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IMEP®

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 (EU-RL-HM). One of its core tasks is to organize interlaboratory comparisons (ILCs) among appointed National Reference Laboratories. This report presents the results of a proficiency test (PT) of the EU-RL-HM which focused on the determination of total Cd, Pb, As, Hg and Sn in feed premixes 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 feed premix which after the appropriate processing was bottled, labelled, numbered accordingly and dispatched to the participants on the 27th of June 2012. Thirty laboratories from 26 countries registered to the exercise of which 25 reported results for total As, 30 for total Cd, 29 for total Pb, 13 for total Hg and 9 for total Sn. 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 analytes investigated.

Between 60 and 90 % of the laboratories reported satisfactory results for total As and total Cd, and 50 % for total Pb. Only 3 participants reported satisfactory results for total Sn (out of 9 laboratories that reported values). Thirteen participants reported results for total Hg although, according to the expert laboratories the mass fraction for that measurand was below their limit of detection.

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1 Introduction

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

IMEP-114 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 "*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-114 was organized to check the analytical capabilities of the National Reference Laboratories to determine low concentrations of total As, Cd, Pb, Hg and Sn in feed premixes.

1.1 Scope

As stated in Regulation N° 882/2004 of the European Parliament and the Council, one of the core duties of the EU-RL-HM is to organise interlaboratory comparisons for the benefit of National Reference Laboratories. The scope of this proficiency test (PT) is to assess the competence of the appointed NRLs to determine the total concentration of Cd, Pb, As, Hg and Sn in feed premixes thereby providing analytical quality assurance in the Member States.

The assessment of the measurement results is performed 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-114

2. Set up of the exercise

The proficiency test was agreed upon by the EU-RL-HM and the Directorate General for Health and Consumers (DG SANCO) when preparing the work program of the EU-RL-HM for 2012. Invitation letters were sent to the participants on the 1st of June 2012 (Annex 1) and a web announcement (Annex 2) was made for the exercise on the IMEP webpage on the same day. The registration deadline was the 15th of June and the samples were dispatched to the participants on the 27th of the same month. The reporting deadline was set to the 7th of September 2012.

2.1 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 Ul kg^{-1} Vitamine A; (E671) 200000 Ul 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-114 and IMEP-36.

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.

2.2 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. 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.

2.3 Distribution

Samples were dispatched to the participants by IRMM on 27th of June 2012. Each participant received:

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

b) A "Confirmation of Receipt" form to be sent back to IRMM after receipt of the test material (Annex 3).

c) A summary of the questionnaire the laboratory would be prompted to answer on-line after reporting the results. (Annex 4)

d) An accompanying letter (Annex 5).

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, Hg and Sn in Feed Premixes 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 6).

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.

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·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 ζ-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 ISO 17043 [13] as follows:

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

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

The 30 laboratories (26 countries) having registered submitted their results and answered the associated questionnaire. 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 no "less than" values were found to be lower than the respective $X_{ref} - U_{ref}$.

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

	Total As	**Total Cd**	**Total Pb**	**Total Sn**	**Total Hg**
Number of participants reported evaluable results	25	30	28	9	13
"less than"	-	-	1	3	10

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 8 to 12, 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 1.

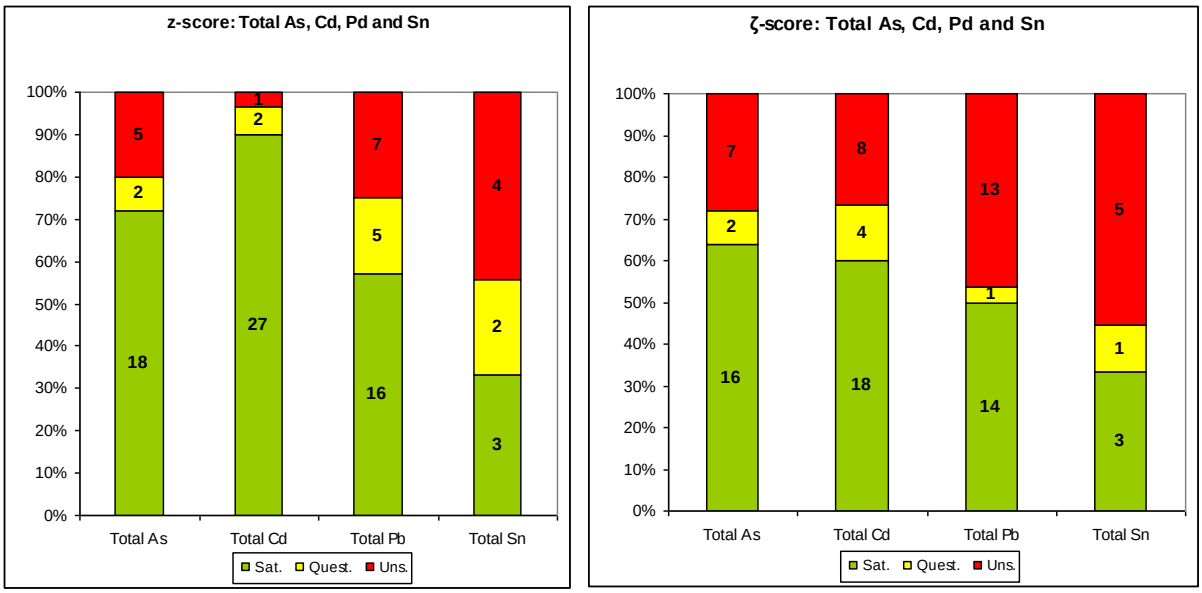

Figure 1: *Number and percentages of laboratories with satisfactory, questionable and unsatisfactory scores. (The numbers on the bars correspond to the exact number of laboratories in a certain scoring category).*

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 ones 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 participants and the certifiers are well above the detection limits of the commonly used methods of analysis.

The results provided by the participants demonstrate that feed premixes proved to be a challenging matrix. The moisture content reported by the participants was of the order of 10.0 ± 3.0 %, ALS reported 11.0 ± 0.2 % (n=30). The overall performance for the results reported for total As (25 participants) and total Cd (30 participants) respectively was adequate. Satisfactory z-scores were obtained by more than 70 and 85% of the participants respectively.

In the case of total Pb, 28 laboratories reported results wherein satisfactory z- and ζ-scores were achieved by almost the half of them. Two thirds of the questionable and/or unsatisfactory results were underestimating 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. Less than one third of the participants reported results, and 3 reported "less than". Among the laboratories that delivered a value for total Sn only three scored satisfactorily, one overestimated the result and the rest (5) underestimated it, as shown in the Kernel density plot (Annex 11). It should also be noted that from the laboratories reporting questionable and/or unsatisfactory results, three 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]. Both effects were evident in the IMEP-114 exercise where two out of the three laboratories scoring satisfactorily have used microwave acid extraction (one with the addition of HF), the third reported to have used an official method (LST EN 15763:2010) which was identified as a pressurized digestion. 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 that fixed the assigned values, were not able to provide results for total Hg in the test item. Nevertheless, a significant number of participants (13) reported values for total Hg ranging from 0.8 to 1.075 µg kg^{-1}. Seven participants did not report a value while 10 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 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).

Fourteen participants performed the analysis following an official method. The information provided by the laboratories about their methods of analysis is summarised in Annex 13. With the exception of total Hg, the influence of the techniques used did not correlate to any of the reported results.

Nineteen laboratories corrected their results for recovery, one did not and 10 did not reply. Those that did, applied one or several of the options shown in Figure 2.

