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Determination of total As, Cd, Pb, Hg, Sn and inorganic As in canned food

IMEP-118 Proficiency test Report

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I. Fiamegkos, B. de la Calle, H. Emteborg,
J. Seghers, M.-F. Tumba, M. Vahcic,
F. Cordeiro, A. Cizek-Stroh, P. Robouch

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

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Abstract

This report presents the outcome of the proficiency test, on the determination of total As, Cd, Pb, Hg, Sn and iAs in canned food (peas in brine). The exercise was organised by the European Union Reference Laboratory for Heavy Metals (EURL-HM) to support the Commission Regulation (EC) 1881:2006 setting maximum levels for certain contaminants in foodstuffs. Participation in the proficiency test was mandatory for the nominated NRLs, and open to other OCLs and interested laboratories. A total of 127 participants from 36 countries registered to the exercise. All NRLs (36) reported results, while 4 non-NRL participants did not.
From the participating laboratories 54 % analysed the drained product and 46 % the solid/liquid composite. Hence, a non-unified analytical approach is observed. The majority of laboratories (more than 74 %) reported satisfactory results for the five scored measurands with measurable concentrations (total As, Cd, Pb, Sn and iAs). The best performances were obtained for total As, Cd and Pb. The interpretation of the respective legislation is not straightforward as indicated by the 32 laboratories that characterised the test item as compliant with the legislation, although it was not.

Erratum

The missing results for total Cd in the drained product (Annex 11, page 54) of laboratory N011 are included in the table of the IMEP-118 (EUR 27145) report.

Dr. IOANNIS FIAMEGKOS
EU-RL Heavy Metals
Coordinator of IMEP-118

European Union Reference Laboratory
Heavy Metals in Feed and Food

Determination of total As, Cd, Pb, Hg, Sn and inorganic As in canned food

IMEP-118 Proficiency Test Report

January 2015

Ioannis Fiamegkos (a)

Beatriz de la Calle (b,c)

Håkan Emteborg

John Seghers

Marie-France Tumba

Mitja Vahcic

Fernando Cordeiro (c)

Aneta Cizek-Stroh (d)

Piotr Robouch (c,e)

(a) ILC coordinator, (b) IMEP programme coordinator,

(c) Technical / scientific support, (d) Logistic support

(e) EURL-HM operating manager

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Executive summary

This report presents the outcome of a proficiency test (PT), on the determination of total As, Cd, Pb, Hg, Sn and iAs in canned food (peas in brine). The exercise was organised by the European Union Reference Laboratory for Heavy Metals for Feed and Food (EURL-HM) to support the implementation of provisions laid down in Commission Regulation (EC) No 1881/2006, which sets maximum levels for certain contaminants in foodstuffs.

National Reference Laboratories (NRLs) requested the EURL-HM to organise a PT with the aim to check: (i) the analytical capabilities of participating laboratories to analyse heavy metals, in particular Sn, in vegetables and (ii) the sample preparation approach applied by NRLs and Official Control Laboratories (OCLs) when analysing canned or jarred vegetables, using the drained product or the solid/liquid composite.

Participation in this PT was mandatory for the nominated NRLs, and open to official control laboratories and other interested laboratories. A total of 127 participants from 36 countries registered to the exercise. All NRLs (36) reported results, while 4 non-NRL participants did not.

Laboratory results were rated using z- and ζ-scores (zeta-scores) in accordance with ISO 13528:2005. The target standard deviation for proficiency assessment (σ), for all measurands was calculated using the Horwitz equation modified by Thompson, except for the case of total Sn where σ was decided by the scientific committee of the PT. In the case of total Hg the certifying laboratories reported "less than" values, therefore no scoring was provided for this measurand.

Two different sample preparation approaches have been identified: 54 % of the participating laboratories analysed the drained product, while 46 % the solid/liquid composite demonstrating the lack of specific sample preparation approach protocol. The majority of laboratories (more than 74 %) reported satisfactory results for total As, Cd, Pb, Sn and iAs. The best performances were obtained for total As, Cd and Pb. The interpretation of the respective legislation is not straightforward as indicated by the 32 laboratories that characterised the test item as compliant with the legislation, although it was not.

1 Introduction

Contamination with toxic elements is a global environmental and food safety concern. The consumption of contaminated food leads to uptake of toxic elements by humans, with the risk increasing proportional to the quantity consumed. Heavy metal toxicity can affect mental development and central nervous system function, alter the blood composition and disturb the function of organs like kidneys, lungs, and liver [1].

Heavy metals may occur in canned foods as a result of naturally incurred contamination of the food commodity or by migration from the packaging material. Metallic food packaging is mostly composed of tinplate (tin coated steel), chromium coated steel, or aluminium, which is mostly coated on the inner side with a resin to protect food from coming into contact with the metal. However, when the metal is exposed to the food as a result of damage of the coating, corrosion is accelerated and elements such as tin (Sn), iron (Fe), cadmium (Cd) and lead (Pb) could be released, increasing their levels in the food [2].

The occurrence of heavy metals in canned food is of great importance and covers a large variety of food commodities [3-7]. More specifically for tin, the general population is exposed to it through the diet with a mean tin intake ranging from <1 up to 15 mg per day. However, maximum daily intakes could reach 50–60 mg / day for individuals frequently consuming canned fruits, vegetables, and juices from un-lacquered cans. Tin levels are usually below 25 mg kg^{-1} in lacquered food cans, but may exceed 100 mg kg^{-1} in un-lacquered ones. Tin concentrations in canned foods increase with storage, time, and temperature [8].

Commission Regulation (EC) No 1881/2006 sets maximum levels for certain contaminants in foodstuffs [9]. The following limits apply: 200 mg kg^{-1} for tin in canned foods; 0.2 mg kg^{-1} for lead in legume vegetables, cereals and pulses; and 0.05 mg kg^{-1} for cadmium in vegetables and fruits. All values refer to wet weight.

A proficiency test (IMEP-118) was organised by the European Union Reference Laboratory for Heavy Metals (EURL-HM), to assess the performance of National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs) on the determination of total arsenic, cadmium, lead, mercury, tin and inorganic arsenic in canned peas. An additional outcome of this exercise consists in the evaluation of the various sample treatment approaches applied by NRLs and OCLs when analysing canned or jarred vegetables, using the drained product or the solid/liquid composite.

This report summarises and evaluates the outcome of IMEP-118.

2 IMEP support to EU policy

The International Measurement Evaluation Programme (IMEP) is run by the Joint Research Centre (JRC) - Institute for Reference Materials and Measurements (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 reference value, which is established according to metrological best practice.

IMEP helps laboratories to assess their estimate of measurement uncertainty. Participants are invited to report the uncertainty of their measurement results. IMEP integrates the uncertainty estimate 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-118 it was organised to support the Directorate General for Health and Consumers (DG SANCO) with the implementation of Commission Regulation (EC) No 1881/2006 [9].

Furthermore, IMEP-118 provided support to the following stakeholders:

- The European Cooperation for Accreditation (EA) in the frame of a Collaboration of Arrangement 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 Inter-American Accreditation Cooperation (IAAC).

3 Scope and aim

As stated in Regulation (EC) No 882/2004 one of the core duties of the European Union Reference Laboratories (EURLs) is to organise proficiency tests (PTs) for the benefit of staff of National Reference Laboratories (NRLs).

The organisation of the present PT – designated as "IMEP-118" - was requested by NRLs at the 8th EURL-HM Workshop held on September 24, 2013 (i) to assess the analytical capabilities of participating laboratories in determining total As, Cd, Pb, Hg, Sn and inorganic As mass fractions in a vegetable food matrix (in particular Sn); and (ii) to evaluate the various sample preparation approaches applied by NRLs and OCLs when analysing canned or jarred vegetables (using the drained product or the solid/liquid composite or any other approach).

The PT was organised following the administrative procedure and logistics defined by IMEP, a PT scheme accredited according to ISO 17043:2010 [10]. The assessment of the reported results was performed on the basis of requirements set by EU legislation [9].

4. Set up of the exercise

4.1 Time frame

IMEP-118 was included in the EURL-HM work program 2014 and was further approved by the Directorate General for Health and Consumers (DG SANCO). Invitation letters were sent to NRLs (Annex 1) on March 10, 2014. On the same day the exercise was announced on the IMEP web page (Annex 2) as well as to the European Cooperation for Accreditation (EA), to the Asian Pacific Laboratory Accreditation Cooperation (APLAC) and to the Inter-American Accreditation Cooperation (IAAC) (Annexes 3 - 5).

Registration was opened till April 14, 2014. The deadline for reporting results was set to June 6, 2014. Dispatch was followed by the web-based parcel tracking system of the courier service.

A preliminary report disclosing the assigned values together with the respective performance scoring was sent by e-mail to participants on July 10, 2014.

4.2 Confidentiality

The following confidentiality statement was made to EA, IAAC and APLAC: "*Confidentiality of the participants and their results towards third parties is guaranteed*". In the case of EA the following was added: "*However, IMEP will disclose details of the participants that have been nominated by EA to you. The EA accreditation bodies may wish to inform the nominees of this disclosure*". A similar clause was provided to those NRLs who wished to appoint OCLs in their respective countries to take part in IMEP-118.

4.3 Distribution

Test items were dispatched to participants on April 22-24 and 28, 2014. Each participant received:

- One glass jar containing approximately 170 g of peas in brine;
- A "Sample accompanying letter" (Annex 6); and
- A "Confirmation of receipt form" to be sent back to IRMM after receipt of the test material (Annex 7).

4.4 Instructions to participants

Detailed instructions were given to participants in the "Sample accompanying letter" mentioned above. The measurands were defined as "Total As, Cd, Pb, Hg, Sn and iAs in canned food".

Laboratories were asked to perform two or three independent measurements and to report the mean, the associated expanded measurement uncertainty, the coverage factor of the associated expanded measurement uncertainty and the technique used to perform the measurements. The measurement results were to be **corrected for recovery**. Participants were asked to follow their routine procedures for the analysis and to report results in the same way (e.g. number of significant figures) as they would report to their customers. All data were to be reported on **wet weight basis**.

Participants received an individual code to access the on-line reporting interface used to report their measurement results and to complete the related questionnaire. The questionnaire was used to extract relevant information related to sample preparation, measurements and laboratories (Annex 8).

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

5 Test item

5.1 Preparation

A total of twenty two kilograms of frozen peas were purchased at a local supermarket for the production of the test material

As a first step a feasibility study was carried out (i) to evaluate the uptake/adsorption of spiked heavy metals on peas during preparation, and (ii) to optimise the peas to brine ratio in the test item. Ten units of 210 mL glass jars were filled with frozen peas (~ 103 g) using a vibrating feeder; then water (~ 75 g) was added. An average peas / water ratio of 1.364 (± 0.014) was obtained. Based on this ratio 17 L of spiked brine solution were prepared in an acid-washed 20 L polyethylene (PE) drum. The brine had the following composition: HCl (0.01 mol L^{-1}) solution with traces of HF (25 µl L^{-1}) containing 0.3 mg L^{-1} As; 0.3 mg L^{-1} Cd; 0.2 mg L^{-1} Pb; 470 mg L^{-1} Sn and 6.9 g L^{-1} of NaCl. In order to achieve a high tin concentration, $SnCl_2 \cdot 2H_2O$ (purity ≥99.995 %) was used. All other elements

were of Certipur ICP standards quality from Merck Millipore (Brussels, Belgium). The brine had a pH of 2 with a salt content of about 0.7 % (w/v). The salt composition is comparable to the one found in commercial canned peas. Similarly 0.5 L of blank solution was prepared in an acid washed PE drum containing the acids and salt but without any spiked elements.

For the production of the main lot, 214 jars were acid cleaned using 2 % (w/v) nitric acid and rinsed with Type 1 water (Milli-Q Advantage 10 system). The jars were then dried in a clean cell and 209 jars were filled manually with ~99 g of frozen green peas. 75 mL of spiked brine solution were added using a BRAND-dispenser. The remaining 5 jars were filled with peas but instead of using the spiked brine solution, the blank solution mentioned above was used. All jars were then closed in a Lenssen Twist Off machine (Sevenum, NL) whereby sterilizable T.O. 66 lids were placed on the jars when transported through a chamber saturated with culinary grade steam. The lids were firmly kept in place by the resulting under-pressure in the head space after cooling down. The integrity of the seal could be confirmed by the "sensor" on the lid or by the "pop" sound of the lid at opening. Four of the jars filled with peas were equipped with Pt-1000 thermocouple probes of an E-Val Flex system (Ellab, Roedovre, DK) to monitor the core temperature in the jar during thermal sterilisation. All jars (including blanks) were thermally sterilised at 121 °C for 12 minutes using a JBTC Pilot AR092 autoclave (Sint Niklaas, BE). The jars were then placed for conditioning for 2 weeks at 60 °C in an Elbanton drying cabinet (Kerkdriel, NL). The elevated temperature was used to accelerate the migration of heavy metals from the liquid to the solid material and to reach equilibrium. The peas in the jars were intact after sterilisation and prior to dispatch.

5.2 Homogeneity and stability

Because of the two different sample preparation approaches foreseen, the homogeneity of both, drained peas and the solid/liquid composite, has been systematically investigated for all measurands. Assuming that the stability of the test item would not depend on the sample preparation approach, only the stability of the solid/liquid composite was monitored.

The measurements for the homogeneity and stability studies were performed by ALS Scandinavia AB (Luleå, Sweden) using inductively coupled plasma sector field mass spectrometry (ICP-SF/MS) after closed microwave digestion of 1 g of sample with a mixture of $HNO_3/H_2O_2/HF$.

The statistical treatment of data was performed at the IRMM.

Homogeneity was evaluated according to ISO 13528: 2005 [11]. Both, the drained product and the solid/liquid composite proved to be adequately homogeneous for all the investigated measurands (Annex 9.1 and 9.2).

The stability study was conducted following an isochronous design [12, 13]. The test material proved to be stable for 5 weeks for total As, Cd, Pb, Hg and Sn which is covering the life-time of the PT. (Annex 9.3). From previous experience (IMEP-107), it was

assumed that the homogeneity and stability of the total As mass fraction are representative of those of iAs.

The contributions from homogeneity (u_{bb}) and stability (u_{st}) to the uncertainty of the assigned value (u_{ref}) were calculated using SoftCRM [14]. The analytical results and the statistical evaluation of the homogeneity and stability studies are presented in Tables 1, 2 and Annex 9.

6. Assigned values and their uncertainties

6.1 Assigned value X_{ref}

The assigned values for the five measurands that were introduced/spiked into the test item (total As, Cd, Pb, Hg, Sn and iAs in canned peas) were determined by five laboratories, selected on their demonstrated measurement capabilities (later referred as expert laboratories):

- *IRMM – Institute for Reference Materials and Measurements, SID unit (Geel, Belgium)*
- *ALS Scandinavia AB (Luleå, Sweden);*
- *SCK-CEN – Studiecentrum voor Kernenergie (Mol, Belgium);*
- *Institut für Chemie, Bereich Analytische Chemie, Karl-Franzens Universität (Graz, Austria); and*
- *Department of Analytical Chemistry, Faculty of Chemistry, University of Barcelona, (Barcelona, Spain).*

Two sets of test items were sent to the expert laboratories: (i) for characterisation of the drained material and (ii) for characterisation of the solid/liquid composite. When applicable the draining protocol described in the AOAC official method 968.30 [15] was to be applied.

