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Annual Workshop of the European Union Reference Laboratory for chemical elements in food of animal origin

Istituto Superiore di Sanità
Rome, October 19-20, 2017

ABSTRACT BOOK

Edited by
A. Sorbo, M. D'Amato,
G. Fornari Luswergh and L. Ciaralli

ISTITUTO SUPERIORE DI SANITÀ

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Department of Food Safety, Nutrition and Veterinary Public Health

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Edited by Angela Sorbo, Marilena D'Amato, Guendalina Fornari Luswergh and Laura Ciaralli
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This volume collects the abstracts of the contributions presented at the annual workshop, organized by the European Union Reference Laboratory for chemical elements in food of animal origin (EURL-CEFAO) hosted by the Department of Food Safety, Nutrition and Veterinary Public Health of the Italian National Institute of Health. The workshop was held in Aula Marotta. The volume provides an overview of the activities carried out by both the EURL-CEFAO and the National Reference Laboratories of the EU Member States working in the field of chemical elements in food of animal origin. In particular, it is focused on proficiency testings, analytical topics and regulatory issues.

Key words: Chemical elements; Proficiency Testings; European Union Reference Laboratory

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Questo volume raccoglie i riassunti dei contributi presentati durante il workshop annuale, organizzato dal Laboratorio Europeo di Riferimento per gli elementi chimici nelle matrici di origine animale (EURL-CEFAO) che ha sede presso il Dipartimento di Sicurezza Alimentare, Nutrizione e Sanità Pubblica Veterinaria dell'Istituto Superiore di Sanità. Il workshop si è tenuto in Aula Marotta. Il volume offre una panoramica sulle attività svolte dall'EURL-CEFAO e dai Laboratori Nazioni di Riferimento degli Stati Membri dell'Unione Europea operanti nell'ambito degli elementi chimici nelle matrici alimentari di origine animale. In particolare, esso è incentrato sui circuiti interlaboratorio, su argomenti analitici e su problemi regolatori.

Parole chiave: Elementi chimici; Circuiti interlaboratorio; Laboratorio di Riferimento dell'Unione Europea

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NOTE FOR THE READER

This volume gathers all the contributions presented at the conference. Abstracts are divided into oral and poster presentations. For easy consultation, oral presentations are listed in the order of the programme.

Posters are listed after the oral presentations. The poster abstracts are numbered with a code including the letter “P” followed by a progressive number.

At the end of the volume, the authors’ index is provided for the reader’s convenience.

PREFACE

The main objective of the European Commission's food safety policy is to ensure a high level of protection of consumers' health by applying the integrated approach "from farm to fork" covering the whole food chain.

In order to achieve this purpose, laboratories dealing with official controls have to work uniformly within the EU. Therefore, the relationships between the European Union Reference Laboratories (EURLs) and the National Reference Laboratories (NRLs) are strongly encouraged as the NRLs are in strict touch with the Official Laboratories (OLs), rooted in the territory, that usually perform the analytical official controls in each EU country.

According to Article 32 of the EU-Regulation 882/2004, the EURLs, among other tasks, have to organize workshops and training addressed to the NRLs.

These workshops contribute to foster the exchange of experiences and ideas among the participants that are called to disseminate information and knowledge to colleagues working in other official monitoring and surveillance laboratories in their and/or in other countries.

In order to accomplish this task, the European Union Reference Laboratory for chemical elements in food of animal origin (EURL-CEFAO) has organized the annual workshop, at the National Institute of Health in Rome on 19-20 October 2017. Scientists of the NRLs coming from the 28 EU Member States, as well as international experts coming from Brazil, Norway, Iceland and Republic of Macedonia were invited to attend the meeting.

They were also encouraged to contribute to the scientific content of the Workshop by oral or poster presentations.

The reaction of participants was very positive, as demonstrated by the number of works presented. Furthermore, for the second time, a poster session has been included in the workshop, considering the very positive acceptance of this proposal by the participants during the previous EURL-CEFAO workshop.

This volume collects scientific contributions on a wide variety of topics related to activities performed by the NRLs. The workshop was partially focused on analytical issues such as arsenic speciation in seafood and validation of a method to quantify methylmercury and inorganic mercury by HPLC-ICMS.

The representative of the German NRL gave a speech about the advantages of European Standards whose application does not require a full method validation allowing the laboratory to save time and resources. In particular, it was stressed that the preparation of a standard method was a long process that should involve laboratories of different countries. Representatives of the EURL-CEFAO gave speeches focusing on the organization of Proficiency Testings as well as on the difficulties connected to the harmonization of compliance assessment for compound and processed food. A survey of the EURL-CEFAO activity was also presented, as well as the outcome of the 26th PT on freeze dried meat. An Italian Official Control Laboratory was also invited to give the audience information on the activity performed at local level to guarantee the food safety.

To better understand the role of the National Focal Point of the European Food Safety Authority, a representative of the Italian Focal Point explained the activity particularly related the EU risk assessment strategy.

During the meeting a TRAINMIC trainer proposed a training on the construction and use of control chart.

This volume gathers the abstracts of the contributions presented at the "*Annual NRLs – EURL Workshop 2017"* organized by the EURL-CEFAO on 19-20 October 2017.

Laura Ciaralli
Workshop chairperson
EURL-CEFAO Director

Oral session 1

Chairperson
Laura Ciaralli

THE LONG STONY ROAD FROM AN IDEA TO A STANDARD

Timo Kapp
Federal Office of Consumer Protection and Food Safety (BVL), Berlin, Germany

In times of legal stipulations of method performance criteria, the importance of standardized methods could be questioned. It involves a high effort to conduct a collaborative trial, and a high amount of time is invested in the documentation of a standardized method. However, all laboratories using the resulting standard save time in the process of method introduction, as only a verification instead of a whole validation is needed. Another advantage of European standards is their acceptance on CCMAS level, which is not given for national standards or in-house methods.

A minor number of standard methods for elements in food originated from the CEN working group (TC275 WG 10). Examples are the mandated standard methods for methylmercury in marine matrices and for inorganic arsenic in food. In the past most European standards were based on national standards (*e.g.* Germany, Spain) or on standards of regional organisations (NMKL, EURL-NRL-network).

