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EURL-SRM

EU Reference Laboratories for Residues of Pesticides
Single Residue Methods

EU Proficiency Test on the analysis of Honey for incurred and spiked residues of pesticides requiring Single Residue Methods

EUPT–SRM18

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Final Report

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EU PROFICIENCY TEST
EUPT-SRM18, 2023

Residues of Pesticides Requiring Single Residue Methods

Test Material: Honey

Final Report

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approved by Michelangelo Anastassiades
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FOREWORD

Regulation (EU) 2017/625 [1] defines the general tasks and duties of the EU Reference Laboratories (EURLs) for Food, Feed and Animal Health[1] including the organisation of comparative tests (proficiency tests = PTs). These PTs are carried out on an annual basis and aim to improve the quality, accuracy and comparability of the analytical results generated by EU Member States within the framework of the EU coordinated control programs as well as national monitoring programs. By participating in PTs laboratories can assess and at the same time demonstrate their analytical performance. The attention to details paid by laboratories during PT-analysis, together with the need to identify errors and to take corrective actions in cases of underperformance, lead to continuous improvements in the quality of analytical results.

According to Article 28 of Reg. (EC) 2005/396 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 frame-work 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 Article 38 (2) of the regulation on official controls (Reg. (EU) 2017/625), where it reads as follows: "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 Reg. (EU) 2017/625 furthermore prescribes the participation of NRLs in these comparative tests: "National reference laboratories shall, in their area of competence: (a) collaborate with the European Union reference laboratories, and participate in training courses and in inter-laboratory comparative tests organised by these laboratories."

Since 2006 the EURL for pesticide residues requiring the use of Single Residue Methods, EURL-SRM, has annually conducted one scheduled Proficiency Test. Six of those 18 EUPT-SRMs were conducted in collaboration with the EURL for pesticide residues in Fruits and Vegetables (EURL-FV) using apple juice (EUPT-SRM1, 2006), carrot homogenate (EUPT-SRM3, 2008), apple purée (EUPT-SRM5, 2010), potato homogenate (EUPT-SRM8, 2013), spinach homogenate (EUPT-SRM11, 2016) and tomato homogenate (EUPT-SRM18, 2022) as test items. Five other EUPT-SRMs were conducted in collaboration with the EURL for pesticide residues in Cereals and Feeding Stuff (EURL-CF) with wheat flour (EUPT-C1/SRM2, 2007), oat flour (EUPT-C3/SRM4, 2009), rice flour (EUPT-C5/SRM6, 2011 and EUPT-SRM15, 2020) and maize flour (EUPT-C9/SRM10, 2015) as test items. Further six EUPT-SRMs were organized by the EURL-SRM unilaterally, two of them were based on commodities of plant origin with low fat/oil 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 EUPT-SRM14 (2019) was based on bovine liver homogenate and the first one EUPT-SRM in cooperation with the EURL for Residues of Pesticides in Food of Animal Origin (EURL-AO), and two PTs used oil seeds as commodities: The EUPT-SRM13 (2018) using flour of whole soybeans was the first one using a commodity with low water and high oil content (about 22 %), the EUPT-SRM16 (2021) using sesame seeds focused, among others, on the analysis of ethylene oxide and 2-chloroethanol in response to the massive recalls of sesame products within the EU in 2020. The present EUPT-SRM18 uses honey as test material since honey was added to the SANTE working document on pesticides to be considered in national control programmes (MANCP)[2] and was carried out alongside a joint venture pilot monitoring programme between the EURL-SRM and the EURL-AO covering honey samples from all over the world.

Participation in EUPT-SRMs is mandatory for all NRLs for pesticides requiring Single Residue Methods (NRL-SRMs) and for all OfLs analysing pesticide residues within the framework of national or EU control programs in commodities represented by the respective EUPT test item. Laboratories in EU Member States analysing

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

[2] SANCO/12745/2013 21 – 22 November 2022 rev. 14(5)

pesticide residues within the frame of import controls according to Reg. (EU) 2019/1793 are also considered to be performing official controls in the sense of Reg. (EU) 2017/625 and are thus also obliged to take part in EUPTs. OfLs from EFTA countries (Iceland and Norway) that are or used to be 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-candidate countries (i.e. Albania, Bosnia and Herzegovina, Montenegro, North Macedonia, Serbia and Turkey) are also invited to take part in EUPTs. A limited number of laboratories from third countries, in particular if they are involved in the control of food or feed exported to EU member states, are allowed to take part in this exercise, too, as long as sufficient test material is available. However, only results submitted by labs from EU and EFTA countries are included in the calculation of the assigned values.

Based on information about the commodity scope and the labs' NRL-function, a tentative list of EU-OfLs considered to be obliged to participate in the EUPTs organised within a PT-season is uploaded onto the PT registration page. The pesticide scope is not taken into account in these lists. NRLs and OfLs can see their participation status during the registration. Laboratories listed as being obliged to participate in an EUPT exercise in a given year but deciding not to take part, are asked to state the reason(s) for their non-participation. The same applies to laboratories originally registering to participate in a certain EUPT but finally not submitting results.

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

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EUROPEAN COMMISSION –
EU-PROFICIENCY TEST ON RESIDUES OF PESTICIDES
REQUIRING SINGLE RESIDUE METHODS
MATRIX: HONEY
EUPT-SRM18, 2023

INTRODUCTION

On 20 February 2023 the Announcement/Invitation Letter (**Appendix 11**) for the 18th EU Proficiency Test for pesticides requiring Single Residue Methods (EUPT-SRM18) was published on the EUPT-SRM18-Website. The invitation was accompanied by a Calendar and a Preliminary Target Pesticides List (**Appendix 10**). All relevant National Reference Laboratories (NRLs) of the 27 EU-Member States (MS), as well as all relevant EU-Official Laboratories (OfLs) whose contact details were available to the organisers were invited to participate.

The preliminary Target Pesticides List following consultation with the EUPT-Scientiffic Committee entailed 17 compulsory and 7 optional analytes. For each of the analytes on the Target Pesticides List a residue definition valid for the PT and the minimum required reporting level (MRRL) were stipulated. The selection of the compounds considered the entries within the SANTE working document on pesticides to be considered in national control programmes[1], the relevance of compounds for honey, the availability of analytical standards, and the capability of laboratories. Due to a partial overlap with the analyte scope of EUPT-AO18 (also with honey as test material), the amitraz metabolites DMF and DMPF were later removed from the Target Pesticides List. The list was, however, supplemented by chloridazon-desphenyl, copper and bromide. Chloridazon-desphenyl was added to the list to address issues of potential false positive, while copper and bromide were marked as "extra analytes" and were kept at natural levels. Copper was added to the target list following a request by DG-SANTE, with the intention to push the OfLs to establish procedures for copper analysis in products sampled and further processed for the purpose of pesticide residue analysis. The background for the wish to entail copper in the PT was the decission to include copper in the EU-coordinated monitoring program on pesticides. It was, however, finally decided not to evaluate the lab results on copper as the analysis of copper at very low levels is affected by cross contaminations and background levels. The final Target Pesticide List entailed 17 compulsory, 7 optional and 2 extra analytes. The updated list was published on 8 May 2023 and the participants were informed.

NRL-SRMs and all OfLs analysing pesticide residues in honey within the framework of official controls, including those involved in the import controls of products listed under Reg. (EU) 2019/1793 as far as they could be tracked in the EURL-DataPool, as well as EU laboratories analysing official organic samples within the frame of Reg. (EC) 2008/889 were called for registration on 13 March 2023. NRLs and OfLs from EFTA and EU candidate countries were also invited if their contact data were available. In addition official and commercial laboratories from third countries, in particular those involved in the export control of foods or feeding stuff to the EU, were also invited and accepted as participants in the present PT. Only the results from EU and EFTA OfLs were, however, taken into account for the establishment of the assigned values of all analytes.

[1] SANCO/12745/2013 21 – 22 November 2022 rev. 14(5)

Based on their commodity scope and NRL-function (NRL-SRMs) all official laboratories were allocated a tentative status as regards their obligation to participate in the EUPT-SRM18. This status was tracked in the DataPool, so that every participant could see it during the registration. To ensure that all concerned official laboratories were informed about this EUPT, the NRLs were asked to forward the invitation to all relevant official laboratories within their countries. It was made clear that the status of the laboratories was only tentative, and the real obligation to participate was based on the respective regulations. From 17 March till 18 April, laboratories obliged or interested to participate in this PT could register using the registration form on the EURL DataPool. Obliged laboratories not intending to participate in the PT had to register for non-participation and state their reason. The SRM18 Specific Protocol (**Appendix 9**) was provided to the participants on 8 May 2023 via hyperlinks in e-mails. The SRM18 Webtool Guideline (**Appendix 12)** was made accessible to the participants via a web-link, which was communicated via email and on the Webtool itself.

In total, 71 official laboratories (including NRLs) from 33 countries (26 EU-Member States, 2 EFTA-countries) and four laboratories from other countries (1 EU candidate country and 3 non-European countries) registered for participation in the EUPT-SRM18 and reported at least one result for this PT.

The robinia blossom honey, that was used to produce the EUPT-SRM18 test material, was purchased from a German honey producer and contained both incurred/natural levels of some analytes. Further compounds were spiked prior to homogenization. Spiking, homogenization and portioning was done in the "Landesanstalt fur Bienenkunde", at the University of Hohenheim, with technical assistance being provided by Dr. Klaus Wallner. More details are given in Chapter 1 "Test Materials".

1. TEST ITEM

1.1 Selection of PT-Commodity

During the evaluation meeting in June 2022, it was decided to use honey as the PT test material both for the EUPT-SRM18 and -AO18. Initially, it was planed to conduct both PTs using the same material. But for logistical and technical reasons, it was finally decided to use separate test materials for the two EUPTs. In Order to avoid the honey test sample crystallising during the PT exercise, which would have led to uncertainty among the labs regarding sample handling, robinia honey was chosen as raw material. Honey of *Robinia pseudoacacia*, often falsely referred to as acacia honey, naturally contains high levels of fructose, which makes it more liquid compared to most other types of honey. In a preliminary test, no crystallization was observed during the storage of robinia honey in the refrigerator over a period of six weeks.

1.2 Sourcing of Honey for PT material production

Approximately 25 kg of robinia honey were purchased from a beekeeper from Eastern Germany with Dr. Klaus Wallner from the State Bee Institute at University of Hohenheim acting as a broker for the purchase. The honey only contained a few of the analytes of interests at trace levels and it was therefore deemed fit for the purpose.

1.3 Selection of Compounds for the Target Pesticides List (TPL)

The compounds to be included in the Target Pesticides List (TPL, **Appendix 10**) were selected by the organiser and the EUPT-Scientific Committee (Advisory Group and Quality Control Group) taking the following points into account:

1) the scope of analytes recommended for the analysis in honey samples within the framework of Multi-Annual National Monitoring Programs (MANCP) as listed in the Working Document (SANCO/12745/2013 rev. 14(5));
2) the scope of the EU-coordinated Multi-Annual Control Program (EU-MACP) as regulated, and the Implementing Reg. (EU) 2022/741;
3) the relevance of certain analytes to matrix (honey) based on the collection of information from various sources and an own pilot monitoring program;
4) the capabilities and interests of the potential participants as revealed through a survey on SRM18 Target Pesticides run among the OfLs in November 2022 (e. g., it was deemed reasonable to skip the analysis of Amitraz (sum) due to the limited number of laboratories employing the common moiety approach);
5) the suggestions/Votes by the EUPT-Scientific Committee;
6) the intention to keep the number of different methods required to cover the full scope of analytes reasonably low;
7) coordination with the EURL-AO (it was, for example, agreed that the two main metabolites of amitraz, DMPF and DMF are to be covered by the EUPT-AO18)

The minimum required reporting levels (MRRLs) were set as shown below (compounds present in the test material are highlighted in bold and italic).

- at 0.01 mg/kg for ***2,4-D (free acid)***, *BAC-C12 chloride*, ***BAC-C14 chloride, chlorate (anion)***, *chlormequat chloride*, ***DDAC-C10 chloride, fluazifop (free acid, sum of isomers)***, *fosetyl*, ***glyphosate***, *haloxyfop (free acid, sum of isomers)*, ***matrine***, *mepiquat chloride*, ***nicotine***, ***oxymatrine***, *AMPA, MCPA (free acid), N-acetyl-glyphosate*, ***perchlorate***, *quizalofop (free acid, sum of isomers)*, ***trinexapac (free acid)***,
- at 0.02 mg/kg for *chloridazon-desphenyl*,
- at 0.03 mg/kg for ***phosphonic acid***,
- no MRRLs for ***bromide anion*** and ***copper*** (both were left at their natural level and were labelled as "extra" analytes)

1.4 Preliminary Investigations: Analyte Stability in Honey

Different portions of honey were spiked and left standing at ambient temperature or at –18 °C for four weeks to check the potential stability of the TPL-compounds during storage. Analysis was conducted by the QuEChERS method as well as by the QuPPe method, with the latter focusing on highly polar analytes. Most compounds proven to be stable, with some exceptions:

a) Fosetyl degraded nearly completely into phosphonic acid when the sample was stored at ambient temperature. Phosphonic acid, spiked separately, was shown to be stable;
b) The free phenoxy-acids (2,4-D, Fluazifop, Haloxyfop, MCPA and Quizalofop) showed marginal losses after storage for four weeks at ambient temperature (6 – 12 %), but a good stability at –18 °C. It couldn't be ruled out that measurement uncertainty had its influence on the deviation, but a conjugation of the acids with sugars from the honey might have also taken place;
c) The stability of amitraz was also checked although this compound was finally excluded from the PT-scope. Amitraz was found to degrade rapidly in honey at ambient temperature leading to the formation of its two degradation products DMF and DMPF.

1.5 Preparation of the Test Material and Bottling of the Test Item

Based on discussions with the Advisory Group, it was decided to include the 14 compounds listed in **Table 1-1** in the EUPT SRM18 test material, of which 10 are mandatory and the remaining two are optional. As only 4 of them were present in trace amounts in the base material, all these 12 compounds were additionally spiked in the laboratory. The two extra analytes, bromide ion and copper, were left at their natural levels.

25 kg of the honey material was warmed to 25 °C in a large plastic container. The honey was homogenized with a Rapido stirrer driven by a drill. A defined amount of the homogenized "blank" honey material was withdrawn to be used for EURL-SRM experiments. The remaining material was spiked with the selected analytes by adding defined amounts of analyte mixtures. ***Chlorate*** and ***nicotine*** were dissolved in water, ***BAC-C14 chloride, DDAC-C10 chloride, glyphosate, matrine, perchlorate*** and ***phosphonic acid*** were dissolved in

Table 1-1: Analytes to be included in the test material and spiked into 25 kg honey for the preparation of EUPT-SRM18 test material

	Analytes to be included in the test material (expr. as required in the TPL)	Residues incurred	Spiked in lab	Compounds applied in lab	Stock Solution		Theor. exp. conc. in the test material [mg/kg]
					Conc. [mg/ml]	Volume employed in the spiking mixture [ml]	
Mandatory	**2,4-D (free acid)**	traces	Yes	2,4-D (free acid)	1.0 [3)]	1.25	0.05
	BAC C14 chloride	No	Yes	BAC-C14 chloride	1.0 [2)]	3.0	0.12
	Chlorate (anion)	traces	Yes	Sodium chlorate	1.0 [1)]	2.5	0.1
	DDAC-C10 chloride	No	Yes	DDAC-C10 chloride	1.0 [2)]	3.75	0.15
	Fluazifop (free acid)	No	Yes	Fluazifop (free acid)	1.0 [3)]	1.5	0.06
	Glyphosate	No	Yes	Glyphosate	1.0 [2)]	2.5	0.1
	Matrine	No	Yes	Matrine	1.0 [2)]	2.25	0.09
	Nicotine	No	Yes	Nicotine	1.0 [1)]	2.25	0.09
	Oxymatrine	No	Yes	Oxymatrine	1.0 [3)]	1.75	0.07
	Phosphonic acid	traces	Yes	Phosphonic acid	1.0 [2)]	5.0	0.2
Optional	**Perchlorate**	traces	Yes	Sodium perchlorate	1.0 [2)]	1.5	0.06
	Trinexapac (free acid)	No	Yes	Trinexapac (free acid)	1.0 [3)]	2.75	0.11
Extra	**Bromide (anion)**	Yes	No	–	–	–	at natural level
	Copper	Yes	No	–	–	–	at natural level

[1)] in water; [2)] in water + 10 % acetonitrile ; [3)] in acetonitrile

water containing 10% acetonitrile, ***2,4-D (free acid), fluazifop (free acid), oxymatrine*** and ***trinexapac (free acid)*** were dissolved in acetonitrile. All solutions were mixed together and diluted with acetone, so that the mixture finally consisted of approx. 10 % acetonitrile, 20 % water and 70 % acetone and with a final volume of around 250 mL. Spiking was done by slowly pouring the mixture to the honey while stirring. The empty vessel was then rinsed twice with a small volume (approx. 25 mL in total) of an acetone mix. The amounts of analytes spiked to the material and the resulting concentrations are shown in **Table 1-1**.

After spiking, the honey was intensively stirred for further 10 min with the Rapido stirrer during which the honey heated up slightly and the acetone evaporated significantly, with only decent smell remaining after stirring. After mixing, the spiked and homogenated honey was filled into a special bucket with a suitable outlet for portioning honey. Portions of ~150 g test material were filled into 150 pre-numbered bottles, which were then sealed with a lid.

1.5.1 Preliminary Analysis

The preparation and bottling of the material took place with ample time before shipment, thus allowing the homogeneity test to be conveniently conducted prior to shipment (see **Section 1.8 Homogeneity Test**). A preliminary check for the presence, concentration and homogeneity of the spiked analytes, prior to bottling, was therefore deemed unnecessary in this case.

1.6 Packaging and Delivery of PT Materials to Participants

Three days prior to their dispatch to the participants, the bottles with the PT-material were packed into cardboard boxes (one each), filled-up with thin paper, without any other cooling pads and stored in a walk-in fridge (at 4 °C) till pick-up by DHL-Express on Friday 19 May for shipment to the laboratories. Two boxes, each containing one Test Item, were sent to laboratories having ordered double amount. Once the parcel was picked-up by DHL, the main PT-contact-person of each participating laboratory received an e-mail from the shipping company (DHL Germany) showing the individual online tracking number of the laboratory. In the Specific Protocol, which was distributed two weeks prior to the shipment of the test material, the participants were informed that the test material for the EUPT-SRM18 will be delivered without any cooling, and that it should be stored in the refrigerator upon arrival. Storage in the refrigerator was deemed adequate for the duration of the EUPT, as the pre-experiment run at ambient temperature showed good stability of the analytes during storage. Furthermore, a parallel experiment showed that the particular honey does not crystallise in the refrigerator at long-term storage.

Among the 81 packages sent to laboratories in EU and EFTA countries, 75 packages (93 %) reached the participating labs on Monday 22 May (= within 3 days). Further 3 packages (4 %) arrived on Tuesday 20 May (= within 4 days) due to the remote location of the affected laboratories. All packages to EU and EFTA-labs and the United Kingdom arrived the participants within 4 days. Due to lengthy customs procedures, one third country laboratory did not receive the package until 26 May, while two laboratories in EU candidate countries received their samples with even longer delays (1 and 23 June). In the latter case the package was fortunately stored in a refrigerator in the customs office. The test materials for the participants in Latin America were handed over to representatives of these labs during the LAPRW conference in Panama on 22 May 2023. All material was accepted by the participants and was reportedly in very good condition. Considering the preliminary experiments on the stability of the analytes and the fact that the EU and EFTA OfLs (the results of which are considered when calculating the assigned value) received the samples within a relatively short period, it was assumed that differences in shipment duration would have most likely no significant influence on the results or the assigned values. It was, therefore, decided not to further investigate the impact of shipment duration on analyte stability (see also **Section 1.10**. **Transport Stability Test, p. 7**). In the special cases of delayed receipt the delayed sample receipt would be highlighted if considered relevant. Details on shipment duration are compiled in **Appendix 2**.

The organisers would like to appeal the participants to track their own parcels via the online tracking tool of the shipping company in order to take any necessary measures in case of delays, e.g., providing the customs with all necessary documents, asking for an acceleration of the clearance procedure or for placing the parcel in a freezer until clearance is granted. The participants are furthermore encouraged to contact the local office of the shipping company to ensure optimal delivery timing.

1.7 Analytical Methods

The analytical methods used by the organisers to check the homogeneity and storage stability of the analytes contained in the test material and to verify the absence of the other TPL-analytes are summarized in **Table 1-2 (p. 5)**. For more details on the methods used, please refer to the EURL-SRM website: https://www.eurl-pesticides.eu (→EURL-SRM Analytical Method Reports and Analytical Observation Reports).

1.8 Homogeneity Test

After filling the test material into bottles, 10 bottles were randomly chosen for the homogeneity test and two analytical portions per bottle were taken for each analytical method. Both the sample extraction and the injection into the measurement instruments were done in random order. Matrix-matched calibration using extracts prepared from blank material or procedural calibration using blank material were applied for quantification. For all compounds, analytical portions of 5 g were used. No homogeneity test was conducted for bromide and copper, which were left at their natural content and regarded as "extra" analytes.

The statistical evaluation of the homogeneity test data was performed according to ISO 13528:2015 ("Statistical methods for use in proficiency testing be interlaboratory comparison") [6]. An overview of the statistical evaluations of the homogeneity test is shown in **Table 1-3 (p. 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 is that the estimate of the between-sample standard deviation s_s is smaller than $0.3 \times \sigma_{pt,}$ where $\sigma_{pt} = 0.3 \times$ FFP-RSD (25 %) × the analytical sampling mean of the analyte. In addition and for informative purpose only, the actual sampling error and repeatability were also calculated and compared. If the between-sample standard deviation s_s is smaller than the check value $\sqrt{c}$, then the batch of the PT test items can be regarded sufficiently homogeneous. The check value c is calculated as $F_1 \times \sigma_{allow}{}^2 + F_2 \times s_w{}^2$, F_1 and F_2 are constants with values of 1.88 and 1.01, respectively, when duplicate samples are taken from 10 bottles. $\sigma_{allow}{}^2 = 0.3 \times$ FFP-RSD (25 %) × the analytical sampling mean of the analyte, and s_w is the within sample standard deviation.

As all target compounds passed the homogeneity test, the test item was considered to be sufficiently homogenous and suitable for the EUPT-SRM18.

1.9 Storage Stability Test

In the Specific Protocol laboratories were recommended storing the bottles with the test item or any analytical portions withdrawn thereof in a refrigerator until the conduction of the analytical procedures.

The bottles randomly selected for the homogeneity test (with three of them being foreseen for the additional stability test) were initially stored under the same conditions as all other bottles. On the day of packing, these pre-selected bottles were set aside and then left at ambient temperature over four days to simulate the shipping duration to the laboratories. Thereafter, the required analytical portions for the homogeneity and the stability tests were portioned from the above selected bottles. The portions to be extracted on days 2 and 3 of the stability test were kept in a refrigerator at 4 °C until the scheduled analysis

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

QuEChERS Method [3]: involving: weighing of 5 g honey homogenate into a sealable vessel, addition of IS/ILIS, addition of water and acetonitrile, shaking, addition and distribution of salt mixture, centrifugation and direct determination by LC-MS/MS.				
Compound	**IS**	**Determinative analysis**		**Notes**
2,4-D (free acid)	2,4-D $^{13}C_6$	LC-MS/MS	EI (pos)	
BAC -C14 chloride	BAC -C14 D_7	LC-MS/MS	ESI (pos)	
DDAC-C10 chloride	DDAC-C10 D_6	LC-MS/MS	EI (pos)	
Fluazifop (free acid, sum of isomers)	Fluazifop D_3	LC-MS/MS	ESI (pos)	
Trinexapac (free acid)	Propyzamide D_3	LC-MS/MS	ESI (pos)	
*BAC-C12 chloride**	*BAC C12 D_6*	*LC-MS/MS*	*ESI (pos)*	
*Haloxyfop (free acid, sum of isomers)**	*Haloxyfop D_4*	*LC-MS/MS*	*ESI (neg)*	
*MCPA (free acid)**	*MCPA D_6*	*LC-MS/MS*	*ESI (pos)*	
*Quizalofop (free acid, sum of isomers)**	*Quizalofop D_3*	*LC-MS/MS*	*ESI (pos)*	
QuPPe-PO Method [5]: involving: weighing of 5 g honey homogenate into a sealable vessel, addition of ILISs, addition of water and methanol containing 1 % formic acid, shaking, centrifugation, filtration and direct determination by LC-MS/MS in the ESI (neg.) or ESI (pos.) mode				
Compound	**IS**	**Determinative analysis**		**Notes**
Chlorate (anion)	Chlorate $^{18}O_3$	LC-MS/MS	ESI (neg)	QuPPe M1.7
Glyphosate	Glyphosate ^{13}C ^{15}N	LC-MS/MS	ESI (neg)	QuPPe M1.6
Matrine	Matrine D_3	LC-MS/MS	ESI (pos)	QuPPe M4.2
Nicotine	Nicotine D_4	LC-MS/MS	ESI (pos)	QuPPe M4.2
Oxymatrine	Oxymatrine D_3	LC-MS/MS	ESI (pos)	QuPPe M4.2
Phosphonic acid	Phosphonic acid $^{18}O_3$	LC-MS/MS	ESI (neg)	QuPPe M1.6
Perchlorate	Perchlorate $^{18}O_3$	LC-MS/MS	ESI (neg)	QuPPe M1.7
*Chlormequat chloride**	*CCC D_4*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.2*
*Fosetyl**	*Fosetyl D_5*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Mepiquat chloride**	*Mepiquat D_3*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.2*
*AMPA**	*AMPA ^{13}C ^{15}N*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Chloridazon desphenyl**	*Chloridazon desphenyl $^{15}N_2$*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.2*
*N-Acetyl-glyphosate**	*N-Acetyl-glyphosate $^{13}C_2$ ^{15}N*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*

* : To check for absence in "Blank" Material (= base material without spiking)

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

	MANDATORY ANALYTES										**OPTIONAL**	
	2,4-D	**BAC-C14 (chloride)**	**Chlorate**	**DDAC-C10 (chloride)**	**Fluazifop**	**Glyphosate**	**Matrine**	**Nicotine**	**Oxymatrine**	**Phosphonic acid**	**Perchlorate**	**Trinexapac**
Analytical portion size [g]	5	5	5	5	5	5	5	5	5	5	5	5
Mean [mg/kg]	0.0535	0.105	0.108	0.145	0.0627	0.104	0.0910	0.0923	0.0724	0.204	0.0611	0.106
between-samples STD	$2.14 \times^{-4}$	$1.06 \times^{-3}$	$2.54 \times^{-4}$	$2.35 \times^{-3}$	0.0	0.0	$1.87 \times^{-4}$	$1.20 \times^{-3}$	$1.47 \times^{-3}$	$4.26 \times^{-3}$	$5.76 \times^{-4}$	$3.98 \times^{-3}$
Check Value	$4.01 \times^{-3}$	$7.91 \times^{-3}$	$8.09 \times^{-3}$	$1.09 \times^{-2}$	$4.70 \times^{-3}$	$7.83 \times^{-3}$	$6.83 \times^{-3}$	$6.92 \times^{-3}$	$5.43 \times^{-3}$	$1.53 \times^{-2}$	$4.58 \times^{-3}$	$7.96 \times^{-3}$
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed	passed	passed	passed	passed

date, as this was the temperature at which the participants were advised to store their test material prior to analysis. No homogeneity test was conducted for bromide and copper, which were left at their natural content and regarded as "extra" analytes.

For the analysis of the stability test samples the methods described in **Section 1.7 (p. 4)** were used. The dates on which extractions were made by each method are shown below:

Extraction day 1: 20 April 2023 (analytes via QuEChERS-method)
27 April 2023 (analytes via QuPPe-method)

Extraction day 2: 25 May 2023 (analytes via QuEChERS-method)
25 May 2023 (analytes via QuPPe-method)

Extraction day 3: 15 June 2023 (analytes via QuEChERS-method)
15 June 2023 (analytes via QuPPe-method)

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 obtained from stability test 1 and σ_{pt} the standard deviation used for proficiency assessment, typically 25 % of the assigned value. In the period between the first and the third stability tests, which was long enough to exceed the duration of the PT, and during which the samples were stored at 4 °C (= recommended conditions), all analytes contained in the test item were shown to be sufficiently stable (**Table 1-4, p. 6**). For the compounds passing the test it was assumed that the time elapsed between sample receipt by a lab and its analysis had a negligible influence on the results, provided that the recommended storage conditions were followed.

The detailed results of all analyses conducted within the framework of the stability test are shown in (**Table 1-4, p. 6**) and **Appendix 5**.

Table 1-4: Results of storage stability test (storage at 4 °C). For the details of each analytes please see the text and **Appendix 4**.

	MANDATORY ANALYTES										OPTIONAL	
	2,4-D	BAC-C14 (chloride)	Chlorate	DDAC-C10 (chloride)	Fluazifop	Glyphosate	Matrine	Nicotine	Oxymatrine	Phosphonic acid	Perchlorate	Trinexapac
	Storage at 4 °C (mean values in mg/kg)											
Extraction day 1	0.0534	0.1041	0.1078	0.1429	0.0625	0.104	0.0914	0.0924	0.0707	0.206	0.0616	0.1114
Extraction day 2	0.0515	0.1065	0.1039	0.1428	0.0598	0.097	0.0946	0.0905	0.0702	not analysed	0.0605	0.1107
Extraction day 3	0.0549	0.0978	0.1079	0.1383	0.0613	0.1075	0.0918	0.0929	0.0712	0.2078	0.061	0.1124
Deviation Analysis 3 vs. Analysis 1 [mg/kg] ([%])	0.00158 (3.0 %)	0.00624 (−6.0 %)	0.00001 (0.0 %)	0.00452 (−3.2 %)	0.0012 (−1.9 %)	0.00347 (3.3 %)	0.00038 (0.4 %)	0.00042 (0.5 %)	0.00054 (0.8 %)	0.00183 (0.9 %)	0.00059 (−1.0 %)	0.00099 (0.9 %)
$0.3 \times \sigma_{pt}$ [mg/kg]	0.0039	0.0089	0.0077	0.0112	0.0045	0.0077	0.0065	0.0066	0.0051	0.0152	0.0041	0.0089
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed	passed	passed	passed	passed

1.10 Transport Stability Test

With the exception of three laboratories outside the EU and EFTA, all other participants received the sample packages within four days and in a very good condition. The results reported by the three laboratories having received the material after 7 days did not imply any significant negative impact of this prolonged transport on the material. In addition, the assigned values for all analytes are calculated on the basis of the results submitted by the EU and EFTA laboratories, all of which having received the samples within four days of dispatch and within a maximum of 36 hours of each other. Based on this knowledge, it was judged that the impact of shipment duration on the assigned values and the stability of the analytes was negligible. Therefore, the organisers decided to skip the transport stability test in this PT.

1.11 Organisational Aspects

1.11.1 Laboratory Status: Mandatory and Optional Participation

Based on available information on NRL-status and commodity scope stored in the EURL-DataPool, the EU and EFTA OfLs, including the NRLs, were preliminarily divided into those with obligation to participate in the PT and those whose participation was on a voluntary basis. The OfLs were asked to update their status and analytical scope a few months prior to the PT. The NRLs were furthermore reminded of their responsibility of ensuring that the information concerning their network is up-to-date and that all obliged OfLs within their network were informed of this EUPT. All NRLs and OfLs were informed that the division into "obliged" and "voluntary" was tentative as the real obligation to participate is derived from the respective regulations and their real scope.

Following instructions from DG-SANTE, obliged laboratories that did not intend to participate in EUPT-SRM18 were asked to provide explanations for their non-participation.

1.11.2 Announcement / Invitation and EUPT-SRM18 Website

The EUPT-SRM18 was scheduled to run from 19 May till 16 June 2023. Within the EURL-Web-Portal an EUPT-SRM18-Website was set up on 27 January, 2023. All documents relevant to this EUPT (i.e., Announcement/ Invitation Letter (**Appendix 11**), Calendar and Target Pesticides List (TPL) (**Appendix 10**), Specific Protocol (**Appendix 9**) and General EUPT Protocol (**Appendix 8**) valid for the present PT were linked to this website. These documents were uploaded both to the EURL-Web-Portal and to the CIRCA BC.

On 19 February 2023 the Announcement/Invitation Letter for the EUPT-SRM18 was published on the EUPT-SRM18-Website and additionally sent to all NRL-SRMs and all OfLs within the EU member states analysing pesticide residues in honey, as these labs were considered obliged to participate in this exercise. NRLs and OfLs from EFTA and EU candidate countries not entailing the above commodities within the routine scope were also invited to participate on a voluntary basis. This also included laboratories involved in the import controls of products listed under Reg. (EU) 1793/2019 and EU laboratories officially analysing organic samples within the frame of Reg. 889/2008/EC. All these laboratories were considered eligible but not obliged to participate. The OfLs were informed that their obligation to participate in EUPTs arises from the respective regulations, irrespective of the content of the tentative list of obliged laboratories.

1.11.3 Registration

As in the previous EUPTs since 2017, the participants were able to register for this EUPT via a website connected to the EURL-DataPool. All laboratories required to participate in the current EUPT, whether or not

they intended to participate in this exercise, were invited to use the same website to either register or provide reasons for non-participation. Upon registration or change of registration status, the laboratories received an electronic confirmation of their participation or non-participation in the current EUPT.

1.11.4 Further instructions to the participants

Shipment of the Test Item: On 8 May (approximately two weeks prior to shipment), the Specific Protocol (**Appendix 9**) was released, providing detailed instructions on how to handle the EUPT-SRM18 test item upon receipt and further details on reporting. On 12 May, one week prior to the dispatch of the test items, a detailed guidance on how to submit results via the Webtool (**Appendix 12)** was also provided to the participants.

Webtool for Results Submission and Confidentiality: The acknowledgement of sample receipt and the submission of PT-results and method information is accomplished via an on-line data submission tool called "Webtool". This platform is run by the DTU on behalf of all four EURLs of the pesticide residues cluster. A main and at least one and up to three alternative contact persons are designated by each PT participant for this purpose. Login to the Webtool requires the use of personalized login credentials, which are unique to the registered email addresses of the PT contact persons. These login credentials are created after a person registrers to the Webtool for the first time and are sent to his/her email address before the Webtool-area of the particular PT becomes accessible for acknowledgement of sample receipt, typically on the date of sample shipment or one day after. Using his/her personal login credentials, the PT-contact person can access the results submission pages of all EUPTs to which he/she has registered using the e-mail address, which was tracked into the system during registration.

Each laboratory participating in a certain EUPT receives a unique lab code, as soon as one of its PT-contact persons, assigned during registration, logs into the particular EUPT-site within the Webtool. Personal login credentials and the unique lab code that differs from PT-to-PT warrantee confidentiality. For further information on confidentiality please refer to the General EUPT Protocol (**Appendix 8**).

The EUPT-SRM18 participants received their login credentials from the Webtool programmer at the DTU on the day of shipment on 19 May. The Webtool was accessible from 22 May, the first day that the packages arrived at the participating laboratories. It was available until 16 June.

After the submission deadline on 16 June, participants were informed on 17 June by the organiser via e-mail about the analytes present in the test material. Participants were instructed to access the Webtool to check if they had obtained any tentative false positive or false negative results. In the latter case, they were asked to report method details for compounds of false negative results via the Webtool between 17 and 22 June.

Actions following Results Submission and Preliminary Report: The Preliminary Report on the EUPT-SRM18 was published and sent to the participants on 10 June. This report entailed the prelminary z scores of the compounds present in the PT material, which were calculated based on the preliminary assigned values (prAV). Laboratories that had submitted false positive results, as well as laboratories that had received preliminary $|z\ scores| > 2$, were asked to investigate the reasons for this underperformance and to provide feedback using a laboratory-specific Excel spreadsheet provided by the organisers.

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 ormally disregarded by the organiser and not regarded as false positives. No z scores are calculated for false positive results. Any results reported for analytes not present in the test material and below the MRRL are normally not considered false positives, even though these results should not have been reported. If these results are additionally lower than the lab's reporting limit, they will be attributed with FR ('False Reporting'), see below.

2.1.2 False Repoertings (FRs)

Numerical results below the laboratory's reporting limit, are assigned as FRs ('False Reportings'). Such results should not be reported. If the analytes concerned are present in the test material, z scores are calculated for FRs as for any other numerical results. Furthermore, these results are included in the population of results for the determination of the assigned value, unless they are excluded for other reasons (e.g. reported by laboratories outside EU or EFTA countries, generated using biased methods, etc.).

2.1.3 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 10th ed., z scores for false negatives are set at "−4.0". In cases of the assigned value (see **Section 2.2**) being less than a factor of 3 times the MRRL, false negatives will typically not be assigned.

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 value xpt of each pesticide in the PT is established using the mean value of robust statistics (x^*) using Algorithm A in ISO 13528:2015 [6] of all results reported by OfLs from EU and EFTA countries. Since the assigned values of the analytes are normally derived from the respective robust mean values of the participants' results and since these results are generated by a variety of analytical methods and standards, the assigned values are metrologically not traceable. Results associated with obvious mistakes and gross errors may be excluded from the population for the establishment of the assigned values.

In the present PT, the add-in "RobStat" provided by Royal Society of Chemistry was used to calculate the assigned values with the convergence criterion = 10^{-6}.

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

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

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

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

Using the assigned value derived from the robust mean, the z scores of the participants' results are calculated using the formula in **Section 2.4.** In the present PT, all results with z scores > 5 were preliminarily regarded as outliers. If they were confirmed by Grubbs' test as outliers, they were excluded from the results population for the establishment of the assigned value, and the corresponding analyte was calculated again without those 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 numerical result for the target analyte (i) reported by the participant;
- 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).

For results regarded as false negatives, z scores are set at −4 (see **2.1.3)**. Any z scores > 5 are set at 5 in calculations of combined z scores (see **2.5.2**).

The z scores are classified as follows:

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

2.5 Laboratory Classification

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

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

2.5.1 Combined z Scores

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

The **Absolute z Scores (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, including z scores assigned for false negative results.
For the calculation, any z score > 5 is set at 5.

3. PARTICIPATION

75 official laboratories, including NRLs, from 33 countries (22 EU-Member States, 2 EFTA-countries, 1 EU candidate country and 3 other countries registered for participation in the EUPT-SRM18 and submitted at least one result. An overview of the participating laboratories and countries is given in **Table 3-1**. All laboratories having participated in this EUPT are listed in **Appendix 1**.

Several gaps in the participation of NRL-SRMs were noticed. The Swedish NRL-SRM explained its decision not to participate this time as the overlap between its own analyte scope and the Target Pesticides List was very small. The Italian NRL-SRM originally registered for the EUPT-SRM18, but finally failed to submit results due to late delivery of purchased analytical standards. The Polish NRL-SRM explained that products of animal origin, including honey, are out of its commodity scope and it has no analytical methods. The same applied to the Bulgarian NRL-SRM, which didn't participate as it only analyses commodities of plant origin. Both Bulgaria and Poland need to ensure, that the full scope of the EURL-SRM is covered by one or more NRL-SRMs. The possibility of designating a different institution as NRL-SRM for products of animal origin should be considered. As a result of Brexit the contract of the former proxy-NRL-SRM of Malta, based in UK, was terminated, and the Maltese Authorities have not yet managed to designate a new proxy-NRL-SRM. Estonia was also one of the countries with no NRL-SRM participation. The former NRL-SRM had to be closed a few months prior to the PT and a major reorganization was underway to implement the NRL-SRM tasks within the LABRIS laboratory, which took part in this PT as OfL.

In total, 108 EU-OfLs, including NRL-SRMs, regardless of their commodity scope, as well as all EU-OfLs analysing for pesticide residues in honey, were initially considered to be obliged to participate in the current EUPT. These laboratories were invited to register their participation in the present EUPT via the online registration page, or to provide an explanation for their intention not to participate. Other EU-OfLs without an obligation to participate were also invited to participate in the current PT on a voluntary basis.

In addition to the Italian NRL-SRM, which was not able to submit results due to delays in the delivery of the necessary standards as mentioned above, two of the obliged OfLs initially registered to participate, but finally withdrew due to defect instruments that prevented them from producing results in time. Another two registered obliged OfLs that also failed to report results have not given any reasons for non-submission, even after repeated requests by the organisers.

The reasons most frequently given by preliminarily obliged OfLs for their decision not to participate in the EUPT-SRM18 were the following: "PT matrix 'honey' out of routine scope of the lab"; "SRM18 target pesticides out of routine scope" and "already participated in the EUPT-AO also based on honey, with MRM-analytes in scope". During the registration period, a total of 33 obliged laboratories communicated their decision not to participate in this PT providing sufficient explanations. Considering these cases, the number of EU-OfLs considered obliged to participate in the EUPT-SRM18 decreased to 76. Among these 76 obliged laboratories finally 60 registered for the PT with 55 among them submitting at least one result. Another 16 EU- or EFTA-OfLs participated on voluntary basis.

20 out of the 75 obliged OfLs (28 %) did neither register for the PT nor provide any explanation for non-participation. These 20 laboratories originated from 12 countries as follows: ES and IT (each 3×), DE, GR, HR and RO (each 2×), as well as each one laboratory each from CZ, EE, FR, PT, SI and SK.

