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Proceedings of the 9th workshop of the European Union Reference Laboratory & National Reference Laboratories for Heavy Metals in Food and Feed

Brussels, 9 September 2014

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Executive Summary

The 9th workshop of the EURL for Heavy Metals in Food and Feed (EURL-HM) was organised in Brussels on September 9, 2014. Forty five participants attended the event, representing 27 National Reference Laboratories (NRLs), the Directorate-General Health and Consumers (DG SANCO) and the EURL-HM.

The activities performed by the EURL-HM in 2014 were reviewed and the work program 2015 submitted to DG SANCO for approval was presented. The support to the European Committee for Standardization (CEN) and other standardisation activities was also summarised. Representatives from DG SANCO presented the recent and future developments of the EU legislation in the field of heavy metals (HM) in food and feed. The main topics of the meetings consisted in the presentation and discussion of the outcomes of the two Proficiency Tests (PTs) organised in 2014 for the determination of HM in canned peas (IMEP-118) and vegetable feed (IMEP-119). NRLs agreed to the organisation in 2105 of two proficiency tests for the determination of HM in chocolate and palm kern expellers. An invited lecture on the design of experiments providing several practical examples completed the agenda. All presentations are included as Annexes to this report.

Table of Contents

EURL
European Union Reference Laboratory
Heavy Metals in Feed and Food

Minutes of the 9th Workshop of the EURL-HM.

Welcome and opening of the event

The 9th annual workshop of the European Union Reference Laboratory for Heavy Metals in Food and Feed (EURL-HM) was organised in Brussels on the 9th of September 2014. P. Robouch, operating manager of the EURL-HM, welcomed the thirty-five National Reference Laboratory (NRL) representatives from 28 countries (including Norway) and the two representatives from DG SANCO: F. Verstraete and F. Swartenbroux, responsible for the European legislation for contaminants in feed and food, respectively. Apologies were received from Malta.

At present, the EURL-HM network consists of 47 NRLs from 28 member states plus Norway. All NRLs were requested to provide by the 1st of October 2014 an updated list of NRL contact person.

The new EURL-HM web page, managed by the Joint Research Centre (JRC) headquarters, was presented to the audience (https://ec.europa.eu/jrc/en/eurl/heavy-metals). Participants were also informed about the CIRCABC platform - the Communication and Information Resource Centre for Administrations, Businesses and Citizens (https://circabc.europa.eu) - to be used in the future to exchange information between the EURL-HM and the NRLs (e.g. announcements of proficiency tests, workshops, or exchange of documents).

EURL-HM 2014 activities and Work Programme 2015

Participants were informed about the activities carried out by the EURL-HM in 2014, namely:

- IMEP-118, a proficiency test (PT) for the determination of total As, Cd, Pb, Hg, Sn and inorganic As (iAs) in canned food.
- IMEP-119: a PT for the determination of total As, Cd, Pb and Hg in vegetable feed.
- Participation in the meetings of the Working Group of National Experts in Environmental and Industrial Contaminants in food.
- Support to standardisation and attending meetings of CEN TC 275/WG 10 (dealing with methods for trace elements in food) and of CEN TC 327/WG 4 (dealing with methods for trace elements in feed).
- Invited lecture by P. Robouch at the VDLUFA annual workshop (April 2014) and at the EURL-CEFAO annual workshop (October 2014).

The activities included in the EURL-HM work program 2015 were also presented:

- IMEP-120, a PT for the determination of total As, Cd, Pb and Hg and of iAs in chocolate.
- IMEP-121, a PT for the determination of total As, Cd, Pb, Hg and iAs in kaolinitic clay.
- Ad hoc support to NRLs, DG SANCO, CEN and EFSA.

The EURL-HM will continue the collaboration with two other EURLs sharing common interests, namely the EURL-CEFAO (ISS, Rome - Italy) and the EURL-Feed Additives (IRMM, Geel - Belgium).

Suggestions for PTs in 2016 and for training courses in 2015

Before the workshop, participants were asked to express their wishes for (i) PTs to be organised in 2016 and (ii) trainings to be included in the 2015 workshop.

A variety of PTs for the determination of HM in food and feed matrices were suggested. Several of them (tea, cereals, bakery commodities, and iAs in seaweed) were already organised in the recent past. From the remaining ones, the EURL-HM proposed to organise the following PTs: (1) HM in seafood (including the determination of methylmercury); and (2) HM in palm kernel expeller. This proposal was unanimously accepted by all participants.

P. Robouch asked for some clarification to the DG SANCO representatives regarding the proper understanding of "Heavy Metals". Should the EURL-HM take into consideration the request made by several NRLs and organise PTs for the determination of Ni, Cr, Co and/or Se in food and/or feed? Knowing that Co and Se are trace elements also authorised as feed additives. F. Verstraete suggested that such issues need to be agreed by the two EURLs on a case-by-case basis.

As for the training needs, the following suggestions were retained:

- Method validation focusing on limit of detection (LOD) and limit of quantification (LOQ) and uncertainty calculations. P. Robouch informed the network that four EURLs supporting the European legislation on contaminants in food and feed (HM, Mycotoxins, PAH's and PCBs & dioxins) were requested by DG SANCO to draft a guidance document on "how to calculate LOD and LOQ".
- Significant digits.

B. de la Calle reminded that a training on homogeneity and stability of PT test items was provided in a previous workshop. The presentations are included in the corresponding proceedings available from from https://ec.europa.eu/jrc/en/eurl/heavy-metals (see "Related Publications").

P. Robouch announced that the second version of the Eurachem guide on "The Fitness for Purpose of Analytical Methods" was recently published (https://www.eurachem.org/index.php/publications/guides/mv).

General information

P. Robouch summarised the outcome of the survey carried out by the EURL-HM to gather information about the activities of the NRLs in the framework of their official mandate, particularly PTs and workshops organised by the NRLs. This survey was requested by DG SANCO. Thirteen NRLs did not organise PTs in their countries due to the small number of official control laboratories (1 or 2). Eleven NRLs organised at least one PT/year. Some others outsourced the organisation of PTs to PT-providers, such as FAPAS, IMEP or EURLs. Similar numbers described the organisation of workshops. Participants were invited to share the reports of their PTs and their workshop proceedings on CIRCABC. Furthermore, NRLs could provide information about their contribution/participation to relevant international fora dealing with HM analysis.

In December, the EURL-HM will send instructions to NRLs on how to access the CIRCABC platform. Only designated NRL representatives will have access.

Recent and future developments of EU legislation in the field of HM in food

F. Swartenbroux presented the recent changes in the European legislation for contaminants in food.

Arsenic

Regarding maximum levels (MLs) for inorganic arsenic (iAs) in rice, the expert committee agreed that the following three categories should be considered:

- White and brown rice
- Puffed rice products
- Rice for infants and young children

Levels range between 0.10 mg kg^{-1} (for rice for infants and young children other than husked/brown rice) and 0.30 mg kg^{-1} (for puffed rice, rice wafers, rice crackers and rice doughnut derived from milled rice). MLs for "brown vs. white rice" and for "parboiled vs. non-parboiled rice" need to be agreed. The iAs content in the bran is higher than in the rice grain, and higher iAs levels seem to occur after steaming of parboiled rice due to the iAs migration from the bran to the grain.

Cadmium:

The revision of Regulation (EC) No 1881/2006 focusses on two commodities having no MLs for Cd:

- Chocolate and cocoa products: MLs will vary from 0.1 to 0.8 mg kg^{-1} depending on the percentage of dry cocoa solids. An ML of 0.60 mg kg^{-1} will also be set for cocoa powder sold to the final consumer.

- Baby food: different MLs will be set for the powdered and the liquid formulae.

Minor adjustments of MLs for Cd were done for some other food commodities. For the moment no MLs were introduced for most of the contributors to exposure (vegetables and cereals). This will allow farmers and business operators to take the right measures in order to decrease Cd levels. However, a recommendation was published to apply existing mitigation measures and to initiate relevant research in the field. The situation will be reassessed before December 2018.

Lead:

According to the EFSA opinion, the intake of Pb from the diet should be reduced to avoid neurodevelopmental problems to unborn children, infants and children. The main Pb contributors to the diet are cereals and vegetables (in particular potatoes) and to a lesser extent meat and fish. As for the children diet, milk and milk products, baby foods, fruits, fruit products and fruit juices are the main Pb contributors. New MLs have been proposed during the recent stakeholder consultation.

Mercury:

Regarding Hg, NRLs were informed that EFSA was request by SANCO to assess the beneficial effects of fish consumption.

Recent and future developments of EU legislation in the field of HM in feed.

F. Verstraete presented the updates of the European legislation for HM in feed. A new sampling procedure for feed and new methods of analysis were included. A review of the methods of analysis included in Regulation (EU) No 691/2013 (superseding Regulation (EC) No 152/2009) is on-going to check whether some of methods included are obsolete. NRLs were asked to read the Regulation and to provide feedback to the EURL-HM. Regulation (EC) No 152/2009 includes one method for the determination of trace elements (Fe, Cu, Mn and Zn) in feed. If major modifications of the methods are deemed necessary, it may be removed from the legislation, since alternative CEN methods, which are ring-trial validated, are available (e.g. CEN 15510, CEN 15550 and CEN 15621).

Amendments of Directive 2002/32/EC and the recent RASFF notifications were shortly presented.

Training course on experimental design

F. Cordeiro gave an invited lecture on the "experimental design" and provided several practical examples.

Support to CEN and other standardisation activities

B. de la Calle informed NRLs about the activities carried out by the CEN TC 275/WG 10 on trace elements in food:

- Validation of a method for the determination of iAs in food by HPLC-ICP-MS (final stage).
- Validation of a method for the determination of methylmercury in food by GC-ID-ICP-MS (ongoing).
- Revision of some existing standards (on-going).
- A technical report will be published on a method for the determination of iAs in rice by HG-AAS.

and by the CEN TC 327/WG 4 on trace elements in feed:

- The work of the EURL-HM network is mentioned/acknowledged in Regulation (EU) No 1275/2013. NRLs were informed that according to that regulation the determination of Pb in feed containing kaolinitic clay is to be carried out applying partial extraction with 5 % HNO_3 at boiling temperature for half an hour. The Standard Operating Procedure (SOP) distributed to participants of IMEPs 103, 105 and 108 (on the determination of total and "partial" contents of Cd and Pb in feed) should be used. This SOP is described in the corresponding IMEP reports (i.e. https://ec.europa.eu/jrc/sites/default/files/eur23711en.pdf).
- In the frame of the mandate M522, the EURL-HM will draft a guide on the application of the "Criteria approach for methods for analysis of HM in feed".

B. de la Calle also presented the outcome of IMEP-41, a collaborative trial for the determination of iAs in food using sequential extraction with further determination by HG-AAS. The method was successfully ring-trial validated and the SOP will be available from the EURL-HM web page in 2015 (https://ec.europa.eu/jrc/en/interlaboratory-comparison/imep-41). This method will complement the HPLC-ICP-MS method currently being validated by CEN.

Outcome of IMEP-118 and IMEP-119

I. Fiamegkos and F. Cordeiro presented the outcome of two PTs organised by the EURL-HM in 2014 for the determination of HM in canned food and in vegetable feed (IMEP-118 and IMEP-119, respectively). The IMEP-118 and IMEP-119 draft reports were sent to participants before the workshop, allowing NRLs to review the report and their performance evaluation. In general, the overall performance of NRLs is better than that of other participating laboratories for all HM in both matrices.

Two enthusiastic discussion groups were formed around the posters of IMEP-118 (chaired by I. Fiamegkos) and IMEP-119 (chaired by F. Cordeiro), where participants actively contributed. The following main topics were later presented in plenary by the two chairs:

IMEP-118 – HM in canned vegetables:

- The discussion started with a question from a participant about *"What is considered the correct sampling approach for this kind of samples - the drained product or the solid/liquid composite?"* This question triggered a lively discussion around the interpretation of existing official methods and the lack of a relevant protocol or guidelines for the analysis of canned food in brine or sauces. The explanation of what is considered "edible in a can" should not rely on the eating habits of the analyst but should be based on clear technical instructions. However, the complexity of the matrix to be analysed must be taken into consideration when drafting such instructions.
- Participants mentioned that only one certified reference material was commercially available for the analysis of Sn in food (ERM-BC084a Sn in tomato paste). A discussion followed on the importance of using appropriate RM for analysis. Spiking the sample could be an alternative in the absence of appropriate RMs.
- The discrepancy between the theoretical (by formulation) and the reference values of iAs in the solid /liquid composite was discussed. Participants explained that such a difference could be attributed to the formation of thioarsenates. During the discussion it became evident that many participants analysed also the brine of the sample and promised to report their results to the EURL-HM. Additional results are expected from one of the laboratories contributing to the reference value assignment of the test item in October.

The Danish NRL for HM in food congratulated the EURL-HM for the "professional organisation of the IMEP-118, a challenging and informative PT".

IMEP-119 – HM in vegetable feed:

- It was suggested that the LOQ should be asked to participants instead of the LOD. These method performance characteristics should be better defined to allow a common interpretation by all participants. As mentioned earlier, a guidance document will be drafted by the EURLs for contaminants in food on "How to calculate LOD and LOQs". This will be useful also for feed analysis.
- Regarding the compliance to Directive 2002/32/EC some concern was raised by the participants since the MLs specified in this Directive refer to a moisture content of 12 %, while the PT test item distributed to participants had a moisture content of less than 4 %. It was not clear whether the results should be corrected for the moisture content (applying the oven drying method provided by the EURL-HM or assuming 12 % by default) before assessing compliance. F. Verstraete informed that, in particular for feed, Directive 2002/32/EC clearly indicates that

MLs are applicable for feed containing 12 % moisture content, so any compliance statement should take this into account. Furthermore, the Dutch NRL for feed pointed out that laboratories could not check the validity of their routine procedure for the moisture content determination when another procedure was recommended by the EURL-HM. It was therefore decided that no SOP for moisture determination will be distributed by the EURL-HM in the future. Laboratories will have to report moisture content as an additional measurand. This information will be used when scrutinising possible biases.

Selenium in feed

The German NRL presented an analytical method for the selective determination of selenium species, (including organic selenium compounds) in feed and discussed the difficulties associated with this type of analyses.

Wrap-up

P. Robouch informed participants that a report on the "Implementation of Art. 33 of Regulation (EC) No 882/2004 in the EU member states in the areas of HM in food and feed" (Report JRC90090) was submitted to DG SANCO and will be distributed to NRLs shortly.

Executive summary.
DG SANCO request to provide (i) an overview of MS which did not appoint NRLs; (ii) a review of NRL activities performed in the frame of their mandate, including the organisation of PT and the follow-up of non-compliant results reported by OCL. The EURL-HM network consists of 47 NRLs. All countries attend systematically the annual workshops and participate to PTs organised by the EURL-HM since 2007. The thorough review of NRL performances for the determination of total mass fraction of As, Cd, Hg and Pb in food and feed matrices clearly demonstrates the high quality of the analytical capabilities of the network laboratories. Some challenging matrices were identified and will be closely monitored. Finally, all NRLs declared fulfilling their mandate set by Regulation (EC) 882/2004, Articles 33.2, but only fourteen of them (managing a network of several national official control laboratories) organise and follow-up non-compliant results on a yearly basis.

In order to comply with the request by DG SANCO regarding the monitoring of under-performing laboratories (having a |zeta-score| > 3) a follow-up form will be sent to the concerned laboratories allowing them to describe (i) the root-cause analysis they have undertaken; (ii) the corrective actions they have implemented; and (iii) the demonstration of effectiveness of these corrective actions (when available).

P. Robouch closed the event wishing participants a safe journey home and asked them to fill at their earliest convenience the e-satisfaction survey (http://ec.europa.eu/eusurvey).

Note: All the presentations included in the Agenda, the two surveys together with the follow-up form are provided in the Annexes.

Minutes prepared by Beatriz de la Calle (24/09/2014)
Minutes reviewed by Piotr Robouch (24/10/2014)

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9th EURL-HM Workshop

Tue. 9 Sep. 2014 - Brussels, CCAB

08:30 - 09:00	Registration	
09:00 – 09:10	Welcome & Opening of the Event	
09:10 – 09:40	2014 Activities & WP 2015	Piotr Robouch
09:40 – 10:10	Legislation on HM in FOOD Legislation on HM in FEED (SANCO)	Frank Swartenbroux Frans Verstraeten
10:10 – 10:30	General Info: Relevant International Activities.	Piotr Robouch
10:30 – 11:00	*Coffee*	
11:00 – 12:00	Experimental Design	Fernando Cordeiro
12:00 – 12:30	HM in Feed & Food - CEN activities	Beatriz de la Calle
12:30 – 14:00	*Lunch*	
14:00 – 14:10	Se in feed – Analytical problems in discriminating organic from total Se	Timo Kapp
14:10 – 14:30	Review of NRL performances	Piotr Robouch
14:30 – 16:00	Presentation & Discussion • IMEP 118 – Canned Vegetables • IMEP 119 – Vegetable feed • **Feedback in plenary** moderated by	 Ioannis Fiamegkos Fernando Cordeiro Beatriz de la Calle
Coffee available during poster presentations & discussion		
16:00 – 16:30	Future activities & Wrap-up	Piotr Robouch
16:30	Closing of the event	

European Union Reference Laboratory
Heavy Metals in Feed and Food

Evaluation of the Satisfaction Survey of the EUL-HM workshop 2014

Participants appreciated the various topics included in the Agenda and acknowledged the quality of the proficiency testing exercises organised in 2014. The constructive comments presented hereafter will be taken into consideration in order to give more time for "networking".

	Best					Worst	
Was the scientific program of the WS 2014 appropriate	1	2	3	4	5	6	
1a. Discussions related to **2014 PT** results (IMEP-118 & IMEP-119)	15	16	1	0	0	0	good/bad
1.b Presentation of (next) PTs included in the Workp Program 2015	11	16	5	0	0	0	good/bad
1c. Identification of future PTs for 2016	6	21	4	1	0	0	good/bad
1d. Training on Statistical Experimental Design	3	6	14	7	2	0	good/bad
1e. Relevance of the various topics to the tasks of your NRL	11	13	8	0	0	0	good/bad
1f. Time dedicated to Presentations & Discussions	11	14	5	0	2	0	good/bad
1g. Overall rating of the organisation & structure of the WS 2014	16	13	2	1	0	0	good/bad
1h. The event provided me with Networking opportunities	6	18	7	1	0	0	agree/disagree
1i. The event improved my knowledge and expertise in my field of science and research	8	15	7	0	2	0	agree/disagree
Your opinion about the 2014 PTs							
2a. Description of samples & tasks of the 2014 PTs	15	14	3	0	0	0	good/bad
2b. Timing of the 2014 PTs	17	14	0	1	0	0	good/bad
2c. Communication with the EURL during the 2014 PTs	21	10	1	0	0	0	good/bad
2e. Evaluation of the PT report(s) to participants	20	10	2	0	0	0	good/bad
2f. Timing of publication of the preliminary PT reports (before the Workshop)	21	9	1	1	0	0	good/bad
2g. Timing of publication of the final PT reports (expected October 2014)	16	13	2	1	0	0	good/bad
2h. Would you agree replacing proceedings (paper) printouts by e-proceedings (pdf)? Please consider ecological reasons & remember that presentations will be available after the WS.	17	10	5	0	0		agree/disagree
2i. Overall rating of the 2014 PTs	14	18	0	0	0	0	good/bad
2j. Capability & handling of the MILC interface for registering and reporting results	13	14	4	0	1	0	good/bad
(optional) Your opinion about chapters, topics or information in the PT reports. Points to be covered in more details? Additional topics to be included? Please specify.	3 comments						
Your opinion about the EURL-HM webpage							
Did you visit the EURL-HM **webpage**?	27	5					yes/no
Do you find the EURL-HM website useful	8	19	4	1	0	0	useful/not useful
According to you, which information is most useful?	**13 comments**						
According to you, which information is MISSING? (which info should be added?)	**3 comment**						
Logistics							
Meeting place	18	12	2	0	0	0	good/bad
Meals	4	12	13	1	1	1	good/bad
Desk assistance during the meeting	21	9	2	0	0	0	good/bad
Communication with the IRMM/EURL - for logistics, transport, hotel, other info	18	12	1	1	0	0	good/bad
How was the IRMM/EURL reaction to your questions?	18	12	2	0	0	0	good/bad
Ypour opinion matters							
Would you like to suggest some improvements/changes	**9 comments**						

Note: All comments transcribed on the next page

Comments

	(optional) Your opinion about chapters, topics or information in the PT reports. Points to be covered in more details? Additional topics to be included? Please specify.
I suppose that question 1j is not related to the PTs but to the workshop (handouts). Replacing the paper printouts by e-proceedings will not necessarily positively influence the environmental aspects: I will e.g. print them out anyway because it is most handy to note comments/remarks directly on the slide handouts.	
First time at the meeting and very satisfied with the organisation and the subjects evoked. More than happy to see a training on statistical experimental design, but for uninitialized persons, the training was not easy to follow. An accompanying example (of the field of expertise of trace elements in food) would have been a valuable plus.	
All information was very useful	(3)

EURL-HM website	According to you, which information is most useful?
list of methods of analysis	
The documentation on the annual workshops	
PT reports, workshop report	
The overview of concerned legislation	
I didn't visit webpage yet.	
legislation information, interlaboratory comparison publications	
up to date legislation	
All of them.	
legislation part but need to be updated and enriched	
It is interesting to see what other PTs there are going on	
Interlaboratory comparisons, legislation and list of official methods	
Past PT's and regulations	
All information in one place about IMEP	(13)

EURL-HM website	According to you, which information is MISSING? (which info should be added?)
events organized by NRLs (national WS, conferences...)	
I miss the latest version of legislation. I would appreciate not only a list of the official methods but also texts of the methods or methods in your use.	
Maybe it is possible to put direct link to last version of "The Fitness for Purpose of Analytical Methods. A Laboratory Guide to Method Validation and Related Topics"	(3)

Would you like to suggest some improvements/changes	
It would be very interesting to meet at the laboratory site of a NRL colleague	
Information about resonsibilities and activities in other EURLs and NRLs involved in monitoring and the analysis of heavy metals and other metals in food and feed would be useful. Also links to thoe web sites.	
Although I like to travel, I like the venue in Brussels because it is easy to reach, while some NRLs might not.	
Put the discussions about PT results earlier in the agenda. Not very much of discussions in plenum. If main objective is to supply information it would be better make a movie/stream the presentations and we would not have to travel to the meeting.	
more time is needed for discussion, and contacts with other NRL's	
The work shop agenda was too busy for 1 day.	
The establishment of a more effective communication network between members should enrich the debate during the annual workshop	
Future the workshop would take place in some NRL	
At the poster session, I found it difficult to access both the presenters and the posters due to space limitation. Is it possible to organize the poster session in the corridor, for example, where there is more space to be able to gather around the posters, please? Thank you.	(9)

List of Participants

Name	Organisation	Country
Ernst Schmeisser	AGES	Austria
Karlien Cheyns	CODA-CERVA	Belgium
Nadia Waegeneers	CODA-CERVA	Belgium
Borislav Blazhev	Central Laboratory for Chemical Testing and Control	Bulgaria
Anica Benutić	Croatian National Institute of Public Health	Croatia
Marija Sedak	Croatian Veterinary Institute	Croatia
Dimitris Stefani	State General Laboratory, Cyprus	Cyprus
Eva Niedobova	CISTA	Czech Republic
Alena Simakova	State Veterinary Institute Olomouc,Lab.Kromeriz	Czech Republic
Rie Romme Rasmussen	Technical University of Denmark, National Food Institute	Denmark
Inge Rokkjær	Danish Veterinary and Food Administration	Denmark
Merike Toome	Agricultural Research Centre	Estonia
Tarja Kousa	Finnish Food Safety Authority Evira	Finland
François Auger	Laboratoire SCL de Bordeaux	France
Rachida Chekri	National Agency	France
Laurent Noel	ANSES	France
Timo Kapp	Federal Office of Consumer Protection and Food saf	Germany
Eleni Psomiadou	General Chemical State Laboratory	Greece
Eva Táborhegyi	National Food and Feed Safaty Office	Hungary
Frederick Davidson	Cork Public Analyst's Laboratory	Ireland
Paola Brizio	IZS PLV	Italy
Konstantins Bavrins	Institute of Food Safety, Animal Health and Enviro	Latvia
Birute Miliauskaite	National Food and Veterinary Risk Assessment Insti	Lithuania
Veronika Fekete	Scientific Institute of Public Healh, Brussels, BE	Belgium/Luxemburg
Martijnvan der Lee	RIKILT	Netherlands
Heidi Amlund	NIFES	Norway
Monika Mania	National Institute of Hygiene (NIPH-NIH)	Poland
Agnieszka Nawrocka	National Veterinary Research Institute	Poland
Gabriela Assis	Laboratório de Controlo da Alimentação Animal	Portugal
Soniea Ciocilteu	Hygiene and Veterinary Public Health	Romania
Peter Očenáš	Veterinary and food institute	Slovakia
Katarina Pavšič Vrtač	National Veterinary Institute	Slovenia
Manuela Mirat	Laboratorio Arbitral Agroalimentario	Spain
Joakim Engman	National Food Administration	Sweden
Malcolm Baxter	The Food and Environmental Research Agency (FERA)	United Kingdom
Aneta Cizek-Stroh	JRC-IRMM	EC
Fernando Cordeiro Raposo	JRC-IRMM	EC
Beatriz De La Calle	JRC-IRMM	EC
Ioannis Fiamegkos	JRC-IRMM	EC
Piotr Robouch	JRC-IRMM	EC
Frans Verstraete	DG SANCO	EC
Frank Swartenbroux	DG SANCO	EC

Annex – Presentations

1. Welcome to the EURL-HM workshop 2014

Piotr Robouch

Welcome

9th Workshop of the EURL for Heavy Metals in Food & Feed

Brussels, 9 Sept. 2014

M.B. de la Calle, A. Cízek-Stroh
F. Cordeiro, I. Fiamegkos,
& P. Robouch

Our Network

European Union Reference Laboratory
Heavy Metals in Feed and Food

- 47 NRLs
- from 28 MS + NO
  - ➢ total of 135 collaborators (Director, Contact, Experts)

- Food of non-animal origin (incl. wild caught fish)
- Feed

Today:
- ✓35 participants
- ✓All MS represented (*apologies from MT*)

New web site

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EURL for Heavy Metals in feed and food

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

EURL

EURL heavy metals

- Legislation
- Network laboratories
- Interlaboratory comparisons
- Contact

EURL mycotoxins

EURL Polycyclic Aromatic

EURL-HM

Heavy metals may reach the food chain through their natural and anthropogenic presence. Some heavy metals have nutritional functions and are essential to health. But others such as lead, cadmium and mercury have no nutritional relevance and need to be monitored as their presence in the atmosphere, soil and water, even in traces can cause serious problems to all organisms. Heavy metals enter the human body mainly through ingestion of water and food. This can have a significant impact on human health.

