European Commission

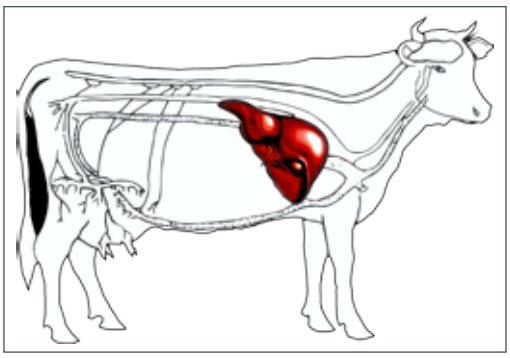
EURL-SRM

EU Reference Laboratories for Residues of Pesticides
Single Residue Methods

EU Proficiency Test on the Analysis of Bovine Liver Homogenate for Residues of Pesticides Requiring Single Residue Methods

EUPT–SRM14

March/April 2019



Final Report

Chemisches und
Veterinäruntersuchungsamt
Stuttgart

cvua STUTTGART

EU PROFICIENCY TEST
EUPT-SRM14, 2019

Residues of Pesticides Requiring Single Residue Methods

Test Item: Bovine Liver Homogenate

Final Report

Resutls Evaluation

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Picture on the book cover: Cattle And Onions Horse Livestock Transprent - Cow Liver @pngkey.com

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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 the analysts 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 analytical quality of the laboratories.

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 residues of pesticides 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. Four 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 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 of plant origin with low lipid 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). The present EUPT-SRM14, based on bovine liver homogenate, is the second one dealing with a commodity of animal origin and the first one EUPT-SRM co-organized with the EURL for Residues of Pesticides in Food of Animal Origin (EURL-AO).

Participation in EUPT-SRMs is mandatory for all NRLs for pesticides requiring Single Residue Meth- ods (NRL-SRMs) and for all OfLs analysing pesticide residues, within the framework of national or EU-coordinated 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. 625/2017/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. A non-matching analyte scope of a lab with the analyte scope of the PT may be a valid reason for a non-participation in a short term, but actions should be taken to expand the scope in the long term.

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

Evaluation Report on the Feedback Survey on EUPT-SRM14

http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_Survey_Evaluation.pdf

European Commission –
EU-Proficiency Test on Residues of Pesticides
Requiring Single Residue Methods
Test Item: Bovine Liver Homogenate
EUPT-SRM14, 2019

INTRODUCTION

Following an urgent request by the COM, on 16 November, 2018 all relevant National Reference Laboratories (NRLs) of the 28 EU-Member States (MS), as well as all relevant EU-Official Laboratories (OfLs) whose contact details were available to the organisers (EURL-SRM and EURL-AO) were informed about the intention to use "bovine liever" as the commodity in the upcoming EUPT-SRM14 and -AO14 and about the planned shipment date. On 18 December, 2018, all concerned laboratories were invited to participate in the 14th European Commission's Proficiency Test Requiring Single Residue Methods (EUPT-SRM14). The EUPT-SRM14-Website initially contained links to the Announcement/Invitation Letter and the Calendar. A link to the EUPT-SRM14 Target Pesticides List, entailing 32 compounds, was added to the website on 18 January 2019. The selection of the compounds took place in close consultation with the EUPT Scientiffic Committee and considered the SANTE working document listing pesticides to be considered in national control programmes[1], the relevance of compounds for liver, the availability of analytical standards and the capability of laboratories. To check the capability and intentions of the potentially participating laboratories and to avoid the inclusion of compounds that are analyzed by too few labs, a survey was conducted among all OfLs analyzing pesticides in food of animal origin. In this survey (start 27/12/2018, end 11/1/2019) the laboratories had to declare which compounds they were intending to analyze in the upcoming PT (see **Appendix 10**). Among the selected compounds, only glyphosate was listed in the EU-coordinated Multiannual Control Program for Pesticide Residues and was thus considered as mandatory within this PT. For each compound a residue definition valid for the PT and the minimum required reporting level (MRRL) were stipulated.

On 21 January 2019 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 "Supplementary Information on Analytes" entailing, among others, information on exemplary sources of analytical standards (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 2019. Two weeks prior to the shipment of the PT material to the laboratories the "EUPT-SRM14 Specific Protocol" was uploaded and a link was 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.

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

Based on commodity scope (food of animal origin) and NRL-status (NRL-SRMs) all laboratories were allocated a status as regards their obligation to participate in the EUPT-SRM14. This tentative 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 that 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.

In total 57 participating labs from EU and EFTA countries and three laboratories outside the 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-SRM14 was conducted using calves' liver originating from Germany. The test itemwas prepared by spiking the finely ground liver, at around 0 – 5 °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 Item and Blank Material"**

1. TEST ITEM AND BLANK MATERIAL

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

Upon an urgent request by the COM at the end of October 2018, bovine liver was chosen as commodity for the EUPT-SRM14.

To ensure that the results reported by the participants would be sufficient for a reliable statistical evaluation, a survey on the analytical scope intended to be covered was conducted in advance among NRL-SRMs and OfLs analysing pesticides residues in food of animal origin. The compounds included in this survey were selected 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 2018); 3) the relevance of pesticides to the specific commodity.

The final selection of the compounds to be included in the Target Pesticides List (**Appendix 10**) were selected by the organiser and the EUPT-Scientific Committee (Advisory Group and Quality Control Group) taking into account the analytical capabilities and intentions of the laboratories as recorded in the survey as well as all the above mentioned aspects.

The minimum required reporting levels (MRRLs) were set at 0.01 mg/kg for *2,4-D*, ***2,4-DB***, 5*-OH-thiabendazole*, ***avermectin B1a***, ***desmethyl-bixafen (M21)***, ***boscalid met. M510F017***, ***bromoxynil***, *chlorate*, *chlormequat*, *dichlorprop*, *emamectin B1a*, ***fenpropimorph carboxylic acid (BF-421-2)***, ***flonicamid Met***. ***TFNA-AM***, *fluazifop*, ***haloxyfop***, *isoxaflutole diketonitrile (RPA 202248)*, ***MCPA***, *MCPB*, ***mepiquat***, *quizalofop*, and *triclopyr*; at 0.02 mg/kg for ***fluopyram-benzamide (M25)***; at 0.03 mg/kg for *BAC-C_{12}* and ***DDAC-C_{10}***; at 0.05 mg/kg for *dicamba*, *ethephon*, ***MPP***, and *N-acetyl-glufosinate*; as well as at 0.1 mg/kg for *glufosinate*, ***glyphosate***, ***AMPA***, and ***N-acetyl-glyphosate***.

For the production of test item and blank material, 60 kg calves' liver were purchased from a slaughterhouse, cleaned from fiber, and cut into pieces. Half of the liver material was used for the production of the blank material and the other half was spiked with 16 compounds (see **Section 1.4, p. 3**) and used as test material. Calves' liver was chosen over liver of adult bovines or swine because of its typically lower levels of contamination. The materials were processed within the facilities of an institute providing vocational education for aspiring butchers. A teacher of that school assisted in the preparation of the material.

1.2 Preliminary Investigations on the Behavior of Analytes during Homogenisation

In order to optimize the homogenization process of the test material and to estimate the behavior of spiked analytes during the preparation of the test material, several preliminary spiking experiments were performed at a small scale.

1.2.1 Optimization of homogenisation

Fresh liver was coarsely cut, immersed into liquid nitrogen and then homogenized into a fine powder by a powerfull knife mill. This liver powder was thawed and 2 kg liver puree was spiked at room temperature with a mixture of a representative analytes listed on the Target Pesticides List plus some other analytes covering a broad polarity range (**Table 1-1, p. 2**). The material was mixed further for 3 minutes with the knife mill and the homogeneity was checked by analyzing 5 analytical portions. Part of the material was processed further to a frozen snow-like powder prepared and also analyzed in quintuplicate. To generate the snow-like

Table 1-1: Small scale investigation on homogeneity of test material (matrix: liver, n = 5)

Analyte	Homogenized at RT	… and processed further to a snow-like powder	Analyte	Homogenized at RT	… and processed further to a snow-like powder
	RSD (%)	RSD (%)		RSD (%)	RSD (%)
2,4-D	3.9	4.7	Etofenprox	3.3	1.9
Chlormequat	0.8	5.6	Mepiquat	2.7	2.0
Chlorpyrifos	2.3	1.8	Metalaxyl	3.2	3.2
Cyhalothrin-lambda	2.3	2.0	Propyzamid	3.6	1.5

material, the puree was filled into a large plastic zip-bags so that each contained a flat 0,5 cm thick layer. The bags were placed in the freezer in a flat position and the frozen liver plates were ground to a snow-like powder using a knife mill and dry ice. Analysis was conducted using the QuEChERS method (n = 5 each).

1.2.2 Investigation on the Behavior of Analytes during Test Material Preparation

This small-scale preliminary investigation was conducted to obtain information about the behavior and the stability of analytes during sample preparation.

Three analytical portions of blank liver puree were spiked at RT and left standing at RT for approx. 1.5 h. The intention was to simulate a long homogenization process under worst case condition and to check what may happen if PT-participants leave their samples to thaw. This experiment was performed with two different analyte mixtures, one containing the parent analytes and the other one the corresponding metabolites. These mixtures were spiked to separate portions of liver puree. In parallel, triplicate recovery experiments were conducted by spiking blank liver homogenates at two different conditions: a) in frozen state (ca. –5- 0°C) and b) at room temperature on blank liver material that was previously left "to age" for ca. 1.5 h at room temeprature to resample, as far as possible, the condition of the liver of the above experiment).

Most analytes included in this test survived the presence in liver homogenate for 1.5 h at room temperature, with the recovery rates ranging between 82 and 110 % compared to the recovery rates of 87 to 108 % obtained for those that were analyzed directly after spiking onto the aged material. Only the spiked esters (fluazifop-butyl and propaquizafop) showed a strong degradation to their respective free acids (fluazifop and quizalofop).

1.2.3 Method Validation

Method validation results for analytes amenable to the QuEChERS method are shown in **Table 1-2 (p. 3)**. For analytes amenable to the QuPPe method please refer to the QuPPe document: https://www.eurl-pesticides.eu/userfiles/file/meth_QuPPe_AO_V3_1.pdf.

1.3 Preparation and Bottling of the Blank Material

The liver homogenate was prepared within the facilities of an institute for vocational education for aspiring butchers located in Freiburg/Germany. Approximately 30 kg of calves' liver free from fiber was pre-cooled to 4 °C, cut into pieces and finely ground using a professional meat grinder and kept at approximately 0 °C.

Table 1-2: Results of method validation using QuEChERS

Recoveries of analytes amenable to QuEChERS method							
Analyte	**Rec (%)**	**RSD (%)**	**Spiking Level**	**Analyte**	**Rec (%)**	**RSD (%)**	**Spiking Level**
Avermectin B1a	104	4	0.01	2.4-DB	102	10	0.01
BAC C12	89	6	0.02	2.4-DP (Dichlorprop)	93	9	0.01
M510F01	98	8	0.01	Bixafen desmethyl	101	16	0.01
Chlormequat	39	6	0.01	Bromoxynil	87	5	0.02
DDAC C10	89	3	0.02	Dicamba	55	11	0.03
Emamectin B1a	103	4	0.01	Fluazifop	108	8	0.01
Fenpropimorph Carboxylic Acid	105	5	0.01	Haloxyfop	99	10	0.01
Fluopyram Benzamide	99	8	0.01	Isoxaflutole, RPA202248	101	10	0.01
Mepiquat	33	8	0.01	MCPA	90	7	0.01
TFNA-AM	91	6	0.01	MCPB	104	9	0.01
Thiabendazole, 5-hydroxy-	75	8	0.01	Quizalofop	96	9	0.01
2,4-D	83	9	0.01	Triclopyr	92	8	0.01
Analytical replicates: 5 except avemectin B1a with only 2 replicates.							

The cold material was placed in its entirety into a large professional coolable meat cutter. After addition of 300 g NaCl with aim to improve the flowing properties, the material was intensively homogenized. The material temperature was kept below 2 °C during this process. Using a professional portionation machine, approximately 200 g portions of the well-mixed liver homogenate were filled into pre-labelled, leakproof screw-capped polyethylene plastic bottles. The sealed bottles were placed into boxes and placed into a large walk-in freezer for several days at −40 °C. Afterwards, the bottles were transported to EURL-SRM in Stuttgart using a special freezer van, and were stored there at −20 °C till their dispatch to the participants.

1.4 Preparation and Bottling of the Test Item

For the preparation of the test item ca. 30 kg of liver were ground and homogenized in a way similar to that described above for the blank material. While the material was being homogenized in the meat cutter 53 ml of a solution containing the target analytes was slowly added and the homogenization was continued for further 3 minutes. The spiking solution contained 16 different compounds and was prepared as described in **Table 1-3 (p. 4)**. The homogenized liver was portioned (approx. 500 g portions) into pressure-lock plastic bags, which were layed down so that the material can evenly distributed within the bag by forming a flat cake. The flat portions were all together placed into a large walk-in freezer at −40 °C, where they were kept for several days. The frozen liver cakes were then transported by the freezer van to the EURL-SRM facilities where they were placed into a walk-in freezer at −20 °C. Using various mixers the material was further homogenized with the use of dry ice to obtain a snow-like free-flowing material. Approximately 200 g portions of this material were manually weighed out into pre-labelled and leak-proof screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about −20 °C till their dispatch to the participants.

1.5 Packaging and Delivery of PT Materials to Participants

On the day of shipment, two frozen bottles, one with test item and the other one with blank material were packed into thermo-insulated polystyrene boxes, filled-up with dry ice pellets (2 – 3 kg in each box) and

Table 1-3: Analytes spiked into 30 kg of calves' liver for the preparation of the test material

Analytes dissolved in 32 ml H_2O containing 10 % ACN, 24 ml thereof used for spiking			Analytes dissolved in 32 ml ACN, 24 ml thereof used for spiking		
Compound	**Amount**	**Theor. Conc. [mg/kg]**	**Compound**	**Amount**	**Theor. Conc. [mg/kg]**
AMPA, glyphosate metabolite	32.03 mg	0.80	2,4-DB	2.4 mg [2)]	0.060
DDAC C-10-Cl	7.18 mg	0.18	Abamectin B1a	2.8 mg [2)]	0.060
Glyphosate	22.62 mg	0.57	Bixafen desmethyl	2.0 mg [2)]	0.050
Mepiquat	2.54 mg	0.064	Boscalid Metabolite M510F01 (free)	3.2 mg [2)]	0.080
MPP, glufosinate metabolite	12.35 mg	0.31	Bromoxynil	2.4 mg [2)]	0.060
			Fenpropimorph carboxylic acid (BF-421-2)	3.2 mg [2)]	0.080
Analytes dissolved in 5 ml H_2O containing 10 % ACN, 5 ml used for spiking			Flonicamid metabolite, TFNA-AM	3.2 mg [2)]	0.080
Compound	**Amount**	**Theor. Conc. [mg/kg]**	Fluopyram-benzamide (M25)	4.0 mg [2)]	0.10
N-acetyl-glyphosate	~ 15 mg [1)]	0.38	MCPA (free acid)	2.0 mg [2)]	0.050
			Haloxyfop	1.83 mg	0.046

ACN = Acetonitrile
1) 10 mg according to the packaging size solved in 5 ml stock solution (1 mg/ml), total amount approx. 15 mg
2) from stock solution 1 mg/ml

shipped by DHL-Express to the laboratories. Where the dry ice transport was not allowed due to IATA regulations, special measures were taken: In these cases ca. 200 g of blank homogenate and 200 g of cryogenically re-homogenized Test Item were weighed into sealable plastic bags, which were placed into pre-cooled (–20 °C) thermoinsulated Dewar vessels (capacity of 8 and 16 Oz). The Dewar vessels were placed into the freezer for several days. One bottle of each blank material and Test Item was then packed into thick-walled thermo-insulated polystyrene boxes and special freezing elements which had been pre-cooled at –80 °C were added. Those shipment units were stored at least for 3 days at –80 °C before they were picked up by DHL for express shipment. Once the parcel was picked up by DHL, the recipients received an e-mail from the shipping company entailing the individual tracking number.

Among the 59 packages sent to laboratories in EU and EFTA countries, 54 (92 %) reached the participating labs within 24 hours, 4 packages within 48 hours and only one package arrived the recipient laboratory on the third day due to remote location. The delivery to the three labs located in countries outside the EU and EFTA zones was accomplished within 48 hours, 4 days and 7 days. 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**. In a shipment simulation experiment the material was found to remain frozen even after 72 h storage of the parcel at room temperature. All material was accepteded by the participants and was reportedly in very good condition, even when arriving on the third or fourth day. Based on these results, it was judged that differences in shipment duration have most likely not had a signifficant influence on the analyte concentrations (and the analytical results of the laboratories), and it was thus decided not to further 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 the whereabouts of their own parcels via the online tracking tool of the shipping company in order to maintain the ability to take the necessary measures in case of delays, e.g., contacting the customs to ask for an acceleration of the clearance procedure or to place the parcel in a cool place until clearance is granted. The participants are furthermore encouraged to contact the local office of the shipping company to ensure optimal delivery 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 in the blank material are summarized in **Table 1-4**. For more details on the methods used, please refer to the EURL-SRM website: http://www.eurl-pesticides.eu (EURL-SRM-website → Services → EURL-SRM Methods / Analytical Observations).

Table 1-4: 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.

QuEChERS method [EN15662]
involving: weighing of 5 g liver homogenate into a sealable vessel, addition of IS / ILISs, extraction with ACN (15 min), addition of partitioning salts (4 g MgSO4, 1 g NaCl, 1 g trisodium citrate dihydrate and 0.5 g disodium hydrogen citrate sesquihydrate), 1 min shaking, centrifugation (at ~4000 rpm for 5 min) and direct determination by LC-MS/MS in the ESI (neg.) and ESI (pos) mode.

Compound	IS	Determinative analysis		Notes
2.4-DB	**BNPU**	**LC-MS/MS**	**ESI (neg)**	
Avermectin B1a	**Chlorpyrifos D_{10}**	**LC-MS/MS**	**ESI (pos)**	
Boscalit metabolite M510F01	**Chlorpyrifos D_{10}**	**LC-MS/MS**	**ESI (pos)**	
Bromoxynil	**Mecoprop D_6**	**LC-MS/MS**	**ESI (neg)**	
DDAC-C10	**Chlorpyrifos D_{10} / DDAC-C_{10} D_6**	**LC-MS/MS**	**ESI (pos)**	
Bixafen desmethyl	**Mecoprop D_6**	**LC-MS/MS**	**ESI (neg)**	
Fenpropimorph Carboxylic Acid (BF-421-2)	**Chlorpyrifos D_{10}**	**LC-MS/MS**	**ESI (pos)**	
Flonicamid metabolite TFNA-AM	**Chlorpyrifos D_{10}**	**LC-MS/MS**	**ESI (pos)**	
Fluopyram Benzamide (M25)	**Chlorpyrifos D_{10}**	**LC-MS/MS**	**ESI (pos)**	
Haloxyfop	**BNPU/ Haloxyfop D_4**	**LC-MS/MS**	**ESI (neg)**	
Mepiquat	**Chlorpyrifos D_{10} / Mepiquat D_3**	**LC-MS/MS**	**ESI (pos)**	
MCPA	**BNPU/ Mecoprop D_6**	**LC-MS/MS**	**ESI (neg)**	
*5-Hydroxythiabendazole**	*Chlorpyrifos D_{10}*	**LC-MS/MS**	**ESI (pos)**	
*BAC-C12**	*Chlorpyrifos D_{10}*	*LC-MS/MS*	*ESI (pos)*	
*Chlormequat**	*Chlorpyrifos D_{10}/ Chlormequat D_4*	*LC-MS/MS*	*ESI (pos)*	
*Emamectin B1a**	*Chlorpyrifos D_{10}*	*LC-MS/MS*	*ESI (pos)*	
*2,4-D**	*BNPU*	*LC-MS/MS*	*ESI (neg)*	
*2.4-DP (Dichlorprop)**	*BNPU*	*LC-MS/MS*	*ESI (neg)*	
*Dicamba**	*Dicamba D_3*	*LC-MS/MS*	*ESI (neg)*	
*Fluazifop**	*BNPU*	*LC-MS/MS*	*ESI (neg)*	
*Isoxaflutole diketonitrile metabolite RPA202248**	*BNPU*	*LC-MS/MS*	*ESI (neg)*	
*MCPB**	*BNPU*	*LC-MS/MS*	*ESI (neg)*	
*Quizalofop**	*BNPU*	*LC-MS/MS*	*ESI (neg)*	
*Triclopyr**	*BNPU*	*LC-MS/MS*	*ESI (neg)*	

QuPPe-AO method [https://www.eurl-pesticides.eu/userfiles/file/meth_QuPPe_AO_V3_1.pdf]
involving: weighing of 10 g liver homogenate into a sealable vessel, addition of ILISs, water adjustment, addition of methanol containing 1 % formic acid, addition of an extra amount of 100 µL 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.) or ESI (pos.) mode.

Compound	IS	Determinative analysis		Notes
Glyphosate	**Glyphosate $^{13}C_2$, ^{15}N**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.6**
Glufosinate metabolite MPP	**MPP D_3**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.6**
Glyphosate metabolite AMPA	**AMPA $^{13}C^{15}N$**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.6**
Glyphosate metabolite N-acetyl-glyphosate	**N-acetyl-glyphosate $^{13}C_2$, ^{15}N**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.6**
*Chlorate**	*Chlorate $^{18}O_3$*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.4*
*Ethephone**	*Ethephone D_4*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Glufosinate**	*Glufosinate D_3*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Glufosinate metabolite N-acetyl-glufosinate**	*N-acetyl-glufosinate D_3*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*

* : To check for absence in Blank Material

Since most participating laboratories where not well familiar with liver as matrix, the organizer distributed an updated version of the QuPPe-AO method, in which a procedure for the analysis of highly polar pesticides in liver was described. This method was distributed at the beginning of the registration period. Nevertheless, the participants were free to use this 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 for analysis. Both the order of sample preparation and the order of extract injection into the analytical instruments were random. Matrix-matched calibration using extract prepared from blank material or procedural calibration using blank material were applied for quantification. Analytical portions of 10 g for QuPPe and 5 g for all other compounds were used.

The statistical evaluation of the homogeneity test data was performed according to the International Harmonized Protocols published by IUPAC, ISO and AOAC [4, 6]. An overview of the statistical evaluations of the homogeneity test is shown in **Table 1-5**. 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

Table 1-5: Statistical evaluation of homogeneity test data (n = 20), details please see **Appendix 3**.

	COMPULSORY	OPTIONAL COMPOUNDS						
	Glyphosate	2,4-DB	Avermectin B1a	Bixafen desmethyl	M510F01 (Boscalid met.)	Bromoxynil	DDAC C10-Cl	Fenpropimorph Carboxylic Acid (BF-421-2)
Analytical portion size [g]	10	5	5	5	5	5	5	5
Mean [mg/kg]	0.536	0.058	0.055	0.052	0.081	0.054	0.188	0.082
s_{sam}^2	1.11 x 10^{-4}	0.00 x 10^{-0}	4.58 x 10^{-6}	0.00 x 10^{-0}	3.16 x 10^{-6}	1.26 x 10^{-5}	1.01 x 10^{-4}	5.07 x 10^{-6}
c	4.02 x 10^{-2}	4.37 x 10^{-3}	4.1 x 10^{-3}	3.92 x 10^{-3}	6.10 x 10^{-3}	4.03 x 10^{-3}	1.41 x 10^{-2}	6.13 x 10^{-3}
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed
OPTIONAL COMPOUNDS								
	TFNA-AM (Flonicamid met.)	Fluopyram-benzamid (M25)	MPP	AMPA	N-Acetyl-Glyphosate	Haloxyfop	MCPA	Mepiquat
Analytical portion size [g]	5	5	10	10	10	5	5	5
Mean [mg/kg]	0.072	0.099	0.294	0.749	0.487	0.034	0.047	0.051
s_{sam}^2	0.00 x 10^{-0}	1.39 x 10^{-6}	1.11 x 10^{-4}	1.84 x 10^{-4}	3.02 x 10^{-5}	1.80 x 10^{-6}	0.00 x 10^{-0}	1.38 x 10^{-6}
c	5.38 x 10^{-3}	7.45 x 10^{-3}	2.20 x 10^{-2}	5.62 x 10^{-2}	3.65 x 10^{-2}	2.55 x 10^{-3}	3.52 x 10^{-3}	3.83 x 10^{-3}
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed

when duplicate samples are taken from 10 bottles. $\sigma_{all} = 0.3 \times$ FFP-RSD (25 %) $\times$ the analytical 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-SRM14.

1.8 Storage Stability Test

In the Specific Protocol laboratories were recommended storing the samples in the freezer until analysis. The bottles with the test items that were used for the stability experiments 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. 5)** are applied for these tests. 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, under repeatability conditions) at a day suitable for the laboratory.

Stability test 1 (extraction shortly before shipment):
07 March 2019 (analytes via QuPPe-AO-Methods)
12 March 2019 (analytes via QuEChERS-Methods)

Stability test 2 (extraction 10 days after shipment):
28 March 2019 (analytes via QuPPe-AO-Methods)
29 March 2019 (analytes via QuEChERS-Methods)

Stability test 3 (extraction at the end of PT):
17 April 2019 (analytes via QuEChERS-Methods)
18 April 2019 (analytes via QuPPe-AO-Methods)

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 1-6, p. 8**). 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 1-6 (p. 8)** and **Appendix 4**.

1.9 Transport Stability Test

With the exception of two laboratories outside the EU all other participants received the sample packages within three days and in very good condition. The results reported by the two laboratories that received the material on the fourth and seventh day did not imply any degradation of compounds. For these reasons, the organizer decided to skip the transport stability test in this PT.

Table 1-6: Results of storage stability test (storage at -18 °C). Please see the text under **Section 1.8 (p. 7)** or **Appendix 4** for the dates of analysis of each analyte.

	Glyphosate	2,4-DB	Avermectin B1a	Bixafen desmethyl	M510F01 (Boscalid met.)	Bromoxynil	DDAC C10-Cl	Fenpropimorph Carboxylic Acid (BF-421-2)
Storage at –18 °C (mean values in mg/kg)								
Analysis 1	0.537	0.061	0.064	0.051	0.079	0.060	0.187	0.082
Analysis 2	0.513	0.057	0.066	0.053	0.074	0.060	0.180	0.087
Analysis 3	0.507	0.056	0.060	0.051	0.076	0.056	0.177	0.081
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.033 (-6.22 %)	0.005 (-7.44 %)	0.004 (-6.25 %)	0.001 (-0.97 %)	0.003 (-4 %)	0.004 (-6.93 %)	0.01 (-5.52 %)	0.0003 (-0.41 %)
$0.3 \times \sigma_{pt}$ [mg/kg]	0.04	0.005	0.004	0.004	0.006	0.004	0.013	0.007
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed
	TFNA-AM (Flonicamid met.)	Fluopyram-benzamid (M25)	MPP	AMPA	N-Acetyl-Glyphosate	Haloxyfop	MCPA	Mepiquat
Storage at –18 °C (mean values in mg/kg)								
Analysis 1	0.075	0.100	0.296	0.762	0.498	0.032	0.052	0.054
Analysis 2	0.079	0.106	0.283	0.732	0.485	0.033	0.051	0.056
Analysis 3	0.075	0.098	0.299	0.728	0.485	0.032	0.051	0.053
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.0005 (0.67 %)	0.002 (-4.07 %)	0.003 (1.13 %)	0.033 (-4.38 %)	0.012 (-2.48 %)	0.0003 (-1.05 %)	0.001 (-1.94 %)	0.002 (-3.07 %)
$0.3 \times \sigma_{pt}$ [mg/kg]	0.005	0.008	0.023	0.057	0.041	0.003	0.003	0.004
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed

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 divited 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 (liver). 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 ensure that the information concerning their network is up-to-date and that all obliged OfLs within their network were informed of this EUPT. All NRLs and OfLs were informed that the division into "obliged" and "voluntary" was tentative and that the real obligation for participation is derived from the respective regulations and their actual scope.

Following DG-SANTE instructions, obliged labs not intending to participate in the EUPT-SRM14 were instructed to provide explanations for their non-participation.

1.10.2 Announcement / Invitation and EUPT-SRM14-Website

Following an urgent request by COM, the orginally planned PT matrix (red cabbage) was changed into bovine liver. Thereafter, all laboratories within the network were informed about this change on 16 November 2018. Within the EURL-Web-Portal an EUPT-SRM14-Website was installed with links to all documents relevant to this EUPT (i.e., Announcement/Invitation Letter, Calendar, Target Pesticides List, Specific Protocol and General EUPT Protocol). These documents were uploaded to the EURL-Web-Portal and the CIRCA BC.

The Announcement/Invitation Letter for the EUPT-SRM14 was published on the EUPT-SRM14-Website in December 2018 and was sent to all NRL-SRMs, all OfLs analysing pesticide residues in food and feeding stuff within the framework of official controls, all laboratories performing import controls according to Reg. 669/2009/EC, as far as they were tracked in the EURL-DataPool, as well as to EU laboratories analysing official organic samples within the frame of Reg. 889/2008/EC. The latter laboratories were considered eligible but not obliged to participate. It was indicated to the OfLs that their obligation to participate in EUPTs arises from 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. Due to expected difficulties with the customs when shipping raw material of animal origin, it was decided not to actively invite laboratories based in countries outside the EU. Still, on request, three selected laboratories outside the EU or EFTA were premitted to take part in this exercise, highlighting the risk of having problems with the clearance of the material by the customs. As always, the results from laboratories outside EU were not taken into accout for the establischment of the assigned values.

1.10.3 Registration

Like in previous PTs in 2017 (EUPT-SRM12) and 2018 (EUPT-SRM13) 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

At the request of DG-SANTE a method for the analysis of highly polar pesticides in products of animal origin (including liver) was distributed among the network. Another document concerning the QuEChERS-based analysis of various metabolites relevant to food of animal origin was publushed earlier.

For their information and convenience, the participants were additionally provided with an Excel file named "Supplementary Information on Analytes". It entailed a list of all compounds within the Target Pesticide List showing their relevance (legals status whether they were listed in the SANTE working document etc.), possible analytical approach, exemplary standard providers and the current MRLs in liver. The aim was to facilitate the preparation of the laboratories for their participation in the EUPT-SRM14 by helping them expanding their analytical scope, which was also of high importance to DG-SANTE.

1.10.5 Distribution of the Test Items and the Blank Material

One bottle of test item (approx. 200 g) and one bottle of blank material (approx. 200 g) were shipped on

18 March, 2019 to each participant in thermo-insulated polystyrene boxes with dry ice. The packages for laboratories in destination countries where according to IATA Dangerous Goods Regulations shipments with dry ice were not allowed contained special freezing elements instead of dry ice and were pre-frozen at −80 °C for three days before shipment.

Two weeks prior to the shipment, detailed instructions on how to treat the test item and blank material upon receipt were provided to the participating laboratories in the Specific Protocol (**Appendix 9**).

1.10.6 Webtool for Results Submission and Confidentiality

A new web-based submission tool allowed participants to acknowledge sample receipt and to submit their results and method information online. This new webtool is used by all pesticide EURLs from 2019 onwards. Following their first registration with the webtool, the PT responsible persons receive unique login credentials, which are linked to the stated e-mail-address. Using their personal login credentials, participant are provided with an overview of all pesticide-related EUPTs and with an access to the results submission templates of the relevant EUPTs.

The lab code of a laboratory for a certain PT is obtained when a participant, either as PT main or alternative contact person, logs in to this PT. The personal login credentials and the unique lab code for a certain PT ensure confidentiality. For further information on confidentiality please refer to the General EUPT Protocol (**Appendix 8**).

Via the Webtool, all participants had access to the websites for sample receipt acknowledgement and results submission. These sites were available from the day following sample shipment until the result submission deadline (23 April, 2019). One week prior to sample shipment, the organizer provided the participants with a guideline on how to use the new Webtool (**Appendix 12**), including information on how to log-in, on how to get the lab code for the EUPT-SRM14, and on which fields would need to be completed (e.g. laboratory's reporting limits, initial sample temperature and extraction time).

After the deadline, participants were informed on the pesticides present in the test item and whether they had false negative results. In the latter case, they were prompted to fill-in their method details via the Webtool. The schedule of sample shipment and submission deadline was embedded in a workflow of the Webtool.

Based on the dates of sample shipment and submission deadline, which were embedded in the Webtool reminder e-mails prompting the labs to make certain actions were automatically sent to the participants. As the Webtool was operated for the first time, it contained a few errors, so that the participants received for example incorrect information on false negative results. The software developers are working to fix these errors.

1.10.7 Actions following Results Submission, Preliminary Report and Survey

One laboratory that had originally registered to participate in the current PT but finally did not submit any results, was asked to provide explanations. On 10 May, 2019, the preliminary report on the EUPT-SRM14 with the preliminary assigned values was sent to the participants. Due to some errors in the access permission policies within the software, the PT-organizers were not able to download the complete data-sets. As this error only concerned very few laboratories, it was not noticed and resulted in “transcription” errors in the preliminary report. An updated report with marginal changes in the assigned values and z-score of 12 compounds was issued on 21 May.

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-score | > 2 and to report them.

In order to obtain feedback from the participants and to improve the service quality in the future, parallel to the release of the preliminary report the organisers invited the participants to participate in a survey on EUPT-SRM14 from 10 to 29 May, 2019. This survey contained 5 questions on the organisation (general, registration, information and instruction provided, shipment/delivery, test item, blank material and webtool), on the relevance of the used matrix (bovine liver) to the routine work, on the assigned values of the analytes, as well as on the preliminary report and wishes as regards the commodities and/or analytes to be included in the upcoming EUPT-SRMs. 46 of 59 participants (78 %) in EU or EFTA countires and one of the three participants outside EU took part in the survey. The evaluation and compilation of comments was published on 12 June and can be downloaded via http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_Survey_Evaluation.pdf.

2. EVALUATION RULES

2.1 False Positives and Negatives

2.1.1 False Positives (FPs)

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

2.1.2 False Negatives (FNs)

These are results of target analytes reported as "analysed" but without reporting numerical values, although they were used by the organiser to prepare the test item and were detected, at or above the MRRL, by the organiser and the overwhelming majority of the participating laboratories. In accordance with the General Protocol z-scores for false negatives are calculated using the MRRL as the result, or using the lab's reporting-limit (RL), if this is lower 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 analyte was present in the sample at a concentration allowing such a judgement (sufficient distance from MRRL) and irrespective of whether the analyte concentration was lower than the RL.

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 (according to 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 being 10^{-6}.

The uncertainty of the assigned value of each analyte $u(x_{pt})$ 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 correction factor 1.25 is based on the typical standard deviation of the median in a large set of results drawn from a normal distribution.

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

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

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

2.4 z-Scores

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

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

Where

- x_i is the result for the target analyte (i) as reported by the participant
  (For results considered as false negatives, x_i is set as equal to the respective minimum required reporting level (MRRL) or the laboratory reporting limit (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 **2.5.1**).