Table 3-1: Number of laboratories listed as being obliged to participate in the EUPT-SRM18, labs that registered to participate in the PT and submitted results (grouped by contracting country)

Contracting Country [1)]	Labs originally considered to be obliged (*based on scope and NRL-status)	Labs having provided explanations for non-participation	Obliged labs non participating w/o giving explanations	Labs finally considered being obliged to participate (taking the provided explanations into account)	Labs having registered for participation and having submitted results obliged + [on voluntary basis]		Notes
					All	NRL-SRM	
EU: NRLs and OfLs							
AT	1	0	0	1	1	1	
BE	4	2	0	2	2	1	
BE; AL	1	0	0	1	1	–	
BE; DE	1	0	0	1	1	–	
BE; FR; LU; BG	1	0	0	1	1	–	
BE; NL	1	0	0	1	1	–	
BG	3	3	0	0	0	0	The NRL-SRM of BG explained that honey, being an AO commodity, is out of its scope
CY	1	0	0	1	1	1	
CZ	4	0	1	4	2+[1]	1	
DE	16	1	2	15	10+[3]	1	
DK	2	0	1	2	1	1	
EE	3	1	1	2	0+[1]	0	The NRL-SRM of EE closed unexpectedly a few months prior to the PT. The transfer of the NRL-SRM tasks to LABRIS was underway. The latter participated in the PT as OfL.
ES	19	3	3	16	7+[3]	2	
ES; MT	1	0	0	1	1	–	
FI	2	1	0	1	1	1	
FR	6	2	1	4	1+[1]	1	
GR	5	1	2	4	2	2	
HR	5	1	0	4	2+[2]	2	
HU	4	0	2	4	2	1	
IE	2	1	0	1	1	1	
IT	14	5	3	9	6	0	The NRL-SRM of IT initially registered but had to withdraw due to delays in the delivery of necessary standards.
LT	2	0	0	2	1+[1]	1	
LU	1	0	0	1	1	1	
LV	1	0	0	1	1	1	
MT	2	0	1	2	1	0*	MT: no subcontracting proxy NRL-SRM, one lab in Germany was appointed as OfL for monitoring activities
NL	1	0	0	1	1	1	
PL	7	3	0	4	1+[3]	0	The NRL-SRM of PL explained that products of animal origin, incl. honey, are out of its commodity scope and it has no methods.
PT	3	1	1	2	1	1	
RO	4	1	2	3	1	1	
SE	2	1	0	1	1	0	The NRL-SRM of SE explained that the overlap between its scope with that of the PT was minimal.
SI	2	0	1	2	1	1	
SK	3	1	1	2	1	1	
EU Total	**124**	**28**	**22**	**96**	**54+[15]**	**24**	

Table 3-1 (cont.): Number of laboratories listed as being obliged to participate in the EUPT-SRM18, labs that registered to participate in the PT and submitted results (grouped by contracting country)

Contracting Country [1)]	Labs originally considered to be obliged (*based on scope and NRL-status)	Labs having provided explanations for non-participation	Obliged labs non participating w/o giving explanations	Labs finally considered being obliged to participate (taking the provided explanations into account)	Labs having registered for participation and having submitted results obliged + [on voluntary basis]		Notes
					All	NRL-SRM	
EFTA: NRLs and OfLs							
CH	–	–	–	–	0+[1]	0	
NO	1	0	0	1	1	1	
EU/EFTA OfLs Total	**126**	**28**	**22**	**98**	**55+[16]**	**25**	
Countries outside Europe							
RS					2		
UY					1		
UK					1		
VN					1		
Countries outside Europe Total					**5**		

4. RESULTS

4.1 Overview of Results

In addition to the compulsory and optional compounds there was an extra compound group of two analytes on the EUPT-SRM18 Target Pesticides List. The two extra compounds, bromide (anion) and copper, were left in the sample at their natural level, despite knowing that many laboratories would have difficulty in accurately analysing these compounds at these levels. Bromide was included to the Target Pesticides List after realizing that the natural level in the test material exceeded the valid MRL. In fact, the vast majority of honeys on the market exceed the bromide MRL, which obviously needs to be adjusted (please see **Section 4.4, p. 35**). On the other hand, copper was added to the Target Pesticides List despite the absence of an MRL in honey, as its inclusion was requested by DG-SANTE. The aim was to push OfLs to establish operational and logistical procedures to include copper in their scope of analytes covered in samples collected and processed for pesticide residue analysis. It was of further interest to the organisers to get informed from the participants on whether copper analyses are delegated to other laboratory sections within the same institution or even sub-contracted to other laboratories.

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. 16)** gives an overview of all results submitted by each of the participating laboratories. The individual numerical results reported by the laboratories are shown in **Table 4-8 (p. 30)**.

Table 4-1: Percentage of EU and EFTA Official Laboratories (OfLs) that have analysed for the compounds in the Target Pesticides List

Compounds		Present in Test Item	EU and EFTA OfLs analyzed for the compunds				
			Obliged OfLs only			Incl. OfLs on Voluntary Basis	
			No. [1]	Based on n = 55 [2]	Based on n = 75 [3]	No. [1]	Based on n = 71 [2]
Compulsory Compounds	**2,4-D (free acid)**	**Yes**	47	85 %	63 %	60	85 %
	BAC-C12 chloride	No	37	67 %	49 %	45	63 %
	BAC-C14 chloride	**Yes**	37	67 %	49 %	45	63 %
	Chlorate (anion)	**Yes**	38	69 %	51 %	48	68 %
	Chlormequat chloride	No	46	84 %	61 %	59	83 %
	DDAC-C10 chloride	**Yes**	37	67 %	49 %	46	65 %
	Fluazifop (free acid)	**Yes**	45	82 %	60 %	59	83 %
	Fosetyl	No	38	69 %	51 %	51	72 %
	Glyphosate	**Yes**	50	91 %	67 %	64	90 %
	Haloxyfop (free acid)	No	46	84 %	61 %	60	85 %
	Matrine	**Yes**	32	58 %	43 %	40	56 %
	Mepiquat chloride	No	45	82 %	60 %	57	80 %
	Nicotine	**Yes**	32	58 %	43 %	42	59 %
	Oxymatrine	**Yes**	28	51 %	37 %	36	51 %
	Phosphonic acid	**Yes**	35	64 %	47 %	45	63 %
Optional Compounds	**AMPA**	No	40	73 %	53 %	51	72 %
	Chloridazon desphenyl	No	18	33 %	24 %	26	37 %
	MCPA (free acid)	No	41	75 %	55 %	54	76 %
	N-Acetyl glyphosate	No	28	51 %	37 %	33	46 %
	Perchlorate	**Yes**	36	65 %	48 %	46	65 %
	Quizalofop (free acid)	No	39	71 %	52 %	52	73 %
	Trinexapac (free acid)	**Yes**	20	36 %	27 %	31	44 %
Extra	**Bromide (anion)**	**Yes**	26	47 %	35 %	34	48 %
	Copper	**Yes**	8	15 %	11 %	12	17 %

1) Laboratories representing more than one country were counted only once.
2) 71 OfLs from EU and EFTA countries (incl. NRLs) have submitted at least one result, among them 55 laboratories were obliged to participate in this PT and 16 participated on voluntary basis.
3) Taking any explanations for non-participation into account, 75 OfLs (including NRLs) from EU and EFTA countries were finally considered obliged to participate in the EUPT-SRM18.

Table 4-2: Scope and categorization of participating laboratories (including laboratories from third countries)

			Compulsory Compounds															
Compounds listed on the Target List			2,4-D (free acid)	BAC-C12 chloride	BAC-C14 chloride	Chlorate (anion)	Chlormequat chloride	DDAC-C10 chloride	Fluazifop (free acid)	Fosetyl	Glyphosate	Haloxyfop (free acid)	Matrine	Mepiquat chloride	Nicotine	Oxymatrine	Phosphonic acid	Analysed / correctly found (Compulsory Compounds): max. possible 15/10
within ...			WD-Honey; MACP.	WD-Honey	WD-Honey	WD-Honey	MACP.	WD-Honey	WD-Honey; MACP.	MACP.	WD-Honey; MACP.	MACP.	WD-Honey	MACP.	MACP. (2024+)	WD-Honey	MACP.	
Present in Test Item			**Yes**	No	**Yes**	**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	
Evaluated in this PT			**Yes**	No	**Yes**	**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	
Lab-Code SRM18-	NRL	Cat.																
2	SRM[AO]	B	V	ND	V		ND	V	V		V	ND		ND				9 / 5
3		B	V	ND	**FN**	V	ND	**FN**	V	ND	V	ND	V	ND	V	V	V	15 / 8
4		B	V	ND	V		ND	V	V		V	ND	V	ND	V	V		12 / 8
5		B	V	ND	V	V	ND	V	V	ND	V	ND	V	ND			V	13 / 8
6	SRM[AO]	B	V			V	ND		V	ND	V	ND	V	ND	V	V	V	12 / 8
7	SRM	B	V				ND		V		V	ND		ND	V			7 / 4
10		B									V							1 / 1
11	SRM	A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
12	SRM[AO]	A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
13	SRM[AO]	B	V				ND		V	ND	V	ND		ND				7 / 3
14	SRM[AO]	A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
15		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
16		B	V				ND		V	ND	V	ND		ND			V	8 / 4
17	SRM[AO]	B	V	ND	V		ND	V	V	ND	V	ND		ND			V	11 / 6
18		B	V			**FN**	ND		V	ND	V	ND	V	ND	V	V	V	12 / 7
19	SRM[AO]	B	V	ND	**FR**	V	ND	V	V	ND	V	ND		ND			V	12 / 7
20		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
21	AO	B									V							1 / 1
22		B								ND	V						V	3 / 2
23		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
24		B									V							1 / 1
25	SRM[AO]	B	V			V	ND		V	ND	V	ND		ND			V	9 / 5
26		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
27	SRM[AO]	A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V		V	14 / 9
28	SRM[AO]	B	V			V	ND		V	ND	V	ND		ND			V	9 / 5
29		B	V						V			ND						3 / 2
30		B	V	ND	V			V				ND			V			6 / 4
31		B		ND	V	V		V			V							5 / 4
32		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
33	SRM[AO]	A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
35		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10

MACP: Multiannual Control Program Regulation. Note that honey is not among the matrices of the MACP.
Link: REGULATION (EU) 2022/741 of 13 May 2022; https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32022R0741&qid=1665421580611&from=EN.
WD: Working document on pesticides to be considered for inclusion in the national control programs to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 21-22 November 2022 rev. 14(5); https://www.eurl-pesticides.eu/userfiles/file/WD/SANCO_12745_2013_rev_14_5.pdf
SRM[AO]: NRL-SRM and NRL-AO
Empty cells: not analysed; **V** = analysed for and submitted a concentration Value for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RL > assigned value; **FR** = reported result < labs' RL.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including laboratories from third countries)

				Optional Compounds								Total	Extra	
	Compounds listed on the Target List			AMPA	Chloridazon desphenyl	MCPA (free acid)	N-Acetyl glyphosate	Perchlorate	Quizalofop (free acid)	Trinexapac (free acid)	Analysed / correctly found (Optional Compounds): max. 7/2	analysed / correctly found (Compulsory + Optional Compounds): max. possible 22/12	Bromide (anion)	Copper
	within ...			WD-Annex II (future RD)	WD-Honey	WD-Annex III	WD-Annex II (future RD)	Contaminant	WD-Annex III	WD (4.1 & Annex II)			MACP.	MACP.
	present in Test Item			No	No	No	No	**Yes**	No	**Yes**			**Yes**	**Yes**
	Evaluated in this PT			No	No	No	No	**Yes**	No	**Yes**			No	No
	Lab-Code SRM18-	NRL	Cat.											
	2	SRM[AO]	B	ND		ND					2 / 0	11 / 5		
	3		B	ND	ND	ND		V	ND	**FN**	6 / 1	21 / 9		
	4		B						ND		1 / 0	13 / 8		
	5		B	ND		ND		V	ND	**FN**	5 / 1	18 / 9	*a. n. f.*	
	6	SRM[AO]	B	ND		ND	ND	V	ND	V	6 / 2	18 / 10	V	
	7	SRM	B	ND					ND		2 / 0	9 / 4		
	10		B								0 / 0	1 / 1		V
	11	SRM	A	ND	ND	ND	ND	**FN**	ND		6 / 0	21 / 10	*a. n. f.*	
	12	SRM[AO]	A	ND		ND	ND	V	ND	V	6 / 2	21 / 12		
	13	SRM[AO]	B			ND			ND	V	3 / 1	10 / 4		
	14	SRM[AO]	A	ND		ND	ND	V	ND	V	6 / 2	21 / 12	*a. n. f.*	
	15		A	ND		ND	ND		ND		4 / 0	19 / 10	*a. n. f.*	
	16		B		ND	ND			ND	**FN**	4 / 0	12 / 4	V	V
	17	SRM[AO]	B	ND		ND					2 / 0	13 / 6	*a. n. f.*	*a. n. f.*
	18		B	ND	ND	ND		V	ND		5 / 1	17 / 8		
	19	SRM[AO]	B	ND		ND	ND	V	ND		5 / 1	17 / 8		
	20		A	ND	ND	ND	ND	V	ND	V	7 / 2	22 / 12	V	V
	21	AO	B	ND							1 / 0	2 / 1		
	22		B	ND			ND				2 / 0	5 / 2		
	23		A			ND		V	ND		3 / 1	18 / 11		
	24		B	ND							1 / 0	2 / 1		
	25	SRM[AO]	B	ND		ND	ND	V			4 / 1	13 / 6		
	26		A	ND	ND	ND	ND	V	ND	**FN**	7 / 1	22 / 11	*a. n. f.*	
	27	SRM[AO]	A	ND		ND					2 / 0	16 / 9		
	28	SRM[AO]	B					V	ND		2 / 1	11 / 6		
	29		B			ND					1 / 0	4 / 2		
	30		B								0 / 0	6 / 4		
	31		B					V			1 / 1	6 / 5		
	32		A	ND	ND	ND	ND	V	ND	V	7 / 2	22 / 12	*a. n. f.*	
	33	SRM[AO]	A	ND		ND	ND	V	ND		5 / 1	20 / 11	*a. n. f.*	
	35		A	ND		ND	ND	V	ND		5 / 1	20 / 11	V	

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SRM[AO]: NRL-SRM and NRL-AO
Empty cells: not analysed; **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"; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RLs > assigned values; *a. n. f.* (= analysed, not found): Results that would be technically considered to be FNs as the lab's RL was higher than the analyte content in the sample (based on own EURL-Results). This judgement is informal and for information only given the very low levels of bromide and copper in the sample.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including laboratories from third countries)

			Compulsory Compounds															
Compounds listed on the Target List			2,4-D (free acid)	BAC-C12 chloride	BAC-C14 chloride	Chlorate (anion)	Chlormequat chloride	DDAC-C10 chloride	Fluazifop (free acid)	Fosetyl	Glyphosate	Haloxyfop (free acid)	Matrine	Mepiquat chloride	Nicotine	Oxymatrine	Phosphonic acid	Analysed / correctly found (Compulsory Compounds): max. possible 15/10
within ...			WD-Honey; MACP.	WD-Honey	WD-Honey	WD-Honey	MACP.	WD-Honey	WD-Honey; MACP.	MACP.	WD-Honey; MACP.	MACP.	WD-Honey	MACP.	MACP. (2024+)	WD-Honey	MACP.	
Present in Test Item			**Yes**	No	**Yes**	**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	
Evaluated in this PT			**Yes**	No	**Yes**	**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	
Lab-Code SRM18-	NRL	Cat.																
36		B	V	ND	V	V		V	V		V	ND						8 / 6
37		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
38		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
39		B					ND				V			ND				3 / 1
40		B	V	ND	V	V		V	V		V	ND						8 / 6
41		B	V			V	ND		V	ND	V	ND	V	ND	V	V	V	12 / 8
42		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
43		B	V				ND				V							3 / 2
44		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
45	SRM[AO]	B	V	ND	V	**FN**	ND	V	V	ND	**FN**	ND	V	ND	V	V	**FN**	15 / 7
46	SRM[AO]	B	V	ND	V		ND	V	V			ND		ND				8 / 4
47		B	V			V	ND		V	ND		ND		ND	V			8 / 4
48		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
50	SRM	B	V				ND		V	ND	V	ND		ND				7 / 3
51	SRM[AO]	B	V	ND	V	V	ND	V	V	ND	V	ND		ND	V		V	13 / 8
52		B	V			V	ND		V		V	ND	**FN**	ND				8 / 4
53		B	V				ND		V	ND	V	ND		ND				7 / 3
54		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
55	SRM[AO]	A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
57	SRM	A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
58		B		ND	**FN**	V	ND	V	**FN**	ND	V	ND			V		V	11 / 5
59		B	V	ND	V		ND	V	V			ND		ND				8 / 4
60	SRM[AO]	B					ND							ND				2 / 0
61		B	V				ND	V	V		V	ND		ND				7 / 4
62		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
63	SRM	B	V			V	ND		V	ND	V	ND	V	ND		V	V	11 / 7
64		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
65	SRM	B									V							1 / 1
66	SRM	B	V			V	ND		V	ND	V	ND	V	ND	V	V	V	12 / 8
67		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V		V	14 / 9

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SRM[AO]: NRL-SRM and NRL-AO
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Table 4-2 (cont.): Scope and categorization of participating laboratories (including laboratories from third countries)

			Optional Compounds								Total	Extra	
Compounds listed on the Target List			AMPA	Chloridazon desphenyl	MCPA (free acid)	N-Acetyl glyphosate	Perchlorate	Quizalofop (free acid)	Trinexapac (free acid)	Analysed / correctly found (Optional Compounds): max. 7/2	analysed / correctly found (Compulsory + Optional Compounds): max. possible 22/12	Bromide (anion)	Copper
within ...			WD-Annex II (future RD)	WD-Honey	WD-Annex III	WD-Annex II (future RD)	Contaminant	WD-Annex III	WD (4.1 & Annex II)			MACP.	MACP.
present in Test Item			No	No	No	No	**Yes**	No	**Yes**			**Yes**	**Yes**
Evaluated in this PT			No	No	No	No	**Yes**	No	**Yes**			No	No
Lab-Code SRM18-	NRL	Cat.											
36		B			ND					1 / 0	9 / 6		
37		A	ND	ND	ND	ND	V	ND	V	7 / 2	22 / 12	*a. n. f.*	
38		A	ND	ND	ND		V	ND	V	6 / 2	21 / 12	*a. n. f.*	V
39		B	ND				V			2 / 1	5 / 2		
40		B	ND	ND	ND	ND	V	ND		6 / 1	14 / 7		
41		B		ND	ND		V	ND	V	5 / 2	17 / 10	*a. n. f.*	
42		A	ND		ND	ND	V	ND	V	6 / 2	21 / 12	*a. n. f.*	
43		B	ND							1 / 0	4 / 2		
44		A	ND		ND	ND	V	ND		5 / 1	20 / 11	*a. n. f.*	
45	SRM^{AO}	B	ND		ND	ND	**FN**	ND	**FN**	6 / 0	21 / 7	*a. n. f.*	V
46	SRM^{AO}	B			ND			ND		2 / 0	10 / 4		
47		B			ND		V	ND	V	4 / 2	12 / 6		
48		A	ND	ND	ND		V	ND	V	6 / 2	21 / 12	*a. n. f.*	
50	SRM	B	ND		ND			ND		3 / 0	10 / 3	*a. n. f.*	V
51	SRM^{AO}	B			ND		V	ND		3 / 1	16 / 9	*a. n. f.*	
52		B	ND	ND	ND	ND	V	ND	V	7 / 2	15 / 6	*a. n. f.*	
53		B	ND	ND	ND	ND		ND	V	6 / 1	13 / 4		
54		A	ND	ND	ND	ND	V	ND	V	7 / 2	22 / 12	*a. n. f.*	V
55	SRM^{AO}	A	ND	ND	ND	ND	V	ND	V	7 / 2	22 / 12	*a. n. f.*	V
57	SRM	A	ND	ND	ND	ND	V	ND	**FN**	7 / 1	22 / 11	*a. n. f.*	
58		B					V	ND		2 / 1	13 / 6		
59		B			ND			ND		2 / 0	10 / 4		
60	SRM^{AO}	B								0 / 0	2 / 0		
61		B		ND	ND			ND	**FN**	4 / 0	11 / 4		
62		A	ND	ND	ND	ND	V	ND	V	7 / 2	22 / 12	*a. n. f.*	
63	SRM	B	ND		ND	ND	V	ND		5 / 1	16 / 8	*a. n. f.*	
64		A	ND	ND	ND	ND	**FN**	ND	V	7 / 1	22 / 11		
65	SRM	B	ND			ND				2 / 0	3 / 1		
66	SRM	B	ND			ND	V			3 / 1	15 / 9	*a. n. f.*	
67		A		ND	ND	ND	V	ND	V	6 / 2	20 / 11	*a. n. f.*	

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Table 4-2 (cont.): Scope and categorization of participating laboratories (including laboratories from third countries)

			Compulsory Compounds															
Compounds listed on the Target List			2,4-D (free acid)	BAC-C12 chloride	BAC-C14 chloride	Chlorate (anion)	Chlormequat chloride	DDAC-C10 chloride	Fluazifop (free acid)	Fosetyl	Glyphosate	Haloxyfop (free acid)	Matrine	Mepiquat chloride	Nicotine	Oxymatrine	Phosphonic acid	Analysed / correctly found (Compulsory Compounds): max. possible 15/10
within ...			WD-Honey; MACP.	WD-Honey	WD-Honey	WD-Honey	MACP.	WD-Honey	WD-Honey; MACP.	MACP.	WD-Honey; MACP.	MACP.	WD-Honey	MACP.	MACP. (2024+)	WD-Honey	MACP.	
Present in Test Item			**Yes**	No	**Yes**	**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	
Evaluated in this PT			**Yes**	No	**Yes**	**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	
Lab-Code SRM18-	NRL	Cat.																
69		B	V	ND	V	**FN**	ND	V	V	ND	V	ND	V	ND	**FN**	V	V	15 / 8
71		B	V				ND		V	ND	V	ND	V	ND	V	V		10 / 6
73		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V		14 / 9
74		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
75		B				V				ND	V						V	4 / 3
77		B		ND	**FN**			**FN**										3 / 0
78		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
79		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
80	SRM^{AO}	A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
82	SRM	A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	**FN***	V	V	15 / 9
3rd-34		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND		V	V	14 / 9
3rd-70		B	V				ND		V	ND	**FN**	ND	**FN**	ND	V	**FN**	V	11 / 4
3rd-72		A	V	ND	V	V	ND	V	V	ND	V	ND	V	ND	V	V	V	15 / 10
3rd-76		B	V			V	ND			ND	V	ND	V	ND	V	V	V	11 / 7
3rd-83		B									V							1 / 1

MACP: Multiannual Control Program Regulation. Note that honey is not among the matrices of the MACP.
Link: REGULATION (EU) 2022/741 of 13 May 2022; https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32022R0741&qid=1665421580611&from=EN.
WD: Working document on pesticides to be considered for inclusion in the national control programs to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 21-22 November 2022 rev. 14(5); https://www.eurl-pesticides.eu/userfiles/file/WD/SANCO_12745_2013_rev_14_5.pdf
SRM^{AO}: NRL-SRM and NRL-AO
Empty cells: not analysed; **V** = analysed for and submitted a concentration Value for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RL > assigned value; **FR** = reported result < labs' RL.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including laboratories from third countries)

			Optional Compounds								Total	Extra	
	Compounds listed on the Target List		AMPA	Chloridazon desphenyl	MCPA (free acid)	N-Acetyl glyphosate	Perchlorate	Quizalofop (free acid)	Trinexapac (free acid)	Analysed / correctly found (Optional Compounds): max. 7/2	analysed / correctly found (Compulsory + Optional Compounds): max. possible 22/12	Bromide (anion)	Copper
	within ...		WD-Annex II (future RD)	WD-Honey	WD-Annex III	WD-Annex II (future RD)	Contaminant	WD-Annex III	WD (4.1 & Annex II)			MACP.	MACP.
	present in Test Item		No	No	No	No	**Yes**	No	**Yes**			**Yes**	**Yes**
	Evaluated in this PT		No	No	No	No	**Yes**	No	**Yes**			No	No
	Lab-Code SRM18-	NRL											
	69		B	ND	ND	ND	ND	**FN**	ND	**FN**	7 / 0	22 / 8	V	V
	71		B	ND		ND			ND	**FN**	4 / 0	14 / 6		
	73		A	ND	ND	ND		V	ND	V	6 / 2	20 / 11	*a. n. f.*	
	74		A	ND	ND	ND	ND	V	ND	V	7 / 2	22 / 12	*a. n. f.*	*a. n. f.*
	75		B	ND				V			2 / 1	6 / 4		
	77		B								0 / 0	3 / 0		V
	78		A	ND		ND		V	ND		4 / 1	19 / 11	*a. n. f.*	
	79		A	ND	ND	ND		V	ND	**FN**	6 / 1	21 / 11	V	
	80	SRM^{AO}	A	ND	ND	ND	ND	V	ND		6 / 1	21 / 11		
	82	SRM	A	ND		ND	ND	V	ND		5 / 1	20 / 10		
	3rd-34		A	ND		ND	ND	V	ND		5 / 1	19 / 10		
	3rd-70		B	ND		ND	ND		ND		4 / 0	15 / 4	*a. n. f.*	V
	3rd-72		A	ND	ND	ND		V	ND	V	6 / 2	21 / 12	*a. n. f.*	*a. n. f.*
	3rd-76		B	ND		ND	ND	V			4 / 1	15 / 8		
	3rd-83		B								0 / 0	1 / 1		

MACP: Multiannual Control Program Regulation. Note that honey is not among the matrices of the MACP.
Link: REGULATION (EU) 2022/741 of 13 May 2022; https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32022R0741&qid=1665421580611&from=EN.
WD: Working document on pesticides to be considered for inclusion in the national control programs to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 21-22 November 2022 rev. 14(5); https://www.eurl-pesticides.eu/userfiles/file/WD/SANCO_12745_2013_rev_14_5.pdf
SRM^{AO}: NRL-SRM and NRL-AO
Empty cells: not analysed; **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"; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RLs > assigned values; *a. n. f.* (= analysed, not found): Results that would be technically considered to be FNs as the lab's RL was higher than the analyte content in the sample (based on own EURL-Results). This judgement is informal and for information only given the very low levels of bromide and copper in the sample.

4.2 Assigned Values and Target Standard Deviations

With the exception of two extra compounds ***bromide (anion)*** and ***copper***, 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 OfLs from EU and EFTA countries, excluding outliers and calculated using Algorithm A [**[5]**, **Appendix 8**]. Results from laboratories of non-EU or EFTA countries (i.e., third countries and EU candidate countries) were not taken into account. Before setting the assigned values, the results within a population of each analyte were checked for outliers based on the z scores calculated using the robust mean of the entire population and the Grubbs' test (alpha = 0.05). Results with z scores >5 or detected by Grubbs' test as outliers were excluded from the population used to establish the assigned values. After excluding the outliers, the robust mean of each analyte was recalculated using the remaining results and established as the assigned value. Among the compulsory and optional compounds, outliers were only identified in the case of ***nicotine***, with no additional outliers being identified in a second iteration. In the case of the two extra compounds ***bromide (anion)*** and ***copper***, no attempt was made to identify outliers, as only few numerical results were reported by the laboratories and it was decided a priori not to calculate z scores.

The uncertainties ($u(x_{pt})$) of the assigned values were calculated as described in **Section 2.2, p. 29,** based on the remaining population after elimination of outliers.

The assigned value (x_{pt}) of each analyte, its uncertainty ($u(x_{pt})$), the uncertainty tolerance as well as the relative standard deviation (CV^* = Coefficient of Variation) calculated applying robust statistics, are shown in **Table 4-3**. These values were calculated on the basis of the entire population, excluding outliers. The

Table 4-3: Assigned values, their uncertainties and CV^* calculated for the compulsory and optional compounds present in the test item as well as some basic figures on the "Extra" (all figures concern labs from EU and EFTA countries).

Assigned Value and CV^* Based on the Entire Population of Results from EU and EFTA Laboratories								
	Compound	No. of FNs | Outliers	No. of numerical results (EU+EFTA)	Assigned Value [mg/kg]	$u(x_{pt})$ [1] [mg/kg]	$u(x_{pt})$ Tolerance [mg/kg]	Judgement for UAV-test	CV^* [2] [%]
Compulsory	2,4-D (free acid)	0 | 0	60	0.052	± 0.0016	0.0039	passed	18.9
	BAC-C14 chloride	3 | 0	42	0.119	± 0.0044	0.0089	passed	19.0
	Chlorate (anion)	3 | 0	45	0.102	± 0.0019	0.0077	passed	9.8
	DDAC-C10 chloride	2 | 0	44	0.149	± 0.007	0.0112	passed	25.0
	Fluazifop (free acid)	1 | 0	58	0.060	± 0.0016	0.0045	passed	16.5
	Glyphosate	1 | 0	63	0.102	± 0.0021	0.0077	passed	12.8
	Matrine	1 | 0	39	0.087	± 0.0029	0.0065	passed	16.9
	Nicotine	2 | 1	40	0.087 [3]	± 0.0043	0.0065	passed	24.8
	Oxymatrine	0 | 0	36	0.068	± 0.0022	0.0051	passed	15.4
	Phosphonic acid	1 | 0	44	0.202	± 0.008	0.0152	passed	20.9
	Average [4] CV^*							**18.0**
Optional	Perchlorate	0 | 0	60	0.052	± 0.0012	0.0041	passed	18.9
	Trinexapac (free acid)	10 | 0	21	0.118	± 0.0047	0.0089	passed	14.7
	Average [4] CV^*							**16.8**
Extra	Bromide ion	*28* | –	6	not established	–	–	–	–
	Copper	*2* | –	10	not established	–	–	–	–

1: $u(x_{pt})$: Uncertainty of assigned value calculated as shown under **Section 2.2 (p. 38)**
2: CV^*: Coefficient of variation (= relative standard deviation) based on robust statistics of entire population excluding outliers,
3: In the case of nicotine, there were signs of a bimodal distribution of the results. Please refer to **Section 4.2** for the final decision for the establishment of this assigned value.
4: 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 relevant and conclusive.

uncertainty of the assigned values of all compounds (compulsory and optional) was within the tolerance, so that the assigned values can regarded as statistically reliable.

In the case of ***nicotine***, there were signs of a bimodal distribution of the results. Looking at the methodology data it was not possible to identify/isolate any subpopulation of laboratories that have used a certain method or a specific analytical step associated with bias. However, there was a notable bias between the robust mean values of the results submitted by labs correcting results for recovery (i. e. via a recovery factor or a procedural calibration or a standard addition to sample portions and/or via the use of ILIS), 0.090 mg/kg, and that of labs not correcting for recovery, 0.084 mg/kg. However, these two subgroups were rather overlapping and could not clearly explain the apparent bimodality.

As the robust mean of the entire population was relatively close to the robust mean of the population applying correction for recovery, the Scientific Committee decided that the robust mean of the whole population should be used as the assigned value based on which the z scores are derived.

Overall, the results of the present EUPT were more narrowly distributed compared to the previous EUPT-SRMs, with all CV^* values of compulsory and optional compounds remaining below the FFP-σ_{pt} of 25 %. In the case of ***chlorate (anion)***, the CV^*-value was even as low as 9.8 %. Further six compulsory compounds (***2,4-D (free acid), BAC-C14 chloride, Fluazifop (free acid), glyphosate, matrine*** and ***oxymatrine***) as well as two optional compounds (***trinexapac (free acid)*** and ***perchlorate***) had CV^*-values between 10 % and 20 %. For the remaining three compulsory compounds (***phosphonic acid, nicotine*** and ***DDAC-C10 chloride)***) the CV^*-values ranged between 20 % and 25 %. This overall good performance is surely related to the fact that honey poses relatively few analytical problems for most of the analytes present in the test material.

As regards the extra analytes ***bromide ion*** and ***copper***, the number of numerical results submitted by the participants was low and the results were broadly distributed. This was expected to some extent, as these two analytes were present at their natural levels. Overall, 34 OfLs form EU- or EFTA-countires reported that they analysed for ***bromide ion***, but 28 laboratories thereof reported that ***bromide*** was not detected in the test material. Only one of these 28 laboratories had a RL for ***bromide ion*** (0.05 mg/kg) that was close to the estimated concentration range for this compound of 0.04 – 0.05 mg/kg (based on results by the participants and the organisers), while the RLs of the remaining 27 laboratories ranged much higher between 0.2 mg/kg and 5 mg/kg. In practice, such results would not be regarded as false negatives. It is highlighted that the concentration of bromide exceeded the current MRL (see discussion this regarding under **Section 4.4, p. 35**).

In the case of ***copper***, 12 OfLs from EU- or EFTA-countries analysed for this compound, with only two laboratories reporting numerical results. Nevertheless, the main objective relating to ***copper***, which was to trigger the OfLs to focus on copper analysis, was largely achieved. Another objective achieved was the collection of information on analytical methods used in laboratories in preparation for EUPT-SRM19 and subsequent EUPTs, which will continue to focus on ***copper***.

No assigned values were calculated for both extra analytes, and the laboratory performance (in terms of z scores) was not assessed. Where laboratories reported 'analysed but not detected' for ***copper***, with their RLs being lower than the estimated copper concentration range in the honey sample (which based on the results by the participants and the organisers was roughly between 0.03 mg/kg and 0.05 mg/kg), the code "a.n.f" (for analyzed, not found) was entered in **Table 4-2 (p. 16)** for information.

4.3 Assessment of Laboratory Performance

4.3.1 False Positives and False Reportings

There were no false positive results in the EUPT-SRM18.

Lab SRM18-19 reported for ***BAC-C14 chloride*** a numerical value below the lab's RL. This result should not be reported and was regarded as a False Reporting (FR).

4.3.2 False Negatives

These concerned cases where a compound was present in the test item at a relevant level and where this compound was analysed by a laboratory but reporting 'not detected' (even in the case where RL > AV) or reporting a concentration that was lower than its own RL or the MRRL. In 27 of those cases, the assigned values were higher than the laboratories' RLs, therefore, they were judged as FNs. Following the General Protocol (10th Ed.) the z scores for FNs were set at −4. The FN results concerned the following analytes: ***trinexapac*** (10×), ***perchlorate*** (4×), ***BAC-C14*** (3×), ***chlorate*** (3×), ***DDAC-C10*** (2×) and one case each for ***fluazifop, glyphosate, matrine, nicotine*** and ***phosphonic acid***. In one case, Lab SRM18-82 had targeted ***nicotine*** and not detected this compound with its RL (0.1 mg/kg) being higher than the assigned value (0.0868 mg/kg). In accordance with the General Protocol(**Appendix 8**), this result was judged as a false negative result but marked with a note. This lab is encouraged to improve its analytical sensitivity for this compound.

Noticeable was the high number of FNs (10×) in the case of ***trinexapac***. Four of the concerned laboratories (SRM18-5, -16, -57 and -79) reported that they had mistakenly targeted "trinexapac-ethyl" instead of ***trinexapac (acid)***. Further two labs (SRM18-69 and -71) indicated that their statement of having analysed for ***trinexapac*** was actually wrong as the compound is not part of their scope, but didn't explicitly state that they have targeted trinexapac-ethyl instead. Among the other four labs with FNs for ***trinexapac,*** lab SRM18-45 stated "lack of experience" and lab SRM18-69 „transcription error" as reasons for their FN results. The remaining two laboratories (SRM18-3 and SRM18-26) didn't provide any reasons. The organisers urge the labs SRM18-3, -26, -45, -61, -69 and -71 to check whether the name mismatching was finally the reason for their FN result. Additional information concerning the mismatching of ***trinexapac*** and trinexapac-ethyl can be found in **Section 4.4, p. 35**.

Furthermore, 28 laboratories analysed for ***bromide ion*** and 2 laboratories for ***copper*** without detecting it. Both analytes were present in the test material at their natural content, which was quite low compared to the RLs of the participating laboratories. The two compounds were therefore classified as "extra" analytes and were not to be included in the official lab performance assesment. In addition, no MRRL was set for these two analytes. Technically, only the ***bromide*** result of lab SRM18-67 could be judged as a FN as its RL was lower than the estimated content of ***bromide*** in the sample. In the case of ***copper***, the two laboratories not detecting copper have indicated RLs that were higher than the estimated content of copper in the test material. Further information on bromide and copper can be found in **Section 4.4, p. 35**.

All false negative results in the EUPT-SRM18 were listed in **Table 4-4, p. 25.** The reasons reported by the laboratories to explain their performance are compiled in **Appendix 7**. It was also checked whether certain laboratories had reported false negative results for many compounds and especially for related compounds, as this could be an indication for a systematic bad practice. Especially noteworthy were the six FN results of laboratory SRM18-45, which happens to be an NRL-SRM, the four FNs of lab SRM18-69, and the three FNs each of SRM18-70 and SRM18-3. The organisers urge these laboratories to introduce QC measures, such as calibrations at low levels and the checking for matrix effects to avoid false negatives in the future.

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

	Compounds	MRRL [mg/kg]	Assigned Value [mg/kg]	PT-Code (SRM18-)	Analysed	Detected	RL‡ [mg/kg]	Judgement
Compulsory	BAC-C14 chloride	0.01	0.119	3	Yes	No	0.01	FN
				58	Yes	No	0.01	FN
				77	Yes	No	0.01	FN
	Chlorate (anion)	0.01	0.102	18	Yes	No	0.01	FN
				45	Yes	No	0.01	FN
				69	Yes	No	0.01	FN
	DDAC-C10 chloride	0.01	0.149	3	Yes	No	0.01	FN
				77	Yes	No	0.01	FN
	Fluazifop (free acid)	0.01	0.0598	58	Yes	No	0.01	FN
	Glyphosate	0.01	0.102	45	Yes	No	0.01	FN
	Matrine	0.01	0.0873	52	Yes	No	0.05	FN
	Nicotine	0.01	0.0868	69	Yes	No	0.01	FN
				82	Yes	No	0.1	FN* (RL > AV)
	Phosphonic acid	0.03	0.202	45	Yes	No	0.03	FN
Optional	Perchlorate	0.01	0.0550	11	Yes	No	0.01	FN
				45	Yes	No	0.01	FN
				64	Yes	No	0.01	FN
				69	Yes	No	0.01	FN
	Trinexapac (free acid)	0.01	0.118	3	Yes	No	0.01	FN
				5	Yes	No	0.01	FN
				16	Yes	No	0.01	FN
				26	Yes	No	0.01	FN
				45	Yes	No	0.01	FN
				57	Yes	No	0.01	FN
				61	Yes	No	0.01	FN
				69	Yes	No	0.01	FN
				71	Yes	No	0.01	FN
				79	Yes	No	0.01	FN
Extra	Bromide (anion)	–	not established (pls. see p. 23)	5	Yes	No	0.5	*a. n. f.*
				11	Yes	No	0.5	*a. n. f.*
				14	Yes	No	1	*a. n. f.*
				15	Yes	No	0.5	*a. n. f.*
				17	Yes	No	0.5	*a. n. f.*
				26	Yes	No	5	*a. n. f.*
				32	Yes	No	2	*a. n. f.*
				33	Yes	No	0.5	*a. n. f.*
				37	Yes	No	1	*a. n. f.*
				38	Yes	No	0.5	*a. n. f.*
				41	Yes	No	0.2	*a. n. f.*
				42	Yes	No	0.5	*a. n. f.*
				44	Yes	No	0.5	*a. n. f.*
				45	Yes	No	0.5	*a. n. f.*
				48	Yes	No	1	*a. n. f.*
				50	Yes	No	2.5	*a. n. f.*
				51	Yes	No	0.5	*a. n. f.*
				52	Yes	No	2.5	*a. n. f.*
				54	Yes	No	0.5	*a. n. f.*
				55	Yes	No	0.5	*a. n. f.*
				57	Yes	No	0.5	*a. n. f.*
				62	Yes	No	0.5	*a. n. f.*
				63	Yes	No	0.5	*a. n. f.*
				66	Yes	No	0.5	*a. n. f.*
				67	Yes	No	0.05	*a. n. f.*
				73	Yes	No	0.5	*a. n. f.*
				74	Yes	No	1	*a. n. f.*
				78	Yes	No	0.5	*a. n. f.*
	Copper	–	not established (pls. see p. 23)	17	Yes	No	0.5	*a. n. f.*
				74	Yes	No	1	*a. n. f.*

‡ as reported by the participants
* Lab's RL >> Assigned value, so that this lab could not determine this compound.
a. n. f. = analysed, not found: analysed for but not detected compounds present in the test material at their natural content, and belonging to the extra compounds for which no assigned value was established. See p. 23 roughly estimated conc. range.

4.3.3 Laboratory Classification Based on Scope

All participating laboratories that reported at least one result were classified into categories A or B according to the rules given in **Section 2.5 (p. 11)** and in the General Protocol (10th Edition, see **Appendix 8**). According to these rules, a laboratory had to fulfil the following conditions in order to be classified into Category A in the present PT: a) analysis of at least 13 out of the 15 compulsory pesticides on the Target Pesticides List; b) correct detection of at least nine out of the ten compulsory pesticides present in the test item, and c) no false positive results.

A total of 28 EU and EFTA laboratories (39 %) were classified into Category A and 43 (61 %) into Category B. Two of the five laboratories (40 %) from one EU candidate country or third countries were classified into Category A.

Considering the compulsory compounds only, the laboratories from EU and EFTA countries classified into Category A achieved an overall AAZ of 0.5 (n = 252), whereas those classified into Category B achieved

Table 4-5: Category A laboratories in EUPT-SRM18, ordered by lab-codes. For the AAZ calculation any z scores > 5 were set at 5.

		COMPULSORY Compounds	2,4-D (free acid)	BAC-C14 chloride	Chlorate (anion)	DDAC-C10 chloride	Fluazifop (free acid)	Glypho-sate	Matrine	Nicotine	Oxy-matrine	Phospho-nic acid	
		Assigned Value [mg/kg]	0.052	0.119	0.102	0.149	0.060	0.102	0.087	0.087	0.068	0.202	
		*CV**	18.9%	19.0%	9.8%	25.0%	16.5%	12.8%	16.9%	24.8%	15.4%	20.9%	
		MRRL [mg/kg]	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.03	
Lab code SRM18-	NRL	Analysed / corr. found, max. 15 / 10	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	AAZ
11	**SRM**	15 / 10	0.4	1.6	0.7	1.1	-1.3	-0.3	1.3	1.2	0.8	0.5	0.9
12	**SRM/AO**	15 / 10	-0.6	0.4	0.2	-0.3	0.3	-0.1	0.8	1.0	1.0	-0.1	0.5
14	**SRM/AO**	15 / 10	0.4	-0.5	0.0	-0.4	-0.2	-0.1	0.2	0.4	0.6	1.9	0.5
15		15 / 10	0.2	-0.6	0.0	-0.6	0.1	-0.4	-0.3	0.4	-0.3	1.1	0.4
20		15 / 10	-0.9	0.3	0.0	-0.4	-0.1	-0.1	-0.2	0.5	0.8	0.2	0.4
23		15 / 10	-0.2	0.0	0.6	0.2	-0.3	-0.3	0.4	-1.2	0.5	-0.3	0.4
26		15 / 10	0.1	-2.7	-0.4	-2.0	-0.7	0.1	0.2	0.7	0.0	-0.2	0.7
27	**SRM/AO**	14 / 9	1.8	0.1	-0.1	0.1	0.6	0.3	-0.6	-0.5		1.6	0.6
32		15 / 10	0.1	-0.4	-0.2	-1.0	-0.2	0.2	0.0	0.2	-0.1	0.0	0.2
33	**SRM/AO**	15 / 10	0.2	0.5	0.2	-0.7	0.1	-0.1	0.6	0.7	1.1	0.0	0.4
35		15 / 10	1.1	-0.6	0.7	-0.9	0.6	0.6	-0.5	-0.6	-1.0	-1.3	0.8
37		15 / 10	-0.1	-0.7	-0.4	-0.1	-0.6	0.1	0.5	-1.2	0.7	-0.3	0.5
38		15 / 10	0.1	-0.4	-0.5	0.2	0.0	0.2	1.0	1.4	-0.1	-0.6	0.5
42		15 / 10	-0.3	-0.3	-0.1	-0.8	-0.5	-0.1	-0.1	-0.6	-0.6	-0.2	0.4
44		15 / 10	0.6	0.1	-0.1	0.6	0.5	0.6	0.4	0.1	0.1	1.0	0.4
48		15 / 10	1.2	1.1	0.1	0.7	1.3	0.3	-0.2	-1.2	0.1	-0.1	0.6
54		15 / 10	-1.2	-0.1	0.2	0.2	-0.8	-0.2	0.7	0.0	-0.5	0.1	0.4
55	**SRM/AO**	15 / 10	0.2	0.8	0.1	0.6	0.3	-0.4	0.2	0.7	-0.1	-0.2	0.4
57	**SRM**	15 / 10	-0.8	-2.1	0.0	-2.1	-0.1	-0.4	-1.0	-1.4	-0.4	-0.1	0.8
62		15 / 10	-0.2	0.1	0.0	0.5	0.0	-0.4	-0.1	0.3	0.0	-0.6	0.2
64		15 / 10	-0.3	-0.1	0.2	0.2	0.1	0.0	-0.7	0.3	-0.1	0.8	0.3
67		14 / 9	-0.1	0.2	0.0	1.0	0.0	0.5	-0.1	0.3		1.1	0.4
73		14 / 9	0.1	-0.2	-0.3	0.0	-0.4	-0.4	-0.6	-1.9	-0.1		0.4
74		15 / 10	-0.2	-0.4	0.0	-1.2	0.0	-0.1	0.6	0.5	-0.7	0.0	0.4
78		15 / 10	-0.6	-0.4	-0.7	0.5	-0.7	-0.1	0.3	1.0	0.1	0.1	0.5
79		15 / 10	0.7	-0.1	-0.2	0.2	0.8	0.1	-1.3	0.6	0.6	-0.4	0.5
80	**SRM/AO**	15 / 10	-0.1	0.4	0.0	0.0	-0.2	-0.2	-0.2	0.7	0.1	1.0	0.3
82	**SRM**	15 / 9	0.2	0.3	0.5	0.2	0.0	0.6	0.3	-FN	0.5	-0.2	0.7
3rd-34		14 / 9	-0.4	0.9	-0.5	0.4	-0.1	-0.7	-0.7		-1.0	-0.6	0.6
3rd-72		15 / 10	-0.1	0.6	-0.3	-0.3	0.6	-0.3	-0.1	-0.4	-0.1	-0.9	0.4

Table 4-6: Category B laboratories in EUPT-SRM18, ordered by lab-codes. Tha AAZs were calculated for laboratories having submitted at least 5 results for the compulsory compounds present in the test material.

