

WORK PROGRAMME of EURL for
PESTICIDE RESIDUES IN
FRUITS AND VEGETABLES

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INTRODUCTION

EU Reference Laboratories (EURLs) aim to ensure high-quality, uniform testing in the EU and support Commission activities on risk management and risk assessment in the area of laboratory analysis.

Regulation (EC) No 625/2017 on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products, defines tasks and responsibilities for all the EURLs. Some of them are: to provide National Reference Laboratories (NRLs) with analytical methods and diagnostic techniques, and coordinate their application; to train staff from NRLs; to provide the Commission with scientific and technical expertise in relation to laboratory analysis and to collaborate with the competent laboratories in non-EU countries. Based on the aforementioned, some of the specific activities of the EURL for pesticide residues in fruits and vegetables are the organisation of proficiency tests, the coordination and edition of the Analytical Quality Control guidelines or the assistance to the Commission and EFSA for Art. 12 MRL reviews.

Every year the EURLs submit their work programmes demonstrating their contribution to the Commission's objectives and priorities and request annual EU funding to fulfil their tasks and functions to cover their operational costs.

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 1.1 Updating the EURL website and the EURL DataPool

Objectives: To update and maintain the EURL-FV webpage, as well as contribute to the design and contents of the EURL DataPool (in cooperation with the other EURLs).

Description: The dedicated webpage "EURL for Fruits and Vegetables": <http://www.eurl-pesticides.eu/docs/public/home.asp?LabID=500&Lang=EN> located at the EURLs common website (<http://www.eurl-pesticides.eu>), designed to support dissemination of information and network activities, is continuously updated. It represents the main source of information exchange between the EURLs and the NRLs as well as with other official EU and third countries laboratories. The EURL-FV website holds information about the activities and events

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carried out by the EURL-FV as well as available published reports and scientific papers. It also holds forms, sheets and other documents ready to fill out on-line, thus facilitating management tasks and quality monitoring as well as direct links to other relevant websites. Constant collaboration between the EURL-FV and the EURL website management is necessary.

Furthermore, the website aids contacts (via specific links) between laboratory researchers and experts providing a valuable tool for dissemination. The website includes different sections, corresponding to the activities of the EURL: Proficiency Tests, Workshops, Services, The EURL-FV Network, AQC Panel and Library.

In 2021 a new online tool will be developed by the EURL-FV with the aim to improve dissemination of scientific knowledge. An **e-learning platform** will facilitate access to new analytical advances, new equipment, and new methods, but also to basic procedures within the analytical process, such as pipetting or estimation of uncertainty. The online platform will contain multimedia content about the most interesting analytical methods proposed in the scientific activities of the working programme, description and handling of lab equipment, sample treatment, etc. This will enable all personnel of the NRLs and OfLs to have full access to online resources of diverse complexity, which can be accessed using the internet anytime and anyplace. Users of this online tool will be able to make any question through the system, and it will be answered by the EURL-FV personnel in a short time. The e-learning platform will be certified in accordance with the ISO-9001:2015 standards.

Expected Output:

- Forms and other information to conduct the 2021 and 2022 EUPTs will be uploaded onto EUPT-FV area.

- Information and main presentations of the webinars to be organized in 2021 and 2022 (see sub-activity 2.5)

- EURL-Webinars will be uploaded onto EUPT-FV area.

[http://www.eurl-](http://www.eurl-pesticides.eu/docs/public/tmpl_article.asp?LabID=500&CntID=933&Theme_ID=1&Pdf=False&Lang=EN)

[pesticides.eu/docs/public/tmpl_article.asp?LabID=500&CntID=933&Theme_ID=1&Pdf=False&Lang=EN](http://www.eurl-pesticides.eu/docs/public/tmpl_article.asp?LabID=500&CntID=933&Theme_ID=1&Pdf=False&Lang=EN)

- Access to the AQC Panel topic in the main EURL website and in our specific area will allow laboratories to consult the “Analytical quality control and validation procedures for pesticide residues analysis in food and feed.” (SANTE/12682/2019). The site will allow constant feedback from the laboratories, so it will be useful in collecting information or suggestions from laboratories on the future revisions of the document. In 202, the new edition of the AQC document will be uploaded to the webpage.

- The results of the scientific activities developed by the EURL-FV will be published as technical or scientific documents, and the most relevant will be disseminated in the EURL-FV website through the Library section making them available for NRLs, OfLs and members of the scientific community.

- E-learning platform where video tutorials and other online tools will be uploaded. All participants will receive an ISO 9001 certificate of attendance.

Duration: Throughout the year

Sub-activity 1.2 *Follow up on requests from NRLs for providing analytical standards*

Objectives: To supply analytical standards to the NRLs under request.

