European Commission

EURL-SRM

EU Reference Laboratories for Residues of Pesticides
Single Residue Methods

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

EUPT – SRM16

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Final Report

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EU PROFICIENCY TEST
EUPT-SRM16, 2021

Residues of Pesticides Requiring Single Residue Methods

Test Item: Milled Sesame

Final Report

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approved by Michelangelo Anastassiades
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Picture on the book cover: Unpeeled sesame, Dr. Eckehard Hiller, CVUA Stuttgart

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FOREWORD

Regulation 625/2007/EC [1] defines the general tasks and duties of the EU Reference Laboratories (EURLs) for Food, Feed and Animal Health[1] including the organisation of comparative tests (proficiency tests = PTs). These PTs are carried out on an annual basis and aim to improve the quality, accuracy and comparability of the analytical results generated by EU Member States within the framework of the EU coordinated control programs as well as national monitoring programs. By participating in PTs laboratories can assess and at the same time demonstrate their analytical performance. The attention to details paid by 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 Regulation 396/2005/EC on maximum residue levels of pesticides in or on food and feed of plant and animal origin [2], all laboratories analysing for pesticide residues within the framework of official controls shall participate in the European Union Comparative Proficiency Tests (EUPTs) for pesticide residues. The participation of OfLs comparative tests organized by the EURLs has been more recently also layed down in Article 38 (2) of the regulation on offical controls (625/2017/EC), where it reads: "Upon request by the European Union reference laboratory or national reference laboratory, official laboratories shall take part in inter-laboratory comparative tests or proficiency tests that are organised for the analyses, tests or diagnoses they perform as official laboratories". Art 101(1)(a) of Regulation 625/2017/EC furthermore prescribes the participation of NRLs in these comparative tests: "National reference laboratories shall, in their area of competence: (a) collaborate with the European Union reference laboratories, and participate in training courses and in inter-laboratory comparative tests organised by these laboratories".

Since 2006 the EURL for pesticide residues requiring the use of Single Residue Methods, EURL-SRM, has annually conducted one scheduled Proficiency Test. Five of those 16 EUPT-SRMs were conducted in collaboration with the EURL for pesticide residues in Fruits and Vegetables (EURL-FV) with apple juice (EUPT-SRM1, 2006), carrot homogenate (EUPT-SRM3, 2008), apple purée (EUPT-SRM5, 2010), potato homogenate (EUPT-SRM8, 2013) and spinach homogenate (EUPT-SRM11, 2016) as test items. Five other EUPT-SRMs were conducted in collaboration with the EURL for pesticide residues in Cereals and Feeding Stuff (EURL-CF) with wheat flour (EUPT-C1/SRM2, 2007), oat flour (EUPT-C3/SRM4, 2009), rice flour (EUPT-C5/SRM6, 2011 and EUPT-SRM15, 2020) and maize flour (EUPT-C9/SRM10, 2015) as test items. One EUPT-SRM was conducted in collaboration with the EURL for Residues of Pesticides in Food of Animal Origin (EURL-AO), with bovine liver homogenate (EUPT-SRM14, 2019) as commodity. The remaining five EUPT-SRMs were organized by the EURL-SRM unilaterally. One of them concerned a commodity of animal origin, namely cow's milk (EUPT-SRM9, 2014), whereas the remaining four PTs were all based on commodities of plant origin, namely strawberry homogenate (EUPT-SRM12, 2017), milled dry lentils (EUPT-SRM7, 2012), milled soybeans (EUPT-SRM13, 2018) and finally milled sesame seeds (EUPT-SRM16, 2021) in the present PT. The later commodity was selected as a reaction to the massive findings of 2-chloroethanol in sesame seeds and products thereof in the second half of 2020.

Participation in EUPTs is mandatory for all NRLs for pesticides requiring Single Residue Meth- ods (NRL-SRMs) and for all OfLs analysing pesticide residues within the framework of national or EU control programs in commodities represented by the respective EUPT test item. Laboratories in EU Member States analysing pesticide residues within the frame of import controls according to 1793/2019/EG are also considered as performing official controls in the sense of VO (EG) 2017/625 and are thus also obliged to take part in EUPTs. OfLs from EFTA countries (Iceland, Norway and Switzerland) contributing data to the EU-coordinated community control programs, EU laboratories analysing official organic samples within the frame of Reg. 889/2008/EC,

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

as well as OfLs from EU-acceding or -candidate countries (FYROM, Montenegro, Serbia and Turkey) are also invited to take part in EUPTs.

Commercial laboratories in EU or EFTA countries willing to be subcontracted by EU Member States for the analysis of ethylene oxide related residues in sesame seed imports were exceptionally accepted to participate in the current PT. A limited number of laboratories from third countries are allowed to take part in this exercise, too. In the present PT and in case of ethylene oxide and 2-chloroethanol results of all participating laboratories were considered in the calculation of the assigned values, regardless of whether they are OfLs from EU/EFTA countries or not. For all other analytes present in the current PT, 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-status, a tentative list of EU-labs considered as obliged to participate in the EUPTs is published at the beginning of each year. The pesticide scope is not taken into account in these lists. NRLs and OfLs can see their participation status on the registration page. Laboratories listed as being obliged to participate in an EUPT exercise in a given year, but deciding not to take part, are always asked to state the reason(s) for their non-participation. The same applies to laboratories originally registering to participate in a certain EUPT but finally not submitting results.

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

CONTENT

EUROPEAN COMMISSION –
EU-PROFICIENCY TEST ON RESIDUES OF PESTICIDES
REQUIRING SINGLE RESIDUE METHODS
TEST ITEM: MILLED SESAME
EUPT-SRM16, 2021

INTRODUCTION

Originally, the EUPT-SRM16 was planned to be conducted using aubergines as a commodity, but following an urgent request by DG-SANTE, this plan was dropped and sesame seeds were used instead. This shift in the planning was related to the massive findings of 2-chloroethanol (2-CE) residues in various dry food products within the EU-market. 2-CE is reaction product of the highly effective fumigant ethylene oxide (EO), that has been banned within the EU since decades for toxicological reasons. The MRLs were thus set at the LOQ. The EO/2-CE incidence emerged in the second half of 2020 and initially mainly affected sesame and sesame products (mainly originating from India), but it was foreseeable that other products would also be affected and that this topic would be occupying the EU-enforcement for some time to come. At the beginning of the insidence, only a hand full of OfLs within the EU were able to analyze for EO/2-CE and even fewer were able to analyze for this compound on a routine basis. This was partly due to the complexity of the traditional analytical procedure. In the private laboratory sector the situation was only slightly better. Thus, when the EO/2-CE-incidence errupted, the pool of available laboratories that could be potentially employed for conducting import controls of sesame for residues of 2-CE/EO (as required by the regulation) was rather small and insufficient. The EURL-SRM was therefore prompted by DG-SANTE to find out which laboratories within the governmental and private sector are able to analyze for EO/2-CE, or willing to implement methods for this purpose. Furthermore, the EURL-SRM was prompted to act towards increasing the analytical capabilities of the labs and to help the labs to demonstrate their profficiency by organizing PTs. A simple QuEChERS-based method was thus developed, allowing the analysis of EO and 2-CE in parallel. Additionally, contact with laboratories in India was established to ensure efficient controls in this country. Already in late Autumn 2020 a first ad-hoc PT based on sesame seeds with incurred levels of EO and 2CE was organized by the EURL-SRM and thereafter a list of laboratories that were able to analyze for EO/2-CE was published at the request of DG-SANTE. Most of the labs that have newly implemented the analytical procedure were highly interrested in the participation in PTs in order to demonstrate proficiency and to facilitate the accreditation process of the newly implemented methods.

It was the wish of DG-SANTE that the EUPTSRM16 was conducted using sesame seeds as commodity, that it includes EO and/or 2-CE on the the target pesticide list and that it is conducted as soon as possible. With the goal to considerably strengthen the controls of EO/2-CE residues in food, EUPT-SRM6-participation should not only include OfLs but also private laboratories that have recently implemented a method for this purpose and have declared their interrest in participating in official import control activities as well as laboratories in India, the country with the most problems as regards residues of EO/2-CE.

In a pre-PT survey the potentially participating laboratories were asked to indicate their preferences as regards the timing of the PT as well as their preferences as regards the other analytes to be included in the Target Pesticides List.

Considering the results of the survey and various additional aspects, the EUPT-SRM16 was scheduled from 22 March till 27 April 2021. Within the EURL-Web-Portal an EUPT-SRM16-Website was set up on 15 February 2021 with links to all documents relevant to this EUPT (i.e., Announcement/Invitation Letter, Calendar, Target Pesticides List, Specific Protocol and General EUPT Protocol). These documents were also uploaded on CIRCA BC.

On 22 February 2021 the "Announcement/Invitation Letter" and "Call for Registration" **(Appendix 11)** for the EUPT-SRM16, accompanied by SRM16 Calendar and Target Pesticides List **(Appendix 10)**, was published on the EUPT-SRM16-Website. These documents were sent to all NRL-SRMs, all OfLs analysing pesticide residues in food of plant origin with high lipid content or of feed within the framework of official controls, including laboratories involved in the import controls of products listed under Reg. (EU) 1793/2019, as far as they were tracked in the EURL-DataPool, as well as to EU laboratories analysing official organic samples within the frame of Reg. 889/2008/EC. NRLs and OfLs from EFTA and EU-candidate countries were also invited if their contact data were available. In agreement with the EU Commission commercial laboratories in EU or EFTA countries willing to be subcontracted by EU Member States for the analysis of ethylene oxide related residues in sesame seed imports were exceptionally accepted to participate in the current PT. In addtion, official and commercial laboratories from 3rd countries, in particular those involved in the export control of Indian sesame to the EU, were also invited and accepted as participants in the present PT. The results from laboratories outside EU and EFTA OfLs are not taken into account for the calculation of the robust mean which is used as the assigned value, but in this particular PT an exception was made in the case of 2CE, EO and EO (sum). Here the numerical results of all participating laboratories were used for the calculation.

Based on commodity scope and NRL-status (NRL-SRMs) all official laboratories were allocated a tentative status as regards their obligation to participate in the EUPT-SRM16. This status was stored 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 that the real obligation to participate is based on the respective regulations. From 23 February till 9 March 2021, laboratories obliged to or interested in this PT could register for their participation using the registration form on www.eurl-pesticides-datapool.eu. Obliged laboratories not intending to participate in the PT were asked to login and state the reason for their non-participation. The SRM16 Specific Protocol **(Appendix 9)** and Webtool Guideline **(Appendix 12)** were provided to the participants on 15 and 18 March, respectively, via hyperlinks in e-mails.

In total, 116 official laboratories (including NRLs) from 33 countries (26 EU-Member States, 2 EFTA-countries, 1 EU candidate country and 4 countries outside Europe) registered for participation in the EUPT-SRM16. In addition, 12 EU commercial laboratories willing to be subcontracted by EU Member States for the analysis of ethylene oxide related residues in sesame seed imports, participated in the current PT, too. All laboratories having registered for the PT had also submitted results before the results submission deadline.

The proficiency test EUPT-SRM16 was conducted using hulled sesame seeds containing incurred levels of 2-CE, which were partly provided by a commercial laboratory and partly by a laboratory of a supermarket chain. Sesame seeds from a local supermarket containing only low levels of 2-CE, but higher residues of chlorate were also used. The test item was prepared by spiking to a portion of the sesame seeds with standard solutions containing in total 11 compounds, followed by through homogenization with the rest of the material, cryo-milling with dry ice and portioning into bottles. More details are given in **Chapter 1 "Test Materials and Blank Material"**.

1. TEST ITEM

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

Given the frequent findings of ethylene oxide residues in sesame from August 2020 onwards, and the multiple recalls and RASFF notifications of sesame seeds, DG-SANTE asked the EURL-SRM to use sesame seeds as the commodity for the EUPT-SRM16, as this would help the official labs to check and demonstrate their performance in this area.

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.) DG-SANTE's wish that the PT-material should contain ethylene oxide (EO) and/or its conversion product 2-chloroethanol (2-CE), preferably in incurred form;
2.) the scope of the present and upcoming EU-coordinated control programs;
3.) the scope of the DG-SANTE working document on pesticides to be considered in national control programmes (SANCO/12745/2013 rev. 10(3); 26 – 27 November 2019);
4.) the relevance of pesticides to the specific commodity;
5.) the results of the survey on the analytical capabilities and intentions of the laboratories.

The minimum required reporting levels (MRRLs) were set as follows:

- at 0.01 mg/kg for *chlormequat chloride*, ***matrine,*** *mepiquat chloride,* ***nicotine, trimesium***;
- at 0.02 mg/kg for ***diquat***, *ethylene oxide (EO)*, *fosetyl,* ***glufosinate***, *MPP, N-acetyl-glufosinate,* ***paraquat***,
- at 0.04 mg/kg for ***chlorate*** for ***ethephone***,
- at 0.04 mg/kg for ***2-chloroethanol (2-C***E), ***ethylene oxide (sum) (EO (sum)***),
- at 0.05 mg/kg for *AMPA,* ***glyphosate***, ***phosphonic acid***, *N-acetyl glyphosate*,
- at 2.0 mg/kg for ***bromide ion***.

1.2 Analysis of the Sesame Intended to be Used to Prepare the PT-Material

In order to prepare the PT-Material, different amounts of sesame seeds from various origins were mixed together. This material was either purchased or provided by two commercial laboratories. The collected material was analysed for any residues of the pesticides on the Target Pesticides using the QuEChERS method for EO/2-CE or the QuPPe method (www.quppe.eu) for the remaining compounds. Considering

Table 1-1: Analytes present in the SRM16 test material and their application history

Analytes	Contained in seeds used?	Spiked in lab	Compounds applied in lab	Analytes	Contained in seeds used?	Spiked in lab	Compounds applied in lab
2-CE	Yes	No	–	Glyphosate	No	Yes	Glyphosate
Bromide	Yes	Yes	Potassium bromide	Matrine	No	Yes	Matrine
Chlorate	Yes	Yes	Sodium chlorate	Nicotine	Yes	Yes	Nicotine
Diquat	No	Yes	Diquat dibromide monohydrate	Paraquat	No	Yes	Paraquat dichloride hydrate
Ethephon	No	Yes	Ethephone	Phosphonic acid	Yes (traces)	Yes	Phosphonic acid
Glufosinate	No	Yes	Glufosinate ammonium	Trimesium	Yes (traces)	Yes	Trimethylsulfonium iodide

the encountered levels of "incurred" ***2-CE, chlorate, bromide ion, nicotine, trimesium*** and ***phosphonic acid*** in these batches, they were mixed together at suitable proportions to obtain a material containing these compounds within the desired concentration range, where possible. Some of these compounds had to be additionally spiked (**Table 1-1, p. 1**).

1.3 Investigations on the Impact of the Proportion of Laboratory-Spiked Sesame Seeds on the Homogeneity of the Final Material

It was initially still doubtful whether intact or milled sesame seeds would be sent to the participants. It was thus considered important to check how the proportion of the spiked material in the final material affects the portion-to-portion variability of the latter. In a visual test, black and white sesame seeds were mixed at a 1:10 proportion and the black seeds present in various sub-samples of 2 g were counted. The portion-to-portion variability of the black seeds was deemed too high for the purpose of the PT and it was thus decided that the material would need to be milled to be on the safe site. It was also contemplated whether this milling should be done by the organizers or the participants. Considering that some participants would opt to use the unmilled material and that the milling procedure would vary considerably among the participants, it was finally decided, that the participants should receive a milled material as this would reduce the number of factors having an influence on the results.

In a separate experiment aiming to investigate how the proportion of the spiked material in the final material affects the homogeneity, various portions of sesame seeds were spiked with different analytes and left to dry overnight. The various spiked portions were then mixed together, diluted with unspiked material, mixed again and then milled cryogenically, using dry ice (the originally planned analysis of intact sesame was omitted as the decision was taken to provide the PT-participants with milled material). Analytical portions of 2 g and 5 g were used to investigate the impact of the analytical portion size on the sub-sampling variability of the milled homogenate (n = 6). Based on the results of this study, it was concluded that even at a 1 % share of the spiked portion in the total material, and even when using analytical portions of 2 g the portion-to-portion variability remains acceptable.

1.4 Material production and preliminary homogeneity test

In total, 42.1 kg commercially available sesame seeds were used to prepare the PT test material. For spiking, 11 compounds dissolved in 1 L of a solvent mixture consisting of water and acetone (1 : 9) (see **Table 1-2**) were evenly spread over four portions of 1 kg sesame seeds each (4 kg of material in total). The spiked material was shaken until the solvent was evaporated and then left standing over night for the seeds to dry-out completely. The spiked seeds were then incorporated to the rest of the material and thoroughly mixed over 5 hours by a drum hoop mixer. For milling, 150 g portions were used each time. The seeds were premixed with dry ice pellets (3 mm) (sample : dry ice ratio = 2 : 1), cryogenically milled into a fine powder and united into a large plastic vessel. The material was then sieved to remove any coagulated material (formed when mixing temperature was high) and mixed repeatedly by hand.

1.4.1 Preliminary analysis prior to bottling

To double-check the spiking levels and to verify that the material is sufficiently homogeneous, prior to bottling 8 subsamples of the homogenate were sampled at 8 different spots and subjected to analysis of selected analytes using the QuEChERS and QuPPe method. The sample material was found to be homogeneous (RSD 4 – 9 %) and ready for packaging.

Table 1-2: Analytes spiked into 42.1 kg sesame seeds for the preparation of EUPT-SRM16 test material

Analytes expr. as required in the TPL	Conc. (Stock Solution) [mg/ml]	Stock solution employed for preparing spiking mixture [ml]	Theoretically expected concentration in the Test Material [mg/kg]	Incurred levels
Bromid	100	9	21.4	yes
Ethephon	1.0	11.5	0.273	
Glufosinate	1.0	12	0.286	
Glyphosate	2.0	16.1	0.763	
Phosphonic acid	2.0	11.5	0.55	yes
Chlorate	1.0	9	0.21	yes
Diquat	1.0	11	0.261	
Nicotine	1.0	13.5	0.321	traces
Paraquat	1.0	11.5	0.273	
Trimethylsulfonium	1.0	3	0.071	traces
Matrine	1.0	6.5	0.154	

1.5 .Bottling and packaging of the Test Item

Approximate 180 g portions were filled into pre-numbered screw-capped polyethylene bottles, with the bottle numbers following the chronological order of bottling. The test items were then sealed and packed into the parcels together with cooling elements and placed in a freezer at -18 °C. Bottling and packing was conducted five days prior to shipment, which ensured that on the day of shipment the cooling elements were deep frozen.

1.6 Delivery of PT Materials to Participants

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

Among the 115 packages sent to OfLs and commercial laboratories in EU and EFTA countries, 102 (89 %) reached the participating labs within 24 hours, 12 packages within 48 hours and only one package arrived the recipient laboratory on the third day due to special security measures at the airport. The 13 deliveries to countries outside the EU and EFTA zones was accomplished in 3 cases within 48 hours and in another 3 cases within 3 days. In the 7 remaining cases delivery was delayed due to lengthy customs clearance procedures and took between 7 and 18 days. The submission deadline of those laboratories, whose PT material took more than one week to arrive, was extended considering the individual arrival day to ensure that these labs would have a similar time at their disposal for processing the samples.

Overall, the vast majority of the parcels arrived at the laboratories within two days. Details on the shipment duration are shown in **Appendix 2**. All dispatched material was accepted by the participants, and they reported its good condition upon arrival.

The organisers would like to appeal to 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 and asking for an acceleration of the clearance procedure or placing the parcel in a cool place until clearance is granted. The participants are furthermore encouraged to contact the local office of the shipping company to ensure optimal delivery timing.

1.7 Analytical Methods

The analytical methods used by the organisers to check the homogeneity and storage stability of the analytes contained in the test item and to verify the absence of the remaining TPL-analytes, are summarized in **Table 1-3**. For more details on the methods used, please refer to the EURL-SRM website: http://www.eurl-pesticides.eu (→EURL-SRM Methods or Analytical Observations).

1.8 Homogeneity Test

After filling the test item into bottles, 10 bottles were randomly chosen for the homogeneity test and two analytical portions per bottle were analysed by each analytical method. Both the order of sample preparation and the order of extract injection into the analytical instruments were random. Matrix-matched

Table 1-3: Analytical methods used by the organisers to check for the homogeneity and storage-stability of the pesticides present in the test item and to demonstrate the absence of other pesticides in the blank material.

QuEChERS method Method [3]: involving: weighing of 2 g sesame homogenate into a sealable vessel, addition of ILIS, water adjustment, addition of acetonitrile, shaking, centrifugation at -10°C, cleanup with PSA and C18, pH adjustment, addition of analyte protectant and direct determination by GC-MS/MS in the EI (pos.) mode.				
Compound	**IS**	**Determinative analysis**		**Notes**
2-Chloroethanol	**2-Chloroethanol D_4**	**GC-MS/MS**	**EI (pos)**	
Ethylene oxide (sum)	**BNPU**	**GC-MS/MS**	**EI (pos)**	
*Ethylene oxide**	*Ethylene oxide D_4*	*GC-MS/MS*	*EI (pos)*	
QuPPe-AO Method [5]: involving: weighing of 2 g sesame homogenate into a sealable vessel, addition of ILISs, water adjustment, addition of methanol containing 1 % formic acid, addition of an extra amount of 100 µL formic acid and EDTA, shaking, freeze-out, centrifugation, dilution 1:1 with ACN, filtration with Ultrafiltration filters and direct determination by LC-MS/MS in the ESI (neg.) or ESI (pos.) mode.				
Compound	**IS**	**Determinative analysis**		**Notes**
Bromide	–	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.7**
Chlorate	**Chlorate $^{18}O_3$**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.7**
Diquat	**Diquat D_8**	**LC-MS/MS**	**ESI (pos)**	**QuPPe M4.1**
Ethephon	**Ethephon D_4**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.6**
Glufosinate	**Glufosinate D_3**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.6**
Glyphosate	**Glyphosate $^{13}C_2{}^{15}N$**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.6**
Matrine	**Matrine D_4**	**LC-MS/MS**	**ESI (pos)**	**QuPPe M4.1**
Nicotine	**Nicotine D_4**	**LC-MS/MS**	**ESI (pos)**	**QuPPe M4.1**
Paraquat	**Paraquat D_8**	**LC-MS/MS**	**ESI (pos)**	**QuPPe M4.1**
Phosphonic acid	**Phosphonic acid $^{18}O_3$**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.7**
Trimesium	**Trimesium D_9**	**LC-MS/MS**	**ESI (pos)**	**QuPPe M4.1**
*AMPA**	*AMPA $^{13}C^{15}N$*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Chlormequat**	*Chlormequat D_4*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.1*
*Fosetyl**	*Fosetyl D_5*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Mepiquat**	*Mepiquat D_3*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.1*
*Glufosinate metabolite MPP**	*MPP D_3*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Glufosinate metabolite N-acetyl-glufosinate**	*N-acetyl-glufosinate D_3*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
*Glyphosate metabolite N-acetyl-glyphosate**	*N-acetyl-glyphosate $^{13}C^{15}N$*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.6*
* : To check for absence in Blank Material				

calibration using extract prepared from blank material or procedural calibration using blank material were applied for quantification. For all compounds, analytical portions of 5 g were used.

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

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

As all target compounds passed the homogeneity test, the test item was considered to be sufficiently homogenous and suitable for the EUPT-SRM16.

Table 1-4: Statistical evaluation of homogeneity test data (n = 10), details please see **Appendix 3**.

	COMPULSORY				
	Bromide ion	Ethephon	Glufosinate	Glyphosate	Phosphonic acid
Analytical portion size [g]	5	5	5	5	5
Mean [mg/kg]	20.6	0.239	0.218	0.500	0.705
between-samples STD	0.2773	3.94×10^{-5}	0.0002	0.0003	0.0003
Check Value	1.5445	0.0179	0.0164	0.0375	0.0529
Passed/Failed	passed	passed	passed	passed	passed

	OPTIONAL COMPOUNDS							
	2-CE	EO (sum)	Chlorate	Diquat	Matrine	Nicotine	Paraquate	Trimesium
Analytical portion size [g]	5	5	5	5	5	5	5	5
Mean [mg/kg]	5.06	2.77	0.985	0.212	0.121	0.315	0.242	0.069
between-samples STD	0.0118	0.0035	0.0002	6.19×10^{-5}	1.43×10^{-5}	8.02×10^{-5}	0.00	7.17×10^{-7}
Check Value	0.3793	0.2075	0.0739	0.0159	0.0091	0.0236	0.0180	0.0051
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed

1.9 Storage Stability Test

In the Specific Protocol laboratories were recommended storing the samples in the freezer until analysis. The stability test samples were thus also stored under the same conditions. Shortly after the shipment of the samples to the participants, three of the spare test item bottles were chosen randomly out ot the bottles used for the homogeneity test. The portions of stability tests 1 were the same as those used in the homogeneity test. The were taken and extracted immediately. The remaining material for the stability tests 2 and 3 were placed in the freezer at –20 °C until performing the tests. The methods described in **Section 1.7 (p. 4)** were applied for the tests. The extracts of all stability test extractions were stored in the freezer at –20 °C and measured iso-chronically (= under repeatability conditions within the same measurement sequence) at a day suitable for the laboratory. The dates of extractions by each method are shown below

Stability test 1 (extraction shortly before shipment):
30 March 2021 (analytes via QuPPe-AO-method)
30 March 2021 (analytes via QuEChERS-method)

Stability test 2 (extraction 29 days after shipment):
20 April 2021 (analytes via QuPPe-AO-method)
23 April 2021 (analytes via QuEChERS-method)

Stability test 3 (extraction shortly after the end of PT):
05 May 2021 (analytes via QuPPe-AO-method)
07 May 2021 (analytes via QuEChERS-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 test, which was long enough to exceed the duration of the PT, and during which the samples were stored at –18 °C (= recommended conditions), all analytes contained in the test item were shown to be sufficiently stable (**Table 1-5, p. 7**). 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 1-5 (p. 7)** and **Appendix 4**.

1.10 Transport Stability Test

With the exception of seven laboratories outside the EU and EFTA, all other participants received the sample packages within three days and in a very good condition. The results reported by the seven laboratories having received the material after 7 till 18 days did not imply any significant negative impact of this prolonged transport on the material. Furthermore, the assigned values of all analytes except 2-CE are calculated on the basis of results submitted by EU and EFTA laboratories, that have almost entirely received the samples within two days. 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 and the organizers therefore decided to skip the transport stability test in this PT.

Table 1-5: Results of storage stability test (storage at -18 °C). For the details of each analytes please see the text and **Appendix 4**.

	Compulsory Compounds				
	Bromide ion	Ethephon	Glufisinate	Glyphosate	Phosphonic acid
Storage at –18 °C (mean values in mg/kg)					
Analysis 1	20.5	0.238	0.221	0.508	0.728
Analysis 2	19.1	0.231	0.207	0.515	0.680
Analysis 3	19.3	0.222	0.213	0.471	0.692
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	1.19 (-5.8%)	0.015 (-6.4%)	0.0084 (-3.8%)	0.037 (-7.3%)	0.036 (-4.9%)
$0.3 \times \sigma_{pt}$ [mg/kg]	1.6	0.017	0.016	0.038	0.051
Passed/Failed	passed	passed	passed	passed	passed

	Optional Compounds							
	2-CE	EO (sum)	Chlorate	Diquat	Matrine	Nicotine	Paraquate	Trimesium
Storage at –18 °C (mean values in mg/kg)								
Analysis 1	5.04	2.76	1.027	0.207	0.117	0.311	0.247	0.070
Analysis 2	5.27	2.88	1.012	0.215	0.110	0.296	0.243	0.066
Analysis 3	5.24	2.87	0.999	0.193	0.110	0.309	0.251	0.065
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.1928 (3.8%)	0.1058 (3.8%)	0.0277 (-2.7%)	0.0138 (-6.7%)	0.0073 (-6.3%)	0.0023 (-0.8%)	0.0045 (1.8%)	0.0052 (-7.4%)
$0.3 \times \sigma_{pt}$ [mg/kg]	0.3368	0.1875	0.0775	0.0183	0.0093	0.0198	0.0198	0.0055
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed

1.11 Organisational Aspects

1.11.1 Laboratory Status – Mandatory and Optional Participation

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

Following DG-SANTE instructions, obliged labs that were not intending to participate in the EUPT-SRM16 were instructed to provide explanations for their non-participation.

1.11.2 Announcement / Invitation and EUPT-SRM16 Website

The initial planning for the EUPT-SRM16 foresaw the use of aubergines as PT-material. However, due to the massive problems caused by residues of 2-CE in sesame seeds DG-SANTE requested to conduct the PT using sesame, and it was decided at the end of January 2021 to run the EUPT-SRM16 based on sesame seeds. The EUPT Scientific Commitee was informed about this change. This PT had to be run as soon as possible and include EO and/or 2-CE in the scope. Commercial laboratories in EU or EFTA countries willing to be subcontracted by EU Member States for the analysis of ethylene oxide related residues in sesame seed imports were also exceptionally accepted to participate in the current PT, as their participation would facilitate their potential designation as OfLs for import controls.

In a survey run among the OfLs within the network, the laboratories were asked about their interest to participate in this PT on sesame seeds, and about their intention to analyse for EO/2-CE. The labs were also requested to indicate the time by which they expect to have managed to establish an analytical procedure and about the preferred period for participation in the EUPT on sesame.

Considering all this information, the EUPT-SRM16 was finally scheduled to run from 22 March till 27 April 2021. Within the EURL-Web-Portal an EUPT-SRM16-Website was set up on 15 February 2021 with links to 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**). These documents were uploaded both to the EURL-Web-Portal and to the CIRCA BC.

On 22 February 2021 the Announcement/Invitation Letter for the EUPT-SRM16 was published on the EUPT-SRM16-Website and sent to all NRL-SRMs and all OfLs analysing pesticide residues in food of plant origin with high lipid content or in feed. Laboratories involved in the import controls of products listed under Reg. (EU) 1793/2019, as far as they were tracked in the EURL-DataPool, as well as to EU laboratories analysing official organic samples within the frame of Reg. 889/2008/EC were also informed. The latter laboratories were considered eligible but not obliged to participate. All these labs were tracked on a list that was prepared by the EURL-SRM at the request of DG-SANTE following another survey. This list was made available on-line for the convenience of authorities involved in EU-import controls. 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. It was indicated to the OfLs, that their obligation to participate in EUPTs arises from the respective regulations, irrespective of the content of the tentative list of obliged laboratories.

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 being obliged to participate in the current EUPT, regardless of whether they were intending to participate in this exercise or not, were requested to either register or to state their reasons for non-participation using the same website. Upon registration or change of registration status, the labs received an electronic confirmation about their participation or non-participation in the current PT.

1.11.4 Distribution of the Test Items

The PT Test Items were shipped to the address indicated by the participants on 22 March, 2021. The participants were given the possibility to order one or two portions of the PT-material. Laboratories having requested one bottle were sent one thermo-insulated polystyrene box containing one bottle of the deeply

frozen test item (180 g test material), including two gel-packs each. Participating laboratories ordering a double amount of the test material received two parcels with one test item each.

On 15 March, detailed instructions on how to treat the test item upon receipt were provided to the participating laboratories in the Specific Protocol (**Appendix 9**).

1.11.5 Webtool for Results Submission and Confidentiality

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

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

The participants received their login credentials from the programmer at the DTU on the day of shipment. It was planned to make the Webtool accessible on 23 March, one day after dispatch. Due to unexpected technical difficulties, the Webtool has not been able to be open till 8 April. The participants were informed of this delay on 23 March and still had sufficient time for submission of their results by the submission deadline on 27 April 2021.

On 15 March, one week prior to the sample shipment the organizer provided together with the specific protocol a guideline on the Webtool, including how to login to the Webtool, how to get the lab code for the EUPT-SRM16, as well as all fields to be filled in. The specific protocol, containing additional information on how to proceed with the PT-sample and the reporting of the results via the Webtool was also distributed on this day.

The participants whose parcels took more than one week to arrive, obtained individually extended submission deadlines that were adjusted on the arrival date of their PT material as stored in the database of the delivery company.

After the submission deadline, participants could check in the Webtool, if they have obtained any tentative false positive or false negative results. In the latter case, they were requested to report method details for compounds of false negative results via the Webtool.

Five laboratories have only targeted two analytes from the TPL within this EUPT, namely chlormequat choride and mepiquat chloride. Both of them were listed on the TPL but not contained in the PT material. Although each of these five laboratories had correctly not detected the concerned analytes, they were not able to submit any numerical values and therefore not able to submit its results via the Webtool. In order to complete the Webtool and submit the results, one of those laboratories entered very small values for the analysed pesticides and informed the organizers about this work-around via email. The other four laboratories contacted the organizers directly. The organizers completed the concerned PTs in the Webtool database and reported the special case to the Webtool team.

1.11.6 Actions following Results Submission and Preliminary Report

On 18 May, 2021, the preliminary report on the EUPT-SRM16 with the preliminary assigned values was released and sent to the participants. Laboratories having submitted false positive results as well as having receiving any |z-score| > 2 were asked to investigate the reasons and to report them using a individuel Excel sheet provided by the organizors.

2. EVALUATION RULES

2.1 False Positives and Negatives

2.1.1 False Positives (FPs)

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

2.1.2 False Negatives (FNs)

These are results of target analytes reported as "analysed" but without reporting numerical values, although they were used by the organiser to prepare the test item and were detected, at or above the MRRL, by the organiser and the overwhelming majority of the participating laboratories. In accordance with the General Protocol z-scores for false negatives are calculated using the MRRL as the result, or using the lab's reporting-limit (RL), if this is lower than MRRL. Following the General Protocol, results reported as "< RL" without providing a numerical value are also judged as false negatives. 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 values x_{pt} of each pesticide in the PT is established using the mean value of robust statistics using Algorithms A (x^*) [6] of all reported results generated by OfLs from EU and EFTA countries. In the present PT and in case of EO and 2-CE, results of all participating laboratories were considered in the calculation of the assigned values, regardless of whether they are OfLs from EU/EFTA countries or not. Results associated with obvious mistakes and gross errors may be excluded from the population for the establishment of the assigned values. The add-in "RobStat" provided by Royal Society of Chemistry was used to calculate the assigned values with the convergence criterion = 10^{-6}.

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

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

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

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

Using the assigned value derived by robust mean, the z-scores of the participants' results were calculated using the formula in **Section 2.4**. All Results achieving z-scores > 5 are regarded preliminarily as outliers. If confirmed by Grubbs' test as outliers, these results are excluded from the results population for the establishment of the assigned value, and the corresponding analyte is 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, the EUPT-Scientific Committee agreed to apply a fixed fit-for-purpose relative standard deviation (FFP-RSD) of 25 % for calculating the z-scores. The fixed target standard deviation using the fit-for-purpose approach ($FFP\text{-}\sigma_{pt}$), for each individual target analyte is calculated by multiplying the assigned value by the FFP-RSD of 25 %. In addition, the robust relative standard deviation of the assigned value (CV^*) is calculated for informative purposes.

2.4 z-Scores

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

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

Where

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

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

The z-scores are classified as follows:

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

For results considered as false negatives, z-scores are calculated using the MRRL or the RL, if RL < MRRL. No z-scores are allocated to false positive results. In case that the MRRL is higher than 25 % of the assigned value and therefore the calculated z-score for a false negative result is higher than -3 and reaches the classification "questionable", the z-score will be set at -3.5.

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-Score: "Average of the Absolute z-Scores (AAZ)"

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 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

116 official laboratories (including NRLs) from 33 countries (26 EU-Member States, 2 EFTA-countries, 1 EU candidate country and 4 countries outside Europa) registered for participation in the EUPT-SRM16. In addition, 12 EU commercial laboratories willing to be subcontracted by EU Member States for the analysis of ethylene oxide related residues in sesame seed imports, participated in the current PT, too. For the first time, all laboratories having registered for the PT had also submitted results. An overview of the participating laboratories and countries is given in **Table 3-1**. All laboratories having participated in this EUPT are given in the list in **Appendix 1**. Malta was represented by one OfL based in Germany with two subsidiaries for its routine analysis.

In total, 150 EU-OfLs, including NRL-SRMs, regardless of their commodity scope, as well as all EU-OfLs analysing for pesticide residues in feed or food of high fat content, were originally considered as being obliged to participate in the present EUPT. These laboratories were invited to register on the online registration page for their participation in the current PT or to provide an explanation for their non-participation. Other EU-OfLs wihtout obligation on participation were also invited to participate in the current PT on voluntary basis.

37 obliged laboratories explained their non-participation with the fact that the matrix sesame or the SRM16 target pesticides or both were out of or not yet included in their routine scope. Excluding those 36 laboratories that provided sufficient explanations, the number of EU-laboratories considered as being obliged decreased to 113, among them 83 participated and submitted results. In addition, 20 EU-OfLs registered for participation on voluntary basis and submitted results. 30 (27 %) of the 113 laboratories finally considered as obliged for participation did neither register for the PT nor provide any explanation for non-participation. These laboratories originated from 8 countries as follows: IT (9×), ES (5×), RO (4×), DE (3×), PL(2×), as well as each one from DK, EE, FR, GR, HR, NO, and SI.

Table 3-1: Number of laboratories listed as being obliged to participate in the EUPT-SRM16, labs that registered to participate in the PT and submitted results (grouped by contracting country)

EU: NRLs and OfLs							
Contracting Country [1)]	Labs originally considered to be obliged (*based on scope and NRL)	Labs providing expl. for non-participation	Obliged labs non particip. w/o giving expl.	Finally considered to be obliged	Registered for Participation and submitted results obliged + [on voluntary basis]		Notes
					All	NRL-SRMs	
AT	1	0	0	1	1	1	
BE	5	1	0	4	4 + [2]	1	
BE; DE	1	0	0	1	1		
BE; FR; LU	1	0	0	1	1		
BE; NL	1	0	0	1	1		
BG	2	1	0	1	1	1	
CY	2	1	0	1	1	1	
CZ	3	0	0	3	3	1	
DE	24	3	3	21	18 + [2]	1	
DK	2	0	1	2	1	1	
EE	3	0	1	3	2	1	
ES	24	7	5	17	12 + [2]	2	
FI	2	0	0	2	2	2	
FR	4	0	1	4	3 + [5]	1	
GR	3	0	1	3	2	2	
HR	7	2	1	5	4 + [2]	1	
HU	4	2	0	2	2	1	
IE	1	0	0	1	1	1	
IT	26	6	9	20	11	1	
LT	1	0	0	1	1	1	
LU	1	0	0	1	1	1	
LV	1	0	0	1	1	1	
MT	2	1	0	1	1		MT: no subcontracting proxy NRL-SRM, one lab in DE with two subsidiaries was subcontracted for routine controls
NL	1	0	0	1	1	1	
PL	10	7	2	3	1 + [5]	1	
PT	2	1	0	1	1	1	
RO	5	1	3	4	1	1	
RO; AL	1	0	1	1			
SE	2	1	0	1	1		
SI	2	0	1	2	1	1	
SK	2	1	0	1	1	1	
SK; AL	1	1	0	0			
EU Total	147	36	29	111	**82 + [18]**	**28**	
EFTA: NRLs and OfLs							
CH					[2]	0	
NO	3	1	1	2	1	1	
EU/EFTA OfLs Total	150	37	30	113	**83 + [20]**	**29**	

Table 3-1 (cont.): Number of laboratories listed as being obliged to participate in the EUPT-SRM16, labs that registered to participate, and labs that finally submitted results (grouped by contracting country)

Countries outside Europa							
					Registered for Participation obliged + [on voluntary basis]		
Contracting Country [1)]	Labs originally considered to be obliged (*based on scope and NRL)	Labs providing expl. for non-participation	Obliged labs non particip. w/o giving expl.	Finally considered to be obliged	All	NRL-SRMs	Notes
Countries outside Europa							
IN					8		
KR					2		
RS					1		
TH					1		
UK					1		
Countries outside Europa Total					**13**		
Commercial Laboratories from EU or EFTA Countires							
CE					1		
CY					1		
DE					4		
IT					4		
ES					2		
EU/EFTA Commercial Laboratories Total					**12**		

4. RESULTS

4.1 Overview of Results

An overview of the percentage of laboratories having targeted each of the analytes present in the Target Pesticides List is shown in **Table 4-1**. **Table 4-2 (p. 21)** gives an overview of all results submitted by each laboratory. The individual numerical results reported by the laboratories are shown in **Table 4-8 (p. 21)**.

Table 4-1: Percentage of EU and EFTA 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 = 83 [2]	Based on n = 113	No. [1]	Based on n = 103 [2]	Based on n = 113
Compulsory Compounds	**Bromide**	**Yes**	47	57 %	42 %	56	54 %	50 %
	Chlormequat-Chloride	No	73	88 %	65 %	88	85 %	78 %
	Ethephon	**Yes**	61	73 %	54 %	76	74 %	67 %
	Fosetyl	No	55	66 %	49 %	70	68 %	62 %
	Glufosinate	**Yes**	60	72 %	53 %	72	70 %	64 %
	Glyphosate	**Yes**	71	86 %	63 %	88	85 %	78 %
	Mepiquat-Chloride	No	72	87 %	64 %	86	83 %	76 %
	MPP (= MPPA)	No	46	55 %	41 %	56	54 %	50 %
	N-Acetyl glufosinate	No	43	52 %	38 %	53	51 %	47 %
	Phosphonic acid	**Yes**	51	61 %	45 %	65	63 %	58 %
Optional Compounds	**2-CE**	**Yes**	25	30 %	22 %	32	31 %	28 %
	EO	No	16	19 %	14 %	19	18 %	17 %
	EO (sum)	**Yes**	25	30 %	22 %	32	31 %	28 %
	AMPA	No	52	63 %	46 %	67	65 %	59 %
	Chlorate	**Yes**	43	52 %	38 %	57	55 %	50 %
	Diquat	**Yes**	25	30 %	22 %	33	32 %	29 %
	Matrine	**Yes**	26	31 %	23 %	34	33 %	30 %
	N-Acetyl glyphosate	No	32	39 %	28 %	38	37 %	34 %
	Nicotine	**Yes**	27	33 %	24 %	36	35 %	32 %
	Paraquat	**Yes**	25	30 %	22 %	30	29 %	27 %
	Trimesium	**Yes**	15	18 %	13 %	24	23 %	21 %

1) Laboratories representing more than one country were counted only once.
2) 103 OfLs from EU and EFTA countries (incl. NRLs) have submitted at least one result, among them 83 laboratories were obliged to participate in this PT and 20 participated on voluntary basis.
3) 113 OfLs (including NRLs) from EU and EFTA countries were finally considered as obliged to participate in the EUPT-SRM16 (taking into account any explanations for non-participation).

