European Commission

EURL-SRM

EU Reference Laboratories for Residues of Pesticides
Single Residue Methods

EU Proficiency Test on the Analysis of Rice Flour for incurred and spiked Pesticides Residues Requiring Single Residue Methods

EUPT–SRM15

February/March 2020

Final Report

Chemisches und
Veterinäruntersuchungsamt
Stuttgart

cvua STUTTGART

EU PROFICIENCY TEST
EUPT-SRM15, 2020

Residues of Pesticides Requiring Single Residue Methods

Test Item: Rice Flour

Final Report

Resutls Evaluation

Michelangelo Anastassiades
Pat Schreiter
Anja Barth
Hubert Zipper

approved by Michelangelo Anastassiades
released on 31 March, 2021

The EURL-SRM is accredited by the DAkkS according to EN ISO/IEC 17043.
The accreditation is valid for the proficiency testing programs listed in the certificate.

DAkkS
Deutsche
Akkreditierungsstelle
D-EP-18585-01-00

Publication of parts of this EUPT-SRM15 final report by individuals or organizations shall always refer to the original document that can be found under:
https://www.eurl-pesticides.eu/library/docs/srm/EUPT_SRM15_FinalReport.pdf

Picture on the book cover: Rice, Image by ImageParty from Pixabay

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

Ann-Kathrin Wachtler, Food Chemist

Hubert Zipper, Senior Food Chemist

Anja Barth, Chemical Technician

Giovanna Cerchia, Chemical Technician

Birget Hanke, Chemical Technician

Dorothea Mack, Chemical Technician

Sonja Lachnit, Chemical Technician

Sahra Söhnholz, Chemical Technician

EURL for Pesticides in Cereals and Feedingstuff, National Food Institute, Technical University of Denmark

Anne-Mette Skovlund, Project Manager of Webtool
Steen Maigaard, Senior Software Developer
Sean Gomes, Software Developer
Nicolaj Graversen Pedersen, Software Developer
Wardan Ghazal, Software Developer

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 625/2007/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. The participation of OfLs comparative tests organized by the EURLs has been more recently also layed down in Art 38 (2) of the regulation on offical controls (625/2017/EC), where it reads: "Upon request by the European Union reference laboratory or national reference laboratory, official laboratories shall take part in inter-laboratory comparative tests or proficiency tests that are organised for the analyses, tests or diagnoses they perform as official laboratories". Art 101(1)(a) of Regulation 625/2017/EC furthermore prescribes the participation of NRLs in these comparative tests: "National reference laboratories shall, in their area of competence: (a) collaborate with the European Union reference laboratories, and participate in training courses and in inter-laboratory comparative tests organised by these laboratories"..

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 14 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. Five other 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 the current one EUPT-SRM15, 2020) and maize flour (EUPT-C9/SRM10, 2015) as test items. Further 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 strawberry homogenate (EUPT-SRM12, 2017). The EUPT-SRM9 (2014) was the first EUPT-SRM using a commodity of animal origin (cow's milk), and the EUPT-SRM15 using bovine liver homogenate as the test commodity was the first one EUPT-SRM in cooperation with the EURL for Residues of Pesticides in Food of Animal Origin (EURL-AO).

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. 625/2007/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 in EUPTs. 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.

[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 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 can see their participation status on the registration page. Laboratories 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

EUROPEAN COMMISSION –
EU-PROFICIENCY TEST ON RESIDUES OF PESTICIDES
REQUIRING SINGLE RESIDUE METHODS
TEST ITEM: BOVINE LIVER HOMOGENATE
EUPT-SRM15, 2020

INTRODUCTION

On 28 October, 2019, 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 were invited to participate in the 15th European Commission's Proficiency Test Requiring Single Residue Methods (EUPT-SRM15). The EUPT-SRM15-Website contained links to the Announcement/Invitation Letter and the Calendar. Following consultation with the EUPT-Scientiffic Committee, the EUPT-SRM15 Target Pesticides List, entailing 31 compounds, was released on 13 November, 2019. The selection of the compounds considered the entries within the SANTE working document on pesticides to be considered in national control programmes[1], the relevance of compounds for rice, the availability of analytical standards, the possibility of application during cultivation and the capability of laboratories. On 6 February, 2020, an update of the Target Pesticides List, excluding bromide ion, was released. Among the selected compounds, there are 13 listed in the EU-coordinated Multiannual Control Program for Pesticide Residues and was thus considered as a mandatory compound within this PT. For each compound a residue definition valid for the PT and the minimum required reporting level (MRRL) were stipulated.

On 21 Janury 2020 an e-mail "call for registration" (see. **Appendix 11**) was sent to the participants (see Appendix 11). This e-mail also contained a link to the latest version of the "General Protocol" containing information common to all EUPTs (see **Appendix 8**) ; a link to the document "SupplementaryInformation on Analytes" entailing, among others, information on exemplary sources of analytical standards material (see **Appendix 13**); and a link to the latest version of the QuPPe-AO document, including an analytical procedure for highly polar compounds. The laboratories were able to register on-line from 18 January to 8 February 2020. Two weeks prior to the shipment of the PT material to the laboratories the "Specific Protocol" valid for the current PT was uploaded and a link sent to the participating laboratories via e-mail (see **Appendix 9**). A guide to the new webtool for the results submission (see **Appendix 12**) was provided to the participants one week before the shipment of the PT materials.

Based on commodity scope (food of animal origin) and NRL-status (NRL-SRMs) all laboratories were allocated a tentative status as regards their obligation to participate in the EUPT-SRM15. This status was stored in the DataPool, so that every participant could 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 the respective regulations. Obliged labs that did not intend to participate were asked to provide an explanation.

[1] SANCO/12745/2013 rev. 10(3); 26 – 27 November 2018

In total 57 participating labs from EU and EFTA countries and three laboratories outside EU submitted results of at least one compound. In addition, two laboratories from EU and EFTA countries registerted for participation but did not submit any result.

The proficiency test EUPT-SRM15 was conducted using calves' liver originated from Germany. The test item-was prepared by spiking the finely ground liver, at around 0°C, with standard solutions containing in total 16 compounds, followed by thorrow homogenization, portionation into plastic bags, freezing, cryo-milling with dry ice and portionation into bottles. More details are given **Chapter 1 "Test Materials and Blank Material"**.

1. TEST ITEM

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

In agreement with the EUPT-Scientic Committe and the EURL-CF, rice flour was chosen as commodity for both the EUPT-SRM15 and -CF14 and the raw material for the production of the test material for both EUPTs was organized together.

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) The scope of the SANTE working document on pesticides to be considered in national control programmes (SANCO/12745/2013 rev. 10(3); 26 – 27 November 2019); 3) the relevance of pesticides to the specific commodity; 4) the results of the survey on the analytical capabilities and intentions of the laboratories.

The minimum required reporting levels (MRRLs) were set at 0.01 mg/kg for ***2,4-D (free acid)***, ***carbofuran (sum)***, ***chlormequat-chlorid***, *ethephon, fluazifop (free acid), haloxyfop (free acid), mepiquat-chloride*, ***TFNA***, *TFNG*, ***2,4-D (sum)***, ***bentazone***, ***fluazifop (sum)***, ***haloxyfop (sum)***, ***imazethapyr (free acid)***, ***MCPA (free acid)***, ***MCPA (sum)***, *MCPB (free acid)*, ***MCPB (sum)***, *mecoprop (free acid)*, ***mecoprop (sum)***, ***quizalofop (free acid)*** and ***quizalofop (sum)***; at 0.02 mg/kg for *diquat* and ***paraquat***, and at 0.03 mg/kg for *glufosinate*, ***glyphosate***, *MPP, N-acetyl glufosinate, AMPA* and *N-acetyl glyphosate.*

Based on the fact that most of the rice consumed in Europe is imported from Asia and that rice can be grown in South of Europe only and that in Europe it is prohibited to apply most of the pesticides found in routine food control of rice, the production of the test material was subcontracted to a company/university in India. According to the instructions of EURL-SRM, part of the analytes were applied during cultivation and part of them post harvest. In parallel, rice without any exposure to pesticides was also grown. After harvesting, the rice grains were precessed (peeled and polished). All three parts, i.e. polished rice, husk and bran, were collected and shipped to EURL-SRM for the production of the PT Material.

There were four lots of the treated materials (Lot A1, Lot A3 Lot B1 and Lot B3) and one lot of blank material. Lots A1/B1 contained lower level of pesticides residues than that of Lots A3/B3. On Lots A other compounds were applied than on Lots B. Of all Lots we received polished rice, husks and bran. Details on portions arrived at EURL-SRM are shown in **Table 1-1.** Approximate half of each portion was delivered to EURL-CF for production of the EUPT-CF14 material.

Table 1-1: Composition and amount of rice material arrived at EURL-SRM for preparation of the test material for the EUPT-SRM15 and EUPT-CF14

	Lower Level of Pesticides Residues		Higher Level of Pesticides Residues		Blank Rice
	A1	A3	B1	B3	
Polished Rice [kg]	21	20.7	21.4	15.5	30
Bran [kg]	5.3	4.9	4.1	4	8.1
Hull [kg]	0.4	0.6	0.8	0.8	0.85

1.2 Preliminary Investigation of Treated Materials

In order to prepare spiking solutions for the PT test materials, the presence of the analytes on the Target Pesticides List as well as their residue levels were investigated in different rice and husk lots using four methods: QuEChERS entailing alkaline hydrolysis, modified QuEChERS for various compounds and QuPPe. As expected, the different lots of rice and bran contained different residue levels as shown in **Table 1-2.**

1.3 Investigation on the Analysis of Conjugates/Esters using QuEChERS after Alkaline Hydrolysis

In order to obtain satisfying results of analytes for which the full residue definition contains esters and conjugates the conditions for QuEChERS entailing alkaline hydrolysis were optimized using the following small scale studies.

1.3.1 Analysis of SPIKED Compounds using QuEChERS after Alkaline Hydrolysis and Determined as Free Acids

The impact of different modifications of QuEChERS entailing alkaline hydrolysis in rice matrix on the recovery rate of analytes spiked as acids, esters and glucosides was investigated. Recovery rates show satisfying yields regardless both of the modification of QuEChERS entailing alkaline hydolysis and the kind of conjugate (ester or glucoside) for compounds spiked on rice matrix. Results please see **Table 1-3.**

1.3.2 Analysis of INCURRED Compounds using QuEChERS after Alkaline Hydrolysis and Determined as Free Acids

The impact of different modifications of QuEChERS entailing alkaline hydrolysis in rice matrix on the yield of the sum parameters of free acids, esters and conjugates after alkaline hydrolysis was investigated. For incurred residues different modifications of QuEChERS entailing alkaline hydrolysis result in different yields of residues. There are conjugates which are easily hydrolyses to the their free acid and other conjugates need stronger conditions of alkaline hydrolysis to deliver satisfying yields. Results please see OBSERVATION and **Figure 1-1.**

Table 1-2: Analytes of the different rice lots treated in the filed

Analytes spiked on test material rice in the field						
Analyte	**sprayed**	**Extraction**	**A1**	**A3**	**B1**	**B3**
2.4-D	YES	Q + 1% FA/ Alk. Hydrol. + Q	0.066	0.14	n.n.	n.n.
Bentazone	YES	Q + 1% FA	n.n.	n.n.	0.41	1.3
Bispyribac-sodium	YES	Q + 1% FA	~0.006	~0.007	n.n.	n.n.
Carbofuran	YES	Q + 1% FA	n.n.	n.n.	0.05	0.25
Chlorothalonil	YES	Q + 1% FA	0.009	0.014	n.n.	n.n.
Chlorothalonil 4-OH	Metabolite	Q + 1% FA	n.b.	0.001	n.n.	n.n.
Haloxyfop (sum)	YES	Alk. Hydrol. + Q	n.n.	n.n.	0.15	0.52
Imazethapyr	YES	Q + 1% FA	n.n.	n.n.	0.23	0.84
Paraquat	YES	QuPPe-pos	n.n.	n.n.	0.06	0.2
Propaquizafop	YES	Q + 1% FA	0.014	0.04	n.n.	n.n.
Pymetrozine	YES	Q + 1% FA	0.006	0.016	n.n.	n.n.
Quizalofop (sum)	Metabolite	Alk. Hydrol. + Q	0.028	0.052	n.n.	n.n.

Table 1-3: Recovery rate of spiked analytes in form of I) acidic analytes, II) esters and III) glucoside-ester in rice following various alkaline hydrolysis at 40 °C for 120 min, referred to the spiked concentration as 100 %

	I) Acidic Analytes				
spiked as	2.4-D	Bentazone	Fluazifop	Haloxyfop	Imazethapyr
determined as	2.4-D	Bentazone	Fluazifop	Haloxyfop	Imazethapyr
A: 1 ml NaOH [5 M] + acidic QuEChERS	90 %	87 %	87 %	89 %	86 %
B: 2 ml NaOH [5 M] + QuEChERS	95 %	98 %	97 %	101 %	86 %
C: 2 ml NaOH [5 M] + acidic QuEChERS	101 %	105 %	97 %	103 %	97 %

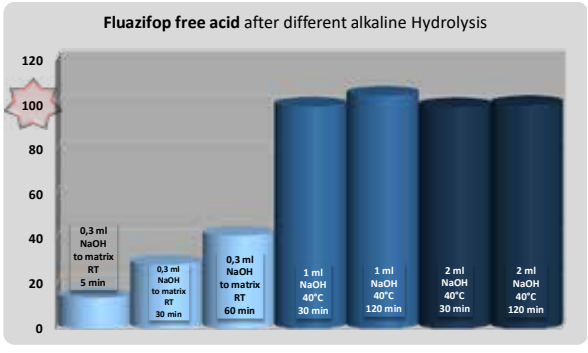
spiked as	MCPA	MCPB	Mecoprop	Quizalofop	
determined as	MCPA	MCPB	Mecoprop	Quizalofop	
A: 1 ml NaOH [5 M] + acidic QuEChERS	87 %	89 %	88 %	91 %	
B: 2 ml NaOH [5 M] + QuEChERS	98 %	111 %	100 %	97 %	
C: 2 ml NaOH [5 M] + acidic QuEChERS	98 %	104 %	102 %	96 %	

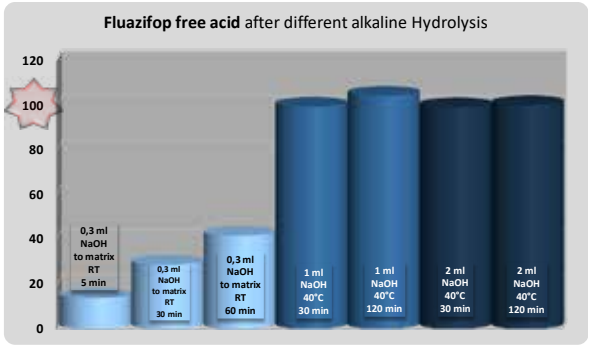
	II) Esters			
spiked as	2,4-D ethylhexyl	Fluazifop butyl	Haloxyfop methyl	MCPA ethylhexyl
determined as	2.4-D	Fluazifop	Haloxyfop	MCPA
A: 1 ml NaOH [5 M] + acidic QuEChERS	95 %	112 %	97 %	90 %
B: 2 ml NaOH [5 M] + QuEChERS	99 %	122 %	106 %	95 %
C: 2 ml NaOH [5 M] + acidic QuEChERS	97 %	115 %	104 %	91 %

spiked as	MCPB ethyl	MCPP trimethylpentyl	Propaquizafop	
determined as	MCPB	Mecoprop	Quizalofop	
A: 1 ml NaOH [5 M] + acidic QuEChERS	95 %	92 %	94 %	
B: 2 ml NaOH [5 M] + QuEChERS	111 %	105 %	106 %	
C: 2 ml NaOH [5 M] + acidic QuEChERS	104 %	104 %	99 %	

	III) Glucoside-Esters		
spiked as	2,4-D-Glucoside	Haloxyfop-Glucoside	MCPA-Glucoside
determined as	2.4-D	Haloxyfop	MCPA
A: 1 ml NaOH [5 M] + acidic QuEChERS	89 %	97 %	84 %
B: 2 ml NaOH [5 M] + QuEChERS	94 %	107 %	91 %
C: 2 ml NaOH [5 M] + acidic QuEChERS	88 %	99 %	81 %

Figure 1-1: Analysis of Incurred Compounds using QuEChERS after Alkaline Hydrolysis and Determined as Free Acids





1.3.3 Method Validation

The method validation for QuEChERS analytes was performed for esters of analytes of interest with QuEChERS entailing alkaline hydolysis. Alkaline Hydrolysis was conducted under different conditions like temperatur, time and the concentration of base during hydrolysis. The best results were obtained by alkaline hydrolysis with 2 ml NaOH [5M] at 40 °C for 120 min (**Table 1-4, p. 4**).

For the QuPPe-Method validation for analytes amenable to QuPPe please see: https://www.eurl-pesticides.eu/userfiles/file/meth_QuPPe_AO_V3_2.pdf.

1.4 Production, Bottling and Packaging of the Test Item

For the preparation of the test item 2 kg of rice blank material were spiked with 205 ml spiking solutions prepared according to **Table 1-5.** After evaporation the spiked rice together with 10 kg of polished rice from Lot A1, Lot A3 and Lot B1 each and 7.26 kg from Lot B3 were mixed with a drum-hoop mixer over night. Afterward, they were milled in portions with a rotor beater mill (Retsch Rotor Beater Mill SR 300) equipped with a 0.5 µm sieve. In order to avoid overheating an process milling continuously, approximately 750 ml rice grains was manually premixed with 250 ml dry ice pellets (3 mm) prior to milling. The milled material was remixed with a drum-hoop mixer over 10 h and weighted out in approx. 180 – 200 g portions into screw capped polyethylene plastic bottles. The bottles were numbered chronologically in the order of filling and sealed. After all test materials were bottled and sealed, one randomly choosen bottle with test item was packed into thermo-insulated polystyrene boxes, filled with two cooling elements and stored in a walk-in freezer at about –20 °C until packaging and dispatch to the participants. Parcel packaging was proceeded three days prior to the sample delivery, so that on the day of delivery the cooling elements were deep frozen.

Table 1-4: Results of method validation for analytes amenable to QuEChERS entailing alkaline hydrolysis with 2 ml NaOH [5M] at 40 °C for 120 min.

Analytes		Rec.	RSD*	Analytes		Rec.	RSD*
spiked at 0.2 ppm	analyzed as	[%]	[%]	spiked at 0.2 ppm	analyzed as	[%]	[%]
2,4,5-T-isooctyl	2,4,5-T	92	5	Fluroxypyr meptyl	Fluroxypyr	91	3
2,4-D ethylhexyl	2,4-D	92	3	Haloxyfop methyl	Haloxyfop	96	2
2,4-DB methyl	2,4-DB	89	3	Ioxynil-octanoate	Ioxynil	96	4
2,4-DP ethylhexyl	2,4-DP	101	4	MCPA ethylhexyl	MCPA	85	4
Bromoxynil-heptanoate	Bromxynil	100	4	MCPB ethyl	MCPB	87	3
Cyhalofop-butyl	Cyhalofop	89	3	Mecoprop trimethylpentyl	Mecoprop	96	1
Diclofop-methyl	Diclofop	98	1	Propaquizafop	Quizalofop	97	8
Fluazifop butyl	Fluazifop	98	3	Triclopyr-2-butoxyethyl ester	Triclopyr	96	4
Analytical replicates: n=3; RSD*: Relative Standard Deviation							

Table 1-5: Analytes spiked into 2 kg blank rice for the preparation of approximate 40 kg test material

Analytes spiked using stock solutions (1 mg/ml H_2O), mixed with ~90 ml acetone for total approx. 40 kg test material				
Analyte	**Amount [ml]**	**Theor. Spiked Conc. [mg/kg]**	**Incurred Conc. [mg/kg]**	**Theo. Final Conc. [mg/kg]**
Chlormequat-Cl ‡	2.8	0.085		0.085
Glyphosate	8.0	0.200	–	0.200
Paraquat	7.2	0.180	~0.055	~0.235
Analytes spiked using stock solutions (1 mg/ml ACN), mixed with ~70 ml acetone for total approx. 40 kg test material				
Analyte	**Amount [ml]**	**Theor. Spiked Conc. [mg/kg]**	**Incurred Conc. [mg/kg]**	**Theo. Final Conc. [mg/kg]**
Carbosulfan ¶	4.8	0.070	~0.065	~0.130
Fluazifop-butyl #	3.0	0.064	–	0.064
MCPA-Glucoside	5.5	0.076*	–	0.076*
MCPB-methyl	2.8	0.066*	–	0.066*
Mecoprop-trimethylpentyl	4.5	0.074*	–	0.074*
Quizalofop	4.0	0.050	~0.020	~0.070
TFNA	3.0	0.075	–	0.075

‡ Stock solution: 1 mg chlormequate / ml; concentration in the spiked material referred to mg chlormequate-chlorid / kg
¶ Stock solution: 1 mg carbosulfan / ml; concentration in the spiked material referred to mg carbofuran / kg
# Stock solution: 1 mg Fluazifop / ml; concentration in the spiked material referred to mg Fluazifop-butyl / kg
* as the corresponding free acid (sum)

1.5 Delivery of PT Materials to Participants

On the day of dispatch, both PT corresponding persons of one participating laboratory received an e-mail from the shipping company (DHL Germany) entailing the individual online tracking number.

Among the 117 packages sent to laboratories in EU and EFTA countries, 105 (90 %) reached the participating labs within 24 hours, 9 packages within 48 hours and only one package arrived the recipient laboratory on the third day due to remote location. The delivery to countries outside the EU and EFTA zones was accomplished within 48 hours in three cases, 3 days in 2 cases as well as 5, 7 and 9 days for each of one case. The latter was, however, due to delays at the customs. Overall, the vast majority of the parcels arrived at the laboratories within two days. Details on the shipment duration are shown in **Appendix 2**. All dispatched material was accepted by the participants, and they reported its good condition when arriving, even arrival on the third or fourth day. Based on this results, it was judged that differences in shipment duration have had most likely no significant influence on the analyte concentrations (and the analytical results of the laboratories), and it was thus decided not to test the impact of shipment duration on analyte stability (see also **1.9 „Transport Stability Test"**).

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., providing the customs with all necessary documents and asking for an acceleration of the clearance procedure or placing 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 timing.

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 which were neither applied in the field nor spiked in the laboratory are summarized in **Table 1-6**. For more details on the methods used, please refer to the EURL-SRM website: http://www.eurl-pesticides.eu (EURL-SRM-website → EURL-SRM Methods or Analytical Observations).

During the investigation on analysis of conjugates/esters via alkaline hydrolysis prior to QuEChERS using the official method varying results were obtained in conjunction with the hydrolysis conditions like hydrolysis temperature and duration. Therefore, on 04.03.2020 the organizer distributed a method version for "Acidic Pesticides following hydrolysis" (SRM-43/(V1): https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EurlSrm_Observation_alkaline_hydrolysis_acidic_herbicides.pdf. The participants were welcome to use this procedure or any other methods.

1.7 Homogeneity Test

After filling the test item into the bottles, 10 bottles were randomly chosen for the homogeneity test and two analytical portions were taken from each bottle 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. For all compounds analytical portions of 5 g 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 1-7**. 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-SRM15.

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. The portions of stability tests 1 were taken and extracted immediately. The remaining material for the stability tests 2 and 3 were placed in the freezer at –20 °C until performing the tests. The methods described in **Section 1.6 (p. 6)** are applied for the tests**4**. The extracts of all stability tests corresponding to one method were stored in the freezer at –20 °C and measured iso-chronically (within the same sequence) at a day suitable for the laboratory.

Table 1-6: 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-D	**Modified QuEChERS-Method (Acidic-QuEChERS) :** weighing of 5 g rice flour into a sealable vessel, addition of 10 mL water and IS / ILISs, extraction with ACN + 1 % formic acid (15 min), addition of partitioning salts (4 g $MgSO_4$, 1 g NaCl), 1 min shaking, centrifugation (~4000 rpm, 5 min) and direct determination by LC-MS/MS in the ESI (neg.) mode.	Mecoprop D_6	LC-MS/MS	ESI (neg)	
TFNA		Mecoprop D6	LC-MS/MS	ESI (neg)	
Bentazone		no IS	LC-MS/MS	ESI (neg)	
Imazethapyr		Mecoprop D_6	LC-MS/MS	ESI (neg)	
Quizalofop		Mecoprop D_6	LC-MS/MS	ESI (neg)	
*Fluazifop**		*Mecoprop D_6*	*LC-MS/MS*	*ESI (neg)*	
*Haloxyfop**		*Haloxyfop D_4*	*LC-MS/MS*	*ESI (neg)*	
*TFNG**		*Mecoprop D_6*	*LC-MS/MS*	*ESI (neg)*	
*MCPA**		*Mecoprop D6*	*LC-MS/MS*	*ESI (neg)*	
*MCPB**		*Mecoprop D_6*	*LC-MS/MS*	*ESI (neg)*	
*Mecoprop**		*Mecoprop D_6*	*LC-MS/MS*	*ESI (neg)*	

Compound	Extraction	IS	Determinative analysis		Notes
Carbofuran sum	**QuEChERS entailing Acidic Hydrolyses :** Using the extract of QuEChERS method. Addition of 10 µL H_2SO_4 5 N to 1 mL extract in a vial before heating for 3 h at 80 °C. Direct determination by LC-MS/MS in the ESI (pos) mode.	Propyzamide D_3	LC-MS/MS	ESI (pos)	SRM-33

Compound	Extraction	IS	Determinative analysis		Notes
2,4-D sum	**Modified QuEChERS Method (Acidic QuEChERS entailing Alkaline Hyadolyses) :** weighing of 5 g rice homogenate into a sealable vessel, optional addition of ILISs, water adjustment, addition of ACN and NaOH 5N. Extraction at 40°C in a waterbath for 120 min. Neutralisation with H_2SO_4, addition of IS, acidification with 100 µl formic acid and addition of partitioning salts without buffer (4 g MgSO4, 1 g NaCl), 1 min shaking, centrifugation (~4000 rpm, 5 min) and direct determination by LC-MS/MS in the ESI (neg).	Mecoprop D_6	LC-MS/MS	ESI (neg)	SRM-43/(V1)
Fluazifop sum		Propyzamide D_3	LC-MS/MS	ESI (neg)	SRM-43/(V1)
Haloxyfop sum		Haloxyfop D_4	LC-MS/MS	ESI (neg)	SRM-43/(V1)
MCPA sum		Mecoprop D_6	LC-MS/MS	ESI (neg)	SRM-43/(V1)
MCPB sum		Mecoprop D_6	LC-MS/MS	ESI (neg)	SRM-43/(V1)
Mecoprop sum		Mecoprop D_6	LC-MS/MS	ESI (neg)	SRM-43/(V1)
Quizalofop sum		Mecoprop D_6	LC-MS/MS	ESI (neg)	SRM-43/(V1)

Compound	Extraction	IS	Determinative analysis		Notes
Chlormequat	**QuPPe-AO Method :** weighing of 5 g rice homogenate into a sealable vessel, addition of ILISs, water adjustment, addition of methanol containing 1 % formic acid, addition of formic acid and EDTA, shaking, freeze-out, centrifugation, clean-up/ precipitation with C18 and ACN, filtration with Ultrafiltration filters and direct determination by LC-MS/MS in the ESI (neg./pos.) mode.	Chlormequat D_4	LC-MS/MS	ESI (neg)	QuPPe M4.1
Glyphosate		Glyphosate $^{13}C^{15}N$	LC-MS/MS	ESI (neg)	QuPPe M1.6
Paraquat		Paraquat D_8	LC-MS/MS	ESI (neg)	QuPPe M4.1
*Ethephon**		*Ethephone D_4*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Glufosinate**		*Glufosinate D_3*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Mepiquat**		*Mepiquat D_3*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M4.1*
*MPP**		*MPP D_3*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*N-acetyl-glufosinate**		*N-acetyl-glufosinate D_3*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*AMPA**		*AMPA $^{13}C^{15}N$*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Diquat**		*Diquat D_8*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M4.1*
*N-acetyl-glyphosate**		*N-acetyl-glyphosate $^{13}C_2$, ^{15}N*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*

* : To check for absence in Blank Material

Stability test 1 (extraction shortly before shipment):
14 February 2020 (analytes via QuPPe-AO-Method)
02 March 2020 (analytes via Acidic QuEChERS-Method/ QuEChERS + Acidic Hydolysis)
02 March 2020 (analytes via QuEChERS entailing Alkaline Hydrolysis)

Stability test 2 (extraction 10 days after shipment):
13 March 2020 (analytes via QuPPe-AO-Methods)
24 March 2020 (analytes via Acidic QuEChERS-Method/ QuEChERS + Acidic Hydolysis)
24 March 2020 (analytes via QuEChERS entailing Alkaline Hydrolysis)

Stability test 3 (extraction at the end of PT):
22 April 2020 (analytes via QuPPe-AO-Methods)
07 May 2020 (analytes via Acidic QuEChERS-Method/ QuEChERS + Acidic Hydolysis)
12 May 2020 (analytes via QuEChERS entailing Alkaline Hydrolysis)

A target compound is considered to be sufficiently 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. Within the stability test, in which the samples were stored at –18 °C (= recommended conditions) over a period exceeding the duration of the PT, all analytes contained in the test item were shown to be sufficiently stable (**Table 1-8, p. 10**). For the compounds passing the test it was 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.

The detailed results of all analyses conducted within the framework of the stability test are shown in **Table 1-8 (p. 10)** and **Appendix 4**.

1.9 Transport Stability Test

Except three laboratories outside the EU and EFTA all other participants received the sample packages within three days and in very good conditions. The results reported by the three laboratories having received the material on the fifth, seventh and ninth day did not imply any degradation of compounds. For these reasons, the organizer decided to skip the transport stability test in this PT.

1.10 Organisational Aspects

1.10.1 Laboratory Status – Mandatory and Optional Participation

Based on available information on NRL-status and commodity scope as recorded in the EURL-DataPool, the EU and EFTA OfLs and NRLs were preliminarily divided into those that were obliged to participate in the particular PT and those whose participation was voluntary. The available information on the pesticide scope covered by the laboratories was not considered due to concerns that it might not be up-to-date and/ or not applicable to the present commodity (rice). The OfLs were asked to update their status and scope several months prior to the PT. The NRLs were furthermore reminded of their responsibility of ensuring that the information concerning their network is up-to-date and that all obliged OfLs within their network were

Table 1-7: Statistical evaluation of homogeneity test data (n = 10), details please see **Appendix 3**.

	COMPULSORY					OPTIONAL COMPOUNDS		
	2,4-D (free acid)	Carbofuran (sum)	Chlormequat-Cl	Glyphosate	TFNA	2,4-D (sum)	Bentazone (free acid)	Fluazifop (sum)
Analytical portion size [g]	5	5	5	5	5	5	5	5
Mean [mg/kg]	0.056	0.097	0.083	0.20	0.064	0.055	0.339	0.067
s_{sam}^2	6.5×10^{-7}	0×10^{-0}	5×10^{-7}	0×10^{-0}	0×10^{-0}	5.5×10^{-7}	6.2×10^{-6}	0×10^{-0}
c	0.0042	0.0073	0.0062	0.015	0.0048	0.0041	0.025	0.005
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed
	OPTIONAL COMPOUNDS							
	Haloxyfop (sum)	Imazethapyr (free acid)	MCPA (sum)	MCPB (sum)	MCPP (sum)	Paraquat	Quizalofop (free acid)	Quizalofop (sum)
Analytical portion size [g]	5	5	5	5	5	5	5	5
Mean [mg/kg]	0.15	0.22	0.064	0.058	0.068	0.233	0.046	0.063
s_{sam}^2	2.4×10^{-6}	5.9×10^{-6}	0×10^{-0}	0×10^{-0}	1.4 x 10-7	1.8 x 10-5	0×10^{-0}	0×10^{-0}
c	0.012	0.016	0.0048	0.0043	0.0051	0.017	0.0035	0.0047
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed

informed of this EUPT. All NRLs and OfLs were informed that the division into "obliged" and "voluntary" was tentative and the real obligation for participation is derived from the respective regulations and their real scope.

Following DG-SANTE instructions, obliged labs that were not intending to participate in the EUPT-SRM15 were instructed to provide explanations for their non-participation.

1.10.2 Announcement / Invitation and EUPT-SRM15-Website

The EUPT-SRM15 was firstly scheduled to run from 27 January till 25 February, 2020. Within the EURL-Web-Portal an EUPT-SRM15-Website was set up on 14 October 2019 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.

On 29 October 2019 the Announcement/Invitation Letter for the EUPT-SRM15 was published on the EUPT-SRM15-Website and 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 analys-

Table 1-8: 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					Optional Compounds		
	2,4-D (free acid)	Carbofuran (sum)	Chlormequat-Cl	Glyphosate	TFNA	2,4-D (sum)	Bentazone (free acid)	Fluazifop (sum)
Storage at –18 °C (mean values in mg/kg)								
Analysis 1	0.059	0.096	0.082	0.202	0.066	0.055	0.35	0.066
Analysis 2	0.056	0.095	0.079	0.205	0.063	0.058	0.35	0.069
Analysis 3	0.056	0.10	0.084	0.205	0.063	0.058	0.34	0.065
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.003 (-5.1%)	0.004 (4.2%)	0.002 (2.4%)	0,003 (1.3%)	0.003 (-4.1%)	0.003 (5.1%)	0.011 (-3.2%)	0.0006 (-1.0%)
$0.3 \times \sigma_{pt}$ [mg/kg]	0.0039	0.00795	0.0069	0.015	0.0045	0.004	0.025	0.005
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed

	Optional Compounds							
	Haloxyfop (sum)	Imazethapyr (free acid)	MCPA (sum)	MCPB (sum)	MCPP (sum)	Paraquat	Quizalofop (free acid)	Quizalofop (sum)
Storage at –18 °C (mean values in mg/kg)								
Analysis 1	0.15	0.22	0.063	0.057	0.068	0.23	0.047	0.062
Analysis 2	0.15	0.21	0.066	0.059	0.073	0.22	0.048	0.061
Analysis 3	0.15	0.21	0.067	0.059	0.072	0.23	0.048	0.059
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.005 (3.1%)	0.007 (-2.9%)	0.004 (5.8%)	0.002 (3.5%)	0.004 (5.9%)	0.009 (-3.9%)	0.0008 (1.8%)	0.003 (-4.6%)
$0.3 \times \sigma_{pt}$ [mg/kg]	0.011	0.015	0.005	0.004	0.005	0.015	0.003	0.005
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed

ing 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 the respective regulations, 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. Eight laboratories outside EU having registered for this PT were accepted to take part in this exercise. As always the results from laboratories outside EU and EFTA were not taken into accout for the establishment of the assigned values.

Due to difficulties to obtain suitable rice material with incurred pesticides for production of the test material for EUPT-SRM15 and EUPT-CF14 and in agreement with EURL-CF, -FV and -AO, the period for the EUPT-SRM15 was shifted from 10 February to 10 March 2020 that was communicated with the SRM15 participants on 19 December 2019. Due to delayed access to the webtool and in order to overcome some technical difficulties, the SRM15 deadline was firstly postponed to 17 March 2020 and finally to 31 March 2020 in order to take account of movement restrictions in conjunction with corona virus outbreak. During this extended submission period the participants had the possibility of accessing to the webtool from their home office and were asked to correct inconclusive/inconsistent method information entries.

1.10.3 Registration

Like in the previous EUPTs since 2017 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.

1.10.4 Additional Information provided to the participants

On 5 March, following some requests, the participants were provided with a document dealing with the analysis of acidic pesticides following alkaline hydrolysis and with links to other recently published documents by the EURL-SRM.

1.10.5 Distribution of the Test Items and the Blank Material

Except one participating laboratory with special agreement with the organiser to which the PT material was dispatched on 12 February, one deeply frozen bottle of test item (approx. 200 g) packed in a thermo-insulated polystyrene box including two gel-packs was shipped on 10 February, 2020 to each participating laboratory.

On 23 January, 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**).

1.10.6 Webtool for Results Submission and Confidentiality

The "Webtool", an online submission tool allowing participants to submit sample acknowledgement and their results via a web browser, was constructed and used 2019 for the first time for all EUPTs on pesticides residues. This Webtool is utilized onwards. The login credentials unique to the registered email address of the PT responsible person are created after registration in the Webtool for the first time. They were sent to the registered email address before the Webtool was open for sample acknowledgement for a certain PT. Using his personal login credentials, the participant has an overview of all EUPTs on pesticides residues under this account and has access to results submission during the PT period.

The lab code of a laboratory for a certain PT is obtained when a participant, either as PT main or alternative contact person, login to this PT. The personal login credentials and the unique lab code for a certain PT warrantee the confidentiality. For further information on confidentiality please refer to the General EUPT Protocol (**Appendix 9**).

Due to unexcepted technical difficulties the Webtool became accessible one week after sample dispatch and the participants have received their login credentials from the programmer at the DTU. Via the Webtool all participants had access to the sample acknowledgement and results submission until the result submission deadline (31 March 2020). One week prior to the sample shipment the organizer provided the A guideline on the Webtool, including how to login to the webtool, how to get the lab code for the EUPT-SRM15, as well as all fields to be filled in., was provided as a link to the participants together with the Specific Protocol on 23 January. After the deadline, participants were informed on the presence of the spiked

pesticides and if they had false negative results. In the latter case, they should report method details for compounds of false negative results via the Webtool.

The schedule of sample shipment and submission deadline was embedded in a workflow of the Webtool. Based on these two dates a reminder of certain activity is automatically sent to the participants. The Webtool for a dry matrix operated for the first time, contained a few errors and once started, the workflow could be stopped only hardly. Due to an error in query, the participants received incorrect information on false negative results. The software developer are working on improvement. In addition, five analytes (free acid of Fluazifop, Haloxyfop, MCPA, MCPB and Mecoprop) were present in the test material but at levels closed to the MRRL and the reporting limits of the most laboratories. The organizers have informed the participants having analysed for but not found these analytes and receiving false negative results that they should not regarded as false negative results.

1.10.7 Actions following Results Submission, Preliminary Report and Survey

Four EU-Laboratories and 1 EFTA-laboratory had originally registered to participate in the current PT but finally were not able to submit results due to corona crisis. One laboratory having started to submit its results was not able to complete its submission due to corona-shotdown. It was going to use its PT-results for internal quality control. Its results were not included in the establishment of the assigned values, and the z-scores of this laboratory were calculated for informative purpose only.

On 5 June, 2020, the preliminary report on the EUPT-SRM15 with the preliminary assigned values was released and sent to the participants. Due to an error that the results form the two participating laboratories in Serbia were not excluded from the results population for the establishment of the assigned values, the assigned values and z-scores in the preliminary report may differ slightly from those in the present Final Report. As the difference is not significant, no update report was followed.

The preliminary report contained comments from the organizers driven from the method information given by the participating laboratory. Those can be helpful to the participants to identify the error sources. 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 make method-based evaluation, 63 selected laboratories were additionally asked for more detailed information about their analytical methods for carbofuran (sum).

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 than MRRL. 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.

Using the assigned value derived by robust mean, the z-scores of the participants' results were calculated

using the formula in **Section 2.4**. All Results achieving z-scores > 5 are regarded as outliers and excluded from the results population for the establishment of the assigned value, and the corresponding analyte is calculated again without those outliers.

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

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.

2.5.1 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

122 laboratories from 37 countries (27 EU-Member States, 3 EFTA-countries, 1 EU candidate country and 6 countries outside Europa) originally registered for participation in the EUPT-SRM15. 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**. Malta was represented by its proxy-NRL-SRM based in the United Kingdom and one laboratory based in Germany with two subsidiaries for its routine analysis.

Among the 114 EU and EFTA OfLs having registered for participation in the current PT one obliged laboratory in France (SRM15-10) was not able to complete its submission due to COVID-19 lockdown. The results reported by this laboratory was not included in the establishment of the assigned values and their evaluation in Chapter 4 was for informative purpose only. From the same reason, two other obliged laboratories (each one from IT and ES) and two voluntary laboratories (each one from PL and ES) and one EFTA laboratory (CH) failed to submit any results. Finally, there were 108 EU/EFTA laboratories having submitted at least one result for this PT.

All 8 laboratories from non-EU/EFTA countries (2 from one EU candidate country and 6 from countries outside Europa) submitted results. The results submitted by these 8 laboratories located outside EU and EFTA countries were not taken into account when calculating the assigned values.

In total, 155 EU-OfLs, including NRL-SRMs, regardless of their commodity scope, as well as all EU-OfLs analysing for pesticide residues in food and feed of plant origin, were originally considered as being obliged to participate in the present EUPT. These laboratories were invited to register on the online registration page 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 (rice) or the SRM15 target pesticides or both were out of their routine scope. Another one obliged laboratory (IT) was not able to participate in the surrent PT due to lack of personnel. Excluding those 31 laboratories that provided sufficient explanations and the additional 3 EU-OfLs that due to COVID-19 lockdown were not able to complete submission or report any results, the number of EU-laboratories considered as being obliged decreased to 121. Among the 98 obliged laboratories that have registered for this PT 98 laboratories finally submitted result. In addition, 13 EU-OfLs registered for participation on voluntary basis, and 11 of them submitted results. Out of the 155 obliged OfLs 26 (17 %) did neither register for the PT nor provide any explanation for non-participation. These laboratories originated from 8 countries as follows: IT (10×), each 4 from PL and ES, each 3 from DE and RO as well as each one from FR and UK.

Table 3-1: Number of laboratories listed as being obliged to participate in the EUPT-SRM15, labs that registered to participate, and labs that finally submitted results (grouped by contracting country)

EU: NRLs and OfLs									
					Registered for Participation obliged + [on voluntary basis]		Submitted Results		
Contracting Country [1)]	Labs originally considered to be obliged (*based on scope and NRL)	Labs providing expl. for non-participation	Obliged labs non particip. w/o giving expl.	Finally considered to be obliged	All	NRL-SRMs	All	NRL-SRMs	Notes
AT	1	0	0	1	1	1	1	1	
BE	6	1	0	5	5+[1]	1	5+[1]	1	
BE; NL	1	0	0	1	1	0	1	0	
BE; BG; FR; LU	1	0	0	1	1	0	1	0	
BG	2	1	0	1	1	1	1	1	
CY	2	1	0	1	1	1	1	1	
CZ	3	0	0	3	3	1	3	1	
DE	22	2	3	20	17+[2]	1	17+[2]	1	
DK	2	1	0	1	1	1	1	1	
EE	2	0	0	2	2	1	2	1	
FI	2	0	0	2	2	2	2	2	
FR	9	0	1	9	8+[1]	1	7+[1]	1	1 none submission (obliged)
GR	3	1	0	2	2	2	2	2	
HR	8	3	0	5	5	1	5	1	
HU	4	0	0	4	4	1	4	1	
IE	1	0	0	1	1	1	1	1	
IT	25	6	10	19	9+[1]	1	8+[1]	1	1 none submission (obliged)
LT	2	0	0	2	2	1	2	1	
LU	1	0	0	1	1	1	1	1	
LV	1	0	0	1	1	1	1	1	
MT	3	2	0	1	1+[1]		1+[1]		MT: subcontracts UK-NRL-SRM as Proxy-NRL + one lab in DE with two subsidiaries for routine controls
NL	1	0	0	1	1	1	1	1	
PL	11	5	4	6	2+[4]	1	2+[3]	1	1 none submission
PT	4	1	0	3	3	1	3	1	
RO	7	2	3	5	2	1	2	1	
SE	2	0	0	2	2	1	2	1	
SI	3	1	0	2	2	1	2	1	
SK	1	0	0	1	1	1	1	1	
ES	23	4	4	19	15+[2]	2	14+[1]	2	2 none submission (one of them obliged)
UK	1	0	1	1					
UK; MT	1	0	0	1	1	1	1	1	UK-NRL-SRM functions also for MT-NRL-SRM
EU Total	155	31	26	124*	**98+[13]**	**30**	**95+[11]**	**30**	

* Excluding 3 obliged EU OfLs that due to COVID-19 lockdown were not able to complete submission or report any result, the number reduced to 121.

Table 3-1 (cont.): Number of laboratories listed as being obliged to participate in the EUPT-SRM15, labs that registered to participate, and labs that finally submitted results (grouped by contracting country)

EFTA: NRLs and OfLs									
					Registered for Participation obliged + [on voluntary basis]		Submitted Results		
Contracting Country [1)]	Labs originally considered to be obliged (*based on scope and NRL)	Labs providing expl. for non-participation	Finally considered to be obliged	Obliged labs non particip. w/o giving expl.	All	NRL-SRMs	All	NRL-SRMs	Notes
CH					[1]	0	0	0	1 none submission
IS					[1]	0	[1]	0	
NO					1	1	1	1	
EU+EFTA Total					**99+[15]**		**96+[12]**		
Countries outside Europa									
BR	1				1		1		
BY	1				1		1		
HK/CN	1				1		1		
CR	1				1		1		
RS	2				2		2		
SG	1				1		1		
TH	1				1		1		
Countries outside Europa Total					**8**		**8**		

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. 21)** 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. 21)**.

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* =108 [3])	No. [2]	% (based on *n* = 121[4])
Compulsory Compounds	**2,4-D (free acid)**	**Yes**	89	82 %	81	67 %
	Carbofuran (sum)	**Yes**	92	85 %	81	67 %
	Chlormequat-Cl	**Yes**	88	81 %	78	64 %
	Ethephon	No	73	68 %	64	53 %
	Fluazifop (free acid)	Trace	85	79 %	77	64 %
	Glufosinate	No	70	65 %	63	52 %
	Glyphosate	**Yes**	84	78 %	76	63 %
	Haloxyfop (free acid)	Trace	87	81 %	78	64 %
	Mepiquat-Chloride	No	87	81 %	78	64 %
	MPP (= MPPA)	No	55	51 %	49	40 %
	N-Acetyl glufosinate	No	51	47 %	45	37 %
	TFNA	**Yes**	72	67 %	64	53 %
	TFNG	No	71	66 %	63	52 %
Optional Compounds	**2,4-D (sum)**	**Yes**	66	61 %	58	48 %
	AMPA	No	70	65 %	62	51 %
	Bentazone	**Yes**	77	71 %	68	56 %
	Diquat, expr. as dication	No	37	34 %	30	25 %
	Fluazifop (sum)	**Yes**	66	61 %	58	48 %
	Haloxyfop (sum)	**Yes**	65	60 %	57	47 %
	Imazethapyr (free acid)	**Yes**	40	37 %	33	27 %
	MCPA (free acid)	Trace	77	71 %	70	58 %
	MCPA (sum)	**Yes**	66	61 %	58	48 %
	MCPB (free acid)	Trace	66	61 %	59	49 %
	MCPB (sum)	**Yes**	60	56 %	52	43 %
	Mecoprop (free acid)	Trace	67	62 %	61	50 %
	Mecoprop (sum)	**Yes**	59	55 %	52	43 %
	N-Acetyl glyphosate	No	34	31 %	31	26 %
	Paraquat	**Yes**	34	31 %	28	23 %
	Quizalofop (free acid)	**Yes**	64	59 %	58	48 %
	Quizalofop (sum)	**Yes**	54	50 %	48	40 %

1) Including official laboratories participating on voluntary basis
2) Laboratories representing more than one country were counted only once.
3) 108 OfLs from EU and EFTA countries (incl. NRLs and OfLs participating on voluntary basis) have submitted at least one result.
4) 121 OfLs (including NRLs) from EU countries were finally considered as obliged to participate in the EUPT-SRM15 (taking into account any explanations for non-participation).

