

Fera NRL Annual Report
Report to the Food Standards Agency
May 2014



Annual Report

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

April 2013 – March 2014

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1. Introduction

1.1. Scope

Following a tender exercise by the Food Standards Agency (FSA), the Central Science Laboratory (CSL), was appointed as the National Reference Laboratory (NRL) for the UK for chemicals in food. The four year agreement was effective from the 1st April 2008.

During 2008, the FSA carried out a separate tender exercise for the appointment of NRLs for feed. CSL was appointed as the NRL for dioxins and PCBs in feed in March 2009.

On 1st April 2009, CSL became part of The Food and Environment Research Agency (Fera).

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.

Under the most recent tender exercise held early in 2013, Fera was appointed as NRL for the food and feed areas set out below for the next four year period 2013 to 2017. There was an extension to the scope of the areas covered with feed being added to the heavy metals NRL area where prior to the new contract only the food area was covered by Fera. The inclusion of 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 areas for food and feed but these have now been 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

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) No882/2004 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 updated annually and placed on the NRL website.

2.2. 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: four Public Analysts, one each to represent England, Scotland, Wales and Northern Ireland; 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.

2.2.1. Seventh Steering Group Committee Meeting

This meeting was held at the FSA in London on 13th November 2013. The meeting was attended by Chelvi Leonard (FSA), Frank Hollywood (Public Analyst, Staffordshire; England), John Robinson (Public Analyst, Minton Treharne and Davis; Wales), Ron Ennion (Public Analyst, Public Analyst Scientific Services Ltd; Northern Ireland), Paul Hancock (Public Analyst, Worcestershire Scientific Services; APA training committee) Susan MacDonald and Irene Leon (NRL). Members of the FSA were present for relevant sections: Alan Dowding, Gavin Shears, Christina Baskaran and Jonathan Briggs from the Contaminants Division and Ray Smith from the Hygiene and Microbiology Division. Will Munro (FSA in Scotland) participated by teleconference.

An update on each area was given including EURL activities, legislation, methodology, training and the NRL website.

2.3. Training Activities

2.3.1. APA Training Committee

Susan MacDonald attended the APA Training Committee Meeting in London on 11th December 2013.

Throughout the year, Susan MacDonald made regular contact with the APA Training Liaison officer.

2.3.2. Food Contact Materials OCL Training Workshop

A workshop was held at Fera on 20th February 2014 for OCL staff. Further details can be found in 7.5.1.

2.3.3. 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

2.4. 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/food/nationalReferenceLaboratory/>

The content associated with each function is reviewed and updated as necessary in the interest of continual improvement.

2.5. 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.

2.6. Future Activities

The following are planned as general activities for 2014-15:

- Participation in EURL PTs and circulation of information to OCLs when appropriate.
- Visits to OCLs will be planned as required.
- Steering Group Committee Meeting.

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 2010 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.

Directive 2002/32/EC on undesirable substances in animal feed sets limits for aflatoxin B₁ and ergot sclerotia bodies in animal feed. In addition Commission Recommendation of 17th August 2006 on the presence of deoxynivalenol, zearalenone, ochratoxin A, T-2 and HT-2 and fumonisins in products intended for animal feeding sets out a table of Guidance Values for these mycotoxins in products intended for animal feeding. The values vary depending on the intended use, with lower levels for more sensitive species.

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

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. Regulations

Regulation (EC) No 1881/2006 has been amended by Regulation (EC) No 212/2014 to include a maximum level of 2000 µg/kg citrinin in food supplements based on rice fermented with red yeast *Monascus purpureus*. The level shall apply 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.

There is a proposal to amend Regulation (EC) No 1881/2006, setting a limit of 500 mg/kg for Ergot sclerotia for cereal grains intended for human consumption except corn and rice. The maximum level would be reviewed before 31st December 2016. The proposal is due

to be voted on at the Standing Committee on the Food Chain and Animal Health (Toxicological Section) shortly.

Regulation (EC) No 401/2006 establishes the criteria for the sampling for the control of the levels of mycotoxins. The sampling legislation for mycotoxins 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 will be amended by Regulation (EC) No 519/2014 and shall apply from 1st July 2014.

Regulation (EU) No 691/2013 was published in the Official Journal, and came into force on 1st January 2014. It amends Regulation (EC) No 152/2009 on methods for sampling and analysis for animal feed.

Since 25th January 2010 imports of certain feed and food of non-animal origin, from certain non-EU countries, that are considered to be 'high-risk' can only occur through specific ports and airports approved as designated points of entry (DPEs). A 'high-risk' product is feed or food that is either a known, or an emerging risk to public health, e.g. specific products at risk of aflatoxin contamination have been included.

The list of the 'high-risk' products, country of origin and the frequency of checks required can be found at Annex I of Commission Regulation (EC) 669/2009, this is reviewed regularly and was most recently amended by Commission Implementing Regulation (EU) 323/2014 of 28th March 2014.

Regulation (EC) No 1152/2009 consolidated a number of 'safeguard measures' and lays down specific measures for control of aflatoxins in products from certain third countries. This will now be amended to reflect some changes in the control frequencies for the import of certain foodstuffs from certain third countries. The amended version will form a new regulation and Regulation (EC) No1152/2009 will be repealed.

3.2.2. Contact with the EURL

There has been a lot of contact via email, web interfaces and at the EURL workshop. In advance of the EURL Workshop several questionnaires were circulated that Fera completed and returned. These were about sampling and sample preparation uncertainty; LOD and LOQ information for different analytes by LC, GC and LC-MS; and method criteria for LC-MS/MS methods. The information from these was collated and presented at the EURL-NRL Workshop.

At the request of DG SANCO, the EURL was requested to collect information from the NRLs about the way Article 33 (concerning performance of NRLs) of Regulation (EC) No 882/2004 is implemented within Member States. The EURL sent a questionnaire asking which methods the NRL has available and accredited, and asked about organisation of PTs for OCLs. The questionnaire was received in September 2013 and returned in October 2013.

3.2.3. Workshop of EURL-NRL Network

The 8th Mycotoxin EURL-NRL Workshop was held in Rome on 10-11th June 2013. A report of the meeting was prepared and circulated to the FSA.

The key points presented by the EURL at the meeting were:

- the results from the 2012 PT
- the arrangements for the next 2013 PT (maize multitoxin)
- plans for future PTs

There were several discussion topics that resulted in working groups being set up. These were:

- Issues raised at ISO17025 audits
- Sampling & sample preparation uncertainty
- Guidelines for criteria for LC-MS/MS methods
- Guidelines for screening methods

In addition details about the web based communication system, CIRCA-BC were presented.