A recent example of an EN standard which had previously been standardized in Germany is "EN16943:2017-Foodstuffs-Determination of calcium, copper, iron, magnesium, phosphorous, potassium, sodium, sulfur and zinc by ICP-OES". The work on this standard started in 2008 in the working group for elements in the food chemistry section of the German Chemical Society (GDCh). This group of experts developed the method by testing for interferences, suitable wavelengths and internal standards. The resulting optimized method was then passed on to the working group for elements according to §64 German Food and Feed Act (Official collection of analytical methods) at the BVL for standardization. A collaborative trial was successfully conducted in 2011. After the reporting of the trial the method was fully documented and published in 2013. In 2014 an English version was submitted to the TC275 WG10 and adopted as a new working item. After two commenting cycles and additional meetings to discuss improvements, EN16943 was published in 2017.

This example shows that, seen from the final stage of publication, the standard has at least a 9-year history. So modern developments need time to get reflected in the standards. In addition to this, a method or technique of some kind has to be established among expert laboratories to enlist enough participants, as for the evaluation of a collaborative trial around 10 laboratories are needed. Even in a large country, it is difficult to obtain cooperation from a sufficient number of experienced participants on a national level. Therefore, the participation of experienced laboratories from other European countries in the elaboration of methods and in collaborative trials is highly appreciated.

Recently, the urgent need for an update of the "EN13806:2002-Foodstuffs-Determination of mercury by cold vapour atomic absorption spectrometry (CV-AAS)" has emerged. This method has become obsolete from a technical point of view, as it is based on the batch technique and not on the flow injection system, which is predominantly used

nowadays. Recent proficiency tests have provided further evidence that the performance parameters of this method are out of date.

In the GDCh-working group, it was agreed that the introduction of a new (modern) method was desired. Therefore the group also prepared a draft for solid-state AAS. When discussing the project in the CEN-WG, the need for a standard using cold vapour atomic fluorescence spectrometry (CV-AFS) emerged. This method was then prepared by Warren Corns from the UK. It was agreed to perform one collaborative trial with all three methods in parallel. For this trial especially participants for the AAS and CV-AFS techniques are welcome.

VALIDATION OF A HPLC-ICPMS METHOD FOR THE DETERMINATION OF METHYLMERCURY AND INORGANIC MERCURY IN FISH AND SEAFOOD

Gerhard Liftinger, Franz Mlynek
AGES GmbH, Linz, Austria

The determination of methylmercury (MeHg) in fish and seafood is an important task for an official control laboratory due to the toxicity of this chemical species. As no HPLC-ICPMS standard methods are available, there is a demand for a robust and suitable method based on this technique. Most of the published methods for this technique foresee the use of harmful chemicals like pyridine or mercaptoethanol as eluent. In order to protect the safety and health of the operators, a robust and sensitive method with nontoxic chemicals for the extraction solution and the eluent for the determination of MeHg and iHg (inorganic mercury) in in fish and seafood was validated by our laboratory. The method was based on the procedure described by Hight and Cheng.

As for instrumentation, HPLC-System (Agilent 1260) with QQQ-ICPMS (Agilent 8800) was used. The samples were extracted with L-Cysteine solution at 0.5% (w/v) in 0.5% HCl (v/v). The elution was performed on a Knauer Eurosphere C18-A column (100 x 4.6 mm) using a L-Cysteine solution at 0.8 % (w/v) in 0.35% HCl (v/v). Hg was determined on mass 202 in nogas mode.

For the estimation of the extraction efficiency the content of total mercury in a fish sample was quantified by ICPMS. The sample was also extracted with the extraction solution and the content of the extracted mercury was determined in the same way. The extraction efficiency resulted to be > 90 %, and thus satisfactory.

The recovery of the column was calculated by comparing the concentration of total Hg in the extracted solution with the sum of the Hg species (MeHg and iHg) after speciation with HPLC-ICPMS. It was found to be satisfactory (> 90 %).

The linearity range was from 0.5 µg/l to 25 µg/l for both inorganic mercury and methylmercury.

The LoD for MeHg is 0.006 mg/kg and for iHg 0.012 mg/kg, the LoQ for MeHg is 0.02 mg/kg and for iHg 0.04 mg/kg.

A certified reference material (CRM) - TORT-2 - Lobster Hepatopancreas from NRC-CNRC was analyzed to assess the trueness of the method. The concentration of MeHg and total Hg lie within the certified range for both CRMs.

Based on the results, the method fitted for the purpose of determining methylmercury and iHg in seafood by using nontoxic chemicals.

TRAINMIC - BUILDING AND USING CONTROL CHARTS

Marina Patriarca
Authorised TrainMiC trainer, Istituto Superiore di Sanità, Rome, Italy

Effective monitoring of the quality of measurement results is an essential part of the requirements for the competence of testing laboratories according to ISO / IEC 17025. The general objective of this training will be to show how to optimize this process through the use of control charts.

To this aim, the theoretical principles and the practical aspects of building, implementing and interpreting control charts will be addressed. The training will be based on material from TrainMiC®, a shared, European-wide, platform for advanced and harmonized professional training in metrology in chemistry, initiated in 2001 as a European project, and will include updated guidance from the ISO 7870 series, ISO Guide 80, the Nordtest TR 569 guide and Eurachem.

Laboratories need to implement suitable internal quality control systems, fulfilling the purposes of continuous monitoring of the quality of data, critical evaluation of individual results and identification of developing problems. Effective quality control planning includes significant elements, such as choice of parameters to be monitored, materials to be used, type of control charts (X cards or R cards), setting of the central line and definition of (statistical or target) alarm and action limits. Clear rules need to be set for the interpretation of the internal quality control results to assure the quality of the analytical results of a single analytical session, monitoring trends and/or detecting variations of analytical performance overtime that may highlight a developing problem.

Finally the training will cover additional uses of control charts, besides monitoring the quality of analytical results. Laboratories can make the most of the information collected in control charts to support the review processes of method validation, Proficiency Test results and estimate of measurement uncertainty, thus maximizing the benefits of the time and effort spent in building and implementing these powerful tools.

Oral session 2

Chairperson
Angela Sorbo

THE EURL-HM AND BEYOND …

Piotr Robouch
European Commission, Joint Research Centre, Directorate Health, Consumers & Reference Materials, Geel, Belgium

The European Union Reference Laboratory for Heavy Metals in feed and food (EURL-HM) - hosted in Geel by the Joint Research Centre (JRC) of the European Commission – organised since 2006 a total of twenty five proficiency tests (PT) for EU National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs).