The z-scores are classified as follows:

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

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

2.5 Laboratory Classification

In current PT it was decided not to classify the laboratories based on the covered analyte scope due to the very limited number of mandatory analytes.

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

62 laboratories from 29 countries (24 EU-Member States, 2 EFTA- countries, and 3 countries outside Europe) originally registered for participation in the EUPT-SRM14. An overview of the participating laboratories and countries is given in **Table 3-1**. A list of all individual laboratories that registered for this EUPT is presented in **Appendix 1**. Croatia was the only EU-country not represented by an NRL-SRM, as the NRL-SRM of this country had not yet been appointed at that time. Malta was represented by its proxy-NRL-SRM based in the United Kingdom.

Out of the 57 EU OfLs having registered for participation in the current PT two laboratories, one performing official food control activities on behalf of Begium and the other one on behalf of Spain, failed to submit any results. The former one reported still before the submission deadline that the matrix was out of its routine scope and that it could not manage to finish the analysis before the deadline. The other laboratory did not provide any explanation for its non-submission of results.

All 5 laboratories from non-EU countries submitted results (2 from EFTA countries and 3 from countries elsewhere). The results submitted by the 3 laboratories located outside EU and EFTA countries were not taken into account when calculating the assigned values.

In total, 123 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 animal 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.

54 obliged laboratories explained their non-participation with the fact that the matrix (bovine liver) or the SRM14 target pesticides or both were out of their routine scope. One obliged laboratory was not able to participate because of a relocation to other premisses. Excluding those 54 laboratories that provided sufficient explanations, the number of EU-laboratories considered as being obliged decreased to 69. Among the 53 obliged laboratories that have registered for this PT, 51 laboratories finally submitted results. In addition, 6 OfLs registered for participation on voluntary basis, and all of them submitted results. Out of the 69 obliged OfLs 16 (23 %) did neither register for the PT nor provide any explanation for non-participation. These laboratories originated from 8 countries as follows: FR (5×), IT (3×), ES (3×), and each one from HR, DE, GR, PL, and RO.

Table 3-1: Number of laboratories listed as being obliged to participate in the EUPT-SRM14, labs that registered to participate, and labs that finally submitted results (grouped by contracting country)

EU: NRLs and OfLs											
				Registered for Participation [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	All	NRL-SRMs/ AOs	NRL-SRMs	NRL-AOs	All	NRLs	Obliged labs non particip. w/o giving expl.	Notes
AT	2	1	1	1	1			1	1		
BE	7	2	5	5 + [1]	1			5	1		
BE; LU	1		1	1				1			
BG	2	2							0*		Matrix out of scope of NRL-SRM
CY	1		1	1	1			1	1		
CZ	4	1	3	3		1	1	3	2		
DE	14	5	9	8 + [1]	1			9	1	1	
DK	2	2							0*		Matrix out of scope of NRL-SRM
EE	2	1	1	1	1			1	1		
FR	12	5	7	2 + [1]	1			3	1	5	
FI	2	1	1	1	1			1	1		
GR	5	2	3	2	2			2	2	1	GR has appointed two NRL-SRMs.
HR	3	1	2	1				1	0*	1	HR has not yet established an NRL-SRM.
HU	4		4	4	1			4	1		
IE	2	1	1	1	1			1	1		
IT	15	7	8	5	1			5	1	3	
LT	2		2	2	1			2	1		
LU	1	1							0*		Matrix out of scope of NRL-SRM
LV	1		1	1	1			1	1		
MT	1*		1*	1*				1*			*MT-NRL-SRM represented by proxy by the UK-NRL-SRM; this obliged OfL in DE is subcontracted by MT for routine analysis.
NL	2		2	2	1		1	2	2		
PL	7	6	1						0*	1	Matrix out of scope of NRL-SRM
PT	2	1	1	1		1		1	1		
RO	4	2	2	1	1			1	1	1	
SE	2		2	2	1			2	1		
SI	1		1	1	1			1	1		
SK	3	2	1	1	1			1	1		
ES	17	10	7	4 + [1]	1			4	1	3	
UK	1	1									
UK; MT	1		1	1	1			1	1		
EU-total	**123**	**54**	**69**	**55 + [4]**	**21**	**2**	**2**	**51 + [4]**	**25**	**16**	

Table 3-1 (cont.): Number of laboratories listed as being obliged to participate in the EUPT-SRM14, labs that registered to participate, and labs that finally submitted results (grouped by contracting country)

EU: NRLs and OfLs											
				Registered for Participation [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	All	NRL-SRMs/AOs	NRL-SRMs	NRL-AOs	All	NRLs	Obliged labs non particip. w/o giving expl.	Notes
NO				[1]	1			[1]	1		
CH				[1]	–			[1]	–		
EU+EFTA Total				53 + [6]				51 + [6]	25	16	
Countries outside Europa											
CA				1				1			
HK/CN				1				1			
TH				1				1			
Countries outside Europa Total				3				3			
Countries outside Europa Total				62				60	25	16	

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. 22)** 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. 34)**. Detailed information about the analytical methods used by the laboratories is shown on the web under "EUPT-SRM14 - Supplementary Information" accessible via the the link: http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_Supplementary_Information.pdf.

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

Compounds		Present in test item	Labs analysed for the compound			
			EU [1]- and EFTA-Labs		EU obliged Labs only	
			No. [2]	% (based on *n* = 57 [3])	No. [2]	% (based on *n* = 69 [4])
	Glyphosate	**Yes**	43	75 %	37	54 %
Optional Compounds	**2,4-D**	No	43	75 %	38	55 %
	2,4-DB	**Yes**	37	65 %	34	49 %
	5 OH-Thiabendazole	No	21	37 %	20	29 %
	Avermectin B1a	**Yes**	40	70 %	35	51 %
	BAC C12	No	33	58 %	28	41 %
	Bixafen desmethyl	**Yes**	20	35 %	19	28 %
	Boscalid Met. M510F017	**Yes**	18	32 %	17	25 %
	Bromoxynil	**Yes**	35	61 %	30	43 %
	Chlorate	No	32	56 %	26	38 %
	Chlormequat	No	44	77 %	38	55 %
	DDAC-C10	**Yes**	31	54 %	27	39 %
	Dicamba	No	29	51 %	25	36 %
	Dichlorprop	No	41	72 %	36	52 %
	Emamectin B1a	No	41	72 %	36	52 %
	Ethephon	No	40	70 %	34	49 %
	Fenpropimorph carb. acid (BF-421-2)	**Yes**	11	19 %	10	14 %
	TFNA-AM (Flonicamid Met.)	**Yes**	24	42 %	22	32 %
	Fluazifop	No	44	77 %	38	55 %
	Fluopyram-benzamide (M25)	**Yes**	23	40 %	22	32 %
	Glufosinate	No	27	47 %	24	35 %
	MPP	**Yes**	20	35 %	18	26 %
	N-Acetyl-glufosinate	No	19	33 %	17	25 %
	AMPA	**Yes**	29	51 %	26	38 %
	N-Acetyl-glyphosate	**Yes**	16	28 %	15	22 %
	Haloxyfop	**Yes**	42	74 %	36	52 %
	Isoxaflutole diketonitrile Met. (RPA202248)	No	18	32 %	18	26 %
	MCPA	**Yes**	45	79 %	40	58 %
	MCPB	No	39	68 %	34	49 %
	Mepiquat	**Yes**	46	81 %	40	58 %
	Quizalofop	No	34	60 %	29	42 %
	Triclopyr	No	39	68 %	34	49 %

1) Including official laboratories participating on voluntary basis
2) Laboratories representing more than one country were counted only once.
3) 57 OfLs from EU and EFTA countries (incl. NRLs and OfLs participating on voluntary basis) have submitted at least one result.
4) 69 OfLs (including NRLs) from EU countries were finally considered as obliged to participate in the EUPT-SRM14 (taking into account any explanations for non-participation).

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

Compound listed on Target List		Glyphosate	2,4-D	2,4-DB	5 OH-Thiabendazole	Avermectin B1a	BAC-C12	Bixafen desmethyl	Boscalid Met. M510F017	Bromoxynil	Chlorate	Chlormequat	DDAC-C10	Dicamba	Dichlorprop	Emamectin B1a	Ethephon
within [1]		MACP-Reg.		NCP-WD			NCP-WD	NCP-WD	NCP-WD		NCP-WD	NCP-WD	NCP-WD	NCP-WD		NCP-WD	
present in Test Item		**Yes**	**No**	Yes	**No**	Yes	**No**	**Yes**	**Yes**	**Yes**	**No**	**No**	**Yes**	**No**	No	No	No
evaluated in this PT		**Yes**	**No**	Yes	**No**	Yes	**No**	**Yes**	**Yes**	**Yes**	**No**	**No**	**Yes**	**No**	No	No	No
Lab-Code SRM14-	NRL																
950		V		V	ND	V	ND	V	V	V	ND	ND	V	ND	ND	ND	ND
956	x	V	ND	**FN**				V	V	V	ND	ND			ND	ND	ND
992		V	ND	V	ND	V	ND	V	V	V	ND	ND	V	ND	ND	ND	ND
1022					ND			V	**FN**								
1024						V										ND	
1072	x	V	ND	V		V	*(FP)*			V		ND	V	ND	ND	ND	ND
1090	x	V	ND	V	ND	V	ND	V	V	V	ND	ND	V	ND	ND	ND	ND
1092	SRM	V	ND	V		V	ND				ND	ND	**FN**	ND	ND		ND
1150				V									V				
1206		V	ND	V	ND	V	ND	V	V	V	ND	ND	V	ND	ND	ND	ND
1214		V	ND	V		V	*(FP)*			V	ND	ND	V	ND	ND	ND	ND
1218		V	ND	V		V	ND			V	ND	ND	V	ND	ND		ND
1224		V			ND	V						ND				ND	
1228		V	ND									ND			ND	ND	ND
1240			ND	V		V								ND		ND	
1244	x	V	ND	V		V				V	ND	ND			ND	ND	ND
1248	AO	V															
1250	x	V	ND		ND	V	ND										
1266		V	ND	V	ND	V	ND	V	V	V	ND	ND	V		ND	ND	ND
1270		V	ND	V		V				V	ND	ND		ND	ND	ND	ND
1274	x	V	ND			V	ND				ND	ND	V	ND	ND	ND	ND
1276		V	ND	V		V	ND			**FN**	ND	ND	V		ND	ND	ND
1278	x	V	ND			V						ND				ND	ND
1290	x		ND	**FN**								ND			ND		
1292		V	ND	V			ND	V	V	V	ND	ND	V	ND	ND	ND	ND
1298			ND	V	ND	V				V				ND	ND	ND	
1300	x	V	ND	V		V	ND			V		ND	V	ND	ND	ND	ND
1302	x		ND	V			ND	V	V		ND	ND	V				

1) MACP-Reg.: REGULATION (EU) 2018/555 of 9 April 2018; 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)

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

		Optional Compounds																Total
Optional / Additional Compound listed in Target List		Fenpropimorph carb. acid (BF-421-2)	TFNA-AM (Flonicamid Met.)	Fluazifop	Fluopyram-benzamide (M25)	Glufosinate	MPP	N-Acetyl-Glufosinate	AMPA	N-Acetyl-glyphosate	Haloxyfop	Isoxaflutole diketonitrile Met. (RPA202248)	MCPA	MCPB	Mepiquat	Quizalofop	Triclopyr	analysed / correctly found within the EUPT-Target Pesticides List (max. 32 / 16)
within [1]		NCP-WD		NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD		
present in Test Item		**Yes**	Yes	**No**	**Yes**	No	Yes	**No**	Yes	**Yes**	**Yes**	**No**	**Yes**	**No**	**Yes**	**No**	**No**	
evaluated in this PT		**Yes**	Yes	**No**	**Yes**	No	Yes	**No**	Yes	**Yes**	**Yes**	**No**	**Yes**	**No**	**Yes**	No	No	
Lab-Code SRM14-	NRL																	
950			V	ND	V	ND	V	ND	V	V	V	ND	V	ND	V	ND	ND	30 / 15
956	x		**FN**	ND	V	ND	V	ND	V	V	V		V	ND	V	ND	ND	25 / 11
992		V	V	ND	V	ND	V	ND	V	V	V		V	ND	V	ND	ND	31 / 16
1022																		3 / 1
1024																		2 / 1
1072	x			ND							V		V	ND	V		ND	18 / 8
1090	x	V	V	ND	V	ND	V	ND	V	V	V	ND	V	ND	V	ND	ND	32 / 16
1092	SRM			ND		ND	V		**FN**	V	V		V	ND	V	ND	ND	22 / 8
1150													V		V			4 / 4
1206		V	V	ND	V	ND	V	ND	V	V	V	ND	V	ND	V	ND	ND	32 / 16
1214				ND		ND	V		V		V	ND	V	ND	V	ND	ND	24 / 10
1218			**FN**	ND		ND			V		V		V	ND	V	ND	ND	22 / 9
1224									V						V			7 / 4
1228			V	ND		ND	V		V		V		V		V	ND	ND	16 / 7
1240			V	ND							V		V	ND		ND		11 / 5
1244	x			ND	V	ND	V	ND	V	V	V		V	ND	V	ND	ND	23 / 11
1248	AO																	1 / 1
1250	x			ND	V								V		V			9 / 5
1266		V	V	ND	V	ND	V	ND	V	V	V	ND	V	ND	V	ND	ND	31 / 16
1270				ND	V						V		V	ND	V	ND	ND	19 / 8
1274	x			ND							V		V	ND	V	ND	ND	18 / 6
1276				ND		ND	V	ND	V	V	V		V	ND	V	ND	ND	24 / 10
1278	x			ND					V		V		V	ND	V			12 / 6
1290	x			ND							V		V	ND	V	ND	ND	11 / 3
1292			V	ND	V	ND		ND	V		V	ND	V	ND	V		ND	26 / 12
1298				ND							V	ND	V	ND	V	ND	ND	16 / 6
1300	x			ND		ND			V		V		V		V		ND	19 / 9
1302	x		**FN**		V								V	ND	V			13 / 7

1) MACP-Reg.: REGULATION (EU) 2018/555 of 9 April 2018; 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)

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

Compound listed on Target List		Glyphosate	2,4-D	2,4-DB	5 OH-Thiabendazole	Avermectin B1a	BAC-C12	Bixafen desmethyl	Boscalid Met. M510F017	Bromoxynil	Chlorate	Chlormequat	DDAC-C10	Dicamba	Dichlorprop	Emamectin B1a	Ethephon
within [1]		MACP-Reg.		NCP-WD			NCP-WD	NCP-WD	NCP-WD		NCP-WD	NCP-WD	NCP-WD	NCP-WD		NCP-WD	
present in Test Item		**Yes**	**No**	Yes	**No**	Yes	**No**	**Yes**	**Yes**	**Yes**	**No**	**No**	**Yes**	**No**	No	No	No
evaluated in this PT		**Yes**	**No**	Yes	**No**	Yes	**No**	**Yes**	**Yes**	**Yes**	**No**	**No**	**Yes**	**No**	No	No	No
Lab-Code SRM14-	NRL																
1306		V					ND			V	ND	ND	V			ND	ND
1310		V	ND	V		V				V	ND	ND		ND	ND	ND	ND
1312	x	V	ND	V	ND	V	ND	V	V	V	ND	ND	V		ND	ND	ND
1318	x	V	ND			**FN**	ND			V		ND	V		ND	ND	ND
1320		V	ND	V	ND	V	ND	V	V	V	ND	ND	V	ND	ND	ND	ND
1322			ND	V	ND	V	ND			V				ND	ND	ND	ND
1324		V	ND			V	ND			V	ND	ND			ND	ND	ND
1330	x	V	ND	V		V	ND			V		ND	V		ND	ND	ND
1332		V	ND	V	ND	V	*(FP)*	V		V	ND	ND	V		ND	ND	ND
1336	x	V															ND
1338	x	V	ND	V				V	V	V	ND	ND		ND	**FP**		ND
1340	x		ND				ND	V				ND	V	ND	ND	ND	
1342	x	V	ND	V		V						ND		ND	ND	ND	ND
1346						V											
1350		V									ND	ND					ND
1352	x	V	ND	V		V				V	ND	ND		ND	ND	ND	ND
1354	x	V	ND	V	ND	V	ND			V	ND	ND		ND	ND	ND	ND
1356		V	ND		ND		ND			V	ND	ND	V	ND	ND	ND	ND
1358		V	ND	V		V	ND	V	V	V	ND	ND	V	ND	ND	ND	ND
1364							ND	V	V				V		ND		
1366		V					ND						V				ND
1368	x	V	ND	V		V	ND			V	ND	ND	V	ND	ND	ND	ND
1370	x				ND	V						ND				ND	
1392	x		ND	V	ND	V				V				ND	ND	ND	
1394		V	ND	V	ND	V	ND	V	V	V	ND	ND	V	**ND**	ND	ND	ND
1396												ND					
1398	AO	V	ND	V	ND	V		V	V	V	ND	ND	V		ND	ND	

1) MACP-Reg.: REGULATION (EU) 2018/555 of 9 April 2018; 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)

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

		Optional Compounds															Total
Optional / Additional Compound listed in Target List		Fenpropimorph carb. acid (BF-421-2)	TFNA-AM (Flonicamid Met.)	Fluazifop	Fluopyram-benzamide (M25)	Glufosinate	MPP	N-Acetyl-Glufosinate	AMPA	N-Acetyl-glyphosate	Haloxyfop	Isoxaflutole diketonitrile Met. (RPA202248)	MCPA	MCPB	Mepiquat	Quizalofop	Triclopyr	analysed / correctly found within the EUPT-Target Pesticides List (max. 32 / 16)
within [1]		NCP-WD		NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD		
present in Test Item		**Yes**	Yes	**No**	**Yes**	No	Yes	**No**	Yes	**Yes**	**Yes**	**No**	**Yes**	**No**	**Yes**	**No**	**No**	
evaluated in this PT		**Yes**	Yes	**No**	**Yes**	No	Yes	**No**	Yes	**Yes**	**Yes**	**No**	**Yes**	**No**	**Yes**	No	No	
Lab-Code SRM14-	NRL																	
1306				ND							V				V			11 / 5
1310				ND							V		V	ND	V	ND	ND	18 / 7
1312	x	V	V	ND	V	ND	V	ND			V	ND	V	ND	V	ND	ND	29 / 14
1318	x			ND					V		V		V	ND	V			16 / 7
1320		V	V	ND	V	ND	V	ND	V	V	V	ND	V	ND	V	ND	ND	32 / 16
1322			V	ND							V	ND	V	ND		ND	ND	18 / 6
1324				ND		ND	V	ND	V		**FN***		V	ND	V	ND	ND	21 / 7
1330	x			ND		ND	V	ND			V		V	ND	V		ND	20 / 9
1332			V	ND	V						V	ND	V	ND	V	ND	ND	24 / 11
1336	x								V									3 / 2
1338	x	V	V	ND	V	ND	V	ND	V	V	V	ND	V	ND	V	ND	ND	27 / 14
1340	x	**FN**		ND	V						V		V		V		ND	15 / 6
1342	x		**FN**	ND							V		V	ND	V	ND	ND	17 / 6
1346																		1 / 1
1350						ND			V						V			7 / 3
1352	x			ND						V	V		V	ND	V	ND	ND	19 / 8
1354	x		V	ND	V						V	ND	V	ND	V	ND	ND	23 / 9
1356			V	ND		**FP**			V		V		V	ND	V	ND	ND	22 / 8
1358				ND	V	ND		ND	V	V	V	ND	V	ND	V		ND	27 / 13
1364					V													6 / 4
1366						ND	V	ND	V	V								9 / 5
1368	x		**FN**	ND		ND	V	ND	V	V	V		V	ND	V	ND	ND	26 / 11
1370	x																	4 / 1
1392	x			ND							V		V	ND		ND	ND	14 / 5
1394		V	V	ND	V	ND			V		V	ND	V	ND	V	ND	ND	29 / 14
1396															V			2 / 1
1398	AO	V	V	ND	V		V	ND				ND	V		V	ND	ND	24 / 13

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within [1]		MACP-Reg.		NCP-WD			NCP-WD	NCP-WD	NCP-WD		NCP-WD	NCP-WD	NCP-WD	NCP-WD		NCP-WD	
present in Test Item		**Yes**	**No**	Yes	**No**	Yes	**No**	**Yes**	**Yes**	**Yes**	**No**	**No**	**Yes**	**No**	No	No	No
evaluated in this PT		**Yes**	**No**	Yes	**No**	Yes	**No**	**Yes**	**Yes**	**Yes**	**No**	**No**	**Yes**	**No**	No	No	No
Lab-Code SRM14-	NRL																
1400		V	ND	V	ND	V	ND	V	V	V	ND	ND	V	ND	ND		ND
1402	SRM	V	ND	V	ND	V	ND	V	V	V	ND	ND	V	ND	ND	ND	ND
978		V	ND			V											ND
1404			ND		ND					V				ND		ND	
1406		V		V	ND	V	**FP**			V		ND	**FN**	ND	ND	ND	

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		Optional Compounds																Total
Optional / Additional Compound listed in Target List		Fenpropimorph carb. acid (BF-421-2)	TFNA-AM (Flonicamid Met.)	Fluazifop	Fluopyram-benzamide (M25)	Glufosinate	MPP	N-Acetyl-Glufosinate	AMPA	N-Acetyl-glyphosate	Haloxyfop	Isoxaflutole diketonitrile Met. (RPA202248)	MCPA	MCPB	Mepiquat	Quizalofop	Triclopyr	analysed / correctly found within the EUPT-Target Pesticides List (max. 32 / 16)
within [1]		NCP-WD		NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD	NCP-WD		
present in Test Item		**Yes**	Yes	**No**	**Yes**	No	Yes	**No**	Yes	**Yes**	**Yes**	**No**	**Yes**	**No**	**Yes**	**No**	**No**	
evaluated in this PT		**Yes**	Yes	**No**	**Yes**	No	Yes	**No**	Yes	**Yes**	**Yes**	**No**	**Yes**	**No**	**Yes**	No	No	
Lab-Code SRM14-	NRL																	
1400			V	ND	**FN**	ND			V		V	ND	V	ND	V	ND	ND	27 / 12
1402	SRM	V	V	ND	V	ND	V	ND	V	V	V	ND	V	ND	V	ND	ND	32 / 16
978						ND							V					6 / 3
1404			V	ND							V		V	ND		ND	ND	12 / 4
1406			V	ND	V	**FP**			V		V	ND	**FN**	ND	V	ND		22 / 9

1) MACP-Reg.: REGULATION (EU) 2018/555 of 9 April 2018; 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 Analysis of Blank Material

In 16 cases (**Table 4-3)** the laboratories reported numerical results for taget pesticides in the blank material. In 12 of those cases, concerning ***DDAC-C10***,(5x), *BAC-C12* (2x), ***glyphosate*** (1x), *chlorate* (1x) and ***AMPA*** (1x) the reported concentrations were lower than the respective MRRLs. In 10 out of those 12 cases the concentrations reported were even lower than the RLs of the laboratories. In 4 out of the 16 cases concerning ***glyphosate***, *BAC-C12*, *glufosinate* and ***AMPA*** (1x each) the reported concentrations in the blank material were higher than the MRRLs and the labs' RLs. All these findings could not be verified by the organizer and were also not reported by other participants. Nevertheless, *BAC-C12*, ***DDAC-C10*** and chlorate are often present as background levels as they are related to sanitation measures and thus ubiquitary. In the case of *BAC-C12* and ***DDAC-C10*** laboratory-borne background levels are not uncommon. Two of the labs having reported *BAC-C12* levels in the blank have also reported false positive results in the test Item (see Table 4-5). Two of the laboratories having reported *BAC-C12* findings in the blank (SRM14-1332 and 1214) have also reported findings in the test item at the very same levels. The affected laboratories are prompted to investigate the reasons for the background levels and to take measures to reduce them. The laboratory with the code SRM14-1406 (3rd) reported positive results in the blank material for 3 highly polar compounds, namely *glufosinate*, ***glyphosate*** and ***AMPA***. Furthermore, this lab has also reported two false positive results in the test item (concerning *glufosinate* and *BAC-C12*) as well as two false negative results. This lab should check its quality criteria as regards compound identification and the possibility of a cross-contamination or an extract mismatch within the lab.

4.3 Assigned Values and Target Standard Deviations

The assigned value (x_{pt}) of each analyte present in the test item was established as the mean of robust statistics (x^*) of all numerical results submitted by laboratories from EU and EFTA countries, excluding outliers, and calculated according to Algorithm A [6, **Appendix 8**]. Results from third country laboratories were not

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

Compound	MRRL [mg/kg]	Assigned Value [mg/kg]	Conc. in Blank Material [mg/kg]	Conc. in Test Item [mg/kg]	RL [mg/kg]	Reported by
Glyphosate	0.1	0.535	0.005	0.502	0.01	SRM14-1400
			1.6	1.8	0.1	SRM14-1406 (3rd)
BAC-C12-Cl	0.03	–	0.001	0.0102	0.03	SRM14-1332
			0.005	0.005	0.005	SRM14-1214
			0.054	–	0.03	SRM14-1340
Chlorate	0.01	–	0.0012	–	0.01	SRM14-1332
DDAC-C10-Cl	0.03	0.177	0.002	0.182	0.03	SRM14-1206
			0.0035	0.199	0.03	SRM14-1332
			0.0134	0.139	0.03	SRM14-1274
			0.015	0.146	0.03	SRM14-1400
			0.018	0.175	0.01	SRM14-1218
			0.02	0.272	0.05	SRM14-1398
			0.014 *	0.144 *	0.005	SRM14-1214
Glufosinate	0.1	–	**0.36**	0.59 (= FP)	0.1	SRM14-1406 (3rd)
AMPA	0.1	0.754	0.006	0.753	0.01	SRM14-1400
			2.3	3.1	0.1	SRM14-1406 (3rd)

* The participant originally reported 14 (in blank material) and 144 (in test item), but in retrospect he noticed that these values referred to µg/kg, not mg/kg as requested.

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

Assigned Value and CV^* Based on the Entire Population of Results from EU and EFTA Laboratories								
	Compound	No. of FNs	No. of numerical results (EU+EFTA)	Assigned Value [mg/kg]	$u(x_{pt})$ [1] [mg/kg]	$u(x_{pt})$ Tolerance [mg/kg]	Judgement for UAV-test	CV^* [2] [%]
	Glyphosate	0	42 + 1*	0.535	+/-0.023776	0.0401	passed	23.3
Optional Compounds	2,4-DB	2	35	0.061	+/-0.0024331	0.0046	passed	19.0
	Avermectin B1a	1	39	0.058	+/-0.0033607	0.0044	passed	29.2
	Bixafen desmethyl	0	19 + 1*	0.05	+/-0.0028052	0.0038	passed	20.0
	Boscalid Met. M510F017	1	17	0.081	+/-0.0032065	0.0061	passed	13.1
	Bromoxynil	1	34	0.059	+/-0.001913	0.0044	passed	15.3
	DDAC-C10-Cl	1	29 + 1*	0.177	+/-0.0079871	0.0133	passed	19.8
	Fenpropimorph carboxylic acid (BF-421-2)	1	10 + 1#	0.089	+/-0.0042635	0.0067	passed	11.4
	Flonicamid Met. TFNA-AM	5	19	0.073	+/-0.0045539	0.0055	passed	21.8
	Fluopyram-benzamide (M25)	1	22	0.101	+/-0.0033405	0.0076	passed	12.4
	MPP	0	20	0.309	+/-0.0176858	0.0232	passed	20.5
	AMPA	1	28	0.754	+/-0.0337584	0.0566	passed	19.0
	N-Acetyl-glyphosate*	0	13 + 3*	0.543	+/-0.0519711	0.0407	failed	27.6
	Haloxyfop	1*	42	0.037	+/-0.0015315	0.0028	passed	21.4
	MCPA	0	45	0.046	+/-0.0021739	0.0035	passed	25.3
	Mepiquat-Cl	0	46	0.051	+/-0.0015894	0.0038	passed	16.9
	Average [3] CV^*							**19.8**

1: $u(x_{pt})$: Uncertainty of assigned value calculated as shown under **Section 2.2 (p. 38)**
2: CV^*: Relative standard deviation based on robust statistics
3: The average CV^* is given for information purposes only. CV^*s of individual compounds or average CV^*s of individual compounds or related compounds over many PTs are more meaningfull and conclusive.
*: Outliers and excluded from the population for establishment of the assigned values
#: The mean value of the homogeneity test was included in order to receive sufficient results for a relaible statistical evaluation.

taken into account. Based on these assigned values, z-scores were calculated for all submitted results using the FFP-approach (**Section 4.4.3, p. 31**), and a preliminary report was released on 10 May 2019. Due to an error in the access permission policies within the software the PT organizers were initially not able to download the complete data-sets. As this error only concerned very few laboratories it was not noticed and resulted in "transcription" errors within the preliminary report. A revised report was released on 21 May, 2019.

The uncertainties ($u(x_{pt})$) of the assigned values were calculated as described under **Section 2.2 (p. 13)**.

Before calculating the assigned values, the result population of each analyte was checked for outliers via Grubbs's test. In the case of ***N-acetyl-glyphosate*** the lowest value (0.0438 mg/kg) and the two highest values (1.889 mg/kg and 2.01 mg/kg) were visually far away from the rest, but based on the Grubb's test they were not significant outliers and, still considered in the AV-calculation for the preliminary report. Nevertheless, any of the two highest values would have been a Grubb's outlier if observed alone. The lowest value was lower than the MRRL (0.1 mg/kg) and by a factor of 8 lower than its closest neighbour (0.358 mg/kg). After consultation with the Scientific Committee these three values were excluded from the population used for the establishment of the assigned value of ***N-acetyl-glyphosate***.

In the case of ***Fenpropimorph carboxylic acid (BF-421-2)*** there were only 10 numerical results reported by the participants with CV^* = 12.1 %. In order to have sufficient results for a reliable statistical caluculation (n = 11), after consultation with the Scientific Committee the average of the homogeneity test was included in the calculation of the assigned value of ***Fenpropimorph carboxylic acid (BF-421-2)***.

For the above mentioned reasons the final assigned values and CV^* of ***N-acetyl-glyphosate*** and ***Fenpropimorph carboxylic acid (BF-421-2)*** differ from those in the preliminary report.

The assigned values and their uncertainties are shown in **Table 4-4**. The CV^* of ***avemectin B1a*** (29.2 %), ***N-acetyl-glyphosate*** (27.6 %) and ***MCPA*** (25.3 %) were slightly higher than the FFP-RSD of 25 %. The CV^*-values of all other analytes were lower than the FFP-RSD. The average CV^*s of all analytes based on the entire population of EU-and EFTA-laboratories was 19.8 %. In particular in the cases of ***fenpropimorph carboxylic acid*** (n = 10 + 1[1], CV^* = 11.4 %), ***fluopyram-benzamide (M25)*** (n = 22, CV^* = 12.4 %) and ***Boscalid Met. M510F017*** (n = 17, CV^* = 13.1 %) the CV^*-values were significantly lower than the FFP-RSD of 25 %, although very few participants reported results for those analytes. This pattern has been observed several times in the past PTs and can be explained by the fact that such "rare" analytes are mainly targeted by very experienced and well-perfroming laboratories which results in a narrow distribution of the PT-results.

4.4 Assessment of Laboratory Performance

4.4.1 False Positives

In total, 7 numerical results were submitted by 6 laboratories for analytes which were not spiked to the test material and which were not detected neither by the organizers nor by the overwhelming majority of the participants. Three of those results, all concerning *BAC-C12*, were lower than the MRRL and were therefore not judged as false positives. The reported values of the other four results, *glufosinate* (2×), *BAC-C12* (1×), and *dichlorprop* (1×), were at least 5 times higher than the respective MRRLs and were therefre judged as false positive results (**Table 4-5**). Two of those four false positive results were reported by EU/EFTA laboratories (1 each) and the other two by one laboratory outside EU.

Table 4-5: Overview of false positive and potentially false positive results reported by participating laboratories

	Compound	PT-Code	Analysed	Reported Result [mg/kg]	RL [mg/kg]	MRRL [mg/kg]	Judgement
Optional Compounds	BAC-C12	SRM14-1072	Yes	0.018	0.03	0.03	–
		SRM14-1214	Yes	0.005	0.005		–
		SRM14-1332	Yes	0.0102	0.03		–
		SRM14-1406 (3rd)	Yes	0.16	0.03		FP
	Dichlorprop	SRM14-1338	Yes	0.0770	0.01	0.01	FP
	Glufosinate	SRM14-1356	Yes	0.135	0.1	0.1	FP
		SRM14-1406 (3rd)	Yes	0.59	0.1		FP

1 The mean value of homogeneity test was added to the result population for the establishment of the assigned value

4.4.2 False Negatives

14 laboratories reported in 17 cases "analysed, but not detected" for target compounds which were spiked to the test item and detected by the majority of the laboratories targeting them (**Table 4-6, p. 31**). This concerned the following compounds ***TFNA-AM*** (5×), ***2,4-DB*** (2×), ***DDAC-C10*** (2×), and ***AMPA***, ***avermectin B1a***, ***boscalid Met. M510F017, bromoxynil, fenpropimorph carboxylic acid (BF-421-2), fluopyram-benzamide (M25), haloxyfop***, and ***MCPA*** (1× each). All these compounds were listed as optional within the target list. Two of the false negative results (***DDAC-C10*** and ***MCPA***) were reported by one participant outside the EU; the other 15 false negative results were reported by laboratories from EU and EFTA countries and represented 3.4 % of the total 437 results reported by this group of laboratories for optional target compounds present in the test item. The false negative result for ***haloxyfop*** resulted from the fact that the laboratory's reporting limit at 0.5 mg/kg was 13.8 times higher than the assigned value 0.036 mg/kg. In accordance with the rules in the General Protocol it was still judged as a false negative result. This laboratory is encouraged to improve its RL for ***haloxyfop***.