Expert laboratories were asked to use the method of analysis of their choice with no further metrological requirements. 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 estimated. Expert laboratories were not requested to report values for all measurands.

- IRMM used microwave digestion with a mixture of HNO_3/HF and applied isotope dilution inductively coupled plasma mass spectrometry (ID-ICP/MS).
- ALS used inductively coupled plasma sector field mass spectrometry (ICP-SF/MS) after closed microwave digestion of the sample with a mixture of HNO_3/H_2O_2/HF applying a modified EPA-method 200.8.
- SCK-CEN used neutron activation analysis (NAA).
- Institut für Chemie of the University of Graz used microwave digestion with HNO_3/H_2O_2 combined with ICP/MS analysis for total As determination. For iAs, samples were heated with a solution of CF_3COOH/ H_2O_2 (95°C for 60 min) and analyzed by HPLC-ICP/MS.

- Department of Analytical Chemistry in Barcelona used microwave digestion (temperature ramp to 95°C – total digestion time 30 min) with HNO_3/H_2O_2 and quantification of the iAs mass fraction via anion exchange chromatography LC-ICP/MS.

For this PT, the mean of the means provided by the expert laboratories was used to derive the assigned values (X_{ref}) according to ISO Guide 35 [16]. Values were reported for all analytes except for total Hg for which expert laboratories reported "less than" values ("< 0.002" mg kg^{-1} by ALS using ICP-SF/MS; "< 0.02" mg kg^{-1} by SCK using NAA). Therefore, no assessment of reported results is performed for total Hg in the two matrices investigated.

According to the assigned values the test item was not compliant with the legislation because of the high total Cd mass fraction for both sample preparation approaches and of the high total Sn content in the drained product.

6.2 Associated standard 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 for homogeneity (u_{bb}) and stability (u_{st}) in compliance with ISO Guide 35 [16]:

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

In all cases (except iAs in the drained product) the expert laboratories reported values with overlapping expanded measurement uncertainties (Table 1 and 2, Fig. 1). u_{char} was then calculated according to ISO 13528:2005 [11]:

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

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

For iAs in the drained product, expert laboratories reported values which did not overlap within their respective expanded measurement uncertainties (Table 1, Figure 1). u_{char} was then calculated according to ISO Guide 35 [16]:

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

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

Tables 1 and 2 present the results 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 and the standard deviation for the PT assessment σ.

Table 1 –*Measurement results reported by the expert laboratories for the* ***drained product****, assigned values, their associated expanded measurement uncertainties and target standard deviations for the PT assessment. All values in mg kg^{-1}.*

	Total As	Total Cd	Total Pb	Total Sn	Inorganic As
Expert 1	*0.111 ± 0.021*	*0.193 ± 0.033*	*0.114 ± 0.022*	*269 ± 37*	*0.106 ± 0.008*
Expert 2		*0.191 ± 0.009*	*0.117 ± 0.006*	*261.2 ± 14.7*	*0.09 ± 0.005*
Expert 3	*0.112 ± 0.015*			*296.43 ± 14.1*	
Expert 4	*0.129 ± 0.005*				
X_{ref}	**0.117**	**0.192**	**0.116**	**275.5**	**0.098**
u_{char}	0.005	0.011	0.007	8.8	0.008
u_{bb}	0.006	0.003	0.006	5.0	0.005
u_{st}	0.004	0.003	0.003	4.7	0.004
u_{ref}	0.009	0.012	0.009	11.1	0.010
U_{ref}	**0.018**	**0.023**	**0.019**	**22.3**	**0.020**
σ	0.026	0.038	0.025	33.1	0.022
σ (%)	22.0%	20.0%	22.0%	12.0%	22.0%

X_{ref} : assigned value; U_{ref}= k·u_{ref} , estimated associated expanded measurement uncertainty; k=2 coverage factor corresponding to a level of confidence of about 95 %.

Table 2 –*Measurement results reported by the expert laboratories for the* ***solid/liquid composite****, assigned values, their associated expanded measurement uncertainties and target standard deviations for the PT assessment. All values in mg kg^{-1}.*

	Total As	Total Cd	Total Pb	Total Sn	Inorganic As
Expert 1	*0.111 ± 0.02*	*0.131 ± 0.024*	*0.091 ± 0.017*	*185 ± 30*	*0.086 ± 0.006*
Expert 2		*0.129 ± 0.002*	*0.092 ± 0.001*	*209 ± 3*	*0.078 ± 0.005*
Expert 3	*0.127 ± 0.008*			*210 ± 10*	
Expert 4	*0.124 ± 0.005*				
X_{ref}	**0.121**	**0.130**	**0.092**	**201.2**	**0.082**
u_{char}	0.005	0.008	0.005	6.6	0.002
u_{bb}	0.003	0.002	0.002	3.2	0.002
u_{st}	0.004	0.002	0.002	3.4	0.003
u_{ref}	0.007	0.008	0.006	8.1	0.004
U_{ref}	**0.014**	**0.016**	**0.012**	**16.2**	**0.008**
σ	0.027	0.028	0.020	24.1	0.018
σ (%)	22.0%	21.5%	22.0%	12.0%	22.0%

X_{ref} : assigned value; U_{ref}= k·u_{ref} , estimated associated expanded measurement uncertainty; k=2 coverage factor corresponding to a level of confidence of about 95 %.

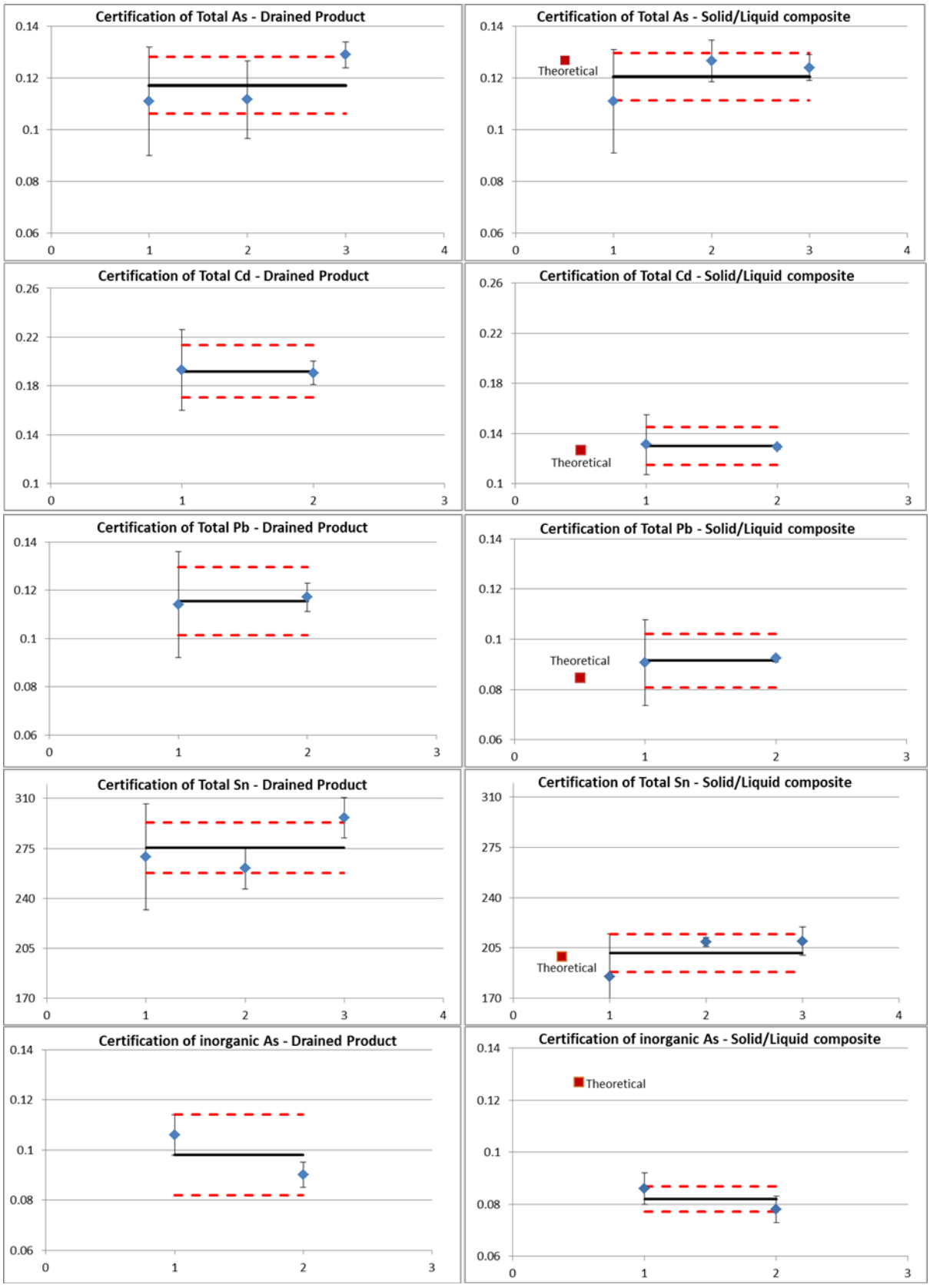


Figure 1: *The assigned values of the PT for the two types of samples (Drained product and Solid/Liquid composite). Blue rhombuses = reported values from the expert laboratories (± U_{cert}); Red square = theoretical concentration of the analyte derived from the spiking process; Black solid line = assigned value (X_{ref}); Red dashed lines = expanded assigned uncertainty interval (X_{ref} ± U_{ref}).*

6.3 Standard deviation of the proficiency test assessment σ

The standard deviation for proficiency test assessment (σ), for all measurands (except Sn) was calculated using the Horwitz equation modified by Thompson [18]. Being aware of specific difficulties associated to the determination of Sn and on the basis of previous experience (IMEP-108, IMEP-114, IMEP-29 and IMEP-39) the EURL-HM set σ to 12 % (instead of 7 % as predicted by Horwitz/Thomson).

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 [11]:

$$z = \frac{x_{lab} - X_{ref}}{\sigma} \qquad \text{Eq. 4}$$

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

where: x_{lab} is the measurement result reported by a participant;

u_{lab} is the standard uncertainty reported by a participant;

X_{ref} is the assigned value (assigned value);

u_{ref} is the standard uncertainty of the assigned value; and

σ is the standard deviation for proficiency assessment

The interpretation of the z- and ζ-score is done according to ISO 17043:2010 [10]:

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

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

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

The standard measurement uncertainty of the laboratory (u_{lab}) was obtained by dividing the reported expanded uncertainty by the reported coverage factor, *k*. When no uncertainty was reported, it was set to zero (u_{lab} = 0). When *k* was not specified, the reported expanded uncertainty was considered as the half-width of a rectangular distribution; u_{lab} was then calculated by dividing this half-width by $\sqrt{3}$, as recommended by Eurachem and CITAC [19].

Uncertainty estimation is not trivial, therefore an additional assessment was provided to each laboratory reporting 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} is set to the standard 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") 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 of homogeneity and stability of the test item. If those components are large, measurement uncertainties smaller than u_{min} are possible and plausible. If $u_{lab} > u_{max}$, (case "c") the laboratory may have overestimated the measurement uncertainty.

An evaluation of this statement can be made when looking at the difference of the reported value and the assigned value: if the difference is 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 Discussion regarding the test item (canned peas).

Preparing and distributing a complex test item such as canned peas is a demanding process. The first concern of a PT provider is that the selected test item must reach all the participants in the same, stable and homogeneous form representing reality as close as possible. In addition, Commission Regulation (EC) No 333/2007 laying down the methods of sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs [20] clearly states that: "[…] *In the case of inorganic tin, care shall be taken to ensure that all the material is taken into*

solution as losses are known to occur readily, particularly because of hydrolysis to insoluble hydrated Sn(IV) oxide species […]."

In this context during the production of the canned peas: i) the test item was incubated for 2 weeks at 60°C to accelerate the migration of the analytes from the brine into the peas [21-23] and b) a pH of 2 was used by means of HF and HCl in order to achieve complete solubilisation of the high concentration of Sn in the sample avoiding its precipitation as insoluble Sn oxides [24].

According to the values reported by the expert laboratories (Tables 1 and 2) 55 % of total As, 84 % of total Cd, 72 % of total Pb and 78 % of total Sn migrated from the brine into the peas. Whether this migration process had reached equilibrium or not by the time of the analysis was out of the scope of this exercise. However, the homogeneity and stability studies, the agreement between the expert laboratories on the assigned values, and the high percentages of satisfactory performances recorded from the participants, confirms the absence of detectable diffusion/adsorption phenomena. The analysis result was not affected even for the cases where participants rinsed the drained product (L028 and N014).

The reported standard uncertainty contributions due to homogeneity (u_{bb}) of the drained product are higher compared to those of the solid/liquid composite for all measurands (Tables 1 and 2). Since all samples were analysed by the same laboratory and in the same way, these differences can be attributed to the different sample preparation procedures alone.

The ranges reported by the expert laboratories are plotted (Figure 1) together with the assigned values ($X_{ref} \pm U_{ref}$), calculated as mentioned in paragraph 6.2. Taking into consideration the spiked concentrations of the analytes in the brine, their theoretical concentrations in the solid/liquid composite were calculated, plotted in the respective graphs (red squares) and used as qualitative indicators. It is important to note the good agreement between the theoretical and the assigned values for total As, Cd, Pb, and Sn.

In the case of iAs, the brine was spiked with As (V) in the form of arsenate. It was therefore assumed that the iAs concentration in the solid/liquid composite would be equal to the theoretical value (Figure 1). However, the iAs mass fraction in the drained product and in the solid liquid composite were found to be lower than the respective total As mass fractions. Especially for the solid/liquid composite the iAs mass fraction was 35 % lower than the theoretical one. This may indicate that the fraction of spiked iAs was transformed into different As species. Peas are legumes with high protein content and the formation of difficultly cleavable organo-arsenates is possible.

The expert laboratories were contacted, in an attempt to clarify this issue. Their answers are transcribed hereafter:

Expert 1. *"Two factors have to be considered to explain the observed discrepancy: spiking procedure and/or extraction of analytes from the matrix.*

The first one is always on discussion as interactions of spiked species can be different than the respective of the native ones, yielding changes in chemical behaviour.

Generally speciation methods are based on extraction procedures that preserve species integrity, so they do not use strong reagents that allow a complete dissolution. On the contrary elemental analysis uses strong reagents that allow complete dissolution but loss of information on species present in the sample.

Both factors together can explain the discrepancies, so unexpected interactions between added arsenic can modify extractions efficiencies and bring to low recoveries of added analytes".

Expert 2. *"If the arsenate is converted to a thio compound, it is not eluted from the Hamilton PRP-X100 column due to a strong interaction between the polymer backbone of this column and the hydrophobic thioarsenate"*.

7.3 Laboratory results and scorings

In total 127 laboratories registered to IMEP-118 of which 123 (36 countries) submitted results (Figure 2) and 113 of them answered the associated questionnaire. Thirty-six NRLs from 27 countries participated in this PT and all of them reported results.