3.2.4. EURL 2012 Proficiency Test - Deoxynivalenol, Zearalenone, T-2 and HT-2 Toxin in Cereal

The results of this PT were presented at the EURL-NRL workshop. Results for deoxynivalenol and zearalenone were very good, most labs attained satisfactory performance. However the consensus results for T-2 and HT-2 toxin of the participating laboratories did not agree with the assigned values that had been established during sample preparation with Isotope Dilution Mass Spectrometry (IDMS). Therefore z-scores were not given in the report. An investigation at the EURL could find no reason, but there was no resource to retest the material using this method. Calculations, using statistics used by other PT schemes to establish assigned 'consensus' values using a robust mean, showed the NRL laboratories were all in good agreement with each other but they did not agree with the values from IDMS. Had the 'consensus' approach been adopted to calculate z-scores, most laboratories (including Fera) would have been deemed to be satisfactory. However it has been decided to leave the report as it stands, and not issue z-scores for T-2 and HT-2 toxins.

3.2.5. EURL Proficiency Test 2013 - Patulin in Apple Juice

This PT was mandatory for NRLs and open to participation by OCLs. The FSA agreed to fund OCL participation as an ad hoc activity, in total nine UK OCLs registered to participate. Test samples (two apple juice samples) were received in April 2013, and results reported in May 2013. The draft report of the study was received in September 2013 and the final report received January 2014. Fera's performance was satisfactory, as were the majority of the OCLs. However two OCLs had either unsatisfactory or questionable results. Both labs were contacted and asked to investigate the cause and report back to the NRL. Both laboratories replied, one sent a page of calculations to review. No problems were identified. The second laboratory reviewed their procedure and could find no problems. However they prepared a new standard and analysed a FAPAS PT sample from a previous round and this time found satisfactory results. A third laboratory did not submit any results to the PT. To date no response has been received from the laboratory.

3.2.6. EURL Proficiency Test 2013 - Maize for Multitoxins

This PT was open to OCL laboratories. Five UK OCLs registered and their participation was funded by the FSA as an ad hoc activity. Samples (two cereals) were received on 1st August 2013. Laboratories were required to test the samples for aflatoxin B₁,

deoxynivalenol and fumonisin B₁. Laboratories could use any method of their choice, this could be a single method or separate methods for each toxin. Results were reported via an on-line portal. It was stated at the EURL workshop that other toxins were present in the sample and these could be reported in addition to the mandatory analyses. Additional mycotoxins were detected during analysis, e.g. aflatoxins B₂, G₁ and G₂, however the on-line reporting procedure did not have any result fields other than for the mandatory toxins, so the other results were not reported.

The EURL issued the draft report and asked for comments, some minor comments were returned. Fera's results were satisfactory for all analytes except fumonisin B₁ in the feed cereal. In this case our reported result of 8.4 mg/kg was significantly lower than the assigned value of 31.2 mg/kg resulting in an unsatisfactory z-score. The majority of laboratories had a problem with this analysis, with only 12/58 achieving a satisfactory score. In total 79% (46/58) of laboratories obtained a z-score outside ± 2.0 . A few of these were >2.0 (8 labs), however the majority of labs obtained scores <-2.0 , with many of the results reported in the range 1-10 mg/kg (24/58 labs) (similar to the Fera result). Initial evaluation of the data by the EURL suggested there may be a link between the method used and result reported, for example labs that reported using immunoaffinity column clean-up appeared to report lower results than those with another or no clean-up. It was stated a full evaluation of the method questionnaires would be carried out and this will be a topic for discussion at the next EURL-NRL workshop.

Five UK OCLs participated in this PT as well as the NRL. Overall results were very good, for aflatoxin B₁ 8/9 OCLs' reported results were satisfactory. For deoxynivalenol 6/10 OCL results were satisfactory, two were questionable and two were unsatisfactory. Both unsatisfactory results were obtained by the same laboratory. One laboratory did not report any fumonisin results. For the high level fumonisin B₁ sample, two OCLs obtained low unsatisfactory z-scores, one OCL obtained a satisfactory result, and one lab reported a result as a greater than value so could not be given a z-score. For the lower level sample three OCLs were satisfactory, the other laboratory obtained a score of -2.1 so was just questionable.

The EURL sent emails with follow up actions for questionable/unsatisfactory results for the PT. The NRL received an email for the fumonisin analysis, the EURL stated they believe the problem of under reporting of high levels to be due to IAC capacity, and requested information for a Corrective Action Plan (CAP). However as they believe the cause of the problem has been identified there will be no follow up PT. The NRL will complete the CAP and return by the due date in May 2014. The EURL has confirmed that it will not follow up UK OCLs. Therefore the NRL will contact the OCLs to ask for root cause analysis to be performed and to ask if they would like any assistance or advice about methods.

3.2.7. EURL Proficiency Test 2013 - Aflatoxins in Coconut

This was the third PT planned for 2013, however this round was postponed until 2014.

3.2.8. Other PT: Dutch NRL Tropane Alkaloids PT

Fera has been invited to participate in a PT for tropane alkaloids organised by RIKILT.

3.2.9. EURL Method Validation Study (MVS) Ochratoxin A in Paprika and Chilli

The report of this study was made available to OCLs via the Fera website. It was discussed at the EURL workshop and has been put forward to CEN TC275 WG5 to be developed as a standardised method.

3.2.10. Pyrrolizidine Alkaloids in Honey and Plant Material

This method validation study for an LC-MS/MS method for pyrrolizidine alkaloids was run by BfR in Germany. Samples for the pre-trial were received in March 2013. As a result of satisfactory performance in the first phase Fera was invited to take part in Phase 2, the full method validation study. Samples were received in September 2013 and results were submitted in November 2013. This was funded as an ad hoc activity. To date, no feedback or results have been received.

3.2.11. CEN Activity (TC275 WG5): CEN Method

CEN TC275 WG5 Biotoxins received a Mandate (M/520) for the development of eleven new methods of analysis for mycotoxins. The call asked for bids to be submitted to NEN (the Dutch Standardisation Institute). Bids were not received for all the projects, therefore a second call was published in July 2013. No decision on the outcome of these bids has been made yet.

Several CEN/ ISO mycotoxin methods underwent periodic review in this period. The documents were reviewed and voting to confirm their continued suitability and applicability was carried out via the CEN website.

3.3. Contact with Other NRLs

3.3.1. General Contact and Collaboration

Irish NRL – ISO17025 assessment experiences

In advance of the EURL-NRL workshop the Irish NRL requested examples of experiences during ISO17025 assessments. Information from Fera was collected from both analysts and members of the quality team, as former ISO17025 assessors, of issues that had been encountered. The information supplied by Fera and other NRLs was presented at the EURL meeting and used as a discussion topic.

Belgian NRL - Multi-mycotoxin Proficiency Test

Details of a PT organised by the Belgian NRL were circulated to UK OCLs for information.

German NRL – PAs Method Validation

Fera took part in the method validation study run by BfR.

Dutch NRL – Tropane Alkaloids PT

Fera has been invited to participate in a PT for tropane alkaloids organised by RIKILT (see 3.2.8).

