

WORK PROGRAMME of EURL for
PESTICIDE RESIDUES IN CEREALS
AND FEEDING STUFF

PERIOD: 2021-22

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INTRODUCTION

The main purpose of the European Union Reference Laboratory on Pesticide Residues in Cereals and Feedingstuff (EURL-CF) is to ensure the quality of the pesticide residue analysis performed on food and feed in the EU Member States (MSs). This includes the use of multi methods as well as harmonized procedures for quality control and validation of methods used. Most of the activities of the EURL-CF aim to implement 1) valid multi methods for analysing pesticide residues in cereals and feeds, 2) improving and boosting the communication in the EURL/NRL/OfL network, 3) strengthen education and training, 4) address knowledge gaps, and 5) ensure harmonization according to Commission Regulation (EU) 2017/625.

In addition, the EURL-CF will provide assistance to the MSs to reduce knowledge gaps and increase both analytical performance as well as the analytical scope. Furthermore, the EURL-CF is, together with EURL-FV, EURL-SRM, EURL-AO editor of the guidance document SANTE/12682/2019 on Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed (latest version).

The EURL-CF is hosted by the Technical University of Denmark, National Food Institute (DTU Food) as part of one of the activities of the Research Group for Analytical Food Chemistry. The Research Group is involved in all aspect of providing chemical data, which is a crucial part of food trust and transparency for healthy choice by consumers as well as for regulation by authorities and innovation by industry. The ambition in the Research Group for Analytical Food Chemistry is to provide and interpret these crucial data by developing and applying reliable, robust and detailed chemical analysis. Our focus is on trace analysis of small organic compounds in food, feed and biological samples. Our core fundament is high performance mass spectrometry coupled to chromatography, applying MS-MS, accurate mass spectrometry and isotope ratio measurements. Our objective will be reached by challenging state-of-the-art mass spectrometry, data processing and sample preparation. Methods for monitoring and metabolomics combine our efforts to:

- Develop cost-efficient, robust and scalable methods for food monitoring and control that are specific and sensitive as well as of value for the official food control.
- Develop screening methods that are comprehensive to discover emerging risk.
- Maintain a metabolomics platform to unravel biological effects and mechanisms of chemicals.
- Co-develop new analytical technologies and strategies.

DTU Food supports the EURL-CF with basic housing as well as administrative support, IT support and basic laboratory necessities. In 2021, DTU Food will, as earlier years, co-finance the overall budget as part of their responsibility as NRLs on pesticide residues. However, the financial system (databases etc.) at the University is not well suited to incorporate different financial sources to the projects. Consequently, the co-finance will not appear in the budget, but in reality still be allocated.

Regulation (EU) 625/2017 Art 94(2):

European Union reference laboratories designated in accordance with Article 93(1) shall be responsible for the following tasks insofar as they are included in the reference laboratories' annual or multiannual work programmes that have been established in conformity with the objectives and priorities of the relevant work programmes adopted by the Commission in accordance with Article 36 of Regulation (EU) No 652/2014:

(taking into account Art 147 of (EU) 625/2017)

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TO ENSURE AVAILABILITY AND USE OF HIGH QUALITY METHODS AND TO ENSURE HIGH QUALITY PERFORMANCE BY NRLs.

Please, provided activities related to Regulation (EU) 2017/625:
(Number of Sub-activity boxes can be adjusted by EURL)

- **Art. 94.2.a** *Providing national reference laboratories with details and guidance on the methods of laboratory analysis, testing or diagnosis, including reference methods.*
- **Art. 94.2.b** *Providing reference materials to national reference laboratories*
- **Art. 94.2.c** *Coordinating the application by the national reference laboratories and, if necessary, by other official laboratories of the methods referred to in point (a), in particular, by organising regular inter-laboratory comparative testing or proficiency tests and by ensuring appropriate follow-up of such comparative testing or proficiency tests in accordance, where available, with internationally accepted protocols, and informing the Commission and the Member States of the results and follow-up to the inter-laboratory comparative testing or proficiency tests.*
- **Art. 94.2.l** *Where relevant for their area of competence, cooperate among themselves and with the Commission, as appropriate, to develop methods of analysis, testing or diagnosis of high standards.*

Sub-activity 1.01 Provide NRLs with details and guidance on the methods of laboratory analysis

Objectives: Provide information to NRLs concerning method, PTs and general knowledge sharing.

Description:

1.01.1 Updating the EURL website and the CIRCA domain

Maintenance of the common web portal <http://www.eurl-pesticides.eu> for the pesticide EURLs (horizontal task – see description in AWP for EURL-SRM).