Challenging

- *To get our dbase of contacts up-to-date*
- *All (relevant) NRLs participating to PTs*
- *All MS represented at the WS*

❑ ***Difficulties to arrange travel*** ➔ *contact EURL asap!*
to ensure (i) participation (ii) at descent price
- avoid last moment arrangements
- OR send official letter for non-participation

❑ ***Future communication through CIRCABC***
i.e calls for PT, WS, exchange of documents, other info

❑ ***Provide all contact info to EURL,*** *by 1st Oct. 2014*
NB: 47 NRLs contacted; 19 pending, some to be updated today

⚐ ***NO news from the EURL since long?***
contact **JRC-IRMM-EURL-HEAVY-METALS@ec.europa.eu**

Administrative formalities

Travel Reimbursement

❖ *Provide all relevant documents*
(travel ticket; legal identity; Bank info)
properly signed/stamped

➢ *Contact Aneta Cizek-Stroh*

⚐ *Please do not delay the process*

The Menu

- *2014 Activities & WP 2015*
- *On-going Legislation (by SANCO)*
- *General Info - cf. international activities*
- *Lecture on Experimental Design*
- *CEN activities – HM in Food & Feed*
- *IMEP-118 & 119: Presentation & Discussion*
- *Future activities*

+ "Se in feed" (by BVL)

Let the Workshop Begin

2. Activities in 2014

Work Program 2015

EURL-HM team

EURL-HM
2014 activities
& WP 2015

M.B. de la Calle, A. Cizek-Stroh,
F. Cordeiro, I. Fiamegkos
& P. Robouch

2014 Activities

Campaign	Description	Type	Status
IMEP-41 2014 Restricted	Determination of inorganic Arsenic in food	Method Validation	Ongoing
IMEP-119 2014 Open to All	Determination of total As, Cd, Pb and Hg in vegetable feed	Proficiency Test	Ongoing
IMEP-118 2014 Open to All	Determination of total As, Cd, Pb, Hg, Sn and iAs in canned food	Proficiency Test	Ongoing

- 2 PTs
- 9th WS
- "Environmental & Ind. Contaminants" expert meeting
- CEN 275/WG10 (food) & 327/WG4 (feed)
- Invited lecture at the VDLUFA Annual WS (Apr 2014)
- Invited Lecture at the EURL-CEFAO WS (Oct 2014)

WP 2015

- PT1(#) = HM in dark **chocolate**
- PT2(#) = HM in **mineral feed** containing kaolinitic clay
- **Follow-up** *unsatisfactory* results reported in 2014 PTs
- Organisation of the **10th WS** (Sep. 2015)
- Support to NRLs (when requested)
- Support to DG SANCO, EFSA and CEN
- Collaboration with EURL-CEFAO & EURL-FA

(#) Results due before July (dates *tbc*)

Your answers

Topics
1. Dinner together (maybe next time)
2a. EURL PTs for 2016
2b.Training for 2015
3a. NRL-PTs in 2014
3b. NRL-WS in 2014
3c. NRL other activities
Did we miss something?

Ready for the EURL WS 2014

PTs for 2016 suggestions

Food	Feed	Fish
• Cereals • fruits/juices • Tea • Backery commodities • Potatoes/root veg • iAs in seaweed • Vegetable oil 4 food	• Vegetable feed (palm kernel expellent) • Vegetable oil 4 feed	• Sea food • Seashell • Shell fish ➔ MetHg

- Several PTs already organised in the recent past
- For matrices of food of animal origin – contact EURL-CEFAO
- Ni, Cr, Co, Se

Training for 2015 suggestions

- *Method validation (**LOD/LOQ**; Measurement Uncertainty)*
- *Quality Control*
- *PT organisation (Homogeneity/Stability)*
- ***Significant Digits***
- *Risk Management*
- *Quantification in practice (calibration, blank subtraction, use of internal standards)*

- https://ec.europa.eu/jrc/en/**trainmic** (in several countries)
- https://ec.europa.eu/jrc/en/event/training-course/**use-reference-materials-and-estimation-measurement-uncertainty**-8-9102014?search (Oct. 2014)
- http://www.**eurachempt2014**.de/ (Oct. 2014)
- http://www.pt-conf.org/ (Sep. 2015)

To be discussed today

- ❑ *Trace elements & Nano (food & feed)*
- ❑ *Analysis of freeze dried food*
- ✓ *Sample Preparation of canned food*
- ✓ *Assignment of Reference Values*
- ✓ *New CEN standards*
- ➢ *LOD/LOQ (soon)*

NRL PTs 2014

	Countries
No PTs	BG; HR; CY; EE; FI; IE; LT; LUX; MT; NO; PT; SI; SE
At least one	BE (wine); CZ (feed); DK (feed); FR (mollusk; feed; food); DE (rice); GE (kidney); HU (mixed feed); IT (Bread CRM); PO (pig liver; veg/meat; iAs in food); RO (Honey); SK (?); ES (14 elements in feed and fish meal)
outsourcing	To FAPAS, IMEP, EURL-HM

- Not all NRLs replied to the Survey
- Please send the **PT e-report** to the EURL for information
  idem for **NRL Annual Report** - when available, no need to translate
- Will be uploaded **CIRCABC**

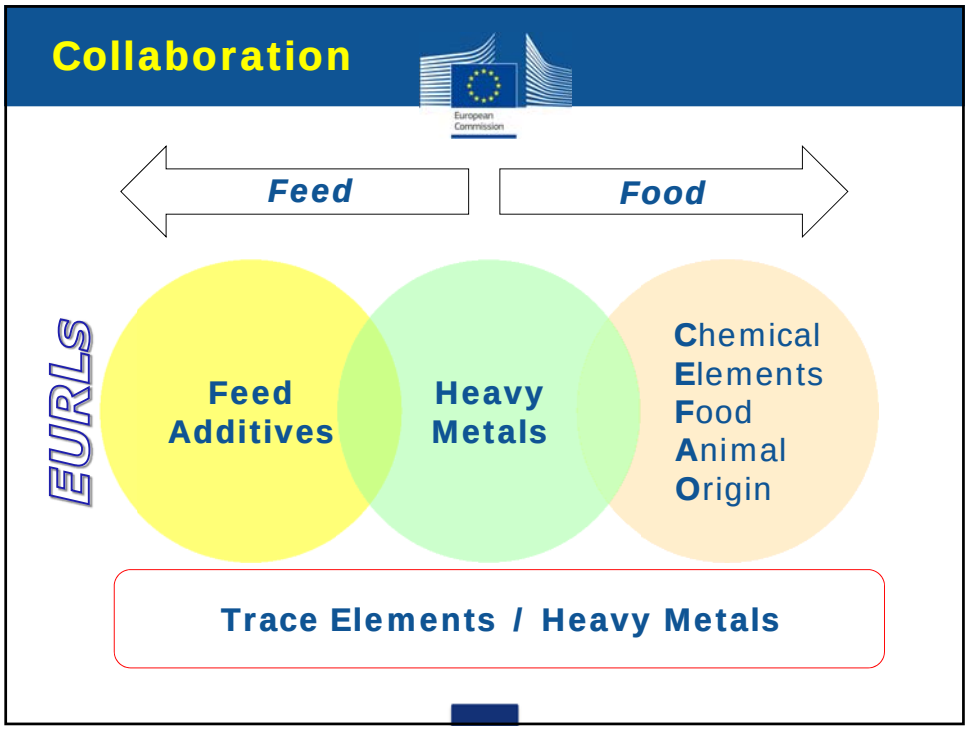
NRL WS 2014

	Countries
Yes	BE; HR; CZ (2); DK(2); DE; GR; IE (informal); IT; PO (2+); RO; SK; ES
No	BG; CY; EE; FI; FR; HU; LT; LUX; MT; NO; PT; SI; SE; UK

- Not all NRLs replied to the Survey
- Could you share your proceedings?
- Could be uploaded on **CIRCABC**

NRL Additional Request

- ✓ *Latest information from SANCO*
- ✓ *Methods for iAs determination*
- ❑ *Distribution of non-commercial reference materials (??)*
- ❑ *Information from EFSA (which one?)*
  - *- risk assessment for Human & Animal Health (cf. BIOCONTAM)*
  - *- collection of data HM in food & Feed (Evidence Management Unit)*


Collaboration
European Commission
Feed
Food
EURLS
Feed Additives
Heavy Metals
Chemical Elements Food Animal Origin
Trace Elements / Heavy Metals

3. Recent & Future developments of EU legislation in the field of HM in food

Frank Swartenbroux (DG SANCO)

Directorate-General for
Health & Consumers

Recent and future developments of EU legislation in the field of heavy metals

Frank Swartenbroux

Arsenic

EFSA CONTAM 2009 scientific opinion

Discussion initiated in expert working group

CCCF suspended work for 2 y (additional data collection)

Further discussion in expert working group

Arsenic stakeholder consultation

Levels suggested for inorganic arsenic in

- Rice and rice products at 0,20 mg/kg

Except

- Brown rice & brown rice products : 0,25 mg/kg
- Rice & rice based foodstuffs for infants and young children other than brown rice: 0,10 mg/kg

Levels suggested for total arsenic in

- edible oils and fats : 0,10 mg/kg (Codex level)

Arsenic stakeholder reactions

On rice:

- **Alignment with international standards**
- **Analytical uncertainty**
- **Food for infants and young children (supply)**
- **Rice bran (ML needed?)**
- **Effect of parboiling ! Limited to white rice !**

On edible oils and fats

- **No major contributor**

Arsenic – expert committee first outcome

No ML needed for edible fats & oils

Differentiated MLs needed for

- **White versus brown rice**
- **Puffed rice products**
- **Rice infants & young children**

Arsenic – suggested levels, still under discussion

3.5	**Arsenic (inorganic)** (50)	
3.5.1	Milled rice and parboiled rice	0,20
3.5.2	Puffed rice, rice wafer, rice cracker and rice doughnut derived from milled rice	0,25
3.5.3	Husked (brown) rice	0,25
3.5.4	Puffed rice, rice wafer, rice cracker and rice doughnut derived from husked (brown) rice	0,30
3.5.5	Rice for infants and young children other than husked (brown) rice	0,10

'(50) Sum of As(III) and As(V)'

Arsenic – to be discussed further in expert committee

Specific ML for parboiled white rice

Feasibility of controls

- **Parboiled vs. non-parboiled**
- **Rice wafers: brown vs. white rice content**

Amendment to 333/2006 as suggested by EURL

Cadmium – EFSA scientific opinion 2009

- **Established a lower tolerable weekly intake** (TWI) of 2.5 µg/kg b.w. (previously 7 µg/kg b.w.)
- Concluded that this **TWI was exceeded by certain population groups**
- Recommended that **exposure to cadmium should be reduced at a population level**

8

Cadmium – main contributors to dietary exposure

All age groups: *potatoes, cereals and vegetables*

Children, adolescents, toddlers and adults: *chocolate ad cocoa products*

Infants: *ready-to-eat meals, infant formulae, milk and dairy products*

Adolescents/Adults: *fish/seafood, meat and offal*

9

Cadmium - review of 1881/2006

Focuses on commodities for which no MLs exist

Chocolate and cocoa products:

- **differentiated MLs depending on the percentage of total dry cocoa solids**
- **Levels applicable as from 1/1/2019**

Baby foods:

- **Differentated MLs for powdered vs. liquid infant formulae and follow-on formulae (from milk and soya) and processed cereal based foods for infants and young children**
- **Levels applicable as from 1/1/2015**

10

Cadmium – Chocolate and cocoa products

Higher levels proposed for darker chocolates

- **0,10 mg/kg < 30 % dry cocoa solids**
- **0,30 mg/kg ≥ 30 % and < 50 % dry cocoa solids**
- **0,80 mg/kg ≥ 50 % dry cocoa solids**
- **0,60 mg/kg cocoa powder sold to the final consumer**

11

Cadmium – Baby foods

Differentiated MLs for powdered versus liquid formulae

- **Powdered formulae from milk (0,010 mg/kg) and from soya (0,020 mg/kg)**
- **Liquid formulae from milk (0,005 mg/kg) and from soya (0,010 mg/kg)**
- **Processed cereal based foods and baby foods for infant and young children (0,04 mg/kg)**

12

Cadmium – Adjustments

For certain fish species:

- **sardine, bichique : 0,05 → 0,10 mg/kg**
- **swordfish, anchovy: 0,30 mg/kg → 0,25 mg/kg**
- **bullet tuna: 0,20 mg/kg → 0,15 mg/kg**
- **Bonito, common two-branded seabream, eel, grey mullet, horse mackerel, louvar, sardinops and wedge sole: 0,10 → 0,05 mg/kg**

For specific vegetables

*** parsnips, horseradish, salsify and celery: 0,10 → 0,20 mg/kg**

Cadmium – What about the major contributors?

No changes to the MLs for vegetables and cereals

to allow farmers and food business operators more time to put appropriate measures in place to bring cadmium levels down

→ Recommendation for application of existing mitigation measures and to initiate research and investigations to fill any possible gaps in knowledge on mitigation measures

→ reassess situation before 31 December 2018

Cadmium – net result

Commission Regulation (EU) No 488/2014 of 12 May 2014 amending Regulation (EC) No 1881/2006 as regards maximum levels of cadmium in foodstuffs (OJ L 138, 13,5,2014, p. 75)

Commission Recommendation of 4 April 2014 on the reduction of the presence of cadmium in foodstuffs (OJ L 104, 8,4,2014, p. 80)

Lead

The EFSA CONTAM Panel March 2010 opinion identified a need to reduce exposure due to concern over possible neurodevelopmental effects in young children

Population group mainly at risk:

- **unborn child**
- **Infants**
- **children**

Lead – main contributors

Very similar to cadmium:

- **Cereals**
- **Vegetables (in particular potatoes)**
- **Meat/Fish (to a lesser extent)**

Additional for infants/children:

- **Milk and milk products**
- **Baby foods (all kinds incl. formulae)**
- **Fruit and fruit products, fruit juices**

Lead – stakeholder consultation

Changes suggested for

- Infant formulae and follow-on formulae: 0,01 mg/kg in liquid product // 0,02 mg/kg in powdered product
- Fresh legumes: 0,2 mg/kg → 0,10 mg/kg
- Fruiting vegetables: 0,1 mg/kg → 0,05 mg/kg
- Brassica vegetables other than leafy brassica: 0,3 mg/kg → 0,10 mg/kg
- Berries and small fruit: 0,20 mg/kg → 0,10 mg/kg except for cranberries, currants, elderberries and strawberry tree

Lead – stakeholder consultation

Changes suggested for

- Fruit juice other than fruit juice from berries and other small fruit: 0,05 mg/kg → 0,03 mg/kg
- Wine: 0,20 mg/kg → 0,15 mg/kg

New maximum levels suggested for

- Processed cereal based foods for infants and young children and other baby foods: 0,1 mg/kg
- Honey: 0,1 mg/kg

No maximum levels suggested anymore for

- Fresh herbs.

Mercury

EFSA CONTAM Opinion (22 November 2012) on the risk for public health related to the presence of mercury and methylmercury in food

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

Assessment by EFSA on the beneficial effects of fish consumption ongoing

20

4. Recent & Future developments of EU legislation in the field of HM in feed

Frans Verstraete (DG SANCO)

Directorate-General for
Health & Consumers

Recent and future developments of EU policy on heavy metals in feed

Frans Verstraete

MLs of heavy metals in Directive 2002/32/EC on undesirable substances in feed

* Maximum levels (MLs) for arsenic (total), cadmium, fluorine, lead and mercury in all feed materials and compound feed
* MLs for arsenic (total), cadmium and lead in feed additives belonging to the functional group of trace elements
* MLs for cadmium and lead and mercury in feed additives belonging to the functional groups of binders and anti-caking agents of trace elements
* ML for fluorine in vermiculite.

Recent and foreseen changes to MLs of heavy metals in feed

* **Regulation (EU) No 744/2012**:
- Maximum levels (MLs) for arsenic (total), fluorine, lead and mercury in the feed material calcium and magnesium carbonate aligned with the existing MLs in calcium carbonate.
- Increase of the ML for arsenic in di copper chloride trihydroxide (tribasic copper chloride, TBSCC) all feed materials and compound feed
*increase of the level for lead in natrolite-phonolite (E566) (alighed with the ML of clinoptilolite of volcanic origin (E567)

Recent and foreseen changes to MLs of heavy metals in feed

* **Regulation (EU) No 744/2012 (continued)**:
- Increase of the ML for arsenic in complete feed for pet animals containing fish, other aquatic animals and products derived thereof and/or seaweed meal and feed materials derived from seaweed (10 mg/kg aligned with complete feed for fish and fur animals)→ footnote "upon request of competent authority, the responsible operator must perform an analysis to demonstrate that the content of inorganic arsenic is lower than 2 ppm)".

Recent and foreseen changes to MLs of heavy metals in feed

* **Regulation (EU) No 1275/2013** :
- Specific higher maximum level of arsenic, cadmium and lead for long-term supply formulations of feed for particular nutritional purposes with a concentration of trace elements higher than 100 times the established maximum content in complete feed (higher than normal complementary mineral feed: arsenic 12 → 30 ppm, cadmium 5→15 ppm and lead 15→ 60 ppm)".
- Increase of maximum level of arsenic in feed additve ferrous carbonate

Recent and foreseen changes to MLs of heavy metals in feed

* **Regulation (EU) No 1275/2013** :
- a significant difference has been identified by the EURL–HM between the analytical results obtained by the application of different extraction methods currently used for the determination of lead in kaolinitic clay and feed containing kaolinitic clay. Before, no significant differences were observed between the levels of heavy metals in mineral feed by the application of different extraction methods.

Recent and foreseen changes to MLs of heavy metals in feed

* **Regulation (EU) No 1275/2013** :

The maximum levels of heavy metals in feed relate "to an analytical determination of lead, whereby extraction is performed in nitric acid (5 % w/w) for 30 minutes at boiling temperature".

Therefore the footnote has been re-introduced to provide for the use of that specific method of extraction (or any other method of extraction with a corresponding extraction efficiency) for the determination of lead in kaolinitic clay.

Recent and foreseen changes to MLs of heavy metals in feed

* **Regulation (EU) No (to be voted in next standing Committee)**:

- Increase of ML of arsenic, fluorine and lead in calcareous marine shells

- Change of maximum level of mercury for fish, other aquatic animals and products derived thereof intended for the production of compound feed for dogs, cats, ornamental fish and fur animals (ML of 0,5 mg/kg 88% DM → wet weight)

RASFF – heavy metals in feed

*** 2014: 4 notifications (cadmium)**
- cadmium in celery stalks, complementary feed, ferrous sulphate and fish meal

*** 2013: 13 notifications**
- arsenic (1): peat for piglets
- cadmium (4): fish meal (2), complementary feed, zinc oxide
- lead (3): palm kernel expeller, processed animal proteins of venison, pet food
- mercury(5): shark cartilage powder, mineral poultry feed, fish meal (3)

RASFF – heavy metals in feed

*** 2012: 24 notifications**
- arsenic (16): sunflower meal, complementary feed (3), manganese oxide, canned pet feed (6), feed lime, fishmeal (2), algae meal, copper sulphate.
- cadmium (3): zinc sulphate (2), fish meal
- lead: mineral feed
- mercury(4): complementary feed for cats (1), canned cat feed (3)

152/2009 sampling and method of analysis

Method of analysis: Regulation 691/2013

Thank you for your attention !

5. General information from International Committees

Piotr Robouch

General Information from International Committees

P. Robouch

Useful links

*http://www.**eurachem**.org/*

- Guides, Leaflets (in different languages)

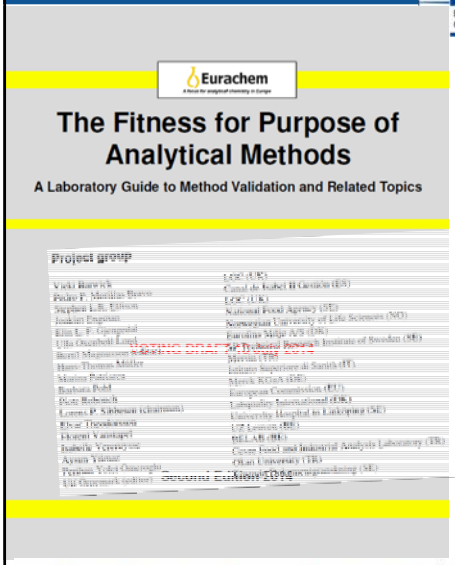
*http://www.**eurolab**.org/*

- cook books; publications

Related to measurement uncertainty, method validation, proficiency testing, traceability, sampling, quality control

Method Validation

*Voting draft **Jul 2014***


Eurachem

The Fitness for Purpose of Analytical Methods

A Laboratory Guide to Method Validation and Related Topics

Project group

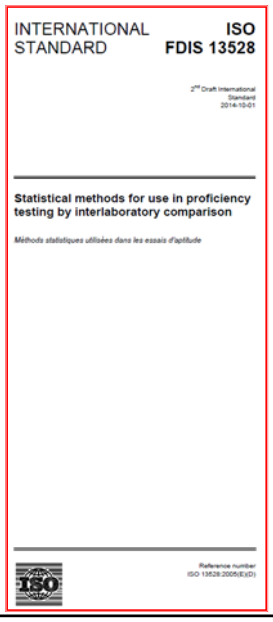
Table of Content *(simplified)*

- *What is method validation?*
- *When should methods be validated or verified?*
- *How should methods be validated*
- *Method performance characteristics*
- *Using validated methods*
- *Documentation*
- *ANOVA*

Provides practical examples & quick references

This publication should be cited" as: "B. Magnusson and U. Örnemark (eds.) Eurachem Guide: The Fitness for Purpose of Analytical Methods - A Laboratory Guide to Method Validation and Related Topics, (2nd ed. 2014). ISBN 978-91-87461-59-0. Available from www.eurachem.org."

ISO 13528


INTERNATIONAL STANDARD

ISO FDIS 13528

Statistical methods for use in proficiency testing by interlaboratory comparison

Méthods statistiques utilisées dans les essais d'aptitude

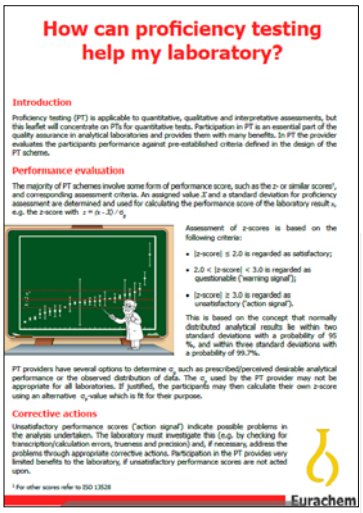
Table of Content *(simplified)*

- *General Principles*
- *Guidelines for Statistical design*
- *Homo; Stab; robust stats*
- *Determination of X_{pt} & u_{pt}*
  formulation; CRM; Results of one lab; consensus of expert labs; consensus from participants
- *Determination of σ_{pt}*
- *Calculation of performance stats*
  z; z'; zeta (ζ); E_n; ~~combined scoring~~
- *Graphical methods*
  histograms; kernel; Youden

Annex E (informative) Illustrative examples

Proficiency Testing

http://www.eurachem.org


How can proficiency testing help my laboratory?

