



Planned work programme for 2016 and 2017 (as submitted on 25.09.2015 and clarified on 08.10.2015)

Explanatory remark

This planned work programme reflects the **tasks** of European Union Reference Laboratories (EURLs) as fixed in REGULATION (EC) No 882/2004 of the European Parliament and of the Council. Furthermore, SANTE/10305/2015 CIS and its ANNEX define **four operational objectives** for the work of the EU Reference Laboratories for 2016 and 2017. In the following planned work programme for 2016 and 2017, the fulfilment of these objectives is indicated for each topic (directly under title of each topic). Then, the above mentioned SANTE document describes **activities** to be funded specifically for each EURL. Also the fulfilment of these activities is indicated.

General Objectives

- A. General tasks**
- B. Development and validation of analytical methods**
- C. Quality assurance and quality control including the organisation and implementation of proficiency tests**
- D. Technical and scientific support to the EU Commission, National Reference Laboratories (NRLs), Official Laboratories (OfLs) and Third Countries**

Working plan for the period January 2016 - December 2017 and multi-annual goals

A. General Tasks

Covering the **operational objectives**:

- (1) To ensure the availability of scientific and technical assistance provided by the EURLs,
- (2) To ensure a sound and efficient management of EURL funding cycle.



1. 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 later.
2. Compilation of the Technical and Financial Report for 2015 by 31 March, 2016.
3. Compilation of an intermediate Technical and Financial Report for 2016 by 31 March 2017 and of a final Technical and Financial Report for 2017 by 31 March 2018.
4. Compilation of the planned activities and estimated budget for 2018 (or 2018 and 2019) by 30 September 2017.
5. For the EURL/NRL network, the list of all NRLs and contact points in the field “pesticides in food of animal origin and commodities with high fat content” will be kept updated. In addition, all OfLs as reported by the NRLs will be added to this list and will be made available to the Commission and NRLs. This task will be performed in close cooperation with the EURL for Single Residue Methods (EURL SRM).
6. Maintenance of the EURL-website in cooperation with the other EURLs for pesticide residues, exchange of information via the website and updating on regular basis with the particular aim of disseminating information to NRLs. Information about important improvements of analytical methodology and major changes in EU legislation.
7. Maintenance of the CIRCA-BC Domain in cooperation with the other EURLs for pesticide residues; continuous provision of the status of the enrolled members.

B. Development and Validation of Analytical Methods

Covering

(1) the operational objectives:

- ✓ **To ensure the development and use of high quality analytical methods across the EU-RL framework,**
- ✓ **To maintain appropriate level of proficiency testing ensuring efficiency of control analysis methods.**

(2) the specific activity for the pesticide EURLs:

- ✓ **development, validation and dissemination of new and improved methods for analysing pesticide residues in food of animal origin, cereals and fruits and vegetables**

Analytical scientific work should cover the improvement of existing, approved multi-methods and the development and implementation of new multi-methods.



1. Certain pesticides (mainly organochlorine, organophosphorous and pyrethroid pesticides) can be considered as introduced in the daily routine of NRLs for pesticides in food of animal origin and high fat commodities. The evaluation of the results of EU Proficiency Tests (EUPTs) of the EURL-AO (EUPT AO-01 to AO-10) shows the progress achieved during the last 5 years. However, there is also further need for improvement for these groups. Therefore, the spectrum of analytes will be extended continuously, also by inclusion of LC-MS-based methods (QuEChERS or SweEt-based).

It is planned to add at least 20 new pesticides¹ into the existing methods for determination and to validate them in two other matrices of interest.

2. The EURL for Fruits and Vegetables (EURL FV) has available software packages to extract matrix information from chromatograms achieved with LC- and GC-ToF-Equipment. In 2014 the EURL AO and the EURL FV started a cooperation evaluating the background of extracts for MRM-pesticides (QuEChERS, SweEt) in food of animal origin. In 2015 GC-MS-ToF was included in the cooperation.

