



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

### **Activity Programme 2014**

#### **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. **Technical and scientific support to DG SANCO, EU Member States and Third Countries including the organisation of Courses and Workshops**

#### **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  
**HRAM:** High-Resolution, Accuracy-Mass

**LOQ:** Limit of quantification  
**MRM:** Multiresidue Method  
**NRL:** National Reference Laboratory  
**OfL:** Official Laboratory  
**PT:** Proficiency Test  
**QC:** Quality Control  
**QCG:** Quality Control Group  
**QS:** Quality System



**2014 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.

**Performance Indicators:**

**-Other Activities:** *Adequate execution of the work programme.*

**Expected-ex-ante:** To achieve more than 90 % of the total work programme execution.

**-Other Activities:** *Properly allocated budget.*

**Expected-ex-ante:** Changes of not more than 10 % in budget allocation are expected.

**Deliverables:** Financial and technical reports.

**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 (over the last year there were 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, QC Panel and Library. Technical support to the DG SANCO, to the NRLs and to all the laboratories that have participated in the EUPTs-FV will be provided through the computer tool developed the previous year and located on the EURL website, in the section *EURL-FV Network*. This tool will contain information gathered since EUPT-FV11 about the performance of the participant NRLs and OfLs, such as combined z-score values and the total number of pesticides within their scope, in order to provide a clear picture of the technical capabilities of the laboratories. Only DG SANCO will be able to access all the data, through a user name and password. The participant laboratories can only access their own data, and the NRLs will be able to see the performance of all the laboratories within their network. The link to this tool is going to be placed in the EURL-FV Network topic. The design of this tool and the EUPTs-FV specific webpages ([www.eupt.es](http://www.eupt.es) and [www.eupt-sm.com](http://www.eupt-sm.com)) will be subcontracted with the computer experts.

In cooperation with the other pesticide residue EURLs and coordinated by EURL-AO, the EURL-FV will collaborate with the maintenance of the CIRCA Domain; especially the movement of content from FISVL-Server (Berlin) to EU-Server (Brussels) as well as confirming the status of the enrolled members.

- Forms and related NRLs and OfLs documentation to conduct the 2014 EUPTs will be uploaded onto a specific web pages designed by the EURL-FV ([www.eupt.es](http://www.eupt.es) and [www.eupt-sm.com](http://www.eupt-sm.com)) and linked to the EUPT-FV area.
- Information and main presentations of the webinars to be organized in 2014 (see activity D6) will be included into the Workshop topic: [EURL-WEBINARS](#).
- 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/xxx/201x). 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 document. Another tool available in 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](http://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](http://www.eurl-pesticides.eu)) through the Library topic making them available for OfLs and members of the scientific community (more than 1,500 entries were received in that section last year). Last publications Link: [LAST PUBLICATIONS](#) List of methods Link: [LIST OF METHODS](#)

**Performance indicators:**

**-FF.PT.5:** *Availability of analytical methods developed or validated by the EURL for use by NRLs/OfLs, or (ii) prescribed for official control by European legislation (if applicable).*

**Expected ex-ante:** Three new analytical methods or partially modified analytical methods will be available on the web page.

**-Other Activities:** *Number of entries on the web page.*

**Expected ex-ante:** 10,000 entries/year or more.

**-Other Activities:** *Number of web page updates with all new activities.*

**Expected ex-ante:** 12 web page updates will be made with the new activities, as well as new documents.

**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.
- Cooperation with the NRL in The Netherlands to improve the MiniLuke method.

**Performance indicators**

**-Other Activities:** *Certificates of collaboration from other organisations.*

**Expected ex-ante:**

- Reports for each specific activity developed by the second national expert in collaboration with EURL-FV.

**Deliverables:** Joint EURL report, technical report



**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.**

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: [LIST OF METHODS](#)

**B1.1 Development of an extremely sensitive LC-QQQ-MS method avoiding matrix effects for pesticide determination in fruits and vegetables:**

The application of micro-liquid chromatography can provide very high sensitivity allowing great matrix dilution (20-50 times dilution) to decrease signal suppression due to matrix effects. The implementation of this technique is much cheaper than using highly sensitive mass spectrometers and the new commercially-available narrow columns and micropumps are very effective in routine work. This instrumentation will be evaluated by a  $\mu$ LC-MS/MS method validation for fruits and vegetables including "difficult matrices" such as spices - this will give us better understanding and improve OfL implementation. Spices (white pepper, black pepper, curry and paprika) are considered "difficult" matrices because their extracts have a high amount of coextractives. This new instrumentation can provide an easy and cheap technology for the pesticides determination in fruits and vegetables by the official laboratories.