EFSA Sterigmatocystin Project

Fera is a partner with the Dutch NRL (RIKILT) in an EFSA project to carry out a survey of sterigmatocystin in cereals across Europe. This project is co-funded by the Contaminants Branch of the FSA.

Swedish NRL – Working Group on Sample Preparation

At the EURL Workshop some Working Groups were established to look at different issues. The Swedish NRL is responsible for the group that will discuss developing common practices and procedures and developing best practice for sample preparation for mycotoxin analysis. Fera will participate in this group. An area has been established on the CIRCA BC website for this activity.

Dutch NRL - Working Group on LC-MS/MS Criteria for Mycotoxin Analysis

The Dutch NRL (RIKILT) agreed at the last EURL-NRL workshop to run the working group to establish method performance criteria for LC-MS/MS analysis specifically for mycotoxins. RIKILT contacted members of the group with a proposed plan of work, suggestions on how to proceed and asking for information about methods currently used. This activity is ongoing.

3.3.2. Advice to/ from Other NRLs

German NRL - Method for Zearalenone in Maize Oil

The German NRL was contacted and asked if they could supply a copy of an analytical method to determine zearalenone in maize oil. The document is in German, so will be translated and made available to the OCLs that participate in the EURL PT.

3.4. Contact with the Competent Authority

3.4.1. Advice and Support

UK Stakeholder Group on Guidance Document for T-2 and HT-2 toxin.

Fera was an active member of a Stakeholder Working Group to draft a UK Guidance document in support of Commission Recommendation 165/2013/EC. Several meetings were attended at the FSA, where the draft document was developed. The document was also reviewed and commented on after changes by other members of the group. The document was submitted to the Commission (DG SANCO) in advance of the EU Mycotoxin Forum. It was presented at the meeting, which was held on 5 - 6th September 2013. The document was well received by the participants of the Forum, and it has now been adopted by DG SANCO as a starting point to be modified into a Europe-wide Guidance document.

Ergot Alkaloid Consultation

Information on methods of analysis, and in particular their performance characteristics, as well as potential analytical capability of UK OCLs for ergot alkaloids was provided to the FSA in response to a European consultation.

Comment on Draft Revision of Regulation (EC) No 401/2006

Fera was asked to provide comments on the proposed amendments to Regulation (EC) No 401/2006.

Advice on Pyrrolizidine Alkaloids (PAs)

Fera was asked to comment on a report produced by BfR on pyrrolizidine alkaloids in herbal teas and infusions in advance of an EU Working Group meeting. Comments were provided.

Comment on Codex Committee on Methods of Analysis and Sampling (CCMAS) Documents

Comments were provided on the CCMAS paper on sampling for deoxynivalenol in response to a request from the FSA.

Comment/ Vote on Retention of ISO Standard for Aflatoxins

As NRL and a member of CEN TC275/WG5, comment and vote on confirming the suitability of the current ISO method for aflatoxins in cereals and peanut products was requested.

3.5. Supporting the UK Official Control Laboratories

3.5.1. Training

Meeting: APA Training Committee

Susan MacDonald attended the APA training Committee (December 2013), where more detailed discussions on NRL-OCL training events were held, including the possibility of preparing another mycotoxin semi-reference material.

Presentation

Susan MacDonald gave a presentation on 'Fusarium mycotoxins, regulations and analysis' at a Workshop held in Droitwich-Spa on 23rd October 2013. There were representatives from several OCLs at the workshop, and practical sessions were held in the Worcestershire Scientific Services Laboratory.

3.5.2. Advice

Advice was given to OCLs on the following topics:

- Advice on sample storage and preparation for patulin in apple juice was provided to an OCL in advance of the EURL PT.
- Query about availability of EURL PT results for Patulin (contacted EURL on their behalf).
- Analysis of nutmeg for aflatoxins.
- Analytical methods for fumonisins.
- Interpretation of results in EURL patulin PT report
- Method information about analysis for ochratoxin A in spices.
- Advice about patulin analysis and standard stability was provided to an OCL after the EURL PT.

3.5.3. Semi Reference Material

Requests were received from two OCLs for additional sachets of NRL_01, Aflatoxins in Chilli Powder semi-reference material. A number of sachets were sent to the laboratories.

3.5.4. RASFF Alerts on NRL Website

Mycotoxin alerts taken from the EU RASFF system were put on the NRL website (see 2.4). These will be updated frequently. An email was sent to all OCLs informing them this new alert system was available.

3.5.5. Meeting: Steering Group Committee

Representatives from four OCLs attended the Steering Group Committee Meeting in November 2013. Useful discussions about the website, and possible training initiatives were held (see also 2.2.1.).

4. Heavy Metals NRL (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.

4.2. Activities of the EURL-NRL Network on Heavy Metals

The EURL is appointed by Commission Regulation (EC) No 776/2006 which amends Regulation (EC) No 882/2004 as regards EURLs. The EURL for heavy metals in feed and food is the Joint Research Centre Institute for Materials and Measurements, (JRC IRMM) in Geel, Belgium. The three types of matrices covered by the EURL are wild caught fish, food of plant origin and animal feed. The EURL website can be found at:

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

Fera is the UK NRL for Heavy Metals in Food and Feed.

4.2.1. Contact with the EURL

A request from the EURL was responded to for confirmation of UK NRL status and NRL activities carried out in the past year. It was confirmed that Fera was the UK NRL for Food and Feed and three documents were forwarded: (1) Fifth Annual Report on Operation of NRL Chemicals in Food and Feed, (2) Heavy Metals in Food NRL Report_Metals in Wheat Flour NRL03, (3) Collated UK OCL PT participation October 2013.

4.2.2. Workshop of the EURL-NRL Network

The 8th EURL Workshop on Heavy Metals in Food and Feed was held on 24th September 2013 in Brussels.

4.2.3. Regulations

Significant changes to maximum levels have been agreed or are proposed and these changes are summarised here for background. The following information was provided in the 8th EURL meeting note:

Review of Regulation (EC) No 1881/2006

Cadmium - Food

Focus mainly on commodities for which no maximum levels exist yet (chocolate and cocoa products*, baby foods**)

- *Proposed maximum levels will depend on the percentage of total dry cocoa solids - milk chocolate (<30%): 0.10 mg/kg; chocolate (<50%): 0.30 mg/kg; chocolate (≥50%): 0.80 mg/kg. Cocoa powder sold to the final consumer: 0.60 mg/kg. Expected to become applicable as from 1st January 2019.
- **Different levels for powdered and liquid infant formulae (from cows' milk: 0.010 and 0.005 mg/kg; from soya (or mixtures): 0.020 and 0.010 mg/kg; processed cereal based foods, baby foods for infants and young children: 0.040 mg/kg. Expected to become applicable as from 1st January 2015.