Table 4-2: Scope and categorization of participating laboratories (including third country laboratories and one laboratory having started submission but was not able to complete it due to COVID-19 lockdown)

			Compulsory Compounds														Optional Compounds		
Compounds listed on the Target List			2,4-D (free acid)	Carbofuran (sum)	Chlormequat-Cl	Ethephon	Fluazifop (free acid)	Glufosinate	Glyphosate	Haloxyfop (free acid)	Mepiquat-Chloride	MPP (= MPPA)	N-Acetyl glufosinate	TFNA	TFNG	analysed / correctly found (Compulsory Compounds)	2,4-D (sum)	AMPA	Bentazone
within MACP			MACP	MACP	MACP + WD	MACP	MACP	MACP	MACP + WD	MACP	MACP + WD	MACP	MACP	MACP	MACP		MACP	WD*	
present in Test Item			Yes	Yes	Yes	No	Trace	No	Yes	Trace	No	No	No	Yes	No		Yes	No	Yes
evaluated in this PT			Yes	Yes	Yes	No	No	No	Yes	No	No	No	No	Yes	No		Yes	No	Yes
Lab-Code SRM15-	NRL	Cat.																	
5		B	FN													1 / 0	V		
8	×	B		V	V	ND		ND	V		ND	ND	ND	V	ND	10 / 4	V	ND	V
9	×	B	V	V	V	ND	ND	ND	V	ND	ND	ND	ND			11 / 4	V	ND	V
11		B	V	V	V		ND		V	ND	ND					7 / 4			
12	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
13	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND		V	ND	12 / 5	V	ND	V
16	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
18		B		V												1 / 1			
19		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
20	×	B	V	V	V				V	ND	ND					6 / 4		ND	V
21	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V		V
22	×	B	V	V	V	ND	ND		V	ND	ND			V	ND	10 / 5	V		V
23	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5		ND	V
24		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
25		B	V	V	V		ND			ND	ND					6 / 3	V		V
26		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
27		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V		V
28		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5		ND	V
30		B	V	V	V	ND	ND	ND	V	ND	ND			V	ND	11 / 5	V	ND	V
31		A	V		V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	12 / 4	V	ND	V
32		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
33		B	FN	V	V			ND	V		ND					6 / 3	V	ND	V
34		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
35		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5		ND	V
36		B							V							1 / 1		ND	
37		B	V	V	V	ND	ND		V	ND	ND			V	ND	10 / 5	V		V
38		B		V	V						ND					3 / 2			
39		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5		ND	V
40		B	V	V			ND			ND				V	ND	6 / 3	V		V
41	×	B	FN	V		ND		ND	V		ND					6 / 2			

*: Future residue definition; **MACP-Reg.**: REGULATION (EU) 2019/533 of 28 March 2019; **NCP-WD**: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 26–27 November 2018rev. 10(3)

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 one laboratory having started submission but was not able to complete it due to COVID-19 lockdown)

			Optional Compounds														Total
Compounds listed on the Target List			Diquat, expr. as dication	Fluazifop (sum)	Haloxyfop (sum)	Imazethapyr (free acid)	MCPA (free acid)	MCPA (sum)	MCPB (free acid)	MCPB (sum)	Mecoprop (free acid)	Mecoprop (sum)	N-Acetyl glyphosate	Paraquat	Quizalofop (free acid)	Quizalofop (sum)	analysed / correctly found (Optional Compounds)	analysed / correctly found (Total)
within MACP			WD	MACP	MACP		WD	WD	WD	WD			WD*	WD	WD	WD*		
present in Test Item			No	**Yes**	**Yes**	**Yes**	Trace	**Yes**	Trace	**Yes**	Trace	**Yes**	No	**Yes**	**Yes**	**Yes**		
evaluated in this PT			No	**Yes**	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**		
Lab-Code SRM15-	NRL	Cat.																
5		B															1/1	2/1
8	×	B	ND	V	V	V		V		V		**FN**	ND	V		**FN**	13/8	23/12
9	×	B		V	V		ND	V			ND	V			V	V	11/8	22/12
11		B															0/0	7/4
12	×	A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17/11	30/16
13	×	A	ND	V	V		ND	V	ND	V	ND	V	ND	V	V	V	16/10	28/15
16	×	A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17/11	30/16
18		B															0/0	1/1
19		A	ND	V	**FN**		ND	V	ND	V	ND	V	ND	V	V	V	16/9	29/14
20	×	B		V			ND				ND				V		6/3	12/7
21	×	A	ND	V	V		ND	V	ND	V	ND	**FN**		V	V	V	14/9	27/14
22	×	B		V	V		ND	V	ND	V	ND	V			V	V	12/9	22/14
23	×	A					ND		ND		ND		ND		V		7/2	20/7
24		A	ND	V	V		ND	V	ND	V	ND	**FN**	ND	V	V	V	16/9	29/14
25		B		V	V		ND	V	ND	V	ND	V			V	V	12/9	18/12
26		A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17/11	30/16
27		A		V	V		ND	V	ND	V	ND	V	ND				11/7	24/12
28		A				V	ND		ND		ND				V		7/3	20/8
30		B	ND	V	V	V	ND	V	ND	**FN**	ND	**FN**		V	V		15/8	26/13
31		A		V	V		ND	V					ND		V	V	10/7	22/11
32		A		V	V		ND	V	ND	V	ND	V	ND		V	V	14/9	27/14
33		B					ND	V	ND	V	ND	V					9/5	15/8
34		A	ND	V	V	V	ND	**FN**	ND	**FN**	ND	**FN**	ND	V	**FN**	V	17/7	30/12
35		A				V	ND		ND		ND		ND		V		8/3	21/8
36		B															1/0	2/1
37		B		V	V		ND	V	ND	V	ND	**FN**			V	V	12/8	22/13
38		B															0/0	3/2
39		A									ND						3/1	16/6
40		B		V	V		ND	V									6/5	12/8
41	×	B		V	V												2/2	8/4

*: Future residue definition; **MACP-Reg.**: REGULATION (EU) 2019/533 of 28 March 2019; **NCP-WD**: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 26–27 November 2018rev. 10(3)

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 one laboratory having started submission but was not able to complete it due to COVID-19 lockdown)

			Compulsory Compounds														Optional Compounds		
Compounds listed on the Target List			2,4-D (free acid)	Carbofuran (sum)	Chlormequat-Cl	Ethephon	Fluazifop (free acid)	Glufosinate	Glyphosate	Haloxyfop (free acid)	Mepiquat-Chloride	MPP (= MPPA)	N-Acetyl glufosinate	TFNA	TFNG	analysed / correctly found (Compulsory Compounds)	2,4-D (sum)	AMPA	Bentazone
within MACP			MACP	MACP	MACP + WD	MACP	MACP	MACP	MACP + WD	MACP	MACP + WD	MACP	MACP	MACP	MACP		MACP	WD*	
present in Test Item			Yes	Yes	Yes	No	Trace	No	Yes	Trace	No	No	No	Yes	No		Yes	No	Yes
evaluated in this PT			Yes	Yes	Yes	No	No	No	Yes	No	No	No	No	Yes	No		Yes	No	Yes
Lab-Code SRM15-	NRL	Cat.																	
42	×	B			V						ND					2/1			
44		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
45	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5		ND	V
46	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
47		B		V	V	ND		ND	V		ND	ND	ND	V	ND	10/4	V	ND	V
48		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
49		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
50		B	V	V	V		ND	ND	V	ND	ND			V	ND	10/5	V	ND	V
51		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	**FN***	ND	13/4	V	ND	V
54		B						ND	V			ND				3/1		ND	
55		B	V	V	V	ND	ND	ND	V	ND	ND			V	ND	11/5	V		V
56		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
57		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
58		B	V	V		ND	ND	ND	V	ND		ND	ND			9/3	V	ND	
59		B	V	V	V		ND		V	ND	ND					7/4	V		V
60		B	V	V			ND			ND						4/2			V
61	×	B	V	V	V		ND		V	ND	ND					7/4		ND	
62	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
63		B				ND		ND	V			ND	ND			5/1		ND	
64		B	V	V	V	ND	ND	ND	V	ND	ND			V	ND	11/5	V	ND	V
65	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	**FN**	ND	13/4		ND	V
66		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
67		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
68	×	B	V		V		ND		V	ND	ND			V	ND	8/4	V	ND	
69		B	V	V				ND	V							4/3		ND	V
70		B		V												1/1			
71	×	B	V	V	V	ND	ND			ND	ND			V	ND	9/4	V		V
74	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
75		B	V	V	V	ND	ND		V	ND	ND			V	ND	10/5		ND	
77	×	B	V	V	V		ND			ND	ND			V	ND	8/4			

*: Future residue definition; **MACP-Reg.**: REGULATION (EU) 2019/533 of 28 March 2019; **NCP-WD**: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 26–27 November 2018rev. 10(3)

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 one laboratory having started submission but was not able to complete it due to COVID-19 lockdown)

			Optional Compounds														Total
Compounds listed on the Target List			Diquat, expr. as dication	Fluazifop (sum)	Haloxyfop (sum)	Imazethapyr (free acid)	MCPA (free acid)	MCPA (sum)	MCPB (free acid)	MCPB (sum)	Mecoprop (free acid)	Mecoprop (sum)	N-Acetyl glyphosate	Paraquat	Quizalofop (free acid)	Quizalofop (sum)	analysed / correctly found (Optional Compounds)	analysed / correctly found (Total)
within MACP			WD	MACP	MACP		WD	WD	WD	WD			WD*	WD	WD	WD*		
present in Test Item			No	**Yes**	**Yes**	**Yes**	Trace	**Yes**	Trace	**Yes**	Trace	**Yes**	No	**Yes**	**Yes**	**Yes**		
evaluated in this PT			No	**Yes**	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**		
Lab-Code SRM15-	NRL	Cat.																
42	×	B	ND											V			2 / 1	4 / 2
44		A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17 / 11	30 / 16
45	×	A	ND				ND		ND								5 / 1	18 / 6
46	×	A		V	V		ND	V	ND	V	ND	**FN**			V	V	13 / 8	26 / 13
47		B	ND	V	V	V		V		V		**FN**		V		V	12 / 9	22 / 13
48		A		V	V	V	ND	V	ND	V	ND	V	ND		V	V	15 / 10	28 / 15
49		A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17 / 11	30 / 16
50		B	ND	V	V	V	ND	V	ND	V	ND	V		V	V	V	16 / 11	26 / 16
51		A	ND	V	V	V	ND	V	ND	V	ND	**FN**	ND	**FN**	V	**FN**	17 / 8	30 / 12
54		B															1 / 0	4 / 1
55		B		V	V	V	ND	V	ND	V	ND	V			V	V	13 / 10	24 / 15
56		A	ND	V	V	V	ND	V	ND	V	ND	V	ND		V	V	16 / 10	29 / 15
57		A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17 / 11	30 / 16
58		B		V	V		ND	V	ND	V			ND		V	V	11 / 7	20 / 10
59		B		V	V		ND	V	ND	V	ND	V			V	V	12 / 9	19 / 13
60		B				V	ND				ND						4 / 2	8 / 4
61	×	B					ND				ND						3 / 0	10 / 4
62	×	A		V	V		ND	V	ND	V	ND	V					11 / 7	24 / 12
63		B															1 / 0	6 / 1
64		B	ND	V	V	V		V		V		**FN**		V	V		12 / 9	23 / 14
65	×	A	ND			V	ND		ND		ND		ND	V	V		10 / 4	23 / 8
66		A	ND				ND	V	ND			**FN**		V	V	V	11 / 6	24 / 11
67		A	ND	V	V		ND	V			ND	V		V	V	V	13 / 9	26 / 14
68	×	B		V	V		ND	V	ND	V	ND	**FN**					10 / 5	18 / 9
69		B					ND										3 / 1	7 / 4
70		B															0 / 0	1 / 1
71	×	B			V	V	ND	V	ND	V	ND				V	V	11 / 8	20 / 12
74	×	A	ND	**FN**	V	V	ND	V	ND	V	ND	**FN**	ND	V	V	V	17 / 9	30 / 14
75		B															1 / 0	11 / 5
77	×	B															0 / 0	8 / 4

*: Future residue definition; **MACP-Reg.**: REGULATION (EU) 2019/533 of 28 March 2019; **NCP-WD**: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 26–27 November 2018rev. 10(3)

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 one laboratory having started submission but was not able to complete it due to COVID-19 lockdown)

			Compulsory Compounds														Optional Compounds		
Compounds listed on the Target List			2,4-D (free acid)	Carbofuran (sum)	Chlormequat-Cl	Ethephon	Fluazifop (free acid)	Glufosinate	Glyphosate	Haloxyfop (free acid)	Mepiquat-Chloride	MPP (= MPPA)	N-Acetyl glufosinate	TFNA	TFNG	analysed / correctly found (Compulsory Compounds)	2,4-D (sum)	AMPA	Bentazone
within MACP			MACP	MACP	MACP + WD	MACP	MACP	MACP	MACP + WD	MACP	MACP + WD	MACP	MACP	MACP	MACP		MACP	WD*	
present in Test Item			Yes	Yes	Yes	No	Trace	No	Yes	Trace	No	No	No	Yes	No		Yes	No	Yes
evaluated in this PT			Yes	Yes	Yes	No	No	No	Yes	No	No	No	No	Yes	No		Yes	No	Yes
Lab-Code SRM15-	NRL	Cat.																	
79		B		V	V				V							3/3			
80		B			V		ND			ND	ND					4/1			
82		B		V	V		ND			ND						4/2			V
84		B	V				ND			ND						3/1	V		
85		B														0/0			
87	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
88	×	B	V	V	V	ND	ND	ND	V	ND	ND					9/4		ND	V
90		B	V	V	V	ND	ND	ND	V	ND	ND			V	ND	11/5	V	ND	V
91	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V		V
92		B	V		V	ND	ND	ND	V	ND	ND			V	ND	10/4	V	ND	V
93		B	V	V			ND			ND				V	ND	6/3	V		V
94		B	V	V	V	ND	ND	ND	V	ND	ND			**FN**	ND	11/4			V
95	×	B	V	V	V		ND		V	ND	ND			V	ND	9/5			V
96		B	V	V	V	ND	ND	ND	V	ND	ND	ND				10/4	V	ND	V
97		B	V	V			ND			ND				V	ND	6/3			
98		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
99		B	**FN**	V	V	ND	ND	ND	V	ND	ND			V	ND	11/4	V	ND	V
100		B		V	V	ND		ND	V	ND	ND	ND	ND	V	ND	11/4	V	ND	V
101		B			V	ND	ND		V	ND	ND					6/2			V
102		B	V	V	V	ND	ND		V	ND	ND					8/4			
103	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND		V	ND	12/5	V		V
104		B		V												1/1			
105	×	B	V	V	V		ND			ND	ND			V	ND	8/4			
107		B	V	V	V	ND	ND	ND	V	ND	ND	ND	ND			11/4		ND	
108	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	
109		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5		ND	
110		B		V												1/1			
111		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V
112	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V		12/5		ND	
113		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13/5	V	ND	V

*: Future residue definition; **MACP-Reg.**: REGULATION (EU) 2019/533 of 28 March 2019; **NCP-WD**: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 26–27 November 2018rev. 10(3)
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 one laboratory having started submission but was not able to complete it due to COVID-19 lockdown)

			Optional Compounds															Total
Compounds listed on the Target List			Diquat, expr. as dication	Fluazifop (sum)	Haloxyfop (sum)	Imazethapyr (free acid)	MCPA (free acid)	MCPA (sum)	MCPB (free acid)	MCPB (sum)	Mecoprop (free acid)	Mecoprop (sum)	N-Acetyl glyphosate	Paraquat	Quizalofop (free acid)	Quizalofop (sum)	analysed / correctly found (Optional Compounds)	analysed / correctly found (Total)
within MACP			WD	MACP	MACP		WD	WD	WD	WD			WD*	WD	WD	WD*		
present in Test Item			No	**Yes**	**Yes**	**Yes**	Trace	**Yes**	Trace	**Yes**	Trace	**Yes**	No	**Yes**	**Yes**	**Yes**		
evaluated in this PT			No	**Yes**	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**		
Lab-Code SRM15-	NRL	Cat.																
79		B															0 / 0	3 / 3
80		B															0 / 0	4 / 1
82		B		V	V												3 / 3	7 / 5
84		B		V	V		ND	V	ND	V	ND	V			V	V	11 / 8	14 / 9
85		B												V			1 / 1	1 / 1
87	×	A		V	V	V	ND	V	ND	V	ND	V	ND		V	V	15 / 10	28 / 15
88	×	B															2 / 1	11 / 5
90		B	ND	V	V	V	ND	V	ND	V	ND	V		V	V	V	16 / 11	27 / 16
91	×	A		**FN**	V		ND	V	ND	**FN**	ND	**FN**	ND		V	V	13 / 6	26 / 11
92		B		V	V	V	ND	V	ND	V	ND	V			V	V	14 / 10	24 / 14
93		B		V	V	V	ND	V	ND	V	ND	V			V	V	13 / 10	19 / 13
94		B		V			ND		ND		ND						5 / 2	16 / 6
95	×	B	ND				ND		ND		ND				V		6 / 2	15 / 7
96		B		V	V	V	ND	V	ND	V	ND	V			V	V	14 / 10	24 / 14
97		B					ND		ND						V		3 / 1	9 / 4
98		A	ND	V	V	V	ND	V	ND	V	ND	V		V	V	V	16 / 11	29 / 16
99		B	ND	V	V	V	ND	V	ND	V				**FN**	**FN**		13 / 7	24 / 11
100		B		V	V	**FN**		V		V		V				V	10 / 8	21 / 12
101		B													V		2 / 2	8 / 4
102		B															0 / 0	8 / 4
103	×	A		V	V	V	ND	V	ND	V	ND	**FN**			V	V	13 / 9	25 / 14
104		B															0 / 0	1 / 1
105	×	B															0 / 0	8 / 4
107		B					ND						ND				3 / 0	14 / 4
108	×	A		V	V		ND	V	ND	V	ND	V	ND				11 / 6	24 / 11
109		A					ND		ND		ND						4 / 0	17 / 5
110		B		V													1 / 1	2 / 2
111		A		V	V	V	ND	V	ND	V	ND	V			V	V	14 / 10	27 / 15
112	×	A					ND						ND				3 / 0	15 / 5
113		A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17 / 11	30 / 16

*: Future residue definition; **MACP-Reg.**: REGULATION (EU) 2019/533 of 28 March 2019; **NCP-WD**: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 26–27 November 2018rev. 10(3)

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 one laboratory having started submission but was not able to complete it due to COVID-19 lockdown)

			Compulsory Compounds													Optional Compounds		
Compounds listed on the Target List			2,4-D (free acid)	Carbofuran (sum)	Chlormequat-Cl	Ethephon	Fluazifop (free acid)	Glufosinate	Glyphosate	Haloxyfop (free acid)	Mepiquat-Chloride	MPP (= MPPA)	N-Acetyl glufosinate	TFNA	TFNG	analysed / correctly found (Compulsory Compounds)	2,4-D (sum)	AMPA	Bentazone
within MACP			MACP	MACP	MACP + WD	MACP	MACP	MACP	MACP + WD	MACP	MACP + WD	MACP	MACP	MACP	MACP		MACP	WD*	
present in Test Item			Yes	Yes	Yes	No	Trace	No	Yes	Trace	No	No	No	Yes	No		Yes	No	Yes
evaluated in this PT			Yes	Yes	Yes	No	No	No	Yes	No	No	No	No	Yes	No		Yes	No	Yes
Lab-Code SRM15-	NRL	Cat.																	
114		B	V	V	V	ND	ND	ND	V	ND	ND			V	ND	11 / 5	V	ND	V
115		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
116	×	B	V	V			ND			ND				V	ND	6 / 3	V		V
118	×	A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
119	×	B	V	V	V	ND	ND	ND	V	ND	ND	ND		V	**FP**	12 / 5	V	ND	V
120		B		V												1 / 1			
121		B	V	V		ND	ND	ND	V	ND				V	ND	9 / 4		ND	V
122		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
123		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
124		B	V		V		ND	ND	V	ND	ND	ND	ND			9 / 3	V	ND	V
125		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
127		B	V		V	ND		ND			ND			V	ND	7 / 3		ND	
128		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
129		A	V	V	V	ND	ND	ND	V	ND	ND	ND	ND	V	ND	13 / 5	V	ND	V
130		B			V				V		ND					3 / 2		ND	
132		B	V		V	ND				ND	ND					5 / 2	V		V
133		B	V	V	V		ND				ND					5 / 3			V
137		B	V	V	V	ND	ND		V	ND	ND		ND	V	ND	11 / 5	V	ND	V
3rd-29		B	V				ND			ND						3 / 1	V		V
3rd-43		B	V		V			ND	V		ND					5 / 3		ND	V
3rd-72		B	V				ND							V	ND	4 / 2			
3rd-73		B	V	V	V	ND	ND	ND	V	ND	ND			V	ND	11 / 5	V	ND	V
3rd-83		B	V		V	ND	ND	ND		ND	ND					7 / 2		ND	V
3rd-86		B	V						V							2 / 2		ND	V
3rd-134		B	V				ND			ND						3 / 1	V		
3rd-135		B		V		ND	ND	ND		ND						5 / 1		ND	
Info-10		B	V	V	V	ND			V	ND	ND			V	ND	9 / 5	V		V

*: Future residue definition; **MACP-Reg.**: REGULATION (EU) 2019/533 of 28 March 2019; **NCP-WD**: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 26–27 November 2018rev. 10(3)
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 one laboratory having started submission but was not able to complete it due to COVID-19 lockdown)

			Optional Compounds															Total
Compounds listed on the Target List			Diquat, expr. as dication	Fluazifop (sum)	Haloxyfop (sum)	Imazethapyr (free acid)	MCPA (free acid)	MCPA (sum)	MCPB (free acid)	MCPB (sum)	Mecoprop (free acid)	Mecoprop (sum)	N-Acetyl glyphosate	Paraquat	Quizalofop (free acid)	Quizalofop (sum)	analysed / correctly found (Optional Compounds)	analysed / correctly found (Total)
within MACP			WD	MACP	MACP		WD	WD	WD	WD			WD*	WD	WD	WD*		
present in Test Item			No	**Yes**	**Yes**	**Yes**	Trace	**Yes**	Trace	**Yes**	Trace	**Yes**	No	**Yes**	**Yes**	**Yes**		
evaluated in this PT			No	**Yes**	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**		
Lab-Code SRM15-	NRL	Cat.																
114		B	ND	V	V	V	ND	V	ND	V	ND	V		V	V	V	16 / 11	27 / 16
115		A	ND	V	V	**FN**	ND	V	ND	V	ND			V	V	V	15 / 9	28 / 14
116	×	B		V	V		ND	V	ND	V	ND	V			**FN***	V	12 / 8	18 / 11
118	×	A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17 / 11	30 / 16
119	×	B		V	V		ND	V	ND	V	ND	**FN**			V	V	13 / 8	25 / 13
120		B															0 / 0	1 / 1
121		B					ND								**FN**		4 / 1	13 / 5
122		A		V	V	V	ND	V	ND	V	ND	**FN**	ND		V	V	15 / 9	28 / 14
123		A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17 / 11	30 / 16
124		B		V	V		ND	V	ND	V	ND	V			V	V	13 / 9	22 / 12
125		A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17 / 11	30 / 16
127		B															1 / 0	8 / 3
128		A	ND	V	V	V	ND	V	ND	V	ND	V	ND	V	V	V	17 / 11	30 / 16
129		A		V	V		ND	V	ND	V	ND	V			V	V	13 / 9	26 / 14
130		B															1 / 0	4 / 2
132		B	ND				ND	V	ND	V	ND	V					9 / 5	14 / 7
133		B			V			V									3 / 3	8 / 6
137		B	ND	V	V		ND	V	ND	**FN***	ND	**FN**	ND	V	V		15 / 7	26 / 12
3rd-29		B		V	V	V	ND	V	ND	V	ND	V			V	V	13 / 10	16 / 11
3rd-43		B	ND			V					ND			V			6 / 3	11 / 6
3rd-72		B				V			ND		ND				V		4 / 2	8 / 4
3rd-73		B	ND	V	V	V	ND	V	ND	V	ND	V		**FN**			14 / 8	25 / 13
3rd-83		B	ND			V	ND		ND		ND						7 / 2	14 / 4
3rd-86		B	ND			V	ND							V			6 / 3	8 / 5
3rd-134		B		**FN**	**FN**		ND		ND								5 / 1	8 / 2
3rd-135		B				**FN**											2 / 0	7 / 1
Info-10		B	ND	V	V		ND	V			ND	**FN**	ND	**FN**			11 / 5	20 / 10

*: Future residue definition; **MACP-Reg.**: REGULATION (EU) 2019/533 of 28 March 2019; **NCP-WD**: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 26–27 November 2018rev. 10(3)

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 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, excluded outliers and calculated using Algorithm A [6, **Appendix 8**]. Results from laboratories outside EU and EFTA countries (i.e. 3rd countries and EU Candidate Countries) should not be taken into account. Unfortunately, results submitted by two laboratories outside EU and EFTA countries were by mistake included in the establishment of the assigned values. Based on these assigned values, z-scores were calculated for all submitted results using the FFP-approach (**Section 4.4.3, p. 28)**, and a preliminary report was released on 5 June 2020.

[Corrigendum]
Immediately after the preliminary report was released, the organizer noticed that the results submitted by two laboratories outside EU and EFTA countries were by mistake included in the establishment of the assigned values. As this error has only minor influence on the concerned assigned values, no revised version of the preliminary report was released. Hence, the assigned values and z-scores in the present final report can differ slightly from those in the preliminary report.

Before setting the assigned values, the results within a population of each analyte were checked for outlier based on the z-scores calculated using robust mean from the entire population. Results obtained z-scores >5 are regarded as outliers and excluded from the population for establishment of assigned values. No further criteria were applied to investigation of outliers. Following exclusion of outliers the robust mean of each analyte was calculated again using the remaining results and established as the assigned value.

The uncertainties ($u(x_{pt})$) of the assigned values were calculated as described under **Section 2.2, p. 29**.

In the case of ***fluazifop (sum)***, ***MCPB (sum)*** and ***mecoprop (sum)*** the distribution of the participants' results was quite broad and visibly not unimodal. Looking at methodological patterns and considering the results obtained for the PT sample by the organizer (EURL-SRM) using different hydrolysis conditions, the participants' results were divided into subpopulations according to their hydrolysis conditions. For the various subpopulations, robust statistics calculations were applied to derive the robust mean and the relative standard deviation (CV*). As expected, the robust mean of subpopulations applying stronger hydrolysis conditions, thus ensuring nearly quantitative conversion rates, were overall closer to the expected (e.g. spiked) levels compared to subpopulations using weak or no hydrolysis. It was therefore decided using the robust means of results generated by methods involving moderate or strong hydrolysis conditions for ***fluazifop (sum)***, ***MCPB (sum)*** and results generated by methods involving strong hydrolysis conditions only for ***mecoprop (sum)*** as the assigned values in the preliminary report. After consultation with the Scientific Committee, these criteria were also applied to establish the final assigned values for these three compounds.

Due to the similar situation the assigned value of ***carbofuran (sum)*** in the preliminary report was calculated using results generated by methods involving acidic hydrolysis only, although the organizer was well aware of the fact that results from laboratories having analyzed carbofuran and carbosulfan separately could also have been added to this group. But as carbosulfan analysis is quite challenging and error-prone, results driven from this analytical approach were firstly not included. After releasing the Preliminary Report, the organizer started a survey on detailed information about the analytical method for ***carbofuran (sum).*** Based on the survey results and after consultation with the Scientific Committee, it was finally decided to use the robust mean of ***carbofuran (sum)*** results generated by hydrolysis or analysis of carbofuran and carbosulfan separately as the final assigned value for ***carbofuran (sum).***

For details please see also method-based evaluation **Section 4.4 „Special Topic: Method-based Evaluation" (p. 48) .**

Table 4-3: 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 / Outlier	No. of numerical results (EU+EFTA)	Result Population for AV | n=	Assigned Value [mg/kg]	$u(x_{pt})$ [1] [mg/kg]	$u(x_{pt})$ Tolerance [mg/kg]	Judgement for UAV-test	CV^* [2] [%]
Compulsory Compounds	2,4-D (free acid)	4 / 0	85	entire | 85	0.052	+/- 0.0015	0.0039	passed	20.8
	Carbofuran (sum)	0 / 2	92	part* | 63	0.107	+/- 0.0032	0.0080	passed	22.8
	Chlormequat-Cl	0 / 3	88	entire | 88	0.092	+/- 0.0021	0.0069	passed	16.8
	Glyphosate	0 / 0	84	entire | 84	0.203	+/- 0.0066	0.0152	passed	23.7
	TFNA	3 / 1	69	entire | 69	0.060	+/- 0.0025	0.0045	passed	27.8
	Average [3] CV^*								**22.4**
Optional Compounds	2,4-D (sum)	0 / 3	66	entire | 66	0.059	+/- 0.0017	0.0044	passed	18.9
	Bentazone	0 / 1	77	entire | 77	0.334	+/- 0.0092	0.0251	passed	19.4
	Fluazifop (sum)	2 / 0	64	part# | 42	0.060	+/- 0.0022	0.0045	passed	18.8
	Haloxyfop (sum)	1 / 0	64	entire | 64	0.151	+/- 0.0038	0.0113	passed	16.2
	Imazethapyr (free acid)	2 / 0	38	entire | 38	0.206	+/- 0.0073	0.0155	passed	17.6
	MCPA (sum)	1 / 2	65	entire | 65	0.068	+/- 0.0021	0.0051	passed	20.2
	MCPB (sum)	4 / 1	56	part# | 40	0.057	+/- 0.0034	0.0043	passed	29.4
	Mecoprop (sum)	18 / 1	41	part‡ | 20	0.067	+/- 0.0043	0.0050	passed	22.4
	Paraquat	2 / 1	32	entire | 32	0.195	+/- 0.0125	0.0146	passed	28.9
	Quizalofop (free acid)	4 / 0	60	entire | 60	0.044	+/- 0.0012	0.0033	passed	16.4
	Quizalofop (sum)	2 / 0	52	entire | 52	0.062	+/- 0.0025	0.0046	passed	22.9
	Average [3] CV^*								**21.0**

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 meaningful and conclusive.
*: Sub population for carbofuran (sum) consisted of results generated by methods involving acidic hydrolysis or analysis of carbofuran and carbosulfan separately.
#: Sub population for fluazifop (sum) and MCPA (sum) consisted of results generated by methods involving moderate or strong hydrolysis conditions.
‡: Sub population for mecoprop (sum) consisted of results generated by methods involving strong hydrolysis conditions only.

The assigned values and their uncertainties are shown in **Table 4-3**. Except ***MCPB (sum)*** (29.4 %), ***paraquat*** (28.9 %) and ***TFNA*** (27.8 %) the CV^*-values of all other analytes were lower than the FFP-RSD (25 %). The average CV^*s of all compulsory analytes based on the results population of EU-and EFTA-laboratories was 22.4 %. In particular ***chlormequat-Cl*** with CV^* of 16.8 % was significantly lower than the FFP-RSD. The average CV^*s of all optional analytes based on the results population of EU-and EFTA-laboratories was 21.0 % with CV^* of 16.2 % for ***haloxyfop (sum)*** and 16.4 % for ***quizalofop (free acid)*** as the lowest values.

4.3 Assessment of Laboratory Performance

4.3.1 False Positives

In this PT, only one numerical result concerning *TFNG* was submitted by one EU laboratory for target compounds being not spiked to the test material and not detected by the organizer or by the overwhelming majority of the participants. The reported value was slightly above the MRRL and lab's reporting limit and was therefore judged as a false positive result (**Table 4-4**).

Table 4-4: The only one false positive result reported in EURL-SRM15

	Compound	PT-Code	Analysed	Reported Result [mg/kg]	RL [mg/kg]	MRRL [mg/kg]	Judgement
Compulsory	TFNG	SRM15-119	Yes	0.011	0.01	0.01	FP

4.3.2 False Negatives

In the case of compulsory compounds 7 laboratories reported in 7 cases (4× ***2,4-D (free acid)*** and 3× ***TFNA***) "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 4-5, p. 33**). Among them, one laboratory's reporting limit for ***TFNA*** (0.1 mg/kg) was higher then the assigned value (0.06 mg/kg). According to the rules in the General Protocol this result was still judged as false negative. This laboratory is encouraged to improve its RL for ***TFNA***. The 7 false negative results accounted for 1.6 % of the total 425 results reported by the EU/EFTA laboratories for the compulsory target compounds.

In the case of optional compounds 36 cases (18× ***mecoprop (sum),*** each 4 cases for ***quizalofop (free acid)*** and ***MCPB (sum),*** each 2 cases for ***paraquat, quizalofop (sum), imazethapyr (free acid)*** and ***fluazifop (sum),*** each one case for ***MCPA (sum)*** and ***haloxyfop (sum)***) reported by 24 laboratories were judged as false negative results (**Table 4-5, p. 33**), since those target compounds which the participating laboratories analysed for but not detected were spiked to the test item and detected by the majority of the laboratories targeting them. Among them, one laboratory's reporting limit for ***MCPB (sum)*** (0.05 mg/kg) was equal to the assigned value (0.05 mg/kg) and another one laboratory had a higher reporting limit for ***quizalofop (free acid)*** (0.05 mg/kg) then the assigned value (0.044 mg/kg). In accordance with the General Protocole these two results were judged as false negative, and the laboratories are encouraged to improve their RLs for the corresponding analytes. The 36 false negative results accounted for 5.5 % of the total 651 results reported by the EU/EFTA laboratories for the optional target compounds. In the case of ***mecoprop (sum)*** the main reason for the 18 false negative result resulted from too weak hydrolysis (14 cases) accompanied by lacking experiences (4 cases) or the analytical procedure was not properly performed.

4.3.3 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 and in the case of ***carbofuran (sum), fluazifop (sum), MCPB (sum)*** and ***mecoprop (sum)*** from the subpopulation of results reported by the EU/EFTA laboratories excluding outliers as described in **Section 4.2 (p. 30)**. **Table 4-6 (p. 34)** shows the overall classification of z-scores achieved by all laboratories for compulsory and optional compounds based on the rules given in **Section 2.4 (p. 31)**.

In average, 89 % of the reported results by EU/EFTA laboratories for the compulsory compounds achieved the "acceptable" z-scores. It ranged from 86 % for ***carbofuran (sum)*** to 94 % for ***chlormequate-Cl***. Considering only the optional compounds and regardless of ***MCPB (sum)*** and ***mecoprop (sum)***, "acceptable" z-scores were achieved by EU/EFTA-laboratories from 82 % for ***fluazifop (sum)*** and ***paraquat*** till 95 % for ***bentazone*** and in average 88 % of the reported results for the optional compounds were able to be classified as acceptable. Considering the results reported by the participating laboratories from EU Candidate or 3rd countries 95 % and 83 % of them could be classified into "acceptable" for the compulsory and optional compounds, respectively.

Table 4-5: Overview of false negative results reported by participating laboratories (including 4 results from 3rd country laboratories)

	Compounds	PT-Code	Analysed	Detected	RL [mg/kg]	MRRL [mg/kg]	Assigned Value [mg/kg]	Judgement
Compulsory	2,4-D (free acid)	SRM15-5	Yes	No	0.025	0.01	0.052	False Negative
		SRM15-33	Yes	No	0.01	0.01	0.052	False Negative
		SRM15-41	Yes	No	0.05	0.01	0.052	False Negative
		SRM15-99	Yes	No	0.01	0.01	0.052	False Negative
	TFNA	SRM15-51	Yes	No	0.1	0.01	0.060	False Negative*
		SRM15-65	Yes	No	0.01	0.01	0.060	False Negative
		SRM15-94	Yes	No	0.01	0.01	0.060	False Negative
Optional	Fluazifop (sum)	SRM15-74	Yes	No	0.01	0.01	0.057	False Negative
		SRM15-91	Yes	No	0.01	0.01	0.057	False Negative
		SRM15-134[3rd]	Yes	No	0.01	0.01	0.057	False Negative
	Haloxyfop (sum)	SRM15-19	Yes	No	0.01	0.01	0.152	False Negative
		SRM15-134[3rd]	Yes	No	0.01	0.01	0.152	False Negative
	Imazethapyr (free acid)	SRM15-100	Yes	No	0.01	0.01	0.207	False Negative
		SRM15-115	Yes	No	0.01	0.01	0.207	False Negative
		SRM15-135[3rd]	Yes	No	0.01	0.01	0.207	False Negative
	MCPA (sum)	SRM15-34	Yes	No	0.01	0.01	0.068	False Negative
	MCPB (sum)	SRM15-30	Yes	No	0.01	0.01	0.050	False Negative
		SRM15-34	Yes	No	0.01	0.01	0.050	False Negative
		SRM15-91	Yes	No	0.01	0.01	0.050	False Negative
		SRM15-137	Yes	No	0.05	0.01	0.050	False Negative*
	Mecoprop (sum)	SRM15-8	Yes	No	0.05	0.01	0.051	False Negative
		SRM15-21	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-24	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-30	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-34	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-37	Yes	No	0.02	0.01	0.051	False Negative
		SRM15-46	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-47	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-51	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-64	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-66	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-68	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-74	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-91	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-103	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-119	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-122	Yes	No	0.01	0.01	0.051	False Negative
		SRM15-137	Yes	No	0.01	0.01	0.051	False Negative
	Paraquat	SRM15-51	Yes	No	0.01	0.02	0.193	False Negative
		SRM15-73[3rd]	Yes	No	0.01	0.02	0.193	False Negative
		SRM15-99	Yes	No	0.02	0.02	0.193	False Negative
	Quizalofop (free acid)	SRM15-34	Yes	No	0.01	0.01	0.044	False Negative
		SRM15-99	Yes	No	0.01	0.01	0.044	False Negative
		SRM15-116	Yes	No	0.05	0.01	0.044	False Negative*
		SRM15-121	Yes	No	0.01	0.01	0.044	False Negative
	Quizalofop (sum)	SRM15-8	Yes	No	0.01	0.01	0.062	False Negative
		SRM15-51	Yes	No	0.01	0.01	0.062	False Negative

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

Table 4-6: Overall performance based on z-score classification

	EU and EFTA laboratories					
	Compound	No. of results [1)]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1)] No. (%)	FNs No.
Compulsory Compounds	2,4-D (free acid)	89	77 (87 %)	5 (6 %)	7 (8 %)	4
	Carbofuran (sum)	92	79 (86 %)	9 (10 %)	4 (4 %)	0
	Chlormequat-Cl	88	83 (94 %)	0 (0 %)	5 (6 %)	0
	Glyphosate	84	77 (92 %)	3 (4 %)	4 (5 %)	0
	TFNA	72	64 (89 %)	3 (4 %)	5 (7 %)	3
	Subtotal	**425**	**380 (89 %)**	**20 (5 %)**	**25 (6 %)**	**7**
Optional Compounds	2,4-D (sum)	66	56 (85 %)	5 (8 %)	5 (8 %)	0
	Bentazone	77	73 (95 %)	1 (1 %)	3 (4 %)	0
	Fluazifop (sum)	66	54 (82 %)	6 (9 %)	6 (9 %)	2
	Haloxyfop (sum)	65	58 (89 %)	5 (8 %)	2 (3 %)	1
	Imazethapyr (free acid)	40	34 (85 %)	3 (8 %)	3 (8 %)	2
	MCPA (sum)	66	61 (92 %)	1 (2 %)	4 (6 %)	1
	MCPB (sum)	60	43 (72 %)	10 (17 %)	7 (12 %)	4
	Mecoprop (sum)	59	28 (47 %)	7 (12 %)	24 (41 %)	18
	Paraquat	34	28 (82 %)	0 (0 %)	6 (18 %)	2
	Quizalofop (free acid)	64	57 (89 %)	1 (2 %)	6 (9 %)	4
	Quizalofop (sum)	54	48 (89 %)	2 (4 %)	4 (7 %)	2
	Subtotal excl. MCPB (sum) and Mecoprop (sum)	**532**	**469 (88 %)**	**24 (5 %)**	**25 (5 %)**	**14**
	Subtotal incl. MCPB (sum) and Mecoprop (sum)	**651**	**540 (83 %)**	**41 (6 %)**	**70 (11 %)**	**36**
Overall EU/EFTA (Average)		**1076**	**920 (86 %)**	**61 (6 %)**	**95 (9 %)**	**43**
	3rd country laboratories					
	Compound	No. of results [1)]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1)] No. (%)	FNs No.
Compulsory Compounds	2,4-D (free acid)	7	6 (86 %)		1 (14 %)	0
	Carbofuran (sum)	2	2 (100 %)		0 (0 %)	0
	Chlormequat-Cl	3	3 (100 %)		0 (0 %)	0
	Glyphosate	3	3 (100 %)		0 (0 %)	0
	TFNA	2	2 (100 %)		0 (0 %)	0
	Subtotal	**17**	**16 (94 %)**		**1 (6 %)**	**0**
Optional Compounds	2,4-D (sum)	3	2 (67 %)		1 (33 %)	0
	Bentazone	5	5 (100 %)		0 (0 %)	0
	Fluazifop (sum)	3	2 (67 %)		1 (0 %)	1
	Haloxyfop (sum)	3	2 (67 %)		1 (0 %)	1
	Imazethapyr (free acid)	7	6 (86 %)		1 (0 %)	1
	MCPA (sum)	2	2 (100 %)		0 (0 %)	0
	MCPB (sum)	2	2 (100 %)		0 (0 %)	0
	Mecoprop (sum)	2	2 (100 %)		0 (0 %)	0
	Paraquat	3	2 (67 %)		1 (0 %)	1
	Quizalofop (free acid)	2	2 (100 %)		0 (0 %)	0
	Quizalofop (sum)	1	1 (100 %)		0 (0 %)	0
	Subtotal excl. MCPB (sum) and Mecoprop (sum)	**29**	**24 (83 %)**		**1 (3 %)**	**4**
	Subtotal incl. MCPB (sum) and Mecoprop (sum)	**33**	**28 (85 %)**		**5 (3 %)**	**4**
Overall 3rd country (Average)		**50**	**44 (88 %)**		**6 (4 %)**	**4**

1) including false negatives (FNs)

Table 4-7: Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compounds

COMPULSORY Compounds				2,4-D (free acid)		Carbofuran (sum)			Chlormequat-Cl		Glyphosate		TFNA	
MRRL [mg/kg]				0.01		0.01		*0.01*	0.01		0.03		0.01	
Assigned Value [mg/kg]				0.052		0.107*		*0.106#*	0.092		0.203		0.060	
*CV**				20.8 %		22.8 %*		*22.1 %#*	16.8 %		23.7 %		27.8 %	
Lab code SRM15-	NRL-SRM	Cat	Analysed / corr. found, max. 13 / 5	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	*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 %)
5		B	1 / 0	FN	-3.2									
8	x	B	10 / 4			0.142	1.3	*1.3*	0.0501	-1.8	0.0651	-2.7	0.101	2.7
9	x	B	11 / 4	0.096	3.4	0.106	0.0	*0.0*	0.119	1.2	0.181	-0.4		
11		B	7 / 4	0.050	-0.1	0.105	-0.1	*-0.1*	0.070	-1.0	0.183	-0.4		
12	x	A	13 / 5	0.047	-0.4	0.108	0.0	*0.1*	0.091	-0.1	0.159	-0.9	0.039	-1.4
13	x	A	12 / 5	0.0557	0.3	0.113	0.2	*0.3*	0.0675	-1.1	0.205	0.0	0.0381	-1.5
16	x	A	13 / 5	0.060	0.7	0.124	0.6	*0.7*	0.102	0.4	0.214	0.2	0.077	1.1
18		B	1 / 1			0.062	-1.7	*-1.7*						
19		A	13 / 5	0.038	-1.1	0.098	-0.3	*-0.3*	0.082	-0.5	0.26	1.1	0.046	-0.9
20	x	B	6 / 4	0.043	-0.7	0.069	-1.4	*-1.4*	0.096	0.2	0.136	-1.3		
21	x	A	13 / 5	0.0577	0.5	0.0738	-1.2	*-1.2*	0.0873	-0.2	0.286	1.6	0.0577	-0.2
22	x	B	10 / 5	0.053	0.1	0.116	0.3	*0.4*	0.096	0.2	0.189	-0.3	0.068	0.5
23	x	A	13 / 5	0.0503	-0.1	0.123	0.6	*0.6*	0.0788	-0.6	0.249	0.9	0.0610	0.1
24		A	13 / 5	0.034	-1.4	0.28	6.5	*6.8*	0.065	-1.2	0.15	-1.1	0.059	-0.1
25		B	6 / 3	0.052	0.0	0.116	0.3	*0.4*	0.092	0.0				
26		A	13 / 5	0.050	-0.1	0.119	0.5	*-0.3#*	0.098	0.2	0.20	-0.1	0.058	-0.2
27		A	13 / 5	0.050	-0.1	0.074	-1.2	*-1.2*	0.109	0.7	0.187	-0.3	0.066	0.4
28		A	13 / 5	0.0557	0.3	0.0841	-0.9	*-0.8*	0.0521	-1.8	0.239	0.7	0.0684	0.5
30		B	11 / 5	0.046	-0.4	0.099	-0.3	*0.0#*	0.095	0.1	0.207	0.1	0.061	0.1
31		A	12 / 4	0.0474	-0.3				0.103	0.5	0.227	0.5	0.0737	0.9
32		A	13 / 5	0.0602	0.7	0.131	0.9	*0.9*	0.111	0.8	0.177	-0.5	0.0734	0.9
33		B	6 / 3	FN	-3.2	0.0488	-2.2	*-2.2*	0.0982	0.3	0.233	0.6		
34		A	13 / 5	0.10	3.8	0.053	-2.0	*-2.0*	0.11	0.8	0.23	0.5	0.07	0.7
35		A	13 / 5	0.0427	-0.7	0.0820	-0.9	*-0.9*	0.0813	-0.5	0.129	-1.5	0.0420	-1.2
36		B	1 / 1								0.196	-0.1		
37		B	10 / 5	0.0374	-1.1	0.0645	-1.6	*-1.6*	0.0916	0.0	0.215	0.2	0.0648	0.3
38		B	3 / 2			0.046	-2.3	*-2.3*	0.097	0.2				
39		A	13 / 5	0.063	0.9	0.049	-2.2	*-2.2*	0.106	0.6	0.254	1.0	0.045	-1.0
40		B	6 / 3	0.0612	0.8	0.0894	-0.7	*-0.6*					0.0661	0.4
41	x	B	6 / 2	FN	-3.2	0.105	-0.1	*0.0*			0.112	-1.8		
42	x	B	2 / 1						0.128	1.5				
44		A	13 / 5	0.045	-0.5	0.098	-0.3	*-0.3*	0.063	-1.3	0.172	-0.6	0.064	0.3
45	x	A	13 / 5	0.0498	-0.1	0.103	-0.1	*-0.1*	0.0763	-0.7	0.242	0.8	0.0858	1.7
46	x	A	13 / 5	0.054	0.2	0.095	-0.4	*-0.4*	0.088	-0.2	0.193	-0.2	0.061	0.1
47		B	10 / 4			0.027	-3.0	*-3.0*	0.063	-1.3	0.195	-0.2	0.030	-2.0
48		A	13 / 5	0.028	-1.8	0.099	-0.3	*-0.3*	0.114	0.9	0.20	-0.1	0.046	-0.9
49		A	13 / 5	0.056	0.3	0.073	-1.3	*-1.2*	0.084	-0.4	0.225	0.4	0.086	1.7
50		B	10 / 5	0.050	-0.1	0.101	-0.2	*-0.3#*	0.083	-0.4	0.2303	0.5	0.21	10.0
51		A	13 / 4	0.039	-1.0	0.061	-1.7	*-1.4#*	0.086	-0.3	0.409	4.1	FN	-3.3
54		B	3 / 1								0.220	0.3		

* based on subpopulation consisting of results generated by methods involving acidic hydrolysis or analysis of carbofuran and carbosulfan separately
‡ due to COVID-19 lockdown lab was not able to complete its submission, evaluation for informative purpose only

based on corrected values, for details please see Section **4.4.2 (p. 51)**. Z-scores were calculated for informative purpose only.

Table 4-7 (cont.): Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compounds

COMPULSORY Compounds				2,4-D (free acid)		Carbofuran (sum)			Chlormequat-Cl		Glyphosate		TFNA	
MRRL [mg/kg]				0.01		0.01		*0.01*	0.01		0.03		0.01	
Assigned Value [mg/kg]				0.052		0.107*		*0.106#*	0.092		0.203		0.060	
*CV**				20.8 %		22.8 %*		*22.1 %#*	16.8 %		23.7 %		27.8 %	
Lab code SRM15-	NRL-SRM	Cat	Analysed / corr. found, max. 13 / 5	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	*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 %)
55		B	11 / 5	0.0400	-0.9	0.179	2.7	*2.7*	0.0846	-0.3	0.162	-0.8	0.0553	-0.3
56		A	13 / 5	0.042	-0.7	0.084	-0.9	*-0.8*	0.103	0.5	0.172	-0.6	0.061	0.1
57		A	13 / 5	0.052	0.0	0.132	0.9	*1.0*	0.102	0.4	0.210	0.1	0.059	-0.1
58		B	9 / 3	0.0535	0.2	0.0788	-1.1	*-1.0*			0.206	0.1		
59		B	7 / 4	0.077	2.0	0.108	0.0	*0.1*	0.077	-0.7	0.272	1.4		
60		B	4 / 2	0.0285	-1.8	0.0805	-1.0	*-1.0*						
61	x	B	7 / 4	0.059	0.6	0.103	-0.1	*-0.1*	0.229	5.9	0.248	0.9		
62	x	A	13 / 5	0.015	-2.8	0.0976	-0.3	*-0.3*	0.0875	-0.2	0.201	0.0	0.064	0.3
63		B	5 / 1								0.092	-2.2		
64		B	11 / 5	0.063	0.9	0.202	3.6	*3.6*	0.080	-0.5	0.258	1.1	0.046	-0.9
65	x	A	13 / 4	0.0350	-1.3	0.111	0.2	*0.2*	0.0917	0.0	0.274	1.4	FN	-3.3
66		A	13 / 5	0.088	2.8	0.051	-2.1	*-2.1*	0.096	0.2	0.103	-2.0	0.030	-2.0
67		A	13 / 5	0.043	-0.7	0.093	-0.5	*-0.5*	0.086	-0.3	0.022	-3.6	0.077	1.1
68	x	B	8 / 4	0.050	-0.1				0.091	-0.1	0.248	0.9	0.061	0.1
69		B	4 / 3	0.0526	0.1	0.074	-1.2	*-1.2*			0.021	-3.6		
70		B	1 / 1			0.091	-0.6	*-0.6*						
71	x	B	9 / 4	0.0520	0.0	0.104	-0.1	*-0.1*	0.128	1.5			0.0694	0.6
74	x	A	13 / 5	0.064	1.0	0.099	-0.3	*-0.3*	0.089	-0.2	0.199	-0.1	0.057	-0.2
75		B	10 / 5	0.012	-3.1	0.15	1.6	*1.7*	0.075	-0.8	0.21	0.1	0.015	-3.0
77	x	B	8 / 4	0.051	-0.1	0.11	0.1	*0.1*	0.076	-0.7			0.071	0.7
79		B	3 / 3			0.109	0.1	*0.1*	0.0861	-0.3	0.220	0.3		
80		B	4 / 1						0.07	-1.0				
82		B	4 / 2			0.0982	-0.3	*-0.3*	0.0963	0.2				
84		B	3 / 1	0.0495	-0.2									
85		B	0 / 0											
87	x	A	13 / 5	0.053	0.1	0.081	-1.0	*-0.9*	0.107	0.6	0.235	0.6	0.054	-0.4
88	x	B	9 / 4	0.078	2.1	0.050	-2.1	*-1.9#*	0.113	0.9	0.20	-0.1		
90		B	11 / 5	0.056	0.3	0.12	0.5	*0.5*	0.1	0.3	0.2	-0.1	0.08	1.3
91	x	A	13 / 5	0.0520	0.0	0.127	0.7	*0.8*	0.0832	-0.4	0.191	-0.2	0.0647	0.3
92		B	10 / 4	0.061	0.7				0.085	-0.3	0.220	0.3	0.053	-0.5
93		B	6 / 3	0.0513	0.0	0.0755	-1.2	*-1.2*					0.0678	0.5
94		B	11 / 4	0.020	-2.5	0.078	-1.1	*-1.1*	0.078	-0.6	0.246	0.8	FN	-3.3
95	x	B	9 / 5	0.0385	-1.0	0.071	-1.3	*-1.8#*	0.782	29.9	0.133	-1.4	0.0310	-1.9
96		B	10 / 4	0.0500	-0.1	0.107	0.0	*0.0*	0.122	1.3	0.122	-1.6		
97		B	6 / 3	0.0562	0.4	0.0520	-2.1	*-2.0*					0.0690	0.6
98		A	13 / 5	0.047	-0.4	0.087	-0.7	*-0.7*	0.099	0.3	0.22	0.3	0.041	-1.3
99		B	11 / 4	FN	-3.2	0.06	-1.8	*-1.9#*	0.09	-0.1	0.18	-0.5	0.06	0.0

* based on subpopulation consisting of results generated by methods involving acidic hydrolysis or analysis of carbofuran and carbosulfan separately
‡ due to COVID-19 lockdown lab was not able to complete its submission, evaluation for informative purpose only

based on corrected values, for details please see Section **4.4.2 (p. 51)**. Z-scores were calculated for informative purpose only.

Table 4-7 (cont.): Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compounds

COMPULSORY Compounds				2,4-D (free acid)		Carbofuran (sum)			Chlormequat-Cl		Glyphosate		TFNA	
MRRL [mg/kg]				0.01		0.01		*0.01*	0.01		0.03		0.01	
Assigned Value [mg/kg]				0.052		0.107*		*0.106#*	0.092		0.203		0.060	
*CV**				20.8 %		22.8 %*		*22.1 %#*	16.8 %		23.7 %		27.8 %	
Lab code SRM15-	NRL-SRM	Cat	Analysed / corr. found, max. 13 / 5	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	*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 %)
100		B	11 / 4			0.090	-0.6	*-0.6*	0.075	-0.8	0.210	0.1	0.040	-1.3
101		B	6 / 2						0.087	-0.2	0.202	0.0		
102		B	8 / 4	0.054	0.2	0.071	-1.3	*-1.3*	0.194	4.4	0.196	-0.1		
103	x	A	12 / 5	0.036	-1.2	0.105	-0.1	*0.0*	0.324	10.0	0.176	-0.5	0.070	0.7
104		B	1 / 1			0.066	-1.5	*-1.5*						
105	x	B	8 / 4	0.0556	0.3	0.135	1.0	*1.1*	0.101	0.4			0.0708	0.7
107		B	11 / 4	0.063	0.9	0.081	-1.0	*-0.9*	0.020	-3.1	0.052	-3.0		
108	x	A	13 / 5	0.061	0.7	0.093	-0.5	*-0.5*	0.103	0.5	0.199	-0.1	0.060	0.0
109		A	13 / 5	0.057	0.4	0.065	-1.6	*-1.6*	0.079	-0.6	0.104	-2.0	0.065	0.3
110		B	1 / 1			0.051	-2.1	*-2.1*						
111		A	13 / 5	0.039	-1.0	0.060	-1.8	*-1.7*	0.088	-0.2	0.245	0.8	0.087	1.8
112	x	A	12 / 5	0.0520	0.0	0.139	1.2	*1.2*	0.0870	-0.2	0.240	0.7	0.0420	-1.2
113		A	13 / 5	0.058	0.5	0.081	-1.0	*-0.9*	0.097	0.2	0.264	1.2	0.064	0.3
114		B	11 / 5	0.055	0.3	0.113	0.2	*0.3*	0.092	0.0	0.200	-0.1	0.072	0.8
115		A	13 / 5	0.055	0.3	0.060	-1.8	*-1.7*	0.10	0.3	0.24	0.7	0.056	-0.3
116	x	B	6 / 3	0.0582	0.5	0.127	0.7	*0.8*					0.031	-1.9
118	x	A	13 / 5	0.062	0.8	0.150	1.6	*0.6#*	0.099	0.3	0.149	-1.1	0.069	0.6
119	x	B	12 / 5	0.040	-0.9	0.108	0.0	*0.1*	0.112	0.9	0.299	1.9	0.047	-0.9
120		B	1 / 1			0.0645	-1.6	*-1.6*						
121		B	9 / 4	0.015	-2.8	0.260	5.7	*5.8*			0.280	1.5	0.093	2.2
122		A	13 / 5	0.0479	-0.3	0.125	0.7	*0.7*	0.104	0.5	0.232	0.6	0.0547	-0.4
123		A	13 / 5	0.063	0.9	0.104	-0.1	*-0.1*	0.097	0.2	0.205	0.0	0.044	-1.1
124		B	9 / 3	0.054	0.2				0.099	0.3	0.247	0.9		
125		A	13 / 5	0.057	0.4	0.112	0.2	*0.2*	0.087	-0.2	0.210	0.1	0.052	-0.6
127		B	7 / 3	0.040	-0.9				0.091	-0.1			0.088	1.9
128		A	13 / 5	0.065	1.0	0.101	-0.2	*-0.2*	0.119	1.2	0.200	-0.1	0.102	2.8
129		A	13 / 5	0.076	1.9	0.166	2.2	*2.3*	0.112	0.9	0.063	-2.8	0.032	-1.9
130		B	3 / 2						0.122	1.3	0.227	0.5		
132		B	5 / 2	0.054	0.2				0.094	0.1				
133		B	5 / 3	0.056	0.3	0.14	1.2	*1.3*	0.089	-0.2				
137		B	11 / 5	0.0611	0.7	0.147	1.5	*1.6*	0.0960	0.2	0.215	0.2	0.0651	0.3
3rd-29		B	3 / 1	0.0598	0.6									
3rd-43		B	5 / 3	0.071	1.5				0.087	-0.2	0.230	0.5		
3rd-72		B	4 / 2	0.0579	0.5								0.0708	0.7
3rd-73		B	11 / 5	0.048	-0.3	0.104	-0.1	*-0.1*	0.123	1.3	0.211	0.2	0.066	0.4
3rd-83		B	7 / 2	0.110	4.5				0.098	0.2				
3rd-86		B	2 / 2	0.0529	0.1						0.114	-1.8		
3rd-134		B	3 / 1	0.04	-0.9									
3rd-135		B	5 / 1			0.11	0.1	*0.1*						
10‡		B	9 / 5	0.064	1.0	0.064	-1.6	*-1.6*	0.094	0.1			0.058	-0.2

* based on subpopulation consisting of results generated by methods involving acidic hydrolysis or analysis of carbofuran and carbosulfan separately
‡ due to COVID-19 lockdown lab was not able to complete its submission, evaluation for informative purpose only

based on corrected values, for details please see Section **4.4.2 (p. 51)**. Z-scores were calculated for informative purpose only.

