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Determination of total As, Cd, Pb and Hg in vegetable feed

IMEP-119 Proficiency Test Report

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Abstract

This report presents the results of a proficiency test round (PT, IMEP-119) of the EURL-HM focussing on the determination of total As, Cd, Pb and Hg in vegetable feed in support to Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed. The PT exercise was also opened to all laboratories who wish to take part in the exercise as a way to benchmark their performance against NRLs and other laboratories.

One hundred and two participants from 45 countries registered to the exercise. Only eight participants did not report their results.

Laboratory results were rated using z- and zeta (ζ-) scores in accordance with ISO 13528. The relative standard deviation for proficiency assessment was set to 15 % for the total As, Cd and Pb mass fractions, and to 22 % for the total Hg mass fraction, respectively.

An overall adequate performance for NRLs and feed control laboratories is shown by the percentage of satisfactory performance (expressed as z-scores). These percentages were ranging from 93 to 74 % for NRLs and from 92 to 69 %, for feed control laboratories, respectively.

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Executive summary

This report presents the results of a proficiency test (PT, IMEP-119), which focussed on the determination of total As, Cd, Pb and Hg in vegetable feed in support to Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed. The present PT was also opened to all laboratories wishing to take part in the exercise to benchmark their performance against NRLs and other laboratories.

One hundred and two participants from 45 countries registered to the exercise. Only eight participants did not report results.

The material used in this exercise as test item was a commercially available feed of vegetable origin (alfalfa meal) which, after appropriate processing, was bottled, labelled and dispatched to the participants at the beginning of May 2014. Four laboratories with demonstrated measurement capabilities in the field provided results to establish the assigned values (X_{ref}). The standard uncertainties associated to the assigned values (u_{ref}) were calculated according to ISO 13528:2005.

Laboratory results were rated using *z*- and zeta (*ζ*-) scores in accordance with ISO 13528. The relative standard deviation for proficiency assessment was set to 15 % for the total As, Cd and Pb mass fractions, and to 22 % for the total Hg mass fraction, respectively.

An overall adequate performance for NRLs and feed control laboratories is shown by the percentage of satisfactory performance (expressed as z-scores). These percentages were ranging from 93 to 74 % for NRLs and from 92 to 69 %, for feed control laboratories, respectively.

1. Introduction

The present proficiency test (PT, IMEP-119) was carried out by the European Union Reference Laboratory for Heavy Metals in Feed and Food (EURL-HM) to assess the performance of National Reference Laboratories (NRLs) and other laboratories (non-NRLs), such as official control laboratories, in the determination of total arsenic, cadmium, lead and mercury in a vegetable feed.

The PT exercise was requested by the Directorate General for Health and Consumers (DG SANCO) and agreed with the NRLs during the 8th EURL-HM workshop (Brussels, the 24th September 2013).

The vegetable feed used in the present proficiency test as test item is alfalfa meal, a product made from the alfalfa plant (a member of the *Fabaceae* pea family called *Medicago sativa)*. Alfalfa meal can be fed to a variety of livestock, poultry and horses. Alfalfa meal can also be used as compost or as natural fertilizer to provide the soil with the basic nitrogen-phosphorous-potassium combination.

Alfalfa has high content of protein, digestible fiber, vitamins and digestible energy, which can be utilized in feed formulations. However, the use of such materials as feeding stuff needs surveillance as it may contain constituents, such as heavy metals, which are considered as undesirable substances.

Directive 2002/32/EC of the European Parliament and of the Council on undesirable substances in animal feed [1], describes "compound feedingstuffs" as the "mixtures of feed materials, whether or not containing additives, which are intended for oral animal feeding as complete or complementary feedingstuffs". The Directive and its amendments [1] set maximum levels (MLs) for undesirable substances in animal feed (organic and inorganic). All the trace elements included as measurands in the present PT are listed, as undesirable substances in feed materials in the above mentioned Directive, with MLs of 2.0, 10.0, 0.1 and 1.0 mg kg^{-1} for the total As, Pb, Hg and Cd mass fractions, respectively.

Considering that all values accepted as the assigned values for the PT assessment are well below the MLs listed above, the test item should be considered as compliant with the European legislation.

This report summarises and evaluates the outcome of the PT exercise.

2. IMEP support to EU policy

The International Measurement Evaluation Programme (IMEP) is operated by the Joint Research Centre - Institute for Reference Materials and Measurements (JRC-IRMM). IMEP provides support to the European measurement infrastructure in the following ways:

IMEP disseminates metrology from the highest level down to the field laboratories. These laboratories can benchmark their measurement result against the IMEP assigned value, which is established according to metrological best practice.

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

IMEP supports EU policies by organising interlaboratory comparisons (ILCs) in the frame of specific EU legislation or on request of a specific EC Directorate-General. In the case of IMEP-119 it was organised to support the Directorate General for Health and Consumers (DG SANCO) with the implementation of the European Commission Directive 2002/32/EC [1].

Furthermore, IMEP-119 provided support to the following stakeholders:

- The European Cooperation for Accreditation (EA) in the frame of a Collaboration Agreement on a number of metrological issues, including the organisation of interlaboratory comparisons. This report does not discern the EA nominees from the other participants. Their results are however summarised in a separate report to EA.

- The Asia Pacific Laboratory Accreditation Cooperation (APLAC), in the frame of the collaboration with APLAC.

- The InterAmerican Accreditation Cooperation (IAAC).

3. Scope and aim

As stated in Regulation (EC) No 882/2004 [2] one of the core duties of the European Union Reference Laboratories (EURLs) is to organise interlaboratory comparisons (ILCs) for the benefit of the staff from National Reference Laboratories (NRLs).

IMEP-119 aimed to test the competences of NRLs and other laboratories (non-NRLs), such as official control laboratories (OCLs), to determine the total arsenic (As), cadmium (Cd), lead (Pb) and mercury (Hg) mass fractions in feed of vegetable origin. In addition, participants were asked to evaluate the conformity of the test item analysed as animal feed stuff according to Directive 2002/32/EC.

The assessment of measurement results is undertaken on the basis of requirements laid down in legislation [1] and follows the administrative and logistic procedures of the International Measurement Evaluation Program (IMEP).

JRC-IMEP is accredited according to ISO 17043:2010 [3]. The name of this proficiency test round is IMEP-119.

4. Set up of the exercise

4.1 Time frame

The organisation of the IMEP-119 exercise was agreed upon by the NRL network at the 8th EURL-HM Workshop held in Brussels on September 24, 2013. Invitation letters were sent to the various participants on March 20, 2014 (Annex 1 to 4) and a web announcement (Annex 5) for the exercise was made on the JRC webpage on the same day. The registration deadline was April 24, 2014. The reporting deadline was set to June 13, 2014. Dispatch was followed by the PT coordinator using the messenger's parcel tracking system on the internet.

4.2 Confidentiality

The following confidentiality statement was made to EA, APLAC and IAAC: *"Confidentiality of the participants and their results towards third parties is guaranteed".* In the case of EA the following was added: "*However, IMEP will disclose details of the participants that have been nominated by EA to you. The EA accreditation bodies may wish to inform the nominees of this disclosure".* A similar clause was provided to those NRLs who wished to appoint official control laboratories in their respective country to take part in IMEP-119.

4.3 Distribution

Test items were dispatched to NRLs on May 5, 2014 and to the other participants on May 6, 12 and 13, 2014. Each participant received:

- One glass bottle containing approximately 25 g of test item;
- A "Sample accompanying letter" (Annex 6); and
- A "Confirmation of receipt form" to be sent back to IRMM after receipt of the test item (Annex 7).

4.4 Instructions to participants

Detailed instructions were given to participants in the "Sample accompanying letter" mentioned above. Measurands were defined as "Total As, Cd, Pb and Hg in vegetable feed".

Participants were asked to perform two or three independent measurements, to correct their measurements for recovery and for moisture content (applying a protocol described in the sample accompanying letter) and to report their calculated mean (x_{lab}, expressed on a dry mass) and its associated measurement uncertainty (u_{lab}).

Participants received an individual code to access the on-line reporting interface, to report their measurement results and to complete the related questionnaire. A dedicated questionnaire was used to gather additional information related to measurements and laboratories (Annex 8).

Participants were informed that the procedure used for the analysis should resemble as closely as possible their routine procedures for this particular matrix, analyte and concentration level.

The laboratory codes were given randomly and communicated to the participants by e-mail.

5. Test item

5.1 Preparation

The test item used was a vegetable feed (Alfalfa-meal) provided by the National Laboratory for Feeding Stuffs of the National Research Institute of Animal Production (Lublin, Poland). About 7 kg of the test item were sent to the JRC-IRMM. Once received, the material was stored at -20 °C until processing.

The material was first cryogenically milled using a Palla VM-KT vibrating mill from Humboldt-Wedag (Köln, Germany). All grinding elements in this system were made of high purity titanium to avoid contamination. After milling, the material was sieved over a 250 µm stainless steel sieve. The resulting coarse fraction was cryogenically milled and sieved in the same conditions. The collected coarse fraction went through a third run of those two steps. All fine fractions (6024 g for the first run, 613 g for the second run and 171 g for the third run) were pooled to produce 6808 g of sieved powder.

The material was then freeze dried in a freeze dryer from Martin Christ model Epsilon 2-100D (Osterode, Germany). Six Teflon coated trays were filled with about 1100 g each of the powder. A total of about 5700 g of dried alfalfa-meal powder was collected. Mixing was performed in a Dynamix CM-200 (WAB, Basel, Switzerland) for one hour.

The Karl Fischer titration and laser diffraction analysis indicate that the material had a water content of 3.8 % (m/m) with a top particle size below 450 µm.

No spiking was necessary since the endogenous content of As, Cd, Pb and Hg was considered appropriate.

Finally, portions of 25 g were filled into 125 ml amber glass acid-washed bottles. The bottles were manually filled using acid washed plastic spoons under an extraction point. The bottles were closed with acid washed inserts and screw caps.

Each vial was identified/labelled following the IMEP procedures to include a unique number and the name of the PT exercise.

5.2 Homogeneity and stability

Measurements for the homogeneity and stability studies were performed by the Centro de Salud Pública de Alicante (CSPA, Alicante, Spain). Inductively coupled plasma mass spectrometry (ICP-MS), after microwave digestion (0.25 g of feed in a mixture of HNO_3/H_2O_2 (30 %)) was used to determine the total As, Cd and Pb mass fractions.
An elemental mercury analyser (EMA) was used to quantify the total Hg mass fraction, using approximately 100 mg of feed per analysis.

The statistical treatment of data was performed at IRMM.

Homogeneity was evaluated according to ISO 13528:2005 [4]. The test item proved to be adequately homogeneous for all the investigated measurands.

The stability study was conducted applying the isochronous design [5, 6]. The test item proved to be adequately stable for all measurands during the 6 weeks that elapsed between the dispatch of the samples and the deadline for reporting.

The contribution from homogeneity (u_{bb}) and stability (u_{st}) to the standard measurement uncertainty of the assigned value (u_{ref}) was calculated using SoftCRM [7]. The analytical results reported by the expert laboratories and the statistical evaluation of the homogeneity and stability studies are presented in Table 1 and in Annex 9.

6. Reference values and their uncertainties

6.1 Assigned value X_{ref}

The assigned values for the four measurands (total As, Cd, Pb and Hg in vegetable feed) were determined by four laboratories, all selected based on their demonstrated measurement capabilities (later referred as expert laboratories):

- *ALS Scandinavia AB (Luleå, Sweden);*
- *SCK-CEN - Studiecentrum voor Kernenergie (Mol, Belgium);*
- *BAM - Bundesanstalt für Materialforschung und – Prüfung (Berlin, Germany);*
- *CSPA - Centro de Salud Pública de Alicante (Alicante, Spain)*

Expert laboratories were asked to use the method of analysis of their choice and no further requirements were imposed regarding methodology. Expert laboratories were also required to report their results together with the associated expanded measurement uncertainty and with a clear and detailed description on how their measurement uncertainty was calculated. Expert laboratories were not requested to report values for all measurands.

- ALS Scandinavia used inductively coupled plasma sector field mass spectrometry (ICP-SFMS) after closed microwave digestion of the sample (approximately 0.4 g in closed Teflon containers) using HNO_3, H_2O_2 and HF.

Analyses were made according to EPA 200.8 method (modified). ALS reported results for the total As, Cd, Pb and Hg mass fractions.

- SCK-CEN used instrumental neutron activation analysis (k_0-INAA) for the determination of total As, Cd and Hg mass fractions. Three test samples of about 1000 mg were taken from each bottle and transferred in standard high-density polyethylene vials. After weighing, samples were placed in the irradiation vials together with six IRMM-530 (Al-0.1 % Au alloy) neutron flux monitors, AMELS II and a BCR 176 validation sample. IRMM-530 monitors were used to determine the neutron flux during irradiation. SMELS II and BCR 176 were used to validate their experimental protocols.

- BAM used quadrupole ICP-MS for the total As, Cd and Pb mass fractions, while cold-vapour atomic fluorescence spectrometry (CV-AFS) was used for the total Hg mass fraction. A test sample of approximately 0.3 g was used for each analysis. Microwave-assisted digestion was used with HNO_3 and HF as digestion mixture. A certified reference material, BCR-482 (lichen) from IRMM was used to assess trueness.

- CSPA used ICP-MS after microwave digestion for the total As, Cd and Pb mass fractions, while elemental mercury analysis (EMA) was used for the total Hg mass fraction. Four certified reference materials were used to assess accuracy and trueness: IRMM 804 (rice flour) and BCR-191 (brown bread) from the IRMM; LGC7162 (strawberry leaves) from the Laboratory of Government Chemist (LGC, UK); and GBW07605 (tea leaves) from National Analysis Centre for Reference Materials (China). For the determination of total As, Cd and Pb mass fractions, approximately 0.25 g of test sample was used for each digestion. HNO_3 and H_2O_2 were used as digestion mixture. For Hg a test sample of 0.10 g was used with HCl as digestion mixture.

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

6.2 Associated uncertainty u_{ref}

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

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

In all cases the expert laboratories reported values with overlapping expanded measurement uncertainties (Table 1), hence u_{char} was calculated according to ISO 13528:2005 [4]:

$$u_{char} = \frac{1.25}{p}\sqrt{\sum_{1}^{p} u_i^2} \qquad \text{Eq. 2}$$

where p is the number of expert laboratories used to assign the reference value; and u_i is the standard measurement uncertainty reported by the experts.

Table 1 presents the average measurement values reported by the expert laboratories and their associated expanded measurement uncertainties, the assigned values (X_{ref}, u_{ref} and U_{ref}), all standard measurement uncertainty contributions (from characterization, homogeneity and stability) and the standard deviation for the PT assessment, σ.

Table 1 – *Average measurement values reported by the expert laboratories, assigned values, their associated expanded measurement uncertainties and the standard deviation for the PT assessment (all values in mg kg^{-1}).*

	As	Cd	Pb	Hg
Expert 1	1.2 ± 0.23	0.12 ± 0.025	3.06 ± 0.67	0.008 ± 0.0008
Expert 2	1.14 ± 0.17	0.122 ± 0.016	3.22 ± 0.31	0.0072 ± 0.00033
Expert 3	1.2 ± 0.07	0.142 ± 0.008	3.23 ± 0.023	
Expert 4	1.19 ± 0.06			
X_{ref}	**1.183**	**0.128**	**3.170**	**0.0076**
u_{char}	0.0470	0.0064	0.1539	0.00027
u_{bb}	0.0248	0.0032	0.0507	0.00023
u_{st}	0.0272	0.0023	0.0634	0.00027
u_{ref}	**0.0597**	**0.0075**	**0.174**	**0.00044**
U_{ref} (*)	0.119	0.015	0.348	0.0009
σ	**0.177**	**0.019**	**0.476**	**0.0017**
σ (%)	15.0%	15.0%	15.0%	22.0%

X_{ref} is the assigned value; $U_{ref} = k \cdot u_{ref}$ is the estimated associated expanded uncertainty; k=2 coverage factor corresponding to a level of confidence of about 95 %.

Note: Expert laboratories do not necessarily correspond to the order they were presented.

6.3 Standard deviation of the proficiency test assessment (σ)

The relative standard deviation for proficiency test assessment (σ, in %) was set for all measurands on the basis of previous PT rounds with similar measurands (IMEP-108, IMEP-111, IMEP-114, IMEP-117 and IMEP-38 [10]). σ was set to 15 % for the total mass fractions of As, Cd and Pb.

For the total Hg mass fraction, σ of 22 % was derived from the Thompson "modified Horwitz" equation [11] to take into consideration the low total Hg mass fraction in the test item.

