

EURL FOR PESTICIDE RESIDUES IN FRUITS AND VEGETABLES (EURL-FV)

Activity Programme 2015

FUNCTIONS AND DUTIES

The functions and duties of the European Commission Reference Laboratory are described in Article 32 of the EU Regulation No 882/2004.

These functions and duties together with the Additional Specifications are gathered together into four groups of activities (A, B, C and D) as follows:

- A. General tasks
- B. **Development and validation of analytical methods**
- C. Quality assurance and quality control programme; including the organisation of Proficiency Tests and Intercomparative Studies
- D. <u>Technical and scientific support to DG SANCO, EU Member States and Third Countries including the organisation of Courses and Workshops</u>

Acronyms

ADVG: Advisory Group

EFSA: European Food Safety Authority
EFTA: European Free Trade Association
EURL-FV/CF/AO/SRM: European Union
Reference Laboratory-Fruits and

Vegetables/Cereals and Feed stuff/ Food of Animal Origin/Single Residue Methods. **FVO:** Food and Veterinary Office

HRMS: High Resolution Mass Spectrometry

LOQ: Limit of quantification

MRM: Multiresidue Method

NRL: National Reference Laboratory

OfL: Official Laboratory
PT: Proficiency Test
QC: Quality Control

QCG: Quality Control Group



2015 Activity Programme.

The four groups of activities mentioned above are developed into different tasks distributed as follows:

A - GENERAL TASKS.

A1. Management of administrative duties

Follow-up tasks:

- Continue oversight by the EURL-FV responsible personnel, controlling and reporting upon all the activities of the work programme, ensuring that they are executed at the right time and in the correct way and their adequate dissemination in the NRL and OfLs network. In addition, the proper allocation of the budget is ensured by the EURL-FV-responsible personnel in collaboration with the corresponding administrative public officer from the University of Almeria.
- Identification of analytical difficulties or sources of errors in the OfL network for an optimized development of the activities B and C.

A2. EURL-FV web page.

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 carried out by the EURL-FV as well as available published reports and scientific papers (in the first semester of 2014 the FV website has received more than 12,000 visits). 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.

- Forms and related NRLs and OfLs documentation to conduct the 2015 EUPTs will be uploaded onto specific web pages designed by the EURL-FV and linked to the EUPT-FV area.
- Information and main presentations of the webinars to be organized in 2015 (see activity D6) will be included into the Workshop topic:

http://www.eurl-pesticides.eu/docs/public/tmplt_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 "Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed" (SANCO/12571/2013). 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. Another tool available about this topic is the Conversion factors e-learning, this e-learning site offers a very useful tool to aid familiarization with the conversion factor calculus: www.eupt.es/e-learning.
- 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 (www.eurl-pesticides.eu) through the Library topic making them available for OfLs and members of the scientific community (more than 1,200 entries were received in that section in the first semester of 2014). Last publications Link:

- A new development in 2015 will be the possibility to search for a specific pesticide in all the technical reports and the scientific publications in order to facilitate the use of the webpage.

A3. Development of bilateral cooperation with other organisations:

Information of mutual interest will be exchanged between the EURLs, EFSA and FVO, in addition to specific cooperation with NRLs, with a view to possible joint projects or to present relevant information/data with the express agreement of DG SANCO.

Follow-up activities:

- Special collaboration with EFSA by agreement with DG SANCO through Ms. Paula Medina Pastor, second national expert in EFSA and former member of the staff at EURL-FV.

New activities:

- -Cooperation with the IRMM-JRC (Institute for Reference Materials and Measurements-Joint Research Centre) in a study of the viability of preparing certified reference material.
- Collaboration with the Dutch Institute of Food Safety (RIKILT) in the study of the variability of ion ratios by GC-MS/MS.

A4. Collaboration with the other pesticide residue EURLs.

Constant collaboration with the other pesticide residue EURLs will be maintained for general management activities and other specific tasks.