**Performance indicators:**

**-FF.ANA.1** *Number of newly available analytical methods disseminated to NRLs/OfLs: description of the situation with specification e.g. of new analytical methods developed by the EURL, or in general; or a description of whether partially modified methods, or completely new methods, are expected.*



**Expected ex-ante:** 1 completely new analytical method ( $\mu$ LC-QQQ-MS method by high dilution of the matrix for determination of pesticides in fruits and vegetables including spices)

Training, a workshop and webinar dissemination (see activities D4, D1 and D6)

**-FF.PT.5** *Availability for use by NRLs/OfLs of analytical methods developed or validated by the EURL, or (ii) prescribed for official control by European legislation (if applicable).*

**Expected ex-ante:** The analytical methods validated will be available on the EURL website (List of methods link: [LIST OF METHODS](#)). Additionally, a specific webinar for the NRLs on this will be organised (see activity D6) + round table during the 2014 EURL-FV workshop.

### **B1.2 Development of a high-resolution accuracy-mass method for the screening of a wide range of pesticides in different fruit and vegetable matrices.**

Following the activity programme of the previous years, screening methods will be evaluated considering the introduction of new technologies (HRAMS) in EU OfLs. Over recent years, such instrumentation has been available at a similar cost to the triple quads introduced into routine laboratories. As a consequence, there will be an increasing trend in the near future. Additionally the use of this instrumentation in routine work is complementary to the triple quads and requires an extension to the identification criteria established in the EU QC Guidelines. Consequently, the evaluation of these technologies by the EURL is relevant to the OfLs. This information is also relevant for updating EU QC Guidelines where the MS-based identification parameters are established.

The EURL-FV will develop and validate a routine HRAM (High-Resolution, Accuracy-Mass) method using Orbitrap technology in four different matrices: orange, tomato, tea and pepper; and at two concentration levels. 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 (17500, 35000 and 70000 FWHM); 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.



**Performance indicators:**

**-FF.ANA.1** *Number of newly available analytical methods disseminated to NRLs/OfLs: description of the situation with specification e.g. of new analytical methods developed by the EURL, or in general, or a description of whether partially-modified methods or completely new methods are expected.*

**Expected ex-ante:** 1 completely new analytical method (high-resolution accuracy-mass method for the screening of a wide range of pesticides in different fruit and vegetable matrices).

Training, a workshop and webinar dissemination (see activities D4, D1 and D6).

**-FF.PT.5** *Availability for use by NRLs/OfLs of analytical methods developed or validated by the EURL, or (ii) prescribed for official control by European legislation (if applicable).*

**Expected ex-ante:** The analytical methods validated will be available on the EURL website (List of methods link: [LIST OF METHODS](#)) + round table during the 2014 EURL-FV workshop.

**B1.3. Construction of database with exact masses of pesticide fragments for use in screening methods on GC-Q-TOF-MS:**

GC-Q-TOF-MS is a high resolution technique based on accurate masses. However, unlike LC-MS, compounds analysed with GC-MS are fragmented in the ion source and the molecular ion is rarely seen or only seen in low abundance. Consequently, it is necessary to look for the fragments instead. These data are currently not available. EURL-FV and EURL-CF will calculate exact masses for 1-5 fragments per pesticides, with a focus on pesticides that are not amenable to LC and construct a database. The data will subsequently be uploaded onto the EURL database. This information will be used in EU-QC-Guidelines updating.

**Performance indicators:**

**-Other activities.** Availability for use by NRLs/OfLs data bases.

**Expected ex-ante:** The analytical database generated will be available on the EURL website (<http://www.eurl-pesticides.eu>) with more than 500 visits per year.