The communication platform supports the coordination activities of the EURLs and serves as contact, reference and service points for the National Reference laboratories and official pesticide residue laboratories in Europe. The website for Cereal and Feeding stuff will be maintained in coordination with the other EURLs and the following items will be updated: Presentation of the EURL, information on proficiency test, training courses, workshop, annual work programme, validation data and analytical methods. The website is accessible for everybody. Additional input will be provided to the development of the DataPool, especially the EUPT-Archive.

Maintenance of the common CIRCA domain (horizontal task – see AWP for EURL-AO). The CIRCA domain is only accessible for a limited number of persons, mainly persons employed in NRLs or OfLs. Consequently, information can be uploaded to this platform if it has a more confidential content. It could e.g. be data that could be published in scientific papers. However, the platform can also send emails when new documents are uploaded. Therefore, information put on the open web portal will also be uploaded to the CIRCA platform.

1.01.2 Updating the EURL DataPool

Validation results will be up loaded to the DataPool and the list of NRLs and OfL will be maintained in cooperation with the EURL-SRM.

Expected Output: New webpages created or webpages updated

Duration: Throughout 2021-22

Sub-activity1.02 Organisation of proficiency tests and follow-up on the results

Objectives: Checking the analytical performance of the NRLs and OfL contributing to the EU monitoring programme.

Description:

1.02.1 Organisation of EUPT-CF15 on rape seed cake

The proficiency test will cover pesticides analysed by multi residue methods. The target pesticides will include 214 pesticides, as agreed on by the Advisory Group and will include, as a minimum, pesticides concerned from the latest version of EU Regulation for the Multi-Annual Control Programme for pesticide residues. The rape seeds, which will be used as test item, were produced by a consultant and will be further spiked in the laboratory. After spiking, the oil will be pressed out of the rape seeds. The oil will be stored and used as Proficiency Test material by EURL-AO, if agreed by the Scientific Committee. The rape seed cake will be milled and homogenized and portions will be weighed out directly into screw-capped polyethylene plastic bottles, sealed, numbered, and stored in a freezer at about -20 °C prior to homogeneity and stability testing. Before shipment of the test items, 20 homogeneity experiments will be performed (double determinations of 10 randomly selected test

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items). Stability test will be performed on several occasions during the PT period, in total 50 experiments.

It is foreseen that around 110 EU NRLs and EU OfLs will participate in the PT and additionally 5-10 official laboratories from Third Countries. One meeting with the Scientific Group is foreseen. The meeting will probably be held in Spain.

1.02.2 Preparation of test items for proficiency test 2022, EUPT-CF16

Field spraying of barley will be performed by Aarhus University in 2021 in order to produce test material for EUPT-CF16 with incurred pesticides. Barley is on the Multi Annual Control Programme 2022.

1.02.3 Organisation of EUPT-CF16 on barley in 2022.

The proficiency test will cover pesticides analysed by multi residue methods. The target pesticides will include more than 214 pesticides, as agreed on by the Advisory Group and will include, as a minimum, pesticides concerned from the latest version of EU Regulation for the Multi Annual Control Programme for pesticide residues. The barley kernels will most likely be further spiked in the laboratory. After spiking, the kernels will be weighed out directly into screw-capped polyethylene plastic bottles, sealed, numbered, and stored in a freezer at about -20 °C prior to homogeneity and stability testing. Before shipment of the test items, 20 homogeneity experiments will be performed (double determinations of 10 randomly selected test items). Stability test will be performed on several occasions during the PT period, in total 50 experiments.

It is foreseen that around 150 EU NRLs and EU OfLs will participate in the PT and additionally 5-10 official laboratories from Third Countries. One meeting with the Scientific Group is foreseen. The meeting will probably be held in Spain.

1.02.4 Preparation of test items for proficiency test 2023, EUPT-CF17

If necessary, field spraying of a relevant cereal or feed crop will be performed by a consultant in order to produce test material for EUPT-CF17. The proficiency test will focus on crops agreed upon by the Advisory Group. However, a relevant feed commodity would be of high interest.

1.02.5. EUPT result submission website and tools

A new database and web tool for the annual proficiency tests (PTs) has been developed during 2018. The web tool has been individually organized for each EURL. Before the PT the organisers (EURLs) will, in coordination with the system programmers, (from the AIT department at the University) set up the database and web tool. Furthermore, it is foreseen that minor improvements of the system will be implemented.

To set up a PT, the EURL must incorporate the dates and deadlines, select the pesticides included in the PT in question from the global pesticide list. If additional or different information from the participants is needed, the programmers will adjust the forms for result submission. Before the system is opened for use, the participants who signed up for the PT via the DataPool will be uploaded to the system by the programmers and lab codes will be generated. After the deadline for result submission, data is extracted and transferred to the EURLs for further statistical evaluation of the laboratories performance. A guide and/or instructive videos on 1) how the EURLs can manage the system and 2) how the participant must enter their data will be prepared.

