European Commission

JRC SCIENTIFIC AND POLICY REPORTS

IMEP-117: Determination of total As, Cd, Pb, and Hg in compound feed

Interlaboratory Comparison Report

Ioannis Fiamegkos, Fernando Cordeiro, Piotr Robouch, Håkan Emteborg, Hanne Leys Aneta Cizek-Stroh, Beatriz de la Calle

October 2013

Report EUR 26195 EN

Joint Research Centre

European Commission
Joint Research Centre
Institute for Reference Materials and Measurements

Contact information
Beatriz de la Calle
Address: Joint Research Centre, Joint Research Centre, Retieseweg 111 2440, Geel, Belgium
E-mail: Maria.De-La-Calle@ec.europa.eu
Tel.: +32 (0) 1457 1252
Fax: +32 (0) 1457 1865

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

JRC85128

EUR 26195 EN

ISBN 978-92-79-33385-9 (pdf)

ISSN 1831-9424 (online)

doi:10.2787/80103

Printed in Belgium

IMEP-117: Determination of total As, Cd, Pb, and Hg in compound feed

Interlaboratory Comparison Report

October 2013

Ioannis Fiamegkos (a)

Fernando Cordeiro (c)

Piotr Robouch (c)

Håkan Emteborg (c)

Hanne Leys (c)

Aneta Cizek-Stroh (d)

Beatriz de la Calle (b,c)

(a) ILC coordinator, (b) IMEP programme coordinator,
(c) technical / scientific support, (d) logistic support

Table of Contents

Executive summary

The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre, a Directorate General of the European Commission, operates the European Union Reference Laboratory for Heavy Metals in Feed and Food (EURL-HM). One of its core tasks is to organize proficiency tests (PTs) among appointed National Reference Laboratories. This report presents the results of a PT, IMEP-117 of the EURL-HM focussing on the determination of total As, Cd, Pb and Hg in compound feed in support to Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed.

Thirty participants from 27 countries registered to the exercise. Only one participant did not report results.

The test material used in this exercise was a commercially available compound feed for cats which was spiked after the appropriate processing, bottled, labelled, and dispatched to the participants on the 23rd of May 2013. Three laboratories with demonstrated experience in the field provided results to establish the assigned values (X_{ref}). The standard uncertainties associated to the assigned values (u_{ref}) were calculated according to ISO/IEC Guide 98:2008 (GUM), ISO 13528:2005 and ISO Guide 35.

Laboratory results were rated with *z*- and zeta (*ζ*-) scores in accordance with ISO 13528. The z-score compares the participant's deviation from the reference value with the target standard deviation for proficiency assessment (σ_p), while the ζ- score states whether the laboratory's result agrees with the assigned value within the respective uncertainty. The standard deviation for proficiency assessment (σ_p), also called target standard deviation, was set to 10 % of the assigned value, for the measurands investigated.

The percentage of satisfactory z-scores was above 79 % for all measurands showing an overall adequate performance for European National Reference Laboratories assuring compliance towards the European legislation related to the determination of the investigated compound feed contaminants.

1 Introduction

The IMEP-117 exercise was carried out by the EURL-HM to assess the performance of National Reference Laboratories (NRLs) in the determination of total arsenic, cadmium, lead and mercury in compound feed. In parallel to IMEP-117, another PT, IMEP-38 was organised using the same test material, in which official control laboratories (OCLs) were allowed to participate. The results submitted to IMEP-38 are not discussed in this report.

IMEP-117 was requested by the Directorate General for Health and Consumers (DG SANCO).

Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed [1], describes as "*compound feedingstuffs*" the "*mixtures of feed materials, whether or not containing additives, which are intended for oral animal feeding as complete or complementary feedingstuffs*". The Directive and its amendments [1] set maximum levels for undesirable substances in animal feed (organic and inorganic). Regarding heavy metals, limits are set only for mercury (0.1 mg kg^{-1}) with the exception of mineral feed (0.2 mg kg^{-1}), compound feed for fish (0.2 mg kg^{-1}) and compound feed for dogs, cats and fur animals (0.3 mg kg^{-1}).

The screening of the selected material for this exercise (cat feed) revealed very low or no naturally incurred heavy metals and thus a spiking approach was choosen. As a result the test material distributed to the participants was not compliant with the legislation.

This report summarises and evaluates the outcome of IMEP-117.

2 Scope and aim

As stated in Regulation (EC) N° 882/2004 [2], one of the core duties of the EURL-HM is to organise interlaboratory comparisons for the benefit of NRLs. The scope of this PT was to assess the competence of the appointed NRLs to determine the total concentration of As, Cd, Pb and Hg in compound feed thereby providing a means of analytical quality assurance for the Member States.

The assessment of the measurement results was performed on the basis of requirements laid down in legislation [1], and follows the administrative procedure and the logistics of the International Measurement Evaluation Program (IMEP) of the IRMM.

IMEP is accredited according to ISO 17043:2010 [3]. The designation of this PT is IMEP-117

3. Set up of the exercise

3.1 Time Frame

The PT was agreed upon by the EURL-HM and the Directorate General for Health and Consumers (DG SANCO) when preparing the work program of the EURL-HM for 2013. Invitation letters were sent to participants on the 4th of April 2013 (Annex 1) and a web announcement (Annex 2) for the exercise was made on the IMEP webpage on the same day. The registration deadline was the 15th of May. The reporting deadline was set to the 30th of June 2013. Dispatch was followed by the PT coordinator using the messenger's parcel tracking system on the internet.

3.2 Distribution

Samples were dispatched to the participants by IRMM on 23rd of May 2013. Each participant received:

a) One bottle containing approximately 20 g of powdered compound feed.
b) An accompanying letter (Annex 3).
c) A "Confirmation of Receipt" form to be sent back to IRMM after receipt of the test material (Annex 4).

3.3 Instructions to participants

Concrete instructions were given to all participants in the above mentioned letter accompanying the test material. The measurands and matrix were defined as "Total As, Cd, Pb and Hg in compound Feed" following Directive 2002/32/EC on undesirable substances in animal feed".

Laboratories were asked to perform two or three independent measurements and to report the mean, the associated expanded uncertainty, the coverage factor of the associated expanded uncertainty and the technique used to perform the measurements. The measurement results were to be corrected for (i) recovery and (ii) moisture, the latter following the procedure described in the sample accompanying letter. Participants were asked to follow their routine procedures for the analysis and to report results in the same way (e.g. number of significant figures) as they would report to their customers. Likewise they were asked to calculate the moisture content of the test material using the recipe provided in the accompanying letter and to report all data based on dry-mass.

The results were to be reported in a dedicated on-line form for which each participant received an individual access code. A questionnaire was attached to this on-line form (Annex 5).

The laboratory codes were given randomly and communicated to each participant by e-mail. The assigned values were disclosed to participants during the 8th EURL-HM Workshop that was held in Brussels on the 24th of September 2013.

4 Test material

4.1 Preparation

The material used as test item was a commercially available feed purchased at the local market in Belgium. The composition reported on the label by the producer is indicated hereafter between brackets:

> Cereals, vegetable proteins, meat and animal sub-products, vegetable sub-products, oil and fats, fish and fish sub-products, yeast, minerals, vegetables.
> Nutritional additives in UI Kg^{-1}: Vit. A (12500), Vit. D3 (1000)
> in mg Kg^{-1}: Fe (48), I (1.5), Cu (9), Mn(5.1), Zn(67), Se(0.1)
> Analytical components: proteins (34.0 %), fat (8.0 %), ash (7.0 %), ash (7.0 %), fibers (4.0 %)

Two bags (4 kg each) of the granular compound feed (cat-food), were emptied in two stainless steel drums which were thereafter immersed in liquid N_2 to cool down the material prior to cryogenic milling. An all-titanium vibrating cryogenic mill was then used to mill the material (Palla VM-KT, Humboldt-Wedag, Köln, Germany).

After milling at temperatures between -196 to -100 °C the material was pre-cooled again and sieved over a 250 µm stainless steel sieve (Russel Finex, London, United Kingdom). Cold sieving was achieved under gentle flow of liquid N_2 to avoid clogging. The resulting powder (7.8 kg, < 250 µm) was placed in an 80 L stainless steel drum in which 32.5 L of tap water were added. The slurry was then mixed, homogenized and spiked with Pb, Hg, As and Cd standard solutions. Pure concentrated standards (Merck, 1000 mg/l ICP standards) with a certified concentration and associated uncertainty were used to obtain the following theoretical concentrations in the final material: 2.36, 0.76, 5.08 and 0.79 mg $kg^{-1}$of As, Cd, Pb and Hg, respectively. The recipient in which the spike was contained was rinsed once with tap water and added to the slurry to ensure a quantitative transfer. The spiked slurry was stirred for 2 hours using an IKA (Janke- Kunkel, Staufen, Germany) stirrer for further homogenisation.

Approximately 1 L of slurry per tray was placed on the freeze drying trays, (31 trays in total) and placed at -20 °C in a freeze cell over-night. After freeze drying the material was found to be sufficiently dry for the next steps (1.13 ± 0.17 % m/m for n = 2) as measured by Karl Fischer titration (KFT).

The dried slurry formed hard cakes on the trays which were crushed using a Teflon pestle inside a plastic drum. Teflon balls were then added to the drum placed in a 3-dimensional mixer for 1 h (Dynamix CM-200, WAB, Basel, Switzerland). The resulting powder-lump mixture was passed over a 710 µm stainless steel sieve and the lumps were crushed on the sieve using sieve inserts and the scoop. The resulting material was sieved over a 250 µm stainless steel sieve. Crushing of lumps and sieving through 710 and 250 µm sieves was repeated until 4.7 kg of powder was obtained. The powder bulk was then homogenized by placing the drum in the 3-dimensional mixer for 30 minutes.

The top particle size in the final material was 241 µm for X_{90} and 346 µm for X_{99} as measured by laser diffraction. Water content in the final material was 1.52 ± 0.22 % (m/m) as measured by KFT. An oven method was developed to provide equivalent result as obtained by KFT. The drying recipe was provided to the participants of the PT-testing round in order to harmonise the drying protocol.

Amber glass 60-ml bottles with a PE insert were filled with slightly more than 20 g each using a vibrating feeder and a balance. Units of IMEP-117 and IMEP-38 were labeled intermittently. In total 200 bottles were filled and kept at 4 ^{o}C until dispatch.

4.2 Homogeneity and stability

The homogeneity and stability studies were performed by ALS Scandinavia AB (Luleå, Sweden) using inductively coupled plasma sector field mass spectrometry (ICP/SFMS) after microwave digestion with a mixture of HNO_3/H_2O_2.

Homogeneity was evaluated according to ISO 13528:2005 [4]. The material proved to be adequately homogeneous for all measurands under study.

The stability study was conducted following the isochronous approach [5, 6]. The material proved to be stable for the 5 weeks that elapsed between the dispatch of the samples and the deadline for submission of results, for total As, Cd, Pb and Hg.

The contribution from homogeneity (u_{bb}) and stability (u_{st}) to the uncertainty of the reference value (u_{ref}) was calculated using SoftCRM [7]. The analytical results and the statistical evaluation of the homogeneity and stability studies are presented in Table 1 and Annex 6.

5. Reference values and their uncertainties

5.1 Assigned value X_{ref}

The assigned values for the four measurands investigated were determined by:
LNE – Laboratoire National de Metrologie et d' Essais (Paris, France);
SCK-CEN – Studiecentrum voor Kernenergie (Mol, Belgium); and
VITO – Vlaamse Instelling voor Technologisch Onderzoek (Mol, Belgium).

Experts were asked to use the method of their choice with no further metrological requirements. Experts were also required to report their results together with the associated expanded uncertainty and with a clear and detailed description on how uncertainty was estimated.

LNE used microwave digestion with a mixture of HNO_3/H_2O_2 with double isotope dilution - inductively coupled plasma mass spectrometry (ID-ICP/MS) for the determination of total Cd, Pb and Hg and standard addition method with ICP/MS for total As.

