European Commission

EURL-SRM

EU Reference Laboratories for Residues of Pesticides
Single Residue Methods

# EU Proficiency Test on the Analysis of Tomato Homogenate for Residues of Pesticides Requiring Single Residue Methods

## EUPT–SRM17

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## Final Report

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cvua STUTTGART

# EU PROFICIENCY TEST
# EUPT-SRM17, 2022

# Residues of Pesticides Requiring Single Residue Methods

## Test Item: Tomato Homogenate

## Final Report

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**approved by Michelangelo Anastassiades**
**released on 20 February, 2024**

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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 organised 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 as follows: "Upon request by the European Union reference laboratory or national reference laboratory, official laboratories shall take part in inter-laboratory comparative tests or proficiency tests that are organised for the analyses, tests or diagnoses they perform as official laboratories". Art 101 (1)(a) of 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. Six of those 17 EUPT-SRMs were conducted in collaboration with the EURL for pesticide residues in Fruits and Vegetables (EURL-FV) using apple juice (EUPT-SRM1, 2006), carrot homogenate (EUPT-SRM3, 2008), apple purée (EUPT-SRM5, 2010), potato homogenate (EUPT-SRM8, 2013), spinach homogenate (EUPT-SRM11, 2016) and tomato homogenate in the present one (EUPT-SRM17, 2022) as test items. Five other EUPT-SRMs were conducted in collaboration with the EURL for pesticide residues in Cereals and Feeding Stuff (EURL-CF) with wheat flour (EUPT-C1/SRM2, 2007), oat flour (EUPT-C3/SRM4, 2009), rice flour (EUPT-C5/SRM6, 2011 and EUPT-SRM15, 2020) and maize flour (EUPT-C9/SRM10, 2015) as test items. One EUPT-SRM (EUPT-SRM14, 2019) was organised in cooperation with the EURL for Residues of Pesticides in Food of Animal Origin (EURL-AO) using bovine liver homogenate as test commodity. Further five EUPT-SRMs were organised by the EURL-SRM unilaterally. One concerning a commodity of animal origin, namely cow's milk (EUPT-SRM9, 2014) and the remaining four concerning commodities of plant origin, namely, milled dry lentils (EUPT-SRM7, 2012), strawberry homogenate (EUPT-SRM12, 2017), soyflour (EUPT-SRM13, 2018) and sesame seeds flour (EUPT-SRM17, 2021). The latter was chosen as a reaction to the massive incidence with residues of ethylene oxide in sesame and other products.

Participation in the respective EUPTs is mandatory for all NRLs for pesticides requiring Single Residue Methods (NRL-SRMs) and for all OfLs analysing pesticide residues within the framework of national or EU control programs in commodities represented by the respective EUPT test item. Laboratories in EU Member States analysing pesticide residues within the frame of import controls according to 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, as well as OfLs from EU-acceding or -candidate countries (North Macedonia,

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

Montenegro, Serbia and Turkey) are also invited to take part in EUPTs. A limited number of laboratories from third countries, in particular if they are involved in the control of food or feeding stuff exported to EU member states are allowed to take part in this exercise, too. However, only results submitted by labs from EU and EFTA countries are included in the calculation of the assigned values.

Based on information about the commodity scope and labs' NRL-status a tentative list of EU-labs considered to be 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 in, are asked to state the reason(s) for their non-participation. The same applies to laboratories originally registering to participate in a certain EUPT but finally not submitting results.

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

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**EUROPEAN COMMISSION –**
**EU-PROFICIENCY TEST ON RESIDUES OF PESTICIDES**
**REQUIRING SINGLE RESIDUE METHODS**
**TEST ITEM: TOMATO HOMOGENATE**
**EUPT-SRM17, 2022**

# INTRODUCTION

On 8 November 2021 the EUPT-SRM17 Announcement/Invitation Letter (**Appendix 11**), as well as the Calendar and the preliminary Target Pesticides List (TPL) were published on the EUPT-SRM17-Website. All relevant National Reference Laboratories (NRLs) of the 27 EU-Member States (MS), as well as all relevant EU-Official Laboratories (OfLs) whose contact details were available to the organisers were invited to participate.

Following consultation with the EUPT-Scientiffic Committee, the preliminary TPL was prepared, which entailed 31 compulsory and 31 optional analytes. All analytes on the preliminary TPL were allocated a residue definition valid for the PT and the minimum required reporting level (MRRL). The preliminary TPL included compounds entailed in the latest Regulation on the EU-coordinated monitoring program, the entries within the SANTE working document on pesticides to be considered in national control programmes, the relevance of compounds for tomato and fruiting vegetables in general, the availability of analytical standards, and the availability of pesticide formulations in Spain, where the tomatoes were grown.

For refining the preliminary TPL, a survey was conducted from the 11$^{th}$ till the 19$^{th}$ of November 2021, among the potential participants (EU/EFTA-OfLs and NRLs), which revealed the actual capability of those laboratories. The survey furthermore allowed to localize the interrest of laboratories on certain compounds to be included in the PT as well as the cases, where the number of expected results would be too low for a reliable statistical evaluation. Based on the survey results and consultation with the EURL-FV in the premisses of which (University of Almería) the tomatoes were cultivated for both the EUPT-SRM17 and the EUPT-FV24, the final TPL was drafted by reducing the number of analytes to 17 compulsory and 17 optional ones. This new TPL was published on 26 November 2021 and updated on 28 January 2022 (**Appendix 10**).

NRL-SRMs and all OfLs analysing pesticide residues in fruits and vegetables within the framework of official controls, including those involved in the import controls of products listed under Reg. (EU) 1793/2019, as far as they could be tracked in the EURL-DataPool, as well as EU laboratories officially analysing organic samples within the frame of Reg. 889/2008/EC, were called for registration on 6 December 2021. NRLs and OfLs from EFTA and EU-candidate countries were also invited if their contact data were available. In addition official and commercial laboratories from 3$^{rd}$ countries, in particular those involved in the export control of foods or feeding stuff to the EU, were also invited and accepted as participants in the present PT. The results from laboratories outside EU and EFTA OfLs were, however, not taken into account for the establishment of the assigned values of all analytes.

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-SRM17. This status was stored in the DataPool, so that every participant could see it during the registration. To ensure that all concerned official labora-

tories were informed about this EUPT, the NRLs were asked to forward the invitation to all relevant OfLs within their countries. It was made clear, that the status of the laboratories was only tentative, and the real obligation to participate was based on the respective regulations. From 6 December till 31 December 2021 laboratories obliged to or interested in paricipating 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 prompted to register for non-participation and state their reason. The SRM17 Specific Protocol **(Appendix 9)** was provided to the participants on 11 January 2022 by distributing a hyperlink via e-mail. The SRM17 Webtool Guideline **(Appendix 12)** was available to the participants through a link entailed in an mail sent by the DTU Webtool Admin and which additionally contained the login credentials and was also available within the main page of the webtool. The link was provided to the participants also on 10 February, when the opening of the webtool was announced.

In total, 123 official laboratories (including NRLs) from 33 countries (26 EU-Member States, 2 EFTA-countries, 1 EU candidate country and 3 countries outside Europe) registered for participation in the EUPT-SRM17 and reported at least one result for this PT.

The tomato used to produce the EUPT-SRM17 test material contained both incurred (field-sprayed) and non-incurred compounds, which were spiked to the homogenate. The cultivation of the tomato was subcontracted to the EURL-FV. According to the agreement with the EURL-SRM the chosen analytes and their corresponding concentrations were applied during growing. After harvesting the tomato was finely chopped and shipped deeply frozen to the EURL-SRM for further processing, which entailed spiking with standard solutions, followed by thorough homogenization and cryo-milling with dry ice and portioning into bottles. More details are given in **Chapter 1 "TEST ITEM"**.

# 1. TEST ITEM

## 1.1 Selection of PT-Commodity

The last EUPT-SRM using a fruit or vegetable as commodity was the EUPT-SRM12, that took place in 2017. Since then, three EUPT-SRMs using dry commodities (sesame, rice and soybean in 2021, 2020 and 2018, respectively) and one using calfs' liver (2019) were conducted. The original plan was to run a PT on a vegetable matrix in 2021 (EUPT-SRM16). During a meeting of the EUPT-Scientiffic Commitee in 2020 aubergines were selected as matrix for the EUPT-SRM16. But due to the ethylene oxide incidence that happened in late 2020, DG-SANTE requested to use sesame seeds as matrix and to focus, among others, on residues related to ethylene oxide fumigation (EO and 2-CE). During a meeting of the EUPT-Scientific Committee in 2021, it was decided to use tomatoes as commodity for the EUPT-SRM17 and the EUPT-FV24, to be organized in 2022 by the EURL-SRM and the EUPT-FV, respectively. The EUPT-FV took over the task of growing the tomatoes in a green house belonging to the University of Almería in Spain.



## 1.2 Production of Tomatoes with incurred pesticides and preparation of homogenate

Tomato seeds were sown in September 2021, transplanted into the greenhouse soil in mid-November, treated with the selected pesticides (see below) in mid-December and harvested in late December. The harvested material was placed in a refrigerator overnight, milled using knife mills, filled into 10 kg buckets, tightly sealed, and put in the freezer. 10 buckets each containing approx. 10 kg of deeply frozen ground tomato were shipped to the EURL-SRM for further processing.

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

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

1) the scope of the present and upcoming EU-coordinated control programs, such as the MACP-Regulation and the latest version of the DG-SANTE working document (SANCO/12745/2013) giving guidance for the design of national monitoring programs;
2) dithiocarbamates-Relevant compounds of toxicological concern;
3) Relevance to matrix (tomato) / matrix group (high water content);
4) the capabilities and interrests of the potential participants as revealed through a survey on SRM17 Target Pesticides run among the OfLs in November 2021;
5) suggestions / votes by EUPT-Scientific Committee;
6) the need to keep the number of different methods, required to cover the full scope of analytes, reasonably low.

The minimum required reporting levels (MRRLs) were set as follows: (Note: the compounds present in the test item are highlighted in bold italic).

- at 0.01 mg/kg for ***2,4-DNOP,*** *avermectin B1a, chlormequat (chloride),* ***chlorothalonil,*** *DDAC-C10 (chloride),* ***dodine, emamectin B1a, ETU,*** *fenbutatin oxide,* ***formetanate-HCl,*** *mepiquat (chloride),* ***meptyldinocap (sum),*** *nicotine,* ***oxymatrine,*** *paraquat,* ***phthalimide,*** *PTU, TFNA, TFNG,* ***THPI,*** *trimesium cation,*
- at 0.02 mg/kg for *BAC-C12 (chloride),* ***captan, chloridazon-desphenyl, cyromazine,*** *dithianon,* ***dithiocarbamates, folpet,*** *matrine,* ***meptyldinocap, pymetrozine;***
- at 0.03 mg/kg for ***bifenazate (sum)*** and *diquat*,
- at 0.05 mg/kg for ***maleic hydrazide***

**Table 1-1:** Analytes present in the SRM17 test material and their application history

| Mandatory Analytes | Residues incurred | Spiked in lab | Compounds applied in lab | Optional Analytes | Residues incurred | Spiked in lab | Compounds applied in lab |
|---|---|---|---|---|---|---|---|
| Captan | Yes | Yes | Captan | Bifenazate | Yes | No | – |
| Tetrahydro phthalimide (THPI) | Yes | No | – | Chloridazon-des-phenyl | No | Yes | Chloridazon-desphenyl |
| Chlorothalonil | Yes | No | – | ETU | No | Yes | ETU (partly from metiram and partly spiked on top) |
| Cyromazine | No | Yes | Cyromazine | Formetanate-HCl | Yes | No | – |
| Dodine | No | Yes | Dodine | Maleic hydrazide | No | Yes | Maleic hydrazide |
| DTC (CS2) | No | Yes | Metiram (CELAFLOR) | Meptyldinocap | No | Yes | Meptyldinocap |
| Emamectin B1a | Yes | Yes | Emamectin benzoate | 2,4-DNOP | No | Yes | 2,4-DNOP (free phenol) |
| Folpet | Yes | Yes | Folpet | Oxymatrine | No | Yes | Oxymatrine |
| Phthalimide (PI) | Yes | Yes | Phthalimide | | | | |
| Pymetrozine | Yes | No | – | | | | |

## 1.4 Analysis of the Tomatoes Intended to be Used to Prepare the PT-Material

Seven days after the treatment of the tomatoes on the field, a small amount of tomatoes were harvested, homogenized and shipped to the EURL-SRM for a preliminary check of the levels contained. For some of the tomatoes it was decided to conduct a second spraying and the harvest date was decided.

Finally, approximately 100 kg of frozen homogenate of the tomatoes was received in December 2021 by the EURL-SRM. Thereafter, the material was analysed for any residues of TPL-pesticides, using the QuEChERS method, QuEChERS variants (for ***meptyldinocap*** and ***bifenazate (sum),*** the QuPPe method and the method for dithiocarbamates involving conversion to carbon disulfide ($CS_2$). Considering the encountered levels of "incurred" pesticides and metabolites, it was decided which of the compounds would need to be overspiked (to increase the present levels) and which ones should be additionally spiked. **Table 1-1** gives an overview of the compounds used in the field and the compounds spiked in the laboratory.

## 1.5 Pre-Test to Check Analyte Stability in Tomato Homogenate

Different portions of tomato homogenate, just thawed but still very cold (< 2 °C), were spiked with the pesticides on the TPL. The spiked portions were left standing for either 0, 4 or 15 hours at room temperature before extraction in order to check the potential instability of the TPL-compounds and decide about the final spiking procedure to be followed. Most compounds were stable with a few exceptions: ***Captan*** was degraded by 40 – 50 % within 4 h and was not detectable anymore after 15 h. ***Folpet*** showed losses of ~5 % after a 4 h delay and losses of ~70 % after 15 h. ***Chlorothalonil*** degraded by ~30 %. ***Formetanate*** and bifenazate degraded by ~20 % each within 15 h storage at room temperature. *Dithianon*, which wasn't present in the PT-Item, degraded by ~50 % after 4 h and was not detectable after 15 h.

## 1.6 Preparation and Bottling of the Test Item and Preliminary Homogeneity Test

Two days before spiking, the buckets containg the 10 kg portions of frozen tomato-homogenate were removed from the freezer and left standing at room temperature to defrost. On the date of spiking, 90.5 kg

**Table 1-2:** Analytes spiked into 95.5 kg tomato homogenate for the preparation of EUPT-SRM17 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 |
|---|---|---|---|---|
| **Captan** | 1.0 | 15.0 | 0.167 | yes |
| **Chloridazon-desphenyl** | 1.0 | 8.0 | 0.089 | |
| **Cyromazine** | 1.0 | 13.0 | 0.144 | |
| **Dodine** | 1.0 | 10.0 | 0.111 | |
| **Emamectin B1a** | 1.0 | 3.0 | 0.033 | yes |
| **Folpet** | 1.0 | 25.0 | 0.278 | yes |
| **Phthalimide** | 1.0 | 7.0 | 0.078 | yes |
| **ETU** | 1.0 | 4.0 | 0.044 | yes |
| **Maleic hydrazide** | 5.0 | 10.0 | 0.556 | |
| **Meptyldinocap** | 1.0 | 9.0 | 0.100 | |
| **2,4-DNOP** | 1.0 | 5.0 | 0.056 | |
| **Metiram (CELAFLOR)** | 0.7 | 76.6 | 0.334 | |
| **Oxymatrine** | 2.0 | 8.5 | 0.189 | |

of the partly defrosted tomato-homogenate, which was to the most part defrosted, was transferred into a large plastic container and mixed using a high shear batch mixer. In parallel, the tomato homogenate portion that was used for spiking was prepared. For this, 5 kg of fridge-cold blank tomatoes were homogenized using a knife mill, and spiked with the selected pesticides except of metiram. These pesticides were dissolved into 150 mL of a solvent mixture composed of water, acetonitril and methanol 80 / 10 / 10 % and after adding them the material was thoroughly mixed for 5 minutes using a powerfull knife mill. The spiked homogenate was then mixed with the rest of the material and mixing continued for further 60 min. The amounts of pesticides spiked to the material are stated in **Table 1-1**. Metiram was spiked directly into the large portion of 95.5 kg (90.5 + 5 kg) in form of an aqueous suspension of a commercial plant protection product. In total 79 mL of the suspension containing 55 mg of metiram (based on the active subtance content on the label) were used. Metiram was spiked with a 40 min delay compared to the other pesticides and therefore only mixed with the entire material for 20 min. During homogenization, the initial temperature of 0 °C raised to 2 °C after one hour of mixing. After mixing, the spiked homogenate was filled into zip seal plastic bags. Ca. 600 – 800 g of material were filled into each bag and the bags were placed in a freezer in a flat position in order to get thin plates that are easy to handle during the final mixing step with dry ice. The initial homogenate was further mixed in 500 – 600 g portions with the help of a knife mill and after the addition of dry ice. This procedure resulted in a free-flowing snow-like material, that could be conveniently portioned by the participants. The material was swiftly filled into numbered bottles, which were sealed with a lid, and swiftly placed into a freezer to ensure that the snow-like consistency is maintained.

### 1.6.1 Preliminary Analysis Prior to Bottling

To double-check the spiking levels and to verify that the material is sufficiently homogeneous, prior to bottling, 8 portions were taken from the initial homogenate (96 kg) and analysed for selected compounds using the dithiocarbamate method, the QuEChERS method and the QuPPe method. The sample material was found to be homogeneous (RSD 2 – 8 %) and fit for further processing and packaging.

## 1.7 Packaging and Delivery of PT Materials to Participants

On the day of dispatch, one test item (bottled PT-material) was packed into one thermo-insulated polystyrene box, filled-up with dry ice pellets (2 – 3 kg in each box) and transported by DHL-Express to each of the participating laboratories. Two boxes, each containing one test item were sent to laboratories having ordered double amount. Once the parcel was picked up by the shipping company (DHL Germany), the main PT corresponding person of each participating laboratory received an e-mail from DHL entailing the individual online tracking number

Among the 123 packages sent to laboratories in EU and EFTA countries, 110 (89 %) reached the participating labs within 24 hours, eight packages (7 %) arrived within 48 hours and five packages arrived the recipient laboratories on the third day due to remote location or thunderstorm. In total, 96 % of the parcels arrived at the EU- and EFTA-laboratories within two days, and thus in frozen condition. All material was accepteded by the participants and was reportedly in very good condition, even when arriving on the third day. Based on these results, it was supposed that differences in shipment duration within the EU most likely didn't signifficantly influence the analyte concentrations and the analytical results of the laboratories. It was, therefore, decided not to further investigate the impact of shipment duration on analyte stability (see also **Section 1.11 „Transport Stability Test", p. 8**).

The five shipments to laboratories located in countries outside the EU and EFTA zones was accomplished within 24 hours (1×), 3 days (1×), 7 days (2×) and 9 days (1×). The latter three cases were due to prolonged customs clearance. In one of the two cases where the shipment lasted 7-days, the test item was maintained in deeply frozen condition thanks to special precautionary measures provided by the transport company. In the other case with 7-day shipment duration the material arrived in defrosted but reportedly cold condition. The test item that was received by the laboratory with a 9 days delay was at ambient temperature on arrival. Since the results reported by laboratories outside the EU and EFTA zones are not taken into account when establishing the assigned values, any changes of the analyte concentrations in the material provided to those participating laboratories will have no influence on the estimate of the assigned values. Still, those special cases need to be considered when evaluating the labs' performance. Details on the shipment duration are shown in **Appendix 2**.

*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 for placing the parcel into the freezer until clearance is granted. The participants are furthermore encouraged to contact the local office of the shipping company to ensure optimal delivery timing.*

## 1.8 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 (p. 5).** 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).

## 1.9 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 taken for each analytical method. Both the order of sample preparation

**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 listed in the TPL.

**QuEChERS Method [3]:**
involving: weighing of 10 g tomato homogenate into a sealable vessel, addition of IS/ILIS, addition of acetonitrile, shaking, addition and distribution of salt mixture, centrifugation and direct determination by GC-MS/MS and LC-MS/MS.

| Compound | IS | Determinative analysis | | Notes |
|---|---|---|---|---|
| **Captan** | **Captan D6** | **GC-MS/MS** | **EI (pos)** | |
| **THPI Tetrahydrophthalimide** | – | **LC-MS/MS** | **ESI (pos)** | |
| **Chlorothalonil** | **Chlorothalonil $^{13}C_2$,$^{15}N_2$** | **GC-MS/MS** | **EI (pos)** | |
| **Dodine** | **Chlorpyrifos $D_{10}$** | **LC-MS/MS** | **ESI (pos)** | |
| **Emamectin B1a** | **Propyzamide $D_3$** | **LC-MS/MS** | **ESI (pos)** | |
| **Folpet** | **Folpet $D_4$** | **GC-MS/MS** | **EI (pos)** | |
| **Phthalimide** | **Propyzamide $D_3$** | **LC-MS/MS** | **ESI (pos)** | |
| **Formetanate-HCl** | **Formetanate $D_7$** | **LC-MS/MS** | **ESI (pos)** | |
| **Meptyldinocap** | – | **LC-MS/MS** | **ESI (neg)** | Special LC-gradient for separation |
| **2,4-DNOP** | – | **LC-MS/MS** | **ESI (neg)** | |
| *Avermectin B1a** | *Propyzamide $D_3$* | *LC-MS/MS* | *ESI (pos)* | |
| *Dithianon** | *Dithianon $D_4$* | *LC-MS/MS* | *ESI (neg)* | |
| *Fenbutatin oxide** | *Propyzamide $D_3$* | *LC-MS/MS* | *ESI (pos)* | |
| *TFNA** | *Propyzamide $D_3$* | *LC-MS/MS* | *ESI (pos)* | |
| *TFNG** | *Propyzamide $D_3$* | *LC-MS/MS* | *ESI (pos)* | |
| *BAC-C12 (chloride) ** | *Propyzamide $D_3$* | *LC-MS/MS* | *ESI (pos)* | |
| *DDAC-C10 (chloride) ** | *Propyzamide $D_3$* | *LC-MS/MS* | *ESI (pos)* | |

**QuPPe-PO Method [5]:**
involving: weighing of 10 g tomato homogenate into a sealable vessel, addition of ILISs, addition of methanol containing 1 % formic acid, shaking, centrifugation, filtration and direct determination by LC-MS/MS in the ESI (neg.) or ESI (pos.) mode

| Compound | IS | Determinative analysis | | Notes |
|---|---|---|---|---|
| **Cyromazine** | **Cyromazine $D_4$** | | | **QuPPe M4.2** |
| **Pymetrozine** | – | **LC-MS/MS** | **ESI (pos)** | **QuPPe M4.2** |
| **Chloridazon-desphenyl** | **Chloridazone-desphenyl $^{15}N_2$** | **LC-MS/MS** | **ESI (pos)** | **QuPPe M4.2** |
| **ETU (ethylene thiourea)** | **ETU $D_4$** | **LC-MS/MS** | **ESI (pos)** | **QuPPe M4.2** |
| **Maleic hydrazide** | **Maleic hydrazide $D_2$** | **LC-MS/MS** | **ESI (pos)** | **QuPPe M4.2** |
| **Oxymatrine** | **Oxymatrine $D_3$** | **LC-MS/MS** | **ESI (pos)** | **QuPPe M4.2** |
| *Chlormequat (chloride)** | *Chlormequat $D_4$* | *LC-MS/MS* | *ESI (pos)* | *QuPPe M4.2* |
| *Mepiquat (chloride) ** | *Mepiquat $D_3$* | *LC-MS/MS* | *ESI (pos)* | *QuPPe M4.2* |
| *Propamocarb*** | *Propamocarb $D_7$* | *LC-MS/MS* | *ESI (pos)* | *QuPPe M4.2* |
| *Diquat** | *Diquat $D_8$* | *LC-MS/MS* | *ESI (pos)* | *QuPPe M4.2* |
| *Matrine** | *Matrine $D_3$* | *LC-MS/MS* | *ESI (pos)* | *QuPPe M4.2* |
| *Nicotine** | *Nicotine $D_4$* | *LC-MS/MS* | *ESI (pos)* | *QuPPe M4.2* |
| *Paraquat** | *Paraquat $D_6$* | *LC-MS/MS* | *ESI (pos)* | *QuPPe M4.2* |
| *PTU (N,N'-(1,2-propylene)thiourea)** | *PTU $D_6$* | *LC-MS/MS* | *ESI (pos)* | *QuPPe M4.2* |
| *Trimesium** | *Trimesium $D_9$* | *LC-MS/MS* | *ESI (pos)* | *QuPPe M4.2* |

**QuEChERS followed by reductive conversion [7]**
involving: conversion step of bifenazate-diazene into bifenazate by adding aqueous ascorbic acid solution (30 % w/w) to an aliquot of the QuEChERS extract in a vial. Incubation overnight. Direct determination by LC-MS/MS

| Compound | IS | Determinative analysis | | Notes |
|---|---|---|---|---|
| **Bifenazate (sum)** | **Bifenazate D5** | **LC-MS/MS** | **ESI (pos)** | **QuPPe M4.2** |

* : To check for absence in Blank Material
**: present in sample material, but removed from TPL

**Table 1-3 (cont.):** Analytical methods used by the organisers to check for the homogeneity and storage-stability of the pesticides present in the test item and to demonstrate the absence of other pesticides listed in the TPL.

| **QuEChERS followed by alkaline hydrolysis [8]**<br>involving: transfer of an aliquot of the QuEChERS extract into a vial, addition of 25 % ammonia solution and incubation for approx. 16 hours at room temperature overnight. The hydrolysate was "neutralized" with concentrated acetic acid, followed by LC-MS/MS analysis. | | | | |
|---|---|---|---|---|
| **Compound** | **IS** | **Determinative analysis** | | **Notes** |
| **Meptyldinocap (sum)** | **–** | **LC-MS/MS** | **ESI (neg)** | **QuPPe M4.2** |
| **Dithiocarbamate method [9]**<br>involving: weighing of 10 g tomato homogenate into a sealable vessel, addition of chloroform (as IS) and 25 ml isooctane and 150 ml $SnCl_2$ /HCl, followed by cleavage to $CS_2$ in a shaking waterbath for 3 hours at 80 °C, followed by GC-MS/MS analysis | | | | |
| **Compound** | **IS** | **Determinative analysis** | | **Notes** |
| **Dithiocarbamates determined and expressed as carbon disulphide ($CS_2$)** | **Chloroform** | **GC-MS/MS** | **EI (pos)** | |

* : To check for absence in Blank Material
**: present in sample material, but removed from TPL

and the order of extract injection into the analytical instruments were random. Quantifications were done either using matrix-matched calibration standards prepared by spiking extracts of blank tomatoes or using procedural calibration standards prepared by spiking analytical portions of blank tomato homoganate. For all compounds, analytical portions of 10 g were used.

The statistical evaluation of the homogeneity test data was performed according to the ISO 13528:2015 "Statistical methods for use in proficiency testing be interlaboratory comparison" [6]. An overview of the statistical evaluations of the homogeneity test is shown in **Table 1-4 (p. 7)**. The individual residue data of the homogeneity test is given in **Appendix 3**.

The acceptance criterion for the test item to be sufficiently homogeneous for the Proficiency Test is that the estimate of the between-sample standard deviation $s_s$ is smaller than $0.3 \times \sigma_{pt}$, where $\sigma_{pt} = 0.3 \times$ FFP-RSD (25 %) × the analytical sampling mean of the analyte. In addition and for informative purposes only, the actual sampling error and repeatability were also calculated and compared. If the between-sample standard deviation $s_s$ is smaller than the check value $\sqrt{c}$, then the batch of the PT test items can be regarded as sufficinetly homogeneous. The check value c is calculated as $F_1 \times \sigma_{allow}{}^2 + F_2 \times s_w{}^2$, with $F_1$ and $F_2$ being constants with values of 1.88 and 1.01, respectively, and applying when duplicate samples from 10 bottles are analysed. $\sigma_{allow}{}^2 = 0.3 \times$ FFP-RSD (25 %) × the analytical sampling mean of the analyte, and $s_w$ is the within sample standard deviation.

As all target compounds passed the homogeneity test, the test item was considered sufficiently homogenous and suitable for the EUPT-SRM17.

## 1.10 Storage Stability Test

Within the Specific Protocol, laboratories were recommended storing the samples or analytical protions in the freezer until performing extraction. The stability test samples were thus also stored in the freezer at –20 °C in the period between extraction day 1 and day 3 of the stability test. Shortly after the sample dispatch to the participants, three of the test item bottles that were spared for the homogeneity test were chosen randomly for the conduction of the stability test and extracted immediately. The analytical results of these three bottles (6 results) were thus used for both tests, the homogeneity test and the stability test (here extraction day 1). The three bottles with the remaining material for the extraction days 2 and 3 of

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

| | COMPULSORY COMPOUNDS | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Captan | Chlorothalonil | Cyromazine | Dithiocarbamates (CS2) | Dodine | Emamectin B1a | Folpet | Phthalimide | Pymetrozine | THPI |
| Analytical portion size [g] | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 |
| Mean [mg/kg] | 0.189 | 0.168 | 0.137 | 0.217 | 0.108 | 0.048 | 0.300 | 0.0933 | 0.191 | 0.614 |
| between-samples STD | 0.0 | $3.69\times^{-3}$ | $1.15\times^{-3}$ | $5.57\times^{-3}$ | $1.61\times^{-3}$ | $1.02\times^{-3}$ | 0.0 | $5.67\times^{-3}$ | 0.0 | 0.0 |
| Check Value | $1.40\times^{-2}$ | $1.30\times^{-2}$ | $1.03\times^{-2}$ | $1.60\times^{-2}$ | $8.00\times^{-3}$ | $4.00\times^{-3}$ | $2.30\times^{-2}$ | $7.00\times^{-3}$ | $1.40\times^{-2}$ | $4.60\times^{-2}$ |
| Passed/Failed | passed | passed | passed | passed | passed | passed | passed | passed | passed | passed |

| | OPTIONAL COMPOUNDS | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Bifenazate (sum) | Chloridazon-desphenyl | ETU | Formetanate-HCl | Maleic hydrazide | Oxymatrine | 2,4-DNOP (free phenol) | Meptyldinocap | Meptyldinocap (sum) |
| Analytical portion size [g] | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 |
| Mean [mg/kg] | 0.323 | 0.0533 | 0.0566 | 0.840 | 0.537 | 0.185 | 0.0562 | 0.0956 | 0.165 |
| between-samples STD | 0.0 | 0.0 | $2.07\times^{-3}$ | 0.0 | 0.0 | $3.87\times^{-3}$ | $7.45\times^{-4}$ | $4.01\times^{-4}$ | $1.83\times^{-3}$ |
| Check Value | $2.40\times^{-2}$ | $4.00\times^{-3}$ | $4.00\times^{-3}$ | $6.30\times^{-2}$ | $4.00\times^{-2}$ | $1.40\times^{-2}$ | $4.00\times^{-3}$ | $7.00\times^{-3}$ | $1.20\times^{-2}$ |
| Passed/Failed | passed | passed | passed | passed | passed | passed | passed | passed | passed |

the stability test were placed in the freezer at –20 °C until performing the tests. The methods described in **Section 1.8 (p. 4)** were applied for the analysis of the stability test samples. The extracts of all stability test extractions were stored in the freezer at –20 °C and measured under repeatability conditions within the same measurement sequence, on a day suitable for the laboratory (isochronous approach). The dates on which extractions for each method were carried out are shown below:

Extraction day 1: 02 February 2022 (dithiocarbamates-method)
01 March 2022 (QuEChERS-method)
08 March 2022 (QuPPe-method)

Extraction day 2: 25 February 2022 (dithiocarbamates-method)
14 March 2022 (QuEChERS-method)
15 March 2022 (QuPPe-method)

Extraction day 3: 11 March 2022 (dithiocarbamates-method)
07 April 2022 (QuEChERS-method)
13 April 2022 (QuPPe-method)

A target compound is considered to be sufficiently stable if $|y_i - y| \leq 0.3 \times \sigma_{pt}$, with $y_i$ being the mean value of the last period of the stability test, $y$ the mean value obtained from stability test 1 and $\sigma_{pt}$ the standard

**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 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Captan | Chlorothalonil | Cyromazine | Dithiocarbamates (CS2) | Dodine | Emamectin B1a | Folpet | Phthalimide | Pymetrozine | THPI |
| **Storage at –18 °C (mean values in mg/kg)** | | | | | | | | | | |
| Extraction day 1 | 0.186 | 0.172 | 0.136 | 0.203 | 0.106 | 0.05 | 0.307 | 0.091 | 0.181 | 0.606 |
| Extraction day 2 | 0.173 | 0.159 | 0.128 | 0.212 | 0.099 | 0.052 | 0.287 | 0.086 | 0.174 | 0.598 |
| Extraction day 3 | 0.179 | 0.170 | 0.133 | 0.200 | 0.106 | 0.052 | 0.300 | 0.091 | 0.180 | 0.641 |
| Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1 | 0.0067 (-3.6 %) | 0.0018 (-1.1 %) | 0.0038 (-2.8 %) | 0.0034 (-1.7 %) | 0.000 (0 %) | 0.0018 (3.6 %) | 0.0068 (-2.2 %) | 0.0003 (-0.4 %) | 0.0009 (-0.5 %) | 0.0355 (5.9 %) |
| $0.3 \times \sigma_{pt}$ [mg/kg] | 0.0131 | 0.0113 | 0.0116 | 0.014 | 0.0075 | 0.0035 | 0.0187 | 0.0073 | 0.0113 | 0.0443 |
| Passed/Failed | passed | passed | passed | passed | passed | passed | passed | passed | passed | passed |

| | OPTIONAL COMPOUNDS | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Bifenazate (sum) | Chloridazon-desphenyl | ETU | Formetanate-HCl | Maleic hydrazide | Oxymatrine | 2,4-DNOP (free phenol) | Meptyldinocap | Meptyldinocap (sum) |
| **Storage at –18 °C (mean values in mg/kg)** | | | | | | | | | |
| Extraction day 1 | 0.298 | 0.053 | 0.056 | 0.838 | 0.531 | 0.184 | 0.054 | 0.105 | 0.171 |
| Extraction day 2 | 0.280 | 0.053 | 0.053 | 0.778 | 0.501 | 0.172 | 0.051 | 0.106 | 0.168 |
| Extraction day 3 | 0.310 | 0.054 | 0.056 | 0.814 | 0.533 | 0.175 | 0.053 | 0.100 | 0.165 |
| Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1 | 0.01 (3.7 %) | 0.001 (1.9 %) | 0.0002 (-0.3 %) | 0.0243 (-2.9 %) | 0.0026 (0.5 %) | 0.0088 (-4.8 %) | 0.0015 (-2.8 %) | 0.0047 (-4.4 %) | 0.0062 (-3.6 %) |
| $0.3 \times \sigma_{pt}$ [mg/kg] | 0.02 | 0.0046 | 0.0047 | 0.0655 | 0.0408 | 0.0149 | 0.0042 | 0.0075 | 0.0127 |
| Passed/Failed | passed | passed | passed | passed | passed | passed | passed | passed | passed |

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**). For the compounds passing the test, it can be assumed that the time elapsed between sample receipt by a lab and its analysis has 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. 8)** and **Appendix 4**.

## 1.11 Transport Stability Test

With the exception of three 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 three laboratories having received the material after 7 or 9 days did not imply any significant negative impact of this pro-

longed transport on the analytes reported. Furthermore, the assigned values of all analytes 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. Therefore, the organisers decided to skip the transport stability test in this PT.



## 1.12 Organisational Aspects

### 1.12.1 Laboratory Status: Mandatory and Optional Participation

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

Following DG-SANTE instructions, obliged labs, that were not intending to participate in the EUPT-SRM17, were instructed to provide explanations for their non-participation.

### 1.12.2 Announcement / Invitation and EUPT-SRM17 Website

The EUPT-SRM17 was scheduled to run from 31 January till 1 March, 2022. Within the EURL-Web-Portal an EUPT-SRM17-Website was set up on 30 October, 2021. 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**) were linked to this website. These documents were uploaded both to the EURL-Web-Portal and to the CIRCA BC.

In order to get informed about the capabilities, limitations and wishes of the potential EUPT-SRM17 participants, an exploratory survey was conducted from 11 to 19 November 2021. The OfLs were asked about their interest to participate in this PT as well as about their capability and intention to analyse the compounds on the preliminary TPL released on 5 November, 2021. Considering the information gathered by this survey and after consultation with the EUPT-SC, a revised TPL of the EUPT-SRM17 was released on 26 November and replaced the old version on the PT-Website and on the CIRCA BC.

On 9 November, 2021 the Announcement/Invitation Letter for the EUPT-SRM17 was published on the EUPT-SRM17-Website and sent to all NRL-SRMs and all OfLs analysing pesticide residues in fruits and vegetables. Laboratories involved in the import controls of products listed under Reg. (EU) 1793/2019, as far as they could be tracked in the EURL-DataPool, as well as EU laboratories officially analysing organic samples within the frame of Reg. 889/2008/EC were also informed. The latter laboratories were not considered obliged to participate. NRLs and OfLs from EFTA and EU-candidate countries not entailing fruits and vegetables within the routine scope (according to the DataPool) 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.

### 1.12.3 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 in case of a change of the registration status, the labs received an electronic confirmation about their participation or non-participation in the current PT.

### 1.12.4 Distribution of the Test Items

The PT test items were shipped to the address indicated by the participants on 31 January, 2022. 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 (approx. 350 g test material) embedded in dry ice. Participating laboratories ordering a double amount of the test material received two parcels with one test item each.

On 11 January, detailed instructions on how to treat the EUPT-SRM17 test item upon receipt were provided to the participating laboratories through the Specific Protocol (**Appendix 9**).

### 1.12.5 Webtool for Results Submission and Confidentiality

The "Webtool", an online data submission tool, allows PT-responsibles to acknowledge sample receipt and to submit PT-results and method information via a web browser. It has been used since 2019 for all EUPTs on pesticides residues. Login to the Webtool requires the use of personalized login credentials, which are unique to the registered email address of the PT responsible person. These login credentials are created after a person registrers to the Webtool for the first time and are sent to his/her email address before the Webtool becomes accessible for acknowledgement of sample receipt (typically on the date of sample shipment). Using his/her personal login credentials, the PT-responsible person can access the results submission pages of all EUPTs to which he/she has been designated as PT contact person using the particular e-mail address.

Each laboratory participating in a certain EUPT receives a unique lab code, as soon as one of its PT-responsibles assigned during registration (either the main or the alternative one) logs into the partucular EUPT-site within the WebTool. 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 EUPT-SRM17 participants received their login credentials from DTU on the day of shipment. It was planned to make the Webtool accessible on 1 February, one day after dispatch. Due to unexpected technical difficulties, the Webtool could not be openned on schedule, and all participants were informed of this delay on 1 February. After eliminating those technical problems the Webtool was finally openned on 10 February, which gave the participants enough time to report their results by the original deadline on 1 March 2022. One week after opening the data submission area, one participant reported to the PT organisers that a few data were not correctly saved in the Webtool. Afterwards, a few other laboratories reported the same observation. This phenomenon happed to a few analytes and to certain participants only, so that the organisers appealed to all participants to check the data carefully and to report any irregularity. In parallel, the IT team of the Webtool worked on a solution. In order to reduce any unnecessary error and stress

accompanied with this interruption in the Webtool, the organisers decided on 23 February to postpone the submission deadline to 8 March and informed all participants about it. The problem could be solved by 24 February, and all participants were informed of it via email.

On 10 February, the participants were sent an email informing them about the opening of the Webtool and providing them with a link to a detailed guideline on how to use the Webtool. In this guideline, the participants could find instruction on how to login to the Webtool, how to get the lab code for the EUPT-SRM17, as well as all fields to be filled in. This guideline was also linked on the Webtool for the EUPT-SRM17.

After the submission deadline, participants could check in the Webtool, if they have obtained any tentatively 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.



### 1.12.6 Actions following Results Submission and Preliminary Report

On 8 April, 2022, the preliminary report on the EUPT-SRM17, was released and sent to the participants. This report entailed the prelminary z scores of the compounds present in the PT material, which were calculated based on the preliminary assigned values (prAV). Laboratories having submitted false positive results or having received preliminary | z scores | > 2 were asked to investigate the reasons for this underperformance, and to give a feedback using a specific Excel sheet that was provided by the organisers. At this stage, the organisers overlooked that in the case of ***phthalimide*** the participants' results generated by LC-MS/MS (one result was FN) formed a separate, relatively narrow distributed population (9 numerical results and one FN, $CV^*$ = 27.0 %), with a robust mean of 0.092 mg/kg, which is significantly lower than that of the rest of the population at 0.140 mg/kg (69 numerical results and 4 FNs, $CV^*$ = 35.2 %) or the total population at 0.134 mg/kg (78 nummerical results, $CV^*$ = 36.8 %). Based on the prAV, those LC-MS/MS results of the participants received negative z scores. In addition, the ***phthalimide*** results generated by the organisers during the homogeneity test using LC-MS/MS (mean = 0.093 mg/kg) also fitted well into the LC-MS/MS population of the participants. The GC-results formed the overwhelming majority of the total population of ***phthalimide*** results (73 out of 83), and were broadly distributed themselves, which partly masked the existing bimodality. Despite the bimodality, which was actually slightly visible as a shoulder in the kernel density histogram, the uncertainty of the prAV of the overall population was within the limits and the robust mean of the total population was therefore used in the preliminary report. After looking at the methodology data more closely and identifying that the LC-based results form a well defined and significantly shifted population. The organisers decided to inform the participants immediately in order to avoid that laboratories having performed well start searching for the reasons of a non-existing underperformance and to make sure that laboratories actually having obtained overestimated results (using GC) will be aware of this. In fact, the risk of obtaining overestimated results of ***THPI*** and ***phthalimide*** in the presence of excess amounts of the respective parent compounds (***Captan*** and ***Folpet***) has been explained extensively in an analytical observation report by the EURL-SRM in 2017 (SRM-07 (GC)). Based on this information, the EURL-SRM decided to proceed with a second evaluation of the participans' results, based on a second preliminary assigned value derived from the LC-results population. The revised preliminary z scores for ***phthalimide*** were released on 9 June in a second version of the preliminary report. In the case of ***THPI***, it was considered that there was no need to calculate new z scores as the parent captan was present at a lower concentration than ***THPI***, resulting in a negligible potential of ***THPI*** overestimation using GC. All participants were informed of this change. For details please refer to **Section 4.2.1 (p. 28).**

During the EUPT-Sceintific Committee Meeting in June 2022, it was decided that in the EUPT-SRM17 Final Report any z scores for ***phthalimide*** should be shown for information only. It was decided to make calculations based on both the robust mean of the entire population as well the robust mean of the population of

results generated laboratories applying LC-methods (**Section 4.2.1, p. 28**). In addition, it was decided to additionally show calculations for the summed concentrations of ***folpet/phthalimide*** expressed as ***folpet*** and of ***captan/THPI***, expressed as ***captan***.

# 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 organisers, 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 organisers 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 present in the test item and determined, at or above the MRRL, by the organisers and the overwhelming majority of the participating laboratories. In accordance with the EUPT General Protocol 9th Ed. 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 the MRRL. Any RLs that are higher than the MRRL are not taken into account. Following the General Protocol, results reported as "< RL" without providing a numerical value are also judged as false negatives if the RL exceeds the MRRL. In cases of the assigned value (see **Section 2.2)** being less than a factor of 3 times the MRRL, false negatives will typically not be assigned.

## 2.2 Assigned Values ($x_{pt}$) and Calculation of the Respective Uncertainties ($u(x_{pt})$)

In accordance with EUPT General Protocol (**Appendix 8**), the assigned value ($x_{pt}$) of each compound in the PT-material is established using the mean value of robust statistics ($x^*$; calculated using using Algorithm A of ISO 13528:2015 [6]) derived from results generated by OfLs (incl. NRLs) from EU and EFTA countries. Since the assigned values of the analytes are generated using the respective robust mean values of the participants' results and since these results are generated by a variety of analytical methods and standards, the assigned values are not metrologically traceable. 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 values, the z scores of the participants' results were calculated applying the formula shown in **Section 2.4**. All results assigned with z scores > 5 based on the *initially* calculated assigned value, were regarded as outlier-candidates. If these oulier candidates were confirmed by Grubbs' test as outliers, they were excluded from the results population for the establishment of the assigned value, and the assigned value of the corresponding analyte was calculated again without those results.

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

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

## 2.4 z Scores

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

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

Where

- $x_i$ is the result for the target analyte ($i$) as reported by the participant;
  (In case of 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).

  In case that the MRRL is higher than 25 % of the assigned value and therefore the calculated z score of the FN is higher than -3 and reaches the classification "questionable", the z score will be set at -3. Any z scores > 5 are set at 5 in calculations of combined z scores (see **2.5.1**).

The z scores are classified as follows:

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

### 2.4.1 z' Scores and Tentative Range of z Scores

In accordance with ISO 13528:2015 "Statistical methods for use in proficiency testing by interlaboratory comparison" **[6]**, for one analyte in the present PT (***ETU***), where the uncertainty of assigned value of the concerned analyte was slightly higher than the tolerance of the assigned value, the organisers calculated also the z' score. Z' score is a common variation on the z score by combining the uncertainty of the assigned value with the standard deviation for proficiency and calculated as follows, applying the uncertainty of assigned value for the benefit of the participant:

$$z_i' = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u^2\left(x_{pt}\right)}}$$

In parallel, the uncertainty of the assigned value ($u(x_{pt})$) was applied to the assigned value to determine the lower- and upper-bound limits of the assigned value: $x_{pt} - u(x_{pt})$ *to* $x_{pt} + u(x_{pt})$, based on the lower- and upper-bound assigned value, the upper and lower-bound limits of the z scores were calculated as follows:

$$z_1 = \frac{\left(x_i - x_{pt}\right)}{FPP\text{-}\sigma_{pt}} - \frac{u\left(x_{pt}\right)}{FPP\text{-}\sigma_{pt}} \quad \text{and} \quad z_2 = \frac{\left(x_i - x_{pt}\right)}{FPP\text{-}\sigma_{pt}} + \frac{u\left(x_{pt}\right)}{FPP\text{-}\sigma_{pt}}$$

This approach shows the best and worst z scores obtained by a lab considering the uncertainty of the assigned value, and is particularly interresting in cases where an analyte marginally fails to meet the uncertainty threshold of the assigned value (e. g. due to the small number of results). In such cases, z scores would normally not be calculated, that means that laboratories performing well would not be able to demonstrate good proficiency. By calculating the z score range of each laboratory, such labs can show that even in a worst-case scenario their z score for the particular analyte is well within the acceptable range.

## 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 for compulsory compounds.

### 2.5.1 Combined z Scores

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

#### Average of the Absolute z Scores (AAZ)

The AAZ is calculated using the following formula:

$$AAZ = \frac{\sum_{i=1}^{n} |z_i|}{n}$$

where "$n$" is the number of each laboratory's z scores that are considered in this formula, including z scores assigned for false negative results.
For the calculation, any z score > 5 is set at 5.

# 3. PARTICIPATION

123 official laboratories (including NRLs) from 33 countries (26 EU-Member States, 2 EFTA-countries, 1 EU candidate country and 3 countries outside Europe) registered for participation in the EUPT-SRM17 and submitted at least one result. An overview of the participating laboratories and countries is given in **Table 3-1**. All laboratories having participated in this EUPT are given in the list in **Appendix 1**.

In total, 160 EU-OfLs, including NRL-SRMs, regardless of their commodity scope, as well as all EU-OfLs analysing for pesticide residues in fruits and vegetables, 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 within the same website. Other EU-OfLs wihtout obligation for participation were also invited to participate in the current PT on voluntary basis.

23 obliged laboratories provided reasons for their non-participation, the most frequently stated reason was that the EUPT-SRM17 target pesticides were generally out of their routine scope. Two other obliged OfLs initially registered for the participation, but finally withdrew for the same reason. Excluding those 25 laboratories that provided sufficient explanations, the number of EU-laboratories considered as being obliged decreased to 135, among which 102 participated and submitted results. Furthermore, 17 EU-OfLs registered for participation on voluntary basis with 16 of them submitting results and the remaining one neither submitting results nor providing any explanations this regarding.

35 out of the 160 obliged OfLs (21 %) did neither register for the PT nor provide any explanation for non-participation. These laboratories originated from 8 countries as follows: ES (14×), IT (8×), HR (3×), FR (2×), PT(2×), as well as one each from BG, DK, PL, RO, and SI.

**Table 3-1:** Number of laboratories listed as being obliged to participate in the EUPT-SRM17, labs that registered to participate in the PT and submitted results (grouped by contracting country)

| EU: NRLs and OfLs | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | | | | Registered for Participation and submitting results obliged + [on voluntary basis] | | |
| Contracting Country [1)] | Labs originally considered to be obliged (based on scope and NRL-function) | Labs providing explanations for non-participation | Obliged labs non participating w/o explanations | Finally considered to be obliged | All | NRL-SRMs | Notes |
| **AT** | 1 | 0 | 0 | 1 | 1 | 1 | |
| **BE** | 5 | 0 | 0 | 5 | 5 | 1 | |
| **BE, LU, FR** | 1 | 0 | 0 | 1 | 1 | | |
| **BE, NL** | 1 | 0 | 0 | 1 | 1 | | |
| **BG** | 3 | 0 | 1 | 3 | 2 | 1 | |
| **CY** | 1 | 0 | 0 | 1 | 1 | 1 | |
| **CZ** | 3 | 0 | 1 | 3 | 2 + [1] | 1 | |
| **DE** | 20 | 4 | 0 | 16 | 16 + [6] | 1 | |
| **DK** | 2 | 0 | 1 | 2 | 1 | 1 | |
| **EE** | 2 | 0 | 0 | 2 | 2 | 1 | |
| **ES** | 36 | 4 | 15 | 32 | 16 | 2 | |
| **FI** | 3 | 0 | 0 | 3 | 3 | 2 | |
| **FR** | 10 | 1 | 1 | 9 | 8 + [1] | 1 | |
| **GR** | 3 | 1 | 0 | 2 | 2 | 2 | |
| **HR** | 9 | 1 | 3 | 8 | 5 | 1 | |
| **HU** | 4 | 0 | 0 | 4 | 4 | 1 | |
| **IE** | 1 | 0 | 0 | 1 | 1 | 1 | |
| **IT** | 25 | 8 | 8 | 17 | 9 | 1 | |
| **LT** | 1 | 0 | 0 | 1 | 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 Germany were appointed as OfL for monitoring activities |
| **NL** | 1 | 0 | 0 | 1 | 1 | 1 | |
| **PL** | 11 | 0 | 3 | 11 | 8 + [4] | 1 | |
| **PT** | 1 | 0 | 0 | 1 | 2 | 1 | |
| **RO** | 7 | 4 | 1 | 3 | 2 | 1 | |
| **SE** | 2 | 0 | 0 | 2 | 2 | 1 | |
| **SI** | 2 | 1 | 0 | 1 | 1 | 1 | |
| **SK** | 1 | 0 | 0 | 1 | 1 | 1 | |
| **EU Total** | 160 | 25 | 34 | 135 | **101 + [13]** | **26** | |
| | | | | | | | |
| **EFTA: NRLs and OfLs** | | | | | | | |
| **CH** | | | | | [3] | | |
| **NO** | 1 | 0 | 0 | 1 | 1 | 1 | NO-NRL regarded as obliged lab due to data submission to EFSA |
| **EU/EFTA OfLs Total** | 161 | 25 | 34 | 136 | **102 + [16]** | **27** | |
| | | | | | | | |
| **Countries outside Europa** | | | | | | | |
| **KR** | | | | | 1 | | |
| **RS** | | | | | 2 | | |
| **TH** | | | | | 1 | | |
| **UK** | | | | | 1 | | |
| **Countries outside Europa Total** | | | | | **5** | | |

# 4. RESULTS

## 4.1 Overview of Results

An overview of the percentage of laboratories having targeted each of the analytes present in the Target Pesticides List is shown in **Table 4-1**. **Table 4-2 (p. 20)** gives an overview of all results submitted by each laboratory. The individual numerical results reported by the laboratories are shown in **Table 4-8 (p. 36)**

**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 analysed for the compunds | | | | |
|---|---|---|---|---|---|---|---|
| | | | Obliged OfLs only | | | Incl. OfLs on Voluntary Basis | |
| | | | No. [1] | Based on n = 102 [2] | Based on n = 135 [3] | No. [1] | Based on n = 118 [2] |
| Compulsory Compounds | **Avermectin B1a** | No | 78 | 76 % | 58 % | 92 | 78 % |
| | **Captan** | **Yes** | 76 | 75 % | 56 % | 90 | 76 % |
| | **Chlormequat (chloride)** | No | 81 | 79 % | 60 % | 94 | 80 % |
| | **Chlorothalonil** | **Yes** | 89 | 87 % | 66 % | 103 | 87 % |
| | **Cyromazine** | **Yes** | 80 | 78 % | 59 % | 92 | 78 % |
| | **Dithianon** | No | 66 | 65 % | 49 % | 78 | 66 % |
| | **Dithiocarbamates (expr. as $CS_2$)** | **Yes** | 83 | 81 % | 61 % | 99 | 84 % |
| | **Dodine** | **Yes** | 82 | 80 % | 61 % | 96 | 81 % |
| | **Emamectin B1a** | **Yes** | 77 | 75 % | 57 % | 91 | 77 % |
| | **Fenbutatin Oxide** | No | 72 | 71 % | 53 % | 82 | 69 % |
| | **Folpet** | **Yes** | 76 | 75 % | 56 % | 90 | 76 % |
| | **Mepiquat (chloride)** | No | 81 | 79 % | 60 % | 93 | 79 % |
| | **Phthalimide** | **Yes** | 71 | 70 % | 53 % | 83 | 70 % |
| | **Pymetrozine** | **Yes** | 88 | 86 % | 65 % | 101 | 86 % |
| | **TFNA** | No | 65 | 64 % | 48 % | 77 | 65 % |
| | **TFNG** | No | 65 | 64 % | 48 % | 77 | 65 % |
| | **THPI** | **Yes** | 68 | 67 % | 50 % | 80 | 68 % |
| Optional Compounds | **BAC-C12 (chloride)** | No | 52 | 51 % | 39 % | 63 | 53 % |
| | **Bifenazate (sum)** | **Yes** | 49 | 48 % | 36 % | 59 | 50 % |
| | **Chloridazon-desphenyl** | **Yes** | 26 | 25 % | 19 % | 32 | 27 % |
| | **DDAC-C10 (chloride)** | No | 52 | 51 % | 39 % | 63 | 53 % |
| | **Diquat (dication)** | No | 35 | 34 % | 26 % | 42 | 36 % |
| | **ETU** | **Yes** | 14 | 14 % | 10 % | 19 | 16 % |
| | **Formetanate-HCl** | **Yes** | 70 | 69 % | 52 % | 81 | 69 % |
| | **Maleic hydrazide** | **Yes** | 38 | 37 % | 28 % | 47 | 40 % |
| | **Matrine** | No | 39 | 38 % | 29 % | 49 | 42 % |
| | **Meptyldinocap** | **Yes** | 24 | 24 % | 18 % | 28 | 24 % |
| | **2,4-DNOP (free phenol)** | **Yes** | 11 | 11 % | 8 % | 15 | 13 % |
| | **Meptyldinocap (sum)** | **Yes** | 11 | 11 % | 8 % | 16 | 14 % |
| | **Nicotine** | No | 37 | 36 % | 27 % | 47 | 40 % |
| | **Oxymatrine** | **Yes** | 27 | 26 % | 20 % | 36 | 31 % |
| | **Paraquat (dication)** | No | 33 | 32 % | 24 % | 40 | 34 % |
| | **PTU** | No | 14 | 14 % | 10 % | 20 | 17 % |
| | **Trimesium (cation)** | No | 23 | 23 % | 17 % | 30 | 25 % |

1) Laboratories representing more than one country were counted only once.
2) 118 OfLs from EU and EFTA countries (incl. NRLs) have submitted at least one result, among them 102 laboratories were obliged to participate in this PT and 16 participated on voluntary basis.
3) 135 OfLs (including NRLs) from EU and EFTA countries were finally considered as obliged to participate in the EUPT-SRM17 (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 | | | Avermectin B1a | Captan | Chlormequat (chloride) | Chlorothalonil | Cyromazine | Dithianon | Dithiocarbamates (expr. as $CS_2$) | Dodine | Emamectin B1a | Fenbutatin Oxide | Folpet | Mepiquat (chloride) | Phthalimide | Pymetrozine | TFNA | TFNG | THPI | analysed / correctly found (Compulsory Compounds, max. 17/10) |
| within MACP/WD | | | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | |
| present in test item | | | No | **Yes** | No | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | **Yes** | **Yes** | No | No | **Yes** | |
| evaluated in this PT | | | No | **Yes** | No | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | (**Yes***) | **Yes** | No | No | **Yes** | |
| Lab-Code SRM17- | NRL | Cat. | | | | | | | | | | | | | | | | | | |
| 3 | x | B | | FN | ND | V | V | | V | V | V | | V | ND | | V | ND | ND | | 12 / 7 |
| 4 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 5 | x | B | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | | V | ND | ND | | 15 / 8 |
| 6 | | B | | | ND | V | | | | | | | | ND | | | | | | 3 / 1 |
| 7 | x | B | ND | | ND | V | V | | V | | V | ND | | ND | | V | | | | 9 / 5 |
| 8 | | B | | V | ND | V | V | | | V | | | V | ND | | V | | ND | | 9 / 6 |
| 9 | | B | ND | V | | V | | | V | V | V | | V | | | | ND | ND | | 9 / 6 |
| 10 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 11 | | A | ND | V | ND | V | V | ND | V | V | V | | V | ND | V | V | ND | ND | V | 16 / 10 |
| 12 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | | 16 / 9 |
| 13 | | B | ND | FN | ND | V | V | ND | V | V | V | ND | FN | ND | V | V | ND | ND | V | 17 / 8 |
| 15 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 16 | | B | ND | V | ND | V | | ND | V | | V | ND | V | ND | V | V | | | V | 13 / 8 |
| 17 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | | | V | 15 / 10 |
| 18 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 19 | x | B | ND | | | | | | | V | V | ND | | ND | | | | | | 5 / 2 |
| 20 | | A | ND | V | ND | V | V | ND | V | V | V | | V | ND | FN | V | ND | ND | V | 16 / 9 |
| 21 | | B | ND | V | ND | V | | ND | V | V | V | ND | V | ND | | V | | | | 12 / 7 |
| 22 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 23 | | B | | | | | | | V | | | | | | | | | | | 1 / 1 |
| 24 | | B | | V | | V | | | V | | | | V | | V | | | | V | 6 / 6 |
| 25 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 26 | | A | ND | V | ND | V | V | ND | V | | V | ND | V | ND | V | V | ND | ND | V | 16 / 9 |
| 28 | x | A | ND | V | ND | V | V | | | V | V | ND | V | ND | V | V | ND | ND | V | 15 / 9 |
| 29 | | B | ND | | | | | | | V | V | | | | | V | | | | 4 / 3 |
| 30 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 31 | | B | | | | V | | | | | | | | | | | | | | 1 / 1 |
| 32 | | B | | | ND | | | | V | | | | | ND | | V | | | | 4 / 2 |
| 33 | | B | | | ND | | | | | V | | ND | | | | V | | | | 4 / 2 |
| 34 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 35 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 36 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |

**MACP-Reg**. (covering 2022-2024): REGULATION (EU) 2021/601 of 13 April 2021; **MACP****: inclusion of MH in the MACP was decided at technical level in 2021; **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)

(**Yes***): evaluation for information only

**V** = analysed for and submitted concentration Value for a pesticide present in the test item at a level allowing statistical evaluations; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RLs > assigned values; **FP** = false positive result

**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 | | | BAC-C12 (chloride) | Bifenazate (sum) | Chloridazon-desphenyl | DDAC-C10 (chloride) | Diquat (dication) | ETU | Formetanate-HCl | Maleic hydrazide | Matrine | Meptyldinocap | 2,4-DNOP (free phenol) | Meptyldinocap (sum) | Nicotine | Oxymatrine | Paraquat (dication) | PTU | Trimesium (cation) | analysed / correctly found (Optional Compounds, max. 17/7) | analysed / correctly found (Total, max. 34/17) |
| within MACP/WD | | | WD | WD | WD | WD | WD | None | MACP | MACP** | WD | WD | WD | WD | WD | WD (2021) | WD | None | WD | | |
| present in test item | | | No | **Yes** | **Yes** | No | No | **Yes** | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | No | No | | |
| evaluated in this PT | | | No | **Yes** | **Yes** | No | No | (**Yes***) | **Yes** | **Yes** | No | No | No | No | No | **Yes** | No | No | No | | |
| Lab-Code SRM17- | NRL | Cat. | | | | | | | | | | | | | | | | | | | |
| 3 | x | B | ND | | FN | ND | ND | | | V | | | | | | | ND | | | 6 / 1 | 18 / 8 |
| 4 | x | A | ND | V | | ND | | | FN | | | V | | | | | | | | 5 / 2 | 22 / 12 |
| 5 | x | B | ND | V | | ND | | | V | V | ND | | | | ND | | | | ND | 8 / 3 | 23 / 11 |
| 6 | | B | ND | | | ND | | | V | | | | | | ND | | | | | 4 / 1 | 7 / 2 |
| 7 | x | B | | | | | | | V | | | | | | ND | | | | | 2 / 1 | 11 / 6 |
| 8 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 9 / 6 |
| 9 | | B | ND | | | ND | | | V | V | | | | | | | | | | 4 / 2 | 13 / 8 |
| 10 | | A | ND | V | V | ND | ND | V | V | V | ND | FN | FN | V | ND | V | ND | ND | ND | 17 / 7 | 34 / 17 |
| 11 | | A | | | V | | ND | | V | | | | | V | | | ND | | | 5 / 3 | 21 / 13 |
| 12 | x | A | ND | V | | ND | ND | FN | FN | V | | V | | | | | ND | | ND | 10 / 3 | 26 / 12 |
| 13 | | B | ND | V | V | ND | ND | | V | | ND | V | | | | V | ND | | | 10 / 5 | 27 / 13 |
| 15 | | A | ND | V | V | ND | | V | V | | ND | | | | ND | V | | ND | ND | 11 / 5 | 28 / 15 |
| 16 | | B | | V | | | ND | | V | V | ND | | | | | | ND | | | 6 / 3 | 19 / 11 |
| 17 | x | A | | | | | | | V | | | | | | | | | | | 1 / 1 | 16 / 11 |
| 18 | x | A | | | | | | | V | | | | | | ND | | | | ND | 3 / 1 | 20 / 11 |
| 19 | x | B | | V | | | | | | | | | | | | | | | | 1 / 1 | 6 / 3 |
| 20 | | A | | | V | | | | | | | | | | | | | | | 1 / 1 | 17 / 10 |
| 21 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 12 / 7 |
| 22 | | A | ND | V | | ND | ND | | V | V | | V | V | V | | | ND | | | 10 / 6 | 27 / 16 |
| 23 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 1 / 1 |
| 24 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 6 / 6 |
| 25 | | A | ND | V | V | ND | ND | | V | V | ND | | V | | ND | V | ND | | ND | 13 / 6 | 30 / 16 |
| 26 | | A | | | | | | | | | ND | | | | | | | | | 1 / 0 | 17 / 9 |
| 28 | x | A | | | | | | | V | | | | | | | | | | | 1 / 1 | 16 / 10 |
| 29 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 4 / 3 |
| 30 | | A | ND | V | FN | ND | ND | V | V | V | ND | FN | V | V | ND | V | ND | ND | ND | 17 / 7 | 34 / 17 |
| 31 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 1 / 1 |
| 32 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 4 / 2 |
| 33 | | B | ND | | | ND | | | | | | | | | | | | | | 2 / 0 | 6 / 2 |
| 34 | | A | ND | | V | ND | ND | | V | V | ND | V | V | V | | V | | | | 11 / 7 | 28 / 17 |
| 35 | x | A | ND | | | ND | | | V | | ND | | | | | | | | ND | 5 / 1 | 22 / 11 |
| 36 | x | A | | | | | | | V | | | | | | | | | | | 1 / 1 | 18 / 11 |

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(**Yes***): evaluation for information only

**V** = analysed for and submitted concentration Value for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RLs > assigned values; **FP** = false positive result

**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 | | | Avermectin B1a | Captan | Chlormequat (chloride) | Chlorothalonil | Cyromazine | Dithianon | Dithiocarbamates (expr. as $CS_2$) | Dodine | Emamectin B1a | Fenbutatin Oxide | Folpet | Mepiquat (chloride) | Phthalimide | Pymetrozine | TFNA | TFNG | THPI | analysed / correctly found (Compulsory Compounds, max. 17/10) |
| within MACP/WD | | | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | |
| present in test item | | | No | **Yes** | No | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | **Yes** | **Yes** | No | No | **Yes** | |
| evaluated in this PT | | | No | **Yes** | No | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | (**Yes***) | **Yes** | No | No | **Yes** | |
| Lab-Code SRM17- | NRL | Cat. | | | | | | | | | | | | | | | | | | |
| 37 | | A | ND | V | ND | V | V | ND | FN | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 9 |
| 38 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 39 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 40 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 41 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 42 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 43 | x | A | ND | V | ND | V | V | ND | V | V | | ND | V | ND | V | V | ND | ND | V | 16 / 9 |
| 44 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | FN | V | ND | ND | V | 17 / 9 |
| 45 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 46 | | B | | | | | V | | | V | | ND | | | | V | | | | 4 / 3 |
| 47 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 48 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 49 | x | B | ND | V | ND | V | | | V | | | | V | ND | | FN | | | | 8 / 4 |
| 50 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 51 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 52 | | B | | | | | V | | V | V | | ND | | | | V | | | | 5 / 4 |
| 53 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 54 | | A | ND | V | ND | V | V | | V | V | V | ND | V | ND | V | V | ND | ND | V | 16 / 10 |
| 55 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | | 16 / 9 |
| 56 | x | B | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | | | | 14 / 9 |
| 57 | | A | ND | | ND | V | V | | V | V | V | ND | V | ND | V | V | ND | ND | V | 15 / 9 |
| 58 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 59 | | B | | | | | | | V | | | | | | | | | | | 1 / 1 |
| 60 | x | B | | | ND | V | V | ND | V | V | V | | | ND | V | V | ND | ND | | 12 / 7 |
| 61 | | A | ND | V | ND | FN | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 9 |
| 62 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 63 | x | B | | | ND | V | | | | | | | | ND | | | ND | ND | | 5 / 1 |
| 64 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 66 | | B | ND | FN* | ND | V | | | V | V | | ND | | ND | | V | | | | 9 / 4 |
| 67 | | A | ND | V | ND | V | V | | V | V | V | ND | V | ND | V | V | ND | ND | V | 16 / 10 |
| 68 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 69 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |

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(**Yes***): evaluation for information only

**V** = analysed for and submitted concentration Value for a pesticide present in the test item at a level allowing statistical evaluations; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RLs > assigned values; **FP** = false positive result

**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 | | | BAC-C12 (chloride) | Bifenazate (sum) | Chloridazon-desphenyl | DDAC-C10 (chloride) | Diquat (dication) | ETU | Formetanate-HCl | Maleic hydrazide | Matrine | Meptyldinocap | 2,4-DNOP (free phenol) | Meptyldinocap (sum) | Nicotine | Oxymatrine | Paraquat (dication) | PTU | Trimesium (cation) | analysed / correctly found (Optional Compounds, max. 17/7) | analysed / correctly found (Total, max. 34/17) |
| within MACP/WD | | | WD | WD | WD | WD | WD | None | MACP | MACP** | WD | WD | WD | WD | WD | WD (2021) | WD | None | WD | | |
| present in test item | | | No | **Yes** | **Yes** | No | No | **Yes** | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | No | No | | |
| evaluated in this PT | | | No | **Yes** | **Yes** | No | No | (**Yes***) | **Yes** | **Yes** | No | No | No | No | No | **Yes** | No | No | No | | |
| Lab-Code SRM17- | NRL | Cat. | | | | | | | | | | | | | | | | | | | |
| 37 | | A | ND | V | | | ND | | | | ND | | | | ND | | ND | ND | | 7 / 1 | 24 / 10 |
| 38 | | A | ND | V | V | ND | ND | V | V | V | ND | | V | V | ND | V | ND | ND | ND | 16 / 8 | 33 / 18 |
| 39 | | A | ND | V | | ND | | | V | V | ND | | | | ND | | | | | 7 / 3 | 24 / 13 |
| 40 | | A | ND | V | | ND | ND | | V | V | ND | V | | | | V | ND | | | 10 / 5 | 27 / 15 |
| 41 | | A | ND | V | V | ND | | | V | V | ND | | | | ND | V | | | | 9 / 5 | 26 / 15 |
| 42 | | A | ND | V | V | ND | ND | V | V | V | ND | V | | | ND | V | ND | ND | ND | 15 / 7 | 32 / 17 |
| 43 | x | A | | | | | ND | | | V | | | | | | | ND | | | 3 / 1 | 19 / 10 |
| 44 | | A | ND | V | V | ND | | | V | V | ND | V | V | V | | V | | | | 11 / 8 | 28 / 17 |
| 45 | | A | ND | V | FN | ND | ND | V | V | V | ND | V | | | ND | V | ND | ND | | 14 / 6 | 31 / 16 |
| 46 | | B | | | | | | | V | | | | | | | | | | | 1 / 1 | 5 / 4 |
| 47 | | A | ND | V | V | ND | ND | V | V | V | ND | | V | V | ND | V | ND | ND | ND | 16 / 8 | 33 / 18 |
| 48 | | A | ND | V | V | ND | ND | | V | V | ND | | | | ND | V | ND | | ND | 12 / 5 | 29 / 15 |
| 49 | x | B | | | | | | | | | | | | | | | | | | 0 / 0 | 8 / 4 |
| 50 | x | A | ND | V | V | ND | ND | V | V | V | ND | FN | | V | ND | V | ND | ND | ND | 16 / 7 | 33 / 17 |
| 51 | | A | ND | | V | ND | ND | V | V | | ND | | | | ND | V | ND | ND | ND | 12 / 4 | 29 / 14 |
| 52 | | B | ND | | V | ND | | | V | | | | | | | | | | | 4 / 2 | 9 / 6 |
| 53 | | A | ND | V | | ND | | | V | V | ND | | | | ND | V | | | ND | 9 / 4 | 26 / 14 |
| 54 | | A | | V | | | | | V | V | ND | V | | | ND | V | | | ND | 8 / 5 | 24 / 15 |
| 55 | x | A | | V | | | ND | | V | V | | | | | | | ND | | | 5 / 3 | 21 / 12 |
| 56 | x | B | | | | | ND | | V | | | | | | | | ND | | | 3 / 1 | 17 / 10 |
| 57 | | A | ND | V | | ND | | V | V | | ND | V | | | | | | ND | | 8 / 4 | 23 / 13 |
| 58 | x | A | | V | V | | | | V | V | ND | | | | | | | | | 5 / 4 | 22 / 14 |
| 59 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 1 / 1 |
| 60 | x | B | | V | | | | | V | | | | | | | | | | | 2 / 2 | 14 / 9 |
| 61 | | A | ND | | | ND | ND | | V | | ND | | | | ND | V | ND | | | 8 / 2 | 25 / 11 |
| 62 | | A | ND | V | V | ND | ND | V | V | V | ND | V | V | V | ND | V | ND | ND | ND | 17 / 9 | 34 / 19 |
| 63 | x | B | | | | | | | V | | | | | | | | | | | 1 / 1 | 6 / 2 |
| 64 | x | A | | | | | ND | | V | | | | | | | | | | ND | 3 / 1 | 20 / 11 |
| 66 | | B | | | | | | | V | | | | | | | | | | | 1 / 1 | 10 / 5 |
| 67 | | A | ND | | | ND | | V | | V | ND | | | | ND | V | | ND | ND | 9 / 3 | 25 / 13 |
| 68 | x | A | | V | | | | | | V | | | | | ND | | | | | 3 / 2 | 20 / 12 |
| 69 | | A | ND | V | V | ND | ND | | V | V | ND | V | V | V | ND | V | ND | | ND | 15 / 8 | 32 / 18 |

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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 | | | Avermectin B1a | Captan | Chlormequat (chloride) | Chlorothalonil | Cyromazine | Dithianon | Dithiocarbamates (expr. as $CS_2$) | Dodine | Emamectin B1a | Fenbutatin Oxide | Folpet | Mepiquat (chloride) | Phthalimide | Pymetrozine | TFNA | TFNG | THPI | analysed / correctly found (Compulsory Compounds, max. 17/10) |
| within MACP/WD | | | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | |
| present in test item | | | No | **Yes** | No | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | **Yes** | **Yes** | No | No | **Yes** | |
| evaluated in this PT | | | No | **Yes** | No | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | (**Yes***) | **Yes** | No | No | **Yes** | |
| Lab-Code SRM17- | NRL | Cat. | | | | | | | | | | | | | | | | | | |
| 70 | | A | ND | V | ND | V | V | ND | | V | V | ND | V | ND | V | V | ND | ND | V | 16 / 9 |
| 71 | | B | | V | ND | V | | ND | V | | V | ND | V | ND | FN | | | | V | 11 / 6 |
| 72 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 73 | | B | ND | FN | ND | V | V | ND | V | V | V | ND | V | ND | | V | | | V | 14 / 8 |
| 74 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 75 | | B | | | ND | | V | | V | V | | | | ND | | V | | | | 6 / 4 |
| 76 | | B | ND | V | | V | V | ND | | V | V | | V | | V | V | | | V | 11 / 9 |
| 77 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 78 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 79 | | B | | V | | | | | | V | | | V | | V | | | | V | 5 / 5 |
| 80 | | B | | | | | | | V | | | | | | | | | | | 1 / 1 |
| 81 | | B | | | | | | | V | | | | | | | | | | | 1 / 1 |
| 82 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 84 | x | B | ND | | ND | V | V | | | V | | | | ND | | | | | | 6 / 3 |
| 85 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 86 | | B | ND | V | | V | | | V | | V | | V | | | V | ND | ND | | 9 / 6 |
| 87 | | B | | | ND | | V | | V | | | | | ND | | | | | | 4 / 2 |
| 88 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 89 | | A | ND | V | ND | V | V | ND | V | V | V | | V | ND | V | V | ND | ND | V | 16 / 10 |
| 90 | | B | ND | V | ND | V | V | | | V | V | | V | ND | V | V | ND | ND | V | 14 / 9 |
| 91 | | A | ND | V | ND | V | V | ND | V | V | V | | V | ND | V | V | ND | ND | V | 16 / 10 |
| 92 | x | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | FN | V | ND | ND | V | 17 / 9 |
| 93 | | A | | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 16 / 10 |
| 94 | | B | ND | V | ND | V | | ND | V | V | V | ND | | ND | V | V | | | V | 13 / 8 |
| 95 | | B | ND | | ND | V | V | ND | V | V | V | ND | | ND | | V | ND | ND | V | 14 / 7 |
| 96 | x | B | ND | V | ND | V | | | | | | | V | ND | V | V | ND | ND | V | 11 / 6 |
| 97 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | | V | 16 / 10 |
| 98 | x | B | ND | | ND | V | V | ND | V | V | V | ND | | ND | | V | | | | 11 / 6 |
| 99 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 100 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 101 | x | B | ND | | | V | V | | | V | V | ND | | | | V | | | | 7 / 5 |
| 102 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 104 | x | B | ND | V | ND | V | V | ND | | V | V | ND | V | ND | FN | V | ND | ND | V | 16 / 8 |

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(**Yes***): evaluation for information only