7. Evaluation of results

7.1 Scores and evaluation criteria

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

$$z = \frac{x_{lab} - X_{ref}}{\sigma} \qquad \text{Eq. 3}$$

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

where:

	x_{lab}	is the measurement result reported by a participant;
	u_{lab}	is the standard measurement uncertainty reported by a participant;
	X_{ref}	is the assigned value;
	u_{ref}	is the standard measurement uncertainty of the assigned value;
	σ	is the standard deviation for proficiency test assessment.

The interpretation of the z- and ζ-score is done according ISO 17043:2010 [3]:

$\|score\| \leq 2$	satisfactory performance	(green in Annexes 10 to 15)
$2 < \|score\| < 3$	questionable performance	(yellow in Annexes 10 to 15)
$\|score\| \geq 3$	unsatisfactory performance	(red in Annexes 10 to 15)

The z-score compares the participant's deviation from the assigned value with the standard deviation for proficiency test assessment (σ) used as common quality criterion.

The ζ-score provides an indication of whether the participant's estimate of uncertainty is consistent with the observed deviation from the assigned value [12]. The denominator is the combined uncertainty of the assigned value (u_{ref}) and the measurement uncertainty as stated by the laboratory (u_{lab}). The ζ-score includes all parts of a measurement result, namely the expected value (assigned value), its measurement uncertainty in 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 measurement uncertainty, or both.

The standard measurement uncertainty of the laboratory (u_{lab}) was obtained by dividing the reported expanded measurement 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 measurement uncertainty was considered as the half-width of a rectangular distribution; u_{lab} was then calculated by dividing this half-width by $\sqrt{3}$, as recommended by Eurachem and CITAC [13].

Uncertainty estimation is not trivial, therefore an additional assessment was provided to each laboratory reporting measurement uncertainty, indicating how reasonable their measurement uncertainty estimation was.

The standard measurement 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}, case "a": $u_{min} \leq u_{lab} \leq u_{max}$). u_{min} is set to the standard measurement uncertainty of the assigned value (u_{ref}). It is unlikely that a laboratory carrying out the analysis on a routine basis would measure the measurand with a smaller measurement uncertainty than the expert laboratories chosen to establish the assigned value. u_{max} is set to the standard deviation accepted for the PT assessment (σ).

If u_{lab} is smaller than u_{min}, (case "b": $u_{lab} < u_{ref}$) the laboratory may have underestimated its measurement uncertainty. Such a statement has to be taken with care as each laboratory reported only measurement uncertainty, whereas the uncertainty associated with the assigned value also includes contributions for homogeneity and stability of the test item. If those are large, measurement uncertainties smaller than u_{min} are possible and plausible.

If u_{lab} is larger than u_{max}, (case "c": $u_{lab} > \sigma$) the laboratory may have overestimated its measurement uncertainty. An evaluation of this statement can be made when looking at the difference between the reported value and the assigned value: if the difference is smaller than U_{ref} then overestimation is likely. If the difference is larger but x_{lab} agrees with X_{ref} within their respective expanded measurement uncertainties, then the measurement uncertainty is properly assessed resulting in a satisfactory performance expressed as a ζ-score, though the corresponding performance, expressed as a z-score, may be questionable or unsatisfactory.

It should be pointed out that u_{max} is a normative criterion when set by legislation.

7.2 General observations

Results were received from 94 participants, from which 32 were NRLs. All registered NRLs (coded as NXX) reported results, while eight registered non-NRLs (coded as LXX) did not report results; two of the later explained that they encountered technical/instrumental difficulties that hindered their reporting.

7.3 Laboratory results and scorings

7.3.1 Performances

Annexes 10 to 13 present for each measurand the reported results as tables and graphs, distinguishing the NRL and non-NRLs populations. The corresponding Kernel density plots are also included, obtained using the software available from the Statistical Subcommittee of the Analytical Methods Committee of the UK Royal Society of Chemistry [14].

Figure 1 presents an overview of the performance of the participants, expressed as z- and ζ-scores for NRLs and non-NRLs (Fig. 1a and 1b, respectively).

The overall performance of the participants in this PT is considered as "satisfactory". The percentage of NRLs reporting results leading to satisfactory performances ($|z| \leq 2$) was 93 % for As and Pb, 90 % for Cd and 74 % for Hg. A similar picture is seen for the non-NRL participants (92 % for Pb, 91 % for As, 86 % for Cd and 69 % for Hg). The lower percentage of satisfactory performances for total Hg analysis for the two populations may be attributed to the low content of Hg in the test item (0.0076 ± 0.0009 mg kg^{-1}).

A similar tendency is observed when looking at "satisfactory" ζ-scores (Figure 1): between 61 % and 85 % for the NRLs; between 56 % and 77 % for non-NRLs. As often observed in previous IMEP rounds the percentage of "satisfactory" z-scores is higher that the ζ-score ones. This clearly identifies once more the need for laboratories to improve their measurement uncertainty calculations.

The evaluation of the measurement uncertainty estimation shows that most of the NRLs (from 55 to 65 %, depending on the measurand) reported "realistic" measurement uncertainties ($u_{ref} \leq u_{lab} \leq \sigma$). One third of the NRLs reported "likely underestimated" measurement uncertainties ($u_{lab} < u_{ref}$), while 10 to 17 % of them reported "likely overestimated" measurement uncertainties ($u_{lab} > \sigma$). As for the non-NRL population, a larger percentage (almost double) reported "likely underestimated" measurement uncertainties. Table 2 summarises the measurement uncertainty assessment based on the three categories defined.

Except for the total Hg mass fraction, only few participants reported "less than X" values. These values were not scored but were further evaluated. If the reported "less than X" value was lower than the corresponding $X_{ref} - U_{ref}$, this statement should be considered as incorrect, since the laboratory should have detected the respective measurand. Such results are flagged in red in Annexes 10 to 13. Most of the participants reported "lower than X" values corresponding to their limit of detection (LOD, in mg kg^{-1}). Others reported 2xLOD (six participants), 3xLOD (two) or 10xLOD (one).

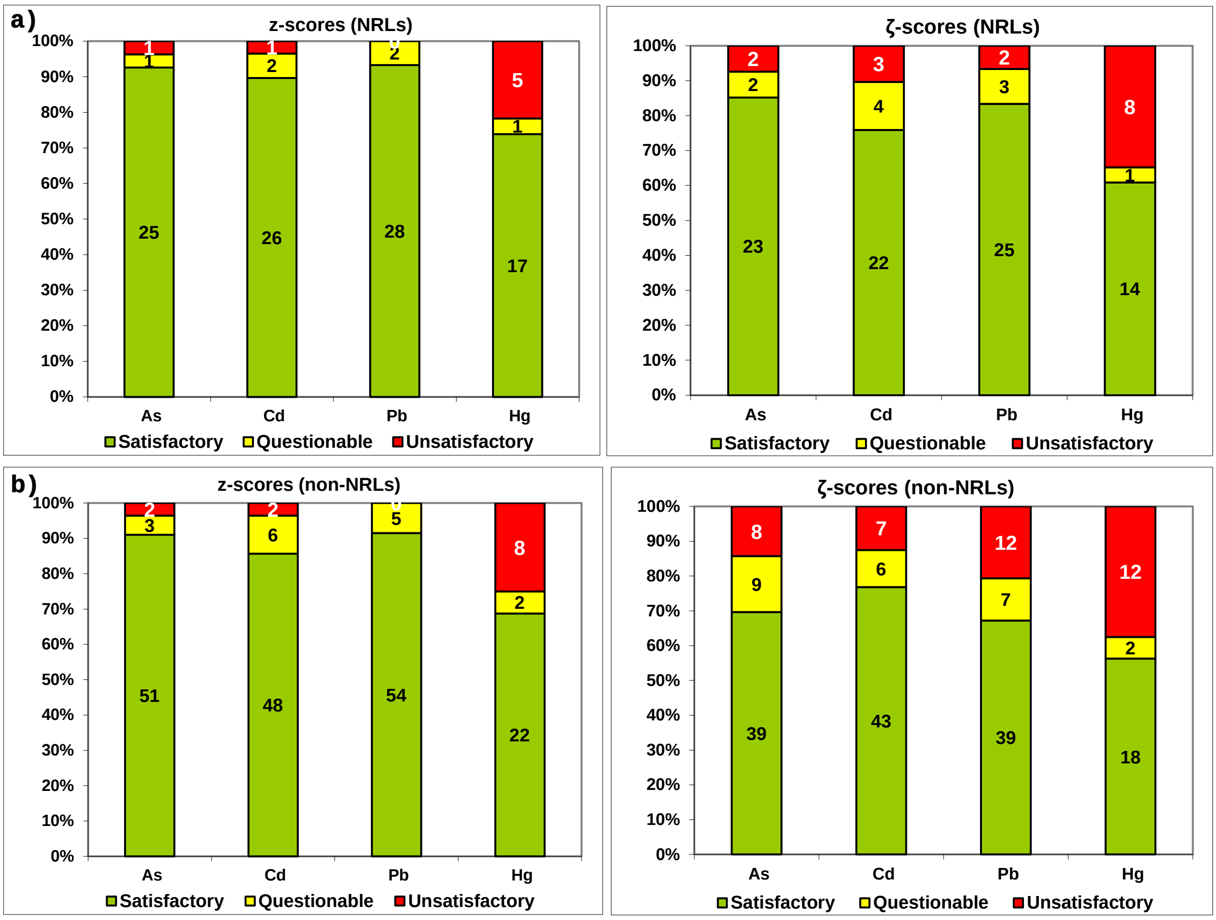

***Figure 1** – Overview of scores: in the number of laboratories and in %, having satisfactory, questionable or unsatisfactory performance. a) NRLs, b) non-NRLs*

***Table 2** – Uncertainty assessment. The figures are the % of participants for each group.*

	Case "a"		Case "b"		Case "c"	
	NRL	*non-NRL*	*NRL*	*non-NRL*	*NRL*	*non-NRL*
As	63	41	26	52	11	7
Cd	55	45	28	43	17	12
Pb	63	36	27	51	10	13
Hg	65	31	22	28	13	41

"a" : u_{min} (u_{ref}) ≤ u_{lab} ≤ u_{max} (σ); "b" : $u_{lab} < u_{min}$; and "c" : $u_{lab} > u_{max}$

7.3.2 Analysis of the information extracted from the questionnaire

When reporting their results participants were asked to answer a set of questions related to the analytical method used and to the quality assurance of their measurements. Annexes 14 (NRLs) and 15 (non-NRLs) summarises the answers to the questionnaire and their relation with the performance calculated for each participant (as z-scores).

No significant trend was observed with the analytical techniques used.

The majority (53 %) of NRLs estimated their analytical recovery (Annex 8, question 3, Q3) using certified reference materials (CRMs). Two groups were identified in the non-NRL population, where 48 % used spiking/fortifying, while 41 % used CRMs.

Most of the participants digested the samples with microwaves in a closed vessel (Annex 8, Q5). Few participants (4 to 8 depending on the measurand) used dry ashing, and some others (3 to 6) applied wet digestion in an open vessel. About half of the results obtained applying "digestion in open vessels" were scored either unsatisfactory or questionable. Considering the low number of data, this observation has low statistical value. However, laboratories using this approach must keep in mind that some heavy metals are volatile as it is the case of Hg, As and Pb, and that special precautions must be taken to avoid loses by volatilisation.

Many laboratories used a mixture of HNO_3 and H_2O_2 to mineralise the sample, although HNO_3 and HCl, H_2O_2 with HNO_3 and HCl (or and HF) and HNO_3 or HCl alone have also been used by some participants (Annex 8, Q6).

Regarding the experience of the participants (Annex 8, Q8) the number of laboratories participating in IMEP-119 who carry out this type of analysis on a regular basis do not differ significantly if analysing 0-50 samples/year or 50-250 samples/year. A smaller number of participants stated to carry out this type of analysis on a regular basis - more than 1000 samples/year. No significant difference could be identified in performances based on the laboratory experience analysing such samples.

On average, NRLs and non-NRLs reported correct values for the moisture content of the test item (Annex 8, Q10).

The majority of NRLs estimated their measurement uncertainty using their in-house method validation data (Annex 8, Q11c) or applying the ISO GUM (Q11a), which resulted in "likely realistic" uncertainty statements ($u_{ref} \leq u_{lab} \leq \sigma$). On the other hand, most of the non-NRLs estimated their measurement uncertainty based on their in-house method validation data (Q11c), or estimating from replicates/precision (Q11d). As already mentioned in previous IMEP reports, measurement uncertainty estimation based only on repeatability/precision data usually lead to "likely underestimated" measurement uncertainty ($u_{lab} < \sigma$), where other sources of uncertainty are ignored. Table 2 clearly shows the higher percentage of non-NRL that have reported "likely underestimated" measurement uncertainty, when compared to the corresponding percentages for NRLs. Annexes 10-13 shows that most of the laboratories with "unsatisfactory" performance (expressed as ζ-scores) reported "likely underestimated" uncertainties. No reliable conclusions can be drawn for total Hg, where "unsatisfactory performance" (expressed as ζ-scores) may be attributed to the low Hg content – close to the quantification capabilities of the laboratories.

Regarding the compliance (Annex 8, Q15) of the test item towards Directive 2002/32/EC, all NRLs correctly assessed the vegetable feed investigated as an animal feedstuff compliant with the European legislation. Not all non-NRLs answered this question, a question relevant mainly to non-EU countries having trade exchanges with the EU market. Only four non-NRLs assessed the sample as non-compliant.

Annexes 14 (NRLs) and 15 (non-NRLs) present the additional experimental details and information extracted from the questionnaire (Annex 8 see Q3.2, Q4, Q5, Q6, Q8, Q10.1 and Q15).

8. Conclusion

Considering the overall satisfactory performance of the participating laboratories in IMEP-119, the analytical capability of NRLs and other laboratories (non-NRLs), such as official control laboratories, for the determination of the undesirable substances in feed of vegetable origin was successfully demonstrated at the investigated concentration levels.

As a whole, the NRL population showed better performance when compared to the other laboratories. This positive outcome may be due to (i) the seventeen PTs organised so far by the EURL-HM and (ii) the various trainings on relevant topics related to the analyses of heavy metals in feed and food provided by the EURL-HM during the annual workshops. This is particularly clear when considering the difference between NRL and non-NRLs performance (expressed as ζ-scores), in which the realistic measurement uncertainty estimation is identified.

Finally, participants are invited to pay due care in the determination of "realistic" limit of detection, for which a very large discrepancy for reported "less than"/LOD was identified within each measurand, even for the same analytical technique. Clear definition and some practical guidance on how to estimate this important method performance characteristic, are necessary.

9. Acknowledgements

The laboratories participating in this exercise, listed below, are kindly acknowledged. The following IRMM colleagues are also acknowledged: P. Conneely, for the determination of the moisture content; M-F. Tumba-Tshilumba for the characterisation of the particle size distribution; C. Contreras for the processing of the test item; F. Ulberth and H. Emteborg for reviewing the manuscript.