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

OPTIONAL Compounds				2,4-D (sum)		Bentazone		Fluazifop (sum)		Haloxyfop (sum)		Imazethapyr (free acid)	
MRRL [mg/kg]				0.01		0.01		0.01		0.01		0.01	
Assigned Value [mg/kg]				0.059		0.334		0.060*		0.151		0.206	
*CV**				18.9 %		19.4 %		18.8 %*		16.2 %		17.6 %	
Lab code SRM15-	NRL-SRM	Cat	Analysed / corr. found, max. 17 / 11	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 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
5		B	1 / 1	0.028	-2.1								
8	x	B	13 / 8	0.0406	-1.2	0.768	5.2	0.0141	-3.1	0.162	0.3	0.222	0.3
9	x	B	11 / 8	0.198	9.5	0.384	0.6	0.096	2.4	0.205	1.4		
11		B	0 / 0										
12	x	A	17 / 11	0.047	-0.8	0.323	-0.1	0.061	0.1	0.168	0.4	0.198	-0.2
13	x	A	16 / 10	0.0578	-0.1	0.329	-0.1	0.0579	-0.1	0.150	0.0		
16	x	A	17 / 11	0.060	0.1	0.403	0.8	0.050	-0.7	0.158	0.2	0.220	0.3
18		B	0 / 0										
19		A	16 / 9	0.037	-1.5	0.26	-0.9	0.046	-0.9	FN	-3.7		
20	x	B	6 / 3			0.270	-0.8	0.056	-0.3				
21	x	A	14 / 9	0.0581	0.0	0.337	0.0	0.0174	-2.8	0.129	-0.6		
22	x	B	12 / 9	0.060	0.1	0.347	0.2	0.065	0.3	0.135	-0.4		
23	x	A	7 / 2			0.369	0.4						
24		A	16 / 9	0.090	2.1	0.127	-2.5	0.056	-0.3	0.27	3.1		
25		B	12 / 9	0.057	-0.1	0.326	-0.1	0.052	-0.5	0.134	-0.5		
26		A	17 / 11	0.081	1.5	0.281	-0.6	0.059	-0.1	0.144	-0.2	0.095	-2.2
27		A	11 / 7	0.056	-0.2	0.305	-0.4	0.056	-0.3	0.153	0.1		
28		A	7 / 3			0.375	0.5					0.211	0.1
30		B	15 / 8	0.048	-0.7	0.704	4.4	0.041	-1.3	0.048	-2.7	0.199	-0.1
31		A	10 / 7	0.0452	-0.9	0.308	-0.3	0.0152	-3.0	0.163	0.3		
32		A	14 / 9	0.0653	0.5	0.339	0.1	0.0741	0.9	0.171	0.5		
33		B	9 / 5	0.187	8.8	0.315	-0.2						
34		A	17 / 7	0.1	2.8	0.35	0.2	0.063	0.2	0.14	-0.3	0.21	0.1
35		A	8 / 3			0.436	1.2					0.194	-0.2
36		B	1 / 0										
37		B	12 / 8	0.0508	-0.5	0.198	-1.6	0.0511	-0.6	0.115	-1.0		
38		B	0 / 0										
39		A	3 / 1			0.392	0.7						
40		B	6 / 5	0.0498	-0.6	0.4200	1.0	0.0698	0.7	0.1354	-0.4		
41	x	B	2 / 2					0.068	0.5	0.066	-2.3		
42	x	B	2 / 1										
44		A	17 / 11	0.09	2.1	0.69	4.3	0.055	-0.3	0.187	1.0	0.23	0.5
45	x	A	5 / 1			0.381	0.6						
46	x	A	13 / 8	0.064	0.4	0.342	0.1	0.020	-2.7	0.199	1.3		
47		B	12 / 9	0.076	1.2	0.329	-0.1	0.033	-1.8	0.152	0.0	0.194	-0.2
48		A	15 / 10	0.070	0.8	0.325	-0.1	0.055	-0.3	0.133	-0.5	0.214	0.2
49		A	17 / 11	0.058	0.0	0.269	-0.8	0.076	1.1	0.146	-0.1	0.185	-0.4
50		B	16 / 11	0.0655	0.5	0.342	0.1	0.0622	0.1	0.1528	0.0	0.1989	-0.1
51		A	17 / 8	0.050	-0.6	0.288	-0.6	0.010	-3.3	0.146	-0.1	0.221	0.3
54		B	1 / 0										
55		B	13 / 10	0.137	5.4	0.388	0.7	0.0811	1.4	0.222	1.9	0.264	1.1

* based on subpopulation, for fluazifop (sum), MCPB (sum) subpopulation consisting results generated by methods involving moderate or strong hydrolysis, for mecoprop (sum) consisting results generated by methods involving strong hydrolysis only

‡ due to COVID-19 shut down lab was not able to complete its submission, evaluation for informative purpose only

Optional Compounds				MCPA (sum)		MCPB (sum)		Mecoprop (sum)		Paraquat		Quizalofop (free acid)		Quizalofop (sum)	
MRRL [mg/kg]				0.01		0.01		0.01		0.02		0.01		0.01	
Assigned Value [mg/kg]				0.068		0.057*		0.067*		0.195		0.044		0.062	
*CV**				20.2 %		29.4 %*		22.4 %*		28.9 %		16.4 %		22.9 %	
Lab code SRM15-	NRL-SRM	Cat	Analysed / corr. found, max. 17 / 11	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 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
5		B	1 / 1												
8	x	B	13 / 8	0.0850	1.0	0.0202	-2.6	FN	-3.4	0.478	5.8			FN	-3.4
9	x	B	11 / 8	0.206	8.2			0.096	1.7			0.091	4.2	0.136	4.8
11		B	0 / 0												
12	x	A	17 / 11	0.075	0.4	0.055	-0.1	0.020	-2.8	0.183	-0.3	0.049	0.4	0.062	0.0
13	x	A	16 / 10	0.0628	-0.3	0.0580	0.1	0.0653	-0.1	0.180	-0.3	0.0389	-0.5	0.0604	-0.1
16	x	A	17 / 11	0.071	0.2	0.063	0.4	0.076	0.5	0.241	0.9	0.046	0.1	0.062	0.0
18		B	0 / 0												
19		A	16 / 9	0.067	0.0	0.055	-0.1	0.039	-1.7	0.13	-1.3	0.043	-0.1	0.063	0.1
20	x	B	6 / 3									0.039	-0.5		
21	x	A	14 / 9	0.0663	-0.1	0.0232	-2.4	FN	-3.4	0.174	-0.4	0.0441	0.0	0.0544	-0.5
22	x	B	12 / 9	0.070	0.2	0.074	1.2	0.073	0.4			0.048	0.3	0.062	0.0
23	x	A	7 / 2									0.0505	0.6		
24		A	16 / 9	0.091	1.4	0.060	0.2	FN	-3.4	0.124	-1.5	0.042	-0.2	0.084	1.4
25		B	12 / 9	0.061	-0.4	0.021	-2.5	0.033	-2.0			0.043	-0.1	0.053	-0.6
26		A	17 / 11	0.068	0.0	0.070	0.9	0.043	-1.4	0.220	0.5	0.020	-2.2	0.057	-0.3
27		A	11 / 7	0.065	-0.2	0.053	-0.3	0.024	-2.6						
28		A	7 / 3									0.0414	-0.3		
30		B	15 / 8	0.053	-0.9	FN	-3.3	FN	-3.4	0.200	0.1	0.036	-0.8		
31		A	10 / 7	0.0536	-0.8							0.0453	0.1	0.0636	0.1
32		A	14 / 9	0.0808	0.8	0.0657	0.6	0.0823	0.9			0.0518	0.7	0.0599	-0.1
33		B	9 / 5	0.212	8.6	0.122	4.6	0.202	8.1						
34		A	17 / 7	FN	-3.4	FN	-3.3	FN	-3.4	0.36	3.4	FN	-3.1	0.066	0.3
35		A	8 / 3									0.0470	0.2		
36		B	1 / 0												
37		B	12 / 8	0.0564	-0.7	0.042	-1.1	FN	-3.4			0.0243	-1.8	0.0404	-1.4
38		B	0 / 0												
39		A	3 / 1												
40		B	6 / 5	0.0512	-1.0										
41	x	B	2 / 2												
42	x	B	2 / 1							0.358	3.3				
44		A	17 / 11	0.046	-1.3	0.039	-1.3	0.031	-2.1	0.212	0.4	0.043	-0.1	0.054	-0.5
45	x	A	5 / 1												
46	x	A	13 / 8	0.077	0.6	0.071	1.0	FN	-3.4			0.080	3.2	0.113	3.3
47		B	12 / 9	0.079	0.7	0.070	0.9	FN	-3.4	0.097	-2.0			0.068	0.4
48		A	15 / 10	0.078	0.6	0.046	-0.8	0.066	-0.1			0.040	-0.4	0.048	-0.9
49		A	17 / 11	0.062	-0.3	0.068	0.8	0.071	0.2	0.173	-0.5	0.036	-0.8	0.050	-0.8
50		B	16 / 11	0.0736	0.4	0.0520	-0.4	0.0645	-0.1	0.155	-0.8	0.0454	0.1	0.0689	0.5
51		A	17 / 8	0.054	-0.8	0.022	-2.5	FN	-3.4	FN	-3.8	0.048	0.3	FN	-3.4
54		B	1 / 0												
55		B	13 / 10	0.144	4.5	0.134	5.4	0.135	4.1			0.0525	0.7	0.0990	2.4

* based on subpopulation, for fluazifop (sum), MCPB (sum) subpopulation consisting results generated by methods involving moderate or strong hydrolysis, for mecoprop (sum) consisting results generated by methods involving strong hydrolysis only

‡ due to COVID-19 lockdown lab was not able to complete its submission, evaluation for informative purpose only

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

OPTIONAL Compounds				2,4-D (sum)		Bentazone		Fluazifop (sum)		Haloxyfop (sum)		Imazethapyr (free acid)	
MRRL [mg/kg]				0.01		0.01		0.01		0.01		0.01	
Assigned Value [mg/kg]				0.059		0.334		0.060*		0.151		0.206	
*CV**				18.9 %		19.4 %		18.8 %*		16.2 %		17.6 %	
Lab code SRM15-	NRL-SRM	Cat	Analysed / corr. found, max. 17 / 11	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 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
56		A	16 / 10	0.053	-0.4	0.333	0.0	0.048	-0.8	0.152	0.0	0.154	-1.0
57		A	17 / 11	0.060	0.1	0.354	0.2	0.075	1.0	0.152	0.0	0.179	-0.5
58		B	11 / 7	0.0547	-0.3			0.0113	-3.2	0.127	-0.6		
59		B	12 / 9	0.068	0.6	0.334	0.0	0.067	0.5	0.148	-0.1		
60		B	4 / 2			0.2625	-0.9					0.1750	-0.6
61	x	B	3 / 0										
62	x	A	11 / 7	0.121	4.3	0.387	0.6	0.073	0.9	0.232	2.1		
63		B	1 / 0										
64		B	12 / 9	0.062	0.2	0.365	0.4	0.049	-0.7	0.144	-0.2	0.178	-0.6
65	x	A	10 / 4			0.209	-1.5					0.140	-1.3
66		A	11 / 6	0.080	1.5	0.372	0.5						
67		A	13 / 9	0.060	0.1	0.392	0.7	0.049	-0.7	0.181	0.8		
68	x	B	10 / 5	0.049	-0.7			0.016	-2.9	0.140	-0.3		
69		B	3 / 1			0.26	-0.9						
70		B	0 / 0										
71	x	B	11 / 8	0.0554	-0.2	0.272	-0.7			0.119	-0.9	0.227	0.4
74	x	A	17 / 9	0.068	0.6	0.352	0.2	FN	-3.3	0.143	-0.2	0.300	1.8
75		B	1 / 0										
77	x	B	0 / 0										
79		B	0 / 0										
80		B	0 / 0										
82		B	3 / 3			0.342	0.1	0.0608	0.1	0.152	0.0		
84		B	11 / 8	0.0544	-0.3			0.0465	-0.9	0.148	-0.1		
85		B	1 / 1										
87	x	A	15 / 10	0.056	-0.2	0.338	0.1	0.057	-0.2	0.143	-0.2	0.195	-0.2
88	x	B	2 / 1			0.27	-0.8						
90		B	16 / 11	0.057	-0.1	0.45	1.4	0.089	1.9	0.20	1.3	0.35	2.8
91	x	A	13 / 6	0.0537	-0.3	0.315	-0.2	FN	-3.3	0.0755	-2.0		
92		B	14 / 10	0.061	0.2	0.300	-0.4	0.056	-0.3	0.139	-0.3	0.200	-0.1
93		B	13 / 10	0.0582	0.0	0.377	0.5	0.0518	-0.5	0.116	-0.9	0.217	0.2
94		B	5 / 2			0.203	-1.6	0.050	-0.7				
95	x	B	6 / 2			0.217	-1.4						
96		B	14 / 10	0.067	0.6	0.387	0.6	0.0630	0.2	0.136	-0.4	0.222	0.3
97		B	3 / 1										
98		A	16 / 11	0.047	-0.8	0.48	1.8	0.056	-0.3	0.14	-0.3	0.024	-3.5
99		B	13 / 7	0.05	-0.6	0.25	-1.0	0.06	0.0	0.17	0.5	0.28	1.4
100		B	10 / 8	0.049	-0.7	0.286	-0.6	0.056	-0.3	0.160	0.2	FN	-3.8
101		B	2 / 2			0.439	1.3						
102		B	0 / 0										
103	x	A	13 / 9	0.036	-1.5	0.228	-1.3	0.018	-2.8	0.044	-2.8	0.183	-0.5
104		B	0 / 0										
105	x	B	0 / 0										

* based on subpopulation, for fluazifop (sum), MCPB (sum) subpopulation consisting results generated by methods involving moderate or strong hydrolysis, for mecoprop (sum) consisting results generated by methods involving strong hydrolysis only

‡ due to COVID-19 shut down lab was not able to complete its submission, evaluation for informative purpose only

Optional Compounds				MCPA (sum)		MCPB (sum)		Mecoprop (sum)		Paraquat		Quizalofop (free acid)		Quizalofop (sum)	
MRRL [mg/kg]				0.01		0.01		0.01		0.02		0.01		0.01	
Assigned Value [mg/kg]				0.068		0.057*		0.067*		0.195		0.044		0.062	
*CV**				20.2 %		29.4 %*		22.4 %*		28.9 %		16.4 %		22.9 %	
Lab code SRM15-	NRL-SRM	Cat	Analysed / corr. found, max. 17 / 11	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 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
56		A	16 / 10	0.060	-0.4	0.031	-1.8	0.014	-3.2			0.034	-0.9	0.050	-0.8
57		A	17 / 11	0.065	-0.2	0.056	-0.1	0.034	-2.0	0.217	0.5	0.043	-0.1	0.065	0.2
58		B	11 / 7	0.061	-0.4	0.0302	-1.9					0.0426	-0.2	0.0530	-0.6
59		B	12 / 9	0.082	0.9	0.08	1.6	0.083	1.0			0.041	-0.3	0.097	2.3
60		B	4 / 2												
61	x	B	3 / 0												
62	x	A	11 / 7	0.0895	1.3	0.074	1.2	0.058	-0.5						
63		B	1 / 0												
64		B	12 / 9	0.062	-0.3	0.049	-0.6	FN	-3.4	0.174	-0.4	0.035	-0.9		
65	x	A	10 / 4							0.431	4.8	0.0288	-1.4		
66		A	11 / 6	0.071	0.2			FN	-3.4	0.142	-1.1	0.046	0.1	0.083	1.4
67		A	13 / 9	0.088	1.2			0.018	-2.9	0.184	-0.2	0.061	1.5	0.085	1.5
68	x	B	10 / 5	0.064	-0.2	0.026	-2.2	FN	-3.4						
69		B	3 / 1												
70		B	0 / 0												
71	x	B	11 / 8	0.0595	-0.5	0.0232	-2.4					0.0516	0.6	0.0572	-0.3
74	x	A	17 / 9	0.075	0.4	0.019	-2.7	FN	-3.4	0.131	-1.3	0.044	0.0	0.051	-0.7
75		B	1 / 0												
77	x	B	0 / 0												
79		B	0 / 0												
80		B	0 / 0												
82		B	3 / 3												
84		B	11 / 8	0.0587	-0.5	0.0515	-0.4	0.0298	-2.2			0.0437	-0.1	0.0603	-0.1
85		B	1 / 1							0.174	-0.4				
87	x	A	15 / 10	0.063	-0.3	0.050	-0.5	0.056	-0.7			0.043	-0.1	0.059	-0.2
88	x	B	2 / 1												
90		B	16 / 11	0.076	0.5	0.073	1.1	0.070	0.2	0.2	0.1	0.044	0.0	0.070	0.5
91	x	A	13 / 6	0.0581	-0.6	FN	-3.3	FN	-3.4			0.0466	0.2	0.0534	-0.6
92		B	14 / 10	0.056	-0.7	0.053	-0.3	0.064	-0.2			0.046	0.1	0.054	-0.5
93		B	13 / 10	0.0705	0.2	0.0381	-1.3	0.0171	-3.0			0.0487	0.4	0.0614	0.0
94		B	5 / 2												
95	x	B	6 / 2									0.0421	-0.2		
96		B	14 / 10	0.0553	-0.7	0.0520	-0.4	0.0523	-0.9			0.0363	-0.7	0.0563	-0.4
97		B	3 / 1									0.0554	1.0		
98		A	16 / 11	0.081	0.8	0.067	0.7	0.078	0.7	0.15	-0.9	0.051	0.6	0.088	1.7
99		B	13 / 7	0.05	-1.0	0.09	2.3			FN	-3.6	FN	-3.1		
100		B	10 / 8	0.051	-1.0	0.034	-1.6	0.025	-2.5					0.060	-0.1
101		B	2 / 2									0.053	0.8		
102		B	0 / 0												
103	x	A	13 / 9	0.054	-0.8	0.032	-1.8	FN	-3.4			0.047	0.2	0.047	-1.0
104		B	0 / 0												
105	x	B	0 / 0												

* based on subpopulation, for fluazifop (sum), MCPB (sum) subpopulation consisting results generated by methods involving moderate or strong hydrolysis, for mecoprop (sum) consisting results generated by methods involving strong hydrolysis only

‡ due to COVID-19 lockdown lab was not able to complete its submission, evaluation for informative purpose only

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

OPTIONAL Compounds				2,4-D (sum)		Bentazone		Fluazifop (sum)		Haloxyfop (sum)		Imazethapyr (free acid)	
MRRL [mg/kg]				0.01		0.01		0.01		0.01		0.01	
Assigned Value [mg/kg]				0.059		0.334		0.060*		0.151		0.206	
*CV**				18.9 %		19.4 %		18.8 %*		16.2 %		17.6 %	
Lab code SRM15-	NRL-SRM	Cat	Analysed / corr. found, max. 17 / 11	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 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
107		B	3 / 0										
108	x	A	11 / 6	0.064	0.4			0.059	-0.1	0.154	0.1		
109		A	4 / 0										
110		B	1 / 1					0.078	1.2				
111		A	14 / 10	0.023	-2.4	0.314	-0.2	0.046	-0.9	0.069	-2.2	0.208	0.0
112	x	A	3 / 0										
113		A	17 / 11	0.065	0.4	0.334	0.0	0.062	0.1	0.154	0.1	0.218	0.2
114		B	16 / 11	0.058	0.0	0.389	0.7	0.092	2.1	0.178	0.7	0.325	2.3
115		A	15 / 9	0.053	-0.4	0.240	-1.1	0.052	-0.5	0.15	0.0	FN	-3.8
116	x	B	12 / 8	0.0652	0.5	0.327	-0.1	0.0351	-1.7	0.183	0.8		
118	x	A	17 / 11	0.055	-0.3	0.323	-0.1	0.062	0.1	0.150	0.0	0.187	-0.4
119	x	B	13 / 8	0.041	-1.2	0.338	0.1	0.063	0.2	0.187	1.0		
120		B	0 / 0										
121		B	4 / 1			0.244	-1.1						
122		A	15 / 9	0.0541	-0.3	0.321	-0.2	0.0514	-0.6	0.133	-0.5	0.176	-0.6
123		A	17 / 11	0.063	0.3	0.293	-0.5	0.067	0.5	0.164	0.3	0.181	-0.5
124		B	13 / 9	0.060	0.1	0.352	0.2	0.063	0.2	0.162	0.3		
125		A	17 / 11	0.059	0.0	0.450	1.4	0.063	0.2	0.152	0.0	0.265	1.1
127		B	1 / 0										
128		A	17 / 11	0.066	0.5	0.355	0.3	0.061	0.1	0.141	-0.3	0.220	0.3
129		A	13 / 9	0.102	3.0	0.404	0.8	0.073	0.9	0.170	0.5		
130		B	1 / 0										
132		B	9 / 5	0.070	0.8	0.39	0.7						
133		B	3 / 3			0.333	0.0			0.160	0.2		
137		B	15 / 7	0.0611	0.2	0.344	0.1	0.0743	1.0	0.155	0.1		
3rd-29		B	13 / 10	0.0603	0.1	0.316	-0.2	0.0544	-0.4	0.153	0.1	0.242	0.7
3rd-43		B	6 / 3			0.294	-0.5					0.207	0.0
3rd-72		B	4 / 2									0.213	0.1
3rd-73		B	14 / 8	0.176	8.0	0.414	1.0	0.072	0.8	0.2	1.3	0.242	0.7
3rd-83		B	7 / 2			0.293	-0.5					0.195	-0.2
3rd-86		B	6 / 3			0.338	0.1					0.237	0.6
3rd-134		B	5 / 1	0.04	-1.3			FN	-3.3	FN	-3.7		
3rd-135		B	2 / 0									FN	-3.8
10‡		B	11 / 5	0.064	0.4	0.364	0.4	0.052	-0.5				

* based on subpopulation, for fluazifop (sum), MCPB (sum) subpopulation consisting results generated by methods involving moderate or strong hydrolysis, for mecoprop (sum) consisting results generated by methods involving strong hydrolysis only

‡ due to COVID-19 shut down lab was not able to complete its submission, evaluation for informative purpose only

Optional Compounds				MCPA (sum)		MCPB (sum)		Mecoprop (sum)		Paraquat		Quizalofop (free acid)		Quizalofop (sum)	
MRRL [mg/kg]				0.01		0.01		0.01		0.02		0.01		0.01	
Assigned Value [mg/kg]				0.068		0.057*		0.067*		0.195		0.044		0.062	
*CV**				20.2 %		29.4 %*		22.4 %*		28.9 %		16.4 %		22.9 %	
Lab code SRM15-	NRL-SRM	Cat	Analysed / corr. found, max. 17 / 11	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 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
107		B	3 / 0												
108	x	A	11 / 6	0.064	-0.2	0.052	-0.4	0.050	-1.0						
109		A	4 / 0												
110		B	1 / 1												
111		A	14 / 10	0.032	-2.1	0.023	-2.4	0.013	-3.2			0.036	-0.8	0.032	-1.9
112	x	A	3 / 0												
113		A	17 / 11	0.102	2.0	0.117	4.2	0.062	-0.3	0.171	-0.5	0.040	-0.4	0.056	-0.4
114		B	16 / 11	0.075	0.4	0.070	0.9	0.070	0.2	0.225	0.6	0.045	0.1	0.065	0.2
115		A	15 / 9	0.060	-0.4	0.054	-0.2			0.24	0.9	0.040	-0.4	0.058	-0.3
116	x	B	12 / 8	0.0918	1.4	0.0324	-1.7	0.0146	-3.1			FN	-3.1	0.0617	0.0
118	x	A	17 / 11	0.061	-0.4	0.056	-0.1	0.061	-0.4	0.141	-1.1	0.050	0.5	0.081	1.2
119	x	B	13 / 8	0.043	-1.5	0.049	-0.6	FN	-3.4			0.037	-0.7	0.047	-1.0
120		B	0 / 0												
121		B	4 / 1									FN	-3.1		
122		A	15 / 9	0.0587	-0.5	0.0447	-0.9	FN	-3.4			0.0343	-0.9	0.0451	-1.1
123		A	17 / 11	0.077	0.6	0.037	-1.4	0.026	-2.4	0.28	1.7	0.037	-0.7	0.038	-1.5
124		B	13 / 9	0.068	0.0	0.052	-0.4	0.049	-1.1			0.046	0.1	0.070	0.5
125		A	17 / 11	0.065	-0.2	0.056	-0.1	0.055	-0.7	0.185	-0.2	0.051	0.6	0.059	-0.2
127		B	1 / 0												
128		A	17 / 11	0.071	0.2	0.056	-0.1	0.069	0.1	0.226	0.6	0.055	1.0	0.055	-0.4
129		A	13 / 9	0.100	1.9	0.046	-0.8	0.099	1.9			0.055	1.0	0.086	1.6
130		B	1 / 0												
132		B	9 / 5	0.092	1.5	0.088	2.2	0.041	-1.6						
133		B	3 / 3	0.062	-0.3										
137		B	15 / 7	0.0633	-0.3	FN	-3.3	FN	-3.4	0.268	1.5	0.0497	0.5		
3rd-29		B	13 / 10	0.0687	0.1	0.0573	0.0	0.0863	1.2			0.0519	0.7	0.0670	0.3
3rd-43		B	6 / 3							0.120	-1.5				
3rd-72		B	4 / 2									0.0477	0.3		
3rd-73		B	14 / 8	0.074	0.4	0.055	-0.1	0.075	0.5	FN	-3.8				
3rd-83		B	7 / 2												
3rd-86		B	6 / 3							0.106	-1.8				
3rd-134		B	5 / 1												
3rd-135		B	2 / 0												
10‡		B	11 / 5	0.007	-3.6			FN	-3.4	FN	-3.6				

* based on subpopulation, for fluazifop (sum), MCPB (sum) subpopulation consisting results generated by methods involving moderate or strong hydrolysis, for mecoprop (sum) consisting results generated by methods involving strong hydrolysis only

‡ due to COVID-19 lockdown lab was not able to complete its submission, evaluation for informative purpose only

A compilation of all individual results and z-scores for each laboratory is shown in **Table 4-7 (p. 35)** for compulsory compounds and **Table 4-8 (p. 38)** for optional compounds. 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**.

4.3.4 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 cited in **Section 2.5 (p. 35)**. Following the rules defined in the General Protocol (8th Edition, see **Appendix 8**), a laboratory had to fulfil the following conditions in order to be classified into Category A in the present PT: a) analysis of at least twelve out of the 13 compulsory pesticides on the Target Pesticides List; b) correct detection of at least four out of the five compulsory pesticides present in the test item, and c) no false positive results.

A total of 44 EU and EFTA laboratories (41 %) were classified into Category A and 64 (59 %) into Category B. All 8 laboratories from EU candidate and third-countries were classified into Category B. Considering only the compulsory compounds the laboratories from EU and EFTA countries classified into Category A achieved an overall AAZ of 0.8 (n = 219), whereas those classified into Category B achieved an overall AAZ of 1.0 (n = 206). When including laboratories from EU candidate and third countries, the AAZ for the compsulsory and optional compounds remains the same, but the number of the submitted results classified into Category B increased to n = 233.

Table 4-9 (p. 45) and **Table 4-10 (p. 46)** show the details of laboratories classified into Category A and B, respectively. For informative purposes, the overall AAZ was calculated for laboratories with 4 or more individual z-scores among the compulsory compounds. For the AAZ calculation any z-scores > 5 were set at 5.

4.3.5 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 ($|z\text{-score}| \geq 3$) or false positive 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 participating 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 order to assist the participants to find the error sources, the organizers stated for the first time in the Preliminary Report comprehensive indications on potential error sources driven from the reported information on methods that have been applied by the participants to analyse the compounds in the current PT.

In total, 1103 numerical results for the analytes present in the test items, 43 false negative results and 1 false positive result were reported by 108 participants from EU/EFTA countries. 67 EU/EFTA laboratories reported 155 cases of poor performance ($|z|>2$, incl. 43 FNs, and 1 FPs). 60 laboratories reported for 134 cases the (possible) reasons or their investigation, even though no clear reasons for the poor performance could be identified. In another 13 cases (each one case for ***fluazifop (sum),*** c***arbofuran (sum)***, ***haloxyfop (sum)***, 3× ***MCPB (sum)*** and 5× ***mecoprop (sum)***) the 6 laboratories did not report their error source after their investigation.

Table 4-9: Category A laboratories in EUPT-SRM15, ordered by lab-codes.

COMPULSORY Compounds			2,4-D (free acid)	Carbofuran (sum)	Chlormequat-Cl	Glyphosate	TFNA	
MRRL [mg/kg]			0.01	0.01	0.01	0.03	0.01	
Assigned Value [mg/kg]			0.052	0.107*	0.092	0.203	0.060	
*CV**			20.8 %	22.8 %*	16.8 %	23.7 %	27.8 %	
Lab code SRM15-	NRL-SRM	Analysed / corr. found, max. 13 / 5	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	AAZ
12	x	13 / 5	-0.4	0.0	-0.1	-0.9	-1.4	0.6
13	x	12 / 5	0.3	0.2	-1.1	0.0	-1.5	0.6
16	x	13 / 5	0.7	0.6	0.4	0.2	1.1	0.6
19		13 / 5	-1.1	-0.3	-0.5	1.1	-0.9	0.8
21	x	13 / 5	0.5	-1.2	-0.2	1.6	-0.2	0.7
23	x	13 / 5	-0.1	0.6	-0.6	0.9	0.1	0.5
24		13 / 5	-1.4	6.5	-1.2	-1.1	-0.1	1.8
26		13 / 5	-0.1	0.5	0.2	-0.1	-0.2	0.2
27		13 / 5	-0.1	-1.2	0.7	-0.3	0.4	0.5
28		13 / 5	0.3	-0.9	-1.8	0.7	0.5	0.8
31		12 / 4	-0.3		0.5	0.5	0.9	0.6
32		13 / 5	0.7	0.9	0.8	-0.5	0.9	0.8
34		13 / 5	3.8	-2.0	0.8	0.5	0.7	1.6
35		13 / 5	-0.7	-0.9	-0.5	-1.5	-1.2	1.0
39		13 / 5	0.9	-2.2	0.6	1.0	-1.0	1.1
44		13 / 5	-0.5	-0.3	-1.3	-0.6	0.3	0.6
45	x	13 / 5	-0.1	-0.1	-0.7	0.8	1.7	0.7
46	x	13 / 5	0.2	-0.4	-0.2	-0.2	0.1	0.2
48		13 / 5	-1.8	-0.3	0.9	-0.1	-0.9	0.8
49		13 / 5	0.3	-1.3	-0.4	0.4	1.7	0.8
51		13 / 4	-1.0	-1.7	-0.3	4.1	-3.3	2.1
56		13 / 5	-0.7	-0.9	0.5	-0.6	0.1	0.6
57		13 / 5	0.0	0.9	0.4	0.1	-0.1	0.3
62	x	13 / 5	-2.8	-0.3	-0.2	0.0	0.3	0.7
65	x	13 / 4	-1.3	0.2	0.0	1.4	-3.3	1.2
66		13 / 5	2.8	-2.1	0.2	-2.0	-2.0	1.8
67		13 / 5	-0.7	-0.5	-0.3	-3.6	1.1	1.2
74	x	13 / 5	1.0	-0.3	-0.2	-0.1	-0.2	0.4
87	x	13 / 5	0.1	-1.0	0.6	0.6	-0.4	0.5
91	x	13 / 5	0.0	0.7	-0.4	-0.2	0.3	0.3
98		13 / 5	-0.4	-0.7	0.3	0.3	-1.3	0.6
103	x	12 / 5	-1.2	-0.1	10.0	-0.5	0.7	1.5
108	x	13 / 5	0.7	-0.5	0.5	-0.1	0.0	0.4
109		13 / 5	0.4	-1.6	-0.6	-2.0	0.3	1.0
111		13 / 5	-1.0	-1.8	-0.2	0.8	1.8	1.1
112	x	12 / 5	0.0	1.2	-0.2	0.7	-1.2	0.7
113		13 / 5	0.5	-1.0	0.2	1.2	0.3	0.6
115		13 / 5	0.3	-1.8	0.3	0.7	-0.3	0.7
118	x	13 / 5	0.8	1.6	0.3	-1.1	0.6	0.9
122		13 / 5	-0.3	0.7	0.5	0.6	-0.4	0.5
123		13 / 5	0.9	-0.1	0.2	0.0	-1.1	0.5
125		13 / 5	0.4	0.2	-0.2	0.1	-0.6	0.3
128		13 / 5	1.0	-0.2	1.2	-0.1	2.8	1.1
129		13 / 5	1.9	2.2	0.9	-2.8	-1.9	1.9

* based on subpopulation consisting of results generated by methods involving acidic hydrolysis or analysis of carbofuran and carbosulfan separately

Table 4-10: Category B laboratories in EUPT-SRM15, ordered by lab-codes.

COMPULSORY Compounds			2,4-D (free acid)	Carbofuran (sum)	Chlormequat-Cl	Glyphosate	TFNA	
MRRL [mg/kg]			0.01	0.01	0.01	0.03	0.01	
Assigned Value [mg/kg]			0.052	0.107*	0.092	0.203	0.060	
*CV**			20.8 %	22.8 %*	16.8 %	23.7 %	27.8 %	
Lab code SRM15-	NRL-SRM	Analysed / corr. found, max. 13 / 5	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	AAZ
5		1 / 0	-3.2					
8	x	10 / 4		1.3	-1.8	-2.7	2.7	2.1
9	x	11 / 4	3.4	0	1.2	-0.4		1.2
11		7 / 4	-0.1	-0.1	-1	-0.4		0.4
18		1 / 1		-1.7				
20	x	6 / 4	-0.7	-1.4	0.2	-1.3		0.9
22	x	10 / 5	0.1	0.3	0.2	-0.3	0.5	0.2
25		6 / 3	0	0.3	0			
30		11 / 5	-0.4	-0.3	0.1	0.1	0.1	0.2
33		6 / 3	-3.2	-2.2	0.3	0.6		
36		1 / 1				-0.1		
37		10 / 5	-1.1	-1.6	0	0.2	0.3	0.6
38		3 / 2		-2.3	0.2			
40		6 / 3	0.8	-0.7			0.4	
41	x	6 / 2	-3.2	-0.1		-1.8		
42	x	2 / 1			1.5			
47		10 / 4		-3	-1.3	-0.2	-2	1.6
50		10 / 5	-0.1	-0.2	-0.4	0.5	10	1.2
54		3 / 1				0.3		
55		11 / 5	-0.9	2.7	-0.3	-0.8	-0.3	1
58		9 / 3	0.2	-1.1		0.1		
59		7 / 4	2	0	-0.7	1.4		1
60		4 / 2	-1.8	-1				
61	x	7 / 4	0.6	-0.1	5.9	0.9		1.6
63		5 / 1				-2.2		
64		11 / 5	0.9	3.6	-0.5	1.1	-0.9	1.4
68	x	8 / 4	-0.1		-0.1	0.9	0.1	0.3
69		4 / 3	0.1	-1.2		-3.6		
70		1 / 1		-0.6				
71	x	9 / 4	0	-0.1	1.5		0.6	0.5
75		10 / 5	-3.1	1.6	-0.8	0.1	-3	1.7
77	x	8 / 4	-0.1	0.1	-0.7		0.7	0.4
79		3 / 3		0.1	-0.3	0.3		
80		4 / 1			-1			
82		4 / 2		-0.3	0.2			
84		3 / 1	-0.2					
85		0 / 0						
88	x	9 / 4	2.1	-2.1	0.9	-0.1		1.3
90		11 / 5	0.3	0.5	0.3	-0.1	1.3	0.5
92		10 / 4	0.7		-0.3	0.3	-0.5	0.4
93		6 / 3	0	-1.2			0.5	
94		11 / 4	-2.5	-1.1	-0.6	0.8	-3.3	1.6
95	x	9 / 5	-1	-1.3	29.9	-1.4	-1.9	2.1
96		10 / 4	-0.1	0	1.3	-1.6		0.7

* based on subpopulation consisting of results generated by methods involving acidic hydrolysis or analysis of carbofuran and carbosulfan separately

‡ due to COVID-19 lockdown lab was not able to complete its submission, evaluation for informative purpose only

COMPULSORY Compounds			2,4-D (free acid)	Carbofuran (sum)	Chlormequat-Cl	Glyphosate	TFNA	
MRRL [mg/kg]			0.01	0.01	0.01	0.03	0.01	
Assigned Value [mg/kg]			0.052	0.107*	0.092	0.203	0.060	
*CV**			20.8 %	22.8 %*	16.8 %	23.7 %	27.8 %	
Lab code SRM15-	NRL-SRM	Analysed / corr. found, max. 13 / 5	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	z-Score (FFP-RSD = 25 %)	AAZ
97		6 / 3	0.4	-2.1			0.6	
99		11 / 4	-3.2	-1.8	-0.1	-0.5	0	1.1
100		11 / 4		-0.6	-0.8	0.1	-1.3	0.7
101		6 / 2			-0.2	0		
102		8 / 4	0.2	-1.3	4.4	-0.1		1.5
104		1 / 1		-1.5				
105	x	8 / 4	0.3	1	0.4		0.7	0.6
107		11 / 4	0.9	-1	-3.1	-3		2
110		1 / 1		-2.1				
114		11 / 5	0.3	0.2	0	-0.1	0.8	0.2
116	x	6 / 3	0.5	0.7			-1.9	
119	x	12 / 5	-0.9	0	0.9	1.9	-0.9	0.9
120		1 / 1		-1.6				
121		9 / 4	-2.8	5.7		1.5	2.2	2.8
124		9 / 3	0.2		0.3	0.9		
127		7 / 3	-0.9		-0.1		1.9	
130		3 / 2			1.3	0.5		
132		5 / 2	0.2		0.1			
133		5 / 3	0.3	1.2	-0.2			
137		11 / 5	0.7	1.5	0.2	0.2	0.3	0.5
3rd-29		3 / 1	0.6					
3rd-43		5 / 3	1.5		-0.2	0.5		
3rd-72		4 / 2	0.5				0.7	
3rd-73		11 / 5	-0.3	-0.1	1.3	0.2	0.4	0.4
3rd-83		7 / 2	4.5		0.2			
3rd-86		2 / 2	0.1			-1.8		
3rd-134		3 / 1	-0.9					
3rd-135		5 / 1		0.1				
10‡		5 / 1		0.1				

* based on subpopulation consisting of results generated by methods involving acidic hydrolysis or analysis of carbofuran and carbosulfan separately

‡ due to COVID-19 lockdown lab was not able to complete its submission, evaluation for informative purpose only

We assumed that they agreed with the reason provided by us: Analytical procedure was inappropriate (hydrolysis conditions too weak).

"Analytical procedure was inappropriate" was the most frequent reason for the poor performance (68 cases) in the present PT. It accounted for 49 out of 71 |z|>2 cased of c***arbofuran (sum)***, ***fluazifop (sum)***, ***MCPB (sum)*** and ***mecoprop (sum)***. It was often accompanied by the next frequent reason "lacking of experience" (25 cases). On the third place was "transcription error (21 cases)". Other often reported error sources were "measurement problems" (13x), "inappropriate/erroneous calibration approach" (9x), "calculation error" (9x), "erroneous analytical standard" (7x), as well as "result not properly corrected for recovery" (6x). In 5 cases the participants reported "Deficient QC-measures" as the reason for poor performance. "Analytical procedure was appropriate but it was not properly performed", "analyte losses during the procedure" and misinterpretation/misevaluation of measurement data" were responsible for each 2 cases. In one case the participant misunderstood the residue definition.

For the first time the organizers of the EUPT-SRMs did not provide the blank material that actually simulated the real lab routine. In 4 cases the participant found that the problem lay in the suppression effect of the blank rice used. Due to the different behaviour of the blank material and that of the test material, special attention has to be paid when using blank material for calibration or determination of recovery rate! And when every possible, using isotope labelled internal stands is the best choice to compensate the matrix effect.

4.4 Special Topic: Method-based Evaluation

On 6 March 2020, during the PT exercise, the EURL-SRM published a document focusing on the analysis of acidic pesticides involving hydrolysis for the cleavage of esters and conjugates[1]. This document summarized various hydrolysis experiments performed by the EURL-SRM (and presented in various EURL-workshops and trainings) for releasing esters and conjugates. The document highlighted, among others, that the cleavage of certain hydrolysis-resistant esters requires hydrolysis conditions stronger than those shown in the QuEChERS standard and that matrix also plays an important role. Certain types of commodities, including cereals, require stronger hydrolysis conditions than most fruits and vegetables. On 13 March the organizers, noticing that the participants were partly using insufficient hydrolysis approaches, decided to make them aware of the document concerning the analysis of acidic pesticides as well as of another document concerning the analysis of ***carbofuran (sum)***[2]. Unfortunately, in view of the announced corona virus lockdown measures, some laboratories had already completed their analyses and result-submission by this time, while others had already a restricted or no access to their labs.

Aiming to obtain an assigned value that is as close as possible to the true value, the participants' results were divided into sub-populations based on the hydrolysis conditions they have applied (see also **Section 4.2, p. 30**). For the various sub-populations, the robust mean and the relative standard deviation (CV^*) were calculated. As expected, the robust mean of sub-populations applying stronger hydrolysis conditions, thus ensuring nearly quantitative conversion rates, were overall closer to the expected (e.g. spiked) levels compared to sub-populations involving weak or no hydrolysis in their procedures.

[1] https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EurlSrm_Observation_alkaline_hydrolysis_acidic_herbicides.pdf

[2] https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EurlSrm_Observations_Carbofuran.pdf

4.4.1 Impact of hydrolysis conditions on the release of acidic ester- or glucoside-bound acidic pesticides

In order to check the degree of hydrolysis achieved for the various acidic pesticides contained in the sample when varying the hydrolysis conditions, the organizers decided to run some experiments using the following hydrolysis conditions:

a). **Mild: 0.3 ml NaOH** +10 mL water added to 5 g matrix; reaction at RT for:
i) 5 min; ii) 30 min and iii) 60 min

b). **Intermediate: 1 ml NaOH** + 10 mL water + 10 mL acetonitrile added to 5 g sample; reaction at 40 °C for
i) 30 min and ii) 120 min

c). **Strong: 2 ml NaOH** + 10 mL water + 10 mL acetonitrile added to 5 g sample; reaction at 40 °C for
i) 30 min and ii) 120 min

These experiments aimed at delivering the scientific evidence needed for facilitating decision-making on behalf of the EUPT-Scientific Committee, as regards the selection of sub-populations for the calculation of assigned values.

As shown in **Figure 4-1** mild extraction conditions were already sufficient for achieving satisfactory hydrolysis yields for ***MCPA*** (spiked as glucoside) and ***Quizalofop*** (spiked as propaquizafop). ***Fluazifop*** (spiked as fluazifop-butyl) and ***MCPB*** (spiked as methyl ester) required intermediate conditions, whereas ***mecoprop*** (spiked as MCPP-trimethylpentyl ester) required the harshest conditions.

The same trend was also observed in the participants' results when subdivided into groups, according on the hydrolysis conditions employed in each case. This is shown in **Table 4-11 (p. 50)**.

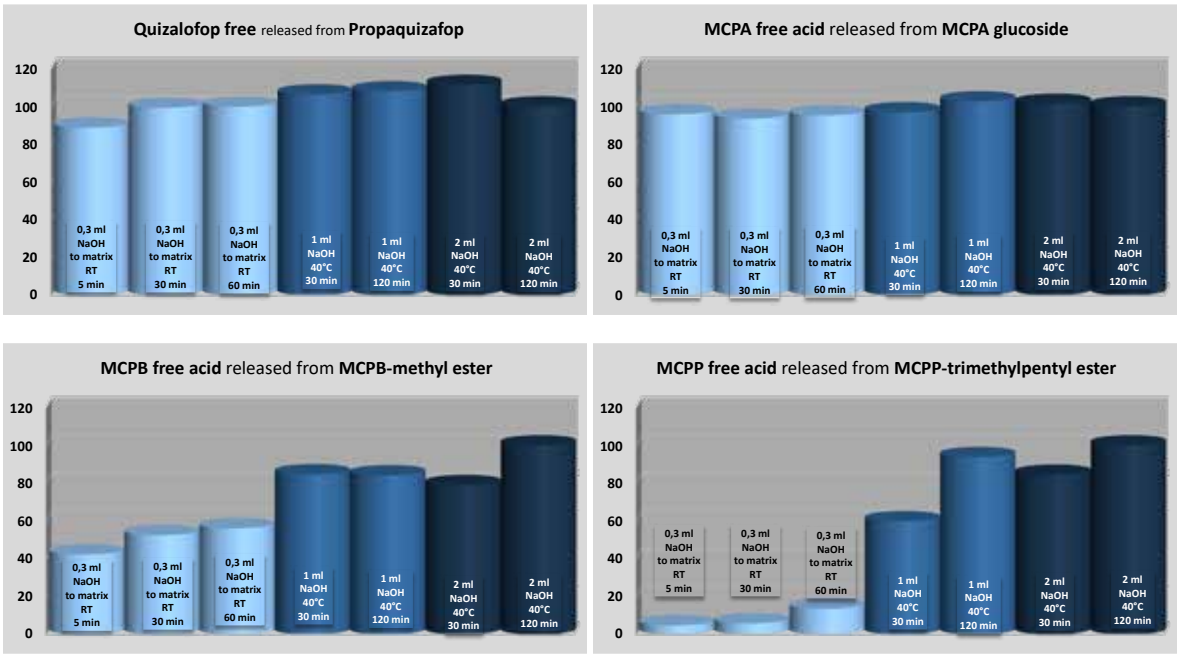


Figure 4-1: Impact of alkaline hydrolysis condition on the relative conversion yields. The yield obtained when applying the strongest conditions c-ii: "2 ml NaOH + 10 mL water + 10 mL acetonitrile, 120 min" was set at 100%

Table 4-11: Impact of hydrolysis condition on the distribution of results for fluazifop (sum), MCPB (sum) and mecoprop (sum) as well as their average bias (only results from EU and EFTA laboratories were taken into account)

	Fluazifop (sum)		
	Whole Pupolation	**Subpopulation with Hydrolysis (strong/moderate)**	**Subpopulation without Hydrolysis or with weak Hydrolysis**
No. of Numerical Results	64	42	22
Outlier thereof [1)]	0	0	0
No. of False Negative Results	2	0	2
Robust Mean [mg/kg]	**0.057**	**0.060**	**0.045**
CV*	**27.1 %**	**18.8 %**	**60.5 %**
AAZ [1)] (average bias in %)	**0.65**	**0.65**	**1.86**
No. (%) of acceptable results [1)]	54 (82 %)	40 (95 %)	11 (50 %)
No. (%) of questionable results [1)]	7 (11 %)	2 (5 %)	8 (36 %)
No. (%) of unacceptable results [1)]	5 (8 %)	0 (0 %)	3 (14 %)
	MCPB (sum)		
	Whole Population	**Subpopulation with Hydrolysis (strong/moderate)**	**Subpopulation without Hydrolysis or with weak Hydrolysis**
No. of Numerical Results	56	40	16
Outlier thereof	3	1	0
No. of False Negative Results	4	0	4
Robust Mean [mg/kg]	**0.050**	**0.057**	**0.039**
CV*	**38.8 %**	**29.4 %**	**53.5 %**
AAZ [1)] (average bias in %)	**1.51**	**1.14**	**1.61**
No. (%) of acceptable results [1)]	43 (72 %)	33 (55 %)	13 (22 %)
No. (%) of questionable results [1)]	10 (17 %)	4 (7 %)	1 (2 %)
No. (%) of unacceptable results [1)]	7 (12 %)	3 (5 %)	2 (3 %)
	Mecoprop (sum)		
	Whole Population	**Subpopulation with strong Hydrolysis**	**Subpopulation without Hydrolysis or with weak or moderate Hydrolysis**
No. of Numerical Results	41	20	21
Outlier thereof	2	1	0
No. of False Negative Results	18	1	17
Robust Mean [mg/kg]	**0.051**	**0.067**	**0.039**
CV*	**51.6 %**	**21.2 %**	**57.8 %**
AAZ [1)] (average bias in %)	**2.33**	**1.30**	**2.35**
No. (%) of acceptable results [1)]	28 (47 %)	16 (27 %)	13 (22 %)
No. (%) of questionable results [1)]	7 (12 %)	1 (2 %)	4 (7 %)
No. (%) of unacceptable results [1)]	24 (41 %)	3 (5 %)	4 (7 %)

1) calculated using the corresponding population

Following consultations with the EUPT-Scientific Committee, it was finally decided to calculate the assigned values of the acidic pesticides, that were spiked in ester or conjugated form, using the following sub-populations of results:

a). ***MCPA (sum)*** and ***quizalofop (sum)***: entire population
b). ***Fluazifop (sum)*** and ***MCPB (sum)***: sub-population of results generated by labs using intermediate or strong hydrolysis conditions
c). ***Mecoprop (sum)***: sub-population of results generated by labs using strong hydrolysis conditions

By reducing the populations, the distribution of the results, expressed as CV^*, improved significantly. For example from 27.5 % to 18.8% in the case of ***fluazifop (sum)*** and from 38.8 % to 29.4 % in the case of ***MCPB (sum)***. The remaining results, which were generated by methods involving weak or no hydrolysis, showed a $CV^* > 50$ % for both analytes.

4.4.2 Carbofuran (sum)

Looking at the participants' results for ***carbofuran (sum)*** a non-unimodal distribution was visible. Looking at the hydrolysis conditions it became clear that the labs not employing hydrolysis submitted strongly biased results. This is shown in **Figure 4-2**

The assigned value of ***carbofuran (sum)*** in the preliminary report was calculated using results generated by methods involving acidic hydrolysis only, although the organizer was well aware of the fact that results from laboratories having analyzed carbofuran and carbosulfan separately could also have been added to this group. But as carbosulfan analysis is quite challenging and error-prone, results driven from this analytical approach were firstly not included. After releasing the Preliminary Report, the organizer started a survey on detailed information about the analytical method for ***carbofuran (sum)*** . Based on the survey results and after consultation with the Scientific Committee, the assigned value for ***carbofuran (sum)*** was finally established as the robust mean of the results generated by labs applying hydrolysis or by labs analysing carbofuran and carbosulfan separately and adding up the result. See also **Section 4.2 (p. 30)**

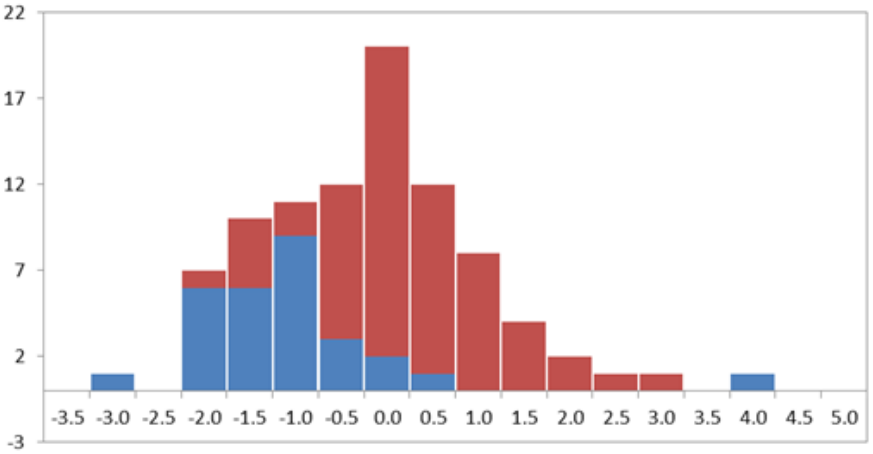


Figure 4-2: Sub-division of results for carbofuran (sum) based on methodologies used. Red: labs applying hydrolysis OR not applying hydrolysis but analyzing CF+CS separately and summing up. Blue: rest of the population

Table 4-12: Results of carbofuran (sum) from laboratories analysing carbofuran and carbosulfan separately and not correctly converted into carbofuran (sum)

	Reported by Laboratories			Corrected Value
Lab-Code SRM15	Carbofuran (CF)	Carbosulfan (CS)	Sum of CF and CS	Sum of CF and CS
26	0.066	0.053	0.119	0.097
30	0.099	0.01	0.099	0.105
50	0.096	0.0033	0.101	0.098
51	0.069	n.n.	0.061	0.069
88	0.05	0.01	0.050	0.056
95	0.039	0.033	0.071	0.058
99	0.01	0.08	0.06	0.057
118	0.085	0.065	0.150	0.123

* Sum of CF and CS = Carbofuran + (Carbosulfan/1.72)

In the feedback on the survey from the laboratories, the organizers found 8 results that were generated by adding up carbofuran and carbosulfan results, but using incorrect conversion factors (Table 4-12). In consultation with the Scientific Committee, the organizers have therefore corrected those results and proceeded with the calculation of the assigned value, CV* and labs z-scores, again using the corrected data . This evaluation is shown in **Table 4-7 (p. 35),** but for informative purpose only.