4.4.3 Laboratory Performance Based on z-Scores

All individual z-scores were calculated using the FFP-RSD of 25 % and the assigned values derived from the entire population of results received from EU/EFTA laboratories excluding outliers with the exception of ***fenpropimorph carboxylic acid (BF-421-2)*** where the population was supplemented with the mean value

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

	Compound	PT-Code	Analysed	Detected	RL [mg/kg]	MRRL [mg/kg]	Assigned Value [mg/kg]	Judgement
Optional Compounds	2,4-DB	SRM14-956	Yes	No	0.01	0.01	0.061	False Negative
		SRM14-1290	Yes	No	0.01			False Negative
	Avermectin B1a	SRM14-1318	Yes	No	0.05	0.01	0.057	False Negative
	Boscalid Met. M510F017	SRM14-1022	Yes	No	0.01	0.01	0.081	False Negative
	Bromoxynil	SRM14-1276	Yes	No	0.01	0.01	0.058	False Negative
	DDAC-C10	SRM14-1092	Yes	No	0.03	0.03	0.178	False Negative
		SRM14-1406 (3rd)	Yes	No	0.03			False Negative
	Fenpropimorph carboxylic acid (BF-421-2)	SRM14-1340	Yes	No	0.01	0.01	0.089	False Negative
	Flonicamid Met. TFNA-AM	SRM14-956	Yes	No	0.01	0.01	0.072	False Negative
		SRM14-1218	Yes	No	0.01			False Negative
		SRM14-1302	Yes	No	0.01			False Negative
		SRM14-1342	Yes	No	0.01			False Negative
		SRM14-1368	Yes	No	0.01			False Negative
	Fluopyram-benzamide (M25)	SRM14-1400	Yes	No	0.02	0.02	0.101	False Negative
	AMPA	SRM14-1092	Yes	No	0.1	0.1	0.751	False Negative
	Haloxyfop	SRM14-1324	Yes	No	0.5	0.01	0.036	* False Negative
	MCPA	SRM14-1406 (3rd)	Yes	No	0.01	0.01	0.046	False Negative

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

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

EU and EFTA laboratories						
	Compound	No. of results [1)]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1)] No. (%)	FNs No.
	Glyphosate	43	38 (88 %)	2 (5 %)	3 (7 %)	0
Optional Compounds	2,4-DB	37	33 (89 %)	2 (5 %)	2 (5 %)	2
	Avermectin B1a	40	36 (90 %)	2 (5 %)	2 (5 %)	1
	Bixafen desmethyl	20	18 (90 %)	1 (5 %)	1 (5 %)	0
	Boscalid Met. M510F017	18	17 (94 %)	0 (0 %)	1 (6 %)	1
	Bromoxynil	35	33 (94 %)	1 (3 %)	1 (3 %)	1
	DDAC-C10	31	27 (87 %)	1 (3 %)	3 (10 %)	1
	Fenpropimorph carboxylic acid (BF-421-2)	11	10 (91 %)	0 (0 %)	1 (9 %)	1
	Flonicamid Met. TFNA-AM	24	17 (71 %)	2 (8 %)	5 (21 %)	5
	Fluopyram-benzamide (M25)	23	22 (96 %)	0 (0 %)	1 (4 %)	1
	MPP	20	17 (85 %)	1 (5 %)	2 (10 %)	0
	AMPA	29	26 (90 %)	2 (7 %)	1 (3 %)	1
	N-Acetyl-glyphosate	16	13 (81 %)	0 (0 %)	3 (19 %)	0
	Haloxyfop	42	39 (93 %)	3 (7 %)	0 (0 %)	1
	MCPA	45	40 (89 %)	5 (11 %)	0 (0 %)	0
	Mepiquat	46	45 (98 %)	0 (0 %)	1 (2 %)	0
Overall EU/EFTA (Average)		**480**	**431 (90 %)**	**22 (5 %)**	**27 (6 %)**	**15**
3rd country laboratories						
	Compound	No. of results [1)]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1)] No. (%)	FNs No.
	Glyphosate	2	1 (50 %)	(0 %)	1 (50 %)	0
Optional Compounds	2,4-DB	1	1 (100 %)	(0 %)	(0 %)	0
	Avermectin B1a	2	1 (50 %)	1 (50 %)	(0 %)	0
	Bromoxynil	2	1 (50 %)	(0 %)	1 (50 %)	0
	DDAC-C10	1	(0 %)	(0 %)	(0 %)	1
	Flonicamid Met. TFNA-AM	2	1 (50 %)	(0 %)	1 (50 %)	0
	Fluopyram-benzamide (M25)	1	(0 %)	(0 %)	1 (100 %)	0
	AMPA	1	(0 %)	(0 %)	1 (100 %)	0
	Haloxyfop	2	1 (50 %)	(0 %)	1 (50 %)	0
	MCPA	3	2 (67 %)	(0 %)	(0 %)	1
	Mepiquat	1	1 (100 %)	(0 %)	(0 %)	0
Overall 3rd country (Average)		**18**	**9 (50 %)**	**3 (17 %)**	**6 (33 %)**	**6**

1) including false negatives (FNs)

of the homogeneity test as described in **Section 4.3 (p. 28)**. **Table 4-7 (p. 32)** shows the overall classification of z-scores achieved by all laboratories for compulsory and optional compounds. The respective rules are shown in **Section 2.4 (p. 14)**. Excluding ***TFNA-AM,*** among the laboratories from EU and EFTA countries "acceptable" z-scores were achieved by 81 % (***N-acetyl-glyphosate***) to 98 % (***mepiquat***) of the labs for the compounds being analysed. Overall, 90 % of the results submitted by EU- and EFTA-countries were acceptable, 5 % questionable and 6 % unacceptable (including false negatives). Among the 18 results reported by the three non-EU/EFTA laboratories 50 % were allocated with acceptable, 17 % with questionable, and 33 % with unacceptable z-scores. The latter group included 2 false negative results.

A compilation of all individual results and z-scores for each laboratory is shown in **Table 4-8 (p. 34)** and **Table 4-9 (p. 38)**. 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**.

4.4.4 Laboratory Classification Based on Scope

Since there was only one compulsory compound within this PT and the matrix "bovine liver" was out of the routine analytical scope of most participating laboratories, no classification of the participating laboratories into Category A and B based on the analytical scope was undertaken in this PT.

4.4.5 Feedback from Laboratories in Case of Poor Results

In the current PT, in total 480 results for the analytes present in the test items and 2 false positive results were reported by 57 participants from EU/EFTA countries. 31 EU/EFTA laboratories reported 51 results indicating poor performance; thereof 49 results with $|z| > 2$ (incl. 17 FNs) and 2 FPs. 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 these 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.

28 out of the 31 laboratories with poor performance gave feedback as regards the possible reasons for poor performance and in some cases about supplementary experiments undertaken to localize the errors. Feedback was therefore provided for 46 out of the 51 cases of poor performance. In many cases no clear reasons for the poor performance could be identified. The most frequent reasons reported were the lack of experience with the matrix (liver) or the analyte-matix combination (22 cases). Other frequent reasons of poor performance were: "absence of a validated method" (13 cases), the "improper compensation of matrix effects" (11 cases), "transcription error" (8 cases), "improper or non-correction of results for recovery" (7 cases) and "improper / unsuitable method" (6 cases). Among the 17 cases of FNs reported by EU/EFTA labs the main reason reported was the "analysis of the wrong analyte" (5 cases). Further reasons reported were "transcription errors" (3 cases), "lack of a validated method" (2 cases) and a "too high RL" (1 case).

Table 4-8: Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compound glyphosate and 7 OPTIONAL compounds

		Compound	Glyphosate		2,4-DB		Avermectin B1a		Bixafen desmethyl	
		MRRL [mg/kg]	0.1		0.01		0.01		0.01	
		Assigned Value [mg/kg]	0.535		0.061		0.058		0.05	
		*CV**	23.3 %		19.7 %		29.2 %		20.0 %	
Lab code SRM14-	NRL	Analysed / corr. found, max. 32 / 16	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 %)
950		30 / 15	0.548	0.1	0.0813	1.3	0.0593	0.1	0.0495	-0.1
956	SRM/A0	25 / 11	0.428	-0.8	FN	-3.3			0.0560	0.5
992		31 / 16	0.570	0.3	0.0880	1.7	0.0700	0.9	0.0510	0.1
1022		3 / 1							0.034	-1.3
1024		2 / 1					0.07	0.9		
1072	SRM/A0	18 / 8	0.465	-0.5	0.065	0.2	0.049	-0.6		
1090	SRM/A0	32 / 16	0.527	-0.1	0.066	0.3	0.030	-1.9	0.055	0.4
1092	SRM	22 / 8	0.688	1.1	0.0358	-1.7	0.0419	-1.1		
1150		4 / 4			0.027	-2.2				
1206		32 / 16	0.511	-0.2	0.068	0.4	0.058	0.0	0.046	-0.3
1214		24 / 10	0.47	-0.5	0.065	0.2	0.064	0.4		
1218		22 / 9	0.570	0.3	0.052	-0.6	0.046	-0.8		
1224		7 / 4	0.620	0.6			0.026	-2.2		
1228		16 / 7	0.416	-0.9						
1240		11 / 5			0.0560	-0.3	0.0354	-1.5		
1244	SRM/A0	23 / 11	0.353	-1.4	0.0437	-1.2	0.0829	1.8		
1248	A0	1 / 1	0.572	0.3						
1250	SRM/A0	9 / 5	0.498	-0.3			0.0565	-0.1		
1266		31 / 16	1.104	4.3	0.064	0.2	0.061	0.2	0.055	0.4
1270		19 / 8	0.928	2.9	0.0621	0.0	0.0456	-0.8		
1274	SRM/A0	18 / 6	0.550	0.1			0.0703	0.9		
1276		24 / 10	0.431	-0.8	0.0653	0.3	0.0832	1.8		
1278	SRM/A0	12 / 6	0.551	0.1			0.059	0.1		
1290	SRM/A0	11 / 3			FN	-3.3				
1292		26 / 12	0.854	2.4	0.0479	-0.9			0.0543	0.3
1298		16 / 6			0.0756	0.9	0.0644	0.5		
1300	SRM/A0	19 / 9	0.529	0.0	0.064	0.2	0.079	1.5		
1302	SRM/A0	13 / 7			0.101	2.6			0.080	2.4
1306		11 / 5	2.33	13.4						
1310		18 / 7	0.321	-1.6	0.046	-1.0	0.098	2.8		
1312	SRM/A0	29 / 14	0.770	1.8	0.061	0.0	0.126	4.8	0.052	0.1
1318	SRM/A0	16 / 7	0.558	0.2			FN	-3.3		
1320		32 / 16	0.483	-0.4	0.064	0.2	0.056	-0.1	0.043	-0.6
1322		18 / 6			0.0588	-0.2	0.0551	-0.2		
1324		21 / 8	0.806	2.0			0.058	0.0		
1330	SRM/A0	20 / 9	0.654	0.9	0.0721	0.7	0.0675	0.7		
1332		24 / 11	0.403	-1.0	0.0600	-0.1	0.0558	-0.1	0.0512	0.1
1336	SRM/A0	3 / 2	0.619	0.6						
1338	SRM/A0	27 / 14	0.463	-0.5	0.0750	0.9			0.132	6.5
1340	SRM/A0	15 / 6							0.030	-1.6
1342	SRM/A0	17 / 6	0.683	1.1	0.057	-0.3	0.071	0.9		
1346		1 / 1					0.0382	-1.3		

* based on 10 numerical results from participants and the mean value of the homogeneity test

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compound glyphosate and 7 OPTIONAL compounds

		Compound	Boscalid Met. M510F017		Bromoxynil		DDAC-C10		Fenpropimorph carboxylic acid (BF-421-2)	
		MRRL [mg/kg]	0.01		0.01		0.03		0.01	
		Assigned Value [mg/kg]	0.081		0.059		0.177		0.088*	
		*CV**	13.1 %		15.3 %		19.8 %		11.4 %	
Lab code SRM14-	NRL	Analysed / corr. found, max. 32 / 16	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 %)
950		30 / 15	0.0754	-0.3	0.0864	1.9	0.175	0.0		
956	SRM/A0	25 / 11	0.0902	0.5	0.0619	0.2				
992		31 / 16	0.0760	-0.2	0.0620	0.2	0.162	-0.3	0.0810	-0.3
1022		3 / 1	FN	-3.5						
1024		2 / 1								
1072	SRM/A0	18 / 8			0.060	0.1	0.216	0.9		
1090	SRM/A0	32 / 16	0.088	0.4	0.063	0.3	0.209	0.7	0.104	0.7
1092	SRM	22 / 8					FN	-3.3		
1150		4 / 4					0.021	-3.5		
1206		32 / 16	0.072	-0.4	0.058	0.0	0.182	0.1	0.081	-0.3
1214		24 / 10			0.056	-0.2	144	3246.8		
1218		22 / 9			0.064	0.4	0.175	0.0		
1224		7 / 4								
1228		16 / 7								
1240		11 / 5								
1244	SRM/A0	23 / 11			0.0728	1.0				
1248	A0	1 / 1								
1250	SRM/A0	9 / 5								
1266		31 / 16	0.084	0.2	0.065	0.4	0.174	-0.1	0.090	0.1
1270		19 / 8			0.0479	-0.7				
1274	SRM/A0	18 / 6					0.139	-0.9		
1276		24 / 10			FN	-3.3	0.221	1.0		
1278	SRM/A0	12 / 6								
1290	SRM/A0	11 / 3								
1292		26 / 12	0.0810	0.0	0.0438	-1.0	0.248	1.6		
1298		16 / 6			0.0580	0.0				
1300	SRM/A0	19 / 9			0.059	0.0	0.174	-0.1		
1302	SRM/A0	13 / 7	0.085	0.2			0.207	0.7		
1306		11 / 5			0.0537	-0.3	0.0864	-2.0		
1310		18 / 7			0.057	-0.1				
1312	SRM/A0	29 / 14	0.075	-0.3	0.056	-0.2	0.159	-0.4	0.079	-0.4
1318	SRM/A0	16 / 7			0.056	-0.2	0.202	0.6		
1320		32 / 16	0.060	-1.0	0.042	-1.1	0.190	0.3	0.082	-0.3
1322		18 / 6			0.0625	0.3				
1324		21 / 8			0.060	0.1				
1330	SRM/A0	20 / 9			0.0453	-0.9	0.204	0.6		
1332		24 / 11			0.0662	0.5	0.199	0.5		
1336	SRM/A0	3 / 2								
1338	SRM/A0	27 / 14	0.0890	0.4	0.0900	2.2			0.112	1.1
1340	SRM/A0	15 / 6					0.144	-0.7	FN	-3.5
1342	SRM/A0	17 / 6								
1346		1 / 1								

* based on 10 numerical results from participants and the mean value of the homogeneity test

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compound glyphosate and 7 OPTIONAL compounds

Compound			Glyphosate		2,4-DB		Avermectin B1a		Bixafen desmethyl	
MRRL [mg/kg]			0.1		0.01		0.01		0.01	
Assigned Value [mg/kg]			0.535		0.061		0.058		0.05	
*CV**			23.3 %		19.7 %		29.2 %		20.0 %	
Lab code SRM14-	NRL	Analysed / corr. found, max. 32 / 16	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 %)
1350		7 / 3	0.60	0.5						
1352	SRM/A0	19 / 8	0.513	-0.2	0.0485	-0.8	0.0483	-0.6		
1354	SRM/A0	23 / 9	0.0618	-3.5	0.0524	-0.6	0.0519	-0.4		
1356		22 / 8	0.305	-1.7						
1358		27 / 13	0.508	-0.2	0.0562	-0.3	0.0428	-1.0	0.0309	-1.5
1364		6 / 4							0.0443	-0.5
1366		9 / 5	0.57	0.3						
1368	SRM/A0	26 / 11	0.562	0.2	0.0691	0.5	0.0596	0.1		
1370	SRM/A0	4 / 1					0.036	-1.5		
1392	SRM/A0	14 / 5			0.0526	-0.6	0.0553	-0.2		
1394		29 / 14	0.520	-0.1	0.067	0.4	0.072	1.0	0.054	0.3
1396		2 / 1								
1398	A0	24 / 13	0.399	-1.0	0.065	0.2	0.066	0.6	0.069	1.5
1400		27 / 12	0.502	-0.2	0.050	-0.7	0.032	-1.8	0.050	0.0
1402	SRM	32 / 16	0.466	-0.5	0.073	0.8	0.053	-0.3	0.058	0.6
978		6 / 3	0.442	-0.7			0.0520	-0.4		
1404		12 / 4								
1406		22 / 9	1.8	9.5	0.05	-0.7	0.026	-2.2		

* based on 10 numerical results from participants and the mean value of the homogeneity test

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compound glyphosate and 7 OPTIONAL compounds

	Compound		Boscalid Met. M510F017		Bromoxynil		DDAC-C10		Fenpropimorph carboxylic acid (BF-421-2)	
	MRRL [mg/kg]		0.01		0.01		0.03		0.01	
	Assigned Value [mg/kg]		0.081		0.059		0.177		0.088*	
	*CV**		13.1 %		15.3 %		19.8 %		11.4 %	
Lab code SRM14-	NRL	Analysed / corr. found, max. 32 / 16	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 %)
1350		7 / 3								
1352	SRM/A0	19 / 8			0.0396	-1.3				
1354	SRM/A0	23 / 9			0.0546	-0.3				
1356		22 / 8			0.0580	0.0	0.0969	-1.8		
1358		27 / 13	0.0460	-1.7	0.0608	0.2	0.151	-0.6		
1364		6 / 4	0.0849	0.2			0.178	0.0		
1366		9 / 5					0.17	-0.2		
1368	SRM/A0	26 / 11			0.0614	0.2	0.178	0.0		
1370	SRM/A0	4 / 1								
1392	SRM/A0	14 / 5			0.0436	-1.0				
1394		29 / 14	0.096	0.8	0.065	0.4	0.156	-0.5	0.095	0.3
1396		2 / 1								
1398	A0	24 / 13	0.094	0.7	0.064	0.4	0.272	2.1	0.089	0.0
1400		27 / 12	0.079	-0.1	0.050	-0.6	0.146	-0.7		
1402	SRM	32 / 16	0.073	-0.4	0.068	0.6	0.194	0.4	0.087	-0.1
978		6 / 3								
1404		12 / 4			0.0596	0.1				
1406		22 / 9			0.015	-3.0	FN	-3.3		

* based on 10 numerical results from participants and the mean value of the homogeneity test

Table 4-9: Results reported and z-scores achieved by all participating laboratories for 8 OPTIONAL compounds

		Compound	Flonicamid Met. TFNA-AM		Fluopyram-benza-mide (M25)		MPP		AMPA	
		MRRL [mg/kg]	0.01		0.02		0.05		0.1	
		Assigned Value [mg/kg]	0.073		0.101		0.309		0.754	
		*CV**	21.8 %		12.4 %		20.5 %		19.0 %	
Lab code SRM14-	NRL	Analysed / corr. found, max. 32 / 16	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 %)
950		30 / 15	0.0498	-1.3	0.105	0.2	0.314	0.1	0.569	-1.0
956	SRM/A0	25 / 11	FN	-3.5	0.0835	-0.7	0.231	-1.0	0.855	0.5
992		31 / 16	0.0820	0.5	0.104	0.1	0.320	0.1	0.820	0.4
1022		3 / 1								
1024		2 / 1								
1072	SRM/A0	18 / 8								
1090	SRM/A0	32 / 16	0.081	0.5	0.102	0.0	0.302	-0.1	0.707	-0.2
1092	SRM	22 / 8					0.651	4.4	FN	-3.5
1150		4 / 4								
1206		32 / 16	0.069	-0.2	0.100	0.0	0.303	-0.1	0.705	-0.3
1214		24 / 10					0.27	-0.5	0.69	-0.3
1218		22 / 9	FN	-3.5					0.795	0.2
1224		7 / 4							1.200	2.4
1228		16 / 7	0.074	0.1			0.313	0.1	0.303	-2.4
1240		11 / 5	0.0342	-2.1						
1244	SRM/A0	23 / 11			0.101	0.0	0.350	0.5	0.791	0.2
1248	A0	1 / 1								
1250	SRM/A0	9 / 5			0.0919	-0.4				
1266		31 / 16	0.079	0.3	0.108	0.3	0.291	-0.2	0.679	-0.4
1270		19 / 8			0.0925	-0.3				
1274	SRM/A0	18 / 6								
1276		24 / 10					0.229	-1.0	0.781	0.1
1278	SRM/A0	12 / 6							0.857	0.5
1290	SRM/A0	11 / 3								
1292		26 / 12	0.0716	-0.1	0.102	0.0			1.00	1.3
1298		16 / 6								
1300	SRM/A0	19 / 9							0.730	-0.1
1302	SRM/A0	13 / 7	FN	-3.5	0.099	-0.1				
1306		11 / 5								
1310		18 / 7								
1312	SRM/A0	29 / 14	0.071	-0.1	0.094	-0.3	0.680	4.8		
1318	SRM/A0	16 / 7							0.790	0.2
1320		32 / 16	0.096	1.3	0.120	0.8	0.309	0.0	0.532	-1.2
1322		18 / 6	0.0576	-0.8						
1324		21 / 8					0.288	-0.3	0.898	0.8
1330	SRM/A0	20 / 9					0.268	-0.5		
1332		24 / 11	0.0690	-0.2	0.0953	-0.2				
1336	SRM/A0	3 / 2							0.58	-0.9
1338	SRM/A0	27 / 14	0.0825	0.5	0.116	0.6	0.376	0.9	0.949	1.0
1340	SRM/A0	15 / 6			0.101	0.0				
1342	SRM/A0	17 / 6	FN	-3.5						
1346		1 / 1								
1350		7 / 3							0.84	0.5
1352	SRM/A0	19 / 8								

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for 8 OPTIONAL compounds

	Compound			N-Acetyl-glyphosate		Haloxyfop		MCPA		Mepiquat	
	MRRL [mg/kg]			0.1		0.01		0.01		0.01	
	Assigned Value [mg/kg]			0.543		0.037		0.046		0.051	
	*CV**			27.6 %		21.4 %		25.3 %		16.9 %	
	Lab code SRM14-	NRL	Analysed / corr. found, max. 32 / 16	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 %)
	950		30 / 15	0.354	-1.6	0.0454	1.0	0.0513	0.5	0.0455	-0.4
	956	SRM/A0	25 / 11	0.0438	-3.7	0.0382	0.2	0.0435	-0.2	0.0334	-1.4
	992		31 / 16	0.460	-0.8	0.0410	0.5	0.0470	0.1	0.0500	-0.1
	1022		3 / 1								
	1024		2 / 1								
	1072	SRM/A0	18 / 8			0.039	0.3	0.052	0.5	0.075	1.9
	1090	SRM/A0	32 / 16	0.693	0.8	0.044	0.8	0.049	0.3	0.053	0.2
	1092	SRM	22 / 8	0.874	2.0	0.0120	-2.7	0.0367	-0.8	0.0480	-0.2
	1150		4 / 4					0.032	-1.2	0.055	0.3
	1206		32 / 16	0.625	0.3	0.038	0.2	0.050	0.3	0.050	-0.1
	1214		24 / 10			0.046	1.0	0.065	1.6	0.052	0.1
	1218		22 / 9			0.040	0.4	0.049	0.3	0.055	0.3
	1224		7 / 4							0.052	0.1
	1228		16 / 7			0.057	2.2	0.078	2.8	0.054	0.2
	1240		11 / 5			0.0392	0.3	0.0377	-0.7		
	1244	SRM/A0	23 / 11	0.517	-0.4	0.0318	-0.5	0.0430	-0.3	0.0427	-0.6
	1248	A0	1 / 1								
	1250	SRM/A0	9 / 5					0.0317	-1.2	0.0588	0.6
	1266		31 / 16	0.665	0.6	0.042	0.6	0.056	0.9	0.042	-0.7
	1270		19 / 8			0.036	-0.1	0.0454	-0.1	0.042	-0.7
	1274	SRM/A0	18 / 6			0.0342	-0.3	0.0356	-0.9	0.0543	0.3
	1276		24 / 10	0.354	-1.6	0.0391	0.3	0.0467	0.1	0.0726	1.7
	1278	SRM/A0	12 / 6			0.038	0.2	0.050	0.3	0.049	-0.2
	1290	SRM/A0	11 / 3			0.02	-1.8	0.02	-2.3	0.05	-0.1
	1292		26 / 12			0.0321	-0.5	0.0286	-1.5	0.0631	1.0
	1298		16 / 6			0.0384	0.2	0.0467	0.1	0.055	0.3
	1300	SRM/A0	19 / 9			0.048	1.2	0.071	2.2	0.048	-0.2
	1302	SRM/A0	13 / 7					0.078	2.8	0.052	0.1
	1306		11 / 5			0.0238	-1.4			0.0479	-0.2
	1310		18 / 7			0.024	-1.4	0.038	-0.7	0.033	-1.4
	1312	SRM/A0	29 / 14			0.042	0.6	0.047	0.1	0.141	7.1
	1318	SRM/A0	16 / 7			0.038	0.2	0.053	0.6	0.032	-1.5
	1320		32 / 16	1.889	9.1	0.032	-0.5	0.039	-0.6	0.032	-1.5
	1322		18 / 6			0.0423	0.6	0.0460	0.0		
	1324		21 / 8			FN*	-2.9	0.054	0.7	0.051	0.0
	1330	SRM/A0	20 / 9			0.0387	0.2	0.0395	-0.6	0.0504	0.0
	1332		24 / 11			0.0390	0.3	0.0534	0.6	0.0421	-0.7
	1336	SRM/A0	3 / 2								
	1338	SRM/A0	27 / 14	0.487	-0.6	0.0356	-0.1	0.0625	1.4	0.0472	-0.3
	1340	SRM/A0	15 / 6			0.022	-1.6	0.031	-1.3	0.042	-0.7
	1342	SRM/A0	17 / 6			0.044	0.8	0.059	1.1	0.076	2.0
	1346		1 / 1								
	1350		7 / 3							0.058	0.6
	1352	SRM/A0	19 / 8	0.496	-0.6	0.0309	-0.6	0.0361	-0.9	0.0542	0.3

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for 8 OPTIONAL compounds

Compound			Flonicamid Met. TFNA-AM		Fluopyram-benza-mide (M25)		MPP		AMPA		
MRRL [mg/kg]			0.01		0.02		0.05		0.1		
Assigned Value [mg/kg]			0.073		0.101		0.309		0.754		
*CV**			21.8 %		12.4 %		20.5 %		19.0 %		
Lab code SRM14-	NRL	Analysed / corr. found, max. 32 / 16	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 %)	
1354	SRM/A0	23 / 9	0.0666	-0.3	0.0766	-1.0					
1356		22 / 8	0.0340	-2.1					0.659	-0.5	
1358		27 / 13			0.0532	-1.9			0.599	-0.8	
1364		6 / 4			0.0934	-0.3					
1366		9 / 5					0.32	0.1	0.87	0.6	
1368	SRM/A0	26 / 11	FN	-3.5			0.237	-0.9	0.833	0.4	
1370	SRM/A0	4 / 1									
1392	SRM/A0	14 / 5									
1394		29 / 14	0.094	1.2	0.114	0.5			0.637	-0.6	
1396		2 / 1									
1398	A0	24 / 13	0.087	0.8	0.114	0.5	0.490	2.3			
1400		27 / 12	0.072	0.0	FN	-3.2			0.753	0.0	
1402	SRM	32 / 16	0.082	0.5	0.117	0.6	0.245	-0.8	0.697	-0.3	
978		6 / 3									
1404		12 / 4	0.0778	0.3							
1406		22 / 9	0.197	6.8	0.291	7.6			3.1	12.5	

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for 8 OPTIONAL compounds

	Compound		N-Acetyl-glyphosate		Haloxyfop		MCPA		Mepiquat	
	MRRL [mg/kg]		0.1		0.01		0.01		0.01	
	Assigned Value [mg/kg]		0.543		0.037		0.046		0.051	
	*CV**		27.6 %		21.4 %		25.3 %		16.9 %	
Lab code SRM14-	NRL	Analysed / corr. found, max. 32 / 16	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 %)
1354	SRM/A0	23 / 9			0.0288	-0.9	0.0405	-0.5	0.0633	1.0
1356		22 / 8			0.0257	-1.2	0.013	-2.9	0.0505	0.0
1358		27 / 13	2.01	9.9	0.0212	-1.7	0.0487	0.2	0.0560	0.4
1364		6 / 4								
1366		9 / 5	0.46	-0.8						
1368	SRM/A0	26 / 11	0.491	-0.6	0.0418	0.6	0.0484	0.2	0.0586	0.6
1370	SRM/A0	4 / 1								
1392	SRM/A0	14 / 5			0.0322	-0.5	0.0416	-0.4		
1394		29 / 14			0.040	0.4	0.054	0.7	0.052	0.1
1396		2 / 1							0.052	0.1
1398	A0	24 / 13					0.056	0.9	0.046	-0.4
1400		27 / 12			0.039	0.3	0.030	-1.4	0.033	-1.4
1402	SRM	32 / 16	0.689	0.8	0.042	0.6	0.053	0.6	0.056	0.4
978		6 / 3					0.0477	0.1		
1404		12 / 4			0.0385	0.2	0.0499	0.3		
1406		22 / 9			0.0055	-3.4	FN	-3.1	0.045	-0.5

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6. REFERENCES

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7. APPENDICES

Appendix 1 List of Laboratories Registered to Participate in the EUPT-SRM14

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

Country (Location)	Analysed on behalf of	Institution	City	NRL
Austria	AT	AGES Innsbruck - Institute for Food Safety	Innsbruck	SRM/AO
Belgium	BE; LU	CER Groupe	Marloie	–
Belgium	BE	LFSAL - Liège	Wandre	–
Belgium	BE	Sciensano	Brussels	SRM/AO
Croatia	HR	Bioinstitut d.o.o., Cakovec	Cakovec	–
Cyprus	CY	SGL (Nicosia)	Nicosia	SRM/AO
Czech Republic	CZ	Czech Agriculture and Food Inspection Authority	Prague	SRM
Czech Republic	CZ	SVI Prague	Prague	AO
Czech Republic	CZ	VSCHT (Praha)	Prague	–
Estonia	EE	Tarfu Laboratory of Health Board	Tartu	SRM/AO
Finland	FI	Finnish Food Authority	Helsinki	SRM/AO
France	FR	Anses - LSAl - PBM	MAISONS-ALFORT Cedex	SRM/AO
France	FR	CERECO (GARONS)	GARONS	–
France	FR	INOVALYS Le Mans	Le Mans	–
France	BE	Phytocontrol (Nimes)	Nimes	–
Germany	DE	NRL Pestizide	Berlin	SRM/AO
Germany	DE	Chemisches- und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe	Münster	–
Germany	DE	Eurofins SOFIA GmbH	Berlin	–
Germany	DE	Institut für Hygiene und Umwelt	Hamburg	–
Germany	DE	LALLF (Rostock)	Rostock	–
Germany	DE	Landesamt für Verbraucherschutz, Halle/Saale	Halle/Saale	–
Germany	DE	LGL Erlangen	Erlangen	–
Germany	DE	LUA Rheinland-Pfalz, Institut für LM-Chemie Speyer	Speyer	–
Germany	DE	State Investigation Institute of Health and Veterinary Saxony	Dresden	–
Germany	LT	GALAB Laboratories GmbH - Hamburg	Hamburg	–
Germany	MT	Eurofins Dr. Specht Laboratorien GmbH	Hamburg	–
Germany	BE	LUFA Kiel	Kiel	–
Greece	GR	Benaki Phytopathological Institute, Kifissia	Kifissia	SRM/AO
Greece	GR	GCSL (Athens)	Athens	SRM/AO
Hungary	HU	National Food Chain Safety Office Food Chain Safety Laboratory Directorate Pesticide Analytical National Reference Laboratory, Velence	Velence	SRM/AO
Hungary	HU	Food Chain Safety Centre Non-profit Ltd. Pesticide Residue Analytical Laboratory, Hódmezővásárhely	Hódmezovásárhely	–
Hungary	HU	FCSCN Ltd., Pesticide Residue Analytical Laboratory, Miskolc	Miskolc	–
Hungary	HU	Food Chain Safety Centre Non-profit Ltd., Pesticide Residue Analytical Laboratory, Szolnok	Szolnok	–
Ireland	IE	Pesticide Control Laboratory	Co. Kildare	SRM/AO
Italy	IT	APPA Bolzano	Bolzano	–
Italy	IT	Italian national Insitute of health (Department of Environment and Health)	Roma	SRM/AO
Italy	IT	IZS Sardegna	Sassari	–
Italy	IT	Istituto Zooprofilattico Sperimentale Abruzzo e Molise	Teramo	–

* 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
Italy	IT	IZSLER	Brescia	–
Latvia	LV	BIOR (Riga)	Riga	SRM/AO
Lithuania	LT	NMVRVI (Vilnius)	Vilnius	SRM/AO
The Netherlands	NL	NVWA - NRL for Pesticide Residues in Food and Feed	Wageningen	SRM/AO
The Netherlands	NL	RIKILT Wageningen University & Research	Wageningen	AO
The Netherlands	BE	Groen Agro Control	Delfgauw	–
The Netherlands	BE	Handelslaboratorium Dr. Verwey	Rotterdam	–
Norway	NO	NIBIO, Pesticides and Natural Products Chemistry	ÅS	SRM/AO
Portugal	PT	Laboratório Regional de Veterinária e Segurança Alimentar	Funchal - Madeira Island	SRM
Romania	RO	Institute for Hygiene and Veterinary Public Health	Bucharest	SRM/AO
Slovakia	SK	State Veterinary and Food Institute (Bratislava)	Bratislava	SRM/AO
Slovenija	SI	National Laboratory of Health, Environment and Food-stuffs - Maribor, Pesticide Lab	Maribor	SRM/AO
Spain	ES	Analytica Alimentaria	Almeria	–
Spain	ES	Lab. Agrario Regional - Junta de Castilla y Leon	Burgos	–
Spain	ES	Laboratori Agència Salut Pública Barcelona	Barcelona	–
Spain	ES	National Center for Technology and Food Safety	San Adrián (Navarra)	–
Spain	ES	National Centre for Food (Majadahonda)	Majadahonda	SRM/AO
Sweden	SE	Eurofins Food & Feed Sweden AB	Lidköping	–
Sweden	SE	SNFA Science Department - Chemistry Division 1	Uppsala	SRM/AO
Switzerland	CH	Kantonales Labor Zurich	Zürich	–
United Kingdom	UK; MT	Fera Science Ltd	York	SRM/AO

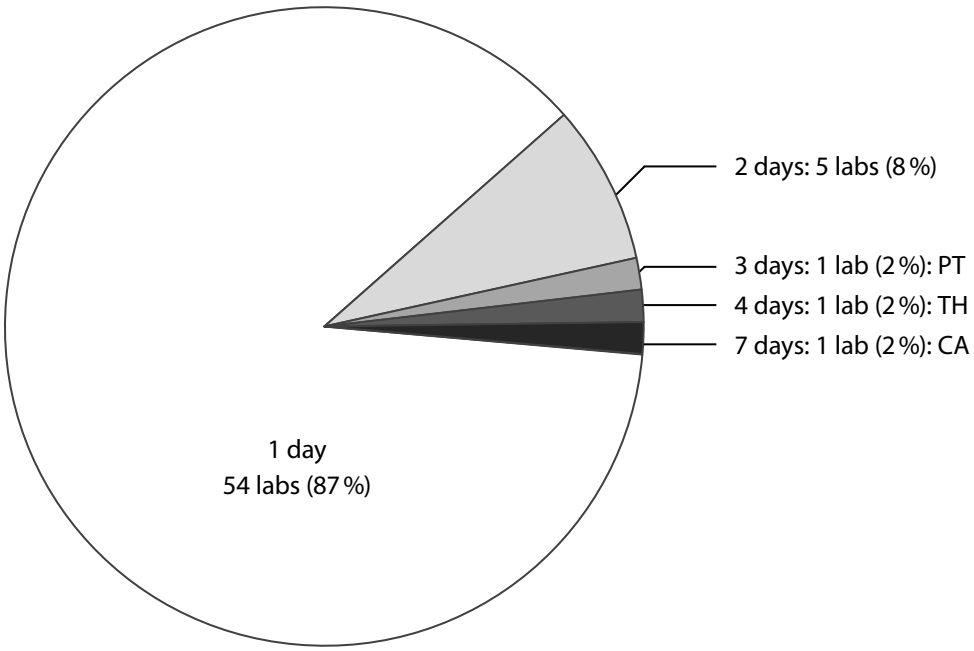
* only for EU-Member States

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

Country	Institution	City
Canada	ISURA	Burnaby
China	Government Laboratory (Hong Kong)	Hong Kong
Thailand	Central Laboratory (Bangkok)	Bangkok