Table 4-2: Scope and categorization of participating laboratories (including third country laboratories and commercial laboratories)

			Compulsory Compounds										
Compounds listed on the Target List			Bromide	Chlormequat-Chloride	Ethephon	Fosetyl	Glufosinate	Glyphosate	Mepiquat-Chloride	MPP (= MPPA)	N-Acetyl glufosinate	Phosphonic acid	Compulsory Compounds analysed / correctly found (max: 10 / 5)
within MACP			MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	
present in Test Item			Yes		Yes		Yes	Yes				Yes	
evaluated in this PT			Yes	No	Yes	No	Yes	Yes	No	No	No	Yes	
Lab-Code SRM16-	NRL	Cat.											
3		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
4		A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
5	x	B	V	ND	V	ND	V	V	ND			V	8 / 5
6	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
7		B		ND	V	ND	V	V	ND			V	7 / 4
8		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
9		B											0 / 0
10	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
11	x	B	V	ND	V		V	V	ND	ND			7 / 4
12		B			V								1 / 1
13		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
14		B	V	ND	**FN**		V	V	ND	ND	ND		8 / 3
15		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
16		B		ND	V	ND		V	ND			V	6 / 3
17		B	V	ND	V	ND	V	V	ND			V	8 / 5
18		B			V	ND	V	V		ND	ND	V	7 / 4
19	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
20	x	B		ND	V		V	V	ND	ND	ND		7 / 3
21	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
22		B	V	ND	V	ND		V	ND			V	7 / 4
23	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
24		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
25		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
26		A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
27		B		ND	V	ND	V	V	ND		ND	**FN**	8 / 3
28		B	**FN**	ND	V	ND	V	V	ND			V	8 / 4
29		B					V						1 / 1
30		B						V					1 / 1
31		B		ND					ND				2 / 0
32		B		**FP**				V					2 / 1
33	x	B		ND	V	ND		V	ND			V	6 / 3
35		A	V	ND	V	ND	V	V	ND	ND	ND		9 / 4

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

			Optional Compounds												Total
Compounds listed on the Target List			2-CE	EO	EO (sum)	AMPA	Chlorate	Diquat	Matrine	N-Acetyl glyphosate	Nicotine	Paraquat	Trimesium	Optional Compounds analysed / correctly found (max: 11 / 8)	Total analysed / correctly found (max: 21 / 13)
within MACP			actual concern	actual concern	actual concern	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD		
present in Test Item			Yes		Yes		Yes	Yes	Yes		Yes	Yes	Yes		
evaluated in this PT			Yes	No	Yes	No	Yes	Yes	Yes	No	Yes	Yes	Yes		
Lab-Code SRM16-	NRL	Cat.													
3		A				*(FP)*			V					2 / 1	12 / 6
4		A				ND	V	V						3 / 2	12 / 6
5	x	B				ND								1 / 0	9 / 5
6	x	A	V	ND	V		V			ND	V		Yes	7 / 4	17 / 9
7		B	**V***		V		V		V					4 / 3	11 / 7
8		A				ND	V	V	V	ND	V		**FN**	7 / 4	17 / 9
9		B					V							1 / 1	1 / 1
10	x	A	V	ND	V	ND	V	V		ND		V		8 / 5	18 / 10
11	x	B				ND					V			2 / 1	9 / 5
12		B												0 / 0	1 / 1
13		A				ND	V	V	V					4 / 3	14 / 8
14		B				ND		V	**FN**	ND		V		5 / 2	13 / 5
15		A				ND	V			ND				3 / 1	13 / 6
16		B				ND	V							2 / 1	8 / 4
17		B	V	ND	V		V		V		V		Yes	7 / 5	15 / 10
18		B	V	ND	V	ND				ND				5 / 2	12 / 6
19	x	A	V	ND	V	ND	V	V	V	ND	V	V	**FN**	11 / 7	21 / 12
20	x	B				ND					V			2 / 1	9 / 4
21	x	A	V		V	ND	V	V	V	ND	V	V	Yes	10 / 7	20 / 12
22		B	**FN**	ND	**FN**	ND	V							5 / 1	12 / 5
23	x	A				ND	V	V	V	ND	V	V	Yes	8 / 5	18 / 10
24		A				ND	V			ND				3 / 1	13 / 6
25		A				ND	V				V		Yes	4 / 2	14 / 7
26		A	**V***		V	ND	V	V				V		6 / 4	15 / 8
27		B	**V***		V	ND				ND				4 / 1	12 / 4
28		B				ND	V	V	V		V	V		6 / 5	14 / 9
29		B												0 / 0	1 / 1
30		B				ND								1 / 0	2 / 1
31		B												0 / 0	2 / 0
32		B	**V***		V									2 / 1	4 / 2
33	x	B					V							1 / 1	7 / 4
35		A												0 / 0	9 / 4

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

			Compulsory Compounds										
Compounds listed on the Target List			Bromide	Chlormequat-Chloride	Ethephon	Fosetyl	Glufosinate	Glyphosate	Mepiquat-Chloride	MPP (= MPPA)	N-Acetyl glufosinate	Phosphonic acid	Compulsory Compounds analysed / correctly found (max: 10 / 5)
within MACP			MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	
present in Test Item			Yes		Yes		Yes	Yes				Yes	
evaluated in this PT			Yes	No	Yes	No	Yes	Yes	No	No	No	Yes	
Lab-Code SRM16-	NRL	Cat.											
36	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
37		B		ND					ND				2 / 0
38		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
39		B					V	V		ND			3 / 2
41		A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
42		B	V			ND	V	V		ND	ND	V	7 / 4
43		B	V	ND	V	ND		V	ND			V	7 / 4
44		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
46		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
48	x	B		ND			V	V	ND		ND		5 / 2
49	x	B	V	ND	V			V	ND				5 / 3
50		B						V					1 / 1
51		B						V					1 / 1
52	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
55	x	B		ND					ND				2 / 0
56		B		ND	V		V	V	ND	ND	ND		7 / 3
57	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
59	x	A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
60		A	V	ND	V	ND	V	V	ND	ND		V	9 / 5
61		B		ND	V	ND		V					4 / 2
62		B		ND					ND				2 / 0
63	x	B		ND	V	**FP**	V	V	ND		ND	V	8 / 4
64	x	A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
65		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
66		B		ND				V	ND				3 / 1
68		A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
69		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
70	x	B		ND	V				ND				3 / 1
71		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
72		B		ND				V	ND				3 / 1
73	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
74		B		ND					ND				2 / 0

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

			Optional Compounds													Total
Compounds listed on the Target List			2-CE	EO	EO (sum)	AMPA	Chlorate	Diquat	Matrine	N-Acetyl glyphosate	Nicotine	Paraquat	Trimesium	Optional Compounds analysed / correctly found (max: 11 / 8)	Total analysed / correctly found (max: 21 / 13)
within MACP			actual concern	actual concern	actual concern	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD		
present in Test Item			Yes		Yes		Yes	Yes	Yes		Yes	Yes	Yes		
evaluated in this PT			Yes	No	Yes	No	Yes	Yes	Yes	No	Yes	Yes	Yes		
Lab-Code SRM16-	NRL	Cat.													
36	x	A				ND	V			ND				3 / 1	13 / 6
37		B												0 / 0	2 / 0
38		A	**V***		V		V		V	ND	V		Yes	7 / 4	17 / 9
39		B				ND								1 / 0	4 / 2
41		A				ND	V		V	ND				4 / 2	13 / 6
42		B					V							1 / 1	8 / 5
43		B				ND	V	V	V		V	V	Yes	7 / 5	14 / 9
44		A	V	ND	V	ND	V	V	V	ND	V	V	Yes	11 / 7	21 / 12
46		A				ND	V	V	V	ND	V	V	Yes	8 / 5	18 / 10
48	x	B				ND				ND				2 / 0	7 / 2
49	x	B				ND								1 / 0	6 / 3
50		B				ND								1 / 0	2 / 1
51		B												0 / 0	1 / 1
52	x	A				ND				ND				2 / 0	12 / 5
55	x	B						V				V		2 / 2	4 / 2
56		B				ND	V							2 / 1	9 / 4
57	x	A	V		**V***	ND		V				V		5 / 3	15 / 8
59	x	A				ND		V						2 / 1	11 / 5
60		A	V	ND	V		V						Yes	5 / 3	14 / 8
61		B											Yes	1 / 0	5 / 2
62		B												0 / 0	2 / 0
63	x	B				**FP**	V	V		ND		V		5 / 3	13 / 7
64	x	A				ND		V				V		3 / 2	12 / 6
65		A				ND	V			ND				3 / 1	13 / 6
66		B				ND					V			2 / 1	5 / 2
68		A	V	ND	V	ND	V	V	V	ND	V	V	Yes	11 / 7	20 / 11
69		A				ND	V	V	V	ND	V	V		7 / 5	17 / 10
70	x	B												0 / 0	3 / 1
71		A	V	ND	**V***	ND	V		V		V		Yes	8 / 4	18 / 9
72		B									V			1 / 1	4 / 2
73	x	A				ND	V		V	ND			Yes	5 / 2	15 / 7
74		B												0 / 0	2 / 0

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

			Compulsory Compounds										
Compounds listed on the Target List			Bromide	Chlormequat-Chloride	Ethephon	Fosetyl	Glufosinate	Glyphosate	Mepiquat-Chloride	MPP (= MPPA)	N-Acetyl glufosinate	Phosphonic acid	Compulsory Compounds analysed / correctly found (max: 10 / 5)
within MACP			MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	
present in Test Item			Yes		Yes		Yes	Yes				Yes	
evaluated in this PT			Yes	No	Yes	No	Yes	Yes	No	No	No	Yes	
Lab-Code SRM16-	NRL	Cat.											
75		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
76	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
78		B		ND					ND				2 / 0
79		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
80		B		ND				V					2 / 1
82		B		ND			V	V	ND	ND			5 / 2
83	x	A	V	ND	V	ND	V	V	ND	ND		V	9 / 5
84	x	A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
86	x	B		ND				**FN**	ND				3 / 0
87		B	V	ND	V	ND		V	ND			V	7 / 4
88		A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
89	x	B		ND					ND				2 / 0
90		B	V	ND	V	ND		V	ND			V	7 / 4
92		A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
93		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
94		B	V	ND			V	V	ND				5 / 3
95	x	B	V										1 / 1
96		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
97		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
99		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
101		B											0 / 0
102		B	V	ND	V	ND	V	V	ND			V	8 / 5
103	x	B	V	ND	V	ND	V	V	ND				7 / 4
104		B		ND		ND	V	V	ND			**FN**	6 / 2
105		A	V	ND	V	ND	V	**FN**	ND	ND	ND	V	10 / 4
107		A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
109		B											0 / 0
111		B			V	ND	V	V					4 / 3
112		B	V	ND	V		V	V	ND			V	7 / 5
113		A		ND	V	ND	V	V	ND	ND	ND	V	9 / 4
114		B	V	ND	V	ND	V	V	ND			V	8 / 5
115		B	V		V	ND	V	V	ND			V	7 / 5
116		B			V								1 / 1

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

			Optional Compounds												Total
Compounds listed on the Target List			2-CE	EO	EO (sum)	AMPA	Chlorate	Diquat	Matrine	N-Acetyl glyphosate	Nicotine	Paraquat	Trimesium	Optional Compounds analysed / correctly found (max: 11 / 8)	Total analysed / correctly found (max: 21 / 13)
within MACP			actual concern	actual concern	actual concern	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD		
present in Test Item			Yes		Yes		Yes	Yes	Yes		Yes	Yes	Yes		
evaluated in this PT			Yes	No	Yes	No	Yes	Yes	Yes	No	Yes	Yes	Yes		
Lab-Code SRM16-	NRL	Cat.													
75		A	V	ND	V	ND	V		V	ND	V		Yes	9 / 5	19 / 10
76	x	A												0 / 0	10 / 5
78		B					V							1 / 1	3 / 1
79		A	**V***		V	ND	V	V	V		V	V		8 / 6	18 / 11
80		B												0 / 0	2 / 1
82		B				ND	V							2 / 1	7 / 3
83	x	A				ND	V	V		ND		V	Yes	6 / 3	15 / 8
84	x	A												0 / 0	10 / 5
86	x	B									V			1 / 1	4 / 1
87		B	V	ND	V	ND	V	V	V	ND		V	Yes	10 / 6	17 / 10
88		A	V	**FP**	V	ND	V	V				V		7 / 5	16 / 9
89	x	B												0 / 0	2 / 0
90		B				ND		V	V		V	V		5 / 4	12 / 8
92		A				ND		V		ND		V		4 / 2	13 / 6
93		A				ND	V			ND	V		Yes	5 / 2	15 / 7
94		B	**V***		V	ND		V				V		5 / 3	10 / 6
95	x	B												0 / 0	1 / 1
96		A	**V***		V	ND	V	V	V	ND	V	V		9 / 6	19 / 11
97		A	V	ND	V		V		V	ND	V			7 / 5	17 / 10
99		A				ND	V			ND				3 / 1	13 / 6
101		B										V		1 / 1	1 / 1
102		B				ND	V		V		V			4 / 3	12 / 8
103	x	B				ND				ND				2 / 0	9 / 4
104		B				ND							Yes	2 / 0	8 / 2
105		A					V		V	ND				3 / 2	13 / 6
107		A				ND	V	V	V	ND	V	V	Yes	8 / 5	17 / 9
109		B	V	**FP**	V									3 / 2	3 / 2
111		B				ND	V							2 / 1	6 / 4
112		B	V	ND	V		V				V			5 / 4	12 / 9
113		A				ND	V	V	V			V	Yes	6 / 4	15 / 8
114		B	V	ND	V	ND	V		V		V			7 / 5	15 / 10
115		B	V		**V***	ND	V		V	ND	V			7 / 4	14 / 9
116		B												0 / 0	1 / 1

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

			Compulsory Compounds										
Compounds listed on the Target List			Bromide	Chlormequat-Chloride	Ethephon	Fosetyl	Glufosinate	Glyphosate	Mepiquat-Chloride	MPP (= MPPA)	N-Acetyl glufosinate	Phosphonic acid	Compulsory Compounds analysed / correctly found (max: 10 / 5)
within MACP			MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	MACP	
present in Test Item			Yes		Yes		Yes	Yes				Yes	
evaluated in this PT			Yes	No	Yes	No	Yes	Yes	No	No	No	Yes	
Lab-Code SRM16-	NRL	Cat.											
118	x	A	V	ND	V	ND	V	V	ND	ND	ND	FN	10 / 4
120		B	FN	ND	V	ND	V	V	ND	FP		V	9 / 4
121	x	A	V	ND	V	ND	V	V	ND	ND	ND		9 / 4
123		B		ND		ND		V	ND				4 / 1
124		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
126		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
3rd-34		B	FN	ND	V	ND	V	V	ND	FP	ND	V	9 / 3
Privat-40		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
3rd-45		A	V	ND	V	ND	V	V	ND	ND	ND		10 / 5
Privat-47		A	V	ND	V	ND	V	V	ND	ND	ND	FN	10 / 5
Privat-53		A	V	ND	V	ND	V	V	ND	ND	ND		10 / 5
Privat-54		A	V	ND	V	ND	V	V	ND	ND	ND	FN	10 / 5
Privat-58		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
Privat-77		A	V	ND	V	ND	V	V	ND	ND	ND		10 / 5
3rd-85		B		ND		ND	V	V	ND	ND	ND		7 / 2
3rd-91		A	V	ND	V	ND	V	V	ND	ND	ND	V	10 / 5
3rd-98		B	V	ND		ND	V	V	ND	ND	ND		8 / 3
3rd-100		A	V	ND	V	ND	V	V	ND		ND	V	9 / 5
3rd-106		B	V	ND	V	ND	V	V	ND				8 / 5
3rd-108		B	V					V					2 / 2
Privat-110		B										V	0 / 0
Privat-117		B		ND		ND		V	ND				5 / 2
Privat-119		B											0 / 0
Privat-122		B											0 / 0
Privat-125		B	V	ND	V		V	V	ND			V	6 / 4
Privat-127		B	V	ND	V	ND		V	ND			V	7 / 4
3rd-128		B	V	ND	FN	ND	V	V	ND	ND	ND	V	10 / 3
3rd-129		B	V	ND		ND	V	V	ND			V	6 / 3
3rd-130		B		ND	FN	ND		V	ND	ND		V	7 / 1
3rd-131		B	V	ND	V	ND	V	V	ND			V	8 / 5
3rd-132		B										V	0 / 0

MACP-Reg.: REGULATION (EU) 2020/585 of 27 April 2020; **WD:** Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 23–24 November 2020 rev. 12(2)

V = analysed for and submitted concentration Value > "MRRL" for a pesticide present in the test item; **V***: value of EO (sum) or 2-CE calculated by the organizers using the reported results of 2-CE or EO (sum), repectively; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FP** = false positive result; *(FP)* = Result reported as "≤ MRRL", therefore, not regarded as FP

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

			Optional Compounds												Total
Compounds listed on the Target List			2-CE	EO	EO (sum)	AMPA	Chlorate	Diquat	Matrine	N-Acetyl glyphosate	Nicotine	Paraquat	Trimesium	Optional Compounds analysed / correctly found (max: 11 / 8)	Total analysed / correctly found (max: 21 / 13)
within MACP			actual concern	actual concern	actual concern	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD	Moni-WD		
present in Test Item			Yes		Yes		Yes	Yes	Yes		Yes	Yes	Yes		
evaluated in this PT			Yes	No	Yes	No	Yes	Yes	Yes	No	Yes	Yes	Yes		
Lab-Code SRM16-	NRL	Cat.													
118	x	A	V	ND	V	ND	V	V	V	ND	V	V		10 / 7	20 / 11
120		B	V	ND	V	ND				ND	V			6 / 3	15 / 7
121	x	A	V		**V***									2 / 1	11 / 5
123		B					V				V			2 / 2	6 / 3
124		A				ND	V	V	V		V	V		6 / 5	16 / 10
126		A	V		V	ND	V	V	V	ND	V	V	Yes	10 / 7	20 / 12
3rd-34		B												0 / 0	9 / 3
Privat-40		A	**V***		V	ND	V	V	V	ND	V	V	Yes	10 / 6	20 / 11
3rd-45		A	V	ND	**V***		V		V	ND				6 / 3	16 / 8
Privat-47		A				ND	V	V	V	ND	V	V		7 / 5	17 / 10
Privat-53		A				ND	V	V		ND		V		5 / 3	15 / 8
Privat-54		A	V	ND	V	ND	V	V	V	ND	V	V	Yes	11 / 7	21 / 12
Privat-58		A	V	ND	V	ND	V	V	V	ND	V	V	Yes	11 / 7	21 / 12
Privat-77		A	V	**FP**	V		V		V		V			6 / 5	16 / 10
3rd-85		B	**V***		V	ND								3 / 1	10 / 3
3rd-91		A	V	ND	V								Yes	4 / 2	14 / 7
3rd-98		B	**V***		V	ND								3 / 1	11 / 4
3rd-100		A	V	ND	V	ND		V		ND	V	**FN**		8 / 4	17 / 9
3rd-106		B	V	ND	V		V	V	V			V		7 / 6	15 / 11
3rd-108		B				ND		V				V		3 / 2	5 / 4
Privat-110		B	V		V									2 / 2	2 / 2
Privat-117		B	V	ND	V	ND	V		V					6 / 4	11 / 6
Privat-119		B	**V***		V									2 / 1	2 / 1
Privat-122		B	**V***		V									2 / 1	2 / 1
privat-125		B	**V***		V	ND	V				V			5 / 3	11 / 7
Privat-127		B	**V***		V		V		V		V			5 / 4	12 / 8
3rd-128		B												0 / 0	10 / 3
3rd-129		B	V	ND	V	ND					V			5 / 3	11 / 6
3rd-130		B	**V***		V	ND		**FN**				**FN**		5 / 1	12 / 2
3rd-131		B	V	ND	V	ND		V			V	V		7 / 5	15 / 10
3rd-132		B	V	ND	V									3 / 2	3 / 2

MACP-Reg.: REGULATION (EU) 2020/585 of 27 April 2020; **WD:** Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 23–24 November 2020 rev. 12(2)

V = analysed for and submitted concentration Value > "MRRL" for a pesticide present in the test item; **V***: value of EO (sum) or 2-CE calculated by the organizers using the reported results of 2-CE or EO ***(sum)***, repectively; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FP** = false positive result; *(FP)* = Result reported as "≤ MRRL", therefore, not regarded as FP

4.2 Assigned Values and Target Standard Deviations

The assigned value (x_{pt}) of each analyte present in the test item was established as the mean of robust statistics (x^*) of the numerical results received, which was calculated using Algorithm A ([**6**] and **Appendix 8**) after the exclusion of obvious outliers. In the case of ***2-CE*** and ***EO (sum)*** results received from all participants were considered in the assigned value calculation (**Section 4.2.1, p. 29**). For all other analytes only the results received from OfLs based in EU and EFTA countries were considered, disregarding results from laboratories from 3rd countries and EU Candidate Countries as well as results from commercial laboratories based in the EU, that are not dessignated as OfLs.

Before setting the assigned values for each analyte, the results were checked for outliers based on the distance to the robust mean of the entire relevant population (z-score >5) or if detected as outliers by the Grubbs' test ($\alpha = 0.05$). These outliers were excluded from the population for establishment of assigned values. Following exclusion of outliers the robust mean of each analyte was calculated again using the remaining results untill the robust mean could be calculated and established as the assigned value.

The uncertainties of the assigned values ($u(x_{pt})$) were calculated as described under **Section 2.2 (p. 11)**.

The assigned values, their uncertainties and the robust relative standard deviations (CV^*) of all analytes present in the PT are shown in **Table 4-3**. Except ***2-CE*** (30.4 %), ***EO (sum)*** (29.5 %), ***matrine*** (26.5 %) and ***nicotine*** (27.5 %) the CV^*-values of all other analytes were at or lower than 25 % (FFP-RSD). The average CV^*s of all compulsory analytes was 20.3 % and for the optional compounds 20.3 %. The narrowest result distributions were obtained in the case of ***glufosinate*** ($CV^* = 17.1$ %) and ***trimesium*** (14.8 %).

Table 4-3: Assigned values, uncertainties of assigned values and CV^* values calculated for all compounds present in the test item

Assigned Value and CV^* Based on the Entire Population of Results from EU and EFTA Laboratories									
	Compound	No. of FNs | Outlier	No. of numerical results (EU+EFTA)	Result Population for AV [1] | n=	Assigned Value [mg/kg]	$u(x_{pt})$ [2] [mg/kg]	$u(x_{pt})$ Tolerance [mg/kg]	Judgement for UAV-test	CV^* [3] [%]
Compulsory Compounds	Bromide	2|1	54	OfLs (EU/EFTA) | 53	21.3	± 0.7579	1.5975	passed	20.7
	Ethephon	1|3	75	OfLs (EU/EFTA) | 72	0.228	± 0.0071	0.0171	passed	21.1
	Glufosinate	0|4	72	OfLs (EU/EFTA) | 68	0.216	± 0.0056	0.0162	passed	17.1
	Glyphosate	2|1	86	OfLs (EU/EFTA) | 85	0.510	± 0.0128	0.0383	passed	18.5
	Phosphonic acid	3|0	62	OfLs (EU/EFTA) | 62	0.676	± 0.0261	0.0507	passed	24.3
	Average [4] CV^*								**20.3**
Optional Compounds	2-CE*	0|1	46	all (-) | 45	4.59	± 0.2598	0.3443	passed	30.4
	EO (sum)*	0|2	46	all (-) | 44	2.50	± 0.1388	0.1875	passed	29.5
	Chlorate	0|0	57	OfLs (EU/EFTA) | 57	1.03	± 0.0353	0.0775	passed	20.7
	Diquat	0|2	33	OfLs (EU/EFTA) | 31	0.244	± 0.0136	0.0183	passed	24.7
	Matrine	1|3	33	OfLs (EU/EFTA) | 30	0.124	± 0.0075	0.0093	passed	26.5
	Nicotine	0|0	36	OfLs (EU/EFTA) | 36	0.255	± 0.0146	0.0191	passed	27.5
	Paraquat	0|0	30	OfLs (EU/EFTA) | 30	0.264	± 0.0150	0.0198	passed	25.0
	Trimesium	2|1	22	OfLs (EU/EFTA) | 21	0.073	± 0.0029	0.0055	passed	14.8
	Average [4] CV^*								**24.9**

1: OfLs (EU/EFTA): The population consisted of numerical results reported by the OfLs form EU or EFTA countries, excl. outliers;
all (-): The population consisted of numerical results reported by all participants, excl. outliers and those 6 results derived from sample extration without water addition in the extraction solvent.
2: $u(x_{pt})$: Uncertainty of assigned value calculated as shown under **Section 2.2 (p. 38)**
3: CV^*: Relative standard deviation based on robust statistics
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.

4.2.1 Evaluation of results for 2-CE and EO (sum)

Different analytical approaches were employed by the participants for the analysis of ***2-CE*** and ***EO (sum)***:

a) ***EO (sum)*** only
b) ***EO (sum)*** and EO (separately)
c) ***2-CE*** only
d) ***2-CE*** and EO (separately)

As the test item did not contain any measurable residues of EO as such, the organizers assumed that any results of ***EO (sum)*** or ***2-CE*** should be interconvertible by applying the molecular weight factor. Part of the labs proceeded with conversion on their own while others did not. In order to obtain a more uniform set of data, the organizers decided to mathematically convert ***EO (sum)*** results to ***2-CE*** (16 cases) and ***2-CE*** results to ***EO (sum)*** (5 cases), as long as this had not been done by the participants allready.

Irrespective on whether the abovementioned back-calculated levels were included in the population or not, the distribution of the participants' results remained quite broad both for ***2-CE*** and ***EO (sum)***. The inexperience of many labs with the analysis of ***2-CE*** and ***EO (sum)*** was surely one of the reasons for this wide scattering, but differences in the methods applied also had an effect. For example, 6 laboratories did not add water to the extraction solvent (acetonitrile) that resulted in insufficient soaking of the material and, therefore, to poor extraction yields, even at prolongued extraction times. One of them has even not detected ***2-CE*** or EO in the test material and obtaiened FNs for these two compounds. These laboratories were contacted by phone to confirm their procedure. In addition, the EURL-SRM confirmed the poor extractability of ***2-CE*** in the absence of water by own experiments. Excluding this sub-population of results, markedly narrowed the result distribution and the robust standard deviation. For the preliminary report released on 18 May, the preliminary assigned values for ***EO (sum)*** and ***2-CE*** were calculated based on the results of 22 laboratories that had previously participated in the ad-hoc EO-PT held in December 2020 as these laboratories had the possibility to gain more experience with their methods for the analysis of ***EO (sum)*** and ***2-CE***. None of these labs had extracted the material without water addition. After consultations with the Scientific Committee on the Evaluation Meeting it was decided to use all reported results, excluding the outliers and the results reported by the 6 laboratories that hadn't added water during the extraction step. The final assigned value of ***2-CE*** and ***EO (sum)*** are slightly lower than those in the Preliminary Report (**Table 4-4**), so that the participants z-scores only shifted slightly compared to the preliminary report.

For details please see also **Section 4.5 „Method-based Evaluation" (p. 47) .**

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

	2-CE			EO (sum)		
Population [1)]	**all**	**all** [(-)] (Final Decision)	**ad-hoc** (in Pre. Report)	**all**	**all** [(-)] (Final Decision)	**ad-hoc** (in Pre. Report)
No. Numerical Results	51	46	22	51	46	22
No. FNs	1	0	0	1	0	0
No. Outliers	1	1	0	2	2	0
AV	4.29	4,59	4,66	2.33	2.50	2.55
CV*	39.1 %	30.4 %	27.3 %	38.2 %	29.5 %	27.7
Uncertainty of AV	0.29655	0.25983	0.33866	0.15926	0.13878	0.18860
Tolerance	0.3218	0.3443	0.3500	0.1750	0.1880	0.1913

Definition of the populations:
all: all results submitted; **all** [(-)]: all results excl. 6 derived from methods not involving water addion during the extration step; **ad-hoc**: results reported by the laboratories that had previously participated in the ad-hoc PT on EO and 2-CE in sesame held in Dec. 2020

4.3 Assessment of Laboratory Performance

4.3.1 False Positives

Five EU/EFTA OfLs reported in six cases numerical results for compounds that were neither spiked to the test material nor detected by the organizer and the overwhelming majority of the participants. These cases concerned twice *EO* and one time each *chlormequat-chloride, MPP, AMPA* and *fosetyl.* In the case of *AMPA*, the participatng lab (LabCode 3) submitted a numerical result lower than MRRL and its own RL. This result was therefore not judged as a FP. In addition, one 3rd country laboratory and one commercial laboratory submitted a numerical result for *AMPA* and *MPP*, respectively. Those were also judged as FPs (**Table 4-5**).

4.3.2 False Negatives

Eleven EU/EFTA OfLs reported in 13 cases that they have analysed for certain target compounds present in the test item, but without detecting them. They concerned 8 compulsory compounds (3× ***phosphonic acid***, each 2× ***bromide*** and ***glyphosate*** as well as 1× ***ethephon***) and 5 optional ones (2× ***trimesium***, each 1× ***2-CE***, ***EO (sum)*** and ***matrine***). Since those compounds were either applied during the cultivation or spiked to the test mterial and detected by the majority of the laboratories targeting them, these reported results were judged as FNs (**Table 4-6, p. 31**). These false negative results accounted for 1.6 % of the total 515 results reported by the EU/EFTA laboratories for compulsory target compounds and for 0.6 % of the 824 results reported by these labs in total.

In addtion, 4 labs from 3rd countries reported in 8 cases results that were also judged as FNs. These concerned ***ethephon, phosphonic acid*** and ***paraquat*** (2× each) as well as ***bromide*** and ***diquat*** (1× each) (**Table 4-6, p. 31**).

The reasons reported by the laboratories for the false negartive or false positive results are compiled in **Appendix 7**.

4.3.3 Laboratory Performance Based on z-Scores

Individual z-scores were calculated using the FFP-RSD of 25 % and the assigned values (AVs). The AVs were derived from the robust mean values of the entire population of results submitted by the EU/EFTA OfLs with the exception of ***2-CE*** and ***EO (sum)***, where the results of all participanting laboratories were consid-

Table 4-5: False positive results reported in EUPT-SRM16

Compound		PT-Code	Analysed	Reported Result [mg/kg]	RL [mg/kg]	MRRL [mg/kg]	Judgement
Compulsory	Chlormequat-Cloride	SRM16-32	Yes	0.012	0.01	0.01	False Positive
	MPP (= MPPA)	SRM16-120	Yes	0.031	0.02	0.02	False Positive
		SRM16-3rd-34	Yes	0.111	0.01	0.02	False Positive
Optional	EO	SRM16-88	Yes	0.346	0.025	0.02	False Positive
		SRM16-109	Yes	0.981	0.005	0.02	False Positive
		SRM16-Privat-77	Yes	0.180	0.02	0.02	False Positive
	AMPA	SRM16-3	Yes	0.0631	0.1	0.1	Not False Positive
		SRM16-63	Yes	0.186	0.1	0.1	False Positive
	Fosetyl	SRM16-63	Yes	0.042	0.01	0.02	False Positive

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

	Compounds	PT-Code	Analysed	Detected	RL [mg/kg]	MRRL [mg/kg]	Assigned Value [mg/kg]	Judgement
Compulsory	Bromide	SRM16-28	Yes	No	2	2	21.3	False Negative
		SRM16-120	Yes	No	9.9	2	21.3	False Negative
		SRM16-3rd-34	Yes	No	0.5	2	21.3	False Negative
	Ethephon	SRM16-14	Yes	No	0.02	0.04	0.228	False Negative
		SRM16-3rd-128	Yes	No	0.02	0.04	0.228	False Negative
		SRM16-3rd-130	Yes	No	0.01	0.04	0.228	False Negative
	Glyphosate	SRM16-86	Yes	No	0.1	0.1	0.51	False Negative
		SRM16-105	Yes	No	0.1	0.1	0.51	False Negative
	Phosphonic acid	SRM16-27	Yes	No	0.1	0.1	0.676	False Negative
		SRM16-104	Yes	No	0.1	0.1	0.676	False Negative
		SRM16-118	Yes	No	0.1	0.1	0.676	False Negative
		SRM16-3rd-128	Yes	No	0.1	0.1	0.676	False Negative
		SRM16-3rd-130	Yes	No	0.1	0.1	0.676	False Negative
Optional	2-CE	SRM16-22	Yes	No	0.05	0.05	4.59	False Negative
	Diquat	SRM16-3rd-130	Yes	No	0.01	0.02	0.244	False Negative
	EO (sum)	SRM16-22	Yes	No	0.05	0.05	2.5	False Negative
	Matrine	SRM16-14	Yes	No	0.05	0.01	0.124	False Negative
	Paraquat	SRM16-3rd-100	Yes	No	0.02	0.02	0.264	False Negative
		SRM16-3rd-130	Yes	No	0.01	0.02	0.264	False Negative
	Trimesium	SRM16-8	Yes	No	0.01	0.01	0.073	False Negative
		SRM16-19	Yes	No	0.02	0.01	0.073	False Negative

ered. In each case outliers were excluded prior to the calculation, as described in **Section 4.2 (p. 28)**. **Table 4-7 (p. 32)** 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. 12).**

In summary, 85 % of the results reported by EU/EFTA-OfLs were within the "acceptable" z-scores. The share of acceptable results ranged from 72 % for ***2-CE*** and ***EO (sum)*** to 91 % for ***chlorate***. Among the compulsory compounds, "acceptable" z-scores were achieved by more than 85 % of the labs in all cases (on average 87 %). Among the optional compounds "acceptable" z-scores were achieved by 82 % of the labs on average. In the case of ***2-CE, EO (sum), diquat*** and ***fluazifop*** acceptable z-score were obtained by less than 80 % of the labs. In the case of ***paraquat, nicotine*** and ***trimesium*** the acceptable z-score rate ranged between 80 % and 90 %. In the case of ***chlorate*** the acceptable z-score rate was 91 %.

In the case of laboratories from EU Candidate or 3rd countries, 83 % and 88 % of the results could be classified as "acceptable" for the compulsory and optional compounds, respectively. In the case of the participating commercial laboratories the share of "acceptable" z-scores was 85 % for both the compulsory and optional compounds.

While only 72 % results submited by the EU/EFTA-OfLs for ***2-CE*** and ***EO (sum)*** could be classified as "acceptable", 90 % from EU Candidate/3rd countries and even 100 % from commercial laboratories achieved "acceptable" z-scores. This is surely related to the lack of experience with the analysis of these particular analytes in many of the OfLs.

A compilation of all individual results and z-scores for each laboratory is shown in **Table 4-8 (p. 33)** for compulsory compounds and **Table 4-9 (p. 36)** for optional compounds. The corresponding kernel density histograms showing the distribution of the reported results are shown in **Appendix 5**. A graphic representation of the z-score distribution of each target analyte present in the test item can be seen in **Appendix 6**.

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

EU and EFTA Official Laboratories						
	Compound	No. of results [1)]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1)] No. (%)	FNs
Compulsory Compounds	Bromide	56	49 (88 %)	4 (7 %)	3 (5 %)	2
	Ethephon	76	67 (88 %)	4 (5 %)	5 (7 %)	1
	Glufosinate	72	61 (85 %)	3 (4 %)	8 (11 %)	0
	Glyphosate	88	76 (86 %)	7 (8 %)	5 (6 %)	2
	Phosphonic acid	65	57 (88 %)	5 (8 %)	3 (5 %)	3
	Subtotal	**357**	**310 (87 %)**	**23 (6 %)**	**24 (7 %)**	**8**
Optional Compounds	2-CE	32	23 (72 %)	3 (9 %)	6 (19 %)	1
	EO (sum)	32	23 (72 %)	3 (9 %)	6 (19 %)	1
	Chlorate	57	52 (91 %)	3 (5 %)	2 (4 %)	0
	Diquat	33	26 (79 %)	2 (6 %)	5 (15 %)	0
	Matrine	34	27 (79 %)	2 (6 %)	5 (15 %)	1
	Nicotine	36	31 (86 %)	2 (6 %)	3 (8 %)	0
	Paraquat	30	24 (80 %)	1 (3 %)	5 (17 %)	0
	Trimesium	24	21 (88 %)	(0 %)	3 (13 %)	2
	Subtotal	**278**	**227 (82 %)**	**16 (6 %)**	**35 (13 %)**	**5**
Overall EU/EFTA		**635**	**537 (85 %)**	**39 (6 %)**	**59 (9 %)**	**13**
3rd Country / EU Candidate Country Laboratories						
	Compound	No. of results [1)]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1)] No. (%)	FNs
Compulsory Compounds	Bromide	10	9 (90 %)	(0 %)	1 (10 %)	1
	Ethephon	8	4 (50 %)	2 (25 %)	2 (25 %)	2
	Glufosinate	10	10 (100 %)	(0 %)	0 (0 %)	0
	Glyphosate	12	11 (92 %)	(0 %)	1 (8 %)	0
	Phosphonic acid	7	5 (71 %)	(0 %)	2 (29 %)	2
	Subtotal	**47**	**39 (83 %)**	**2 (4 %)**	**6 (13 %)**	**5**
Optional Compounds	2-CE	10	9 (90 %)	1 (10 %)	0 (0 %)	0
	EO (sum)	10	9 (90 %)	1 (10 %)	0 (0 %)	0
	Chlorate	3	3 (100 %)	(0 %)	0 (0 %)	0
	Diquat	6	5 (83 %)	(0 %)	1 (17 %)	1
	Matrine	3	3 (100 %)	(0 %)	0 (0 %)	0
	Nicotine	4	4 (100 %)	(0 %)	0 (0 %)	0
	Paraquat	6	4 (67 %)	(0 %)	2 (33 %)	2
	Trimesium	1	1 (100 %)	(0 %)	0 (0 %)	0
	Subtotal	**43**	**38 (88 %)**	**2 (5 %)**	**3 (7 %)**	**3**
Overall 3rd country / EU Candidate Country		**90**	**77 (86 %)**	**4 (4 %)**	**9 (10 %)**	**8**
Commercial Laboratories from EU/EFTA Countries (not designated as OfLs)						
	Compound	No. of results [1)]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1)] No. (%)	FNs
Compulsory Compounds	Bromide	8	6 (75 %)	(0 %)	2 (25 %)	2
	Ethephon	8	8 (100 %)	(0 %)	(0 %)	0
	Glufosinate	7	7 (100 %)	(0 %)	(0 %)	0
	Glyphosate	9	9 (100 %)	(0 %)	(0 %)	0
	Phosphonic acid	8	8 (100 %)	(0 %)	(0 %)	0
	Subtotal	**40**	**38 (95 %)**	**(0 %)**	**2 (5 %)**	**2**
Optional Compounds	2-CE	10	10 (100 %)	(0 %)	(0 %)	0
	EO (sum)	10	10 (100 %)	(0 %)	(0 %)	0
	Chlorate	9	9 (100 %)	(0 %)	(0 %)	0
	Diquat	5	5 (100 %)	(0 %)	(0 %)	0
	Matrine	7	5 (71 %)	1 (14 %)	1 (14 %)	1
	Nicotine	7	6 (86 %)	1 (14 %)	(0 %)	0
	Paraquat	5	5 (100 %)	(0 %)	(0 %)	0
	Trimesium	3	3 (100 %)	(0 %)	(0 %)	0
	Subtotal	**56**	**53 (95 %)**	**2 (4 %)**	**1 (2 %)**	**1**
Commercial Laboratories		**96**	**91 (95 %)**	**2 (2 %)**	**3 (3 %)**	**3**

1) including false negatives (FNs)

Table 4-8: Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compounds. Individual z-scores were calculated using the FFP-RSD of 25 % and the assigned values.

COMPULSORY Compounds				Bromide		Ethephon		Glufosinate		Glyphosate		Phosphonic acid	
Assigned Value [mg/kg]				21.3		0.228		0.216		0.510		0.676	
*CV**				20.7 %		21.1 %		17.1 %		18.5 %		24.3 %	
MRRL [mg/kg]				2		0.04		0.02		0.1		0.1	
Lab code SRM16-	NRL-SRM	Cat	Analysed / corr. found, max. 10 / 5	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score
3		A	10 / 5	18.0	-0.6	0.196	-0.6	0.177	-0.7	0.488	-0.2	0.487	-1.1
4		A	9 / 4			0.174	-1.0	0.178	-0.7	0.545	0.3	0.722	0.3
5	x	B	8 / 5	19.89	-0.3	0.276	0.8	0.237	0.4	0.533	0.2	0.771	0.6
6	x	A	10 / 5	19.6	-0.3	0.232	0.1	0.206	-0.2	0.488	-0.2	0.671	0.0
7		B	7 / 4			2.56	40.9	0.672	8.4	0.219	-2.3	0.705	0.2
8		A	10 / 5	31.4	1.9	0.079	-2.6	0.26	0.8	0.88	2.9	0.92	1.4
9		B	0 / 0										
10	x	A	10 / 5	15.4	-1.1	0.217	-0.2	0.185	-0.6	0.528	0.1	0.791	0.7
11	x	B	7 / 4	24.1	0.5	0.232	0.1	0.222	0.1	0.452	-0.5		
12		B	1 / 1			0.192	-0.6						
13		A	10 / 5	0.042	-4.0	0.219	-0.2	0.180	-0.7	0.745	1.8	0.923	1.5
14		B	8 / 3	24.4	0.6	FN	-3.7	0.557	6.3	0.201	-2.4		
15		A	10 / 5	14.3	-1.3	0.214	-0.3	0.238	0.4	0.768	2.0	0.681	0.0
16		B	6 / 3			0.10	-2.3			0.56	0.4	0.30	-2.2
17		B	8 / 5	20.9	-0.1	0.202	-0.5	0.251	0.7	0.424	-0.7	0.648	-0.2
18		B	7 / 4			0.232	0.1	0.201	-0.3	0.482	-0.2	0.635	-0.2
19	x	A	10 / 5	11.3	-1.9	0.197	-0.5	0.186	-0.6	0.434	-0.6	0.605	-0.4
20	x	B	7 / 3			0.267	0.7	0.215	0.0	0.497	-0.1		
21	x	A	10 / 5	25.1	0.7	0.301	1.3	0.199	-0.3	0.479	-0.2	0.921	1.5
22		B	7 / 4	13.220	-1.5	0.203	-0.4			0.511	0.0	0.696	0.1
23	x	A	10 / 5	12.7	-1.6	0.219	-0.2	0.227	0.2	0.573	0.5	0.825	0.9
24		A	10 / 5	18.3	-0.6	0.204	-0.4	0.221	0.1	0.413	-0.8	0.715	0.2
25		A	10 / 5	34.9	2.6	0.203	-0.4	0.200	-0.3	0.570	0.5	0.364	-1.9
26		A	9 / 4			0.28	0.9	0.22	0.1	0.41	-0.8	0.99	1.9
27		B	8 / 3			0.31	1.4	0.43	4.0	1.3	6.2	FN	-3.4
28		B	8 / 4	FN	-3.6	0.35	2.1	0.16	-1.0	0.35	-1.3	0.36	-1.9
29		B	1 / 1					0.00001	-4.0				
30		B	1 / 1							0.428	-0.6		
31		B	2 / 0										
32		B	2 / 1							0.543	0.3		
33	x	B	6 / 3			0.175	-0.9			0.516	0.1	0.519	-0.9
35		A	9 / 4	20.4	-0.2	0.175	-0.9	0.252	0.7	0.447	-0.5		
36	x	A	10 / 5	34.5	2.5	0.19	-0.7	0.19	-0.5	0.56	0.4	0.53	-0.9
37		B	2 / 0										
38		A	10 / 5	27.1	1.1	0.225	-0.1	0.224	0.2	0.682	1.4	0.460	-1.3
39		B	3 / 2					0.247	0.6	0.488	-0.2		
41		A	9 / 4			0.217	-0.2	0.195	-0.4	0.444	-0.5	0.758	0.5
42		B	7 / 4	20.4	-0.2			0.216	0.0	0.524	0.1	0.631	-0.3
43		B	7 / 4	24.3	0.6	0.24	0.2			0.62	0.9	0.80	0.7
44		A	10 / 5	17.8	-0.7	0.235	0.1	0.206	-0.2	0.517	0.1	0.960	1.7
46		A	10 / 5	17.09	-0.8	0.191	-0.7	0.214	0.0	0.564	0.4	0.616	-0.4
48	x	B	5 / 2					0.042	-3.2	0.089	-3.3		

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compounds. Individual z-scores were calculated using the FFP-RSD of 25 % and the assigned values.

COMPULSORY Compounds				Bromide		Ethephon		Glufosinate		Glyphosate		Phosphonic acid	
Assigned Value [mg/kg]				21.3		0.228		0.216		0.510		0.676	
*CV**				20.7 %		21.1 %		17.1 %		18.5 %		24.3 %	
MRRL [mg/kg]				2		0.04		0.02		0.1		0.1	
Lab code SRM16-	NRL-SRM	Cat	Analysed / corr. found, max. 10 / 5	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score
49	x	B	5 / 3	22.0	0.1	0.339	2.0			0.511	0.0		
50		B	1 / 1							0.496	-0.1		
51		B	1 / 1							0.82	2.4		
52	x	A	10 / 5	23.95	0.5	0.169	-1.0	0.332	2.2	0.412	-0.8	0.559	-0.7
55	x	B	2 / 0										
56		B	7 / 3			0.213	-0.3	0.215	0.0	0.561	0.4		
57	x	A	10 / 5	17.3	-0.8	0.116	-2.0	0.236	0.4	0.213	-2.3	0.593	-0.5
59	x	A	9 / 4			0.203	-0.4	0.180	-0.7	0.490	-0.2	0.668	-0.1
60		A	9 / 5	20.7	-0.1	0.250	0.4	0.198	-0.3	0.448	-0.5	0.538	-0.8
61		B	4 / 2			0.29	1.1			0.36	-1.2		
62		B	2 / 0										
63	x	B	8 / 4			0.987	13.3	0.232	0.3	0.562	0.4	0.578	-0.6
64	x	A	9 / 4			0.130	-1.7	0.104	-2.1	0.555	0.4	0.179	-2.9
65		A	10 / 5	20.2402	-0.2	0.4015	3.0	0.2950	1.5	0.6897	1.4	0.7593	0.5
66		B	3 / 1							0.513	0.0		
68		A	9 / 4			0.254	0.5	0.217	0.0	0.566	0.4	0.697	0.1
69		A	10 / 5	25.3	0.8	0.24	0.2	0.22	0.1	0.54	0.2	0.82	0.9
70	x	B	3 / 1			0.22	-0.1						
71		A	10 / 5	23.3	0.4	0.222	-0.1	0.211	-0.1	0.507	0.0	0.729	0.3
72		B	3 / 1							0.539	0.2		
73	x	A	10 / 5	24.7	0.6	0.307	1.4	0.288	1.3	0.540	0.2	0.732	0.3
74		B	2 / 0										
75		A	10 / 5	19.9	-0.3	0.215	-0.2	0.21	-0.1	0.52	0.1	0.665	-0.1
76	x	A	10 / 5	22.9	0.3	0.293	1.1	0.210	-0.1	0.500	-0.1	0.594	-0.5
78		B	2 / 0										
79		A	10 / 5	19	-0.4	0.23	0.0	0.27	1.0	0.74	1.8	0.78	0.6
80		B	2 / 1							0.466	-0.4		
82		B	5 / 2					0.222	0.1	0.460	-0.4		
83	x	A	9 / 5	22.5	0.2	0.282	1.0	0.260	0.8	0.489	-0.2	0.962	1.7
84	x	A	10 / 5	10.2	-2.1	0.142	-1.5	0.166	-0.9	0.363	-1.2	0.189	-2.9
86	x	B	3 / 0							FN	-3.2		
87		B	7 / 4	21.8	0.1	0.211	-0.3			0.539	0.2	1.06	2.3
88		A	9 / 4			0.248	0.4	0.190	-0.5	0.799	2.3	0.781	0.6
89	x	B	2 / 0										
90		B	7 / 4	14.16	-1.3	0.275	0.8			0.611	0.8	1.132	2.7
92		A	9 / 4			0.237	0.2	0.220	0.1	0.533	0.2	0.699	0.1
93		A	10 / 5	22.4	0.2	0.24	0.2	0.241	0.5	0.613	0.8	0.607	-0.4
94		B	5 / 3	25.4	0.8			1.104	16.4	0.813	2.4		
95	x	B	1 / 1	21.8	0.1								
96		A	10 / 5	17.6	-0.7	0.205	-0.4	0.209	-0.1	0.568	0.5	0.611	-0.4
97		A	10 / 5	21.010	-0.1	0.270	0.7	0.234	0.3	0.458	-0.4	0.654	-0.1
99		A	10 / 5	19.0	-0.4	0.277	0.9	0.228	0.2	0.480	-0.2	0.625	-0.3
101		B	0 / 0										

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for the COMPULSORY compounds. Individual z-scores were calculated using the FFP-RSD of 25 % and the assigned values.