B - DEVELOPMENT AND VALIDATION OF ANALYTICAL METHODS

B1. Development of validated procedures (including the use of new instrumentation)

Important EU OfLs challenges are related to three aspects:

- (i) The challenge of achieving adequate scope. This is evidenced by the EUPT-FV results where around 50% of OfLs typically are in category B (meaning they analyzed less than 90% of the target list).
- (ii) The challenge of improving instrumentation sensitivity as a consequence of difficulties buying highly updated instrumentation.
- (iii) Problems in analyzing "difficult" commodities because of the high number of coextractives obtained by the application of the current validated MRMs.

The development of the new procedures is focused on at least partial improvement of these three challenges.

List of methods link:

 $http://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=500\&CntID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=828\&Theme_ID=8$

B1.1 Update of the GC-Q-TOF-MS database of exact masses of pesticide fragments in El mode and development of a GC-Q-TOF-MS database in NCI mode. Validation of the corresponding screening methods.

GC-Q-TOF-MS is a high resolution technique based on accurate masses. During 2014 a database in electron impact mode was created for 110 pesticides with the exact masses of 1-5 fragments per pesticide. During 2015 the database will be updated with at least 50 new pesticides and their fragments in collaboration with the EURL-CF. The selection of the pesticides will be made in order to cover as many compounds as possible of the EU multi-annual control programme (chlorothalonil, chlorfenvinphos, flusilazone, Isofenphos-methyl, etc). Furthermore, a new database in negative chemical ionisation mode will be developed. The data will subsequently be uploaded onto the EURL database. Additionally, both databases will be used to develop and validate screening methods based on GC-HRMS, and the information obtained from those methods will be used for the update of the EU-QC-Guidelines.

B1.2 Validation of a LC-QTOF HRMS method (request of CEN standardised method).

Following the activity programme of the previous years, screening methods will be evaluated considering the introduction of new technologies (HRMS) in EU OfLs. The EURL-FV will validate a routine HRMS method using new and highly sensitive QTOF technology. The main MRMs used by the EU-OfLs (QuEChERS, SweEt, and MiniLuke) will be included in the evaluation. The method will be developed and optimized using full-scan spectral acquisition in MS/MS mode. MS and MS/MS parameters optimized for a wide range of pesticides will be critically evaluated.



Sensitivity, linearity and repeatability will be studied at different resolution levels, as this is one of the main aspects for automatic identification of pesticides, especially in complex matrices. In addition, experiments will be conducted to detect possible failures in the software used for automatic data processing. In cooperation with EURL-CF, the method will be requested as a CEN standardised method.

B1.3. Study of the efficiency of extensive clean-ups using high speed centrifugation or specific sorbents based on Zr or others.

The removal of matrix co-extracted components from the extract before analysis is necessary for several reasons: those components interfere with the analysis and can produce broad peaks which overlap analyte peaks and can also increase matrix effects. Furthermore, small amounts of lipids, for example, can damage the column, source and detector. In those cases an extensive clean-up can be the solution. The EURL-FV will test different approaches such as changing operational parameters or extensive clean up options using high speed centrifugation or specific sorbents based on Zr or other kind of particles. Additionally, the study will be performed with commercial fruit and vegetable-based baby food.

This activity will be performed in cooperation with the EURL-CF.

B1.4. Development of a "components map" for all the commodity groups in Document SANCO 12571/2013.

During 2014 the background of relevant commodities affecting the performance of the analytical method applied was evaluated by making use of software programs that extract matrix information from chromatograms achieved with LC-TOF-Equipment in order to help OfLs to avoid matrix effects. In 2015 the capability of GC-QTOF-HRMS for the same purpose will be investigated. Additionally, a components map will be developed for all the typical representative commodities within the different commodity groups in Document SANCO 12571/2013. This activity will be performed in cooperation with the EURL-AO and EURL-CF (they will provide the different extracts and the EURL-FV will analyse and evaluate them)

B1.5 Evaluation of interferences between matrix-analyte for the correct identification of the pesticides by GC-QQQ-MS/MS and LC-QQQ-MS/MS

The complexity of certain vegetable matrices can cause not only problems with the ionization efficiency of the analytical instruments, but also it may happen that some matrix component with very similar mass to the target analyte gives a signal at the same retention time than that specific pesticide. That problem could be a major drawback for an unequivocal identification and in some cases it may lead to false positive occurrences. For this reason the EURL-FV will evaluate the signal given by blanks of all the fruit and vegetables representative matrices when analysed by all the MRM available in the laboratory.