COMPULSORY Compounds			2,4-D (free acid)	BAC-C14 chloride	Chlorate (anion)	DDAC-C10 chloride	Fluazifop (free acid)	Glypho-sate	Matrine	Nicotine	Oxy-matrine	Phospho-nic acid	
Assigned Value [mg/kg]			0.052	0.119	0.102	0.149	0.060	0.102	0.087	0.087	0.068	0.202	
*CV**			18.9%	19.0%	9.8%	25.0%	16.5%	12.8%	16.9%	24.8%	15.4%	20.9%	
MRRL [mg/kg]			0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.03	
Lab code SRM18-	NRL	Analysed / corr. found, max. 15 / 10	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	z score (FFP-RSD = 25 %)	AAZ
2	**SRM/AO**	9 / 5	-1.1	0.3		0.7	0.5	-0.1					0.5
3		15 / 8	1.0	-FN	-0.9	-FN	-0.1	1.1	-0.2	0.4	0.2	0.2	1.2
4		12 / 8	-1.9	0.2		1.4	-1.5	0.3	-0.4	-0.9	0.0		0.8
5		13 / 8	-1.4	-1.5	0.5	-1.5	-0.9	2.0	-0.3			0.0	1.0
6	**SRM/AO**	12 / 8	-0.1		0.2		0.7	-0.4	1.5	0.5	0.6	0.4	0.6
7	**SRM**	7 / 4	0.4				-0.1	0.3		-1.3			--
10		1 / 1						3.8					--
13	**SRM/AO**	7 / 3	-0.1				0.9	0.2					--
16		8 / 4	-0.3				0.7	0.0				-0.5	--
17	**SRM/AO**	11 / 6	0.6	-1.1		-1.4	0.5	-0.2				-2.0	1.0
18		12 / 7	-0.2		-FN		0.0	0.7	-0.3	-1.2	-1.1	0.2	1.0
19	**SRM/AO**	12 / 7	-0.4	-2.7	-0.4	-2.5	-0.1	-1.0				-0.3	1.1
21	**AO**	1 / 1						0.0					--
22		3 / 2						-0.4				0.4	--
24		1 / 1						-0.9					--
25	**SRM/AO**	9 / 5	1.3		1.5		-0.9	0.4				-0.4	0.9
28	**SRM/AO**	9 / 5	1.2		-0.9		0.6	-1.4				-1.6	1.1
29		3 / 2	1.1				4.6						--
30		6 / 4	0.0	1.5		6.0				11.9			--
31		5 / 4		-0.5	0.1	-0.3		FN					--
36		8 / 6	-0.2	-0.1	0.1	-0.2	-0.5	-0.2					0.2
39		3 / 1						-0.2					--
40		8 / 6	-0.2	-0.1	-0.4	0.7	-0.6	-1.1					0.5
41		12 / 8	0.2		0.0		0.8	0.6	-1.0	-1.4	-0.2	-0.5	0.6
43		3 / 2	-0.6					1.0					--
45	**SRM/AO**	15 / 7	0.6	0.5	-FN	0.6	-0.2	-FN	-1.7	-0.4	-2.1	-FN	1.8
46	**SRM/AO**	8 / 4	-1.4	4.8		1.7	-1.1						--
47		8 / 4	-1.3		0.3		-1.1			0.9			--
50	**SRM**	7 / 3	0.1				0.1	-0.5					--
51	**SRM/AO**	13 / 8	0.5	0.1	0.4	-0.9	0.8	-0.4		-0.5		-1.1	0.6
52		8 / 4	0.7		0.0		0.3	0.2	-FN				1.0
53		7 / 3	0.4				-0.3	0.2					--
58		11 / 5		-FN	7.2	1.3	-FN	5.9		4.3		5.3	4.1
59		8 / 4	1.4	1.0		1.5	0.8						--
61		7 / 4	-1.9			-0.8	-0.6	-0.7					--
63		11 / 7	-1.0		-0.4		-0.7	-1.5	-0.6		-0.8	-1.7	1.0
65		1 / 1						-0.2					--
66		12 / 8	-0.1		0.2		0.1	0.2	0.7	0.5	0.2	5.5	0.9
69		15 / 8	-0.1	1.3	-FN	0.0	0.2	-1.1	0.7	-FN	-0.1	0.4	1.2
71		10 / 6	0.0				3.5	-0.5	-0.1	-0.7	-0.8		0.9
75		4 / 3			-0.7			-0.1				-1.7	--
77		3 / 0		-FN		-FN							--
3rd-70		11 / 4	0.1				0.1	-FN	-FN	2.1	-FN	-0.3	2.1
3rd-76		11 / 7	-1.1		0.2			-0.2	3.0	0.4	2.6	-0.4	1.1
3rd-83		1 / 1						2.4					--

an overall AAZ of 1.1 (n = 208). The two laboratories from EU candidate and/or third countries classified into Category A achieved an overall AAZ of 0.5 (n = 19), whereas the other three classified into Category B achieved an overall AAZ of 1.7 with only 15 results.

Table 4-5 (p. 26) and **Table 4-6 (p. 27)** show the details of laboratories classified into Category A and B, respectively. For informative purposes, the overall AAZs were calculated for laboratories with 5 or more individual z scores among the compulsory compounds. For the AAZ calculation, any z scores > 5 were set at 5.

4.3.4 Laboratory Performance Based on z Scores

As described before, no assessment was carried out for the two extra compounds ***bromide ion*** and ***copper***. For all other compulsory and optional compounds, the individual z scores were calculated using the FFP-RSD of 25 % and the assigned values derived from the entire population of EU/EFTA OfLs excluding outliers and using robust statistics. **Table 4-7 (p. 29)** shows the overall classification of z scores achieved by all laboratories for compulsory and optional compounds based on the rules given in **Section 2.4 (p. 31)**.

91 % of the results reported by EU/EFTA-OfLs were classified as "acceptable". The acceptability rate ranged from 65 % for ***trinexapac (free acid)*** to 100 % for ***2,4-D (free acid)***. Among the compulsory compounds, 93 % of the reported results were "acceptable". For all analytes but ***BAC-C14 chloride*** (84 %) and ***DDAC-C10 chloride*** (89 %) the percentage of "acceptable" z scores exceeded the 90 % mark, ranging from 90 % for ***nicotine*** to 100 % for ***2,4-D (free acid)***. Among the optional compounds, 78 % of the reported results were classified as "acceptable" (i.e. 65 % in the case of ***trinexapac (free acid)*** and 87 % in the case of ***perchlorate***). It is interesting to note that in the case of ***trinexapac (free acid)*** 10 of the 11 z scores that were not classified as acceptable were FNs, with only one questionable result reported. As a result, the CV^* value of this population was still very satisfactory at 14.7 %.

A compilation of all individual results and z scores for each laboratory is shown in **Table 4-8 (p. 30)** for compulsory, optional and extra compounds persent in the test material. The corresponding kernel density histograms showing the distribution of the reported results for the compulsory and optional compounds 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**.

(continoued on p. 34)

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

EU and EFTA Official Laboratories								
	Compounds	No. of results[1]	Acceptable No. (%)	Questionable No. (%)	Unacceptable[1] No. (%)	FNs	AAZ (incl. FNs)	AAZ (excl. FNs)
Compulsory	2,4-D (free acid)	60	60 (100%)	(0%)	0 (0%)	0	0.6	0.6
	BAC-C14 chloride	45	38 (84%)	3 (7%)	4 (9%)	3	1.0	0.7
	Chlorate (anion)	48	44 (92%)	(0%)	4 (8%)	3	0.6	0.4
	DDAC-C10 chloride	46	41 (89%)	2 (4%)	3 (7%)	2	1.0	0.8
	Fluazifop (free acid)	59	56 (95%)	(0%)	3 (5%)	1	0.7	0.6
	Glyphosate	64	60 (94%)	(0%)	4 (6%)	1	0.7	0.6
	Matrine	40	39 (98%)	(0%)	1 (3%)	1	0.6	0.5
	Nicotine	42	38 (90%)	(0%)	4 (10%)	2	1.1	0.9
	Oxymatrine	36	35 (97%)	1 (3%)	0 (0%)	0	0.5	0.5
	Phosphonic acid	45	42 (93%)	(0%)	3 (7%)	1	0.9	0.8
	Subtotal	**485**	**453 (93%)**	**6 (1%)**	**26 (5%)**	**14**	**0.8**	**0.7**
Optional	Perchlorate	46	40 (87%)	(0%)	6 (13%)	4	0.9	0.6
	Trinexapac (free acid)	31	20 (65%)	1 (3%)	10 (32%)	10	1.6	0.5
	Subtotal	**77**	**60 (78%)**	**1 (1%)**	**2 (3%)**	**14**	**1.3**	
Overall EU/EFTA		**562**	**513 (91%)**	**7 (1%)**	**14 (2%)**	**28 (5%)**	**0.9**	**0.6**

	Compounds	No. of analysed[2]	Reporting Num. Result	Not Reporting Num. Result
Extra	Bromide ion	34	6	28
	Copper	12	10	2
	Subtotal	**46**	**16**	**30**

Third Country / EU Candidate Country Laboratories								
	Compounds	No. of results[1]	Acceptable No. (%)	Questionable No. (%)	Unacceptable[1] No. (%)	FNs	AAZ (incl. FNs)	AAZ (excl. FNs)
Compulsory	2,4-D (free acid)	4	4 (100%)	(0%)	0 (0%)	0	–	–
	BAC-C14 chloride	2	2 (100%)	(0%)	0 (0%)	0	–	–
	Chlorate (anion)	3	3 (100%)	(0%)	0 (0%)	0	–	–
	DDAC-C10 chloride	2	2 (100%)	(0%)	0 (0%)	0	–	–
	Fluazifop (free acid)	3	3 (100%)	(0%)	0 (0%)	0	–	–
	Glyphosate	5	3 (60%)	1 (20%)	1 (20%)	1	1.5	0.9
	Matrine	4	2 (50%)	(0%)	2 (50%)	1	–	–
	Nicotine	3	2 (67%)	1 (33%)	0 (0%)	0	–	–
	Oxymatrine	4	2 (50%)	1 (25%)	1 (25%)	1	–	–
	Phosphonic acid	4	4 (100%)	(0%)	0 (0%)	0	–	–
	Subtotal	**34**	**27 (79%)**	**3 (9%)**	**4 (12%)**	**3**	–	–
Optional	Perchlorate	3	3 (100%)	(0%)	0 (0%)	0	–	–
	Trinexapac (free acid)	1	1 (100%)	(0%)	0 (0%)	0	–	–
	Subtotal	**4**	**4 (100%)**	**(0%)**	**0 (0%)**	**0**	–	–
Overall EU/EFTA		**38**	**31 (82%)**	**3 (8%)**	**4 (11%)**	**3 (8%)**	**–**	**–**

	Compounds	No. of analysed[2]	Reporting Num. Result	Not Reporting Num. Result
Extra	Bromide ion	2	0	2
	Copper	2	1	1
	Subtotal	**4**	**1**	**3**

1) including false negatives (FNs),
2) Number of laboratories having reported "analysed" for this compound

Table 4-8: Results reported and z scores achieved by all participating laboratories for the compounds present in the test material

				Compulsory													
Compounds				2,4-D (free acid)		BAC-C14 chloride		Chlorate (anion)		DDAC-C10 chloride		Fluazifop (free acid)		Glyphosate		Matrine	
Assigned Value [mg/kg]				0.052		0.119		0.102		0.149		0.060		0.102		0.087	
*CV**				18.9%		19.0%		9.8%		25.0%		16.5%		12.8%		16.9%	
MRRL [mg/kg]				0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Lab code SRM18-	NRL	Cat	Analysed / corr. found, max. 15 / 10	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)
2	SRM/AO	B	9 / 5	0.038	-1.1	0.129	0.3			0.176	0.7	0.068	0.5	0.100	-0.1		
3		B	15 / 8	0.066	1.0	FN	-FN	0.079	-0.9	FN	-FN	0.058	-0.1	0.13	1.1	0.084	-0.2
4		B	12 / 8	0.028	-1.9	0.126	0.2			0.200	1.4	0.037	-1.5	0.11	0.3	0.078	-0.4
5		B	13 / 8	0.0341	-1.4	0.0757	-1.5	0.115	0.5	0.0947	-1.5	0.0462	-0.9	0.154	2.0	0.0801	-0.3
6	SRM/AO	B	12 / 8	0.0510	-0.1			0.106	0.2			0.0700	0.7	0.0930	-0.4	0.120	1.5
7	SRM	B	7 / 4	0.057	0.4							0.058	-0.1	0.11	0.3		
10		B	1 / 1											0.198	3.8		
11	SRM	A	15 / 10	0.0572	0.4	0.167	1.6	0.120	0.7	0.190	1.1	0.0403	-1.3	0.0935	-0.3	0.115	1.3
12	SRM/AO	A	15 / 10	0.044	-0.6	0.131	0.4	0.107	0.2	0.138	-0.3	0.064	0.3	0.099	-0.1	0.105	0.8
13	SRM/AO	B	7 / 3	0.0509	-0.1							0.0733	0.9	0.107	0.2		
14	SRM/AO	A	15 / 10	0.0576	0.4	0.104	-0.5	0.102	0.0	0.135	-0.4	0.0567	-0.2	0.100	-0.1	0.0927	0.2
15		A	15 / 10	0.0551	0.2	0.101	-0.6	0.103	0.0	0.126	-0.6	0.0612	0.1	0.0921	-0.4	0.0802	-0.3
16		B	8 / 4	0.048	-0.3							0.070	0.7	0.103	0.0		
17	SRM/AO	B	11 / 6	0.0598	0.6	0.0849	-1.1			0.0986	-1.4	0.0676	0.5	0.0975	-0.2		
18		B	12 / 7	0.05	-0.2			FN	-FN			0.06	0.0	0.12	0.7	0.08	-0.3
19	SRM/AO	B	12 / 7	0.0474	-0.4	0.0389	-2.7	0.0910	-0.4	0.0567	-2.5	0.0584	-0.1	0.0773	-1.0		
20		A	15 / 10	0.041	-0.9	0.129	0.3	0.101	0.0	0.134	-0.4	0.058	-0.1	0.100	-0.1	0.082	-0.2
21	AO	B	1 / 1											0.103	0.0		
22		B	3 / 2											0.093	-0.4		
23		A	15 / 10	0.050	-0.2	0.118	0.0	0.117	0.6	0.155	0.2	0.055	-0.3	0.095	-0.3	0.097	0.4
24		B	1 / 1											0.0797	-0.9		
25	SRM/AO	B	9 / 5	0.069	1.3			0.139	1.5			0.047	-0.9	0.112	0.4		
26		A	15 / 10	0.054	0.1	0.040	-2.7	0.092	-0.4	0.074	-2.0	0.049	-0.7	0.104	0.1	0.091	0.2
27	SRM/AO	A	14 / 9	0.0756	1.8	0.122	0.1	0.100	-0.1	0.153	0.1	0.0682	0.6	0.110	0.3	0.0743	-0.6
28	SRM/AO	B	9 / 5	0.0681	1.2			0.0803	-0.9			0.0684	0.6	0.0671	-1.4		
29		B	3 / 2	0.067	1.1							0.128	4.6				
30		B	6 / 4	0.052	0.0	0.164	1.5			0.372	6.0						
31		B	5 / 4			0.104	-0.5	0.105	0.1	0.137	-0.3			0.203	FN		
32		A	15 / 10	0.054	0.1	0.108	-0.4	0.097	-0.2	0.110	-1.0	0.057	-0.2	0.108	0.2	0.087	0.0
33	SRM/AO	A	15 / 10	0.055	0.2	0.135	0.5	0.108	0.2	0.123	-0.7	0.061	0.1	0.100	-0.1	0.100	0.6
35		A	15 / 10	0.0664	1.1	0.102	-0.6	0.121	0.7	0.116	-0.9	0.0688	0.6	0.117	0.6	0.0763	-0.5
36		B	8 / 6	0.0502	-0.2	0.115	-0.1	0.105	0.1	0.142	-0.2	0.0529	-0.5	0.0971	-0.2		
37		A	15 / 10	0.051	-0.1	0.097	-0.7	0.093	-0.4	0.147	-0.1	0.051	-0.6	0.104	0.1	0.099	0.5
38		A	15 / 10	0.054	0.1	0.108	-0.4	0.089	-0.5	0.157	0.2	0.060	0.0	0.106	0.2	0.110	1.0
39		B	3 / 1											0.097	-0.2		
40		B	8 / 6	0.0501	-0.2	0.116	-0.1	0.0914	-0.4	0.174	0.7	0.0505	-0.6	0.0748	-1.1		
41		B	12 / 8	0.0547	0.2			0.102	0.0			0.0720	0.8	0.117	0.6	0.0651	-1.0
42		A	15 / 10	0.048	-0.3	0.11	-0.3	0.1	-0.1	0.12	-0.8	0.053	-0.5	0.1	-0.1	0.085	-0.1
43		B	3 / 2	0.0441	-0.6									0.127	1.0		
44		A	15 / 10	0.060	0.6	0.122	0.1	0.099	-0.1	0.173	0.6	0.068	0.5	0.118	0.6	0.095	0.4
45	SRM/AO	B	15 / 7	0.060	0.6	0.134	0.5	FN	-FN	0.173	0.6	0.057	-0.2	FN	-FN	0.050	-1.7
46	SRM/AO	B	8 / 4	0.0345	-1.4	0.263	4.8			0.211	1.7	0.0428	-1.1				
47		B	8 / 4	0.0351	-1.3			0.109	0.3			0.0440	-1.1				

				Compulsory							Optional				Extra	
			Compounds	Nicotine		Oxymatrine		Phosphonic acid		Compounds	Perchlorate		Trinexapac (free acid)		Bromide (anion)	Copper
			Assigned Value [mg/kg]	0.087		0.068		0.202		AV [mg/kg]	0.055		0.118		not assessed	
			*CV**	24.8%		15.4%		20.9%		CV*	11.6%		14.7%		not assessed	
			MRRL [mg/kg]	0.01		0.01		0.03			0.01		0.01		none	none
	Lab code SRM18-	NRL	Cat	Analysed / corr. found, max. 15 / 2	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Analysed / corr. found, max. 7 / 2	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	
	2	SRM/AO	B	9 / 5							2 / 0						
	3		B	15 / 8	0.095	0.4	0.072	0.2	0.21	0.2	6 / 1	0.051	-0.3	FN	-FN		
	4		B	12 / 8	0.067	-0.9	0.068	0.0			1 / 0						
	5		B	13 / 8					0.201	0.0	5 / 1	0.0530	-0.1	FN	-FN	*a. n. f.*	
	6	SRM/AO	B	12 / 8	0.0985	0.5	0.0784	0.6	0.223	0.4	6 / 2	0.0510	-0.3	0.136	0.6	0.943	
	7	SRM	B	7 / 4	0.058	-1.3					2 / 0						
	10		B	1 / 1							0 / 0						0.49
	11	SRM	A	15 / 10	0.113	1.2	0.0809	0.8	0.225	0.5	6 / 0	FN	-FN			*a. n. f.*	
	12	SRM/AO	A	15 / 10	0.108	1.0	0.085	1.0	0.197	-0.1	6 / 2	0.071	1.2	0.121	0.1		
	13	SRM/AO	B	7 / 3							3 / 1			0.118	0.0		
	14	SRM/AO	A	15 / 10	0.0950	0.4	0.0786	0.6	0.300	1.9	6 / 2	0.0627	0.6	0.121	0.1	*a. n. f.*	
	15		A	15 / 10	0.0951	0.4	0.0638	-0.3	0.259	1.1	4 / 0					*a. n. f.*	
	16		B	8 / 4					0.176	-0.5	4 / 0			FN	-FN	0.065	0.05
	17	SRM/AO	B	11 / 6					0.100	-2.0	2 / 0					*a. n. f.*	*a. n. f.*
	18		B	12 / 7	0.06	-1.2	0.05	-1.1	0.21	0.2	5 / 1	0.06	0.4				
	19	SRM/AO	B	12 / 7					0.1844	-0.3	5 / 1	0.0480	-0.5				
	20		A	15 / 10	0.098	0.5	0.081	0.8	0.212	0.2	7 / 2	0.053	-0.1	0.070	-1.6	0.80	0.036
	21	AO	B	1 / 1							1 / 0						
	22		B	3 / 2					0.22	0.4	2 / 0						
	23		A	15 / 10	0.060	-1.2	0.076	0.5	0.189	-0.3	3 / 1	0.078	1.7				
	24		B	1 / 1							1 / 0						
	25	SRM/AO	B	9 / 5					0.184	-0.4	4 / 1	0.052	-0.2				
	26		A	15 / 10	0.101	0.7	0.068	0.0	0.193	-0.2	7 / 1	0.049	-0.4	FN	-FN	*a. n. f.*	
	27	SRM/AO	A	14 / 9	0.0770	-0.5			0.285	1.6	2 / 0						
	28	SRM/AO	B	9 / 5					0.123	-1.6	2 / 1	0.0566	0.1				
	29		B	3 / 2							1 / 0						
	30		B	6 / 4	0.344	11.9					0 / 0						
	31		B	5 / 4							1 / 1	0.060	0.4				
	32		A	15 / 10	0.091	0.2	0.066	-0.1	0.200	0.0	7 / 2	0.045	-0.7	0.133	0.5	*a. n. f.*	
	33	SRM/AO	A	15 / 10	0.102	0.7	0.086	1.1	0.204	0.0	5 / 1	0.055	0.0			*a. n. f.*	
	35		A	15 / 10	0.0731	-0.6	0.0515	-1.0	0.136	-1.3	5 / 1	0.0580	0.2			0.0770	
	36		B	8 / 6							1 / 0						
	37		A	15 / 10	0.061	-1.2	0.080	0.7	0.188	-0.3	7 / 2	0.060	0.4	0.118	0.0	*a. n. f.*	
	38		A	15 / 10	0.118	1.4	0.066	-0.1	0.171	-0.6	6 / 2	0.060	0.4	0.114	-0.1	*a. n. f.*	0.057
	39		B	3 / 1							2 / 1	0.055	0.0				
	40		B	8 / 6							6 / 1	0.0578	0.2				
	41		B	12 / 8	0.0567	-1.4	0.0640	-0.2	0.179	-0.5	5 / 2	0.0513	-0.3	0.140	0.7	*a. n. f.*	
	42		A	15 / 10	0.074	-0.6	0.058	-0.6	0.19	-0.2	6 / 2	0.052	-0.2	0.109	-0.3	*a. n. f.*	
	43		B	3 / 2							1 / 0						
	44		A	15 / 10	0.089	0.1	0.070	0.1	0.253	1.0	5 / 1	0.053	-0.1			*a. n. f.*	
	45	SRM/AO	B	15 / 7	0.078	-0.4	0.033	-2.1	FN	-FN	6 / 0	FN	-FN	FN	-FN	*a. n. f.*	0.230
	46	SRM/AO	B	8 / 4							2 / 0						
	47		B	8 / 4	0.107	0.9					4 / 2	0.0520	-0.2	0.128	0.3		

Table 4-8 (cont.): Results reported and z scores achieved by all participating laboratories for the compounds present in the test material

				Compulsory													
Compounds				2,4-D (free acid)		BAC-C14 chloride		Chlorate (anion)		DDAC-C10 chloride		Fluazifop (free acid)		Glyphosate		Matrine	
Assigned Value [mg/kg]				0.052		0.119		0.102		0.149		0.060		0.102		0.087	
*CV**				18.9%		19.0%		9.8%		25.0%		16.5%		12.8%		16.9%	
MRRL [mg/kg]				0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Lab code SRM18-	NRL	Cat	Analysed / corr. found, max. 15 / 10	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)
48		A	15 / 10	0.068	1.2	0.152	1.1	0.105	0.1	0.175	0.7	0.079	1.3	0.109	0.3	0.082	-0.2
50	SRM	B	7 / 3	0.0530	0.1							0.0620	0.1	0.0890	-0.5		
51	SRM/AO	B	13 / 8	0.0594	0.5	0.121	0.1	0.111	0.4	0.114	-0.9	0.0724	0.8	0.0920	-0.4		
52		B	8 / 4	0.0617	0.7			0.102	0.0			0.0645	0.3	0.108	0.2	FN	-FN
53		B	7 / 3	0.0575	0.4							0.056	-0.3	0.107	0.2		
54		A	15 / 10	0.036	-1.2	0.115	-0.1	0.106	0.2	0.157	0.2	0.048	-0.8	0.096	-0.2	0.102	0.7
55	SRM/AO	A	15 / 10	0.055	0.2	0.142	0.8	0.104	0.1	0.170	0.6	0.064	0.3	0.091	-0.4	0.092	0.2
57	SRM	A	15 / 10	0.042	-0.8	0.056	-2.1	0.103	0.0	0.071	-2.1	0.059	-0.1	0.093	-0.4	0.065	-1.0
58		B	11 / 5			FN	-FN	0.285	7.2	0.199	1.3	FN	-FN	0.253	5.9		
59		B	8 / 4	0.0712	1.4	0.149	1.0			0.206	1.5	0.0720	0.8				
60	SRM/AO	B	2 / 0														
61		B	7 / 4	0.027	-1.9					0.121	-0.8	0.051	-0.6	0.085	-0.7		
62		A	15 / 10	0.0497	-0.2	0.122	0.1	0.103	0.0	0.166	0.5	0.0598	0.0	0.0926	-0.4	0.0855	-0.1
63	SRM	B	11 / 7	0.0394	-1.0			0.0919	-0.4			0.0492	-0.7	0.0650	-1.5	0.0747	-0.6
64		A	15 / 10	0.049	-0.3	0.116	-0.1	0.107	0.2	0.158	0.2	0.062	0.1	0.101	0.0	0.071	-0.7
65	SRM	B	1 / 1											0.0973	-0.2		
66	SRM	B	12 / 8	0.051	-0.1			0.107	0.2			0.061	0.1	0.108	0.2	0.103	0.7
67		A	14 / 9	0.0510	-0.1	0.124	0.2	0.103	0.0	0.188	1.0	0.0603	0.0	0.114	0.5	0.0847	-0.1
69		B	15 / 8	0.051	-0.1	0.157	1.3	FN	-FN	0.150	0.0	0.063	0.2	0.075	-1.1	0.103	0.7
71		B	10 / 6	0.052	0.0							0.112	3.5	0.089	-0.5	0.086	-0.1
73		A	14 / 9	0.0532	0.1	0.113	-0.2	0.0951	-0.3	0.148	0.0	0.0536	-0.4	0.0923	-0.4	0.0737	-0.6
74		A	15 / 10	0.05	-0.2	0.106	-0.4	0.103	0.0	0.105	-1.2	0.06	0.0	0.099	-0.1	0.1	0.6
75		B	4 / 3					0.085	-0.7					0.099	-0.1		
77		B	3 / 0			FN	-FN			FN	-FN						
78		A	15 / 10	0.044	-0.6	0.107	-0.4	0.084	-0.7	0.166	0.5	0.049	-0.7	0.1	-0.1	0.093	0.3
79		A	15 / 10	0.0614	0.7	0.117	-0.1	0.0970	-0.2	0.157	0.2	0.0723	0.8	0.105	0.1	0.0597	-1.3
80	SRM/AO	A	15 / 10	0.0507	-0.1	0.132	0.4	0.102	0.0	0.150	0.0	0.0575	-0.2	0.0975	-0.2	0.0840	-0.2
82	SRM	A	15 / 9	0.055	0.2	0.127	0.3	0.114	0.5	0.156	0.2	0.060	0.0	0.117	0.6	0.093	0.3
3rd-34		A	14 / 9	0.0470	-0.4	0.146	0.9	0.0888	-0.5	0.165	0.4	0.0580	-0.1	0.0849	-0.7	0.0710	-0.7
3rd-70		B	11 / 4	0.053	0.1							0.061	0.1	FN	-FN	FN	-FN
3rd-72		A	15 / 10	0.051	-0.1	0.138	0.6	0.094	-0.3	0.137	-0.3	0.069	0.6	0.094	-0.3	0.085	-0.1
3rd-76		B	11 / 7	0.0378	-1.1			0.107	0.2					0.0967	-0.2	0.153	3.0
3rd-83		B	1 / 1											0.162	2.4		

3rd-: Laboratories from third countries or EU candidate countries

				Compulsory							Optional				Extra	
	Compounds			Nicotine		Oxymatrine		Phosphonic acid		Compounds	Perchlorate		Trinexapac (free acid)		Bromide (anion)	Copper
	Assigned Value [mg/kg]			0.087		0.068		0.202		AV [mg/kg]	0.055		0.118		not assessed	
	*CV**			24.8%		15.4%		20.9%		CV*	11.6%		14.7%		not assessed	
	MRRL [mg/kg]			0.01		0.01		0.03			0.01		0.01		none	none
	Lab code SRM18-	NRL	Cat	Analysed / corr. found, max. 15 / 2	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Analysed / corr. found, max. 7 / 2	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	z score (FFP-RSD = 25 %)	Conc. [mg/kg]	
	48		A	15 / 10	0.061	-1.2	0.07	0.1	0.199	-0.1	6 / 2	0.057	0.1	0.127	0.3	*a. n. f.*	
	50	SRM	B	7 / 3							3 / 0					*a. n. f.*	0.0220
	51	SRM/AO	B	13 / 8	0.0751	-0.5			0.147	-1.1	3 / 1	0.0571	0.2			*a. n. f.*	
	52		B	8 / 4							7 / 2	0.0510	-0.3	0.110	-0.3	*a. n. f.*	
	53		B	7 / 3							6 / 1			0.128	0.3		
	54		A	15 / 10	0.086	0.0	0.059	-0.5	0.207	0.1	7 / 2	0.060	0.4	0.096	-0.7	*a. n. f.*	0.044
	55	SRM/AO	A	15 / 10	0.101	0.7	0.066	-0.1	0.191	-0.2	7 / 2	0.056	0.1	0.186	2.3	*a. n. f.*	0.079
	57	SRM	A	15 / 10	0.056	-1.4	0.062	-0.4	0.195	-0.1	7 / 1	0.040	-1.1	FN	-FN	*a. n. f.*	
	58		B	11 / 5	0.18	4.3			0.471	5.3	2 / 1	0.03	-1.8				
	59		B	8 / 4							2 / 0						
	60	SRM/AO	B	2 / 0							0 / 0						
	61		B	7 / 4							4 / 0			FN	-FN		
	62		A	15 / 10	0.0925	0.3	0.0683	0.0	0.172	-0.6	7 / 2	0.0555	0.0	0.127	0.3	*a. n. f.*	
	63	SRM	B	11 / 7			0.0544	-0.8	0.114	-1.7	5 / 1	0.0505	-0.3			*a. n. f.*	
	64		A	15 / 10	0.094	0.3	0.066	-0.1	0.243	0.8	7 / 1	FN	-FN	0.109	-0.3		
	65	SRM	B	1 / 1							2 / 0						
	66	SRM	B	12 / 8	0.098	0.5	0.072	0.2	0.480	5.5	3 / 1	0.112	4.1			*a. n. f.*	
	67		A	14 / 9	0.0937	0.3			0.260	1.1	6 / 2	0.0543	-0.1	0.0977	-0.7	*a. n. f.*	
	69		B	15 / 8	FN	-FN	0.066	-0.1	0.221	0.4	7 / 0	FN	-FN	FN	-FN	0.110	0.046
	71		B	10 / 6	0.071	-0.7	0.054	-0.8			4 / 0			FN	-FN		
	73		A	14 / 9	0.0453	-1.9	0.0667	-0.1			6 / 2	0.0571	0.2	0.0870	-1.1	*a. n. f.*	
	74		A	15 / 10	0.097	0.5	0.057	-0.7	0.203	0.0	7 / 2	0.07	1.1	0.11	-0.3	*a. n. f.*	*a. n. f.*
	75		B	4 / 3					0.118	-1.7	2 / 1	0.046	-0.7				
	77		B	3 / 0							0 / 0						0.0443
	78		A	15 / 10	0.109	1.0	0.07	0.1	0.206	0.1	4 / 1	0.048	-0.5			*a. n. f.*	
	79		A	15 / 10	0.0991	0.6	0.0780	0.6	0.183	-0.4	6 / 1	0.0508	-0.3	FN	-FN	0.0438	
	80	SRM/AO	A	15 / 10	0.102	0.7	0.0694	0.1	0.255	1.0	6 / 1	0.0555	0.0				
	82	SRM	A	15 / 9	FN*	-FN	0.077	0.5	0.190	-0.2	5 / 1	0.099	3.2				
	3rd-34		A	14 / 9			0.0510	-1.0	0.170	-0.6	5 / 1	0.0535	-0.1				
	3rd-70		B	11 / 4	0.132	2.1	FN	-FN	0.189	-0.3	4 / 0					*a. n. f.*	0.058
	3rd-72		A	15 / 10	0.079	-0.4	0.067	-0.1	0.159	-0.9	6 / 2	0.049	-0.4	0.150	1.1	*a. n. f.*	*a. n. f.*
	3rd-76		B	11 / 7	0.0947	0.4	0.113	2.6	0.180	-0.4	4 / 1	0.0643	0.7				
	3rd-83		B	1 / 1							0 / 0						

4.3.5 Laboratory Feedback on Poor Results

In the EUPT-SRM18, no false positive results were reported by the participating laboratories. Like in the previous EUPT-SRMs, with the publication of the preliminary report, all participating laboratories having obtained questionable ($2 < |z\ score < 3$) or unacceptable ($|z\ score \geq 3$) results were asked to investigate the reasons for their poor performance and to report them to the organisers. The aim of this follow-up is to sensitise laboratories to investigate the causes of errors in a timely manner in order to avoid making the same errors in the future.

Concerning results from EU/EFTA-OfLs on compulsory and optional compounds only, there were 25 labs which reported in 49 cases results indicating poor performance, among them 28× FNs, which more than one third of these FNs being linked to ***trinexapac (free acid)***. Concerning participating laboratories from third countries, 3 labs reported in 7 cases results indicating poor performance, among them 3× FNs. **Table 4-9** gives a summary of the number of poor results received by the labs for each of the analytes present in the test material.

In case of the two extra compounds ***bromide ion*** and ***copper*** that were present at their very low natural levels in the test material, 28 and 2 EU/EFTA-OfLs reported "analysed, but not found". Using the lab's RLs for evaluation (as no MRRLs were set) and an orientation level of 0.04 mg/kg for ***bromide ion*** and 0.05 mg/kg for ***copper***, the number of quasi "FNs" (for labs that have analyzed for the these compounds without reporting a result despite their RL being lower than the orientation level) was 0 in the case of ***bromide*** (lowest RL was 0.05 mg/kg) and 2 in the case of ***copper*** (RL = 0.01 mg/kg each). However, this evaluation is for information only.

Following a request by the organisers, 25 laboratories (incl. third country labs) have reported the (possible) reasons for their poor performance. The comments concerned 42 results (86 % of the cases). The most frequent reason reported was "lack of experience with the analyte or matrix or the combination of both" (13×). It should be noted that some of the labs utilized the EUPT-SRM as an opportunity to verify newly introduced methods under development which were not fully validated. The organisers always encourage labs to test new methods and hope that the PT exercise, as well as the error tracing activities of these laboratories, will help them to improve their new methods. Another frequent reason was "Misinterpretation / Misevaluation of measurement data / Misunderstanding of the target pesticide" (10×), e.g. the first run of column equilibration was used by mistake as a reference, or instead of ***trinexapac (free acid)*** trinexapac-

Table 4-9: Number of poor results obtained by the laboratories for each of analytes present in the SRM18 test material. In this PT there were no false positive results.

		# labs with poor results / therein FNs or FN*	
	Compounds	**EU/EFTA-OfLs**	**Labs from third Countries**
Compulsory	2,4-D (free acid)	0 / 0	0 / 0
	BAC-C14 chloride	7 / 3	0 / 0
	Chlorate (anion)	4 / 3	0 / 0
	DDAC-C10 chloride	5 / 2	0 / 0
	Fluazifop (free acid)	3 / 1	0 / 0
	Glyphosate	4 / 1	2 / 1
	Matrine	1 / 1	2 / 1
	Nicotine	4 / 2	1 / 0
	Oxymatrine	1 / 0	2 / 1
	Phosphonic acid	3 / 1	0 / 0
	Subtotal	**32 /14**	**7 / 3**
Optional	Perchlorate	6 / 4	0 / 0
	Trinexapac (free acid)	11 / 10	0 / 0
	Subtotal	**17 / 14**	**0 / 0**

ethyl was sought for. The later explanation was given as a reason by 5 of the 10 laboratories reporrting FNs for ***trinexapac (free acid)***. It is likely that some other laboratories had a FN for the same reason, but were not aware of this at the time the feedback was reported.

A compilation of the feedback received by the laboratories is given in **Appendix 7**. The aim of this compilation is to make all participating laboratories aware of common and potential sources of errors so that they can be avoided or eliminated in the future. This information also provides input to the NRLs on how to better assist OfLs within the network to improve their performance.

4.4 Special Topics

Use of ILIS

Using isotope labelled internal standards (ILISs) typically helps to improve accuracy, especially when it comes to correcting for low absolute recoveries and/or for strong matrix effects that were not or not fully covered by the calibration approach used. As in previous EUPTs, this aspect was also checked by comparing the statistical evaluations of data submitted by labs using ILIS with data of labs not using ILIS (**Table 4-10, p. 36**). Overall, ILIS was most frequently employed in the case of ***glyphosate*** (71 % of labs submitting quantitative results), followed by ***chlorate*** and ***perchlorate*** (60 % each), ***phosphonic acid*** (50 %), ***nicotine*** (30 %) and ***matrine*** (21 %). In the vast majority of these cases, the ILIS was employed at the beginning of the procedure. In all cases an improvement in data dispersion could be noticed, however, with only marginal shift of the robust mean values.

Even though the advantage of using ILISs in honey was overall less pronounced compared to previous PT-matrices (probably because matrix effects are less pronounced in honey), the organisers would like to urge laboratories once again to use ILISs where this is beneficial (e.g., when poor recovery rates and/or strong matrix effects are not adequately addressed otherwise).

Compared to some previous EUPT-SRMs, the EUPT-SRM18 posed few problems for the participants. Below are some issues that the organisers would like to draw attention to, so that laboratories can take them into account in routine controls or future PTs:

Trinexapac

Many of the false negative results on ***Trinexapac (free acid)*** were related to a mix-up between trinexapac and trinexapac-ethyl. This error is likely to be attributed to the following issues:

a) the rather confusing entry in the EU database, which at the time of the PT was "Trinexapac (aka cimetacarb ethyl)";
b) the fact that "trinexapac-ethyl" (the ester form) is defined as the active substance within the EU pesticides database (in contrast to other acidic herbicides and growth regulators, where the free acids are listed as active substances);;
c) the fact that in various online databases, and some websites of vendors of analytical standards, one of the two CAS numbers of trinexapac (104273-73-6) is falsely attributed to trinexapac-ethyl.

All these aspects may have led some laboratories to assume that the name "trinexapac" actually refers to the ethyl ester.

As a reaction to the trinexapac / trinexapac-ethyl mismatching problems observed in the present EUPT, the organisers have contacted DG-SANTE to raise awareness of the problem. As a result, the database entry was changed to 'Trinexapac (Aka cimetacarb)' in October 2023, to avoid confusions.

Bromide Ion

Bromide was present in the honey used to prepare the test item at its natural level without any additional bromide being added. Only six of the 34 laboratories having analyzed for bromide reported results. Due to the small number of numerical results received (six results), it was not possible to statistically establish the assigned value. The median of these results was 0.094 mg/kg. Excluding two deviating results (SRM18-6: 0.943 mg/kg and SRM18-20: 0.8 mg/kg), the median drops to 0.071 mg/kg, which deviates from the level de-

Table 4-10: Comparison of the statistical evaluation of the results obtained with the use of ILIS with that of the results obtained without the use of ILIS.

	Chlorate		
	Whole Population[1)]	ILIS-Yes	ILIS-No
No. of Numerical Results	47	28	19
Mean [mg/kg]	0.106	0.102	0.111
Median [mg/kg]	0.103	0.103	0.102
Robust Mean [mg/kg]	**0.102**	**0.102**	**0.102**
*CV**	**10.1 %**	**8.3 %**	**14.5 %**
RSD	**27.4 %**	**8.7 %**	**39.7 %**
	Glyphosate		
	Whole Population[1)]	ILIS-Yes	ILIS-No
No. of Numerical Results	66	46	20
Mean [mg/kg]	0.107	0.105	0.110
Median [mg/kg]	0.100	0.100	0.100
Robust Mean [mg/kg]	**0.102**	**0.102**	**0.100**
*CV**	**13.4 %**	**12.8 %**	**16.0 %**
RSD	**28.0 %**	**21.5 %**	**38.7 %**
	Nicotine		
	Whole Population[1)]	ILIS-Yes	ILIS-No
No. of Numerical Results	42	12	30
Mean [mg/kg]	0.0950	0.0845	0.0993
Median [mg/kg]	**0.0939**	**0.0918**	**0.0951**
Robust Mean [mg/kg]	**0.0884**	**0.0853**	**0.0901**
*CV**	**25.7 %**	**23.2 %**	**27.5 %**
RSD	**48.6 %**	**22.8 %**	**53.4 %**
	Perchlorate		
	Whole Population[1)]	ILIS-Yes	ILIS-No
No. of Numerical Results	44	28	16
Mean [mg/kg]	0.0573	0.0583	0.0598
Median [mg/kg]	**0.0547**	**0.0553**	**0.0525**
Robust Mean [mg/kg]	**0.0547**	**0.0552**	**0.0535**
*CV**	**11.4 %**	**8.5 %**	**18.8 %**
RSD	**23.2 %**	**17.8 %**	**29.6 %**
	Phosphonic acid		
	Whole Population[1)]	ILIS-Yes	ILIS-No
No. of Numerical Results	47	23	24
Mean [mg/kg]	0.208	0.195	0.221
Median [mg/kg]	**0.197**	**0.199**	**0.196**
Robust Mean [mg/kg]	**0.200**	**0.197**	**0.206**
*CV**	**20.8 %**	**14.9 %**	**29.7 %**
RSD	**33.7 %**	**18.4 %**	**41.2 %**

1) including results reported by laboratories from third countries / EU candidate countries

termined by the organisers (0.049 mg/kg). Based on this data, it can be assumed that the level of bromide in the sample was close to or even higher than the existing MRL for bromide in honey of 0.05 mg/kg. In any case, the current MRL is not only difficult to control (as can be judged from the poor results) but also too low, as it does not properly consider the natural background levels in honey samples.