Introduction

Proficiency testing (PT) is applicable to quantitative, qualitative and interpretative assessments, but this leaflet will concentrate on PTs for quantitative tests. Participation in PT is an essential part of the quality assurance in analytical laboratories and provides them with many benefits. In PT the provider evaluates the participants performance against pre-established criteria defined in the design of the PT scheme.

Performance evaluation

The majority of PT schemes involve some form of performance score, such as the z- or similar scores[1], and corresponding assessment criteria. An assigned value X and a standard deviation for proficiency assessment are determined and used for calculating the performance score of the laboratory result x_i, e.g. the z-score with $z = (x_i - X) / \sigma_p$

Assessment of z-scores is based on the following criteria:

- $|z\text{-score}| \leq 2.0$ is regarded as satisfactory;
- $2.0 < |z\text{-score}| < 3.0$ is regarded as questionable ('warning signal');
- $|z\text{-score}| \geq 3.0$ is regarded as unsatisfactory ('action signal').

This is based on the concept that normally distributed analytical results lie within two standard deviations with a probability of 95 %, and within three standard deviations with a probability of 99.7%.

PT providers have several options to determine σ_p such as prescribed/perceived desirable analytical performance or the observed distribution of data. The σ_p used by the PT provider may not be appropriate for all laboratories. If justified, the participants may then calculate their own z-score using an alternative σ_p-value which is fit for their purpose.

Corrective actions

Unsatisfactory performance scores ('action signal') indicate possible problems in the analysis undertaken. The laboratory must investigate this (e.g. by checking for transcription/calculation errors, trueness and precision) and, if necessary, address the problems through appropriate corrective actions. Participation in the PT provides very limited benefits to the laboratory, if unsatisfactory performance scores are not acted upon.

[1] For other scores refer to ISO 13528

Eurachem

Leaflets (L) & Guides

- Proficiency Testing Schemes (L)
- How can PT help my laboratory? (L)
- Pre & Post analytical PTs (L)
- Selection, Use & interpretation of PTs (2011)
- Quantifying Uncertainty (QUAM 2012)
- ...

Availability

- Download Croationa version
- Download Czech version
- Download English version
- Download Estonian version
- Download Italian version
- Download Serbian version
- Download Slovakian version
- Download Spanish version
- Download Swedish version
- Download Turkish version
- Download Ukrainian version

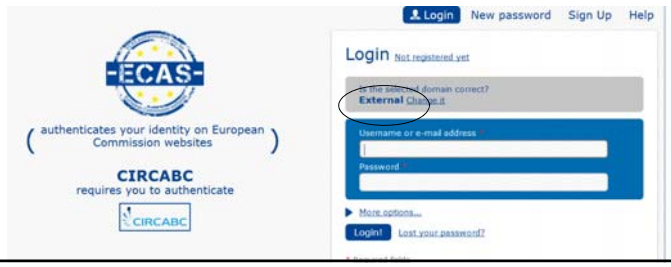
To which committees does your NRL participate *(related to HM)*

- *ISO, CEN,*
- *CODEX*
- *AOAC*
- *EFSA expert group*
- *Other?*

Knowledge Sharing
Document Exchange Platform

CIRCABC

https://circabc.europa.eu/

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EUROPA > European Commission > CIRCABC
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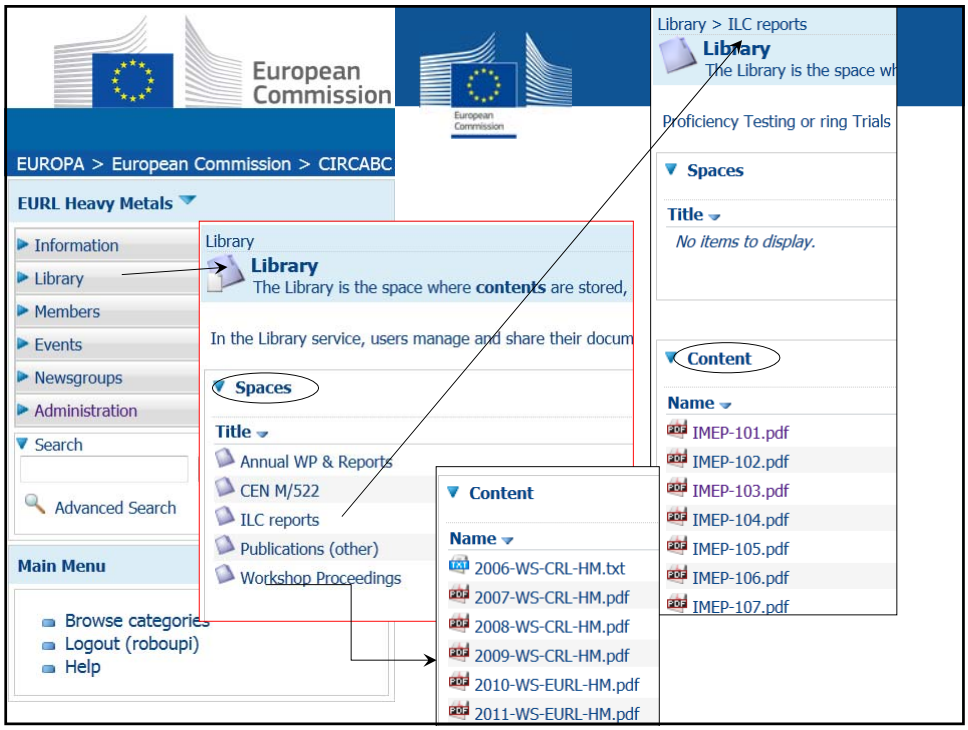
Others
List of the users categories and interest groups.
Category
User does not have any role category level
Category
Interest Group
Health and Consumers
Contaminants in food
Health and Consumers
Standing Committee on Plants, Animals, Food and Feed
Joint Research Centre
EEE-PT Working Group
Joint Research Centre
EURL for Feed Additives
Joint Research Centre
EURL for PAHs
Joint Research Centre
EURL Heavy Metals


European Commission
EUROPA > European Commission > CIRCABC
EURL Heavy Metals
Information
Library
Members
Events
Newsgroups
Administration
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Advanced Search
Main Menu
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Logout (roboupi)
Help
Library
Library
The Library is the space where contents are stored,
In the Library service, users manage and share their docum
Spaces
Title
Annual WP & Reports
CEN M/522
ILC reports
Publications (other)
Workshop Proceedings
Content
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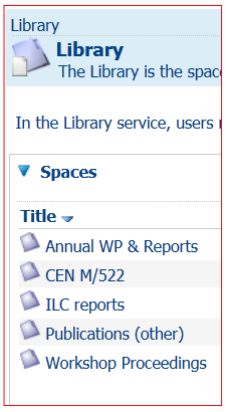
2006-WS-CRL-HM.txt
2007-WS-CRL-HM.pdf
2008-WS-CRL-HM.pdf
2009-WS-CRL-HM.pdf
2010-WS-EURL-HM.pdf
2011-WS-EURL-HM.pdf
Library > ILC reports
Library
The Library is the space wh
Proficiency Testing or ring Trials
Spaces
Title
No items to display.
Content
Name
IMEP-101.pdf
IMEP-102.pdf
IMEP-103.pdf
IMEP-104.pdf
IMEP-105.pdf
IMEP-106.pdf
IMEP-107.pdf

CIRCABC (continued)



- ✓ EURL PT reports
- ✓ WS proceedings
- ✓ Annual report & WPs
- ❑ NRL PT reports (?)
- ❑ NRL proceedings (?)
- ❑ NRL Annual Reports (?)
- ? which documents YOU wish share (no Copyrighted docs)

Under Library > Publications (other)

https://circabc.europa.eu/sd/a/b2a82372-8de6-4f71-b4b6-d163c175f943/**2014-NRL-performance-report%20(JRC90090)**.pdf

6. Statistical Experimental Design

Fernando Cordeiro

Statistical Experimental Design (SED)

How to make method development/validation more efficiently?

www.jrc.ec.europa.eu

F. Cordeiro
EC – JRC – IRMM, SFB Unit

Serving society
Stimulating innovation
Supporting legislation

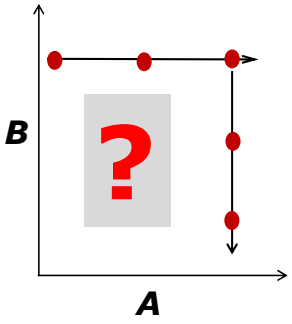
Method development / validation

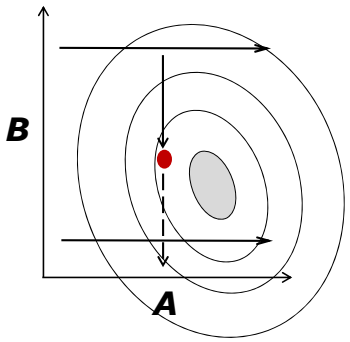
Finding the "best" experimental conditions ...

Where to start?

- ***Literature search,***
  Identical matrix/analyte combination,
- ***Previous experience,***
  Ask your boss!

Classical approach: changing one factor at a time *(keeping other factors constant)*





Effect of A depends on the level of B!

Classical approach (drawbacks)

- ***Too many experiments!***
- ***You do not investigate the complete experimental region!***
- ***You "ignore" interactions!***
- ***You may miss the real optimum!***
- ***You do not (really) plan your experiments!***

Statistical Experimental Design *(SED)*

Depending on your objective!

Plan a set of measurements to extract the maximum information with a minimum of experiments!

Whereby all influencing factors are *varied simultaneously*!

SED is used for (define your objective):

Screening

Which influencing factors (among many!) are having a significant effect on your response(s)?

Linear or linear with interaction models

Optimisation *(response surface modeling, RSM)*

Which are the optimum experimental conditions?

Quadratic + interaction → Central Composite Designs

Robustness

Are small, but controlled, variations of the experimental conditions affecting the response(s)?

Linear models

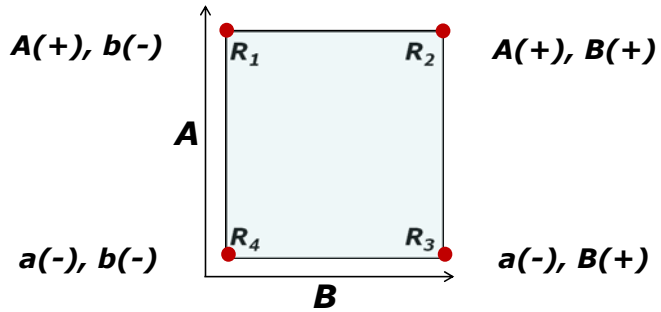
How? Planning your work!

- ***Define your objective,***
- ***Identify all influencing variables*** *(factors) –*
  knowledge about your measurement procedure required!,
- ***Set your experimental region*** *(low, high),*
- ***Select your response(s)*** *(e.g., peak area),*
- ***Select an appropriate design!*** *(minimum / reasonable N)*

Two factors *(k=2)* at 2 levels: *low -, high +*

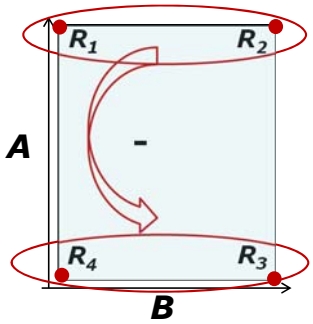
Experimental region is "fully" covered by $2^k = 4$ exp.!

(full factorial designs, FFD)



Main effects?

Effect of A (E_A) on the response, R *(peak area):*

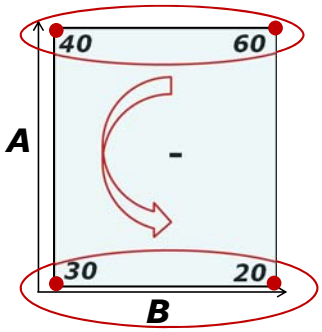


$$E_A = \frac{\sum A}{n_A} - \frac{\sum a}{n_a} = \frac{(R_1+R_2)-(R_3+R_4)}{2}$$

Main effects (E_A, E_B) on *R:*

$$E_A = \frac{(40+60)-(30+20)}{2} = \frac{100-50}{2} = 25$$

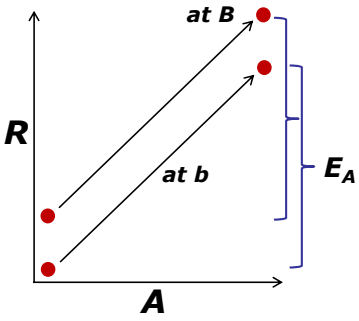
$$E_B = \frac{(R_2+R_3)-(R_1+R_4)}{2} = \frac{(20+60)-(30+40)}{2} = 5$$



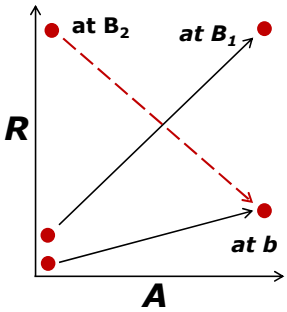
When changing factor A from low to high level R increases by 25!

Interaction effect (E_{AB})?

If E_A depends on which level B is set!



No interaction!

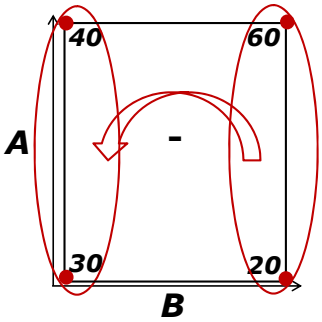


$E_A\ B > E_A\ b$

Significant interaction E_{AB}

Interaction effect?

$$E_{AB} = \frac{E_A\,(at\ B) - E_A\,(at\ b)}{2} = \frac{(R_2 - R_3) - (R_1 - R_4)}{2} = \frac{(60-20)-(40-30)}{2} = +\,15$$



Interaction effects (E_{AB}) may be larger than one of the main effects (E_A or E_B)!

Response Surface model equation

$$R = \beta_o + \beta_A A + \beta_B B + \beta_{AB} AB + \varepsilon$$

Randomise!

Exp.	β_o	A β_A	B β_B	AB β_{AB}	Response R
1	+	+	+	+	60
3	+	+	-	-	40
2	+	-	+	-	20
4	+	-	-	+	30

Model matrix

$\beta_o = (60+40+20+30)/4 = 37.5$

$\beta_A = (60+40-20-30)/4 = 12.5$

$\beta_B = (60-40+20-30)/4 = 2.5$

$\beta_{AB} = (60-40-20+30)/4 = 7.5$

$\beta_{AB} = 3\beta_B$

$\beta_{AB} < \beta_A$

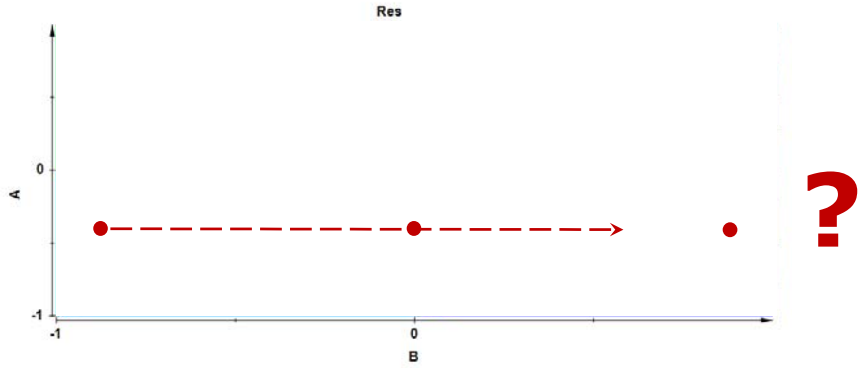
$$E_X = 2\beta_X$$

β_X estimated as the slope (from 0 to 1)

E_X estimated from -1 to 1 (low to high)

Use the response surface model equation

Find the real optimum!



Investigate the complete experimental region!

Doubts? Systematic but critical!

3 factors (A, B, C) at 2 levels (-, +) → (FFD, N=8) R to be max.!

Test a → A with b + B keeping c ***(N=4),***

Test a → A with b + B keeping C ***(+4),***

If higher R with A …

Test b → B with c + C keeping A ***(+4),***

If higher R with B …

Test c → C with A + B (n=2) → ***N=14!***

And how about c → C at a, b (n+2)?
and b → B with c, C at a (+4)? ***N=20!***

Randomise!

Exp.	A	B	C	β_0	A β_A	B β_B	C β_C	Resp. R
1	160	20	C_A	+	-	-	-	60
4	180	20	C_A	+	+	-	-	72
3	160	40	C_A	+	-	+	-	54
2	180	40	C_A	+	+	+	-	68
8	160	20	C_B	+	-	-	+	52
7	180	20	C_B	+	+	-	+	83
5	160	40	C_B	+	-	+	+	45
6	180	40	C_B	+	+	+	+	80
9	170	30	C_B	+	0	0	+	66
10	170	30	C_B	+	0	0	+	65
11	170	30	C_B	+	0	0	+	68
12	170	30	C_B	+	0	0	+	66

Model matrix

R_c

$$R = 64.25 + 11.50\,A - 2.50\,B \pm 0.75\,(C_B, C_A)$$

$R_c = 66.25,\ 1.26\ (S_c) \rightarrow \pm t.S_c/\sqrt{4}\ (\pm 2.0)$

$R_{model} = 64.25 + 0.75 = 65.0$ *(model validation I)*

Are the effects significant?

(model validation II)

$$F_{test} = \frac{E^2/(m-1)}{S_e^2/(n-1)}$$

$F_{test} > F_{crit}$ = significant

m = *number of measurements for the model,*
n = *number of measurements for the random variability,* S_e

E_A *(p < 0.001),* E_B *(p < 0.05),* E_C *not significant!*

Variability S_e under intermediate precision conditions:

$$S_e = \sqrt{\frac{S_A^2}{n_A} + \frac{S_a^2}{n_a}}$$

Or:

S_R *(from validation studies)*

$\sim E_{ABC}$ **or**

$\sim E_{dummy}$ *(hypothetical)*

ANOVA (multiple linear regression)

(model validation III)

- **Total Sum of Squares (SS),** T_{SS}
- **SS due to regression,** SS_{Reg} – Variance explained by the model
- **Residual SS,** SS_{Res} – Variance not explained by the model
  - SS due to the pure error (random), SS_{PE}
  - SS due to the lack of fit, SS_{LoF}

Balanced (*full factorial*) designs with *k* factors at 2 levels!

$N = 2^k$ $k = 2, N = 4 (+ S_e)$,

$k = 3, N = 8$,

$k = 4, N = 16$,

$k = 7, N = 128$!!

Reduce the number of experiments by selecting fractional factorial designs ($N = 2^{k-p}$)

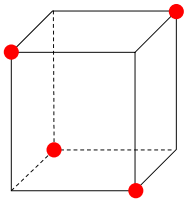
Fractional factorial designs (*k=4*)

Full factorial design (FFD at 2 levels) = 2^4, N = 16

Fractional factorial design: 2^{4-1}, N = 8 ! *(- 50 %)*

Randomise!

Exp	A	B	C	D	R	ABC
8	-	-	-	-	R_8	-
2	+	+	-	-	R_2	-
1	+	-	+	-	R_1	-
4	-	+	+	-	R_4	-
7	+	-	-	+	R_7	+
6	-	+	-	+	R_6	+
3	-	-	+	+	R_3	+
5	+	+	+	+	R_5	+

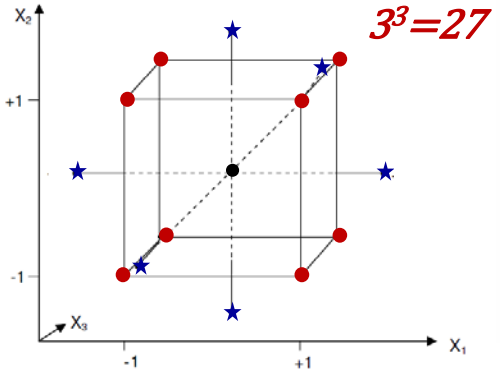


D = "ABC" (D is "confounded with" ABC = cannot be estimated separately)

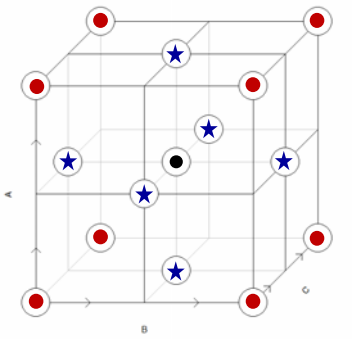
B = "ACD", C = "ABD" and A = "BCD" (resolution IV)

Central Composite (CC) Designs

(k=3, N = 14+3 → 8 FFD + 6 star (X^2)+ 3 center point!)



$3^3=27$



CC Face-centered (CCF)

Main + quadratic + interaction effects = optimisation!

How to do when having many factors?

- ***Identify all "potential" influencing factors*** *(k ~ 20!)*
- ***Run a two-level Screening Design to identify which ones have a significant effect on the response***
  (Pareto ~ 20 % factors → 80 % variability)
- ***Estimate the main effects*** *(interactions are not relevant)*
- ***Run a Optimisation (RS) Design using only significant factors*** *(k < 6)* *(narrow / refine the experimental region – use fractional factorial designs)*

Use the information from the screening design
C_A → C_B , β_{CB} = + 0.75 *(set at C_B)*

Questions?

Screening designs

How to find the really influencing factors (among many!)

Disc brake pads – *Objective: Screening*
10 influencing factors – Are all significant?

X_1	resin type	slow	fast
X_2	press type	old	new
X_3	press time	short	long
X_4	press pressure	low	high
X_5	press temperature	low	high
X_6	oven temperature	low	high
X_7	oven time	short	long
X_8	scorching time	short	long
X_9	scorching temperature	low	high
X_{10}	pressure at high temp.	low	high

R = compressibility *(to be minimized)*

Are all these 10 effects having a significant effect on the compressibility?

FFD (at 2 levels) requires …

2^{10} = 1024 experiments!

Plackett Burman designs

Plackett Burman designs are fractional factorial designs generated with 4, 8, 12 …, runs

Support only linear models (no interactions)

Number of experiments = the first multiple of 4 greater than the number of factors!

The first row … *(must be respected!)*

N = 8	+ - - + - + +	
N = 12	**+ + - + + + - - - + -**	***→ 11 factors***
N = 16	+ + + + - + - + + - - + - - -	

	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	D_1	R
1	+	+	-	+	+	+	-	-	-	+	-	163
2	-	+	+	-	+	+	+	-	-	-	+	121
3	+	-	+	+	-	+	+	+	-	-	-	152
4	-	+	-	+	+	-	+	+	+	-	-	100
5	-	-	+	-	+	+	-	+	+	+	-	93
6	-	-	-	+	-	+	+	-	+	+	+	173
7	+	-	-	-	+	-	+	+	-	+	+	133
8	+	+	-	-	-	+	-	+	+	-	+	131
9	+	+	+	-	-	-	+	-	+	+	-	157
10	-	+	+	+	-	-	-	+	-	+	+	157
11	+	-	+	+	+	-	-	-	+	-	+	101
12	-	-	-	-	-	-	-	-	-	-	-	236

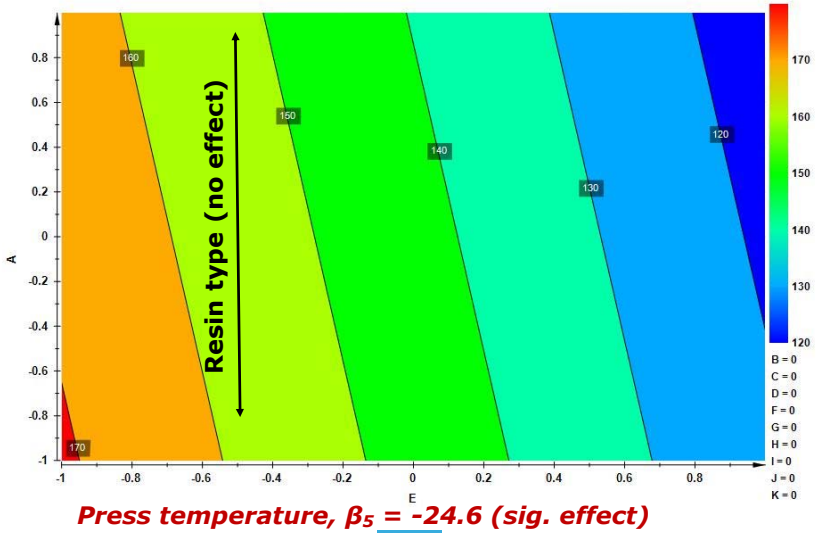
$R = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \ldots + \beta_D D_1$ *(Dummy = hypothetical)*

Only linear terms, no interactions!