**B1.4. Evaluation of extract backgrounds for MRM-pesticides (QuECHERS, SweEt) in fruits and vegetables and in food of animal origin (in cooperation with EURL-AO)**

The evaluation of the background of relevant commodities affecting the performance of the analytical method applied will be developed as a powerful





tool to help OfLs to avoid ion suppression effects. Six FV commodities will be evaluated with different degrees of difficulty. In addition cooperation with EURL AO: evaluating the background of extracts for MRM-pesticides (QuEChERS, SweEt) in food of animal origin. EURL-FV has available software packages to extract matrix information from chromatograms achieved with LC-TOF-Equipment. EURL AO will analyse samples of animal origin from all commodity groups using LC-MS-(Q)-TOF. The resulting data (chromatograms) will be evaluated at EURL-FV to get information of the number of the matrix components being present through the whole chromatogram. This information will be used for interpretation of matrix effects and for improvement of the clean-up with the aim to reduce matrix effects in GC- and LC-MS for a better quantification.

**Performance indicators:**

**-Other activities** Availability for use by NRLs/OfLs databases and better evaluation of the clean-ups applied.

**Expected ex-ante:** *The obtained information will be available on the EURL website (<http://www.eurl-pesticides>), with more than 500 visits per year*

**Deliverables:** 4 Technical Reports

**B2. Follow-up of the implementation of the developed methods in 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 (see activity D1).

**Performance indicators:**

**-FF.PT.6** *Reported use by NRLs/OfLs in relation to EURL's specific promotional activities of analytical methods (i) developed or validated by the EURL (expected take-up), or (ii) prescribed for official control by European legislation (if applicable).*

**Expected ex-ante:** In general, it is expected that at least 70 % of the laboratories will use, to some extent, or take into consideration the updated methods published by the EURL-FV for future validation procedures.



For specific methods such as the ones described in activity B1.1, due to the difficulty of the implementation, it is expected that, during the first year, at least 25 % of the laboratories will use some of the modifications of the methods. Regarding the screening method (Activity B1.2), the method will be presented to the NRLs and OfLs to continue with the implementation of screening methods. Its implementation will be tracked, and it is expected that an implementation of more than 50 % of the laboratories with screening activities will be maintained.

**Deliverables:** survey + webinar + round table during the 2014 EURL-FV workshop.

### **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](http://www.eurl-pesticides.eu)), through the Library topic making them available for OfLs and members of the scientific community (more than 1,500 entries were received in that section last year). EUPTs-FV hard copies for each OfLs will be subcontracted.

Last publications link: [LAST PUBLICATIONS](#). List of methods link: [LIST OF METHODS](#)

#### **Performance indicators:**

**-Other Activities:** *Number of relevant publications in peer-reviewed journals.*

**Expected-ex-ante:** Three scientific papers are expected to be published related to the analytical activities described.

**-Other Activities:** *Number of technical documents available on the EURL website.*

**Expected-ex-ante:** Four technical documents are expected to be published related to the analytical activities described.

**-Other Activities:** *Number of entries on this web page section.*

**Expected ex-ante:** 1,500 entries/year or more.

**Deliverables:** 3 scientific papers + 4 Technical documents + updating these two webpages ([LAST PUBLICATIONS](#) and [LIST OF METHODS](#))



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

**C1. Organisation and Development of Proficiency Tests and Intercomparative studies.**

**C1.1 Development and conduction of European Proficiency Test FV-16:**

The EURL European Proficiency Test on fruits and vegetables 16, in accordance with previous schemes and statements (EUPT-FV-15) will be open to all OfLs, especially the NRLs of EU Member States. Additionally, laboratories from EFTA countries and other third countries which are big exporters to the EU 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 FV16 was chosen in the EUPT Panel meeting that took place in Uppsala (Sweden) on 26<sup>th</sup> and 27<sup>th</sup> June 2013. The test material will be pepper and the EUPT will be conducted with 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-16 link). And all the forms will be available through this specific webpage ([www.eupt.es](http://www.eupt.es)) using a username and a password for each one of the participating laboratories.

**- Production of the matrix.**

The pepper to prepare 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 pepper free of pesticides and treated pepper with the selected pesticides.

The crop will be at least 300 kg of pepper free of pesticides and 300 kg of treated pepper.

**- 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 suggests. The proficiency test material is cut into smaller 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](http://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 (ADV G) 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 medians 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 (<http://www.eurl-pesticides.eu>) (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.

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.