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 the EDV-Team at DTU: Anne-Mette Skovlund, Steen Maigaard, Sean Gomes, Nicolaj Graversen Pedersen and Wardan Ghazal, for the development and establishment of the online result submission tool as well as for the support during and after PT period.

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-SRM15

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

Country (Location)	Analysed on behalf of	Institution	City	NRL*
Austria	AT	Department for Pesticide and Food Analytics (PLMA)	Innsbruck	x
Belgium	BE; BG; FR; LU	PRIMORIS (Phytolab) - Belgium	Gent - Zwijnaarde	–
Belgium	BE	Sciensano - Pesticide Lab	Brussels	x
Bulgaria	BG	CLCTC - Sofia | Pesticide Lab	Sofia	x
Croatia	HR	Bioinstitut d.o.o., Cakovec	Cakovec	–
Croatia	HR	Croatiakontrola - Pesticide Lab	Zagreb	–
Croatia	HR	Dr. Andrija Štampar - Pesticide Lab	Zagreb	x
Croatia	HR	INSPECTO d.o.o. Laboratorij (Osijek)	Osijek	–
Croatia	HR	Sample Control - Pesticide Lab	Lučko	–
Cyprus	CY	SGL - Pesticide Lab (Nicosia)	Nicosia	x
Czech Republic	CZ	CAFIA - Pesticide Lab (Praha)	Praha	x
Czech Republic	CZ	Pesticide Lab (Brno)	Brno	–
Czech Republic	CZ	VSCHT / UCT Prague - Food Analysis (323)	Praha	–
Denmark	DK	Laboratoriet Ringsted - Pesticide Lab	Ringsted	x
Estonia	EE	Agricultural Research Center - Estonia, Saku	Saku	–
Estonia	EE	Tartu Laboratory of Health Board	Tartu	x
Finland	FI	Finnish Customs Laboratory	Espoo	x
Finland	FI	Finnish Food Authority	Helsinki	x
France	BE	Phytocontrol (Nimes) - Pesticide Lab	Nimes	–
France	FR	ANSES - LSAI (Unité PBM)	MAISONS-ALFORT Cedex	x
France	FR	CAMP Méditerrannée (Perpignan)	PERPIGNAN	–
France	FR	CAPINOV (Landerneau)	Landerneau	–
France	FR	CERECO (GARONS)	GARONS	–
France	FR	GIRPA-POLLENIZ - Pesticide Lab	Beaucouzé	–
France	FR	INOVALYS Le Mans - Pesticide Lab	Le Mans	–
France	FR	SCL - Massy Cedex	Massy Cedex	–
France	FR	SCL (Montpellier)	Montpellier	–
Germany	DE	BVL Unit 504 NRL for Pesticide Residues	Berlin	x
Germany	DE	CVUA RRW - Pesticide Lab (Krefeld)	Krefeld	–
Germany	DE	KWALIS Fulda - Pesticide Lab	Dipperz	–
Germany	DE	Labor Friedle - Germany, Tegernheim	Tegernheim	–
Germany	DE	LALLF - Pesticide Lab (Rostock)	Rostock	–
Germany	DE	Landesamt für Verbraucherschutz, Halle/Saale	Halle/Saale	–
Germany	DE	Landeslabor Berlin-Brandenburg, Potsdam	Potsdam	–
Germany	DE	Landeslabor Schleswig-Holstein, Neumünster	Neumünster	–
Germany	DE	LAVES - Pesticide Lab (Oldenburg)	Oldenburg	–
Germany	DE	LAVES - Pesticide Lab (Stade)	Stade	–
Germany	DE	LGL Erlangen - Pesticide Lab	Erlangen	–
Germany	DE	LHL - Pesticide Lab (Kassel)	Kassel	–
Germany	DE	LLG - Pesticide Lab	Halle/Saale	–
Germany	DE	LTZ Augustenberg - Organic Analysis	Karlsruhe	–
Germany	DE	LUA Rheinland-Pfalz, Institut für LM-Chemie Speyer	Speyer	–
Germany	DE	LUA Sachsen - Pesticide Lab	Dresden	–
Germany	DE	LUFA Speyer	Speyer	–

* 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*
Germany	DE	Pesticide Lab (Nossen)	Nossen	–
Germany	DE	Thüringer Landesanstalt für Landwirtschaft, Jena	Jena	–
Germany	BE	LUFA Kiel - Pesticide Lab	Kiel	–
Germany	FR	Intertek Food Services - Bremen	Bremen	–
Germany	LT	GALAB Laboratories GmbH - Hamburg	Hamburg	–
Germany	MT	Eurofins - Germany, Hamburg, Großmoorbogen	Hamburg	–
Germany	MT	Eurofins Dr.Specht Express GmbH - Hamburg	Hamburg	–
Greece	GR	Benaki Phytopathological Institute, Kifissia	Kifissia	x
Greece	GR	GCSL - Pesticide Lab (Athens)	Athens	x
Hungary	HU	NFCSO - Pesticide Lab (Velence)	Velence	x
Hungary	HU	NFCSO Pesticide Lab (Hódmezovásárhely)	Hódmezovásárhely	–
Hungary	HU	NFCSO Pesticide Lab (Miskolc)	Miskolc	–
Hungary	HU	NFCSO Pesticide Lab (Szolnok)	Szolnok	
Iceland	IS	Matís - Iceland, Reykjavík	Reykjavík	–
Ireland	IE	Pesticide Control Lab - Ireland, Co. Kildare	Co. Kildare	x
Italy	IT	APPA Bolzano - Pesticide Lab	Bolzano	–
Italy	IT	APPA-Puglia | Polo Alimenti Bari - Pesticide Lab	Bari	–
Italy	IT	ARPA Veneto (Laboratorio di Verona)	Verona	–
Italy	IT	ASF - Pesticide Lab	Firenze	–
Italy	IT	ISS - Pesticide Lab	Roma	x
Italy	IT	IZS LT - Italy, Rome	Roma	–
Italy	IT	IZSAM - Pesticide Lab	Teramo	–
Italy	IT	IZSLER - Pesticide Lab	Brescia	–
Italy	IT	Laboratorio di Prevenzione (Bergamo)	Bergamo	–
Latvia	LV	BIOR (Riga) - Pesticide Lab	Riga	x
Lithuania	LT	NMVRVI - Pesticide Lab (Vilnius)	Vilnius	x
Luxembourg	LU	LNS Food lab	Dudelange	x
Norway	NO	NIBIO - Department of Pesticide Chemistry	ÅS	
Poland	PL	InHort (Skierniewice) - Pesticide Lab	Skierniewice	–
Poland	PL	IPP-NRI - Pesticide Lab (Poznan)	Poznan	–
Poland	PL	SGS Sp. z o.o. Laboratorium Srodowiskowe	pszczyna	–
Poland	PL	UO-Technologia (Grojec) - Pesticide Lab	Grojec	–
Poland	PL	VSES Warszawa - Pesticide Lab	Warszaw	x
Portugal	PT	Labiagro – Laboratório Químico	Oeiras - Lisboa	–
Portugal	PT	Pesticide Lab (Funchal - Madeira Island)	Funchal - Madeira Island	x
Romania	RO	IISPV (Bucharest) - Pesticide Lab	Bucharest	x
Romania	RO	LRCRPPPV (Tirgu Mures) - Pesticide Lab	Tirgu Mures	–
Slovakia	SK	State Veterinary and Food Institute (Bratislava)	Bratislava	x
Slovenia	SI	Nat. Lab for Health, Environment and Food, Maribor	Ljubljana	–
Slovenia	SI	Pesticide Lab - Maribor	Maribor	x
Spain	ES	Ainia (Valencia)	Valencia	–
Spain	ES	Analytica Alimentaria GmbH - Almeria, Spain	Almeria	–
Spain	ES	EUROFINS ECOSUR - Pesticide Lab	Lorquí	–
Spain	ES	Lab. Agrario Regional - Junta de Castilla y Leon	Burgos	–
Spain	ES	Laboratori Agència Salut Pública Barcelona	Barcelona	–
Spain	ES	Laboratorio Agroalimentario - Spain, Valencia	Valencia	–
Spain	ES	Laboratorio Agroalimentario de Extremadura	Cáceres	–
Spain	ES	Laboratorio Agroambiental de Zaragoza	Zaragoza	–

* 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*
Spain	ES	Laboratorio Analítico Bioclínico - Spain, Almeria	Almeria	–
Spain	ES	Laboratorio Arbitral Agroalimentario, Madrid	Madrid	x
Spain	ES	Laboratorio de Salud Pública de Galicia, Lugo	Lugo	–
Spain	ES	LABORATORIO KUDAM, S.L.	Pilar de la Horadada (Alicante)	–
Spain	ES	LAC - Generalitat de Catalunya	Cabrils	–
Spain	ES	National Center for Technology and Food Safety	San Adrián (Navarra)	–
Spain	ES	National Centre for Food (Majadahonda)	Majadahonda	x
Spain	PT	Labs & Technological Services AGQ - Burguillos	Burguillos	–
Sweden	SE	Eurofins Food & Feed - Pesticide Lab (Lidköping)	Lidköping	–
Sweden	SE	National Food Agency - Sweden, Uppsala	Uppsala	x
The Netherlands	NL	WFSR - NRL for Pesticides	Wageningen	x
The Netherlands	BE	Groen Agro Control - Netherlands	Delfgauw	–
The Netherlands	BE	Handelslaboratorium Dr. Verwey - Pesticide Lab	Rotterdam	–
The Netherlands	BE	NofaLab - Pesticide Lab	Schiedam	–
The Netherlands	BE; NL	Eurofins Lab Zeeuws-Vlaanderen B.V. - Pesticiden	Graauw	–
United Kingdom	UK	Concept Life Sciences - United Kingdom, Cambridge	Bar Hill	–
United Kingdom	UK; MT	FERA - Pesticide Lab	York	x

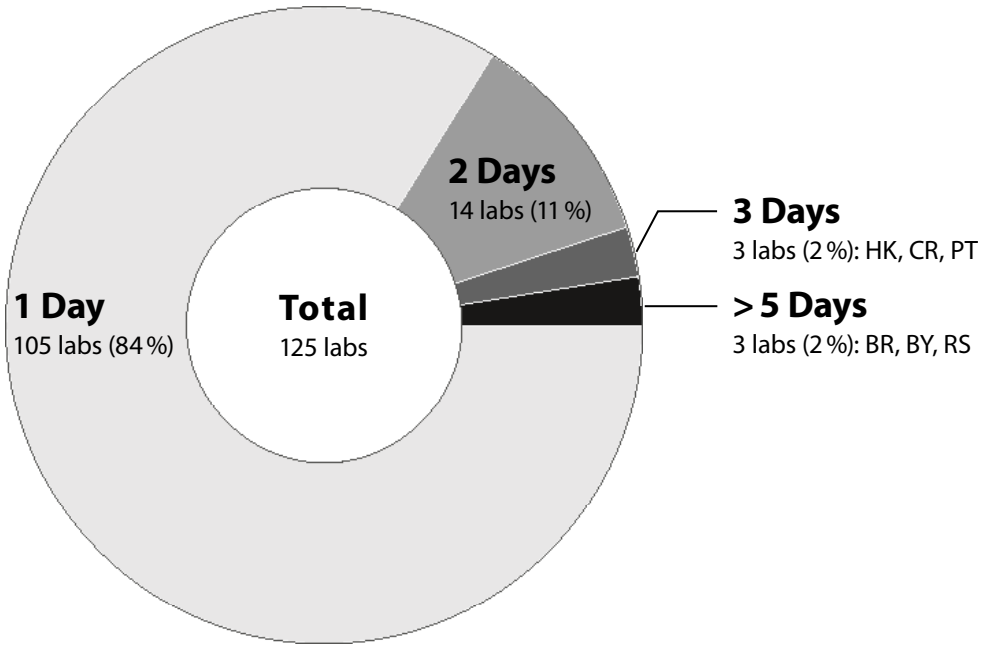
* only for EU-Member States

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

Country	Institution	City
Belarus	Pesticide Lab (Minsk)	Minsk
Brazil	MAPA - Pesticide Lab - Brazil, Pedro Leopoldo	Pedro Leopoldo - MG
China (Hong Kong)	Government Laboratory (Hong Kong)	Hong Kong
Costa Rica	SFE - Pesticide Lab (San Jose)	San Jose
Serbia	Inst. of Public Health of Belgrade - Pesticide Lab	Belgrade
Serbia	SP Laboratorija - Pesticide Lab	BECEJ
Singapore	SINGAPORE FOOD AGENCY - Pesticide Lab	Singapore
Thailand	Central Laboratory - Pesticide Lab (Bangkok)	Bangkok

Appendix 2 Shipment Evaluation

Compilation of shipment duration



Appendix 3 Data of Homogeneity Test

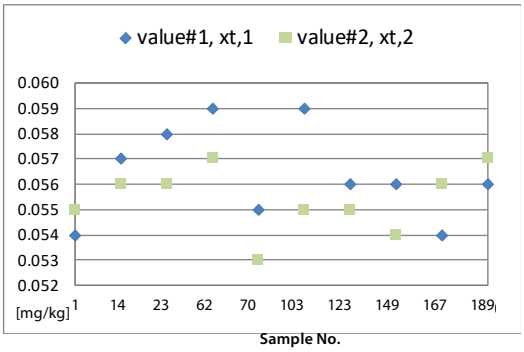
Compulsory Compounds

2,4-D (free acid)			Carbofuran (sum)			Chlormequat-Cl			Glyphosate		
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]
1	0.054	0.055	1	0.101	0.095	1	0.084	0.085	1	0.201	0.199
14	0.057	0.056	14	0.098	0.090	14	0.080	0.083	14	0.199	0.200
23	0.058	0.056	23	0.100	0.098	23	0.083	0.084	23	0.200	0.195
62	0.059	0.057	62	0.097	0.100	62	0.083	0.084	62	0.213	0.200
70	0.055	0.053	70	0.097	0.095	70	0.082	0.086	70	0.207	0.201
103	0.059	0.055	103	0.097	0.098	103	0.083	0.082	103	0.204	0.193
123	0.056	0.055	123	0.092	0.100	123	0.080	0.082	123	0.209	0.202
149	0.056	0.054	149	0.097	0.098	149	0.083	0.084	149	0.198	0.206
167	0.054	0.056	167	0.097	0.100	167	0.083	0.084	167	0.190	0.204
189	0.056	0.057	189	0.099	0.099	189	0.084	0.085	189	0.199	0.214
mean / AV*	0.056 / 0.052		mean / AV*	0.097 / 0.107‡		mean / AV*	0.083 / 0.092		mean / AV*	0.202 / 0.203	

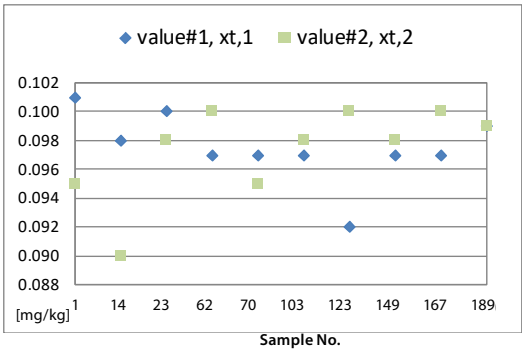
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories
‡ AV based on subpopulation of results generated by methods including hydrolysation or analysis of carbofuran and carbosulfan separately only

Graphical presentation of the results:

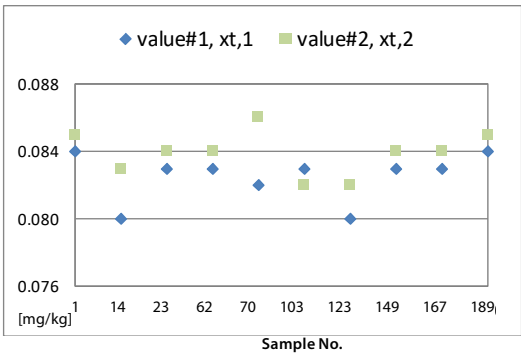
2,4-D (free Acid)



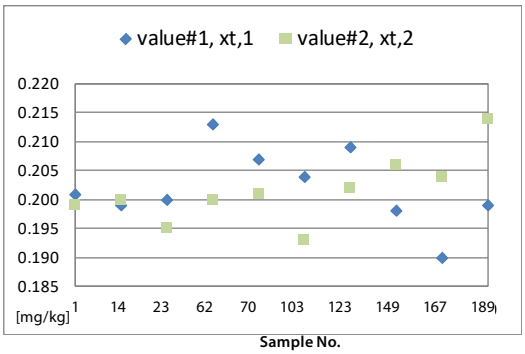
Carbofuran (sum)



Chlormequat-Cl



Glyphosate



Appendix 3 (cont.): Data of Homogeneity Test

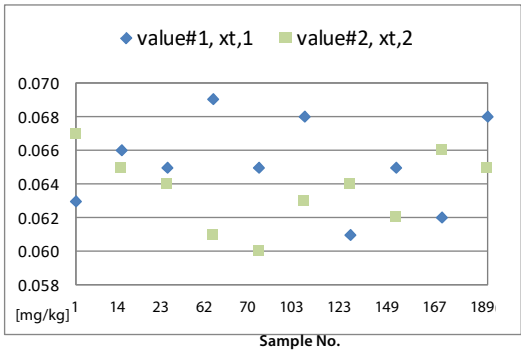
Compulsory Comp. | **Optional Compounds**

TFNA			2,4-D (sum)			Bentazone (free acid)			Fluazifop (sum)		
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]
1	0.063	0.067	1	0.057	0.055	1	0.337	0.345	1	0.068	0.069
14	0.066	0.065	14	0.056	0.053	14	0.333	0.334	14	0.067	0.069
23	0.065	0.064	23	0.059	0.055	23	0.343	0.329	23	0.064	0.069
62	0.069	0.061	62	0.055	0.054	62	0.329	0.332	62	0.067	0.068
70	0.065	0.060	70	0.057	0.056	70	0.344	0.334	70	0.068	0.069
103	0.068	0.063	103	0.056	0.054	103	0.339	0.342	103	0.069	0.066
123	0.061	0.064	123	0.057	0.056	123	0.340	0.342	123	0.066	0.068
149	0.065	0.062	149	0.051	0.054	149	0.339	0.338	149	0.062	0.067
167	0.062	0.066	167	0.053	0.056	167	0.340	0.351	167	0.067	0.066
189	0.068	0.065	189	0.054	0.054	189	0.342	0.341	189	0.065	0.066
mean / AV*	0.064 / 0.060		mean / AV*	0.055 / 0.059		mean / AV*	0.339 / 0.334		mean / AV*	0.067 / 0.060‡	

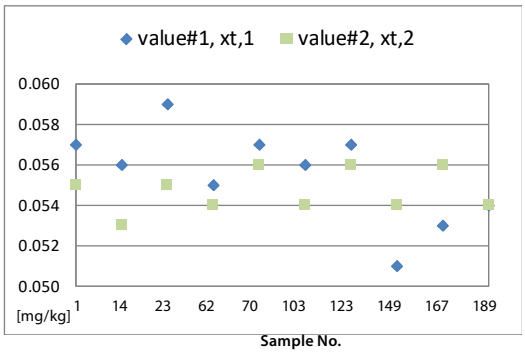
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories
‡ AV based on subpopulation of results generated by methods including moderate and strong hydrolysis only

Graphical presentation of the results:

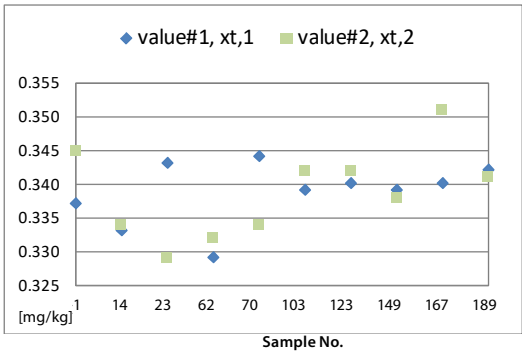
TFNA



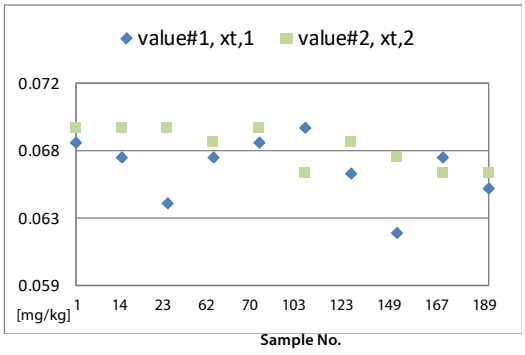
2,4-D (sum)



Bentazone (free acid)



Fluazifop (sum)



Appendix 3 (cont.): Data of Homogeneity Test

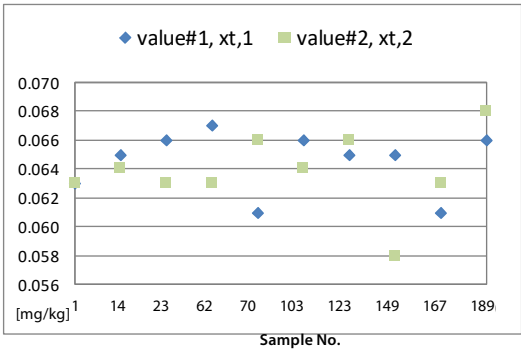
Optional Compounds

Haloxyfop (sum)			Imazethapyr (free acid)			MCPA (sum)			MCPB (sum)		
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]
1	0.159	0.160	1	0.211	0.212	1	0.063	0.063	1	0.057	0.058
14	0.153	0.158	14	0.222	0.221	14	0.065	0.064	14	0.054	0.057
23	0.151	0.156	23	0.218	0.206	23	0.066	0.063	23	0.059	0.057
62	0.159	0.160	62	0.219	0.219	62	0.067	0.063	62	0.061	0.058
70	0.152	0.147	70	0.214	0.214	70	0.061	0.066	70	0.056	0.059
103	0.154	0.153	103	0.231	0.215	103	0.066	0.064	103	0.060	0.057
123	0.160	0.152	123	0.208	0.211	123	0.065	0.066	123	0.059	0.061
149	0.150	0.152	149	0.224	0.212	149	0.065	0.058	149	0.060	0.055
167	0.160	0.147	167	0.211	0.219	167	0.061	0.063	167	0.055	0.060
189	0.151	0.155	189	0.222	0.219	189	0.066	0.068	189	0.057	0.058
mean / AV*	0.154 / 0.151		mean / AV*	0.216 / 0.206		mean / AV*	0.064 / 0.068		mean / AV*	0.058 / 0.057‡	

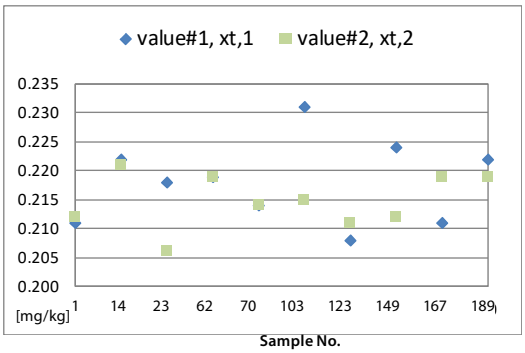
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories
‡ AV based on subpopulation of results generated by methods including moderate and strong hydrolysis only

Graphical presentation of the results:

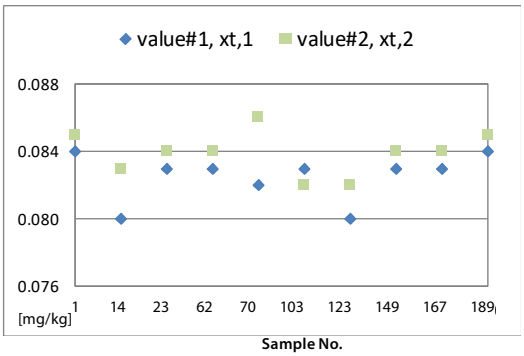
Haloxyfop (sum)



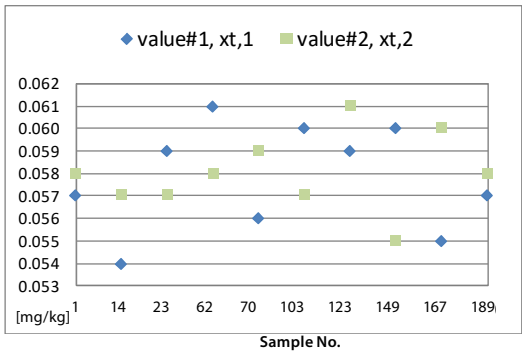
Imazethapyr (free acid)



MCPA (sum)



MCPB (sum)



Appendix 3 (cont.): Data of Homogeneity Test

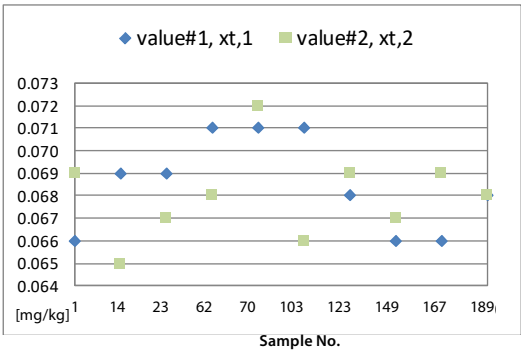
Optional Compounds

MCPP (sum)			Paraquat (dication)			Quizalofop (free acid)			Quizalofop (sum)		
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]
1	0.066	0.069	1	0.229	0.230	1	0.045	0.045	1	0.065	0.060
14	0.069	0.065	14	0.227	0.226	14	0.047	0.046	14	0.065	0.061
23	0.069	0.067	23	0.217	0.229	23	0.049	0.045	23	0.065	0.061
62	0.071	0.068	62	0.238	0.229	62	0.047	0.047	62	0.064	0.064
70	0.071	0.072	70	0.238	0.244	70	0.047	0.043	70	0.061	0.061
103	0.071	0.066	103	0.241	0.241	103	0.048	0.047	103	0.063	0.063
123	0.068	0.069	123	0.226	0.236	123	0.046	0.046	123	0.064	0.061
149	0.066	0.067	149	0.240	0.227	149	0.047	0.046	149	0.060	0.063
167	0.066	0.069	167	0.234	0.241	167	0.044	0.047	167	0.065	0.064
189	0.068	0.068	189	0.236	0.229	189	0.049	0.046	189	0.062	0.064
mean / AV*	0.068 / 0.067‡		mean / AV*	0.233 / 0.195		mean / AV*	0.046 / 0.044		mean / AV*	0.063 / 0.062	

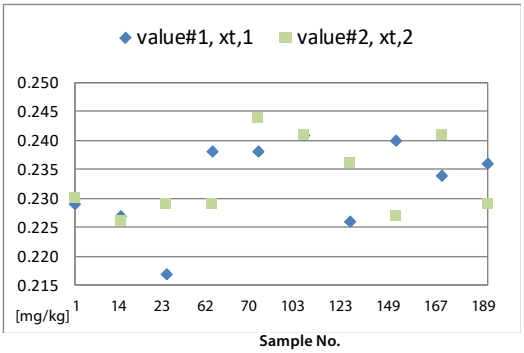
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories
‡ AV based on subpopulation of results generated by methods including strong hydrolysis only

Graphical presentation of the results:

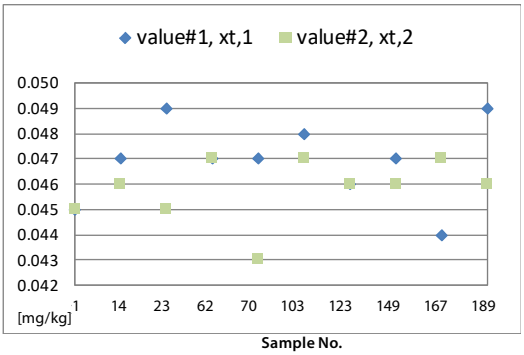
MCPP (sum)



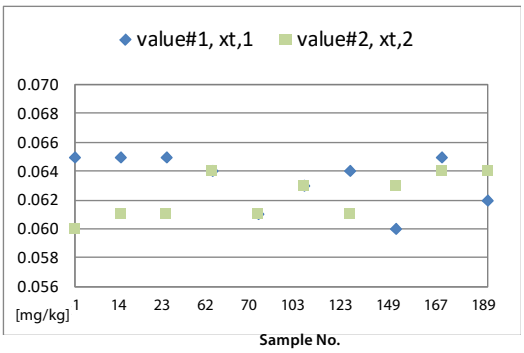
Paraquat (dication)



Quizalofop (free acid)



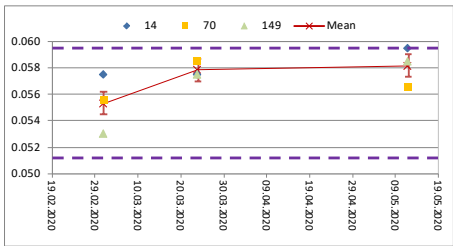
Quizalofop (sum)



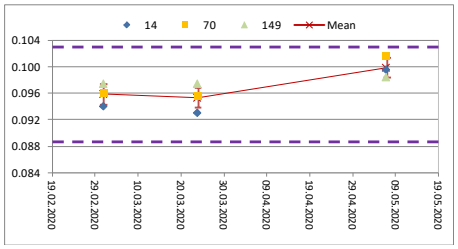
Appendix 4 Data of Stability Test

Compulsory Compounds												
2,4-D (free acid)							**Carbofuran (sum)**					
AV [mg/kg]	0.052						AV [mg/kg]	0.107‡				
Date	02.03.2020		24.03.2020		07.05.2020		Date	02.03.2020		24.03.2020		07.05.2020
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 014	0.059	0.060	0.053	0.056	0.055	0.056	No. 014	0.098	0.090	0.093	0.093	0.098	0.101
No. 070	0.059	0.059	0.057	0.057	0.056	0.055	No. 070	0.097	0.095	0.095	0.096	0.100	0.103
No. 149	0.057	0.058	0.055	0.056	0.055	0.057	No. 149	0.097	0.098	0.098	0.097	0.098	0.099
Mean [mg/kg]	**0.059**		**0.056**		**0.056**		**Mean** [mg/kg]	**0.096**		**0.095**		**0.100**	
RSD* [%]	**1.8%**		**2.3%**		**0.5%**		**RSD*** [%]	**1.8%**		**2.4%**		**1.5%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-5.1%**		**-5.1%**			**—**		**-0.5%**		**4.2%**	
							‡ AV based on subpopulation of results generated by methods including hydrolysation or analysis carbofuran and carbosulfan separately only					

2,4-D (free acid)



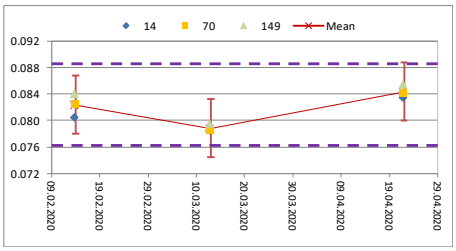
Carbofuran (sum)



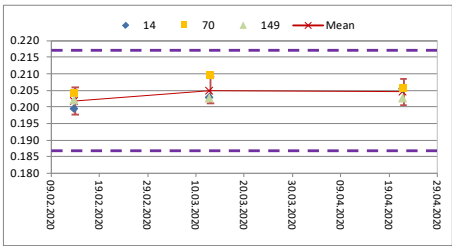
– – – : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

Compulsory Compounds												
Chlormequat-Cl							**Glyphosate**					
AV [mg/kg]	0.092						AV [mg/kg]	0.203				
Date	14.02.2020		13.03.2020		22.04.2020		Date	14.02.2020		13.03.2020		22.04.2020	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 014	0.083	0.078	0.082	0.075	0.087	0.080	No. 014	0.199	0.200	0.200	0.206	0.203	0.208
No. 070	0.085	0.080	0.082	0.075	0.082	0.086	No. 070	0.207	0.201	0.217	0.202	0.210	0.201
No. 149	0.084	0.084	0.077	0.082	0.083	0.088	No. 149	0.198	0.206	0.194	0.211	0.205	0.200
Mean [mg/kg]	**0.082**		**0.079**		**0.084**		**Mean** [mg/kg]	**0.202**		**0.205**		**0.205**	
RSD* [%]	**2.1%**		**0.7%**		**1.2%**		**RSD*** [%]	**1.1%**		**1.9%**		**0.8%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-4.3%**		**2.4%**			**—**		**1.6%**		**1.3%**	

Chlormequat-Cl



Glyphosate



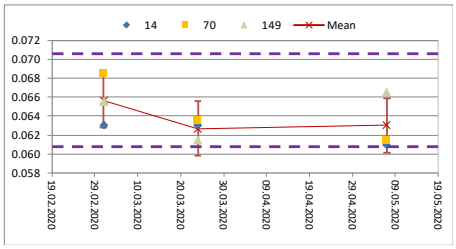
– – – : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

* RSD = relative standard diviation

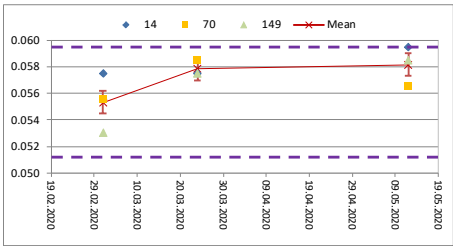
Appendix 4 (cont.): Data of Stability Test

Compulsory Compound							Optional Compound						
TFNA							2,4-D (sum)						
AV [mg/kg]	0.060						AV [mg/kg]	0.059					
Date	02.03.2020		24.03.2020		07.05.2020		Date	02.03.2020		24.03.2020		12.05.2020	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 014	0.063	0.063	0.062	0.064	0.060	0.062	No. 014	0.058	0.057	0.057	0.058	0.058	0.061
No. 070	0.070	0.067	0.063	0.064	0.062	0.061	No. 070	0.056	0.055	0.060	0.057	0.057	0.056
No. 149	0.065	0.066	0.063	0.060	0.065	0.068	No. 149	0.053	0.053	0.056	0.059	0.057	0.060
Mean [mg/kg]	**0.066**		**0.063**		**0.063**		**Mean** [mg/kg]	**0.055**		**0.058**		**0.058**	
RSD* [%]	**4.2%**		**1.7%**		**4.8%**		**RSD*** [%]	**4.1%**		**1.0%**		**2.6%**	
Diviation [%] (ref. 1st Anaylsis)	—		**-4.6%**		**-4.1%**			—		**4.5%**		**5.1%**	

TFNA



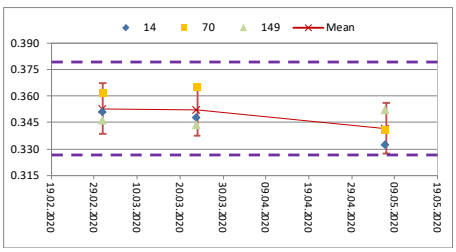
2,4-D (sum)



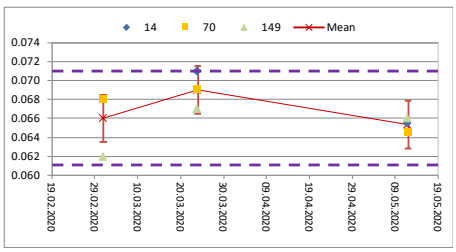
- - - : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

Optional Compounds													
Bentazone (free acid)							Fluazifop (sum)						
AV [mg/kg]	0.334						AV [mg/kg]	0.060‡					
Date	02.03.2020		24.03.2020		07.05.2020		Date	02.03.2020		24.03.2020		12.05.2020	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 014	0.349	0.353	0.346	0.350	0.325	0.340	No. 014	0.067	0.069	0.070	0.072	0.063	0.068
No. 070	0.363	0.359	0.369	0.361	0.340	0.341	No. 070	0.064	0.064	0.072	0.068	0.063	0.066
No. 149	0.343	0.350	0.346	0.341	0.346	0.358	No. 149	0.063	0.063	0.069	0.065	0.067	0.065
Mean [mg/kg]	**0.353**		**0.352**		**0.342**		**Mean** [mg/kg]	**0.066**		**0.069**		**0.065**	
RSD* [%]	**2.1%**		**3.2%**		**2.9%**		**RSD*** [%]	**5.2%**		**2.9%**		**1.2%**	
Diviation [%] (ref. 1st Anaylsis)	—		**-0.2%**		**-3.2%**			—		**4.5%**		**-1.0%**	
							‡ AV based on subpopulation of results generated by methods including strong or moderate hydrolysation only						

Bentazone (free acid)



Fluazifop (sum)



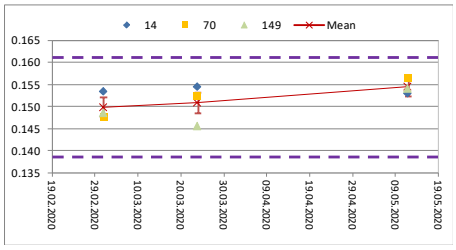
- - - : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

* RSD = relative standard diviation

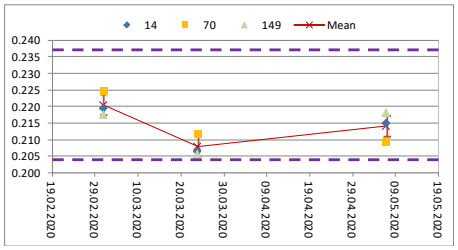
Appendix 4 (cont.): Data of Stability Test

Optional Compounds												
Haloxyfop (sum)							**Imazethapyr (free acid)**					
AV [mg/kg]	0.151						AV [mg/kg]	0.206				
Date	02.03.2020		24.03.2020		12.05.2020		Date	02.03.2020		24.03.2020		07.05.2020
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]
No. 014	0.157	0.150	0.152	0.157	0.152	0.154	No. 014	0.216	0.223	0.200	0.213	0.212	0.218
No. 070	0.153	0.142	0.158	0.147	0.157	0.156	No. 070	0.227	0.222	0.213	0.210	0.215	0.203
No. 149	0.148	0.149	0.136	0.155	0.149	0.159	No. 149	0.215	0.220	0.207	0.204	0.214	0.222
Mean [mg/kg]	**0.150**		**0.151**		**0.155**		**Mean** [mg/kg]	**0.221**		**0.208**		**0.214**	
RSD* [%]	**2.1%**		**3.1%**		**1.2%**		**RSD*** [%]	**1.6%**		**1.5%**		**2.1%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**0.7%**		**3.1%**			**—**		**-5.7%**		**-2.9%**	

Haloxyfop (sum)



Imazethapyr (free acid)

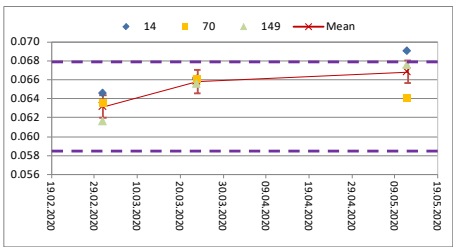


– – – : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

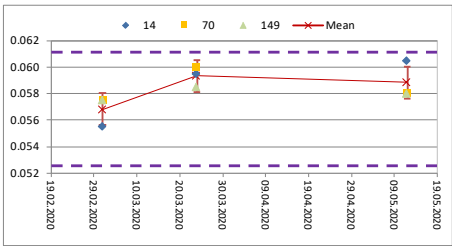
Optional Compounds												
MCPA (sum)							**MCPB (sum)**					
AV [mg/kg]	0.068						AV [mg/kg]	0.057‡				
Date	02.03.2020		24.03.2020		12.05.2020		Date	02.03.2020		24.03.2020		12.05.2020
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]
No. 014	0.065	0.064	0.065	0.067	0.067	0.071	No. 014	0.054	0.057	0.057	0.062	0.059	0.062
No. 070	0.061	0.066	0.066	0.066	0.063	0.065	No. 070	0.056	0.059	0.060	0.060	0.056	0.060
No. 149	0.065	0.058	0.065	0.066	0.067	0.068	No. 149	0.060	0.055	0.057	0.060	0.056	0.060
Mean [mg/kg]	**0.063**		**0.066**		**0.067**		**Mean** [mg/kg]	**0.057**		**0.059**		**0.059**	
RSD* [%]	**2.4%**		**0.4%**		**3.8%**		**RSD*** [%]	**2.0%**		**1.3%**		**2.5%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**4.2%**		**5.8%**			**—**		**4.4%**		**3.5%**	

‡ AV based on subpopulation of results generated by methods including strong or moderate hydrolysation only

MCPA (sum)



MCPB (sum)



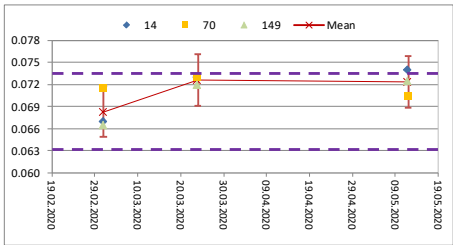
– – – : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

* RSD = relative standard diviation

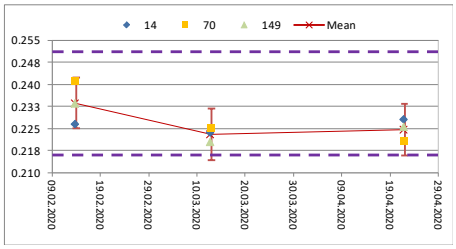
Appendix 4 (cont.): Data of Stability Test

Optional Compounds												
MCPP (sum)							**Paraquat (dication)**						
AV [mg/kg]	0.067‡						AV [mg/kg]	0.195					
Date	02.03.2020		24.03.2020		12.05.2020		Date	14.02.2020		13.03.2020		22.04.2020	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 014	0.069	0.065	0.071	0.075	0.071	0.077	No. 014	0.227	0.226	0.226	0.221	0.230	0.226
No. 070	0.071	0.072	0.075	0.071	0.068	0.073	No. 070	0.238	0.244	0.226	0.224	0.229	0.212
No. 149	0.066	0.067	0.073	0.071	0.070	0.075	No. 149	0.240	0.227	0.209	0.232	0.224	0.227
Mean [mg/kg]	**0.068**		**0.073**		**0.072**		**Mean** [mg/kg]	**0.234**		**0.223**		**0.225**	
RSD* [%]	**4.0%**		**0.8%**		**2.4%**		**RSD*** [%]	**3.1%**		**1.0%**		**1.7%**	
Diviation [%] (ref. 1st Anaylsis)	—		**6.3%**		**5.9%**			—		**-4.6%**		**-3.9%**	
‡ AV based on subpopulation of results generated by methods including strong hydrolysation only													

MCPP (sum)



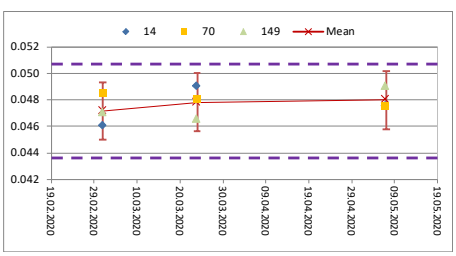
Paraquat (dication)



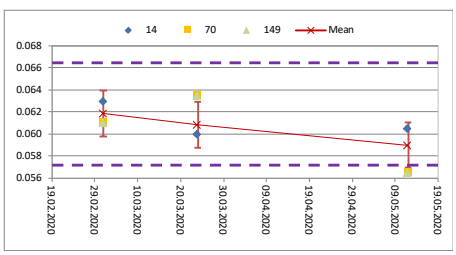
– – – : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

Optional Compounds												
Quizalofop (free acid)							**Quizalofop (sum)**						
AV [mg/kg]	0.044						AV [mg/kg]	0.062					
Date	02.03.2020		24.03.2020		07.05.2020		Date	02.03.2020		24.03.2020		12.05.2020	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 014	0.045	0.047	0.049	0.049	0.047	0.048	No. 014	0.065	0.061	0.060	0.060	0.057	0.064
No. 070	0.049	0.048	0.048	0.048	0.048	0.047	No. 070	0.061	0.061	0.063	0.064	0.054	0.059
No. 149	0.046	0.048	0.046	0.047	0.048	0.050	No. 149	0.060	0.063	0.058	0.060	0.058	0.062
Mean [mg/kg]	**0.047**		**0.048**		**0.048**		**Mean** [mg/kg]	**0.062**		**0.061**		**0.059**	
RSD* [%]	**2.7%**		**2.6%**		**1.8%**		**RSD*** [%]	**1.7%**		**3.9%**		**3.7%**	
Diviation [%] (ref. 1st Anaylsis)	—		**1.4%**		**1.8%**			—		**-1.6%**		**-4.6%**	

Quizalofop (free acid)



Quizalofop (sum)



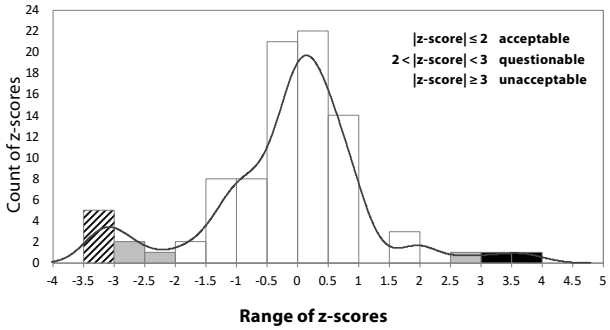
– – – : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

* RSD = relative standard diviation

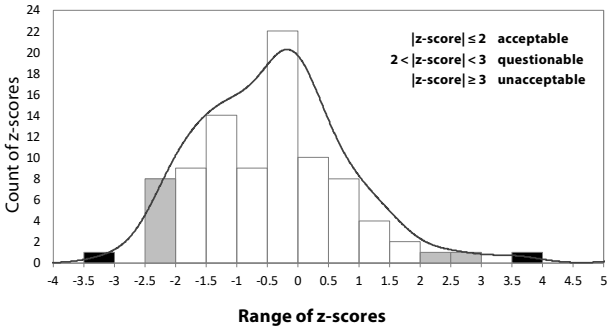
Appendix 5 Histograms and Kernel Density Estimates of z-score* Distributions
(Results from EU and EFTA Laboratories only)

Compulsory Compounds

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

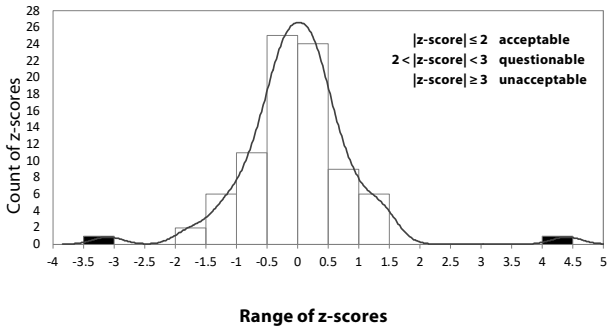


Carbofuran (sum)
(AV derived from subpopulation*)

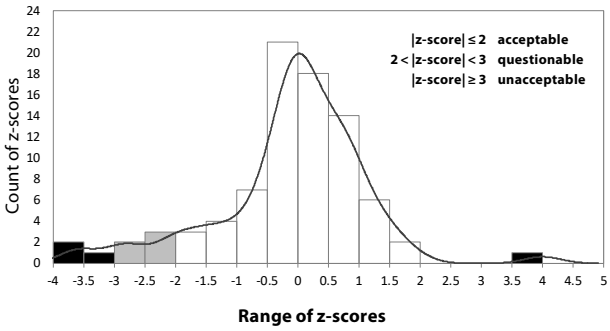


* The subpopulation included results generated by methods including hydrolysation or analysis of carbofuran and carbosulfan separately only

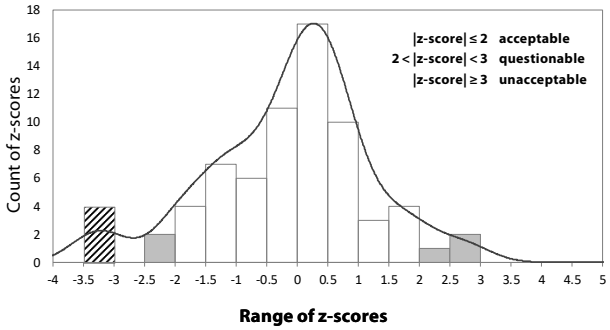
Chlormequat-Cl
(AV derived from entire population excl. 3 outliers)



Glyphosate
(AV derived from entire population excl. 1 outlier)



TFNA
(AV derived from entire population excl. 1 outlier)

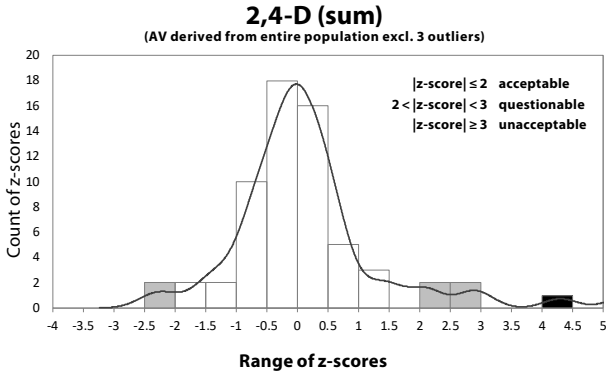


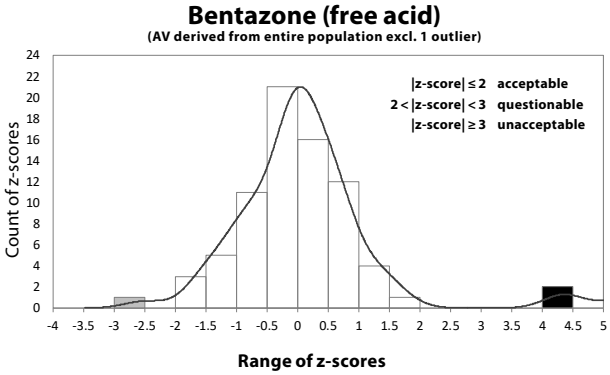
* Cut-off at z-score = 5; ▨: false negative results

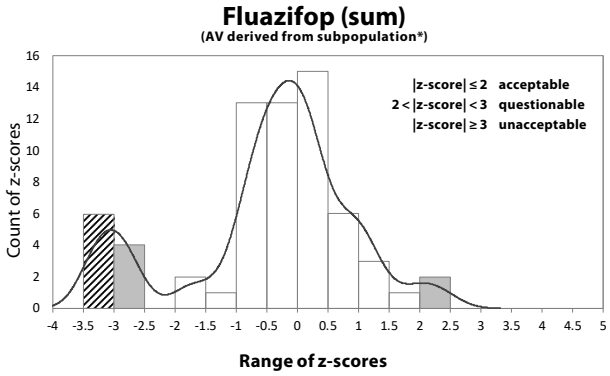
Appendix 5 (cont.) Histograms and Kernel Density Estimates of z-score* Distributions

(Results from EU and EFTA Laboratories only)

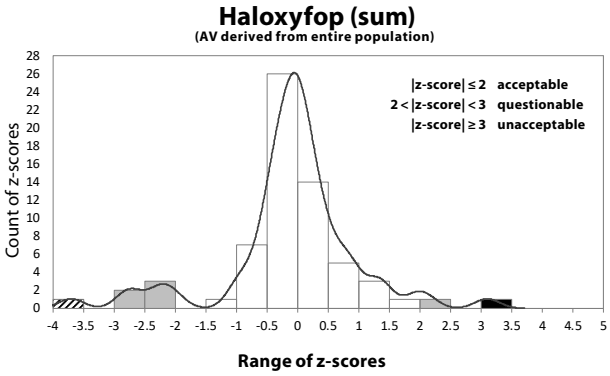
Optional Compounds

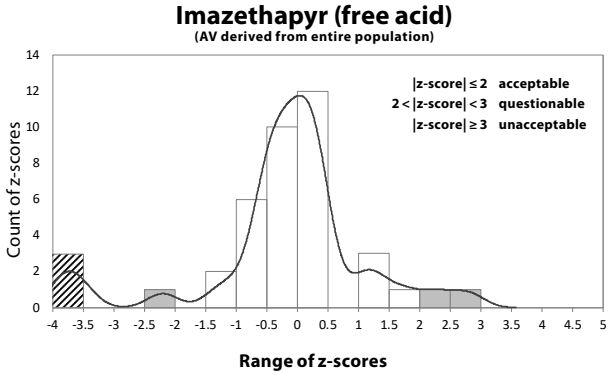


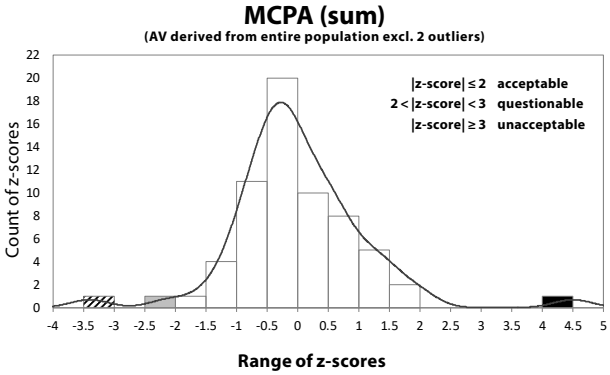




* The subpopulation included results generated by methods including moderate or strong hydrolysis only