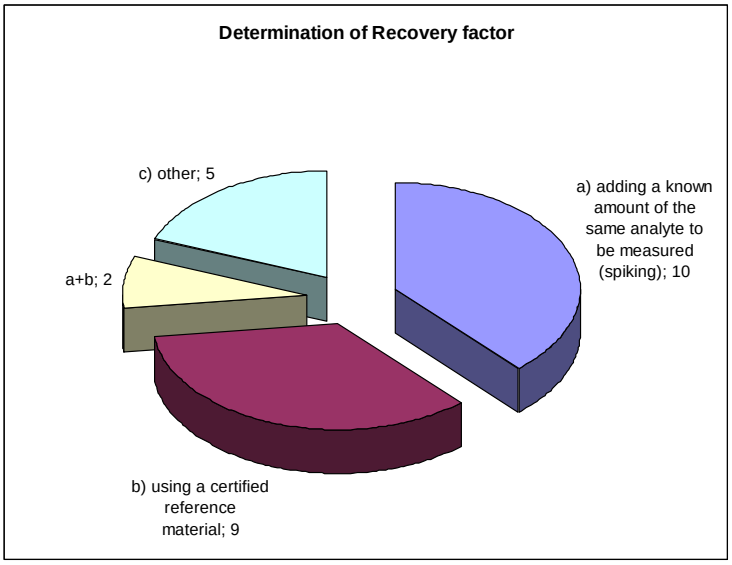

Figure 2: *Distribution of laboratories according to the procedure used to calculate the recovery.*

Sixteen laboratories reported the recovery used to correct their results. The recoveries reported were in the range 70-107.3 %. 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].

Twenty 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 about their usefulness regarding the specific analysis.

All participants but three (L10, L13 and L14) corrected their results for the moisture content, determined using the protocol described in the accompanying letter (Annex 5).

Table 3 - *List of reference materials used by the participants in IMEP-114 for method validation and/or calibration purposes.*

Lab ID	Which reference material?
L01	IMEP 105 *(mineral feed for piglet)*, IMEP 111 *(BCR 032-Moroccan phosphate rock)*
L02	UKZUZ 2011 (3 period)
L03	BCR 463 *(Tuna fish)*
L04	NIST 1547 *(peach leaves)*
L05	BCR 482 *(Lichen)*, BCR 279 *(Sea Lettuce)*
L06	CRM BCR 191 *(Brown bread)*
L07	ZC73012 *(Cabbage)*
L08	LGC 7162 *(Strawberry Leaves)*, Tort 2 *(Lobster hepatopangreas marine RM)*, NIST 1548a *(typical diet)*
L09	IMEP-111 *(BCR 032-Moroccan phosphate rock)*
L10	NIST Rice Flour, NIST Wheat Flour, NIST Pine Needles
L12	LICHEN 482 – BCR, TEA CBW 10016; TOMATO LEAVES 1573a NIST
L14	BCR 463 *(Tuna fish)*, CRM 2976 *(Mussel tissue)*, IMEP 111 *(BCR 032-Moroccan phosphate rock)*, BCR 151 *(Skim milk powder)*
L17	Natural moraccan phosphate Rock BCR 032 and Trace Elements in Multi Element-Nutrient Fertilizer SRM 695
L18	IRMM 804 *(Rice flour)* because we have no feed reference material available in the lab (we do not analyse feed on a regular basis)
L21	ERM-CD281 *(Rye grass)*
L22	CRM CZ 9003 (1N), CZ 9089 MIX 009 *(Certified Reference Solutions)*
L24	internal reference, IRMM
L25	SRM 3280 Multivitamin Tablets
L26	IMEP 105 (mineral feed test material) , IMEP 110 (NIST SRM 1570a - spinach leaves), FAPAS T07105 (chilli powder)
L27	Old proficiency material
L28	CPAchem multi-element 10 mg/l *(Certified Reference Solution)*
L29	Oyster Tissue, Tort-2, Bovine Liver
L30	BCR-279 (sea lettuce), NIST 1570A (spinach leaves)

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

Various approaches were used to scrutinise the measurement uncertainty (Table 4). Twenty one laboratories usually report uncertainty to their customers while 9 do not.

When asked about the level of confidence covered by the reported coverage factor (*k*), 25 participants reported 95 % and one reported 99 %.

Regarding the experience of the laboratories with this kind of analysis 27 participants answered that they carry out this type of analysis on a regular basis while three do not. The distribution in terms of number of analysis per year is shown in Figure 3.

Table 4 - *Approaches used by the participants in IMEP-114 to estimate the uncertainty of their measurements.*

Approach followed for uncertainty calculation	Number of labs.
Uncertainty budget calculated according to ISO-GUM	6
Known uncertainty of the standard method	4
Uncertainty of the method as determined by in-house validation	16
Measurement of replicates (i.e. precision)	14
Estimation based on judgement	2
Use of intercomparison data	2
Other	2

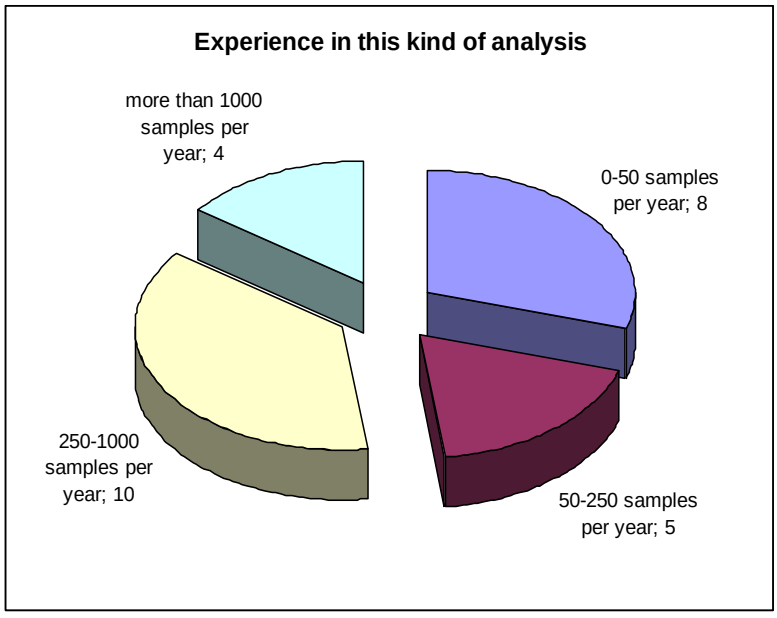

Figure 3*: Participants' experience in this type of analysis expressed as number of analysis per year.*

All participants stated that they have a quality system in place based on ISO 17025. In two cases the quality system is also based on ISO 9000. Twenty-one participants are accredited for the methods of analysis used in this exercise, although a number of laboratories indicated that they are not accredited for Sn analysis (Table 5). Most of the laboratories regularly take part in PTs (23 out 30). Finally, table 5 summarises the comments of the participants regarding the IMEP-114 exercise.

Table 5 - *Comments of the laboratories participating in the IMEP-114 ILC.*

Lab ID	Comments
L02	Results were not corrected for recovery, because we do not do it in routine sample analysis.
L03	Our laboratory is accredited for Hg analysis and Cd/Pb method is under final validation.
L08	This Laboratory not yet accredited for Tin analyses. It is not accredited for Feed Premixes, only Food matrices. Analytical problems were encountered because of high quantity of insoluble matter in the acid digested sample which resulted in further difficulties with Arsenic and Mercury analyses.
L08	This Laboratory not yet accredited for Tin analyses. It is not accredited for Feed Premixes, only Food matrices. Analytical problems were encountered because of high quantity of insoluble matter in the acid digested sample which resulted in further difficulties with Arsenic and Mercury analyses.
L12	Pb, Cd and As is not accredited: validation in progress
L15	We don´t correct the results for recovery
L18	The method we used is only validated and used on a regular basis for food matrices. This SOP is not validated for feed
L20	We have send the material to a subcontracter for analysis of As, Cd, Pb and Hg. The aim was to check the subcontracter.
L22	Determination of Sn is not accredited because our laboratory do not provide this deteremination in feed.
L28	Our laboratory is accredited for food matrix, we don't analysis feed matrix

5 Conclusions

From the results obtained for the IMEP-114 exercise, 60 and 90 % of the participating laboratories reported satisfactory results for total As and total Cd. Total Pb proved to be a difficult measurand since less satisfactory results were obtained with an obvious trend to underestimation of the assigned value. Only one third of the participants reported results for total Sn from which only 33 % did it satisfactorily. The digestion method is evidently influencing the Sn recovery from the matrix. It is not unusual that tin forms insoluble tin oxide especially if only digested with HNO_3. Thirteen 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 methods used. Interference by tungsten oxide could be the explanation for those high results. NRLs are advised to have a look at the report of the workshop organized by the EU-RL-HV in 2010, when a training on interferences on trace element determination by ICP-MS was given.