Setting independent assigned values, evaluating the measurement uncertainties reported, taking into account truncated values ("less than"), and addressing compliance are the main constituents of the rigorous approach implemented by the EURL-HM. Several other EURLs and PT providers are gradually implementing similar approaches.

This presentation will summarise what we learned from the PTs of the EURL-HM in the past decade.

DETERMINATION OF SIX ARSENIC SPECIES IN SEAFOOD

Agnieszka Nawrocka, Maciej Durkalec, Miroslawa Kmiecik, Andrzej Posyniak
Department of Pharmacology and Toxicology, National Veterinary Research Institute, Pulawy, Poland

Arsenic (As) is one of the most toxic elements naturally occurring in the Earth's crust and environmentally ubiquitous by natural and anthropogenic emissions. This element occurs in the environment in various chemical forms differing in toxicity (As(III)> As(V)> MMA> DMA> AsB> AsC> TMAO> arsenolipids> arsenosugars). Marine organisms, due to their sedentary or sessile lifestyle, are specifically exposed to arsenic through waterborne and dietary routes. This fact may pose a risk associated with the consumption of this type of food. Therefore, the speciation analysis is necessary for the assessment of the potential consumer's health risk associated with the seafood consumption.

The aim of this study was to develop an analytical method for the identification and quantification of six arsenic species: arsenite As(III), arsenate As(V), monomethylarsonic acid (MMA), dimethylarsinic acid (DMA), arsenobetaine (AsB), and arsenocholine (AsC) in seafood tissues. Speciation analysis was carried out by high performance liquid chromatography combined with inductively coupled plasma mass spectrometry (HPLC-ICP-MS) with an anion exchange column (Hamilton PRPX-100). The microwave assisted extraction was used as a preparation method of samples to analyse. Extraction efficiency was confirmed by determination of total arsenic in the obtained extracts carried out via ICP-MS.

The proposed method was validated and validation parameters (LoD, LoQ, repeatability, within-laboratory reproducibility and accuracy) were assessed. The expanded uncertainty of the measurement was estimated as well. Reliability of obtained results was verified by analysis of spiked samples and certified reference materials (CRMs).

Oral session 3

Chairperson
Laura Ciaralli

PLANNING OF 2018 PTs

Laura Ciaralli
European Union Reference Laboratory for Chemical Elements in Food of Animal Origin, EURL-CEFAO, Istituto Superiore di Sanità, Rome, Italy

Regulation (EC) No 882/2004, which will be replaced by Regulation (EU) No 2017/625, lists the tasks that the European Union Reference Laboratories (EURLs) have to take on. The organization of inter-laboratory comparative tests or Proficiency Testings (PTs) is considered a key point in the current Regulation as well as in the new one in which the importance of this activity is further stressed. As a consequence, each EURL is requested to include the management of PTs in its work programme. Obviously this activity, addressed to the National Reference Laboratories (NRLs), is performed by each EURL within its field of competence. The organization of PTs requires an accurate planning so as to provide participants with profitable exercises adequate to verify the performances of their analytical methods. In particular, the legislation in force as well as emerging problems should influence the selection of matrix/analytes combinations on which PTs are based. This aspect is especially considered in the activity of the EURL-CEFAO as PT Provider. In fact, as of 2005 it has organized 28 PTs devoted to its network taking into account both the needs/requests of the NRLs and the revision of the EU regulations.

The annual workshop is an important occasion for the EURL-CEFAO to collect the NRLs' requirements by administering a questionnaire. This is a simple but effective way to gather information on the network's preferences regarding matrices and analytes to be included in the exercises. The EURL-CEFAO evaluates the outcomes and takes them into account in drafting the work programme for the following year. However, in general terms the two annual PTs are planned considering not only the current legislation but also the entering in force of new Maximum Limits.

During the workshop, the exercises planned for 2018 will be presented, detailing the reason of the choice, the goal to be achieved and giving a very preliminary time scheduling.

On the basis of this information, the NRLs could plan their work and, if necessary, could select further commercial PTs that cover matrix/analytes not included in the 2018 EURL-CEFAO PTs.

THE NATIONAL FOCAL POINT OF THE EUROPEAN FOOD SAFETY AUTHORITY AND ITS ROLE IN THE RISK ASSESSMENT STRATEGY IN EU

Luca Busani, Camilla Marchiafava
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The Focal Points (FPs) act as an interface between the European Food Safety Authority (EFSA) and national food safety authorities, research institutes and other stakeholders. All 28 EU Member States, Iceland and Norway, as well as observers from Switzerland and EU candidate countries have EFSA FPs, that support scientific cooperation and networking activities between and among Member States and EFSA by:

- assisting in the exchange of scientific information and experts;
- advising on cooperation activities and scientific projects;
- promoting training in risk assessment;
- raising EFSA's scientific visibility and outreach in Member States.

Among the strategic activities that at EU level are promoted and supported by the FPs and EFSA, the European Risk Assessment agenda, developed within the 2020 EFSA's cooperation strategy summarized as "Working together and exchanging knowledge between food safety experts ensures excellence and efficiency and maximises Europe's risk assessment capacity and potential. We believe that the whole of food safety expertise is greater than the sum of its individual parts" is one of the key initiatives.

The agenda is the result of a framework process for initiating joint projects with Member States (and other partners) on topics that are agreed as being priorities. This is the basis of the EU Risk Assessment Agenda which will be on ongoing activity in the coming years.

The key partners of this and others initiatives described in the 2020 EFSA's cooperation strategy are of interest also for research institutes and laboratories, that can participate and promote their competences and expertise in EU.

HARMONIZATION OF COMPLIANCE ASSESSMENT FOR COMPOUND AND PROCESSED FOOD: IS IT AN ISSUE?

Andrea Colabucci
European Union Reference Laboratory for Chemical Elements in Food of Animal Origin, EURL-CEFAO, Istituto Superiore di Sanità, Rome, Italy

In order to guarantee that food and feed are safe and wholesome, the EU legislation foresees official controls at any stage of production. High quality and uniformity of analytical results are a key to ensure a high level of protection of consumers' health. To this aim, reference laboratories have been designed both at European Union and at national level (EURL and NRLs).

