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Ioannis Fiamegkos,
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Hanne Leys, Bibi Kortsen,
Beatriz de la Calle

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**Contact information**
Ms. Beatriz de la Calle
Joint Research Centre, Retieseweg 111, 2440 Geel, Belgium
E-mail: maria.de-la-calle@ec.europa.eu
Tel.: +32 (0) 14 571252
Fax: +32 (0) 14 571865

http://irmm.jrc.ec.europa.eu/
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Ioannis Fiamegkos (c)
Fernando Cordeiro (c)
Piotr Robouch *(c)*
Håkan Emteborg (c)
Jean Charoud-Got (c)
Hanne Leys (c)
Bibi Kortsen (d)
Beatriz de la Calle (a,b,c)

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

IMEP®

## Summary

The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre, a Directorate General of the European Commission, operates the International Measurement Evaluation Programme (IMEP). IMEP organizes interlaboratory comparisons (ILCs) in support to EU policies. This report presents the results of the proficiency test (PT) which focused on the determination of total Cd, Pb, As, Hg and Sn in feed premixes according 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 commercially available feed premix which after the appropriate processing was bottled, labelled, numbered accordingly and dispatched to the participants on the 9$^{th}$ of July 2012. Fifty laboratories from 22 countries registered to the exercise of which 45 reported results and answered the respective questionnaire. Laboratories were asked to perform two or three independent measurements and to report the mean, the associated uncertainty, the coverage factor of the associated uncertainty and the technique used to perform the measurements. Laboratory results were rated using z- and ζ-scores (zeta-scores) in accordance with ISO 13528. The assigned values ($X_{ref}$) for the measurands were determined as the mean of the values reported by two expert laboratories both of them National Metrology Institutes (NMI). These laboratories were the Federal Institute for Materials Research and Testing (BAM) (Germany) and LGC Limited (UK). The stability (isochronous) and homogeneity study was conducted by ALS Scandinavia AB (Sweden). The standard deviation for proficiency assessment ($\hat{\sigma}$), also called target standard deviation, was set to 15 % of the assigned value, for the analysis investigated.

The results obtained by the participants were optimum in the case of total Cd and less satisfactory for total As and total Pb. For total Sn 16 participants reported results, from which one third scored satisfactorily. Twenty one participants reported results for total Hg although, the expert laboratories reported that the mass fraction for that measurand was below their limit of detection. Hence, no scoring was provided for total Hg.

## IMEP support to EU policy

IMEP is owned by the JRC – IRMM and provides support to the European measurement infrastructure in the following ways:

IMEP **distributes metrological traceability** from the highest level down to the routine laboratories. Laboratories can benchmark their measurement result against the IMEP reference value which is established according to metrological best practice.

IMEP helps laboratories to assess their estimate of **measurement uncertainty**. Participants are invited to report the uncertainty of their measurement result. IMEP integrates the estimate into the scoring, and provides assistance for the interpretation.

IMEP supports EU policies by **organising interlaboratory comparisons** (ILC) in the frame of specific EU legislation, or on request of a specific Directorate-General. IMEP-36 provided specific support to the following stakeholders:

- To the European Co-operation for Accreditation (EA), Interamerican Accreditation Cooperation (IAAC) and Asia Pacific Laboratory Accreditation Cooperation (APLAC) in the frame of a formal collaboration on a number of metrological issues, including the organisation of interlaboratory comparisons. Mr. Paul Greenwood from the United Kingdom Accreditation Service liaised between EA and IMEP for this ILC, Mrs. Barbara Belzer for IAAC and Aparna Dawan for APLAC (Annexes 1-3) This report does not discern the EA nominees from the other participants. Their results are however summarised in a separate report to EA.
- To the European Union Reference Laboratory for Heavy Metals in Feed and Food (EU-RL-HM) in the frame of the support to the National Reference Laboratories (NRLs). The exercise was announced to the network of NRLs who were invited to distribute the information between control laboratories in their respective countries.

IMEP is accredited according to ISO 17043:2010.

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# 1 Introduction

The IMEP-36 exercise was organized aiming to assess the performance of the food control laboratories on the determination of total cadmium, lead, arsenic, mercury and tin in feed premixes. This proficiency test (PT) was carried out in collaboration with the European Union Reference Laboratory for Heavy Metals (EU-RL-HM), who organised in parallel the PT IMEP-114 for its network of National Reference Laboratories (NRLs), using the same test material. The results submitted to IMEP-114 are not discussed in this report.

Both exercises were 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 "premixtures" as the "mixtures of additives or mixtures of one or more additives with substances used as carriers, intended for the manufacture of feedingstuffs". The various commercially available feed premixes as well as the diversity of production ways may lead to contaminated and/or dangerous end-products, introducing undesirable substances into the food chain. The Directive and its amendments [1] set maximum levels for undesirable substances in animal feed and feed premixes (organic and inorganic). Regarding heavy metals, limits are set only for lead (200 mg $kg^{-1}$) and cadmium (15 mg $kg^{-1}$).

From the analytical point of view the scarce publications found in scientific literature about the analysis of feed premixes are dealing with the determination of anticoccidials [2-4], antibiotics [5], vitamins [6-8], phthalates [9], sulfonamides [10] and iodinated casein [11], but not with the analysis of heavy metals. The determination of essential metals (e.g. Zn, Cu, Mn and Fe) in premix samples was reported for the evaluation of enriched preparations of animal feeds [12].

IMEP-36 was organized to check the analytical capabilities of the European and International food control laboratories to determine low concentrations of total As, Cd, Pb, Hg and Sn in feed premixes.

## *1.1 Scope*

The scope of this PT was to test the competence of the participating laboratories to determine total As, Cd, Pb, Hg and Sn in feed premixes. Measurements were to be done on a commercially available feed premix that was processed by the IRMM to reach adequate homogeneity.

The assessment of the measurement results is undertaken on the basis of requirements laid down in legislation [1], and follows the administrative procedure and

logistics of the International Measurement Evaluation Program (IMEP) of the IRMM of the European Commission Directorate Joint Research Centre. IMEP is accredited according to ISO 17043:2010 [13]. The designation of this PT is IMEP-36.

## 2. Set up of the exercise

An invitation letter for participation was sent to the EA, IAAC and APLAC coordinators (Annex 1-3) on the 1st of June 2012 for distribution to nominated and interested laboratories. A web announcement (Annex 4) was made for the exercise on the IMEP webpage on the 7th of June 2012.

Laboratories could register until the 3rd of July and the test items were dispatched to the participants on the 9th of July 2012. The reporting deadline was the 15th of September 2012.

### *2.1 Confidentiality*

The following confidentiality statement was made to EA, IAAC and APLAC: *"Confidentiality of the participants and their results towards third parties is guaranteed".* In the case of EA the following was added: "*However, IMEP will disclose details of the participants that have been nominated by EA to you. The EA accreditation bodies may wish to inform the nominees of this disclosure".*

### *2.2 Test material - Preparation*

The commercially available feed premix (for dairy cattle) was purchased at the local market in Geel, Belgium. The producer reported the following composition:

| |
|---|
| ***Genetically modified soya*** |
| ***Analytical Constituents****: 0.22% Ca; 3.95%Total P; 8% Mg; 3.01% Na* |
| ***Nutritional additives****: 1000000 UI kg$^{-1}$ Vitamine A; (E671) 200000 UI kg$^{-1}$ Vitamine D3; 4400 mg kg$^{-1}$ Vitamine E; 2000 mg kg$^{-1}$ $C_5H_{14}ClNO$; 20 mg kg$^{-1}$ $Ca(IO_3)_2$/ $I_2$; 15 mg kg$^{-1}$ $CoSO_4{\cdot}7H_2O$/ Co; 1000 mg kg$^{-1}$ $CuSO_4{\cdot}5H_2O$/ Cu, 1250 mg kg$^{-1}$ MnO/Mn; 2500 mg kg$^{-1}$ $ZnSO_4(H_2O)$/Zn; 40 mg kg$^{-1}$ $Na_2SeO_3$/Se; 100 mg kg$^{-1}$ BHT.* |

The 25-kg bag of the feed premix was opened and 7.5 kg of this material was first crushed using a jaw-crusher Retsch (Haan, Germany). The distance between the jaws was about 1 mm in order to break the pellets into smaller pieces. Next the resulting material was milled in a 100 UPZ mill (Hosokawa Alpine, Augsburg, Germany) without a sieve insert. By subsequent sieving over a 250 µm nylon sieve and re-milling of the coarse fractions using the 100 UPZ mill 7.05 kg was finally obtained of the fine fraction.

The resulting 7.05 kg were then carefully mixed for 30 minutes in a Dynamix CM200 3D-mixer (WAB, Basel, Switzerland) before filling. The filling of 20 g per unit into 60 ml amber glass bottles was achieved using a vibrating feeder (Fritsch, Idar-Oberstein, Germany) and a balance. After filling the glass bottle was closed with a PE-insert and a screw-cap lid. In total 210 units of this powder were filled and labelled both for IMEP-36 and IMEP-114.

Analysis of the particle size and water revealed that the top-particle size in the powder was smaller than 365 µm. Water content was about 10 % (m/m) as measured by Karl Fischer titration (KFT). No attempt was made to dry the material further. Based on the Karl Fischer data an oven method was devised for the benefit of the participants in this inter-comparison. The oven method if correctly applied would thereby result in a mass loss corresponding to the water content as measured by KFT.

### *2.3 Distribution*

Samples were dispatched to the participants by IRMM on the 9$^{th}$ of July 2012. Each participant received:

a) One bottle containing approximately 20 g of powdered feed premix

b) An accompanying letter (Annex 5).

c) A "Confirmation of Receipt" form to be sent back to IRMM after receipt of the test material (Annex 6).

d) A summary of the questionnaire the laboratory would be prompted to answer on-line after reporting the results (Annex 7).

Concrete instructions were given to all participants in the above mentioned letter accompanying the test material. The measurands and matrix were defined as “Total Cd, Pb, Hg, As and Sn in Feed Pre-mixes following Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed”.

Laboratories were asked to perform two or three independent measurements and to report the mean, the associated uncertainty, the coverage factor of the associated uncertainty and the technique used to perform the measurements. The measurement results were to be corrected for (i) recovery and (ii) moisture, following the procedure described therein. 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 perform the drying using the drying recipe provided and report all data as based on dry-mass.

The results were to be reported in a special on-line form for which each participant received an individual access code. A questionnaire was attached to this on-line form (Annex 8).

The laboratory codes were given randomly and communicated to the participants by e-mail. The assigned values were disclosed to the participants in an e-mail sent on the 15th of November 2012.

### *2.4 Homogeneity and stability*

The homogeneity and stability studies were performed by ALS Scandinavia AB (Luleå, Sweden). Homogeneity was evaluated according to ISO 13528 [14]. The material proved to be adequately homogeneous for all measurands under study (Annex 9). The contribution from homogeneity *($u_{bb}$)* to the uncertainty of the reference value *($u_{ref}$)* was calculated using SoftCRM [15].

The stability study was conducted following the isochronous approach [16, 17]. The evaluation of the stability of the test item was made using the software SoftCRM [15]. The material proved to be stable for the 10 weeks that elapsed between the dispatch of the samples and the deadline for submission of results, for total Cd, Pb, As, Hg and Sn (Annex 9).

## 3. Reference values and their uncertainties

### *3.1 Assigned value $X_{ref}$*

The assigned values for the five measurands were determined by BAM and LGC.

BAM used a microwave-assisted digestion with a mixture of $HNO_3$/HCl/HF and inductively coupled plasma–mass spectrometry (ICP-MS) for the analysis. Values were reported for all measurands except for total Hg for which after analysing the test item with ICP-MS, advanced mercury analyzer (AMA-254) and cold vapour–atomic fluorescence spectrometry (CV-AFS) the laboratory reported less than 1.5 µg $kg^{-1}$. LGC Ltd used Microwave-assisted digestion with a mixture of $HNO_3$/HCl/$H_2O_2$/HF and ICP-MS for the metal determination. Values were reported in all cases except for total Hg for which the laboratory reported less than 0.8 µg $kg^{-1}$ based on cold vapour – inductively coupled plasma mass spectrometry (CV-ICP-MS) analysis.

### *3.2 Associated uncertainty $u_{ref}$*

The associated uncertainties ($u_{ref}$) of the assigned values in the milled feed premix samples were calculated combining the uncertainty of the characterization ($u_{char}$) with the contributions for homogeneity ($u_{bb}$) and stability ($u_{st}$):

$$u_{ref} = \sqrt{u_{char}^2 + u_{bb}^2 + u_{st}^2}$$

For all measurands except Hg, $u_{char}$ was estimated combining the reported uncertainties by BAM ($u_{BAM}$) and LGC ($u_{LGC}$) according to the ISO Guide for the Expression of Uncertainty in Measurement (GUM) [18], as follows:

$$u_{char} = \frac{1}{2}\sqrt{u_{LGC}^2 + u_{BAM}^2}$$

### *3.3 Target standard deviation* $\hat{\sigma}$

On the basis of previous experience for this type of analysis the standard deviations for proficiency assessment $\hat{\sigma}$ (also called target standard deviation) was set to 15 % of the respective assigned values.

An overview of all reference values ($X_{ref}$, $u_{ref}$, $U_{ref}$, $\hat{\sigma}$) is given in Table 1.