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/or 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 in the following table are kindly acknowledged.

Table - 6: *The laboratories participated in IMEP-114 and their respective countries of origin.*

Organisation	Country
AGES GmbH	AUSTRIA
CODA-CERVA	BELGIUM
BFSA CLVCE	BULGARIA
State Veterinary Institute Olomouc	CZECH REPUBLIC
CISTA	CZECH REPUBLIC
Danish Veterinary and Food Administration Chemical Laboratory	DENMARK
AGRICULTURAL RESEARCH CENTRE	ESTONIA
Veterinary and Food Laboratory	ESTONIA
Finnish Food Safety Authority Evira	FINLAND
Laboratoire SCL de Bordeaux	FRANCE
Federal Office of Consumer Protection and Food Safety (BVL)	GERMANY
GENERAL CHEMICAL STATE LABORATORY	GREECE
REGIONAL CENTRE OF PLANT PROTECTION & QUALITY CONTROL OF MAGNISIA	GREECE
National Food ChainSafety Office, Food and Feed Safety Directorate	HUNGARY
National Food Chain Safety Office	HUNGARY
HEALTH SERVICE EXECUTIVE	IRELAND
Istituto Zooprofilattico Sperimentale del Piemonte Liguria e Valle d'Aosta	ITALY
Institute of Food Safety, Animal Health and Environment	LATVIA
National food and veterinary risk assessment institute	LITHUANIA
Public Health Laboratory	MALTA
RIKILT	NETHERLANDS
NIFES	NORWAY
National Veterinary Research Institute	POLAND
Laboratório Nacional de Investigação Veterinária	PORTUGAL
HYGIENE AND VETERINARY PUBLIC HEALTH INSTITUTE	ROMANIA
State Veterinary and Food Institute	SLOVAKIA
National Veterinary Institute	SLOVENIA
Laboratorio Arbitral Agroalimentario	SPAIN
National Veterinary Institute	SWEDEN
LGC Ltd	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

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
EU reference laboratory for heavy metals in feed and food

European Union Reference Laboratory
Heavy Metals in Feed and Food

Geel, 1 June 2012
JRC.D6/BCa/bk/ARES(2012)

«Title» «M_1st_name» «last_name»
«Institute»
«Department»
«Address»
«ZIP» «City»
«COUNTRY»

Dear Madam / Sir,

Inter-laboratory comparison for EU-RL Heavy Metals in Feed and Food

On behalf of the EU-RL Heavy Metals in Feed and Food, I would like to invite you to participate in the Proficiency Test IMEP-114 for the **"Determination of total Cd, Pb, As, Hg and Sn in mineral feed pre-mixes"**.

I would like to remind you that – according to Regulation (EC) No 882/2004 - you have the duty as NRL to participate in PTs organised by the EU-RL-HM if you hold a mandate for the type of matrix investigated.

Please register electronically for this inter-laboratory comparison using the following link:
https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=920

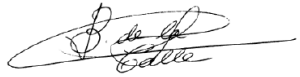
Your participation is free of charge.

Once you have submitted your registration electronically, please follow the procedure indicated: a) print your registration form; b) sign it; and c) fax it to us. **Your fax is the confirmation of your participation.**

The **deadline for registration is 15 June 2012**. Samples will be sent to participants during the second half of June. The deadline for submission of results is 7 September 2012.

I am the project leader for this inter-laboratory comparison. In case of questions/doubts, do not hesitate to contact me.

Yours sincerely



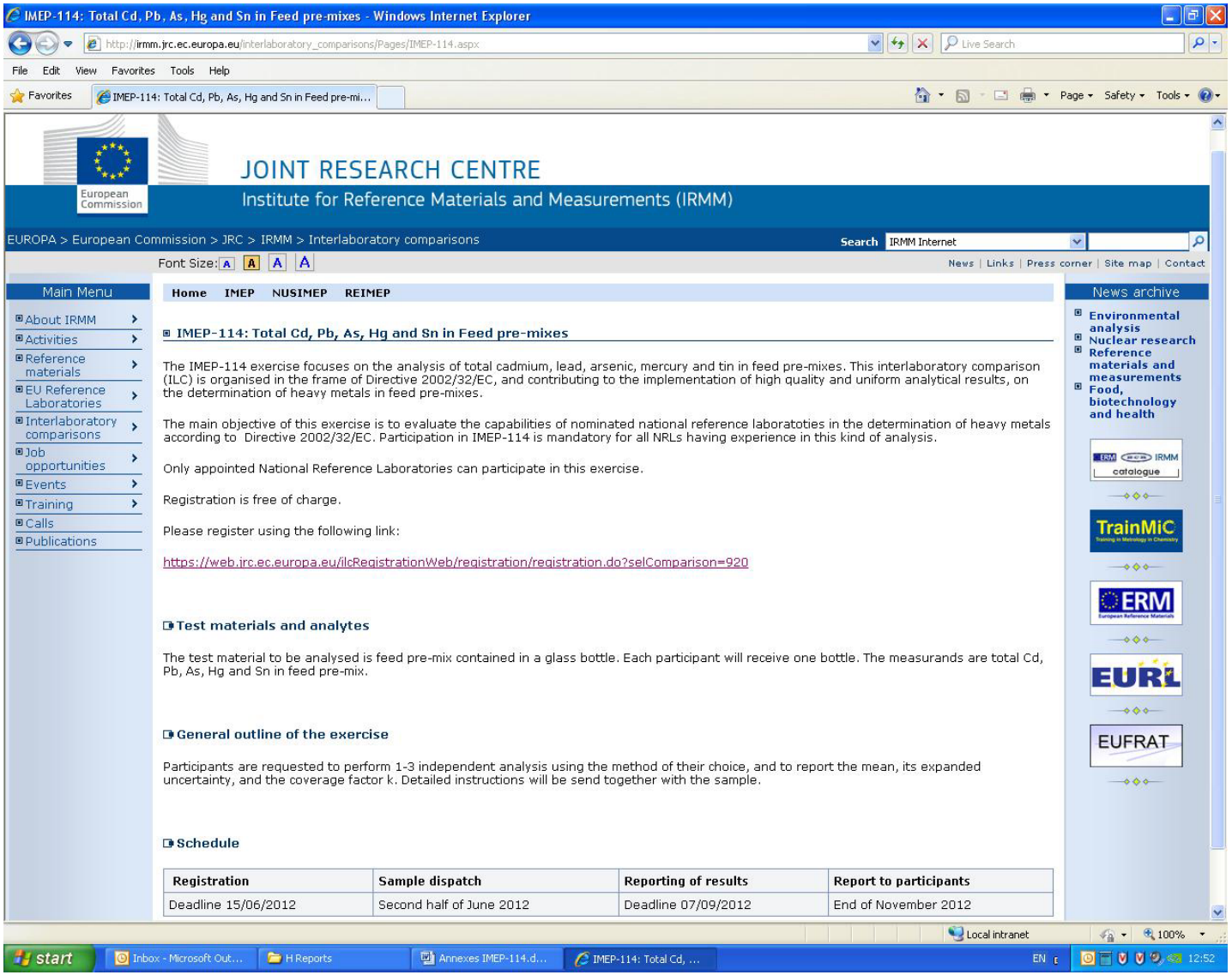
Dr. M.B. de la Calle
Operating Manager EU-RL-HM

Cc: Franz Ulberth

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 299. Fax: (32-14) 571 865.