As for the field of interest of the EURL for chemical elements in food of animal origin (CEFAO), the Commission Regulation (EC) 333/2007 lays down the methods of sampling and analysis. In particular, the point D.2 states that the lot/sublot (object of the control) is accepted if the analytical result of the laboratory sample does not exceed the respective maximum level for the chemical element/matrix combinations laid down in the Annex of the Commission Regulation (EC) 1881/2006 taking into account the expanded measurement uncertainty and, when relevant, the correction of the result for recovery.

This rule can be applied in a uniform way when the laboratory sample exactly matches the element/matrix combinations reported in the Annex (e.g. cadmium in meat).

Anyway, official controls can be carried on more complex foods. What happens if the laboratory sample is composed of more than one ingredient that may have undergone an industrial process (i.e. drying)? How can be the compliance assessed in such cases?

Article 2 of the CR 1881/2006 states that, the changes of the concentration of the contaminant caused by the processing and the relative proportions of the ingredients in the product as well as the analytical limit of quantification are to be taken into account.

The scope of this presentation is to raise a discussion among workshop participants, to verify if a harmonized point of view can be reached.

26TH PROFICIENCY TESTING ON FREEZE-DRIED MEAT: STATISTICAL EVALUATIONS AND RESULTS

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Harmonization and improvement of the analytical performances of the National Reference Laboratories (NRLs) designated by the EU Member States are priority objectives of the EURL-CEFAO activity. The periodical repetition of Proficiency Testings (PTs) on the same matrix/analytes combination is a crucial tool to monitor the performances of the network over the years. In particular, it is crucial in case the laboratories modify the analytical techniques they use or new laboratories enter the network. According to this policy, the EURL-CEFAO based the 26th PT on the determination of cadmium (Cd), lead (Pb), total mercury (Hg) and copper (Cu) in freeze dried meat. Notably, for Cd and Pd, maximum levels in meat are set in the Commission Regulation 1881/2006, whilst Cu and Hg are frequently included in the EU Member States (MSs) annual National Residue Monitoring Plans (NRMPs). The same analytes/matrix combination was previously chosen for the 18th PT on frozen meat. This choice allowed the EURL-CEFAO to highlight possible variations of the participants' performances depending on the physical form of the sample.

All except one NRL belonging to the network participated to the 26th PT. In addition, four extra-network laboratories participated to the exercise.

The statistical evaluation of the results was performed in accordance with ISO 13528:2015. The data of Hg and Cd were normally distributed and the datasets resulted roughly symmetric and unimodal. For Pb, dataset did not have a normal distribution due to the presence of two outliers. Finally, the distribution of Cu results was neither normal or unimodal. However, the observed minor mode was hardly significant as it was due to a single result. The robust mean was set as the assigned value for all the analytes except for Hg. In this case, the indicators of central tendency (mean, median and mean) coincided and the mean was selected as the assigned value, considering the data distribution and the consistence of the indicators of central tendency. The standard deviations for proficiency assessment (σpt) were calculated using the specific equations developed by the EURL-CEFAO, which provide values lower than those calculated using Horwitz/Thompson equation. This choice is based on the fact that the performances of the NRLs are expected to be better than those of routine control laboratories. The general performance of the network was confirmed to be satisfactory for Cd, Cu and Pb. This is an important outcome, mainly for Pb, taking into account the physical form of the material (freeze-dried meat) and the intrinsic difficulties of the Pb determination (e.g., overestimation due to contamination). The worst performance was obtained for Hg (two censored results, two unsatisfactory and two questionable z-scores), but this was probably due to the low concentration value of this analyte in the freeze-dried sample.

The statistical evaluation carried out on the 26th PT data will be illustrated and the performances of the current and the previous PTs on the same matrix (15th PT on freeze-dried meat and 18th PT on frozen meat) will be compared. As for Cd and Pb, participants were also requested to state the sample compliance taking into account the EU legislation in force and, therefore, the relevant outcome will be discussed.

THE EURL-CEFAO AS PROFICIENCY TESTING PROVIDER: ACTIVITY SURVEY

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The European Union Reference Laboratory for Chemical Elements in Food of Animal Origin (EURL-CEFAO) started its activity as organizer of Proficiency Testings (PTs) in the mid-'90s. In that time, the availability of commercial PTs and Certified Reference Materials (CRMs) containing chemical elements in food matrices was scarce. Therefore, the organization of interlaboratory comparisons to evaluate the performance of the National Reference Laboratories (NRLs) as well as the production of materials to be used as reference for internal quality control were key issues.

The EURL-CEFAO, formerly CRL-ISS, has invested time and resources in this activity since the beginning. The very first exercises were only explorative as the participants were requested to quantify the concentration of chemical elements in acid aqueous solutions and digestion-simulating samples. After ascertaining the ability of the NRLs to analyse simple solutions the subsequent PTs were based on real samples (meat, fish and honey). This activity significantly boosted in 2010 when the EURL-CEFAO was accredited as PT provider according to ISO-Guide 43-1 and subsequently according to ISO/IEC 17043:2010. Over the years, specific procedures have been implemented to produce materials that were stable and homogeneous taking into account the physical form of the final samples (freeze-dried; liquid; frozen). A lot of preliminary batches have been prepared so as to overcome any possible drawbacks occurring during the preparation of the PT material. In the period from 2005 to 2017, twenty-eight PTs have been organized and roughly 4000 PT items have been produced.

As for the procedure of statistical elaboration of the participants results, it has been continuously updated and improved. In particular, specific algorithms for deriving the standard deviations for proficiency assessment were set on the basis of the analyte/concentration/matrix combination. Over the years, these equations have been modified and made stricter considering the good performance obtained by the network. The significant improvement of the NRLs' performance can be easily deduced from the control charts of z-scores that have been updated after each PT. In the last five years new matrices (*e.g.* infant formula; honey) and analytes (*e.g.* Mo, Ni) of particular interest to the network have been included in the EURL-CEFAO PTs.

An overview of this activity will be presented pointing out the improvement of the network in terms of performance, data dispersion and statement of sample compliance. Some preliminary information on the results of the 27th PT will be given as well.

