

# JRC Scientific and Technical Reports

## Report of the eighth interlaboratory comparison organised by the Community Reference Laboratory for Heavy Metals in Feed and Food

Total Cd, Pb, As, Hg and Sn and extractable Cd and Pb in feed of plant origin

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## **Heavy Metals in Feed and Food**

Report of the eighth interlaboratory comparison  
Total Cd, Pb, As, Hg and Sn and extractable Cd and Pb  
in feed of plant origin



January 2010

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## **1 Summary**

The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre, a Directorate General of the European Commission, operates the Community Reference Laboratory for Heavy Metals in Feed and Food (CRL-HM). One of its core tasks is to organise interlaboratory comparisons (ILCs) among appointed National Reference Laboratories (NRLs). This report presents the results of the eighth ILC of the CRL-HM which focused on the determination of total Cd, Pb, As, Hg and Sn and extractable amounts of Cd and Pb in feed of plant origin following Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed<sup>1</sup>.

The test material used in this exercise was a candidate reference material, the matrix being rye grass. The material was relabelled and dispatched to the participants on the second half of October 2009. Each participant received approximately 10 g of test material. Thirty participants from 25 member states registered to the exercise of which 27 reported results for total Cd, Pb and Hg, 23 for extractable Pb, 22 for extractable Cd and for total As and 16 for total Sn. One laboratory did not report results due to a breakdown of the instrumentation, and one laboratory reported results only for Hg for the same reason.

The assigned values ( $X_{\text{ref}}$ ) for total Cd, Pb, As, Hg and Sn were the candidate certified values as obtained during the certification. The assigned values for extractable Cd and Pb were provided by IRMM using isotope dilution-inductively coupled plasma-mass spectrometry (ID-ICP-MS).

Participants were invited to report the uncertainty of their measurements, which was provided by the majority of them. The laboratory performance was evaluated using z- and zeta-scores in accordance with ISO 13528<sup>2</sup>. The standard deviation for proficiency assessment (also called target standard deviation),  $\hat{\sigma}$ , was fixed to 15 % of the assigned values for all measurands on the basis of the outcome of previous ILCs organised by the CRL-HM for the same population.

Between 85 and 95 % of the laboratories performed to a satisfactory level taking into consideration the z-scores for total and extractable Cd and Pb. The figures are not equally positive for the same measurands when looking at the zeta-scores only. 65 to 69 % of the reported results were satisfactory when the associated uncertainties are taken into account.

## **2 Introduction**

Metals occur in varying amounts as natural ingredients of the earth's crust and they can, therefore, also be determined in variable concentrations in all natural plants and animals and consequently also in food and feed. The concentration of the various elements in plant food and feed is influenced by different factors: the genetic properties of the plant, the nature of the soil on which the plants are grown, the climate and the degree of maturity of the plant at the time of harvesting, have a considerable influence on the content of elements in the plant. Apart from genetic factors the nature of the soil has the most important influence on the content of elements in plant food and feed. Depending on the existing geological conditions of the particular region the element content of the soil can vary by several degrees of magnitude. The concentration of elements in plants is largely correlated to the corresponding concentrations in the soil on which they grow, though differences in the acid content, the humus layer, and the structure of the soil itself will also have some influence. The heavy

metal content of the soil can be affected by anthropogenic activities, such as the use of contaminated mineral fertilisers, or spreading sewage sludge contaminated with heavy metals or sediments dredged from polluted rivers. In certain polluted areas the heavy metal content can reach levels several times higher than normal, so that the plants grown there can be used neither as feed nor food according to the applicable legislation<sup>3</sup>.

To overcome problems associated with a high heavy metal content in feed, maximum levels for trace elements in several commodities have been laid down in Directive 2002/32/EC, and a network has been built up to ensure quality and comparability in official controls throughout the European Union. In March 2006 a footnote was introduced in Directive 2002/32/EC in which it is stated that "*Maximum levels refer to an analytical determination of lead and cadmium whereby extraction is performed in nitric acid 5 % (W/W) for 30 min at boiling temperature*".

In the second half of 2007 and 2008 the CRL-HM organised two proficiency tests (PTs), IMEP-103 and IMEP-105, respectively, for the network of appointed NRLs to determine total and extractable Cd and Pb in compound feed for fish (IMEP-103) and in mineral feed (IMEP-105). The outcome of IMEP-103 was that total and extractable Cd concentrations in that test material were identical. The Youden plots constructed with participant's results for total and extractable Pb, indicated no method dependence although more studies would be needed before extracting any definitive conclusion on the Pb matter due to a lack of homogeneity for Pb in the test material. The outcome of IMEP-105 lead to the same conclusion than IMEP-103 regarding total and extractable Cd, and showed that extractable Pb accounted for about 80 % of total Pb in the mineral feed tested in that exercise.

With the aim of expanding the previously mentioned studies to a wider variety of feed matrices, the CRL-HM organised in 2009 a PT for the determination of total Cd, Pb, As, Hg and Sn and extractable Cd and Pb in feed of plant origin. The two latter measurands were to be determined using the same extraction procedure than in IMEP-103 and in IMEP-105 which was agreed upon by the CRL-HM and the network of NRLs, and which is in agreement with the requirements laid down in Directive 2002/32/EC.

### **3 Scope**

As stated in Regulation (EC) No 882/2004 of the European Parliament and of the Council<sup>4</sup>, one of the core duties of the CRL-HM is to organise interlaboratory comparisons for the benefit of staff from National Reference Laboratories. The scope of this ILC is to test the competence of the appointed NRLs to determine the total concentration of Cd, Pb, As, Hg and Sn and of extractable Cd and Pb according to Directive 2002/32/EC in feed of plant origin.

The assessment of the measurement results is undertaken on the basis of requirements laid down in legislation<sup>1</sup>, and follows the administrative and logistic procedures of IMEP, the International Measurement Evaluation Program of the IRMM of the European Commission Directorate General Joint Research Centre. IMEP is accredited according to ISO Guide 43. The designation of this ILC is IMEP-108.

## **4 Time frame**

The interlaboratory comparison was agreed upon by the NRL network at the 3rd CRL-HM workshop held on 25-26 September 2008. Specific details of the exercise were refined during the 4th CRL-HM workshop held on 1-2 October 2009. Invitation letters were sent to the participants on 13<sup>th</sup> October 2009 (Annex 1). The samples were dispatched to the participants on the second half of October 2009. Reporting deadline was 30<sup>th</sup> November 2009.

## **5 Material**

### **5.1 Preparation**

The rye grass used had been harvested in 1983. The original material was processed by jet milling and made separated in 25 g portions. The finely ground powder was further processed by drying at 50 °C (to reach the water content below 4 % (m/m)), homogenising and filling into amber 60 mL glass vials with a lyo-insert and an aluminium cap under argon atmosphere. Capping and labelling was performed according to the filling sequence. Every unit contained about 10 g of the rye grass material.

The particle size distribution of the final product was assessed using a Sympatec Helos laser diffraction instrument (Clausthal Zellerfeld, DE). The maximum particle size was 175 µm. A sieve analysis demonstrated presence of larger particles of 200 µm-3 mm. In relation to the total number of particles, their relative abundance is far below 1 %.

### **5.2 Homogeneity and stability**

Homogeneity and stability studies were conducted in the frame of the certification project for the total content of heavy metals. On the basis of previous experience of the CRL-HM, extractable Cd and Pb behave the same as total Cd and Pb, respectively, in terms of homogeneity and stability.

### **5.3 Distribution**

The samples were dispatched to the participants on the second half of October 2009. Each participant received: a) one glass bottle containing approximately 10 g of the test material, b) a form that has to be sent back to the organiser after receipt of the test material to confirm its arrival (Annex 2) and c) an accompanying letter with instructions on sample handling and reporting (Annex 3).

## **6 Instructions to participants**

Details on this intercomparison were discussed with the NRLs at the fourth workshop organised by the CRL-HM, held in Geel on 1-2 October 2009. Concrete instructions were given to all participants in a letter that accompanied the test material. The measurands and matrix were defined as "*total Cd, Pb, As, Hg and Sn and extractable amounts of Cd and Pb in feed of plant origin 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 report them, together with the mean of the results and its associated uncertainty. The measurement results were to be corrected for moisture (following a procedure described in the accompanying letter which had been optimised at IRMM by the Reference Materials Unit), and for recovery. Participants were asked to follow their routine procedures. The results were to be reported in the same manner (eg. number of significant figures) as those normally reported to the customer.

The results were to be reported in a special on-line form for which each participant received an individual access code. A specific questionnaire was attached to this on-line form. The questionnaire was intended to provide further information on the measurements and the laboratories. A copy of the questionnaire is presented in Annex 5.

## **7 Reference values and their standard uncertainties**

The assigned values ( $X_{\text{ref}}$ ) for total Cd, Pb, As, Hg and Sn were the certified reference values as obtained during the certification of the of the candidate reference material that was finalised in the same period of time than IMEP-108 took place. The assigned values for extractable Cd and Pb were provided by IRMM using isotope dilution-inductively coupled plasma-mass spectrometry (ID-ICP-MS). The assigned values for all the measurands covered in this exercise are summarised in Table 1.

The uncertainty in the candidate certified values ( $u_{\text{ref}}$ ) for the total content of Cd, Pb, As, Hg and Sn, is calculated according to the principles described in ISO Guide 35. For extractable Pb the  $u_{\text{ref}}$  indicated in the certificate for total Pb was taken because the total and the extractable amounts of Pb in the test material were found to be identical.

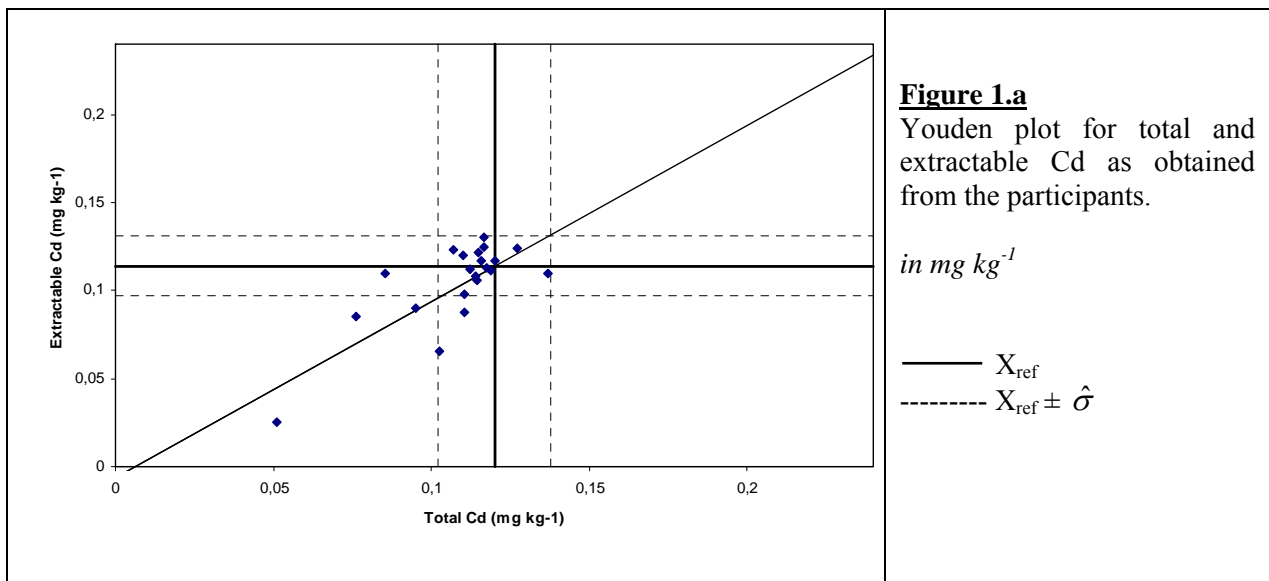
The analytical uncertainty of  $X_{\text{ref}}$  ( $u_{\text{char}}$ ) for extractable Cd was calculated according to the ISO Guide to the Expression of Uncertainty in Measurement (GUM)<sup>5</sup>. The uncertainty of the assigned value for extractable Cd was calculated combining the analytical uncertainty ( $u_{\text{char}}$ ) with a contribution for the between-bottle homogeneity ( $u_{\text{bb}}$ ) as provided by the CRM producer. No contribution for the short-term stability of the materials was added because the material proved to be stable for the five weeks that elapsed between the dispatch of the test material and the deadline for submission of results.

