



## Fera NRL Annual Report 2016 to 2017

Report to the Food Standards Agency



# Annual Report

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## Annual Report on Operation of National Reference Laboratory (Chemical Safety in Food and Feed) by Fera Science Ltd.

April 2016 – March 2017

Report Number	Fera/NRL/2015/09
Authors	S. MacDonald, M. Walls, M. Rose, J. Holland, E. Bradley and I. Leon
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Sponsor	Food Standards Agency Aviation House 125 Kingsway London WC2B 6NH
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Principal Workers	S. MacDonald, M. Baxter, M. Walls, M. Rose, J. Holland, E. Bradley and I. Leon
Project Manager	S. MacDonald
Distribution:	1. Mrs Chelvi Leonard (FSA) 2. Dr Andrew Damant (FSA) 3. Mr Mark Bond (FSA) 4. Mrs Bhavna Parmar (FSA) 5. Dr Christina Baskaran (FSA) 6. Ms Susan MacDonald (Fera)

# 1. Introduction

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## 1.1. Scope

Fera was appointed as NRL for the food and feed areas set out below for the four year period 2013 to 2017 under a tender exercise. Feed was added to the heavy metals and mycotoxins NRLs where previously only food was covered. Dioxins and PCBs NRL also covers food and feed.

Fera acts as the UK NRL for the following groups of chemicals:

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

Fera was relaunched as Fera Science Ltd. on 1<sup>st</sup> April 2015 as the result of a joint venture between Defra and Capita PLC. The NRL contract transferred to Fera Science Ltd. on this date.

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 29<sup>th</sup> 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 Food Standards Agency (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). The MANCP for the United Kingdom (April 2013 to March 2018) was updated and extended in 2016 and is available from the Food Standards Agency:

[www.food.gov.uk/enforcement/regulation/europeleg/feedandfood/ncpuk](http://www.food.gov.uk/enforcement/regulation/europeleg/feedandfood/ncpuk)

### **1.2.3. Official Controls**

These 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. DG Health and Food Safety Audits and Analysis) 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.

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 through Regulation (EC) No 776/2006 that amends Regulation (EC) No 882/2004.

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 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.

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 relevant individual UK NRLs operated by Fera are given in Appendix 2.

## 2. Fera National Reference Laboratory (NRL)

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### 2.1. General and Contract Activities

There is an open and standing invitation from Fera to OCLs inviting them to participate in Fera NRL visits to them or for them to visit Fera for bespoke individual training. This is communicated to OCLs at the Steering Group Meetings. Fera NRL contact information is available in Appendix 2.

### 2.2. Contact with the FSA

#### 2.2.1. DG Health and Consumers Audit

DG Health and Food Safety carried out an Audit of UK controls for contaminants in October 2016. In preparation for the Audit, Fera provided the FSA with help, advice and information. Questionnaires on NRL performance were also completed and information on support and training for UK OCLs was provided.

Susan MacDonald attended the Opening Meeting, accompanied the audit team on a visit to an OCL and participated in the closing meeting via teleconference.

As a follow-up to the Audit, training for OCLs was planned.

#### 2.2.2. NRL Meeting

Chelvi Leonard (FSA) visited Fera in December 2016 to discuss current and upcoming NRL work.

### 2.3. Contact with Official Control Laboratories (OCLs)

#### 2.3.1. MChemA

In April 2016, Susan MacDonald and Emma Bradley gave presentations to OCL MChemA course delegates at Reading University.

#### 2.3.2. Proficiency Test Information

In preparation for the DG Health and Food Safety Audit (October 2016), OCLs were asked to provide information on their proficiency test participation and performance.

#### 2.3.3. OCL Training Workshop

As a follow-up to the DG Health and Food Safety Audit, a one day training workshop was arranged for OCLs. This was held at Fera on 7<sup>th</sup> March 2017. The Workshop covered sampling, sample preparation, reporting, measurement uncertainty and EURL documentation on LOD and LOQ. Excellent feedback was received from the delegates.

## 2.4. Training Activities

### 2.4.1. APA Training Committee

Fera contacted the Committee with a view to attending an APA training meeting and to discuss future training activities. It was agreed to hold a training event involving reporting for regulations, LOD/LOQ and sampling. A successful event was held in March 2017 (see Section 2.3.3).

## 2.5. Website

The Fera NRL website is seen as a cohesive part of the NRL function and the content associated with each function is reviewed and updated as necessary in the interest of continual improvement.

<http://fera.co.uk/about-us/national-reference-laboratory/index.cfm>

Alterations are planned as the main Fera website is updated: <https://www.fera.co.uk/>

## 2.6. Support/ Contact with Other NRLs

There has been regular contact with a number of NRLs across the year with Fera providing advice.

### 2.6.1. Workshops and Working Groups

EURL Workshops were held in this period for all NRL food and feed contaminants and materials and articles in contact with food areas. More information is given in each of the relevant sections.