Poster Contributions

P01 NICKEL IN BIVALVE MOLLUSCS PRODUCED IN PORTUGAL: AN EVALUATION OF PORTUGUESE CONSUMER EXPOSURE

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Recently, the Hellenic Food Authority asked the European Food Safety Authority (EFSA) for an assessment of the risks to human health posed by the presence of nickel (Ni) in food, including bivalve molluscs. However, the data collected by this entity are still insufficient in order to adopt any risk management measures. Accordingly, the European Union recommendation (EU 2016/1111) on the monitoring of nickel in food has been published.

The main objective of this work was to collect data on levels of Ni in bivalve molluscs produced in Portugal so as to evaluate the risk associated with their consumption taking into account the tolerable daily intake for adults of 2.8 ug Ni/kg body weight per day (EFSA, 2015).

All data were obtained from the analysis of bivalve molluscs harvested between 2014 and 2016 within the scope of IPMA's National Bivalve Mollusc System (SNMB), in several production areas in Portugal, both coastal and estuarine-lagoons. In this context, levels of Ni were determined by flame atomic absorption spectrometry, based on the methodology described by L. Jorhem (2000).

As for the results, median levels of Ni ranged from 0.08 to 1.7 mg/kg and the highest values were recorded in samples of Japanese clams (2.9 mg/kg) and cockles (2.5 mg/kg). Considering the maximum values of Ni obtained for the various bivalves analyzed and considering a meal of 100g per day, and a person of 69 kg (mean body weight of the Portuguese citizen) the tolerable daily intake of Ni is achieved when some samples of cockle, Japanese clam and blue mussel are consumed.

In conclusion, this study seems to indicate some risk in the consumption of bivalves produced in some areas of Portugal as the tolerable daily intake of Ni is sometimes exceeded. However, it would be unlikely for a person to consume the mentioned amount of these species per day, since the consumption of bivalves in Portugal is usually considered as a snack or as an entree and not a main course. Therefore, the values obtained can be considered overestimated and its consumption does not guide to any concerns.

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P02 BIOMONITORING OF ELEMENTS IN THE MUSSEL MYTILUS GALLOPROVINICIALIS FROM HARVESTING AREAS IN SLOVENIA

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The concentration of some trace elements (Zn, As, Mn, Cu, Cr, Ni, Co, Cd, Pb, Hg and Fe) was determined in the mussel *Mytilus galloprovinicialis* from harvesting areas in Slovenia- The study was conducted so as to determine differences among sites, seasonal variations and to examine the potential risks for human health. The Slovenian Sea is part of the Gulf of Trieste, the northern part of the Adriatic Sea. It is affected by urban and industrial pollution as well as by tourism. Mussels are so-called filter-feeders and for this reason they are often used as bioindicator of environmental pollution. Mussels (*Mytilus galloprovincialis*) were collected once per month during the period between January and October 2015 from three Slovenian shellfish harvesting areas (at Seča, Strunjan and Debeli Rtič) and from one location of wild Mediterranean mussels (at the lighthouse near Debeli Rtič). Ten mussels with similar length were selected from each sampling location. Mussels were cleaned and washed with deionized water. The length and wet weight of the independent shells was measured. The whole muscle tissues of ten mussels were pooled together, homogenized and digested in a microwave oven. The concentrations of elements were determined with Varian 820-MS ICP-MS. External calibration curve was used. The following internal standards were used to correct the spectral interferences: Sc for Cr, Mn, Fe, Co, Ni, Cu, As and Zn; In for Cd; Bi for Hg and Pb . To eliminate polyatomic interferences, Collision Reaction Interface (CRI) was used for measurement of ^{75}As. The accuracy of measurements was tested by using Community Bureau of Reference (BCR) certified reference material (ERM - CE278k mussel tissue). Two-sides ANOVA was used to test the influence of season and location on the element concentration in mussel. The level of significance was set at $\alpha=0.05$. The mussels from all investigated samples contained the highest amount of Fe and the content of other elements decrease in following order: Zn>As>Mn>Cu>Cr>Ni>Co>Cd>Pb>Hg. Statistical analysis revealed that the time of sampling significantly affects the concentrations of Cr, Mn, Fe, Ni, Hg and Pb in *M. galloprovincialis*. The concentration of these elements was the highest between January and March and the lowest between May and September. However, sampling time had no significant effect on the Zn, Co, Cu and Cd concentration in mussels. Slightly higher or lower concentration of these elements was observed, but these changes are unlikely to be related to seasonal variations. Thus, the concentration of chromium was slightly higher in May, especially in mussels from Strunjan, and the concentration of As a little lower in January. The lowest level of elements was determined at Seča. The highest levels of Hg and Pb were determined in mussels from the Debeli rtič. The higher mercury content in this area is associated with a higher content of Hg in sediment. This part of Adriatic Sea is influenced by the natural and anthropogenic load of Hg from polluted River Soča (Isonzo), the watershed of which contains the world's second largest Hg mine in Idrija, which is

closed since 1988. Nevertheless, mussels were suitable for human consumption and in accordance with Commission Regulation No 1881/2006 since all samples contained less mercury than 0.50 mg/kg of wet weight, less Cd than 1.0 mg/kg and less Pb than 1.5 mg/kg of wet weight.

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P03 ANALYSIS AND RISK CHARACTERIZATION OF ARSENIC SPECIES IN FOOD SUPPLEMENTS BASED ON CLAY

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Fifteen clay-containing Food Supplements (FS), which contain also natural ingredients such as plants, were collected on the Belgian market and analyzed for total arsenic (Astot), inorganic Arsenic (i-As), AB (Arsenobetaine), DMA (Dimethyl Arsenate) and MA (Monomethyl Arsenate). Arsenic (As) speciation analysis was conducted by HPLC-ICP-MS, by means of a method which is commonly used for As speciation analysis in food (cfr method CEN:16802; further referred to as "food-method"). In addition, the bioaccessible As fraction in these samples was determined by use of the Unified Barge Model (UBM) protocol, an in vitro simulation of the gastro-intestinal tract. Total As in the samples was analyzed by ICP-MS.

Clay-containing FS form a particular group of FS in relation to potential As toxicity, because a large fraction of arsenic in these samples is expected to be present in the most toxic inorganic form (i-As). In terms of risk, the most important question is not which species are present, but rather what is the bioaccessibility of i-As. The results revealed that bioaccessibility of i-As varied between 7.7% and 50.9 % among all clay-containing samples, and that -overall- the food method was only a poor predictor of the bioaccessible i-As fraction despite the significant relationship.