SCK-CEN used neutron activation analysis for the determination of total As and Hg.

VITO used digestion in a high pressure asher using quartz vessels with a mixture of HNO_3/H_2O_2 and inductively coupled plasma atomic emission spectroscopy (ICP/AES) for the determination of total As, Cd and Pb and cold vapour atomic absorption spectrometry after thermal decomposition and amalgamation for the determination of total Hg.

For this PT, the mean of the independent means provided by the expert laboratories was used to derive the assigned values (X_{ref}) according to ISO Guide 35 [8].

5.2 Associated uncertainty u_{ref}

The associated uncertainties (u_{ref}) of the assigned values were calculated combining the uncertainty of the characterization (u_{char}) with the contributions for homogeneity (u_{bb}) and stability (u_{st}) in compliance with ISO/IEC Guide 98 (GUM) [9] using Eq.1:

$$u_{ref} = \sqrt{u_{char}^2 + u_{bb}^2 + u_{st}^2} \qquad \text{Eq. 1}$$

In the case of total Pb and Hg the expert laboratories reported values with overlapping expanded uncertainties (Table 1). u_{char} was calculated according to ISO 13528:2005 [4]:

$$u_{char} = \frac{1.25}{p}\sqrt{\sum_1^p u_i^2} \qquad \text{Eq. 2}$$

where p refers to the number of expert laboratories used to assign the reference value.

For total As and Cd the experts reported non-overlapping values (Table 1). u_{char} was then calculated according to ISO Guide 35 [8]:

$$u_{char} = \frac{s}{\sqrt{p}} \qquad \text{Eq. 3}$$

where s refers to the standard deviation of the values obtained by the expert laboratories

Table 1 presents the results reported by the expert laboratories, standard uncertainty contributions, the reference values (X_{ref}, u_{ref} and U_{ref}) and the standard deviation for the PT assessment σ_p.

***Table 1** – Reported values by the expert laboratories, assigned values, their associated expanded uncertainties and target standard deviations for the measurands of this ILC (all values in mg kg^{-1}).*

	total-As	total-Cd	total-Pb	total-Hg
Expert lab 1	*2.61 ± 0.075*	*0.866 ± 0.011*	*5.639 ± 0.085*	*0.787 ± 0.025*
Expert lab 2	*3.02 ± 0.21*	*0.892 ± 0.014*	*5.67 ± 0.37*	*0.815 ± 0.058*
Expert lab 3	*2.84 ± 0.14*			*0.87 ± 0.07*
X_{ref}	**2.823**	**0.879**	**5.655**	**0.824**
u_{char}	0.119	0.013	0.119	0.020
u_{bb}	0.079	0.011	0.040	0.012
u_{st}	0.062	0.008	0.017	0.008
u_{ref}	0.156	0.019	0.126	0.024
U_{ref}	**0.311**	**0.037**	**0.252**	**0.048**
σ_p	0.282	0.088	0.565	0.082
σ_p (%)	10%	10%	10%	10%

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 %.

5.3 Standard deviation of the proficiency test assessment σ_p

On the basis of previous experience for this type of analysis the standard deviations for proficiency assessment σ_p (also called target standard deviation) was set to 10 % of the respective assigned values (Table 1).

6 Evaluation of results

6.1 Scores and evaluation criteria

Individual laboratory performance was expressed in terms of z- and ζ-scores in accordance with ISO 13528:2005 [4]:

$$z = \frac{x_{lab} - X_{ref}}{\sigma_p} \quad \text{Eq. 4 and} \qquad \zeta = \frac{x_{lab} - X_{ref}}{\sqrt{u_{ref}^2 + u_{lab}^2}} \quad \text{Eq. 5}$$

where:
- x_{lab} is the measurement result reported by a participant;
- u_{lab} is the standard uncertainty reported by a participant;
- X_{ref} is the reference value (assigned value);
- u_{ref} is the standard uncertainty of the reference value; and
- σ_p is the standard deviation for proficiency assessment

The interpretation of the z- and ζ-score is done according ISO 17043:2010 [13]:

|score| ≤ 2	satisfactory result	(green in Annexes 7 to 12)
2 < |score| < 3	questionable result	(orange in Annexes 7 to 12)
|score| ≥ 3	unsatisfactory result	(red in in Annexes 7 to 12)

The z-score compares the participant's deviation from the reference value with the target standard deviation for proficiency assessment (σ_p) used as common quality criterion. σ_p is defined by the PT organizer as the maximum acceptable standard uncertainty.

The ζ-score states whether the laboratory's result agrees with the assigned value within the respective uncertainty. The denominator is the combined uncertainty of the assigned value and the measurement uncertainty as stated by the laboratory. The ζ-score is therefore the most relevant evaluation parameter, as it includes all parts of a measurement result, namely the expected value (assigned value), its uncertainty and the unit of the result as well as the uncertainty of the reported values. An unsatisfactory ζ-score can either be caused by an inappropriate estimation of the concentration or of its uncertainty, or both.

The standard uncertainty of the laboratory (u_{lab}) was estimated by dividing the reported expanded uncertainty by the reported coverage factor, *k*. When no uncertainty was reported, it was set to zero ($u_{lab} = 0$). When *k* was not specified, the reported expanded uncertainty was considered as the half-width of a rectangular distribution; u_{lab} was then calculated by dividing this half-width by $\sqrt{3}$, as recommended by Eurachem and CITAC [10].

Uncertainty estimation is not trivial, therefore an additional assessment was provided to each laboratory reporting uncertainty, indicating how reasonable their uncertainty estimate is. The standard uncertainty from the laboratory (u_{lab}) is most likely to fall in a range between a minimum uncertainty (u_{min}), and a maximum allowed (u_{max}). u_{min} is set to the standard uncertainty of the reference value. It is unlikely that a laboratory carrying out the analysis on a routine basis would measure the measurand with a smaller uncertainty than the expert laboratories chosen to establish the assigned value. u_{max} is set to the target standard deviation (σ_p) accepted for the PT. If u_{lab} is smaller than u_{min}, (case "b") the laboratory may have underestimated its uncertainty. Such a statement has to be taken with care as each laboratory reported only measurement uncertainty, whereas the uncertainty of the reference value also includes contributions of homogeneity and stability. If those are large, measurement uncertainties smaller than u_{min} are possible and plausible. If $u_{lab} > u_{max}$, (case "c") the laboratory may have overestimated the uncertainty. An evaluation of this statement can be made when looking at the difference of the reported value and the assigned value: if the difference is small and the uncertainty is large, then overestimation is likely. If, however, the deviation is large but is covered by the uncertainty, then the uncertainty is properly assessed but large. It should be pointed out that u_{max} is only a normative criterion if set down by legislation.

6.2 General observations

From the 30 laboratories (27 countries) having registered, 29 submitted results and answered the associated questionnaire (25 for total As, 29 for total Cd, 29 for total Pb and 28 for total Hg).

Most of the participants performed the analysis following an official method. The experimental details provided by the laboratories for the methods used, are summarised in Annex 11. The influence of the methods and techniques used did not correlate to the quality of the reported results.

Annexes 7 to 10 present the reported results as a table and as a graph. Furthermore, the graphs include the corresponding Kernel density plots, obtained using the software available from the Statistical Subcommittee of the Analytical Methods Committee of the UK Royal Society of Chemistry [11].

6.3 Laboratory results and scorings

The overall performance of the participants regarding the z- and ζ-scores, is summarised in Figure 1. More than 79% of the NRLs performed satisfactorily to this exercise for the determination of the target measurands.

It should also be mentioned that in the case of total As, Cd and Pb the number of satisfactory ζ-scores are the same as the respective z-scores. A minor decrease is observed only in the case of total Hg (86% / 75%, z / ζ).

The uncertainty assessment ("*a*", "*b*" and "*c*") is presented in Anexes 7 to10. In the case of total As, only half of the laboratories that performed satisfactorily obtained an "a". Their performance was better in the cases of total Cd, Pd and Hg where 74 % of the participants reported reasonable uncertainty estimates (case "a"). Only few (3 for total As, 1 for total Pb and 4 for total Hg) reported under-estimated uncertainties (case "b"), probably based on their repeatability.

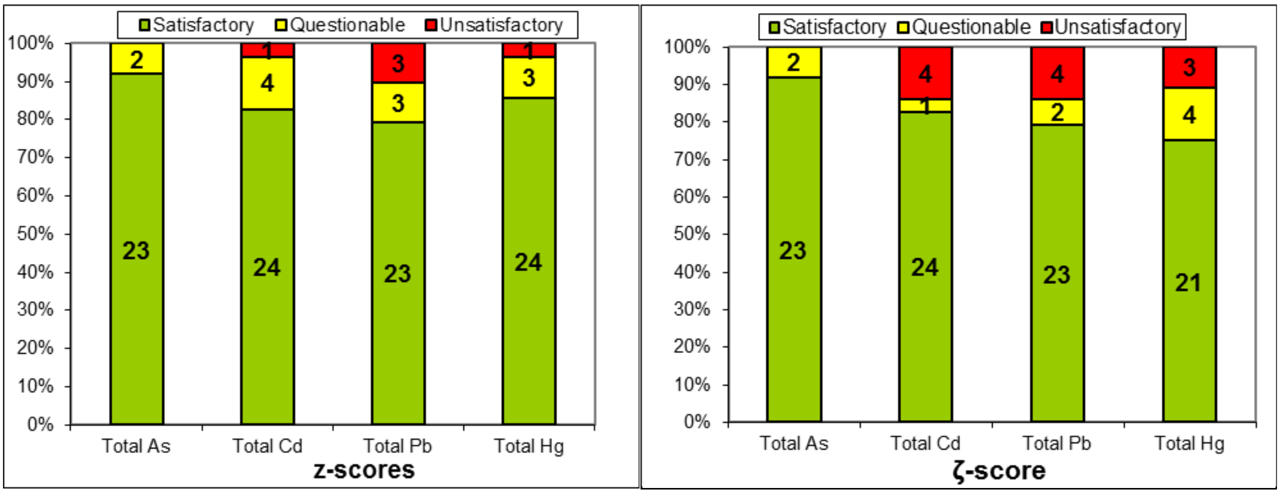


Figure 1: *Number and percentages of laboratories with satisfactory, questionable and unsatisfactory scores. (The numbers on the bars correspond to the exact number of laboratories in a certain scoring category).*

Table 2 - *Approaches used by the participants in IMEP-117 to estimate the uncertainty of their measurements.*

Approach followed for uncertainty calculation	Number of labs.
Uncertainty budget (ISO-GUM), validation	**9**
Known uncertainty of the standard method (ISO 21748)	1
Uncertainty of the method (in-house)	**17**
Measurement of replicates (precision)	**9**
Estimation based on judgment	1
Use of intercomparison data	4
Other	3

Various approaches were used to evaluate measurement uncertainties (Table 2). Most of the laboratories having reported satisfactory results either applied ISO-GUM to estimate the combined/expanded uncertainties or used intermediate precision derived from their method validation studies. Twenty one laboratories usually report uncertainty to their customers while 8 do not.

6.4 Additional information extracted from the questionnaire

According to the answers collected from the questionnaire (Annex 5) all participants (except one) stated that they have a quality system in place based on ISO 17025. In three cases the quality system is also based on ISO 9000. Most of the laboratories regularly take part in PTs (26 out 29).

The participants were asked to report the LoDs of the methods that they have used for the determination of the four measurands. These LoDs together with the respective techniques used are presented in Annex 12. Large discrepancies are observed even if laboratories used the same technique.

All participants but three (L11, L14 and L23) corrected their results for the moisture content, determined using the protocol described in the accompanying letter (Annex 3). The moisture content values reported ranged from 0.3 to 2.31 %.

Twenty seven participants determined the recovery factor applying one or several of the options shown in Table 3. Twenty five of them corrected their results for recovery. Nineteen laboratories reported the recovery used to correct their results which were in the range 72-117.8 %. Laboratories that reported recoveries lower than 80 % must be aware that such recoveries indicate that the method is significantly biased and that corrective actions should be undertaken [12].