B1.6 Development of an analytical method by LC-microflow coupled to HRMS (QTOF).

The use of microfluidic chromatography devices is a novel technique that has been implemented in the EURL-FV laboratory coupled with tandem mass spectrometry (µLC-QQQ-MS/MS), allowing high sensitivity and shorter times of analysis. In 2015 the same type of microflow device will be coupled to HRMS (µLC-QTOF-MS) with the aim to obtain the same advantages that with tandem mass spectrometry but with the additional gain of working with HRMS. A new method will be developed in different representative commodities and its advantages over the traditional chromatographic device will be evaluated.

B2. Follow-up of the implementation of the developed methods by the Official Laboratories.

The EURL-FV will evaluate the number of entries to the web page in the method section (using google statistics tool). In addition, the EURL-FV will contact the OfLs (via a survey) in order to check the degree of implementation of the new methods developed and published - with the aim of learning any possible difficulties found in the implementation process. This information will be very useful for more effective workshop planning (Activity D1).

B3. Publication of technical reports and scientific papers.

The results of the scientific activities developed by the EURL-FV will be published as technical or scientific documents, depending on the publishing and editorial company. The most relevant will be disseminated in the EURL-FV website (www.eurl-pesticides.eu), through the Library topic making them available for OfLs and members of the scientific community (more than 1,200 entries were received in that section in the first semester of 2014). The main EURL-FV contributions to international conferences will also be uploaded to the EURL-FV website

- Last publications Link:
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- List of methods Link:
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- Conference Contributions Link:
 - $http://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=500\&CntID=904\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=904\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=904\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=904\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=904\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=904\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=904\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=904\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=904\&Theme_ID=904\&Theme_ID=1\&Pdf=False\&Lang=ENLabID=904\&Theme_ID=904\%Theme_ID=904\%Theme_I$



C -QUALITY ASSURANCE AND QUALITY CONTROL PROGRAMME, INCLUDING THE ORGANISATION OF PROFICIENCY TESTS AND INTERCOMPARATIVE STUDIES.

C1. Update of EU Guidelines on Quality Control Procedures.

In order to continue the process of achieving complete harmonisation measures for pesticide residue analysis within the EU, the SANCO document "Analytical quality control and method validation procedures for pesticide residues analysis in food and feed" (SANCO/12571/2013) needs to be revised and updated on continuous basis, especially when difficulties arise.

Therefore, the aim is to carry on with the specific forum (QC Panel) on the EURL-FV website (http://www.eurl-pesticides.eu) so as to facilitate the discussion and to point out difficulties and improvements on the EU QC 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 QC Guidelines, to be presented in the workshop.

Through the QC Panel the laboratories can ask any question related to the "Analytical quality control and method validation procedures for pesticide residues analysis in food and feed" (Document N° SANCO 12571/2013). The four EURLs will coordinate the answers and will publish them on the website.

Regarding the Quality Control Procedures, the expected activities of the EURL-FV in 2015 are:

- To collect changes for the new version, apart than those from the QC Panel, from the Joint Workshop with EURL-SRM, CF and AO organised in Stuttgart in September 2015 and from the specific training tailored to Screening Methods in Almería.
- To implement the above mentioned changes. In a final meeting that will take place in Stuttgart with the ADVG together with the EURL-FV, all the possible modifications to the SANCO Guidelines will be discussed with the aim to produce the new version of the document at the end of the year.
- To edit and distribute electronically and in hard copy among NRLs and OfLs the new version of SANCO Guidelines: SANCO/XXXX/2015. This work will be in coordination with the other three EURLs.

C2. Organisation and Development of Proficiency Tests and Intercomparative studies.

C2.1 Development and conduction of European Proficiency Test FV-17 (EUPT-FV17).

The EURL European Proficiency Test on fruits and vegetables 17, in accordance with previous schemes and statements (EUPT-FV-16) 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 FVO mission recommendation and by request of DG SANCO.