COMPULSORY Compounds				Bromide		Ethephon		Glufosinate		Glyphosate		Phosphonic acid	
Assigned Value [mg/kg]				21.3		0.228		0.216		0.510		0.676	
*CV**				20.7 %		21.1 %		17.1 %		18.5 %		24.3 %	
MRRL [mg/kg]				2		0.04		0.02		0.1		0.1	
Lab code SRM16-	NRL-SRM	Cat	Analysed / corr. found, max. 10 / 5	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score
102		B	8 / 5	22	0.1	0.163	-1.1	0.398	3.4	0.573	0.5	0.482	-1.2
103	x	B	7 / 4	23.0	0.3	0.230	0.0	0.220	0.1	0.500	-0.1		
104		B	6 / 2					0.14	-1.4	0.36	-1.2	FN	-3.4
105		A	10 / 4	25.7	0.8	0.228	0.0	0.193	-0.4	FN	-3.2	0.803	0.8
107		A	9 / 4			0.249	0.4	0.219	0.1	0.391	-0.9	0.768	0.5
109		B	0 / 0										
111		B	4 / 3			0.345	2.1	0.276	1.1	0.305	-1.6		
112		B	7 / 5	19.0	-0.4	0.257	0.5	0.211	-0.1	0.486	-0.2	0.769	0.6
113		A	9 / 4			0.195	-0.6	0.21	-0.1	0.51	0.0	0.472	-1.2
114		B	8 / 5	27	1.1	0.18	-0.8	0.04	-3.3	0.59	0.6	0.57	-0.6
115		B	7 / 5	33.9	2.4	0.193	-0.6	0.178	-0.7	0.488	-0.2	0.696	0.1
116		B	1 / 1			0.227	0.0						
118	x	A	10 / 4	24.5	0.6	1.01	13.7	0.14	-1.4	0.59	0.6	FN	-3.4
120		B	9 / 4	FN	-3.6	0.317	1.6	0.36	2.7	0.493	-0.1	0.767	0.5
121	x	A	9 / 4	20	-0.2	0.23	0.0	0.20	-0.3	0.27	-1.9		
123		B	4 / 1							1.00	3.8		
124		A	10 / 5	24.0	0.5	0.206	-0.4	0.247	0.6	0.532	0.2	0.548	-0.8
126		A	10 / 5	21.64	0.1	0.203	-0.4	0.256	0.7	0.460	-0.4	0.582	-0.6
3rd-34		B	9 / 3	FN	-3.9	0.172	-1.0	0.247	0.6	0.513	0.0		
3rd-45		A	10 / 5	20.6	-0.1	0.243	0.3	0.243	0.5	0.532	0.2	0.737	0.4
Private-40		A	10 / 5	40.3	3.6	0.218	-0.2	0.189	-0.5	0.447	-0.5	0.936	1.5
Private-47		A	10 / 5	21.9	0.1	0.262	0.6	0.216	0.0	0.530	0.2	0.682	0.0
Private-53		A	10 / 5	20.8	-0.1	0.230	0.0	0.210	-0.1	0.525	0.1	0.635	-0.2
Private-54		A	10 / 5	22.1	0.2	0.261	0.6	0.248	0.6	0.528	0.1	0.770	0.6
Private-58		A	10 / 5	20.5	-0.2	0.22	-0.1	0.22	0.1	0.45	-0.5	0.69	0.1
Private-77		A	10 / 5	38.6	3.3	0.270	0.7	0.180	-0.7	0.530	0.2	0.530	-0.9
3rd-85		B	7 / 2					0.208	-0.2	0.442	-0.5		
3rd-91		A	10 / 5	23.2	0.4	0.156	-1.3	0.217	0.0	0.544	0.3	0.628	-0.3
3rd-98		B	8 / 3	21.7	0.1			0.201	-0.3	0.467	-0.3		
3rd-100		A	9 / 5	22.89	0.3	0.075	-2.7	0.22	0.1	0.521	0.1	0.82	0.9
3rd-106		B	8 / 5	24.8	0.7	0.295	1.2	0.312	1.8	0.609	0.8	0.538	-0.8
3rd-108		B	2 / 2	19.503	-0.3					0.115	-3.1		
Private-110		B	0 / 0										
Private-117		B	5 / 2							0.47	-0.3	0.65	-0.2
Private-119		B	0 / 0										
Private-122		B	0 / 0										
Private-125		B	6 / 4	19.3	-0.4	0.23	0.0	0.18	-0.7	0.55	0.3		
Private-127		B	7 / 4	20.4	-0.2	0.19	-0.7			0.49	-0.2	0.43	-1.5
3rd-128		B	10 / 3	17.39	-0.7	FN	-3.7	0.184	-0.6	0.506	0.0	FN	-3.4
3rd-129		B	6 / 3	19.09	-0.4			0.195	-0.4	0.579	0.5		
3rd-130		B	7 / 1			FN	-3.8			0.428	-0.6	FN	-3.4
3rd-131		B	8 / 5	20.89	-0.1	0.066	-2.8	0.226	0.2	0.511	0.0	0.88	1.2
3rd-132		B	0 / 0										

3rd: Laboratories from 3rd countries or EU candidate countries; Privat: EU Commercial laboratories

Table 4-9: Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds. Individual z-scores were calculated using the FFP-RSD of 25 % and the assigned values.

OPTIONAL Compounds				2-CE		EO (sum)		Chlorate		Diquat	
Assigned Value [mg/kg]				4.59		2.50		1.03		0.244	
*CV**				30.4 %		29.5 %		20.7 %		24.7 %	
MRRL [mg/kg]				0.05		0.05		0.04		0.02	
Lab code SRM16-	NRL-SRM	Cat	Analysed / corr. found, max. 11 / 8	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score
3		A	2 / 1								
4		A	3 / 2					1.12	0.3	0.231	-0.2
5	x	B	1 / 0								
6	x	A	7 / 4	5.30	0.6	2.90	0.6	1.22	0.7		
7		B	4 / 3	5.61 (cal.)	0.9	3.07	0.9	1.26	0.9		
8		A	7 / 4					1.2	0.7	0.47	3.7
9		B	1 / 1					0.76	-1.1		
10	x	A	8 / 5	4.94	0.3	2.71	0.3	0.998	-0.1	0.298	0.9
11	x	B	2 / 1								
12		B	0 / 0								
13		A	4 / 3					1.265	0.9	0.154	-1.5
14		B	5 / 2							0.236	-0.1
15		A	3 / 1					1.13	0.4		
16		B	2 / 1					0.73	-1.2		
17		B	7 / 5	3.70	-0.8	2.02	-0.8	1.13	0.4		
18		B	5 / 2	4.53	-0.1	2.48	0.0				
19	x	A	11 / 7	1.33	-2.8	0.723	-2.8	0.785	-1.0	0.230	-0.2
20	x	B	2 / 1								
21	x	A	10 / 7	4.55	0.0	2.49	0.0	1.25	0.8	0.248	0.1
22		B	5 / 1	FN	-4.0	FN	-3.9	0.703	-1.3		
23	x	A	8 / 5					0.886	-0.6	0.245	0.0
24		A	3 / 1					1.03	0.0		
25		A	4 / 2					1.90	3.4		
26		A	6 / 4	6.09 (cal.)	1.3	3.33	1.3	0.98	-0.2	0.30	0.9
27		B	4 / 1	2.19 (cal.)	-2.1	1.2	-2.1				
28		B	6 / 5					0.35	-2.6	0.2	-0.7
29		B	0 / 0								
30		B	1 / 0								
31		B	0 / 0								
32		B	2 / 1	0.393 (cal.)	-3.7	0.215	-3.7				
33	x	B	1 / 1					1.11	0.3		
35		A	0 / 0								
36	x	A	3 / 1					1.7	2.6		
37		B	0 / 0								
38		A	7 / 4	4.02 (cal.)	-0.5	2.20	-0.5	1.27	0.9		
39		B	1 / 0								
41		A	4 / 2					0.889	-0.6		
42		B	1 / 1					1.04	0.0		
43		B	7 / 5					0.93	-0.4	0.73	8.0
44		A	11 / 7	4.84	0.2	2.65	0.2	1.14	0.4	0.206	-0.6
46		A	8 / 5					1.099	0.3	0.223	-0.3
48	x	B	2 / 0								

(cal.): value calculated by the organizer and added to the population for the establishment of the assigned value

	Optional Compounds				Matrine		Nicotine		Paraquat		Trimesium	
	Assigned Value [mg/kg]				0.124		0.255		0.264		0.073	
	*CV**				26.5 %		27.5 %		25.0 %		14.8 %	
	MRRL [mg/kg]				0.01		0.01		0.02		0.01	
	Lab code SRM16-	NRL-SRM	Cat	Analysed / corr. found, max. 11 / 8	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score
	3		A	2 / 1	0.0790	-1.5						
	4		A	3 / 2								
	5	x	B	1 / 0								
	6	x	A	7 / 4			0.280	0.4			0.0825	0.5
	7		B	4 / 3	0.0782	-1.5						
	8		A	7 / 4	0.096	-0.9	0.12	-2.1			FN	-3.5
	9		B	1 / 1								
	10	x	A	8 / 5					0.296	0.5		
	11	x	B	2 / 1			0.14	-1.8				
	12		B	0 / 0								
	13		A	4 / 3	0.146	0.7						
	14		B	5 / 2	FN	-3.7			0.306	0.6		
	15		A	3 / 1								
	16		B	2 / 1								
	17		B	7 / 5	0.139	0.5	0.200	-0.9			0.0956	1.2
	18		B	5 / 2								
	19	x	A	11 / 7	0.145	0.7	0.277	0.4	0.221	-0.7	FN	-3.5
	20	x	B	2 / 1			0.230	-0.4				
	21	x	A	10 / 7	0.134	0.3	0.269	0.2	0.256	-0.1	0.071	-0.1
	22		B	5 / 1								
	23	x	A	8 / 5	0.139	0.5	0.271	0.3	0.264	0.0	0.069	-0.2
	24		A	3 / 1								
	25		A	4 / 2			0.266	0.2			0.0718	-0.1
	26		A	6 / 4					0.34	1.2		
	27		B	4 / 1								
	28		B	6 / 5	0.36	7.6	0.17	-1.3	0.23	-0.5		
	29		B	0 / 0								
	30		B	1 / 0								
	31		B	0 / 0								
	32		B	2 / 1								
	33	x	B	1 / 1								
	35		A	0 / 0								
	36	x	A	3 / 1								
	37		B	0 / 0								
	38		A	7 / 4	0.154	1.0	0.280	0.4			0.078	0.3
	39		B	1 / 0								
	41		A	4 / 2	0.102	-0.7						
	42		B	1 / 1								
	43		B	7 / 5	0.057	-2.2	0.070	-2.9	0.49	3.4	0.067	-0.3
	44		A	11 / 7	0.149	0.8	0.303	0.8	0.217	-0.7	0.084	0.6
	46		A	8 / 5	0.094	-1.0	0.236	-0.3	0.167	-1.5	0.060	-0.7
	48	x	B	2 / 0								

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds. Individual z-scores were calculated using the FFP-RSD of 25 % and the assigned values.

OPTIONAL Compounds				2-CE		EO (sum)		Chlorate		Diquat	
Assigned Value [mg/kg]				4.59		2.50		1.03		0.244	
*CV**				30.4 %		29.5 %		20.7 %		24.7 %	
MRRL [mg/kg]				0.05		0.05		0.04		0.02	
Lab code SRM16-	NRL-SRM	Cat	Analysed / corr. found, max. 11 / 8	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score
49	x	B	1 / 0								
50		B	1 / 0								
51		B	0 / 0								
52	x	A	2 / 0								
55	x	B	2 / 2							0.283	0.6
56		B	2 / 1					1.01	-0.1		
57	x	A	5 / 3	6.83	2.0	3.74 (cal.)	2.0			0.306	1.0
59	x	A	2 / 1							0.170	-1.2
60		A	5 / 3	3.19	-1.2	1.75	-1.2	0.934	-0.4		
61		B	1 / 0								
62		B	0 / 0								
63	x	B	5 / 3					1.32	1.1	0.083	-2.6
64	x	A	3 / 2							0.440	3.2
65		A	3 / 1					1.1530	0.5		
66		B	2 / 1								
68		A	11 / 7	1.12	-3.0	0.613	-3.0	1.13	0.4	0.259	0.3
69		A	7 / 5					1.1	0.3	0.22	-0.4
70	x	B	0 / 0								
71		A	8 / 4	4.64	0.0	2.54 (cal.)	0.1	0.929	-0.4		
72		B	1 / 1								
73	x	A	5 / 2					1.12	0.3		
74		B	0 / 0								
75		A	9 / 5	3.66	-0.8	2.0	-0.8	1.004	-0.1		
76	x	A	0 / 0								
78		B	1 / 1					1.10	0.3		
79		A	8 / 6	6.03 (cal.)	1.3	3.3	1.3	1.0	-0.1	0.27	0.4
80		B	0 / 0								
82		B	2 / 1					1.14	0.4		
83	x	A	6 / 3					1.24	0.8	0.616	6.1
84	x	A	0 / 0								
86	x	B	1 / 1								
87		B	10 / 6	3.98	-0.5	2.15	-0.6	0.806	-0.9	0.240	-0.1
88		A	7 / 5	3.78	-0.7	2.41	-0.1	0.933	-0.4	0.414	2.8
89	x	B	0 / 0								
90		B	5 / 4							0.154	-1.5
92		A	4 / 2							0.241	-0.1
93		A	5 / 2					1.03	0.0		
94		B	5 / 3	6.25 (cal.)	1.5	3.42	1.5			0.230	-0.2
95	x	B	0 / 0								
96		A	9 / 6	3.56 (cal.)	-0.9	1.95	-0.9	0.87	-0.6	0.225	-0.3
97		A	7 / 5	3.768	-0.7	2.059	-0.7	1.108	0.3		
99		A	3 / 1					1.11	0.3		

(cal.): value calculated by the organizer and added to the population for the establishment of the assigned value

	Optional Compounds				Matrine		Nicotine		Paraquat		Trimesium	
	Assigned Value [mg/kg]				0.124		0.255		0.264		0.073	
	*CV**				26.5 %		27.5 %		25.0 %		14.8 %	
	MRRL [mg/kg]				0.01		0.01		0.02		0.01	
	Lab code SRM16-	NRL-SRM	Cat	Analysed / corr. found, max. 11 / 8	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score
	49	x	B	1 / 0								
	50		B	1 / 0								
	51		B	0 / 0								
	52	x	A	2 / 0								
	55	x	B	2 / 2					0.303	0.6		
	56		B	2 / 1								
	57	x	A	5 / 3					0.285	0.3		
	59	x	A	2 / 1								
	60		A	5 / 3							0.057	-0.9
	61		B	1 / 0							0.069	-0.2
	62		B	0 / 0								
	63	x	B	5 / 3					0.045	-3.3		
	64	x	A	3 / 2					0.086	-2.7		
	65		A	3 / 1								
	66		B	2 / 1			0.177	-1.2				
	68		A	11 / 7	0.441	10.2	0.313	0.9	0.222	-0.6	0.083	0.6
	69		A	7 / 5	0.17	1.5	0.30	0.7	0.26	-0.1		
	70	x	B	0 / 0								
	71		A	8 / 4	0.144	0.7	0.308	0.8			0.0710	-0.1
	72		B	1 / 1			0.230	-0.4				
	73	x	A	5 / 2	0.354	7.4					0.090	0.9
	74		B	0 / 0								
	75		A	9 / 5	0.13	0.2	0.234	-0.3			0.066	-0.4
	76	x	A	0 / 0								
	78		B	1 / 1								
	79		A	8 / 6	0.131	0.2	0.26	0.1	0.31	0.7		
	80		B	0 / 0								
	82		B	2 / 1								
	83	x	A	6 / 3					0.520	3.9	0.0810	0.4
	84	x	A	0 / 0								
	86	x	B	1 / 1			0.446	3.0				
	87		B	10 / 6	0.195	2.3			0.242	-0.3	0.249	9.6
	88		A	7 / 5					0.537	4.1		
	89	x	B	0 / 0								
	90		B	5 / 4	0.140	0.5	0.240	-0.2	0.252	-0.2		
	92		A	4 / 2					0.253	-0.2		
	93		A	5 / 2			0.316	1.0			0.065	-0.4
	94		B	5 / 3					0.201	-1.0		
	95	x	B	0 / 0								
	96		A	9 / 6	0.12	-0.1	0.272	0.3	0.265	0.0		
	97		A	7 / 5	0.144	0.7	0.281	0.4				
	99		A	3 / 1								

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds. Individual z-scores were calculated using the FFP-RSD of 25 % and the assigned values.

OPTIONAL Compounds				2-CE		EO (sum)		Chlorate		Diquat	
Assigned Value [mg/kg]				4.59		2.50		1.03		0.244	
*CV**				30.4 %		29.5 %		20.7 %		24.7 %	
MRRL [mg/kg]				0.05		0.05		0.04		0.02	
Lab code SRM16-	NRL-SRM	Cat	Analysed / corr. found, max. 11 / 8	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score
101		B	1 / 1								
102		B	4 / 3					0.354	-2.6		
103	x	B	2 / 0								
104		B	2 / 0								
105		A	3 / 2					0.896	-0.5		
107		A	8 / 5					0.723	-1.2	0.193	-0.8
109		B	3 / 2	8.70	3.6	5.74	5.2				
111		B	2 / 1					0.641	-1.5		
112		B	5 / 4	2.99	-1.4	1.64	-1.4	1.20	0.7		
113		A	6 / 4					1.463	1.7	0.459	3.5
114		B	7 / 5	5.47	0.8	3.0	0.8	0.24	-3.1		
115		B	7 / 4	2.90	-1.5	1.59 (cal.)	-1.5	1.26	0.9		
116		B	0 / 0								
118	x	A	10 / 7	0.94	-3.2	0.51	-3.2	0.96	-0.3	0.27	0.4
120		B	6 / 3	17.2	11.0	9.4	11.0				
121	x	A	2 / 1	2.0	-2.3	1.09 (cal.)	-2.3				
123		B	2 / 2					1.09	0.2		
124		A	6 / 5					1.07	0.1	0.243	0.0
126		A	10 / 7	5.659	0.9	3.237	1.2	0.796	-0.9	0.209	-0.6
3rd-34		B	0 / 0								
3rd-45		A	6 / 3	6.33	1.5	3.46 (cal.)	1.5	1.16	0.5		
Private-40		A	10 / 6	6.25 (cal.)	1.5	3.42	1.5	1.37	1.3	0.282	0.6
Private-47		A	7 / 5					1.18	0.6	0.2	-0.7
Private-53		A	5 / 3					0.895	-0.5	0.200	-0.7
Private-54		A	11 / 7	5.00	0.4	2.74	0.4	1.18	0.6	0.242	0.0
Private-58		A	11 / 7	5.32	0.6	2.93	0.7	1	-0.1	0.23	-0.2
Private-77		A	6 / 5	3.00	-1.4	1.83	-1.1	1.09	0.2		
3rd-85		B	3 / 1	6.54 (cal.)	1.7	3.58	1.7				
3rd-91		A	4 / 2	3.88	-0.6	2.13	-0.6				
3rd-98		B	3 / 1	5.67 (cal.)	0.9	3.10	1.0				
3rd-100		A	8 / 4	4.05	-0.5	2.21	-0.5			0.212	-0.5
3rd-106		B	7 / 6	1.69	-2.5	0.925	-2.5	1.18	0.6	0.281	0.6
3rd-108		B	3 / 2							0.157	-1.4
Private-110		B	2 / 2	4.412	-0.2	2.418	-0.1				
Private-117		B	6 / 4	5.44	0.7	2.98	0.8	1.08	0.2		
Private-119		B	2 / 1	3.08 (cal.)	-1.3	1.683	-1.3				
Private-122		B	2 / 1	4.75 (cal.)	0.1	2.60	0.2				
Private-125		B	5 / 3	6.03 (cal.)	1.3	3.3	1.3	1.0	-0.1		
Private-127		B	5 / 4	3.16 (cal.)	-1.3	1.73	-1.2	0.77	-1.0		
3rd-128		B	0 / 0								
3rd-129		B	5 / 3	4.19	-0.4	2.2	-0.5				
3rd-130		B	5 / 1	3.07 (cal.)	-1.3	1.68	-1.3			FN	-3.8
3rd-131		B	7 / 5	4.04	-0.5	2.18	-0.5			0.224	-0.3
3rd-132		B	3 / 2	3.953	-0.6	2.174	-0.5				

(cal.): value calculated by the organizer and added to the population for the establishment of the assigned value

	Optional Compounds				Matrine		Nicotine		Paraquat		Trimesium	
	Assigned Value [mg/kg]				0.124		0.255		0.264		0.073	
	*CV**				26.5 %		27.5 %		25.0 %		14.8 %	
	MRRL [mg/kg]				0.01		0.01		0.02		0.01	
	Lab code SRM16-	NRL-SRM	Cat	Analysed / corr. found, max. 11 / 8	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score	Conc. [mg/kg]	z-Score
	101		B	1 / 1					0.284	0.3		
	102		B	4 / 3	0.091	-1.1	0.132	-1.9				
	103	x	B	2 / 0								
	104		B	2 / 0							0.075	0.1
	105		A	3 / 2	0.079	-1.5						
	107		A	8 / 5	0.153	0.9	0.362	1.7	0.205	-0.9	0.080	0.4
	109		B	3 / 2								
	111		B	2 / 1								
	112		B	5 / 4			0.544	4.5				
	113		A	6 / 4	0.139	0.5			0.497	3.5	0.059	-0.8
	114		B	7 / 5	0.03	-3.0	0.06	-3.1				
	115		B	7 / 4	0.138	0.5	0.298	0.7				
	116		B	0 / 0								
	118	x	A	10 / 7	0.11	-0.5	0.23	-0.4	0.26	-0.1		
	120		B	6 / 3			0.271	0.3				
	121	x	A	2 / 1								
	123		B	2 / 2			0.32	1.0				
	124		A	6 / 5	0.132	0.3	0.225	-0.5	0.268	0.1		
	126		A	10 / 7	0.130	0.2	0.291	0.6	0.221	-0.7	0.069	-0.2
	3rd-34		B	0 / 0								
	3rd-45		A	6 / 3	0.136	0.4						
	Private-40		A	10 / 6	0.0530	-2.3	0.258	0.1	0.241	-0.4	0.0749	0.1
	Private-47		A	7 / 5	0.102	-0.7	0.255	0.0	0.207	-0.9		
	Private-53		A	5 / 3					0.195	-1.1		
	Private-54		A	11 / 7	0.163	1.3	0.245	-0.2	0.217	-0.7	0.067	-0.3
	Private-58		A	11 / 7	0.22	3.1	0.4	2.3	0.34	1.2	0.07	-0.2
	Private-77		A	6 / 5	0.160	1.2	0.320	1.0				
	3rd-85		B	3 / 1								
	3rd-91		A	4 / 2							0.0754	0.1
	3rd-98		B	3 / 1								
	3rd-100		A	8 / 4			0.28	0.4	FN	-4.0		
	3rd-106		B	7 / 6	0.147	0.7			0.242	-0.3		
	3rd-108		B	3 / 2					0.146	-1.8		
	Private-110		B	2 / 2								
	Private-117		B	6 / 4	0.18	1.8						
	Private-119		B	2 / 1								
	Private-122		B	2 / 1								
	Private-125		B	5 / 3			0.25	-0.1				
	Private-127		B	5 / 4	0.09	-1.1	0.29	0.6				
	3rd-128		B	0 / 0								
	3rd-129		B	5 / 3			0.202	-0.8				
	3rd-130		B	5 / 1					FN	-3.9		
	3rd-131		B	7 / 5			0.255	0.0	0.21	-0.8		
	3rd-132		B	3 / 2								

4.3.4 Laboratory Classification Based on Scope and Combined z-Scores

All participating laboratories having reported at least one result were classified into categories A or B according to the rules defined in the General Protocol (9th Edition, see **Appendix 8**), see also **Section 2.5 (p. 13)**. Following 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 eleven out of the twelve compulsory pesticides on the Target Pesticides List; b) correct detection of at least four out of the five compulsory pesticides present in the test item, and c) no false positive results.

A total of 47 EU and EFTA laboratories (46 %) were classified into Category A and 56 (54 %) into Category B. In the case of laboratories from one EU candidate or 3rd countries, only 3 out of the 13 participating laboratories (23 %) were classified into Category A. Half of the 12 non-OfL-commercial laboratories in EU/EFTA-countries were also categorized into Category A, but it should be noted that some of these labs only focused on the analysis of EO, ***EO (sum)*** and ***2-CE*** in this PT.

Looking at the 29 participating NRL-SRMs overall 16 laboratories (55 %) were classified into Category A and 13 laboratories (45 %) into Category B.

Looking at compulsory compounds only, the laboratories from EU and EFTA countries classified into Category A achieved an overall average of absolute z-scores (AAZ) of 0.75 (n = 223), and those classified into Category B an overall AAZ of 1.17 (n = 134). The overall AAZ of the total population of participating labs classified into Category A and B was 0.71 (n = 268) and 1.13 (n = 176), respectively.

Table 4-10 shows the detailed z-and AAZ-scores of laboratories classified into Category A with the focus being on compulsory compounds. **Table 4-11 (p. 44)** shows the overall AAZ achhieved by all participating laboratories, based on all compounds present in the test item (with the exception of ***2-CE***). The ***2-CE*** results were excluded from this calculation in order to avoid a disproportionate weighting of EO-related

Table 4-10: Category A laboratories in EUPT-SRM16, ordered by lab-codes. AAZs were calculated only for laboratories with at least 5 z-scores for analytes present in the test item. For the AAZ calculation, any z-scores > 5 were set at 5.

COMPULSORY Compounds			Bromide	Ethephon	Glufosinate	Glyphosate	Phosphonic acid	
Assigned Value [mg/kg]			21.3	0.228	0.216	0.51	0.676	
*CV**			20.7 %	21.1 %	17.1 %	18.5 %	24.3 %	
MRRL [mg/kg]			2	0.04	0.02	0.1	0.1	
Lab code SRM16-	NRL-SRM	Analysed / corr. found, max. 10 / 5	z-Score	z-Score	z-Score	z-Score	z-Score	AAZ
3		10 / 5	-0.6	-0.6	-0.7	-0.2	-1.1	0.6
4		9 / 4		-1.0	-0.7	0.3	0.3	
6	x	10 / 5	-0.3	0.1	-0.2	-0.2	0.0	0.2
8		10 / 5	1.9	-2.6	0.8	2.9	1.4	1.9
10	x	10 / 5	-1.1	-0.2	-0.6	0.1	0.7	0.5
13		10 / 5	-4.0	-0.2	-0.7	1.8	1.5	1.6
15		10 / 5	-1.3	-0.3	0.4	2.0	0.0	0.8
19	x	10 / 5	-1.9	-0.5	-0.6	-0.6	-0.4	0.8
21	x	10 / 5	0.7	1.3	-0.3	-0.2	1.5	0.8
23	x	10 / 5	-1.6	-0.2	0.2	0.5	0.9	0.7
24		10 / 5	-0.6	-0.4	0.1	-0.8	0.2	0.4

Table 4-10 (cont.): Category A laboratories in EUPT-SRM16, ordered by lab-codes. AAZs were calculated only for laboratories with at least 5 z-scores for analytes present in the test item. For the AAZ calculation, any z-scores > 5 were set at 5.

COMPULSORY Compounds			Bromide	Ethephon	Glufosinate	Glyphosate	Phosphonic acid	
Assigned Value [mg/kg]			21.3	0.228	0.216	0.51	0.676	
*CV**			20.7 %	21.1 %	17.1 %	18.5 %	24.3 %	
MRRL [mg/kg]			2	0.04	0.02	0.1	0.1	
Lab code SRM16-	NRL-SRM	Analysed / corr. found, max. 10 / 5	z-Score	z-Score	z-Score	z-Score	z-Score	AAZ
25		10 / 5	2.6	-0.4	-0.3	0.5	-1.9	1.1
26		9 / 4		0.9	0.1	-0.8	1.9	
35		9 / 4	-0.2	-0.9	0.7	-0.5		
36	x	10 / 5	2.5	-0.7	-0.5	0.4	-0.9	1.0
38		10 / 5	1.1	-0.1	0.2	1.4	-1.3	0.8
41		9 / 4		-0.2	-0.4	-0.5	0.5	
44		10 / 5	-0.7	0.1	-0.2	0.1	1.7	0.6
46		10 / 5	-0.8	-0.7	0.0	0.4	-0.4	0.5
52	x	10 / 5	0.5	-1.0	2.2	-0.8	-0.7	1.0
57	x	10 / 5	-0.8	-2.0	0.4	-2.3	-0.5	1.2
59	x	9 / 4		-0.4	-0.7	-0.2	-0.1	
60		9 / 5	-0.1	0.4	-0.3	-0.5	-0.8	0.4
64	x	9 / 4		-1.7	-2.1	0.4	-2.9	
65		10 / 5	-0.2	3.0	1.5	1.4	0.5	1.3
68		9 / 4		0.5	0.0	0.4	0.1	
69		10 / 5	0.8	0.2	0.1	0.2	0.9	0.4
71		10 / 5	0.4	-0.1	-0.1	0.0	0.3	0.2
73	x	10 / 5	0.6	1.4	1.3	0.2	0.3	0.8
75		10 / 5	-0.3	-0.2	-0.1	0.1	-0.1	0.2
76	x	10 / 5	0.3	1.1	-0.1	-0.1	-0.5	0.4
79		10 / 5	-0.4	0.0	1.0	1.8	0.6	0.8
83	x	9 / 5	0.2	1.0	0.8	-0.2	1.7	0.8
84	x	10 / 5	-2.1	-1.5	-0.9	-1.2	-2.9	1.7
88		9 / 4		0.4	-0.5	2.3	0.6	
92		9 / 4		0.2	0.1	0.2	0.1	
93		10 / 5	0.2	0.2	0.5	0.8	-0.4	0.4
96		10 / 5	-0.7	-0.4	-0.1	0.5	-0.4	0.4
97		10 / 5	-0.1	0.7	0.3	-0.4	-0.1	0.3
99		10 / 5	-0.4	0.9	0.2	-0.2	-0.3	0.4
105		10 / 4	0.8	0.0	-0.4	-3.2	0.8	1.0
107		9 / 4		0.4	0.1	-0.9	0.5	
113		9 / 4		-0.6	-0.1	0.0	-1.2	
118	x	10 / 4	0.6	13.7	-1.4	0.6	-3.4	2.2
121	x	9 / 4	-0.2	0.0	-0.3	-1.9		0.6
124		10 / 5	0.5	-0.4	0.6	0.2	-0.8	0.5
126		10 / 5	0.1	-0.4	0.7	-0.4	-0.6	0.4
3rd-45		10 / 5	-0.1	0.3	0.5	0.2	0.4	0.3
Private-40		10 / 5	3.6	-0.2	-0.5	-0.5	1.5	1.3
Private-47		10 / 5	0.1	0.6	0.0	0.2	0.0	0.2
Private-53		10 / 5	-0.1	0.0	-0.1	0.1	-0.2	0.1
Private-54		10 / 5	0.2	0.6	0.6	0.1	0.6	0.4
Private-58		10 / 5	-0.2	-0.1	0.1	-0.5	0.1	0.2
Private-77		10 / 5	3.3	0.7	-0.7	0.2	-0.9	1.2
3rd-91		10 / 5	0.4	-1.3	0.0	0.3	-0.3	0.5
3rd-100		9 / 5	0.3	-2.7	0.1	0.1	0.9	0.8

Table 4-11: Compilation of AAZ-scores of all laboratories for compulsory analytes and for all analytes excluding 2-CE. For details please see Section 4.3.4, p. 42. For the AAZ calculation, any z-scores > 5 were set at 5.

Lab code SRM16-	NRL-SRM	Cat	COMULSORY Analysed / corr. found, max. 10 / 5	OPTIONAL Analysed / corr. found, max. 11 / 8	TOTAL Analysed / corr. found, max. 21 / 13	FPs	FNs	AAZ (COMULSORY Analytes)	AAZ (ALL Analytes excl. 2-CE)
3		A	10 / 5	2 / 1	12 / 6			0.6	0.8
4		A	9 / 4	3 / 2	12 / 6				0.5
5	x	B	8 / 5	1 / 0	9 / 5			0.5	0.5
6	x	A	10 / 5	7 / 4	17 / 9			0.2	0.3
7		B	7 / 4	4 / 3	11 / 7				2.3
8		A	10 / 5	7 / 4	17 / 9		1	1.9	2.1
9		B	0 / 0	1 / 1	1 / 1				
10	x	A	10 / 5	8 / 5	18 / 10			0.5	0.5
11	x	B	7 / 4	2 / 1	9 / 5				0.6
12		B	1 / 1	0 / 0	1 / 1				
13		A	10 / 5	4 / 3	14 / 8			1.6	1.4
14		B	8 / 3	5 / 2	13 / 5		2		2.3
15		A	10 / 5	3 / 1	13 / 6			0.8	0.7
16		B	6 / 3	2 / 1	8 / 4				
17		B	8 / 5	7 / 5	15 / 10			0.4	0.6
18		B	7 / 4	5 / 2	12 / 6				0.2
19	x	A	10 / 5	11 / 7	21 / 12		1	0.8	1.1
20	x	B	7 / 3	2 / 1	9 / 4				
21	x	A	10 / 5	10 / 7	20 / 12			0.8	0.5
22		B	7 / 4	5 / 1	12 / 5		2		1.2
23	x	A	10 / 5	8 / 5	18 / 10			0.7	0.5
24		A	10 / 5	3 / 1	13 / 6			0.4	0.4
25		A	10 / 5	4 / 2	14 / 7			1.1	1.2
26		A	9 / 4	6 / 4	15 / 8				0.9
27		B	8 / 3	4 / 1	12 / 4		1		3.2
28		B	8 / 4	6 / 5	14 / 9		1	2.0	2.0
29		B	1 / 1	0 / 0	1 / 1				
30		B	1 / 1	1 / 0	2 / 1				
31		B	2 / 0	0 / 0	2 / 0				
32		B	2 / 1	2 / 1	4 / 2	1			
33	x	B	6 / 3	1 / 1	7 / 4				
35		A	9 / 4	0 / 0	9 / 4				
36	x	A	10 / 5	3 / 1	13 / 6			1.0	1.3
37		B	2 / 0	0 / 0	2 / 0				
38		A	10 / 5	7 / 4	17 / 9			0.8	0.7
39		B	3 / 2	1 / 0	4 / 2				
41		A	9 / 4	4 / 2	13 / 6				0.5
42		B	7 / 4	1 / 1	8 / 5				0.1
43		B	7 / 4	7 / 5	14 / 9				1.7
44		A	10 / 5	11 / 7	21 / 12			0.6	0.6
46		A	10 / 5	8 / 5	18 / 10			0.5	0.6
48	x	B	5 / 2	2 / 0	7 / 2				
49	x	B	5 / 3	1 / 0	6 / 3				
50		B	1 / 1	1 / 0	2 / 1				
51		B	1 / 1	0 / 0	1 / 1				
52	x	A	10 / 5	2 / 0	12 / 5			1.0	1.0
55	x	B	2 / 0	2 / 2	4 / 2				
56		B	7 / 3	2 / 1	9 / 4				
57	x	A	10 / 5	5 / 3	15 / 8			1.2	1.2

Table 4-11 (cont.): Compilation of AAZ-scores of all laboratories for compulsory analytes and for all analytes excluding 2-CE. For details please see Section 4.3.4, p. . For the AAZ calculation, any z-scores > 5 were set at 5.

Lab code SRM16-	NRL-SRM	Cat	COMULSORY Analysed / corr. found, max. 10 / 5	OPTIONAL Analysed / corr. found, max. 11 / 8	TOTAL Analysed / corr. found, max. 21 / 13	FPs	FNs	AAZ (COMULSORY Analytes)	AAZ (ALL Analytes excl. 2-CE)
59	x	A	9 / 4	2 / 1	11 / 5				0.5
60		A	9 / 5	5 / 3	14 / 8			0.4	0.6
61		B	4 / 2	1 / 0	5 / 2				
62		B	2 / 0	0 / 0	2 / 0				
63	x	B	8 / 4	5 / 3	13 / 7	2			1.9
64	x	A	9 / 4	3 / 2	12 / 6				2.2
65		A	10 / 5	3 / 1	13 / 6			1.3	1.2
66		B	3 / 1	2 / 1	5 / 2				
68		A	9 / 4	11 / 7	20 / 11				1.1
69		A	10 / 5	7 / 5	17 / 10			0.4	0.5
70	x	B	3 / 1	0 / 0	3 / 1				
71		A	10 / 5	8 / 4	18 / 9			0.2	0.3
72		B	3 / 1	1 / 1	4 / 2				
73	x	A	10 / 5	5 / 2	15 / 7			0.8	1.3
74		B	2 / 0	0 / 0	2 / 0				
75		A	10 / 5	9 / 5	19 / 10			0.2	0.3
76	x	A	10 / 5	0 / 0	10 / 5			0.4	0.4
78		B	2 / 0	1 / 1	3 / 1				
79		A	10 / 5	8 / 6	18 / 11			0.8	0.6
80		B	2 / 1	0 / 0	2 / 1				
82		B	5 / 2	2 / 1	7 / 3				
83	x	A	9 / 5	6 / 3	15 / 8			0.8	1.6
84	x	A	10 / 5	0 / 0	10 / 5			1.7	1.7
86	x	B	3 / 0	1 / 1	4 / 1		1		
87		B	7 / 4	10 / 6	17 / 10				1.2
88		A	9 / 4	7 / 5	16 / 9	1*			1.4
89	x	B	2 / 0	0 / 0	2 / 0				
90		B	7 / 4	5 / 4	12 / 8				1.0
92		A	9 / 4	4 / 2	13 / 6				0.2
93		A	10 / 5	5 / 2	15 / 7			0.4	0.4
94		B	5 / 3	5 / 3	10 / 6				1.8
95	x	B	1 / 1	0 / 0	1 / 1				
96		A	10 / 5	9 / 6	19 / 11			0.4	0.4
97		A	10 / 5	7 / 5	17 / 10			0.3	0.4
99		A	10 / 5	3 / 1	13 / 6			0.4	0.4
101		B	0 / 0	1 / 1	1 / 1				
102		B	8 / 5	4 / 3	12 / 8			1.3	1.5
103	x	B	7 / 4	2 / 0	9 / 4				
104		B	6 / 2	2 / 0	8 / 2		1		
105		A	10 / 4	3 / 2	13 / 6		1	1.0	1.0
107		A	9 / 4	8 / 5	17 / 9				0.8
109		B	0 / 0	3 / 2	3 / 2	1			
111		B	4 / 3	2 / 1	6 / 4				
112		B	7 / 5	5 / 4	12 / 9			0.4	1.1
113		A	9 / 4	6 / 4	15 / 8				1.3
114		B	8 / 5	7 / 5	15 / 10			1.3	1.8
115		B	7 / 5	7 / 4	14 / 9			0.8	0.8
116		B	1 / 1	0 / 0	1 / 1				

* FP was due to an optional compound and was therefore not consindered in the evaluation of performance/categorization.

Table 4-11 (cont.): Compilation of AAZ-scores of all laboratories for compulsory analytes and for all analytes excluding 2-CE. For details please see Section 4.3.4, p. . For the AAZ calculation, any z-scores > 5 were set at 5.

Lab code SRM16-	NRL-SRM	Cat	COMULSORY Analysed / corr. found, max. 10 / 5	OPTIONAL Analysed / corr. found, max. 11 / 8	TOTAL Analysed / corr. found, max. 21 / 13	FPs	FNs	AAZ (COMULSORY Analytes)	AAZ (ALL Analytes excl. 2-CE)
118	x	A	10 / 4	10 / 7	20 / 11		1	2.2	1.4
120		B	9 / 4	6 / 3	15 / 7	1	1	1.7	2.0
121	x	A	9 / 4	2 / 1	11 / 5				0.9
123		B	4 / 1	2 / 2	6 / 3				
124		A	10 / 5	6 / 5	16 / 10			0.5	0.4
126		A	10 / 5	10 / 7	20 / 12			0.4	0.6
3rd-34		B	9 / 3	0 / 0	9 / 3	1	1		
3rd-45		A	10 / 5	6 / 3	16 / 8			0.3	0.5
Private-40		A	10 / 5	10 / 6	20 / 11			1.3	1.1
Private-47		A	10 / 5	7 / 5	17 / 10			0.2	0.4
Private-53		A	10 / 5	5 / 3	15 / 8			0.1	0.4
Private-54		A	10 / 5	11 / 7	21 / 12			0.4	0.5
Private-58		A	10 / 5	11 / 7	21 / 12			0.2	0.7
Private-77		A	10 / 5	6 / 5	16 / 10	1*		1.2	1.0
3rd-85		B	7 / 2	3 / 1	10 / 3				
3rd-91		A	10 / 5	4 / 2	14 / 7			0.5	0.3
3rd-98		B	8 / 3	3 / 1	11 / 4				
3rd-100		A	9 / 5	8 / 4	17 / 9		1	0.8	1.1
3rd-106		B	8 / 5	7 / 6	15 / 11			1.1	1.0
3rd-108		B	2 / 2	3 / 2	5 / 4				
Private-110		B	0 / 0	2 / 2	2 / 2				
Private-117		B	5 / 2	6 / 4	11 / 6				0.7
Private-119		B	0 / 0	2 / 1	2 / 1				
Private-122		B	0 / 0	2 / 1	2 / 1				
private-125		B	6 / 4	5 / 3	11 / 7				0.4
Private-127		B	7 / 4	5 / 4	12 / 8				0.8
3rd-128		B	10 / 3	0 / 0	10 / 3		2	1.7	1.7
3rd-129		B	6 / 3	5 / 3	11 / 6				0.5
3rd-130		B	7 / 1	5 / 1	12 / 2		4		2.8
3rd-131		B	8 / 5	7 / 5	15 / 10			0.9	0.7
3rd-132		B	0 / 0	3 / 2	3 / 2				

* FP was due to an optional compound and was therefore not consindered in the evaluation of performance/categorization.

results, considering that most of the ***EO (sum)*** results were mathemetically converted from ***2-CE*** results, or vice versa, and the z-scores of both analytes were therefore equal or very close. Also here, the AAZ was only calculated for laboratories with 5 or more z-scores for analytes present in the test material and any z-scores > 5 were set at 5 for the AAZ calculation.

Looking at all compounds present in the test item (the compulsory and optional ones); the overall AAZ of labs classified into Category A calculates at 0.78 (n = 481) and of the labs classified into Category B at 1.22 (n = 288). Overall, this indicates that laboratories covering a larger scope (the main criterion for the classification into Category A and B) also tend to achieve lower z-scores. Looking at the NRL-SRMs, the overall AAZ obtained (for all compounds) was 0.98 (n = 166), which is slightly worse, but still comparable to the overall AAZ of all other labs, which calculates at 0.93 (n = 603). Also here, the AAZ-scores were calculated including results of ***EO (sum)*** and excluding results of ***2-CE***.

4.4 Feedback from Laboratories in Case of Poor Results

Like in the previous EUPT-SRMs, as a follow-up measure to this EUPT, all participating laboratories having achieved questionable ($2 < |z\text{-score}| < 3$) or unacceptable ($|z\text{-score}| \geq 3$) or false positive results were asked to investigate the reasons for their poor performance and to report them to the organisers. The aim of this measure is to sensibilize the laboratories to investigate the sources of errors and to improve analytical know-how and performance in the long run.