From the participating laboratories 67 (54 %) analysed the drained product and 56 (46 %) the solid/liquid composite (NRLs: 21 and 15, respectively). Table 3 provides a general overview of reported results for each measurand in the two samples analysed by the NRLs and non-NRLs.

Different sample preparation approaches (drained product or solid/liquid composite) were used even by laboratories coming from the same country (17 countries). This may be attributed to unclear specific regulations or guidelines at European and/or national levels. European standard EN 13804:2013 recommends the following sample preparation strategy: "*Remove the sauce, brine or other packing medium which is normally not intended to be eaten, by draining. Include the sauce/juice when intended to be eaten*" [25]. The EURL-HM asked NRLs to provide additional information concerning the existence of national regulations on this matter. The majority of the laboratories verified that their sample preparation strategy is based on common sense, about what is intended for consumption and what not. This ambiguous situation of the analytical laboratories was further confirmed by the various comments collected in question 15 of the PT questionnaire (Annex 17). It is worth mentioning the comment of a participant:

> *"We analysed the drained product and liquid separately, reporting a composite value. This proficiency was a conundrum for us. Regulations did not specifically state to drain off liquid. Commission Regulation (EC) No 333/2007, Part B, Sampling Methods, B.2. Sampling Plans, Table 4 has the comment "The maximum levels for inorganic tin apply to the contents of each can" so we have reported composite."*

Only in Spain a protocol for handling and analysis of such samples clearly specifies that processed food, canned in its natural liquid or in sauces, should be drained before analysis while for processed canned fruits the liquid should be included in the analysis [26].

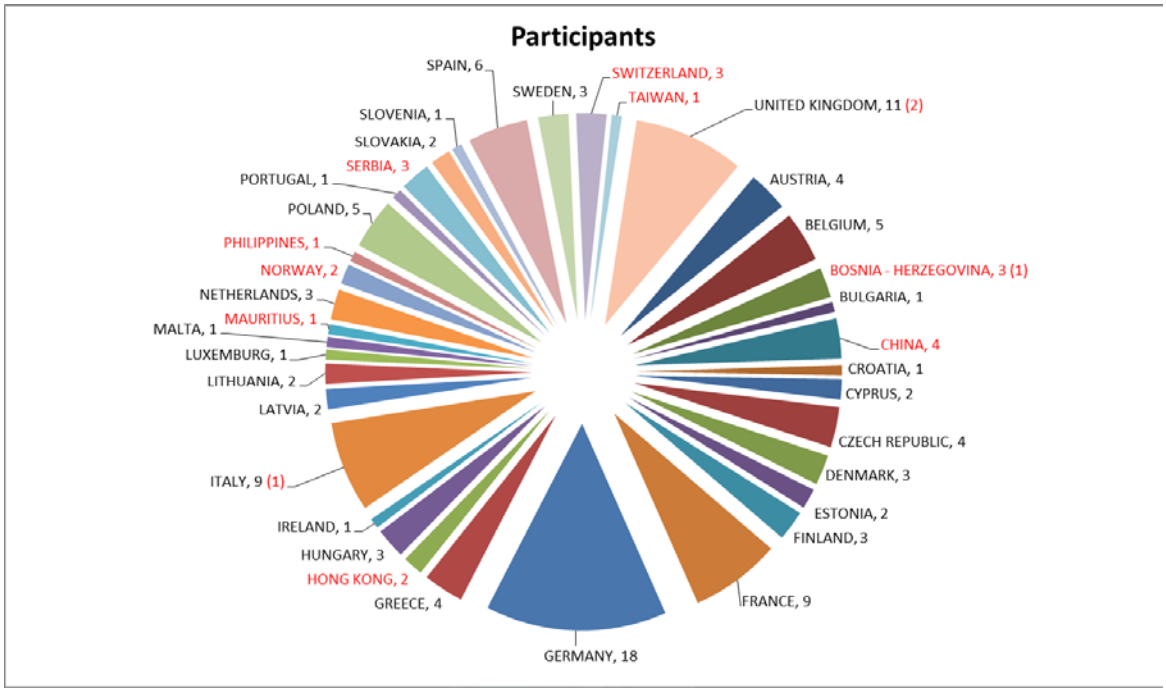


Figure 2: *Country distribution in IMEP-118 based on number of participants (127) having registered to the PT. Countries outside the European Union are depicted in red. The number of laboratories that did not return results is indicated in parentheses.*

Table 3- *Extracted information concerning the number of PT participants, the data obtained from them and their performance in the respective analysis.*

		Reported Values / less than		z - scores						ζ - scores					
				NRLs			non - NRLs			NRLs			non - NRLs		
		NRL	non-NRL	S	Q	U	S	Q	U	S	Q	U	S	Q	U
Drained Product	As	18 / 2	33 / 6	16(89%)	1(6%)	1(6%)	31(94%)	0	2(6%)	13(72%)	2(11%)	3(17%)	25(76%)	5(15%)	3(9%)
	Cd	20 / 0	46 / 0	20(100%)	0	0	41(91%)	2(4%)	2(4%)	20(95%)	1(5%)	0	38(84%)	1(2%)	6(13%)
	Pb	21 / 0	43 / 2	20(95%)	1(5%)	0	39(91%)	0	4(9%)	17(81%)	2(10%)	2(10%)	38(88%)	1(2%)	4(9%)
	Hg	4 / 15	5 / 34												
	Sn	15 / 0	35 / 0	12(80%)	2(13%)	1(7%)	25(71%)	5(14%)	5(14%)	10(67%)	3(20%)	2(13%)	19(54%)	4(11%)	12(34%)
	iAs	12 / 0	7 / 2	10(83%)	1(8%)	1(8%)	6(86%)	0	1(14%)	9(75%)	2(17%)	1(8%)	5(71%)	1(14%)	1(14%)
Solid/Liquid composite	As	14 / 0	37 / 2	12(86%)	1(7%)	1(7%)	30(81%)	4(11%)	3(8%)	8(57%)	4(29%)	2(14%)	29(78%)	3(8%)	5(14%)
	Cd	15 / 0	39 / 2	12(80%)	2(13%)	1(7%)	36(92%)	3(8%)	0	11(73%)	0	4(27%)	36(92%)	1(3%)	2(5%)
	Pb	15 / 0	40 / 0	13(87%)	1(7%)	1(7%)	34(85%)	2(5%)	4(10%)	11(73%)	1(7%)	3(20%)	31(78%)	4(10%)	5(13%)
	Hg	3 / 9	11 / 27												
	Sn	11 / 0	35 / 1	9(82%)	1(9%)	1(9%)	27(77%)	4(11%)	4(11%)	6(55%)	1(9%)	4(36%)	26(74%)	2(6%)	7(20%)
	iAs	6 / 1	16 / 2	5(83%)	1(17%)	0	12(75%)	3(19%)	1(6%)	2(33%)	2(33%)	2(33%)	7(44%)	6(38%)	3(19%)

Where S,Q,U: Satisfactory, Questionable, Unsatisfactory.

Taking into consideration the differences in assigned values of the two sample preparation approaches (Tables 1 and 2) it is conceivable that contradictory assessments of compliance of the test item may be reached. In the case of total Sn the test item may be declared either compliant or not, depending on the applied sample preparation protocol.

Annexes 10 – 15 present the reported results as tables and as graphs. The graphs include the corresponding Kernel density plots, obtained using the software available from the Statistical Subcommittee of the Analytical Methods Committee of the UK Royal Society of Chemistry [27].

The overall performance of the participants regarding the z- and ζ-scores, is summarised in Table 3 and Figure 3. The participants performed satisfactorily in this exercise for the determination of total As, Cd and Pb for both sample preparation approaches (drained and solid/liquid composite). Only 32 laboratories (13 NRLs) reported results for all five measurands from which 20 performed satisfactorily for all of them (9 NRLs). In the case of total Sn and iAs, there is room for further improvement in terms of performance and number of laboratories performing the analysis.

In all cases, the percentage of satisfactory ζ-scores is lower than that of the satisfactory z-scores (in the case of iAs: 39 % and 74 % satisfactory ζ- and z-scores, respectively).

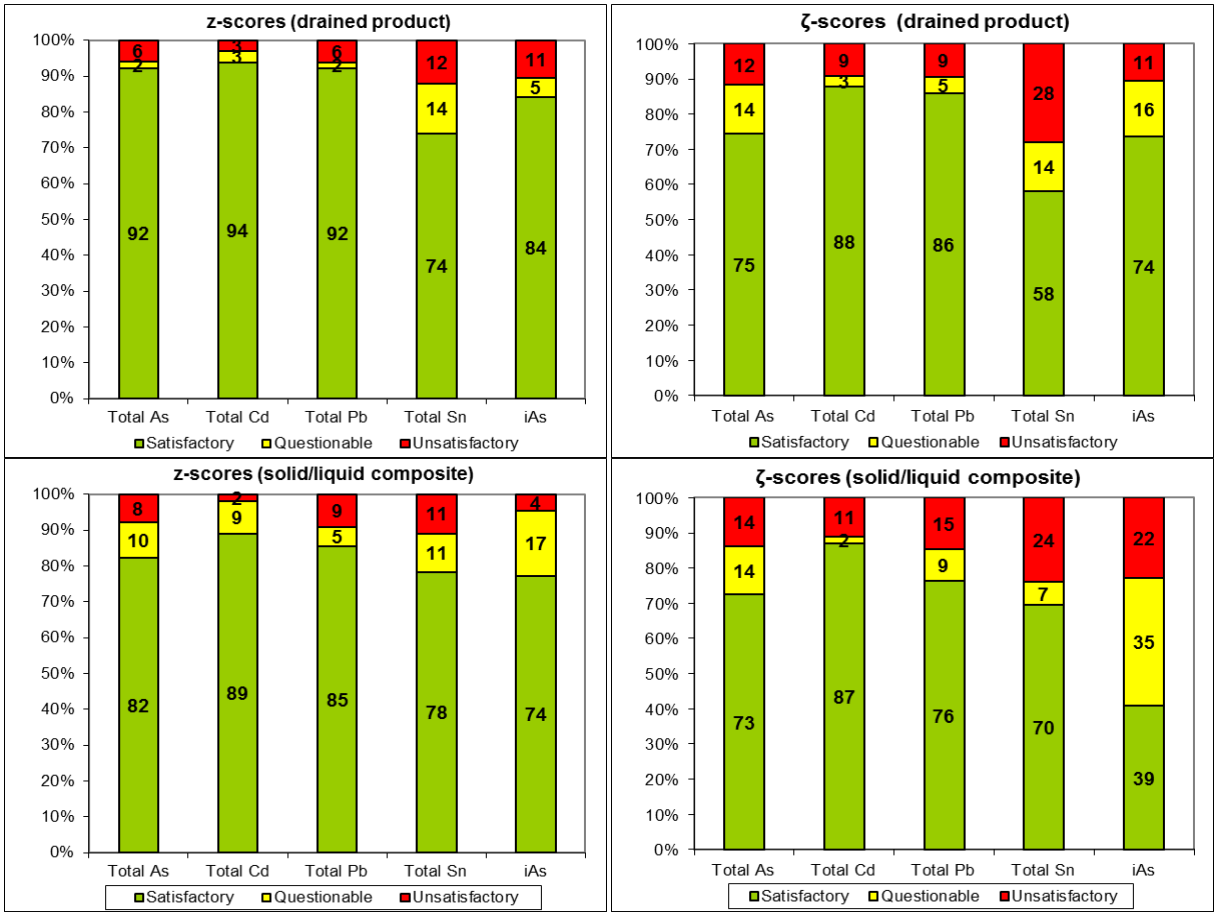


Figure 3: *Percentages of laboratories with satisfactory, questionable and unsatisfactory performance for the analysis of the drained product and the solid/liquid composite.*

The uncertainty assessment ("a": $u_{ref} \leq u_{lab} \leq \sigma$; "b": $u_{lab} < u_{ref}$; and "c": $u_{lab} > \sigma$) is presented in Table 4 and Annexes 10-15. Most of the NRL laboratories reported realistic measurement uncertainty estimates (case "a") - from 47 to 83 % - compared to non-NRLs (from 36 to 65 %). On the other hand, only a few NRLs (below 13 %) reported overestimated uncertainties (case "c") for the five analytes in the two matrices.

Table 4 – *Uncertainty assessment (in terms of "a", "b", "c") for the participating NRLs and non-NRLs, where* "a": $u_{ref} \leq u_{lab} \leq \sigma$; "b": $u_{lab} < u_{ref}$; and "c": $u_{lab} > \sigma$

Uncertainty assessment		A		b		c	
		NRLs	Non-NRLs	NRLs	Non-NRLs	NRLs	Non-NRLs
Drained product	As	12(67%)	12(36%)	6(33%)	17(55%)	0	4(12%)
	Cd	15(71%)	20(44%)	5(24%)	18(40%)	1(5%)	7(16%)
	Pb	17(81%)	19(44%)	3(14%)	16(37%)	1(5%)	8(19%)
	Sn	12(80%)	16(46%)	3(20%)	17(49%)	0	2(6%)
	iAs	6(50%)	2(29%)	6(50%)	4(57%)	0	1(14%)
Solid/liquid composite	As	8(57%)	19(51%)	5(36%)	12(32%)	1(7%)	6(16%)
	Cd	7(47%)	24(62%)	6(40%)	12(31%)	2(13%)	3(8%)
	Pb	8(53%)	26(65%)	5(33%)	9(23%)	2(13%)	5(13%)
	Sn	6(55%)	17(49%)	5(45%)	13(37%)	0	5(14%)
	iAs	5(83%)	10 (59%)	1(17%)	3(18%)	0	3(18%)

Table 5 - *Approaches used by the participants in IMEP-118 to estimate the uncertainty of their measurements. Multiple selections were possible.*

Approach followed for uncertainty calculation	Number of labs.
Uncertainty budget (ISO-GUM), validation	19
Known uncertainty of the standard method (ISO 21748)	3
Uncertainty of the method (in-house)	71
Measurement of replicates (precision)	44
Estimation based on judgment	4
Use of intercomparison data	25
Other: Based on certified reference material: 1 lab Horwitz equation: 7 labs Based on certified reference material: 3 labs FDA Elemental Analysis Manual (EAM): 1 lab Nordtest TR 537: 1 lab Control samples, spiking and recovery: 3 labs	16

Several approaches were used to evaluate measurement uncertainties (Table 5). The majority of the NRLs (31) report uncertainty to their customers, while 3 do not. Forty-one non-NRLs report uncertainty to their customers while 38 do not. Laboratories which report measurement uncertainties to their customers performed better in terms of measurement uncertainty estimation (67 % obtained "a") when compared to the laboratories that do not report uncertainty (41 % obtained "a").

For all the measurands considered in this PT the laboratories reporting "less than" and "0" values were not included in the evaluation. However, reported "less than" values were compared with the corresponding $X_{ref} - U_{ref}$ values. If the reported limit value is lower than the corresponding $X_{ref} - U_{ref}$, this statement is considered incorrect (flagged in red in Annexes 10 - 15), since the laboratory should have detected the respective element. In this exercise three laboratories reported incorrect "less than" values: - L087 (0.06 mg kg^{-1} for iAs in the drained product), L031 (0.1 mg kg^{-1} for total As in the solid liquid composite) and L047 (0.01 mg kg^{-1} for total Cd in the solid liquid composite).