Some amendments for different fish species

- sardines: 0.10 → 0.25 mg/kg
- bichique: 0.050 → 0.10 mg/kg
- swordfish, anchovy: 0.30 → 0.25 mg/kg
- bullet tuna: 0.10 → 0.15 mg/kg
- bonito, common two-banded seabream, eel, grey mullet, horse mackerel, louvar, sardinops, wedge sole: 0.10 → 0.05 mg/kg

Some amendments for specific vegetables

- parsnips, horseradish, salsify, celery: 0.10 → 0.20 mg/kg

No changes to the Maximum Levels (MLs) of other vegetables and cereals so that:

- Farmers and food business operators have time to put appropriate measures in place to bring cadmium levels down.
- Recommend application of existing mitigation measures and initiate research/ investigations to fill any gaps in knowledge on mitigation measures (for example, cadmium from phosphate fertilisers).

Lead - Food

The CONTAM panel of EFSA adopted an opinion in March 2010

- Identified the need to reduce exposure - concern over possible neuro-developmental effects in young children.
- Population group mainly at risk: unborn child, infants, children.
- Main contributors to exposure broadly similar to cadmium. Cereals, vegetables (especially potato) and to a lesser extent meat/fish.
- Important sources for children, milk, milk products, baby foods (all kinds including formulae), fruit and fruit products, fruit juices, cocoa and chocolate products.

Current discussions in CODEX on maximum levels of lead.

Maximum levels at step 5.

- Fruit juice from berries and other small fruit: 0.05 mg/kg
- Other fruit juices: 0.03 mg/kg

- Canned fruits and vegetables: 0.1 mg/kg as consumed (with the exception of leafy, brassica and legume vegetables, berries and other small fruit).
- Milk and cereals (no change in MLs): 0.02 and 0.2 mg/kg respectively.

Maximum levels under discussion.

- Infant formulae: 0.01 mg/kg
- Fruits, vegetables, milk products for discussion next year onwards.

Once adopted in CODEX, EU Regulations would likely follow.

Arsenic - Food

The CONTAM panel of EFSA adopted an opinion in October 2009

- Focus on inorganic arsenic
- CODEX discussions on MLs in rice, raw and unpolished: 0.2 mg/kg (inorganic), 0.3 mg/kg (total)
- Other food commodities of relevance for discussion at EU level: rice based products (in particular for infants and young children), other cereals, certain algae, food supplements.

Mercury/ Methylmercury - Food

The CONTAM panel adopted a scientific opinion on 22nd November 2012 on the risk for public health related to the presence of mercury and methylmercury in food

- TWI for inorganic mercury of 4 µg/kg bw
- TWI for methylmercury of 1.3 µg/kg bw, expressed as mercury.
- Unborn children constitute the most vulnerable group.

Assessment by EFSA on the beneficial effects of fish consumption ongoing.

Undesirable substances in Feed Regulation (EU) No 744/2012 - Feed

Higher MLs for calcium and magnesium carbonate (alignment with existing levels for calcium carbonate).

- arsenic: 2 → 15 mg/kg
- fluorine: 150 → 350 mg/kg
- lead: 10 → 20 mg/kg
- mercury: 0.1 → 0.3 mg/kg

Higher ML in di copper chloride trihydroxide

- arsenic: 30 → 50 mg/kg

Higher ML in natrolite-phonolite

- lead: 30 → 60 mg/kg

Higher ML in complementary and complete feed for pet animals containing fish, other aquatic animals and products derived thereof and/or seaweed meal and feed materials derived from seaweed.

- arsenic: 4 → 10(*) mg/kg for complementary feed
- arsenic: 2 → 10(*) mg/kg for complete feed.

(*) Upon request of the competent authorities, the responsible operator must perform an analysis to demonstrate that the content of inorganic arsenic is lower than 2 mg/kg.

Undesirable substances in Feed Regulation (EU) */2013 - Feed**

Specific MLs for long term supply formulations of feed for particular nutritional purposes with a concentration of trace elements higher than 100 times the established maximum content in complete feed:

- arsenic: 30 mg/kg (2.5 x the ML for mineral feed)
- cadmium: 15 mg/kg (2 to 3 x the ML for mineral feed)
- lead: 60 mg/kg (4 x the ML for mineral feed)

Higher ML in the feed additive ferrous carbonate

- arsenic: 30 → 50 mg/kg

For the determination of lead in kaolinitic clay and in feed containing kaolinitic clay, the ML refers to a procedure whereby extraction is performed in nitric acid (5% w/w) for 30 minutes at boiling temperature. Equivalent extraction procedures can be applied (e.g. microwave based) if these have been demonstrated to have an equal extraction efficiency. But suppliers can request the use of the original soft extraction procedure in cases where the regulations are exceeded and kaolinitic clay is present.

New CEN Mandates for Animal Nutrition (Topics Related to Heavy Metals) - Feed

- Criteria approach for methods of analysis for heavy metals in feed
- Determinations of trace elements, heavy metals and other inorganic elements in feed by ICP-MS (multi-method)
- Determination of inorganic arsenic in feed materials and compound feed by HPLC-ICP-MS

Regulation (EU) No 691/2013 New Sampling Rules - Feed

- In the case of the analysis of undesirable substances, if the result of the first determination is significantly lower (> 50%) than the specification to be controlled, no additional determinations are necessary. This is on the condition that appropriate quality procedures are applied.
- In other cases a duplicate analysis (second determination) is necessary to exclude the possibility of internal cross-contamination or an accidental mix-up of samples. The mean of the two determinations, taking into account the measurement uncertainty is used for verification of compliance.

4.2.4. SCHEMA 30 04 Lead and Cadmium Migration From Ceramics PT

Participation in this scheme was a joint effort with the Materials and Articles in Contact with Food NRL area conducting the main migration experiments on the ceramic samples (7.2.13). Cadmium and lead were determined in a spiked simulant and the metals migrated from ceramic articles.

4.2.5. EURL IMEP Proficiency Tests

There were two IMEP inter laboratory comparisons held within the year: IMEP-116 Total As, Cd, Hg, Pb and iAs in mushrooms and IMEP-117 Total As, Cd, Hg and Pb in

compound feed. Fera participated successfully in both. In all cases, the Reference Value (X_{ref}) lay within our limits of uncertainty.

Planned tests are IMEP-118: Total As, Cd, Pb, Hg, Sn and iAs in canned food and IMEP-119: Determination of total As, Cd, Pb and Hg in vegetable feed. Both these tests are being offered as open rounds and therefore UK OCLs will be encouraged to take part.