**Table 1 -** *Assigned values, their associated uncertainties and target standard deviations for the measurands of this ILC (all values in mg kg$^{-1}$). - Mercury results are not included as the $X_{ref}$ was found by the two expert laboratories to be "less than" their respective limit of detection.*

| | | Total As ($X_n \pm U_n$) | Total Cd ($X_n \pm U_n$) | Total Pb ($X_n \pm U_n$) | Total Sn ($X_n \pm U_n$) |
|---|---|---|---|---|---|
| Certifiers | BAM | 1.96 ± 0.09 | 1.12 ± 0.06 | 0.613 ± 0.041 | 0.85 ± 0.07 |
| | LGC | 1.911 ± 0.077 | 1.103 ± 0.027 | 0.658 ± 0.036 | 0.733 ± 0.017 |
| $X_{ref}$ | | 1.936 | 1.112 | 0.636 | 0.792 |
| $u_{char}$ | | 0.030 | 0.016 | 0.014 | 0.018 |
| $u_{bb}$ | | 0.046 | 0.008 | 0.018 | 0.021 |
| $u_{st}$ | | 0.102 | 0.021 | 0.022 | 0.047 |
| $u_{ref}$ | | 0.116 | 0.028 | 0.032 | 0.055 |
| $U_{ref}$ (k=2)* | | 0.231 | 0.056 | 0.063 | 0.109 |
| $\hat{\sigma}$ (Set 15 %) | | 0.290 | 0.167 | 0.095 | 0.119 |

*$X_{ref}$ 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 %.*

## 4 Evaluation of results

### *4.1 Scores and evaluation criteria*

Individual laboratory performance was expressed in terms of z- and $\zeta$-scores in accordance with ISO 13528 [14].

$$z = \frac{x_{lab} - X_{ref}}{\hat{\sigma}} \qquad \text{and} \qquad \zeta = \frac{x_{lab} - X_{ref}}{\sqrt{u_{ref}^2 + u_{lab}^2}}$$

where: $x_{lab}$ is the measurement result reported by a participant
$X_{ref}$ is the reference value (assigned value)
$u_{ref}$ is the standard uncertainty of the reference value
$u_{lab}$ is the standard uncertainty reported by a participant
$\hat{\sigma}$ is the standard deviation for proficiency assessment

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

| | | |
|---|---|---|
| \|score\| ≤ 2 | satisfactory result | (green in the tables of Annexes 10 - 14) |
| 2 < \|score\| < 3 | questionable result | (orange in the tables of Annexes 10 - 14) |
| \|score\| ≥ 3 | unsatisfactory result | (red in the tables of Annexes 10 - 14) |

The z-score compares the participant's deviation from the reference value with the target standard deviation for proficiency assessment ($\hat{\sigma}$) used as common quality criterion. $\hat{\sigma}$ 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 √3, as recommended by Eurachem and CITAC [19].

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 ($\hat{\sigma}$) accepted for the PT. If $u_{lab}$ is smaller than $u_{min}$, 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 $u_{lab} > u_{max}$, 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.

### *4.2 General observations*

From the 50 laboratories (22 countries) that have registered, 45 submitted their results and answered the associated questionnaire (figure 1).

Those reporting "less than" and "0" values were not included in the evaluation (Table 2). However, reported "less than" values were compared with the corresponding $X_{ref} - U_{ref}$ values. If the reported limit value is lower than the corresponding $X_{ref} - U_{ref}$, this statement is considered incorrect, since the laboratory should have detected the respective element. In this exercise laboratory L12 reported incorrectly "less than" 0.2 mg $kg^{-1}$ for total Lead ($X_{ref}$-$U_{ref}$ = 0.57 mg $kg^{-1}$) and L02 "less than" 0.05 mg $kg^{-1}$ for total Tin ($X_{ref}$-$U_{ref}$ = 0.68 mg $kg^{-1}$).

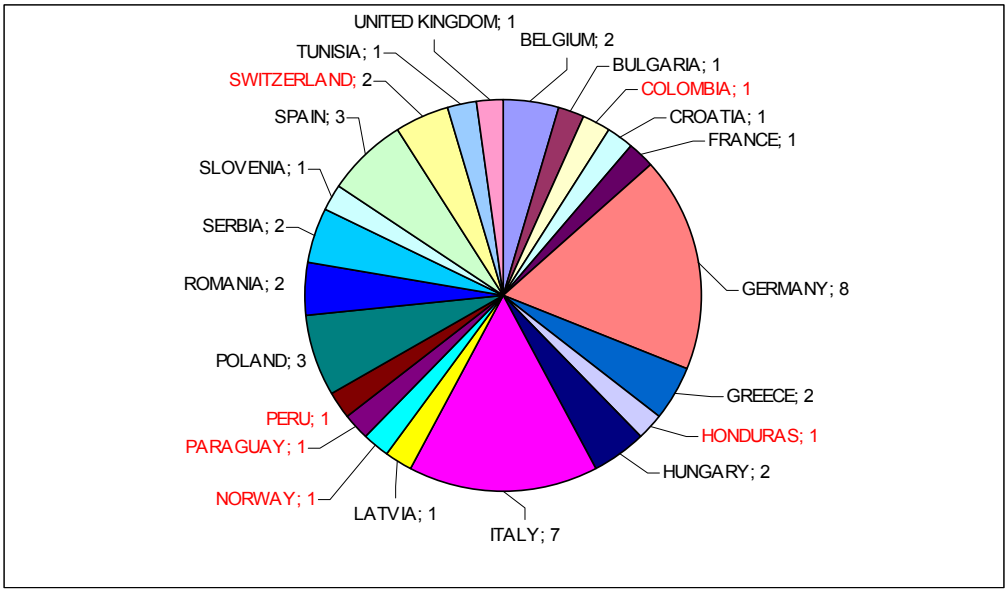


***Figure 1**: Country distribution in IMEP-36 based on number of participants(45) having submitted results. Non-EU countries are indicated in red.*

**Table 2 -** *Number of reported results,"less than" values.*

| | Total As | Total Cd | Total Pb | Total Sn | Total Hg |
|---|---|---|---|---|---|
| Number of participants reported evaluable results | 40 | 44 | 40 | 16 | 21 |
| "less than" | 1 | - | 4 | 5 | 14 |

### *4.3 Laboratory results and scorings*

The results as reported by the participants for total As, Cd, Pb, Hg and Sn are listed in Annexes 10 to 14, together with the z- and ζ-scores. The Kernel distribution plots, obtained using a software tool developed by AMC [20] are also presented in the respective figures therein.

The overall performance of the participants regarding the z- and ζ-scores, is summarised in Figure 2.

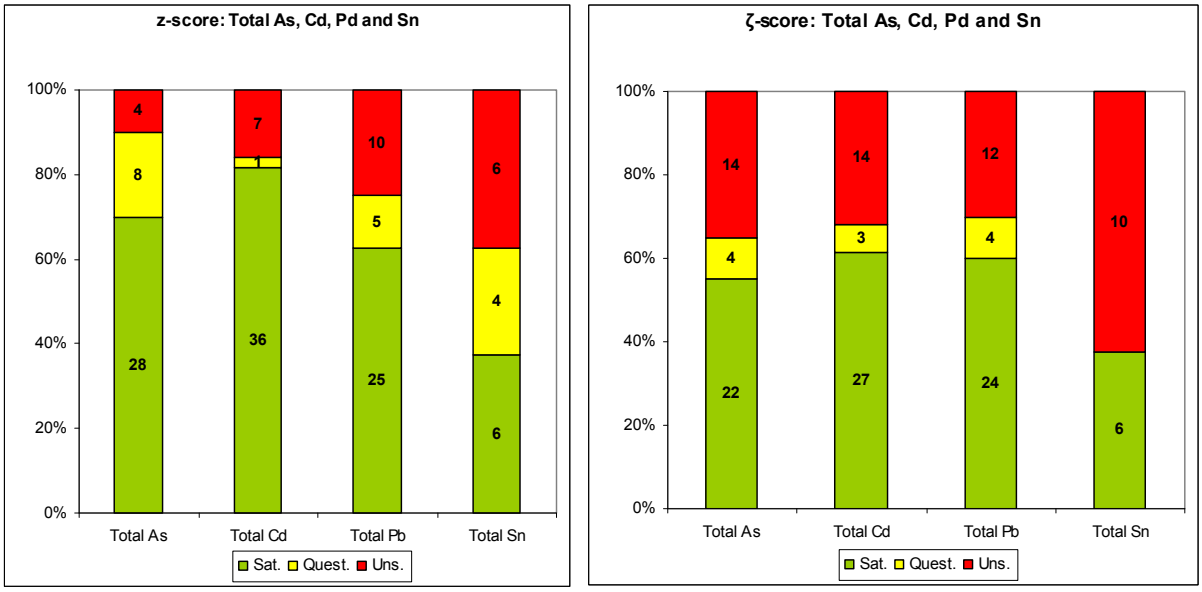


***Figure 2:*** *Number and percentages of laboratories with satisfactory, questionable and unsatisfactory scores. (The numbers on the bars correspond to the exact number of laboratories in a certain scoring category).*

The values provided by the two expert laboratories for total As, Cd, Pb and Sn were in good agreement between each other. The reference values ($X_{ref}$) were calculated as the average of the two reported by the expert laboratories according to the ISO 13528. The assigned and reported values for total Cd and total Pb in the samples were well under the limits set by the legislation. For total As and total Sn, where no limit are set by the legislation, the values provided by the certifiers are well above the detection limits of the commonly used methods of analysis.

The results provided by the participants demonstrate that the feed premix used as test item proved to be a challenging matrix. The moisture content reported by the

participants was of the order of 9.6 ± 1,9 %, ALS reported 11.0 ± 0.2 % (n=30). The overall performance for the results reported for total Cd (44 participants) was adequate since satisfactory z-scores were obtained by 80 % of the participants (ζ-scores: 60 %). For total As (40 participants) satisfactory z-scores were obtained by more than 65 % of the participants (ζ-scores: 58 %). In the case of total Pb, (40 laboratories reported results) satisfactory z- and ζ-scores were achieved by more than 60 %. For all the measurands the majority of the questionable and unsatisfactory results were due to underestimation of the mass fraction.

Some laboratories might have used mild digestion methods which did not allow a quantitative determination of the total mass fraction. In the last update of the Directive 2002/32/CEC the footnote on partial extraction with 5 % $HNO_3$ acid at boiling temperature has been deleted. Laboratories must be aware that in some types of feed matrices, such as those containing kaolinitic clay, the total mass fraction might be significantly different from that obtained with mild digestion procedures.

For total Sn the performance of the participating laboratories was not satisfactory. One third of the participants reported evaluable results, and 5 reported "less than". From the 16 laboratories that delivered a value for total Sn only 6 scored satisfactorily, one overestimated the result and the rest (10) underestimated it, as can also be seen in the Kernel density plot (Annex 13). It should also be noted that from the 10 laboratories reporting questionable and/or unsatisfactory results, 7 were using reference materials for the validation of the method applied and/or for the calibration of the instrument. In addition, there seems to be a correlation between the Sn concentration and the digestion procedure used. As indicated in the literature, the efficiency of Sn extraction by this kind of matrices increases when microwave acid digestion is used, also the addition of HF improves the recovery [21]. In the IMEP-36 exercise 3 out of 5 laboratories that scored satisfactorily have used microwave acid extraction (one with the addition of HF), while for the remaining two no clear information was given. Nevertheless, the low number of reported values for this measurand does not allow any deeper analysis of the influencing factors and hence no conclusive remarks can be extracted.

As mentioned, the expert laboratories were not able to provide results for total Hg in the test item. Nevertheless, a significant number of participants (21) reported values for total Hg ranging from 0.7 μg $kg^{-1}$ to 0.4 mg $kg^{-1}$. Ten participants did not report a value while 14 reported "less than". A first remark is that half of the reported values for total Hg are lower than almost all the LoDs reported by the participants that stated a "less than" value. Also very low uncertainty values were reported by these laboratories. One could assume that those very low values correspond even to the blank values given by the electronic noise of the instrument and to their associated standard deviation.

Secondly, the higher values reported for total Hg, in most cases, were acquired by analysing the sample by ICP-MS. Interestingly, a participating laboratory in the parallel exercise IMEP-114 communicated the following [private communication]:

*"We analyzed the Hg content in the Imep 114, using the new bottles you send us. In line with the reported value for the PT last time, we found concentrations < lod (analyzed using a mercure aparatus CVAFS).*
*On the ICPMS we found the following:*
*Based on mass 200 and 202 we had an interference of Wolfram oxide. This interference increased the Hg concentration. If we use mass 201 for the calibration of Hg, we do not have interference of W oxide, and <LOD was found in the imep 114 sample. This results show you the error due to the interference even when the collision cell (reactor cell of ICPMS) was used to remove those interferences."*

One last remark is that the number of satisfactory ζ -scores is lower than that of the z-scores. Thus, laboratories should put effort in making a rational estimation of the uncertainty associated to their measurements.

### *4.4 Additional information extracted from the questionnaire*

Additional information was gathered from the questionnaire filled in by the participants (Annex 6).

Twenty one participants performed the analysis following an official method. The information provided by the laboratories about their methods of analysis is summarised in Annex 15. With the exception of total Hg, the influence of the techniques used did not correlate to any of the reported results.

Thirty three laboratories reported the use of reference materials for the purpose of method validation and/or calibration and these materials are presented in Table 3. Attention must be paid to the CRMs used since they must match the matrix of the test samples as much as possible. Although there are no feed premix reference materials and the participants used a variety of RMs for the purpose of this PT no safe conclusions can be drawn out about their usefulness regarding the specific analysis.

Fifteen laboratories corrected their results for recovery, 23 did not and 7 didn't reply. Sixteen laboratories reported the recovery used to correct their results. The recoveries reported were in the range 70-107 %. 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 engaged [22].