Fera is a member of EURL working groups for Liquid Chromatography-Mass Spectrometry LC-MS methods for mycotoxin analysis, dioxins and PCBs and Food Contact Materials.

Fera is a member of CEN Working Groups TC327 WG5, CEN TC275 WG5, WG10 and WG13.

## 2.7. Future Activities

The following are planned as general activities for 2017-18:

- Participation in EURL PTs
- Visits to OCLs will be planned as requested
- Steering Group Committee Meeting

## 3. Mycotoxins NRL (Susan MacDonald)

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### 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 amendments Commission Regulation (EU) No 178/2010 and Commission Regulation (EU) No 519/2014.

### 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 remit of the EURL was extended by the European Commission to also cover plant toxins such as pyrrolizidine alkaloids and tropane alkaloids.

The EURL website can be found at:

[http://irmm.jrc.ec.europa.eu/EURLs/eurl\\_mycotoxins/Pages/index.aspx](http://irmm.jrc.ec.europa.eu/EURLs/eurl_mycotoxins/Pages/index.aspx)

#### 3.2.1. Contact with the EURL

There was contact from the EURL about the various PTs that were underway (tropane alkaloids in cereals and herbal teas and infusions, aflatoxins and multimycotoxins). The Guidance Document on LOD/LOQ was distributed by the EURL, it was distributed to the OCLs.

#### 3.2.2. EURL Workshop

The 11th workshop was held at the IRMM JRC in Geel on 5th October 2016. It was held in conjunction with the EURL-NRL workshops for metals and PAHs. In addition, a 10th anniversary symposium was held for all members of the three networks. A note of the meeting was prepared.

A science day for participants at the EURL workshops for Metals, PAHs and Mycotoxins was held from 6th-7th October 2016. All participants from the Workshops attended and the agenda included presentations on alkaloids, inorganic arsenic, dioxins, toxicants formed during food processing, mycotoxins and elemental speciation as well as presentations from representatives from DGSANTE, the European Commission and



Nestle. It gave a good overview of the various issues faced by industry, regulators and laboratories / enforcers.

### **3.2.3. CEN TC275 WG5**

A meeting was held in Brussels on 22nd – 23rd June 2016. The status of all the projects running under Mandate M520 was reviewed. A Fera meeting report was prepared. Several projects have completed full method validation studies, e.g. multitoxin screening method, ochratoxin A in liquorice, spice and cocoa, while the others were at different stages of the process.

Fera hosted CEN TC275 WG5 on 7th to 8th December where all the projects under Mandate M520 were discussed. A meeting report was produced.

### **3.2.4. CEN TC327 WG5**

A telephone meeting was held to discuss the project on theobromine in feed led by LGC. The meeting agreed to proceed with the method validation for both LC-MS/MS and LC-UV methods as little additional work would be required and the availability of both methods would be beneficial. Further meetings were held in October 2016 and March 2017 where method validation studies being run under Mandates M521 and M522 were discussed.

## **3.3. Contact with Other NRLs**

### **3.3.1. Method Validation Studies and Proficiency Tests**

Several of the Method Validation Studies that Fera is collaborating with are being run by other NRLs, so there has been contact about these. Fera is also project leader for two projects under Mandate M520 and we have been in contact with the NRLs about these.

### **3.3.2. Advice**

The LC-MS criteria document produced by the EURL/NRL working group was reviewed in conjunction with the other NRLs that produced it.

## **3.4. Contact with the Competent Authority**

### **3.4.1. EURL Work Programme and CEN activities**

Copies of information from the EURL, including meeting agendas and reports, as well as documents such as the Guidance Document on LOD/LOQ were sent to the FSA. Meeting reports were also produced after each CEN meeting attended.

### **3.4.2. General Advice**

Information was provided about the forthcoming FVO mission to the UK. As NRL for contaminants Fera collected information from the OCLs on their capacity for testing for contaminants listed in Regulation (EC) No 1881/2006 (amended) as well as an updated list of OCL accreditation status.

Fera attended the opening meeting for the DGSANTE Contaminants Audit and gave a presentation on the role of the UK Contaminants NRLs covering the training and events

the NRL has undertaken to support the OCLs. Fera also accompanied the Competent Authority staff on a visit to an OCL with the DGSANTE audit team.

## 3.5. Interlaboratory Comparisons

### 3.5.1. EURL PT - Tropane Alkaloids in Cereals

The EURL ran a PT for tropane alkaloids in cereals in early 2016. The PT was offered to all interested laboratories free of charge as the European Commission wanted to know the capacity and performance of laboratories carrying out this analysis. A presentation about the PT was given by the EURL at the World Plant Toxin conference, satisfactory performance (z-score  $\leq 2$ ) was 95% for total tropane alkaloids and 99% of results were  $\leq 3$ . Therefore, it was concluded there is no issue with analytical performance for these analytes at the low levels in the Regulation.