$\beta_D = (121+173+133+131+157+101-163-152-100-93-157-236)/12 = -7.1$

Average Compressibility	$\beta_0 = 143.1$
resin type	$\beta_1 = -3.6$
press type	$\beta_2 = -4.9$
press time	$\beta_3 = -12.9$
press pressure	$\beta_4 = -2.1$
press temperature	$\beta_5 = -24.6$
oven temperature	$\beta_6 = -4.2$
oven time	$\beta_7 = -3.8$
scorching time	$\beta_8 = -15.4$
scorching temperature	$\beta_9 = -17.2$
pressure at high temperature	$\beta_{10} = +2.9$
Dummy	$\beta_D = -7.1$

Only effects X having $\beta_X > \beta_D$ are significant!



Press temperature, $\beta_5 = -24.6$ (sig. effect)

What to do next?

*New experimental design taking **ONLY** the significant factors*

Optimisation = response surface modeling

(use the information from the screening design)

Central Composite Designs …

Response Surface Modeling (RSM)

How to find (real) optimal experimental conditions?

Method Development

- ***With a reduced number of factors (k<6) one might not need to run a Screening design!***
- ***Run a Optimisation (RSM) Design***

If k is 2-3 a full factorial design can be feasible,

If k ~ 3-6 select fractional factorial designs –

Central Composite Designs

For optimisation (*Response Surface Modelling*) we need designs which are capable to estimate …

Main effects (E)+

interactions (E_{AB})+

quadratic terms (E_A^2)!

Solubility – *Objective: RS Modeling*

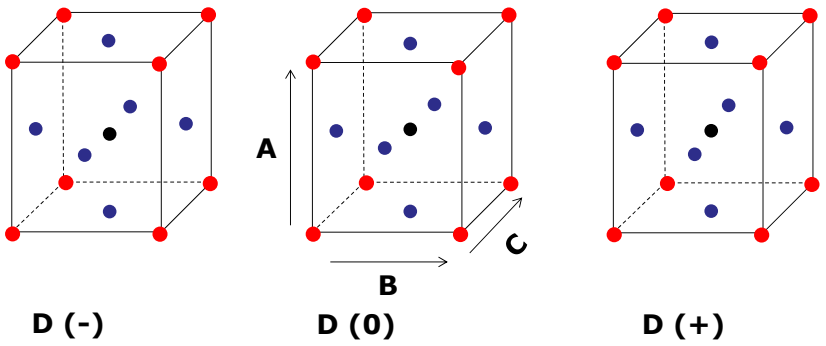
4 sig. factors – Find optimal experimental conditions!

EtOH	Et	Quantitative	Controlled 7 to 22
H2O	Wa	Quantitative	Controlled 0 to 0.5
Temp	Te	Quantitative	Controlled 5 to 25
H_3PO_4	Ac	Quantitative	Controlled 0 to 0.006

R = Solubility (µg mg^{-1})

→ Central Composite Face-Centered design

Model estimates main + (2-factor) interactions + quadratic effects



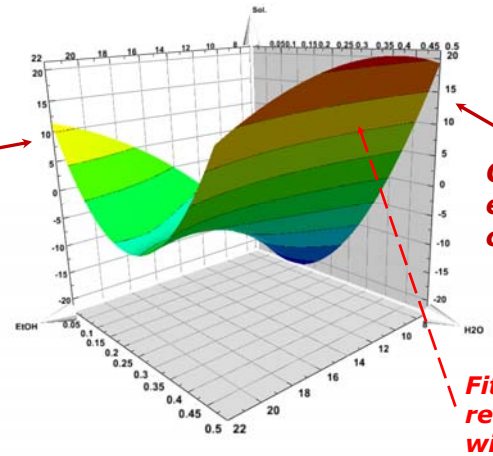
N = 8 + 6 + 1CP + (12) = 15 (+12) x 3 = 45 (or 75)

CCF selecting N = 24 + 3 CPs

(balanced design for 4 factors!)

Response surface

(saddle type)



Local optimum!

does it fit your requirements?

Optimal experimental conditions

Fits the requirements with less costs?

Robustness

Are minor changes in the factors (around the optimal conditions) affecting (significantly) the response(s)?

Robustness

All influencing variables –

quantitative *(time, concentration, …),*
qualitative *(e.g., analytical column),*
should be tested for ***robustness.***

The objective is to minimise the potential increase in the observed variability (from diff. laboratories) due to allowances in the SOP *(e.g., 6.5 ± 1.0)*

Appropriate designs for robustness assessment?

Objective is *(identical)* to screening:

Fractional factorial designs,

Plackett Burman designs

HPLC method – *Objective: Robustness*

8 factors – Are the selected ranges affecting R?

R = retention time + resolution (*critical pair*)

Make a balanced design at 2 levels (-, +)

Factor				Limits	Low	High	Nominal
A	Buffer pH	pH		± 0.3	6.5	7.1	6.8
B	Manufacturer	CO			All (-)	Pro (+)	All2
C	Column Temp.	TP	°C	± 0.5	23	33	28
D	% org solvent start	OS	%	± 1.0	24	26	25
E	% org. solvent end	OE	%	± 2.0	41	45	43
F	Flow	FL	ml min^{-1}	± 0.1	1.4	1.6	1.5
G	Wavelength	WL	nm	± 5	260	270	265
H	Buffer conc.	BC	% m v^{-1}	± 0.025	0.225	0.275	0.25
D1	Dummy 1	D1			-1	1	
D2	Dummy 2	D2			-1	1	
D3	Dummy 3	D3			-1	1	

Plackett Burman design (N=12)

8 + (3 D_i) factors

A) Follow Decision 2002/32/EC

$$D_A = A - a = \Sigma(A_i) - \Sigma(a_i) \qquad E_A = D_A/n_A$$

$$S_{Di} = \sqrt{2 * \sum(\frac{D^2_i}{n})}$$

If S_{Di} is significantly larger than the within-lab S_R

*"**<u>all factors together</u>** have an effect on the result **even if every single factor does not show a significant influence** and that the method is **<u>not sufficiently robust against the chosen modifications</u>**."*

Decision 2002/657/EC

B) Assessing each factor independently…

$$t_{test} = \frac{|E|}{S_R}$$

$t_{test} > t_{crit}$ = significant

t_{crit} **for 95 % CL and degrees of freedom for** S_R
(if a PB design is selected use S_{Dummy}*)*

Reduce the range for any effect which is responsible for the lack of method robustness, e.g., pH 6.8 ± 0.3!

A) Follow Decision 2002/657/EC

(calculate S_{Di} *and use a* F_{test} *to compare with* S_{dummy} ***or*** S_R*)*

$$F_{test} = \frac{S_{Di}{}^2/11}{S_{Dummy}{}^2/2} \qquad S_E = S_{Dummy} = \sqrt{\frac{\sum E_{Dummy}{}^2}{n_{Dummy}}}$$

N=12 and 3 D factors!
It should be compared with S_R

B) Use a t_{test} for each factor *(and for each response)*

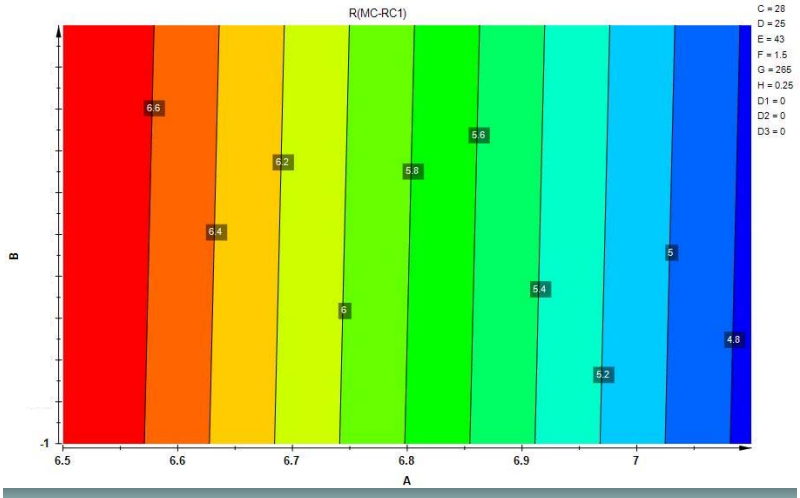
$$t_{test} = \frac{|E_i|}{S_E}$$

t_{crit} *for 95 % and* D_f *for estimating* E_D *(11)* ***or*** S_R

	R(MC-RC1)	Effect	S_E	t-test	t_c 95%	S_{Di}
A	pH	-2.12	0.539	-3.26	2.201	7.08
B	CO	0.03		0.05		
C	TP	0.78		-0.70		
D	OS	0.14		0.26		
E	OE	0.48		1.18		
F	FL	0.07		-0.80		
G	WL	0.17		1.20	F_{test}	F_{crit}
H	BC	-0.46		0.21	*31.4*	*19.405*
	D_1	-0.52				
	D_2	-0.10				
	D_3	0.77				

The method is <u>not</u> robust if allowing the pH to vary ± 0.3 around the optimal!

No effect for column manufacturer!



Significant effect if allowing pH to vary from 6.5 to 7.1!

What to do next?

Reduce the range for quantitative factors!

pH 6.8 ± 0.2 *(keeping all the other factors at their nominal value (optimal) → n=2*

Decide for qualitative factors!

How about a "classical" design?

With 8 factors to test for robustness one would require a minimum of N=16 exp.!

For each factor test at low (-) and high (+) level,
(keeping all the others at their optimal level)

With PB designs each effect is estimated with n=6 experiments at each level!

(precision improved by a factor of √6/√1 ~ x 2.4)

Suggestions ….

Identify properly all influencing factors (-, +),

Required knowledge about your measurement principle!

Identify and set your requirements,

Target values to be achieved

Select designs requiring the minimum number of experiments, ….

WHY?

You may fulfil your requirements!

save resources (low N)

Designs can be complemented!

… insert X^2 / interactions,

eliminate confounding effects

Have fun using SED!

7. HM in Feed & Food – CEN Activities

Beatriz de la Calle

HM in Feed & Food - CEN activities

M.B. de la Calle, I. Fiamegkos, F. Cordeiro, A. Cizek-Stroh and P. Robouch.

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CEN TC 275 / WG10 (Trace elements in food)

Since the last workshop of this network representatives of the EURL-HM attended the meetings of the working group in:

- Paris 18/10/2013
- London 23/05/2014

Validation of two methods on-going for the determination of:

- Inorganic arsenic in food matrices by HPLC-ICP-MS

Technical note for the determination of inorganic arsenic in rice by HG-AAS in of publication

- Methylmercury by GC-ID-ICPMS

The working group to decide before the next meeting in March 2015 if the method validated by the EURL-HM for the determination of methylmercury (IMEP-115) will be standardised.

- Revision of some existing standards on-going

CEN TC 327/WG4 (Trace elements in feed)

Since the last workshop of this network representatives of the EURL-HM attended the meeting of the working group in:

- Brussels 24/04/2014

Discussion on-going about:
- EN 15510:2007 (Trace elements by ICP-AES)
- EN 15550:2007 (Cd and Pb by GF-AAS after pressure digestion)

The work of this workshop is mentioned in Commission Regulation (EU) No 1275/2013

Pb & Kaolinitic Clay

COMMISSION REGULATION (EU) No 1275/2013

of 6 December 2013

amending Annex I to Directive 2002/32/EC of the European Parliament and of the Council as regards maximum levels for arsenic, cadmium, lead, nitrites, volatile mustard oil and harmful botanical impurities

(4) Recently, a significant difference has been identified by the European Union Reference Laboratory for heavy metals in feed and food (EURL-HM) between the analytical results obtained by the application of different extraction methods currently used for the determination of lead in kaolinitic clay and feed containing kaolinitic clay [2]. Before, no significant differences were observed between the levels of heavy metals in mineral feed by the application of different extraction methods [3]. The maximum levels of heavy metals in feed relate *'to an analytical determination of lead, whereby extraction is performed in nitric acid (5 % w/w) for 30 minutes at boiling temperature'*. It is therefore appropriate to provide for the use of that method of extraction for the determination of lead in kaolinitic clay.

[2] Determination of extractable and total lead in kaolinitic clay. Technical support from the EURL-HM to the Directorate-General for Health and Consumers – JRC 69122 – Joint Research Centre – Institute for Reference Materials and Measurements.

[3] IMEP-111: Total cadmium, lead, arsenic, mercury and copper and extractable cadmium and lead in mineral feed. Report of the eleventh interlaboratory comparison organised by the European Union Reference Laboratory for heavy metals in Feed and Food – EUR 24758 EN — Joint Research Centre – Institute for Reference Materials and Measurements.

CEN TC 327/WG4 (Trace elements in feed)

Mandate M522

Criteria approach for methods of analysis for heavy metals

Milestones

- Draft the document (Autumn 2014)
- Circulate it among NRLs for comments
  - Discussion of the results of the work within CEN/TC 327
  - Preparation of a draft Technical specification according to CEN rules
  - Evaluation of technical and editorial comments given during meetings and during the Enquiry stage of the draft technical specification
  - Evaluation of editorial comments given during meetings and during Formal Vote stage of the draft technical specification

8. Outcome IMEP-41

Ring-trial validation iAs in Food

Beatriz de la Calle

The outcome of IMEP-41 (validation of a method for the determination of iAs in food)

F. Cordeiro, I. Fiamegkos, P. Robouch, M.B. de la Calle.

European Commission, Joint Research Centre (JRC), Institute for Reference Materials and Measurements (IRMM), Retieseweg 111, 2440 Geel, Belgium.

How did the EURL-HM and IMEP get involved in the iAs issue?

A **scientific paper** was published by a research group at the **University of Aberdeen** questioning the safety of current levels of **iAs in rice** for the health of babies. The **European Parliament** asked the EC what actions would be given to address this problem.

IMEP-107: PT for iAs in rice

European Commission

IMEP-107

DG SANCO

- Is it possible to measure inorganic As in rice?
- How trustable are the date published in the literature?
- Can they be used for sound risk assessment?
- Is it feasible to set maximum levels for inorganic As in the legislation?

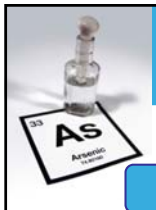
Accreditation: EA, APLAC

- A PT was organised:
- 6-7 expert laboratories in As speciation were asked to certify the test material.
- EA and APLAC were invited to appoint laboratories for participation in this exercise
- NRLs informed control laboratories in their respective countries abou the exercise

Standardisation:

- Information of the outcome of IMEP-107 will be provided to CEN TC 275/WG 10
- so that they can judge about what method to validate for the future standard

Joint Research Centre



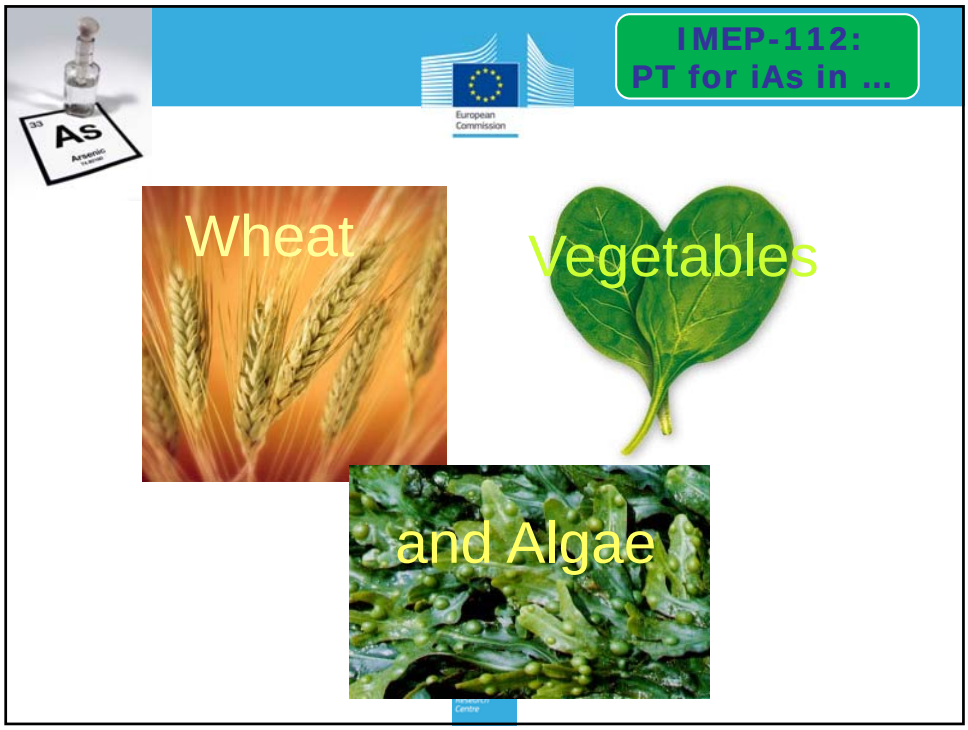
European Commission

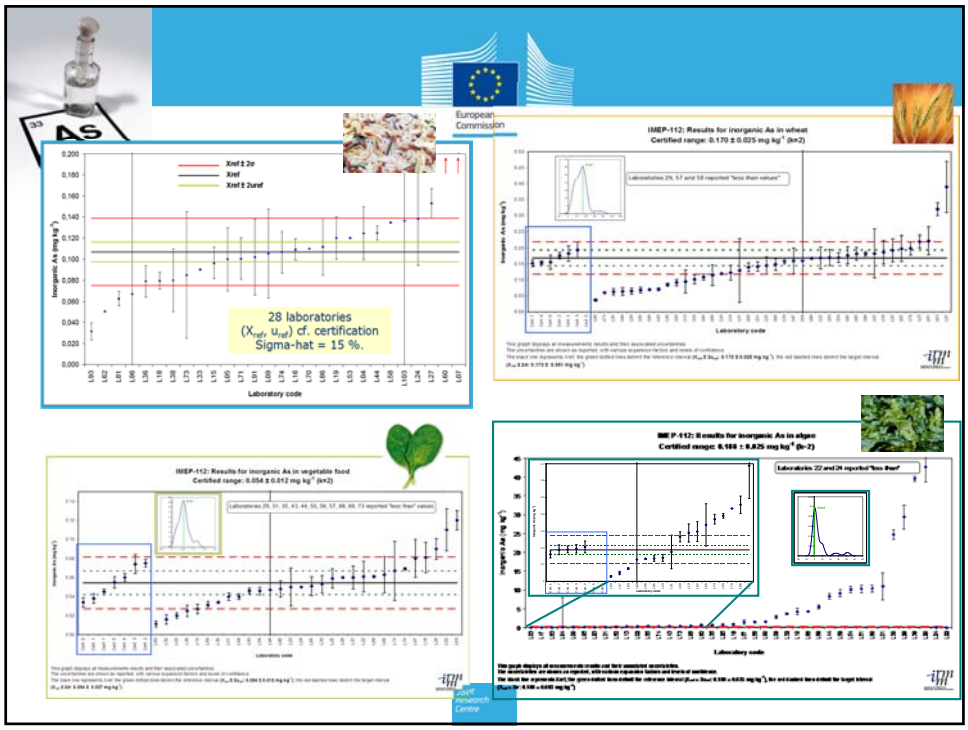
Main contributors to iAs exposure in Europe

- Cereal grains and cereal based products, other than rice, (approx. 50%)
- Food for special dietary uses
- Bottled water, coffee and bier
- Rice grains and rice based products
  (Implications for babies)
- Fish and vegetables

Scientific Opinion on As in food of the European Food Safety Authority (EFSA)
(2009) EFSA Journal 7(10):1351-1549

Joint Research Centre


IMEP-112:
PT for iAs in …
European Commission
Wheat
Vegetables
and Algae


European Commission
Xref ± 2σ
Xref
Xref ± 2uref
28 laboratories
(Xref, uref) cf. certification
Sigma-hat = 15 %.
Laboratory code
IMEP-112: Results for inorganic As in wheat
Certified range: 0.170 ± 0.025 mg kg-1 (k=2)
IMEP-112: Results for inorganic As in vegetable food
Certified range: 0.054 ± 0.012 mg kg-1 (k=2)
IMEP-112: Results for inorganic As in algae
Certified range: 0.188 ± 0.025 mg kg-1 (k=2)

Standardised methods

EN 15517(2008): iAs in seaweed by HG-AAS after acid extraction

"Nearly selective method":
(DMA and MMA may interfere)

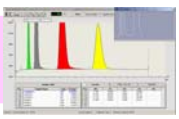
EN 16278(2012): iAs by HG-AAS after microwave extraction and SPE for feed. ***Validated in the range 0.190-2.7 mg kg^{-1}***

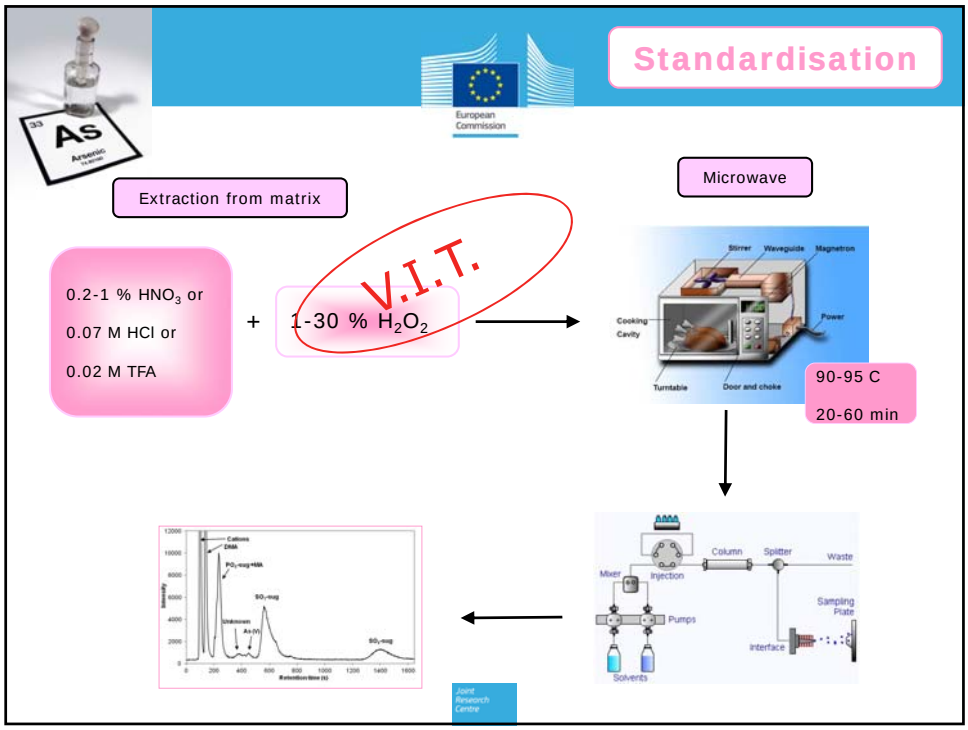
GB/T5009.11-2003: Determination of total arsenic and abio-arsenic in foods, issued by Ministry of Health, the Standardisation Administration of China

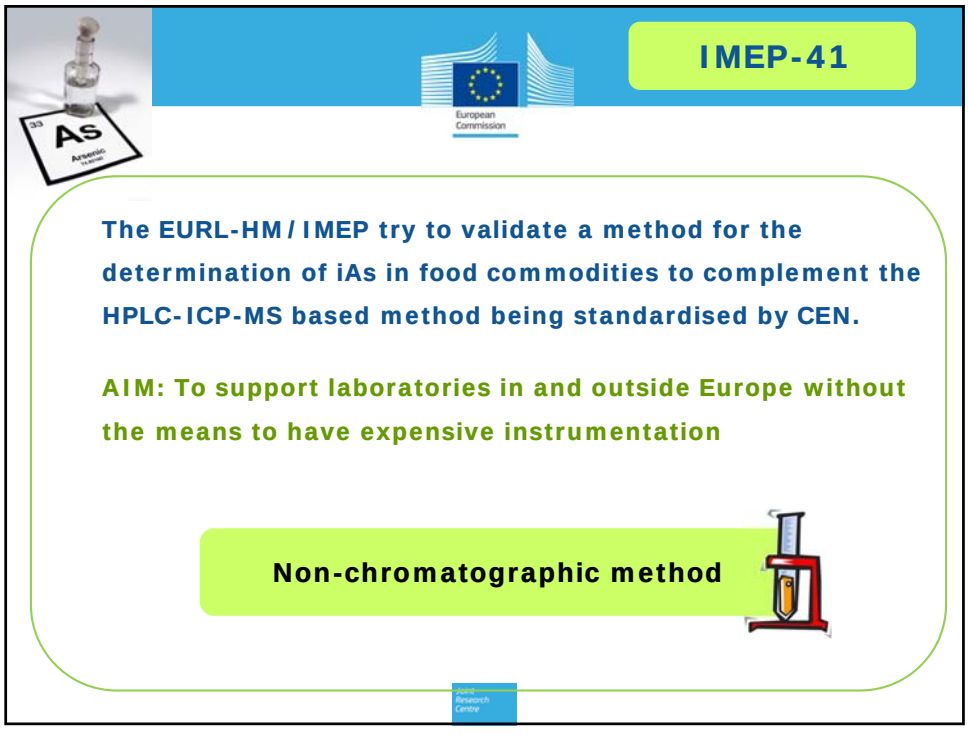
Standardisation

At present and under a mandate from the European Commission, **CEN TC 275 WG 10** *(Trace Elements and their Species in Food)* is in the process of standardising a method for the determination of **inorganic arsenic in food** commodities based on

