

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

#### **Activity Programme 2016-2017**

#### **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. <u>Development and validation of analytical methods</u>
- C. Quality assurance and quality control programme; including the organisation of Proficiency Tests and Intercomparative Studies
- D. <u>Technical and scientific support to DG SANTE, EU Member States and Third</u>
  <u>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 LOQ: Limit of quantification

MRM: Multiresidue Method

NRL: National Reference Laboratory

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

**QCG:** Quality Control Group



#### 2016-2017 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 (from January to September the FV website has received more than 14000 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 2016-2017 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 2016-2017 (see activity D6) will be included into the Workshop topic: EURL-WEBINARS:

 $\label{lem:http://www.eurl-pesticides.eu/docs/public/tmplt\_article.asp?LabID=500&CntID=933&Theme_ID=1&Pdf=False\&Lang=ENdf=False\&Lang=Endf=Fa$ 



- 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" (\$ANTE/11945/2015). 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 elearning, 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 4000 entries were received in those sections from January to September 2015). Last publications Link:

http://www.eurl-pesticides.eu/docs/public/tmplt\_article.asp?LabID=500&CntID=828&Theme\_ID=1&Pdf=False&Lang=EN Conference Contributions Link:

 $\label{lem:http://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=500&CntID=904&Theme_ID=1&Pdf=False&Lang=ENdf=False&Lang=Endf=Fa$ 

#### New activity:

- EUPT Evaluation Tools. The EURL-FV together with the EURL-SRM will design new online-tools for EURLs and NRLs in order to allow them to view and assess the EUPT-performance of the labs within their network, both on a laboratory basis as well as overall. The result of this collaboration will be discussed within all EURLs and eventually the EUPT-Advisory Group.

#### 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 SANTE.

#### New activity:

- Collaboration with the French NRL (Laboratoire du SCL de Montpellier) in the validation of high sensitive methods for the analysis of pesticides in baby food based mainly on fruits and/or vegetables (2016) (See Activity B.1.1).

#### Follow-up activities:

- Special collaboration with EFSA during 2016 and 2017 in agreement with DG SANTE through Ms. Paula Medina Pastor, scientific officer in EFSA Pesticide Unit. From July 2015, she has become part of the EUPT-Scientific Committee (EUPT-SC) as part of the Quality Control Group (QCG). This link with EFSA Pesticide Unit among the four EURLs will facilitate the communication and collaboration with EFSA, improving the cooperation among the EURLs work and EFSA's work identifying issues to be improved.



-Cooperation with the IRMM-JRC (Institute for Reference Materials and Measurements-Joint Research Centre) in the evaluation of certified materials of plant origin (2017)

#### 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. Additionally, every year the four EURLs will meet in order to discuss specific issues like the EURLs web page, 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. Date and place of these events will be decided at a later stage.



#### **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=EN

#### Last publications Link:

http://www.eurl-pesticides.eu/docs/public/tmplt\_article.asp?LabID=500&CntID=665&Theme\_ID=1&Pdf=False&Lang=EN

#### Conference Contributions Link:

### B.1.1 Development of a highly sensitive analytical method for baby food and organic products.

Ensuring baby food safety is a great concern for the member states, given the vulnerability of such population group. In most of the cases baby food based in fruits and vegetables is produced from organic samples, as the companies want to ensure that the final product is free of pesticides. The control of such low levels of concentrations of pesticide residues requires the use of highly sensitive analytical methodologies. For that reason the EURL-FV will develop a highly sensitive analytical method with limits of quantification below 0.010 mg/kg, including those pesticides in baby food EU legislation (COMMISSION DIRECTIVE 2006/125/EC of 5 December 2006 on processed cereal-based foods and baby foods for infants and young children). Additionally, the method developed will be validated in baby food and organic products.

This activity will be performed during 2016 and 2017.

#### B.1.2 Development of an accurate mass analytical method using GC-orbitrap.

The current analytical systems based on Orbitrap analysers are interfaced to liquid chromatography. Very recently, a new system has been developed combining gas chromatography with Orbitrap analyser, providing high mass accuracy at low concentration levels. Because of the novelty of that technology, no analytical methods are available for pesticide residue analysis in fruits and vegetables using GC-orbitrap-MS/MS. The EURL-FV will evaluate the effectiveness



of that technology for the GC amenable pesticides included in the MACP, and will develop and validate a method.

This activity will be performed during 2017.