The final report of this study has not been received yet, however draft results of laboratory performance have been received, Fera obtained satisfactory results for both analytes.

### 3.5.2. EURL PT – Tropane Alkaloids in Herbal Teas and Infusions

Due to the high number of laboratories that wanted to participate in the PT for tropane alkaloids an additional PT for analysis of tropane alkaloids in herbal teas and infusions was also organised. Preliminary results were received, Fera results were all satisfactory, as were most of the results in the study. The final report of the study has not been received yet.

### 3.5.3. EURL PT – Aflatoxins in Defatted Peanut Meal

The EURL announced a PT on aflatoxins in defatted peanut meal, this was also offered to OCLs at a cost. OCLs were informed of the PT and nine UK OCLs registered to participate with their registration costs covered by the FSA. The study had two parts, a calibration solution to be measured on three occasions and peanut test materials. All test materials were analysed and reported in this reporting period.

### 3.5.4. EURL PT – Regulated Mycotoxins and Beauvericin and Enniatins in Maize

The PT was open to OCLs, therefore the OCLs were invited to participate, and three UK OCLs registered. At least one OCL stated they will only analyse some of the mycotoxins in the test sample as they do not have the capacity to analyse them all. OCL participation was funded via Ad Hoc funding by the FSA. The test materials were received and results submitted by the reporting deadline of 28th October 2016.

## 3.6. Method Validation Studies

### 3.6.1. Phomopsins in Lupins

Under CEN Mandate M520, RIKILT is project leader to develop and validate a method for phomopsins in lupin products. The scope of the method was limited to phomopsin A as this was the only commercially available phomopsin analytical standard. Feedback on the pre-trial was given at the CEN TC275 WG5 meeting held on 22nd -23rd June 2016, and on the main trial at the CEN TC275 WG5 meeting held on 7th-8th December 2016. The results were compared to the identification criteria document and found to be compliant for

ion ratio, retention time etc. The reproducibility of the method RSDR was 10-18% for most samples and was 26% for the lowest concentration sample which was near the LOQ for many laboratories. Overall the results were very good considering there was no isotopically labelled standard.

### **3.6.2. Ochratoxin A in Meat Products**

The Italian NRL (ISS) is project leader to develop and validate a method for ochratoxin A in meat products under Mandate M520. Fera participated in this method validation study, samples for both pre-trial and main trial were analysed and the data reported in this period. Results were presented at a CEN TC275WG5 meeting. Overall the data was good with acceptable method performance.

### **3.6.3. Multi-toxin Method for Fusarium Toxins in Food by LC-MS/MS**

Under CEN Mandate M520 ISPA/CNR in Italy is the project leader to develop and validate a method for Fusarium toxins in food by LC-MS/MS. Fera participated, results for the pre-trial for this study were reported. Feedback on the pre-trial was given at the CEN TC275 WG5 meeting held on 22nd - 23rd June 2016. There were issues with the preparation of the test materials for the main trial and this was delayed, however the test materials for the main trial were analysed and reported in this period.

### **3.6.4. LC-MS/MS Method for Tropane Alkaloids and Ergot Alkaloids in Feed**

Under Mandate M521 from CEN TC327 WG5, RIKLT are project leaders to develop and validate a method for ergot and tropane alkaloids in feed. Fera participated in this method validation study. Pre-trial test samples were analysed and reported in this period. The study co-ordinator contacted all laboratories requesting detailed information about the analysis and the instruments used. This was provided.

Samples for the full method validation study were also analysed and reported in this period. No feedback on the method performance had been received at the time of reporting.

### **3.6.5. Theobromine in Animal Feed by LC-MS/MS and LC-UV**

Under Mandate M521/522, LGC are project leaders to validate a method for theobromine in animal feed. The initial method chosen was LC-MS/MS but the remit was expanded to analyse the samples by both LC-MS/MS and LC-UV. Fera participated in the method validation study. Samples for the pre-trial were analysed and reported. Samples for the main trial were sent to laboratories in January 2017, and analysed in this reporting period.

### **3.6.6. Gossypol in cotton seed and animal feed by LC-MS/MS**

Under Mandate M521/522, RIKILT are project leaders to develop and validate a method for gossypol in cotton seed and animal feed. Fera participated in the method validation study. Pre-trial samples were analysed and reported in this reporting period.

## 3.7. Supporting the UK Official Control Laboratories

### 3.7.1. Advice

- Information about the EURL PT for aflatoxins in defatted peanut meal was circulated to the OCLs. Nine OCLs were registered to participate with funding from FSA AdHoc funds.
- Information about the EURL PT for regulated mycotoxins and enniatins and beauvericin in maize was circulated to the OCLs. Three OCLs were registered to participate, with funding from FSA AdHoc funds.
- Advice was given to an OCL about the PT for regulated mycotoxins and enniatins and beauvericin in maize.
- Fera visited an OCL that was being audited as part of the DGSANTE Contaminants controls audit to provide additional support where required.

