



Newsletter of CRL for Polycyclic Aromatic Hydrocarbons

Issue 1/2009

Edited by: Donata Lerda

Operating Manager: Thomas Wenzl



The mission of the IRMM is to promote a common and reliable European measurement system in support of EU policies.

European Commission
Joint Research Centre
Institute for Reference Materials and Measurements

Contact information

Address: Retieseweg, 111 - 2440 Geel (Belgium)
E-mail: donata.lerda@ec.europa.eu
Tel.: 0032-014-571826
Fax: 0032-014-571787

<http://irmm.jrc.ec.europa.eu/>
<http://www.jrc.ec.europa.eu/>

Legal Notice

Neither the European Commission nor any person acting on behalf of the Commission is responsible for the use which might be made of this publication, for errors or deficiencies in the information reported and/or in the webpages listed. No preference had been expressed through choice of information delivered: it is intended as a non-exhaustive example of available information.

Operating manager foreword



Welcome to the first issue of the CRL PAHs newsletter!

I am confident it will lead to a better exchange of information and news, and that it will be the product of a combined effort of the CRL and NRLs. You will find some sections that are specifically dedicated to the news from the NRLs, but of course your input is very much welcomed also for other sections.

In addition to the newsletter we are regularly updating the CRL PAHs webpage (http://irmm.jrc.ec.europa.eu/html/CRLs/crl_pah/index.htm). In addition, a Technical Report with general information about PAHs, from chemical structure to analytical methods and many other technical items, is in the making. Your feedback on the website and the Technical Report is encouraged and will be appreciated.

Some key upcoming tasks of interest to the consortium are:

- the test material for the 2009 Proficiency Test (PT) on PAHs will be fish products
- in 2009 there will be a second round for PAHs in oil PT, as a follow-up to the 2007 PT.
- the 2009 workshop for the CRL-NRLs will take place 24-25 March in Geel.
- in 2010 a method validation study by collaborative trial will be conducted. It will concern the determination of EU priority PAHs in edible oil, meat and fish products.
- in 2010 a PT is foreseen, focussing on PAHs in either herbs, baby food or cereal products (final decision will be taken during the March 2009 workshop).
- one or more best practice guidelines concerning various analytical topics specific to PAH determination shall be issued with the collaboration of NRLs.

The work programme will be presented in detail and discussed during the next workshop for NRLs.

The whole CRL PAHs team is eager to contribute to efficient communication within the CRL-NRL network and beyond. To help us make the best possible product I would like to ask you, when reading the newsletter, to take note of any possible improvements and to send your comments and suggestions to the following mail box:

jrc-irmm-crl-pah@ec.europa.eu

Meanwhile, I wish you enjoyable reading.

Thomas Wenzl
CRL PAHs Operating Manager

Table of contents

Operating manager foreword	1
Main feature of the issue	3
The CRL/NRLs PAHs network	5
Important dates	5
News from the NRLs	5
Questions to the CRL-NRL Network	5
Activities at JRC- IRMM	5
National Reference Laboratories (NRLs) contribution "Organisation of food control in different European countries"	6
Germany – food and feed control in a federal state.....	6
How food control is organised in the UK.....	8
Recently published papers	9
Upcoming International Events	15
Past Events	16
Upcoming Training Activities	16
Acknowledgements	16

DISCLAIMER

This newsletter is published for information purposes only. The European Commission or any person acting on their behalf shall not be held responsible for any use third parties make of this information. The links presented should not be seen as any kind of endorsement by the European Commission. Users of the newsletter are free to read and download its contents for information or educational purposes. They shall not, however, be allowed to sell copies of the content without permission from the European Commission.

Main feature of this issue

This first issue of the CRL PAHs Newsletter highlights the EU legislation approach to food safety relevant for contamination from PAHs.

[Regulation \(EC\) No 178/2002 of the European Parliament and of the Council](#) constitutes the foundation of food control legislation, "laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety".

Its main points are consequent to the Commission guiding principles for the European food safety policy set out in the [White paper on Food Safety - COM \(1999\)](#). The application of an integrated approach to guarantee food quality through a controlled chain "from farm to fork" is the important novelty of this document. Its proposals cover all sectors, including feed production, primary production, food processing, storage, transport, and retail sale.

The functions and duties of the CRLs and NRLs, as defined in [Regulation \(EC\) No 882/2004 of the European Parliament and the Council](#), fall within that general frame. The network of CRLs, NRLs and official control laboratories facilitates an area where food safety is optimised, through the common effort of ensuring homogeneous and acceptable level of food and feed controls across all the Member States of the European Union. All necessary controls on commodities from third countries and in third countries are regulated. Regulation (EC) 882/2004 also takes into consideration animal welfare.