In a second step an exposure and risk assessment of As for FS consumers was performed. Exposure was calculated for each FS by multiplying the concentration of these compounds with the maximal recommended dose. Risks related to the intake of As species were evaluated by comparing the calculated exposure to the reference values suggested by ATSDR (2007), JECFA (2011) and EFSA (2009). If the reference value was a MRL-value, the conclusion 'concern' was given if the calculated exposure was higher than the MRL value. If the reference value was a BMDL-value, then a 'Margin-of-exposure' (MOE=BDML/exposure) was calculated and the conclusion 'concern' was given when the MOE<100. For Asi exposure we worked with the assumption 'AsTot = total inorganic As'; and with the bioaccessible As. Regarding MA and DMA no (sub)chronic risk was present, and no risk for acute toxicity of i-As was detected.

We concluded that intake of bioaccessible i-As due to chronic consumption of clay-containing food supplements (intended for oral use) is of concern for the general population in case of 43% of the tested food supplements, while for high risk groups it is of concern for 71% of the tested food supplements.

P04 VALIDATION OF A SIMPLE AND RAPID METHOD FOR TOXIC ELEMENTS DETERMINATION IN RAW MILK BY ICP-MS

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The MERCOSUL regulation n. 12/2011 sets 50, 50 and 20 µg kg-1 as the Maximum Levels (MLs) for Arsenic (As), Cadmium (Cd) and Lead (Pb) in milk, respectively. Therefore, the development of analytical methods to quantify low concentration values of these contaminants in milk is of critical importance. Inductively coupled plasma mass spectrometry (ICP-MS) has become accepted as the most powerful analytical tool for the determination of trace elements due to its high sensitivity and selectivity.

Alternatively to the conventional microwave sample preparation, procedures using bases (*e.g.* tetramethylammonium hydroxide) and acids (*e.g.* formic acid), have been reported to solubilize biological samples. The solubilization of milk with formic acid improves the sensitivity of the method by enhancing the ionization of the analytes with minimal handling and low time consumption.

Furthermore, this procedure minimizes the possible sample contamination caused by the reuse of the digester's vessels (a crucial point on traces analyses), The aim of this study was to validate a simple dilute-and-shoot method for the determination of As, Cd and Pb in milk by ICP-MS.

Samples of raw milk were solubilized with formic acid in an ultrasonic bath. Quantification of the analytes in milk samples was performed by ICP-MS (Agilent's 7700x) and was based onthe following isotopes: 75As, 111Cd and 208Pb (as sum of 206, 207 and 208) . Additionally, germanium and rhodium were used as internal standards. The following validation parameters were assessed : linearity range, specificity, quantification limit (LoQ), repeatability, within-laboratory reproducibility and recuperation accuracy by recovery rates. Regression curves (0.5 to 10.0 µg L-1 range) showed good linear correlation coefficients ($R > 0.999$) for all the tested elements.

As for specificity , no matrix effects were observed. The LoQs obtained for As, Cd and Pb were 5.0, 5.0 and 7.5 µg kg-1, respectively; at these levels of addition, the recoveries ranged from 89% to 97%. The following results were obtained for repeatability (n=14) : As, 3.1% and 3.4%; Cd, 2.5% and 1.3%; Pb 6.7% and 8.6% at low and high levels of analyte's spike, respectively. The results for within-laboratory reproducibility (n=35) were: As, 3.2% and 3.4%; Cd, 2.9% and 2.4%; Pb, 8.2% and 7.0% at low and high levels of spike, respectively. Recoveries for all elements ranged from 95% to 100%. After the validation process, our laboratory took part in the 25th Proficiency Testing on determination of As, Cd and Pb in milk organized by the European Union Reference Laboratory for Chemical Elements in Food of Animal Origin

(EURL-CEFAO) in order to verify the performance of the method. The z-scores obtained were satisfactory for all analytes (As, - 0.2; Cd, - 0.4; Pb, - 0.6) proving the adequacy of the method.

The developed method for determination of As, Cd and Pb in raw milk has been shown to be accurate and reproducible, also reaching quantification limits that meet international regulations. Furthermore, the satisfactory results in the proficiency testing validated the use of this method by the Brazilian Ministry of Agriculture, Livestock and Food Supply for official control of these contaminants in milk.

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P05 THE ITALIAN NATIONAL REFERENCE LABORATORY FOR HEAVY METALS IN FOOD: ACHIEVEMENTS AND FUTURE DEVELOPMENTS

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The Istituto Superiore di Sanità, Department of Food safety, nutrition and veterinary public health, was designated as the National Reference Laboratory (NRL) for heavy metals in food by the Italian Minister of Health on the 16th of February 2010, to comply with the tasks established by art. 33 of Regulation EC 882/2004. The activities carried out over seven years will be presented.

The NRL implemented and accredited analytical methods for the determination of the heavy metals in food for which maximum limits are set by Regulation (EC) 1881/2006 and demonstrated its competence through the successful participation in the comparative tests organised by the EURL. With respect to the coordination of the OCLs, the NRL organised inter-laboratory comparisons covering a range of matrices, with assigned values traceable to the Network of the NRLs. To this aim, a dedicated application was developed, allowing on-line registration of participants, electronic transmission of results and related information and distribution of final reports on the test. As part of these exercises, details of the analytical techniques applied and their performance characteristics were collected to verify that the requirements of Regulation (EC) 333/2007 were satisfied. The exercises also included an assessment of the interpretation of the results in terms of compliance with limits. To ensure the dissemination of information to both the competent authorities and the OCLs, the NRL set up a dedicated website, regularly updated with the relevant information, and organised a series of annual workshops to present and discuss new information and criticalities. Strict collaboration is in place with the Ministry of Health and advice was provided on a number of technical issues.

Following the recommendations to monitor the levels of inorganic arsenic and nickel in food, current and future developments are devoted to provide support to the OCLs and the competent authorities on best practices for sampling, analytical measurements and interpretation of results for these elements.

P06 SELENIUM LEVELS IN CATTLE AND PIG IN FINNISH SELENIUM MONITORING PROGRAMME

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Due to an extremely low Selenium (Se) intake in the 1970s in Finland (0.025 mg/day), a decision was made in 1984 to supplement multinutrient fertilizers with Se in the chemical form of sodium selenate. Almost all fertilizers used in Finland since 1985 have been fortified with Se.