Table 4 summarises the comments of the participants regarding the IMEP-117 exercise.

Table 3 - *Methods applied by the laboratories to determine the recovery factors of the exercise.*

How did you determine the recovery factor?	Number of labs.
adding a known amount of the same analyte to be measured (spiking)	5
using a certified reference material	9
other	9
adding a known amount of the same analyte to be measured (spiking) and using a certified reference material	3
adding a known amount of the same analyte to be measured (spiking) and other	1

Table 4 - *Comments of the laboratories participating in the IMEP-117 ILC.*

Lab ID	Comments
L101	We shall register in the FAPAS scheme for future Feed rounds.
L102	The web-pages did not work at all gave empty sheet!
L112	Laboratory does not routinely analyse feed samples.
L123	After dry ashing, ash sample was gray
L128	Tab. 1.2. reports twice Total Hg
L130	We have send the material to a subcontracter for analysis of As, Cd, Pb, Hg. The aim was to check the subcontracter.

7 Conclusions

The results collected in the frame of the IMEP-117 exercise indicate that participating laboratories performed satisfactorily for the determination of total As (92 %), for total Cd (82 %), for total Pb (79%) and total Hg (86 %). Thus, the analytical capability of NRLs for the determination of the investigated food contaminants at the investigated levels of concentration was successfully demonstrated. When comparing NRL performances to those obtained in IMEP-38 (a parallel PT open to food control laboratories using the same test samples and applying the same evaluation criteria) the overall rates of satisfactory performance obtained by the NRLs (expressed as z-scores) were 10 % (for total Pb) to 32 % (for total As) higher than the respective rates in IMEP-38.

No direct correlations between the methods of analysis used and the performances of the laboratories could be identified.

Significant discrepancies were observed for limits of detections reported, even for similar analytical methods. It is not the first time that the EURL-HM identifies problems in the calculation of the LOD. This issue will be tackled in the near future and clear information will be provided to the NRLs on the way how to determine the LOD of an analytical method.

For the first time in the seven years that the EURL-HM runs PTs for the network of NRLs, no significance difference in the performance of the laboratories in terms of z- and ζ-scores was detected. This means that the information distributed to the NRLs in trainings and in the reports of the PTs starts to pay back. This is confirmed by the small number of laboratories having underestimated their uncertainties.

8 Acknowledgements

C. Contreras and P. Connely from the Standards for Innovation and Sustainable Development (SID) Unit of the IRMM are acknowledged for their support in the isochronous study and in optimizing the method to measure the moisture content, respectively. F. Ulberth is also acknowledged for revising the manuscript.

The laboratories participating in this exercise, listed in the following table, are kindly acknowledged.

Table - 5: *Laboratories that participated in IMEP-117 and their respective countries of origin.*

Organisation	Country
AGES GmbH	Austria
CODA-CERVA	Belgium
Central Laboratory of Veterinary Control and Ecology	Bulgaria
State Veterinary Institute Olomouc	Czech Republic
CISTA	Czech Republic
Danish Veterinary and Food Administration	Denmark
Agricultural Research Centre	Estonia
Finnish Food Safety Authority Evira	Finland
Laboratoire SCL de Bordeaux - FRANCE	France
Federal Office of Consumer Protection and Food Safety (BVL)	Germany
Regional centre of plant protection & quality control of magnesia	Greece
National Food Chain Safety Office, Food and Feed Safety Directorate	Hungary
Health Service Executive	Ireland
Istituto Zooprofilattico Sperimentale del Piemonte, Liguria e Valle d'Aosta	Italy
Istituto Superiore di Sanità	Italy
Institute of Food Safety, Animal Health and Environment	Latvia
National food and veterinary risk assessment institute	Lithuania
Environmental Health Directorate	Malta
RIKILT	Netherlands
NIFES	Norway
National Veterinary Institute in Pulawy	Poland
Instituto Nacional de Investigação Agrária e Veterinaria	Portugal
Hygiene and Veterinary Public Health Institute	Romania
State Veterinary and Food Institute	Slovakia
National Veterinary Institute	Slovenia
Zavod za zdravstveno varstvo Maribor	Slovenia
Laboratorio Arbitral Agroalimentario	Spain
National Veterinary Institute	Sweden
The Food and Environment Research Agency	United Kingdom

9 Abbreviations

AMC	Analytical Methods Committee of the Royal Society of Chemistry
BIPM	Bureau International des Poids et Mesures
CITAC	Co-operation for International Traceability in Analytical Chemistry
CONTAM	Panel on Contaminants in the Food Chain
DG SANCO	Directorate General for Health and Consumer Protection
EA	European Co-operation for Accreditation
EFSA	European Food Safety Authority
ETAAS	Electrothermal atomic absorption spectrometry
EU	European Union
EURL-HM	European Union Reference Laboratory for Heavy Metals in Feed and Food
GUM	Guide for the Expression of Uncertainty in Measurement
ID-ICP-MS	Isotope dilution - inductively coupled plasma - mass spectrometry
ILC	Interlaboratory Comparison
IMEP	International Measurement Evaluation Programme
IRMM	Institute for Reference Materials and Measurements
JRC	Joint Research Centre
LoD	Limit of detection
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PT	Proficiency Test
RM	Reference material

10 References

1 Commission Directive 2002/32/EC of the European Parliament and of the Council of 7 May 2002 on undesirable substances in animal feed & Commission Regulation (EU) No 744/2012 of 16 August 2012 amending Directive 2002/32/EC of the European Parliament and of the Council of 7 May 2002 on undesirable substances in animal feed.

2 Commission Regulation (EC) 882/2004 of the European parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules, Official Journal of the European union.

3 ISO 17043:2010 – "Conformity assessment – General requirements for proficiency testing", issued by ISO-Geneva (CH), International Organization for Standardization.

4 ISO 13528:2005 - "Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons", issued by ISO-Geneva (CH), International Organization for Standardization.

5 Lamberty A., Schimmel H., Pauwels J. (1998) "The study of the stability of reference materials by isochronous measurements", Fresenius' Journal of Analytical Chemistry 360(3-4): 359-361.

6 Linsinger T. P. J., Pauwels J., Lamberty A., Schimmel H. G., Van Der Veen A. M. H., Siekmann L. (2001) "Estimating the uncertainty of stability for matrix CRMs", Analytical and Bioanalytical Chemistry 370(2-3): 183-188.

7 http://www.eie.gr/iopc/softcrm/index.html, (Accessed at date of publication of this report).

8 ISO Guide 35 Reference Materials – general and statistical principles for certification (2006), issued by ISO-Geneva (CH), ISO-Geneva (CH).

9 ISO/IEC Guide 98:2008, "*Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement*" (GUM 1995), issued by International Organisation for Standardisation, Geneva.

10 Eurachem/CITAC (2000) "Quantifying Uncertainty in Analytical Measurement", http://www.eurachem.org.

11 AMC/RSC (2006), "Representing data distributions with Kernel density estimates", Issued by the Statistical Subcommittee of the Analytical Methods Committee (AMC) of the Royal Society of Chemistry (RSC), AMC Technical Brief.

12 Commission Decision of 12 August 2002 implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results.

Annexes

Annex 1: Invitation letter to NRLs

Ref. Ares(2013)544517 - 04/04/2013

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements
European Union Reference Laboratory for Heavy Metals

Geel, 4 April 2013
JRC.D.5/PRO/IF/acs/ARES

<u>Subject</u> : **IMEP-117: Total As, Cd, Pb and Hg in compound feed**

Dear National Reference Laboratory representative,

We would like to invite you on behalf of the EURL Heavy Metals in Feed and Food, to participate in the Proficiency Test IMEP-117 for the **"Determination of <u>total</u> As, Cd, Pb and Hg in compound feed"**.

You are kindly reminded that according to Regulation (EC) No 882/2004 it is your duty as NRL to participate in PTs organised by the EURL-HM if you hold a mandate for the type of matrix investigated.

Your participation is free of charge.

Please register electronically for this proficiency test round using the following link:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1020

Once you have submitted your registration electronically, please (1) print your registration form, (2) sign it, and (3) fax it to us. <u>Your fax is the confirmation of your participation.</u>

The **deadline for registration is 15 May 2013**. Samples will be sent to participants during the second half of May 2013. The deadline for submission of results is **30 June 2013**.

Do not hesitate to contact us, in case of questions/doubts,

Yours sincerely

Dr. Ioannis Fiamegkos
IMEP-117 Coordinator

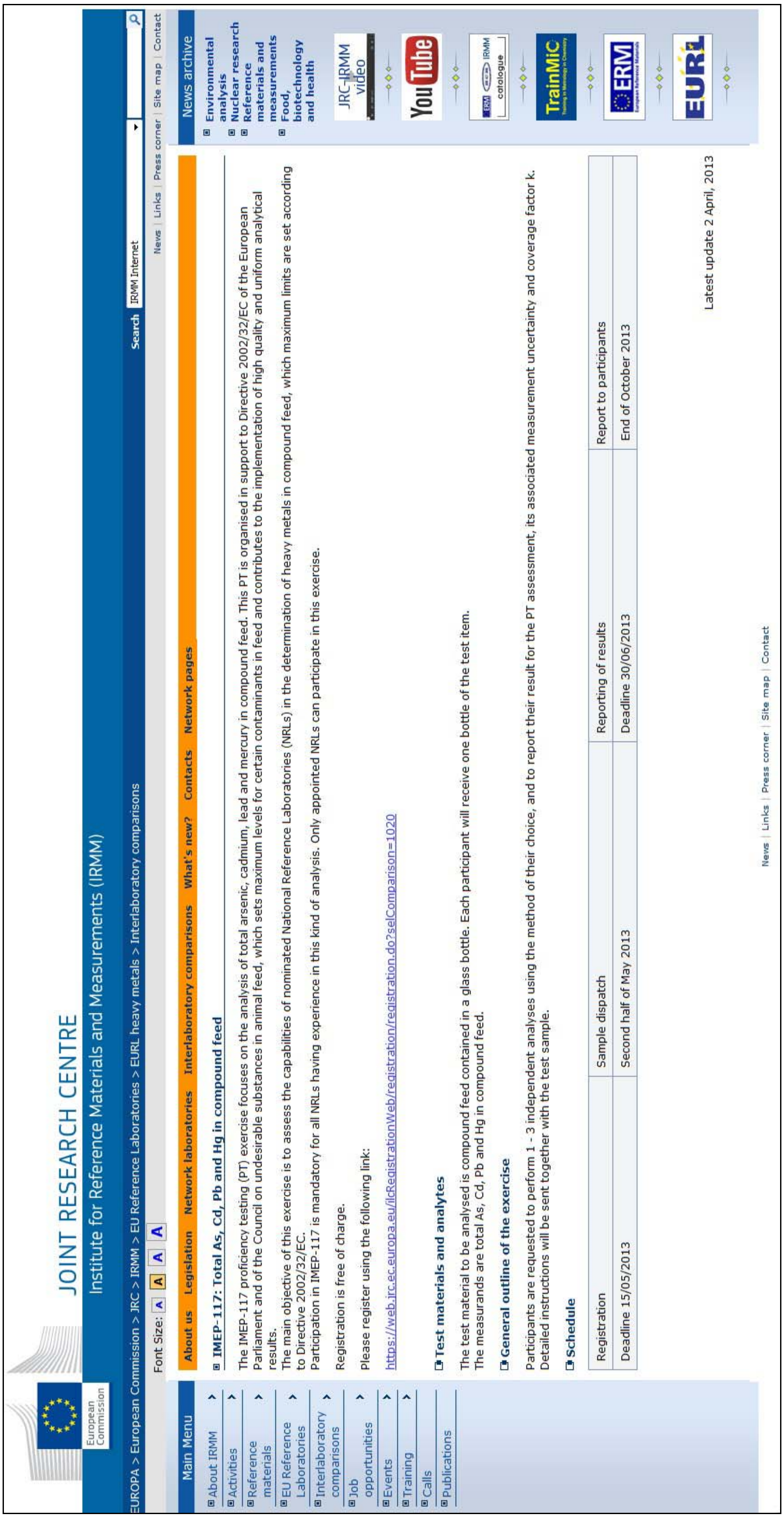
Dr. Piotr Robouch
Operating Manager EURL-HM

Cc: Franz Ulberth (Head of Unit SFB)

Retieseweg 111, B-2440 Geel - Belgium. Telephone: +32-(0)14-571 211.
Telephone: direct line +32-(0)14-571 687, Fax: +32-(0)14-571 865.