For an overview of EU legislation concerning PAHs in various matrices, the following paper could be a valuable guide for its interpretation in relation with the challenges required to analytical methods:

[T. Wenzl et al., Analytical methods for polycyclic aromatic hydrocarbons \(PAHs\) in food and the environment needed for the new food legislation in the European Union. Trends in Analytical Chemistry, 2006. 25: p. 716-724](#)

In particular, the legislation concerning PAHs in food covers both the definition of maximum levels ([Commission Regulation \(EC\) No 1881/2006](#) - Text with European Economic Area - EEA relevance) for various foodstuffs, [Regulation \(EC\) No 2065/2003 of the European Parliament and of the Council](#) specific for primary smoke products) and the requirements for the methods of sampling and analysis to be used for the determination of PAHs levels in foodstuff and primary smoke products ([Commission Regulation \(EC\) No 333/2007](#) and [Commission Regulation \(EC\) No 627/2006](#) respectively).

In the Commission Regulation (EC) No 1881/2006 the maximum levels for PAHs, set for Benzo[a]pyrene (BaP) concentrations only, are listed in the Annex, Section 6. BaP "is used as a marker for the occurrence and effect of carcinogenic polycyclic aromatic hydrocarbons" and the maximum levels range from 1.0 µg/Kg, for food intended for infants, to 10.0 µg/Kg, for bivalve molluscs. All technical references to the choice of the limited PAHs and foodstuffs concerned are reported and discussed in the points (57) to (63) of the introduction part of this Regulation.

The Regulation (EC) No 2035/2003 lays down maximum contents for BaP and benz[a]anthracene (respectively 10 and 20 µg/kg – note the number of significant figures given).

The harmonisation of food controls in the European Union could be achieved through, besides the definition of the maximum levels and of the concerned PAHs, the harmonisation of the quality parameters required for methods of analysis.

We would like to focus here on the provisions laid down for methods of analysis.

No reference to a standard method is included in the mentioned legislation, but specifications for performance parameters which should characterise the method in use are set.

In the case of primary smoke flavourings, the Commission Regulation (EC) No 627/2006 reports in the Annex, Table 2 "Minimum method quality criteria for analysing Polycyclic Aromatic Hydrocarbons (PAHs)". These criteria (see point (8) of the introduction) are derived from the collaborative trials for method validation carried out by the Institute for Reference Materials and Measurements (IRMM) for a method intended the quantification of polycyclic aromatic hydrocarbons in primary smoke condensates. The parameters taken into account are: RSD_i (relative single-laboratory repeatability standard deviation), RSD_r (relative average repeatability standard deviation), RSD_R (relative reproducibility standard deviation), LOD (limit of detection), LOQ (limit of quantification), analytical range, and recovery.

As far as the analytical result itself is concerned, point (12) of the introduction of the same Regulation states: "Following the recommendation given in the ISO, IUPAC, and AOAC International Harmonized Guidelines for the Use of Recovery Information in Analytical Measurement, the analytical results should be corrected for recovery."

As far as the food categories included in the Commission Regulation (EC) No 1881/2006 are concerned, focusing again onto requirements for analytical methods to be used to determine BaP, in the Commission Regulation (EC) No 333/2007 (Text with EEA relevance) - Part C of the Annex general requirements, specific requirements (performance criteria and fitness-for-purpose approach) are laid down.

General requirements are described at point C.3.2.: methods of analysis used for food control purposes shall comply with the provisions of points 1 (list of parameters the analytical methods should be characterised for) and 2 (requirements for precision) of Annex III to Regulation (EC) No 882/2004.

Specific requirements are reported at point C.3.3. The regulated performance criteria are: applicability, LOD, LOQ, precision (HORRAT Values for repeatability and reproducibility), recovery, and specificity. The 'Fitness-for-purpose' approach foresees a maximum standard measurement uncertainty calculated from the LOD of the method and the concentration of benzo[a]pyrene in the sample.

As regards analytical results reporting, the point D.1.1., states that they shall be expressed in the same units and with the same number of significant figures as the maximum levels laid down in Regulation (EC) No 1881/2006. The correction of the result for recovery is compulsory (D.1.2.) whenever the analytical method includes an extraction step and, in this case, the level of recovery shall be reported. In case no extraction step is applied, the result may be reported uncorrected for recovery under specified conditions; in this case in the report shall be mentioned that the result is not correct for recovery. Uncertainty boundaries shall accompany all the reported analytical results; expanded measurement uncertainty, using a coverage factor of 2, which gives a level of confidence of approximately 95 %, is used.

A complete description of the legislative process, of the involved bodies and institutions and of the active food legislation in Europe is reported in:

[European Food Law Handbook, B. v.d. Meulen and M v.d. Velde. 2008: Wageningen Academic Publishers, The Netherlands. Editors](#)

In the Technical Note "PAHs Factsheet" on the CRL webpage, links to the active legislation for PAHs in food are reported.