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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 | | | BAC-C12 (chloride) | Bifenazate (sum) | Chloridazon-desphenyl | DDAC-C10 (chloride) | Diquat (dication) | ETU | Formetanate-HCl | Maleic hydrazide | Matrine | Meptyldinocap | 2,4-DNOP (free phenol) | Meptyldinocap (sum) | Nicotine | Oxymatrine | Paraquat (dication) | PTU | Trimesium (cation) | analysed / correctly found (Optional Compounds, max. 17/7) | analysed / correctly found (Total, max. 34/17) |
| within MACP/WD | | | WD | WD | WD | WD | WD | None | MACP | MACP** | WD | WD | WD | WD | WD | WD (2021) | WD | None | WD | | |
| present in test item | | | No | **Yes** | **Yes** | No | No | **Yes** | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | No | No | | |
| evaluated in this PT | | | No | **Yes** | **Yes** | No | No | (**Yes***) | **Yes** | **Yes** | No | No | No | No | No | **Yes** | No | No | No | | |
| Lab-Code SRM17- | NRL | Cat. | | | | | | | | | | | | | | | | | | | |
| 70 | | A | ND | V | | ND | | | V | V | | | | | ND | | | | | 6 / 3 | 22 / 12 |
| 71 | | B | ND | | | ND | | | FN | | | V | | | | | | | | 4 / 1 | 15 / 7 |
| 72 | x | A | ND | V | | ND | ND | | V | | | | | | ND | | ND | | | 7 / 2 | 24 / 12 |
| 73 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 14 / 8 |
| 74 | | A | ND | V | | ND | ND | | V | V | ND | V | | | ND | V | FP | | ND | 12 / 5 | 29 / 15 |
| 75 | | B | FP | | | FP | | | V | V | | | | | | | | | | 4 / 2 | 10 / 6 |
| 76 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 11 / 9 |
| 77 | | A | | | | | | | V | | | | | | | | | | | 1 / 1 | 18 / 11 |
| 78 | x | A | ND | | | ND | | | V | | | | | V | | | | | | 4 / 2 | 21 / 12 |
| 79 | | B | | | | ND | | | V | | | | | | | | | | | 2 / 1 | 7 / 6 |
| 80 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 1 / 1 |
| 81 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 1 / 1 |
| 82 | | A | ND | V | V | ND | | | V | V | ND | | | | ND | V | | | | 9 / 5 | 26 / 15 |
| 84 | x | B | | | | | ND | | V | | | | | | | | ND | | | 3 / 1 | 9 / 4 |
| 85 | | A | ND | V | | ND | ND | | V | V | ND | | | V | ND | V | ND | | | 11 / 5 | 28 / 15 |
| 86 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 9 / 6 |
| 87 | | B | ND | | V | ND | | | | | | | | | ND | | | | | 4 / 1 | 8 / 3 |
| 88 | | A | | V | | | | | V | V | | V | | | ND | | | | | 5 / 4 | 22 / 14 |
| 89 | | A | ND | V | | ND | | | | V | ND | | | | ND | | | | | 6 / 2 | 22 / 12 |
| 90 | | B | ND | V | | ND | | | V | | ND | | | | ND | | | | | 6 / 2 | 20 / 11 |
| 91 | | A | | | | | ND | | | V | | V | | | | | ND | | | 4 / 2 | 20 / 12 |
| 92 | x | A | ND | | | ND | | | V | | | | | | | | | | | 3 / 1 | 20 / 10 |
| 93 | | A | | V | | | | | | | ND | | | | | | | | | 2 / 1 | 18 / 11 |
| 94 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 13 / 8 |
| 95 | | B | ND | | V | ND | | | V | | ND | | | | | V | | | ND | 7 / 3 | 21 / 10 |
| 96 | x | B | ND | | | ND | ND | | V | | | | | | | | ND | | | 5 / 1 | 16 / 7 |
| 97 | | A | ND | | | ND | ND | | | V | ND | V | | | ND | | ND | | | 8 / 2 | 24 / 12 |
| 98 | x | B | ND | V | | ND | ND | | | V | | | | | ND | | ND | | | 7 / 2 | 18 / 8 |
| 99 | | A | ND | V | FN | ND | | | V | | ND | | | | | V | | | | 7 / 3 | 24 / 13 |
| 100 | | A | | V | V | | ND | V | V | V | ND | V | V | V | ND | V | ND | ND | ND | 15 / 9 | 32 / 19 |
| 101 | x | B | | V | | | | | V | | | | | | | | | | | 2 / 2 | 9 / 7 |
| 102 | | A | ND | V | V | ND | ND | | V | | FP | V | V | V | ND | V | ND | ND | | 14 / 7 | 31 / 17 |
| 104 | x | B | ND | | V | ND | ND | | V | | ND | | | | ND | V | ND | | ND | 10 / 3 | 26 / 11 |

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(**Yes***): evaluation for information only

**V** = analysed for and submitted concentration Value for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RLs > assigned values; **FP** = false positive result

**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 | | | Avermectin B1a | Captan | Chlormequat (chloride) | Chlorothalonil | Cyromazine | Dithianon | Dithiocarbamates (expr. as $CS_2$) | Dodine | Emamectin B1a | Fenbutatin Oxide | Folpet | Mepiquat (chloride) | Phthalimide | Pymetrozine | TFNA | TFNG | THPI | analysed / correctly found (Compulsory Compounds, max. 17/10) |
| within MACP/WD | | | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | MACP | |
| present in test item | | | No | **Yes** | No | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | **Yes** | **Yes** | No | No | **Yes** | |
| evaluated in this PT | | | No | **Yes** | No | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | (**Yes***) | **Yes** | No | No | **Yes** | |
| Lab-Code SRM17- | NRL | Cat. | | | | | | | | | | | | | | | | | | |
| 105 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 106 | | A | ND | FN | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 9 |
| 107 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 109 | | A | ND | V | ND | V | V | ND | FN | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 9 |
| 110 | | A | ND | FN | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 9 |
| 111 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 112 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 113 | | B | | | ND | V | V | | V | V | | | | ND | | V | | | | 7 / 5 |
| 114 | | B | ND | V | | V | V | ND | V | V | V | | V | | | V | ND | ND | | 12 / 8 |
| 115 | | B | ND | | | V | V | | V | V | | | | | | V | | | | 6 / 5 |
| 117 | | A | ND | V | ND | V | V | ND | | V | V | ND | V | ND | V | V | ND | ND | V | 16 / 9 |
| 118 | | B | ND | V | ND | V | V | ND | V | V | V | | V | | V | V | | | V | 13 / 10 |
| 119 | | B | | | | | | | V | | | | | | | | | | | 1 / 1 |
| 120 | | A | ND | V | | V | V | ND | V | V | V | ND | V | | V | V | ND | ND | V | 15 / 10 |
| 122 | | B | | | | | | | V | | | | | | | | | | | 1 / 1 |
| 123 | x | B | | V | | V | V | | V | | | | V | | V | V | | | V | 8 / 8 |
| 124 | | B | | V | | V | | | V | | | | V | | V | | | | V | 6 / 6 |
| 125 | | B | ND | V | | V | | | V | V | V | ND | V | | | V | | | | 9 / 7 |
| 126 | | B | | | | V | V | | | | V | | V | | V | V | | | | 6 / 6 |
| 127 | | B | ND | | ND | V | V | ND | V | V | V | ND | | ND | V | V | ND | ND | V | 15 / 8 |
| 128 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | | | V | 15 / 10 |
| 3rd-14 | | B | ND | | | | | | | | V | ND | | | | V | | | | 4 / 2 |
| 3rd-27 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 3rd-83 | | A | ND | V | ND | V | V | ND | V | V | V | ND | V | ND | V | V | ND | ND | V | 17 / 10 |
| 3rd-103 | | B | ND | FN* | ND | FN* | V | ND | | V | V | ND | V | ND | V | V | FP | ND | V | 16 / 7 |
| 3rd-108 | | A | ND | V | ND | V | V | ND | V | V | V | ND | | ND | V | V | ND | ND | V | 16 / 9 |
| 3rd-128 | | B | V | ND | FN | ND | V | | | | | | | | V | ND | ND | ND | V | 10 / 3 |

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(**Yes***): evaluation for information only

**V** = analysed for and submitted concentration Value for a pesticide present in the test item at a level allowing statistical evaluations; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RLs > assigned values; **FP** = false positive result

**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 | | | BAC-C12 (chloride) | Bifenazate (sum) | Chloridazon-desphenyl | DDAC-C10 (chloride) | Diquat (dication) | ETU | Formetanate-HCl | Maleic hydrazide | Matrine | Meptyldinocap | 2,4-DNOP (free phenol) | Meptyldinocap (sum) | Nicotine | Oxymatrine | Paraquat (dication) | PTU | Trimesium (cation) | analysed / correctly found (Optional Compounds, max. 17/7) | analysed / correctly found (Total, max. 34/17) |
| within MACP/WD | | | WD | WD | WD | WD | WD | None | MACP | MACP** | WD | WD | WD | WD | WD | WD (2021) | WD | None | WD | | |
| present in test item | | | No | **Yes** | **Yes** | No | No | **Yes** | **Yes** | **Yes** | No | **Yes** | **Yes** | **Yes** | No | **Yes** | No | No | No | | |
| evaluated in this PT | | | No | **Yes** | **Yes** | No | No | (**Yes***) | **Yes** | **Yes** | No | No | No | No | No | **Yes** | No | No | No | | |
| Lab-Code SRM17- | NRL | Cat. | | | | | | | | | | | | | | | | | | | |
| 105 | | A | ND | V | | ND | | V | V | V | | | | | ND | | | ND | ND | 9 / 4 | 26 / 14 |
| 106 | | A | | V | | | | | V | | ND | V | | | ND | | | | | 5 / 3 | 22 / 12 |
| 107 | | A | ND | V | | ND | ND | | | V | ND | | | | | V | ND | | ND | 9 / 3 | 26 / 13 |
| 109 | | A | | V | | | | | V | FN | | V | | | | | | | | 4 / 3 | 21 / 12 |
| 110 | | A | ND | V | FN | ND | ND | V | V | V | ND | | V | | ND | V | ND | ND | | 14 / 6 | 31 / 15 |
| 111 | | A | ND | V | | ND | | V | V | V | ND | | | | ND | V | | ND | ND | 11 / 5 | 28 / 15 |
| 112 | | A | ND | V | | ND | ND | V | FN | V | ND | | FN | | ND | V | ND | ND | ND | 14 / 4 | 31 / 14 |
| 113 | | B | | | | | | | FN | | | | | | | | | | | 1 / 0 | 8 / 5 |
| 114 | | B | | V | FN | | | | | | | | | | | | | | | 2 / 1 | 14 / 9 |
| 115 | | B | ND | | | ND | | | V | | | | | | ND | | | | | 4 / 1 | 10 / 6 |
| 117 | | A | ND | V | | ND | ND | | V | | | V | V | V | | | ND | | ND | 10 / 5 | 26 / 14 |
| 118 | | B | | V | | | | | V | | | | | | | | | | ND | 3 / 2 | 16 / 12 |
| 119 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 1 / 1 |
| 120 | | A | ND | V | FN | ND | | | V | | ND | V | | | ND | V | | | | 9 / 4 | 24 / 14 |
| 122 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 1 / 1 |
| 123 | x | B | | | | | | | | | | | | | | | | | | 0 / 0 | 8 / 8 |
| 124 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 6 / 6 |
| 125 | | B | ND | V | | ND | | | | | | | | | | | | | | 3 / 1 | 12 / 8 |
| 126 | | B | ND | V | | ND | | | V | | | | | | | | | | | 4 / 2 | 10 / 8 |
| 127 | | B | | V | | | ND | | V | V | ND | V | | | ND | V | ND | | | 9 / 5 | 24 / 13 |
| 128 | | A | | | | | | V | V | | | | | | | | | ND | | 3 / 2 | 18 / 12 |
| 3rd-14 | | B | | | | | ND | | | | | | | | | | ND | | | 2 / 0 | 6 / 2 |
| 3rd-27 | | A | ND | V | V | ND | ND | | V | V | ND | | | | ND | | ND | | | 10 / 4 | 27 / 14 |
| 3rd-83 | | A | ND | | | ND | ND | V | FN | V | ND | | | | | V | ND | ND | | 10 / 3 | 27 / 13 |
| 3rd-103 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 16 / 7 |
| 3rd-108 | | A | ND | | | ND | ND | | V | | ND | | | | ND | V | ND | | | 8 / 2 | 24 / 11 |
| 3rd-128 | | B | | | | | | | | | | | | | | | | | | 0 / 0 | 10 / 3 |

**MACP-Reg**. (covering 2022-2024): REGULATION (EU) 2021/601 of 13 April 2021; **MACP****: inclusion of MH in the MACP was decided at technical level in 2021; **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)

(**Yes***): evaluation for information only

**V** = analysed for and submitted concentration Value for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = FN because of labs' RLs > assigned values; **FP** = false positive result

## 4.2 Assigned Values and Target Standard Deviations

Except ***meptyldinocap, 2,4-DNOP*** and ***meptyldinocap (sum)*** (see **Section 4.2.2, p. 30**) as well as ***phthalimide*** (see **Section 4.2.1, p. 28**), the assigned value ($x_{pt}$) of each analyte present in the test item was established as the mean of robust statistics ($x^*$) of all numerical results submitted by OfLs from EU and EFTA countries, excluding outliers and using Algorithm A for calculation ([6], **Appendix 8**). Results from laboratories outside EU and EFTA countries (i.e., 3rd countries and EU Candidate Countries) were not taken into account. Before setting assigned values, the results population of each analyte was checked for outliers based on the z scores calculated using the robust mean of the entire population and the Grubbs' test (alpha = 0.05). Results assigned with z scores > 5 (using the initial robust mean as a basis) and confirmed as outliers by the Grubbs' test were excluded from the population for establishing the assigned values. Following exclusion of outliers, the robust mean of each analyte was calculated again using the remaining results and established as the assigned value.

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

The assigned values and their uncertainties are shown in **Table 4-3**. In four cases ***meptyldinocap, 2,4-DNOP***, ***meptyldinocap (sum)*** and ***ETU***), the uncertainty of the assigned value exceeded the tolerance and the assigned values were considered too uncertain for properly evaulating the laboratory performance. After consultation with the Scientific Committee at the Evaluation Meeting, no z scores should be calculated for these four analytes in the final report. Alternative "Assigned values" were calculated for informative purposes. For ***meptyldinocap, 2,4-DNOP*** and ***meptyldinocap (sum)***, the spiking levels were employed as "assigned values". For ***ETU*** the uncertainty of the assigned value only slightly exceeded the threshold. In this case, both the z´-scores (z-prime scores) as well as a z score range was calculated by considerung the uncertainty of the assigned value, but both of them were for information only.

**Table 4-3** additionally shows the coefficients of variation ($CV^*$) calculated for each analyte by applying robust statistics to the entire population of results, excluding outliers. The $CV^*$ was also calculated for the analytes that didn't pass the criteria as regards the uncertainty of the assigned value. Among compulsory compounds, except ***captan*** (34.2 %), ***folpet*** (34.5 %), and ***phthalimide*** (36.8 %) the $CV^*$-values were close to or lower than the FFP-RSD (25 %). Excluding ***phtahlimid***, the assigned value of which was based on the sum of the incurred and the spiked level, the average $CV^*$s of all compulsory analytes based on the results population of EU-and EFTA-laboratories was 26.4 %. Among the optional compounds, excluding ***meptyldinocap, 2,4-DNOP***, ***meptyldinocap (sum)***, the average $CV^*$ of data reported by EU-and EFTA-laboratories was 25.2 %. Among all compounds in this PT, the lowest $CV^*$ (16.6 %) was observed for ***maleic hydrazide*** .

### 4.2.1 Evaluation of Results of Captan, THPI, Folpet and Phthalimide

In the EUPT-SRM17 exercise, the overall distribution of the results of ***phthalimide***, even after the exclusion of 7 outliers, was still relatively broad ($CV^*$ = 36.8 %). Knowing about the difficulty of accurately quantifying ***phthalimide*** in the presence of its parent ***folpet***, especially when GC-techniques are used, a relatively broad distribution of the ***phthalimide*** results was indeed expected. As the distribution of the participants' results was not strongly distorted and the uncertainty of the robust mean was clearly within the established limits (UAV-test), the robust mean 0.134 mg/kg was firstly taken as the preliminary assigned value (prAV) in the preliminary report released on 8 April 2022. Unfortunately, at this stage the organisers didn't notice, that all LC-based results submitted by the participants for ***phthalimide*** and the LC-MS/MS results generated by the organisers were towards the low side of the distribution (negative z scores using the prAV). Due to the overwhelmingly high share of GC-based results within the total population (73 out of 83), and due to the broad distribution of the GC-results themselves, the slight indications of bimodality in the result distribution remained unnoticed at this stage.

**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 \| Outliers | No. of Numerical Results (EU+EFTA) | Result Population for AV-Estimation [1] | Assigned Value [mg/kg] | $u(x_{pt})$ [2] [mg/kg] | $u(x_{pt})$ Tolerance [mg/kg] | Judgement for UAV-test | $CV^*$ [3] [%] |
| Compulsory Compounds | Captan | 6 \| 5 | 84 | EU/EFTA-OfLs | 0.174 | ± 0.0080 | 0.0131 | passed | 32.9 |
| | Chlorothalonil | 1 \| 1 | 102 | EU/EFTA-OfLs | 0.151 | ± 0.0046 | 0.0113 | passed | 24.4 |
| | Cyromazine | 0 \| 0 | 92 | EU/EFTA-OfLs | 0.154 | ± 0.0041 | 0.0116 | passed | 20.3 |
| | DTCs (expr. as $CS_2$) | 2 \| 2 | 97 | EU/EFTA-OfLs | 0.187 | ± 0.0068 | 0.014 | passed | 28.4 |
| | Dodine | 0 \| 0 | 96 | EU/EFTA-OfLs | 0.100 | ± 0.0029 | 0.0075 | passed | 23.1 |
| | Emamectin B1a | 0 \| 2 | 91 | EU/EFTA-OfLs | 0.0461 | ± 0.0013 | 0.0035 | passed | 21.5 |
| | Folpet | 1 \| 2 | 89 | EU/EFTA-OfLs | 0.249 | ± 0.0115 | 0.0187 | passed | 34.5 |
| | Phthalimide | 5 \| 7* | 78 | Sum of incurred and spiked level | *0.098* | *± 0.0072* | *0.0074* | passed | *36.2* [5] |
| | Pymetrozine | 1 \| 1 | 100 | EU/EFTA-OfLs | 0.150 | ± 0.0053 | 0.0113 | passed | 28.2 |
| | THPI | 0 \| 1 | 80 | EU/EFTA-OfLs | 0.590 | ± 0.0193 | 0.0443 | passed | 23.2 |
| | **Average [4] $CV^*$** | | | | | | | | **26.4** [6] |
| Optional Compounds | Bifenazate (sum) | 0 \| 2 | 59 | EU/EFTA-OfLs | 0.296 | ± 0.0127 | 0.0222 | passed | 26.0 |
| | Chloridazon-desphenyl | 7 \| 1 | 25 | EU/EFTA-OfLs | 0.0616 | ± 0.0037 | 0.0046 | passed | 23.7 |
| | ETU | 1 \| 0 | 18 | EU/EFTA-OfLs | *0.0629* | *± 0.0061* | *0.0047* | failed | *33.1* |
| | Formetanate-HCl | 5 \| 7 | 76 | EU/EFTA-OfLs | 0.873 | ± 0.0336 | 0.0655 | passed | 25.5 |
| | Maleic hydrazide | 1 \| 0 | 46 | EU/EFTA-OfLs | 0.544 | ± 0.0167 | 0.0408 | passed | 16.6 |
| | Oxymatrine | 0 \| 0 | 36 | EU/EFTA-OfLs | 0.198 | ± 0.0093 | 0.0149 | passed | 22.4 |
| | 2,4-DNOP (free phenol) | 2 \| 2 | 13 | Spiking Level | *0.056* | *± 0.0087* | *0.0042* | failed | *36.5* |
| | Meptyldinocap | 3 \| 12 [6] | 25 | Spiking Level | *0.100* | *± 0.0161* | *0.0075* | failed | *89.8* |
| | Meptyldinocap (sum) | 0 \| 1 | 16 | Spiking Level | *0.169* | *± 0.0275* | *0.0127* | failed | *44.0* |
| | **Average [4] $CV^*$** | | | | | | | | **25.2** [6] |

1: **EU/EFTA-OfLs**: The population consisted of numerical results reported by the OfLs form EU or EFTA countries, but excuding outliers;
**Sum of incurred and spiked level**: The sum of incurred level analysed before spiking and the theoretical spiking level was set as the assigned values and for informative purpose only (Please see also **Section 4.2.1**)
**Spiking Leve**: The theoretical spiking level was set as the assigned value, but for informative purposes only (Please see also **Section 4.2.2**).
2: $u(x_{pt})$**:** Uncertainty of assigned value calculated as shown under **Section 2.2 (p. 13))**
3: $CV^*$ **:** Coefficient of variation (= relative standard deviation) based on robust statistics of entire population excluding outliers, also in case of meptyldinocap, 2,4-DNOP and meptyldinocap (sum) as well as phthalimide where the assigned values were not established using robust mean.
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.
5: The $CV^*$ of phthalimide was excluded for the calculation of the average $CV^*$ of the compulsory compounds, the $CV^*$ of 2,4-DNOP, meptyldinocap and meptyldinocap (sum) were excluded for the calculation of the average $CV^*$ of the optional compounds.
4: calculated using robust approach; the relative deviation to the reference value 0.098 = 51.5 %
6: Taking the spiking level as assigned value, 12 results obtained z score > 5. When applying robust statistics and excluding results with z scores > 5, the very same 12 results were iteratively identified as outliers, too.

As already described in the Final Report on SRM12 and in our analytical observation report (Report SRM-07 (GC), Report SRM-42 (LC)), ***phthalimide*** is generated via the thermal decomposition of ***folpet*** during GC-injection, and this may lead to overestimated ***phthalimide*** results if this effect is not properly considered in calibration. This overestimation tends to be stronger the higher the levels of the intact parent compound (***folpet***) are. In parallel, the decomposition of ***folpet*** would theoretically lead to underestimated results, however, here the losses are mostly well and straightforwardly compensated by the labs, by using a calibration that corrects for matrix effects (e.g. matrix-matched, procedural, standard addition, ILIS-based). In the case of ***phthalimide***, compensating for the increased signal is not straightforward, as the obtained ***phthalimide*** signal entails the signal share of the initial ***phthalimide*** in the extract and the signal share of the ***phthalimide*** formed during ***folpet*** decomposition in the injector (see Report SRM-07 (GC)). Labs not considering this aspect are likely to have overestimated the ***phthalimide***-concentration.

**Table 4-4:** Evaluation of phthalimide based on different results population

| | Basis for Calculation of Robust Mean | | | |
|---|---|---|---|---|
| | All results received | Results generated by GC-based methods | Results generated by LC-based methods (plus the result of homogeneity test) | Sum of Incurred[1)] and spiked level |
| No. Numerical Values | 78 | 69 | 9+1 | 78 |
| No. of Outliers | 2 | 2 | 0 | 7 |
| FN | 5 | 4 | 1 | 5 |
| Robust Mean [mg/kg] | 0.134 | 0.140 | 0.092 | 0.098 [1)] |
| *CV** | 36.8 % | 35.2 % | 27.0 % | 36.2 % [2)] |

1) The incurred level was determined in the sample prior to spiking the sample in the lab using LC-MS/MS
2) calculated using robust approach; the relative deviation to the reference value 0.098 = 51.5 %

For this reason, a revised version of the preliminary report was released on 9 Juni with a new preliminary AV of 0.098 mg/kg for ***phthalimide.*** This value was derived from the ***phthalimide*** concentration measured in the sample homogenate prior to spiking and the ***phthalimide*** amount spiked to the sample during homogenization, given that ***folpet*** remained stable during a simulated homogenization and homogenate storage step. The new assigned value is close to the result obtained by the Organisers in the homogeneity test (0.093 mg/kg) and, furthermore, close to the robust mean of the population of results derived from LC-based measurements supplemented with the result of homogeneity test that was also derived by LC-based measurement. But one should keep in mind, that the number of data within this LC-population is actually too low for applying robust statistics.

After consultation with the Scientific Committee, in the final report the z scores for ***phthalimide*** should be calculated and shown using both the robust mean of the entire population as well as the sum of incurred and spiked levels as assigned value, but for informative purposes only. Following the suggestion of the Scientific Committee, the organisers additionally evaluated the results of the "sum of ***folpet*** and ***phthalimide***" (see **Section 4.4, (p. 54)** ).

In the case of ***THPI*** this effect of overestimation because of thermo-degradation during GC injection was much less pronounced as the ratio of parent (***captan***) to degradant (***THPI)*** was much smaller. Therefore, no change in the assigned value of ***THPI*** was introduced, and ***THPI*** is evaluated regularly using the robust mean of the entire rerults population. However, the organisers evaluated additionally here also the "sum of ***captan*** and ***THPI***" (**Section 4.4, (p. 54)** ).

### 4.2.2 Evaluation of Results of Meptyldinocap, 2,4-DNOP, Meptyldinocap (sum)

The analysis of ***meptyldinocap*** is analytically challenging for various reasons, including its instability in solution and the presence of its degradant ***2,4-DNOP (free phenyl***) in ***meptyldinocap*** standards. ***Meptyldinocap*** and its degradant share the same LC-MS/MS mass-transitions due to in-source fragmentation of ***meptyldinocap***, but the degradant shows a much higher detection sensitivity, which often leads to errors in peak allocation and therefore often to errors in quantification (for details please see "Analysis of Meptyldinocap by QuEChERS followed by alkaline hydrolysis and LC-MS/MS measurement" and **Section 4.4, (p. 62)** ).

In the present PT, the participants were asked to report results for ***meptyldinocap, 2,4-DNOP*** and ***meptyldinocap (sum)***. Due to the lack of experience with the analysis of these compounds or labs' unawareness of the tricky analysis, the quality of the results was rather poor. To support the evaluation of ***meptyldinocap, 2,4-DNOP*** and ***meptyldinocap (sum)***, the organisers run a small-scale survey among the laboratories having analysed at least one of these three analytes. The survey was launched on 23 March 2022. In this survey

the participants were asked to give information on the stock and working solution used as well as on the composition of the sample extracts. Unfortunately, no meaningful conclusion could be made from the answers.

Since the results distribution of ***meptyldinocap*** and ***meptyldinocap (sum)*** was very broad and the number of numerical results for ***2,4-DNOP (free phenyl)*** was not sufficient for a reliable statistical evaluation, the organisers decided to use the spiking levels of these three compounds as preliminary assigned values (prAV) within the preliminary report. The fact that the concentrations determined by the organisers during the homogeneity test were close to the spiked levels supported this decission. Following consultation with EUPT-Scientiffic Committee, it was decided not to calculate z scores for these three analytes in the final report, but rather to plot the concentrations reported highlighting the robust mean as well as the spiked level. The $CV^*$ should also be calculated and shown in the final report.

### 4.2.3 Evaluation of results of ETU

For ***ETU***, the uncertainty of the robust mean of the reported results slightly failed to meet the criterion to qualify as an assigned value. This was mainly due to the small number of results and due to a few deviating results in the upper range. Even after supplementing the results population by the average result of the homogeneity test, the uncertainty remained above the limit of ± 7.5 %, yet still below ± 10 %.

As the uncertainty of the ***ETU*** robust mean only slightly exceeded the limit, it was decided to calculate for information purposes the following: a) the z' score (in accordance with ISO 13528:2015 "Statistical methods for use in proficiency testing by interlaboratory comparison" **[6]**); and b) the z score range for each reported result, considering the uncertainty of the robust mean. The robust mean plus or minus the uncertainty of the assigned value was used to establish the upper or lower limit of the tentative assigned value. All these additional scores were also ***for informative purpose only***. Please see also **Section 2.4.1 (p. 15).** Laboratories that, even when applying the worst-case calculation, achieved a z score well within the acceptable range may assume that they have performed well in the analysis of ***ETU***.

## 4.3 Assessment of Laboratory Performance

### 4.3.1 False Positives

Among the results received from EU/EFTA-OfLs, 4 results submitted by 3 laboratories (one each for *BAC-C12 (chloride), DDAC-C10 (chloride), matrine* and *paraquat (dication)*) were preliminarily judged as FPs. All of them concerned optional analytes. 7 EU/EFTA-OfLs and one 3rd country laboratory reported numerical results for *TFNG*, three of them were in the range between MRRL and 2× MRRL or equal to the MRRL, the other 5 were lower than the lab's RL. Since *TFNG* was present in trace amount in the test material, all these 8 results were not judged as FP, but the later 5 cases with reported results ≤ RL were classified as false reporting. Although *TFNA* was also present in trace amount in the test material, the numerical result for *TFNA* reported by one 3rd country laboratory was 10 times higher than the MRRL and thus judged as FP. All these results are listed in **Table 4-5 (p. 32)**.

### 4.3.2 False Negatives

26 EU/EFTA-OfLs reported in 35 cases results that were preliminarily judged as FNs. These concerned compounds that were present in the test item at relevant levels and were analysed by the labs without report-

**Table 4-5:** False positive results reported in EUPT-SRM17

| | Compound | PT-Code (SRM17-) | Analysed | Reported Result [mg/kg] | RL [mg/kg] | MRRL [mg/kg] | Judgement |
|---|---|---|---|---|---|---|---|
| Compulsory | TFNA | 3rd-103 | Yes | 0.115 | 0.01 | 0.01 | False Positive |
| | TFNG | 70 | Yes | 0.0147 | 0.01 | 0.01 | Not FP. TFNG was present in trace amount in the test material and the reported result close or equal to MRRL |
| | | 89 | Yes | 0.011 | 0.01 | 0.01 | |
| | | 90 | Yes | 0.010 | 0.01 | 0.01 | |
| | | 15 | Yes | 0.00639(FR) | 0.01 | 0.01 | Not FP, TFNG was present in trace amount in the test material, but false reporting (FR) since the reported result results < RL. |
| | | 30 | Yes | 0.004(FR) | 0.01 | 0.01 | |
| | | 47 | Yes | 0.004(FR) | 0.01 | 0.01 | |
| | | 112 | Yes | 0.006(FR) | 0.01 | 0.01 | |
| | | 3rd-103 | Yes | 0.0087(FR) | 0.01 | 0.01 | |
| Optional | BAC-C12 (chloride) | 75 | Yes | 0.68 | 0.02 | 0.02 | False Positive |
| | DDAC-C10 (chloride) | 75 | Yes | 0.099 | 0.01 | 0.01 | False Positive |
| | Matrine | 102 | Yes | 0.029 | 0.02 | 0.02 | False Positive |
| | Paraquat (dication) | 74 | Yes | 0.92 | 0.01 | 0.01 | False Positive |

(FR): False Reporting, reported concentration was lower than the lab's reporting limit

ing any numerical results. In 34 of those cases, the assigned values are higher than the laboratories' RL, therefore, they were finally judged as FNs. For these results z scores were calculated using the corresponding MRRL in the target pesticide list or the RL, if this was lower. The FN results concerned in 7 cases ***chloridazon-desphenyl***, in 5 cases each ***captan, phthalimide*** and ***formetanate-HCl***, in 3 cases ***meptyldinocap***, in 2 cases each ***2,4-DNOP (free phenol)*** and ***dithiocarbamates***, and one case each ***chlorothalonil, ETU, folpet, maleic hydrazide and pymetrozine***. Lab SRM-66 had targeted ***captan*** and reported not detected with its RL being higher than the assigned value. Following the rules layed down in the General Protocol, this result was judged as a false negative, but marked with a note. This laboratory is encouraged to lower its reporting limit for ***captan***.

Furthermore, two laboratories reported numerical results lower than their RLs, Lab 30 in case of ***captan*** and Lab 41 in case of ***chloridazon-desphenyl***. In practice, results lower than the RLs are regarded as not detected and therefore schould not be reported. After consultation with the Scientific Committe these two should be classified as "false reportings" and coded as "(FR)", with their z scores being calculated using the reported values.

Two laboratories from 3rd countries reported false negative results. Lab 3rd-103 reported FNs for ***captan*** and ***chlorothalonil***, whereas Lab 3rd-83 reported a FN result for ***formetanate-HCl***.

All false negative results in the EUPT-SRM17 are listed in **Table 4-6 (p. 33).** 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

As mentioned in **Section 4.2 (p. 28)**, in the case of ***2,4-DNOP, meptyldinocap*** and ***meptyldinocap (sum)*** only the robust mean and the robust standard deviation $CV^*$ of the outlier-cleaned results of EU/EFTA OfLs were calculated. These are shown along with participants' results ***for informative purposes only***. No individual z scores were calculated for these three compounds.

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

| | Compounds | MRRL [mg/kg] | Assigned Value [mg/kg] | PT-Code (SRM17-) | Analysed | Detected | RL [mg/kg] | Judgement |
|---|---|---|---|---|---|---|---|---|
| Compulsory | Captan | 0.02 | 0.174 | 3 | Yes | No | 0.02 | False Negative |
| | | | | 13 | Yes | No | 0.01 | False Negative |
| | | | | 66 | Yes | No | 0.5 | False Negative* |
| | | | | 73 | Yes | No | 0.02 | False Negative |
| | | | | 106 | Yes | No | 0.02 | False Negative |
| | | | | 110 | Yes | No | 0.01 | False Negative |
| | Chlorothalonil | 0.01 | 0.151 | 61 | Yes | No | 0.01 | False Negative |
| | DTCs (expr. as $CS_2$) | 0.02 | 0.187 | 37 | Yes | No | 0.01 | False Negative |
| | | | | 109 | Yes | No | 0.02 | False Negative |
| | Folpet | 0.02 | 0.249 | 13 | Yes | No | 0.01 | False Negative |
| | Phthalimide | 0.01 | 0.134 | 20 | Yes | No | 0.01 | False Negative |
| | | | | 44 | Yes | No | 0.01 | False Negative |
| | | | | 71 | Yes | No | 0.01 | False Negative |
| | | | | 92 | Yes | No | 0.01 | False Negative |
| | | | | 104 | Yes | No | 0.01 | False Negative |
| | Pymetrozine | 0.02 | 0.150 | 49 | Yes | No | 0.02 | False Negative |
| Optional | Chloridazon-desphenyl | 0.02 | 0.0616 | 3 | Yes | No | 0.02 | False Negative |
| | | | | 30 | Yes | No | 0.02 | False Negative |
| | | | | 45 | Yes | No | 0.01 | False Negative |
| | | | | 99 | Yes | No | 0.01 | False Negative |
| | | | | 110 | Yes | No | 0.01 | False Negative |
| | | | | 114 | Yes | No | 0.02 | False Negative |
| | | | | 120 | Yes | No | 0.01 | False Negative |
| | ETU | 0.01 | 0.0629 | 12 | Yes | No | 0.01 | False Negative |
| | Formetanate-HCl | 0.01 | 0.873 | 4 | Yes | No | 0.01 | False Negative |
| | | | | 12 | Yes | No | 0.01 | False Negative |
| | | | | 71 | Yes | No | 0.01 | False Negative |
| | | | | 112 | Yes | No | 0.01 | False Negative |
| | | | | 113 | Yes | No | 0.01 | False Negative |
| | Maleic hydrazide | 0.05 | 0.544 | 109 | Yes | No | 0.05 | False Negative |
| | 2,4-DNOP (free phenol) | 0.01 | 0.056 | 10 | Yes | No | 0.01 | False Negative |
| | | | | 112 | Yes | No | 0.01 | False Negative |
| | Meptyldinocap | 0.02 | 0.100 | 10 | Yes | No | 0.02 | False Negative |
| | | | | 30 | Yes | No | 0.02 | False Negative |
| | | | | 50 | Yes | No | 0.02 | False Negative |

* Lab's RL >> Assigned value, so that this lab could not determine this compound, still judged as FN following the rules of the EUPT General Protocol.

For ***phthalimide***, informative z scores were calculated based on two different reference values: a) the robust mean derived from the outlier-cleaned results population of EU/EFTA OfLs, and b) the sum of the incurred and the spiked level. The latter is also close to the robust mean of the population of results generated using LC-MS/MS, a technique that is considered more accurate for this analysis (see also **Section 4.2.1, p. 28**). Both z score calculations are shown for ***informative purposes only***.

In the case of ***ETU,*** where the uncertainty of the robust mean did not pass the limit for being accepted as an assigned value, z scores, z' scores, and z score ranges that consider the uncertainty of the robust mean were calculated ***for informative purposes***.

**Table 4-7:** Overall performance based on z score classification. Following the decision on the Evaluation Meeting, no z scores were calculated for 2,4-DNOP, meptyldinocap and meptyldinocap (sum), z scores for phthalimide and ETU were calculated for informative purposes only and not included in the subtotal.

| EU and EFTA Official Laboratories | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Compound | No. of results [1] | Acceptable No. (%) | Questionable No. (%) | Unacceptable [1] No. (%) | FNs | AAZ [2] | AAZ [3] |
| Compulsory Compounds | Captan | 90 | 64 (71%) | 7 (8%) | 19 (21%) | 6 | 1.5 | 1.4 |
| | Chlorothalonil | 103 | 94 (91%) | 4 (4%) | 5 (5%) | 1 | 0.9 | 0.9 |
| | Cyromazine | 92 | 84 (91%) | 6 (7%) | 2 (2%) | 0 | 0.7 | 0.7 |
| | Dithiocarbamates (expr. as $CS_2$) | 99 | 81 (82%) | 8 (8%) | 10 (10%) | 2 | 1.1 | 1.1 |
| | Dodine | 96 | 90 (94%) | 5 (5%) | 1 (1%) | 0 | 0.8 | 0.8 |
| | Emamectin B1a | 91 | 85 (93%) | (0%) | 6 (7%) | 0 | 0.8 | 0.8 |
| | Folpet | 90 | 69 (77%) | 8 (9%) | 13 (14%) | 1 | 1.4 | 1.3 |
| | ***Phthalimide*** (AV based on Robust Mean of entire population) | 83 | *68 (82%)* | *4 (5%)* | *11 (13%)* | 5 | *1.5* | *1.3* |
| | ***Phthalimide*** (AV based on incurred + spiked level) | 83 | *45 (54%)* | *13 (16%)* | *25 (30%)* | 5 | *2.0* | *1.9* |
| | Pymetrozine | 101 | 93 (92%) | 5 (5%) | 3 (3%) | 1 | 0.9 | 0.9 |
| | THPI | 80 | 74 (93%) | 4 (5%) | 2 (3%) | 0 | 0.8 | 0.8 |
| | **Subtotal** (Excl. phthalimide) | **842** | **734 (87%)** | **47 (6%)** | **61 (7%)** | 11 | **1.0** | **1.0** |
| Optional Compounds | Bifenazate (sum) | 59 | 53 (90%) | 4 (7%) | 2 (3%) | 0 | 1.0 | 1.0 |
| | Chloridazon-desphenyl | 32 | 23 (72%) | (0%) | 9 (28%) | 7 | 1.5 | 1.0 |
| | ***ETU*** | 19 | *15 (79%)* | *3 (16%)* | *1 (5%)* | 1 | *1.1* | *1.0* |
| | Formetanate-HCl | 81 | 58 (72%) | 7 (9%) | 16 (20%) | 5 | 1.5 | 1.3 |
| | Maleic hydrazide | 47 | 45 (96%) | 1 (2%) | 1 (2%) | 1 | 0.6 | 0.5 |
| | Oxymatrine | 36 | 34 (94%) | 2 (6%) | 0 (0%) | 0 | 0.7 | 0.7 |
| | ***2,4-DNOP (free phenol)*** | 15 | | | | | | |
| | ***Meptyldinocap*** | 28 | | | | | | |
| | ***Meptyldinocap (sum)*** | 16 | | | | | | |
| | **Subtotal** (Excl. ETU, 2,4-DNOP, meptyldinocap, meptyldinocap (sum)) | **255** | **213 (84%)** | **14 (5%)** | **28 (11%)** | **13** | **1.1** | **0.8** |
| **Overall EU/EFTA** | | **1097** | **947 (86%)** | **61 (6%)** | **89 (8%)** | **24** | **1.0** | **0.9** |
| **3rd Country / EU Candidate Country Laboratories** | | | | | | | | |
| | Compound | No. of results [1] | Acceptable No. (%) | Questionable No. (%) | Unacceptable [1] No. (%) | FNs | AAZ [2] | AAZ [3] |
| Compulsory Compounds | Captan | 4 | 2 (50%) | 1 (25%) | 1 (25%) | 1 | – | – |
| | Chlorothalonil | 4 | 3 (75%) | (0%) | 1 (25%) | 1 | – | – |
| | Cyromazine | 4 | 4 (100%) | (0%) | 0 (0%) | 0 | – | – |
| | Dithiocarbamates (expr. as $CS_2$) | 3 | 2 (67%) | 1 (33%) | 0 (0%) | 0 | – | – |
| | Dodine | 4 | 4 (100%) | (0%) | 0 (0%) | 0 | – | – |
| | Emamectin B1a | 5 | 5 (100%) | (0%) | 0 (0%) | 0 | 1.1 | 1.1 |
| | Folpet | 3 | 3 (100%) | (0%) | 0 (0%) | 0 | – | – |
| | ***Phthalimide*** (AV based on Robust Mean of entire population) | 4 | *2 (50%)* | *1 (25%)* | *1 (25%)* | 0 | – | – |
| | ***Phthalimide*** (AV based on incurred + spiked level) | 4 | *0 (0%)* | *1 (25%)* | *3 (75%)* | 0 | – | – |
| | Pymetrozine | 5 | 5 (100%) | (0%) | 0 (0%) | 0 | 1.0 | 1.0 |
| | THPI | 4 | 4 (100%) | (0%) | 0 (0%) | 0 | – | – |
| | **Subtotal** (Excl. phthalimide) | **36** | **32 (89%)** | **2 (6%)** | **2 (6%)** | **2** | **–** | **–** |
| Optional Compounds | Bifenazate (sum) | 1 | (0%) | 1 (100%) | 0 (0%) | 0 | – | – |
| | Chloridazon-desphenyl | 1 | (0%) | 1 (100%) | 0 (0%) | 0 | – | – |
| | ***ETU*** | 1 | *1 (100%)* | *(0%)* | *0 (0%)* | 0 | – | – |
| | Formetanate-HCl | 3 | 2 (67%) | (0%) | 1 (33%) | 1 | – | – |
| | Maleic hydrazide | 2 | 2 (100%) | (0%) | 0 (0%) | 0 | – | – |
| | Oxymatrine | 2 | 2 (100%) | (0%) | 0 (0%) | 0 | – | – |
| | ***2,4-DNOP (free phenol)*** | 0 | | | | | | |
| | ***Meptyldinocap*** | 0 | | | | | | |
| | ***Meptyldinocap (sum)*** | 0 | | | | | | |
| | **Subtotal** (Excl. ETU, 2,4-DNOP, meptyldinocap, meptyldinocap (sum)) | **9** | **6 (67%)** | **2 (22%)** | **1 (11%)** | **1** | **1.8** | **1.8** |
| **Overall 3rd country / EU Candidate Country** | | **45** | **38 (84%)** | **4 (9%)** | **3 (7%)** | **3** | **1.4** | **1.4** |

1) including false negatives (FNs)
2) AAZ calculated for results population ≥ 5 only and included FNs
3) AAZ calculated for results population ≥ 5 only but excluded FNs

For all other compounds, individual z scores were calculated using the robust mean of the outlier-cleaned population of EU/EFTA OfLs as the assigned value and the FFP-RSD of 25 % as target standard deviation. **Table 4-7 (p. 34)** shows the overall classification of z scores achieved by all laboratories for compulsory and optional compounds, based on the rules given in **Section 2.4 (p. 14)**.

Disregarding the data of the compounds that could not be evaluated properly, namely ***phthalimide, ETU, 2,4-DNOP, meptyldinocap*** and ***meptyldinocap (sum)***, 86 % of the z scores obtained by EU/EFTA-OfLs were within the "acceptable" range. Among the compulsory compounds, the overall rate was 87 % and among the optional ones it was 84 %. The analytes showing the lowest rate of acceptable results were ***captan*** (71 %), ***formetanate-HCl*** (72 %), ***folpet*** (77 %) and ***dithiocarbamates*** (82 %). The highest rate of acceptable z scores was achieved for ***maleic hydrazide*** (96 %), followed by ***dodine*** and ***oxymatrine*** (94 % each) and ***Emamectin B1a*** and ***THPI*** (93 % each) and ***pymetrozine*** (92 %). Out of the 14 analytes that were evaluated, a rate of acceptable z scores > 90 % was reached for 9 analytes (64 % of all), between 80 and 90 % for one analyte (7 % of all), and between 70 and 80 % for 4 analytes (28 % of all).

Looking at the results reported by the participating laboratories from EU Candidate or 3rd countries, the rate of "acceptable" z scores was 89 % for compulsory compounds and 67 % the optional ones.

A compilation of all individual results and z scores for each laboratory is shown in **Table 4-8 (p. 36)** for compulsory compounds and **Table 4-9 (p. 42)** for optional compounds. The corresponding kernel density histograms showing the distribution of the reported results are shown in **Appendix 5**. A graphic representation of the z score distribution of each target analyte present in the test item can be seen in **Appendix 6.**

### 4.3.4 Laboratory Classification Based on Scope

All participating laboratories having reported at least one result were classified into categories A or B according to the rules cited in **Section 2.5 (p. 15)**. Following the rules defined in the General Protocol (9th Edition, see **Appendix 8**), a laboratory had to fulfil the following conditions in order to be classified into Category A in the present PT: a) analysis of at least 15 out of the 17 compulsory pesticides on the Target Pesticides List; b) correct detection of at least nine out of the ten compulsory pesticides present in the test item, and c) no false positive results among any of the compounds (compulsory and optional).

A total of 66 EU and EFTA laboratories (56 %) were classified into Category A and 52 (44 %) into Category B. Three of the five laboratories (60 %) from one EU candidate country or 3rd countries were classified into Category A.

Considering the compulsory compounds only but excluding ***phthalimide***, the laboratories from EU and EFTA countries classified into Category A achieved an overall AAZ of 1.0 (n = 586), whereas those classified into Category B achieved an overall AAZ of 1.1 (n = 256). The three laboratories from EU candidate and third countries, classified into Category A achieved an overall AAZ of 0.9 (n = 26), whereas the other two, classified into Category B, achieved an overall AAZ of 1.7 with only 10 results.

**Table 4-10 (p. 48)** and **Table 4-11 (p. 49)** show detailed compilations of the results achieved by laboratories classified into Category A and B, respectively.. For informative purposes, the AAZ was calculated for laboratories with 5 or more individual z scores among the compulsory compounds. For the AAZ calculation any z scores > 5 were set at 5, and the z scores for ***phthalimide*** were excluded. It is highlighted, that performance evaluations referring to individual analytes are of higher importance than combined performance evaluations.

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

| COMPULSORY Compounds | | | | Captan | | Chlorothalonil | | Cyromazine | | Dithiocarbamates (expr. as $CS_2$) | | Dodine | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | | 0.174 | | 0.151 | | 0.154 | | 0.187 | | 0.100 | |
| *CV** | | | | 32.9% | | 24.4% | | 20.3% | | 28.4% | | 23.1% | |
| MRRL [mg/kg] | | | | 0.02 | | 0.01 | | 0.02 | | 0.02 | | 0.01 | |
| Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 10 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) |
| **3** | x | B | 12 / 7 | FN | -3.5 | 0.150 | 0.0 | 0.172 | 0.5 | 0.173 | -0.3 | 0.086 | -0.6 |
| **4** | x | A | 17 / 10 | 0.199 | 0.6 | 0.164 | 0.3 | 0.177 | 0.6 | 0.157 | -0.6 | 0.0973 | -0.1 |
| **5** | x | B | 15 / 8 | 0.34 | 3.9 | 0.152 | 0.0 | 0.145 | -0.2 | 0.216 | 0.6 | 0.153 | 2.1 |
| **6** | | B | 3 / 1 | | | 0.146 | -0.1 | | | | | | |
| **7** | x | B | 9 / 5 | | | 0.42 | 7.1 | 0.16 | 0.2 | 0.21 | 0.5 | | |
| **8** | | B | 9 / 6 | 0.168 | -0.1 | 0.112 | -1.0 | 0.140 | -0.4 | | | 0.087 | -0.5 |
| **9** | | B | 9 / 6 | 0.218 | 1.1 | 0.177 | 0.7 | | | 0.211 | 0.5 | 0.105 | 0.2 |
| **10** | | A | 17 / 10 | 0.076 | -2.2 | 0.140 | -0.3 | 0.148 | -0.2 | 0.608 | 9.0 | 0.110 | 0.4 |
| **11** | | A | 16 / 10 | 0.208 | 0.8 | 0.167 | 0.4 | 0.173 | 0.5 | 0.218 | 0.7 | 0.072 | -1.1 |
| **12** | x | A | 16 / 9 | 0.113 | -1.4 | 0.115 | -1.0 | 0.185 | 0.8 | 0.351 | 3.5 | 0.0761 | -1.0 |
| **13** | | B | 17 / 8 | FN | -3.8 | 0.1224 | -0.8 | 0.1384 | -0.4 | 0.1813 | -0.1 | 0.0906 | -0.4 |
| **15** | | A | 17 / 10 | 0.149 | -0.5 | 0.129 | -0.6 | 0.166 | 0.3 | 0.0703 | -2.5 | 0.0848 | -0.6 |
| **16** | | B | 13 / 8 | 0.153 | -0.4 | 0.149 | -0.1 | | | 0.197 | 0.2 | | |
| **17** | x | A | 15 / 10 | 0.29 | 2.7 | 0.14 | -0.3 | 0.29 | 3.5 | 0.17 | -0.4 | 0.12 | 0.8 |
| **18** | x | A | 17 / 10 | 0.179 | 0.2 | 0.147 | -0.1 | 0.155 | 0.0 | 0.147 | -0.9 | 0.0803 | -0.8 |
| **19** | x | B | 5 / 2 | | | | | | | | | 0.068 | -1.3 |
| **20** | | A | 16 / 9 | 0.218 | 1.1 | 0.110 | -1.1 | 0.148 | -0.2 | 0.307 | 2.6 | 0.154 | 2.2 |
| **21** | | B | 12 / 7 | 0.20 | 0.7 | 0.19 | 1.0 | | | 0.17 | -0.4 | 0.12 | 0.8 |
| **22** | | A | 17 / 10 | 0.151 | -0.5 | 0.141 | -0.3 | 0.152 | -0.1 | 0.168 | -0.4 | 0.0911 | -0.4 |
| **23** | | B | 1 / 1 | | | | | | | 0.18 | -0.1 | | |
| **24** | | B | 6 / 6 | 0.223 | 1.2 | 0.156 | 0.1 | | | 0.163 | -0.5 | | |
| **25** | | A | 17 / 10 | 0.109 | -1.5 | 0.190 | 1.0 | 0.162 | 0.2 | 0.165 | -0.5 | 0.148 | 1.9 |
| **26** | | A | 16 / 9 | 0.190 | 0.4 | 0.120 | -0.8 | 0.130 | -0.6 | 0.160 | -0.6 | | |
| **28** | x | A | 15 / 9 | 0.150 | -0.5 | 0.114 | -1.0 | 0.160 | 0.2 | | | 0.116 | 0.6 |
| **29** | | B | 4 / 3 | | | | | | | | | 0.101 | 0.0 |
| **30** | | A | 17 / 10 | 0.008[FR] | -3.8 | 0.115 | -1.0 | 0.13 | -0.6 | 0.189 | 0.0 | 0.098 | -0.1 |
| **31** | | B | 1 / 1 | | | 0.219 | 1.8 | | | | | | |
| **32** | | B | 4 / 2 | | | | | | | 0.177 | -0.2 | | |
| **33** | | B | 4 / 2 | | | | | | | | | 0.121 | 0.8 |
| **34** | | A | 17 / 10 | 0.134 | -0.9 | 0.134 | -0.5 | 0.106 | -1.2 | 0.181 | -0.1 | 0.088 | -0.5 |
| **35** | x | A | 17 / 10 | 0.198 | 0.6 | 0.176 | 0.7 | 0.196 | 1.1 | 0.203 | 0.3 | 0.084 | -0.6 |
| **36** | x | A | 17 / 10 | 0.193 | 0.5 | 0.103 | -1.3 | 0.173 | 0.5 | 0.190 | 0.1 | 0.0672 | -1.3 |
| **37** | | A | 17 / 9 | 0.159 | -0.3 | 0.105 | -1.2 | 0.166 | 0.3 | FN | -3.8 | 0.108 | 0.3 |
| **38** | | A | 17 / 10 | 0.130 | -1.0 | 0.147 | -0.1 | 0.160 | 0.2 | 0.200 | 0.3 | 0.113 | 0.5 |
| **39** | | A | 17 / 10 | 0.276 | 2.4 | 0.191 | 1.1 | 0.179 | 0.6 | 0.211 | 0.5 | 0.0993 | 0.0 |
| **40** | | A | 17 / 10 | 0.026 | -3.4 | 0.112 | -1.0 | 0.087 | -1.7 | 0.151 | -0.8 | 0.099 | 0.0 |
| **41** | | A | 17 / 10 | 0.063 | -2.5 | 0.12 | -0.8 | 0.075 | -2.1 | 0.2 | 0.3 | 0.137 | 1.5 |
| **42** | | A | 17 / 10 | 2.2 | 46.6 | 0.18 | 0.8 | 0.20 | 1.2 | 0.24 | 1.1 | 0.085 | -0.6 |
| **43** | x | A | 16 / 9 | 0.211 | 0.9 | 0.0914 | -1.6 | 0.144 | -0.3 | 0.600 | 8.8 | 0.107 | 0.3 |
| **44** | | A | 17 / 9 | 0.218 | 1.1 | 0.22 | 1.8 | 0.145 | -0.2 | 0.2 | 0.3 | 0.13 | 1.2 |
| **45** | | A | 17 / 10 | 0.174 | 0.0 | 0.211 | 1.6 | 0.164 | 0.3 | 0.129 | -1.2 | 0.133 | 1.3 |
| **46** | | B | 4 / 3 | | | | | 0.197 | 1.1 | | | 0.088 | -0.5 |
| **47** | | A | 17 / 10 | 0.147 | -0.6 | 0.139 | -0.3 | 0.194 | 1.0 | 0.159 | -0.6 | 0.091 | -0.4 |

[FR]: false reporting, since the reported result < lab's reporting limit and in practice should not have been reported.

| COMPULSORY Compounds | | | | Emamectin B1a | | Folpet | | Pymetrozine | | THPI | | Phthalimide* | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | | 0.0461 | | 0.249 | | 0.150 | | 0.590 | | | *0.134** | *0.098** |
| *CV** | | | | 21.5% | | 34.5% | | 28.2% | | 23.2% | | | *36.8%** | *36.2%** |
| MRRL [mg/kg] | | | | 0.01 | | 0.02 | | 0.02 | | 0.01 | | 0.01 | | |
| Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 10 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) |
| **3** | x | B | 12 / 7 | 0.038 | -0.7 | 0.218 | -0.5 | 0.187 | 1.0 | | | | | |
| **4** | x | A | 17 / 10 | 0.0387 | -0.6 | 0.309 | 1.0 | 0.165 | 0.4 | 0.690 | *0.7* | *0.190* | *1.7* | *3.8* |
| **5** | x | B | 15 / 8 | 0.0457 | 0.0 | 0.51 | 4.2 | 0.196 | 1.2 | | | | | |
| **6** | | B | 3 / 1 | | | | | | | | | | | |
| **7** | x | B | 9 / 5 | 0.064 | 1.6 | | | 0.24 | 2.4 | | | | | |
| **8** | | B | 9 / 6 | | | 0.230 | -0.3 | 0.120 | -0.8 | | | | | |
| **9** | | B | 9 / 6 | 0.036 | -0.9 | 0.299 | 0.8 | | | | | | | |
| **10** | | A | 17 / 10 | 0.048 | 0.2 | 0.250 | 0.0 | 0.155 | 0.1 | 0.712 | *0.8* | *0.197* | *1.9* | *4.0* |
| **11** | | A | 16 / 10 | 0.048 | 0.2 | 0.312 | 1.0 | 0.184 | 0.9 | 0.677 | *0.6* | *0.169* | *1.0* | *2.9* |
| **12** | x | A | 16 / 9 | 0.0548 | 0.8 | 0.141 | -1.7 | 0.105 | -1.2 | | | *0.169* | *1.0* | *2.9* |
| **13** | | B | 17 / 8 | 0.0568 | 0.9 | FN | -3.8 | 0.1176 | -0.9 | 0.8506 | *1.8* | *0.3776* | *7.3* | *11.4* |
| **15** | | A | 17 / 10 | 0.0249 | -1.8 | 0.204 | -0.7 | 0.173 | 0.6 | 0.594 | *0.0* | *0.170* | *1.1* | *2.9* |
| **16** | | B | 13 / 8 | 0.041 | -0.4 | 0.225 | -0.4 | 0.160 | 0.3 | 0.645 | *0.4* | *0.159* | *0.7* | *2.5* |
| **17** | x | A | 15 / 10 | 0.085 | 3.4 | 0.37 | 1.9 | 0.20 | 1.3 | 0.40 | *-1.3* | *0.15* | *0.5* | *2.1* |
| **18** | x | A | 17 / 10 | 0.0420 | -0.3 | 0.216 | -0.5 | 0.169 | 0.5 | 0.790 | *1.4* | *0.124* | *-0.3* | *1.1* |
| **19** | x | B | 5 / 2 | 0.04 | -0.5 | | | | | | | | | |
| **20** | | A | 16 / 9 | 0.039 | -0.6 | 0.340 | 1.5 | 0.183 | 0.9 | 0.536 | *-0.4* | *FN* | *-3.7* | *-3.6* |
| **21** | | B | 12 / 7 | 0.055 | 0.8 | 0.29 | 0.7 | 0.095 | -1.5 | | | | | |
| **22** | | A | 17 / 10 | 0.0361 | -0.9 | 0.213 | -0.6 | 0.153 | 0.1 | 0.639 | *0.3* | *0.0864* | *-1.4* | *-0.5* |
| **23** | | B | 1 / 1 | | | | | | | | | | | |
| **24** | | B | 6 / 6 | | | 0.263 | 0.2 | | | 0.517 | *-0.5* | *0.121* | *-0.4* | *0.9* |
| **25** | | A | 17 / 10 | 0.219 | 15.0 | 0.167 | -1.3 | 0.155 | 0.1 | 0.575 | *-0.1* | *0.129* | *-0.1* | *1.3* |
| **26** | | A | 16 / 9 | 0.0490 | 0.3 | 0.175 | -1.2 | 0.168 | 0.5 | 0.136 | *-3.1* | *0.108* | *-0.8* | *0.4* |
| **28** | x | A | 15 / 9 | 0.0455 | 0.0 | 0.227 | -0.4 | 0.186 | 1.0 | 0.594 | *0.0* | *0.0766* | *-1.7* | *-0.9* |
| **29** | | B | 4 / 3 | 0.043 | -0.3 | | | 0.080 | -1.9 | | | | | |
| **30** | | A | 17 / 10 | 0.040 | -0.5 | 0.12 | -2.1 | 0.104 | -1.2 | 0.57 | *-0.1* | *0.079* | *-1.6* | *-0.8* |
| **31** | | B | 1 / 1 | | | | | | | | | | | |
| **32** | | B | 4 / 2 | | | | | 0.141 | -0.2 | | | | | |
| **33** | | B | 4 / 2 | | | | | 0.180 | 0.8 | | | | | |
| **34** | | A | 17 / 10 | 0.028 | -1.6 | 0.301 | 0.8 | 0.158 | 0.2 | 0.519 | *-0.5* | *0.054* | *-2.4* | *-1.8* |
| **35** | x | A | 17 / 10 | 0.045 | -0.1 | 0.291 | 0.7 | 0.169 | 0.5 | 0.706 | *0.8* | *0.152* | *0.5* | *2.2* |
| **36** | x | A | 17 / 10 | 0.0464 | 0.0 | 0.265 | 0.3 | 0.152 | 0.1 | 0.609 | *0.1* | *0.185* | *1.5* | *3.6* |
| **37** | | A | 17 / 9 | 0.037 | -0.8 | 0.214 | -0.6 | 0.143 | -0.2 | 0.648 | *0.4* | *0.088* | *-1.4* | *-0.4* |
| **38** | | A | 17 / 10 | 0.045 | -0.1 | 0.227 | -0.4 | 0.130 | -0.5 | 0.560 | *-0.2* | *0.170* | *1.1* | *2.9* |
| **39** | | A | 17 / 10 | 0.0506 | 0.4 | 0.320 | 1.1 | 0.220 | 1.9 | 0.456 | *-0.9* | *0.102* | *-1.0* | *0.2* |
| **40** | | A | 17 / 10 | 0.049 | 0.3 | 0.157 | -1.5 | 0.112 | -1.0 | 0.311 | *-1.9* | *0.097* | *-1.1* | *0.0* |
| **41** | | A | 17 / 10 | 0.048 | 0.2 | 0.21 | -0.6 | 0.08 | -1.9 | 0.77 | *1.2* | *0.15* | *0.5* | *2.1* |
| **42** | | A | 17 / 10 | 0.059 | 1.1 | 0.22 | -0.5 | 0.14 | -0.3 | 0.65 | *0.4* | *0.15* | *0.5* | *2.1* |
| **43** | x | A | 16 / 9 | | | 0.227 | -0.4 | 0.157 | 0.2 | 0.443 | *-1.0* | *0.0663* | *-2.0* | *-1.3* |
| **44** | | A | 17 / 9 | 0.047 | 0.1 | 0.167 | -1.3 | 0.142 | -0.2 | 0.531 | *-0.4* | *FN* | *-3.7* | *-3.6* |
| **45** | | A | 17 / 10 | 0.058 | 1.0 | 0.243 | -0.1 | 0.186 | 1.0 | 0.529 | *-0.4* | *0.106* | *-0.8* | *0.3* |
| **46** | | B | 4 / 3 | | | | | 0.101 | -1.3 | | | | | |
| **47** | | A | 17 / 10 | 0.029 | -1.5 | 0.282 | 0.5 | 0.203 | 1.4 | 0.631 | *0.3* | *0.113* | *-0.6* | *0.6* |

* z scores based on different "assigned values" for phthalimide for informative purposes only.
0.134 mg/kg: robust mean of entire population from EU-/EFTA OfLs with $CV^*$ = 36.8 %
0.098 mg/kg = sum of the incurred and spiked level with $CV^*$ = 36.2 % and relative deviation to the reference value 0.098 = 51.5 %

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

| COMPULSORY Compounds | | | | Captan | | Chlorothalonil | | Cyromazine | | Dithiocarbamates (expr. as $CS_2$) | | Dodine | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | | 0.174 | | 0.151 | | 0.154 | | 0.187 | | 0.100 | |
| *CV** | | | | 32.9% | | 24.4% | | 20.3% | | 28.4% | | 23.1% | |
| MRRL [mg/kg] | | | | 0.02 | | 0.01 | | 0.02 | | 0.02 | | 0.01 | |
| Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 10 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) |
| **48** | | A | 17 / 10 | 0.180 | 0.2 | 0.245 | 2.5 | 0.160 | 0.2 | 0.250 | 1.3 | 0.085 | -0.6 |
| **49** | x | B | 8 / 4 | 0.289 | 2.7 | 0.165 | 0.4 | | | 0.191 | 0.1 | | |
| **50** | x | A | 17 / 10 | 0.183 | 0.3 | 0.143 | -0.2 | 0.167 | 0.3 | 0.301 | 2.4 | 0.094 | -0.2 |
| **51** | | A | 17 / 10 | 0.184 | 0.3 | 0.168 | 0.5 | 0.190 | 0.9 | 0.240 | 1.1 | 0.092 | -0.3 |
| **52** | | B | 5 / 4 | | | | | 0.161 | 0.2 | 0.237 | 1.1 | 0.149 | 2.0 |
| **53** | | A | 17 / 10 | 0.220 | 1.1 | 0.170 | 0.5 | 0.201 | 1.2 | 0.213 | 0.6 | 0.089 | -0.4 |
| **54** | | A | 16 / 10 | 0.192 | 0.5 | 0.166 | 0.4 | 0.162 | 0.2 | 0.156 | -0.7 | 0.0579 | -1.7 |
| **55** | x | A | 16 / 9 | 0.212 | 0.9 | 0.167 | 0.4 | 0.128 | -0.7 | 0.172 | -0.3 | 0.108 | 0.3 |
| **56** | x | B | 14 / 9 | 1.402 | 28.6 | 0.264 | 3.0 | 0.166 | 0.3 | 0.299 | 2.4 | 0.108 | 0.3 |
| **57** | | A | 15 / 9 | | | 0.0240 | -3.4 | 0.0605 | -2.4 | 0.124 | -1.3 | 0.0889 | -0.4 |
| **58** | x | A | 17 / 10 | 0.210 | 0.9 | 0.127 | -0.6 | 0.155 | 0.0 | 0.219 | 0.7 | 0.071 | -1.2 |
| **59** | | B | 1 / 1 | | | | | | | 0.152 | -0.7 | | |
| **60** | x | B | 12 / 7 | | | 0.086 | -1.7 | 0.148 | -0.2 | 0.412 | 4.8 | 0.0698 | -1.2 |
| **61** | | A | 17 / 9 | 0.220 | 1.1 | FN | -3.7 | 0.132 | -0.6 | 0.360 | 3.7 | 0.087 | -0.5 |
| **62** | | A | 17 / 10 | 0.175 | 0.1 | 0.186 | 0.9 | 0.138 | -0.4 | 0.371 | 3.9 | 0.123 | 0.9 |
| **63** | x | B | 5 / 1 | | | 0.13 | -0.6 | | | | | | |
| **64** | x | A | 17 / 10 | 0.187 | 0.3 | 0.155 | 0.1 | 0.125 | -0.8 | 0.0488 | -3.0 | 0.0893 | -0.4 |
| **66** | | B | 9 / 4 | FN* | -3.5 | 0.053 | -2.6 | | | 0.099 | -1.9 | 0.154 | 2.2 |
| **67** | | A | 16 / 10 | 0.110 | -1.4 | 0.131 | -0.5 | 0.164 | 0.3 | 0.265 | 1.7 | 0.121 | 0.8 |
| **68** | x | A | 17 / 10 | 0.158 | -0.3 | 0.200 | 1.3 | 0.203 | 1.3 | 0.187 | 0.0 | 0.083 | -0.7 |
| **69** | | A | 17 / 10 | 0.189 | 0.4 | 0.160 | 0.2 | 0.164 | 0.3 | 0.077 | -2.4 | 0.112 | 0.5 |
| **70** | | A | 16 / 9 | 0.242 | 1.6 | 0.197 | 1.2 | 0.170 | 0.4 | | | 0.106 | 0.2 |
| **71** | | B | 11 / 6 | 0.245 | 1.7 | 0.151 | 0.0 | | | 0.181 | -0.1 | | |
| **72** | x | A | 17 / 10 | 0.135 | -0.9 | 0.163 | 0.3 | 0.159 | 0.1 | 0.180 | -0.1 | 0.108 | 0.3 |
| **73** | | B | 14 / 8 | FN | -3.5 | 0.228 | 2.0 | 0.225 | 1.8 | 0.161 | -0.6 | 0.078 | -0.9 |
| **74** | | A | 17 / 10 | 0.15 | -0.5 | 0.12 | -0.8 | 0.067 | -2.3 | 0.20 | 0.3 | 0.071 | -1.2 |
| **75** | | B | 6 / 4 | | | | | 0.118 | -0.9 | 0.146 | -0.9 | 0.049 | -2.0 |
| **76** | | B | 11 / 9 | 0.0170 | -3.6 | 0.0975 | -1.4 | 0.0691 | -2.2 | | | 0.103 | 0.1 |
| **77** | | A | 17 / 10 | 0.143 | -0.7 | 0.138 | -0.3 | 0.141 | -0.3 | 0.209 | 0.5 | 0.0882 | -0.5 |
| **78** | x | A | 17 / 10 | 0.183 | 0.3 | 0.117 | -0.9 | 0.194 | 1.0 | 0.202 | 0.3 | 0.099 | 0.0 |
| **79** | | B | 5 / 5 | 0.238 | 1.5 | | | | | | | 0.0952 | -0.2 |
| **80** | | B | 1 / 1 | | | | | | | 0.225 | 0.8 | | |
| **81** | | B | 1 / 1 | | | | | | | 0.165 | -0.5 | | |
| **82** | | A | 17 / 10 | 0.120 | -1.2 | 0.148 | -0.1 | 0.158 | 0.1 | 0.196 | 0.2 | 0.114 | 0.6 |
| **84** | x | B | 6 / 3 | | | 0.168 | 0.5 | 0.152 | -0.1 | | | 0.047 | -2.1 |
| **85** | | A | 17 / 10 | 0.156 | -0.4 | 0.144 | -0.2 | 0.160 | 0.2 | 0.134 | -1.1 | 0.098 | -0.1 |
| **86** | | B | 9 / 6 | 0.178 | 0.1 | 0.161 | 0.3 | | | 0.179 | -0.2 | | |
| **87** | | B | 4 / 2 | | | | | 0.223 | 1.8 | 0.0986 | -1.9 | | |
| **88** | | A | 17 / 10 | 0.279 | 2.4 | 0.192 | 1.1 | 0.076 | -2.0 | 0.251 | 1.4 | 0.110 | 0.4 |
| **89** | | A | 16 / 10 | 0.198 | 0.6 | 0.206 | 1.5 | 0.123 | -0.8 | 0.136 | -1.1 | 0.116 | 0.6 |
| **90** | | B | 14 / 9 | 1.450 | 29.7 | 0.039 | -3.0 | 0.071 | -2.2 | | | 0.150 | 2.0 |
| **91** | | A | 16 / 10 | 0.025 | -3.4 | 0.139 | -0.3 | 0.117 | -1.0 | 0.292 | 2.2 | 0.133 | 1.3 |
| **92** | x | A | 17 / 9 | 0.221 | 1.1 | 0.214 | 1.7 | 0.150 | -0.1 | 0.170 | -0.4 | 0.119 | 0.8 |

| COMPULSORY Compounds | | | | Emamectin B1a | | Folpet | | Pymetrozine | | THPI | | Phthalimide* | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | | 0.0461 | | 0.249 | | 0.150 | | 0.590 | | | *0.134** | *0.098** |
| *CV** | | | | 21.5% | | 34.5% | | 28.2% | | 23.2% | | | *36.8%** | *36.2%** |
| MRRL [mg/kg] | | | | 0.01 | | 0.02 | | 0.02 | | 0.01 | | | 0.01 | |
| Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 10 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) |
| **48** | | A | 17 / 10 | 0.057 | 1.0 | 0.255 | 0.1 | 0.155 | 0.1 | 0.700 | *0.7* | *0.180* | *1.4* | *3.3* |
| **49** | x | B | 8 / 4 | | | 0.148 | -1.6 | FN | -3.5 | | | | | |
| **50** | x | A | 17 / 10 | 0.063 | 1.5 | 0.247 | 0.0 | 0.127 | -0.6 | 0.463 | *-0.9* | *0.066* | *-2.0* | *-1.3* |
| **51** | | A | 17 / 10 | 0.050 | 0.3 | 0.291 | 0.7 | 0.188 | 1.0 | 0.705 | *0.8* | *0.148* | *0.4* | *2.0* |
| **52** | | B | 5 / 4 | | | | | 0.162 | 0.3 | | | | | |
| **53** | | A | 17 / 10 | 0.056 | 0.9 | 0.287 | 0.6 | 0.155 | 0.1 | 0.636 | *0.3* | *0.0964* | *-1.1* | *-0.1* |
| **54** | | A | 16 / 10 | 0.0443 | -0.1 | 0.329 | 1.3 | 0.159 | 0.2 | 0.535 | *-0.4* | *0.0862* | *-1.4* | *-0.5* |
| **55** | x | A | 16 / 9 | 0.050 | 0.3 | 0.327 | 1.3 | 0.163 | 0.3 | | | *0.069* | *-1.9* | *-1.2* |
| **56** | x | B | 14 / 9 | 0.045 | -0.1 | 1.164 | 14.7 | 0.198 | 1.3 | | | *0.140* | *0.2* | *1.7* |
| **57** | | A | 15 / 9 | 0.0356 | -0.9 | 0.0368 | -3.4 | 0.0828 | -1.8 | 0.544 | *-0.3* | *0.0786* | *-1.7* | *-0.8* |
| **58** | x | A | 17 / 10 | 0.042 | -0.3 | 0.281 | 0.5 | 0.170 | 0.5 | 0.688 | *0.7* | *0.081* | *-1.6* | *-0.7* |
| **59** | | B | 1 / 1 | | | | | | | | | | | |
| **60** | x | B | 12 / 7 | 0.0645 | 1.6 | | | 0.107 | -1.1 | | | *0.239* | *3.1* | *5.8* |
| **61** | | A | 17 / 9 | 0.039 | -0.6 | 0.342 | 1.5 | 0.194 | 1.2 | 0.426 | *-1.1* | *0.108* | *-0.8* | *0.4* |
| **62** | | A | 17 / 10 | 0.046 | 0.0 | 0.306 | 0.9 | 0.136 | -0.4 | 0.473 | *-0.8* | *0.094* | *-1.2* | *-0.2* |
| **63** | x | B | 5 / 1 | | | | | | | | | | | |
| **64** | x | A | 17 / 10 | 0.0379 | -0.7 | 0.284 | 0.6 | 0.181 | 0.8 | 0.660 | *0.5* | *0.0808* | *-1.6* | *-0.7* |
| **66** | | B | 9 / 4 | | | | | 0.032 | -3.1 | | | | | |
| **67** | | A | 16 / 10 | 0.047 | 0.1 | 0.181 | -1.1 | 0.181 | 0.8 | 0.627 | *0.3* | *0.106* | *-0.8* | *0.3* |
| **68** | x | A | 17 / 10 | 0.039 | -0.6 | 0.290 | 0.7 | 0.124 | -0.7 | 0.720 | *0.9* | *0.120* | *-0.4* | *0.9* |
| **69** | | A | 17 / 10 | 0.046 | 0.0 | 0.301 | 0.8 | 0.136 | -0.4 | 0.547 | *-0.3* | *0.125* | *-0.3* | *1.1* |
| **70** | | A | 16 / 9 | 0.0402 | -0.5 | 0.546 | 4.8 | 0.145 | -0.1 | 0.556 | *-0.2* | *0.145* | *0.3* | *1.9* |
| **71** | | B | 11 / 6 | 0.0477 | 0.1 | 0.567 | 5.1 | | | 0.524 | *-0.4* | *FN* | *-3.7* | *-3.6* |
| **72** | x | A | 17 / 10 | 0.0539 | 0.7 | 0.225 | -0.4 | 0.181 | 0.8 | 0.638 | *0.3* | *0.298* | *4.9* | *8.2* |
| **73** | | B | 14 / 8 | 0.044 | -0.2 | 0.240 | -0.1 | 0.143 | -0.2 | 0.721 | *0.9* | | | |
| **74** | | A | 17 / 10 | 0.036 | -0.9 | 0.21 | -0.6 | 0.073 | -2.1 | 0.75 | *1.1* | *0.16* | *0.8* | *2.5* |
| **75** | | B | 6 / 4 | | | | | 0.137 | -0.3 | | | | | |
| **76** | | B | 11 / 9 | 0.0898 | 3.8 | 0.0336 | -3.5 | 0.0849 | -1.7 | 0.752 | *1.1* | *0.189* | *1.6* | *3.7* |
| **77** | | A | 17 / 10 | 0.0505 | 0.4 | 0.267 | 0.3 | 0.102 | -1.3 | 0.561 | *-0.2* | *0.201* | *2.0* | *4.2* |
| **78** | x | A | 17 / 10 | 0.044 | -0.2 | 0.233 | -0.3 | 0.212 | 1.7 | 0.548 | *-0.3* | *0.081* | *-1.6* | *-0.7* |
| **79** | | B | 5 / 5 | | | 0.286 | 0.6 | | | 0.541 | *-0.3* | *0.124* | *-0.3* | *1.1* |
| **80** | | B | 1 / 1 | | | | | | | | | | | |
| **81** | | B | 1 / 1 | | | | | | | | | | | |
| **82** | | A | 17 / 10 | 0.063 | 1.5 | 0.240 | -0.1 | 0.180 | 0.8 | 0.542 | *-0.3* | *0.171* | *1.1* | *3.0* |
| **84** | x | B | 6 / 3 | | | | | | | | | | | |
| **85** | | A | 17 / 10 | 0.041 | -0.4 | 0.244 | -0.1 | 0.219 | 1.8 | 0.471 | *-0.8* | *0.155* | *0.6* | *2.3* |
| **86** | | B | 9 / 6 | 0.040 | -0.5 | 0.178 | -1.1 | 0.157 | 0.2 | | | | | |
| **87** | | B | 4 / 2 | | | | | | | | | | | |
| **88** | | A | 17 / 10 | 0.054 | 0.7 | 0.430 | 2.9 | 0.090 | -1.6 | 0.621 | *0.2* | *0.096* | *-1.1* | *-0.1* |
| **89** | | A | 16 / 10 | 0.061 | 1.3 | 0.285 | 0.6 | 0.152 | 0.1 | 0.549 | *-0.3* | *0.120* | *-0.4* | *0.9* |
| **90** | | B | 14 / 9 | 0.040 | -0.5 | 0.080 | -2.7 | 0.076 | -2.0 | 0.720 | *0.9* | *0.110* | *-0.7* | *0.5* |
| **91** | | A | 16 / 10 | 0.064 | 1.6 | 0.084 | -2.7 | 0.089 | -1.6 | 0.596 | *0.0* | *0.176* | *1.3* | *3.2* |
| **92** | x | A | 17 / 9 | 0.139 | 8.1 | 0.112 | -2.2 | 0.151 | 0.0 | 0.180 | *-2.8* | *FN* | *-3.7* | *-3.6* |