4.2.6. CEN Activity

The 23rd CEN/TC 275/WG10 was attended in Paris on 18th October 2013. Topics covered were:

- Determination of inorganic arsenic in food of marine and plant origin
- Determination of methylmercury in food of marine origin: WI 00275237
- Discussion of comments on CEN Enquiry/ preparation of a final draft of prEN 13805 Foodstuffs - Determination of trace elements - Pressure digestion
- Elaboration of the first working-document of "Determination of hydride-reactive arsenic compounds in rice by atomic absorption spectrometry (Hydride-AAS) following acid extraction"
- Determination of minerals – German proposal for a new work item
- Results of the systematic review of EN 14332 Foodstuffs - Determination of trace elements - Determination of arsenic in seafood by graphite furnace atomic absorption spectrometry (GFAAS), EN 14546 Foodstuffs - Determination of Trace Elements - Determination Of Total Arsenic By Hydride Generation Atomic Absorption Spectrometry (hgaas) and EN 14627 Foodstuffs - Determination of trace elements - Determination of total arsenic and selenium by hydride generation atomic absorption.
- AI-Task-Group CEN/TC 275/WG 10

Two revised method protocols for inorganic arsenic and methyl mercury were circulated for comment from CEN TC275 WG10. These documents were draft SOP's and details of collaborative trials. Collated comments will be discussed at the next CEN meeting to be held at BSI, London, in May 2014 with the intention to create draft Standards for review at CEN/TC 275.

4.3. Supporting the UK Official Control Laboratories

4.3.1. Advice

An OCL was tendering for a new instrument (ICP-MS) and requested advice about technical specifications/ performance tests that would be useful in their selection criteria. No feedback has been received to date.

4.3.2. Participation in Proficiency Tests

As both EURL-HM proficiencies tests for 2014/5 are being offered as open rounds, the UK OCLs will be encouraged to take part in them with their registration costs met by the FSA via the Fera NRL. Fera will monitor OCL performance as their results will be available for Fera to study and if necessary, act upon.

5. Dioxins and PCBs in Food and Animal Feed NRL (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 is:

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

5.2.1. Regulations

The Commission is keen to see a strengthening of enforcement of the provisions for dioxins and PCB regulations.

The EURL-NRL network was involved in discussions resulting in DG SANCO document 11950/2013 Revision 2 and its Annex amending Regulation (EC) No 152/2009 on analytical criteria for determination of dioxins and PCBs in feed. This document was unanimously supported by all Member States at the SCFCAH meeting section "Animal Nutrition" on 16th December 2013.

With this amendment, all proposed amendments on analytical criteria as unanimously derived by the EURL-NRL network were also supported for animal feeds, including the proposed amendments put forward by the EURL-NRL network. The same amendments had already been unanimously accepted for food.

The following maximum levels for liver were adopted:

Food	Maximum level PCDD/Fs	Maximum level PCDD/Fs + dl-PCBs	Maximum level ndl-PCBs
Liver of terrestrial animals referred to in 5.1 with the exception of sheep, and derived products thereof	0.30 pg/g wet weight	0.50 pg/g wet weight	3.00 ng/g wet weight
Liver of sheep referred to in 5.1, and derived products thereof	1.25 pg/g wet weight	2.00 pg/g wet weight	3.00 ng/g wet weight

For sheep this means an increase of four times compared to the previous maximum level, but public health will still be protected. The revisions will establish a higher level that will be enforced rather than keep the current strict level that is not always enforced by Member States.

A review of Recommendation 2011/5016/EU is intended with the goal

- to align for certain foods the provisions to which the action levels refer with what is provided for the maximum levels.
- to recommend particular attention in the monitoring of:
 - a) free range and organic eggs
 - b) liver of sheep and lamb
 - c) Chinese mitten crab
 - muscle meat from appendages (separately)
 - brown meat (separately)
 - Calculation of the level in the complete crab by taking into account the relative proportion of muscle meat and brown meat and the levels found in these parts.
 - d) dried herbs (feed and food)
 - e) clays sold as food supplement
 - f) vitamins/ precursors of vitamins
- to express action levels consistently with two decimal places.
- to express the action level for “fruits, vegetables and cereals” on “wet weight” instead of “product” basis. Also herbs will be included in this food category.
- to add an action level for clays.

Currently maximum and action levels are set for clays and herbs in feed law, but not in food law. As these products frequently are involved in RASFF notifications, it seems to be appropriate to establish action levels in food law.

5.2.2. Contact with the EURL

There were two meetings of the EURL-NRL network during the year. The first was hosted by the Italian NRL and took place from 7th to 8th May, 2013. The second was hosted by the EURL in Freiburg and took place from 26th to 27th November 2013.

5.2.3. Measurement Uncertainty (MU) Working Group (WG)

Alongside both of the EURL-NL network meetings, the working group on measurement uncertainty also held meetings. Fera is an active participant in this working group. The WG has been preparing data to use to explore different approaches to MU.

5.2.4. Data Base for Congener Patterns WG

The intention of this WG is to produce a tool to help with congener pattern recognition in order to help identify sources of incidents. A draft form for collection of data for a congener pattern database has been developed. Fera has volunteered to join this WG but a programme of meetings and distribution of work is yet to be established.

5.2.5. EURL Proficiency Tests (PT)

The first EURL PT on PCDD/Fs and PCBs for the year was in feed fat. The second for the year was for milk. Fera participated in these exercise and results for the milk exercise were discussed at the Freiburg Workshop. The milk fat results were typical of PT exercises but the milk powder exercise gave rise to a much more unusual distribution of results. Reasons for this are still subject to investigation.

The EURL has organised a PT for the mineral feed additive sepiolite to be performed in 2014.

5.2.6. Other PT

Draft results were received from the Dutch NRL for the PT exercise that they coordinated on dioxin and dioxin-like PCBs in fats. This was directed primarily at Benelux commercial laboratories with selected expert laboratories from the NRL network acting as reference laboratories. Fera participates in the 'Dioxins in food' inter-laboratory scheme organised by the Norwegian Public Health Laboratory and results for this were obtained during this period.

Fera had good performance in all of these exercises.

5.3. Contact with Other NRLs

Fera hosted a week long visit from the Belgian NRL to work on GC-MS/MS methods for dioxins and PCBs.

Fera was involved with discussion/ advice to the Portuguese NRL on dealing with samples non-compliant for only some congeners, for example if a sample was found compliant when analysed for PCDD/Fs only but non-compliant when later analysed for PCDD/Fs plus PCBs.

6. Polycyclic Aromatic Hydrocarbons in Food NRL (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. Activities of the EURL-NRL Network on Polycyclic Aromatic Hydrocarbons

The Joint Research Centre Institute for Reference Materials and Measurements (IRMM), Geel, Belgium, is the European Union Reference Laboratory (EURL) for Polycyclic Aromatic Hydrocarbons. The EURL website can be found at:

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

6.2.1. Revision of Regulation (EC) No 882/2004

The European Commission has published proposals to change Regulation (EC) No 882/2004, which lays down provision on the official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules.

Regarding the execution of official controls, it is emphasised that the requirement for accreditation of official laboratories according to ISO 17025 will remain. However, the scope of accreditation will have to include in future all analysis methods that are applied in the course of official controls.