The CRL/NRLs PAHs network

Important dates

The Proficiency Test (PT) 2008 organised for the NRLs and concerning the determination of 15+1 EU priority PAHs in meat and solvent solution, closed its results reporting stage on the 15 September, and the report will be issued within December 2008.

The 2009 workshop of the CRL PAHs/NRLs network will take place at the JRC-IRMM in Geel (Belgium) from 24 to 25 March.

A follow-up PT will be provided to NRLs in 2008 for the determination of PAHs in olive oil (first PT for that matrix was delivered in 2007).

The 2009 PT for NRLs will concern the determination of PAHs in fish products.

News from the NRLs

From the next issue of the newsletter, this section will be dedicated to briefs from the NRLs concerning general information (change of address or denomination of the NRL) or technical information (new analytical activities, new foodstuffs of interest, analytical techniques tested, etc).

Questions to the CRL-NRL Network

Questions from the NRLs and other control laboratories of general interest will be collected, published as interesting issues and answers provided: **please, participate!**

Activities at JRC- IRMM

The CRL PAHs was established at the JRC Institute for Reference Materials and Measurements (IRMM) in 2006.

IRMM is one of the seven institutes of the Joint Research Centre (JRC), a Directorate-General of the European Commission (EC). The main fields of its activity are: production of reference materials, delivery of expert advice in food safety and quality and bio-analysis as well as providing of reference measurement data. For more detailed information, please visit the IRMM web-page.

<http://irmm.jrc.ec.europa.eu/>

National Reference Laboratories (NRLs) contribution

"Organisation of food control in different European countries"

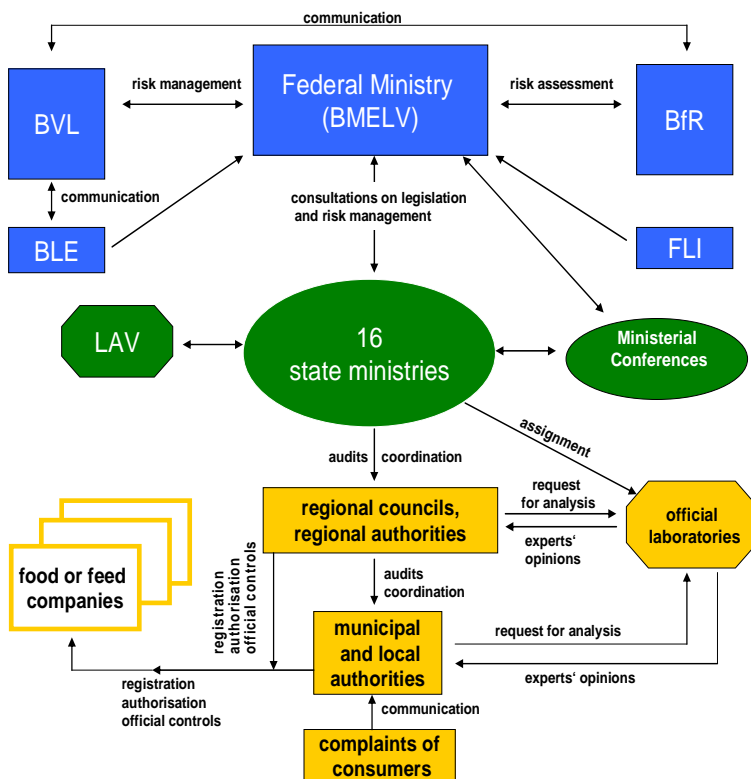
NRLs are requested for a contribution on the above mentioned subject. The first issues of the newsletter will feature the description of the food control organisation in the different Member States of the European Union. In this issue, food and feed control structure is presented for Germany and United Kingdom.

Germany – food and feed control in a federal state

Author: Martina Senger, Bundesamt für Verbraucherschutz und Lebensmittelsicherheit, NRL for Germany

The Federal Republic of Germany consists of 16 federal states (*Bundesländer*) with varying administrative structures. The responsibility for food and feed control as well as for plant health, animal health and animal welfare lies with the states, according to the German Basic Law (Constitution). The federal states assign the duties and coordinate the different tasks of control to districts, municipalities and accredited laboratories. The German Federal Government does not have any directive authority over the states in the above mentioned fields of duty.

The following schema presents an overview on institutions involved on Federal Government (national) level (blue), the authorities and panels on state level (green) and such on district and municipal level (yellow) as well as their respective structures of communication.



The Federal Ministry of Food, Agriculture and Consumer Protection (BMELV) almost entirely assumes the responsibilities in the sectors of food and feed safety, plant health, animal health and

animal welfare on the national level. Subordinate to the Federal Ministry are various superior federal authorities, institutes and research facilities.