**Performance indicators:**

**-FF.PT.1** *Number of comparative tests following internationally standardized criteria ("high quality comparative tests") to be organised.*

**Expected-ex-ante:** The EURL will organize the European Union Proficiency Test on fruits and vegetables 16 (EUPT-FV-16).

**-FF.PT.2** *Grading addressing the complexity of the PT with the corresponding justification.*

**Expected-ex-ante:** 2 - more challenging analytes, concentration and matrices.

**-FF.PT.3** *Average rates of NRL/OfL success in relation to PT'S grade of complexity and methods and activities to ensure follow-up of poor results.*

**Expected ex-ante:** 160 participant laboratories are expected to take part in EUPT-FV-16, among them NRLs, OfLs and third-country laboratories. More than 75 % of the laboratories are expected to obtain z-scores values between 0 and 2.

**-FF.PT.4** *Progress made by NRLs/OfLs on similar comparative tests with possible discussion of influencing factors:*

**Expected-ex-ante:** At least 10 % of the laboratories are expected to progress positively in comparison with previous EUPT-FVs.

**-FF.ORG.1** *Number of workshops/ training courses/PTs to be organised with a third country focus.*

**Expected ex-ante:** EUPT-FV-16.

**-FF.ORG.2** *Number of third countries invited for each workshop/training course/PT with a third country focus.*

**Expected ex-ante:** 8 third countries will be invited to participate in the EUPTs.

**-FF.ORG.3** *Attendance rate and average rates of NRL/OfL PT success in relation to PT's grade of complexity.*



**Expected ex-ante:** See FF.PT.3

**-FF.R&D.1** *Provision of high quality communication items to NRLs on follow-up of research other than that related to analytical method.*

**Expected ex-ante:** A final report containing the results of the EUPT-FV-16 will be sent to participant laboratories.

**Qualification Indicators:**

**-FF.PT.QI:** *Presence of additional specific quality assurance schemes (type of training that staff involved in this type of activities would receive, ISO acquisition planned etc...)*

**Expected ex-ante:** The European Union Proficiency Tests (EUPTs) will follow the Quality Control Norm ISO/IEC 17043: Conformity assessment - General requirements for proficiency testing. This accreditation has been officially requested by the Spanish Accreditation Body (ENAC).

Following the Quality Control Norm ISO/IEC 17043, a questionnaire of satisfaction is offered to all the participants once the EUPT is finished; more than 85% satisfactory opinions are expected.

**Deliverables:** 1 Proficiency Test Report (Final report) + webinar

**C1.2 EU Intercomparative Study on Mass Spectrometry Screening Methods 06.**

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, (EUPT-FV-SM06 link). And all the forms will be available through this specific webpage ([www.eupt-sm.com](http://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 four 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 was chosen by the EUPT Panel, which took place in Uppsala (Sweden) on 26<sup>th</sup> and 27<sup>th</sup> June 2013. The test material will be pepper. In any case, the commodity and the sample treatment will be the same as the one employed for EUPT-FV-16 but with incurred pesticides. 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.

**Performance Indicators:**

*-FF.PT.1 Number of comparative tests following internationally standardized criteria ("high quality comparative tests") to be organised.*

**Expected ex-ante:** The EURL will organize the European Union Proficiency Test for screening methods 6 (EUPT-SM-06).

*-FF.PT.2 Grading addressing the complexity of each PT with corresponding justification.*

**Expected ex-ante:** 2 - more challenging analytes, concentrations and matrices.

*-FF.PT.3 Average rates of NRL/OfL success in relation to PT's grade of complexity and methods and activities to ensure follow-up of poor results.*

**Expected ex-ante:** 50 participant laboratories are expected to take part in the EUPT-SM-05. At least 70 % of the laboratories are expected to be able to detect at least 70 % of the pesticides present in the test material.

*-FF.PT.4 Progress made by NRLs/OfL on similar comparative tests with possible discussion of influencing factors.*

**Expected ex-ante:** An improvement of between 5 to 10 % is expected in comparison with previous EUPT-SM results.



**-FF.ORG.1** *Number of workshops/ training courses/PTs to be organised with a third country focus.*

**Expected ex-ante:** EUPT-SM-06.

**-FF.ORG.2** *Number of third countries invited for each workshop/training course/PT with a third country focus.*

**Expected ex-ante:** 8 third countries will be invited to participate in the EUPTs.

**-FF.ORG.3** *Attendance rate and average rates of NRL/OfL PT success in relation to PT's grade of complexity.*

**Expected ex-ante:** See FF.PT.3

**-FF.R&D.1** *Provision of high quality communication items to NRLs on follow-up of research other than that related to the analytical method.*

**Expected ex-ante:** A final report containing the results of the EUPT-SM-06 will be sent to the participant laboratories.