Description: In order to promote the enlargement of the NRLs’ analytical scope and to offer them the possibility to verify their standard solutions, we will provide them with the analytical standards that they request.

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Expected Output: With the publication of EUPT-FV23, FV24, EUPT-SC05 and SC06 target lists (mandatory and voluntary), we expect to receive requests from NRLs to send them analytical standards of those pesticides newly included in the lists of possible pesticides. Furthermore, during the year, for example, with the publication of the coordinated multiannual control programme and the working documents 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, we will provide them with the requested substances.

Duration: Throughout the year

Sub-activity 1.3 *Organisation of proficiency tests and follow up on the results*

Objectives: To organise proficiency tests simulating, as far as possible, the real sample conditions and to follow up on the results obtained by the NRLs and OfLs, emphasizing on unacceptable results.

Description: The European Proficiency Test on fruits and vegetables EUPT-FV23 and FV24, in accordance with previous schemes and statements, will be open to all OfLs, especially the NRLs of EU Member States. Additionally, laboratories from EFTA countries and other third countries will be invited to participate, so quality assurance can reach them on the basis of the proficiency test. These countries might be invited to take part after *Health and Food Audit and Analysis* recommendation and by request of DG SANTE.

These EUPTs will be carried out in a way which simulates, as far as possible, the real sample conditions that arrive at a laboratory in its routine work such as: the use of commercial formulations for pesticide treatment; homogeneity of intra-samples and the consideration of all classes/types of compounds.

The commodity used for the test material of EUPT-FV23 will be aubergine, and tomato in the case of EUPT-FV24. The test material will contain incurred pesticides. The whole organisation of the EUPT will be very similar to that of previous EUPTs performed by the EURL-FV.

Additionally, other intercomparative studies using screening methods (EUPT-SM13 and SM14) will be organized in parallel to EUPT-FV23 and FV24, with the intention to promote the rapid screening of a large number of pesticide residues in the EU control laboratories over a very short period of time (72 h). In this way, the scope of the methods in screening mode could reach 500-700 compounds in a rapid inexpensive way. This information supports OfLs in checking their performance in these situations. It allows the EURL to identify the large scope laboratories ("scouting laboratories"). This activity is well accepted by OfLs as can be confirmed by the increasing participation (approx 60 EU OfLs) in previous rounds.

Participation in the screening PT remains on a voluntary basis; nevertheless, all NRLs and OfLs involved in the determination of pesticide residues in fruit and vegetables for the EU-coordinated monitoring programme, or for their own national programmes and third countries will be invited to take part.

A third type of proficiency test will be organised in order to offer the NRLs and OfLs the possibility to test their methods with special commodities such as baby food, herbs, spices, etc. and evaluate their performance with regard to those commodities (EUPT-SC04 and SC05). Participation in this PT remains on a voluntary basis.

These Proficiency Tests will be based on the Quality Control Norm ISO/IEC 17043: Conformity assessment - General requirements for proficiency testing.

Once a year, the EURL-FV will organise a meeting of the EUPT-Panel (EURLs + EUPTs Scientific Committee) to discuss the evaluation of the EUPT results and to decide about the following years' EUPTs.

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Expected Output: Six proficiency tests will be organised during 2021 and 2022: EUPT-FV23, EUPT-FV24, EUPT-SM13, EUPT-SM14, EUPT-SC05 and EUPT-SC06. Their final reports will be uploaded to the EURL-FV webpage.

Duration: Throughout the year

Sub-activity 1.4 *Cooperation and meetings with other EURLs*

Objectives: To maintain a smooth channel of communication between the EURLs for pesticide residues.

Description: Constant collaboration with the other pesticide residue EURLs will be maintained for general management activities and other specific tasks. Additionally, every year the four EURLs will meet in order to discuss specific issues like the EURLs webpage, EUPTs or joint workshops. Inter-EURL-meetings in some cases in presence of DG SANTE representatives will be carried out with the aim to discuss, plan, coordinate or evaluate EURL-activities such as the preparation of work programs, EUPTs or web-applications. In certain cases, online-meetings or tele-conferences will be carried out.

Expected Output: The four EURLs for pesticide residues will meet, at least, for the AQC expert meeting, the EUPT Panel meeting and the Joint EURL/NRLs Workshop, in 2021. In 2022 they will meet during the EUPT Panel meeting and at the EPRW 2022.

Duration: Throughout the year

Sub-activity 1.5 *Development and validation of analytical methods: Troubleshooting in GC-MS/MS*

Objectives: The general objective is to offer NRLs and OfLs useful tools to facilitate and improve the workflow of the laboratory and quality of the results. Two specific objectives within this activity would be: to assess the use of acetonitrile in combination with other solvents in the analysis of samples by gas chromatography; and to evaluate and compare the features of several split/splitless liners offered by specialized firms.