Soils in Finland are not exceptionally poor in Se, but the soil and climatic conditions depress Se uptake by plants. The pH of soils is relatively low, the prevailing temperatures are also low and the high humidity, characteristics of the climate in Finland, results that selenium in soils is stored in reduced forms, which are poorly available to plants.

Since 1985 the Se content of fertilizers has been revised three times (1990, 1998, and 2007). The latest amendment was necessary due to the decreasing use of fertilizers. The revision in 2007 from 10 to 15 mg/kg fertilizers has slightly increased the Secontent of foods and feeds. Currently all crop fertilizers contain 15 mg Se/kg. Finland is the only country in the world where the Se supplementation of fertilizers is in use.

The effect of Se supplementation of fertilizers on Se concentration in cattle and pig has been very clear. In 1985 Se concentrations (wet weight) in meat and liver of cattle were 0.07 mg/kg and 0.28 mg/kg. The concentrations in 2016 were 0.17 mg/kg and 0.52 mg/kg, respectively. In 1985 selenium concentrations in meat and liver of pig were 0.08 mg/kg and 0.49 mg/kg and in 2016 the concentrations were 0.22 mg/kg and 0,63 mg/kg.

The selenium supplementation of fertilizers has been an effective, safe and economical way to increase the selenium intake of humans and food producing animals. The activity has been controlled by systematic follow-up studies focused to main agricultural products and human serum levels. The average daily Se intake has been about 0.08 mg/day in recent years, which meet well the recommendations and is considered adequate and safe level.

This work reports in detail the level of Se in liver and mussel of cattle and pig in more than thirty-year monitoring activity.

P07 NATIONAL REFERENCE LABORATORY FOR HEAVY METALS AT VETERINARY AND FOOD INSTITUTE IN KOŠICE

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The Unit of Ecotoxicology, Testing and Monitoring of Foreign Substances at Veterinary and Food Institute in Košice (VFI) is the National Reference Laboratory for heavy metals in food and feed of Slovak Republic. Heavy metals are determined using ICP-MS Agilent 7500, 7900 and direct mercury analyzer. All samples are digested in microwave digestion system Berghof speedwave ENTRY. Reaccreditation on VFI according to general requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005) by Slovak National Accreditation Service in 2016 was succesfully processed. VFI regularly organizes proficiency tests and collaborates with partner Institutes in Dolný Kubín and Bratislava and Regional Public Health Authorities and other testing laboratories in Slovak Republic. VFI also collaborates with Faculty of Science at Pavol Jozef Šafárik University in Košice and University of Veterinarny Medicine and Pharmacy in Košice.

Every year VFI usually determines lead, cadmium, arsenic, mercury and nickel in approximately one thousand samples of food and feed which are being sent from the state sector - monitoring of game and fish, national program for residue control, general controls, target controls during Easter and Christmas periods and samples from private owners.

VFI was involved in state target control of methylmercury in twenty four samples of frozen marine fish from supermarkets in Slovak Republic in 2016.

Seventy three samples originating from monitoring of game and fish were analyzed during year 2016. VFI is involved in national monitoring of mercury in fresh-water fish from rivers and lakes which are attractive for tourists and fishermen in the eastern and central part of Slovak Republic. In particular, lakes are monitored in areas where, in the past (1870), industries were located in which mercury was a secondary product from copper and silver mining. Mercury concentration in three samples from Lake Ružín (tourist attraction/dam) with mining history and one sample from lake Liptovská Mara (tourist attraction/dam) was not compliant with Regulation (EC) No. 1881/2006.

P08 ASSESSING THE LEVELS OF NICKEL IN FOOD: A NEW CHALLENGE FOR THE OFFICIAL CONTROL LABORATORIES

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Recently, the European Food Safety Authority (EFSA) issued a scientific opinion on the risks to human health arising from the presence of nickel (Ni) in foodstuffs and drinking water. Based on the examination of data on reproductive and developmental toxicity in experimental animals, a tolerable daily intake of 2.8 mg Ni / kg body weight was identified, which should be further reduced in case of individuals susceptible to allergic reactions. EU legislation sets no limit for the content of Ni in foods, whereas the level of Ni in drinking water intended for human consumption and in natural mineral waters must not exceed 20 μg/L.

At present, the available data are not representative of the presence of Ni in food throughout the EU (80% of data comes from Germany). In addition, the occurrence data for food groups considered as the main source of Ni exposure are limited. Therefore, in July 2016, the Commission and the European Union issued Recommendation (EU) 2016/1111 on the monitoring of the presence of Ni in food, to be carried out in 2016, 2017 and 2018 in order to build an objective framework on the Ni content in food products in view of possible future risk management measures. The monitoring activities concern a variety of food products of vegetable origin and also bivalve molluscs.

The measures envisaged in the Recommendation in particular what is required in terms of sampling and analysis, were presented to the Italian OCLs at the last annual workshop in December 2016. Furthermore, to support the implementation of new analytical methods and data quality assurance, a comparative test for the determination of Ni in freeze-dried mussels was launched. The test material, prepared by the EURL-CEFAO as part of the EURL-CEFAO proficiency testing activities, had not been spiked with Ni, thus providing an example of naturally occurring Ni levels, but neither tested for Ni. However, according to ISO 13528:2015, the availability of homogeneity data for other, more critical elements (As, Cd, Hg and Pb) was deemed satisfactory to propose the material as a test item in this exercise. Ten Italian OCLs participated in this exercise using methods of their choice. Assigned values for Ni were calculated using Algorithm A. Analytical performance was assessed by z-scores using the standard deviation for proficiency assessment (σp) derived from the Horwitz equation. All laboratories obtained a satisfactory z-score confirming their capability to deal with the task of providing reliable analytical results on the content of Ni in bivalve molluscs.

P09 RESULTS OF THE ITALIAN PROFICIENCY TEST ON PESTICIDE RESIDUES IN PULPED TOMATOES

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Pesticides are a broad class of bioactive compounds used in agriculture, food preservation, and human health. They differ from other chemical substances because they are spread deliberately into the environment. In Europe, it is necessary to perform a risk assessment process before a pesticide can be used on a food crop . The risks associated with the use of pesticides are controlled by carrying on monitoring plans for pesticide residues in foodstuff, in each Member States by a network of official laboratories. Current EU legislation on pesticides in and on food requires the official laboratory participation in specific proficiency tests, particularly in those organized by the Reference Laboratories.