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

Annex 2 : IRMM – IMEP web announcement



Annex 3 : Acknowledgment of receipt form:

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
EU reference laboratory for heavy metals in feed and food

European Union Reference Laboratory
Heavy Metals in Feed and Food

Annex to
JRC.D5/BDC/bk/ARES(2012)/772941

«TITLE» «FIRSTNAME» «SURNAME»
«ORGANISATION»
«DEPARTMENT»
«ADDRESS»
«ADDRESS2»
«ADDRESS3»
«ADDRESS4»
«ZIP» «TOWN»
«COUNTRY»

EU-RL-HM-14 / IMEP-114

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-114 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. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 299. Fax: (32-14) 571 865.

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

Annex 4 : Summary of the Questionnaire

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
Food Safety & Quality

European Union Reference Laboratory
Heavy Metals in Feed and Food

Annex to JRC. D5/BDC/bk/ARES(2012)/772941

FOR INFORMATION ONLY – SUMMARY QUESTIONNAIRE IMEP-114

- Which are the correction factors for Total Cd, Pb, As, Hg and Sn.
- How did you determine the recovery factor (R)?
- What is the level of confidence (in %) reflected by the coverage (k) given by your results?
- What is the basis of your uncertainty estimate (multiple answers are possible)?
- 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 an interlaboratory comparison for this type of analysis on a regular basis.
- Does your laboratory use a reference material for this type of analysis?
- Do you have any comments? Please let us know: …

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

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 687. Fax: (32-14) 571 865.

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

IMEP

Annex 5 : Sample accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
EU reference laboratory for heavy metals in feed and food

European Union Reference Laboratory
Heavy Metals in Feed and Food

Geel, June 2012
JRC.D05/BDC/bk/ARES(2012)772941

«TITLE» «FIRSTNAME» «SURNAME»
«ORGANISATION»
«DEPARTMENT»
«ADDRESS»
«ADDRESS2»
«ADDRESS3»
«ADDRESS4»
«ZIP» «TOWN»
«COUNTRY»

Participation in IMEP-114, 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-114 interlaboratory comparison for the determination of **Total Cd, Pb, Hg, As and Sn in Feed Pre-mixes.**

This exercise takes place in the frame of the EU-RL Heavy Metals in Feed and Food.

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.
e) 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 Pre-mixes.**

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

«PARTKEY»

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 252. Fax: (32-14) 571 865.

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

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:

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.*

Note 1: perform the measurements of the water content in triplicate.
*Note 2: **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 per measurand. Correct the measurement results for recovery and moisture content, and report on the reporting website:
- the **corrected mean** of your two or three measurement results,
- the associated expanded **uncertainty**,
- 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. 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: **«PARTKEY»**. 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 07/09/2012.

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-114 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

Cc: F. Ulberth

Annex 6 : Questionnaire

Submission Form

1.

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. **How did you determine the recovery factor (R)? By:**

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

2.1. If other, please specify:

3. **What is the level of confidence (in %) reflected by the coverage (k) given by your results?**

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

- ☐ 1. uncertainty budget calculated according to iso-gum
- ☐ 2. known uncertainty of the standard method
- ☐ 3. uncertainty of the method as determined in-house validation
- ☐ 4. measurement of replicates (i.e. precision)
- ☐ 5. estimation based on judgement
- ☐ 6. use of intercomparison data
- ☐ 7. other

4.1. If other, please specify

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

- ○ no
- ○ yes

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

- ○ no
- ○ yes

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

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

7. **Did you analyse the sample according to an official method?**

- ○ no
- ○ yes

7.1. If Yes, which:

7.2. If no, please describe (in max. 150 characters for each reply) your

7.2.1. sample pre-treatment

7.2.2. digestion step

7.2.3. extraction / separation step

7.2.4. instrument calibration step

8. **Does your laboratory carry out this type of analysis (as regards the anlytes, matrix and methods) on a regular basis?**

- ○ no
- ○ yes

8.1. If Yes, please estimate the number of samples (As, Cd, Pb, Hg, Cu measurements together):

- ○ a) 0-50 samples per year
- ○ b) 50-250 samples per year
- ○ c) 250-1000 samples per year
- ○ d) more than 1000 samples per year

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

- ○ no
- ○ yes

9.1. If Yes, which:

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

9.1.1. If other, please specify

10. **Is your laboratory accredited for this type of analysis?**

- ○ No
- ○ Yes

11. **Does your laboratory take part in an interlaboratory comparison for this type of analysis on a regu**

- ○ no
- ○ yes

11.1. If yes, which one(s)

12. **Does your laboratory use a reference material for this type of analysis?**

- ○ no
- ○ yes

12.1. If yes, which one(s)

12.2. Is the material used for the validation of procedures?

- ○ no
- ○ yes

12.3. Is the material used for calibration of instruments?

- ○ no
- ○ yes

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

Annex 7 : Homogeneity and stability studies

7.1 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	

7.2 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

7.3 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	

7.4 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

7.5 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	

7.6 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

7.7 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	

7.8 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 8 : Results for Total Arsenic

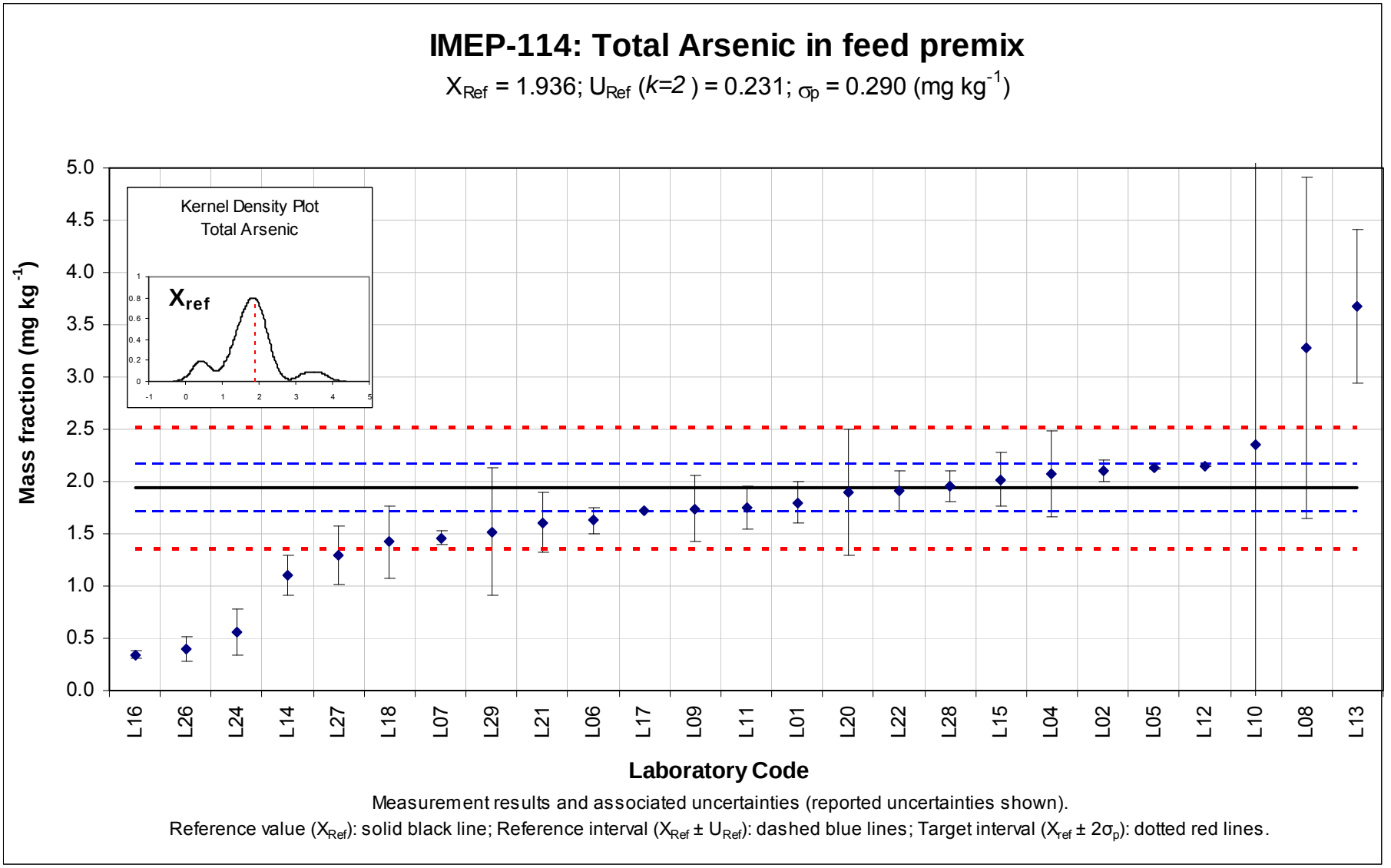
Assigned range: X_{ref} = 1,936, U (k = 2) = 0.231, $\hat{\sigma}$ = 0.290 (all values in mg kg^{-1})