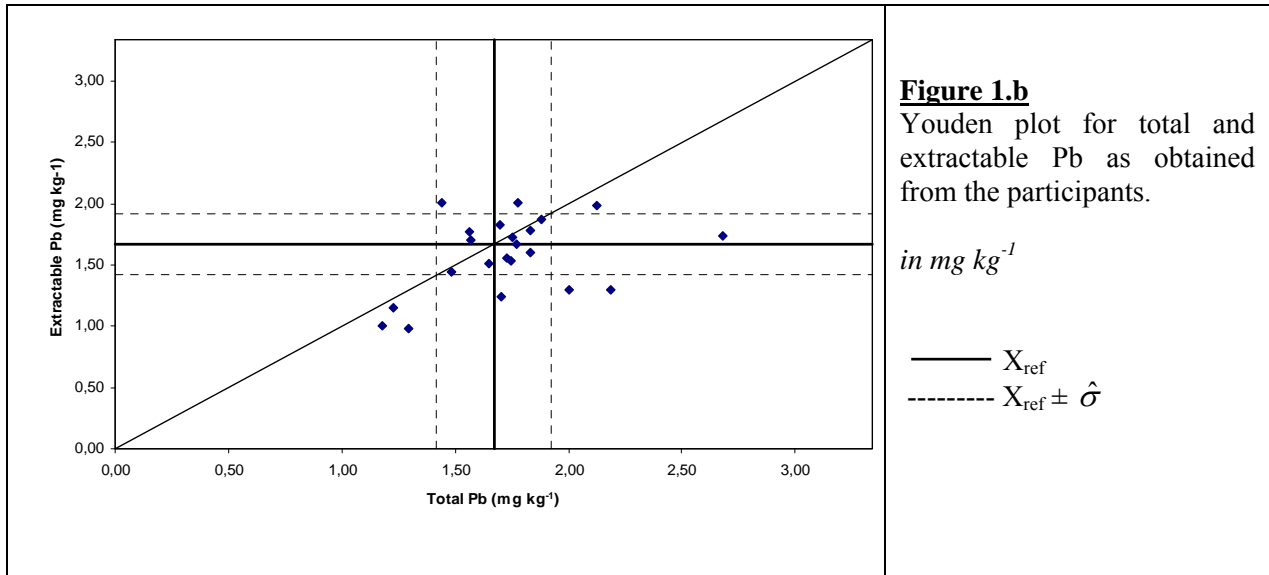
**Table 1:** Assigned values and their associated standard and associated uncertainties for the measurands of this ILC.

Measurand	$X_{ref}$ (mg kg <sup>-1</sup> )	$U_{ref}$ (mg kg <sup>-1</sup> )
Total Cd	0.120	0.007
Extractable Cd	0.114	0.003
Total Pb	1.67	0.11
Extractable Pb	1.67	0.11
Total As	0.042	0.010
Total Hg	0.0164	0.0022
Total Sn	0.062	0.011

$X_{ref}$  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 %.

As summarised in Table 1, total digestion and partial extraction of the test material, following the procedure described in the accompanying letter to the participants, provided identical Pb concentrations. In the case of Cd, the reference value obtained by IRMM for extractable Cd is about 94 % of the total concentration. These findings are supported by the Youden plots, Fig. 1.a and b, constructed with the results provided by the participants in this exercise for Cd and Pb, respectively. One cloud of points is observed on both axes around the reference value and within the square formed by the reference values  $\pm \hat{\sigma}$ , when total vs the extractable contents are plotted.





## 8 Evaluation of results

### 8.1 General observations

Thirty participants from 25 member states registered to the exercise of which 27 reported results for total Cd, Pb and Hg (2, 1 and 5 out of the 27 reported "less than" for total Cd, total Pb and total Hg, respectively), 23 for extractable Pb, 22 for extractable Cd and for total As (3 out of the 22 reported "less than" for total As) and 16 for total Sn (5 out of the 16 reported "less than"). One laboratory did not report results due to a break down of the instrument that was to be used for the analysis, and one laboratory reported results only for Hg for the same reason. All laboratories responded to the questionnaire included in the on-line reporting form.

### 8.2 Scores and evaluation criteria

Individual laboratory performance is expressed in terms of z and zeta scores in accordance with ISO 13528<sup>2</sup> and the International Harmonised Protocol<sup>6</sup>.

$$z = \frac{x_{lab} - X_{ref}}{\sigma} \quad \text{Eq. 1}$$

$$zeta = \frac{x_{lab} - X_{ref}}{\sqrt{u_{ref}^2 + u_{lab}^2}} \quad \text{Eq. 2}$$

where:

$x_{lab}$  is the mean measurement result reported by a participant calculated by the ILC organiser

$X_{ref}$  is the certified reference value (assigned value)

$u_{ref}$  is the standard uncertainty of the reference value

$u_{\text{lab}}$  is the standard uncertainty reported by a participant

$\hat{\sigma}$  is the standard deviation for proficiency assessment

The z score compares the participant's deviation from the reference value with the standard deviation accepted for the proficiency test,  $\hat{\sigma}$ . For z-scores, the standard deviation  $\hat{\sigma}$  is used as common quality criterion. Metrologically speaking,  $\hat{\sigma}$  is the maximum acceptable standard uncertainty as defined by the organiser of the proficiency test. This uncertainty consists of two parts, namely the acceptable laboratory uncertainty and potential contributions for homogeneity and stability. The Harmonised Protocol<sup>6</sup> states that the method for homogeneity testing must be repeatable enough to render the homogeneity contribution negligible compared to the measurement uncertainty of the laboratory. Based on feedback from experts on the state-of-the-art and on discussions amongst the PT organisers, values for  $\hat{\sigma}$  were set as 15 % of the assigned value for all measurands.

Should participants feel that these  $\hat{\sigma}$  values are not fit for their purpose they can recalculate their scorings with a standard deviation matching their requirements.

If  $\hat{\sigma}$  is regarded as satisfactory, the z-score can be interpreted as:

$ z  \leq 2$	satisfactory result
$2 <  z  \leq 3$	questionable result
$ z  > 3$	unsatisfactory result

The zeta score states if the laboratory result agrees with the assigned value within the respective uncertainties. The numerator of equation 2 describes the combined uncertainty of the assigned value and the measurement uncertainty as stated by the laboratory. The zeta-score is therefore the most relevant evaluation parameter, as it includes all parts of a measurement result, namely the expected value, its uncertainty and the unit of the result as well as the uncertainty of the assigned values. If a result receives a zeta-score  $>2$  then the result is wrong at a 95 %, if the zeta-score is larger then 3, then the result is wrong on a 99.4 % confidence level. This wrong result can either be caused by a wrong estimation of the expected value (the value before the " $\pm$ ") or its uncertainty.

Accordingly, the interpretation of the zeta score is similar to the interpretation of the z-score:

$ \text{zeta}  \leq 2$	satisfactory result
$2 <  \text{zeta}  \leq 3$	questionable result
$ \text{zeta}  > 3$	unsatisfactory result

It is a well-established fact that uncertainty estimation is not trivial. Therefore an additional assessment was given as an indication of the plausibility of its uncertainty estimate for each laboratory providing an uncertainty estimate: The standard uncertainty is most likely to fall in a range between a minimum uncertainty ( $u_{\text{min}}$ ), and a maximum allowed ( $u_{\text{max}}$ ).  $u_{\text{min}}$  is set to the standard uncertainty of the reference value. It is unlikely (but not impossible) that a laboratory carrying out the analysis on a routine basis could measure the measurand with a smaller uncertainty than the reference laboratory itself.  $u_{\text{max}}$  is set to the standard deviation accepted for the PT,  $\hat{\sigma}$ . If the standard uncertainty from the laboratory,  $u_{\text{lab}}$ , is smaller than  $u_{\text{min}}$ , the laboratory probably underestimated its uncertainty. However, this statement has to taken with care as each laboratory will report only the uncertainty of its measurement, whereas the uncertainty of the reference value also includes contributions of homogeneity and stability. If those are large, measurement uncertainties smaller than  $u_{\text{min}}$  are possible and also plausible. If  $u_{\text{lab}} > u_{\text{max}}$ , the laboratory possibly overestimated the uncertainty. An evaluation of this statement can be made when looking at the comparison of the reported value and the

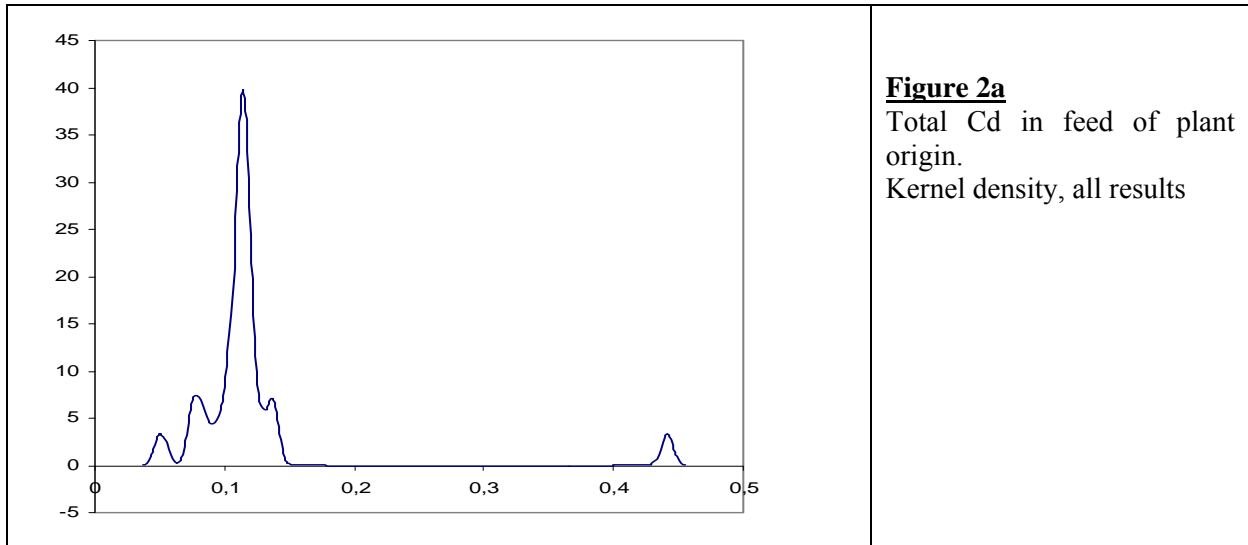
assigned value: if the difference is small but the uncertainty is large, then overestimation is likely. If, however, the deviation is large but is covered by the uncertainty, then the uncertainty was assessed correctly but is large. Naturally, this assessment will gain from pooling data from various intercomparisons. It should be pointed out that  $u_{\max}$  is not a normative criterion: Which uncertainty is acceptable for a certain measurement is the decision of the customer of the respective result.

The standard uncertainty of the laboratory,  $u_{\text{lab}}$ , was estimated by dividing the reported expanded uncertainty by the reported coverage factor,  $k$ . When no uncertainty was reported, it was set to zero ( $u_{\text{lab}} = 0$ ). When  $k$  was not specified, the reported expanded uncertainty was considered as the half-width of a rectangular distribution;  $u_{\text{lab}}$  was then calculated by dividing this half-width by  $\sqrt{3}$ , as recommended by Eurachem and CITAC<sup>7</sup>.

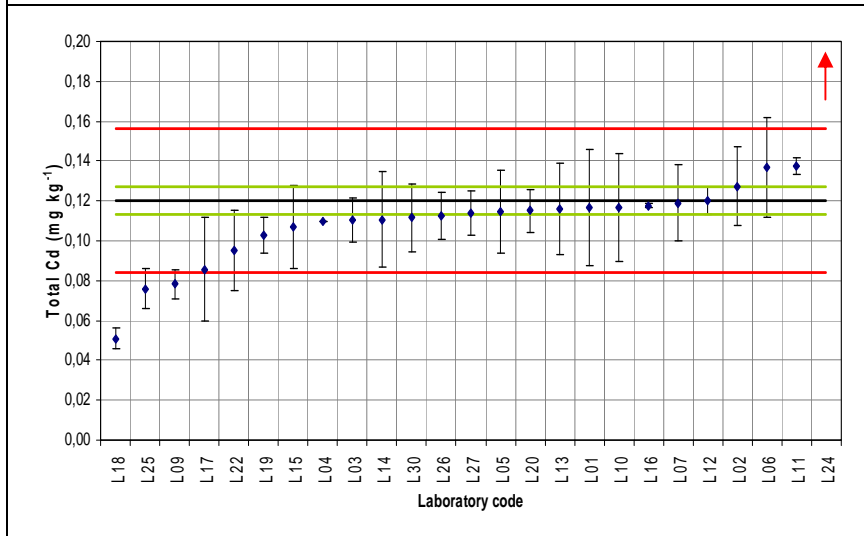
### **8.3 Laboratory results and scorings**

The results as reported by the participants are summarised in Table 2 a-g for total Cd, total Pb, total As, total Hg and total Sn and for extractable Cd and Pb, respectively, together with the z- and zeta scores. Laboratory codes were given randomly.