Appendix 2 Shipment Evaluation

Compilation of shipment duration



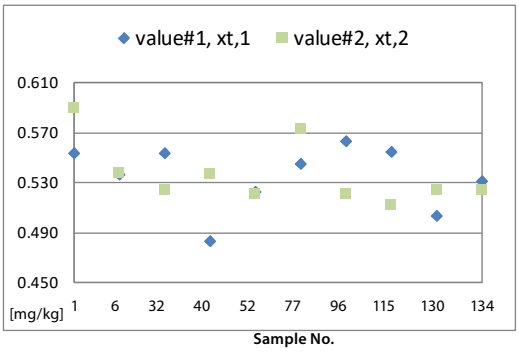
Appendix 3 Data of Homogeneity Test

Glyphosate			2,4-DB			Avermectin B1a			Bixafen desmethyl		
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	0.553	0.590	1	0.059	0.068	1	0.071	0.064	1	0.054	0.054
6	0.537	0.538	6	0.055	0.062	6	0.069	0.064	6	0.051	0.057
32	0.554	0.524	32	0.053	0.054	32	0.062	0.065	32	0.052	0.047
40	0.483	0.537	40	0.059	0.053	40	0.065	0.069	40	0.055	0.051
52	0.523	0.521	52	0.053	0.065	52	0.063	0.061	52	0.060	0.043
77	0.545	0.573	77	0.055	0.053	77	0.071	0.070	77	0.049	0.056
96	0.563	0.520	96	0.066	0.061	96	0.063	0.064	96	0.052	0.052
115	0.555	0.512	115	0.058	0.058	115	0.062	0.062	115	0.044	0.054
130	0.503	0.524	130	0.057	0.062	130	0.065	0.062	130	0.055	0.044
134	0.531	0.524	134	0.064	0.051	134	0.060	0.062	134	0.054	0.063
mean / AV*	0.536 / 0.535		mean / AV*	0.058 / 0.061		mean / AV*	0.065 / 0.058		mean / AV*	0.052 / 0.050	

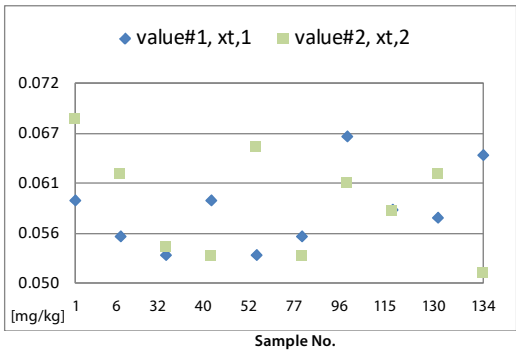
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Grafical presentation of the results:

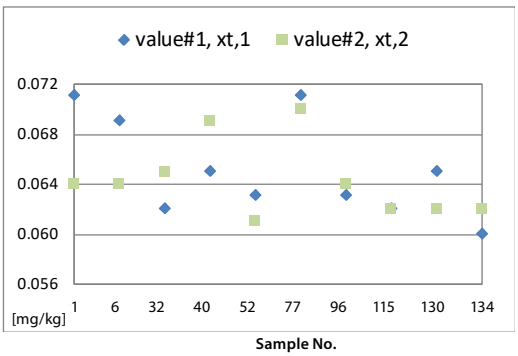
Glyphosate



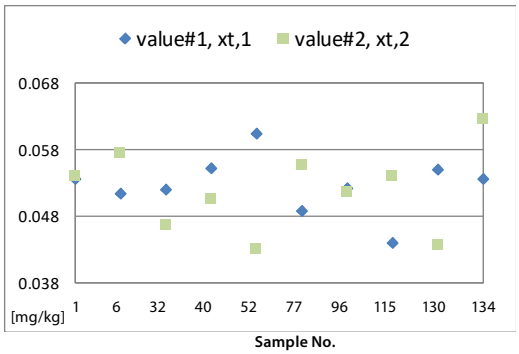
2,4-DB



Avermectin B1a



Bixafen desmethyl

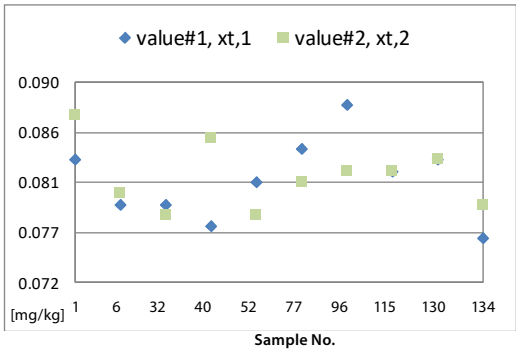


Appendix 3 (cont.): Data of Homogeneity Test

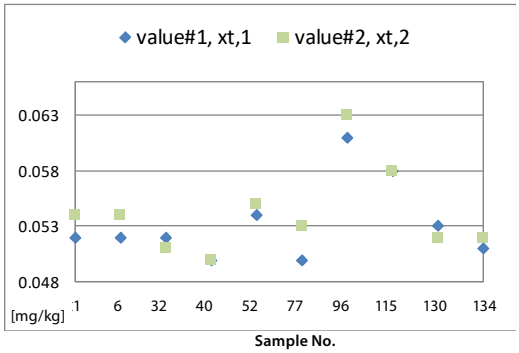
Boscalid Met. M510F01			Bromoxynil			DDAC-C10-Cl			Fenpropimorph carboxylic acid (BF-421-2)		
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]
1	0.083	0.087	1	0.052	0.054	1	0.199	0.198	1	0.084	0.091
6	0.079	0.080	6	0.052	0.054	6	0.194	0.178	6	0.077	0.081
32	0.079	0.078	32	0.052	0.051	32	0.198	0.185	32	0.078	0.081
40	0.077	0.085	40	0.050	0.050	40	0.182	0.167	40	0.076	0.081
52	0.081	0.078	52	0.054	0.055	52	0.163	0.184	52	0.086	0.078
77	0.084	0.081	77	0.050	0.053	77	0.218	0.198	77	0.086	0.084
96	0.088	0.082	96	0.061	0.063	96	0.215	0.194	96	0.084	0.084
115	0.082	0.082	115	0.058	0.058	115	0.182	0.182	115	0.079	0.079
130	0.083	0.083	130	0.053	0.052	130	0.182	0.174	130	0.082	0.084
134	0.076	0.079	134	0.051	0.052	134	0.185	0.180	134	0.080	0.080
mean / AV*	0.081 / 0.081		mean / AV*	0.054 / 0.059		mean / AV*	0.188 / 0,177		mean / AV*	0.082 / 0.088	
* 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											

Grafical presentation of the results:

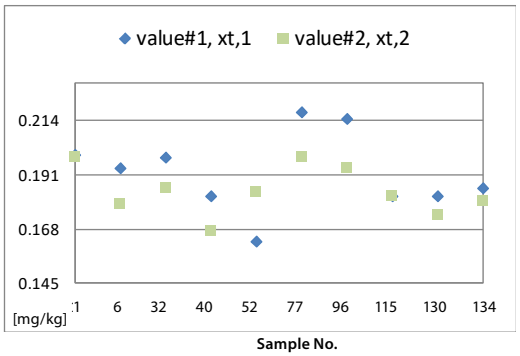
Boscalid Met. M510F01



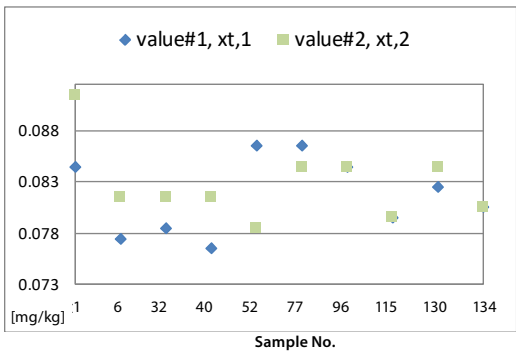
Bromoxynil



DDAC-C10-Cl



Fenpropimorph carb. acid (BF-421-2)



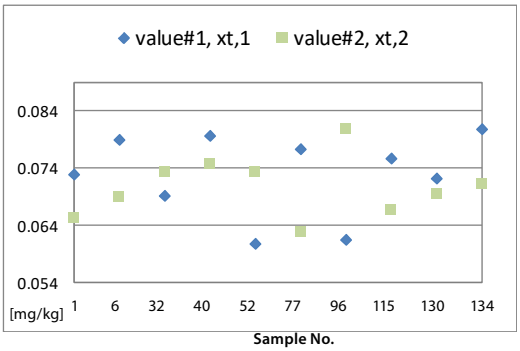
Appendix 3 (cont.): Data of Homogeneity Test

Flonicamid Met. TFNA-AM			Fluopyram-benzamide (M25)			MPP			AMPA		
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	0.073	0.065	1	0.097	0.090	1	0.309	0.333	1	0.763	0.823
6	0.079	0.069	6	0.107	0.100	6	0.283	0.298	6	0.790	0.753
32	0.069	0.073	32	0.099	0.101	32	0.287	0.267	32	0.771	0.748
40	0.080	0.075	40	0.103	0.096	40	0.281	0.274	40	0.728	0.722
52	0.061	0.073	52	0.099	0.103	52	0.287	0.284	52	0.708	0.707
77	0.077	0.063	77	0.103	0.094	77	0.296	0.334	77	0.730	0.774
96	0.061	0.081	96	0.099	0.112	96	0.314	0.286	96	0.738	0.778
115	0.076	0.067	115	0.096	0.093	115	0.289	0.310	115	0.698	0.771
130	0.072	0.069	130	0.094	0.099	130	0.292	0.287	130	0.737	0.728
134	0.081	0.071	134	0.104	0.099	134	0.278	0.281	134	0.730	0.779
mean / AV*	0.072 / 0.073		mean / AV*	0.099 / 0.101		mean / AV*	0.294 / 0.309		mean / AV*	0.749 / 0.754	

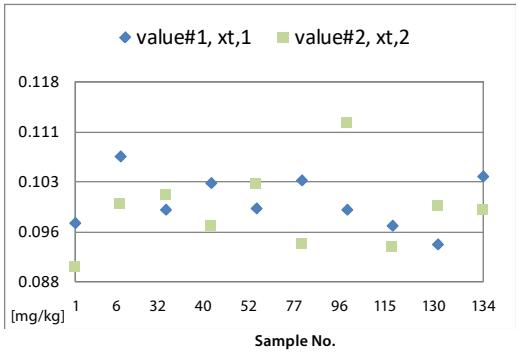
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Grafical presentation of the results:

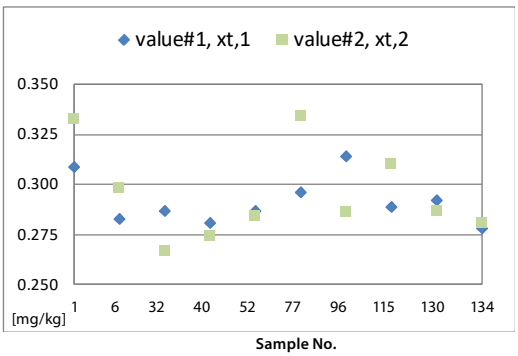
Flonicamid Met. TFNA-AM



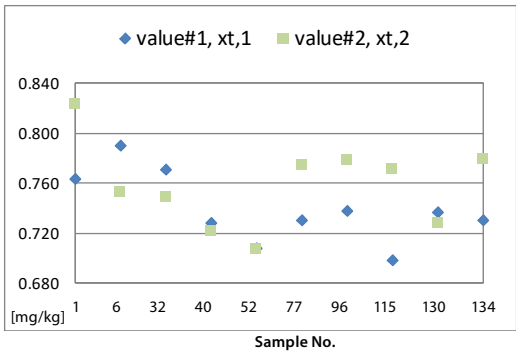
Fluopyram-benzamide (M25)



MPP



AMPA



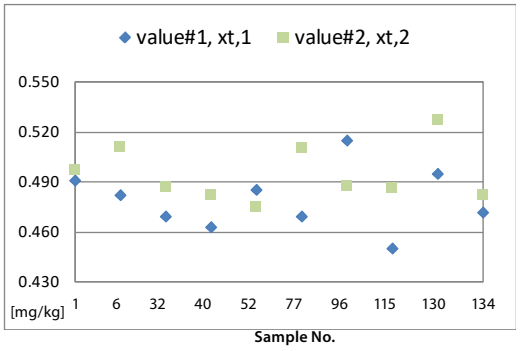
Appendix 3 (cont.): Data of Homogeneity Test

N-Acetyl-glyphosate			Haloxyfop			MCPA			Mepiquat-Cl		
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]
1	0.491	0.497	1	0.038	0.036	1	0.050	0.045	1	0.052	0.056
6	0.482	0.511	6	0.034	0.036	6	0.046	0.044	6	0.051	0.054
32	0.469	0.487	32	0.034	0.033	32	0.047	0.044	32	0.052	0.048
40	0.463	0.482	40	0.032	0.033	40	0.050	0.046	40	0.048	0.050
52	0.485	0.475	52	0.033	0.032	52	0.046	0.045	52	0.049	0.052
77	0.469	0.510	77	0.034	0.032	77	0.046	0.048	77	0.054	0.052
96	0.515	0.488	96	0.039	0.034	96	0.044	0.045	96	0.052	0.052
115	0.450	0.486	115	0.032	0.032	115	0.050	0.046	115	0.049	0.049
130	0.495	0.527	130	0.035	0.035	130	0.046	0.049	130	0.050	0.050
134	0.472	0.482	134	0.032	0.035	134	0.055	0.047	134	0.050	0.052
mean / AV*	0.478 / 0.543 ‡		mean / AV*	0.034 / 0.037		mean / AV*	0.047 / 0.046		mean / AV*	0.051 / 0.051	

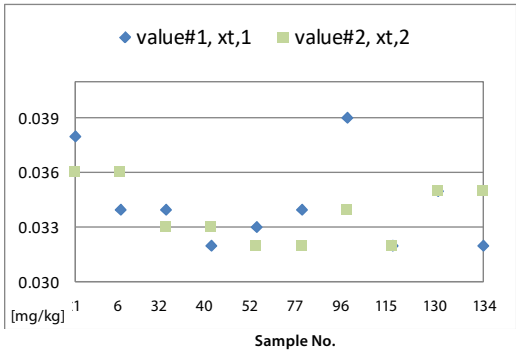
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories
‡ based on 10 nemerical results from the participating laboratories and the mean value of the homogeneity test

Grafical presentation of the results:

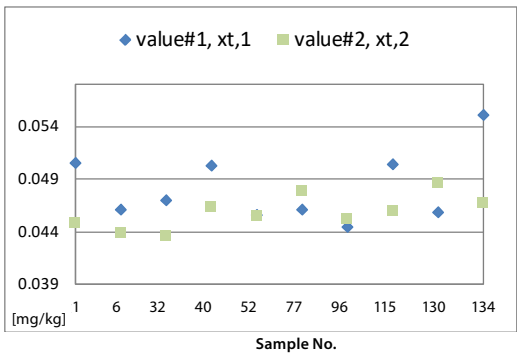
N-Acetyl-glyphosate



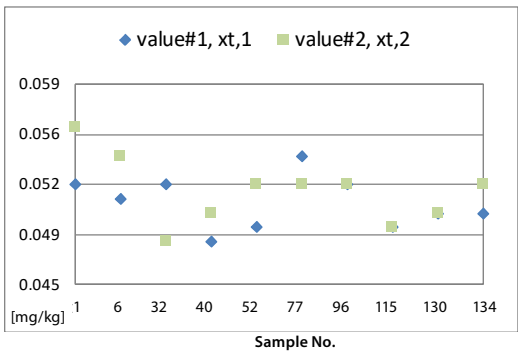
Haloxyfop



MCPA



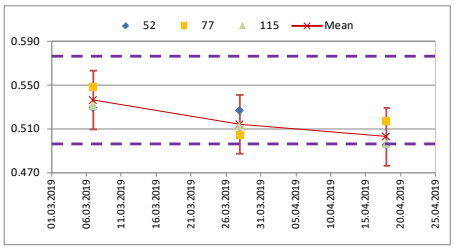
Mepiquat-Cl



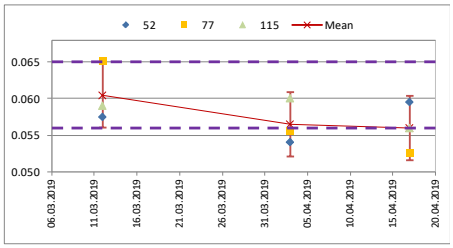
Appendix 4 Data of Stability Test

Glyphosate							2,4-DB						
AV [mg/kg]	0.535						AV [mg/kg]	0.061					
Date	07.03.2019		28.03.2019		18.04.2019		Date	12.03.2019		03.04.2019		17.04.2019	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 052	0.528	0.530	0.529	0.524	0.493	0.498	No. 052	0.058	0.057	0.053	0.055	0.059	0.060
No. 077	0.542	0.554	0.512	0.496	0.508	0.525	No. 077	0.068	0.062	0.055	0.056	0.054	0.051
No. 115	0.537	0.525	0.510	0.514	0.495	0.497	No. 115	0.058	0.060	0.065	0.055	0.059	0.053
Mean [mg/kg]	**0.536**		**0.514**		**0.503**		**Mean** [mg/kg]	**0.061**		**0.057**		**0.056**	
RSD* [%]	**1.9%**		**2.2%**		**2.4%**		**RSD*** [%]	**6.6%**		**5.5%**		**6.3%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-4.1%**		**-6.2%**			**—**		**-6.6%**		**-7.4%**	

Glyphosate



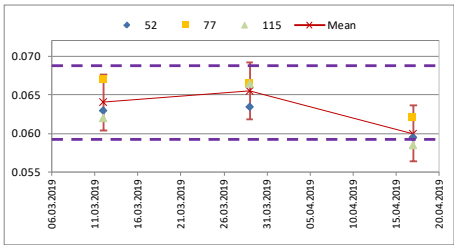
2,4-DB



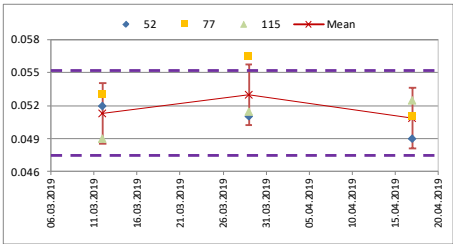
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25%

Avermectin B1a							Bixafen desmethyl						
AV [mg/kg]	0.058						AV [mg/kg]	0.050					
Date	12.03.2019		29.03.2019		17.04.2019		Date	12.03.2019		29.03.2019		17.04.2019	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 052	0.066	0.060	0.063	0.064	0.055	0.064	No. 052	0.052	0.052	0.050	0.052	0.052	0.046
No. 077	0.071	0.063	0.068	0.065	0.067	0.057	No. 077	0.050	0.056	0.059	0.054	0.050	0.052
No. 115	0.062	0.062	0.066	0.067	0.055	0.062	No. 115	0.044	0.054	0.048	0.055	0.052	0.053
Mean [mg/kg]	**0.064**		**0.066**		**0.060**		**Mean** [mg/kg]	**0.051**		**0.053**		**0.051**	
RSD* [%]	**4.1%**		**2.6%**		**3.0%**		**RSD*** [%]	**4.1%**		**5.7%**		**3.5%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**2.3%**		**-6.3%**			**—**		**3.2%**		**-1.0%**	

Avermectin B1a



Bixafen desmethyl



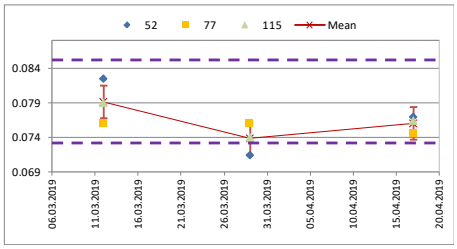
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25%

* RSD = relative standard diviation

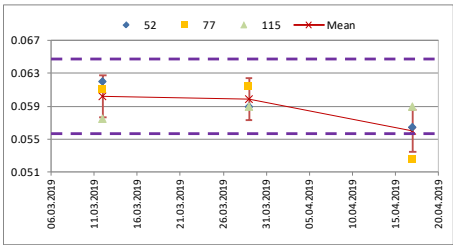
Appendix 4 (cont.): Data of Stability Test

Boscalid metabolite M510F01							Bromoxynil						
AV [mg/kg]	0.081						AV [mg/kg]	0.059					
Date	12.03.2019		29.03.2019		17.04.2019		Date	12.03.2019		29.03.2019		17.04.2019	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 052	0.083	0.082	0.078	0.065	0.076	0.078	No. 052	0.060	0.064	0.058	0.060	0.058	0.055
No. 077	0.071	0.081	0.076	#	0.076	0.073	No. 077	0.063	0.059	0.066	0.057	0.052	0.053
No. 115	0.077	0.081	#	0.074	0.076	0.077	No. 115	0.054	0.061	0.055	0.063	0.058	0.060
Mean [mg/kg]	**0.079**		**0.074**		**0.076**		**Mean** [mg/kg]	**0.060**		**0.060**		**0.056**	
RSD* [%]	**4.1%**		**3.1%**		**1.7%**		**RSD*** [%]	**3.9%**		**2.4%**		**5.9%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-6.7%**		**-4.0%**			**—**		**-0.6%**		**-6.9%**	

Boscalid metabolite M510F01



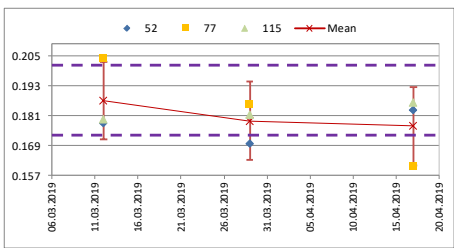
Bromoxynil



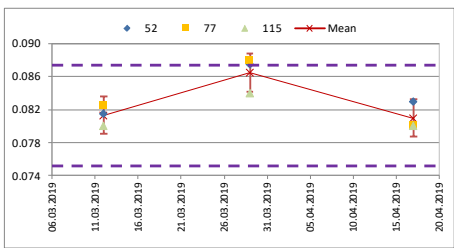
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25%

DDAC C10-Cl							Fenpropimorph Carboxylic Acid (BF-421-2)						
AV [mg/kg]	0.177						AV [mg/kg]	0.088					
Date	12.03.2019		29.03.2019		17.04.2019		Date	12.03.2019		29.03.2019		17.04.2019	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 052	0.172	0.184	0.174	0.166	0.182	0.185	No. 052	0.082	0.081	0.087	0.088	0.076	0.090
No. 077	0.221	0.187	0.191	0.180	0.168	0.153	No. 077	0.086	0.079	0.089	0.087	0.081	0.079
No. 115	0.188	0.171	0.186	0.177	0.182	0.191	No. 115	0.079	0.081	0.082	0.086	0.076	0.084
Mean [mg/kg]	**0.187**		**0.179**		**0.177**		**Mean** [mg/kg]	**0.081**		**0.087**		**0.081**	
RSD* [%]	**7.8%**		**4.5%**		**8.0%**		**RSD*** [%]	**1.5%**		**2.5%**		**2.1%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-4.4%**		**-5.5%**			**—**		**6.4%**		**-0.4%**	

DDAC C10-Cl



Fenpropimorph Carb. Acid



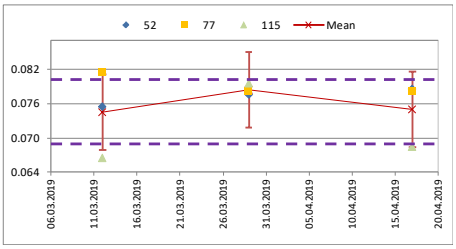
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25%

* RSD = relative standard diviation

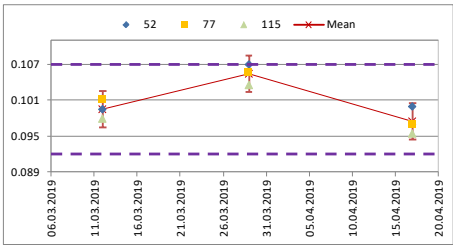
Appendix 4 (cont.): Data of Stability Test

Flonicamid metabolite TFNA-AM							Fluopyram-benzamid (M25)						
AV [mg/kg]	0.073						AV [mg/kg]	0.101					
Date	12.03.2019		29.03.2019		17.04.2019		Date	12.03.2019		29.03.2019		17.04.2019	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 052	0.074	0.077	0.075	0.080	0.065	0.092	No. 052	0.104	0.095	0.105	0.109	0.094	0.106
No. 077	0.086	0.077	0.080	0.076	0.088	0.068	No. 077	0.103	0.099	0.108	0.103	0.099	0.095
No. 115	0.070	0.063	0.079	0.080	0.065	0.072	No. 115	0.097	0.099	0.106	0.101	0.094	0.097
Mean [mg/kg]	**0.075**		**0.078**		**0.075**		**Mean** [mg/kg]	**0.100**		**0.105**		**0.098**	
RSD* [%]	**10.1%**		**1.3%**		**7.5%**		**RSD*** [%]	**1.5%**		**1.7%**		**2.4%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**5.1%**		**0.7%**			**—**		**5.9%**		**-2.0%**	

Flonicamid metabolite TFNA-AM



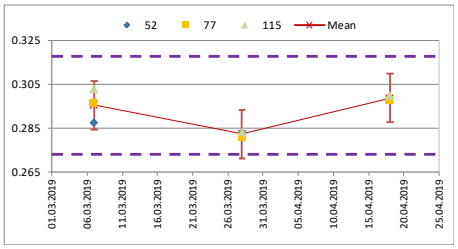
Fluopyram-benzamid (M25)



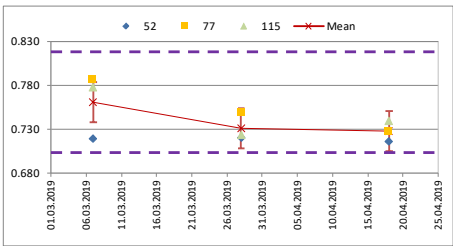
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25%

MPP							AMPA						
AV [mg/kg]	0.309						AV [mg/kg]	0.754					
Date	07.03.2019		28.03.2019		18.04.2019		Date	07.03.2019		28.03.2019		18.04.2019	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 052	0.288	0.287	0.261	0.305	0.308	0.289	No. 052	0.747	0.693	0.705	0.737	0.731	0.701
No. 077	0.295	0.297	0.275	0.287	0.298	0.298	No. 077	0.773	0.800	0.736	0.763	0.714	0.742
No. 115	0.301	0.305	0.290	0.277	0.298	0.302	No. 115	0.773	0.782	0.756	0.692	0.758	0.722
Mean [mg/kg]	**0.296**		**0.283**		**0.299**		**Mean** [mg/kg]	**0.761**		**0.732**		**0.728**	
RSD* [%]	**2.6%**		**0.5%**		**0.3%**		**RSD*** [%]	**4.7%**		**2.1%**		**1.6%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-4.4%**		**1.1%**			**—**		**-3.9%**		**-4.4%**	

MPP



AMPA



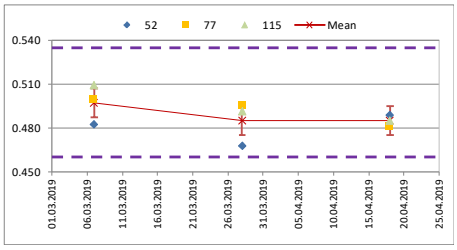
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25%

* RSD = relative standard diviation

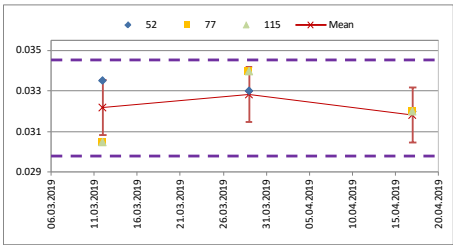
Appendix 4 (cont.): Data of Stability Test

N-Acetyl-glyphosate							Haloxyfop						
AV [mg/kg]	0.543						AV [mg/kg]	0.037					
Date	07.03.2019		28.03.2019		18.04.2019		Date	12.03.2019		29.03.2019		17.04.2019	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 052	0.500	0.466	0.466	0.470	0.482	0.496	No. 052	0.031	0.035	0.031	0.034	0.032	0.031
No. 077	0.497	0.503	0.510	0.481	0.499	0.464	No. 077	0.029	0.031	0.033	0.034	0.032	0.031
No. 115	0.498	0.521	0.502	0.481	0.472	0.498	No. 115	0.029	0.035	0.030	0.032	0.032	0.030
Mean [mg/kg]	**0.498**		**0.485**		**0.485**		**Mean** [mg/kg]	**0.032**		**0.032**		**0.031**	
RSD* [%]	**2.7%**		**3.1%**		**0.8%**		**RSD*** [%]	**4.8%**		**3.9%**		**0.9%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-2.5%**		**-2.5%**			**—**		**2.1%**		**-1.1%**	

N-Acetyl-glyphosate



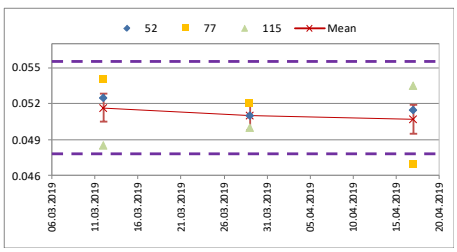
Haloxyfop



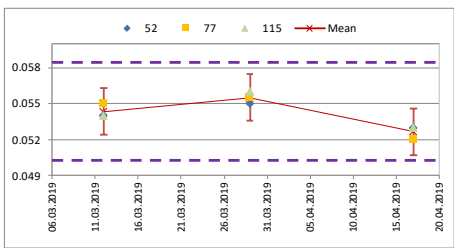
- - - : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25%

MCPA							Mepiquat-Cl						
AV [mg/kg]	0.046						AV [mg/kg]	0.051					
Date	12.03.2019		29.03.2019		17.04.2019		Date	12.03.2019		29.03.2019		17.04.2019	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 052	0.052	0.053	0.051	0.051	0.052	0.051	No. 052	0.051	0.057	0.053	0.057	0.052	0.054
No. 077	0.057	0.051	0.056	0.048	0.050	0.044	No. 077	0.055	0.055	0.055	0.056	0.051	0.053
No. 115	0.047	0.050	0.047	0.053	0.052	0.055	No. 115	0.055	0.053	0.055	0.057	0.052	0.054
Mean [mg/kg]	**0.052**		**0.051**		**0.051**		**Mean** [mg/kg]	**0.054**		**0.056**		**0.053**	
RSD* [%]	**5.5%**		**2.0%**		**6.6%**		**RSD*** [%]	**1.1%**		**0.9%**		**1.1%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-1.3%**		**-1.9%**			**—**		**2.1%**		**-3.1%**	

MCPA



Mepiquat-Cl

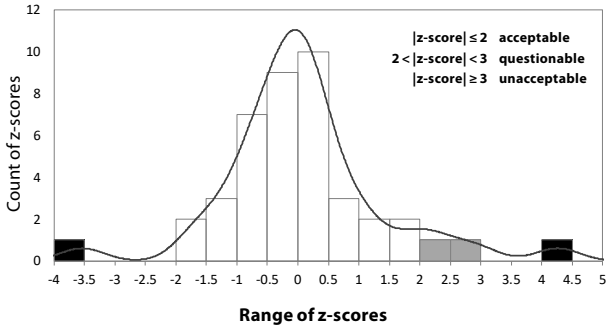


- - - : upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25%

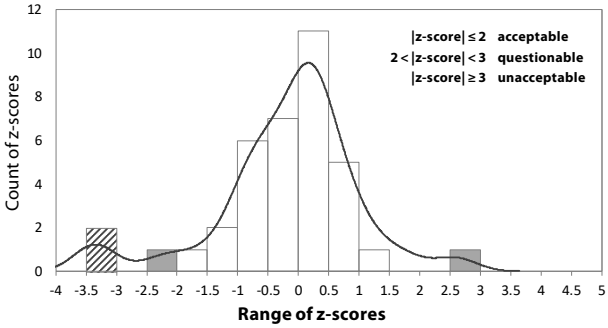
* RSD = relative standard diviation

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

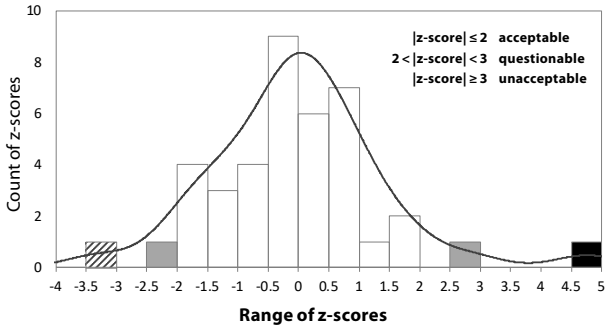
Glyphosate
(AV derived from entire population excl. one outlier)



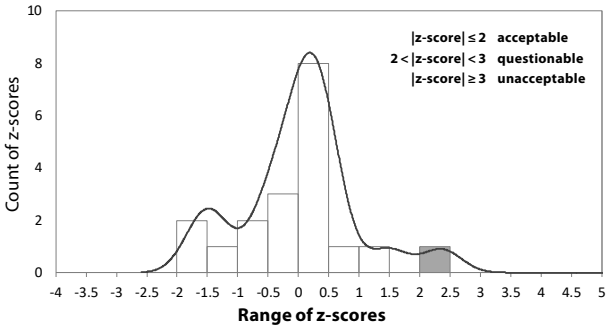
2,4-DB
(AV derived from entire population)



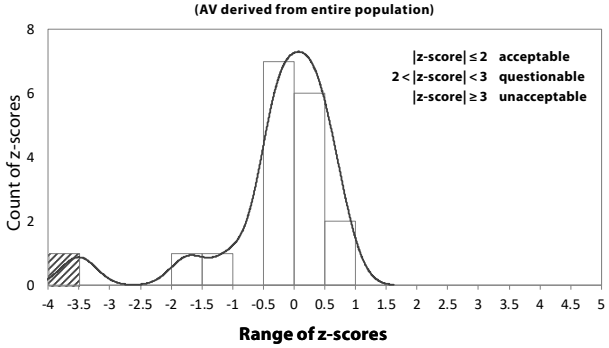
Avermectin B1a
(AV derived from entire population)



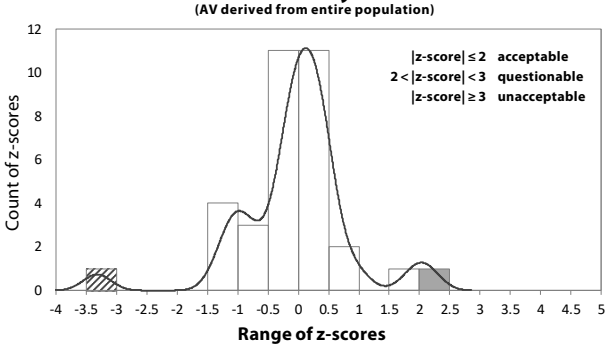
Desmethyl bixafen
(AV derived from entire population excl. one outlier)