7.4 Discussion on the reported results

No direct correlation could be found between the analytical methods used by the participants and the quality of their reported results. Regardless of the satisfactory performance exhibited by the majority of the participants a critical factor that could potentially influence the quality of the reported results is the sample preparation, namely the homogenization of the sample (e.g. use of lyophilisation, knife milling, hand blender, ceramic homogenizer etc. full list included in annex 16). The use of improper homogenizing means could lead to sample contamination and to overestimation of the concentrations.

Concentrating on the reported results by the participants the main observations are summarised hereafter.

For the total As mass fraction the performance of the participants analysing the drained product was better than those analysing the solid/liquid composite (92 *vs*. 82%). A tendency to underestimate the total As mass fraction is observed in the figure of Annex 10 which may be attributed to the formation of thio-bound As(V) species difficult to cleave even under the harsh mineralization conditions used for total As determination.

The iAs mass fraction was analysed only by 41 laboratories (18 NRLs and 23 non-NRLs). Satisfactory performance was achieved by 84 % of all the laboratories analysing the drained product and by 74 % analysing the solid/liquid composite. For the NRLs the respective percentage was 83 % for both approaches. The figure of Annex 15 shows that the reported results are in good agreement with the assigned value of the drained product. For the solid liquid composite a tendency of overestimation is identified by the participants. According to the expert laboratories, the use of strong reagents for the analysis may lead to loss of information on the As species present in the sample and to overestimation of the actual value.

For the total Cd and total Pb mass fractions, the participants performed satisfactorily. Although the majority of the reported results are in good agreement with the assigned value for cadmium in both matrices (Annex 11), they are slightly higher in the case of lead (Annex 12). The PT provider has full confidence in the assigned value for total Pb based on a set of results obtained using the ID-ICP/MS method. In the case of the solid/liquid composite, the assigned value is in good agreement with the spiking/theoretical value (Figure 3). Lead contamination in laboratories may contribute to the positively biased results.

For the total Hg mass fraction, both certifiers reported "less than" values (0.02 and 0.002 mg kg^{-1}). However, Annex 13 shows that 23 participants (7 NRLs) reported values for total Hg (9 for the drained product and 14 for solid/liquid composite). Twelve participants reported values that were well above the "less than" 0.002 mg kg^{-1} (N010, N025, L051, L053, L069, L072, L092, L100, L109, L114, L118, L126). Two participants (L069, L104) reported values that were at the level of the reported LODs (Annex 16). Finally two participants (L055, L126) reported total Hg values lower that their reported LODs.

For the total Sn mass fraction, 96 participants reported results (26 NRLs and 70 non-NRLs). The majority of the participants (74 and 78 %) performed satisfactorily for the analysis of the drained product and solid/liquid composite respectively, (80 and 81 % for the NRLs). However, a larger dispersion of results than for other measurands was observed. For example, in the case of the drained product, results ranged from 2 to 315 mg kg^{-1}. This could be attributed to the combination of inherent analytical issues [28, 29] with the lack of appropriate reference material available on the market.

7.5 Discussion on the information extracted from the questionnaire

The associated questionnaire was answered by 113 of the participants. Laboratories were asked to report LODs of the methods that they used for the determination of the six measurands. The LODs together with the respective techniques and general experimental conditions used are presented in Annex 16. Large discrepancies in reported LODs were observed even among laboratories using the same technique.

Thirty-five laboratories corrected their results for recovery while 78 did not. For the whole population of participants the recoveries reported ranged from 20 to 130 %. NRLs applied recoveries in the range of 60 - 130 %. Laboratories that reported recoveries lower than 80 % and higher than 120 % must be aware that such recoveries indicate that the analytical method used is significantly biased and that corrective actions should be undertaken. The 34 participants that reported to have calculated a recovery factor applied one or several of the options shown in Table 6.

Table 6 - *Methods applied by the laboratories to determine the recovery factors of the exercise. Multiple selections were possible.*

How did you determine the recovery factor?		**Number of labs.**
adding a known amount of the same analyte to be measured (spiking)		18
using a certified reference material		19
Other : (labs)	- "Using internal standard or RM" – (2) - "Use of Interlaboratory Comparison samples" – (1) - "Control card dates" – (1)	4

The participants (107) answered that they are accredited for one or more of the measurands under study. The performance of the accredited laboratories was slightly better than for the non-accredited ones (accredited/non-accredited: 89 /84 % for total As, 93 / 84 % for total Cd, 90 / 83 % for total Pb, 81 / 62 % for total Sn and 82 to 75 % for iAs). All laboratories which answered to the questionnaire except 3 have a quality system in place based on ISO 17025. In five cases the quality system is also based on ISO 9000. The majority of the laboratories regularly take part in PTs. Seventeen out of 65 unsatisfactory scores of IMEP-118 (26 %) were reported by laboratories that do not participate in ILCs.

In the case of total As analysis, 50% of the laboratories having unsatisfactory performance, stated to have limited (or non-existing) experience in this specific analysis. For all the other measurands no correlation between performance and experience existed.

8. Compliance assessment of the test item

According to the assigned values (Tables 1 and 2) the test item is not compliant with the maximum levels (ML) given in Regulation (EC) No 1881/2006 because of the high concentration of total Cd (above the maximum legal limit for legumes) in both the drained product and the solid/liquid composite and because of the high concentration of total Sn in the drained product (above maximum legal limits for canned foods). The concentration of total Sn in the solid/liquid composite is equal to the ML set by the legislation (taking into consideration the uncertainty of the assigned value). Seventy-two laboratories declared the test item non-compliant with the legislation for several reasons (Table 7). Thirty-two laboratories (including 6 NRLs) reported that the sample item was compliant with the legislation and could be consumed, while 19 participants (of which 4 NRLs) did not answer to the question.

Table 7 - *Question 14 of the questionnaire: Considering the reported level for the investigated trace elements in the specific food matrix (canned peas) and the maximum levels of certain contaminants in foodstuffs (Commission Regulation (EC) No 1881/2006) would you accept the present sample?*

<table>
<tr><th></th><th colspan="3">Compliance of the test item</th></tr>
<tr><td>Yes (test item compliant)</td><td colspan="3">32 (6)</td></tr>
<tr><td>Did not answer</td><td colspan="3">19 (4)</td></tr>
<tr><td rowspan="3">No (with reason)</td><td rowspan="3">Overall
72 (26)</td><td>35 (14): because of Sn</td><td>6 (3): Sn, Cd, Pb
15 (6):Sn, Cd
1: Sn, Pb
13 (5): Sn</td></tr>
<tr><td colspan="2">32 (10): because of Cd and/or Pb</td></tr>
<tr><td colspan="2">5 (2): no reason</td></tr>
</table>

Numbers in brackets indicate the respective number of NRLs.

According to Commission Regulation, (EC) No 333 / 2007 [20] a sample should be considered as compliant with the legislation when *" the analytical result of the laboratory does not exceed the respective maximum level as laid down in Regulation (EC) No 1881/2006 taking into account the expanded measurement uncertainty"*.

Figure 4 presents the conformity assessment of participants having declared the test item as compliant or not compliant based on their total Sn results. The red horizontal line represents the ML given in Commission Regulation (EC) No 1881/2006 while the red boxes highlight laboratories having made a wrong conformity assessment in contradiction with their reported results / ranges.

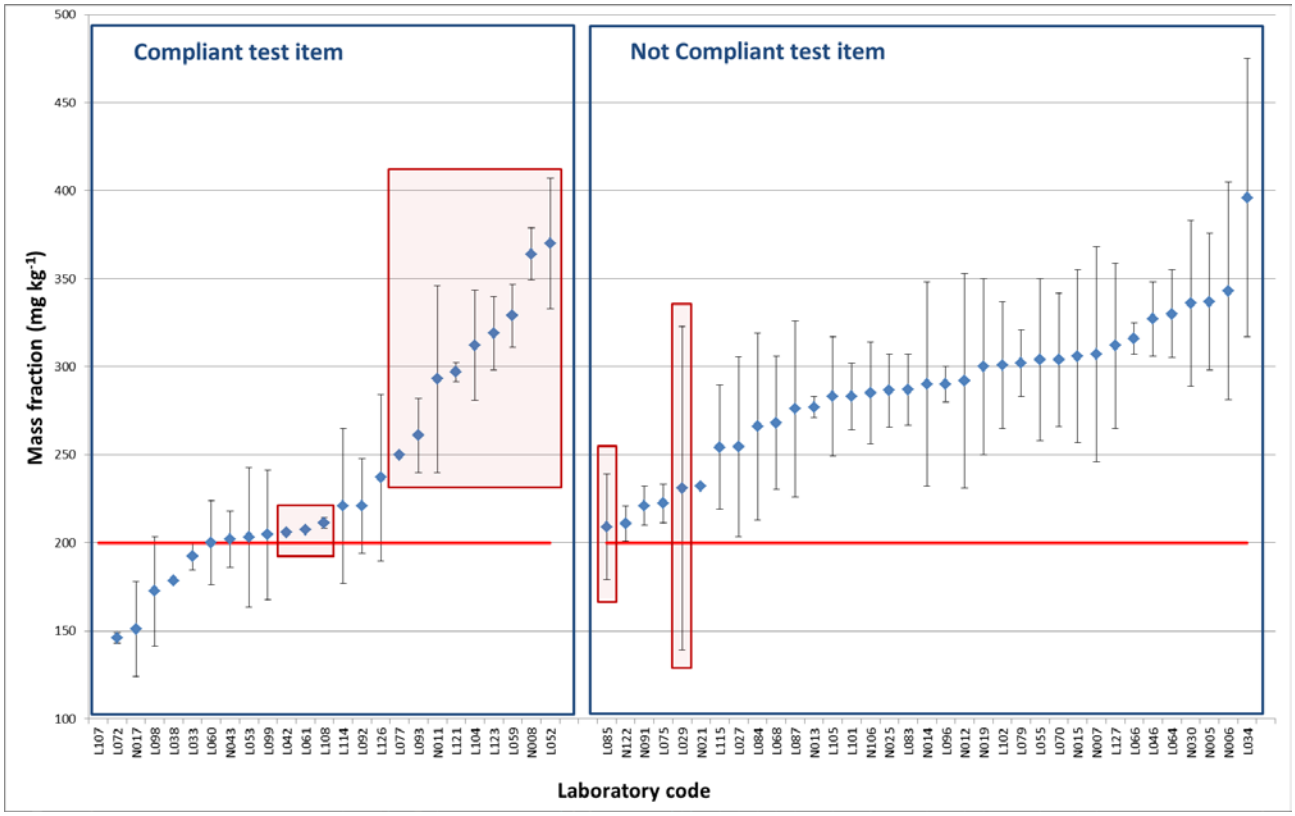


Figure 4: *Responses of the participants to Q 14 of the questionnaire concerning compliance of the test item in correlation to their respective reported results / ranges for total Sn (diamonds). The red line corresponds to the ML set by regulation (200 mg kg^{-1}). The red boxes highlight laboratories having made a wrong conformity assessment in contradiction with their reported results / ranges.*

8 Conclusions

The outcome of IMEP-118 clearly identified that guidelines are needed on the sample preparation protocol to be used when analysing canned food, drained product or solid/liquid composite. According to information collected from the NRLs and OCLs taking part in this PT, only Spain has such a guidance document.

IMEP-118 evaluated how the reporting laboratories have assessed compliance with the maximum limits given in Regulation (EC) No 1881/2006. Although the test item was not compliant with legislation, 32 laboratories (of which 6 NRLs) would have allowed the product to be placed on the European market.

The performance of the participating laboratories to determine the total amount (mass fraction) of As, Cd, Pb, Hg, Sn and inorganic As was satisfactory for both sample preparation approaches. In the case of total Sn and iAs there is room for improvement, regarding not only the performance but also the number of laboratories carrying out the analyses (only 33 % of the participants reported values for iAs).

Once again the need for an extra effort was identified in the evaluation of uncertainties associated to the results, as the number of questionable and unsatisfactory ζ-scores is systematically higher than those of z-scores for all analytes. NRLs performed better than non-NRLs estimating the uncertainties of the measurands. Measurement uncertainty is of paramount importance in cases of litigation and therefore the capability of control laboratories to estimate it correctly is a fundamental requirement.

Another area in which action must be taken relates to the determination of the LOD of the method of analysis used. Significant discrepancies were observed for the limits of detections reported, even for similar analytical methods. There is a clear confusion between the LOD of the method and the instrumental LOD.

9 Acknowledgements

C. Contreras from the Standards for Innovation and Sustainable Development (SID) Unit of the IRMM is acknowledged for her support in the isochronous study. F. Ulberth and J. Charoud-Got are also acknowledged for reviewing the manuscript.

The laboratories participating in this exercise, listed in Table 9, are kindly acknowledged.