EURL-FV analyses extracts with ToF-systems provided by EURL-AO. The resulting data (chromatograms) will be evaluated at EURL FV to get information about the density of matrix components being present through the whole chromatogram. The 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.

This cooperation will be continued in 2016 and 2017. Especially the evaluation of extracts derived with Zentrimix-equipment is of interest for all matrices of animal origin (see B.4).

3. In 2013 EURL AO developed a multi-residue method (MRM-method) for analysis of less polar pesticides (organochlorine, organophosphorous and pyrethroids) in liver and honey applying GC-MS/MS. In 2014 the method was extended to the matrix egg and in 2015 to milk (including infant formula und milk powder) and muscle (including meat containing baby food).

¹ At least 20 pesticides from the following: 1-(4-Chlorphenyl)urea, 4-Hydroxy-Cyprodinil CGA304075, Aldicarb-sulfoxid, Ametryne (Ametrex), Aminocarb, Baycor (Bitertanol), Benalaxyl, Bendiocarb, Benzoximate, Bifenazate (D 2341), Bupirimate, Butafenacil, Carbetamid, Carbofuran, Carbofuran-3-hydroxy, Carfentrazone-ethyl, Chlorantraniliprole, Chlorfluazurone, Chlortoluron, Clethodim, Cyazofamid, Cycluron, Dichlobutrazol (Diclobutrazol), Dicrotophos (Bidrin), Diethofencarb, Dimoxystrobin, Diniconazole(I), Dinotefuran, Di-oxacarb, Diuron, Etaconazole(I), Ethiofencarb, Ethiprole, Ethirimol, Etoxazole, Fenarimol, Fenazaquin, Fenpropidin-CGA289267, Fenpyroximate, Fenuron, Flonicamid, Flubendiamide, Flufenacet (Fluthiamide) (BAY FOE 5043), Flufenoxuron, Fluometuron, Fluoxastrobin, Forchlorfenuron, Furalaxyl, Halofenozide, Hydramethylnon, Iprovalicarb, Isocarbophos, Isoprocab, Isoproturon, Kresoxim, Kresoxim-methyl, Mandipropamid, Mefenacet (Rancho), Mepronil, Mesotrione, Methabenzthiazuron, Methamidophos (Metamidophos), Methoprotrolyne, Methoxyfenozid, Metobromuron, Metribuzin, Mevinphos (Phosdrin), Mexacarbate (Zectran), Monocrotophos (Azodrin), Monolinuron (phenylurea), Myclobutanil-RH9090, Nuarimol, Oxadixyl, Oxamyl, Penconazole(I), Pencycuron, Picoxystrobin, Piperonyl-butoxide, Promecarb, Propamocarb, Propargite, Propiconazole, Propoxur, Pymetrozine, Pyracarbolid, Pyraclostrobin, Pyridaben, Pyrimethanil, Pyriproxyfen, Rotenone, Siduron, Simetryn, Spinetoram, Spirodiclofen, Spiromesifen, Spirotetramat-BYI03380-enol, Spirotetramat-BYI03380-monohydroxy, Sulfentrazone, Tebuthiuron, Temephos (Abate), Terbumeton, Terbutryn, Thiabendazole-5-hydroxy, Vamidothion



The extraction step is based on a mixture of ethyl acetate and cyclohexane (1 + 1/v + v) as solvent. One big advantage of this mixture is the possibility to use an aliquot of the extract directly for further clean-up by gel permeation chromatography. Thus, the new extraction procedure helps to save time during the sample extraction procedures and can be understood as a new module for the extraction step of the method according EN 1528. In 2016 and 2017 the method will be further developed, if necessary modified and validated for at least three of the following matrices of interest: fat of animal origin, vegetable oil, kidney or fermented milk products (e.g. cheese, yogurt). After successful tests the method will be validated following the Quality Control Procedures for Pesticide Residues Analysis (Document N° SANCO/2013/12751).