* z scores based on different "assigned values" for phthalimide for informative purposes only.
0.134 mg/kg: robust mean of entire population from EU-/EFTA OfLs with $CV^*$ = 36.8 %
0.098 mg/kg = sum of the incurred and spiked level with $CV^*$ = 36.2 % and relative deviation to the reference value 0.098 = 51.5 %

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

| COMPULSORY Compounds | | | | Captan | | Chlorothalonil | | Cyromazine | | Dithiocarbamates (expr. as $CS_2$) | | Dodine | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | | 0.174 | | 0.151 | | 0.154 | | 0.187 | | 0.100 | |
| *CV** | | | | 32.9% | | 24.4% | | 20.3% | | 28.4% | | 23.1% | |
| MRRL [mg/kg] | | | | 0.02 | | 0.01 | | 0.02 | | 0.02 | | 0.01 | |
| Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 10 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) |
| **93** | | A | 16 / 10 | 0.164 | -0.2 | 0.172 | 0.6 | 0.160 | 0.2 | 0.111 | -1.6 | 0.164 | 2.6 |
| **94** | | B | 13 / 8 | 0.202 | 0.7 | 0.113 | -1.0 | | | 0.174 | -0.3 | 0.0838 | -0.6 |
| **95** | | B | 14 / 7 | | | 0.181 | 0.8 | 0.179 | 0.6 | 0.213 | 0.6 | 0.107 | 0.3 |
| **96** | x | B | 11 / 6 | 0.178 | 0.1 | 0.178 | 0.7 | | | | | | |
| **97** | | A | 16 / 10 | 0.128 | -1.0 | 0.232 | 2.1 | 0.140 | -0.4 | 0.050 | -2.9 | 0.099 | 0.0 |
| **98** | x | B | 11 / 6 | | | 0.14 | -0.3 | 0.15 | -0.1 | 0.28 | 2.0 | 0.096 | -0.2 |
| **99** | | A | 17 / 10 | 0.086 | -2.0 | 0.165 | 0.4 | 0.140 | -0.4 | 0.210 | 0.5 | 0.110 | 0.4 |
| **100** | | A | 17 / 10 | 0.309 | 3.1 | 0.194 | 1.1 | 0.099 | -1.4 | 0.141 | -1.0 | 0.058 | -1.7 |
| **101** | x | B | 7 / 5 | | | 0.126 | -0.7 | 0.296 | 3.7 | | | 0.024 | -3.0 |
| **102** | | A | 17 / 10 | 0.182 | 0.2 | 0.131 | -0.5 | 0.142 | -0.3 | 0.116 | -1.5 | 0.072 | -1.1 |
| **104** | x | B | 16 / 8 | 0.250 | 1.8 | 0.169 | 0.5 | 0.160 | 0.2 | | | 0.096 | -0.2 |
| **105** | | A | 17 / 10 | 0.17 | 0.0 | 0.17 | 0.5 | 0.153 | 0.0 | 0.23 | 0.9 | 0.076 | -1.0 |
| **106** | | A | 17 / 9 | FN | -3.5 | 0.124 | -0.7 | 0.139 | -0.4 | 0.146 | -0.9 | 0.109 | 0.4 |
| **107** | | A | 17 / 10 | 0.144 | -0.7 | 0.153 | 0.1 | 0.158 | 0.1 | 0.1883 | 0.0 | 0.0874 | -0.5 |
| **109** | | A | 17 / 9 | 0.046 | -2.9 | 0.20 | 1.3 | 0.075 | -2.1 | FN | -3.6 | 0.072 | -1.1 |
| **110** | | A | 17 / 9 | FN | -3.8 | 0.0501 | -2.7 | 0.168 | 0.4 | 0.17 | -0.4 | 0.123 | 0.9 |
| **111** | | A | 17 / 10 | 0.19 | 0.4 | 0.174 | 0.6 | 0.14 | -0.4 | 0.135 | -1.1 | 0.13 | 1.2 |
| **112** | | A | 17 / 10 | 0.585 | 9.4 | 0.152 | 0.0 | 0.158 | 0.1 | 0.272 | 1.8 | 0.138 | 1.5 |
| **113** | | B | 7 / 5 | | | 0.110 | -1.1 | 0.136 | -0.5 | 0.20 | 0.3 | 0.109 | 0.4 |
| **114** | | B | 12 / 8 | 0.213 | 1.0 | 0.169 | 0.5 | 0.154 | 0.0 | 0.175 | -0.3 | 0.0942 | -0.2 |
| **115** | | B | 6 / 5 | | | 0.14 | -0.3 | 0.11 | -1.1 | 0.19 | 0.1 | 0.089 | -0.4 |
| **117** | | A | 16 / 9 | 0.117 | -1.3 | 0.133 | -0.5 | 0.174 | 0.5 | | | 0.118 | 0.7 |
| **118** | | B | 13 / 10 | 0.191 | 0.4 | 0.186 | 0.9 | 0.163 | 0.2 | 0.193 | 0.1 | 0.102 | 0.1 |
| **119** | | B | 1 / 1 | | | | | | | 0.243 | 1.2 | | |
| **120** | | A | 15 / 10 | 0.03 | -3.3 | 0.143 | -0.2 | 0.221 | 1.7 | 0.038 | -3.2 | 0.113 | 0.5 |
| **122** | | B | 1 / 1 | | | | | | | 0.259 | 1.5 | | |
| **123** | x | B | 8 / 8 | 0.173 | 0.0 | 0.163 | 0.3 | 0.215 | 1.6 | 0.0813 | -2.3 | | |
| **124** | | B | 6 / 6 | 0.041 | -3.0 | 0.079 | -1.9 | | | 0.208 | 0.4 | | |
| **125** | | B | 9 / 7 | 0.089 | -1.9 | 0.150 | 0.0 | | | 0.165 | -0.5 | 0.081 | -0.8 |
| **126** | | B | 6 / 6 | | | 0.169 | 0.5 | 0.172 | 0.5 | | | | |
| **127** | | B | 15 / 8 | | | 0.12 | -0.8 | 0.11 | -1.1 | 0.12 | -1.4 | 0.09 | -0.4 |
| **128** | | A | 15 / 10 | 1.437 | 29.4 | 0.162 | 0.3 | 0.156 | 0.1 | 0.205 | 0.4 | 0.096 | -0.2 |
| **3rd-14** | | B | 4 / 2 | | | | | | | | | | |
| **3rd-27** | | A | 17 / 10 | 0.11 | -1.4 | 0.13 | -0.6 | 0.184 | 0.8 | 0.19 | 0.1 | 0.148 | 1.9 |
| **3rd-83** | | A | 17 / 10 | 0.0795 | -2.2 | 0.192 | 1.1 | 0.122 | -0.8 | 0.090 | -2.1 | 0.121 | 0.8 |
| **3rd-103** | | B | 16 / 7 | FN* | -3.5 | FN* | -3.7 | 0.128 | -0.7 | | | 0.110 | 0.4 |
| **3rd-108** | | A | 16 / 9 | 0.100 | -1.7 | 0.141 | -0.3 | 0.159 | 0.1 | 0.186 | 0.0 | 0.145 | 1.8 |

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

| COMPULSORY Compounds | | | | Emamectin B1a | | Folpet | | Pymetrozine | | THPI | | Phthalimide* | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | | 0.0461 | | 0.249 | | 0.150 | | 0.590 | | | *0.134** | *0.098** |
| *CV** | | | | 21.5% | | 34.5% | | 28.2% | | 23.2% | | | *36.8%** | *36.2%** |
| MRRL [mg/kg] | | | | 0.01 | | 0.02 | | 0.02 | | 0.01 | | 0.01 | | |
| Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 10 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) |
| **93** | | A | 16 / 10 | 0.058 | 1.0 | 0.251 | 0.0 | 0.179 | 0.8 | 0.368 | *-1.5* | *0.095* | *-1.2* | *-0.1* |
| **94** | | B | 13 / 8 | 0.0369 | -0.8 | | | 0.169 | 0.5 | 0.32 | *-1.8* | *0.127* | *-0.2* | *1.2* |
| **95** | | B | 14 / 7 | 0.082 | 3.1 | | | 0.188 | 1.0 | 0.699 | *0.7* | | | |
| **96** | x | B | 11 / 6 | | | 0.274 | 0.4 | 0.162 | 0.3 | 0.576 | *-0.1* | *0.117* | *-0.5* | *0.8* |
| **97** | | A | 16 / 10 | 0.054 | 0.7 | 0.442 | 3.1 | 0.179 | 0.8 | 0.697 | *0.7* | *0.697* | *16.8* | *24.4* |
| **98** | x | B | 11 / 6 | 0.041 | -0.4 | | | 0.16 | 0.3 | | | | | |
| **99** | | A | 17 / 10 | 0.045 | -0.1 | 0.260 | 0.2 | 0.170 | 0.5 | 0.817 | *1.5* | *0.121* | *-0.4* | *0.9* |
| **100** | | A | 17 / 10 | 0.038 | -0.7 | 0.486 | 3.8 | 0.130 | -0.5 | 0.436 | *-1.0* | *0.141* | *0.2* | *1.8* |
| **101** | x | B | 7 / 5 | 0.047 | 0.1 | | | 0.257 | 2.9 | | | | | |
| **102** | | A | 17 / 10 | 0.043 | -0.3 | 0.296 | 0.8 | 0.209 | 1.6 | 0.611 | *0.1* | *0.105* | *-0.9* | *0.3* |
| **104** | x | B | 16 / 8 | 0.038 | -0.7 | 0.459 | 3.4 | 0.136 | -0.4 | 0.305 | *-1.9* | *FN* | *-3.7* | *-3.6* |
| **105** | | A | 17 / 10 | 0.034 | -1.0 | 0.23 | -0.3 | 0.11 | -1.1 | 0.83 | *1.6* | *0.17* | *1.1* | *2.9* |
| **106** | | A | 17 / 9 | 0.031 | -1.3 | 0.232 | -0.3 | 0.175 | 0.7 | 0.744 | *1.0* | *0.19* | *1.7* | *3.8* |
| **107** | | A | 17 / 10 | 0.037 | -0.8 | 0.219 | -0.5 | 0.141 | -0.2 | 0.474 | *-0.8* | *0.248* | *3.4* | *6.1* |
| **109** | | A | 17 / 9 | 0.045 | -0.1 | 0.11 | -2.2 | 0.12 | -0.8 | 0.63 | *0.3* | *0.18* | *1.4* | *3.3* |
| **110** | | A | 17 / 9 | 0.04 | -0.5 | 0.1021 | -2.4 | 0.066 | -2.2 | 1.001 | *2.8* | *0.226* | *2.7* | *5.2* |
| **111** | | A | 17 / 10 | 0.051 | 0.4 | 0.84 | 9.5 | 0.096 | -1.4 | 0.70 | *0.7* | *0.21* | *2.3* | *4.6* |
| **112** | | A | 17 / 10 | 0.087 | 3.6 | 0.458 | 3.4 | 0.109 | -1.1 | 0.247 | *-2.3* | *0.063* | *-2.1* | *-1.4* |
| **113** | | B | 7 / 5 | | | | | 0.209 | 1.6 | | | | | |
| **114** | | B | 12 / 8 | 0.0419 | -0.4 | 0.284 | 0.6 | 0.145 | -0.1 | | | | | |
| **115** | | B | 6 / 5 | | | | | 0.13 | -0.5 | | | | | |
| **117** | | A | 16 / 9 | 0.0275 | -1.6 | 0.194 | -0.9 | 0.151 | 0.0 | 0.482 | *-0.7* | *0.109* | *-0.7* | *0.4* |
| **118** | | B | 13 / 10 | 0.0460 | 0.0 | 0.267 | 0.3 | 0.0890 | -1.6 | 0.507 | *-0.6* | *0.110* | *-0.7* | *0.5* |
| **119** | | B | 1 / 1 | | | | | | | | | | | |
| **120** | | A | 15 / 10 | 0.049 | 0.3 | 0.294 | 0.7 | 0.197 | 1.3 | 0.941 | *2.4* | *0.13* | *-0.1* | *1.3* |
| **122** | | B | 1 / 1 | | | | | | | | | | | |
| **123** | x | B | 8 / 8 | | | 0.273 | 0.4 | 0.164 | 0.4 | 0.347 | *-1.6* | *0.0823* | *-1.5* | *-0.6* |
| **124** | | B | 6 / 6 | | | 0.100 | -2.4 | | | 0.566 | *-0.2* | *0.174* | *1.2* | *3.1* |
| **125** | | B | 9 / 7 | 0.053 | 0.6 | 0.131 | -1.9 | 0.071 | -2.1 | | | | | |
| **126** | | B | 6 / 6 | 0.060 | 1.2 | 0.130 | -1.9 | 0.157 | 0.2 | | | *0.187* | *1.6* | *3.6* |
| **127** | | B | 15 / 8 | 0.03 | -1.4 | | | 0.41 | 6.9 | 0.61 | *0.1* | *0.16* | *0.8* | *2.5* |
| **128** | | A | 15 / 10 | 0.042 | -0.3 | 0.587 | 5.4 | 0.152 | 0.1 | 1.437 | *5.7* | *0.291* | *4.7* | *7.9* |
| **3rd-14** | | B | 4 / 2 | 0.0372 | -0.8 | | | 0.0880 | -1.7 | | | | | |
| **3rd-27** | | A | 17 / 10 | 0.055 | 0.8 | 0.19 | -0.9 | 0.195 | 1.2 | 0.633 | *0.3* | *0.21* | *2.3* | *4.6* |
| **3rd-83** | | A | 17 / 10 | 0.0362 | -0.9 | 0.260 | 0.2 | 0.136 | -0.4 | 0.760 | *1.2* | *0.240* | *3.2* | *5.8* |
| **3rd-103** | | B | 16 / 7 | 0.069 | 2.0 | 0.153 | -1.5 | 0.191 | 1.1 | 0.793 | *1.4* | *0.150* | *0.5* | *2.1* |
| **3rd-108** | | A | 16 / 9 | 0.055 | 0.8 | | | 0.169 | 0.5 | 0.547 | *-0.3* | *0.171* | *1.1* | *3.0* |

* z scores based on different "assigned values" for phthalimide for informative purposes only.
0.134 mg/kg: robust mean of entire population from EU-/EFTA OfLs with $CV^*$ = 36.8 %
0.098 mg/kg = sum of the incurred and spiked level with $CV^*$ = 36.2 % and relative deviation to the reference value 0.098 = 51.5 %

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

| COMPULSORY Compounds | | | | Bifenazate (sum) | | Chloridazon-desphenyl | | Formetanate-HCl | | Maleic hydrazide | | Oxymatrine | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | | 0.296 | | 0.0616 | | 0.873 | | 0.544 | | 0.198 | |
| *CV** | | | | 26.0% | | 23.7% | | 25.5% | | 16.6% | | 22.4% | |
| MRRL [mg/kg] | | | | 0.03 | | 0.02 | | 0.01 | | 0.05 | | 0.01 | |
| Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 9 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) |
| **3** | x | B | 6 / 1 | | | FN | -3.5 | | | 0.330 | -1.6 | | |
| **4** | x | A | 5 / 2 | 0.266 | -0.4 | | | FN | -4.0 | | | | |
| **5** | x | B | 8 / 3 | 0.454 | 2.1 | | | 0.77 | -0.5 | 0.60 | 0.4 | | |
| **6** | | B | 4 / 1 | | | | | 0.789 | -0.4 | | | | |
| **7** | x | B | 2 / 1 | | | | | 0.91 | 0.2 | | | | |
| **8** | | B | 0 / 0 | | | | | | | | | | |
| **9** | | B | 4 / 2 | | | | | 0.854 | -0.1 | 0.506 | -0.3 | | |
| **10** | | A | 17 / 7 | 0.289 | -0.1 | 0.075 | 0.9 | 1.146 | 1.3 | 0.545 | 0.0 | 0.197 | 0.0 |
| **11** | | A | 5 / 3 | | | 0.058 | -0.2 | 0.863 | 0.0 | | | | |
| **12** | x | A | 10 / 3 | 0.206 | -1.2 | | | FN | -4.0 | 0.603 | 0.4 | | |
| **13** | | B | 10 / 5 | 0.2492 | -0.6 | 0.0360 | -1.6 | 0.6740 | -0.9 | | | 0.1492 | -1.0 |
| **15** | | A | 11 / 5 | 0.369 | 1.0 | 0.0556 | -0.4 | 0.361 | -2.3 | | | 0.207 | 0.2 |
| **16** | | B | 6 / 3 | 0.376 | 1.1 | | | 0.787 | -0.4 | 0.679 | 1.0 | | |
| **17** | x | A | 1 / 1 | | | | | 0.89 | 0.1 | | | | |
| **18** | x | A | 3 / 1 | | | | | 2.31 | 6.6 | | | | |
| **19** | x | B | 1 / 1 | 0.227 | -0.9 | | | | | | | | |
| **20** | | A | 1 / 1 | | | 0.080 | 1.2 | | | | | | |
| **21** | | B | 0 / 0 | | | | | | | | | | |
| **22** | | A | 10 / 6 | 0.183 | -1.5 | | | 0.768 | -0.5 | 0.551 | 0.1 | | |
| **23** | | B | 0 / 0 | | | | | | | | | | |
| **24** | | B | 0 / 0 | | | | | | | | | | |
| **25** | | A | 13 / 6 | 0.506 | 2.8 | 0.057 | -0.3 | 0.706 | -0.8 | 0.685 | 1.0 | 0.326 | 2.6 |
| **26** | | A | 1 / 0 | | | | | | | | | | |
| **28** | x | A | 1 / 1 | | | | | 0.759 | -0.5 | | | | |
| **29** | | B | 0 / 0 | | | | | | | | | | |
| **30** | | A | 17 / 7 | 0.148 | -2.0 | FN | -3.5 | 0.653 | -1.0 | 0.603 | 0.4 | 0.161 | -0.7 |
| **31** | | B | 0 / 0 | | | | | | | | | | |
| **32** | | B | 0 / 0 | | | | | | | | | | |
| **33** | | B | 2 / 0 | | | | | | | | | | |
| **34** | | A | 11 / 7 | | | 0.041 | -1.3 | 0.832 | -0.2 | 0.493 | -0.4 | 0.222 | 0.5 |
| **35** | x | A | 5 / 1 | | | | | 0.945 | 0.3 | | | | |
| **36** | x | A | 1 / 1 | | | | | 0.896 | 0.1 | | | | |
| **37** | | A | 7 / 1 | 0.226 | -0.9 | | | | | | | | |
| **38** | | A | 16 / 8 | 0.230 | -0.9 | 0.067 | 0.4 | 1.26 | 1.8 | 0.427 | -0.9 | 0.179 | -0.4 |
| **39** | | A | 7 / 3 | 0.316 | 0.3 | | | 0.729 | -0.7 | 0.478 | -0.5 | | |
| **40** | | A | 10 / 5 | 0.329 | 0.4 | | | 0.717 | -0.7 | 0.477 | -0.5 | 0.193 | -0.1 |
| **41** | | A | 9 / 5 | 0.26 | -0.5 | 0.005[FR] | -3.7 | 2.88 | 9.2 | 0.55 | 0.0 | 0.17 | -0.6 |
| **42** | | A | 15 / 7 | 0.37 | 1.0 | 0.053 | -0.6 | 0.71 | -0.7 | 0.52 | -0.2 | 0.16 | -0.8 |
| **43** | x | A | 3 / 1 | | | | | | | 0.520 | -0.2 | | |
| **44** | | A | 11 / 8 | 0.267 | -0.4 | 0.067 | 0.4 | 0.8 | -0.3 | 0.56 | 0.1 | 0.15 | -1.0 |
| **45** | | A | 14 / 6 | 0.260 | -0.5 | FN | -3.4 | 0.848 | -0.1 | 0.617 | 0.5 | 0.216 | 0.4 |

[FR]: false reporting, since the reported result < lab's reporting limit and in practice should not be reported.

| | COMPULSORY Compounds | | | | ETU # | | | | | 2,4-DNOP (free phenol) | Meptyl-dinocap | Meptyl-dinocap (sum) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Reference value [mg/kg] | | | | 0.0629 | | | Possible range of reference value considering uncertainty of robust mean | | 0.056* | 0.100* | 0.169* |
| | | | | CV* | 33.1% | | | Lower value: | Upper value: | 45.1% | 54.6% | 50.5% |
| | | | | MRRL [mg/kg] | 0.01 | | | 0.0568 | 0.0690 | 0.01 | 0.02 | 0.01 |
| | Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 9 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | z' score (FFP-RSD = 25 %) | z Score range considering uncertainty of robust mean | | Conc. [mg/kg] | Conc. [mg/kg] | Conc. [mg/kg] |
| | 3 | x | B | 6 / 1 | | | | | | | | |
| | 4 | x | A | 5 / 2 | | | | | | | 0.0842 | |
| | 5 | x | B | 8 / 3 | | | | | | | | |
| | 6 | | B | 4 / 1 | | | | | | | | |
| | 7 | x | B | 2 / 1 | | | | | | | | |
| | 8 | | B | 0 / 0 | | | | | | | | |
| | 9 | | B | 4 / 2 | | | | | | | | |
| | 10 | | A | 17 / 7 | 0.104 | 2.6 | 2.4 | 2.2 | 3.0 | FN | FN | 0.162 |
| | 11 | | A | 5 / 3 | | | | | | | | 0.082 |
| | 12 | x | A | 10 / 3 | FN | -3.4 | -3.1 | -3.8 | -3.0 | | 0.0721 | |
| | 13 | | B | 10 / 5 | | | | | | | 0.7500 | |
| | 15 | | A | 11 / 5 | 0.0677 | 0.3 | 0.3 | -0.1 | 0.7 | | | |
| | 16 | | B | 6 / 3 | | | | | | | | |
| | 17 | x | A | 1 / 1 | | | | | | | | |
| | 18 | x | A | 3 / 1 | | | | | | | | |
| | 19 | x | B | 1 / 1 | | | | | | | | |
| | 20 | | A | 1 / 1 | | | | | | | | |
| | 21 | | B | 0 / 0 | | | | | | | | |
| | 22 | | A | 10 / 6 | | | | | | 0.0321 | 0.0621 | 0.105 |
| | 23 | | B | 0 / 0 | | | | | | | | |
| | 24 | | B | 0 / 0 | | | | | | | | |
| | 25 | | A | 13 / 6 | | | | | | 0.164 | | |
| | 26 | | A | 1 / 0 | | | | | | | | |
| | 28 | x | A | 1 / 1 | | | | | | | | |
| | 29 | | B | 0 / 0 | | | | | | | | |
| | 30 | | A | 17 / 7 | 0.037 | -1.7 | -1.5 | -2.0 | -1.3 | 0.049 | FN | 0.112 |
| | 31 | | B | 0 / 0 | | | | | | | | |
| | 32 | | B | 0 / 0 | | | | | | | | |
| | 33 | | B | 2 / 0 | | | | | | | | |
| | 34 | | A | 11 / 7 | | | | | | 0.064 | 0.088 | 0.147 |
| | 35 | x | A | 5 / 1 | | | | | | | | |
| | 36 | x | A | 1 / 1 | | | | | | | | |
| | 37 | | A | 7 / 1 | | | | | | | | |
| | 38 | | A | 16 / 8 | 0.056 | -0.4 | -0.4 | -0.8 | 0.0 | 0.174 | | 0.385 |
| | 39 | | A | 7 / 3 | | | | | | | | |
| | 40 | | A | 10 / 5 | | | | | | | 0.329 | |
| | 41 | | A | 9 / 5 | | | | | | | | |
| | 42 | | A | 15 / 7 | 0.080 | 1.1 | 1.0 | 0.7 | 1.5 | | 0.28 | |
| | 43 | x | A | 3 / 1 | | | | | | | | |
| | 44 | | A | 11 / 8 | | | | | | 0.03 | 0.1 | 0.13 |
| | 45 | | A | 14 / 6 | 0.102 | 2.5 | 2.3 | 2.1 | 2.9 | | 0.156 | |

# The uncertainty of the robust mean of ETU is larger than what is required for being regarded as assigned value, the z scores are therefore calculated for informative purposes only. Next to the z score based on the robust mean, additionally, z' scores and the range of z scores (considering the uncertainty range of the robust mean) were also calculated.

* For informative purposes, calculations were made where the spiking levels were designated as "assigned values" for 2,4-DNOP, meptyldinocap and meptyldinocap (sum). After consultation with the Scientific Committee, no z scores for these three analytes were calculated.

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

| COMPULSORY Compounds | | | | Bifenazate (sum) | | Chloridazon-desphenyl | | Formetanate-HCl | | Maleic hydrazide | | Oxymatrine | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | | 0.296 | | 0.0616 | | 0.873 | | 0.544 | | 0.198 | |
| *CV** | | | | 26.0% | | 23.7% | | 25.5% | | 16.6% | | 22.4% | |
| MRRL [mg/kg] | | | | 0.03 | | 0.02 | | 0.01 | | 0.05 | | 0.01 | |
| Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 9 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) |
| **46** | | B | 1 / 1 | | | | | 0.642 | -1.1 | | | | |
| **47** | | A | 16 / 8 | 0.313 | 0.2 | 0.074 | 0.9 | 0.831 | -0.2 | 0.463 | -0.6 | 0.308 | 2.2 |
| **48** | | A | 12 / 5 | 0.410 | 1.5 | 0.060 | -0.1 | 6.0 | 23.5 | 0.444 | -0.7 | 0.150 | -1.0 |
| **49** | x | B | 0 / 0 | | | | | | | | | | |
| **50** | x | A | 16 / 7 | 0.223 | -1.0 | 0.054 | -0.5 | 0.868 | 0.0 | 0.733 | 1.4 | 0.169 | -0.6 |
| **51** | | A | 12 / 4 | | | 0.061 | 0.0 | 0.860 | -0.1 | | | 0.225 | 0.5 |
| **52** | | B | 4 / 2 | | | 0.668 | 39.8 | 0.929 | 0.3 | | | | |
| **53** | | A | 9 / 4 | 0.319 | 0.3 | | | 0.732 | -0.6 | 0.706 | 1.2 | 0.293 | 1.9 |
| **54** | | A | 8 / 5 | 0.302 | 0.1 | | | 1.344 | 2.2 | 0.430 | -0.8 | 0.191 | -0.1 |
| **55** | x | A | 5 / 3 | 0.337 | 0.6 | | | 0.778 | -0.4 | 0.496 | -0.4 | | |
| **56** | x | B | 3 / 1 | | | | | 0.766 | -0.5 | | | | |
| **57** | | A | 8 / 4 | 0.842 | 7.4 | | | 40.6 | 182.0 | | | | |
| **58** | x | A | 5 / 4 | 0.272 | -0.3 | 0.052 | -0.6 | 1.02 | 0.7 | 0.518 | -0.2 | | |
| **59** | | B | 0 / 0 | | | | | | | | | | |
| **60** | x | B | 2 / 2 | 0.186 | -1.5 | | | 0.850 | -0.1 | | | | |
| **61** | | A | 8 / 2 | | | | | 1.79 | 4.2 | | | 0.230 | 0.6 |
| **62** | | A | 17 / 9 | 0.398 | 1.4 | 0.052 | -0.6 | 0.677 | -0.9 | 0.539 | 0.0 | 0.177 | -0.4 |
| **63** | x | B | 1 / 1 | | | | | 0.55 | -1.5 | | | | |
| **64** | x | A | 3 / 1 | | | | | 1.48 | 2.8 | | | | |
| **66** | | B | 1 / 1 | | | | | 0.344 | -2.4 | | | | |
| **67** | | A | 9 / 3 | | | | | | | 0.561 | 0.1 | 0.198 | 0.0 |
| **68** | x | A | 3 / 2 | 0.303 | 0.1 | | | | | 0.623 | 0.6 | | |
| **69** | | A | 15 / 8 | 0.360 | 0.9 | 0.077 | 1.0 | 0.767 | -0.5 | 0.544 | 0.0 | 0.165 | -0.7 |
| **70** | | A | 6 / 3 | 0.358 | 0.8 | | | 1.79 | 4.2 | 0.554 | 0.1 | | |
| **71** | | B | 4 / 1 | | | | | FN | -4.0 | | | | |
| **72** | x | A | 7 / 2 | 0.402 | 1.4 | | | 0.777 | -0.4 | | | | |
| **73** | | B | 0 / 0 | | | | | | | | | | |
| **74** | | A | 12 / 5 | 0.47 | 2.4 | | | 0.63 | -1.1 | 0.48 | -0.5 | 0.13 | -1.4 |
| **75** | | B | 4 / 2 | | | | | 0.374 | -2.3 | 0.83 | 2.1 | | |
| **76** | | B | 0 / 0 | | | | | | | | | | |
| **77** | | A | 1 / 1 | | | | | 1.25 | 1.7 | | | | |
| **78** | x | A | 4 / 2 | | | | | 1.037 | 0.8 | | | | |
| **79** | | B | 2 / 1 | | | | | 1.44 | 2.6 | | | | |
| **80** | | B | 0 / 0 | | | | | | | | | | |
| **81** | | B | 0 / 0 | | | | | | | | | | |
| **82** | | A | 9 / 5 | 0.346 | 0.7 | 0.067 | 0.4 | 1.361 | 2.2 | 0.518 | -0.2 | 0.209 | 0.2 |
| **84** | x | B | 3 / 1 | | | | | 1.294 | 1.9 | | | | |
| **85** | | A | 11 / 5 | 0.301 | 0.1 | | | 1.64 | 3.5 | 0.690 | 1.1 | 0.200 | 0.0 |
| **86** | | B | 0 / 0 | | | | | | | | | | |
| **87** | | B | 4 / 1 | | | 0.0665 | 0.4 | | | | | | |
| **88** | | A | 5 / 4 | 0.272 | -0.3 | | | 3.15 | 10.4 | 0.620 | 0.6 | | |
| **89** | | A | 6 / 2 | 0.246 | -0.7 | | | | | 0.602 | 0.4 | | |

| | COMPULSORY Compounds | | | | ETU # | | | | | 2,4-DNOP (free phenol) | Meptyl-dinocap | Meptyl-dinocap (sum) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Reference value [mg/kg] | | | | 0.0629 | | | Possible range of reference value considering uncertainty of robust mean | | 0.056* | 0.100* | 0.169* |
| | CV* | | | | 33.1% | | | Lower value: | Upper value: | 45.1% | 54.6% | 50.5% |
| | MRRL [mg/kg] | | | | 0.01 | | | 0.0568 | 0.0690 | 0.01 | 0.02 | 0.01 |
| | Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 9 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | z' score (FFP-RSD = 25 %) | z Score range considering uncertainty of robust mean | | Conc. [mg/kg] | Conc. [mg/kg] | Conc. [mg/kg] |
| | **46** | | B | 1 / 1 | | | | | | | | |
| | **47** | | A | 16 / 8 | 0.039 | *-1.5* | *-1.4* | *-1.9* | *-1.1* | 0.077 | | 0.159 |
| | **48** | | A | 12 / 5 | | | | | | | | |
| | **49** | x | B | 0 / 0 | | | | | | | | |
| | **50** | x | A | 16 / 7 | 0.058 | *-0.3* | *-0.3* | *-0.7* | *0.1* | | FN | 0.332 |
| | **51** | | A | 12 / 4 | 0.070 | *0.4* | *0.4* | *0.1* | *0.8* | | | |
| | **52** | | B | 4 / 2 | | | | | | | | |
| | **53** | | A | 9 / 4 | | | | | | | | |
| | **54** | | A | 8 / 5 | | | | | | | 0.613 | |
| | **55** | x | A | 5 / 3 | | | | | | | | |
| | **56** | x | B | 3 / 1 | | | | | | | | |
| | **57** | | A | 8 / 4 | 0.0295 | *-2.1* | *-2.0* | *-2.5* | *-1.7* | | 1.91 | |
| | **58** | x | A | 5 / 4 | | | | | | | | |
| | **59** | | B | 0 / 0 | | | | | | | | |
| | **60** | x | B | 2 / 2 | | | | | | | | |
| | **61** | | A | 8 / 2 | | | | | | | | |
| | **62** | | A | 17 / 9 | 0.069 | *0.4* | *0.4* | *0.0* | *0.8* | 0.081 | 0.093 | 0.202 |
| | **63** | x | B | 1 / 1 | | | | | | | | |
| | **64** | x | A | 3 / 1 | | | | | | | | |
| | **66** | | B | 1 / 1 | | | | | | | | |
| | **67** | | A | 9 / 3 | 0.066 | *0.2* | *0.2* | *-0.2* | *0.6* | | | |
| | **68** | x | A | 3 / 2 | | | | | | | | |
| | **69** | | A | 15 / 8 | | | | | | 0.054 | 0.093 | 0.128 |
| | **70** | | A | 6 / 3 | | | | | | | | |
| | **71** | | B | 4 / 1 | | | | | | | 0.213 | |
| | **72** | x | A | 7 / 2 | | | | | | | | |
| | **73** | | B | 0 / 0 | | | | | | | | |
| | **74** | | A | 12 / 5 | | | | | | | 0.48 | |
| | **75** | | B | 4 / 2 | | | | | | | | |
| | **76** | | B | 0 / 0 | | | | | | | | |
| | **77** | | A | 1 / 1 | | | | | | | | |
| | **78** | x | A | 4 / 2 | | | | | | | | 0.111 |
| | **79** | | B | 2 / 1 | | | | | | | | |
| | **80** | | B | 0 / 0 | | | | | | | | |
| | **81** | | B | 0 / 0 | | | | | | | | |
| | **82** | | A | 9 / 5 | | | | | | | | |
| | **84** | x | B | 3 / 1 | | | | | | | | |
| | **85** | | A | 11 / 5 | | | | | | | | 0.208 |
| | **86** | | B | 0 / 0 | | | | | | | | |
| | **87** | | B | 4 / 1 | | | | | | | | |
| | **88** | | A | 5 / 4 | | | | | | | 3.55 | |
| | **89** | | A | 6 / 2 | | | | | | | | |

# The uncertainty of the robust mean of ETU is larger than what is required for being regarded as assigned value, the z scores are therefore calculated for informative purposes only. Next to the z score based on the robust mean, additionally, z' scores and the range of z scores (considering the uncertainty range of the robust mean) were also calculated.

* For informative purposes, calculations were made where the spiking levels were designated as "assigned values" for 2,4-DNOP, meptyldinocap and meptyldinocap (sum). After consultation with the Scientific Committee, no z scores for these three analytes were calculated.

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

| COMPULSORY Compounds | | | | Bifenazate (sum) | | Chloridazon-desphenyl | | Formetanate-HCl | | Maleic hydrazide | | Oxymatrine | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | | 0.296 | | 0.0616 | | 0.873 | | 0.544 | | 0.198 | |
| *CV** | | | | 26.0% | | 23.7% | | 25.5% | | 16.6% | | 22.4% | |
| MRRL [mg/kg] | | | | 0.03 | | 0.02 | | 0.01 | | 0.05 | | 0.01 | |
| Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 9 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) |
| **90** | | B | 6 / 2 | 0.744 | 6.1 | | | 2.230 | 6.2 | | | | |
| **91** | | A | 4 / 2 | | | | | | | 0.403 | -1.0 | | |
| **92** | x | A | 3 / 1 | | | | | 0.653 | -1.0 | | | | |
| **93** | | A | 2 / 1 | 0.191 | -1.4 | | | | | | | | |
| **94** | | B | 0 / 0 | | | | | | | | | | |
| **95** | | B | 7 / 3 | | | 0.056 | -0.3 | 1.02 | 0.7 | | | 0.226 | 0.6 |
| **96** | x | B | 5 / 1 | | | | | 0.860 | -0.1 | | | | |
| **97** | | A | 8 / 2 | | | | | | | 0.658 | 0.8 | | |
| **98** | x | B | 7 / 2 | 0.26 | -0.5 | | | | | 0.54 | 0.0 | | |
| **99** | | A | 7 / 3 | 0.320 | 0.3 | FN | -3.3 | 1.100 | 1.0 | | | 0.200 | 0.0 |
| **100** | | A | 15 / 9 | 0.227 | -0.9 | 0.038 | -1.5 | 0.767 | -0.5 | 0.452 | -0.7 | 0.132 | -1.3 |
| **101** | x | B | 2 / 2 | 0.318 | 0.3 | | | 1.64 | 3.5 | | | | |
| **102** | | A | 14 / 7 | 0.335 | 0.5 | 0.088 | 1.8 | 0.881 | 0.0 | | | 0.156 | -0.8 |
| **104** | x | B | 10 / 3 | | | 0.08 | 1.2 | 0.879 | 0.0 | | | 0.253 | 1.1 |
| **105** | | A | 9 / 4 | 0.25 | -0.6 | | | 2.21 | 6.1 | 0.53 | -0.1 | | |
| **106** | | A | 5 / 3 | 0.318 | 0.3 | | | 0.746 | -0.6 | | | | |
| **107** | | A | 9 / 3 | 0.408 | 1.5 | | | | | 0.411 | -1.0 | 0.179 | -0.4 |
| **109** | | A | 4 / 3 | 0.26 | -0.5 | | | 0.78 | -0.4 | FN | -3.6 | | |
| **110** | | A | 14 / 6 | 0.457 | 2.2 | FN | -3.3 | 0.643 | -1.1 | 0.54 | 0.0 | 0.183 | -0.3 |
| **111** | | A | 11 / 5 | 0.29 | -0.1 | | | 0.635 | -1.1 | 0.548 | 0.0 | 0.197 | 0.0 |
| **112** | | A | 14 / 4 | 0.182 | -1.5 | | | FN | -4.0 | 0.498 | -0.3 | 0.253 | 1.1 |
| **113** | | B | 1 / 0 | | | | | FN | -4.0 | | | | |
| **114** | | B | 2 / 1 | 0.237 | -0.8 | FN | -3.5 | | | | | | |
| **115** | | B | 4 / 1 | | | | | 0.82 | -0.2 | | | | |
| **117** | | A | 10 / 5 | 0.319 | 0.3 | | | 0.928 | 0.3 | | | | |
| **118** | | B | 3 / 2 | 0.271 | -0.3 | | | 1.00 | 0.6 | | | | |
| **119** | | B | 0 / 0 | | | | | | | | | | |
| **120** | | A | 9 / 4 | 0.320 | 0.3 | FN | -3.3 | 1.113 | 1.1 | | | 0.274 | 1.5 |
| **122** | | B | 0 / 0 | | | | | | | | | | |
| **123** | x | B | 0 / 0 | | | | | | | | | | |
| **124** | | B | 0 / 0 | | | | | | | | | | |
| **125** | | B | 3 / 1 | 0.279 | -0.2 | | | | | | | | |
| **126** | | B | 4 / 2 | 0.21 | -1.2 | | | 1.02 | 0.7 | | | | |
| **127** | | B | 9 / 5 | 0.28 | -0.2 | | | 0.85 | -0.1 | 0.51 | -0.3 | 0.24 | 0.8 |
| **128** | | A | 3 / 2 | | | | | 0.883 | 0.0 | | | | |
| **3rd-14** | | B | 2 / 0 | | | | | | | | | | |
| **3rd-27** | | A | 10 / 4 | 0.501 | 2.8 | 0.105 | 2.9 | 0.94 | 0.3 | 0.483 | -0.4 | | |
| **3rd-83** | | A | 10 / 3 | | | | | FN | -4.0 | 0.425 | -0.9 | 0.156 | -0.8 |
| **3rd-103** | | B | 0 / 0 | | | | | | | | | | |
| **3rd-108** | | A | 8 / 2 | | | | | 0.805 | -0.3 | | | 0.133 | -1.3 |

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

| | COMPULSORY Compounds | | | | ETU # | | | | | 2,4-DNOP (free phenol) | Meptyl-dinocap | Meptyl-dinocap (sum) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Reference value [mg/kg] | | | | 0.0629 | | | Possible range of reference value considering uncertainty of robust mean | | 0.056* | 0.100* | 0.169* |
| | | | | CV* | 33.1% | | | Lower value: | Upper value: | 45.1% | 54.6% | 50.5% |
| | | | | MRRL [mg/kg] | 0.01 | | | 0.0568 | 0.0690 | 0.01 | 0.02 | 0.01 |
| | Lab code SRM17- | NRL-SRM | Cat | Analysed / corr. found, max. 17 / 9 | Conc. [mg/kg] | z Score (FFP-RSD = 25 %) | z' score (FFP-RSD = 25 %) | z Score range considering uncertainty of robust mean | | Conc. [mg/kg] | Conc. [mg/kg] | Conc. [mg/kg] |
| | **90** | | B | 6 / 2 | | | | | | | | |
| | **91** | | A | 4 / 2 | | | | | | | 0.190 | |
| | **92** | x | A | 3 / 1 | | | | | | | | |
| | **93** | | A | 2 / 1 | | | | | | | | |
| | **94** | | B | 0 / 0 | | | | | | | | |
| | **95** | | B | 7 / 3 | | | | | | | | |
| | **96** | x | B | 5 / 1 | | | | | | | | |
| | **97** | | A | 8 / 2 | | | | | | | 0.149 | |
| | **98** | x | B | 7 / 2 | | | | | | | | |
| | **99** | | A | 7 / 3 | | | | | | | | |
| | **100** | | A | 15 / 9 | 0.069 | *0.4* | *0.4* | *0.0* | *0.8* | 0.058 | 0.186 | 0.257 |
| | **101** | x | B | 2 / 2 | | | | | | | | |
| | **102** | | A | 14 / 7 | | | | | | 0.047 | 0.053 | 0.111 |
| | **104** | x | B | 10 / 3 | | | | | | | | |
| | **105** | | A | 9 / 4 | 0.053 | *-0.6* | *-0.6* | *-1.0* | *-0.2* | | | |
| | **106** | | A | 5 / 3 | | | | | | | 0.764 | |
| | **107** | | A | 9 / 3 | | | | | | | | |
| | **109** | | A | 4 / 3 | | | | | | | 3.5 | |
| | **110** | | A | 14 / 6 | 0.043 | *-1.3* | *-1.2* | *-1.7* | *-0.9* | 0.044 | | |
| | **111** | | A | 11 / 5 | 0.085 | *1.4* | *1.3* | *1.0* | *1.8* | | | |
| | **112** | | A | 14 / 4 | 0.064 | *0.1* | *0.1* | *-0.3* | *0.5* | FN | | |
| | **113** | | B | 1 / 0 | | | | | | | | |
| | **114** | | B | 2 / 1 | | | | | | | | |
| | **115** | | B | 4 / 1 | | | | | | | | |
| | **117** | | A | 10 / 5 | | | | | | 0.122 | 3.23 | 3.38 |
| | **118** | | B | 3 / 2 | | | | | | | | |
| | **119** | | B | 0 / 0 | | | | | | | | |
| | **120** | | A | 9 / 4 | | | | | | | 1.274 | |
| | **122** | | B | 0 / 0 | | | | | | | | |
| | **123** | x | B | 0 / 0 | | | | | | | | |
| | **124** | | B | 0 / 0 | | | | | | | | |
| | **125** | | B | 3 / 1 | | | | | | | | |
| | **126** | | B | 4 / 2 | | | | | | | | |
| | **127** | | B | 9 / 5 | | | | | | | 0.53 | |
| | **128** | | A | 3 / 2 | 0.055 | *-0.5* | *-0.5* | *-0.9* | *-0.1* | | | |
| | **3rd-14** | | B | 2 / 0 | | | | | | | | |
| | **3rd-27** | | A | 10 / 4 | | | | | | | | |
| | **3rd-83** | | A | 10 / 3 | 0.0730 | *0.6* | *0.6* | *0.3* | *1.0* | | | |
| | **3rd-103** | | B | 0 / 0 | | | | | | | | |
| | **3rd-108** | | A | 8 / 2 | | | | | | | | |

# The uncertainty of the robust mean of ETU is larger than what is required for being regarded as assigned value, the z scores are therefore calculated for informative purposes only. Next to the z score based on the robust mean, additionally, z' scores and the range of z scores (considering the uncertainty range of the robust mean) were also calculated.

* For informative purposes, calculations were made where the spiking levels were designated as "assigned values" for 2,4-DNOP, meptyldinocap and meptyldinocap (sum). After consultation with the Scientific Committee, no z scores for these three analytes were calculated.

**Table 4-10:** Category A laboratories in EUPT-SRM17, ordered by lab-codes. For the AAZ calculation any z scores > 5 were set at 5. Z-scores for phthalimide based on two different assigned values (for details please see **Section 4.2.1**) are calculated for informative purposes only and excluded in the AAZ calculation.

| COMPULSORY Compounds | | | Captan | Chloro-thalonil | Cyro-mazine | DTCs (expr. as $CS_2$) | Dodine | Ema-mectin B1a | Folpet | Pyme-trozine | THPI | Phthalimide | | AAZ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | 0.174 | 0.151 | 0.154 | 0.187 | 0.100 | 0.0461 | 0.249 | 0.150 | 0.590 | *0.134* | *0.098* | |
| *CV** | | | 32.9% | 24.4% | 20.3% | 28.4% | 23.1% | 21.5% | 34.5% | 28.2% | 23.2% | *36.8%* | *36.2%*[1)] | |
| MRRL [mg/kg] | | | 0.02 | 0.01 | 0.02 | 0.02 | 0.01 | 0.01 | 0.02 | 0.02 | 0.01 | 0.01 | | |
| Lab code SRM17- | NRL-SRM | Analysed / corr. found, max. 17 / 10 | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | | |
| **4** | x | 17 / 10 | 0.6 | 0.3 | 0.6 | -0.6 | -0.1 | -0.6 | 1.0 | 0.4 | 0.7 | *1.7* | *3.8* | 0.5 |
| **10** | | 17 / 10 | -2.2 | -0.3 | -0.2 | 9.0 | 0.4 | 0.2 | 0.0 | 0.1 | 0.8 | *1.9* | *4.0* | 1.0 |
| **11** | | 16 / 10 | 0.8 | 0.4 | 0.5 | 0.7 | -1.1 | 0.2 | 1.0 | 0.9 | 0.6 | *1.0* | *2.9* | 0.7 |
| **12** | x | 16 / 9 | -1.4 | -1.0 | 0.8 | 3.5 | -1.0 | 0.8 | -1.7 | -1.2 | | *1.0* | *2.9* | 1.4 |
| **15** | | 17 / 10 | -0.5 | -0.6 | 0.3 | -2.5 | -0.6 | -1.8 | -0.7 | 0.6 | 0.0 | *1.1* | *2.9* | 0.8 |
| **17** | x | 15 / 10 | 2.7 | -0.3 | 3.5 | -0.4 | 0.8 | 3.4 | 1.9 | 1.3 | -1.3 | *0.5* | *2.1* | 1.7 |
| **18** | x | 17 / 10 | 0.2 | -0.1 | 0.0 | -0.9 | -0.8 | -0.3 | -0.5 | 0.5 | 1.4 | *-0.3* | *1.1* | 0.5 |
| **20** | | 16 / 9 | 1.1 | -1.1 | -0.2 | 2.6 | 2.2 | -0.6 | 1.5 | 0.9 | -0.4 | *-3.7* | *-3.6* | 1.2 |
| **22** | | 17 / 10 | -0.5 | -0.3 | -0.1 | -0.4 | -0.4 | -0.9 | -0.6 | 0.1 | 0.3 | *-1.4* | *-0.5* | 0.4 |
| **25** | | 17 / 10 | -1.5 | 1.0 | 0.2 | -0.5 | 1.9 | 15.0 | -1.3 | 0.1 | -0.1 | *-0.1* | *1.3* | 1.3 |
| **26** | | 16 / 9 | 0.4 | -0.8 | -0.6 | -0.6 | | 0.3 | -1.2 | 0.5 | -3.1 | *-0.8* | *0.4* | 0.9 |
| **28** | x | 15 / 9 | -0.5 | -1.0 | 0.2 | | 0.6 | 0.0 | -0.4 | 1.0 | 0.0 | *-1.7* | *-0.9* | 0.5 |
| **30** | | 17 / 10 | -3.8 | -1.0 | -0.6 | 0.0 | -0.1 | -0.5 | -2.1 | -1.2 | -0.1 | *-1.6* | *-0.8* | 1.0 |
| **34** | | 17 / 10 | -0.9 | -0.5 | -1.2 | -0.1 | -0.5 | -1.6 | 0.8 | 0.2 | -0.5 | *-2.4* | *-1.8* | 0.7 |
| **35** | x | 17 / 10 | 0.6 | 0.7 | 1.1 | 0.3 | -0.6 | -0.1 | 0.7 | 0.5 | 0.8 | *0.5* | *2.2* | 0.6 |
| **36** | x | 17 / 10 | 0.5 | -1.3 | 0.5 | 0.1 | -1.3 | 0.0 | 0.3 | 0.1 | 0.1 | *1.5* | *3.6* | 0.5 |
| **37** | | 17 / 9 | -0.3 | -1.2 | 0.3 | -3.8 | 0.3 | -0.8 | -0.6 | -0.2 | 0.4 | *-1.4* | *-0.4* | 0.9 |
| **38** | | 17 / 10 | -1.0 | -0.1 | 0.2 | 0.3 | 0.5 | -0.1 | -0.4 | -0.5 | -0.2 | *1.1* | *2.9* | 0.4 |
| **39** | | 17 / 10 | 2.4 | 1.1 | 0.6 | 0.5 | 0.0 | 0.4 | 1.1 | 1.9 | -0.9 | *-1.0* | *0.2* | 1.0 |
| **40** | | 17 / 10 | -3.4 | -1.0 | -1.7 | -0.8 | 0.0 | 0.3 | -1.5 | -1.0 | -1.9 | *-1.1* | *0.0* | 1.3 |
| **41** | | 17 / 10 | -2.5 | -0.8 | -2.1 | 0.3 | 1.5 | 0.2 | -0.6 | -1.9 | 1.2 | *0.5* | *2.1* | 1.2 |
| **42** | | 17 / 10 | 46.6 | 0.8 | 1.2 | 1.1 | -0.6 | 1.1 | -0.5 | -0.3 | 0.4 | *0.5* | *2.1* | 1.2 |
| **43** | x | 16 / 9 | 0.9 | -1.6 | -0.3 | 8.8 | 0.3 | | -0.4 | 0.2 | -1.0 | *-2.0* | *-1.3* | 1.2 |
| **44** | | 17 / 9 | 1.1 | 1.8 | -0.2 | 0.3 | 1.2 | 0.1 | -1.3 | -0.2 | -0.4 | *-3.7* | *-3.6* | 0.7 |
| **45** | | 17 / 10 | 0.0 | 1.6 | 0.3 | -1.2 | 1.3 | 1.0 | -0.1 | 1.0 | -0.4 | *-0.8* | *0.3* | 0.8 |
| **47** | | 17 / 10 | -0.6 | -0.3 | 1.0 | -0.6 | -0.4 | -1.5 | 0.5 | 1.4 | 0.3 | *-0.6* | *0.6* | 0.7 |
| **48** | | 17 / 10 | 0.2 | 2.5 | 0.2 | 1.3 | -0.6 | 1.0 | 0.1 | 0.1 | 0.7 | *1.4* | *3.3* | 0.7 |
| **50** | x | 17 / 10 | 0.3 | -0.2 | 0.3 | 2.4 | -0.2 | 1.5 | 0.0 | -0.6 | -0.9 | *-2.0* | *-1.3* | 0.7 |
| **51** | | 17 / 10 | 0.3 | 0.5 | 0.9 | 1.1 | -0.3 | 0.3 | 0.7 | 1.0 | 0.8 | *0.4* | *2.0* | 0.7 |
| **53** | | 17 / 10 | 1.1 | 0.5 | 1.2 | 0.6 | -0.4 | 0.9 | 0.6 | 0.1 | 0.3 | *-1.1* | *-0.1* | 0.6 |
| **54** | | 16 / 10 | 0.5 | 0.4 | 0.2 | -0.7 | -1.7 | -0.1 | 1.3 | 0.2 | -0.4 | *-1.4* | *-0.5* | 0.6 |
| **55** | x | 16 / 9 | 0.9 | 0.4 | -0.7 | -0.3 | 0.3 | 0.3 | 1.3 | 0.3 | | *-1.9* | *-1.2* | 0.6 |
| **57** | | 15 / 9 | | -3.4 | -2.4 | -1.3 | -0.4 | -0.9 | -3.4 | -1.8 | -0.3 | *-1.7* | *-0.8* | 1.7 |
| **58** | x | 17 / 10 | 0.9 | -0.6 | 0.0 | 0.7 | -1.2 | -0.3 | 0.5 | 0.5 | 0.7 | *-1.6* | *-0.7* | 0.6 |
| **61** | | 17 / 9 | 1.1 | -3.7 | -0.6 | 3.7 | -0.5 | -0.6 | 1.5 | 1.2 | -1.1 | *-0.8* | *0.4* | 1.6 |
| **62** | | 17 / 10 | 0.1 | 0.9 | -0.4 | 3.9 | 0.9 | 0.0 | 0.9 | -0.4 | -0.8 | *-1.2* | *-0.2* | 0.9 |
| **64** | x | 17 / 10 | 0.3 | 0.1 | -0.8 | -3.0 | -0.4 | -0.7 | 0.6 | 0.8 | 0.5 | *-1.6* | *-0.7* | 0.8 |
| **67** | | 16 / 10 | -1.4 | -0.5 | 0.3 | 1.7 | 0.8 | 0.1 | -1.1 | 0.8 | 0.3 | *-0.8* | *0.3* | 0.8 |
| **68** | x | 17 / 10 | -0.3 | 1.3 | 1.3 | 0.0 | -0.7 | -0.6 | 0.7 | -0.7 | 0.9 | *-0.4* | *0.9* | 0.7 |
| **69** | | 17 / 10 | 0.4 | 0.2 | 0.3 | -2.4 | 0.5 | 0.0 | 0.8 | -0.4 | -0.3 | *-0.3* | *1.1* | 0.6 |
| **70** | | 16 / 9 | 1.6 | 1.2 | 0.4 | | 0.2 | -0.5 | 4.8 | -0.1 | -0.2 | *0.3* | *1.9* | 1.1 |
| **72** | x | 17 / 10 | -0.9 | 0.3 | 0.1 | -0.1 | 0.3 | 0.7 | -0.4 | 0.8 | 0.3 | *4.9* | *8.2* | 0.4 |
| **74** | | 17 / 10 | -0.5 | -0.8 | -2.3 | 0.3 | -1.2 | -0.9 | -0.6 | -2.1 | 1.1 | *0.8* | *2.5* | 1.1 |
| **77** | | 17 / 10 | -0.7 | -0.3 | -0.3 | 0.5 | -0.5 | 0.4 | 0.3 | -1.3 | -0.2 | *2.0* | *4.2* | 0.5 |
| **78** | x | 17 / 10 | 0.3 | -0.9 | 1.0 | 0.3 | 0.0 | -0.2 | -0.3 | 1.7 | -0.3 | *-1.6* | *-0.7* | 0.6 |
| **82** | | 17 / 10 | -1.2 | -0.1 | 0.1 | 0.2 | 0.6 | 1.5 | -0.1 | 0.8 | -0.3 | *1.1* | *3.0* | 0.5 |

1) calculated using robust approach; the relative deviation to the reference value 0.098 = 51.5 %

**Table 4-10 (cont.):** Category A laboratories in EUPT-SRM17, ordered by lab-codes. For the AAZ calculation any z scores > 5 were set at 5. Z-scores for phthalimide based on two different assigned values (for details please see Section 4.2.1) are calculated for informative purposes only and excluded in the AAZ calculation.

| COMPULSORY Compounds | | | Captan | Chloro-thalonil | Cyro-mazine | DTCs (expr. as $CS_2$) | Dodine | Ema-mectin B1a | Folpet | Pyme-trozine | THPI | Phthalimide | | AAZ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | 0.174 | 0.151 | 0.154 | 0.187 | 0.100 | 0.0461 | 0.249 | 0.150 | 0.590 | *0.134* | *0.098* | |
| *CV** | | | 32.9% | 24.4% | 20.3% | 28.4% | 23.1% | 21.5% | 34.5% | 28.2% | 23.2% | *36.8%* | *36.2%[1)]* | |
| MRRL [mg/kg] | | | 0.02 | 0.01 | 0.02 | 0.02 | 0.01 | 0.01 | 0.02 | 0.02 | 0.01 | 0.01 | | |
| Lab code SRM17- | NRL-SRM | Analysed / corr. found, max. 17 / 10 | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | | |
| **85** | | 17 / 10 | -0.4 | -0.2 | 0.2 | -1.1 | -0.1 | -0.4 | -0.1 | 1.8 | -0.8 | *0.6* | *2.3* | 0.6 |
| **88** | | 17 / 10 | 2.4 | 1.1 | -2.0 | 1.4 | 0.4 | 0.7 | 2.9 | -1.6 | 0.2 | *-1.1* | *-0.1* | 1.4 |
| **89** | | 16 / 10 | 0.6 | 1.5 | -0.8 | -1.1 | 0.6 | 1.3 | 0.6 | 0.1 | -0.3 | *-0.4* | *0.9* | 0.8 |
| **91** | | 16 / 10 | -3.4 | -0.3 | -1.0 | 2.2 | 1.3 | 1.6 | -2.7 | -1.6 | 0.0 | *1.3* | *3.2* | 1.6 |
| **92** | x | 17 / 9 | 1.1 | 1.7 | -0.1 | -0.4 | 0.8 | 8.1 | -2.2 | 0.0 | -2.8 | *-3.7* | *-3.6* | 1.6 |
| **93** | | 16 / 10 | -0.2 | 0.6 | 0.2 | -1.6 | 2.6 | 1.0 | 0.0 | 0.8 | -1.5 | *-1.2* | *-0.1* | 0.9 |
| **97** | | 16 / 10 | -1.0 | 2.1 | -0.4 | -2.9 | 0.0 | 0.7 | 3.1 | 0.8 | 0.7 | *16.8* | *24.4* | 1.3 |
| **99** | | 17 / 10 | -2.0 | 0.4 | -0.4 | 0.5 | 0.4 | -0.1 | 0.2 | 0.5 | 1.5 | *-0.4* | *0.9* | 0.7 |
| **100** | | 17 / 10 | 3.1 | 1.1 | -1.4 | -1.0 | -1.7 | -0.7 | 3.8 | -0.5 | -1.0 | *0.2* | *1.8* | 1.6 |
| **102** | | 17 / 10 | 0.2 | -0.5 | -0.3 | -1.5 | -1.1 | -0.3 | 0.8 | 1.6 | 0.1 | *-0.9* | *0.3* | 0.7 |
| **105** | | 17 / 10 | 0.0 | 0.5 | 0.0 | 0.9 | -1.0 | -1.0 | -0.3 | -1.1 | 1.6 | *1.1* | *2.9* | 0.7 |
| **106** | | 17 / 9 | -3.5 | -0.7 | -0.4 | -0.9 | 0.4 | -1.3 | -0.3 | 0.7 | 1.0 | *1.7* | *3.8* | 1.0 |
| **107** | | 17 / 10 | -0.7 | 0.1 | 0.1 | 0.0 | -0.5 | -0.8 | -0.5 | -0.2 | -0.8 | *3.4* | *6.1* | 0.4 |
| **109** | | 17 / 9 | -2.9 | 1.3 | -2.1 | -3.6 | -1.1 | -0.1 | -2.2 | -0.8 | 0.3 | *1.4* | *3.3* | 1.6 |
| **110** | | 17 / 9 | -3.8 | -2.7 | 0.4 | -0.4 | 0.9 | -0.5 | -2.4 | -2.2 | 2.8 | *2.7* | *5.2* | 1.8 |
| **111** | | 17 / 10 | 0.4 | 0.6 | -0.4 | -1.1 | 1.2 | 0.4 | 9.5 | -1.4 | 0.7 | *2.3* | *4.6* | 1.2 |
| **112** | | 17 / 10 | 9.4 | 0.0 | 0.1 | 1.8 | 1.5 | 3.6 | 3.4 | -1.1 | -2.3 | *-2.1* | *-1.4* | 2.1 |
| **117** | | 16 / 9 | -1.3 | -0.5 | 0.5 | | 0.7 | -1.6 | -0.9 | 0.0 | -0.7 | *-0.7* | *0.4* | 0.8 |
| **120** | | 15 / 10 | -3.3 | -0.2 | 1.7 | -3.2 | 0.5 | 0.3 | 0.7 | 1.3 | 2.4 | *-0.1* | *1.3* | 1.5 |
| **128** | | 15 / 10 | 29.4 | 0.3 | 0.1 | 0.4 | -0.2 | -0.3 | 5.4 | 0.1 | 5.7 | *4.7* | *7.9* | 1.8 |
| **3rd-27** | | 17 / 10 | -1.4 | -0.6 | 0.8 | 0.1 | 1.9 | 0.8 | -0.9 | 1.2 | 0.3 | *2.3* | *4.6* | 0.9 |
| **3rd-83** | | 17 / 10 | -2.2 | 1.1 | -0.8 | -2.1 | 0.8 | -0.9 | 0.2 | -0.4 | 1.2 | *3.2* | *5.8* | 1.1 |
| **3rd-108** | | 16 / 9 | -1.7 | -0.3 | 0.1 | 0.0 | 1.8 | 0.8 | | 0.5 | -0.3 | *1.1* | *3.0* | 0.7 |

1) calculated using robust approach; the relative deviation to the reference value 0.098 = 51.5 %

**Table 4-11:** Category B laboratories in EUPT-SRM17, ordered by lab-codes. The AAZs were calculated for laboratories having submitted at least 5 results for the evaluated compulsory compounds present in the test material. For the AAZ calculation any z scores > 5 were set at 5. Z scores for phthalimide based on two different assigned values (for details please see **Section 4.2.1**) are calculated for informative purposes only and excluded in the AAZ.

| COMPULSORY Compounds | | | Captan | Chloro-thalonil | Cyro-mazine | DTCs (expr. as $CS_2$) | Dodine | Ema-mectin B1a | Folpet | Pyme-trozine | THPI | Phthalimide | | AAZ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | 0.174 | 0.151 | 0.154 | 0.187 | 0.100 | 0.0461 | 0.249 | 0.150 | 0.590 | *0.134* | *0.098* | |
| *CV** | | | 32.9% | 24.4% | 20.3% | 28.4% | 23.1% | 21.5% | 34.5% | 28.2% | 23.2% | *36.8%* | *36.2%[1)]* | |
| MRRL [mg/kg] | | | 0.02 | 0.01 | 0.02 | 0.02 | 0.01 | 0.01 | 0.02 | 0.02 | 0.01 | 0.01 | | |
| Lab code SRM17- | NRL-SRM | Analysed / corr. found, max. 17 / 10 | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | | |
| **3** | x | 12 / 7 | -3.5 | 0.0 | 0.5 | -0.3 | -0.6 | -0.7 | -0.5 | 1.0 | | | | 0.9 |
| **5** | x | 15 / 8 | 3.9 | 0.0 | -0.2 | 0.6 | 2.1 | 0.0 | 4.2 | 1.2 | | | | 1.5 |
| **6** | | 3 / 1 | | -0.1 | | | | | | | | | | -- |
| **7** | x | 9 / 5 | | 7.1 | 0.2 | 0.5 | | 1.6 | | 2.4 | | | | 1.9 |
| **8** | | 9 / 6 | -0.1 | -1.0 | -0.4 | | -0.5 | | -0.3 | -0.8 | | | | 0.5 |
| **9** | | 9 / 6 | 1.1 | 0.7 | | 0.5 | 0.2 | -0.9 | 0.8 | | | | | 0.7 |
| **13** | | 17 / 8 | -3.8 | -0.8 | -0.4 | -0.1 | -0.4 | 0.9 | -3.8 | -0.9 | 1.8 | *7.3* | *1.8* | 1.4 |
| **16** | | 13 / 8 | -0.4 | -0.1 | | 0.2 | | -0.4 | -0.4 | 0.3 | 0.4 | *0.7* | *0.4* | 0.3 |

1) calculated using robust approach; the relative deviation to the reference value 0.098 = 51.5 %

**Table 4-11 (cont.):** Category B laboratories in EUPT-SRM17, ordered by lab-codes. The AAZs were calculated for laboratories having submitted at least 5 results for the evaluated compulsory compounds present in the test material. For the AAZ calculation any z scores > 5 were set at 5. Z scores for phthalimide based on two different assigned values (for details please see Section 4.2.1) are calculated for informative purposes only and excluded in the AAZ.

| COMPULSORY Compounds | | | Captan | Chloro-thalonil | Cyro-mazine | DTCs (expr. as $CS_2$) | Dodine | Ema-mectin B1a | Folpet | Pyme-trozine | THPI | Phthalimide | | AAZ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Assigned Value [mg/kg] | | | 0.174 | 0.151 | 0.154 | 0.187 | 0.100 | 0.0461 | 0.249 | 0.150 | 0.590 | *0.134* | *0.098* | |
| *CV** | | | 32.9% | 24.4% | 20.3% | 28.4% | 23.1% | 21.5% | 34.5% | 28.2% | 23.2% | *36.8%* | *36.2%*[1)] | |
| MRRL [mg/kg] | | | 0.02 | 0.01 | 0.02 | 0.02 | 0.01 | 0.01 | 0.02 | 0.02 | 0.01 | 0.01 | | |
| Lab code SRM17- | NRL-SRM | Analysed / corr. found, max. 17 / 10 | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | z Score (FFP-RSD = 25 %) | | |
| **19** | x | 5 / 2 | | | | | -1.3 | -0.5 | | | | | | -- |
| **21** | | 12 / 7 | 0.7 | 1.0 | | -0.4 | 0.8 | 0.8 | 0.7 | -1.5 | | | | 0.8 |
| **23** | | 1 / 1 | | | | -0.1 | | | | | | | | -- |
| **24** | | 6 / 6 | 1.2 | 0.1 | | -0.5 | | | 0.2 | | -0.5 | *-0.4* | *-0.5* | 0.5 |
| **29** | | 4 / 3 | | | | | 0.0 | -0.3 | | -1.9 | | | | -- |
| **31** | | 1 / 1 | | 1.8 | | | | | | | | | | -- |
| **32** | | 4 / 2 | | | | -0.2 | | | | -0.2 | | | | -- |
| **33** | | 4 / 2 | | | | | 0.8 | | | 0.8 | | | | -- |
| **46** | | 4 / 3 | | | 1.1 | | -0.5 | | | -1.3 | | | | -- |
| **49** | x | 8 / 4 | 2.7 | 0.4 | | 0.1 | | | -1.6 | -3.5 | | | | 1.7 |
| **52** | | 5 / 4 | | | 0.2 | 1.1 | 2.0 | | | 0.3 | | | | 0.9 |
| **56** | x | 14 / 9 | 28.6 | 3.0 | 0.3 | 2.4 | 0.3 | -0.1 | 14.7 | 1.3 | | *0.2* | | 2.2 |
| **59** | | 1 / 1 | | | | -0.7 | | | | | | | | -- |
| **60** | x | 12 / 7 | | -1.7 | -0.2 | 4.8 | -1.2 | 1.6 | | -1.1 | | *3.1* | | 1.8 |
| **63** | x | 5 / 1 | | -0.6 | | | | | | | | | | -- |
| **66** | | 9 / 4 | -3.5 | -2.6 | | -1.9 | 2.2 | | | -3.1 | | | | 2.7 |
| **71** | | 11 / 6 | 1.7 | 0.0 | | -0.1 | | 0.1 | 5.1 | | -0.4 | *-3.7* | *-0.4* | 1.2 |
| **73** | | 14 / 8 | -3.5 | 2.0 | 1.8 | -0.6 | -0.9 | -0.2 | -0.1 | -0.2 | 0.9 | | *0.9* | 1.1 |
| **75** | | 6 / 4 | | | -0.9 | -0.9 | -2.0 | | | -0.3 | | | | 1.0 |
| **76** | | 11 / 9 | -3.6 | -1.4 | -2.2 | | 0.1 | 3.8 | -3.5 | -1.7 | 1.1 | *1.6* | *1.1* | 2.2 |
| **79** | | 5 / 5 | 1.5 | | | | -0.2 | | 0.6 | | -0.3 | *-0.3* | *-0.3* | 0.7 |
| **80** | | 1 / 1 | | | | 0.8 | | | | | | | | -- |
| **81** | | 1 / 1 | | | | -0.5 | | | | | | | | -- |
| **84** | x | 6 / 3 | | 0.5 | -0.1 | | -2.1 | | | | | | | -- |
| **86** | | 9 / 6 | 0.1 | 0.3 | | -0.2 | | -0.5 | -1.1 | 0.2 | | | | 0.4 |
| **87** | | 4 / 2 | | | 1.8 | -1.9 | | | | | | | | -- |
| **90** | | 14 / 9 | 29.7 | -3.0 | -2.2 | | 2.0 | -0.5 | -2.7 | -2.0 | 0.9 | *-0.7* | *0.9* | 2.3 |
| **94** | | 13 / 8 | 0.7 | -1.0 | | -0.3 | -0.6 | -0.8 | | 0.5 | -1.8 | *-0.2* | *-1.8* | 0.8 |
| **95** | | 14 / 7 | | 0.8 | 0.6 | 0.6 | 0.3 | 3.1 | | 1.0 | 0.7 | | *0.7* | 1.0 |
| **96** | x | 11 / 6 | 0.1 | 0.7 | | | | | 0.4 | 0.3 | -0.1 | *-0.5* | *-0.1* | 0.3 |
| **98** | x | 11 / 6 | | -0.3 | -0.1 | 2.0 | -0.2 | -0.4 | | 0.3 | | | | 0.6 |
| **101** | x | 7 / 5 | | -0.7 | 3.7 | | -3.0 | 0.1 | | 2.9 | | | | 2.1 |
| **104** | x | 16 / 8 | 1.8 | 0.5 | 0.2 | | -0.2 | -0.7 | 3.4 | -0.4 | -1.9 | *-3.7* | *-1.9* | 1.1 |
| **113** | | 7 / 5 | | -1.1 | -0.5 | 0.3 | 0.4 | | | 1.6 | | | | 0.8 |
| **114** | | 12 / 8 | 1.0 | 0.5 | 0.0 | -0.3 | -0.2 | -0.4 | 0.6 | -0.1 | | | | 0.4 |
| **115** | | 6 / 5 | | -0.3 | -1.1 | 0.1 | -0.4 | | | -0.5 | | | | 0.5 |
| **118** | | 13 / 10 | 0.4 | 0.9 | 0.2 | 0.1 | 0.1 | 0.0 | 0.3 | -1.6 | -0.6 | *-0.7* | *-0.6* | 0.5 |
| **119** | | 1 / 1 | | | | 1.2 | | | | | | | | -- |
| **122** | | 1 / 1 | | | | 1.5 | | | | | | | | -- |
| **123** | x | 8 / 8 | 0.0 | 0.3 | 1.6 | -2.3 | | | 0.4 | 0.4 | -1.6 | *-1.5* | *-1.6* | 0.9 |
| **124** | | 6 / 6 | -3.0 | -1.9 | | 0.4 | | | -2.4 | | -0.2 | *1.2* | *-0.2* | 1.6 |
| **125** | | 9 / 7 | -1.9 | 0.0 | | -0.5 | -0.8 | 0.6 | -1.9 | -2.1 | | | | 1.1 |
| **126** | | 6 / 6 | | 0.5 | 0.5 | | | 1.2 | -1.9 | 0.2 | | *1.6* | | 0.9 |
| **127** | | 15 / 8 | | -0.8 | -1.1 | -1.4 | -0.4 | -1.4 | | 6.9 | 0.1 | *0.8* | *0.1* | 1.5 |
| **3rd-14** | | 4 / 2 | | | | | | -0.8 | | -1.7 | | | | -- |
| **3rd-103** | | 16 / 7 | -3.5 | -3.7 | -0.7 | | 0.4 | 2.0 | -1.5 | 1.1 | 1.4 | *0.5* | *1.4* | 1.8 |

1) calculated using robust approach; the relative deviation to the reference value 0.098 = 51.5 %

### 4.3.5 Feedback from Laboratories in Case of Poor Results

Like in the previous EUPT-SRMs, with the publication of the preliminary report all participating laboratories having obtained questionable ($2 < |z\ score| < 3$), unacceptable ($|z\ score| \geq 3$) 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 follow-up measure is to sensibilize the laboratories to timely investigate the sources of errors in order to avoid making the same errors in the future. As described in **Section 4.2.1**, two months after releasing the EUPT-SRM17 preliminay report, the organisers recognized that in the case of ***phthalimide*** the robust mean of the entire population, which was used as a "preliminary assigned value" in the preliminary report, was not suitable as a reference value for checking the performance of the laboratories. The z scores for ***phthalimide*** were therefore recalculated using a new reference value that was based on the sum of incurred and spiked ***phthalimide***, and a new version of the preliminary report was released. All participants were informed about this amendment and the laboratories having analysed for ***phthalimide*** were additionally informed about the background reasons and about the need for adjusting their follow-up activities based on the new preliminary z scores for ***phthalimide***, and if these were questionable or unacceptable to provide the reason(s) for poor performance.

Excluding ***meptyldinocap, 2,4-DNOP*** and ***meptyldinocap (sum)***, for which it was decided not to include z scores in the final report (due to the uncertainty of the robust means), but including ***ETU*** and ***phthalimide*** (in latter case using the sum of incurred and spiked level as reference), whose z scores were calculated for informative purposes only, 81 OfLs from EU/EFTA countries reported 196 results suggesting poor performance. This includes 77 results with questionable z scores, 115 results with unacceptable z scores (incl. 31 FNs), and 4 FPs. If, for the sake of interrest, the spiking level was used as reference value for ***meptyldinocap, 2,4-DNOP*** and ***meptyldinocap (sum)***, 29 results reported by 23 laboratories would fall into the poor performance category (among them 5 FNs). Note: Experiments carried out by the EURL-SRM suggest that the spiking levels of these compounds would be suitable estimates of the assigned values.

73 laboratories having obtained poor performance scores reported in 181 cases (=90 %) the reasons for poor performance. 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 compilation also provides input to NRLs on how to better assist OfLs within the network so that they can improve their performance.

## 4.4 Special Topics

As already mentioned before, certain analytes on the TPL present a big challenge not only in this PT but also in the routine analysis. The organizers outlined the most pressing issues in this section. For more details please refer to the presentation at the Joint EURL-FV/SRM Workshop 2022

**Impact of using ILIS on the analytical results**

The use of isotope labelled analogues of the target analytes as internal standards (ILISs) is probably the most efficient way for overcoming errors. Precondition is that the analytical methods involve mass-spectrometry, and that the ILIS is added at an early stage of the procedure. Following the example of previous EUPT-SRM reports and EURL/NRL-workshops, the positive impact of using ILIS is also highlighted here, by comparing the results generated by labs using ILIS with the results generated by the rest of the population.

