



# Planned work programme for 2015 (as submitted on 08.08.2014 and revised on 26.09.2014)

### Legal functions and duties

The functions and duties of the European Union Reference Laboratory are described in Regulation No 882/2004. Objectives for the period January - December 2015 will be:

### **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 NRLs/ EU Official labs, Commission and third countries

### Working plan for the period January - December 2015 and multi-annual goals

#### A. General Tasks

### Covering the operational objectives:

- (3) To ensure the availability of scientific and technical assistance provided by the EU-RLs,
- (4) To ensure a sound and efficient management of EU-RL funding cycle.
- 1. Participation in co-ordinating meetings and general management activities of EURLs for residues. The EURL "pesticides in food of animal origin and commodities with

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high fat content" (EURL-AO) needs a close cooperation with the other pesticide EURLs.

- 2. Compilation of the Technical and Financial Report for 2014 by 31 March 2015.
- 3. Compilation of the planned activities and estimated budget for 2016 by 31 August 2015.
- 4. 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 connection to EURL SRM.
- 5. 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.
- 6. Maintenance of the CIRCA Domain in cooperation with the other EURLs for pesticide residues; continuous proving the status of the enrolled members.

#### B. Development and Validation of Analytical Methods

### Covering the operational objectives

- (1) To ensure the development and use of high quality analytical methods across the EU-RL framework.
- (2) To maintain appropriate level of proficiency testing ensuring efficiency of control analysis methods.

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

1. Three groups of pesticides (organochlorine, organophosphorous and pyrethroid pesticides) can be considered to have been introduced to a certain degree in the daily routine of NRLs for pesticides in food of animal origin and high fat commodities. The evaluation of the results of EUPTs AO-01 to AO-09 showed that there were some advancements during the last 5 years but there is further need for improvement also for these groups. 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 pesticides<sup>1</sup> into the existing determination methods and to validate them in 2 other matrices (honey, muscle).
- 2. In 2014 EURL AO and EURL FV started a cooperation 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. From 2015 on, GC-MS-ToF will be included in the cooperation. EURL-FV will analyse extracts 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 cleanup with the aim to reduce matrix effects in GC- and LC-MS for a better quantification. This cooperation will be continued in 2015.
- 3. In 2013 EURL AO developed a MRM-method for analysing less polar pesticides (organo chlorine, organo phosphorous and pyrethroids) in liver and honey applying GC-MS/MS. The extraction step is based on a mixture of ethyl acetate and cyclohexane (1 + 1(v + v) as solvent. As an advantage of using this mixture, an aliquot of the extract can be used directly for a further cleanup by gel chromatography (and, if necessary with an optional silica in combination with zirconium oxide clean-up step for difficult matrices). 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 2014 the method was extended to the matrix egg.

In 2015 the method shall be tested (and if necessary, modified) for at least 80 pesti-

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<sup>&</sup>lt;sup>1</sup> 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, Dioxacarb, 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, Isoprocarb, Isoproturon, Kresoxim, Kresoxim-methyl, Mandipropamid, Mefenacet (Rancho), Mepronil, Mesotrione, Methabenzthiazuron, Methamidophos (Metamidophos), Methoprotryne, 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





cides (mainly organo chlorine, organo phosphorous and pyrethroids<sup>2</sup>) in the matrices milk and muscle. After successfully test 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 cheaper standard-detectors (ECD, single MS) for identification and determination of the pesticides in the sample extracts will be checked.

- 4. Improvement of extraction and cleanup steps (QuEChERS, SweEt) for food of animal origin.
  - Actual 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 can improve the applicability of the methods. This will be tested for two other matrices AO (suggestion milk and muscle) and about 80 pesticides<sup>3</sup>. Co-extracted matrix compounds interfere with the target analytes (matrix suppression in LC-MS or matrix enhancement in GC-MS) may lead to wrong results. For these two matrixes (suggestion milk and muscle) further cleanup by using of alternative add ins for cleanup will be tested for about 80 pesticides and, if successful, validated.
- EURL will start to develop one multi-residue-methods for the determination of residues of semi-polar and one for more polar pesticides in fish (all MRM-pesticides included in the Multiannual Coordinated Control Program 2015 and 2016 for matrices