**Table 3 -** *List of reference materials used by the participants in IMEP-36 for method validation and/or calibration purposes.*

| LCode | Does your laboratory use a reference material for this type of analysis? | If yes, which one(s) | Is the material used for the validation of procedures? | Is the material used for calibration of instruments? |
|---|---|---|---|---|
| L03 | yes | Feed material from Ministery of Agriculture | no | no |
| L05 | yes | Pig feed BCR-709, Soya bean NCS ZC73011, Milk powder NCS ZC73015 | yes | yes |
| L07 | yes | CRM from BIPEA and FAPAS | yes | no |
| L08 | yes | BCR - 191 Brown bread | yes | no |
| L09 | yes | Phosphate procured from CEN validation studies, Bipea samples type premixes we use as internal reference material | yes | no |
| L11 | yes | FAPAS | yes | no |
| L13 | yes | BCR 185R *(bovine liver)* | yes | no |
| L15 | yes | BgVV tomate puree as QS standard for Sn and collaborative tested feed materials from studies of VDLUFA, ALVA, IAG | no | no |
| L16 | yes | ILC sample material | yes | no |
| L17 | yes | CRM BCR-032 *(Moroccan phosphate rock)* | yes | no |
| L18 | yes | CRM's IRMM, NIST, IAEA | yes | no |
| L20 | yes | interlaboratory comparisons excess | no | no |
| L21 | yes | mineral feed test material from a former LGC interlaboratory comparison | yes | no |
| L22 | yes | MERCK standard solution | | yes |
| L23 | yes | CERTIFICATED STANDARD SOLUTIONS | yes | yes |
| L26 | yes | Samples from ring tests | yes | yes |
| L27 | yes | Oyster Tissue, Tort-2, Bovine Liver | yes | no |
| L28 | yes | sample of interlaboratory comparison | no | no |
| L29 | yes | Fapas and CREAA | yes | no |
| L30 | yes | INTERLABORATORY THEM APPROVED | yes | yes |
| L31 | yes | inorganic ventures, 71A, 71B, Hg of 10 ppm | | |
| L35 | yes | DORM-3 (NRC), NIST | yes | yes |
| L36 | yes | This one obteined from intercomparation exercices | yes | yes |
| L37 | yes | BCR skim milk powder | no | no |
| L38 | yes | | yes | no |
| L39 | yes | INCT-MPH-2, NCS ZC 80002b( wheat flour), NCS ZC73009(GSB-2 wheat), NCS ZC73010 mealie | yes | no |
| L40 | yes | NCS ZC 73013 *(spinage)* | yes | no |
| L42 | yes | IAEAV 185R DORM TORT 150 .... | yes | no |
| L45 | yes | | yes | yes |
| L46 | yes | FAPAS | yes | no |
| L47 | yes | the rest of PT material in the last year | no | no |
| L48 | yes | IRMM | yes | yes |
| L49 | yes | Material from interlaboratory comparisons | yes | no |

*In italics information about the matrix of used CRM are given when applicable*

All participants but 4 (L08, L30, L44 and L48) corrected their results for the moisture content, determined using the protocol described in the accompanying letter (Annex 5).

Various approaches were used to scrutinise the measurement uncertainty (Table 4). Eighteen laboratories usually report uncertainty to their customers while 21 do not.

**Table 4 -** *Approaches used by the participants in IMEP-36 to estimate the uncertainty of their measurements.*

| Approach followed for uncertainty calculation | Number of labs. |
|---|---|
| Uncertainty budget calculated according to ISO-GUM | 9 |
| Known uncertainty of the standard method | 5 |
| Uncertainty of the method as determined by in-house validation | 22 |
| Measurement of replicates (i.e. precision) | 9 |
| Estimation based on judgement | 3 |
| Use of intercomparison data | 8 |
| Other | 5 |

Regarding the experience of the laboratories with this kind of analysis 35 participants answered that they carry out this type of analysis on a regular basis while 4 do not.

Thirty eight participants stated that they have a quality system in place based on ISO 17025. In 5 cases the quality system is also based on ISO 9000. Thirty two participants are accredited for the methods of analysis used in this exercise, although one laboratory indicated that they are not accredited for Sn analysis and 2 other reported that they do not analyse Sn routinely. Most of the laboratories (36) regularly take part in PTs.

## 5 Conclusions

From the results obtained for the IMEP-36 exercise, the participating laboratories reported satisfactorily for total Cd. Total As and Pb proved to be difficult measurands since less satisfactory results were obtained with an obvious trend to underestimation of the assigned values. This could be attributed to inadequate digestion/extraction procedures. Only one third of the participants reported results for total Sn from which only 33 % obtained satisfactory score. The digestion method is evidently influencing the Sn recovery from the matrix. Twenty one participants reported results for total Hg although the expert laboratories stated that the mass fraction for that measurand was below the limit of detection of the method used. Interference by tungsten oxide could be the explanation for those high results.

Once again the need for an extra effort was identified in the evaluation of uncertainties associated to the results, since the number of questionable and unsatisfactory ζ-scores is systematically higher than those of z-scores for all analytes. The measurement uncertainty is of paramount importance in cases of litigation and so its sound calculation is fundamental for control laboratories.

## 6 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 below are kindly acknowledged.

| Organisation | Country |
|---|---|
| FAVV - FLVVG | BELGIUM |
| SCK-CEN | BELGIUM |
| SGS Bulgaria Ltd | BULGARIA |
| IVONNE BERNIER LABORATORIO LTDA | COLOMBIA |
| EUROINSPEKT CROATIAKONTROLA d.o.o. | CROATIA |
| CONSEIL GENERAL DE VENDEE | FRANCE |
| Bavarian health and food safety authority | GERMANY |
| Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit | GERMANY |
| Institut fuer Hygiene und Umwelt | GERMANY |
| LTZ Augustenberg | GERMANY |
| University of Hohenheim | GERMANY |
| BLS Analytik GmbH & Co. KG | GERMANY |
| Berghof Analytik + Umweltengineering GmbH & Co.KG | GERMANY |
| Landeslabor Berlin-Brandenburg | GERMANY |
| FOOD ALLERGENS LABORATORY | GREECE |
| AGROLAB SA | GREECE |
| Laboratorio Nacional de Análisis de Residuos, LANAR-OIRSA | HONDURAS |
| Food Analytica Ltd. | HUNGARY |
| Central Agricultural Office | HUNGARY |
| SILLIKER ITALIA SPA | ITALY |
| BIOCHEMIELAB SRL | ITALY |
| Istituto Zooprofilattico Sperimentale - Puglia e Basilicata | ITALY |
| NEOTRON SPA | ITALY |
| Istituto Caporale Teramo | ITALY |
| Istituto Zooprofilattico Sperimentale delle Regioni Lazio e Toscana | ITALY |
| IZS SARDEGNA | ITALY |
| Ltd LATSERT | LATVIA |
| National Institute of Nutrition and Seafood Research | NORWAY |
| Díaz Gill Medicina Laboratorial S.A. | PARAGUAY |
| Instituto Tecnológico pesquero del Perú | PERU |
| Wojewódzki Inspektorat Weterynarii w Białymstoku | POLAND |
| Zakład Higieny Weterynaryjnej | POLAND |
| Wojewodzki Inspektorat Weterynarii | POLAND |
| DSVSA DOLJ | ROMANIA |
| DSVSA-LSVSA Calarasi | ROMANIA |

| Organisation | Country |
|---|---|
| SP LABORATORIJA A.D. | SERBIA |
| INSTITUTE OF MEAT HYGIENE AND TECHNOLOGY | SERBIA |
| KMETIJSKI INSTITUT SLOVENIJE | SLOVENIA |
| LABORATORI AGROALIMENTARI - DAAM (Generalitat de Catalunya) | SPAIN |
| LABORATORIO AGRARIO REGIONAL. JUNTA DE CASTILLA Y LEÓN. | SPAIN |
| Consejeria de Ganaderia, Pesca y Desarrollo Rural | SPAIN |
| Agroscope ALP | SWITZERLAND |
| COOP Zentrallabor | SWITZERLAND |
| LCAE LABORATOIRE CENTRAL D'ANALYSES ET D'ESSAIS | TUNISIA |
| Eurofins food testing UK limited | UNITED KINGDOM |

## 7. 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 |
| EURACHEM | A focus for Analytical Chemistry in Europe |
| EU-RL-HM | European Union Reference Laboratory for Heavy Metals in Feed and Food |
| GUM | Guide for the Expression of Uncertainty in Measurement |
| GF-AAS | Graphite furnace atomic absorption spectrometry |
| 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 |
| PE | Polyethylene |
| PT | Proficiency Test |
| PTWI | Provisional Tolerable Weekly Intake |
| RM | Reference material |

## 8 References

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## Annexes

# Annex 1: Invitation to EA to nominate laboratories

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements

IMEP

1st June 2012

Mr Paul Greenwood
United Kingdom Accreditation Service
21-47 High Street
Feltham
Middlesex
TW13 4UN
UNITED KINGDOM

Dear Paul,

**Interlaboratory comparison for the determination of total Heavy metals in feed pre-mixes**

The Institute for Reference Materials and Measurements (IRMM) organises IMEP-36, an interlaboratory comparison for the "**Determination of the total cadmium, lead, arsenic, mercury and tin in feed pre-mixes**" in support to the *Directive No 2002/32/EC of the European Parliament and the Council on undesirable substances in animal feed*.

In the frame of the EA-IRMM collaboration agreement, IRMM kindly invites EA to nominate laboratories for free participation. They should hold (or be in the process of obtaining) an accreditation for this type of measurement.

I suggest that you forward this invitation to the national EA accreditation bodies for their consideration. There is a limited number of samples at your disposal and the number of nominees should not exceed 2-3 laboratories per country.

Confidentiality of the participants and their results towards third parties is guaranteed. However, IMEP will disclose details of the participants that have been nominated by EA to you. The EA accreditation bodies may wish to inform the nominees of this disclosure.

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 682. Fax: (32-14) 571 865.

E-mail: jrc-irmm-imep@ec.europa.eu

The registration page for laboratories appointed by EA is open until 3 **July 2012**. Distribution of the samples is foreseen for the second week of July 2012. The deadline for submission of results is **15 September 2012**.

In order to register, laboratories must:

1. **Enter** their details online:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=940

2. **Print** the completed form when the system asks to do so and clearly indicate on the printed form that you have been appointed by the European Cooperation for Accreditation to take part in this exercise **otherwise your laboratory will be invoiced 320 € for participation,** tariff that will be charged to non-appointed laboratories.

3. **Send** the printout to both the IMEP-36 and the EA-IMEP-36 coordinators:

| **IMEP-36 coordinator** | **EA-IMEP-36 coordinator** |
|---|---|
| Dr. Beatriz de la Calle | Mr. Paul Greenwood |
| Fax +32 14 571865 | Fax +44 208 917 8500 |
| E-mail jrc-irmm-imep@ec.europa.eu | E-mail pg@UKAS.com |

Please contact me if you have any questions or comments. We are looking forward to our cooperation!

With kind regards

Beatriz de la Calle
IMEP-36 Coordinator

# Annex 2: Invitation to IAAC to nominate laboratories

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements

IMEP

Geel, 07 June 2012

To: Babara Belzer
IAAC Lab Committee

**Intercomparison for Total cadmium, lead, arsenic, mercury and tin in feed pre-mixes**

Dear Mrs. Belzer,

The Institute for Reference Materials and Measurements (IRMM) organises an interlaboratory comparison for the **"Determination of the total cadmium, lead, arsenic, mercury and tin in feed pre-mixes ", (IMEP-36).**

IRMM kindly invites IAAC to nominate 10 laboratories for free participation. However, they should hold (or be in the process of obtaining) an accreditation for this type of measurement. I suggest that you forward this invitation to a selection of specialised laboratories in this area.

In addition to the 10 laboratories above mentioned, other laboratories may take part in IMEP-36 paying a registration fee of 320 €.

Confidentiality of the participants and their results towards third parties is guaranteed.

Registration of participants is open until **3 July 2012**. Distribution of the samples is foreseen for the second week of July 2012, and the deadline for submission of results is **15 September 2012**.

In order to register, laboratories must:

1. **Enter** their details online:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=940

2. **Print** the completed form when the system asks to do so and clearly indicate on the printed form that they have been appointed by APLAC to take part in this exercise **otherwise your laboratory will be invoiced 320 € for participation** normally applied for non-appointed laboratories.

3. **Send** the printout to both the IMEP-33 and the APLAC coordinators:

| **IMEP-36 coordinator** | **IAAC coordinator** |
|---|---|
| Beatriz de la Calle | Barbara Belzer |
| Fax +32 14 571 865 | |
| E-mail: jrc-irmm-imep@ec.europa.eu | E.Mail: barbara.belzer@nist.gov |

Please contact me if you have any questions or comments. We are looking forward to our cooperation!

With kind regards

Beatriz de la Calle
IMEP-36 Coordinator

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 715. Fax: (32-14) 571 865.

E-mail: jrc-irmm-imep@ec.europa.eu

# Annex 3: Invitation to APLAC to nominate laboratories

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements

IMEP

Geel, 01 June 2012

To: Aparna Dhawan
APLAC PT Committee

**Intercomparison for Total cadmium, lead, arsenic, mercury and tin in feed pre-mixes**

Dear Aparna,

The Institute for Reference Materials and Measurements (IRMM) organises an interlaboratory comparison for the **"Determination of the total cadmium, lead, arsenic, mercury and tin in feed pre-mixes ", (IMEP-36)**.

IRMM kindly invites APLAC to nominate 10 laboratories for free participation. However, they should hold (or be in the process of obtaining) an accreditation for this type of measurement. I suggest that you forward this invitation to a selection of specialised laboratories in this area.

In addition to the 10 laboratories above mentioned, other laboratories may take part in IMEP-36 paying a registration fee of 320 €.

Confidentiality of the participants and their results towards third parties is guaranteed.

Registration of participants is open until **3 July 2012**. Distribution of the samples is foreseen for the second week of July 2012, and the deadline for submission of results is **15 September 2012**.

In order to register, laboratories must:

1. **Enter** their details online:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=940

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 715. Fax: (32-14) 571 865.

E-mail: jrc-irmm-imep@ec.europa.eu

2. **Print** the completed form when the system asks to do so and clearly indicate on the printed form that they have been appointed by APLAC to take part in this exercise **otherwise your laboratory will be invoiced 320 € for participation** normally applied for non-appointed laboratories.