### 3.7.2. Training

Susan MacDonald gave a presentation on Contaminants at the MChemA course at Reading University in April 2016. This was well attended with approximately 15 delegates from OCLs from across the UK, including several from Scotland. The presentation covered the background of EU and UK regulation on contaminants and touched on risk assessment and controls. Specific information on groups of contaminants, e.g. metals, dioxins, processing contaminants and mycotoxins was also given

## 4. NRL Heavy Metals

### Malcolm Baxter and Mike Walls

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#### 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.

The EURL website can be found at:

<https://ec.europa.eu/jrc/eurl/heavy-metals>

#### 4.2. Activities of the EURL/NRL Network Heavy Metals

##### 4.2.1. Contact with the EURL

There has been contact at the EURL Workshops on Heavy Metals in Geel and EURL inter-laboratory comparisons.

##### 4.2.2. EURL Workshop

Mike Walls attended the EURL Workshop on Heavy Metals in Geel (Belgium) from 5<sup>th</sup> to 7<sup>th</sup> October 2016. This was a 10<sup>th</sup> anniversary event held in conjunction with the EURL's for PAH and mycotoxin groups. As part of the preparation for the Workshop the EURL requested posters from the participants and one was provided by Fera.

##### 4.2.3. CEN Meeting

Malcolm Baxter attended the 26<sup>th</sup> meeting of CEN/TC 275/WG 10 held on 28<sup>th</sup> to 29<sup>th</sup> April 2016 in Utrecht, Netherlands as a technical expert.

#### 4.3. Interlaboratory Comparisons

##### 4.3.1. EURL-HM-22: Determination of Total As, Cd, Pb, Hg, MeHg and iAs in Fish

Results for this PT were reported for all analytes except methyl mercury (which is not fully method validated). Good results were achieved.

#### **4.3.2. EURL-HM-23: Determination of Total As, Cd, Pb, Hg and iAs in Palm Kernel Expeller**

Performance in this PT was good but a small negative bias was traced to an incorrect moisture correction formula (feedstuff regulations assume feed contains 12% moisture); this has now been rectified in procedures.

#### **4.3.3. ILC01 2016 - Determination of Total Al, Mn, Fe, Co, Cu, Zn, Cd, Sb, Ba, and Pb in 4% Acetic Acid Leachates.**

A number of leachates were analysed as part of an EURL FCM ILC; good performance was achieved.

#### **4.3.4. EURL-HM-24 - "Herbal Supplement" Determination of Total Cd, Pb, As, Hg**

Registration to participate in this PT has been completed and analysis is underway.

#### **4.3.5. EURL-HM-25 - "Complete Feed for Fish" Determination of Total Cd, Pb, As, Hg and Inorganic As.**

Fera have registered to participate in this PT.

### **4.4. Method Validation Studies**

#### **4.4.1. Methyl Mercury Method Development**

Fera are currently adapting a method validated for a direct mercury analyser for use with ICP-MS. The method at present gives consistent extraction results from sample matrices of marine origin and thus unknowns can be accurately quantified using the CRM as a recovery estimate. Validation of the method is on-going.

### **4.5. Contact with the Competent Authority**

#### **4.5.1. NRL Meeting**

Malcolm Baxter and Mike Walls attended the meeting with Chelvi Leonard at Fera (07/12/16) to discuss upcoming NRL work.

### **4.6. Supporting the UK Official Control Laboratories (OCLs)**

#### **4.6.1. Advice to OCL: Aluminium Analysis**

Malcolm Baxter responded to a request from an OCL currently using open block digestion. Reservations on this type of method were passed on and the latest methodology discussed at CEN TC275/WG10 - Trace Elements, using high digestion temperatures and wetting the sample prior to digestion was given. Hydrofluoric acid may also be used but has health and safety considerations.

#### **4.6.2. Advice to OCL: Iodine in Seaweed Analysis**

Malcolm Baxter supplied enough detail for an OCL to implement Fera's halide analysis using tetramethylammonium hydroxide (TMAH) digestion.

Acid digestion was discussed and although other workers have reported success, Fera does not use this method.

#### **4.6.3. Advice to OCL: Sample Blanks for Determining LOD/LOQ**

Information was provided on procedures currently used by the NRL and the reference for a document was supplied for suggested reading on the subject: "JRC Guidance Document on the Estimation of LOD and LOQ for Measurements in the Field of Contaminants in Feed and Food".