Planned EUPTs 2021: EUPT-CF15, FV23, AO16 and SRM16

Planned EUPTs 2022: EUPT-CF16, FV24, AO17 and SRM17

Maintenance during the PTs:

When the EUPTs are running the result submission will be supervised by the AIT department at the DTU to help and support the participants if required. After deadline for result submission, the data will be extracted from the database for statistical processing. This is done by the individual EURLs.

Expected Output: Each year; Draft report, final report, certificates to the participants, around 70 kg rape seed cake and 30 kg rape seed oil Test Item with incurred and spiked pesticide residues, 50 kg of barley kernels with incurred and spiked pesticide residues and web tool user guides.

Duration: Throughout 2021-22

Sub-activity 1.03 Method development employing Accurate Mass Spectrometry

Objectives: Further development of screening method using Accurate Mass Spectrometry

1.03.1 Increasing performance of screening method using GC Accurate Mass Spectrometry and applicability of the simultaneous screening and quantitative analyses by GC accurate mass instrumentation

In 2019, a high resolution accurate mass database was created and updated on the new purchased GC-Orbitrap-MS. The high resolution (>60000), the high mass accuracy (mass error < 1ppm), the fast scans, and the high sensitivity offered by the GC-Orbitrap-MS, give this instrument advantages over others to be used for both screening and quantitation purposes. In 2020, using the updated database, a combined screening and quantitation method was validated for 100 pesticides using target screening and quantitation methods developed on the TraceFinder software.

In 2021, with the possibility of screening for thousands of analytes in a single run, and in the frame of enlarging the screening scope of analysis, using this high technology and using the updated database, more pesticides will be validated for screening and quantitation on the GC-Orbitrap-MS. For the quantitation, the focus will be on pesticides included in the MACP, and data will be processed using the quantitation methods. Whether for screening, the focus will be on pesticides not included in the MACP, and data will be processed using target screening methods.

Within the frame of exploring new tools for screening of pesticides in food, a new software, "compound discoverer" from Thermo Scientific has been installed on the GC-Orbitrap-MS. The software allows the identification of unknowns, spectral library search, and elucidation of structure. It also allows performing statistical analysis. This software will also be explored in the screening analysis of pesticide residues in food and feed.

This feature is of highly relevance for the Sub-activity 1.06 Development of method for analysis of residues of co-formulants.

1.03.2 Increasing performance of screening method and applying simultaneous screening and quantitation analyses using high resolution LC-Q-ToF-MS?

In 2020, DTU-Food department has purchased a high resolution LC-ESI-QToF-MS Maxis II™ from Bruker. The instrument has a resolution of 60 000 FWHM which generally provides a mass error <1 ppm for almost all LC amenable compounds. The instrument offers higher resolution and higher sensitivity than the LC-QToF from Agilent, which improves the performance and reliability of pesticide screening methods. LC-QToF-MS Maxis allows working in Broadband Collision Induced Dissociation (bbCID) mode. The bbCID is a data acquisition process where both TOF-MS full scan data and MS/MS fragments are continuously generated in two independent, rapidly alternating data channels, without either precursor ion or threshold selection. The Maxis ToF database used for target screening analysis does include retention times, contrary to the PCDL provided by the Agilent instrument. The Maxis pesticides database contain a minimum of 2 fragment ions for each analyte, with a total of 942 compounds. Fragments in the database are marked if generated from the FullScan or from bbCID. The

instrument is equipped with a Target Analysis for Screening and Quantitation (TASQ) software that is used for data processing allowing qualitative and quantitative batch processing. In the coming year, experimental spectra will be recorded for, tentatively, the 942 pesticides.

In 2020, the acquisition method was developed on the LC-QTOF-MAXIS. The chromatographic acquisition parameters, such as flow gradient, and the MS parameters were optimized. The database retention times and fragments were adjusted and verified for more than 500 compounds by injecting standard mixes. In 2021, the work on this instrument will be carried on. Last year, using the optimized method and the updated database, 50 pesticides were validated for screening using the criteria for HRMS for identification and confirmation. In the coming year, a larger number of pesticides (at least another 50) are going to be included in the screening validation method.

1.03.3 Further development of methods for quantification of pesticides from marker compounds using Accurate Mass Spectrometry

The Marker Compound project in 2019-2020 concluded the use of marker compounds to calculate content of other compounds seem promising as more than 90% of the 86 pesticide were semi quantified with a difference below <25%. However, the study included a limited number of data and more research in this area is needed.