3. **Send** the printout to both the IMEP-33 and the APLAC coordinators:

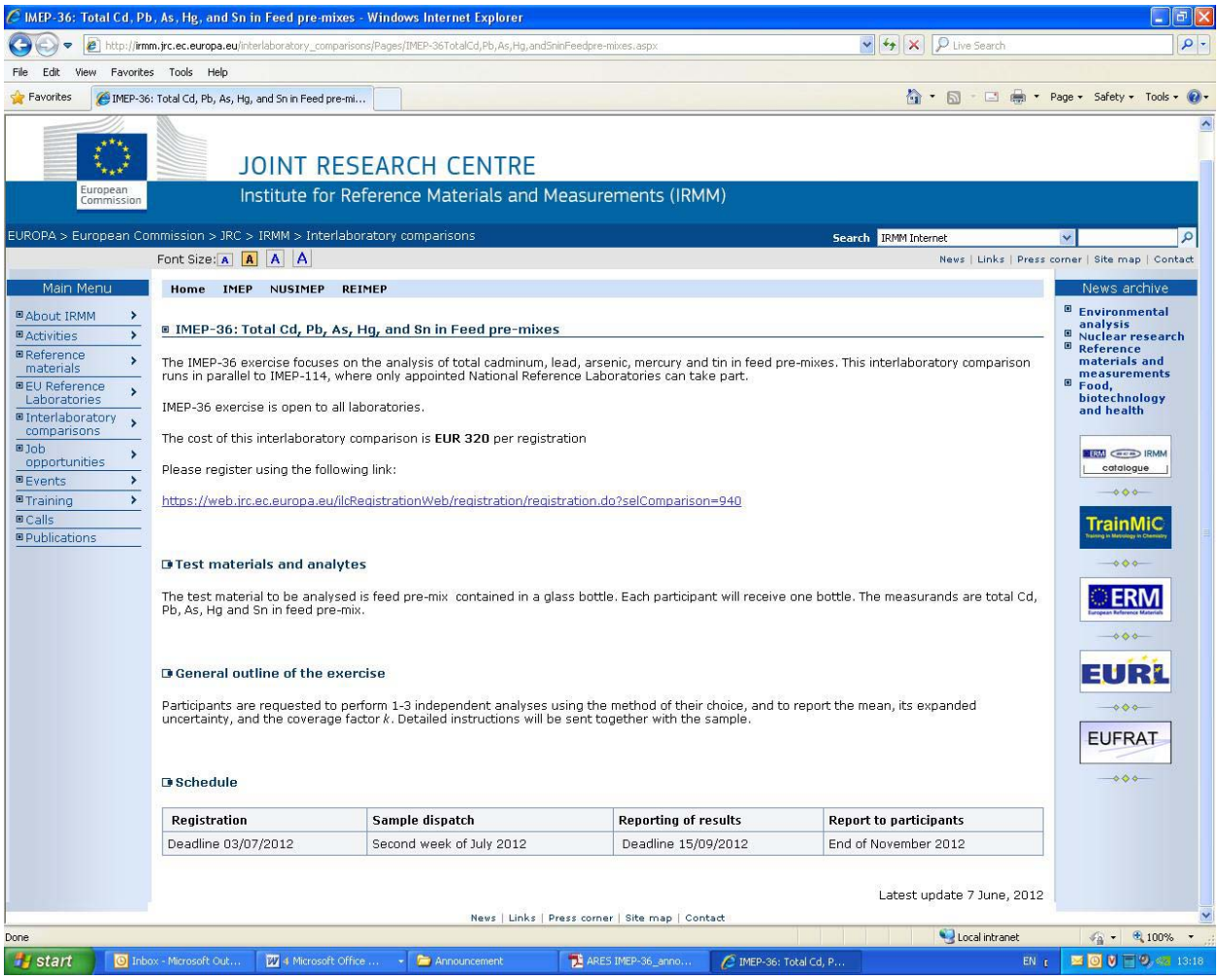
| | |
|---|---|
| **IMEP-36 coordinator** | **APLAC coordinator** |
| Beatriz de la Calle | Aparna Dhawan |
| Fax +32 14 571 865 | |
| E-mail: jrc-irmm-imep@ec.europa.eu | E.Mail: aparna@nabl-india.org |

Please contact me if you have any questions or comments. We are looking forward to our cooperation!

With kind regards

Beatriz de la Calle
IMEP-36 Coordinator

# Annex 4: Announcement on IRMM - IMEP website



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
**Food Safety & Quality**

IMEP

Geel, 9 July 2012
JRC.D5/IF/bk/ARES(2012)821552

«TITLE» «FIRSTNAME» «SURNAME»
«ORGANISATION»
«DEPARTMENT»
«ADDRESS»
«ADDRESS2»
«ZIP» «TOWN»
«COUNTRY»

**Participation in IMEP-36, a proficiency test exercise for the determination of Total Cd, Pb, Hg, As and Sn in Feed Pre-mixes**

Dear «TITLE» «SURNAME»,

Thank you for participating in the IMEP-36 proficiency test for the determination of **Total Cd, Pb, Hg, As and Sn in Feed Premixes.**

This parcel contains:

a) One bottle containing approximately 20 g of powdered feed pre-mix
b) A "Confirmation of Receipt" form
c) A summary of the questionnaire you will be prompted to answer on-line after reporting your results.
d) This accompanying letter

Please check whether the bottle containing the test material remained undamaged during transport. Then fax (at +32-14-571865) or e-mail the "Confirmation of receipt" form. You should store the samples in a dark and cool place (not more than 4 °C) until analysis.

The measurands are: **Total Cd, Pb, Hg, As and Sn in Feed Premixes.**

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

Please perform two or three independent measurements per measurand. Correct the measurement results for recovery and moisture content and report the **corrected mean** on the reporting website. The results should be reported in the same way (e.g., number of significant figures) as normally reported to your customers.

The results in the powder are to be reported referring to dry mass and thus corrected for humidity. To calculate the **water content** in the test material, please apply the following procedure:

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 262. Fax: (32-14) 571 865.

E-mail: jrc-irmm-crl-heavy-metals@ec.europa.eu

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 an oven for 4 h ± 5 min at 130 ± 1 °C.*
3. *Place the glass container covered with a lid in a desiccator and wait 30 min before weighing the test material again.*

**Reporting of results**

Please perform two or three independent measurements per measurand. Correct the measurement results for recovery, and report on the reporting website:

- **the corrected mean**,
- the associated expanded **uncertainty**,
- the **coverage factor** and
- the **technique** you used

Mean and uncertainty are to be reported in the same unit.

You can find the reporting website at https://irmm.jrc.ec.europa.eu/ilc/ilcReporting.do

To access this webpage you need a personal password key, which is: **«Part_key»**. The system will guide you through the reporting procedure. Please enter for each measurand the **mean** of your two or three measurement results, the **uncertainty of the mean**, the **coverage factor** and the **technique** you used. After entering all results, please complete also the relating questionnaire.

**Do not forget to submit and confirm always when required.**

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 definitive confirmation.

**The deadline for submission of results is 15/09/2012.**

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

Dr. M.B. de la Calle
IMEP-36 Co-ordinator

Enclosures: a) One bottle containing 20 g of sample, b) A "Confirmation of Receipt" form, c) A summary of the questionnaire and e) This accompanying letter

## Annex 6: 'Confirmation of receipt' form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
**Food Safety & Quality**

Geel, 9 July 2012
JRC.D5/IF/bk/ARES(2012)/821552

«TITLE» «FIRSTNAME» «SURNAME»
«ORGANISATION»
«DEPARTMENT»
«ADDRESS»
«ADDRESS2»
«ZIP» «TOWN»
«COUNTRY»

**IMEP-36**
**Total Cd, Pb, Hg, As and Sn in Feed Premixes**

**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:**
Dr. M.B. de la Calle

IMEP-36 Coordinator
EC-JRC-IRMM
Retieseweg 111
B-2440 GEEL, Belgium

Fax : +32-14-571865
e-mail : JRC-IRMM-CRL-HEAVY-METALS@ec.europa.eu

## Annex 7: Summary questionnaire sent with sample

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
**Food Safety & Quality**

Annex to JRC.D5/IF/bk/ARES(2012)/821552

**FOR INFORMATION ONLY** – SUMMARY QUESTIONNAIRE IMEP-36

- Have you corrected your results for recovery?
- If yes, Which are the correction factors for Total Cd, Pb, Hg, As and Sn
- What is the basis of your uncertainty estimate?
- Do you usually provide an uncertainty statement to your customers for this type of analysis?
- Did you correct for the moisture content of the sample?
- Did you analyse the sample according to an official method?
- Does your laboratory carry out this type of analysis (as regards the anlytes, matrix and methods) on a regular basis?
- Does your laboratory have a quality system in place?
- Is your laboratory accredited for this type of analysis?
- Does your laboratory take part in interlaboratory comparisons for this type of analysis on a regular basis?
- Does your laboratory use a reference material for this type of analysis?
- How have you heard about this exercise?
- Do you have any comments?

**Please – complete the questionnaire online, when submitting your results !**

# Annex 8: Online Questionnaire

Questionnaire questions

1. **Have you corrected your results for recovery?** *[Q:105533: RADIO]*

- no *[A:179]*
- yes *[A:124]*

1.1. If no, why? *[Q:105534: TEXT]*

1.2. If yes, * if parent A:124 checked *[Q:105636: CUSTOM TABLE]*

**Which are the correction factors for:**

Please enter the recovery factors that you have used to correct your results.

| Questions/Response table | Total Cd | Total Pb | Total As | Total Hg | Total Sn |
|---|---|---|---|---|---|
| Recovery % | | | | | |

2. **What is the basis of your uncertainty estimate (multiple answers are possible)?** *[Q:105506: CHECKBOX]*

- 1. uncertainty budget calculated according to iso-gum *[A:238]*
- 2. known uncertainty of the standard method *[A:239]*
- 3. uncertainty of the method as determined in-house validation *[A:240]*
- 4. measurement of replicates (i.e. precision) *[A:241]*
- 5. estimation based on judgement *[A:964]*
- 6. use of intercomparison data *[A:243]*
- 7. other *[A:244]*

2.1. If other, please specify *[Q:105507: TEXT]*

3. **Do you usually provide an uncertainty statement to your customers for this type of analysis?** *[Q:105508: RADIO]*

- no *[A:179]*
- yes *[A:288]*

4. **Did you correct for the moisture content of the sample?** *[Q:105509: RADIO]*

- no *[A:179]*
- yes *[A:124]*

4.1. If yes, what is the moisture content (in % of the sample mass)? *[Q:105510: TEXT]*

4.2. If no, what was the reason not to do this? *[Q:105511: TEXT]*

5. **Did you analyse the sample according to an official method?** *[Q:105512: RADIO]*

- no *[A:179]*
- yes *[A:124]*

5.1. If yes, which: *[Q:105513: TEXT]*

5.2. If no, please describe (in max. 150 characters for each reply) your *[Q:105514: GROUP]*

5.2.1. sample pre-treatment *[Q:105515: TEXT]*

5.2.2. digestion step *[Q:105516: TEXT]*

5.2.3. extraction / separation step *[Q:105517: TEXT]*

5.2.4. instrument calibration step *[Q:105518: TEXT]*

6. **Does your laboratory carry out this type of analysis (as regards the anlytes, matrix and methods) on a regular basis?** *[Q:105519: RADIO]*

- no *[A:179]*
- yes *[A:124]*

6.1. If Yes, please estimate the number of samples (As, Cd, Pb, Hg, Cu measurements together): *[Q:105520: RADIO]*

- a) 0-50 samples per year *[A:245]*
- b) 50-250 samples per year *[A:246]*
- c) 250-1000 samples per year *[A:249]*
- d) more than 1000 samples per year *[A:248]*

7. **Does your laboratory have a quality system in place?** *[Q:105521: RADIO]*

- no *[A:179]*
- yes *[A:124]*

7.1. If yes, which: *[Q:105522: CHECKBOX]*

- a) ISO 17025 *[A:350]*
- b) ISO 9000 series *[A:351]*
- c) Other *[A:352]*

7.1.1. If other, please specify *[Q:105523: TEXT]*

8. **Is your laboratory accredited for this type of analysis?** *[Q:105524: RADIO]*

- no *[A:179]*
- yes *[A:124]*

8.1. If yes, by which accreditation body * if parent A:124 checked *[Q:105638: TEXT]*

9. **Does your laboratory take part in interlaboratory comparisons for this type of analysis on a regular basis** *[Q:105525: RADIO]*

- no *[A:179]*
- yes *[A:124]*

9.1. If yes, which one(s) *[Q:105526: TEXT]*

10. **Does your laboratory use a reference material for this type of analysis?** *[Q:105527: RADIO]*

- no *[A:179]*
- yes *[A:124]*

10.1. If yes, which one(s) *[Q:105528: TEXT]*

10.2. Is the material used for the validation of procedures? *[Q:105529: RADIO]*

- no *[A:179]*
- yes *[A:124]*

10.3. Is the material used for calibration of instruments? *[Q:105530: RADIO]*

- no *[A:179]*
- yes *[A:124]*

11. **How have you heard about this exercise?** *[Q:105532: TEXT]*

12. **Do you have any comments? Please let us know: ...** *[Q:105531: TEXT]*

# Annex 9: Homogeneity and stability studies

## 9.1 Homogeneity study for total Arsenic

| | Total As (mg $Kg^{-1}$) | |
|---|---|---|
| **Bottle ID** | **Replicate 1** | **Replicate 2** |
| 55 | 1.97 | 2.01 |
| 66 | 2.12 | 2.07 |
| 42 | 1.93 | 2.04 |
| 77 | 2.10 | 1.97 |
| 121 | 2.07 | 2.09 |
| 114 | 2.05 | 1.96 |
| 98 | 2.01 | 1.91 |
| 109 | 1.98 | 1.98 |
| 114 | 1.93 | 1.95 |
| 41 | 1.88 | 1.93 |
| **Mean of 20 results** | 2.00 | |
| $\hat{\sigma}$ | 15% (0.290) | |
| **Homogeneity test according to ISO 13528 [14]** | | |
| **0,3** $\hat{\sigma}$ | 0.087 | |
| $s_x$ | 0.059 | |
| $s_w$ | 0.052 | |
| $s_s$ | 0.046 | |
| $s_s \leq 0{,}3\sigma$ ? | Yes | |
| **Test result** | Passed | |