Superior federal Authorities subordinate to the Federal Ministry (BMELV)

The Federal Office of Consumer Protection and Food Safety (BVL) has major tasks in assisting the cooperation of ministries and federal states in fields of food safety, coordination of control, surveillance and monitoring programs, authorisation of food additives, pesticides and veterinary medicines, and consumer protection. As the national contact point of the European Rapid Alert System for Food and Feed (RASFF), the BVL supports the Ministry (BMELV) in situations of crisis. The BVL also assesses notifications for the experimental use of genetically modified organisms (GMO) and is involved in the approval of GMOs in connection with food and feed.

The Federal Institute for Risk Assessment (BfR) is a scientific institute working out statements and independent scientific opinions and providing expertise for action to minimise potential risks on the basis of internationally approved scientific criteria. The BfR informs and gives advice to the BMELV, state authorities, institutions of the European Community, industry and public within the wide range of its responsibilities. The BfR cooperates with other scientific institutions on national and international level especially with the European Food Safety Authority (EFSA).

The Federal Institute for Agriculture and Nutrition (BLE) is the coordinating administration for common market organisations existing in the EU in the sectors of grain, rice, sugar, fruit and vegetables, seeds, wine alcohol, beef, pork and meat of lamb, milk and dairy products, fishing products.

The Friedrich Loeffler Institute, Federal Research Institute for Animal Health (FLI) is responsible for research and risk assessment concerning infectious diseases in animals including zoonoses, assistance in inspections of animals and animal by-products for import and export, and epidemiologic investigations in case of animal and epizootic diseases. The FLI takes part in the European project of "TRACES" for the documentation of transports of animals and animal by-products within the European Union as well as from third countries.

Control of food and feed

The Federal Ministry of Food, Agriculture and Consumer Protection is working out legal provisions on the national level. Assisted by the BVL, the ministry prepares administrative regulations. The BVL is the management authority for health-related consumer protection, assisting in and coordinating cooperation on food safety among ministries, the states and the European Community. While the BfR is responsible for the assessment of food-borne risks to public health, risk communication is a task of both the management authorities (BMELV, BVL, states) and the assessment authority BfR.

Control of food and feed is the responsibility of the 16 federal states. Enforcement of controls is incumbent on the municipal and local authorities. The competent authorities of the states appoint laboratories to analyse food and feed samples drawn by food control officers. Furthermore they register and authorise feed establishments and maintain accounts of all authorised and registered operators.

The supreme authorities of the 16 states communicate and cooperate in the States' Working Group on Consumer Protection (LAV), which has eight different sections working on different subjects.

How food control is organised in the UK

Author: Susan MacDonald, Central Science Laboratory, NRL for the United Kingdom

The UK National Control Plan (NCP) principal objectives are in line with those established in EC Regulation 882/2004.

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). This includes (but not only) rules on hygiene, additives, contaminants, labelling and composition, GM feed and food, and food contact materials. It also includes rules on feed and food produced and sold within the EU, and public health requirements for feed and food imported from outside the Community.

Defra (and its agencies) and the Agriculture/Rural Affairs Departments in the Devolved Administrations have responsibility between them for feed and food legislation that falls outside the FSA's remit, including standards for organic products, and residues of pesticides and veterinary drugs. The various central Government and local authorities that are responsible for organising and undertaking official controls constitute the 'competent authorities'.

Responsibility for monitoring and verifying compliance (official controls) and enforcement of feed and food law is divided. For the most part, this function in respect of food (including imported food) is delegated to local and port health authorities in the UK (this involves 469 local authorities). Port health authorities (PAHs) are local authorities that inspect food at points of import.

Areas outside local authority remit include the monitoring, designation and classification of shellfish harvesting areas in the UK (FSA), the national monitoring and surveillance programmes for pesticide residues in food and drink (Pesticide Safety Directorate (PSD)), and the veterinary drug residue surveillance programmes (Veterinary Medicines Directorate (VMD)).

The competent authorities are assisted in carrying out their various functions by National Reference Laboratories (NRLs) and by official laboratories, and also by a number of independent third parties to which specific control tasks have been delegated - these are termed 'control bodies' under Regulation 882/2004.

NRLs are responsible for co-ordinating the activities of official laboratories and they provide scientific and technical assistance to the central competent authorities. Official laboratories are designated by the central competent authorities for the purposes of chemical analysis or microbiological examination of feed or food samples taken by enforcement practitioners. Designation may only be granted if the laboratory meets certain standards (i.e. is accredited to the European Standards specified in Regulation 882/2004). In the UK, accreditation is undertaken by the United Kingdom Accreditation Service (UKAS). Laboratories that undertake work for the central competent authorities and their agencies, such as the PSD and the VMD, are also designated official laboratories.