HPLC-ICP-MS




Standardisation
Extraction from matrix
Microwave
0.2-1 % HNO3 or
0.07 M HCl or
0.02 M TFA
+
1-30 % H2O2
V.I.T.
90-95 C
20-60 min
Stirrer
Waveguide
Magnetron
Cooking Cavity
Power
Turntable
Door and choke
Column
Splitter
Waste
Mixer
Injection
Sampling Plate
Pumps
Interface
Solvents


IMEP-41
The EURL-HM / IMEP try to validate a method for the determination of iAs in food commodities to complement the HPLC-ICP-MS based method being standardised by CEN.
AIM: To support laboratories in and outside Europe without the means to have expensive instrumentation
Non-chromatographic method

IMEP-41

European Commission

CSIC

Non-chromatographic method for iAs determination

1st step. Hydrolisis

Polypropilen tubes (50 mL)

4.1 mL deionised water

18.4 mL HCl cc (9M)

Shake 1 h

0.5-1 g sample

Left overnight for a complete sample digestion

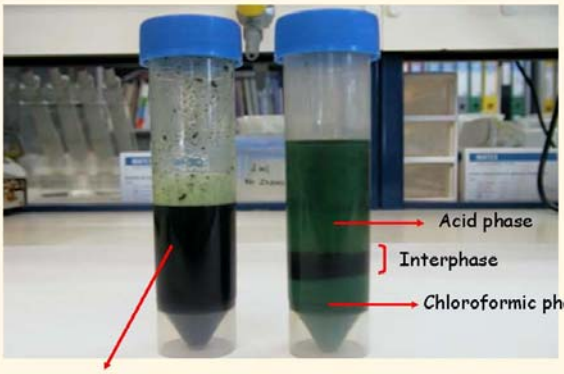
Courtesy of D. Velez & V. Devesa (IATA-CSIC)

Joint Research Centre

IMEP-41

European Commission

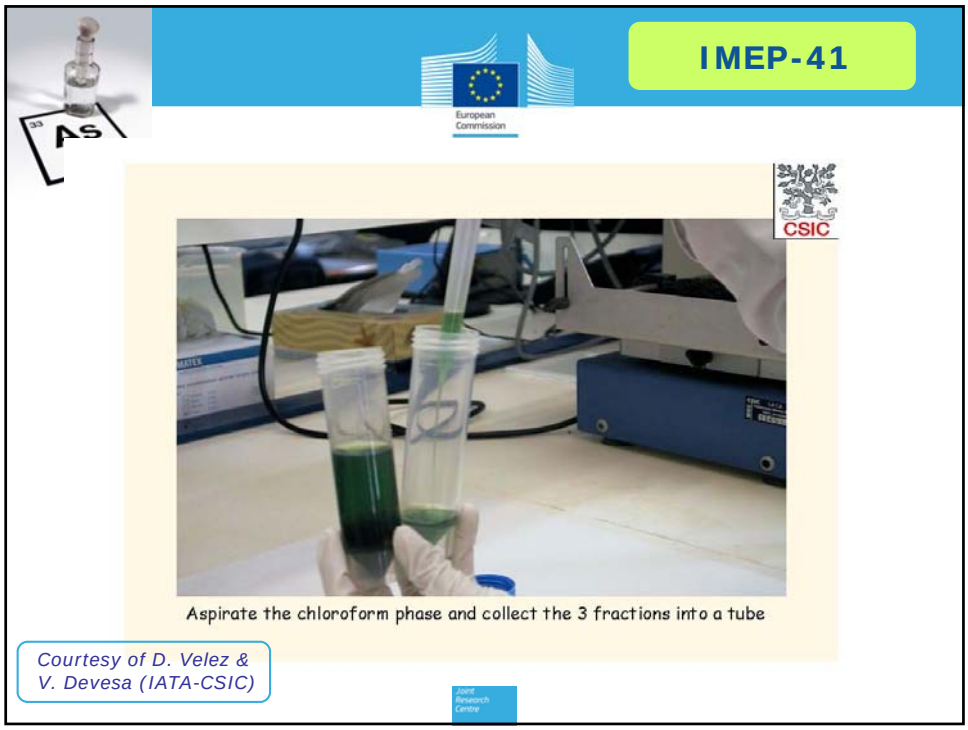
CSIC

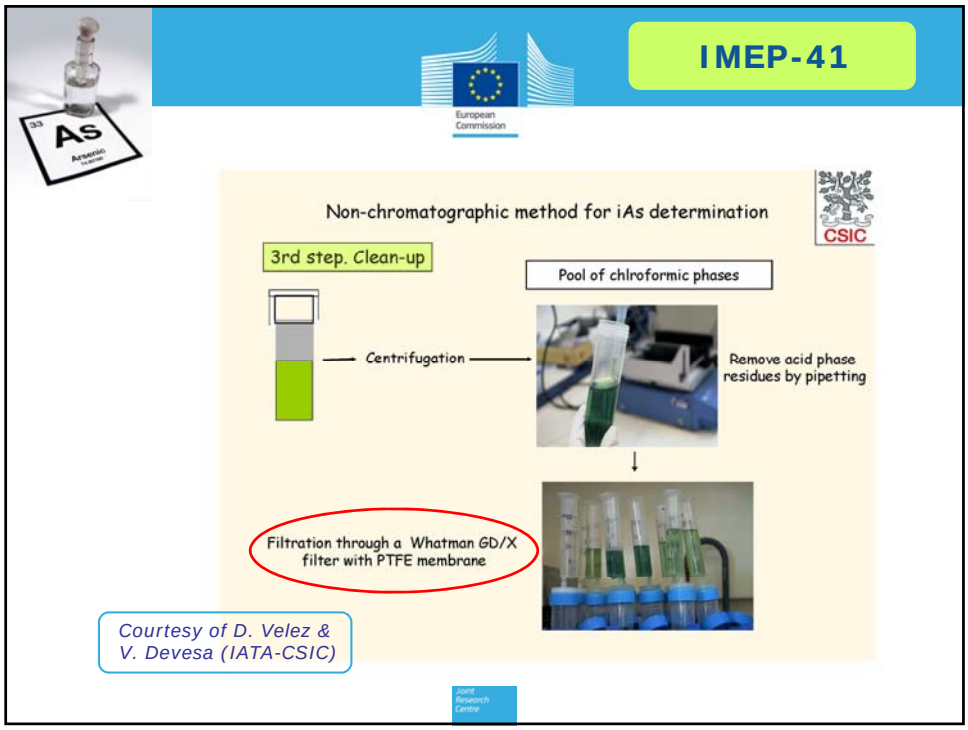


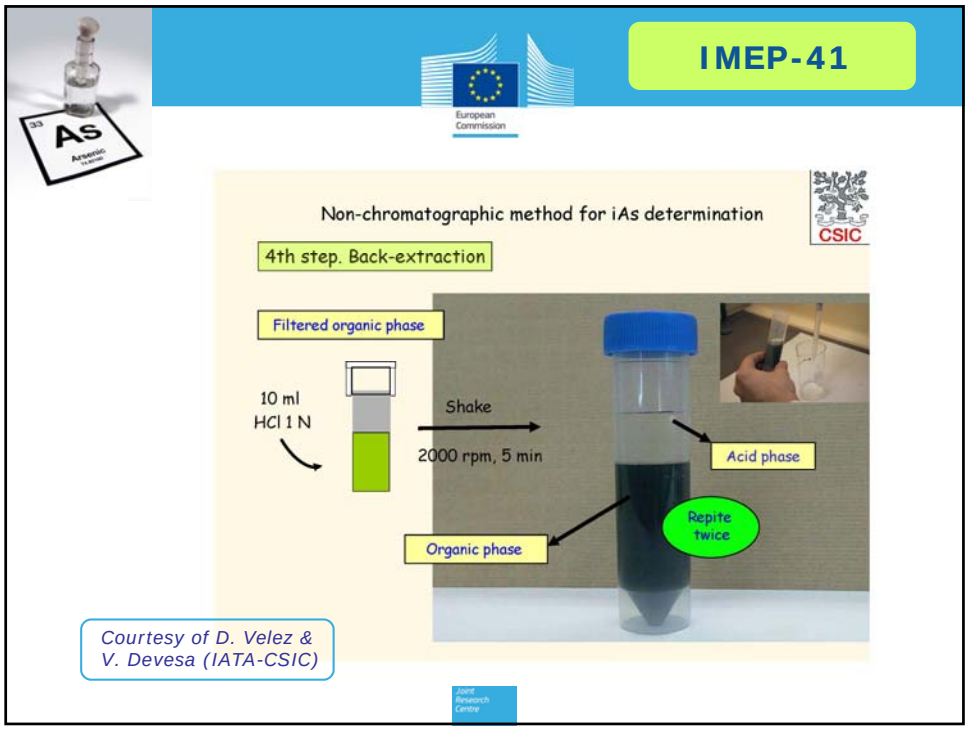
Hydrolised sample

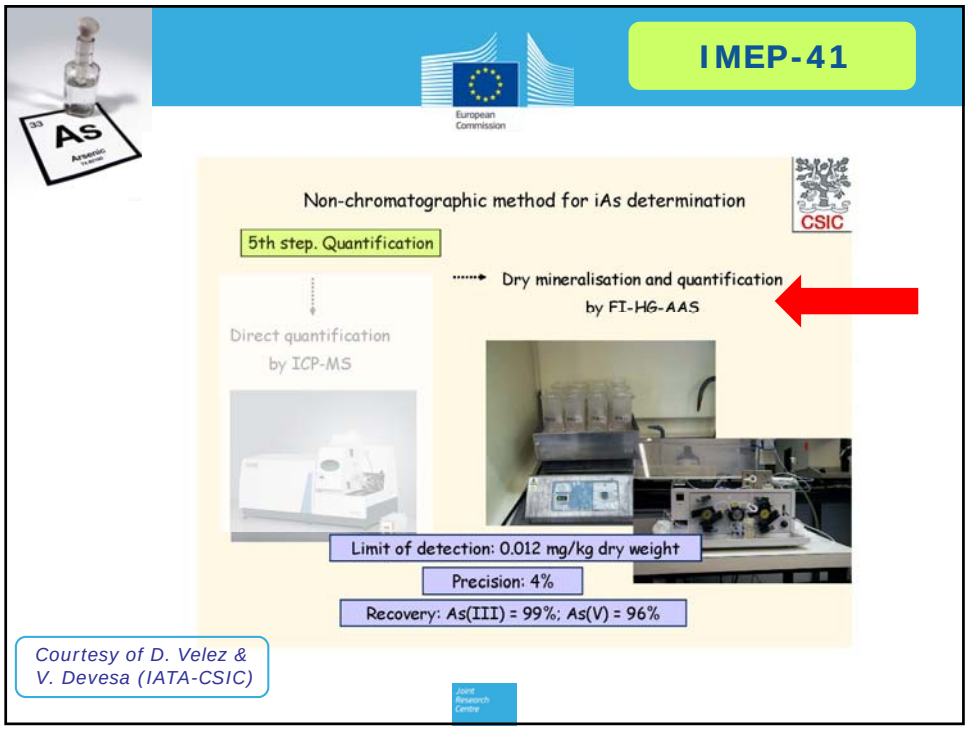
Courtesy of D. Velez & V. Devesa (IATA-CSIC)

Joint Research Centre


IMEP-41
European Commission
CSIC
Aspirate the chloroform phase and collect the 3 fractions into a tube
Courtesy of D. Velez & V. Devesa (IATA-CSIC)


IMEP-41
European Commission
CSIC
Non-chromatographic method for iAs determination
3rd step. Clean-up
Pool of chloroformic phases
Centrifugation
Remove acid phase residues by pipetting
Filtration through a Whatman GD/X filter with PTFE membrane
Courtesy of D. Velez & V. Devesa (IATA-CSIC)


IMEP-41
European Commission
Non-chromatographic method for iAs determination
CSIC
4th step. Back-extraction
Filtered organic phase
10 ml
HCl 1 N
Shake
2000 rpm, 5 min
Acid phase
Repite twice
Organic phase
Courtesy of D. Velez & V. Devesa (IATA-CSIC)


IMEP-41
European Commission
Non-chromatographic method for iAs determination
CSIC
5th step. Quantification
Dry mineralisation and quantification
by FI-HG-AAS
Direct quantification
by ICP-MS
Limit of detection: 0.012 mg/kg dry weight
Precision: 4%
Recovery: As(III) = 99%; As(V) = 96%
Courtesy of D. Velez & V. Devesa (IATA-CSIC)

IMEP-41

Expert laboratories which analysed the samples in parallel using methods of their choice based on HPLC-ICP-MS

- University of Aberdeen (UK)
- University of Barcelona (ES)
- Technical University of Denmark (DK)
- University of Graz (AT)
- Istituto Superiore di Sanita (IT)

IMEP-41

- **Seven samples included in the exercise covering:**

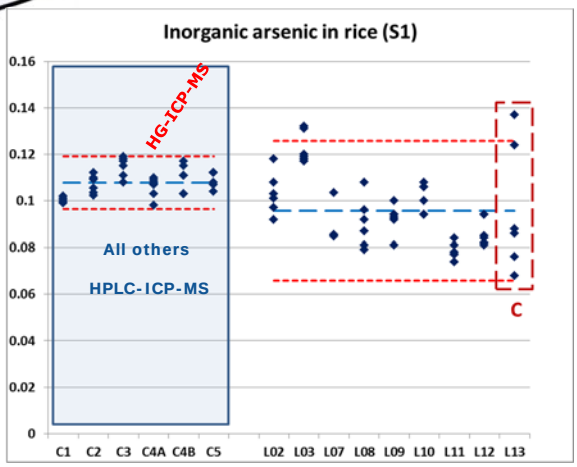
  Rice
  Wheat
  Mussels
  Cabbage
  Mushroom
  Seaweed
  Fish

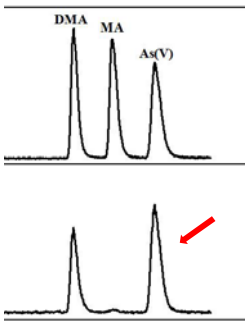
- **Twelve laboratories from nine European countries participated in IMEP-41 of which …**

IMEP-41

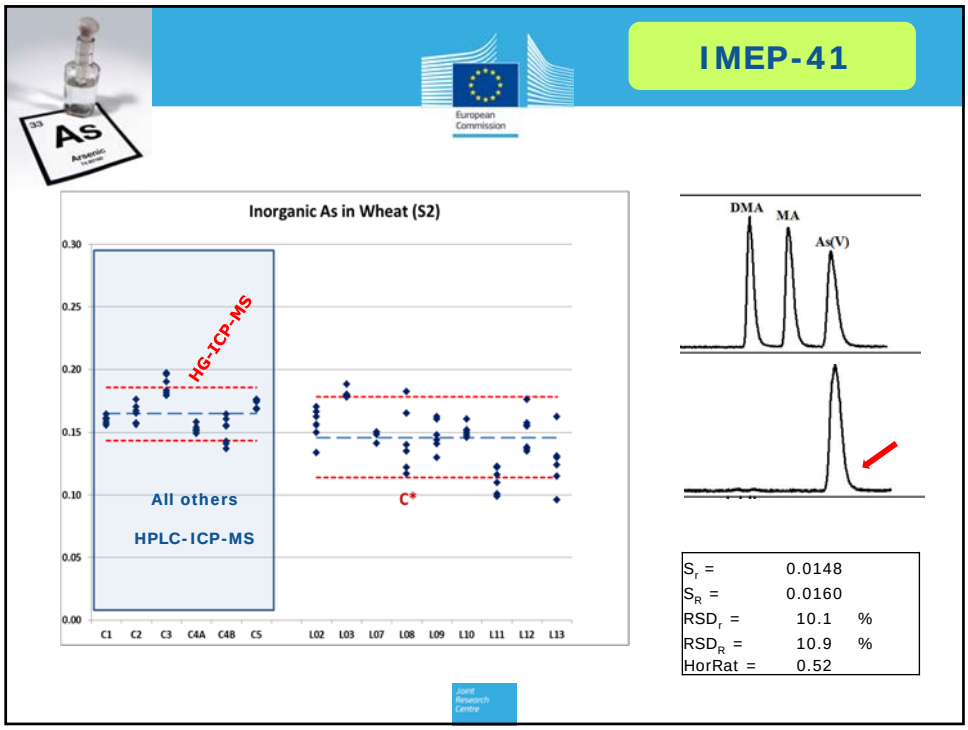
- **One laboratory did not use HG-AAS and was subsequently excluded from further calculations.**
- **One laboratory used an old HG-AAS apparatus not equipped with FI-HG-AAS** *(excluded from the study due to large variance in almost all sets of results)*
- **One laboratory did not use the method as described in the SOP** *(no filter used to separate the aqueous phase from the chloroform after the first extraction)*.

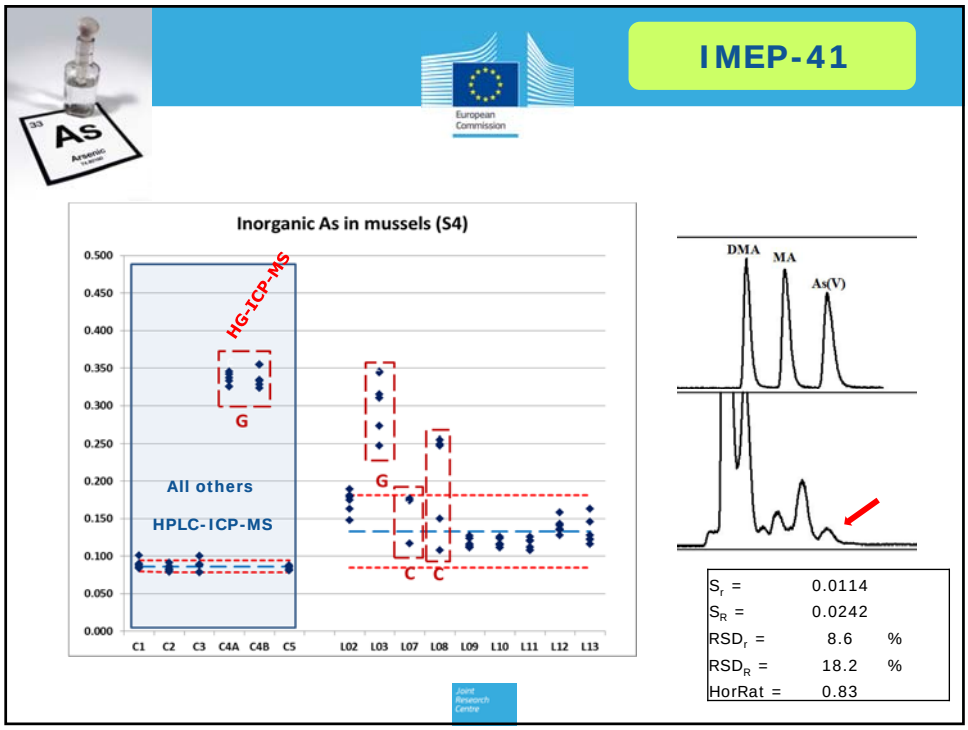
IMEP-41

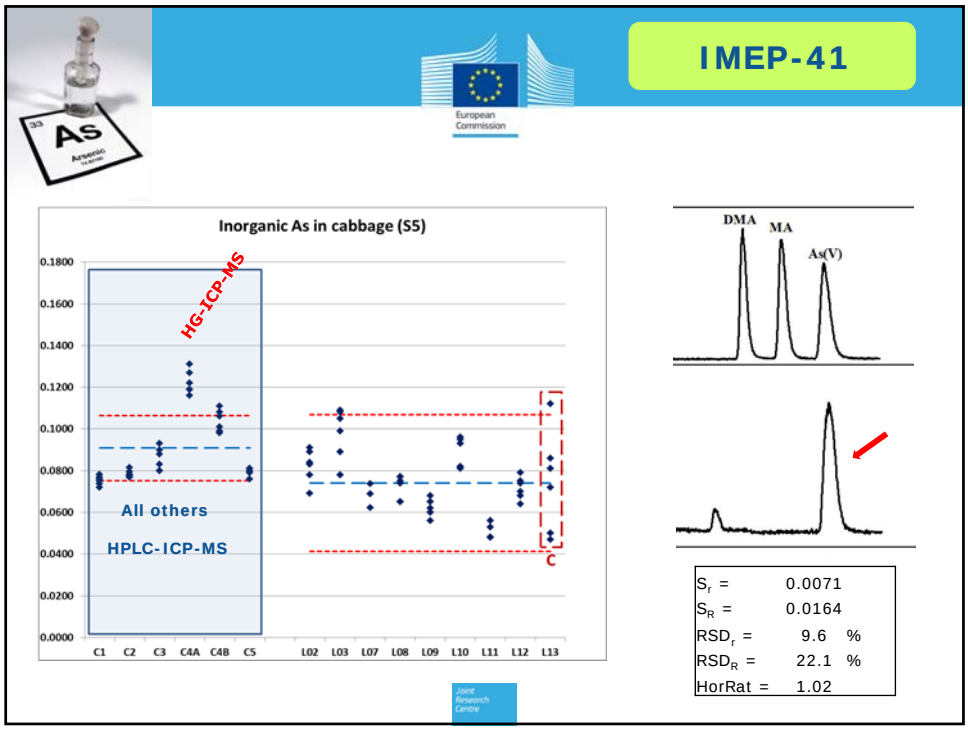


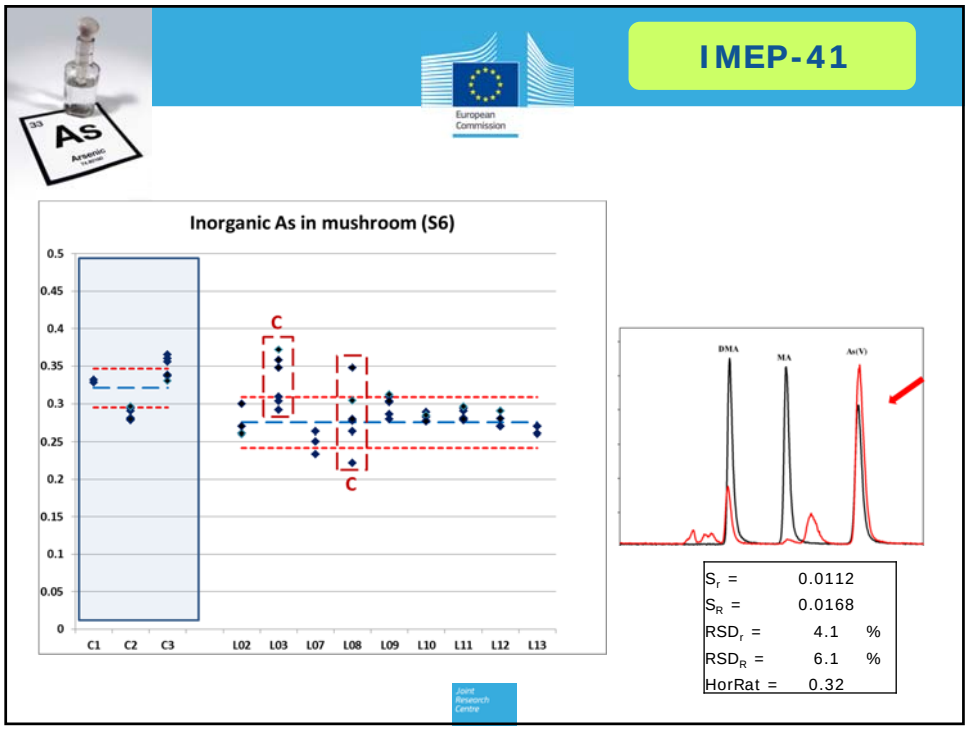


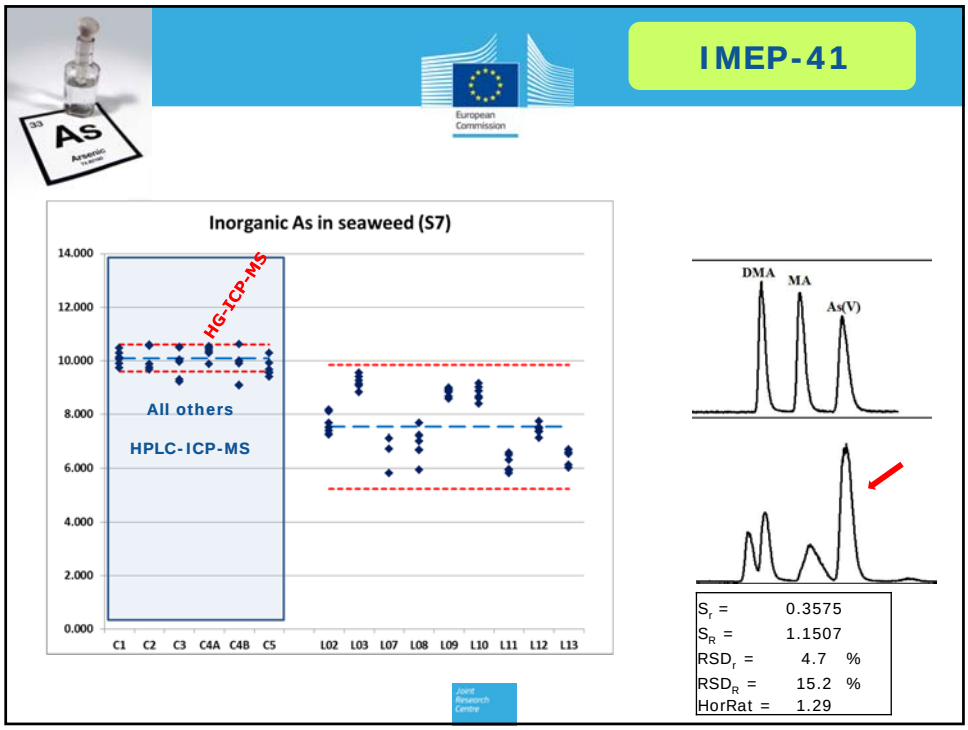
S_r =	0.0075	
S_R =	0.0150	
RSD_r =	7.8	%
RSD_R =	15.6	%
HorRat =	0.71	