The work with quantification without standards will be continued with the aim to test the robustness of the approach, over time. A continuously analyzes of calibration standards and spike samples will be analysed each month over an 18 month period will enlarge the amount of data and thereby decrease the uncertainty of the results. As the relationships between the responses of the marker compounds and the compounds in question may vary depending on the condition of the instrument and/or instrument settings the calibration standards and spike samples will be inject before and after analyses of samples, to investigated how clean or dirty ion sources, new or old columns, liners etc. will influence the outcome. Until now only 86 compounds has been included in the study, by analysis on the GC-Orbitrap-MS. However, more compounds will be included and these will cover more pesticides from the MACP which also allow quantification of PT test materials to be include in the calculations.

Expected Output: Reports uploaded to CIRCA platform

Duration: 2021-22

Sub- activity 1.04 Studies on possibilities to include pesticides in multimethod for cereals/feeds

Objectives: Studies on possibilities to implement pesticides in multi residue method and gaining knowledge on achievable LOQs

Description: Inclusion of more pesticides in the quantitative LC-MS/MS and GC-MS/MS methods is a constant request. New pesticides are authorised for use in EU, the number of pesticides included in the MACP are generally expanded, the pesticides relevant in regard to Regulation 2019/1793 is changing etc. Furthermore, EFSA is continuously collecting data on analytical methods and LOQs achieved to renew the MRLs, to update the residue definition by including relevant metabolites, and to refine pesticide residues intake calculations and this will be possible due to more sensitive GC-MS/MS and LC-MS/MS instruments. Consequently, the work on optimising the multi residue method for cereals and feeds will therefore be continued.

1.04.1 Studies on the possibility to include new pesticides/metabolites in multimethod for cereals.

Each year a validation study will be performed using the QuEChERS method or other relevant multi residue methods for around 20 pesticides. The pesticides chosen will mainly be from the EFSA Progress report MRL reviews (Article 12 of Regulation (EC) No 396/2005), MACP, the Working Document

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SANCO/12745/2013 and new authorisations. However, also compounds for which an MRL is set based on actual use of the pesticides is. The final decision on which pesticides to include will be based on last minute information on the most relevant pesticides to cover. However, e.g. the following pesticides would be of interest: 4-bromophenylurea, 8-Hydroxyquenoline, Aminopyralid, Azadirachtin, Bifenazate, Bifenazate-diazene, Chloridazon-desphenyl, Cinerin, Clopyralid, Diclofop, Diclofop-methyl, Dithianon, Eugenol, Geraniol, Fenobucarb, Flupyradifurone, Ioxynil, Isofetamid, Isoxaben, Jasmoline, Lenacil, Oxadiazon, Picloram, Pinoxaden, Pinoxaden Metabolite SYN502836, Pinoxaden Metabolite SYN505164, Pyraclonil, Pyrethrin, Rimsulfuron, Halosulfuron-methyl, Thymol.

The studies will additionally enable the EURL to contribute to the Art. 12 process by establishing new MRLs for the pesticides in question. The study will include minimum three cereal matrices and be spiked at concentration level at ≥ 0.005 mg/kg. The validation data will be generated, converted in the specific format and uploaded to the common database. This information is important for EFSA and the official laboratories.

1.04.3 Study on lowering the LOQ in cereal based babyfood.

Validation performed in 2020 on rice based babyfood showed that very low LOQs were achievable for many pesticides. Out of 67 pesticides/isomers 80% were validated with an LOQ of 0.0005 mg/kg and 16% at 0.001 mg/kg. The EURL has in relation to the experiments in 2020 prepared extract for validation of additional >300 pesticides. These extracts have been stored at -80 °C and will be analysed on GC-MS/MS and LC-MS/MS. From the recovery, repeatability, reproducibility a LOQs will be calculated in accordance with SANTE/12682/2019.

Expected Output: Reports and updates of the DataPool.

Duration: Throughout 2021-22

Sub-activity 1.05 Development of a microSPE clean-up method

Objectives: MicroSPE clean-up of cereal and rapeseed sample extract using TriPlus RSH MicroSPE Purification Platform

1.05.1 Automatic clean-up of cereal samples extracts

Description: In 2020, DTU-Food department has invested in a stand-alone TriPlus RSH MicroSPE Purification Platform. The equipment is an automatic system that can be used to clean-up sample extract from e.g. QuEChERS extraction by using a μ SPE column. The standard clean-up procedure following QuEChERS extraction and applied in our lab consists of the use of d-SPE. A mix of $MgSO_4$ and PSA is added directly to the extract and the extract is shaken and centrifuged, and the clean supernatant is ready to analyse. The μ -SPE is a simple scale-down or miniaturization of the normal size cartridge-SPE and it consists of a bed of sorbents (C_{18} , PSA, $MgSO_4$, and CarbonX, or other combinations). What makes the use of the μ -SPE more interesting in routine analysis is that it is mounted on a robotic arm, allowing automated sample clean-up and thus significant reduction of labour. It will improve working environment as the technician can avoid repeating pipetting that might over the years end damaging their shoulders. The system also can save time because it is automatic and can function automatically outside the normal work schedule. Besides the reduction of labour, and the time-saving, more importantly, the μ SPE in comparison to both the conventional cartridge-SPE clean-up and the newer d-SPE clean-up, is expected to reduce human error and to give more accurate results. Therefore the system could be of great importance for routine laboratories with high through put of samples.