Lab Code	X_{lab}	±	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L01	1.80	0.20	0.95	HG-AAS	0.211	-0.47	-0.56	a
L02	2.100	0.1	2	ICP-MS	0.050	0.57	1.30	b
L04	2.07	0.41	2	ICP-MS	0.205	0.46	0.57	a
L05	2.13	0.0085	2	ETAAS	0.004	0.67	1.68	b
L06	1.626	0.130	2	HG-AAS	0.065	-1.07	-2.33	b
L07	1.46	0.07	2	ICP-MS	0.035	-1.64	-3.93	b
L08	3.275	1.63	2	ICP-MS	0.815	4.61	1.63	c
L09	1.74	0.32	2	HG-AAS	0.160	-0.67	-0.99	a
L10	2.36	30	√3	ICP-MS	17.321	1.46	0.02	c
L11	1.75	0.205	2	ICP-MS	0.103	-0.64	-1.20	b
L12	2.14	0.00	√3	ICP-MS	0.000	0.70	1.77	b
L13	3.6756	0.7351	√3	ICP-MS	0.424	5.99	3.96	c
L14	1.102	0.188	2	HG-AAS	0.094	-2.87	-5.59	b
L15	2.02	0.26	2	ICP-MS	0.130	0.29	0.49	a
L16	0.35	0.04	2	HG-AAS	0.018	-5.48	-13.59	b
L17	1.72	0.006	2	ICP-MS	0.003	-0.74	-1.86	b
L18	1.42	0.34	2	ICP-MS	0.170	-1.78	-2.51	a
L20	1.90	0.6	2	ICP-MS	0.300	-0.12	-0.11	c
L21	1.61	0.28	2	ICP-MS	0.140	-1.12	-1.79	a
L22	1.91	0.19	2	ICP-MS	0.095	-0.09	-0.17	b
L24	0.56	0.22	2	ETAAS	0.110	-4.74	-8.61	b
L26	0.40	0.12	2	ETAAS	0.060	-5.29	-11.78	b
L27	1.29	0.28	2	HG-AAS	0.140	-2.22	-3.55	a
L28	1.957	0.15	2	ICP-MS	0.075	0.07	0.16	b
L29	1.52	0.608	2	ICP-MS	0.304	-1.43	-1.28	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-114: Total Arsenic in feed premix
X_{Ref} = 1.936; U_{Ref} (*k*=2) = 0.231; σ_p = 0.290 (mg kg^{-1})
Kernel Density Plot
Total Arsenic
X_{ref}
Mass fraction (mg kg^{-1})
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
L16
L26
L24
L14
L27
L18
L07
L29
L21
L06
L17
L09
L11
L01
L20
L22
L28
L15
L04
L02
L05
L12
L10
L08
L13
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 9 : Results for Total Cadmium

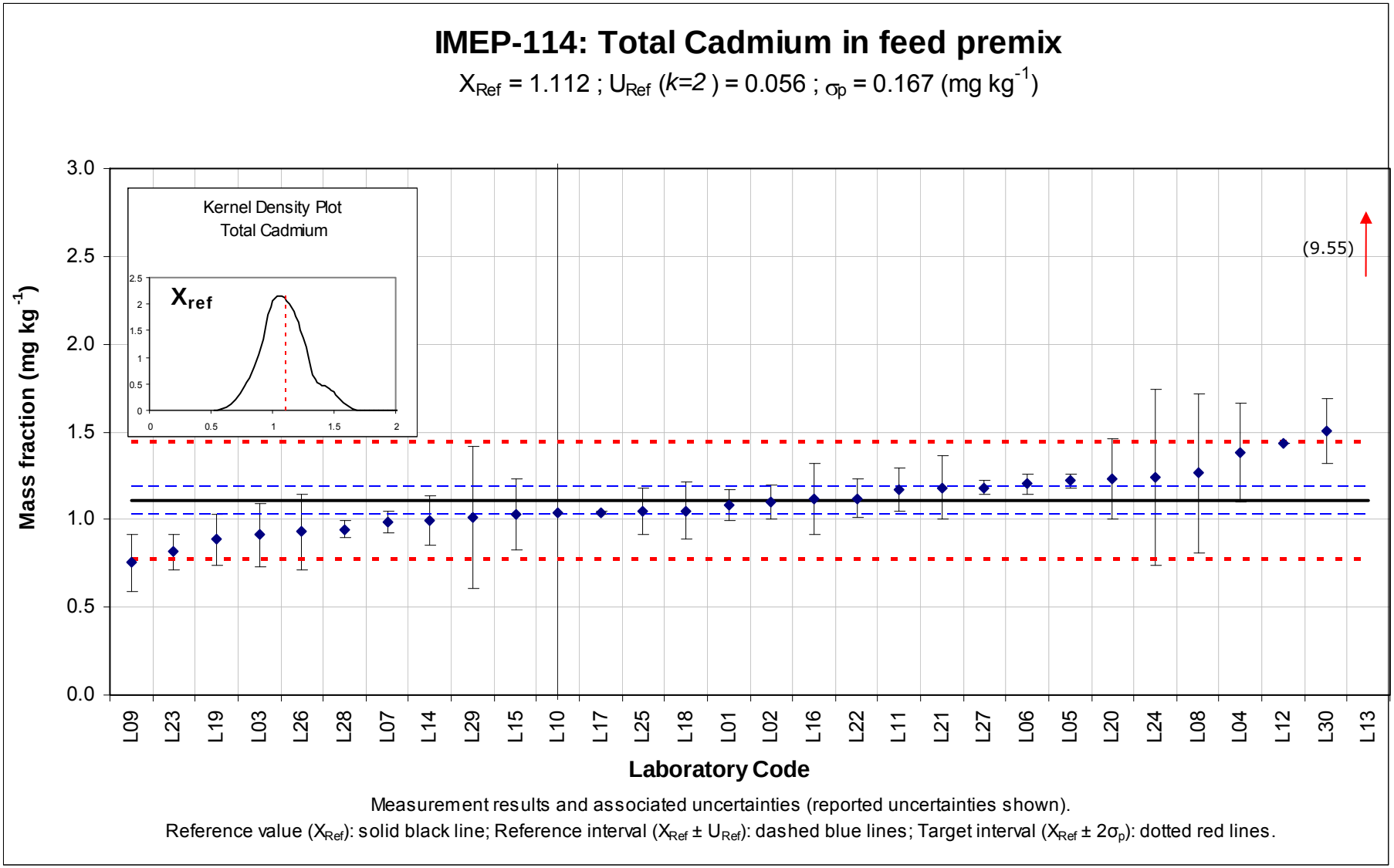
Assigned range: X_{ref} = 1,112, U (k = 2) = 0.056, $\hat{\sigma}$ = 0.167 (all values in mg kg^{-1})

