Fera NRL Annual Report 2014 to 2015

Report to the Food Standards Agency

May 2015




Annual Report on Operation of National Reference Laboratory (Chemical Safety in Food and Feed) by The Food and Environment Research Agency

April 2014 – March 2015

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1. Introduction

1.1. Scope

Fera (formerly the Central Science Laboratory (CSL)), was appointed as the National Reference Laboratory (NRL) for the UK for chemicals in food under a four year agreement that was effective from the 1st April 2008.

Towards the end of 2011/12, a one year extension to the chemicals in food NRL contract was established by the FSA to cover 2012/13. This was to align the renewal dates across NRL contracts.

Fera was appointed as NRL for the food and feed areas set out below for the next four year period 2013 to 2017 under a second tender exercise. There was an extension to the scope of the areas covered with feed being added to the heavy metals NRL where prior to the new contract only food was covered by Fera. The provision for feed along with food had been implicit in the mycotoxins area but this was also formalised to cover food and feed. Previously for dioxins and PCBs there were two separate contracts for food and feed but these were amalgamated.

Fera acts as the UK NRL for the following groups of chemicals that can be found in food and feed:

- mycotoxins
- heavy metals
- dioxins (PCDD/Fs) and polychlorinated biphenyls (PCBs)
- polycyclic aromatic hydrocarbons (PAHs) in food
- materials and articles in contact with food

Fera acts as proxy NRL for Malta for all of the above five contaminants areas. In addition Fera is the proxy NRL for Malta for some pesticides and veterinary drugs.

Some background information is provided below on the establishment of European Union and National Reference Laboratories along with the legislative, administrative and scientific framework within which they work, since it is within this frame that this Annual Report of activities should be viewed.

1.2. Legislative Framework

1.2.1. Regulation (EC) No 882/2004

Regulation (EC) No 882/2004 of The European Parliament and of the Council of 29th April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules provides legislation to ensure feed and food is safe and wholesome. The Regulation establishes a harmonised framework of rules for Member States to adhere to at a Community level. It also provides the legal basis for the European Commission to assess the effectiveness of national arrangements for official controls.

Regulation (EC) No 882/2004 can be found at:
http://eur-lex.europa.eu/legal-content/EN/TXT/?qid=1399359846222&uri=CELEX:02004R0882-20130701

1.2.2. Competent Authorities

The UK competent authorities responsible for official controls in respect of feed and food law are designated formally in domestic legislation that gives effect to Regulation (EC) No 882/2004 at a national level. In the UK, responsibility for official feed and food controls is held centrally. The FSA has responsibility at central Government level for the main body of feed and food law in the UK (both domestic and EU).

Regulation (EC) No 882/2004 stipulates that each Member State should establish and implement a Multi-Annual National Control Plan (MANCP). This MANCP should cover the national official feed and food, and animal health and welfare control arrangements as well as plant health controls. It provides an overview of how these authorities and other bodies work together and sets out the strategic objectives and the planned activities of the various authorities for the period of the plan. The MANCP for the United Kingdom (April 2013 to March 2015) is available from the Food Standards Agency:
www.food.gov.uk\enforcement\regulation\europeleg\feedandfood\ncpuk

In practice, responsibility for monitoring and verifying compliance (official controls) and enforcement of feed and food law is divided between central and local authorities. Most enforcement for food (including imported food) is carried out by local and port health authorities (this involves over 400 local authorities). Local Government Regulation, part of Local Government Association, is the local government central body responsible for overseeing local authority regulatory and related services in the UK. A similar pattern exists for feed enforcement in Great Britain. However, in Northern Ireland DARD (Department of Agriculture and Rural Development) has this responsibility.

1.2.3. Official Controls

These are specifically defined for the purposes of Regulation (EC) No 882/2004 at Article 2(1). They are checks carried out by the competent authorities in the Member States to monitor compliance by feed and food businesses with the requirements set out in 'feed law' and 'food law'. These checks might include inspections, audits, sampling and analysis.

Official controls also relate to the checks carried out by the European Commission's Inspection Services (e.g. Food and Veterinary Office, FVO) to assess the performance of national control authorities and national control systems.

1.2.4. The Rapid Alert System for Food and Feed (RASFF)

The Rapid Alert System for Food and Feed (RASFF) was put in place by the European Union to provide food and feed control authorities with an effective tool to exchange information about measures taken responding to serious risks detected in relation to food or feed. This exchange of information helps Member States to act more rapidly and in a coordinated manner in response to a health threat caused by food or feed. The FSA is a member of the RASFF network and is the UK contact point for RASFF notifications. The RASFF Portal website and its online searchable database of RASFF notifications provide users with a multitude of search and selection criteria.

1.2.5. Official Control Laboratories (OCLs)

Central competent authorities designate official laboratories for the purposes of chemical analysis or microbiological examination of feed or food samples taken by enforcement practitioners. Control bodies are independent third party organisations to which specific control tasks have been delegated by the competent authority. Delegated tasks might include chemical analysis, inspection, or sampling. The competent authority retains the responsibility for the work and for taking any formal enforcement action should non-compliance be found. Control bodies are subject to audit or inspection by the competent authorities in respect of the control tasks delegated to them.

Designation may only be granted if the laboratory meets certain standards (i.e. is accredited to the European Standards specified in Article 12 of Regulation (EC) No 882/2004). In the UK, accreditation is undertaken by the United Kingdom Accreditation Service (UKAS). A list of official feed and food laboratories that undertake chemical analysis or microbiological examination of samples on behalf of local authorities and district councils is published on the Food Standards Agency website. The Association of Public Analysts (APA) website also gives contact details for official control laboratories:

http://www.publicanalyst.com/

1.2.6. European Union Reference Laboratories (EURLs)

EURLs are appointed by the Commission. Regulation (EC) No 776/2006 that amends Regulation (EC) No 882/2004 pertains as regards EURLs.

EURLs assist the harmonisation process by increasing the current analytical scope throughout the EU in quantity and quality of the results. Summarising Article 32 of Regulation (EC) No 882/2004, EURLs for feed and food are responsible for:

a) Providing NRLs with details of analytical methods, including reference methods;
b) Coordinating application by the NRLs of the methods referred to in (a), in particular by organising comparative testing and by ensuring an appropriate follow-up of such comparative testing in accordance with internationally accepted protocols, when available;
c) Coordinating, within their area of competence, practical arrangements needed to apply new analytical methods and informing NRLs of advances in this field;
d) Conducting initial and further training courses for the benefit of staff from NRLs and of experts from developing countries;
e) Providing scientific and technical assistance to the Commission, especially in cases where Member States contest the results of analyses.

Details of the EURLs relevant to this report are given in Appendix 1.

1.2.7. National Reference Laboratories (NRLs)

The European Commission has created a network of NRLs coordinated by the EURLs. This network of laboratories is responsible for setting up EU-wide standards for routine procedures and reliable testing methods in the areas of feed and food and animal health. Each Member State must designate an NRL to correspond to each EURL, although the NRL does not have to be located in the designating Member State. These laboratories must collaborate with the EURL in their particular area of expertise and disseminate nationally the information provided by the EURL. In addition, they provide scientific and technical assistance to the national competent authorities.

According to Article 33 of Regulation (EC) No 882/2004, duties of the NRLs include collaboration with the EURLs and coordination of the activities of official laboratories, organisation of comparative tests and ensuring appropriate follow-up, dissemination to the competent authority and official national laboratories information that the EURLs supply, provision of scientific and technical assistance.

The NRL role:
It is a requirement of Regulation (EC) No 882/2004 that NRLs:

a) Collaborate with the EURL in their area of competence;
b) Coordinate, for their area of competence, the activities of official laboratories responsible for the analysis of samples;
c) Organise comparative tests between the official national laboratories and ensure an appropriate follow-up of such comparative testing;
d) Ensure the dissemination to the competent authority and official national laboratories of information that the EURL supplies;
e) Provide scientific and technical assistance to the competent authority for the implementation of coordinated control plans adopted in accordance with Article 53 (coordinated control plans).

EURLs establish a network between EURLs, NRLs and OCLs. The overall objective of the EURLs and NRLs is to improve the quality, accuracy and comparability of the results at OCLs. Full details of UK NRLs can be found in the Appendices of the UK Multi-Annual National Control Plan (see 1.2.2). Contact information for the individual UK NRLs operated by Fera are given in Appendix 2.

2. Fera National Reference Laboratory (NRL)

2.1. General and Contract Activities

An open and standing invitation from Fera to OCLs inviting them to participate in Fera NRL visits to them is reviewed regularly and is available on the NRL website http://fera.co.uk/about-us/national-reference-laboratory/

2.2. Contact with the FSA

2.2.1. Annual Report

The previous Annual Report of Fera NRL activities during 2013 to 2014 was produced and is available on the NRL website.