## 9.2 Stability study for total Arsenic

**Stability Study – Total As**

***TEMPERATURE = 18°C***

Meas.Unit: mg $kg^{-1}$

| | Time in Weeks | | | |
|---|---|---|---|---|
| samples | 0 | 3 | 5 | 8 |
| 1 | 1.86 | 1.86 | 1.86 | 1.86 |
| 2 | 2.02 | 2.02 | 2.02 | 2.02 |

**CALCULATION OF $U_{lts}$ for given $X_{shelf}$**

Given $X_{shelf}$ = 10 Weeks

$u_{lts}$ = 0.102 mg $kg^{-1}$

$u_{lts}$[%] = 5.3%

**REGRESSION LINE PARAMETERS**

Slope = 0.003
SE Slope = 0.011
Intercept = 1.895
SE Intercept = 0.054
Correlation Coefficient =0.014

Slope of the linear regression significantly <> 0 (95%) :No
Slope of the linear regression significantly <> 0 (99%) :No

## 9.3 Homogeneity study for total Cadmium

| | Total Cd (mg $Kg^{-1}$) | |
|---|---|---|
| **Bottle ID** | **Replicate 1** | **Replicate 2** |
| 55 | 1.12 | 1.14 |
| 66 | 1.10 | 1.13 |
| 42 | 1.11 | 1.13 |
| 77 | 1.11 | 1.11 |
| 121 | 1.09 | 1.12 |
| 114 | 1.11 | 1.09 |
| 98 | 1.12 | 1.12 |
| 109 | 1.11 | 1.09 |
| 114 | 1.11 | 1.13 |
| 41 | 1.08 | 1.12 |
| **Mean of 20 results** | 1.11 | |
| $\hat{\sigma}$ | 15% (0.167) | |
| **Homogeneity test according to ISO 13528 [14]** | | |
| **0,3** $\hat{\sigma}$ | 0.050 | |
| $\mathbf{s_x}$ | 0.010 | |
| $\mathbf{s_w}$ | 0.016 | |
| $\mathbf{s_s}$ | $MS_{Bb} < MS_{Wb}$ | |
| $\mathbf{s_s \leq 0{,}3\sigma}$ **?** | Yes | |
| **Test result** | Passed | |

## 9.4 Stability study for total Cadmium

**Stability Study – Total Cd**

***TEMPERATURE = 18°C***

Meas.Unit: mg $kg^{-1}$

| | Time in Weeks | | | |
|---|---|---|---|---|
| samples | 0 | 3 | 5 | 8 |
| 1 | 1.10 | 1.10 | 1.13 | 1.08 |
| 2 | 1.13 | 1.09 | 1.11 | 1.11 |

**CALCULATION OF $U_{lts}$ for given $X_{shelf}$**

Given $X_{shelf}$ = 10 Weeks

$u_{lts}$ = 0.021 mg $kg^{-1}$

$u_{lts}$[%] = 1.9 %

**REGRESSION LINE PARAMETERS**

Slope = -0.002
SE Slope = 0.002
Intercept = 1,.113
SE Intercept = 0.011
Correlation Coefficient =0.081

Slope of the linear regression significantly <> 0 (95%) :No
Slope of the linear regression significantly <> 0 (99%) :No

## 9.5 Homogeneity study for total Lead

| | Total lead (mg kg$^{-1}$) | |
|---|---|---|
| **Bottle ID** | **Replicate 1** | **Replicate 2** |
| 55 | 0.65 | 0.68 |
| 66 | 0.65 | 0.69 |
| 42 | 0.66 | 0.68 |
| 77 | 0.64 | 0.66 |
| 121 | 0.64 | 0.65 |
| 72 | 0.63 | 0.64 |
| 98 | 0.68 | 0.69 |
| 109 | 0.65 | 0.66 |
| 33 | 0.61 | 0.63 |
| 41 | 0.64 | 0.65 |
| **Mean of 20 results** | 0.65 | |
| $\hat{\sigma}$ | 15% (0.095) | |
| **Homogeneity test according to ISO 13528 [14]** | | |
| $0.3\,\hat{\sigma}$ | 0.029 | |
| $S_x$ | 0.020 | |
| $S_w$ | 0.013 | |
| $S_s$ | 0.018 | |
| $S_s \leq 0.3\ \hat{\sigma}$ ? | Yes | |
| Test result | Passed | |

## 9.6 Stability study for total Lead

**Stability Study – Total Pb**

***TEMPERATURE = 18°C***

Meas.Unit: mg kg$^{-1}$

| | Time in Weeks | | | |
|---|---|---|---|---|
| samples | 0 | 3 | 5 | 8 |
| 1 | 0.617 | 0.642 | 0.606 | 0.625 |
| 2 | 0.638 | 0.653 | 0.655 | 0.648 |

**CALCULATION OF $U_{lts}$ for given $X_{shelf}$**

Given $X_{shelf}$ = 10 Weeks

$u_{lts}$ = 0.022 mg kg$^{-1}$

$u_{lts}$[%] = 3.4%

**REGRESSION LINE PARAMETERS**

Slope = 0.001
SE Slope = 0.002
Intercept = 0.633
SE Intercept = 0.011
Correlation Coefficient =0.010

Slope of the linear regression significantly <> 0 (95%) :No
Slope of the linear regression significantly <> 0 (99%) :No

## 9.7 Homogeneity study for total Tin

| | Total Tin (mg kg$^{-1}$) | |
|---|---|---|
| **Bottle ID** | **Replicate 1** | **Replicate 2** |
| 55 | 0.73 | 0.77 |
| 66 | 0.77 | 0.77 |
| 42 | 0.72 | 0.81 |
| 77 | 0.71 | 0.78 |
| 121 | 0.77 | 0.74 |
| 72 | 0.76 | 0.77 |
| 98 | 0.72 | 0.74 |
| 109 | 0.72 | 0.60 |
| 33 | 0.70 | 0.69 |
| 41 | 0.74 | 0.73 |
| **Mean of 20 results** | 0.74 | |
| $\hat{\sigma}$ | 15% (0.119) | |
| **Homogeneity test according to ISO 13528 [14]** | | |
| $0.3\,\hat{\sigma}$ | 0.036 | |
| $S_x$ | 0.035 | |
| $S_w$ | 0.039 | |
| $S_s$ | 0.021 | |
| $S_s \leq 0.3\ \hat{\sigma}$ ? | Yes | |
| Test result | Passed | |

## 9.8 Stability study for total Tin

**Stability Study – Total Sn**

***TEMPERATURE = 18°C***

Meas.Unit: mg kg$^{-1}$

| | Time in Weeks | | | |
|---|---|---|---|---|
| samples | 0 | 3 | 5 | 8 |
| 1 | 0.688 | 0.688 | 0.688 | 0.688 |
| 2 | 0.648 | 0.648 | 0.648 | 0.648 |

**CALCULATION OF $U_{lts}$ for given $X_{shelf}$**

Given $X_{shelf}$ = 10 Weeks

$u_{lts}$ = 0,047 mg kg$^{-1}$

$u_{lts}$[%] = 7.0 %

**REGRESSION LINE PARAMETERS**

Slope = 0.006
SE Slope = 0.004
Intercept = 0.656
SE Intercept = 0.022
Correlation Coefficient =0.251

Slope of the linear regression significantly <> 0 (95%) :No
Slope of the linear regression significantly <> 0 (99%) :No

## Annex 10: Results for Total Arsenic

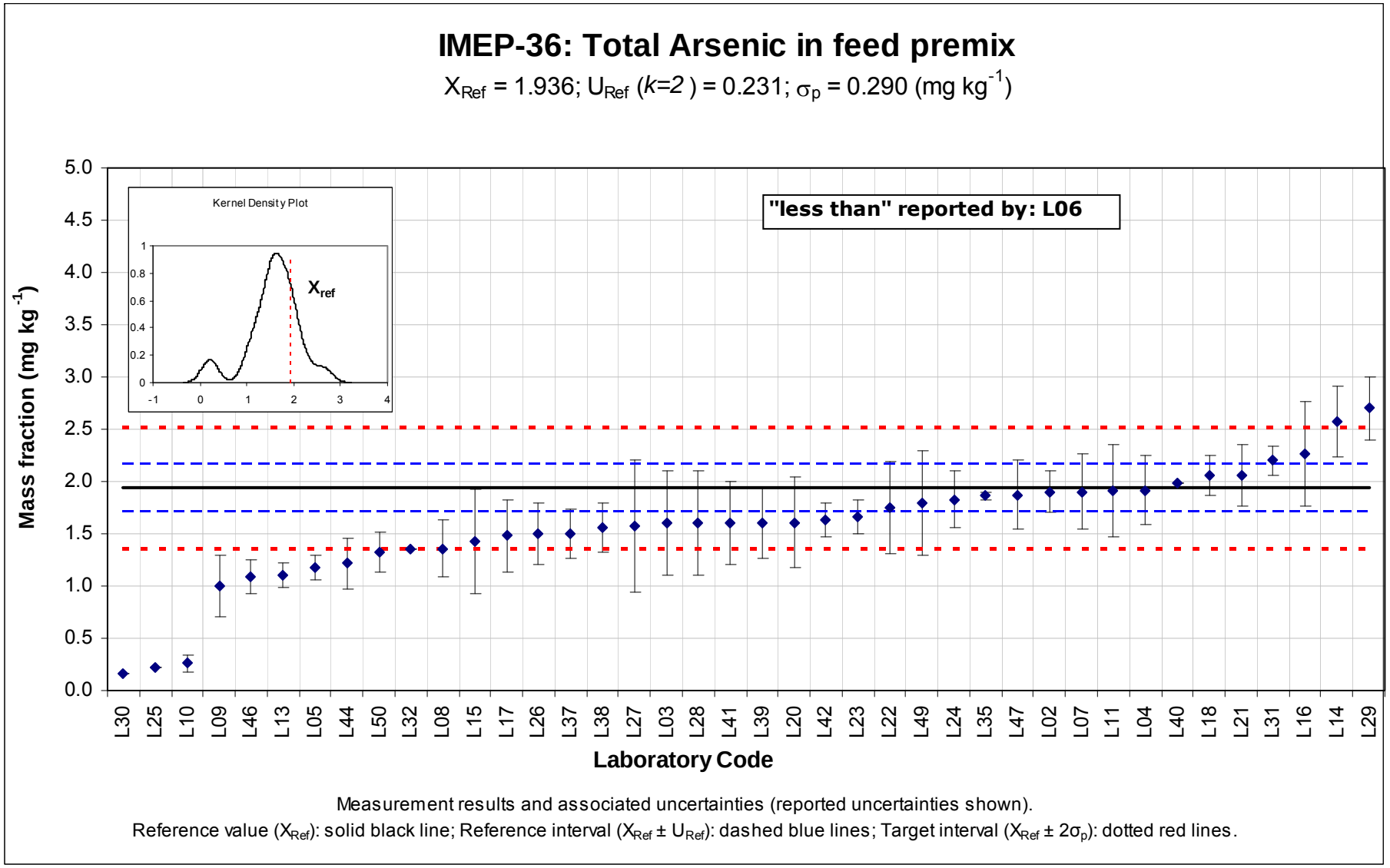
Assigned range: $X_{ref}$ = 1,936, U (k = 2) = 0.231, $\sigma_p$= 0.29 (all values in mg $kg^{-1}$)