In addition, the possibility of applying standard-detectors (ECD, single MS) for identification and determination of the pesticides in the sample extracts will be checked. The advantages of standard detectors like ECD or single MS are their significant lower costs with regard to purchase and routine operation.

4. Improvement of extraction and clean-up steps (QuEChERS, SweEt) for food of animal origin.

4a. Speeding up

Present MRM-methods for more polar pesticides like QuEChERS and SweEt are fast and well introduced in a quite number of laboratories. Combining extraction and centrifugation in one step (Zentrimix) can improve the applicability of the methods. In 2015 the clean-up was tested for milk including infant formula (milk powder) and for muscle including meat-containing baby food with about 80 pesticides². At least two out of the matrices kidney, fat and egg will tested and validated in 2016 - 2017.

² At least 80 pesticides from the following list: Acetamiprid, Aldicarb, Amitraz, Azinphos-ethyl, Azoxystrobin, Bixafen, Boscalid, Bromuconazol, Buprofezin, Butoxycarboxim, Carbaryl, Carbendazim, Carbofuran, Carbofuran-3-hydroxy, Carboxin, Chlordimeform, Chlorpropham, Chlorpyrifos-methyl, Clofentezin, Clothianidin, Coumaphos, Coumaphos-oxon, Cymiazol, Cymoxanil, Cyproconazol, Cyprodinil, Difenconazol, Diflubenzuron, Dimethoat, Dimethomorph, Dimoxystrobin, DMP-formamid, 2,4-, DMP-me-formamidin, 2,4-, Epoxiconazol, Etofenprox, Etoprophos, Famoxadon, Fenamidon, Fenbuconazol, Fenhexamid, Fenoxycarb, Fenpropidin, Fenpropimorph, Fenthion, Fenthion-oxon, Fenthion-oxon-sulfon, Fenthion-oxon-sulfoxid, Flua-zifop-butyl, Fluoxastrobin, Fluquinconazol, Flusilazol, Flutolanil, Flutriafol, Fluvalinat, tau-, Formetanat, Fosthiazat, Haloxyfop, Haloxyfop-2-ethoxyethyl, Haloxyfop-methyl, Hexaconazol, Hexythiazox, Imazalil, Imidacloprid, Indoxacarb, Iprovalicarb, Isoproturon, Linuron, Lufenuron, Malaaxon, Malathion, Mepanipyrim, Metalaxyl, Metamitron, Metazachlor, Metconazol, Methiocarb, Methiocarb-sulfon, Methiocarb-sulfoxid, Methomyl, Myclobutanil, Nitenpyram, Omethoat, Oxydemeton-Methyl, Paclobutrazol, Pendimethalin, Phosalon, Phosmet, Phosmet-oxon, Phoxim, Pirimicarb, Pirimicarb-desmethyl, Pirimiphos-methyl, Prochloraz, Propamocarb, Propargit, Propiconazol, Propyzamid, Pyraclostrobin, Pyrimethanil, Pyriproxyfen, Quinoxifen, Spinosyn A, Spinosyn D, Spirotetramat, Spiroxamin, Tebuconazol, Tebufenozid, Tebufenpyrad, Tepraloxym, Terbutylazin, Tetraconazol, Thiabendazol, Thiacloprid, Thiamethoxam, Thiophanat-methyl, Triadimefon, Triadimenol, Trichlorfon, Tricyclazol, Trifloxystrobin, Triflumizol, Triflumuron, Triticonazol, Zoxamid



4b. Clean-up

Co-extracted matrix compounds interfering with the target analytes (matrix suppression in LC-MS/MS or matrix enhancement in GC-MS/MS) may lead to wrong results. For at least two of the matrixes milk, muscle, kidney, fat and egg further clean-up steps will be tested by use of commercially available alternative extraction kits for clean-up for about 80 pesticides² and, if successful, validated.