2.2.2. EFSA Article 36 Workshop

Susan MacDonald and Claire Charlton from Fera attended a joint workshop organised between the FSA and EFSA on Article 36 grant funded projects. Susan MacDonald gave a presentation on experiences of working on Article 36 funded projects.

2.2.3. T-2 and HT-2 Toxin Stakeholder Meeting

Susan MacDonald and Phil Jennings (Fera) attended the Stakeholder meeting to discuss the T-2 and HT-2 toxin in UK oats results from the 2014 harvest.

2.3. Steering Group Committee

The Steering Group Committee exists to maintain an overview of the NRL activities and ensure good communication with the OCLs. The Committee comprises Public Analysts, and staff from the FSA and Fera. The Public Analysts are seen as key members of the Steering Group Committee as end users of the results of much of the work of the NRL.

2.3.1. Eighth Steering Group Committee Meeting

This was held at Fera on 18th September 2014. The meeting was attended by Susan MacDonald, Martin Rose, Malcolm Driffield, Malcolm Baxter, Joe Holland, and Irene Leon all from Fera. David Mortimer attended from the FSA, and William Munro (FSA Scotland) participated remotely via telephone. This meeting was held in a modified format with representatives invited from all the OCLs. The following OCL representatives were present:
Steve Appleton, Aberdeen Scientific Services Laboratory; Duncan Campbell, West Yorkshire Analytical Services; Ron Ennion, Public Analyst Scientific Services; Jen Green, Hampshire Scientific Services (representing Kent Scientific Services); Nigel Payne, Public Analyst Scientific Services; John Robinson, Minton, Treharne and Davies Ltd; Andrew Smith, Lancashire County Scientific Services.

An update on each area was given including EURL activities, legislation, methodology, training and the NRL website. Future training needs of the OCLs and possible activities the NRL could undertake to meet these were also discussed. It was a useful meeting and several good recommendations came from the OCLs. They agreed that the communications we use, i.e. mostly email were the most effective way to contact them.

They would like more notice of EURL PTs, and they also asked if the NRL and the training provided could be more closely linked to the Annual Monitoring Plans, so the NRL could provide advice, training and prepare reference materials ahead of the work being conducted in the plan. This would require the NRLs being more closely involved with the plan, and being informed about its contents to allow time to offer assistance to the OCLs. The Food Standards Agency representative agreed this was a good proposal and all parties agreed to work towards this in the future.

2.4. Contact with Official Control Laboratories (OCLs)

2.4.1. OCL Contact Details Check

All OCLs were contacted and asked to confirm or amend their contact details. The contact list held by the NRL was updated accordingly.

2.4.2. OCL Accreditation Check

The accreditation status of OCLs was checked. This was done by checking UKAS Testing Laboratories Schedules for each laboratory. Information was collected and tabulated for the five areas covered under the Fera NRL contract (food and feed). Information on analytical capability was also collected and changes including additions and removals were noted.

Some differences noted since August 2012 include:

- 2 laboratories have closed (Somerset Scientific Services and Cardiff Scientific Services)
- 3 laboratories no longer obviously quoting the use of APA VEMS
- Public Analyst Scientific Services Ltd have changed their UKAS laboratory codes (Wolverhampton & Norwich)
- Mycotoxins (food): 1 laboratory has dropped Aflatoxin M_1, but 6 laboratories have increased mycotoxins testing to different extents
- There have been small changes to metals tested
- There was no obvious mention of PAHs or dioxins
- Food Contact Materials (FCM): 1 laboratory has dropped the test they did; 1 laboratory has become more specific in the types of overall migration tested; 2 laboratories have increased testing to offer PAAs and formaldehyde from kitchenware.

This information was given to the FSA in March 2015.

2.4.3. Advice

A visit was made to an OCL to provide support during an FVO inspection visit concerning eggs.

2.5. Training Activities

2.5.1. MChemA Course

Susan MacDonald and Emma Bradley gave presentations at the MChemA training course on 30th April 2014 at the University of Reading.

2.5.2. Future Activities

Further activities and training identified are:

- Measurement Uncertainty
- More semi-reference materials and associated semi-formal PTs along the lines as previously provided by the NRL for OCLs.

Training was discussed in detail at the Steering Group Meeting. It was agreed that the preparation and circulation of a rice material containing metals would be the most useful material. It was agreed to include As, Pb, Cd and Hg as more laboratories would be able to participate in the PT and could use the material as an aid to setting up a method for arsenic/inorganic arsenic in the future. A proposal and costing were prepared subsequently and sent to the FSA for approval.

2.6. Website

The Fera NRL website is seen as a key part of the NRL function, in terms of dissemination, as a source of resources and also as a means of communication and building a network of official control laboratories within the UK.

On 1st November 2013, Fera launched a new website for its corporate and agri-food commercial services. This is also home to the Fera NRL website:

http://fera.co.uk/about-us/national-reference-laboratory//

The content associated with each function is reviewed and updated as necessary in the interest of continual improvement. The website was reviewed at the Steering Group Meeting, including the additional alert notification information that is available on the mycotoxins page. This was thought to be useful, and may be considered for other areas.

2.7. Support/ Contact with Other NRLs

There has been regular contact with a number of NRLs across the year. Advice has particularly been provided for mycotoxins and materials and articles in contact with food covering a variety of topics.

2.7.1. Proficiency Tests

Fera were invited and participated in a Proficiency Test (PT) being organised by RIKILT for ochratoxin A in pig kidney. Fera was invited to participate in several PT studies run by other NRLs (The Netherlands and Belgium).

2.7.2. Workshops and Working Groups

Four EURL Workshops were held in this period; mycotoxins, PAHs, Food Contact Materials and dioxins and PCBs. More information is given in each of the sections.

Fera is a member of a working group on criteria for Liquid Chromatography-Mass Spectrometry LC-MS methods for mycotoxin analysis that produced a document in this period, as well as working groups for dioxins and PCBs and Food Contact Materials.

2.7.3. CEN Working Groups

There was contact with a number of NRLs at several CEN Working Groups. Fera is a member of CEN TC327 WG5, CEN TC275 WG5, WG10 and WG13.

In the Food Contact Materials area a workshop on ceramics was held in which Fera had a key role.

2.8. Future Activities

The following are planned as general activities for 2015-16:

- Participation in EURL PTs and circulation of information to OCLs when appropriate.
- Visits to OCLs will be planned as requested.
- Steering Group Committee Meeting.
- Preparation of reference material for As in rice
- PT for As in rice and training workshop for metals

3. Mycotoxins NRL (Susan MacDonald)

3.1. Introduction

Mycotoxins are secondary metabolites produced by some moulds. They can occur in a wide range of foods, often with no visible signs of mould spoilage to the food. They have a wide range of chemical properties and toxicities to humans and food-producing animals. Exposure to some mycotoxins is controlled through European and National Legislation. The Contaminants in Food (England) Regulations 2013 provide for the enforcement of European Commission Regulation (EC) No 1881/2006, setting maximum levels for certain contaminants in foodstuffs. There are similar domestic Regulations for Scotland, Wales and Northern Ireland. Methods to be used for sampling and analysis for enforcement purposes are prescribed in Commission Regulation (EC) No 401/2006 and its subsequent amendment Commission Regulation (EU) No 178/2010.

There have been a number of changes in European legislation relating to mycotoxins in the past year, these are summarised below.

3.1.1. Regulations

Regulation (EC) No 1881/2006 was amended to include a maximum level of 2000 µg/kg citrinin in food supplements based on rice fermented with red yeast *Monascus purpureus*, the level applied from 1st April 2014. The maximum level is to be reviewed before 1st January 2016 to consider information on exposure to citrinin from other foodstuffs and updated information on the toxicity of citrinin in particular as regards carcinogenicity and genotoxicity.

In addition, Regulation (EC) No 401/2006 on sampling and analysis for the control of the levels of mycotoxins was amended with respect to the sampling of large lots, spices and food supplements, performance criteria for T-2, HT-2 toxin and citrinin and screening methods of analysis; the amendments applied from 1st April 2014.

Regulation (EC) No 594/2012, amending Commission Regulation (EC) No 1881/2006, set the maximum levels (MLs) for ochratoxin A in spices and mixtures of spices at 15µg/kg. For paprika (*Capsicum spp.*) a higher limit of 30µg/kg was allowed until January to allow Good Manufacturing Processes (GMP) to be introduced to meet the lower limit. Data has shown that many producers still cannot meet the lower limit and so a request has been made to retain the higher level.

Commission Implementing Regulation (EU) No 884/2014 was published on 13th August 2014. This regulation stipulates special conditions about the import of certain food and feed from third countries due to a risk from aflatoxin contamination. It repeals Regulation (EC) No 1152/2009. Conditions are set out on a number of issues including the frequency of checks of certain products from specified countries and how often results of official controls have to be reported to the Commission. This Regulation is now in force.