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The system will be tested on extracts of different cereal matrices. A validation study of at least 50 pesticides, with three spiking concentration levels of 0.005, 0.01 and 0.05 mg/kg will be performed and the extracts will be cleaned-up by both d-SPE and μ SPE. The results will be compared with focus on recovery, repeatability, and removal of matrix compounds.

Besides the clean-up, the system offers the possibility of preparing robotically standard calibration levels which will reduce labour, reduce the time, and minimize human error. However, first, a specific workflow will be set, and this feature will be thoroughly tested.

Objectives: Automatic clean-up of fatty feed samples extracts

Description: In 2020, a validation of soya and rape seeds were performed using QuEChERS method extraction and dSPE clean-up with EMR. Both soya and rape seeds are difficult matrices with high fat content. Although the validation showed acceptable results we will investigate if the automatic μ SPE system are comparable or even superior the dSPE clean-up with EMR. The pesticides included will primarily be from the MACP and/or Directive 2002/32/EC and the spike will be performed at 3 concentration levels ≥ 0.005 mg/kg.

Expected Output: Reports and updates of the DataPool. One publication in per reviewed journal.

Duration: 2021-22

Sub-activity 1.06 Development of method for analysis of residues of co-formulants

Objectives: Gain knowledge on occurrence of residues of co-formulants in food and feed (postponed from 2019-2020 work programme)

Description:

In 2018 a project was initiated to gain knowledge on the occurrence of residues of co-formulants (additives/adjuvants/co-formulants) in food and feed. Co-formulants are used for the preparation of the pesticide formulations (or added prior to spraying) in order to obtain a product which is practical, safe and efficient to use. Co-formulants aid e.g. the distribution, uptake, stability and efficiency of the active ingredient (a.i.). More than 1500 adjuvants including co-formulants are represented in authorised plant protection products. Many of these are mixtures of several compounds, thus the number of compounds in use as co-formulants is high. In 2021 study will be initiated by toxicologists at DTU National Food Institute (outside the EURL framework), aiming to generate a list of co-formulants authorised for use with formulations authorised for use nationally. By gathering information on amounts consumed, physical/chemical and toxicological data it will aid the evaluation of which adjuvants are most likely to be of relevance for human and/or animal safety, due to likely occurrence of residues of toxicological relevance. Co-formulants belonging e.g. to the group of solvents of low vapour pressure are not likely to leave residues at the time of neither harvest nor consumption.

To our knowledge studies on whether residues of adjuvants, e.g. surfactants, occur on harvested crops are not available. However, the study EURL-CF performed in 2019 show that residues of some of the ingredients in the selected test formulations do occur in detectable levels on treated hay and it is therefore found relevant to continue the work.

The project was planned to be continued in 2020, but due to both the postponement of the toxicological project and the challenges with the Cov-Sars2 virus, the project was postponed to 2021 in agreement with the Commission. In 2021 the EURL-CF will continue the work on studying the occurrence of residues of co-formulants on food/feed. The above mentioned list of co-formulants used

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nationally, will aid the selection of a number of co-formulants, which are widely used and evaluated likely to leave detectable residues, and analytical methods will be sought developed for these selected co-formulants. Selected samples of food/feed will then be analysed for residues using the developed method. Generally residues are assumed most likely to occur on commodities treated with plant protection products at a time where the eatable parts are already formed and on eatable parts with a large surface area.

Expected Output: Analytical method for commonly used co-formulants and a report describing the employed extraction and analytical method.

Duration: 2021-22

Sub-activity 1.07 Development of method for analysis of residues in insects

Objectives: To develop a multi method that can detect and quantify pesticides in insects used for feed. Furthermore, the method will be used to quantify pesticide levels in insects from a feeding pre-study.

Background: According to the United Nation's Food and Agriculture Organization (FAO) projections, the world population will approach 9 billion by 2050. To meet demands for feeding the growing population, food production will need to increase by 70% requiring more land supply and water use, thus leading to negative environmental effects. To address this, alternative food and feed sources are developing quickly worldwide. Specifically, there has been a growing interest in using insects as food and feed to address food insecurity and environmental sustainability issues. More than 2000 insect species are edible and are common delicacies in Asia, Africa and South America. Although their consumption in the EU MS is not common, it has recently been gaining interest. However, the use of this protein source will, most likely, be as feed and not as food. Practices of utilizing food waste to feed insects will contribute to a circular economy. In Denmark, a discussion has started about whether it should be allowed to use food waste from canteens to feed insects. However, also side streams from productions, earlier considered to be waste, could also be introduced to the food chain via feed to insects, and then be used as feed for livestock. So far only very scattered analytical methods for pesticides in insects are published and consequently only few data on pesticide levels in insects are found in the literature.