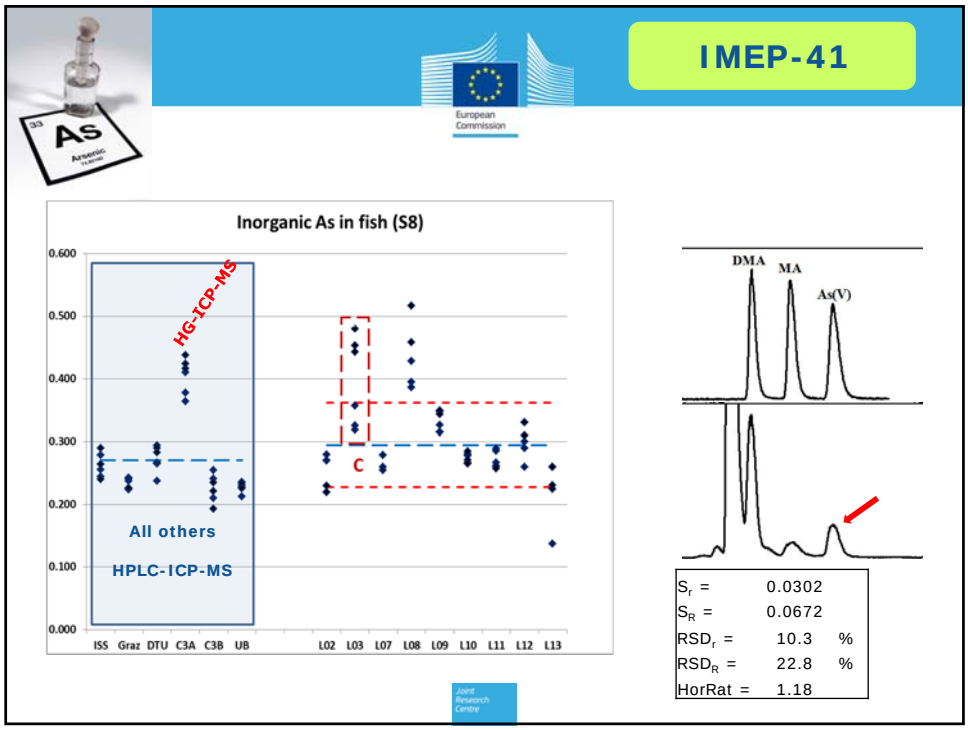

IMEP-41
European Commission
Inorganic As in Wheat (S2)
HG-ICP-MS
All others
HPLC-ICP-MS
C*
C1 C2 C3 C4A C4B C5 L02 L03 L07 L08 L09 L10 L11 L12 L13
DMA
MA
As(V)
S_r = 0.0148
S_R = 0.0160
RSD_r = 10.1 %
RSD_R = 10.9 %
HorRat = 0.52


IMEP-41
European Commission
Inorganic As in mussels (S4)
HG-ICP-MS
G
All others
HPLC-ICP-MS
G
C
C
C1 C2 C3 C4A C4B C5 L02 L03 L07 L08 L09 L10 L11 L12 L13
DMA
MA
As(V)
S_r = 0.0114
S_R = 0.0242
RSD_r = 8.6 %
RSD_R = 18.2 %
HorRat = 0.83


IMEP-41
European Commission
Inorganic As in cabbage (S5)
HG-ICP-MS
All others
HPLC-ICP-MS
C
C1 C2 C3 C4A C4B C5 L02 L03 L07 L08 L09 L10 L11 L12 L13
DMA
MA
As(V)
S_r = 0.0071
S_R = 0.0164
RSD_r = 9.6 %
RSD_R = 22.1 %
HorRat = 1.02


IMEP-41
European Commission
Inorganic As in mushroom (S6)
C
C
C1 C2 C3 L02 L03 L07 L08 L09 L10 L11 L12 L13
DMA
MA
As(V)
S_r = 0.0112
S_R = 0.0168
RSD_r = 4.1 %
RSD_R = 6.1 %
HorRat = 0.32


IMEP-41
European Commission
Inorganic As in seaweed (S7)
14.000
12.000
10.000
8.000
6.000
4.000
2.000
0.000
HG-ICP-MS
All others
HPLC-ICP-MS
C1 C2 C3 C4A C4B C5
L02 L03 L07 L08 L09 L10 L11 L12 L13
DMA
MA
As(V)
Sr = 0.3575
SR = 1.1507
RSDr = 4.7 %
RSDR = 15.2 %
HorRat = 1.29


IMEP-41
European Commission
Inorganic As in fish (S8)
0.600
0.500
0.400
0.300
0.200
0.100
0.000
HG-ICP-MS
All others
HPLC-ICP-MS
C
ISS Graz DTU C3A C3B UB
L02 L03 L07 L08 L09 L10 L11 L12 L13
DMA
MA
As(V)
Sr = 0.0302
SR = 0.0672
RSDr = 10.3 %
RSDR = 22.8 %
HorRat = 1.18

IMEP-41

Drawbacks

- Tedious
- It implies the use of chloroform
- Interference of MMA
(Not abundant in most matrices)

IMEP-41

Advantages

- Low cost instrumentation
- Pre-treatment uses basic analytical chemistry
- The method is **robust** and can be used in **complex matrices**, even with **low concentration** of iAs, without adjustment

Some comments we got from you about the method:

We do not recommend this method for routine analysis because is very time consuming, quite complicated and because of the chemicals used.

We would like to accredit the method and use it in our lab.

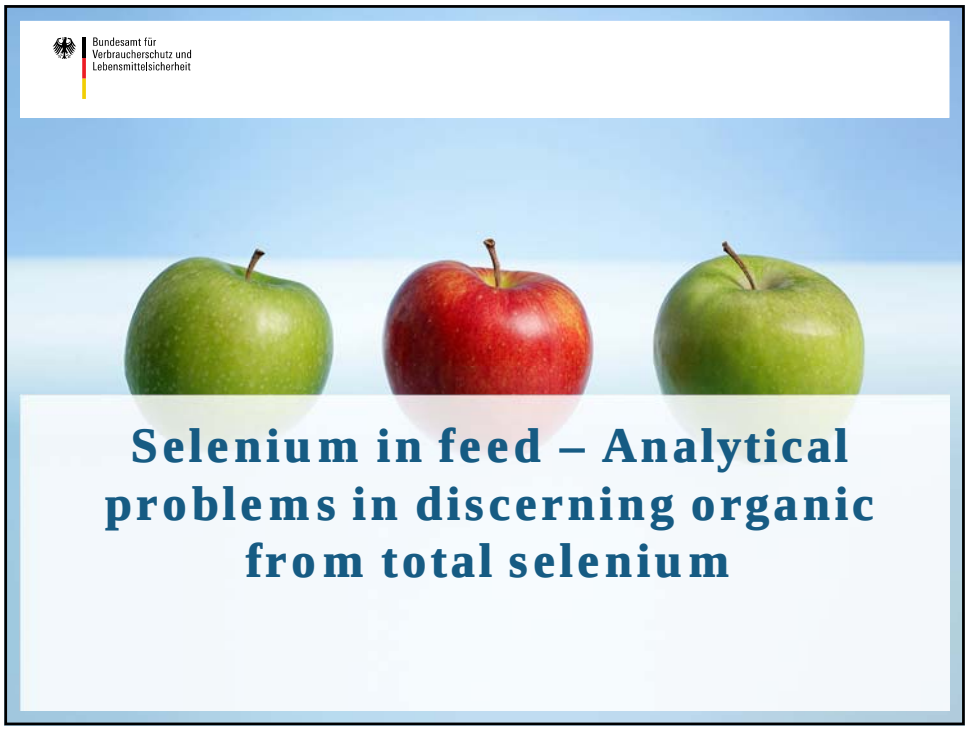
IMEP-41

Conclusions

- Method **successfully validated** with **Horrat values < 1.5** for all the tested matrices. The SOP will be downloadable from the EURL-HM web page.
- Since the different matrices were analysed using other techniques, quite a lot of information can be gathered on the analytical determination of inorganic arsenic in food commodities.
- Report expected in October 2014.

9. Selenium in feed
Analytical problems in discriminating organic from total Selenium

Timo Kapp (BvL)



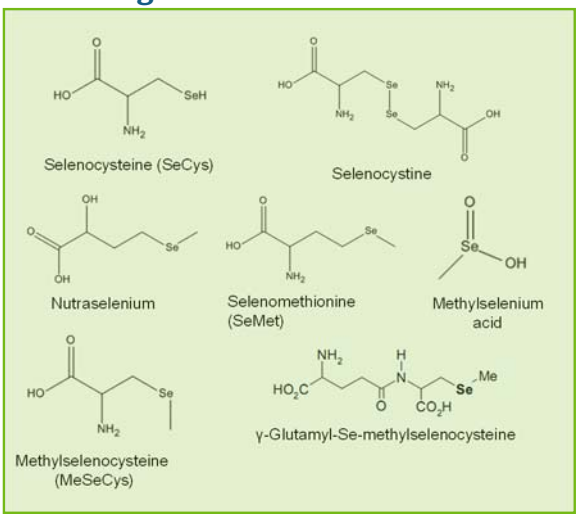
Bundesamt für
Verbraucherschutz und
Lebensmittelsicherheit

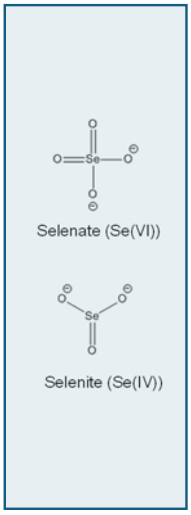
background - general

- Selenium is an essential element for human and animal
- Sources for selenium: fish, meat, eggs, legume, nuts, mushrooms, etc
- Especially in animal production selenium is often supplemented conventionally with inorganic forms of selenium
- In recent years organic selenium sources (selenomethionine, selenium yeast etc.) were introduced
- EFSA raised concerns about higher carry over rates caused by the organic selenium forms. This may exceed the safe exposition of some consumer populations, as selenium has got a small therapeutic width.
- Therefore the supplementation of organic selenium forms is limited to 0.2mg/kg feeding stuff (Regulation 427/2013)

Bundesamt für Verbraucherschutz und Lebensmittelsicherheit

Background – some selenium species





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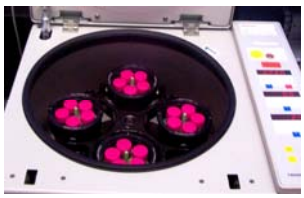
Analytical approach



Predigest with Amylase

Protein digest with Subtilisin

Protein digest with Prot. Asp. Oryzae

Ultrafiltration

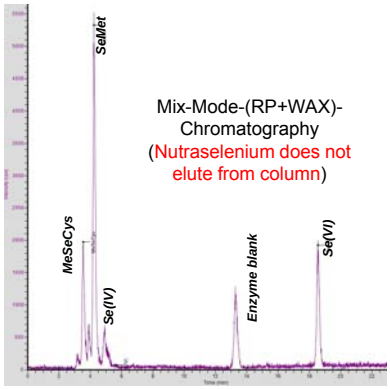
LC-ICP-MS (RP + WAX)

LC-ICP-MS (IP)

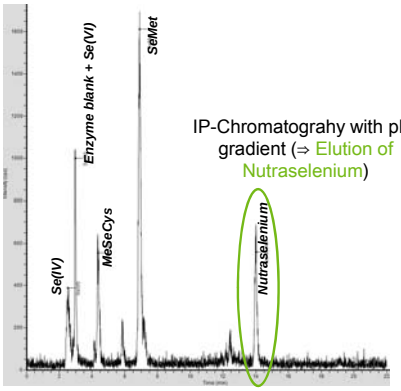
Bundesamt für Verbraucherschutz und Lebensmittelsicherheit

Examples of chromatograms

Lentils spiked



Mix-Mode-(RP+WAX)-Chromatography (Nutraselenium does not elute from column)



IP-Chromatograhy with pH-gradient (⇒ Elution of Nutraselenium)

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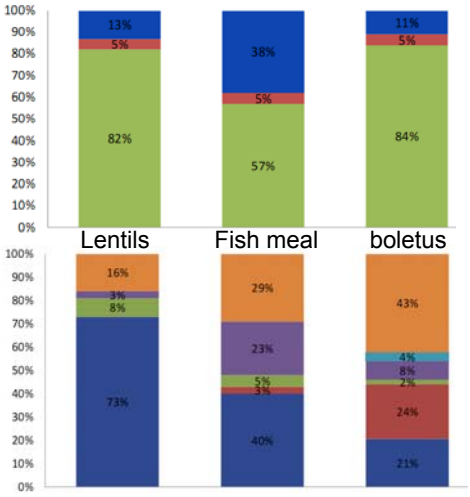
Results with "real" samples

extraction efficiency

- insoluble residue
- selenium in supernatant
- selenium in ultrafiltrate

species composition

- unknown
- Methylselenium acid
- Methylseleniumcysteine
- Selenate (Se(VI)
- Selenite (Se(IV))
- Selenomethionine



Bundesamt für Verbraucherschutz und Lebensmittelsicherheit

Characteristics of the method

Substanz	LOQ [mg Se/kg]	Recovery	Precision
Selenomethionine	0,061	70-110% (spiked) 104±10% (nat)	6% at 2,3mg/kg 5% at 1,6mg/kg 6% at4,5mg/kg
Methylselenocysteine	0,033	90-110%	4% at 1,0mg/kg
Selenate	0,021	80-100%	11% at 95µg/kg
Nutraselenium	n.b.	90-110%	9% for 0,9mg/kg spiked
Selenite	0,117	10% bis max. 50%	40% at 95µg/kg 10% at 3,2mg/kg
Methylselenium acid	0,094	ca. 50%	9% für 0,9mg/kg spiked

Bundesamt für Verbraucherschutz und Lebensmittelsicherheit

Problem reference material

- **one certified reference material available: NRC-SELM-1**

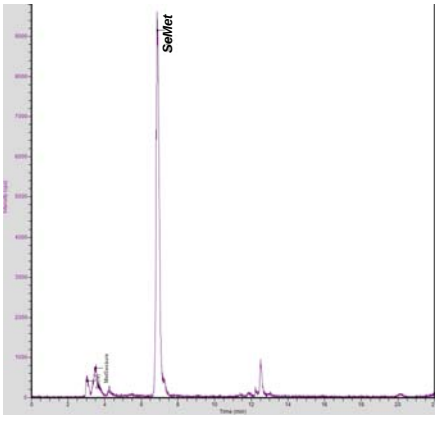
National Research Council Canada | Conseil national de recherches Canada

SELM-1

Selenium Enriched Yeast Certified Reference Material

CERTIFIED VALUES (milligram/kilogram)

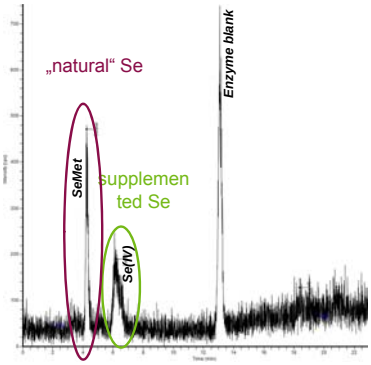
Total Selenium	2059	± 64
Selenomethionine	3448	± 146
Methionine	5758	± 277



Bundesamt für Verbraucherschutz und Lebensmittelsicherheit

Example complex feed (1)

- **Feed for dairy cows (Rapeseed, soya, bean supplemented with selenite 0,3mg Se/kg) (total-Se 0,55mg/kg)**

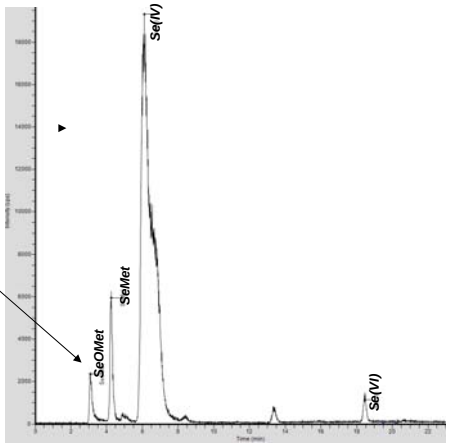


Bundesamt für Verbraucherschutz und Lebensmittelsicherheit

Example complex feed(2)

- **Mineral feed (supplemented with 30mg Se/kg selenite and org. Se 10mg Se/kg from selenium yeast)**

Large portion of the supplemented SeMet oxidized to SeOMet ⇒ lowers the bioavailability!



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Summary and questions

- Authorized additives were well quantifiable
- The availability of standard substances impedes the analysis

⇒ The sums of organic selenium is not directly determinable

? How should the producer control and the official labaratories the content of organic selenium with no official method available?

? What to do with natural organic selenium contents above 0.2mg/kg?

? How to discriminate between added and natural organic selenium?

Bundesamt für Verbraucherschutz und Lebensmittelsicherheit

Thank yor for your attention!

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10. Review of NRL performances

Piotr Robouch

Implementation of Art.33 of Regulation (EC) No 882/2004 in the EU Member States in the area of Heavy Metals in Food and Feed

P. Robouch , I. Fiamegkos, F. Cordeiro, B. de la Calle

Report JRC90090

Executive Summary

DG SANCO request to provide
(i) an overview of MS which did not **appoint NRLs**;
(ii) a review of NRL activities performed in the frame of their mandate, including the **organisation of PT** and the **follow-up** of non-compliant results reported by OCL.

The EURL-HM network consists of **47 NRLs**.
All countries attend systematically the annual workshops and **participate to PTs** organised by the EURL-HM since 2007.

The thorough review of NRL performances for the determination of total mass fraction of **As, Cd, Hg and Pb in food and feed** matrices clearly demonstrates the **high quality of the analytical capabilities of the network laboratories**.
Some **challenging matrices** were identified and will be closely monitored.

Finally, all **NRLs declared fulfilling their mandate** set by Regulation (EC) 882/2004, Articles 33.2, but only fourteen of them (managing a network of several national official control laboratories) organise and follow-up non-compliant results on a yearly basis.

Highlights (1 / 6)

The updated list of NRLs presented in Table 1 shows that:

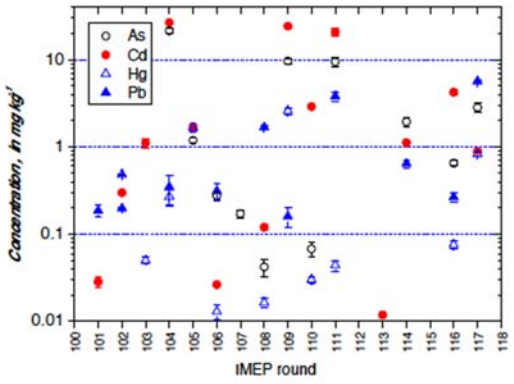
- 12 countries nominated one NRL to cover the three matrices (food + feed + fish);
- 16 countries nominated two NRLs (food & fish ; feed),
- 1 country (Greece) nominated three NRLs.

- 29 NRLs participate also to the EURL-CEFAO network, while 18 NRLs (mainly related to feed) don't.

Table 2 presents the overview of attendance for the various countries since 2009. In general, excluding few last minute cancelations, all member states systematically attend the EURL-HM workshops.

Highlights (2 / 6)

Figure 1: Concentrations ranges investigated in the frame of the 17 PTs organised by the EURL-HM



Highlights (3 / 6)

Food matrices: Determination of Cd, Hg & Pb in food matrices well under control. Only few problems identified for low concentrations (Hg in IMEP-116; Pb in IMEP-109).

Feed matrices: Most NRLs provide satisfactory results for Cd.
Determination of Hg and Pb challenging in mineral feed and feed premix matrices containing high contents of these elements.

Arsenic: Maximum levels for As set only for feed. Most of the NRLs implement(ed) validated methods for determination of As in food matrices. Positive improvement in performances evidenced for the two sets of matrices. Remaining non-compliances occurred for low As concentrations (ca. 0.05 mg kg-1) in spinach leaves (IMEP-110) and feed of plant origin (IMEP-108).

Inorganic arsenic: NRLs were requested by SANCO to determine iAs in IMEP-107 (rice); IMEP-109 (seafood) and IMEP-112 (wheat, spinach and seaweed) and IMEP-116 (mushrooms). Since most of the NRLs did not have an extensive experience in the field no thorough evaluation is included in this report. However, a significant increase in the number of NRLs reporting results for iAs was observed along the years: from 10 in IMEP-107 to 16 in IMEP-116, of which 13 were evaluated as "satisfactory".

Highlights (4 / 6)

REGULATION (EC) No 882/2004 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL

[...]

Article 33: National reference laboratories

1. Member States shall arrange for the designation of one or more national reference laboratories for each Community reference laboratory referred to in Article 32. A Member State may designate a laboratory situated in another Member State or European Free Trade Association (EFTA) Member and a single laboratory may be the national reference laboratory for more than one Member State.

2. These national reference laboratories shall:
   (a) collaborate with the Community reference laboratory in their area of competence;
   (b) coordinate, for their area of competence, the activities of official laboratories responsible for the analysis of samples in accordance with Article 11;
   (c) where appropriate, 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 Community reference laboratory supplies;
   (e) provide scientific and technical assistance to the competent authority for the implementation of coordinated control plans adopted in accordance with Article 53;
   (f) be responsible for carrying out other specific duties provided for in accordance with the procedure referred to in Article 62(3), without prejudice to existing additional national duties.

[...]

Highlights (5 / 6)

As expected, **100 % of the NRLs** having replied:

(i) Coordinate, when relevant, the activities of their national official control laboratory, OCLs (cf. Art.33.2(b));

(ii) Disseminate systematically all pertinent information from the Commission, from the EURL and/or from the CEN committees to the relevant authorities (cf. Art.33.2(d)); and

(iii) Provide technical & scientific assistance to competent authorities when needed (cf. Art.33.2(e)).

As for the organisation of PTs (cf. Art.33.2(c_1)) and the proper follow-up of non-compliant results (Art.33.2.(c_2)), the following types of replies were obtained (cf. Table 1 and Annex 6):

a) **28** NRLs **do not organise** any PT, since no (or not enough) OCLs exist in their country for the specific field. No follow-up is then required.

b) **4** NRLs (from DK, HU, SE and UK) **outsource** this activity to other PT providers, such as FAPAS, BEPEA or IMEP/EURL-HM, and monitor systematically non-compliant performances of their OCLs.

c) **15** NRLs (from BE, CZ, DE, DK, ES, FR, HU, IT, PL, RO) organise at least one PT per year for the determination of heavy metals in food or feed. The OCL performances are evaluated and discussed in the frame of dedicated workshops. Moreover, non-compliant performances are systematically followed-up.

Highlights (6 / 6)

EURL-HM, Geel - Questionnaire

Dec 2013 – Apr 2014

Contents

Based on the evidence presented, the performance of the network of NRLs for heavy metals in food and feed is **very satisfactory**.