<sup>2</sup> Beside others, it is planned to include the following pesticides: Hexachlorbenzol HCB, HCH, alpha-, HCH, beta-, Lindan gamma-HCH, HCH, delta-, trans-Nonachlor, DDE, op-, DDE, pp-, DDT, op-, DDT, pp-, DDD, op-, DDD, pp-, Aldrin, Dieldrin, Heptachlor, Heptachlorepoxid, cis-, Heptachlorepoxid, trans-, Endosulfan, alpha-, Endosulfan, beta-, Endosulfan-sulfat, Chlordan, alpha(cis)-, Chlordan, gamma(trans)-, Oxychlordan, Endrin, delta-Ketoendrin, Iodofenphos, Methoxychlor, Nitrofen, Pendimethalin, Tecnazen, Quintozen, Fenpropathrin, Lambda-Cyhalothrin,, Cyphenothrin, cis-Permethrin, trans-Permethrin, Cyfluthrin, , Cypermethrin, , Fenvalerat / Esfenvalerat, , Deltamethrin, Chlorpyrifos-methyl, Parathion-methyl, Pirimiphos-methyl, Chlorpyriphos(-ethyl), Parathion(-ethyl), Pirimiphos-ethyl, Fenthion

<sup>&</sup>lt;sup>3</sup> 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, Difenoconazol, Diflubenzuron, Dimethoat, Dimethomorph, Dimoxystrobin, DMP-formamid, 2,4-, DMP-me-formamidin, 2,4-, Epoxiconazol, Etofenprox, Etoprophos, Famoxadon, Fenamidon, Fenbuconazol, Fenhexamid, Fenoxycarb, Fenpropidin, Fenthion, Fenthion, Fenthion-oxon, Fenthion-oxon-sulfon, Fenthion-oxon-sulfoxid, Fluazifop-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, Malaoxon, 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, Quinoxyfen, Spinosyn A, Spinosyn D, Spirotetramat, Spiroxamin, Tebuconazol, Tebufenozid, Tebufenpyrad, Tepraloxydim, Terbuthylazin, Tetraconazol, Thiabendazol, Thiacloprid, Thiamethoxam, Thiophanat-methyl, Triadimefon, Triadimenol, Trichlorfon, Tricyclazol, Trifloxystrobin, Triflumizol, Triflumuron, Triticonazol, Zoxamid





of animal origin will be included). Method validation follows the Quality Control Procedures for Pesticide Residues Analysis (Document N° SANCO/2013/12751 or succeeding document).

#### C. Quality Assurance and Quality Control

### Covering the operational objectives

- (1) To ensure the development and use of high quality analytical methods across the EU-RL framework,
- (2) To maintain appropriate level of proficiency testing ensuring efficiency of control analysis methods,
- (3) To ensure the availability of scientific and technical assistance provided by the EU-RLs.
- 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 proves this 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. The PT will be performed with all NRLs and OfLs of the EU Member States and Associated States and the results will be discussed with NRLs at the workshop in Freiburg. The PT will be based on a matrix of animal origin. The PT 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 list comprises of 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. Results will be discussed at the workshop (see D.8). The schedule will be coordinated with the other pesticide EURLs and the Commission to avoid overlapping periods.
- 5. EURLs for FV, CF, SRM and AO will continue to establish defined criteria to evaluate whether a laboratory has had underperformance in the EUPTs, and if so, this laboratory could be a potential attendee 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° SAN-CO/12751/2013): Participation in meetings of the Advisory Group for the improvement of the document (at least 2) and contributions by mail contacts.

## D. Technical and Scientific Support to NRLs/EU Official labs, the Commission and Third Countries

### Covering the operational objectives

- (1) To ensure the development and use of high quality analytical methods across the EU-RL framework,
- (2) To maintain appropriate level of proficiency testing ensuring efficiency of control analysis methods,
- (3) To ensure the availability of scientific and technical assistance provided by the EU-RLs.
- 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 confirming critical results in crisis situations.
- 4. Support to the Commission for drafting of the EU coordinated working programme.
- 5. Scientific support to NRLs
  - NRLs will be supported with general scientific information and esp. in case of problems with methods for MRM-pesticides or the pesticides themselves (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 form previous EUPTs (test material service) or by exchange of samples and analysing them in parallel to show the validity of the results.
- 7. Missions to NRLs (at least one), if necessary (e.g. as an outcome of tasks C.4 and C.5).
- 8. A joint workshop of all 4 EURLs for residues of pesticides with NRL-OFL-networks in 2015 in Stuttgart, Germany (see details in the planned work programme of EURL SRM).
- 9. A trainee workshop in Freiburg in close connection with the joint workshop in Stuttgart.
- 10. At least one webinar will be organized individually or in collaboration with other EURLs for the network of NRLs and OfLs.