In a recent EURL-SRM study with 157 honey samples from all continents being analyzed, the current bromide MRL of 0.05 mg/kg was exceeded by more than 90 % of the samples, with the median being at 0.39 mg/kg and the 95th percentile at 1.45 mg/kg. The highest level encountered was as high as 2.8 mg/kg.

Among the 34 EU/EFTA OfLs having analyzed for bromide in this exercise, 22 laboratories (65 %) reported a RL of 0.5 mg/kg and 32 laboratories (94 %) a RL ≥ 0.2 mg/kg. The highest RL reported was at 5 mg/kg (SRM18-26). Two laboratories (6 %) indicated a RL of 0.05 mg/kg and thus low enough for controlling the MRL of 0.05 mg/kg as well as for analysing the estimated concentration of bromide in the test item which was in the range of 0.05 - 0.1 mg/kg. One of these two labs even reported a false negative result for bromide. There were no labs with RLs < 0.05 mg/kg. Interestingly, three (SRM18-79, -35, -69) of the six labs that had reported numerical results for bromide, reported RLs exceeding the actual result reported. These results could be considered as false reportings ("FR"). In any case, the need to adjust an MRL for bromide in honey is obvious, not only because the natural levels of bromide in honey are mostly higher than 0.05 mg/kg, but also because laboratories are obviously not able to analyse bromide properly at such low levels. If the MRL was raised to e. g. 2 mg/kg, a level that could encompass the vast majority of the honeys on the market, only 3 of the 34 labs submitting data (9 %) wouldn't have been able to covercover this level based on the RLs reported in this PT. The need to revise the MRL for bromide in honey was communicated to DG-SANTE by the organisers.

Copper

Copper was also present in the test item at its natural level. Only 12 laboratories indicated analysing for copper with ten of them reporting quantitative results (median level 0.048 mg/kg). Excluding two deviating results (SRM18-45: 0.23 mg/kg and SRM18-10 0.49 mg/kg), the median drops to 0.041 mg/kg, which is close to the level determined by the organisers (0.038 mg/kg). All ten laboratories reporting results indicated RLs at 0.01 mg/kg. The two laboratories not reporting quantitative results (SRM18-17 and SRM18-74), reported RLs of 0.5 and 1 mg/kg, respectively. The results of these two laboratories are thus technically not false negatives. It should be highlighted that during the experiments by the organisers it was noticed that the background levels fluctuate and that LOQs < 0.05 mg/kg can only be maintained when applying certain measures, which may include the thorough cleaning of the reaction/digestion vessels, the use of certain vessels exclusively for low level analyses, and the use of very clean reagents.

Overview of Noteworthy or Critical Issues recorded for each Laboratory

An overview of noteworthy or critical aspects recorded for each laboratory during the EUPT-SRM18 (e.g. on performance or reporting), is given in **Table 4-11 (p. 38)**. Entries to the table are made in the following cases:

1) Poor sensitivity of the method if the laboratory RL is higher than the MRRL;
2) Poor results, including questionable and unacceptable z scores, FNs and FPs (not applicable in this EUPT);
3) False Reportings (if the reported concentration is lower than the RL);
4) Non-submission of results;
5) Failure to submit method information for FNs in the Webtool during the additional information period.
6) Late sample arrival

Table 4-11: Overview of notable or critical issues in laboratory performance or reporting

		Compulsory Compounds																
Compounds listed on the Target List		2,4-D (free acid)	BAC-C12 chloride	BAC-C14 chloride		Chlorate (anion)		Chlormequat chloride	DDAC-C10 chloride		Fluazifop (free acid)		Fosetyl	Glyphosate		Haloxyfop (free acid)	Matrine	
Present in Test Item		Yes	No	Yes		Yes		No	Yes		Yes		No	Yes		No	Yes	
MRRL [mg/kg]		0.01	0.01	0.01		0.01		0.01	0.01		0.01		0.01	0.01		0.01	0.01	
Lab-Code SRM18-	non-Submission / No Meth Data for FNs / No Feedback for Poor Results / Delayed Sample Arrival	Poor Sensitivity (RL > MRRL)	Poor Sensitivity (RL > MRRL)	Poor Sensitivity (RL > MRRL)	Poor Result and/or FR	Poor Sensitivity (RL > MRRL)	Poor Result and/or FR	Poor Sensitivity (RL > MRRL)	Poor Sensitivity (RL > MRRL)	Poor Result and/or FR	Poor Sensitivity (RL > MRRL)	Poor Result and/or FR	Poor Sensitivity (RL > MRRL)	Poor Sensitivity (RL > MRRL)	Poor Result and/or FR	Poor Sensitivity (RL > MRRL)	Poor Sensitivity (RL > MRRL)	Poor Result and/or FR
2																		
3					FN					FN								
4																		
5																		
6																		
7																		
8																		
9																		
10															AZ ≥ 3			
11																		
12																		
13																		
14																		
15																		
16																		
17																		
18							FN											
19					AZ > 2 and FR					AZ > 2								
20																		
21																		
22																		
23																		
24																		
25																		
26					AZ > 2													
27																		
28																		
29											AZ ≥ 3							
30										AZ ≥ 3								
31															FN			
32																		
33																		
35																		
36																		
37																		
38																		
39																		
40																		
41																		
42																		

FN*: FN because of labs' RL > assigned value

	Optional Compounds										Extra Compound
	AMPA	Chloridazon desphenyl	MCPA (free acid)	N-Acetyl glyphosate	Perchlorate		Quizalofop (free acid)	Trinexapac (free acid)			Bromide (anion)
	No	No	No	No	Yes		No	Yes			Yes
	0.01	0.02	0.01	0.01	0.01		0.01	0.01			none
	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR		Poor Result and/or FR
									FN		
									FN		
					FN						
									FN		
									FN		
		AZ ≥ 3									
											FR

Table 4-11 (cont.): Overview of notable or critical issues in laboratory performance or reporting

		Compulsory Compounds																
Compounds listed on the Target List		2,4-D (free acid)	BAC-C12 chloride	BAC-C14 chloride		Chlorate (anion)		Chlormequat chloride	DDAC-C10 chloride		Fluazifop (free acid)		Fosetyl	Glyphosate		Haloxyfop (free acid)	Matrine	
Present in Test Item		Yes	No	Yes		Yes		No	Yes		Yes		No	Yes		No	Yes	
MRRL [mg/kg]		**0.01**	0.01	**0.01**		**0.01**		0.01	**0.01**		**0.01**		**0.01**	**0.01**		**0.01**	**0.01**	
Lab-Code SRM18-	non-Submission / No Meth Data for FNs / No Feedback for Poor Results / Delayed Sample Arrival	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poo Resu and/or
43																		
44																		
45							FN								FN			
46					AZ≥3													
47																		
48																		
49																		
50																		
51																		
52																		FN
53																		
54																		
55																		
57					AZ>2					AZ>2								
58					FN		AZ≥3					FN			AZ≥3			
59																		
60																		
61																		
62																		
63																		
64																		
65																		
66																		
68																		
69							FN											
71												AZ≥3						
73																		
74																		
75																		
77					FN					FN								
78																		
79																		
80																		
81																		
82																		
3rd-34																		
3rd-70															FN			FN
3rd-72																		
3rd-76																		AZ≥
3rd-83															AZ>2			
3rd-84																		

FN*: FN because of labs' RL > assigned value

	Optional Compounds												Extra Compound
	Mepiquat chloride	Nicotine		Oxymatrine		Phosphonic acid		AMPA	Chloridazon desphenyl	MCPA (free acid)	N-Acetyl glyphosate	Perchlorate		Quizalofop (free acid)	Trinexapac (free acid)		Bromide (anion)
	No	Yes		Yes		Yes		No	No	No	No	Yes		No	Yes		Yes
	0.01	0.01		0.01		0.03		0.01	0.02	0.01	0.01	0.01		0.01	0.01		none
	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Sensitivity (RL>MRRL)	Poor Sensitivity (RL>MRRL)	Poor Result and/or FR	Poor Result and/or FR
					AZ > 2		FN					FN				FN	
																AZ > 2	
																FN	
			AZ ≥ 3				AZ ≥ 3										
																FN	
												FN					
			AZ ≥ 3									AZ ≥ 3					
			FN									FN				FN	FR
																FN	
																FN	FR
			FN*									AZ ≥ 3					
			AZ > 2		FN												
3					AZ > 2												

5. ACKNOWLEDGEMENTS

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6. REFERENCES

[1] Regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March 2017 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 L 95, 7.4.2017, pp. 1 – 142

[2] Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin; published at OJ L 70, 16.3.2005, pp. 1 – 16.

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

[4] https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EurlSrm_meth_QuPPe_PO_V12_1.pdf

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

7. APPENDICES

Appendix 1 List of Laboratories Registered to Participate in the EUPT-SRM18

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

Country (Location)	Analysed on behalf of	Institution	City	NRL Activities
Austria	AT	AGES - Innsbruck	Innsbruck	SRM/AO
Belgium	BE	Sciensano	Brussels	SRM
Belgium	BE; FR; LU; BG	Primoris Belgium	Gent - Zwijnaarde	–
Croatia	HR	Croatian Veterinary Institute - Krizevci	Krizevci	SRM
Croatia	HR	Dr. Andrija Štampar - Pesticide Lab	Zagreb	SRM
Croatia	HR	Eurofins Croatiakontrola	Zagreb	–
Croatia	HR	Sample Control - Pesticide Lab	Lučko	–
Cyprus	CY	State General Laboratory of Cyprus	Nicosia	SRM/AO
Czech Republic	CZ	Czech Agriculture and Food Inspection Authority	Praha	SRM
Czech Republic	CZ	ÚKZÚZ	Brno	–
Czech Republic	CZ	UCT Prague - Metrological and Testing Laboratory	Praha	–
Denmark	DK	Danish Veterinary and Food Administration	Ringsted	SRM
Estonia	EE	LABRIS - Laboratory of Chemistry (Tallinn)	Tallinn	–
Finland	FI	Finnish Food Authority	Helsinki	SRM/AO
France	FR	ANSES - LSAI (Unité PBM)	MAISONS-ALFORT Cedex	SRM/AO
France	BE	Phytocontrol (Nimes) - Pesticide Lab	Nimes	–
Germany	DE	BVL Unit 504 NRL for Pesticide Residues	Berlin	SRM/AO
Germany	DE	CVUA Freiburg	Freiburg	–
Germany	DE	CVUA-RRW	Krefeld	–
Germany	DE	Eurofins Dr.Specht Express GmbH - Hamburg	Hamburg	–
Germany	DE	Landesamt für Landwirt. Lebensmittelsicherheit und Fischerei	Rostock	–
Germany	DE	Landesamt für Verbraucherschutz, Halle/Saale	Halle/Saale	–
Germany	DE	Landeslabor Schleswig-Holstein	Neumünster	–
Germany	DE	LAVES, Lebensmittel- und Veterinärinstitut Oldenburg	Oldenburg	–
Germany	DE	LGL Erlangen	Erlangen	–
Germany	DE	LTZ Augustenberg	Karlsruhe	–
Germany	DE	Landesuntersuchungsamt ILC Speyer	Speyer	–
Germany	DE	LUA Sachsen - Pesticide Lab, Dresden	Dresden	–
Germany	DE	LUFA Speyer	Speyer	–
Germany	FR	Intertek Food Services GmbH, Bremen	Bremen	–
Germany	BE	AGROLAB LUFA Kiel - Pesticide Lab	Kiel	–
Germany	LT	GALAB Laboratories GmbH - Hamburg	Hamburg	–
Germany	MT	Eurofins - Germany, Hamburg	Hamburg	–
Greece	GR	Benaki Phytopathological Institute, Kifissia	Kifissia	SRM/AO
Greece	GR	GENERAL CHEMICAL STATE LABORATORY	Athens	SRM/AO
Hungary	HU	Plant Protection and Soil Conservation NRL site in Velence	Velence	SRM/AO
Hungary	HU	NFCSO FCSLD PPSCNRL, Site in Miskolc	Miskolc	–
Ireland	IE	The Food Chemistry Laboratories - DAFM	Celbridge	SRM/AO
Italy	IT	APPA Bolzano	Bolzano	–
Italy	IT	IZSLT - Rome	Roma	–
Italy	IT	IZSAM - Pesticide Lab	Teramo	–

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

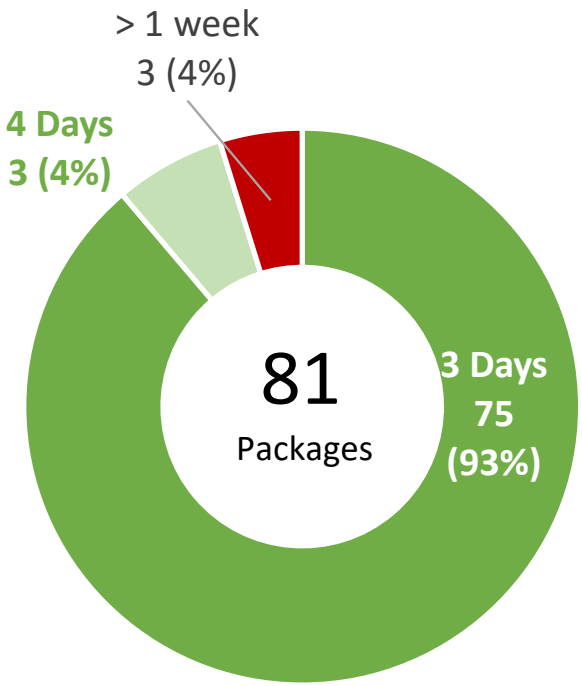
Country (Location)	Analysed on behalf of	Institution	City	NRL Activities
Italy	IT	Istituto Zooprofil Sperimentale Lombardia ed Emilia Romagna	Brescia	–
Italy	IT	IZSVe - Pesticide Lab	Legnaro (Padova)	–
Latvia	LV	BIOR (Riga) - Pesticide Lab	Riga	SRM/AO
Lithuania	LT	NFVRAI	Vilnius	SRM/AO
Luxembourg	LU	LNS Food lab	Dudelange	SRM
Norway	NO	NIBIO Pesticides & Natural Products Chemistry	ÅS	SRM/AO
Poland	PL	InHort (Skierniewice) - Pesticide Lab	Skierniewice	–
Poland	PL	IPP-NRI - Pesticide Lab (Poznan)	Poznan	–
Poland	PL	Laboratory of Food & Feed Safety in Bialystok	Bialystok	–
Poland	PL	Nat. Vet. Research Institute - Poland, Pulawy	Puławy	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
Slovenia	SI	Pesticide Lab - Maribor	Maribor	SRM/AO
Spain	ES	Lab. Arbitral Agroalimentario	Madrid	SRM
Spain	ES	Centro Nacional de Alimentación	Majadahonda	SRM/AO
Spain	ES	Ainia (Valencia)	Valencia	–
Spain	ES	Analytica Alimentaria GmbH - Almeria, Spain	Almeria	–
Spain	ES	EURL-FV - Pesticide Residue Research Group	Almeria	–
Spain	ES	Laboratori de l'Agència de Salut Pública de Barcelona (ASPB)	Barcelona	–
Spain	ES	Laboratorio Agroalimentario de Extremadura	Cáceres	–
Spain	ES	Laboratorio Analítico Bioclínico - Spain, Almeria	Almeria	–
Spain	ES	Laboratorios Apinevada S.L - Spain, Escuzar	Escuzar (Granada)	–
Spain	ES	National Center for Technology and Food Safety (CNTA)	San Adrián (Navarra)	–
Spain	ES	SALUD PUBLICA (LSP - MADRID SALUD)	Madrid	–
Sweden	SE	Eurofins Food and Feed Testing Sweden	Lidköping	–
Switzerland	CH	Kantonales Laboratorium Zürich	Zürich	–
The Netherlands	NL	Wageningen Food Safety Research	Wageningen	SRM/AO
The Netherlands	BE; NL	Eurofins Lab Zeeuws-Vlaanderen	Graauw	–
The Netherlands	BE	Groen Agro Control - Netherlands	Delfgauw	–

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

Country	Institution	City
Serbia	A BIO TECH LAB - Serbia, Sremska Kamenica	Sremska Kamenica
Serbia	Field Test - Serbia, Belgrade	Belgrade
United Kingdom	FERA - Pesticide Lab	York
Uruguay	UdelaR - Faculty of Chemistry (Montevideo)	Montevideo
Vietnam	SGS - Vietnam, Ho Chi Minh	Ho Chi Minh

Appendix 2 Shipment Evaluation

Compilation of information on shipment duration



- **Within 3 days: 93 %**
- **Within 4 days:** 3 EU (2× ES, 1× CY) remote location
- **3 Parcels more than 7 days:** EU candidate and third countries only, one parcel was kept frozen at local DHL and one was kept in a fridge during customs clearance

Appendix 3 Data of Homogeneity Test

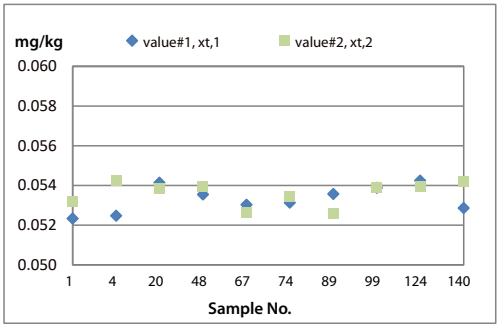
Compulsory Compounds

	2,4-D (free acid)		BAC-C14-Cl		Chlorate (anion)		DDAC-C10-Cl		Fluazifop (free acid)	
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]
001	0.0523	0.0532	0.104	0.106	0.109	0.108	0.143	0.144	0.0627	0.0617
004	0.0525	0.0542	0.105	0.106	0.106	0.108	0.144	0.145	0.0636	0.0635
020	0.0541	0.0538	0.108	0.106	0.107	0.109	0.149	0.149	0.0631	0.0630
048	0.0535	0.0539	0.108	0.106	0.107	0.110	0.142	0.145	0.0618	0.0637
067	0.0530	0.0527	0.103	0.103	0.109	0.107	0.139	0.145	0.0625	0.0620
074	0.0531	0.0534	0.106	0.104	0.106	0.108	0.150	0.145	0.0640	0.0614
089	0.0536	0.0526	0.106	0.106	0.107	0.109	0.145	0.143	0.0635	0.0610
099	0.0539	0.0539	0.105	0.103	0.108	0.109	0.142	0.143	0.0627	0.0606
124	0.0543	0.0540	0.106	0.105	0.110	0.108	0.146	0.145	0.0630	0.0620
140	0.0529	0.0542	0.106	0.108	0.106	0.107	0.150	0.151	0.0624	0.0648
mean / AV*	0.0535 (0.0523)		0.105 (0.119)		0.108 (0.102)		0.145 (0.149)		0.0627 (0.0598)	

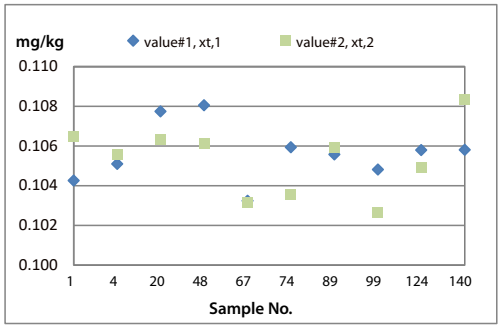
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Graphical presentation of the results:

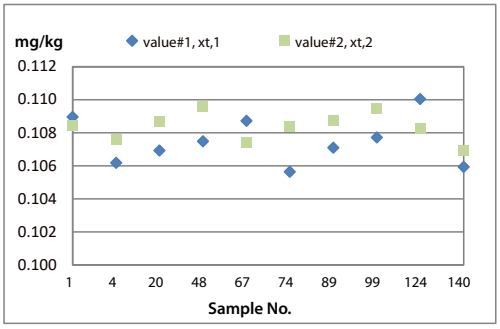
2,4-D (free acid)



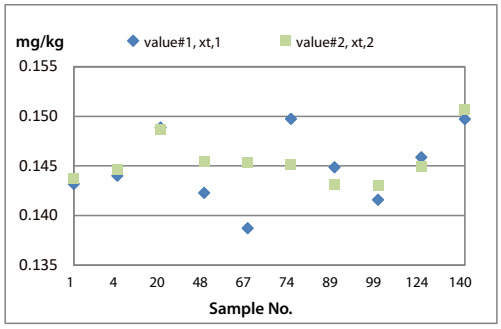
BAC-C14 chloride



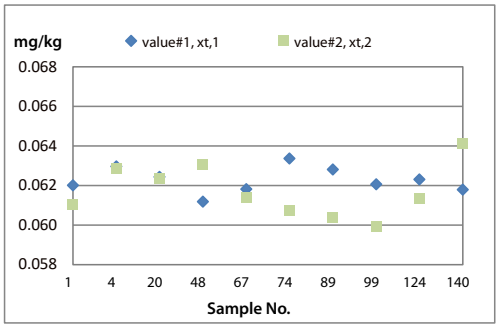
Chlorate (anion)



DDAC-C10 chloride



Fluazifop (free acid)



Appendix 3 (cont.): Data of Homogeneity Test

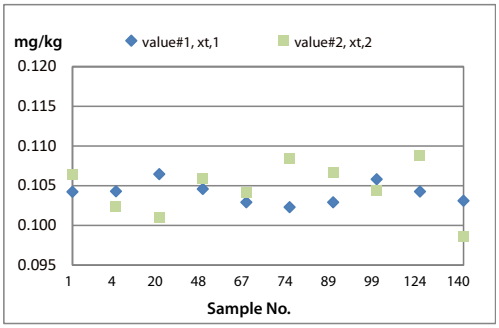
Compulsory Compounds

	Glyphosate		Matrine		Nicotine		Oxymatrine		Phosphonic acid	
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]
011	0.104	0.106	0.0934	0.0947	0.0883	0.0910	0.0733	0.0769	0.211	0.207
034	0.104	0.102	0.0887	0.0942	0.0917	0.0957	0.0725	0.0705	0.209	0.212
050	0.106	0.101	0.0886	0.0909	0.0923	0.0922	0.0727	0.0700	0.196	0.191
073	0.105	0.106	0.0912	0.0894	0.0912	0.0921	0.0719	0.0697	0.200	0.210
096	0.103	0.104	0.0885	0.0919	0.0932	0.0917	0.0767	0.0723	0.207	0.199
112	0.102	0.108	0.0907	0.0903	0.0922	0.0926	0.0702	0.0719	0.194	0.195
143	0.103	0.107	0.0905	0.0920	0.0899	0.0914	0.0712	0.0697	0.211	0.201
156	0.106	0.104	0.0916	0.0936	0.0921	0.0902	0.0777	0.0738	0.203	0.206
183	0.104	0.109	0.0922	0.0908	0.0966	0.0933	0.0671	0.0724	0.208	0.193
200	0.103	0.099	0.0861	0.0914	0.0938	0.0936	0.0744	0.0729	0.207	0.211
mean / AV*	0.104 (0.102)		0.0910 (0.0873)		0.0923 (0.0877)		0.0724 (0.0681)		0.204 (0.202)	

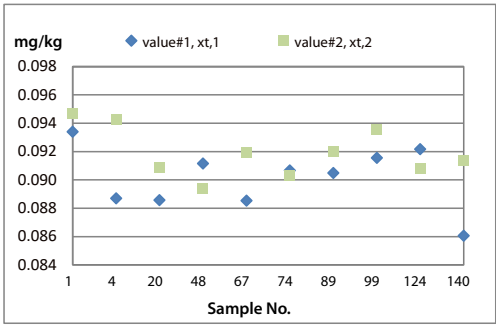
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Graphical presentation of the results:

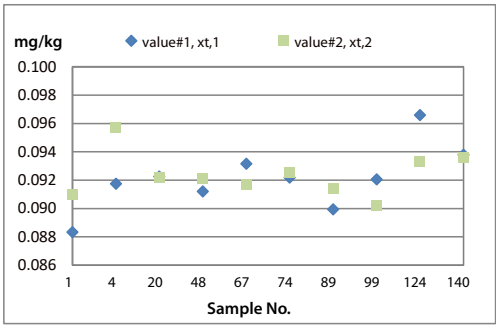
Glyphosate



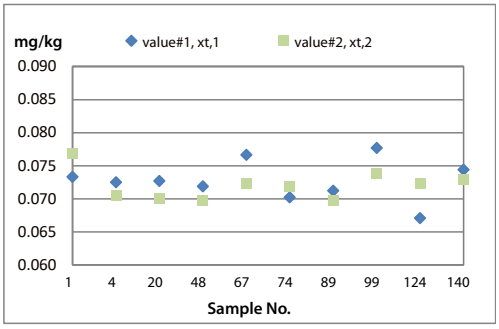
Matrine



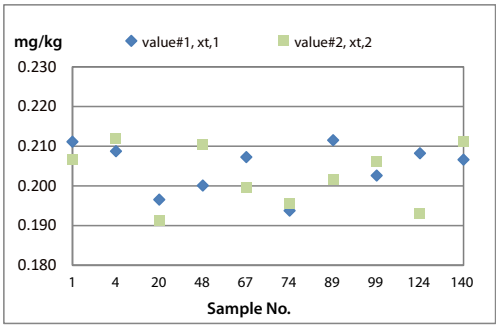
Nicotine



Oxymatrine



Phosphonic acid



Appendix 3 (cont.): Data of Homogeneity Test

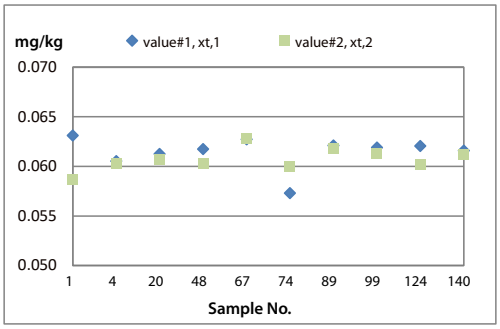
Optional Compounds

	Perchlorate		Trinexapac (free acid)	
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]
001	0.0631	0.0587	0.108	0.101
004	0.0605	0.0603	0.128	0.119
020	0.0613	0.0607	0.115	0.097
048	0.0618	0.0603	0.111	0.103
067	0.0627	0.0628	0.107	0.101
074	0.0573	0.0600	0.095	0.107
089	0.0621	0.0618	0.113	0.106
099	0.0619	0.0613	0.118	0.098
124	0.0621	0.0602	0.105	0.085
140	0.0616	0.0612	0.106	0.099
mean / AV*	0.0611 (0.0550)		0.106 (0.118)	

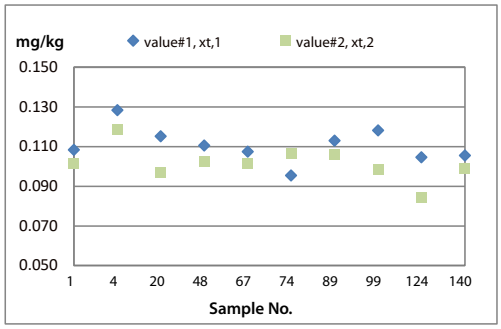
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Graphical presentation of the results:

Perchlorate



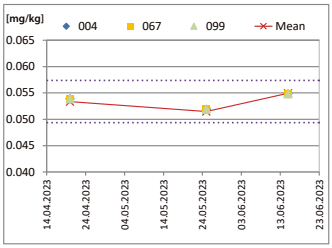
Trinexapac (free acid)



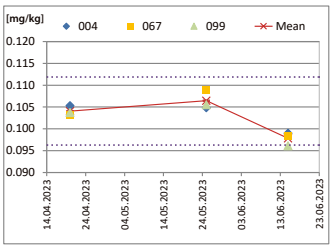
Appendix 4 Data of Stability Test

	Compulsory Compounds																	
	2,4-D (free acid)						BAC-C14-Cl						Chlorate (anion)					
AV [mg/kg]	0.052						0.119						0.102					
Date	20.04.2023		25.05.2023		15.06.2023		20.04.2023		25.05.2023		15.06.2023		27.04.2023		25.05.2023		15.06.2023	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 004	0.0525	0.0542	0.0503	0.0522	0.0552	0.0552	0.105	0.106	0.103	0.106	0.097	0.101	0.106	0.108	0.103	0.103	0.104	0.110
No. 067	0.0530	0.0527	0.0504	0.0521	0.0544	0.0551	0.103	0.103	0.111	0.107	0.098	0.099	0.109	0.107	0.105	0.106	0.105	0.111
No. 099	0.0539	0.0539	0.0506	0.0532	0.0547	0.0550	0.105	0.103	0.107	0.104	0.097	0.095	0.108	0.109	0.102	0.103	0.107	0.110
Mean [mg/kg]	0.0534		0.0515		0.0549		0.104		0.106		0.098		0.108		0.104		0.108	
RSD* [%]	1.0%		0.8%		0.4%		1.1%		2.1%		1.5%		0.8%		1.4%		0.9%	
Diviation [%] (ref. 1st Anaylsis)	—		-3.5%		3.0%		—		2.3%		-6.0%		—		-3.7%		0.0%	

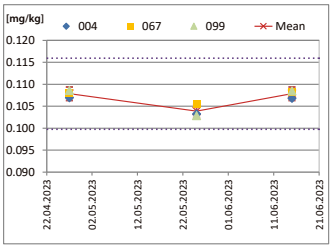
2,4-D (free acid)



BAC-C14 chloride



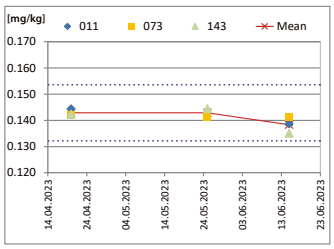
Chlorate (anion)



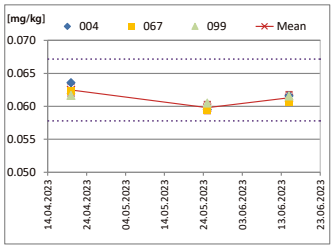
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

	Compulsory Compounds																	
	DDAC-C10-Cl						Fluazifop (free acid)						Glyphosate					
AV [mg/kg]	0.149						0.06						0.102					
Date	20.04.2023		25.05.2023		15.06.2023		20.04.2023		25.05.2023		15.06.2023		27.04.2023		25.05.2023		15.06.2023	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 004	0.144	0.145	0.142	0.144	0.141	0.136	0.0636	0.0635	0.0584	0.0606	0.0624	0.0607	0.104	0.102	0.100	0.100	0.103	0.111
No. 067	0.139	0.145	0.141	0.141	0.143	0.140	0.0625	0.0620	0.0576	0.0612	0.0603	0.0611	0.103	0.104	0.0937	0.0917	0.109	0.111
No. 099	0.142	0.143	0.145	0.143	0.136	0.134	0.0627	0.0606	0.0601	0.0609	0.0623	0.0609	0.106	0.104	0.0980	0.0988	0.107	0.104
Mean [mg/kg]	0.143		0.143		0.138		0.062		0.060		0.061		0.104		0.097		0.107	
RSD* [%]	0.9%		1.1%		2.3%		1.6%		1.0%		0.8%		0.9%		3.9%		2.3%	
Diviation [%] (ref. 1st Anaylsis)	—		0.0%		-3.2%		—		-4.3%		-1.9%		—		-6.8%		3.3%	
	‡ other portions (11, 73, 143) used																	

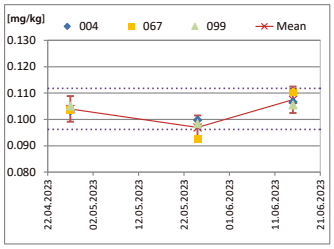
DDAC-C10 chloride



Fluazifop (free acid)



Glyphosate



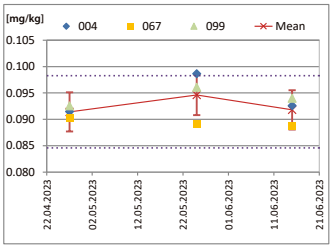
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

* RSD = relative standard diviation

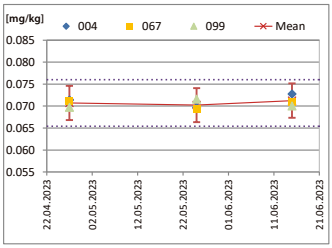
Appendix 4 (cont.): Data of Stability Test

	Compulsory Compounds																	
	Matrine						Oxymatrine						Phosphonic acid					
AV [mg/kg]	0.087						0.068						0.202					
Date	27.04.2023		25.05.2023		15.06.2023		27.04.2023		25.05.2023		15.06.2023		27.04.2023		25.05.2023		15.06.2023	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 004	0.091	0.099	0.093	0.0931	0.0917	0.0957	0.0727	0.0700	0.0686	0.0707	0.0746	0.0709	0.186	0.180	0.175	0.172	0.177	0.181
No. 067	0.090	0.089	0.089	0.0899	0.0921	0.0902	0.0702	0.0719	0.0734	0.0654	0.0710	0.0709	0.184	0.175	0.173	0.173	0.188	0.179
No. 099	0.093	0.096	0.094	0.0871	0.0938	0.0936	0.0671	0.0724	0.0719	0.0713	0.0702	0.0699	0.183	0.177	0.175	0.173	0.176	0.177
Mean [mg/kg]	0.091		0.095		0.092		0.071		0.070		0.071		0.181		0.174		0.180	
RSD* [%]	1.3%		5.2%		2.9%		1.2%		1.7%		1.9%		1.1%		0.3%		1.9%	
Diviation [%] (ref. 1st Anaylsis)	—		3.5%		0.4%		—		-0.7%		0.8%		—		-3.9%		-0.5%	

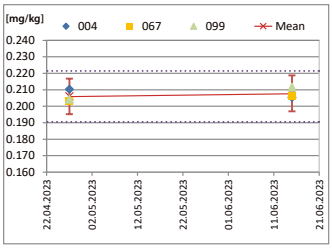
Matrine



Oxymatrine



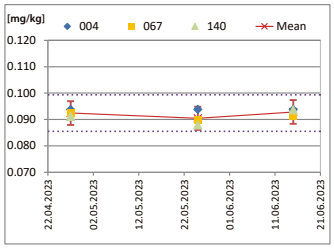
Phosphonic acid



– – –: 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 %

	Compulsory Compounds					
	Nicotine					
AV [mg/kg]	0.087					
Date	27.04.2023		25.05.2023		15.06.2023	
Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 004	0.0917	0.0957	0.0945	0.0931	0.0917	0.0957
No. 067	0.0932	0.0917	0.0894	0.0899	0.0921	0.0902
No. 104	0.0921	0.0902	0.0888	0.0871	0.0938	0.0936
Mean [mg/kg]	0.092		0.090		0.093	
RSD* [%]	1.4%		3.3%		1.6%	
Diviation [%] (ref. 1st Anaylsis)	—		-2.1%		0.5%	

Nicotine

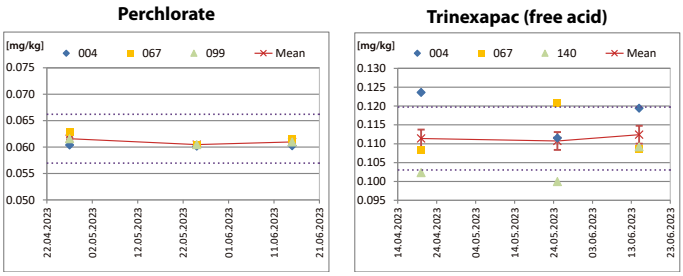


– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

* RSD = relative standard diviation

Appendix 4 (cont.): Data of Stability Test

	Optional Compounds											
	Perchlorate						Trinexapac (free acid)					
AV [mg/kg]	0.052						0.118					
Date	27.04.2023		25.05.2023		15.06.2023		20.04.2023		25.05.2023		15.06.2023	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 004	0.0605	0.0603	0.0604	0.0600	0.0616	0.0590	0.128	0.119	0.110	0.113	0.116	0.123
No. 067	0.0627	0.0628	0.0602	0.0605	0.0614	0.0616	0.118	0.098	0.128	0.114	0.112	0.105
No. 099 / 140	0.0619	0.0613	0.0622	0.0595	0.0609	0.0617	0.106	0.099	0.118	0.082	0.125	0.093
Mean [mg/kg]	0.0616		0.0605		0.0610		0.111		0.111		0.112	
RSD* [%]	1.9%		0.6%		1.0%		9.9%		9.4%		5.4%	
Diviation [%] (ref. 1st Anaylsis)	—		-1.8%		-1.0%		—		-0.6%		0.9%	



– – –: 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 %

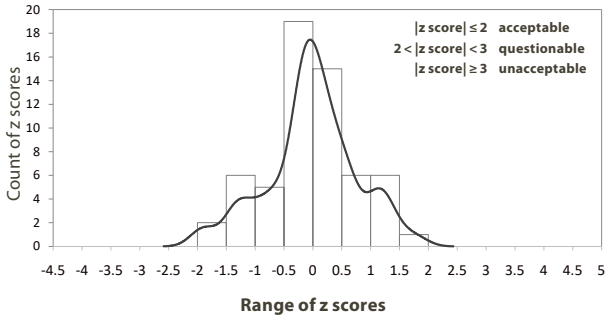
A4
STABILITY

* RSD = relative standard diviation

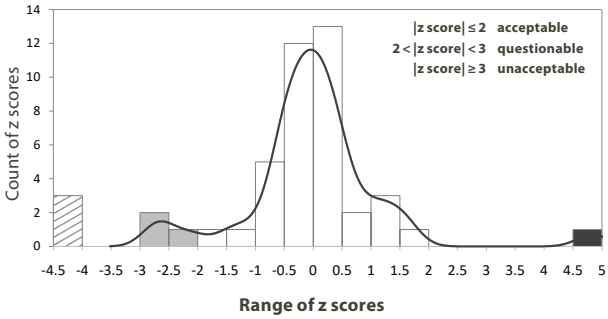
Appendix 5 Histograms and Kernel Density Estimates of z score* Distributions
(Results from EU and EFTA Laboratories only)

Compulsory Compounds

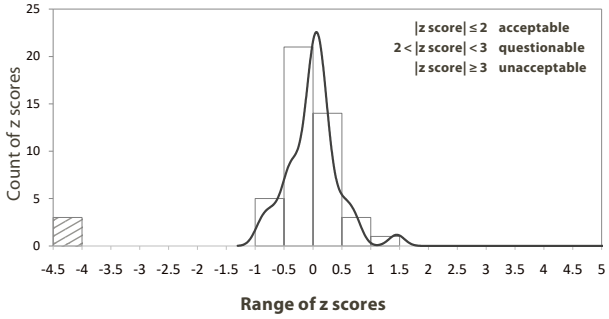
2,4-D (free acid)
(AV derived from entire population)



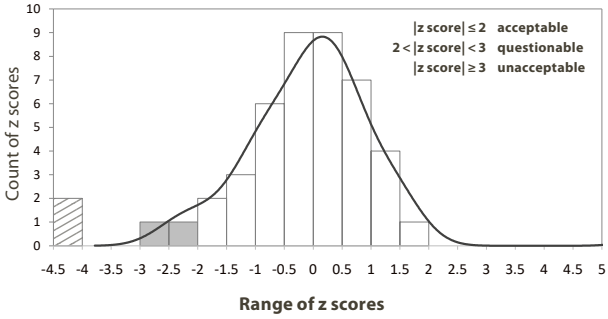
BAC-C14 chloride
(AV derived from entire population)



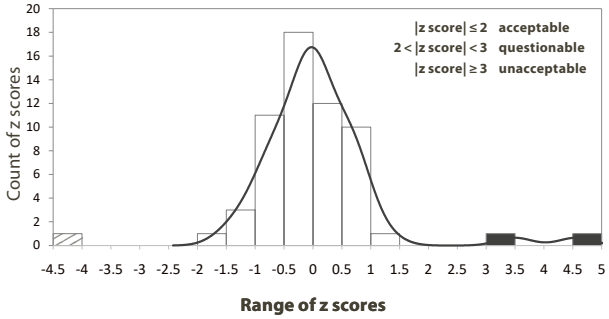
Chlorate
(AV derived from entire population)



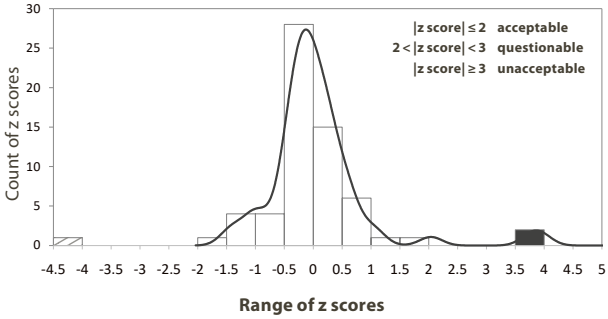
DDAC-C10 chloride
(AV derived from entire population)



Fluazifop (free acid)
(AV derived from entire population)



Glyphosate
(AV derived from entire population)



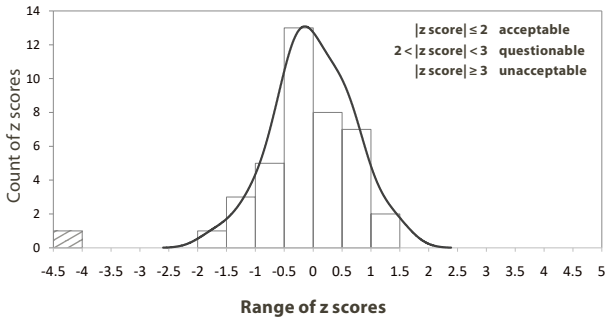
* Cut-off at z score = 5; ▨: false negative results

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

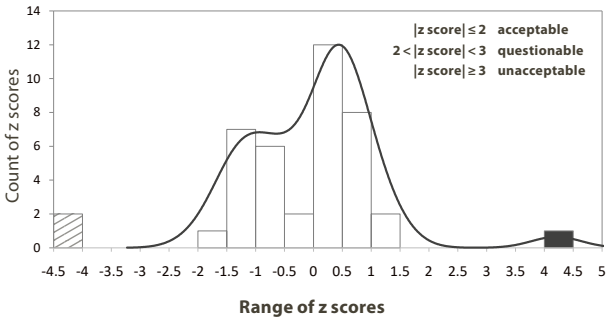
(Results from EU and EFTA Laboratories only)

Compulsory Compounds (cont.)