SANCO new WGs

I. *Development & Evaluation of **NRL** workshop and training programmes*

II. *Development of performance monitoring and **peer review** schemes (for EURLs)*

III. *Development **best practices** (for EURLs,NRLs)*

DG SANCO Meeting with Directors of the EURL in food, feed and animal health Sectors
Brussels, July 4, 2014.

Update NRL info

- ✓ *Effective communication with NRLs required*
- ✓ *Up-to-date list of Members*
- ✓ *Up-to-date contact info (email, address, affiliation)*

- ➢ *Exchange of info through CIRCABC*

Should we go social?

11. IMEP-118

HM in canned peas

Ioannis Fiamegkos

IMEP-118: Determination of total As, Cd, Pb, Hg, Sn and inorganic As in canned food

I. Fiamegkos, B. de la Calle, F. Cordeiro, P. Robouch

European Commission, Directorate General Joint Research Centre, Institute for Reference Materials and Measurements, B-2440 Geel, Belgium

9 September 2014

IMEP-118: Determination of total As, Cd, Pb, Hg, Sn and inorganic As in canned food

Scope and aim

PT requested by the network of NRLs at the 8th EURL-HM Workshop (24th of September 2013) aiming to:

- Test the competences of the appointed NRLs to determine the total As, Cd, Pb, Hg, Sn and inorganic As mass fraction in a **vegetable food matrix** and in the **determination of Sn**.
- Gather information about how NRLs deal with the analysis of canned/jarred food: **drained product** or **solid/liquid composite**

Samples

Reference values

Participants

Measurement results

Test item

Commercially available frozen peas jarred (IRMM) with spiked brine solution

Commission Regulation (EC) 1881:2006 sets maximum levels for certain contaminants in foodstuffs :

Regarding heavy metals in canned food, limits are set only for tin (200 mg kg^{-1} wet weight).

- Pb in legume vegetables (0.2 mg kg^{-1} wet weight)
- Cd in vegetables and fruits (0.05 mg kg^{-1} wet weight).

Test item

Preparation

- Preliminary studies preparing ten units of 210-mL glass jars
- Main production of 209 units peas/brine ratio: 1.364, (RSD < 1 %)
- Spiked brine solution (17 L) was prepared with the following composition:
  - < 0.01 mol L^{-1} HCl solution with traces of HF (25 µl L^{-1})
  - 0.3 mg L^{-1} As;
  - 0.3 mg L^{-1} Cd;
  - 0.2 mg L^{-1} Pb;
  - 470 mg L^{-1} Sn and
  - 6.9 g L^{-1} of NaCl.
- Jars were placed at 60ºC for 2 weeks for equilibrium (HMs in peas/solution) to be reached.

Time frame

Samples dispatched on 22 – 28 of April

Deadline for submitting the results - 6th of June.

Preliminary report sent to participants - 10th of July.

Reference values

Certifiers (two sets of jars were sent to obtain assigned values for drained product and solid/liquid composite)

- Homogeneity and stability studies were undertaken by:
  ALS Scandinavia AB (Luleå, Sweden)
- Assigned values for total As, Cd, Pb, Hg and Sn by:
  IRMM – Institute for Reference Materials and Measurements, SID unit (Geel, Belgium)
  ALS Scandinavia AB (Luleå, Sweden); and
  SCK-CEN – Studiecentrum voor Kernenergie (Mol, Belgium)
- Assigned values for inorganic As by:
  Institut für Chemie, Bereich Analytische Chemie, Karl-Granzens Universität (Graz, Austria); and
  Department of Analytical Chemistry, Faculty of Chemistry, University of Barcelona, (Barcelona, Spain)

Reference values – Drained product

Drained product	Total As	Total Cd	Total Pb	Total Sn	Inorganic As
Certifier 1	*0.111 ± 0.021*	*0.193 ± 0.033*	*0.114 ± 0.022*	*269 ± 37*	*0.106 ± 0.008*
Certifier 2		*0.191 ± 0.009*	*0.117 ± 0.006*	*261.2 ± 14.7*	*0.09 ± 0.005*
Certifier 3	*0.112 ± 0.015*			*296.43 ± 14.1*	
Certifier 4	*0.129 ± 0.005*				
X_{Ref}	**0.117**	**0.192**	**0.116**	**275.5**	**0.098**
u_{char}	0.005	0.011	0.007	8.8	0.008
u_{hom}	0.006	0.003	0.006	5.0	0.005
u_{st}	0.004	0.003	0.003	4.7	0.004
u_{Ref}	0.009	0.012	0.009	11.1	0.010
U_{Ref} (*)	**0.018**	**0.023**	**0.019**	**22.3**	**0.020**
σ_p	0.026	0.038	0.025	33.1	0.022
σ_p (%)	22.0%	20.0%	22.0%	12.0%	22.0% ← Horwitz

X_{ref} is the reference value and $U_{ref} = k \cdot u_{ref}$ is the estimated associated expanded uncertainty; with a coverage factor k= 2 corresponding to a level of confidence of about 95 %.

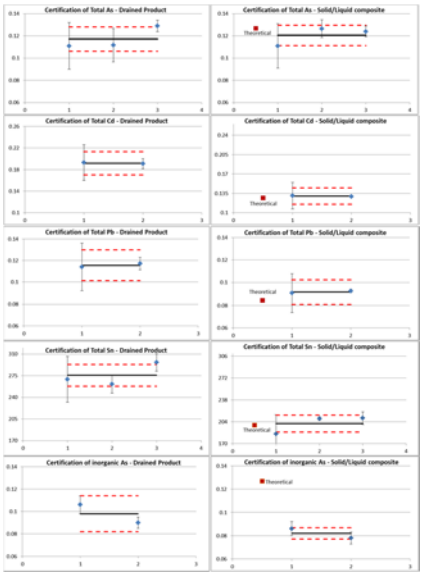
Reference values – Solid/Liquid composite

olid / Liquid composite	Total As	Total Cd	Total Pb	Total Sn	Inorganic As
Certifier 1	*0.111 ± 0.02*	*0.131 ± 0.024*	*0.091 ± 0.017*	*185 ± 30*	*0.086 ± 0.006*
Certifier 2		*0.129 ± 0.002*	*0.092 ± 0.001*	*209 ± 3*	*0.078 ± 0.005*
Certifier 3	*0.127 ± 0.008*			*210 ± 10*	
Certifier 4	*0.124 ± 0.005*				
X_{Ref}	**0.121**	**0.130**	**0.092**	**201.2**	**0.082**
u_{char}	0.005	0.008	0.005	6.6	0.002
u_{hom}	0.003	0.002	0.002	3.2	0.002
u_{st}	0.004	0.002	0.002	3.4	0.003
u_{Ref}	0.007	0.008	0.006	8.1	0.004
U_{Ref} (*)	**0.014**	**0.016**	**0.012**	**16.2**	**0.008**
σ_p	0.027	0.028	0.020	24.1	0.018
σ_p (%)	22.0%	21.5%	22.0%	12.0%	22.0%

← Horwitz

X_{ref} is the reference value and U_{ref}= k·u_{ref} is the estimated associated expanded uncertainty; with a coverage factor k= 2 corresponding to a level of confidence of about 95 %.

Interesting!!



	As	Cd	Pb	Sn	iAs
Drained					
uchar	0.005	0.011	0.007	8.799	0.008
uhom	0.006	0.003	0.006	4.960	0.005
uRef	0.009	0.012	0.009	11.134	0.010
URef (*)	0.018	0.023	0.019	22.268	0.020
Solid / Liquid					
uchar	0.005	0.008	0.005	6.609	0.002
uhom	0.003	0.002	0.002	3.219	0.002
uRef	0.007	0.008	0.006	8.108	0.004
URef (*)	0.014	0.016	0.012	16.216	0.008

- There is a disagreement between spiked and observed concentrations of iAs in the S/L composite
- The sampling procedure is critical for this kind of analysis.

Samples

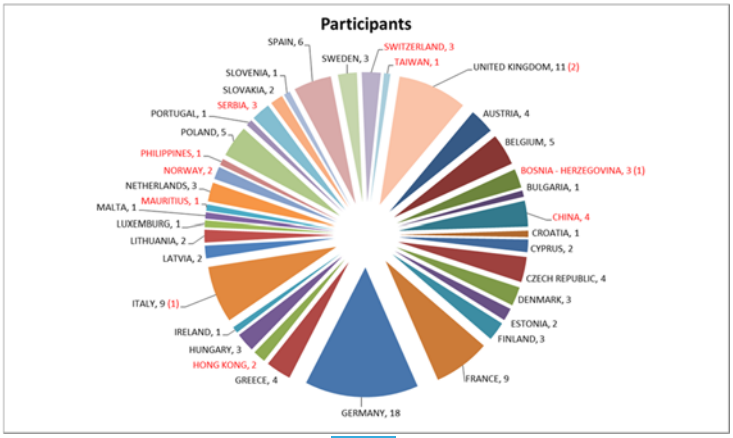
Reference values

Participants

Measurement results

IMEP-118

- Registered 127 laboratories (123 reported results) from 36 countries
  - 36 NRLs from 26 countries (all reported results)



Samples

Reference values

Participants

Measurement results

Drained product *Vs.* solid / liquid composite ?

Laboratories from 17 countries followed both approaches

Drained	Country	Solid / liquid
4	BELGIUM	1
1	BOSNIA - HERZEGOVINA	1
3	CHINA	1
2	CZECH REPUBLIC	2
2	DENMARK	1
1	ESTONIA	1
1	FINLAND	2
4	FRANCE	5
3	GERMANY	15
2	GREECE	2
6	ITALY	2
1	LATVIA	1
1	NORWAY	1
2	POLAND	3
2	SWEDEN	1
1	SWITZERLAND	2
6	UNITED KINGDOM	3

Laboratories from 9 countries followed one of the two approaches

Drained			Solid / Liquid	
AUSTRIA	4		BULGARIA	1
CYPRUS	2		CROATIA	1
HONG KONG	2		LUXEMBURG	1
IRELAND	1		HUNGARY	3
LITHUANIA	2		MAURITIUS	1
MALTA	1		PHILIPPINES	1
NETHERLANDS	3		SERBIA	3
PORTUGAL	1		SLOVAKIA	2
SLOVENIA	1		TAIWAN	1
SPAIN	5			

General information

	Whole population	Non-NRLs	NRLs		
Registered	127	91	36		
Reported	123	87	36		

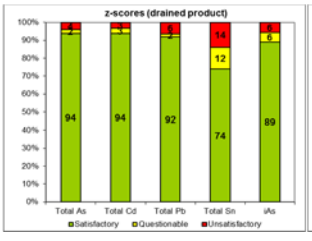
Drained product

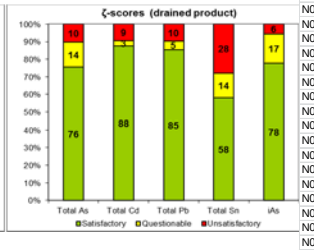
	Whole population	Non-NRLs	NRLs	Non-NRLs	NRLs
	67	46	21		
	General information			*Performance*	
	Values / less than			*S(%) / Q / U*	
As	51 / 8	33 / 6	18 / 2	31(93) / 0 / 2	10(89) / 1 / 1
Cd	66 / 0	45 / 0	21 / 0	41(91) / 2 / 2	21(100) / 0 / 0
Pb	64 / 2	43 / 2	21 / 0	39(91) / 0 / 4	20(95) / 1 / 0
Hg	9 / 49	5 / 34	4 / 15	-	-
Sn	50 / 0	35 / 0	15 / 0	25(71) / 5 / 5	12(80) / 2 / 1
iAs	19 / 2	7 / 2	12 / 0	6(86) / 0 / 1	10(83) / 1 / 1

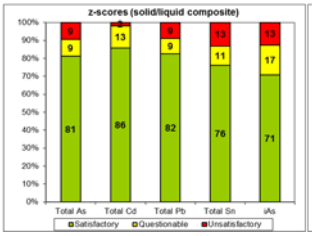
Solid/Liquid composite

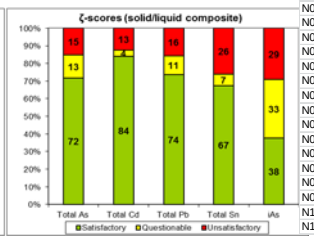
	Whole population	Non-NRLs	NRLs	Non-NRLs	NRLs
	56	41	15		
	General information			*Performance*	
	Values / less than			*S(%) / Q / U*	
As	51 / 2	37 / 2	14 / 0	30(81) / 4 / 3	12(86) / 1 / 1
Cd	54 / 2	39 / 2	15 / 0	36(92) / 3 / 0	12(80) / 2 / 1
Pb	55 / 0	40 / 0	15 / 0	34(85) / 2 / 4	13(86) / 1 / 1
Hg	14 / 36	11 / 27	3 / 9	-	-
Sn	46 / 1	35 / 1	11 / 0	27(77) / 4 / 4	9(81) / 1 / 1
iAs	22 / 3	16 / 2	6 / 1	12(71) / 3 / 1	5(83) / 1 / 0

Overall performances



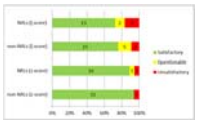


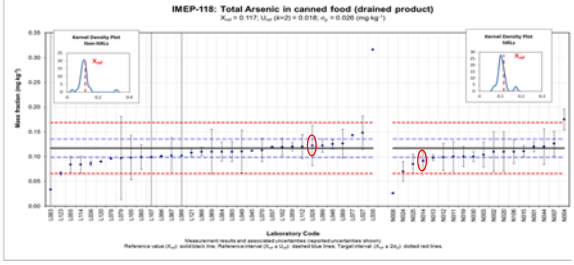


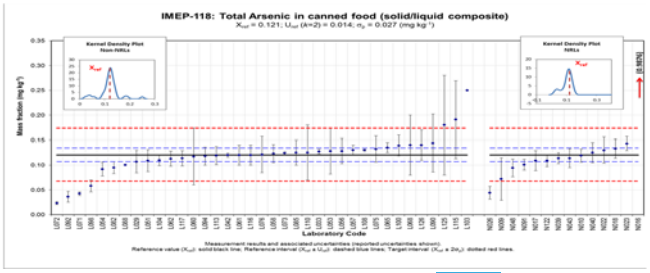


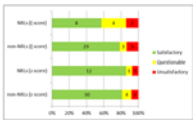
	Total As	Total Cd	Total Pb	Total Sn	iAs
N001	0.11	0.73	1.36		1.48
N002	-0.28	-0.05	-0.81		-1.30
N003	-0.51	-0.10	0.41		-0.70
N004	2.22	0.60	1.63		
N005		0.47	0.96	1.86	
N006		0.73	2.14	2.04	
N007	0.34	-0.02	0.77	0.95	2.60
N008	-3.53	-0.07	0.73	2.68	-4.38
N009	-1.80	2.07	0.81		
N010	-0.06	0.39	2.30		
N011	-0.66	-0.31	-0.22	0.53	
N012	-0.70	-0.57	-0.22	0.50	0.00
N013	-0.74	-0.05	0.18	0.04	-0.23
N014	-1.00	0.21	0.14	0.44	
N015	-0.24	0.37	1.79	0.92	0.00
N016	32.12	23.45	27.64		
N017	-0.43	1.25	-0.67	-2.08	
N018	0.46	0.18	0.32	-2.00	-1.22
N019	-0.66	-0.05	-0.22	0.74	0.56
N020	-0.28	-1.35	-1.04	-4.70	0.09
N021		-2.47	-0.08	1.28	
N022	0.35	0.02	0.63		
N023	0.83	0.39	-0.77	-0.09	
N024	-1.81	-0.86	0.18		
N025	-1.24	-0.07	-0.14	0.33	-0.97
N026	-2.83	-1.11	-1.66	-4.22	-2.39
N030	-0.66	0.99	0.57	1.83	
N039	-0.24	-1.18	0.17	-0.26	0.72
N040	0.17	0.14	-0.08	0.36	1.78
N041		-0.05	0.57		
N043	-0.24	0.14	-0.18	0.03	-0.33
N044	0.11	0.73	0.96	1.95	
N048	-0.97	-1.47	-0.03	0.15	
N091	-0.72	-0.54	0.37	0.82	
N106	-0.28	-0.44	0.57	0.29	0.56
N122	-0.43	-0.40	0.07	0.41	0.72

IMEP-118: Total As

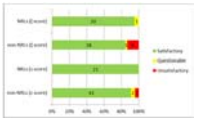


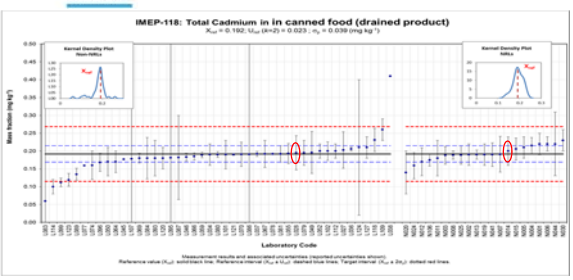


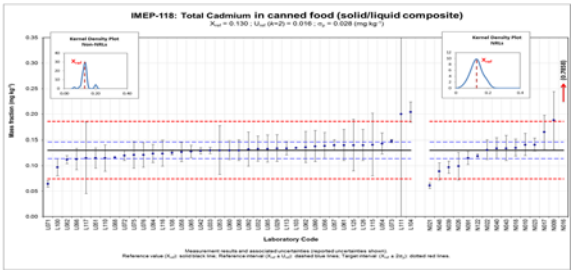




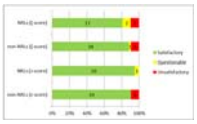
IMEP-118: Total Cd

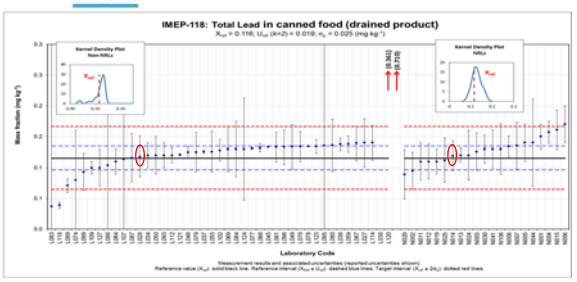


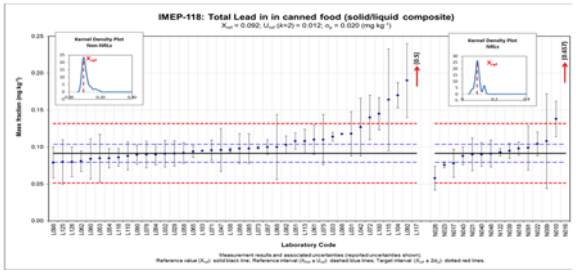


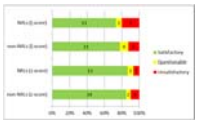


IMEP-118: Total Pb









IMEP-118: Total Hg

Certifiers reported:

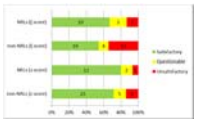
<0.002 mg kg^{-1}

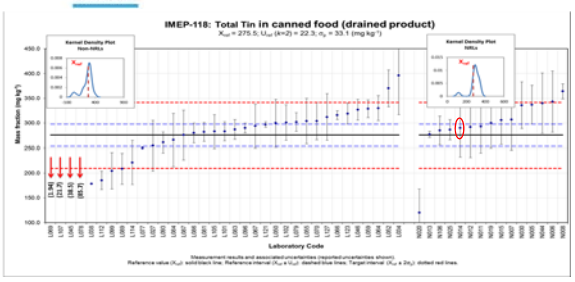
<0.02 mg kg^{-1}

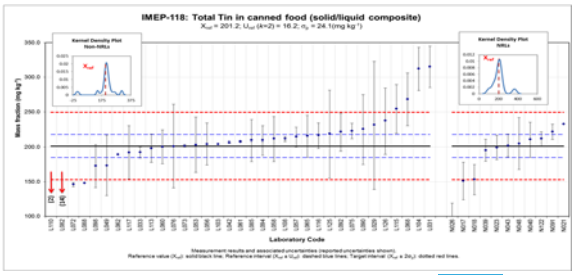
23 participants (7 NRLs), reported results

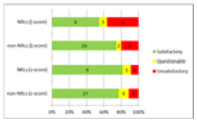
Sample	Xlab	LODs	U	K	Technique
D	0.0002	0.000051	0.00006	2	DMA
S/L	0.013	0.0000723	0.003	2	DMA
D	0.0005	0.0002	0.0001	2	DMA
D	0.001		0.001	2	ICP-MS
D	0.009	0.005	0.001	2	CV-AAS
S/L	0.0004	0.0001	0.0001	2	DMA
S/L	0.00038	0.00002	0.000021		DMA
S/L	0.0199	0.006	0.0029	2	ICP-MS
S/L	0.01	0.0005	0.004	2	CV-AAS
D	0.00073	0.0017	0.00008	2	DMA
S/L	0.002				ICP-MS
D	0.03	0.02	0.041	2	ICP-MS
S/L	0.01	0.01	0.002	2	CV-AAS
S/L	0.0014	0.0002	0.002	2	DMA
S/L	0.0011				CV-AAS
S/L	0.0047	0.001	0.0014	2	CV-AAS
S/L	0.0042		0.00067	1	DMA
S/L	0.00012	0.0001	0.00001	1	FIMS
D	0.13	0.02	0.02	2	DMA
D	0.032	0.01	0.006	2	FAAS-MHS
S/L	0.0007	0.0002	0.0001	2	CV-AAS
D	0.09	0.005	0.017	2	CV-AAS
S/L	0.01	0.05	0.002	2	ICP-MS

IMEP-118: Total Sn

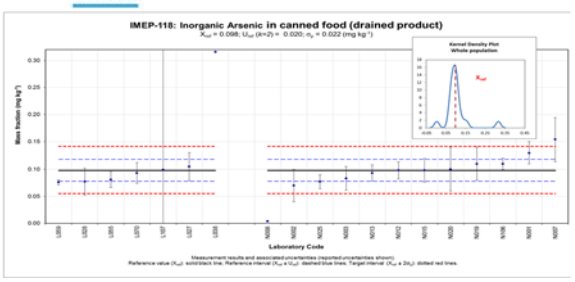


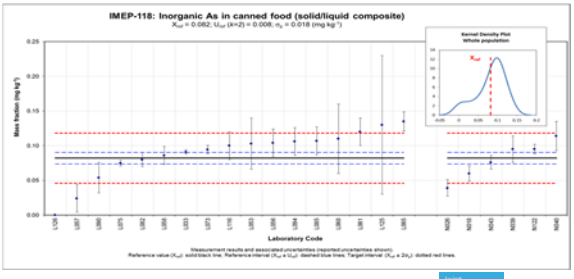


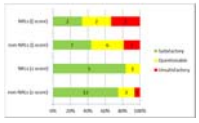




IMEP-118: inorganic As

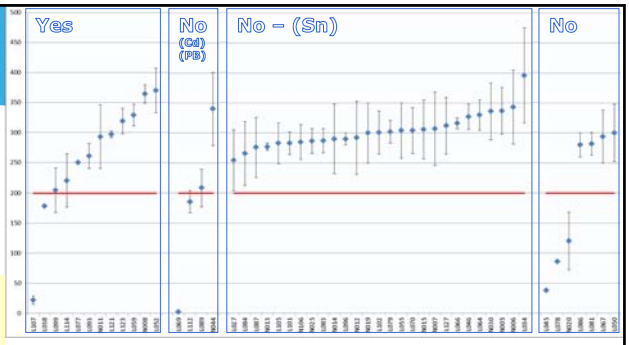






Is the test item compliant?

Drained product



Is the test item compliant?

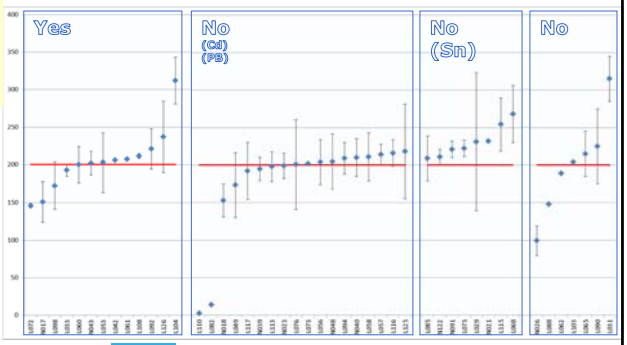
72 (26) : NO

32 (6) : YES

19 (4) : no reply

Labs (NRLs)

Solid/Liquid composite



Joint Research Centre

European Commission

Conclusions IMEP-118:

1. The participants were divided into two categories for the analysis of the test item.
2. The performance of the participating laboratories for both sampling approaches was satisfactory with NRLs performing slightly better.
3. A protocol or guidelines should be drawn taking into consideration:
   - The different sampling approaches.
   - Canned products are chemically active environments (migration of analytes, binding of As(V)).
4. In the case of total Sn and iAs, although the situation improved, it can be even better.
5. An extra effort is needed for the evaluation of uncertainties.
6. Significant discrepancies were observed for the limits of detections reported.