Three sets of figures are provided for total Cd, Pb, As, Hg and Sn and for extractable Cd and Pb (Figure 2-8). Each set includes (a) the Kernel density plot, (b) the individual mean value and associated expanded uncertainty, (c) the z- and zeta scores. The black line represents the assigned value, the green lines delimit the reference interval ( $X_{\text{ref}} \pm 2 u_{\text{ref}}$ ) and the red lines delimit the target interval ( $X_{\text{ref}} \pm 2 \sigma$ ). The Kernel plots were obtained using a software tool developed by AMC<sup>8</sup>.

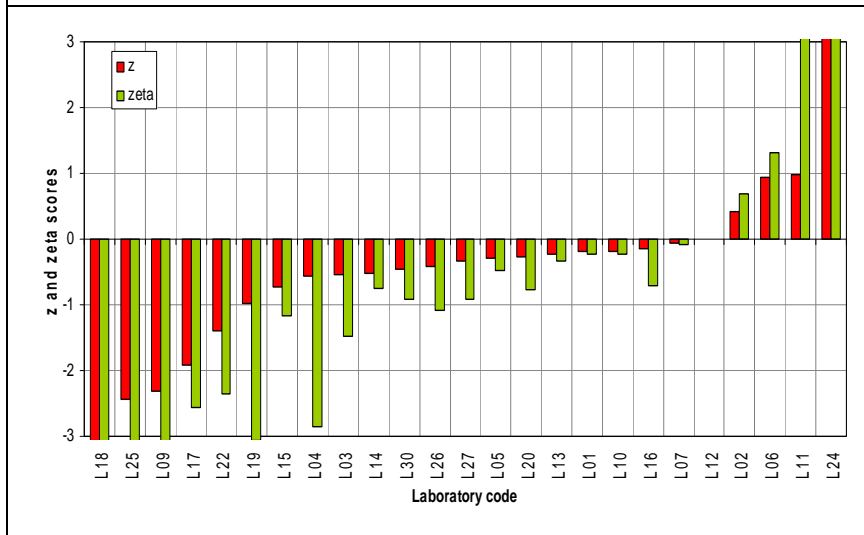


**Figure 2a**  
Total Cd in feed of plant origin.  
Kernel density, all results

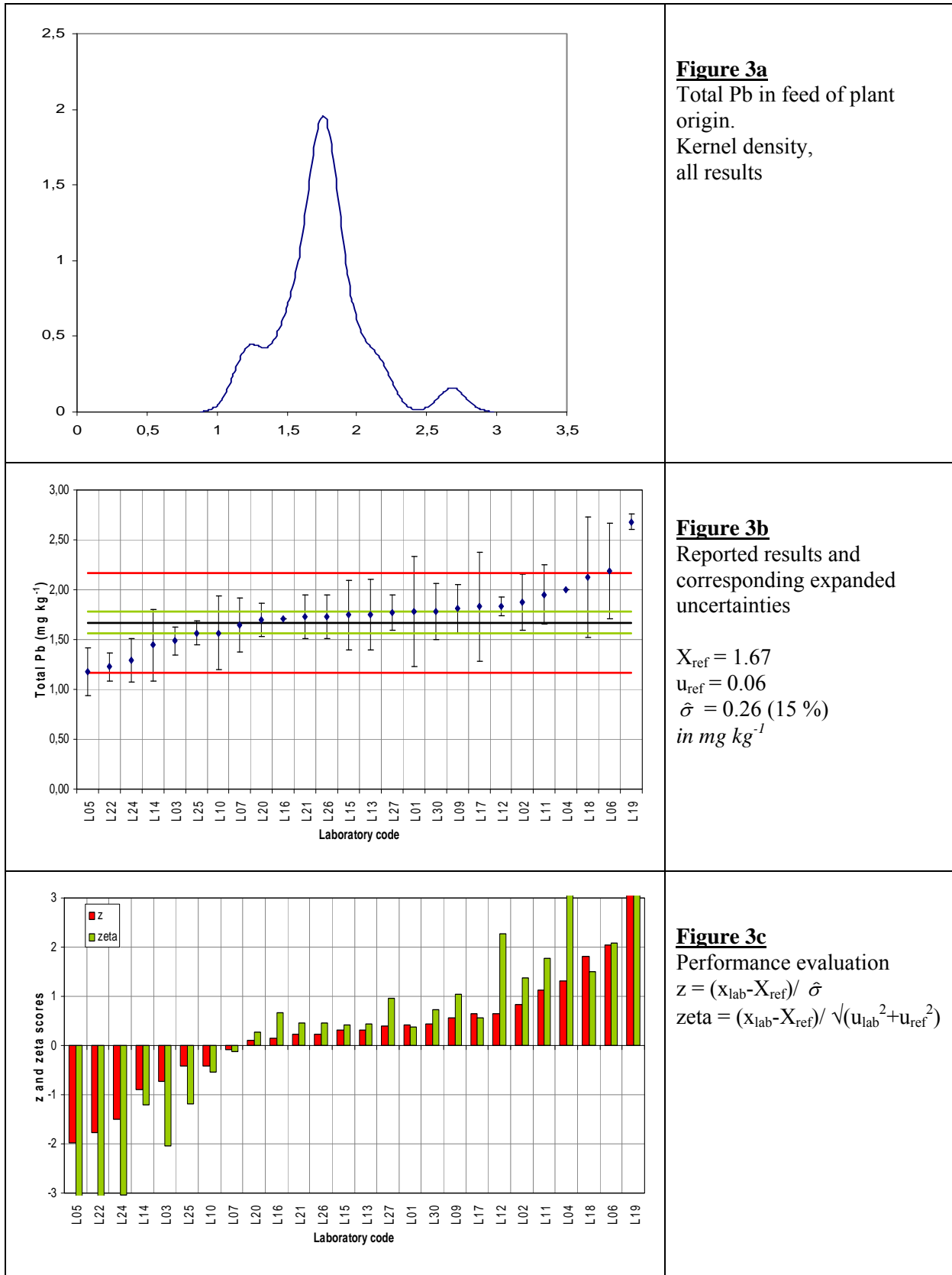


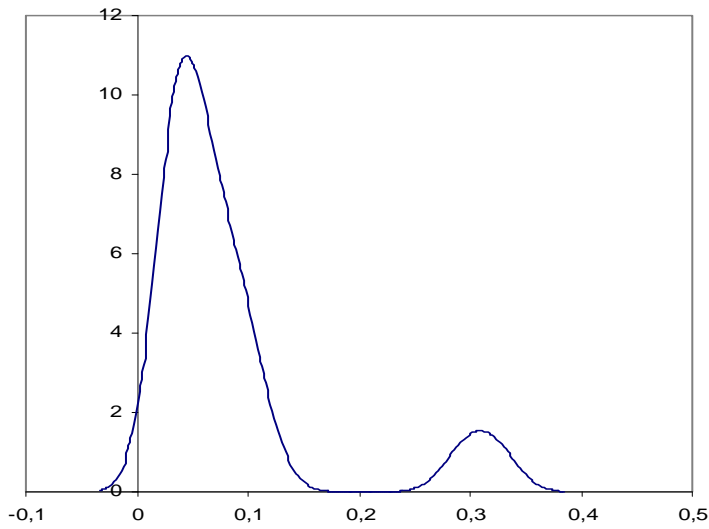
**Figure 2b**  
Reported results and corresponding expanded uncertainties

$X_{ref} = 0.120$   
 $u_{ref} = 0.004$   
 $\hat{\sigma} = 0.018$  (15 %)  
in  $mg\ kg^{-1}$

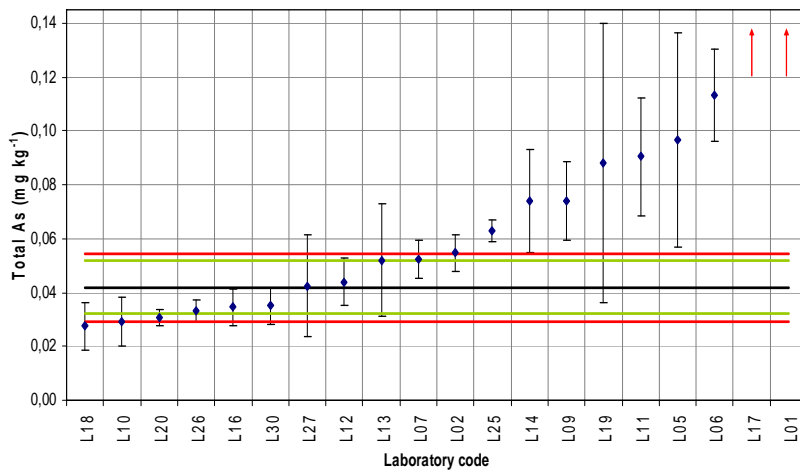


**Figure 2c**  
Performance evaluation  
 $z = (x_{lab} - X_{ref}) / \hat{\sigma}$   
 $zeta = (x_{lab} - X_{ref}) / \sqrt{(u_{lab}^2 + u_{ref}^2)}$



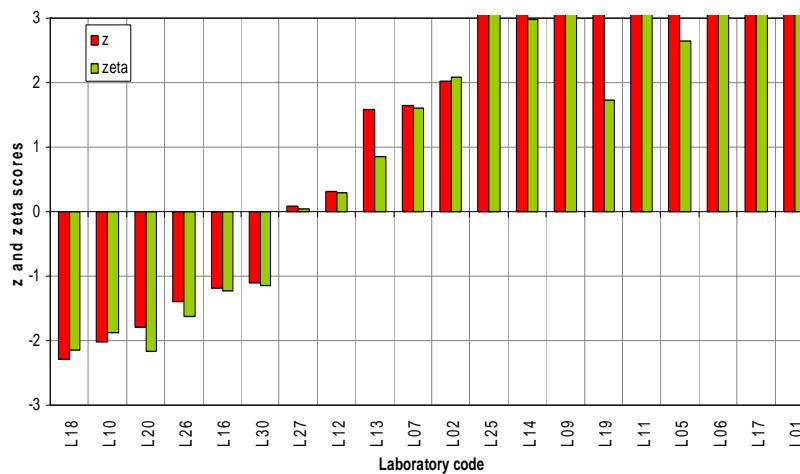


**Figure 4a**  
Total As in feed of plant origin.  
Kernel density,  
all results



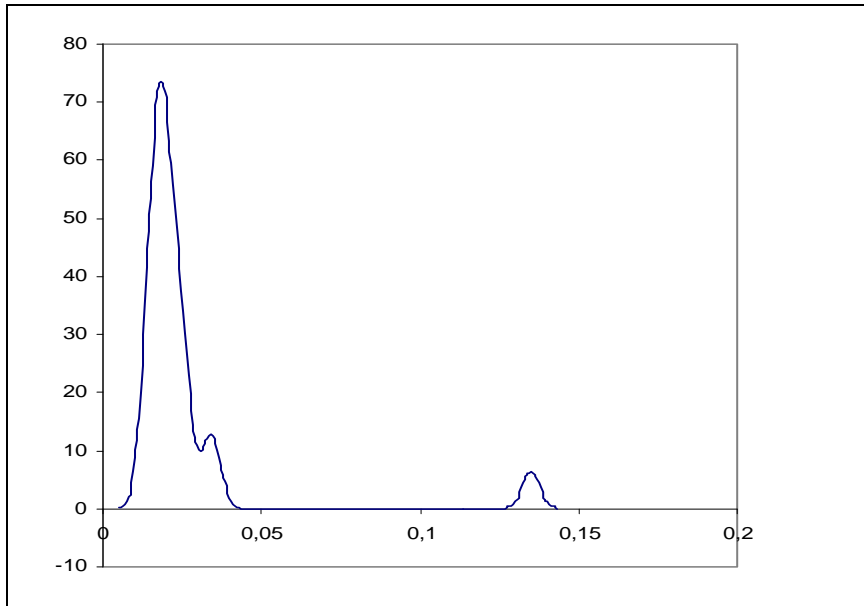
**Figure 4b**  
Reported results and  
corresponding expanded  
uncertainties