5. EURL AO will continue its work for development of multi-residue methods for the determination of residues of semi-polar and for more polar pesticides in fish (all MRM-pesticides included in the Multiannual Coordinated Control Program 2016 till 2018 for matrices of animal origin will be included). The increase of the spectrum will comprise pesticides listed in footnotes 1 and 2. Method validation follows the Quality Control Procedures for Pesticide Residues Analysis (Document N° SANCO/2013/12751 or succeeding document).
6. The EURL AO will conduct two feeding studies for the matrices egg and fish in cooperation with EURL for Cereals and Feedingstuffs (EURL CF). Pesticide residues in feed can be transferred to the animals. For most feed/pesticide/animal combinations the transfer will not result in pesticide residue levels measurable in the animals. However, residues in eggs and in fish could be of interest. The following two task focus on this:
 - 6a. Pesticide residues in feed to laying hens and pesticide residues in eggs (this part will be performed preferable in 2016): In 2011 the EURL AO and EURL CF initiated a study with the aim to detect pesticide residues in egg when known pesticides residues are present in the feed provided for the laying hens. However, the feeding study was stopped after few days due to a high weight loss for the hens. This was probably due to high concentrations of especially organochlorines (aldrin and lindane). A repetition of the feeding study on laying hens with feed containing lower residues of pesticides will be organised. EURL CF will produce the feed and EURL AO will organize the feeding experiments and analyse the resulting residues in the eggs. The study will enable the estimation of the risk for consumers in relation to pesticide residues in eggs.
 - 6b. Pesticide residues in feed for farmed fish and pesticide residues in fish (this part will be performed preferable in 2017 after the survey on feed for farmed fish of the EURL CF): So far only few data exist on pesticide residues in feed for farmed fish and in fish. Fish feed consists mainly of cereals and fish meal. Both constituents contain pesticide residues. To elucidate the correlation between feed and fish, the EURL AO and EURL FV will cooperate on the feeding study with known pesticides residues in feed. EURL CF will produce the feed and EURL AO will organize the feeding experiments and analyse the resulting residues in the fish. The study will enable the estimation of the risk for consumers in relation to pesticide residues in fish.



C. Quality Assurance and Quality Control

Covering

(1) the operational objectives

- ✓ To ensure the development and use of high quality analytical methods across the EU-RL framework,
- ✓ To maintain appropriate level of proficiency testing ensuring efficiency of control analysis methods,
- ✓ To ensure the availability of scientific and technical assistance provided by the EU-RLs.

(2) the specific activity for the pesticide EURLs

- ✓ development, validation and dissemination of new and improved methods for analysing pesticide residues in food of animal origin, cereals and fruits and vegetables

1. As multi-annual goal, the availability of validated methods for the most important groups of pesticide residues in the most important matrices will be checked and promoted within the EURL / NRL network. Therefore, EURL AO will check on a regularly basis if
 - new methods for pesticides in food of animal origin or
 - information for improvement/enhancement of existing methods are published.

EURL AO examines these methods theoretically and, in few cases, by some practical tests. Thereafter the information will be spread to the network through CIRCA (and via a link on the EURL homepage), during the workshop or by providing the data through the EURL Data Pool.

2. Maintenance of in-house QA/QC activities in consequence of the ISO 17025 accreditation of all analytical work done within the EURL.
3. Maintenance of proficiency testing in consequence of the ISO 17043 accreditation for performing EUPTs.
4. Performance of a proficiency test (PT) for MRM-pesticides in 2016 and in 2017. The PTs will be performed with all NRLs and OfLs of the EU Member States and Associated States. Both PTs will be designed for 90 to 130 participating laboratories (intention 110: 28 NRLs, 78 EU-OfLs, 2 EFTA laboratories, 1 Candidate State laboratory and 1 Third Country laboratory) and the target analytes lists will comprise about 50 to 80 MRM-pesticides (intention 65). The PT will be performed and evaluated in accordance with the General Protocol for EU proficiency tests for pesticide residues in food and feed. The time schedule will be coordinated with the other pesticide EURLs and the Commission to avoid overlapping periods.