**Qualification Indicators:**

**-FF.PT.QI:** *Presence of additional specific quality assurance schemes (type of training that staff involved in this type of activities would receive, ISO acquisition planned etc...).*

**Expected ex-ante:** The European Union Proficiency Tests (EUPTs) will follow the Quality Control Norm ISO/IEC 17043: Conformity assessment - General requirements for proficiency testing. This accreditation has been officially requested by the Spanish Accreditation Body (ENAC).

Following the Quality Control Norm ISO/IEC 17043, a questionnaire of satisfaction is offered to all the participants once the EUPT is finished; more than 80% satisfactory opinions are expected.

**Deliverables:** 1 Intercomparative Study Report (Final Report) + webinar

**C1.3 EU Intercomparative Study on incurred Spices samples 01 (EUPT-SP01).**

Analysis of spices is a great challenge to OfLs, which need to check the performance of their current methods for that purpose. However, given the specific and significant difficulties of these commodities, they have not been included in previous regular EUPTs. 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 spice analysis performance, the EURL-FV will organise an intercomparative study in spices (to be chosen between paprika or red pepper).

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 a commercial incurred spice sample (up to 30 kg) 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, (EUPT-FV-SP01 link), (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, with the peculiarity that, in this case, the sample will be a commercial incurred matrix.

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

**Performance Indicators:**

**-FF.PT.1** *Number of comparative tests following internationally standardized criteria ("high quality comparative tests") to be organised.*

**Expected ex-ante:** The EURL will organize the first intercomparative study on spices.

**-FF.PT.2** *Grading addressing the complexity of each PT with corresponding justification.*

**Expected ex-ante:** 3 - more complex combination of analytes and matrix.

**-FF.PT.3** *Average rates of NRL/OfL success in relation to PT grade of complexity and methods and activities to ensure follow-up of poor results.*

**Expected ex-ante:** 40 participant laboratories are expected to take part in EUPT-T-01. At least 70 % of laboratories are expected to have satisfactory results in this EUPT.

**-FF.ORG.1** *Number of workshops/ training courses/PTs to be organized with a third country focus.*

**Expected ex-ante:** EUPT-Spices-01

**-FF.ORG.2** *Number of third countries invited for each workshop/training/PT with third country focus.*

**Expected ex-ante:** 8 third countries will be invited to participate in the EUPTs.

**-FF.ORG.3** *Attendance rate and average rates of NRL/OfL PT success in relation to PT grade of complexity.*

**Expected ex-ante:** See FF.PT.3

**-FF.R&D.1** *Provision of high quality communication items to NRLs on follow-up of research other than that related to the analytical method.*

**Expected ex-ante:** A final report containing the results of the EUPT-Spices-01 will be sent to participant laboratories.

**Qualification Indicators:**

**-FF.PT.QI:** *Presence of additional specific quality assurance schemes (type of training that staff involved in this type of activity would receive, ISO acquisition planned etc...).*

**Expected ex-ante:** The European Union Proficiency Tests (EUPTs) will follow the Quality Control Norm ISO/IEC 17043: Conformity assessment - General requirements for proficiency testing. This accreditation has been officially requested by the Spanish Accreditation Body (ENAC).

Following Quality Control Norm ISO/IEC 17043, a questionnaire of satisfaction is offered to all participants once the EUPT is finished; more than 80% satisfactory opinions are expected.

**Deliverables:** 1 Proficiency Test Report (Final report) + webinar

**C1.4 Standard Solution Stock Service:**

This service will be open from the date of publication of the EUPT-FV target list until the end of the proficiency test.



The main objective of this service is to facilitate the laboratories' work; the EURL-FV considers it very useful offering them a stock solution with the new compounds introduced in the EUPT target list. That service can assist the speeding up of analytical evaluation from those compounds not included in the validated methods of some OfLs. Laboratories will be able to request pesticide solutions from our stock of standards. Upon request by the laboratories, a vial with a concentrated solution of the pesticide in the chosen solvent will be sent to them. Only the shipment will be charged.