$X_{ref} = 0.042$   
 $u_{ref} = 0.005$   
 $\hat{\sigma} = 0.006$  (15 %)  
in  $mg\ kg^{-1}$

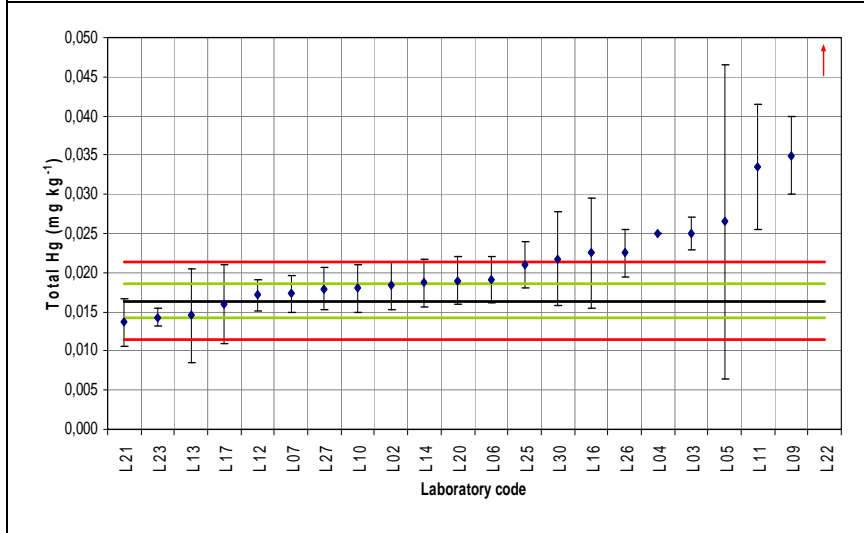


**Figure 4c**  
Performance evaluation  
 $z = (x_{lab} - X_{ref}) / \hat{\sigma}$   
 $zeta = (x_{lab} - X_{ref}) / \sqrt{(u_{lab}^2 + u_{ref}^2)}$



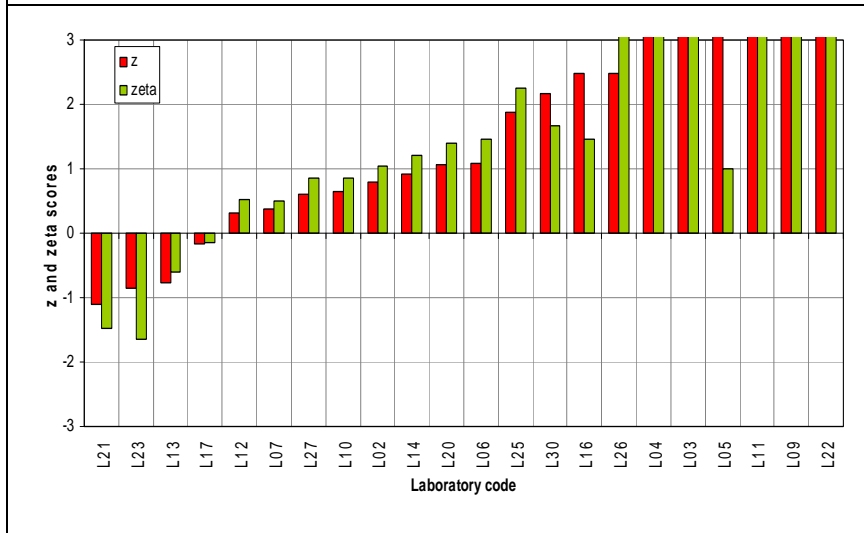


**Figure 5a**  
Total Hg in feed of plant origin.  
Kernel density,  
all results

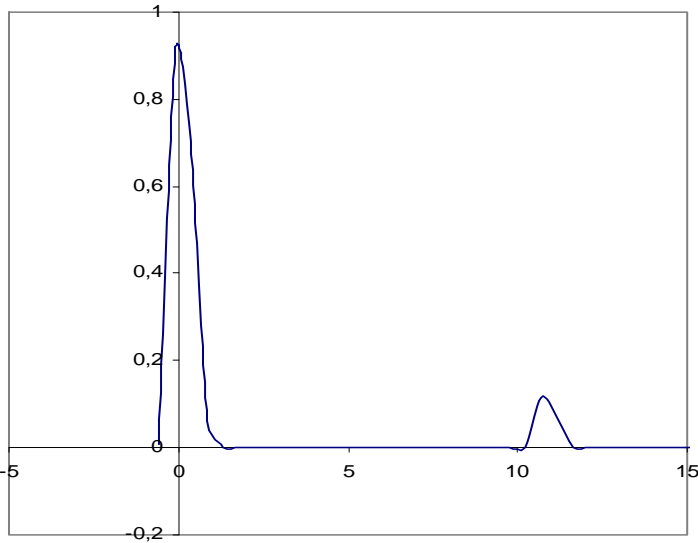


**Figure 5b**  
Reported results and  
corresponding expanded  
uncertainties

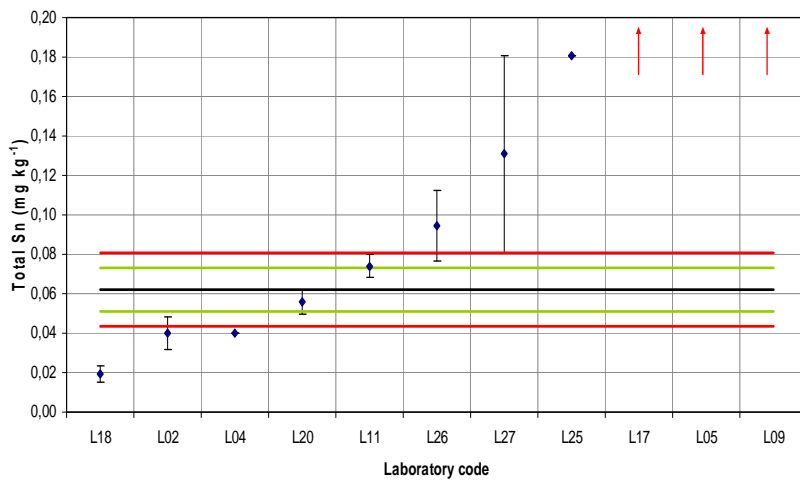
$X_{ref} = 0.0164$   
 $u_{ref} = 0.0011$   
 $\hat{\sigma} = 0.0025$  (15 %)  
in  $mg\ kg^{-1}$



**Figure 5c**  
Performance evaluation  
 $z = (x_{lab} - X_{ref}) / \hat{\sigma}$   
 $zeta = (x_{lab} - X_{ref}) / \sqrt{(u_{lab}^2 + u_{ref}^2)}$

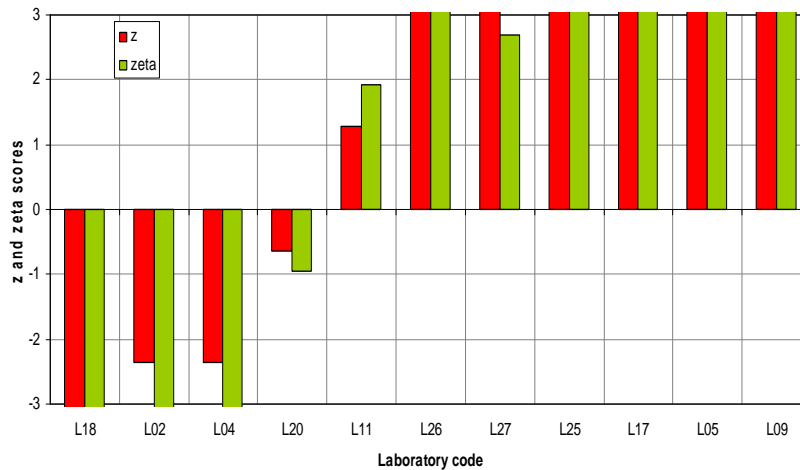


**Figure 6a**  
Total Sn in feed of plant origin.  
Kernel density,  
all results

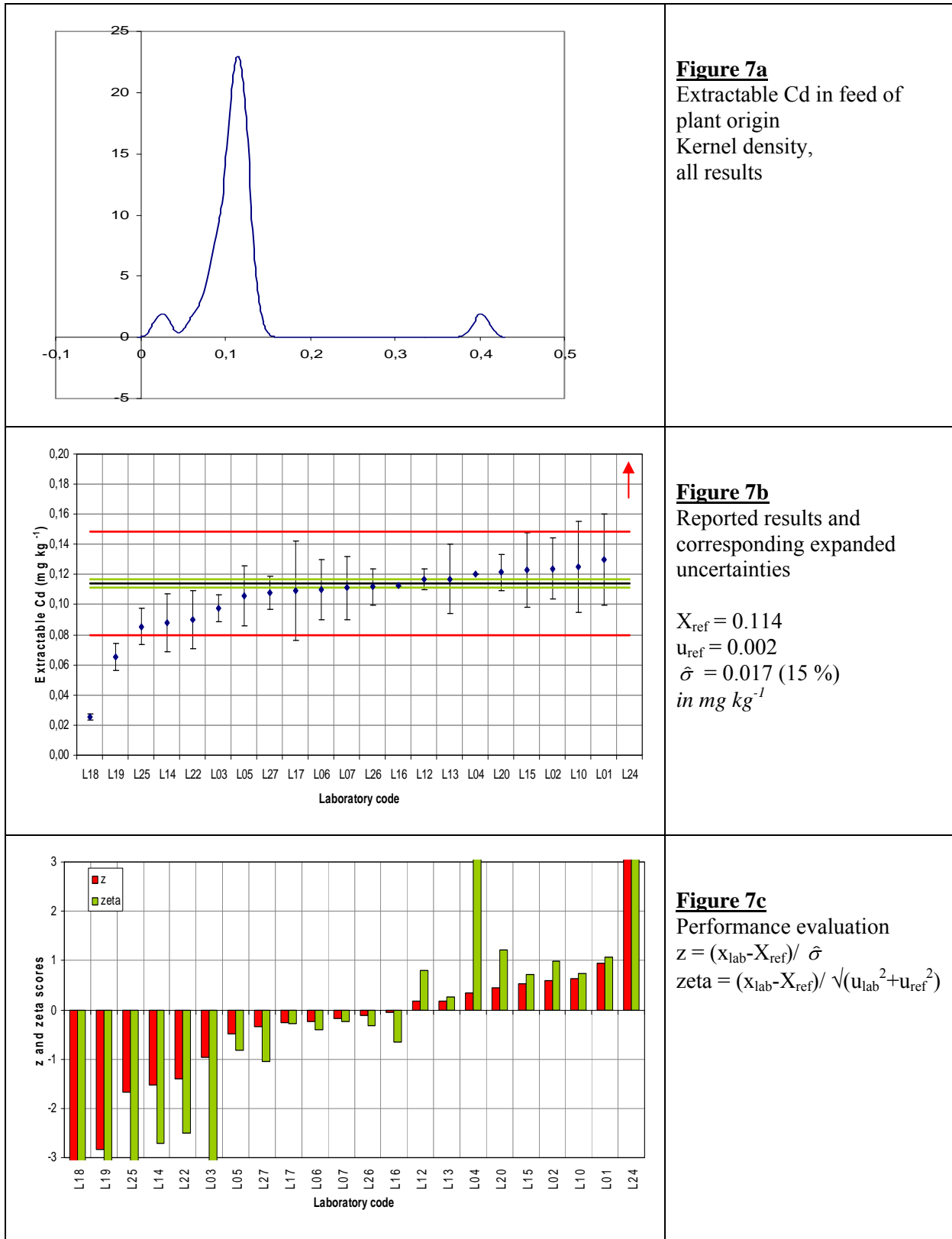


**Figure 6b**  
Reported results and  
corresponding expanded  
uncertainties

$X_{ref} = 0.062$   
 $u_{ref} = 0.006$   
 $\hat{\sigma} = 0.009$  (15 %)  
in  $mg\ kg^{-1}$