***** Cut-off at z-score = 5; ▨: false negative results

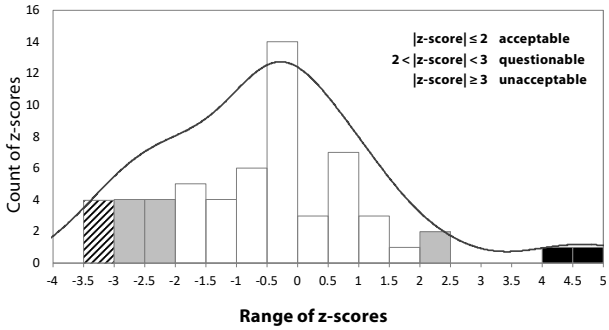
Appendix 5 (cont.) Histograms and Kernel Density Estimates of z-score* Distributions

(Results from EU and EFTA Laboratories only)

Optional Compounds

MCPB (sum)

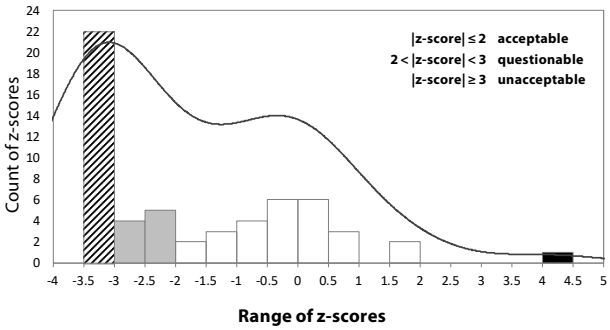
(AV derived from subpopulation* excl. 1 outlier)



* The subpopulation included results generated by methods including moderate or strong hydrolysis only

MCPP (sum)

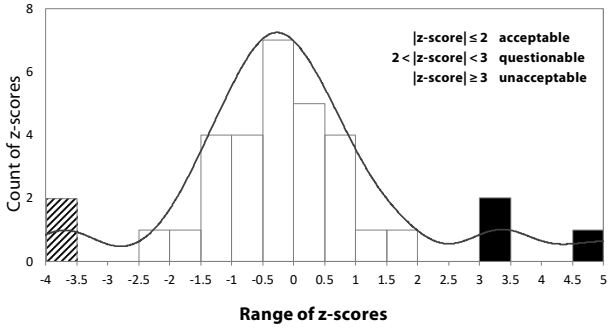
(AV derived from subpopulation* excl. 1 outlier)



* The subpopulation included results generated by methods including strong hydrolysis only

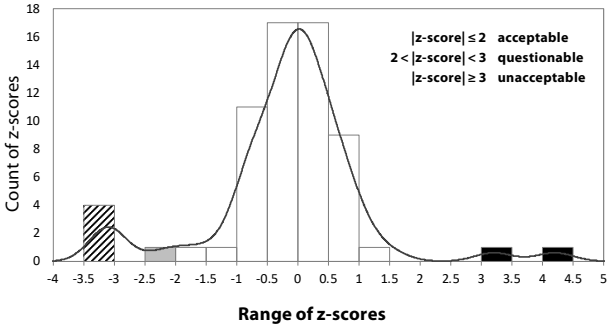
Paraquat (dication)

(AV derived from entire population excl. 1 outlier)



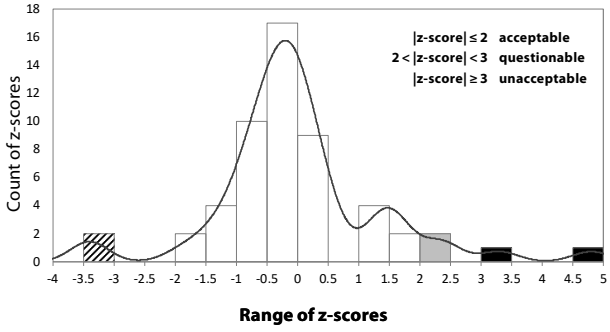
Quizalofop (free acid)

(AV derived from entire population)



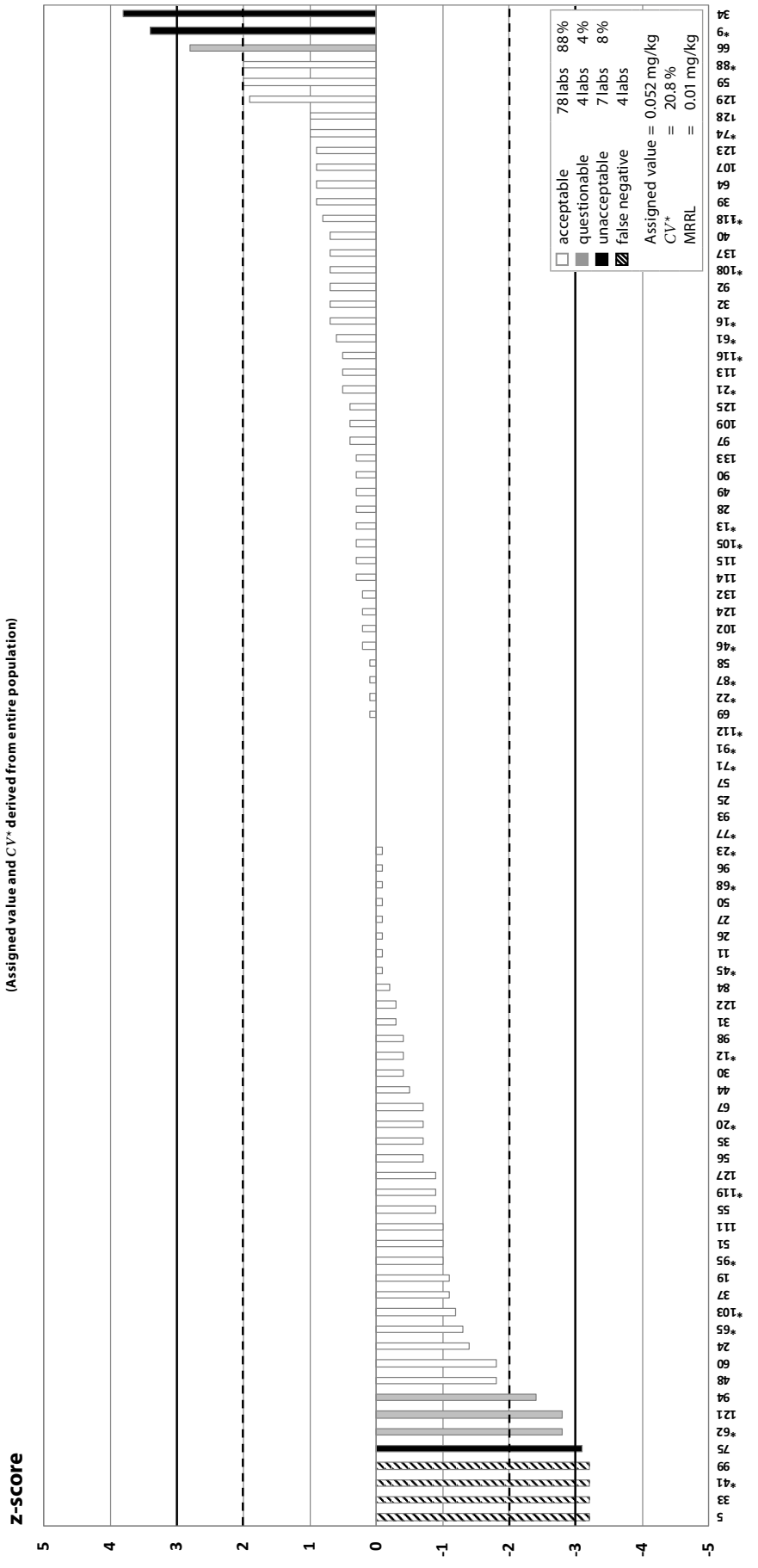
Quizalofop (sum)

(AV derived from entire population)

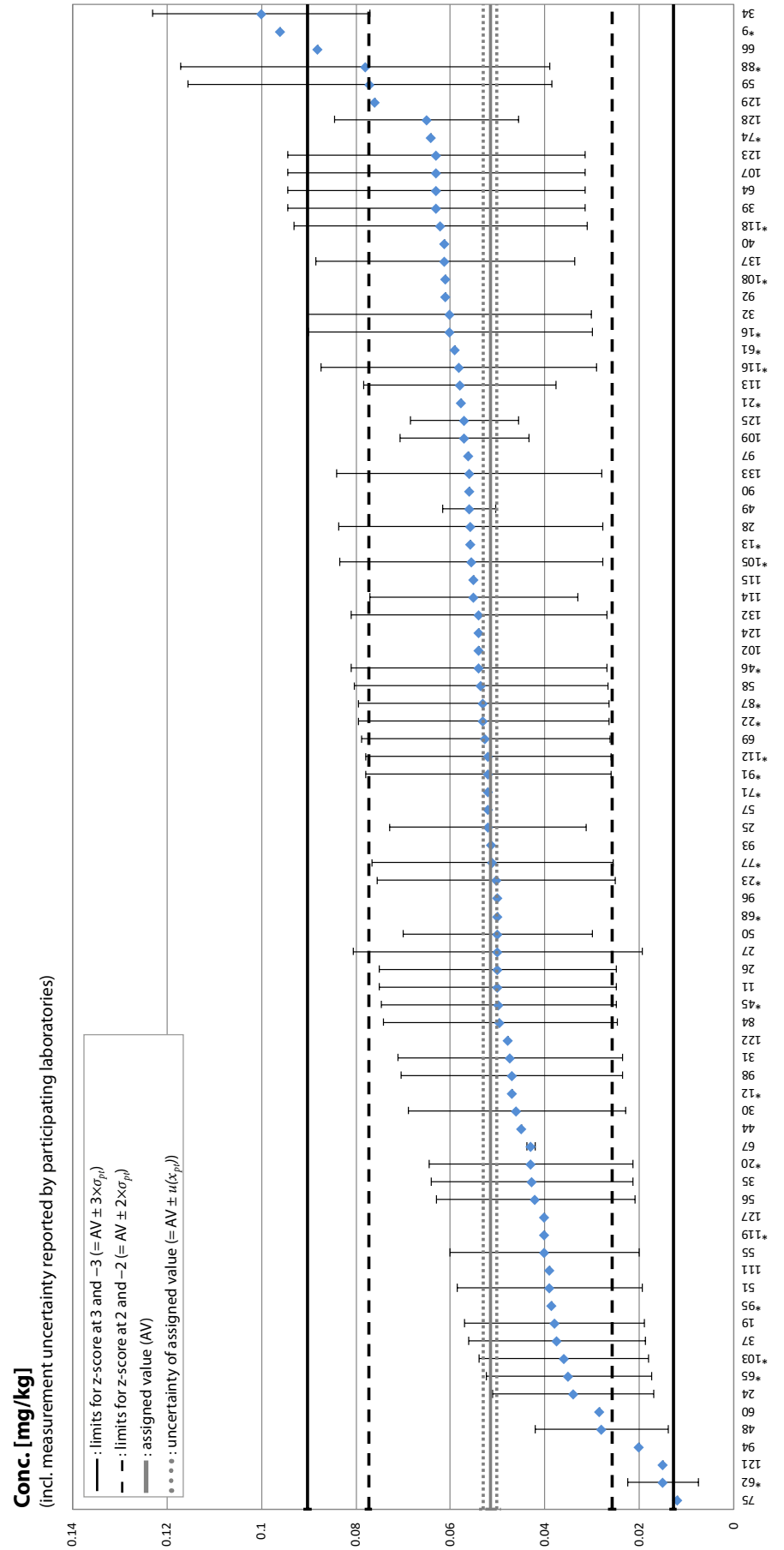


* Cut-off at z-score = 5; ▨: false negative results

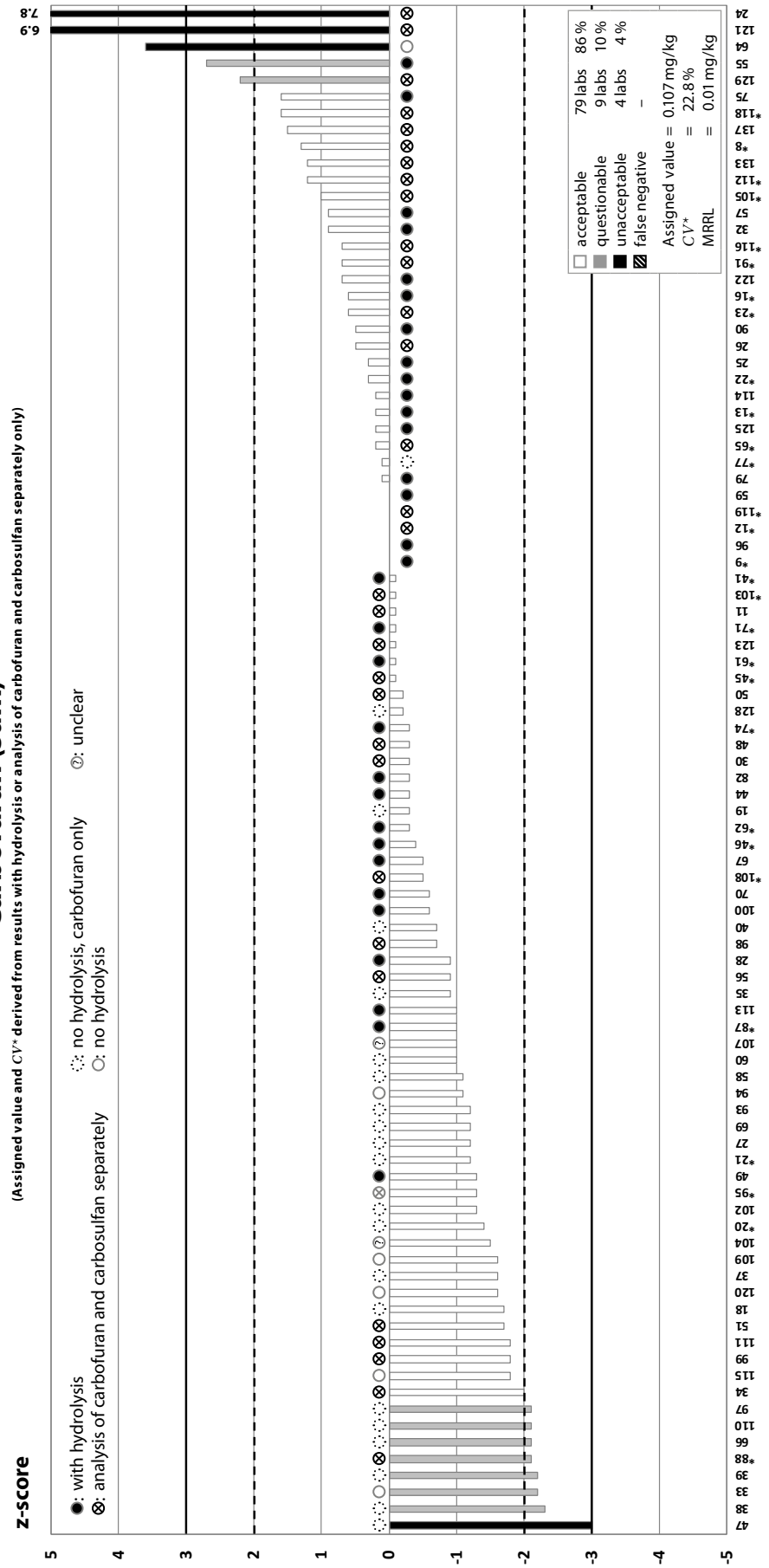
Appendix 6 Graphic Presentation of z-Scores: Compulsory Compounds(Results from EU and EFTA Laboratories only, * = NRL)



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

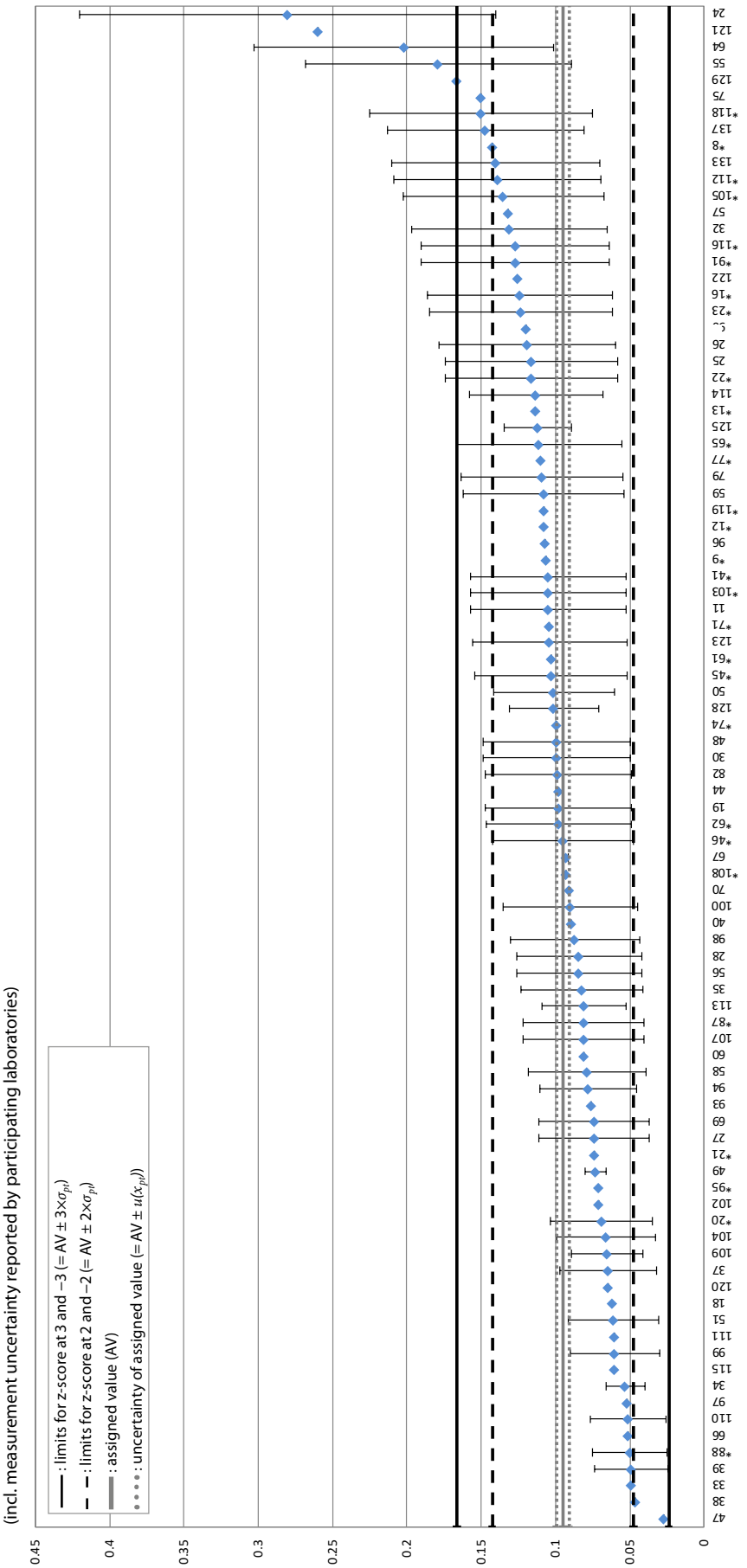


Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)

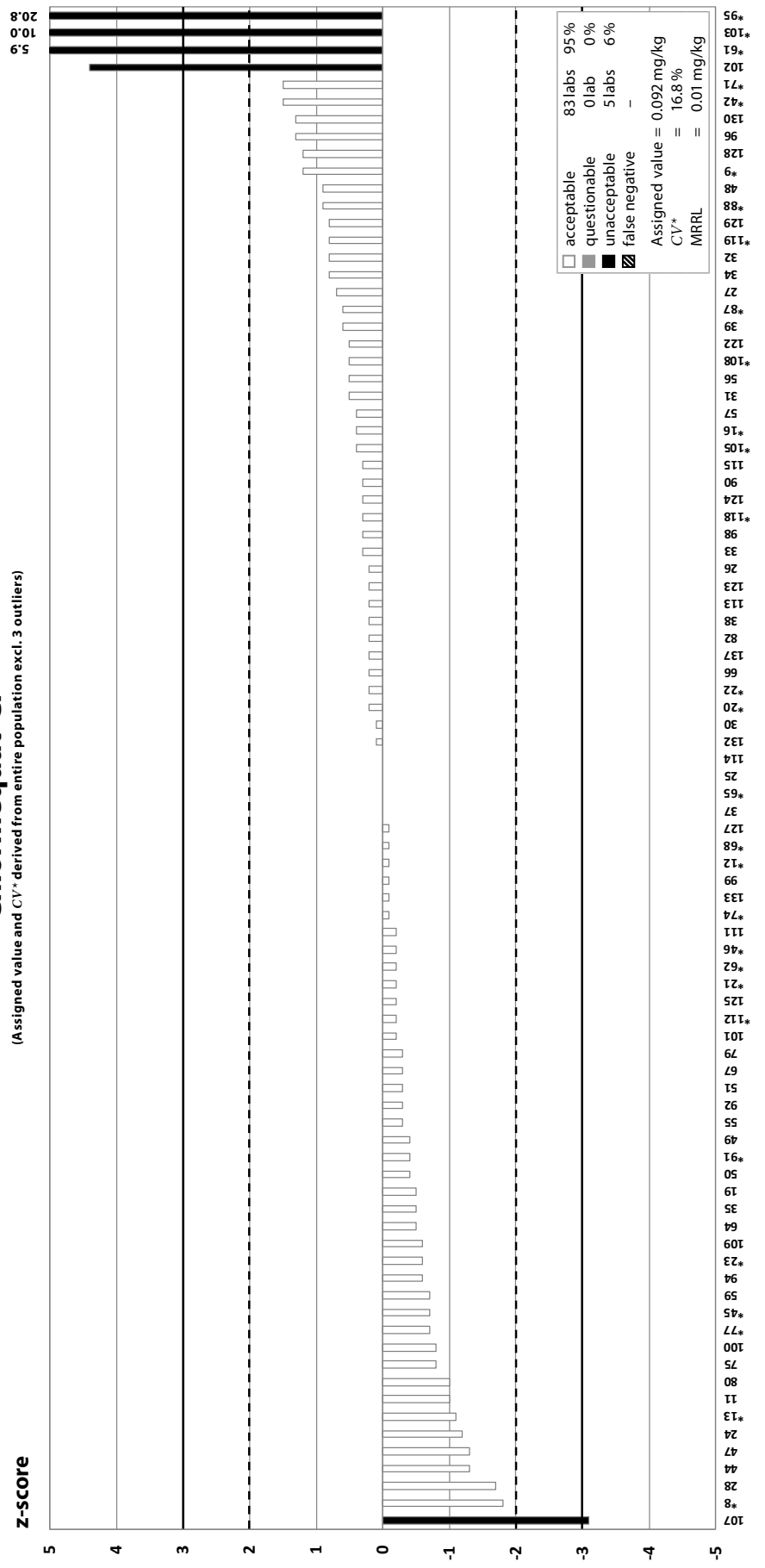


A-18

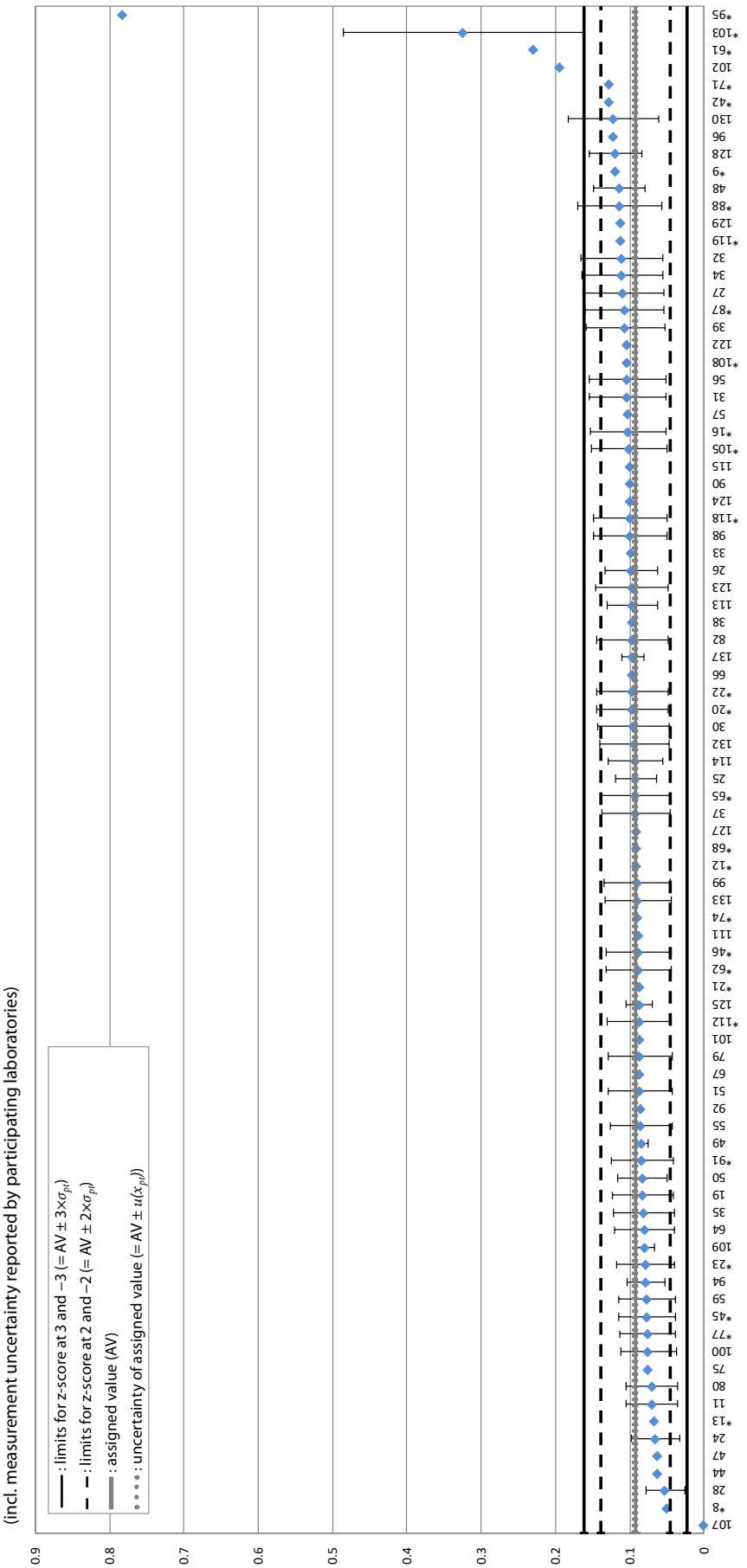
Appendix 6 (cont.) **Graphic Presentation of Results: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



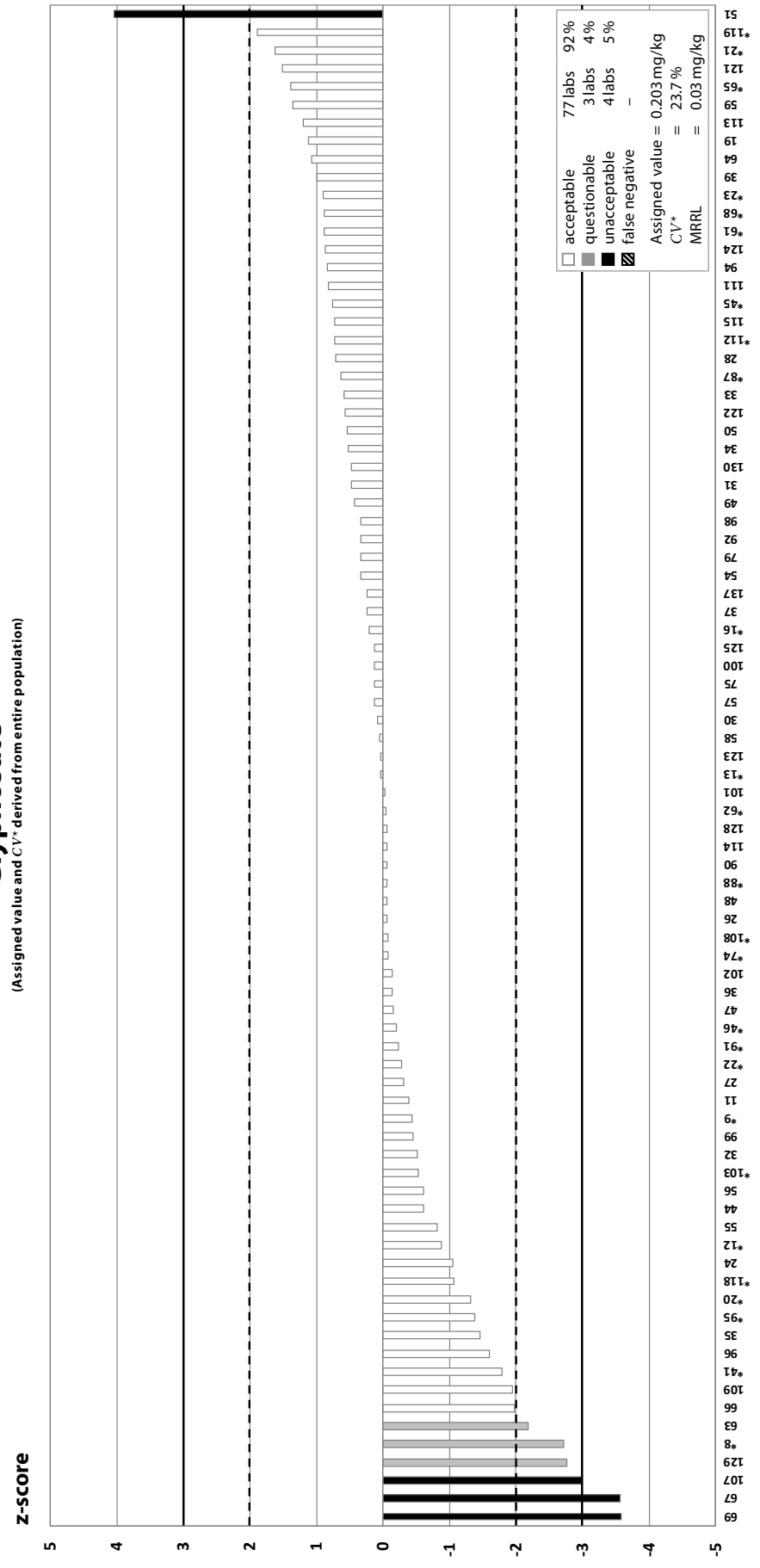
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



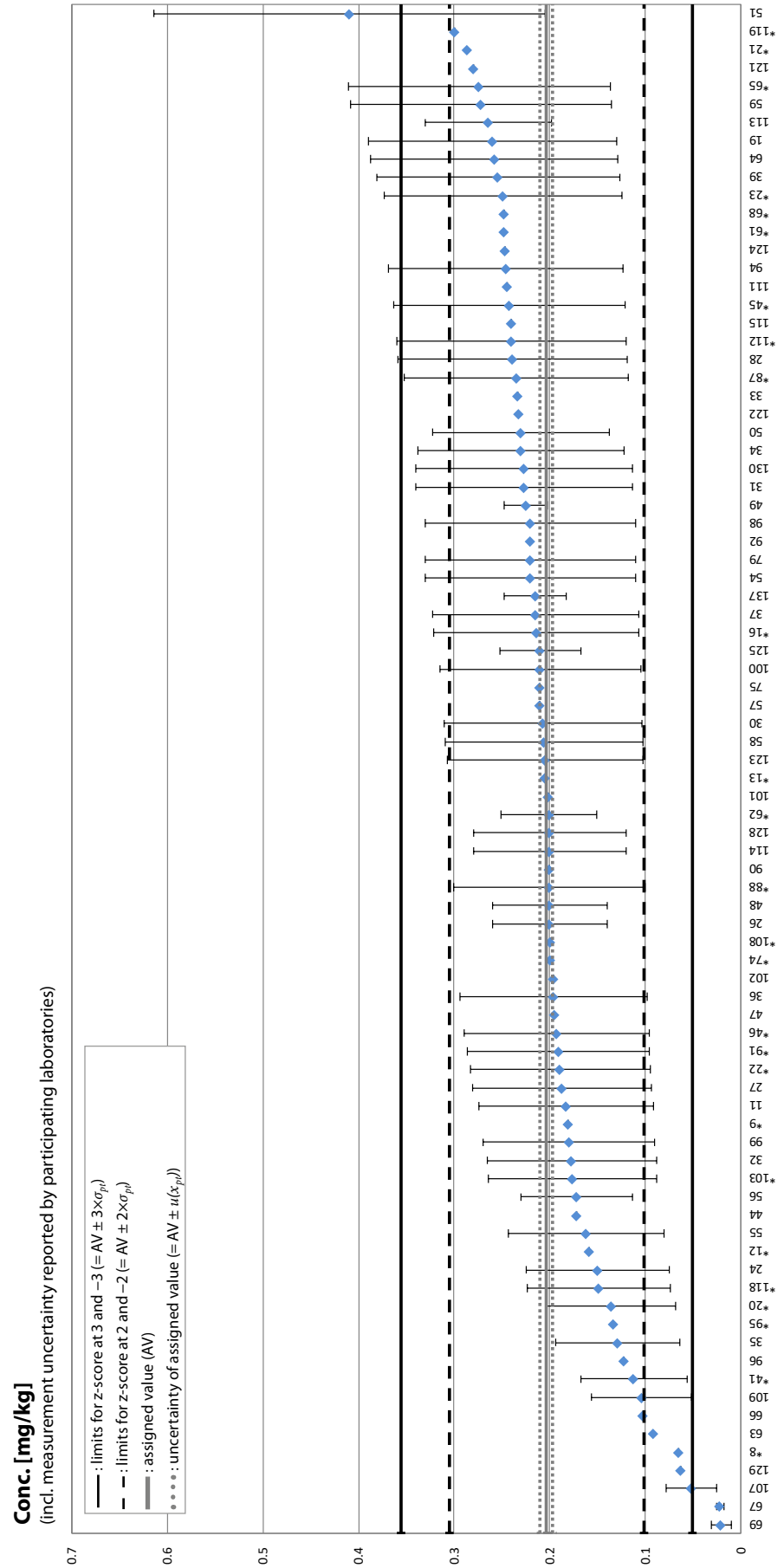
Appendix 6 (cont.) **Graphic Presentation of Results: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



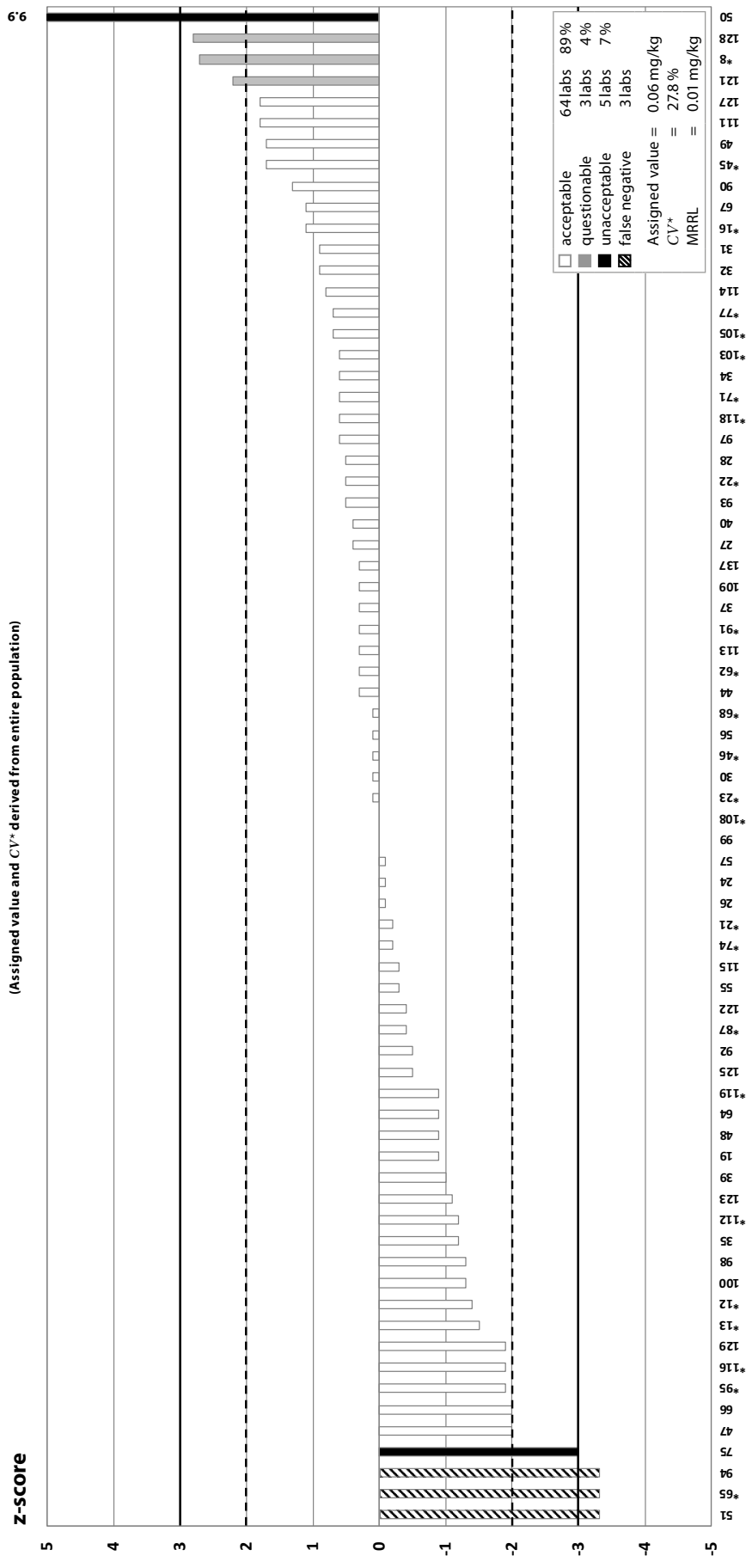
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



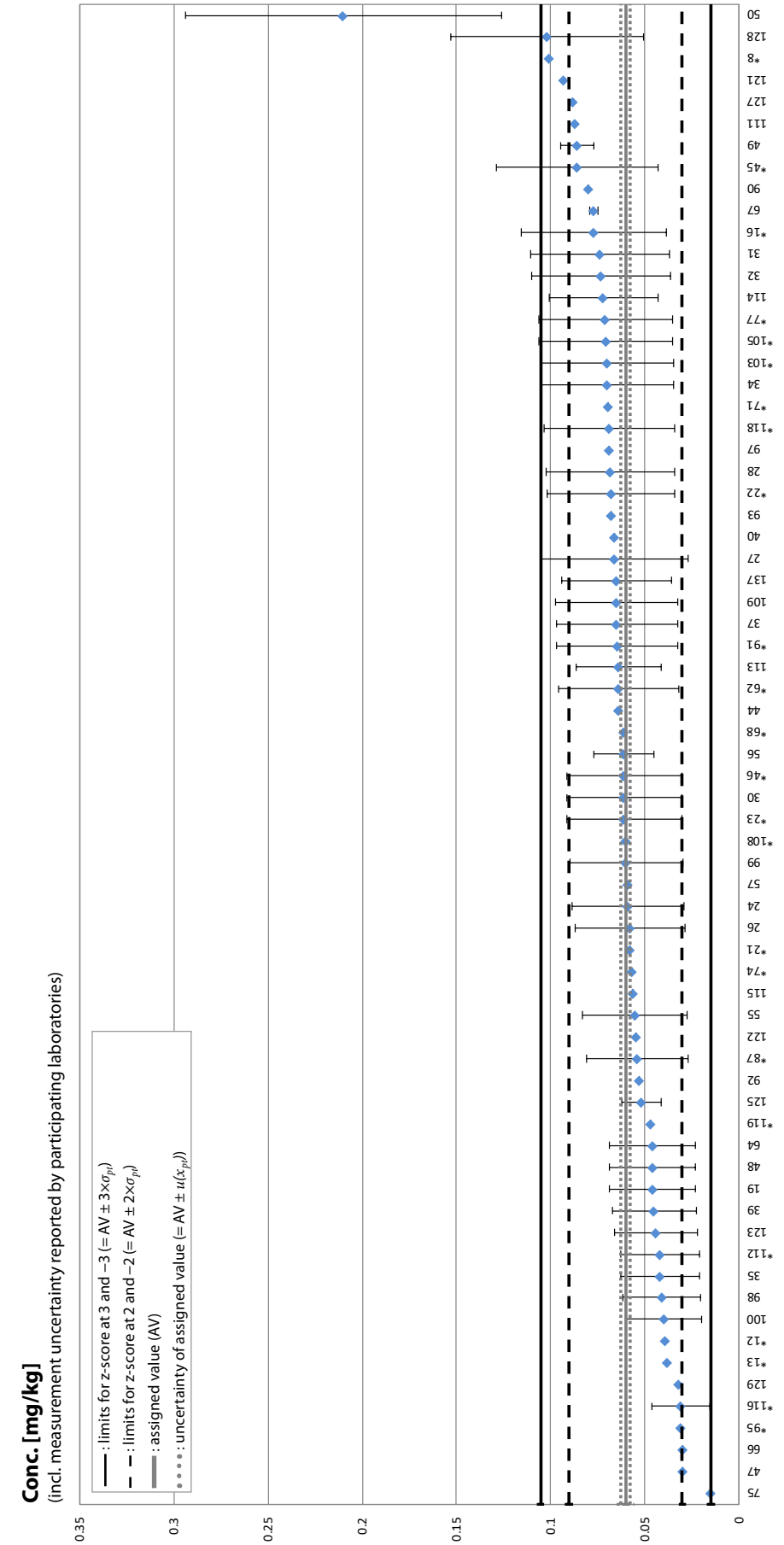
Appendix 6 (cont.) **Graphic Presentation of Results: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



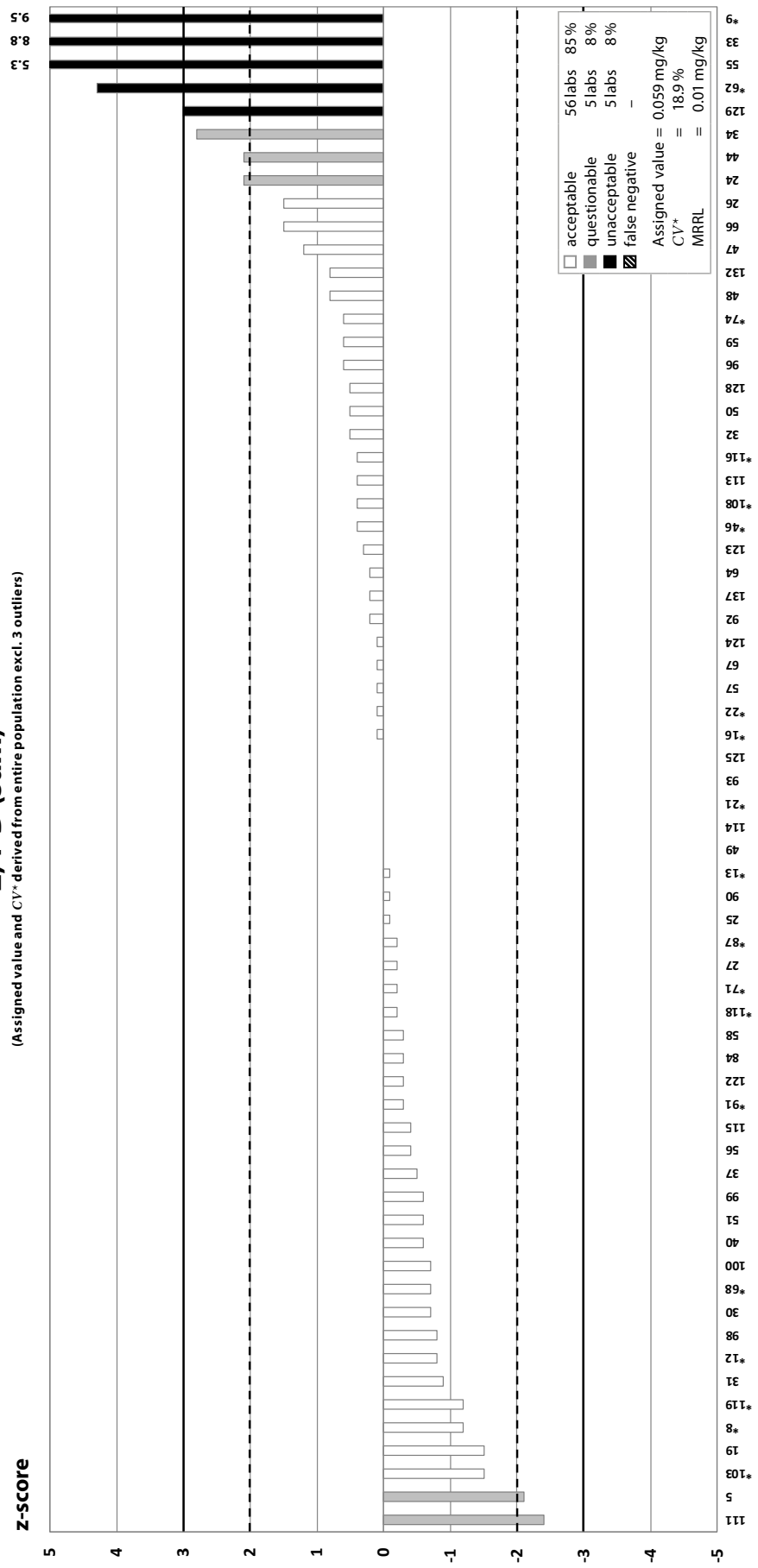
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



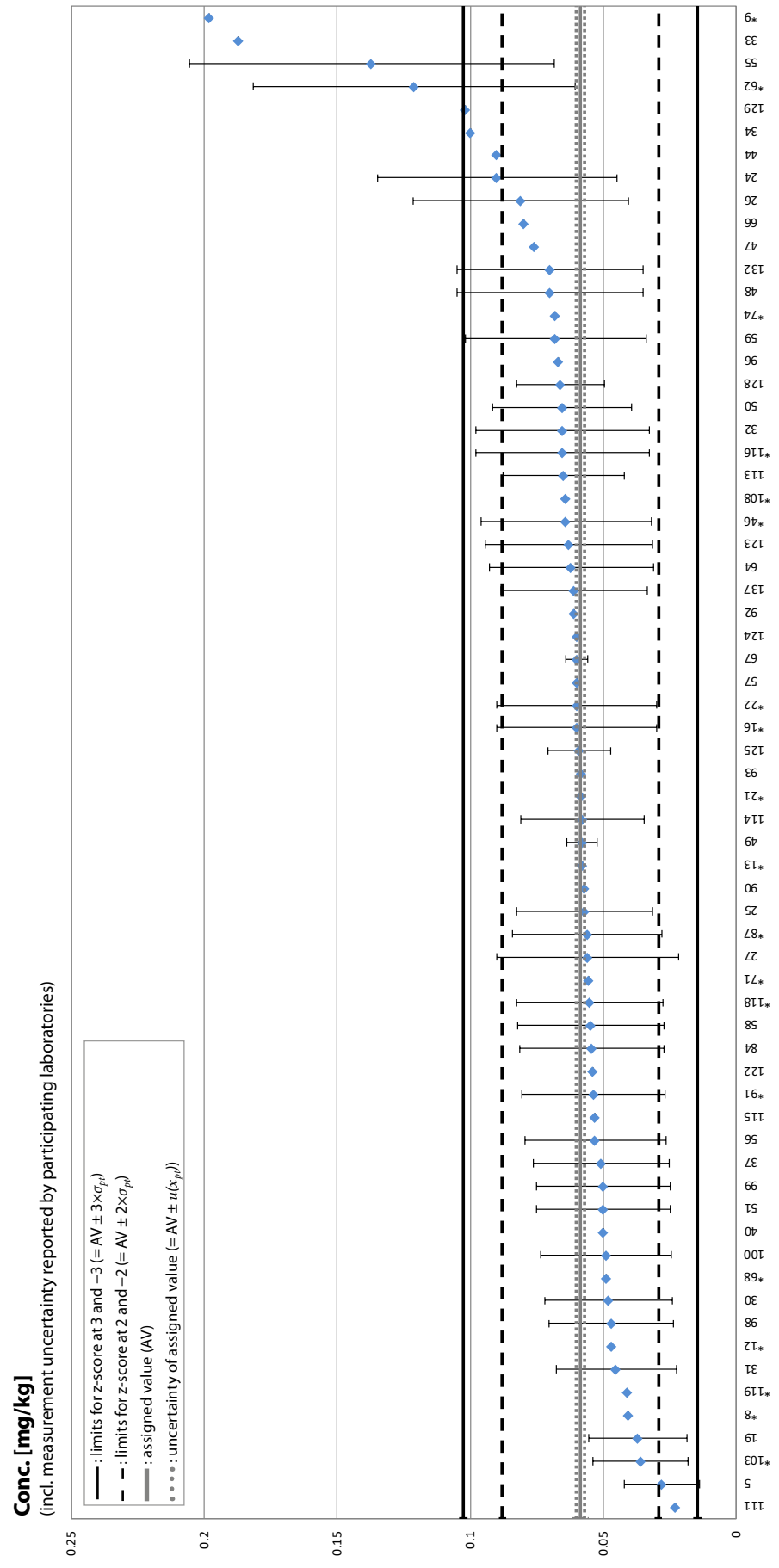
Appendix 6 (cont.) **Graphic Presentation of Results: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



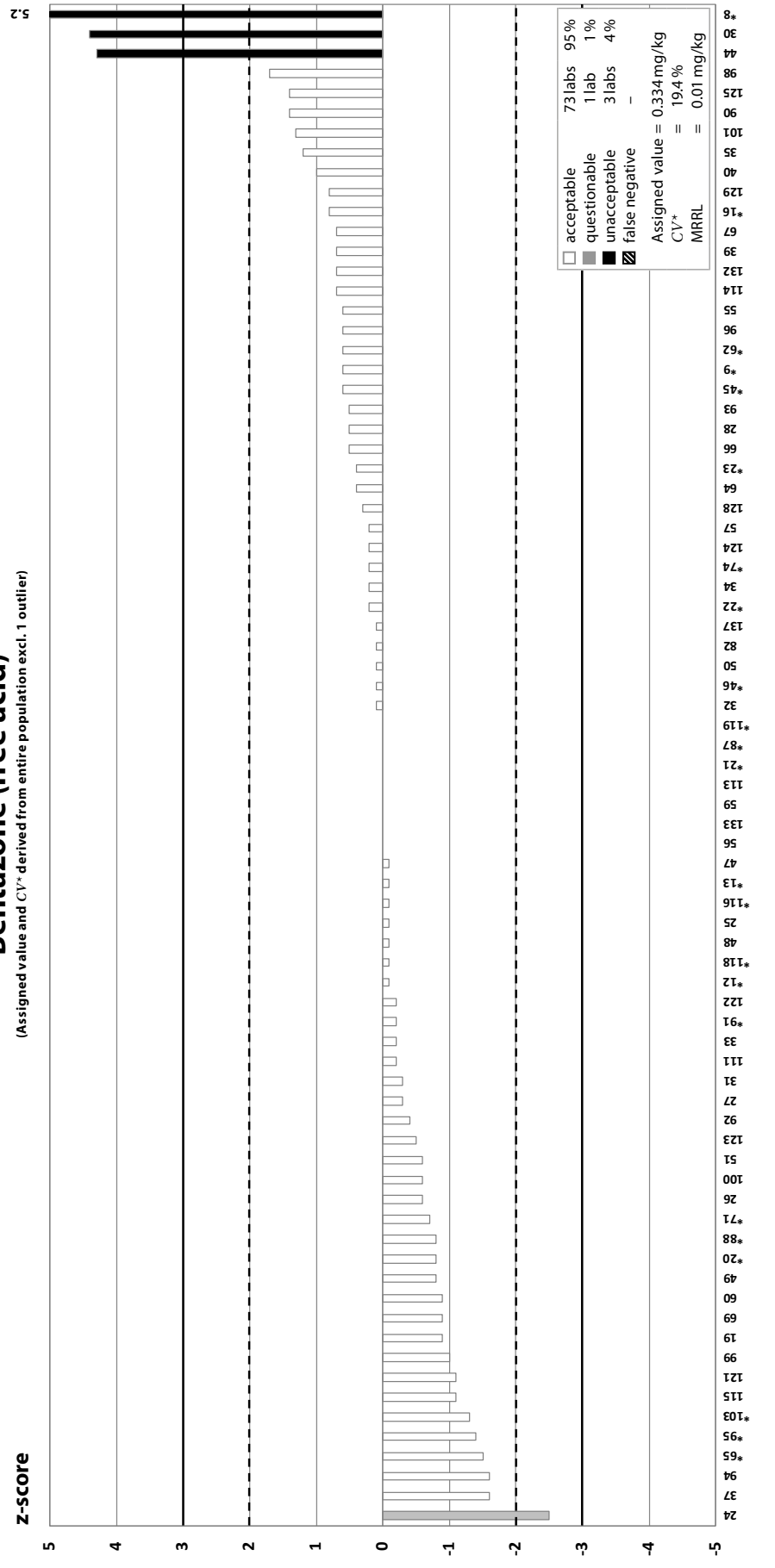
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