E-mail: jrc-irmm-crl-heavy-metals@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

Annex 2: IRMM – IMEP web announcement



Annex 3: Sample accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements
European Union Reference Laboratory for Heavy Metals

Geel, 24 May 2013
JRC.D5/IF/acs/Ares(2013)1384593

«Title» «Firstname» «Surname»
«Organisation»
«Department»
«Address»
«Address2»
«Zip» «Town»
«Country»

Participation in IMEP-117, a proficiency test exercise for the determination of total arsenic (As), cadmium (Cd), lead (Pb) and mercury (Hg) in compound feed.

Dear «Title» «Surname»,

Thank you for participating in the IMEP-117 proficiency test for the determination of total As, Cd, Pb and Hg in compound feed. This exercise takes place in the frame of the EURL Heavy Metals in Feed and Food and is organised in support to Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed.

Please keep this letter. You need it to report your results.

This parcel contains:
a) One bottle containing approximately 20 g of the test material
b) A "Confirmation of Receipt" form
c) This accompanying letter.

Please check whether the bottle containing the test material remained undamaged during transport. Then, please send the "Confirmation of receipt" form back (fax: +32-14-571865, e-mail: jrc-irmm-crl-heavy-metals@ec.europa.eu). You should store the sample in a dark place at ≤4 C until analysis.

The measurands are total As, Cd, Pb and Hg in compound feed.

The procedure used for the analyses should resemble as closely as possible the one that you use in routine analyses.

The results are to be reported with correction for moisture (in dry mass).

Retieseweg 111, B-2440 Geel - Belgium. Telephone: +32-(0)14-571 211.
Telephone: direct line +32-(0)14-571 687, Fax: +32-(0)14-571 865.

E-mail: jrc-irmm-crl-heavy-metals@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

To calculate the **water content** in the test material, please apply the following procedure:

1. Weigh accurately 1 g of test material in a glass container of 5-7 cm diameter. Preferably with a lid because when the prescribed drying time has passed, the glass container must cool down about 30 minutes in a desiccator before weighing.
2. Place it in a checked and calibrated drying oven for 1 h ± 5 min at 105 ± 1 °C.
3. Place the glass container covered with a lid in a desiccator and wait 30 min before weighing the test material again.
4. Calculate the average mass loss from the dried material in percentage of the initial mass.

Please note that this drying method is devised to result in a mass loss that corresponds to the water content in % (m/m) as measured by Karl Fischer titration which is specific for water. Therefore it is not necessary to dry and continue weighing until constant mass. Keeping the material longer than one hour in the oven will result in an excessive mass loss and an erroneous dry-mass correction.

Note : do not use for the heavy metal determinations the aliquots of test material that you have used for the water content determination!

Reporting of results

Please perform two or three independent measurements, correct the measurements results for recovery and for the moisture content and report on the reporting website:

- the **mean** of your two or three measurement results (mg kg^{-1}, as dry mass)
- the associated expanded **uncertainty** (mg kg^{-1}),
- the **coverage factor** and
- the **technique** you used.

The results should be reported in the same form (e.g. number of significant figures) as those normally reported to the customer.

The reporting website is https://irmm.jrc.ec.europa.eu/ilc/ilcReporting.do

To access the webpage you need a personal password key, which is: **«Part_key».** The system will guide you through the reporting procedure. After entering your results, please complete also the relating questionnaire.

Do not forget to submit and confirm always when required.

Retieseweg 111, B-2440 Geel - Belgium. Telephone: +32-(0)14-571 211.
Telephone: direct line +32-(0)14-571 374, Fax: +32-(0)14-571 865.

E-mail: jrc-irmm-crl-heavy-metals@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

Directly after submitting your results and the questionnaire information online, you will be prompted to print the completed report form. Please do so, **sign the paper version and return it to IRMM by fax (at +32-14-571-865) or by e-mail**. Check your results carefully for any errors before submission, since this is your last definitive confirmation.

The **deadline** for submission of results is **30/06/2013.**

Please keep in mind that collusion is contrary to professional scientific conduct and serves only to nullify the benefits of proficiency tests to customers, accreditation bodies and analysts alike.

Your participation in this project is greatly appreciated. If you have any remaining questions, please contact me by e-mail: jrc-irmm-crl-heavy-metals@ec.europa.eu

With kind regards,

Ioannis Fiamegkos (PhD)

IMEP-117 Coordinator

Cc: F. Ulberth (SFB HoU)

Retieseweg 111, B-2440 Geel - Belgium. Telephone: +32-(0)14-571 211.
Telephone: direct line +32-(0)14-571 374, Fax: +32-(0)14-571 865.

E-mail: jrc-irmm-crl-heavy-metals@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

Annex 4: Confirmation of receipt form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements
European Union Reference Laboratory For Heavy Metals

Annex to
JRC.D5/IF/acs/ARES(2013)1384593

«Title» «Firstname» «Surname»
«Organisation»
«Address»
«Address2»
«Zip» «Town»
«Country»

IMEP-117

Total arsenic (As), cadmium (Cd), lead (Pb) and mercury (Hg) in compound feed

Confirmation of receipt of the samples

Please return this form at your earliest convenience.
This confirms that the sample package arrived.
In case the package is damaged,
please state this on the form and contact us immediately.

ANY REMARKS ....
....

Date of package arrival ....

Signature ....

Please return this form to:

Ioannis Fiamegkos

IMEP-117 Coordinator
EC-JRC-IRMM
Retieseweg 111
B-2440 GEEL, Belgium

Fax : +32-14-571865

e-mail : JRC-IRMM-CRL-HEAVY-METALS@ec.europa.eu

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211
Telephone: direct line (32-14) 571 687, Fax: (32-14) 571 865

E-mail: jrc-irmm-crl-heavy-metals@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

Annex 5: Questionnaire

Comparison for IMEP-117

Please fill in this questionnaire

Submission Form

1. **How did you determine the recovery factor? By:**

- ☐ a) adding a known amount of the same analyte to be measured (spiking)
- ☐ b) using a certified reference material
- ☐ c) other

1.1. If "Other" please specify

1.2. Please enter the correction factors used and the LODs of your methods

Analytical recovery (in %) and limit of detection (LoD in mg / kg)

Questions/Response table	Total As	Total Cd	Total Pb	Total Hg	Total Hg
Recovery %					
LoDs (mg/Kg)					

2. **Experimental details for the analysis**

2.1. Does your laboratory use reference material for this type of analysis?

- ○ a) Yes
- ○ b) No

2.1.1. If "Yes" which one?

2.1.2. The reference material was used for: (multiple answers are possible)

- ☐ the calibration of instruments
- ☐ the validation of the procedure

2.2. Did you use an official method?

- ○ a) Yes
- ○ b) No

2.2.1. If "Yes", Which one(s)? Please refer to the help button (?) for an example

2.3. Which type of digestion did you use for each element?

Questions/Response table	Closed Microwave Dig.	Dry Ashing	Open Microwave Dig.	Open Wet Dig.	Pressure Bomb Dig.	*Info*
Total As	○	○	○	○	○	
Total Pb	○	○	○	○	○	
Total Cd	○	○	○	○	○	
Total Hg	○	○	○	○	○	

2.4. What kind of digestion mixture did you use for each element? (Multiple selections are possible)

Questions/Response table	H2O2	H2SO4	HCl	HClO4	HF	HNO3	*Info*
Total As	☐	☐	☐	☐	☐	☐	
Total Pb	☐	☐	☐	☐	☐	☐	
Total Cd	☐	☐	☐	☐	☐	☐	
Total Hg	☐	☐	☐	☐	☐	☐	

2.5. Does your laboratory carry out ths type of analysis on a regular basis? (samples per year)

Questions/Response table	a) Never	b) 0-50	c) 50-250	d) 250-1000	e) more than 1000	Info
Total As	○	○	○	○	○	
Total Pb	○	○	○	○	○	
Total Cd	○	○	○	○	○	
Total Hg	○	○	○	○	○	

2.6. Additional remarks/comments on the method(s) of analysis.

3. **Did you correct for the moisture content of the sample?**

- a) Yes
- b) No

3.1. If "Yes", what is the moisture content of the sample (in % of the sample mass)?

3.2. If "no", what was the reason not to do this?

4. **What is the basis of your uncertainty estimate (multiple answers are possible)?**

- a) Uncertainty budget (ISO-GUM)
- b) Known uncertainty of the standard method (ISO 21748)
- c) Uncertainty of the method (in-house validation)
- d) Measurement of replicates (precision)
- e) Estimation based on judgemnt
- f) Use of intercomparison data
- g) Other

4.1. If other, please specify

5. **What is the level of confidence (in %) reflected by the coverage (k) assigned to your reported uncertainty**

6. **Do you usually provide an uncertainty statement to your customers for this type of analysis?**

- a) Yes
- b) No

7. **Does your laboratory have a quality system in place?**

- a) Yes
- b) No

7.1. If "Yes", which:

- a) ISO 17025
- b) ISO 9000 series
- c) Other

7.1.1. If other, please specify

8. **Does your laboratory take part in interlaboratory comparison scheme for this type of analysis?**

- a) Yes
- b) No

8.1. If "yes", which one(s)?

9. **Do you have any comments? Please let us know: ...**

Annex 6: Homogeneity and stability studies

6.1 Homogeneity studies

	Total As		Total Cd		Total Pb		TotalHg	
Bottle ID	*R1*	*R2*	*R1*	*R2*	*R1*	*R2*	*R1*	*R2*
46	2.71	2.72	0.906	0.890	5.92	5.92	0.819	0.808
9	2.56	2.63	0.871	0.887	5.83	5.82	0.787	0.816
37	2.76	2.74	0.884	0.891	5.76	5.92	0.795	0.818
72	2.73	2.89	0.868	0.894	5.72	5.99	0.773	0.81
16	2.68	2.70	0.872	0.869	5.92	5.72	0.784	0.795
117	2.72	2.77	0.873	0.901	5.80	5.84	0.812	0.827
57	2.84	2.88	0.894	0.928	5.95	6.04	0.82	0.837
70	2.73	2.82	0.917	0.912	5.91	6.04	0.834	0.841
97	2.64	2.64	0.885	0.890	5.91	5.97	0.819	0.794
23	2.64	2.62	0.890	0.871	5.93	5.90	0.813	0.809
Mean	2.72		0.890		5.89		0.811	
σ_p	0.28		0.088		0.57		0.082	
0.3* σ_p	0.08		0.026		0.17		0.025	
Critical value	0.015		0.0015		0.067		0.0013	
s_x	0.08		0.014		0.07		0.015	
s_w	0.05		0.013		0.09		0.014	
s_s	0.08		0.010		0.01		0.011	
$s_s \leq 0.3 * s_p$	Pass		Pass		Pass		Pass	
s_s^2 < critical	Pass		Pass		Pass		Pass	

Where σ_p is the standard deviation for the PT assessment,
s_x is the standard deviation of the sample averages,
s_w is the within-sample standard deviation,
s_s is the between-sample standard deviation,