***Table 9**: Participating Laboratories in IMEP-118*

Organisation	Country
AGES GmbH	AUSTRIA
LVA GmbH	AUSTRIA
ILV Kärnten	AUSTRIA
MA 38 - Lebensmitteluntersuchungsanstalt der Stadt Wien	AUSTRIA
CODA-CERVA	BELGIUM
Scientific Institute of Public Health Belgium	BELGIUM
FAVV	BELGIUM
INAGRO	BELGIUM
Laboratorium ECCA NV	BELGIUM
LOVAP NV	BELGIUM

FEDERAL AGROMEDITERRANEAN INSTITUTE OF MOSTAR	BOSNIA - HERZEGOVINA
Federal Institute of Agriculture	BOSNIA - HERZEGOVINA
Central Laboratory for Chemical Testing and Control /CLCTC/	BULGARIA
Beijing Municipal Center for Diseases Prevention and Control	CHINA
Guangdong Provincial center for disease control an prevention	CHINA
China National Center for Food Safety Risk Assessment	CHINA
Shenzhen Center for Disease Control & Prevention,	CHINA
Croatian National Institute of Public Health	CROATIA
STATE GENERAL LABORATORY	CYPRUS
Aristos Loucaides Chemical Laboratory Ltd.	CYPRUS
State Veterinary Institute Olomouc	CZECH REPUBLIC
Ustav pro vysetrovani potravin spol. s r.o.	CZECH REPUBLIC
Statni veterinarni ustav Praha	CZECH REPUBLIC
LITOLAB, spol. s r.o.	CZECH REPUBLIC
National Food Institute (DTU Food)	DENMARK
Danish Veterinary and Food Administration	DENMARK
Eurofins Environment A/S	DENMARK
Agricultural Reasearch Centre	ESTONIA
Veterinary and Food laboratory	ESTONIA
Finnish Customs Laboratory	FINLAND
City of Jyväskylä	FINLAND
Metropolilab ltd.	FINLAND
Laboratoire SCL de Bordeaux	FRANCE
ANSES - French Agency for Food, Environmental and Occupational Health and Safety	FRANCE
LASAT	FRANCE
Eurofins Analytics France	FRANCE
SGS MULTILAB	FRANCE
INOVALYS	FRANCE
CAMP 66	FRANCE
La drôme laboratoire	FRANCE
LABORATOIRE DE L'ENVIRONNEMENT ET DE L'ALIMENTATION	FRANCE
Federal Office for Consumer Protection and Food Safety (BVL)	GERMANY
Chemisches und Veterinäruntersuchungsamt Westfalen	GERMANY
Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit	GERMANY
Lebensmittel- und Veterinärinstitut Oldenburg	GERMANY
LAV Sachsen-Anhalt	GERMANY
Landesuntersuchungsanstalt Sachsen	GERMANY
Landesamt fuer Umwelt- und Arbeitsschutz	GERMANY
Landeslabor Schleswig-Hplstein (LSH)	GERMANY
Landesbetrieb Hessisches Landeslabor	GERMANY
TLV Bad Langensalza	GERMANY
Dr. Graner & Partner GmbH	GERMANY
LAVES	GERMANY
Chemisches Labor Dr. Wirts + Partner GmbH	GERMANY
Bayer. Landesamt f. Gesundheit	GERMANY
GLUmbH	GERMANY
Landesuntersuchungsamt für Chemie, Hygiene und Veterinärmedizin	GERMANY
CVUA-OWL	GERMANY
Office of Consumer Protection	GERMANY
REGIONAL CENTRE OF PLANT PROTECTION AND QUALITY CONTROL OF MAGNISSIA	GREECE
AGENT	GREECE
General Chemical State Laboratory	GREECE
GENERAL CHEMICAL STATE LABORATORY	GREECE
Enviro Labs Limited	HONG KONG
ALS Technichem (HK) Pty Ltd	HONG KONG
National Food Chain Safety Office	HUNGARY
National Food Chain Safety Office	HUNGARY
National Food Chain Safety Office	HUNGARY
HEALTH SERVICE EXECUTIVE	IRELAND
ISTITUTO ZOOPROFILATTICO SPERIMENTALE PIEMONTE, LIGURIA E VALLE D'AOSTA	ITALY
ISS- Istituto Superiore di Sanità	ITALY
ARPA FVG	ITALY
PROVINCIA AUTONOMA DI BOLZANO	ITALY
ISTITUTO ZOOPROFILATTICO SPERIMENTALE DELLA PUGLIA E BASILICATA	ITALY
Istituto Zooprofilattico Sperimentale della Lombardia ed Emilia Romagna (IZSLER)	ITALY
ARPA PIEMONTE	ITALY
Laboratorio di Prevenzione di Milano	ITALY
Institute of Food Safety, Animal Health and Environment	LATVIA

Latvian Certification Centre Ltd.	LATVIA
National Food and Veterinary Risk Assessment Institute	LITHUANIA
JSC Labtarna	LITHUANIA
Environmental Health Directorate	MALTA
Princes Tuna (Mauritius) Limited	MAURITIUS
RIKILT	NETHERLANDS
Food & Consumer Products Safety Authority	NETHERLANDS
TNO Triskelion	NETHERLANDS
LabNett Skien	NORWAY
NIFES	NORWAY
SentroTek Corporation	PHILIPPINES
National Institute of Public Health-National Institute of Hygiene	POLAND
The National Veterinary Research Institute	POLAND
Wojewódzka Stacja Sanitarno-Epidemiologiczna w Krakowie	POLAND
Wojewódzka Stacja Sanitarno - Epidemiologiczna	POLAND
Oddział Laboratoryjny Tarnobrzeg WSSE Rzeszów	POLAND
ControlVet	PORTUGAL
Institute of Public Health of Vojvodina	SERBIA
Jugoinspekt Beograd ad	SERBIA
JUGOINSPEKT-NOVI SAD	SERBIA
Veterinary and food institute in Košice	SLOVAKIA
Regional Authority of Public Health	SLOVAKIA
National Laboratory of Health, Environment and Food	SLOVENIA
Laboratorio Arbitral Agroalimentario	SPAIN
ainia	SPAIN
GOBIERNO DEL PRINCIPADO DE ASTURIAS - CONSEJERÍA DE SANIDAD	SPAIN
CENTRO DE SALUD PÚBLICA DE ALICANTE	SPAIN
Laboratory of the Public Health Agency of Barcelona	SPAIN
MADRID SALUD	SPAIN
National Food Agency	SWEDEN
ALS Scandinavia	SWEDEN
Eurofins environment testing AB	SWEDEN
Coop Central Laboratory, nominated by SAS	SWITZERLAND
SQTS - Swiss Quality Testing Services	SWITZERLAND
Labor der Urkantone	SWITZERLAND
A.G.V.PRODUCTS.CORP.	TAIWAN
Minton, Treharne and Davies Limited.	UNITED KINGDOM
Food and Environment Research Agency	UNITED KINGDOM
TAYSIDE SCIENTIFIC SERVICES	UNITED KINGDOM
Lancashire County Scientific Services	UNITED KINGDOM
Staffordshire County Council	UNITED KINGDOM
Worcestershire Scientific Services	UNITED KINGDOM
Glasgow Scientific Services	UNITED KINGDOM
Hampshire County Council	UNITED KINGDOM
Kent County Council	UNITED KINGDOM

10. Abbreviations

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

11 References

1. Hajeb, P., Sloth J.J., Shakibazadeh S., Mahyudin N.A.Afsah-Hejri L., *Toxic Elements in Food: Occurrence, Binding, and Reduction Approaches.* Comprehensive Reviews in Food Science and Food Safety, 2014. 13(4): p. 457-472.

2. Kassouf, A., Chebib H., Lebbos N.Ouaini R., *Migration of iron, lead, cadmium and tin from tinplate-coated cans into chickpeas.* Food Additives and Contaminants - Part A Chemistry, Analysis, Control, Exposure and Risk Assessment, 2013. 30(11): p. 1987-1992.

3. Amjadi, M., Manzoori J.L.Hamedpour V., *Optimized Ultrasound-Assisted Temperature-Controlled Ionic Liquid Microextraction Coupled with FAAS for Determination of Tin in Canned Foods.* Food Analytical Methods, 2013. 6(6): p. 1657-1664.

4. LaKind, J.S., *Can coatings for foods and beverages: Issues and options.* International Journal of Technology, Policy and Management, 2013. 13(1): p. 80-95.

5. Mol, S., *Determination of trace metals in canned anchovies and canned rainbow trouts.* Food and Chemical Toxicology, 2011. 49(2): p. 348-351.

6. Obeid, P.J., Saliba C., Younis M., Aouad S.El-Nakat J., *Comparative analysis of lead and cadmium levels in various brands of canned and processed meat products in Lebanon.* WIT Transactions on Ecology and the Environment, 2013. 170: p. 135-146.

7. Russo, R., Voi A.L., De Simone A., Serpe F.P., Anastasio A., Pepe T., Cacace D.Severino L., *Heavy metals in canned tuna from Italian markets.* Journal of Food Protection, 2013. 76(2): p. 355-359.

8. CICAD 65, on *Tin and inorganic tin compounds*, the *Concise International Chemical Assessment Documents*. 2005. p. i-73.

9. Commission Regulation (EC) No 1881/2006 *setting maximum levels for certain contaminants in foodstuffs, issued by the European Commission, .* Official Journal of the European Union, L364/5 (2006).

10. ISO 17043:2010, I., *Conformity assessment - General requirements for proficiency testing.* issued by ISO-Geneva (CH), International Organization for Standardization.

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

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

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

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

15. AOAC method 968.30, *Canned Vegetables, Drained Weight, Procedure 1968.* AOAC Official Methods of Analysis, 1995. chapter 42: p. 1-2.

16. ISO Guide 35, *Reference Materials - General and statistical principles for certification (2006), issued by ISO-Geneva (CH).*

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

18. Thompson, M., Analyst, 2000. 125: p. 385-386.

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

20. Commission Regulation, *(EC) No 333 / 2007 laying down the methods of sampling and analysis for the official control of the levels of led, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs.* Official Journal of the European Union, L88/29 (2007).

21. Buculei, A., Amariei S., Oroian M., Gutt G., Gaceu L.Birca A., *Metals migration between product and metallic package in canned meat.* LWT - Food Science and Technology, 2013.

22. Knápek, J., Herman V., Buchtová R.Vošmerová D., *Determination of tin in canned foods by atomic absorption spectrometry.* Czech Journal of Food Sciences, 2009. 27(SPEC. ISS.): p. S407-S409.

23. Trandafir, I., Nour V.Ionica M.E., *Determination of tin in canned foods by inductively coupled plasma-mass spectrometry.* Polish Journal of Environmental Studies, 2012. 21(3): p. 749-754.

24. Blunden, S.Wallace T., *Tin in canned food: A review and understanding of occurrence and effect.* Food and Chemical Toxicology, 2003. 41(12): p. 1651-1662.

25. 2013, E., *Foodstuffs - Determination of elements and their chemical species - General considerations and specific requirements.* EUROPEAN STANDARD 2013.

26. Protocolo de preparacion previa de muestras de alimentos, piensos y sus materias primas para analisis de contaminantes metalicos. Ministerio de Agricultura, Alimentacion y medio ambiente. 2012, *Spain*.

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

28. Perring, L.Basic-Dvorzak M., *Determination of total tin in canned food using inductively coupled plasma atomic emission spectroscopy.* Analytical and Bioanalytical Chemistry, 2002. 374(2): p. 235-243.

29. Boutakhrit, K., Crisci M., Bolle F.van Loco J., *Comparison of four analytical techniques based on atomic spectrometry for the determination of total tin in canned foodstuffs.* Food Additives and Contaminants - Part A Chemistry, Analysis, Control, Exposure and Risk Assessment, 2011. 28(2): p. 173-179.

Annexes

Annex 1: Invitation letter to NRLs

Ref. Ares(2014)641686 - 10/03/2014

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

Geel, 10 March 2014
JRC.D.5/PRO/IF/acs/ARES

Subject : IMEP-118: Total As, Cd, Pb, Hg, Sn and iAs in canned food

Dear National Reference Laboratory representative,

We would like to invite you on behalf of the EURL Heavy Metals in Feed and Food, to participate in the Proficiency Test IMEP-118 for the **"Determination of total As, Cd, Pb, Hg, Sn and iAs in canned food"**.

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=1165

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 14 April 2014**. Samples will be sent to participants during the second half of April 2014. The deadline for submission of results is **6 June 2014**.

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

Yours sincerely

Dr. Ioannis Fiamegkos
IMEP-118 Coordinator

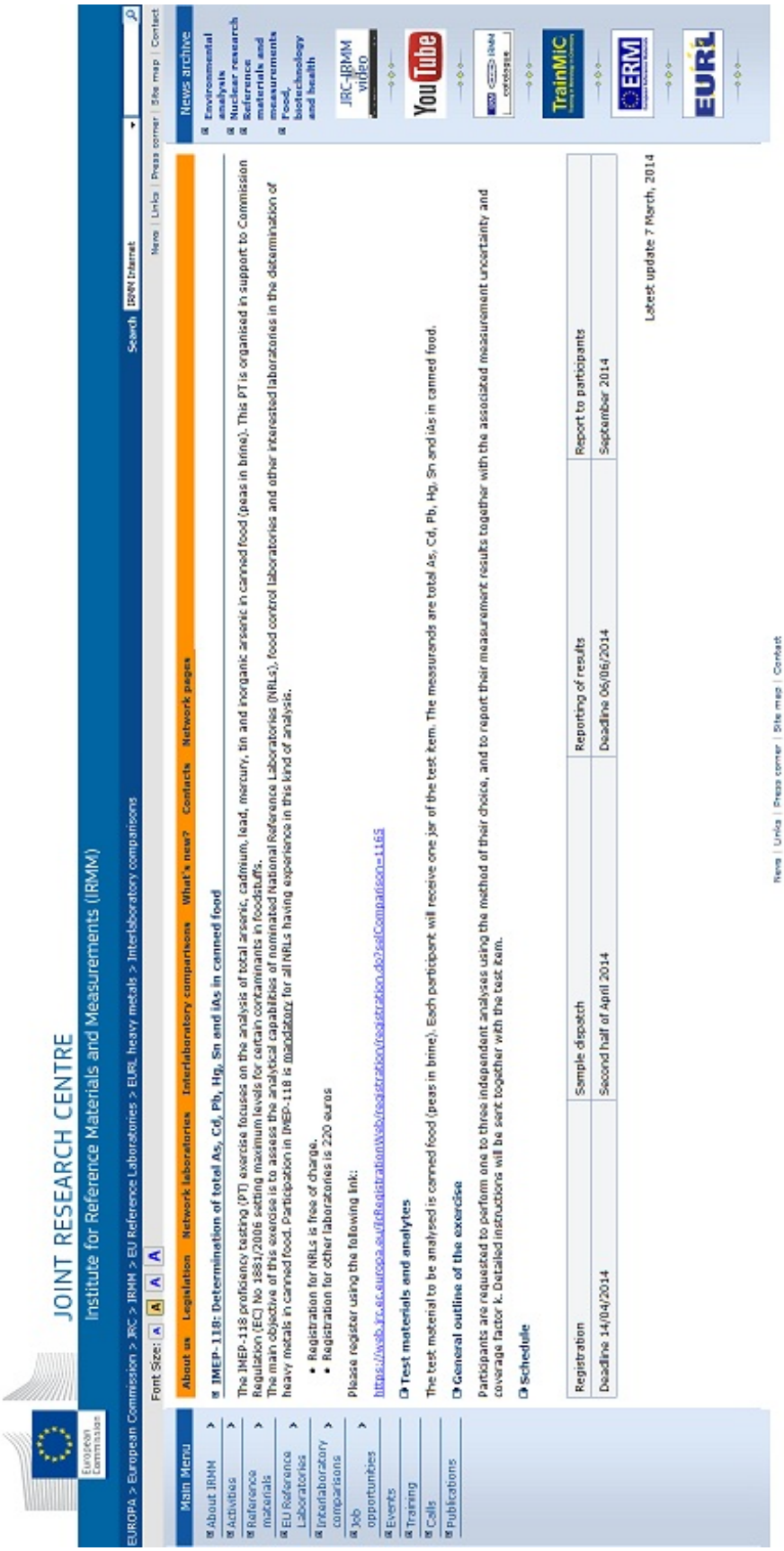
Dr. Piotr Robouch
Operating Manager EURL-HM

Cc: Franz Ulberth (Head of Unit SFB)

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

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

Annex 2: IRMM – IMEP web announcement



Annex 3: Invitation letter to EA

Ref. Ares(2014)644990 - 10/03/2014

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

Mrs Hanna Tugi
PCA – Polskie Centrum Akredytacji
ul. Szczotkarska 42
01-382 Warszawa
POLAND

IMEP-118: Interlaboratory comparison exercise for the determination of total As, Cd, Pb, Hg, Sn and iAs in canned food

Dear Mrs Tugi,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"IMEP-118: Determination of total As, Cd, Pb, Hg, Sn and iAs in canned food"** in support to the *"Commission Regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs"*.

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 **14 April 2014**. Distribution of the samples is foreseen for the second half of April 2014. The deadline for submission of results is **6 June 2014**.
More information about this PT following the link:
http://irmm.jrc.ec.europa.eu/EURLs/EURL_heavy_metals/interlaboratory_comparisons/Pages/IMEP-118DeterminationoftotalAs,Cd,Pb,Hg,SnandiAsincannedfood.aspx

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211
Telephone: direct line (32-14) 571 371. Fax: (32-14) 571 865

E-mail: jrc-irmm-mep@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=1165

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 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.