Description: In 2022 a method will be developed using larvae of black soldier fly (*Hermetia illucens*). Blank samples will be spiked with 20 pesticides frequently found in food from traded in EU. Five parallel recovery samples will be performed and repeated 3 times. The samples will be spiked at 3 levels, 0.01, 0.02 and 0.05 mg/kg. The extract will be analysed on GC-MSMS and LC-MSMS. From recovery, repeatability, reproducibility the LOQs will be calculated in accordance with SANTE/12682/2019. The method will be used to analyse three samples from the feeding pre-study of black soldier fly larvae. These have been fed on chicken feed, spiked with boscalid, etofenprox and fluopyram.

Expected Output: Validation report including results from feeding pre-study.

Duration: 2021-22

Sub-activity 1.08 Follow-up on EUPT results in relation to different calibration approaches

Objectives: Examine changes in results by using calibration standards prepared in solvent, in same type of matrix, in other type of matrix, using procedural calibration or standard addition

Description: Results from the EUPT-CF15 on rapeseed cake showed very high standard deviation between the participants. Rapeseed cake was expected to be a difficult commodity to analyse and the results clearly confirmed that given Robust Standard Deviation (Algorithm A standard deviation) between 25-35%. Typically the STD in EUPT-CF is lower than 20%. Part of the uncertainty was due to different calibration approach, e.g. standard addition, procedural calibration, matrix matched calibration or calibration in pure solvent. Those participants using procedural calibration or standard addition got 20% higher results for several of the pesticides. Also the standard deviation was lower. On the contrary, those laboratories using calibration standards in solvent obtained 25% lower results.

To elucidate if these results can be confirmed under repeatability condition in the EURL-CF laboratory the EUPT-CF15 test material will be analysed and quantified using different calibration approaches. The repeatability condition will cover analytical method (extraction and clean-up), instrument, date, personnel. The calibration approaches will be: calibration standards 1) in solvent, 2) in matrix (using the blanks of the same rapeseed cake as used for the PT and in oat), 3) procedural calibration (using the blanks of the same rapeseed cake as used for the PT and in oat and 4) standard addition. The participant did not received the blank rapeseed cake and oat is selected for this experiment because it has a relatively high fat content and it was actually also used by one of the participants.

The results of the different calibration approaches will be compared in terms of matrix effect, signal suppression/enhancement. The evaluation will also investigate peak shapes, areas, calibration curves slopes. From the results the best calibration approach will be indicated taken into account the possibility given in the routing laboratories due access of blank materials, complexity of analytical batch (different commodities analysed in the same batch) and time available.

Expected Output: Report and presentation at EURL-CF workshop

Duration: 2021-22

Sub-activity 1.09 Coordination project to between DataPool and EUPT database

Objectives: Harmonization of EURL-DataPool and EUPT-Database to facilitate mutual data-transfer and to minimize work-intensive and error-prone manual editing.

Background: The EURL-DataPool and the EUPT-Database are independent databases run by the EURL-SRM and the EURL-CF, respectively. Both include among others a list of laboratories, a list of pesticides as well as EUPT-related data. For synchronization data is periodically transferred from the EURL-Datapool to the EUPT-Database and vice versa. In absence of common unique identifiers (coding system) between the databases, data transfer between the databases currently involves extensive manual editing and is prone to errors. For example pesticide names (English) can be spelled and misspelled in many ways, e.g.: endosulfan alpha; endosulfan alfa; endosulfan, alpha-; endosulfan-alpha; alpha endosulfan; α -endosulfan and α endosulfan.

Description: This task will involve coordination between the affected EURLs and the necessary changes within the two databases (e.g. installation of a joint coding system) in order to obtain the desired/achievable degree of harmonization and a simpler data-transfer. The possibilities of installing tools for semi-automatic data synchronization/transfer between the databases in the future will be

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examined at a technical level. Incorporation of information concerning the EUPT Target Pesticides within the databases and development of a tool for exporting the target pesticide list as well as EUPT-participation certificates.

Expected Output: Harmonization of databases. Implementation of a tool for generating the EUPT-participation certificates and EUPT-target pesticide lists by the end of 2022

Duration: 2021-22

Sub-activity 1.10 Cooperation with other EURLs

Objectives: Optimize utilization of resources; enhance mutual exchange of knowledge and experience.