3.2. Activities of the EURL-NRL Network on Mycotoxins

The Joint Research Centre (JRC) Institute for Reference Materials and Measurements (IRMM) is the EURL for mycotoxins and is located in Geel, Belgium. It works together with appointed NRLs of the EU Member States. The EURL for mycotoxins aims to facilitate the implementation of European legislation related to monitoring of mycotoxins in food of plant origin and animal feed. The EURL website can be found at:

http://irmm.jrc.ec.europa.eu/EURLs/eurl_mycotoxins/Pages/index.aspx

3.2.1. Contact with the EURL

The main contact with the EURL related to two PTs for zearalenone in maize oil and aflatoxin B1 in copra that they organised in this period. In addition to NRLs, OCLs were able to take part so there was correspondence regarding that. In addition the EURL Workshop was held in October 2014. The EURL invited laboratories to take part in some method validation studies. Fera replied to express an interest in taking part in a method validation study for *Alternaria* Toxins and a proposed EURL ring trial on screening methods for mycotoxins. An EURL online survey on LOD and LOQ was completed in January 2015.

3.2.2. EURL Workshop

The Workshop was held at the IRMM JRC, Geel, Belgium in October 2014. A note of the meeting has been prepared and was sent to the FSA with documents from the meeting. The topics discussed were the results of the PTs from 2014, follow up from the PTs from 2013, presentations on a method validation study for *Alternaria* toxins, working group reports on sample preparation, criteria for LC-MS methods as well as discussions on proposals for LOD/LOQ estimation, validation of screening methods and a harmonisation of the approach on calculation of measurement uncertainty.

Fera is a member of two working groups, LC-MS criteria and sample preparation. The LC-MS group produced a document before the meeting that was discussed. The sample preparation working group agreed to carry on with more work.

The work programme for 2015 was also presented. The PT for 2015 will be citrinin, in two different samples possibly two red yeast rice. The working groups on sample preparation and LC-MS will continue, there will be an evaluation of some rapid test kits for DON, training on LC-MS will be provided for 6 NRLs, and last year's PTs will be followed up. The usual activities including a workshop, representing and advising the Commission and supporting CEN activities will also continue.

3.3. Contact with Other NRLs

3.3.1. General Contact and Collaboration

There was a lot of contact with RIKILT on a number of topics including participation in their PT, and collaboration for various projects including EFSA projects on sterigmatocystin and tropane alkaloids.

3.3.2. Method Criteria Working Group for the EURL-NRL network

Information about method parameters such as tolerances for ion ratios, retention times, recovery etc. was requested from laboratories in the EURL-NRL network. Fera collated

information from different types of LC-MS/MS batches, i.e. using solvent calibration; matrix matched calibration and 13C internal standard addition. Ion ratios, retention time drift and other information was collated and sent to RIKILT. A guidance document suggesting criteria that can be used for LC-MS/MS analysis for mycotoxins was produced.
Several NRLs were involved; the group was led by RIKILT. This guidance document was an agenda item at the EURL Workshop.

3.3.3. Sample Preparation Working Group

Fera was a member of the sample preparation working group; this was led by the Swedish NRL. Following the EURL Workshop the group prepared some recommendations to help standardise approaches in sample preparation.

3.3.4. CEN TC327 WG5

As NRL for mycotoxins in animal feed, Fera requested nomination to become a member of the newly formed CEN Animal Feed Working Group 5 for mycotoxins and natural toxins. Susan MacDonald was accepted and became a member of this CEN working group. The convenor is Hans Mol from RIKILT (NRL, The Netherlands). The meeting was also attended by the EURL, LGC and representatives from Germany, the Czech Republic, Belgium, Austria and Ireland. Following the meeting, feedback was sent to FSA.

3.3.5. CEN Mandate M521 / 522

An invitation to participate in an EURL pretrial for a collaborative study for multimycotoxins in animal feed (CEN Mandate M521/522) was received in March 2015. Participation in this study was agreed.

3.3.6. CEN Mandate M520

Fera was awarded two projects under this mandate, these were a method for ergot alkaloids in cereals and cereal products and a method for aflatoxins in spices included in Regulation (EC) No 1881/2006 other than paprika and capsicum spp. In addition the Mandate M520 has awarded a range of method validation projects to a number of European laboratories including the EURL. The EURL will develop and validate a method for *Alternaria* toxins. Other methods that will be developed and validated include: Citrinin in red yeast rice; Ochratoxin A in meat products; Ochratoxin A in spices and cocoa; Phomopsins in foods; Multitoxin methods by LC-MS (a screening method and a quantitative method) and a method for Fusarium toxins by LC-MS/MS.

3.3.7. CEN TC275 WG5

TC275 WG5 is for horizontal methods of analysis of Biotoxins in food, several NRLs are also members of this Working Group. A meeting of CEN TC275 WG5 was held in Brussels on 10th to 11th July 2014, where all new project leaders under Mandate M520 attended to discuss their proposed work. A note of the meeting was prepared and sent to the FSA.

3.4. Contact with the Competent Authority

3.4.1. General Advice

Information was provided about the method performance for analysis of ergot alkaloids in cereals and cereal products. Specifically, information was requested about measurement

uncertainty. This was provided along with a copy of a publication by Fera about method validation for ergot alkaloids.
Advice was provided to the FSA for a Codex Electronic Working group on how to handle measurement uncertainty for multiple analytes. A presentation and information from an EURL Workshop was forwarded to the FSA.

3.4.2. T-2 and HT-2 toxin Stakeholder Meetings

Susan MacDonald attended two Stakeholder meetings about T-2 and HT-2 toxins (one via telephone). Information about the data collection exercise to collect data on T-2 and HT-2 toxin in response to EU Recommendation 165/2013 was discussed, as well as some initial results from industry that had been found so far this year. Results from 2014 results were reviewed and a plan on how investigations should be conducted was discussed.

3.4.3. AMC Animal Feed Sub Committee

Susan MacDonald attended the AMC Animal Feed Sub Committee meeting chaired by the FSA and held in London in November 2014. The possible future role and tasks the committee could undertake were discussed.

3.5. Interlaboratory Comparisons

3.5.1. Aflatoxin B_1 in Copra

This PT was organised by the EURL for the NRL network but was also open to OCLs. Therefore an email invitation was sent to all UK OCLs offering them registration paid by the FSA. Six UK OCLs registered to participate in the PT. Samples were received in April, and results were reported in June 2014. Fera obtained satisfactory z-scores. Four OCLs participated and all obtained satisfactory z-scores. Some OCL labs had high zeta scores showing that there may be an issue with their measurement uncertainty calculation, however the overall performance was good and no follow up action was required. In January 2015, the feedback form was completed and returned to the EURL.

3.5.2. Determination of Ochratoxin A in Pig Kidney

This was organised by the Dutch NRL, RIKILT. The email advertising the PT was circulated to all UK OCLs and they were asked to register directly with RIKILT if interested. An enquiry was received from an OCL requesting a method as they were considering participating. Results were submitted electronically in November 2014. The report of the study was received in March 2015, Fera's overall performance classified as 'optimal'.

3.5.3. Determination of Multi Toxins in Cereal

Fera was invited to participate in a multi-mycotoxin PT organized by ISPA-CNR (Italy) in the framework of the project "New Strategies for Improvement of Food Safety: Prevention, Control, Correction" (S.I.Mi.S.A. PON_02_00186_3417512, project of the Italian Ministry of Education, Universities and Research) and promoted by the MoniQA Association (Monitoring and Quality Assurance in the Total Food Supply Chain, www.moniqa.org).

The main objective of the PT was to provide interested laboratories with an opportunity to test their multi-mycotoxin methods and to compare their results with those of other laboratories. The use of LC-MS(MS) methods, although not strictly required, was highly recommended.

Fera's results were submitted in July 2014. Twenty one laboratories had registered and eighteen labs returned results. Fera submitted two sets of results, one using immunoaffinity column clean-up and the other using a screening method that did not use any clean-up. For the main toxins, the Fera results were satisfactory for both methods. In some cases no z-score was given for Fera results as less than values had been reported where the samples were below the reporting limit of the method. Due to some data being excluded, combined with a number of results being reported as 'less than' some data sets were too small to allow proper data evaluation. Comments on the report were returned to the co-ordinator.

3.5.4. EURL PT Zearalenone in Maize Oil

This PT was organised by the EURL for the NRL network but was also open to OCLs. An email invitation was sent to all UK OCLs offering them registration paid by the FSA. Three UK OCLs registered to participate in the PT. There is no available standard method for this analysis, however a German laboratory (ILCTR, LUA, Trier) is undertaking a method validation study. They were contacted and kindly provided a copy of the SOP, this was translated from German and supplied to the UK OCLs that were taking part in the PT. Sample results were submitted in July 2014.