This EUPT 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. In order to facilitate analytical performance control to the laboratories, a "blank" sample will be provided in each EUPT.

The commodity used for the test material of this EUPT FV17 will be chosen in the EUPT Panel meeting that will take place in Madrid (Spain) on 30th September and 1st October 2014. 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. This Proficiency Test will be based on the Quality Control Norm ISO/IEC 17043: Conformity assessment - General requirements for proficiency testing.

The process of the development of the EUPT involves the following steps:

- Contact with the participants.

All the information about this EUPT will appear in the Proficiency Test Topic inside the common EURL webpage and in our area, (EUPT-FV-17 link). And all the forms will be available through this specific webpage (www.eupt.es) using a username and a password for each one of the participating laboratories.

- Production of the matrix.

The matrix intended for the test material will be grown in a dedicated green house subcontracted with that purpose.

The green house will be divided in two separate areas, to cultivate matrix free of pesticides and treated matrix with the selected pesticides.

The crop will be at least 600 kg of matrix in total.

- Concentration Level tests.

Different concentration test levels are performed in small amounts of matrix in order to assess the required concentration levels.

- Preparation of the proficiency test material (both blank and spiked samples).

The organising staff will prepare approximately 200 Kg of homogeneous proficiency test items, treated with the selected incurred pesticides. According to the matrix chosen, it needs to be prepared as the Codex indicates. The proficiency test material is cut into small pieces. This improves the next step which is freezing, using liquid nitrogen, to avoid degradation of the pesticides. It is vital that the cold chain is not broken from this point until the reception of the sample by the laboratory and subsequent analysis.



Once the matrix is completely frozen, it is chopped using a mincer until it becomes powder. After everything has been minced, it is placed in a big homogenising container where it continues to be mixed for 1 hour. Once the hour has elapsed, the mixture is sub-sampled into polyethylene bottles that have been coded. Security screw caps are placed on top and once the bottles are cleaned, they are stored in freezers at -20°C prior to shipment.

The blank proficiency test items are prepared, too, in the same way with another 200 Kg of matrix, this time untreated.

- Homogeneity and Stability tests.

Ten of the bottles containing the test material will be chosen randomly, and analysed to check for homogeneity. The test material will be stored frozen (-20°C) prior to shipment to participants. Two bottles, again chosen randomly, will be analysed over a period of time to confirm the stability of the pesticides in the test material (firstly, when the test materials are shipped, then a few days after the receipt deadline for participants' results). There will be one further analysis during this period reproducing the sample shipment i.e. maintaining the sample at room temperature for a few days to see if there is degradation of any of the pesticides present in the test material. These results will not be included in the proficiency test's statistical analysis. The aim is solely to check pesticide stability during the shipping process and over the duration of the proficiency test.

- Shipment to participants.

Up to 300 g aliquots of test and blank samples will be distributed to each participant that has been accepted to participate in the test, without any information about the residue type or level present in the sample. These samples are sent to the participants by courier packed in dry ice inside special boxes.

- Reception of results.

Once the laboratory has analysed the test material and is ready to submit their data, they must enter their results by accessing the restricted area on the EURL – FV web site: www.eupt.es (see activity A.2).

- Statistical treatment of the results.

The results provided by the laboratories are treated and assessed (estimation of the true concentration, Standard deviation of the assigned value, individual z-scores and combined z-scores calculations etc...) and are included in the Final report. The Advisory group (ADVG) and Quality Control Group (QCG) will support the EURL-FV in all evaluation steps and as a guarantee of transparency.

- Final report and Certificates of participation.

The EURLs will publish a preliminary report, containing tentative assigned values and z-score values for all pesticides present in the test sample, within 2 months of



the deadline for results submission. In addition, this preliminary report will be presented in a specific webinar.

The Final Report will be published after the EUPT-Panel has discussed the results.

Along with the Final Report, the EURL Organiser will deliver an official Certificate of Participation to each participating laboratory with the z-score achieved for each pesticide and the combined z-scores calculated (if any) together with the classification into Category A and B.