**Figure 6c**  
Performance evaluation  
 $z = (x_{lab} - X_{ref}) / \hat{\sigma}$   
 $zeta = (x_{lab} - X_{ref}) / \sqrt{(u_{lab}^2 + u_{ref}^2)}$

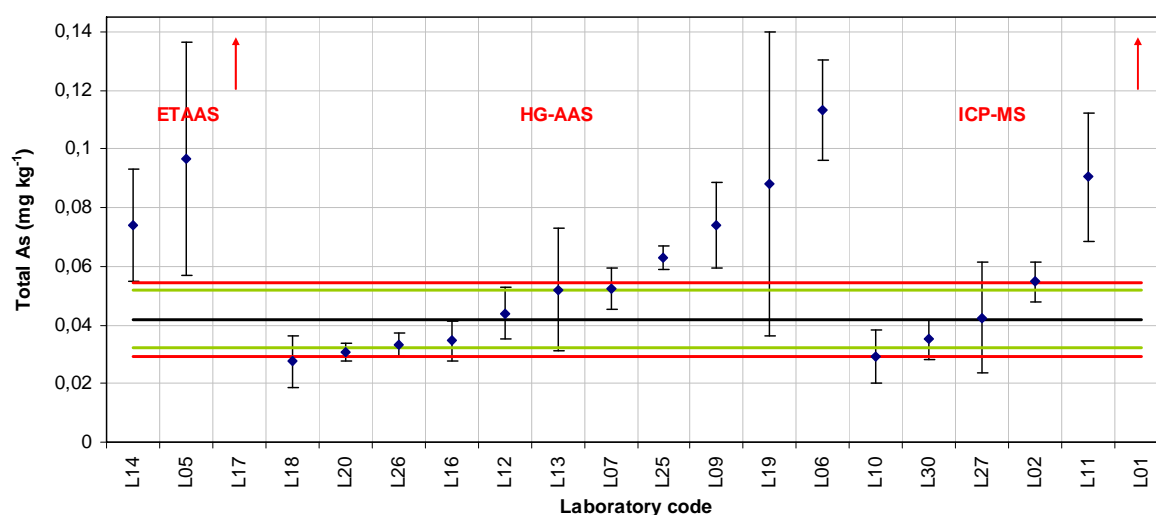


	<p><b>Figure 8a</b> Extractable Pb in feed of plant origin. Kernel density, all results</p>
	<p><b>Figure 8b</b> Reported results and corresponding expanded uncertainties</p> <p><math>X_{ref} = 1.67</math> <math>u_{ref} = 0.06</math> <math>\hat{\sigma} = 0.25</math> (15 %) in <math>mg\ kg^{-1}</math></p>
	<p><b>Figure 8c</b> Performance evaluation</p> <p><math>z = (x_{lab} - X_{ref}) / \hat{\sigma}</math> <math>zeta = (x_{lab} - X_{ref}) / \sqrt{(u_{lab}^2 + u_{ref}^2)}</math></p>

Regarding the z and zeta scores, the results for total Cd, Pb, As, Hg and Sn are summarised in Table 3. Between 85 and 95 % of the laboratories performed satisfactory taking into

consideration the z-scores for total and extractable Cd and Pb. However, when looking at the zeta-scores only, 65 to 69 % of the reported results were satisfactory when the associated uncertainties are taken into account.

In previous exercises organised by the CRL-HM for the same population, a high share of satisfactory z and zeta-scores were obtained for total As. That has not been the case in IMEP-108, with only 50 % of the laboratories obtaining a satisfactory z-score, and 45 % a satisfactory zeta-score, the concentration of total As reported frequently being too high. It is well known that when analysing As by ICP-MS,  $^{40}\text{Ar}^{35}\text{Cl}^+$ , which has the same mass as As, can interfere. However, after comparing the results reported by laboratories that use ICP-MS with those obtained by hydride generation-atomic absorption spectrometry (HG-AAS) no significant difference was observed, Figure 9. A contamination problem mainly coming from the reagents used for the analysis could be at the source of the overestimated As concentrations. Such a contamination problem would have more impact on the results reported for total As in IMEP-108 than in the previous ILC's organised by the CRL-HM, such as IMEP-104, -105 and -106, due to the comparatively low concentration of As in the test material used in IMEP-108.



**Figure 9:** Comparison of the results reported for total As according to the technique used for the analysis.

A positive bias was also observed in some of the results reported for total Hg, which resulted in 27 % of the laboratories obtaining unsatisfactory z and zeta-scores. This problem was already identified in IMEP-106 (heavy metals in food supplements). As indicated, the positive bias is probably due to a contamination problem, to the presence of interferences or to problems in calibration at such low concentrations.

The network of NRLs requested during the 4<sup>th</sup> workshop organised by the CRL-HM, to include tin as measurand in future ILC's. IMEP-108 is the first ILC organised by the CRL-HM in which participants are invited to report values for total Sn. Eleven participants reported values for this measurand and only two out of them obtained satisfactory z and zeta-scores. The concentration of Sn in the test material was low,  $0.062 \text{ mg kg}^{-1}$ , when compared to the maximum allowed levels given in the legislation for food<sup>9</sup> ( $50\text{-}200 \text{ mg kg}^{-1}$  depending on the food commodity). Sn is not a contaminant for which maximum levels have been set in the European legislation for contaminants in feed<sup>1</sup>. Unfortunately, it is not always possible to find

a test material with appropriate concentrations for all the measurands covered in an ILC and the CRL-HM rather prefers to avoid the option of using spiked test materials in a proficiency test to avoid artificial high recoveries which do not correspond to the actual recoveries on non-spiked samples.

Additional information was gathered from the questionnaire that participants were asked to fill in (Annex 4). Nine participants corrected their results for recovery by one of the following options or a combination of them: seven used a certified reference material and five added a known amount of the same analyte to the sample. Different justifications were given by those that did not correct for recovery: five indicated that the recovery as calculated with a CRM was close to 100 %, one said that they do not have a CRM and blank matrix for organic feed, one said that they never do, one indicated that they do not do it on a routine basis because they evaluate the recovery during the validation of the method, and one indicated that the recovery of the determination for all elements was in the range covered by the uncertainty.

For uncertainty estimates, various combinations of one or more options (question 3 of the questionnaire shown in Annex 4) were given. Eight laboratories calculated the uncertainty according to ISO-GUM. Seventeen use the uncertainty as calculated during the in-house validation of the method. Eleven laboratories use the uncertainty obtained by measuring replicates (i.e. precision). Four laboratories made use of intercomparison data. Two laboratories used the expert guesstimate (which corresponds to "estimation based on judgement", as defined in the Eurachem/CITAC guide on Quantifying Uncertainty in Analytical Measurements<sup>7</sup>). One participant calculates the uncertainty according to ISO 5725. Sixteen participants report uncertainty to their customers although one of the laboratories that declared to do so did not report any uncertainty to IMEP-108.

All participants but two have corrected their results for the moisture content. Out of the two that did not correct for the moisture content, one said that they have dried the sample before analysing it and the other indicated that the sample was not sufficient for the moisture determination. The moisture contents reported varied in the range 2.6-6.9 %. The way in which the moisture content of the test material was to be calculated was described in detail in the accompanying letter (Annex 3).

Thirteen laboratories analysed the test material following an official method. One participant did not answer to this question. The information reported by the remaining laboratories about their method of analysis is summarised in Annex 5.

Twenty-four participants carry out this type of analysis (as regards the measurands, matrix and methods) on a routine basis, three do not and two did not answer.

All participants but one have a quality system in place based on ISO 17025, two based on ISO 17025 and ISO 9000 series. One laboratory did not answer.

Twenty-five participants use on a routine basis total digestion and three partial digestion according to legislation<sup>1</sup> (none of the NRLs that took part in IMEP-103, which took place in 2008, declared to use partial extraction in routine analysis). One laboratory did not reply to this question. Twenty-four laboratories are accredited for the type of sample treatment that they use on a routine basis, four are not.

Twenty-five laboratories take part in ILCs for this type of analysis and three do not.

Twenty-five participants use a reference material for this type of analysis, twenty-one use it for the validation procedure and nine for calibration purposes.

The comments made by the participants are summarised in Table 4. Several participants indicated that not enough test material had been distributed. It is frequently said that the outcome of proficiency tests does not reflect the real situation in analytical laboratories because participants do not treat PT test materials as they would treat normal samples. For instance participants in a PT would perform more replicates than in routine analysis. In order to check if that pre-assumption is correct, the CRL-HM carried out an experiment with IMEP-108. Namely, without informing participants, the minimum amount of test material strictly needed to perform the requested analysis plus the moisture determination, was distributed to the participating laboratories. Participants had to carefully plan the analysis to be performed in order to have enough test material. Two participants requested more test material and they received some extra bottles. No significant difference in the overall performance of the laboratories in IMEP-108 and in the previous exercises organised by the CRL-HM was observed other than the poor performance in total As determination, which as previously described, is most likely related to a contamination problem at low concentrations. It is then possible to conclude that the PT's organised by the CRL-HM reflect the real state-of-the-art regarding the performance of the network of NRL's.

No influence of the techniques used (mainly ZETAAS, HG-AAS and ICP-MS) was detected for any of the measurands covered in the ILC.

**Table 2a:** Total Cd, quantitative information reported by the participants plus the laboratory scorings provided by the organiser.

**Total Cd content:  $0.120 \pm 0.007 \text{ mg kg}^{-1}$**

Lab code	x1	x2	x3	x4	U <sub>lab</sub>	k	Mean-calc	Technique	z	zeta
L01	0,12	0,12	0,11		0,029	2	0,12	ICP-MS	-0,2	-0,2
L02	0,129	0,126	0,127		0,02	2	0,127	ICP-MS	0,4	0,7
L03	0,110	0,112	0,109		0,011	2	0,110	AAS	-0,5	-1,5
L04	0,11				0	√3	0,11	ETAAS	-0,6	-2,9
L05	0,1207	0,1109	0,1123		0,021	2	0,115	ETAAS-Z	-0,3	-0,5
L06	0,136	0,137	0,138		0,025	2	0,137	ETAAS	0,9	1,3
L07	0,127	0,121	0,109		0,019	√3	0,119	ETAAS	-0,1	-0,1
L08	<0,25	<0,25					<0,25	ZETAAS		
L09	0,0781	0,0820	0,0747		0,0071	2	0,0783	ETAAS	-2,3	-8,4
L10	0,12	0,12	0,11		0,027	2	0,12	ICP-MS	-0,2	-0,2
L11	0,147	0,128			0,004	2	0,138	ICP-MS	1,0	4,3
L12	0,126	0,114	0,120		0,007	2	0,120	ICP-MS	0,0	0,0
L13	0,119	0,113			0,023	2	0,116	ETAAS	-0,2	-0,3
L14	0,111	0,108	0,113		0,024	2	0,111	ETAAS	-0,5	-0,7
L15	0,098	0,109	0,114		0,021	2	0,107	ICP-AES	-0,7	-1,2
L16	0,117	0,118			0,001	2	0,118	ETAAS	-0,1	-0,7
L17	0,084	0,087			0,026	2	0,086	ETAAS	-1,9	-2,6
L18	0,049	0,053			0,005	2	0,051	ETAAS	-3,8	-16,0
L19	0,111	0,094			0,009	2	0,103	ETAAS	-1,0	-3,1
L20	0,110	0,114	0,121		0,011	2	0,115	ICP-MS	-0,3	-0,8
L21	<0,232	<0,232	<0,232				<0,232	FAAS		
L22	0,092	0,100	0,093		0,020	2	0,095	ETAAS	-1,4	-2,4
L24	0,442	0,441	0,442		0,111	2	0,442	ETAAS	17,9	5,8
L25	0,091	0,061			0,010	√3	0,076	ICP-AES	-2,4	-6,5
L26	0,113	0,110	0,114	0,113	0,012	2	0,113	ETAAS	-0,4	-1,1



*CRL-HM in Feed and Food. Total Cd, Pb, As, Hg and Sn and extractable Cd and Pb in feed of plant origin*

<b>Lab code</b>	<b>x1</b>	<b>x2</b>	<b>x3</b>	<b>x4</b>	<b>U<sub>lab</sub></b>	<b>k</b>	<b>Mean-calc</b>	<b>Technique</b>	<b>z</b>	<b>zeta</b>
L27	0,111	0,117			0,011	2	0,114	ICP-MS	-0,3	-0,9
L30	0,107	0,119	0,109		0,017	2	0,112	ETAAS	-0,5	-0,9

*All results in mg kg<sup>-1</sup>*

**Table 2b:** Total Pb, quantitative information reported by the participants plus the laboratory scorings provided by the organiser.