Another change concerns the harmonisation of methods used for sampling and analyses by defining a new hierarchy. In the absence of Union rules, preference shall be given (in hierarchical order) to internationally recognised rules and protocols (such as from CEN), in their absence to methods developed or recommended by the European Union Reference

Laboratories, and validated according to internationally accepted scientific protocols, and only then to methods which comply with relevant rules established on national level. The hierarchy contains two more levels which concern validated methods proposed by the National Reference Laboratory, and finally methods which were properly validated.

The complete proposals can be downloaded from:

http://ec.europa.eu/dgs/health_consumer/pressroom/animal-plant-health_en.htm

6.2.2. Expression of Results for Cocoa and Cocoa Products - Outcome of Discussions with the Experts from the EU Member States

The results of the 2012 proficiency test on the determination of four EU marker PAHs in cocoa butter and chocolate revealed that the variability of results expressed for the chocolate test material on fat basis was higher than the variability of results expressed on product basis.

Consequently the EURL-PAH was requested to investigate whether it would be possible to express results gained for food covered by class 6.1.2 on product basis, as it is the case for PAHs for all other regulated food products.

The EURL evaluated among the NRLs the level of support of such a request. In fact, all NRLs expressed their support for this initiative. Hence, a letter describing the issues related to the expression of results was prepared by the EURL and sent to DG SANCO. It was discussed on 24th June 2013 in the meeting of the Experts Committee on Industrial and Environmental Contaminants. The Experts did not find agreement on a potential change of legislation, which caused postponing of any decision to the meeting on 4th October 2013. In that meeting, DG SANCO presented different risk management options, e.g. setting one maximum level (ML) for all products covered by class 6.1.2, setting fat content dependent levels for the different products covered by class 6.1.2, or keeping the actual legislation without introducing any modification. The first option was rejected as this might decrease the level of protection of the consumer, compared to the current legislation. The second option was rejected as well because it would cause the complication of the existing legislation by introducing additional subcategories. Additionally, food products would have to be classified for the identification of the applicable ML, which might again require fat determination.

The point of expression of results was taken up again in the meeting of the Experts on 6th December 2013, in which DG SANCO reported on a letter received from the European Cocoa Association (ECA) and from CAOISCO (Chocolate, Biscuits & Confectionery of Europe). In essence, industry does not support changes in the approach for expression of results. Industry recommends harmonisation of fat determination methods, which was also expressed by the Experts in the meeting in October 2013. However the final decision on the way of expressing analysis results for food falling under category 6.1.2 will be taken in the next meeting of the Expert Committee.

6.2.3. LOD/LOQ Guide

The guidance document for the determination of the limit of detection (LOD) and limit of quantification (LOQ), which was requested by DG SANCO from the EURLs in the area of food contaminants, is in preparation. The EURL-PAH became responsible for drafting the text. Guidance which is partially available in a different field was taken into consideration. The guidance documents contains both information on the experimental prerequisites and mathematical operations for deriving realistic LOD/LOQ estimates. Additionally

development of templates is planned, which can be directly applied for the calculation of the estimates based on a defined experimental setup.

The draft document was sent by the EURL for internal revision. It will be submitted to the other contaminant EURLs. The guidance document will be discussed and upon agreement by the NRLs will be endorsed at the 2014 workshop. The draft document will be distributed to the NRLs in the first quarter of 2014.

6.2.4. Status of Standardisation of Methods for the Determination of PAHs in Food at CEN

The analysis method for the determination of the four EU marker PAHs in food by GC-MS was submitted to CEN at the beginning of the year. The national standardisation bodies had to comment on the draft standard by the beginning of November 2013. The EURL received and compiled comments. They will be implemented as far as possible. The revised draft standard will be presented to the CEN working group at the beginning of February 2014.

6.2.5. Workshop of the EURL-NRL Network

This year's workshop was hosted by the Czech Republic's NRL, and took place in Prague on 14th & 15th May 2013. A brief workshop report was prepared and circulated.

6.2.6. PAHs in Olive Oil 2012 PT

Fera received the final report for the inter-laboratory comparison study organised by the EURL-PAH on the determination of the 4 EU priority PAHs in olive oil. The target analytes were the 4 EU marker PAHs benz[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, and chrysene. Results were reported corrected for recovery and accompanied by the respective measurement uncertainty for the individual PAHs as well as for the sum of the four PAHs.

Fera's performance was satisfactory for this PT.

6.2.7. PAHs in Bivalve Molluscs (ILC-1060) & PAHs in Olive Oil (ILC-1061) 2013

This year both inter-laboratory comparisons were run at the same time. The target analytes were the 4 EU marker PAHs benz[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, and chrysene. Results were reported corrected for recovery and accompanied by the respective measurement uncertainty for the individual PAHs as well as for the sum of the four PAHs. Fera received the final reports for the ILCs, achieving excellent z-scores in both.

6.2.8. Spanish NRL Method Validation Study (MVS)

The draft standard on the determination of the four EU marker PAHs by HPLC-FD will be subjected to method validation by collaborative trial. Pedro Burdaspal from the Spanish NRL for PAHs is in charge of this project.

7. Materials and Articles in Contact with Food NRL (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:

http://ihcp.jrc.ec.europa.eu/our_labs/eurl_food_c_m

7.2.1. Contact with the EURL

The excellent working relationship with the EURL was maintained. Fera staff contributed 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.

The current (2014) work programme of the EURL can be found at:

http://ihcp.jrc.ec.europa.eu/our_labs/eurl_food_c_m/european-network/FCM%20EURL%20Proposal_WP%202014_CS_V3-final.pdf

7.2.2. Regulations

Commission Regulation (EU) No 202/2014 amending Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food was published in March 2014. The revised text includes amendments to Table 1 (the Union list of authorised monomers, other starting substances, macromolecules obtained from microbial fermentation, additives and polymer production aids) and Table 2 (Group restriction of substances).

Technical guidelines have been prepared in support of Regulation (EU) No 10/2011 and sent out for comment to the Member States and the National Reference Laboratories.

DG SANCO has proposed amendments to the rules for ceramic materials and articles and work is ongoing to define the new rules.

7.2.3. EURL Plenary Meetings

Two plenary EURL-NRL network meetings were held in June and November 2013 to discuss the 2013 work programme and to plan the 2014 work programme. Topics covered

included: a presentation of and a discussion on the outcomes of the EURL-FCM PTs carried out in 2013, a review of the deliverables from the 2013 work programme, a discussion on the work carried out on ceramics, how the 2014 work programme will be implemented, the exchange of information between the NRLs as well as an update on the other activities of the EURL and other NRLs.