**Performance indicators:**

**-Other Activities:** *Number of requested standard solutions.*

**Expected ex-ante:** At least 10 standard solutions are expected to be requested.

**C2. Establishment of criteria for defining underperformance in multiresidue methods.**

The EURL-FV, in cooperation with EURL-CF, EURL-AO and EURL-SRM in agreement with the DG SANCO will develop 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. These criteria will be set, through meetings or by e-mail, starting from the Basic Agreement with DG SANCO.

**Performance indicators:**

**-Other Activities:** *Number of meetings and people involved*

**Expected ex-ante:** Two meetings (EURL-FV and ADVG)

**Deliverables:** 1 Report



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

**D1. EURL- FV Workshop for Pesticide Residues in Fruits and vegetables.**

A workshop will be held in Almería; it will be focused on updating MRMs applied to “difficult matrices” from FV and screening methods. Extensive interaction with all NRLs that can attend will be the main objective. Other updating activities suggested by the NRLs will also be considered. The workshop will be held over two days and will consist of technical and scientific communications and round tables. 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.

Within 2 months of the workshop, a technical report, which will include feedback evaluation, will be submitted. The technical report will be presented on the EURL-FV website ([LINK EURL-FV WEBSITE](#)).

**Performance indicators:**

**-FF.NRL.1** *Number of participating NRLs/OfLs in the annual workshop (attendance rate)/ Actions taken to ensure all NRL/OfLs participate.*

**Expected ex-ante:** 28 official invitations by e-mail, and by specific request, if necessary. If the laboratory does not participate, it will be asked for a document justifying their non-participation, which will be available if requested, by DG SANCO.

**-FF.NRL.2** *Number of positive satisfaction surveys above 85% received for the annual workshop.*

**Expected ex-ante:** More than 85 % of the attendees satisfied with the workshop.

**-FF.NRL.3** *Measures to address relevant negative feedback from satisfaction surveys.*

**Expected ex-ante:** In case of relevant negative feedback from satisfaction surveys, the EURL-FV will send an e-mail to the laboratories consulting them on which points were unsatisfactory. The findings will be gathered in a report and will be sent to the other EURLs and DG SANCO in order to avoid repeating the same errors in further workshops.

**-FF.R&D.1** *Provision of high quality communication items to NRLs on follow-up of research other than related to the analytical method.*



**Expected ex-ante:** 4 oral communications and 2 round tables concerning general NRL and OfL problems not related to the analytical methods.

**Deliverables:** 1 report + Web page

## **D2. Advisory Group Workshop**

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 October). 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.

### **Performance Indicator:**

**-FF.NRL.5** *Number of workshops/training courses to be organised other than the annual workshop within the framework of EURL activity as described in the AWP (third country involvement to be stated as laid out in the AWP).*

**Expected ex-ante:** 1 ADVG Workshop.

**-FF.NRL.6** *Attendance rate and number of positive satisfaction surveys above 85% received for such workshops.*

**Expected ex-ante:** 10 participants.

**Deliverables:** 1 Minute Report + web page ([ADVG Workshop](#)).

## **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 EPRW, MGPRW, 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).

#### **Performance Indicator:**

**-FF.COM.1** *Number of qualified staff in terms of preparedness with relevant completed training able to travel, to assist during outbreaks/ crisis situations and/or to engage the EURL on-site.*

**Expected ex-ante:** 5 highly qualified staff, most of them with PhD degrees, available to meet with the DG SANCO and who can participate and interchange e-mails with DG SANCO.

**-FF.COM.2** *Adequacy of response to requests in terms of 1) content and 2) timely delivery.*

**Expected ex-ante:** The EURL-FV has established a "help desk" - 8 hours a day, 220 days a year.



**-Other Activities:** *Certificates of collaboration from other organisations.*

**Expected ex-ante:**

- LOQs or other analytical parameters of approximately 50 substances will be forwarded to the DG SANCO and EFSA upon request. In addition to some 5 new substances where no analytical information is available; these will be evaluated experimentally in the laboratory: see analytical part (B1.1).

- Reports for each specific activity developed by the second national expert in collaboration with EURL-FV.