6.2 Stability studies

	Time in Weeks				u_{st}
	0	**3**	**5**	**8**	
As	2.7	2.67	2.45	2.64	**2.2%**
	2.7	2.5	2.67	2.57	
Cd	0.852	0.862	0.872	0.837	**0.9%**
	0.861	0.842	0.865	0.848	
Pb	5.82	5.81	5.75	5.69	**0.3%**
	5.86	5.8	5.82	5.7	
Hg	0.8	0.805	0.771	0.772	**1.0%**
	0.83	0.784	0.782	0.775	

Annex 7: Results for total As

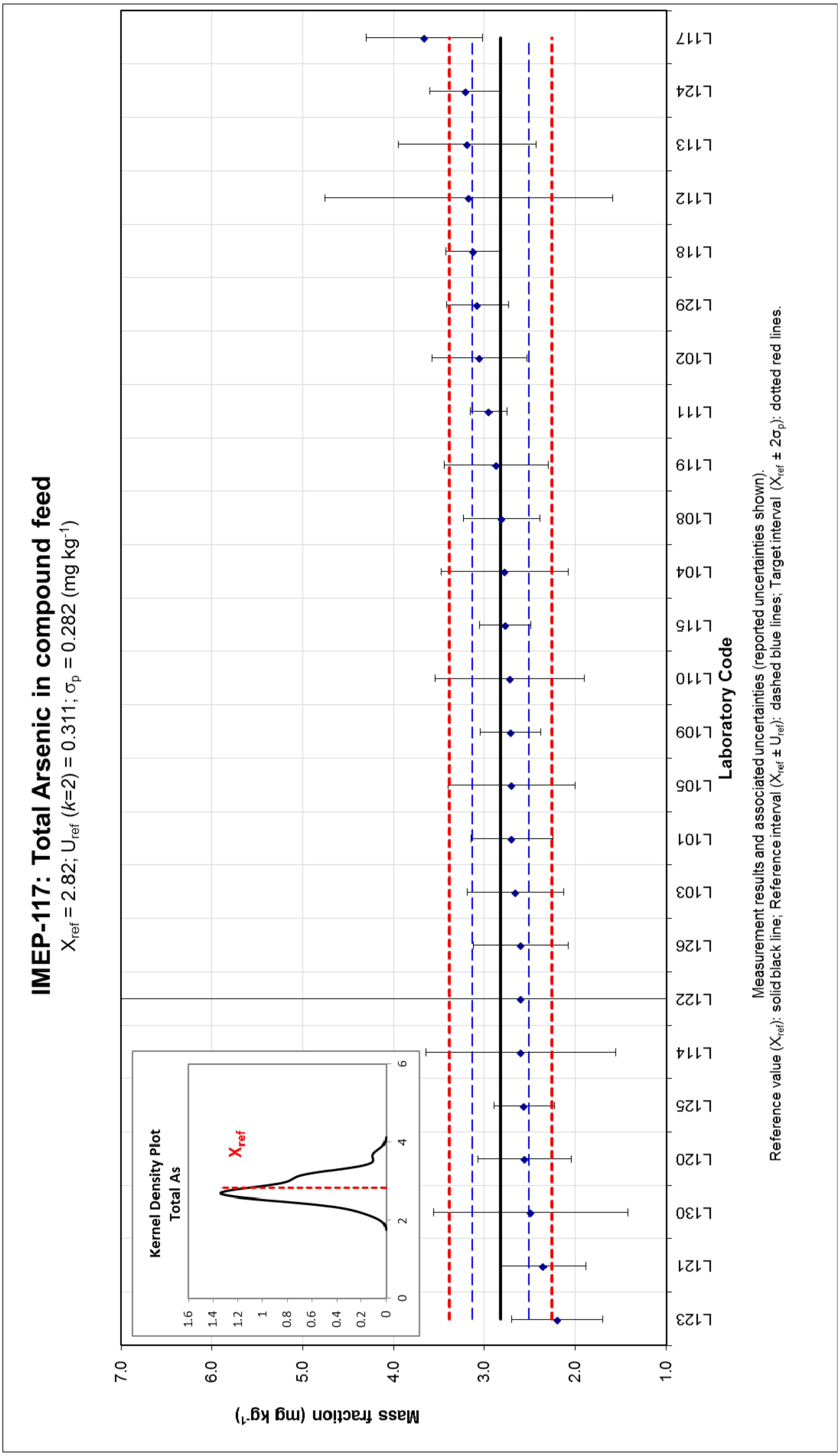
Assigned range: X_{ref} = 2.82; U_{Ref} (k=2) = 0.311; σ_p = 0.282 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L101	2.7	0.45	2	ICP-QMS	0.225	-0.44	-0.45	a
L102	3.05	0.52	2	ICP-MS	0.26	0.80	0.75	a
L103	2.66	0.53	2	HG-AAS	0.265	-0.58	-0.53	a
L104	2.78	0.70	2		0.35	-0.15	-0.11	c
L105	2.7	0.7	2	ICP-MS	0.35	-0.44	-0.32	c
L108	2.81	0.42	2	HG-AAS	0.21	-0.05	-0.05	a
L109	2.71	0.33	2	ETAAS	0.165	-0.40	-0.50	a
L110	2.72	0.82	2	ICP-AES	0.41	-0.37	-0.24	c
L111	2.951	0.20	2	ICP-MS	0.1	0.45	0.69	b
L112	3.17	1.58	2	ICP-MS	0.79	1.23	0.43	c
L113	3.19	0.76	2	ICP-QMS	0.38	1.30	0.89	c
L114	2.6	1.04	2	ICP-QMS	0.52	-0.79	-0.41	c
L115	2.77	0.28	2	ICP-QMS	0.14	-0.19	-0.25	b
L117	3.66	0.64	2	ICP-MS	0.32	2.96	2.35	c
L118	3.125	0.300	2	HG-AAS	0.15	1.07	1.40	b
L119	2.873	0.574	√3	ICP-MS	0.331399	0.18	0.14	c
L120	2.561	0.512	2	H-AAS	0.256	-0.93	-0.88	a
L121	2.35	0.47	2	ICP-MS	0.235	-1.68	-1.68	a
L122	2.6	14	2	ICP-QMS	7	-0.79	-0.03	c
L123	2.2	0.5	2	HG-AAS	0.25	-2.21	-2.12	a
L124	3.21	0.39	2	HG-AAS FIAS	0.195	1.37	1.55	a
L125	2.563	0.333	2	ICP-MS	0.1665	-0.92	-1.14	a
L126	2.6	0.52	2	ET-AAS	0.26	-0.79	-0.74	a
L129	3.08	0.34	2	HG-AAS	0.17	0.9	1.1	a
L130	2.49	1.07	2	ICP-MS	0.535	-1.2	-0.6	c

[a] √3 is set by the ILC coordinator when no expansion factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k*=√3.

[b] **Satisfactory, Questionable, Unsatisfactory**

[c] **a** : $u_{min} \leq u_{lab} \leq u_{max}$; **b** : $u_{lab} < u_{min}$; and **c** : $u_{lab} > u_{max}$


IMEP-117: Total Arsenic in compound feed
X_ref = 2.82; U_ref (k=2) = 0.311; σ_p = 0.282 (mg kg-1)
Mass fraction (mg kg-1)
7.0
6.0
5.0
4.0
3.0
2.0
1.0
Kernel Density Plot
Total As
X_ref
L123
L121
L130
L120
L125
L114
L122
L126
L103
L101
L105
L109
L110
L115
L104
L108
L119
L111
L102
L129
L118
L112
L113
L124
L117
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_ref): solid black line; Reference interval (X_ref ± U_ref): dashed blue lines; Target interval (X_ref ± 2σ_p): dotted red lines.

Annex 8: Results for total Cd

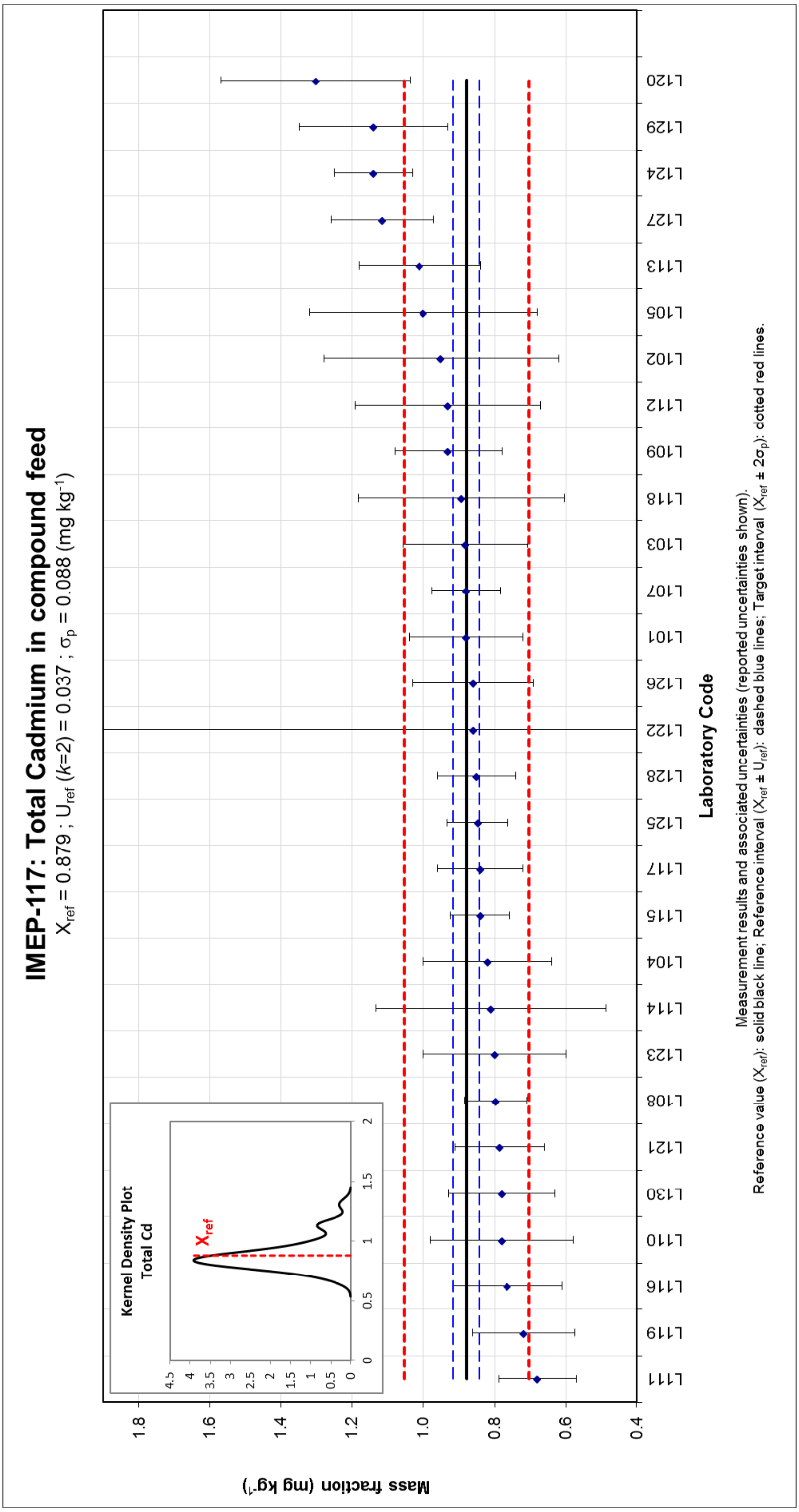
Assigned range: X_{ref} = 0.879 ; U_{ref} (k=2) = 0.037 ; s_p = 0.088 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L101	0.88	0.16	2	ICP-QMS	0.080	0.01	0.01	a
L102	0.95	0.33	2	ICP-MS	0.165	0.81	0.43	c
L103	0.881	0.176	2	ET-AAS	0.088	0.02	0.02	c
L104	0.819	0.18	2		0.090	-0.68	-0.65	c
L105	1.00	0.32	2	ETAAS	0.160	1.38	0.75	c
L107	0.88	0.096	2	ET-AAS	0.048	0.01	0.02	a
L108	0.796	0.088	2	ET-AAS	0.044	-0.94	-1.74	a
L109	0.93	0.15	2	ETAAS	0.075	0.58	0.66	a
L110	0.78	0.2	2	ICP-AES	0.100	-1.13	-0.97	c
L111	0.679	0.11	2	ICP-AES	0.055	-2.28	-3.45	a
L112	0.93	0.26	2	ICP-MS	0.130	0.58	0.39	c
L113	1.01	0.17	2	ICP-QMS	0.085	1.49	1.51	a
L114	0.81	0.324	2	ICP-QMS	0.162	-0.78	-0.42	c
L115	0.840	0.084	2	ICP-QMS	0.042	-0.44	-0.85	a
L116	0.764	0.153	2	FAAS	0.077	-1.31	-1.46	a
L117	0.84	0.12	2	ICP-MS	0.060	-0.44	-0.62	a
L118	0.893	0.290	2	ETAAS	0.145	0.16	0.10	c
L119	0.718	0.143	√3	ICP-MS	0.083	-1.83	-1.90	a
L120	1.303	0.266	2	ET-AAS	0.133	4.82	3.16	c
L121	0.785	0.126	2	ICP-MS	0.063	-1.07	-1.43	a
L122	0.86	18	2	ICP-QMS	9.000	-0.22	0.00	c
L123	0.8	0.2	2	FAAS	0.100	-0.90	-0.78	c
L124	1.14	0.11	2	ETAAS	0.055	2.97	4.50	a
L125	0.847	0.085	2	ICP-MS	0.043	-0.36	-0.69	a
L126	0.86	0.17	2	ET-AAS	0.085	-0.22	-0.22	a
L127	1.116	0.145	2	ETAAS	0.073	2.70	3.17	a
L128	0.85	0.11	2	ICP-MS	0.055	-0.33	-0.50	a
L129	1.14	0.21	2	ET-AAS	0.105	2.97	2.45	c
L130	0.780	0.149	2	ICP-MS	0.075	-1.13	-1.29	a