The competent authorities have Emergency or Incident Response Plans outlining the procedures that should be followed during food and feed incidents and emergencies. Food Alerts are issued by the FSA to let local authorities (and consumers) know about problems associated with feed and food and, where appropriate, provide details of specific action to be taken. These are issued under two categories, 'Food Alerts: for Action' and 'Food Alerts: for Information'.

Arrangements are in place in the UK to ensure mutual assistance and co-operation between the competent authorities of the different Member States and with the Commission. This is separate to the arrangements concerning food hazards and incidents that are communicated via the Commission's RASFF system (Rapid Alert System for Feed and Food). The FSA is a member of the RASFF network and its Incident Branch is the UK contact point for RASFF notifications.

More information on the UK National Control Plan can be found at the link on the FSA website: Single integrated national control plan for the United Kingdom, January 2007 to March 2011 (Revision 2 - issued February 2008)

<http://www.food.gov.uk/multimedia/pdfs/uknationalcontrolplan.pdf>

Recently published papers



In the following pages some of the papers read by the CRL PAHs team members, within the on-going scientific projects, are described. Both "fundamental" papers and new literature are reported.

YEAR OF PUBLICATION	2008
First Author	V. Fernandez-Gonzalez
Other authors	E. Concha-Grana, S. Muniategui-Lorenzo, P. Lopez-Mahia, D. Prada-Rodriguez
Title of the paper	A multivariate study of the programmed temperature vaporization injection-gas chromatographic-mass spectrometric determination of polycyclic aromatic hydrocarbons. Application to marine sediments analysis
Journal, Volume, Pages	Talanta, 74, 1096-1103
Category	Method development
Ref. to Official method	
Official method	
Contaminant(s)	26 alkylated and non-alkylated PAHs (including 9 of the 15 + 1 EU-priority PAHs).
Matrix	Real marine sediments and SRM 1941b.
Extraction method	Microwave assisted extraction (MAE).
Extraction solvent	30 mL of hexane:acetone (1:1, v/v)
Conditions of extraction (T, P, sorbent)	The microwave program begins at room temperature, reaches 130 C in 10 min (held for 20 min), and finishes with a cooldown time of 10 min.
Purification method	SPE
Purification step	30 ml of a mixture of dichloromethane:hexane (20:80; v/v).
Conditions of purification (T, P, stationary phase, etc)	The extracts were cleaned with a 5 g alumina (10% deactivated) column. The eluate was concentrated in a rotary evaporator, dried under a nitrogen stream, and redissolved in 250 µl of perylene-d12 (0.25 10g/mL in hexane) before the GC-MS analysis.
Analytical Technique	PTV-GC-MS
More on analytical technique	PTV-GC-MS: Thermo-Finnigan Trace GC chromatograph equipped with a GC PAL autosampler, PTV and split/splitless injectors and coupled to an ion trap mass spectrometer (Polaris Q). Separation achieved with a DB-XLB column (60m × 0,25*0,25)
Instrumental conditions	50 C 3 min, then 4 C/min to 325 C (hold for 20 min). Mass spectrometer in Full scan and in SIM mode. Ionization voltage:70 eV, transfer line T= 300 C and ion source T= 250 C. Injector initial temperature:55 C Heating rate optimized in this work
Quality parameters (LOD, LOQ, etc)	The instrumental detection and quantification limits were calculated as Xb +3Sb and Xb +10Sb respectively, where Xb is the average value and Sb the standard deviation of the background in a low concentration standard

	(0.005 ug/mL).
Guideline for Calibration	Acenaphthene-d10, chrysene-d12, phenanthrene-d10 and naphthalene-d8 were used as labelled internal standards for quantitation.
Guideline for Validation	Not described. The precision of the PTV method (repeatability and intermediate precision) was calculated with standard solutions. Trueness of the whole method was evaluated through the analysis of the SRM 1941b.
Guideline for Uncertainty	Not described
Interesting points	The paper describes the optimization of large-volume injection (PTV) for the analysis of different PAHs by GC-MS. PTV is optimised in splitless mode. Although applied to marine sediments, the optimised PTV method might be also applied to other matrices.
Additional interesting points	Experimental design strategy is followed to optimise PTV injection. Main advantage is the reduction in the number of experiments to be run and the identification of possible interactions amid the parameters involved in the PTV injection. An improvement of 50- and 100-fold, for the injection of 10 and 25µL, respectively, was achieved for PAHs LOD in comparison with splitless injection.
Comments	None