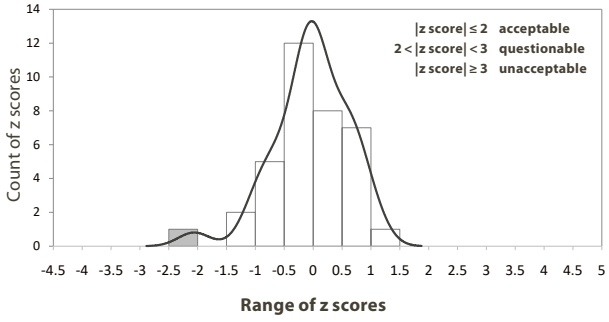
Matrine
(AV derived from entire population)



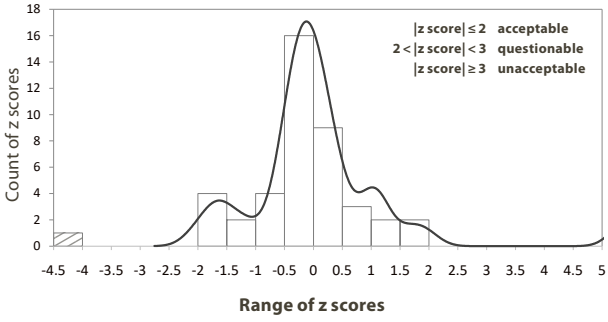
Nicotine
(AV derived from entire population excl. one outlier)



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

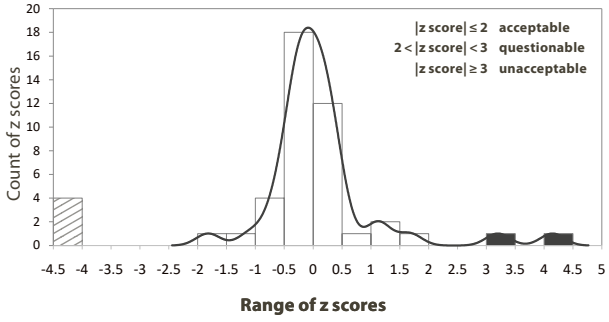


Phosphonic acid
(AV derived from entire population)

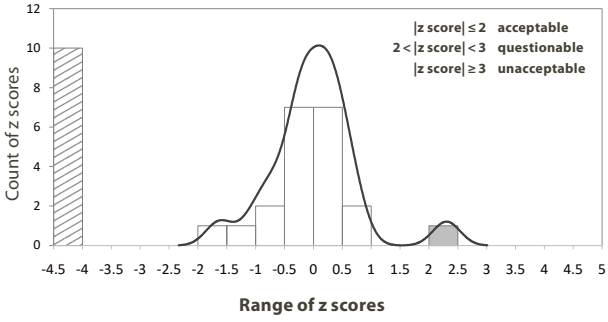


Optional Compounds

Perchlorate
(AV derived from entire population)

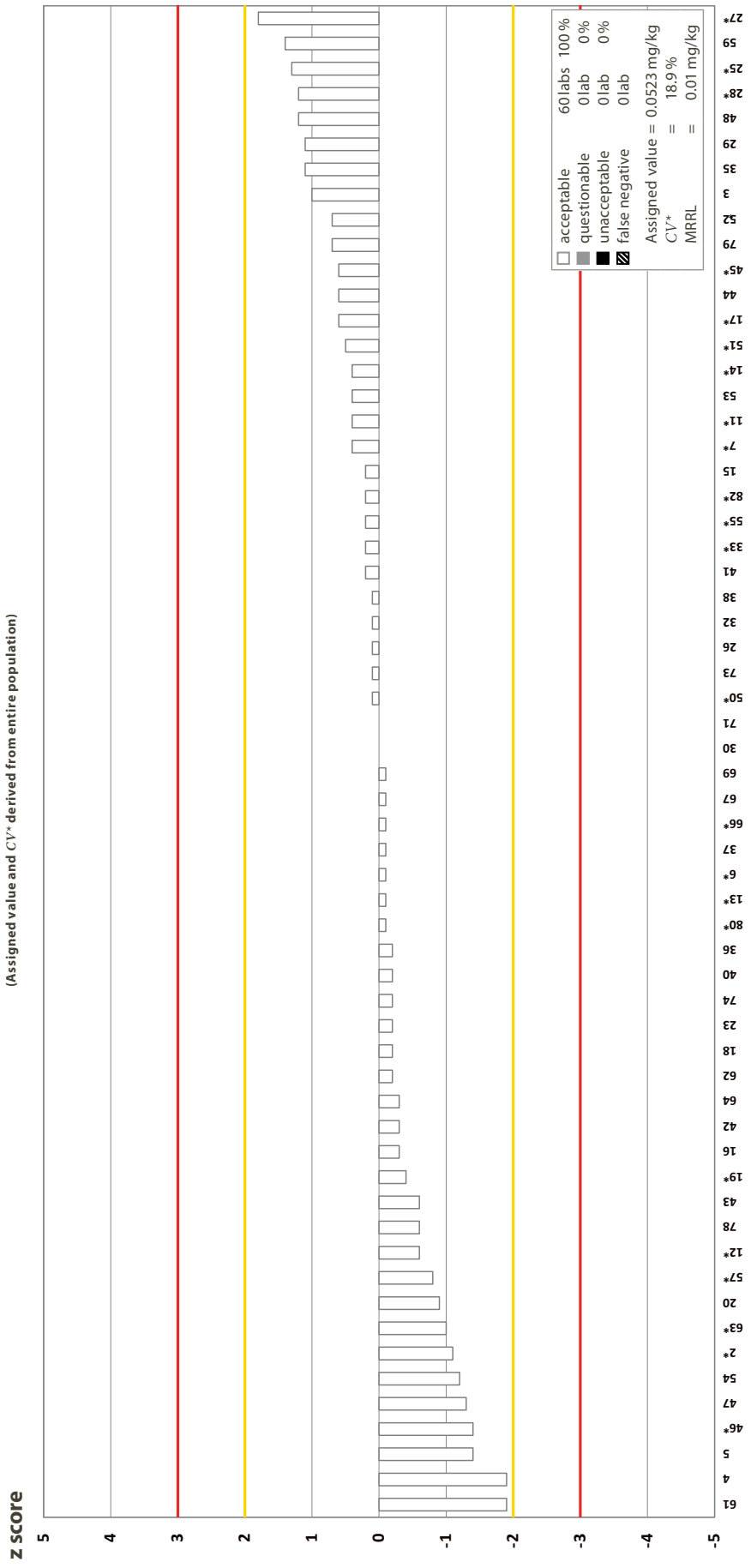


Trinexapac (free acid)
(AV derived from entire population)

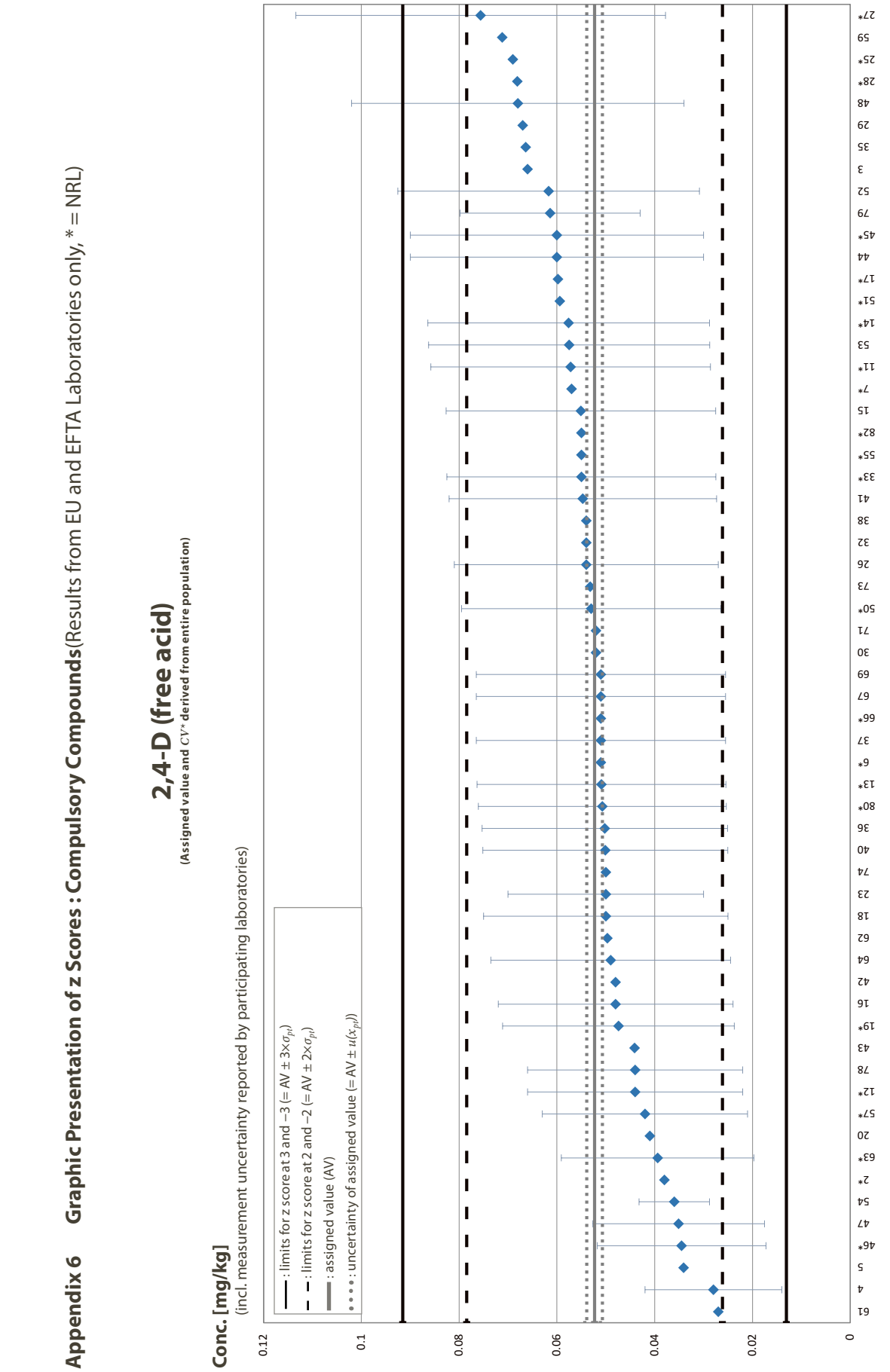


* Cut-off at z score = 5; ▨: false negative results

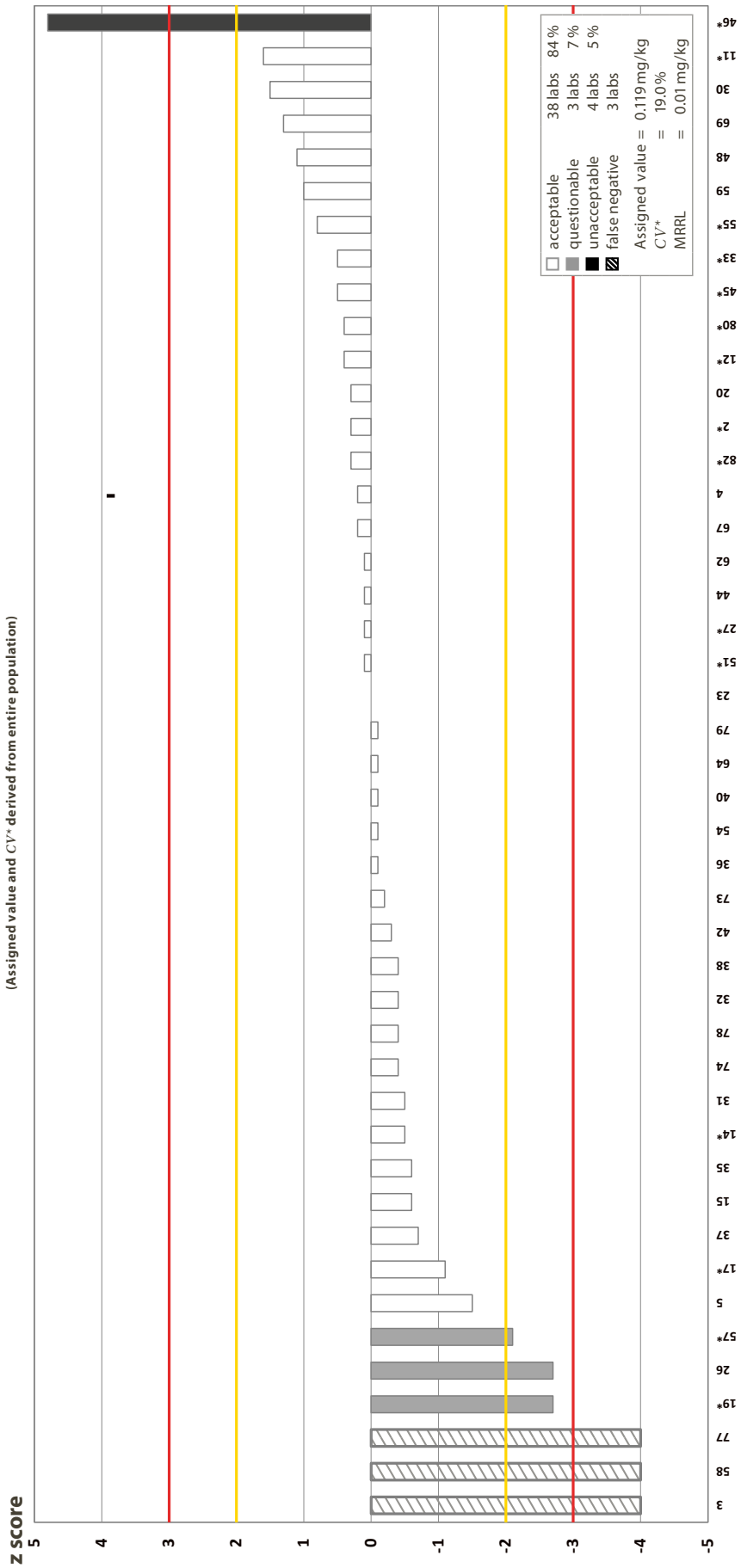
Appendix 6 Graphic Presentation of z Scores: Compulsory Compounds(Results from EU and EFTA Laboratories only, * = NRL)



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

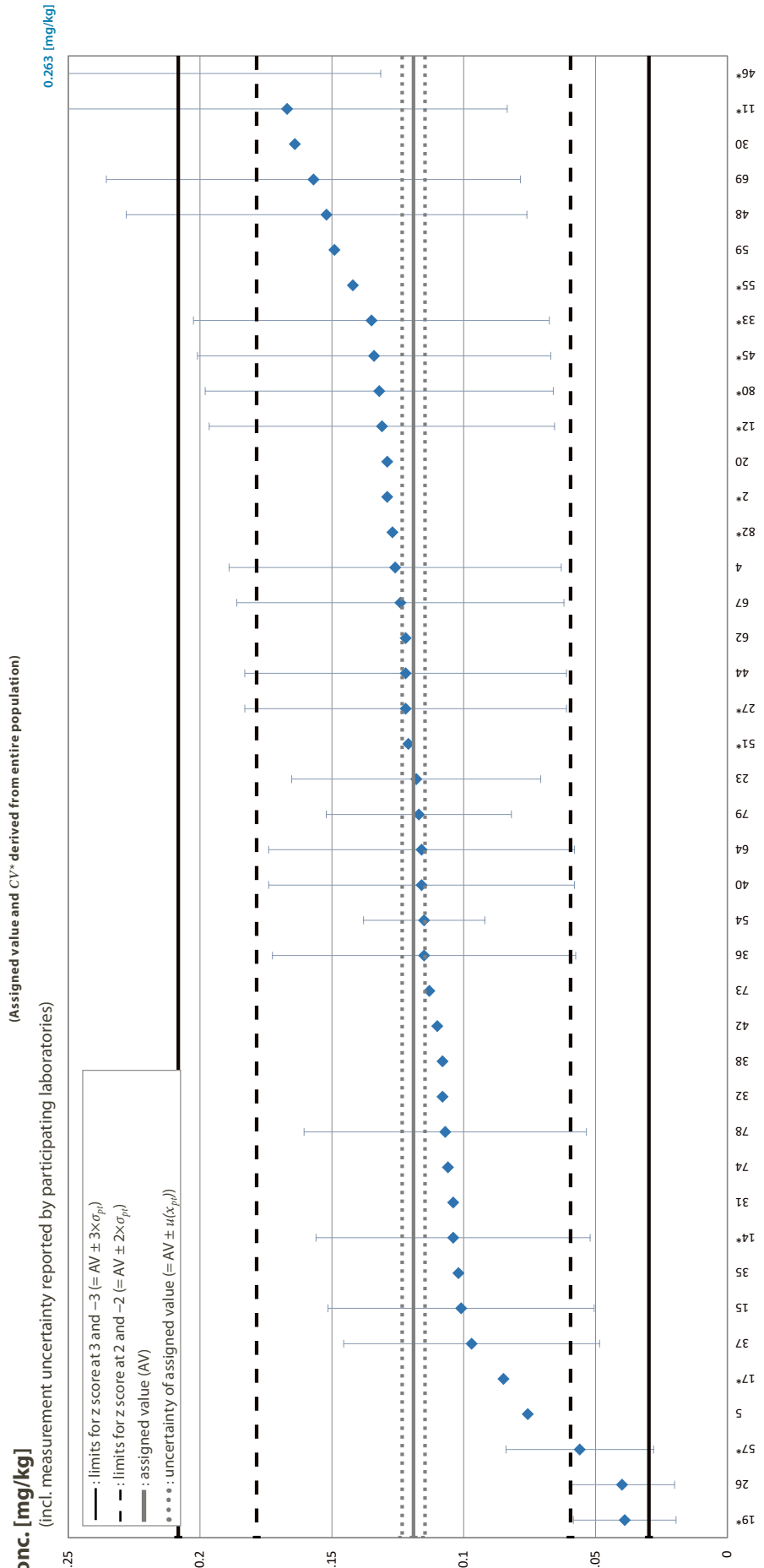


Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)

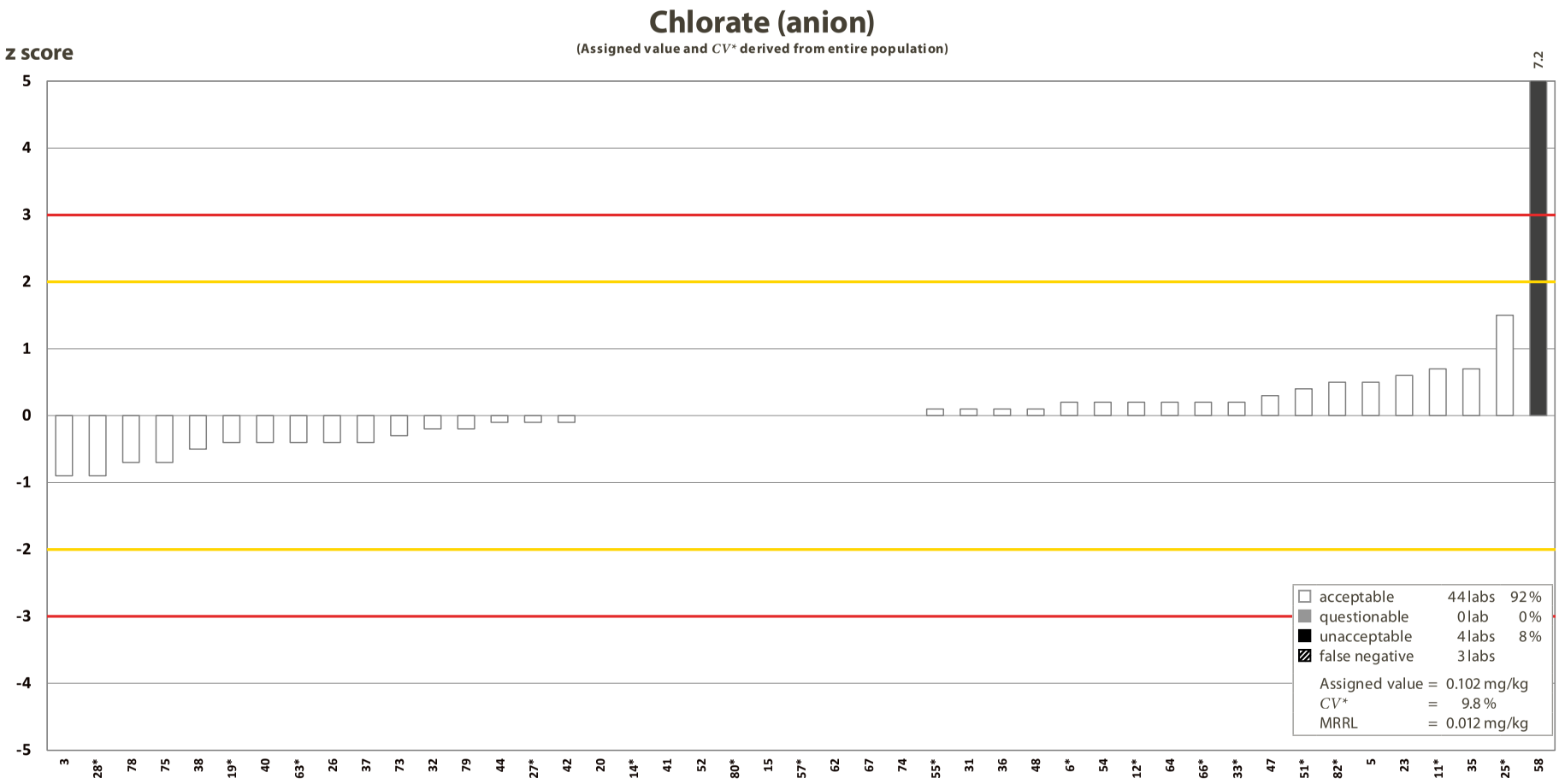


56

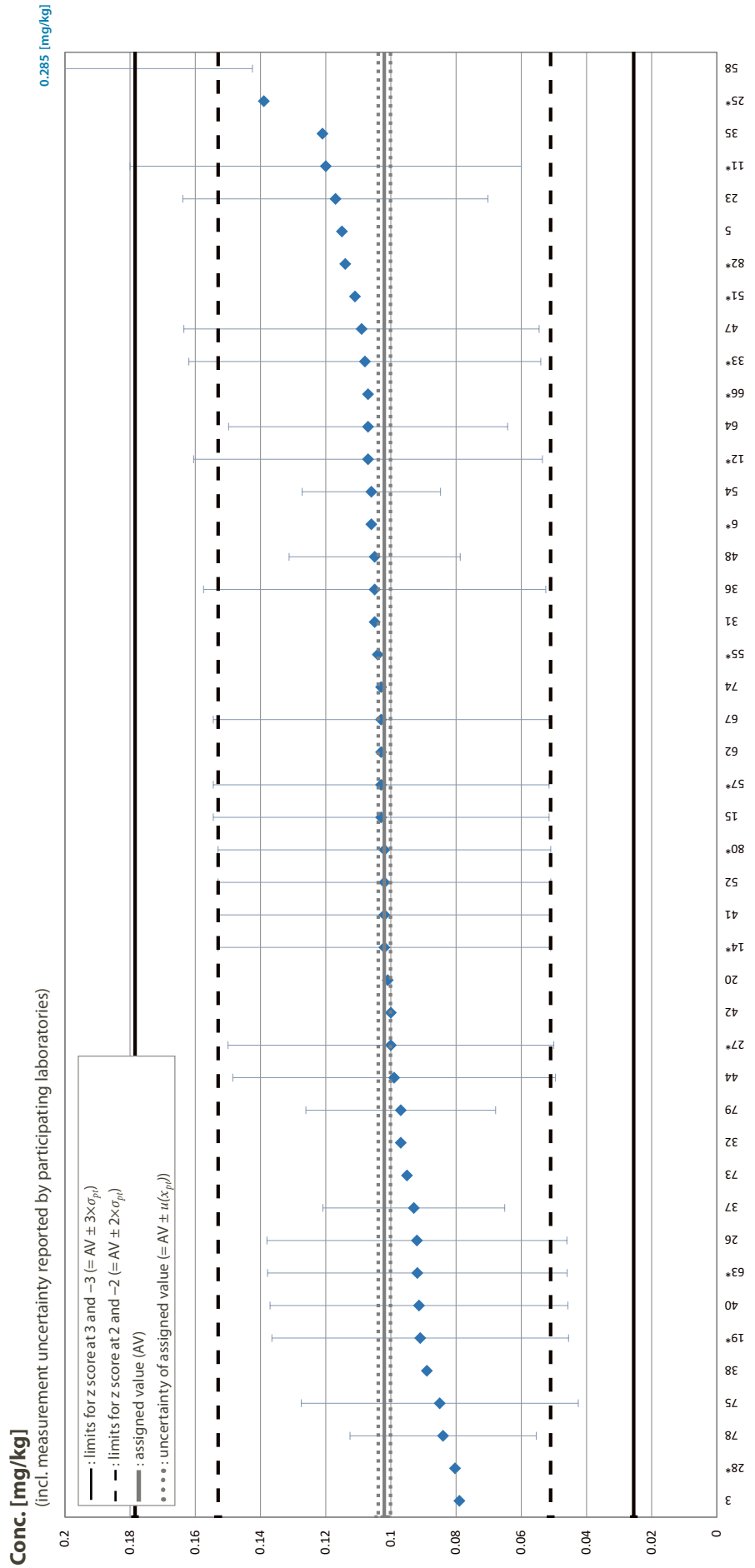
Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



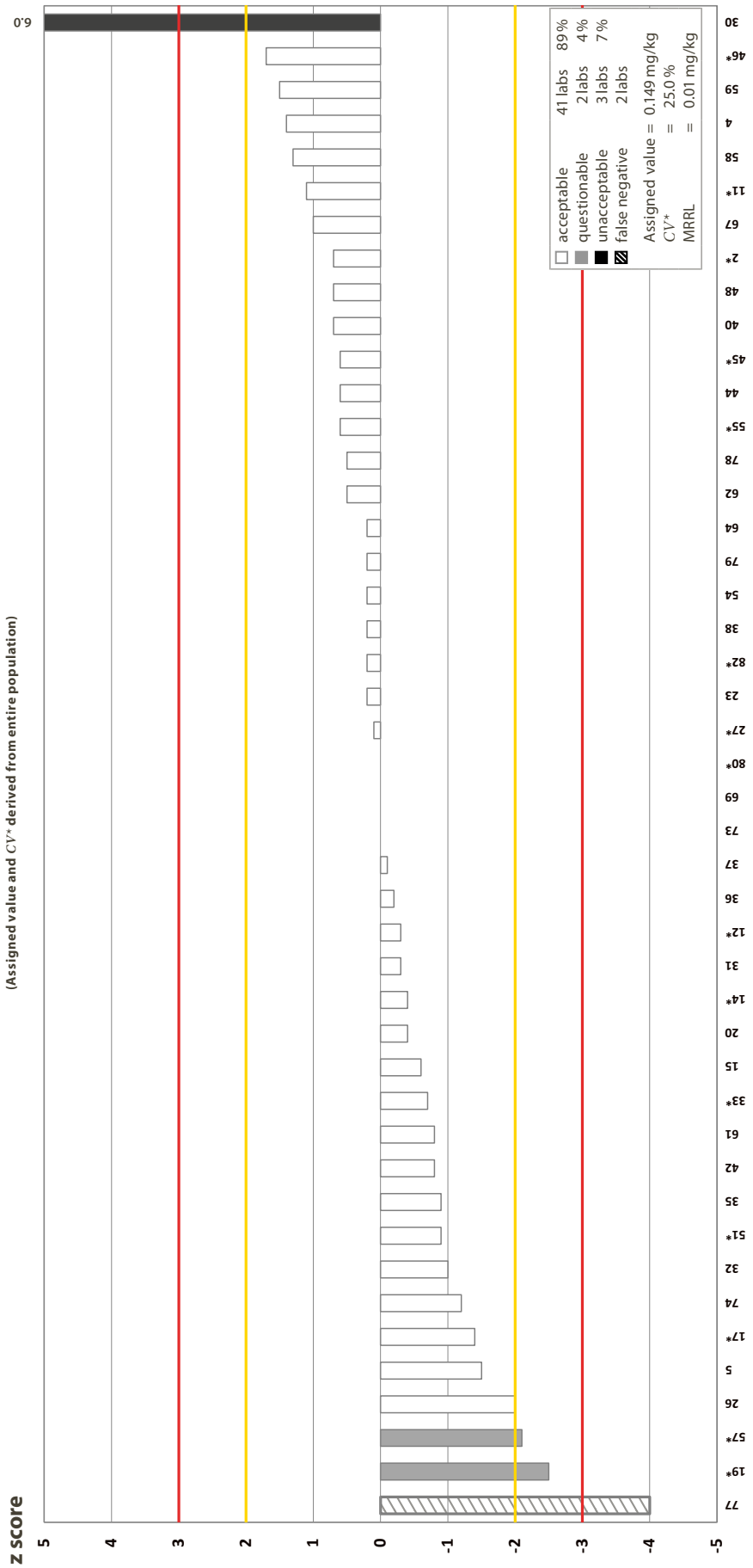
Appendix 6 (cont.) **Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



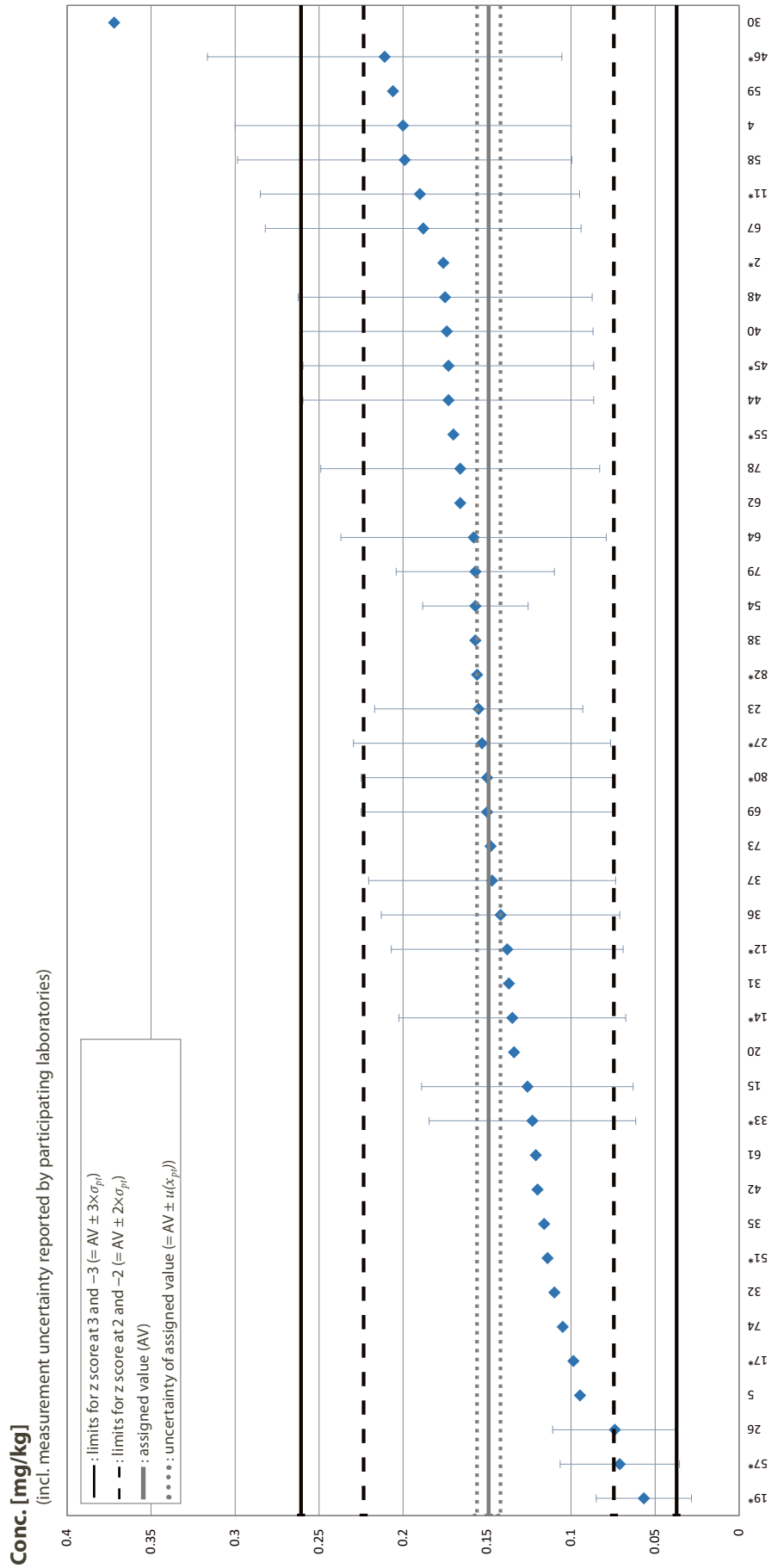
Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



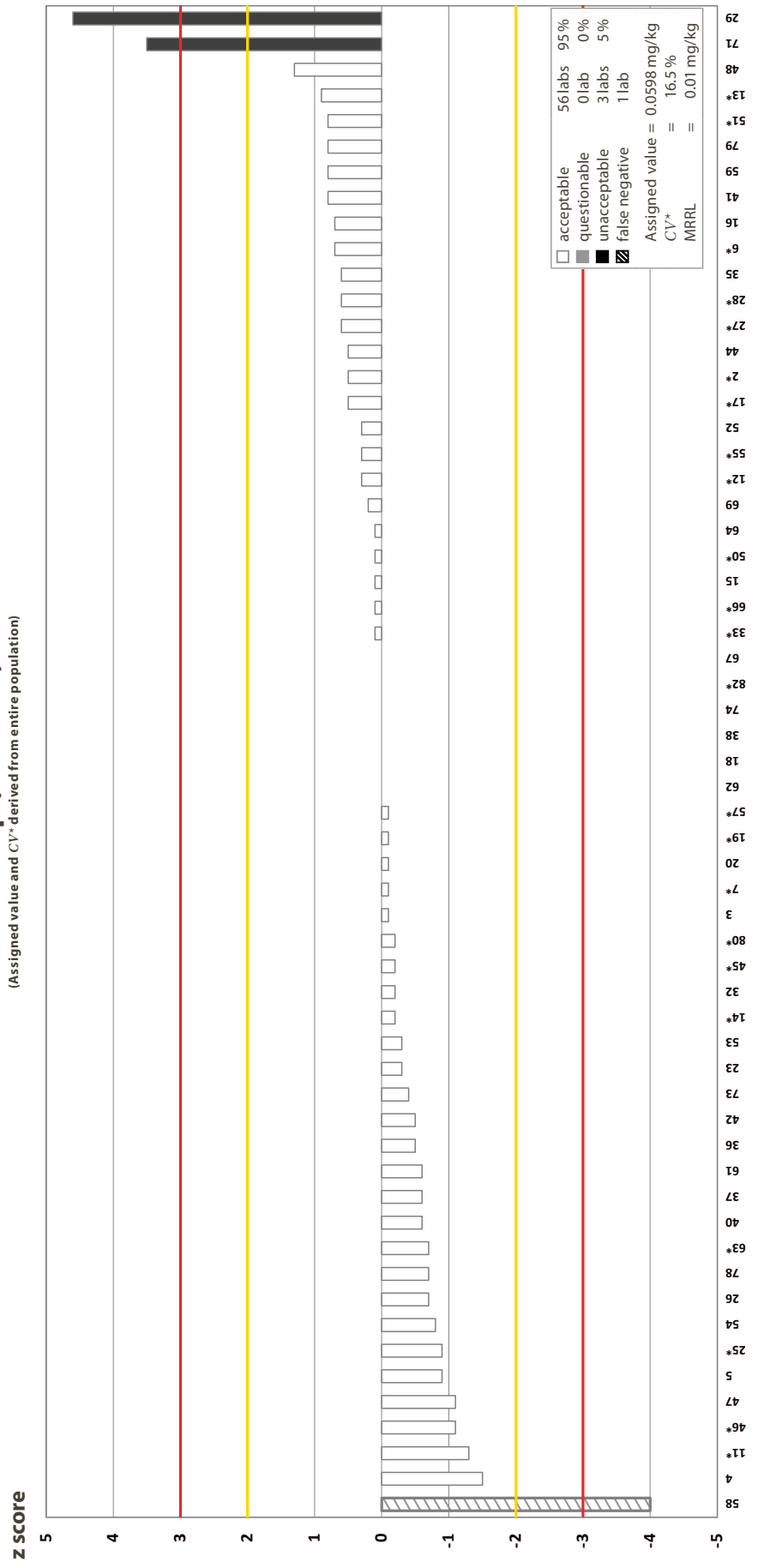
Appendix 6 (cont.) **Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



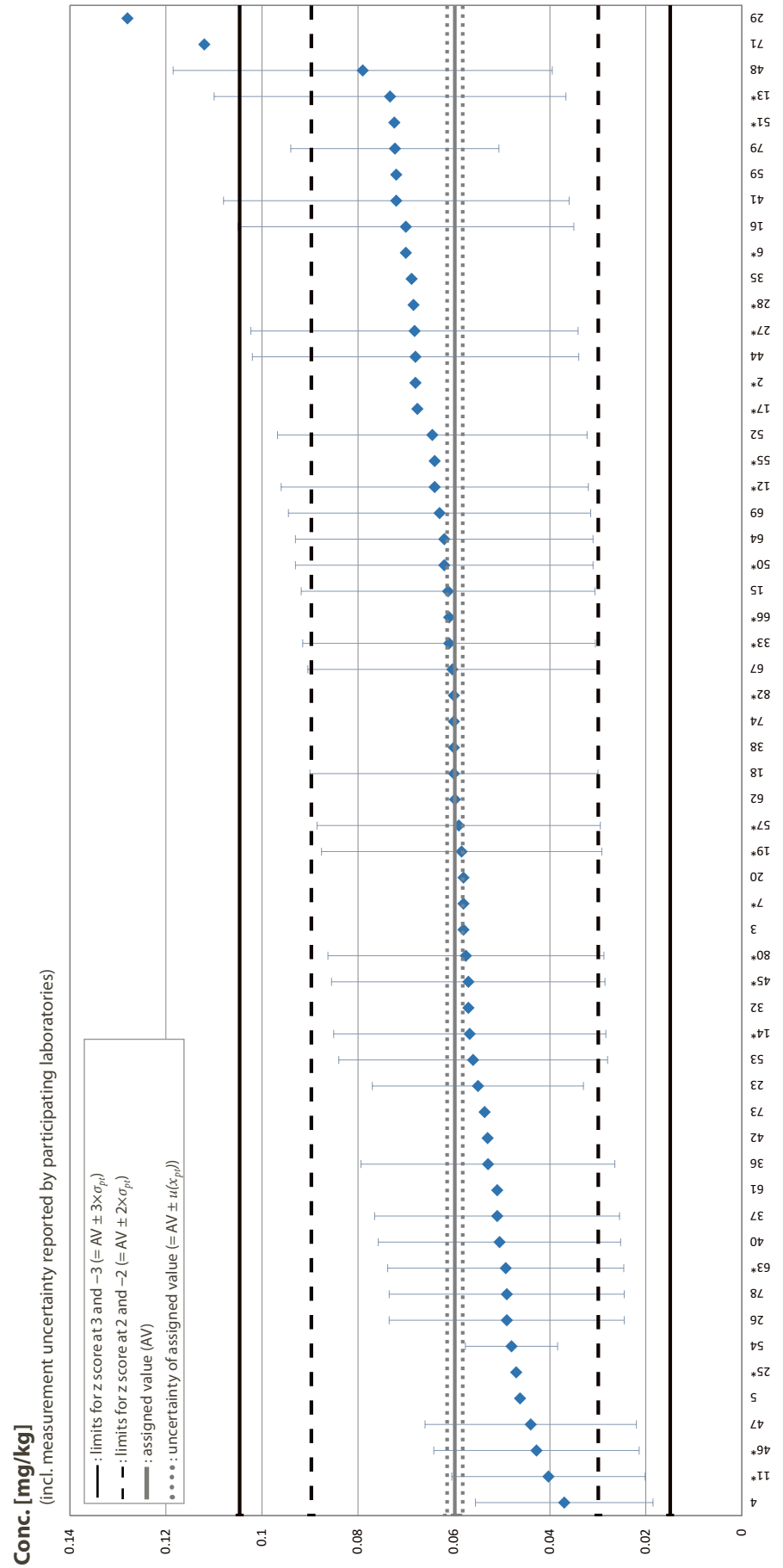
Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



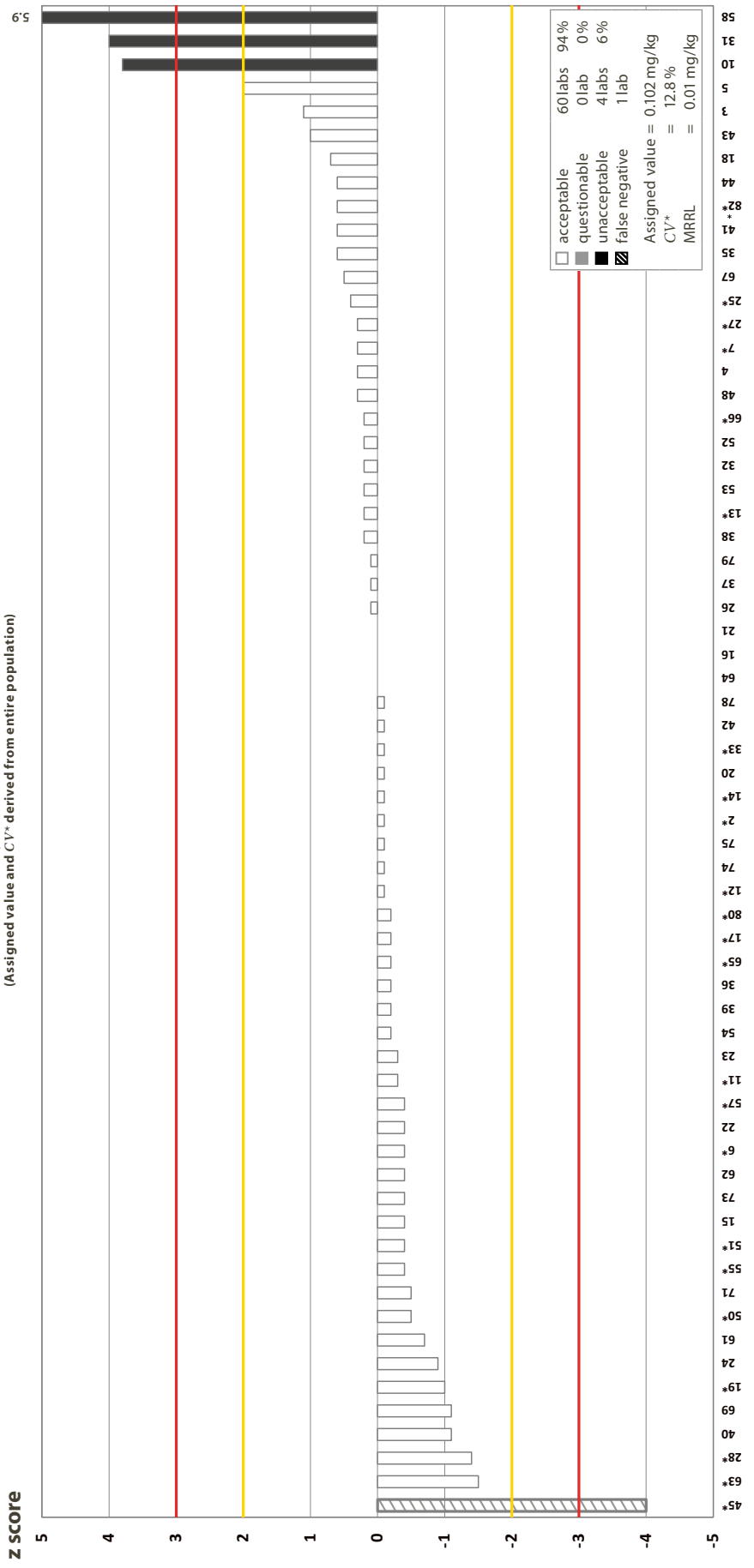
Appendix 6 (cont.) **Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