7.2.4. EURL Meeting - Multi-Analyte Method EURL-FCM Task Force Meeting Under Regulation EU No 10/2011

It is recognised that it is not possible to develop, validate, ring trial and standardise methods of analysis for every single substance permitted for use in plastic food contact materials and articles. As a result there is a need to develop strategies for the development and validation of more cost-effective multi-analyte methods. A work item was agreed in the 2013 work programme of the EURL to examine strategies to develop a means of prioritising multi-analyte methods. Fera methodology developed in FSA funded surveillance work for the determination of volatile substances was proposed as one possible multi-analyte method. This methodology was written in CEN standard format within CEN/TC194/SC1 in 2007. Additional editorial work is required to finalise the document.

7.2.5. EURL Meeting - Ceramics

Following on from a workshop hosted by the EURL and European Commission in Brussels in February 2013 at which proposed changes to legislation on ceramics was discussed with industry, NRLs, stakeholders and Member States, several of the NRLs attended a follow up meeting to discuss the technical issues associated with the proposed changes. Discussions concentrated on assessing methods and on devising an experimental project plan to compare migration into the existing simulant (4% aqueous acetic acid) with a potential new simulant (0.5% aqueous citric acid) and into foods, taking into account the repeat use of ceramic articles.

7.2.6. EURL Workshop - Declaration of Compliance (DoC) and Supporting Documentation

A training workshop on Declaration of Compliance (DoC) and supporting documentation was organised by the EURL. The workshop comprised of a series of presentations in which NRLs and other laboratories presented ongoing work in this area.

7.2.7. EURL Activities - Maintenance of the Databank of Substances and Analytical Methods

The objectives laid out in the EURL work programme include expanding the databank of substances and methods for the analysis of plastic monomers and additives to include substances and methods for potential migrants from other (i.e. non-plastic) food contact materials. The importance of this databank was assessed by questionnaire and it was agreed that this was a necessary resource required to support official controls. A list of the substances included in the databank and methods for their analysis are provided on the EURL-FCM website at:

http://ihcp.jrc.ec.europa.eu/our_databases/eurl-fcm-ref-coll/reference-substances

7.2.8. EURL Comparative Trials

The EURL organised two comparative trials between April 2013 and March 2014. These exercises were carried out to demonstrate that the skills of the NRLs are appropriate for the enforcement of the EU laws on materials and articles in contact with foods.

Copies of the final reports can be found at:

http://ihcp.jrc.ec.europa.eu/our_labs/eurl_food_cm/publications/publications#inter-laboratory-comparison-exercise

The EURL organised four interlaboratory comparison exercises that were completed in 2013-2014. These were:

- Interlaboratory comparison exercise 2013_001: comparative testing (PT) on the dry simulant Tenax containing a mystery cocktail of 6 substances
- Interlaboratory comparison exercise 2013_002: identification of polymeric materials of unknown nature
- Interlaboratory comparison exercise 2013_003: determination of surface contact area
- Interlaboratory comparison exercise 2013_004: follow-up of the 2012 exercise on the determination of melamine and formaldehyde in 3% acetic acid migration solutions

7.2.9. Interlaboratory Comparison Exercise 2013_001: Comparative Testing (PT) on the Dry Simulant Tenax Containing a Mystery Cocktail of 6 Substances

The work carried out in the 2012 ILC on extraction from and migration into Tenax was considered successful but only looked at a limited number of analytes and at one non-polar extraction solvent. The aim of the 2013 exercise was to further expand the scope of the methodology to more polar substances and more polar extraction solvent(s). The spiked Tenax also contained two unknown substances for the NRLs to identify using analytical screening methodology. This exercise was carried out to provide more data to support the validation of the methodology as well as assessing a laboratory's ability to quantify migrants in Tenax and identify unknowns in migration extracts. Fera did not return results for this exercise. The EURL carried out a study to compare the concentrations measured in different solvent extracts – acetone, ethanol, hexane and methanol were used. Hexane provided satisfactory extraction capacity for all substances except the most polar substance (caprolactam). Acetone was reported to provide satisfactory extraction capacity for all substances except the least polar substance (1-octene). Therefore to ensure complete coverage it was proposed that more than one extraction solvent is necessary. It is foreseen that there will be a follow-up exercise as the results of the quantification were not considered satisfactory (57%). This will be included in the 2014 work programme. The final report can be found at:

http://publications.jrc.ec.europa.eu/repository/bitstream/111111111/30400/1/jrc87459%20report%20ilc001%202013%20tenax_131213-final-pubsy.pdf

7.2.10. Interlaboratory Comparison Exercise 2013_002: Identification of Polymeric Materials of Unknown Nature

To demonstrate compliance with Regulation EU No 321/2011 restricting bisphenol A use in plastic infant feeding bottles requires the ability to determine the polymer type of the baby bottle. This exercise was planned to test the NRLs abilities to identify the polymer type for a range of plastics that may be used as baby bottles as well as other FCM articles. The analysis and identification could be done by using any analytical technique considered appropriate for the exercise. Fera used FT-IR to determine the polymer type. The final report, which can be found at:

<http://publications.jrc.ec.europa.eu/repository/bitstream/111111111/30401/1/lbna26467enn.pdf>

This describes the determination of the polymer type for 12 mono-layer materials.

Qualitative scores were awarded to allow data interpretation. The scores were:

- Very good = 2 points = exactly correct;
- Acceptable = 1 point = correct but not exact identification (e.g. reported the polymer as PE rather than LDPE or HDPE);
- Poor = 0 points = incorrect identification.

Of the 12 mono-layer samples tested Fera scored two points for 10 of the samples and 1 point for the other 2 samples, i.e. all results were considered satisfactory.

7.2.11. Interlaboratory Comparison Exercise (ILC) 2013_003: Determination of Surface Contact Area

The 2012 EURL work programme included one ILC on the determination of the concentration of melamine and formaldehyde in migration solutions derived from the exposure of melamineware spoons to the simulant 3% acetic acid. Nineteen volunteer laboratories from the NRL-FCM network participated in an exploratory optional task to determine the contact area of the spoon used to generate the test solutions. The results ranged from contact areas of 0.73 to 1.99 dm². When determining compliance of migration from kitchen utensils such as the spoon tested the contact area is considered and therefore determination of the surface area is an integral part of the compliance assessment. ILC 2013_003 was organised as a follow up to this voluntary test. The NRLs were asked to provide the EURLs with the methods used to determine the surface area and these were compiled and to complete this ILC the NRLs were requested to determine the contact area of five utensils using each of the methods provided to them. Rather than being a proficiency test the aim of this exercise was to establish best practice in determination of the surface area to help to achieve comparability of results across the EU. Of the methods tested calculation of the contact area by breaking the utensils down into geometric shapes, measuring the dimensions of each and calculating the area of each part using conventional mathematical formulae gave the best results. Fera's performance was satisfactory in 29 of the 30 results reported for the determination of the surface area of utensil A to E. The two methods used at Fera to determine surface area involve wrapping the utensil in paper or cutting shapes in paper consistent with the contact surface and comparing the weight of the areas cut with the weight of a known area of the paper. All of the z-scores for the two Fera methods were satisfactory. The final report can be found at: <http://publications.jrc.ec.europa.eu/repository/bitstream/111111111/30398/1/jrc87418%20%20fcm%20eurl%20ilc03%202013%20final%20report-rev.pdf>