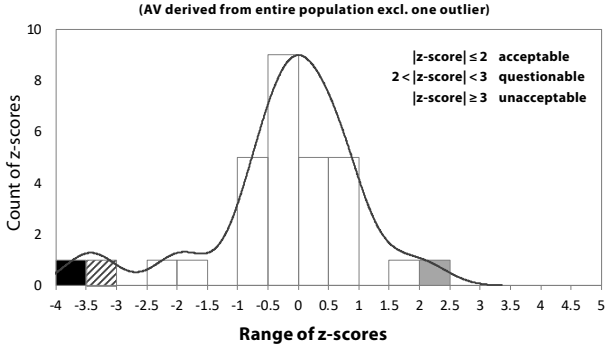
Boscalid Met. M510F017
(AV derived from entire population)



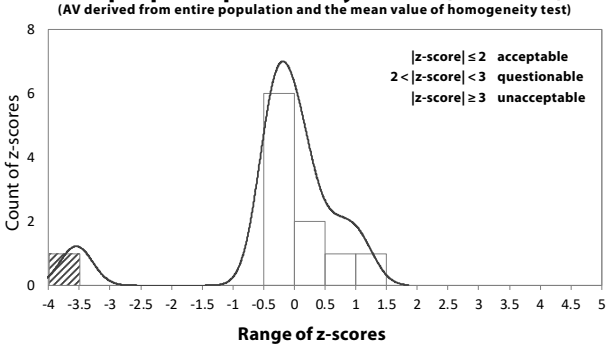
Bromoxynil
(AV derived from entire population)



DDAC-C10-Cl
(AV derived from entire population excl. one outlier)



Fenpropimorph carboxylic acid (BF-421-2)
(AV derived from entire population and the mean value of homogeneity test)



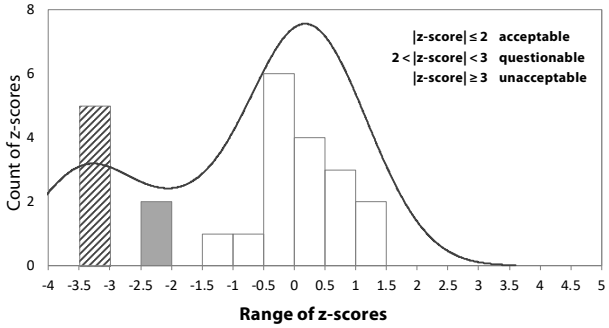
* Cut-off at z-score = 5

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

(Results from EU and EFTA Laboratories only)

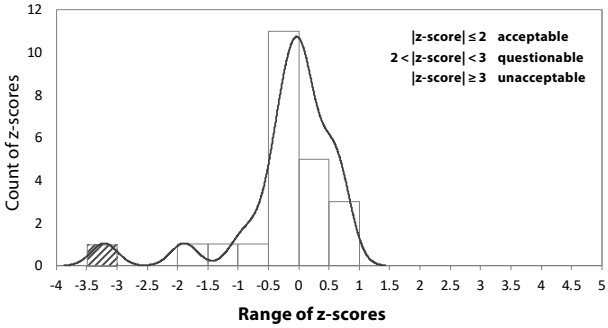
Flonicamid Met. TFNA-AM

(AV derived from entire population)



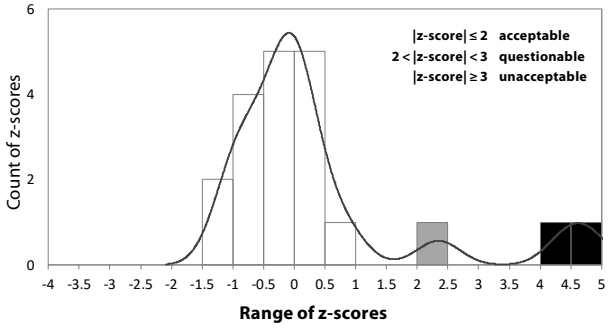
Fluopyram-benzamide (M25)

(AV derived from entire population)



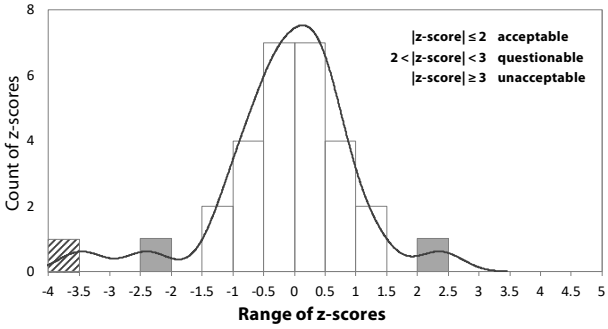
MPP

(AV derived from entire population)



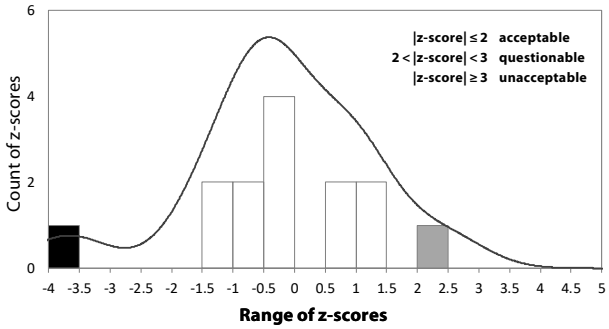
AMPA

(AV derived from entire population)



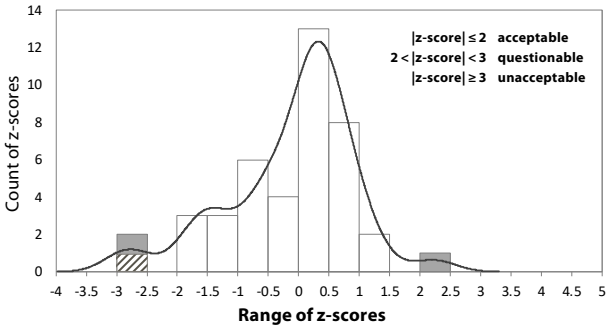
N-Acetyl-glyphosate

(AV derived from entire population excl. 3 outliers)



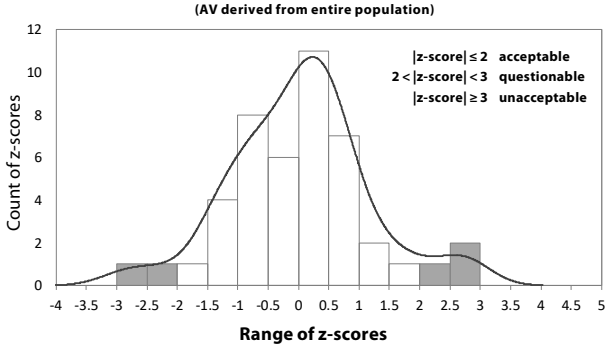
Haloxyfop

(AV derived from entire population)



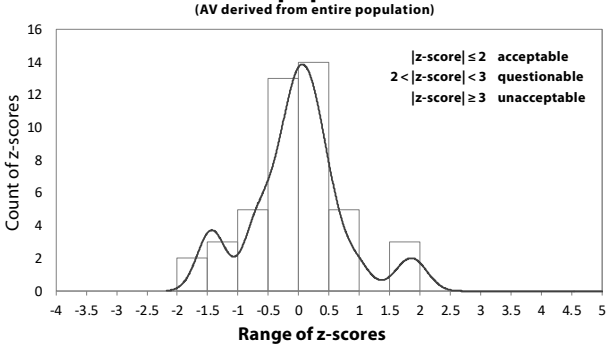
MCPA

(AV derived from entire population)



Mepiquat-Cl

(AV derived from entire population)



* Cut-off at z-score = 5

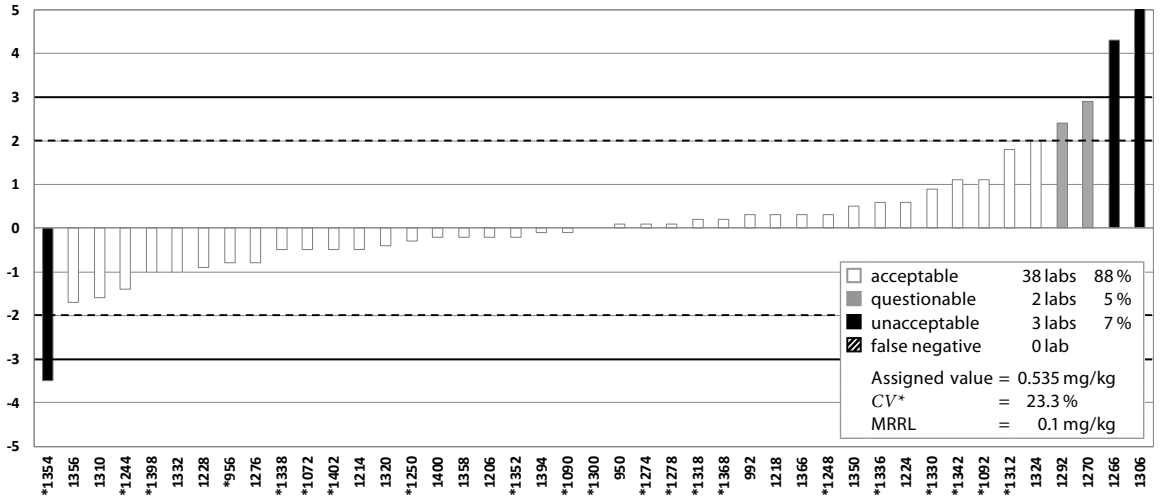
Appendix 6 Graphic Presentation of z-Scores and Results

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

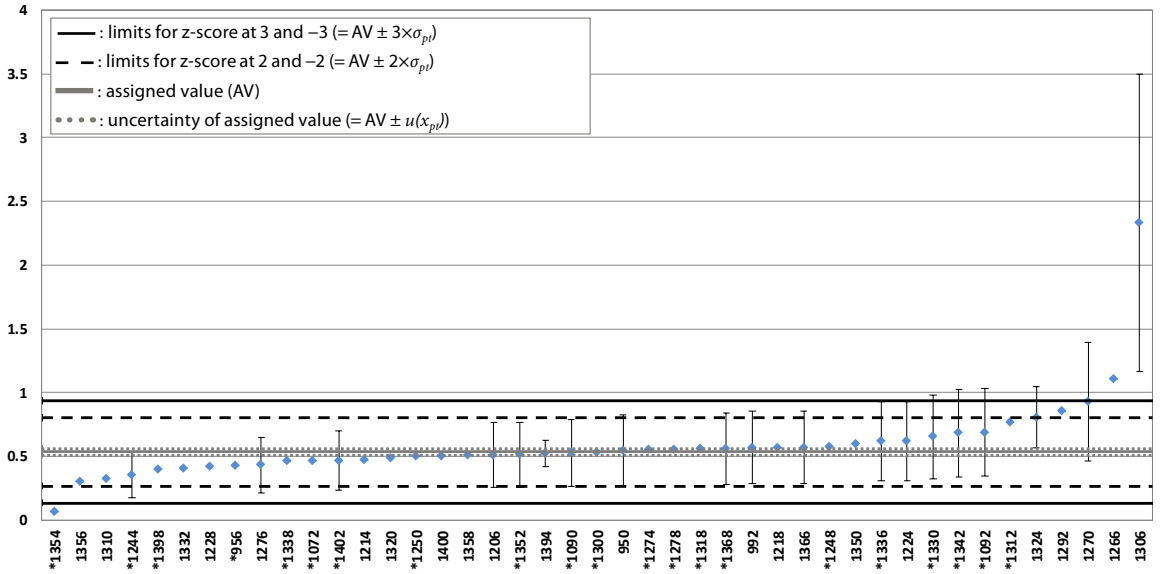
Glyphosate

(Assigned value and CV^* derived from entire population excl. 1 outlier)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



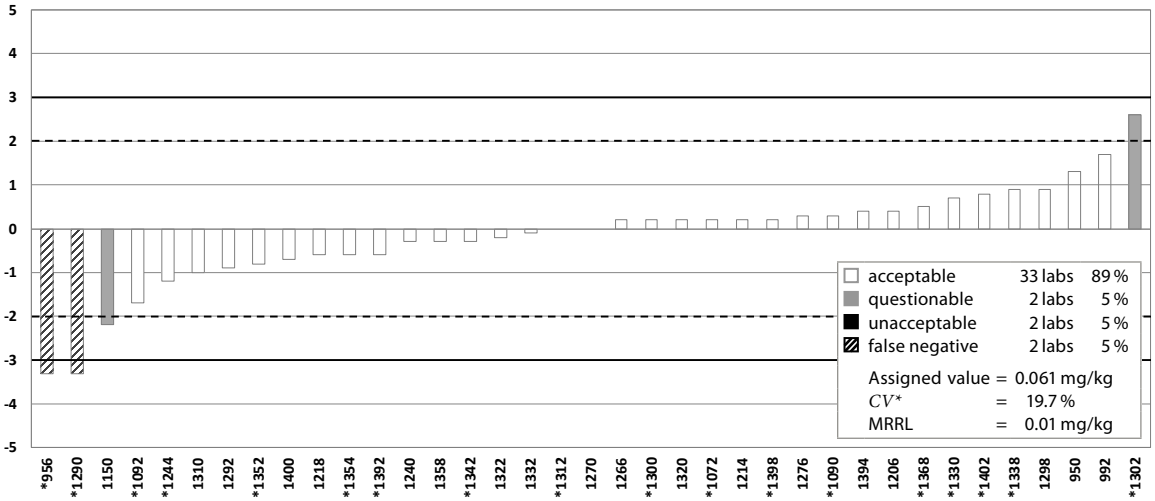
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

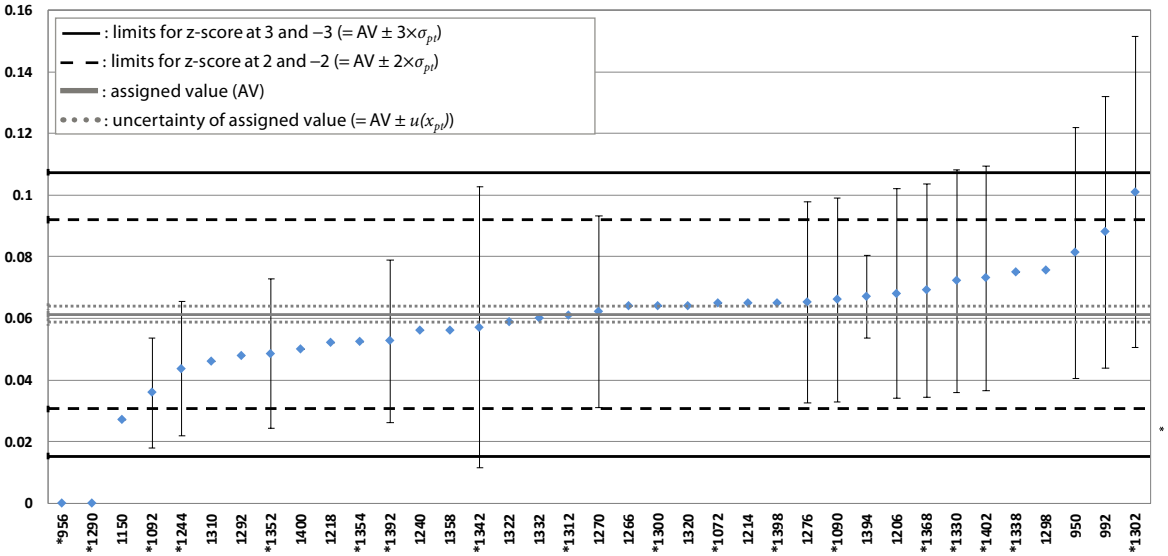
2,4-DB

(Assigned value and CV^* derived from entire population)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



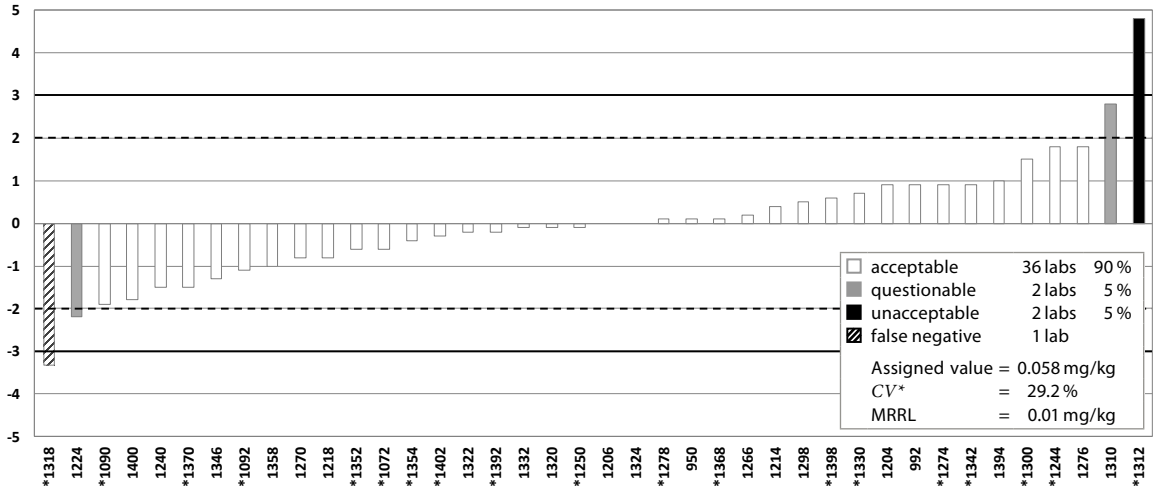
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

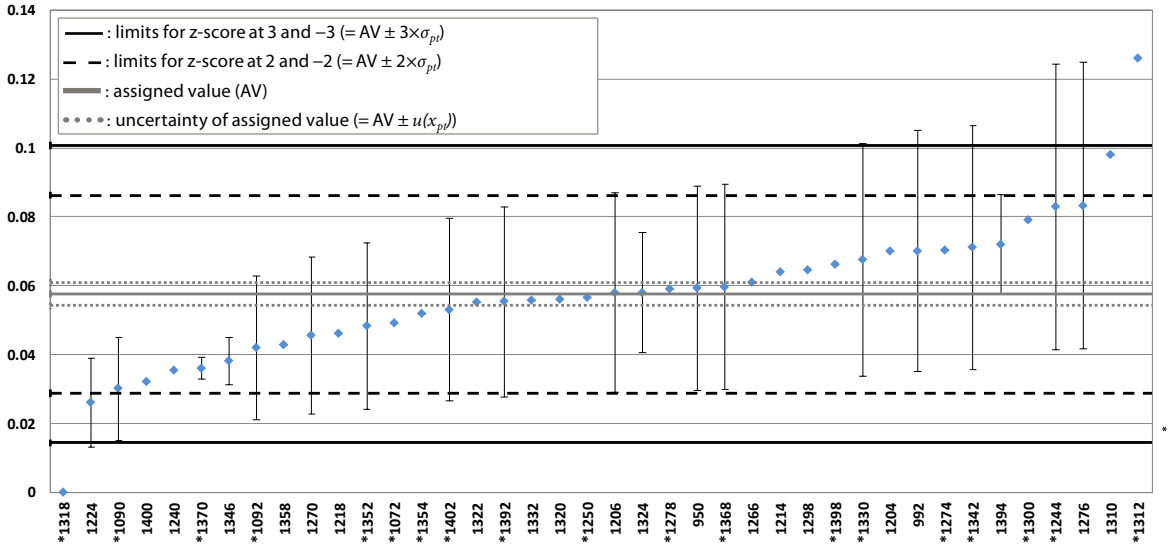
Avermectin B1a

(Assigned value and CV^* derived from entire population)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



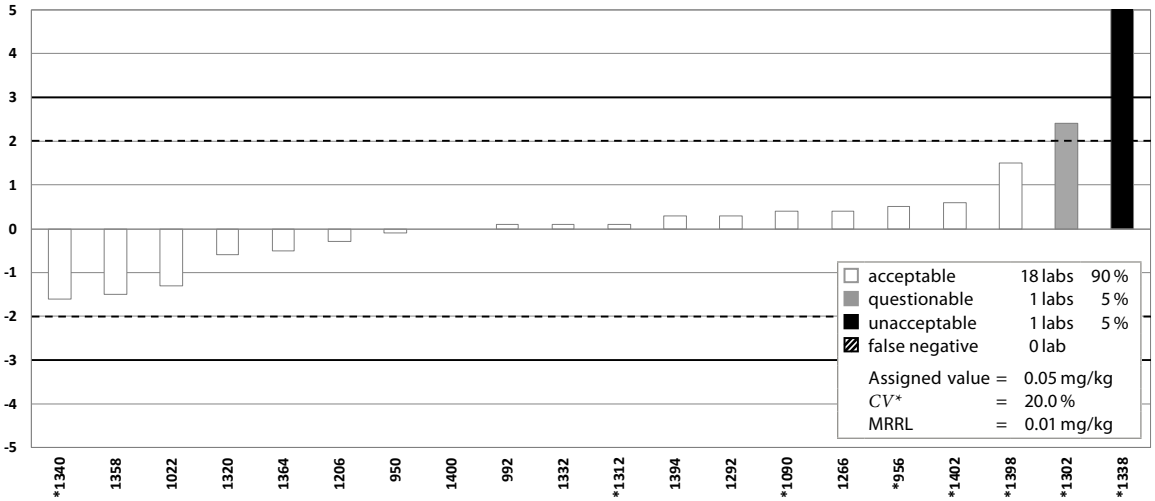
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

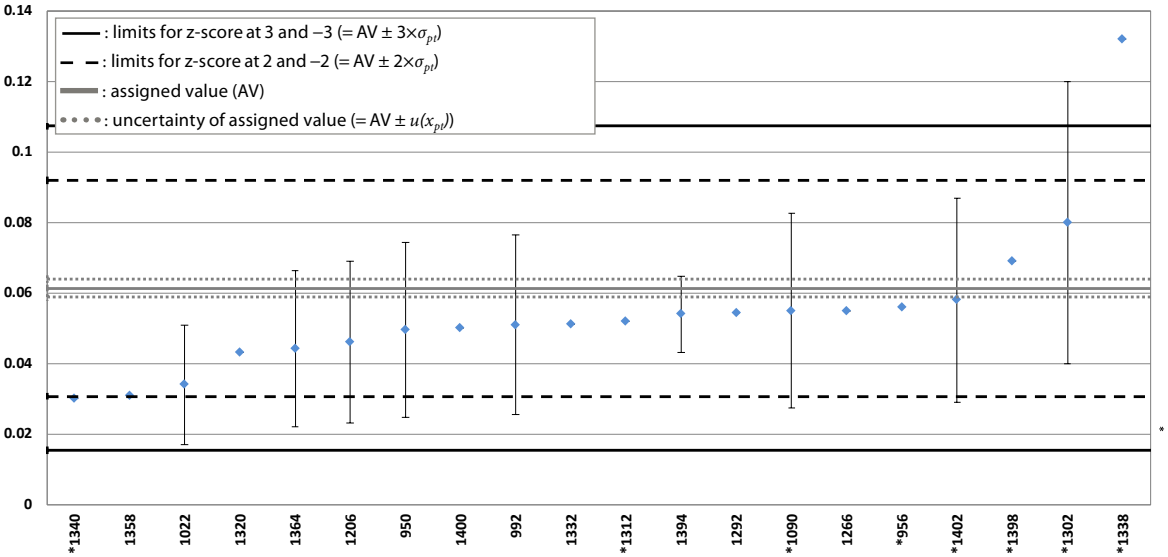
Desmethyl bixafen

(Assigned value and CV^* derived from entire population excl. one outlier)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



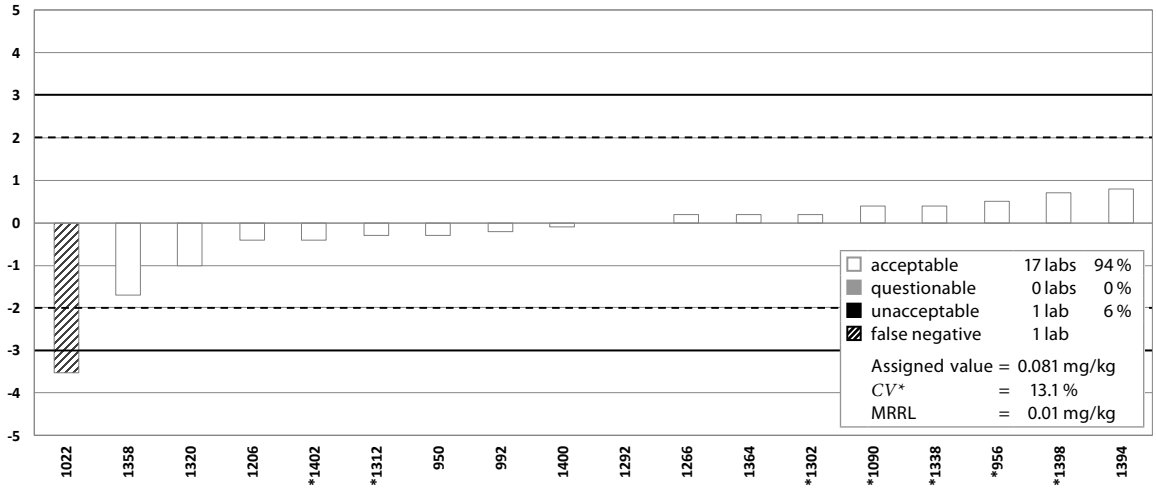
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

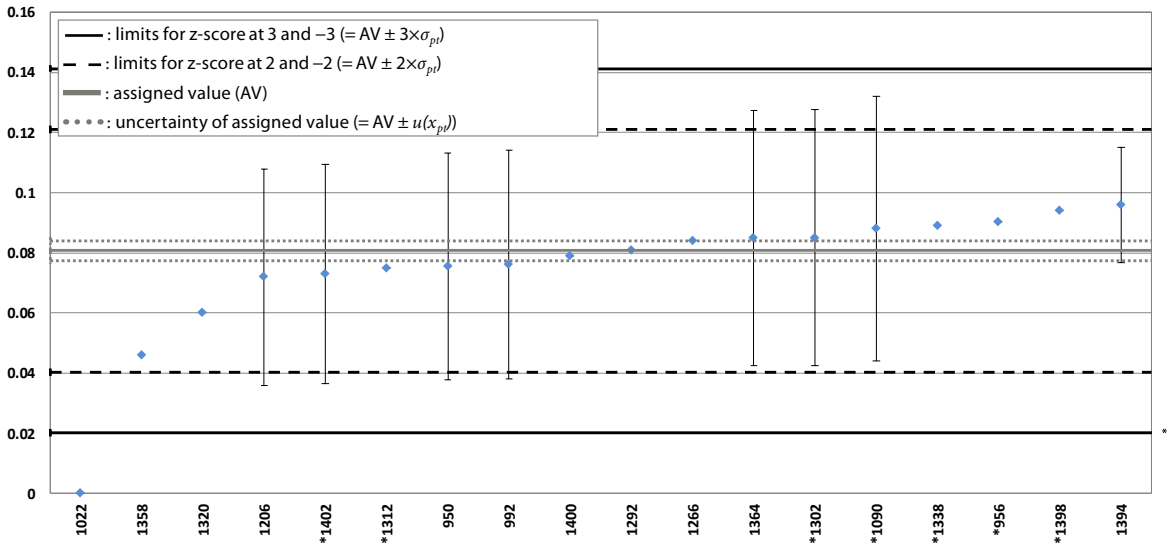
Boscalid Met. M510F017

(Assigned value and CV^* derived from entire population)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



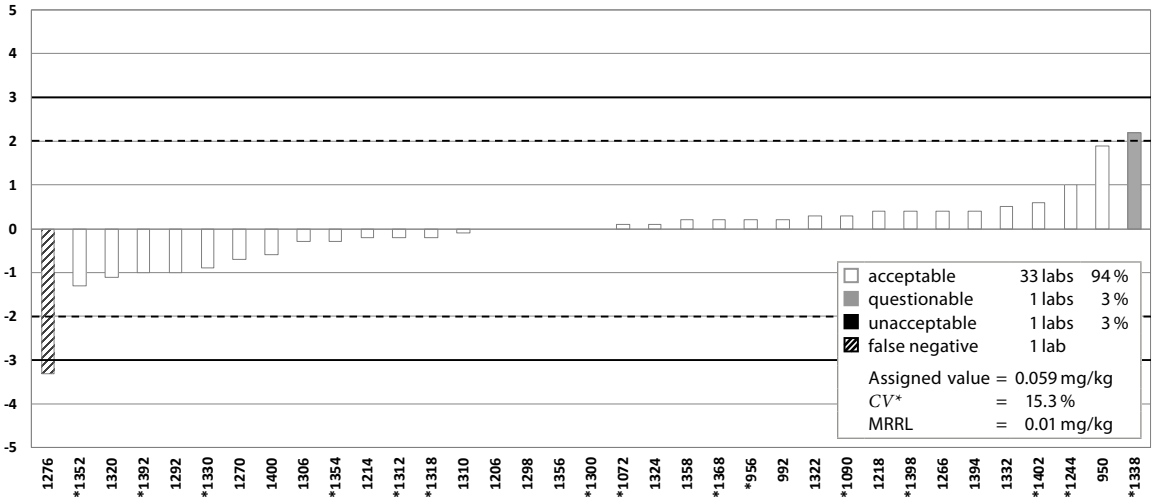
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

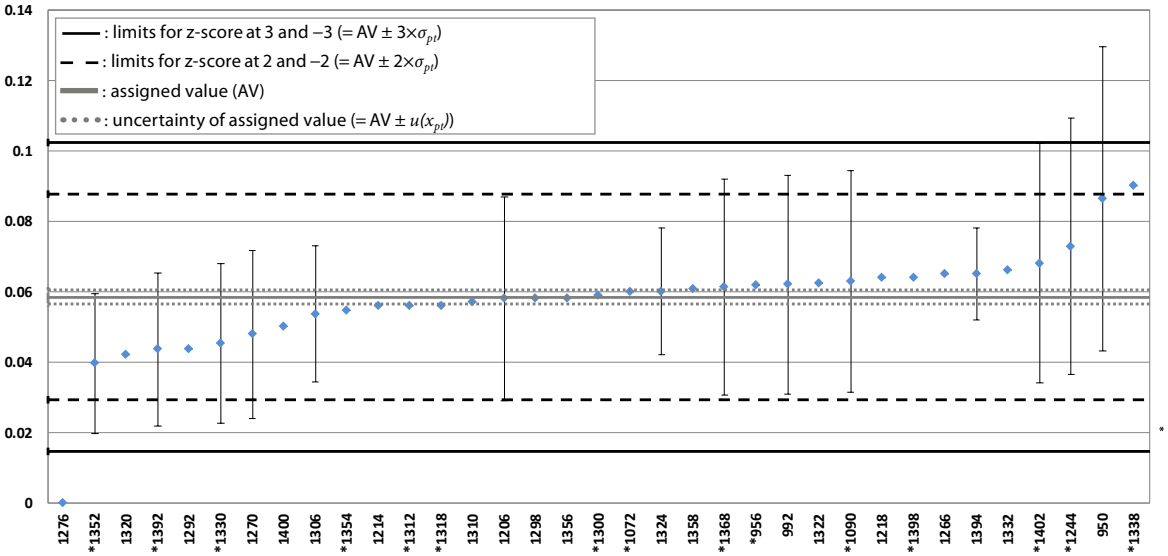
Bromoxynil

(Assigned value and CV^* derived from entire population)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



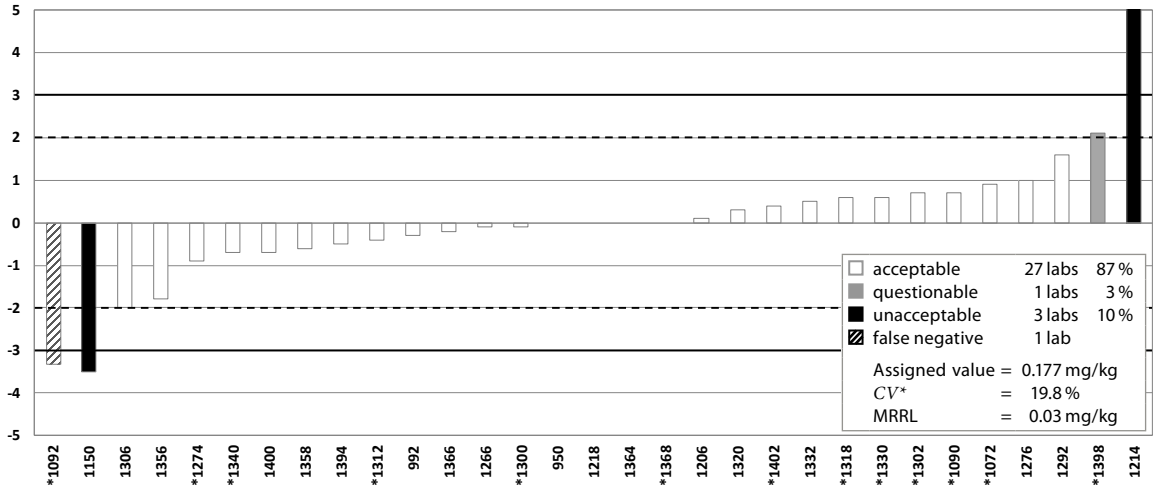
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

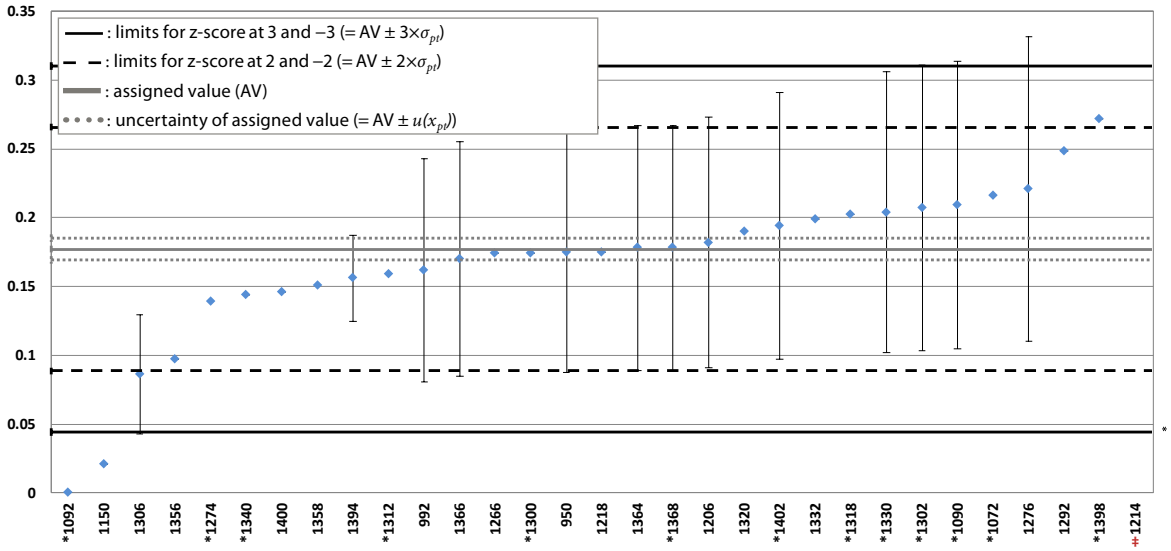
DDAC-C10-Cl

(Assigned value and CV^* derived from entire population excl. 1 outlier)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