Description: *Evaluation of mixed injection solvents in gas chromatography as a measure to avoid solvent change during sample treatment.* The most commonly used extraction methods for routine analysis use the solvent acetonitrile. This entails the need of an addition step during the sample treatment prior to GC analysis: a solvent change. Acetonitrile is not the preferred solvent for GC analyses, as it can damage the chromatographic columns as a consequence of its high expansion volume, and thus make it necessary to perform maintenance tasks frequently. The use of analyte protectants to avoid this task has not been very satisfactory for many laboratories. The EURL-FV will assess the combination of solvents, together with different injection modes including dual layer injection and/or solvent vent) as a way to:

- 1) Avoid the need of evaporating samples, thus reducing the loss of volatile analytes and reducing the time needed for sample preparation.
- 2) Maximize the durability of the instruments between maintenance tasks.

Description: *Testing of different commercially available liners for the injection in gas chromatography.* With the exception of the on-column injection, liners play an essential role in the injection of samples for gas chromatography analyses. The different variants can be purchased from various companies,

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but there can be differences in their performance. The EURL-FV will compare a variety of the most commonly used split-splitless liners in terms of inertia, durability and general performance for the analysis of pesticides at various concentration levels in different vegetable matrices.

Expected Output: 2 Technical reports and/or scientific publications.

Duration: 4 months

Sub-activity 1.6 Development and validation of analytical methods: Troubleshooting in LC-MS/MS

Objectives: The general objective is to offer NRLs and OfLs useful tools to facilitate and improve the workflow of the laboratory. Two specific objectives within this activity would be: to develop a method for the preservation in the injection vials of analytes known to suffer from degradation and/or transformation and allow their correct detection and quantitation in multiresidue methods; and to assess the cost-benefit of not applying clean-up and evaluate the negative effect of clean-up sorbents.

Description: *Use of antioxidants to prevent degradation of labile analytes prior to LC-MS analysis.*

Degradation of target analytes in multiresidue analysis can take place at different steps throughout sample reception and analysis: during sample processing, sample extraction, and even sample analysis in certain cases. In LC-MS, standard procedure in many laboratories is to dilute the organic extracts with water prior to their injection in the chromatographic system, and these extracts can be queued for hours before being analysed. While being queued and in this organic solvent-water mixture, certain analytes can suffer from various degradation processes which significantly reduce their analytical response- even below the detection limit of the analytical instrument. Some target analytes known to suffer from degradation under these conditions include cymoxanil, dazomet, and formetanate, for instance.

A feasible strategy to overcome this issue is to include an antioxidant to the water to be employed for the vial preparation, such as ascorbic acid or similar. In cases where the compounds are appropriately stabilised, their analysis can be performed as usual, and in cases where in-vial degradation is inevitable, it is expected that it can be decelerated enough to allow the analyst to detect it and take the appropriate measures for its correct quantitation, such as preparing the injection vial right at the moment of injection and after the external calibration curve.

Description: *Assesment of the cost-benefit of not applying clean-up and evaluation of the negative effect of clean-up sorbents on the analytes.*

With the appearance of new and more sensitive instrumentation, the clean-up step of multiresidue methods might be obviated, reducing the costs and time of analysis, and avoiding loses of specific compounds. For example, PSA should be avoided with some pesticides of baby food directive, such as haloxyfop, and it can also produce degradation of some other pesticides like dichlofluanid or tolylfluanid. The EURL-FV will carry out a detailed evaluation of the negative effects of the different sorbents used during clean-up, always comparing with the possibility of not using clean-up, and trying to assess this way the cost-benefit of not cleaning the extracts.

Expected Output: 2 Technical reports and/or scientific publications.

Duration: 4 months

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Sub-activity 1.7 *Development and validation of analytical methods: New developments for the automatisisation of critical steps of the analytical process*

Objectives: The general objective is to offer NRLs and OfLs useful tools to facilitate and improve the workflow of the laboratory and reproducibility of the results, that affect the possibility to enlarge the scope under Iso 17025, in this case by automatisation of diverse steps in the analytical process, which will derive in an improvement of the quality control. Within this general purpose, the main specific objective would be to automate the extraction method for high water content commodities, with the increase sample throughput.

Description: *Development and validation of an automated extraction method for the analysis of high-water content commodities.*

Automated extraction instruments are garnering increasing interest from laboratories due to their increased robustness and reproducibility compared to manual sample treatment methodologies. However, currently their use is limited to specific commodities or target analytes, and traditional solid-liquid extraction techniques such as QuEChERS are still the most commonly employed sample extraction approaches. In this regard, new instruments based on pressurized liquid extraction allow the extraction of traditionally difficult commodities without requiring costly clean-up steps. One such example is the development and validation of a routine MRM method for high fat content commodities of plant origin by the EURL-FV.