Organisation	Country
JLA: ARGENTINA S.A.	ARGENTINA
Dairy Technical Services	AUSTRALIA
AGES GmbH	AUSTRIA
FAVV - FLVVG	BELGIUM
Inagro vzw	BELGIUM
Institut Ernest Malvoz	BELGIUM
CODA-CERVA	BELGIUM
Federal Institute of Agriculture	BOSNIA - HERZEGOVINA
Bioensaios Análises e Consultoria Ambiental Ltda.	BRAZIL
M. CASSAB COMÉRCIO E INDÚSTRIA LTDA.	BRAZIL
Central Laboratory of Veterinary Control and Ecology	BULGARIA
RPC	CANADA
Laboratorio Corthorn Quality S.A.	CHILE
Tecnimicro laboratorio de análisis S.A.S	COLOMBIA
Croatian Veterinary Institute	CROATIA
Croatian National Institute of Public of Health	CROATIA
Department of Agriculture	CYPRUS
State Veterinary Institute Olomouc	CZECH REPUBLIC
CISTA	CZECH REPUBLIC
Eurofins Miljø A/S	DENMARK
Danish Veterinary and Food Adminstration	DENMARK
Nestle Ecuador S.A.	ECUADOR
Agricultural Reasearch Centre	ESTONIA
Finnish Food Safety Authority Evira	FINLAND
Laboratoire SCL Bordeaux	FRANCE
Center for Public Health	FYR OF MACEDONIA
JZU Centar za javno zdravje Skopje	FYR OF MACEDONIA
Landesanstalt für Landwirtschaft, Forsten und Gartenbau Sachsen-Anhalt (LLFG)	GERMANY
Bioanalytik Weihenstephan - TUM	GERMANY
Nds. Landesamt für Verbraucherschutz und Lebensmittelsicherheit (LAVES)	GERMANY
LUFA Speyer	GERMANY
Thüringer Landesanstalt für Landwirtschaft	GERMANY
University of Hohenheim	GERMANY
Staatliche Betriebsgesellschaft für Umwelt und Landwirtschaft	GERMANY
CVUA-Westfalen AöR Standort Arnsberg	GERMANY
Federal Office of Consumer Protection and Food Safety (BVL)	GERMANY
REGIONAL CENTRE FOR PLANT PATHOLOGY AND QUALITY CONTROL OF MAGNISSIA	GREECE
Instituto de Investigaciones Químicas, Biológicas, Biomédicas y Biofísicas de la Universidad Mariano	GUATEMALA
National Food Chain Safety Office	HUNGARY
Milouda & Migal laboratories Limited Partnership	ISRAEL
The Standards Institution of Israel	ISRAEL
Istituto Zooprofilattico Sperimentale della Sicilia	ITALY
ISTITUTO ZOOPROFILATTICO SPERIMENTALE DELLA PUGLIA E DELLA BASILICATA	ITALY
ISS - Istituto Superiore di Sanità -	ITALY
ISTITUTO ZOOPROFILATTICO SPERIMENTALE DEL PIEMONTE, LIGURIA E VALLE D'AOSTA	ITALY
Institute of Food Safety, Animal Health and Environment	LATVIA
JSC Labtarna	LITHUANIA
National Public Health Surveillance Laboratory	LITHUANIA
National Food and Veterinary Risk Assessment Institute	LITHUANIA
Environmental Health Directorate	MALTA
Food & Consumer Products Safety Authority	NETHERLANDS
RIKILT	NETHERLANDS

Organisation	Country
NIFES	NORWAY
LabNett Skien	NORWAY
Diaz Gill Medicina Laboratorial S.A.	PARAGUAY
Polcargo International	POLAND
Cracow's Veterinary Inspectorate	POLAND
Wroclaw University of Technology, Chemical Laboratory of Multielemental Analysis	POLAND
National Veterinary Research Institute	POLAND
INIAV	PORTUGAL
SUPREME COUNCIL OF HEALTH	QATAR
HYGIENE AND VETERINARY PUBLIC HEALTH INSTITUTE	ROMANIA
Institute of Public Health Leskovac	SERBIA
Zavod za javno zdravlje Subotica-Public Health Institute	SERBIA
Jugoinspekt Beograd	SERBIA
Veterinary and food institute in Košice	SLOVAKIA
KMETIJSKI INSTITUT SLOVENIJE	SLOVENIA
Jozef Stefan Institute	SLOVENIA
National Laboratory of Health, Environment and Food	SLOVENIA
National Veterinary Institute	SLOVENIA
Laboratorio Regional de Salud Pública Comunidad de Madrid	SPAIN
GOBIERNO DEL PRINCIPADO DE ASTURIAS-CONSEJERÍA DE SANIDAD	SPAIN
Laboratorio Agroalimentario y de Sanidad Animal	SPAIN
ANFACO-CECOPESCA	SPAIN
TROUW NUTRITION ESPAÑA	SPAIN
LABORATORIO ARBITRAL AGROALIMENTARIO	SPAIN
Eurofins Environment	SWEDEN
National Veterinary Institute	SWEDEN
LABORATORIO CANTONALE	SWITZERLAND
MSM (SGS Mersin) Food Control Laboratory	TURKEY
İstanbul Food Control Laboratory	TURKEY
TAYSIDE SCIENTIFIC SERVICES	UNITED KINGDOM
Staffordshire County Council	UNITED KINGDOM
Public Analyst Scientific Services	UNITED KINGDOM
Worcestershire Scientific Services	UNITED KINGDOM
Glasgow Scientific Services	UNITED KINGDOM
Minton, Treharne and Davies Limited	UNITED KINGDOM
Lancashire County Scientific Services	UNITED KINGDOM
Kent County Council	UNITED KINGDOM
EUROFINS FOOD TESTING UK LIMITED	UNITED KINGDOM
The City of Edinburgh Council	UNITED KINGDOM
Food and Environment Research Agency	UNITED KINGDOM
Certified Laboratories	UNITED STATES

10. Abbreviations

AAS	Atomic Absorption Spectroscopy
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV-AAS	Cold Vapour Atomic Absorption Spectrometry
CV-AFS	Cold-Vapour Atomic Fluorescence Spectrometry
EMA	Elemental Mercury Analyser
ETAAS	Electro Thermal Atomic Absorption Spectrometry
EURL-HM	European Union Reference Laboratory for Heavy Metals in Feed and Food
EU	European Union
FAAS	Flame Atomic Absorption Spectroscopy
FI-HGAAS	Flow Injection Hydride-Generation Atomic Absorption Spectrometry
GF-AAS	Graphite Furnace Atomic Absorption Spectroscopy
GUM	Guide to the expression of Uncertainty in Measurement
HG-AAS	Hydride Generation Atomic Absorption Spectroscopy
ICP-MS	Inductively-Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ICP-IDMS	Inductively Coupled Plasma Isotope Dilution Mass Spectrometry
ICP-SFMS	Inductively Coupled Plasma Sector Field Mass Spectrometry
ILC	Interlaboratory Comparison
IMEP	International Measurement Evaluation Programme
IRMM	Institute for Reference Materials and Measurements
ISO	International Organisation for Standardisation
JRC	Joint Research Centre
k_0-INAA	k_0-Instrumental Neutron Activation Analysis
NRL	National Reference Laboratory
OCL	Official Control Laboratories
PT	Proficiency Testing
Q-ICP-MS	Quadrupole Inductively Coupled Plasma Mass Spectroscopy

11. References

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Annexes

Annex 1: Invitation letter to NRLs

Ref. Ares(2014)818858 - 20/03/2014

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
European Union Reference Laboratory for Heavy Metals

Geel, 20 March 2014
JRC.D.5/PRO/FCR/acs/ARES

Subject : IMEP-119: Total As, Cd, Pb and Hg in vegetable feed

Dear National Reference Laboratory representative,

We would like to invite you on behalf of the EURL Heavy Metals in Feed and Food, to participate in the Proficiency Test (PT) IMEP-119 for the **"Determination of total As, Cd, Pb and Hg in vegetable feed"**.

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

Your participation is free of charge.

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

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1201

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

The **deadline for registration is the 24th April 2014**. Samples will be sent to participants during the first half of May 2014. The deadline for submission of results is the **13th June 2014**.

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

Yours sincerely

Dr. Fernando Cordeiro
IMEP-119 Coordinator

Dr. Piotr Robouch
Operating Manager EURL-HM

Cc: Franz Ulberth (Head of Unit SFB)

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

E-mail: JRC-IRMM-EURL-HEAVY-METALS@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

Annex 2: Invitation letter to EA

Ref. Ares(2014)843218 - 21/03/2014

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
European Union Reference Laboratory for Heavy Metals

Mr. Baran Bozoglu
Turkak
Esat Cad. No 41 Küçükesat
06660 ANKARA
TURKEY

IMEP-119: Interlaboratory comparison exercise for the determination of total As, Cd, Pb, and Hg in vegetable feed

Dear Mr. Bozoglu,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"IMEP-119: Determination of total As, Cd, Pb and Hg in vegetable feed"** in support to the Commission Directive 2002/32/EC on undesirable substances in animal feed.

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

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

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

The registration page for laboratories appointed by EA is open until the **24th April 2014**. Distribution of the samples is foreseen for the first half of May 2014. The deadline for submission of results is the **13th June 2014**.
More information about this PT following the link:

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

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

http://irmm.jrc.ec.europa.eu/EURLs/EURL_heavy_metals/interlaboratory_comparisons/Pages/IMEP-119DeterminationoftotalAs,Cd,PbandHginvegetablefeed.aspx

In order to register, laboratories must:

1. **Enter** their details online:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1201

Print the completed form when the system asks to do so.

2. **Clearly indicate on the printed form that they have been appointed by the European Cooperation for Accreditation** to take part in this exercise **otherwise the laboratory will be invoiced 220 € for participation** as charged to the non-appointed laboratories.

3. **Send** the printout to both the IMEP-119 and the EA-IMEP-119 coordinators:

IMEP-119 coordinator	EA-IMEP-119 coordinator
Dr. F. Cordeiro	Mr. Baran Bozoglu
E-mail: jrc-irmm-eurl-heavy-metals@ec.europa.eu	E-mail: bbozoglu@turkak.org.tr

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

With kind regards

Fernando Cordeiro
IMEP-119 Coordinator

2

Annex 3: Invitation letter to APLAC

Ref. Ares(2014)842555 - 21/03/2014

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
European Union Reference Laboratory for Heavy Metals

To: Ms. Cynthia Chen
APLAC PT Committee

IMEP-119: Interlaboratory comparison exercise for the determination of total As, Cd, Pb and Hg in vegetable feed

Dear Ms. Chen,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"IMEP-119: Determination of total As, Cd, Pb and Hg in vegetable feed".**

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

In addition to the 10 laboratories above mentioned, other laboratories may take part in IMEP-119 **paying a registration fee of 220 €.**

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

Registration of participants is open until the **24th April 2014**. Distribution of the samples is foreseen for the first half of May 2014, and the deadline for submission of results is the **13th June 2014**.

More information about this PT following the link:

http://irmm.jrc.ec.europa.eu/EURLs/EURL_heavy_metals/interlaboratory_comparisons/Pages/IMEP-119DeterminationoftotalAs,Cd,PbandHginvegetablefeed.aspx

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

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

In order to register, laboratories must:

1. **Enter** their details online:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1201

2. **Print** the completed form when the system asks to do so.

3. **Clearly indicate on the printed form that they have been appointed by APLAC** to take part in this exercise **otherwise the laboratory will be invoiced 220 € for participation** normally applied for non-appointed laboratories.

4. **Send** the printout to both the IMEP-119 and the APLAC coordinators:

IMEP-119 coordinator Fernando Cordeiro (Ph.D) E-mail: jrc-irmm-eurl-heavy-metals@ec.europa.eu	**APLAC coordinator** Cynthia Chen E-mail: cynthia_chen@taftw.org

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

With kind regards,

Dr. F. Cordeiro

IMEP-119 Coordinator

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Annex 4: Invitation letter to IAAC

Ref. Ares(2014)842665 - 21/03/2014

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
European Union Reference Laboratory for Heavy Metals

To: Barbara Belzer
IAAC Lab Committee

IMEP-119: Interlaboratory comparison exercise for the determination of total As, Cd, Pb and Hg in vegetable feed

Dear Mrs. Belzer,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"IMEP-119: Determination of total As, Cd, Pb and Hg in vegetable feed".**

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

In addition to the 10 laboratories above mentioned, other laboratories may take part in IMEP-119 paying a registration fee of 220 €.

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

Registration of participants is open until the **24th April 2014**. Distribution of the samples is foreseen for the first half of May 2014, and the deadline for submission of results is the **13th June 2014**.
More information about this PT following the link:

http://irmm.jrc.ec.europa.eu/EURLs/EURL_heavy_metals/interlaboratory_comparisons/Pages/IMEP-119DeterminationoftotalAs,Cd,PbandHginvegetablefeed.aspx

In order to register, laboratories must:

1. **Enter** their details online:

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

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

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1201

2. **Print** the completed form when the system asks to do so.

3. **Clearly indicate on the printed form that they have been appointed by IAAC** to take part in this exercise **otherwise the laboratory will be invoiced 220 € for participation** normally applied for non-appointed laboratories.

4. **Send** the printout to both the IMEP-119 and the IAAC coordinators:

IMEP-119 coordinator	**IAAC coordinator**
Fernando Cordeiro (*Ph.D*)	Barbara Belzer
E-mail: jrc-irmm-eurl-heavy-metals@ec.europa.eu	E-mail: barbara.belzer@nist.gov

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

With kind regards

Dr. F. Cordeiro

IMEP-119 Coordinator

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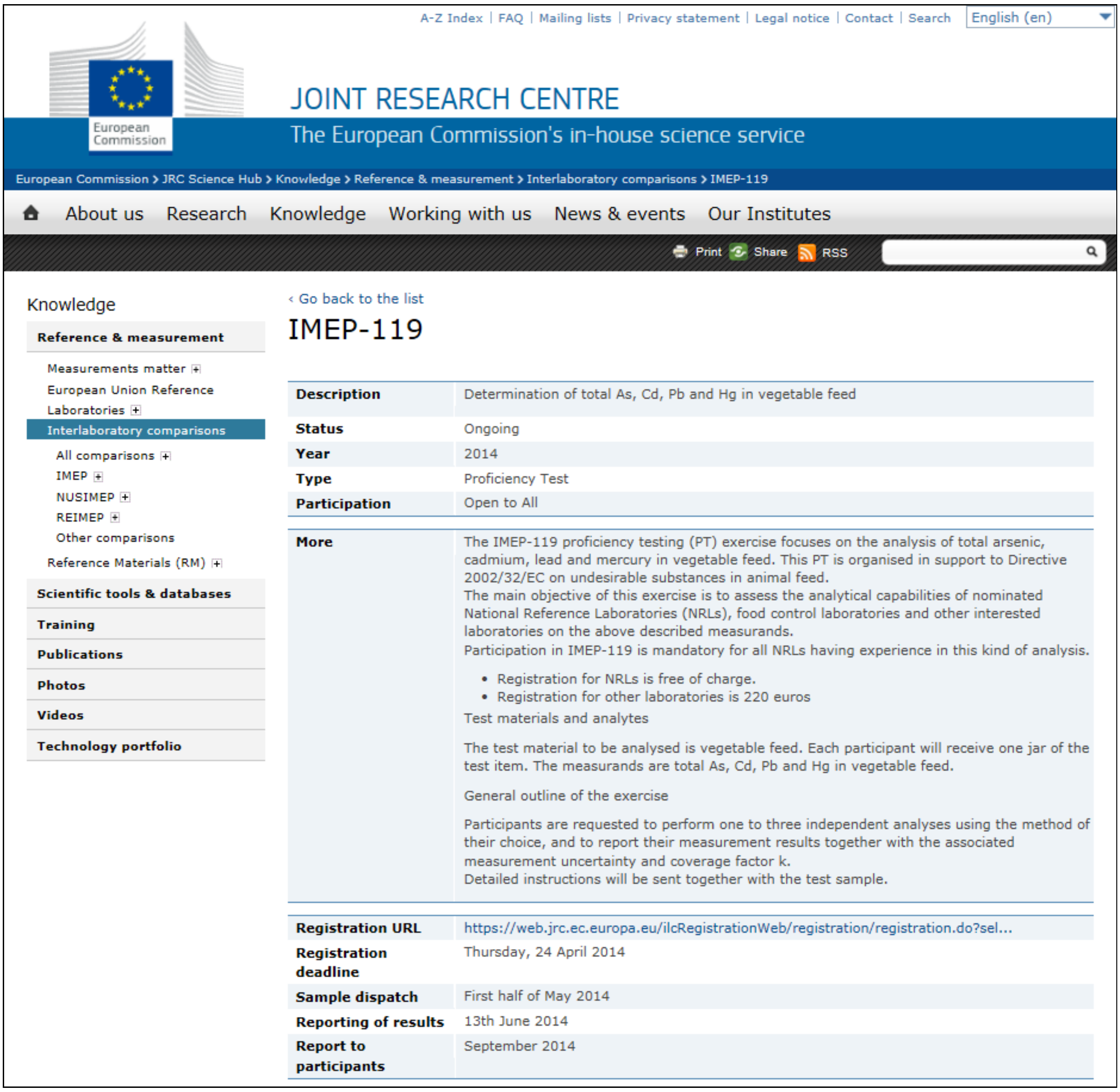
Annex 5: JRC web announcement



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IMEP-119

Description	Determination of total As, Cd, Pb and Hg in vegetable feed
Status	Ongoing
Year	2014
Type	Proficiency Test
Participation	Open to All

More

The IMEP-119 proficiency testing (PT) exercise focuses on the analysis of total arsenic, cadmium, lead and mercury in vegetable feed. This PT is organised in support to Directive 2002/32/EC on undesirable substances in animal feed.
The main objective of this exercise is to assess the analytical capabilities of nominated National Reference Laboratories (NRLs), food control laboratories and other interested laboratories on the above described measurands.
Participation in IMEP-119 is mandatory for all NRLs having experience in this kind of analysis.

- Registration for NRLs is free of charge.
- Registration for other laboratories is 220 euros

Test materials and analytes

The test material to be analysed is vegetable feed. Each participant will receive one jar of the test item. The measurands are total As, Cd, Pb and Hg in vegetable feed.

General outline of the exercise

Participants are requested to perform one to three independent analyses using the method of their choice, and to report their measurement results together with the associated measurement uncertainty and coverage factor k.
Detailed instructions will be sent together with the test sample.