As a questionable result had been obtained by Fera for one test material, we were given the option of carrying out a Root Cause Analysis (RCA) and preparing a Corrective Action Plan (CAP) to be submitted to the EURL or participating in a follow up PT organised by the EURL. As this PT had been the first time that Fera had carried out this method it was decided to participate in the follow up PT to gain more experience and because there would be very little to review in the RCA.

Samples were received in February 2015 with results submitted on 16th March 2015. The report of the follow up was received at the end of March and showed that Fera had a questionable result. The EURL advised that root cause analysis should be completed to determine the cause, and offered support if required. Further analysis was carried out at Fera to establish the method and validate it in-house.

3.6. Method Validation Studies

3.6.1. Multitoxin in Animal Feed Method by LC-MS/MS - Pretrial

This method validation study was run by the EURL as part of Mandate M521 for CEN TC 327/WG5. Fera agreed to participate in the pre-trial in March 2015, with ad hoc funding.

3.6.2. Pyrrolizidine Alkaloids in Herbal Products and Honey by SPE and LC-MS/MS

Fera participated in Phase 2, the full method validation study for pyrrolizidine alkaloids (PAs) organised by BfR, Germany. Samples were received in September 2013 and results were submitted in November 2013. A report was circulated in October 2014. The method validation was successful and Fera's results were included in the data evaluation.

3.6.3. Alternaria Toxins - Pretrial

Under CEN Mandate 520 the EURL will develop and validate a method for *Alternaria* toxins. In February 2015, the SOP for *Alternaria* toxin pretrial was reviewed. Analytical standards were also received in order to establish MS conditions.

3.7. Supporting the UK Official Control Laboratories

3.7.1. Advice

Advice was given to OCLs on the following topics:

- OCLs emailed to inform them of PTs run by EURL and RIKILT
- SOP for zearalenone in maize oil supplied to OCLs taking part in the EURL PT.
- Presentation on Contaminants given at MChemA Training course at Reading University
- Advice on sample preparation by the slurry method was given.
- Method for ochratoxin A in pig kidney was supplied to one OCL who was interested in taking part in the RIKILT PT

3.7.2. Training

A presentation on Contaminants was given at the MChemA training course at Reading University in April 2014.

4. NRL Heavy Metals

Malcolm Baxter

4.1. Introduction

Contaminants such as heavy metals are substances that have not been intentionally added to food. These substances may be present in food as a result of the various stages of its production, packaging, transport or holding. They also might result from environmental contamination. Since contamination generally has a negative impact on the quality of food and may imply a risk to human health, European legislation lays down maximum allowed limits in foodstuffs. EU regulations cover the following heavy metals: cadmium, lead, mercury and inorganic tin. Legislation can be found in European Commission Regulation (EC) No 1881/2006, amended by Commission Regulation (EU) No 420/2011.

Sampling methods and the methods of analysis for the official control of the levels of cadmium, lead, mercury and inorganic tin are given in Commission Regulation (EC) No 333/2007.

4.1.1. Regulations - On-going Legislation for Food

Arsenic:

Following Stakeholder discussion on rice and edible fats and oils, the expert committee proposed differentiated Maximum Levels (MLs) for rice, (white versus brown, puffed rice products and rice for infants) but decided no ML was required for oils and fats. Issues to be discussed further by the expert committee were (a) a specific ML for parboiled rice (the process can increase inorganic arsenic levels in the rice) and (b) rice wafers, brown versus white rice content.

At present the following suggested levels are still under discussion for inorganic arsenic:

3.5	Description	Sum of As (III) and As (V)
3.5.1	Milled rice and parboiled rice	0.2 mg/kg
3.5.2	Puffed rice, rice wafer, rice cracker and rice doughnut derived from milled rice	0.25 mg/kg
3.5.3	Husked (brown) rice	0.25 mg/kg
3.5.4	Puffed rice, rice wafer, rice cracker and rice doughnut derived from husked (brown) rice	0.3 mg/kg
3.5.5	Rice for infants and young children other than husked (brown) rice	0.1 mg/kg

Cadmium:

EFSA scientific opinion 2009 established a lower tolerable weekly intake (TWI) of 2.5 µg/kg b.w. (previously 7 µg/kg b.w.). It concluded this TWI was exceeded by certain population groups and recommended exposure be reduced at a population level. Main contributors to dietary exposure for all age groups were potatoes, cereals, vegetables, chocolate and cocoa products. Ready-to-eat meals, infant formulae, milk and dairy products were a source for infants. A review of Regulation (EC) No 1881/2006 focused on

commodities for which no MLs exist. Chocolate and cocoa products would have differentiated MLs depending on the percentage of total dry cocoa solids, applicable from 1/1/2019. Baby foods would have differentiated MLs for powdered versus liquid formula (from milk and soya) and processed cereal based foods for infants and young children, applicable from 1/1/2015. Other various adjustments for specific fish and vegetables were also given. These are all contained in Commission Regulation (EU) No 488/2014 of 12 May 2014, amending Regulation (EC) No 1881/2006.

Lead

The EFSA CONTAM Opinion (March 2010) identified a need to reduce exposure due to concern over neurodevelopmental effects in young children. Main contributors were similar to cadmium along with fruit and fruit products, fruit juices. Changes have been suggested after Stakeholder consultation with similar differentiation between solid and liquid formulae. New MLs suggested for processed cereal based foods for infants and young children and other baby foods (0.1 mg/kg) and honey (0.1 mg/kg).

Mercury

The EFSA CONTAM Opinion (November 2012) on the risk for public health related to the presence of mercury and methylmercury in food issued a TWI for inorganic mercury of 4 µg/kg b.w. and for methylmercury 1.3 µg/kg b.w. Assessment by EFSA on the beneficial effects of fish consumption is on-going, to try to counteract the negative slant methylmercury has on this matrix.

4.1.2. Regulations - On-going Legislation for Feed

Regulation (EU) No 744/2012:

Recent and foreseen changes to this Regulation focused on matrices or additives that are essentially the same but had different MLs, making alignments of MLs where possible. There was also an increase of the ML for arsenic in complete feed for pet animals containing fish, other aquatic animals and products derived thereof and/or seaweed meal and feed materials derived from seaweed (10 mg/kg aligned with complete feed for fish and fur animals). A footnote was added “upon request of competent authority, the responsible operator must perform an analysis to demonstrate that the content of inorganic arsenic is lower than 2 ppm”.

Regulation (EU) No 1275/2013:

Specific higher MLs for arsenic, cadmium and lead in long-term supply formulations of feed for particular nutritional purposes with a concentration of trace elements higher than 100 times the established maximum content of complete feed (higher than normal complementary mineral feed: arsenic 12 ⇨ 30 mg/kg, cadmium 5 ⇨ 15 mg/kg, lead 15 ⇨ 60 mg/kg). There was also an increase of ML for arsenic in feed additive ferrous carbonate.

A significant difference has been identified by the EURL-HM between the analytical results obtained by the application of different extraction methods currently used to determine lead in kaolinitic clay and feed containing kaolinitic clay. Before, no significant differences were observed in mineral feed using different extraction methods. The MLs of heavy metals in feed relate to “an analytical determination of lead, whereby extraction is performed in nitric acid (5 % w/w) for 30 minutes at boiling temperature”. In view of this finding, a footnote has been re-introduced to provide for the use of this specific method of

extraction (or method of similar extraction efficiency) when determining lead in kaolinitic clay.

The following are to be voted at the next standing committee. Increase of ML for arsenic, fluorine and lead in calcareous marine shells and change of ML of mercury for fish, other aquatic animals and products derived thereof intended for the production of compound feed for dogs, cats, ornamental fish and fur animals (ML of 0.5 mg/kg).

Future trend to will be to introduce MLs for inorganic arsenic in feed but these will stay with total arsenic at present.

Finally, Regulation (EC) No 152/2009 sampling and method of analysis has been superseded by Regulation (EU) No 691/2013 which entered into force January 2014.

4.2. Activities of the EURL-NRL Network

4.2.1. EURL Workshop

The 9th EURL- HM Workshop was held in Brussels (9th September 2014). The meeting covered the following topics:

4.2.2. 2014 Activities & WP 2015

A general overview of EURL activities for 2014 was presented. Matrices selected for 2015 will be dark chocolate and a mineral feed containing kaolinitic clay. The former in response to a forthcoming regulation for cadmium and the latter due to method issues with feed containing the clay. Suggestions from NRLs for 2016 included cereal, fruit/juices, tea, bakery commodities, potatoes/root vegetables, vegetable feed (from palm kernel expellant), vegetable oil for feed, and various sea foods. Our request was for speciation, inorganic arsenic and methylmercury which could help us with testing/training of OCL labs.

4.2.3. General Information About EURL Website

The location of the EURL website for NRL information and documentation will be moving onto another platform called CIRCABC (communication and information resource centre for administrations, businesses and citizens). No definite timescale has been given for this move but is likely to be within a year.