With the gained experience, an extraction method for high-water content commodities based on automated pressurized liquid extraction will be developed. This method will allow for faster sample throughput and decreased or unnecessary clean-up steps. The method will also be completely validated in high water content commodities to ease its introduction in routine laboratories. The main advantages for its implementation in routine laboratories are (i) significantly increased sample throughput, at about 7 min per sample in the worst scenario -and potentially down to 5 min per sample depending on the commodity-, up to 70 to 96 samples processed in 8 h; (ii) serial sample treatment, as opposed to parallel sample treatment of typical extraction methods, where n samples are processed concurrently and only at the end of the process can all be analysed, which means a batch analysis can begin just minutes after the first sample has been extracted; (iii) ease of implementation under ISO/IEC 17025 standard; and (iv) reduced or eliminated need for expensive clean-up steps, as demonstrated by our group in the case of tea, cocoa and coffee, where no hydration or clean-up were required for the developed multiresidue method (technical report 2019-M34 and DOI 10.1039/D0AY01962C).

Expected Output: 1 Technical report and/or scientific publication.

Duration: 4 months

Sub-activity 1.8 *Development and validation of analytical methods: Advantages and disadvantages of Liquid chromatography vs supercritical fluid chromatography.*

Objectives: To provide information that helps laboratories determine which chromatography is more efficient for each pesticide and matrices group.

Description: Supercritical Fluid Chromatography offers a dramatical reduction of solvent usage compared to a typical LC instrument, reducing waste and economical cost, in a manner that complies with the principles of green chemistry.

Numerous studies have been published evaluating different parameters of reverse-phase liquid chromatography or supercritical fluid chromatography both coupled to mass spectrometry for pesticide residue analysis, and it has created a great interest in laboratories for further evaluation in

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routine. The EURL-FV will evaluate both types of chromatography for multiresidue analytes, but using the same mass spectrometer platform, being the main difference between them the mobile phase gradient pumped across the system. The capability to work at low temperature in the ion source facilitates the analysis of thermolabile compounds. Through the injection of the same vials, separate experiments will be conducted using both chromatographies. The pesticides will be compared in terms of sensitivity in pure solvent and in a variety of matrices. The compounds that exposed the highest sensitivity in each chromatography will be evaluated through their substance groups and polarity values. In addition, how matrix effects interact with analytes in each technique will be studied. The results of this study could help to choose the type of chromatography coupled to mass spectrometry who fits better with the scope and the complexity of matrices to be analyzed.

Expected Output: 1 Technical report and/or scientific publication.

Duration: 4 months

Sub-activity 1.9 Development and validation of analytical methods: Validation of the main MRM extraction methods in dry legumes.

Objectives: To validate analytical methods for the analysis of pesticide residues in representative dry legumes.

Description: Dried beans are one of the products of plant origin to be sampled in 2023 according to the EU coordinated multiannual control programme for 2021, 2022 and 2023, but there is little information about the best methodology of analysis. Dry legumes belong to the *High starch and/or protein and low water and fat content* commodity group of the EU AQC Guidelines. In order to give support to the NRLs and OfLs with their analysis, the EURL-FV will validate the three main multiresidue (MRM) extraction methods (QuEChERS, SweEt and NL-miniLuke) for the analysis of dry legumes by both gas and liquid chromatography coupled to tandem mass spectrometry (GC-MS/MS and LC-MS/MS). The scope of the methods will cover at least those MRM amenable pesticides included in the MACP and working document.

Expected Output: 1 Technical report and/or scientific publication.

Duration: 4 months

Sub-activity 1.10 Development and validation of analytical methods: Impact of the different calibration approaches in the quality of the results.

Objectives: To evaluate different ways of calibration for quantification, in order to evaluate the impact that they have in the quality of the results.

Description: In document SANTE/12682/2019 for quality control, different approaches for quantification are described: multiple- or single-level calibration, procedural calibration, standard addition or internal standard calibration, among others. All of them are acceptable, as far as they are fit for purpose. However, in multiresidue analysis, it's a reality that in some cases labs have to take a compromise solution that tries to satisfy both, the quality of the results and the workload of the laboratory. The use of one or other way of calibration for quantification might imply the introduction

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of errors in the analysis. The EURL-FV will assess the influence that different ways of calibration have in the quality of the results.

Expected Output: 1 Technical report and/or scientific publication.

Duration: 4 months

Sub-activity 1.11 Development and validation of analytical methods: Evaluation of the three main multiresidue methods for the validation of new substances of SANCO/12745/2013 and those with low analytical coverage.