The distribution of the results submitted by laboratories using ILIS is more narrow than that of laboratories not using it. The $CV^*$ of the population using ILIS compared to the population not using it was 7.6 % (n = 21) vs. 21.9 % (n = 25) in the case of ***maleic hydrazide***, 9.9 % (n = 19) vs. 23.5 % (n = 73) in the case of ***cyromazine***, 20.8 % (n = 22) vs. 40.3 % (n = 67) in the case of ***folpet,*** and 30.1 % (n = 22) vs. 34.9 % (n = 67) in the case of

**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)

| | Cyromazine | | |
|---|---|---|---|
| | **Whole Population** | **Matching ILIS[1)]** | **No ILIS + Other IS[2)] (Calibration Approach)** |
| No. of Numerical Results | 92 | 19 (4× QuEChERS, 15× QuPPe) | 73<br>Procedural \| 15<br>StAdd-SP[3)] \| 11<br>StAdd-EA[3)] \| 4<br>MM[3)] \| 15<br>SB[3)] \| 17 |
| Thereof outliers[1)] | 0 | 0 | 0 |
| No. of False Negative Results | 0 | 0 | 0 |
| **Robust Mean [mg/kg]** | **0.154** | **0.161** | **0.152** |
| ***CV**** | **20.3%** | **9.9%** | **23.5%** |
| Uncertainty of Robust Mean | 0.0041 | 0.0046 | 0.0053 |
| UAV-Tolerance | 0.0116 | 0.0121 | 0.0114 |
| Judgement (in relation to UAV tolerance) | passed | passed | passed |
| | **Folpet** | | |
| | **Whole Population** | **Matching ILIS[1)]** | **No ILIS + Other IS[2)] (Calibration Approach)** |
| No. of Numerical Results | 89 | 22 | 67<br>Procedural \| 10<br>StAdd-SP[3)] \| 4<br>StAdd-EA[3)] \| 2<br>MM[3)] \| 41 (1°)<br>SB[3)] \| 8 (1°) |
| Thereof outliers[1)] | 2 | 0 | 2 |
| No. of False Negative Results | 1 | 0 | 1 |
| **Robust Mean [mg/kg]** | **0.249** | **0.261** | **0.242** |
| ***CV**** | **34.5%** | **20.8%** | **40.3%** |
| Uncertainty of Robust Mean | **0.0115** | **0.0144** | **0.0151** |
| UAV-Tolerance | 0.0187 | 0.0196 | 0.0182 |
| Judgement (in relation to UAV tolerance) | passed | passed | passed |

1) Isotope-Labelled-Internal Standard (analogue to target analyte) added at beginning of procedure or at an intermediate step
2) Other type of Internal Standard
3) **StAdd-SP**: Standard addition to sample portions; **StAdd-EA**: Standard addition to extract aliquots; **MM**: Matrix-Matched calibration; **SB**: Solvent-Based calibration
4) ILIS added at beginning of procedure or during an intermediate step
5) Due to the small population of results the robust mean of this subpopulation is too uncertain and wouldn't qualify as an AV

***captan***. More detailed data about these cases can be see in **Table 4-12**. In the latter two cases, the distribution of the results is still relatively broad despite the use of ILIS, and this can be attributed to the fact that in many laboratories the test-sample was left to thaw prior to analysis, and thus prior to adding any ILIS. So, the ILIS was not able to compensate for the losses which have occurred during thawing.

### Impact of Calibration on Cyromazine Quantification

***Cyromazine*** is a relatively polar compound, that shows recovery rates around 40 % in QuEChERS. Many laboratories thus employ QuPPe-style methods for this analyte. In the present PT, equal numbers of laboratories have employed QuEChERS, and QuPPe (47 laboratories each). Among the 19 laboratories using

**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)

| | Captan | | |
|---|---|---|---|
| | **Whole Population** | **Matching ILIS[1)]** | **No ILIS + Other IS (Calibration Approach)** |
| No. of Numerical Results | 84 | 25 | 59<br>Procedural \| 7<br>StAdd-SP \| 4<br>StAdd-EA \| 1<br>MM \| 34 (4°)<br>SB \| 9 |
| Thereof outliers[1)] | 5 | 1 | 4 |
| No. of False Negative Results | 5 | 0 | 5 |
| **Robust Mean [mg/kg]** | **0.174** | **0.177** | **0.171** |
| ***CV**** | **32.9%** | **30.1%** | **37.5%** |
| Uncertainty of Robust Mean | 0.0080 | 0.0136 | 0.0108 |
| UAV-Tolerance | 0.0131 | 0.0133 | 0.0128 |
| Judgement (in relation to UAV tolerance) | passed | failed [5)] | passed |
| | **Maleic hydrazide** | | |
| | **Whole Population** | **Matching ILIS[1)]** | **No ILIS + Other IS [2)] (Calibration Approach)** |
| No. of Numerical Results | 46 | 22 | \| 25<br>Procedural \| 6<br>StAdd-SP \| 3<br>StAdd-EA \| 2<br>MM \| 11<br>SB \| 3 |
| Thereof outliers[1)] | 0 | 0 | 0 |
| No. of False Negative Results | 1 | 0 | 1 |
| **Robust Mean [mg/kg]** | **0.544** | **0.523** | **0.563** |
| ***CV**** | **16.6%** | **7.6%** | **21.9%** |
| Uncertainty of Robust Mean | 0.0167 | 0.0106 | 0.0308 |
| UAV-Tolerance | 0.0408 | 0.0392 | 0.0422 |
| Judgement | passed | passed | passed |

1) Isotope-Labelled-Internal Standard (analogue to target analyte) added at beginning of procedure or at an intermediate step
2) Other type of Internal Standard
3) **StAdd-SP**: Standard addition to sample portions; **StAdd-EA**: Standard addition to extract aliquots; **MM**: Matrix-Matched calibration; **SB**: Solvent-Based calibration
4) ILIS added at beginning of procedure or during an intermediate step
5) Due to the small population of results the robust mean of this subpopulation is too uncertain and wouldn't qualify as an AV

ILIS, 15 employed it in combination with the QuPPe method and 4 labs in combination with QuEChERS (**Table 4-12**). Among the 73 laboratories not using ILIS 23 labs corrected for recovery either via a procedural calibration or via standard addition to sample portions. Among the 50 remaining laboratories, 17 laboratories corrected for recovery using a recovery factor, thereof 14 laboratories using QuEChERS. The average recovery rate reported by these 14 laboratories was 43 ± 9 % with all but one of these labs achieving acceptable results after correction for recovery (the corrected results finally reported by these labs deviated from the assigned value by 7.5 % on average, with 1 outlier). In total, 64 % of the laboratories analysing for cyromazine have employed one or another approach for correcting their result for recovery. Among the labs not correcting, the ones employing QuPPe extraction in combination with matrix-matched calibration formed the largest group.

**Table 4-13:** Comparison of the results of dithiocarbamates generated by various analytical approaches

| | Dithiocarbamates (expr. as CS2) | | | |
|---|---|---|---|---|
| | Whole Population | HS + SPME | LLP | Spectrophoto-metric |
| No. of Numerical Results | 97 | 32 | 42 | 18 |
| Thereof outliers | 2 | 1 | 1 | 0 |
| No. of False Negative Results | 2 | 2 | 0 | 0 |
| **Robust Mean [mg/kg]** | **0.187** | **0.205** | **0.183** | **0.175** |
| $CV^*$ | **28.4%** | **31.8%** | **31.1%** | **20.0%** |
| Uncertainty of Robust Mean | 0.0068 | 0.0147 | 0.0111 | 0.0103 |
| UAV-Tolerance | 0.0140 | 0.0154 | 0.0137 | 0.0131 |
| Judgement (in relation to UAV tolerance) | passed | passed | passed | passed |

## Impact of Analytical Methods on the Results of Dithiocarbamates

In one of the past EUPTs (EUPT-SRM11, 2016), in which a polymeric dithiocarbamate (propineb) was spiked, it was observed that the conditions of the cleavage step (acid strength, reagent/matrix ratio temperature and duration) influenced the yields.

In the present PT, where ***metiram*** was spiked, the results submitted by the laboratories were not as broadly dispersed. Among the 97 laboratories reporting results, 42 have reported results involving liquid-liquid partitioning into a non-polar solvent prior to GC analysis (LLP-methods). 32 laboratories have employed headspace analysis involving either direct or SPME-assisted sampling (HS methods). Finally, 18 laboratories have employed methods involving spectrophotometric detection (SpPhM methods) (**Table 4-13**).

Interestingly, the population of results determined by SpPhM methods was the least dispersed one, with a robust standard deviation ($CV^*$) of just 20.0 % (n = 18). The respective $CV^*$ figures for the LLP and the HS population were 31.1 % and 31.8 %, respectively. The robust mean value of the SpPhM method population and the LLP-method population (0.175 vs 0.183 mg/kg) were comparable, with the robust mean of the headspace method population (0.205 mg/kg) deviating towards the higher end. For determining the assigned value, the robust mean of the entire population was used.

## Challenges in the analysis of Folpet (sum) and Captan (sum)

The TPL did not foresee the submission of results for the sum of ***folpet*** and ***phthalimide*** and the sum of ***captan*** and ***THPI***. In the EUPT-SC meeting it was decided that an evaluation of the summed results would be of interest and should be included in the final report for information purposes. An additional evaluation for the sum of ***folpet*** and ***phthalimide*** as well as the sum of ***captan*** and ***THPI*** was therefore carried out by the organisers. For this, the organizers have only used results of laboratories submitting results for both the parent and its respective degradant. The degradant was expressed as parent and the sum was calculated.

In total, 74 laboratories have submitted results for both ***folpet*** and ***phthalimide*** (see **Table 4-14**) and 73 laboratories have submitted results for both ***captan*** and ***THPI*** (see **Table 4-15, p. 55**). The robust means of the summed values of the respective subpopulations were designated as the assigned values for ***folpet (sum)*** and ***captan (sum)***, and used as a basis for calculating the corresponding z scores. Pro forma, z scores were also calculated for laboratories reporting results for only one of these two compounds. No z score was calculated for the two laboratories having reported FN results for ***captan*** , without analyzing for ***THPI***.

The z scores calculated for ***folpet (sum)*** are shown in **Table 4-16 (p. 56)** as well as in **Figure 4-1 (p. 58)** and **Figure 4-2 (p. 58)** with the former highlighting the cases where only one of the two analytes was

**Table 4-14:** Statistical evaluation of folpet, phthalimide and the sum of folpet and phthalimide expressed as folpet. The latter was calculated by the organisers using the results of participants having analysed for both folpet and phthalimide.

| | Folpet | Phthalimide | Phthalimide | Folpet + Phthalimide (expr. as folpet) |
|---|---|---|---|---|
| No. Numerical Results | 89 | 78 | 78 | 74* |
| No. of Outliers | 2 | 2 | 7 | 4 |
| Remaining results after outlier elimination | 87 | 76 | 71 | 70 |
| FN | 1 | 5 | 5 | 0 |
| Reference Value Used [mg/kg] (Source) | 0.249 (robust mean) | 0.134 (robust mean) | 0.098** (incurred + spiked) | 0.519 (robust mean) |
| *CV** | 34.5 % | 36.8 % | 36.2 %*** | 19.5 % |

*: 74 laboratories have analysed for both folpet and phthalimide; their results were used to establish the robust mean for the sum.
**: sum of the incurred level of phthalimide in the tomato homogenate 0.020 mg/kg (determined using LC-MS/MS prior to spiking in the lab ) and the spiked level of 0.078 mg/kg.
***: calculated using robust approach; the relative deviation to the reference value 0.098 = 51.5 %

**Table 4-15:** Statistical evaluation of captan, THPI and the sum of captan and THPI expressed as captan. The latter was calculated by the organisers using the results of participants having analysed for both captan and THPI.

| | Captan | THPI | Captan (sum) (Captan + THPI* (expr. as captan)) |
|---|---|---|---|
| No. Numerical Values | 84 | 80 | 73 |
| No. of Outliers | 5 | 1 | 2 |
| Remaining results after outlier elimination | 79 | 79 | 71 |
| FN | 6 | 0 | 2 |
| Robust Mean [mg/kg] | 0.174 | 0.590 | 1.32 (as Captan) |
| *CV** | 32.9 % | 23.2 % | 20.9 % |

targeted and the latter showing the share of each analyte to the sum. The z scores calculated for ***captan (sum)*** are shown in (**Table 4-17, p. 59**) as well as in **Figure 4-3 (p. 61)** and **Figure 4-4 (p. 61)** with the former highlighting the cases where only one of the two analytes was targeted and the latter showing the share of each analyte to the sum.

As described in **Section 4.2.1 (p. 28)**, using GC techniques ***folpet*** and ***captan*** decompose upon contact with the hot surface of the injector to ***phthalimide*** and ***THPI,*** respectively. ***Phthalimide*** and ***THPI*** formed through decomposition in the injector co-elute with ***phthalimide*** and ***THPI*** originally present in the sample extracts leading to an overestimation of the ***phthalimide*** or ***THPI*** content, unless measures are taken to correct for this. A calibration procedure taking this aspect into account and deducting the share of the degradant formed in the injector was developed by the EURL-SRM (see Calibration approach 1 and Calibration approach 2). Details on the principles of these calibration approaches are found under document SRM-07.

The overestimation of the degradant concentration is typically not accompanied by an underestimation of the parent, as the parent losses are matched for via the calibration (e.g. matrix-matching or ILIS-based).

The problems with the overestimation of the concentration of degradants become more prominent, the higher the concentration ratio of parent to degradant is. They become, however, irrelevant if LC-MS/MS is used for measurement, where no degradation takes place. A document describing an LC-MS/MS approach was published by the EURL Report SRM-49.

**Table 4-16:** Compilation of laboratory results of folpet, phthalimide and the sum thereof expressed as folpet. The latter was calculated by the organisers. The z scores of phthalimide and the sum are for information only. Where laboratories have not analysed for both components, the lab code, the result for the sum as well as the z score for the sum are highlighted in red.

| | Folpet | | | Phthalimide | | | Folpet (sum) (Folpet + PI* (expr. as folpet)) | |
|---|---|---|---|---|---|---|---|---|
| Lab-Code SRM17- | Conc. [mg/kg] | z score | Determ. Techn. | Conc. [mg/kg] | z score | Determ. Techn. | Conc. [mg/kg] | z score |
| 92* | 0.112 | -2.2 | GC-MS/MS (QQQ) | FN | -3.6 | LC-MS/MS (QQQ) | 0.112 | -3.1 |
| 125 | 0.131 | -1.9 | GC-MS/MS (QQQ) | | | | 0.131 | -3.0 |
| 49* | 0.148 | -1.6 | GC-MSD | | | | 0.148 | -2.8 |
| 44 | 0.167 | -1.3 | GC-MS/MS (QQQ) | FN | -3.6 | GC-MS/MS (QQQ) | 0.167 | -2.7 |
| 86 | 0.178 | -1.1 | GC- (μ) ECD | | | | 0.178 | -2.6 |
| 57 | 0.0368 | -3.4 | GC-MS/MS (QQQ) | 0.0786 | -0.8 | GC-MS/MS (QQQ) | 0.195 | -2.5 |
| 3* | 0.218 | -0.5 | GC-Orbitrap | | | | 0.218 | -2.3 |
| 8 | 0.230 | -0.3 | GC-MSD | | | | 0.230 | -2.2 |
| 73 | 0.240 | -0.1 | GC-MS/MS (QQQ) | | | | 0.240 | -2.1 |
| 94 | | | | 0.127 | 1.2 | GC-MS/MS (QQQ) | 0.256 | -2.0 |
| 30 | 0.12 | -2.1 | LC-MS/MS (QQQ) | 0.079 | -0.8 | LC-MS/MS (QQQ) | 0.279 | -1.8 |
| 114 | 0.284 | 0.6 | GC- (μ) ECD | | | | 0.284 | -1.8 |
| 21 | 0.29 | 0.7 | GC-MS/MS (QQQ) | | | | 0.290 | -1.7 |
| 9 | 0.299 | 0.8 | GC- (μ) ECD | | | | 0.299 | -1.7 |
| 90 | 0.080 | -2.7 | GC-MS/MS (QQQ) | 0.110 | 0.5 | GC-MS/MS (QQQ) | 0.302 | -1.6 |
| 127 | | | | 0.16 | 2.5 | GC-MS/MS (QQQ) | 0.323 | -1.5 |
| 20 | 0.340 | 1.5 | GC-MS/MS (QQQ) | FN | -3.6 | GC-MS/MS (QQQ) | 0.340 | -1.3 |
| 40 | 0.157 | -1.5 | LC-MS/MS (QQQ) | 0.097 | 0.0 | GC-MS/MS (QQQ) | 0.353 | -1.2 |
| 43* | 0.227 | -0.4 | GC-MS/MS (QQQ) | 0.0663 | -1.3 | GC-MS/MS (QQQ) | 0.361 | -1.2 |
| 50* | 0.247 | 0.0 | GC-MS/MS (QQQ) | 0.066 | -1.3 | GC-MS/MS (QQQ) | 0.380 | -1.0 |
| 28* | 0.227 | -0.4 | GC-MS/MS (QQQ) | 0.0766 | -0.9 | GC-MS/MS (QQQ) | 0.381 | -1.0 |
| 22 | 0.213 | -0.6 | GC-MS/MS (QQQ) | 0.0864 | -0.5 | GC-MS/MS (QQQ) | 0.387 | -1.0 |
| 37 | 0.214 | -0.6 | GC-MS/MS (QQQ) | 0.088 | -0.4 | GC-MS/MS (QQQ) | 0.391 | -0.9 |
| 26 | 0.175 | -1.2 | GC-MS/MS (QQQ) | 0.108 | 0.4 | GC-MS/MS (QQQ) | 0.393 | -0.9 |
| 67 | 0.181 | -1.1 | GC- (μ) ECD | 0.106 | 0.3 | GC-MS/MS (QQQ) | 0.395 | -0.9 |
| 78* | 0.233 | -0.3 | GC-MS/MS (QQQ) | 0.081 | -0.7 | GC-MS/MS (QQQ) | 0.396 | -0.9 |
| 34 | 0.301 | 0.8 | LC-MS/MS (QQQ) | 0.054 | -1.8 | LC-MS/MS (QQQ) | 0.410 | -0.8 |
| 117 | 0.194 | -0.9 | GC-MS/MS (QQQ) | 0.109 | 0.4 | GC-MS/MS (QQQ) | 0.414 | -0.8 |
| 76 | 0.0336 | -3.5 | GC-MS/MS (QQQ) | 0.189 | 3.7 | GC-MS/MS (QQQ) | 0.415 | -0.7 |
| 25 | 0.167 | -1.3 | GC-Orbitrap | 0.129 | 1.3 | GC-Orbitrap | 0.427 | -0.7 |
| 91 | 0.084 | -2.7 | GC-MS/MS (QQQ) | 0.176 | 3.2 | GC-MS/MS (QQQ) | 0.439 | -0.6 |
| 123* | 0.273 | 0.4 | GC- (μ) ECD | 0.0823 | -0.6 | GC- (μ) ECD | 0.439 | -0.6 |
| 93 | 0.251 | 0.0 | GC-MS/MS (QQQ) | 0.095 | -0.1 | GC-MS/MS (QQQ) | 0.442 | -0.5 |
| 58* | 0.281 | 0.5 | GC-MS/MS (QQQ) | 0.081 | -0.7 | GC-MS/MS (QQQ) | 0.444 | -0.5 |
| 64* | 0.284 | 0.6 | GC-MS/MS (QQQ) | 0.0808 | -0.7 | LC-MS/MS (QQQ) | 0.447 | -0.5 |
| 124 | 0.100 | -2.4 | GC-Ion Trap | 0.174 | 3.1 | GC-Ion Trap | 0.451 | -0.5 |
| 45 | 0.243 | -0.1 | GC-MS/MS (QQQ) | 0.106 | 0.3 | GC-MS/MS (QQQ) | 0.457 | -0.4 |
| 104* | 0.459 | 3.4 | GC-MS/MS (QQQ) | FN | -3.6 | GC-MS/MS (QQQ) | 0.459 | -0.4 |
| 18* | 0.216 | -0.5 | GC-MS/MS (QQQ) | 0.124 | 1.1 | LC-MS/MS (QQQ) | 0.466 | -0.3 |
| 55* | 0.327 | 1.3 | LC-(HR)-TOF-MS | 0.069 | -1.2 | LC-(HR)-TOF-MS | 0.466 | -0.3 |
| 109 | 0.11 | -2.2 | GC-MS/MS (QQQ) | 0.18 | 3.3 | GC-MS/MS (QQQ) | 0.473 | -0.3 |
| 53 | 0.287 | 0.6 | GC-MS/MS (QQQ) | 0.0964 | -0.1 | GC-MS/MS (QQQ) | 0.481 | -0.2 |
| 60* | | | | 0.239 | 5.8 | GC-MS/MS (QQQ) | 0.482 | -0.2 |
| 12* | 0.141 | -1.7 | GC- (μ) ECD | 0.0823 | -0.6 | GC- (μ) ECD | 0.482 | -0.2 |
| 118 | 0.267 | 0.3 | GC-MS/MS (QQQ) | 0.110 | 0.5 | GC-MS/MS (QQQ) | 0.489 | -0.2 |
| 62 | 0.306 | 0.9 | LC-MS/MS (QQQ) | 0.094 | -0.2 | LC-MS/MS (QQQ) | 0.495 | -0.1 |

*: Z-scores for the sum were calculated also in those cases where only one of the two compounds were analysed. Some of the laboratories within this subgroup reported that they have analysed for the sum via the degradation product phthalimide.

**Table 4-16 (cont.):** Compilation of laboratory results of folpet, phthalimide and the sum thereof expressed as folpet. The latter was calculated by the organisers. The z scores of phthalimide and the sum are for information only. Where laboratories have not analysed for both components, the lab code, the result for the sum as well as the z score for the sum are highlighted in red.

| | Folpet | | | Phthalimide | | | Folpet (sum) (Folpet + PI* (expr. as folpet)) | |
|---|---|---|---|---|---|---|---|---|
| Lab-Code SRM17- | Conc. [mg/kg] | z score | Determ. Techn. | Conc. [mg/kg] | z score | Determ. Techn. | Conc. [mg/kg] | z score |
| 54 | 0.329 | 1.3 | GC-MS/MS (QQQ) | 0.0862 | -0.5 | GC-MS/MS (QQQ) | 0.503 | -0.1 |
| 99 | 0.260 | 0.2 | GC-MSD | 0.121 | 0.9 | GC-MS/MS (QQQ) | 0.504 | 0.0 |
| 126 | 0.130 | -1.9 | GC-MS/MS (QQQ) | 0.187 | 3.6 | GC-MS/MS (QQQ) | 0.507 | 0.0 |
| 24 | 0.263 | 0.2 | GC-MS/MS (QQQ) | 0.121 | 0.9 | GC-MS/MS (QQQ) | 0.507 | 0.0 |
| 102 | 0.296 | 0.8 | GC-MS/MS (QQQ) | 0.105 | 0.3 | GC-MS/MS (QQQ) | 0.508 | 0.0 |
| 5* | 0.247 | 0.0 | GC-MS/MS (QQQ) | | | | 0.510 | 0.0 |
| 96* | 0.274 | 0.4 | GC- (μ) ECD | 0.117 | 0.8 | LC-MS/MS (QQQ) | 0.510 | 0.0 |
| 47 | 0.282 | 0.5 | LC-MS/MS (QQQ) | 0.113 | 0.6 | LC-MS/MS (QQQ) | 0.510 | 0.0 |
| 41 | 0.21 | -0.6 | GC-MSD | 0.15 | 2.1 | GC-MSD | 0.512 | 0.0 |
| 42 | 0.22 | -0.5 | GC-MS/MS (QQQ) | 0.15 | 2.1 | GC-MS/MS (QQQ) | 0.522 | 0.1 |
| 39 | 0.320 | 1.1 | GC-MS/MS (QQQ) | 0.102 | 0.2 | GC-MS/MS (QQQ) | 0.526 | 0.1 |
| 89 | 0.285 | 0.6 | GC-MS/MS (QQQ) | 0.120 | 0.9 | GC-MS/MS (QQQ) | 0.527 | 0.1 |
| 68* | 0.290 | 0.7 | GC-MS/MS (QQQ) | 0.120 | 0.9 | GC-MS/MS (QQQ) | 0.532 | 0.2 |
| 74 | 0.21 | -0.6 | GC-MS/MS (QQQ) | 0.16 | 2.5 | GC-MS/MS (QQQ) | 0.533 | 0.2 |
| 79 | 0.286 | 0.6 | GC-MS/MS (QQQ) | 0.124 | 1.1 | GC-MS/MS (QQQ) | 0.536 | 0.2 |
| 16 | 0.225 | -0.4 | GC- (μ) ECD | 0.159 | 2.5 | GC-MS/MS (QQQ) | 0.545 | 0.3 |
| 15 | 0.204 | -0.7 | GC-MS/MS (QQQ) | 0.170 | 2.9 | GC-MS/MS (QQQ) | 0.547 | 0.3 |
| 69 | 0.301 | 0.8 | GC-MSD | 0.125 | 1.1 | GC-MS/MS (QQQ) | 0.553 | 0.3 |
| 85 | 0.244 | -0.1 | GC-MS/MS (QQQ) | 0.155 | 2.3 | GC-MS/MS (QQQ) | 0.556 | 0.4 |
| 120 | 0.294 | 0.7 | GC-MS/MS (QQQ) | 0.13 | 1.3 | GC-MS/MS (QQQ) | 0.556 | 0.4 |
| 110 | 0.1021 | -2.4 | GC-MS/MS (QQQ) | 0.226 | 5.2 | GC-MS/MS (QQQ) | 0.558 | 0.4 |
| 61 | 0.342 | 1.5 | GC-MS/MS (QQQ) | 0.108 | 0.4 | GC-MS/MS (QQQ) | 0.560 | 0.4 |
| 71 | 0.567 | 5.1 | GC-MS/MS (QQQ) | FN | -3.6 | GC-MS/MS (QQQ) | 0.567 | 0.4 |
| 38 | 0.227 | -0.4 | GC-MS/MS (QQQ) | 0.170 | 2.9 | GC-MS/MS (QQQ) | 0.570 | 0.5 |
| 105 | 0.23 | -0.3 | GC-MS/MS (QQQ) | 0.17 | 2.9 | GC-MS/MS (QQQ) | 0.573 | 0.5 |
| 82 | 0.240 | -0.1 | GC-MSD | 0.171 | 3.0 | GC-MSD | 0.585 | 0.6 |
| 112 | 0.458 | 3.4 | GC-MS/MS (QQQ) | 0.063 | -1.4 | GC-MS/MS (QQQ) | 0.585 | 0.6 |
| 51 | 0.291 | 0.7 | GC-MS/MS (QQQ) | 0.148 | 2.0 | GC-MS/MS (QQQ) | 0.589 | 0.6 |
| 35* | 0.291 | 0.7 | GC-TOF-MS (unit resol.) | 0.152 | 2.2 | GC-TOF-MS (unit resol.) | 0.597 | 0.7 |
| 106 | 0.232 | -0.3 | GC-MS/MS (QQQ) | 0.19 | 3.8 | GC-MS/MS (QQQ) | 0.615 | 0.8 |
| 48 | 0.255 | 0.1 | GC-MS/MS (QQQ) | 0.180 | 3.3 | GC-MS/MS (QQQ) | 0.618 | 0.8 |
| 88 | 0.430 | 2.9 | GC-MS/MS (QQQ) | 0.096 | -0.1 | LC-MS/MS (QQQ) | 0.624 | 0.9 |
| 36* | 0.265 | 0.3 | GC-MS/MS (QQQ) | 0.185 | 3.6 | GC-MS/MS (QQQ) | 0.638 | 1.0 |
| 10 | 0.250 | 0.0 | GC-MS/MS (QQQ) | 0.197 | 4.0 | GC-MS/MS (QQQ) | 0.647 | 1.1 |
| 11 | 0.312 | 1.0 | GC-MS/MS (QQQ) | 0.169 | 2.9 | GC-MS/MS (QQQ) | 0.653 | 1.1 |
| 77 | 0.267 | 0.3 | GC-MS/MS (QQQ) | 0.201 | 4.2 | GC-MS/MS (QQQ) | 0.672 | 1.3 |
| 17* | 0.37 | 1.9 | GC-MS/MS (QQQ) | 0.15 | 2.1 | GC-MS/MS (QQQ) | 0.672 | 1.3 |
| 4* | 0.148 | -1.6 | GC-MSD | | | | 0.692 | 1.4 |
| 107 | 0.219 | -0.5 | GC- (μ) ECD | 0.248 | 6.1 | GC-MS/MS (QQQ) | 0.719 | 1.6 |
| 13 | FN | -3.8 | GC-MS/MS (QQQ) | 0.3776 | 11.4 | GC-MS/MS (QQQ) | 0.761 | 2.0 |
| 100 | 0.486 | 3.8 | GC-MS/MS (QQQ) | 0.141 | 1.8 | GC-MS/MS (QQQ) | 0.770 | 2.0 |
| 72* | 0.225 | -0.4 | GC-MS/MS (QQQ) | 0.298 | 8.2 | GC-MS/MS (QQQ) | 0.826 | 2.5 |
| 70 | 0.546 | 4.8 | GC-MS/MS (QQQ) | 0.145 | 1.9 | GC-MS/MS (QQQ) | 0.838 | 2.6 |
| 128 | 0.587 | 5.4 | GC-MS/MS (QQQ) | 0.291 | 7.9 | GC-MS/MS (QQQ) | 1.174 | 5.2 |
| 111 | 0.84 | 9.5 | GC-MSD | 0.21 | 4.6 | GC-MS/MS (QQQ) | 1.263 | 5.9 |
| 56* | 1.164 | 14.7 | GC-MS/MS (QQQ) | 0.140 | 1.7 | GC-MS/MS (QQQ) | 1.446 | 7.3 |
| 97 | 0.442 | 3.1 | GC-MS/MS (QQQ) | 0.697 | 24.4 | GC-MS/MS (QQQ) | 1.847 | 10.5 |

*: Z-scores for the sum were calculated also in those cases where only one of the two compounds were analysed. Some of the laboratories within this subgroup reported that they have analysed for the sum via the degradation product phthalimide.

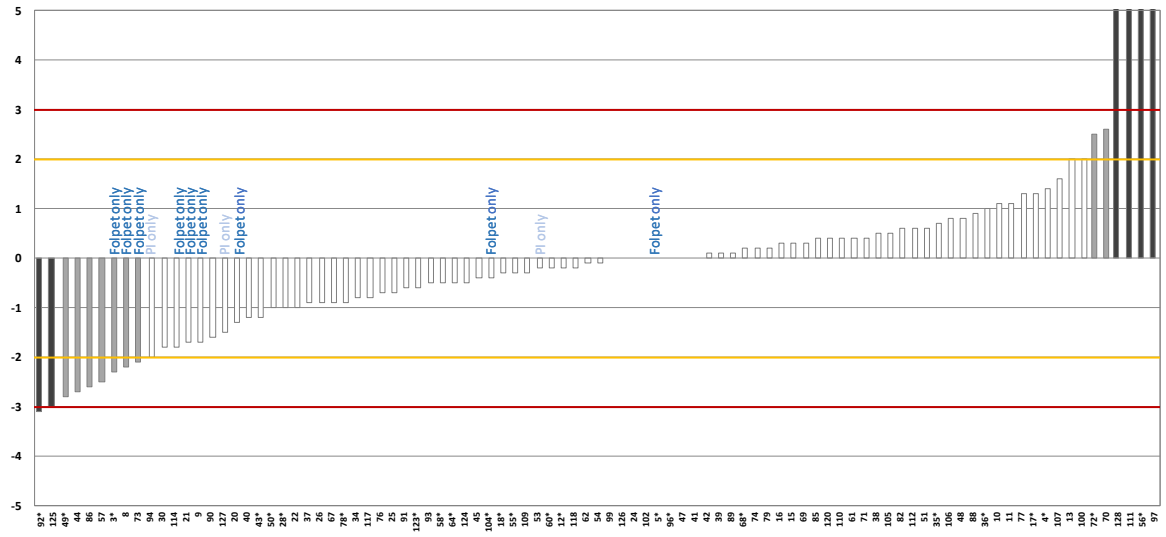



**Figure 4-1:** Graphic presentation of z scores achieved by participating laboratories (the robust mean for folpet (sum) shown in **Table 4-14 (p. 55)** was used as reference value for z score calculation). Laboratories having reported only for one of the two components are highlighted.

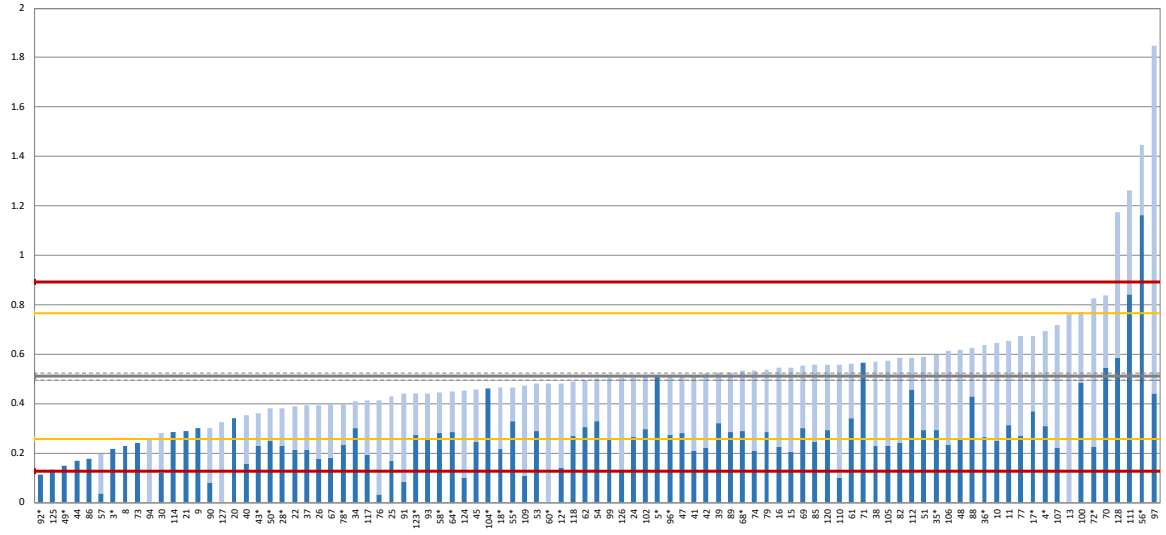



**Figure 4-2:** Graphic presentation of folpet (sum) concentrations as caluclated by the organisers using the results for folpet and phthalimide. The share of results of folpet reported by the participants are shown in dark blue. The share of the reported phthalimide results (expressed as folpet) are shown in pale blue.

**Table 4-17:** Compilation of laboratory results of captan, THPI and the sum thereof expressed as captan. The latter was calculated by the organisers. The z scores of the sum are for information only. Where laboratories have not analysed for both components, the lab code, the result for the sum as well as the z score for the sum are highlighted in red.

| Lab-Code SRM17- | Captan | | | THPI | | | Captan (sum) (Captan + THPI* (expr. as captan)) | |
|---|---|---|---|---|---|---|---|---|
| | Conc. [mg/kg] | z score | Determ. Techn. | Conc. [mg/kg] | z score | Determ. Techn. | Conc. [mg/kg] | z score |
| 3* | FN | -3.5 | GC-MS/MS (QQQ) | | | | – | – |
| 66 | FN*, RL (Lab) > AV | -3.5 | GC-MS/MS (QQQ) | | | | – | – |
| 125 | 0.089 | -2.0 | GC-MS/MS (QQQ) | | | | 0.089 | -3.7 |
| 12* | 0.113 | -1.4 | GC- (μ) ECD | | | | 0.113 | -3.7 |
| 8 | 0.168 | -0.1 | GC-MSD | | | | 0.168 | -3.5 |
| 86 | 0.178 | 0.1 | GC- (μ) ECD | | | | 0.178 | -3.5 |
| 21 | 0.20 | 0.6 | GC-MS/MS (QQQ) | | | | 0.200 | -3.4 |
| 55* | 0.212 | 0.9 | LC-(HR)-TOF-MS | | | | 0.212 | -3.4 |
| 114 | 0.213 | 0.9 | GC- (μ) ECD | | | | 0.213 | -3.4 |
| 9 | 0.218 | 1.0 | GC- (μ) ECD | | | | 0.218 | -3.3 |
| 49* | 0.289 | 2.6 | GC-MSD | | | | 0.289 | -3.1 |
| 5* | 0.183 | 0.2 | GC-MS/MS (QQQ) | | | | 0.340 | -3.0 |
| 26 | 0.190 | 0.4 | GC-MS/MS (QQQ) | 0.136 | -3.1 | GC-MS/MS (QQQ) | 0.460 | -2.6 |
| 92* | 0.221 | 1.1 | GC- (μ) ECD | 0.180 | -2.8 | LC-MS/MS (QQQ) | 0.579 | -2.2 |
| 40 | 0.026 | -3.4 | LC-MS/MS (QQQ) | 0.311 | -1.9 | GC-MS/MS (QQQ) | 0.644 | -2.0 |
| 94 | 0.202 | 0.6 | GC-MS/MS (QQQ) | 0.32 | -1.8 | GC-MS/MS (QQQ) | 0.838 | -1.5 |
| 104* | 0.250 | 1.7 | GC-MS/MS (QQQ) | 0.305 | -1.9 | GC-MS/MS (QQQ) | 0.856 | -1.4 |
| 123* | 0.173 | 0.0 | GC- (μ) ECD | 0.347 | -1.6 | GC- (μ) ECD | 0.863 | -1.4 |
| 93 | 0.164 | -0.2 | GC-MS/MS (QQQ) | 0.368 | -1.5 | GC-MS/MS (QQQ) | 0.896 | -1.3 |
| 61 | 0.220 | 1.1 | GC-MS/MS (QQQ) | 0.426 | -1.1 | GC-MS/MS (QQQ) | 1.067 | -0.8 |
| 117 | 0.117 | -1.3 | GC-MS/MS (QQQ) | 0.482 | -0.7 | GC-MS/MS (QQQ) | 1.075 | -0.7 |
| 112 | 0.585 | 9.4 | GC-MS/MS (QQQ) | 0.247 | -2.3 | GC-MS/MS (QQQ) | 1.076 | -0.7 |
| 57 | | | | 0.544 | -0.3 | GC-MS/MS (QQQ) | 1.082 | -0.7 |
| 17* | 0.29 | 2.7 | GC-MS/MS (QQQ) | 0.40 | -1.3 | GC-MS/MS (QQQ) | 1.085 | -0.7 |
| 107 | 0.144 | -0.7 | GC- (μ) ECD | 0.474 | -0.8 | GC-MS/MS (QQQ) | 1.087 | -0.7 |
| 43* | 0.211 | 0.9 | GC-MS/MS (QQQ) | 0.443 | -1.0 | GC-MS/MS (QQQ) | 1.092 | -0.7 |
| 85 | 0.156 | -0.4 | GC-MS/MS (QQQ) | 0.471 | -0.8 | GC-MS/MS (QQQ) | 1.093 | -0.7 |
| 50* | 0.183 | 0.2 | GC-MS/MS (QQQ) | 0.463 | -0.9 | GC-MS/MS (QQQ) | 1.104 | -0.6 |
| 62 | 0.175 | 0.0 | LC-MS/MS (QQQ) | 0.473 | -0.8 | LC-MS/MS (QQQ) | 1.116 | -0.6 |
| 30 | 0.008 (FR) | -3.8 | LC-MS/MS (QQQ) | 0.57 | -0.1 | LC-MS/MS (QQQ) | 1.141 | -0.5 |
| 34 | 0.134 | -0.9 | LC-MS/MS (QQQ) | 0.519 | -0.5 | LC-MS/MS (QQQ) | 1.166 | -0.5 |
| 124 | 0.041 | -3.1 | GC-Ion Trap | 0.566 | -0.2 | GC-Ion Trap | 1.166 | -0.5 |
| 100 | 0.309 | 3.1 | GC-MS/MS (QQQ) | 0.436 | -1.0 | GC-MS/MS (QQQ) | 1.176 | -0.4 |
| 39 | 0.276 | 2.3 | GC-MS/MS (QQQ) | 0.456 | -0.9 | GC-MS/MS (QQQ) | 1.183 | -0.4 |
| 82 | 0.120 | -1.2 | GC-MSD | 0.542 | -0.3 | GC-MSD | 1.198 | -0.4 |
| 118 | 0.191 | 0.4 | GC-MS/MS (QQQ) | 0.507 | -0.6 | GC-MS/MS (QQQ) | 1.199 | -0.4 |
| 91 | 0.025 | -3.4 | GC-MS/MS (QQQ) | 0.596 | 0.0 | GC-MS/MS (QQQ) | 1.210 | -0.3 |
| 127 | | | | 0.61 | 0.1 | GC-MS/MS (QQQ) | 1.213 | -0.3 |
| 45 | 0.174 | 0.0 | GC-MS/MS (QQQ) | 0.529 | -0.4 | GC-MS/MS (QQQ) | 1.226 | -0.3 |
| 38 | 0.130 | -1.0 | GC-MS/MS (QQQ) | 0.560 | -0.2 | LC-MS/MS (QQQ) | 1.244 | -0.2 |
| 24 | 0.223 | 1.1 | GC-MS/MS (QQQ) | 0.517 | -0.5 | GC-MS/MS (QQQ) | 1.251 | -0.2 |
| 25 | 0.109 | -1.5 | GC-Orbitrap | 0.575 | -0.1 | GC-Orbitrap | 1.252 | -0.2 |
| 54 | 0.192 | 0.4 | GC-MS/MS (QQQ) | 0.535 | -0.4 | GC-MS/MS (QQQ) | 1.256 | -0.2 |
| 77 | 0.143 | -0.7 | GC-MS/MS (QQQ) | 0.561 | -0.2 | GC-MS/MS (QQQ) | 1.259 | -0.2 |
| 78* | 0.183 | 0.2 | GC-MS/MS (QQQ) | 0.548 | -0.3 | GC-MS/MS (QQQ) | 1.273 | -0.1 |
| 44 | 0.218 | 1.0 | GC-MS/MS (QQQ) | 0.531 | -0.4 | GC-MS/MS (QQQ) | 1.274 | -0.1 |

**Table 4-17 (cont.):** Compilation of laboratory results of captan, THPI and the sum thereof expressed as captan. The latter was calculated by the organisers. The z scores of the sum are for information only. Where laboratories have not analysed for both components, the lab code, the result for the sum as well as the z score for the sum are highlighted in red.

| Lab-Code SRM17- | Captan | | | THPI | | | Captan (sum) (Captan + THPI* (expr. as captan)) | |
|---|---|---|---|---|---|---|---|---|
| | Conc. [mg/kg] | z score | Determ. Techn. | Conc. [mg/kg] | z score | Determ. Techn. | Conc. [mg/kg] | z score |
| 69 | 0.189 | 0.3 | GC-MSD | 0.547 | -0.3 | GC-MS/MS (QQQ) | 1.277 | -0.1 |
| 20 | 0.218 | 1.0 | GC-MS/MS (QQQ) | 0.536 | -0.4 | GC-MS/MS (QQQ) | 1.284 | -0.1 |
| 71 | 0.245 | 1.6 | GC-MS/MS (QQQ) | 0.524 | -0.4 | GC-MS/MS (QQQ) | 1.287 | -0.1 |
| 89 | 0.198 | 0.6 | GC-MS/MS (QQQ) | 0.549 | -0.3 | GC-MS/MS (QQQ) | 1.290 | -0.1 |
| 109 | 0.046 | -2.9 | GC-MS/MS (QQQ) | 0.63 | 0.3 | GC-MS/MS (QQQ) | 1.299 | -0.1 |
| 79 | 0.238 | 1.5 | GC-MS/MS (QQQ) | 0.541 | -0.3 | GC-MS/MS (QQQ) | 1.314 | 0.0 |
| 96* | 0.178 | 0.1 | GC- (μ) ECD | 0.576 | -0.1 | LC-MS/MS (QQQ) | 1.323 | 0.0 |
| 15 | 0.149 | -0.6 | GC-MS/MS (QQQ) | 0.594 | 0.0 | GC-MS/MS (QQQ) | 1.330 | 0.0 |
| 28* | 0.150 | -0.6 | GC-MS/MS (QQQ) | 0.594 | 0.0 | GC-MS/MS (QQQ) | 1.331 | 0.0 |
| 70 | 0.242 | 1.6 | GC-MS/MS (QQQ) | 0.556 | -0.2 | GC-MS/MS (QQQ) | 1.348 | 0.1 |
| 67 | 0.110 | -1.5 | GC- (μ) ECD | 0.627 | 0.3 | GC-MS/MS (QQQ) | 1.357 | 0.1 |
| 95 | | | | 0.699 | 0.7 | GC-MS/MS (QQQ) | 1.390 | 0.2 |
| 102 | 0.182 | 0.2 | GC-MS/MS (QQQ) | 0.611 | 0.1 | GC-MS/MS (QQQ) | 1.397 | 0.2 |
| 56* | 1.402 | 28.2 | GC-MS/MS (QQQ) | | | | 1.402 | 0.3 |
| 47 | 0.147 | -0.6 | LC-MS/MS (QQQ) | 0.631 | 0.3 | LC-MS/MS (QQQ) | 1.402 | 0.3 |
| 72* | 0.135 | -0.9 | GC-MS/MS (QQQ) | 0.638 | 0.3 | GC-MS/MS (QQQ) | 1.404 | 0.3 |
| 36* | 0.193 | 0.4 | GC-MS/MS (QQQ) | 0.609 | 0.1 | GC-MS/MS (QQQ) | 1.404 | 0.3 |
| 22 | 0.151 | -0.5 | GC-MS/MS (QQQ) | 0.639 | 0.3 | GC-MS/MS (QQQ) | 1.422 | 0.3 |
| 73 | FN | -3.5 | GC-MS/MS (QQQ) | 0.721 | 0.9 | GC-MS/MS (QQQ) | 1.434 | 0.4 |
| 16 | 0.153 | -0.5 | GC- (μ) ECD | 0.645 | 0.4 | GC-MS/MS (QQQ) | 1.436 | 0.4 |
| 37 | 0.159 | -0.3 | GC-MS/MS (QQQ) | 0.648 | 0.4 | GC-MS/MS (QQQ) | 1.447 | 0.4 |
| 106 | FN | -3.5 | GC-MS/MS (QQQ) | 0.744 | 1.0 | GC-MS/MS (QQQ) | 1.479 | 0.5 |
| 53 | 0.220 | 1.1 | GC-MS/MS (QQQ) | 0.636 | 0.3 | GC-MS/MS (QQQ) | 1.485 | 0.5 |
| 10 | 0.076 | -2.3 | GC-MS/MS (QQQ) | 0.712 | 0.8 | GC-MS/MS (QQQ) | 1.492 | 0.5 |
| 64* | 0.187 | 0.3 | GC-MS/MS (QQQ) | 0.660 | 0.5 | LC-MS/MS (QQQ) | 1.499 | 0.5 |
| 76 | 0.0170 | -3.6 | GC-MS/MS (QQQ) | 0.752 | 1.1 | GC-MS/MS (QQQ) | 1.512 | 0.6 |
| 97 | 0.128 | -1.1 | GC-MS/MS (QQQ) | 0.697 | 0.7 | GC-MS/MS (QQQ) | 1.514 | 0.6 |
| 88 | 0.279 | 2.4 | GC-MS/MS (QQQ) | 0.621 | 0.2 | LC-MS/MS (QQQ) | 1.514 | 0.6 |
| 11 | 0.208 | 0.8 | GC-MS/MS (QQQ) | 0.677 | 0.6 | GC-MS/MS (QQQ) | 1.554 | 0.7 |
| 4* | 0.199 | 0.6 | GC- (μ) ECD | 0.690 | 0.7 | GC-Ion Trap | 1.571 | 0.8 |
| 48 | 0.180 | 0.1 | GC-MS/MS (QQQ) | 0.700 | 0.7 | GC-MS/MS (QQQ) | 1.572 | 0.8 |
| 58* | 0.210 | 0.8 | GC-MS/MS (QQQ) | 0.688 | 0.7 | GC-MS/MS (QQQ) | 1.578 | 0.8 |
| 111 | 0.19 | 0.4 | GC-MSD | 0.70 | 0.7 | GC-MS/MS (QQQ) | 1.582 | 0.8 |
| 51 | 0.184 | 0.2 | GC-MS/MS (QQQ) | 0.705 | 0.8 | GC-MS/MS (QQQ) | 1.586 | 0.8 |
| 68* | 0.158 | -0.4 | GC-MS/MS (QQQ) | 0.720 | 0.9 | GC-MS/MS (QQQ) | 1.590 | 0.8 |
| 41 | 0.063 | -2.6 | GC-MSD | 0.77 | 1.2 | GC-MSD | 1.594 | 0.8 |
| 35* | 0.198 | 0.6 | GC-TOF-MS (unit resol.) | 0.706 | 0.8 | GC-TOF-MS (unit resol.) | 1.602 | 0.9 |
| 74 | 0.15 | -0.6 | GC-MS/MS (QQQ) | 0.75 | 1.1 | GC-MS/MS (QQQ) | 1.641 | 1.0 |
| 13 | FN | -3.8 | GC-MS/MS (QQQ) | 0.8506 | 1.8 | GC-MS/MS (QQQ) | 1.691 | 1.1 |
| 99 | 0.086 | -2.0 | GC-MSD | 0.817 | 1.5 | GC-MS/MS (QQQ) | 1.711 | 1.2 |
| 18* | 0.179 | 0.1 | GC- (μ) ECD | 0.790 | 1.4 | LC-MS/MS (QQQ) | 1.750 | 1.3 |
| 105 | 0.17 | -0.1 | GC-MS/MS (QQQ) | 0.83 | 1.6 | GC-MS/MS (QQQ) | 1.820 | 1.5 |
| 120 | 0.03 | -3.3 | GC-MS/MS (QQQ) | 0.941 | 2.4 | GC-MS/MS (QQQ) | 1.901 | 1.8 |
| 110 | FN | -3.8 | LC-MS/MS (QQQ) | 1.001 | 2.8 | GC-MS/MS (QQQ) | 1.990 | 2.0 |
| 90 | 1.450 | 29.3 | GC-MS/MS (QQQ) | 0.720 | 0.9 | GC-MS/MS (QQQ) | 2.882 | 4.7 |
| 42 | 2.2 | 46.6 | GC-MS/MS (QQQ) | 0.65 | 0.4 | GC-MS/MS (QQQ) | 3.492 | 6.6 |
| 128 | 1.437 | 29.0 | GC-MS/MS (QQQ) | 1.437 | 5.7 | GC-MS/MS (QQQ) | 4.294 | 9.0 |

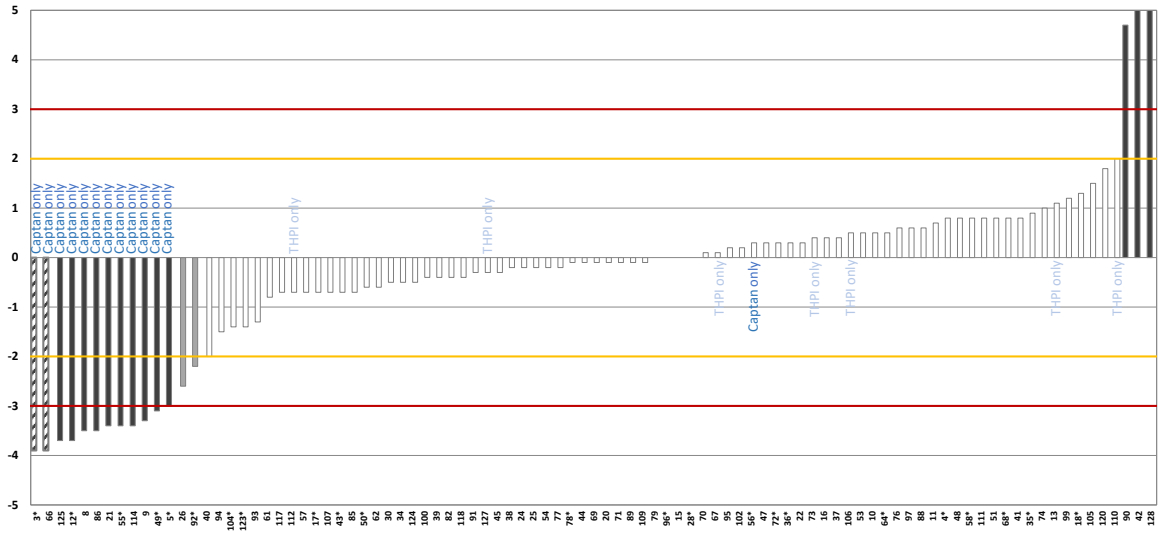



**Figure 4-3:** Graphic presentation of z scores achieved by participating laboratories (the robust mean for captan (sum) shown in **Table 4-15 (p. 55)** was used as reference value for z-score calculation). Laboratories having reported only for one of the two components are highlighted.

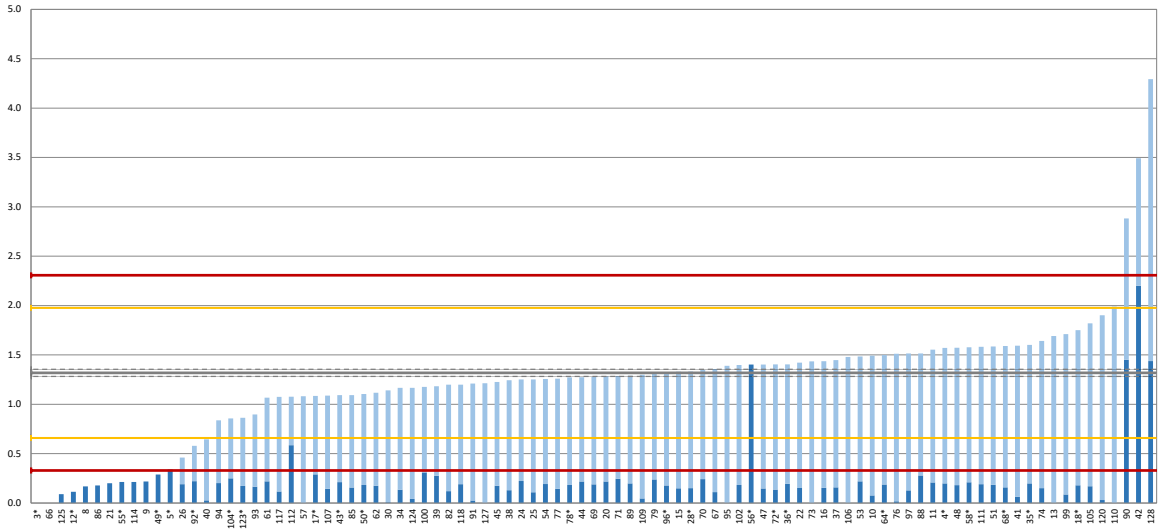



**Figure 4-4:** Graphic presentation captan (sum) concentrations as caluclated by the organisers using the results for captan and THPI. The share of results of captan reported by the participants are shown in dark blue. The share of the reported THPI results (expressed as captan) are shown in pale blue.

In contrast to the results of ***phthalimide***, the results of ***THPI*** were more narrowly distributed. This is due to the much higher mol-ratio between parent and degradant in the case of ***folpet*** / ***phthalimide*** (1.26) compared to ***captan*** / ***THPI*** (0.15). The higher this ratio is (i.e. the more predominant the parent component is), the higher the share of the degradant formed during GC-injection and the higher the risk of an overestimation of the degradant. In the case of ***captan,*** the degradant formed during injection was negligible compared to the degradant originally present in the extracts.

Degradation of analytical standards is another source for errors. Overestimated results for the parents may be the outcome in this case. If mixed calibrations standards of parent and degradants are used, the calibration standards will contain more degradant than they should and the concentration of the degradants in the samples will be underestimated. Degradation of the parents are most prominent in standards prepared in acetonitrile. To avoid degradation acetonitrile standards need to be acidified. The need for acidification has lost significance in the last few years due to a change in the quality of commercially available acetonitrile, which is nowadays typically not as alkaline as in the past.

Another aspect which may have strong impact on the results of ***captan*** / ***THPI*** and ***folpet*** / ***phthalimide*** is the decomposition of the parents within thawed sample homogenates. Despite the advice to the participants to keep the samples frozen till analysis, many participants have let their samples thaw. Looking at the methodological data submitted, there is a clear trend towards underestimations for labs having left the test item thaw. The impact was more pronounced in the case of ***captan***. Due to the multifactorial impact on analytical results this trend is, however, not fully conclusive for all labs. But experiments run by the organisers using the sample homogenate have clearly demonstrated a progressive conversion of ***captan*** to ***THPI*** and of ***folpet*** to ***phthalimide*** as the tomato homogenates containing ***folpet*** and ***captan*** were left standing over several hours at room temperature (see **Section 1.5, p. 2**). The conversion yields of ***captan*** and ***folpet*** to ***THPI*** and ***phthalimide***, respectively, were very high, which means that the results for the summed residue are only minimally affected by degradations in homogenates (see also evaluation of summed concentrations, **p. 54**). Furthermore, due to the shift in the parent / degradant ratio the risk of overestimation of the degradant concentration in GC analysis decreases.

As ***captan*** and ***folpet*** behave very similarly during analysis, it was checked whether the results of the participants followed similar trends as regards over- or underestimations. **Figure 4-5** shows that this was largely the case. In the majority of the cases the results for ***captan*** and ***folpet*** correlated well with each other. 36 out of the 91 laboratories having reported results for both ***captan*** and ***folpet*** showed clear trends towards a signifficant positive or negative deviation for these two analytes. Most other laboratories showed either a less signifficant trends or no trend. Only very few labs achieved z scores deviating strongly towards opposite directions.

### Analytical Challenge in case of Meptyldinocap & Co.

In principle, there are two approaches for the analysis: a) analysis of ***meptyldinocap*** and ***2,4-DNOP*** separately; and b) Analysis following conversion of ***meptyldinocap*** to ***2,4-DNOP***. The analysis of ***meptyldinocap*** next to its degradant ***2,4-DNOP*** is tricky as ***meptyldinocap*** is also analysed as ***2,4-DNOP*** as it readily degrades within the ESI ion source. So both compounds share the same MRM transitions and are not separated mass-spectrometrically. To avoid partial co-elution of the two compounds, it is recommended running comparably "slow" LC-gradients to ensure sufficient chromatographic separation. ***Meptyldinocap*** is sensitive to degradation and analytical standards need to be stabilized with some acid. Still, even freshly prepared ***meptyldinocap*** standards contain a small amount of ***2,4-DNOP*** (e.g. ~3 %). Despite the small share of ***2,4-DNOP*** in ***meptyldinocap*** standards, ***2,4-DNOP*** forms the largest peak upon injection. The reason for this is that ***2,4-DNOP*** when analysed as such is ca. 50 to 100-fold more sensitive than ***2,4-DNOP*** originating

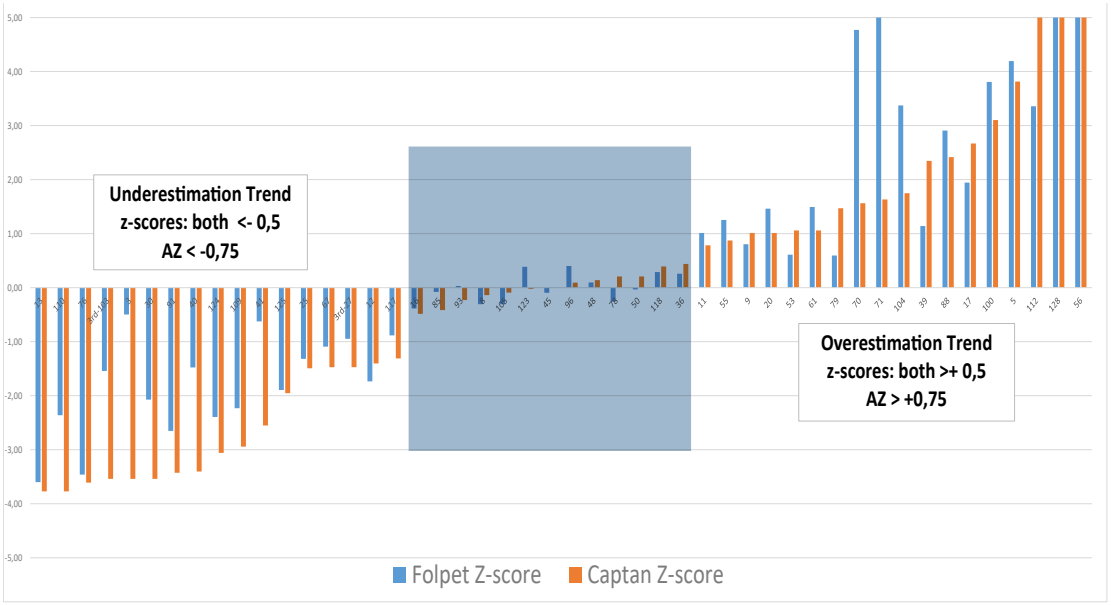



**Figure 4-5:** Bias-Correlation between captan and folpet. Folpet results shown as blue columns and captan results as green columns.

from ***meptyldinocap*** through in-source fragmentation which appears at a later retention time. Laboratories often get confused misallocating the retention time of ***2,4-DNOP*** to ***meptyldinocap***. This leads to wrong quantifications. Even if retention times are correctly allocated, it can happen that the peak at the retention time of ***meptyldinocap*** is overlooked as the peak for ***2,4-DNOP*** is typically much-much larger, so that the ***2,4-DNOP*** peak is taken. This scenario may happen if the two compounds elute very closely ("fast" elution gradient) so that ***2,4-DNOP*** appears close to the centre of the data review window of ***meptyldinocap***.

A method entailing conversion of ***meptyldinocap*** to ***2,4-DNOP*** was developed by the EURL-SRM (Method SRM-47).

### Challenges in Analysis of Chloridazon-Desphenyl

Being highly polar, ***chloridazon-desphenyl*** shows poor absolute recovery rates using multiresidue methods, such as QuEChERS. Correction for bias may be accomplished using the isotope-labelled analogue of ***chloridazon-desphenyl*** as internal standard (ILIS). Using QuPPe-style methods, the absolute recovery is sufficient, but care must be taken to correct for matrix effects. The use of ILIS is also helpful in this case. Problematic in the analysis of ***chloridazon-desphenyl*** is that the qualifier mass-transitions show poor sensitivity which leads to problems with interferences, thus impacting quantification and identification. According to the observations by the organisers, interferences on the qualifier peaks of ***chloridazon-desphenyl*** were rather moderate in the particular sample type and analyte concentration of the PT, but chromatographic separation and detection sensitivities vary from lab to lab. Another problem that was noticed in the PT was that some laboratories confused ***chloridazon-desphenyl*** with chloridazon (parent), reporting false negative results.

Information about the analysis of ***chloridazon-desphenyl*** was published by the EURL-SRM (Document SRM-51)

### Challenges in Analysis of Bifenazate (sum)

***Bifenazate*** is an oxidizable compound with ***bifenazate*** diazene being the oxidized form. The reaction is fully reversible. Depending on the matrix ***bifenazate*** may be mainly present in its reduced form (***bifenazate***) or mainly present in its oxidized form (***bifenazate diazene***). Redox reactions may also take place in extracts or matrix-based calibration standards, which leads to concentration shifts and quantification errors. The safest approach to ensure acurate quantification is to ensure that ***bifenazate*** is present in one of its two forms prior to injection. A method entailing simple conversion of ***bifenazate*** diazene to ***bifenazate***, through addition of ascorbic acid into autosampler vials containing sample extracts and calibration solutions, was published by the EURL-SRM (Method SRM-34).

### Challenges in Analysis of Pymetrozine

***Pymetrozine*** has a pH-dependent polarity and is too polar for QuEChERS-type methods at pH values < 6. There is currently no ILIS available for this analyte. A QuEChERS-based method for the analysis of ***pymetrozine***, involving partitioning at higher pH, was developped by the EURL-SRM (Document SRM-42)

### Challenges in the Analysis of Formetanate

***Formetanate*** is prone to degradation at various stages of the procedure. If degradation takes place during extraction or within the final extract it may lead to underestimated results or even false negatives. In EUPT-SRM17, five labs reported false negative results and additional three labs reported results with a z score < –2. A degradation of ***formetanate*** taking place within the analytical standard solution or calibration standards may, in contrast, lead to overestimated results. In total, 14 laboratories obtained z scores > 2 and 10 laboratories z scores > 3. Twelve of these 14 labs obtaining z scores > 2 reported that they had observed degradation of ***formetanate*** within the standard solution used for calibration.

### Challenges in the Analysis of Emamectin

***Emamectin*** tends to attach to surfaces, which may lead to underestimated calibration curves and overestimated results. Most affected are low-concentrated solutions. Among the six labs having obtained z scores > 3, five labs reported losses in the analytical standards used for calibration. The remaining lab reported a strongly overestimated recovery rate, which also points in the same direction. Preparing calibration solutions in plastic rather than glass vials and the use of blank matrix extracts for preparing the calibration solutions minimizes this effect.