#### **4.6.4. Request for Advice: New Zealand**

A request was received for information regarding sampling and analysis procedures for the EU as a follow-up for RASFF notifications of Cd in squid. To get a full understanding of the EU procedures for sampling and testing seafood, Malcolm Baxter suggested contacting Piotr Robouch at the EURL for Heavy Metals in Feed and Food; contact details were supplied.

#### **4.6.5. OCL Training Workshop**

The metals team contributed to the training day that was arranged for the OCLs in response to the DG Health and Food Safety audit of UK controls for contaminants.

## 5. NRL Dioxins and PCBs

### Martin Rose

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#### 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.

The EURL website can be found at:

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

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

##### 5.2.1. EURL Workshop

There were two EURL-NRL workshops for Dioxins and PCBs in Feed and Food. The first was hosted by the National Food Agency (as National Reference Laboratory) Uppsala, Sweden on 18<sup>th</sup> to 19<sup>th</sup> May 2016, and the second was held at the EURL in Freiburg, Germany on 29<sup>th</sup> to 30<sup>th</sup> November 2017.

#### 5.3. Interlaboratory Comparisons

##### 5.3.1. EURL PT 2015: Determination of PCDD/Fs and PCBs in Animal Feed

The final proficiency test (PT) organised by the EURL during 2015/16 was the subject of activity during this period and a follow-up report was produced. This covered an investigation of the reason for high deviations of analytical results. All participants of this EURL proficiency test were asked to report which stationary phases were used for GC separation, quantification and, if applicable, confirmation of results; to report any co-elutions of interfering substances with analytes of interest, in particular 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF, that were observed for certain stationary phases; to provide chromatograms of relevant congeners, in particular 2,3,7,8-TCDF and 2,3,4,7,8-PeCDF, and to provide any other relevant information.

##### 5.3.2. EURL PTs: 2016/17

There were two new proficiency tests organised by the EURL during 2016/17. These were obligatory for NRLs and were also available to Official Control Laboratories in order to help demonstrate their competence in analysis. Details are given below.



### **5.3.3. EURL PT 2016: Determination of PCDD/Fs and PCBs in Halibut Filet and Fish Oil**

This exercise showed that some laboratories including NRLs were still clearly not calculating MU correctly. Whilst some bioassay results were far from consensus values, because levels were high, there was good assignment when these were used to assess compliance with legal limits.

### **5.3.4. EURL PT 2016: Feed of Plant Origin**

This exercise took place in the second half of 2016. Fera performed well as UK NRL.

### **5.3.5. EURL PT 2017: Determination of PCDD/Fs, Dioxin-like PCBs and Indicator PCBs in Palm Fatty Acid Distillate**

This exercise took place during early 2017 and results have still not been analysed.

## **5.4. Working Group (WG) Activities**

### **5.4.1. Working Group on LOD/LOQ**

The activities of this WG concluded during 2016, and the final document, agreed between the EURL-NRL networks for dioxins and PCBs, mycotoxins, metals and PAHs was published. It is available on the EURL website:

<http://www.crl-freiburg.eu/dioxin/news.html#2016>

### **5.4.2. Working Group on Measurement Uncertainty**

This working group also concluded its activities towards the end of the 2016/17 period of activity with the final guidance document published in March 2017. The document is specifically focussed on PCDD/F and PCB analysis and will soon be available via the EURL and NRL websites.

### **5.4.3. Working Group on Congener Patterns**

This working group was established to collect together knowledge that can be used to help identify the source of dioxin / PCB contamination during an incident, or when elevated levels are found. Links were made with the German Federal Environmental Agency (UBA) where there is on-going work to relate PCDD/F-PCB congener patterns of sources with environmental samples such as water and soil.

During 2016, the Commission asked the Working Group to use some of the methods proposed to evaluate PCDD/PCDF congener patterns by applying them to dried feed products from Ukraine. This was due to the fact that elevated dioxin levels were found in several dried products from Ukraine during the last few years, possibly related to the drying processes used. The EURL was asked to take a look at the congener patterns in order to check if there is an underlying problem and to assess whether further follow-up is needed. If a potential problem is identified, it could result in an increased level of official controls on imports of dried products from Ukraine.

A paper has been drafted for publication in the scientific literature.

#### **5.4.4. Working Group on Chlorinated Paraffins**

A new WG was established to cover chlorinated paraffins (CP). The first meeting took place and was held in Freiburg in December 2016. The working group was formed at the request of the Commission, due to increased concern about this class of chemicals. The meeting considered the current state of knowledge on CP analysis with respect to the large number of compounds included in the class, extraction and measurement techniques, analytical standards and comparability of processes. It was evident due to the huge complexity of the issue that even on a global basis there is very little progress on any of these parameters. The EURL is planning an inter-laboratory exercise on CPs for the second half of 2017.

### **5.5. Contact with Other NRLs**

#### **5.5.1. Advice: Swedish and Latvian NRLs**

During 2016/17, Fera responded to a request for help and support from the Swedish NRL on the analysis of BFRs in food, and also provided support to the Latvian NRL on the use of MU spreadsheet / calculation.