Objectives: To validate new compounds included in Working Document SANCO/12745/2013 and those with a low analytical coverage, by the three main multiresidue methods.

Description: The three main multiresidue extraction methods used in the EU (QuEChERS, new-Luke and SweEt) will be evaluated in three commodity groups (high water content, high water and acid content and high fat content) at low concentration levels in order to support the EU NRLs/OfLs in the enlargement of their analytical scope, especially with those pesticides newly included in Chapter 4 of the working document SANCO/12745/2013 (cyflumetofen, diuron, forchlorfenuron, flupyradifuron and matrine), those with poor or medium analytical coverage (4-CPA, aclonifen, benzovindiflupyr, bifenazate, chlorfluazuron, clomazone, cyantraniliprole, diafenthiuron, dinotefuran, fenobucarb, fencicoxamid, florpypauxyfen benzyl, flutianil, fluxapyroxad, isofetamid, isopyrazam, mefentrifluconazole, mercury compounds, novaluron, oxathiapiprolin, penflufen, penthiopyrad, pyrethrins, pyriofenone, quintozone, rotenone, tolfenpyrad, triflumizole and trinexapac, plus those included in the 2021 revision), and those MRM amenable included in Annex II of the working document. The new compounds will be validated at least at two concentration levels, being the lowest one at least 5 µg/kg. The validation report will include all the information about validation parameters, transitions, ion ratios, etc.

Expected Output: 1 Technical report and/or scientific publication.

Duration: 4 months

Sub-activity 1.12 Development and validation of analytical methods: Optimisation of the preparation of working solutions of analytical standards and impact of the cocktail effects.

Objectives: To evaluate the cocktail effect produced by the mixture of compounds in the analytical working solutions, which might affect the stability of the analytes in the mixes.

Description: The preparation of mixes of analytical standards (stock solutions) is a crucial step that may have a great impact on pesticide residue quantitation in samples. Typically, laboratories prepare pesticide mixes at high concentrations (10 - 100 mg/L) containing a significant number of pesticides (10-100), which are kept and employed for a long period of time (6 - 12 months). These mixes are afterwards combined into working mixes of lower concentrations (0.5 - 5 mg/L) with a shorter expiration date (approx. 1 week). The physicochemical nature of the pesticides included in the high concentration mixes is often overlooked, besides some standard measures such as avoiding the combination of metabolites and precursor molecules within the same mix.

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The effect of combining pesticides of similar and different physicochemical properties (fundamentally, pKa) will be checked throughout a year after their preparation. With this, it will be possible to determine whether there is a correlation and causation between high concentration mixes shelf life and the physicochemical properties of the pesticides within the mix. This information will be helpful to the laboratories in the preparation of their standard solutions.

Expected Output: 1 Technical report and/or scientific publication.

Duration: 14 months

Sub-activity 1.13 Development and validation of analytical methods: Guidance for overcoming difficulties in the quantification of complex residue definitions containing isomers

Objectives: To achieve a correct quantification of spinosad, spinetoram or similar cases (standards containing component mixes) by the analytical laboratories.

Description: In 2020, one of the activities of the EURL-FV working programme was the evaluation of analytical signals of individual isomers to verify the most appropriate quantification mode. The most common approach laboratories employ is using the mixture of isomers instead of the individual components. It was proven that, in some cases -such as for spinosad-, it is necessary to quantitate spinosyn A and spinosyn D separately, either by the use of individual standards, or by the correct application of their respective purities within the spinosad standard mixture.

During the EUPT-FV23 proficiency test, spinosad was applied to the test item. The evaluation of the results demonstrated bimodality for this compound as a consequence of the different approaches laboratories followed for its quantification. A survey answered by the participants showed that 46 % of the respondents were not appropriately quantitating Spinosad. Furthermore, after the complete evaluation of the results with the newfound information from the survey, the high dispersion of spinosyn D results might be explained by its low proportion in some of the technical mixtures of spinosad. An alternative, more worrisome cause for this dispersion might be the inaccuracy of the certified ratio of spinosyn A and spinosyn D by the standard suppliers. The goal of the EURL-FV is to investigate the latter possibility by purchasing spinosad analytical standards (mixture of spinosyn A and D) from different certified standard providers, after which they will be employed to quantitate PT samples.

Similar experiments will be done for spinetoram and analogous compounds.

Expected Output: 1 Technical report and/or scientific publication.

Duration: 4 months

Sub-activity 1.14 Development and validation of analytical methods: Automatisation of the clean-up step of multiresidue methods in GC-MS.

Objectives: To evaluate the automatization of mini-SPE clean-up cartridges for the typically dirty extracts from the QuEChERS sample extraction process to achieve ease-of-use and extended sample throughput with less maintenance.