**Total Pb content:  $1.67 \pm 0.11 \text{ mg kg}^{-1}$**

Lab code	x1	x2	x3	x4	Ulab	k	Mean-calc	Technique	z	zeta
L01	1,79	1,79	1,75		0,552	2	1,78	ICP-MS	0,4	0,4
L02	1,887	1,886	1,872		0,282	2	1,878	ICP-MS	0,8	1,4
L03	1,479	1,496	1,485		0,141	2	1,487	AAS	-0,7	-2,1
L04	2,0				0	$\sqrt{3}$	2,0	ETAAS	1,3	6,0
L05	1,17	1,12	1,24		0,24	2	1,18	ETAAS-Z	-2,0	-3,7
L06	2,184	2,184	2,182		0,48	2	2,183	ETAAS	2,0	2,1
L07	1,60	1,55	1,80		0,27	$\sqrt{3}$	1,65	ETAAS	-0,1	-0,1
L08	<1,80	<1,80	<1,80				<1,80	ZETAAS		
L09	1,794	1,821	1,810		0,243	2	1,808	ETAAS	0,6	1,0
L10	1,6	1,5	1,6		0,37	2	1,6	ICP-MS	-0,4	-0,5
L11	1,96	1,94			0,295	2	1,95	ICP-MS	1,1	1,8
L12	1,840	1,832	1,826		0,092	2	1,833	ICP-MS	0,6	2,3
L13	1,78	1,72			0,35	2	1,75	ETAAS	0,3	0,4
L14	1,45	1,44	1,44		0,36	2	1,44	ETAAS	-0,9	-1,2
L15	1,63	1,74	1,87		0,35	2	1,75	ICP-AES	0,3	0,4
L16	1,740	1,673			0,001	2	1,707	ETAAS	0,1	0,7
L17	1,79	1,87			0,55	2	1,83	ETAAS	0,6	0,6
L18	2,14	2,11			0,6	2	2,125	ETAAS	1,8	1,5
L19	2,697	2,634	2,710		0,081	2	2,680	ETAAS	4,0	14,8
L20	1,72	1,71	1,66		0,17	2	1,70	ICP-MS	0,1	0,3
L21	1,736	1,686	1,759		0,217	2	1,727	FAAS	0,2	0,5
L22	1,19	1,14	1,35		0,14	2	1,23	ETAAS	-1,8	-5,0
L24	1,293	1,270	1,314	1,299	0,221	2	1,294	ETAAS	-1,5	-3,0
L25	1,533	1,596			0,120	$\sqrt{3}$	1,565	ICP-AES	-0,4	-1,2
L26	1,68	1,74	1,71	1,78	0,22	2	1,73	ETAAS	0,2	0,5
L27	1,76	1,78			0,18	2	1,77	ICP-MS	0,4	0,9

*CRL-HM in Feed and Food. Total Cd, Pb, As, Hg and Sn and extractable Cd and Pb in feed of plant origin*

Lab code	x1	x2	x3	x4	Ulab	k	Mean-calc	Technique	z	zeta
L30	1,771	1,782	1,787		0,28	2	1,78	ETAAS	0,4	0.7
<i>All results in mg kg<sup>-1</sup></i>		Satisfactory			Questionable		Unsatisfactory			

**Table 2c:** Total As, quantitative information reported by the participants plus the laboratory scorings provided by the organiser.

**Total As content:  $0.042 \pm 0.010 \text{ mg kg}^{-1}$**

Lab code	x1	x2	x3	x4	Ulab	k	Mean-calc	Technique	z	zeta
L01	0,29	0,30	0,38		0,074	2	0,323	ICP-MS	44,7	7,5
L02	0,053	0,056	0,055		0,007	2	0,055	ICP-MS	2,0	2,1
L04	<0,1						<0,1	ETAAS		
L05	0,116	0,098	0,076		0,04	2	0,10	ETAAS-Z	8,7	2,7
L06	0,121	0,109	0,110		0,017	2	0,113	HG-AAS	11,3	7,2
L07	0,056	0,048	0,053		0,007	$\sqrt{3}$	0,052	HG-AAS	1,6	1,6
L08	<0,85	<0,85	<0,85				<0,85	ZETAAS		
L09	0,0749	0,0739	0,0733		0,0148	2	0,0740	HG-AAS	5,1	3,6
L10	0,033	0,027	0,028		0,009	2	0,029	ICP-MS	-2,0	-1,9
L11	0,099	0,082			0,022	2	0,091	ICP-MS	7,7	4,0
L12	0,043	0,044	0,045		0,009	2	0,044	HG-AAS	0,3	0,3
L13	0,055	0,049			0,021	2	0,052	HG-AAS	1,6	0,9
L14	0,070	0,077	0,075		0,019	2	0,074	ETAAS	5,1	3,0
L16	0,033	0,036			0,007	2	0,035	HG-AAS	-1,2	-1,2
L17	0,29	0,3			0,12	2	0,30	ETAAS	40,2	4,2
L18	0,027	0,028			0,009	2	0,0275	HG-AAS	-2,3	-2,2
L19	0,114	0,062			0,052	2	0,088	HG-AAS	7,3	1,7
L20	0,030	0,032	0,030		0,003	2	0,031	HG-AAS	-1,7	-2,1
L22	<0,030	<0,030					<0,030	ETAAS		
L25	0,065	0,061			0,004	$\sqrt{3}$	0,063	HG-AAS	3,3	3,8
L26	0,033	0,033	0,033	0,034	0,004	2	0,033	HG-AAS	-1,4	-1,6
L27	0,043	0,042			0,019	2	0,043	ICP-MS	0,1	0,0
L30	0,0356	0,0348	0,0347		0,007	2	0,035	ICP-MS	-1,1	-1,1

*All results in  $\text{mg kg}^{-1}$*

**Table 2d:** Total Hg, quantitative information reported by the participants plus the laboratory scorings provided by the organiser.

**Total Hg content:  $0.0164 \pm 0.0022 \text{ mg kg}^{-1}$**

Lab code	x1	x2	x3	x4	Ulab	k	Mean-calc	Technique	z	zeta
L01	<0,05	<0,05	<0,05				<0,05	ICP-MS		
L02	0,019	0,018	0,018		0,003	2	0,018	AMA	0,8	1,0
L03	0,025	0,026	0,024		0,002	2	0,025	HG-AAS	3,5	5,8
L04	0,025				0	√3	0,025	CV-AFS	3,5	7,8
L05	0,025	0,028			0,02	2	0,027	CV-AAS	4,1	1,0
L06	0,0170	0,0191	0,0211		0,0029	2	0,0191	CV-AAS	1,1	1,5
L07	0,0168	0,0172	0,0179		0,0024	√3	0,0173	CV-AAS	0,4	0,5
L08	<0,034	<0,034	<0,034				<0,034	TDA-AAS		
L09	0,0356	0,0369	0,0324		0,005	2	0,0350	CV-AAS	7,5	6,8
L10	0,017	0,019			0,003	2	0,018	Autoana. AAS	0,7	0,9
L11	0,034	0,033			0,008	2	0,034	ICP-MS	7,0	4,1
L12	0,0163	0,0184	0,0168		0,002	2	0,0172	CV-AAS	0,3	0,5
L13	0,015	0,014			0,006	2	0,015	CV-AAS	-0,8	-0,6
L14	0,018	0,019	0,019		0,003	2	0,019	AMA-254	0,9	1,2
L15	<0,015	<0,015					<0,015	CV-ICP-AES		
L16	0,022	0,023			0,007	√3	0,023	AMA254	2,5	1,5
L17	0,017	0,015			0,005	2	0,016	Amalgamation	-0,2	-0,1
L18	<0,1	<0,1					<0,1	CV-AAS		
L19	<0,081	<0,081					<0,081	HG-AAS		
L20	0,018	0,020			0,003	2	0,019	ICP-MS	1,1	1,4
L21	0,012	0,014	0,015		0,003	2	0,014	CV-AAS	-1,1	-1,5
L22	0,134	0,136			0,020	2	0,135	HG-AAS	48,2	11,8
L23	0,0144	0,0140	0,0145		0,0011	√3	0,0143	AMA	-0,9	-1,7
L25	0,019	0,023			0,003	√3	0,021	CV-AAS	1,9	2,2
L26	0,022	0,023	0,022	0,023	0,003	2	0,023	CV-AAS	2,5	3,3

*CRL-HM in Feed and Food. Total Cd, Pb, As, Hg and Sn and extractable Cd and Pb in feed of plant origin*

<b>Lab code</b>	<b>x1</b>	<b>x2</b>	<b>x3</b>	<b>x4</b>	<b>Ulab</b>	<b>k</b>	<b>Mean-calc</b>	<b>Technique</b>	<b>z</b>	<b>zeta</b>
L27	0,0180	0,0178			0,0027	2	0,0179	AMA 254	0,6	0,9
L30	0,0239	0,0203	0,021		0,006	2	0,022	AMA	2,2	1,7

*All results in mg kg<sup>-1</sup>*

**Table 2e:** Total Sn, quantitative information reported by the participants plus the laboratory scorings provided by the organiser.

**Total Sn content:  $0.062 \pm 0.011 \text{ mg kg}^{-1}$**

Lab ID	x1	x2	x3	x4	Ulab	k	Mean-calc	Technique	z	zeta
L01	<10	<10	<10				<10	ICP-MS		
L02	0,043	0,036	0,041		0,008	2	0,040	ICP-MS	-2,4	-3,2
L04	0,04				0	$\sqrt{3}$	0,04	ICP-MS	-2,4	-4,0
L05	11,212	10,978	10,490		0,74	2	10,89	ETAAS-Z	1164,7	29,3
L09	120,68	116,78	118,73		23,74	2	118,73	FAAS	12760,0	10,0
L10	<0,050	<0,050					<0,050	ICP-MS		
L11	0,065	0,083			0,006	2	0,074	ICP-MS	1,3	1,9
L13	<1	<1					<1	ICP-AES		
L17	0,52	0,53			0,1	2	0,53	ICP-AES	49,8	9,2
L18	0,016	0,023			0,004	2	0,0195	ETAAS	-4,6	-7,3
L20	0,055	0,057			0,006	2	0,056	ICP-MS	-0,6	-1,0
L22	<0,100	<0,100					<0,100	ETAAS		
L25	0,197	0,164			0	$\sqrt{3}$	0,181	ICP-AES	12,7	21,5
L26	0,084	0,100	0,093	0,102	0,018	2	0,095	ICP-MS	3,5	3,1
L27	0,114	0,148			0,050	2	0,131	ICP-MS	7,4	2,7
L30	<3	<3	<3				<3	ICP-AES		

*All results in  $\text{mg kg}^{-1}$*

**Table 2f:** Extractable Cd, quantitative information reported by the participants plus the laboratory scorings provided by the organiser.