Participants will be able to follow the exercise in real time and receive documents and instructions through the specific section of the EURL website (see activity A2). Each participant will receive a detailed electronic report, which will allow the laboratories to interpret their results and to identify possible sources of procedural errors. EUPTs-FV hard copies for each OfLs will be subcontracted.

A final report summarising the scope, results, data treatment and additional information of the methods used will be prepared and made available to every participant laboratory.

Additionally, a critical evaluation on a laboratory-basis and on a country-basis will be prepared and made available to the DG SANCO in the shortest time possible after completing the last stage of the exercise.

The progress made by the NRLs or OfLs will be evaluated, and in those cases where evidence of no advance is found, the EURL-FV will carry out appropriate follow-up following the underperformance criteria agreed by DG-SANCO.

C2.2 EU Intercomparative Study on Mass Spectrometry Screening Methods 07 (EUPT-FV-SM07).

The aim of this intercomparative study is 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 high participation (more than 50 EU OfLs) in previous rounds.

Participation in this 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.

All the information about this EUPT will appear in the Proficiency Test Topic inside the common EURL webpage and in our area, and all the forms will be available through this specific webpage (www.eupt-sm.com) using a username and a password for each one of the participating laboratories.

This intercomparative test, already conducted over the last six years, will be based on the delivery of a matrix treated with around 20 pesticides. In this case



the laboratories will not receive a fixed target list of pesticides, so they will have to analyse the sample in full-scan mode by LC-TOF-MSD, GC-Q-MSD or GC-TOF-MSD systems, for example, or using any other procedure that they may have.

The commodity used for the test material of this EUPT FV17 will be chosen in the EUPT Panel meeting that will take place in Madrid (Spain) on 30th September and 1st October 2014. The commodity and the sample treatment will be the same as the one employed for EUPT-FV-17 but with spiked pesticides instead of incurred. This Proficiency Test will be based on Quality Control Norm ISO/IEC 17043: Conformity assessment - General requirements for proficiency testing.

After sending the treated sample to the participants for analysis, the laboratory has to submit the results in a short period of time after the reception of the samples (72 hours). The aim of this PT is to check if screening methods are run rapidly and efficiently on a routine basis by the laboratories.

A final report summarising the scope, results, data treatment and additional information of the methods used will be prepared and made available to every participant laboratory.

C2.3 EU Intercomparative Study on incurred fresh herbs 01 (EUPT-FV-FH01).

Fresh herbs analyses can present a great challenge to OfLs, but given the specific and significant difficulties of these commodities, they have not been included in previous regular EUPTs. Moreover, those commodities are present in Annez I of Regulation 669 of import controls, making clear the need of OfLs to check the performance of their current methods for that purpose. This year, our intention is to help laboratories evaluate their performance with regard to these commodities and to apply certain modification of the available methods if their results are poor.

To facilitate NRLs and OfLs in achieving acceptable criteria for their fresh herbs analysis performance, the EURL-FV will organise an intercomparative study in that commodity.

This intercomparative study 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. The sample sent to the laboratories for analysis of the present pesticides using their multiresidue methods will be an incurred fresh herb sample containing at least five pesticides previously evaluated by the EURL-FV with adequate amounts of each while considering the interest in having GC and LC-amenable residues in the selected sample.

Participation in this intercomparative study will be on a voluntary basis; nevertheless, all NRLs and OfLs involved in the determination of pesticide residues in fruit and vegetables and third countries will be invited to take part.

Participants will be able to follow the exercise in real time and receive documents and instructions via the specific section in the Proficiency Test Topic, inside the common EURL webpage and in our area (see activity A2).



A final report summarising the scope, results, data treatment and additional information of the methods used will be prepared and made available to every participant laboratory, so each participant will receive a detailed electronic report, allowing laboratories to interpret their results and also to identify possible sources of procedural errors.

Additionally, a critical evaluation on a laboratory-basis and on a country-basis will be prepared and made available to the NRLs and the DG SANCO in the shortest time possible after completing the last stage of the exercise.

The entire organisation of the intercomparative study will be very similar to that of previous EUPTs performed by the EURL-FV.