Description: The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method is the most employed by the EU laboratories. It involves two steps: sample extraction, and clean-up of the extracts in order to remove undesired matrix compounds that may cause analytical interferences. Currently, many labs possess very sensitive instruments that allow them to skip the clean-up step in the case of

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LC-MS/MS analysis. However, GC-MS/MS systems are more affected by certain matrix components like a wax, fat, and proteins. For such matrices, the clean-up step is critical due to the impact on the glass liner that can affect the correct quantification of the target compounds. Therefore, developing an automated procedure for this clean-up step is expected to be of considerable interest for routine laboratories in terms of time saving and quality improvement of the results. A novel solution to automate the clean-up step of complex matrixes of QuEChERS extracts has been developed. The EURL-FV will evaluate the application of the automated mini-SPE sample handling system, which employs clean-up cartridges to run online sample clean-up and injection into gas chromatography instrument. The main advantage routine laboratories could expect from the mini-SPE cleaning method would be the increased sample throughput and reduced instrumental maintenance. While the sample sequence is running, the prep-ahead function of the system begins the preparation of the next sample during the current chromatographic run, which saves sample preparation time.

Expected Output: 1 Technical report and/or scientific publication.

Duration: 4 months

Sub-activity 1.15 Development and validation of analytical methods: Studies of the main degradation processes of pesticides in commodities during the milling/extraction steps.

Objectives: To evaluate the ideal approach to sample milling that reduces or eliminates pesticide degradation.

Description A specific, troublesome step that might cause laboratories to report false positives/negatives or an incorrect quantitation is the transformation of compounds during the milling and/or extraction steps. This is often consequence of the liberation of cellular content, fundamentally enzymes, which can be the source of analyte transformation and/or degradation. Such effects are commonly unpredictable and happen at different rates, depending on the commodity. With this study, we will provide enough information to avoid this difficulty for the main combinations of pesticide-commodity (for example, thiodicarb degradation to methomyl in onion).

Expected Output: 1 Technical report and/or scientific publication.

Duration: 4 months

Sub-activity 1.16 Development and validation of analytical methods: HRMS mix mode approach.

Objectives: To develop a working mode in High Resolution Mass Spectrometry (HRMS) that can be easily applicable in routine analysis.

Description: HRMS offers different ways of data acquisition. Generally, screening employing acquisition modes like All Ion Fragmentation (AIF) is the preferred approach by laboratories using this instrumentation. However, with the new development of high resolution technology, the combination of various acquisition modes has become feasible. One such example is the concurrent target and non-target data acquisition, which might be the ideal alternative for laboratories looking to implement high resolution analyses in their scope. The low sensitivity normally associated to HRMS instrumentation (one of the reasons why this new instrumentation has currently a limited application in routine laboratories) is avoided for analytes that require a more sensitive, target data acquisition and are not amenable for non-target data modes. Thus, this new data acquisition strategy may be implemented in

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routine laboratories, enlarging the scope of analyses they can perform and avoiding the reporting of false negatives.

Expected Output: 1 Technical report and/or scientific publication.

Duration: 4 months

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 2.1 *Providing technical and scientific support to NRLs*

Objectives: To support the NRLs in the development of their analytical methods and the enlargement of their scope of analysis.

Description: The results of the scientific activities developed by the EURL-FV will be published as technical or scientific documents, depending on the impact of the activities. They will be disseminated in the EURL-FV website (www.eurl-pesticides.eu), through the “Library” section, making them available for OfLs and members of the scientific community. The main EURL-FV contributions to international conferences will also be uploaded to the EURL-FV website.

Additional assistance to the NRLs will be supported by constant communication via e-mail and telephone.

The new e-learning platform will enable the NRLs to have full access to online resources of diverse complexity, which can be accessed using the internet anytime and anyplace. The more relevant scientific activities of the working programme will be included in the e-learning platform.

Expected Output: In 2021 and 2022 at least 14 new technical reports and/or scientific papers will be published on the website. At least four of the sub-activities 1.5 to 1.16 will be presented in a multimedia way in the e-learning platform.

Duration: Throughout the year.

Sub-activity 2.2 *Organisation of workshops*

Objectives: To organise a workshop with the NRLs to act as a platform for information exchange.

Description: In 2021, the annual EURL/NRLs-FV workshop will be celebrated together with the other EURLs for pesticide residues, in Almería, Spain, and organised by EURL-FV (October 2021). It will consist of technical and scientific communications and round tables. Extensive interaction with all NRLs that will attend will be the main objective. Attention will also be paid to the evaluation of the EUPT results

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and their relation with the various analytical methods applied by the NRLs and OfLs establishing actions for improvement.

NRLs representatives from all the EU Member States will participate in the workshops.