#### **5.5.2. NRL Activities at Dioxin 2016 Conference**

Fera was represented at Dioxin 2017 and co-authored 7 presentations including 2 that were given relating to EURL-NRL network activities on LOD/LOQ guidance, and on congener patterns. These abstracts are available on-line as part of the 'Organohalogen compounds' database at [www.dioxin20xx.org](http://www.dioxin20xx.org)

### **5.6. Supporting the UK Official Control Laboratories (OCLs)**

#### **5.6.1. OCL Dioxin Analysis**

A consortium of UK OCLs have decided to set up a facility in-house for dioxins analysis and as the UK NRL, Fera have offered support and advice for this in terms of training and advice, in particular relating to sample extraction and clean-up and laboratory management.

#### **5.6.2. OCL Training Workshop**

A presentation was given during the event held on 7<sup>th</sup> March 2017.

### **5.7. Laboratory Safety Requirements for Working with Dioxins as Advised by Leading Competent Bodies**

Following a legal case expressing concerns relating to workers in laboratories handling dioxins, the EURL produced a document: 'Developmental and Immunotoxic Effects from Single Low Doses of TCDD based on U.S. EPA's Reanalysis of Key Issues Related to Dioxin Toxicity (2012); Laboratory Safety Requirements as advised by leading Competent Bodies.' It includes basic advice for safe working and also calculates 'Worst-case accidental theoretical Exposure'.

## 6. NRL PAHs in Food

### Joe Holland

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#### 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.

The EURL website can be found at:

<https://ec.europa.eu/jrc/eurl/pahs>

#### 6.2. Contact with the EURL

##### 6.2.1. EURL Workshop

The 11<sup>th</sup> EURL Workshop was held on 5<sup>th</sup> October 2016 at JRC, Geel and began with an update about the re-organisation of the JRC which will no longer be an institute. Instead there will be 10 directorates which will include European Reference Laboratories (EURLs) for food and feed hosted by the JRC:

- Feed additives
- Food contact materials
- GMOs
- Heavy metals in feed and food
- Mycotoxins
- Polycyclic aromatic hydrocarbons and process contaminants

Thomas Wenzl, the operating manager for the EURL PAH will be moving to food authenticity and will be replaced by Christoph von Holst. 2017 will be the final year the JRC will be hosting the EURLs.

There were then presentations covering PAHs and processing contaminants, updates on EU legislation, the work programme for 2016/17 and the LOD/LOQ guide.

##### 6.2.2. Training: Acrylamide

Fera registered for hands-on training for the determination of acrylamide in food. This training is due to take place in April 2017.

## 6.3. Interlaboratory Comparisons

### 6.3.1. EURL-PAH 2016 PT- PAHs in Smoked Pepper

Fera successfully participated in the interlaboratory comparison study organised by the EURL PAH on the determination of the 4 marker PAHs in smoked black pepper.

One UK OCL participated in this interlaboratory comparison but had questionable results.

### 6.3.2. EURL ILC MCPD Esters and Glycidyl Esters in Food

Samples were received for this new PT under the processing contaminants extended scope of the EURL PAHs. Due to various unforeseen issues Fera did not submit results for this PT.

Two UK OCLs participated in this PT.

### 6.3.3. ILC Four EU Marker PAHs in Coconut Oil

This interlaboratory comparison study was organised by the EURL PAH on the determination of the 4 marker PAHs in coconut oil; Fera participated with successful results.

OCLs did not show any interest to participate.

### 6.3.4. Inter-Laboratory Comparison on the Determination of Acrylamide in Food

Fera has registered for participation in this inter-laboratory comparison study organised by the EURL PAH on the determination of acrylamide in food.

## 6.4. Contact with Other NRLs

### 6.4.1. Advice

PAH NRL Ireland contacted Fera seeking advice for validating a method for erucic acid. Fera replied with answers based on our experience with this subject.

## 6.5. Supporting the UK Official Control Laboratories (OCLs)

### 6.5.1. EURL-PAH 2016 PT- PAHs in smoked pepper

One OCL participated in this interlaboratory comparison but had questionable results. Fera has attempted to contact the OCL several times but have had no response regarding this issue to date.

### 6.5.2. OCL Training Workshop

Information including PAH sampling and preparation, analysis and reporting was presented during the training day on 7<sup>th</sup> March 2017.

## 7. NRL Materials and Articles in Contact with Foods

### Emma Bradley

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#### 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 European Commission Joint Research Centre 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 participated in a workshop on ceramics hosted by the EURL and DG SANTE. The EURL-NRL network is working together to establish a mechanism by which information, methodologies, etc can be shared, i.e. working together to achieve efficiencies in enforcement.