4. **Send** the printout to both the IMEP-118 and the EA-IMEP-118 coordinators:

IMEP-118 coordinator	**EA-IMEP-118 coordinator**
Dr. Ioannis Fiamegkos	Mrs Hanna Tugi
Fax +32 14 571865	Fax +22 355 70 18
E-mail: jrc-irmm-imep@ec.europa.eu	E-mail: h.tugi@pca.gov.pl

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

With kind regards

Ioannis Fiamegkos
IMEP-118 Coordinator

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Annex 4: Invitation letter to APLAC

Ref. Ares(2014)644894 - 10/03/2014

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

To: Ms Cynthia Chen
APLAC PT Committee

IMEP-118: Interlaboratory comparison exercise for the determination of total As, Cd, Pb, Hg, Sn and iAs in canned food

Dear Ms Chen,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"IMEP-118: Determination of total As, Cd, Pb, Hg, Sn and iAs in canned food".**

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-38 paying a registration fee of 220 €.

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

Registration of participants is open until **14 April 2014**. Distribution of the samples is foreseen for the second half of April 2014, and the deadline for submission of results is **06 June 2014**.
More information about this PT following the link:
http://irmm.jrc.ec.europa.eu/EURLs/EURL_heavy_metals/interlaboratory_comparisons/Pages/IMEP-118DeterminationoftotalAs,Cd,Pb,Hg,SnandiAsincannedfood.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) 571 273. Fax: (32-14) 571 865

E-mail: jrc-irmm-imep@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1165

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-118 and the APLAC coordinators:

IMEP-118 coordinator	**APLAC coordinator**
Ioannis Fiamegkos	Cynthia Chen
Fax +32 14 571 865	
E-mail: jrc-irmm-imep@ec.europa.eu	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. Ioannis Fiamegkos
IMEP-38 Coordinator

2

Annex 5: Invitation letter to IAAC

Ref. Ares(2014)645087 - 10/03/2014

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

To: Barbara Belzer
IAAC Lab Committee

IMEP-118: Interlaboratory comparison exercise for the determination of total As, Cd, Pb, Hg, Sn and iAs in canned food

Dear Mrs. Belzer,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"IMEP-118: Determination of total As, Cd, Pb, Hg, Sn and iAs in canned food".**

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-38 paying a registration fee of 220 €.

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

Registration of participants is open until **11 April 2014**. Distribution of the samples is foreseen for the second half of April 2014, and the deadline for submission of results is **02 June 2014**.
More information about this PT following the link:
http://irmm.jrc.ec.europa.eu/EURLs/EURL_heavy_metals/interlaboratory_comparisons/Pages/IMEP-118DeterminationoftotalAs,Cd,Pb,Hg,SnandiAsincannedfood.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) 571 273. Fax: (32-14) 571 865

E-mail: jrc-irmm-imep@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1165

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-118 and the IAAC coordinators:

IMEP-118 coordinator	**IAAC coordinator**
Ioannis Fiamegkos	Barbara Belzer
Fax +32 14 571 865	
E-mail: jrc-irmm-imep@ec.europa.eu	E-mail: barbara.belzer@nist.gov

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

With kind regards

Dr. Ioannis Fiamegkos
IMEP-118 Coordinator

2

Annex 6: Sample accompanying letter

Ref. Ares(2014)1191459 - 15/04/2014

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

Geel, 22 April 2014
JRC.D5/IF/acs/Ares(2014)

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

Participation in IMEP-118, a proficiency test exercise for the determination of total arsenic (As), cadmium (Cd), lead (Pb), mercury (Hg), tin (Sn) and inorganic arsenic (iAs) in canned food.

Dear «Title» «Surname»,

Thank you for participating in the IMEP-118 proficiency test for the determination of total As, Cd, Pb, Hg, Sn and iAs in canned food (peas in brine). This proficiency test (PT) exercise is organised in support to the EU Regulation 1881:2006 which sets maximum levels for certain contaminants in foodstuffs.

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

This parcel contains:
a) One jar containing approximately 100 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-EURL-HEAVY-METALS@ec.europa.eu). You should store the sample in a dark place at 4°C until analysis.

The measurands are total As, Cd, Pb, Hg, Sn and iAs in canned food (jars with peas).

The procedure used for the analyses should resemble as closely as possible the one that you use in routine analyses. Keep in mind that one of the objectives of this exercise is to check, how laboratories sample complex canned food for analysis do. Accordingly, you will not receive any further instructions on how to sample the test item for further analysis.

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

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

Reporting of results

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

- 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 **06/06/2014**.

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-EURL-HEAVY-METALS@ec.europa.eu

With kind regards,

Ioannis Fiamegkos (PhD)
IMEP-118 Coordinator

Cc: F. Ulberth (SFB HoU)

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

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

Annex 7: Confirmation of receipt form

Ref. Ares(2014)1191459 - 15/04/2014

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

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

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

IMEP-118

Total arsenic (As), cadmium (Cd), lead (Pb), mercury (Hg), Tin (Sn) and inorganic arsenic (iAs) in canned food

Confirmation of receipt of the samples

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

ANY REMARKS .....
.....

Date of package arrival .....

Signature .....

Please return this form to:

Ioannis Fiamegkos

IMEP-118 Coordinator
EC-JRC-IRMM
Retieseweg 111
B-2440 GEEL, Belgium

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

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

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

Annex 8: Questionnaire

Comparison for IMEP-118

Please fill in the 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 Hg**	**Total Pb**	**Total Sn**	**iAs**	*Info*
Accredited for:	☐	☐	☐	☐	☐	☐	

3. **Have you corrected your results for recovery?**

○ a) Yes
○ b) No

3.1. If Yes, How did you estimate the recovery?

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

3.2. If "Other" please specify

3.3. If No, what was the reason?

3.4. 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	Total Sn	iAs
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	Total Sn	iAs
Validation of measurement procedure						
Instrument calibration						

5. **What exactly have you sampled and analysed?**

☐ a) The solid/liquid composite
☐ b) The drained product
☐ c) Other

5.1. Describe briefly how you treated the can (jar) in order to sample for your analysis.

6. **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	☐	☐	☐	☐	☐	
Total Sn	☐	☐	☐	☐	☐	

7. **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	☐	☐	☐	☐	☐	☐	☐	
Total Sn	☐	☐	☐	☐	☐	☐	☐	

8. **If "Other" please specify.**

9. **Describe briefly the analytical method used for the determination of iAs**

10. **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	☐	☐	☐	☐	☐	
Total Sn	☐	☐	☐	☐	☐	
iAs	☐	☐	☐	☐	☐	

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

- ○ a) Yes
- ○ b) No

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

- ○ a) Yes
- ○ b) No

13.1. If "Yes", which:

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

13.1.1. If "Other" please specify.

14. **Considering the reported level for the investigated trace elements in the specific food matrix (canned peas) and the maximum levels of certain contaminants in foodstuffs (Commission Regulation No 1881/2006) would you accept the present sample?**

- ☐ a) Yes
- ☐ b) No

14.1. If "No" please explain why

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

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

Annex 9: Homogeneity and stability studies

9.1 Homogeneity studies (drained product)

	As		Cd		Pb		Sn	
Bottle ID	*R1*	*R2*	*R1*	*R2*	*R1*	*R2*	*R1*	*R2*
142	0.146	0.135	0.192	0.190	0.132	0.125	270	269
99	0.125	0.136	0.188	0.198	0.127	0.129	266	258
10	0.104	0.125	0.176	0.179	0.108	0.116	247	248
72	0.133	0.134	0.194	0.185	0.120	0.123	265	263
15	0.132	0.117	0.187	0.192	0.124	0.122	274	255
180	0.113	0.115	0.192	0.184	0.114	0.119	255	252
56	0.125	0.125	0.187	0.193	0.127	0.134	261	267
32	0.125	0.129	0.183	0.199	0.123	0.131	246	268
123	0.115	0.123	0.190	0.186	0.128	0.134	265	272
190	0.125	0.129	0.185	0.194	0.134	0.131	262	264
Mean	0.13		0.189		0.13		261.4	
σ	0.03		0.038		0.03		33.1	
0.3* σ	0.01		0.012		0.01		9.9	
Critical value	0.0001		0.0001		0.000		166.7	
s_x	0.008		0.004		0.01		6.8	
s_w	0.007		0.006		0.00		7.1	
s_s	0.007		0.001		0.01		4.6	
$s_s \leq 0.3 * \sigma$	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 Homogeneity studies (solid / liquid composite)

	As		Cd		Pb		Sn	
Bottle ID	*R1*	*R2*	*R1*	*R2*	*R1*	*R2*	*R1*	*R2*
24	0.10	0.11	0.137	0.128	0.087	0.086	185	185
6	0.10	0.10	0.128	0.127	0.081	0.084	180	182
134	0.11	0.10	0.128	0.113	0.084	0.079	183	179
68	0.10	0.10	0.129	0.129	0.086	0.082	179	191
172	0.10	0.10	0.134	0.129	0.083	0.087	183	190
60	0.11	0.11	0.130	0.135	0.088	0.089	188	186
84	0.11	0.11	0.128	0.132	0.082	0.092	174	194
109	0.11	0.11	0.133	0.129	0.085	0.079	182	179
192	0.09	0.10	0.125	0.128	0.077	0.084	177	188
158	0.11	0.11	0.125	0.130	0.083	0.089	184	183
Mean	0.10		0.129		0.08		183.6	
σ	0.027		0.028		0.02		24.1	
0.3* σ	0.01		0.008		0.01		7.2	
Critical value	0.00004		0.00005		0.00003		94.8	
s_x	0.004		0.004		0.003		2.3	
s_w	0.005		0.005		0.004		6.1	
s_s	0.002		0.002		0.000		0.0	
$s_s \leq 0.3 * \sigma$	Pass		Pass		Pass		Pass	

Where s_p is the standard deviation for the PT assessment,

s_x is the standard deviation of the sample averages,

s_w is the within-sample standard deviation,

s_s is the between-sample standard deviation,

9.3 Stability studies (solid / liquid composite)

	Time in Weeks				u_{st}
	0	3	5	8	
As	0.101	0.111	0.118	0.105	**3.6%**
	0.104	0.106	0.098	0.11	
Cd	0.134	0.128	0.131	0.128	**1.7%**
	0.131	0.132	0.135	0.124	
Pb	0.0872	0.0881	0.0862	0.0844	**2.3%**
	0.0868	0.0953	0.0865	0.0852	
Sn	184	181	188	179	**1.7%**
	187	193	179	179	

Annex 10: Results for total As

Drained product

Assigned range: X_{ref} = 0.117; U_{ref} (*k=2*) = 0.018; σ = 0.026 (all values in mg kg^{-1})

Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L027	0.148	0.034	2	ICP-MS	0.017	1.19	1.59	a
L028	0.122	0.04	2	ICP-MS	0.02	0.19	0.22	a
L034	<0.3			ICP-AES				
L036	0.086	0.0047	2	H-AAS	0.002	-1.21	-3.28	b
L037	0.119	0.002	√3	ICP-MS	0.001	0.07	0.19	b
L038	0.316			ICP-AES	0	7.71	21.57	b
L045	0.112			ICP-MS	0	-0.20	-0.57	b
L046	0.125	0.009	2	ICP-MS	0.004	0.30	0.76	b
L049	0.1101	0.043	2	H-AAS	0.0215	-0.28	-0.30	a
L052	<2.5			ICP-AES				
L055	0.084	0.016	2	ICP-MS	0.008	-1.29	-2.72	b
L059	0.12	0.008	2	ICP-MS	0.004	0.11	0.28	b
L064	<0.5			FAAS-MHS				
L066	0.11	0.008	2	ICP-MS	0.004	-0.28	-0.72	b
L067	0.102	0.036	2	ICP-MS	0.018	-0.59	-0.75	a
L069	0.11	0.045	2	ICP-MS	0.0225	-0.28	-0.30	a
L070	0.113	0.023	2	ICP-MS	0.0115	-0.16	-0.29	a
L074	<0.1			AAS				
L077	0.143			ICP-MS	0	1.00	2.80	b
L078	0.096			ICP-MS	0	-0.82	-2.30	b
L079	0.097	0.084	2	ICP-MS	0.042	-0.78	-0.47	c
L080	0.099	0.025	3	ICP-MS	0.008	-0.71	-1.47	b
L083	0.0336	25	√3	ICP-MS	14.434	-3.24	-0.01	c
L084	0.11	0.019	2	ETAAS	0.009	-0.28	-0.55	a
L086	0.102	40	√3	ICPMS	23.094	-0.59	0.00	c
L089	0.126	0.029	2	HG-AAS	0.0145	0.34	0.51	a
L093	0.11	0.02	2	ICP-MS	0.01	-0.28	-0.53	a
L096	0.101	0.001	√3	ICP-MS	0.0006	-0.63	-1.76	b
L099	0.122	0.013	1	HG-AAS	0.013	0.19	0.30	a
L101	<0.5			ICP-AES				
L102	0.119	0.017	2	ICP-MS	0.008	0.07	0.14	b
L105	0.098	0.045	2	HG-AAS	0.022	-0.75	-0.79	a
L107	0.099	28	2	ICP-MS	14	-0.71	0.00	c
L112	0.12	0.024	2	ICP-MS	0.012	0.11	0.18	a
L114	0.084	0.017	2	ICP-AES	0.008	-1.29	-2.65	b
L118	<0.01							
L120	0.09		2	ICP-MS	0	-1.06	-2.95	b
L121	0.108	0.007	2	ICP-MS	0.003	-0.36	-0.93	b
L123	0.066	0.004	2	HG-AAS	0.002	-1.99	-5.43	b
N001	0.12	0.01	2	ICP-MS	0.005	0.11	0.27	b
N002	0.11	0.04	2	ETAAS	0.02	-0.28	-0.33	a
N003	0.104	0.025	2	ICP-MS	0.013	-0.51	-0.85	a
N004	0.175	0.02	2	ICP-MS	0.011	2.24	4.14	a
N005	<0.2			ICP-MS				
N006	<0.85			ETAAS				
N007	0.126	0.025	2	ICP-MS	0.012	0.34	0.57	a
N008	0.0261	0.0012	2		0.0006	-3.53	-9.86	b
N011	0.1	0.03	2	ICP-MS	0.015	-0.67	-0.98	a
N012	0.099	0.027	2	ICP-MS	0.0135	-0.71	-1.11	a
N013	0.098	0.01	2	ICP-MS	0.003	-0.75	-1.98	b
N014	0.0912	0.0182	2	ICP-MS	0.009	-1.01	-2.01	b
N015	0.111	0.011	2	HG-AAS	0.005	-0.24	-0.58	b
N019	0.1	0.02	2	ICP-MS	0.01	-0.67	-1.27	a
N020	0.11	0.04	2	ICP-MS	0.02	-0.28	-0.33	a
N024	0.07	0.02	2	AAS	0.01	-1.83	-3.47	a
N025	0.085	0.02	2	HG-AAS	0.01	-1.25	-2.37	a
N030	0.1	0.01	2	AAS	0.005	-0.67	-1.64	b
N044	0.12	0.036	2	ICP-MS	0.018	0.11	0.14	a
N106	0.11	0.024	2	ETAAS	0.012	-0.28	-0.48	a