103 OfLs from EU/EFTA countries reported 623 numerical results for analytes present in the test item, among them 85 results were allocated with a $|z\text{-scores}| > 2$. In addition, there were 13 cases of FN results that were alocated with a z-score lower than -3 and 6 FP results. Thus in total, there were 104 cases of poor performance, concerning 43 EU/EFTA OfLs that had to be investigated. Overall, 28 laboratories gave feedback in 67 cases (64 % out of 104 cases) about the possible reasons for their poor performance and some of them even reported details about their investigations. In many cases, no clear reasons for the poor performance could be identified or non-specific reasons (such as lack of experience) were provided.

In fact, "Lack of experience" (21 cases) was the most frequent reason for poor performance reported by the labs in the present PT. Some of those participants had no or little experience with the analysis of polar compounds in high oil content matrices or with the analysis of ***EO*** or ***2-CE*** in general. In 10 cases each, the following reasons for poor performance were reported: "analytical procedure was inappropriate", "analytical procedure was appropriate but it was not properly performed" and "calculation errors". Other often reported error sources for poor performance were: "measurement problems" (9×), "erroneous analytical standard" (6×), "analyte losses during the procedure" (5×), and "inappropriate/erroneous calibration approach" (4×). One participant reported a "transcriptional error" as the reason for its 3 poor z-scores. In one case each "misinterpretation/misevaluation of measurement data", "result not properly corrected for recovery", "misinterpretation/misevaluation of measurement data" and "deficient QC-measures " were reported as reasons.

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

4.5 Method-based Evaluation

4.5.1 Impact of using ILIS on the analytical results

The usefulness of employing ILISs for reducing error sources in analysis was repeatedly highlighted in past PT reports and is also stressed here in the case of the highly polar compounds ***chlorate***, ***diquat***, ***ethephon***, ***glufosinate***, ***glyphosate***, ***nicotine***, ***paraquat*** and ***phosphonic acid***. In the case of ***trimesium*** the population of laboratories using ILIS was too small for reliable statistics. **Table 4-12** shows the comparison of the statistical figures obtained for the total population and the sub-populations using or not using ILIS. Overall, the distance between the robust mean values of the two sub-populations to that of the total population was < 5 % with the excption of ***nicotine*** (7 % distance between robust mean of sub-population using ILIS to robust mean of total population). When comparing the robust means of the two sub-populations among them, the largest distance was observed in the case of ***nicotine*** 11 % (w. ILIS: 0.273 mg/kg; w/o ILIS: 0.246 mg/kg) and ***phosphonic acid*** 8 % (w. ILIS: 0.704 mg/kg; w/o ILIS: 0.650 mg/kg).

Table 4-12: Impact of ILISs or other ISs on the distribution of results and the average bias (only results from EU and EFTA laboratories were taken into account)

	Ethephon		
	Whole Population	**Matching ILIS[1)]**	**No IS or other IS (Calibration Approach)**
No. of Numerical Results	75	41	29 Procedural | 5 StAdd-SP | 8 StAdd-EA | 2 MM | 9 (2°°) SB | 3
Outlier(s) thereof [1)]	3	1	2
No. of False Negative Results	1	1	0
Robust Mean [mg/kg]	**0.228**	**0.235**	**0.233**
CV*	**21.1 %**	**18.0 %**	**27.9 %**
UAV	0.007	0.008	0.016
Tolerance	0.0171	0.0176	0.0175
Judgement	passed	passed	passed
	Glufosinate		
	Whole Population	**Matching ILIS[1)]**	**No IS or other IS (Calibration Approach)**
No. of Numerical Results	72	35	32 Procedural | 5 StAdd-SP | 8 StAdd-EA | 2 MM | 13 SB | 2 (2°°)
Outlier(s) thereof [1)]	4	2	2
No. of False Negative Results	0	0	0
Robust Mean [mg/kg]	**0.216**	**0.218**	**0.213**
CV*	**17.1 %**	**13.4 %**	**29.4 %**
UAV	0.0056	0.0063	0.0143
Tolerance	0.0162	0.0164	0.016
Judgement	passed	passed	passed
	Glyphosate		
	Whole Population	**Matching ILIS[1)]**	**No IS or other IS (Calibration Approach)**
No. of Numerical Results	86	53	29 Procedural | 4 (1°°) StAdd-SP | 8 StAdd-EA | 3 MM | 11 SB | 2
Outlier(s) thereof [1)]	1	0	1
No. of False Negative Results	2	2	0
Robust Mean [mg/kg]			
CV*	**0.510**	**0.514**	**0.499**
UAV	**18.5 %**	**19.5 %**	**15.5 %**
Tolerance	0.0128	0.0172	0.0183
Judgement	0.0383	0.0386	0.0374
Judgement	passed	passed	passed

1) ILIS added at beginning of procedure or in a intermediate step

Table 4-12 (cont.): Impact of ILISs or other ISs on the distribution of results and the average bias (only results from EU and EFTA laboratories were taken into account)

	Phosphonic acid		
	Whole Population	**Matching ILIS[1)]**	**No IS or other IS (Calibration Approach)**
No. of Numerical Results	62	29	28 Procedural | 5 StAdd-SP | 8 StAdd-EA | 3 MM | 8 SB | 4
Outlier(s) thereof [1)]	0	0	0
No. of False Negative Results	3	0	3
Robust Mean [mg/kg]	**0.676**	**0.704**	**0.651**
CV*	**24.3 %**	**23.1 %**	**24.9 %**
UAV	0.0261	0.0378	0.0383
Tolerance	0.0507	0.0528	0.0488
Judgement	passed	passed	passed
	Chlorate		
	Whole Population	**Matching ILIS[1)]**	**No IS or other IS (Calibration Approach)**
No. of Numerical Results	57	32	20 Procedural | 2 StAdd-SP | 3 StAdd-EA | 3 MM | 8 SB | 4
Outlier(s) thereof [1)]	0	0	0
No. of False Negative Results	0	0	0
Robust Mean [mg/kg]	**1.033**	**1.023**	**1.053**
CV*	**20.7 %**	**19.4 %**	**24.2 %**
UAV	0.0353	0.0439	0.0712
Tolerance	0.0775	0.0767	0.079
Judgement	passed	passed	passed
	Diquat		
	Whole Population	**Matching ILIS[1)]**	**No IS or other IS (Calibration Approach)**
No. of Numerical Results	33	16	16 Procedural | 3 StAdd-SP | 5 StAdd-EA | 0 MM | 4 (1°°) SB | 3
Outlier(s) thereof [1)]	2	1	1
No. of False Negative Results	0	0	0
Robust Mean [mg/kg]	**0.244**	**0.25**	**0.269**
CV*	**24.7 %**	**11.1 %**	**51.1 %**
UAV	0.0136	0.0089	0.0444
Tolerance	0.0183	0.0188	0.0202
Judgement	passed	passed	failed

1) ILIS added at beginning of procedure or in a intermediate step

Table 4-12 (cont.): Impact of ILISs or other ISs on the distribution of results and the average bias (only results from EU and EFTA laboratories were taken into account)

	Nicotine		
	Whole Population	**Matching ILIS[1)]**	**No IS or other IS (Calibration Approach)**
Outlier(s) thereof [1)]	36	9	27
No. of False Negative Results			Procedural | 2
Robust Mean [mg/kg]			StAdd-SP | 6
CV*			StAdd-EA | 2
UAV			MM | 13
Tolerance			SB | 4
Judgement	0	0	0
No. of False Negative Results	0	0	0
Robust Mean [mg/kg]	**0.255**	**0.273**	**0.246**
CV*	**27.5 %**	**23.3 %**	**32.6 %**
UAV	0.0146	0.0266	0.0193
Tolerance	0.0191	0.0205	0.0185
Judgement	passed	failed	failed
	Paraquat		
	Whole Population	**Matching ILIS[1)]**	**No IS or other IS (Calibration Approach)**
No. of Numerical Results	30	15	15
			Procedural | 3
			StAdd-SP | 5
			StAdd-EA | 0
			MM | 4
			SB | 3
Outlier(s) thereof [1)]	0	0	0
No. of False Negative Results	0	0	0
Robust Mean [mg/kg]	**0.264**	**0.273**	**0.266**
CV*	**25 %**	**17.8 %**	**57.8 %**
UAV	0.015	0.0157	0.0497
Tolerance	0.0198	0.0205	0.02
1) ILIS added at beginning of procedure or in a intermediate step			
Judgement	passed	passed	failed
1) ILIS added at beginning of procedure or in a intermediate step			

The impact of using ILIS was much more visible when looking at the robust relative standard deviation (CV^*). Here the CV^*-figure of the sub-population using ILIS was on average almost 50 % lower than that of the sub-population not using ILIS (18.2 % versus 32.9 %). It should be further noted, that the labratories not employing ILIS have partly used other means for correcting their results for recovery or matrix effects, such as standard additions, procedural calibrations and matrix matching.

The most pronounced impact of using ILIS on thethe CV^* figures were observed for ***diquat*** (All results: 24.7 %; w/o ILIS 51.1 % and w. ILIS 11.1 %), ***paraquat*** (All results: 25.0 %; w/o ILIS 57.8 % and w. ILIS 17.8 %) and ***glufosinate*** (All results: 17.1 %; w/o ILIS 29.4 % and w. ILIS 13.4 %). Rather marginal differences were observed in the case of ***Phosphonic acid*** (All results: 24.3 %; w/o ILIS 24.9 % and w. ILIS 23.1 %), ***chlorate*** (All results: 20.7 %; w/o ILIS 24.2 % and w. ILIS 19.4 %) and ***glyphosate*** (All results: 18.5 %; w/o ILIS 15.5 % and w. ILIS 19.5 %) with the latter being the only compound where a reverse trend was observed.

4.5.2 Impact on non-adding water during extraction of 2-CE when using the QuPPe method

2-CE tends to undergo strong adhere to the matrix via hydrogen bonds. The addition of water during the extraction helps to interrupt those interactions thus speeding up extraction and increasing extraction yields. In absence of water, extraction is hampered, which leads to underestimated results. In the current PT, several laboratories employing the QuOil method did not add water during the extraction step. These labs either submitted strongly underestimated results and obtained z-scores < -2 or even reported FN results. As shown in **Figure 4-2**, the extractability of EO is highly facilitated when water is added during this step. This sub-population of results in highlighted in **Figure 4-1**.

The laboratories affected were contacted by the organizers shortly after the PT, and were made aware that the non-addition of water can lead to severely underestimated results. Furthermore, the EUPT-AdvG decided that these results should be excluded from the population used to calculate the assigned value for ***2-CE*** and ***EO (sum)***, as these results were generated using a methodology that traceably leads to biased results. As can be seen in **Table 4-13**. This measure has notably shifted the AV towards higher values and has also reduced the variability of the results (reduced CV^*).

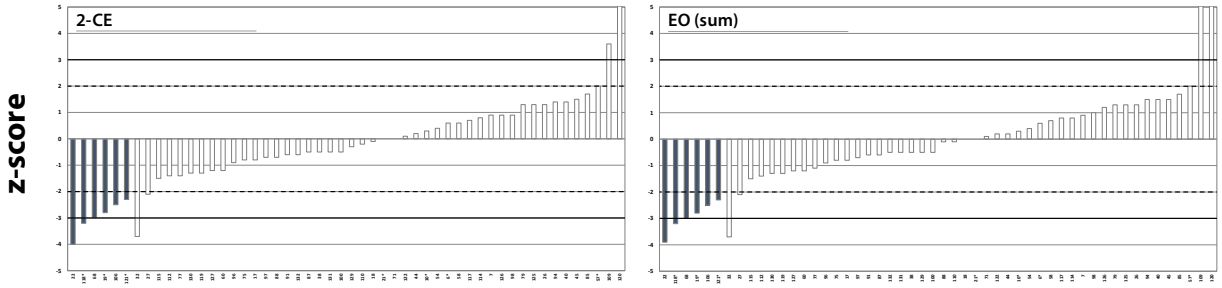


Figure 4-1: Distribution of z-Scores. ■: Laboratories without water addtion during extration step

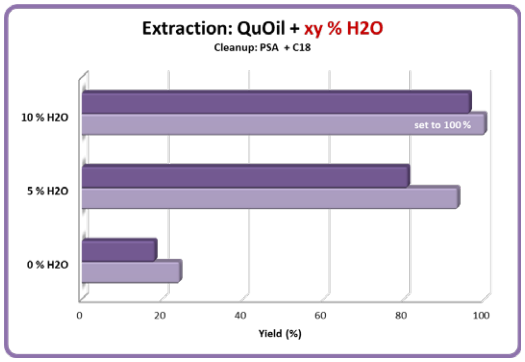


Figure 4-2: Impact of water addtion and extration time on the extractbility of 2-CE

Table 4-13: Impact of water addition during extracion step on the analysis of 2-CE and EO.

	2-CE		EO (sum)	
	Whole Population	excl. results w/o water addtion during extration	Whole Population	excl. results w/o water addtion during extration
No. of Numerical Results	34 + 17*	29 + 17*	46 + 5*	42 + 4*
Outlier(s) thereof [1)]	1	1	2	2
No. of False Negative Results	1	0	1	0
Robust Mean [mg/kg]	**4.30**	**4.59**	**2.33**	**2.50**
CV^*	**39.1 %**	**30.4 %**	**38.2 %**	**29.5%**
* mathemetically converted by the organizer from 2-CE to EO (sum) or vice versa.				

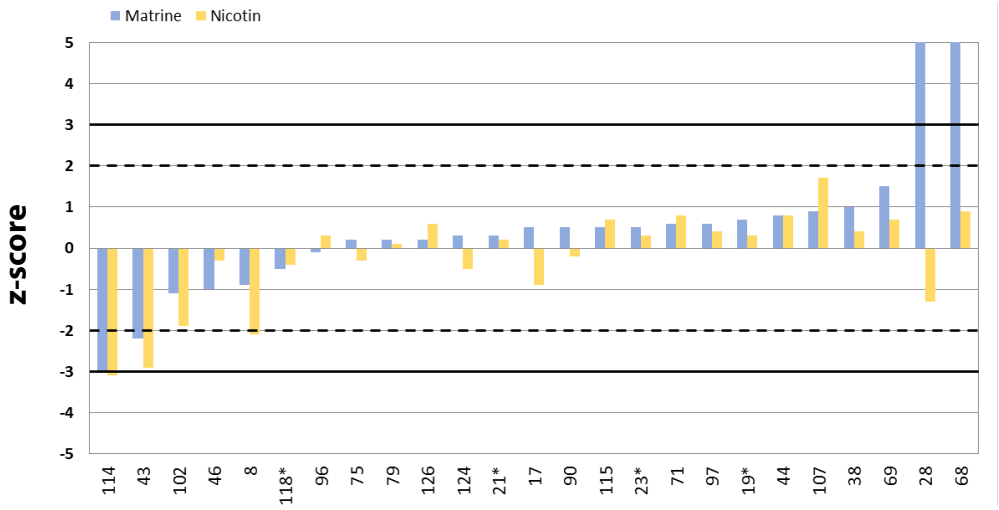


Figure 4-3: Impact of water addtion and extration time on the extractbility of 2-CE

4.5.3 Performance correlation between Matrine and Nicotine results

Matrine and nicotine both share high polarity and bacicity. QuEChERS recoveries are poor unless pH is raised to ensure deprotonation. In QuPPe-like methods matrix effects need to be taken into account. Correction for recovery or matrix-effects is possible via ILIS or other means. In this PT, 25 participating laboratories submitted results for both analytes and in many cases performance of them correlated well to each other (**Figure 4-3**). Labs having submitted underestimated results for both nicotine and matrine should check whether this underestimation can be traced back to certain steps within the methodology used.

4.5.4 Long-term Trends in Analytical Coverage and Performance

Over the years, the analytical scope of the laboratories within the official network has improved considerably. In addition, the analytical performance has also improved overall. **Figure 4-4** shows the EUPT-SRM history of ethephon and glyphosate over the past years: Compared to the EUPT-SRM4 in 2009, the number of laboratories analyzing these two pesticides increased more than 10 times. The results distribution depends on the complexity of the matrix used, but still a clear improvement in the CV^* can be noted overall, although many labs with little experience with the analysis of these compounds were added to the population in every PT.

Long-term Performance: Ethephon

	SRM4	SRM5	SRM6	SRM7	SRM10	SRM16
Matrix	Oats	Apple Juice	Rice	Lentils	Maiz	Sesame
Ethephon						
No. Results	4	28	28	32	61	76
% of Participants	8.3%	35%	36%	29%	59%	74%
CV* [%]	–	23.0%	29.7%	25.5%	30.8%	21.1%

Avg CV*= 26.0 %

Long-term Performance: Glyphosate

	SRM4	SRM6	SRM7	SRM8	SRM10	SRM12	SRM13	SRM14	SRM15	SRM16
Matrix	Oats	Rice	Lentils	Potatoes	Maiz	Straw-berries	Soy-beans	Liver	Rice	Sesame
Glyphosate										
No. Results	6	34	39	45	62	86	83	43	84	88
% of Participants	13%	34%	35%	41%	60%	67%	76%	75%	78%	88%
CV* [%]	--	40.6%	34.5%	24.5%	22.8%	20.9%	22.6%	23.3%	23.7%	18.5%

Avg CV*= 22.3 %

Figure 4-4: Long-term trends in the performance and coverage of ethephon and glyphosate

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

- The use of ILISs is highly recommended as it has been repeatedly shown over the years that signifficantly contibute to reduce average bias.

- When analyzing 2-CE residues in dry commodities, such as sesame, using the QuOil method, use acetonitrile containing water (e.g., 5 % as prescibed) as it facilitates the extraction by weakening the interactions of this analyte with the matrix. Also, make sure to have a long enough extraction time (e.g. 10 – 30 minutes) in order to ensure good extraction yields.

- Issues concerning results submission:
  - » Based on the "General procedures for reporting results" in the General Protocol, laboratories should not report results below their reporting limits.
  - » In accordance with the Specific Protocols for the EUPT-SRMs, quantitative results of the pesticide residues must be reported in mg/kg using THREE significant figures: e.g. 0.0582 mg/kg; 0.156 mg/kg, 1.64 mg/kg or 20.3 mg/kg.

- Organisational aspects relevant for future EUPT-SRMs:
  - » In accordance with the the General Protocol, the official language used in all EUPTs is English. This includes all types of communication including about analytical, organisational or administrative (like payment) issues.
  - » The PT-organisers like to appeal to the participants to track their own parcels via the online tracking tool of the shipping company in order to maintain the ability to take any necessary measures in case of delays, e.g., providing the customs with all necessary documents and asking for an acceleration of the clearance procedure, or placing the parcel in a cool place until clearance is granted. The participants are furthermore encouraged to contact the local office of the shipping company to ensure optimal delivery timing.
  - » 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-Organisers 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 in your laboratory, please fill-in the forms yourself based on the data in our financial identification note (https://www.eurl-pesticides.eu/library/docs/srm/SRM-Bank_m_Financial_Identification.pdf) and send us the filled form. After verification, and if necessary after correction, we will return it with signature and stamp.
  - » If no payment or no proof of payment is received and no explanation is given to the PT-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.

5. ACKNOWLEDGEMENTS

The organisers wish to thank the members of the EUPT Scientific Committee (Quality Control Group and Advisory Group) for their valuable advice. Special thanks go to Mette Poulson and Helen Fodnæss at the EURL-CF for coordination with the EDV-Team at DTU, Anne-Mette Skovlund, Steen Maigaard, Sean Gomes, Nicolaj Graversen Pedersen and Wardan Ghazal, wo continued the development and update the webtool for the present PT and supported the PT organizers, whenever they needed help regarding the results submission tool.

6. REFERENCES

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

[2] Regulation (EC) N° 396/2005, published at OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published at OJ of the EU L234 of 30.08.2008.

[3] https://www.eurl-pesticides.eu/library/docs/srm/EurlSrm_Observation_EO_V1.pdf

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

[5] https://www.eurl-pesticides.eu/userfiles/file/meth_QuPPe_AO_V3_1.pdf

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

7. APPENDICES

Appendix 1 List of Participating Laboratories in the EUPT-SRM16

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

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

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

Country (Location)	Analysed on behalf of	Institution	City	NRL
Germany	DE	LTZ Augustenberg - Organic Analysis	Karlsruhe	–
Germany	DE	LUA Rheinland-Pfalz, Institut für LM-Chemie Speyer	Speyer	–
Germany	DE	LUA Sachsen ((Dresden)	Dresden	–
Germany	DE	LUFA Speyer	Speyer	–
Germany	DE	Thüringer Landesanstalt für Landwirtschaft, Jena	Jena	–
Germany	DE	ZInstSanBw Kiel - Pesticide Lab	Kronshagen	–
Germany	MT	Eurofins - Germany, Hamburg	Hamburg	–
Greece	GR	Benaki Phytopathological Institute, Kifissia	Kifissia	x
Greece	GR	GCSL (Athens)	Athens	x
Hungary	HU	FCSCN Ltd Pesticide Res. Anal. Lab. Miskolc	Miskolc	–
Hungary	HU	NFCSO (Velence)	Velence	x
Ireland	IE	The Food Chemistry Laboratories - DAFM	Co. Kildare	x
Italy	IT	APPA Bolzano	Bolzano	–
Italy	IT	APPA-Puglia | Polo Alimenti Bari	Bari	–
Italy	IT	ARPA FVG - Pesticide Lab (Udine)	Udine	–
Italy	IT	ARPA Lazio (sez. Latina)	Latina	–
Italy	IT	ARPA Veneto (Verona)	Verona	–
Italy	IT	ASF	Firenze	–
Italy	IT	Istituto Superiore di Sanità - Roma	Roma	x
Italy	IT	IZS LT - Italy, Rome	Roma	–
Italy	IT	IZSAM	Teramo	–
Italy	IT	IZSLER	Brescia	–
Italy	IT	Laboratorio di Prevenzione (Bergamo)	Bergamo	–
Latvia	LV	BIOR (Riga)	Riga	x
Lithuania	LT	NMVRVI (Vilnius)	Vilnius	x
Luxembourg	LU	LNS Food lab	Dudelange	x
Norway	NO	NIBIO - Department of Pesticide Chemistry	ÅS	x
Poland	PL	Hamilton UO-Technologia	Grójec	–
Poland	PL	InHort (Skierniewice)	Skierniewice	–
Poland	PL	IPP-NRI - Pesticide Lab (Poznan)	Poznan	–
Poland	PL	Laboratory of Food & Feed Safety in Bialystok	Bialystok	–
Poland	PL	SGS Sp. z o.o. Laboratorium Srodowiskowe	pszczyna	–
Poland	PL	VSES Warszawa	Warszaw	x
Portugal	PT	Pesticide Lab (Funchal - Madeira Island)	Funchal - Madeira Island	x
Romania	RO	IISPV (Bucharest)	Bucharest	x
Slovakia	SK	State Veterinary and Food Institute (Bratislava)	Bratislava	x
Slovenia	SI	National laboratory of Health, Environment and Food (Maribor)	Maribor	x
Spain	ES	AINIA (Valencia)	Valencia	–
Spain	ES	Analytica Alimentaria GmbH - Almeria, Spain	Almeria	–
Spain	ES	EURL-FV - Pesticide Residue Research Group	Almeria	–
Spain	ES	EUROFINS ECOSUR	LORQUI - MURCIA	–
Spain	ES	Laboratori Agència Salut Pública Barcelona	Barcelona	–
Spain	ES	Laboratorio Agroalimentario de Valencia	Burjassot, Valencia	–
Spain	ES	Laboratorio Agroalimentario de Extremadura	Cáceres	–
Spain	ES	Laboratorio Agroambiental de Zaragoza	Zaragoza	–
Spain	ES	Laboratorio Analítico Bioclínico (Almería)	Almeria	–
Spain	ES	Laboratorio Arbitral Agroalimentario, Madrid	Madrid	x
Spain	ES	LABORATORIO KUDAM, S.L.	Pilar de la Horadada (Alicante)	–

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

Country (Location)	Analysed on behalf of	Institution	City	NRL
Spain	ES	Labs & Technological Services AGQ - Burguillos	Burguillos	–
Spain	ES	National Center for Technology and Food Safety	San Adrián (Navarra)	–
Spain	ES	National Centre for Food (Majadahonda)	Majadahonda	x
Sweden	SE	Eurofins Food & Feed - Pesticide Lab (Lidköping)	Lidköping	–
Switzerland	CH	Kantonales Laboratorium Bern	Bern	–
Switzerland	CH	Kantonales Laboratorium Zürich	Zürich	–
The Netherlands	BE	Groen Agro Control - Netherlands	Delfgauw	–
The Netherlands	BE	Handelslaboratorium Dr. Verwey - Pesticide Lab	Rotterdam	–
The Netherlands	BE	NofaLab - Pesticide Lab	Schiedam	–
The Netherlands	BE; NL	Eurofins Lab Zeeuws-Vlaanderen B.V. - Pesticiden	Graauw	–
The Netherlands	NL	Wageningen Food Safety Research (WFSR)	Wageningen	x

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

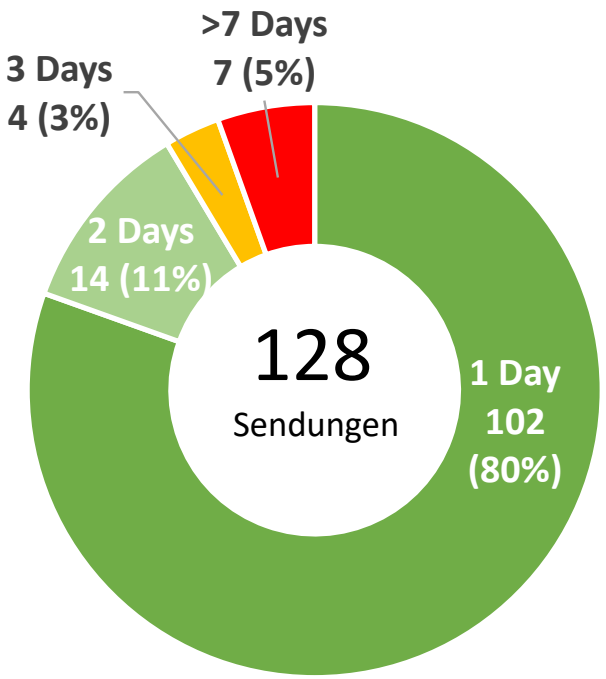
Country	Institution	City
India	Eureka - India, Bangalore	Bangalore
India	Eureka - India, Kundli	Kundli
India	Eureka - India, Unjha	Unjha
India	Eurofins - India, Gurgaon	Gurgaon
India	EUROFINS, India, Bangalore	Bangalore
India	Geo Chem - India, Mumbai	Mumbai
India	SGS - India, Gujarat	Gujarat
India	SGS - Pesticide Lab (Chennai)	CHENNAI
Korea, Republic of	Korea Institute of Toxicology (KIT) - Jinju	JINJU
Korea, Republic of	Nat. Institute of Agriculture - Suwon, Gyeonggi	Suwon, Gyeonggi
Serbia	SP Laboratorija	BECEJ
Thailand	Central Laboratory (Bangkok)	Bangkok
United Kingdom	FERA	York

Appendix 1-c: Commercial labs from EU / EFTA countries and participating in the EUPT-SRM16

Country	Institution	City
Cyprus	FAFAL - Analytical Laboratory	Larnaca
Germany	Analytica Alimentaria GmbH - Lab (Kleinmachnow)	Kleinmachnow
Germany	Eurofins Institut Dr. Appelt - Leipzig	Leipzig
Germany	Gesellschaft für Bioanalytik Hamburg	Hamburg
Germany	PhytoLab GmbH & Co KG - Vestenbergsgreuth	Vestenbergsgreuth
Italy	EPTA NORD SRL - Italy, Conselve	Conselve
Italy	Neotron (Modena)	Modena
Italy	pH srl, TÜV Italia - Italy, Florence	Florence
Italy	Water & Life Lab S.R.L. - Lab	Entratico
Spain	Labcolor-Coexphal - Spain, Almeria	La Mojonera, Almeria
Spain	Reactiva Lab (Almeria)	Almeria
Switzerland	COOP central Laboratory - CH, Pratteln	Pratteln

Appendix 2 Shipment Evaluation

Compilation of shipment duration



Appendix 3 Data of Homogeneity Test

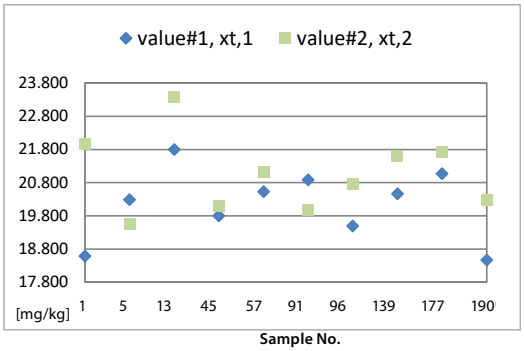
Compulsory Compounds

	Bromide ion		Ethephon		Glufosinate		Glyphosate		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]
1	18.589	21.975	0.233	0.245	0.207	0.210	0.462	0.535	0.689	0.680
5	20.282	19.557	0.242	0.232	0.209	0.218	0.558	0.450	0.680	0.693
13	21.793	23.365	0.236	0.247	0.206	0.217	0.462	0.495	0.692	0.730
45	19.799	20.101	0.229	0.237	0.212	0.215	0.454	0.477	0.704	0.705
57	20.524	21.128	0.251	0.250	0.245	0.243	0.630	0.519	0.722	0.709
91	20.887	19.980	0.238	0.235	0.211	0.236	0.501	0.496	0.730	0.723
96	19.496	20.766	0.230	0.243	0.215	0.222	0.484	0.482	0.672	0.731
139	20.463	21.612	0.242	0.241	0.225	0.234	0.460	0.529	0.777	0.697
177	21.068	21.733	0.243	0.252	0.226	0.227	0.527	0.559	0.720	0.731
190	18.469	20.282	0.219	0.227	0.190	0.195	0.458	0.467	0.650	0.664
mean / AV*	20.6 / 21.3		0.239 / 0.228		0.218 / 0.216		0.500 / 0.510		0.705 / 0.676	

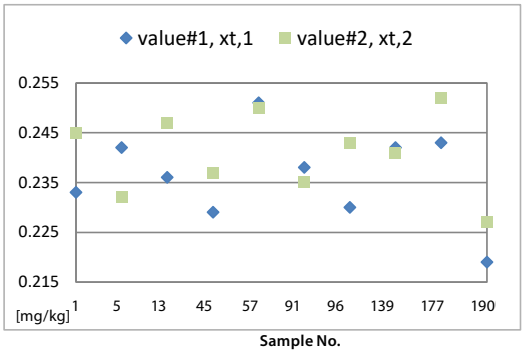
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Graphical presentation of the results:

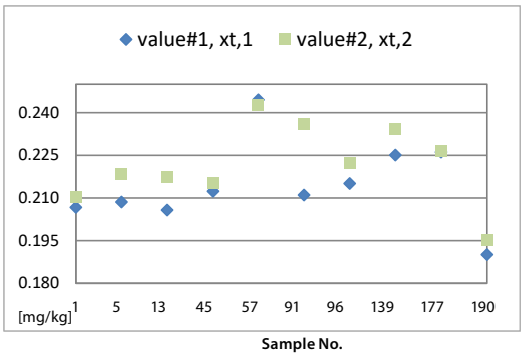
Bromide ion



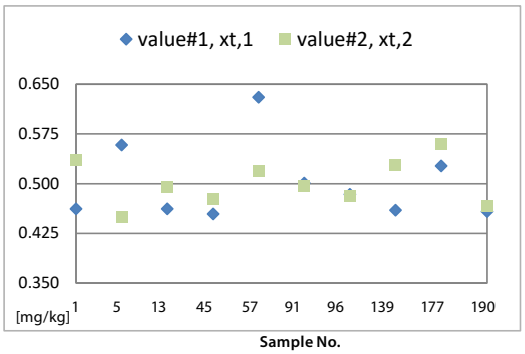
Ethephon



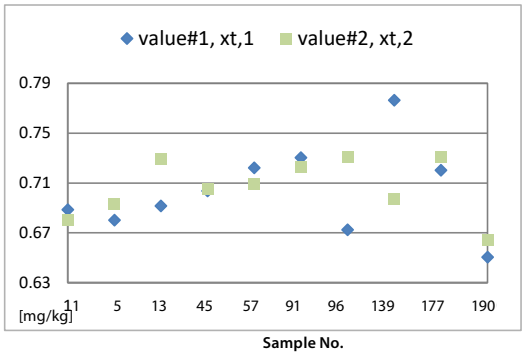
Glufosinate



Glyphosate



Phosphonic acid



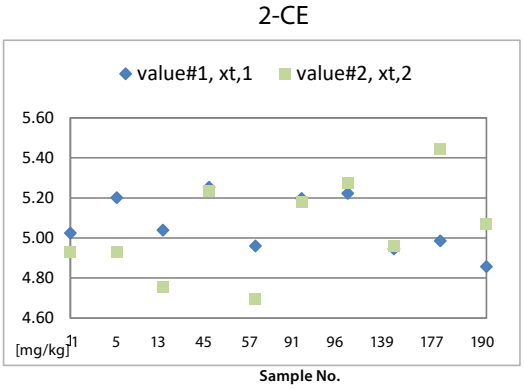
Appendix 3 (cont.): Data of Homogeneity Test

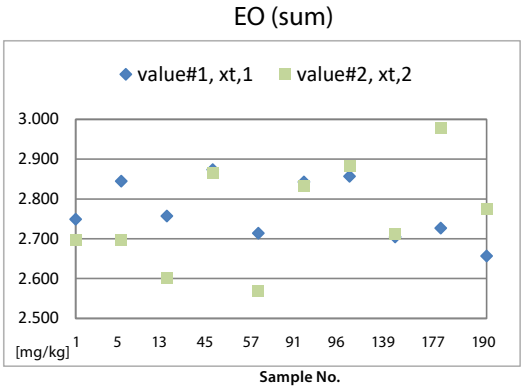
Optional Compounds

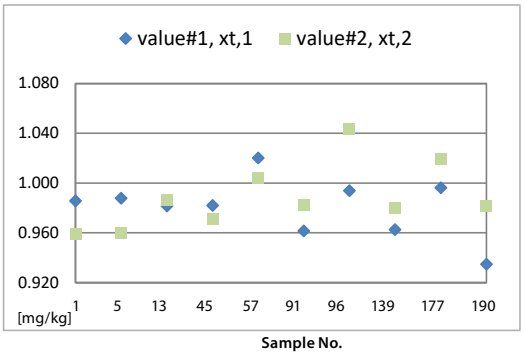
	2-CE		EO (sum)		Chlorate		Diquat	
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Portion 1 [mg/kg]	Portion 2 [mg/kg]
1	5.024	4.931	2.749	2.697	0.986	0.960	0.208	0.223
5	5.201	4.928	2.845	2.696	0.988	0.960	0.206	0.187
13	5.039	4.754	2.757	2.601	0.982	0.986	0.212	0.223
45	5.253	5.236	2.874	2.865	0.982	0.971	0.207	0.186
57	4.960	4.696	2.714	2.569	1.020	1.004	0.222	0.220
91	5.197	5.179	2.843	2.833	0.962	0.982	0.223	0.199
96	5.222	5.272	2.857	2.884	0.994	1.043	0.203	0.214
139	4.945	4.958	2.705	2.712	0.963	0.980	0.219	0.212
177	4.985	5.444	2.727	2.978	0.996	1.019	0.226	0.236
190	4.856	5.070	2.657	2.774	0.935	0.981	0.199	0.211
mean / AV*	5.06 / 4.49 ‡		2.77 / 2.50 ‡		0.985 / 1.03		0.212 / 0.244	

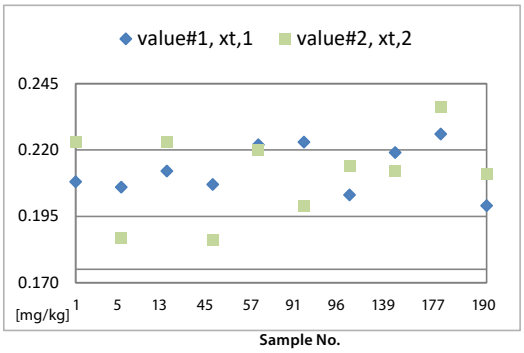
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories
‡ AV based on sub-population of results generated by methods including water addition only

Graphical presentation of the results:









Appendix 3 (cont.): Data of Homogeneity Test

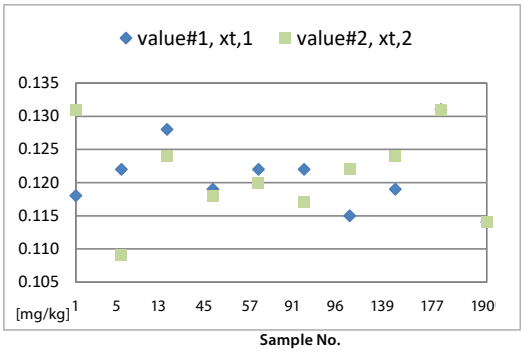
Optional Compounds

	Matrine		Nicotine		Paraquat		Trimesium	
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]
1	0.118	0.131	0.301	0.330	0.251	0.244	0.065	0.074
5	0.122	0.109	0.314	0.305	0.250	0.240	0.068	0.065
13	0.128	0.124	0.317	0.339	0.224	0.227	0.071	0.073
45	0.119	0.118	0.301	0.304	0.217	0.233	0.064	0.069
57	0.122	0.120	0.325	0.323	0.261	0.236	0.072	0.071
91	0.122	0.117	0.332	0.323	0.258	0.249	0.068	0.072
96	0.115	0.122	0.325	0.309	0.237	0.268	0.066	0.069
139	0.119	0.124	0.309	0.314	0.240	0.224	0.069	0.067
177	0.131	0.131	0.340	0.311	0.233	0.269	0.070	0.068
190	0.114	0.114	0.284	0.296	0.234	0.239	0.064	0.067
mean / AV*	0.121 / 0.124		0.315 / 0.255		0.242 / 0.264		0.069 / 0.073	

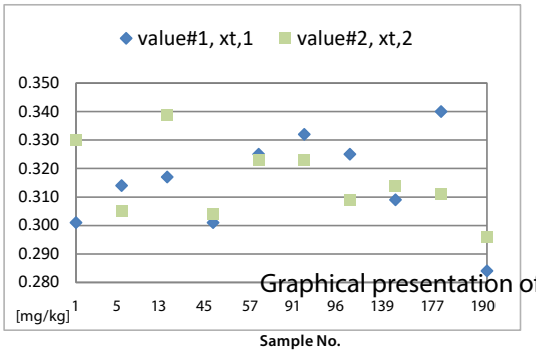
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Graphical presentation of the results:

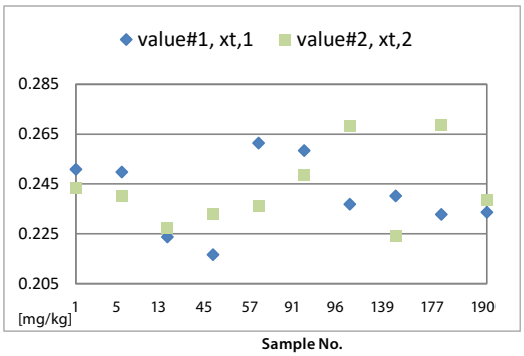
Matrine



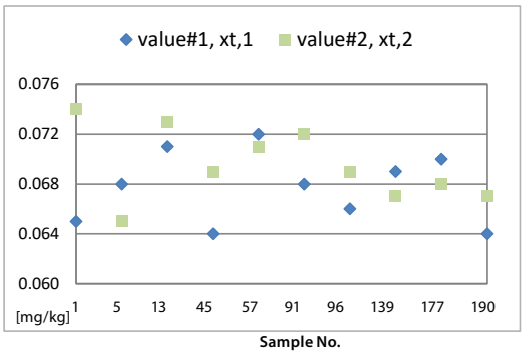
Nicotine



Paraquat



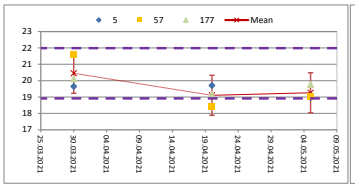
Trimesium



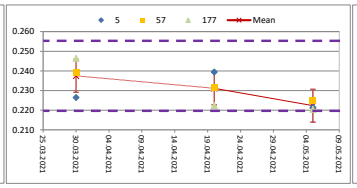
Appendix 4 Data of Stability Test

	Compulsory Compounds																	
	Bromide ion						Ethephon						Glufosinate					
AV [mg/kg]	21.3						0.228						0.216					
Date	30.03.2021		20.04.2021		05.05.2021		30.03.2021		20.04.2021		05.05.2021		30.03.2021		20.04.2021		05.05.2021	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 005	20.08	19.22	19.73	19.69	18.00	19.93	0.231	0.222	0.247	0.232	0.223	0.220	0.205	0.209	0.223	0.197	0.217	0.220
No. 057	22.05	21.11	19.32	17.57	18.43	19.69	0.246	0.233	0.226	0.237	0.223	0.227	0.229	0.224	0.206	0.205	0.215	0.218
No. 177	20.48	19.77	18.44	19.88	19.38	20.16	0.247	0.246	0.221	0.224	0.213	0.228	0.233	0.229	0.202	0.207	0.205	0.205
Mean [mg/kg]	20.45		19.10		19.26		0.238		0.231		0.222		0.221		0.207		0.213	
RSD* [%]	4.9%		3.3%		2.3%		4.3%		3.7%		1.1%		5.6%		1.4%		3.4%	
Diviation [%] (ref. 1st Anaylsis)	—		-6.6%		-5.8%		—		-2.7%		-6.4%		—		-6.6%		-3.8%	

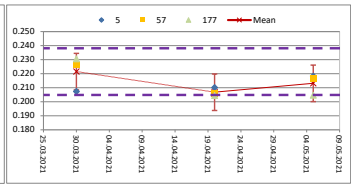
Bromide ion



Ethephon



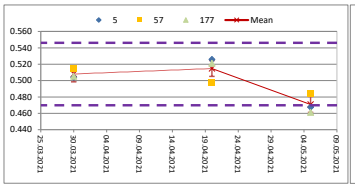
Glufosinate



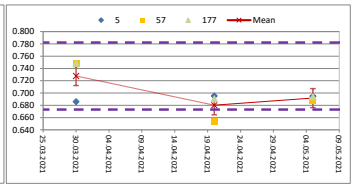
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

	Compulsory Compounds											
	Glyphosate						Phosphonic acid					
AV [mg/kg]	0.510						0.676					
Date	30.03.2021		20.04.2021		05.05.2021		30.03.2021		20.04.2021		05.05.2021	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 005	0.509	0.499	0.558	0.494	0.475	0.460	0.704	0.668	0.696	0.695	0.691	0.698
No. 057	0.534	0.495	0.492	0.503	0.489	0.479	0.744	0.753	0.654	0.655	0.674	0.700
No. 177	0.521	0.490	0.502	0.540	0.470	0.452	0.740	0.757	0.675	0.706	0.678	0.710
Mean [mg/kg]	0.508		0.515		0.471		0.728		0.680		0.692	
RSD* [%]	1.2%		2.9%		2.5%		5.0%		3.3%		0.6%	
Diviation [%] (ref. 1st Anaylsis)	—		1.3%		-7.3%		—		-6.5%		-4.9%	

Glyphosate



Phosphonic acid



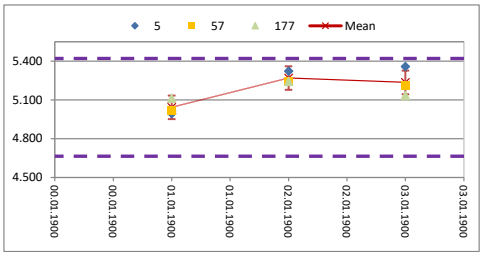
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

* RSD = relative standard diviation

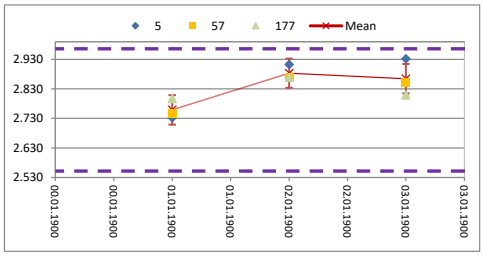
Appendix 4 (cont.): Data of Stability Test

	Optional Compounds											
	2-CE						EO (sum)					
AV [mg/kg]	0.449‡						0.250‡					
Date	30.03.2021		23.04.2021		07.05.2021		30.03.2021		23.04.2021		07.05.2021	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 005	4.93	5.05	5.26	5.39	5.29	5.43	2.70	2.77	2.88	2.95	2.89	2.97
No. 057	5.01	5.03	5.43	5.06	5.36	5.07	2.74	2.75	2.97	2.77	2.93	2.77
No. 177	5.18	5.05	5.07	5.42	4.92	5.35	2.83	2.76	2.77	2.96	2.69	2.93
Mean [mg/kg]	**5.04**		**5.27**		**5.24**		**2.76**		**2.88**		**2.86**	
RSD* [%]	**1.3%**		**0.9%**		**2.2%**		**1.2%**		**0.9%**		**2.2%**	
Diviation [%] (ref. 1st Anaylsis)	—		**4.5%**		**3.8%**		—		**4.5%**		**3.8%**	
	‡ AV based on sub-population of results generated by methods including water addition only											