# 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 developing and updating the webtool for the present PT and supported the PT organisers, whenever they needed help regarding this 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/EurlSRM/EurlSrm_meth_QuPPe_PO_V12_1.pdf

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

[7] https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/meth_Bifenazate_EurlSRM.pdf

[8] https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EurlSrm_Observation_Meptyldinocap_V1_2.pdf

[9] https://www.eurl-pesticides.eu/library/docs/srm/meth_DithiocarbamatesCs2_EurlSrm.pdf

# 7. APPENDICES

## Appendix 1 List of Laboratories Registered to Participate in the EUPT-SRM17

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

| Country (Location) | Analysed on behalf of | Institution | City | NRL |
|---|---|---|---|---|
| Austria | AT | AGES - Innsbruck | Innsbruck | x |
| Belgium | BE | LOVAP NV - Belgium, Geel | Geel | – |
| Belgium | BE | Sciensano - Pesticide Lab | Brussels | x |
| Belgium | BE, LU, FR | PRIMORIS (Phytolab) - Belgium, Gent | Gent - Zwijnaarde | – |
| Bulgaria | BG | CLCTC - Sofia \| Pesticide Lab | Sofia | x |
| Bulgaria | BG | Primoris - Bulgaria, Plovdiv | Plovdiv | – |
| Croatia | HR | Bioinstitut d.o.o., Cakovec | Cakovec | – |
| Croatia | HR | Dr. Andrija Štampar - Pesticide Lab | Zagreb | x |
| Croatia | HR | Eurofins Croatiakontrola | Zagreb | – |
| Croatia | HR | INSPECTO d.o.o. Laboratorij (Osijek) | Osijek | – |
| Croatia | HR | Sample Control - Pesticide Lab | Lučko | – |
| Cyprus | CY | SGL - Pesticide Lab (Nicosia) | Nicosia | x |
| Czech Republic | CZ | CAFIA - Pesticide Lab (Praha) | Praha | x |
| Czech Republic | CZ | 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 |
| Finland | FI | MetropoliLab - Pesticide Lab | Helsinki | – |
| 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 | Laboratoire SCL de PARIS | Massy Cedex | – |
| France | FR | SCL (Montpellier) | Montpellier | – |
| France | BE | Phytocontrol (Nimes) - Pesticide Lab | Nimes | – |
| Germany | FR | Intertek Food Services - Bremen | Bremen | – |
| Germany | BE | AGROLAB LUFA Kiel - Pesticide Lab | Kiel | – |
| Germany | DE | Analytica Alimentaria GmbH - Lab (Kleinmachnow) | Kleinmachnow | – |
| Germany | DE | Bundeswehr - Pesticide Lab (Garching-Hochbrück) | Garching-Hochbrück | – |
| 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 | Gesellschaft für Bioanalytik Hamburg | Hamburg | – |
| Germany | DE | IHU - Pesticide Lab (Hamburg) | Hamburg | – |
| Germany | DE | KWALIS Fulda - Pesticide Lab | Dipperz | – |
| Germany | DE | Labor Friedle - Germany, Tegernheim | Tegernheim | – |
| Germany | DE | Labor Mang - Pesticide Lab | Frankfurt | – |
| Germany | DE | LALLF - Pesticide Lab (Rostock) | Rostock | – |
| Germany | DE | Landesamt für Verbraucherschutz, Halle/Saale | Halle/Saale | – |
| Germany | DE | Landeslabor Berlin-Brandenburg, Frankfurt (Oder) | Frankfurt (Oder) | – |

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

| Country (Location) | Analysed on behalf of | Institution | City | NRL |
|---|---|---|---|---|
| Germany | DE | Landeslabor Schleswig-Holstein, Neumünster | Neumünster | – |
| Germany | DE | LAVES - Pesticide Lab (Oldenburg) | Oldenburg | – |
| Germany | DE | LGL Erlangen - Pesticide Lab | Erlangen | – |
| Germany | DE | LHL - Pesticide Lab (Kassel) | Kassel | – |
| Germany | DE | LTZ Augustenberg - Organic Analysis | Karlsruhe | – |
| Germany | DE | LUA Rheinland-Pfalz, Institut für LM-Chemie Speyer | Speyer | – |
| Germany | DE | LUA Sachsen - Pesticide Lab, Dresden | Dresden | – |
| Germany | DE | LUFA Speyer | Speyer | – |
| Germany | LT | GALAB Laboratories GmbH - Hamburg | Hamburg | – |
| Germany | MT | Eurofins - Germany, Hamburg | Hamburg | – |
| Germany | MT | Eurofins Dr. Specht Express GmbH - Hamburg | Hamburg | – |
| Greece | GR | Benaki Phytopathological Institute, Kifissia | Kifissia | x |
| Greece | GR | GCSL - Pesticide Lab (Athens) | Athens | x |
| Hungary | HU | FCSCN Ltd Pesticide Res. Anal. Lab. Miskolc | Miskolc | – |
| Hungary | HU | NFCSO - Pesticide Lab (Velence) | Velence | x |
| Hungary | HU | NFCSO Pesticide Lab (Hódmezovásárhely) | Hódmezovásárhely | – |
| Hungary | HU | NFCSO Pesticide Lab (Szolnok) | Szolnok | – |
| Ireland | IE | The Food Chemistry Laboratories - DAFM | Co. Kildare | x |
| Italy | IT | APPA Bolzano - Pesticide Lab | Bolzano | – |
| Italy | IT | APPA-Puglia \| Polo Alimenti Bari - Pesticide Lab | Bari | – |
| Italy | IT | ARPA-ER - Pesticide Lab | Ferrara | – |
| Italy | IT | Azienda Sanitaria Locale di Firenze | Firenze | – |
| Italy | IT | Istituto Superiore di Sanità - Roma | Roma | x |
| Italy | IT | IZS LT - Italy, Rome | Roma | – |
| Italy | IT | IZSAM - Pesticide Lab | Teramo | – |
| Italy | IT | IZSLER - Pesticide Lab | Brescia | – |
| Italy | IT | Laboratorio di Prevenzione (Bergamo) | Bergamo | – |
| Latvia | LV | BIOR (Riga) - Pesticide Lab | Riga | x |
| Lithuania | LT | NMVRVI - Pesticide Lab (Vilnius) | Vilnius | x |
| Luxembourg | LU | LNS Food lab | Dudelange | x |
| Norway | NO | NIBIO - Department of Pesticide Chemistry | ÅS | x |
| Poland | PL | AGROLAB Polska - Poland, Deblin | Deblin | – |
| Poland | PL | Hamilton UO-Technologia | Grójec | – |
| Poland | PL | InHort (Skierniewice) - Pesticide Lab | Skierniewice | – |
| Poland | PL | IPP-NRI - Pesticide Lab (Poznan) | Poznan | – |
| Poland | PL | IPP-NRI - Pesticide Lab (Sosnicowice) | Sosnicowice | – |
| Poland | PL | Laboratory of Food & Feed Safety in Bialystok | Bialystok | – |
| Poland | PL | PIORIN - Central Laboratory (Torun) | Torun | – |
| Poland | PL | VSES Lodz - Pesticide Lab | Lodz | – |
| Poland | PL | VSES Opole - Pesticide Lab | Opole | – |
| Poland | PL | VSES Warszawa - Pesticide Lab | Warszaw | x |
| Poland | PL | VSES Wroclaw - Pesticide Lab | Wroclaw | – |
| Poland | PL | WSSE - Poland, Bydgoszcz | Bydgoszcz | – |
| Portugal | PT | Pesticide Lab (Funchal - Madeira Island) | Funchal - Madeira Island | x |
| Romania | RO | IISPV (Bucharest) - Pesticide Lab | Bucharest | x |
| Romania | RO | NATIONAL PHITOSANITARY AUTHORITY | Bucharest | – |
| Slovakia | SK | State Veterinary and Food Institute (Bratislava) | Bratislava | x |
| Slovenia | SI | Pesticide Lab - Maribor | Maribor | x |
| Spain | ES | Agricultural and Phytopathological Lab. of Galicia | Abegondo. A Coruña | – |

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

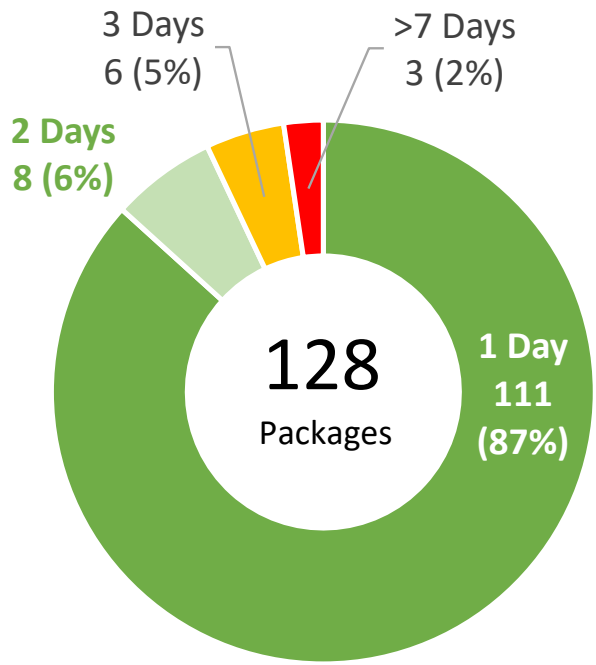
| Country (Location) | Analysed on behalf of | Institution | City | NRL |
|---|---|---|---|---|
| Spain | ES | Ainia (Valencia) | Valencia | – |
| Spain | ES | Analytica Alimentaria GmbH - Almeria, Spain | Almeria | – |
| Spain | ES | EUROFINS ECOSUR - Pesticide Lab | LORQUI - MURCIA | – |
| Spain | ES | Eurofins SiCA AgriQ - Almeria, Vícar | Almeria | – |
| Spain | ES | Lab. Agrario Regional - Junta de Castilla y Leon | Burgos | – |
| Spain | ES | Laboratori Agència Salut Pública Barcelona | Barcelona | – |
| Spain | ES | Laboratori Agroalimentari - Generalitat Valenciana | Burjassot, Valencia | – |
| Spain | ES | Laboratorio Agroalimentario de Extremadura | Cáceres | – |
| Spain | ES | Laboratorio Agroambiental de Zaragoza | Zaragoza | – |
| Spain | ES | Laboratorio Analítico Bioclínico - Spain, Almeria | Almeria | – |
| Spain | ES | Laboratorio Arbitral Agroalimentario, Madrid | Madrid | x |
| Spain | ES | Laboratorio de Residuos, Inst. Tecnol. de Canarias | Agüimes, Gran Canaria | – |
| Spain | ES | National Center for Technology and Food Safety | San Adrián (Navarra) | – |
| Spain | ES | National Centre for Food (Majadahonda) | Majadahonda | x |
| Spain | ES | SALUD PUBLICA (LSP - MADRID SALUD) | Madrid | – |
| Spain | PT | Labs & Technological Services AGQ - Burguillos | Burguillos | – |
| Sweden | SE | Eurofins Food & Feed - Pesticide Lab (Lidköping) | Lidköping | – |
| Sweden | SE | National Food Agency - Sweden, Uppsala | Uppsala | x |
| Switzerland | CH | Amt für Verbraucherschutz Aargau | Aargau | – |
| Switzerland | CH | Kantonales Laboratorium Bern | Bern | – |
| Switzerland | CH | Kantonales Laboratorium Zürich | Zürich | – |
| The Netherlands | BE | Groen Agro Control - Netherlands | Delfgauw | – |
| 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 |
|---|---|---|
| Korea, Republic of | Korea Institute of Toxicology (KIT) - Jinju | JINJU |
| Serbia | Field Test - Serbia, Belgrade | Belgrade |
| Serbia | SP Laboratorija - Pesticide Lab | BECEJ |
| Thailand | Central Laboratory - Pesticide Lab (Bangkok) | Bangkok |
| United Kingdom | FERA - Pesticide Lab | York |

## Appendix 2 Shipment Evaluation

**Compilation of shipment duration**




- **Within 2 days: 93 %**
- **Within 3 days:**
  1 EU remote Location
  1 EU Candidate Country
  4 to Spain due to thunderstorm (2 days delay at DHL Leipzig)
- **7 and 9 days:** EU Candidate and 3rd Countries only
  TH (7 days): was kept frozen at local DHL
  RS (7 days): thawed, but still cold
  KR (9 days): thawed, slightly cool

## Appendix 3 Data of Homogeneity Test

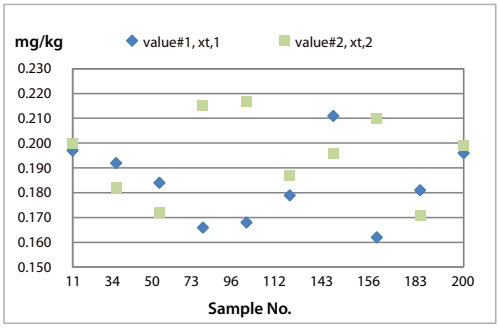
### Compulsory Compounds

| | Captan | | Chlorothalonil | | Cyromazine | | Dithiocarbamates ($CS_2$) | | Dodine | |
|---|---|---|---|---|---|---|---|---|---|---|
| Sample No. | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] |
| 011 | 0.197 | 0.200 | 0.173 | 0.177 | 0.134 | 0.138 | 0.211 | 0.214 | 0.110 | 0.113 |
| 034 | 0.192 | 0.182 | 0.166 | 0.164 | 0.139 | 0.137 | 0.218 | 0.222 | 0.109 | 0.108 |
| 050 | 0.184 | 0.172 | 0.169 | 0.166 | 0.146 | 0.138 | 0.215 | 0.237 | 0.098 | 0.107 |
| 073 | 0.166 | 0.215 | 0.165 | 0.160 | 0.138 | 0.136 | 0.189 | 0.210 | 0.111 | 0.112 |
| 096 | 0.168 | 0.217 | 0.153 | 0.170 | 0.140 | 0.136 | 0.211 | 0.214 | 0.107 | 0.114 |
| 112 | 0.179 | 0.187 | 0.167 | 0.184 | 0.135 | 0.134 | 0.208 | 0.221 | 0.105 | 0.105 |
| 143 | 0.211 | 0.196 | 0.173 | 0.171 | 0.142 | 0.137 | 0.221 | 0.235 | 0.106 | 0.112 |
| 156 | 0.162 | 0.210 | 0.176 | 0.171 | 0.138 | 0.137 | 0.210 | 0.217 | 0.103 | 0.109 |
| 183 | 0.181 | 0.171 | 0.156 | 0.165 | 0.135 | 0.134 | 0.221 | 0.222 | 0.103 | 0.109 |
| 200 | 0.196 | 0.199 | 0.169 | 0.168 | 0.130 | 0.138 | 0.216 | 0.229 | 0.111 | 0.108 |
| mean / AV* | 0.189 (0.172) | | 0.168 (0.151) | | 0.137 (0.154) | | 0.217 (0.187) | | 0.108 (0.100) | |

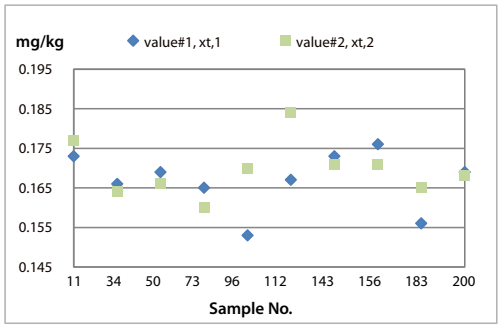
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Graphical presentation of the results:

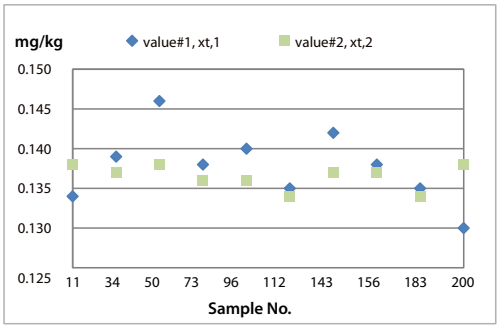
**Captan**



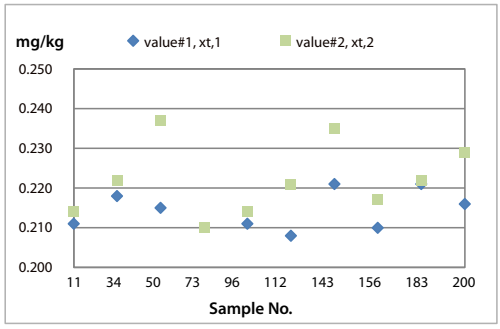

**Chlorothalonil**



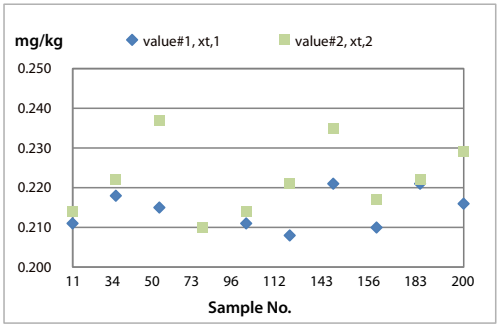

**Cyromazine**




**Dithiocarbamates (expr. as $CS_2$)**




**Dodine**



## Appendix 3 (cont.): Data of Homogeneity Test

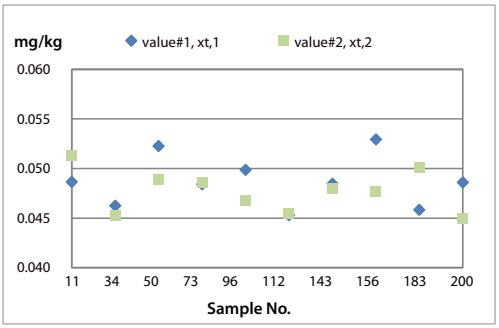
### Compulsory Compounds

| | Emamectine B1a | | Folpet | | Phthalimide | | Pymetrozine | | THPI | |
|---|---|---|---|---|---|---|---|---|---|---|
| Sample No. | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] |
| 011 | 0.049 | 0.051 | 0.315 | 0.314 | 0.087 | 0.077 | 0.179 | 0.196 | 0.617 | 0.604 |
| 034 | 0.046 | 0.045 | 0.320 | 0.301 | 0.080 | 0.071 | 0.188 | 0.195 | 0.618 | 0.590 |
| 050 | 0.052 | 0.049 | 0.277 | 0.323 | 0.076 | 0.072 | 0.201 | 0.193 | 0.648 | 0.628 |
| 073 | 0.048 | 0.049 | 0.310 | 0.309 | 0.072 | 0.075 | 0.196 | 0.193 | 0.623 | 0.565 |
| 096 | 0.050 | 0.047 | 0.277 | 0.314 | 0.066 | 0.075 | 0.192 | 0.184 | 0.634 | 0.624 |
| 112 | 0.045 | 0.045 | 0.301 | 0.303 | 0.078 | 0.071 | 0.191 | 0.190 | 0.640 | 0.582 |
| 143 | 0.048 | 0.048 | 0.254 | 0.309 | 0.079 | 0.082 | 0.197 | 0.196 | 0.654 | 0.629 |
| 156 | 0.053 | 0.048 | 0.292 | 0.294 | 0.074 | 0.078 | 0.181 | 0.194 | 0.571 | 0.620 |
| 183 | 0.046 | 0.050 | 0.285 | 0.308 | 0.074 | 0.073 | 0.190 | 0.190 | 0.633 | 0.569 |
| 200 | 0.049 | 0.045 | 0.294 | 0.309 | 0.076 | 0.080 | 0.189 | 0.192 | 0.638 | 0.593 |
| mean / AV* | 0.048 (0.046) | | 0.300 (0.249) | | 0.076 (0.134) | | 0.191 (0.150) | | 0.614 (0.590) | |

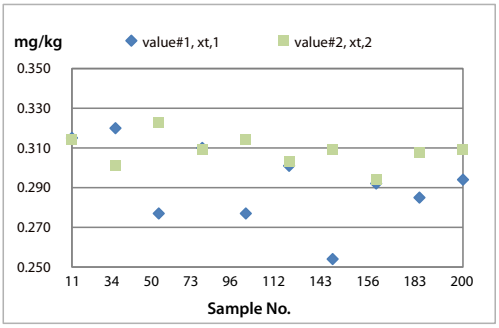
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Graphical presentation of the results:

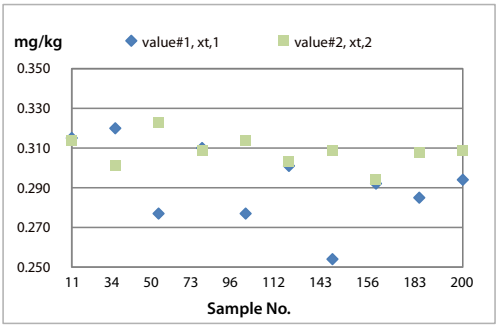
**Emamectin B1a**



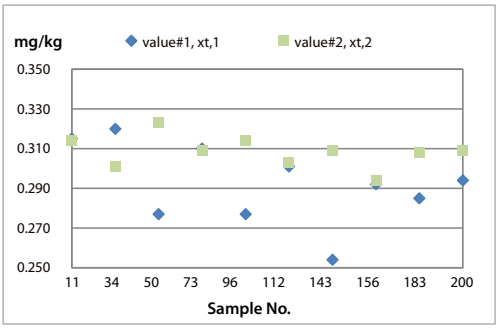

**Folpet**



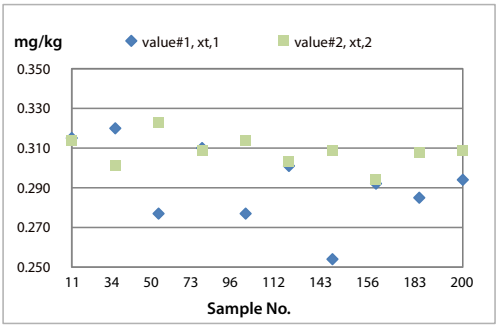

**Phthalimide**




**Pymetrozine**




**THPI**



## Appendix 3 (cont.): Data of Homogeneity Test

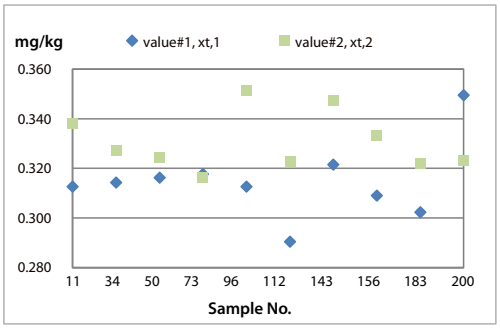
**Optional Compounds**

| | Bifenazate (sum) | | Chloridazon-desphenyl | | ETU | | Formetanate-HCl | | Maleic hydrazide | |
|---|---|---|---|---|---|---|---|---|---|---|
| Sample No. | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] | Portion 1 [mg/kg] | Portion 2 [mg/kg] |
| 011 | 0.313 | 0.338 | 0.052 | 0.052 | 0.057 | 0.053 | 0.861 | 0.777 | 0.482 | 0.590 |
| 034 | 0.314 | 0.327 | 0.049 | 0.056 | 0.056 | 0.055 | 0.836 | 0.830 | 0.514 | 0.595 |
| 050 | 0.316 | 0.324 | 0.056 | 0.052 | 0.060 | 0.057 | 0.852 | 0.844 | 0.591 | 0.528 |
| 073 | 0.318 | 0.316 | 0.052 | 0.051 | 0.050 | 0.054 | 0.836 | 0.867 | 0.506 | 0.507 |
| 096 | 0.313 | 0.351 | 0.055 | 0.052 | 0.056 | 0.059 | 0.801 | 0.832 | 0.494 | 0.555 |
| 112 | 0.290 | 0.323 | 0.055 | 0.051 | 0.055 | 0.053 | 0.861 | 0.833 | 0.592 | 0.534 |
| 143 | 0.322 | 0.347 | 0.050 | 0.055 | 0.060 | 0.060 | 0.880 | 0.856 | 0.555 | 0.546 |
| 156 | 0.309 | 0.333 | 0.055 | 0.058 | 0.053 | 0.057 | 0.820 | 0.843 | 0.458 | 0.539 |
| 183 | 0.302 | 0.322 | 0.054 | 0.054 | 0.059 | 0.057 | 0.838 | 0.818 | 0.536 | 0.491 |
| 200 | 0.350 | 0.323 | 0.051 | 0.055 | 0.057 | 0.063 | 0.846 | 0.866 | 0.543 | 0.591 |
| mean / AV* | 0.323 (0.296) | | 0.053 (0.061) | | 0.057 (0.063) | | 0.840 (0.873) | | 0.537 (0.544) | |

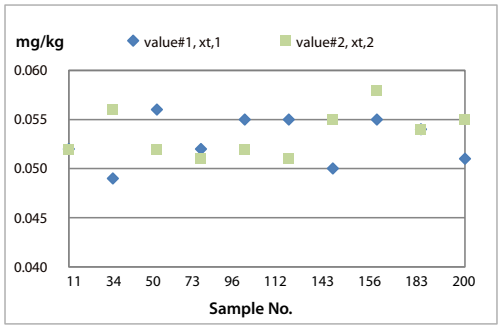
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Graphical presentation of the results:

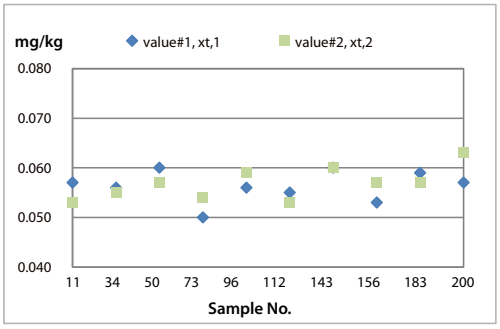
**Bifenazate (sum)**



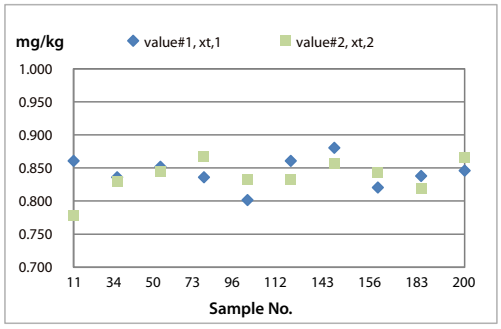

**Chloridazon-desphenyl**



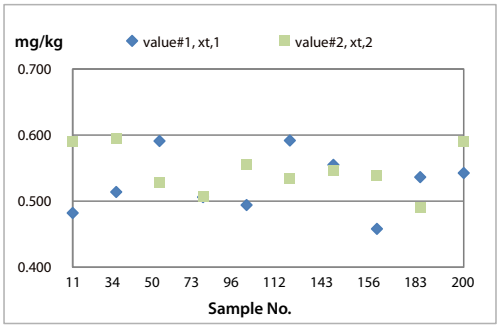

**ETU**




**Formetanate-HCl**




**Maleic hydrazide**



## Appendix 3 (cont.): Data of Homogeneity Test

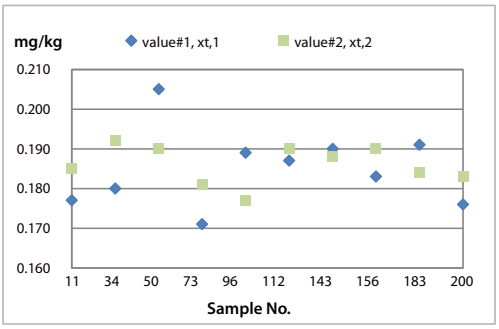
**Optional Compounds**

| | Oxymatrine | | 2,4-DNOP (free phenol) | | Meptyldinocap | | Meptyldinocap (sum) | |
|---|---|---|---|---|---|---|---|---|
| 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] |
| 011 | 0.177 | 0.185 | 0.301 | 0.330 | 0.251 | 0.244 | 0.165 | 0.167 |
| 034 | 0.180 | 0.192 | 0.314 | 0.305 | 0.250 | 0.240 | 0.154 | 0.167 |
| 050 | 0.205 | 0.190 | 0.317 | 0.339 | 0.224 | 0.227 | 0.158 | 0.165 |
| 073 | 0.171 | 0.181 | 0.301 | 0.304 | 0.217 | 0.233 | 0.155 | 0.158 |
| 096 | 0.189 | 0.177 | 0.325 | 0.323 | 0.261 | 0.236 | 0.164 | 0.167 |
| 112 | 0.187 | 0.190 | 0.332 | 0.323 | 0.258 | 0.249 | 0.158 | 0.158 |
| 143 | 0.190 | 0.188 | 0.325 | 0.309 | 0.237 | 0.268 | 0.155 | 0.166 |
| 156 | 0.183 | 0.190 | 0.309 | 0.314 | 0.240 | 0.224 | 0.157 | 0.164 |
| 183 | 0.191 | 0.184 | 0.340 | 0.311 | 0.233 | 0.269 | 0.158 | 0.164 |
| 200 | 0.176 | 0.183 | 0.284 | 0.296 | 0.234 | 0.239 | 0.161 | 0.153 |
| mean / AV* | 0.185 (0.198) | | | | 0.242 / 0.264‡ | | 0.161 / 0.169‡ | |

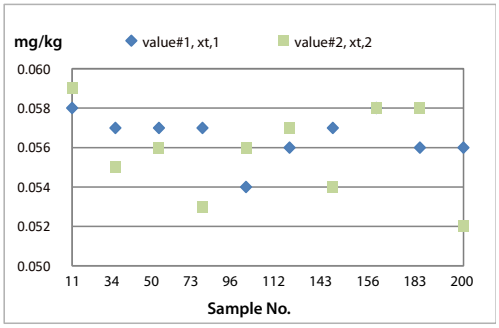
* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories
‡ AV driven from spiking level, fro details please see XXX

Graphical presentation of the results:

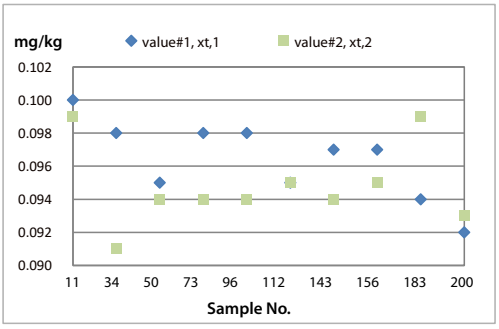
**Oxymatrine**



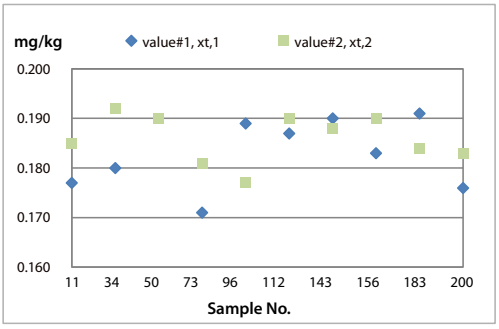

**2,4-DNOP (free phenol)**




**Meptyldinocap**




**Meptyldinocap (sum)**

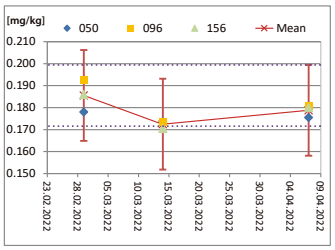



A3 HOMOGENEITY

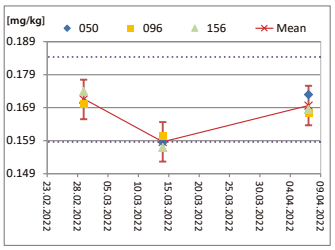
## Appendix 4 Data of Stability Test

| | Compulsory Compounds | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Captan | | | | | | Chlorothalonil | | | | | | Cyromazine | | | | | |
| AV [mg/kg] | 0.172 | | | | | | 0.151 | | | | | | 0.154 | | | | | |
| Date | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | | 08.03.2022 | | 15.03.2022 | | 13.04.2022 | |
| Sample | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | |
| **No. 050** | 0.184 | 0.172 | 0.173 | 0.174 | 0.170 | 0.181 | 0.171 | 0.170 | 0.163 | 0.154 | 0.171 | 0.175 | 0.139 | 0.138 | 0.131 | 0.123 | 0.130 | 0.139 |
| **No. 096** | 0.168 | 0.217 | 0.176 | 0.171 | 0.184 | 0.178 | 0.165 | 0.175 | 0.161 | 0.160 | 0.169 | 0.166 | 0.135 | 0.137 | 0.128 | 0.130 | 0.135 | 0.132 |
| **No. 156** | 0.162 | 0.210 | 0.166 | 0.175 | 0.183 | 0.177 | 0.174 | 0.174 | 0.160 | 0.154 | 0.169 | 0.168 | 0.131 | 0.138 | 0.124 | 0.129 | 0.131 | 0.128 |
| **Mean [mg/kg]** | **0.186** | | **0.173** | | **0.179** | | **0.172** | | **0.159** | | **0.170** | | **0.136** | | **0.128** | | **0.133** | |
| **RSD* [%]** | **3.9%** | | **1.0%** | | **1.6%** | | **1.3%** | | **1.1%** | | **1.7%** | | **1.5%** | | **1.0%** | | **2.0%** | |
| **Deviation [%] (ref. 1st Anaylsis)** | — | | **-7.0%** | | **-3.6%** | | — | | **-7.5%** | | **-1.1%** | | — | | **-6.5%** | | **-2.8%** | |

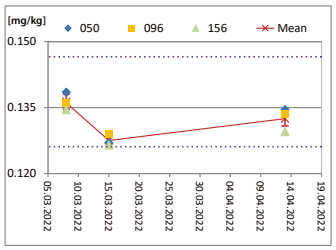
**Captan**




**Chlorothalonil**



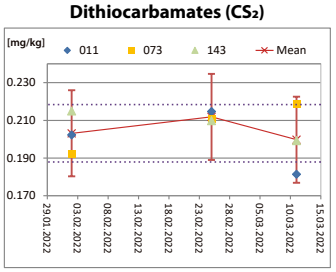

**Cyromazine**



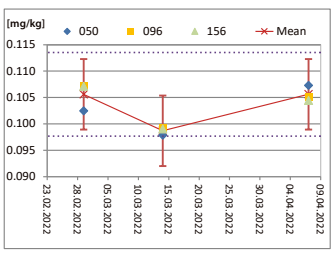

– – –: upper and lower tolerance of the stability test
calculated as mean value of the first stability test ± 0.3× standard Deviation based on FPP-RSD of 25 %

| | Compulsory Compounds | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Dithiocarbamates ($CS_2$) | | | | | | Dodine | | | | | | Emamectin B1a | | | | | |
| AV [mg/kg] | 0.187 | | | | | | 0.100 | | | | | | 0.046 | | | | | |
| Date | 02.02.2022 | | 25.02.2022 | | 11.03.2022 | | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | |
| Sample | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | |
| **No. 050 ‡** | 0.199 | 0.206 | 0.223 | 0.206 | 0.181 | 0.181 | 0.100 | 0.105 | 0.101 | 0.095 | 0.106 | 0.108 | 0.051 | 0.050 | 0.053 | 0.050 | 0.053 | 0.050 |
| **No. 096 ‡** | 0.181 | 0.203 | 0.207 | 0.215 | 0.220 | 0.217 | 0.104 | 0.110 | 0.101 | 0.097 | 0.103 | 0.107 | 0.047 | 0.049 | 0.052 | 0.050 | 0.055 | 0.051 |
| **No. 156 ‡** | 0.207 | 0.223 | 0.204 | 0.216 | 0.195 | 0.204 | 0.107 | 0.108 | 0.101 | 0.097 | 0.104 | 0.105 | 0.051 | 0.051 | 0.052 | 0.053 | 0.052 | 0.049 |
| **Mean [mg/kg]** | **0.203** | | **0.212** | | **0.200** | | **0.106** | | **0.099** | | **0.106** | | **0.050** | | **0.052** | | **0.052** | |
| **RSD* [%]** | **5.6%** | | **1.2%** | | **9.3%** | | **2.6%** | | **0.7%** | | **1.4%** | | **3.1%** | | **1.3%** | | **2.0%** | |
| **Deviation [%] (ref. 1st Anaylsis)** | — | | **4.3%** | | **-1.7%** | | — | | **-6.5%** | | **0.0%** | | — | | **3.6%** | | **3.6%** | |
| | ‡ other portions (11, 73, 143) used | | | | | | | | | | | | | | | | | |

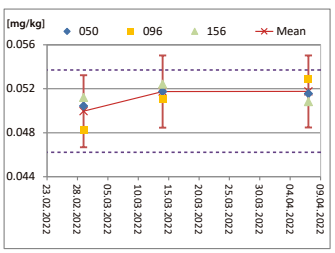
**Dithiocarbamates ($CS_2$)**




**Dodine**




**Emamectin B1a**



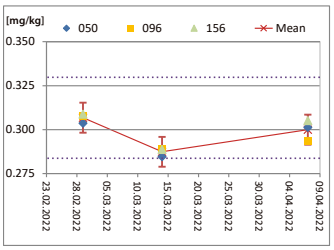

– – –: upper and lower tolerance of the stability test
calculated as mean value of the first stability test ± 0.3× standard Deviation based on FPP-RSD of 25 %

* RSD = relative standard Deviation

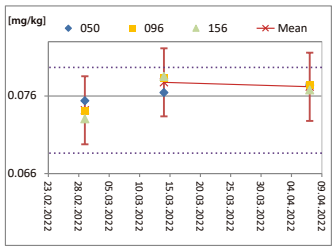
## Appendix 4 (cont.): Data of Stability Test

| | Compulsory Compounds | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Folpet | | | | | | Phthalimide | | | | | | Pymetrozine | | | | | |
| AV [mg/kg] | 0.249 | | | | | | 0.134 | | | | | | 0.15 | | | | | |
| Date | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | | 08.03.2022 | | 15.03.2022 | | 13.04.2022 | |
| Sample | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | |
| No. 050 | 0.301 | 0.307 | 0.289 | 0.280 | 0.302 | 0.301 | 0.073 | 0.076 | 0.075 | 0.077 | 0.078 | 0.076 | 0.186 | 0.180 | 0.175 | 0.172 | 0.177 | 0.181 |
| No. 096 | 0.298 | 0.318 | 0.292 | 0.286 | 0.303 | 0.285 | 0.074 | 0.073 | 0.077 | 0.079 | 0.076 | 0.078 | 0.184 | 0.175 | 0.173 | 0.173 | 0.188 | 0.179 |
| No. 156 | 0.304 | 0.314 | 0.289 | 0.289 | 0.297 | 0.314 | 0.070 | 0.075 | 0.074 | 0.082 | 0.077 | 0.076 | 0.183 | 0.177 | 0.175 | 0.173 | 0.176 | 0.177 |
| Mean [mg/kg] | 0.307 | | 0.287 | | 0.300 | | 0.074 | | 0.077 | | 0.077 | | 0.181 | | 0.174 | | 0.180 | |
| RSD* [%] | 0.9% | | 0.9% | | 2.0% | | 1.6% | | 1.5% | | 0.4% | | 1.1% | | 0.3% | | 1.9% | |
| Deviation [%] (ref. 1st Anaylsis) | — | | -6.3% | | -2.2% | | — | | 4.9% | | 4.1% | | — | | -3.9% | | -0.5% | |

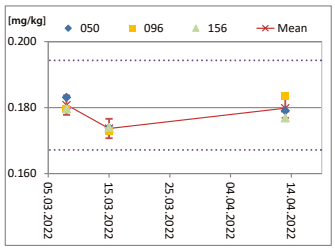
**Folpet**




**Phthalimide**



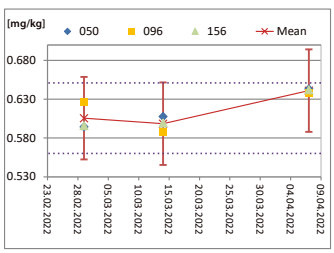

**Pymetrozine**




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

| | Compulsory Compounds | | | | | |
|---|---|---|---|---|---|---|
| | THPI | | | | | |
| AV [mg/kg] | 0.590 | | | | | |
| Date | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | |
| Sample | [mg/kg] | | [mg/kg] | | [mg/kg] | |
| No. 050 | 0.600 | 0.589 | 0.617 | 0.598 | 0.642 | 0.644 |
| No. 096 | 0.654 | 0.599 | 0.596 | 0.580 | 0.622 | 0.654 |
| No. 156 | 0.603 | 0.589 | 0.580 | 0.619 | 0.646 | 0.639 |
| Mean [mg/kg] | 0.606 | | 0.598 | | 0.641 | |
| RSD* [%] | 3.0% | | 1.6% | | 0.5% | |
| Deviation [%] (ref. 1st Anaylsis) | — | | -1.2% | | 5.9% | |

**THPI**

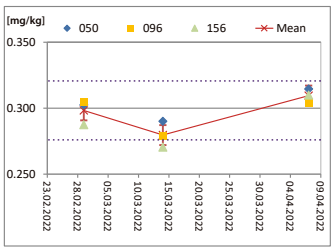



– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard Deviation based on FPP-RSD of 25 %

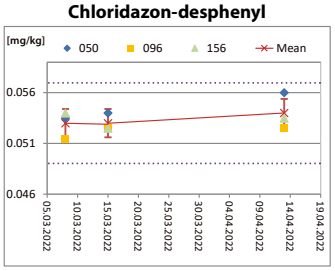
## Appendix 4 (cont.): Data of Stability Test

| | Optional Compounds | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Bifenazate (sum) | | | | | | Chloridazon-desphenyl | | | | | | ETU | | | | | |
| AV [mg/kg] | 0.296 | | | | | | 0.061 | | | | | | 0.063 | | | | | |
| Date | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | | 08.03.2022 | | 15.03.2022 | | 13.04.2022 | | 08.03.2022 | | 15.03.2022 | | 13.04.2022 | |
| Sample | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | |
| **No. 050** | 0.302 | 0.303 | 0.312 | 0.268 | 0.314 | 0.315 | 0.055 | 0.052 | 0.054 | 0.054 | 0.053 | 0.059 | 0.055 | 0.055 | 0.048 | 0.056 | 0.055 | 0.054 |
| **No. 096** | 0.299 | 0.311 | 0.282 | 0.275 | 0.299 | 0.309 | 0.050 | 0.053 | 0.055 | 0.050 | 0.053 | 0.052 | 0.058 | 0.057 | 0.054 | 0.049 | 0.058 | 0.052 |
| **No. 156** | 0.279 | 0.296 | 0.279 | 0.262 | 0.307 | 0.313 | 0.054 | 0.054 | 0.053 | 0.052 | 0.052 | 0.055 | 0.055 | 0.056 | 0.055 | 0.055 | 0.058 | 0.058 |
| **Mean [mg/kg]** | **0.298** | | **0.280** | | **0.310** | | **0.053** | | **0.053** | | **0.054** | | **0.056** | | **0.053** | | **0.056** | |
| **RSD* [%]** | **3.2%** | | **3.5%** | | **1.7%** | | **2.5%** | | **1.6%** | | **3.3%** | | **2.4%** | | **3.6%** | | **3.4%** | |
| **Deviation [%] (ref. 1st Anaylsis)** | **—** | | **-6.3%** | | **3.7%** | | **—** | | **0.0%** | | **1.9%** | | **—** | | **-5.7%** | | **-0.3%** | |

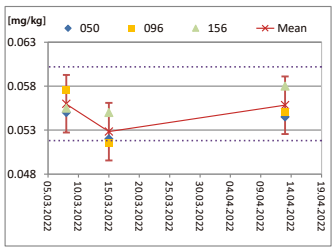
**Bifenazate (sum)**




**Chloridazon-desphenyl**



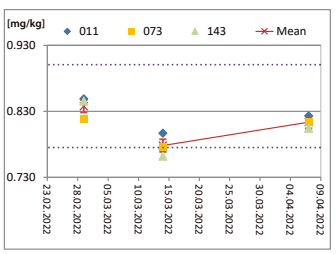

**ETU**



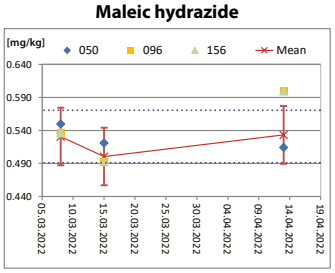

– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard Deviation based on FPP-RSD of 25 %

| | Optional Compounds | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Formetanate-HCl | | | | | | Maleic hydrazide | | | | | | Oxymatrine | | | | | |
| AV [mg/kg] | 0.873 | | | | | | 0.544 | | | | | | 0.198 | | | | | |
| Date | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | | 08.03.2022 | | 15.03.2022 | | 13.04.2022 | | 08.03.2022 | | 15.03.2022 | | 13.04.2022 | |
| Sample | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | |
| **No. 050** | 0.855 | 0.843 | 0.798 | 0.795 | 0.800 | 0.846 | 0.558 | 0.542 | 0.538 | 0.505 | 0.493 | 0.536 | 0.191 | 0.188 | 0.177 | 0.170 | 0.172 | 0.178 |
| **No. 096** | 0.821 | 0.818 | 0.767 | 0.784 | 0.809 | 0.818 | 0.527 | 0.545 | 0.499 | 0.487 | 0.577 | 0.623 | 0.180 | 0.181 | 0.174 | 0.169 | 0.189 | 0.171 |
| **No. 156** | 0.848 | 0.843 | 0.776 | 0.749 | 0.803 | 0.805 | 0.518 | 0.495 | 0.493 | 0.483 | 0.482 | 0.490 | 0.177 | 0.185 | 0.172 | 0.170 | 0.166 | 0.173 |
| **Mean [mg/kg]** | **0.838** | | **0.778** | | **0.814** | | **0.531** | | **0.501** | | **0.533** | | **0.184** | | **0.172** | | **0.175** | |
| **RSD* [%]** | **1.9%** | | **2.3%** | | **1.2%** | | **4.2%** | | **3.6%** | | **11.1%** | | **2.8%** | | **0.8%** | | **3.0%** | |
| **Deviation [%] (ref. 1st Anaylsis)** | **—** | | **-7.1%** | | **-2.9%** | | **—** | | **-5.7%** | | **0.5%** | | **—** | | **-6.4%** | | **-4.8%** | |

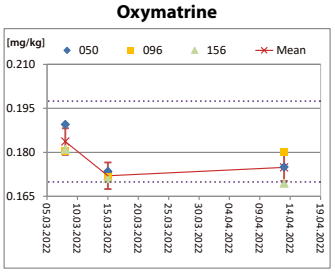
**Formetanate-HCl**




**Maleic hydrazide**




**Oxymatrine**



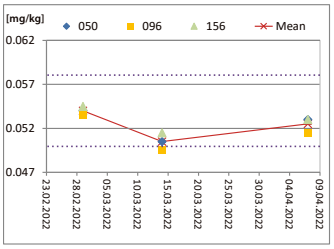

– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard Deviation based on FPP-RSD of 25 %

* RSD = relative standard Deviation

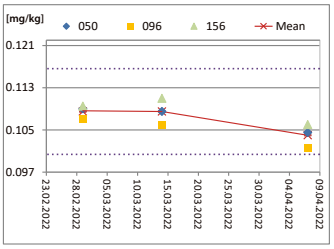
## Appendix 4 (cont.): Data of Stability Test

| | Optional Compounds | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 2,4-DNOP (free phenol) | | | | | | Meptyldinocap | | | | | | Meptyldinocap (sum) | | | | | |
| AV [mg/kg] | 0.056 | | | | | | 0.1 | | | | | | 0.169 | | | | | |
| Date | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | | 01.03.2022 | | 14.03.2022 | | 07.04.2022 | |
| Sample | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | | [mg/kg] | |
| No. 050 | 0.054 | 0.054 | 0.050 | 0.051 | 0.052 | 0.054 | 0.108 | 0.110 | 0.109 | 0.108 | 0.105 | 0.104 | 0.158 | 0.155 | 0.161 | 0.159 | 0.163 | 0.163 |
| No. 096 | 0.053 | 0.054 | 0.050 | 0.049 | 0.051 | 0.052 | 0.106 | 0.108 | 0.106 | 0.106 | 0.102 | 0.101 | 0.158 | 0.161 | 0.162 | 0.157 | 0.166 | 0.159 |
| No. 156 | 0.054 | 0.055 | 0.052 | 0.051 | 0.052 | 0.054 | 0.109 | 0.110 | 0.110 | 0.112 | 0.103 | 0.109 | 0.162 | 0.159 | 0.167 | 0.161 | 0.162 | 0.170 |
| **Mean [mg/kg]** | **0.054** | | **0.051** | | **0.053** | | **0.109** | | **0.109** | | **0.104** | | **0.159** | | **0.161** | | **0.164** | |
| **RSD* [%]** | **0.9%** | | **2.0%** | | **1.6%** | | **1.2%** | | **2.3%** | | **2.2%** | | **1.3%** | | **1.5%** | | **1.2%** | |
| **Deviation [%] (ref. 1st Anaylsis)** | **—** | | **-6.5%** | | **-2.8%** | | **—** | | **0.0%** | | **-4.1%** | | **—** | | **1.5%** | | **3.1%** | |

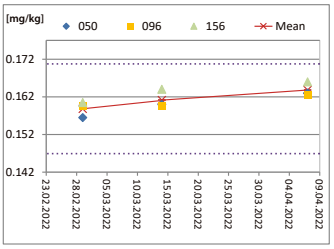
**2,4-DNOP (free phenol)**




**Meptyldinocap**




**Meptyldinocap (sum)**



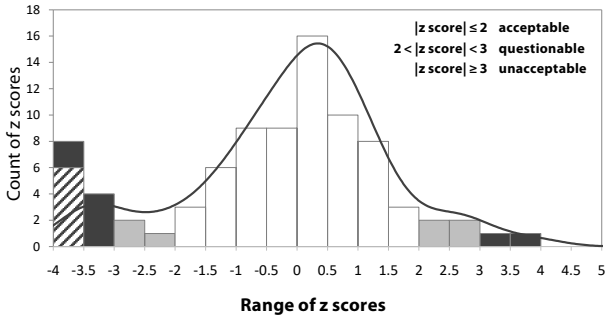

– – –: upper and lower tolerence of the stability test
calculated as mean value of the first stability test ± 0.3× standard Deviation based on FPP-RSD of 25 %

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

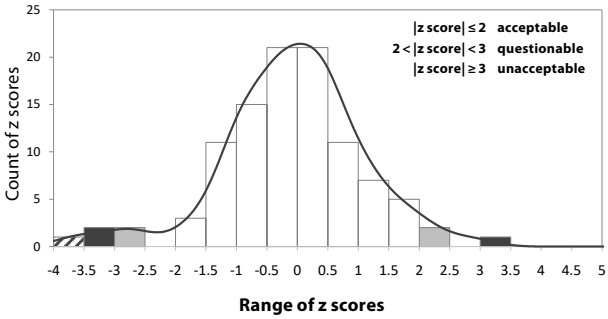
## Appendix 5 Histograms and Kernel Density Estimates of z score* Distributions
(Results from EU and EFTA Laboratories only)

## Compulsory Compounds

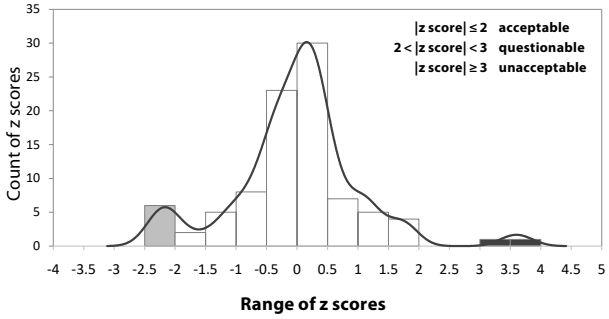
### Captan
(AV derived from entire population excl. 5 outliers)



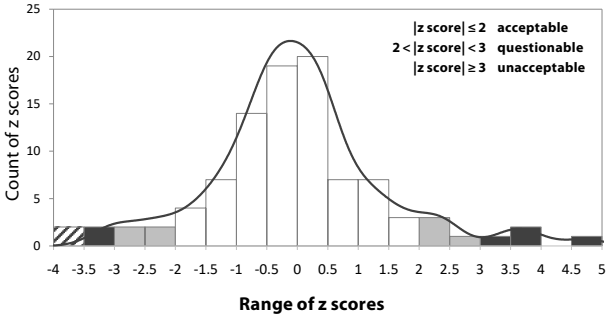

### Chlorothalonil
(AV derived from entire population excl. one outlier)



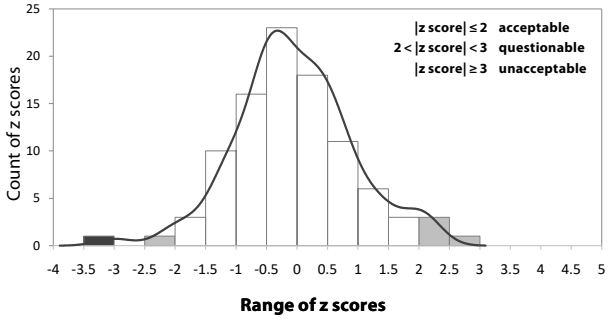

### Cyromazine
(AV derived from entire population)



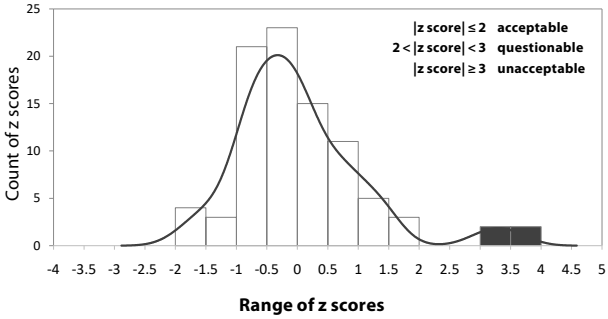

### Dithiocarbamates (expr. as CS2)
(AV derived from entire population excl. two outliers)




### Dodine
(AV derived from entire population)




### Emamectin B1a
(AV derived from entire population excl. two outliers)



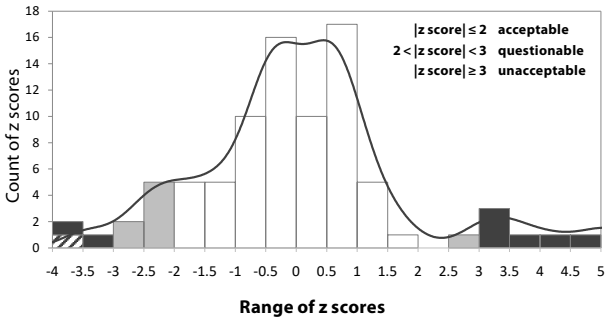

* Cut-off at z score = 5; ▨: false negative results

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

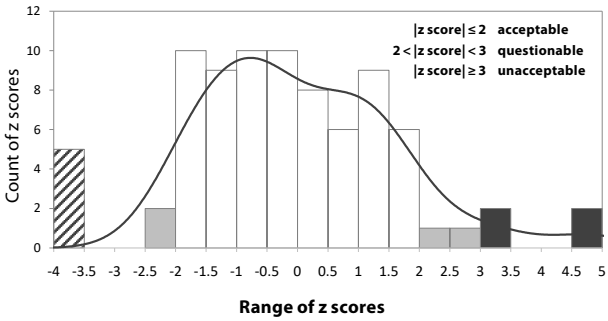
(Results from EU and EFTA Laboratories only)

### Compulsory Compounds (cont.)

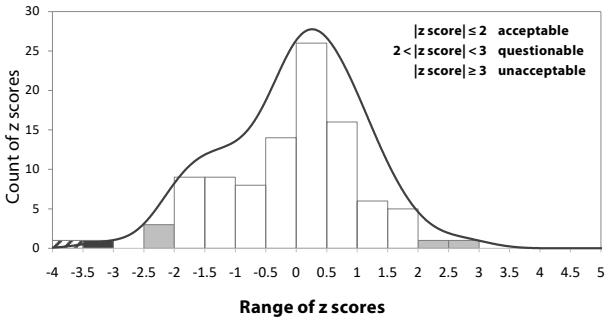
**Folpet**
(AV derived from entire population excl. two outliers)



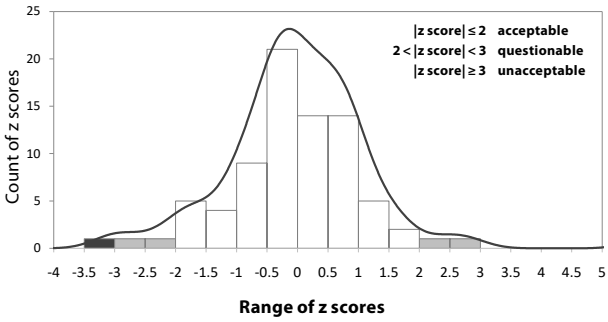

**Phthalimide**
(AV derived from entire population excl. two outliers)




**Pymetrozine**
(AV derived from entire population excl. one outlier)




**THPI**
(AV derived from entire population excl. one outlier)






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

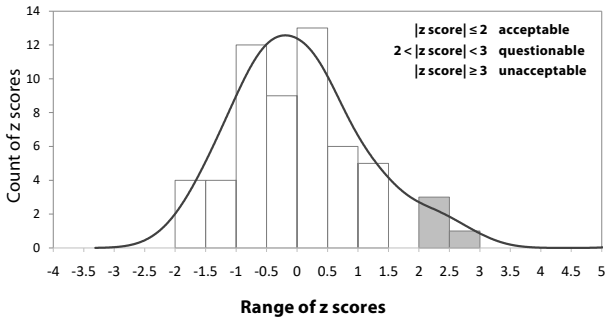
## Appendix 5 (cont.) Histograms and Kernel Density Estimates of z score* Distributions

(Results from EU and EFTA Laboratories only)

## Optional Compounds

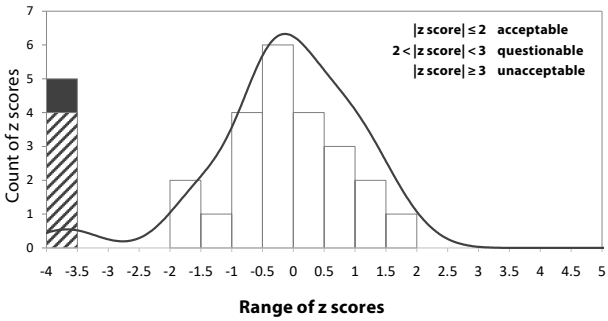
### Bifenazate (sum)

(AV derived from entire population excl.two outliers)




### Chloridazon-desphenyl

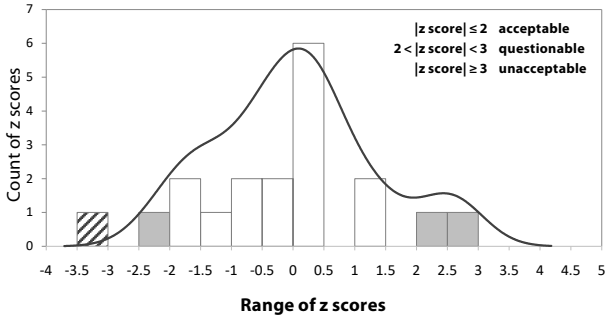
(AV derived from entire population excl.one outlier)




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

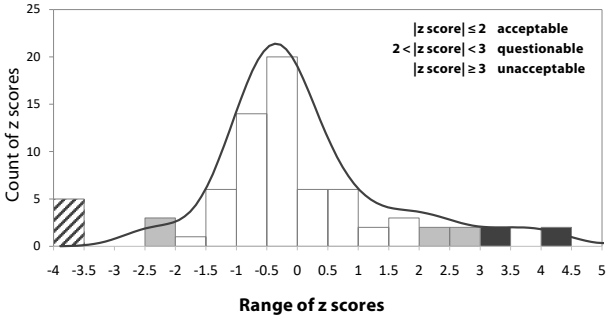
### ETU

(AV derived from entire population)



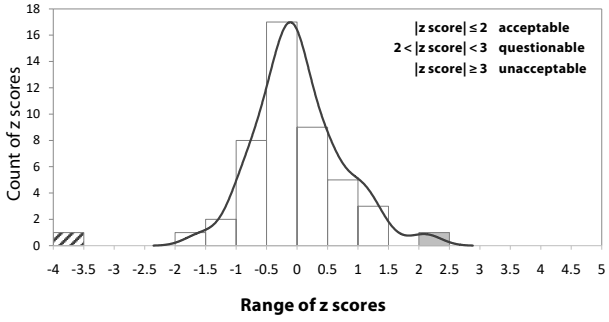

### Formetanate-HCl

(AV derived from entire population excl. five outliers)



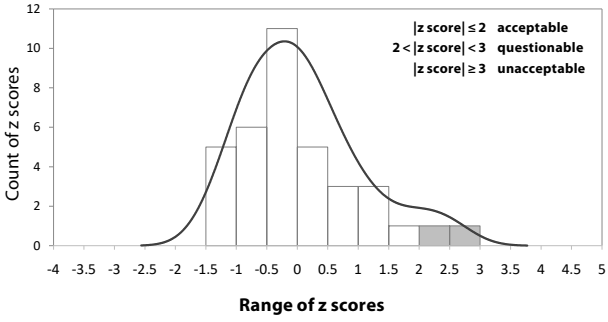

### Maleic hydrazide

(AV derived from entire population excl. one outlier)




### Oxymatrine

(AV derived from entire population)

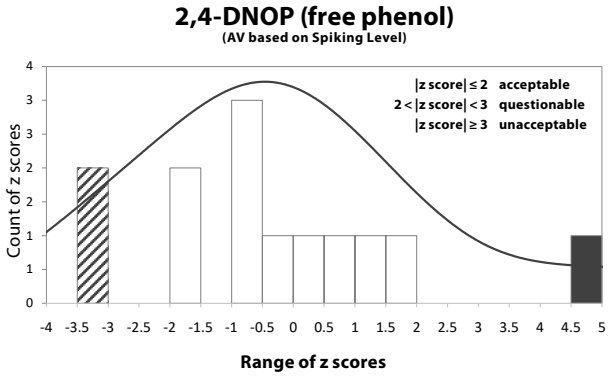


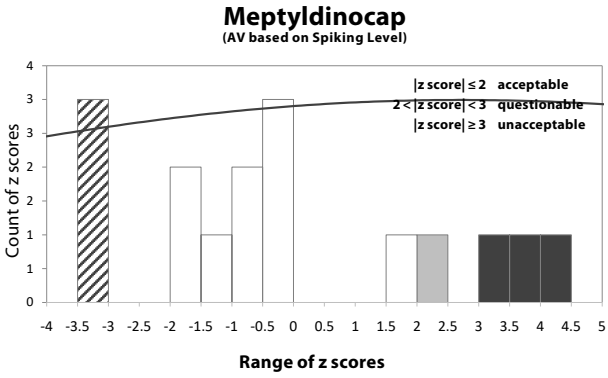

***** Cut-off at z score = 5; ▨: false negative results

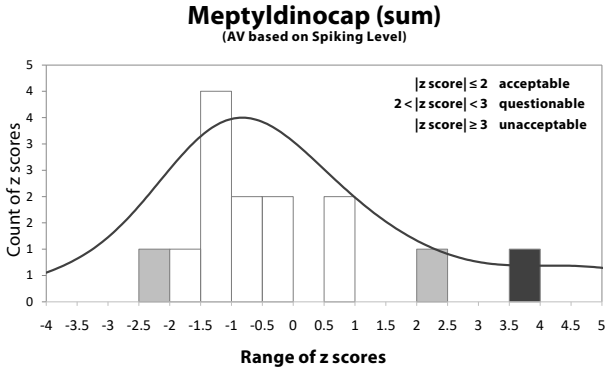
## Appendix 5 (cont.) Histograms and Kernel Density Estimates of z score* Distributions

(Results from EU and EFTA Laboratories only)

As described in Chapter 4, the results distribution of meptyldinocap and meptyldinocap (sum) was very broad and the number of the numerical results for 2,4-DNOP (free phenyl) was not sufficient for a reliable statistical evaluation, therefore, the theoretical spiking levels of these three compounds were designated as the preliminary assigned values and used to calculate the preliminary z scores. The z scores used in the following graphs were only calculated for being able to visualize the distribution of the results.

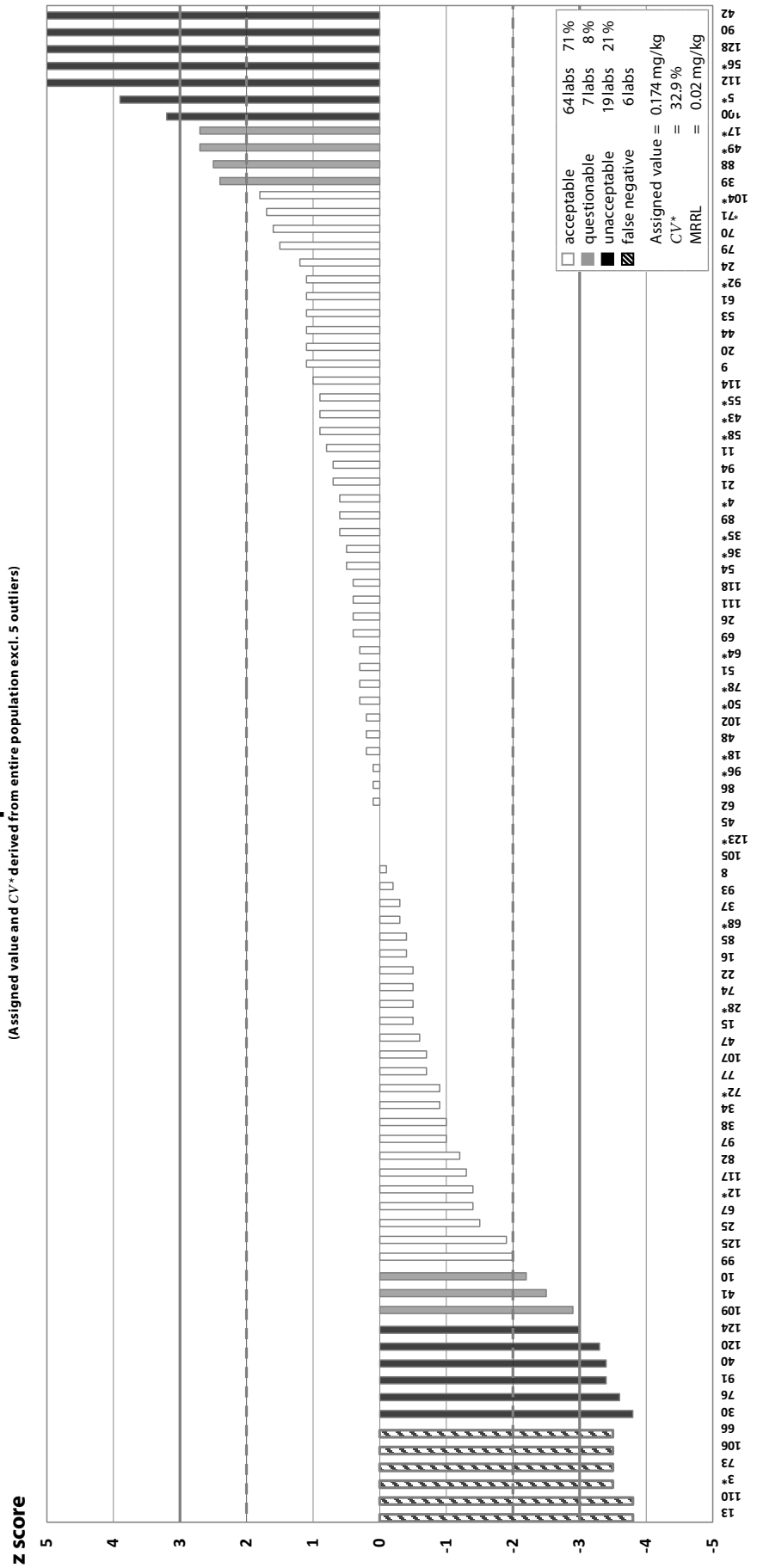



* Cut-off at z score = 5; ▨: false negative results

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



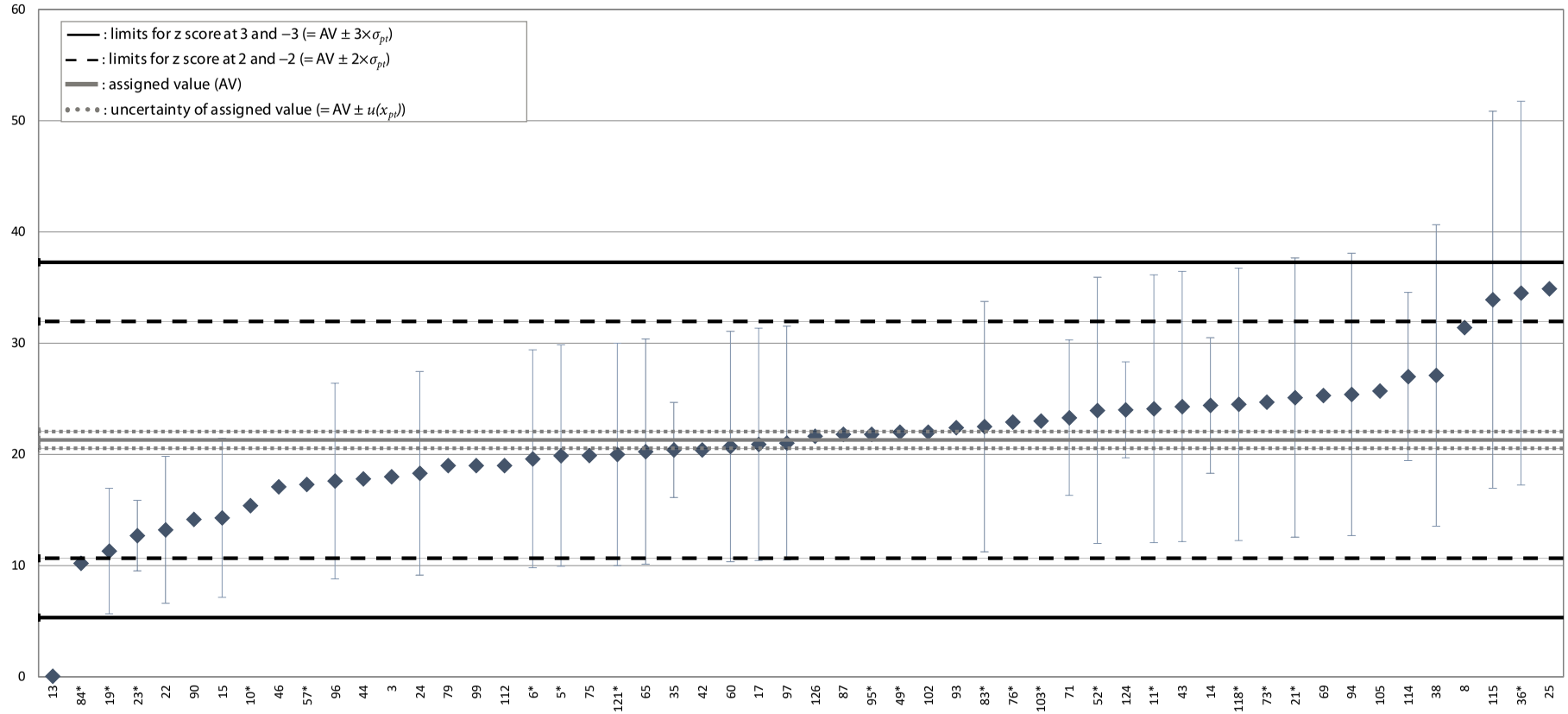
## Appendix 6 Graphic Presentation of Results: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)

# Captan

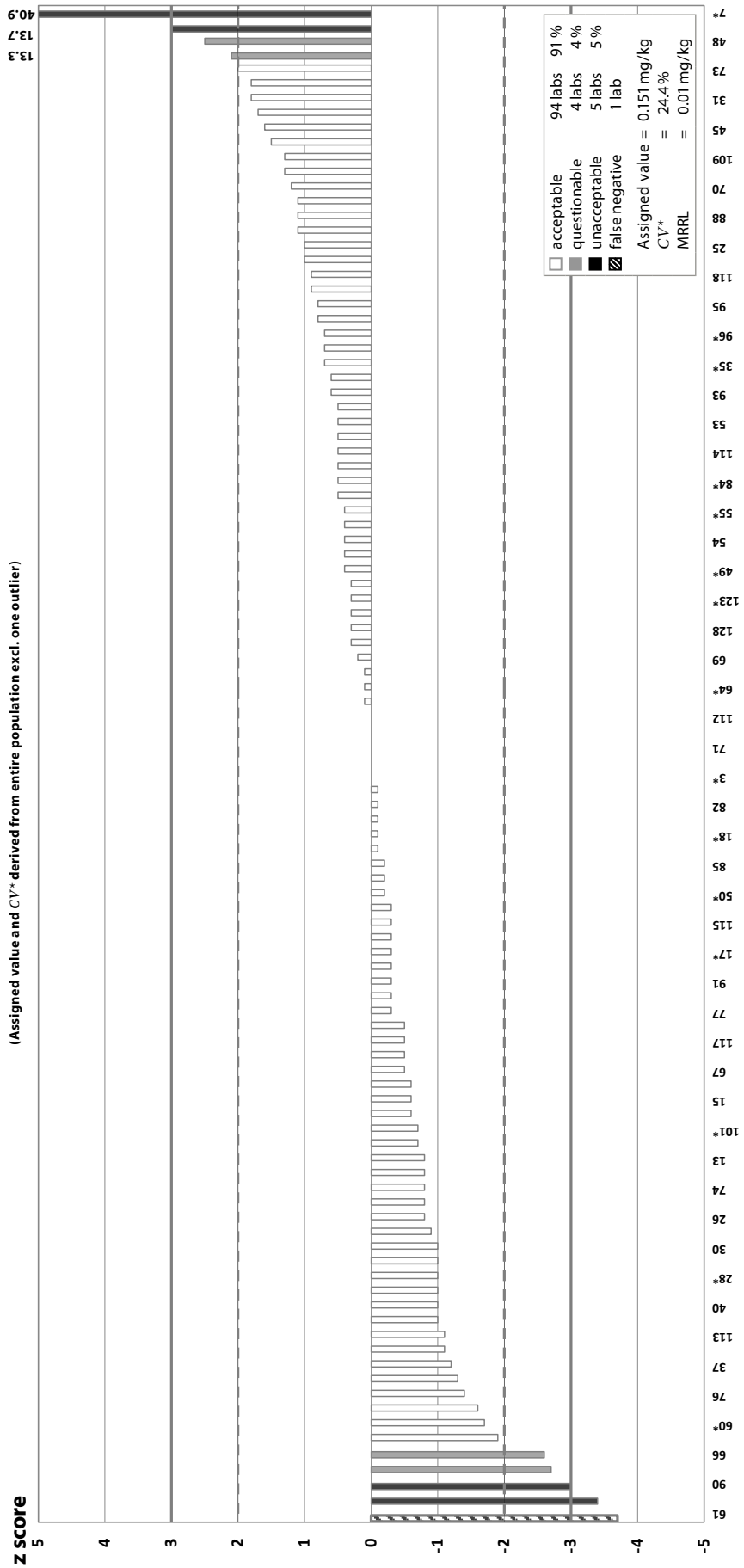
**(Assigned value and $CV^*$ derived from entire population excl. 5 outliers)**

**Conc. [mg/kg]**

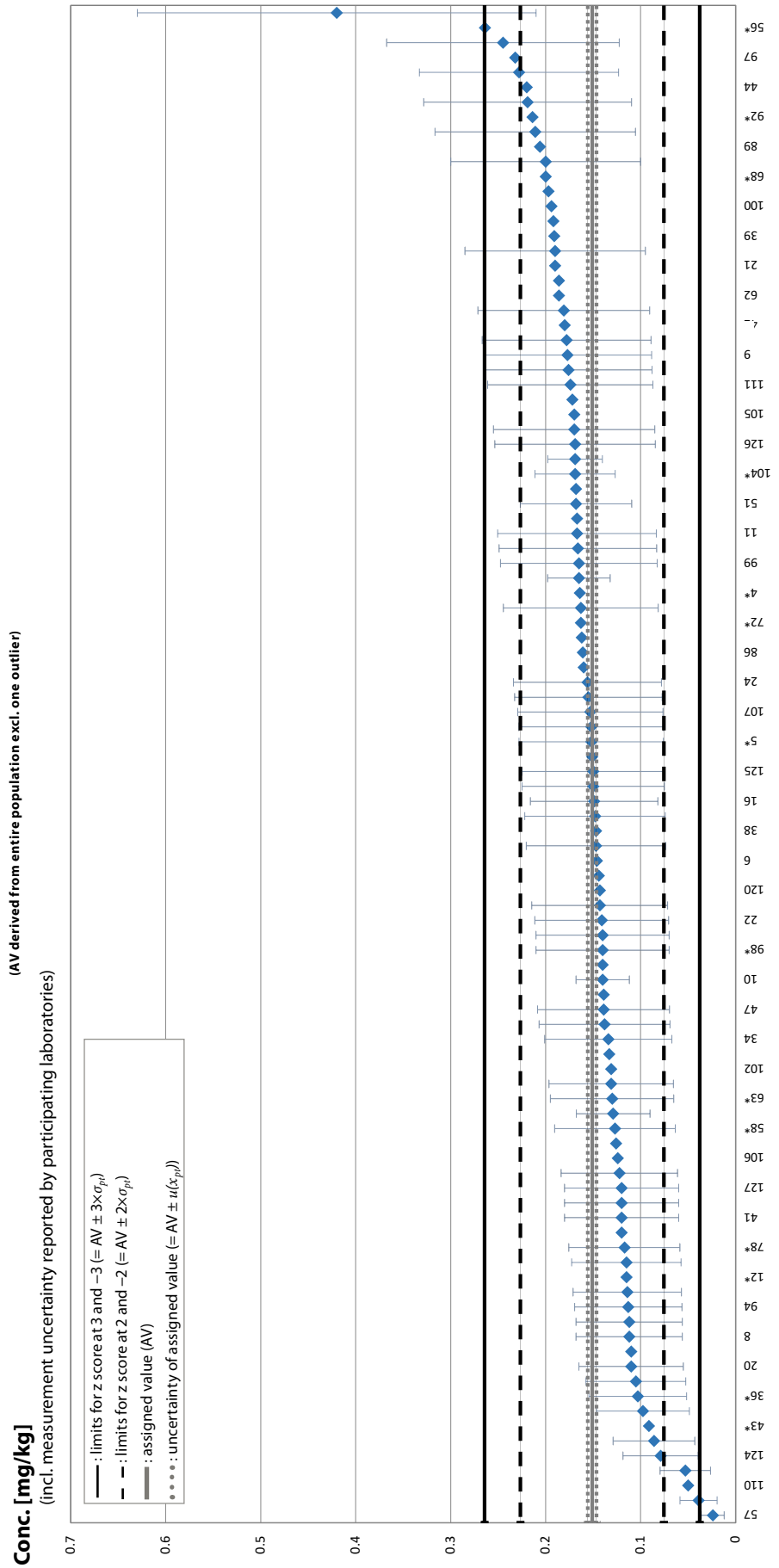
(incl. measurement uncertainty reported by participating laboratories)



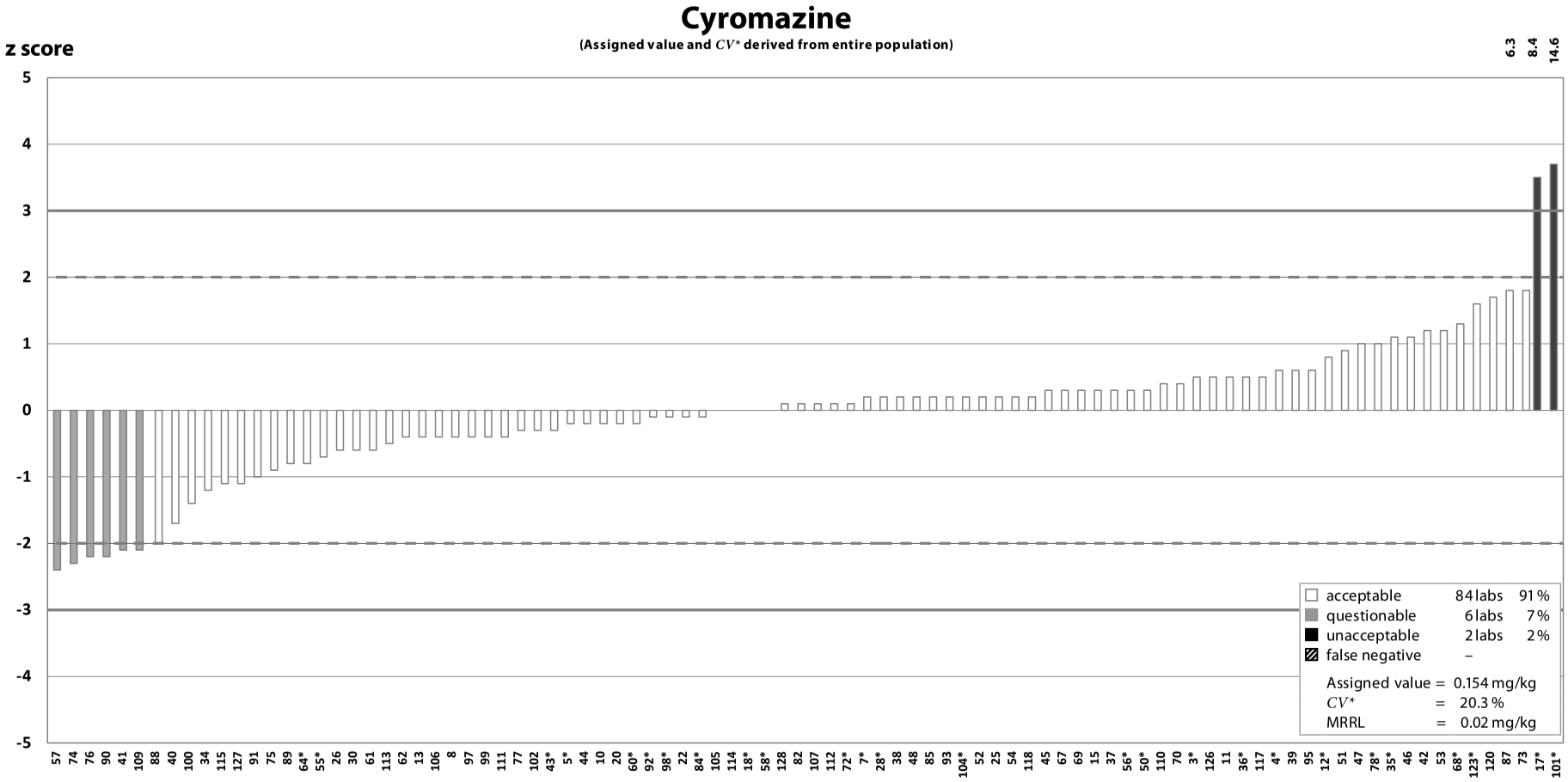
**Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



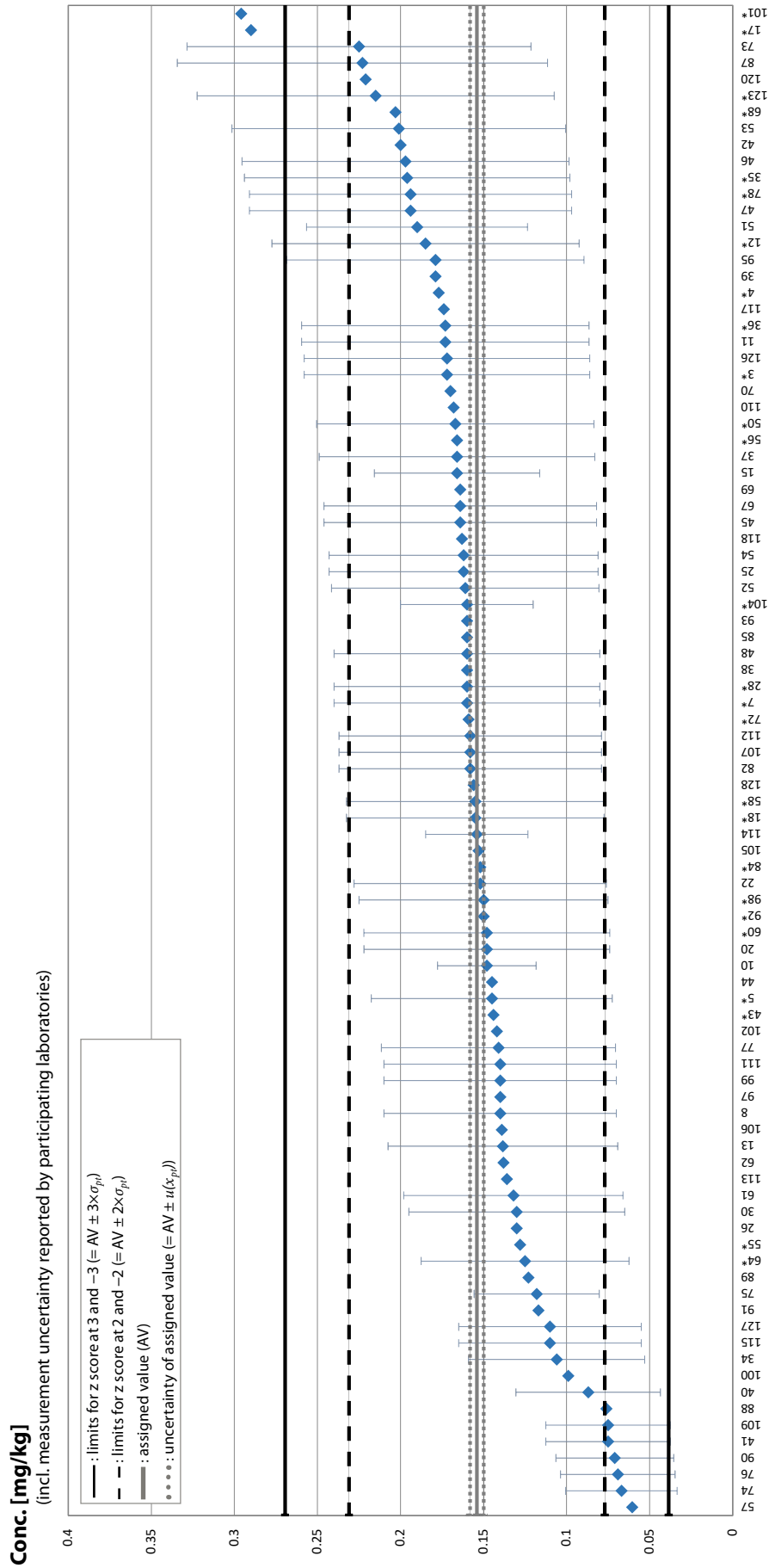
**Appendix 6 Graphic Presentation of Results: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