YEAR OF PUBLICATION	2008
First Author	W. Jira
Other authors	K. Ziegenhals and K. Speer
Title of the paper	Gas chromatography-mass spectrometry (GC-MS) method for the determination of 16 European priority polycyclic aromatic hydrocarbons in smoked meat products and edible oils
Journal, Volume, Pages	Food Additives and Contaminants, 25, 704-713
Category	Method development
Ref. to Official method	
Official method	
Contaminant(s)	15 + 1 EU priority PAHs
Matrix	Smoked meat products and edible oils
Extraction method	Pressurised liquid extraction (PLE)
Extraction solvent	n-hexane
Conditions of extraction (T, P, sorbent)	100 °C and 100 bars at a static time of 10 min. The flush volume was 60% and the purge time 120 s. Two static cycles were accomplished.
Purification method	Gel Permeation + SPE.
Purification step	Cyclohexane/ethylacetate (50:50 v/v) for the GPC and 10 mL of cyclohexane for the PAHs elution from the SPE silica cartridges.
Conditions of purification (T, P, stationary phase, etc)	The GPC column (25mm i.d.) was filled with Bio-Beads S-X3 (weight of filling 60 g). Flow rate of 5 mL/min. For the SPE cartridges the stationary phase was 1 g of silica (dried for 12 h at 550 °C and deactivated with 15% water).
Analytical Technique	GC-HRMS and GC-MS

More on analytical technique	GC-HRMS: HP 5890 II gas chromatograph with a split/splitless injection port. The GC was equipped with a VF-17ms capillary column (60m, 0.25mm i.d., 0.25 mm film thickness). GC/MS: Agilent 7890 GC and Agilent 5975 inert mass.
Instrumental conditions	GC/MS and GC-HRMS: 50 °C (1 min hold), then to 280 °C at 30 C/min, to 340 °C at 1 °C/min and to 350 °C at 1.5 °C/min kept constant for 2 min. Different programmed temperature was applied to separate chrysene and triphenylene on a VF-Xms, 60m, 0.25, 0.25
Quality parameters (LOD, LOQ, etc)	GC/MS: LODs of 0.01-0.02 µg/Kg and LOQs of. 0.03-0.06 µg/kg. GC-HRMS: LODs of 0.003-0.01 µg/Kg and LOQs of. 0.009-0.03 µg/kg.
Guideline for Calibration	Not described
Guideline for Validation	Not described
Guideline for Uncertainty	Not described
Interesting points	GC-MS method is able to analyse the 15+1 EU-PAHs in smoked meat and edible oils. Good resolution is achieved for all PAHs using mid-polarity column (VF-Xms, 60m, 0.25, 0.25).The GC method also offers a good separation of different methylchrysene isomers.
Additional interesting points	Information is also given on the separation of chrysene and triphenylene. This method is based on another method (Jira, 2004, Eur. Food Res Tech. 218:208-212) applied for the analysis of liquid smoke flavourings and smoked meat products. The performance of the method for edible oils was demonstrated in a proficiency test organised by the Institute for Reference Materials and Methods (http://www.irmm.jrc.be/html/interlaboratory_comparisons/index.htm).
Comments	None

YEAR OF PUBLICATION	2007
First Author	Nobuyasu Itoh
Other authors	Masahiko Numata, Yoshie Aoyagui, Takashi Yarita
Title of the paper	Comparison of the behaviour of the 13C- and deuterium-labelled polycyclic aromatic hydrocarbons in analyses by isotope dilution mass spectrometry in combination with pressurized liquid extraction
Journal, Volume, Pages	Journal of Chromatography A, 1138, 26-31
Category	
Ref. to Official method	
Official method	
Contaminant(s)	PAHs
Matrix	Sediments
Extraction method	Pressurized liquid extraction (PLE)
Extraction solvent	n-hexane, n-hexane:acetone (1:1), toluene
Conditions of extraction (T, P, sorbent)	150 C, 15 Mpa, static time 10 min, 2 cycles
Purification method	SPE coupled with a Zymark RapidTrace automation system
Purification step	6 mL of n-hexane

Conditions of purification (T, P, stationary phase, etc)	The extract from ASE was concentrated to 1mL and loaded in the SPE cartridges 500mg /3mL Isolute Silica. PAHs were eluted with 6-mL of n-hexane., which were evaporated and replaced by toluene.
Analytical Technique	GC-MS
More on analytical technique	GC-MS: Agilent 6890/5973N. The column used was a DB-17MS capillary column (30 m x 0.25 mm i.d x 0.25 micrometres film thickness: J&W Scientific, CA, USA) attached via a 1-m deactivated retention gap. MS operated in EI ionization mode.
Instrumental conditions	Splitless mode of injection. Injector T = 300 C; transfer line T= 300 C. Carrier gas: Helium; flow constant at 1.0 mL/min. The oven T programme was 50 C (2 min hold), 10 C/min up to 240 C and 1.25 C/min up to 300 C (10 min hold). MS
Quality parameters (LOD, LOQ, etc)	Not described
Guideline for Calibration	Not described
Guideline for Validation	Not described
Guideline for Uncertainty	Not described
Interesting points	Special care should be taken when using PAHs-d as internal standards. This study demonstrated that the measured concentrations of PAHs were significantly lower when using PAHs-d instead of 13C-PAHs, due to their higher recoveries.
Comments	13C-PAHs might also introduce an underestimation of PAHs concentration when PLE is performed at low temperatures.