‡ reported by mistake 144 mg/kg and not shown in the graph

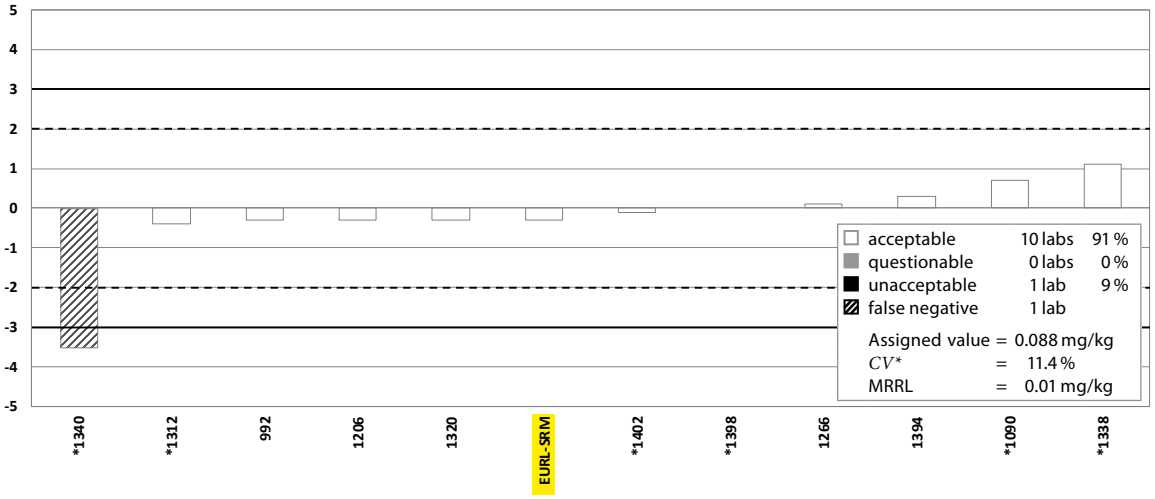
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

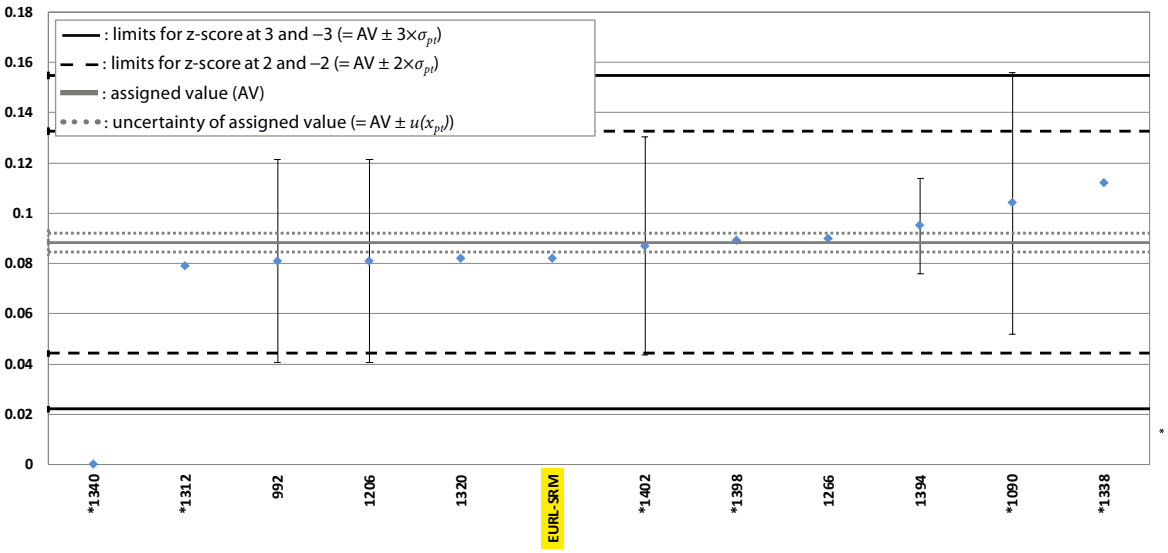
Fenpropimorph carboxylic acid (BF-421-2)

(Assigned value and CV^* derived from entire population the mean value of homogeneity test)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



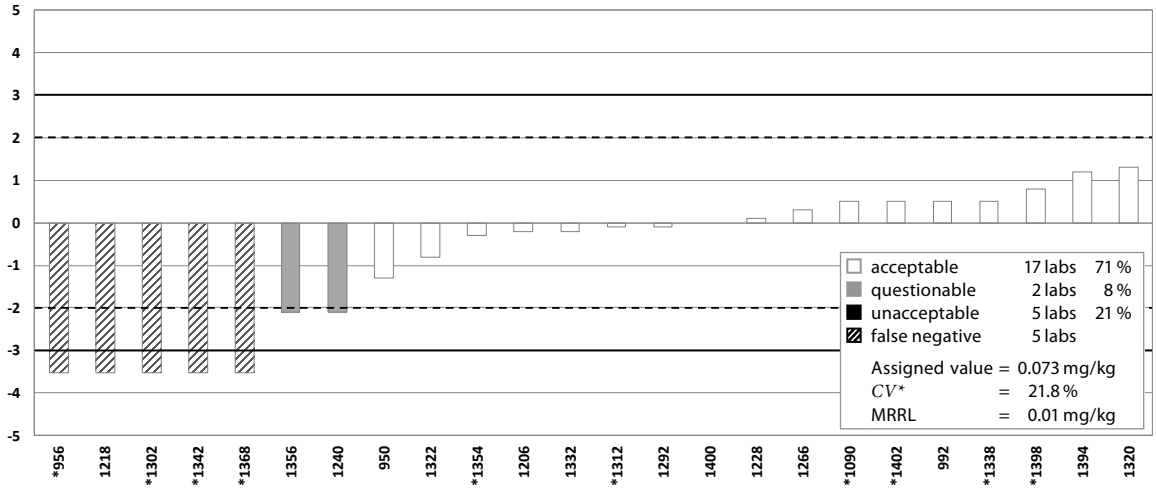
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

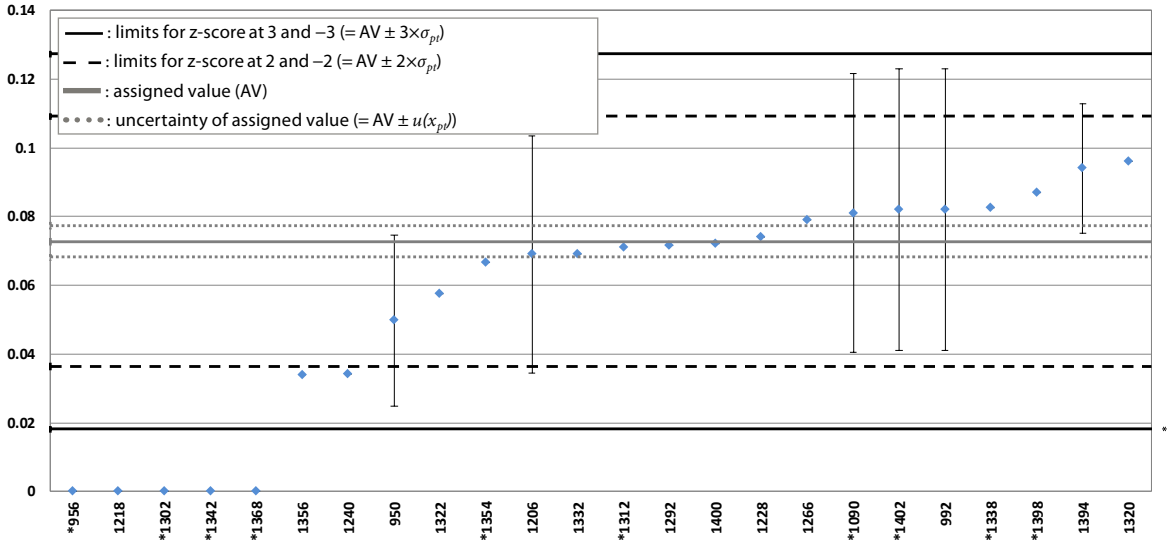
Flonicamid Met. TFNA-AM

(Assigned value and CV^* derived from entire population)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



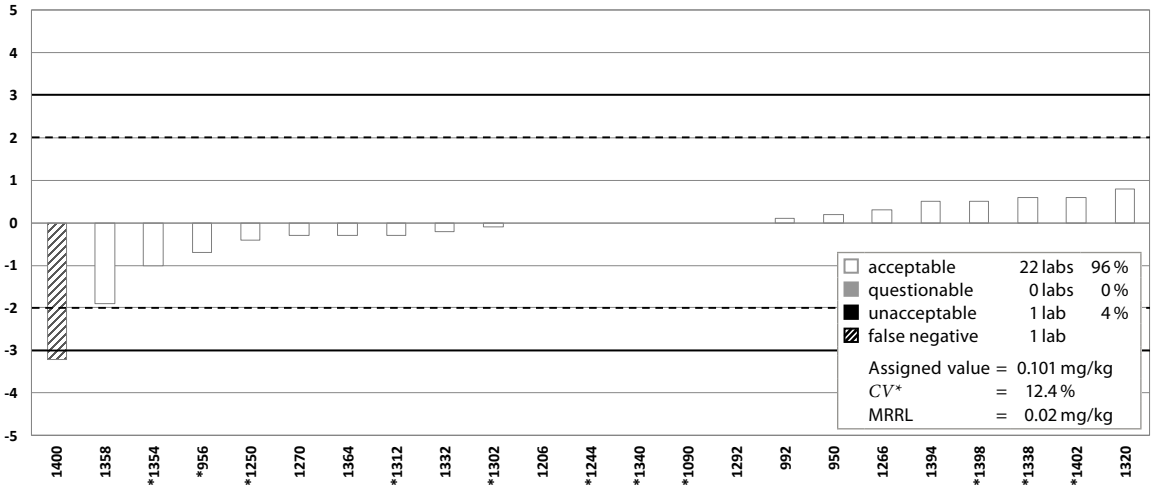
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

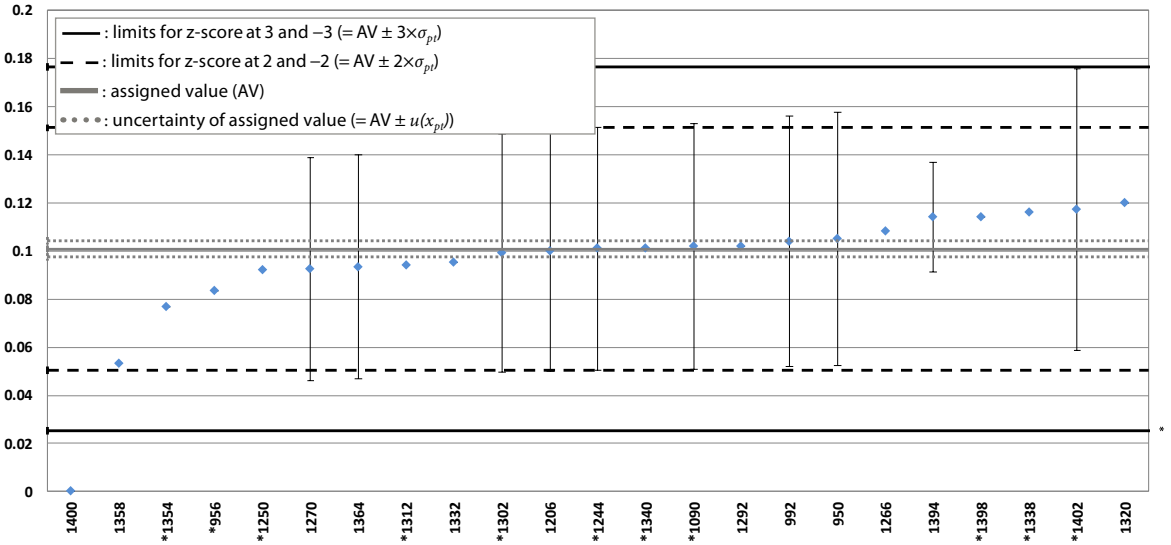
Fluopyram-benzamide (M25)

(Assigned value and CV^* derived from entire population)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



A6 Z-SCORE DISTRIBUTION

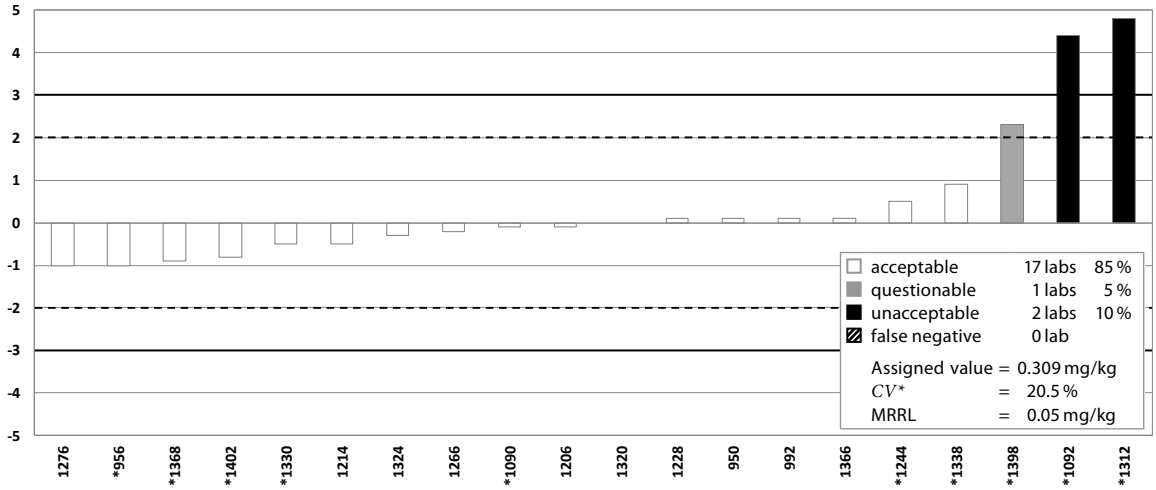
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

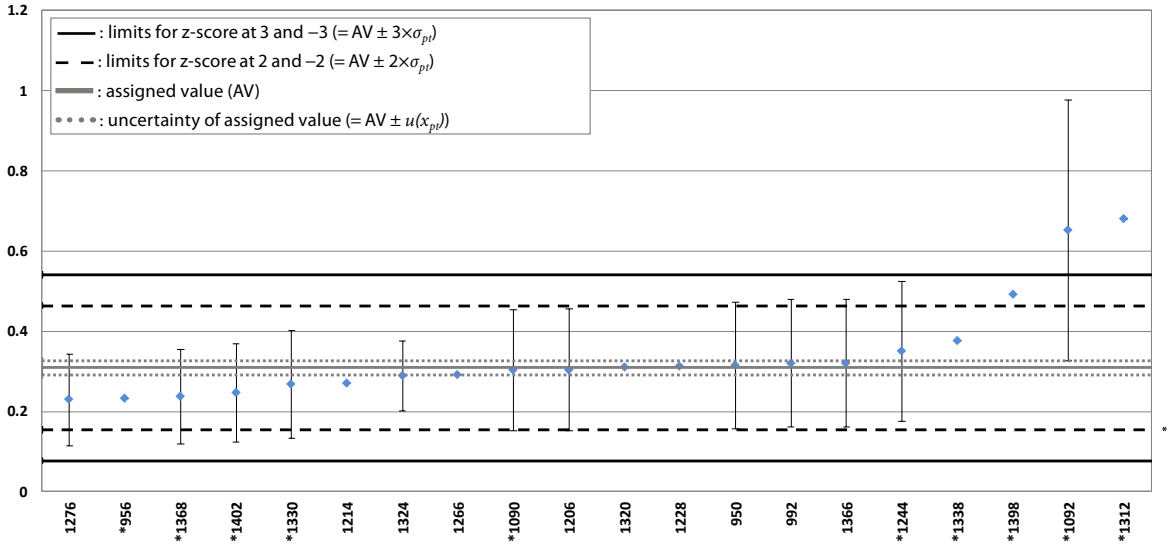
MPP

(Assigned value and CV^* derived from entire population excl. 1 outlier)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



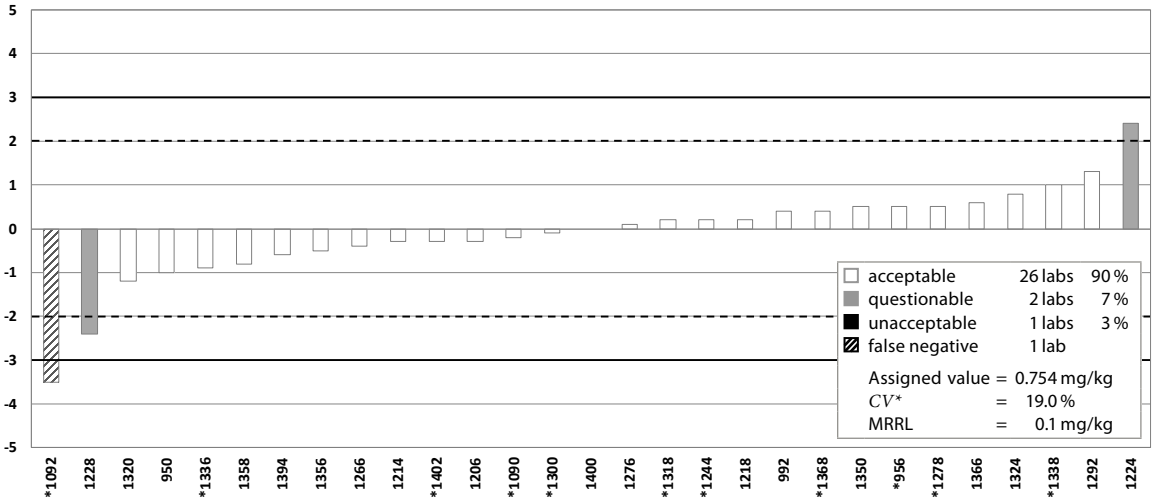
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

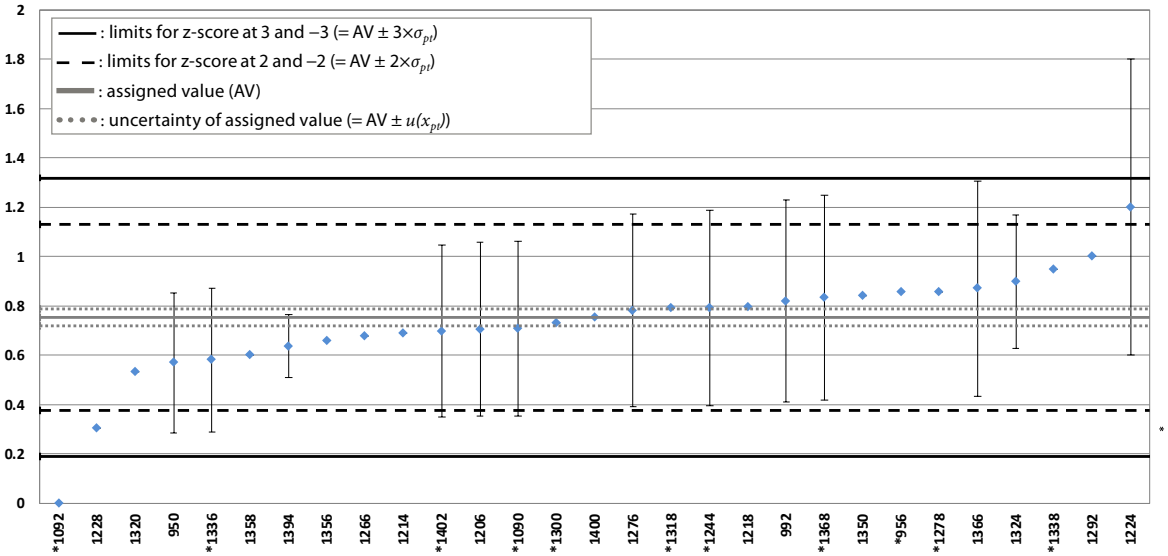
AMPA

(Assigned value and CV^* derived from entire population)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



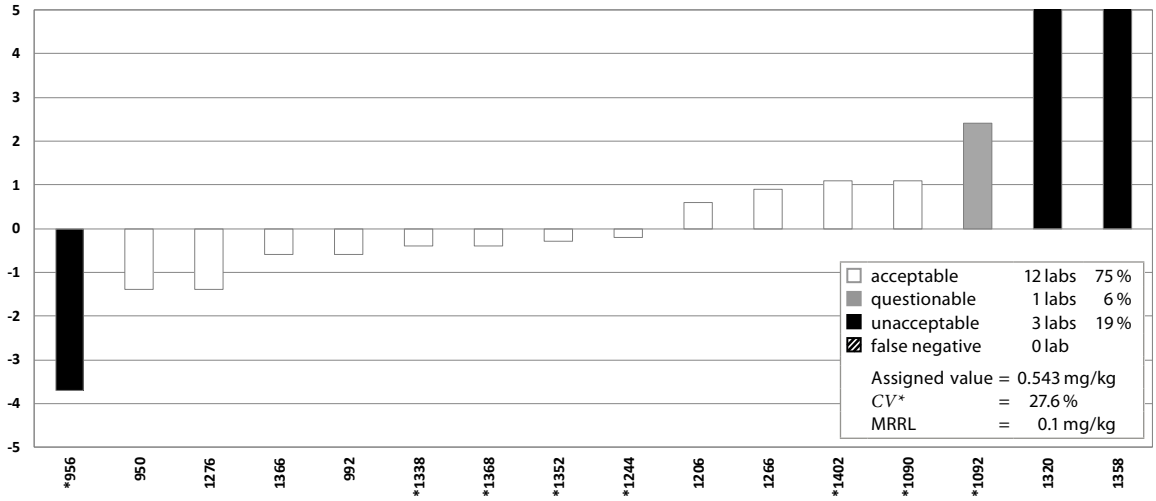
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

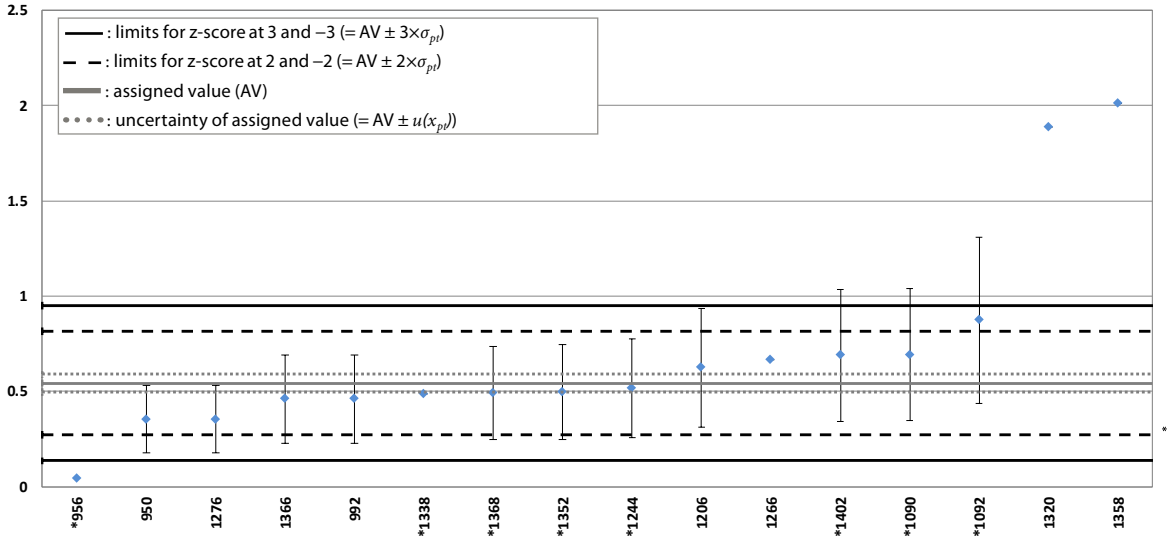
N-Acetyl-glyphosate

(Assigned value and CV^* derived from entire population excl. 3 outliers)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



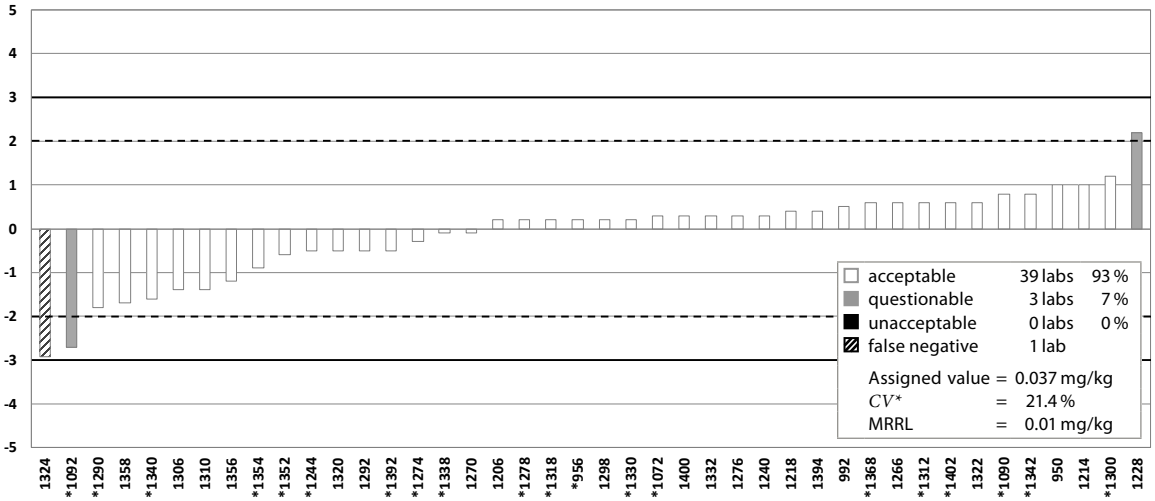
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

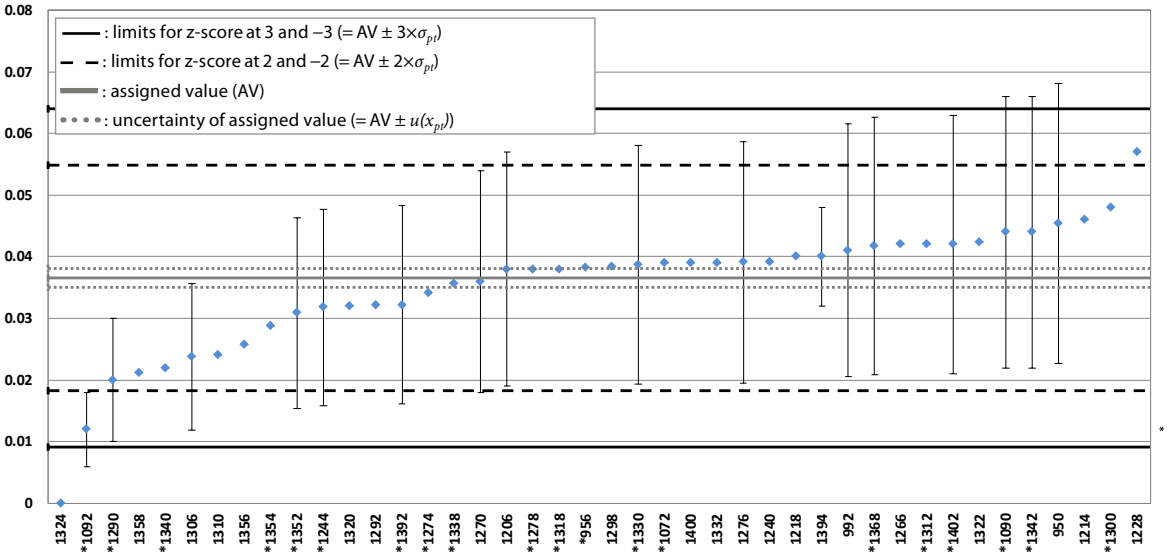
Haloxyfop

(Assigned value and CV^* derived from entire population)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



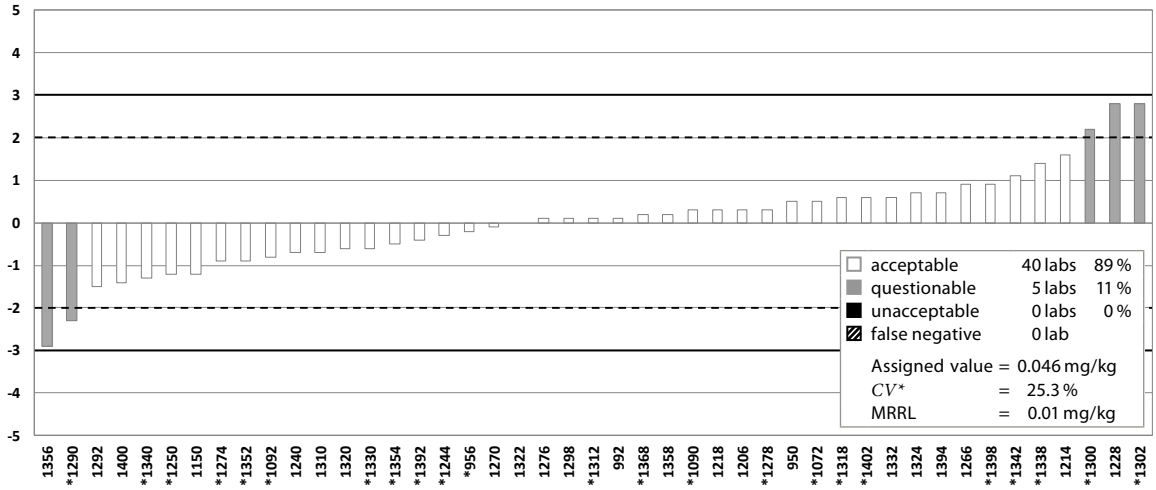
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

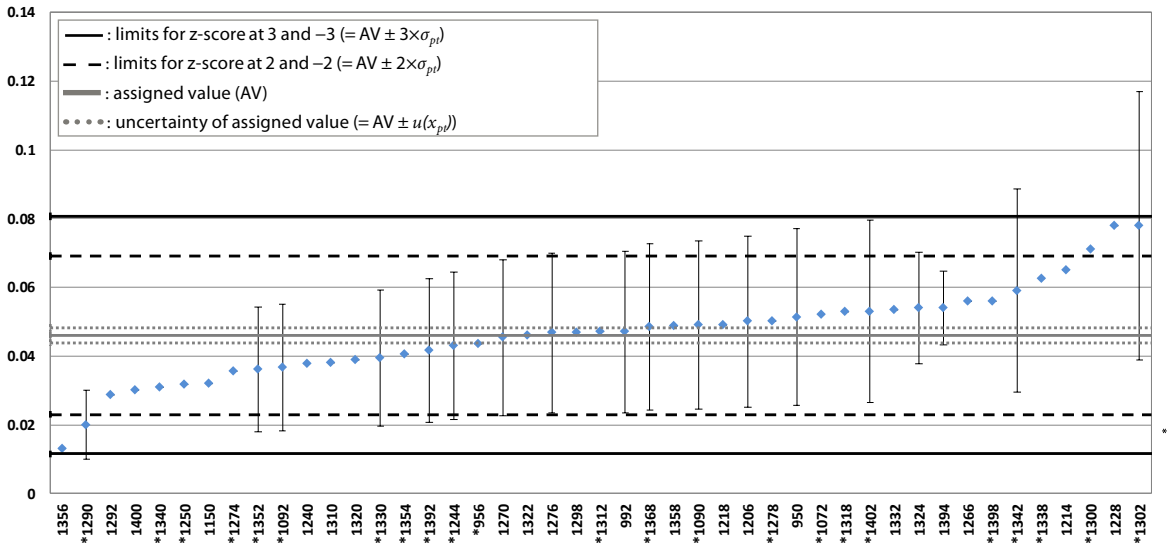
MCPA

(Assigned value and CV^* derived from entire population excl. 1 outlier)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



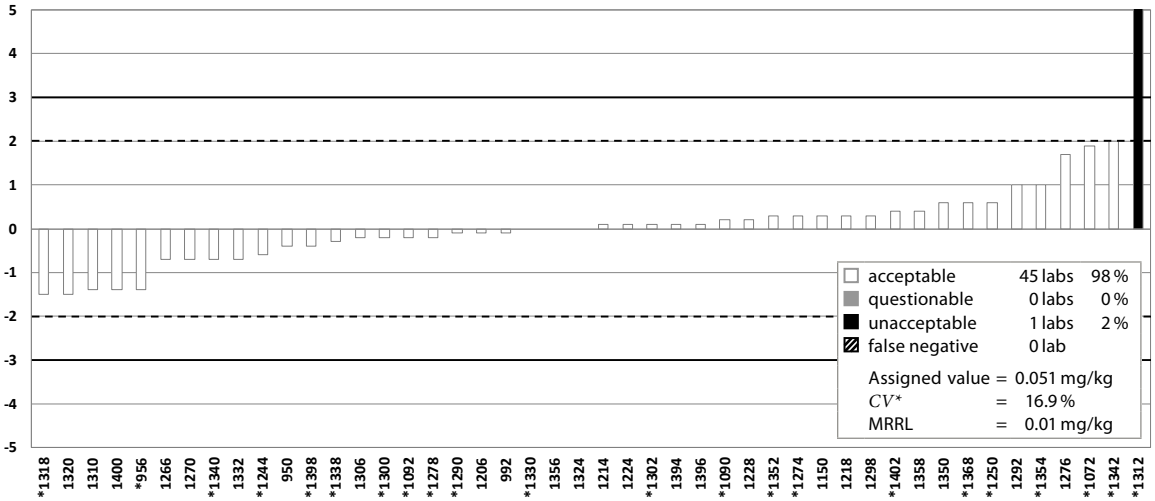
Appendix 6 (cont.) Graphic Presentation of z-Scores and Results