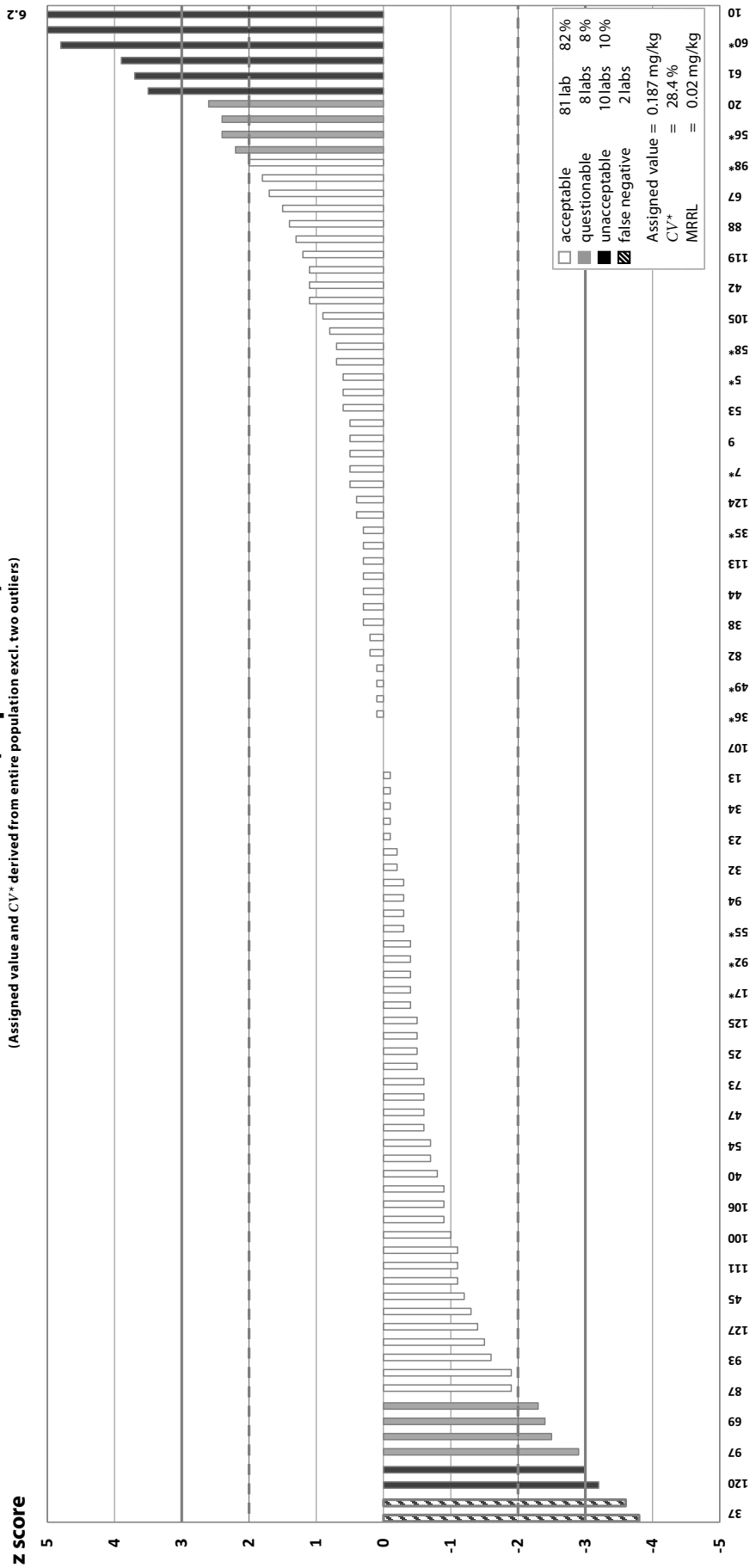
**Appendix 6 (cont.)** **Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



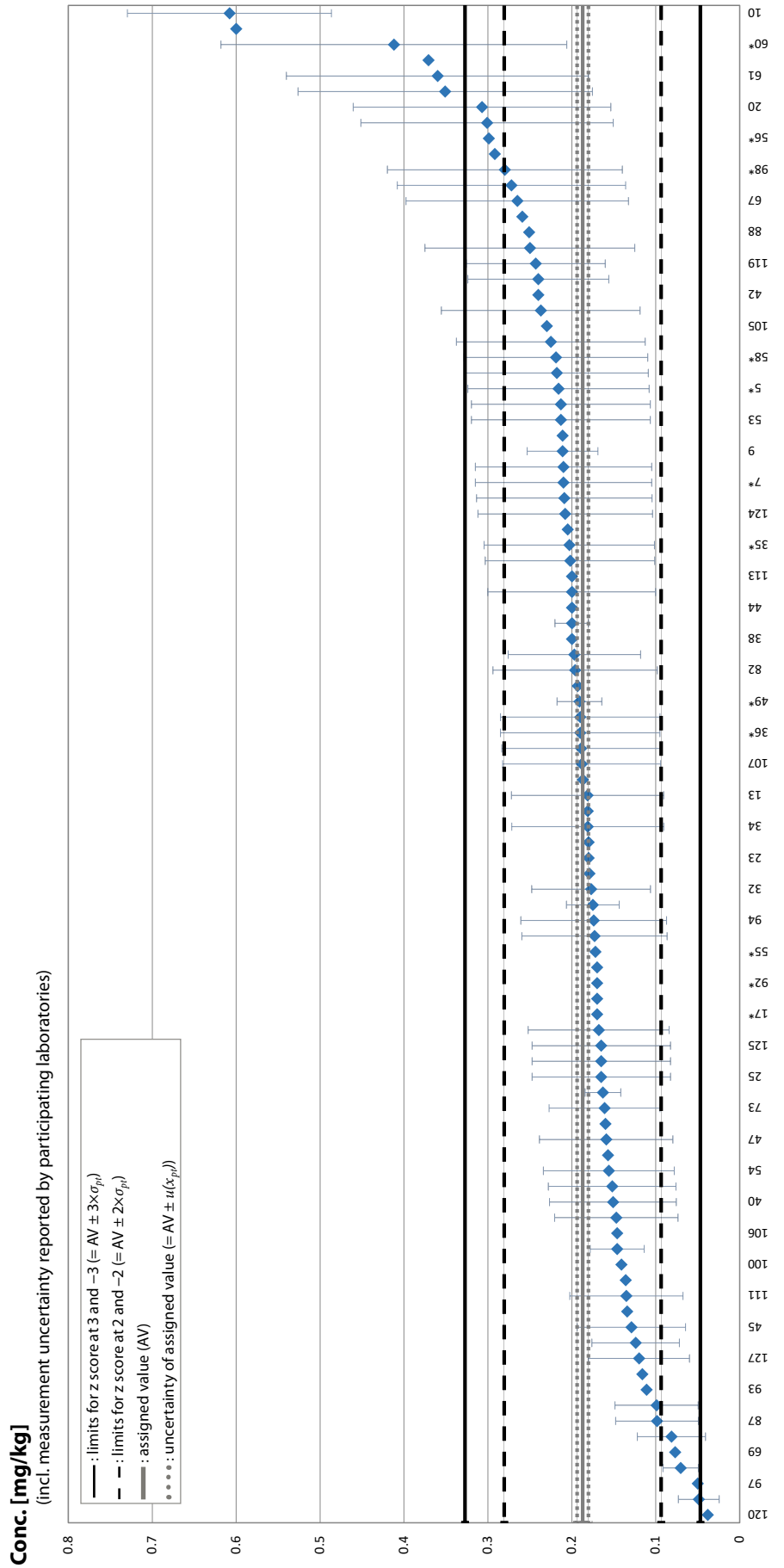
**Appendix 6 Graphic Presentation of Results: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



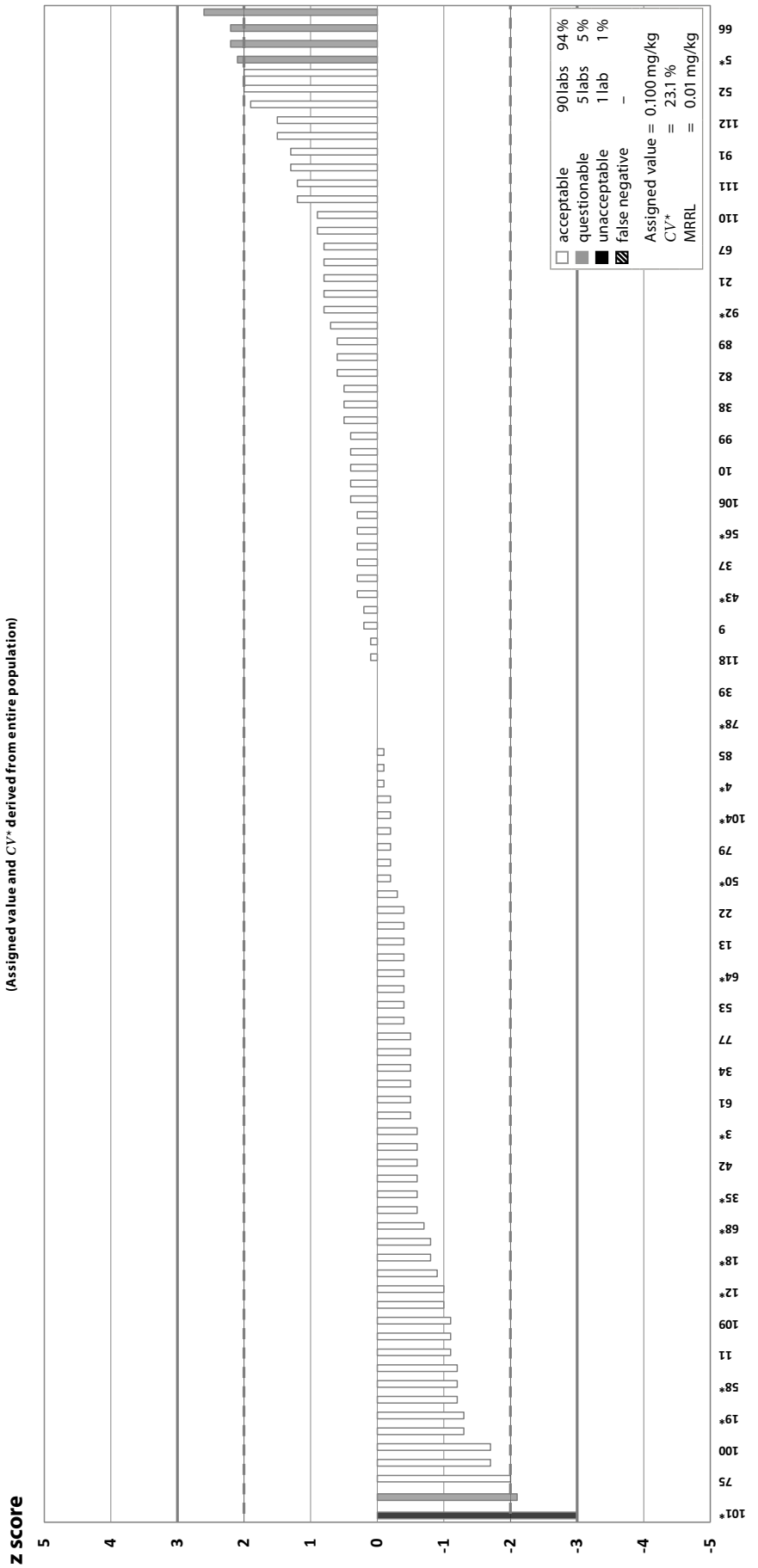
**Appendix 6 (cont.)** **Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



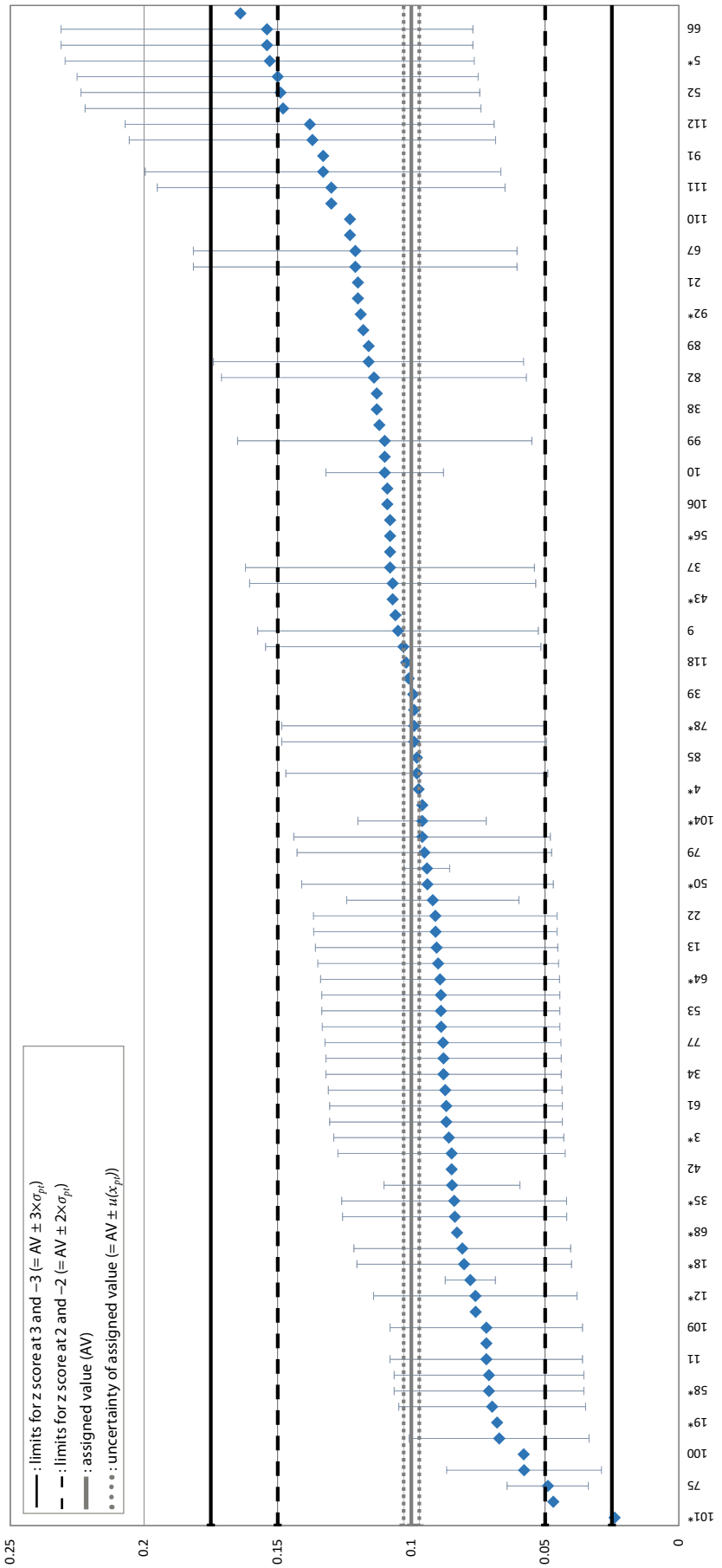
**Appendix 6 Graphic Presentation of Results: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



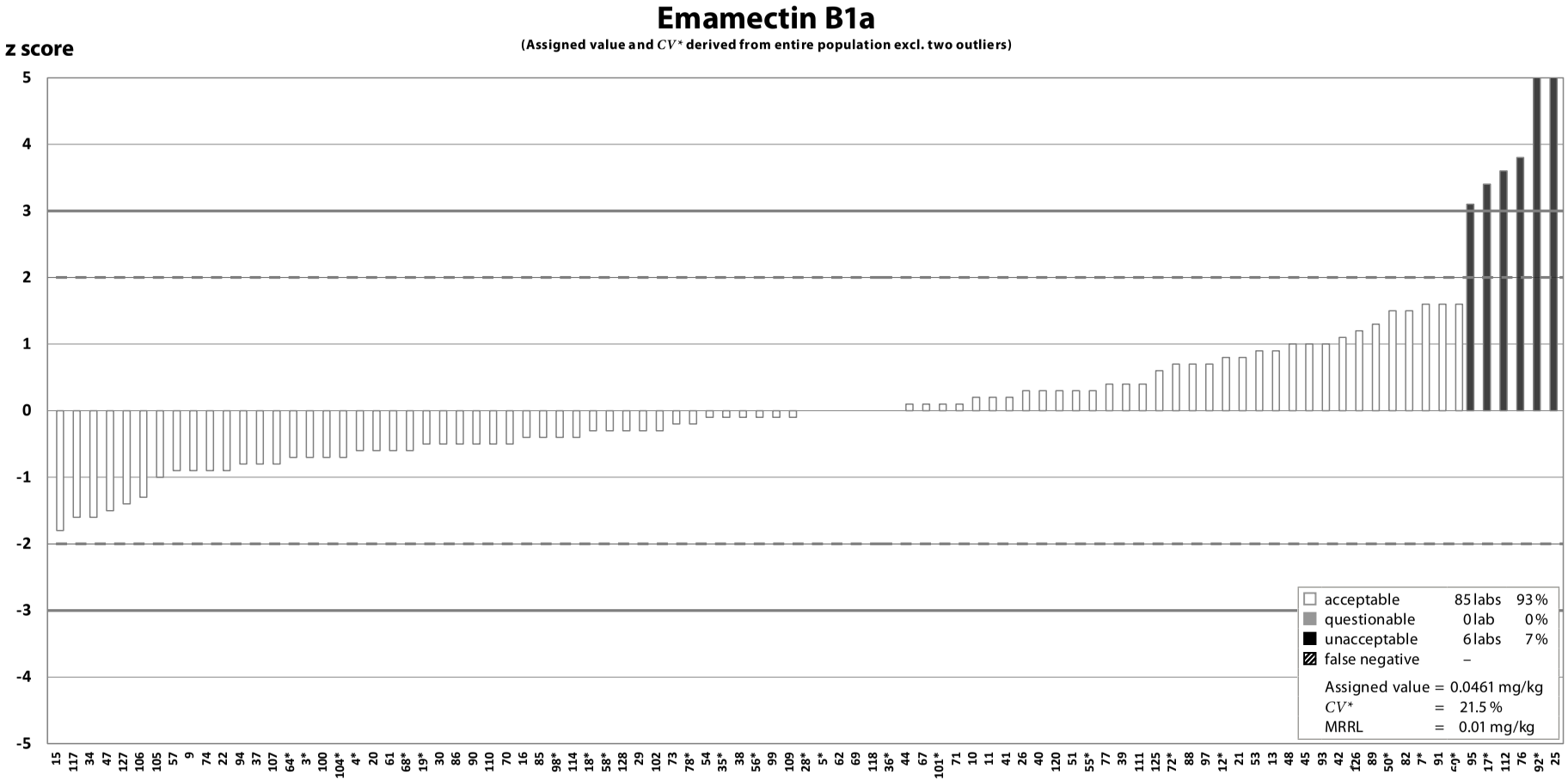
**Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



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



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



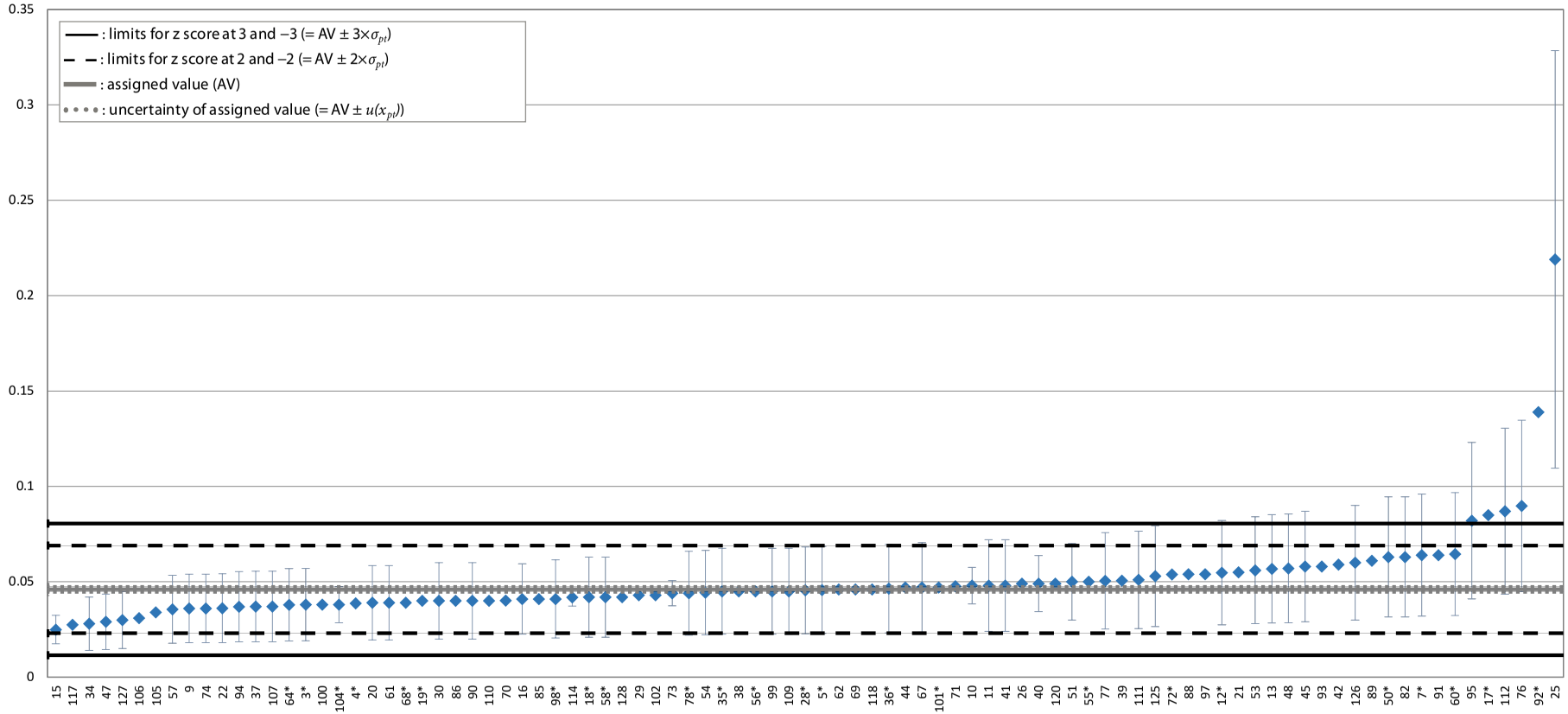
## Appendix 6 Graphic Presentation of Results: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)

### Emamectin B1a

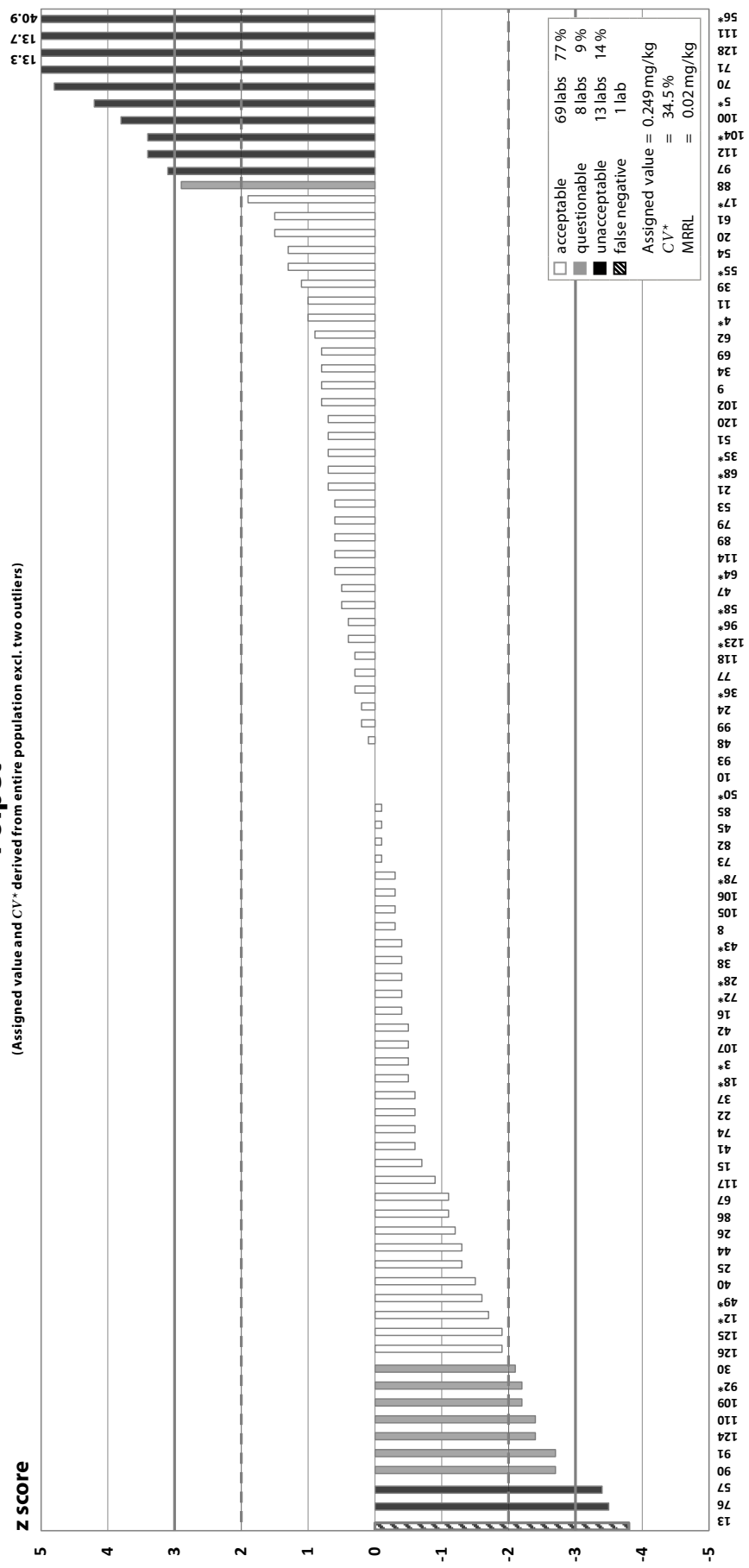
**(Assigned value and $CV^*$ derived from entire population excl. two outliers)**

**Conc. [mg/kg]**

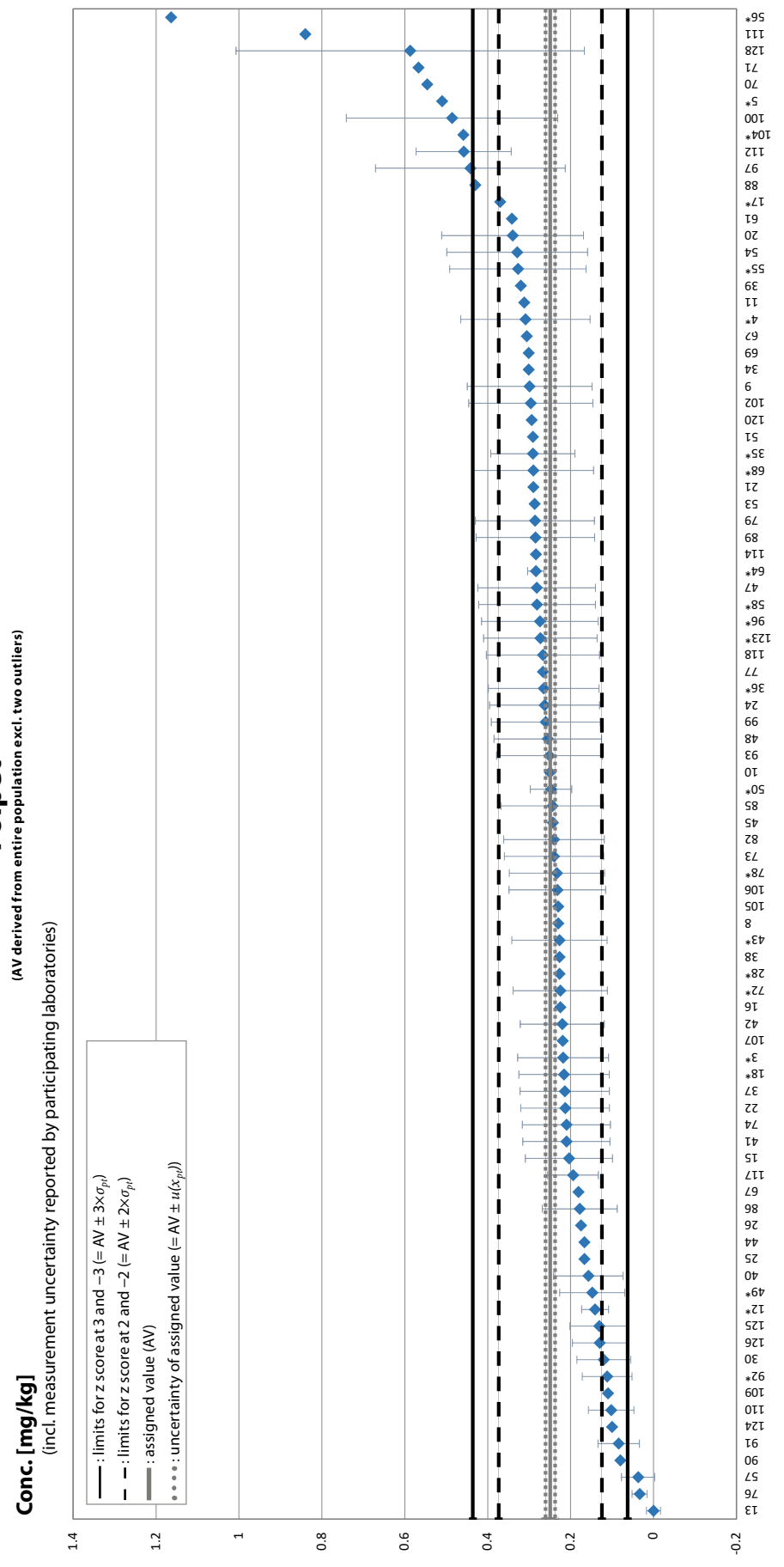
(incl. measurement uncertainty reported by participating laboratories)

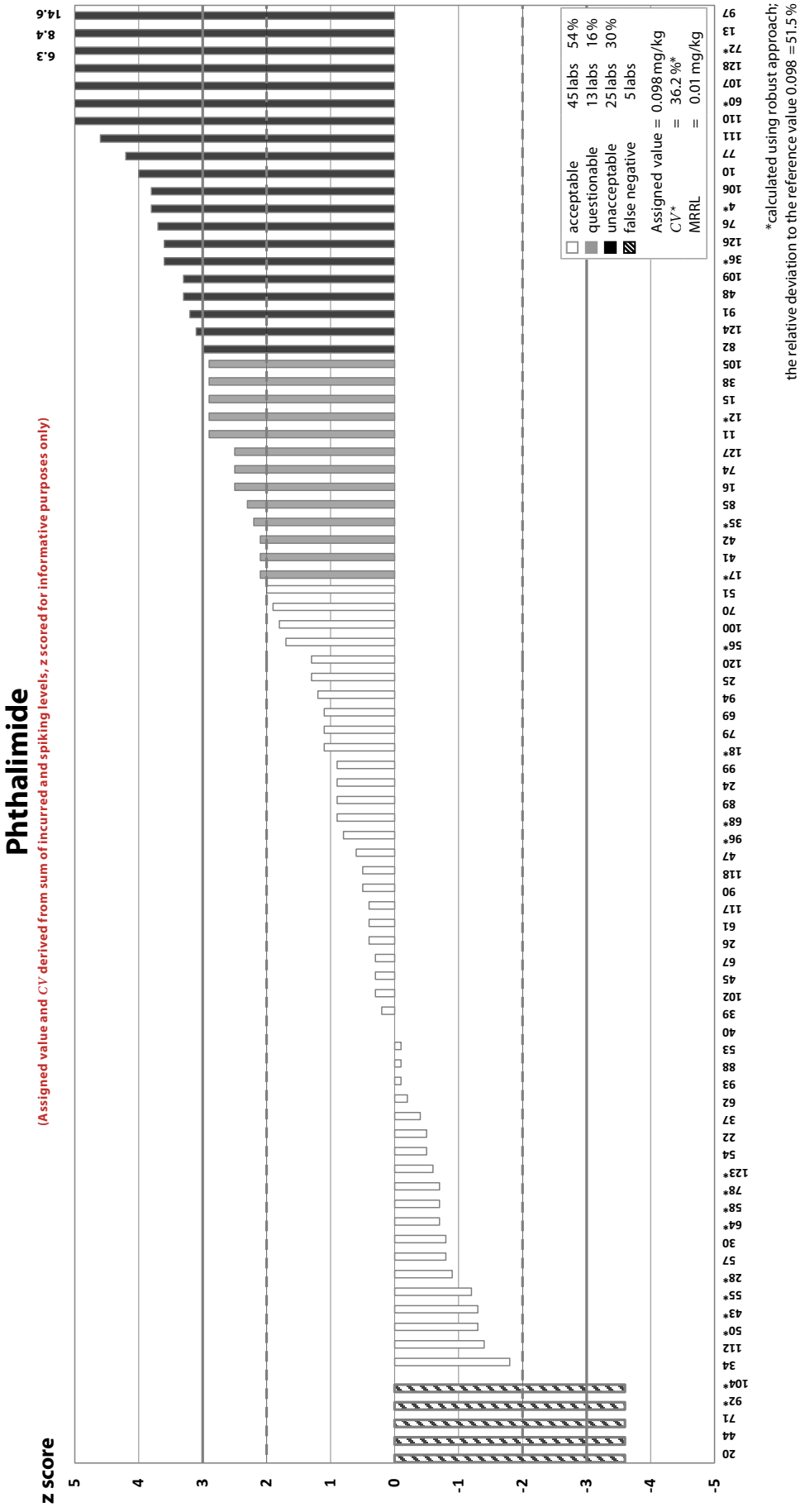


**Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



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




Phthalimide
(Assigned value and CV derived from sum of incurred and spiking levels, z scored for informative purposes only)
z score
5
4
3
2
1
0
-1
-2
-3
-4
-5
acceptable 45 labs 54 %
questionable 13 labs 16 %
unacceptable 25 labs 30 %
false negative 5 labs
Assigned value = 0.098 mg/kg
CV* = 36.2 %*
MRRL = 0.01 mg/kg
*calculated using robust approach;
the relative deviation to the reference value 0.098 = 51.5 %

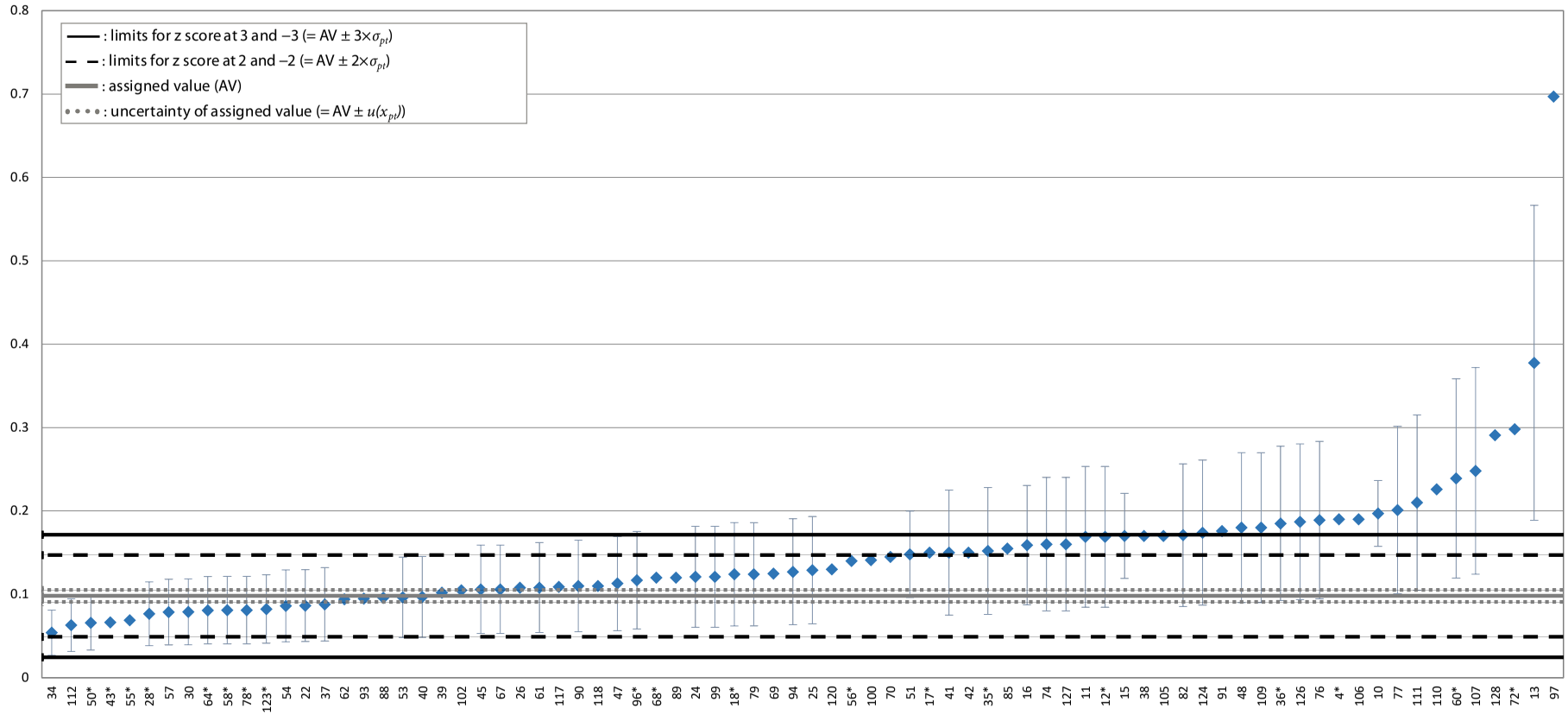
## Appendix 6 Graphic Presentation of Results: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)

# Phthalimide

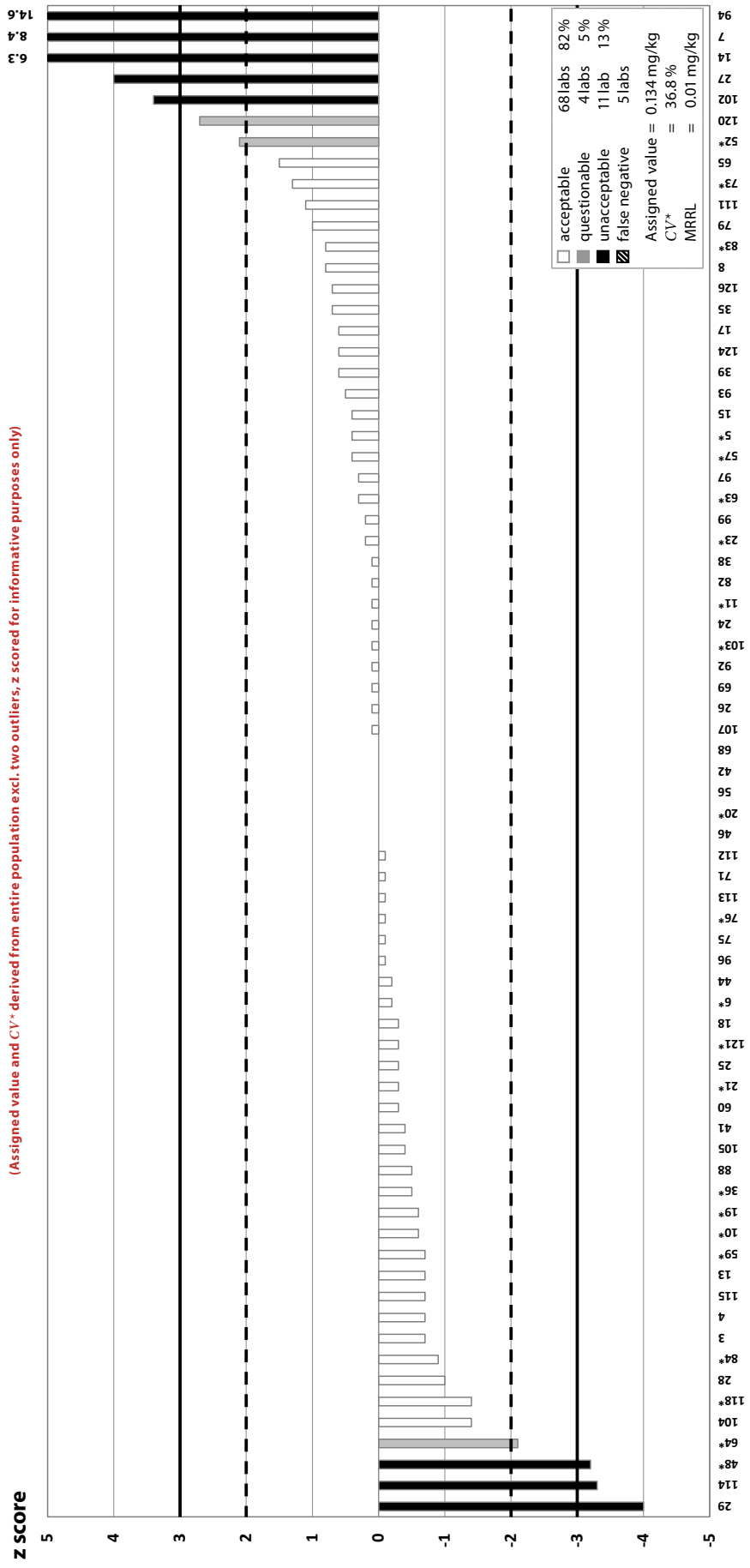
(Assigned value and $CV$ derived from sum of incurred and spiking levels, z scored for informative purposes only)

**Conc. [mg/kg]**

(incl. measurement uncertainty reported by participating laboratories)



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



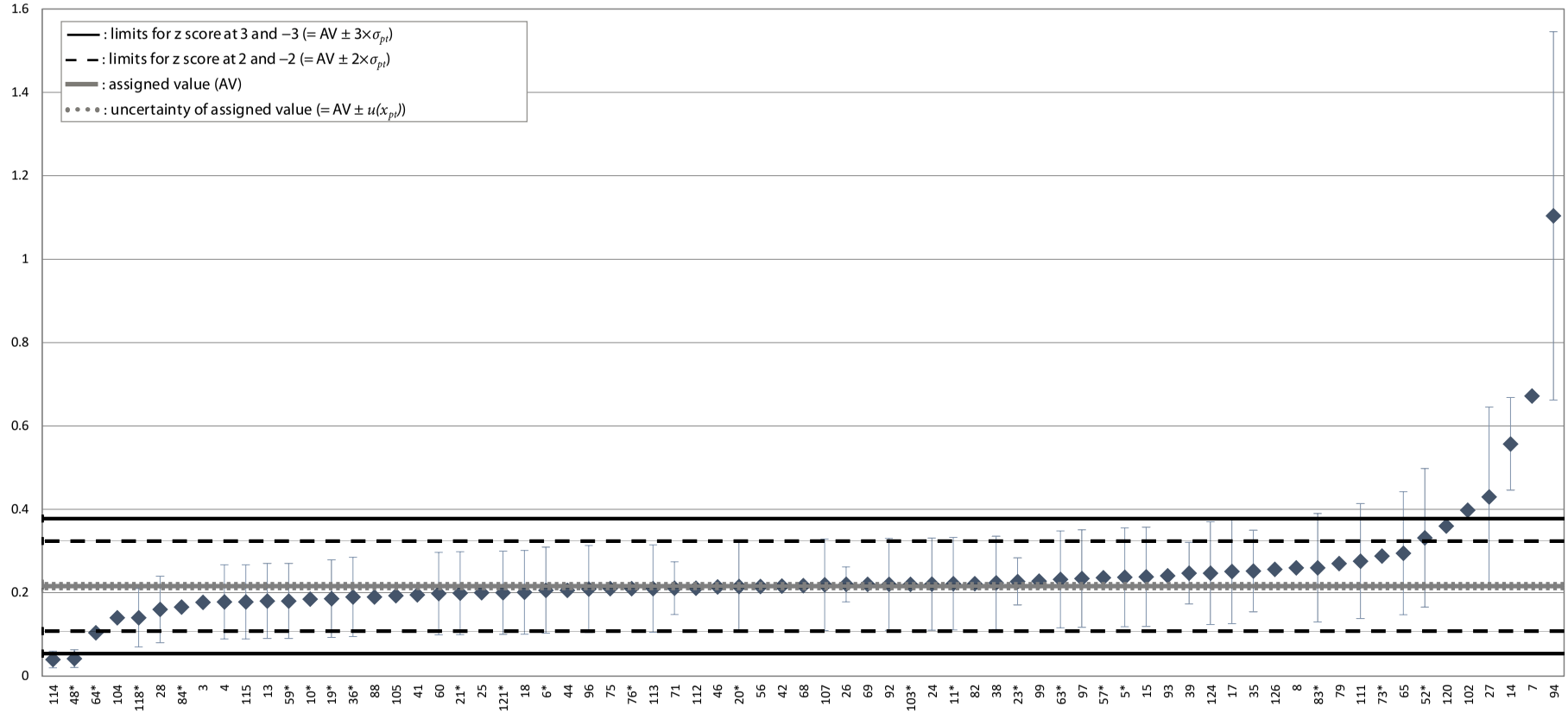
## Appendix 6 Graphic Presentation of Results: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)

# Phthalimide

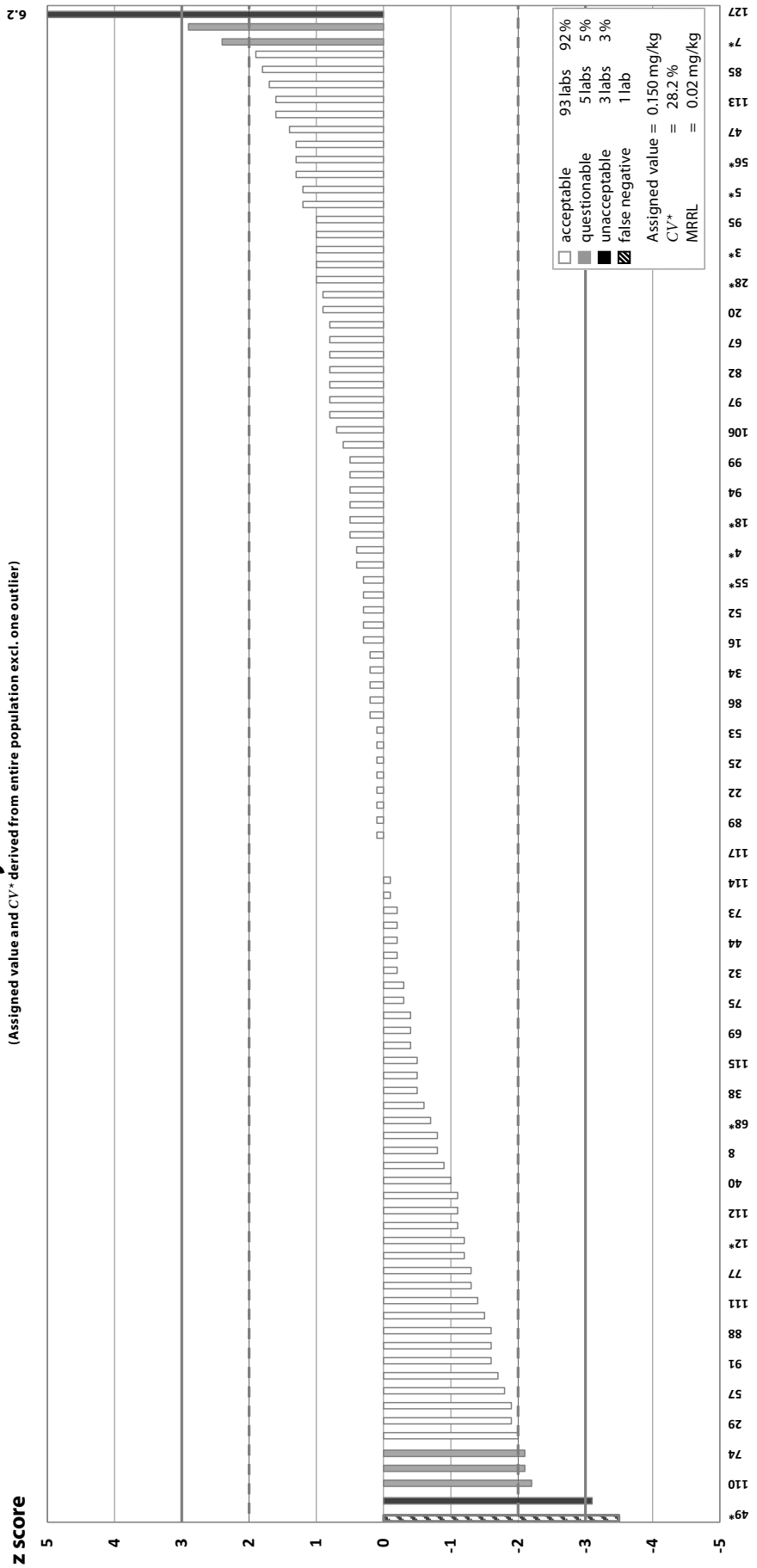
**(Assigned value and $CV^*$ derived from entire population excl. two outliers, z scored for informative purposes only)**

**Conc. [mg/kg]**

(incl. measurement uncertainty reported by participating laboratories)



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



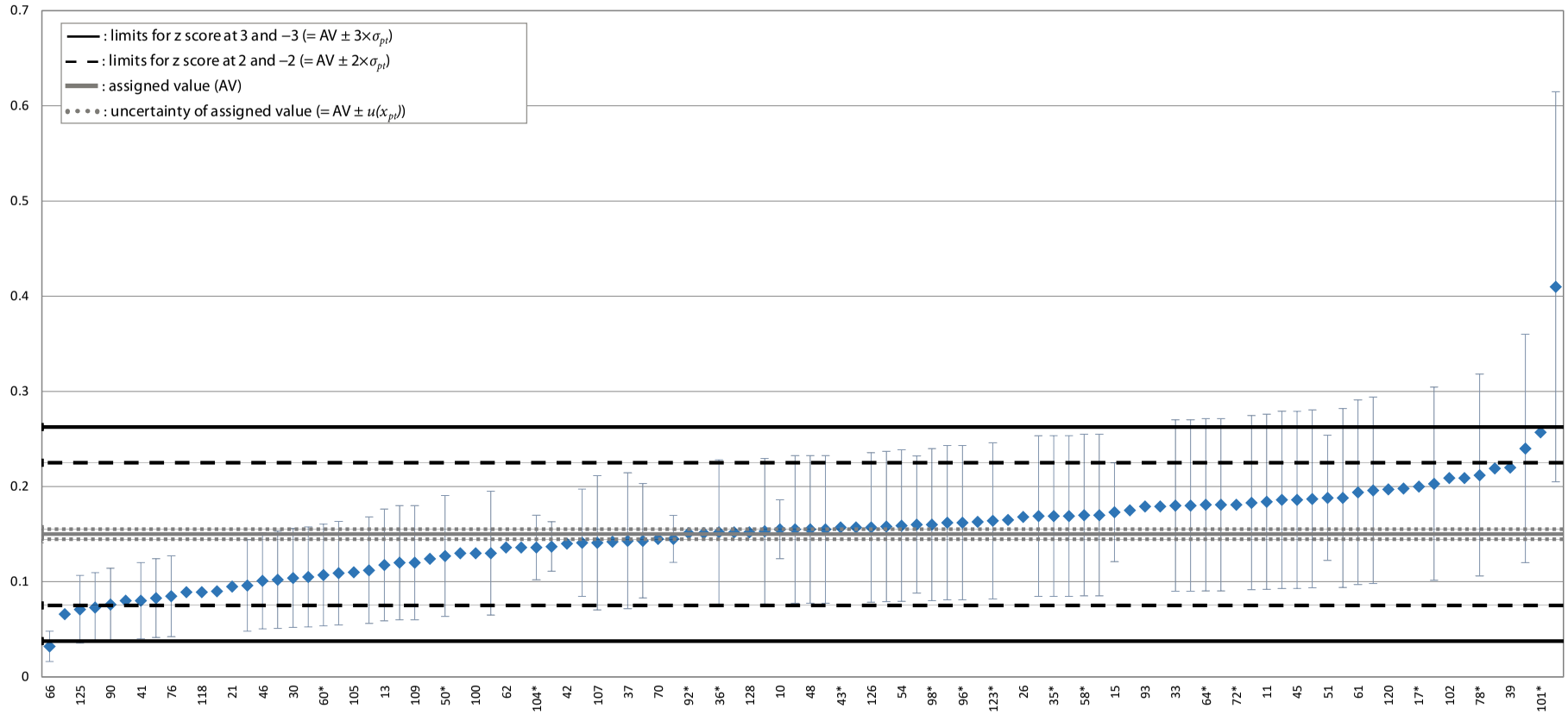
## Appendix 6 Graphic Presentation of Results: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)

# Pymetrozine

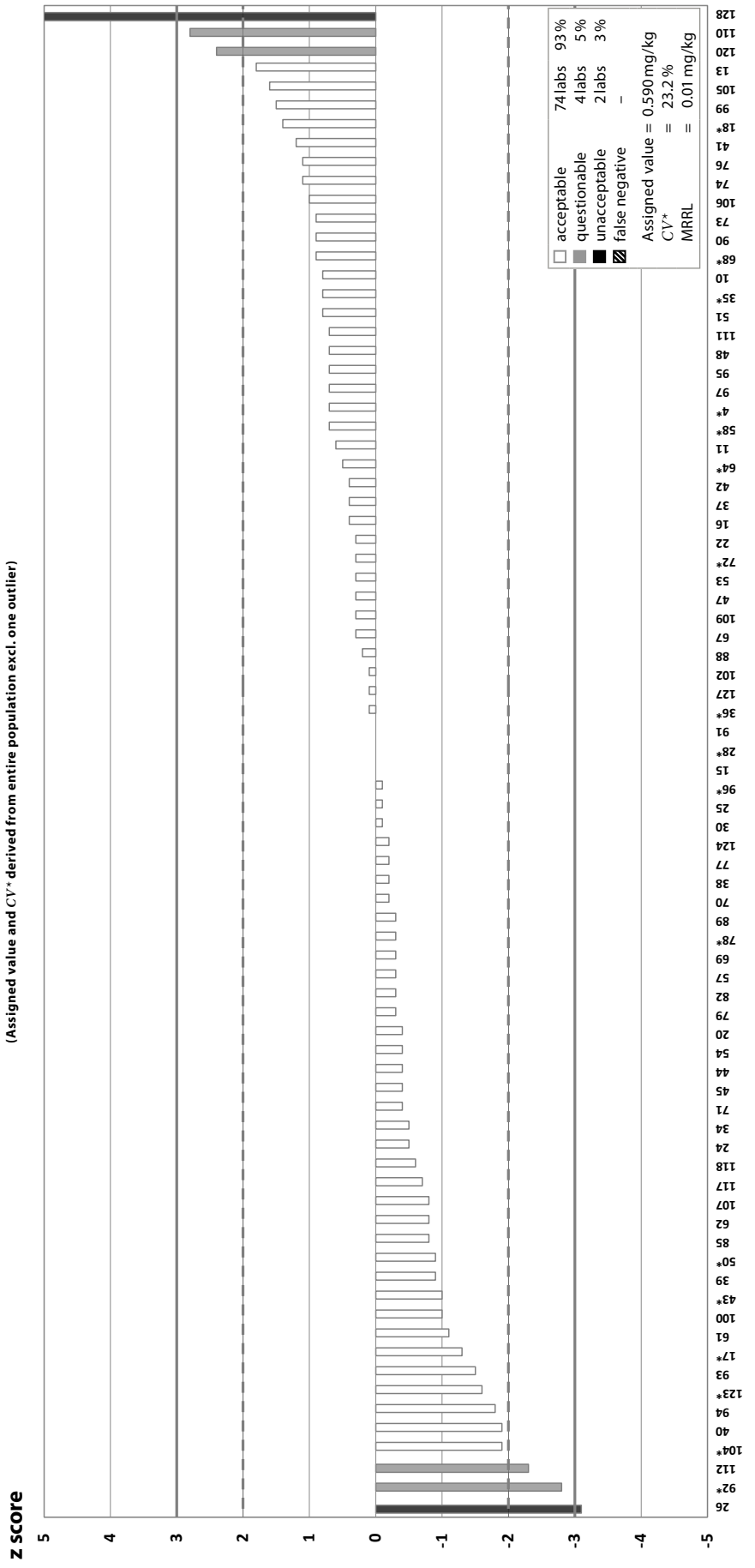
**(Assigned value and $CV^*$ derived from entire population excl. one outlier)**

**Conc. [mg/kg]**

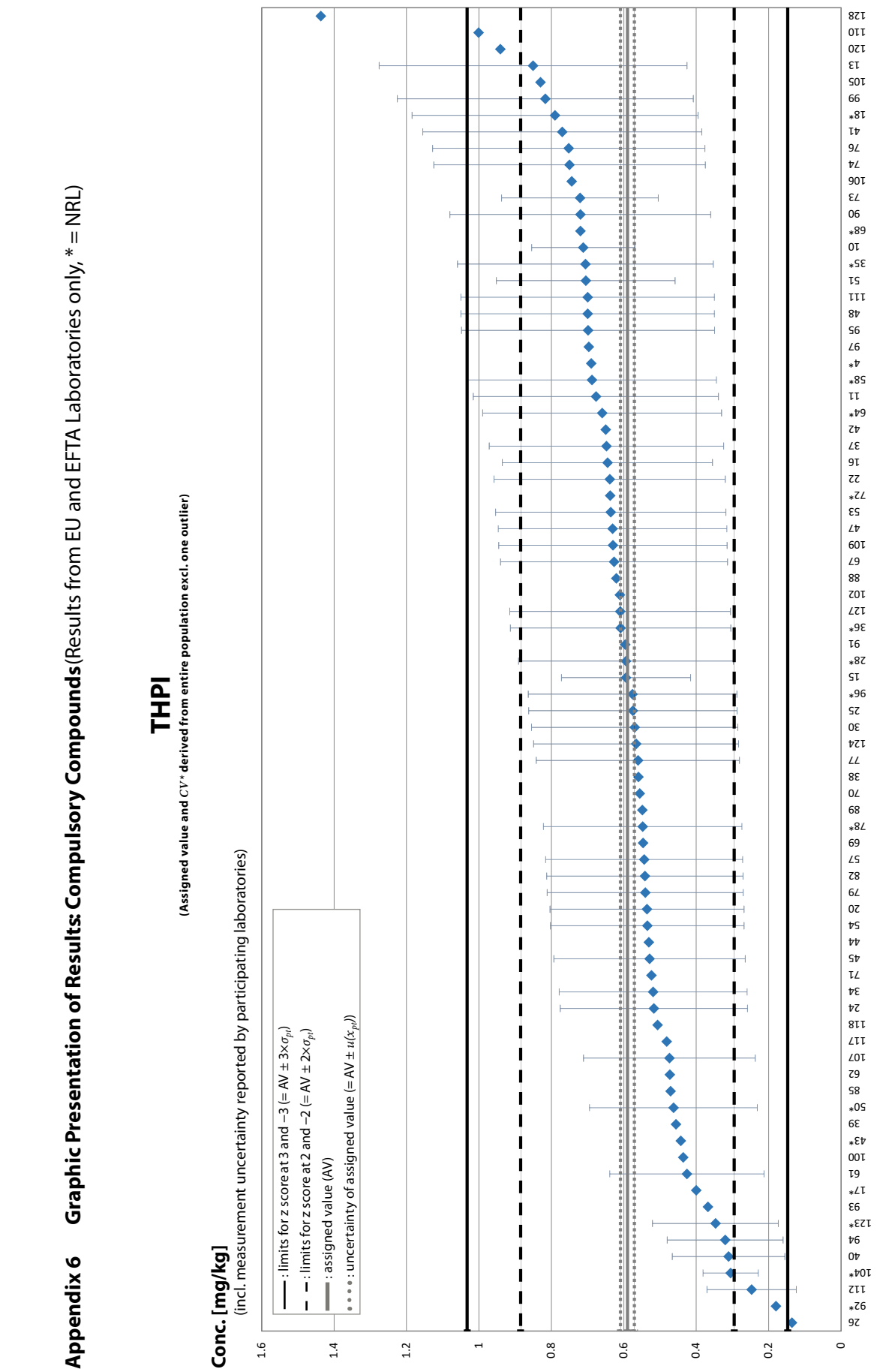
(incl. measurement uncertainty reported by participating laboratories)



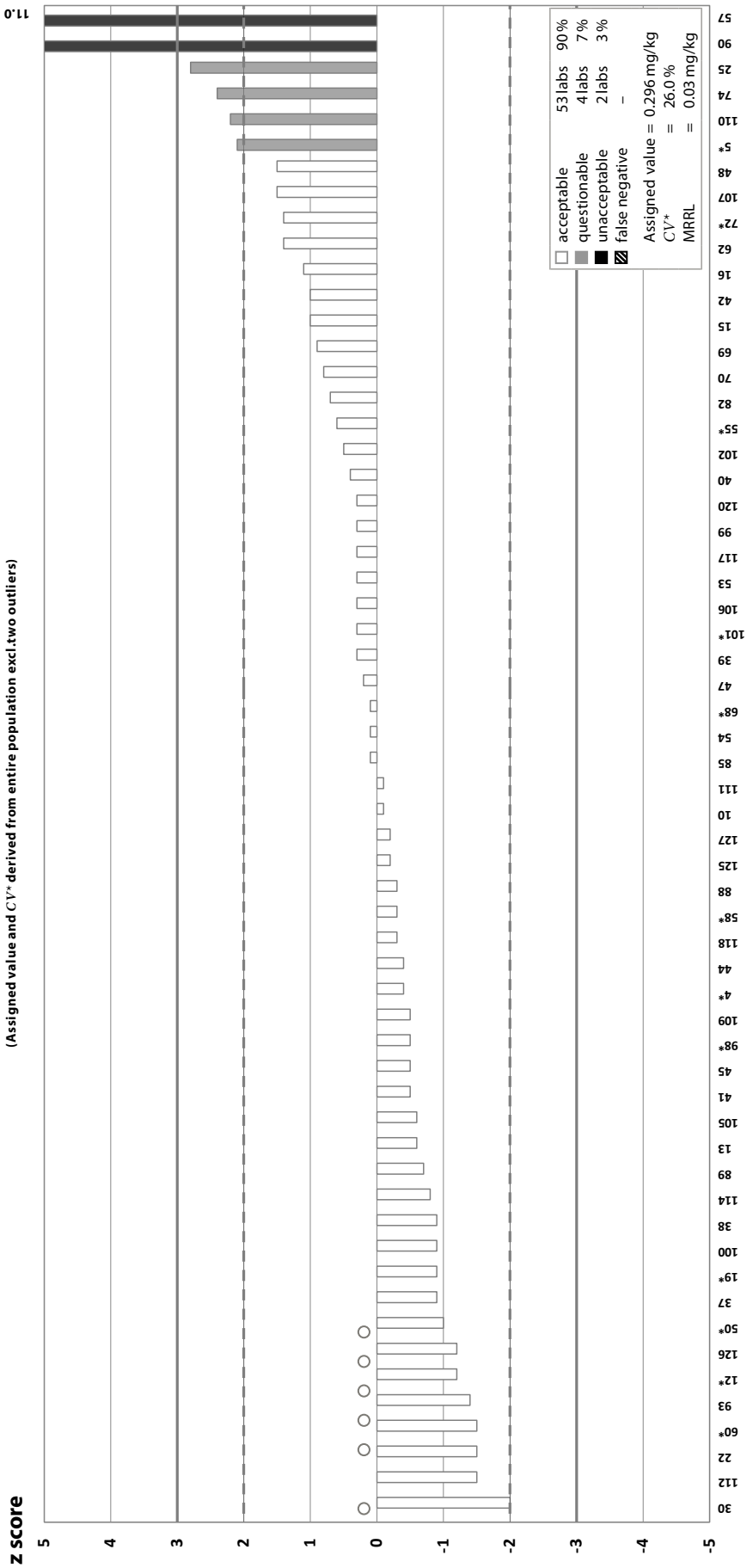
**Appendix 6 (cont.) Graphic Presentation of z Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



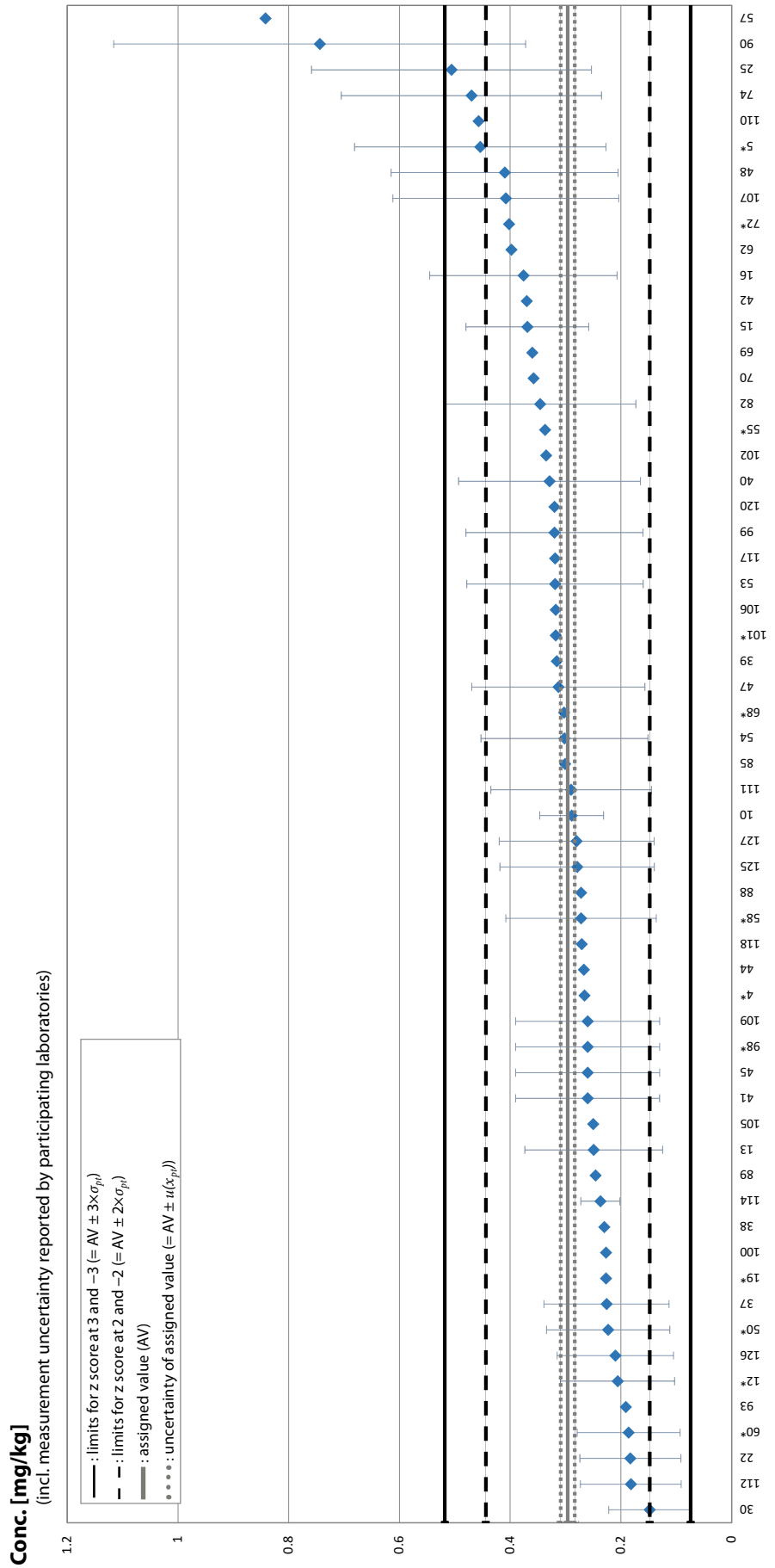
**Appendix 6 Graphic Presentation of Results: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



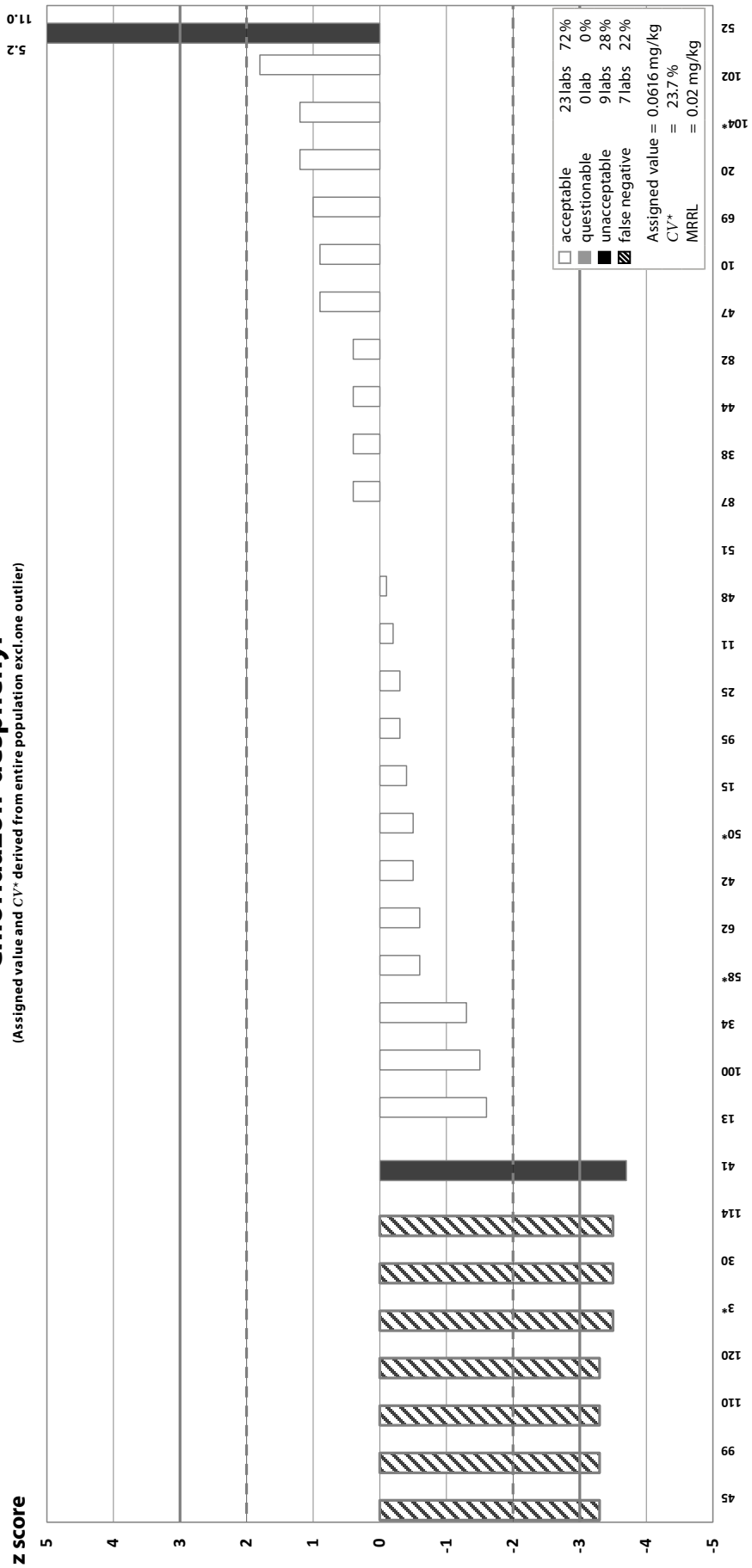
**Appendix 6 (cont.)** **Graphic Presentation of z Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