4.2.4. Proficiency Tests Held in 2014

IMEP-118: Determination of Total As, Cd, Pb, Hg, Sn and iAs in Canned Food
The sample supplied was whole peas in brine to represent a real sample. One aim of the PT was to gather information about how NRLs dealt with the sample, performing the analysis on drained solid or a solid liquid composite. At present there is no guidance at an EU level on how to treat such samples and the PT highlighted this quandary. Nominally a 50:50 split in the choice of reporting was observed.

IMEP-119: Determination of Total As, Cd, Pb, and Hg in Vegetable Feed
The sample supplied was an animal feed made from Alfalfa-meal. Vegetable feed is a relatively easy matrix to analyse. Only mercury appeared to create any problems for some participants, probably due to the relatively low concentration.

4.2.5. Selenium in Feed

A short talk given by Dr Timo Kapp, of BVL described the analytical problems faced when discerning organic from total selenium (Se). The background to this subject, selenium is an essential element with sources from fish, meat, eggs, legume, nuts, mushrooms etc. Especially in animal production, the feed is often supplemented with inorganic sources of Se but in recent years organic sources have been used (selenomethionine, selenium yeast etc.). EFSA raised concerns about higher carry over rates caused by the organic Se forms as Se has a small therapeutic width. Therefore the supplementation of organic selenium forms is limited to 0.2 mg/kg feeding stuff (Commission Implementing Regulation (EU) No 427/2013). The current analytical approach involves enzyme digestion, ultrafiltration and LC-ICP-MS. To date, the method was able to quantify authorised additives but the lack of standard substances impeded the analysis. Questions raised were: (a) how should Official Laboratories control the content of organic selenium with no official method available (b) what to do with natural organic selenium contents above 0.2 mg/kg (c) how to discriminate between added and natural organic selenium.

4.2.6. Statistical Experimental Design (SED)

In essence, SED is a plan to get the maximum information from the minimum number of experiments. The presentation explained ways to reduce the number of measurements required to achieve the same outcome as an experiment without this statistical reduction applied. An interesting topic but would require some thought before being able to apply the technique successfully.

A date for the next EURL-HM Workshop was set for 28/29th September 2015 in Brussels.

4.3. Interlaboratory Comparisons

4.3.1. IMEP-118: Determination of Total As, Cd, Pb, Hg, Sn and iAs in Canned Food

The material supplied was peas in liquor. Fera analysed the peas and liquor separately, reporting a composite value after studying regulations. There was no reference to draining off liquid in regulations and the only specific reference was found in (EC) 333/2007, Part B, Sampling methods, that stated “The maximum levels of inorganic tin apply to the contents of each can”, hence our decision. The EURL chose the material to highlight this very dilemma. The material was characterised by the EURL for both drained product and as a composite to cover both options. Fera results were satisfactory for all analytes. No scores were issued for mercury as the concentration was too low for a meaningful value to be estimated.

4.3.2. IMEP-119: Determination of Total As, Cd, Pb, and Hg in Vegetable Feed

The sample supplied was an animal feed made from Alfalfa-meal. No issues were encountered when analysing this material. Fera results were satisfactory for all analytes.

4.3.3. NRL04, Inorganic Arsenic and Other Elements in Brown Rice

As a forerunner to a future speciation training workshop, a proposal to prepare a brown rice material (NRL04) for iAs and other elements was suggested. The material could then be used as a semi-reference material by OCLs after the PT. The PT would indicate the current status of this analysis amongst the OCLs. A suitable matrix was identified in the FAPAS programme and after discussion it was agreed to combine our NRL PT with the FAPAS round. The benefits would be reduced preparation costs and a robust statistical

analysis of the matrix would also be obtained. The material has been prepared and a stability trial conducted.

4.3.4. EURL-HM-20 and EURL-HM-21 (Food and Feed Matrices)

Fera has received notification and registered for two 2015 PT materials; EURL-HM-20 for the determination of total As, Cd, Pb, Hg and iAs in chocolate, EURL-HM-21 for the determination of total As, Cd, Hg, iAs and extractable Pb in kaolinitic clay. Participation is open to OCLs too, so Fera has circulated the details and actively encouraged their participation.

4.4. Method Validation Studies

4.4.1. CEN_BVL Collaborative Trial "Aluminium in Food"

A ring-test was organised to check the ruggedness of an analytical method for the determination of aluminium in foodstuffs. Participants were supplied with a method description, reporting template and eight test samples. Instructions were to follow the method as closely as possible using the laboratories own equipment, thereby testing the method in a more general situation. We have completed all the tasks and await the outcome of this trial.

4.4.2. CEN Meeting

Malcolm Baxter attended the 24th meeting of CEN/TC 275/WG10 in London as a technical expert (23rd May 2014). The main topics discussed included the following: Determination of inorganic arsenic in food of marine and plant origin and Determination of methylmercury in food of marine origin: WI 00275237. Document FprCEN/TS 16731 “Determination of hydride-reactive arsenic compounds in rice by (HG-AAS) was awaiting CEN Enquiry (24th April 2014 to 24th June 2014). FprEN 13805 was awaiting formal CEN vote (1st May 2014 to 1st July 2014).

Other items discussed were NWIP – Determination of minerals using ICP-OES, and systematic review of three standards (EN 15763, EN 15764 and EN 15765). The majority vote from members was in favour of no change therefore the three standards were confirmed for a further 5 years.

A method for Determination of MeHg in seafood by elemental mercury analysis was put forward by JRC-IRMM for consideration as a new working item. Due to the lack of time at the meeting this method will now be circulated for comments at the next WG10 meeting. Germany proposed a method for determining mercury using cold vapour AFS as a future project. They are currently undertaking pre-work for this method and invited interested laboratories to participate. A ring test will be carried out later in the year where more laboratories will be approached. The next meeting will be held sometime in April/May 2015 in Berlin once the mandated projects have gone through CEN Enquiry.

4.5 Supporting the UK Official Control Laboratories (OCLs)

4.5.1. Advice

An OCL sought advice/information on replacement certified reference materials for food/feed analysis. Having looked through the literature, a few suggestions were offered for their consideration.

An OCL requested information on a number of topics pertaining to inorganic arsenic analysis (methods, forthcoming Regulations, possible training events, etc.). They have been furnished with a copy of the method used at Fera and the proposed Regulations concerning rice and rice products.

4.5.2. Proficiency Testing of OCLs

As part of our NRL duties, Fera encouraged the OCLs to participate in the EURL PTs as they were both “open” rounds. In response, 8 laboratories took part in IMEP-118 and 9 in IMEP-119. At present, draft reports for both PTs and the participant codes and have been received and the findings will be collated into a suitable report for the Competent Authority (FSA).

5. NRL Dioxins and PCBs

Martin Rose

5.1. Introduction

Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), collectively referred to as dioxins (PCDD/Fs), along with polychlorinated biphenyls (PCBs) are a group of toxic and persistent chemicals. Their effects on human health and on the environment include dermal toxicity, immunotoxicity, reproductive effects and teratogenicity, endocrine disrupting effects and carcinogenicity. There is considerable public, scientific and regulatory concern over the negative effects on human health and on the environment of long-term exposure to even the smallest amounts of dioxins and PCBs. Over the past two decades the European Commission has proposed wide ranging legislation aimed at directly or indirectly reducing the release of these compounds into the environment, with the objective of reducing human exposure and protecting human health and the environment.

5.2. Activities of the EURL-NRL Network on Dioxins and PCBs

The European Union Reference Laboratory (EURL) for Dioxins and PCBs in Feed and Food is the State Institute for Chemical and Veterinary Analysis of Food (Chemisches und Veterinäruntersuchungsamt, CVUA) Freiburg, Germany. The EURL website can be found at:

http://www.crl-freiburg.eu/dioxin/index.html

The NRL network for dioxins and PCBs has been asked by the Commission to cover brominated flame retardants (BFRs) on an interim basis until a permanent solution can be found.

5.2.1. Contact with the EURL - Workshop of EURL-NRL Network

There were two meetings of the EURL-NRL network during the year. The first was hosted by the French NRL and took place during May 2014. The second was hosted by the EURL in Freiburg and took place in December 2014.

Alongside both of these meetings, the working group on measurement uncertainty also held meetings. Fera is an active participant in meetings of this working group.

5.2.2. Regulations (EU) No 589/2014 and 709/2014

Some amendments were made during the course of the year, especially with respect to bio-analytical methods. Many were editorial in nature, and to make it clear that bioanalytical methods such as CALUX can only be used to screen compliant samples – suspect non-compliant samples must be subject to confirmatory chemical analysis. There was also a need to remove confusion between maximum and action limits, limits for PCDD/Fs and PCBs and for PCDD/Fs only, and between TEQ (calculated using WHO TEFs) and BEQ (calculated using bioanalytical response) terminology.