**Deliverables:** Reports + PowerPoint of technical-scientific contributions in the library section in our webpage.

**D4. Training for the NRLs.**

The EURL-FV will support the NRLs with technical "lab activities". This technical assistance will consist of the selection of a limited group of NRLs (up to 8) to develop technical training of 1-2 days duration at the EURL-FV laboratory. New analytical methods will be demonstrated in a practical way attending to the questions and details from the NRLs. The selection of the laboratories will be fixed following consultation with DG SANCO, giving emphasis to NRLs who underperformed in the previous EUPT-FVs and EUPT-SMs; or as a consequence of specific results and difficulties in their network. New methodologies to evaluate "difficult matrices" and screening updated methods are initially selected for 2014. However, the scope is open to some specific requests of great interest to the NRLs.

In addition to this, the EURL-FV will visit one NRL where it was noticed that the country network has deficits in the areas of EUPT-performance and scope. The country to be visited will be specified at a later stage in consultation with DG-SANCO (This activity is considered as a mission). In 2014 a visit to Croatia in collaboration with the EURL-SRM is expected.

**Performance Indicator:**

**-FF.NRL.4** *Number of NRLs for training.*

**Expected ex-ante:** Up to 8 NRLs.

An NRL satisfaction survey is expected to receive tailored training from the EURL-FV.

**-Other Activities:** *Visit to the Croatian NRL*

**Expected ex-ante:** 1 technical report





**Deliverables:** 1 survey + 1 Technical Report

**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. Joint EURL-CF/SRM/AO/FV Webinars**

The EURL-FV in collaboration with EURL-CF and 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 2014, each EURL will organize at least two webinars individually, or in collaboration with other EURLs, the main relevant topics being activities related to the Work Programme 2014. 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.

**Performance Indicator:**

*-FF.R&D.1 Provision of high quality communication items to NRLs on follow-up of research other than that related to the analytical method.*

**Expected ex-ante:** At least 2 webinars.

**Deliverables:** 2 Recorded webinars + web page ([EURL-Webinars](#))

**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).



**Performance Indicator:**

**-FF.COM.1** *Number of qualified staff in terms of preparedness with relevant completed training able to travel, to assist during outbreaks/ crisis situations and/or to engage the EURL on-site.*

**Expected ex-ante:** 5 highly qualified staff, most of them with PhD degrees, available to meet with the DG SANCO and who can participate and interchange e-mails with DG SANCO.

**-FF.COM.2** *Adequacy of response to requests in terms of 1) content and 2) timely delivery.*

**Expected ex-ante:** The EURL-FV has established a "help desk" - 8 hours a day, 220 days a year. In case of arbitration in the event of litigation, submission of the results of the analyses to the DG SANCO will be within a period of time less than 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.
  - B1.1 Development of an extremely sensitive LC-QQQ-MS method avoiding matrix effects for pesticide determination in fruits and vegetables
  - B1.2 Development of a high-resolution accuracy-mass method for the screening of a wide range of pesticides in different fruit and vegetable matrices
  - B1.3. Construction of database with exact masses of pesticide fragments for use in screening methods on GC-Q-TOF-MS
  - B1.4. Evaluation of extract backgrounds for MRM-pesticides (QuECHERS, SweEt) in fruits and vegetables and in food of animal origin (in cooperation with EURL-AO)
- B2.** Follow up of the implementation of the developed methods in 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.** Organisation and development of Proficiency Tests and Intercomparative Studies.
  - C1.1. Development and conduction of European Proficiency Test FV-16.
  - C1.2. EU Intercomparative Study on Mass Spectrometry Screening Methods 06.
  - C1.3. EU Intercomparative Study on incurred Spices samples 01 (EUPT-SP01).
  - C1.4. Standard Solution Stock Service.
- C2.** Establishment of criteria for defining underperformance in multiresidue methods.

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

- D1.** EURL-FV Workshop for Pesticide Residues in fruits and vegetables.
- D2.** Advisory Group Workshop.
- D3.** Technical assistance to DG SANCO.

**EURL FOR PESTICIDE RESIDUES IN FRUITS AND VEGETABLES (EURL-FV)**  
**2014 Activity Programme**



D3.1. Support to COM and EFSA

D3.2. 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.** Joint EURL-CF/SRM/AO/FV Webinars.

**D7.** Arbitration in the event of litigation.