### B.1.3 Application of accurate mass to the analysis of "difficult" compounds of low molecular weight.

The analysis of compounds of low molecular weight by mass spectrometry can be challenging as the high specificity provided by the analysis decreases with the molecular weight; and mostly in fruit and vegetable matrices, where a high number of natural components are co-extracted and are potential interferents. In those cases, accurate mass offers a good solution, enabling unambiguous identification of the pesticides analysed. The method will be validated for difficult" compounds of low molecular weight such as phosphonic acid.

This activity will be performed during 2016.

### B.1.4 Development of a MRM method for vegetable oils by both LC-MS/MS and GC-MS/MS.

Very often extraction methods for vegetable oils are intended for pesticides only amenable by gas chromatography, due to the apolarity of those matrices. However, some polar compounds are also used in the plantations destined for the production of vegetable oils, like dimethoate. Moreover, many laboratories still apply GPC clean-up for oil extraction, which is a long and tedious method that constitutes the bottle-neck of the analytical workflow. The removal of matrix co-extracted components from the extract before analysis is necessary, especially in oily samples, which can damage the analytical systems.

The EURL-FV will develop clean-up methods for vegetable oils as simple as possible, avoiding the use of GPC, in order to facilitate its implementation in the workflow of the majority of the OfLs.

This activity will be performed during 2016.

### B.1.5 Use of accurate mass analytical methods combining screening with quantitative detection of frequently found pesticides.

The introduction of new mass spectrometry platforms in the laboratory based on mass spectrometry requires new studies to evaluate pesticide residues in terms of identification and quantification. The combination of accurate mass screening with quantitative detection presents the important advantage of the application to commodities with a high number of co-extractives such as spices, herbs, oily matrices, etc. The EURL-FV in cooperation with the EURL-CF will develop a method based on this new technology, and will validate it in such complex matrices.

This activity will be performed during 2017.



### B.1.6 Development of a CEN standardised method for liquid chromatography coupled to accurate mass spectrometry

During 2015 the EURL-FV validated a routine method based on accurate mass using new and highly sensitive QTOF technology. The method was developed and optimized using full-scan spectral acquisition in MS/MS mode. In cooperation with EURL-CF, the method was included in the detection module of the modular QuEChERS CEN standardised method.

During 2016 and 2017 the EURL-FV together with the EURL-CF will extensively work on the full validation of the CEN method, including inter-laboratory validation.

### B.1.7 Update of the "components map" for typical representative commodities of each commodity group in Document SANTE/11945/2015.

During 2015 the background of one commodity of each different commodity group of Document SANCO/12571/2013 (High water content, High acid content and high water content, High sugar and low water content, High oil content and very low water content, High oil content and intermediate water content, High starch and/or protein content and low water and fat content, "Difficult or unique commodities", Meat (muscle) and Seafood, Milk and milk products, Eggs, Fat from food of animal origin) was evaluated in cooperation with the EURL-AO and EURL-CF by making use of software programs that extract matrix information from chromatograms achieved with LC-TOF-Equipment. Additionally a components map was developed for each one of those commodities.

In 2016 this database of components maps will be enlarged for typical representative commodities of each commodity group in Document SANTE/11945/201, again in cooperation with the EURL-AO and EURL-CF (they will provide the different extracts and the EURL-FV will analyse and evaluate them). At least 35 matrices will be evaluated, one of each typical representative commodity: Pome fruit, Stone fruit, Other fruit, Alliums , Fruiting vegetables/cucurbits, Brassica vegetables, Leafy vegetables and fresh herbs, Stem and stalk vegetables, Forage/fodder crops, Fresh legume vegetables, Leaves of root and tuber vegetables, Fresh Fungi, Root and tuber vegetables or feed, Citrus fruit, Small fruit and berries, Fruit pomace, Honey, dried fruit, Tree nuts, Oil seeds, Pastes of tree nuts and oil seeds, Oily fruits and products, Dry legume vegetables/pulses, Cereal grain and products thereof, Cereal grain and products thereof, incl. cereal based composite feed, Pulses, Red muscle, White muscle, Offal, Fish, Milk, Cheese, Dairy products, Eggs, Fat from meat, Milk fat)



# B.1.8 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. In 2014 a database in electron impact mode was created for 163 pesticides with the exact masses of 1-5 fragments per pesticide. During 2015 the database was updated with 53 new pesticides and their fragments in collaboration with the EURL-CF. Throughout 2016 and 2017 this database will be continuously updated with at least 100 more pesticides and their fragments. The selection of the pesticides will be made in order to cover as many compounds as possible of the EU multi-annual control programme. 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. At the end of 2017 it is expected to have a database containing at least 316 pesticides and their fragments.