5.2.3. ISO 17025 Accreditation v Commission Regulation (EU) No 589/2014

The risk of reporting unreliable analytical results by an accredited laboratory that does not comply with EU analytical criteria was identified as a real possibility because of the confusion of the role and overlap of both requirements. Meeting ISO 17025 is not sufficient to demonstrate competence for analysis of dioxins, dl-PCBs and ndl-PCBs in food and feed as demanded by EU legislation. A short document was produced by the EURL outlining this requirement.

A clarification of the analytical criteria with regards to the need for duplicate analysis was also made. There is a need for duplicate analysis where the analytical result is more than 50 % of the relevant limit for screening purposes, with a higher limit for confirmatory methods.

5.3. Activities of the EURL-NRL Network

5.3.1. Working Group on Measurement Uncertainty (MU)

This is a very active working group that met several times during the year to discuss not only MU but also definitions for limit of detection (LOD) and limit of quantification (LOQ).

The main objective of the working group on MU is to provide the EURL-NRL network with the tools for a harmonised approach to estimating measurement uncertainty (MU) e.g. a working document with guidelines, including practical examples. The approach to be adopted will work around the principles drafted in the working document on “Evaluation of Measurement Uncertainty for dioxins and PCBs using Isotope Dilution Techniques”. This document contains an agreement on inclusion of trueness (specifically performance testing - PT) data in the MU budget. The proposal of a moving time window of ~3-5 years of PT data including a minimum of six participations will be included. Most members were in favour (Fera have already trialled the inclusion) as it allows a reflection of current laboratory performance, rather than historical data. PT providers will be asked to assess uncertainty on the assigned values as given in the ISO/IEC17043 standards and the IUPAC recommendations for PT. The group still needs to establish the best way of estimating MU on the TEQ, and to provide worked examples of different approaches including principles agreed

The activities on LOD / LOQ arose from a request from the Commission to harmonise the various approaches to determining the limit of quantification (LOQ) among the “contaminant based” EURLs (PAHs, mycotoxins, heavy metals and dioxins/PCBs). As the dioxin/PCB EURL and the MU core working group were not primarily tasked with this requirement, the draft document was prepared by the PAH EURL (JRC) and circulated to the dioxin/PCB network at the last meeting in Freiburg (see Meeting Report Dec 2014). The initial responses from members at this meeting were that the draft did not meet the requirements of the dioxin/PCB network. Consequently it was proposed that the core working group would investigate suitable approaches from within the network and provide a separate amendment to the draft that would be applicable to dioxin/PCB work. The timescale for the amendment currently stands at the end of 2015

5.3.2. Database of Congener Profiles

There are plans to link the EURL-NRL initiative with a German Environment Agency project with similar objectives to establish a database of congener profiles associated with

contamination incidents. Such a database may be useful to identify the source of contamination associated with future contamination incidents. Some progress has been made and there is a potential that the German database may be modified to be suitable to the needs of the NRL network, with minimal additional effort. A working group will be established with a proposed meeting in Berlin, spring 2015, including representation from the UK NRL.

5.4. Interlaboratory Comparisons

There have been several interlaboratory comparisons over the year including the first EURL PT exercise for BFRs. Early in the year final results for a PT exercise on dioxins and PCBs in sepiolite (used in animal feed) were circulated. The second exercise was for PCDD/Fs and PCBs in cod liver and fish liver oil and this was later amended to include BFRs. There is an on-going PT exercise on dioxins and PCBs in animal feed which will be reported in the next financial year. Fera performed well in all exercises.

5.5. Contact with Other NRLs

Fera is regularly contacted by members of the network for advice particularly about MU and methodology, and for issues relating to new and emerging contaminants where we have more experience that most members of the network.

Fera has agreed to host a visit from a member of staff from the Latvian NRL in May 2015. This related to the analysis of brominated and mixed halogenated dioxins and the visit is because of our strength in that area of research.

The EURL is conducting some work comparing chemical analysis results with bioassay results in a project similar to one taking place at the UK NRL (funded by the FSA) and preliminary arrangements have been made for Fera to host a visit from the PhD student conducting this work with other staff from the EURL later this year. A comparison of efforts on these projects may result in useful collaboration and may form some kind of cross validation of approaches.

5.6. Contact with the UK Competent Authority and UK Official Control Laboratories (OCLs)

The UK NRL was represented at an FVO inspection visit concerning eggs that took place at Eurofins in Wolverhampton. This was to support the FSA and Eurofins as an Official Control Laboratory.

5.7. Publication

Fera along with another partner of the NRL network had a paper published about the European strategy and developments following incidents with dioxins in the food and feed chain.

Ron Hoogenboom, Wim Traag, Alwyn Fernandes and Martin Rose (2015). **European developments following incidents with dioxins and PCBs in the food and feed chain.** *Food Control* **50** 670-683.

6. NRL PAHs in Food
Joe Holland

6.1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) constitute a large class of organic compounds containing two or more fused aromatic rings made up of carbon and hydrogen atoms. Hundreds of individual PAHs may be formed and released during incomplete combustion or pyrolysis of organic matter, during industrial processes and other human activities. PAHs are also formed in natural processes, such as carbonisation.

In food, PAHs may be formed during processing and domestic food preparation, such as smoking, drying, roasting, baking, frying or grilling. Vegetables may be contaminated by the deposition of airborne particles or by growth in contaminated soil. Meat, milk, poultry and eggs will normally not contain high levels of PAHs due to rapid metabolism of these compounds in the species of origin. However, some marine organisms, such as mussels and lobsters are known to adsorb and accumulate PAHs from water, which may be contaminated, for example by oil spills. Of the many hundreds of PAHs, the most studied is benzo[a]pyrene and exposure to this was controlled by European Commission Regulation (EC) No. 1881/2006, setting maximum levels for certain contaminants in food stuffs. From September 2012 this was amended by Commission Regulation EU No. 835/2011 stating new maximum levels for the sum of four substances (PAH4) (benzo(a)pyrene, benz(a)anthracene, benzo(b)fluoranthene and chrysene) whilst maintaining a separate maximum level for benzo(a)pyrene.

6.2. Contact with the EURL

The EURL asked NRLs for details of how LOD and LOQ were calculated with a view to harmonising the approach across the network.

6.2.1. EURL Meeting

The annual meeting of the National Reference Laboratories (NRLs) for Polycyclic Aromatic Hydrocarbons (PAHs) was hosted by the Dutch NRL at RIKILT, Wageningen; 13th to 14th October 2014.

At the EURL coordination meeting, it was made clear by DG SANCO that OCLs should be invited to participate in EURL organised PT exercises on a fee basis. Fera will therefore ensure that UK OCLs are invited, and will seek advice from the FSA about arrangements for covering their costs. The EURL has formalised the process for following up underperformance on PT exercises.

Many NRLs are accredited for only few food/feed types and these were encouraged to extend their scope of accreditation.

The EURL presented results of a survey on food supplements and proposed to include these in EU regulations. The French NRL presented results of the French Total Diet Study for PAHs and concluded highest exposure came from molluscs, oils and fats and condiments (Mayonnaise). The lowest margin of exposure was for children where the 95%ile value was around 70,000.

The UK gave a brief summary of recent FSA funded surveys for banana chips and cocoa products, and then a longer presentation on the formation of PAHs as a result of home cooking. This work was done several years ago as part of an FSA funded research project, but has only recently been prepared for publication.

The Greek NRL raised some legal issues about enforcement of regulations, all around the problem of what limit to apply for products that do not consist of a single ingredient, e.g. canned fish in oil.

The Polish NRL presented work done on PAHs in traditionally smoked meat products, and included on overview of different production methods used.

The EURL informed the network that they would be asking NRLs for details of how LOD and LOQ were calculated with a view to harmonising the approach across the network. They also had performed some work on MU in response to concerns that there may be some correlation between PAH4 compounds resulting in an underestimation of uncertainties. The EURL gave a model calculation for NRLs to make their own assessment, but concluded that effects are likely to be very small.

There was discussion about fat extraction from cocoa products, methods for food supplements, and changes in legislation with some concern that the term 'traditionally smoked' is still not defined. Several countries including the UK have a derogation for new limits applied to traditionally smoked products, but only when these are not exported.

The outcome of the PT exercises held in 2013 and 2014 were discussed in some detail. PT exercises planned for 2015 are for cocoa products and smoked fish, and ideas were requested for 2016. There was a proposal for herbs and spices, and the UK suggested barbecued beefburgers as a second material.

6.2.2. Regulations

From 1st September 2014 the maximum limits for cocoa beans and derived products, smoked meat, smoked meat products and muscle meat of smoked fish and smoked fishery products have changed. For cocoa products the PAH4 SUM maximum limit changes from 35 to 30 µg/kg (fat). All other food stuffs mentioned; benzo(a)pyrene maximum limit changes from 5 to 2 µg/kg and PAH4 SUM maximum limit changes from 30 to 12 µg/kg. There are derogations for traditional products of smoked meat and fish for those that asked for it over three years and as long as products were not marketed widely. This included products from eastern European Member States with Protected Geographical Indication (PGI). If OCLs pick up non compliances from small producers the UK has a derogation.