[a] √3 is set by the ILC coordinator when no expansion factor k is reported. The reported uncertainty was assumed to have a rectangular distribution with $k=\sqrt{3}$.

[b] **Satisfactory, Questionable, Unsatisfactory**

[c] **a** : $u_{min} \leq u_{lab} \leq u_{max}$; **b** : $u_{lab} < u_{min}$; and **c** : $u_{lab} > u_{max}$


IMEP-117: Total Cadmium in compound feed
Xref = 0.879 ; Uref (k=2) = 0.037 ; σp = 0.088 (mg kg-1)
Mass fraction (mg kg-1)
Laboratory Code
L111 L119 L116 L110 L130 L121 L108 L123 L114 L104 L115 L117 L125 L128 L122 L126 L101 L107 L103 L118 L109 L112 L102 L105 L113 L127 L124 L129 L120
Kernel Density Plot
Total Cd
Xref
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (Xref): solid black line; Reference interval (Xref ± Uref): dashed blue lines; Target interval (Xref ± 2σp): dotted red lines.

Annex 9: Results for total Pb

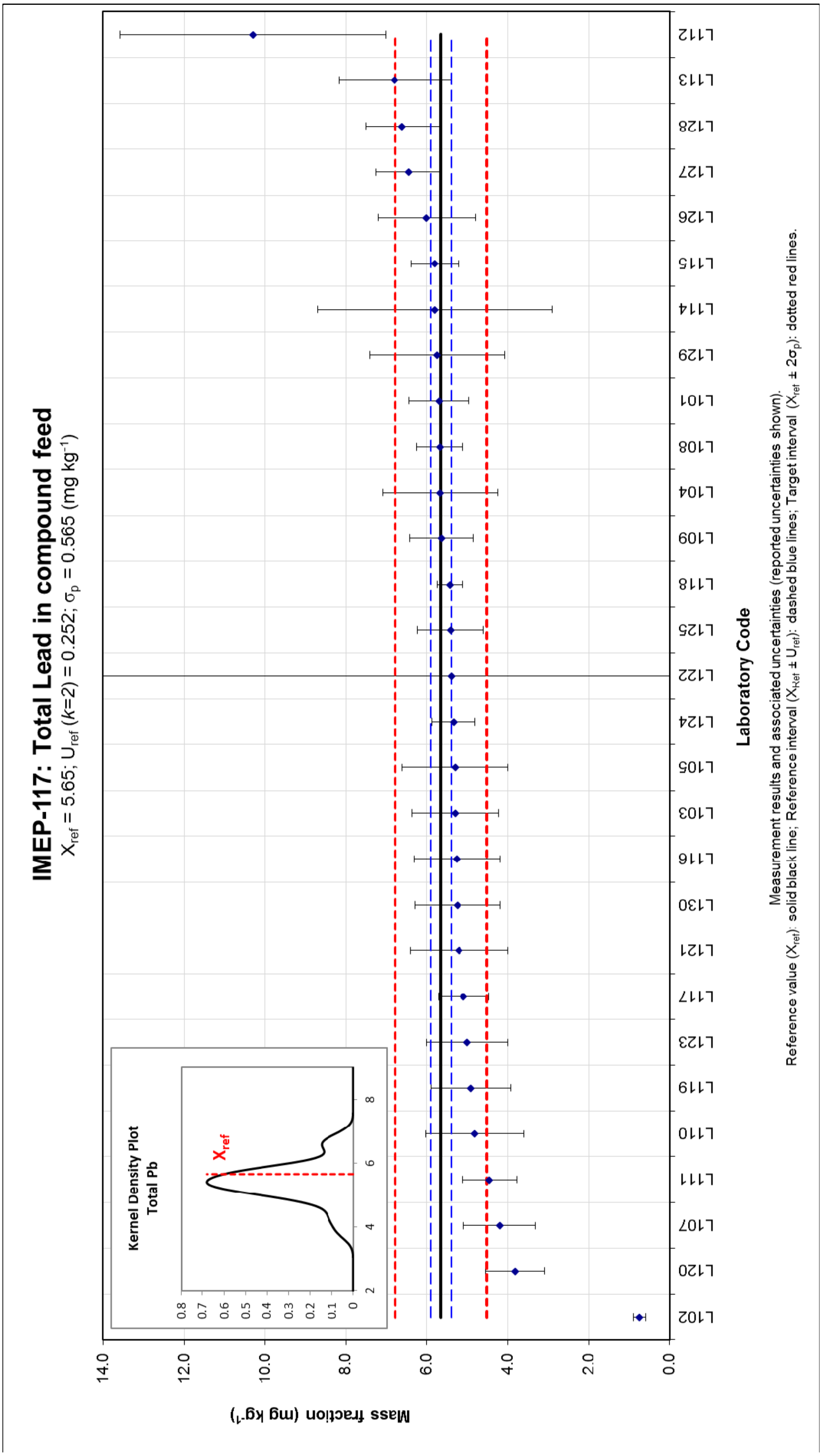
Assigned range: X_{ref} = 5.65; U_{ref} (k=2) = 0.252; σ_p = 0.565 (all values in mg kg^{-1})

Lab Code	X_{lab}	±	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L101	5.7	0.74	2	ICP-QMS	0.370	0.08	0.12	a
L102	0.76	0.15	2	ICP-MS	0.075	-8.66	-33.34	b
L103	5.30	1.06	2	ET-AAS	0.530	-0.63	-0.65	a
L104	5.67	1.42	2		0.710	0.03	0.02	c
L105	5.3	1.3	2	ICP-MS	0.650	-0.63	-0.54	c
L107	4.20	0.89	2	ET-AAS	0.445	-2.57	-3.14	a
L108	5.68	0.57	2	HG-AAS	0.285	0.05	0.08	a
L109	5.64	0.79	2	ETAAS	0.395	-0.03	-0.03	a
L110	4.82	1.21	2	ICP-AES	0.605	-1.48	-1.35	c
L111	4.449	0.67	2	ICP-AES	0.335	-2.13	-3.37	a
L112	10.3	3.29	2	ICP-MS	1.645	8.22	2.82	c
L113	6.79	1.37	2	ICP-QMS	0.685	2.01	1.63	c
L114	5.8	2.9	2	ICP-QMS	1.450	0.26	0.10	c
L115	5.8	0.58	2	ICP-QMS	0.290	0.26	0.46	a
L116	5.256	1.051	2	FAAS	0.526	-0.70	-0.74	a
L117	5.09	0.62	2	ICP-MS	0.310	-1.00	-1.69	a
L118	5.424	0.317	2	ETAAS	0.159	-0.41	-1.14	a
L119	4.913	0.982	√3	ICP-MS	0.567	-1.31	-1.28	c
L120	3.822	0.730	2	ET-AAS	0.365	-3.24	-4.74	a
L121	5.20	1.20	2	ICP-MS	0.600	-0.80	-0.74	c
L122	5.4	16	2	ICP-QMS	8.000	-0.45	-0.03	c
L123	5	1	2	FAAS	0.500	-1.16	-1.27	a
L124	5.34	0.53	2	ETAAS	0.265	-0.56	-1.07	a
L125	5.417	0.813	2	ICP-MS	0.407	-0.42	-0.56	a
L126	6	1.2	2	ET-AAS	0.600	0.61	0.56	c
L127	6.448	0.806	2	ETAAS	0.403	1.40	1.88	a
L128	6.6	0.9	2	ICP-MS	0.450	1.67	2.02	a
L129	5.75	1.66	2	ET-AAS	0.830	0.17	0.11	c
L130	5.24	1.05	2	ICP-MS	0.525	-0.73	-0.77	a

[a] √3 is set by the ILC coordinator when no expansion factor k is reported. The reported uncertainty was assumed to have a rectangular distribution with $k=\sqrt{3}$.

[b] **Satisfactory, Questionable, Unsatisfactory**

[c] **a** : $u_{min} \leq u_{lab} \leq u_{max}$; **b** : $u_{lab} < u_{min}$; and **c** : $u_{lab} > u_{max}$



Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{ref}): solid black line; Reference interval ($X_{Ref} \pm U_{ref}$): dashed blue lines; Target interval ($X_{ref} \pm 2\sigma_p$): dotted red lines.