**Extractable Cd content:  $0.114 \pm 0.003 \text{ mg kg}^{-1}$**

Lab ID	x1	x2	x3	x4	Ulab	k	Mean-calc	Technique	z	zeta
L01	0,13	0,13			0,03	2	0,13	ICP-MS	0,9	1,1
L02	0,123	0,125			0,020	2	0,124	ICP-MS	0,6	1,0
L03	0,096	0,099	0,098		0,009	2	0,098	AAS	-1,0	-3,5
L04	0,12				0	$\sqrt{3}$	0,12	ETAAS	0,4	4,1
L05	0,1047	0,1070	0,1053		0,020	2	0,106	ETAAS-Z	-0,5	-0,8
L06	0,115	0,105			0,020	2	0,110	ETAAS	-0,2	-0,4
L07	0,115	0,119	0,099		0,021	$\sqrt{3}$	0,111	ETAAS	-0,2	-0,2
L10	0,13	0,12			0,030	2	0,13	ICP-MS	0,6	0,7
L12	0,120	0,114			0,007	2	0,117	ICP-MS	0,2	0,8
L13	0,116	0,118			0,023	2	0,117	ETAAS	0,2	0,3
L14	0,086	0,087	0,091		0,019	2	0,088	ETAAS	-1,5	-2,7
L15	0,126	0,120			0,025	2	0,123	ICP-AES	0,5	0,7
L16	0,112	0,114			0,001	2	0,113	ETAAS	-0,1	-0,7
L17	0,112	0,107			0,033	2	0,110	ETAAS	-0,3	-0,3
L18	0,026	0,025			0,002	2	0,0255	ETAAS	-5,2	-50,2
L19	0,065	0,066			0,009	2	0,066	ETAAS	-2,8	-10,3
L20	0,121	0,122			0,012	2	0,122	ICP-MS	0,4	1,2
L22	0,090	0,090			0,019	2	0,090	ETAAS	-1,4	-2,5
L24	0,401	0,399	0,410	0,398	0,040	2	0,402	ETAAS	16,8	14,4
L25	0,085	0,086			0,012	$\sqrt{3}$	0,086	ICP-AES	-1,7	-4,0
L26	0,112	0,113	0,112	0,111	0,012	2	0,112	ETAAS	-0,1	-0,3
L27	0,109	0,107			0,011	2	0,108	ICP-MS	-0,4	-1,1

*All results in  $\text{mg kg}^{-1}$*



**Table 2g:** Extractable Pb, quantitative information reported by the participants plus the laboratory scorings provided by the organiser.

**Extractable Pb content:  $1.67 \pm 0.11 \text{ mg kg}^{-1}$**

Lab ID	x1	x2	x3	x4	Ulab	k	Mean-calc	Technique	z	zeta
L01	1,95	2,06			0,62	2	2,01	ICP-MS	1,3	1,1
L02	1,834	1,907			0,281	2	1,871	ICP-MS	0,8	1,3
L03	1,436	1,424	1,468		0,143	2	1,443	AAS	-0,9	-2,5
L04	1,3				0	$\sqrt{3}$	1,3	ETAAS	-1,5	-6,7
L05	1,02	0,98	1,00		0,20	2	1,00	ETAAS-Z	-2,7	-5,9
L06	1,260	1,343			0,286	2	1,302	ETAAS	-1,5	-2,4
L07	1,38	1,62	1,52		0,24	$\sqrt{3}$	1,51	ETAAS	-0,7	-1,1
L08	1,82	1,91			0	$\sqrt{3}$	1,87	ZETAAS	0,8	3,5
L10	1,8	1,6			0,29	2	1,7	ICP-MS	0,1	0,2
L12	1,482	1,548	1,782		0,080	2	1,604	ICP-MS	-0,3	-1,0
L13	1,73	1,73			0,35	2	1,73	ETAAS	0,2	0,3
L14	1,99	1,98	2,04		0,50	2	2,00	ETAAS	1,3	1,3
L15	1,65	1,42			0,31	2	1,54	ICP-AES	-0,5	-0,8
L16	1,265	1,209			0,001	2	1,237	ETAAS	-1,7	-7,9
L17	1,88	1,69			0,53	2	1,79	ETAAS	0,5	0,4
L18	1,99	1,98			0,4	2	1,985	ETAAS	1,3	1,5
L19	1,728	1,751			0,081	2	1,740	ETAAS	0,3	1,0
L20	1,83	1,82			0,18	2	1,83	ICP-MS	0,6	1,5
L22	1,13	1,18			0,13	2	1,16	ETAAS	-2,1	-6,0
L24	0,971	0,994	0,979		0,03	2	0,981	ETAAS	-2,7	-12,1
L25	1,780	1,767			0,213	$\sqrt{3}$	1,774	ICP-AES	0,4	0,8
L26	1,50	1,65	1,52	1,58	0,22	2	1,56	ETAAS	-0,4	-0,9
L27	1,66	1,68			0,17	2	1,67	ICP-MS	0,0	0,0

*All results in  $\text{mg kg}^{-1}$*

**Table 3:** Number and percentage of laboratories reporting results not "*< than*" with satisfactory, questionable and unsatisfactory scores.

	Total Cd		Extr. Cd		Total Pb		Extr. Pb		Total As		Total Hg		Total Sn	
	N°	%	N°	%	N°	%	N°	%	N°	%	N°	%	N°	%
<i>z</i>														
S	21	84	19	86	25	96	20	87	10	50	13	59	2	18
Q	2	8	1	5	0	0	3	13	1	5	3	14	2	18
U	2	8	2	9	1	4	0	0	9	45	6	27	7	64
<i>zeta</i>														
S	16	64	14	64	18	69	15	65	9	45	15	68	2	18
Q	3	12	2	9	4	15	2	9	5	25	1	5	1	9
U	6	24	6	27	4	15	6	26	6	30	6	27	8	73

N°: Number of laboratories, S: Satisfactory, Q: Questionable, U: Unsatisfactory.

**Table 4:** Comments submitted by the participants to this exercise.

Lab ID	Comments
L01	We found that the extractable Lead results were much higher than the total lead results (they would normally be the same or slightly lower) The extractable acid solution was deeply coloured ( orange – brown) and had a lot of precipitate. Whereas the totally digested solutions were colourless – pale yellow With no precipitate. We carried out some investigations and found that we obtained the same results each time. This may present an interesting topic for next years workshop, providing that this was the outcome in
L04	The amount of sample was not sufficient
L07	Not enough material for determination 5 metals
L08	As: uncertainty 12 % with k=2,04; Hg: uncertainty 15 % and k=2,18; Pb: uncertainty 14 % and k=2,00, Cd: uncertainty 16 % and k=2,00
L15	The analysis of Cd, Pb and Hg are not accredited for this kind of sample
L25	There was not enough sample material; the procedure was only constructed for microwave digestion regarding to sample mass
L26	Sample amount was quite low
L27	Determination of Sn in feed is not accredited because this determination is not provided by our laboratory
L30	We are accredited for the analysis of Cd, Pb and Hg in food but not in feed

## 9 Conclusions

The main conclusion that can be extracted from this exercise is that special attention needs to be paid to contamination issues when analysis of low level materials is performed, as it has been proven in this exercise with the results reported for total As.

Once more it became evident that an extra effort is needed in the evaluation of uncertainties associated to the results, since the number of questionable and unsatisfactory zeta-scores is systematically higher than those of z-scores for all measurands. The uncertainty associated to a certain result is of paramount importance in cases of litigation and so it is fundamental for control laboratories to be able to report a sound uncertainty statement.

It can also be derived from this exercise that the concentrations of total and extractable Pb in the test material used are identical, while the concentration of total and extractable Cd are slightly different, namely extractable Cd accounts for 94 % of the total. It must be kept in mind that the previously described findings apply to the particular material used in IMEP-108 but might be different in another material.

## 10 Acknowledgements

C. Contreras from the Reference Materials Unit is acknowledged for her support in the processing of the test material. The coordinators of the exercise also thank the Reference Materials Unit for the provision of the candidate Reference Material used as test material in this exercise. I. Baer, F. Cordeiro and Anne-Mette Jensen are thanked for revising the manuscript.

The NRLs participating in this exercise, listed below are kindly acknowledged.

Organisation	Country
AGES, Kompetenzzentrum Elemente	Austria
AGES, Zentrum Analytik und Mikrobiologie	Austria
CODA-CERVA	Belgium
Central Laboratory of Veterinary Control and Ecology	Bulgaria
CISTA	Czech Republic
State Veterinary Institute Olomouc	Czech Republic
The Danish Plant Directorate	Denmark
Agricultural Research Centre	Estonia
Evira	Finland
Laboratoire SCL de Bordeaux	France
Federal Office of Consumer Protection and Food Safety (BVL)	Germany
General Chemical State Laboratory	Greece
Regional Center of Plant Protection And Quality Control of Magnisia	Greece
Central Agricultural Office, Food and Feed Safety Directorate	Hungary
Health Service Executive	Ireland
Istituto Zooprofilattico Sperimentale del Piemonte, Liguria e Valle D'Aosta	Italy
National Diagnostic Centre	Latvia
National Food and Veterinary Risk Assessment Institute	Lithuania
Scientific Institute of Public Health	Luxembourg
Public Health Laboratory Malta	Malta
Food and Consumer Product Safety Authority (VWA)	Netherlands
RIKILT- Institute of Food Safety	Netherlands
National Veterinary Research Institute	Poland
Laboratório Nacional de Investigação Veterinária	Portugal
Hygiene Institute of Veterinary Public Health	Romania
State Veterinary and Food Institute - Koaice	Slovakia
Laboratorio Arbitral Agroalimentario	Spain
National Veterinary Institute (SVA)	Sweden

Countries not appearing on the above list did not report results for this ILC.

## 11 References

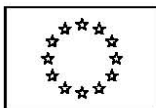
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- <sup>1</sup> Directive 2002/32/EC of the European Parliament and of the Council of 7 May 2002 on undesirable substances in animal feed.
- <sup>2</sup> ISO 13528:2005; Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons.
- <sup>3</sup> P. Weigert, Metal Loads of Food and Vegetable Origin Including Mushrooms, in: Metals and their Compounds in the Environment. Occurrence, Analysis and Biological Relevance, Ed. E. Merian, VCH, Weinheim, 1991.
- <sup>4</sup> Regulation (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare.
- <sup>5</sup> Evaluation of measurement data — Guide to the expression of uncertainty in measurement. JCGM 100, (2008)
- <sup>6</sup> M. Thompson, S.L.R. Ellison, R. Wood, *Pure Appl. Chem.*, (2006), **78(1)**, 145-196.
- <sup>7</sup> Eurachem/CITAC guide, Quantifying Uncertainty in Analytical Measurements, 2000 ([www.eurachem.ul.pt](http://www.eurachem.ul.pt)).
- <sup>8</sup> The software to calculate Kernel densities is provided by the Statistical Subcommittee of the Analytical Methods Committee (AMC) of the Royal Society of Chemistry and described in the AMC Technical Brief "Representing data distributions with Kernel density estimates" (2006), see [www.rsc.org/amc](http://www.rsc.org/amc)
- <sup>9</sup> Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs.

**Annexes**

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## Annex 1: Invitation letter



EUROPEAN COMMISSION  
JOINT RESEARCH CENTRE

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



Geel, 13 October 2009  
JRC.D08/BCa/ive/ARES(2009)275812

«Title» «M\_1st\_name» «last\_name»  
«Institute»  
«Department»  
«Address»  
«DHL\_delivery\_address»  
«ZIP» «City»  
«COUNTRY»

Dear Madam / Sir,

### **Inter-laboratory comparison for CRL Heavy Metals in Feed and Food**

On behalf of the CRL Heavy Metals in Feed and Food, I would like to invite you to participate in the Proficiency Test [IMEP-108] for the "**Determination of total Cd, Pb, As, Hg and Sn and extractable amounts of Cd and Pb in feed of plant origin following Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed**".

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 CRL if you hold a mandate for the type of matrix investigated.

Please register electronically for this inter-laboratory comparison using the following link: <https://irmm.jrc.ec.europa.eu/ilc/ilcRegistration.do?selComparison=339>  
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 23 October 2009**. Samples will be sent to participants during the second half of October. The deadline for submission of results is 30 November 2009.

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](mailto:jrc-irmm-crl-heavy-metals@ec.europa.eu)

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

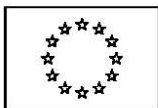
Yours sincerely

A handwritten signature in black ink, appearing to read 'Dr. M.B. de la Calle', with a long horizontal flourish extending to the right.

Dr. M.B. de la Calle  
Operating Manger CRL-HM

Cc: Franz Ulberth

## Annex 2: Acknowledgement of receipt form



EUROPEAN COMMISSION  
JOINT RESEARCH CENTRE

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



Annex to JRC.D08/BCa/ive/ARES(2009)/288628

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

### CRL-HM-08 / IMEP-108

**total Cd, Pb, As, Hg and Sn and  
extractable amounts of Cd and Pb in mineral feed**

### Confirmation of receipt of the samples

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

ANY REMARKS .....

Date of package arrival .....

Signature .....

**Please return this form to:**

Dr Beatriz de la Calle

IMEP-108 Coordinator  
EC-JRC-IRMM  
Retieseweg 111  
B-2440 GEEL, Belgium

Fax : +32-14-571865

e-mail : [JRC-IRMM-CRL-HEAVY-METALS@ec.europa.eu](mailto:JRC-IRMM-CRL-HEAVY-METALS@ec.europa.eu)

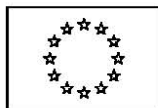
Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. <http://irmm.jrc.ec.europa.eu>  
Telephone: direct line (32-14) 571 252. Fax: (32-14) 571 865.