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

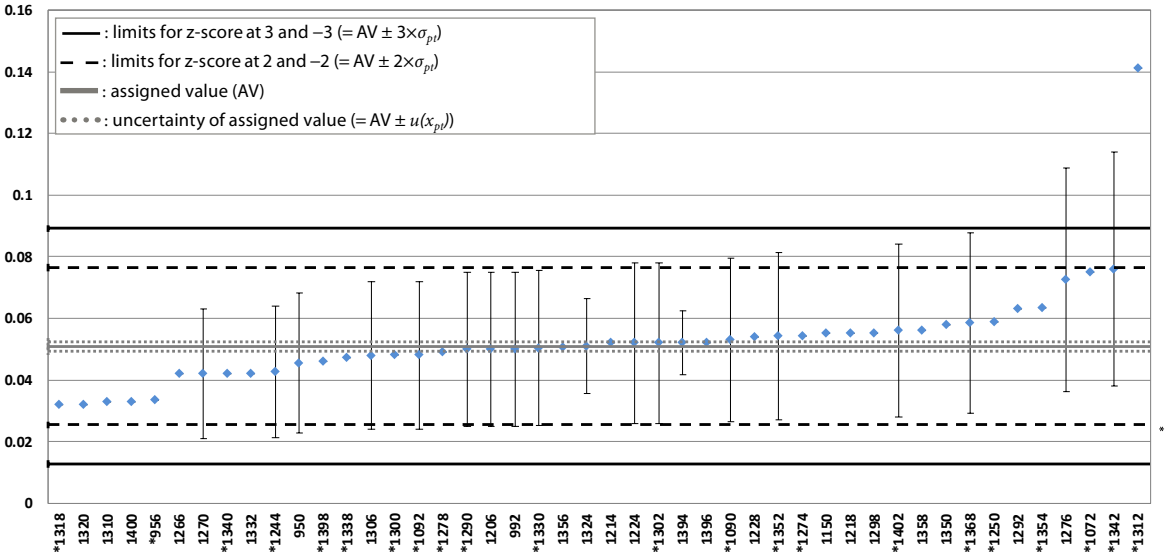
Mepiquat-Cl

(Assigned value and CV^* derived from entire population)

z-score



Conc. [mg/kg] (incl. measurement uncertainty reported by participating laboratories)



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

A: Lack of Experience
B: Improper / unsuitable method
C: Matrix effect not properly compensated
D: Error in concentration of calibration
E: Incorrect calculation
F: Improper recovery correction
G: Interference in chromatogram
H: Technical problem
I: Transcription error
J: No validation
K: False compound analyzed
L: Reporting lebel too high or near assigned value

Glyphosate Assigned value: 0.535 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
1354	-3.5	Yes	The poor performance you mentioned comes from incorrect data recording. Our glyphosate result is 0.0535 mg/kg, and not 0.535 mg/kg, but I mistyped a number when the result was entered (Anm. Pat: In der Antwort Zahlen wieder verdreht!)	I
1292	2.4	(Yes)	Die Aufarbeitung der Proben wurden mit der QuPPe AO Method V 3 durchgeführt, wobei auf den Zentrifugationsschritt mit dem Ultrafiltrationsfilter (Amicon Ultra 4 von Merck) verzichtet wurden, da die Zentrifugation 30 min und länger dauerte, um ausreichend Eluat zu erhalten. Bei Messung auf der Hypercarb-Säule kam es zu enormen Störungen durch mitextrahierte Begleitstoffe und RZ-Schwankungen, die die Auswertung selbst im Bereich 0,5 mg/kg (Processierte Kalibration, Zusatz auf Matrix Bio-Leber) sehr erschwerten. Im Einzelnen lagen die ermittelten Gehalte zwischen 0,666 und 1,07 mg/kg, die Korrelation zweier Kalibration lagen bei r^2 0,896 und r^2 0,963. Für weitere Versuche fehlte leider die Zeit, es wäre angedacht gewesen, die Extraktion statt mit saurem Methanol mit saurem Wasser durchzuführen, um weniger Begleitstoffe zu eluieren, die später zu den beobachteten starken Matrixeffekten geführt haben. Die im derartig hohen Konzentrationsbereich (0,5 mg/kg Glyphosat) geschilderten Probleme führen zur Zeit dazu, dass Validierungen in Bereichen von 5 – 10 µg/kg (Konzentrationsbereich, der in der Realität ggf. relevant werden könnte)mit unseren Mitteln und der bisher verwendeten Methode QuPPe Tier, nicht weiter verfolgt werden. Wir sind gespannt auf die fachliche Auswertung des PT´s, besonders im Hinblick auf erheblich geringere Konzentrationsbereiche, die eigentlich erreicht werden müssten.	G; C
1270	2.9	No	Es konnte nicht ermittelt werden, wo der Fehler lag. Die Stamm- und Arbeitsstandards waren i.O., Berechnungsfehler konnten auch nicht festgestellt werden. Die Quantifizierung erfolgte nicht über den Internen Standard: Glyphosat 13C2 15 N sondern über Perchlorat 18O4. Ohne Berücksichtigung des ISTD Perchlorat 18O4 beträgt der ermittelte Glyphosatgehalt 0,829 mg/kg. Die Bestimmung von Glyphosat in Matrix Leber ist in unserem Labor nicht validiert.	A; J
1266	4.3	No	We have no good explanation for our poor result concerning Glyphosate in liver. Best guess is "no expirience with the matrix-analyte combination". The sample extract has been measured with two different analysis-methods applying a Thermo Hypercarb (100x2.1 mm, 5 µm) and a Dionex IonPac AS11 (250x2 mm, 13 µm) with an appropiate pre column. Both methods yielded similar results. However, we will gather more expirience with the matrix-analyte combination by trying the QuPPe Version 10 method.	A; J
1406	9.5	Yes	Insufficient derivatization of isotopically labelled internal standards. Since the bovine liver samples contains a lot more proteins than our typical samples, the derivatization reagents added was not enough to react with all of the proteins and the isotopically labelled internal standards. As a result, the internal standards are under-derivatized, and that led to higher than actual calculated concentrations of Glyphosate and its metabolites	C; A; B

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

Glyphosate Assigned value: 0.535 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1306	13.4	Yes	The main reason of our unacceptable result of glyphosate in the EUPT-SRM14 bovine liver could be that we didn't follow the extraction method described for animal origin tissues. The specific method involved cryogenic milled and subsequent addition of EDTA, something that meant a deviation from our laboratory routine so this is why we decided to proceed with the plant origin QuPPe method. We already obtained satisfactory results in cow's milk with the routine methodology.	A; B

2,4-DB Assigned value: 0.061 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
956	-3.3 (FN)	Yes	The lab detected this compound below the lab reporting level. The concentration in the sample was less than the lowest confirmed calibration standard. The lab does not have validation data for this compound in liver	A; C
1290	-3.3 (FN)	(Yes)	"No signal was observed in the sample. We observed the signal of the matrix standard but not in the sample injected twice. Also the recoveries in matrix were not satisfactory (less than 30 %). General remark: we have no more time to study this kind of matrix that was decided at last moment. "	J; L
1150	-2.2	Yes	Poor results in EUPT-SRM14 were because we perform SRM only in food of plant origin - fruits and vegetables and we didn't validate these methods and parametres in animal matrices.	A; J
1302	2.6	Yes	With regard to our questionable results (2,4-DB, Bixafen desmethyl and MCPA) It was the first time we had ever analyzed these compounds and, apart from that, our equipments are quite old, so they don't have enough sensibility for the required levels	A; J; H

Avermectin B1a Assigned value: 0.058 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1318	-3.3 (FN)	Yes	We reported reporting limit 0.05 mg/kg for the avermectin B1 and it's same as LOQ of our method. We analyze abamectin routinely with the multiresidue method, which was used in the test too. Detection limit with the multiresidue method is relatively high. Reason for the false negative is that concentration level of abamectin in the liver sample was below LOQ of our method	J; L
1224	-2.2	Yes	This method used in routine is not a quantitative method	B
1406	-2.2	Yes	We used a dSPE clean up tube (MgSO4, PSA, C18). Pesticides in sample solution is reduced by clean up. After taking into account of Spike recovery, Z-Score are much better.	F

Bixafen Met. 6 Assigned value: 0.050 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1302	2.4	Yes	With regard to our questionable results (2,4-DB, Bixafen desmethyl and MCPA) It was the first time we had ever analyzed these compounds and, apart from that, our equipments are quite old, so they don't have enough sensibility for the required levels	A; J; H
1338	6.5	Yes	no experience on this compound ; the provided result comes from the diluted extract (1/5)), but the non-diluted extract gave a result (0.052 mg/kg) closer to the assigned value and would have given a satisfactory z-score. For this reason, we exclude a problem on the standard solution itself	A; C

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

A: Lack of Experience
B: Improper / unsuitable method
C: Matrix effect not properly compensated
D: Error in concentration of calibration
E: Incorrect calculation
F: Improper recovery correction
G: Interference in chromatogram
H: Technical problem
I: Transcription error
J: No validation
K: False compound analyzed
L: Reporting lebel too high or near assigned value

Boscalid Met. M510F017 Assigned value: 0.081mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1022	-3.5 (FN)	Yes	the poor performance of Boscalid Metabolite in the proficiency test EUPT-SRM14 (Bovine Liver) is a result of a wrong order of the substance. We have ordered Hydroxy-Boscalid by the firm HPC. Due to the result of the test we found out that the ordered substance is Metabolite M510F0 and not the required one. Now, we have ordered Boscalid Met. M510F017. The next step will be to include the analyte in our method and to measure the sample of the proficiency test again to identify the Boscalid Metabolite M510F017.	K

Bromoxynil Assigned value: 0.059 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1276	-3.3 (FN)	Yes	After investigating the possible causes we noted, that unfortunately there was an transcription error in the acquisition method (values for collision energy, EP, CXP and declustering potential were interchanged). After correcting the method and redoing the measurement, we obtained a result for Bromoxynil, that would have been acceptable (0.060 mg/kg).	I
1406	-3.0	Yes	We used a dSPE clean up tube (MgSO4, PSA, C18). Pesticides in sample solution is reduced by clean up. After taking into account of Spike recovery, Z-Score are much better.	F
1338	2.2	Yes	no experience on this compound/matrix combination ; the provided result comes from the diluted extract (1/5), but the non-diluted extract gave a result (0.062 mg/kg) closer to the assigned value and would have given a satisfactory z-score. For this reason, we exclude a problem on the standard solution itself. For future analyses of bromoxynil, we could use bromoxynil-D2 as ILIS	A; C

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

DDAC-C10 Assigned value: 0.177 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1150	-3.5	Yes	Poor results in EUPT-SRM14 were because we perform SRM only in food of plant origin - fruits and vegetables and we didn't validate these methods and parametres in animal matrices.	A; J
1092	-3.3 (FN)	Yes	1) Lack of sensitivity of equipment due to malfunction of nitrogen generator and 2) poor performance of the column	H
1406	-3.3 (FN)	Yes	Transcription error (It was the result for BAC-C12)	I
1398	2.1	No (but o.k.)	The QC samples were assessed at 0.01 and 0.05 mg/kg. At 0.01 mg/kg too high background was present and no data for recoveries were reported. For 0.05 mg/kg, recoveries of 105 % were achieved. Although not really necessary for levels >0.05 mg/kg, a second analysis was done using a pre-column which solved the background issue. In order to check for correct calibration solution preparation, a new solution prepared from the reference material was compared to the one used to quantify. The difference between them was 9 %. As the recoveries for the QC samples were within the acceptable range and also the solutions were comparable, the result was reported. Recovery, linearity, standards solutions, background were all OK. Calculations were re-checked and found to be OK. All in all, no cause for the deviating z-score could be found.	–
1214	3246	Yes	the error is due to a unit error. We entered the result in µg/kg when asked in mg/kg	I

Fenpropimorph carboxylic acid (BF-421-2) Assigned value: 0.088 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1340	-3.5 (FN)	Yes	Our failure was unintentional, since this particular substance is neither in our possession nor in our scope.	I

Flonicamid Met. TFNA-AM Assigned value: 0.073 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
956	-3.5 (FN)	Yes	Scope selection error. We analyse for TFNA and not TFNA-AM	K
1302	-3.5 (FN)	Yes	have analyzed Flonicamid instead of Flonicamid metabolite	K
1342	-3.5 (FN)	Yes	We looked for "TFNA" and not for "TFNA-AM	K
1368	-3.5 (FN)	Yes	The flonicamid metabolite TFNA-AM is not within routine scope, we do not look for this compound in our animal product work & therefore have no experience of this compound in bovine liver used in this proficiency round	K
1240	-2.1	No	As feedback, I can only say, that we have very short experience with the analysis of this metabolite, especially in animal tissue. That's why we don't have too much idea yet about the possible reason of the bias of our result, whether it's caused by poor efficiency of extraction, analyte degradation, calibration standard problem or some instrument-related trouble.	A
1356	-2.1	Yes	The turbo pump of LC-MS exploded and caused damages. After repair only a few days were available to analyse the PT sample. Although the results of tests of instrument were good we found that it did not work perfectly during measurement. There were problem with stability of ions and with sensitivity. We repeated the extraction of parallel test sample portions and applied standard addition, too. The recoveries were very low in almost all cases but for lack of ILIS we were not able to correct the results automatically. So we used recovery correction, and where we used it the Z-score were good. In case oTFNA, although we detected this residue in PT sample, we could not produce reliable recovery even after multiple attempts. Although we knew the result would be low, we did not use correction due to lack of knowledge of its rate."	F; A
1406	6.8	Yes	we used a dSPE clean up tube (MgSO4, PSA, C18). Pesticides in sample solution is reduced by clean up. After taking into account of Spike recovery, Z-Score are much better.	F

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

A: Lack of Experience
B: Improper / unsuitable method
C: Matrix effect not properly compensated
D: Error in concentration of calibration
E: Incorrect calculation
F: Improper recovery correction
G: Interference in chromatogram
H: Technical problem
I: Transcription error
J: No validation
K: False compound analyzed
L: Reporting lebel too high or near assigned value

Fluopyram-benzamide (M25) Assigned value: 0.101 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1400	-3.2 (FN)	Yes	The OOS-procedure O-19-0023 showed that we have Fluopyram-benzamide (M25) new in our scope, but unfortunately the peak was overlooked. The routine procedure for new pesticides was not fulfilled. The recalculation of the peak showed a result of 0,123 mg/kg (would be a Z-Score value about 0,8). As corrective action we revise the procedure for establishing new pesticides.	A; B
1406	7.6	Yes	we used a dSPE clean up tube (MgSO4, PSA, C18). Pesticides in sample solution is reduced by clean up. After taking into account of Spike recovery, Z-Score are much better.	F

MPP Assigned value: 0.309 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1398	2.3	No	"The QC samples were assessed at 0.05 and 0.1 mg/kg resulting in 110 and 92 % recovery, respectively. The correctness of solution preparation was also checked presented a difference of less than 1 %. As isotopically labelled internal standard were used and taking into consideration the results obtained for the QC samples, the results was reported. Recovery, linearity, standards solutions were all OK. Calculations were re-checked and found to be OK. All in all, no cause for the deviating z-score could be found."	–
1092	4.4	Yes	1) Bad precision of equipment due to lack of sensitivity - malfunction of nitrogen generator; 2) Test amount not enough to perform standard addition to sample portions for all requested methods	H; C

AMPA Assigned value: 0.754 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1092	-3.5 (FN)	Yes	1) Lack of sensitivity of equipment due to malfunction of nitrogen generator and 2) poor performance of the column	H
1228	-2.4	(Yes)	It is a new matrix that we have never done before. An internal standard like AMPA C13N15 could be added to reduce the matrix effect.	A; J; C
1224	2.4	Yes	this method is not validated for liver, and we didn't used the labelled AMPA as internal standard (too expensive), but now we bought it.	A; C
1406	12.5	Yes	to insufficient derivatization of isotopically labelled internal standards. Since the bovine liver samples contains a lot more proteins than our typical samples, the derivatization reagents added was not enough to react with all of the proteins and the isotopically labelled internal standards. As a result, the internal standards are under-derivatized, and that led to higher than actual calculated concentrations of Glyphosate and its metabolites	C; A; B

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

N-Acetyl glyphosate Assigned value: 0.543 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
956	-3,7	Yes	a reporting error. This result was reported as 0.0438mg/kg. The result obtained in-house was 0.438mg/kg which would result in an acceptable z-score.	I
1320	9.1	Yes	We have detected that our standard of N-acetyl-glyphosat was not ok when we quantified the test with it. This substance was not in our routine control program and that is why its conditions was not under control. We have bought a new standard solid and a new liquid has been prepared. Differences in their responses have been detected. Quantifying the EURL-SRM 14 test that we had with the new standard, the results are quite similar to the assigned value. So we conclude that we can now quantify propertly the n-acetyl-Glyphosate	A; D
1358	9.9	Yes	I would like to kindly inform you that the cause that determined this result (Conc. = 2.01 mg/kg and z-score = 9,9) has to be attributed to the use of a standard stock solution expired in 2018, and, because the analytical standard was also finished, the standard test stability could not have been performed. In addition, at the moment of EUPT-SRM14 arrival, our laboratory was unable to buy a new analytical standard in due time (also because the latter was not commercially available in that period by our provider) | Moreover, I would like to give you notice that the analytical determination of this compound has been carried out even if the analytical standard was expired, because, at the moment of EUPT invitation, this determination was not included among the analytes under mandatory assessment for our laboratory and also because we have taken this opportunity as interlaboratory exercise, providing you with the result for informative purpose only.	D

Haloxyfop Assigned value: 0.037 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1406	-3.4	Yes	we used a dSPE clean up tube (MgSO4, PSA, C18). Pesticides in sample solution is reduced by clean up. After taking into account of Spike recovery, Z-Score are much better.	F
1324	-2.9 (FN)	Yes	limit of detection is much higher than the assigned value	J; L
1092	-2.7	Yes	1) Quantification performed by using haloxyfop and haloxofop-P in same calibration solution; 2) Lack of training	D
1228	2.2	(Yes)	It is a new matrix that we have never done before.	A; J

MCPA Assigned value: 0.046 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
1406	-3.1 (FN)	Yes	we used a dSPE clean up tube (MgSO4, PSA, C18). Pesticides in sample solution is reduced by clean up. No recovery of spike	F
1356	-2.9	Yes	The turbo pump of LC-MS exploded and caused damages. After repair only a few days were available to analyse the PT sample. Although the results of tests of instrument were good we found that it did not work perfectly during measurement. There were problem with stability of ions and with sensitivity. We repeated the extraction of parallel test sample portions and applied standard addition, too. The recoveries were very low in almost all cases but for lack of ILIS we were not able to correct the results automatically. So we used recovery correction, and where we used it the Z-score were good. In case of MCPA, the recovery at 2nd extraction series was 70 %. Therefore we accepted the result of this serie and gave it without correction.	F
1290	-2.3	(Yes)	For MCPA we obtained very high recoveries more than 300 %, so we decided to correct the results by recovery. We observed a very high matrix effect. General remark: we have no more time to study this kind of matrix that was decided at last moment.	A; C; F
1300	2.2	Yes	There was a mistake in the calculations of concentration of the standard solution of MCPA, that possibly is the cause of this deviation	E
1228	2.8	(Yes)	It is a new matrix that we have never done before.	A; J
1302	2.8	Yes	With regard to our questionable results (2,4-DB, Bixafen desmethyl and MCPA) It was the first time we had ever analyzed these compounds and, apart from that, our equipments are quite old, so they don't have enough sensibility for the required levels	A; J; H

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

A: Lack of Experience
B: Improper / unsuitable method
C: Matrix effect not properly compensated
D: Error in concentration of calibration
E: Incorrect calculation
F: Improper recovery correction
G: Interference in chromatogram
H: Technical problem
I: Transcription error
J: No validation
K: False compound analyzed
L: Reporting lebel too high or near assigned value

False Positive Results

Analyte	LabCode	Reason / Remarks	
BAC-C12	1406	Transcription error (It was the result for DDAC-C10)	I
Dichlorprop	1338	FP: due to a human error when building the MS acquisition method: the transitions for 2,4-DB (present in the test item) have been copied-pasted instead of those of 2,4-DP (dichlorprop)	I
Glufosinate	1356	Standards, two parallel test sample portion and fortified control test sample and control sample of laboratory with its fortified version were extracted and derivatized side by side. TPP as IS was added to final extract. The RT of TPP was same in all cases (no shifts). The RT of glufosinate in standards and 2 parallel test portion was same (13.847 min). Only a small shift was observed at 2 fortified sample with differet ΔRT: 13.821 (at fortified control sample of laboratory) and 13.873 (at fortified control test sample). At this moment the column was new, after conditions only some system suitability test solution were injected. Later we checked the MPP at this PFPD condition but its RT was 7.0 min. So, there was no possibility to identify MPP as glufosinate (as false). Unfortunatelly, our test sample sold out so there was no possibility to analyse again and to find the cause of false results.	–
Glufosinate	1406	insufficient derivatization of isotopically labelled internal standards. Since the bovine liver samples contains a lot more proteins than our typical samples, the derivatization reagents added was not enough to react with all of the proteins and the isotopically labelled internal standards. As a result, the internal standards are under-derivatized, and that led to higher than actual calculated concentrations of Glyphosate and its metabolites.	C; A; B

GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues in Food and Feed

Introduction

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

The following four EURLs for pesticide residues were appointed by DG-SANTE based on regulation (EC) 625/2017[3]:

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

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

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

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

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

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

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

EUPT-Organisers and Scientific Committee

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

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

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

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

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

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

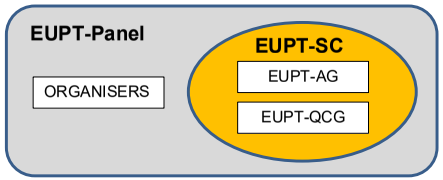
The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and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

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

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

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

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



The decisions of the EUPT-Panel will be documented.

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

EUPT Participants

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

- Art. 28 of Reg. 396/2005/EC[6] (for all OfLs analysing for pesticide residues within the framework of official controls[7] of food or feed)
- Art. 101 (1)(a) of Reg. (EC) 625/2017 (for all NRLs)

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

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

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

EURL EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

8th Edition: Revised 23th January, 2018

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

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

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

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

Confidentiality and Communication

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

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

As laid down in Regulation 625/2017, NRLs are responsible for supporting and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.

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

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

www.eurl-pesticides.eu

The official language used in all EUPTs is English.

Announcement / Invitation Letter

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

Target Pesticide List

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

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

Specific Protocol

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

Homogeneity of the Test Item

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

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

results, the analytical difficulties faced during the test, knowledge of the analytical behaviour of the pesticide question) may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report.

Stability of the analytes contained in the Test Item

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

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

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

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

Considering knowledge about the expected susceptibility of pesticides in the Test Item to possible losses, the Organisers will choose the shipment conditions to be such that pesticide losses are minimised (e.g. shipment of frozen samples, addition of dry ice). As shipment time can differ

between labs/countries it is recommended that the Organisers conduct additional stability tests at conditions simulating shipment. Should critical losses be detected for certain pesticides the EUPT-SC will be informed (or the EUPT-QCG before or during the test). Case-by-case decisions may be taken considering all relevant aspects including the shipment time of the samples to each laboratory.

Methodologies to be used by the participants

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

General procedures for reporting results

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

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

Correction of results for recovery

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

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

EURL EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

8th Edition: Revised 23th January, 2018

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

Methodology information

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

Results evaluation

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

- ***False Positive results***

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

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

– ***False Negative results***

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

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

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

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

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

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

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

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

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

Uncertainty of the assigned value

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

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

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

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

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

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

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

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

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

– ***z scores***

This parameter is calculated using the following formula:

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

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

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

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

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

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

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

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

EURL EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

8th Edition: Revised 23th January, 2018

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

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

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

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

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

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

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

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

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

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

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

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

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

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

EURL EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

8th Edition: Revised 23th January, 2018

Publication of results

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

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

Certificates of participation

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

Feedback

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

Correction of errors

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

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

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

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

Follow-up activities

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

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

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

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

Phase 1:

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

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

EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

8th Edition: Revised 23th January, 2018

- Actions: On the spot visits and training if necessary and repetition of the comparative test if feasible and close the assessment of results by the EURL.

Phase 2:

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

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

Disclaimer

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

SPECIFIC PROTOCOL

for the 14th EU Proficiency Test
on Pesticides requiring Single Residue Methods
EUPT – SRM14 (2019)
(update on 9 April 2019)

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. 8**" covering all EUPTs in 2019.
The EUPT-SRM14 is organised by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL–SRM) in cooperation with EU Reference Laboratory for residues of pesticides in food of animal origin and commodities with high fat content (EURL-AO). Both EURLs are accredited according to ISO 17043 as providers of proficiency tests (Please see **EURL-SRM accreditation** and **EURL-AO accreditation**).
The EUPT-SRM14 deals with the analysis of SRM-pesticides in bovine liver and is to be performed by all National Reference Laboratories for Single Residue Methods (NRL-SRMs) as well as by all official EU laboratories (OfLs) involved in official pesticide residue controls, as far as their scope overlaps with that of the EUPT-SRM14. A special **EUPT-SRM14-Website** containing links to the most important documents of relevance was constructed.

A preliminary classification of laboratories into obliged and non-obliged to participate in the present PT was prepared based on information within the EURL DataPool. NRL-SRMs and OfLs performing pesticide residue analyses in food of animal origin within the frame of National and EU official controls were considered as tentatively obliged to participate in this PT. The laboratories were asked to update this information prior to the EUPT-registration period. This tentative classification was only based on the commodity scope (not the pesticide scope) of the laboratories and was also visible to the participants within the registration page. OfLs listed as "obliged to participate in the EUPT-SRM14" but not intending to participate had to state their reasons for non-participation during the online registration of the EUPT-SRM14, which lasted from 18 January till 8 February, 2019. The feedback received during registration, especially details considering the scope, will be considered in the final list of obliged laboratories.

Test Item and Blank Material

The Test Item of this EUPT is **Bovine Liver**.

Participants will receive two bottles containing:

1) ~ 200 g **Test Item containing spiked analytes** from the **Target Pesticides List**. This **Test Item** will be prepared by cryogenic milling and will be shipped in a snow-like condition within a 500ml plastic jar [1].

2) ~ 200 g **Blank Material**, that can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards. This **Blank Material** will be shipped as an ice block within a 300ml plastic jar [1].

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

Target Analytes and MRRLs

The Test Item will contain several pesticides from the **EUPT-SRM14 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-SRM14 Target Pesticides List before starting with analysis and result reporting**.

Shipment of Test Item

Test item and Blank Material are planned to be shipped on 18 March, 2019.
Frozen Test Item and Blank Material will be packed in thermo-boxes together with dry ice[1] and shipped from Germany via DHL express parcel to the participants. Prior to shipment a reminder will be sent to the participating laboratories by e-mail.
The participating laboratories must make their own arrangements for the receipt of the package. They should inform the Organisers of any public holidays in their country/city during the week of the shipment, and must make the necessary arrangements to receive the shipment, even if the laboratory is closed.

Should any complications during shipment, delivery or the customs be expected, the participating laboratories should provide the Organizers in advance with contact information of possible contact persons of the lab (e.g. mobile phone numbers) as well as instructions in local language explaining the need to keep the package in freezer during delay in transit and delivery. This information will be attached to the package.

After the packages will be 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.

Once unexpected delay is noticed in the customs or within the recipient's country, the participant himself is strongly encouraged to contact his local DHL Express office and/or the customs in order to accelerate the clearance and delivery procedures.

Instructions on handling the Test Item

Once arrived, both the Test Item and the Blank Material should be stored deeply frozen (at -18°C or lower) until analysis in order to avoid any possible deterioration/spoilage of the sample and to minimize pesticide degradation.

The **Test Item** was homogenized via cryogenic milling. It should normally arrive in a snow-like condition so that it can be easily loosened up with a spatula and the analytical portions can be conveniently withdrawn. Any coagulations of the

[1] PT-Material for participating laboratories in countries or locations where the shipment with dry ice is not allowed will be packed in thermo-isolated jars, pre-cooled at -70°C and shipped in thermo-boxes additionally containing freeze elements.

Appendix 9. Specific Protocol of EUPT-SRM14

homogenate are not expected to have any impact on the homogeneity, so in principle there is no need of re-mixing the Test Item in its entirety.

The **Blank Material** is provided as an ice block. Prior to taking analytical portions, it is recommended leaving the Blank Material at ambient temperature for approx. 1 hour to partly defrost followed by a mixing with a knife mill or leaving the material to almost entirely defrost (e.g. 2.5 – 3 hours at ambient temperature) followed by stirring with a spatula while still cold.

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-SRM14 result submission webtool.

The homogeneity tests will be conducted using 10 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.

Results submission webtool

Sample receipt acknowledgement, analytical results and method information are to be submitted via the EUPT-SRM14 result submission webtool:

- **Sample receipt acknowledgement: accessible from 19 March till 28 March, 2019.**
- **Analytical results and method information: accessible from 19 March till 15 April midnight (CEST), 2019.**
- **The deadline for result submission is 23 April 15 h (3 p.m.) (CEST), 2019.**
- **Additional information on the methods used for tentatively false negative results: accessible from 24 April till 2 May, 2019.**

A guideline for the new EUPT-SRM14 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-SRM14 result submission webtool, participants must use their personal login credentials (username and password). The link to the EUPT-SRM14 result submission webtool and the personal login credentials will be provided to the PT-contact persons approx. two weeks prior to sample shipment.

The lab's unique lab code for the EUPT-SRM14 will be provided to the participants after accessing to EUPT-SRM14 result submission webtool for the first time.

- Acknowledgement of Package Receipt and Acceptance of PT-Materials

Once the laboratory has received the package, it must report to the organiser via the EUPT-SRM14 result submission webtool the date of receipt, the condition of the Test Item, and its acceptance. The page Sample Acknowledgement will remain open till 28 March. If a laboratory does not respond by this deadline, the Organisers will assume that Test Item and Blank Material have been received and accepted.

Any participants having not received the Test Items by the Fri. 22 March at noon must inform the Organiser via e-mail (EURL-SRM@cvuas.bwl.de) by Fri. 22 March 2:30 pm. The Organiser will consult the shipping company to localize the package and decide on further actions including new shipment, if necessary.

- Reporting qualitative and quantitative Results

To report their results, laboratories must access the EUPT-SRM14 result submission webtool.
All results must be reported on this website by 23 April 15 h (3 p.m.) (CEST), 2019. 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-SRM14 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 considered as „Not Detected".

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

  Recovery-corrected results should be reported only where this reflects the routine lab's procedure; otherwise the non-recovery-corrected result should be reported. Where a **result was corrected for recovery,** the approach(es) followed to achieve this correction (e.g. standard additions to sample portions, procedural calibration, recovery 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-SRM14 Target Pesticides List determined in the Blank Material (even at levels below the MRRL).

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

- Reporting Information on Analytical Methodology

On the page of "**Edit methods"** of EUPT-SRM14 result submission webtool the participating laboratories must provide information on the analytical method(s) applied to pesticides which were analysed and detected either in the Test Item or in the Blank Material.

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

For detailed information on the columns on the page of "**Edit methods" please refer to the guideline for results submission that will be issued and provided to you in due time**.

- Submission of results

Once you have entered all your results and checked their correctness, you have to submit them by clicking the bottom "Final submission" 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 for entering the method information for the pesticide(s) in question after the results submission period is closed.

Subcontracting

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

a) Generation of the login credentials
b) Administration of EUPT-SRM14 result submission website

Follow-up actions

After the distribution of the EUPT-SRM14 Preliminary Report, laboratories with poor results (high absolute z-scores, false negatives or false positives) will be asked to provide information concerning the reasons for this and possible corrective actions. This information will be forwarded to the corresponding NRL-SRMs upon request. All EUPT-SRM14-participants are welcome to ask the EURL-SRM for technical assistance.

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

According to instructions by DG-SANTE, the "**Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with Community reference laboratories (CRLs) activities**" will be followed by NRLs.

Documents

All documents related to the EUPT–SRM14 can be found in the **EURL-Document Repository (CIRCA-BC)**. Links to the documents can also be found in the **EUPT-SRM14 Website**.
For further information please contact the organizers **EURL-SRM@cvuas.bwl.de**
Please check the **EUPT-SRM14 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: 250 €
- Labs based in third countries: 400 €

An invoice issued to the "invoice address" stated in the registration form will be sent two weeks 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 of payment will be 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 organiser.

If for any reason payment cannot be carried out before this date, please contact the Organizer to give explanations.

If no payment or no proof of payment is received and no explanation is given to the Organizers, the Organizers reserve the right to exclude the results of the concerned laboratories from the Final EUPT-Report or to refuse its participation in future EUPT-SRMs.

Bank Details:

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

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 participated in both the EUPT-SRM14 and the EUPT-AO14, 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 the invoice. Without this text, your payment will not be able to reach the correct EURL.

More details for bank-remittance are given in the invoices.