A similar workshop will be organised in Fall 2022, but in this case together with the EURL-SRM, considering the networks of NRLs-FV and NRLs-SRM.

Expected Output: Report on Workshop, pdf of presentations on website, Evaluation forms (satisfaction index of participants and their comments).

Duration: 2 months

Sub-activity 2.3 Organisation of training courses

Objectives: To organise a training course for staff from national reference laboratories in order to provide them with scientific and technical assistance.

Description: The EURL-FV will support the NRLs with technical “lab activities”. This technical assistance will consist on the selection of a limited group of NRLs (approx. eight) to develop technical training of 1-2 days duration at the EURL-FV laboratory (Almería, Spain). The topic of the training courses will be decided at a later stage, in consultation with DG SANTE, if necessary. The trainings will be celebrated once a year.

Expected Output: 1 training in 2021 and 1 training in 2022. Training material (presentations, excel files) on website, Evaluation forms (satisfaction index of participants and their comments).

Duration: 2 months

Sub-activity 2.4 Visits to NRLs

Objectives: On the spot visits to the NRLs in order to give them technical and scientific support.

Description: Each year, the EURL-FV will visit one NRL with deficits in the areas of EUPT-performance, analytical scope or country network of OfLs. The NRLs to be visited will be selected based on the results of the different EUPTs-FV, and will be specified at a later stage in consultation with DG SANTE.

Expected Output: Mission Reports 2021+2022 and follow-up reports.

Duration: 2 weeks

Sub-activity 2.5 Organisation of webinars

Objectives: To disseminate scientific and technical results in an interactive way.

Description: The EURL-FV will conduct webinars with the aim to disseminate technical information to the NRLs and OfLs in a cost effective but still interactive way. These webinars will be coordinated by the EURL-FV and will be especially focused on dissemination of EUPT results and the main analytical methods developed.

During 2021 and 2022 the EURL-FV will organize at least six webinars, being the main relevant topics, those activities related to the Work Programme 2021-2022, such as the results of the EUPT-FV23/FV24, EUPT-FV-SM13/14, EUPT-FV-SC05/SC06.

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Expected Output: Webinar presentations uploaded to the EURL-FV webpage.

Duration: 2 weeks

Sub-activity 2.6 Updating and publication of the list of NRLs

Objectives: To update the network of NRLs and OfLs.

Description: The network of NRLs and OfLs is constantly changing, and for this reason it is necessary to keep it updated. Every year before the participation in EUPT-FV, the EURL-FV contacts the NRLs in order to obtain the detailed list of OfLs. In parallel, the EURL DataPool also gathers information about possible changes in the list.

Expected Output: Updated list of NRLs published in the EURL-FV website.

Duration: 1 week

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

Objectives: To give technical and scientific support to the Commission when requested.

Description: This horizontal task with the four EURLs and coordinated by EURL-SRM and EURL-CF will give scientific support to the Commission as regards complex residue definitions or other analytical parameters such as LOQs for Art. 12 of Regulation (EC) No 396/2005 proposals, new substances and other substances.

Expected Output: In 2021 and 2022 this activity will continue as in previous years.

Duration: Throughout the year

Sub-activity 3.2 *Assistance to COM for the EU MACP and the monitoring working document*

Objectives: To give technical and scientific support to the Commission in the drafting of the EU MACP.

Description: Assistance to the European Commission will continue regarding the selection of the number of analyses, commodities and pesticide lists to be monitored by the Member States in the coordinated multiannual control programmes of the Union published in 2022. This assistance will also be related to the update of the list of pesticides included in the monitoring working document.

Expected Output: To contribute to the new versions of the MACP and monitoring working documents.

Duration: 2 weeks

Sub-activity 3.3 *Contribution to the revision of the analytical quality control guidelines*

Objectives: Update and edition of EU Guidelines on Quality Control Procedures.

Description: In order to continue the process of achieving complete harmonisation measures for pesticide residue analysis within the EU, the SANTE document “Analytical quality control and method validation procedures for pesticide residues analysis in food and feed” (SANTE/11813/2017) needs to be revised and updated on continuous basis.

Therefore, the aim is to carry on with the specific forum (AQC Panel) on the EURL-FV website to facilitate the discussion and to point out difficulties and improvements on the EU AQC Guidelines. This network will provide interaction among EURLs-NRLs-OfLs. The outcome of the discussion in this specific forum will improve and facilitate further updated revisions of the EU AQC Guidelines, to be presented in the joint workshop every two years.

Expected Output:

The AQC Scientific Committee together with the four EURLs for pesticide residues will meet three times in 2021 in order to discuss about the possible changes and modifications to the SANTE Guidelines. The three meetings will be organised by EURL-FV. The second one will be in combination with the EUPTs Scientific Committee expert meeting. The third AQC meeting of 2021 will take place in Almería, prior to the Joint workshop, where the preliminary document will be produced for the final voting of the NRLs. If it succeeds, after approval by the COM, the new version of the document will be edited at the end of the year 2021.