6.2.3. CEN Method

There are 2 CEN methods in development; 4 PAHs by GCMS should be published in 2015; 4 PAHs by HPLC-FD should be published by 2017.

6.3. Interlaboratory Comparisons

6.3.1. Interlaboratory Comparisons on the Determination of Four EU Marker PAHs in Food Supplements and Smoked Meat

The final reports for the interlaboratory comparison studies organised by the EURL PAH on the determination of the 4 marker PAHs in food supplements and smoked meat were received.

Food supplements - there were two supplements analysed, a fish oil and spirulina. Satisfactory z-scores were achieved for the spirulina but incorrect data for the fish oil was reported to EURL due to an administrative error. Actual analytical values produced were in good agreement with the consensus values.

Smoked meat – satisfactory z-scores achieved.

7. NRL Materials and Articles in Contact with Foods Emma Bradley

7.1. Introduction

The term materials and articles in contact with food describes any material that may come into contact with a foodstuff, either directly or indirectly. The most obvious example is food packaging but the term also encompasses materials (and articles) used in food processing, transport, preparation and consumption. Any chemical constituents present have the potential to transfer to the foods with which they come into contact. This transfer is known as chemical migration. The migration of chemicals from food contact materials and articles is controlled by EU legislation which has been implemented in the United Kingdom.

7.2. Activities of the EURL-NRL Network on Food Contact Materials

Regulation (EC) No. 882/2004 on Feed and Food Controls establishes the JRC as the European Union Reference Laboratory for Food Contact Materials (EURL-FCM). The EURL-FCM website can be found at:

https://ec.europa.eu/jrc/en/eurl/food-contact-materials

7.2.1. Contact with the EURL

The excellent working relationship with the EURL was maintained. Fera staff continued to contribute to the drafting of technical guidelines to support Regulation EU (No) 10/2011 and have participated in workshops and work planning on ceramics hosted by the EURL and DG SANCO.

7.2.2. EURL Workshop

The EURL organised two Stakeholder Workshops on Ceramics, one in June 2014 and one in December 2014.

Background. The European Commission has proposed that lower limits for Cd and Pb will be introduced in EU legislation taking into account revised EFSA opinions for these elements. On the basis of the EFSA assessments then the limits should be reduced considerably however the Commission have indicated that more information on methods and the questions “are the existing protocols appropriate to mimic migration into foods?” and “can the lower levels of interest (10 µg/kg for Pb and 5 µg/kg for Cd) be met using the instrumentation available for industry and enforcement laboratories?” need to be answered before they can progress with the legal text. In addition, the inclusion of migration limits for other elements is being considered.

Limits of detection in 4% acetic acid. To assess whether or not the lower levels of interest for Cd and Pb could be met the EURL-NRL network carried out an interlaboratory comparison exercise to determine the concentrations of selected elements in 4% acetic acid. Results were good for the 8 elements tested (Pb, Cd, Co, Ni, As, Ba, Al and Mn). For Pb (spiked at 10 µg/kg) 94% of laboratories that returned results (47 out of 50) obtained satisfactory z-scores. For Cd (spiked at 5 µg/kg) 98% of laboratories that returned results (50 out of 51) obtained satisfactory z-scores. Therefore it could be concluded that the detection limits can be met in 4% acetic acid. Additional detail is provided below.

Test conditions. Current testing for ceramic articles involves the exposure of the article to 4% acetic acid for 24 hours at 22°C. These test conditions are considered to result in the extraction of the elements from the ceramic article and are not related to the migration that occurs into foodstuffs. If the limits for Pb and Cd are reduced as proposed then the test conditions may need to be altered to reflect the migration that occurs into a foodstuff. A programme of experimental work comparing migration into potential food simulants with that into foodstuffs has been carried out by the EURL, NRL-DE, NRL-BE, NRL-UK and industry. The test programme followed involved the determination of the migration of Cd and Pb (and other elements) from a range of ceramic and crystal glass articles that had been provided by industry to represent the range of products available on the market under the following conditions:

- 3 x 24 hours at 22°C into 4% (v/v) aqueous acetic acid (existing extraction test conditions but three successive exposures to represent repeat use)
- 5 hours at 22°C into 10% (v/v) aqueous acetic acid followed by 24 hours at 22°C into 4% (v/v) aqueous acetic acid (5 hours at 22°C into 10% acetic acid was proposed by industry as a means of pre-conditioning articles to reduce the testing time)
- 3 x 2 hours at 70°C into 0.5% (w/v) aqueous citric acid (consistent with test conditions included in the Council of Europe Resolution for metals and alloys)
- 3 x 2 hours at 70°C into tomato sauce at pH 3.2 for ceramics (repeat use hot fill conditions for a worst case acidic foodstuff as specified in existing EU legislation for plastic materials and articles)
- 3 x 24 hours at 22°C into wine for crystal glass (repeat use conditions for a worst case acidic beverage)
- Rim tests according to EN1388-2:1995 and ISO 4531-1/2:1998 (standard test methods)

In general migration into 0.5% citric acid > migration into 4% acetic acid > migration into tomato sauce on the third exposure. Some exceptions did exist which may be explained by the article type and whether or not the pigment is applied under, in or on the glaze.
Problems were encountered by all laboratories using 0.5% citric acid at 70°C due to controlling the temperature of the simulant and the exposure phase. Even covering the flatware articles resulted in some losses of simulant by evaporation which not only reduced the volume of the simulant and made the contact area more difficult to control/determine but also had an effect on the simulant temperature due to evaporative cooling. Data for 0.5% citric acid at other exposure temperatures would be of interest as the issues that arose were due to the exposure temperature rather than the acid itself (which is considered a more relevant food acid).
In general a higher migration of Pb and Cd was observed following pre-conditioning and exposure to 4% acetic acid than for the third successive exposure to 4% acetic acid and so this test was not considered a good estimate of the migration than will occur into food. The aim of the preconditioning was to get the articles in the same state that they would be at the end of the second exposure, i.e. ready to do the third exposure but much more quickly however the conditions used did not achieve this. It was proposed that further work should be carried out to determine a more appropriate means of pre-conditioning the sample to achieve this. It should also be noted that the articles are all different in terms of the glaze and pigments applied and so what is found appropriate for one article may not be appropriate for all and so a lot of testing would be required to demonstrate generic applicability of pre-conditioning for all article types. Data for migration from lead crystal followed the same trends as for the ceramic articles with migration into 0.5% citric acid > migration into 4% acetic acid > migration into wine. The relevance of exposure to wine for

24 hours at 22°C was questioned, however it was used as a comparison at this stage rather than being realistic for the exposure. Despite an extensive testing programme additional work is required before appropriate test conditions that provide a good estimate of the migration that occurs into foods from all ceramic article types can be defined.

7.2.3. EURL Meetings

Two EURL-NRL network plenary meetings were held. Discussions covered the work on ceramics, the inter-laboratory comparison exercises which are both described elsewhere in this report as well as an update on the EURL activities on the databank of substances and methods. Information on the databank of substances can be found at:

https://ec.europa.eu/jrc/en/eurl/food-contact-materials/substance-database

Information on the test methods can be found at:

https://ec.europa.eu/jrc/en/eurl/food-contact-materials/test-methods

In addition to the plenary meetings the EURL organised two workshops on "Safety of food contact materials: migration testing and exposure assessment of chemicals in foods". One of the workshops specifically involved training in the use of the FACET (Flavourings, Additives, food contact materials Exposure Tool) which was created within the FP7 EU funded research project. The second workshop involved the presentation of the technical guidelines that have been prepared in support of Regulation 10/2011.

7.2.4. Regulations

There have been no amendments to the EU Regulations for food contact materials and articles during this reporting period. Several NRLs (including NRL-UK) continued to be involved in the preparation of technical guidelines to support Regulation EU No 10/2011. This document was sent out for consultation in March 2014, the comments received were addressed, the guidelines were presented to the NRLs at the October 2014 plenary meeting and were discussed at the December 2014 Member States meeting. It is expected that they will be finalised in 2015.

7.3. Interlaboratory Comparisons

Four ILCs were carried out in 2014.