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

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

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

Solid / Liquid composite

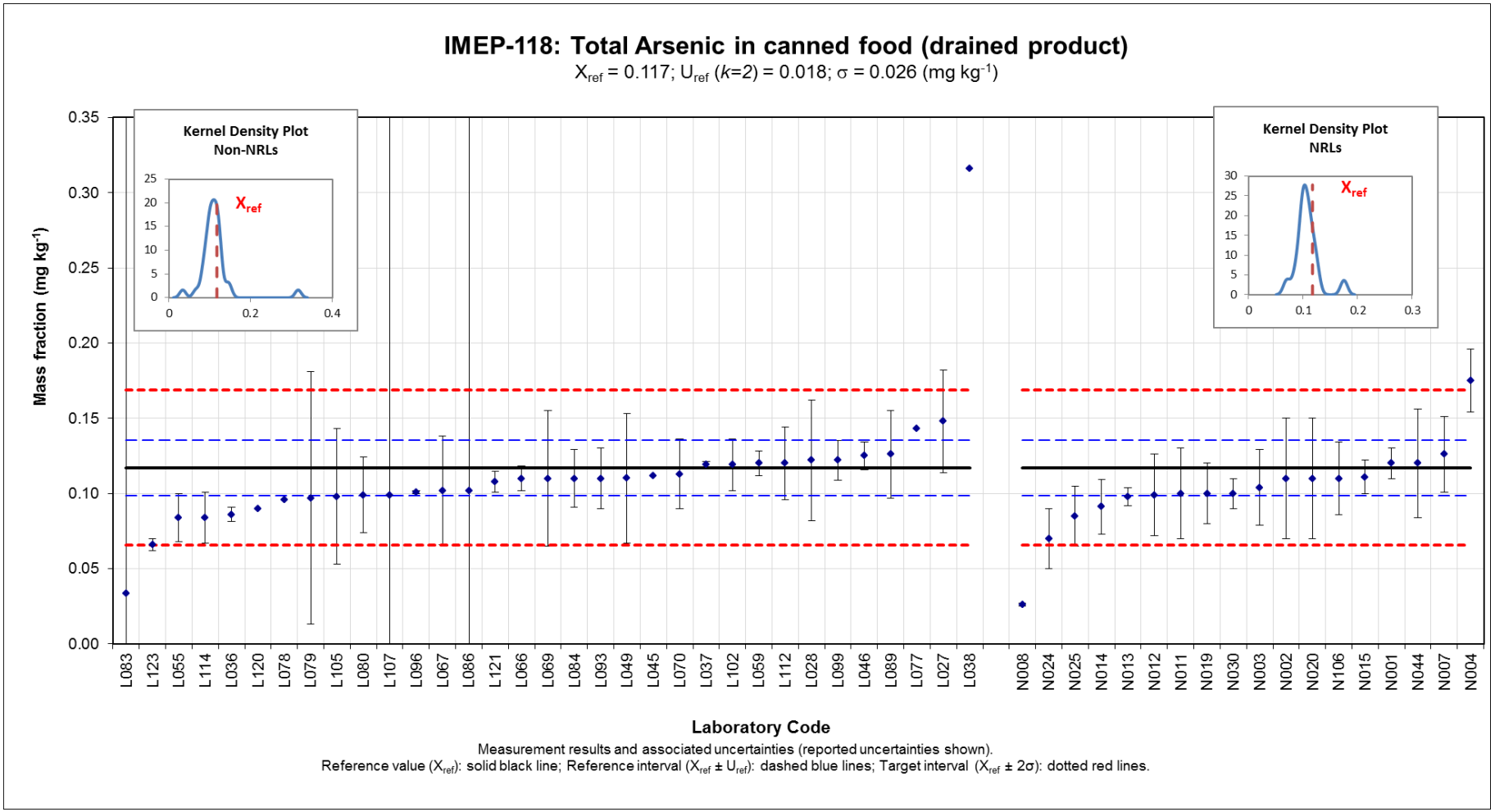
Assigned range: X_{ref} = 0.121; U_{ref} (*k*=2) = 0.014; σ = 0.027 (all values in mg kg^{-1})

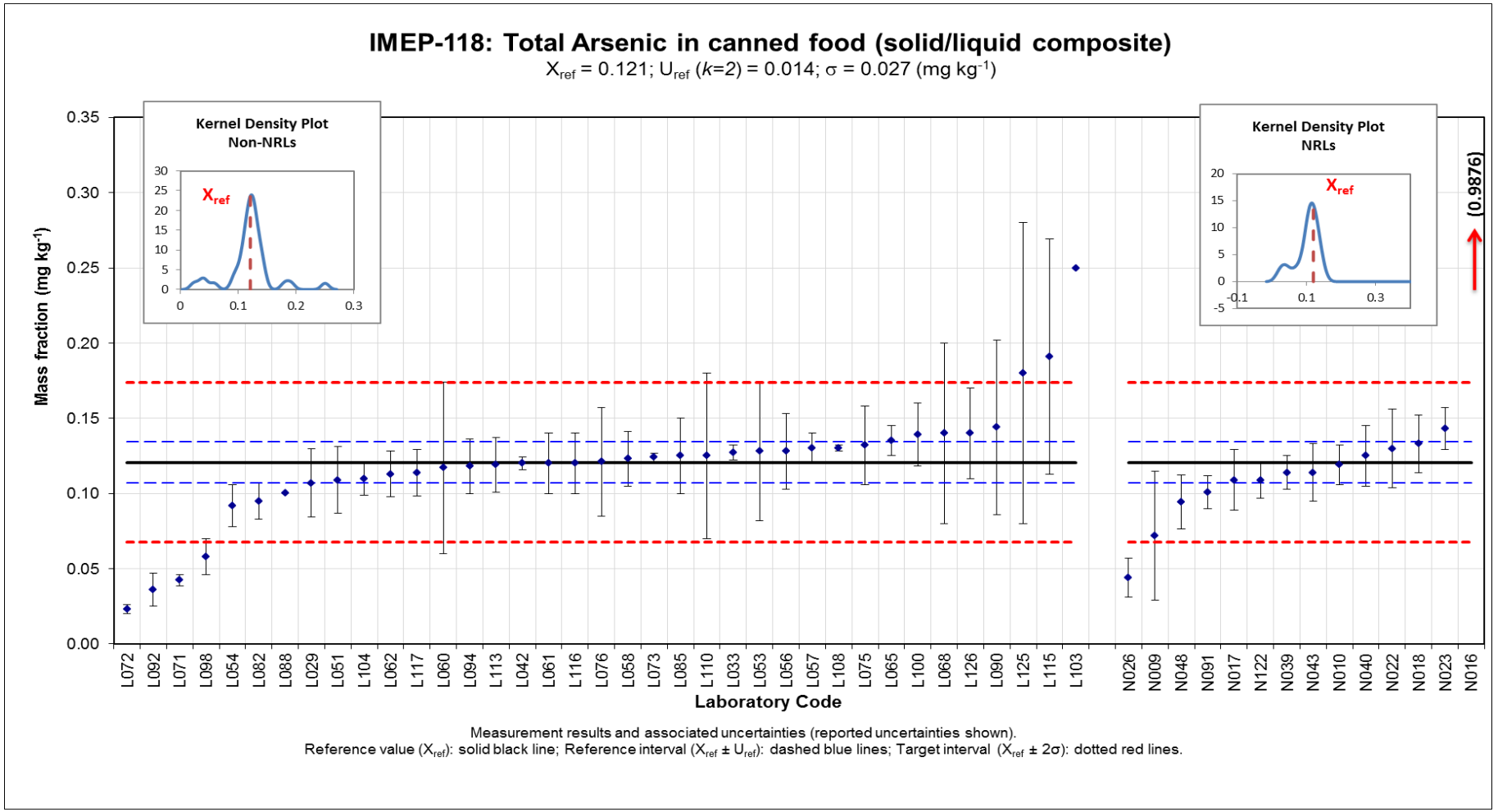
Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L029	0.107	0.0227	2	SFICP-MS	0.011	-0.51	-1.02	a
L031	<0.1			ICP-MS				
L032	<0.3			ICP-MS				
L033	0.127	0.01	2	ICP-MS	0.003	0.24	0.89	b
L042	0.12	0.004	2	ICPMS	0.002	-0.02	-0.07	b
L051	0.109	0.022	2	ICP-MS	0.011	-0.43	-0.89	a
L053	0.128	0.046	2	HG-AAS	0.023	0.28	0.31	a
L054	0.092	0.014	2	FIAS-AAS	0.007	-1.08	-2.91	a
L056	0.128	0.03	2	ICP-MS	0.013	0.28	0.52	a
L057	0.13	0.01	√3	ICP-MS	0.006	0.36	1.06	b
L058	0.123	0.018	2	ICP-MS	0.009	0.09	0.22	a
L060	0.117	0.057	2	G-AAS	0.029	-0.13	-0.12	c
L061	0.12	0.02	2	ICP-MS	0.010	-0.02	-0.04	a
L062	0.113	0.015	2	ICP-MS	0.008	-0.28	-0.74	a
L065	0.135	0.01	√3	AAS	0.006	0.55	1.61	b
L068	0.14	0.060	2	HGA-AA	0.030	0.73	0.63	c
L071	0.0424	0.0037	2	ETAAS	0.002	-2.95	-10.99	b
L072	0.023	0.003	2	AFS	0.002	-3.68	-13.89	b
L073	0.124	0.0026	95	HG-AAS	0.00003	0.13	0.51	b
L075	0.132	0.026	2	ICP-MS	0.013	0.43	0.78	a
L076	0.121	0.036	2	HG-AAS	0.018	0.02	0.02	a
L082	0.095	0.012	√3	AAS	0.007	-0.96	-2.62	a
L085	0.125	0.025	2	SFICP-MS	0.013	0.17	0.31	a
L088	0.1005			ICP-AES	0	-0.76	-2.92	b
L090	0.144	0.058	2	ICP-MS	0.029	0.89	0.79	c
L092	0.036	0.011	2	HG-AAS	0.006	-3.19	-9.61	b
L094	0.118	0.018	2	ICP-MS	0.009	-0.10	-0.22	a
L098	0.058	0.012	2	ICP-OES	0.006	-2.36	-6.86	b
L100	0.139	0.021	1	ICP-AES	0.021	0.70	0.84	a
L103	0.25			CV-AAS	0	4.88	18.87	b
L104	0.11	0.011	1	ICP-MS	0.011	-0.40	-0.81	a
L108	0.13	0.002	3		0.001	0.36	1.37	b
L110	0.125	0.055	2	ICP-MS	0.028	0.17	0.16	c
L113	0.119	0.018	2	ICP-MS	0.009	-0.06	-0.13	a
L115	0.191	0.078	2	ETAAS	0.039	2.66	1.78	c
L116	0.12	0.02	2	HG-AAS	0.010	-0.02	-0.04	a
L117	0.1138	0.0154	2	HG-AAS	0.008	-0.25	-0.65	a
L125	0.18	0.1	2	HG-AAS	0.050	2.24	1.18	c
L126	0.14	0.03	2	ICP-MS	0.015	0.73	1.18	a
N009	0.072	0.043	2	ETAAS	0.022	-1.83	-2.15	a
N010	0.119	0.013	2	ICP-MS	0.007	-0.06	-0.16	b
N016	0.9876	0.2	√3	ICP-MS	0.115	32.70	7.50	c
N017	0.109	0.02	2	HG-AAS	0.010	-0.43	-0.95	a
N018	0.133	0.019	2	ICP-MS	0.010	0.47	1.06	a
N022	0.1299	0.026	2	ICP-MS	0.013	0.35	0.64	a
N023	0.143	0.014	2	HG-AAS	0.007	0.85	2.29	a
N026	0.044	0.013	2	HG-AAS	0.007	-2.89	-8.10	b
N039	0.114	0.011	2	ICP-MS	0.006	-0.25	-0.74	b
N040	0.125	0.02	2	ICP-MS	0.010	0.17	0.37	a
N043	0.114	0.019	2	ICP-MS	0.010	-0.25	-0.56	a
N048	0.0944	0.0179	√3	ICP-MS	0.010	-0.99	-2.11	a
N091	0.101	0.011	2	HG-AAS	0.006	-0.74	-2.22	b
N122	0.109	0.012	2	ICP-MS	0.006	-0.43	-1.26	b

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

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

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


IMEP-118: Total Arsenic in canned food (drained product)
Xref = 0.117; Uref (k=2) = 0.018; σ = 0.026 (mg kg-1)
Kernel Density Plot
Non-NRLs
Xref
Kernel Density Plot
NRLs
Xref
Mass fraction (mg kg-1)
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
L083 L123 L055 L114 L036 L120 L078 L079 L105 L080 L107 L096 L067 L086 L121 L066 L069 L084 L093 L049 L045 L070 L037 L102 L059 L112 L028 L099 L046 L089 L077 L027 L038
N008 N024 N025 N014 N013 N012 N011 N019 N030 N003 N002 N020 N106 N015 N001 N044 N007 N004
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (Xref): solid black line; Reference interval (Xref ± Uref): dashed blue lines; Target interval (Xref ± 2σ): dotted red lines.


IMEP-118: Total Arsenic in canned food (solid/liquid composite)
X_{ref} = 0.121; U_{ref} (*k*=2) = 0.014; σ = 0.027 (mg kg^{-1})
Kernel Density Plot
Non-NRLs
X_{ref}
Kernel Density Plot
NRLs
X_{ref}
(0.9876)
Mass fraction (mg kg^{-1})
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
L072 L092 L071 L098 L054 L082 L088 L029 L051 L104 L062 L117 L060 L094 L113 L042 L061 L116 L076 L058 L073 L085 L110 L033 L053 L056 L057 L108 L075 L065 L100 L068 L126 L090 L125 L115 L103
N026 N009 N048 N091 N017 N122 N039 N043 N010 N040 N022 N018 N023 N016
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{ref}): solid black line; Reference interval (X_{ref} ± U_{ref}): dashed blue lines; Target interval (X_{ref} ± 2σ): dotted red lines.