Joint Research Centre

12. IMEP-119

HM in vegetable feed

Fernando Cordeiro

Joint Research Centre

The European Commission's in-house science service

IMEP-119: Determination of total As, Cd, Pb and Hg in vegetable feed

www.jrc.ec.europa.eu

F. Cordeiro, I. Fiamegkos, A. Cizec-Stroh, B. De la Calle, P. Robouch

Serving society
Stimulating innovation
Supporting legislation

Test items

Reference values

Instructions to participants

Participants

Measurement results

Test item

- Vegetable feed (Alfalfa-meal) – sieved, milled, homogenised and freeze dried,
- > 25 g of material in 125 mL amber glass vials.

Homogeneity & stability

- Measurements for homogeneity and stability studies were undertaken by the Centro de Salud Pública de Alicante (Spain).
- Evaluation according to ISO 13528:2005 (IRMM).

Certifiers:

- *Federal Institute for Materials Research and Testing (Germany)*
  Quadrupole ICP-MS (As, Cd, Pb) + CV-AFS (Hg)
- *Centro de Salud Pública de Alicante (Spain)*
  ICP-MS (As, Cd, Pb) + Elemental Hg analyser (Hg)
- *ALS Scandinavia (Sweden)*
  ICP-Sector field-MS
- *SCK-CEN Studiecentrum voor Kernenergie (Belgium)*
  Neutron Activation Analysis (As, Cd, Hg)

Reference values

	As	Cd	Pb	Hg
Certifier 1	1.2 ± 0.23	0.12 ± 0.025	3.06 ± 0.67	0.008 ± 0.0008
Certifier 2	1.14 ± 0.17	0.122 ± 0.016	3.22 ± 0.31	0.0072 ± 0.00033
Certifier 3	1.2 ± 0.07	0.142 ± 0.008	3.23 ± 0.023	
Certifier 4	1.19 ± 0.06			
X_{ref}	**1.183**	**0.128**	**3.170**	**0.0076**
u_{char}	0.0470	0.0064	0.1539	0.00027
u_{bb}	0.0248	0.0032	0.0507	0.00023
u_{st}	0.0272	0.0023	0.0634	0.00027
u_{ref}	**0.0597**	**0.0075**	**0.174**	**0.00044**
U_{ref} (*)	0.119	0.015	0.348	0.0009
σ_p	**0.177**	**0.019**	**0.476**	**0.0017**
σ_p (%)	15.0%	15.0%	15.0%	22.0%

Modified Horwitz

Instructions to participants

- Participants were allowed to use their own procedures
- The **measurement results** were to be **corrected for;**

  i) **recovery** and,

  ii) **moisture content**, following procedure provided

Test item

Instruction to participants

Reference values

Participants

Measurement results

- **Registered,**

32 NRLs *(from 27 countries),*

14 feed control laboratories,

45 nominated by EA,

10 nominated by IAAC,

1 nominated by APLAC

102 laboratories from **45** countries

(**31 participants from non EU**)

Test item

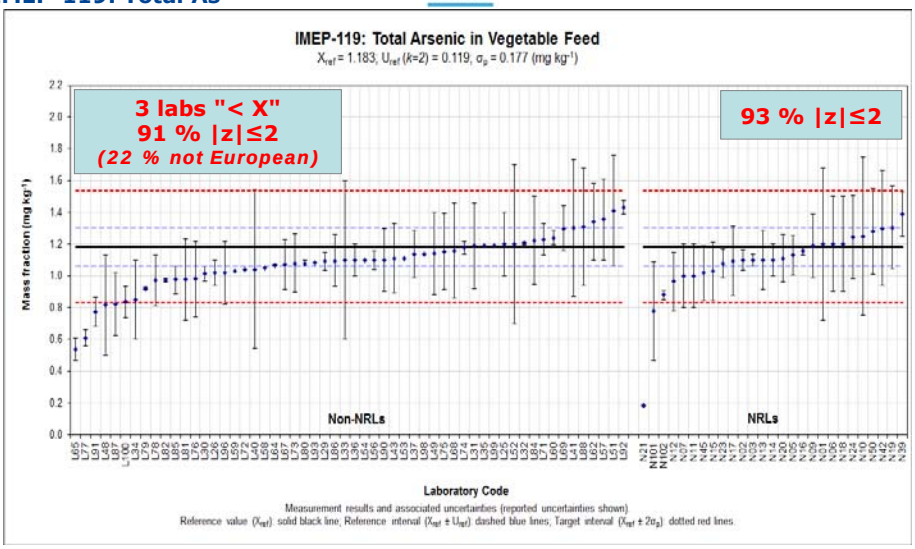
Instruction to participants

Reference values

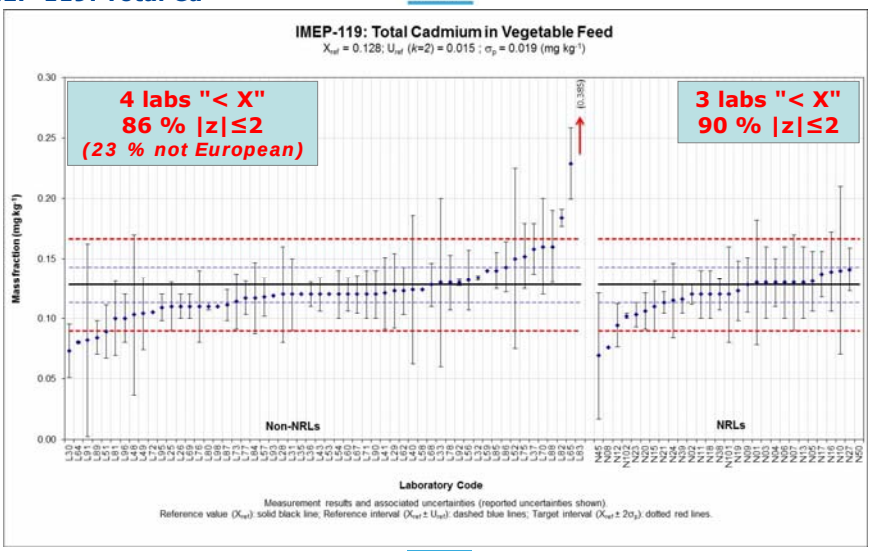
Participants

Measurement results

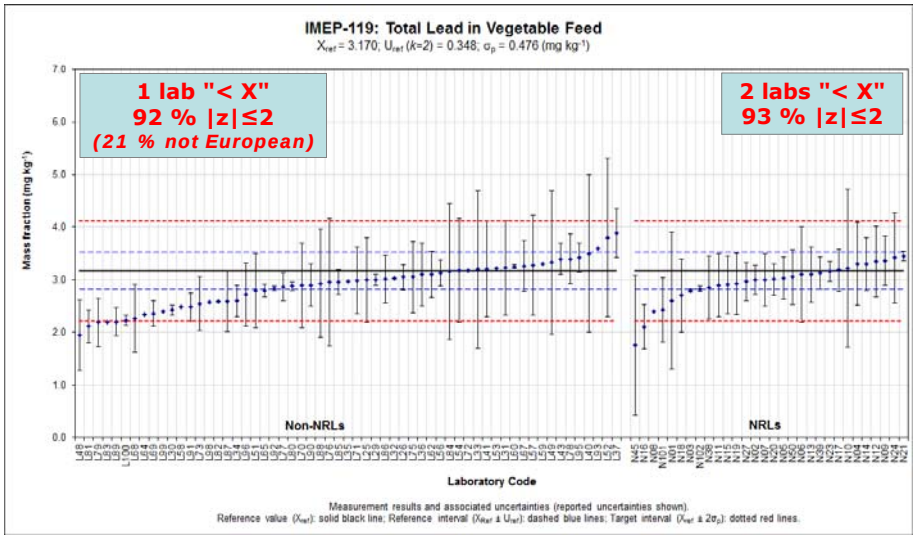
IMEP-119: Total As



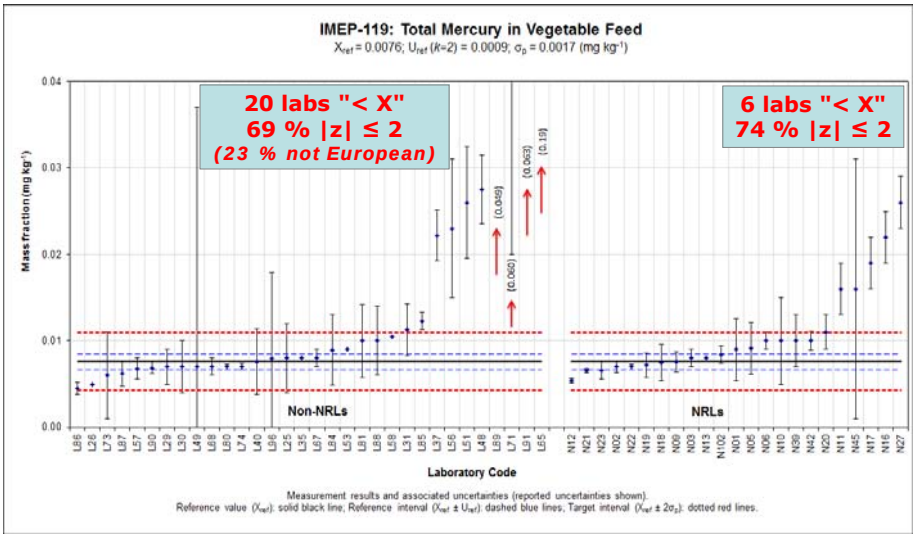
IMEP-119: Total Cd



IMEP-119: Total Pb



IMEP-119: Total Hg



	Technique			
	As	Cd	Pb	Hg
N01				
N02	SFICP-MS	SFICP-MS	SFICP-MS	SFICP-MS
N03	ETAAS	ETAAS	ETAAS	CV-AFS
N04		Q-ICP-MS	Q-ICP-MS	FIMS (< X)
N05	Q-ICP-MS	Q-ICP-MS	Q-ICP-MS	Q-ICP-MS
N06	ICP-OES	ICP-OES	ICP-OES	EMA
N07	ICP-MS	ICP-MS	ICP-MS	
N08				
N09	ICP-MS	ICP-MS	ICP-MS	ICP-MS
N10	ICP-MS	ICP-MS	ICP-MS	CV-AAS
N11	SFICP-MS	SFICP-MS	SFICP-MS	EMA
N12	ICP-MS	ICP-MS	ICP-MS	EMA
N13	ICP-MS	ICP-MS	ICP-MS	EMA
N14	ETAAS	ETAAS (< X)	ETAAS	EMA (< X)
N15	SFICP-MS	SFICP-MS	SFICP-MS	SFICP-MS (< X)
N16	HG-AAS	ETAAS	ETAAS	CV-AAS
N17	SFICP-MS	SFICP-MS	SFICP-MS	EMA
N18	ICP-MS	ICP-MS	ICP-MS	EMA
N19	ICP-MS	ICP-MS	ICP-MS	CV-AFS
N20	SFICP-MS	SFICP-MS	SFICP-MS	CV-AAS
N21				
N22		AAS (< X)	AAS (< X)	EMA
N23	ICP-MS	ICP-MS	ICP-MS	EMA
N24	HG-AAS	GETAAS	ETAAS	HG-AAS (< X)
N27		AAS	AAS	CV-AAS
N38		AAS	AAS	
N39	ICP-MS	ICP-MS	ICP-MS	EMA
N42	HG-AAS	FAAS (< X)	FAAS (< X)	CV-AAS
N45	HG-AAS	ETAAS	ETAAS	CV-AAS
N50	ICP-MS	ICPMS	ICP-MS	ICP-MS (< X)
N101	ETAAS	ETAAS	ETAAS	EMA (< X)
N102				

NRL performance (z-) & technique

- **Conclusions:**

  - ✓ **Overall good performance** *(92 % As & Pb, 87 % Cd, 71 % Hg, particularly for NRLs)*
  - ✓ **NRLs estimated better their MU**.
    More satisfactory performance (ζ-scores) and higher % of $u_{ref} < u_{lab} < \sigma_p$ *than Non-NRLs*
  - ✓ **No significant influence from the technique used, however …**

    All unsatisfactory (2) or questionable (3) performance for **As** used **AAS / ETAAS**

    38 % of the participants having unsatisfactory performance for **Hg** used **CV-AAS.**

  - **Inconsistency in LoD** *(0.0004 – 2.5 mg* kg^{-1} *for As).*
    Majority of "less than X" → X = LoD

13. Future Activities
2015 & beyond

Piotr Robouch

Future Activities
2015 & beyond

P. Robouch

2015

- *PT* ***chocolate***
- *PT* ***Mineral Feed*** *(incl. kaolinitic clay)*
- ***10th WS****; in Brussels? In September?*
  Invite SANCO & EFSA; EURL-CEFAO & EURL-FA
- ***LOD/LOQ***
- ***NRL Report*** *[2015]: PTs, WS, other activities*
- *Share of information through* ***CIRCABC***
- *Draft "****Criteria Approach for HM in feed****"*
- *Review* ***COM 152/2009 for Fe, Cu, Mn, Zn***
- *Support to SANCO, EFSA, CEN, NRL*

2016

- *PT1: Fish/seafood/seashell (MetHg)*
- *PT2:*
- *11th WS: EURL-HM 10th Anniversary*
- *Training needs to be defined in 2015*

Satisfaction Survey (1/3)

1. ***Was the scientific program of the Workshop 2014 appropriate?***
   - *1a. Discussions related to 2014 PT results (IMEP-118 & IMEP-119)*
   - *1.b Presentation of (next) PTs included in the Work Program 2015*
   - *1c. Identification of future PTs for 2016*
   - *1d. Relevance to the tasks of your NRL*
   - *1e. Time dedicated to Presentations & Discussions*
   - *1f. Overall rating of the organisation & structure of the WS 2014*
   - *1g. Would you agree replacing proceedings (paper) printouts by e-proceedings (pdf)?*

➔ *rate Very GOOD (1) to very bad (5)*

Satisfaction Survey (2 / 3)

*2. **Your opinion about the 2014 PTs***

- *2a. Description of samples & tasks of the 2014 PTs*
- *2b. Timing of the 2014 PTs*
- *2c. Communication with the EURL during the 2014 PTs*
- *2d. Capability & handling of the MILK interface for registering and reporting results*
- *2e. Evaluation of the PT report(s) to participants*
- *2f. Timing of publication of the PT reports*
- *2g. Overall rating of the 2014 PTs*

➔ *rate Very GOOD (1) to very bad (5)*

Satisfaction Survey (3 / 3)

Logistics

- *Meeting place*
- *Meals*
- *Desk assistance during the meeting*
- *Communication with the IRMM/EURL*
- *How was the IRMM reaction to your questions? (logistics, transport, hotel, various info)*

Your opinion matters

- *Would you like to suggest some improvements/changes*

ⓘ Satisfaction Survey (LINK) will be sent to you by email

Follow-up (|z| > 3)

EURL
European Union Reference Laboratory
Heavy Metals in Feed and Food

Follow-up Form

Confidential

❶ **Root Cause Analysis.** Present the main causes identified (250 words max.)

Example: reporting blunder, poor quality reagents/calibrants, sample prep or instrumental problems, other.

❷ **Corrective Actions Implemented.** When relevant, present solutions implemented (250 words max.)

❸ **Demonstrated Effectiveness** Provide experimental evidences, if available (250 words max.)

Name		*Signature*
Function		
Date		

Please Fill, Print, Sign the form. Then Scan and Send it to jrc-irmm-eurl-heavy-metals@ec.europa.eu

Please send when available

- *Annual Report of the NRL*
- *Report to participants of NRL PTs*
- *Workshop proceedings*

Confidentiality? Upload on CIRCABC ? Y/N

Duty of the NRL contact person:

to maintain up-to-date list of members

Each member to update their contact info

Thank you for your continued support !

Other EURL-HM e-forms

Survey: Getting Ready for the EURL-HM Workshop 2014

In order to organise a fruitful EURL-HM Workshop 2014
we would like to hear already some of your expectations.
Thank you for answering the questions below.
Looking forward to meet you soon in Brussels.
/ piotr Robouch, on behalf of the EURL-HM

2 - Topics for the Workshop 2014

2a - Specify which **Proficiency Tests** (PTs) you would like to be organised by the EURL in **2016**?
NB: "HM in chocolate" & "HM in mineral feed" are planned in 2015*

2b - Specify which **training** you would like to have during the (next) **WS in 2015**?
NB: "Experimental Design" is on the agenda this year*

2c - Do you have any **specific topic** you would like to **discuss** during **this WS 2014**?*

2d - Would you like to give a **short presentation** (10 min+Q) on a technical issue relevant to your NRL? (at this WS 2014)
If yes, please provide a short Title. ("NO" for none)*

3 - NRL activities in 2014

3a - Which PTs did your NRL organise in 2014?
Specify for **each** PT
(i) analytes and matrix;
(ii) nr of participants;
(iii) summary overview of performances of participating labs;
(iv) when was the PT organised.
Please provide report to participants (when available) by email to the EURL*

3b - NRL workshop 2014 for national official control labs
Specify (i) When, (ii) Where, (iii) Nr of participants, (iv) oral presentations and (v) problems identified/discussed*

3c - Any other relevant NRL activities in 2014*

4 - Other comments

Did we miss something? Would you like to suggest something else?

Satisfactio Survey EURL-HM-2014

Fields marked with * are mandatory.

EURL-HM / Customer Satisfaction 2014

Dear Colleague,
The EURL-HM is committed to deliver high quality services to the NRL network. We are therefore very interested in your opinion related to the workshop 2014 and the two PT exercises organised this year. Thank you for your evaluation.

European Union Reference Laboratory
Heavy Metals in Feed and Food

1. Was the scientific program of the Workshop 2014 appropriate?

1a. Discussions related to 2014 PT results (IMEP-118 & IMEP-119)*
1 - VERY GOOD 2 3 4 5 6 - very bad

1.b Presentation of (next) PTs included in the Workp Program 2015*
1 - VERY GOOD 2 3 4 5 6 - very bad

1c. Identification of future PTs for 2016*
1 - VERY GOOD 2 3 4 5 6 - very bad

1d. Training on Statistical Experimental Design*
1 - VERY GOOD 2 3 4 5 6 - very bad

1e. Relevance of the various topics to the tasks of your NRL*
1 - VERY GOOD 2 3 4 5 6 - very bad

1f. Time dedicated to Presentations & Discussions*
1 - VERY GOOD 2 3 4 5 6 - very bad

1g. Overall rating of the organisation & structure of the WS 2014*

1 - VERY GOOD 2 3 4 5 6 - very bad

1h. The event provided me with Networking opportunities*

1 - Strongly AGREE 2 3 4 5 6 - strongly desagree

1i. The event improved my knowledge and expertise in my field of science and research*

1 - Strongly AGREE 2 3 4 5 6 - strongly desagree

2. Your opinion about the 2014 PTs

2a. Description of samples & tasks of the 2014 PTs*

1 - VERY GOOD 2 3 4 5 6 - very bad

2b. Timing of the 2014 PTs*

1 - VERY GOOD 2 3 4 5 6 - very bad

2c. Communication with the EURL during the 2014 PTs*

1 - VERY GOOD 2 3 4 5 6 - very bad

2e. Evaluation of the PT report(s) to participants*

1 - VERY GOOD 2 3 4 5 6 - very bad

2f. Timing of publication of the preliminary PT reports (before the Workshop)*

1 - VERY GOOD 2 3 4 5 6 - very bad

2g. Timing of publication of the final PT reports (expected October 2014)*

1 - VERY GOOD 2 3 4 5 6 - very bad

1j. Would you agree replacing proceedings (paper) printouts by e-proceedings (pdf)? Please consider ecological reasons & remember that presentations will be available after the WS.*

1 - AGREE 2 3 - do NOT agree

2h. Overall rating of the 2014 PTs*

1 - VERY GOOD 2 3 4 5 6 - very bad

2d. Capability & handling of the MILC interface for registering and reporting results*

1 - VERY GOOD 2 3 4 5 6 - very bad

(optional) Your opinion about chapters, topics or information in the PT reports. Points to be covered in more details? Additional topics to be included? Please specify.

The EURL-HM webpage - LINK

Did you visit the EURL-HM webpage?*

- Yes
- No

Do you find the EURL-HM website useful*

1 - VERY USEFUL 2 3 4 5 6 - not useful at all

According to you, which information is most useful?

According to you, which information is MISSING? (which info should be added?)

Logistics

Meeting place*

1 - VERY GOOD 2 3 4 5 6 - very bad

Meals*

1 - VERY GOOD 2 3 4 5 6 - very bad

Desk assistance during the meeting*

1 - VERY GOOD 2 3 4 5 6 - very bad

Communication with the IRMM/EURL - for logistics, transport, hotel, other info*

1 - VERY GOOD 2 3 4 5 6 - very bad

How was the IRMM/EURL reaction to your questions?*

1 - VERY GOOD 2 3 4 5 6 - very bad

Your Opinion Matters

Would you like to suggest some improvements/changes

EUROPEAN COMMISSION
DIRECTORATE GENERAL
JOINT RESEARCH CENTRE
Directorate D: Institute for Reference Materials and Measurements
European Union Reference Laboratory for Heavy Metals

European Union Reference Laboratory
Heavy Metals in Feed and Food

Geel, xx/xx/xxxx

xxx NRL-name xxx
xxx City, Country xxxx
xxx e-mail xxxx
(sent by e-mail)

Subject: Follow-up xxxx

To whom it may concern,

The results you submitted (X_{lab}, below) in the frame of the xxx PT code xxx "xxx PT name/title xxx" were evaluated as *unsatisfactory* ($|z| > 3$). More details to be found in the corresponding report for participants[1].

Analyte	X_{ref}	σ_p	X_{lab}	*z-score (*)*

(*) $z = (X_{ref}-X_{lab})/\sigma_p$

Regulation 882/2004 Art. 32 or the European Parliament and Council states that "*the EURL shall be responsible for organising comparative testing and ensuring an appropriate **follow-up** of such comparative testing in accordance with internationally accepted protocols*". Hence, according to clause 4.9 on non-conformity of the ISO 17025 standard your laboratory shall perform a **root-cause analysis** (RCA) for each analyte listed above to determine the causes that contributed to the unsatisfactory results. Your laboratory shall then list the root cause(s) and identify any relevant **corrective action(s)** necessary to prevent the non-conforming work to occur again. The RCA shall be (i) as specific as possible; (ii) reasonably identifiable and (iii) able to be managed/controlled. Finally your laboratory shall present, if available, the follow-up actions performed to demonstrate the **effectiveness** of the improvement actions undertaken.

Please fill the form attached to this letter (following page), sign it and send it to the EURL **before the xx/xx/xxxx.**

For additional information, do not hesitate to contact the EURL.
Thank you for your collaboration

Piotr Robouch
EURL-HM, Operating Manager

(1) http://irmm.jrc.ec.europa.eu/EURLs/EURL_heavy_metals/interlaboratory_comparisons

Retieseweg 111, B - 2440 Geel – Belgium
Tel: +32 14 571 211; Direct line +32 14 571 980
jrc-irmm-eurl-heavy-metals@ec.europa.eu
http://irmm.jrc.ec.europa.eu/EURLs/EURL_heavy_metals

Follow-up Form

Confidential

❶ **Root Cause Analysis.** Present the main causes identified (250 words max.)

Example: reporting blunder, poor quality reagents/calibrants, sample prep or instrumental problems, other.

❷ **Corrective Actions Implemented.** When relevant, present solutions implemented (250 words max.)

❸ **Demonstrated Effectiveness** Provide experimental evidences, if available (250 words max.)

Name		*Signature*
Function		
Date		

Please Fill, Print, Sign the form. Then Scan it and Send to jrc-irmm-eurl-heavy-metals@ec.europa.eu

European Commission
JRC92903 – Joint Research Centre – Institute for Reference Materials and Measurements

Title: **Proceedings of the 9th workshop of the European Union Reference Laboratory & National Reference Laboratories for Heavy Metals in Food and Feed – Brussels, 9 September 2014**

Authors: Beatriz de la Calle, Fernando Cordeiro, Yiannis Fiamegkos, Aneta Cizek-Stroh, Mitja Vahčič and Piotr Robouch

2014 – 148 pp. – 21.0 x 29.7 cm

Abstract
A total of forty-five participants attended the 9th workshop of the EURL-HM held in Brussels on September 9, 2014. The 2014 activities of the EURL-HM were reviewed and the outcome of the two proficiency tests organised for the determination of heavy metals in canned peas and vegetable feed were discussed. Ten oral presentations constituted the agenda of the event, together with extensive discussions. The summary of the workshop and all the presentations are included in this report.

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