E-mail: [jrc-irmm-crl-heavy-metals@ec.europa.eu](mailto:jrc-irmm-crl-heavy-metals@ec.europa.eu)





## Annex 3: Accompanying letter



EUROPEAN COMMISSION  
JOINT RESEARCH CENTRE

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



Geel, October 2009  
JRC-D08/BCa/ive/ARES(2009)288628

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

**Participation to IMEP-108, a proficiency test exercise for the determination of total Cd, Pb, As, Hg and Sn and of extractable amounts of Cd and Pb in mineral feed**

Dear «TITLE» «SURNAME»,

Thank you for participating in the IMEP-108 intercomparison for the determination of total Cd, Pb, As, Hg and Sn and extractable amounts of Cd and Pb in mineral feed. This exercise takes place in the frame of the CRL Heavy Metals in Feed and Food.

This parcel contains:

- a) One glass bottle containing approximately 10 g of the test material
- b) A "Confirmation of Receipt" form
- c) This accompanying letter

Please check whether the bottle containing the test material remained undamaged during transport. Then fax (at +32-14-571865) or send the "Confirmation of receipt" form back. You should store the samples in a dark and cold place (not more than 18 °C) until analysis.

The measurands are: total Cd, Pb, As, Hg and Sn and extractable amounts of Cd and Pb according to Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed, in a mineral feed matrix.

As agreed upon during the workshop held in September, the determination of the extractable amounts of Cd and Pb shall be carried out by strictly applying the following procedure:

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

E-mail: [jrc-imm-crl-heavy-metals@ec.europa.eu](mailto:jrc-imm-crl-heavy-metals@ec.europa.eu)

«PARTKEY»

**Protocol for the partial extraction of Cd and Pb in mineral feed**

**(IMEP-105)**

1. Weigh about 2 g of the prepared test sample to the nearest 1 mg into a 250 mL beaker.
2. Add 85 mL of a 5 % (w/w) HNO<sub>3</sub> solution (see note for the preparation of the HNO<sub>3</sub> solution).
3. Cover the beaker with a watch-glass and boil for 30 min on a hot plate (make sure that the plate warms up homogeneously all over the surface).
4. Allow to cool. Decant the liquid into a 100 mL volumetric flask, rinsing the beaker and the watch-glass several times with 5 % (w/w) HNO<sub>3</sub>.
5. Dilute to the mark with 5 % (w/w) HNO<sub>3</sub>.
6. After homogenising, filter through a fry folded filter paper into a dry container. Use the first portion of the filtrate to rinse the glassware and discard that part. If the determination is not carried out immediately, the container with filtrate shall be stoppered.
7. Carry out a blank test at the same time as the extraction, with only the reagents and follow the same procedure as for the samples.

To construct the calibration curve dilute the standards in 5 % (w/w) HNO<sub>3</sub>.

NOTE: To prepare 1 kg stock of 5 % (w/w) HNO<sub>3</sub> (density ~ 1.0257 kg/l): mix 77 g of 65 % (w/w) HNO<sub>3</sub> with 923 g water. Use a balance of two digits for the weighing.

For the determination of the **total** content of Cd, Pb, As, Hg and Sn the procedure that you use 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 report the corrected values, plus their mean on the reporting website. The results should be reported in the same form (e.g., number of significant figures) as those normally reported to the customer.

The results 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:

*Weigh 2 g of test material and dry it at 103 ± 2 °C for 4 hours in triplicate*

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 parameter the two or three measurement results plus the technique you used, but do not report the uncertainty for each individual measurement. In addition, please report the mean of the results with technique and with uncertainty information in the allocated space for "measurement 4". After entering all results, please also complete the relating questionnaire. Do not forget to save, 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 30/11/2009.**

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](mailto:JRC-IRMM-CRL-HEAVY-METALS@ec.europa.eu)

With kind regards

A handwritten signature in black ink, appearing to read 'D. de la Calle', with a long horizontal line extending to the right.

Dr. M.B. de la Calle  
IMEP-108 Co-ordinator

Enclosures: 1) one glass bottle containing the test material; 2) confirmation of receipt form; 3) Accompanying letter.

Cc: F. Ulberth

## Annex 4: Questionnaire

Comparison for MRCF 169  
This questionnaire is offline

Please fill in the questionnaire

Submission Form

1. Did you apply a recovery factor to correct your measurement results?

no  
 yes

1.1. If yes, what are the recovery factors (R, in %) you used:

1.1.1. for Cd (in %)

1.1.2. for Pb (in %)

1.1.3. for As (in %)

1.1.4. for Hg (in %)

1.1.5. for Sn (in %)

1.2. If yes, did you determine R by:

1. adding a known amount of the same analyte to the sample  
 2. using a certified reference material  
 3. other

1.3. If no, please state why?

2. What is the level of confidence reflected by the coverage (k) factors stated above? (in %)

3. 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. expert guess/estimate  
 6. use of intercomparison data  
 7. other

3.1. If other, please specify

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

no  
 yes

5. Did you correct for the water content of the sample?

no  
 yes

5.1. If yes, what is the water content (in % of the sample mass)?

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

6. Did you modify the prescribed protocol for the partial digestion?

no  
 yes

6.1. If yes, please specify the modifications introduced

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

no  
 yes

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

7.1.1. sample pre-treatment

7.1.2. digestion step

7.1.3. extraction / separation step

7.1.4. instrument calibration step

7.2. If Yes, which:

8. Does your laboratory carry out this type of analysis (as regards the analytes, matrix and methods) o

no  
 yes

8.1. If Yes, please estimate the number of samples (As, Cd, Pb, Hg, Sn 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. Which type of sample treatment do you routinely use for such samples?

partial digestion (according to the legislation)  
 total digestion

11. Is your laboratory accredited for the sample treatment that you specify in question 10?

No  
 Yes

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

no  
 yes

12.1. If yes, which one(s)

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

no  
 yes

13.1. If YES, is the material used for the validation of procedures?

no  
 yes

13.2. If YES, is the material used for calibration of instruments?

no  
 yes

13.3. If yes, which one(s):

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

## Annex 5: Experimental details

Lab ID	SOP?	If yes, which	Sample pre-treatment	Digestion step	Extraction/separation step	Instrument calibration set
L01	Yes	JAOAC INTERNATIONAL Vol. 85, No 1, 2002				
L02	No		Shaking	Microwave assisted	Nitric acid	Yes
L03	No		Add 2 mL 65 % HNO <sub>3</sub> + 2 mL H <sub>2</sub> O <sub>2</sub>	Microwave	Dilution	4 step
L04	No			Microwave	See protocol IMEP-108	
L05	Yes	For Cd, Pb ASU L 00,00- 19/3, for Hg ASU L 00,00- 19/4				
L06	Yes					
L07	Yes	AOAC 999,11 for Cd, Pb; AOAC 971,21 for Hg				
L08	No			Pb, Cd, As: microwave high pressure digestion with H <sub>2</sub> O <sub>2</sub> (30 %) and HNO <sub>3</sub> conc. And HF conc.		Add. Method: std solution: Cd 2 ppb, Pb 50 ppb, As 20 ppb, Hg: non-linearity calibration from 25 ppb to 5 ppm
L09	Yes	SR EN 14546/2005, SR EN 14082/2003, SR EN 14083/2003, SR EN ISO 6869/2002, SR EN 13806/2003				
L10	No		No	Microwave with nitric acid		External calibration
L11	No		Homogeneise	Microwave digestion	n.a.	calibration using certified standard solutions
L12	Yes	AOAC				
L13	No			HNO <sub>3</sub> /HF for total Pb/Cd, HNO <sub>3</sub> /HClO <sub>4</sub> for As/Hg, HNO <sub>3</sub> /HCl for Sn, HNO <sub>3</sub> for extraction Pb/Cd		External calibration for all methods

CRL-HM in Feed and Food. Total Cd, Pb, As, Hg and Sn and extractable Cd and Pb in feed of plant origin

L14	No		Drying in oven	Pb and Cd ashing in muffle furnace at 450 °C, disolved in HCl; As ashing with magnesium nitrate hexahydrate solution		Calibration curve for Pb (10-60 µg/L; Cd (1-10 µg/L; As (3-25 µg/L) Hg (0,05-0,50 µg/L)
L15	No		Cd and Pb 0,4 g sample and 3 mL conc HNO <sub>3</sub> . Hg 1 g sample and HNO <sub>3</sub> and HClO <sub>4</sub>	Cd and Pb bomb digestion. Hg wet digestion in open vessels		External calibration. No standard addition
L16	Yes					
L17	Yes					
L18	Yes	MSZ EN 15550:2008, Hungarian Feed Codex (Hg)				
L19	No		None	Using 5 mL conc. nitric acid employing microwave digestion and open tube for Hg	As per the method provided for Pb and Cd	None
L20	No		None	High pressure microwave with conc HNO <sub>3</sub> and H <sub>2</sub> O <sub>2</sub>		External calibration, internal standard indium
L21	Yes	EN 14082, EN 13806				
L22	No			0,5 g of material, add nitric acid, microwave digestion, dilution to 25 mL		External standard calibration, check graph with CRM
L23	Yes					
L24	Yes	AOAC 999,10				
L25	No		All samples are handled according to EF152/2009	CD & Pb according to EF 152/2009 (Cu, Fe, Mn, Zn). As: Ashing of sample and Hg wet digestion	Hg: reduction of Hg(II) to Hg(0) with SnCl <sub>2</sub> . As: reduction of As(V) to As(III) with KI.	External calibration
L26	Yes					
L27	No					ICP-MS measurements with octopole reaction system
L30	No		No	HNO <sub>3</sub> + H <sub>2</sub> O <sub>2</sub> for tin analysis: set to volume with HCl 6 N	No	External calibration

European Commission

**EUR 24278 EN – Joint Research Centre – Institute for Reference Materials and Measurements**

Title: Report of the eighth interlaboratory comparison organised by the Community Reference laboratory for Heavy Metals in Feed and Food. Total Cd, Pb, As, Hg and Sn and extractable Cd and Pb in feed of plant origin

Author(s): M.B. de la Calle, A. Krata, C. Quénel, T. Linsinger, E. Perez Przyk, I. Verbist

Luxembourg: Publications Office of the European Union

2010 – 45 pp. – 21 x 29.7 cm

EUR – Scientific and Technical Research series – ISSN 1018-5593

ISBN 978-92-79-15223-8

DOI 10.2787/24640

**Abstract**

The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre, a Directorate General of the European Commission, operates the Community Reference Laboratory for Heavy Metals in Feed and Food (CRL-HM). One of its core tasks is to organise interlaboratory comparisons (ILCs) among appointed National Reference Laboratories (NRLs). This report presents the results of the eighth ILC of the CRL-HM which focused on the determination of total Cd, Pb, As, Hg and Sn and extractable amounts of Cd and Pb in feed of plant origin following 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 candidate reference material, the matrix being rye grass. The material was relabelled and dispatched to the participants on the second half of October 2009. Each participant received approximately 10 g of test material. Thirty participants from 25 member states registered to the exercise of which 27 reported results for total Cd, Pb and Hg, 23 for extractable Pb, 22 for extractable Cd and for total As and 16 for total Sn. One laboratory did not report results due to a breakdown of the instrumentation, and one laboratory reported results only for Hg for the same reason.

The assigned values ( $X_{\text{ref}}$ ) for total Cd, Pb, As, Hg and Sn were the candidate certified values as obtained during the certification. The assigned values for extractable Cd and Pb were provided by IRMM using isotope dilution-inductively coupled plasma-mass spectrometry (ID-ICP-MS).

Participants were invited to report the uncertainty of their measurements, which was provided by the majority of them. The laboratory performance was evaluated using z- and zeta-scores in accordance with ISO 13528. The standard deviation for proficiency assessment (also called target standard deviation),  $\hat{\sigma}$ , was fixed to 15 % of the assigned values for all measurands on the basis of the outcome of previous ILCs organised by the CRL-HM for the same population.

Between 85 and 95 % of the laboratories performed to a satisfactory level taking into consideration the z-scores for total and extractable Cd and Pb. The figures are not equally positive for the same measurands when looking at the zeta-scores only. 65 to 69 % of the reported results were satisfactory when the associated uncertainties are taken into account.



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