Description: To utilize the resources granted to the EURLs on pesticide residues, each EURL has different horizontal task that covers all EURLs. This includes the EURL portal, CIRCA domain, PT result submission database and webpage etc. and the Scientific Group. To coordinate the activities meetings are organized and emails are used. Two coordination meetings are foreseen. The meetings will probably be held in Spain, one of them in continuation of the Scientific Group meeting.

In 2021 the SANTE document on Method Validation & Quality Control Procedures for Pesticide Residues Analysis in Food & Feed (SANTE/12682/2019) will be updated and additional meetings will be organised with the Scientific Committee.

Expected Output: Not measurable, but short minutes from meetings will be written

Duration: 2021-22

Sub-activity 1.11 Administration and accreditation

Objectives: Fulfilment of administrative duties as well as accreditation requirements.

Description:

1.08.01 Administrative duties such as drafting budget and work programme, as well as compilation of annual technical and financial report, will be performed according to the requirement issued by the Commission.

1.08.2 Maintenance of in-house QA/QC activities in according to ISO 17025 accreditation of all analytical work done within the EURL and ISO 17043 in relation to the proficiency test organized by the EURL. Next audit by the Danish Accreditation Body DANAK will be in January 2022

Expected Output: Budget 2021-22, work programme 2021-22, financial report 2019-20 and 2021, Technical report 2019--20 and 2021.

Duration: on-going

100% financed by DTU and not included in the budget

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TO PROVIDE SCIENTIFIC AND TECHNICAL ASSISTANCE TO NRLs

Please, provided activities related to Regulation (EU) 2017/625:
(Number of Sub-activity boxes can be adjusted by EURL)

- **Art. 94.2.d** *Coordinating practical arrangements necessary to apply new methods of laboratory analysis, testing or diagnosis, and informing national reference laboratories of advances in this field.*
-
- **Art. 94.2.e** *Conducting training courses for staff from national reference laboratories and, if needed, from other official laboratories, as well as of experts from third countries.*
-
- **Art. 94.2.g** *Providing information on relevant national, Union and international research activities to national reference laboratories.*

Sub-activity 2.01 Providing technical and scientific support to NRLs

Objectives: Service to NRL on request – knowledge sharing.

Description: NRLs occasionally contact the EURL and ask for advice e.g. on purchase of new instrument, analytical problems, mainly via mails

Expected Output: Short report on the number and type of requests from the NRLs

Duration: Throughout 2021-22

Sub-activity 2.02 Organisation of Joint workshop 2021

Objectives: Enhance the skills of staff from NRLs and OfL involved in pesticide control.

Description: A joint workshop will be organised for the NRLs and OfL for pesticide residues. The main organiser will be EURL-FV

Duration: 2021

Sub-activity 2.03 Organisation of workshop 2022

Objectives: Enhance the skills of staff from NRLs involved in pesticide control.

Description: A workshop will be organised for the NRLs and OfL for pesticide residues. The workshop will be held in Copenhagen in 2022. Participation for eligible members of EU-NRLs for pesticide residues will be free of charge.

EURL-CF

The agenda for the workshop will include results from proficiency test, discussion on coordinated monitoring programme, analytical challenges, accreditation issues and other relevant issues. One expert from each country will be reimbursed.

Expected Output: Workshop minutes, pdf of oral presentations, evaluation schemes

Duration: 2022

Sub-activity 2.04 Organisation of training courses 2021

Objectives: Organisation of training course

Description: In 2021, a 2-3 days training will be organised at the premises of the EURL. The training will presumably be on basic pesticide residues analyses including sample processing. The training will include hands on laboratory training on extraction, clean-up using dSPE and μ SPE and MS/MS analyses. Six NRLs will be invited and one participant from each NRL can join the training. The participants will be chosen among the EUPT Category B NRLs.

Expected Output: Pdf of oral presentations and evaluation schemes

Duration: 2021

Sub-activity 2.05 Organisation of training courses 2022

Objectives: Organisation of training course

Description: One day training at the premises of the EURL on basic pesticide residues analyses including on specific subjects from the SANTE/12682/2019 or later. The training will be organised in cooperation with EURL-AO.

Expected Output: Pdf of oral presentations and evaluation schemes

Duration: 2022

Sub-activity 2.06 Visits to NRLs

Objectives: Visit to an NRL that underperforms in PTs in order to help and support them to overcome obstacles and problems that they encounter.

Description: One NRL visit each year will be conducted to a laboratory selected in agreement with the COM, where the EUPTs results have been problematic over the last years. The task could be performed in collaboration with other EURLs.

Expected Output: Mission report

Duration: 2021-22

Sub-activity 2.07 Organisation of webinar

Objectives: To disseminate knowledge on specific subject.