YEAR OF PUBLICATION	2005
First Author	S. Martinez-Lopez
Other authors	A. Morales-Noe, A. Pastor-Garcia, A. Morales-Rubio, M. de la Guarda
Title of the paper	Sample Preparation Improvement in Polycyclic Aromatic Hydrocarbons Determination in Olive Oils by Gel Permeation Chromatography and Liquid Chromatography with Fluorescence Detection
Journal, Volume, Pages	Journal of AOAC International, 88, 1247-1254
Category	Method development
Ref. to Official method	
Official method	
Contaminant(s)	EPA-PAHs
Matrix	Olive Pommace oil
Extraction method	LLE
Extraction solvent	Method (1): ACN, Method (2): CH ₂ Cl ₂ , Method (3): ACN
Conditions of extraction (T, P, sorbent)	Methods 1&3:1:10 dilution in solvent, than 3 min vortex, 5 min in ice bath and centrifuged at 2500rpm. Supernatant evaporated and resolved in 1:Hex, 3: CH ₂ Cl ₂ . Method 2: filtration through 0.22 um PP filter and GPC.
Purification method	1: SPE, 2: GPC, 3: GPC
Purification step	1: Hex, 2&3: CH ₂ Cl ₂

Conditions of purification (T, P, stationary phase, etc)	1: SPE on Si Plus Sep-Pak, final solvent in ACN, filtered 0.22 um PP filter. 2&3: GPC on Envirogel 300x19 and 150x19 mm in series, 10 nm pore size, 15 um nominal particle size. Final solution for all was ACN.
Analytical Technique	HPLC-FLD
More on analytical technique	Vydac C18 201TP52 250x2.1 mm, 5 um column, gradient elution with initial 50% ACN/H2O
Instrumental conditions	FLD with fixed emission wavelength 420 nm and multi excitation wavelengths
Quality parameters (LOD, LOQ, etc)	LOD for 1: 0.1-0.9 ng/g, 2: 0.07-0.6 ng/g, 3: 0.05-0.5 ng/g; Recovery for 1: 76-105%, 2: 75-111%, 3: 86-109%
Guideline for Calibration	external calibration between 0.2-10 ng/g
Guideline for Validation	
Guideline for Uncertainty	
Interesting points	Efficiency of extraction is better when extracting with ACN followed by GPC as powerful preparation method
Additional interesting points	
Comments	

YEAR OF PUBLICATION	2007
First Author	Y.-Y. Lin
Other authors	G. Liu, C.M. Wai, Y. Lin
Title of the paper	Magnetic beads-based bioelectrochemical immunoassay of polycyclic aromatic hydrocarbons
Journal, Volume, Pages	Electrochemistry Communications, 9, 1547-1552
Category	Survey study (environment)
Ref. to Official method	
Official method	
Contaminant(s)	PAH(s)
Matrix	Water
Extraction method	competitive immunoassay
Extraction solvent	200 µl of magnetic beads (MB) coated PAH-Ab + 100 µl PAH-horseradish peroxidase (HRP)conjugate + 100 µl PAH analyte
Conditions of extraction (T, P, sorbent)	solvents were mixed in centrifuge tube and shaken for 2 sec. Incubation at 30 min at room temperature under gentle shaking. PAH- and PAH-HRP associated MBs were poured out with magnetic field. MBs mixture was transferred to electrochemical cell.
Purification method	
Purification step	
Conditions of purification (T, P, stationary phase, etc)	
Analytical Technique	electrochemical measurement
More on analytical technique	Substrate 3,3',5,5'-tetramethylbenzidine-H2O2

Instrumental conditions	Three electrode system: working electrode: glassy carbon, reference electrode: Ag/AgCl, wire counter electrode: platinum
Quality parameters (LOD, LOQ, etc)	LOD: 0.02 ppb, precision: 2.4%RSD (n=6)
Guideline for Calibration	in range 0.05-1 ppb
Guideline for Validation	
Guideline for Uncertainty	
Interesting points	immunoaffinity based sample preparation with good sensitivity.
Additional interesting points	
Comments	rather laborious, however would be advantageous to utilize the specificity of immune reactions