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



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



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

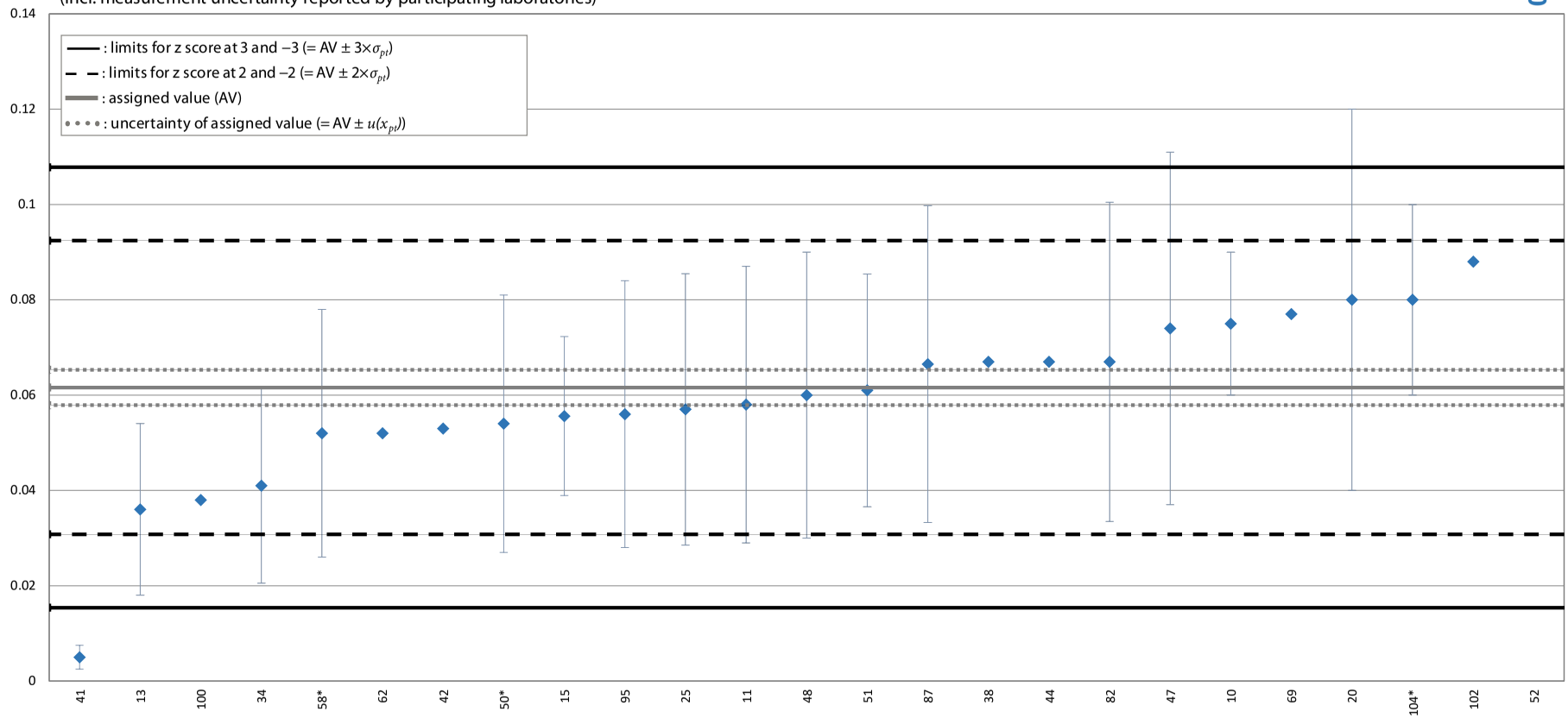
## Chloridazon-desphenyl

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

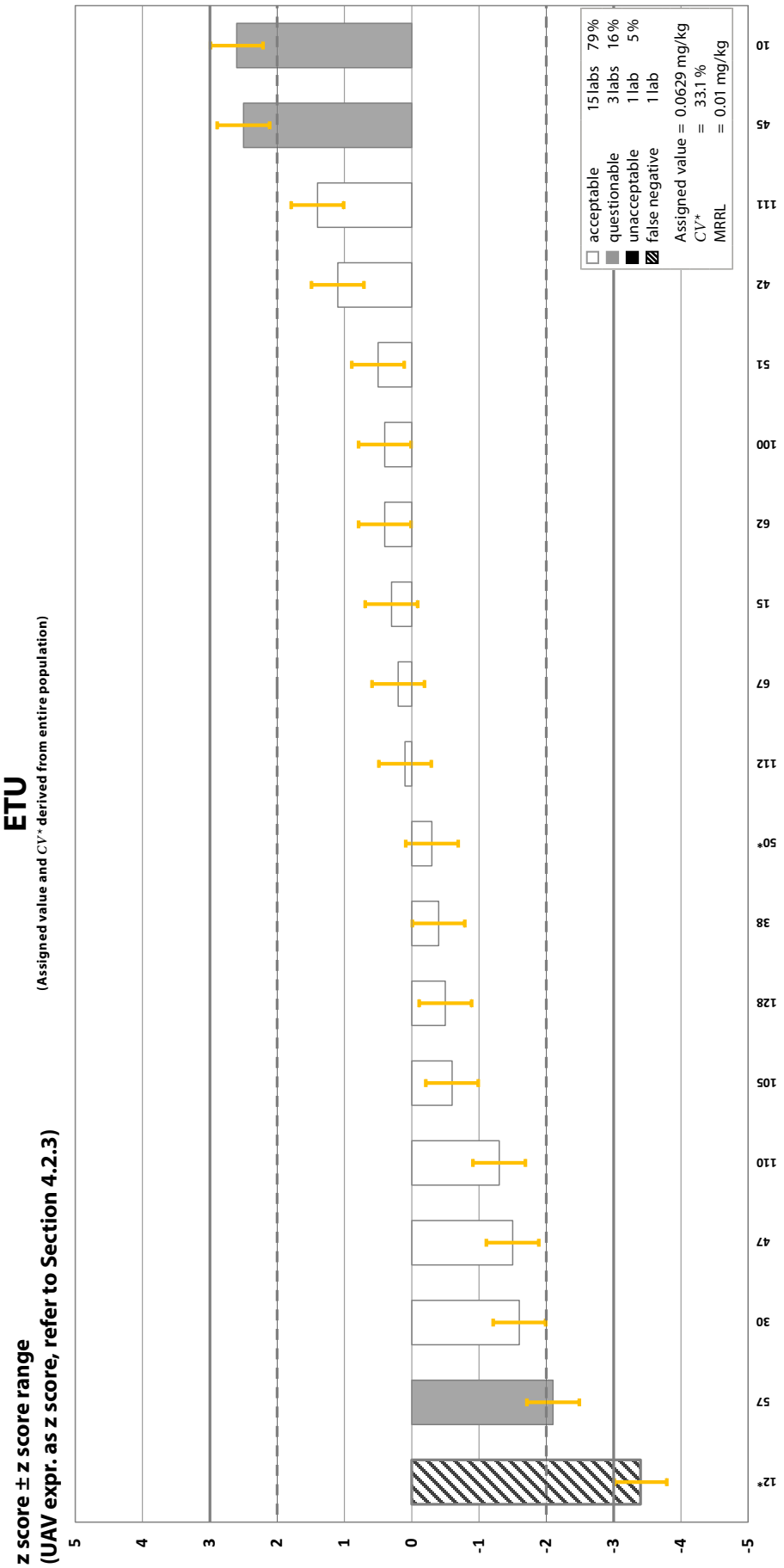
**0.668 ± 0.334**

**Conc. [mg/kg]**

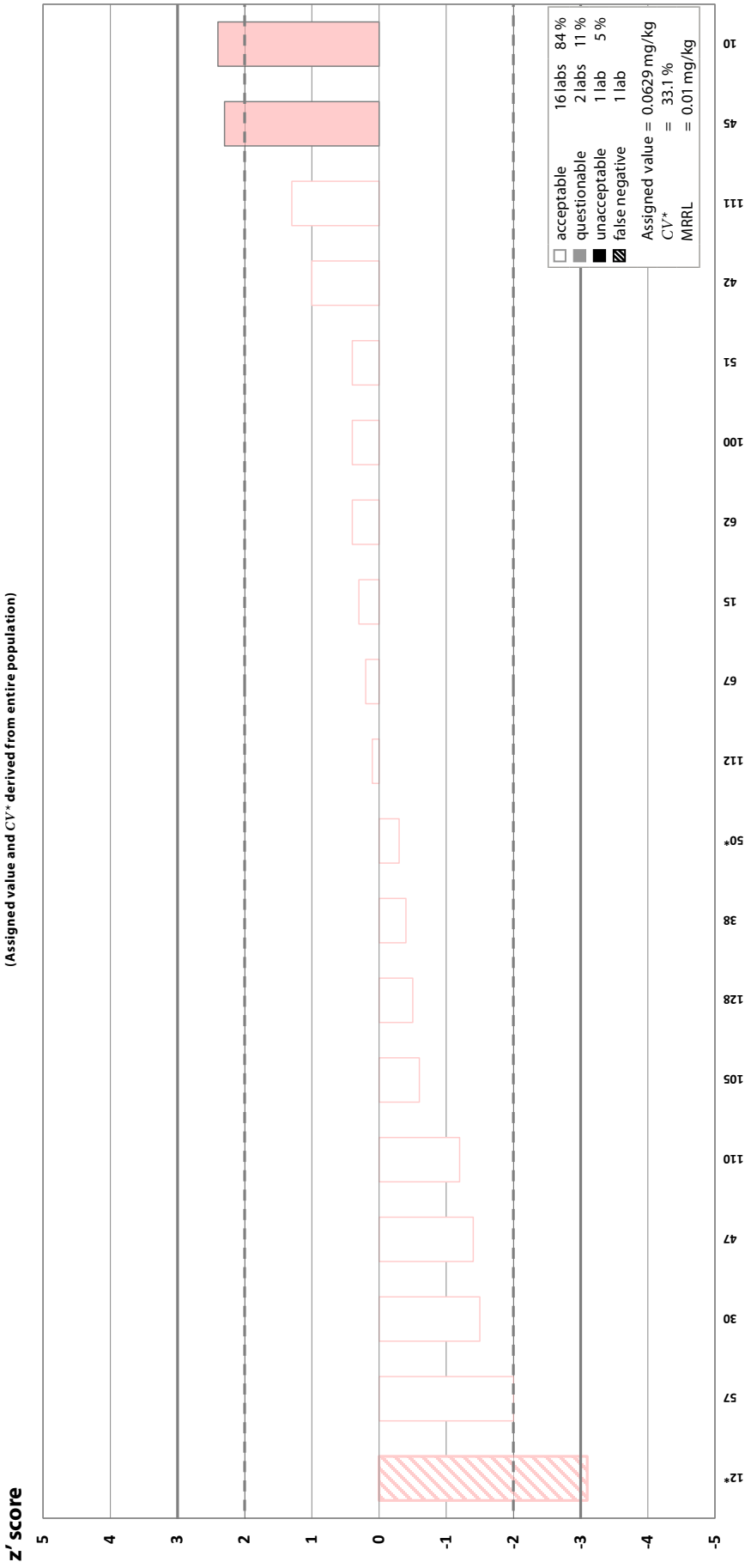
(incl. measurement uncertainty reported by participating laboratories)



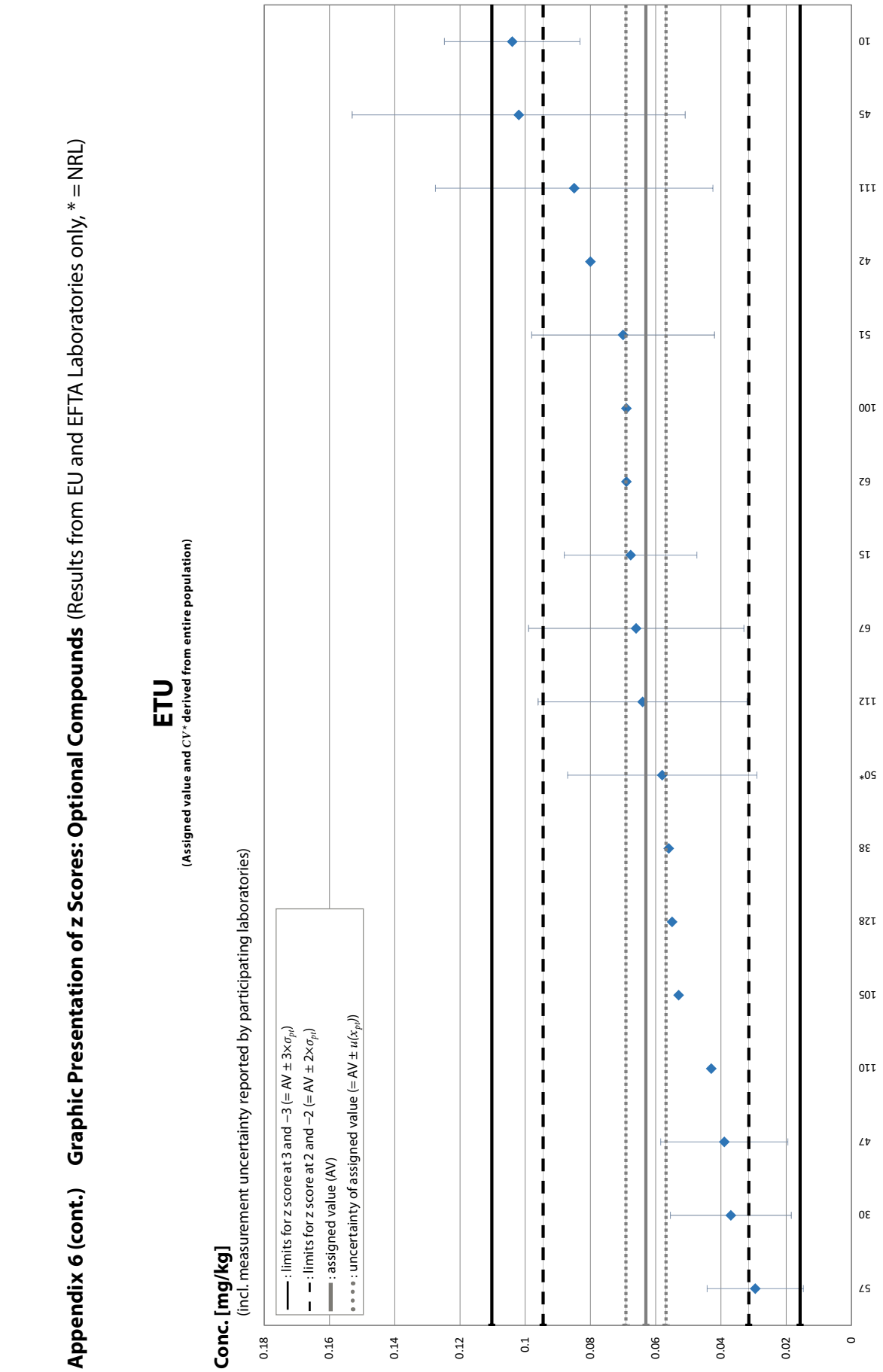
**Appendix 6 (cont.) Graphic Presentation of z Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



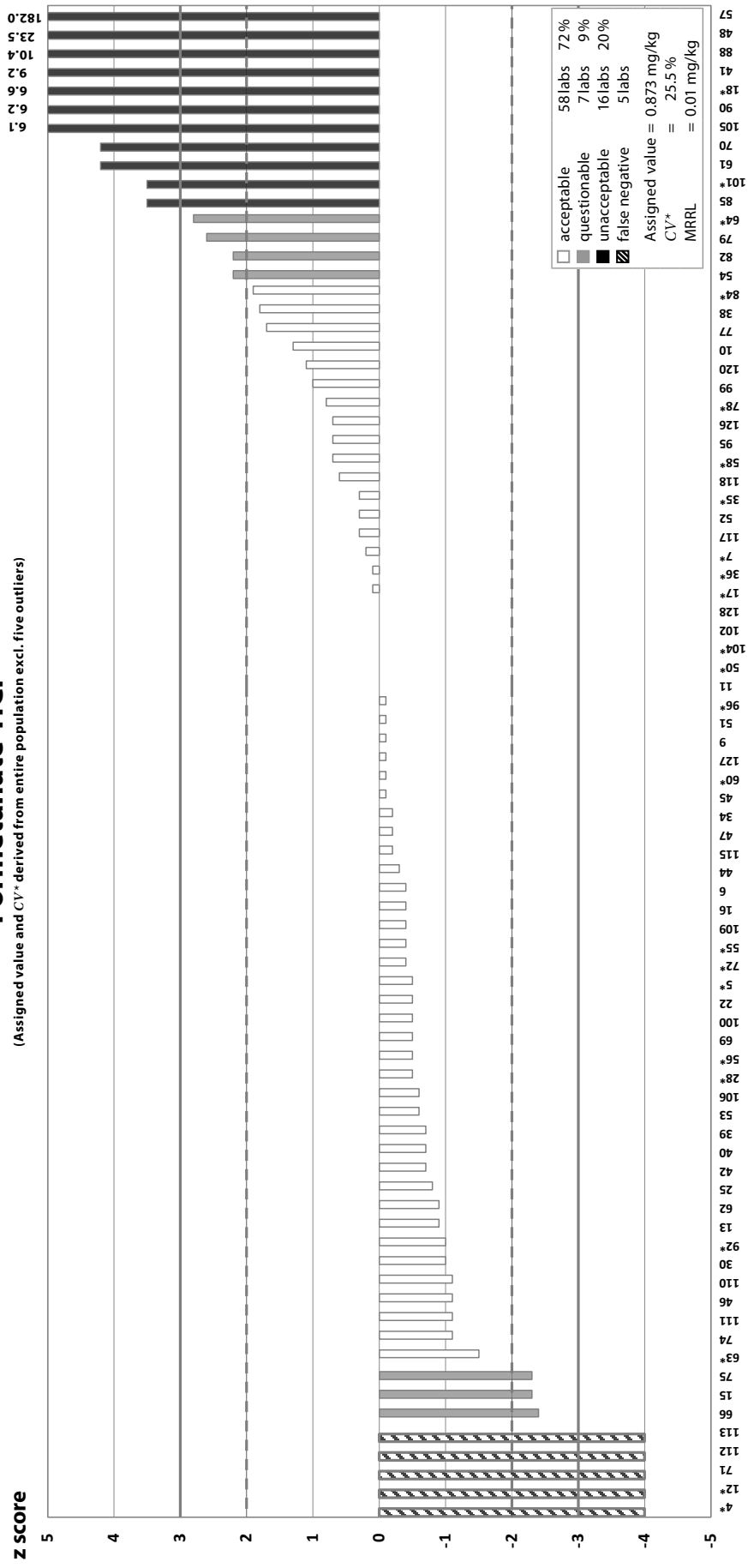
**Appendix 6 (cont.) Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



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



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



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

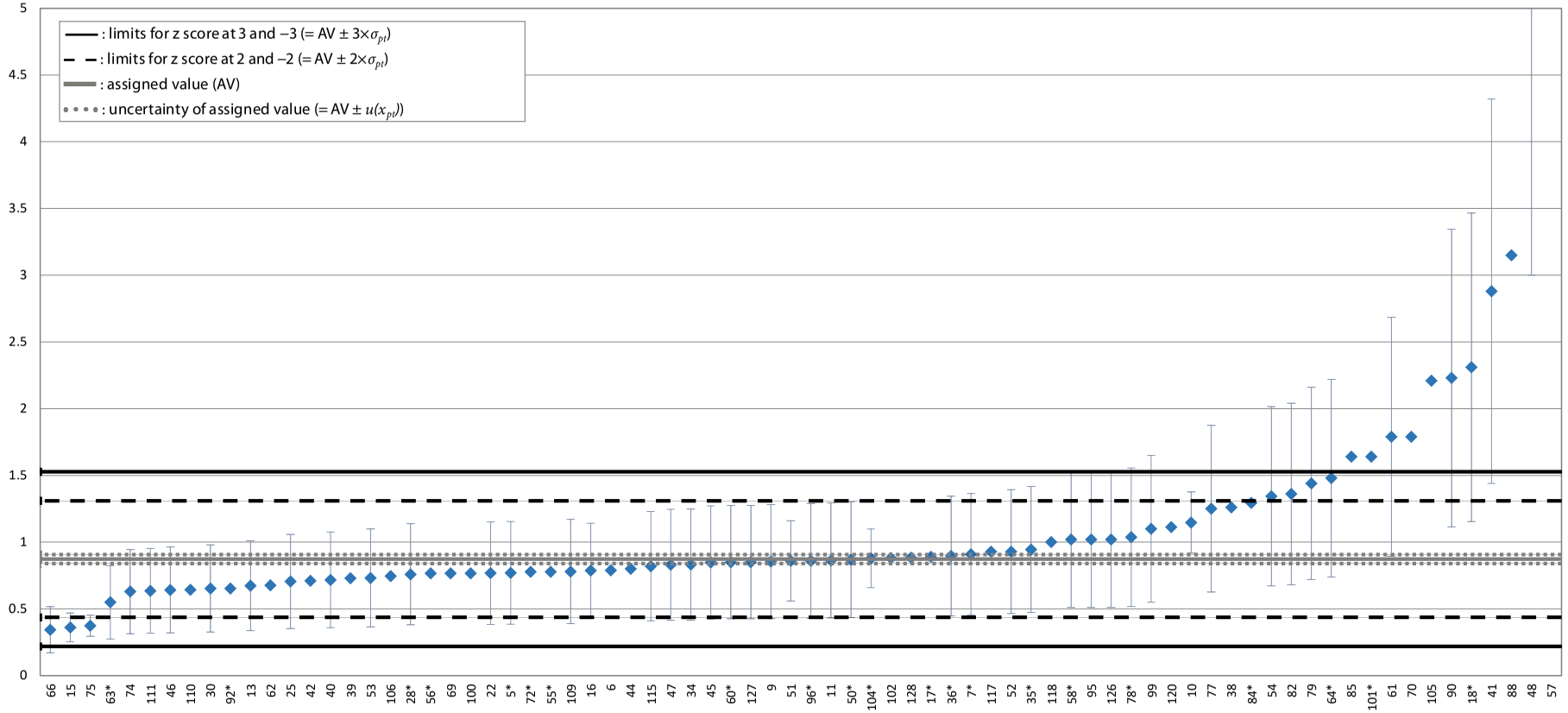
## Formetanate-HCl

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

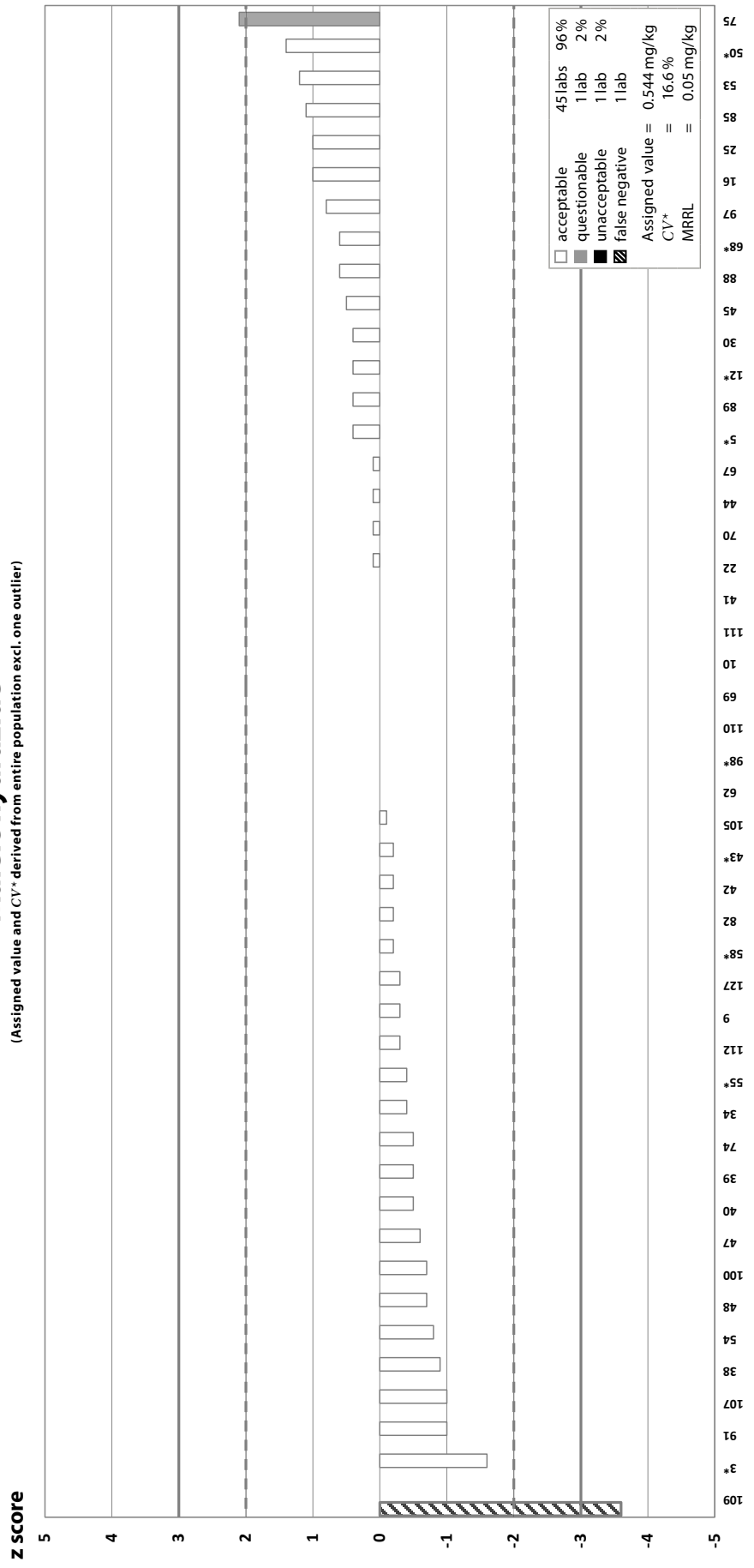
**Conc. [mg/kg]**

(incl. measurement uncertainty reported by participating laboratories)

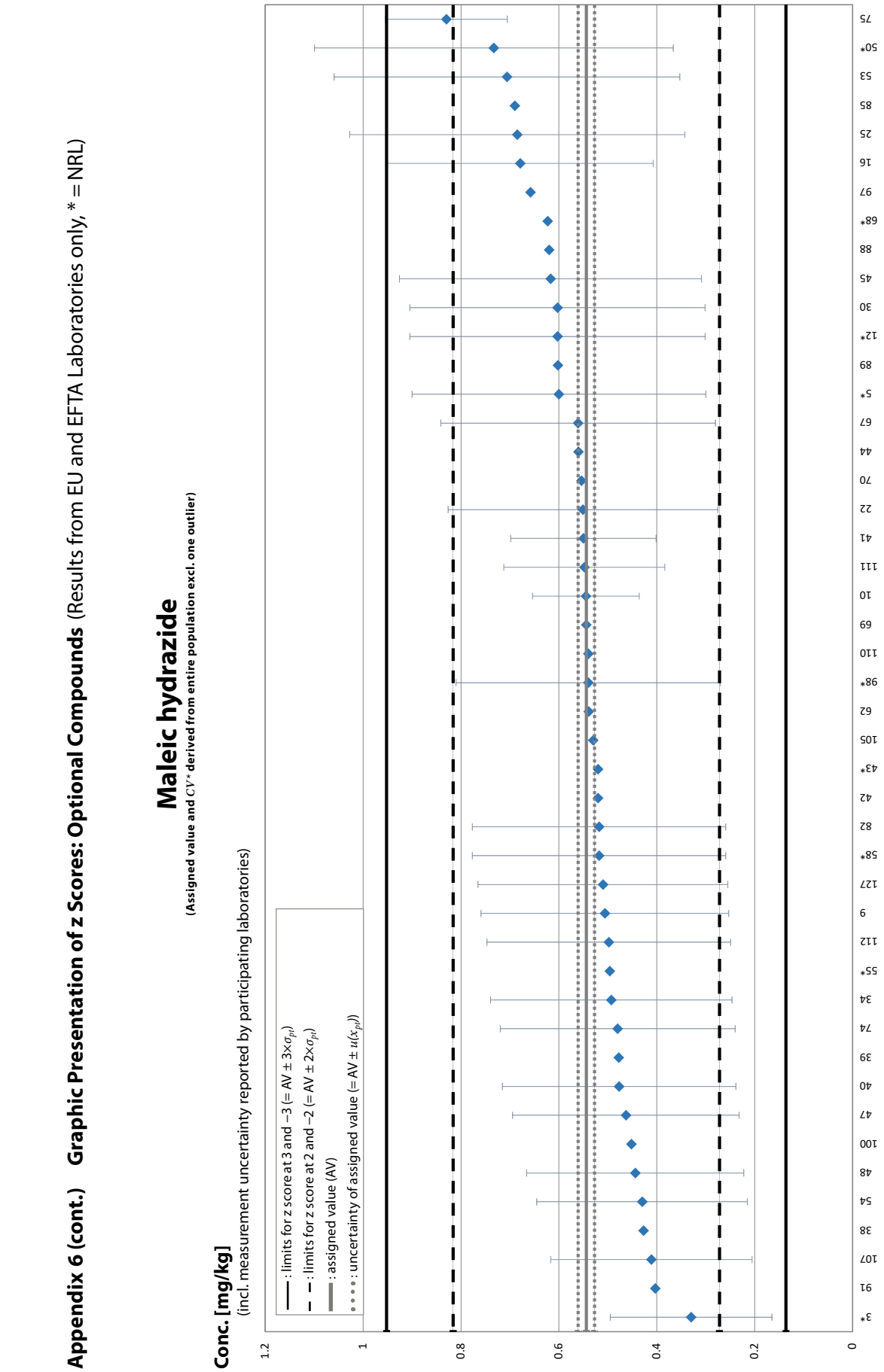
**6.0 ± 3.0**
**40.6 ± 20.3**



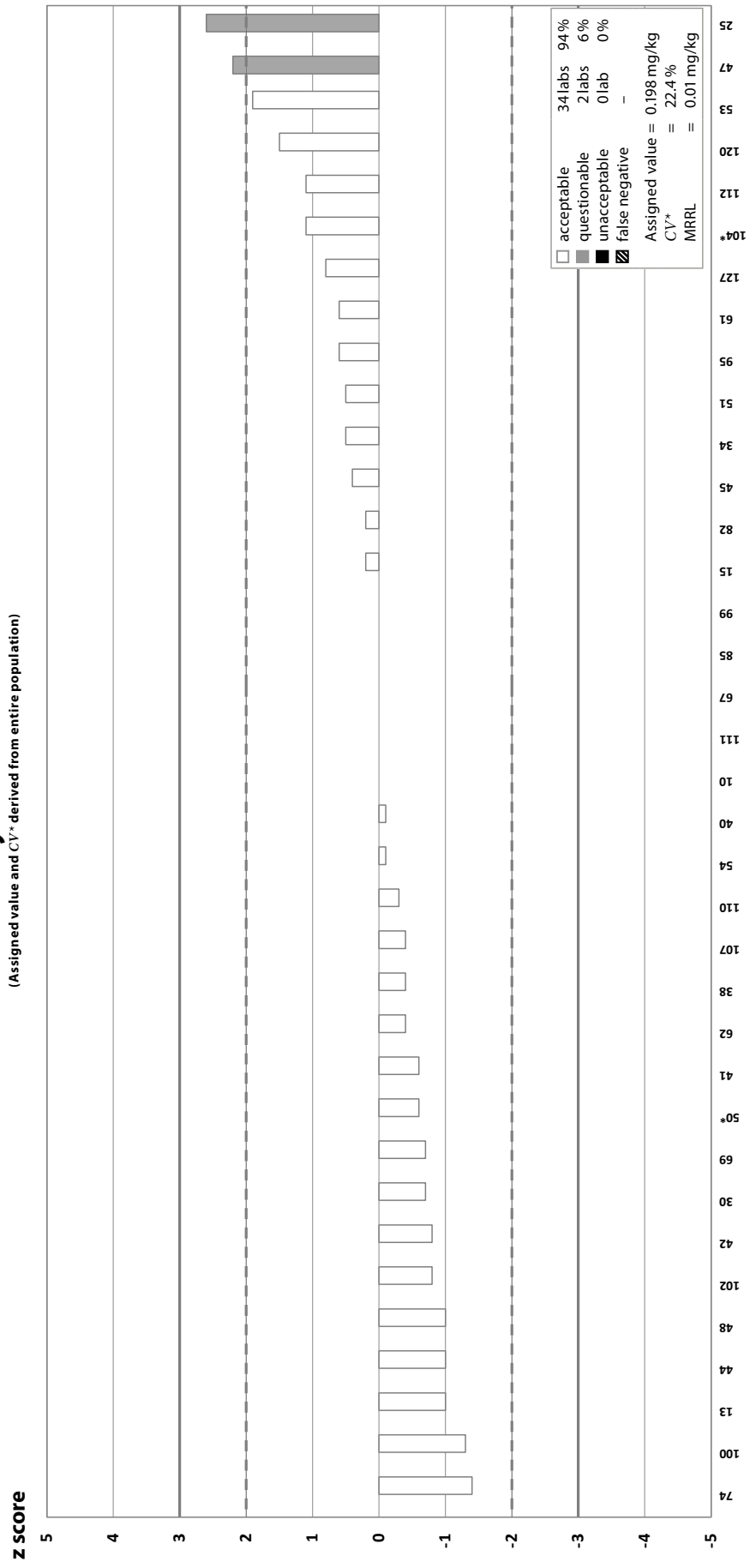
**Appendix 6 (cont.) Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



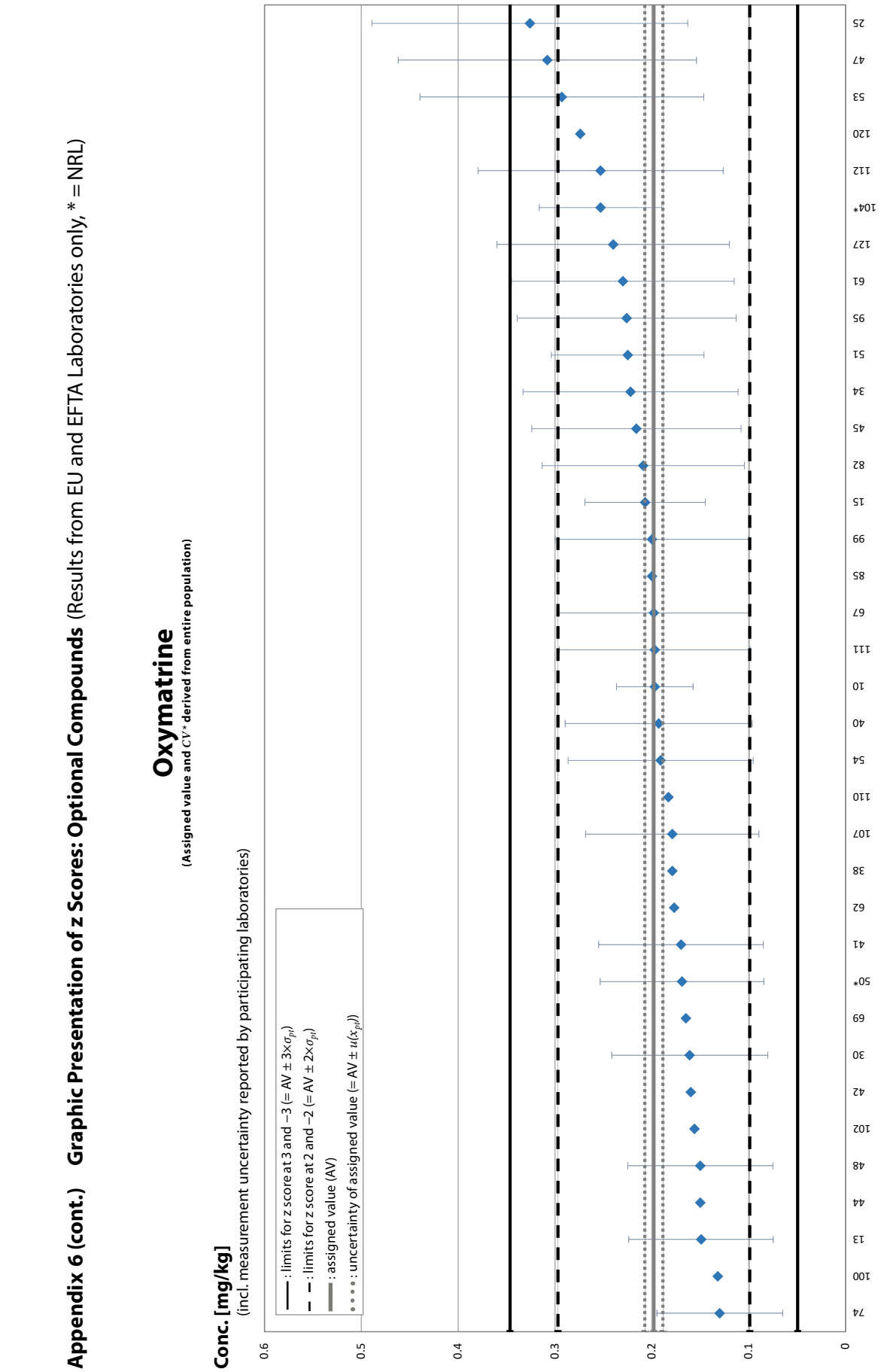
**Appendix 6 (cont.)** **Graphic Presentation of z Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



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



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



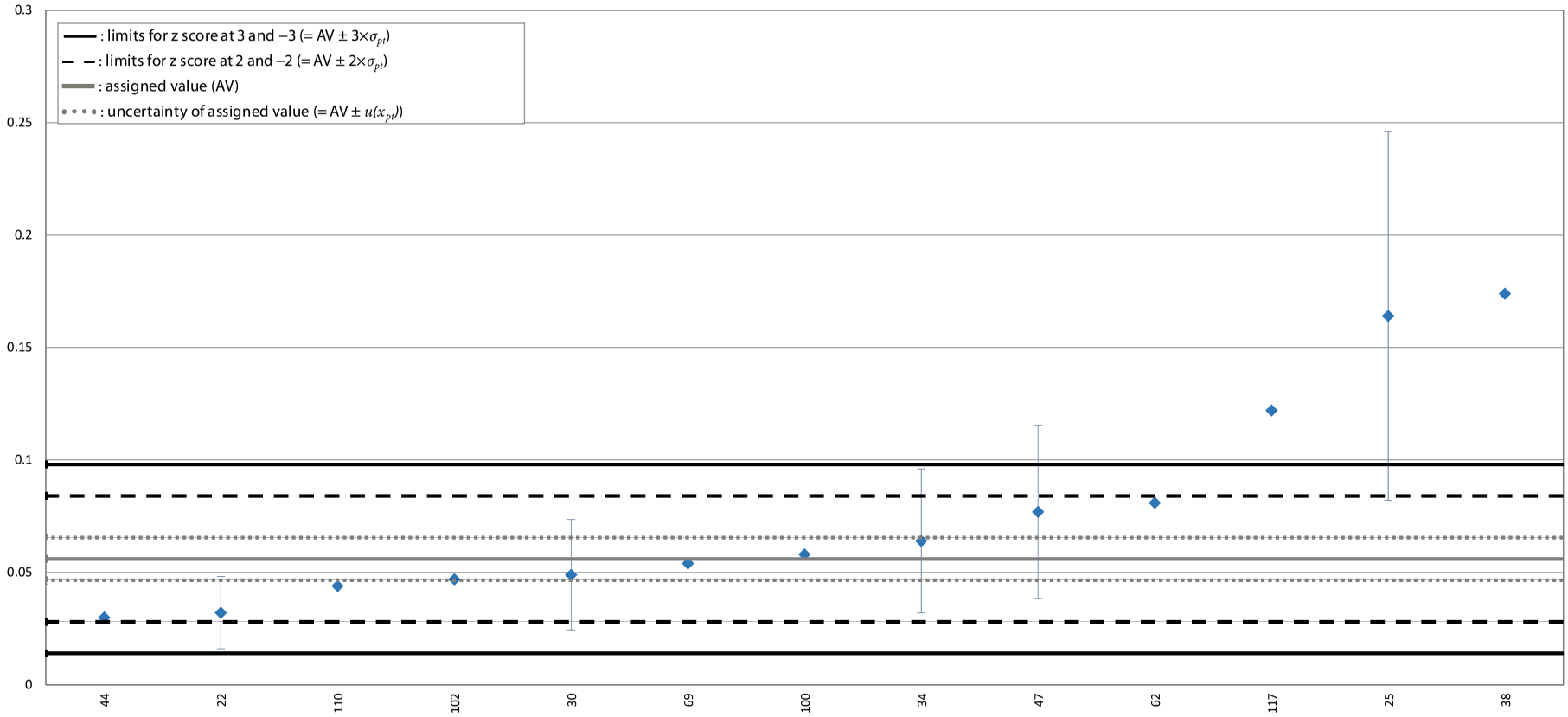
**Appendix 6 (cont.)** **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)

## 2,4-DNOP (free phenol)

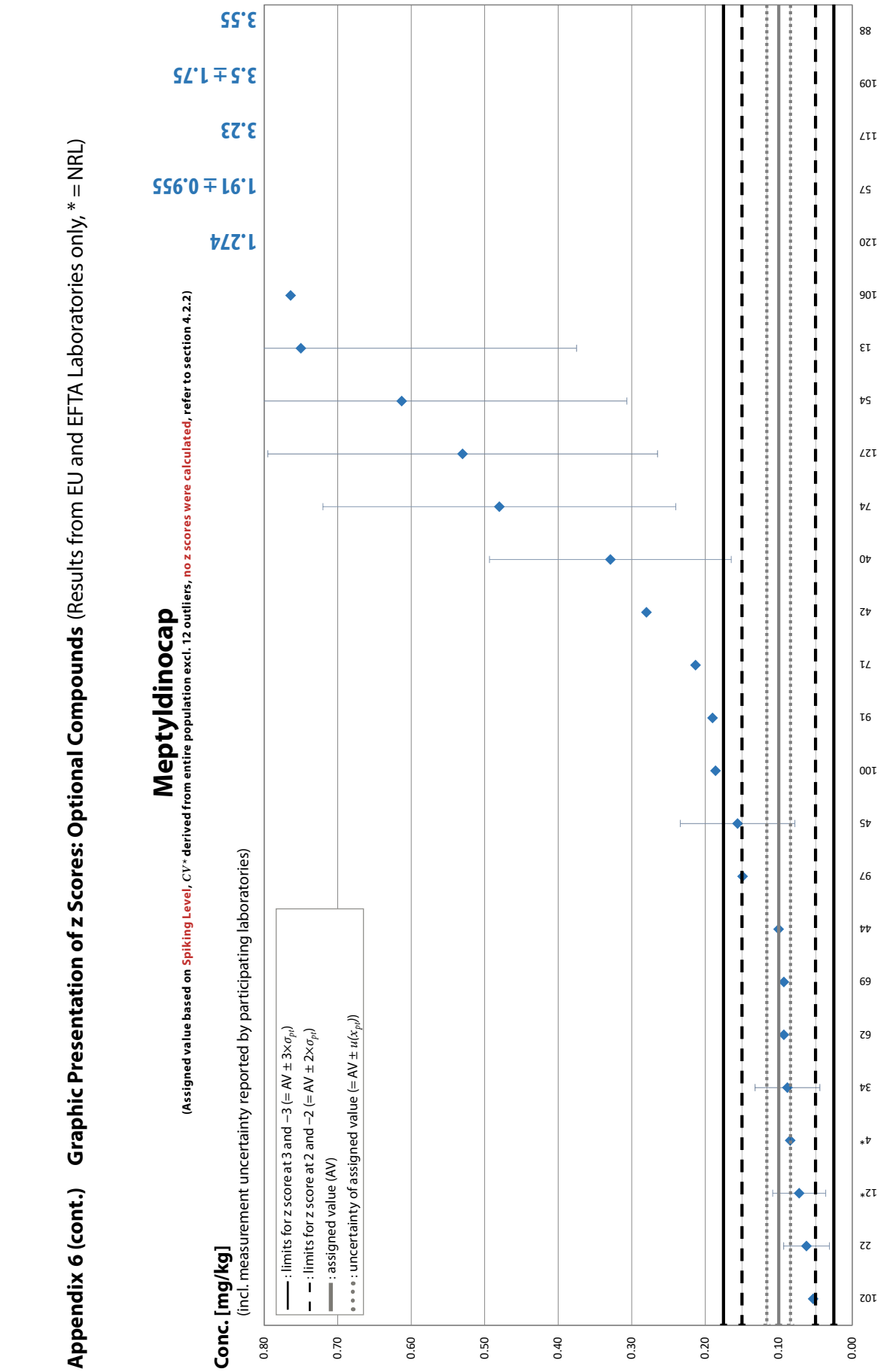
**(Assigned value based on Spiking Level, $CV^*$ derived from entire population excl. 2 outliers, no z scores were calculated, refer to section 4.2.2)**

**Conc. [mg/kg]**

(incl. measurement uncertainty reported by participating laboratories)



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



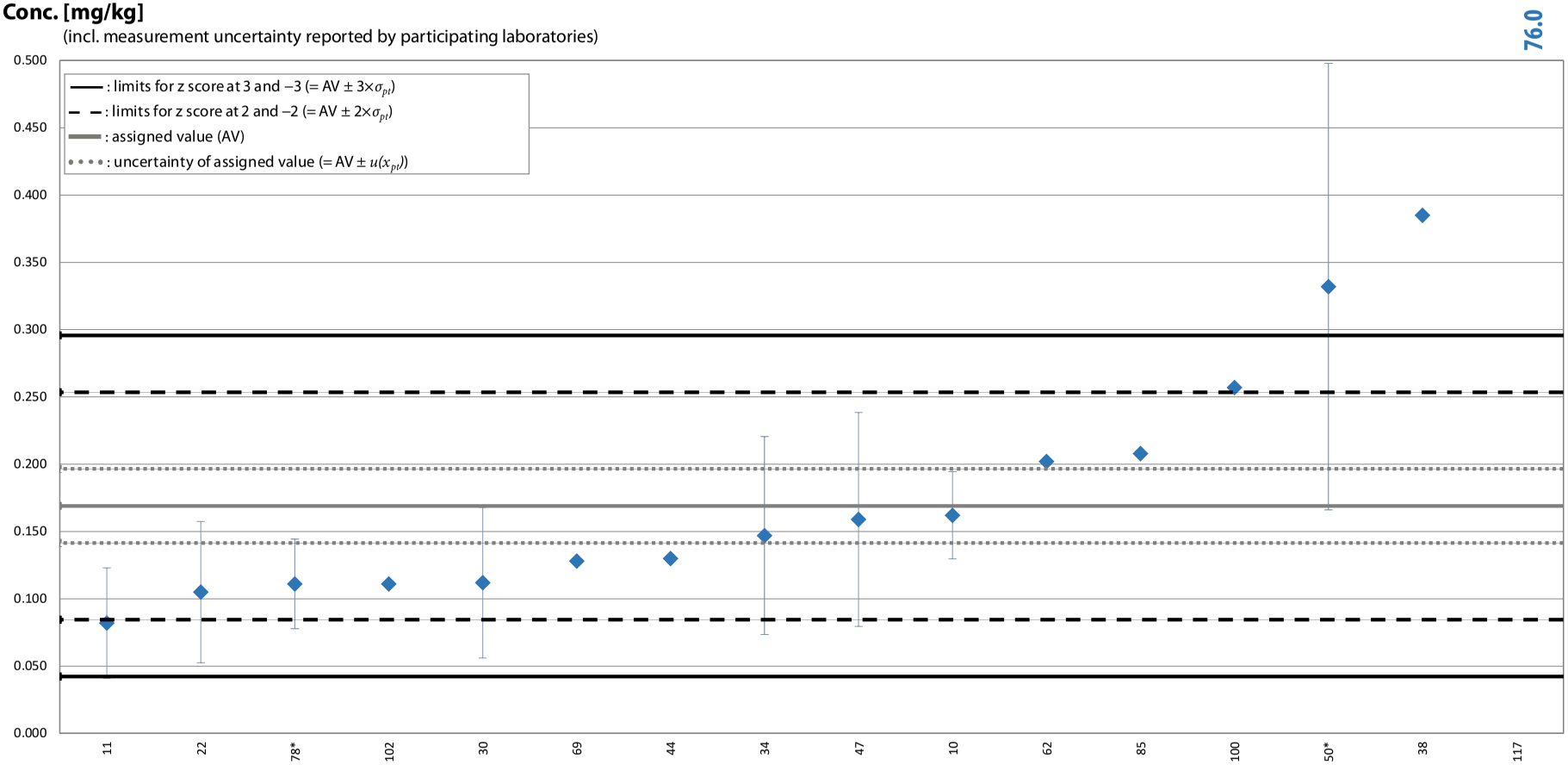
**Appendix 6 (cont.)** **Graphic Presentation of Results: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)

## Meptyldinocap (sum)

**(Assigned value based on Spiking Level, $CV^*$ derived from entire population excl. 1 outlier, no z scores were calculated, refer to section 4.2.2)**

**Conc. [mg/kg]**

(incl. measurement uncertainty reported by participating laboratories)



## Appendix 7 Possible Reasons Reported for Poor Performance (ordered by z score)

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

| **Captan** Assigned value: 0.174 mg/kg, $CV^*$: 32.9 % | | | |
|---|---|---|---|
| LabCode | z Score | Reason | Remarks/Details |
| 13 | -3.8 (FN) | D | |
| 30 | -3.8 | D, E | Possible degradation in the extract after sample prepaparation. Captan is analysed using a selective method on LC-MSMS. We have noticed during the validation that folpet and captan are unstabile in the LC-MSMS extract. Besides that, the sample was for 3 days in the refrigerator before starting the extraction for the determination of captan with LC-MSMS. It is not known by our lab if captan can break down in the refrigerator during storage.<br>**[Note by the Organizer:** A degradation of folpet and captan during storage of thawed homogenates was noticed by the organizers irrespective if the homogenates were stored in the refrigerator or on the bench. Keep your samples frozen untill analysis!. If in your routine work sample homogenates are kept in a non-frozen state prior to analysis, consider minimizing the time interval until the start of the extraction. Alternatively, consider switching to cryogenic processing. Furthermore, note that you generally shouldn't report results that are lower than your own RL.] |
| 76 | -3.6 | D, E | Degradation of Captan to THPI in the GC-Injector (and maybe during analysis), sum of THPI (expressed as Captan) and Captan is correct (112% of preAV sum of both analytes).<br>[**Note by the Organizer**: Having added ILIS at the beginning of the procedure any losses during analysis (incl. measurement) would have been noticed and accounted for. Your calibration via StAdd to extract aliquots would have additionally corrected for any decomposition in the GC-injector. It rather looks as if captan decomposed prior to ILIS addition. Most of the above also apply to folpet, which shows the same trend.] |
| 3 | -3.5 (FN) | E | [**Note by the Organizer**: It is noted that you haven't reported any recovery rate for this compound. Please check whether comment "K: Deficient QC-measures that would have helped to recognize that method generates FNs ..." applies in your case.] |
| 66 | -3.5 (FN) | B, D, E | Problems caused by the Covid-19 pandemic, including acute lack of personnel and necessary consumables, significantly affected our ability to fully participate in this proficiency test. Single residue methods for the compounds in the PT scope were under validation. For this we reported some of the results from our multi residue analysis.<br>[**Note by the Organizer**: With your RL being higher than the AV (0.5 versus 0.174 mg/kg), your result wouldn't be technically a FN in a routine setup. There is an urgent need to adjust your methodology in a way that ensures that you RLs cover all MRLs of this substance in food products.]. In any case, irrespective of the method used, QC measures should be such to avoid false negatives. Therefore, please check whether comment "K: Deficient QC-measures..." applies in your case.] |
| 73 | -3.5 (FN) | D | [**Note by the Organizer**: Please read the organizers-comment for laboratory LabCode 106] |
| 106 | -3.5 (FN) | M, B | Captan is measured in our laboratory as the degradation product THPI. The THPI detected by our analysis contains both the THPI in the sample and Captan degraded to THPI. Since the residue definition for Captan is ""Captan (Sum of captan and THPI, expressed as captan)"" (Reg. (EU) 2019/1015), in routine analyses only the total amount of Captan is relevant, not the separate amounts of undegraded Captan and THPI.<br>In the proficiency test SRM-17, the result of our lab for the sum of THPI and Captan is 1,481 mg/kg Captan. The assigned values (taken from the preliminary report) sum up to 1,346 mg/kg Captan. The difference for the total Captan residue (as used in the residue definition) is just 10%.<br>**[Note by the Organizer:** Indeed, your THPI result expressed as captan is close to the "AV" of Captan (sum). As you do not analyze for captan as such, you shouldn't have reported that you are analyzing captan. In future PTs the option for reporting captan (sum) will be given. In the case of folpet it looks as if your are analyzing differently, as you have reported results for both phthalimide and folpet] |
| 3rd-103 | -3.5 | C | [**Note by the Organizer**: It is noted that you haven't reported any recovery rate for this compound. Please check whether comment "K: Deficient QC-measures that would have helped to recognize that method generates FNs ..." applies in your case.] |

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

**Captan** Assigned value: 0.174 mg/kg, *CV**: 32.9 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 40 | -3.4 | M, D | For Reason 1 : This parameter degrades rapidly at room temperature. During the conduct of this test, the SRM17 sample spent approximately 18 hours on the bench without being re-frozen when it should have been. The analysis of Captan had only been carried out after this long thawing period. We thought this would be offset by the significant increase in THPI concentration. This information was transmitted when the results were reported on the EUPT website.<br>Series controls are good. No other issues identified.<br>[**Note by the Organizer**: We agree with your conclusions as regards captan. Your captan losses should normally be accompanied by an increase of THPI. This is, however, not reflected in your results. With your RL being higher than the AV (0.5 versus 0.174 mg/kg), your result wouldn't be technically a FN in a routine setup. Nevertheless, your very high RL compromises your ability to properly check for the compliance of most food to the MRL of captan. There is an urgent need to adjust your methodology in a way that ensures that you RL covers all food products.] |
| 120 | -3.3 | D, E | Problem with degradation of Captan to THPI in inlet injector during analysis, lab doesn't use isotopically labelled standards yet. |
| 41 | -2.5 | D, E | [**Note by the Organizer**: Defrosting your homogenate over many hours has obviously led to your underestimated result. See comments under LabCode 30.] |
| 10 | -2.2 | D, E | Analyte loss during inappropriate sample handling (multiple inappropriate thawing of sample). THPI-Level increased due to degradation of Captan. Sum (Captan/THPI) in a comparable concentration range as assigned value.<br>[**Note by the Organizer**: See comment under LabCode 30] |
| 3rd-83 | -2.2 | M | No issue found with the data. The combined z score for captan and THPI (residue definition) would have been with ±2 |
| 39 | 2.4 | F | The sample extract was measured both undiluted and diluted. By mistake, the result of the undiluted extract was reported although this was outside the calibration range. For the diluted extract, a result of 0,166 mg/kg was obtained, which was conform (z score = -0,14) |
| 88 | 2.5 | J, E | [**Note by the Organizer**: Consider checking the stability of your stock and working solutions and adjust your storage conditions where needed. Note that Captan and Folpet are sensitive to high pH and to high temperatures. Acidification of standard solutions, e.g. with acetic acid, often helps to increase stability.] |
| 17 | 2.7 | J | [**Note by the Organizer**: See note under LabCode 88.] |
| 49 | 2.7 | H | Captan and Folpet recoveries were mixed and Captan result was corrected with wrong recovery. Actually Captan result needed no correction and was 0,191 and Folpet result should have been 0,224 after correction. In this case Z-scores would have been 0,5 for Captan and -0,55 for Folpet |
| 100 | 3.2 | E, F | No reason identified clearly after several re-analysis. The results are not repeatable but rather high. Amelioration with a LC analysis will be tested in the coming weeks. The utilisation of captan D6 in another considered option for an amelioration.<br>[**Note by the Organizer**: See note under LabCode 88.] |
| 5 | 3.9 | C, E, M | We are in the process of identifying the exact cause of the problem.<br>[**Note by the Organizer**: See note under LabCode 88.] |
| 112 | 9.6 | M | Sum of Captan and THPI calculated as Captan is nearly the same as in the preAV; we have decided to transfer results, we had with the first analytical approach<br>[**Note by the Organizer**: See note under LabCode 88.] |
| 56 | 28.6 | A, E, F | New value : 0,203 mg/kg<br>[**Note by the Organizer**: See note under LabCode 88.] |
| 90 | 29.7 | B, A, J | [**Note by the Organizer**: See note under LabCode 88.] |
| 42 | 47.2 | J | Captan standard partially degraded in THPI, hence the measurement of captan in the sample.<br>[**Note by the Organizer**: Your insights seem plausible. See also note under LabCode 88.] |

**Chlorothalonil** Assigned value: 0.151 mg/kg, CV*: 24.4 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 61 | -3.7 (FN) | D | [**Note by the Organizer**: It is noted that you haven't reported any recovery rate for this compound. It is also highlighted that chlorothalonil is known not to give sensitive signals in LC-MS. Check the appropriateness of your method. Also please check whether comment "K: Deficient QC-measures that would have helped to recognize that method generates FNs ..." applies in your case.] |
| 3rd-103 | -3.7 | F, M | There was mistyping in the result sheet. The detection concentration of chlorothalonil was 0.096 mg/kg. |

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

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

**Chlorothalonil** Assigned value: 0.151 mg/kg, CV*: 24.4 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 57 | -3.4 | D | [**Note by the Organizer**: It is highlighted that chlorothalonil is known to be sensitive to degradation, especially at high pH. The fact that you left your sample defrosted for many hours and that you have conducted dSPE with PSA may have affected your result.Extracts need to be quickly re-acidified following dSPE with PSA.] |
| 90 | -3.0 | B | |
| 66 | -2.6 | B, D, G | Problems caused by the Covid-19 pandemic, including acute lack of personnel and necessary consumables, significantly affected our ability to fully participate in this proficiency test right now. Single residue methods for the compounds in the PT scope were under validation. For this we reported some of the results from our multi residue analysis. |
| 48 | 2.5 | M | No causes identified so far; solution intact; Chlorothalonil, Captan and Folpet were analyzed by Mini Luke; Recoveries were OK<br>[**Note by the Organizer**: Consider checking the stability of your stock and working solutions and adjust your storage conditions where needed. Note that Chlorothalonil is sensitive to high pH and to high temperatures. Acidification of standard solutions, e.g. with acetic acid, often helps to increase stability.] |
| 56 | 3.0 | H, J | New value : 0,176 mg/kg<br>[**Note by the Organizer**: Consider checking the stability of your stock and working solutions and adjust your storage conditions where needed. Note that Chlorothalonil is sensitive to high pH and to high temperatures. Acidification of standard solutions, e.g. with acetic acid, often helps to increase stability.] |
| 7 | 7.1 | H, M | Result corrected for low recovery in PT sample (46%). Recovery in pear in same sequence 105%. Standard used was prepared December 13, new standard prepared April 20 showed a deviation of 36% from the old (lower signal in old)<br>[**Note by the Organizer**: It seems as if the solvent/conditions used for the analytical standard solutions are not appropriate for long-term storage. Chlorothalonil is sensitive to high pH and to high temperatures. Acidification of standard solutions, e.g. with acetic acid, often helps to increase stability.] |

**Cyromazine** Assigned value: 0.154 mg/kg, CV*: 20.3 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 57 | -2.4 | J | [**Note by the Organizer**: Your underestimated result is not plausible at first sight, considering that you have employed an ILIS, which was added at the beginning of the procedure. It is, however, noted that you haven't reported any recovery figure. Consider checking whether the QC measures undertaken were appropriate to recognize any systematic bias] |
| 76 | -2.2 | H | Result not corrected for recovery (41%) |
| 90 | -2.2 | E | [**Note by the Organizer**: Consider the use of ILIS to compensate for matrix effects, especially when calibrating against solvent or against a generic matrix.] |
| 41 | -2.1 | E | [**Note by the Organizer**: See comments under LabCode 90.] |
| 17 | 3.5 | J | [**Note by the Organizer**: See comments under LabCode 90.] |
| 101 | 3.7 | M | Standard addition was used, suspected problem with matrix effects on a new instrument. Investigation is on going.<br>[**Note by the Organizer**: Matrix effects and even recovery bias, should have been addressed by the standard addition to sample portions approach you have employed. The use of ethylacetate as solvent may have resulted in poor recoveries and a high variability. A high variability (poor linearity) increases the uncertainty in the case of a standard additions approach. This remark also applies to dodine, where, using the same calibration approach, the result was also strongly biased. Also consider using an ILIS to better compensate for spurious and systematic errors.] |

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

| **Dithiocarbamates (expr. as $CS_2$)** Assigned value: 0.187 mg/kg, CV*: 28.4 % | | | |
|---|---|---|---|
| LabCode | z Score | Reason | Remarks/Details |
| 37 | -3.8 | L | result analysis for Dithiocarbamate was 0,127 mg/kg |
| 120 | -3.2 | D | |
| 64 | -3 | C, J | |
| 69 | -2.4 | I | |
| 123 (FN) | -2.3 | I | The sample was reconstituted in doble the volume it should. |
| 3rd-83 | -2.1 | M | No issue found with the data. The z score is bordeline. |
| 50 | 2.4 | F, G | |
| 56 | 2.4 | A | New value : 0,254 mg/kg |
| 20 | 2.6 | H | |
| 12 | 3.5 | E | Equipment with sensitivity oscilation<br>[**Note by the Organizer**: Consider checking whether your CS2 stock and working standards are prepared and stored in a way that they are correct and stable. Keeping low temperatures, tightly closing vessels, keeping the headspace volume small and avoiding frequent reopening of vessels is helpfull.] |
| 61 | 3.7 | I | [**Note by the Organizer**: See comments under LabCode 12.] |
| 62 | 3.9 | B | [**Note by the Organizer**: See comments under LabCode 12.] |
| 60 | 4.8 | M | The standards were checked and the PT-material was re-analysed. However, we obtained the result in the same concentration level than during PT-round. The instrument was performing well, blank matrix was OK, calibration was appropriate, and all calculations were also double-checked. So far no specific reason has been found for poor result. We will prepare still a new Thiram standard and test/compare it with the old one. We participated dithiocarbamate-PT (lettuce, FAPAS) in September 2021 with the same analytical method and the result was good (Z 1,2).<br>[**Note by the Organizer**: See comments under LabCode 12.] |
| 43 | 8.8 | A | We usually never perform the $CS_2$ method (only for EUPTs), the method is not optimised in our lab.<br>[**Note by the Organizer**: See comments under LabCode 12.] |
| 10 | 9 | G, H | [**Note by the Organizer**: See comments under LabCode 12.] |

| **Dodine** Assigned value: 0.1 mg/kg, CV*: 23.1 % | | | |
|---|---|---|---|
| LabCode | z Score | Reason | Remarks/Details |
| 101 | -3.0 | M | Standard addition was used, suspected problem with matrix effects on a new instrument. Investigation is on going<br>[**Note by the Organizer**: See comments on Cyromazine regarding the standard addition to sample portions.] |
| 84 | -2.1 | A, B | This is a compound normally analysed as SRM but in our case was used a multiresidual method with 570 compounds recently developed |
| 5 | 2.1 | C, E, M | We are in the process of identifying the exact cause of the problem.<br>[**Note by the Organizer**: Consider checking whether any of the remarks under LabCode 20 applies to you.] |
| 20 | 2.2 | H | [**Note by the Organizer**: Please consider that dodine tends to interact with surfaces both in glass vials and within the LC-injector. Such interactions are more pronounced in absence of matrix and may lead to underestimated signals when injecting solvent based calibration standards or preparing standards based using a diluted standard solution. The losses of dodine when using solvent-based calibration standards (as in your case) lead to oevestimated results. Such types of losses should normally be recognizable through overestimated recovery rates (see Analytical Observation Report SRM-15). Unfortunately, no recovery rate was reported. Therefore, please consider that "K: Deficient QC-measures ..." may be a possible point to address.] |
| 66 | 2.2 | H, I, B | If the result had been corrected for recovery and a factor of 0.791 (free dodine in CAS 2439-10-3) had been used for calculation, the result would have been 0.073 mg/kg (inside the range of method MU 50 %). Because of the unusual circumstances at the time of this PT this procedure was not followed.<br><br>Problems caused by the Covid-19 pandemic, including acute lack of personnel and necessary consumables, significantly affected our ability to fully participate in this proficiency test right now. Single residue methods for the compounds in the PT scope were under validation. For this we reported some of the results from our multi residue analysis.<br>[**Note by the Organizer**: Consider checking whether any of the remarks under LabCode 20 applies to you. Your overestimated recovery rate of 168% points towards this direction.] |
| 93 | 2.6 | H | [**Note by the Organizer**: See comments under LabCode 20.] |

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

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

**Folpet** Assigned value: 0.249 mg/kg, CV*: 34.5 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 13 (FN) | -3.8 | D | [**Note by the Organizer**: At first sight it is not obvious why you have not detected Folpet. Consider introducing QC measures that would highlight the risk of a false negative result. E.g. calibration level and recovery at or below the RL. Also please check the comments on LabID 57 as regards losses in non-frozen homogenates. |
| 76 | -3.5 | D, E | Degradation of Folpet to Phthalimide in the GC-injektor (and maybe during analysis), sum of Phthalimide expressed as Folpet and Folpet is correct (80% of preAV sum of both analytes)<br>[**Note by the Organizer**: As you have extracted using FA-QuEChERS, a degradation during the procedure is unlikely. A degradation during measurement should normally be addressed by proper calibration. By using the ILIS for Captan to quantify Folpet you surely introduced an error source. Also please check the comments on LabID 57 as regards losses in non-frozen homogenates. |
| 57 | -3.4 | D | [**Note by the Organizer**: A degradation of folpet was noticed by the organisers irrespective if the homogenates were stored in the refrigerator or on the lab bench. Ideally, keep your samples frozen untill analysis to minimize losses. If in your routine work sample homogenates are kept in a non-frozen state prior to analysis, consider minimizing the time interval until the start of the extraction. Alternatively, consider switching to cryogenic processing. |
| 90 | -2.7 | B | [**Note by the Organizer**: The reason for the underestimation is not obvious at first sight. Consider re-acidifying quickly after dSPE with PSA. Also read the comments on LabID 57 regarding degradation in homogenates. |
| 92 | -2.2 | M | Not enough amount of sample to make more repetition<br>[**Note by the Organizer**: See the comments made under LabCode 57. Also consider introducing a claibration approach that corrects for matrix effects] |
| 109 | -2.2 | | |
| 30 | -2.1 | D, E | Possible degradation in the extract after sample prepaparation. Folpet is analysed using a selective method on LC-MSMS. We have noticed during the validation that folpet and captan are unstabile in the LC-MSMS extract. Besides that, the sample was for 3 days in the refrigerator before starting the extraction for the determination of folpet with LC-MSMS. It is not known by the lab if folpet can break down in the refrigerator during storage.<br>[**Note by the Organizer**: See the comments made on your input on Captan] |
| 88 | 2.9 | J, E | [**Note by the Organizer**: Consider checking the stability of your stock and working solutions and adjust your storage conditions where needed. Note that Captan and Folpet are sensitive to high pH and to high temperatures. Acidification of standard solutions, e.g. with acetic acid, often helps to increase stability.] |
| 104 | 3.4 | A, M | The determination and calculations were performed following the method published by EURL-SRM (SRM-07/(V3.1) dated 06/04/2017).<br>At thee end of 2021 we commisioned a new GC-MSMS-System (TSQ9000).<br>Unfortunately the measurement method could not be transferred 1:1 from the previous system (TSQ) to the new system due to the change of the injection system from Thermo-PTV to Gerstel-KAS. The results were obtained by GC-SRM as well as GC-NCI using the SSL injector. During the period of EUPT-SRM 17, a validated method for the determination of captan, folpet, THPI and phthalimide was not yet available. Further investigations, method development and method validation must be done.<br>[**Note by the Organizer**: check the comments under LabCode 88.] |
| 112 | 3.4 | M | Sum of Folpet and Phthalimide calculated as Folpet is nearly the same as in the preAV; we have decided to transfer results, we had with the first analytical approach<br>[**Note by the Organizer**: check the comments under LabCode 88.] |
| 100 | 3.8 | E, F | No reason identified clearly after several re-analysis. The results are not repeatable but rather high. Amelioration with a LC analysis will be tested in the coming weeks. The utilisation of folpet D4 in another considered option for an amelioration.<br>[**Note by the Organizer**: The use of Folpet ILIS will surely improve any error sources during analysis. Check the comments under LabCode 88.] |

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

| **Folpet** Assigned value: 0.249 mg/kg, CV*: 34.5 % | | | |
|---|---|---|---|
| LabCode | z Score | Reason | Remarks/Details |
| 5 | 4.2 | C, E, M | We are in the process of identifying the exact cause of the problem. |
| 70 | 4.8 | J, M | J) The stock solution used for calibration and standard additions wa checked and a crystalline sediment was noticed. After dissolving the sediment, the folpet concentration corresponded in the solution of the concentration of a freshly prepared clear stock solution. The concentration of the standard used for the PT dropped by approx. 50% as a result of crystallization, which led to an overestimated result by a factor of 2.<br>M) Individual Error: Insufficient Visual inspection of the standard solution before use and obviously not the optimal solvent for the active substance<br>[**Note by the Organizer**: This text was translated into English by the organizers, check the comments under LabCode 88.] |
| 71 | 5.1 | F | [**Note by the Organizer**: check the comments under LabCode 88.] |
| 128 | 5.4 | | Despite the long exposition of the sample homogenate at room temperature, you have optained an overestimations of the folpet concentration. This indicates suggests the presence additional more error source(s) with opposite impact. Check the stability of your analytical standard. |
| 111 | 9.5 | B, E | [**Note by the Organizer**: check the comments under LabCode 88.] |
| 56 | 14.7 | A, E, D | New value : 0,380 mgkg<br>[**Note by the Organizer**: check the comments under LabCode 88.] |

| **Emamectin B1a** Assigned value: 0.0461 mg/kg, CV*: 21.5 % | | | |
|---|---|---|---|
| LabCode | z Score | Reason | Remarks/Details |
| 95 | 3.1 | J | testing of new stock solutions was performed a couple of days after reporting the results. The response of Emamectin benzoate in the standard solution used for the PT was at 43% which explaines the poor performance.<br>[**Note by the Organizer**: Your observations would explain the overestimation. Consider that losses of emamectin may occur in low concentrated standards in glas vials, which may lead to overestimated results. As the loss-rate is typically higher in lower concentrated standards, which are used to prepare the calibration solutions, such effects are often acompanied and recognaziable by high recovery rates. In your case, however, the recovery rate did not reveal any anomaly.] |
| 17 | 3.4 | J | [**Note by the Organizer**: See comments under LabID 95.] |
| 112 | 3.6 | J | [**Note by the Organizer**: See comments under LabID 95.] |
| 76 | 3.8 | I | used wrong concentration of the standard (0,1 µg/mL instead of 0,05 µg/mL) for the calculation<br>[**Note by the Organizer**: Please also consider the possibility of having experienced losses in the standard used. Your overestimated recoverey rate also poiunts towards this direction. See also comments under LabID 95.] |
| 92 | 8.1 | J | degradation of standard in glass bottle (better use plastic)<br>[**Note by the Organizer**: This is indeed as point to consider. See also comments under LabID 95.] |
| 25 | 15 | J | interlaboratory comparison of standard solution for bifenazat with higher deviation (concentration of own standardsolution too low)<br>[**Note by the Organizer**: Assuming that this comment refers to emamectin, the overestimation is explained. Please also consider the comments under labID 95.] |

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

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

| **Phthalimide** Assigned value*: 0.098 mg/kg, CV*: 51.5 % | | | |
|---|---|---|---|
| LabCode | z Score* | Reason | Remarks/Details |
| 20 | -3.6 | A | signal supression by matrix, repeated analysis gave result of 0.103 mg/kg |
| 44 | -3.6 | E | The laboratory employed the SRM-07 method to analyse captan sum and folpet sum. Phthalimide was detected but, following the indications of the SRM-07, it was necessary to subtract the amount of phthalimide generated in the injector through the "external calibration" calculation excell provided by EURL-SRM. The resulting concentration of phtamilde in the sample was calculated lower than limit of cuantification. The analytical technique employed was GC-MS/MS with EI, because of the laboratory does not have CI and the method with this ionization mode presents low sensitivity |
| 71 | -3.6 | F | |
| 92 | -3.6 | A | |
| 104 | -3.6 | A, M | The determination and calculations were performed following the method published by EURL-SRM (SRM-07/(V3.1) dated 06/04/2017).<br>At thee end of 2021 we commisioned a new GC-MSMS-System (TSQ9000).<br>Unfortunately the measurement method could not be transferred 1:1 from the previous system (TSQ) to the new system due to the change of the injection system from Thermo-PTV to Gerstel-KAS. The results were obtained by GC-SRM as well as GC-NCI using the SSL injector. During the period of EUPT-SRM 17, a validated method for the determination of captan, folpet, THPI and phthalimide was not yet available. Further investigations, method development and method validation must be done. |
| 42 | 2.1 | B | The value returned with acid extraction, with classical quecher extraction results in 0.11 mg/kg<br>[**Note by the Organizer**: Consider quantifying phthalimide by LC-MS/MS using SRM-49. If you would like to continue using GC-MS(/MS), consider using the approach described in SRM-07, which involves accurate determinantion of folpet (using folpet D4 as IS), and calculation of the share of detected phthalimide originating from folpet degradation in the injector (with the help of a suitable calibration). This share can then be deducted from the original phthalimid result (e.g. by SRM-07-ExtCal or SRM-07-StdAdd).] |
| 35 | 2.2 | G | Inappropriate internal standard was used for phthalimide. We used triphenyl phosphate instead of folpet-D4 for calculation of phthalimide concentration.<br>[**Note by the Organizer**: Folpet D4 would be a good choice for the quantification of folpet, but not for phthalimid. Considertesting the approach described in SRM-07, which involves accurate determinantion of folpet (using folpet D4 as IS), and calculation of the share of detected phthalimide originating from folpet degradation in the injector (with the help of a suitable calibration). This share can then be deducted from the original phthalimid result (e.g. by SRM-07-ExtCal or SRM-07-StdAdd). Alternatively, consider quantifying phthalimide by LC-MS/MS using SRM-49] |
| 85 | 2.3 | F | As already described in your mail from 10.06., the Folpet decomposition during GC injection was compensated through standard addition approach, unfortunately the PI result was not corrected accordingly for the generated PI during GC injection<br>[**Note by the Organizer**: See comments under LabID 42] |
| 16 | 2.5 | M | In the multimethod, phthalimide is analysed on GC-MS MS. Phthalimide is used as a marker for folpet. If we detect phthalimide in the sample, folpet is quantified on GC-ECD where there is no degradation to phthalimide. Phthalimide is not converted to folpet (sum). We do this to avoid reporting false positive results or overestimate the result for folpet. A high z-value shows that phthalimide works well as a marker so that we don´t report false negative results. Degradation to phthalimide occurs during analysis on GC-MSMS and phthalimide is formed during the production of processed foods.<br>[**Note by the Organizer**: If conversion to phthalimide is quantitative and the procedure validated spiking folpet, THPI and both, you may work this way to determine folpet (sum). However keep in mind, that you cannot report this results neither as "folpet" nor as "phthalimide". See also comments under LabID 42] |

* Assigned value based on the sum of incurred and spiking level and z scores were calculated for informative purpose only. Please refer to Section 4.2.1

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

| **Phthalimide** Assigned value*: 0.098 mg/kg, CV*: 51.5 % | | | |
|---|---|---|---|
| LabCode | z Score* | Reason | Remarks/Details |
| 11 | 2.9 | A | [**Note by the Organizer**: See comments under LabID 42] |
| 12 | 2.9 | M, B | The rate of the thermal decomposition of folpet (F) in phthalimide (PI) was tested by using blank extracts vials with 0.032 ng/µL of F in tomato matrix, quantified with PI standards.<br>The calculated concentration of PI produced by degradation (PId) was 0.0126 ng/µL (n=3; CV=7) so, if [F] is the concentration of folpet, we have<br>[PId] = 0.0126/0.032 * [F] = 0.393 [F] ng/µL<br>Being<br>[PI] = ""incurred"" PI<br>[PIt] = ""total"" PI reported<br>we can say that<br>[PIt] =[PI] + 0.393 [F]<br>Since our reported result for PI was 0.169 mg/kg and the report result for F was 0.141 mg/kg, we can write<br>0.169 = [PI] + 0.393 * 0.141<br>[PI] = 0.113<br><br>The z score would be (0.113- 0.098)/(0.25*0.098) = 0.61 (good)<br><br>Note:<br>- We observed that thermal degradation of folpet was worst by using ACN as solvent instead EtAcet.<br>- We didn't observed a folpet degradation correlation with the concentration.<br>- Folpet and phtalimid peak areas are very unstable; one way to reduce this instability is by diluting extracts (and calibration solutions).<br>The reason for our questionable perfomance was inexperience of laboratory since the normal practice is reporting folpet as a sum, according residue definition.<br>[**Note by the Organizer**: Thank you for your comments and insights. Indeed, degradation in ACN is typically stronger. Consider the use of Analyte Protectants (APs). Please note, that the decomposition rate of folpet (or captan) in the GC-injector is not constant. It may vary depending on the condition of the injector and the degree of protection provided by the co-injected matrix components. The degradation rate is typically greater at very low concentration. As concentration increases the decomposition-rate decreases ("auto-protection effect") . See also comments under LabID 42]. |
| 15 | 2.9 | M | Phthalimide was quantified by GC-MS/MS using an ILIS and C-spikes (addition to the test solution) with 3 concentrations. This led to a result of 0.170 mg/kg. Due to the better sensitivity on GC-MS/MS, this result was interpreted as the best.<br>However, the sample was also analysed for phthalimide by LC-MS/MS, no ILIS was used and quantification was done by A-spiking (addition to the sample material before extraction). This gave a result of 0.116 mg/kg.<br>LC-MS/MS is the method of choice for phthalimide. Due to the low sensitivity, the result obtained by GC-MS/MS was nevertheless reported.<br>[**Note by the Organizer**: Thank you for your comments and insights, which seem to be plaussible.]. |
| 38 | 2.9 | F | High spread of result in GC and a bad final decision. We considerer that the analyte losses are corrected when using matrix match analysis.<br>[**Note by the Organizer**: Matrix matching will address matrix effects for the parent (phthalimide) but it will not prevent an overestimation of phthalimide when using GC. For the latter consider the comments under LabID 42] |
| 105 | 2.9 | B | The reason for the overestimated PI result could be the GC technique used to determine this compound. We suppose that the overestimation of the PI determination may have been due to the thermal decay of folpet in the liner during the injection of the sample into the GC.<br>[**Note by the Organizer**: See comments under LabID 42] |
| 82 | 3.0 | B | PI results not submitted in rountine analysis because of possible non-PSM sources<br>[**Note by the Organizer**: There are indeed some non-pesticide-sources of phthalimide, such its formation furing the drying of food products. Phthalimide formation in the hot GC-injector has been reported in presence of N-containing compounds. However, the EURL-SRM has observed that APs will suppress this formation. See also comments under LabID 42]. |
| 36 | 3.6 | D, E | Degradation in GC injector<br>[**Note by the Organizer**: See comments under LabID 42] |
| 76 | 3.7 | B, E | Degradation of Folpet to Phthalimide in the GC-injektor (and maybe during analysis), sum of Phthalimide expressed as Folpet and Folpet is correct (93% of preAV sum of both analytes)<br>[**Note by the Organizer**: See comments under LabID 42] |
| 4 | 3.8 | E | signal interefered by matrix<br>[**Note by the Organizer**: See comments under LabID 42] |

* Assigned value based on the sum of incurred and spiking level and z scores were calculated for informative purpose only. Please refer to Section 4.2.1