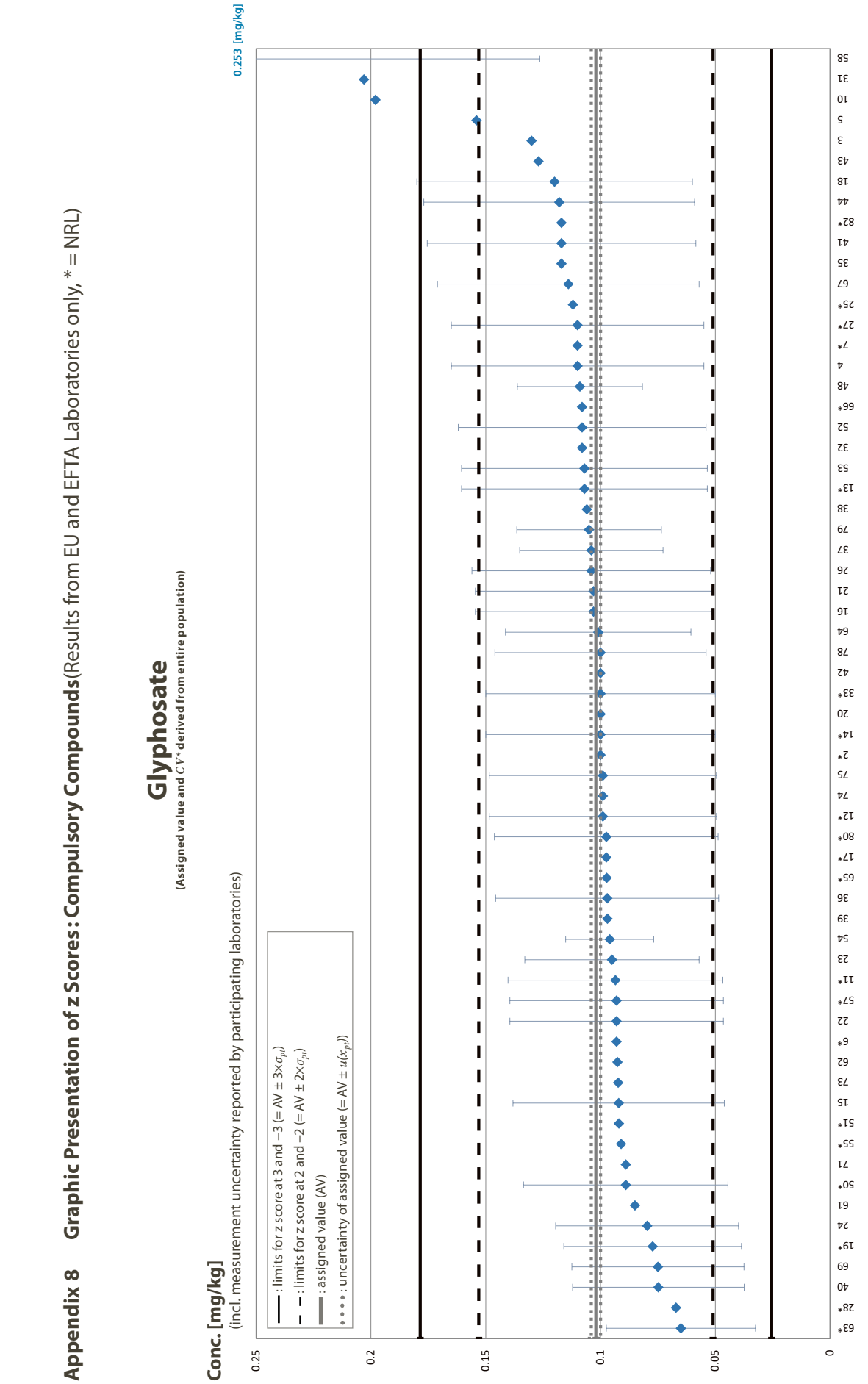
Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



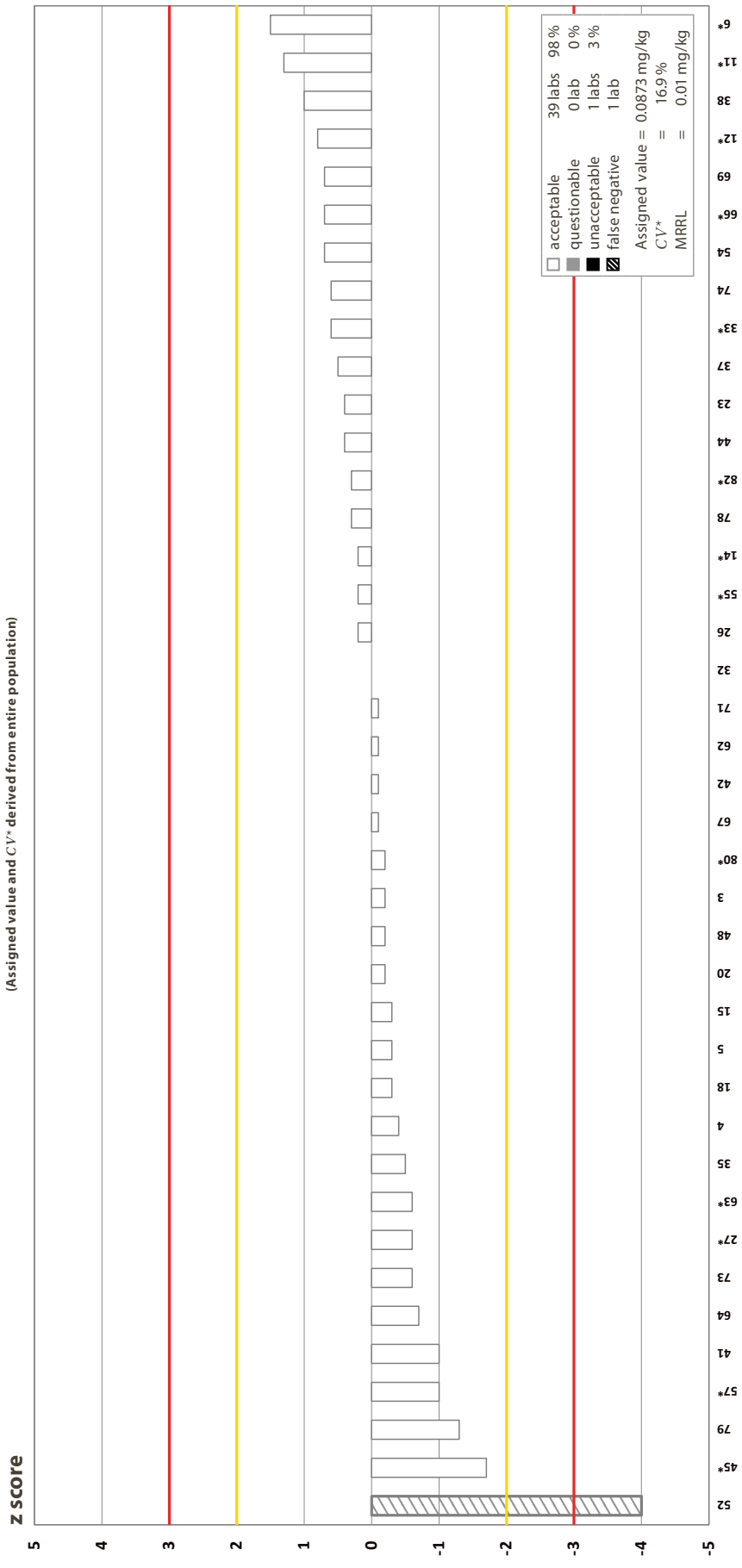
Appendix 7 Graphic Presentation of z Scores: Compulsory Compounds(Results from EU and EFTA Laboratories only, * = NRL)



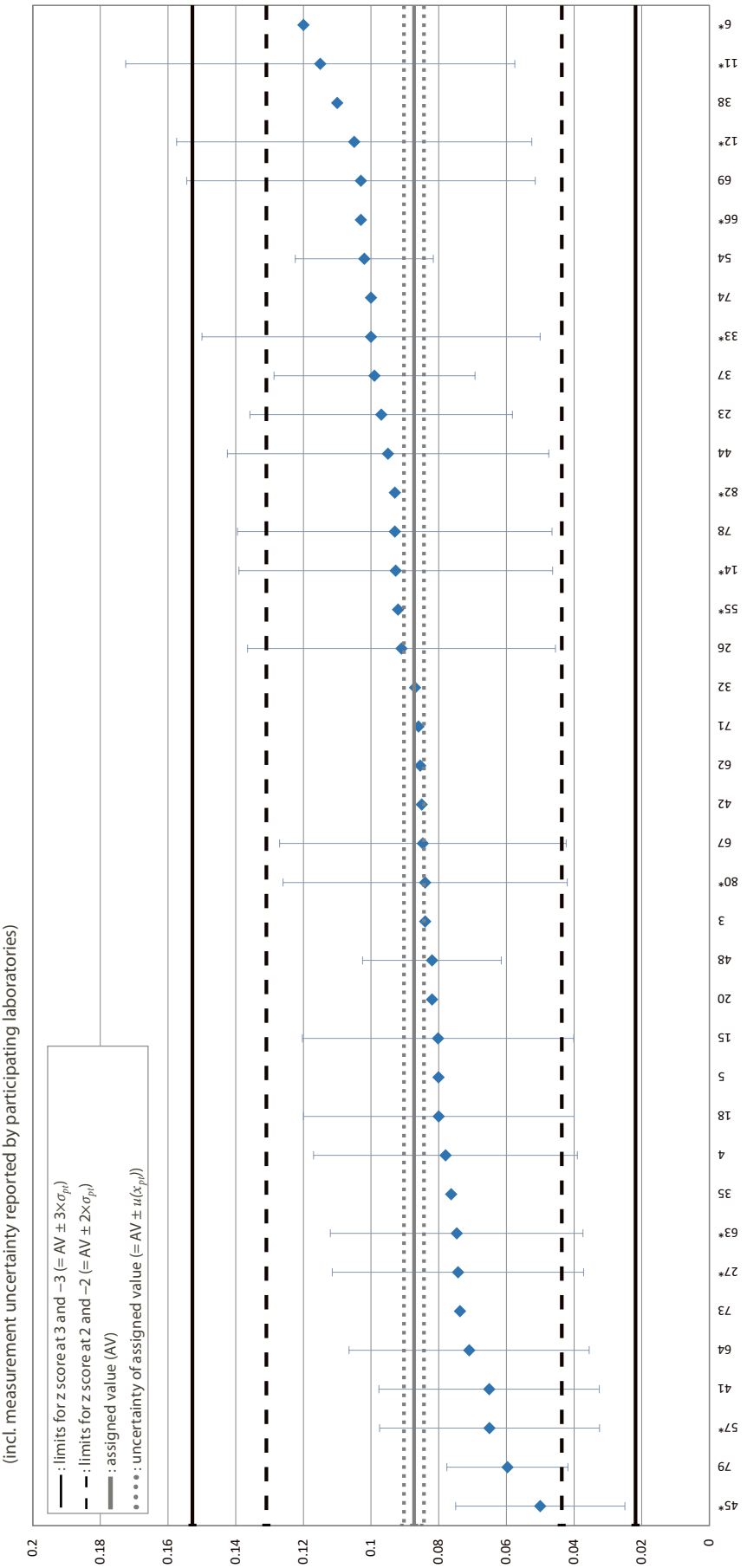
Appendix 8 Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)

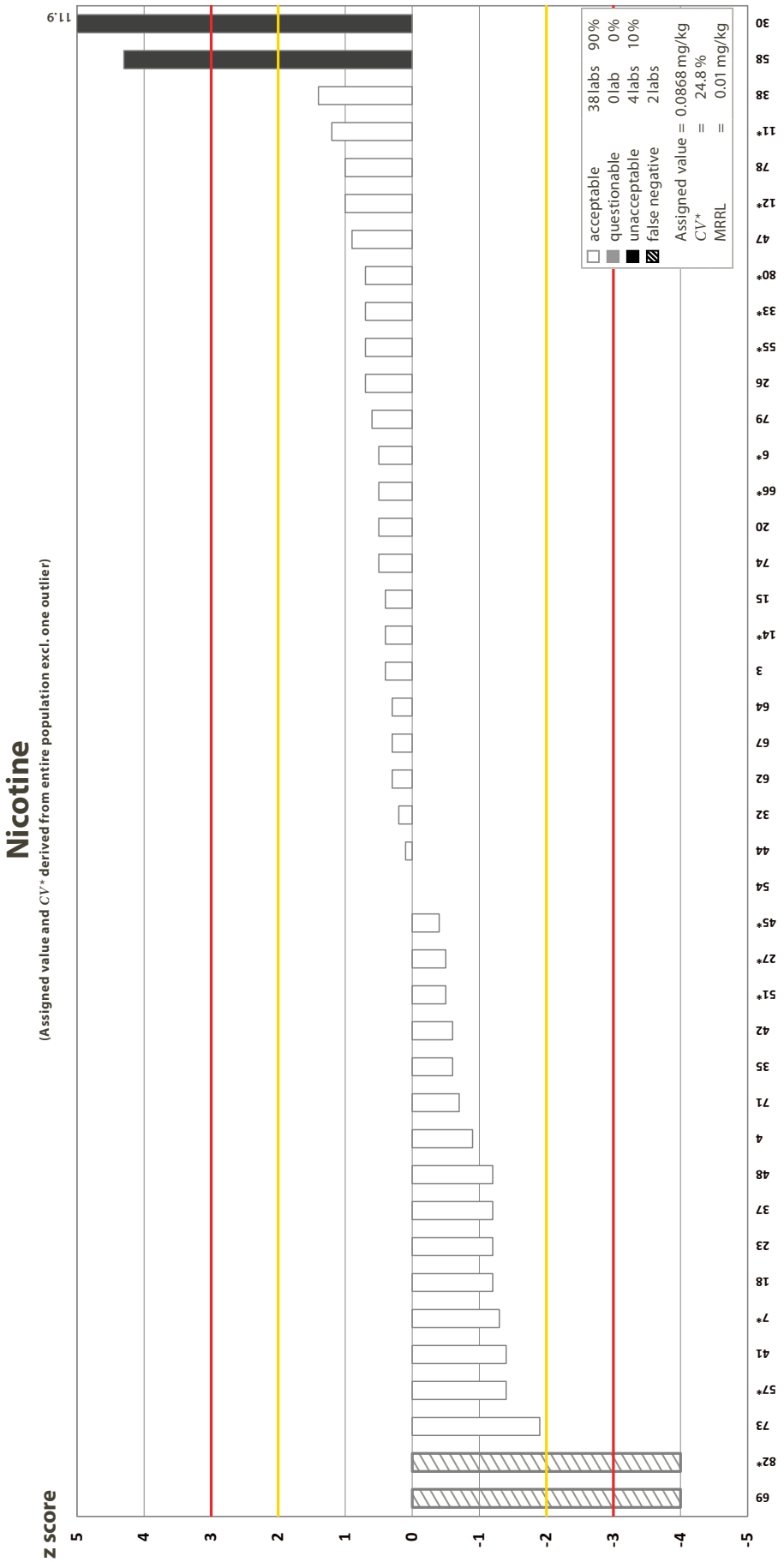


Appendix 6 (cont.) **Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)

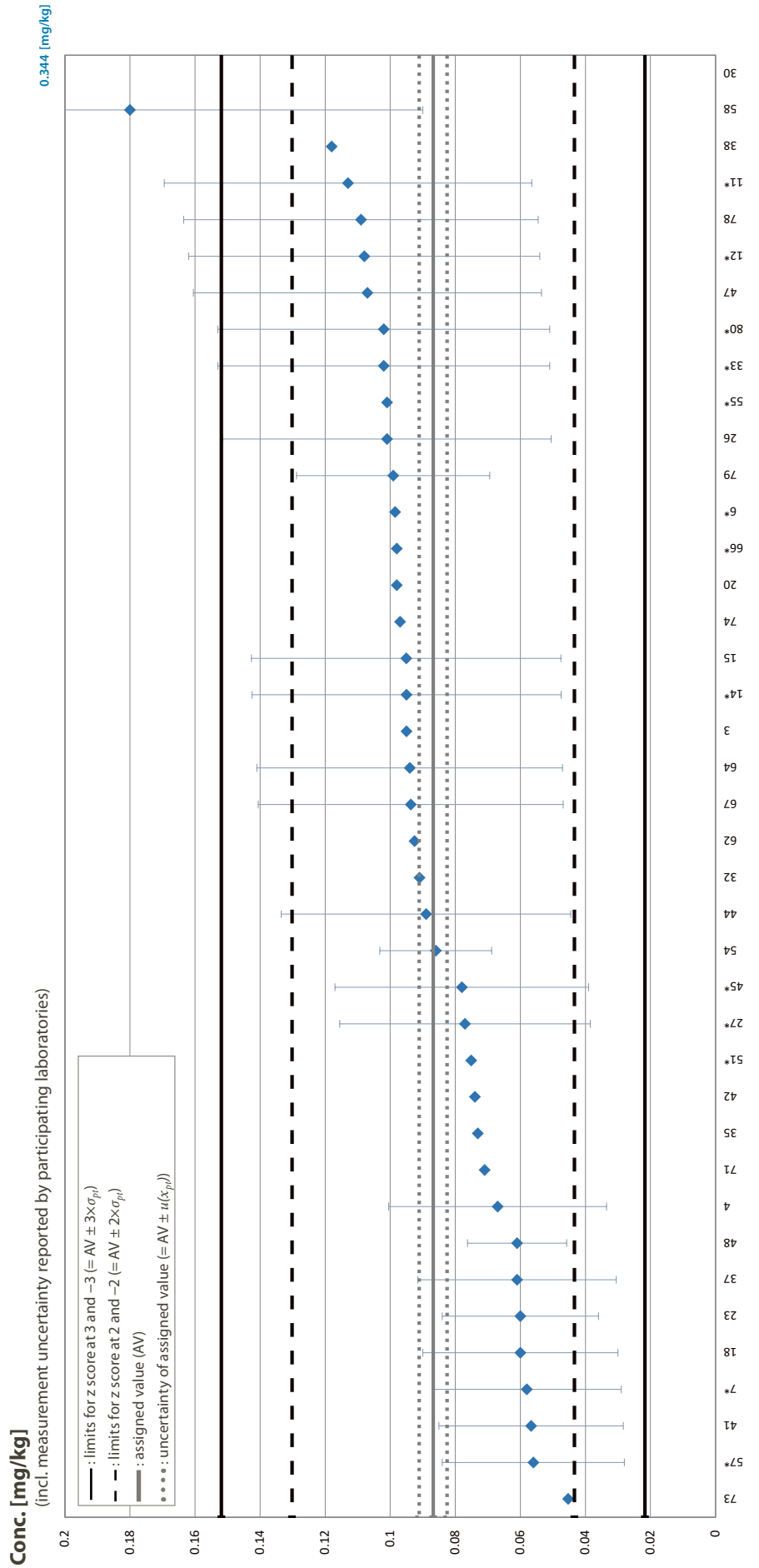


Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)

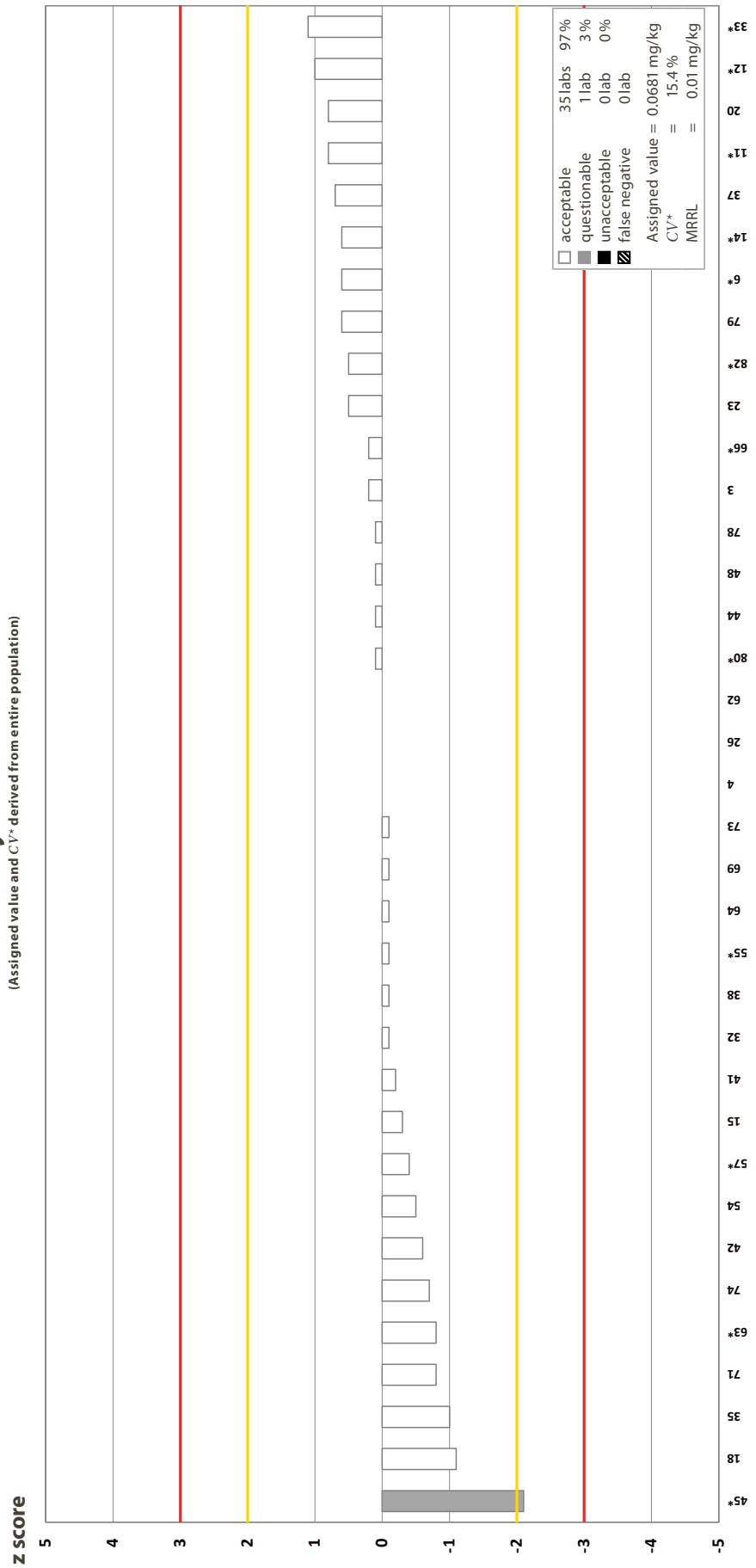



Nicotine
(Assigned value and CV* derived from entire population excl. one outlier)
z score
11.9
5
4
3
2
1
0
-1
-2
-3
-4
-5
30
58
38
11*
78
12*
47
80*
33*
55*
26
79
6*
66*
20
74
15
14*
3
64
67
62
32
44
54
45*
27*
51*
42
35
71
4
48
37
23
18
7*
41
57*
73
82*
69
acceptable 38 labs 90 %
questionable 0 lab 0 %
unacceptable 4 labs 10 %
false negative 2 labs
Assigned value = 0.0868 mg/kg
CV* = 24.8 %
MRRL = 0.01 mg/kg

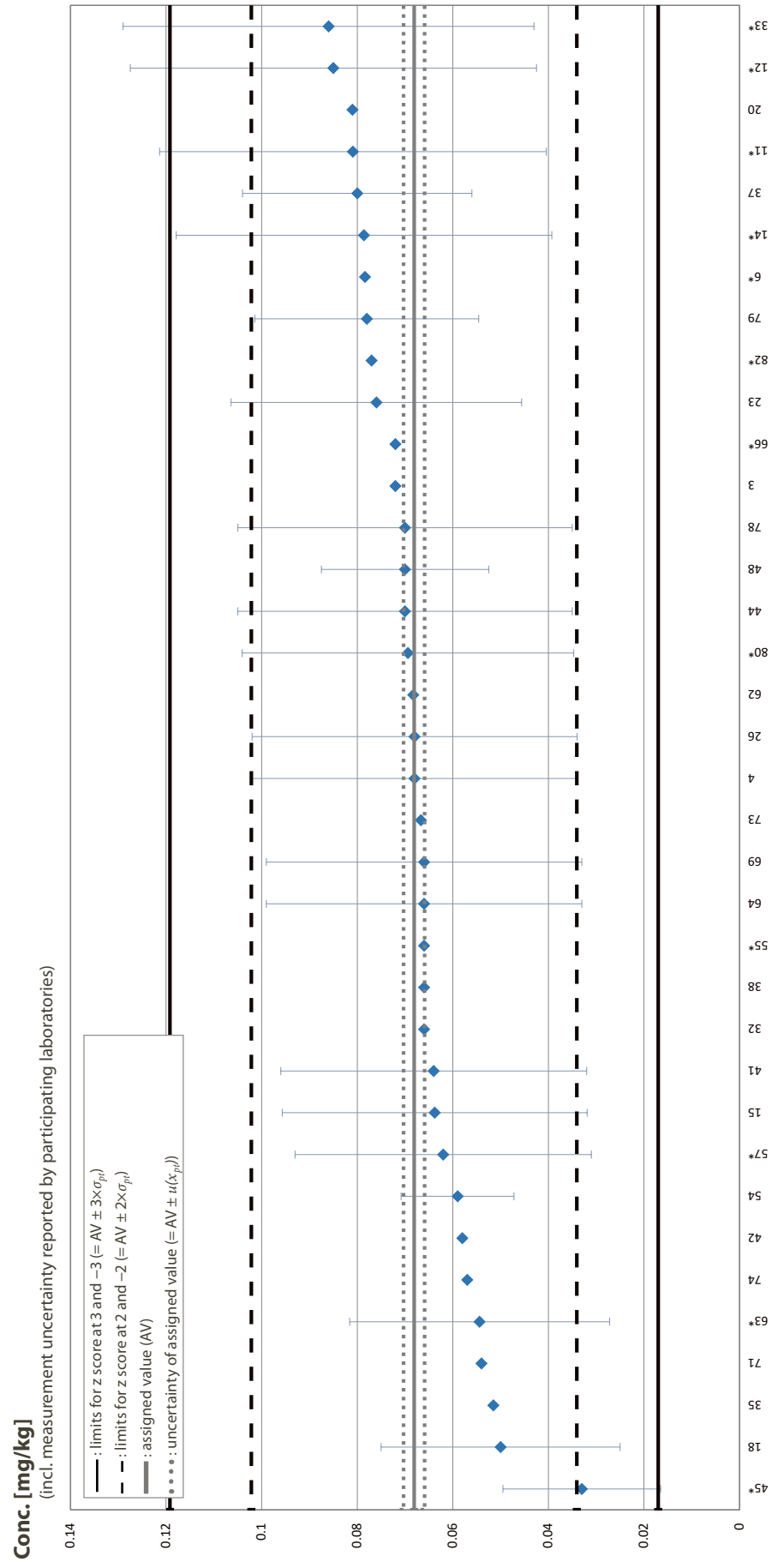
Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



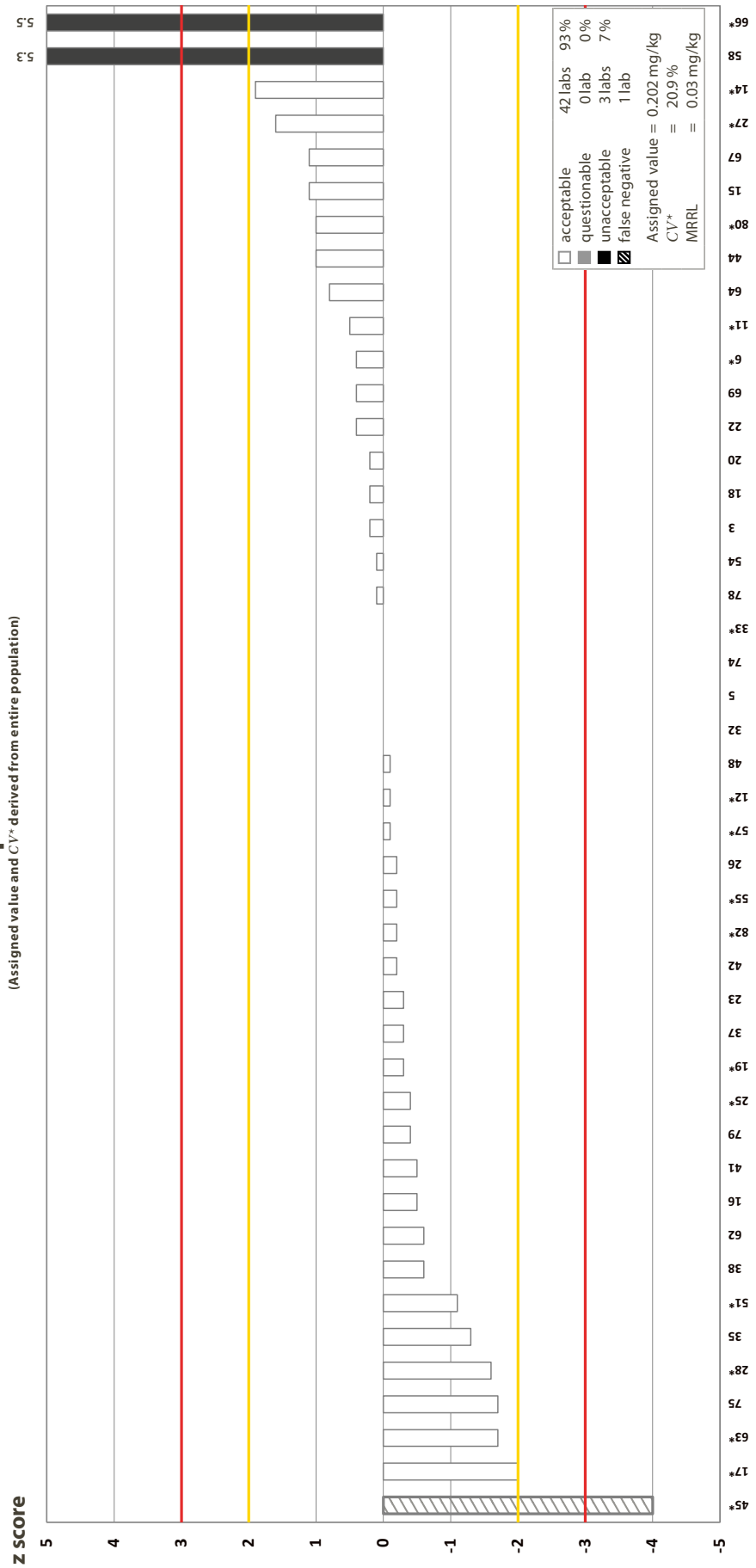
Appendix 6 (cont.) **Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



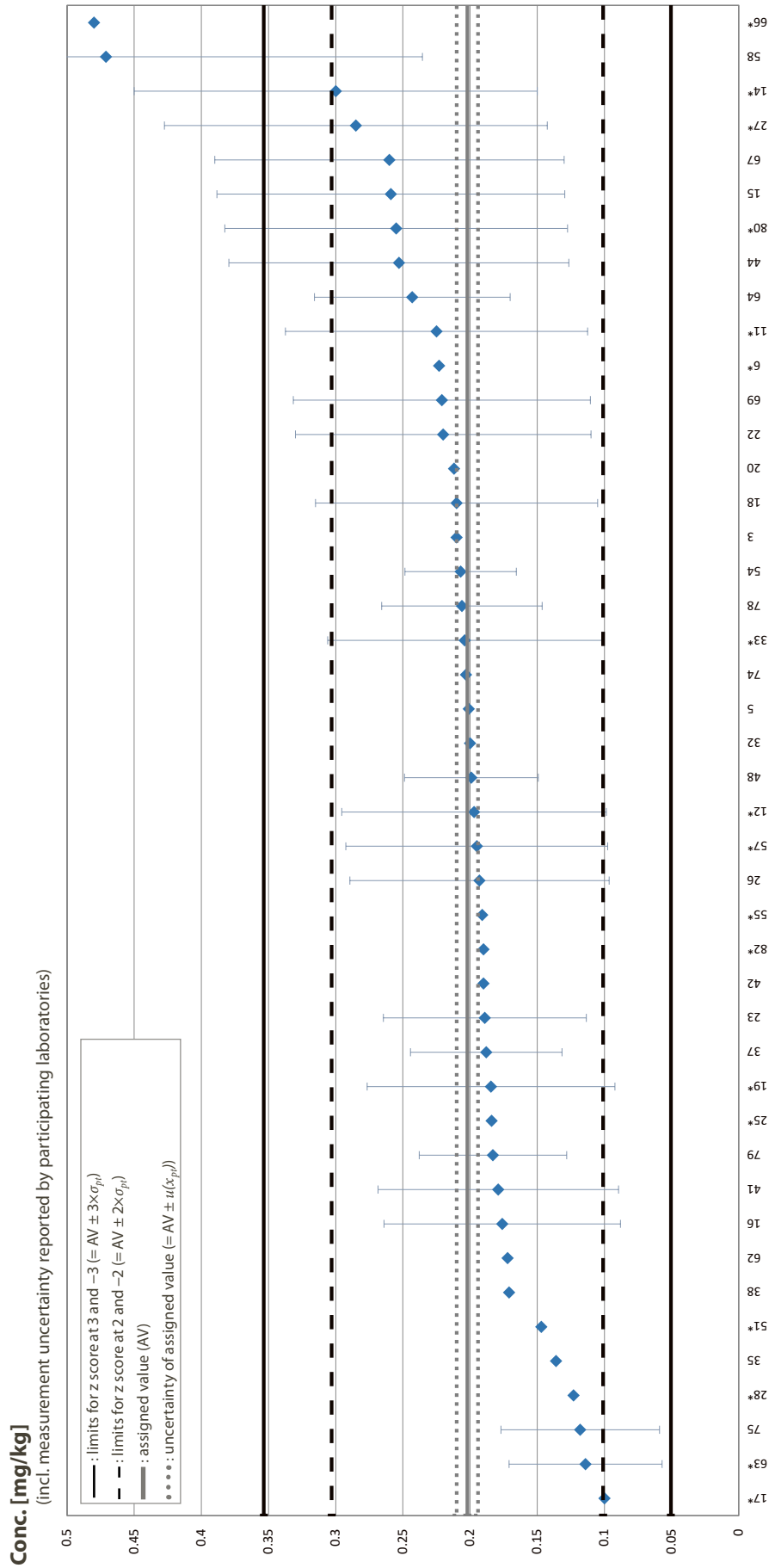
Appendix 6 (cont.) **Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



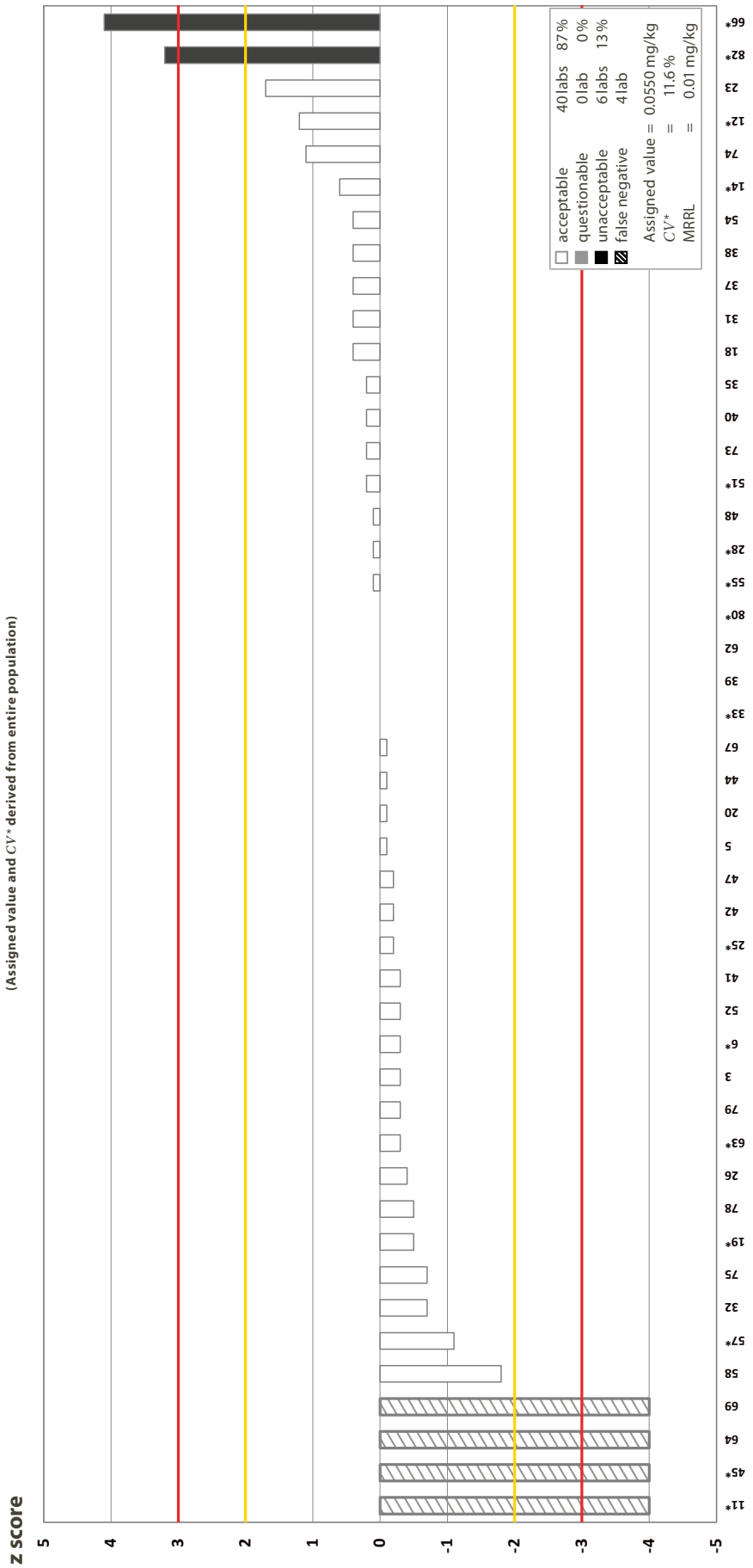
Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



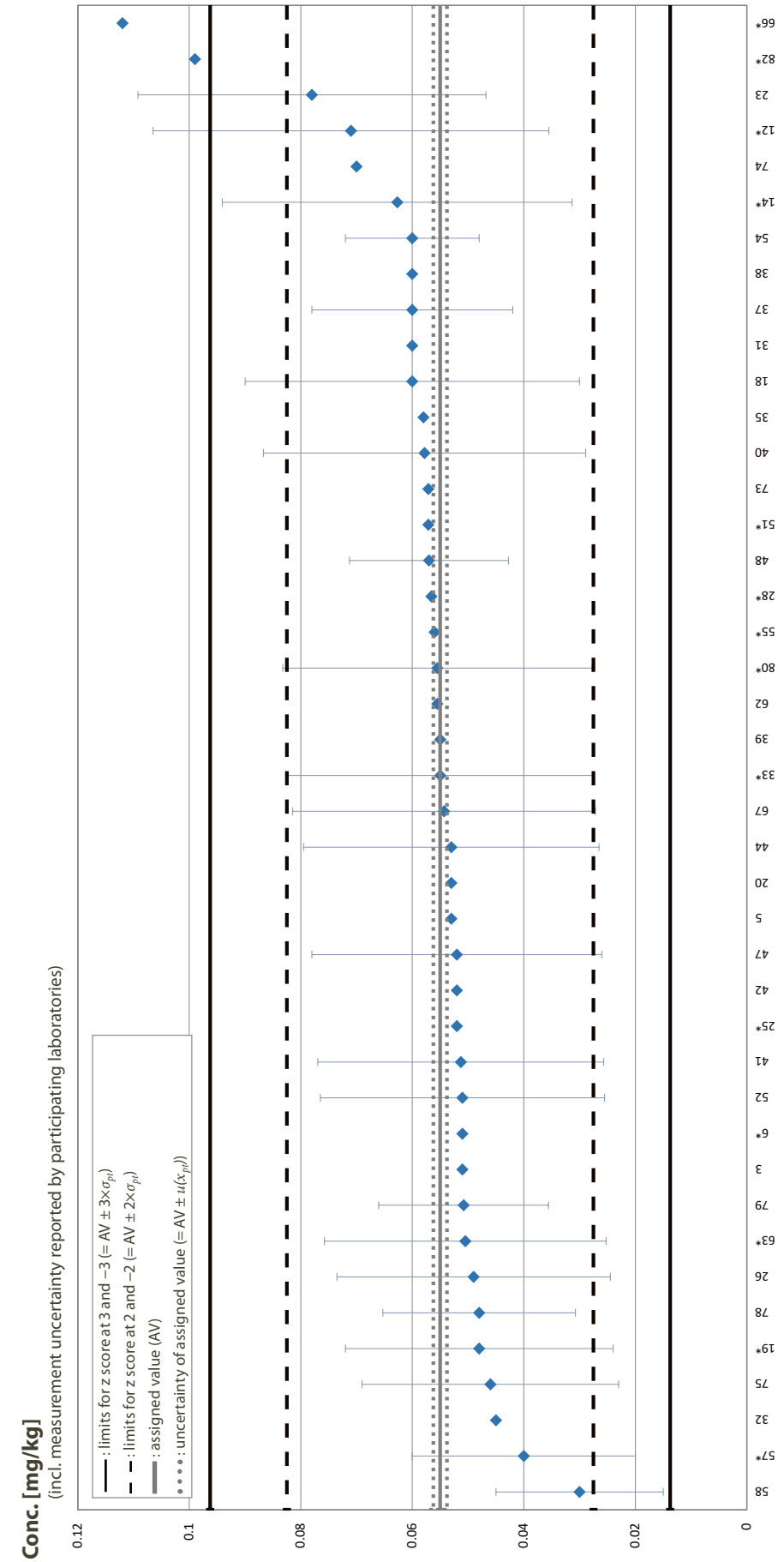
Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