Registration URL	https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?sel...
Registration deadline	Thursday, 24 April 2014
Sample dispatch	First half of May 2014
Reporting of results	13th June 2014
Report to participants	September 2014

Annex 6: Sample accompanying letter

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
International Measurement Evaluation Program

Geel, 28 April 2014
JRC.D5/IF/acs/Ares(2014)1323930

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

Participation in IMEP-119, a proficiency test exercise for the determination of total arsenic (As), cadmium (Cd), lead (Pb) and mercury (Hg) in vegetable feed.

Dear «Title» «Surname»,

Thank you for participating in the IMEP-119 proficiency test for the determination of total As, Cd, Pb and Hg in vegetable feed. This proficiency test (PT) exercise is organised in support to Directive 2002/32/EC on undesirable substances in animal feed.

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

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

Please check whether the bottle containing the test item remained undamaged during transport. Then, send the "Confirmation of receipt" form back (fax: +32-14-571865, e-mail: JRC-IRMM-IMEP@ec.europa.eu). You should store the sample in a dark place at 4°C until analysis.

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

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

Reporting of results

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

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

E-mail: JRC-IRMM-IMEP@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

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

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

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

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

Do not forget to submit and confirm always when required.

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

The **deadline** for submission of results is **13/06/2014**.

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-IMEP@ec.europa.eu

With kind regards,

Fernando Cordeiro (Ph.D.)
IMEP-119 Coordinator

Cc: F. Ulberth (SFB HoU)

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

E-mail: JRC-IRMM-IMEP@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

Annex 7: Confirmation of receipt form

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
International Measurement Evaluation Program

Annex to
JRC.D5/IF/acs/ARES(2014)1323930

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

IMEP-119

Total arsenic (As), cadmium (Cd), lead (Pb) and mercury (Hg) in vegetable 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:

Fernando Cordeiro (Ph.D.)

IMEP-119 Coordinator
EC-JRC-IRMM
Retieseweg 111
B-2440 GEEL, Belgium

Fax : +32-14-571865
JRC-IRMM-IMEP@ec.europa.eu

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

E-mail: JRC-IRMM-IMEP@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

Annex 8: Questionnaire

Submission Form

1. **Are you a National Reference Laboratory (NRL)?**

- a) Yes
- b) No

1.1. If "No" have you been nominated by your National Accreditation Body (NAB) or by your NRL?

- a) Yes
- b) No

1.1.1. If "Yes" please identify NAB or NRL.

2. **Are you accredited for this type of matrix/analyte?**

Questions/Response table	Total As	Total Cd	Total Pb	Total Sn	Info
Accredited for:	☐	☐	☐	☐	

3. **How did you estimate the recovery?**

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

3.1. If "Other" please specify

3.2. Please provide the estimated analytical recovery (%) and the LODs of your methods

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

Questions/Response table	Total As	Total Cd	Total Pb	Total Hg
Recovery %				
LOD (mg/kg)				

4. **Did you use a (certified) reference material for method validation or for instrument calibration? Which one?**

(Certified) reference materials:

Questions/Response table	Total As	Total Cd	Total Pb	Total Hg
Validation of measurement procedure				
Instrument calibration				

5. **Which type of sample digestion did you use?**

Questions/Response table	Closed microwave	Dry ashing	Open microwave	Open wet	Pressure bomb	Info
Total As	☐	☐	☐	☐	☐	
Total Cd	☐	☐	☐	☐	☐	
Total Pb	☐	☐	☐	☐	☐	
Total Hg	☐	☐	☐	☐	☐	

6. **Which type of digestion mixture did you use? (multiple selections are possible)**

Questions/Response table	H2O2	H2SO4	HCL	HCLO4	HF	HNO3	Other	Info
Total As	☐	☐	☐	☐	☐	☐	☐	
Total Cd	☐	☐	☐	☐	☐	☐	☐	
Total Pb	☐	☐	☐	☐	☐	☐	☐	
Total Hg	☐	☐	☐	☐	☐	☐	☐	

7. **If "Other" please specify.**

8. **Does your laboratory carry out this type of analysis on a regular basis? (samples per year)**

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

9. **Additional remarks/comments regarding the method of analysis?**

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

- a) Yes
- b) No

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

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

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

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

11.1. If "Other" please specify.

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

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

- a) Yes
- b) No

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

- a) Yes
- b) No

14.1. If "Yes", which:

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

14.1.1. If "Other" please specify.

15. **Considering the reported level for the investigated trace elements in the feed matrix and the maximum levels of undesirable**

- a) Yes
- b) No

15.1. If "No" please explain why

16. **Does your laboratory take part in interlaboratory comparisons (ILCs) for this type of analysis?**

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

Which official method?

Questions/Response table
Which official method did you followed?

	Total As	Total Cd	Total Pb	Total Hg

Annex 9: Homogeneity and stability studies (all values in mg kg^{-1})

9.1 Homogeneity studies

	As		Cd		Pb		Hg	
Bottle ID	***R₁***	***R₂***	***R₁***	***R₂***	***R₁***	***R₂***	***R₁***	***R₂***
12	1.11	1.09	0.119	0.114	3.11	3.19	0.0069	0.0077
23	1.12	1.09	0.118	0.113	3.26	3.18	0.0063	0.0073
48	1.13	1.04	0.112	0.116	3.11	3.21	0.0068	0.0065
76	1.05	1.09	0.115	0.111	3.22	3.17	0.0067	0.0064
93	1.04	1.03	0.118	0.119	3.31	3.30	0.0064	0.0071
110	1.08	1.13	0.117	0.121	3.25	3.29	0.0065	0.0063
134	1.01	1.10	0.141	0.116	3.33	3.29	0.0065	0.0065
158	1.05	1.08	0.115	0.119	3.31	3.25	0.0079	0.0076
172	1.15	1.00	0.115	0.119	3.26	3.27	0.0081	0.0069
186	1.02	1.06	0.120	0.124	3.25	3.41	0.0068	0.0068
Mean	**1.07**		**0.118**		**3.25**		**0.0069**	
σ	**0.18**		**0.019**		**0.48**		**0.0017**	
0.3*σ	**0.05**		**0.006**		**0.14**		**0.0005**	
s_x	**0.026**		**0.004**		**0.06**		**0.0005**	
s_w	**0.048**		**0.006**		**0.05**		**0.0004**	
s_s	**0.0000**		**0.001**		**0.05**		**0.0003**	
$s_s \leq 0.3*\sigma$	**Pass**		**Pass**		**Pass**		**Pass**	

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

9.2 Stability studies (at 18 °C)

	Time in Weeks				u_{st}
As	0	3	5	8	
	1.09	1.08	1.12	1.10	
	1.04	1.07	1.07	1.16	**2.3%**
Cd	0	3	5	8	
	0.115	0.118	0.109	0.113	
	0.117	0.112	0.115	0.113	**1.8%**
Pb	0	3	5	8	
	3.43	3.23	3.13	3.19	
	3.17	3.19	3.21	3.21	**2.0%**
Hg	0	3	5	8	
	0.0062	0.0065	0.0064	0.0070	
	0.0065	0.0070	0.0062	0.0064	**3.5%**

Where: u_{st} is the standard measurement uncertainty due to stability (6 weeks, expressed as a %)

Annex 10: Results for total As

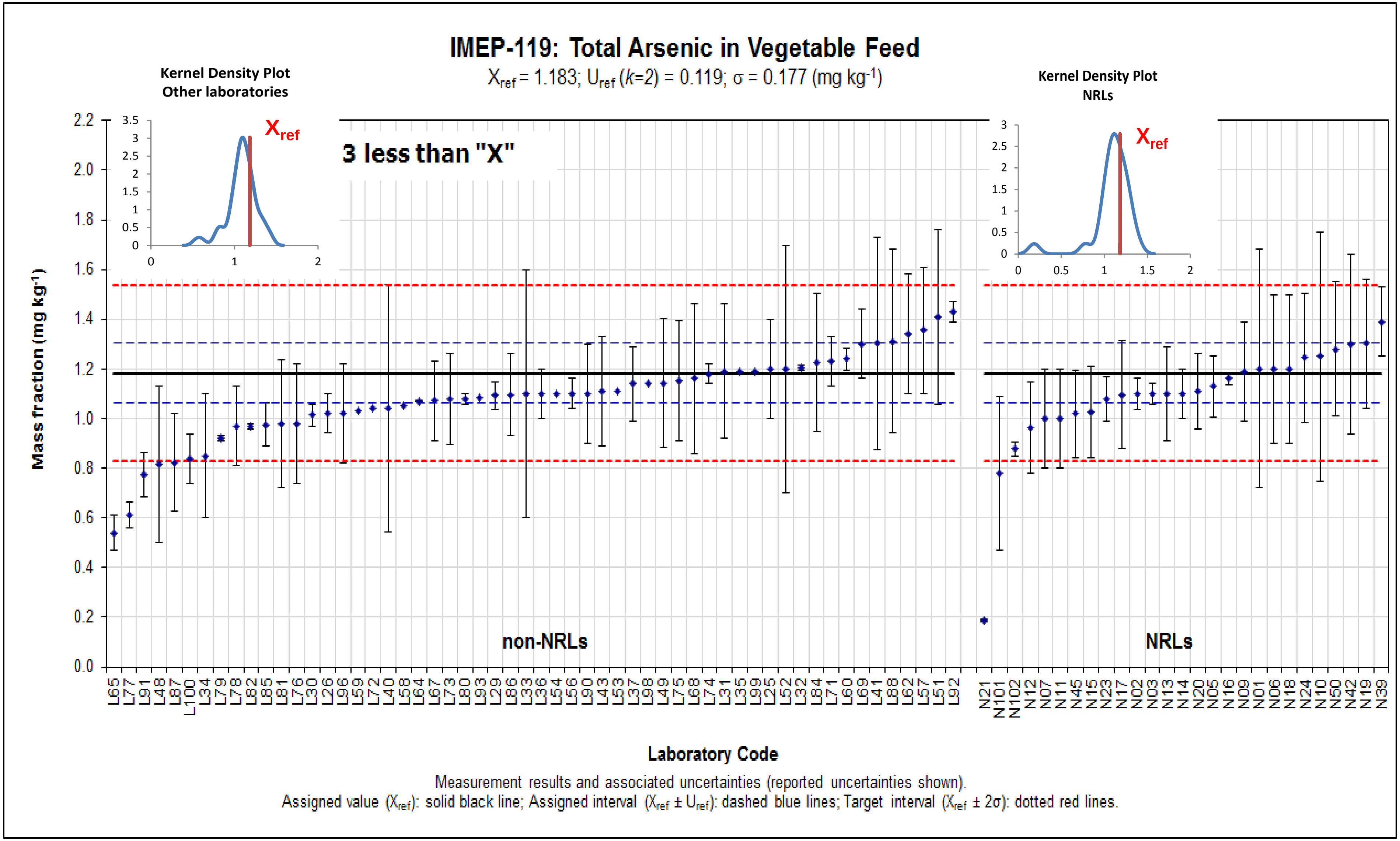
Assigned range: X_{ref} = 1.183; U_{ref} (k=2) = 0.119; σ = 0.177 (all values in mg kg^{-1})

Lab Code	X_{lab}	±	k [a]	Technique	u_{lab}	z-score [b]	ζ-score [b]	unc. [c]
L25	1.2	0.2	100	HG-AAS	0.002	0.10	0.29	b
L26	1.02	0.08	2	ICP-IDMS	0.04	-0.92	-2.26	b
L29	1.093	0.056	2	ICP-IDMS	0.028	-0.50	-1.36	b
L30	1.014	0.046	2	HG-AAS	0.023	-0.95	-2.63	b
L31	1.19	0.27	2	ICP-MS	0.135	0.04	0.05	a
L32	1.205	0.01	√3	ICP-MS	0.005774	0.13	0.38	b
L33	1.1	0.5	2		0.25	-0.47	-0.32	c
L34	0.85	0.25	2	FAAS	0.125	-1.87	-2.40	a
L35	1.19		√3	HG-AAS	0.00	0.04	0.13	b
L36	1.1	0.1	2	ICP-MS	0.05	-0.47	-1.06	b
L37	1.14	0.15	2	ICP-MS	0.075	-0.24	-0.44	a
L40	1.043	0.5	3	HG-AAS	0.166667	-0.79	-0.79	a
L41	1.302	0.43	2	SFICP-MS	0.215	0.67	0.54	c
L43	1.11	0.22	2	ICP-IDMS	0.11	-0.41	-0.58	a
L46	< 2.5			ICP-OES				
L48	0.817	0.316	2		0.158	-2.06	-2.16	a
L49	1.142	0.26	2	SFICP-MS	0.13	-0.23	-0.28	a
L51	1.41	0.35	2	ICP-OES	0.175	1.28	1.23	a
L52	1.2	0.5	2	FAAS	0.25	0.10	0.07	c
L53	1.11			HG-AAS	0.00	-0.41	-1.21	b
L54	1.1		2	ICP-IDMS	0.00	-0.47	-1.38	b
L56	1.1	0.06	2		0.03	-0.47	-1.24	b
L57	1.355	0.254	2	ICP-IDMS	0.13	0.97	1.23	a
L58	1.05			SFICP-MS	0.00	-0.75	-2.22	b
L59	1.029			HG-AAS	0.00	-0.87	-2.57	b
L60	1.24	0.045	2	ICP-IDMS	0.02	0.32	0.90	b
L62	1.34	0.24	2	ICP-MS	0.12	0.89	1.18	a
L64	1.07	0.002	2	ICP-OES	0.00	-0.63	-1.88	b
L65	0.54	0.0696	2	AAS	0.0348	-3.62	-9.30	b
L67	1.071	0.16	2	ICP-IDMS	0.08	-0.63	-1.12	a
L68	1.16	0.3	2	ICP-OES	0.15	-0.13	-0.14	a
L69	1.3	0.14	2	ETAAS	0.07	0.66	1.28	a
L70	< 1.6			ICP-OES				
L71	1.23	0.1	2	HG-AAS	0.05	0.27	0.61	b
L72	1.04			ICP-OES	0.00	-0.80	-2.39	b
L73	1.08	0.184	2	HG-AAS	0.092	-0.58	-0.93	a
L74	1.18	0.04	1	k0-INAA	0.04	-0.01	-0.03	b
L75	1.152	0.24	2	GF AAS	0.12	-0.17	-0.23	a
L76	0.98	0.24	2	ICP-MS	0.12	-1.14	-1.51	a
L77	0.61	0.052	2	ETAAS	0.026	-3.23	-8.80	b
L78	0.97	0.16	2	ETAAS	0.08	-1.20	-2.13	a
L79	0.92	0.01	2	ICP-MS	0.005	-1.48	-4.38	b
L80	1.08	0.02	2	ICP-MS	0.01	-0.58	-1.69	b
L81	0.979	0.257	√3	SFICP-MS	0.148	-1.15	-1.27	a
L82	0.97	0.01	2	HG-AAS	0.005	-1.20	-3.55	b
L84	1.224	0.2792	2	ICP-MS	0.1396	0.23	0.27	a
L85	0.976	0.088	2	HG ICP OES	0.044	-1.16	-2.79	b
L86	1.096	0.164	√3	SFICP-MS	0.095	-0.49	-0.77	a
L87	0.822	0.197	2	HG-AAS	0.0985	-2.03	-3.13	a
L88	1.31	0.37	2	SFICP-MS	0.185	0.72	0.66	c
L89	< 0.01			HG-AAS				
L90	1.1	0.2	2	ICP-IDMS	0.1	-0.47	-0.71	a
L91	0.774	0.09	√3	ETAAS	0.052	-2.30	-5.16	b
L92	1.43	0.043	2	ICP-IDMS	0.0215	1.40	3.90	b
L93	1.086			ICP-MS	0.00	-0.54	-1.62	b
L96	1.02	0.2	2	ICP-MS	0.1	-0.92	-1.40	a
L98	1.14			AAS	0.00	-0.24	-0.71	b
L99	1.19			ICP-OES	0.00	0.04	0.13	b
L100	0.836	0.1	2	ICP-IDMS	0.05	-1.95	-4.45	b
N01	1.2	0.48	2		0.24	0.10	0.07	c
N02	1.1	0.062	2	SFICP-MS	0.031	-0.47	-1.23	b
N03	1.1	0.04	2	ETAAS	0.02	-0.47	-1.31	b
N05	1.13	0.124	2	Q-ICP-MS	0.062	-0.30	-0.61	a
N06	1.2	0.3	2	ICP-OES	0.15	0.10	0.11	a
N07	1.0	0.2	2	ICP-MS	0.1	-1.03	-1.57	a
N09	1.19	0.2	2	ICP-MS	0.1	0.04	0.06	a
N10	1.25	0.5	2	ICP-MS	0.25	0.38	0.26	c
N11	1.0	0.2	2	SFICP-MS	0.1	-1.03	-1.57	a
N12	0.964	0.1831	√3	ICP-MS	0.106	-1.23	-1.80	a
N13	1.1	0.19	2	ICP-MS	0.095	-0.47	-0.74	a
N14	1.1	0.1	2	ETAAS	0.05	-0.47	-1.06	b
N15	1.028	0.184	2	SFICP-MS	0.092	-0.87	-1.41	a
N16	1.16	0.026	2	HG-AAS	0.013	-0.13	-0.37	b
N17	1.0956	0.218	2	SFICP-MS	0.109	-0.49	-0.70	a
N18	1.2	0.3	2	ICP-MS	0.15	0.10	0.11	a
N19	1.304	0.26	√3	ICP-MS	0.150	0.68	0.75	a
N20	1.11	0.15	2	SFICP-MS	0.075	-0.41	-0.76	a
N21	0.186	0.006	2		0.003	-5.62	-16.68	b
N23	1.08	0.09	2	ICP-MS	0.045	-0.58	-1.37	b
N24	1.244	0.261	2	HG-AAS	0.131	0.35	0.43	a
N39	1.39	0.14	2	ICP-MS	0.07	1.17	2.26	a
N42	1.3	0.362	2	HG-AAS	0.181	0.66	0.62	c
N45	1.019	0.174	2	HG-AAS	0.087	-0.92	-1.55	a
N50	1.28	0.27	2	ICP-MS	0.135	0.55	0.66	a
N101	0.78	0.31	2	ETAAS	0.155	-2.27	-2.42	a
N102	0.878	0.028	3		0.009	-1.72	-5.04	b