This intercomparative study will be based on the Quality Control Norm ISO/IEC 17043: Conformity assessment - General requirements for proficiency testing.

C2.4 Ring Test of certified standard solution of EUPT-FV17 (EU-RT-FV17).

One important part of the EUPTs is to detect the possible sources of error in the results. In many cases bad quantification is a consequence of the quality of the working standard solutions of the laboratories. In order to clarify that we will offer the EUPT-FV17 participants (with preference to those who have obtained unsatisfactory results) certified standard solutions so they can check the quality of the working solutions used in their laboratories. A ring test will be organised in the same way as for EU-RT-FV16 (The certified standard solution will be delivered in ampoules by courier with a simple protocol on how to perform the analysis). Participation will be voluntary for those laboratories participating in the EUPT-FV17.

C3. Establishment of criteria for defining underperformance in multiresidue methods.

The EURL-FV in agreement with the DG SANCO will make a proposal of defined criteria to evaluate whether a laboratory has had underperformed in the EUPTs, and if so, this laboratory could be a potential attendee for activity D4.



<u>D - TECHNICAL AND SCIENTIFIC SUPPORT TO DG SANCO, EU MEMBER STATES AND THIRD COUNTRIES INCLUDING THE ORGANISATION OF COURSES AND WORKSHOPS.</u>

D1. Joint EURL-SRM/FV/CF/AO Workshop for Pesticide Residues.

A joint workshop of the four EURLs will be held in Stuttgart, Germany, in September 2015, organised by EURL-SRM. The workshop will be held over two days and 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 and their relation with the various analytical methods applied by the NRLs and OfLs establishing actions for improvement.

Link workshop overview:

http://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=100&CntID=795&Lang=EN

D2. Advisory Group Expert Meeting

As a horizontal task in cooperation with EURL-SRM, EURL-CF and EURL-AO, the members of the scientific group (ADVG) will meet with the four EURL heads of staff once a year, at the end of all the EUPTs (probably in July 2015). The ADVG is made up of scientific experts from various EU countries and has the task of supervising the Proficiency Tests, the statistical data treatment and the performance criteria related to the topic as well as new updating of the EU QC Guidelines.

The Quality control group (QCG) consists of two scientific experts, independent from the laboratory work, who will be in charge of deciding, together with the EURL-FV head of staff, the pesticides and concentrations used to treat the EUPT-FV samples. The QCG will meet with the EURL-FV head of staff twice a year. One of these meetings will be together with the ADVG Workshop; the other will be a computer-assisted virtual meeting.

D3. Technical assistance to DG SANCO.

D3.1. Support to COM and EFSA

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 SANCO upon request. These activities will include:

- Involvement in the EFSA residue evaluation process on behalf of the European Commission by giving opinions and advice, especially regarding residue definition and post registration analytical methods. In the case of new substances it is estimated to carry out experimental analytical work with up to five substances if requested by the DG-SANCO.



- Horizontal task with the four EURLs and coordinated by the EURL-SRM to give scientific support to the Commission as regards residue definitions or other analytical parameters such as LOQs for Art. 12 of Regulation (EC) No 396/2005 proposals.
- Attendance to the Standing Committee (SCFCAH) as the DG SANCO requests (This activity is considered as a mission).
- If the Food and Veterinary Office (FVO) so request it, the EURL will accompany the FVO inspectors in the audit visits giving technical support as a "national expert".

Relevant participation in some international workshops such as RAFA, LAPRW, etc, will be considered as a mission after the agreement of the DG-SANCO.

D3.2. Assistance to COM in drawing up the coordinated multiannual control programme of the Union.

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 agreement with Commission Implementing Regulation (EU) No 788/2012 of 31 August 2012 concerning a coordinated multiannual control programme of the Union for 2013, 2014 and 2015 to ensure compliance with maximum residue pesticide levels and to assess consumer exposure to pesticide residues in and on food of plant and animal origin. Furthermore, this assistance will also be related to the criteria in updating the list of compounds to carry out the EU multiannual control programme and will be linked to previous tasks (B and C).

D4. Training for the NRLs.