7.2.12. Interlaboratory Comparison Exercise 2013_004: Follow-up of the 2012 Exercise on the Determination of Melamine and Formaldehyde in 3% Acetic Acid Migration Solutions

Several laboratories obtained unsatisfactory results in the 2012 interlaboratory comparison exercise on the determination of melamine and formaldehyde in 3% acetic acid. In general results were satisfactory at low (< 15 mg/kg) concentrations but not at high concentrations and so a follow-up exercise was carried out to establish the cause. Two errors were identified, one due to dilution prior to analysis rather than at the start of the experiment, i.e. before derivatisation and for others because they were measuring a concentration above 15 mg/kg in which the method was found to underestimate the concentration due to lack of linearity of the standards at these concentrations. Performance was improved in the follow-up exercise and the 88% success rate of the laboratories that took part was considered satisfactory. The final report can be found at: <http://publications.jrc.ec.europa.eu/repository/bitstream/111111111/30254/1/lbna26206enn.pdf>

7.2.13. Other PT - SCHEMA 30 04. Migration of Cd and Pb from Ceramics

Fera participated in a SCHEMA 30 04 proficiency test (PT) to determine the migration of Cd and Pb from ceramics. The laboratories received four ceramic articles and one simulant solution containing cadmium and lead (in acetic acid 4%). Samples were received in June 2013 and reported in July 2013. In total fifty five laboratories returned results. As mentioned in section 4.2.4, the FCM-NRL at Fera conducted the migration exposures of the ceramic articles to the simulant and the heavy metal NRL analysed the simulant samples for Cd and Pb. The Fera z-scores were satisfactory for Cd and Pb in simulant solutions and migrating from ceramic ware. Notably, the distribution of results for the migration of Cd fell into two distinct groups. The submitted results and homogeneity data showed the same feature. This highlights the difficulty in obtaining homogeneous articles for migration test purposes.

7.3. Contact with Other NRLs

7.3.1. Advice

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

- Provision of methodology for the determination of photoinitiators to NRL Sweden.
- Provision of methodology for the determination of antioxidants to NRL Germany.
- Provision of information to NRL France on irradiation Regulations in the UK.

7.4. Contact with the Competent Authority

7.4.1. Advice

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

- Provision of advice on the calculation of the overall migration from caps applied to glass jars.
- Liaison with FSA regarding testing migration from stainless steel and developments within the Council of Europe for dissemination to OCLs.
- A meeting was held between the FSA leads on food contact materials and articles and Fera. Topics discussed included: Mineral Oil Hydrocarbons – German legislation, future work, proposed changes to the ceramics legislation, a discussion on future work activities and priorities, FACET project conclusions and status of the software as well as the final report and FSIS of a survey carried out on selected trace elements in food packaged in aluminium.
- Provision of advice to FSA NI regarding the Council of Europe metals and alloys guidelines.
- Provision of information to FSA on OCL capability for testing nylon kitchenware and formaldehyde from melamineware.
- Provision of information to the FSA to aid the completion of document "Consultation on task to provide analytical methods and calibration standards by the European Reference Laboratory for Food Contact Materials".
- Provision of information to the FSA on ceramics sample distribution for EURL testing.
- Provision of advice to FSA on thermoplastic elastomers.

7.5. Supporting the UK Official Control Laboratories

7.5.1. Training: Food Contact Materials and Articles Workshop

A one day Food Contact Materials and Articles Workshop was held at Fera on 20th February 2014. There were seven delegates from Public Analyst Laboratories. Speakers were present from different organisations including the Food Standards Agency, the Laboratory of the Government Chemist and Fera. The agenda for the day included presentations entitled:

- Overview of FCM activities including current status of non-harmonised areas
- Enforcement of Regulation (EU) No 10/2011
- Government Chemist Programme
- Declaration of Compliance
- EURL update
- Question and answer session

The presentations given at the Training Workshop have been made available to all of the OCLs via the APA website.

7.5.2. Advice

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

- Provision of advice on interpretation of Regulation (EU) No 10/2011 with respect to repeat use testing for determination of compliance for primary aromatic amine (PAA) migration
- Provision of advice on metal migration from stainless steel
- Provision of advice on overall and specific migration testing from laminated films
- Provision of advice on test conditions for gloves
- Provision of advice on test conditions for melamineware
- Provision of advice on PAA methodology
- Provision of advice on testing sets of utensils with respect to the FSA sampling guidance for the nationally coordinated food sampling programme 2013-14
- Provision of advice on monomer migration methodology
- Provision of advice on PAA test conditions
- Provision of advice on migration testing of lava stones used for cooking steak etc.

7.5.3. Information

- Provision of ILC01 2012 report on Formaldehyde and Melamine in 3% acetic acid migration solution, to participating OCLs
- Provision of ILC 2013_003: determination of surface contact area report to participating OCLs
- Provision of Technical Guidelines for migration testing document for comment

7.5.4. Support

- Collection of sample from an OCL as contingency to ensure on time delivery of primary aromatic amine migration results following their instrument breakdown. The issue was resolved in time and so no analysis by Fera was required.

Appendix 1: EURL Contact Information

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

Contaminant	EURL
Polycyclic Aromatic Hydrocarbons - PAHs	<p>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</p> <p>Tel.: +32 (0)14 571 320 Fax: +32 (0)14 571 783</p> <p>Operating Manager: Thomas Wenzl</p> <p>E-mail: JRC-IRMM-EURL-PAH@ec.europa.eu</p>
Materials and Articles in Contact with Food	<p>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</p> <p>Tel.: +39 0332 785889 Fax: +39 0332 785707</p> <p>Operating Manager: Dr Catherine Simoneau</p> <p>E-mail: JRC-IHCP-EURL-FCM@jrc.ec.europa.eu</p>

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.gsi.gov.uk
Head NRL Chemical Safety in Food and Feed	Susan MacDonald Tel: +44 (0)1904 462558 E-mail: susan.macdonald@fera.gsi.gov.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.gsi.gov.uk
Dioxins and PCBs in Feed and Food NRL	Martin Rose Tel: +44 (0)1904 462655 E-mail: martin.rose@fera.gsi.gov.uk
Polycyclic Aromatic Hydrocarbons NRL	Joe Holland Tel: +44 (0)1904 462230 E-mail: joe.holland@fera.gsi.gov.uk
Materials and Articles in Contact with Food NRL	Emma Bradley Tel: +44 (0)1904 462604 E-mail: emma.bradley@fera.gsi.gov.uk

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