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

[b] performance: satisfactory, questionable, unsatisfactory,

[c] a : u_{min} (u_{ref}) ≤ u_{lab} ≤ u_{max} (σ); b : u_{lab} < u_{min}; and c : u_{lab} > u_{max}


IMEP-119: Total Arsenic in Vegetable Feed
X_ref = 1.183; U_ref (k=2) = 0.119; σ = 0.177 (mg kg-1)
Kernel Density Plot
Other laboratories
X_ref
3 less than "X"
Kernel Density Plot
NRLs
X_ref
Mass fraction (mg kg-1)
non-NRLs
NRLs
L65 L77 L91 L48 L87 L100 L34 L79 L78 L82 L85 L81 L76 L30 L26 L96 L59 L72 L40 L58 L64 L67 L73 L80 L93 L29 L86 L33 L36 L54 L56 L90 L43 L53 L37 L98 L49 L75 L68 L74 L31 L35 L99 L25 L52 L32 L84 L71 L60 L69 L41 L88 L62 L57 L51 L92
N21 N101 N102 N12 N07 N11 N45 N15 N23 N17 N02 N03 N13 N14 N20 N05 N16 N09 N01 N06 N18 N24 N10 N50 N42 N19 N39
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Assigned value (X_ref): solid black line; Assigned interval (X_ref ± U_ref): dashed blue lines; Target interval (X_ref ± 2σ): dotted red lines.

Annex 11: Results for total Cd

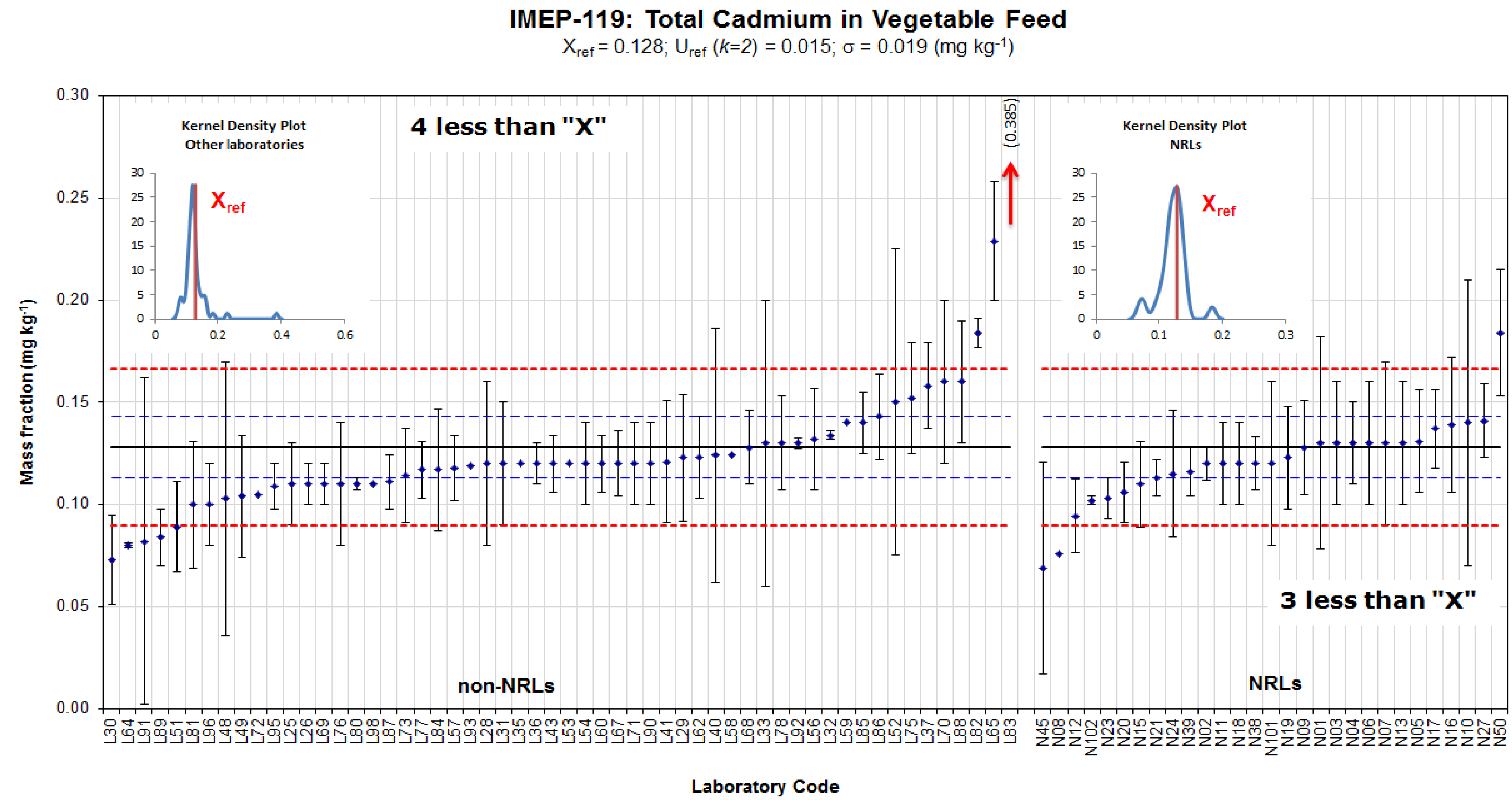
Assigned range: X_{ref} = 0.128; U_{ref} (k=2) = 0.015; σ = 0.019 (all values in mg kg^{-1})

Lab Code	X_{lab}	±	k [a]	Technique	u_{lab}	z-score [b]	ζ-score [b]	unc. [c]
L25	0.11	0.02	100	ICP-MS	0.000	-0.94	-2.39	b
L26	0.11	0.01	2	ICP-IDMS	0.005	-0.94	-1.99	b
L28	0.12	0.04	2	AAS	0.020	-0.42	-0.37	c
L29	0.123	0.031	2	ICP-IDMS	0.016	-0.26	-0.29	a
L30	0.073	0.022	2	ETAAS	0.011	-2.86	-4.13	a
L31	0.12	0.03	2	ICPMS	0.015	-0.42	-0.48	a
L32	0.134	0.002	√3	ICP-MS	0.001	0.31	0.79	b
L33	0.13	0.07	2	ICP-MS	0.035	0.10	0.06	c
L34	< 0.13			AAS				
L35	0.12			ETAAS	0.000	-0.42	-1.06	b
L36	0.12	0.01	2	ICP-MS	0.005	-0.42	-0.89	b
L37	0.158	0.021	2	ICP-MS	0.011	1.56	2.32	a
L40	0.124	0.062	3	ETAAS	0.021	-0.21	-0.18	c
L41	0.121	0.03	2	SFICP-MS	0.015	-0.36	-0.42	a
L43	0.12	0.014	2	ICP-IDMS	0.007	-0.42	-0.78	b
L48	0.103	0.067	2	ICP-OES	0.034	-1.30	-0.73	c
L49	0.104	0.03	2	SFICP-MS	0.015	-1.25	-1.43	a
L51	0.089	0.022	2	ICP-OES	0.011	-2.03	-2.93	a
L52	0.15	0.075	2	ICP-MS	0.038	1.15	0.58	c
L53	0.12			ETAAS	0.000	-0.42	-1.06	b
L54	0.12	0.02	2	ICP-IDMS	0.010	-0.42	-0.64	a
L56	0.132	0.025	2	ICPMS	0.013	0.21	0.27	a
L57	0.118	0.016	2	ETAAS	0.008	-0.52	-0.91	a
L58	0.124			SFICP-MS	0.000	-0.21	-0.53	b
L59	0.14			AAS	0.000	0.63	1.60	b
L60	0.12	0.014	2	ICP-IDMS	0.007	-0.42	-0.78	b
L62	0.123	0.02	2	ICP-MS	0.010	-0.26	-0.40	a
L64	0.08	0.001	2	ICP-OES	0.001	-2.50	-6.37	b
L65	0.229	0.0293	2	ICP-OES	0.015	5.26	6.13	a
L67	0.12	0.016	2	ICP-IDMS	0.008	-0.42	-0.73	a
L68	0.128	0.018	2	ICP-OES	0.009	0.00	0.00	a
L69	0.11	0.01	2	ETAAS	0.005	-0.94	-1.99	b
L70	0.16	0.04	2	AAS	0.020	1.67	1.50	c
L71	0.12	0.02	2	AAS	0.010	-0.42	-0.64	a
L72	0.105			AAS	0.000	-1.20	-3.06	b
L73	0.114	0.023	2	ETAAS	0.012	-0.73	-1.02	a
L75	0.152	0.027	2	ETAAS	0.014	1.25	1.55	a
L76	0.11	0.03	2	ICP-MS	0.015	-0.94	-1.07	a
L77	0.117	0.014	2	ETAAS	0.007	-0.57	-1.07	b
L78	0.13	0.023	2	ETAAS	0.012	0.10	0.15	a
L79	< 0.25		2	ICP-OES				
L80	0.11	0.003	2	ICP-MS	0.002	-0.94	-2.35	b
L81	0.1	0.031	√3	SFICP-MS	0.018	-1.46	-1.44	a
L82	0.184	0.007	2	AAS	0.004	2.92	6.75	b
L83	0.385			ICP-OES	0.000	13.39	34.17	b
L84	0.117	0.0298	2	ICP-MS	0.015	-0.57	-0.66	a
L85	0.14	0.015	2	ICP-OES	0.008	0.63	1.13	b
L86	0.143	0.021	√3	SFICP-MS	0.012	0.78	1.05	a
L87	0.111	0.013	2	ETAAS	0.007	-0.89	-1.71	b
L88	0.16	0.03	2	SFICP-MS	0.015	1.67	1.91	a
L89	0.084	0.014	2	ETAAS	0.007	-2.29	-4.28	b
L90	0.12	0.02	2	ICP-OES	0.010	-0.42	-0.64	a
L91	0.082	0.08	√3	ETAAS	0.046	-2.40	-0.98	c
L92	0.13	0.0026	2	ICP-IDMS	0.001	0.10	0.26	b
L93	0.119			ICP-MS	0.000	-0.47	-1.20	b
L95	0.109	0.011	√3	AAS	0.006	-0.99	-1.93	b
L96	0.1	0.02	2	ICP-MS	0.010	-1.46	-2.24	a
L98	0.11			AAS	0.000	-0.94	-2.39	b
L99	< 0.05			ICP-OES				
L100	< 0.005			ICP-IDMS				
N01	0.13	0.052	2		0.026	0.10	0.07	c
N02	0.12	0.0084	2	SFICP-MS	0.004	-0.42	-0.93	b
N03	0.13	0.03	2	ETAAS	0.015	0.10	0.12	a
N04	0.13	0.02	2	Q-ICP-MS	0.010	0.10	0.16	a
N05	0.131	0.025	2	Q-ICP-MS	0.013	0.16	0.21	a
N06	0.13	0.03	2	ICP-OES	0.015	0.10	0.12	a
N07	0.13	0.04	2	ICP-MS	0.020	0.10	0.09	c
N08	0.076				0.000	-2.71	-6.91	b
N09	0.128	0.023	2	ICP-MS	0.012	0.00	0.00	a
N10	0.14	0.07	2	ICP-MS	0.035	0.63	0.34	c
N11	0.12	0.02	2	SFICP-MS	0.010	-0.42	-0.64	a
N12	0.0944	0.01793	√3	ICP-MS	0.010	-1.75	-2.63	a
N13	0.13	0.03	2	ICP-MS	0.015	0.10	0.12	a
N14	< 0.25			ETAAS				
N15	0.11	0.021	2	SFICP-MS	0.011	-0.94	-1.39	a
N16	0.139	0.033	2	ETAAS	0.017	0.57	0.61	a
N17	0.137	0.0191	2	SFICP-MS	0.010	0.47	0.74	a
N18	0.12	0.02	2	ICP-MS	0.010	-0.42	-0.64	a
N19	0.123	0.025	√3	ICP-MS	0.014	-0.26	-0.31	a
N20	0.106	0.015	2	SFICP-MS	0.008	-1.15	-2.07	b
N21	0.113	0.009	2		0.005	-0.78	-1.71	b
N22	< 0.5			AAS				
N23	0.103	0.01	2	ICP-MS	0.005	-1.30	-2.77	b
N24	0.115	0.031	2	GETAAS	0.016	-0.68	-0.75	a
N27	0.141	0.018	2	AAS	0.009	0.68	1.11	a
N38	0.12	0.013	2	AAS	0.007	-0.42	-0.80	b
N39	0.116	0.012	2	ICP-MS	0.006	-0.63	-1.25	b
N42	< 0.15			FAAS				
N45	0.069	0.052	2	ETAAS	0.026	-3.07	-2.18	c
N50	0.184	0.031	2	ICP-MS	0.016	2.92	3.25	a
N101	0.12	0.04	2	ETAAS	0.020	-0.42	-0.37	c
N102	0.102	0.002	3		0.001	-1.35	-3.44	b

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

[b] performance: satisfactory, questionable, unsatisfactory,

[c] a : u_{min} (u_{ref}) ≤ u_{lab} ≤ u_{max} (σ); b : u_{lab} < u_{min}; and c : u_{lab} > u_{max}



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

Annex 12: Results for total Pb

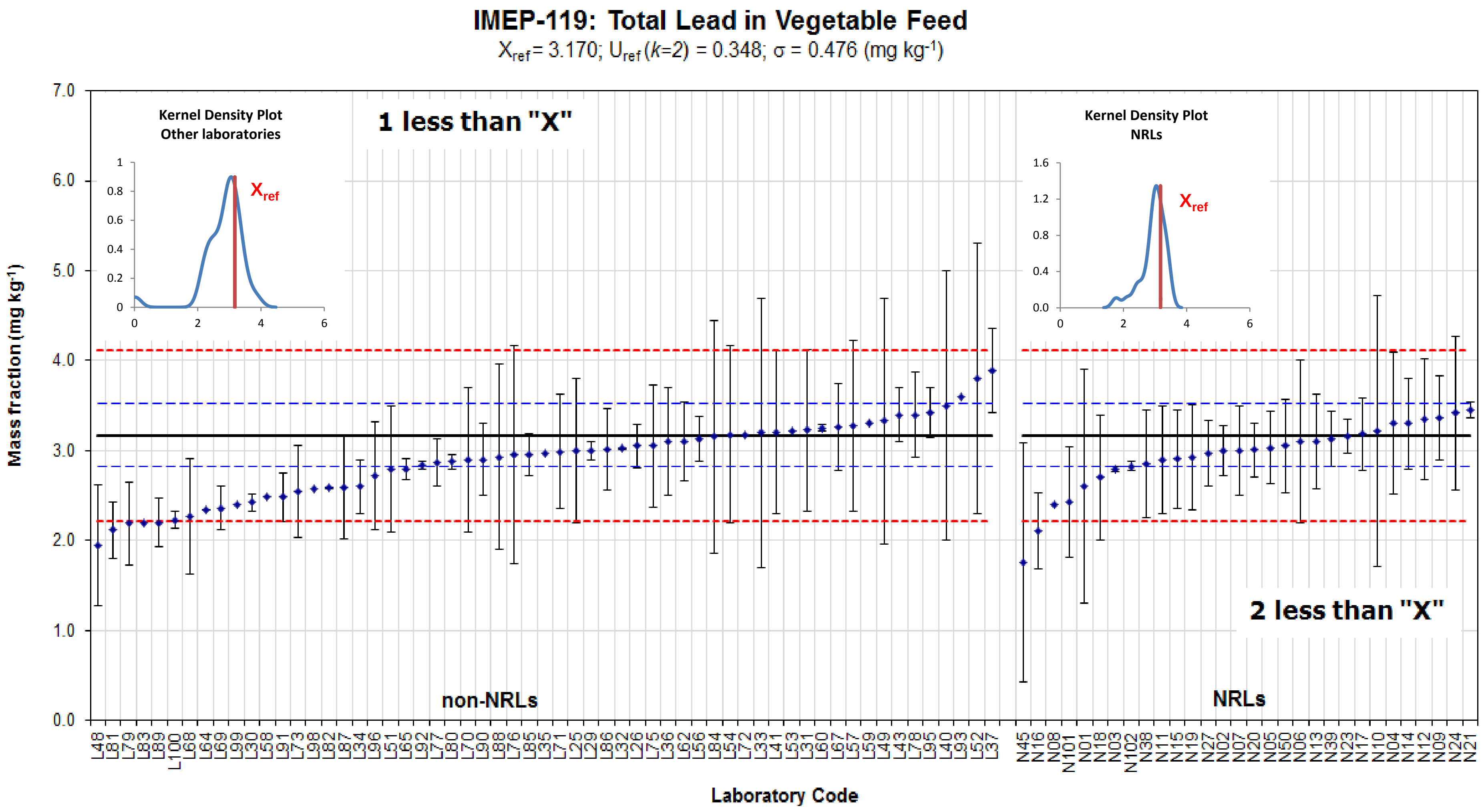
Assigned range: X_{ref} = 3.170; U_{ref} ($k=2$) = 0.348; σ = 0.476 (all values in mg kg^{-1})