The EURL-FV will support the NRLs with technical "lab activities" in collaboration with the EURL-CF. This technical assistance will consist of the selection of a limited group of NRLs (up to 4) to develop technical training of 1-2 days duration at the EURL-CF laboratory (Copenhagen, Denmark). Given the importance of new methodologies the training will be focused on high resolution mass spectrometry techniques, their application to screening methods and validation procedures. In addition to this, the EURL-FV will visit one NRL. The country to be visited will be specified at a later stage in consultation with DG-SANCO. (This sub-activity is considered as a mission).

D5. Assistance to Third Countries.

This task will promote the international networking and dissemination of information and activities from the EURL-FV, especially in countries with intensive European export-import relationships. 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 to participate in EUPT will come from FVO as a consequence of their inspections.

D6. Webinars

The EURL-FV in collaboration with EURL-AO/CF/SRM, will conduct webinars with the aim of disseminating information to the NRLs and some OfLs in a cost effective but still interactive way. In 2015, the EURL-FV will organize at least three webinars, the main relevant topics being activities related to the Work Programme 2015, such as the results of the EUPT-FV17, EUPT-FV-SM07, EUPT-FV-FH01 or EU-RT-FV17. These webinars will be coordinated by the EURL-FV and will be especially focused on dissemination of PT results and the main analytical methods developed. Virtual conference services of these activities will be subcontracted.

D7. Arbitration in the event of litigation

In the case of disputes involving EU Member States, as well as non-EU countries, the EURL-FV counts on highly-qualified personnel available for emergency situations occurring within the European Union, thus being ready to provide its assistance and arbitration within the field of its competence: in particular, to analyse controversial samples for those residues under its mandate in the relevant matrices. These analyses will be performed in the EURL-FV laboratory in the shortest period of time after receiving the sample (< 2 days).



LIST OF TASKS

A - GENERAL TASKS.

- A1. Management of administrative duties.
- A2. EURL-FV Web Page.
- A3. Development of bilateral cooperation with other organisations.
- A4. Collaboration with the other pesticide residue EURLs.

B - DEVELOPMENT AND VALIDATION OF ANALYTICAL METHODS

- **B1.** Development of validated procedures (including the use of new instrumentation)
- B1.1 Update of the GC-Q-TOF-MS database of exact masses of pesticide fragments in El mode and development of a GC-Q-TOF-MS database in NCI mode. Validation of the corresponding screening methods.
- B1.2 Validation of a LC-QTOF HRMS method (request of CEN standardised method).
- B1.3. Study of the efficiency of extensive clean-ups for cereals and feeding stuff, using high speed centrifugation or specific sorbents based on Zr or others.
- B1.4. Development of a "components map" for all the commodity groups in Document SANCO 12571/2013.
- B1.5. Evaluation of interferences between matrix-analyte for the correct identification of the pesticides by GC-QQQ-MS/MS and LC-QQQ-MS/MS
- B1.6 Development of an analytical method by LC-microflow coupled to HRMS (QTOF).
 - **B2.** Follow up of the implementation of the developed methods by the Official Laboratories.
 - B3. Publication of technical reports and scientific papers.

C -QUALITY ASSURANCE AND QUALITY CONTROL PROGRAMME, INCLUDING THE ORGANISATION OF PROFICIENCY TESTS AND INTERCOMPARATIVE STUDIES.

- C1. Update of EU Guidelines on Quality Control Procedures.
- C2. Organisation and development of Proficiency Tests and Intercomparative Studies.
- C2.1. Development and conduction of European Proficiency Test FV-17 (EUPT-FV17).
- C2.2. EU Intercomparative Study on Mass Spectrometry Screening Methods 07 (EUPT-FV-SM07).
- C2.3. EU Intercomparative Study on incurred fresh herbs 01 (EUPT-FV-FH01).
- C2.4. Ring Test of certified standard solution of EUPT-FV17 (EU-RT-FV17).
 - C3. Establishment of criteria for defining underperformance in multiresidue methods.

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 - **D4.** Training for the NRLs.
 - **D5.** Assistance to Third Countries.
 - **D6.** Webinars.
 - **D7.** Arbitration in the event of litigation.