| Lab Code | $X_{lab}$ | ± | k[a] | technique | $u_{lab}$ | z-score[b] | ζ-score[b] | uncert.[c] |
|---|---|---|---|---|---|---|---|---|
| L02 | 1.90 | 0.20 | 2 | ICP-AES | 0.100 | -0.12 | -5.98 | b |
| L03 | 1.6 | 0.5 | 2 | ICP-AES | 0.250 | -1.16 | -1.22 | a |
| L04 | 1.918 | 0.327 | 2 | ICP-MS | 0.164 | -0.06 | -0.09 | a |
| L05 | 1.179 | 0.118 | 2 | ICP-MS | 0.059 | -2.61 | -5.82 | b |
| L06 | < 2.5 | | | ICP-AES | | | | |
| L07 | 1.90 | 0.361 | 2 | ICP-MS | 0.181 | -0.12 | -0.17 | a |
| L08 | 1.36 | 0.27 | 2 | ICP-AES | 0.135 | -1.98 | -3.24 | a |
| L09 | 1.0 | 0.3 | 2 | HG-AAS | 0.150 | -3.22 | -4.94 | a |
| L10 | 0.26 | 0.08 | 2 | AAS | 0.040 | -5.77 | -13.68 | b |
| L11 | 1.907 | 0.440 | 2 | ICP-MS | 0.220 | -0.10 | -0.11 | a |
| L13 | 1.105 | 0.115 | 2 | HG-AAS | 0.058 | -2.86 | -6.43 | b |
| L14 | 2.57 | 0.34 | 2 | ICP-MS | 0.170 | 2.19 | 3.09 | a |
| L15 | 1.42 | 0.5 | 1 | HG-AAS | 0.500 | -1.78 | -1.00 | c |
| L16 | 2.27 | 0.50 | 2 | ICP-MS | 0.250 | 1.15 | 1.21 | a |
| L17 | 1.481 | 0.347 | 2 | HG-AAS | 0.174 | -1.57 | -2.18 | a |
| L18 | 2.06 | 0.19 | 2 | k0-NAA | 0.095 | 0.43 | 0.83 | b |
| L20 | 1.61 | 0.43 | 2 | AFS | 0.215 | -1.12 | -1.33 | a |
| L21 | 2.06 | 0.30 | 2.35 | ICP-MS | 0.128 | 0.43 | 0.72 | a |
| L22 | 1.75 | 0.44 | 2 | HG-AAS | 0.220 | -0.64 | -0.75 | a |
| L23 | 1.660 | 0.166 | 2 | ICP-MS | 0.083 | -0.95 | -1.93 | b |
| L24 | 1.83 | 0.27 | 2 | ICP-AES | 0.135 | -0.36 | -0.59 | a |
| L25 | 0.222 | 0 | 2 | GF-AAS | 0.000 | -5.90 | -14.80 | b |
| L26 | 1.5 | 0.3 | 1 | ICP-MS | 0.300 | -1.50 | -1.35 | c |
| L27 | 1.58 | 0.632 | 2 | | 0.316 | -1.22 | -1.06 | c |
| L28 | 1.6 | 0.5 | 2 | HG-AAS | 0.250 | -1.16 | -1.22 | a |
| L29 | 2.7 | 0.3 | 2 | ICP-AES | 0.150 | 2.63 | 4.04 | a |
| L30 | 0.169 | 0 | √3 | HG-AAS | 0.000 | -6.08 | -15.26 | b |
| L31 | 2.201 | 0.14 | 2 | ICP-MS | 0.070 | 0.91 | 1.96 | b |
| L32 | 1.3531 | 0.0001 | 2 | HG-AAS | 0.000 | -2.01 | -5.03 | b |
| L35 | 1.865 | 0.0388 | 2 | HG-AAS | 0.019 | -0.24 | -0.60 | b |
| L37 | 1.5 | 0.23 | 2 | ETAAS | 0.115 | -1.50 | -2.67 | b |
| L38 | 1.56 | 0.24 | 2 | HG-AAS | 0.120 | -1.29 | -2.25 | a |
| L39 | 1.604 | 0.334 | 2 | HG-AAS | 0.167 | -1.14 | -1.63 | a |
| L40 | 1.986 | 0 | √3 | ICP-MS | 0.000 | 0.17 | 0.44 | b |
| L41 | 1.6 | 0.4 | 2 | ETAAS | 0.200 | -1.16 | -1.45 | a |
| L42 | 1.630 | 0.163 | 2 | ICP-MS | 0.082 | -1.05 | -2.16 | b |
| L44 | 1.217 | 0.243 | 2 | ICP-MS | 0.122 | -2.47 | -4.28 | a |
| L46 | 1.09 | 0.16 | √3 | HG-AAS | 0.092 | -2.91 | -5.71 | b |
| L47 | 1.87 | 0.33 | 2 | HG-AAS | 0.165 | -0.23 | -0.32 | a |
| L49 | 1.787 | 0.5 | 2 | HG-AAS | 0.250 | -0.51 | -0.54 | a |
| L50 | 1.324 | 0.198 | 2 | ETAAS | 0.099 | -2.11 | -4.01 | b |

[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** : $u_{lab} > u_{max}$




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: Results for Total Cadmium

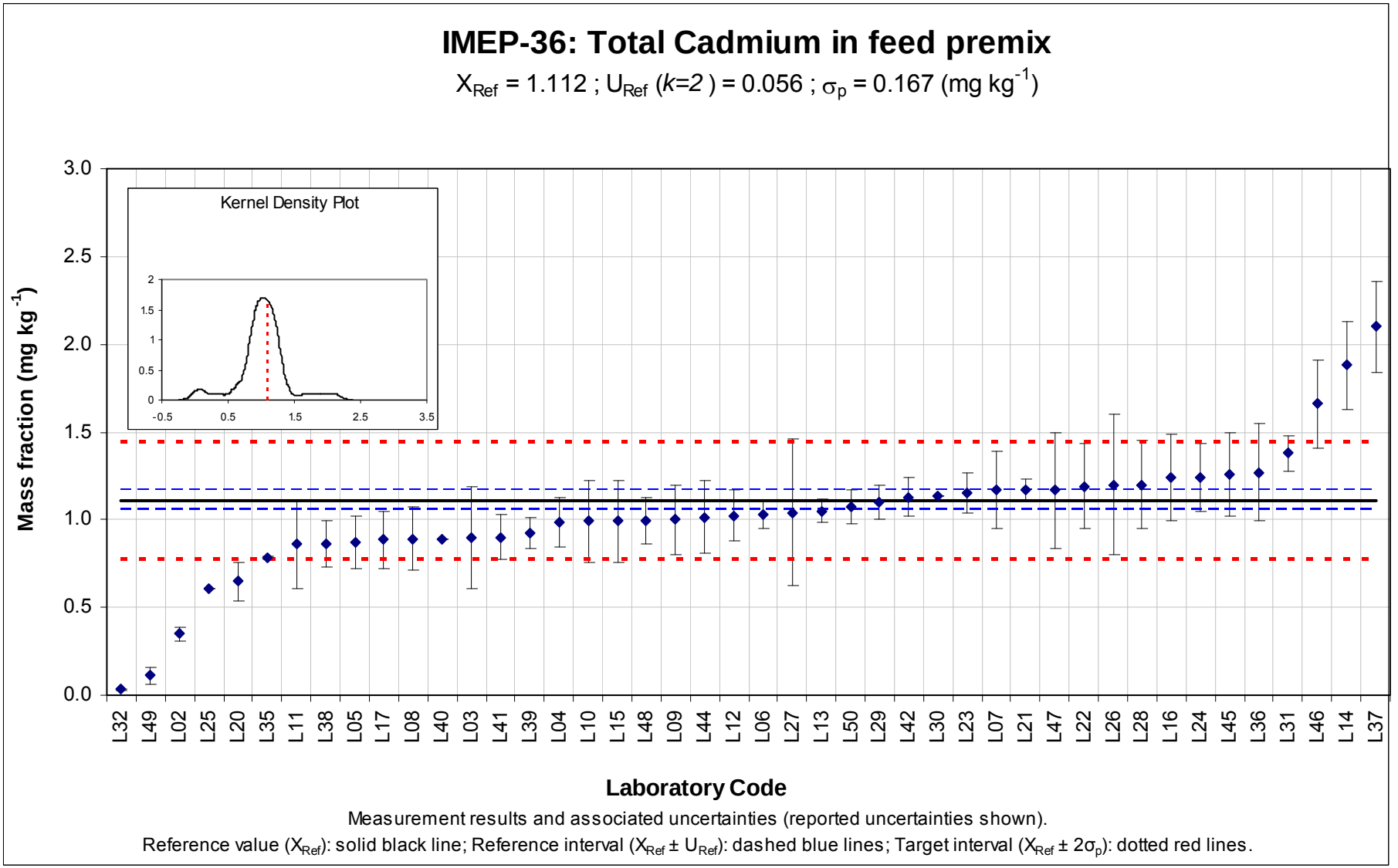
Assigned range: $X_{ref}$ = 1,112, U (k = 2) = 0.056, $\sigma_p$= 0.167 (all values in mg $kg^{-1}$)

| Lab Code | $X_{lab}$ | ± | k[a] | technique | $u_{lab}$ | z-score[b] | ζ-score[b] | uncert.[c] |
|---|---|---|---|---|---|---|---|---|
| L02 | 0.35 | 0.04 | 2 | ICP-AES | 0.020 | -4.57 | -22.21 | b |
| L03 | 0.9 | 0.29 | 2 | ICP-AES | 0.145 | -1.27 | -1.43 | a |
| L04 | 0.983 | 0.141 | 2 | ICP-MS | 0.071 | -0.77 | -1.70 | a |
| L05 | 0.869 | 0.148 | 2 | ICP-MS | 0.074 | -1.45 | -3.07 | a |
| L06 | 1.03 | 0.08 | 2 | ETAAS | 0.040 | -0.49 | -1.67 | a |
| L07 | 1.17 | 0.222 | 2 | ICP-MS | 0.111 | 0.35 | 0.51 | a |
| L08 | 0.89 | 0.18 | 2 | ICP-AES | 0.090 | -1.33 | -2.35 | a |
| L09 | 1.0 | 0.2 | 2 | ICP-AES | 0.100 | -0.67 | -1.07 | a |
| L10 | 0.99 | 0.23 | 2 | AAS | 0.115 | -0.73 | -1.03 | a |
| L11 | 0.858 | 0.249 | 2 | ICP-MS | 0.125 | -1.52 | -1.99 | a |
| L12 | 1.024 | 0.143 | 1 | ETAAS | 0.143 | -0.52 | -0.60 | a |
| L13 | 1.05 | 0.063 | 2 | ETAAS | 0.032 | -0.37 | -1.46 | a |
| L14 | 1.88 | 0.25 | 2 | ICP-MS | 0.125 | 4.61 | 6.00 | a |
| L15 | 0.99 | 0.23 | 1 | ETAAS | 0.230 | -0.73 | -0.52 | c |
| L16 | 1.24 | 0.25 | 2 | ICP-MS | 0.125 | 0.77 | 1.00 | a |
| L17 | 0.885 | 0.163 | 2 | FAAS | 0.082 | -1.36 | -2.63 | a |
| L20 | 0.65 | 0.11 | 2 | ETAAS | 0.055 | -2.77 | -7.49 | a |
| L21 | 1.17 | 0.06 | 2.35 | ICP-MS | 0.026 | 0.35 | 1.55 | b |
| L22 | 1.190 | 0.24 | 2 | ETAAS | 0.120 | 0.47 | 0.64 | a |
| L23 | 1.15 | 0.115 | 2 | ICP-MS | 0.058 | 0.23 | 0.60 | a |
| L24 | 1.24 | 0.19 | 2 | ICP-AES | 0.095 | 0.77 | 1.30 | a |
| L25 | 0.61 | 0 | 2 | GF-AAS | 0.000 | -3.01 | -18.01 | b |
| L26 | 1.2 | 0.4 | 1 | ICP-MS | 0.400 | 0.53 | 0.22 | c |
| L27 | 1.04 | 0.417 | 2 |  | 0.209 | -0.43 | -0.34 | c |
| L28 | 1.2 | 0.25 | 2 | ICP-MS | 0.125 | 0.53 | 0.69 | a |
| L29 | 1.1 | 0.1 | 2 | ICP-MS | 0.050 | -0.07 | -0.20 | a |
| L30 | 1.132 | 0 | √3 | AAS | 0.000 | 0.12 | 0.74 | b |
| L31 | 1.377 | 0.1 | 2 | ICP-MS | 0.050 | 1.59 | 4.64 | a |
| L32 | 0.031 | 0.001 | 2 | GF-AAS | 0.001 | -6.48 | -38.79 | b |
| L35 | 0.7834 | 0.0064 | 2 | FAAS | 0.0032 | -1.97 | -11.70 | b |
| L36 | 1.27 | 0.28 | √3 | AAS | 0.162 | 0.95 | 0.97 | a |
| L37 | 2.1 | 0.26 | 2 | ETAAS | 0.130 | 5.93 | 7.44 | a |
| L38 | 0.864 | 0.13 | 2 | AAS | 0.065 | -1.48 | -3.50 | a |
| L39 | 0.925 | 0.089 | 2 | FAAS | 0.045 | -1.12 | -3.55 | a |
| L40 | 0.89 | 0 | √3 | ETAAS | 0.000 | -1.33 | -7.95 | b |
| L41 | 0.9 | 0.13 | 2 | ETAAS | 0.065 | -1.27 | -2.99 | a |
| L42 | 1.13 | 0.113 | 2 | ICP-MS | 0.057 | 0.11 | 0.29 | a |
| L44 | 1.016 | 0.203 | 2 | ICP-MS | 0.102 | -0.57 | -0.91 | a |
| L45 | 1.260 | 0.24 | 2 | ETAAS | 0.120 | 0.89 | 1.21 | a |
| L46 | 1.66 | 0.25 | √3 | AAS | 0.144 | 3.29 | 3.73 | a |
| L47 | 1.17 | 0.33 | 2 | ETAAS | 0.165 | 0.35 | 0.35 | a |
| L48 | 0.995 | 0.134 | 2 | AAS | 0.067 | -0.70 | -1.61 | a |
| L49 | 0.111 | 0.05 | 2 | ETAAS | 0.025 | -6.00 | -26.73 | b |
| L50 | 1.075 | 0.095 | 2 | ETAAS | 0.048 | -0.22 | -0.66 | 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*=√3.