Lab Code	X_{lab}	±	k [a]	Technique	u_{lab}	z-score [b]	ζ-score [b]	unc. [c]
L25	3.0	0.8	100	ICP-MS	0.008	-0.36	-0.98	b
L26	3.05	0.24	2	ICP-IDMS	0.120	-0.25	-0.57	b
L29	3.001	0.105	2	ICP-IDMS	0.053	-0.36	-0.93	b
L30	2.422	0.089	2	ETAAS	0.045	-1.57	-4.17	b
L31	3.23	0.9	2	ICPMS	0.450	0.13	0.12	a
L32	3.021	0.009	√3	ICP-MS	0.005	-0.31	-0.86	b
L33	3.2	1.5	2	ICP-MS	0.750	0.06	0.04	c
L34	2.6	0.3	2	AAS	0.150	-1.20	-2.48	b
L35	2.97			ETAAS	0.000	-0.42	-1.15	b
L36	3.1	0.6	2	ICP-MS	0.300	-0.15	-0.20	a
L37	3.89	0.47	2	ICP-MS	0.235	1.51	2.46	a
L40	3.5	1.5	3	ETAAS	0.500	0.69	0.62	c
L41	3.204	0.906	2	SFICP-MS	0.453	0.07	0.07	a
L43	3.4	0.306	2	ICP-IDMS	0.153	0.48	0.99	b
L46	< 2.5			ICP-OES				
L48	1.95	0.671	2	ICP-OES	0.336	-2.57	-3.23	a
L49	3.33	1.37	2	SFICP-MS	0.685	0.34	0.23	c
L51	2.79	0.7	2	ICP-OES	0.350	-0.80	-0.97	a
L52	3.8	1.5	2	ICP-MS	0.750	1.32	0.82	c
L53	3.22			ETAAS	0.000	0.11	0.29	b
L54	3.18	0.98	2	ICP-IDMS	0.490	0.02	0.02	c
L56	3.13	0.25	2	ICPMS	0.125	-0.08	-0.19	b
L57	3.27	0.95	2	ETAAS	0.475	0.21	0.20	a
L58	2.48			SFICP-MS	0.000	-1.45	-3.97	b
L59	3.3			AAS	0.000	0.27	0.75	b
L60	3.25	0.037	2	ICP-IDMS	0.019	0.17	0.46	b
L62	3.1	0.44	2	ICP-MS	0.220	-0.15	-0.25	a
L64	2.34	0.001	2	ICP-OES	0.001	-1.75	-4.77	b
L65	2.793	0.1168	2	AAS	0.058	-0.79	-2.05	b
L67	3.26	0.48	2	ICP-IDMS	0.240	0.19	0.30	a
L68	2.27	0.64	2	ICP-OES	0.320	-1.89	-2.47	a
L69	2.36	0.24	2	ETAAS	0.120	-1.70	-3.83	b
L70	2.9	0.8	2	AAS	0.400	-0.57	-0.62	a
L71	2.99	0.63	2	AAS	0.315	-0.38	-0.50	a
L72	3.18			AAS	0.000	0.02	0.06	b
L73	2.55	0.51	2	ICP-OES	0.255	-1.30	-2.01	a
L75	3.05	0.674	2	GF AAS	0.337	-0.25	-0.32	a
L76	2.95	1.21	2	ICP-MS	0.605	-0.46	-0.35	c
L77	2.867	0.269	2	ETAAS	0.135	-0.64	-1.38	b
L78	3.4	0.47	2	ETAAS	0.235	0.48	0.79	a
L79	2.19	0.46	2	ICP-MS	0.230	-2.06	-3.40	a
L80	2.88	0.08	2	ICP-MS	0.040	-0.61	-1.62	b
L81	2.117	0.309	√3	SFICP-MS	0.178	-2.21	-4.23	a
L82	2.584	0.005	2	AAS	0.003	-1.23	-3.37	b
L83	2.1918			ICP-OES	0.000	-2.06	-5.62	b
L84	3.152	1.2989	2	ICP-MS	0.649	-0.04	-0.03	c
L85	2.952	0.236	2	ICP-OES	0.118	-0.46	-1.04	b
L86	3.015	0.452	√3	SFICP-MS	0.261	-0.33	-0.49	a
L87	2.585	0.569	2	ETAAS	0.285	-1.23	-1.75	a
L88	2.93	1.03	2	SFICP-MS	0.515	-0.50	-0.44	c
L89	2.2	0.27	2	ETAAS	0.135	-2.04	-4.40	b
L90	2.9	0.4	2	ICP-OES	0.200	-0.57	-1.02	a
L91	2.485	0.27	√3	ETAAS	0.156	-1.44	-2.93	b
L92	2.84	0.043	2	CV-AFS	0.022	-0.69	-1.88	b
L93	3.601			ICP-MS	0.000	0.91	2.48	b
L95	3.42	0.28	√3	AAS	0.162	0.53	1.05	b
L96	2.72	0.6	2	ICP-MS	0.300	-0.95	-1.30	a
L98	2.58			AAS	0.000	-1.24	-3.39	b
L99	2.4			ICP-OES	0.000	-1.62	-4.43	b
L100	2.231	0.1	2	ICP-IDMS	0.050	-1.97	-5.19	b
N01	2.6	1.3	2		0.650	-1.20	-0.85	c
N02	3.0	0.28	2	SFICP-MS	0.140	-0.36	-0.76	b
N03	2.8	0.03	2	ETAAS	0.015	-0.78	-2.12	b
N04	3.3	0.79	2	Q-ICP-MS	0.395	0.27	0.30	a
N05	3.032	0.403	2	Q-ICP-MS	0.202	-0.29	-0.52	a
N06	3.1	0.9	2	ICP-OES	0.450	-0.15	-0.15	a
N07	3.0	0.5	2	ICP-MS	0.250	-0.36	-0.56	a
N08	2.4				0.000	-1.62	-4.43	b
N09	3.36	0.47	2	ICP-MS	0.235	0.40	0.65	a
N10	3.22	1.5	2	ICP-MS	0.750	0.11	0.06	c
N11	2.9	0.6	2	SFICP-MS	0.300	-0.57	-0.78	a
N12	3.345	0.669	√3	ICP-MS	0.386	0.37	0.41	a
N13	3.1	0.53	2	ICP-MS	0.265	-0.15	-0.22	a
N14	3.3	0.5	2	ETAAS	0.250	0.27	0.43	a
N15	2.905	0.542	2	SFICP-MS	0.271	-0.56	-0.82	a
N16	2.11	0.42	2	ETAAS	0.210	-2.23	-3.89	a
N17	3.1837	0.3979	2	SFICP-MS	0.199	0.03	0.05	a
N18	2.7	0.7	2	ICP-MS	0.350	-0.99	-1.20	a
N19	2.926	0.59	√3	ICP-MS	0.341	-0.51	-0.64	a
N20	3.01	0.3	2	SFICP-MS	0.150	-0.34	-0.70	b
N21	3.45	0.09	2		0.045	0.59	1.56	b
N22	< 3			AAS				
N23	3.16	0.19	2	ICP-MS	0.095	-0.02	-0.05	b
N24	3.419	0.855	2	ETAAS	0.428	0.52	0.54	a
N27	2.97	0.37	2	AAS	0.185	-0.42	-0.79	a
N38	2.85	0.6	2	AAS	0.300	-0.67	-0.92	a
N39	3.13	0.31	2	ICP-MS	0.155	-0.08	-0.17	b
N42	< 4			FAAS				
N45	1.761	1.328	2	ETAAS	0.664	-2.96	-2.05	c
N50	3.05	0.52	2	ICP-MS	0.260	-0.25	-0.38	a
N101	2.43	0.61	2	ETAAS	0.305	-1.56	-2.11	a
N102	2.828	0.048	3		0.016	-0.72	-1.96	b

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

[b] performance: satisfactory, questionable, unsatisfactory,

[c] a : u_{min} (u_{ref}) ≤ u_{lab} ≤ u_{max} (σ); b : u_{lab} < u_{min}; and c : u_{lab} > u_{max}



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

Annex 13: Results for total Hg

Assigned range: X_{ref} = 0.0076; U_{ref} (k=2) = 0.0009; σ = 0.0017 (all values in mg kg^{-1})

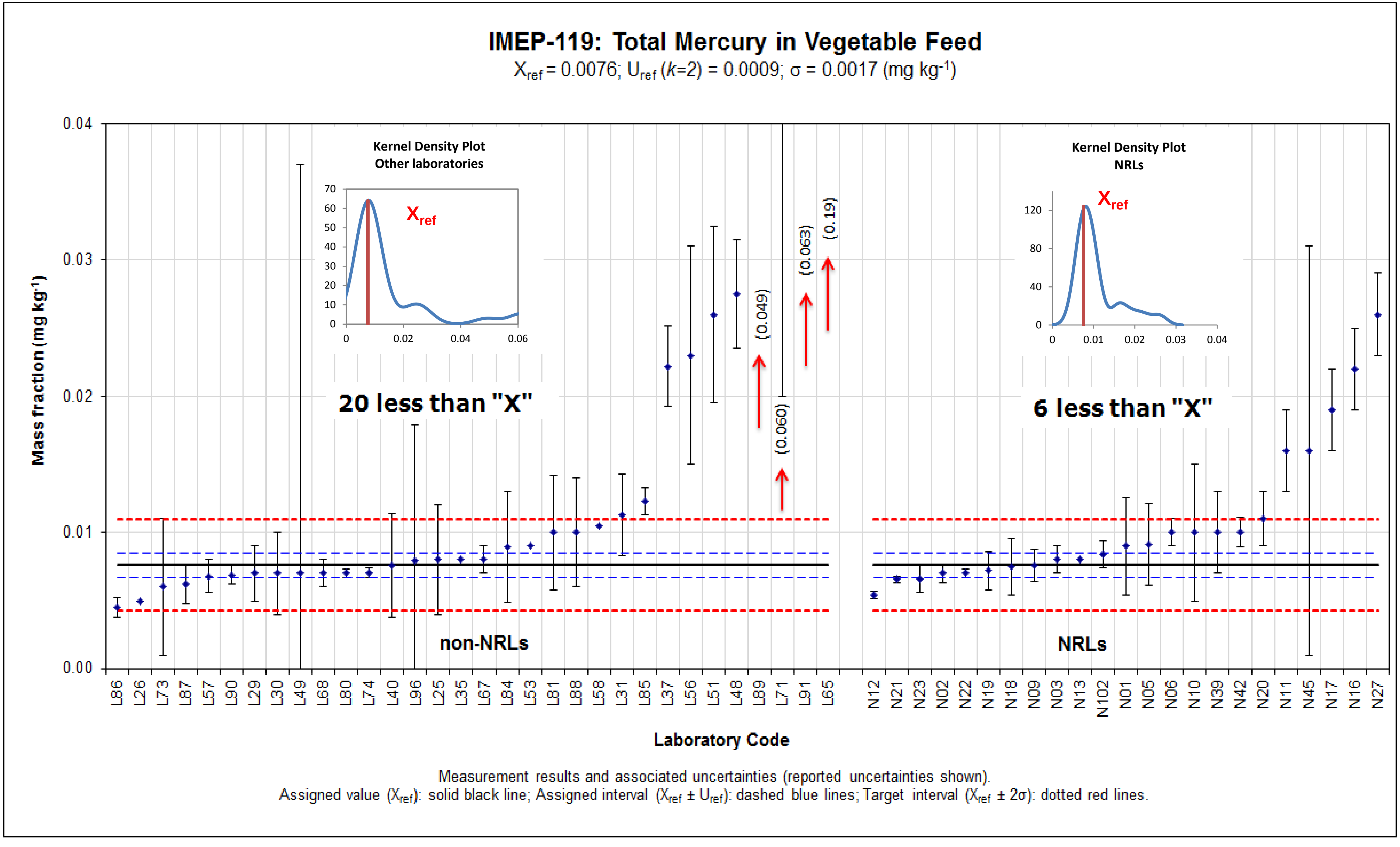
Lab Code	X_{lab}	±	k [a]	Technique	u_{lab}	z-score [b]	ζ-score [b]	unc. [c]
L25	0.008	0.004	100	CV-AAS	0.00004	0.24	0.90	b
L26	0.005	0	2	EMA	0.000	-1.56	-5.87	b
L29	0.007	0.002	2	EMA	0.001	-0.36	-0.55	a
L30	0.007	0.003	2	CV-AAS	0.0015	-0.36	-0.38	a
L31	0.0113	0.003	2	EMA	0.0015	2.21	2.37	a
L32	< 0.01			ICP-MS				
L33	< 0.01			CV-AAS				
L35	0.008			CV-AAS	0.000	0.24	0.90	b
L36	< 0.04			ICP-MS				
L37	0.0222	0.0029	2	ICP-MS	0.00145	8.73	9.63	a
L40	0.0076	0.0038	3	CV-AAS	0.001267	0.00	0.00	a
L41	< 0.01			FIMS				
L43	< 0.0126			CV-AAS				
L46	< 0.05			CV-AAS				
L48	0.0275	0.004	2	EMA	0.002	11.90	9.71	c
L49	0.007	0.03	2	SFICP-MS	0.015	-0.36	-0.04	c
L51	0.026	0.0065	2	ICP-OES	0.00325	11.00	5.61	c
L52	< 0.02			FAAS-MHS				
L53	0.009			CV-AAS	0.000	0.84	3.16	b
L56	0.023	0.008	2	ICP-MS	0.004	9.21	3.83	c
L57	0.0068	0.0012	2	CV-AAS	0.0006	-0.48	-1.07	a
L58	0.0105			SFICP-MS	0.000	1.73	6.55	b
L59	< 0.01			CV-AAS				
L60	< 0.02			ICP-IDMS				
L62	< 0.075			ICP-MS				
L64	< 0.001			HG-ICP				
L65	0.19	0.0298	2	FAAS-MHS	0.0149	109.09	12.24	c
L67	0.00805	0.001	2	ICP-IDMS	0.0005	0.27	0.67	a
L68	0.007	0.001	2	EMA	0.0005	-0.36	-0.90	a
L70	< 0.05			EMA				
L71	0.06	0.04	2	HG-AAS	0.02	31.34	2.62	c
L72	< 0.04			ICP-OES				
L73	0.006	0.005	2	CV-AAS	0.0025	-0.96	-0.63	c
L74	0.00708	0.00036	1	CV-AAS	0.00036	-0.31	-0.91	b
L76	< 0.05			ICP-MS				
L78	< 0.1			CV-AAS				
L79	< 0.058		2	AAS-F				
L80	0.007	0.0003	2	CV-AAS	0.00015	-0.36	-1.28	b
L81	0.01	0.0042	√3	SFICP-MS	0.002425	1.44	0.97	c
L84	0.00893	0.0041	2	ICP-MS	0.00205	0.80	0.63	c
L85	0.0123	0.001	2	HG ICP OES	0.0005	2.81	7.04	a
L86	0.0045	0.0007	√3	AAS	0.000404	-1.85	-5.17	b
L87	0.0062	0.0014	2	EMA	0.0007	-0.84	-1.69	a
L88	0.01	0.004	2	SFICP-MS	0.002	1.44	1.17	c
L89	0.049	0.008	2	CV-AAS	0.004	24.76	10.29	c