YEAR OF PUBLICATION	2007
First Author	K.-C. Hung
Other authors	B.-H. Chen, L.E. Yu
Title of the paper	Cloud-point extraction of selected polycyclic aromatic hydrocarbons by nonionic surfactants
Journal, Volume, Pages	Separation and Purification Technology, 57, 1-10
Category	Survey study (environment)
Ref. to Official method	
Official method	
Contaminant(s)	Nph, Acp, Flr, Ant, Pnt, Fla, Pyr, BaA, BaP
Matrix	Water
Extraction method	cloud-point extraction
Extraction solvent	Tergitol 15-S-9, Neodol 25-7, Tergitol 15-S-7 surfactants
Conditions of extraction (T, P, sorbent)	~30 ml solution was extracted at 25°C for 48 hrs. Surfactant and PAHs were mixed previously. 20 ul aliquot from surfactant-rich phase directly injected into HPLC
Purification method	no additional purification
Purification step	
Conditions of purification (T, P, stationary phase, etc)	
Analytical Technique	HPLC-UV/VIS
More on analytical technique	Column: Tosoh ODS-100S, 250x4.6 mm, 5 um
Instrumental conditions	80% ACN/H2O as mobile phase at 1 ml/min
Quality parameters (LOD, LOQ, etc)	LOD~ 0.1 ug/ml; Recovery values between 80-130%
Guideline for Calibration	
Guideline for Validation	
Guideline for Uncertainty	

Interesting points	unconventional extraction method
Additional interesting points	
Comments	interesting from scientific point of view, but at the moment no practical application to complex food

Upcoming International Events

"CIES International Food Safety Conference"

4 to 6 February 2009 –Barcelona (Spain)

[CIES Conference](#)

"237th ACS National Meeting"

22 to 26 March 2009 - Salt Lake City, Utah (USA)

[ACS-AGFD Meeting](#)

"Second SAFE consortium International Congress on Food Safety: Novel Technologies and Food Quality, Safety and Health"

27 to 29 April 2009 – Girona (Spain)

[SAFE International Congress 2009](#)

"14th International Congress of Metrology: Added value through better measurement"

22 to 25 June 2009 – Paris (France)

[Metrology 2009](#)

"123rd Annual Meeting & Exposition"

13 to 16 September 2009 - Philadelphia Marriott Philadelphia – PA (USA)

[AOAC Annual meeting](#)

"ISPAC Conference"

20 to 24 September 2009 - Charleston – SC (USA)

[ISPAC 22](#)

"46th Congress of the European Societies of Toxicology"

13 to 17 September 2009 – Dresden (Germany)

[Eurotox 2009](#)

"ILAC/IAF Conference"

9 to 21 October 2009 – Vancouver (Canada)

[ILAC / IAF 2009](#)

"4th International Symposium on Recent Advances in Food Analysis (RAFA 2009)."

4 to 6 November 2009 – Prague (Czech Republic)

[Food Analysis Symposium](#)

Past Events

35th International Symposium on Environmental Analytical Chemistry - GDANSK (Poland), 22-26 June 2008. The conference was a good opportunity to meet a broad range of scientists developing and applying analytical techniques to investigate the impact of man's activities on different environmental compartments. Although focus on environmental samples, several oral presentations and posters dealt with the analysis of PAHs by using different analytical techniques such as HPLC with fluorescence and photodiode array detectors or GC coupled to mass spectrometry in selected ion monitoring (SIM) mode. Many of these studies focused on the analysis of the 16 PAHs recommended as priority pollutants by the Environmental Protection Agency (EPA) in air samples.

The CRL on PAHs participated to the conference with one poster:

"Optimisation of programmed temperature vaporization (PTV) injection for the analysis of the 15+1 EU Polycyclic aromatic hydrocarbons (PAHs)" by Jose Angel Gomez-Ruiz, Fernando Cordeiro and Thomas Wenzl"

Upcoming Training Activities

6 to 7 May 2009 - Geel (BE)

Use of reference material and the estimation of measurement uncertainty.

This course provides participants with the theoretical basis for the estimation of measurement uncertainty and establishment of traceability. The course is intended for laboratory managers and practitioners in analytical laboratories who use reference materials for statistical quality control, method validation and calibration and need to assess measurement uncertainties on customer's demand or as requirement of ISO 17025.

[Course leaflet](#)

Acknowledgements

For the compilation of this newsletter, the contributions of Patricia Lopez Sanchez, Jose Gomez Ruiz and Laszlo Hollosi for the review of some interesting papers, of Jose also for the "Past Events" session and of the NRLs of Germany and United Kingdom for the description of the Food Control Organisation in their countries are acknowledged.

Abstract

The Regulation 882/2004 reports at the point (18) of the introduction the following sentence: "The designation of Community and national reference laboratories should contribute to a high quality and uniformity of analytical results. This objective can be achieved by activities such as the application of validated analytical methods, ensuring that reference materials are available, the organisation of comparative testing and the training of staff from laboratories."

The CRL PAHs newsletter aims to help achieving this uniformly high level of competence in food control through an improved sharing of scientific information, implementing a mean of communication between the CRL and the NRLs and towards all the interested professionals (the newsletter will be published onto the CRL web-page) and encouraging collaboration from NRLs with contributions since the first issue of the newsletter.

The mission of the JRC is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.