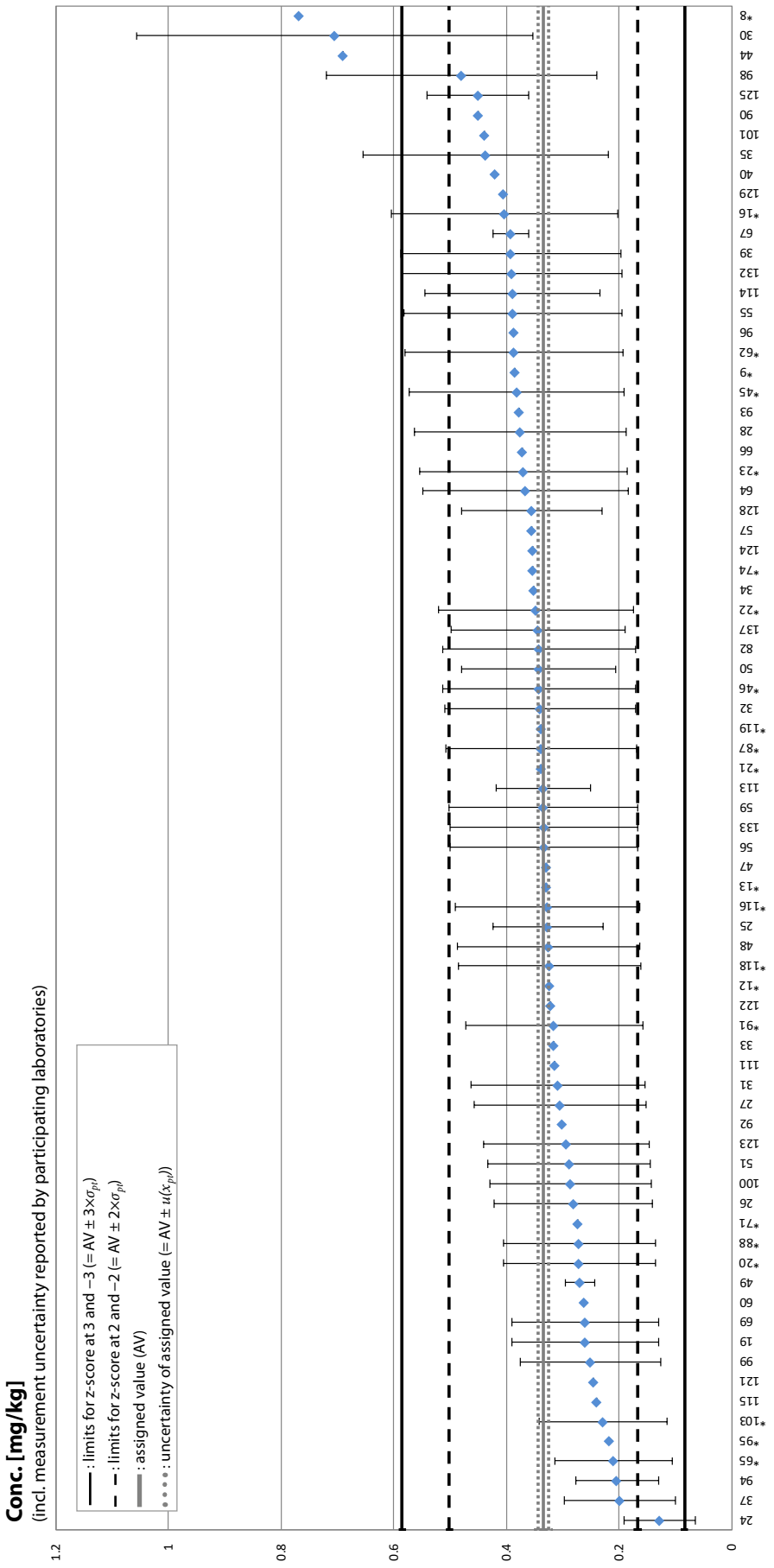
Appendix 6 (cont.) **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



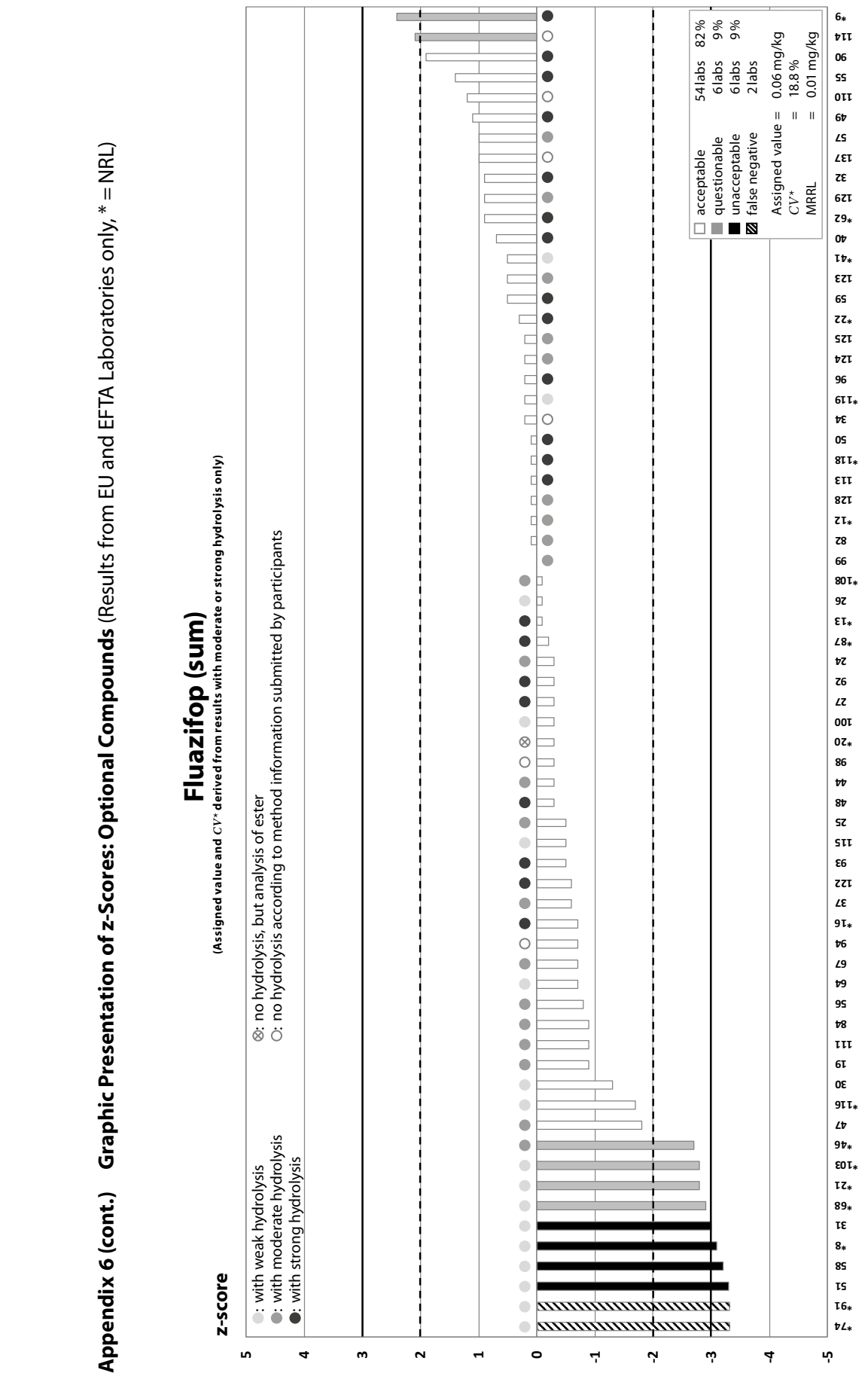
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



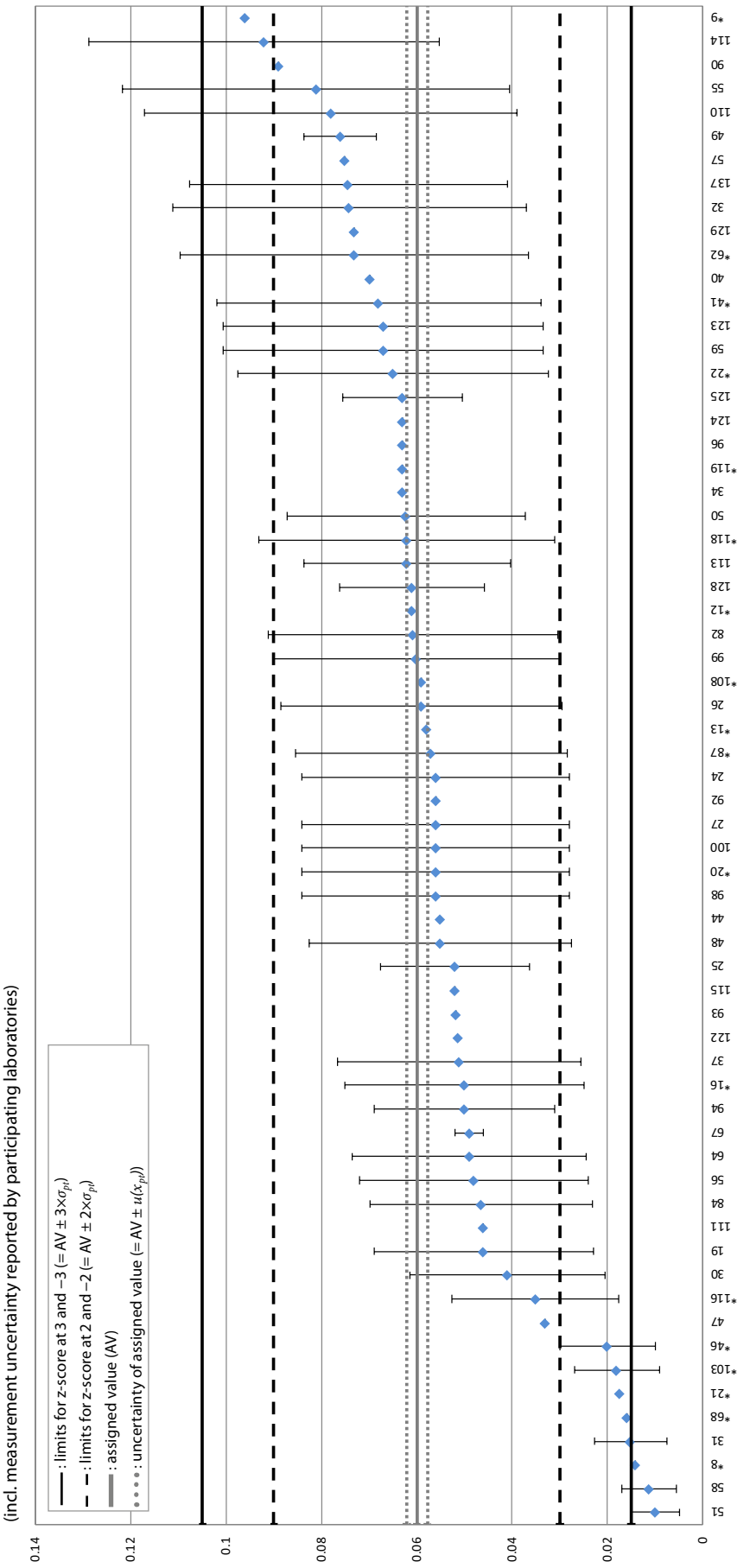
Appendix 6 (cont.) **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



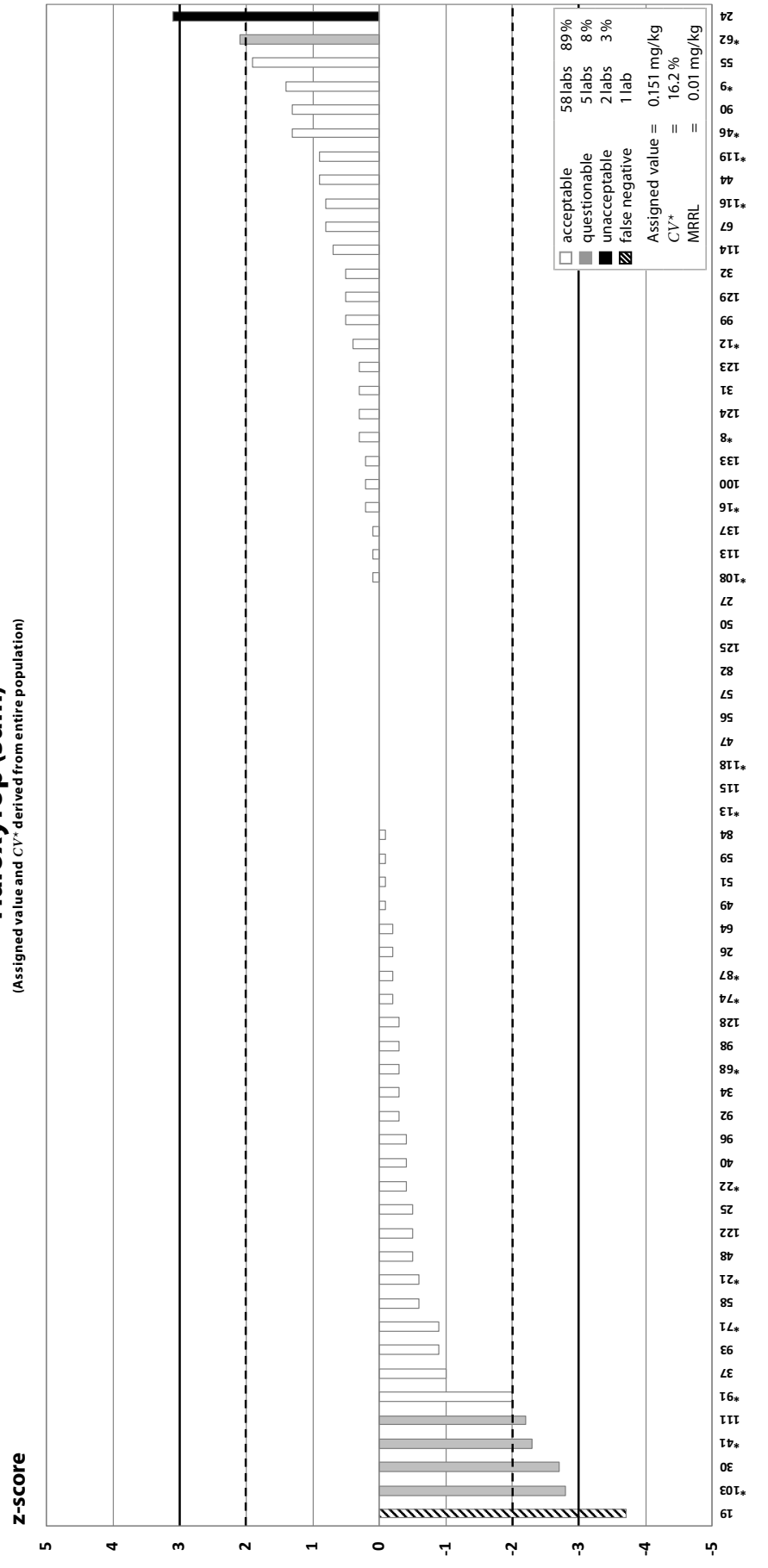
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



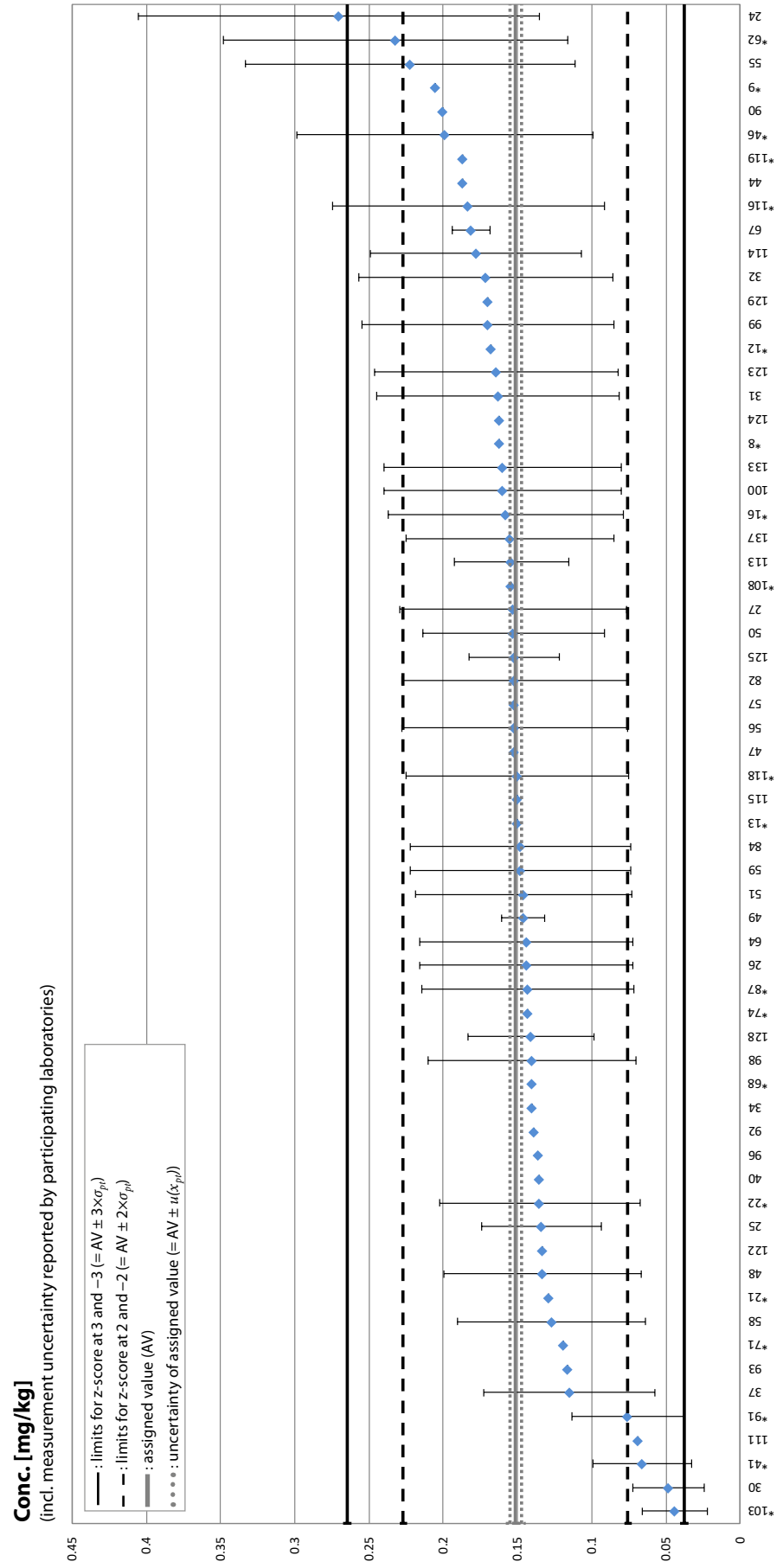
Appendix 6 (cont.) **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



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

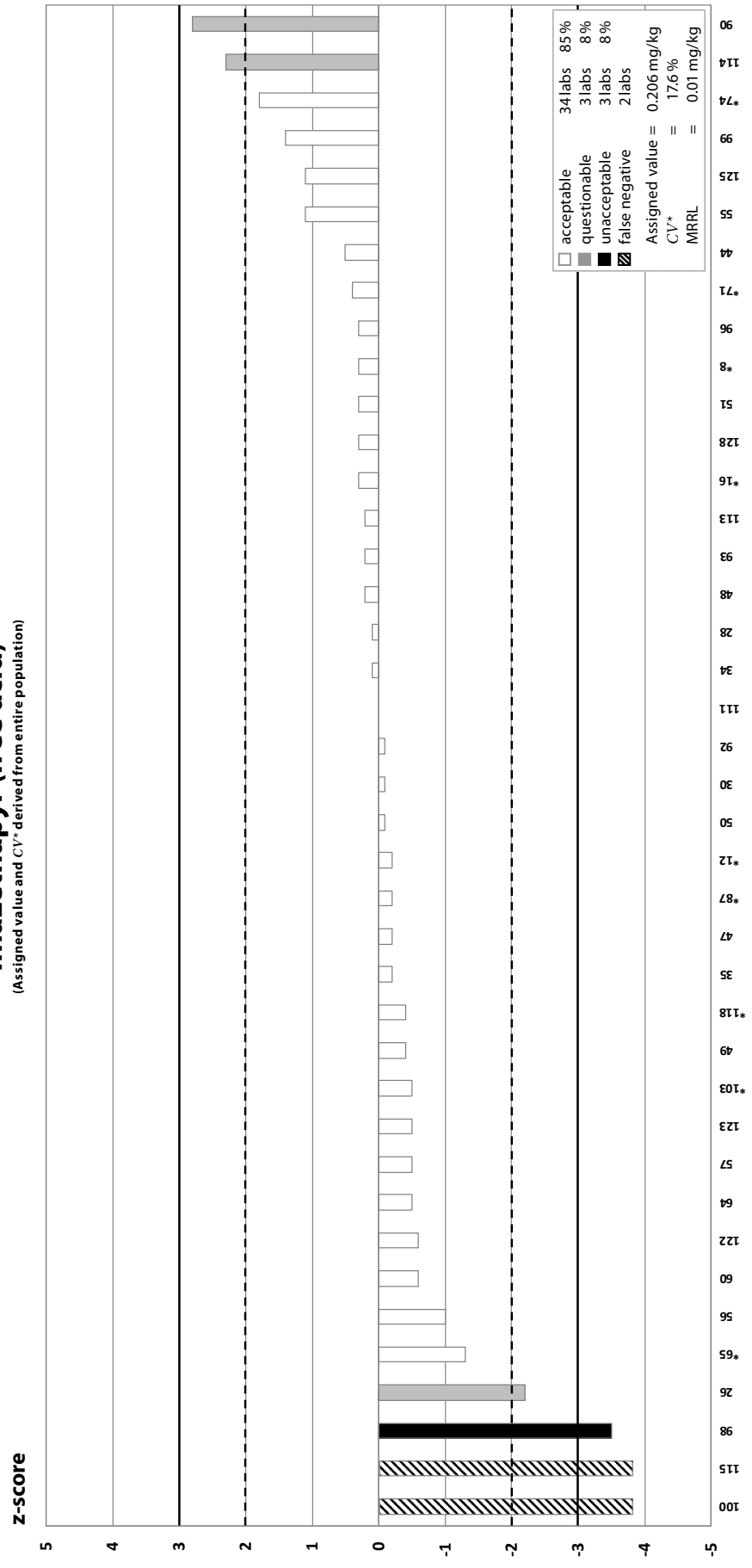


Appendix 6 (cont.) Graphic Presentation of Results: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)

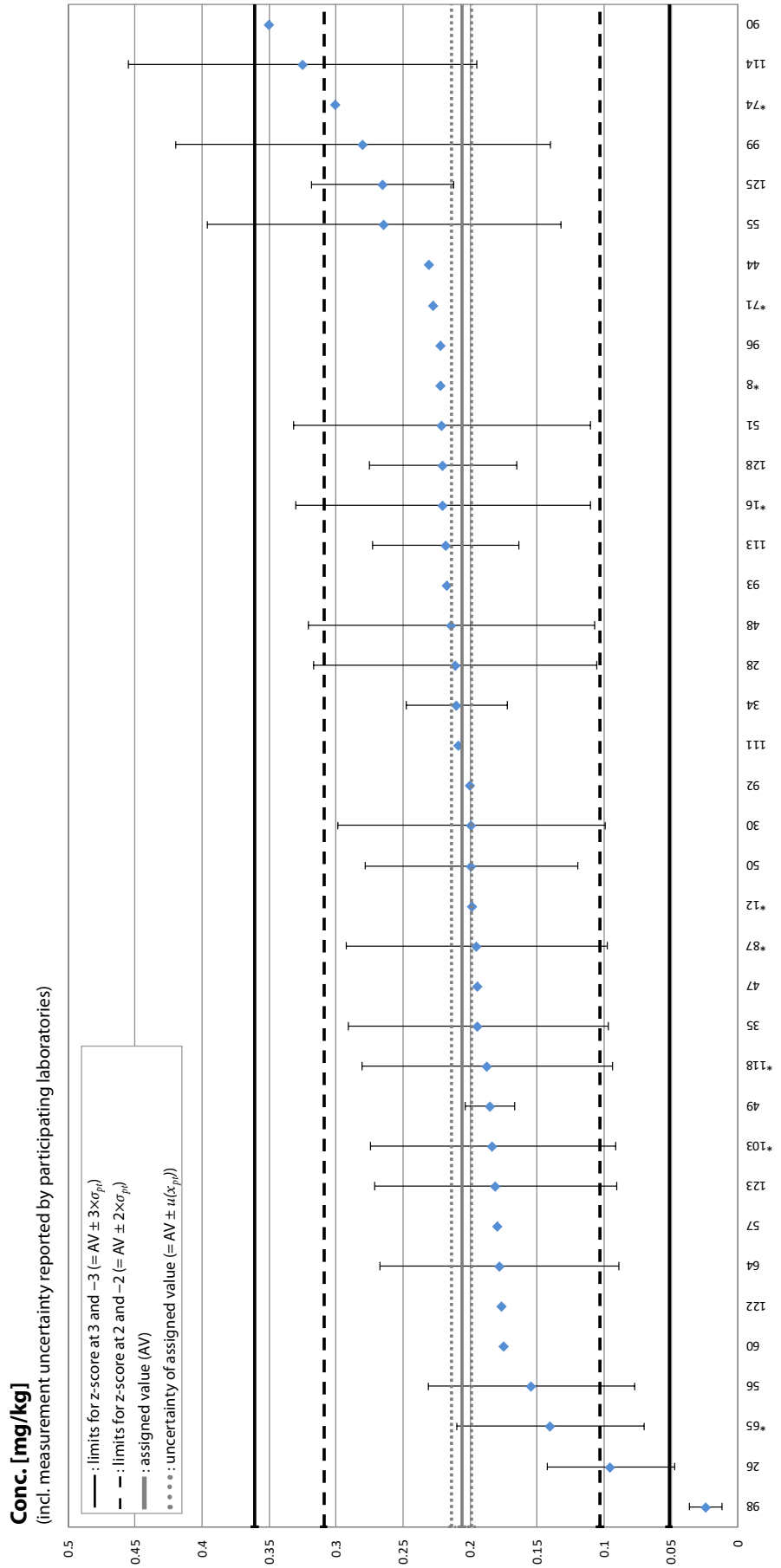


A-33

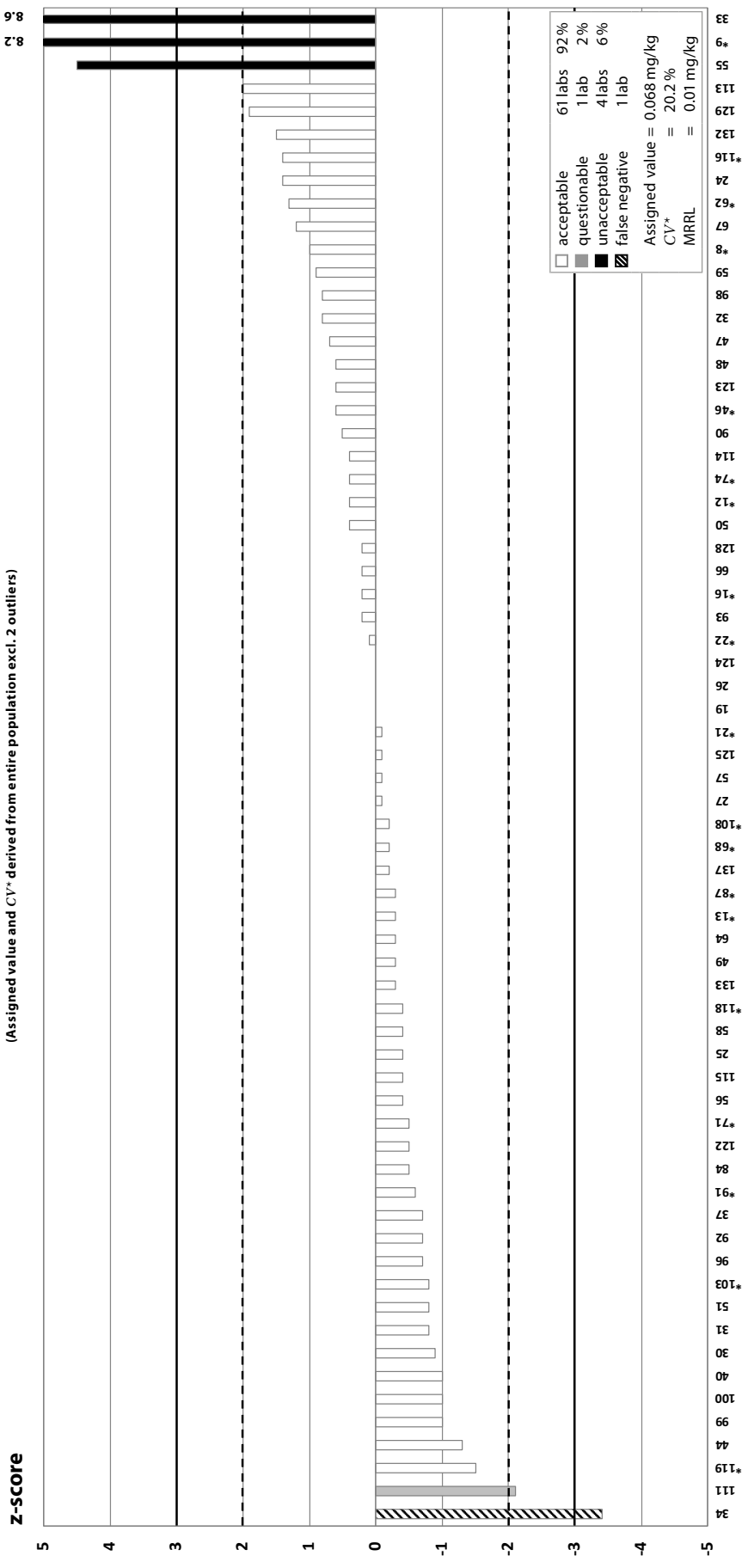
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



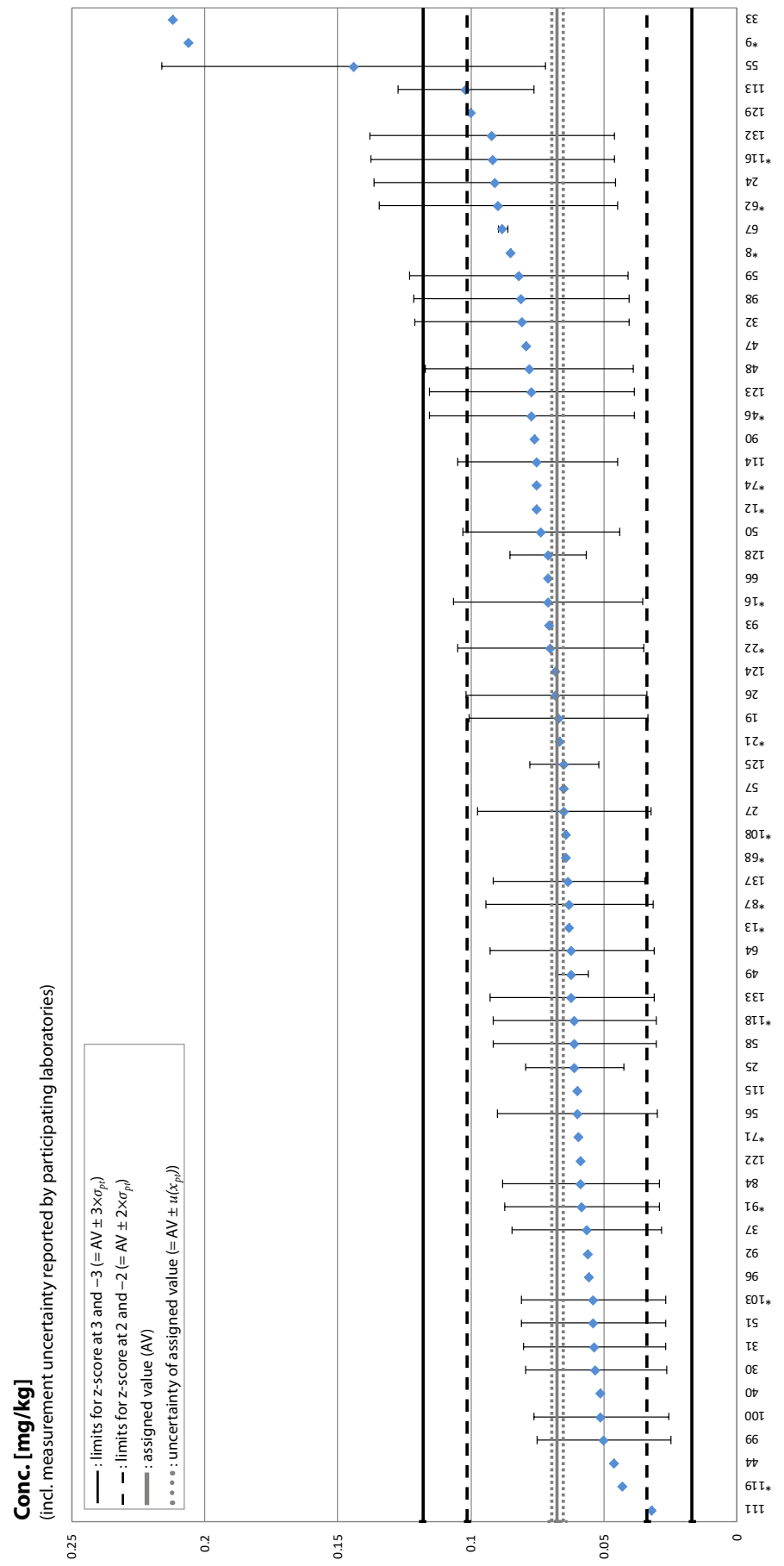
Appendix 6 (cont.) **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



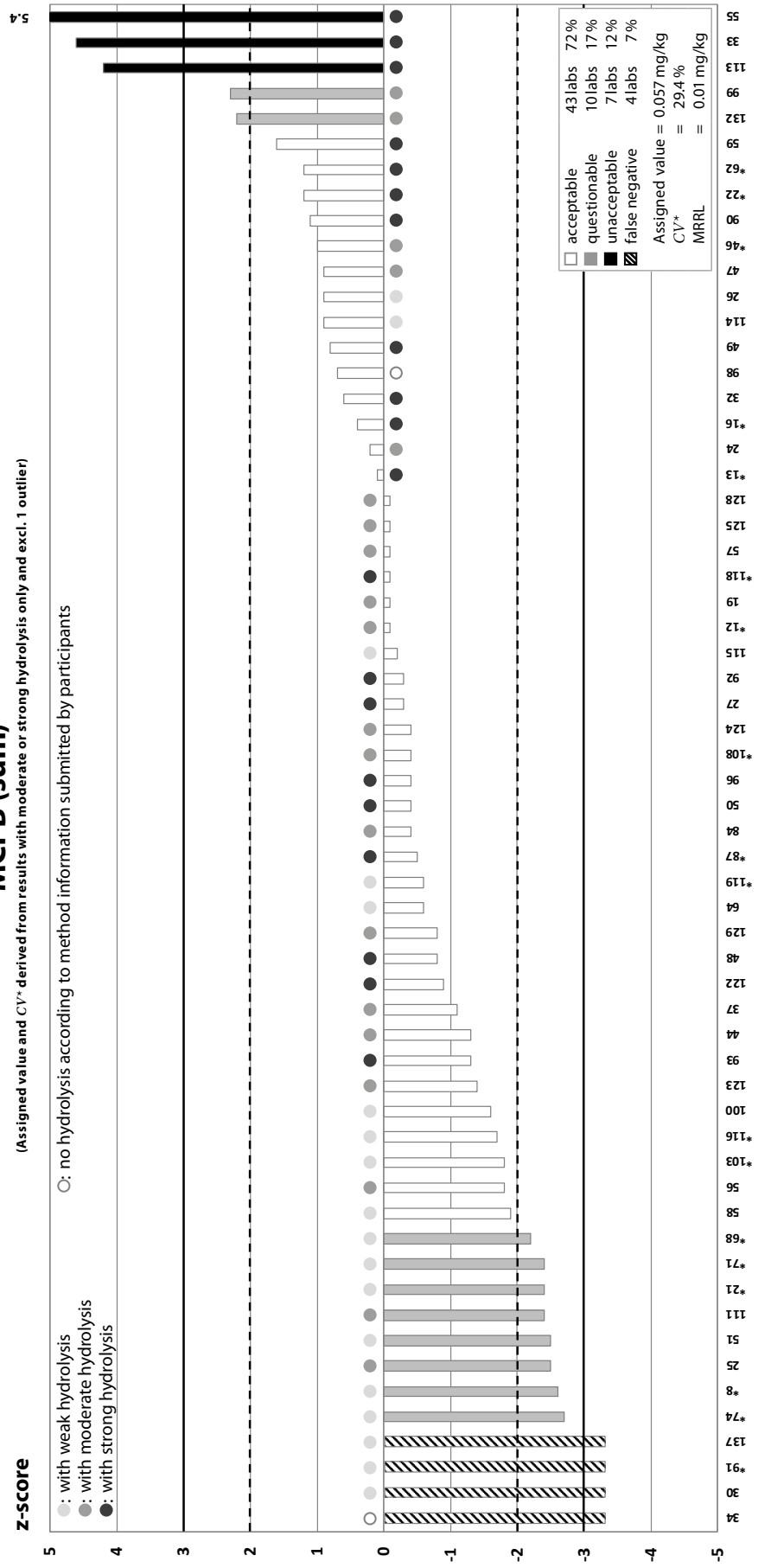
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



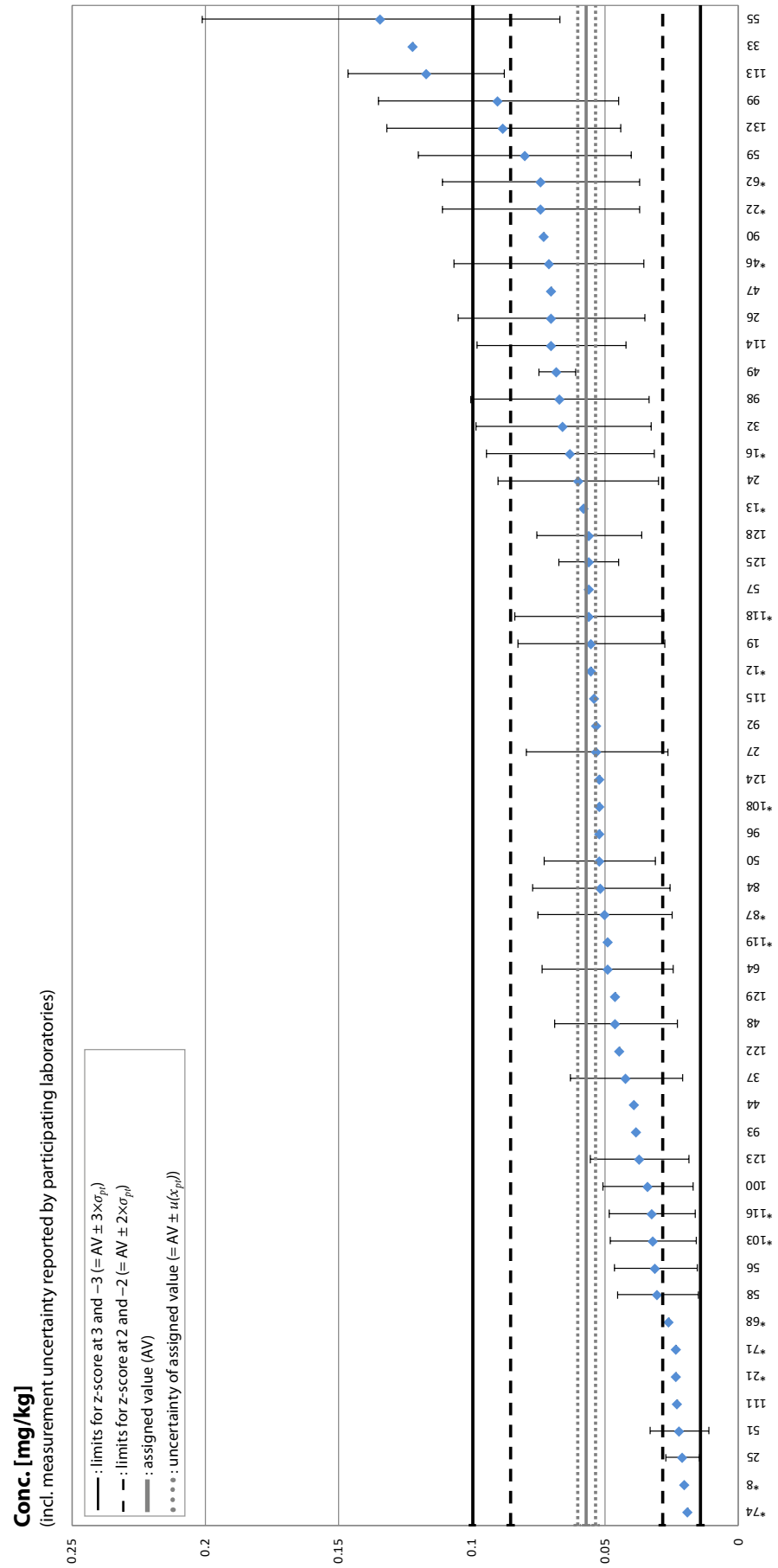
Appendix 6 (cont.) **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



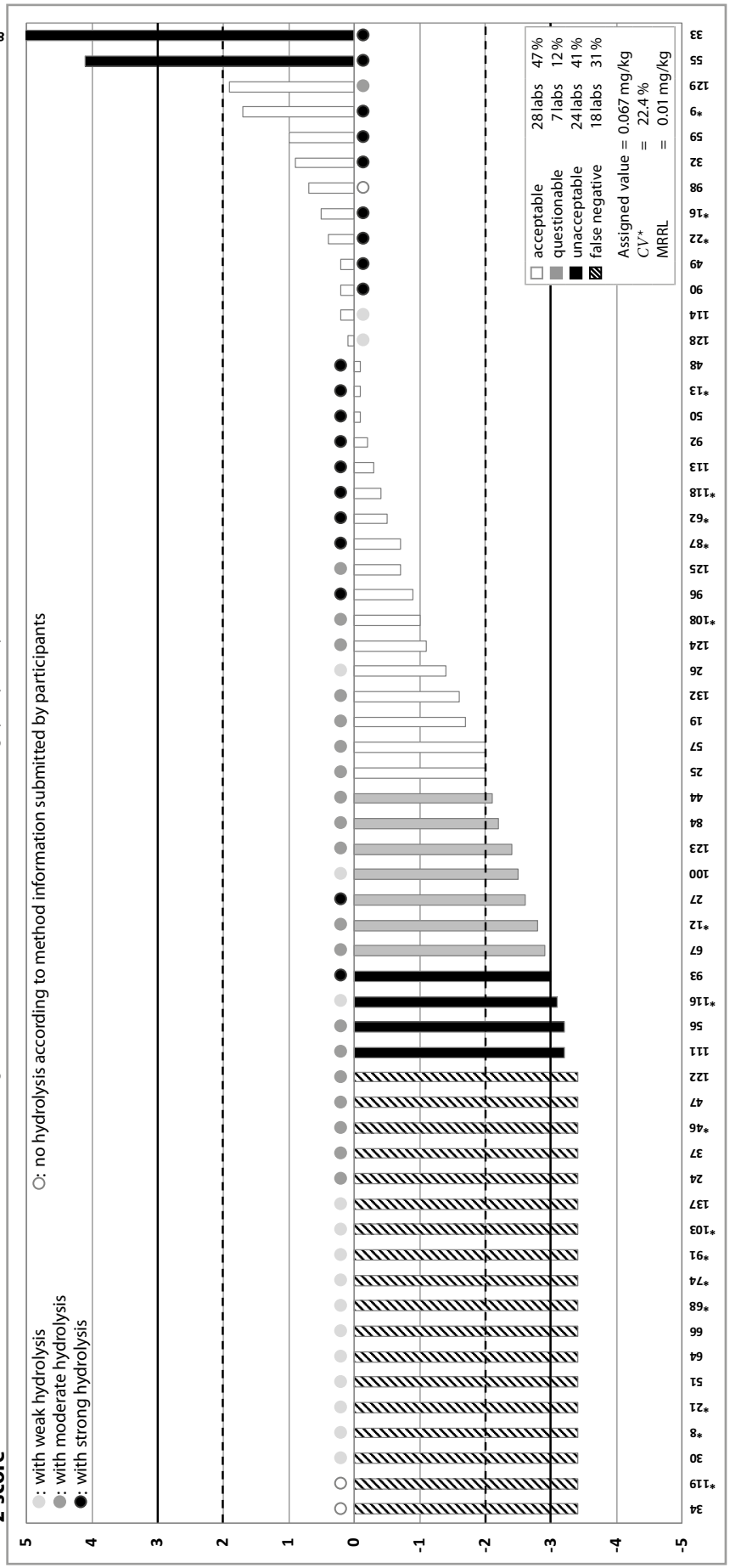
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



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

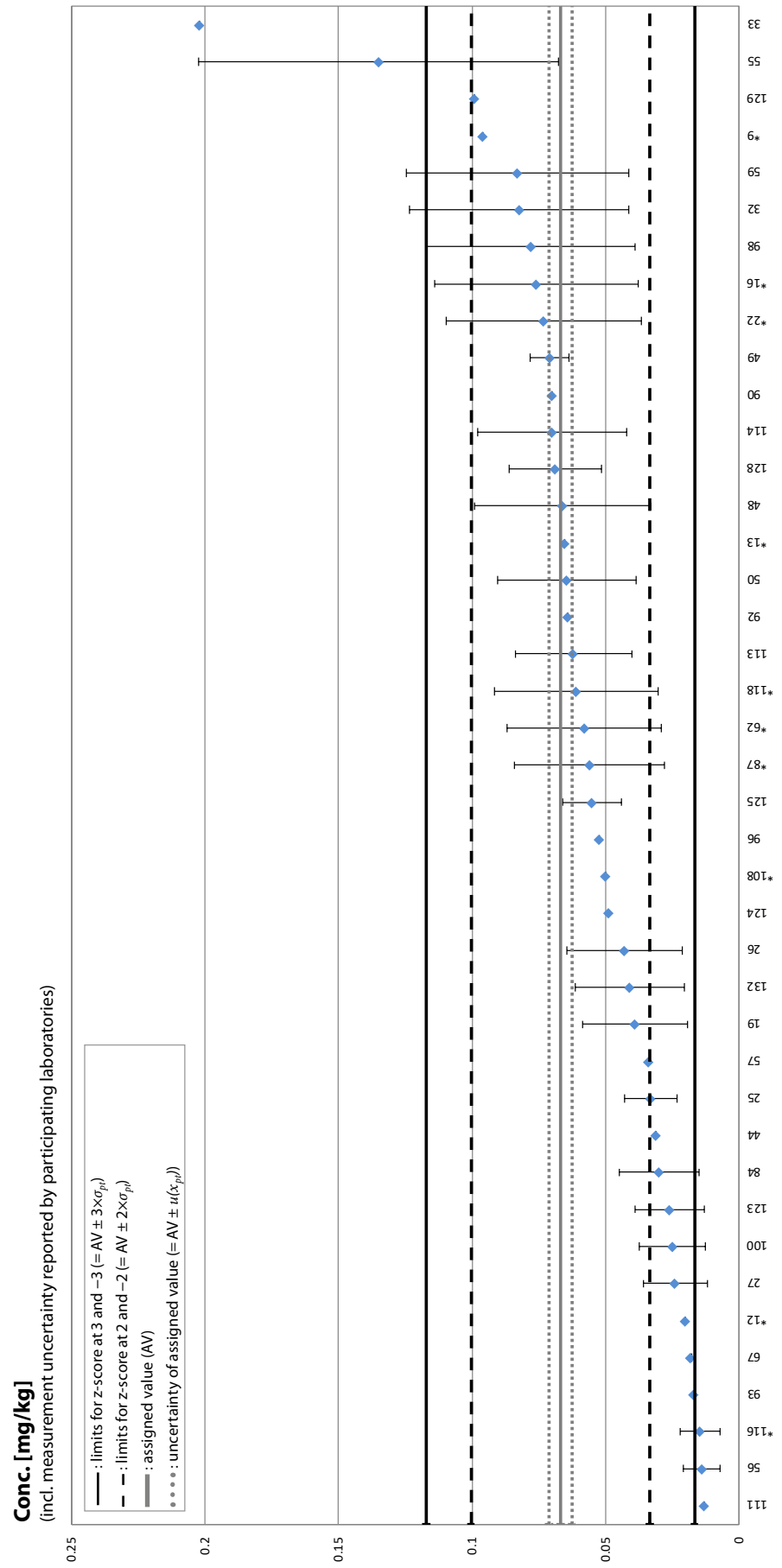


Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)