Lab Code	X_{lab}	±	k [a]	Technique	u_{lab}	z-score [b]	ζ-score [b]	unc. [c]
L90	0.0069	0.0007	2	AAS	0.00035	-0.42	-1.24	b
L91	0.063	0.012	√3	CV-AAS	0.006928	33.13	7.98	c
L92	< 0.02			ICP-IDMS				
L96	0.00791	0.01	2	ICP-MS	0.005	0.19	0.06	c
L98	< 0.048			CV-AAS				
L99	< 0.02			ICP-OES				
L100	< 0.004			ICP-IDMS				
N01	0.009	0.0036	2		0.0018	0.84	0.76	c
N02	0.007	0.0007	2	SFICP-MS	0.00035	-0.36	-1.06	b
N03	0.008	0.001	2	CV-AFS	0.0005	0.24	0.60	a
N04	< 0.006			FIMS				
N05	0.0091	0.003	2	Q-ICP-MS	0.0015	0.90	0.96	a
N06	0.01	0.001	2	EMA	0.0005	1.44	3.59	a
N09	0.0076	0.0012	2	ICP-MS	0.0006	0.00	0.00	a
N10	0.01	0.005	2	CV-AAS	0.0025	1.44	0.95	c
N11	0.016	0.003	2	EMA	0.0015	5.02	5.37	a
N12	0.0054	0.00029	√3	EMA	0.000167	-1.32	-4.65	b
N13	0.008			EMA	0.000	0.24	0.90	b
N14	< 0.034			EMA				
N15	< 0.01			SFICP-MS				
N16	0.022	0.003	2	CV-AAS	0.0015	8.61	9.21	a
N17	0.019	0.003	2	EMA	0.0015	6.82	7.29	a
N18	0.0075	0.0021	2	EMA	0.00105	-0.06	-0.09	a
N19	0.0072	0.0014	√3	CV-AFS	0.000808	-0.24	-0.43	a
N20	0.011	0.002	2	CV-AAS	0.001	2.03	3.11	a
N21	0.00656	0.00022	2		0.00011	-0.62	-2.28	b
N22	0.007	0.00028	2	EMA	0.00014	-0.36	-1.29	b
N23	0.0066	0.001	2	EMA	0.0005	-0.60	-1.50	a
N24	< 0.05			HG-AAS				
N27	0.026	0.003	2	CV-AAS	0.0015	11.00	11.77	a
N39	0.01	0.003	2	EMA	0.0015	1.44	1.53	a
N42	0.01	0.0011	2	CV-AAS	0.00055	1.44	3.40	a
N45	0.016	0.015	2	CV-AAS	0.0075	5.02	1.12	c
N50	< 0.025			ICP-MS				
N101	< 0.01			EMA				
N102	0.0084	0.001	2		0.0005	0.48	1.20	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] performance: satisfactory, questionable, unsatisfactory,

[c] a : u_{min} (u_{ref}) ≤ u_{lab} ≤ u_{max} (σ); b : u_{lab} < u_{min}; and c : u_{lab} > u_{max}



IMEP-119: Total Mercury in Vegetable Feed
X_ref = 0.0076; U_ref (k=2) = 0.0009; σ = 0.0017 (mg kg-1)
Mass fraction (mg kg-1)
Kernel Density Plot
Other laboratories
Xref
Kernel Density Plot
NRLs
Xref
20 less than "X"
6 less than "X"
(0.049)
(0.060)
(0.063)
(0.19)
non-NRLs
NRLs
L86 L26 L73 L87 L57 L90 L29 L30 L49 L68 L80 L74 L40 L96 L25 L35 L67 L84 L53 L81 L88 L58 L31 L85 L37 L56 L51 L48 L89 L71 L91 L65
N12 N21 N23 N02 N22 N19 N18 N09 N03 N13 N102 N01 N05 N06 N10 N39 N42 N20 N11 N45 N17 N16 N27
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Assigned value (Xref): solid black line; Assigned interval (Xref ± Uref): dashed blue lines; Target interval (Xref ± 2σ): dotted red lines.

Annex 14: Experimental details for NRLs and scoring (z-scores)

Lab ID		Official method	LOD (mg kg[-1])	Moisture (% w/w)	CRM for validation of measurement procedure	CRM for instrument calibration	Sample digestion	Digestion mixture	Experience	Technique	Compliant material?
N01	As	NMKL procedure nr 186, 20					Closed microwave	HNO_3	> 1000		
	Cd	NMKL procedure nr 186									
	Pb										
	Hg										
N02	As	No	0,0025	3.38%		Multi Nist 695	Closed microwave	HNO_3	50-250	SFICP-MS	Yes
	Cd		0,0007								
	Pb		0,002								
	Hg		0,0005								
N03	As	SOP 1057/1058	0.03	3.19%			Closed microwave	HNO_3	250-1000	ETAAS	Yes
	Cd		0.003								
	Pb		0.03								
	Hg	SOP 1057/1060	0.005							CV-AFS	
N04	As			2.5							Yes
	Cd		0.08		MR 1 g/l	MR 1 g/l	Closed microwave	H_2O_2 + HNO_3	0-50	Q-ICP-MS	
	Pb		0.52						0-50		
	Hg		0.003				Closed microwave	H_2O_2 + HNO_3	0-50	FIMS	
N05	As		0.027	2,3	3xPT-Material		Closed microwave	HNO_3	0-50	Q-ICP-MS	Yes
	Cd		0.0025								
	Pb		0.017								
	Hg		0.002								
N06	As	Modifided CEN/TS 15621	0.5	3.4			Closed microwave	H_2O_2 + HNO_3	250-1000	ICP-OES	Yes
	Cd		0.1								
	Pb		1								
	Hg	AMA254 technics	0.01						50-250	EMA	
N07	As	LST EN 15763:2010	0.1	3.63			Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	Yes
	Cd		0.01								
	Pb		0.01								
	Hg		0.007								
N08	As										Yes
	Cd	CYS EN 15550:2007					Closed microwave	H_2O_2 + HNO_3	0-50		
	Pb	CYS EN 15550:2007					Closed microwave	H_2O_2 + HNO_3	0-50		
	Hg										
N09	As	In-house	0.0005	3.27	NIST1548a, CE278K		Closed microwave	HNO_3 + HCl	0-50	ICP-MS	Yes
	Cd		0.0001								
	Pb		0.0007								
	Hg		0.0003					HNO_3			
N10	As	VDLUVA - VII 2.2.2.5.	0,01	3,20 %	IMEP 108		Closed microwave	HNO_3	250-1000	ICP-MS	Yes
	Cd		0,0025		NIST 1547						
	Pb		0,01								
	Hg	EN 16277	0,0025				Open wet			CV-AAS	

Lab ID		Official method	LOD (mg kg^{-1})	Moisture (% w/w)	CRM for validation of measurement procedure	CRM for instrument calibration	Sample digestion	Digestion mixture	Experience	Technique	Compliant material?
N11	As	EN 15763	0,010	3.1	SRM 3256, NCS ZC73012		Closed microwave	H_2O_2 + HNO_3	50-250	SFICP-MS	Yes
	Cd		0,005						250-1000		
	Pb		0,010								
	Hg	EPA 7473	0,005						50-250	EMA	
N12	As		0.00231	2.83			Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	Yes
	Cd		0.0016								
	Pb		0.00135								
	Hg		0.00002							EMA	
N13	As	No		3.64%			Closed microwave	HNO_3	250-1000	ICP-MS	Yes
	Cd										
	Pb										
	Hg									EMA	
N14	As	ISTISAN 34/96	0.18	3.36	Lichen BCR 482		Closed microwave	H_2O_2 + HNO_3 + HF	250-1000	ETAAS	Yes
	Cd	IN HOUSE	0.075		Lichen BCR 482		Closed microwave	H_2O_2 + HNO_3 + HF	250-1000	ETAAS	
	Pb	IN HOUSE	0.499		spike		Closed microwave	H_2O_2 + HNO_3 + HF	250-1000	ETAAS	
	Hg	EPA 7473/1998	0.01		tomato leaves NIST1573a				250-1000	EMA	
N15	As	200.8 (ICP-SMS), modified	0.01	3.10%	DORM-2		Closed microwave	H_2O_2 + HNO_3 + HF	50-250	SFICP-MS	Yes
	Cd		0.003								
	Pb		0.02								
	Hg		0.005				Closed microwave	H_2O_2 + HNO_3	50-250	SFICP-MS	
N16	As	EN14546	0.05	3.53			Dry ashing		0-50	HG-AAS	Yes
	Cd	EN15550	0.05		FAPAS 07116		Closed microwave	H_2O_2 + HNO_3		ETAAS	
	Pb	EN15550	0.2		IMEP-110		Closed microwave	H_2O_2 + HNO_3	0-50	ETAAS	
	Hg	EN13806	0.01		IMEP-110		Closed microwave	H_2O_2 + HNO_3	0-50	CV-AAS	
N17	As		0,0005	4,85	SRM 1643e	VAR CAL2	Closed microwave	H_2O_2 + HNO_3	0-50	SFICP-MS	Yes
	Cd		0,0005								
	Pb		0,0005								
	Hg		0,001		TORT 2		Dry ashing		0-50	EMA	
N18	As		0.0006	3.10%	IRMM-804		Closed microwave	HNO_3	0-50	ICP-MS	Yes
	Cd		0.00015		IRMM-804, NIST-1515						
	Pb		0.0009		IRMM-804, NIST-1515						
	Hg		0.000051		BCR-150					EMA	
N19	As			4,32			Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	
	Cd										
	Pb										
	Hg									CV-AFS	
N20	As		0.01	0.05%	IMEP-117		Closed microwave	H_2O_2 + HNO_3	250-1000	SFICP-MS	Yes
	Cd		0.005								
	Pb		0.01								
	Hg	AOAC 971.21	0.005		IMEP-116		Open wet	HNO_3	250-1000	CV-AAS	
N21	As		0.008	3.575	Soya Flour, FAPAS 770		Closed microwave	H_2O_2 + HNO_3	0-50		Yes
	Cd	EN 14084:2003	0.006				Closed microwave	H_2O_2 + HNO_3	0-50		
	Pb	EN 14084:2003	0.02				Closed microwave				
	Hg	In house	0,0005						0-50		

Lab ID		Official method	LOD ($mg\ kg^{-1}$)	Moisture (% w/w)	CRM for validation of measurement procedure	CRM for instrument calibration	Sample digestion	Digestion mixture	Experience	Technique	Compliant material?
N22	As										
	Cd		0.5	0.36%	AAFCO		Dry ashing		50-250	AAS	Yes
	Pb		3								
	Hg		0.0015		BCR 463				50-250	EMA	
N23	As		0.001		DORM-4		Closed microwave	H_2O_2 + HNO_3	250-1000	ICP-MS	Yes
	Cd		0.0003	3.68%	DORM-4						
	Pb		0.004		IAEA-336						
	Hg		0.0001		CZ9024					EMA	
N24	As	MSZ EN 16206:2012	0,040			MerckCRM	Closed microwave	H_2O_2 + HNO_3	50-250	HG-AAS	Yes
	Cd	MSZ EN 15550:2008	0,040	3,39						GETAAS	
	Pb	MSZ EN 15550:2008	0,040							ETAAS	
	Hg	CEN/TC327 N1119	0,050			CaPurCRM	Closed microwave	H_2O_2 + HNO_3	50-250	HG-AAS	
N27	As										
	Cd			8,9		Yes	Closed microwave	HNO_3	250-1000	AAS	Yes
	Pb										
	Hg						Closed microwave	HNO_3 + HCl	250-1000	CV-AAS	
N38	As										
	Cd	AOAC 999.10	0.0023	6.30%	IMEP111, IMEP117		Closed microwave	HNO_3	0-50	AAS	Yes
	Pb	AOAC 999.10	0.01		IMEP111, IMEP114		Closed microwave	H_2O_2 + HNO_3			
	Hg										
N39	As		0.02			CZ9003(1N)					
	Cd	EN 15763:2009	0.02	4.56	GBW7604	CZ9010(1N)	Open microwave	HNO_3	50-250	ICP-MS	Yes
	Pb		0.3			CZ9041(1N)					
	Hg		0.001			CZ9024(1N)				EMA	
N42	As	SR EN 14546	0.1		BCR 32		Dry ashing	HNO_3 + HCl	0-50	HG-AAS	
	Cd	SR EN 14082	0.15		BCR 32	STD 1000 mg/L	Dry ashing	HCl	0-50	FAAS	Yes
	Pb		2								
	Hg	SR EN 13806	0.003		BCR 32		Closed microwave	H_2O_2 + HNO_3 + HCl	0-50	CV-AAS	
N45	As	MSA EN 14546:2005	0.1				Dry ashing	HNO_3 + HCl	0-50	HG-AAS	
	Cd		0.01	3.27%	Past PT material		Closed microwave	H_2O_2 + HNO_3	0-50	ETAAS	Yes
	Pb	In-house	0.2				Closed microwave	H_2O_2 + HNO_3	0-50	ETAAS	
	Hg		0.01				Open wet	H_2O_2 + HNO_3	0-50	CV-AAS	
N50	As		0.0079				Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	
	Cd		0.0014	3.04	ERM-CD281		Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	Yes
	Pb		0.0045				Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	
	Hg		0.025		SRM 1570a		Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	
N101	As	Intenal standard	0.04		Internal standard		Closed microwave	HNO_3	250-1000	ETAAS	
	Cd		0.002	0.4		Internal standard	Closed microwave	HNO_3	250-1000	ETAAS	Yes
	Pb	Internal standard	0.04		standard						
	Hg		0.005						250-1000	EMA	
N102	As		0,250								
	Cd	In-house	0,025	2,75%			Closed microwave	HNO_3 + HF	0-50		Yes
	Pb		0,250								
	Hg		0,001								

Annex 15: Experimental details for non-NRLs and scoring (z-scores)

Lab ID		Official method	LOD (mg kg[-1])	Moisture (% w/w)	CRM for validation of measurement procedure	CRM for instrument calibration	Sample digestion	Digestion mixture	Experience	Technique	Compliant material?
L25	As	DIN EN ISO 11969	0.100	2.20%			Closed microwave	HNO_3	250-1000	HG-AAS	
	Cd	VDLUFA MB VII 2.2.2.5	0.100							ICP-MS	
	Pb		0.100							ICP-MS	
	Hg	DIN EN 1483	0.002							CV-AAS	
L26	As									ICP-IDMS	
	Cd										
	Pb										
	Hg									EMA	
L28	As			3.3							Yes
	Cd		0.04				Closed microwave	H_2O_2 + HNO_3	0-50	AAS	
	Pb										
	Hg										
L29	As	No	0.00004	2.8	NIST 1548a	Yes	Closed microwave	HNO_3 + HCl	50-250	ICP-IDMS	Yes
	Cd		0.000009								
	Pb		0.00005								
	Hg		0.0005		In-house					EMA	
L30	As	VDLUFA VII 2.2.2.10	0.02	1.10%	Enquete samples		Closed microwave	H_2O_2 + HNO_3 + HCl	> 1000	HG-AAS	Yes
	Cd	DIN EN 15550:2007	0.01				Closed microwave	H_2O_2 + HNO_3	> 1000	ETAAS	
	Pb		0.05				Closed microwave	H_2O_2 + HNO_3	> 1000	ETAAS	
	Hg	VDLUFA VII 2.2.2.9	0.01							CV-AAS	
L31	As	Digestion: EN15550	0.1	3.3	Bipea, samples	custom made solutions	Closed microwave	H_2O_2 + HNO_3	0-50	ICP-MS	Yes
	Cd		0.1						50-250		
	Pb		0.1						50-250		
	Hg	none	0.005				Dry ashing		50-250	EMA	
L32	As	CHE01-WV838	0.066	2.05%	Yes		Closed microwave	H_2O_2 + HNO_3	0-50	ICP-MS	
	Cd		0.005								
	Pb		0.008								
	Hg		0.01				Closed microwave	H_2O_2 + HNO_3	0-50	ICP-MS	
L33	As	VDLUFA III 17.9.1	0.5	3.70%	div.		Closed microwave	H_2O_2 + HNO_3	> 1000		Yes
	Cd		0.1							ICP-MS	
	Pb		0.5							ICP-MS	
	Hg	DIN EN 16277	0.01				Closed microwave	H_2O_2 + HNO_3	> 1000	CV-AAS	
L34	As		0.05	3.61	TORT-3			HNO_3 + HCl	0-50	FAAS	Yes
	Cd		0.13				Dry ashing	HCl	0-50	AAS	
	Pb		1.25				Dry ashing	HCl	0-50	AAS	
	Hg										
L35	As		0,05	2,97	NCS73014		Pressure bomb	HNO_3	50-250	HG-AAS	Yes
	Cd		0,01						250-1000	ETAAS	
	Pb		0,1								
	Hg		0,005							CV-AAS	