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

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




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 12: Results for Total Lead

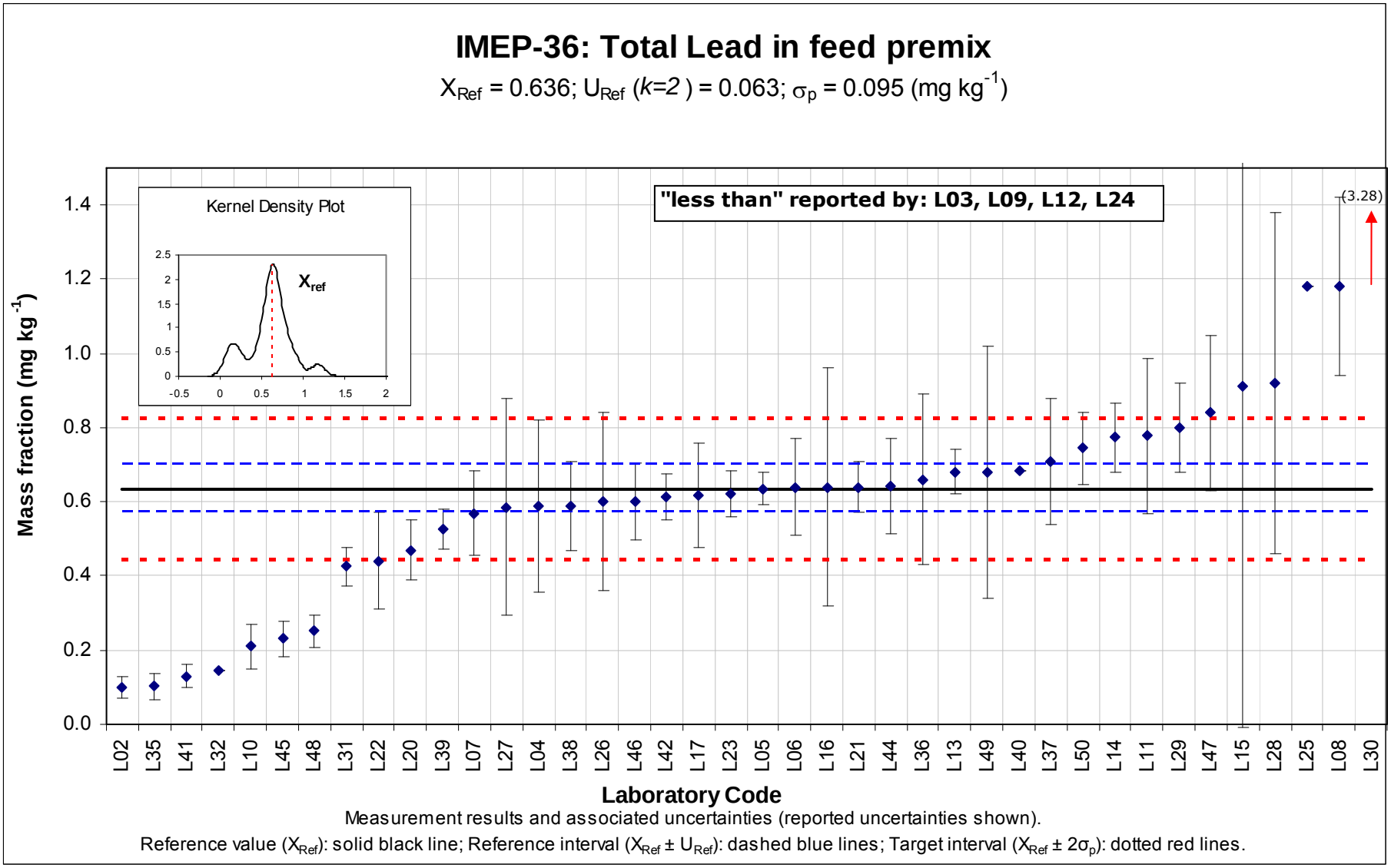
Assigned range: $X_{ref}$ = 0.636, U (k = 2) = 0.063, $\sigma_p$= 0.095 (all values in mg $kg^{-1}$)

| Lab Code | $X_{lab}$ | ± | k[a] | technique | $u_{lab}$ | z-score[b] | ζ-score[b] | uncert.[c] |
|---|---|---|---|---|---|---|---|---|
| L02 | 0.10 | 0.03 | 2 | ICP-AES | 0.015 | -5.62 | -15.34 | b |
| L03 | < 1 | | | ICP-AES | | | | |
| L04 | 0.589 | 0.231 | 2 | ICP-MS | 0.1155 | -0.49 | -0.39 | c |
| L05 | 0.636 | 0.045 | 2 | ICP-MS | 0.0225 | 0.01 | 0.01 | b |
| L06 | 0.64 | 0.13 | 2 | ETAAS | 0.065 | 0.05 | 0.06 | a |
| L07 | 0.57 | 0.114 | 2 | ICP-MS | 0.057 | -0.70 | -1.02 | a |
| L08 | 1.18 | 0.24 | 2 | ICP-AES | 0.12 | 5.71 | 4.39 | c |
| L09 | < 2 | | | ETAAS | | | | |
| L10 | 0.21 | 0.06 | 2 | AAS | 0.03 | -4.46 | -9.78 | b |
| L11 | 0.777 | 0.210 | 2 | ICP-MS | 0.105 | 1.48 | 1.29 | c |
| L12 | < 0.2 | | | ETAAS | | | | |
| L13 | 0.68 | 0.06 | 2 | ETAAS | 0.03 | 0.47 | 1.02 | b |
| L14 | 0.773 | 0.093 | 2 | ICP-MS | 0.0465 | 1.44 | 2.45 | a |
| L15 | 0.91 | 0.46 | 1 | ETAAS | 0.46 | 2.88 | 0.60 | c |
| L16 | 0.64 | 0.32 | 2 | ICP-MS | 0.16 | 0.05 | 0.03 | c |
| L17 | 0.618 | 0.142 | 2 | FAAS | 0.071 | -0.18 | -0.23 | a |
| L20 | 0.47 | 0.08 | 2 | ETAAS | 0.04 | -1.74 | -3.25 | a |
| L21 | 0.64 | 0.08 | 2.35 | ICP-MS | 0.034043 | 0.05 | 0.10 | a |
| L22 | 0.44 | 0.13 | 2 | ETAAS | 0.065 | -2.05 | -2.71 | a |
| L23 | 0.62 | 0.062 | 2 | ICP-MS | 0.031 | -0.16 | -0.35 | b |
| L24 | < 0.67 | | | ICP-AES | | | | |
| L25 | 1.179 | 0 | 2 | GF-AAS | 0 | 5.70 | 17.24 | b |
| L26 | 0.600 | 0.12 | 1 | ICP-MS | 0.12 | -0.37 | -0.29 | c |
| L27 | 0.586 | 0.293 | 2 | | 0.1465 | -0.52 | -0.33 | c |
| L28 | 0.92 | 0.46 | 2 | ICP-MS | 0.23 | 2.98 | 1.23 | c |
| L29 | 0.8 | 0.12 | 2 | ICP-MS | 0.06 | 1.73 | 2.43 | a |
| L30 | 3.28 | 0 | √3 | AAS | 0 | 27.74 | 83.88 | b |
| L31 | 0.425 | 0.05 | 2 | ICP-MS | 0.025 | -2.21 | -5.23 | b |
| L32 | 0.145 | 0.001 | 2 | GF-AAS | 0.0005 | -5.15 | -15.56 | b |
| L35 | 0.1024 | 0.035 | 2 | FAAS ? | 0.0175 | -5.59 | -14.78 | b |
| L36 | 0.66 | 0.2 | √3 | AAS | 0.11547 | 0.26 | 0.20 | c |
| L37 | 0.71 | 0.17 | 2 | ETAAS | 0.085 | 0.78 | 0.82 | a |
| L38 | 0.59 | 0.12 | 2 | AAS | 0.06 | -0.48 | -0.67 | a |
| L39 | 0.527 | 0.054 | 2 | ETAAS | 0.027 | -1.14 | -2.61 | b |
| L40 | 0.683 | 0 | √3 | ETAAS | 0 | 0.50 | 1.51 | b |
| L41 | 0.13 | 0.03 | 2 | ETAAS | 0.015 | -5.30 | -14.48 | b |
| L42 | 0.613 | 0.061 | 2 | ICP-MS | 0.0305 | -0.24 | -0.51 | b |
| L44 | 0.642 | 0.128 | 2 | ICP-MS | 0.064 | 0.07 | 0.09 | a |
| L45 | 0.230 | 0.046 | 2 | ETAAS | 0.023 | -4.25 | -10.39 | b |
| L46 | 0.6 | 0.09 | √3 | AAS | 0.051962 | -0.37 | -0.58 | a |
| L47 | 0.84 | 0.21 | 2.00 | ETAAS | 0.105 | 2.15 | 1.87 | c |
| L48 | 0.252 | 0.043 | 2 | AAS | 0.0215 | -4.02 | -10.05 | b |
| L49 | 0.681 | 0.34 | 2 | ETAAS | 0.17 | 0.48 | 0.26 | c |
| L50 | 0.745 | 0.097 | 2 | ETAAS | 0.0485 | 1.15 | 1.89 | 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*=√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-36: Total Lead in feed premix
X_Ref = 0.636; U_Ref (k=2 ) = 0.063; σ_p = 0.095 (mg kg-1)
"less than" reported by: L03, L09, L12, L24
(3.28)
Kernel Density Plot
X_ref
Mass fraction (mg kg-1)
Laboratory Code
L02 L35 L41 L32 L10 L45 L48 L31 L22 L20 L39 L07 L27 L04 L38 L26 L46 L42 L17 L23 L05 L06 L16 L21 L44 L36 L13 L49 L40 L37 L50 L14 L11 L29 L47 L15 L28 L25 L08 L30
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 13: Results for Total Tin

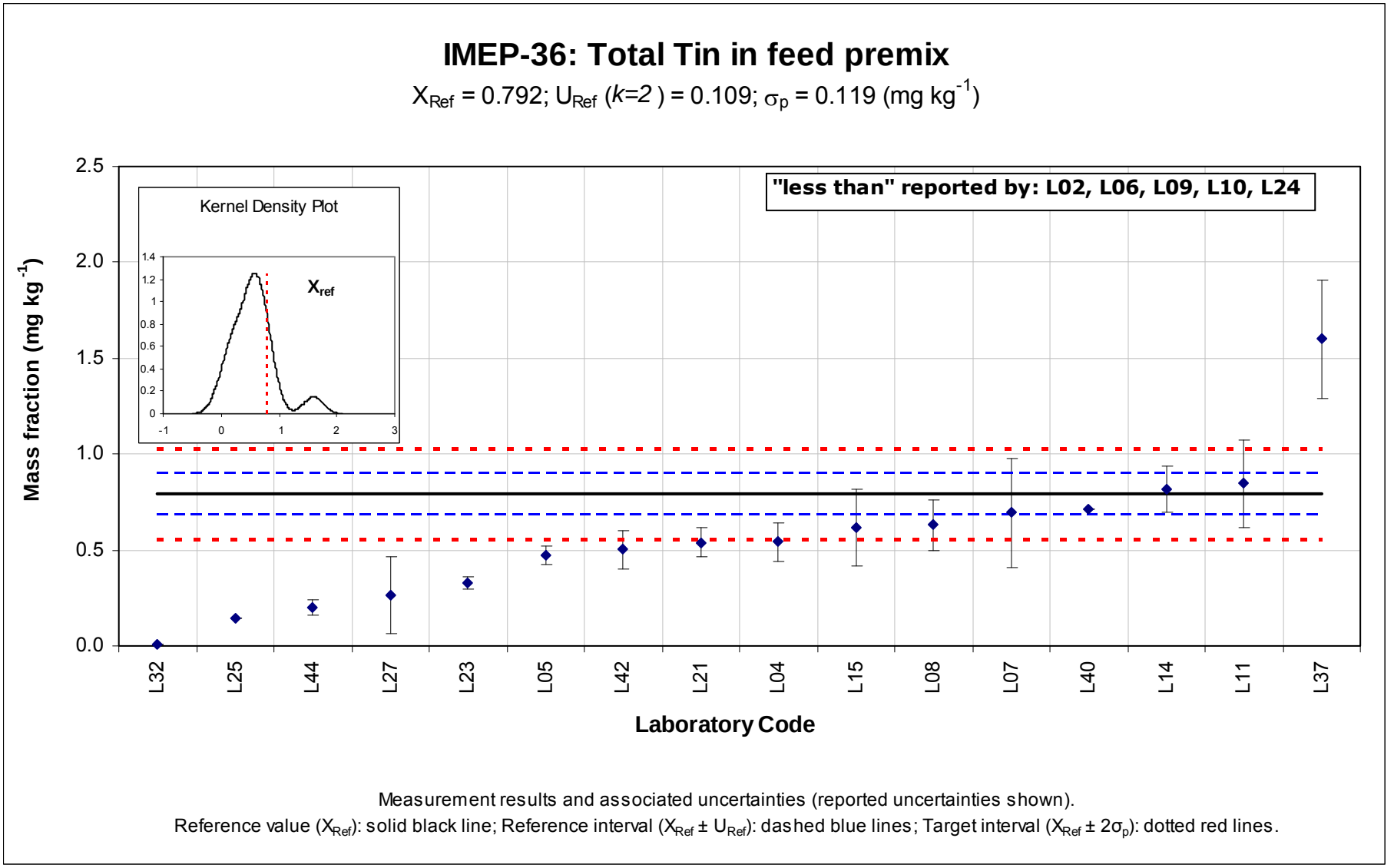
Assigned range: $X_{ref}$ = 0.792, U (k = 2) = 0.109, $\sigma_p$= 0.119 (all values in mg $kg^{-1}$)

| Lab Code | $X_{lab}$ | ± | k[a] | technique | $u_{lab}$ | z-score[b] | ζ-score[b] | uncert.[c] |
|---|---|---|---|---|---|---|---|---|
| L02 | < 0.05 | | | ICP-AES | | | | |
| L04 | 0.542 | 0.103 | 2 | ICP-MS | 0.052 | -2.10 | -3.33 | b |
| L05 | 0.474 | 0.047 | 2 | ICP-MS | 0.024 | -2.67 | -5.35 | b |
| L06 | < 5 | | | ICP-AES | | | | |
| L07 | 0.70 | 0.285 | 2 | ICP-MS | 0.143 | -0.81 | -0.63 | c |
| L08 | 0.63 | 0.13 | 2 | ICP-AES | 0.065 | -1.36 | -1.90 | a |
| L09 | < 2.5 | | | ICP-AES | | | | |
| L10 | < 1 | | | ICP-AES | | | | |
| L11 | 0.848 | 0.229 | 2 | ICP-MS | 0.115 | 0.48 | 0.45 | a |
| L14 | 0.82 | 0.12 | 2 | ICP-MS | 0.060 | 0.24 | 0.35 | a |
| L15 | 0.615 | 0.10 | 1 | ICP-MS | 0.100 | -1.49 | -1.55 | a |
| L21 | 0.54 | 0.09 | 2.35 | ICP-MS | 0.038 | -2.12 | -3.77 | b |
| L23 | 0.331 | 0.033 | 2 | ICP-MS | 0.017 | -3.88 | -8.08 | b |
| L24 | < 1.68 | | | ICP-AES | | | | |
| L25 | 0.144 | 0 | 2 | GF-AAS | | -5.45 | -11.87 | b |
| L27 | 0.263 | 0.201 | 2 | | 0.101 | -4.45 | -4.62 | a |
| L32 | 0.007 | 0.001 | 2 | GF-AAS | 0.001 | -6.61 | -14.38 | b |
| L37 | 1.6 | 0.31 | 2 | ETAAS | 0.155 | 6.81 | 4.92 | c |
| L40 | 0.713 | 0 | | ICP-MS | 0.000 | -0.66 | -1.44 | b |
| L42 | 0.501 | 0.1 | 2 | ICP-MS | 0.050 | -2.45 | -3.93 | b |
| L44 | 0.202 | 0.04 | 2 | ICP-MS | 0.020 | -4.97 | -10.15 | b |