2-CE



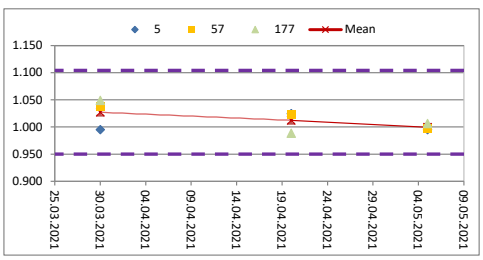
EO (sum)



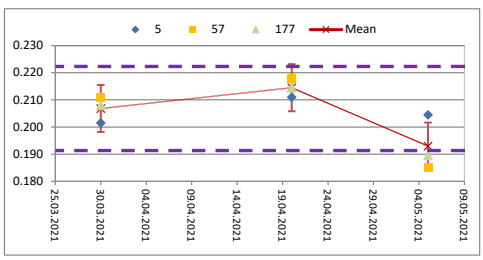
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

	Optional Compounds											
	Chlorate						Diquat					
AV [mg/kg]	1.03						0.244					
Date	30.03.2021		20.04.2021		05.05.2021		30.03.2021		20.04.2021		05.05.2021	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 005	0.982	1.008	1.053	0.996	0.998	0.992	0.218	0.185	0.211	0.211	0.204	0.205
No. 057	1.057	1.017	1.016	1.032	0.984	1.009	0.215	0.207	0.215	0.221	0.188	0.182
No. 177	1.052	1.046	0.966	1.011	1.018	0.995	0.208	0.208	0.212	0.217	0.187	0.192
Mean [mg/kg]	**1.027**		**1.012**		**0.999**		**0.207**		**0.215**		**0.193**	
RSD* [%]	**2.8%**		**2.0%**		**0.6%**		**2.3%**		**1.6%**		**5.3%**	
Diviation [%] (ref. 1st Anaylsis)	—		**-1.4%**		**-2.7%**		—		**3.7%**		**-6.7%**	

Chlorate



Diquat



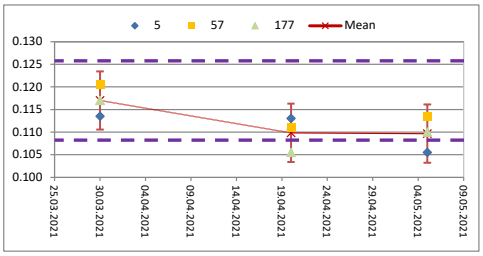
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

* RSD = relative standard diviation

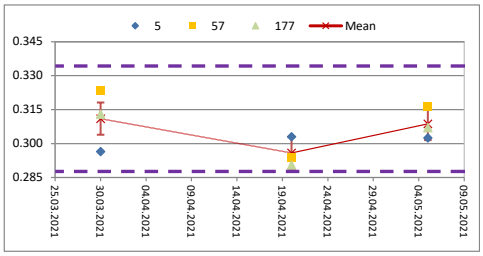
Appendix 4 (cont.): Data of Stability Test

	Optional Compounds											
	Matrine						**Nicotine**					
AV [mg/kg]	0.124						0.255					
Date	30.03.2021		20.04.2021		05.05.2021		30.03.2021		20.04.2021		05.05.2021	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 005	0.114	0.113	0.117	0.109	0.101	0.110	0.307	0.286	0.306	0.300	0.309	0.296
No. 057	0.121	0.120	0.113	0.109	0.108	0.119	0.326	0.321	0.293	0.295	0.304	0.329
No. 177	0.121	0.113	0.102	0.109	0.103	0.117	0.318	0.308	0.287	0.294	0.297	0.317
Mean [mg/kg]	**0.117**		**0.110**		**0.110**		**0.311**		**0.296**		**0.309**	
RSD* [%]	**3.0%**		**3.5%**		**3.7%**		**4.4%**		**2.2%**		**2.3%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-6.1%**		**-6.3%**		**—**		**-4.9%**		**-0.8%**	

Martine



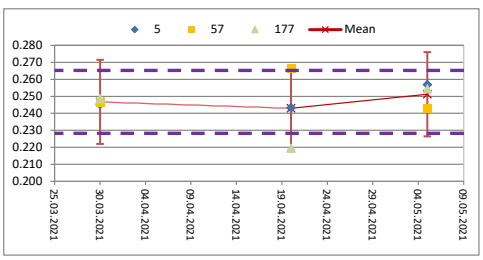
Nicotine



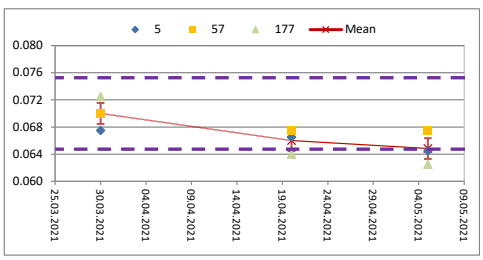
– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard diviation based on FPP-RSD of 25 %

	Optional Compounds											
	Paraquat						**Trimesium**					
AV [mg/kg]	0.264						0.073					
Date	30.03.2021		20.04.2021		05.05.2021		30.03.2021		20.04.2021		05.05.2021	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]		[mg/kg]	
No. 005	0.248	0.243	0.250	0.236	0.265	0.249	0.069	0.066	0.066	0.067	0.066	0.063
No. 057	0.255	0.238	0.264	0.268	0.267	0.218	0.070	0.070	0.067	0.068	0.066	0.069
No. 177	0.253	0.244	0.234	0.204	0.269	0.240	0.075	0.070	0.065	0.063	0.061	0.064
Mean [mg/kg]	**0.247**		**0.243**		**0.251**		**0.070**		**0.066**		**0.065**	
RSD* [%]	**0.7%**		**9.7%**		**3.0%**		**3.6%**		**2.7%**		**3.9%**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-1.5%**		**1.8%**		**—**		**-5.7%**		**-7.4%**	

Paraquat



Trimesium



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

* RSD = relative standard diviation

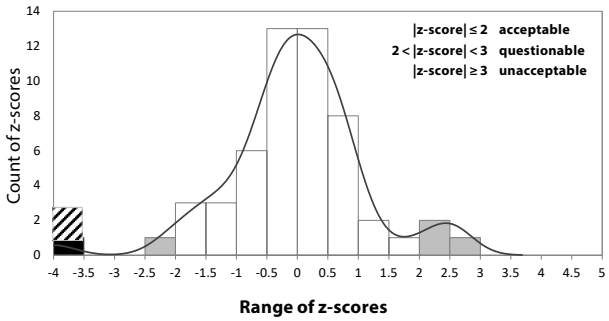
Appendix 5 Histograms and Kernel Density Estimates of z-score* Distributions

(Results from EU and EFTA Laboratories only)

Compulsory Compounds

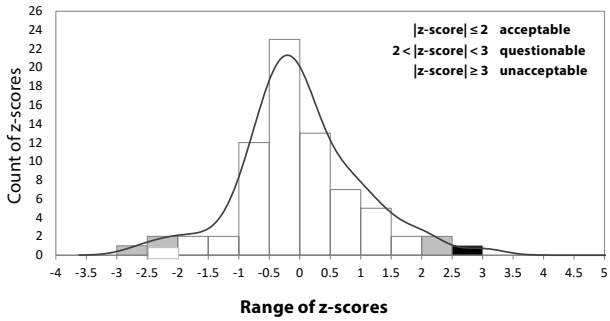
Bromide ion

(AV derived from entire population excl. one outlier)



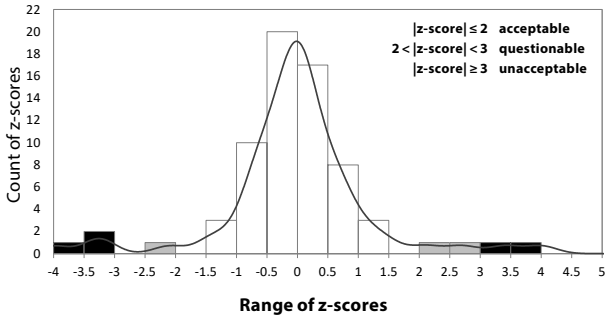
Ethephon

(AV derived from entire population excl. three outliers)



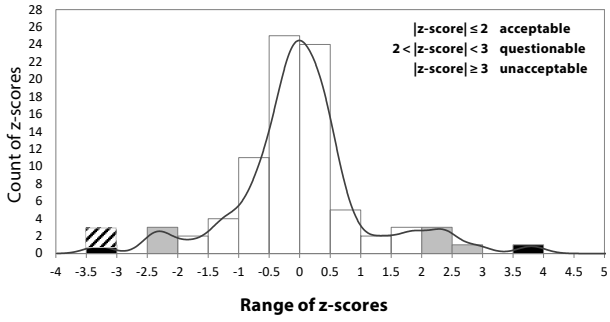
Glufosinate

(AV derived from entire population excl. four outliers)



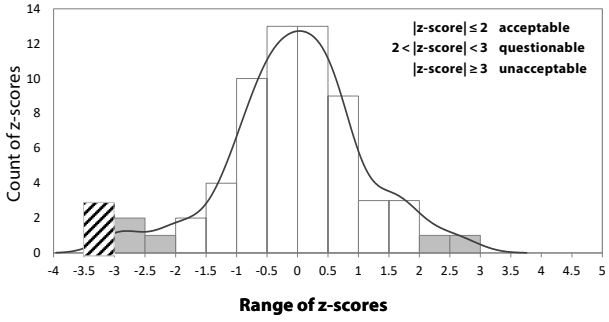
Glyphosate

(AV derived from entire population excl. one outlier)



Phosphonic acid

(AV derived from entire population)

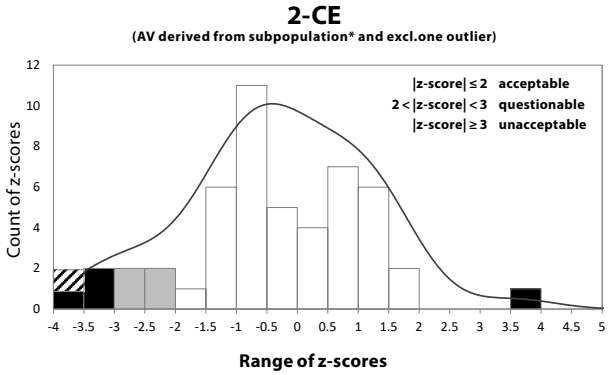


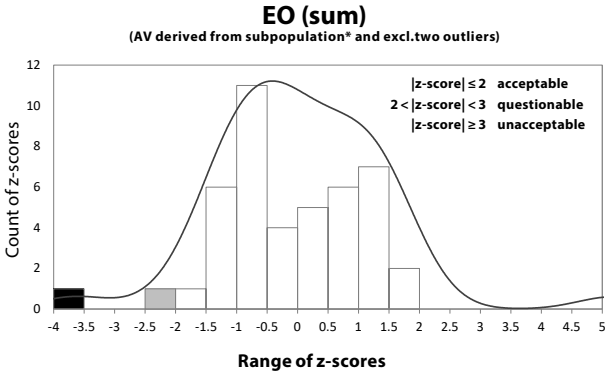
* Cut-off at z-score = 5; ▨: false negative results

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

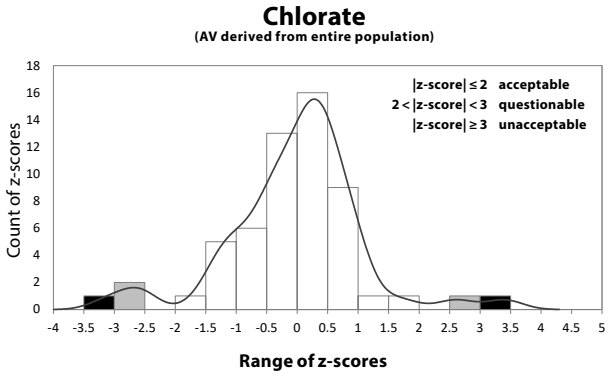
(Results from EU and EFTA Laboratories only)

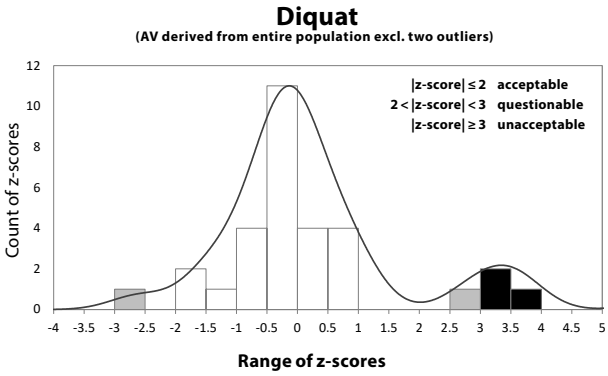
Optional Compounds

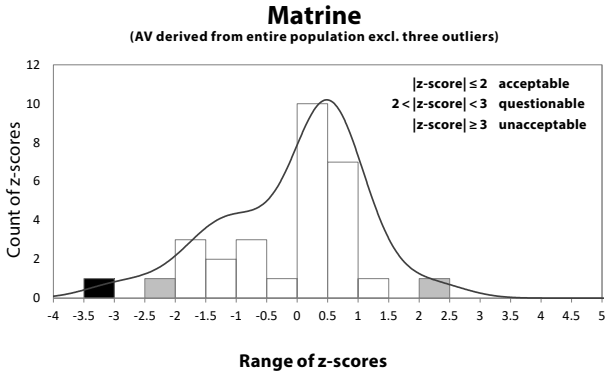


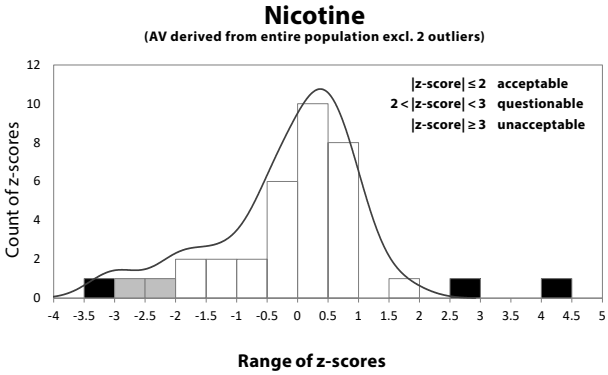


* The sub-population included results generated by methods including water addition only





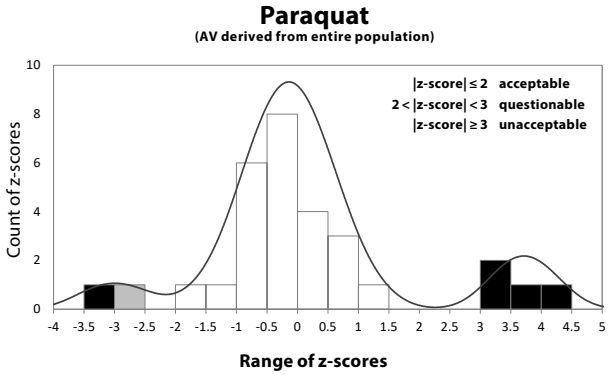


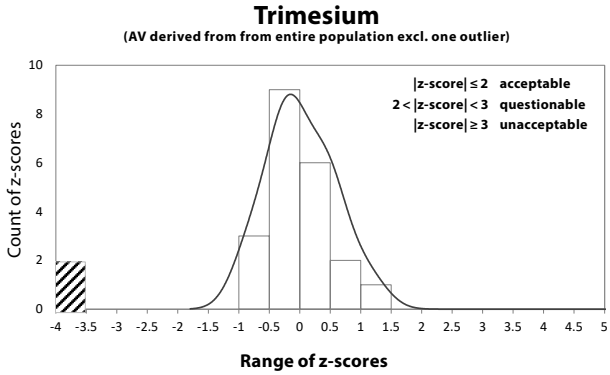


***** Cut-off at z-score = 5; ▨: false negative results

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

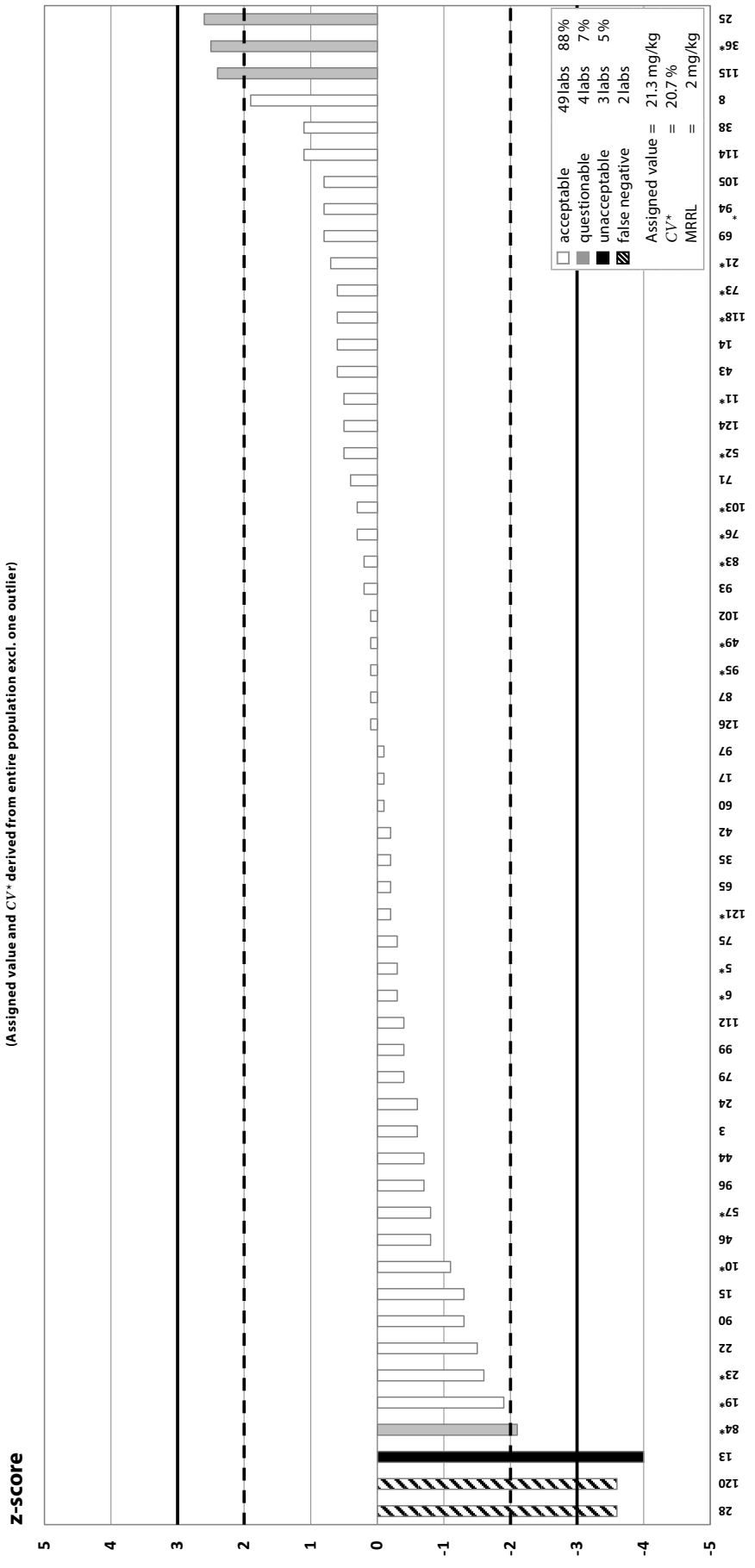
Optional Compounds



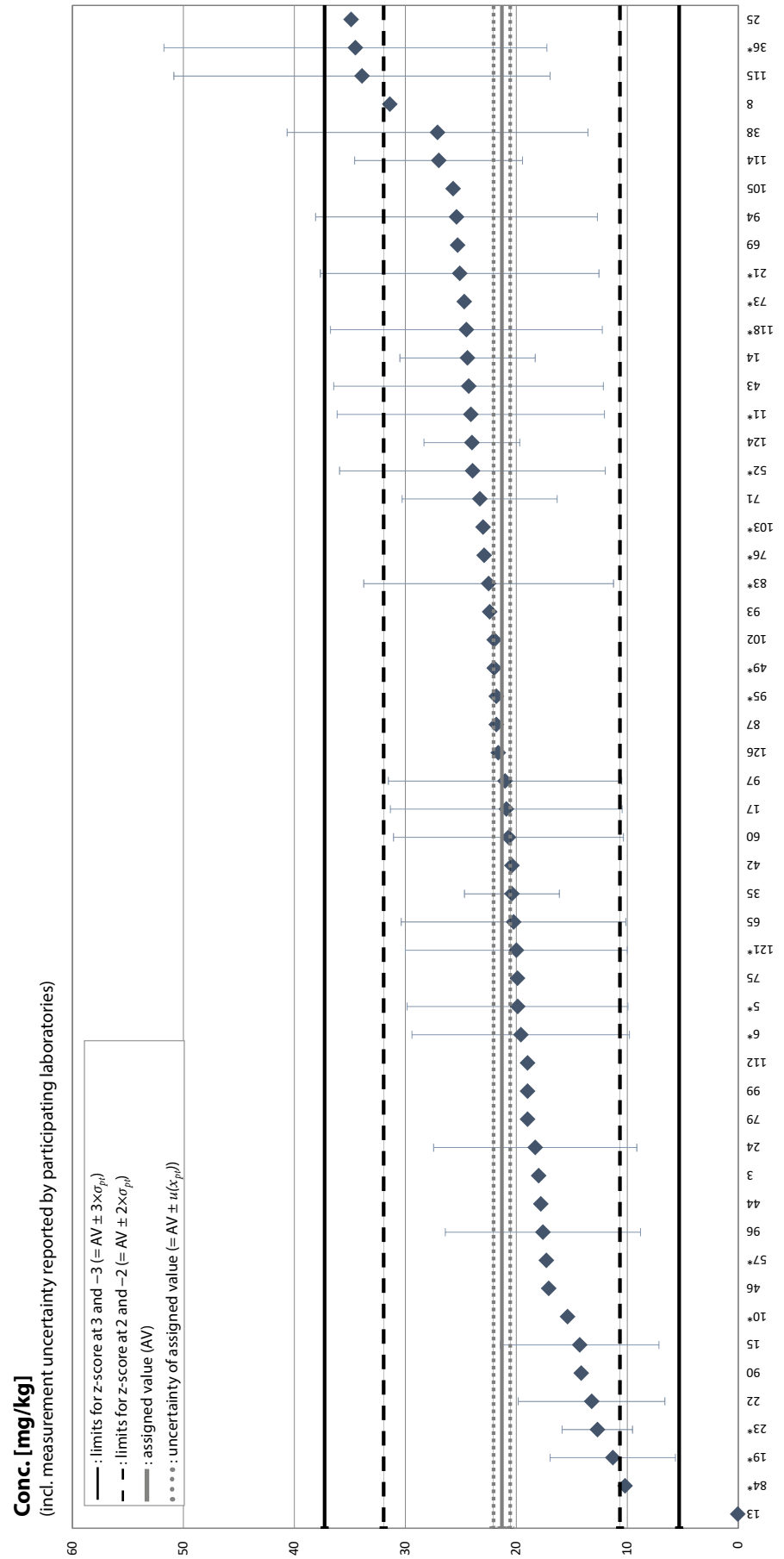


* Cut-off at z-score = 5; ▨: false negative results

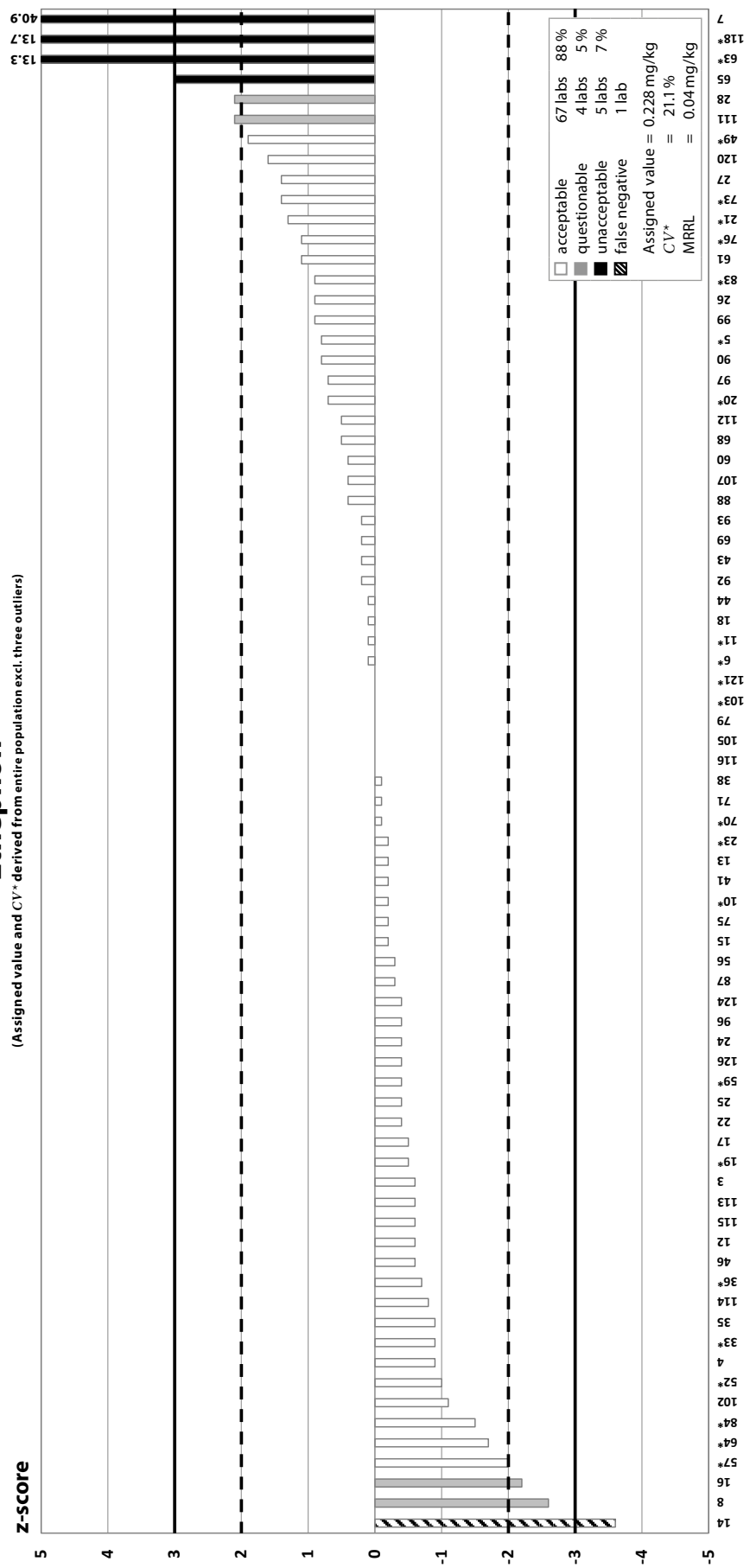
Appendix 6 Graphic Presentation of z-Scores: Compulsory Compounds(Results from EU and EFTA Laboratories only, * = NRL)



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)



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Z-SCORE DISTRIBUTION

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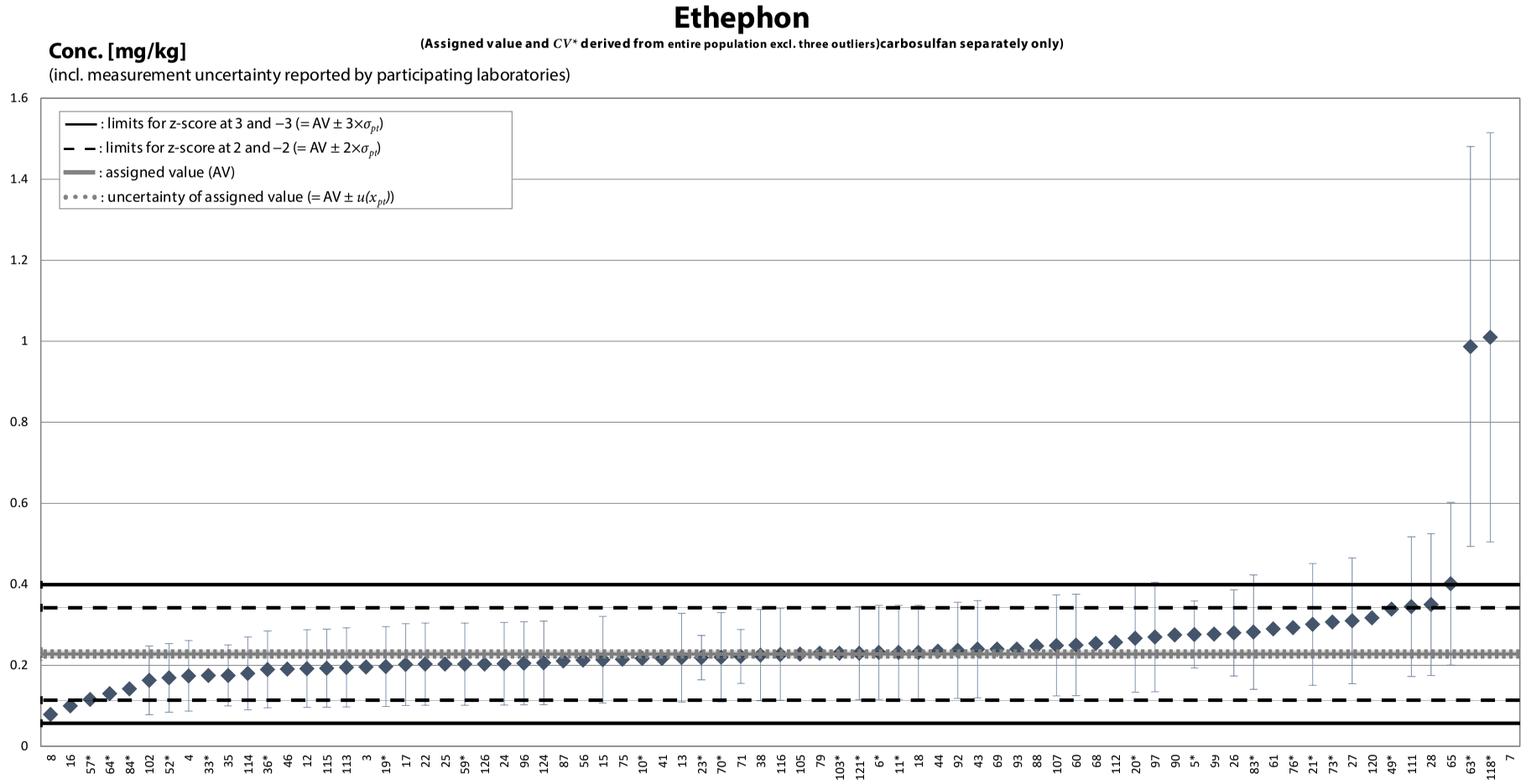
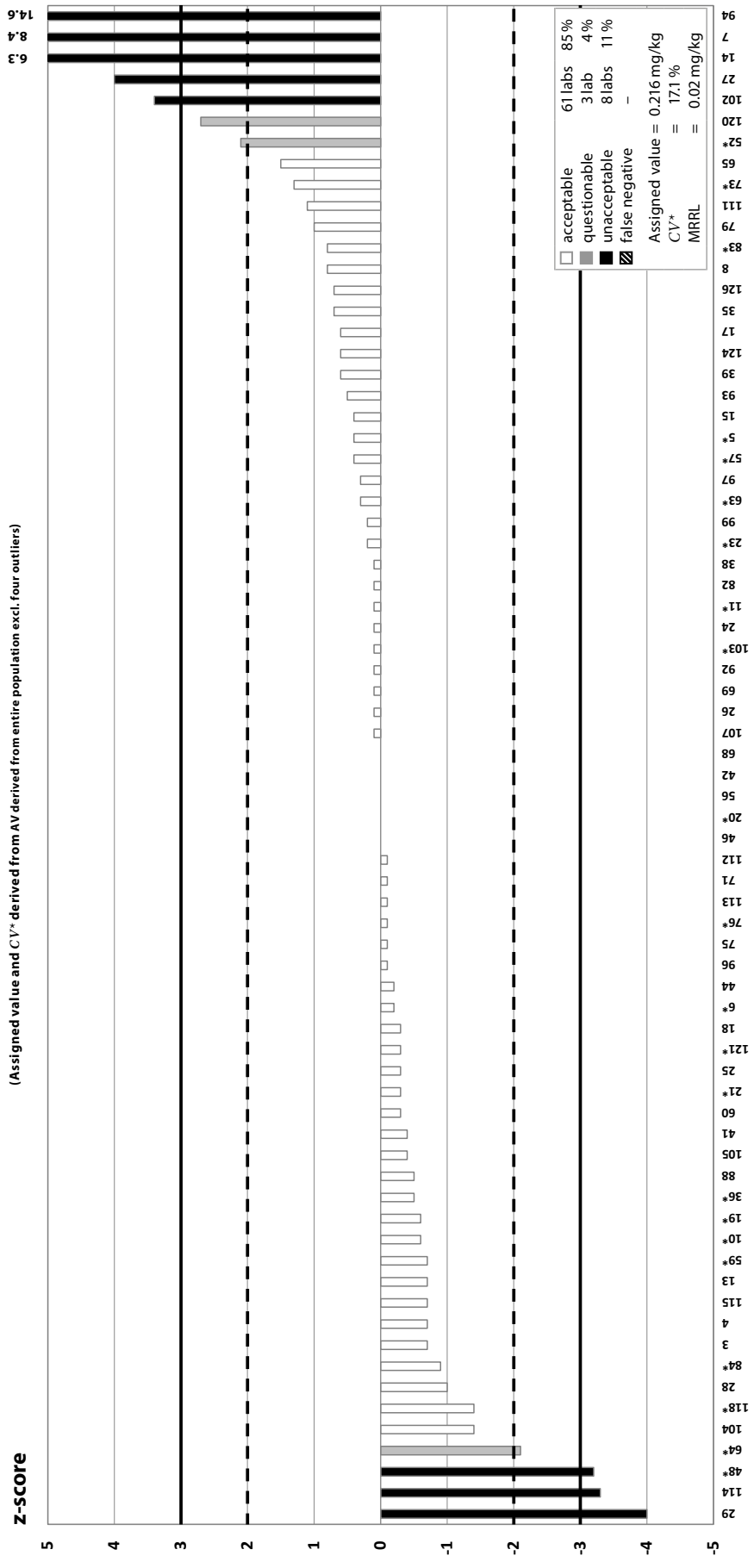
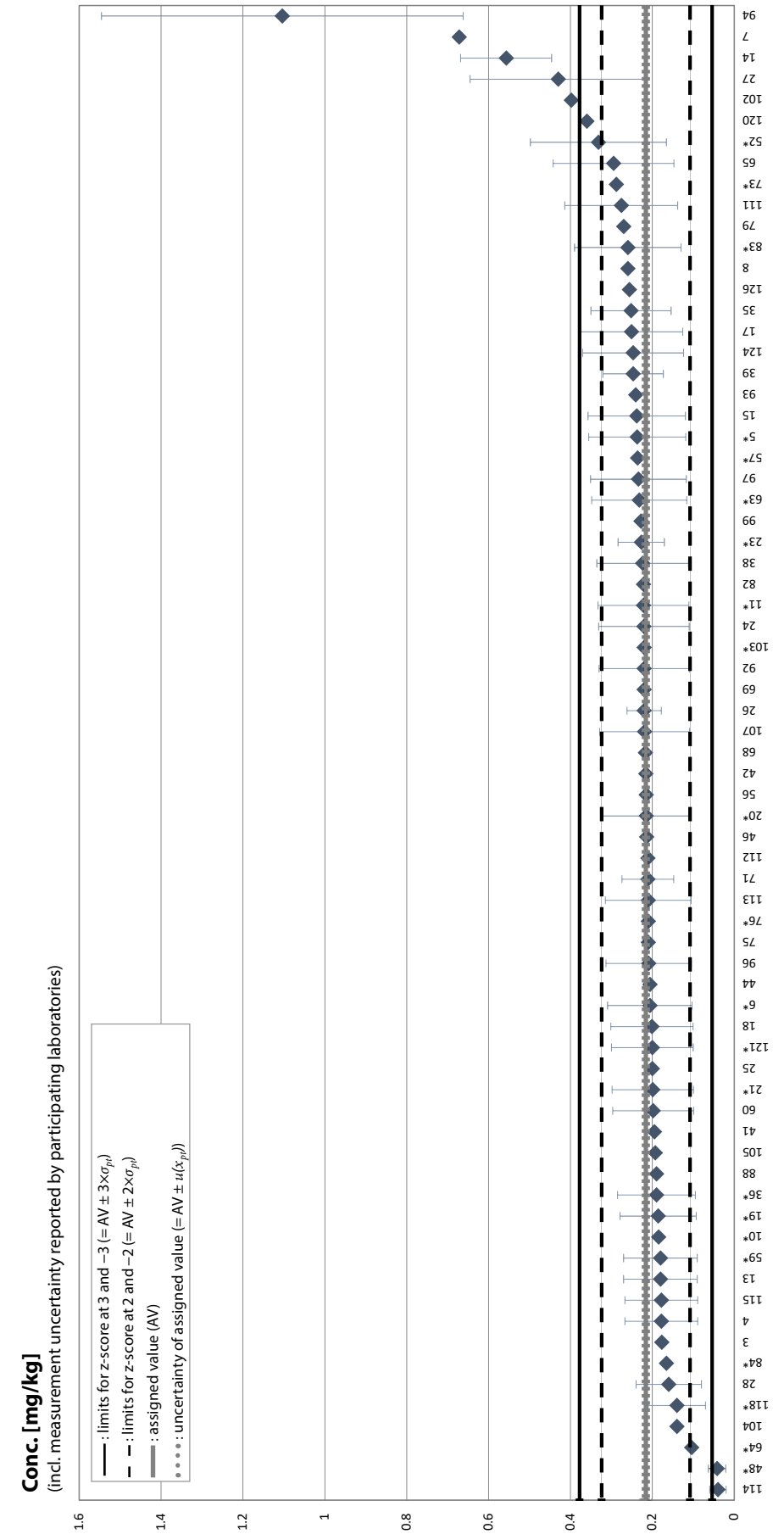
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



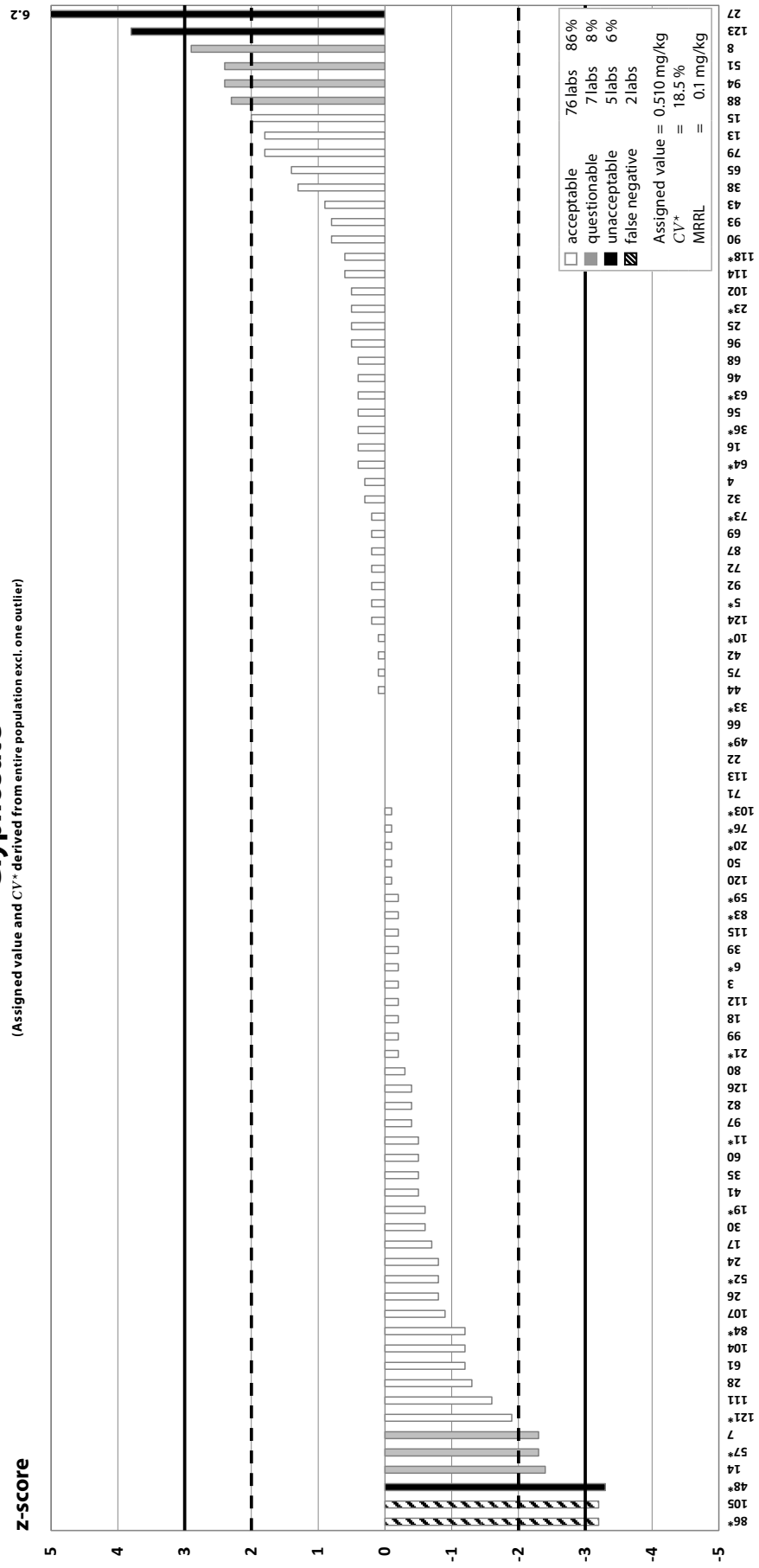
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



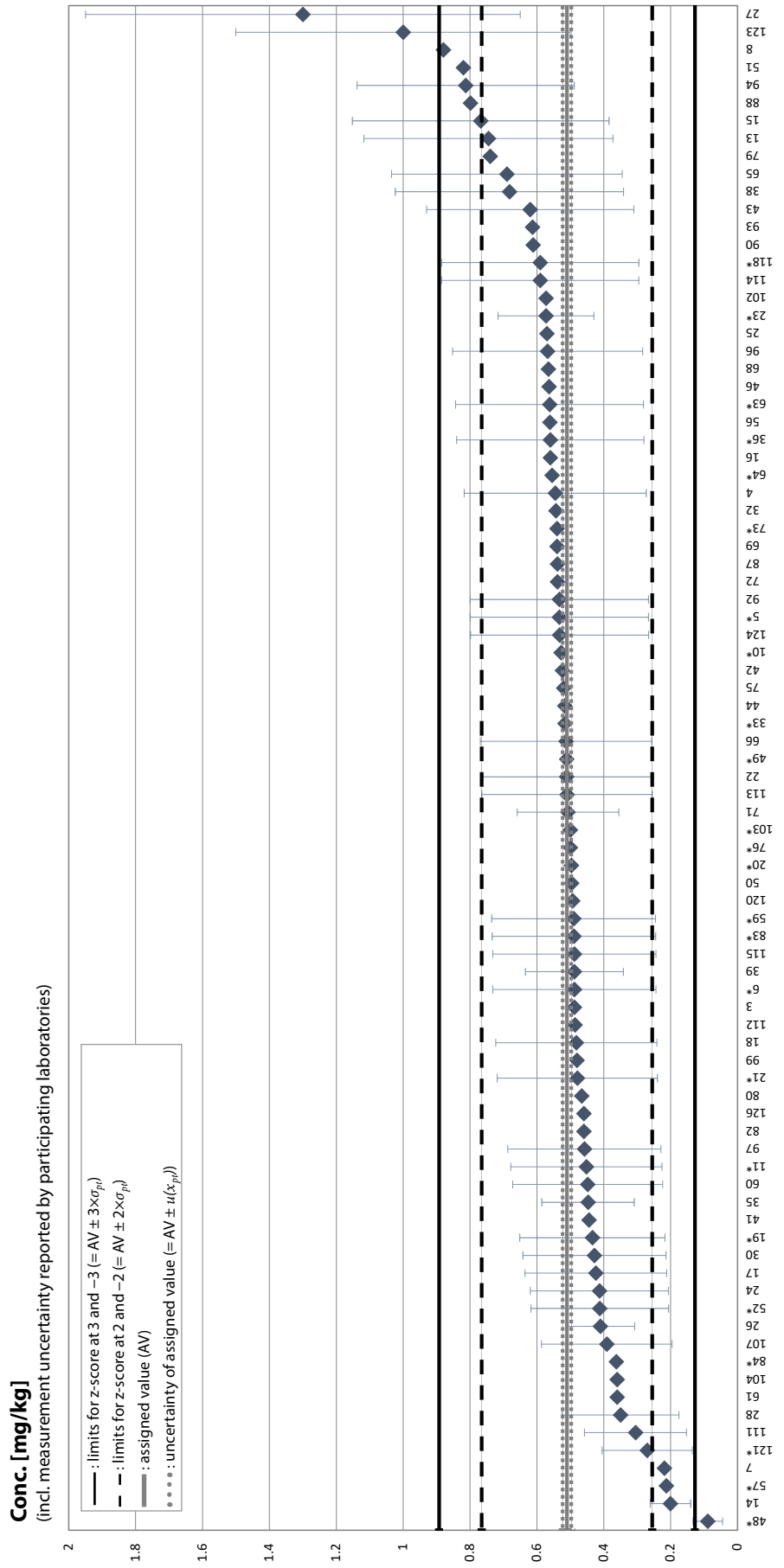
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