In 2022, the AQC meeting will meet once, in combination with the EUPTs Scientific Committee expert meeting.

Duration: 1 month

Sub-activity 3.4 *General technical support to the Commission*

Objectives: To provide technical and scientific support to the Commission when requested

Description: Technical and scientific support to the Commission will be provided when requested. Constant communication will be established via e-mail, phone calls or meetings. Whenever the need arises, technical advice will be provided to the DG SANTE upon request.

Expected Output: Attendance to the Standing Committee (PAFF) meetings at request of the DG SANTE and assistance to the audit team of the department *Health and Food Audits and Analysis* if they so request it, by accompanying the inspectors in the audit visits giving technical support as a “national expert”.

Duration: Throughout the year

Sub-activity 3.5 *Collaboration with European and international organisations (EFSA, CEN, ISO, ...)* *Comments to EFSA on LOQs, standards and methods at the stage of the draft reasoned opinion.*

Objectives: To provide scientific support to EFSA.

Description: Involvement in the EFSA residue evaluation process by giving opinions and advice, especially regarding residue definition and post registration analytical methods. In the case of new

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substances, it is estimated to carry out experimental analytical work if requested by the DG SANTE. This is a horizontal task with the four EURLs and coordinated by EURL-SRM and EURL-CF.

Expected Output: In 2021 and 2022 this activity will continue as in previous years.

Duration: Throughout the year

Sub-activity 3.6 Collaboration with European and international organisations (EFSA, CEN, ISO, ...) Participation in the EFSA networking group on pesticides residues monitoring

Objectives: To provide technical and scientific support to EFSA in the Network of Chemical Monitoring Data Collection.

Description: The EURL-FV yearly collaborates with EFSA with the attendance to the meetings of the Network of Chemical Monitoring Data Collection, with presence of the Member States, the EFTA countries, the European Commission and EFSA. The technical and scientific assistance includes all matters related to pesticide residues monitoring covered by Regulation (EC) No 396/2005, including the preparation of the EFSA Annual Reports on Pesticide Residues and the review of the EFSA standardised data model for reporting the monitoring results.

Expected Output: In 2021 and 2022, one representative from the EURL-FV will attend the meeting of the networking group.

Duration: 1 week

Sub-activity 3.7 Collaboration with European and international organisations (EFSA, CEN, ISO, ...) Participation in the meetings of the CEN/TC 275/WG 3 Working group Pesticides.

Objectives: To participate in the development of standardised methods (CEN methods)

Description: Since 2015, the EURL-FV participates in the Working group 3 (Pesticides, CEN/TC 275/WG 3) dedicated to the standardization of methods for the determination of pesticide residues in food. Furthermore, the EURL-FV is directly involved in the modular QuEChERS, being the project leader of that CEN method.

Expected Output: Attendance to one meeting per year in the German institute for standardization, DIN (Berlin, Germany).

Duration: 2 weeks

Sub-activity 3.8 Collaboration with Third Countries.

Objectives: To promote the international networking and dissemination of information and activities from the EURL-FV, especially in countries with intensive European export-import relationships.

Description: This assistance will be supported by, at least, constant communication via e-mail and telephone. Selected third countries will be invited to participate in the workshops and training courses as well as to visit the laboratories in relevant cases. Important information for selection of laboratories

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to participate in EUPT will come from the *Health and Food Audits and Analysis* section as a consequence of their inspections.

Laboratories from EFTA countries and other third countries will be invited to participate in the EUPTs-FV. These countries might be invited to take part after *Health and Food Audit and Analysis* recommendation and by request of DG SANTE.

Expected Output: E-mail exchange with third countries laboratories, invitation to 2021 and 2022 EUPTs-FV and workshop.

Duration: Throughout the year

Sub-activity 3.9 (*Participation in symposiums, workshops and seminars for the dissemination of scientific information.*)

Objectives: To disseminate the EURL-FV activities to the scientific community.

Description: The most relevant results of the scientific activities developed by the EURL-FV will be presented as posters and/or oral presentations in international workshops.

Expected Output: Oral presentations by the EURL-FV of the scientific activities developed in the laboratory will be presented in 2021 in the Latin American Pesticide Residue Workshop (online, LAPRW 2021), in Eurachem's 10th Workshop on Proficiency Testing in Analytical Chemistry, Microbiology and Laboratory Medicine (Fall 2022, Windsor, UK) or in RAFA (Recent Advances in Food Analysis 2021, 2-5 November 2021, Prague, Czech Republic), among others.

Duration: Throughout the year