##### 7.2.2. EURL Workshop

The EURL organised one Stakeholder Workshop on Ceramics in October 2016. The work carried out by the EURL over the past few years was considered to be successful; the technical report can be found at:

<https://ec.europa.eu/jrc/en/publication/eur-scientific-and-technical-research-reports/testing-approaches-release-metals-ceramic-articles-support-revision-ceramic-directive>

The studies have confirmed that there are no issues quantifying the trace elements of interest at the low levels proposed. Test conditions of three successive exposures to 4% acetic acid had been found to be suitable to mimic exposure into foods and one exposure to 4% acetic acid had been found to be suitable to mimic migration into acidic foods at elevated temperature (as long as the cooking/heating time was < 2 hours).

##### 7.2.3. EURL Meeting

One EURL-NRL network plenary meeting was held in June 2016. 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>

#### 7.2.4. Regulations

The 6th amendment to Regulation EU No. 10/2011 was published in the Official Journal in August 2016.

[http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L\\_.2016.230.01.0022.01.ENG&toc=OJ:L:2016:230:TOC](http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2016.230.01.0022.01.ENG&toc=OJ:L:2016:230:TOC)

This amendment included the addition of newly approved monomers and additives following EFSA evaluation but also updated the original text.

Commission Recommendation (EU) 2017/84 of 16 January 2017 on the monitoring of mineral oil hydrocarbons in food and in materials and articles intended to come into contact with food was published in January 2017. The EURL has been asked to prepare a technical guidance document to support this Recommendation. A task force was formed but no outputs have been presented to date.

#### 7.2.5. CEN Methods

This year DG-SANTE has proposed that a CEN work programme and mandate be approved for the standardisation of methods. The Commission has indicated that the priority areas are multi-analyte methods and methods for the determination of oligomers.

### 7.3. Interlaboratory Comparisons

The following reports were issued between April 2016 and March 2017:

- ILC01\_2015. Temperature Control (article filling)
- ILC02\_2015. Specific migration in Simulant A

These will be made available at:

<https://ec.europa.eu/jrc/en/eurl/food-contact-materials/interlaboratory-comparisons>

A description of the rationale and the work carried out was given in the 2015/16 annual report.

#### 7.3.1. ILC01 2016 – Migration of Elements from Tableware

Two solutions: 4% acetic acid containing Pb, Cd, Co, Ba, Mn and Al; 3% acetic acid containing Cu, Fe, Zn and Sb were received from the EURL and analysed by ICP-MS to demonstrate the ability to measure these elements in food simulants at or close to the proposed legislative restrictions. Migration of the same elements from glass bowls and ceramic cups was determined to demonstrate the ability to determine migration from food contact articles as well as in migration solutions. Once data evaluation is complete the report will be made available at:

<https://ec.europa.eu/jrc/en/eurl/food-contact-materials/interlaboratory-comparisons>

#### 7.3.2. ILC02 2016 – Inter-Laboratory Comparison Exercise on Temperature Control During Migration Tests and Migration of FCM Substance No 500 by Article Filling

The results obtained in ILC01\_2015 in which the temperature of a food simulant was monitored during an exposure set for 2 hours at 70°C demonstrated that the temperature could not be controlled in accordance with the EU Regulations for the entire 2 hour contact period by a several participants. Therefore, this ILC was set up as a follow-up of ILC01\_2015. In addition the ILC was used to test a laboratories performance in

determining the specific migration of FCM substance No 500 (2,5-bis(5-tert-butyl-2-benzoxazolyl)thiophene). Once data evaluation is complete the report will be made available at:

<https://ec.europa.eu/jrc/en/eurl/food-contact-materials/interlaboratory-comparisons>

## 7.4. Contact with Other NRLs

### 7.4.1. Advice

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

- Provision of advice to NRL Sweden on method validation and accreditation.
- Provision of advice to NRL Ireland regarding simulant D2 choice and specifications.

## 7.5. Contact with the Competent Authority

### 7.5.1. Advice

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

- Provision of advice to industry on mineral hydrocarbons.
- Provision of advice to industry on migration test conditions and the use of substitute simulants in the context of Regulation 10/2011.
- Updating the Competent Authority on the work of the EURL, NRL's and industry on ceramics.

## 7.6. Supporting the UK Official Control Laboratories (OCLs)

### 7.6.1. Advice

- Provision of advice on testing silicones (following clarification with NRL-Germany).
- Provision of advice to OCL on polyaminoamide-epichlorohydrin resin use in food contact applications.
- Provision of advice to OCL on primary aromatic amine migration from repeat use articles.

### 7.6.2. Training: MChemA

A presentation entitled "Materials and articles in contact with food" was given on the MChemA course.