[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** : $u_{lab} > u_{max}$

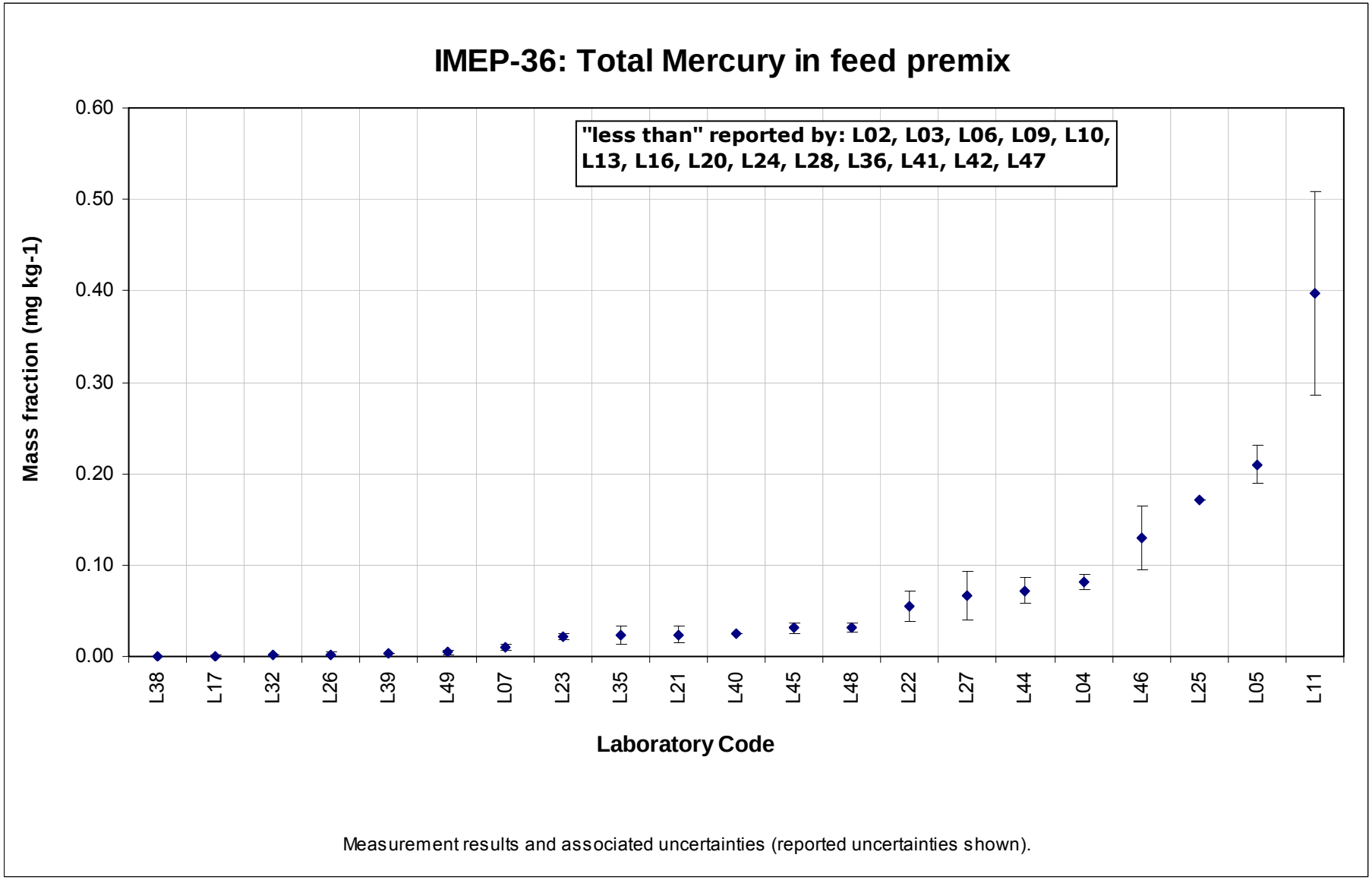



Measurement results and associated uncertainties (reported uncertainties shown).
Reference value ($X_{Ref}$): solid black line; Reference interval ($X_{Ref} \pm U_{Ref}$): dashed blue lines; Target interval ($X_{Ref} \pm 2\sigma_p$): dotted red lines.

# Annex 14: Results for Total Mercury

| Lab Code | $X_{lab}$ | ± | k | technique | $u_{lab}$ |
|---|---|---|---|---|---|
| L02 | < 0.05 | | | ICP-AES | |
| L03 | < 0.01 | | | AAS | |
| L04 | 0.081 | 0.008 | 2 | ICP-MS | 0.004 |
| L05 | 0.21 | 0.021 | 2 | ICP-MS | 0.0105 |
| L06 | < 0.1 | | | CV-AAS | |
| L07 | 0.01 | 0.004 | 2 | ICP-MS | 0.002 |
| L09 | < 0.01 | | | AMA 254 | |
| L10 | < 0.05 | | | CV-AAS | |
| L11 | 0.397 | 0.111 | 2 | ICP-MS | 0.0555 |
| L13 | < 0.025 | | | CV-AAS | |
| L16 | < 0.01 | | | CV-AAS | |
| L17 | 0.0008 | 0.0002 | 2 | AMA 254 | 0.0001 |
| L20 | < 0.05 | | | AMA 254 | |
| L21 | 0.024 | 0.01 | 2.35 | CV-AAS | 0.004255 |
| L22 | 0.055 | 0.017 | 2 | CV-AAS | 0.0085 |
| L23 | 0.022 | 0.003 | 2 | ICP-MS | 0.0015 |
| L24 | < 0.56 | | | ICP-AES | |
| L25 | 0.171 | 0.000 | 2 | HG-AAS | 0.000 |
| L26 | 0.002 | 0.0012 | 1 | AFS | 0.0012 |
| L27 | 0.066 | 0.0264 | 2 | | 0.0132 |
| L28 | < 0.02 | | | CV-AAS | |
| L32 | 0.001 | 0.0005 | 2 | HG-AAS | 0.00025 |
| L35 | 0.0227 | 0.01 | 2 | AFS ? | 0.005 |
| L36 | < 0.05 | | | HG-AAS | |
| L38 | 0.0007 | 0.0001 | 2 | AAS | 0.00005 |
| L39 | 0.00342 | 0.00034 | 2 | CV-AAS | 0.0002 |
| L40 | 0.025 | 0.000 | | ICP-MS | 0.000 |
| L41 | < 0.03 | | | CV-AAS | |
| L42 | < 0.055 | | | ICP-MS | |
| L44 | 0.072 | 0.014 | 2 | ICP-MS | 0.007 |
| L45 | 0.031 | 0.006 | 2 | HG-AAS | 0.003 |
| L46 | 0.13 | 0.03 | √3 | HG-AAS | 0.017 |
| L47 | < 0.02 | | | CV-AFS | |
| L48 | 0.032 | 0.005 | 2 | CV-AAS | 0.003 |
| L49 | 0.0043 | 0.002 | 2 | HG-AAS | 0.001 |

[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 $k=\sqrt{3}$.




Measurement results and associated uncertainties (reported uncertainties shown).

## Annex 15: Experimental details (Annex 8, Question 5)

| Lab code | Did you analyse the sample according to an official method? | sample pre-treatment | digestion step | extraction / separation step |
|---|---|---|---|---|
| L03 | No | addition of nitric acid and $H_2O_2$ | microwave from 80ºC to 220ºC during 22 minuts, and 15 minuts at 220ºC | no |
| L04 | No | none | microwave acid digestion | none |
| L05 | based on SRPS CEN/TS 15621:2009; method is validated because we are using for determination ICP/MS | | | |
| L06 | no | | microwave digestion($HNO_3$/$H_2O_2$ ) | |
| L07 | no | no | Microwave with $HNO_3$ and $H_2O_2$ | na |
| L08 | ISO 15510:2007 | | | |
| L09 | Cd and Pb: EN15510:2007 | Hg, Sn, As: milling, sieve 0.5 mm | Sn: $HNO_3$ as Cd and Pb; Hg: no digestion step; As: reflux digestion using $H_2SO_4$, $HNO_3$ and $H_2O_2$ | not applicable |
| L11 | no | | microwave digestion with nitric acid and hydrochloric acid, EPA method 3051A 2007 | after microwave digestion we carry out a filtration with syrnge filter 0.45um |
| L13 | no | NITRIC ACID AND HYDROGEN PEROXIDE | MICROWAVE 180°C | |
| L14 | no | No pre-treatment | 8 ml of $HNO_3$, 2 ml of $H_2O_2$, 0.1 ml of HF in pressure vessels (microwaves) | not applicable |
| L15 | As: EN 16206:2010 - Cd and Pb: EN 15550:2007 - Hg: EN 16277:2011 - Sn: inhouse-methode comparable to § 64LFGB-methods | | | |
| L16 | VDLUFA III 17.9.1, VDLUFA III 10.8.1, DIN EN 16277 | | | |
| L17 | no | sample weighed and dried in 150°C | dry minaralization | dissolved in acids |

| | | | | |
|---|---|---|---|---|
| L18 | no | Samples were transfered into standard High Density Polyethylene vials. After irradiation no other sample treatment nor transfer of the samples into non irradiated vials was preformed | N.A. | N.A. |
| L20 | no | | | |
| L21 | DINN EN 13805, DIN EN 13806, DIN EN 15763 | | | |
| L22 | yes | | | |
| L23 | no | -1.0% | PRESSURE MICROWAVE DIGESTION FOR AS CD HG PB | ACQUAREGIA ONLY FOR SN |
| L26 | VDLUFA MB VII 2.2.2.5 | | | |
| L27 | NMKL procedure nr 186;2007 | | | |
| L28 | VDLUFA Method Book VII 2.2.2.9 (Hg), 2.2.2.19 (As), 2.2.2.5 (Pb, Cd) | | | |
| L29 | no | no | mineralization by microwave | no |
| L30 | AOAC | he sample is calcined in a furnace for 16 hours | following day and steamers are removed by carbon rsiduos 8 hours, the sample was adicifica with nitric acid and leads back to the muffle | the residue dissolved in hydrochloric acid and filtered and volume with the same acid |
| L31 | no | 0.25 grams of sample | 7 mL of nitric acid and 1 ml of peroxide hydrogen and dilution 1/10 prior injection with ICP/MS | |
| L35 | no | Let the sample take the room temperature, homogeneize it into the frask and weight | for As and Hg wet digestion and for Pb and Cd digestion in furnace | |
| L36 | no | | $HNO_3$ and $H_2O_2$ | |
| L37 | ASU §64 LFGB | | | |

| | | | | |
|---|---|---|---|---|
| L38 | no | Pb i Cd - the samples are ashed at 450 C, As - samples with Mg(NO3)2 are ashed at 550 C. | As - samples are dissolved in 4,5 mol HCl. Thereafter that solutions of KJ and ascorbic acid are added, Pb and Cd- samples are dissolved in 1% HNO3 | |
| L39 | FAAS, CVAAS, HGAAS, ETAAS | | | |
| L40 | DIN EN ISO 17294-2;DIN EN 15763; DIN EN 5961; DIN 38406 E6 | | | |
| L41 | no | none. | 0.5 g of sample + 8 ml HNO3 conc. + 2 ml H2O2. Digestion assisted by microwaves. | Final volume : 25 ml. For Cd determination, a further 8x dilution was indispensable. |
| L42 | NF EN 13805 NF EN15763 NF EN 15765 | | | |
| L44 | EPA 6020 | | | |
| L45 | SR En 14084 | smooth test according to EN 13804 | | |
| L46 | Hungarian Feed Codex | | | |
| L47 | MSZ EN 15550:2008 for Pb and Cd, MTK_2004_III_27 for As and Hg (Codex Pabularis Hungaricus 3. issue) | | | |
| L48 | SR EN ISO 6869/2002 | | | |
| L49 | As DIN EN 16206, Hg DIN EN 16277, Cd u. Pb DIN EN 15550 | | | |
| L50 | no | grinding | microwave digestion; 0,25g sample, 5 ml HNO3 + 1 ml H2O2 | dilution to 10 ml |

European Commission

**EUR 25784 – Joint Research Centre – Institute for Reference Material and Measurements**

Title: IMEP-36: Determination of total Cd, Pb, As, Hg and Sn in feed premixes

Author(s): Ioannis Fiamegkos, Fernando Cordeiro, Piotr Robouch, Håkan Emteborg, Jean Charoud-Got, Hanne Leys, Bibi Kortsen, Beatriz de la Calle

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**Abstract**

This report presents the results of the proficiency test IMEP-36 which focused on the determination of total Cd, Pb, As, Hg and Sn in feed premixes according to Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed. Fifty laboratories from 22 countries registered to the exercise of which 45 reported results and answered the respective questionnaire.

Laboratories were asked to perform two or three independent measurements and to report the mean, the associated uncertainty, the coverage factor of the associated uncertainty and the technique used to perform the measurements. Laboratory results were rated using z- and ζ-scores (zeta-scores) in accordance with ISO 13528. The assigned values ($X_{ref}$) for the measurands were determined as the mean of the values reported by two expert laboratories both of them National Metrology Institutes (NMI).

The results obtained by the participants were optimum in the case of total Cd and less satisfactory for total As and total Pb. For total Sn 16 participants reported results, from which one third scored satisfactorily. Twenty one participants reported results for total Hg although, the expert laboratories reported that the mass fraction for that measurand was below their limit of detection. Hence, no scoring was provided for total Hg.

LA-NA-25784-EN-N

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