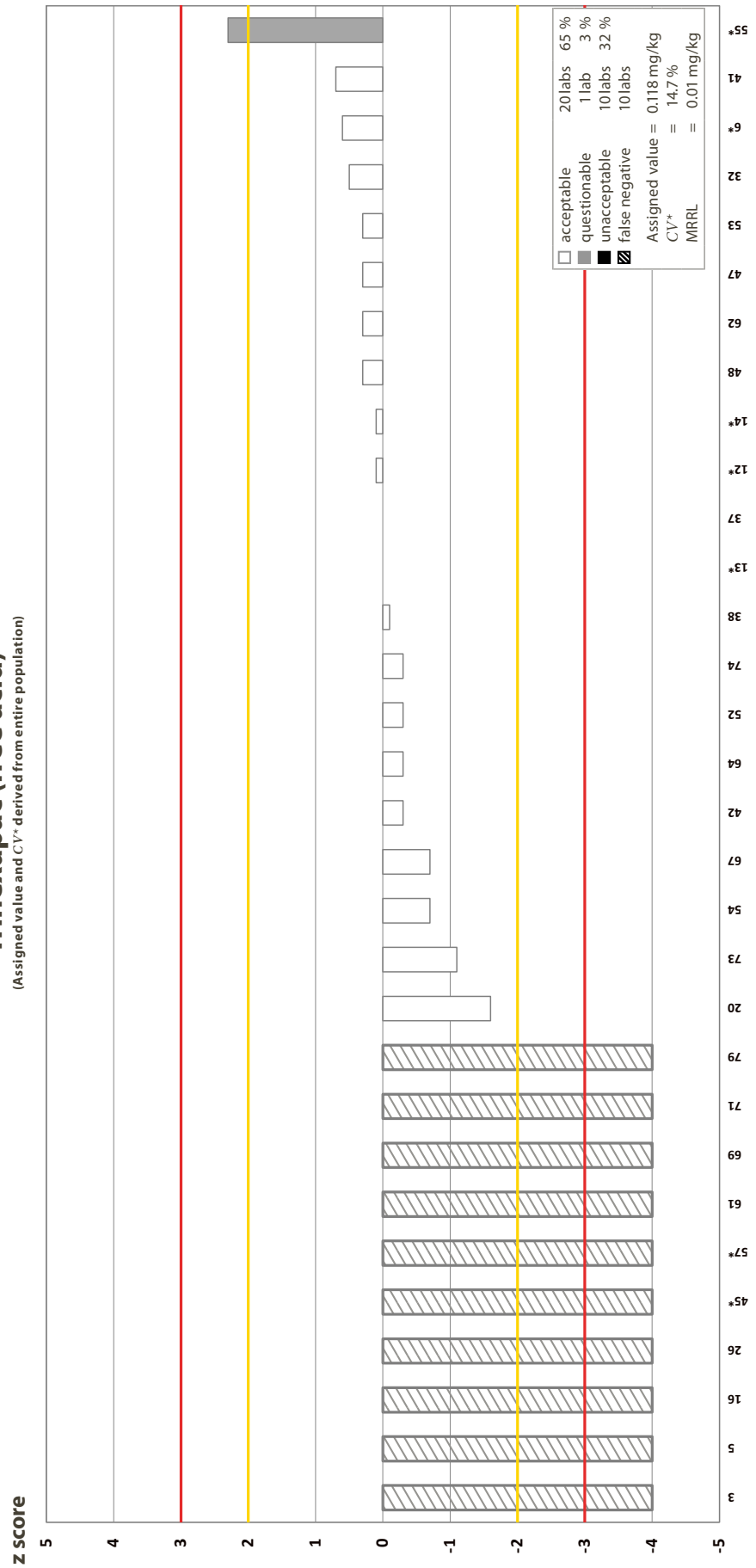
Appendix 6 (cont.) **Graphic Presentation of z Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



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



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



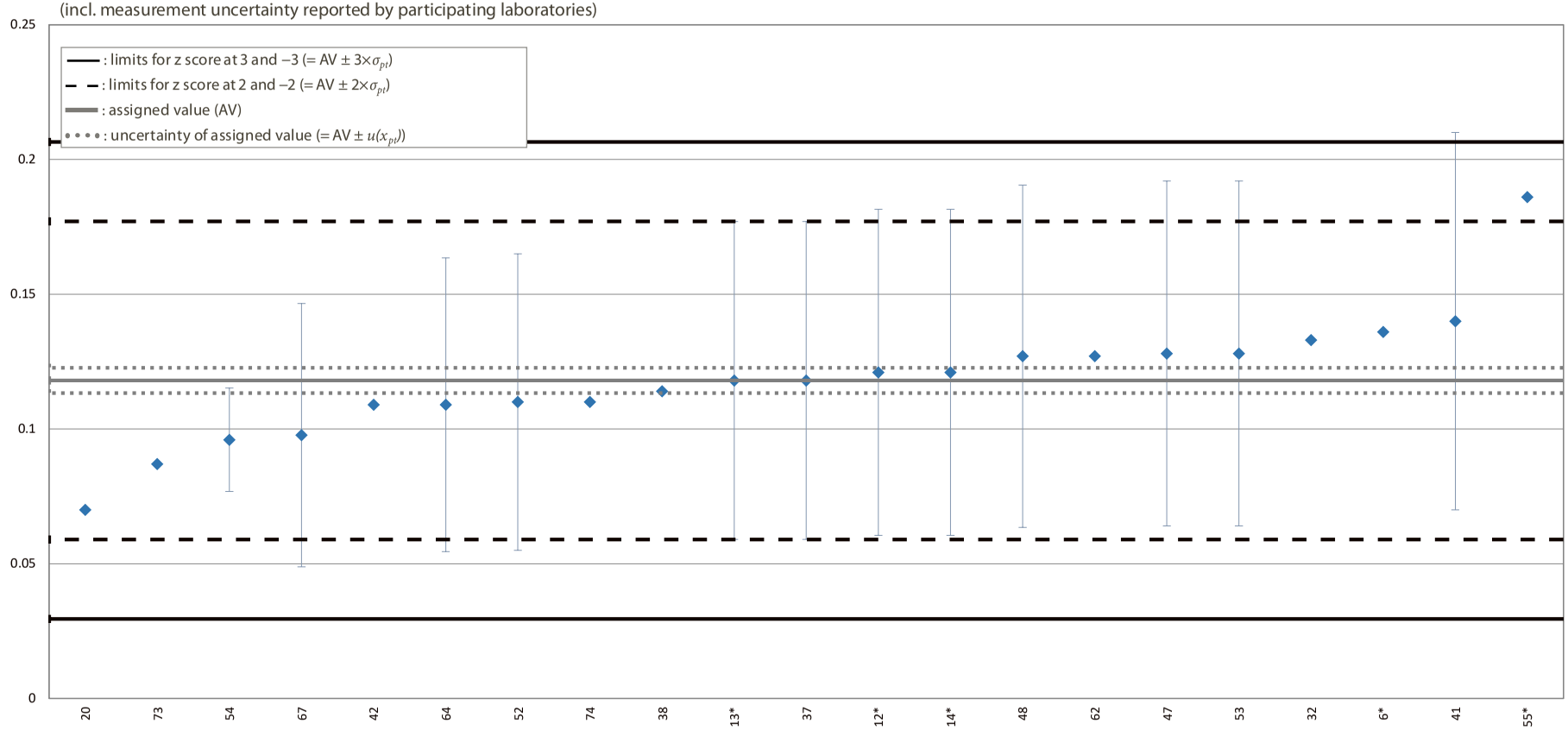
Appendix 6 (cont.) **Graphic Presentation of z Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)

Trinexapac (free acid)

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

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

BAC-C14 chloride Assigned value: 0.119 mg/kg, CV^*: 19.0 %

LabCode	z Score	Reason	Remarks/Details
3	-4.0 (FN)		**Comment from the organisers:** You have neither provided any method information nor any feedback regarding your FN result. Please note that the non-submission of method information or feedback may result in a refusal of the organisers to accept your participation in future PTs. Consider adjusting your QC procedures in a way that reduces the risk of FNs.
58	-4.0 (FN)	C	**Comment from the organisers:** It seems as if the procedure that you have employed (QuPPe) is principally suitable for QACs (as your result for DDAC-C10 was acceptable), but it seems that you need to review your QC measures to avoid FNs. Unlike for DDAC-C10 you haven't reported any recovery figure for BAC-C14.
77	-4.0 (FN)	C	We have broad experience with this compounds but in other foods. Honey was treated as vegetable (without water addition), and therefore we got a poor performance. As the quality control was done in other matrix we didn´t realize about the problem. After receiving your information about our poor performance the test was repeated (the sample was treated as a cereal, ie with water adition), obtaining the followind results: BAC14: 114 ug/kg and DDAC10: 162 ug/kg. So the problem is solved. **Comment from the organisers:** It is good that you managed to find the source of the problem and to take suitable corrective measures. Indeed, the analysis of honey via QuEChERS requires the addition of water. You haven't submitted any recovery data, so we assume that this was due to the fact that these were done on another matrix type. It is very important to use the same or at least a very similar matrix for QC experiments, especially since honey is not optimally represented other primary commodities. In your methodology information you indicated the use of a maching ILIS (BAC-C14 (D_7), which you have added at the beginning of the procedure. Despite this really optimal approach, you havent managed to avoid the false negative result. Normally, the ILIS should have given you a hint that something is wrong with the procedure. It seems that you need to adjust your QC measures.
19	-2.7	B	SRM18 was re-extracted and the same result for BAC-C14 chloride was obtained. Our analytical procedure does not work well for this pesticide and is not part of our accredited scope. We are looking at moving this pesticide from our multi residue method to a single residue method. **Comment from the organisers:** Indeed Luke/S19 type methods do not work well for QACs. Consider employing QuEChERS or to check whether you can screen or even fully cover those analytes by your method for highly polar compounds.
26	-2.7		**Comment from the organisers:**The procedure employed (QuEChERS) is principally suitable for QACs, but you may need to check whether matrix effects could be the cause for the bias. The same applies for BAC-C12 where you also had a negative bias.
57	-2.1	A	**Comment from the organisers:**The procedure employed (QuEChERS) is principally suitable for QACs. You seem to have taken care of matrix effects, by conducting calibration via standard additions. You havent reported any recovery figure, though.
46	4.8	I	The mistake was due to the following reason. As the MRRL for BAC14 and DDAC was half our LOQ/RL, we prepared a solution half the usual concentration. We did that to work the most similar way we usually do (it was easier for us to follow the usual steps and then taking it into account in the calculations). We used that solution to prepare the calibration curve. Then we used the usual Excel sheet to calculate the final concentration, but we forgot to divide by 2 the obtained result. So the correct value we should have sent for BAC14 is 0.263 /2 = 0.131 mg/kg. We recalculated its Z score and the value is 0.4. Also DDAC was in the same case, although the PT Z score was 1.7. The correct value for this compound should have been 0.105 mg/kg and the corresponding Z score would have been -1.2. **Comment from the organisers:** Good that you have traced back the source of the error. It indeed happens that labs need to deviate from their typical routine procedures when analysing PT samples, causing calculation or other errors that would not normally happen in a routine situation. Still, such errors help to increase vigilance.

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

Chlorate Assigned value: 0.102 mg/kg, *CV**: 9.8 %

LabCode	z Score	Reason	Remarks/Details
18	-4.0 (FN)	A, G	**Comment from the organisers:** Unfortunately, you failed to report the method information for this FN. Please provide this information in future cases to help the Organizers to potentially recognise the error sources and to suggest corrective actions. Consider adjusting your QC procedures in a way that reduces the risk of FNs.
45	-4.0 (FN)	F	Column equilibration, 1 injection in screening **Comment from the organisers:** In view of your FN results for chlorate, perchlorate, glyphosate, phosphonic acid and trinexapac, please consider checking your reporting limits and consider introducing additional QC measures to reduce the risk of FNs.
69	-4.0 (FN)	L	The measured value of Chlorate is 0,110mg/kg, human error in documentation and transcription.
58	7.2	C	**Comment from the organisers:** Consider employing an ILIS for reducing the impact of matrix effects on quantification. Also given the many highly biased results (chlorate, glyphosate, phosphonic acid, nicotine) consider checking for systematic calculation errors. Also consider receiving training on the proper use of the procedure and on quality control measures in general.

DDAC-C10 chloride Assigned value: 0.149 mg/kg, CV*: 25.0 %

LabCode	z Score	Reason	Remarks/Details
3	-4.0 (FN)		**Comment from the organisers:** Check organisers' remarks on your result for BAC-C14
77	-4.0 (FN)	C	We have broad experience with this compounds but in order foods. Honey was treated as vegetable (without water adition), and therefore we got a poor performance. As the quality control was done in other matrix we didn´t realize about the problem. After receiving your information about our poor performance the test was repeated (the sample was treated as a cereal, ie with water adition), obtaining the followind results: BAC14: 114 ug/kg and DDAC10: 162 ug/kg. So the problem is solved **Comment from the organisers:** Check the organisers' remarks on your result for BAC-C14
19	-2.5	B	SRM18 was re-extracted and the same result for DDAC-C10 chloride was obtained. Our analytical procedure does not work well for this pesticide and is not part of our accredited scope. We are looking at moving this pesticide from our multi residue method to a single residue method. **Comment from the organisers:** Check organisers' remarks on your result for BAC-C14
57	-2.1	A	**Comment from the organisers:** Check comments on BAC-C14
30	6.0	A	The analysis of these analytes in honey falls outside the scope of our laboratory, and the method employed has not been previously validated. **Comment from the organisers:** Even if you are not yet experienced with this analyte, it is still worthwhile checking for the source of the error. In many cases, the same type of error affects more than one analyte.

Fluazifop (free acid) Assigned value: 0.0598 mg/kg, CV*: 16.5 %

LabCode	z Score	Reason	Remarks/Details
58	-4.0 (FN)	B, D	**Comment from the organisers:** When analysing acidic pesticides, you shouldn't use PSA in dSPE cleanup. Nevertheless, it is douptful whether the dSPE losses were the main reason for your FN result as Fluazifop can be measured very sensitively by LC-MS/MS. Even with an 80% loss of fluazifop its level should remain higher than 0.01 mg/kg (your RL). In fact, dSPE with PSA will typically only remove parts of the fluazifop, especially where there is competition by matrix components (in this case sugars). Therefore, consider adjusting your QC procedures in a way that reduces the risk of FNs.
71	3.5	J	As the recoveries were good, lab's attention was directed to analytical standard checking, because it seemed to be the only reason for a mistake. The laboratory has initiated corrective actions. Results of repeated analyses are shown in the file. **Comment from the organisers:** Congratulations for tracing back the error-source and for taking corrective actions. This shows how valuable PT-participation can be. There is indeed an issue with fluazifop partly precipitating during the storage of stock solutions in acetonitrile. Prior to withdrawal of an aliquot for the preparation of the working solutions, any precipitate needs to be redissolved.
29	4.6		**Comment from the organisers:** Consider checking whether fluazifop has precipitated during the storage of your stock solution. This tends to happen when using acetonitrile as a solvent. See also the organizers' comment on fluazifop directed to lab SRM-71.

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

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

Glyphosate Assigned value: 0.102 mg/kg, CV*: 12.8 %			
LabCode	z Score	Reason	Remarks/Details
45	-4.0 (FN)	F	Column equilibration, 1 injection in screening **Comment from the organisers:** See the organisers' remarks on your FN result for chlorate.
3rd-70	-4.0 (FN)		**Comment from the organisers:** The organizers acknowledge that the delayed arrival of the sample has caused stress and gave too little time for addressing special issues. Judging from your method information, it seems you have taken measures to reduce bias, e.g. by using a matching ILIS and by calibrating via the approach of standard addition to sample portions. Still, you have obtained a false negative result for glyphosate. As you have also reported a FN result for oxymatrine, please consider introducing additional QC measures to reduce the risk of FNs and consider rechecking your reporting limits.
3rd-83	2.4	A, C, G	The amount of ILIS was different in the calibration and in the sample, we did not adjust the amount added to calibration vials with the final dilution we performed of the extracts When the sample was re-analyzed with the correct amount of ILIS we obtained the result 0.115 mg/kg **Comment from the organisers:** Congratulations for recognizing the error-source. This is the gain from participating in PTs.
10	3.8	J, A, K	We was starting with the analysis of Glyphosate. Now we are improving our method **Comment from the organisers:** Procedures involving derivatisation (FMOC in your case) need a lot of experience and attention to details. Consider the possibility of implementing an approach allowing direct analysis of glyphosate such as (QuPPe). Also consider the use of an ILIS to correct for various possible sources of errors.
31	4.0	J	Analytical standard was already three years old and probably concentrated. We prepared new calibration standards and have reanalysed the PT sample. By using the new standard we determined 0,131 mg/kg. **Comment from the organisers:** If your standard has concentrated down (due to evaporation), its concentration should have increased, leading to an underestimated rather than an overestimated result. Check whether your standard experienced losses. Losses may occur e.g. due to precipitation (e.g. if your solvent was not polar/protic) enough, or adsorption to glas surface (most noticable in low concentrated standards) or due to reaction (e.g. if methanol was used as solvent this may have resulted in methylation of the acid group or if an organic acid was added to the standard this may have resuled in an acylation of the amino-group). Consider the possibility of implementing an approach allowing direct analysis of glyphosate, such as (QuPPe).
58	5.9	C	**Comment from the organisers:** Check comments on your result on chlorate.

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

Matrine Assigned value: 0.187 mg/kg, CV*: 16.9 %

LabCode	z Score	Reason	Remarks/Details
52	-4.0 (FN)	C	We couldn't do a retest due to lack of material. **Comment from the organisers:** Note that there is a possibility of ordering a double amount of test material. Please also consider introducing suitable QC measures to reduce the risk of FN results. This may include the use of an ILIS.
3rd-70	-4.0 (FN)		**Comment from the organisers:** Check the organisers' remarks on your result on glyphosate and consider introducing the use of an ILIS.
3rd-76	3.0	A, G, M	We used mepiquat D_3 as IS and it may have been inappropriate for this compound, we didn't have adequate IS; Also we had only few days for analysis as the sample arrived on 29.06, and we submitted results on 07.07. **Comment from the organisers:** Indeed, mepiquat-D3 as IS will mainly correct for volume deviations, not for matrix effects, which can be very different from matrix to matrix and from analyte to analyte within the same matrix..

Oxymatrine Assigned value: 0.0681 mg/kg, CV*: 15.4 %

LabCode	z Score	Reason	Remarks/Details
3rd-70	-4.0 (FN)		**Comment from the organisers:** See the organizers remark on your FN result for glyphosate.
45	-2.1	F	Column equilibration, 1 injection in screening **Comment from the organisers:** It is not clear what has caused the bias in your case, as you have employed an appropriate ILIS at the beginning of the procedure. It would make sense to check whether there is any error in the calculation. As you have used the same procedure for matrine and oxymatrine and as you obtained a simmilar bias for both, it would be reasonable to include both in your investigations for the sources of error. The two compounds show very similar physicochemical properties and a similar analytical behavior. Interrestingly, your bias in the case of nicotine (which also behaves similarly as the above two) was moderate. But the procedure used here seems to be different by the addition of base instead of acid.
3rd-76	2.6	A, G, M	We used mepiquat D_3 as IS and it may have been inappropriate for this compound, we didn't have adequate IS. **Comment from the organisers:** Check the organisers' remarks on your results on matrine.

Nicotine Assigned value: 0.0868 mg/kg, CV*: 24.8 %

LabCode	z Score	Reason	Remarks/Details
69	-4.0 (FN)	E, L	Not possible to measure due to problems with analytical instrument, Information should have been given within the transcription process, human error in documentation and transcription
82	-4.0 (FN)	A, B	Poor sensitivity of applied analytical method **Comment from the organisers:** Your QC measures should be such that any poor sensitivity in the analysis of one or many target compounds (including a temporarily poor sensitivity) is recognised so that the reporting of FN results is avoided. Recovery experiments as well as calibration standards at the RL are helpfull measures. The use of an ILIS would have also helped to recognize a potential severe signal suppression. Using the very same procedure you have achieved good results for matrine and oxymatrine at <0.1 mg/kg levels. But for nicotine, which is similarly sensitive in LC-MS/MS, and which was present roughly at the same level as matrine and oxymatrine, you strangely indicated sensitivity problems. Please recheck your measurement settings.
3rd-70	2.1		**Comment from the organisers:** The use of an ILIS would have helped to reduce the bias
58	4.3	C	**Comment from the organisers:** Check the organiser's comments on your result on chlorate.
30	11.9	A	The analysis of these analytes in honey falls outside the scope of our laboratory, and the method employed has not been previously validated. **Comment from the organisers:** Using the PT as a possibility to implement and test new methodologies is promoted by the organisers. The use of an ILIS would have helped to reduce bias. Check the correctness of your standard solutions. Cross contaminations of the ubiquitous nicotine can occur but typically at much lower levels.

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

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

Phosphonic acid Assigned value: 0.202 mg/kg, CV*: 20.9 %

LabCode	z Score	Reason	Remarks/Details
45	-4.0 (FN)	F	Column equilibration, 1 injection in screening **Comment from the organisers:** See the organisers' remarks on your FN result for chlorate.
58	5.3	C	**Comment from the organisers:** Check the organisers' remarks on your result on chlorate.
66	5.5	E	**Comment from the organisers:** The use of standard addition to extract aliquots should normally have corrected for matrix effects. The use of an ILIS would have provided additional safety, by showing the exact retention time and peak shape of phosphonic acid. Check the correctness of the analytical standard.

Perchlorate Assigned value: 0.0550 mg/kg, CV*: 11.6 %

LabCode	z Score	Reason	Remarks/Details
11	-4.0 (FN)	L, M	Perchlorate was analyzed by the lab and the result was introduced on platform (0.0525 mg/kg). For some reason (communication problems ?) the result disappears. The main reason for this "poor performance" was lack of double check of results. If it was the case, the situation could be communicated to the organization, in time.
45	-4.0 (FN)	F	Column equilibration, 1 injection in screening **Comment from the organisers:** See the organisers' remarks on your FN result for chlorate.
64	-4.0 (FN)	L	internal result was 0.058 mg/kg
69	-4.0 (FN)	L	The measured value of perchlorate is 0.062 mg/kg, human error in documentation and transcription
82	3.2	G	We quantified perchlorate in honey sample using matrix-matched calibration standard. When we re-analyzed the sample employing standard addition approach we achieved result for perchlorate 0.053 mg/kg. This result would be complying. **Comment from the organisers:** It is difficult to understand this effect. Having used an ILIS in your original matrix-matched calibration, any matrix effect differences between the PT sample and your blank should have been taken into account. Background levels of the analyte in the blank can potentially be a source of bias in the case of ubiquitous compounds such as perchlorate, but such levels would lead to underestimated results in the sample.
66	4.1	A	**Comment from the organisers:** You have indicated the use of QuEChERS for this analysis, which was most likely an error. In any case, use of ILIS is strongly recommended.

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

Trinexapac (free acid)* Assigned value: 0.118 mg/kg, CV*: 14.7 %			
LabCode	z Score	Reason	Remarks/Details
3	-4.0 (FN)		
5	-4.0 (FN)	F	We have analyzed trinexapac ethyl
16	-4.0 (FN)	F	There was a mistake in our lab pesticide list. Lab analysed TRINEXAPAC-ETHYL not TRINEXAPAC (free acid)
26	-4.0 (FN)		
45	-4.0 (FN)	A	**Comment from the organisers:** See the organisers' remark on your FN result for chlorate.
57	-4.0 (FN)	F	wrong molecule in the method : trinexapac-ethyl instead of trinexapac
61	-4.0 (FN)		
69	-4.0 (FN)	L	Not in our Scope, so no results could have been transmitted, Information should have been given within the trancription process, human error in documentation and transcription
71	-4.0 (FN)	F	During the test scope selection by non-attention this compound was marked as tested. The entered scope for proficiency testing was not checked before result submission. The compound is not tested by the laboratory. The laboratory does not have an analytical standard of trinexapac (free acid). **Comment from the organisers:** Please note that the EUPT-SRMs do not have a scope selection period prior to the PT, unlike the EUPTs organised by the other three EURLs of the pesticides cluster (FV, CF, AO).
79	-4.0 (FN)	F	Due to a misunderstanding in the laboratory, the ""trinexapac (free acid)"" indicated on the target list was mistakenly interpreted as trinexapac-ethyl. The sample was analysed for trincexapac-ethyl, and not for Trinexapac (free acid). Therefore, trinexapac (free acid) could not be detected in the sample. Since trinexapac (free acid) is not part of the spectrum of our laboratory, no subsequent analysis can be carried out.
55	2.3	A, E, J	no experience for pesticide in matrix honey (pesticide not in accredited sope); poor analytical signal; analytical standard will be reassessed)
* Some laboratories have analysed for trinexapac-ethyl instead of trinexapac. The confusion is partly due to mislabelling of standards by some suppliers and a confusing entry in the EU pesticide database, which has been changed (see also Section 4.4).			

EURL EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

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 organised for 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 the official controls Regulation (EU) No. 2017/625[3]:

- EURL for Fruits and Vegetables (EURL-FV),
- EURL for Cereals and Feedingstuff (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 they can use to demonstrate their analytical performance and compare themselves with other participating laboratories.

[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

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EUPT-organisers and Scientific Committee

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

An **Organising Team** (in the following named organisers) is appointed by the EURL(s) in charge. This team is responsible for all administrative and technical matters concerning the organisation of the Proficiency Test (PT), e.g. the PT-announcement, the production of the PT-material (Test Item), the undertaking of homogeneity and stability tests, the packing and shipment of the PT-materials, the handling and evaluation of the results and method information submitted by the participants, the drafting of the preliminary and final reports as well as generation and distribution of EUPT-participation certificates.

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

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

a) An independent **Quality Control Group** (EUPT-QCG) and
b) An **Advisory Group** (EUPT-AG).

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

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 analytes to be present in the Test Item and the approximate concentrations at which they should be present.

The EUPT-SC typically meets once a year, after all EUPTs of the season have been conducted and preliminarily evaluated by the four pesticide EURLs. The aim of these meetings is to discuss the EUPT-results, especially where case-by-case decisions are needed. PT plans for the next EUPT season and, if needed, possible changes in the EUPT-General Protocol are also discussed during these meetings. The main topics and decisions on these meetings are documented.

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

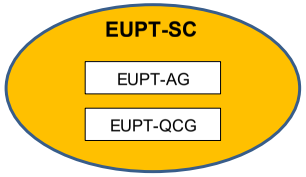

Figure 1: Composition of EUPT-Scientific Committee

The present EUPT General Protocol (EUPT-GP) was drafted by the EURLs and reviewed by the EUPT-SC. Follow the link to access a website giving an *overview of the GP-versions*.

EUPT Participants

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

- Art 38 (2) of Regulation (EU) No. 2017/625[3] Art. 28 (3) of Reg. (EC) No. 2005/396 (for all OfLs analysing for pesticide residues within the framework of official controls of food or feed[6])
- Art. 101 (1)(a) of Regulation (EU) No. 2017/625[3] (for all NRLs)

Every year, shortly before launching the registration period of the first of the four EUPTs in a given EUPT-Season, all OfLs and NRLs are asked to update their routine scope of commodities as well their contact information within the EURL-DataPool. Based on this information the OfLs are classified into those that are obliged and those that are eligible participate in each of the EUPTs to be conducted within a given year..

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

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

[6] Official controls in the sense of Regulation (EU) 2017/625. This includes labs involved in controls within the framework of national and/or EU programs, as well as labs involved in import controls according to Regulation (EU) 2019/1793 (which repealed Regulation (EC) No. 2009/669).

Labs that are obliged to participate in a given EUPT, and that are not able to participate, must provide the reasons for their non-participation This also applies to any participating laboratories that fail to report results.

OfLs not paying the EUPT sample delivery fee will be initially warned that their participation in subsequent EUPTs could be denied. In case of a repetitive non-payment, the EUPT organisers will inform the corresponding NRL to take action.

Confidentiality and Communication

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

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

As laid down in Regulation (EU) No. 2017/625[3], NRLs are responsible for evaluating and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.

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

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

The official language used in all EUPTs is English.

Announcement / Invitation Letter

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

Target Pesticide List

This list contains all analytes (pesticides and metabolites) to be sought for, along with the Minimum Required Reporting Levels (MRRLs) valid for the specific EUPT. The MRRLs are typically based upon the lowest MRLs found either in Regulation (EC) No. 2005/396 and Regulation (EU) No. 2016/128 (Baby Food Directive). Labs must express their results as stated in the Target Pesticides List.

Specific Protocol

For each EUPT the organising 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 analysis of two replicate analytical portions, taken from at least ten randomly chosen units of treated Test Item. Measurements should be conducted in random order. The homogeneity test data are statistically evaluated according to ISO 13528:2022, Annex B[7] or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC[8]. 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 analytes spiked at the same time, the overall distribution of the participants' results (CV^*), the analytical difficulties faced during the test, knowledge of the analytical behaviour of the compound in question), may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report. For certain analytes with comparable properties, an equivalent distribution within the sample can be expected if they were spiked/used at simultaneously. The homogeneity test, of one or more of these analytes, may thus be skipped or simplified. If, however, the distribution of participants' results for an analyte that was not or not fully tested for homogeneity, is found to be atypically broad, compared to the tested analytes, the EUPT-SC may decide that a homogeneity test should be performed a posteriori.

[7] ISO 13528:2022: 'Statistical methods for use in proficiency testing by interlaboratory comparisons", International Organization for Standardization.

[8] Thompson M., Ellison S.L.R., Wood R., "The International Harmonized Protocol for the proficiency testing of analytical chemistry laboratories" (IUPAC Technical Report). Pure Appl. Chem. 2006, 78, 145 – 196

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Stability of the analytes contained in the Test Item

The Test Items will also be tested for stability - according to ISO 13528:2022, Annex B[7]. 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 analytes 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 EUPT-Report, considering all relevant aspects such as the distribution of the participant's results (CV^*).

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

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

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

Stability during shipment: Considering knowledge about the expected susceptibility of analytes in the Test Item to possible losses, the organisers will choose the shipment conditions to be such that analyte losses are minimised (e.g. shipment of frozen samples, addition of dry ice). As shipment-duration can differ between labs/countries it is recommended that the organisers keep track of the shipment duration and then decide whether it is reasonable to conduct additional stability tests at conditions simulating shipment. Should critical losses be detected for certain analytes, the EUPT-SC will be informed (or the EUPT-QCG before or during the test). Case-by-case decisions may be

taken by the EUPT-SC considering all relevant aspects including the duration and conditions of the shipment to the laboratory as well as the feedback by the laboratory.

Methodologies to be used by the participants

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

General procedures for reporting results

Participating laboratories are responsible for reporting their own quantitative results to the organiser within the stipulated deadline. Any analyte 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 analytes detected should be expressed in 'mg/kg' unless indicated otherwise in the specific protocol. Laboratories should not report results below their reporting limits.

Correction of results for bias

According to the DG-SANTE Guidelines, the result of an analyte needs to be adjusted for method bias if the bias exceeds 20%. Unless a method is used that inherently accounts for method bias (see cases a-c below), laboratories are required to report the recovery (in percent), and whether their results were corrected mathematically using a recovery factor reflecting the reported recovery.

The EUPT-Panel will examine whether results, for which no correction for recovery was undertaken, should be omitted from the population used for calculating the assigned value.

When the laboratory uses any of the following approaches inherently accounting for method bias, this needs to be indicated in the appropriate fields within the Web-Tool. In such cases, reporting of the recovery rate is not mandatory.

a) use of stable isotope labelled analogues of the target analytes as Internal Standard (ILISs), added to the analytical portion at an early stage of the procedure

b) 'procedural calibration' approach

c) 'standard addition' approach with additions of analyte(s) to the analytical portions before extraction.

Methodology information

All laboratories are requested to provide information on the analytical method(s) they have used. The Web-Tool, which also serves for submitting analytical results, is typically used for collecting method information.

The collection of method information is considered very important by the EUPT-SC, as it facilitates the interpretation of results and the identification of analytical patterns associated with systematically biased results. A compilation of the methodology information submitted by all participants may be presented in an Annex of the Final EUPT-Report or in a separate report. Where the initial method information provided by the participating laboratories is not sufficient for evaluating methodology-related errors, or where additional information critical for results evaluation is needed, the EURLs and/or the EUPT-Panel may decide to conduct specific follow-up surveys among the concerned laboratories. If no sufficient information on the methodology used is provided, the organisers reserve the right not to accept the analytical results reported by the participants concerned or even refuse participation in the following PT.

Where necessary the methods are evaluated and discussed within the EUPT-SC, especially in those cases where the result distribution is not unimodal or very broad (e.g. $CV^* > 35\ \%$).

Results evaluation

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

– ***False Positive (FP) results***

These are results of analytes 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 analytes. In certain instances, case-by-case decisions by the EUPT-SC 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 (FN) results***

These are results for analytes 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 analytes 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-SC 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-SC may decide to take case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the RLs of the affected labs.

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

To minimise the influence of out-lying results on the statistical evaluation, the assigned value x_{pt} (= consensus concentration) will typically be estimated using the robust estimate of the participant's mean (x^*) as described in ISO 13528:2022[9], taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide including results submitted by laboratories not belonging to the EU-/EFTA-OfLs network or to even to only use the results of a subgroup of ('expert') laboratories that have previously repeatedly demonstrated good performance for the specific or similar compounds.

Furthermore, the EUPT-Panel may decide to eliminate certain results traceably associated with bias or gross errors for establishing the assigned value (see 'Omission or Exclusion of results' below).

Since the assigned values of the EUPT analytes are typically generated using robust mean concentrations of participant results (x_{pt}), which are generated by a variety of analytical standards and methods, the assigned values of EUPTs are typically metrologically not traceable.

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

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

[9] ISO 13528:2022 '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).

generated result, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Results may also be omitted e.g. if an inappropriate method has been used even if they are not outliers. All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the Final EUPT-Report. However, z scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.

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

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

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

The uncertainty of the assigned values $u(x_{pt})$ is calculated according to ISO 13528:2022 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-SC 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 13528:2022 concerning the uncertainty of the assigned value will be taken into account.

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

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

Based on experience from previous EUPTs[10], a percentage FFP-RSD of 25 % is currently used for all analyte-matrix combination, with the target standard deviation being calculated as follows:

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

[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. DOI:10.1021/jf104060h

The EUPT-SC reserves the right to also employ other FFP-RSDs or other approaches for setting the assigned value on a case-by-case basis, considering analytical difficulties and experience gained from previous proficiency tests.

For informative purposes the robust relative standard deviation (CV*) of the participants results is calculated according to ISO 13528:2022; Chapter 7.7 following Algorithm A in Annex C (so called "consensus approach").

– ***z scores***

This parameter is calculated using the following formula:

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

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

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

Following ISO 17043:2010[11], z scores will be classified as follows::

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

All false negatives will be assigned a z score of -4. These z scores will typically appear in the z score histograms and will be used in the calculation of combined z scores.

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

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

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

– ***Category classification***

The EUPT-SC will decide if and how to classify the laboratories into categories based on their scope and/or performance. Currently, a scope-based classification into Category A and Category B is employed. Laboratories that a) are able to analyse at least 90% of the compulsory analytes in the target pesticides list, b) have correctly detected and quantified a sufficiently high percentage of the analytes present in the Test Item (at least 90 %) and c) reported no false positives, will have demonstrated 'sufficient scope' and will be therefore classified into Category A. For the 90% criterion, the number of analytes needed to be correctly analysed to have sufficient scope will be calculated by multiplying the number of compulsory analytes 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 analytes from the Target Pesticides List needed to be targeted or analytes present in the Test Item that need to be correctly detected and quantified to have sufficient scope.

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

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

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

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z score (AZ^2)[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 AZ^2, z scores > 5 will be set as 5. Based on the AZ^2 achieved, the laboratories are classified as follows:

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

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

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

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

Publication of results

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

[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.

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The Final EUPT-Report will be published after the EUPT-SC has discussed the results. Taking into account that the EUPT-SC meets normally only once a year (typically in late summer or autumn) to discuss the results of all EUPTs organised by the EURLs earlier in the year, the Final EUPT-Report may be published up to 12 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 EUPT organiser will deliver a Certificate of Participation to each participating laboratory showing the z scores achieved for each individual analyte, the classification into Categories, and if deemed necessary also combined z scores. The certificates of participation will be uploaded onto the EURL-DataPool where they can be accessed by the concerned laboratories only.

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-SC will decide whether a corrigendum will be issued and how this should look like. The online version of the Final EUPT report will be replaced by the new one and all affected labs will be contacted.

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

Follow-up activities

Laboratories are expected to undertake follow-up activities to trace back the sources of erroneous or strongly deviating results (typically those with $|z| > 2.0$) - including all false positives. In exceptional cases, follow-up activities may even be indicated for results within $|z| \leq 2.0$ (e.g. if two errors with opposed tendency cancel each other leading to acceptable results).

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

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

NRLs will be considered as **underperforming in relation to scope** if in at least two of the last four EUPTs falling within their responsibility area they: a) haven't participated, or b) targeted less than 90% of the compulsory analytes in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the test items (80% for SRM-compounds). Additionally, NRLs that obtained AZ^2 higher than 3 (AAZ higher than 1.3 for SRM-compounds) in two consecutive EUPTs of the last four EUPTs, will be considered as **underperforming in accuracy**. As soon as underperformance of an NRL is detected, a two-step protocol established by DG-SANTE will be applied[14]:

Phase 1:

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

Phase 2:

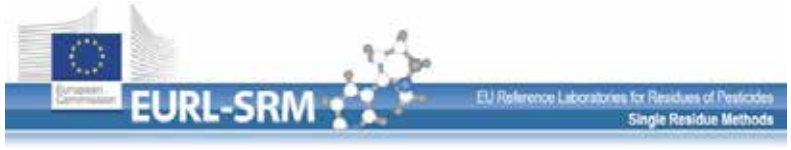
- If the results still reveal underperformance the Commission shall be informed officially by the EURL including a report of the main findings and corrective actions.
- The Commission shall inform the Competent Authority and require that appropriate actions are taken.

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

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

Disclaimer

The EUPT-SC 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 18th EU Proficiency Test
on Pesticides requiring Single Residue Methods
EUPT – SRM18 (2023)
(update on 12 May 2023)

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. 10**" covering all EUPTs in 2023.
This PT is organized by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM). The EURL-SRM is accredited as provider of proficiency tests according to ISO 17043 (please see **EURL-SRM accreditation**).

The EUPT-SRM18 deals with the analysis of SRM-pesticides in honey. Participation is obligatory for all National Reference Laboratories for Single Residue Methods (NRL-SRMs), as well as for all official EU laboratories (OfLs) involved in the official analyses of pesticide residues in honey. The tentative classification of labs into "obliged" and "not obliged" to participate in this PT was based on information on the scope of commodities covered by each laboratory, as stated within the EURL DataPool. Prior to the classification, the laboratories were asked to update this information within the DataPool and the responsible NRLs were asked to verify this information.

The registration of the labs to the PT was run through the EUPT-Registration website, which is connected to the EURL-DataPool. All laboratories classified as obliged were notified that they should enter the online registration platform irrespective if they intend to participate or not. In the latter case, the labs had to state their reasons for non-participation. The reasons for non-participation received from obliged laboratories during registration, especially details considering the scope, will be considered in the final list of obliged laboratories.

The most important documents related to this PT can be accessed via the **EUPT-SRM18-Website**.

Test Item

The Test Item of this EUPT is "Honey".

Participants will receive one Test Item bottle containing ~150 g **honey at ambient temperature with incurred and spiked analytes** from the **Target Pesticides List**. The honey was spiked with selected compounds, homogenized well through intensive steering and filled into plastic bottles. Shipment will be done in non-cooled boxes. NO Blank material will be sent to the participants for this PT.

Using randomly chosen bottles, the Organizers will check the Test Item for sufficient homogeneity and for the stability of the analytes contained over the period of the exercise.

Target Analytes and MRRLs

The Test Item will contain several analytes from the mandatory and optional section of the **EUPT-SRM18 Target Pesticide List** (TPL). Laboratories should read the TPL carefully as it shows how the residues should 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. **Make sure to download the latest version of the EUPT-SRM18 Target Pesticides List before starting with analysis and reporting results**.

Shipment of Test Item

Dispatch of the Test Item is planned on 19 May 2023.
Test Item will be shipped via DHL Express from Germany to the participants in a cushion-packed box at ambient temperature. Prior to shipment, a reminder will be sent to the participating laboratories by e-mail.
The participating laboratories must make their own arrangements for the receipt of the package. They should inform the Organizers of any public holidays in their country/city during the week of the shipment, and must make the necessary arrangements to receive the shipment, even if the laboratory is closed.

Where complications during customs clearance or shipment are expected, the participants should provide the Organizer in advance (by 10 May) with **detailed contact Information** (e.g. mobile phone numbers of laboratory personnel) and all **necessary documents** to be stuck/attached on the package to ensure smooth customs clearance. Such documents may be e.g. a permission for importing organic material for scientific purposes (lab analysis) or an **instruction in local language** explaining the need to keep the package in a refrigerator in case of delay during shipment or clearance at customs.

After the shipment is tracked within the DHL delivery system and the waybill is printed out, the main contact person for the PT will be informed by DHL on the tracking number of his package. The participants can track their own package online and must make any necessary arrangements to receive the delivery.

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

Instructions on handling the Test Item

Once arrived, the Test Item should be stored in a refrigerator (2 – 8 °C) until analysis in order to avoid any possible deterioration/spoilage of the sample material and to minimize analyte losses.
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-SRM18 result submission Webtool**.

The homogeneity tests will be conducted using 5 g portions. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can be guaranteed only for sample portions roughly 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-SRM18 Result Submission Webtool:**

- **Sample receipt acknowledgement: From 22 May, 2023 onwards.**
- **Reporting of Analytical results and method information: 22 May – 16 June 11 am (11 h) CEST.**
- **Deadline for result submission is 16 June, 11 am (11 h) (CEST), 2023.**
- **Reporting of additional information on methods used for tentatively false negative results: 17 – 22 June, 2023.**

A guideline for the new **EUPT-SRM18 result submission Webtool** will be provided to the participants in due time and a link to it can also be found in the info-box on the Webtool. The **participants are urged to read it carefully before submitting their results**.

- Login credentials and lab code

To access the EUPT-SRM18 Result Submission Webtool, participants must use their PERSONAL LOGIN CREDENTIALS (username and password). The personal username[1] is linked to the email and will be provided to the PT-contact persons by DTU prior to the opening of the Webtool for the EUPT-SRM18.
The password can be retrieved via the link: https://guest.dtu.dk/Sites/GuestLogin/RetrievePassword.aspx
Every PT participant can change his/her password using the link: **https://guest.dtu.dk/Sites/GuestLogin/Default.aspx**
For security reasons please update your password once a year.

The lab's unique lab code for the EUPT-SRM18 will be provided to the participants following the first access to EUPT-SRM18 Result Submission Webtool.

- Acknowledgement of package receipt and acceptance of PT-materials

Once the laboratory has received the package with the PT material, it must report to the organizer via the **EUPT-SRM18 Result Submission Webtool** the date of receipt, the condition of the package, the condition of the test material at arrival, whether the material is accepted or not, and any other comments concerning the test material. This task should be finalized by **26 May 11:00 am CEST**. If a laboratory does not respond by this deadline, the Organizers will assume that the Test Item has been received and accepted. **In case of problems with the sample receipt or condition**, please contact the organizers **additionally via e-mail ASAP** (**eurl-srm@cvuas.bwl.de**) to ensure that corrective actions are taken as early as possible. Please note that completing and saving the sample receipt form is a pre-requisite for accessing the results submission areas. However, you can still access sample receipt form and edit it later.

Participants are encouraged to follow the whereabouts of their parcels using the tracking code of the shipping company, that they will receive via e-mail, and to intervene at the shipping company, the customs or the organizers if they notice any delays. Any participants not having received the Test Item by the afternoon of Thu. 25 May must inform the Organizer via e-mail (EURL-SRM@cvuas.bwl.de) immediately but not later than Fri. 26 May 11:00 am CET. The Organizer will consult the shipping company to localize the package and decide on further actions, including new shipment, if necessary.

[1] Correction in the Version 2 from 12 May, 2023: not login credentials, but login username.

- Reporting qualitative and quantitative results

To report their results, laboratories must access the **EUPT-SRM18 Result Submission Webtool**.
All results must be reported on this website by 16 June, 11 am (CEST), 2023. The pages for the "scope, detected and results" will not be accessible after this deadline, and no results submitted afterwards will be accepted.

Before entering the results, please study the **EUPT-SRM18 Target Pesticides List** carefully. Please note, that the residue definitions applying to the EUPT may appear in a shortened form on the result submission website.