Lab Code	X_{lab}	±	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L01	1.08	0.09	0.94	ETAAS	0.096	-0.19	-0.32	a
L02	1.1	0.1	2	ICP-MS	0.050	-0.07	-0.20	a
L03	0.911	0.182	2	FAAS	0.091	-1.20	-2.11	a
L04	1.38	0.28	2	ICP-MS	0.140	1.61	1.88	a
L05	1.22	0.042	2	ETAAS	0.021	0.65	3.11	b
L06	1.202	0.055	2	ETAAS	0.028	0.54	2.31	b
L07	0.987	0.059	2	ICP-MS	0.030	-0.75	-3.07	a
L08	1.263	0.45	2	ICP-MS	0.225	0.91	0.67	c
L09	0.753	0.166	2	ICP-AES	0.083	-2.15	-4.09	a
L10	1.04	30	√3	ICP-MS	17.321	-0.43	0.00	c
L11	1.17	0.12	2	ICP-MS	0.060	0.35	0.88	a
L12	1.43	0.00	√3	ICP-MS	0.000	1.91	11.44	b
L13	9.5465	1.9093	√3	ICP-MS	1.102	50.59	7.65	c
L14	0.996	0.142	2	ETAAS	0.071	-0.69	-1.51	a
L15	1.03	0.2	2	ICP-MS	0.100	-0.49	-0.79	a
L16	1.119	0.201	2	ETAAS	0.101	0.04	0.07	a
L17	1.04	0.007	2	ICP-MS	0.004	-0.43	-2.55	b
L18	1.05	0.16	2	ICP-MS	0.080	-0.37	-0.73	a
L19	0.887	0.145	2	ETAAS	0.073	-1.35	-2.89	a
L20	1.23	0.23	2	ICP-MS	0.115	0.71	1.00	a
L21	1.18	0.18	2	ICP-MS	0.090	0.41	0.73	a
L22	1.12	0.11	2	ICP-MS	0.055	0.05	0.14	a
L23	0.816	0.100	2	ETAAS	0.050	-1.77	-5.16	a
L24	1.24	0.50	2	ETAAS	0.250	0.77	0.51	c
L25	1.047	0.13	2	ICP-MS	0.065	-0.39	-0.91	a
L26	0.930	0.214	2	ETAAS	0.107	-1.09	-1.64	a
L27	1.18	0.04	2	ETAAS	0.020	0.41	2.00	b
L28	0.943	0.050	2	ICP-MS	0.025	-1.01	-4.50	b
L29	1.01	0.404	2	ICP-MS	0.202	-0.61	-0.50	c
L30	1.503	0.185	2	AAS	0.093	2.35	4.05	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** : $u_{lab} > u_{max}$



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 Lead

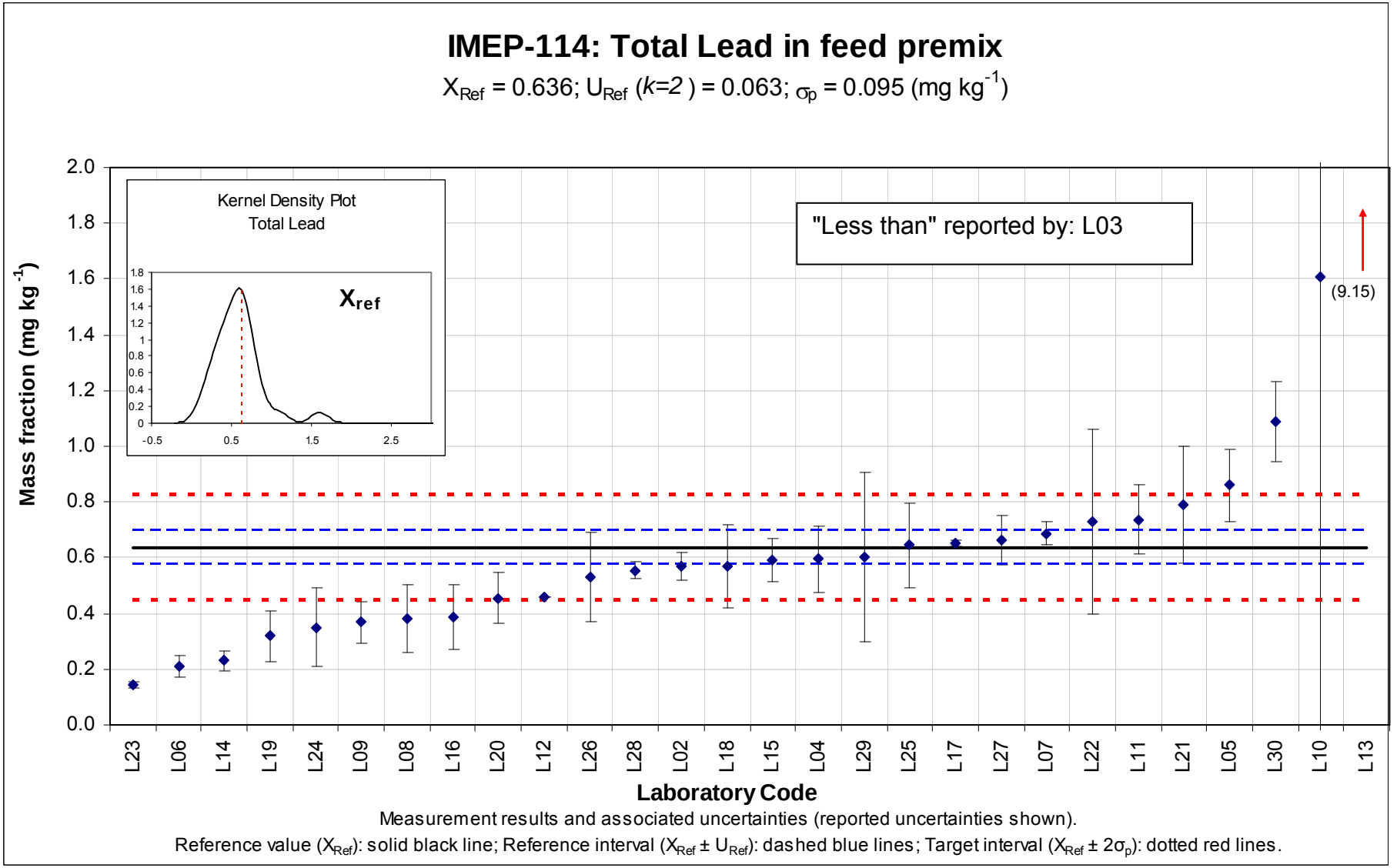
Assigned range: X_{ref} = 0.636, U (k = 2) = 0.063, $\hat{\sigma}$ = 0.095 (all values in mg kg^{-1})

Lab Code	X_{lab}	±	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L02	0.57	0.05	2	ICP-MS	0.025	-0.69	-1.63	b
L03	< 1			FAAS				
L04	0.594	0.119	2	ICP-MS	0.060	-0.44	-0.62	a
L05	0.86	0.13	2	ETAAS	0.065	2.36	3.11	a
L06	0.210	0.037	2	ETAAS	0.019	-4.46	-11.64	b
L07	0.687	0.041	2	ICP-MS	0.021	0.54	1.37	b
L08	0.381	0.12	2	ICP-MS	0.060	-2.67	-3.75	a
L09	0.369	0.074	2	ETAAS	0.037	-2.80	-5.48	a
L10	1.61	25	√3	ICP-MS	14.434	10.22	0.07	c
L11	0.737	0.124	2	ICP-MS	0.062	1.06	1.46	a
L12	0.46	0.000	√3	ICP-MS	0.000	-1.84	-5.57	b
L13	9.1540	1.8308	√3	ICP-MS	1.057	89.36	8.06	c
L14	0.231	0.036	2	ETAAS	0.018	-4.24	-11.14	b
L15	0.59	0.078	2	ICP-MS	0.039	-0.48	-0.91	a
L16	0.386	0.116	2	ETAAS	0.058	-2.62	-3.78	a
L17	0.652	0.010	2	ICP-MS	0.005	0.17	0.52	b
L18	0.57	0.15	2	ICP-MS	0.075	-0.69	-0.81	a
L19	0.319	0.091	2	ETAAS	0.046	-3.32	-5.72	a
L20	0.454	0.092	2	ICP-MS	0.046	-1.90	-3.25	a
L21	0.79	0.21	2	ICP-MS	0.105	1.62	1.41	c
L22	0.73	0.33	2	ICP-MS	0.165	0.99	0.56	c
L23	0.143	0.013	2	ETAAS	0.007	-5.17	-15.30	b
L24	0.35	0.14	2		0.070	-3.00	-3.72	a
L25	0.644	0.15	2	ICP-MS	0.075	0.09	0.10	a
L26	0.53	0.16	2	ETAAS	0.080	-1.11	-1.23	a
L27	0.663	0.09	2	ETAAS	0.045	0.29	0.50	a
L28	0.554	0.030	2	ICP-MS	0.015	-0.85	-2.33	b
L29	0.603	0.302	2	ICP-MS	0.151	-0.34	-0.21	c
L30	1.088	0.142	2	AAS	0.071	4.75	5.82	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-114: Total Lead in feed premix
X_Ref = 0.636; U_Ref (k=2) = 0.063; σp = 0.095 (mg kg-1)
Kernel Density Plot
Total Lead
Xref
"Less than" reported by: L03
(9.15)
Mass fraction (mg kg-1)
Laboratory Code
L23 L06 L14 L19 L24 L09 L08 L16 L20 L12 L26 L28 L02 L18 L15 L04 L29 L25 L17 L27 L07 L22 L11 L21 L05 L30 L10 L13
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 ± 2σp): dotted red lines.