Calendar of EUPT-SRM14

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_Calendar.pdf)

Target Pesticides List of EUPT-SRM14

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_TargetPesticideList.pdf)

Supplementary Information on EUPT-SRM14 Analytes

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_Suppl_Info.xls)

Contact information

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Quality Control Group

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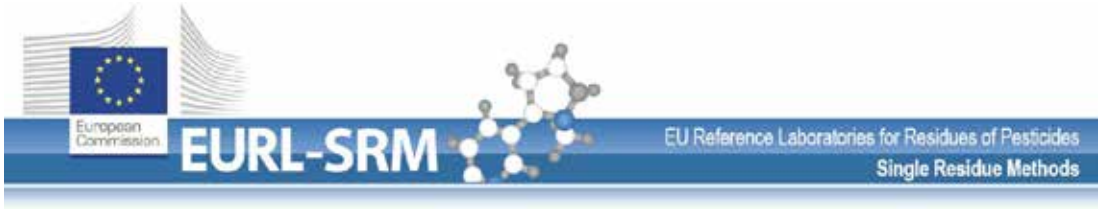
Paula Medina	European Food Safety Authority (EFSA)

Appendix 10 Calendar and Target Pesticides List of EUPT-SRM14



CALENDAR for the EUPT – SRM14

Bovine Liver Homogenate

(update on 9/4/2019)

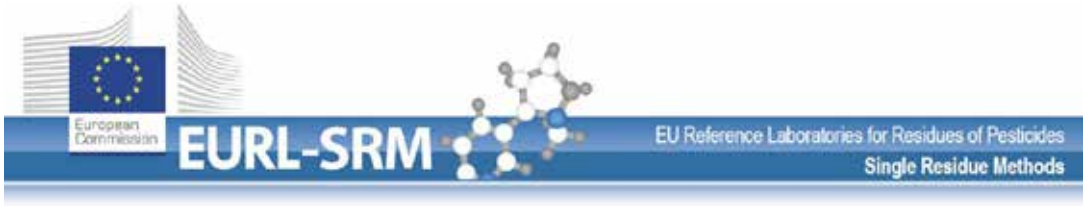
Activity	Who?	Dates
Opening of the **EUPT-SRM14 Website** with Advance Information links to all relevant documents (**Calendar**, **General Protocol**, **Target Pesticides List**)	EURL-SRM	16 Nov. 2018 (Advance Information) 18 Dec. 2018 (Announcement)
Publishing of new methods for SRM-Analytes in matrices of animal origin (**QuPPe-AO-V3**, **Analytical Observations Report on Various Metabolites using QuEChERS**)	EURL-SRM	Till End of Jan. 2019
Registration via "**EUPT-Registration Website**" (Note: Obliged Laboratories MUST enter this Website and either register or give **explanations for non-participation**)	**All Laboratories interested in participation and all obliged labs even if not interested**	**18 Jan. – 8 Feb. 2019**
Dispatch of: • **EUPT-SRM14-Specific Protocol** • **Links to EUPT-SRM14 result submission webtool** • **Personal login credentials**	EURL-SRM	~ 4 March 2019
Preparation of EUPT-SRM14-Test Item (preliminary tests Spiking / Homogenization)	EURL-SRM	Nov. 2018 – March 2019
Homogeneity Tests	EURL-SRM	March 2019
Stability Tests	EURL-SRM	March – May 2019
Shipment of EUPT-SRM14 Test Item (+reminder of upcoming parcel arrival)	EURL-SRM	**18 March 2019**
Confirmation of sample Receipt and acceptance via "**EUPT-SRM14 result submission webtool**"	**Participating Labs**	**19 March – 28 March 2019**
Result Submission (Pesticide scope, Results, Method Info) in "**EUPT-SRM14 result submission webtool**"	**Participating Labs**	**19 March – 23 April 2019**
Additional Information (Method info on tentatively false negative results) in "**EUPT-SRM14 result submission webtool**"	**Participating Labs**	**24 April – 2 May 2019**
Preliminary Report (only compilation of results and preliminary assigned values)	EURL-SRM	May 2019
Survey to collect reasons for underperformance and missing information on methods	EURL-SRM / **Participating Labs**	April / May 2019
EUPT Evaluation Meeting	EUPT-SC, DG-SANTE	19 – 21 June 2019
Final Report	EURL-SRM	Dec. 2019

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@cvuas.bwl.de**

The EUPT-SRM Team

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

Appendix 10 (cont.) Calendar and Target Pesticides List of EUPT-SRM14



TARGET PESTICIDE LIST

for the EUPT–SRM14 2019, Bovine Liver Homogenate

(update on 31.01.2019)

There will be no Cat. A/B classification in EUPT-SRM14, in the order of analytical group

Compounds Potentially Present in Test Item		Listed in	MRRL (mg/kg)
GROUP 1: Compounds with **ca. 75% probability** of being present in test-item (7-8 out of 10 compounds present).	**Boscalid metabolite M510F01** (free phenol, no hydrolysis step to be applied)	WD for NCPs	0.01
	Chlormequat (expressed as chloride salt)	WD for NCPs	0.01
	Bixafen metabolite, desmethyl bixafen	WD for NCPs	0.01
	Fenpropimorph carboxylic acid (BF-421-2)	WD for NCPs	0.01
	Fluopyram-benzamide (M25)	WD for NCPs	0.02
	Glyphosate	MACP-Reg.	0.1
	AMPA (metabolite of glyphosate)	WD for NCPs	0.1
	N-Acetyl-glyphosate (metabolite of glyphosate)	WD for NCPs	0.1
	Isoxaflutole diketonitrile metabolite (RPA202248)	WD for NCPs	0.01
	Mepiquat (expressed as chloride salt)	WD for NCPs	0.01
GROUP 2: Compounds with **ca. 50% probability** of being present in test item (6-8 out of 14 compounds present).	**2,4-D** (free acid, no hydrolysis step to be applied)		0.01
	2,4-DB (free acid, no hydrolysis step to be applied)	WD for NCPs	0.01
	Avermectin B1a		0.01
	BAC-C12 (benzyldimethyldodecylammonium chloride; expressed as chloride salt)	WD for NCPs	0.03
	Chlorate (anion)	WD for NCPs	0.01
	DDAC-C10 (didecyldimethylammonium chloride; expressed as chloride salt)	WD for NCPs	0.03
	Dichlorprop incl. dichlorprop-P (=2,4-DP; free acid, no hydrolysis step to be applied)	WD for NCPs	0.01
	Emamectin B1a		0.01
	Ethephon		0.05
	Flonicamid metabolite TFNA-AM		0.01
	Fluazifop incl. fluazifop-P (free acid, no hydrolysis step to be applied)	WD for NCPs	0.01
	Haloxyfop, incl. Haloxyfop-P (free acid, no hydrolysis step to be applied)	WD for NCPs	0.01
	MCPA (free acid, no hydrolysis step to be applied)	WD for NCPs	0.01
	MCPB (free acid, no hydrolysis step to be applied)	WD for NCPs	0.01
GROUP 3: Compounds with **ca. 25% probability** of being present in test item (1-3 out of 8 compounds present).	**5-Hydroxythiabendazole**		0.01
	Bromoxynil		0.01
	Dicamba		0.05
	Glufosinate	WD for NCPs	0.1
	MPP (metabolite of glufosinate)	WD for NCPs	0.05
	N-Acetyl-glufosinate (metabolite of glufosinate)	WD for NCPs	0.05
	Quizalofop, incl. quizalofop-P (free acid, no hydrolysis step to be applied)	WD for NCPs	0.01
	Triclopyr (free acid)		0.01

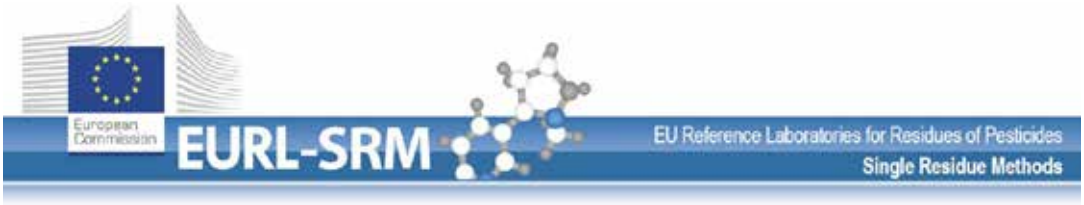
MACP-Reg.: REGULATION (EU) 2018/555 of 9 April 2018
NCP-WD: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 26–27 November 2018rev. 10(3)

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

For any further clarification don't hesitate to contact us under **eurl-srm@cvuas.bwl.de**
The EUPT-SRM14 Organising Team

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

Appendix 10 (cont.) Calendar and Target Pesticides List of EUPT-SRM14



TARGET PESTICIDE LIST

for the EUPT–SRM14 2019, Bovine Liver Homogenate

(update on 12.03.2019)

There will be no Cat. A/B classification in EUPT-SRM14, alphabetically sorted

Compounds Potentially Present in Test Item	GROUP*	Listed in	MRRL (mg/kg)
Glyphosate	1	MACP-Reg.	0.1
2,4-D (free acid, no hydrolysis step to be applied)	2		0.01
2,4-DB (free acid, no hydrolysis step to be applied)	2	WD for NCPs	0.01
5-Hydroxythiabendazole	3		0.01
Avermectin B1a	2		0.01
BAC-C12 (benzyldimethyldodecylammonium chloride; expressed as chloride salt)	2	WD for NCPs	0.03
Bixafen metabolite, desmethyl bixafen	1	WD for NCPs	0.01
Boscalid metabolite M510F01 (free phenol, no hydrolysis step to be applied)	1	WD for NCPs	0.01
Bromoxynil	3		0.01
Chlorate (anion)	2	WD for NCPs	0.01
Chlormequat (expressed as chloride salt)	1	WD for NCPs	0.01
DDAC-C10 (didecyldimethylammonium chloride; expressed as chloride salt)	2	WD for NCPs	0.03
Dicamba	3		0.05
Dichlorprop incl. dichlorprop-P (=2,4-DP; free acid, no hydrolysis step to be applied)	2	WD for NCPs	0.01
Emamectin B1a	2		0.01
Ethephon	2		0.05
Fenpropimorph carboxylic acid (BF-421-2)	1	WD for NCPs	0.01
Flonicamid metabolite TFNA-AM	2		0.01
Fluazifop incl. fluazifop-P (free acid, no hydrolysis step to be applied)	2	WD for NCPs	0.01
Fluopyram-benzamide (M25)	1	WD for NCPs	0.02
Glufosinate	3	WD for NCPs	0.1
Glufosinate metabolite (MPP)	3	WD for NCPs	0.05
Glufosinate metabolite (N-Acetyl-glufosinate)	3	WD for NCPs	0.05
Glyphosate metabolite (AMPA)	1	WD for NCPs	0.1
Glyphosate metabolite (N-Acetyl-glyphosate)	1	WD for NCPs	0.1
Haloxyfop, incl. Haloxyfop-P (free acid, no hydrolysis step to be applied)	2	WD for NCPs	0.01
Isoxaflutole diketonitrile metabolite (RPA202248)	1	WD for NCPs	0.01
MCPA (free acid, no hydrolysis step to be applied)	2	WD for NCPs	0.01
MCPB (free acid, no hydrolysis step to be applied)	2	WD for NCPs	0.01
Mepiquat (expressed as chloride salt)	1	WD for NCPs	0.01
Quizalofop, incl. quizalofop-P (free acid, no hydrolysis step to be applied)	3	WD for NCPs	0.01
Triclopyr (free acid)	3		0.01

MACP-Reg.: REGULATION (EU) 2018/555 of 9 April 2018
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)

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

GROUP*: Please see the definition of analytical group in the table on the left side.

For any further clarification don't hesitate to contact us under **eurl-srm@cvuas.bwl.de**
The EUPT-SRM14 Organising Team

Appendix 11 Call for Registration

Betreff: Call for Registration: EUPT-SRM14 (Bovine Liver)
Von: CVUA-S EURL-SRM Pesticides <EURL-SRM@cvuas.bwl.de>
Datum: 21.01.2019, 14:32
An: CVUA-S EURL-SRM Pesticides <EURL-SRM@cvuas.bwl.de>

Dear Colleagues,

the EUPT-SRM14 using Bovine Liver as matrix is now open for registration.

- **For registration, please access the registration page: www.eupt-registration.eu and login using your EURL DataPool login credentials.**
- **This registration page is accessible till 8 February, 2019 at the latest.**
- **Instructions on how to complete the registration form** can be found in the attached pdf-files and on the registration page. There is separate instructions for labs participating on mandatory and labs participating on voluntary basis.

Based on the data stored within the OfL-Network Database concerning commodity scope and lab status (e.g. OfL, NRL) and a checkup by the respective NRLs **all labs were classified as tentatively obliged or non-obliged to participate in the EUPT-SRM14** (obliged = NRL-SRMs and OfLs analyzing food of animal origin). This classification has been uploaded within the EUPT-Registration page. **If your lab is obliged to participate in this PT but does not intend to participate, you still have to enter the registration form and choose "No" under "I want to REGISTER my lab for this EUPT" and provide an explanation for your non-participation** (requirement by DG-SANTE). **If you think that there you lab's EUPT-SRM14 CLASSIFICATION IS ERRORNEOUS please contact your NRL and the eurl-srm@cvuas.bwl.de.**

Please note:

1. The **Target Pesticides List for EUPT-SRM14** can be found via the following link: http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_TargetPesticideList.pdf).
   Supplementary information on the EUPT-SRM14 compounds can be found here: http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_Suppl_Info.xls (e.g. exemplary providers of analytical standards, exemplary methodologies)

2. The **lab name for shipment is limited to 60 letters** (e.g.: instead of "Chemical and Veterinary Investigations Office, Stuttgart (CVUAS), Dept. Residues" the short acronyms "CVUA Stuttgart, Abt. RK" is preferred.)
   It may be written in your local language phonetics, but LATIN characters MUST be used (please do not use language-specific letters, e.g. Greek or Cyrillic, as this may cause problems for preparing waybills).

3. The "**City**" in the sample delivery address MUST be written **in English characters** !

4. **During the registration period you and any member of your laboratory can change any of your laboratory's entries in the registration page as often as you like.** Following any change of your registration data, you will receive a new email confirming registration for participation / non-participation.

5. The link and **your login credentials for the data submission website** to the sample acknowledgement and results submission pages will be sent to you later.

6. **Labs are free to use any methods with preference to the methods used in routine.** Nevertheless, the following methods, published by the EURL-SRM, are put to your attention as they may be helpful to you
   - QuPPe-AO-V3: http://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=200&CntID=1112&Theme_ID=1&Pdf=False&Lang=EN
   - Analytical Observations Report on Various Metabolites using QuEChERS: http://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EurlSrm_Observation_QuEChERS-AO_V1.pdf

For **further information** on the EUPT-SRM14 please visit the EUTP-SRM14 Website: http://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=200&CntID=1100&Theme_ID=1&Pdf=False&Lang=EN

The **Specific Protocol** for the EUPT-SRM14 will be dispatched two weeks prior to the shipment.

If there is any questions, please contact us EURL-SRM@cvuas.bwl.de

Best regards,

The EURL-SRM Team

Guide to EUPT-SRM14 Results Submission Webtool

Version: 2019-01, Date: 9-4-2019, Init: Schr (changing of submission deadline and period for additional information)

Please read this guideline carefully so you are familiar with the webtool before you start entering your data.

MAJOR CHANGES COMPARED TO THE FORMER WEBTOOL:

- **You cannot use Internet explorer.** The Webtool is only validated for **Chrome** and **Firefox** browsers.
- **Your data is automatically saved as soon as you move from one edited cell to another.** Therefore, almost all pages and tables do not have any save button.
- You can access to 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 are NOT able to change your entries anymore!**

Browser requirements: The system works only with the following browsers

Browser	Oldest supported version*
Google Chrome	44.0
Firefox	39.0

* latest version is recommended

Please don't use Internet Explorer!

Guide to EUPT-SRM Results Submission Webtool

Getting start

Link to webtool: www.eurl.dtu.dk

Choose Guest and others

Log in to the Webtool by using your **personal username and password** sent to you by email in connection to any of the EUPTs on pesticides (EUPT-CF, -FV, AO or SRM).
Don't change your password or request for a new one before we have given a notice on this.

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

Appendix 12. Guide to EUPT-SRM14 Results Submission Webtool

Proficiency Test Overview

On the ***Proficiency Test Overview* page**, 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) concerning the currently active EUPT as well as EUPTs in which your lab has participated in the past.

By clicking on EUPT-SRM14 under "*My proficiency tests"*, you will be able to view (but not edit) the **current scope for this EUPT**. The mandatory compounds are listed first, in alphabetic order, followed by the voluntary compounds, also in alphabetic order.

In contrast to other EUPTs organized by EURL-CF, -FV and -AO**, no compound selection before sample shipment is requested for the EUPT-SRM14. You will be able to edit your targeted compounds only after the Webtool opens for results submission.**

Sample reciept and acceptance

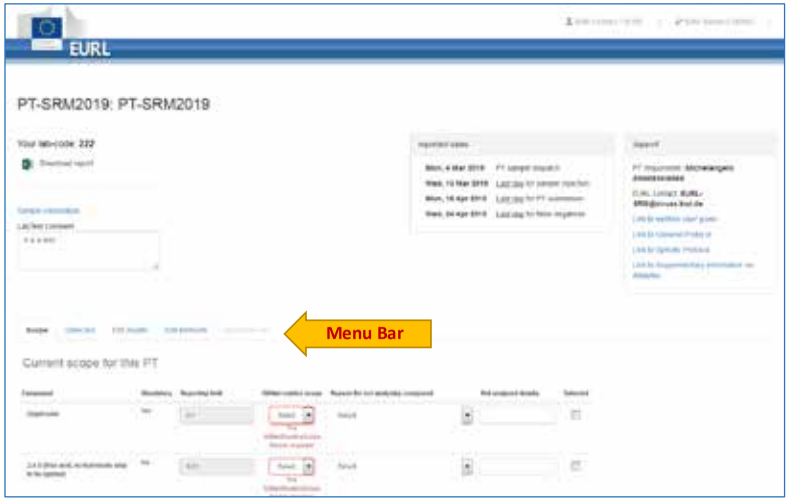
The **Webtool for the EUPT-SRM14 result submission** will expectedly open on **19 March**.

Once you have received the parcel with the PT-materials, please click on EUPT-SRM14 under *My proficiency tests* to open the pop-up window "**Edit sample Receipt"**. Please fill in the information requested within this po-up-window:

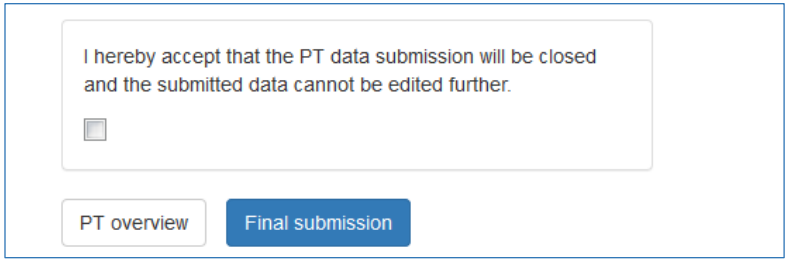
- Sample Number (bottle number of the Test Item you received)
- Blank Sample Number (bottle number of the Blank Material you received)
- **Material Accepted** (based on condition upon receipt) **If the PT-materials are not accepted please additionally contact the PT-Organizers via E-mail.**
- Sample received (enter the date of receipt of the sample)
- Remarks:
  Please state **whether dry ice was still contained in the box upon arrival and describe the condition of the Test Item, when the bottle was first opened (e.g. frozen/partly defrosted/defrosted, if possible measure and indicate the temperature). In case of delayed opening of bottles, please provide information on storage conditions and whereabouts of the parcel or bottles until bottle opening**.

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 28 March**. This page will be shown at the very beginning only. Once you have clicked "*Save and Close*" this page will not be shown any more. Therefore, please enter all remarks concerning your samples or parcel that you want to pass to the organizer at this step.

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 comments** you want to pass to the organizer in relation to this particular PT. On the right side of the page, you find **important dates** and **links**. At the bottom of the page, 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 when you have already entered all your data for this PT and want to submit them for the PT evaluation.
Afterwards you are not able to change your data any more.

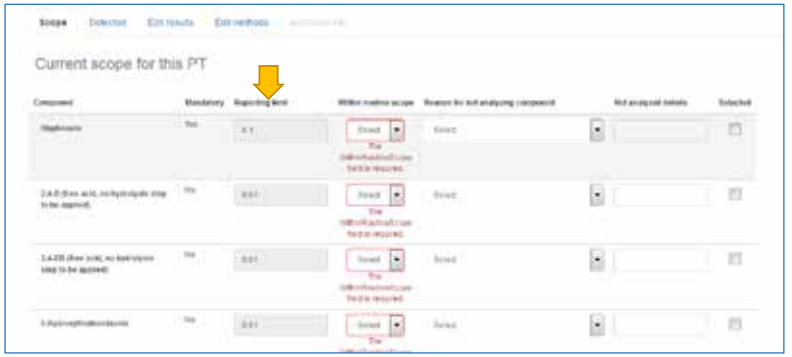


Guide to EUPT-SRM Results Submission Webtool

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.

"Selected" on this table corresponds to analyzed and is used as a filter for the table "detected".



On this table please firstly select the analytes you have targeted within the EUPT-SRM14 and enter your Reporting Limit (RL) of each.

As default the MRRLs were set as Reporting Limits. For each pesticide within your PT Scope please **change the Reporting Limit** to that of your laboratory.

Please also state which of the analytes are "*Within your routine scope*". This column is mandatory for all compounds on the Target Pesticides List regardless of whether they are within your PT Scope. 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*". Any additional information may be added as a free text under "*Not analysed details*".

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

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.

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 "12.345"; use points for decimal separation. **Numerical values have 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.
Concentration blank	mg/kg	Concentration in Blank Material; (mandatory if marked as detected in blank); Syntax "12.345"; use points for decimal separation.
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 correction are covered by other questions**
Recovery %	%	(Mean) recovery rate used to obtain the recovery-corrected result reported for the Test Item (in %), Syntax "123"
Recovery Obtained		Please 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 indicate how the recovery rate was obtained; indicate the matrix used if not matched, spiked compound, spiking level/range

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.
Use the edit function to get an overview of all method-information fields of a selected pesticide.
Use copy function to copy the information from one pesticide to another.

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

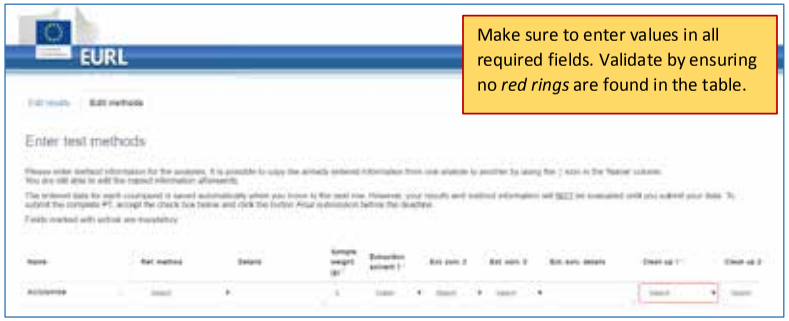
Field(s)	Unit	Explanation
Ref. method		Choose from the drop-down list; if you have used a modified form of the mth pls. give details under "Mth Details"
Ref. Method modified		Specify if you have introduced any noteworthy modifications to the selected reference method
Mth. details		Describe you method shortly if it is not on the dropdown menu or indicate shortly the modifications introduced to the reference method
Experience with this compound		Experience of your lab with the analysis of this pesticide (with any type of commodity)
Initial Sample Temp	°C	Initial Temperature of Test Item portion employed in procedure (choose closest value)
Sample thawed prior to analysis		Please indicate if and for how long approximately your sample was left in a THAWED state between reception until analysis of the compound
Details on sample thawing		Please provide any details relevant to the thawing of the sample (e.g. "thawed over night in refrigerator")
Sample Weight (g)	g	Enter the sample weight used. Syntax "3". Mandatory

Field(s)	Unit	Explanation
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 solvent
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	minutes	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
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 menu
Clean up 2		Choose the clean-up approach employed from the drop down menu - if you use more than one clean up step
Clean up 3		Choose the clean-up approach employed from the drop down menu - if you use more than two clean up step
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 CS2,
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 and any other details of importance such as differences between sample extract and calibration solution (e.g. different cleanup, dilution etc.)
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 Analysis)		Please enter here any general comments concerning the analysis of the selected compound

Guide to EUPT-SRM Results Submission Webtool

Final Submission

Please fill in all required fields, otherwise you cannot submit your data. Check carefully that no *red rings* are found indicating missing entries and fill in the missing information – see example below

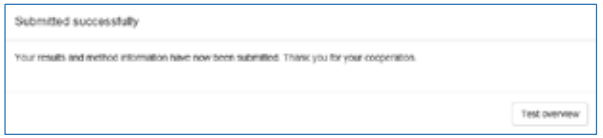


When all fields are filled out and you have checked their correctness, you are ready to submit your results. Accept and submit your final results by clicking the check box and then click on *Final submission*.

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. 23 April, 15 h (3 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 Excel file attached, 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*

Additional Information

After the PT deadline, if you have submitted tentatively false negative result(s) on this PT, the PT-row will be highlighted in 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 2 May.**

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

Tentatively false positives and will be marked with red colour.

Appendix 13 Supplementary Information on Analytes

Group	Compound	Residue definition Liver	Approved in EU	Listed in MACP-Reg. AO	Listed in WD19 (AO)	Anal. Approach (exemplary)	CAS	Standard Providers (exemplary)	Liver MRLs
2,4-D	**2,4-D (free)**	2,4-D (sum of 2,4-D, its salts, its esters and its **conjugates,** expressed as 2,4-D)	**Yes**	no	No	QuEChERS-ESI-Neg	94-75-7	Various	Poultry: 0.05*; Swine, Bovine, Sheep, Goat, Equine: **5 mg/kg**; Other: 0.2
2,4-DB	**2,4-DB (free)**	2,4-DB-code 1000000 except 1040000: sum of 2,4-DB and its **conjugates,** expressed as 2,4-DB	**yes**	no	**Yes**	QuEChERS-ESI-Neg	94-82-6	Various	Swine, Poultry: 0.05*; Bovine and Other: **0.4 mg/kg**
Abamectin	**Avermectin B1a**	Abamectin — code 1000000 except 1040000: avermectin B1a	**Yes (plus vet drug)**	no	No	QuEChERS-ESI-Pos	65195-55-3	HPC (B1a individually)	Poultry and swine 0.01*; sheep 0.025; Bovine and Other: **0.02 mg/kg**
BAC	**BAC C12**	Benzalkonium chloride (mixture of alkylbenzyldimethylammonium chlorides with alkyl chain lengths of C8, C10, C12, C14, C16 and C18)	**No (but biocide)**	no	**Yes**	QuEChERS-ESI-pos	139-07-1	Sigma, HPC, LGC, TRC	All commodities: **0.1 mg/kg (sum)**
Bixafen	**Desmethyl bixafen**	Bixafen- code 1000000 except 1040000: sum of bixafen and desmethyl bixafen expressed as bixafen	**Yes**	no	**Yes**	QuEChERS-ESI-Pos	1655498-06-8	Sigma (Bixafen Metabolite BYF00587), LGC, Kanto	Poultry 0.05*; Bovine and Other: **4 mg/kg**
Boscalid	**Boscalid Metabolite M510F01 (free)**	Sum of boscalid and its hydroxy metabolite 2-chloro-N-(4'-chloro-5-hydroxybiphenyl-2-yl)nicotinamide (free and **conjugated)** expressed as boscalid	**yes**	no	**Yes**	QuEChERS-ESI-Neg	661463-87-2	HPC, Sigma, LGC (Boscalid-5-hydroxy), TRC	Poultry: 0.15 mg/kg; Bovine and Other: **0.2 mg/kg**
Bromoxynil	**Bromoxynil**	Bromoxynil and its salts, expressed as bromoxynil	**Yes**	no	No	QuEChERS-ESI-Neg	1689-84-5	Various	Poultry: 0.05*; Swine: 0.1; Bovine and Other: **0.5 mg/kg**
Chlorate	**Chlorate**	Chlorate	**No (but biocide)**	no	**Yes**	QuPPe-ESI-Neg	7775-09-9 (sodium salt)	Various	Default MRL **0.01 mg/kg** (Art 18(1)(b) Reg 396 / 2005)
Chlormequat	**Chlormequat**	Chlormequat	**Yes**	no	**Yes**	QuPPe-ESI-Pos	999-81-5, 7003-89-6 (chloride salt)	Various	Goat, Sheep, Swine, Bovine **0.15;** Poultry 0,1; Other: 0.5 mg/kg
DDAC	**DDAC C10**	Didecyldimethylammonium chloride (mixture of alkyl-quaternary ammonium salts with alkyl chain lengths of C8, C10 and C12)	**No (but biocide)**	no	**Yes**	QuPPe-ESI-Pos	7173-51-5 (chloride salt)	HPC, Sigma, LGC, TRC	All commodities: **0.1 mg/kg (sum)**
Dicamba	**Dicamba**	Dicamba	**Yes**	no	No	QuEChERS-ESI-Neg	1918-00-9	Various	Swine, Poultry: 0.07; Bovine and Other: **0.7 mg/kg**
Dichlorprop	**Dichlorprop incl. dichlorprop-P (free)**	Dichlorprop — code 1000000 except 1040000: Sum of dichlorprop (including dichlorprop-P) and its salts, expressed as dichlorprop	**Yes**	no	**Yes**	QuEChERS-ESI-Neg	120-36-5 (racemate); 15165-67-0 (-P)	Various	Poultry, Swine: 0.05*; Bovine and Other: **0.06 mg/kg**
Emamectin	**Emamectin B1a**	Emamectin benzoate B1a, expressed as emamectin	**Yes**	no	No	QuEChERS-ESI-Pos	121124-29-6 (B1a, individually); 155569-91-8 (mixture as benzoate salt)	Emamectin benzoate isomeric mix: various; Emamectin B1a, individually: TRC, HPC	Poultry 0.01*; Bovine and Other: **0.08 mg/kg**
Ethephon	**Ethephon**	Ethephon	**Yes**	no	No	QuPPe-ESI-Neg	16672-87-0	Various	Poultry 0.08; Bovine and Other: **0.4 mg/kg**
Fenpropimorph	**Fenpropimorph carboxylic acid (BF-421-2)**	Fenpropimorph - code 1000000: Fenpropimorph carboxylic acid (BF 421-2) expressed as fenpropimorph	**Yes**	no	**Yes**	QuEChERS-ESI-Pos	121098-45-1	HPC, TRC, SCBT, carbosynth	Swine: 0.03; Poultry 0,01*; Bovine and Other: **3 mg/kg**
Flonicamid	**Flonicamid metabolite, TFNA-AM**	Sum of flonicamid and TFNA-AM, expressed as flonicamid	**yes**	no	No	QuEChERS-ESI-Pos	158062-71-6	HPC, Interchim, WAKO, TRC, Apollo Scientific	Poultry 0.1; Bovine and Other **0.2 mg/kg**
Fluazifop	**Fluazifop incl. Fluazifop P (free)**	Fluazifop-P (sum of all the constituent isomers of fluazifop, its esters and its **conjugates,** expressed as fluazifop)	**Yes**	no	**Yes**	QuEChERS-ESI-Neg	83066-88-0 (-P), 69335-91-7 (racemate)	Various	Poultry 0,04 mg/kg; Bovine and Other: **0.03 mg/kg**
Fluopyram	**Fluopyram-benzamide (M25)**	Fluopyram - code 1000000 except 1040000: sum fluopyram and fluopyram-benzamide (M25) expressed as fluopyram	**Yes**	no	**Yes**	QuEChERS-ESI-Pos	360-64-5	Sigma, Apollo Scientific: 2-(Trifluoromethyl)benzamide; HPC (Fluopyram-benzamide)	Poultry: 2 mg/kg ; Swine, bovine, sheep, goat: **5 mg/kg;** Other: 0.7 mg/kg
Glufosinate	**Glufosinate**	Glufosinate-ammonium (sum of glufosinate, its salts, MPP and NAG expressed as glufosinate equivalents)	No (exp. 07/18)	no	**Yes**	QuPPe-ESI-Neg	51276-47-2; 77182-82-2 (ammonium salt)	Various	Poultry 0.1 mg/kg; Bovine and Other: **3 mg/kg**
Glufosinate	**Glufosinate metabolite, MPP**	Glufosinate-ammonium (sum of glufosinate, its salts, MPP and NAG expressed as glufosinate equivalents)	No (exp. 07/18)	no	**Yes**	QuPPe-ESI-Neg	15090-23-0	Various (3-methylphosphinicopropionic acid)	Poultry 0.1 mg/kg; Bovine and Other: **3 mg/kg**

Appendix 13 (cont.) Supplementary Information on Analytes

Group	Compound	Residue definition Liver	Approved in EU	Listed in MACP-Reg. AO	Listed in WD19 (AO)	Anal. Approach (exemplary)	CAS	Standard Providers (exemplary)	Liver MRLs
Glufosinate	**N-Acetyl-Glufosinate**	Glufosinate-ammonium (sum of glufosinate, its salts, MPP and NAG expressed as glufosinate equivalents)	No (exp. 07/18)	no	**Yes**	QuPPe-ESI-Neg	73634-73-8	Various	Poultry 0.1 mg/kg; Bovine and Other: **3 mg/kg**
Glyphosate	**Glyphosate**	**Current RD:** Glyphosate **Future residue definition:** Glyphosate (sum of glyphosate, AMPA and N-acetylglyphosate)	**Yes**	**yes**	**Yes**	QuPPe-ESI-Neg	1071-83-6; 40465-66-5 (Ammonium-Salz)	Various	Ruminant: 0.2, Bovine and Other: 0.05* **EFSA 2018;16(5):526:** Swine: 0.4; Bovine, Equine: **0.7mg/k** Sheep, Goat: 0.9; Poultry 0.2* mg/kg
Glyphosate	**Glyphosate metabolite, AMPA**	**Current RD:** Glyphosate **Future residue definition:** Glyphosate (sum of glyphosate, AMPA and N-acetylglyphosate)	**Yes**	**yes**	**Yes**	QuPPe-ESI-Neg	1066-51-9	Various	Ruminant: 0.2, Bovine and Other: 0.05* **EFSA 2018;16(5):526:** Swine: 0.4; Bovine, Equine: **0.7mg/k** Sheep, Goat: 0.9; Poultry 0.2* mg/kg
Glyphosate	**N-Acetyl-Glyphosate**	**Current RD:** Glyphosate **Future residue definition:** Glyphosate (sum of glyphosate, AMPA and N-acetylglyphosate)	**Yes**	**yes**	**Yes**	QuPPe-ESI-Neg	129660-96-4	Various	Ruminant: 0.2, Bovine and Other: 0.05* **EFSA 2018;16(5):526:** Swine: 0.4; Bovine, Equine: **0.7mg/k** Sheep, Goat: 0.9; Poultry 0.2* mg/kg
Haloxyfop	**Haloxyfop, incl. Haloxyfop-P (free)**	Haloxyfop (Sum of haloxyfop, its esters, salts and **conjugates** expressed as haloxyfop (sum of the R- and S-isomers at any ratio)) (F) (R)	**Yes**	no	**Yes**	QuEChERS-ESI-Neg	69806-34-4 (racemate); 95977-29-0 (-P)	Various	All **0.03 mg/kg**
Isoxaflutole	**Isoxaflutole metabolite (RPA202248)**	Isoxaflutole (sum of isoxaflutole and its diketonitrile-metabolite, expressed as isoxaflutole)	**Yes**	no	Yes	QuEChERS-ESI-NEG	143701-75-1	HPC (Isoxaflutole diketonitrile RPA 202248), TRC	Poultry: 0.2; Bovine and Other: **0.1 mg/kg**
MCPA	**MCPA (free acid)**	MCPA and MCPB - code 1000000: MCPA, MCPB and MCPA thioethyl expressed as MCPA	**Yes**	no	**Yes**	QuEChERS-ESI-Neg	94-74-6	Various	Poultry: 0.1*; Bovine and Other: **3 mg/kg**
MCPB	**MCPB (free acid)**	MCPA and MCPB - code 1000000: MCPA, MCPB and MCPA thioethyl expressed as MCPA	**Yes**	no	**Yes**	QuEChERS-ESI-Neg	94-81-5	Various	Poultry: 0.1*; Bovine and Other: **3 mg/kg**
Mepiquat	**Mepiquat**	Mepiquat (sum of mepiquat and its salts, expressed as mepiquat chloride)	**Yes**	no	**Yes**	QuPPe-ESI-Pos	15302-91-7 ; 24307-26-4 (chloride salt)	Various	Poultry 0,05* mg/kg; Bovine and Other: **0.5 mg/kg**
Quizalofop	**Quizalofop, incl. Quizalofop-P (free)**	***396:*** *Quizalofop, incl. quizalfop-P* ***EFSA Art12 Report (Dec 17):*** *sum of quizalofop, its salts, its esters (including propaquizafop) and its* ***conjugates,*** *expressed as quizalofop (any ratio of constituent isomers)*	**Yes**	no	**Yes**	QuEChERS-ESI-Neg	94051-08-8 (-P); 76578-12-6 (racemate)	Various	***396:*** *All 0.05*;* ***EFSA Art12 Report (Dec 17):*** Swine: 0.02*; Bovine and Other: **0.03 mg/kg;** Poultry: 0.04 (different RD)
Thiabendazole	**5-Hydroxythiabendazole**	Thiabendazole - code 1000000: Sum of thiabendazole and 5-hydroxythiabendazole	**Yes**	no	No	QuEChERS-ESI-Pos	948-71-0	Sigma, HPC, TRC, kanto	Poultry 0.2; Bovine: **0.3 mg/kg;** swine, goat, sheep other 0.15 mg/kg
Triclopyr	**Triclopyr**	Triclopyr	**Yes**	No	No	QuEChERS-ESI-Neg	55335-06-3	Various	Swine, Poultry: 0.01*; Bovine and Other: **0.06 mg/kg**

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