This activity will be performed during 2016 and 2017.

### 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 4000 entries were received in those sections from January to September 2015). The main EURL-FV contributions to international conferences will also be uploaded to the EURL-FV website

- Last publications Link:
  - http://www.eurl-pesticides.eu/docs/public/tmplt\_article.asp?LabID=500&CntID=665&Theme\_ID=1&Pdf=False&Lang=EN
- List of methods Link:
- Conference Contributions Link:
  - $http://www.eurl-pesticides.eu/docs/public/tmplt\_article.asp?LabID=500\&CntID=904\&Theme\_ID=1\&Pdf=False\&Lang=ENED=1&Pdf=False\&Lang=EN$



### 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 SANTE document "Analytical quality control and method validation procedures for pesticide residues analysis in food and feed" (SANTE/11945/2015) 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) 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 joint workshop every two years.

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° SANTE/11945/2015). 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 2016 and 2017 are:

- To collect changes for the new version, apart than those from the QC Panel, from the Joint Workshops with EURL-SRM organised in Almería in 2016, and of the four EURLs organised in Freiburg in 2017, and from the specific trainings to the NRLs organised in Almería.
- To implement the above mentioned changes. The AQC ADVG together with the four EURLs will meet once in 2016 and twice in 2017 in order to discuss about the possible changes and modifications to the SANTE Guidelines. In 2017 there will be three meetings. The first one will take place during the first semester of 2017, the second one will be organised in combination with the EUPTs ADVG expert meeting and the final meeting will be held in Freiburg prior to the Joint workshop (no budget has been requested for this last one), where the preliminary document will be produced for the final voting of the NRLs, and if it succeeds, after approval by the COM, the new version of the document will be edited at the end of the year 2017.
- To edit and distribute electronically and in hard copy among NRLs and OfLs the new version of SANTE Guidelines: SANTE/XXXX/2017. This work will be in coordination with the other three EURLs.



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

### C.2.1 Development and conduction of European Proficiency Test FV-18/FV-19 (EUPT-FV18/EUPT-FV19).

The EURL European Proficiency Test on fruits and vegetables 18 and 19, 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 FVO mission recommendation and by request of DG SANTE.

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 materials of these EUPT FV18 and EUPT-FV19 will be chosen in the EUPT Panel meetings that will take place in 2016 and 2017. 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 these EUPTs 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 (<a href="www.eupt.es">www.eupt.es</a>) 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. 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. Both 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 SANTE in the shortest time possible after completing the last stage of the exercise, if they so request it.

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 SANTE.

### C.2.2 EU Intercomparative Study on Mass Spectrometry Screening Methods 08 and 09 (EUPT-FV-SM08/EUPT-FV-SM09).

The aim of these intercomparative studies 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 increasing participation (more than 60 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 seven years, will be based on the delivery of a matrix treated with around 10-20 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 commodities used for the test materials in 2016 and 2017 will be chosen in the corresponding EUPT Panel meetings. The commodities and the sample treatment will be the same as the one employed for EUPT-FV-18 and 19 but with spiked pesticides instead of incurred. These Proficiency Tests 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 these PTs 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.

### C.2.3 EU Intercomparative Study on special commodities (baby food, herbs, spices, etc).

In order to offer the NRLs and OfLs the possibility to test their methods with special commodities and evaluate their performance with regard to those commodities, the EURL-FV will organise an intercomparative study on special commodities such as baby food, herbs, spices, etc.

Those intercomparative studies 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 contain at least five pesticides 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 SANTE in the shortest time possible after completing the last stage of the exercise, if they so request it.

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

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

### C.2.4 Ring Test of certified standard solution of EUPT-FV18 and EUPT-FV19 (EU-RT-FV18/19).

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-FV18 and FV19 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-FV17 (The certified standard solution will be delivered in ampoules by courier with a simple protocol on how to perform the analysis).



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

#### D1. Workshops for Pesticide Residues.

### D.1.1 Joint EURL/NRLs-FV-SRM Workshop 2016 for Pesticide Residues in Fruits and Vegetables.

In 2016, the annual EURL/NRLs-FV workshop will be celebrated together with the EURL-SRM in Almería, Spain, and organised by EURL-FV. 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.