Annex 11 : Results for Total Tin

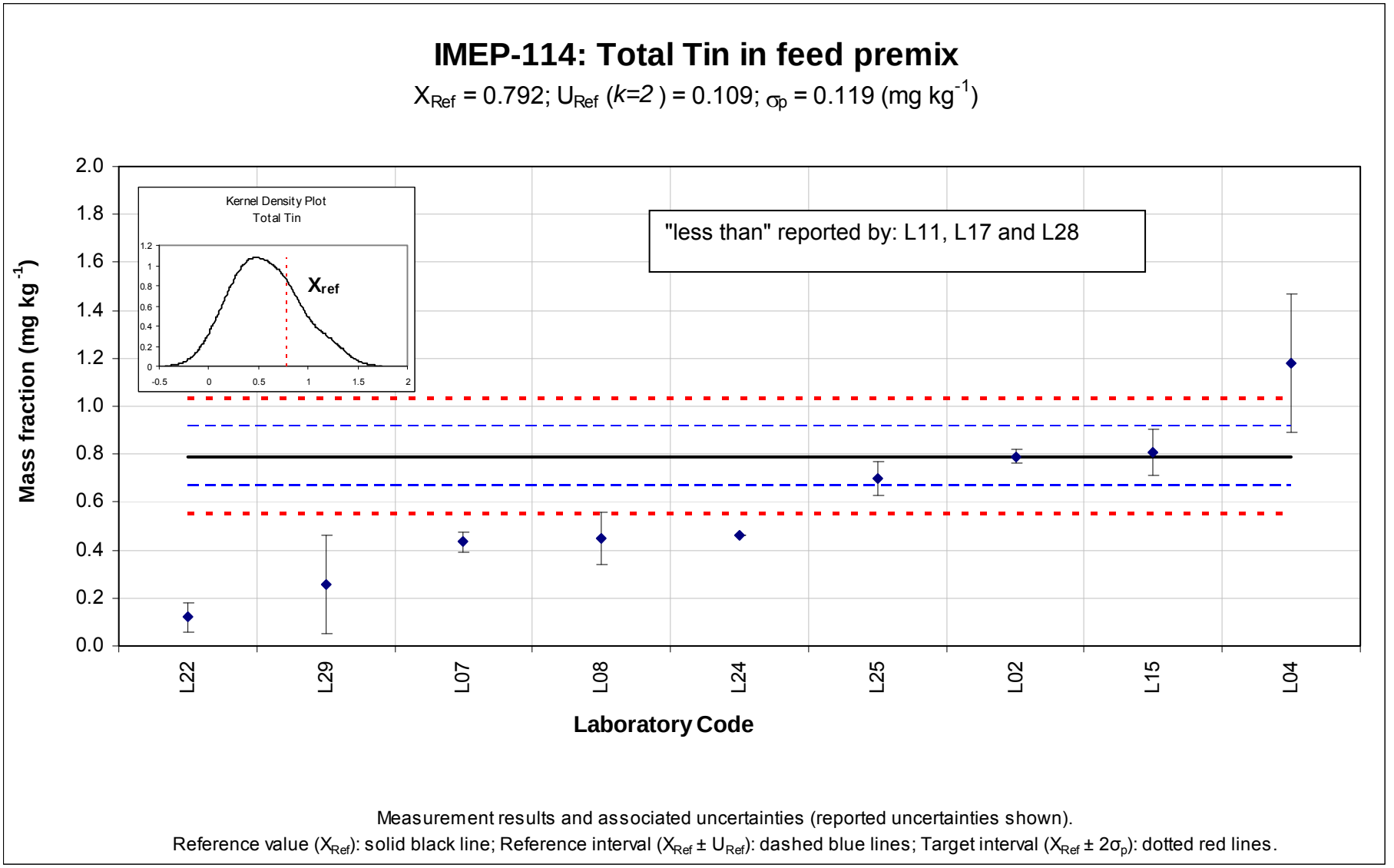
Assigned range: X_{ref} = 0.792, U (k = 2) = 0.109, $\hat{\sigma}$ = 0.119 (all values in mg kg^{-1})

Lab Code	X_{lab}	±	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L02	0.79	0.03	2	ICP-MS	0.015	-0.01	-0.03	b
L04	1.18	0.29	2	ICP-MS	0.145	3.27	2.51	c
L07	0.434	0.043	2	ICP-MS	0.022	-3.01	-6.10	b
L08	0.447	0.11	2	ICP-MS	0.055	-2.90	-4.45	a
L11	< 1			ICP-MS				
L15	0.81	0.097	2	ICP-MS	0.049	0.16	0.25	b
L17	< 3.2			ICP-AES				
L22	0.12	0.06	2	ICP-MS	0.030	-5.66	-10.79	b
L24	0.46	0.000	√	ICP-AES	0.000	-2.79	-6.08	b
L25	0.700	0.07	2	ICP-MS	0.035	-0.77	-1.41	b
L28	< 2			ICP-MS				
L29	0.256	0.205	2	ICP-MS	0.103	-4.51	-4.61	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** : $u_{lab} > u_{max}$