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

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

**Phthalimide** Assigned value*: 0.098 mg/kg, CV*: 51.5 %

| LabCode | z Score* | Reason | Remarks/Details |
|---|---|---|---|
| 106 | 3.8 | A, M, B | The Phthalimide and Folpet results in the proficiency test were obtained on a GC-MSMS by using separate standards spiked with either Folpet or Phthalimide. This method did not account for degradation of parts of the Folpet to additional Phthalimide in the sample, since there was no Folpet in the Phthalimide standards.<br>An additional experiment performed now with several standards spiked with the same amount of Phthalimide, but different amounts of Folpet showed that the presence of Folpet causes an increase in the Phthalimide signal. After that, the SRM-17 test material was tested again for Phthalimide, using Phthalimide standards that all contained the same amount of Folpet as the test material. This resultet in a Phthalimide amount of 0,083. This result would lead to an acceptable z score (near -0,7). Folpet and Phthalimide are currently not in the spectrum of our laboratory, but currently efforts are on the way for adding these compunds to the spectrum. Should a sample contain both Folpet and Phthalimide, two additional steps will be needed for Quantification:<br>1) Quantification of Folpet using Standards spiked with Folpet<br>2) Quantification of Phthalimide using Standards spiked with Phthalimide and all containing the amount of Folpet determined in step 1)<br>[**Note by the Organizer**: The procedure you are proposing seems viable. You may also consider the procedures mentioned in the comments on LabCode 42. |
| 10 | 4.0 | B | [**Note by the Organizer**: See comments under LabID 42] |
| 111 | 4.6 | E, B, F | [**Note by the Organizer**: See comments under LabID 42] |
| 3rd-27 | 4.6 | E | [**Note by the Organizer**: See comments under LabID 42] |
| 3rd-83 | 5.8 | M | No issue found with the data. The combined z score for folpet and PHI (residue definition) would have been with ±2<br>[**Note by the Organizer**: Please take note of the comments under LabID 42, but also check for other possible sources leading to an overestimation of phthalimide] |
| 13 | 11.4 | D, E | Folpet degradation<br>[**Note by the Organizer**: Please take note of the comments under LabID 42, but also check for other possible sources leading to an overestimation of phthalimide] |
| 60 | 11.4 | H | Phthalimide was analysed two times during PT-round. In both measurements, the spiked blank's result was not repeatable, and perhaps the calculated recovery (78%) was thereforee not correct. The average result of Phthalimide without recovery correction is 0,186 that would be acceptable result.<br>[**Note by the Organizer**: Correcting for recovery based on a recovery factor of 78% would not lead to such an overestimation. Please take note of the comments under LabID 42, but also check for other possible sources leading to an overestimation of phthalimide] |
| 72 | 11.4 | J, E | We verify the standard solutions and re-analyse, on medium terms we want to implement LC/MS, at least for PI and THPI<br>Additional comments:<br>We mainly suspected that something was wrong with our PI standard. So we ordered new reference material from ""HPC Standards GmbH "", prepared new standard solutions and performed a complete re-analysis.<br>The new analysis gave us a phthalimide concentration of 0,11 mg/kg.<br><br>This result confirms that our suspicion (bad PI standard) was correct. (Note of the organizer: Thank you for this insight.) |
| 97 | 24.4 | A, C, H | [**Note by the Organizer**: Please take note of the comments under LabID 42, but also check for other possible sources leading to an overestimation of phthalimide] |

* Assigned value based on the sum of incurred and spiking level and z scores were calculated for informative purpose only. Please refer to Section 4.2.1

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

**Pymetrozine** Assigned value: 0.15 mg/kg, CV*: 28.2 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 49 | -3.5 | D | analyte was present in spiked sample (in all parallels) but nothing was seen in PT sample |
| 66 | -3.1 | B, D, H | "Problems caused by the Covid-19 pandemic, including acute lack of personnel and necessary consumables, significantly affected our ability to fully participate in this proficiency test right now. Single residue methods for the compounds in the PT scope were under validation. For this we reported some of the results from our multi residue analysis.<br><br>If the result had been corrected for recovery, the result would have been 0.089 mg/kg (inside the range of method MU 50 %). Because of the unusual circumstances at the time of this PT this procedure was not followed."<br>[**Note by the Organizer**: This indeed a typical recovery range achieved by standard citrate-buffered QuEChERS and would have reduced your bias. Please also check the QuEChERS variant SRM-32] |
| 125 | -2.1 | B | The recoveries are below 60% using Quechers EN (usually in the range 50-60%)<br>[**Note by the Organizer**: This indeed a typical recovery range achieved by standard citrate-buffered QuEChERS. Please also check the QuEChERS variant SRM-32] |
| 7 | 2.4 | H, M | Result corrected for low recovery in PT sample spiked at 0,1 mg/kg (46%). Recovery in cucumber in same sequence 69%. Average recovery in cucumber control spike at 0,1 mg/kg in 120 series is 64% (RSD% = 13), average recovery in tomato control spike at 0,1 mg/kg in 4 serie 63% (RSD%=7).<br>[**Note by the Organizer**: The recovery ranges you give are within the typical recovery range achieved by standard citrate-buffered QuEChERS. Please also check the QuEChERS variant SRM-32] |
| 101 | 2.9 | M | Standard addition was used, suspected problem with matrix effects on a new instrument. Investigation is on going |
| 127 | 6.9 | J | |

**THPI** Assigned value: 0.59 mg/kg, CV*: 23.2 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 26 | -3.1 | A | |
| 92 | -2.8 | A | |
| 112 | -2.3 | M | Sum of Captan and THPI calculated as Captan is nearly the same as in the preAV; we have decided to transfer results, we had with the first analytical approach |
| 120 | 2.4 | D, E | Problem with degradation of Captan to THPI in inlet injector during analysis, lab doesn't use isotopically labelled standards yet<br>[**Note by the Organizer**: Consider quantifying phthalimide by LC-MS/MS using SRM-49. If you would like to continue using GC-MS(/MS), consider using the approach described in SRM-07, which involves accurate determinantion of folpet (using folpet D4 as IS), and calculation of the share of detected phthalimide originating from folpet degradation in the injector (with the help of a suitable calibration). This share can then be deducted from the original phthalimid result (e.g. by SRM-07-ExtCal or SRM-07-StdAdd).] |

**Bifenazate (sum)** Assigned value: 0.296 mg/kg, CV*: 26 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 5 | 2.1 | A, I, M | We are in the process of identifying the exact cause of the problem. |
| 25 | 2.8 | J | interlaboratory comparison of standard solution for bifenazat without higher deviation |
| 3rd-27 | 2.8 | F | |
| 90 | 6.1 | A, B, M | We analyse this pesticide using estándar quechers method and sum bifenazata and bifenazate diazene<br>[**Note by the Organizer**: Consider transforming bifenazate diazene to bifenazate using ascorbic acid. Please also check the QuEChERS variant SRM-34] |
| 57 | 7.4 | B | |

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

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

**Chloridazon-desphenyl** Assigned value: 0.0616 mg/kg, CV*: 23.7 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 41 | -3.7 (FN) | F, E | [**Note by the Organizer**: Check whether chloridazon rarther than chloridazon-desphenyl was targeted] |
| 3 | -3.5 (FN) | A | Compound is not on our scope of analysis<br>[**Note by the Organizer**: Check whether chloridazon rarther than chloridazon-desphenyl was targeted] |
| 30 | -3.5 (FN) | L | Mistake by the scope selection. Compound are not included in our scope (not analysed) |
| 114 | -3.5 (FN) | A, E | Due to the actual changes of pesticide definition, this compund has just gained to our scope, so we do not have enough measuring experience with this compound. We also have ackowledged the problems with the chromatographic separation about this compund, but we have already improved our measuring methods, and we have already quite good results from that. |
| 45 | -3.3 (FN) | L | Assumption that chloridazon and chloridazon-desphenyl are the same component. |
| 99 | -3.3 (FN) | L | This pesticide was not accreditted in our scope, so it was decided not to submit any result. By mistake it was not selected "not analysed" for chloridazone desphenyl when submitting results. The obtained result for chloridazone desphenyl by our lab was 0.048 mg/kg, |
| 120 | -3.3 (FN) | L | Incorrect RL given in scope (0,01mg/kg), correct in our lab RL is 0,05mg/kg, detected value in lab higer than 0,01mg/kg and lower than 0,05mg/kg<br>[**Note by the Organizer**: Check whether chloridazon rarther than chloridazon-desphenyl was targeted] |
| 3rd-27 | 2.9 | E | |
| 52 | 39.8 | A, J | Chloridazon-desphenyl is a new standard, no experience exist, thereforee routinely Standard matching not possible. Standard indicates precipitation in high measure (was overlooked during EUPT), so that the analysis of EUPT lead to higher values (flatter slope of calibration curve). Chloridazon-desphenyl is now dissolved in MeOH, EUPT was replicated with Standard addition. This leads to a value of 0,0612 mg/kg, which should me correct.<br>[**Note by the Organizer**: Thank you very much for your insights about the poor solubility issues. |

**ETU** Assigned value*: 0.06290 mg/kg, CV*: 33.1 %

| LabCode | z Score* | Reason | Remarks/Details |
|---|---|---|---|
| 12 | -3.4 | D | There are two reasons for our false negative result:<br>i) Unexpected high signal supression observed with transition 103>44.1 (%SSE = 11%). This value is outside or normal tolerance.<br>ii) Signal interference in tomato matrix, observed with the transitions 103>86 (% SSE = 530 %) and 103>60 (%SSE = 33%) resulting ion ratios totally different to from those ion ratios observed using solvent.<br>[**Note by the Organizer**: Mass transitions involving the same parent mass normally experience similar matrix effects. You may have a matrix interference.] |
| 57 | -2.1 | J | |
| 45 | 2.5 | I | Spike was too low |
| 10 | 2.6 | G, H | |

* Assigned value statistically uncertain and the z scores were calculated for informative purpose only.
Please refer to Section 4.2.3

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

| **Formetanate-HCl** Assigned value: 0.873 mg/kg, CV*: 25.5 % | | | |
|---|---|---|---|
| LabCode | z Score | Reason | Remarks/Details |
| 4 | -4.0 | E | |
| 12 | -4.0 | G | Our formetanate is in a solution mix with other coumponds. When we made the PT our formetanate appears on RT=14.2min and there wasn't peak, at that time, in the PT sample.<br>When we have prepared a new standard of formetanate we observed a peak at 9.39 min and a similar peak in the PT sample.<br>Using the the peak at (RT=9.39) our result for EUPT_SMR17 would be 0.821 mg/Kg (z score ~0.2)." |
| 71 | -4.0 | E | Problems with capillary (e.g. poor sensitivity due to partial clogging) |
| 112 | -4.0 | F | Chromatographic problems and additionally a matrix peak in our spiking solution which seems to be Formetanate. |
| 113 | -4.0 | L | The reason was simple....I just missed to report that analyte. |
| 3rd-83 | -4.0 | M | Human errror wrong retention time was put into processing method for formetanate.<br>[**Note by the Organizer:** Check if the reason with the code K also applies] |
| 66 | -2.4 | H, I, B | If the result had been corrected for recovery and a factor of 1.165 (formetanate-HCl instead of formetanate) had been used for calculation, the result would have been 0.573 mg/kg (inside the range of method MU 50 %). Because of the unusual circumstances at the time of this PT this procedure was not followed.<br>Problems caused by the Covid-19 pandemic, including acute lack of personnel and necessary consumables, significantly affected our ability to fully participate in this proficiency test right now. Single residue methods for the compounds in the PT scope were under validation. For this we reported some of the results from our multi residue analysis. |
| 75 | -2.3 | B | |
| 54 | 2.2 | J | |
| 82 | 2.2 | E, F | |
| 79 | 2.6 | F, I | |
| 64 | 2.8 | J | |
| 85 | 3.5 | J | analytical standard was degraded, a standard addition with a fresh prepared solution quantified 1,18 mg/kg, which would be inside the acceptable +-2 z score range<br>[**Note by the Organizer**: Formetanate is known to be susceptiple to degradation in certain solvents as well as in certain extracts with light, water presence and high pH increasing degradation rates. Stability in ACN seems OK but solubility is limited. Acidification helps to increase shelf life. Make sure to improve QC measures to allow recognizing such a bias] |
| 101 | 3.5 | M | The sample was diluted with blankmatrix. Suspected problem with matrix effects on a new instrument. Investigation is on going |
| 61 | 4.2 | J | [**Note by the Organizer**: Check comments under LabCode 85.] |
| 70 | 4.2 | J, M | J) The formetanate standard solution was checked prior to use according to the internal QM system and approved for use. In the efforts to find the source of the overestimated PT result, in mid-April, it was recognized that the analyte had apparently degraded by 2/3, so the concentration was only about 33% of the original value. At the time of the PT (february), the degradation rate of the standard was probably lower, and this may explain why the reported results was overestimated by a factor of 2. M) The shelf life specified in the internal QM system for stock solutions is too long for formetanate, which has not been recognized so far.<br>[**Note by the Organizer**: this text was translated into English by the organizers][**Note by the Organizer**: Check comments under LabCode 85.] |
| 105 | 6.1 | J | The reason that influenced of this result was the degradation of the analytical standard in the mixture. A new mixture was prepared and the result was recalculated. The result obtained on the basis of the curve from the new mixture was 0.61 mg / kg.<br>[**Note by the Organizer**: Your comment is plausible. Check comments under LabCode 85.] |
| 90 | 6.2 | D | [**Note by the Organizer**: Check comments under LabCode 85.] |
| 18 | 6.6 | J, M | Intermediate standard kept in acidified Methanol has a response difference 100% than the standard kept in pure Methanol. This observation will be checked with new standard preparation.<br>[**Note by the Organizer**: Your comment is plausible. Check comments under LabCode 85.] |
| 41 | 9.2 | D, I | [**Note by the Organizer**: Check comments under LabCode 85.] |
| 88 | 10.4 | A, J | [**Note by the Organizer**: Check comments under LabCode 85.] |
| 48 | 23.5 | J | our follow-up examination 1st batch: 0.831 ppm; 2nd approach: 0.863 ppm (with fresh stock solution).<br>[**Note by the Organizer**: this text was translated into English by the organizers, Check comments under LabCode 85.] |
| 57 | 182.0 | J | |

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

**A:** Lack of experience; **B:** Analytical procedure was inappropriate (e. g., hydrolysis conditions too weak; recovery too low; sensitivity too poor, RL < AV); **C:** Analytical procedure was appropriate, but 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 / Misunderstanding of the target pesticide; **G:** Inappropriate / erroneous calibration approach (e. g. matrix effects not properly compensated); **H:** Result not properly corrected for recovery; **I:** Calculation error (e. g. use of wrong factor to express residue as required in PT or to address dilutions etc.); **J:** Erroneous analytical standard (e. g. due to degradation, wrong purity, wrong dilution); **K:** Deficient QC-measures that would have helped to recognize that method generates FNs, FPs or strongly biased results (e. g. no recovery test); **L:** Transcription- / documentation-/ communication-/ error; **M:** other reason.

**Maleic hydrazide** Assigned value: 0.544 mg/kg, CV*: 16.6 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 75 | 2.1 | C | |

**Oxymatrine** Assigned value: 0.198 mg/kg, CV*: 22.4 %

| LabCode | z Score | Reason | Remarks/Details |
|---|---|---|---|
| 47 | 2.2 | J | We have performed a compare of the quantification standard with a new standard of Oxymatrine. The recovery of the quantification standard was 75%.The corrected result is 0,231mg/kg Oxymatrine. |
| 25 | 2.6 | E | |

**2,4-DNOP (free phenol)** Assigned value*: 0.056 mg/kg, CV*: 45.1 %

| LabCode | Conc. [mg/kg] | Reason | Remarks/Details |
|---|---|---|---|
| 10 | FN | L | Only detection of Sum (Meptyldinocap) after alkaline hyrolysis. |
| 112 | FN | L | Its not in our scope. We submitted the "possibility to analyse" by mistake. |
| 117 | 0.122 | A | Not in the lab scope, first trial |
| 25 | 0.164 | J, E, F | |
| 38 | 0.174 | A | |

* Assigned value statistically uncertain, no z score was calculated, please refer also to Section 4.2.2

**Meptyldinocap** Assigned value*: 0.100 mg/kg, *CV**: 54.6 %

| LabCode | Conc. [mg/kg] | Reason | Remarks/Details |
|---|---|---|---|
| 10 | FN | L | Only detection of Sum (Meptyldinocap) after alkaline hyrolysis.<br>[**Note by the Organizer**: The webtool allowed separate reporting for "Meptyldinocap", "2,4-DNOP" and "Meptyldinocap (sum)".If you only analyzed for "Meptyldinocap (sum)", i.e. after hydrolysis you should have opted for "Analyzed" and "Detected" only for "Meptyldinocap (sum)". By reporting analyzed and detected for "Meptyldinocap" a concentration is expected. We might need to make this more clear in future PTs.] |
| 30 | FN | B, L | Meptyldinocap cannot be selective analysed as target but only the degradation product 2,4 DNOP. The sample contains Meptyldinocap and free 2,4 DNOP. If we detect meptyldinocap or 2,4-DNOP than we always do a additional hydrolysis of the extract to quantify to total sum of meptyldinocap. We have therefore decided to report as meptyldinocap sum as mentioned in the residue definition. It was not possible to mark meptyldinocap (selective) as not analysed.<br>[**Note by the Organizer**: Consider the comments under LabCode 10.] |

* Assigned value statistically uncertain, no z score was calculated, please refer also to Section 4.2.2

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

| **Meptyldinocap** Assigned value*: 0.100 mg/kg, *CV**: 54.6 % | | | |
|---|---|---|---|
| LabCode | Conc. [mg/kg] | Reason | Remarks/Details |
| 50 | FN | L | Results for Metyldinocap were poor and inconsitent. Therefore the messurement for Metyldinocap was skiped and the new methode for [Sum] was performed.<br>[**Note by the Organizer**: Consider the comments under LabCode 10.] |
| 45 | 0.156 | L | We cannot distinguish a difference between the sub-components meptyldinocap and 2,4-DNOP. In our method we measure the in-source fragment which is 2,4-DNOP. This means that methyldinocap is converted to its metabolite and we measure this as meptyldinocap sum. The value of 0,156 mg/kg is in fact the sum that we have found.<br>[**Note by the Organizer**: The webtool allowed separate reporting for Meptyldinocap, 2,4-DNOP and Meptyldinocap (sum).If you only analyzed for "Meptyldinocap (sum)" i.e. after hydrolysis you shouldn't have opted "Analyzed" and "Detected" for "Meptyldinocap (sum)" only. We might need to make this more clear in future PTs.] |
| 100 | 0.186 | M | Quechers analysis with no conversion. No reason identified for the moment after several re-analysis (problems with recovery too high but no correlation with the residues found in the sample), analytical standard will be tested soon (we are waiting for a new one for a control)<br>[**Note by the Organizer**: Be aware that meptyldinocap standards are sensitive to degradation. Acidification of meptyldinocap and stock and working solutions helps to minimize losses] |
| 71 | 0.213 | E | Problems with standard and capillary, too (the result given was pre-result analysed by old standard, but there was no possibility to quantify the sample again by a new one in time due to clogging of the capillary) |
| 42 | 0.28 | J, A | We suspected that our standard meptyl dinocap is partially degrading to DNOP, causing an higher value of meptyl dinocap<br>[**Note by the Organizer**: Consider the comments under LabCode 100.] |
| 40 | 0.329 | M | "As indicated in the preliminary report, many laboratories are in the same situation as our laboratory.<br>We did not analyse 2,4-DNOP, a degradation product, or assess the sum of these two parameters. The controls of the series are good. Parallel spiking gives 97% of recovery. No problem identified. Awaiting final report to conclude."<br>[**Note by the Organizer**: We haven't understood your comment, in any case, consider the comments under LabCode 100.] |
| 127 | 0.53 | A | [**Note by the Organizer**: Consider the comments under LabCode 100.] |
| 54 | 0.613 | J, A | new validated substance, little experience about this difficult substance<br>[**Note by the Organizer**: Consider the comments under LabCode 100.] |
| 13 | 0.7500 | G, E | [**Note by the Organizer**: Consider the comments under LabCode 100.] |
| 106 | 0.764 | A, M | "eptyldinocap is not part of the spectrum of active substances of our laboratory.<br>The attempt to quantify it in the proficiency test SRM-17 failed, and subsequent attempts to get a satisfactory result, using a different standard solution, have not found the source of the problem. We will not add Meptyldinocap to our spectrum unless we can find and fix the problem.<br>[**Note by the Organizer**: Consider the comments under LabCode 100.] |
| 120 | 1.274 | B | [**Note by the Organizer**: Consider the comments under LabCode 100.] |
| 57 | 1.91 | B | [**Note by the Organizer**: Consider the comments under LabCode 100.] |
| 117 | 3.23 | A | Not in the lab scope, first trial<br>[**Note by the Organizer**: Consider the comments under LabCode 100.] |
| 88 | 3.55 | J | [**Note by the Organizer**: Consider the comments under LabCode 100.] |
| * Assigned value statistically uncertain, no z score was calculated, please refer also to Section 4.2.2 | | | |

| **Meptyldinocap (sum)** Assigned value*: 0.169 mg/kg, CV*: 50.5 % | | | |
|---|---|---|---|
| LabCode | Conc. [mg/kg] | Reason | Remarks/Details |
| 11 | 0.082 | A | |
| 100 | 0.257 | A | Quechers analysis with no conversion, 2,4- DNOP first time tested with meptyldinocap |
| 50 | 0.332 | J, A | First time for the work with the new methode. |
| 38 | 0.385 | K | |
| 117 | 3.38 | A | Not in the lab scope, first trial |
| * Assigned value statistically uncertain, no z score was calculated, please refer also to Section 4.2.2 | | | |

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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).

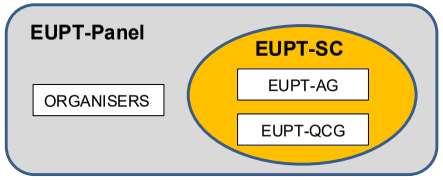
The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and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





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 $\sigma_{pt}$ 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 ($\text{FFP-}\sigma_{pt}$) 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.





– ***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 $\text{FFP-}\sigma_{pt}$ 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.



www.eurl-pesticides.eu

**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 17th EU Proficiency Test**
**on Pesticides requiring Single Residue Methods**
**EUPT – SRM17 (2022)**
(Update on 23 February 2022)

## 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, 2021 and 2022.
This PT is organized by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM). The EU Reference Laboratory for pesticides residues in Fruits and Vegetables (EURL-FV)) has collaborated in the production of PT Test Item. Both EURLs are accredited as providers of proficiency tests according to ISO 17043 (please see **EURL-SRM accreditation** see **EURL-FV accreditation**).

The EUPT-SRM17 deals with the analysis of SRM-pesticides in tomatoes. Participation is obligatory for all National Reference Laboratories for Single Residue Methods (NRL-SRMs), as well as for all official EU laboratories (OfLs) involved in the official analyses of pesticide residues in fruits and vegetables. 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 6 December and ended on 31 December 2021.

The most important documents related to this PT can be accessed via the **EUPT-SRM17-Website**.

## Test Item

The Test Item of this EUPT is "Tomatoes Homogenate".

Participants will receive one Test Item bottle containing ~350 g deeply frozen **tomatoes homogenate with incurred and spiked analytes** from the **Target Pesticides List**. The field-treated tomatoes were milled at ambient temperature, then spiked with additional pesticides and homogenized further first at ambient temperature and then in addition cryogenically





using dry ice. The final material was filled into the bottles in a snow-like state, so that the analytical portions can be easily withdrawn. Shipment will be done in frozen condition with the addition of dry ice into the parcels. NO Blank material will be sent to the participants for this PT.

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

## Target Analytes and MRRLs

The Test Item will contain several analytes from the mandatory and optional section of the **EUPT-SRM17 Target Pesticides List**. Laboratories should read the Target 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 identify false positive and false negative results and for the calculation of z-scores for false negatives. **Make sure to download the latest version of the EUPT-SRM17 Target Pesticides List before starting with analysis and reporting results**.

## Shipment of Test Item

**Dispatch of the Test Item is planned on 31 January 2022.**
Test Item will be packed into thermo-boxes together with dry ice 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 21 January) with **detailed contact Information** (e.g. mobile phone numbers of laboratory personnel) and all **necessary documents** to be stuck/attached on the package to ensure smooth customs clearance. Such documents may be e.g. a permission for importing organic material for scientific purposes (lab analysis) or an **instruction in local language** to explain the need to keep the package in a freezer in case of delay during shipment or clearance at customs.

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

**The Test Item was prepared and shipped in way allowing analytical portions to be taken directly without additional milling or thawing. Should you notice the formation of ice-crystals or signs that the material was thawn during transport,**







Appendix 9. Specific Protocol of EUPT-SRM17

**it is recommended mixing the material thoroughly in its entirety before analytical portions are taken. While mixing, keep temperatures as low as possible to avoid thawing and the potential loss of unstable pesticides.**

**Participating laboratories are recommended using their routine standard operating procedures for extraction, clean-up and analytical measurement as well as their own reference standards for identification and quantification purposes.** Laboratories may also employ methods not yet implemented routinely, for example, if they are in the test phase of implementing them. In this case, the limited experience and the non-inclusion of the analytes in the routine scope should be indicated in the **EUPT-SRM17 result submission webtool**.

The homogeneity tests will be conducted using 10 g or 20 g portions. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can be guaranteed only for sample portions roughly equal to or bigger than the portion size used in the homogeneity test.

## Results submission webtool

Sample receipt acknowledgement, analytical results and method information are to be submitted via the **EUPT-SRM17 Result Submission Webtool:**

- **Sample receipt acknowledgement: From 1 Feb. 2022 onwards.**
- **Reporting of Analytical results and method information: 1 Feb. – 8 Mar. 23:30 h CET.**
- **Deadline for result submission is 8 March, 11.30 pm (CET), 2022.**
- **Reporting of additional information on methods used for tentatively false negative results: 9 – 16 March, 2022.**

A guideline for the new **EUPT-SRM17 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-SRM17 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 by DTU prior to opening of the webtool for the EUPT-SRM17.**

**The lab's unique lab code for the EUPT-SRM17 will be provided to the participants following the first access to EUPT-SRM17 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-SRM17 Result Submission Webtool** the date of receipt, the condition of the package, the condition of the test material at arrival, whether the material is accepted or not, and any other comments concerning the test material. This task should be finalized by **4 February 11:00 am CET**. If a laboratory does not respond by this deadline, the Organizers will assume that the Test Item has been received and accepted. **In case of problems with the sample receipt or condition**, please contact the organizers **additionally via e-mail ASAP** (**eurl-srm@cvuas.bwl.de**) to ensure that corrective actions are taken as early as possible. Please note that completing the sample receipt form is a pre-requisite for accessing the results submission areas. However, you can still access sample receipt form and edit it later.





**Participants are encouraged to follow the whereabouts of their parcels using the tracking code of the shipping company that they will receive via e-mail and intervene at the shipping company, the customs or the organizers if they notice any delays. Any participants not having received the Test Item by the Fri. 4 February, at 10:00 am CET, must inform the Organizer via e-mail (EURL-SRM@cvuas.bwl.de) immediately but not later than Fri. 4 February 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-SRM17 Result Submission Webtool**.
**All results must be reported on this website by 8 March, 11.30 pm (CET), 2022.** The pages for the "scope, detected and results" will not be accessible after this deadline, and no results submitted afterwards will be accepted.

Before entering the results, please study the **EUPT-SRM17 Target Pesticides List** carefully. Please note, that the residue definitions applying to the EUPT may appear in a shortened form on the result submission website.

If a lab routinely subcontracts analyses of one or more compounds to another lab, this subcontracted lab may (and is even encouraged to) also take part in the EUPT exercise.

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

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

- "**Concentration in mg/kg":** the numerical pesticide concentrations that would be reported in routine work.
  If a pesticide was not detected or if it was detected but the quantitative result is below the RL (Reporting Limit) of the laboratory or the MRRL, no result should be reported. In accordance with 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 on the target pesticide list is defined as the sum of two or more components, a result for this "summed target analyte" should only be reported if
  a) the method used covers the entire residue definition of this "summed target analyte", e.g. if the method involves a chemical conversion to one component, or
  b) if all individual components entailed in this residue definition were targeted.

  In the latter case, the concentrations of the individual components of the "summed target analyte" should be added-up and expressed as stated in the residue definition on the target pesticide list. If at least one of the components within the "summed target analyte" was not analysed, this "summed target analyte" should be marked as "not analysed". In case one of the components within the complex residue definition was not encountered at a 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 or, if these are not available, in "Comments" field of the sum analyte. 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-SRM17 Result Submission Webtool** the participating laboratories have to provide information on the analytical method(s) applied for the analysis of the target analytes detected in the Test Item.

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

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

**For detailed information on how to fill-in the columns on the "Edit methods" page, please refer to the Guideline for Results Submission that will be distributed to all participants in due time. A link to this guideline can also be found in the info-box on the Result Submission webtool**. Please also read the **mouse-over messages** popping-up when your mouse cursor meets a field name in the table header for a few seconds.

### - Submission of results

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

**IMPORTANT:**
**Following "Final Submission", you will NOT be able to change your data anymore.**
**Without "Final Submission" your results and method information will not be included in the evaluation!**

### - Additional Information

After the results submission deadline, **if a laboratory has obtained a tentatively false negative result**, it will be asked to enter the method information for this analyte, within 7 working days.





## 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. For the establishment of the assigned values only results submitted by OfLs from EU and EFTA countries are taken into account.

## Subcontracting

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

a) Generation of login credentials
b) Programming and administration of EUPT-SRM17 result submission website

## Follow-up actions

After the distribution of the EUPT-SRM17 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-SRM17-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–SRM17 can be downloaded from **EUPT-SRM17 Website** or **EURL-Document Repository (CIRCA-BC)**.
For any questions, please contact the organizers **EURL-SRM@cvuas.bwl.de**

**IMPORTANT**:
Please check the **EUPT-SRM17 Website** before starting with the analysis in order to **make sure, that you have the latest version of all documents available**. In case of major changes, the participants will be informed via e-mail.

## Participation fees and payment details

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

- OfLs (including NRLs) from EU countries, EU-candidate countries and EFTA countries: 250 €
- Labs based in third countries: 400 €

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

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

**IMPORTANT:**

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

**The EURL-SRM will not complete any special form required by the participating laboratories for their financial department or payment office and does not agree to give any personal or private data for this purpose. If completion of such forms is prerequisite for payment 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 correction, we will return it with signature and stamp.**

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

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

**If no payment or no proof of payment is received and no explanation is given to the Organizers, the Organizers reserve the right not to issue the participation certificate for the concerned laboratory, to exclude its results and its name from the Final EUPT-Report, and to refuse its participation in future EUPT-SRMs.**

**Bank Details:**

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





**Please note:**

- **Please do not make any remittance before you receive the invoice with the Payee Identification Text.**
- **EURL-AO 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-SRM17 and EUPT-AO17), 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-SRM17

(please see https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EUPT-SRM17_Calendar.pdf)

## Target Pesticides List of EUPT-SRM17

(please see http://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EUPT-SRM17_TargetPesticideList.pdf)

## Contact information

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

e-mail: EURL-SRM@cvuas.bwl.de

**Organizing Group at the EURL-SRM (Stuttgart)**

| | |
|---|---|
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| Giovanna Cerchia | phone: +49 3426 1120 |

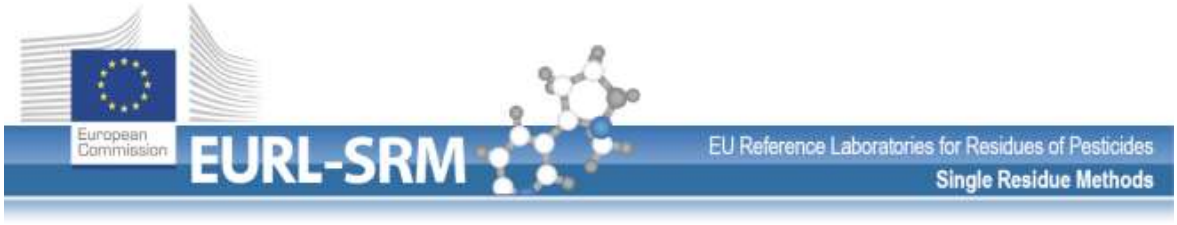
**Quality Control Group**

| | |
|---|---|
| Paula Medina | European Food Safety Authority (EFSA) |
| Antonio Valverde | Professor at 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 |
| Magnus Jezussek | LGL; Erlangen, Germany |
| André de Kok | Formerly working at NVWA, Wageningen, The Netherlands |
| Björn Hardebusch | Head of EURL AO, CVUA Freiburg, Freiburg, Germany |
| Ralf Lippold | Head of CVUA Freiburg, Germany |
| Hermann Unterluggauer | AGES; Institute for Food Safety Innsbruck, Austria. |
| Hans Mol | Wageningen Food Safety Research, Wageningen, The Netherlands |
| Finbarr O'Regan | Pesticide Control Laboratory, Celbridge Co. Kildare Ireland |
| Patrizia Pelosi | Istituto Superiore di Sanità, Rome, Italy |
| Tuija Pihlström | National Food Agency, Uppsala, Sweden |
| Mette Erecius Poulsen | Head of EURL-CF, DTU National Food Institute, Lyngby, Denmark |
| Radim Štěpán | Czech Agriculture and Food Inspection Authority, Prague, Czech Republic |

## Appendix 10 Calendar and Target Pesticides List of EUPT-SRM17




# CALENDAR for the EUPT – SRM17

# Tomato Homogenate

(Update on 23/02/2022)

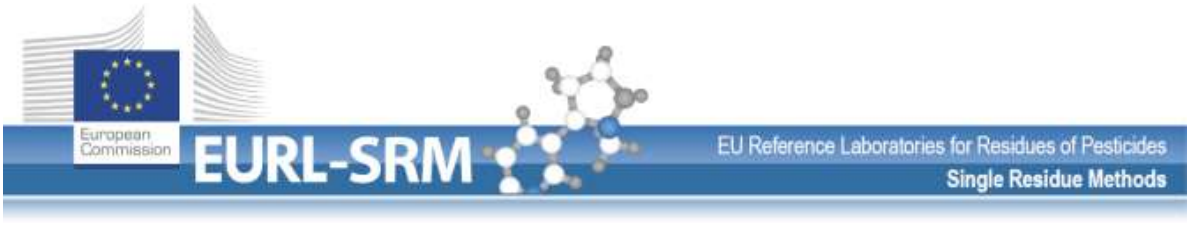
| Activity | Dates |
|---|---|
| **Announcement of the EUPT-SRM17**<br>opening of the **EUPT-SRM17 Website** with links to all relevant documents | 05 Nov. 2021 |
| **Registration Period for EUPT-SRM17**<br>via "**EUPT-Registration Website**"<br>**Labs classified as "OBLIGED" to participate in the EUPT-SRM17 MUST enter the EUPT-Registration Website and either register OR give explanations for non-participation** | 06 – 31 Dec. 2021, 23:30 h CET* |
| **Dispatch of EUPT-SRM17-Specific Protocol** | by 17 Jan. 2022 |
| **Shipment of EUPT-SRM17 Test Item** | 31 Jan. 2022 |
| **Confirming Sample Receipt and Acceptance**<br>via "**EUPT-SRM17 Result Submission Webtool**" | From 1 Feb. 2022 onwards |
| **Submission of Results (Pesticide scope, Results, Method Info)**<br>via "**EUPT-SRM17 Result Submission Webtool**" | 1 Feb. – **8** Mar. 23:30 h CET |
| **Submission of Additional/Missing Information**<br>e.g. Method info on tentatively false negative results<br>via "**EUPT-SRM17 Result Submission Webtool**" | **9** Mar. – **17** Mar. 2022 |
| **Dispatch of Preliminary Report**<br>containing results as well as preliminary assigned values and z-scores only | Within 3 weeks after the submission deadline |
| **Collection of reasons for underperformance and missing information on methods** | Apr. 2022 |
| **Dispatch of Final Report** | Dec. 2022 |

* Please make sure to register for the EUPT by the deadline 31 Dec 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 changes importantly affecting the participants, the participants will be informed via e-mail. However, please still check periodically our website for possible updates in case the email does not get through to you.
Contact: **eurl-srm@cvuas.bwl.de**

**The EUPT-SRM Team**

**Appendix 10 (cont.) Calendar and Target Pesticides List of EUPT-SRM17**




# TARGET PESTICIDE LIST

**for the EUPT–SRM17 (2022), Tomato Homogenate**

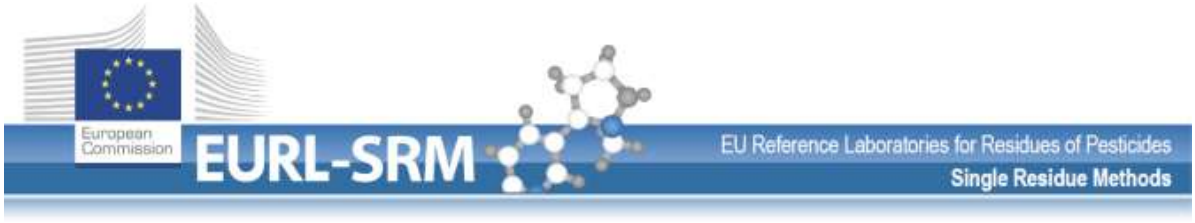
(update released on 28.01.2022)

## I: Analytes are grouped into Mandatory and Optional*

(1 of 2)

| MANDATORY ANALYTES | | | |
|---|---|---|---|
| **Analytes Name** | **Residue definition for the PT and additional remarks** | **MACP/WD** | **MRRL (mg/kg)** |
| **Avermectin B1a** | Main component of abamectin; expressed as avermectin B1a | **MACP** | 0.01 |
| **Captan** | | **MACP** | 0.02 |
| **THPI** | Tetrahydrophthalimide (degradant of captan), expressed as THPI | **MACP** | 0.01 |
| **Chlormequat (chloride)** | Expressed as chlormequat **chloride salt** | **MACP** | 0.01 |
| **Chlorothalonil** | | **MACP** | 0.01 |
| **Cyromazine** | | **MACP** | 0.02 |
| **Dithianon** | | **MACP** | 0.02 |
| **Dodine** | Expressed as dodine (free base) | **MACP** | 0.01 |
| **Dithiocarbamates** | Determined and expressed as carbon disulphide ($CS_2$) | **MACP** | 0.02 |
| **Emamectin B1a** | Main component of emamectin; expressed as emamectin B1a (free base) | **MACP** | 0.01 |
| **Fenbutatin oxide** | | **MACP** | 0.01 |
| **Folpet** | | **MACP** | 0.02 |
| **Phthalimide** | Degradant of Folpet, expressed as phthalimide | **MACP** | 0.01 |
| **Mepiquat (chloride)** | Expressed as mepiquat **chloride salt** | **MACP** | 0.01 |
| removed **~~Propamocarb~~** | ~~Expressed as propamocarb (free base)~~ | ~~**MACP**~~ | ~~0.01~~ |
| **Pymetrozine** | | **MACP** | 0.02 |
| **TFNA** | Metabolite of flonicamid, expressed as TFNA (free acid) | **MACP** | 0.01 |
| **TFNG** | Metabolite of flonicamid, expressed as TFNG (free acid) | **MACP** | 0.01 |

## Appendix 10 (cont.) Calendar and Target Pesticides List of EUPT-SRM17




### I: Analytes are grouped into Mandatory and Optional* (Cont.)

(2 of 2)

| OPTIONAL ANALYTES | | | |
|---|---|---|---|
| **Analytes Name** | **Residue definition for the PT and additional remarks** | **MACP/WD** | **MRRL (mg/kg)** |
| **BAC-C12 (chloride)** | Benzyldimethyldecylammonium chloride, expressed as **chloride salt** | **WD** | 0.02 |
| **Bifenazate (sum)** | Sum of bifenazate and bifenazate-diazene (expressed as bifenazate) | **WD** | 0.03 |
| **Chloridazon-desphenyl** | Expressed as chloridazon-desphenyl | **WD** | 0.02 |
| **DDAC-C10 (chloride)** | Didecyldimethylammonium chloride, expressed as **chloride salt** | **WD** | 0.01 |
| **Diquat** | Expressed as diquat **dication** | **WD** | 0.03 |
| **ETU** | Ethylene thiourea, degradant of ethylene-bis-dithiocarbamates | **None** | 0.01 |
| **Formetanate-HCl** | Expressed as formetanate **hydrochloride salt** | **MACP** | 0.01 |
| **Maleic hydrazide** | Expressed as maleic-hydrazide (free acid) | **MACP**** | 0.05 |
| **Matrine** | Expressed as matrine (free base) | **WD** | 0.02 |
| **Meptyldinocap** | | **WD** | 0.02 |
| **2,4-DNOP** | Metabolite of meptyldinocap, expressed as **2,4-DNOP (free phenol)** | **WD** | 0.01 |
| **Meptyldinocap (sum)** | Sum of meptyldinocap and 2,4-DNOP following chemical conversion to 2,4-DNOP (expressed as **meptyldinocap**) | **WD** | 0.01 |
| **Nicotine** | Expressed as nicotine (free base) | **WD** | 0.01 |
| **Oxymatrine** | Expressed as oxymatrine (free base) | **WD (2021)** | 0.01 |
| **Paraquat** | Expressed as paraquat **dication** | **WD** | 0.01 |
| **PTU** | N,N'-(1,2-propylene)thiourea, degradant of propylene-bis-dithiocarbamates | **None** | 0.01 |
| **Trimesium cation** | Expressed as trimesium **cation** | **WD** | 0.01 |

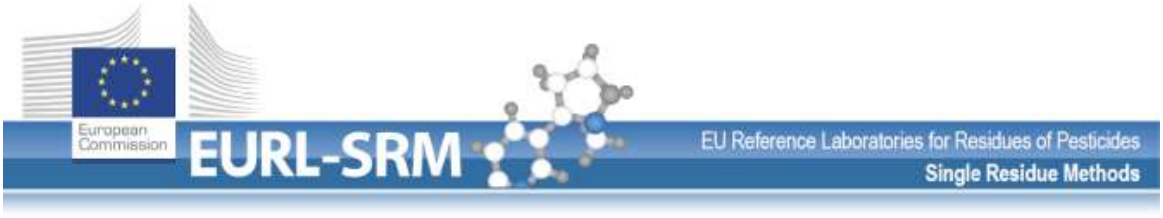
Typo Corrected!

**MACP-Reg.:** REGULATION (EU) 2020/585 of 27 April 2020
**WD:** Working document on pesticides to be considered for inclusion in the national control programs to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin; SANCO/12745/2013; 23–24 November 2020 rev. 12(2)
*** Only mandatory (=compulsory) analytes will be considered in the scope-based classification, optional (=voluntary) analytes not. Please also refer to the EUPT General Protocol.**
** inclusion of MH in the MACP was decided at technical level in 2021

**For alphabetical sorting in the same order as in the Webtool: See Pages 3 and 4.**


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-SRM17




# TARGET PESTICIDE LIST

### for the EUPT–SRM17 (2022), Tomato Homogenate

(update released on 28.01.2022)

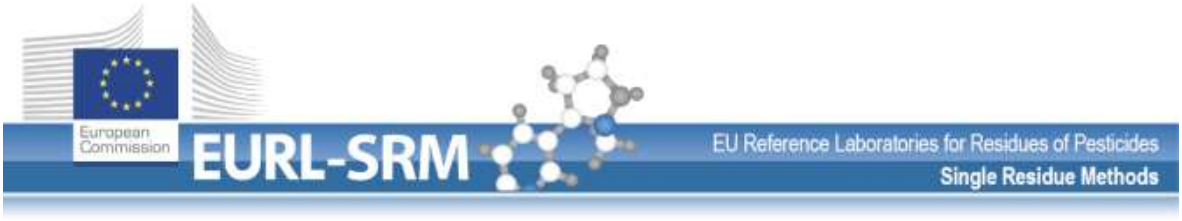
### II: Sorted alphabetically in the same order as in the Webtool

M: Mandatory*; O: Optional (=Voluntary)

(1 of 2)

| | Analytes Name | Residue definition for the PT and additional remarks | MACP/WD | MRRL (mg/kg) |
|---|---|---|---|---|
| O | **2,4-DNOP** | Metabolite of meptyldinocap, expressed as **2,4-DNOP (free phenol)** | **WD** | 0.01 |
| M | **Avermectin B1a** | Main component of abamectin; expressed as avermectin B1a | **MACP** | 0.01 |
| O | **BAC-C12 (chloride)** | Benzyldimethyldecylammonium chloride, expressed as **chloride salt** | **WD** | 0.02 |
| O | **Bifenazate (sum)** | Sum of bifenazate and bifenazate-diazene (expressed as bifenazate) | **WD** | 0.03 |
| M | **Captan** | | **MACP** | 0.02 |
| O | **Chloridazon-desphenyl** | Expressed as chloridazon-desphenyl | **WD** | 0.02 |
| M | **Chlormequat (chloride)** | Expressed as chlormequat **chloride salt** | **MACP** | 0.01 |
| M | **Chlorothalonil** | | **MACP** | 0.01 |
| M | **Cyromazine** | | **MACP** | 0.02 |
| O | **DDAC-C10 (chloride)** | Didecyldimethylammonium chloride, expressed as **chloride salt** | **WD** | 0.01 |
| O | **Diquat** | Expressed as diquat **dication** | **WD** | 0.03 |
| M | **Dithianon** | | **MACP** | 0.02 |
| M | **Dithiocarbamates** | Determined and expressed as carbon disulphide ($CS_2$) | **MACP** | 0.02 |
| M | **Dodine** | Expressed as dodine (free base) | **MACP** | 0.01 |
| O | **ETU** | Ethylene thiourea, degradant of ethylene-bis-dithiocarbamates | **None** | 0.01 |
| M | **Emamectin B1a** | Main component of emamectin; expressed as emamectin B1a (free base) | **MACP** | 0.01 |
| M | **Fenbutatin oxide** | | **MACP** | 0.01 |
| M | **Folpet** | | **MACP** | 0.02 |
| O | **Formetanate - HCl** | Expressed as formetanate **hydrochloride salt** | **MACP** | 0.01 |

## Appendix 10 (cont.) Calendar and Target Pesticides List of EUPT-SRM17




## II: Sorted alphabetically in the same order as in the Webtool (Cont.)

M: Mandatory*; O: Optional (=Voluntary)



| | Analytes Name | Residue definition for the PT and additional remarks | MACP/WD | MRRL (mg/kg) |
|---|---|---|---|---|
| O | **Maleic hydrazide** | Expressed as maleic-hydrazide (free acid) | **MACP**** | 0.05 |
| O | **Matrine** | Expressed as matrine (free base) | **WD** | 0.02 |
| M | **Mepiquat (chloride)** | Expressed as mepiquat **chloride salt** | **MACP** | 0.01 |
| O | **Meptyldinocap** | | **WD** | 0.02 |
| O | **Meptyldinocap (sum)** | Sum of meptyldinocap and 2,4-DNOP following chemical conversion to 2,4-DNOP (expressed as **meptyldinocap**) | **WD** | 0.01 |
| O | **Nicotine** | Expressed as nicotine (free base) | **WD** | 0.01 |
| O | **Oxymatrine** | Expressed as oxymatrine (free base) | **WD (2021)** | 0.01 |
| O | **PTU** | N,N'-(1,2-propylene)thiourea, degradant of propylene-bis-dithio-carbamates | **None** | 0.01 |
| O | **Paraquat** | Expressed as paraquat **dication** | **WD** | 0.01 |
| M | **Phthalimide** | Degradant of Folpet, expressed as phthalimide | **MACP** | 0.01 |
| M | **Pymetrozine** | | **MACP** | 0.02 |
| M | **TFNA** | Metabolite of flonicamid, expressed as TFNA (free acid) | **MACP** | 0.01 |
| M | **TFNG** | Metabolite of flonicamid, expressed as TFNG (free acid) | **MACP** | 0.01 |
| M | **THPI** | Tetrahydrophthalimide (degradant of captan), expressed as THPI | **MACP** | 0.01 |
| O | **Trimesium cation** | Expressed as trimesium **cation** | **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 programs 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 (=compulsory) analytes will be considered in the scope-based classification, optional (=voluntary) analytes not. Please also refer to the EUPT General Protocol.**
** inclusion of MH in the MACP was decided at technical level in 2021

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

**The EUPT-SRM17 Organising Team**

# Appendix 11 Announcement and Call for Registration for the EUPT-SRM17

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

## ANNOUNCEMENT/INVITATION

### EUPT – SRM17

### (Matrix: Tomato Homogenate)

(update on: 23/02/2022)

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-SRM17). This exercise is organized by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM) in cooperation with EU Reference Laboratory for pesticides residues in Fruits and Vegetables (EURL-FV).

**The EUPT-SRM17 is scheduled to run from 31 Jan. till 8 March, 2022.**

All relevant documentation is linked within the **EUPT-SRM17-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 deeply frozen **Tomato Homogenate.** 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 250 and 300 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 (at 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-SRM17-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 working days after the final EUPT deadline 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 **31 January 2022**.
**If any laboratory will be on holiday in the week of the shipment, please inform the organizer by 15 January in order to arrange an alternative shipment.**

Participants must check the integrity and condition of the materials upon receipt and requested to report **within 48 h** if they accept the materials or not. For this, please use the "**EUPT-SRM17 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-SRM17 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 **fruits and vegetables** 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 import controls of fruits and vegetables** listed under Reg. (EU) 1793/2019[1].

Based on the data stored in the Lab-Network Database about the commodity scope and the status of each lab, all official laboratories were categorized as "**obliged" or "not obliged"** to take part in this PT. This information can also be found on the EUPT-Registration page. In case an erroneous classification is noticed this shall 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;
- NRLs and OfLs from **EU-candidate countries** and **EFTA countries**;
- **laboratories analysing official organic samples** within the frame of Reg. 889/2008/EC[2];
- Laboratories from **Third Countries** (countries outside EU) as long as they are **involved in controls of products destined for export to the EU**.

From the latter two groups of labs may be requested to **provide a proof of their function** (e.g. scan copy of a document stating official appointment)

## REGISTRATION

The registration for the EUPT-SRM17 will run through the **EUPT-Registration Website** that is connected with the **EURL-DataPool**. To register for the EUPT-SRM17, please login to the **EUPT-Registration Website** using your **EURL-DataPool login credentials**. If you are not yet registered in the EURL-DataPool, you have to register into the EURL-DataPool first. If you have lost your EURL-DataPool login credentials, please use the "**forgot password" feature**.

The registration period is open from 06 to 31 December, 2021. An instruction on EUPTs registration can be found on the **EUPT-Registration Website** and on the **EUPT-SRM17-Website**.

For more information on how to register, you may consult 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.**

[2] Internally classified as „889-labs"





## IMPORTANT DATES

- **The EUPT-SRM16 registration form within the "EUPT-Registration Website" will be accessible from 06 – 31 December, 2021.**
- The **shipment** of the Test Items is planned on **31 January 2022.**
- **Results and method information** should be submitted by **08 March 2022 at 23:30 h (11:30 p.m.) CET** on the "**EUPT-SRM17 Result Submission website**".

## PARTICIPATION FEE and PAYMENT

A general fee of **250 €** 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 **400 €**.

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 in charge of the payment. **Details on payment will be given in the invoices**.

**Additional costs may apply 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-SRM17 will be uploaded onto the **CIRCA-BC platform** and linked to the **EUPT-SRM17-Website.**

The schedule for all activities and deadlines within this PT can be found in the **EUPT-SRM17 Calendar**.

The pesticides potentially present in the Test Item can be found in the **EUPT-SRM17 Target Pesticides List.**

The **EUPT-SRM17 Specific Protocol** will be published by 17 January, 2022. This should be read carefully. Please also refer to the valid version of the **General EUPT Protocol**, which entails the general evaluation rules of the EUPTs.

## GENERAL INFORMATION, CONFIDENTIALITY, DISCLAIMER

The EUPT-SRM17 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-SRM17 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. **However, please still check periodically the EUPT-SRM17-Website for possible updates** in case the email does not get through to you.
- By registering for this EUPT, the laboratories accept all above conditions and provisions.

## SUPPORT AND CONTACT INFORMATION

The EUPT-SRM17 organizing Team is always at your disposal to answer any questions and give you technical support. For any further questions about the EUPT-SRM17, please mail to eurl-srm@cvuas.bwl.de.

**EURL-SRM**
c/o Chemisches und Veterinäruntersuchungsamt Stuttgart
Schaflandstrasse 3/2
DE-70736 Fellbach
E-Mail: eurl-srm@cvuas.bwl.de

**EURL-SRM Organizing Team**

Michelangelo Anastassiades, phone: +49-711-3426-1124; Pat Schreiter, ext. -1029; Ann-Kathrin Wachtler, ext. -1151; Hubert Zipper, ext. -1141; Anja Barth, ext. -1125; Giovanna Cerchia, ext. -1120

## 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 |
| Magnus Jezussek | LGL; Erlangen, Germany |
| André de Kok | Formerly working at NVWA, Wageningen, The Netherlands |





| | |
|---|---|
| Björn Hardebusch | Head of EURL AO, CVUA Freiburg, Freiburg, Germany |
| Ralf Lippold | Head of CVUA Freiburg, Germany |
| Hermann Unterluggauer | AGES; Institute for Food Safety Innsbruck, Austria. |
| Hans Mol | Wageningen Food Safety Research, Wageningen, The Netherlands |
| Finbarr O'Regan | Pesticide Control Laboratory, Celbridge Co. Kildare Ireland |
| Tuija Pihlström | National Food Agency, Uppsala, Sweden |
| Patrizia Pelosi | Istituto Superiore di Sanità, Rome, Italy |
| Mette Erecius Poulsen | Head of EURL-CF, DTU National Food Institute, Lyngby, Denmark |
| Radim Štěpán | Czech Agriculture and Food Inspection Authority, Prague, Czech Republic |

Best regards,

The EUPT-SRM17 Organising Team

**Von:** Schreiter, Pat (CVUA-S) <Pat.Schreiter@cvuas.bwl.de> im Auftrag von EURL-SRM Pesticides (CVUA-S) <EURL-SRM@cvuas.bwl.de>
**Gesendet:** Sonntag, 5. Dezember 2021 23:15
**An:** EURL-SRM Pesticides (CVUA-S)
**Betreff:** Call for Registration (EUPT-SRM17) | EU/EFTA-OfLs

Dear Colleagues from EU- and EFTA-OfLs,

The EUPT-SRM17 using tomato as matrix is now open for registration.
**Please read this email carefully, before you register.**

**UPDATED TPL : Following a survey among the OfLs, we have updated Target Pesticides List for the EUPT-SRM17 with major changes.** You can access it on the EUPT-SRM17 Website.
Please check, if your laboratory can analyse at least one of the target pesticides, before you register for the participation in the EUPT-SRM17. Once your laboratory registered for participation and received the test material, the total participation fee must be paid, even if you later decide to withdraw your participation (e.g. because none of the pesticides is within your scope or none of the pesticides you have targeted is present in the sample).

**AMOUNT OF TEST MATERIAL : One unit of the test material for the EUPT-SRM17 contains ~350 g tomato homogenate.**
Erratum in the Announcement: Results Submission deadline is 1 March, 2022.

**NEW - SIMULTANEOUS REGISTRATION: From today onwards your laboratory can register for the EUPTs on pesticides residues to be organized by the 4 EURLs (SRM, FV, CF, AO) in 2022.**

- **Note**: the closing deadlines for registration differ from EUPT to EUPT.
- **For the EUPT-SRM17 the deadline is 31 December 2021, 11.30 pm (CET).**
- **To register please access the registration page: www.eupt-registration.eu and login using your EURL DataPool login credentials.** Instructions on how to complete the registration form can be found on the registration page and on the EUPT-SRM17 Website.

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

**Obliged laboratories (in case of EUPT-SRM17: NRLs-SRM and OfLs analyzing fruits and vegetables)**

Labs classified as **obliged** to participate in a PT **need to enter the registration form**.
**Obliged labs that do not intend to participate**, still have to access the registration page and choose "No" under "I want to REGISTER my lab for this EUPT" and **provide an explanation for non-participation** (requirement by DG-SANTE).
If your lab is classified as **non-obliged** and you are not going to participate in this PT, you don't have to do anything.
If you think that the EUPT-SRM17 classification of your laboratory is erroneous, please contact your NRL and the eurl-srm@cvuas.bwl.de.

**Special delivery Information for OfLs in Croatia:**
The test material will be sent with dry ice and by DHL. Shipment with dry ice can be sent to the destination Zagreb, Split, Rijeka, Pula, Zadar, Varazdin, Osijek, and Slavonski Brod only. If your laboratory is not located in one of these cities and in order to receive the parcel with dry ice, please indicate a delivery address within these cities, and state this delivery address in your registration. Advice the recipient to make sure that the sample will not defrost.

**During your registration:**

You can use a short acronym and write it in your local language phonetics, but only LATIN letters can be accepted (please do not use language-specific letters, e.g. Š, Å, í … Greek or Cyrillic, as those are not accepted to prepare the waybills).

2. **The "City" in the sample delivery address MUST be written in English (e.g. "Rome", "Prague" instead of "Roma (IT) or "Praha (CZ)")!**
3. Please **check carefully the sample delivery address in the registration confirmation** sent to you upon your registration. For any error please correct it on the registration form by the registration deadline. During the registration period you and any member of your laboratory can change any of your laboratory's entries on the registration page as often as you like. Following any change of your registration data, you will receive a new email confirming registration for participation / non-participation with the changed data.

For further information (e.g. Target Pesticides List, Calendar, Participation Fee…) please visit the EUPT-SRM17 Website: http://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=200&CntID=1192&Theme_ID=1&Pdf=False&Lang=EN
The Specific Protocol for the EUPT-SRM17 will be dispatched two weeks prior to the shipment.
If you have any questions, please contact us EURL-SRM@cvuas.bwl.de

Best regards,
The EURL-SRM Team

## Guide to EUPT-SRM17 Results Submission Webtool

Version: 2022-02, Date: 23-02-2022, Init: Schr

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

### General Information:

- **Please only use GOOGLE CHROME or FIREFOX Web-browsers under INCOGNITO mode.**
  The latest version of these browser softwares is recommended.
  **DON'T USE Internet Explorer or MS Edge or other browsers as the** Webtool isn't validated for those.
- **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!**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**Edit results** ..... 7
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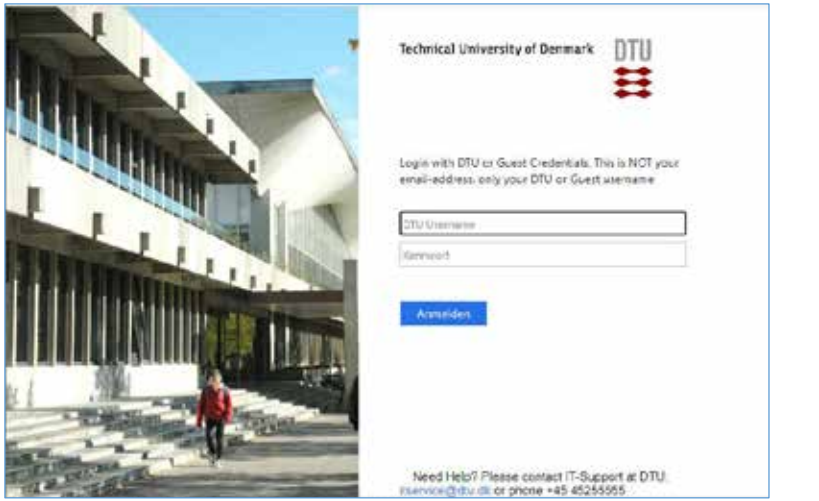
Guide to EUPT-SRM Results Submission Webtool

Getting started

**Link to Webtool: www.eurl.dtu.dk**

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


**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).






Appendix 12. Guide to EUPT-SRM17 Results Submission Webtool

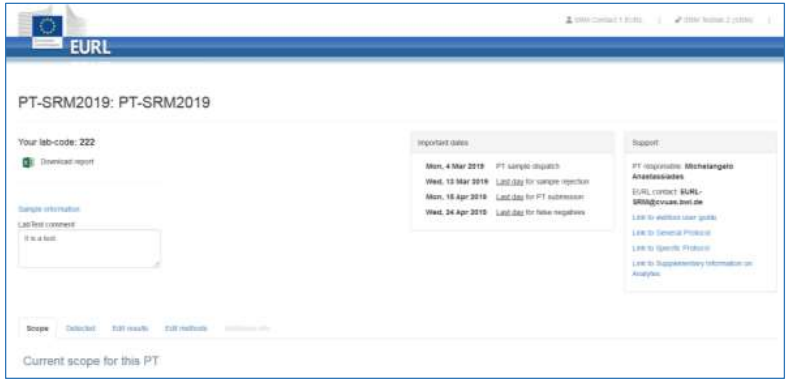
After signing in you will be guided to the *Proficiency Test Overview* page

***Proficiency Test Overview***

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


By clicking on "**SRM17 | Tomato Homogenate**" under "*My proficiency tests*", you will see the **current scope for this EUPT** in alphabetic order. (The screenshots in this Webtool guide are exemplary only and the PT names may differ from "SRM17, Tomato Homogenate".)

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.**



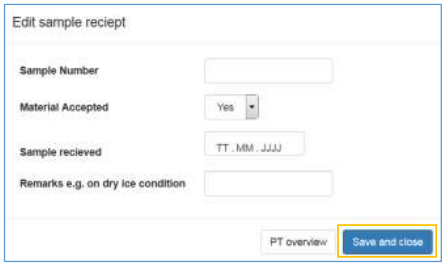



***Sample receipt and acceptance***

The **Webtool for the EUPT-SRM17 result submission** will expectedly open on **4 February 2022**.

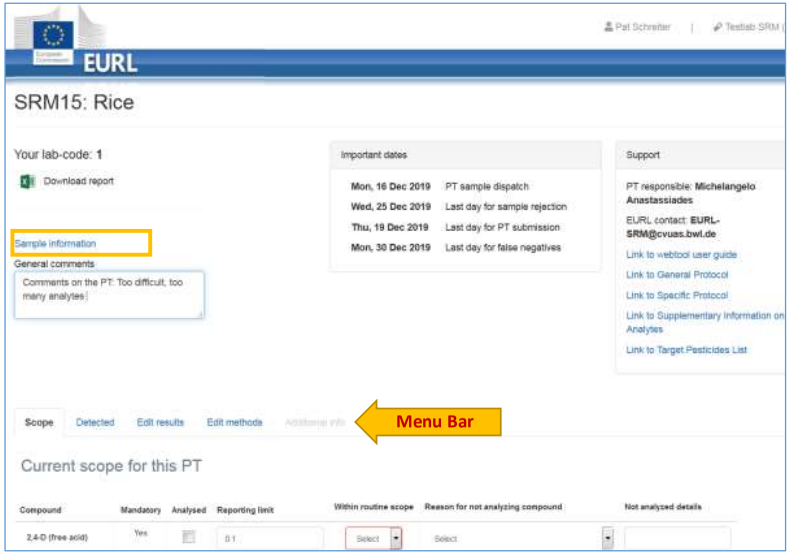
Once you have received the parcel with the PT-materials, please click on EUPT-SRM17 under "*My proficiency tests*" to open the pop-up window "**Edit sample Receipt"**. Please fill-in the information requested within this pop-up-window:

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

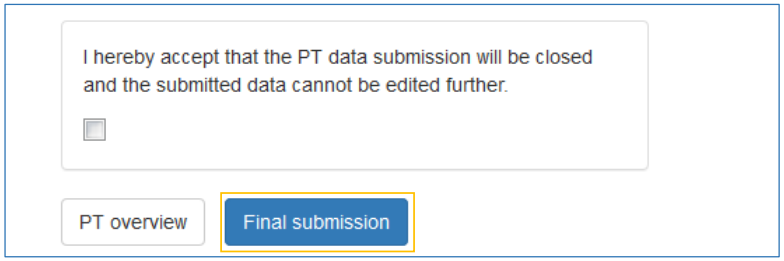



Completing the "*Edit Sample Receipt*" window is a **precondition for being able to continue the submission page.** This should be done **ideally shortly after parcel receipt**. You can, however, access and edit all the entries on "*Edit sample receipt*" throughout the PT-period under "*Sample information*"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

Upon clicking on "*Save and close*" you will be guided to the following page 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.**



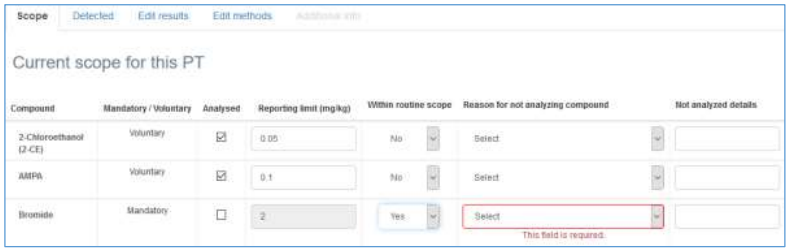





***Scope***

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".



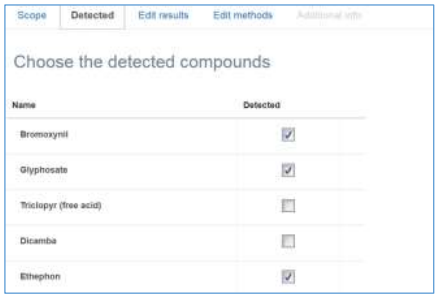

In this table, please firstly select the analytes you have targeted within the EUPT-SRM17 and then enter your Reporting Limit (RL) of each analyte within your PT Scope. The MRRLs were set as default reporting limits. For each pesticide within your PT Scope please **change the Reporting Limit to that of your laboratory**.

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

Even if your laboratory doesn't 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***

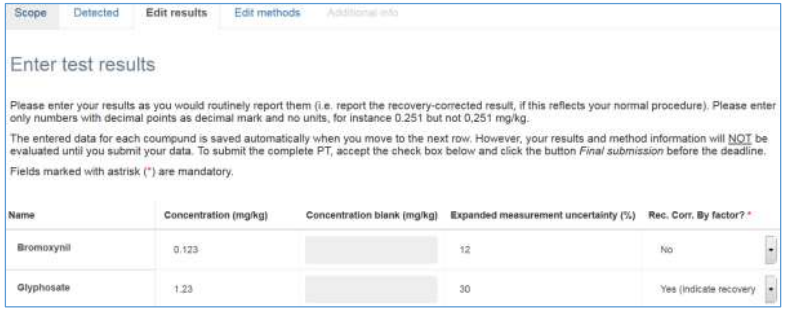
This page will list only analytes that were selected as "analysed" under the table "Scope". Please mark the analytes that you have detected in the Test Item. These selections are used as filters for the subsequent "*Edit results*" table.



## 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.** A 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** **(= Comments on results and on subcontracting, where applicable)** | Here you may add any remarks concerning aspects covered by this subpage for the concerned analyte as well as **any information concerning the SUBCONTRACTING of this analysis to another lab**. Please name subcontracted lab and indicate if this subcontracting reflects routine procedures |

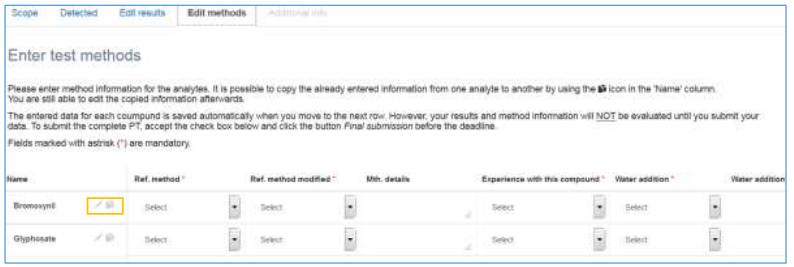




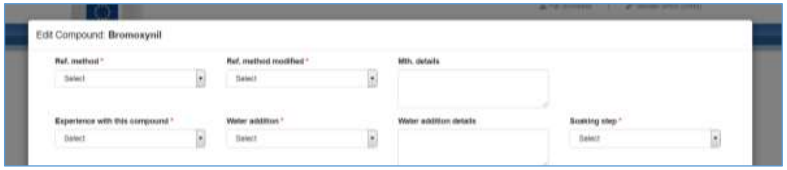
## Edit methods

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

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




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




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

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

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

| Field(s) | Explanation |
|---|---|
| **Ref. method** | Choose from the dropdown list. If you have used a modified form of the mth, pls. give details under "Mth Details" |
| **Ref. method modified** | Specify if you have introduced any noteworthy modifications to the selected reference method. If yes, pls. give brief details of the modification under "Mth. details". |
| **Mth. details** | Describe your method shortly if it is not on the dropdown menu or indicate shortly the modifications introduced to the selected reference method. |
| **Experience with this compound** | Experience of your lab with the analysis of this pesticide (with any type of commodity). |
| **Initial Sample Temp** | Initial Temperature of Sample when starting the extraction procedure (choose closest value) |
| **Sample thawed prior to anal** | Please indicate if and for how long approximately your sample was left in a THAWED state after reception until analysis of the compound |

| Field(s) | Explanation |
|---|---|
| **Details on sample thawing** | Please provide any relevant details relevant to the thawing to the sample (e.g. "thawed over night in refrigerator") |
| **Sample Weight (g)** | Enter the weight (in gram) for the analytical portion. |
| **Extraction/partitioning solvent 1** | Choose the solvent from the dropdown menu |
| **Extraction/partitioning solvent 2** | Choose the solvent from the dropdown menu, if you use more than one solvent. |
| **Extraction/partitioning solvent 3** | Choose the solvent from the dropdown menu, if you use more than two solvents. |
| **Extraction solvent details** | Enter details on solvents used in extraction or partitioning steps or if the solvent is not in the drop down menu |
| **Extraction Time** [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 "Chemical transformation Time" |
| **Extraction approach** | Choose extraction approach from dropdown list |
| **Partitioning salts used** | Choose partitioning salt used from dropdown list |
| **pH modified** | Indicate if you have modified the pH at any stage of the procedure (e.g. by buffering, acid/base addition) |
| **pH modified details** | Please give details on pH modification step(s) |
| **Clean up 1** | Choose the clean-up approach employed from the dropdown list |
| **Clean up 2** | Choose the clean-up approach employed from the dropdown list, if you use more than one clean up step |
| **Clean up 3** | Choose the clean-up approach employed from the dropdown list, if you use more than two clean up steps |
| **Clean up details** | Please give details on clean-up step or describe your clean up procedure if it is not listed in the dropdown menu |
| **Chemical transformation** | Mark if your procedure included a chemical transformation e.g. hydrolysis, derivatization, reductive cleavage to $CS_2$, etc. |
| **Chemical transformation Time** | Please chose closest time from dropdown list. **If extraction and chem. transf. were combined, indicate closest combined time here.** |
| **Chemical transformation Temp.** | Please choose the closest temperature from the dropdown list |
| **Chem. Transf. Details** | Please give details on chemical transformation step(s) conducted |
| **Calibration approach** | Choose the calibration approach used.<br>**NOTE:** "Procedural calibration" and "Standard additions to sample portions" involve correction for recovery. |
| **Determination Technique** | Choose the instrumental technique used to generate your quantitative result |
| **Other Approaches to Corr. PT-Result for Recov.** | Shortly describe any OTHER APPROACHES employed for correction of results for recovery.<br>**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. |



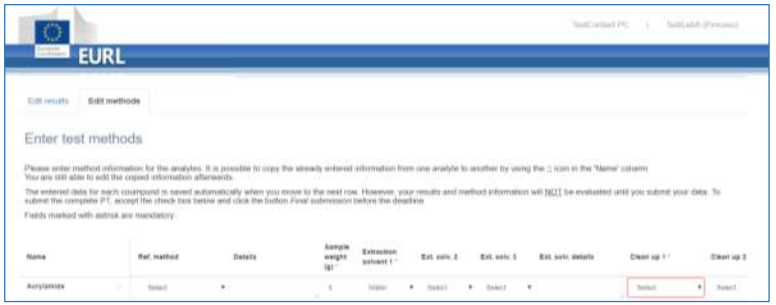




| Field(s) | Explanation |
|---|---|
| **IS used** | **Please choose "No" if no "IS" was used or if the IS was only used for quality control purposes and not for the calculation of the target analyte result**. Please choose one of the two "Yes" options if the IS was used for calculating the target analyte 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**<br>**(= Comments on methods and on subcontracting, where applicable)** | Please enter here any comments concerning the analytical method of the selected compound. **Here you can also indicate if the respective analyte was analyzed by a subcontract lab and if this reflects the routine approach. Please name the subcontracted lab**. |

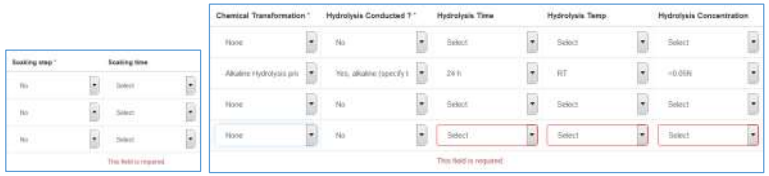
***Final Submission***

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




**Please note:**

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



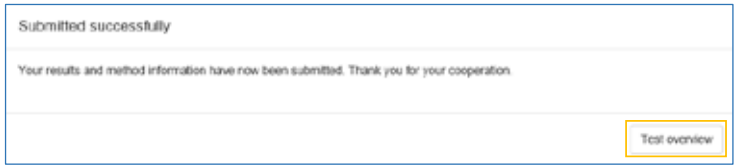
**In both screenshots, the entries are correct**, since no soaking step or hydrolysis was conducted and soaking time or hydrolysis time/temp/concentration are actually not required.
To "remove" the red rings or the sentence "This field is required": **just click on "select".**

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:**

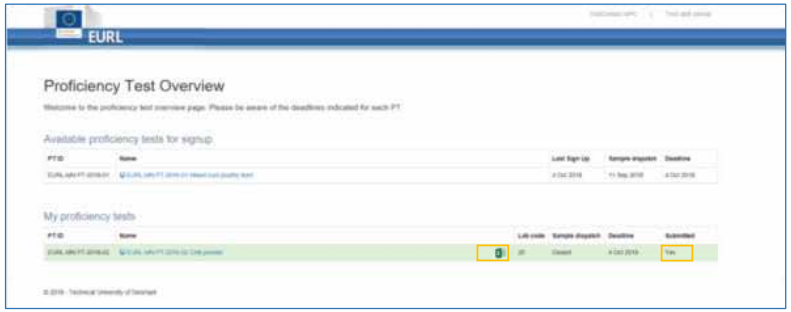
You will **NOT** be able to edit your data after the final submission!
Your data have to be submitted before the deadline on Tue. **8 March 2022, 23:30 h (11:30 p.m.), CET.**

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.




After final submission, you will not be able to edit your data any more! If you find any errors in the exported Excel-file and it is before the submission deadline, please contact the SRM17 organizers per Mail.



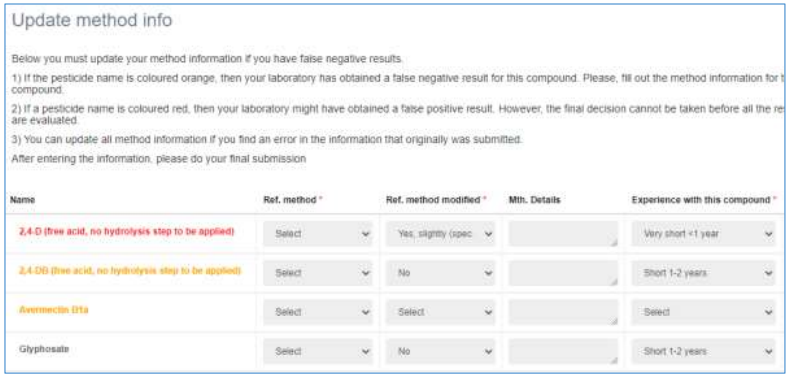


***Additional Information***

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:




**Tentatively false positives are written in red colour.**

**Tentatively false negatives are written in yellow/orange colour.**

Please fill in **the missing method information for the compounds identified as tentatively false negatives. Submit this information by 17 March.**

During this period all method information on this page "Additional information" can be modified.