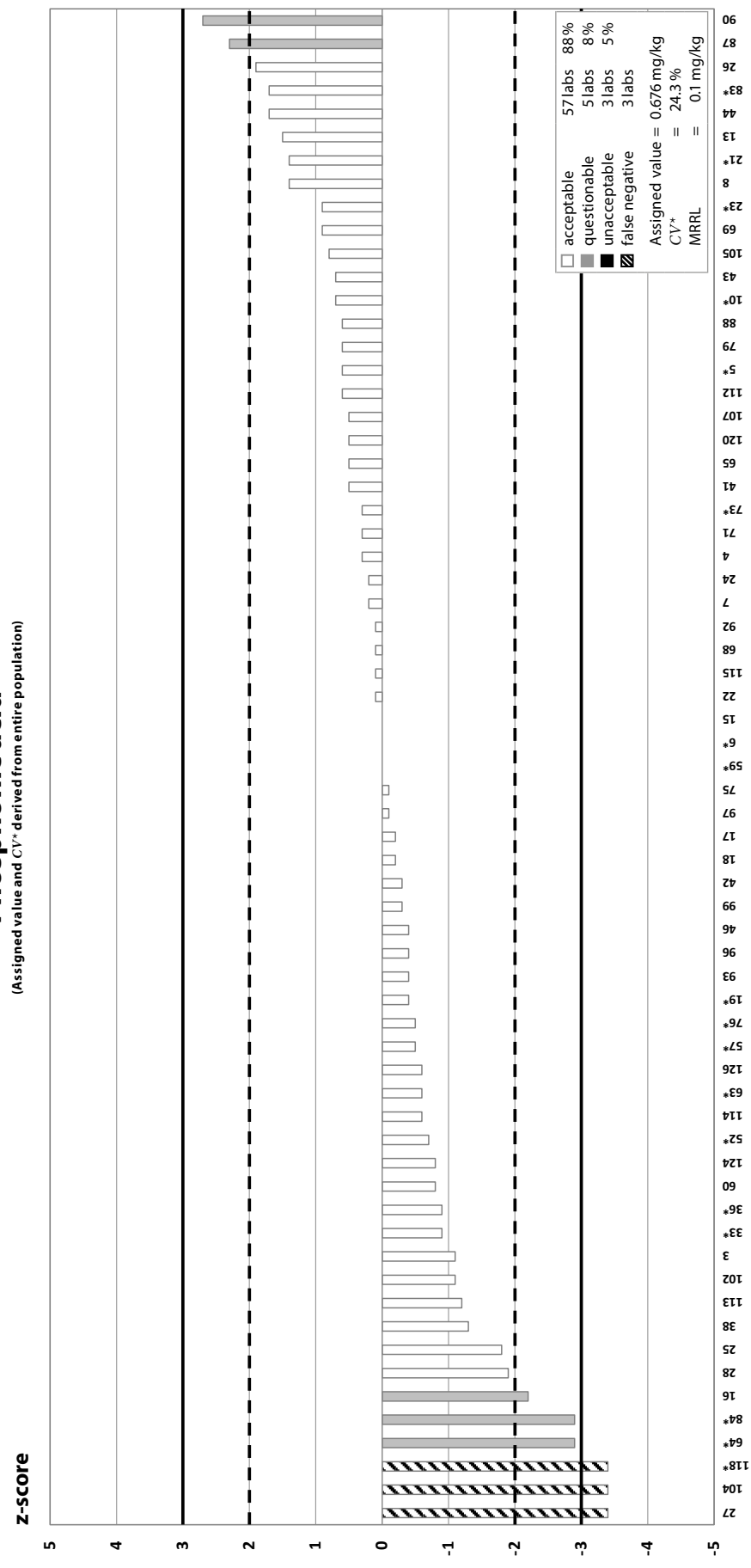
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



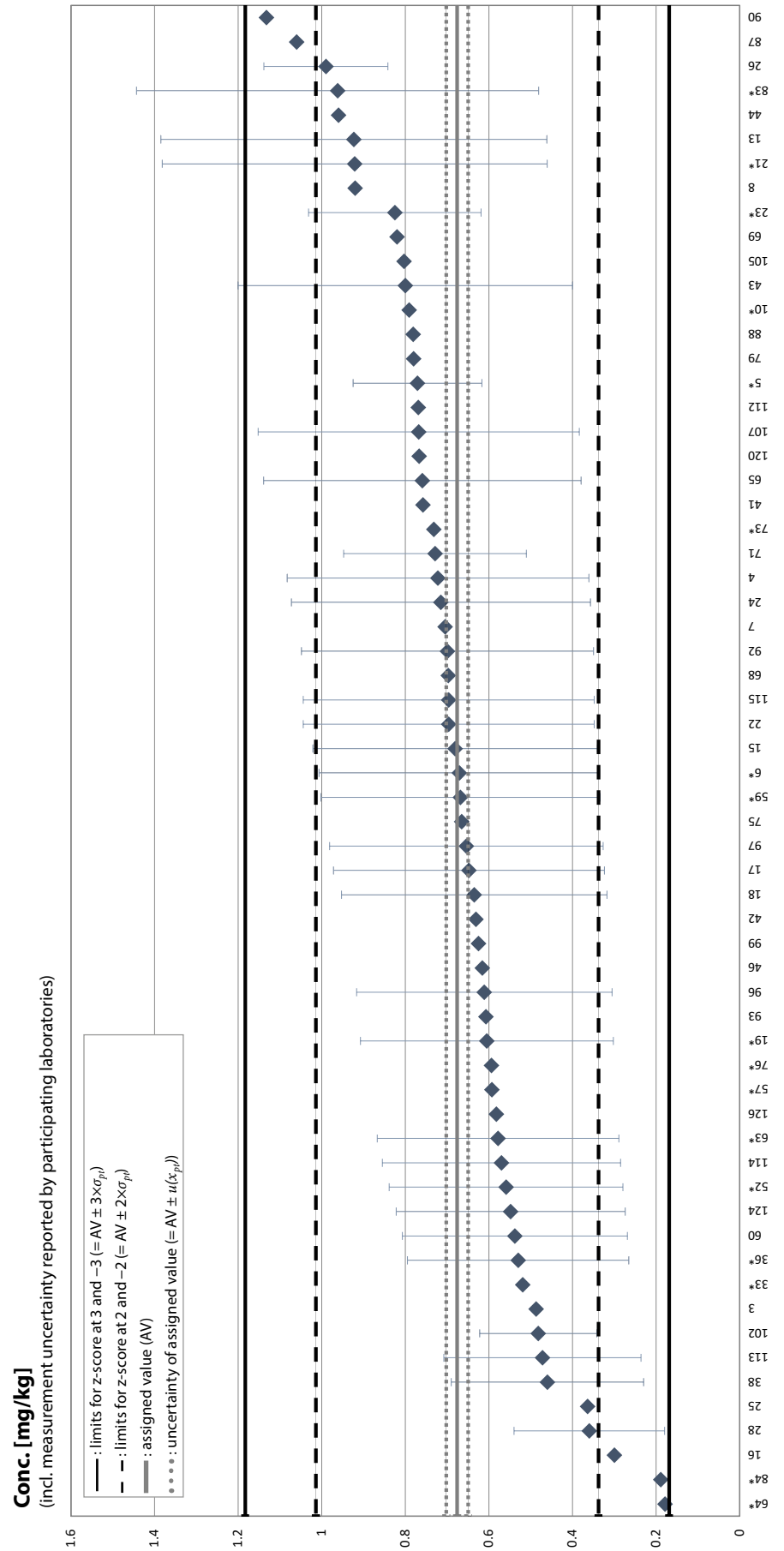
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



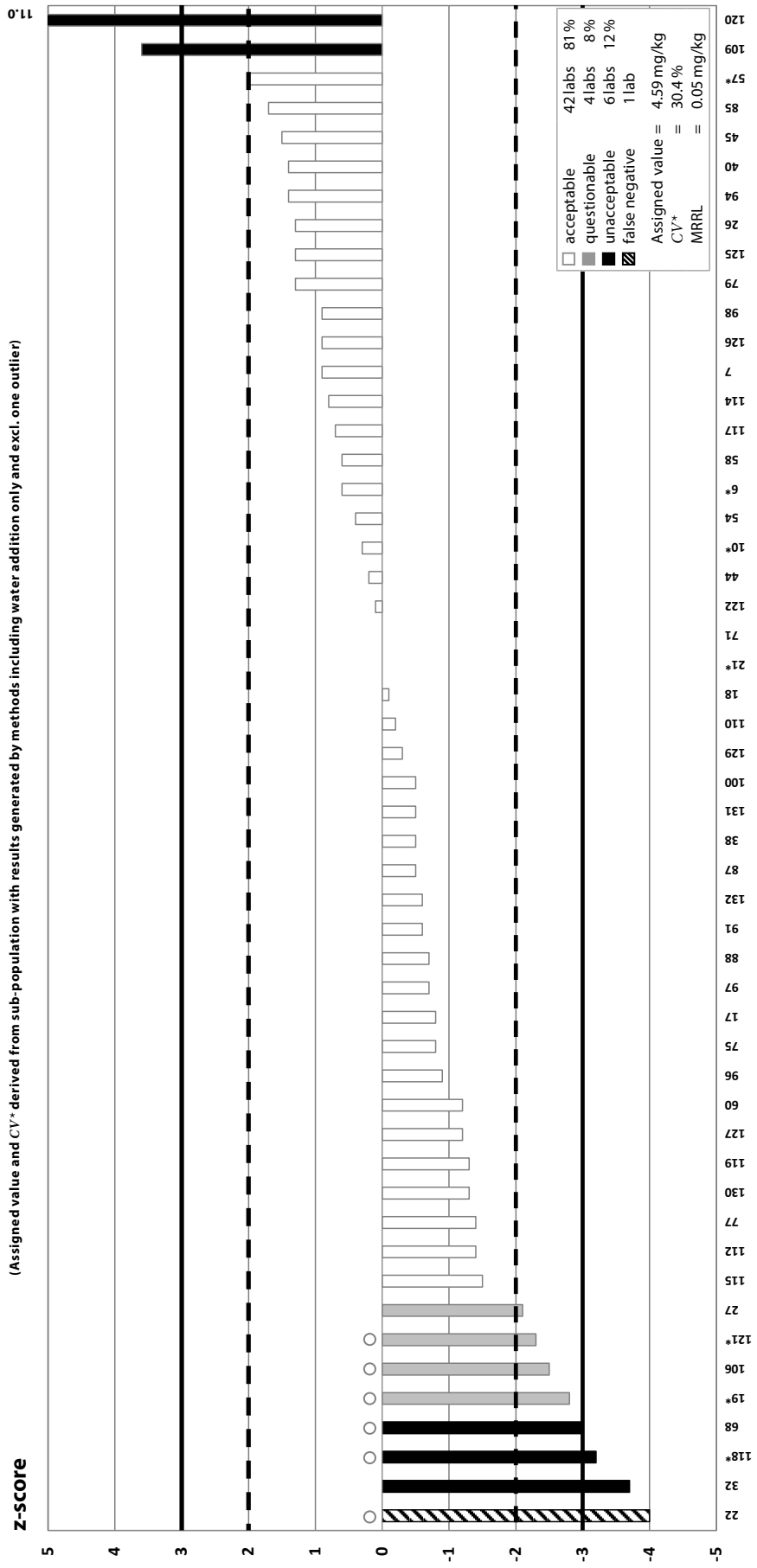
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



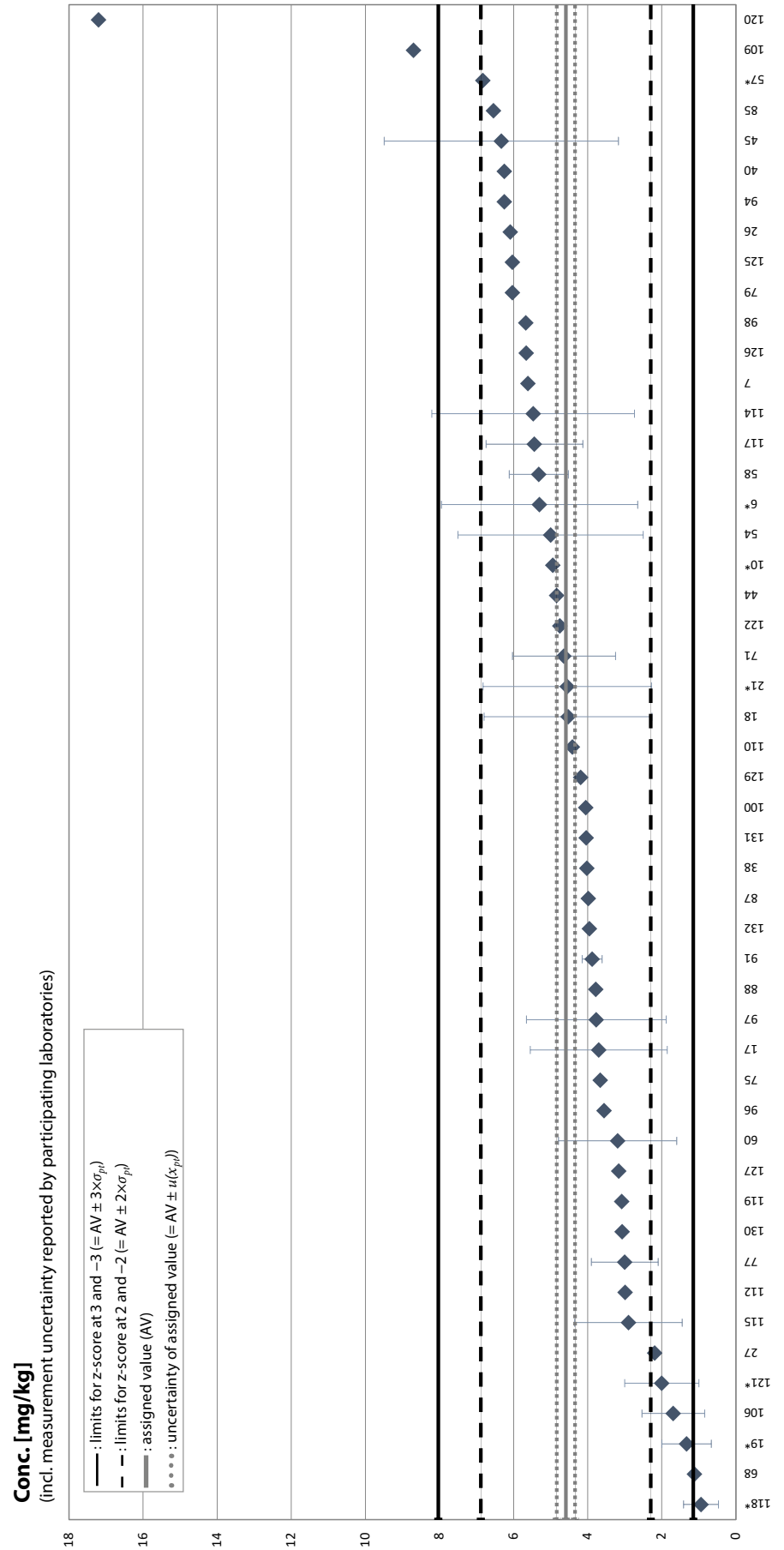
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



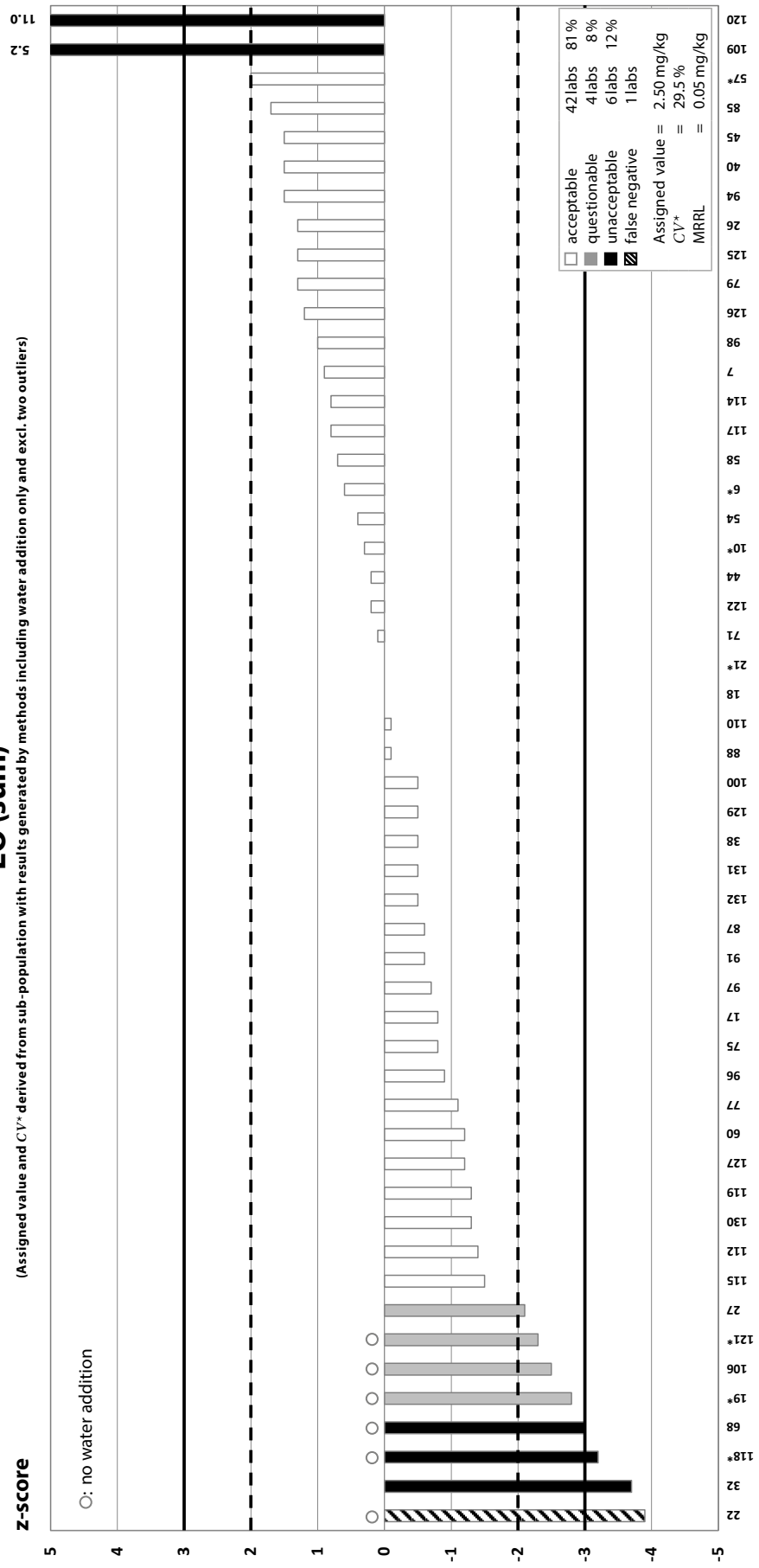
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



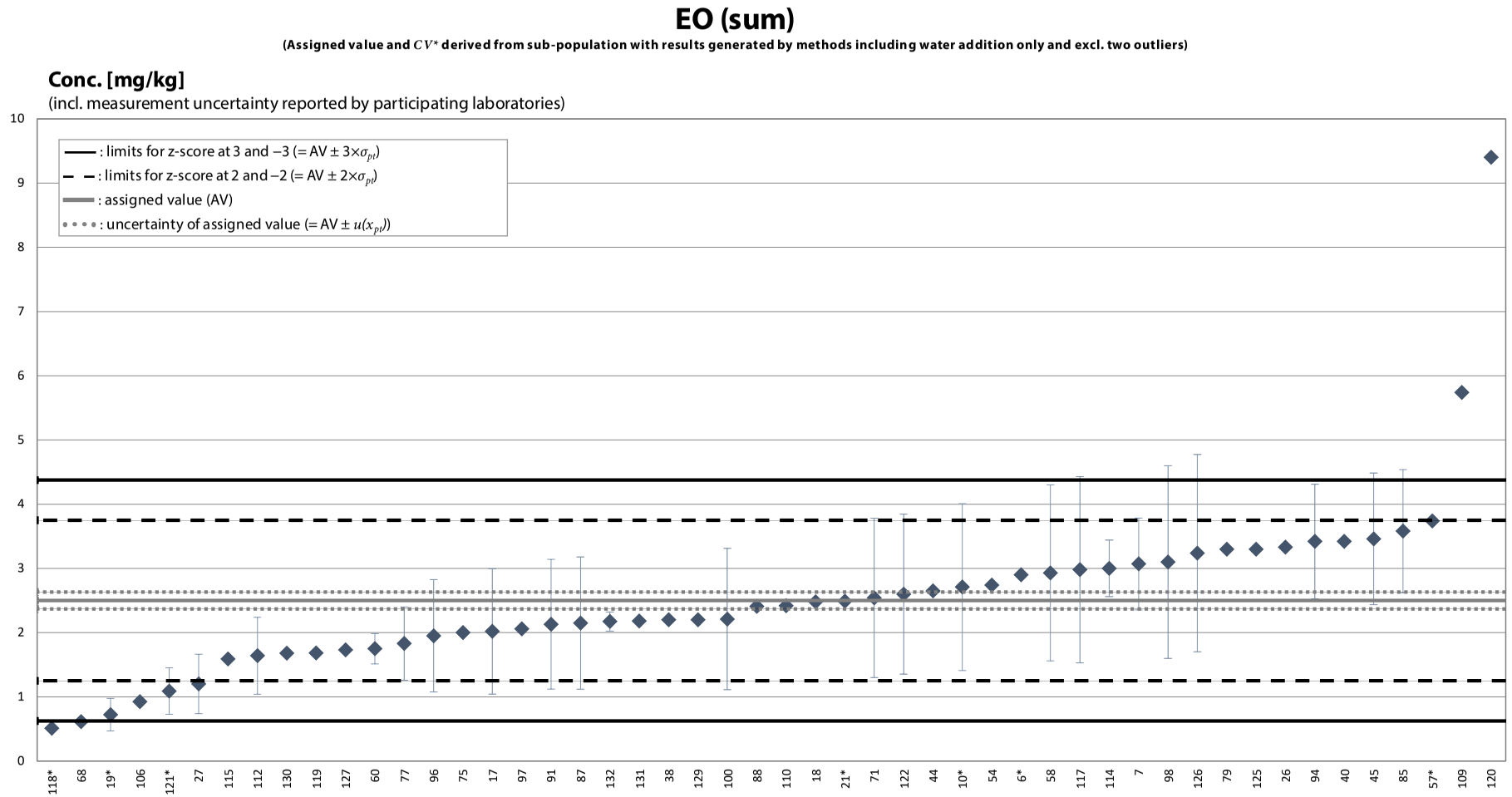
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



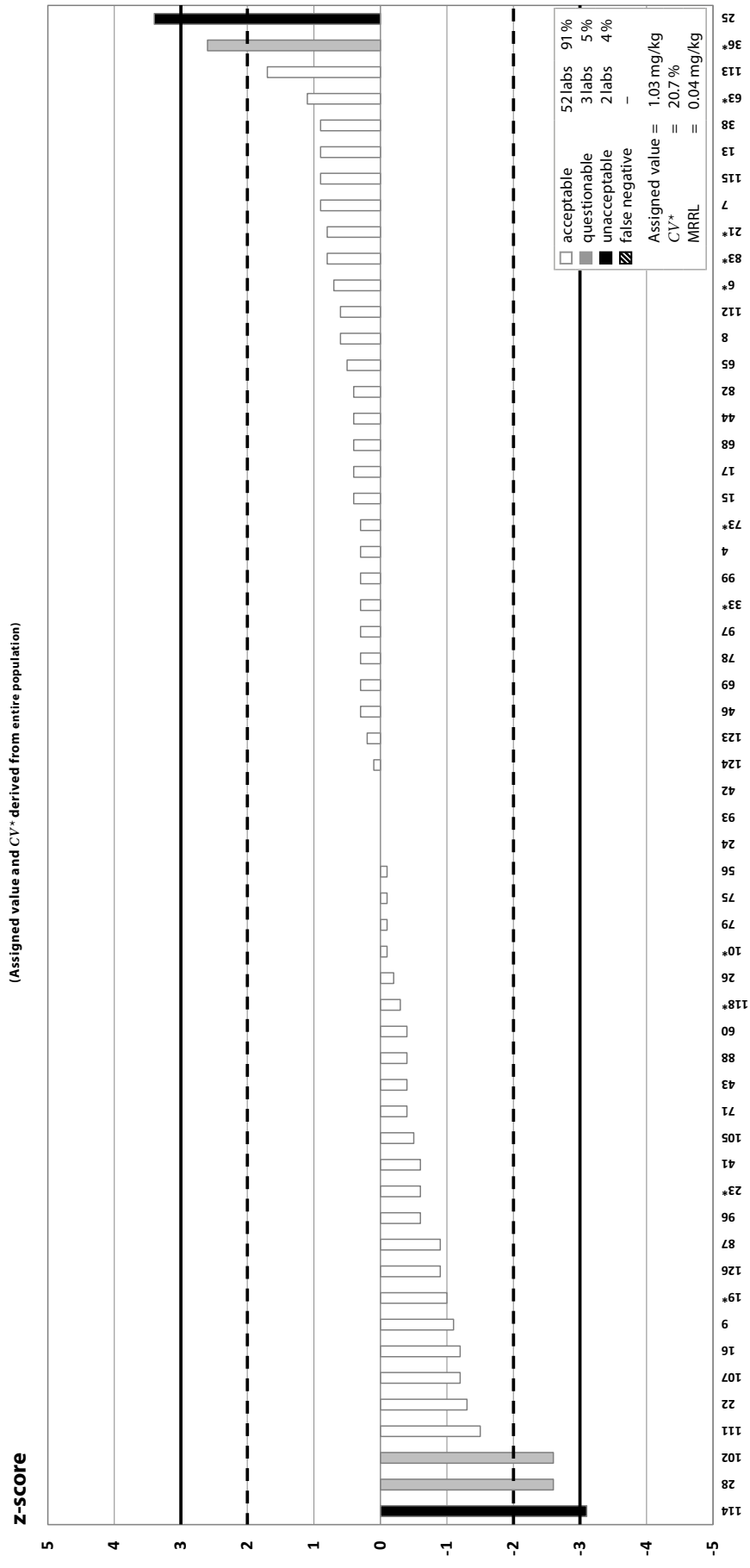
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



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)



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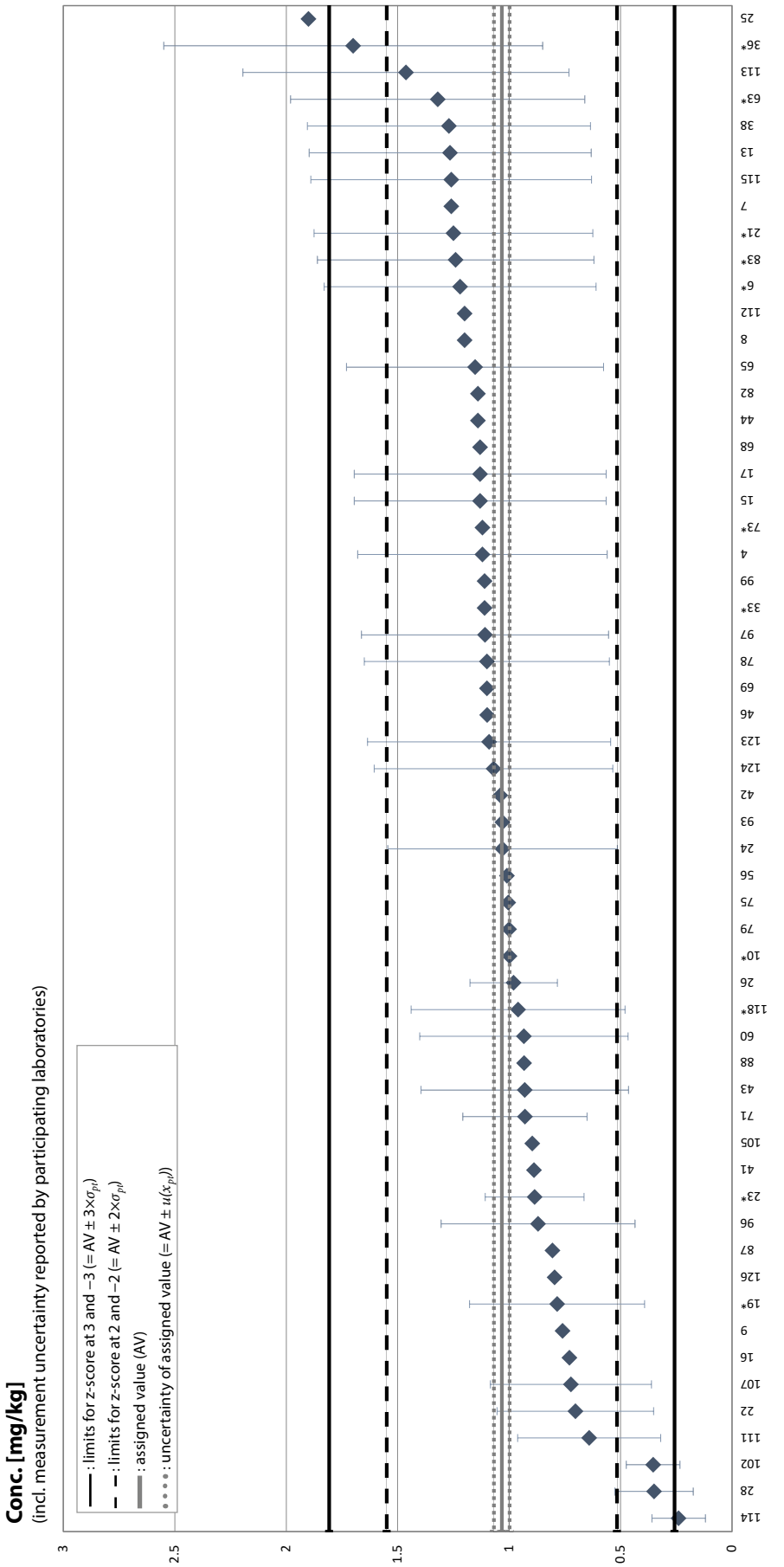
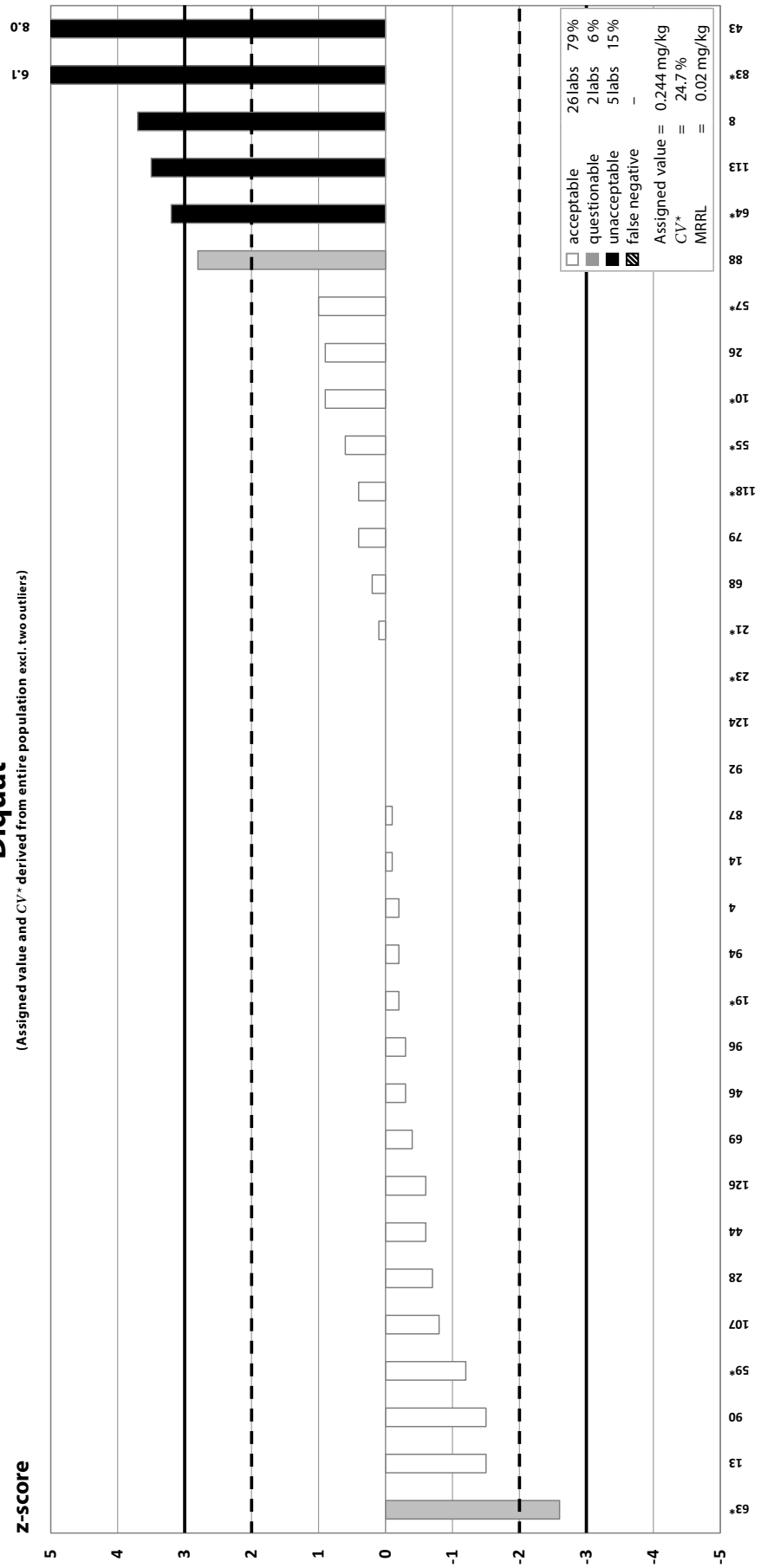
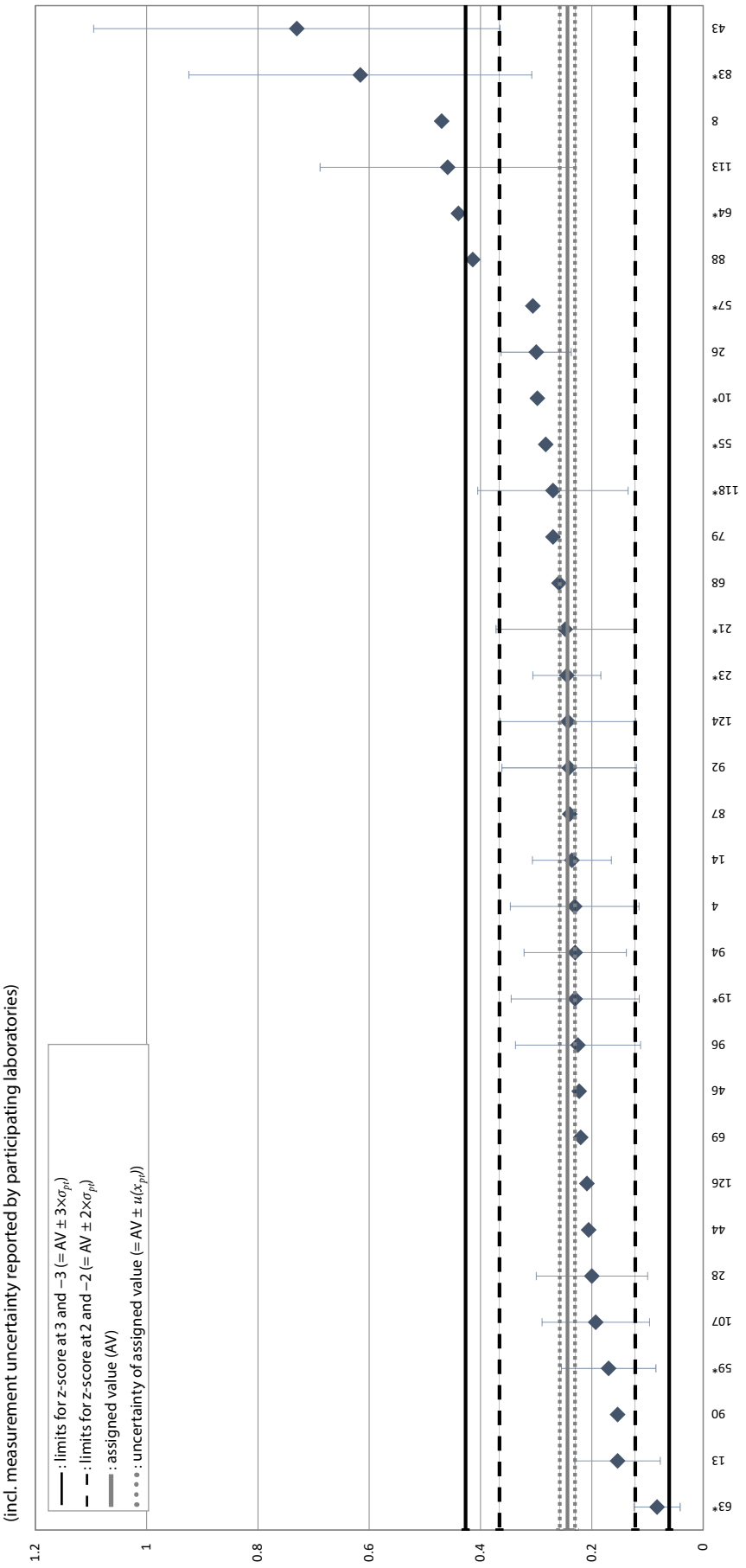
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



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)



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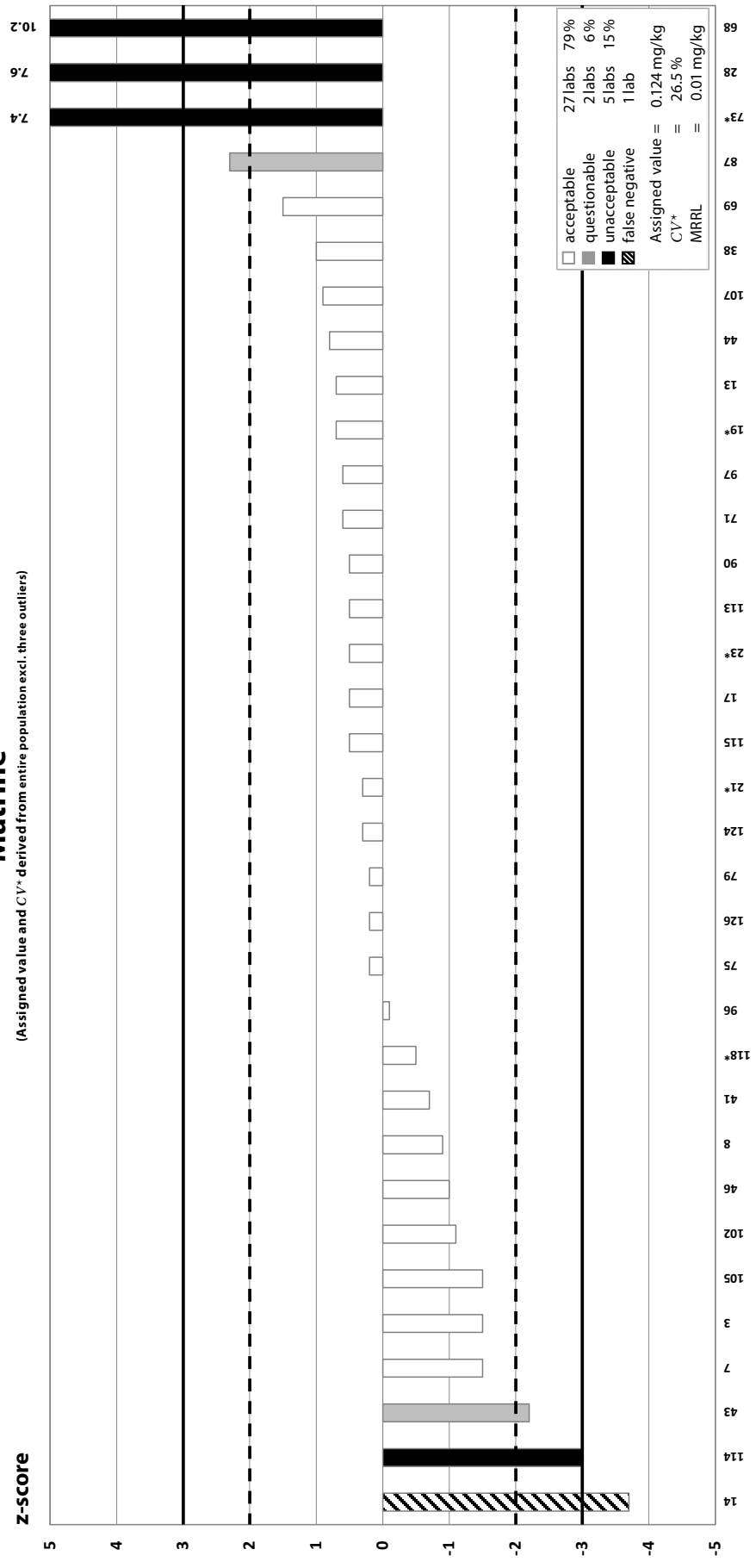
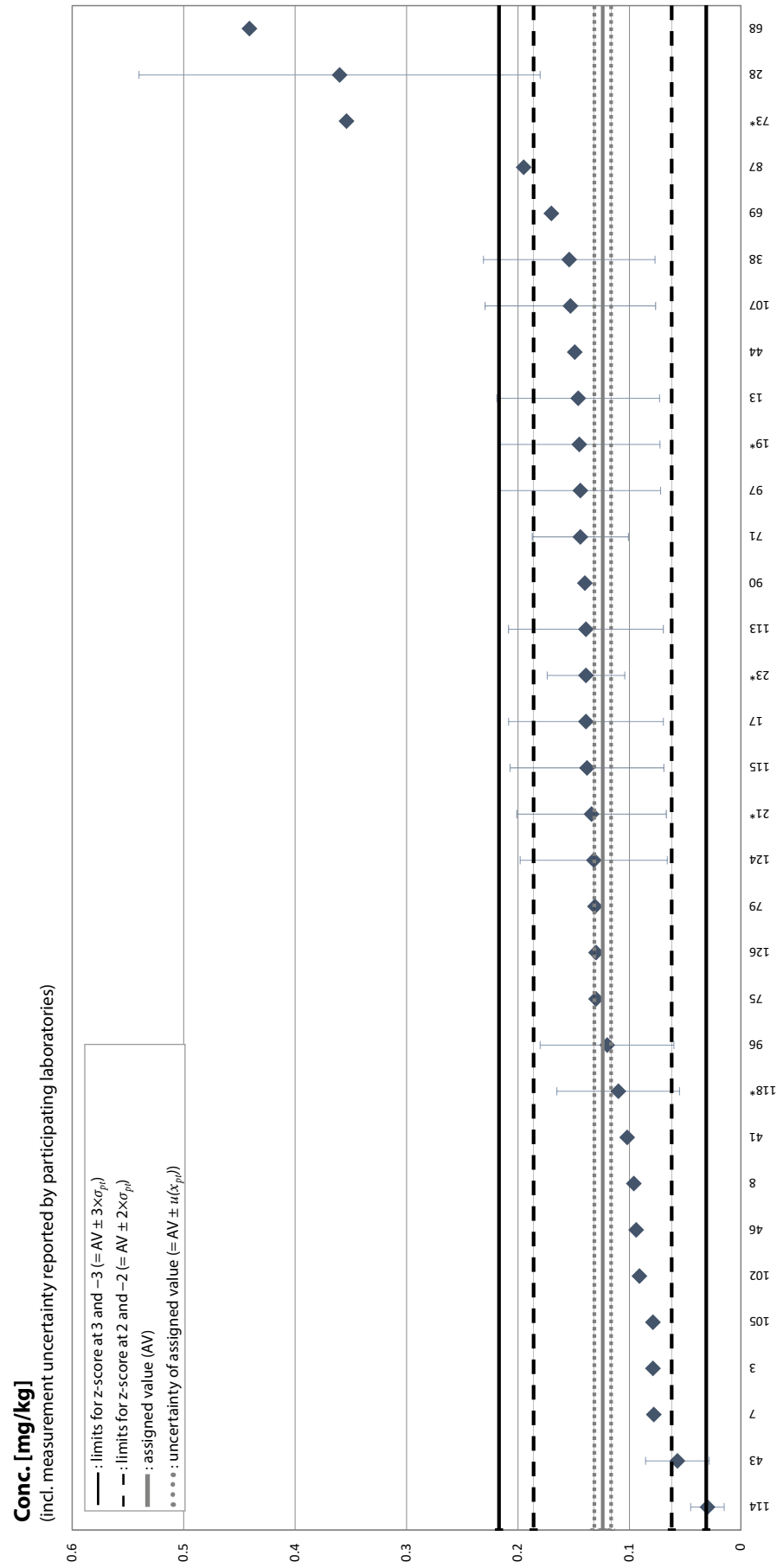
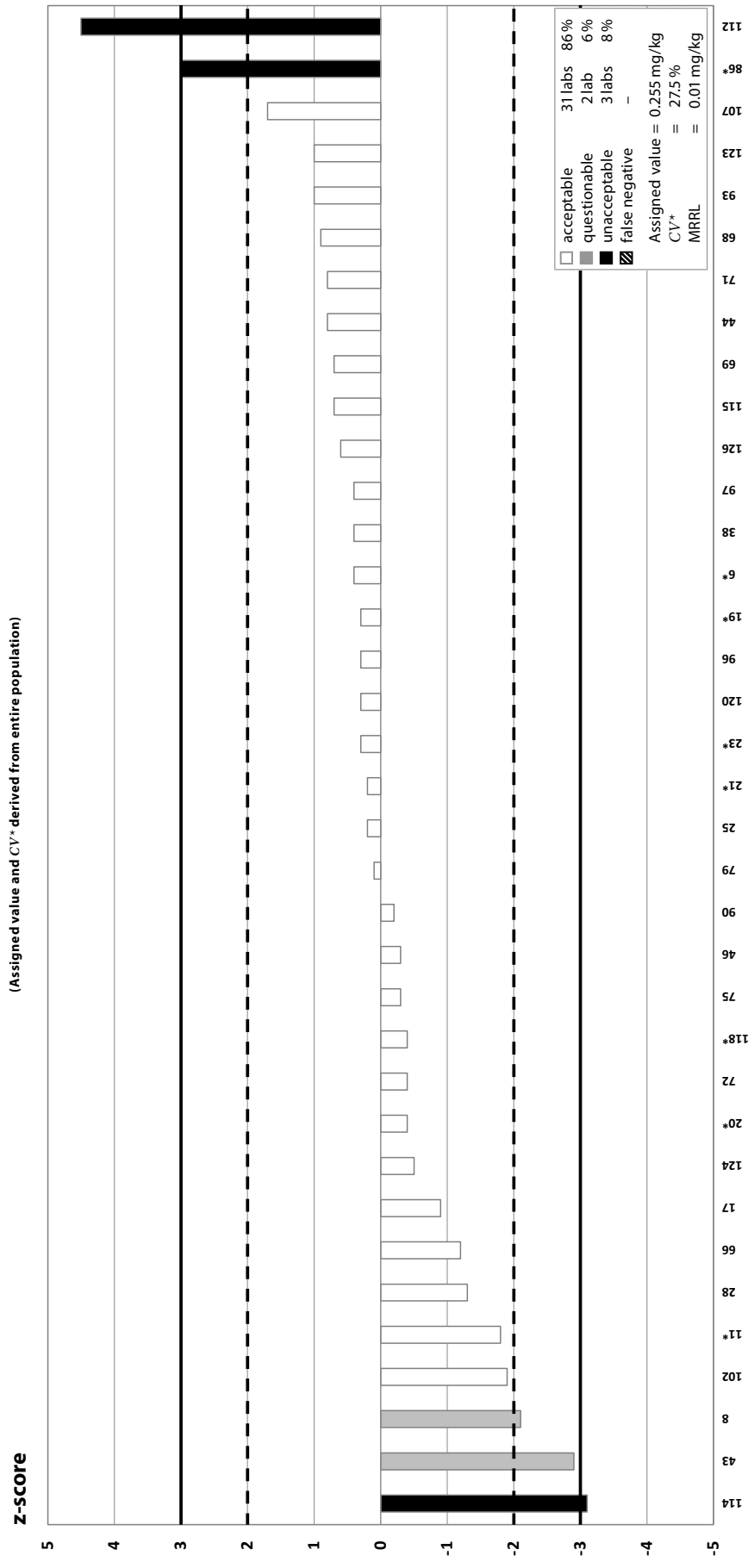
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