Annex 10: Results for total Hg

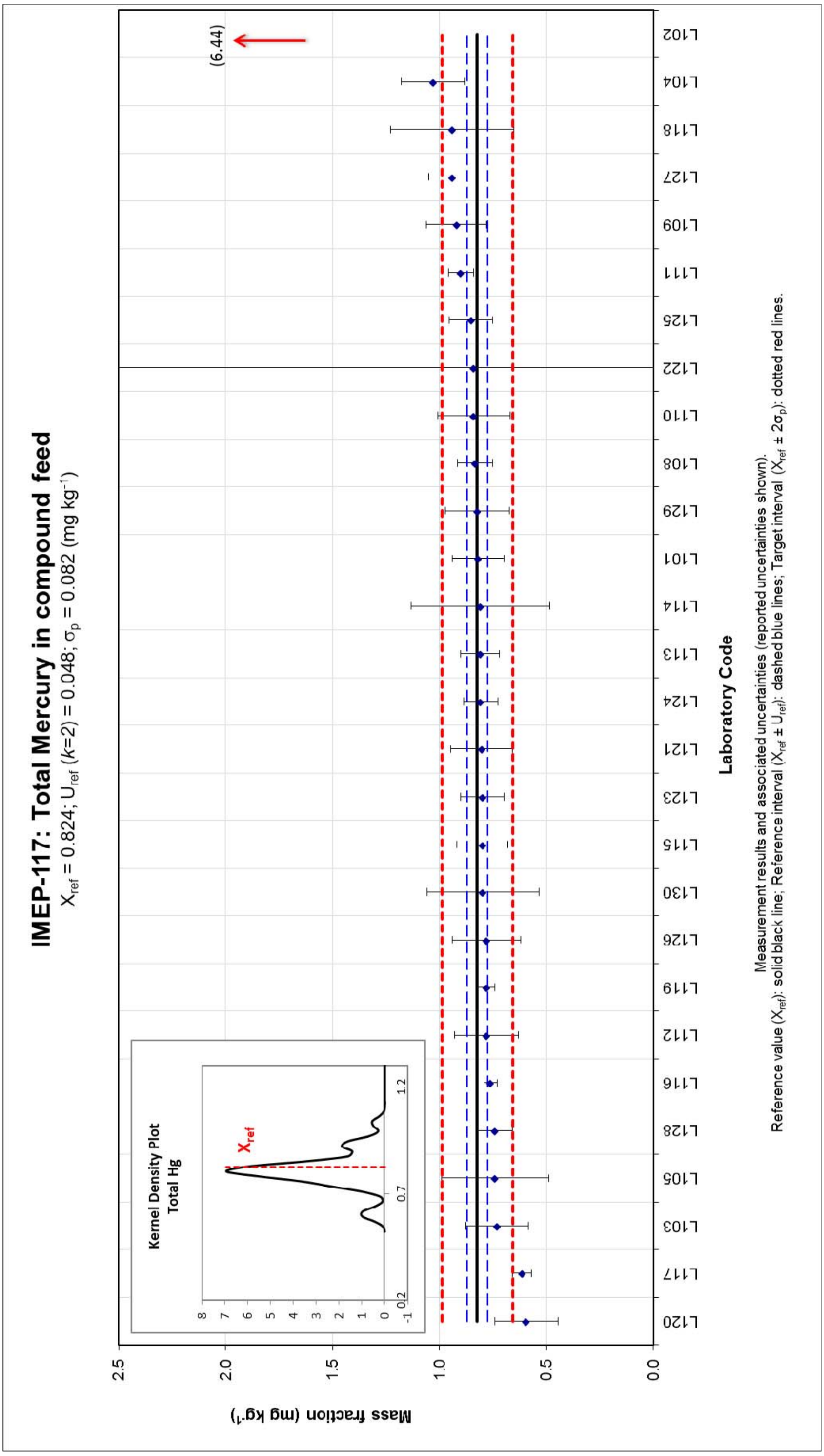
Assigned range: X_{ref} = 0.824; U_{ref} (k=2) = 0.048; σ_p = 0.082 (al values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L101	0.82	0.12	2	ICP-QMS	0.06	-0.05	-0.06	a
L102	6.44	0.52	2	ICP-MS	0.26	68.16	21.51	c
L103	0.730	0.146	2	CV-AAS	0.073	-1.14	-1.22	a
L104	1.03	0.15	2		0.075	2.50	2.61	a
L105	0.74	0.25	2	CV-AAS	0.125	-1.02	-0.66	c
L108	0.834	0.083	2	Direct mercury analyzer	0.0415	0.12	0.21	a
L109	0.92	0.14	2	TDA-AAS	0.07	1.17	1.30	a
L110	0.84	0.17	2	AMA 254	0.085	0.19	0.18	c
L111	0.9000	0.058	2	ICP-MS	0.029	0.92	2.01	a
L112	0.78	0.15	2	ICP-MS	0.075	-0.53	-0.56	a
L113	0.81	0.09	2	Direct Mercury Analyzer	0.045	-0.17	-0.27	a
L114	0.81	0.324	2	ICP-QMS	0.162	-0.17	-0.09	c
L115	0.800	0.12	2	AMA	0.06	-0.29	-0.37	a
L116	0.7611	0.0304	2	AMA 254	0.0152	-0.76	-2.20	b
L117	0.614	0.045	2	ICP-MS	0.0225	-2.55	-6.35	b
L118	0.941	0.288	2	CV-AAS	0.144	1.42	0.80	c
L119	0.780	0.039	√3	Direct mercury analysis	0.022517	-0.53	-1.33	b
L120	0.594	0.149	2	CV-AAS	0.0745	-2.79	-2.94	a
L121	0.802	0.144	2	AAS	0.072	-0.27	-0.29	a
L122	0.84	20	2	ICP-QMS	10	0.19	0.00	c
L123	0.8	0.1	2	CV-AAS	0.05	-0.29	-0.43	a
L124	0.808	0.081	2	CV-AAS	0.0405	-0.19	-0.34	a
L125	0.853	0.102	2	CV-AFS	0.051	0.35	0.51	a
L126	0.78	0.16	2	CV-AFS	0.08	-0.53	-0.53	a
L127	0.939	0.113	2	CV-AAS	0.0565	1.4	1.9	a
L128	0.74	0.08	2	FIMS	0	-1.0	-3.5	b
L129	0.823	0.148	2	AAS AMA 254	0.074	0.0	0.0	a
L130	0.797	0.262	2	ICP-MS	0.131	-0.3	-0.2	c

[a] √3 is set by the ILC coordinator when no expansion factor k is reported. The reported uncertainty was assumed to have a rectangular distribution with $k=\sqrt{3}$.

[b] **Satisfactory, Questionable, Unsatisfactory**

[c] **a** : $u_{min} \leq u_{lab} \leq u_{max}$; **b** : $u_{lab} < u_{min}$; and **c** : $u_{lab} > u_{max}$


IMEP-117: Total Mercury in compound feed
X_{ref} = 0.824; U_{ref} (k=2) = 0.048; σ_p = 0.082 (mg kg^{-1})
Mass fraction (mg kg^{-1})
2.5
2.0
1.5
1.0
0.5
0.0
Kernel Density Plot
Total Hg
X_{ref}
(6.44)
L120
L117
L103
L105
L128
L116
L112
L119
L126
L130
L115
L123
L121
L124
L113
L114
L101
L129
L108
L110
L122
L125
L111
L109
L127
L118
L104
L102
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{ref}): solid black line; Reference interval ($X_{ref} \pm U_{ref}$): dashed blue lines; Target interval ($X_{ref} \pm 2\sigma_p$): dotted red lines.

Annex 11: Experimental details and scoring

Lab. code	Official method	Reference material and its usage	Digestion type	Digestion acids	Technique	Analysis frequency	z-scoring
L101	b) No	BCR185R bovine liver, OBTL-5 tobacco leaves, FAPAS 07160 canned crabmeat for the validation of the procedure. *Additional info*: Feed is rarely analysed here, we will obtain more suitable CRMs for future work.	Closed Microwave Dig.	HCl, HNO_3	ICP-MS	b) 0-50	Total Hg
			Closed Microwave Dig.	HCl, HNO_3	ICP-MS	b) 0-50	Total Cd
			Closed Microwave Dig.	HCl, HNO_3	ICP-MS	b) 0-50	Total Pb
			Closed Microwave Dig.	HCl, HNO_3	ICP-MS	b) 0-50	Total As
L102	b) No	NIST (RICE FLOUR 1568a, PINE NEEDLES 1575a) for the quality control	Dry Ashing		ICP-MS	d) 250-1000	Total Hg
			Closed Microwave Dig.	HNO_3	ICP-MS	d) 250-1000	Total Cd
			Closed Microwave Dig.	HNO_3	ICP-MS	d) 250-1000	Total Pb
			Closed Microwave Dig.	HNO_3	ICP-MS	d) 250-1000	Total As
L103	As-EN 16206, Pb-EN 15550, Cd-EN 15550, Hg-EN 16277	NIST 1547 for the validation of the procedure	Open Wet Dig.	$HClO_4$, HNO_3	CV-AAS	d) 250-1000	Total Hg
			Closed Microwave Dig.	HNO_3	ET-AAS	c) 50-250	Total Cd
			Closed Microwave Dig.	HNO_3	ET-AAS	c) 50-250	Total Pb
			Open Wet Dig.	$HClO_4$, HNO_3	HG-AAS	d) 250-1000	Total As
L104	EN 13806, EN14546, EN15550	b) No	Closed Microwave Dig.	H_2O_2, HNO_3		d) 250-1000	Total Hg
			Closed Microwave Dig.	H_2O_2, HNO_3		d) 250-1000	Total Cd
			Closed Microwave Dig.	H_2O_2, HNO_3		d) 250-1000	Total Pb
			Dry Ashing	HCl, HNO_3		d) 250-1000	Total As
L105	LST EN 15550:2008 (Cd); LST EN 15763:2010 (As, Pb)	PT residues IMEP, FAPAS for the validation of the procedure	Closed Microwave Dig.	H_2O_2, HNO_3	CV-AAS	c) 50-250	Total Hg
			Closed Microwave Dig.	H_2O_2, HNO_3	ET-AAS	c) 50-250	Total Cd
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total Pb
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total As
L107	AOAC 999.10	FAPAS 7152 for the validation of the procedure	Pressure Bomb Dig.	H_2O_2, HNO_3	ET-AAS	c) 50-250	Total Hg
							Total Cd
			Pressure Bomb Dig.	H_2O_2, HNO_3	ET-AAS	c) 50-250	Total Pb
L108	AOAC	ZC73012 - Cabbage, TORT2 - Tort, 1577c - Bovine Liver for the validation of the procedure	Dry Ashing	HNO_3	Direct mercury analyzer	e) more than 1000	Total Hg
			Dry Ashing	HNO_3	ET-AAS	e) more than 1000	Total Cd
			Dry Ashing	HNO_3	HG-AAS	e) more than 1000	Total Pb
			Dry Ashing	HNO_3	HG-AAS	d) 250-1000	Total As
L109	b) No	tomato leaves NIST 1573a; lichen BCR 482; Sea lettuce BCR 279 for the validation of the procedure			TDA-AAS	c) 50-250	Total Hg
			Closed Microwave Dig.	H_2O_2, HF, HNO_3	ETAAS	c) 50-250	Total Cd
			Closed Microwave Dig.	H_2O_2, HF, HNO_3	ETAAS	c) 50-250	Total Pb
			Closed Microwave Dig.	H_2O_2, HF, HNO_3	ETAAS	c) 50-250	Total As
L110	b) No	internal reference material, IMEP, FAPAS for the calibration of instruments, the validation of the procedure			AMA 254	d) 250-1000	Total Hg
			Closed Microwave Dig.	HNO_3	ICP-AES	d) 250-1000	Total Cd
			Closed Microwave Dig.	HNO_3	ICP-AES	d) 250-1000	Total Pb
			Closed Microwave Dig.	HNO_3	ICP-AES	d) 250-1000	Total As
L111	EF/152/200	NIST 695 for the validation of the	Closed Microwave Dig.	HNO_3	ICP-MS	d) 250-1000	Total Hg