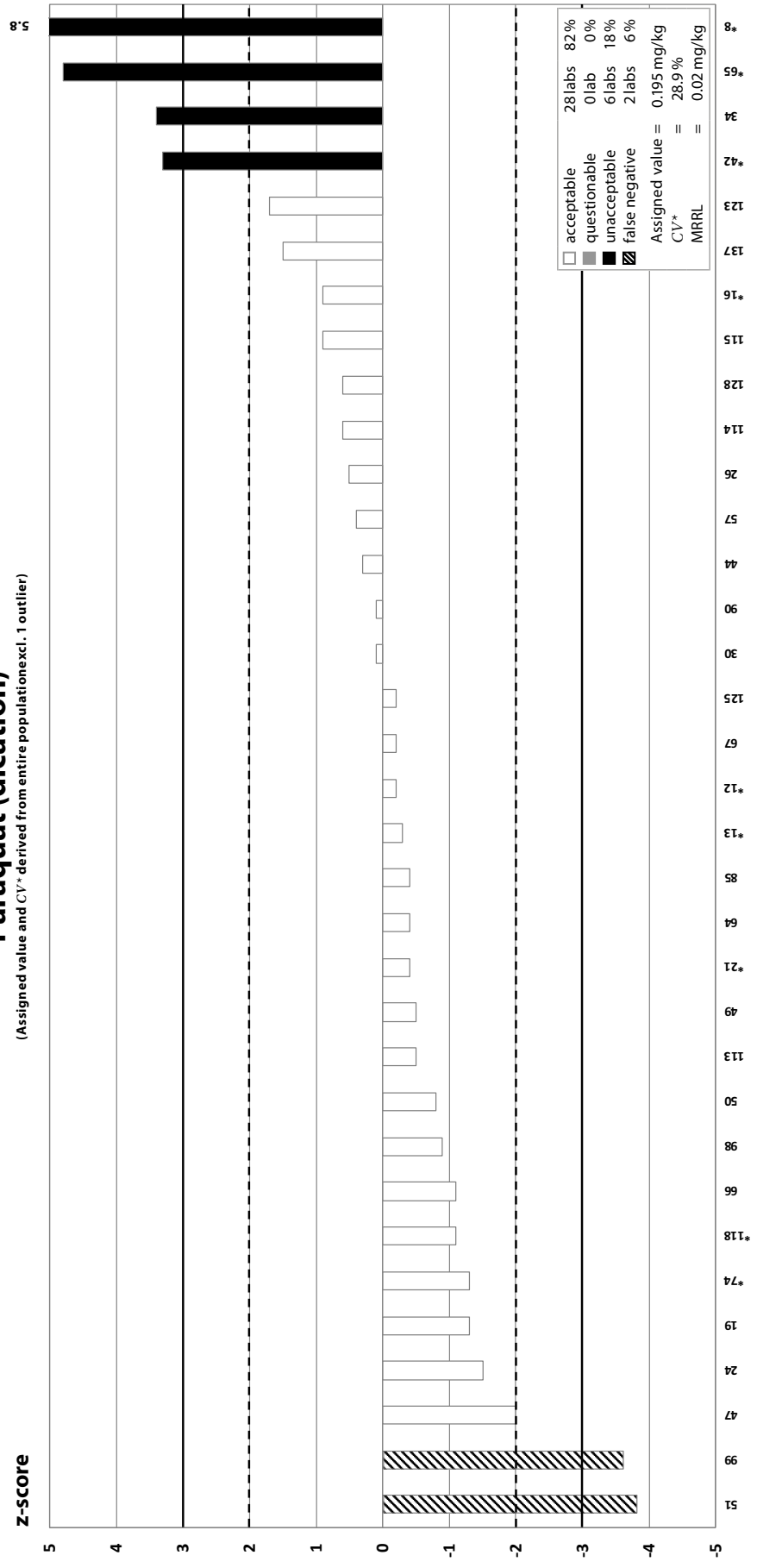


A-40

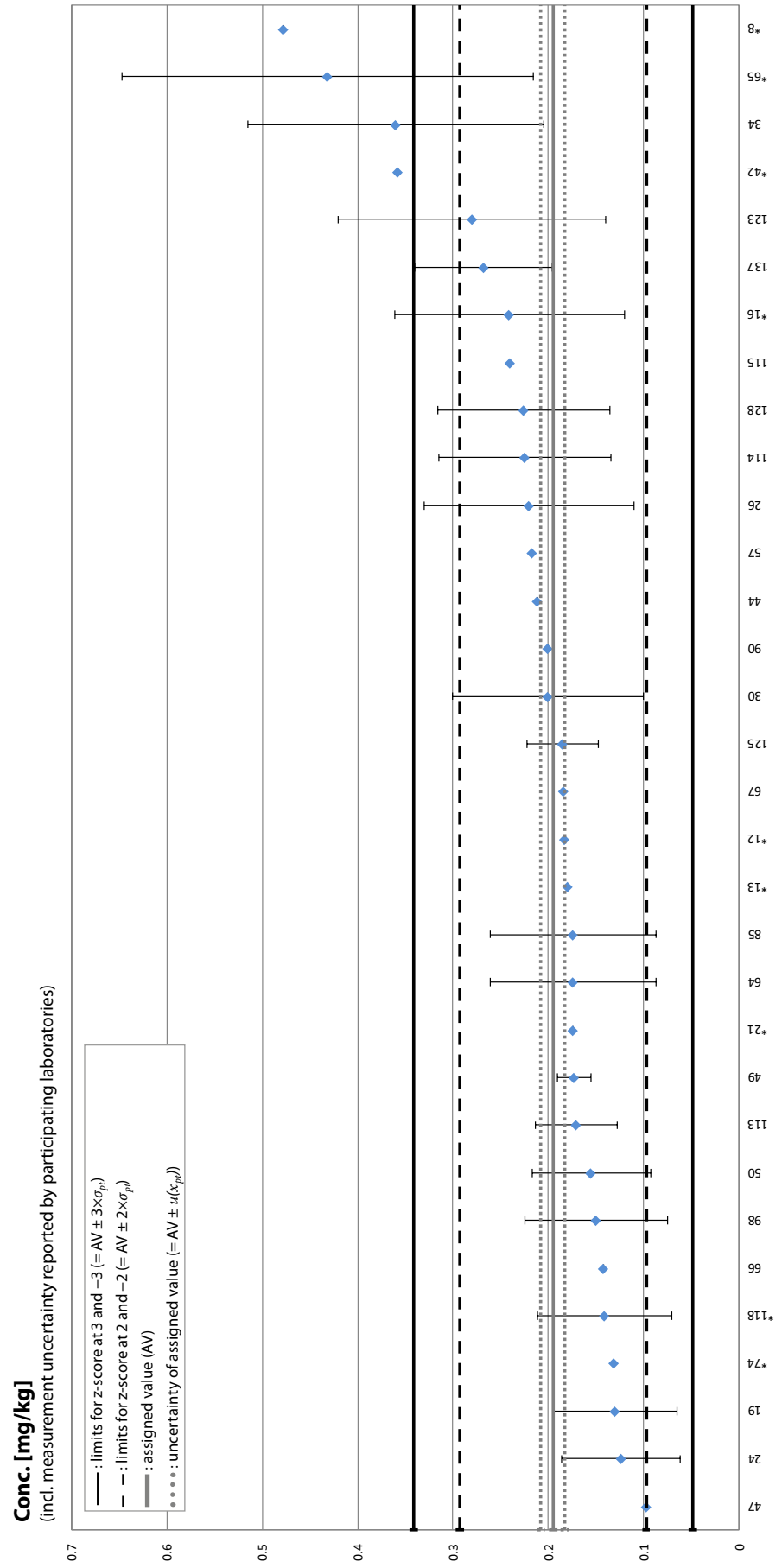
Appendix 6 (cont.) **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



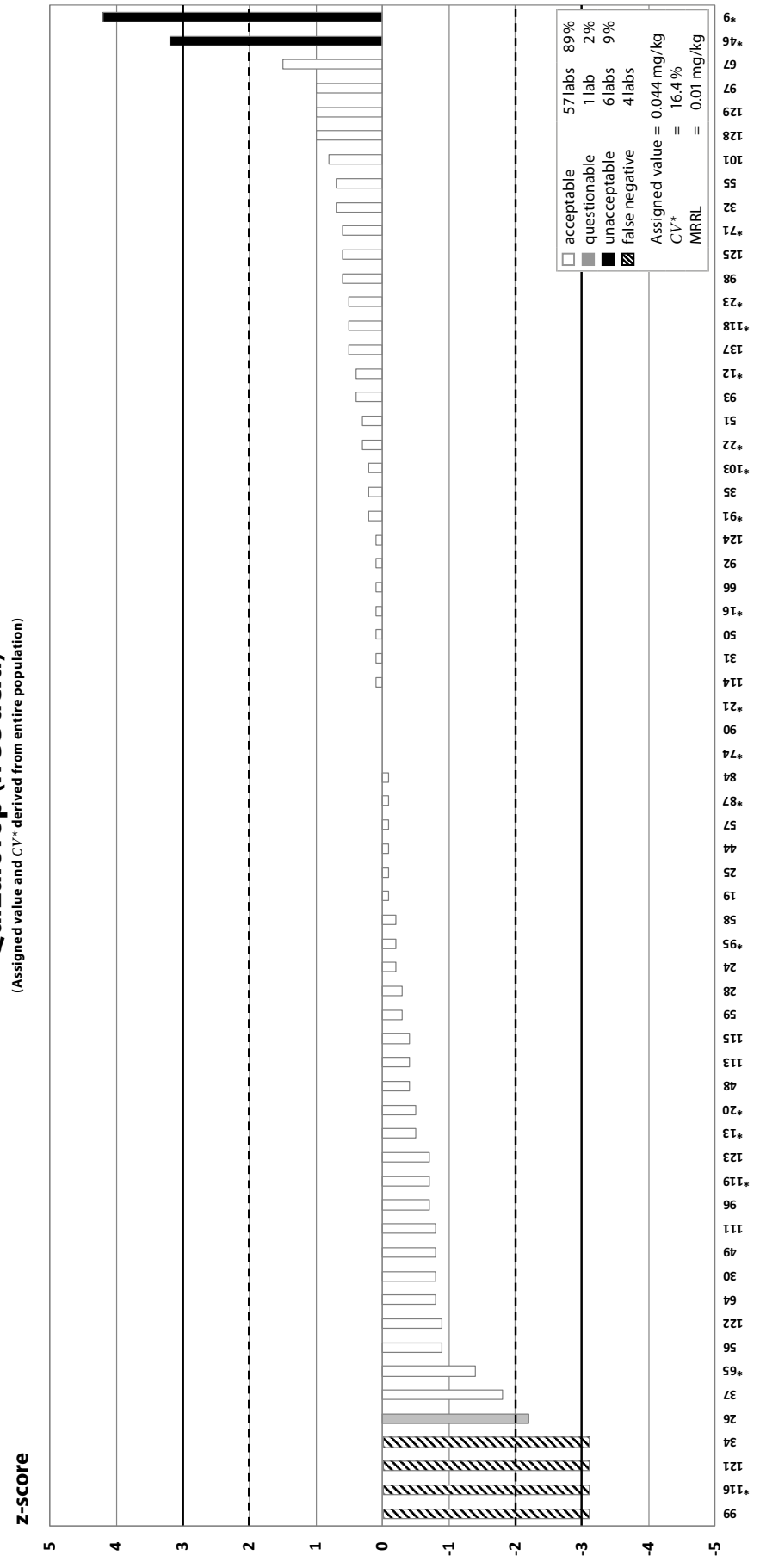
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



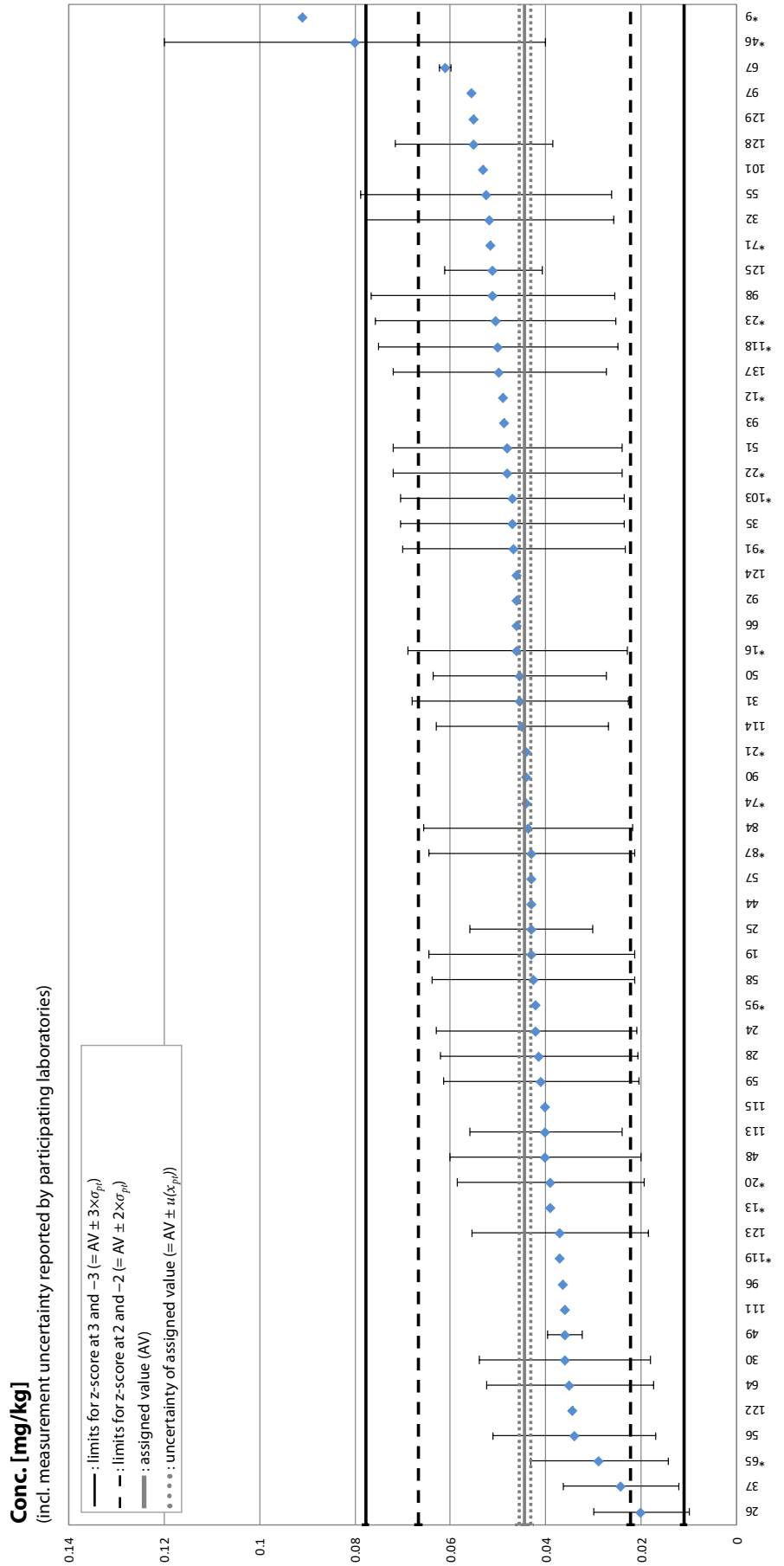
Appendix 6 (cont.) **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



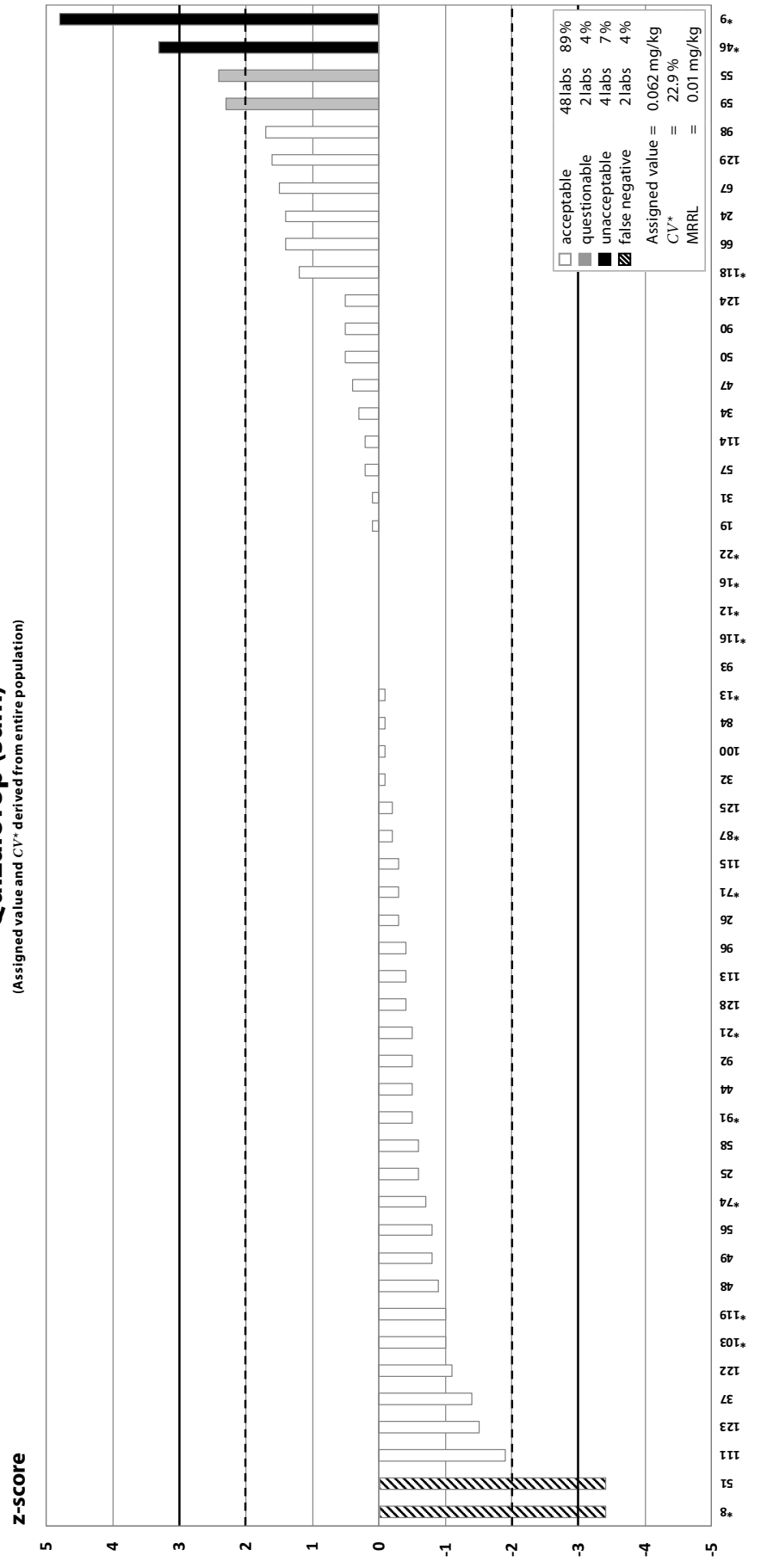
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



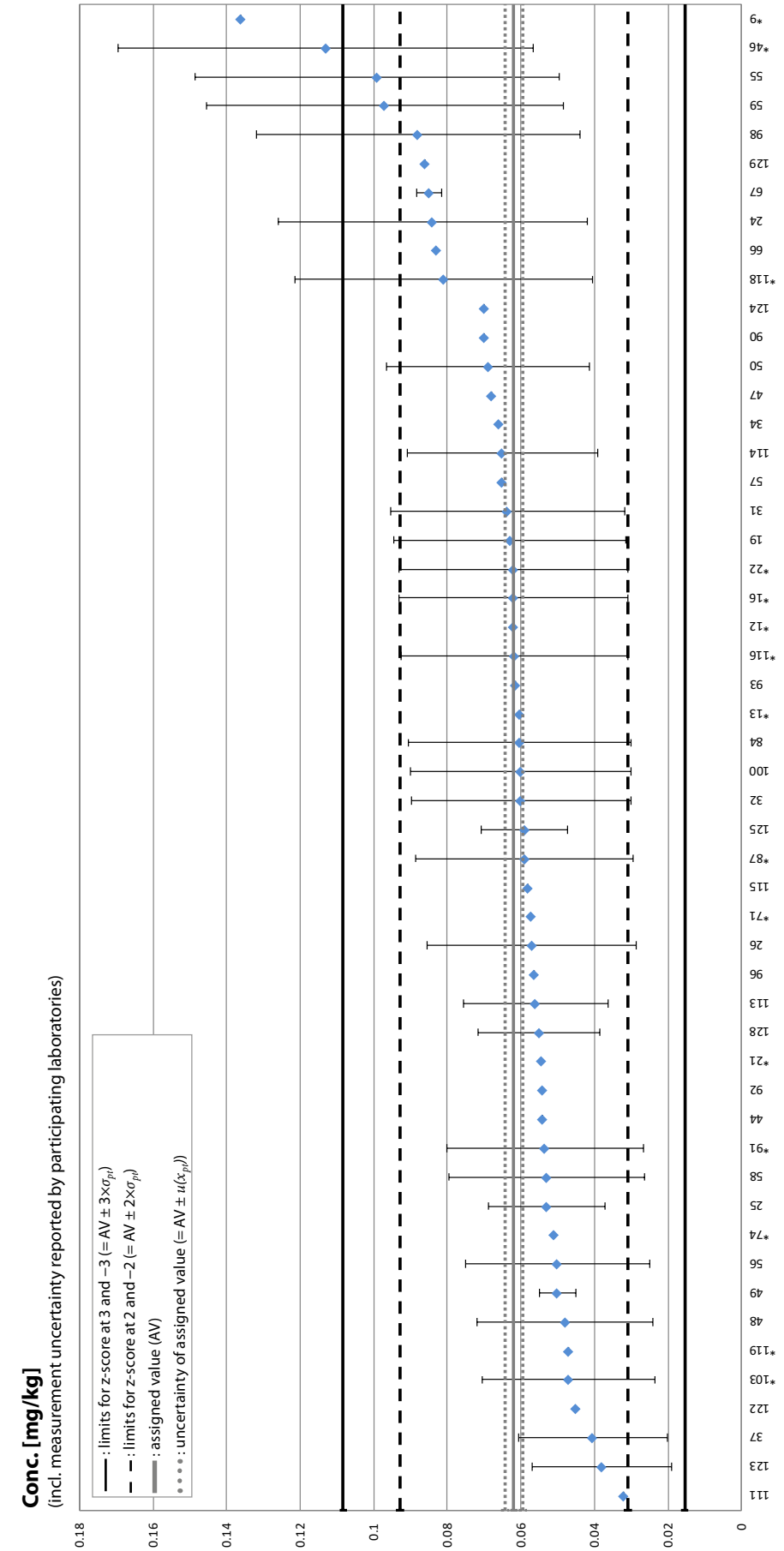
Appendix 6 (cont.) **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



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



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



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

A: Lack of experience
B: Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak; recovery too low; sensitivity too poor, RL<AV)
C: Analytical procedure was appropriate but it was not properly performed
D: Analyte losses during the procedure (e.g due to degradation, unfavourable partitioning, adsorption)
E: Measurement problems (e.g. poor chromatographic separation, poor sensitivity, signal interfered by matrix)
F: Misinterpretation / Misevaluation of measurement data
G: Inappropriate / erroneous calibration approach (e.g. Matrix effects not properly compensated)
H: Result not properly corrected for recovery
I: Calculation error (e.g. use of wrong factor, to express residue as required in PT; to address dilutions etc.)
J: Erroneous analytical standard (e.g. due to degradation, wrong purity, wrong dilution)
K: Deficient QC-measures that would have helped recognize that the method applied generates FNs, FPs or strongly biased results (e.g. no recovery test)
L: Transcription- / documentation- / communication-/ error
M: other
N: misunderstanding of RD

2,4-D (free acid) Assigned value: 0.052 mg/kg, CV^*: 20.8 %

LabCode	z-Score	Reason / Remarks	
5	-3.2 (FN)	L: Transcription-/ documentation-/ communication-/ error	L
33	-3.2 (FN)	B: Analytical procedure was inappropriate: Use of PSA (malpractice for acidic pesticide)	B
41	-3.2 (FN)	Code E Measurement problems (Analytical equipment which we use to determin 2,4-D is Agilent Technologies 7890 A GC System doesn't have so good sensitivity. Our RL is 0.05 mg/kg and we use double dilution. When we receive an analytical result under RL, we accept that it is negative.)	E
99	-3.2 (FN)	L. Transcription- / documentation-/ communication-/ error We do not analyse 2,4-D (free acid), we analyse 2,4-D (sum). The z-score of 2,4-D sum is -0,6.	L
75	-3.1	Result not properly corrected for recovery	H
62	-2.8	We used for analysis the original Quechers method with 2 salts only (withouth hydrolysis) as we use it routinely and in the previous tests we did not have any isues with it. The standards were checked and were OK). We did not track any error in measurment or evaluation. After we got our preliminary results we tried to check everything again and for control analysed some previous Fapas test with the same analytical procedure with fluazifop free acid and got pretty close result (116ug/kg - aceptable interval 58-150). Reanalysis of the test gave us result of 0,068 mg/kg.	?
121	-2.8	B: Analytical procedure was inappropriate	B
94	-2.5	Analytical procedure was inappropriate (hydrolysis conditions too weak)	B
66	2.8	OK with Organizer's Comments: Use of PSA (malpractice for acidic pesticide)	B
9	3.4	Measurement problems (signal interfered by matrix influence on lc-ms/ms performance, problem in system calibration)	E, G?
34	3.7	If calculated with new software without internal standard: +/- 0,06 mg/kg.	G?, I?

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

Carbofuran (sum) Assigned value: 0.107 mg/kg, CV^*: 22.8 %			
LabCode	z-Score	Reason / Remarks	
47	-3.0	no hydrolysis, nor sum of Carbofuran, Carbofuran-3-OH, Carbosulfan, Benfuracarb und Furathiocarb, Measurement problems, Communication error	B, E, L
38	-2.3	No hydrolysis. Effect of IS response? See e-mail explanation.	B
33	-2.2	B: Analytical procedure was inappropriate	B
39	-2.2	NO hydrolysis, no application mono method EURL. We haven´t carried out hydrolysis, so as the Organizer has commented, maybe that's the reason for the low value. I have reviewed previous proficiency tests where Carbofuran and 3-OH-carbofuran where present and we obtained without hydrolysis very good results (see attached Excel-sheet). I have no idea if the matrix (previous tests where fruits and vegetables) or the compounds you used for spiking the samples, where also influencing the necessity of hydrolysis here, compared to other tests. As far as I have seen, there are many laboratory with the same problem. As you can see in the attached word, we have performed the analysis by duplicate, but as far as the criteria for IS is accepted we always inform of the first sample (sample A). The response of the IS in sample A is 120% with respect to sample B, where the response of the IS compared to the calibration curve is 100%. That difference also influenced on the results, because as you can see in sample A a result of 0.046 mg/Kg was obtained, whereas the results for sample B is 0.064 mg/Kg. Both samples where obtained with the same procedure. Which z-score would be obtained with the results from sample B? Which internal standard may you recommend for the analysis of "carbofuran sum""?	B
66	-2.1	OK with Organizer's Comments: NO hydrolysis (may have contributed to result underestimation)	B
88	-2.1	maybe organizers' comments: NO hydrolysis (may have contributed to result underestimation)	(B)
110	-2.1	B: Analytical procedure was inappropriate	B
129	2.3	Assumption : Carbosulfan analytical standard is degraded (supplier order in progress for this analytical standard)	J?
55	2.8	J: Standard für die Kalibrierung war fehlerhaft (zu stark) verdünnt worden	J
64	3.6	Rootcause for the high concentration of carbofuran is due to the fact that the target pesticide list of SRM15, we had to include the concentration of carbosulfan. So the value of carbosulfan was also taken into account (expressed as carbofuran) and added to the concentration of carbofuran, hence elevating the concentration of carbofuran-sum. In routine analysis this situation does not occur since the residu definition does not include the concentration of carbosulfan. --> Question to SRM-organization: Did the other labs correctly apply these requirements? Since the individual reporting ofcarbosulfan was not part of the target pesticide scope.	s. private discussion with An
121	5.8	B: Analytical procedure was inappropriate	B
24	6.6	Analyse de routine au laboratoire: pas d'hydrolyse effectuée, sommation des valeurs individuelles. Nous n'avions pas la connaissance/expertise de la méthode d'hydrolyse acide au H_2SO_4 au moment du PT	A

Chlormequat-Cl Assigned value: 0.092 mg/kg, CV^*: 16.8 %			
LabCode	z-Score	Reason / Remarks	
107	-3.1	G: Inappropriate / erroneous calibration approach and H: Result not properly corrected for recovery	G, H
102	4.4	Lack of experience. Misevaluation of measurement data. Calculation error.	A, F, I
61	5.9	G (result reported was obtained with external calibration. Result with standard addition: 0,102mg/kg)	G
95	29.8	Code E - Measurement problems. We had multiple levels matrix matched calibration. Due to matrix effect the slope of the calbration curve was high. We have changed the method that if we have result of high concentrations then we have to check with different analytical column.	E

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

A: Lack of experience
B: Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak; recovery too low; sensitivity too poor, RL<AV)
C: Analytical procedure was appropriate but it was not properly performed
D: Analyte losses during the procedure (e.g due to degradation, unfavourable partitioning, adsorption)
E: Measurement problems (e.g. poor chromatographic separation, poor sensitivity, signal interfered by matrix)
F: Misinterpretation / Misevaluation of measurement data
G: Inappropriate / erroneous calibration approach (e.g. Matrix effects not properly compensated)
H: Result not properly corrected for recovery
I: Calculation error (e.g. use of wrong factor, to express residue as required in PT; to address dilutions etc.)
J: Erroneous analytical standard (e.g. due to degradation, wrong purity, wrong dilution)
K: Deficient QC-measures that would have helped recognize that the method applied generates FNs, FPs or strongly biased results (e.g. no recovery test)
L: Transcription- / documentation- / communication-/ error
M: other
N: misunderstanding of RD

Glyphosate Assigned value: 0.203 mg/kg, CV^*: 23.7 %

LabCode	z-Score	Reason / Remarks	
67	-3.6	documentation/transcription error, we determined 0.216 but unfortunately reported 0.0216	L
69	-3.6	The laboratory method used for glyphosate, AMPA and glufosinate, is based on derivatitation using FMOC. This method could be inappropriate for this matrix. A new procedure using QuPPe-PO method and hypercarb column will be implemented this year. (Pat: komische Erklärung. Wenn Fmoc im Überschuß ist, ist doch egal)	B (?)
107	-3.0	G: Inappropriate / erroneous calibration approach and H: Result not properly corrected for recovery	G, H
129	-2.8	H : Result not properly corrected for recovery	H
63	-2.2	Calculation error (use of wrong factor); it should be 0.184 mg/kg	I
51	4.0	Our glyphosate standard exceeded the expiry date, we ordered a new Glyphosate standard which lead to improved intensity by factor two	J

TFNA Assigned value: 0.060 mg/kg, CV^*: 27.8 %

LabCode	z-Score	Reason / Remarks	
51	-3.3 (FN)	Our extract was prepared with acetonitrile (QuEChERS). We did not use acidic QuEChERS.	D?
65	-3.3 (FN)	Very poor recoveries for TFNA in all grain/cereal samples using current technique. Known problem compound that requires method development works.	B
94	-3.3 (FN)	Analytical procedure was inappropriate (hydrolysis conditions too weak) Measurement problems (poor sensitivity)	B, E
75	-3.0	Result not properly corrected for recovery	H
121	2.2	B: Analytical procedure was inappropriate	B
128	2.8	I: Calculation Error: When calculating the final result, a value was unfortunately included that was significantly too high due to the incorrect addition of the internal standard. This was clearly marked, but unfortunately not taken into account in the calculation. It should be 0.071 mg/kg; new analysis on 08.06.2020: 0.065 mg/kg	I
50	9.9	The result should be 0.021 mg/kg instead of 0.21 mg/kg	L

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

2,4-D (sum) Assigned value: 0.059 mg/kg, CV^*: 18.9 %			
LabCode	z-Score	Reason / Remarks	
111	-2.4	(I) -due to sample weight used in the extraction results gained for the analytes by hydrolysis required a correction factor calculation. This was not done in error.	I
5	-2.1	L: Transcription-/ documentation-/ communication-/ error; B: Analytical procedure was inappropriate; E: Measurement problems	B, E, L
24	2.1	Très faible expérience de l'hydrolyse. La revue des données brutes, ne permet pas d'identifier une source d'erreur. La relance effectué lors d'un test d'un autre protocole donne une valeur beaucoup plus faible (<0,010 mg/kg).	A
44	2.1	Moderate hydrolysis	B
34	2.8	has to reanalysed "with Hydrolysis" after the instrument is set up (fire accident at the beginning of Mai)	B?
129	3.0	Analysis was conducted with 2 ml NaOH 5M addition (yes, two different hydrolysis conditions were carried out for extraction.)	?
62	4.3	Originaly we analysed sums with the hydrolysis step for fruit/and vegetables (300ulNaOH/30min) and got some lower results.The result for 2,4D was 0,045 mg/kg, 98% recovery). We also got low result for fluazifop sum (0,02), but we had som fluazifp butyl left (0,04). We did not even see the mecoprop sum. As soon as you released the new version for cereals we repeated this analysis with the stronger hydrolysis conditions (2ml NaOH/120min) and we got the higher results, we were able to evaluate the mecoprop either, the flazifop buthyl disapeared and we got the fluazifop sum result 0,073 mg/gk), but we got very high recovery rates in IRMs (130-151%). We tried to evaluate with different ways (standard adiotion, processed calibration, normal matrix matched calibration and got very similar results with all three. For lack of time we had not further investigations. We cannot explain what happened, because all other results perforemd the same way (for sums) were correct and better than the original results without the strong hydolysis. We would like to know your opinion, why the same method worked well for some of the analytes and give high result for 2,4D and haloxyfop sums.	s. private discussion with An
55	5.4	M/K: Zur alkalischen Hydrolyse wurde in unserem Labor bisher die AH+QuEChERS CRL-SRM (2007) angewendet, so auch bei der LVU-Probe. Dann erhielten wir im Verlauf der LVU mit der EURL-Mail vom 5.03.2020 die Information, dass nur mit der SRM-43 Ester vollständig hydrolisiert werden. Daraufhin wiederholten wir die Analysen mit SRM-43 AH-FA-QuECHERS und erhielten damit auch höhere Ergebnisse, die wir dann übermittelten. Zu diesem Zeitpunkt blieb uns aber bis zum Meldeschluss nicht mehr genügend Zeit, um die Ergebnisse durch Wiederfindungsexperimente zu validieren. Unser AH+QuEChERS CRL-SRM (2007)-Ergebnis lautete: 0,052 mg/kg	K, M
3rd-73	8.0	B (Analytical procedure was inappropriate; We changed the sample preparation according to the EURL-SRM method from 4-3-2020 (with 2ml 5M NaOH; 120 min on 400C and 1ml 5M H2SO4); First result (with 1ml 5M NaOH; 30 min on 400C and 1ml 5M H2SO4) was 0,06mg/kg)	B
33	8.8	A: Lack of experience	A
9	9.5	Measurement problems (signal interfered by matrix influence on lc-ms/ms performance, problem in system calibration)	E, G?

Bentazone Assigned value: 0.334 mg/kg, CV^*: 19.4 %			
LabCode	z-Score	Reason / Remarks	
24	-2.5	Analyse de routine au laboratoire avec Quechers classique et purification classique, la valeur indiquée a été rendue avec la prise en compte d'un rendement en conditions identique. La valeur obtenue sans purification au PSA donne effectivement une valeur de 0,218 mg/kg, valeur plus proche de la valeur cible	D
44	4.3	Misinterpretation / Misevaluation of measurement data	F
30	4.4	Possible: Code J. Internal mistakes during the analysis could not be found. A new standard solution was ordered to check the used standard solution."	J?

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

A: Lack of experience
B: Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak; recovery too low; sensitivity too poor, RL<AV)
C: Analytical procedure was appropriate but it was not properly performed
D: Analyte losses during the procedure (e.g due to degradation, unfavourable partitioning, adsorption)
E: Measurement problems (e.g. poor chromatographic separation, poor sensitivity, signal interfered by matrix)
F: Misinterpretation / Misevaluation of measurement data
G: Inappropriate / erroneous calibration approach (e.g. Matrix effects not properly compensated)
H: Result not properly corrected for recovery
I: Calculation error (e.g. use of wrong factor, to express residue as required in PT; to address dilutions etc.)
J: Erroneous analytical standard (e.g. due to degradation, wrong purity, wrong dilution)
K: Deficient QC-measures that would have helped recognize that the method applied generates FNs, FPs or strongly biased results (e.g. no recovery test)
L: Transcription- / documentation- / communication-/ error
M: other
N: misunderstanding of RD

Fluazifop (sum) Assigned value: 0.060 mg/kg, CV^*: 18.8 %

LabCode	z-Score	Reason / Remarks	
74	-3.3 (FN)	A - B (cf EURL comment)	A, B
91	-3.3 (FN)	Lack of experience, hydrolysis conditions too weak	A, B
3rd-134	-3.3 (FN)	L: Transcription- / documentation-/ communication-/ error. Our lab did not do the hydrolysis step	B, L?
51	-3.3	B: Analytical procedure was inappropriate	B
58	-3.3	We confirm the Organizer's Comments: A = Lack of experience; B = Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak)	A, B
8	-3.1	maybe organizers' comments: Weak hydrolysis (most likely contributed to the underestimated result)	(B)
31	-3.0	Yes, I agree with the organizers. However, we tried the both types of hydrolysis (our weak established and new strong from 2020), but unfortunately we got similar results, the both were below 20 ppb. I assume a random error. We will test it again and establish the procedure with strong hydrolysis in our lab.	?
68	-2.9	B: Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak; recovery too low; sensitivity too poor, RL<AV)	B
21	-2.8	maybe organizers' comments: Weak hydrolysis (most likely contributed to the underestimated result)	(B)
103	-2.8	maybe organizers' comments: Weak hydrolysis (most likely contributed to the underestimated result). Hydrolysis conditions do not match with reference method stated, please clarify.	(B)
46	-2.7	The problem regarding the false results has now been solved. The reason is that the blank-rice-matrix we used in a test acts differently compared to the rice-matrix we had as EUPT sample. Conserning some analytes, the suppression matrix-effect was dissimilar in our blank-rice compared to the EUPT rice sample. The suppression was even stronger when using more powerful hydrolysis. We have now conducted the test with the stronger hydrolysis by means of standard-addition-method. Fluazifop (sum) result (stronger hydrolysis, standard addition) is 0,066 mg/kg	"M? diff. Behaviour of blank"
114	2.1	It was realized a weak Acidic Hydrolysis during extraction (10min, 40ºC and 5N). The working solution (mixture) stability was revised and we obtained a -7,7 of difference respect the new working solution for Fluacifop. It´s probably that this light difference (although less than 10), produce an overestimate results. The proficiency test was re-analyzed with a new stock and working solution and it was obtained a result of 0.070 mg/Kg.	B, J
9	2.4	Measurement problems (signal interfered by matrix influence on lc-ms/ms performance, problem in system calibration)	E, G?

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

Haloxyfop (sum) Assigned value: 0.151 mg/kg, CV^*: 16.2 %			
LabCode	z-Score	Reason / Remarks	
19	-3.7 (FN)	I have deleted the wrong parameter in the result table. If transmitted correctly our result for Haloxyfop would have been 0,165 mg/kg with an recovery rate of 108 %	L
3rd-134	-3.7 (FN)	L: Transcription- / documentation-/ communication-/ error. Our lab did not do the hydrolysis step	B, L?
103	-2.8	maybe organizers' comments	(B)
30	-2.7	Code L: The wrong result was submitted. The correct result was 0,129 mg/kg.	L
41	-2.3	Code A Lack of experience	A
111	-2.2	(I)-due to sample weight used in the extraction results gained for the analytes by hydrolysis required a correction factor calculation. This was not done in error.	I
62	2.1	Please see explanation under 2,4-D (sum)	s. private discussion with An
24	3.1	Très faible expérience de l'hydrolyse. La revue des données brutes, ne permet pas d'identifier une source d'erreur. La relance effectué lors d'un test d'un autre protocole donne une valeur beaucoup plus plus proche de la valeur cible, soit 0,156 mg/kg	A

Imazethapyr (free acid) Assigned value: 0.206 mg/kg, CV^*: 17.6 %			
LabCode	z-Score	Reason / Remarks	
115	-3.8 (FN)	this residue is being development by R&D department and had not been added to our routine scope. So, we did not look for it--> this is an error when filling our scope	L
3rd-135	-3.8 (FN)	B (recovery too low), leading to L (result shouldn't have been reported as "not detected", but "not tested"; it wouldn't be reported as well in routine condition)	B, L
98	-3.5	measured value is 0,236 mg/kg. mistake by filling results in the portal	L
26	-2.2	our problem was the use of PSA, after check the sample again we get the right result. 0.1965 mg/kg	B
114	2.3	The working solution (mixture) stability was revised and we obtained a -9,7 of difference respect the new working solution for Imazethapyr. It´s probably that this light difference (although less than 10), produce an overestimate results. The proficiency test was re-analyzed with a new stock and working solution and it was obtained a result of 0.23 mg/Kg. It was analyzed without hydrolisis because the definition of residue is only free.	J
90	2.8	The sample was analyzed with hydrolisis (80ºC, 60 min, 300 uL NaOH). This compound is included in a method of acid herbicides although the definition of residue is only free. The proficiency test was re-analyzed without hydrolysis and was obtained a result of 0.18 mg/kg.	B
62	2.1	Please see explanation under 2,4-D (sum)	s. private discussion with An
24	3.1	Très faible expérience de l'hydrolyse. La revue des données brutes, ne permet pas d'identifier une source d'erreur. La relance effectué lors d'un test d'un autre protocole donne une valeur beaucoup plus plus proche de la valeur cible, soit 0,156 mg/kg	A

MCPA (sum) Assigned value: 0.068 mg/kg, CV^*: 20.2 %			
LabCode	z-Score	Reason / Remarks	
34	-3.4 (FN)	has to reanalysed "with Hydrolysis" after the instrument is set up (fire accident at the beginning of Mai)	B?
111	-2.1	(I) -due to sample weight used in the extraction results gained for the analytes by hydrolysis required a correction factor calculation. This was not done in error.	I
55	4.5	M (other)/K (Deficient QC-measures): Details s. explanation unter 2,4-D. Unser AH+QuEChERS CRL-SRM (2007)-Ergebnis lautete: 0,086 mg/kg	K, M
9	8.2	Measurement problems (signal interfered by matrix influence on lc-ms/ms performance, problem in system calibration)	E, G?
33	8.5	A: Lack of experience	A

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

A: Lack of experience
B: Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak; recovery too low; sensitivity too poor, RL<AV)
C: Analytical procedure was appropriate but it was not properly performed
D: Analyte losses during the procedure (e.g due to degradation, unfavourable partitioning, adsorption)
E: Measurement problems (e.g. poor chromatographic separation, poor sensitivity, signal interfered by matrix)
F: Misinterpretation / Misevaluation of measurement data
G: Inappropriate / erroneous calibration approach (e.g. Matrix effects not properly compensated)
H: Result not properly corrected for recovery
I: Calculation error (e.g. use of wrong factor, to express residue as required in PT; to address dilutions etc.)
J: Erroneous analytical standard (e.g. due to degradation, wrong purity, wrong dilution)
K: Deficient QC-measures that would have helped recognize that the method applied generates FNs, FPs or strongly biased results (e.g. no recovery test)
L: Transcription- / documentation- / communication-/ error
M: other
N: misunderstanding of RD

MCPB (sum) Assigned value: 0.057 mg/kg, CV^*: 29.4 %

LabCode	z-Score	Reason / Remarks	
30	-3.3 (FN)	"Code M and L Analysis without hydrolysis . Internal procedure prescribes doing a second analysis with hydrolysis when first result reaches the internal stated limit value. Wrong submitted as ""sum"". (Pat: Das Labor hat nicht verstanden, daß man bei Säure kein PSA cleanup durchführen darf! Ich habe die Erklärung nicht wirklich verstanden.)"	L, M
34	-3.3 (FN)	has to reanalysed "with Hydrolysis" after the instrument is set up (fire accident at the beginning of Mai)	B?
91	-3.3 (FN)	Lack of experience, hydrolysis conditions too weak	A, B
137	-3.3 (FN)	maybe organizers' comments: Weak hydrolysis (most likely contributed to the FN result)	(B)
74	-2.7	A - B (cf EURL comment)	A, B
8	-2.6	maybe organizers' comments: Weak hydrolysis (most likely contributed to the FN result)	(B)
25	-2.5	A: Lack of experience, maybe also B: Analytical procedure was inappropriate	(B)
51	-2.5	B: Analytical procedure was inappropriate	B
21	-2.4	maybe organizers' comments: Weak hydrolysis (most likely contributed to the FN result)	(B)
71	-2.4	B Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak; sensitivity too poor)	B
111	-2.4	(I) -due to sample weight used in the extraction results gained for the analytes by hydrolysis required a correction factor calculation. This was not done in error.	I
68	-2.2	B: Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak; recovery too low; sensitivity too poor, RL<AV)	B
99	2.3	"We have analysed again the sample and result is lower. We are going to check the reproducibility of this pesticide."	?
113	4.2	"L - by misunderstanding, legal RD used (residue definition according to the Reg. 396/2005/EC) Due to this misunderstanding, also result reported for MCPA is biased. Could You please kindly consider these (high) numbers as a transcription error (caused by misunderstanding of RD) and to replace both values by recalculated results indicated in the form ? (Pat: not L, but N: missunderstanding of RD! Und ""NO, we can't accept the recal. results.)"	N
33	4.6	A: Lack of experience	A
55	5.4	M (other)/K (Deficient QC-measures): Details s. explanation unter 2,4-D. Unser AH+QuEChERS CRL-SRM (2007)-Ergebnis lautete: 0,044 mg/kg	K, M

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

Mecoprop (sum) Assigned value: 0.067 mg/kg, CV^*: 22.4 %			
LabCode	z-Score	Reason / Remarks	
8	-3.4 (FN)	maybe organizers' comments: Weak hydrolysis (most likely contributed to the FN result)	(B)
21	-3.4 (FN)	maybe organizers' comments: Weak hydrolysis (most likely contributed to the FN result)	(B)
24	-3.4 (FN)	Très faible expérience de l'hydrolyse. La revue des données brutes, ne permet pas d'identifier une source d'erreur. Une valeur aux alentours de 0,004 mg/kg (traces) a été retrouvé au départ. La relance effectué lors d'un test d'un autre protocole donne une valeur de l'ordre de traces également. Une plus forte hydrolyse, telle qu'indiquée dans le protocole eurl (04/03/2020) devra être testé.	A
30	-3.4 (FN)	Code M and L Analysis without hydrolysis . Internal procedure prescribes doing a second analysis with hydrolysis when first result reaches the internal stated limit value. Wrong submitted as "sum". (Pat: Das Labor hat nicht verstanden, daß man bei Säure kein PSA cleanup durchführen darf! Die ERklärung habe ich nicht wirklich verstanden.)"	L, M
34	-3.4 (FN)	has to reanalysed "with Hydrolysis" after the instrument is set up (fire accident at the beginning of Mai)	(B)
37	-3.4 (FN)	Code A, the alcaline hydrolysis is not validated in our Lab, we habe no experience, how strong the hydrolysis is needed for Mecoprop (sum) , our result for Mecoprop is 0,012 mg/kg - therefore <RL.	A, B
46	-3.4 (FN)	The problem regarding the false results has now been solved. The reason is that the blank-rice-matrix we used in a test acts differently compared to the rice-matrix we had as EUPT sample. Conserning some analytes, the suppression matrix-effect was dissimilar in our blank-rice compared to the EUPT rice sample. The suppression was even stronger when using more powerful hydrolysis. We have now conducted the test with the stronger hydrolysis by means of standard-addition-method. Mecoprop (sum) result is 0,097 mg/kg	"M? diff. Behaviour of blank"
47	-3.4 (FN)	hydrolysis condition too weak, Mecoprop free acid not analysed due to communication problems during the corona pandemic	B
51	-3.4 (FN)	B: Analytical procedure was inappropriate	B
64	-3.4 (FN)	The residue definition of mecoprop does not include esters, so these esters should not be analyzed in routine analysis. Actually we should have only reported the mecoprop free acid, since our method is not optimized for the mecoprop-ester analysis. On the other hand we will re-evaluate our method in order to perform a hydrolysis at 40°C.	B
66	-3.4 (FN)	OK with Organizer's Comments: Weak hydrolysis (most likely contributed to the FN result)	B
68	-3.4 (FN)	B: Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak; recovery too low; sensitivity too poor, RL<AV)	B
74	-3.4 (FN)	A - B (cf EURL comment)	A, B
91	-3.4 (FN)	Lack of experience, hydrolysis conditions too weak	A, B
103	-3.4 (FN)	maybe organizers' comments: Weak hydrolysis (most likely contributed to the FN result). Hydrolysis conditions do not match with reference method stated, please clarify.	(B)
119	-3.4 (FN)	B/C : Analytical procedure was inappropriate /Analytical procedure was appropriate but it was not properly performed: - Hydrolysis procedure was weak 30min, T° ambiente, 300 µl 5N NaOH - Moreover Inadervertently H2SO4 was added before hydrolysis step We repeat experiments with 2ml NaOH 5N, 120 min, 40°C => it is ok"	B, C
122	-3.4 (FN)	L: Transcription- / documentation-/ communication-/ error. Value found: 0,063 mg/kg. We did not transcript the value due to our error during the result submission.	L
137	-3.4 (FN)	maybe organizers' comments: Weak hydrolysis (most likely contributed to the FN result)	(B)
56	-3.2	Lack of experience (first time hydrolysis tested for this new complex definition).	A, B
111	-3.2	(I) -due to sample weight used in the extraction results gained for the analytes by hydrolysis required a correction factor calculation. This was not done in error. (B)- Hydrolysis conditions too weak	B, I
116	-3.1	Ester was NOT used for calibration curve. The hydrolysis was only performed on the sample to verify the presence of the ester.	B
93	-3	L: Transkription Error: Unfortunately there was transcription error. You can see the attachment file (EUPT-SRM15_Measurement result_Mecoprop sum.xlsx). I sent mecoprop (sum) result 0.0171 mg/kg, but the real result is 0.071	L

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

A: Lack of experience
B: Analytical procedure was inappropriate (e.g. hydrolysis conditions too weak; recovery too low; sensitivity too poor, RL<AV)
C: Analytical procedure was appropriate but it was not properly performed
D: Analyte losses during the procedure (e.g due to degradation, unfavourable partitioning, adsorption)
E: Measurement problems (e.g. poor chromatographic separation, poor sensitivity, signal interfered by matrix)
F: Misinterpretation / Misevaluation of measurement data
G: Inappropriate / erroneous calibration approach (e.g. Matrix effects not properly compensated)
H: Result not properly corrected for recovery
I: Calculation error (e.g. use of wrong factor, to express residue as required in PT; to address dilutions etc.)
J: Erroneous analytical standard (e.g. due to degradation, wrong purity, wrong dilution)
K: Deficient QC-measures that would have helped recognize that the method applied generates FNs, FPs or strongly biased results (e.g. no recovery test)
L: Transcription- / documentation- / communication-/ error
M: other
N: misunderstanding of RD

Mecoprop (sum) Assigned value: 0.067 mg/kg, CV^*: 22.4 %

LabCode	z-Score	Reason / Remarks	
67	-2.9	analytical procedure was inappropriate, hydrolysis conditions too weak hydrolysis was repeated: 2 ml 5N NaOH, 120 min, 40 °C --> new value: 0,0696 mg/kg"	B
12	-2.8	B: hydrolysis conditions too weak, we have reanalysed the sample and our result is 0,064 mg/kg.	B
27	-2.6	Hydrolysis was performed with 1 ml 5N NaOH (not 1N as stated in column "Hydrolysis Concentration") at 40 °C for 120 min. You suggest an amount of 2 ml for Mecoprop in rice (see table 7 in https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EurlSrm_Observation_alkaline_hydrolysis_acidic_herbicides.pdf). Also: lack of experience"	B
100	-2.5	maybe organizers' comments: Weak hydrolysis (most likely contributed to the underestimated result). Hydrolysis conditions do not match with reference method stated, please clarify.	(B)
123	-2.5	Result not properly corrected for recovery	H
44	-2.2	Moderate hydrolysis	B
84	-2.2	Lack of experience; hydrolysis conditions too weak	A, B
55	4	M/K: s. Explanation under 2,4-D. Unser AH+QuEChERS CRL-SRM (2007)-Ergebnis lautete: 0,070 mg/kg	K, M
33	8	A: Lack of experience	A

Paraquat Assigned value: 0.195 mg/kg, CV^*: 28.9 %

LabCode	z-Score	Reason / Remarks	
51	-3.8 (FN)	At the usual retention time we did not detect paraquat. Now we check the column and the standard.	E
3rd-73	-3.8 (FN)	L (communication error; we didn't put the result for Paraquat in table)	L
99	-3.6 (FN)	L. Transcription- / documentation-/ communication-/ error Paraquat is not included in the accreditation scope of the laboratory, we have not analised this pesticide.	L
42	3.4	No experience with this compound. Errors in the concentration of standard solutions . We used expired analitycal standard (2015) and glass volumetric flask for the preparation of stock and working solutions. The solvent used for preparation of the standard solutions was methanol+1% formic acid.	A, C, J
34	3.5	has to reanalysed after the instrument is set up (fire accident at the beginning of Mai)	B?
65	5	Not routinely analysed. Calculation error, standard concentrations were not corrected for the taregt analyte Paraquat (dication). When corrected a z-score of 2 is obtained.	A, I

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

Quizalofop (free acid) Assigned value: 0.044 mg/kg, CV^*: 16.4 %			
LabCode	z-Score	Reason / Remarks	
34	-3.1 (FN)	has to reanalysed "with Hydrolasis" after the instrument is set up (fire accident at the beginning of Mai)	B?
99	-3.1 (FN)	L. Transcription- / documentation-/ communication-/ error There was an error to send the results, we sent "not detected". We detected 0,05 mg/Kg of quizalofop (free acid)and 0,07 mg/Kg of quizalofop (sum)"	L
116	-3.1 (FN)	In our laboratory it´s validated and accredited at 0.05 mg/kg. We need to re-validate the analyte at lower level and do the analyses on a more sensitive instrument.	E
121	-3.1 (FN)	B: Analytical procedure was inappropriate	B
26	-2.2	our problem was the use of PSA, after check the sample again we get the right result. 0.040 mg/Kg	B
46	3.2	The problem regarding the false results has now been solved. The reason is that the blank-rice-matrix we used in a test acts differently compared to the rice-matrix we had as EUPT sample. Conserning some analytes, the suppression matrix-effect was dissimilar in our blank-rice compared to the EUPT rice sample. The suppression was even stronger when using more powerful hydrolysis. We have now conducted the test with the stronger hydrolysis by means of standard-addition-method. Quizalofop (sum) result is 0,062 mg/kg"	"M? diff. Behaviour of blank"
9	4.2	Measurement problems (signal interfered by matrix influence on lc-ms/ms performance, problem in system calibration)	E, G?

Quizalofop (sum) Assigned value: 0.062mg/kg, CV^*: 22.9 %			
LabCode	z-Score	Reason / Remarks	
51	-3.4 (FN)	We had a mistake in the data transmission. Our result would have been 0.061 mg/kg (n=3)	L
59	2.3	unknown / A Lack of experience	A
55	2.4	M (other)/K (Deficient QC-measures): Details s. explanation unter 2,4-D. Unser AH+QuEChERS CRL-SRM (2007)-Ergebnis lautete: 0,070 mg/kg	K, M
46	3.3	The problem regarding the false results has now been solved. The reason is that the blank-rice-matrix we used in a test acts differently compared to the rice-matrix we had as EUPT sample. Conserning some analytes, the suppression matrix-effect was dissimilar in our blank-rice compared to the EUPT rice sample. The suppression was even stronger when using more powerful hydrolysis. We have now conducted the test with the stronger hydrolysis by means of standard-addition-method. Quizalofop (sum) result is 0,062 mg/kg	"M? diff. Behaviour of blank"
9	4.8	Measurement problems (signal interfered by matrix influence on lc-ms/ms performance, problem in system calibration)	E, G?