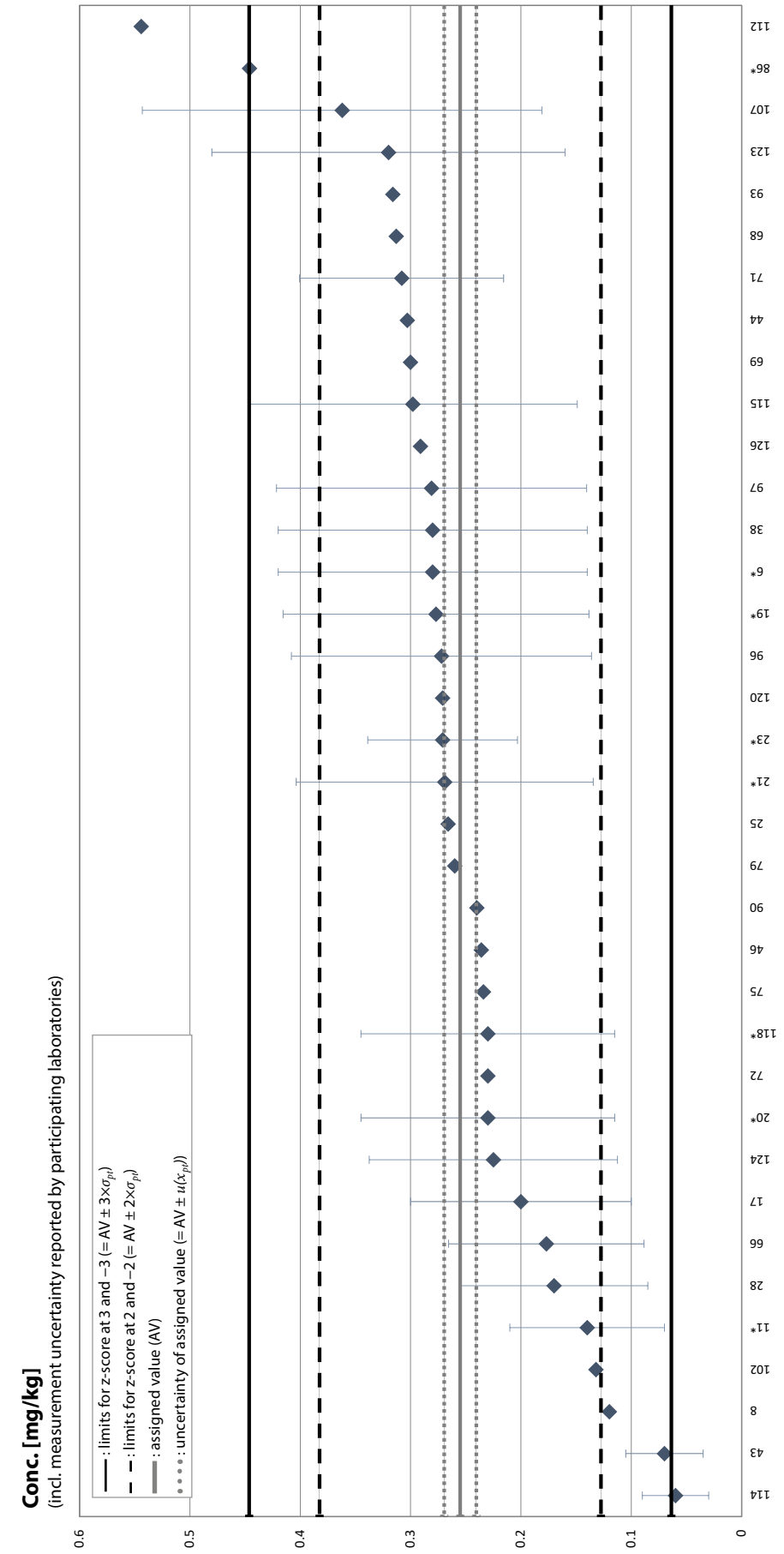
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



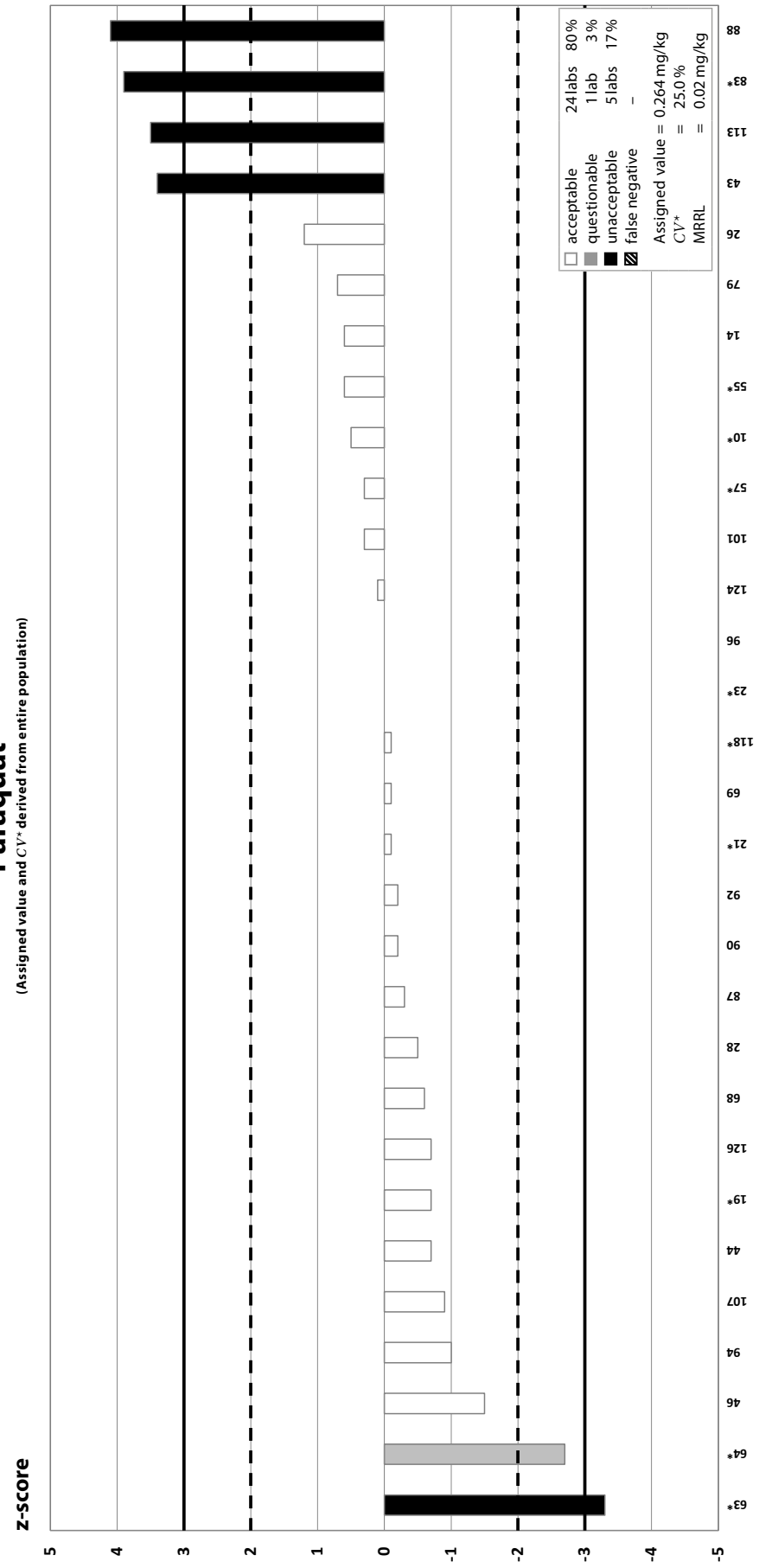
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



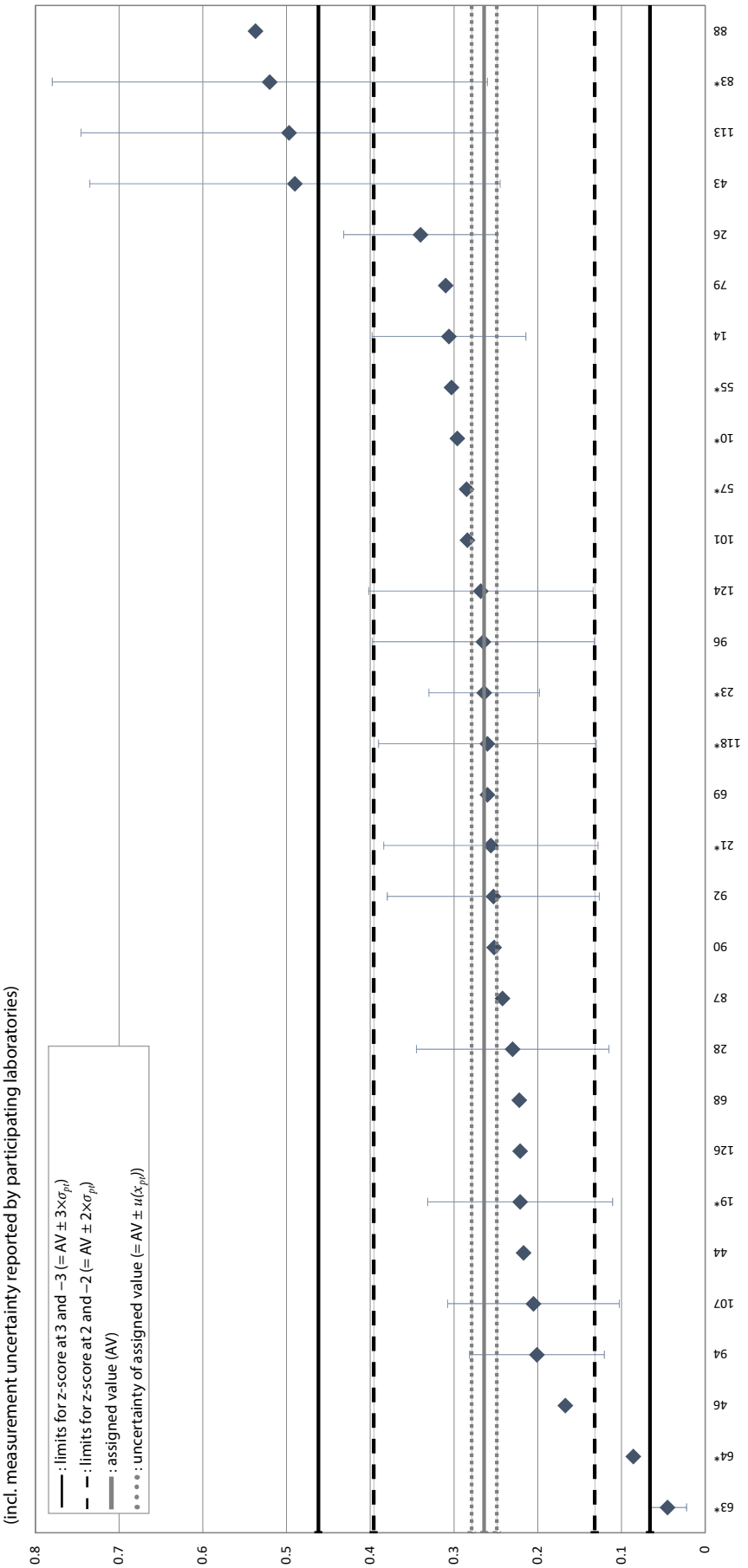
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



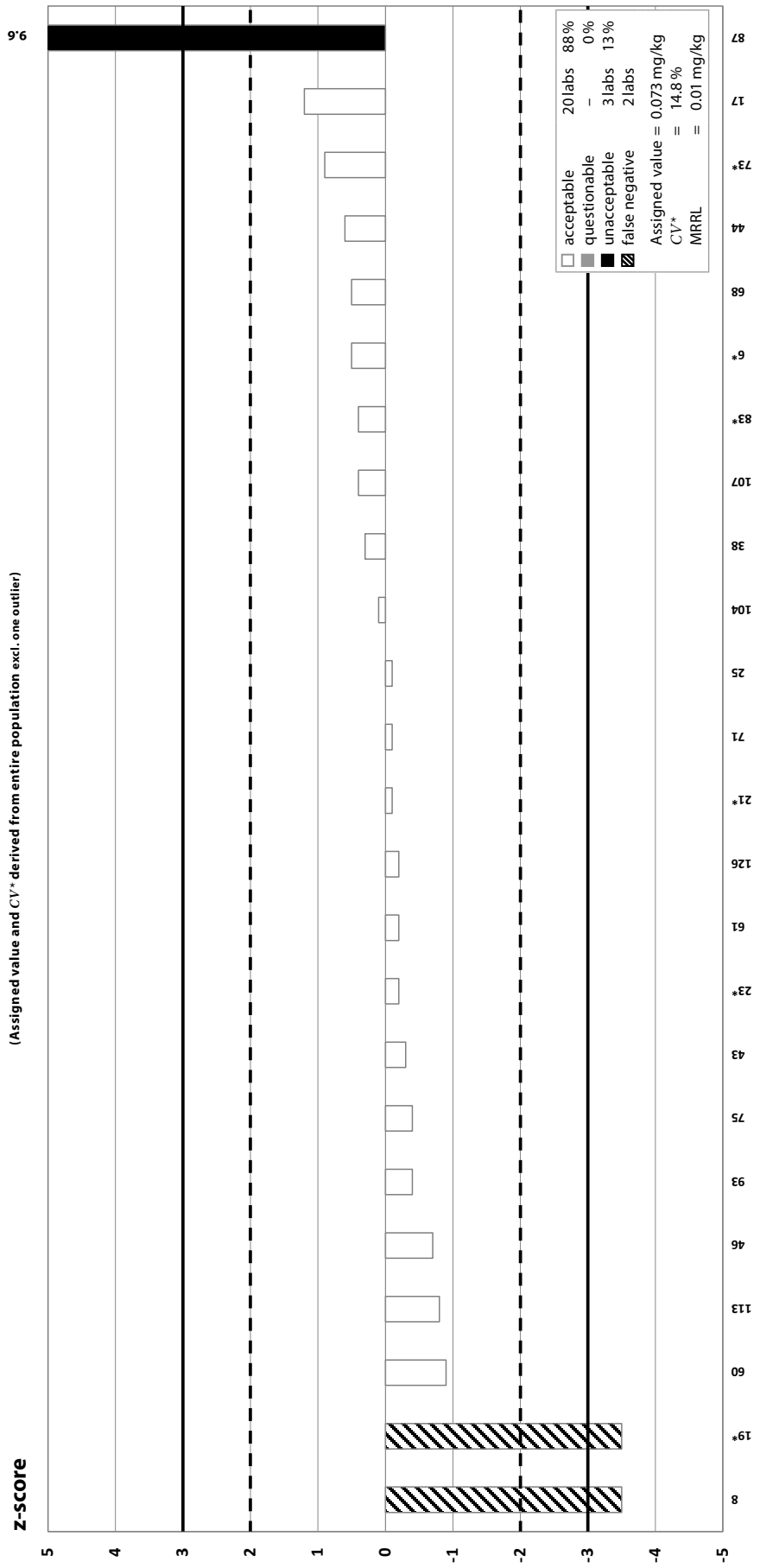
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



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



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



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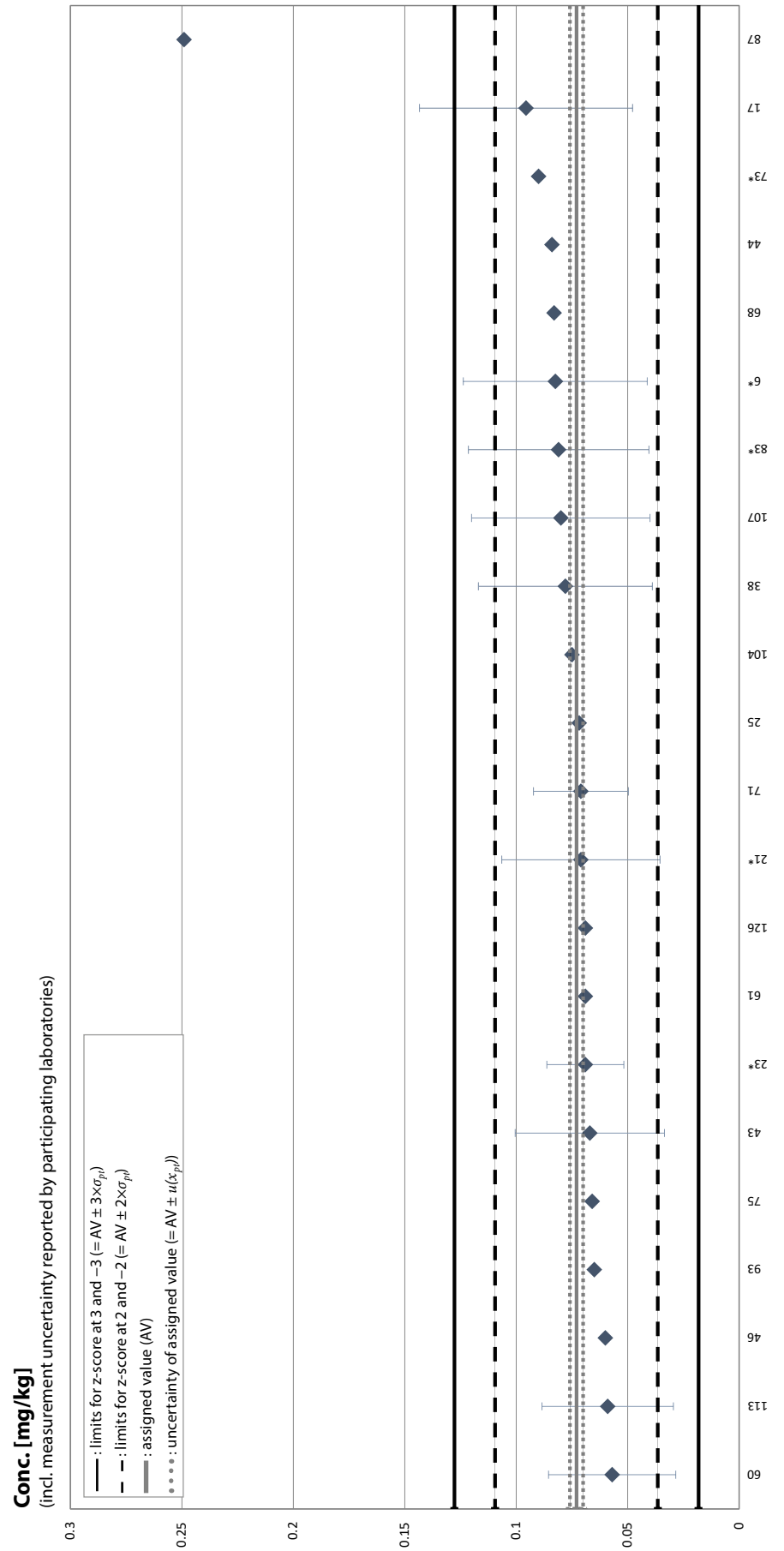
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



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

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

Bomide ion Assigned value: 21.3 mg/kg, CV^*: 20.7 %

LabCode	z-Score	Reason	Remarks/Details
13	-4.0	A, B	- in dailies analisis we does not analyze bromides on oil-rich products - After receiving the SRM results, we developed the analysis of the bromides by LCMSMS. On 01/06/21, this analysis gave 19.9mg/kg. We have identified an interferent by IC-conductivity for this type of matrix.
28	-3.6 (FN)	K	
120	-3.6 (FN)	E, A	Lack of experience with this matrix
115	2.4	G	
36	2.5	A, J	
25	2.6	M	We have not found until now the reason for the overquantification. We have reviewed the calculations and the standards. The recovery should be 100% since we used the standard addition approach (addition to sample portions). Next step: we'll repeate the standard addition and measure both with QuPPe 4.2 (validated) and 1.7 (new).
private-40	3.6	I	Cause Root analysis: the unsatisfactory result is due to an incorrect dilution factor. Corrective action: specific training to the analysts (IONIC Team) performed by Head of Department together with the Quality Assurance function

Ethephon Assigned value: 0.228 mg/kg, CV^*: 21.1 %

LabCode	z-Score	Reason	Remarks/Details
3rd-130	-3.8 (FN)	B	The method performed for the analysis of polar compounds (diquat , paraquat ethephon and phosphonic acid) in oil seed products was inappropriate. We have reanalysed the samples as per the QuPPe method and found out the satisfactory results.
14	-3.7 (FN)	L, M	Used wrong evaluation method where Ethefon was not included.
16	-2.3	A	
28	2.1	C	
111	2.1	A, B, M	Recovery samples were used as calibration standards for this analyte. (The calibration using matrix-standards were not good, the standard signal were too low)
63	13.3	F	Quantitation was carried out using an Internal Standard. Quantifying the results using external calibration would have given an acceptable result

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

Glufosinate Assigned value: 0.216 mg/kg, CV^*: 17.1 %

LabCode	z-Score	Reason	Remarks/Details
114	-3.3	G	Follow up actions undertaken: use internal standard and a matrix-matched calibration curve in sesame seed blank extract
48	-3.2	I	Unfortunately, I left an incorrect calculation factor in the used sheet. Applying the right calculation factor the results is 0,210
64	-2.1	A	
52	2.2	A, H	wi didn't received IS on time an we performed aalysis without it
120	2.7	E	Poor sensitivity below the reporting limit
102	3.4	A	
14	6.3	L, M	Reported Glyphosate results as Glufosinate

Glyphosate Assigned value: 0.510 mg/kg, CV^*: 18.5 %

LabCode	z-Score	Reason	Remarks/Details
48	-3.3	I	Unfortunately, I left an incorrect calculation factor in the used sheet. Applying the right calculation factor the results is 0,458
86	-3.2	A, E, D	Laboratory didn`t have any experience with sesame seeds (matrix is not in usual scope) and new staff member didn`t use validated accreditied laboratory method, but used the EURL QuPPe method directly . Laboratory has performed re-analysis accreditied method and achieved slightly better results.
105	-3.2	B, E	glyphosate could not be validated in commodity group high fat content (validation was performed at the same time as analysis of EUPT material)
14	-2.4	L, M	Reported Gufosinate results as Glyphosate
57	-2.3	E	

Phosphonic acid Assigned value: 0.676 mg/kg, CV^*: 24.3 %

LabCode	z-Score	Reason	Remarks/Details
104	-3.4 (FN)	J	After receiving the results regarding unacceptable results for phosphonic acid in sesame seeds test material (EUPT-SRM16) with z-score -3.4, Laboratory has been taken many steps to find and explain the source of the error. Laboratory has reviewed the documents and decided to prepare sample and analytical standards one more time. After re-analysis the result was 0,58 mg/kg. It was found that the main reason that Laboratory obtained unacceptable results was mistake during phosphonic acid analytical standard preparation. After the analyzes we performed, we found that the previously prepared standards were erroneously serially diluted. Laboratory has obtained results for phosphonic acid 0.056 mg/kg and it was below our limit of quantification, so we didn't report result. Hence the result was 10 times lower than expected.
3rd-130	-3.4 (FN)	B	The method performed for the analysis of polar compounds (diquat , paraquat ethephon and phosphonic acid) in oil seed products was inappropriate. We have reanalysed the samples as per the QuPPe method and found out the satisfactory results.
64	-2.9	A	
16	-2.2	C	
87	2.3	E	On the Hypercarb column phosphonic acid gives a double peak. However phosphonic acid 18O3 gives one broad peak. Thereof, we assume that this led to erroneous results. With standard addition a conc. of 0.69 mg/kg was obtained.

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

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

2-CE Assigned value: 4.59 mg/kg, CV^*: 30.4 %

LabCode	z-Score	Reason	Remarks/Details
32	-3.7	A, C	
68	-3.0	B, D, M	No water addition during extraction. In the new optimized procedure, hydrated matrix is extracted by ethylacetate (z-score: -0,6) (re-analysed: 3.92 mg/kg)
19	-2.9	A, C	Sample was reanlysed with addtion of water, obtain result was correct 2-CE- 5.29 mg/kg
3rd-106	-2.6	A, C	
121	-2.3	A, C	
109	3.5	M	Unpredictable interferences during the first measurements may have occurred. The sample was analysed again (21/05/21). For this second measurement, a concentration of 6,53 mg/kg was found for 2-CE, resulting in an acceptable z-score of 1,47.
120	10.8	J	Wrong standard addition

EO (sum) Assigned value: 2.50 mg/kg, CV^*: 29.5 %

LabCode	z-Score	Reason	Remarks/Details
32	-3.7	A, C	with 10% water in the extraction solvent: 2.7 mg/kg
68	-3.0	B, D, M	No water addition during extraction. In the new optimized procedure, hydrated matrix is extracted by ethylacetate (z-score: -0,6) (re-analysed: 2.15 mg/kg)
19	-2.9	A, C	Sample was reanlysed with addtion of water, obtain result was correct - EO sum - 2.89 mg/kg
3rd-106	-2.6	A, C	
121	-2.3	A, C	
109	5.0	M	The result for EO (sum) is calculated from the results of EO and 2-CE. Because EO was found to be false positive and the concentration of 2-CE was too high in the first measurements, this results in a too high concentration for the sum. The sample was analysed again (21/05/21). For this second measurement, a concentration of EO (sum) of 3,58 mg/kg was calculated, resulting in an acceptable z-score of 1,46.
120	10.8	J	Wrong standard addition

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

Chlorate Assigned value: 1.03 mg/kg, CV^*: 20.7 %			
LabCode	z-Score	Reason	Remarks/Details
114	-3.1	G	Follow up actions undertaken: use internal standard and a matrix-matched calibration curve in sesame seed blank extract
28	-2.6	C	
102	-2.6	E, A	A) with matrix sesam
36	2.6	A, J	
25	3.4	M	We have not found until now the reason for the overquantification. We have reviewed the calculations and the standards. The recovery should be 100% since we used the standard addition approach (addition to sample portions). Next step: we'll repeate the standard additions for Chlorate and Bromide and measure both with QuPPe 4.2 (validated) and 1.7 (new).

Diquat Assigned value: 0.244 mg/kg, CV^*: 24.7 %			
LabCode	z-Score	Reason	Remarks/Details
3rd-130	-3.8	B	The method performed for the analysis of polar compounds (diquat , paraquat ethephon and phosphonic acid) in oil seed products was inappropriate. We have reanalysed the samples as per the QuPPe method and found out the satisfactory results.
63	-2.6	B	Investigation of new method using cation analysis on IC to replace this method
64	3.2	A	
113	3.5	I	the conversion factor has not been applied to results of the ring test SRM16, was a problem in our Lims system.After correction with the conversion factor of diquat (CF = 0.50881) and paraquat (CF = 0.7241), the results were close to Assigned Value. The correct results of Diquat/paraquat: Paraquat = 0.36 mg/kg and Diquat = 0.234 mg/kg. And Z-score for Diquat -0.2 and for Paraquat for 1.5
83	6.1	M	The reason of poor perfomance with this analyte is a combination of i) lack of experience with ILIS and ii) ILIS degradation. Under normal conditions, we never use ILIS for quantifications. Due to the result obtained in a proficiency test carried out previously with these 2 compounds, we concluded that our method for soybean meal showed very low recoveries. For this reason we decided to use the ILIS for diquat in order to correct our results for recovery. Analyzing the results obtained, we think that the ILIS used was degraded and one of the degradation products was the diquat. It was quantified with the diquat present in test-sample. Simultaneously with the quantification of the other compounds present in the test, we proceeded to quantify diquat using the standard addition method without using ILIS and the results obtained can be seen in the sheet2. Median for diquat =0.278 mg/kg (CV=25%)

Matrine Assigned value: 0.164 mg/kg, CV^*: 24.5 %			
LabCode	z-Score	Reason	Remarks/Details
14	-3.7 (FN)	E	
114	-3.0	B	Currently developing the single residue method for this compound in this commodity group
private-40	-2.3	B	Cause Root analysis: to evaluate the accuracy of the Matrine analytical method, only recovery tests and no tests on certified reference material were used; since the recovery tests were in conformity, the poor extraction efficiency of the method was not noticed. Corrective action: the analytical method has been already updated with a new extraction procedure (different alkaline conditions). The Ptest sample was retested with the new procedure obtaining a result in compliance with the assigned value (result: 0,112 , z-score: -0,4) A specific reference material to use together the samples in order to verify the performance (accuracy) of the method will be researched.
87	2.3	C	EDTA is not used routinely. With use of EDTA a conc. of 0.146 mg/kg was obtained.
private-58	3.1	M	For this molecule we gived the result obtained with appropriate procedure (Basic QuEChERs), parallel we did also standard QuPPe and the obtained result was 0,11 mg/kg
73	7.4	G	The value 0.354 mg/kg was calculated via standard addition without isotopically labelled internal standard (matrine D3) which was not available in our laboratory during the time of EUPT-SRM16. Repeated analysis of matrine calculated by standard addition including calibration to matrine D3 performed in August 2021 showed result of matrine 0.150 mg/kg
28	7.6	G	
68	10.2	J	HPC Standards GmbH, Lot 787945 (exp. 03/2023)

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

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

Nicotine Assigned value: 0.255 mg/kg, CV^*: 27.5 %

LabCode	z-Score	Reason	Remarks/Details
114	-3.1	B	Currently developing the single residue method for this compound in this commodity group
private-58	2.3	M	For this molecule we gived the result obtained with appropriate procedure (Basic QuEChERs), parallel we did also standard QuPPe and the obtained result was 0,28 mg/kg
86	3.0	A, E, D	Laboratory didn`t have any experience with sesame seeds (matrix is not in usual scope) and new staff member didn`t use validated accreditied laboratory method, but used the EURL QuPPe method directly . Laboratory has performed re-analysis accreditied method and achieved slightly better results.
112	4.5	I	Bei der Kalibrierung zur Berechnung des Nikotingehaltes wurde statt der wahren Einwaage von 5 g die üblicherweise bei der Untersuchung von getrockneten Pilzen und Tee verwendete Einwaage von 2 g verwendet, so dass das gemeldete Ergebnis um den Faktor 2,5 zu hoch ist. Die entsprechende Umrechnung ergibt einen Wert von 0,216 mg/kg. Bei der Wiederholungsanalyse des Restmaterials aus der LVU wurde bei korrekter Auswertung ein Gehalt von 0,212 mg Nikotin/kg ermittelt. Beide Ergebnisse hätten zu einem befriedigenden z-score <|2| geführt.

Paraquat Assigned value: 0.264 mg/kg, CV^*: 25.0 %

LabCode	z-Score	Reason	Remarks/Details
3rd-100	-4.0	L	
3rd-130	-3.9	B	The method performed for the analysis of polar compounds (diquat , paraquat ethephon and phosphonic acid) in oil seed products was inappropriate. We have reanalysed the samples as per the QuPPe method and found out the satisfactory results.
63	-3.3	B	Investigation of new method using cation analysis on IC to replace this method
64	-2.7	A	
113	3.5	I	the conversion factor has not been applied to results of the ring test SRM16, was a problem in our Lims system.After correction with the conversion factor of diquat (CF = 0.50881) and paraquat (CF = 0.7241), the results were close to Assigned Value. The correct results of Diquat/paraquat: Paraquat = 0.36 mg/kg and Diquat = 0.234 mg/kg. And Z-score for Diquat -0.2 and for Paraquat for 1.5
83	3.9	M	The reason of poor perfomance with this analyte is a combination of i) lack of experience with ILIS and ii) ILIS degradation. Under normal conditions, we never use ILIS for quantifications. Due to the result obtained in a proficiency test carried out previously with these 2 compounds, we concluded that our method for soybean meal showed very low recoveries. For this reason we decided to use the ILIS for paraquat in order to correct our results for recovery. Analyzing the results obtained, we think that the ILIS used was degraded and one of the degradation products was the paraquat. It was quantified with the paraquat present in test-sample. Simultaneously with the quantification of the other compounds present in the test, we proceeded to quantify paraquat using the standard addition method without using ILIS and the results obtained can be seen in the sheet2. Median for paraquat =0.264 mg/kg (CV=21%)

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

Trimesium Assigned value: 0.073 mg/kg, CV^*: 14.8 %			
LabCode	z-Score	Reason	Remarks/Details
19	-3.5 (FN)	B, D	
87	9.6	I	The conversion into Trimesium was not applied. The conversion of 0.249 mg/kg results in a conc. of 0.078 mg/kg.

False Positive Results Assigned value: 0.073 mg/kg, CV^*: 14.8 %				
Analytes	LabCode	Reported Conc. [mg/kg]	Reason	Remarks/Details
AMPA	63	0.186	A	New method recently validated
EO	109	0.981	M	Unpredictable interferences during the first measurements may have occurred. The sample was analysed again (21/05/21). For this second measurement, no ethylene oxide was found.
EO	private-77	0.180	M	See file .docx attached to the mail
Fosetyl	63	0.042	A	New method recently validated

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GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues in Food and Feed

Introduction

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

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

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

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

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

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

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

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

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

EUPT-Organisers and Scientific Committee

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

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

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

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

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

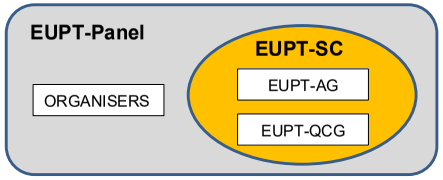
The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and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

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

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

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

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



The decisions of the EUPT-Panel will be documented.

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

EUPT Participants

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

- Art 38 (b) of Reg. 625/2017/EC and Art. 28 of Reg. 396/2005/EC[6] (for all OfLs analysing for pesticide residues within the framework of official controls[7] of food or feed)
- Art. 101 (1)(a) of Reg. 625/2017/EC (for all NRLs)

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

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

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

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

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

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

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

Confidentiality and Communication

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

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

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

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

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

The official language used in all EUPTs is English.

Announcement / Invitation Letter

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

Target Pesticide List

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

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

Specific Protocol

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

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Homogeneity of the Test Item

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

The homogeneity test data are statistically evaluated according to ISO 13528, Annex B or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC. The results of all homogeneity tests are presented to the EUPT-SC. In special cases, where the above homogeneity test criteria are not met, the EUPT-Panel, considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution of the participants' results (CV*), the analytical difficulties faced during the test, knowledge of the analytical behaviour of the pesticide question), may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report. For certain analytes with comparable properties, an equivalent distribution within the sample can be expected if they were spiked/used at simultaneously. The homogeneity test, of one or more of these analytes, may thus be skipped or simplified. If, however, the distribution of participants' results for an analyte that was not or not fully tested for homogeneity, is found to be atypically broad, compared to the tested analytes, the EUPT-SC may decide that a homogeneity test should be performed a posteriori by the EURL.

Stability of the analytes contained in the Test Item

The Test Items will also be tested for stability - according to ISO 13528, Annex B. The time delay between the first and the last stability test must exceed the period of the EUPT-exercise. Typically the first analysis is carried out shortly before the shipment of the Test Items and the last one shortly after the deadline for submission of results. To better recognise trends and gain additional certainty one or more additional tests may be conducted by the Organisers. At least 6 sub-samples (analytical portions) should be analysed on each test day (e.g. 2 analytical portions withdrawn from three randomly chosen containers OR 6 portions withdrawn from a single container). In principle all pesticides contained in the Test Item should be checked for stability. However, in individual cases, where sufficient knowledge exists that the stability of a certain analyte is very unlikely to be significantly affected during storage (e.g. based on experience from past stability tests or knowledge of its physicochemical properties), the Organisers, after consultation with the EUPT-QCG, may decide to omit a specific stability test. The EUPT-Panel will finally decide whether

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

A pesticide is considered to be adequately stable if $|y_i - y| \leq 0.3 \times \sigma_{pt}$, 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 pesticide question) may decide to overrule the test. The reasons of this overruling will be transparently explained in the Final EUPT-Report.

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

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

Methodologies to be used by the participants

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

General procedures for reporting results

Participating laboratories are responsible for reporting their own quantitative results to the Organiser within the stipulated deadline. Any pesticide that was targeted by a participating laboratory should be reported as "analysed". Each laboratory will be able to report only one result for each analyte detected in the Test Item. The concentrations of the pesticides detected should be expressed in 'mg/kg' unless indicated otherwise in the specific protocol. Laboratories should not report results below their reporting limits.

Correction of results for recovery

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

a) the use of recovery correction factors,
b) the use of stable isotope labelled analogues of the target analytes as Internal Standards (ILISs),
c) the 'procedural calibration' approach as well as
d) the approach of 'standard addition' with additions of analyte(s) being made to analytical portions.

Results may be corrected for recovery only in cases where this correction is applied in routine practice (including cases of MRL-violations). Laboratories are required to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery rate (in percentage) must also be reported. If one or more of the approaches b), c) and d) were employed, in which correction for recovery is inherent to the procedures, the apparent recovery figures obtained during validation experiments are not mandatory, and the approached followed are to be reported in the appropriate fields within the data submission tool.

Methodology information

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

Results evaluation

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

- ***False Positive results***

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

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

- ***False Negative results***

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

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

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

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value x_{pt} (= consensus concentration) will typically be estimated using the robust estimate of the

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participant's mean (x^*) as described in ISO 13528:2015[8], taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors (see "Omission or Exclusion of results" below) or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific or similar compounds in the past.

- ***Omission or Exclusion of results***

Before estimating the assigned value, results associated with obvious mistakes have to be examined to decide whether they should be removed from the population. Such gross errors may include incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits, incorrect unit), calculation errors (e.g. missing factors), analysis of a wrong sample/extract (e.g. a spiked blank), use of wrong concentrations of standard solutions, incorrect data processing (e.g. integration of wrong peak), inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate analytical steps or procedures that demonstrably lead to significantly biased results (e.g. employing inappropriate internal standards or analytical steps or conditions leading to considerable losses, due to degradations, adsorptions, incomplete extractions, partitioning etc.). Where the Organisers (e.g. after the publication of the preliminary report) receive information of such gross errors, having a significant impact on a generated result, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Results may also be omitted e.g. if an inappropriate method has been used even if they are not outliers. All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the Final EUPT-Report. However, z scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.

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

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

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

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

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

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

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

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

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

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

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

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

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

For informative purposes the robust relative standard deviation (CV^*) of the participants results is calculated according to ISO 13528:2015; Chapter 7.7 following Algorithm A in Annex C (so called "consensus approach").

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

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– ***z scores***

This parameter is calculated using the following formula:

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

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

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

Z scores will be interpreted in the following way, as is set in the ISO 17043:2010[10]:

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

For results considered as false negatives, z scores will be calculated using the MRRL or RL (the laboratory's Reporting Limit) if RL < MRRL. Where, using this approach, the calculated z scores for false negatives are > −3 (still questionable), they will be fixed at −3.5 to underline that these are unacceptable results. These z-scores will typically appear in the z-score histograms and used in the calculation of combined z-scores.

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

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

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

– ***Category classification***

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

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

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

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

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

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z score (AZ^2)[11,12] (see below) will be used. The AZ^2 is calculated as follows:

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

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

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

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

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

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

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

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

Publication of results

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

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

Certificates of participation

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

Feedback

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

Correction of errors

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

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

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

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

Follow-up activities

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

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

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

NRLs will be considered as **underperforming in relation to scope** if in at least two of the last four EUPTs falling within their responsibility area they: a) haven't participated, or b) targeted less than 90% of the compulsory pesticides in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the test items (80% for SRM-compounds). Additionally, NRLs that obtained AZ^2 higher than 3 (AAZ higher than 1.3 for SRM-compounds) in two consecutive EUPTs of the last four EUPTs, will be considered as **underperforming in accuracy**. A two-step protocol established by DG-SANTE will be applied as soon as underperformance of an NRL is detected[13]:

Phase 1:

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

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

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

Phase 2:

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

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

Disclaimer

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

SPECIFIC PROTOCOL

for the 16th EU Proficiency Test
on Pesticides requiring Single Residue Methods
EUPT – SRM16 (2021)

(released and revised on 15 March 2021)

Introduction

This protocol is complementary to the valid version of the "**General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed, Ed. 9**" covering all EUPTs in 2020 and 2021.
The EUPT-SRM16 is organized by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL– SRM) that is accredited according to ISO 17043 as a provider of proficiency tests (please see **EURL-SRM accreditation**).

The EUPT-SRM16 deals with the analysis of SRM-pesticides in sesame seeds. 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[1] analyses of pesticide residues in food or feed products of plant origin with high lipid content. 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, 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 from obliged laboratories received during registration, especially details considering the scope, will be considered in the final list of obliged laboratories. The registration period started on 23 February and ended on 8 March 2021.

The most important documents related to this PT can be accessed via the **EUPT-SRM16-Website**.