Lab. code	Official method	Reference material and its usage	Digestion type	Digestion acids	Technique	Analysis frequency	z-scoring
	9 - DS/EN 15510:2007	procedure	Closed Microwave Dig.	HNO_3	ICP-AES	d) 250-1000	Total Cd
			Closed Microwave Dig.	HNO_3	ICP-AES	d) 250-1000	Total Pb
			Closed Microwave Dig.	HNO_3	ICP-MS	d) 250-1000	Total As
L112	b) No	LGC 7162, NRC TORT2 for the validation of the procedure	Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	a) Never	Total Hg
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	a) Never	Total Cd
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	a) Never	Total Pb
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	a) Never	Total As
L113	b) No	DORM-3, IRMM-804 the validation of the procedure			Direct Mercury Analyzer	a) Never	Total Hg
			Closed Microwave Dig.	HNO_3	ICP-QMS	a) Never	Total Cd
			Closed Microwave Dig.	HNO_3	ICP-QMS	a) Never	Total Pb
			Closed Microwave Dig.	HNO_3	ICP-QMS	a) Never	Total As
L114	NMKL procedure nr 186, 2007	Oyster Tissue and Tort-2	Closed Microwave Dig.	HNO_3	ICP-QMS	e) more than 1000	Total Hg
			Closed Microwave Dig.	HNO_3	ICP-QMS	e) more than 1000	Total Cd
			Closed Microwave Dig.	HNO_3	ICP-QMS	e) more than 1000	Total Pb
			Closed Microwave Dig.	HNO_3	ICP-QMS	e) more than 1000	Total As
L115	b) No	CRM ASTASOL (ANALYTIKA) for the calibration of instruments			AMA	c) 50-250	Total Hg
			Open Microwave Dig.	HNO_3	ICP-QMS	c) 50-250	Total Cd
			Open Microwave Dig.	HNO_3	ICP-QMS	c) 50-250	Total Pb
			Open Microwave Dig.	HNO_3	ICP-QMS	c) 50-250	Total As
L116	b) No	BAR 463 and AAFCO samples for the validation of the procedure			AMA - 254	c) 50-250	Total Hg
				HNO_3	FAAS	c) 50-250	Total Cd
			Dry Ashing	HNO_3	FAAS	c) 50-250	Total Pb
L117	b) No	ERM-CD 281 for the validation of the procedure	Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total Hg
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total Cd
				H_2O_2, HNO_3	ICP-MS	c) 50-250	Total Pb
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total As
L118	As- EN 14546:2005	EU-RL(HM-CEFAO) Interlaboratory Comparison samples for the validation of the procedure	Open Wet Dig.	HNO_3	CV-AAS	b) 0-50	Total Hg
			Open Wet Dig.	HNO_3	ETAAS	b) 0-50	Total Cd
			Open Wet Dig.	HNO_3	ETAAS	b) 0-50	Total Pb
				HCl, HNO_3	HG-AAS	b) 0-50	Total As
L119	STN EN 15763	internal reference materials from the previous tests			Direct mercury analysis	c) 50-250	Total Hg
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total Cd
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total Pb
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total As
L120	As MSZ EN 16206;Pb,Cd Msz EN 15550	BCR191,IMEP114 for the validation of the procedure		H_2O_2, HNO_3	CV-AAS	c) 50-250	Total Hg
			Closed Microwave Dig.	H_2O_2, HNO_3	ET-AAS	c) 50-250	Total Cd
			Closed Microwave Dig.	H_2O_2, HNO_3	ET-AAS	c) 50-250	Total Pb
			Closed Microwave Dig.	H_2O_2, HNO_3	H-AAS	c) 50-250	Total As

Lab. code	Official method	Reference material and its usage	Digestion type	Digestion acids	Technique	Analysis frequency	z-scoring
L121	SIST EN 15763, EPA 7473	NCS ZC73009 for the validation of the procedure			AAS	b) 0-50	Total Hg
				H_2O_2, HNO_3	ICP-MS	b) 0-50	Total Cd
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	b) 0-50	Total Pb
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	b) 0-50	Total As
L122	b) No	Spare sample from previous proficiency test for the validation of the procedure			ICP-QMS	c) 50-250	Total Hg
			Closed Microwave Dig.	HNO_3	ICP-QMS	c) 50-250	Total Cd
				HNO_3	ICP-QMS	c) 50-250	Total Pb
			Closed Microwave Dig.	HNO_3	ICP-QMS	c) 50-250	Total As
L123	SR EN 13806, SR EN 14082, SR EN 14546	BCR 32- for Cd, Hg, As and CRM 463 for Hg	Closed Microwave Dig.	H_2O_2, HNO_3	CV-AAS	c) 50-250	Total Hg
			Dry Ashing	HCl	FAAS	d) 250-1000	Total Cd
			Dry Ashing	HCl	FAAS	d) 250-1000	Total Pb
				HCl, HNO_3	HG-AAS	d) 250-1000	Total As
L124	AOAC 999.11, AOAC971.21	b) No	Open Wet Dig.	H_2SO_4, HNO_3	CV-AAS	c) 50-250	Total Hg
			Dry Ashing	HNO_3	ETAAS	c) 50-250	Total Cd
			Dry Ashing	HNO_3	ETAAS	c) 50-250	Total Pb
			Dry Ashing	HNO_3	HG-AAS FIAS	c) 50-250	Total As
L125	b) No	BCR-032, VDLUFA-PT-Material 388Qd, national Monitoring-PTmaterials curly cale and beetroot for the validation of the procedure		H_2O_2, HNO_3	CV-AFS	b) 0-50	Total Hg
			Closed Microwave Dig.	H_2O_2, HF, HNO_3	ICP-MS	b) 0-50	Total Cd
			Closed Microwave Dig.	H_2O_2, HF, HNO_3	ICP-MS	b) 0-50	Total Pb
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	b) 0-50	Total As
L126	b) No	BCR Lichen 482 for the calibration of instruments, for the validation of the procedure		HNO_3	CV-AFS	d) 250-1000	Total Hg
			Closed Microwave Dig.	HNO_3	ET-AAS	d) 250-1000	Total Cd
			Closed Microwave Dig.	HNO_3	ET-AAS	d) 250-1000	Total Pb
			Closed Microwave Dig.	HNO_3	ET-AAS	d) 250-1000	Total As
L127	b) No	b) No		HCl, HNO_3	CV-AAS	d) 250-1000	Total Hg
			Closed Microwave Dig.	HNO_3	ETAAS	d) 250-1000	Total Cd
			Closed Microwave Dig.	HNO_3	ETAAS	d) 250-1000	Total Pb
L128	b) No	Material from other Proficiency Test For the validation of the procedure	Pressure Bomb Dig.	H_2O_2, HNO_3	FIMS	b) 0-50	Total Hg
			Pressure Bomb Dig.	H_2O_2, HNO_3	ICP-MS	b) 0-50	Total Cd
			Pressure Bomb Dig.	H_2O_2, HNO_3	ICP-MS	b) 0-50	Total Pb
L129	b) No	IMEP 114 for the validation of the procedure	Dry Ashing		AAS AMA 254	b) 0-50	Total Hg
			Closed Microwave Dig.	H_2O_2, HNO_3	ET-AAS	b) 0-50	Total Cd
			Closed Microwave Dig.	H_2O_2, HNO_3	ET-AAS	b) 0-50	Total Pb
			Dry Ashing	HCl, HNO3	HG-AAS	b) 0-50	Total As
L130	EPA-method (modified) 200.7 (ICP-AES) and 200.8 (ICP-SFMS)	b) No	Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total Hg
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total Cd
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total Pb
			Closed Microwave Dig.	H_2O_2, HNO_3	ICP-MS	c) 50-250	Total As

Annex 12: Techniques used and respective LoDs

LAB ID	Total As		Total Cd		Total Pb		Total Hg	
	Technique	LODs (mg/kg)	Technique	LODs (mg/kg)	Technique	LODs (mg/kg)	Technique	LODs (mg/kg)
L101	ICP-QMS	0.01	ICP-QMS	0.0004	ICP-QMS	0.006	ICP-QMS	0.007
L102	ICP-MS	0.0002	ICP-MS	0.0001	ICP-MS	0.0014	ICP-MS	0.005
L103	HG-AAS	0.056	ET-AAS	0.0025	ET-AAS	0.025	CV-AAS	0.004
L104		0.05		0.05		0.2		0.01
L105	ICP-MS	0.075	ETAAS	0.006	ICP-MS	0.015	CV-AAS	0.006
L107			ET-AAS		ET-AAS			
L108	HG-AAS	0.011	ET-AAS	0.001	HG-AAS	0.006	Direct mercury analyzer	0.0003
L109	ETAAS	0.85	ETAAS	0.25	ETAAS	1.8	TDA-AAS	0.034
L110	ICP-AES	0.05	ICP-AES	0.001	ICP-AES	0.01	AMA 254	0.005
L111	ICP-MS	0.25	ICP-AES	0.01	ICP-AES	0.5	ICP-MS	0.052
L112	ICP-MS	0.1	ICP-MS	0.01	ICP-MS	0.06	ICP-MS	0.03
L113	ICP-QMS		ICP-QMS		ICP-QMS		Direct Mercury Analyzer	
L114	ICP-QMS		ICP-QMS		ICP-QMS		ICP-QMS	
L115	ICP-QMS	0.006	ICP-QMS	0.006	ICP-QMS	0.09	AMA	0.0003
L116			FAAS		FAAS		AMA 254	
L117	ICP-MS	0.05	ICP-MS	0.02	ICP-MS	0.2	ICP-MS	0.1
L118	HG-AAS	0.06	ETAAS	0.013	ETAAS	0.25	CV-AAS	0.15
L119	ICP-MS	0.00005	ICP-MS	0.00001	ICP-MS	0.00002	Direct mercury analysis	0.008
L120	H-AAS	0.04	ET-AAS	0.04	ET-AAS	0.05	CV-AAS	0.05
L121	ICP-MS	0.02	ICP-MS	0.001	ICP-MS	0.01	AAS	0.005
L122	ICP-QMS		ICP-QMS		ICP-QMS		ICP-QMS	
L123	HG-AAS	0.3	FAAS	0.15	FAAS	2	CV-AAS	0.003
L124	HG-AAS FIAS	0.01	ETAAS	0.01	ETAAS	0.02	CV-AAS	0.002
L125	ICP-MS	0.03	ICP-MS	0.02	ICP-MS	0.03	CV-AFS	0.01
L126	ET-AAS		ET-AAS		ET-AAS		CV-AFS	
L127			ETAAS		ETAAS		CV-AAS	
L128			ICP-MS	0.08	ICP-MS	0.52	FIMS	
L129	HG-AAS	0.002	ET-AAS	0.001	ET-AAS	0.002	AAS AMA 254	0.001
L130	ICP-MS	0.01	ICP-MS	0.003	ICP-MS	0.02	ICP-MS	0.01

European Commission
EUR 26195 EN – Joint Research Centre – Institute for Reference Materials and Measurements

Title: **IMEP-117: Determination of total As, Cd, Pb, and Hg in compound feed - Interlaboratory Comparison Report**

Author(s): Ioannis Fiamegkos, Fernando Cordeiro, Piotr Robouch, Håkan Emteborg, Hanne Leys, Aneta Cizek-Stroh, Beatriz de la Calle

Luxembourg: Publications Office of the European Union

2013 – 39 pp. – 21.0 x 29.7 cm

EUR – Scientific and Technical Research series – ISSN 1831-9424 (online)

ISBN 978-92-79-33385-9 (pdf)

doi:10.2787/80103

Abstract
The Institute for Reference Materials and Measurements of the Joint Research Centre, a Directorate General of the European Commission, operates the European Union Reference Laboratory for Heavy Metals in Feed and Food. One of its core tasks is to organize interlaboratory comparisons among appointed National Reference Laboratories. This report presents the results of a proficiency test, IMEP-117 of the EURL-HM which focused on the determination of total As, Cd, Pb and Hg in compound feed in support to Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed.
The test material used in this exercise was a commercially available compound feed for cats which was spiked after the appropriate processing, bottled, labelled, and dispatched to the participants on the 23rd of May 2013. Three laboratories with demonstrated experience in the field provided results to establish the assigned values (X_{ref}). The standard uncertainties associated to the assigned values (u_{ref}) were calculated according to ISO/IEC Guide 98:2008 (GUM) and ISO 13528:2005.
Laboratory results were rated with *z*- and zeta (ζ-) scores in accordance with ISO 13528:2005. The standard deviation for proficiency assessment (σ_p), also called target standard deviation, was set to 10 % of the assigned value, for the measurands investigated.
The percentage of satisfactory z-scores was above 79 % for all measurands showing an overall adequate performance for European National Reference Laboratories assuring compliance towards the European legislation related to the determination of the investigated compound feed contaminants.

LA-NA-26195-EN-N

As the Commission's in-house science service, the Joint Research Centre's mission is to provide EU policies with independent, evidence-based scientific and technical support throughout the whole policy cycle.

Working in close cooperation with policy Directorates-General, the JRC addresses key societal challenges while stimulating innovation through developing new standards, methods and tools, and sharing and transferring its know-how to the Member States and international community.

Key policy areas include: environment and climate change; energy and transport; agriculture and food security; health and consumer protection; information society and digital agenda; safety and security including nuclear; all supported through a cross-cutting and multi-disciplinary approach.

Publications Office

ISBN 978-92-79-33385-9