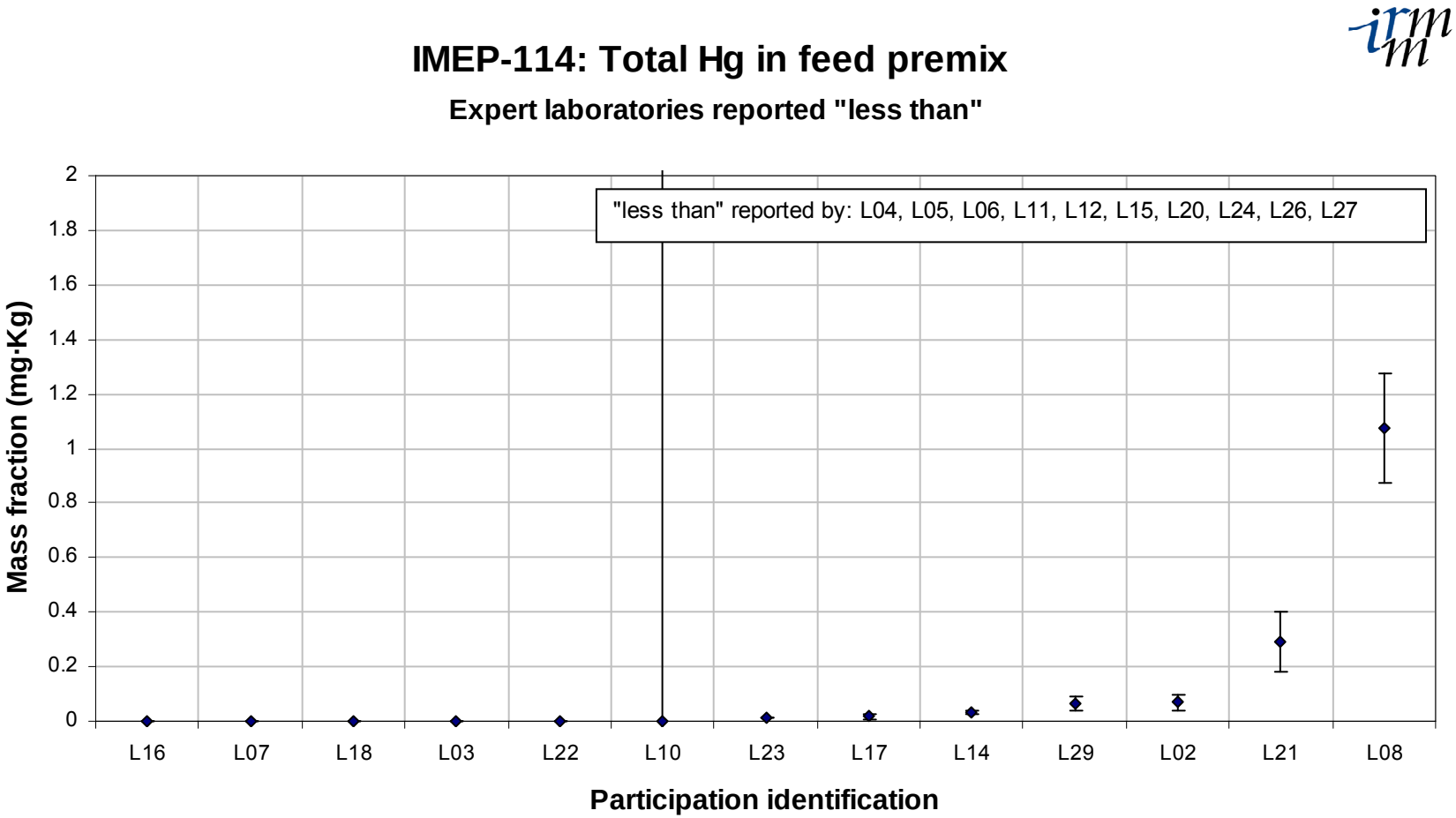


Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{Ref}): solid black line; Reference interval ($X_{Ref} \pm U_{Ref}$): dashed blue lines; Target interval ($X_{Ref} \pm 2\sigma_p$): dotted red lines.

Annex 12 : Results for Total Mercury

Lab Code	X_{lab}	±	k[a]	technique	u_{lab}
L02	0.07	0.03	2	ICP-MS	0.015
L03	0.0016	0.00006	2	AMA 254	0.000
L04	< 0.004			CV-AAS	
L05	< 0.005			CV-AFS	
L06	< 0.05			CV-AAS	
L07	0.0008	0.0002	2	AMA	0.000
L08	1.075	0.20	2	ICP-MS	0.100
L10	0.003	20	√3	AMA254	11.547
L11	< 0.005			CV-AFS	
L12	< 0.034			TDA-AAS	
L14	0.034	0.0059	2	CV-AAS	0.003
L15	< 0.01			Direct Mercury Analyzer	
L16	0.0008	0.0001	2	AAS - AMA 254	0.000
L17	0.0166	0.011	2	ICP-MS	0.006
L18	0.0012	0.0003	2	AMA	0.000
L20	< 0.01			ICP-MS	
L21	0.29	0.11	2	ICP-MS	0.055
L22	0.002	0.001	2	AMA-254	0.001
L23	0.013	0.002	2	CV-AAS	0.001
L24	< 0.01			AMA 254	
L26	< 0.01			CV-AAS	
L27	< 0.028			CV-AAS	
L29	0.064	0.026	2	ICP-MS	0.013

[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 $k=\sqrt{3}$.



This graph displays all measurement results and their associated uncertainties. The uncertainties are shown as reported.

Annex 13: Experimental details (Annex 6, Question 7)

Lab ID	Official Method	Sample pretreatment	Digestion	Extraction/Separation	Instrument calibration
L01	no	Open wet digestion in HNO3 for Cd, dry ashing for As			External nonlinear
L02	LST EN 15763:2010				
L03	EN 14082				
L04	yes	not nessesary	Microwave Digestion with HNO_3+HCl for Sn and HNO_3 for the other elements		CV-AAS for Hg, ICP-MS for the other elements
L05	no	none	microwave digestion		Cd-external calibration line ; As,Pb-standard addition
L06	As CEN/TC 327 N565;Cd,Pb MSZ EN 15550:2008;Hg CEN/TC 327 N561				
L07	AOAC				
L08	no	No sample pre-treatment	Digested with Nitric Acid and Hydrogen Peroxide (As, Cd, Hg, Pb). For Tin Nitric Acid and Hydrochloric Acid Mix used.	None used	ICP-MS Calibrated with standard solutions
L09	EN 15550, EN 14546	HNO_3, $H2O_2$	microwave	filtration	with standard solutions
L10	no	0.5g/ 3 ml HNO3 + 3 ml H2O	Micro-wave digestion	dilution to 100 ml aqua	XSII xt, CCT ED 3,5 ml/min.
L11	yes				
L12	no		FOR PB, CD, AS:HIGH PRESSURE MICROWAVE DIGESTION WITH HNO_3, H_2O_2, HF		FOR Pb, Cd, As ICP-MS: STD 1 - 50 ppb; FOR Hg DTA-AAS STD 25-5000 ppb
L13	standard method for determination of total Cd, Pb, As by ICP-MS				
L14	SR EN 13806; SR EN 14082; SR EN 14083; SR EN 14546;				
L15	no	shake the sample to rehomogenize it	microwave mineralization with nitric acid		external calibration
L16	no	H_2O_2=HNO_3	microvawe digestion		Calibration curve for Pb (10-60 ug/l); Cd (1-10 ug/l); Hg (0.5-5 ug/l); As (3-25 ug/l)
L17	Methods from the Danish Veterinary and Food Administration				
L18	no	shaking of the bottle		microwave assited extraction with HNO_3	external calibration of linear type

Lab ID	Official Method	Sample pretreatment	Digestion	Extraction/Separation	Instrument calibration
L19	no		microwave digestion with nitric acid		external standard
L20	see point 13. - microwave (HNO_3/H_2H_2); EPA-metoder (modified) 200.7 (ICP-AES) and 200.8 (ICP-SFMS)				
L21	no	Addition of nitric acid and hydrogen peroxide, let it stand for 1 hour	Microwave	Dilution	External calibration
L22	no				ICP-MS with octopole reaction system
L23	no				
L24	no		HNO_3 (DigiPREP sample digestion system at T=90°C)		ETAAS
L25	no	None	Microwave Digestion with HNO_3 + HCL +HF	None	external calibration standards
L26	EVS EN ISO 15586:2004				
L27	No - Only Arsenic was analysed using official method - EN 14546:2005	None	Open tube digestion, with Nitric Acid	None	External standards for As and Cd; Standard addition for Pb
L28	EN 13805:2002 , EN 15763:2009, EN 15765:2009				
L29	NMKL procedure nr 186; 2007				
L30	no	sample was shaken	6ml concentrated HNO_3+2ml H_2O_2, microwave heating for 20min at 200°C in sealed vessels [ramp time 20min]	dilution with deionized water, filtration	method of standard additions

European Commission

EUR 25669 – Joint Research Centre – Institute for Reference Material and Measurements

Title: IMEP-114: 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-114 of the EU-RL-HM which focused on the determination of total Cd, Pb, As, Hg and Sn in feed premixes in support to Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed. Thirty laboratories from 26 countries registered to the exercise of which 25 reported results for total As, 30 for total Cd, 29 for total Pb, 13 for total Hg and 9 for total Sn. 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. The standard deviation for proficiency assessment ($\hat{\sigma}$), also called target standard deviation, was set to 15 % of the assigned value, for the analytes investigated.

Between 60 and 90 % of the laboratories reported satisfactory results for total As and total Cd, and 50 % for total Pb. Only 3 participants reported satisfactory results for total Sn (out of 9 laboratories that reported values). Thirteen participants reported results for total Hg although, according to the expert laboratories the mass fraction for that measurand was below their limit of detection.

LA-NA-25669-EN-N

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