Test Item

The Test Item of this EUPT is **milled, hulled non-roasted white sesame seeds (hereinafter called "sesame seeds").**

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

[1] Within the frame of National and/or EU coordinated official control programs according to Art. 38 (2) of Reg. (EC) 625/2017 and of Art. 28 of Reg. (EC) 396/2005

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

Target Analytes and MRRLs

The Test Item will contain several pesticides from the **EUPT-SRM16 Target Pesticides List.** Laboratories should read the Target List list 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 identifying false positive and false negative results and for the calculation of z-scores for false negatives. **Make sure to download the latest version of the EUPT-SRM16 Target Pesticides List before starting with analysis and reporting results**.

Shipment of Test Item

Dispatch of the Test Item is planned on 22 March, 2021.
Test Item will be packed into thermo-boxes together with frozen gel packs and will be shipped from Germany via DHL Express to the participants. Prior to shipment, a reminder will be sent to the participating laboratories by e-mail.
The participating laboratories must make their own arrangements for the receipt of the package. They should inform the Organizers of any public holidays in their country/city during the week of the shipment, and must make the necessary arrangements to receive the shipment, even if the laboratory is closed.

Where complications during customs clearance or shipment are expected, the participants should provide the Organizers in advance (by 16 March) with detailed contact Information (e.g. mobile phone numbers of laboratory personnel) and all necessary documents to be stuck on the package. Such documents may be e.g. a permission for import the commodity for lab analysis or an **instruction in local language** to explain the need to keep the package in a freezer in case of delay during shipment or clearance delay at customs.

After the shipment is stored in the DHL delivery system and the waybill 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 packages 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 deeply frozen (at -18°C or lower) until analysis in order to avoid any possible deterioration/spoilage of the sample material and to minimize analyte losses.

Before analytical portions are taken for analysis, the Test Item should be mixed thoroughly in its entirety. Afterwards, analytical portions can be taken from the Test Item directly. No additional milling is necessary.

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.

Appendix 9. Specific Protocol of EUPT-SRM16

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-SRM16 result submission webtool**.

The homogeneity tests will be conducted using 2 g as well as 5 g portions. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can be guaranteed only for sample portions equal to or bigger than the portion size used in the homogeneity test.

Results submission webtool

Sample receipt acknowledgement, analytical results and method information are to be submitted via the **EUPT-SRM16 Result Submission Webtool**:

- **Sample receipt acknowledgement: 23 March - 27 March.**
- **Reporting of Analytical results and method information: 23 March - 27 April, 2021.**
- **Deadline for result submission is 27 April, 11.30 pm (CEST), 2021.**
- **Reporting of Additional information on methods used for tentatively false negative results: 28 April - 6 May, 2021.**

A guideline for the new **EUPT-SRM16 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-SRM16 Result Submission Webtool, participants must use their personal login credentials (username and password). The personal login credentials will be provided to the PT-contact persons on 23 March.

The lab's unique lab code for the EUPT-SRM16 will be provided to the participants following the first access to EUPT-SRM16 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-SRM16 Result Submission Webtool** the date of receipt, whether the material is accepted or not, and any other comments concerning the test material. This task should be finalized by 27 March. 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 additionally contact the organizers via e-mail ASAP (**eurl-srm@cvuas.bwl.de**) to ensure that corrective actions are taken as early as possible. Please note that completing the sample receipt form is a pre-requisite for accessing the website areas for results submission. However, you can still access sample receipt form and edit it later.

The 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 intervene at the shipping company, the customs or the organizers if they notice any delays. Any participants not having received the Test Items by the Fri. 26 March at noon, must inform the Organizer via e-mail (EURL-SRM@cvuas.bwl.de) by Fri. 26 March 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.

- Reporting qualitative and quantitative results

To report their results, laboratories must access the **EUPT-SRM16 Result Submission Webtool**.
All results must be reported on this website by 27 April, 11.30 pm (CEST), 2021. The page 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-SRM16 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 subcontracting may also (and even encouraged to) take place within the EUPT exercise.

IMPORTANT:
The participants are obliged to inform the organizers of all cases of subcontracted analyses, and to provide details on the subcontracting laboratory.

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. Following the General Protocol, results reported as "< RL" or "< #.# mg/kg" will be judged as "false negatives" if the concerned analyte is present in the test material at levels ≥ 3 times the MRRL.

  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 within 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", 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 not encountered at the quantifiable level (<RL), its concentration should be considered equal to zero when calculating the summed result.

  Recovery-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. 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-SRM16 Result Submission Webtool** the participating laboratories have to provide information on the analytical method(s) applied to pesticides, which were analysed and detected in the Test Item.

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

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.

- 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 tentatively false negative result(s), it will be asked to enter the method information for the analytes(s) in question within 7 workdays.

Establishment of assigned values

In addition to OfLs from EU Member States, EFTA countries, a limited number of laboratories from EU candidate countries and third countries are allowed to take part in this exercise. Furthermore, commercial labs ready to be subcontracted by EU Member States for the analysis of ethylene oxide related residues in sesame seed imports were also invited to participate in the current PT. For establishing the assigned values of EO, EO (sum) and 2-CE, all submitted results are taken into account. For all other analytes, the assigned values are based on results submitted by OfLs from EU and EFTA countries only.

Subcontracting

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

a) Generation of the login credentials
b) Programming and administration of EUPT-SRM16 result submission website

Follow-up actions

After the distribution of the EUPT-SRM16 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 report their insights and possible corrective actions to the Organizer. This information will be forwarded to the corresponding NRL-SRMs upon request. All EUPT-SRM16-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 relevant to the evaluation and interpretation of the PT.

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

Documents

All documents related to the EUPT–SRM16 can be downloaded from **EURL-Document Repository (CIRCA-BC)** or **EUPT-SRM16 Website.**
For any questions, please contact the organizers **EURL-SRM@cvuas.bwl.de**

IMPORTANT:
Please check the **EUPT-SRM16 Website** before starting with the analysis in order to **make sure, that you have the latest version of all documents available**. In case of major changes, the participants will be informed via e-mail.

Participation fees and payment details

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

- OfLs (including NRLs) from EU countries, EU-candidate countries and EFTA countries: 200 €
- Commercial Labs based in EU or EFTA countries: 200 €
- Labs based in third countries: 350 €

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

Additional cost may occur if extra services are requested in relation to the payment or if invoices have to be changed due to new information, or requesting of it being resent.

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

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

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

Bank Details:

Bank account holder:	**Landesoberkasse Baden Wuerttemberg**
Bank Name :	**Baden Wuerttembergische Bank**
IBAN:	**DE 02 6005 0101 7495 5301 02**
BIC/SWIFT:	**SOLADESTXXX**
Payee identification text:	***See invoice (important and MUST be indicated!)***
VAT 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 based in CVUA Freiburg and EURL-SRM based in CVUA Stuttgart belong to the same ministry and have thus the same bank account.**
  If your laboratory is participating in both PTs (EUPT-SRM16 and EUPT-AO16), 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.

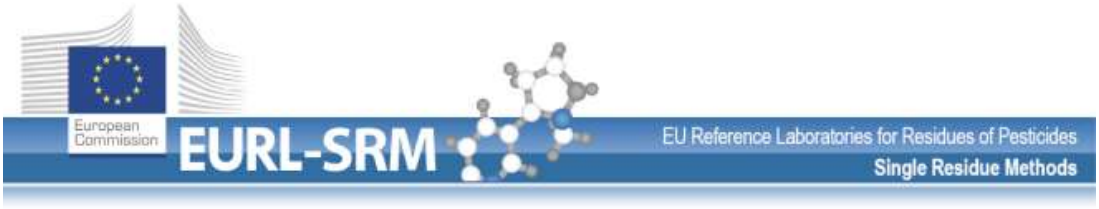
Calendar of EUPT-SRM16

(please see https://www.eurl-pesticides.eu/userfiles/file/EUPT-SRM16_Calendar.pdf)

Target Pesticides List of EUPT-SRM16

(please see https://www.eurl-pesticides.eu/userfiles/file/EUPT-SRM16_TargetPesticideList.pdf)

Appendix 10 Calendar and Target Pesticides List of EUPT-SRM16



CALENDAR for the EUPT – SRM16

Sesame Seeds

(Released on 22/02/2021)

Activity	Dates
Announcement of the EUPT-SRM16 opening of the **EUPT-SRM16 Website** with links to all relevant documents	22 Feb
Registration Period for EUPT-SRM16 via "**EUPT-Registration Website**" **Obliged Labs MUST enter this website and either register OR give explanations for non-participation**	23 Feb – 08 Mar, 23:30 h CEST*
Dispatch of EUPT-SRM16-Specific Protocol	~ 15 Mar
Shipment of EUPT-SRM16 Test Item	22 Mar
Confirming Sample Receipt and Acceptance via "**EUPT-SRM16 Result Submission Webtool**"	From 23 Mar
Submission of Results (**Pesticide scope, Results, Method Info**) via "**EUPT-SRM16 Result Submission Webtool**"	30 Mar – 27 Apr, 23:30 h CEST
Submission of Additional/Missing Information e.g. Method info on tentatively false negative results via "**EUPT-SRM16 Result Submission Webtool**"	28 Apr – 05 May
Dispatch of Preliminary Report containing results as well as preliminary assigned values and z-scores only	Within ~ 3 weeks after submission deadline
Collection of reasons for underperformance and missing information on methods	June 2021
Dispatch of Final Report	Dec 2021

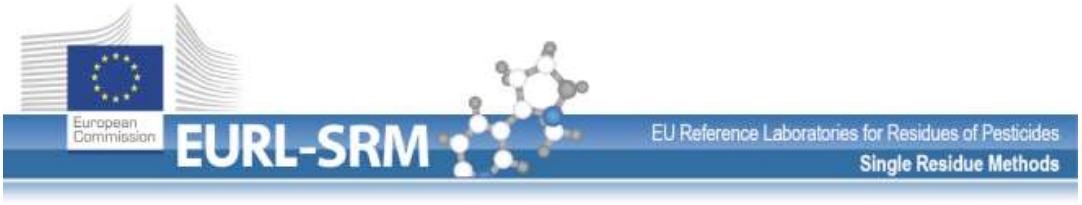
* Please make sure to register for the EUPT by the deadline 08 March 2021 via "**EUPT-Registration Website**". Any wish for registration after this deadline or not using the registration website cannot be considered.

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

The EUPT-SRM Team

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

Appendix 10 (cont.) Calendar and Target Pesticides List of EUPT-SRM16



TARGET PESTICIDE LIST

for the EUPT–SRM16 2021, Sesame Seeds

(update on 17.03.2021)

Sorted alphabetically like in the Webtool, M: Mandatory*; O: Optional (=Voluntary)

	Analytes Name	Residue definition for the PT and additional remarks	MACP/WD	MRRL (mg/kg)
O	**2-Chloroethanol (2-CE)**		Of actual concern	0.05
O	**AMPA**	Glyphosate metabolite; future RD	Monitoring-WD	0.1
M	**Bromide**	Expressed as bromide anion	MACP	2
O	**Chlorate**	Expressed as anion	Monitoring-WD	0.04
M	**Chlormequat chloride**	Expressed as chlormequat chloride	MACP	0.01
O	**Diquat**	Expressed as dication	Monitoring-WD	0.02
M	**Ethephon**		MACP	0.04
O	**Ethylene oxide (EO)**		Of actual concern	0.02
O	**Ethylene oxide (sum) (EO (sum))**	Sum of Ethylene oxide and 2-Chloroethanol **expressed as ethylene oxide**	Of actual concern	0.05
M	**Fosetyl**	Expressed as fosetyl (free acid)	MACP	0.02
M	**Glufosinate**		MACP	0.02
M	**Glyphosate**		MACP	0.1
M	**MPP**	Glufosinate metabolite, also known as **MPPA** CAS-No.: 15090-23-0	MACP	0.02
O	**Matrine**		Monitoring-WD	0.01
M	**Mepiquat chloride**	Expressed as mepiquat chloride	MACP	0.01
M	**N-Acetyl-glufosinate**	Glufosinate metabolite	MACP	0.02
O	**N-Acetyl glyphosate**	Glyphosate metabolite; future RD	Monitoring-WD	0.05
O	**Nicotine**		Monitoring-WD	0.01
	Paraquat	Expressed as dication	Monitoring-WD	0.02
M	**Phosphonic acid**	Fosetyl metabolite, expressed as acid	MACP	0.1
O	**Trimesium**	Expressed as cation	Monitoring-WD	0.01

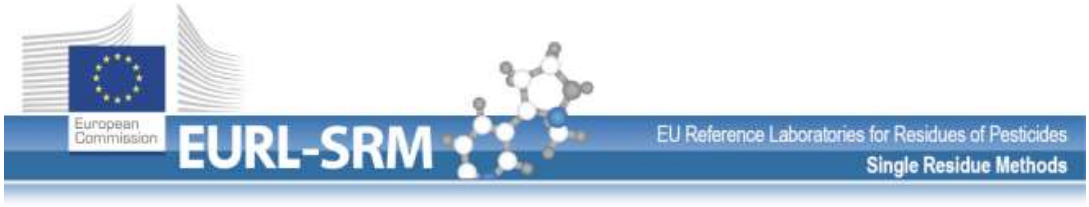
MACP-Reg.: REGULATION (EU) 2020/585 of 27 April 2020
WD: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 23–24 November 2020 rev. 12(2)
*** Only mandatory (=composry) analytes will be considered in the scope-based classification, optional (=voluntary analytes not. (please refer also to the EUPT General Protocol)**

Note: This document may be subject to minor changes. In case of significant changes, the organizers will send e-mails. In any case, please check our website periodically to make sure you are using the latest available version. For any further clarification, don't hesitate to contact us under **eurl-srm@cvuas.bwl.de**

The EUPT-SRM16 Organising Team

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

Appendix 10 (cont.) Calendar and Target Pesticides List of EUPT-SRM16



TARGET PESTICIDE LIST

for the EUPT–SRM16 2021, Sesame Seeds

(update on 17.03.2021)

Ordered according to Mandatory/Optional* Analytes

MANDATORY ANALYTES			
Analytes Name	**Residue definition for the PT and additional remarks**	**MACP/WD**	**MRRL (mg/kg)**
Bromide	Expressed as bromide anion	MACP	2
Chlormequat chloride	Expressed as chlormequat chloride	MACP	0.01
Ethephon		MACP	0.04
Fosetyl	Expressed as fosetyl (free acid)	MACP	0.02
Glufosinate		MACP	0.02
Glyphosate		MACP	0.1
Mepiquat chloride	Expressed as mepiquat chloride	MACP	0.01
MPP	Glufosinate metabolite, also known as **MPPA** CAS-No.: 15090-23-0	MACP	0.02
N-Acetyl-glufosinate	Glufosinate metabolite	MACP	0.02
Phosphonic acid	Fosetyl metabolite, expressed as acid	MACP	0.1
OPTIONAL ANALYTES			
Analytes Name	**Residue definition for the PT and additional remarks**	**MACP/WD**	**MRRL (mg/kg)**
AMPA	Glyphosate metabolite; future RD	Monitoring-WD	0.1
Chlorate	Expressed as anion	Monitoring-WD	0.04
Diquat	Expressed as dication	Monitoring-WD	0.02
Ethylene oxide		Of actual concern	0.02
2-Chloroethanol		Of actual concern	0.05
Ethylene oxide (sum)	Sum of Ethylene oxide and 2-Chloroethanol **expressed as ethylene oxide**	Of actual concern	0.05
Matrine		Monitoring-WD	0.01
N-Acetyl Glyphosate	Glyphosate metabolite; future RD	Monitoring-WD	0.05
Nicotine		Monitoring-WD	0.01
Paraquat	Expressed as dication	Monitoring-WD	0.02
Trimesium	Expressed as cation	Monitoring-WD	0.01

MACP-Reg.: REGULATION (EU) 2020/585 of 27 April 2020
WD: Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 23–24 November 2020 rev. 12(2)
*** Only mandatory (=composry) analytes will be considered in the scope-based classification, optional (=voluntary analytes not. (please refer also to the EUPT General Protocol)**

Note: This document may be subject to minor changes. In case of significant changes, the organizers will send e-mails. In any case, please check our website periodically to make sure you are using the latest available version. For any further clarification, don't hesitate to contact us under **eurl-srm@cvuas.bwl.de**

The EUPT-SRM16 Organising Team

Appendix 11 Announcement and Call for Registration for the EUPT-SRM16

Schreiter, Pat (CVUA-S)

Von: Schreiter, Pat (CVUA-S) <Pat.Schreiter@cvuas.bwl.de> im Auftrag von EURL-SRM Pesticides (CVUA-S) <EURL-SRM@cvuas.bwl.de>
Gesendet: Montag, 22. Februar 2021 10:12
An: EURL-SRM Pesticides (CVUA-S)
Betreff: EUPT-SRM16 (Sesame Seeds): Announcement and Invitation (OfLs)

Dear Colleagues from EU/EFTA-OfLs,

We herewith cordially invite you to participate in the upcoming European Proficiency Test **EUPT-SRM16** on the analysis of pesticides requiring Single Residue Methods organised by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM).

Key Data:

- EUPT-SRM16-Website with links to all relevant documents
- Material: one unit of **Sesame Seeds** (~150-200 g) containing both incurred and spiked compounds.
- **Registration: via the "**EUPT-Registration Website**" from tomorrow 23 Feb till 8 March, 2021.**
- **Dispatch** of **Test Material**: **22 March, 2021**.
- **Submission deadline** for results and method information: **27 April, 2021** on the "**EUPT-SRM16 Result Submission Website**" **(not yet active!)**.
- Fee: **200 €** for one unit

Participation rules:

The following labs are considered **obliged** to participate in the EUPT-SRM16:

- all NRLs for pesticides requiring Single Residue Methods (**NRL-SRMs**), see Art. 101 (1)(a) of Reg. (EC) 625/2017,
- all Official Laboratories (**OfLs**) performing pesticide residue analyses of **commodities of plant origin with high lipid content or of feed** within the frame of National and EU official controls, see Art 38 (2) of Reg. (EC) 625/2017 and Art. 28 of Reg. (EC) 396/2005. This includes laboratories involved in official import controls within the frame of Reg (EC) 669/2009.

Please check on the registration page ("EUPT-Registration Website"), if your laboratory is classified as obliged to participate in the EUPT-SRM16. Errors should be reported to your NRL as well as to the EURL-SRM.

- **In case your laboratory is obliged to participate, but you don't intend to participate, you have to state the reasons for non-participation on the "EUPT-Registration Website" as expected by DG-SANTE.**
  Please do not report your reason via e-mail. If you do so, you will still be prompted to access the website and enter your explanation there.
- In case the participation of your laboratory on this EUPT is on voluntary basis and you are not going to take part in it, you don't have to do anything.
  OfLs that are not obliged to participate in this PT are still welcome to register for the EUPT-SRM16, the acceptance of their registration will, however, depend on the availability of PT-Material.

A general fee of **200 €** will be charged to each participating OfL in the EUPT-SRM16 to cover the costs of test material handling and shipment.
Double amount of material can be requested via e-mail (address: **EUPT-SRM@cvuas.bwl.de**) or on the registration website. In this case the above fees will be doubled.

All scheduled activities and deadlines of this PT can be found on the EUPT-SRM16_Calendar (https://www.eurl-pesticides.eu/userfiles/file//EUPT-SRM16_Calendar.pdf)
The list of analytes potentially contained in the Test Item is shown in the EUPT-SRM16 Target Pesticides List (https://www.eurl-pesticides.eu/userfiles/file//EUPT-SRM16_TargetPesticideList.pdf).

For detailed information please refer to the EUPT-SRM16_Announcement (https://www.eurl-pesticides.eu/userfiles/file//EUPT-SRM16_Announcement.pdf)**.**

Best regards,
Your EURL-SRM Team

ANNOUNCEMENT/INVITATION

EUPT – SRM16

(Matrix: Sesame Seeds)

(update on: 05/03/2021)

Dear Colleagues,

We herewith cordially invite you to participate in the upcoming European Proficiency Test on the analysis of residues of pesticides requiring single residue methods (EUPT-SRM16). This exercise is organized by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM).

The EUPT-SRM16 is scheduled to run from 22 March till 27 April, 2021.

All relevant documentation is linked within the **EUPT-SRM16-Website.**

AIMS

Participation in proficiency tests is part of the QA/QC system of laboratories. It provides them with an assessment of their analytical performance and allows them to make a comparison with the performance of other laboratories. The general aim is to help laboratories demonstrate adequate analytical performance and, in case of underperformance, to help them identify sources of errors, so that the necessary measures for quality improvement can be taken.

TEST ITEM and BLANK MATERIAL

The commodity for the Test Item will be **milled sesame seeds (hulled non-roasted white sesame).** The Test Item will foreseeably contain spiked and incurred pesticides.

One Test Item containing either incurred or spiked pesticides or both will be sent to each participating lab. The content of the Test Item will be expectedly in the range between 150 and 200 g. NO Blank material will be sent to the participants.

As the amount of available test material is limited, additional Test Item can be provided (against extra charge) only if sufficient explanations are given by the requesting laboratory, and only if excess material is available. To request double amount of material (two Test Items) please enter your request in the **EUPT-Registration Website** and contact the **eurl-srm@cvuas.bwl.de**.

TARGET ANALYTES

Analytes potentially contained in the Test Item are shown in the **Target Pesticides List.** For each of the analytes a specific minimum required reporting level (MRRL) is given. The Target Pesticide List may be updated prior to the start of the PT. The latest version will always be accessible within the **EUPT-SRM16-Website.** In case of important changes, the registered participants will be informed via e-mail.

DISCLOSURE OF INFORMATION

The names of the compounds contained in the Test Item will be disclosed to the participants within 3 days after the EUPT via e-mail. The preliminary assigned concentrations will be disclosed in the preliminary report, which will be released within 3 weeks after the deadline of the test.

METHODS TO BE USED

The use of routinely employed methods is preferred. However, participants are encouraged to use the EUPTs as a starting point for the expansion of their scope through the introduction of new methods and are, therefore, free in the choice of the methods applied in the EUPT.

SHIPMENT AND RECEIPT OF TEST ITEM:

The shipment of the Test Item is planned to start on **22 March 2021**.
If any laboratory will be on holiday in the week of the shipment, please inform the organizer by 12 March in order to arrange an alternative shipment.

Participants must check the integrity and condition of the materials upon receipt and to report **within 48 h** if they accept the materials or not. For this, please use the "**EUPT-SRM16 Result Submission website**". In case of problems with the sample receipt or condition, please additionally contact the organizers via e-mail (**eurl-srm@cvuas.bwl.de**) to ensure that corrective actions are taken as early as possible. In case of no reaction, the organizers will assume that the material has been accepted.

OBLIGED AND ELIGIBLE LABS

Participation in the EUPT-SRM16 is mandatory for:

- all NRLs for pesticides requiring Single Residue Methods (**NRL-SRMs**), see Art. 101 (1)(a) of Reg. (EC) 625/2017,
- all Official Laboratories (**OfLs**) performing pesticide residue analyses of commodities of plant origin with high lipid content or of feed, within the frame of National and EU official controls, see Art 38 (2) of Reg. (EC) 625/2017 and Art. 28 of Reg. (EC) 396/2005. This includes laboratories involved in the import controls of products listed under Reg. (EU) 1793/2019[1]; and especially those labs who are involved in import controls of sesame samples.

Based on the data stored in the Lab-Network Database about the commodity scope and the status of each lab, each laboratory is classified as obliged or not obliged to take part in this PT. This information can also be found on the EUPT-Registration page. Errors should be reported to the corresponding NRL and to **eurl-srm@cvuas.bwl.de**, accompanied by a brief explanation.

[1] Reg. (EU) 1793/2019 repealed Reg. (EC) No 669/2009. Labs conducting official analyses within the frame of this regulation were internally classified as "669-Labs"

This EUPT is furthermore open to the following laboratories as long as sufficient material is available:

- **any other OfLs from EU countries** that are not covered by the above obligations to participate;
- **laboratories analysing official organic samples** within the frame of Reg. 889/2008/EC[2];
- NRLs and OfLs from **EU-candidate countries** and **EFTA countries**;
- Laboratories from **Third Countries** (countries outside EU) as long as they are **involved in controls of products destined for export to the EU**.

REGISTRATION

The registration for the EUPT-SRM16 will run through the **EUPT-Registration Website** that is connected with the **EURL-DataPool**. To register for the EUPT-SRM16, you will need to login to the **EUPT-Registration Website** using your **EURL-DataPool login credentials**. If you are not yet registered in the EURL-DataPool, you will need to register firstly. If you have lost your login credentials, please use the "forgot password" feature.

The registration period is open from 23 February to 08 March, 2021. An instruction on EUPTs registration can be found on the **EUPT-Registration Website**.

For more information on how to register, you may consult to the following documents:

- **Obliged EU-Official Laboratories**
- **Voluntary Laboratories**

OBLIGED LABS NOT PARTICIPATING:

DG-SANTE expects from all **obliged labs not intending to participate** in this EUPT to **give an explanation**. The aim is to track all explanations for non-participation provided by the affected labs within the database. Therefore, please **enter this information only directly into the EUPT-Registration Website** and please **do NOT submit it via e-mail**. If you do so, you will still be prompted to access the website and enter your explanation there.

All obliged labs should thus access the Registration Website, regardless of whether they intend to participate or not.

IMPORTANT DATES

- **The EUPT-SRM16 registration form within the "EUPT-Registration Website" will be accessible from 23 February to 08 March, 2021.**
- The **shipment** of the Test Items is planned to start on **22 March, 2021.**
- **Results and method information** should be submitted by **27 April at 23:30 h (11:30 p.m.) CET** on the "**EUPT-SRM16 Result Submission website**".

[2] Internally classified as „889-labs"

PARTICIPATION FEE and PAYMENT

A general fee of **200 €** for one bottle Test Item will be charged to each participating laboratory **from EU Member States, EU-candidate countries or EFTA countries**, to cover the costs of handling and shipment. The fee for labs from **third countries** is set at **350 €**.

An invoice issued for the "invoice address" stated in the registration form will be sent after the shipment to the e-mail address(es) of the person(s) responsible for the PT and, if stated during registration, also to the person responsible for the payment. **Details on payment will be given in the invoices**.

Additional cost may occur if extra services are requested in relation to the payment or if invoices have to be changed due to new information, or requesting of it being resent.

RELEVANT DOCUMENTS

All documents relating to EUPT-SRM16 will be uploaded onto the **CIRCA-BC platform** and linked to the **EUPT-SRM16-Website.**

The schedule for all activities and deadlines within this PT can be found in the **EUPT-SRM16 Calendar**.

The pesticides potentially present in the Test Item can be found in the **EUPT-SRM16 Target Pesticides List.**

One week prior to the start of the EUPT-SRM16, the **EUPT-SRM16 Specific Protocol** will be published. This should be read carefully. Please also refer to the valid version of the **General EUPT Protocol**, which entails the general evaluation rules of EUPTs.

GENERAL INFORMATION, CONFIDENTIALITY, DISCLAIMER

The EUPT-SRM16 is organized by the EURL-SRM on behalf of DG-SANTE. DG-SANTE is the proprietor of all EUPT data and has thus access to all information. This also includes the Directorate on Health and Food Audits and Analysis.

- In each EUPT, the participating laboratories are given a unique code, initially only known to themselves and the organisers. In the final EUPT-Report, the list of participating laboratories will not be linked to their laboratory codes.
- **The participating laboratories are not allowed to communicate with each other on matters concerning the EUPT from the start of the EUPT until the publication of the preliminary report.**
- **The organizers are allowed to provide NRLs with the EUPT-SRM16 codes of all OfLs in their respective networks.**
- The organizers further reserve the right to share EUPT results and codes with other EURLs.
- The organisers may further present the EUPT-results on a country-by-country basis. For those EU **countries where only one laboratory has participated**, the identification of certain laboratories could thus be indirectly revealed.
- All laboratories are requested to **provide information on the analytical methods used**. 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 to exclude the lab from the final report.

- Please note that **all documents mentioned above may be subject to minor changes**. In the case of important changes, participants will be informed by e-mail. **But please still** check **periodically the EUPT-SRM16-Website for possible updates** in case the email does not get through to you.

SUPPORT AND CONTACT INFORMATION

The EUPT-SRM16 organizing Team is always at your disposal to answer any questions and give you technical support. For any further questions about the EUPT-SRM16, please mail to eurl-srm@cvuas.bwl.de.

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EUPT SCIENTIFIC COMMITTEE

Quality Control Group

Paula Medina	European Food safety Authority (EFSA)
Antonio Valverde	University of Almería, Spain

Advisory Group

Amadeo R. Fernández-Alba	Co-Head of EURL FV, University of Almería, Spain
Carmen Ferrer Amate	Co-Head of EURL FV, University of Almería, Spain
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André de Kok	Formerly working at NVWA, Wageningen, The Netherlands
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Best regards,

The EUPT-SRM16 Organising Team

Appendix 11 (cont.) Announcement and Call for Registration for the EUPT-SRM16

CALL FOR REGISTRATION A11

Guide to EUPT-SRM16 Results Submission Webtool

Version: 2021-02, Date: 19-03-2021, Init: Schr

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

General Information:

- **Please use Chrome or Firefox as Web-browser and incognito mode.**
  The Webtool is not validated for internet explorer, Microsoft Edge or other browsers.
- **Your data is automatically saved as soon as you move the cursor from one edited line 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 entries anymore!**

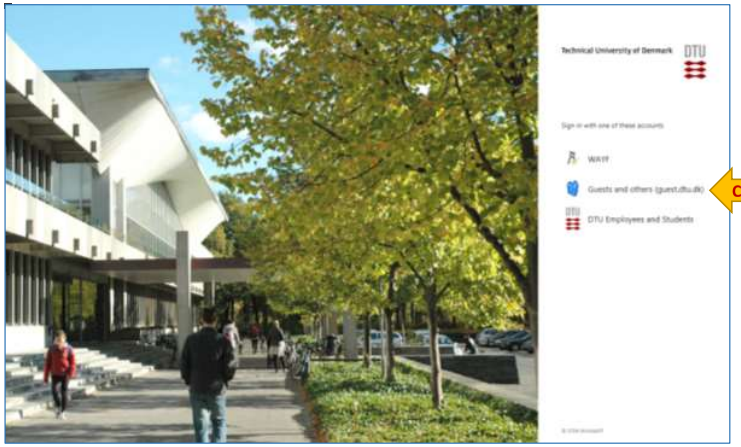
Browser requirements: The system works only with the browsers Google Chrome and Firefox.
The latest version is recommended.
Please access the Webtool using the incognito mode.

Please don't use Internet Explorer or Edge!

Getting started

Link to Webtool: www.eurl.dtu.dk

Choose "**Guest and others**"



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

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

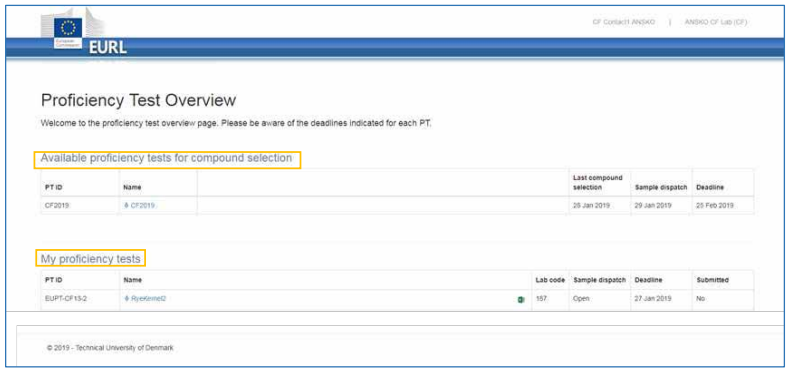
Please note:
The PT-Organizers have access only to your original login credentials. Any changed or new password is known to you yourself only. Therefore, we recommend never using "change password".

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

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

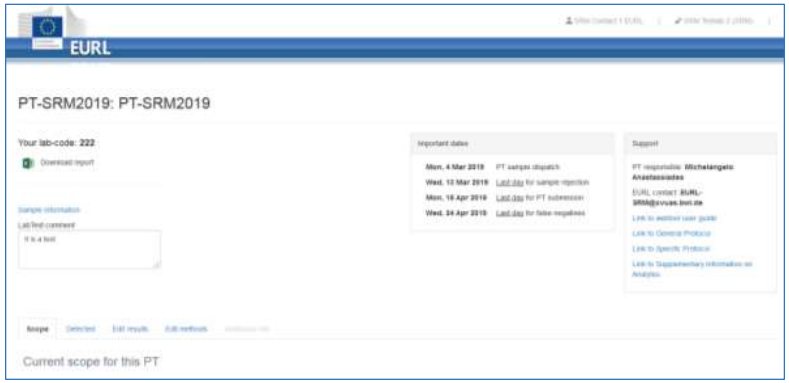
Proficiency Test Overview

On the **page *"Proficiency Test Overview"***, you will see on the top the section "*Available proficiency tests for compound selection*" with the PTs that are available for compound selection and below the section "*My proficiency tests*" showing information 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 concerned PT is open and you login to the Webtool for the first time.



By clicking on "EUPT-SRM16 | Sesame Seeds" under "*My proficiency tests*", you will see the **current scope for this EUPT** in alphabetic order.

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 are able to edit your targeted compounds from opening of the Webtool to the deadline for result submission.**



Sample receipt and acceptance

The **Webtool for the EUPT-SRM16 result submission** will expectedly open on **23 March 2021**.

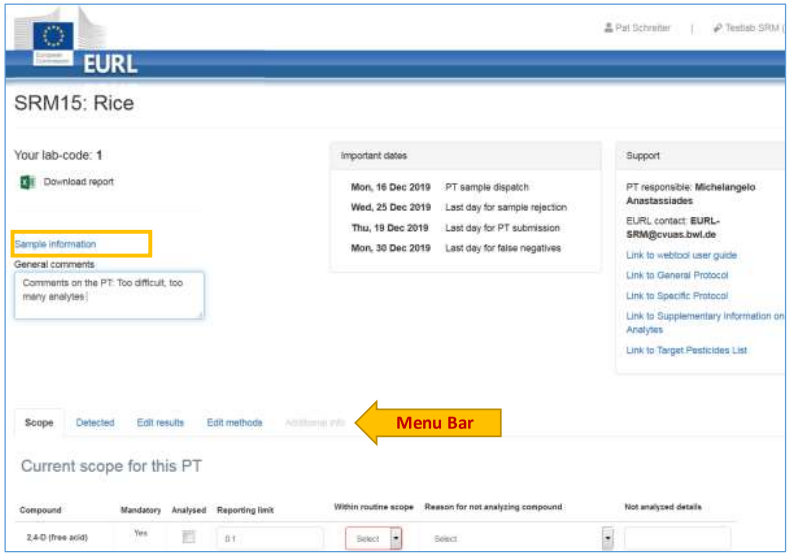
Once you have received the parcel with the PT-materials, please click on EUPT-SRM16 under "*My proficiency tests*" to open the pop-up window "**Edit sample Receipt"**. Please fill-in the information requested within this pop-up-window:

- **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 in your institution.
- **Remarks e.g. on dry ice condition:** Please enter here any remarks concerning the condition of the parcels and sample bottles, the temperature, etc.
  Please note: No dry ice is used for the shipment of the SRM16 material.

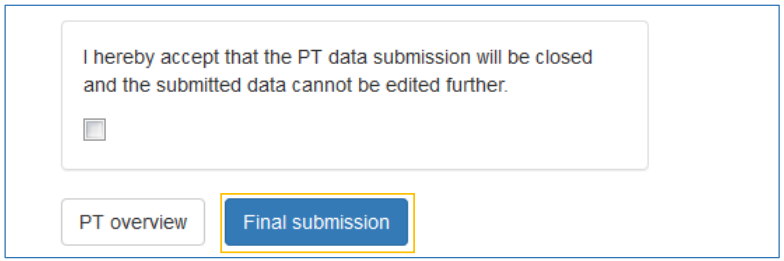
Completing the "*Edit Sample Receipt*" window is a **precondition for being able to continue the submission page.** This should be done **ideally shortly after parcel receipt** and **not later than 27 March**. You can, however, access and edit all the entries on "*Edit sample receipt*" through the PT-period under "*Sample information*" (please see next page, left navigation bar).

Appendix 12. Guide to EUPT-SRM16 Results Submission Webtool

Upon clicking on "*Save and close*" you will be guided to the following page in which you can see your **Lab-code,** a **button for downloading the report (=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"***.



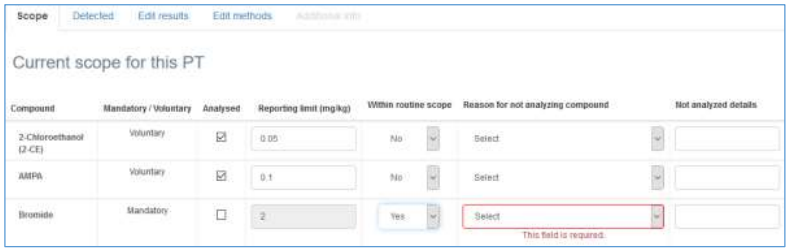
On the bottom of each table you will see the button for ***Final submission***.
Use this button only after you have already entered all your data for this PT and want to submit them for the PT evaluation.
After the Final submission you will NOT be able to change your data any more.



Scope

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

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



In this table, please firstly select the analytes you have targeted within the EUPT-SRM16 and then enter your Reporting Limit (RL) of each.

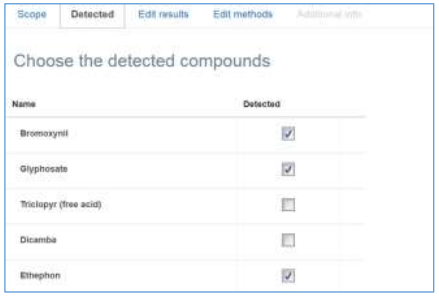
The MRRLs were set as default reporting limits. For each pesticide within your PT Scope please **change the Reporting Limit to that of your laboratory**.

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

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

Detected

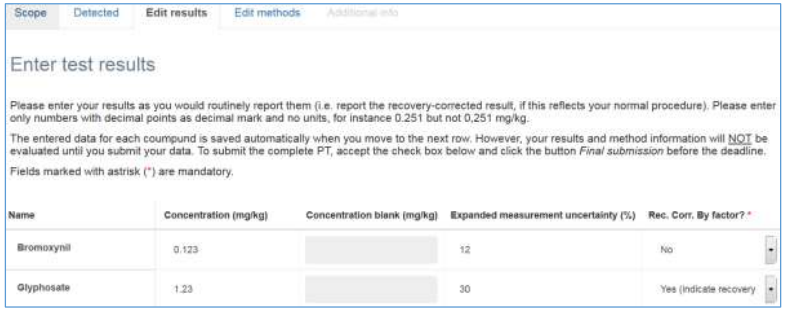
This page will list only analytes that were selected as "analysed" under the table "Scope". Please mark the analytes that you have detected in the Test Item. These selections are used as filters for the subsequent "*Edit results"* table.



Edit results

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

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



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

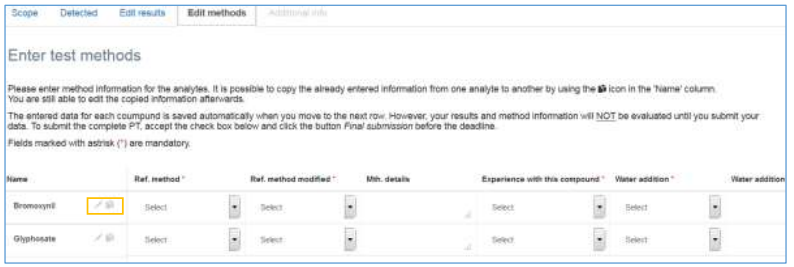
Field(s)	Explanation
Concentration [mg/kg] **(=Concentration in Test Item)**	Concentration in Test Item in mg/kg, (syntax: e.g., 0.345) **Only numerical values are accepted; pls. use points for decimal separation.** An not numerical entry, such as "< RL", may be judged as a "False Negative" result if the compound is present in the Test Item and your RL is higher than the MRRL or the assigned value.
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 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	Here you may add any remarks concerning aspects covered by this subpage as well as **any information concerning the SUBCONTRACTING of this analysis to another lab**. Please name subcontracted lab and indicate if this subcontracting reflects routine procedures

Guide to EUPT-SRM Results Submission Webtool

Edit methods

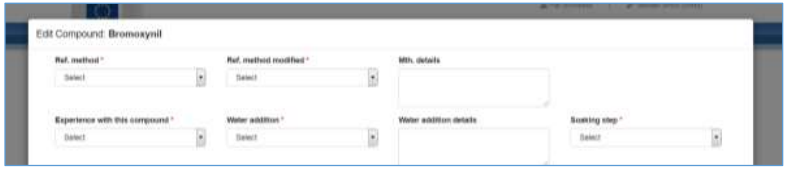
Click on the "*Edit methods*" tab to start reporting the method information.

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



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

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



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

Field(s)	Explanation
Ref. method	Choose from the drop-down list. If you have used a modified form of the mth, pls. give details under "Mth Details"
Ref. method modified	Specify if you have introduced any noteworthy modifications to the selected reference method. 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)
Soaking step prior to extraction?	Please indicate whether your sample was left to soak with water or the extraction solvent prior to the extraction step.
Soaking Time [min]	Soaking time (in minutes) of the sample with water/water containing solvent. Please choose the closest value.
Sample Weight (g)	Enter the weight (in gram) for the analytical portion.
Extraction/partitioning solvent 1	Choose the solvent from the drop down menu

Field(s)	Explanation
Extraction/partitioning solvent 2	Choose the solvent from the drop down menu, if you use more than one solvent.
Extraction/partitioning solvent 3	Choose the solvent from the drop down menu, if you use more than two solvents.
Extraction solvent details	Enter details on solvents used in extraction or partitioning steps or if the solvent is not in the drop down menu
Extraction Time [min]	Duration (in minutes) of main extraction step including any waiting time after addition of solvent (Please choose the closest value). If extr. is combined w. a chem. transformation then chose "Combined w. chem. transformation" and indicate the time under "Hydrolysis /Trasformation time"
Extraction approach	Choose extraction approach from drop-down list
Partitioning salts used	Choose partitioning salt used from drop-down list
pH modified	Indicate if you have modified the pH at any stage of the procedure (e.g. by buffering, acid/base addition)
pH modified details	Please give details on pH modification step(s)
Clean up 1	Choose the clean-up approach employed from the drop-down list
Clean up 2	Choose the clean-up approach employed from the drop-down list, if you use more than one clean up step
Clean up 3	Choose the clean-up approach employed from the drop-down 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 drop down 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
Headspace or SPME	Here you can choose your headspace or SPME method
Determ./Headspace/SPME Details	Here you can add any relevant details on Determination technique used e.g. columen, mobile phase, Headspace or SPME sampling (incubation time/temperature; injected volume; SPME fiber type)
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.

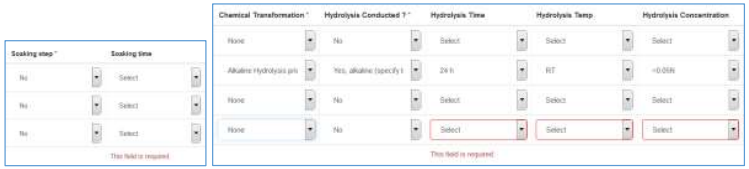
IS used	**Please choose "No" if no "IS" was used or if the IS was only used for quality control purposes and not for the calculation of the target analyte result**. Please choose one of the two "Yes" options if the IS was used for calculating the target analyte concentration.
IS Name	Please give details on the IS used
When was IS added?	Mark at what stage of the procedure the IS was added
Comments **(=General Comments on method 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

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.

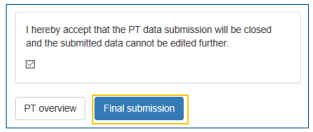
Please note:

- The red rings or information showing that a field is required are not always automatically updated after entering or saving data. You may have to actively click the cells to see the updated status.
- In some cases, in particular the fields concerning soaking step and hydrolysis/transformation, 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".

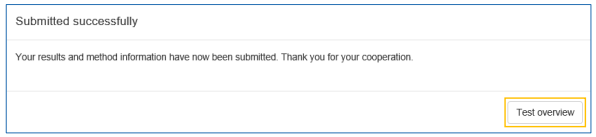
When all fields are filled out and you have checked their correctness, you are ready to submit your results. Accept and submit your final results by clicking the check box and then click on "*Final submission*".



IMPORTANT:

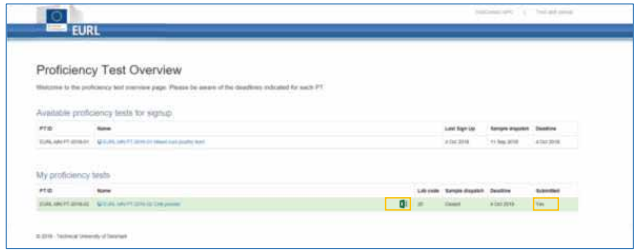
You will **NOT** be able to edit your data after the final submission!
Your data have to be submitted before the deadline on Tue. **27 April 2021, 23:30 h (11:30 p.m.), CEST.**

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.



Additional Information

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

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

Click on the yellow-marked EUPT and fill in **the missing method information for the compounds identified as tentatively false negatives. Submit this information by 6 May.**

Tentatively false positives are written in red colour.

Tentatively false negatives are written in yellow colour.

European Union Reference Laboratory
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