Description: One webinar on demand is intended to disseminate information to the NRLs and official laboratories in a cost effective but still interactive way will be organised.

Expected Output: Pdf of oral presentation

Duration: 2021-22

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TO PROVIDE SCIENTIFIC AND TECHNICAL ASSISTANCE TO THE EUROPEAN COMMISSION AND OTHER ORGANISATIONS

Please, provided activities related to Regulation (EU) 2017/625:
(Number of Sub-activity boxes can be adjusted by EURL)

- *Art. 94.2.f Providing scientific and technical assistance to the Commission within the scope of their mission.*
- *Art. 94.2.h Collaborating within the scope of their mission with laboratories in third countries and with the European Food Safety Authority (EFSA), the European Medicines Agency (EMA) and the European Centre for Disease Prevention and Control (ECDC).*
- *Art. 94.2.i Assisting actively in the diagnosis of outbreaks in Member States of foodborne, zoonotic or animal diseases, or of pests of plants, by carrying out confirmatory diagnosis, characterisation and taxonomic or epizootic studies on pathogen isolates or pest specimens.*

Sub-activity 3.01 Technical and scientific assistance to the Commission

Objectives: Support the Commission on relevant issues.

Description:

3.01.1 Information on LOQs, residue definitions and standards for Art. 12 MRL reviews, new active substances and other substances, when requested by COM.

Coordinate input for the four EURLs, or provide information to EURL-SRM for coordinated input, on achievable LOQs, availability of standards and comments to suggested residue definition in connection with approval of Reasoned Opinion in connection with art. 12 reviews.

3.01.2 Assistance to COM for the EU MACP and the monitoring working document

Comments and input is given concerning the coordinated multiannual control programme in regard to choice of commodities, scope, candidate compounds, availability of standards etc. Contributions will be made on request and in connection with participation in the annual Expert Working group meeting on pesticides monitoring.

3.01.3 Contribution to the revision of the analytical quality control guidelines

Every second year an updated version of the guideline is issued and made available on the EURL website. Whether relevant improvements and adjustments should be made is an ongoing process and the document is discussed among the EURLs as well as with the NRLs/OfL network on various occasions. Suggestions for improvement/adjustments are registered and presented in connection with the biennially drafting of the new versions. Two meetings in Spain organised by EURL-FV are foreseen.

EURL-CF

3.01.4 General technical support to the Commission

On request

Expected Output: Various mails and brief reports

Duration: 2021-22

Sub-activity 3.02 Collaboration with European and international organisations (EFSA, CEN, ISO, ...) and Third Countries

Objectives: Scientific support to European and international organisations when relevant.

Description:

Comments to EFSA on LOQs, standards and methods at the stage of the draft reasoned opinion.

Input will be provided on achievable LOQs, availability of standards and comments to proposed residue definitions in connection with approval of Draft Reasoned Opinion (art. 12 reviews) and other matters.

Expected Output: Various mail and brief report

Duration: 2021-22

Sub-activity 3.03 Participation in symposiums, workshops and seminars for the dissemination of scientific information.

Objectives: To promote EU and the EURLs(-CF), disseminate information on activities and scientific results as well as to upgrade the knowledge and skill of the EURL staff in order to support the Commission with the latest knowledge.

Description: Participation in the International workshop and symposiums by the staff of the EURL-CF

10th International Workshop on Proficiency Testing, UK, October 2022 (1 person)

14th European Pesticide Residue Workshop 2022 (2 persons)

Other workshops or symposiums could be relevant

Expected Output: Pdf file of oral and/or poster presentations

Duration: 2021

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REAGENTS AND REFERENCE COLLECTIONS

Please, provided activities related to Regulation (EU) 2017/625:
(Number of Sub-activity boxes can be adjusted by EURL)

- **Art. 94.2.j** ***Coordinating or performing tests for the verification of the quality of reagents and lots of reagents used for the diagnosis of foodborne, zoonotic or animal diseases and pests of plants.***

- **Art. 94.2.k** ***Where relevant for their area of competence, establishing and maintaining:***
 - i. reference collections of pests of plants and/or reference strains of pathogenic agents;***
 - ii. reference collections of materials intended to come into contact with food used to calibrate analytical equipment and provide samples thereof to national reference laboratories;***
 - iii. up-to-date lists of available reference substances and reagents and of manufacturers and suppliers of such substances and reagents.***

Sub-activity 4.1 (*name of Sub-activity*)

Objectives:
Description:
Expected Output:
Duration:

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REQUIREMENTS RELATED TO OTHER LEGISLATION

Please specify applicable legislation:
(Number of Sub-activity boxes can be adjusted)

Sub-activity 5.1 (*name of Sub-activity*)

Objectives: Description: Expected Output: Duration:

REMARKS

(if necessary)