In 2015 the National Reference Laboratory for pesticide residues in fruit and vegetables organized a Proficiency Test in pulped tomatoes. The participants in this PT are Italian official control laboratories active in the analysis of pesticide residues in fruit and vegetables and involved in the National and European monitoring programs. Twenty one laboratories participated to the PT. The exercise consisted in the determination of seven different pesticides in a pulped tomato sample spiked with a specific level of concentration (> 0.1 mg/kg). These pesticides were chosen from a list of forty-six pesticide residues.

To assess the performance of the participating laboratories, z-scores were used. In this PT the assigned value for each pesticide present in the sample was the median. A fit-for-purpose relative target standard deviation (FFP-RSD) of 25% was chosen to calculate the target standard deviation. Furthermore, the Robust Standard Deviation (Robust RSD) in accordance with the ISO 13528 was estimated.

Information of analytical methodologies used was also requested to the participants. Thus, the effects on the results using different analytical procedures were investigated. The results and information received from the participants have provided indications on satisfactory and unsatisfactory performance and potential analytical problems.

P10 ARSENIC SPECIATION ANALYSIS OF VEGETABLE SAMPLES WITHIN THE "SAFE & SMART" PROJECT

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Safe & Smart Project “New enabling technologies for food safety and integrity of agrifood supply chains in a global scenario” (National Technology Cluster Agrifood D.D. 257/Ric and D.D. 414/Ric 2012 MIUR - 2014-ongoing) has the general objective of improving food safety through prevention, control, technological innovation and training actions. It aims at promoting materials and products with low content of chemical and biological contaminants or other undesired substances, while guiding the agrifood system towards an advanced, integrated scenario of systems for risk prevention and rapid diagnosis of contaminants. The challenge is to turn food safety from a problem into an element of competitiveness and development for the agri-food industry, with substantial spillover benefits for other industrial and technological service sectors. The project foresees research activities in the two areas of “Diagnostic” and “Prevention” Particularly concerning the “Prevention” side, the project foresees - among others - the specific objective of developing methodologies for qualifying the agroecosystem of primary production with respect to the risk of transferring environmental contaminants to the food chain. In this frame, a specific activity is devoted to: i) evaluation of available methods for studying toxic element speciation with a preliminary study on techniques for Arsenic (As) extraction and speciation on vegetable products; ii) development of a method for As speciation in vegetable matrixes and application to samples collected in different geographic areas (subjected or not to different contamination sources). To this purpose, a method for speciation and quantification of inorganic As (i-As) was developed and validated. The samples of vegetal origin underwent a microwave assisted digestion and an extraction in a water bath with the mixture HNO3/H2O2.

Preliminary to the speciation analysis, the total As concentration was assessed in both the digested and extracted samples by Inductively Coupled Plasma Spectrometry (ICP-MS) in Kinetic Energy Discriminator mode (KED) and the quantifiable results ranged from 5 µg/kg to 430 µg/kg.

As for the speciation, the method was based on the complete oxidation of As(III) into As(V) and on the determination of i-As, measured as As(V), by means of High Performance Liquid Chromatography ICP-MS (HPLC-ICP-MS) using an anion exchange column (PRP-X100) and the following eluting mixture: 10 mM NH4H2PO4, 10mM NH4NO3 2% (v/v) CH3OH, pH 5.5 adjusted with aqueous NH4OH. Seven different types of vegetable, harvested in different areas, were analysed and the i-As content ranged from <LoQ (5.0 µg/kg) in fennel to 372 µg/kg in chard. The order of

concentration of i-As in the samples subject of this study was: fennel < aubergine (area CS) = courgette < broccoli = chilli pepper < cardoon < aubergine (area GR) < chard (area VP) < chard (area CAS).

P11 THE MATERIAL FOR THE 23RD EURL-CEFAO PROFICIENCY TEST ON FREEZE DRIED FISH: FROM THE PLANNING TO THE DISTRIBUTION OF THE TEST ITEMS

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Since 2005, the European Union Reference Laboratory for Chemical Elements in Food of Animal Origin (EURL-CEFAO) organizes proficiency tests (PTs) in its field of competence, with the aim to harmonize the quality of the results produced by the EU National Reference Laboratories (NRLs) belonging to its network. To make the exercises the most profitable as possible, ad-hoc PT materials are prepared in-house. Special care is given to adjust the mass fractions of the analytes to values of interest, taking into account both legal and analytical requirements. This poster describes the preparation of the freeze dried fish sample for the 23th PT, from the choice of the matrix up to the distribution of the PT test items. As for the analytes, cadmium, mercury and lead were chosen being maximum levels set in the relevant legislation (Commission Regulation 1881/2006); arsenic was also included since the analysis of this element in fish is routinely performed by laboratories.

In order to adjust the analyte concentrations, the mixing of different fish as well as the spiking of inorganic solutions were considered. Therefore, after exploring the natural content of the four elements in some species, a preliminary lot was prepared mixing a sea fish (swordfish: As, Hg ~1 mg/kg, Cd ~ 0.2, Pb < LoQ) and a fresh water fish (pangasius < LoQ for all the elements). This preliminary lot was prepared to evaluate if a proper homogenization among the fish could be achieved, notwithstanding the different tissue structure. An appropriate amount of water was added to facilitate the amalgamation; swordfish, pangasius and a mix of the two species were then lyophilized to assess the yield of the process; all this information was used to prepare the final batch.

In order to achieve a proper amount of Cd, Hg and As, 30 parts of the fresh-water fish were mixed with one part of swordfish. As for Pb, before the mixing, a spiked solution was added to the swordfish to obtain the planned value. After the freeze-drying process, the sample was gathered, sieved, quartered, bottled, and labelled. The ninety-five items produced were then γ-irradiated to enhance the material shelf-life. The sufficient homogeneity of the sample was verified analyzing ten bottles in duplicate by Inductively Coupled Plasma Mass Spectrometry (ICP-MS for As, Cd and Pb) and by Cold Vapour Atomic Absorption Spectrometry (CV-AAS for Hg).

The results show the achievement of the target values for all the elements; moreover, congruity of data can be also demonstrated by comparing EURL data with the assigned values obtained by consensus from the PT participants. The outcome of the exercise shows the fitness of the procedure and the capability of the EURL to obtain the planned concentrations.

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