If a lab routinely subcontracts analyses of one or more compounds to another lab, this subcontracted lab may (and is even encouraged to) to get involved in the EUPT exercise by analysing the relevant analytes in the test material.

IMPORTANT:
The participants are obliged to inform the organizers of all cases where PT results were generated by subcontracted labs, and to provide details on the subcontracting laboratory. This also applies where the subcontracted lab belongs to the same institution/company but runs its own quality control system.

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.
  If a pesticide was not detected or **if it was detected but the quantitative result is below the RL (Reporting Limit) of the laboratory or the MRRL, no result should be reported.**

  The residue levels of the pesticides must be reported in mg/kg using **three significant figures**:
  e.g. 0.0582; 0.156, 1.64, 20.3 mg/kg.

  Where a target analyte on the target pesticide list is defined as the sum of two or more components, a result for this "summed target analyte" should only be reported if
  a) the method used covers the entire residue definition of this "summed target analyte", e.g. if the method involves a chemical conversion to one component, or
  b) if all individual components entailed in this residue definition were targeted.
  In the latter case, the concentrations of the individual components of the "summed target analyte" should be added-up and expressed as stated in the residue definition on the target pesticide list. If at least one of the components within the "summed target analyte" was not analysed, this "summed target analyte" should be marked as "not analysed". In case one of the components within the complex residue definition was targeted but not encountered at a quantifiable level (<RL), its concentration should be considered equal to zero when calculating the summed result.

  Recovery (Bias)-corrected results should be reported only if this reflects the lab's actual or envisaged routine procedure. Where a **result was corrected for recovery,** the approach(es) applied 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.

- **Reporting Limit (RL) in mg/kg:** the lab's reporting limit for an analyte.
  Where two or more components **of a complex residue definition** are analyzed individually, the **RL of the sum** is also formally required. It should be calculated by summing up the individual RLs of the constituent components expressed as prescribed by the residue definition (applying conversion factors based on the molecular weight of the components). The individual RLs of each component (without conversion) can be reported in the respective

fields of the individual components or, if these are not available, in the "Comments" field of the analyte with the complex residue definition. Where the analytical method for the analysis of a complex residue definition involves a chemical transformation, thus generating a single analytical result, the RL of the method is to be reported, but again expressed as prescribed by the residue definition.

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

- Reporting Information on Analytical Methodology

On the page of "**Edit methods"** of **EUPT-SRM18 Result Submission Webtool** the participating laboratories have to provide information on the analytical method(s) applied for the analysis of the target analytes detected in the Test Item.

The participating laboratories are urged to thoroughly fill-in all requested information.

IMPORTANT:
If entries in required fields within the Result Submission Webtool are missing, you will not be able to proceed with the final submission. Therefore, please fill-in your method information in due time to be on the safe side.

For detailed information on how to fill-in the columns on the "Edit methods" page, please refer to the Guideline for Results Submission that will be distributed to all participants in due time. A link to this guideline can also be found in the info-box on the Result Submission Webtool.

IMPORTANT:
Please also read the **mouse-over messages** popping-up when your mouse cursor meets a field name in the table header for a few seconds.

- Submission of results

Once you have entered all your results and checked their correctness, you have to submit them by clicking "Final Submission" button before the submission deadline. The "Final submission" button can be found at the bottom of each page. To avoid accidents, a confirmation is requested after clicking the "Final Submission" button.

IMPORTANT:
Following "Final Submission", 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

After the results submission deadline, **if a laboratory has obtained a tentatively false negative result**, it will be asked to enter the method information for this analyte, within 4 working days.

Establishment of assigned values

In addition to OfLs from EU Member States, EFTA countries, a limited number of laboratories from EU candidate countrie and third countries are allowed to take part in this exercise. For the establishment of the assigned values, typically onl results submitted by OfLs from EU and EFTA countries are taken into account.

Subcontracting /

The following tasks were conducted by the EURL-CF, Lyngby, Denmark:

a) Generation of login credentials
b) Programming and administration of EUPT-SRM18 result submission website

Follow-up actions

After the distribution of the EUPT-SRM18 Preliminary Report, laboratories having submitted poor results (high absolute z scores, false negatives or false positives) will be asked to investigate the reason behind the poor performance, and to repor their insights and possible corrective actions to the Organizer. This information will be forwarded to the corresponding NRL SRMs upon request. All EUPT-SRM18-participants are welcome to ask the EURL-SRM for technical assistance.

In the course of results evaluation, the organizer may ask laboratories to provide additional methodology information rel evant to the evaluation and interpretation of the PT.

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

Documents

All documents related to the EUPT–SRM18 can be downloaded from **EUPT-SRM18 Website** or **EURL-Document Repositor (CIRCA-BC).**
For any questions, please contact the organizers **EURL-SRM@cvuas.bwl.de**

IMPORTANT:
Please check the **EUPT-SRM18 Website** before starting with the analysis in order to **make sure, that you have the lates 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: 225 €
- Labs based in third countries: 375 €

An invoice in PDF format, issued to the "invoice address" stated in the registration form, will be sent after the results submission deadline to the "invoice e-mail address" stated in the registration form and to the PT main contact person. If the payment is being taken care of by another department/institution, the recipient of the invoice is requested to timely forward the invoice to the responsible persons. Details on payment are given in the invoices.

Every lab that has registered for participation in the EUPT-SRM18 and received the test material in good condition has to pay the total fee, irrespective of whether results are submitted or not. This also includes cases where a lab realizes within the course of the PT, that none of the compounds it has targeted is present at a quantifiable level in the PT-material, or if it realizes that for whatever reasons it cannot conduct any analyses or it cannot submit any results.

Additional cost may occur if extra services are requested in relation to the payment, such as the completion of additional paperwork and the generation of a new modified invoice in order to include information that was missing or incorrectly provided during registration.

IMPORTANT:

The EURL-SRM will issue digital invoices in PDF format only and without any electronic signature.
If, due to locally applying legal requirements, a participating laboratory needs an electronic invoice (e.g. certificed or signed e-invoice in XML), it has to provide the PT-Organizers a suitable and free tool to generate the necessary e-invoice and provide full assistance in case this tool requires the use of a language other than English. Otherwise, the PT-Organizer will not issue an e-invoice. Depending on the incurring extra workload, the participating laboratory may be charged for this extra service.

The EURL-SRM will not complete any special form required by the participating laboratories for their financial department or payment office and does not agree to give any personal or private data for this purpose. If completion of such forms is prerequisite for payment, the participants are requested to fill-in the forms themselves,
based on data in the financial identification note (https://www.eurl-pesticides.eu/library/docs/srm/SRM-Bank_m_Financial_Identification.pdf) and to send us the filled form. After verification, and if necessary correction, we will return it with signature and stamp.

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 the participant and the Organizers.

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

If no payment or no proof of payment is received and no explanation is given to the Organizers, the Organizers reserve the right not to issue the participation certificate for the concerned laboratory, to exclude its results and its name from the Final EUPT-Report, and 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 (This number MUST be indicated in the payment!)***
VAT of CVUA Stuttgart	**DE 811 600 510**

Please note:

- **Please do not make any remittance before you receive the invoice with the Payee Identification Text.**
- **EURL-AO (@ CVUA Freiburg) and EURL-SRM (@ CVUA Stuttgart) belong to the same Ministry and have thus the same bank account.**
  If your laboratory is participating in both PTs (EUPT-SRM18 and EUPT-AO18), please ask your financial department to transfer the fee for each of the PTs separately using the corresponding payee identification text (= invoice number) given in each invoice. Without this text, your payment will not be able to reach the correct EURL.

Calendar of EUPT-SRM18

(please see https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EUPT-SRM18_Calendar.pdf)

Target Pesticides List of EUPT-SRM18

(please see https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EUPT-SRM18_TargetPesticideList.xlsx)

Contact information

EU Reference Laboratory for Single Residue Methods (EURL-SRM)
Chemisches und Veterinäruntersuchunsamt Stuttgart
Schaflandstr. 3/2
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e-mail: EURL-SRM@cvuas.bwl.de

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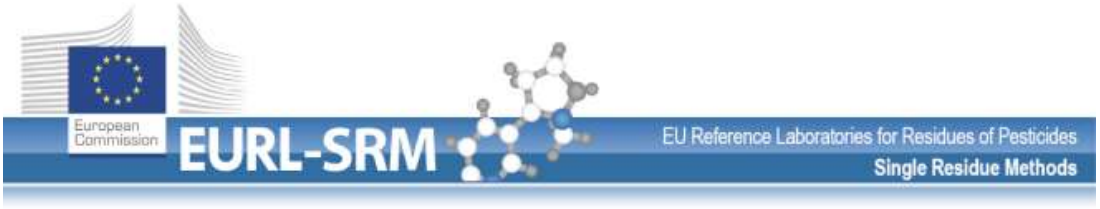
Radim Štěpán	Czech Agriculture and Food Inspection Authority, Prague, Czech Republic
Hermann Unterluggauer	AGES; Institute for Food Safety Innsbruck, Austria.

Appendix 10 Calendar and Target Pesticides List of EUPT-SRM18



CALENDAR for the EUPT – SRM18

Matrix: Honey

(released on 19/02/2023)

Activity	Dates
Announcement of the EUPT-SRM18 opening of the **EUPT-SRM18 Website** with links to all relevant documents	19 February 2023
Registration Period for EUPT-SRM18 via "**EUPT-Registration Website**" **Labs classified as "OBLIGED" to participate in the EUPT-SRM18 MUST enter the EUPT-Registration Website and either register OR give explanations for non-participation**	17 March – 6 April 2023, 23:30 h CEST*
Dispatch of EUPT-SRM18-Specific Protocol	by 5 May 2023
Shipment of EUPT-SRM18 Test Item	19 May 2023
Confirming Sample Receipt and Acceptance via "**EUPT-SRM18 Result Submission Webtool**"	From 22 May 2023 onwards
Submission of Results (Pesticide scope, Results, Method Info) via "**EUPT-SRM18 Result Submission Webtool**"	22 May – 16 June 11 h (11 a.m.) CEST
Submission of Additional/Missing Information e.g. Method info on tentatively false negative results via "**EUPT-SRM18 Result Submission Webtool**"	19 – 22 June 2023
Dispatch of Preliminary Report containing results as well as preliminary assigned values and z-scores only	Within 3 weeks after the submission deadline
Collection of reasons for underperformance and missing information on methods	July & August 2023
Dispatch of Final Report	Dec. 2023

*Please make sure to register for the EUPT from 17 March to the deadline 06 April 2023 via "**EUPT-Registration Website**". Any wish for registration after this deadline or not using the registration website cannot be considered.

REMARK:
Please note that the dates given above may be subject to minor changes**.** In case of changes importantly affecting the participants, the participants will be informed via e-mail. However, 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

EURL-SRM — EU Reference Laboratories for Residues of Pesticides — Single Residue Methods

Target Pesticides List

for the EUPT–SRM18 (2023), Honey
(updated on 08.05.2023)

Analytes are grouped into Mandatory, Optional and Extra
For alphabetical sorting in the same order as in the Webtool: See "Ordered from A to Z"

a new category "Extra (E) is introduced:
Extra analyte, not for lab evaluation (07.05.2023)

Only mandatory (=compulsory) analytes will be considered in the scope-based classification, optional (=voluntary) and extra analytes not.
Please also refer to the EUPT General Protocol.

M: Mandatory O: Optional E: Extra	Analytes	Notes	Listed in	MRRL [mg/kg]	
M	**2,4-D (free acid)**	No hydrolysis required	WD-Honey and MACP-Reg.	**0.01**	
M	**BAC-C12 chloride**	Benzyldimethyldodecylammonium-chloride	WD-Honey	**0.01**	
M	**BAC-C14 chloride**	Benzyldimethyltetradecylammonium-chloride	WD-Honey	**0.01**	
M	**Chlorate (anion)**		WD-Honey	**0.01**	
M	**Chlormequat chloride**	Expressed as chloride salt!	MACP-Reg.	**0.01**	
M	**DDAC-C10 chloride**	Didecyldimethylammonium-chloride	WD-Honey	**0.01**	
~~M~~	~~**DMF**~~	~~N-(2,4-Dimethyl-phenyl)-formamide (Amitraz-metabolite)~~	~~WD-Honey~~	~~**0.01**~~	removed from the TPL (05.04.2023)
~~M~~	~~**DMPF**~~	~~N-2,4-Dimethylphenyl-N'-methylformamidine (Amitraz-metabolite)~~	~~WD-Honey~~	~~**0.01**~~	removed from the TPL (05.04.2023)
M	**Fluazifop (free acid, sum of isomers)**	No hydrolysis required	WD-Honey and MACP-Reg.	**0.01**	
M	**Fosetyl**		MACP-Reg.	**0.01**	
M	**Glyphosate**		WD-Honey and MACP-Reg.	**0.01**	
M	**Haloxyfop (free acid, sum of isomers)**	No hydrolysis required	MACP-Reg.	**0.01**	
M	**Matrine**		WD-Honey	**0.01**	
M	**Mepiquat chloride**	Expressed as chloride salt!	MACP-Reg.	**0.01**	
M	**Nicotine**		MACP-Reg. (2024 onwards)	**0.01**	
M	**Oxymatrine**		WD-Honey	**0.01**	
M	**Phosphonic acid**		MACP-Reg.	**0.03**	
O	**AMPA**		WD-Annex II (future RD)	**0.01**	
O	**Chloridazon desphenyl**		WD-Honey	**0.02**	New! (05.04.2023)
O	**MCPA (free acid)**	No hydrolysis required	WD-Annex III	**0.01**	
O	**N-Acetyl-glyphosate**		WD-Annex II (future RD)	**0.01**	
O	**Perchlorate**		Contaminant	**0.01**	
O	**Quizalofop (free acid, sum of isomers)**	No hydrolysis required	WD-Annex III	**0.01**	
O	**Trinexapac (free acid)**	No hydrolysis required	WD (4.1 and Annex II)	**0.01**	
E	**Bromide (anion)**	Analysis of natural content for information only (no z-score calculations)	MACP-Reg.	**none**	Changed to category "Extra" (07.05.2023)
E	**Copper**	Analysis of natural content for information only (no z-score calculations)	MACP-Reg.	**none**	New on the TPL in the category "Extra"! (08.05.2023)

MACP-Reg.: Multiannual Control Program Regulation. Note that honey is not among the matrices of the MACP.
Link: REGULATION (EU) 2022/741 of 13 May 2022; https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32022R0741&qid=1665421580611&from=EN.
WD: Working document on pesticides to be considered for inclusion in the national control programs to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 21-22 November 2022 rev. 14(5); https://www.eurl-pesticides.eu/userfiles/file/WD/SANCO_12745_2013_rev_14_5.pdf

EURL-SRM — EU Reference Laboratories for Residues of Pesticides — Single Residue Methods

Target Pesticides List

for the EUPT–SRM18 (2023), Honey
(updated on 08.05.2023)

Analytes are sorted alphabetically in the same order as in the Webtool.
For grouping into Mandatory and Optional (incl. Extra): See sheet "Grouped into Mandatory-Optional"

a new category "Extra (E) is introduced:
Extra analyte, not for lab evaluation (07.05.2023)

Only mandatory (=compulsory) analytes will be considered in the scope-based classification, optional (=voluntary) and extra analytes not.
Please also refer to the EUPT General Protocol.

M: Mandatory O: Optional E: Extra	Analytes	Notes	Listed in	MRRL [mg/kg]	
M	2,4-D (free acid)	No hydrolysis required	WD-Honey and MACP-Reg.	0.01	
O	AMPA		WD-Annex II (future RD)	0.01	
M	BAC-C12 chloride	Benzyldimethyldodecylammonium-chloride	WD-Honey	0.01	
M	BAC-C14 chloride	Benzyldimethyltetradecylammonium-chloride	WD-Honey	0.01	
E	Bromide (anion)	Analysis of natural content for information only (no z-score calculations)	MACP-Reg.	none	Changed to category "Extra" (07.05.2023)
M	Chlorate (anion)		WD-Honey	0.01	
O	Chloridazon desphenyl		WD-Honey	0.02	New! (05.04.2023)
M	Chlormequat chloride	Expressed as chloride salt!	MACP-Reg.	0.01	
E	Copper	Analysis of natural content for information only (no z-score calculations)	MACP-Reg.	none	New on the TPL in the category "Extra"! (08.05.2023)
M	DDAC-C10 chloride	Didecyldimethylammonium-chloride	WD-Honey	0.01	
~~M~~	~~DMF~~	~~N-(2,4-Dimethyl-phenyl)-formamide (Amitraz-metabolite)~~	~~WD-Honey~~	~~0.01~~	removed from the TPL (05.04.2023)
~~M~~	~~DMPF~~	~~N-2,4-Dimethylphenyl-N'-methylformamidine (Amitraz-metabolite)~~	~~WD-Honey~~	~~0.01~~	removed from the TPL (05.04.2023)
M	Fluazifop (free acid, sum of isomers)	No hydrolysis required	WD-Honey and MACP-Reg.	0.01	
M	Fosetyl		MACP-Reg.	0.01	
M	Glyphosate		WD-Honey and MACP-Reg.	0.01	
M	Haloxyfop (free acid, sum of isomers)	No hydrolysis required	MACP-Reg.	0.01	
M	Matrine		WD-Honey	0.01	
O	MCPA (free acid)	No hydrolysis required	WD-Annex III	0.01	
M	Mepiquat chloride	Expressed as chloride salt!	MACP-Reg.	0.01	
O	N-Acetyl-glyphosate		WD-Annex II (future RD)	0.01	
M	Nicotine		MACP-Reg. (2024 onwards)	0.01	
M	Oxymatrine		WD-Honey	0.01	
O	Perchlorate		Contaminant	0.01	
M	Phosphonic acid		MACP-Reg.	0.02	
O	Quizalofop (free acid, sum of isomers)	No hydrolysis required	WD-Annex III	0.01	
O	Trinexapac (free acid)	No hydrolysis required	WD (4.1 and Annex II)	0.01	

MACP-Reg.: Multiannual Control Program Regulation. Note that honey is not among the matrices of the MACP.
Link: REGULATION (EU) 2022/741 of 13 May 2022; https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32022R0741&qid=1665421580611&from=EN.
WD: Working document on pesticides to be considered for inclusion in the national control programs to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 21-22 November 2022 rev. 14(5); https://www.eurl-pesticides.eu/userfiles/file/WD/SANCO_12745_2013_rev_14_5.pdf

Appendix 11 Call for Registration for the EUPT-SRM18

Von: Schreiter, Pat (CVUA-S) im Auftrag von EURL-SRM Pesticides (CVUA-S)
An: EURL-SRM Pesticides (CVUA-S)
Betreff: Call for Registration (EUPT-SRM18, Honey) (EU/EFTA_OfLs)
Datum: Donnerstag, 16. März 2023 23:55:24

Dear Colleagues from EU- and EFTA-OfLs,

The EUPT-SRM18 using honey as matrix is now open for registration and your laboratory is welcome to register for the EUPT-SRM18 **by 06 April, 2023, 11.30 p.m.**
Please read this email carefully, before you register.

Based on the data stored in the OfL-Network Database concerning commodity scope and lab status (e.g. OfL, NRL) and verified by the NRLs, all OfLs labs were tentatively classified as "obliged" or "non-obliged" to participate in a certain EUPT on pesticides residues. The classification of your lab for each PT is displayed on the EUPT-Registration page after logging in.

- **NRLs-SRM and OfLs analyzing honey were classified as obliged to participate in the EUPT-SRM18.**
- **All obliged laboratories have to enter the registration form**.
- **Obliged labs not intending to participate** in the EUPT-SRM18 still have to access the registration page and choose "No" under "I want to REGISTER my lab for this EUPT" and **provide an explanation for non-participation** (requirement by DG-SANTE).
- If your lab is classified as **non-obliged** and you are not going to participate in this PT, you don't have to do anything.
- If you think that the EUPT-SRM18 classification of your laboratory is erroneous, please contact your NRL and the eurl-srm@cvuas.bwl.de.
- **Please check first, if your laboratory is able to analyse at least one of the compounds on the Target Pestidies List,** before you register for participation in the EUPT-SRM18. Every lab that has registered for participation in the EUPT-SRM18 and received the test material has to pay the total fee, even though it recognized later that it is not able to analyse any one of the compounds in this PT.

During your registration:

1. The **lab name for shipment** is limited to **40** letters.
   You can use a short acronym and write it in your local language phonetics, but only LATIN letters can be accepted (please do not use language-specific letters, e.g. Š, Å, í … Greek or Cyrillic, as those are not accepted to prepare the waybills).
2. **The "City" in the sample delivery address MUST be written in English (e.g. "Rome", "Prague" instead of "Roma" or "Praha")**!
3. Please **check carefully the sample delivery address in the registration confirmation** sent to you upon your registration. For any error please correct it on the registration form by the registration deadline. During the registration period you and any member of your laboratory can change any of your laboratory's entries on 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 with the changed data.

For further information (e.g. Target Pesticides List, Calendar, Participation Fee…) on the EUPT-SRM18 please visit the EUPT-SRM18 Website.
The Specific Protocol for the EUPT-SRM18 will be dispatched two weeks prior to the shipment.

If you have any questions, please contact us EURL-SRM@cvuas.bwl.de

Best regards,
The EURL-SRM Team

Guide to EUPT-SRM18 Results Submission Webtool

Version: 2023-01, Date: 12-05-2023, Init: Schr

In order to get familiar with the Webtool, please read this guideline carefully before you start entering your data.

General Information:

- **Please only use GOOGLE CHROME or FIREFOX Web-browsers under INCOGNITO mode.**
  The latest version of these browser softwares is recommended.
- **Your data is automatically saved as soon as you move the cursor from one edited field to another.** Therefore, almost all pages and tables do not have any save button.
- You can access the Webtool as often as you need during the results submission period. However, **before deadline you must submit your results and method information by clicking "*Final Submission*". Otherwise, your result will not be included in the evaluation!**
- **After Final Submission you will NOT be able to change your results anymore!**

Getting started 2
Proficiency Test Overview 3
Sample receipt and acceptance 4
Scope 6
Detected 6
Edit results 7
Edit methods 8
Final Submission 10
Additional Information 12

Guide to EUPT-SRM Results Submission Webtool

Getting started

Link to Webtool: www.eurl.dtu.dk

Choose "**DTU Employees Students and Guests**"

PT-contact persons can **login** to the Webtool using their personal login credentials, which are valid for all EUPTs-CF, -FV, -AO, -SRM, -MN and –PC and linked to your email address.

As a reminder, PT-contact persons will receive an e-mail sent by DTU with their **personal username** shortly prior to the opening of the EUPT-SRM18 Webtool.

If you have forgotten your password you may **retrieve** it using this link: https://guest.dtu.dk/Sites/GuestLogin/RetrievePassword.aspx.

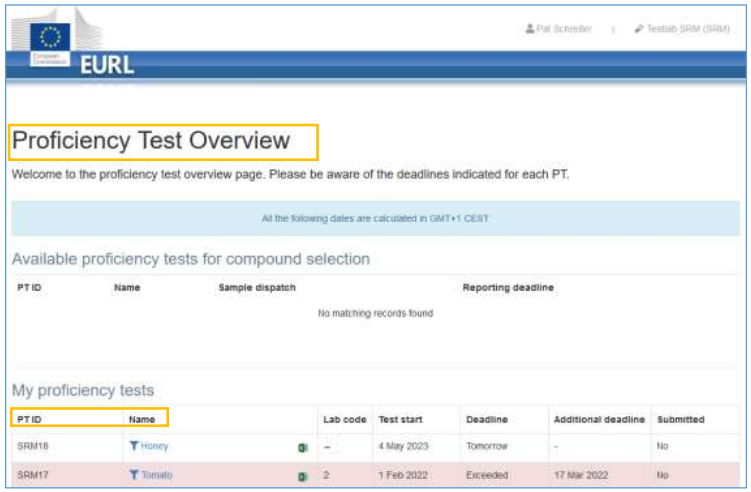
You can **change your password** using this link: https://guest.dtu.dk/Sites/GuestLogin/Default.aspx

In order to increase security we recommend changing your password once a year.

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

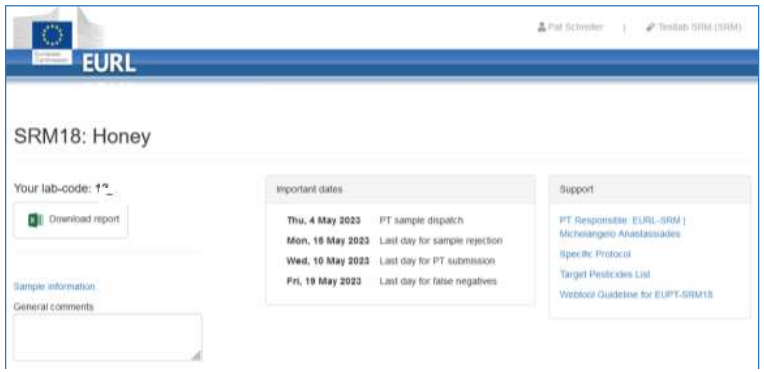
Proficiency Test Overview

On the **page *"Proficiency Test Overview"***, you will see on the top the section "*Available proficiency tests for compound selection*" with the PTs that are available for compound selection and below the section "*My proficiency tests*" showing information on the currently active EUPT(s), as well as on EUPTs in which your lab has participated in the past. The lab code for a new PT is automatically generated when the Webtool of the concerned PT is open and you log into it for the first time.



By clicking on "**SRM18 | Honey**" under "*My proficiency tests*", you will see the **current scope for this EUPT** in alphabetic order.

NO NEED TO INDICATE YOUR ANALYTE SCOPE IN ADVANCE!
In contrast to other EUPTs organized by EURL-CF, -FV and -AO**, in the case of EUPT-SRMs you don't need to select the analyte scope before sample shipment. You will be able to edit your targeted compounds from opening of the Webtool till the deadline for result submission.**

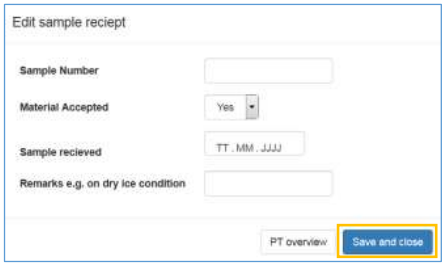


Sample receipt and acceptance

The **Webtool for the EUPT-SRM18 result submission** will expectedly open on **22 May, 2023**.

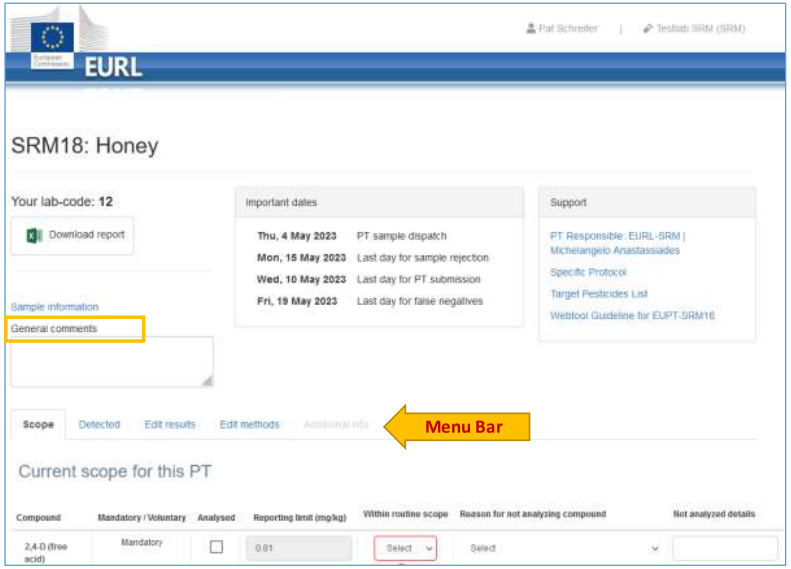
Once you have received the parcel with the PT-materials, please click on EUPT-SRM18 under "*My proficiency tests*" to open the pop-up window "**Edit sample Receipt"**. Please fill-in the information requested within this pop-up-window:

- **Sample Number**: Please enter the bottle number of the Test Item you received.
- **Material Accepted**: Based on condition upon receipt please indicate "Yes" or "No".
  If the PT-materials are not accepted, please additionally contact the PT-Organizers via E-mail.
- **Sample received:** Please enter the date when the parcel arrived at your institution.
- **Remarks e.g. on dry ice condition:** Please enter here any remarks concerning the condition of the parcels and sample bottles, such as leakages, temperature, state of material, if dry ice is still present in the parcel (not applicable for EUPT-SRM18), etc.

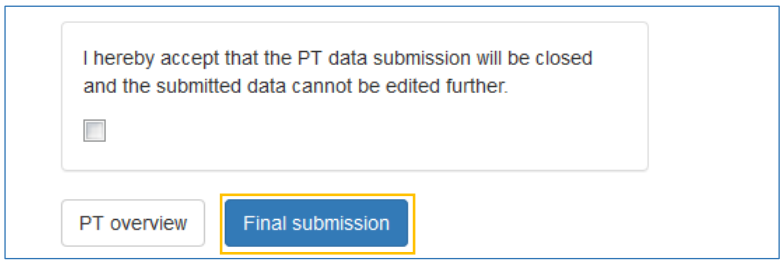


Completing the "*Edit Sample Receipt*" window and accepting the sample is a **precondition for being able to continue the submission page.** This should be done **ideally shortly after parcel receipt**. You can, however, access and edit all the entries on "*Edit sample receipt*" throughout the PT-period under "*Sample information*" (please see next page, left navigation bar).

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



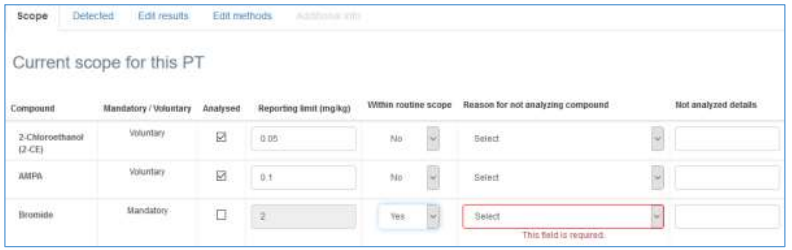
On the bottom of each table you will see the button for ***Final submission.***
Use this button only after you have already entered all your data for this PT and want to submit them for the PT evaluation.
After the Final Submission you will NOT be able to change your results any more.



Scope

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

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



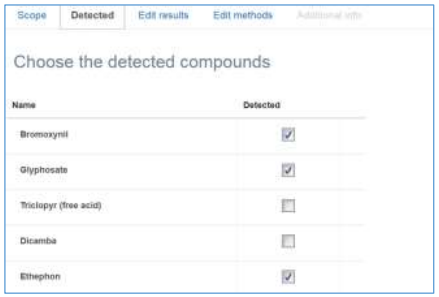
In this table, please firstly select the analytes you have targeted within the EUPT-SRM18 and then enter your Reporting Limit (RL) of each analyte within your PT Scope. The MRRLs were set as default reporting limits. For each pesticide within your PT Scope please **change the Reporting Limit to that of your laboratory**.

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

Even if your laboratory doesn't analyze for certain compounds routinely, you are encouraged to analyze them in this PT and to use this opportunity as a starting point for assessing your newly introduced methods or for expanding your scope.

Detected

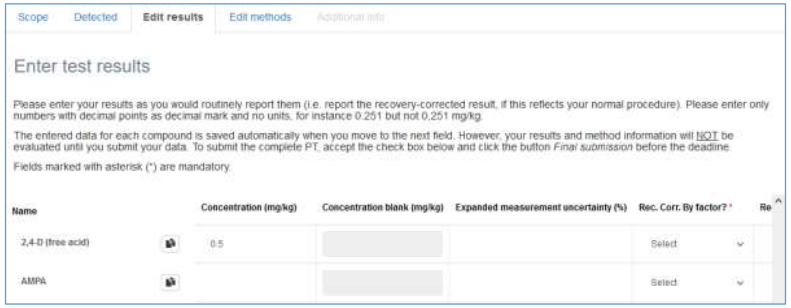
This page will only list analytes that were selected as "analysed" under the table "Scope". Please mark the analytes that you have detected in the Test Item. These selections are used as filters for the subsequent "*Edit results*" table.



Edit results

Click on the *"Edit results"* tab to enter your quantitative results detected analytes in this PT. You can see only those analytes that have been marked on the "*Detected*" table.

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



- Use the scroll bar to reach other parts of the table.
- Use copy function to copy the information from one pesticide to others.

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

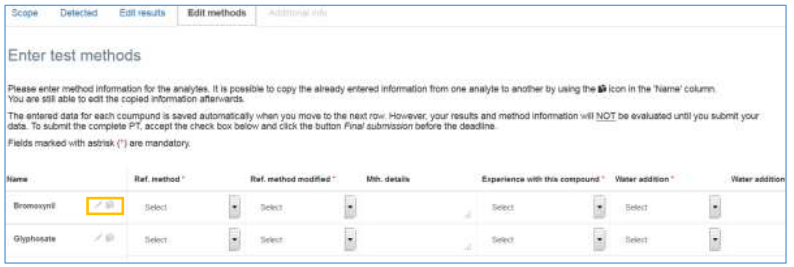
Further information about the "*Edit test results*" table is summarized below.

Field(s)	Explanation
Concentration [mg/kg] **(=Concentration in Test Item)**	Concentration in Test Item in mg/kg, (syntax: e.g., 0.345) **Only numerical values are accepted; pls. use a dot as a decimal separator.**
Concentration blank [mg/kg]	Deactivated, since no blank material was sent to the participants.
Expanded measurement uncertainty [%]	Please indicate the expanded measurement uncertainty value in % (syntax: e.g., "125") 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 rate % [%]	(Mean) recovery rate or bias in % (syntax: e.g., "125") used for deriving the recovery corrected result that will be submitted.
Recovery Obtained	Please choose among the dropdown-options to indicate how the recovery rate used for recovery correction was obtained
Recovery individuals	Number of replicate experiments conducted to obtain the recovery rate/factor used for the correction of results
Recovery details	Please give brief details of, e.g., how the reported recovery rate was obtained, the matrix used if not matching, the spiked compound, the measured compound, the spiking level/range etc.
Comments (= Comments on results and on subcontracting, where applicable)	Here you may add any remarks concerning aspects covered by this subpage for the concerned analyte as well as **any information concerning the SUBCONTRACTING of this analysis to another lab**. Please name the subcontracted lab and indicate if this subcontracting reflects routine procedures.

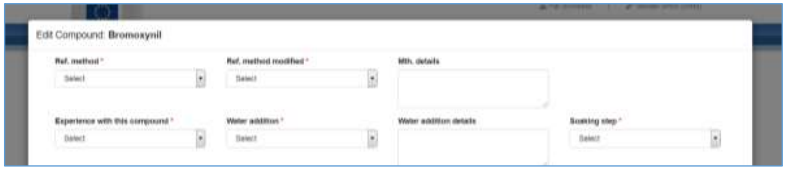
Edit methods

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

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



- Use the scroll bar to reach other parts of the table.
- You can get short description about the columns via mouse-over messages. In some systems or browsers these mouse-over messages do not show up. In this case, please refer to the table below for the explanation and further information about the fields on this page.
- Use the edit function to get an overview of all method-information fields of a selected pesticide in a different format (see screenshot below). However, please note that there is no mouse-over information on the edit view.
- Please note that you need to complete all mandatory fields for the selected compound, before being able to save or close this overview site and return to the initial table view.



- Use copy function to copy the information from one pesticide to another.

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

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

Field(s)	Explanation
Ref. method	Choose from the dropdown 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. If yes, pls. give brief details of the modification under "Mth. details".
Mth. details	Describe your method shortly if it is not on the dropdown menu or indicate shortly the modifications introduced to the selected reference method.
Experience with this compound	Experience of your lab with the analysis of this pesticide (with any type of commodity).
Water addition	Please choose "Yes" if water or a water-containing solvent mixture was added to the sample to assist extraction.
Water addition details	Details on water addition (e.g. amount in mL, step of addition)
Sample Weight (g)	Enter the weight (in gram) for the analytical portion.

Field(s)	Explanation
Extraction/partitioning solvent 1	Choose the solvent from the dropdown menu
Extraction/partitioning solvent 2	Choose the solvent from the dropdown menu, if you use more than one solvent.
Extraction/partitioning solvent 3	Choose the solvent from the dropdown menu, if you use more than two solvents.
Extraction solvent details	Enter details on solvents used in extraction or partitioning steps or if the solvent is not in the drop down menu
Extraction Time	Duration of main extraction step including any waiting time after addition of solvent (Please choose the closest value). If extraction is combined with a chemical transformation, then chose "Combined w. chem. transformation" and indicate the time under "Chemical transformation Time"
Extraction approach	Choose extraction approach from dropdown list
Partitioning salts used	Choose partitioning salt used from dropdown list
pH modified	Indicate if you have modified the pH at any stage of the procedure (e.g. by buffering, acid/base addition)
pH modified details	Please give details on pH modification step(s)
Clean up 1	Choose the clean-up approach employed from the dropdown list
Clean up 2	Choose the clean-up approach employed from the dropdown list, if you use more than one clean up step
Clean up 3	Choose the clean-up approach employed from the dropdown list, if you use more than two clean up steps
Clean up details	Please give details on clean-up step or describe your clean up procedure if it is not listed in the dropdown menu
Chemical transformation	Mark if your procedure included a chemical transformation e.g. hydrolysis, derivatization, reductive cleavage to CS_2, etc.
Chemical transformation Time	Please chose closest time from dropdown list. **If extraction and chem. transf. were combined, indicate closest combined time here.**
Chemical transformation Temp.	Please choose the closest temperature from the dropdown list
Chem. Transf. Details	Please give details on chemical transformation step(s) conducted
Calibration approach	Choose the calibration approach used. **NOTE:** "Procedural calibration" and "Standard additions to sample portions" involve correction for recovery.
Determination Technique	Choose the instrumental technique used to generate your quantitative result
Other Approaches to Corr. PT-Result for Recov.	Shortly describe any OTHER APPROACHES employed for correction of results for recovery. **NOTE:** Corrections for recovery via ILIS or via RECOVERY FACTOR are covered by other specific questions. "PROCEDURAL calibration" and "STANDARD ADDITIONS TO SAMPLE PORTIONS" are covered under "calibration"
Matrix used for calibration	Blank commodity used for matrix-based, matrix-matched or procedural calibration
Matrix calibration details	Please name the blank commodity used to prepare the calibration solutions and any other details of importance, such as differences between sample extract and calibration solution (e.g. in cleanup, dilution etc.)
Compound(s) used for Calibration	Here you can choose your compounds used for calibration
Compound(s) used for Calibration Detail	Here you can specify your compounds used for calibration, in particular, if it is not within the dropdown options.
IS used	**Please choose "No" if no "IS" was used or if the IS was used only 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 calculating the target analyte concentration.
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 **(= Comments on methods and on subcontracting, where applicable)**	Please enter here any comments concerning the analytical method of the selected compound. **Here you can also indicate if the respective analyte was analyzed by a subcontract lab and if this reflects the routine approach. Please name the subcontracted lab**.

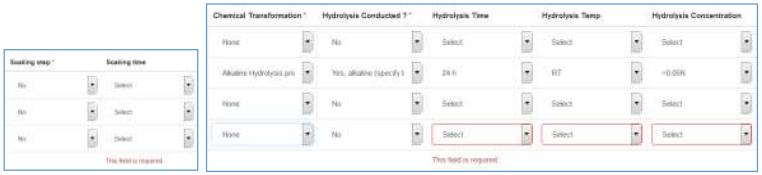
Final Submission

VERY IMPORTANT REMARK!

Make sure to enter values in all required fields. Validate by ensuring *no red rings* are found in the table. Otherwise, you are not able to submit your data.

Please note:

- The red rings or information showing that a field is required are not always automatically and immediately updated after entering or saving data. You may have to **actively click the cells to see the updated status**.
- In some cases, you may probably see the following situation:



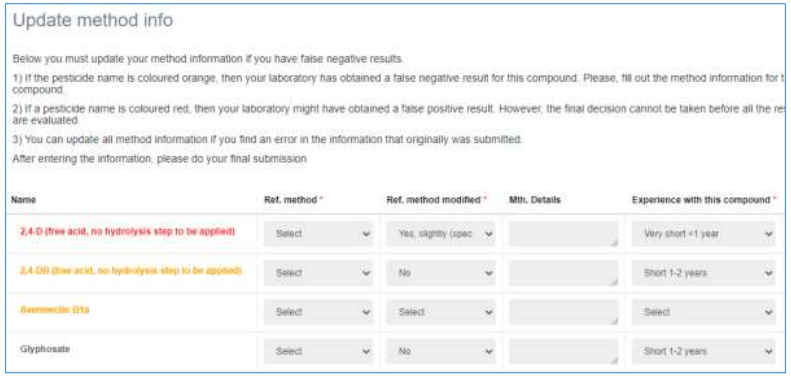
In both screenshots, the entries are correct, since no soaking step or hydrolysis was conducted and soaking time or hydrolysis time/temp/concentration are actually not required.
To "remove" the red rings or the sentence "This field is required": **just click on "select".**

Additional Information

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

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

Click on the yellow-marked EUPT:



Tentatively false positives are written in red colour.

Tentatively false negatives are written in yellow/orange colour.

Please fill-in **the missing method information for the compounds identified as tentatively false negatives. Submit this information by 22 June.**

Please note that during this period, you can not only edit the method information of the analytes preliminarily judged as FNs, but also of all other analytes listed in the table.

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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*".

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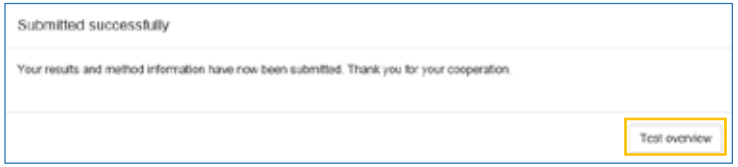
IMPORTANT:

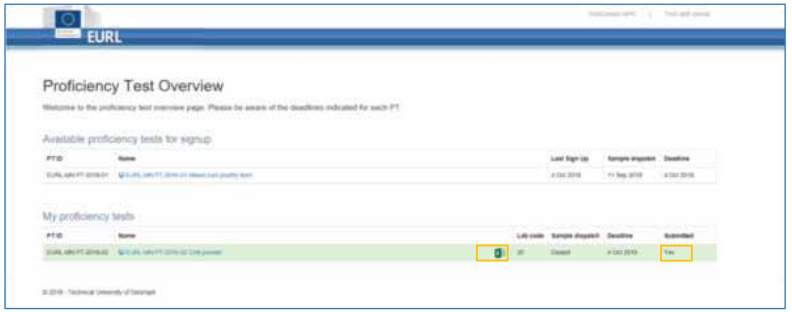
You will **NOT** be able to edit your results after the final submission!
Your data have to be submitted before the deadline on Tue. **16 June 2023, 11 h (11 a.m.), CEST.**

Upon clicking "Final submission" the following pop-up window confirming successful submission of the data will appear on the screen. In parallel, you will receive an email with an attached Excel file, in which your submitted data is compiled. You can also download it form the "*Test Overview*" (please see below).



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

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



IMPORTANT:

After final submission, you will not be able to edit your results any more!
If you find any errors in the exported Excel-file and it is before the submission deadline, please contact the SRM18 organizers per Mail.

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