9th Edition: Released on 15 November 2019

GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues in Food and Feed

Introduction

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

The following four EURLs for pesticide residues were appointed by DG-SANTE based on regulation 882/2004/EC that was repealed by regulation 625/2017/EC[3]:

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

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

[1] DG-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.

9th Edition: Released on 15 November 2019

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 collaboration.

An Organising Team (in the following named Organisers) 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, the production of the PT-material (Test Item), the undertaking of homogeneity and stability tests, the packing and shipment of the PT-materials, the handling and evaluation of the results and method information submitted by the participants, the drafting of the preliminary and final reports as well as generation and distribution of EUPT-participation certificates.

To complement the internal expertise of the EURLs, a group of external consultants forming 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 and the affiliation of each of its members is shown on the EURL-Website. The members of the EUPT-SC are also 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).

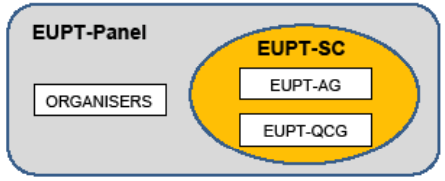
The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and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

[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-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 approximate concentrations at which they should be present.

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 assist 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.

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 38 (b) of Reg. 625/2017/EC and 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. 625/2017/EC (for all NRLs)

[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. 625/2017/EC. This includes labs involved in controls within the framework of national and/or EU-controlled programmes as well as labs involved in import controls according to Regulation 669/2009/EC.

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.

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

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 This also applies to any participating laboratories that fail to report results.

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 corresponding NRL 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/EC, NRLs are responsible for evaluating and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.

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

For each EUPT the organising EURL prepares a specific EUPT-Website where all PT-relevant documents in their latest version are linked. In case of important modifications on any of these documents, the participating laboratories will be informed via e-mail. In any case, as soon as the PT-period starts the participants are encouraged to visit the particular EUPT-Website, to make sure that they are using the latest versions of all PT-relevant documents.

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 for, 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.

EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

9th Edition: Released on 15 November 2019

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-Panel, considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution of the participants' results (CV^{wb}), 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. For certain analytes with comparable properties, an equivalent distribution within the sample can be expected if they were spiked/used at simultaneously. The homogeneity test, of one or more of these analytes, may thus be skipped or simplified. If, however, the distribution of participants' results for an analyte that was not or not fully tested for homogeneity, is found to be atypically broad, compared to the tested analytes, the EUPT-SC may decide that a homogeneity test should be performed a posteriori by the EURL.

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-Panel will finally decide whether

analytes for which the stability test was not undertaken will be included in the Final EUPT-Report, considering all relevant aspects such as the distribution of the participant's results (CV%).

A pesticide is considered to be adequately stable if $|\bar{y}_1 - \bar{y}| \leq 0.3 \times \sigma_{pt}$, with $\bar{y}_1$ being the mean value of the results of the last phase of the stability test, $\bar{y}$ being the mean value of the results of the first phase of the stability test and σ_{pt} being 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.

Stability during shipment: 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 keep track of the shipment duration and then decide whether it is reasonable to 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 by the EUPT-Panel considering all relevant aspects including the duration and conditions of the shipment to the laboratory as well as the feedback by the 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. Laboratories should not report results below their reporting limits.

Correction of results for recovery

Correction of results for recovery is recommended if the average recovery rate significantly deviates from 100 % (typically if outside the 80–120% range). Approaches for recovery correction explicitly stated in the DG-SANTE document are

a) the use of recovery correction factors,
b) the use of stable isotope labelled analogues of the target analytes as Internal Standards (ILISs),
c) the 'procedural calibration' approach as well as
d) 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. If one or more of the approaches b), c) and d) were employed, in which correction for recovery is inherent to the procedures, the apparent recovery figures obtained during validation experiments are not mandatory, and the approached followed are to be reported in the appropriate fields within the data submission tool.

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 EUPT-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 Organisers reserve 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 the robust estimate of the

EURL EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

9th Edition: Released on 15 November 2019

participant's mean (x*) as described in ISO 13528:2015[4], 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 or similar compounds in the past.

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

Before estimating the assigned value, results associated with obvious mistakes have to be examined to decide whether they should be removed from the population. Such gross errors may include incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits, incorrect unit), calculation errors (e.g. missing factors), analysis of a wrong sample/extract (e.g. a spiked blank), use of wrong concentrations of standard solutions, incorrect data processing (e.g. integration of wrong peak), inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate analytical steps or procedures that demonstrably lead to significantly biased results (e.g. employing inappropriate internal standards or analytical steps or conditions leading to considerable losses, due to degradations, adsorptions, incomplete extractions, partitioning etc.). 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").

Results reported by laboratories from non EU member states are typically excluded from the population that is used to derive the assigned value (see also "Estimation of the assigned value").

[4] 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).

– ***Uncertainty of the assigned value***

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

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

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).

Based on experience from previous EUPTs9, a percentage FFP-RSD of 25 % is currently used for all analyte-matrix combination, with the target standard deviation being calculated as follows:

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

The EUPT-Panel reserves the right to also employ other FFP-RSDs or other approaches for setting the assigned value 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*) of the participants results is calculated according to ISO 13528:2015; Chapter 7.7 following Algorithm A in Annex C (so called "consensus approach").

[9] 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), 7809-7819.

– ***z scores***

This parameter is calculated using the following formula:

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

where x_i is the value reported by the laboratory, x_{pt} is the assigned value, and FFP-σ_{pt} is the standard deviation using the 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 the combined z-scores will be 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[10]:

$\lvert z \rvert \leq 2.0$	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 RL < MRRL. Where, using this approach, the calculated z scores for false negatives are > −3 (still questionable), they will be fixed at −3.5 to underline that these are unacceptable results. These z-scores will typically appear in the z-score histograms and used in the calculation of combined z-scores.

– **Collection of measurement uncertainty (MU) figures**

The participating labs will be asked to report the MU figure they would routinely report with each EUPT result. The EUPT-Panel will decide whether and how to evaluate these figures and whether indications will be made to the laboratories in this respect.

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

– ***Category classification***

The EUPT-Panel will decide if and how to classify the laboratories into categories based on their scope and/or performance. Currently a scope-based classification into Category A and Category B is employed. Laboratories that a) are able to analyse at least 90% of the compulsory pesticides in the target pesticides list, b) have correctly detected and quantified a sufficiently high percentage of the pesticides present in the Test Item (at least 90 %) and c) reported no false positives, will have demonstrated 'sufficient scope' and will be therefore 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	

The EUPT-Panel reserves the right to develop and apply alternative classification rules.

– ***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)[11,12] (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 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. The z-scores appointed to false negatives will be also included in the calculation of the combined z-scores.

Laboratories within Category B will be typically ranked according to the total number of pesticides 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.

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

[12] 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.

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 EUPT-Report may be published up to 10 months after the deadline for results submission. Results submitted by non-EUMEFTA laboratories might not always be used in the tables or figures in the Final EUPT-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 Categories.

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 like. The online version of the Final EUPT 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. In exceptional cases, follow-up activities may even be indicated for results within $|z| \leq 2.0$ (e.g. where two errors with opposed tendency cancel each other leading to acceptable results).

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 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 (AAZ higher than 1.3 for SRM-compounds) 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[13]:

Phase 1:

- Identifying the origin of the bad results (failure in EUPTs).

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

Appendix 8 (cont.) General EUPT Protocol (9th Ed.)

- 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.

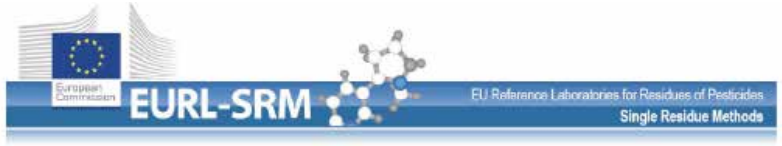
Phase 2:

- 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 CRLs 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 15th EU Proficiency Test
on Pesticides requiring Single Residue Methods
EUPT – SRM15 (2020)
(update on 17 March 2020)

Introduction

This protocol is complementary to the valid version of the "General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed, Ed. 9" covering all EUPTs in 2020.
The EUPT-SRM15 is organized by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM) that is accredited according to ISO 17043 as a provider of proficiency tests (please see EURL-SRM accreditation).

The EUPT-SRM15 deals with the analysis of SRM-pesticides in rice flour 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) performing pesticide residue analyses of cereals or feeding stuff within the frame of National and EU official controls, see Art 38 (b) of Reg. (EC) 625/2017 and Art. 28 of Reg. (EC) 396/2005. The most important documents related to this PT can be accessed via the EUPT-SRM15-Website.

Laboratories were classified into those that are tentatively obliged and those tentatively non-obliged to participate in the present PT, based on information within the EURL DataPool. NRL-SRMs and OfLs performing pesticide residue analyses of cereals or feeding stuff, within the frame of National and EU official controls, were considered as being obliged to participate. Prior to classification the laboratories were asked to update this information. This tentative classification was only based on the commodity scope (not the pesticide scope) of the laboratories and was also visible to the participants during the PT registration process. OfLs listed as "obliged to participate in the EUPT-SRM15" but not intending to participate had to state their reasons for non-participation during the online registration of the EUPT-SRM15. The registration period lasted from 25 November till 23 December, 2019. The feedback received during registration, especially details considering the scope, will be considered in the final list of obliged laboratories.

Test Item

The Test Item of this EUPT is Rice Flour.

Participants will receive one bottle Test Item containing 150 – 200 g rice flour with incurred and spiked analytes from the Target Pesticides List. NO Blank material will be sent to the participants for this PT.

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.

Target Analytes and MRRLs

The Test Item will contain several pesticides from the EUPT-SRM15 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-SRM15 Target Pesticides List before starting with analysis and result reporting.

Shipment of Test Item

Test Item is planned to be shipped on 10 February, 2020.
Test Item will be packed in thermo-boxes together with freeze gel packs and shipped from Germany via DHL Express to the participants. Prior to shipment a reminder will be sent to the participating laboratories by e-mail.
The participating laboratories must make their own arrangements for the receipt of the package. They should inform the Organizers 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.

Where complications during customs clearance or shipment are expected, the participating laboratories should provide the Organizers in advance with suitable contact information (e.g. mobile phone numbers of laboratory personnel) as well as instructions in local language explaining the need to keep the package in the freezer in case of delays during shipment or customs clearance. This information will be attached to the package.

After the packages is 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 and must make any necessary arrangements to receive the delivery.

In case of unusual delays in the customs or within the recipient's country, the participants themselves are strongly encouraged to contact the local DHL Express office and/or the customs in order to help accelerate the clearance and delivery procedures.

Instructions on handling the Test Item

Once arrived, the Test Item should be stored deeply frozen (at –18°C or lower) until analysis in order to avoid any possible deterioration/spoilage of the sample material and to minimize analyte losses.

Before analytical portions are taken for analysis, the Test Item should be mixed thoroughly in its entirety.

Participating laboratories are recommended using 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-SRM15 result submission webtool.

The homogeneity tests will be conducted using 5 g for both 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 portion size used in the homogeneity test. Where smaller sample portions are employed, there will be uncertainty as to whether the portion-to-portion variability is still acceptable

Results submission webtool

Sample receipt acknowledgement, analytical results and method information are to be submitted via the EUPT-SRM15 result submission webtool:

- Sample receipt acknowledgement: accessible from 10 February and should be complete by 19 February.
- Analytical results and method information: accessible from 10 February till 10 March.
- The deadline for result submission is 31 March, 11.30 pm (CEST), 2020.
- Additional information on the methods used for tentatively false negative results: accessible from 1 April till 9 April, 2020.

A guideline for the new EUPT-SRM15 result submission webtool will be provided to the participants in due time. The participants are urged to read it carefully before submitting their results.

- Login credentials and lab code

To access the EUPT-SRM15 result submission webtool, participants must use their personal login credentials (username and password). The link to the EUPT-SRM15 result submission webtool and the personal login credentials will be provided to the PT-contact persons on the day of sample shipment.

The lab's unique lab code for the EUPT-SRM15 will be provided to the participants following the first access to EUPT-SRM15 result submission webtool.

- Acknowledgement of package receipt and acceptance of PT-materials

Once the laboratory has received the package with the PT material, it must report to the organizer via the EUPT-SRM15 result submission webtool the date of receipt, whether the material is accepted or not, and any other comments concerning the test material. This task should be finalized by 19 February. If a laboratory does not respond by this deadline, the Organizers will assume that Test Item has been received and accepted. Please note that completing the sample receipt and acceptance acknowledgement information is a pre-requisite for accessing the website areas in which results and method information is submitted.

Any participants not having received the Test Items by the Fri. 14 February at noon must inform the Organizer via e-mail (EURL-SRM@cvuas.bwl.de) by Fri. 14 February 2:30 pm. The Organizer will consult the shipping company to localize the package and decide on further actions including new shipment, if necessary.

- Reporting qualitative and quantitative results

To report their results, laboratories must access the EUPT-SRM15 result submission webtool.
All results must be reported on this website by 31 March, 11.30 pm (CEST), 2020. 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 EUPT-SRM15 Target Pesticides list carefully, in particular the residue definitions that apply to the EUPT, which may not be given in full on the result submission website.

Among others, the following fields will be available for reporting the quantitative results:

- "Concentration in mg/kg": the numerical 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 judged as "false negatives".

  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 lab's actual (or projected in case of new analytes) routine 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 factor, use of ILIS) must be reported in the respective fields.

- "Conc. in blank in mg/kg": concentration values of any pesticides from the EUPT-SRM15 Target Pesticides List determined in the Blank Material (even at levels below the MRRL).

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

- Reporting Information on Analytical Methodology

On the page of "Edit methods" of EUPT-SRM15 result submission webtool the participating laboratories must provide information on the analytical method(s) applied to pesticides, which were analysed and detected in the Test Item.

The participating laboratories are urged to thoroughly fill-in all requested information. If entries in required fields are missing, you cannot submit your results.

For detailed information on the columns on the page of "Edit methods" please refer to the guideline for results submission, that will be distributed to all participants in due time, and that can also be download from the support box on the webtool.

- Submission of results

Once you have entered all your results and checked their correctness, you have to submit them by clicking "Final submission" button that can be found at the bottom of each page. This has to be done before the submission deadline, afterwards, you will NOT be able to change your data anymore. Without "Final submission" your results and method information will not be included in the evaluation!

- Additional Information

If the laboratory has obtained tentatively false negative result(s), it will be asked to enter the method information for the analytes(s) in question after the results submission period is closed.

Subcontracting

The following tasks were subcontracted to the EURL-CF, Lyngby, Denmark:

a) Generation of the login credentials
b) Programming and administration of EUPT-SRM15 result submission website

Follow-up actions

After the distribution of the EUPT-SRM15 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 the poor performance, and to report possible corrective actions. This information will be forwarded to the corresponding NRL-SRMs upon request. All EUPT-SRM15-participants are welcome to ask the EURL-SRM for technical assistance.

The Organizer might ask laboratories to provide missing methodology information that is important for the evaluation and interpretation of the PT.

According to instructions from DG-SANTE, the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of NRLs" is to be followed by NRLs.

Documents

All documents related to the EUPT-SRM15 can be found in the EURL-Document Repository (CIRCA-BC). Links to the documents can also be found in the EUPT-SRM15 Website.
For further information please contact the organizers EURL-SRM@cvuas.bwl.de
Please check the EUPT-SRM15 Website before starting with the analysis in order 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 approximately one week after sample shipment to the invoice e-mail address stated in the registration form. Should the payment being taken care of by another department/institution, the recipient of the invoice is requested to forward the invoice accordingly.
Details on payment are given in the invoices.

Payment is expected to be made within 30 days upon the invoice issue date, unless special information was provided by the participant during registration and/or otherwise agreed between participant and the Organizers.

If, for any reason, payment cannot be carried out before this date, please contact the Organizers 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 laboratory 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 nf CVUA Stuttgart	DE 811 680 510

Please note:
EURL-AO based in CVUA Freiburg and EURL-SRM based in CVUA Stuttgart belong to the same ministry and have thus the same bank account.
If your laboratory is participating in both PTs (EUPT-SRM15 and EUPT-AO15), please ask your financial department to transfer the fee for each of the PTs separately using the corresponding payee identification text (= invoice number) given in each invoice. Without this text, your payment will not be able to reach the correct EURL.

Calendar of EUPT-SRM15

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM15_Calendar.pdf)

Target Pesticides List of EUPT-SRM15

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM15_TargetPesticideList.pdf)

Contact information

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

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

Organizing Group at the EURL-SRM (Stuttgart)

Michelangelo Anastassiades	**phone: +49 3426 1124**
Pat Schreiter	**phone: +49 3426 1029**
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**
Magnus Jezussek	**Bavarian Health and Food Safety Authority (LGL), Erlangen, DE**
André de Kok	**Wageningen Food Safety Research (WFSR), Wageningen, 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 Rodriguez	**University of Almería (UAL), Spain**

Quality Control Group

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

Appendix 10 Calendar and Target Pesticides List of EUPT-SRM15

EU Reference Laboratories for Residues of Pesticides
Single Residue Methods

CALENDAR for the EUPT – SRM15

RICE

(Tentative, Released on 17/03/2020)

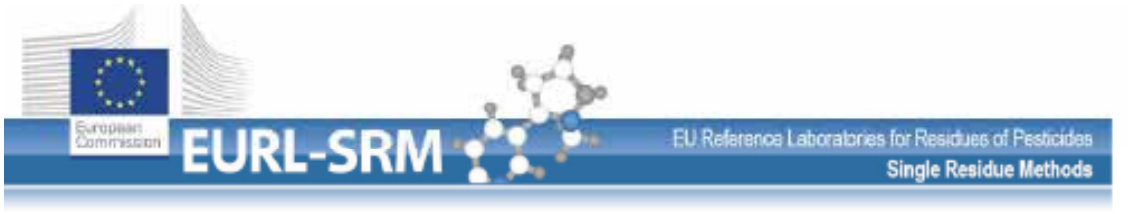
Activity	Who?	Dates
Opening of the **EUPT-SRM15 Website** with links to all relevant documents	EURL-SRM	28 Oct 2019
Period for Registration and for Explanations for Non-Participating (via "EUPT-Registration Website") (Obliged Labs MUST enter this Website and either register OR give explanations for non-participation)	All laboratories interested in participation and all obliged labs even if not interested	25 Nov – 23 Dec 2020
Dispatch of: • EUPT-SRM15-Specific Protocol • Links to EUPT-SRM15 Result Submission Webtool • Personal login credentials	EURL-SRM	~ 27 Jan 2020
Preparation of EUPT-SRM15-Test Item (Preliminary tests: Spiking, Homogenization, Stability)	EURL-SRM	Dec 2019 – Feb 2020
Homogeneity Tests	EURL-SRM	Jan – Feb 2020
Stability Tests	EURL-SRM	Jan – Mar 2020
Shipment of EUPT-SRM15 Test Item (+reminder of upcoming parcel arrival)	EURL-SRM	10 Feb 2020
Period for Confirming Sample Receipt and Acceptance (via "EUPT-SRM15 Result Submission Webtool")	Participating Labs	From 10 Feb 2020
Period for Submission of Results (Pesticide scope, Results, Method Info) (via "EUPT-SRM15 Result Submission Webtool")	Participating Labs	10 Feb – 31 Mar 2020
Period for Submission of Additional/Missing Information (e.g. Method info on tentatively false negative results via "EUPT-SRM15 Result Submission Webtool")	Participating Labs	1 Apr – 9 Apr 2020
Dispatch of Preliminary Report (only compilation of results and preliminary assigned values)	EURL-SRM	~ 04 May2020
Survey to collect reasons for underperformance and missing information on methods	EURL-SRM / Participating Labs	May 2020
EUPT Evaluation Meeting	EUPT-SC, DG-SANTE	Jun 2020
Dispatch of Final Report	EURL-SRM	Dec 2020

REMARK:
Please note that the dates mentioned above may be subject to minor changes. In case of major 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@cvua.bwl.de

The EUPT-SRM Team

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

Appendix 10 (cont.) Calendar and Target Pesticides List of EUPT-SRM15



Target Pesticide List

for the EUPT–SRM15 2020, Rice

(update on 07.02.2020)

MANDATORY ANALYTES			
Analytes Name	Residue definition for the PT and additional remarks	MACP/WD	MRRL (mg/kg)
2,4-D	free acid	MACP	0.01
Carbofuran sum	sum of carbofuran, carbosulfan, benfuracarb and furathiocarb expressed as carbofuran	MACP	0.01
Chlormequat	expressed as chlormequat chloride	MACP + WD	0.01
Ethephon		MACP	0.01
Fluazifop	free acid	MACP	0.01
Glufosinate		MACP	0.03
Glyphosate		MACP + WD	0.03
Haloxyfop	free acid	MACP	0.01
Mepiquat	expressed as mepiquat chloride	MACP + WD	0.01
MPP	glufosinate metabolite (=3-(Methylphosphinico)propionic acid, CAS Number 15090-23-0, commonly known as MPPA)	MACP	0.03
N-Acetyl-glufosinate		MACP	0.03
TFNA		MACP	0.01
TFNG		MACP	0.01

OPTIONAL ANALYTES			
Analytes Name	Residue definition for the PT and additional remarks	MACP/WD	MRRL (mg/kg)
2,4-D sum	sum of free acid, esters and conjugates analyzed as free acid following hydrolysis, expressed as 2,4-D	MACP	0.01
AMPA	glyphosate metabolite	WD*	0.03
Bentazone			0.01
Diquat	expressed as dication	WD	0.02
Fluazifop sum	sum of free acid, esters and conjugates analyzed as free acid following hydrolysis, expressed as fluazifop	MACP	0.01
Haloxyfop sum	sum of free acid, esters and conjugates analyzed as free acid following hydrolysis, expressed as haloxyfop	MACP	0.01
Imazethapyr	free acid		0.01
MCPA	free acid	WD	0.01
MCPA sum	sum of free acid, esters and conjugates analyzed as free acid following hydrolysis, expressed as MCPA – (*deviates from legal RD, which includes MCPB*)	WD	0.01
MCPB	free acid	WD	0.01
MCPB sum	sum of free acid esters and conjugates analyzed as free acid following hydrolysis, expressed as MCPB – (*deviates from legal RD, which includes MCPA*)	WD	0.01
Mecoprop "sum"	sum of free acid, esters and conjugates analyzed as free acid following hydrolysis, expressed as mecoprop – (*deviates from legal RD which does only include the free acid*)		0.01

EU Reference Laboratory Requiring Single Residue Methods (EURL-SRM)
CVUA Stuttgart, Schaflandstr. 1/2, D-70736 Fellbach, Germany

Appendix 10 (cont.) Calendar and Target Pesticides List of EUPT-SRM15

OPTIONAL ANALYTES (cont.)			
Analytes Name	Residue definition for the PT and additional remarks	MACP/WD	MRRL (mg/kg)
Mecoprop	free acid		0.01
N-Acetyl-glyphosate		WD*	0.03
Paraquat	expressed as dication	WD	0.02
Quizalofop	free acid	WD	0.01
Quizalofop sum	sum of free acid, esters and conjugates analyzed as free acid following hydrolysis expressed as quizalofop	WD*	0.01

*Future residue definition
MACP-Reg: REGULATION (EU) 2020/589 of 28 March 2020
MCP-WD: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2020; 25-27 November 2020 rev. 2020

Note: This document may be subject to minor changes. In case of significant changes the organisers 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@cvua-bwl.de

The EUPT-SRM15 Organising Team

Appendix 11 Call for Registration for the EUPT-SRM15

Schreiter, Pat (CVUA-S)

Von: Schreiter, Pat (CVUA-S) <Pat.Schreiter@cvuas.bwl.de> im Auftrag von CVUA-S EURL-SRM Pesticides <EURL-SRM@cvuas.bwl.de>
Gesendet: Dienstag, 29. Oktober 2019 15:56
An: CVUA-S EURL-SRM Pesticides
Betreff: EUPT-SRM15 (Rice): Announcement and Invitation

Dear Colleagues,

We herewith cordially invite you to participate in the upcoming European Proficiency Test EUPT-SRM15 on the analysis of pesticides requiring Single Residue Methods organised by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM).

- **Test Item: Rice containing probably both incurred and spiked compounds.**
- **Registration: via the "EUPT-Registration Website" (not yet active!) from 25 November to 23 December, 2019.**
- **Planned day of shipment of Test Material: 27 January, 2020.**
- **Submission deadline for results and method information: 25 February, 2020 on the "EUPT-SRM15 Result Submission Website".**

Participation rules:
The following labs are considered obliged to participate in the EUPT-SRM15:

- **all NRLs for pesticides requiring Single Residue Methods (NRL-SRMs), see Art. 101 (1)(a) of Reg. (EC) 625/2017,**
- **all Official Laboratories (OfLs) performing pesticide residue analyses of cereals and feeding stuff within the frame of National and EU official controls, see Art 38 (b) of Reg. (EC) 625/2017and Art. 28 of Reg. (EC) 396/2005.**

Obliged labs that do not intend to participate in this test are expected by DG-SANTE to state the reasons for non-participation. These reasons should be reported on the "EUPT-Registration Website".

The following labs are welcome to register for the EUPT-SRM15 (participation will, however, depend on the availability of PT-Material):

- **any other OfLs from EU countries that are not covered by the above obligations to participate;**
- **laboratories analysing official organic samples within the frame of Reg. 889/2008/EC;**
- **NRLs and OfLs from EU-candidate countries and EFTA countries;**
- **Laboratories from Third Countries (countries outside EU) as long as they are involved in controls of products destined for export to the EU.**

All scheduled activities and deadlines of this PT can be found on the EUPT-SRM15 Calendar (http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM15_Calendar.pdf)
The list of analytes potentially contained in the Test Item is shown in the EUPT-SRM15 Target Pesticides List, that will be published on 2 November 2019.
All documents relating to EUPT-SRM15 will be uploaded onto the EUPT-SRM15 Website (http://www.eurl-pesticides.eu/docs/public/tmolt_article.asp?CntID=1139&LabID=200&Lang=EN) and the CIRCA-BC platform.

A general fee of 200 € (350 € for the labs from 3rd countries) **will be charged to each participating laboratory in the EUPT-SRM15 to cover the costs of test material handling and shipment. Double amount of material can be requested via e-mail (address: EUPT-SRM@cvuas.bwl.de) or on the registration website. In this case the above fees will be doubled.**

For more detailed information please refer to the EUPT-SRM15 Announcement (http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM15_Announcement.pdf).

Best regards,
Your EURL-SRM Team

Guide to EUPT-SRM15 Results Submission Webtool

Version: 2020-03, Date: 17-03-2020, Init: Schr

Please read this guideline carefully in order to get familiar with the Webtool before you start entering your data.

General Information:

- Please use Chrome or Firefox as Web-browser .
  The Webtool is not validated for internet explorer or other browsers.
- **Your data is automatically saved as soon as you move from one edited line to another.** Therefore, almost all pages and tables do not have any save button.
- You can access the Webtool as many times as you need during the results submission period. However, **before deadline you must submit your results and method information by clicking "*Final submission*". Otherwise, your result will not be included in the evaluation!**
- **After Final submission you will NOT be able to change your entries anymore!**

Browser requirements: The system works only with the following browsers

Browser	
Google Chrome	newest version
Firefox	newest version

* latest version is recommended

Please don't use Internet Explorer!

Getting started

link to Webtool: www.eurl.dtu.dk

Choose "Guest and others"

Log in to the Webtool using your personal username and password sent to you by email in connection to the present or a previous[1] EUPT on pesticides (EUPT-CF, -FV, AD or SRM).

If you forgot your login credentials and you have never changed your password, you can ask the PT-Organizers for your original login credentials.
If you have changed your password and don't remember it any more, you have to ask for a new password using the button "Forget the password".

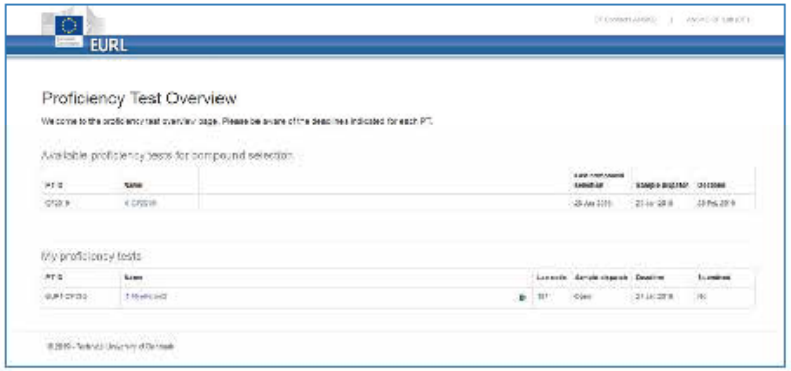
Please note: The PT-Organizers have access only to your original login credentials. Any changed or new password is known to you yourself only.

After signing in you will be guided to the *Proficiency Test Overview* page

[1] Typically the first EUPT you have participated in since 2019.

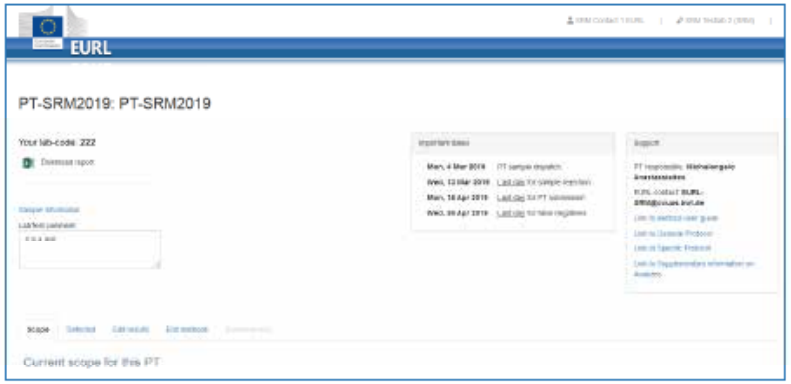
Proficiency Test Overview

On the page *"Proficiency Test Overview"*, you will see on the top the section *"Available proficiency tests for compound selection"* with the PTs that are available for compound selection and below the section *"My proficiency tests"* showing information (including lab codes which will be given automatically after you enter the respective PT-page for the first time) on the currently active EUPT(s), as well as on EUPTs in which your lab has participated in the past.



By clicking on "EUPT-SRM15 | Rice" under *"My proficiency tests"*, you will be able to view (but not edit) the current scope for this EUPT. The mandatory analytes are listed first, in alphabetic order, followed by the voluntary analytes, also in alphabetic order.

In contrast to other EUPTs organized by EURL-CF, -FV and -AO, the analyte scope does not need to be selected before sample shipment in the case of EUPT-SRMs. You will be able to edit your targeted compounds from the point the Webtool is open for results submission to the deadline for result submission.

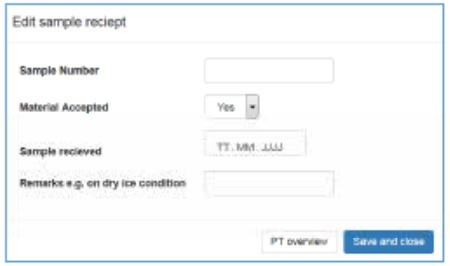


Sample receipt and acceptance

The Webtool for the EUPT-SRM15 result submission will be expectedly open on 10 February 2020.

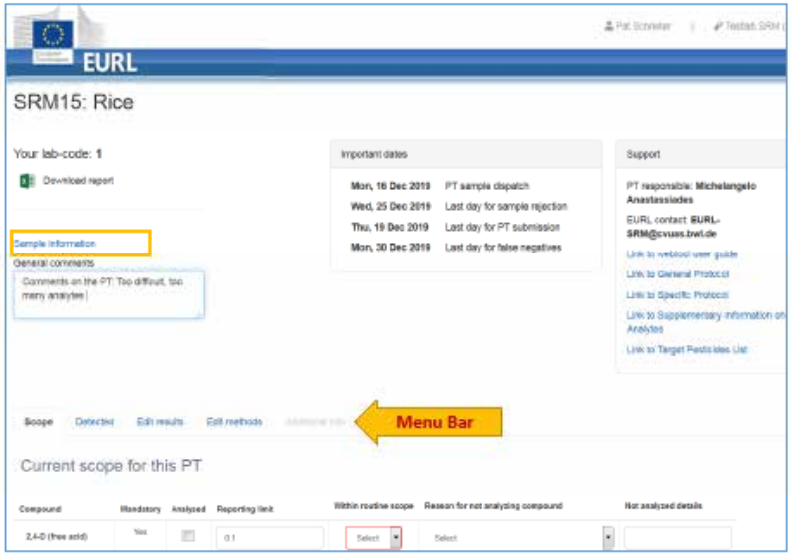
Once you have received the parcel with the PT-materials, please click on EUPT-SRM15 under "My *proficiency tests*" to open the pop-up window "Edit sample Receipt". Please fill in the information requested within this pop-up-window:

- **Sample Number: Please enter the bottle number of the Test Item you received.**
- **Material Accepted: Based on condition upon receipt please indicate "Yes" or "No".**
  If the PT-materials are not accepted, please additionally contact the PT-Organizers via E-mail.
- **Sample received: Please enter the date in which the parcel arrived in your institution.**
- **Remarks e.g. on dry ice condition: Please enter here any remarks concerning the condition of the boxes and sample bottles, the temperature, whether dry ice was left in the box (where applicable) etc.**
  Please note: No dry ice will be used for the shipment of the SRM15 material.

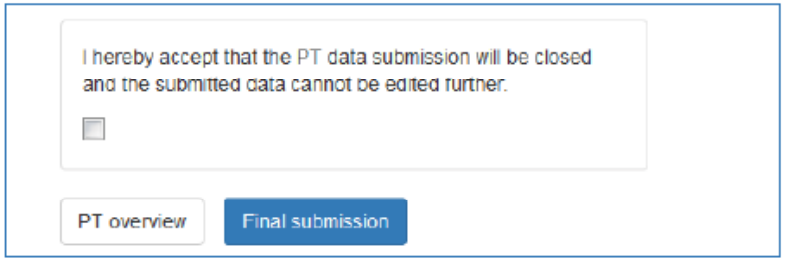


Completing the *"Edit Sample Receipt"* window is a precondition for being able to continue the submission page. This should be done ideally shortly after parcel receipt and not later than 19 February. You can, however, access and edit all the entries on "Edit sample receipt" through the PT-period under "Sample information" (please see next page, left navigation bar).

Upon clicking on *"Save and Close"* you will be guided to the following page in which you can see your Lab-code, a button for downloading the report, and a text field for any comments that you may want to pass to the organizer in relation to this particular PT. On the right side of the page, you can find important dates and Supporting information with useful links. If you scroll further down, you will find a Menu Bar with the following tabs: *"Scope"*, *"Detected"*, *"Edit results"*, *"Edit methods"* and *"Additional info"*.



On the bottom of each table you will see the button for *Final submission*.
Use this button only after you have already entered all your data for this PT and want to submit them for the PT evaluation.
After the Final Submission you will NOT be able to change your data any more.



Scope

In case of EUPT-SRMs this table remains accessible and editable during the whole results submission period. Thus, you can change your scope selection at any time.

Only analytes marked as "Analyzed" on this page will show up in the table "Detected".

In this table please firstly select the analytes you have targeted within the EUPT-SRM15 and enter your Reporting Limit (RL) of each. You can use the function "select/deselect all" to make a quick change for all analytes.

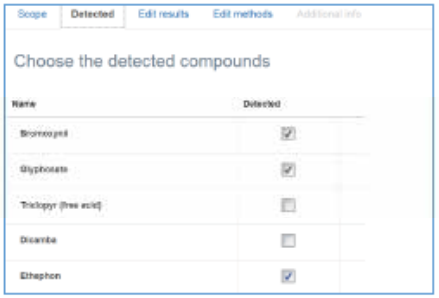
The MRRLs were set as default reporting limits. For each pesticide within your PT Scope please change the Reporting Limit to that of your laboratory.

Please also state for each analyte whether it is "*within your routine scope*" or not. This information is mandatory for all compounds on the Target Pesticides List regardless of whether they were targeted within this PT or not. In case that a compound is within your routine scope but skipped in the PT, please state the "*Reason for not analyzing compound within your scope*".

Even if your laboratory doesn't analyse for certain compounds routinely, you are encouraged to use this PT as a starting point for assessing your methods or for expanding your scope.

Detected

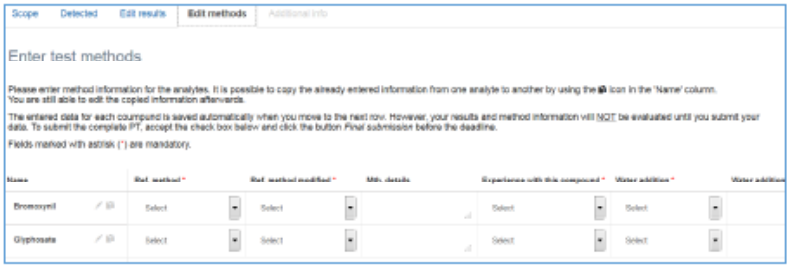
This page will list only analytes that were selected as "analysed" under "Scope". Please mark the analytes that you have detected in the Test Item. These selections will be used as filters for the subsequent *"Edit results"* table.



Edit methods

Click on the *"Edit methods"* tab to start reporting the method information.

NOTE: the system will automatically save your inputs when moving from one row to another.



Use the scroll bar to reach other parts of the table.
You can get short description about the columns via mouse-over messages.
Use the edit function to get an overview of all method-information fields of a selected pesticide.
However, there is no mouse-over information on the edit view.
Use copy function to copy the information from one pesticide to another.

The copy function works only if all mandatory fields for the template compound were filled in.
Otherwise, the icon of copy function becomes red.

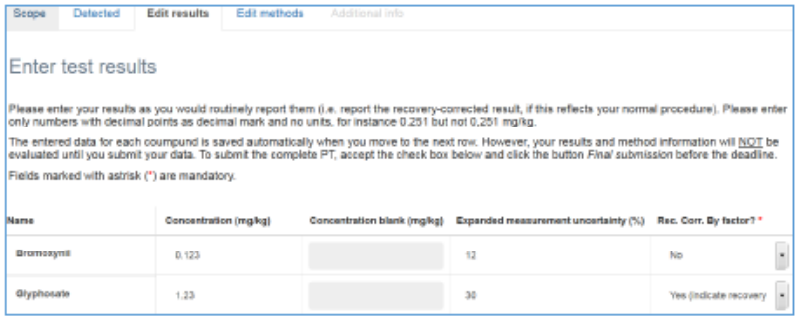
Further information about the *"Edit test methods"* table is summarized below.

Field(s)	Unit	Explanation
Ref. method		**Choose from the drop-down list; if you have used a modified form of the mth pls. give details under "Mth Details"**
Ref. method modified		**Specify if you have introduced any noteworthy modifications to the selected reference method. Give brief details on the modification under "Mth. details".**
Mth. details		**Describe you method shortly if it is not on the dropdown menu or indicate shortly the modifications introduced to the selected reference method**
Experience with this compound		**Experience of your lab with the analysis of this pesticide (with any type of commodity)**
Water Addition		**Please choose "Yes" if water or a water-containing solvent mixture was added to the sample to assist extraction.**
Water Addition Details		**Details on water addition (e.g. amount in mL, step of addition)**
Soaking step prior to extraction?		**Please indicate whether your sample was left to soak with water or the extraction solvent prior to the extraction step..**
Soaking Time	**min**	**Soaking time of the sample with water/water containing solvent. Choose closest value.**

Edit results

Click on the *"Edit results"* tab to enter the table where you can enter the quantitative results of the detected pesticides. You will only see the pesticides that are marked on the *"Detected"* table. Use the scroll bar to reach other parts of the table.

NOTE: The system will automatically save your inputs when moving from one row to another.



Further information about the *"Edit test results"* table is summarized below.

Field(s)	Unit	Explanation
Concentration (=Concentration in Test Item)	**mg/kg**	**Concentration in Test Item (mandatory if marked as detected); Syntax "0.345"; use points for decimal separation. Only numerical values are to be reported here. An entry such as "<RL" may be judged as a "False Negative" result if the compound is present in the Test Item and RL>MRRL or the assigned value.**
Concentration blank	**mg/kg**	**Deactivated, since there is no SRM15 blank material sent to the participants.**
Expanded measurement uncertainty	**%**	**Please indicate the % expanded measurement uncertainty value (Syntax "123") that you would report for the specific compound-matrix combination (e.g. in case of an MRL-Violation)**
Rec. Corr. by factor?		**Please indicate "yes" only if the result reported was corrected using a RECOVERY FACTOR. Other means of recovery based corrections are covered by other questions**
Recovery rate %	**%**	**(Mean) recovery rate used to derive the recovery corrected result that was reported for the Test Item (in %, Syntax "123"**
Recovery Obtained		**Please choose among the dropdown-options to indicate how the recovery rate used for recovery correction was obtained**
Recovery individuals		**"No. of replicate experiments conducted to obtain the recovery rate/factor used for the correction of results"**
Recovery details		**Please give brief details how the reported recovery rate was obtained; indicate the matrix used if not matching, the spiked compound, the spiking level/range**
Comments		**Comments on the analysis of the selected analyte**

Appendix 12 (cont.) Guide to EUPT-SRM15 Results Submission Webtool

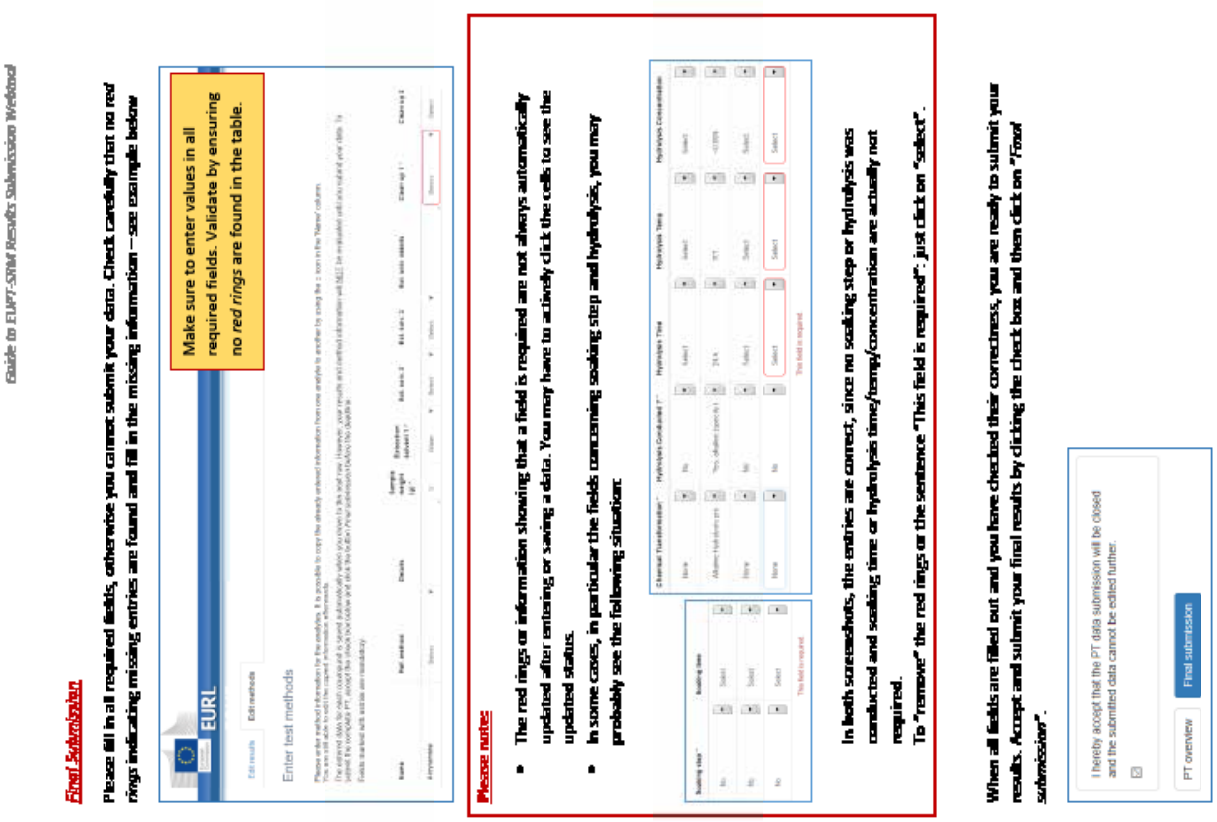
Field(s)	Unit	Explanation
Sample Weight (g)	g	Enter the sample weight used. Example: "3".
Extraction/partitioning solvent 1		Choose the solvent from the drop down menu
Extraction/partitioning solvent 2		Choose the solvent from the drop down menu, if you use more than one solvent
Extraction/partitioning solvent 3		Choose the solvent from the drop down menu, if you use more than two solvents
Extraction solvent details		Enter details on solvents used in extraction or partitioning steps or if the solvent is not in the drop down menu
Extration Time	min	Duration of main Extraction step including any waiting time after addition of solvent [min] (choose closest value)
Extraction approach		Choose Extraction approach from drop-down list
Partitioning salts used		Choose partitioning salt used from drop-down list
pH modified		Indicate if you have modified the pH at any stage of the procedure (e.g. by buffering, acid/base addition)
pH modified details		Please give details on pH modification step(s)
Clean up 1		Choose the clean-up approach employed from the drop-down list
Clean up 2		Choose the clean-up approach employed from the drop-down list, if you use more than one clean up step
Clean up 3		Choose the clean-up approach employed from drop-down list, if you use more than two clean up steps
Clean up details		Please give details on clean-up step or describe the way you clean up if it is not listed in the drop down menu
Chemical transformation		Mark if your procedure included a chemical transformation e.g. hydrolysis, derivatization, reductive cleavage to CS_2
Hydrolysis conducted?		Please indicate whether a hydrolysis step was conducted and if yes what type of hydrolysis was conducted
Hydrolysis time		Please choose the closest time from dropdown list
Hydrolysis temp.		Please choose the closest temperature from dropdown list
Hydrolysis concentration		Please choose from dropdown list the closest value of the acid / base normality during hydrolysis step; e.g. if the recipe was 2 mL of 5N NaOH + 20 mL (Water/ACN 1:1), the Normality would be ~0.5N)
Chem. transf. details		Please give details on chemical transformation step(s) conducted
Calibration		Choose the calibration approach used. NOTE: "Procedural calibration" and "Standard additions to sample portions" involve correction for recovery.
Technique [=Determination technique]		Choose the technique used to generate your quantitative result
Determination Details		Here you can add any relevant details on Determination technique used
LC-Details		e.g. column type and mobile phase used
Other Approaches to Corr. PT-Result for Recov.		Shortly describe any OTHER APPROACHES employed for correction of results for recovery. NOTE: Corrections for recovery via ILIS or via RECOVERY FACTOR are covered by other specific questions. "PROCEDURAL calibration" and "STANDARD ADDITIONS TO SAMPLE PORTIONS" are covered under "calibration"
Matrix used for calibration		Blank commodity used for matrix-based, matrix-matched or procedural calibration
Matrix calibration details		Please name the blank commodity used to prepare the calibration solutions and any other details of importance, such as differences between sample extract and calibration solution (e.g. in cleanup, dilution etc.)

Field(s)	Unit	Explanation
IS used		Please choose "No" if no "IS" was used or if the IS was only used for quality control purposes and not for the calculation of the target analyte result. Please choose one of the two "Yes" options if the IS was used for calculation of the result of the target analyte.
IS Name		Please give details on the IS used
When was IS added?		Mark at what stage of the procedure the IS was added
Comments (=General Comments on Analyte)		Please enter here any general comments concerning the analysis of the selected compound

Please notes

- The four fields concerning hydrolysis (marked red in the table) are new.
- By mistake there are two fields for chemical transformation: 1) **"chemical transformation"** and 2) **"Hydrolysis conducted?"**. "
  Hydrolysis conducted" is actually unnecessary, but both fields are mandatory. Contradictory in these two fields should be avoided.
- The red rings or information showing that a field is required are not always updated automatically after entering or saving a data.

Appendix 12 (cont.) Guide to EUPT-SRM15 Results Submission Webtool



Important guidance for uniform reporting in case of methods involving hydrolysis:

If you have analysed an acidic pesticide (e.g. 2,4-D) **without applying hydrolysis (directly)**, please report the result and respective method information under "… free acid" (e.g. *"2,4-D (free acid)"*).

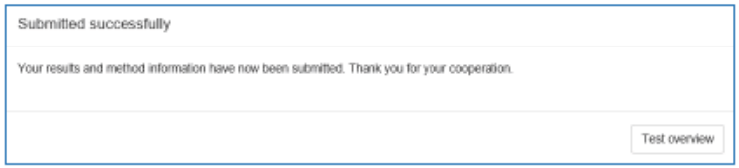
If you have analysed an acidic pesticide using a **procedure involving hydrolysis** (e.g. alkaline), please report the result under "… (sum)" (e.g. *"2,4-D (sum), expr. as 2,4-D"*)

Method used (exemplary)	Hydrolysis Conditions for cereals in brief	Scope of method	Volumes involved	Dilution factor of acid/base	Calculated acid/base Strength	Entries into Data Submission Tool			
						"Reference Method (drop down)"	*"Method Details"*	*"Hydrolysis acid/base strength"*	*"Chemical Transformation Details"*
SRM-43 (link) a. AH-CH-QuEChERS (p.20); b. AH-FA-QuEChERS (p.21)	2mL 5N NaOH 90°C 120 min	free acids, esters and conjugates (e.g. "2,4-D (sum), expr. as 2,4-D")	+ 8 mL water + 2 mL 5N NaOH + 10 mL ACN TOTAL = ~20 mL	~10	~0.5 N	AH-QuEChERS – involving alkaline hydrolysis; EURL method SRM-43	AH-CH-QuEChERS OR AH-FA-QuEChERS + any other relevant info	0.5 N (during hydrolysis step)	"AH after to ACN-addition" + any other relevant info
EN15662; module E5 (NOTE: In "difficult" commodities (e.g. citrus, pulses, cereals) resistant esters are only partly hydrolyzed, applying this approach)	8 mL 5N NaOH 90°C 30 min	free acids, esters (partly) and conjugates"	+ 10 mL water + 1 mL 5N NaOH + 10 mL ACN TOTAL = ~21 mL	~21	0.24 N	QuEChERS – Citrate buffered (EN 15662)	Module E5 + any other relevant info	0.25 N (during hydrolysis step)	"AH after to ACN-addition" + any other relevant info
AH+QuEChERS CH.-SRM (2007) (NOTE: This method was optimized only for conjugates, not for esters)	2.3 mL 5N NaOH RT 30min	free acids, esters (partly) and conjugates"	+ 10 mL water + 300 µL 5N NaOH	~33	0.15 N	Other, please specify under "Method details" (unfortunately, no drop-down option for this method was foreseen)	AH+QuEChERS CH.-SRM (2007) + any other relevant info	0.1 N or 0.2 N (unfortunately exactly between two choices) (during hydrolysis step)	"AH prior to ACN-addition" + any other relevant info
SRM-23 (link); SRM-33 (link)	500 µL 5N H_2SO_4 80°C 180 min	"Carbofuran (sum)"	+ 1 mL QuEChERS Extract +10 µL 5N H_2SO_4	~100	0.05 N	Carbofuran (sum): QuEChERS-based Method involving acidic hydrolysis by EURL-SRM (SRM-23)	any relevant info	0.05 N (during hydrolysis step)	any relevant info

IMPORTANT:

You will **NOT** be able to edit your data after the final submission!
Your data has to be submitted before the deadline on Tue. 17 March 2020, 23:30 h (11:30 p.m.), CEST.

Upon final result submission the following pop-up window confirming successful submission of the data will appear on the screen. In parallel, you will receive an email with an attached Excel file, in which your submitted data is compiled.



By clicking on the "*Test Overview*" button of the pop-up message you will be able to return to the *Proficiency test overview* page. The status of the PT will now be *Submitted*: "yes".

By clicking on the excel-icon you can download your submitted data, even for the exceeded PTs.

Guide to EUPT-SRM Results Submission Webtool

Additional information

Pesticides present in the Test Item and reported as "analysed", but not reported as "detected" are regarded as tentatively false negatives. For those compounds method information is required.

After the PT deadline, if you have submitted tentatively false negative or false positive result(s) in this PT, the PT-row will be highlighted in pastel yellow on the *Proficiency Test Overview* page.

Click on the yellow-marked EUPT and fill in the missing method information for the compounds identified as tentatively false negatives. Submit this information by 26 March.

Tentatively false positives will be marked with red color.

Appendix 12. Guide to EUPT-SRM15 Results Submission Webtool

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