- ILC01_2014 Inter-Laboratory Comparison (ILC) exercise on food simulant E
- ILC02_2014 Composition of Multilayer Packaging Films
- ILC03_2014 Elements from food contact materials
- ILC05_2014 Surface area determination

7.3.1. ILC001_2014 Inter-Laboratory Comparison (ILC) Exercise on Food Simulant E

In this exercise the aim was to determine the concentrations of the substances spiked into the Tenax (one blank and one spiked Tenax sample were provided along with one of the standards that was no longer commercially available). A list of ten substances was provided and laboratories were informed that seven of these were spiked into the Tenax. Although the aim of the exercise was a PT and so laboratories could use any method, the EURL provided a method description in which the Tenax was extracted with acetone. Fera followed the methodology provided and correctly identified dibutyl sebacate, benzophenone, caprolactam, di-(2-ethylhexyl) phthalate, di-(2-ethylhexyl) adipate, 2,6-diisopropylnaphthalene and 2-ethyl-1-hexanol in the spiked Tenax.

- 73% of NRLs participated in the exercise
- 82% of those that returned results correctly identified the substances present

- 55% of those that returned results quantified all substances within tolerance limits

All Fera results were satisfactory.

7.3.2. ILC02_2014 Composition of Multilayer Packaging Films

This ILC was carried out as a follow-up to a 2013 ILC. The 2013 ILC was considered to be a success for the mono-layer materials (in which NRLs correctly identified the polymer present) but not for the multi-layer materials as laboratories could not separate and identify the individual layers present in the materials tested. In this exercise a protocol for separation and identification of the layers was provided by the EURL and was followed by Fera. Samples were sent to 38 laboratories and 31 submitted results (23 NRLs and 8 guest laboratories). Results showed a satisfactory laboratory performance of the participants and an improvement compared to the exercise in 2013. Although only few laboratories were able to identify the composition of the samples completely correctly, the majority were able to locate and identify most of the plastic layers. To assess performance the EURL awarded points for different layer assignments. If a laboratory achieved 50% of the points available for a given material then performance was considered to be satisfactory for that film. The overall performance of the laboratories was considered to be satisfactory if results for two or more of the three films were satisfactory. Applying the above criteria Fera results were satisfactory for each film and overall. Participants who managed to separate the different plastic layers of a sample were also, for the most part, able to characterise the polymer type correctly. For laminated layers, the separation did not seem to be problematic whereas the separation of coextruded layers was of much greater difficulty. Another issue seemed to be the recognition of adhesive layers.

7.3.3. ILC03_2014 Elements from Food Contact Materials

This ILC involved the determination of metals in simulants from ceramics (4% acetic acid) and plastics (3% acetic acid). Spiking levels were chosen to be in the range of those included in the Council of Europe metals and alloys Resolution and Commission discussion starting values (DSV's) being discussed in the context of proposed changes to the ceramics legislation. The spiked 3% acetic acid contained Ba, Co, Cu, Fe, Mn, Zn, Li and Sb (not declared – laboratories were requested to identify an undisclosed element). The spiked 4% acetic acid contained Ba, Co, Mn, Pb, Cd, Ni, As, Al. 55 laboratories participated in this exercise and 54 returned results. Fera performance was satisfactory.

7.3.4. ILC05_2014 Critical Aspects in the Determination of the Surface in Contact with Foods for Migration Testing of Kitchen Utensils

A follow up exercise on ILC003 2013 Determination of the surface area of kitchen utensils was carried out. Many of the participants in the 2013 ILC returned unsatisfactory or questionable results and in this exercise were asked to provide additional information on the measurements made when determining surface area and the calculation used. The main problem was identified to be the determination of the height of the sample which will foreseeably be in contact with the food. Consideration of the questionnaire responses identified mistakes in calculation of this parameter itself or in the calculation of the default value for the handle height. It was agreed that a more straightforward approach should be used to define the length of the handle as a convention to overcome these errors. Other errors in the determination of the surface area were linked to individual laboratory calculation errors and for the envelope volume determination were due to differences in the depth of the article and the point at which this was determined. Again this was due to an individual's judgement. Based on the results obtained and the determination methods that returned the best results, the EURL will prepare a guidance document for the determination of surface area.

7.4. Contact with Other NRLs

7.4.1 Advice

The following advice/support was given during the reporting period:

- Provision of methodology on photoinitiators to NRL-SK.
- Provision of advice to NRL-IT on conversion of HMTA to formaldehyde during melamine-ware testing.

7.5. Contact with the Competent Authority

7.5.1. Advice

The following advice/support was given during the reporting period:

- At the request of the FSA provided information to industry on determination of sulphite in food contact materials and articles (tea and coffee filters) and the legislative requirements.
- Discussions with industry on determination of sulfite in food contact materials and articles (tea and coffee filters) and the legislative requirements (following an FSA request).
- Attendance at EFSA FIP network meeting on non-plastics.
- Provision of advice to FSA on migration testing for unpeeled fruit.

7.6. Supporting the UK Official Control Laboratories (OCLs)

7.6.1. Advice

The following advice/support was given during the reporting period:

- Provision of advice to LGC on testing melamine trays for the migration of formaldehyde.
- Provided guidance to OCL on immersion of food contact articles in simulant and exposure area.
- Provided guidance to an OCL on the feasibility of testing a plastic film and monomer (ethylene glycol) to assure compliance EU legislation.
- Provision of advice to OCL on overall migration into olive oil.
- Provision of advice to OCL on testing nylon utensils.
- Provision of advice to OCL on interpretation of formaldehyde migration results.
- Provision of advice to OCL on migration testing for multi-layer plastics.

7.6.2. Training

A presentation on Materials and Articles in Contact with Food was delivered on the MChemA course (April 2014).

Appendix 1: EURL Contact Information

Contaminant	EURL
Mycotoxins	**European Union Reference Laboratory for Mycotoxins** JRC-IRMM Joint Research Centre Institute for Reference Materials and Measurements Retieseweg 111 B-2440 Geel, Belgium Tel.: +32 (0)14 571 229 Fax: +32 (0)14 571 783 E-mail: jrc-irmm-info@ec.europa.eu Operating Manager: Jörg Stroka
Heavy Metals in Feed and Food	**European Union Reference Laboratory for Heavy Metals in Feed and Food** JRC-IRMM Joint Research Centre Institute for Reference Materials and Measurements Retieseweg 111 B-2440 Geel, Belgium Tel.: +32 (0)14 571 252 Fax: +32 (0)14 571 865 Operating Manager: Beatriz De la Calle Piotr Robouch (From 1st January 2013) E-mail: JRC-IRMM-EURL-HEAVY-METALS@ec.europa.eu
Dioxins and PCBs in Feed and Food	**European Union Reference Laboratory for Dioxins and PCBs in Feed and Food** c/o State Institute for Chemical and Veterinary Analysis of Food Bissierstrasse 5 D-79114 Freiburg - Germany Tel.: +49 761 8855 500 Fax: +49 761 8855 100 **Dr. Rainer Malisch** Dr. Johannes Haedrich Dr. Alexander Kotz E-mail: info@crl-freiburg.eu

Contaminant	EURL
Polycyclic Aromatic Hydrocarbons - PAHs	**European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons** European Commission Joint Research Centre Institute for Reference Materials and Measurements Retieseweg 111 B-2440 Geel, Belgium Tel.: +32 (0)14 571 320 Fax: +32 (0)14 571 783 Operating Manager: Thomas Wenzl E-mail: JRC-IRMM-EURL-PAH@ec.europa.eu
Materials and Articles in Contact with Food	**European Union Reference Laboratory for Food Contact Materials** European Commission Joint Research Centre Institute for Health and Consumer Protection Food Contact Materials Group TP 260 Via E. Fermi 2749 I-21027 Ispra (VA) Italy Tel.: +39 0332 785889 Fax: +39 0332 785707 Operating Manager: Dr Catherine Simoneau E-mail: JRC-IHCP-EURL-FCM@jrc.ec.europa.eu

Appendix 2: Fera NRL Contact Information

Area	Name and Contact Details
General enquiries and information	Food and Environment Research Agency Sand Hutton York YO41 1LZ Tel: +44 (0)1904 462000 Fax: +44 (0)1904 462111 Website: http://fera.co.uk/food/nationalReferenceLaboratory/ E-mail: nrl@fera.co.uk
Head NRL Chemical Safety in Food and Feed	Susan MacDonald Tel: +44 (0)1904 462558 E-mail: susan.macdonald@fera.co.uk
Mycotoxins in Food and Feed NRL	Susan MacDonald (as above)
Heavy Metals in Food and Feed NRL	Malcolm Baxter Tel: +44 (0)1904 462667 E-mail: malcolm.baxter@fera.co.uk
Dioxins and PCBs in Feed and Food NRL	Martin Rose Tel: +44 (0)1904 462655 E-mail: martin.rose@fera.co.uk
Polycyclic Aromatic Hydrocarbons NRL	Joe Holland Tel: +44 (0)1904 462230 E-mail: joe.holland@fera.co.uk
Materials and Articles in Contact with Food NRL	Emma Bradley Tel: +44 (0)1904 462604 E-mail: emma.bradley@fera.co.uk

Note: Whilst care has been taken to ensure that the web links contained in this report are correct at the time of issue changes may occur.