Lab ID		Official method	LOD ($mg\ kg^{-1}$)	Moisture (% w/w)	CRM for validation of measurement procedure	CRM for instrument calibration	Sample digestion	Digestion mixture	Experience	Technique	Compliant material?
L36	As		0.04	3.68%		1000 mg/L	Closed microwave	H_2O_2 + HNO_3	0-50	ICP-MS	
	Cd		0.0005								
	Pb		0.03								
	Hg		0.005				Closed microwave	H_2O_2 + HNO_3	0-50	ICP-MS	
L37	As		0.044	2.97%	NIST 1570a Spinach leaves	N.A.	Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	Yes
	Cd		0.0014								
	Pb		0.0057								
	Hg		0.0075				Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	
L40	As	DIN EN 16206	0,010	3.55	IPE 149	Merck-Standard	Closed microwave	H_2O_2 + HNO_3	50-250	HG-AAS	Yes
	Cd	DIN EN 15550	0,002			Kraft-Standard				ETAAS	
	Pb		0,05			Kraft					
	Hg	DIN EN 16277	0,0005			Merck				CV-AAS	
L41	As		0.05	3.48%	NIST1570a Spinach leaves		Closed microwave	H_2O_2 + HNO_3	> 1000	SFICP-MS	Yes
	Cd	NBN EN 13805, NEN-EN 1576	0.01		IRMM 804 Rice						
	Pb		0.02								
	Hg	(NEN-EN 13806 -	0.01		NIST1570a Spinach leaves		Closed microwave	H_2O_2 + HNO_3	> 1000	FIMS	
L43	As		0.05			ICP-MS	Closed microwave	HNO_3		ICP-IDMS	
	Cd		0.012								
	Pb		0.05								
	Hg		0.006			CV-AAS	Closed microwave	HNO_3		CV-AAS	
L46	As	AOAC 999.10	2.5	3.3	No	Yes	Closed microwave	HNO_3	0-50	ICP-OES	Yes
	Cd		0.5				Closed microwave	HNO_3	0-50		
	Pb		2.5				Closed microwave	HNO_3	0-50	ICP-OES	
	Hg		0.05							CV-AAS	
L48	As		0.06	2.77%	Yes		Open wet	HNO_3 + HCl	50-250		No
	Cd		0.0005				Open wet	HCl	50-250	ICP-OES	
	Pb		0.005				Open wet	HCl	50-250	ICP-OES	
	Hg		0.0001			Yes			250-1000	EMA	
L49	As	N/A	0.0004	3.84	Yes	Yes	Closed microwave	HNO_3	> 1000	SFICP-MS	Yes
	Cd		0.0002								
	Pb		0.001								
	Hg		0.0002								
L51	As		0.01	4.33			Dry ashing	HNO_3 + HCl	50-250	ICP-OES	Yes
	Cd		0.01				Dry ashing	HNO_3 + HCl	50-250	ICP-OES	
	Pb		0.01				Dry ashing	HNO_3 + HCl	50-250	ICP-OES	
	Hg		0.01				Open wet	H_2O_2 + HNO_3	0-50	ICP-OES	
L52	As	VDLUFA MB VII 2.2.2.10	0.4	3.30%	Sample from ring trial	Sample from ring trial	Closed microwave	H_2O_2 + HNO_3	50-250	FAAS	Yes
	Cd	VDLUFA MB VII 2.2.2.5	0.1							ICP-MS	
	Pb		1								
	Hg	VDLUFA MB VII 2.2.2.9	0.02				Closed microwave	H_2O_2 + HNO_3	50-250	FAAS-MHS	
L53	As	VDLUFA MB VII 2.2.2.10	0,003	4.4	CRM	Yes	Closed microwave	H_2O_2 + HNO_3	250-1000	HG-AAS	Yes
	Cd	DIN EN ISO 5961-3	0,002							ETAAS	
	Pb	EN ISO 15586	0,004								
	Hg	VDLUFAMB VII 2.2.2.9	0,008							CV-AAS	

Lab ID		Official method	LOD (mg kg^{-1})	Moisture (% w/w)	CRM for validation of measurement procedure	CRM for instrument calibration	Sample digestion	Digestion mixture	Experience	Technique	Compliant material?
L54	As		0.03		Dorm 4		Closed microwave	H_2O_2 + HNO_3	250-1000	ICP-IDMS	Yes
	Cd		0.006								
	Pb		0.02								
	Hg										
L56	As		0.07	3.01%		Romil 1000 ppm	Closed microwave	HNO_3	0-50		Yes
	Cd		0.007			Romil				ICPMS	
	Pb		0.07								
	Hg		0.007				Closed microwave	HNO_3	0-50	ICP-MS	
L57	As		0.0057		Yes	Yes	Closed microwave	H_2O_2 + HNO_3	250-1000	ICP-IDMS	Yes
	Cd		0.0006							ETAAS	
	Pb		0.0019								
	Hg		0.0004					HNO_3		CV-AAS	
L58	As			4.55				H_2O_2 + HNO_3	250-1000	SFICP-MS	
	Cd								250-1000		
	Pb								> 1000		
	Hg								250-1000		
L59	As									HG-AAS	
	Cd									AAS	
	Pb										
	Hg									CV-AAS	
L60	As		0.2	2.9	TNRL03		Open wet	HNO_3	50-250	ICP-IDMS	Yes
	Cd		0.1								
	Pb		0.5								
	Hg		0.02				Open wet	HNO_3	50-250	ICP-IDMS	
L62	As		0.15	1.60%	InorganicVentures71A10ppm		Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	Yes
	Cd		0.05								
	Pb		0.1								
	Hg		0.05		Inorganic VenturesHg10ppm		Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	
L64	As	SM 3120B	0.001	3,70	Certified standard of As	Certified standard of As	Closed microwave	H_2O_2 + HNO_3	0-50	ICP-OES	Yes
	Cd		0.001		Certified standard or Cd	Certified standard or Cd	Closed microwave	H_2O_2 + HNO_3	0-50	ICP-OES	
	Pb		0.001		certified standard of Pb	Certified standard of Pb	Closed microwave	H_2O_2 + HNO_3	0-50	ICP-OES	
	Hg	USP 36-NF 31 <261> II B	0.001		Certified standard of Hg	Certified standard of Hg	Closed microwave	H_2O_2 + HNO_3	0-50	HG-ICP	
L65	As	A.O.A.C 19 ed, 2012	0.0013	2.947	Standard Solution	Standard Solution	Open wet	HNO_3	250-1000	AAS	Yes
	Cd		0.005							ICP-OES	
	Pb		0.034				Open wet	HNO_3	250-1000	AAS	
	Hg		0.00035				Open wet	HNO_3	250-1000	FAAS-MHS	
L67	As	FDA EAM 4.7	0.01		NIST2976	IV-ICPMS-71A	Closed microwave	H_2O_2 + HNO_3	> 1000	ICP-IDMS	No
	Cd		0.01						250-1000		
	Pb		0.01						> 1000		
	Hg		0.01			MSHG-10PPM			250-1000		
L68	As									ICP-OES	
	Cd										
	Pb										
	Hg									EMA	

Lab ID		Official method	LOD (mg kg^{-1})	Moisture (% w/w)	CRM for validation of measurement procedure	CRM for instrument calibration	Sample digestion	Digestion mixture	Experience	Technique	Compliant material?
L69	As		0,05	34,8 g/kg	Yes	Yes	Closed microwave	H_2O_2 + HNO_3	0-50	ETAAS	Yes
	Cd		0,004			No					
	Pb		0,16								
	Hg										
L70	As									ICP-OES	
	Cd									AAS	
	Pb										
	Hg									EMA	
L71	As	KSS M34 in house	0.025	3.14%	Yes	No	Dry ashing	HNO_3 + HCl	50-250	HG-AAS	Yes
	Cd	KSS M30 in house	0.004					HCl		AAS	
	Pb		0.037								
	Hg	KSS M35 in house	0.02				Open wet	HNO_3 + HCl	50-250	HG-AAS	
L72	As									ICP-OES	
	Cd									AAS	
	Pb										
	Hg									ICP-OES	
L73	As	CEN TC 275	0.01	0.8			Dry ashing	HNO_3		HG-AAS	
	Cd	SLMB 45	0.006				Closed microwave	H_2O_2 + HNO_3		ETAAS	
	Pb	SLMB 45	0.1							ICP-OES	
	Hg	SLMB 45	0.02							CV-AAS	
L74	As	None	0.1	4.03%	NIST SRM-1547				50-250	k0-INAA	Yes
	Cd										
	Pb										
	Hg	None	0.0002		NIST SRM-1570a		Open wet	HNO_3	250-1000	CV-AAS	
L75	As	ISO 14 083			CertiPUR As standard	CertiPUR As standard	Closed microwave	H_2O_2 + HNO_3 + HCl		GF AAS	Yes
	Cd				CertiPUR Cd standard	CertiPUR Cd standard			250-1000	ETAAS	
	Pb				CertiPUR Pb standard	CertiPUR Pb standard			250-1000	GF AAS	
	Hg										
L76	As	AOAC 2013.06	0.02	3.55		PerkinElmer	Closed microwave	H_2O_2 + HNO_3	250-1000	ICP-MS	Yes
	Cd		0.02						50-250		
	Pb		0.02						250-1000		
	Hg		0.02				Closed microwave	H_2O_2 + HNO_3	50-250	ICP-MS	
L77	As	FDA	0.12	2.53	Yes		Closed microwave	H_2O_2 + HNO_3	0-50	ETAAS	Yes
	Cd		0.006				Closed microwave	H_2O_2 + HNO_3	0-50	ETAAS	
	Pb		0.14								
	Hg										
L78	As		0.6	3.45%	FAPAS	Scahrlau	Closed microwave	H_2O_2 + HNO_3	250-1000	ETAAS	Yes
	Cd		0.06		BCR						
	Pb		0.4								
	Hg		0.05		FAPAS	MERCK	Closed microwave	H_2O_2 + HNO_3	50-250	CV-AAS	
L79	As	ISO 27085:2009	0.014	6.82%			Closed microwave	HNO_3	> 1000	ICP-MS	Yes
	Cd	NMKL-161	0.25				Closed microwave	HNO_3	> 1000	ICP-OES	
	Pb		0.65				Closed microwave	HNO_3	> 1000	ICP-MS	
	Hg	NMKL 170	0.058				Closed microwave	HNO_3	> 1000	AAS-F	

Lab ID		Official method	LOD (mg kg^{-1})	Moisture (% w/w)	CRM for validation of measurement procedure	CRM for instrument calibration	Sample digestion	Digestion mixture	Experience	Technique	Compliant material?
L80	As	EPA3051/200.8	0.015	3.1	NIST 1547	NIST 1547	Closed microwave	HNO_3	250-1000	ICP-MS	Yes
	Cd		0.001								
	Pb		0.001								
	Hg	EPA3051/245.6	0.005							CV-AAS	
L81	As	ICP-MS	0.018	2.75	FAPAS	MULTI-STANDARD	Closed microwave	HNO_3	> 1000	SFICP-MS	Yes
	Cd		0.005								
	Pb		0.007				Closed microwave	HNO_3	> 1000	SFICP-MS	
	Hg		0.003				Closed microwave	HNO_3	> 1000	SFICP-MS	
L82	As	EN 14546:2005	0.0052	3.42	Control material	JT Baker	Dry ashing	H_2O_2 + HCl	50-250	HG-AAS	Yes
	Cd	EN 14084:2003	0.0024		LGC CS-M-2	Accutrace	Closed microwave	H_2O_2 + HNO_3	250-1000	AAS	
	Pb	EN 14084:2003	0.0036		Mushroom powder	Accutrace	Closed microwave	H_2O_2 + HNO_3	250-1000	AAS	
	Hg										
L83	As										Yes
	Cd	ISO 14082	0.42			CRM	Dry ashing	HNO_3 + HCl	250-1000	ICP-OES	
	Pb		0.38				Dry ashing	HNO_3 + HCl	250-1000	ICP-OES	
	Hg										
L84	As	N/A	0.0004	3.53	Yes	Yes	Closed microwave	HNO_3	> 1000	ICP-MS	Yes
	Cd		0.0002								
	Pb		0.001								
	Hg		0.0002								
L85	As		0.02	3.285			Closed microwave	H_2O_2 + HNO_3		HG ICP OES	
	Cd		0.04							ICP-OES	
	Pb		0.06								
	Hg		0.005				Closed microwave	H_2O_2 + HNO_3		HG ICP-OES	
L86	As									SFICP-MS	
	Cd										
	Pb										
	Hg									AAS	
L87	As	PN-EN 14546:2005	0,002	1,90 %	Yes	No	Dry ashing	HNO_3 + HCl	50-250	HG-AAS	Yes
	Cd	PN-EN 14082:2004	0,001				Dry ashing	HNO_3	50-250	ETAAS	
	Pb		0,001								
	Hg	EPA 7473	0,0001						50-250	EMA	
L88	As		0.03	3.02		Yes	Closed microwave	HNO_3	0-50	SFICP-MS	Yes
	Cd		0.03								
	Pb		0.09								
	Hg		0.03								
L89	As	EN 14546	0.01	3.20%	PT material	PT material	Closed microwave	H_2O_2 + HNO_3	0-50	HG-AAS	Yes
	Cd	EN 15550	0.001		BCR-191	BCR-191	Closed microwave	H_2O_2 + HNO_3	50-250	ETAAS	
	Pb		0.01				Closed microwave	H_2O_2 + HNO_3	50-250	ETAAS	
	Hg	EN 13806	0.005				Closed microwave	H_2O_2 + HNO_3	0-50	CV-AAS	
L90	As	Own Research Procedure	0,0017	3.45%	Yes	Yes	Closed microwave	HNO_3	0-50	ICP-IDMS	Yes
	Cd		0,0050							ICP-OES	
	Pb		0,20								
	Hg		0,0001				Dry ashing			AAS	

Lab ID		Official method	LOD (mg kg^{-1})	Moisture (% w/w)	CRM for validation of measurement procedure	CRM for instrument calibration	Sample digestion	Digestion mixture	Experience	Technique	Compliant material?
L91	As		0.1	2.81			Closed microwave	H_2O_2 + HNO_3	50-250	ETAAS	Yes
	Cd		0.01				Closed microwave	H_2O_2 + HNO_3	50-250	ETAAS	
	Pb		0.1				Closed microwave	H_2O_2 + HNO_3	50-250	ETAAS	
	Hg		0.01				Closed microwave	H_2O_2 + HNO_3	50-250	CV-AAS	
L92	As	NMKL161 1998	0,05	4%	No	No	Closed microwave	H_2O_2 + HNO_3	50-250	ICP-IDMS	
	Cd		0,01							ICP-IDMS	
	Pb		0,02							CV-AFS	
	Hg	SS-EN16277:2012 annex D	0,02				Open wet	H_2O_2 + HNO_3	50-250	ICP-IDMS	
L93	As			3,26			Closed microwave	H_2O_2 + HNO_3 + HF	0-50	ICP-MS	
	Cd										
	Pb										
	Hg										
L95	As			3.32							No
	Cd	LST EN 14084:2003	0.01		BCR No 191		Closed microwave	H_2O_2 + HNO_3	0-50	AAS	
	Pb		0.1								
	Hg										
L96	As	EN13805	0,1		NIST8436		Closed microwave	H_2O_2 + HNO_3	> 1000	ICP-MS	
	Cd		0,01					H_2O_2 + HNO_3 + HCl			
	Pb		0,05								
	Hg		0,005								
L98	As	IH	0.8		No	No	Closed microwave	HNO_3	250-1000	AAS	No
	Cd		0.016								
	Pb		0.12								
	Hg		0.048				Closed microwave	HNO_3	250-1000	CV-AAS	
L99	As			2.29%			Closed microwave	H_2O_2 + HNO_3	50-250	ICP-OES	
	Cd						Closed microwave	H_2O_2 + HNO_3	50-250	ICP-IDMS	
	Pb						Closed microwave	H_2O_2 + HNO_3	50-250	ICP-OES	
	Hg						Closed microwave	H_2O_2 + HNO_3	50-250	ICP-OES	
L100	As		0.7	2.29			Closed microwave	H_2O_2 + HNO_3		ICP-IDMS	
	Cd		0.005				Closed microwave	H_2O_2 + HNO_3		ICP-OES	
	Pb		2.1				Closed microwave	H_2O_2 + HNO_3		ICP-IDMS	
	Hg		0.004				Closed microwave	H_2O_2 + HNO_3		ICP-IDMS	

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