The preliminary results will be discussed at the joint meeting of the four pesticide EURLs for evaluation of the EUPT results (see C.5). The final results will be discussed with NRLs at the workshops (see D.8).

Pig fat (lard) will be the matrix for the test items in EUPT AO 11 (2016).

The matrix for the test items in EUPT AO 12 (2017) will be fixed during the joint meeting for evaluating EUPTs (see C.5) or during the workshop with NRLs in Freiburg in 2016.

5. Participation in joint meeting to discuss and evaluate EUPT results, and overall PT-performance (horizontal activity). EURLs for FV, CF, SRM and AO will further develop the criteria for evaluation underperformance of laboratories in the EUPTs. Laboratories with indication of underperformance could be potential candidates for activity D.7
6. Contribution to the revision of “Method Validation and Quality Control Procedures for Pesticide Residue Analysis in Food and Feed” (Document N° SANCO/12751/2013): Participation in all meetings of the Advisory Group for the improvement of the document and contributions by mail contacts.

D. Technical and Scientific Support to the Commission, NRLs, EU Official laboratories and Third Countries

Covering

(1) the operational objectives

- ✓ **To ensure the development and use of high quality analytical methods across the EU-RL framework,**
- ✓ **To maintain appropriate level of proficiency testing ensuring efficiency of control analysis methods,**
- ✓ **To ensure the availability of scientific and technical assistance provided by the EU-RLs.**

(2) the specific activity

- ✓ **provision of scientific and technical assistance to the Commission, especially concerning limits of quantification and residue definitions in the framework of the review of all existing MRLs laid down in Article 12 of Regulation (EC) 396/2005**

1. General technical and scientific support to the Commission and its offices, as requested by the Commission.
2. Scientific support to the Commission and EFSA as regards the evaluation of possible applicability of proposed post-registration methods in routine analysis laboratories and residue definitions, esp. in the case of Art. 12 proposals.



3. Analytical support to the Commission upon request.
Analyses of official samples submitted by EU Member States in case of dispute between Member States or in case of analytical problems with a responsible NRL.

Upon request of the Commission EURL AO will analyse samples from other Member States for confirmation of critical results in crisis situations.
4. Support to the Commission for drafting of the EU coordinated working programme.
Tasks performed together with the EURLs CF, FV and SRM.
 - Survey on analytical capability for the substances in chapter 4 of the monitoring working document.
 - Establishment of a database connecting the individual pesticides with information about available methods for their analysis.
5. Scientific support to NRLs.

General scientific information will be provided to NRLs. In particular in case of problems, NRLs will be supported with methods for MRM-pesticides. In certain cases also the analytes could be supplied (e.g. in case of lack of information about pesticides and the availability of the standards, degradation of standard).
6. Analytical support to NRLs upon request.

EURL AO will assist NRLs to establish new methods by providing them with test materials from previous EUPTs (test material service) or by exchange of samples and analysis in parallel to show the validity of the results.
7. Missions to NRLs (at least one in 2016 and at least one in 2017), e.g. as an outcome of tasks C.4 and C.5.
8. A workshop of the all EURL-AO network will be held in Freiburg in 2016.

In 2017 the Joint Workshop of the EURL-NRL-OfL-network for pesticides residues will be held in Freiburg organized by EURL-AO.
9. A trainee workshop in Freiburg in close connection EURL-AO workshop 2016 for up to 8 participants.

In 2017 a trainee workshop will be offered in Freiburg independent from the Joint Workshop of the EURL-NRL-OfL-network for pesticides residues for up to 9 participants.
10. At least one webinar per year will be organized individually or in collaboration with other EURLs for the network of NRLs and OfLs.