#### D.1.2 Joint EURL-SRM/FV/CF/AO Workshop 2017 for Pesticide Residues.

A joint workshop of the four EURLs will be held in Freiburg, Germany, in 2017, organised by EURL-AO. The workshop will have a duration of two to three 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 an 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 (in both 2016 and 2017). The ADVG is made up of eight 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 from the ADVG, 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.

The budget for these ADVG meetings will only include eight external experts; the EURLs will budgetise these meetings as "missions" of their own budget.



#### D3. Technical assistance to DG SANTE.

#### D.3.1. 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 SANTE 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 SANTE.
- Horizontal task with the four EURLs and coordinated by the EURL-SRM and EURL-CF to 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.
- Horizontal task with the four EURLs in the conduction of a survey on analytical capabilities of the OfLs for the substances in chapter 4 of the monitoring working document.
- Establishment of a database connecting pesticides and information about available methods for their analysis. This task will be performed together with the EURLs CF, AO and SRM.
- Attendance to the Standing Committee (SCFCAH) as the DG SANTE requests (This activity will be 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".

### D.3.2. 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) 2015/595 of 15 April 2015 concerning a coordinated multiannual control programme of the Union for 2016, 2017 and 2018 to ensure compliance with maximum residue levels of pesticides and to assess the 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". This technical assistance will consist on the selection of a limited group of NRLs (between 4 and 8) to develop technical training of 1-2 days duration at the EURL-FV laboratory (Almería, Spain). In 2016 the training will be focused on baby food analysis, with special emphasis on the validation of the pesticides included in the baby food Commission Directive 2006/141/EC at the levels set by the directive or lower, including specific strategies in order to achieve such low levels.

In 2017 the training course will be devoted to accurate mass spectrometry techniques, their application to screening methods and validation procedures, and will be organised jointly with EURL-CF. Each EURL will invite up to four NRLs from their NRL-network. The training course will also aim to introduce modifications in the SANTE document as regard the identification and validation procedures for accurate mass spectrometry.

In addition to this, the EURL-FV will visit one NRL each year. The countries to be visited will be specified at a later stage in consultation with DG SANTE. (This subactivities will be considered as missions).

#### 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. During 2016 and 2017, the EURL-FV will organize at least six webinars, being the main relevant topics activities related to the Work Programme 2016 and 2017, such as the results of the EUPT-FV18/19, EUPT-FV-SM08/9, EUPT-FV-SpecialCommoditiy1/2 or EU-RT-FV18/9. 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).
- B.1.1 Development of a highly sensitive analytical method for baby food and organic products.
- B.1.2 Development of an accurate mass analytical method using GC-orbitrap.
- B.1.3 Application of accurate mass to the analysis of "difficult" compounds of low molecular weight.
- B.1.4 Development of a MRM method for vegetable oils by both LC-MS/MS and GC-MS/MS.
- B.1.5 Use of accurate mass analytical methods combining screening with quantitative detection of frequently found pesticides.
- B.1.6 Development of a CEN standardised method for liquid chromatography coupled to accurate mass spectrometry.
- B.1.7 Update of the "components map" for typical representative commodities of each commodity group in Document SANTE/11945/2015.
- B.1.8 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.
  - 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.
- C.2.1. Development and conduction of European Proficiency Test FV-18/FV-19 (EUPT-FV18/EUPT-FV19).
- C.2.2. EU Intercomparative Study on Mass Spectrometry Screening Methods 08 and 09 (EUPT-FV-SM08/EUPT-FV-SM09).
- C.2.3. EU Intercomparative Study on special commodities (baby food, herbs, spices, etc).
- C.2.4. Ring Test of certified standard solution of EUPT-FV18 and EUPT-FV19 (EU-RT-FV18/19).

### D - TECHNICAL AND SCIENTIFIC SUPPORT TO DG SANTE, EU MEMBER STATES AND THIRD COUNTRIES INCLUDING THE ORGANISATION OF COURSES AND WORKSHOPS

- **D1.** Workshops for Pesticide Residues.
- D.1.1 Joint EURL/NRLs-FV-SRM Workshop 2016 for Pesticide Residues in Fruits and Vegetables.
- D.1.2 Joint EURL-SRM/FV/CF/AO Workshop 2017 for Pesticide Residues
  - D2. Advisory Group Expert Meeting.
  - D3. Technical assistance to DG SANTE.
- D.3.1. Support to COM and EFSA
- D.3.2. Assistance to COM in drawing up the coordinated multiannual control programme of the Union.
  - **D4.** Training for the NRLs.
  - **D5.** Assistance to Third Countries.
  - D6. Webinars
  - **D7.** Arbitration in the event of litigation.