### 7.6.3. Training: OCL BPA Training

A workshop on bisphenol A analysis was held at Fera following a specific request from one of the OCLs received after a legislative update was given on the MChemA training course. An update on the current legal status and proposed future legal status for the use of bisphenol A in food contact materials and articles was provided along with an overview of the published methods and the Fera in-house methodology for the determination of bisphenol A in foods, simulants and food contact materials. The critical points of the analysis were highlighted and demonstrated to the five attendees.

## Appendix 1: EURL Contact Information

Contaminant	EURL
Mycotoxins	<p><b>European Union Reference Laboratory for Mycotoxins</b>            European Commission            Joint Research Centre            Directorate F – Health, Consumers and Reference Materials            Retieseweg 111            B-2440 Geel, Belgium</p> <p>Tel.: +32 (0)14 571 229</p> <p>E-mail: <a href="mailto:JRC-EURL-MYCOTOX@ec.europa.eu">JRC-EURL-MYCOTOX@ec.europa.eu</a></p> <p>Operating Manager: Jörg Stroka</p>
Heavy Metals in Feed and Food	<p><b>European Union Reference Laboratory for Heavy Metals in Feed and Food</b>            European Commission            Joint Research Centre            Directorate F – Health, Consumers and Reference Materials            Retieseweg 111            B-2440 Geel, Belgium</p> <p>Tel.: +32 (0)14 571 980</p> <p>E-mail: <a href="mailto:JRC-EURL-HEAVY-METALS@ec.europa.eu">JRC-EURL-HEAVY-METALS@ec.europa.eu</a></p> <p>Operating Manager: Piotr Robouch</p>
Dioxins and PCBs in Feed and Food	<p><b>European Union Reference Laboratory for Dioxins and PCBs in Feed and Food</b>            c/o State Institute for Chemical and Veterinary Analysis            (CVUA Freiburg)            Bissierstrasse 5            D-79114 Freiburg - Germany</p> <p>Tel.: +49 761 8855 500</p> <p>E-mail: <a href="mailto:info@eurl-freiburg.eu">info@eurl-freiburg.eu</a></p> <p>Dr. Rainer Malisch (Director)</p>



Contaminant	EURL
Polycyclic Aromatic Hydrocarbons - PAHs	<p><b>European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons</b>  European Commission  Joint Research Centre  Directorate – General Joint Research Centre  Directorate F – Health, Consumers &amp; Reference Materials  Retieseweg 111  B-2440 Geel, Belgium</p> <p>Tel.: +32 (0)14 571 221</p> <p>E-mail: <a href="mailto:JRC-EURL-PAH@ec.europa.eu">JRC-EURL-PAH@ec.europa.eu</a></p> <p>Operating Manager: Christoph von Holst</p>
Materials and Articles in Contact with Food	<p><b>European Union Reference Laboratory for Food Contact Materials</b>  European Commission  Directorate General Joint Research Centre  Directorate F – Health, Consumers and Reference Materials  Unit Food and Feed Compliance  Food Contact Materials Group  TP 260  Via E. Fermi 2749  I-21027 Ispra (VA)  Italy</p> <p>Tel.: +39 0332 785319</p> <p>E-mail: <a href="mailto:JRC-FCM@ec.europa.eu">JRC-FCM@ec.europa.eu</a></p> <p>Operating Manager: Eddo Hoekstra</p>

## Appendix 2: Fera NRL Contact Information

Area	Name and Contact Details
General enquiries and information	Fera Science Ltd Sand Hutton York YO41 1LZ  Tel: +44 (0)1904 462000  Website: <a href="https://www.fera.co.uk/national-reference-laboratory">https://www.fera.co.uk/national-reference-laboratory</a> E-mail: <a href="mailto:nrl@fera.co.uk">nrl@fera.co.uk</a>
Head NRL Chemical Safety in Food and Feed	Susan MacDonald  Tel: +44 (0)1904 462558 E-mail: <a href="mailto:susan.macdonald@fera.co.uk">susan.macdonald@fera.co.uk</a>
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: <a href="mailto:malcolm.baxter@fera.co.uk">malcolm.baxter@fera.co.uk</a>  Mike Walls Tel: +44 (0)1904 462150 E-mail: <a href="mailto:michael.walls@fera.co.uk">michael.walls@fera.co.uk</a>
Dioxins and PCBs in Feed and Food NRL	Martin Rose  Tel: +44 (0)1904 462655 E-mail: <a href="mailto:martin.rose@fera.co.uk">martin.rose@fera.co.uk</a>
Polycyclic Aromatic Hydrocarbons NRL	Joe Holland  Tel: +44 (0)1904 462230 E-mail: <a href="mailto:joe.holland@fera.co.uk">joe.holland@fera.co.uk</a>
Materials and Articles in Contact with Food NRL	Emma Bradley  Tel: +44 (0)1904 462604 E-mail: <a href="mailto:emma.bradley@fera.co.uk">emma.bradley@fera.co.uk</a>

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