Annex 11: Results for total Cd

Drained product

Assigned range: X_{ref} = 0.192; U_{ref} (*k=2*) = 0.023 ; σ = 0.039 (all values in mg kg^{-1})

Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L027	0.203	0.051	2	ICP-MS	0.026	0.29	0.40	a
L028	0.195	0.048	2	ICP-MS	0.024	0.08	0.12	a
L034	0.19	0.03	2	ICP-AES	0.015	-0.05	-0.10	a
L036	0.2059	0.0056	2	ETAAS	0.003	0.37	1.18	b
L037	0.192	0.007	√3	ICP-MS	0.004	0.00	0.01	b
L038	0.41			ICP-AES	0	5.69	18.85	b
L045	0.177			ICP-MS	0	-0.39	-1.28	b
L046	0.183	0.011	2	ICP-MS	0.006	-0.23	-0.69	b
L049	0.196	0.059	2	ICP-AES	0.030	0.11	0.13	a
L050	0.17	0.04	2	ICP-MS	0.020	-0.57	-0.95	a
L052	0.200	0.02	2	ICP-AES	0.010	0.21	0.53	b
L055	0.193	0.028	2	ICP-MS	0.014	0.03	0.06	a
L059	0.189	0.008	2	ICP-MS	0.004	-0.07	-0.23	b
L064	0.17	0.03	2	ICP-AES	0.015	-0.57	-1.15	a
L066	0.185	0.01	2	ICP-MS	0.005	-0.18	-0.54	b
L067	0.192	0.038	2	ICP-MS	0.019	0.00	0.01	a
L069	0.18	0.014	2	ICP-MS	0.007	-0.31	-0.88	b
L070	0.191	0.034	2	ICP-MS	0.017	-0.02	-0.04	a
L074	0.16	0.04	√3	AAS	0.023	-0.83	-1.23	a
L077	0.159			ICP-MS	0	-0.86	-2.84	b
L078	0.192			ICP-MS	0	0.00	0.01	b
L079	0.195	0.035	2	ICP-MS	0.018	0.08	0.15	a
L080	0.19	0.008	3	ICP-MS	0.003	-0.05	-0.16	b
L081	0.192	0.023	2	ICP-AES	0.012	0.00	0.01	b
L083	0.0597	25	√3	ICP-MS	14.434	-3.44	-0.01	c
L084	0.18	0.058	2	ETAAS	0.029	-0.31	-0.38	a
L086	0.191	40	√3	ICPMS	23.094	-0.02	0.00	c
L087	0.182	0.118	2	ICP-AES	0.059	-0.26	-0.16	c
L089	0.135	0.017	2	ETAAS	0.009	-1.48	-3.96	b
L093	0.18	0.05	2	ICP-MS	0.025	-0.31	-0.43	a
L095	0.181	10	√3	AAS	5.774	-0.28	0.00	c
L096	0.168	0.025	2	AAS	0.013	-0.62	-1.40	a
L099	0.112	0.012	1	ETAAS	0.012	-2.08	-4.79	a
L101	0.19	0.04	1	ICP-AES	0.040	-0.05	-0.04	c
L102	0.2	0.026	2	ICP-MS	0.013	0.21	0.47	a
L107	0.178	50	2	ICP-MS	25.000	-0.36	0.00	c
L109	0.26	0.03	2	ETAAS	0.015	1.78	3.60	a
L112	0.2	0.02	2	ICP-MS	0.010	0.21	0.53	b
L114	0.1	0.02	2	ICP-AES	0.010	-2.39	-6.01	b
L118	0.231	0.033	2	ETAAS	0.017	1.02	1.94	a
L120	0.18	0.03	2	ICP-MS	0.015	-0.31	-0.63	a
L121	0.19	0.002	2	ICP-MS	0.001	-0.05	-0.16	b
L123	0.119	0.018	2	FAAS	0.009	-1.90	-4.97	b
L124	0.21	0.19	2	ICP-MS	0.095	0.47	0.19	c
L127	0.21	0.03	2	ETAAS	0.015	0.47	0.96	a
N001	0.22	0.03	2	ICP-MS	0.015	0.73	1.49	a
N002	0.19	0.03	2	ETAAS	0.015	-0.05	-0.10	a
N003	0.188	0.025	2	ICP-MS	0.013	-0.10	-0.23	a
N004	0.215	0.023	2	ICP-MS	0.012	0.60	1.42	b
N005	0.21	0.03	2	ICP-MS	0.015	0.47	0.96	a
N006	0.22	0.02	2	ICP-MS	0.010	0.73	1.84	b
N007	0.191	0.05	2	ICP-MS	0.025	-0.02	-0.03	a
N008	0.189	0.012	2		0.006	-0.07	-0.22	b
N011	0.18	0.05	2	ICP-MS	0.025	-0.31	-0.43	a
N012	0.17	0.039	2	ICP-MS	0.020	-0.57	-0.96	a
N013	0.19	0.013	2	ICP-MS	0.007	-0.05	-0.14	b
N014	0.2	0.04	2	ICP-MS	0.020	0.21	0.35	a
N015	0.206	0.031	2	ETAAS	0.016	0.37	0.73	a
N019	0.19	0.03	2	ICP-MS	0.015	-0.05	-0.10	a
N020	0.140	0.06	2	ICP-MS	0.030	-1.35	-1.61	a
N024	0.159	0.037	2	AAS	0.019	-0.86	-1.51	a
N025	0.189	0.026	2	ETAAS	0.013	-0.07	-0.16	a
N030	0.23	0.03	2	AAS	0.015	0.99	2.01	a
N041	0.19	0.033	2	ETAAS	0.017	-0.05	-0.09	a
N044	0.22	0.089	2	ICP-MS	0.045	0.73	0.61	c
N106	0.175	0.017	2	ETAAS	0.009	-0.44	-1.17	b

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

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

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

Solid / Liquid composite

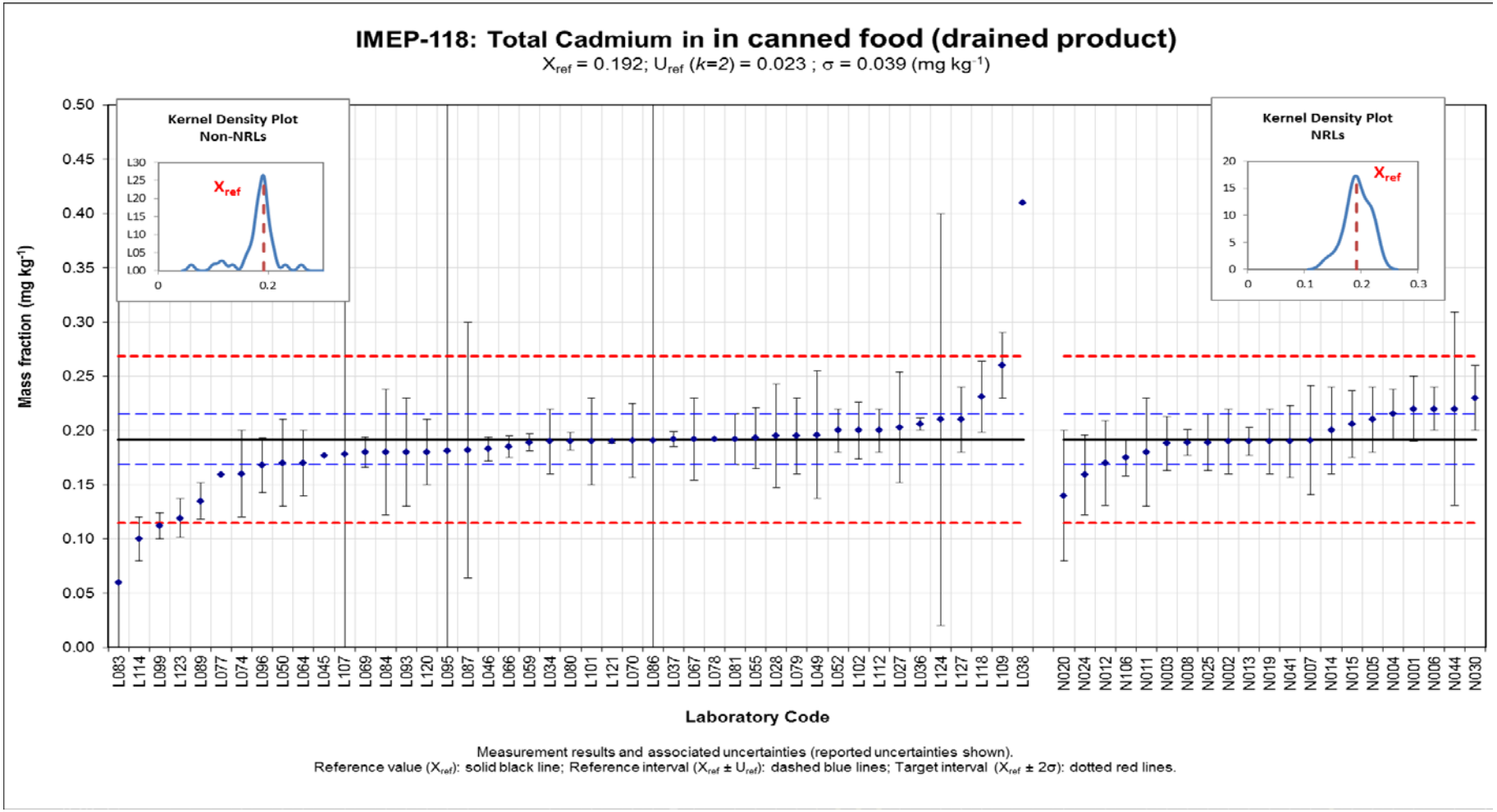
Assigned range: X_{ref} = 0.130 ; U_{ref} (k=2) = 0.016 ; σ = 0.028 (all values in mg kg^{-1})

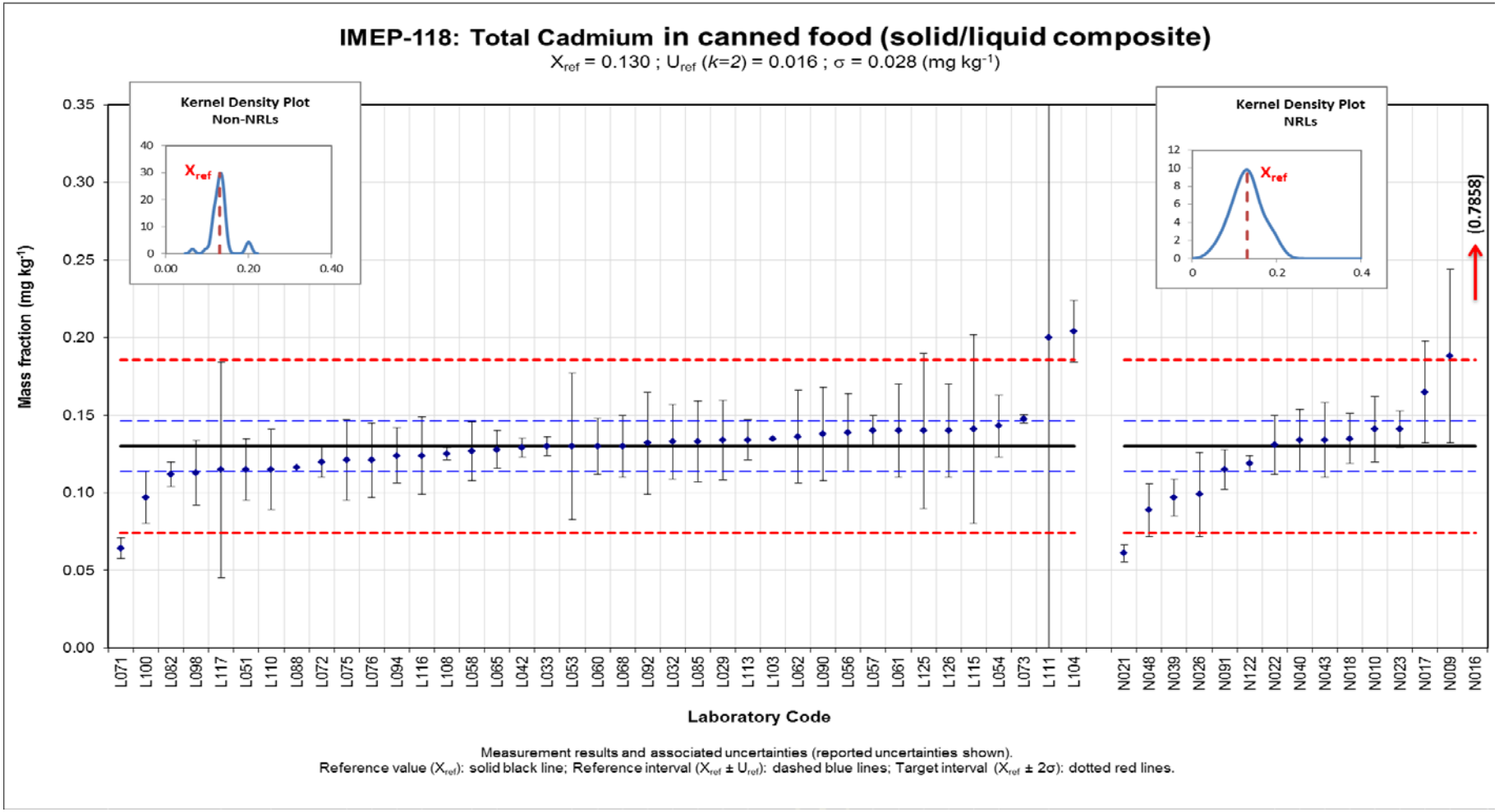
Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L029	0.134	0.0257	2	SFICP-MS	0.013	0.14	0.26	a
L031	<0.5			ICP-MS				
L032	0.133	0.024	2	ICP-MS	0.012	0.11	0.20	a
L033	0.13	0.006	2	ICP-MS	0.003	0.00	-0.01	b
L042	0.13	0.0061	2	ICPMS	0.003	-0.04	-0.12	b
L047	<0.01			ETAAS				
L051	0.115	0.02	2	ICP-MS	0.010	-0.54	-1.17	a
L053	0.13	0.047	2	ICP-AES	0.024	0.00	0.00	a
L054	0.143	0.02	2	ETAAS	0.010	0.46	1.00	a
L056	0.139	0.025	2	ICP-MS	0.013	0.32	0.60	a
L057	0.14	0.01	√3	ICP-MS	0.006	0.36	1.00	b
L058	0.127	0.019	2	ICP-MS	0.010	-0.11	-0.24	a
L060	0.130	0.018	2	G-AAS	0.009	0.00	0.00	a
L061	0.14	0.03	2	AAS	0.015	0.36	0.58	a
L062	0.136	0.03	2	ICP-MS	0.015	0.21	0.35	a
L065	0.128	0.012	√3	AAS	0.007	-0.07	-0.19	b
L068	0.13	0.02	2	HGA-AA	0.010	0.00	0.00	a
L071	0.0643	0.0067	2	GF-AAS	0.003	-2.35	-7.46	b
L072	0.12	0.01	2	ICP-AES	0.005	-0.36	-1.05	b
L073	0.1478	0.0026	90	ETAAS	0.000	0.63	2.18	b
L075	0.121	0.026	2	ICP-MS	0.013	-0.32	-0.59	a
L076	0.121	0.024	2	AAS	0.012	-0.32	-0.62	a
L082	0.112	0.008	√3	AAS	0.005	-0.65	-1.93	b
L085	0.133	0.026	2	SFICP-MS	0.013	0.11	0.19	a
L088	0.1165			ICP-AES	0	-0.48	-1.66	b
L090	0.138	0.03	2	ICP-MS	0.015	0.28	0.47	a
L092	0.132	0.033	2	ETAAS	0.017	0.07	0.11	a
L094	0.124	0.018	2	ICP-MS	0.009	-0.22	-0.50	a
L098	0.113	0.021	2	ICP-OES	0.011	-0.61	-1.28	a
L100	0.097	0.017	1	ICP-AES	0.017	-1.18	-1.75	a
L103	0.135			AAS	0	0.18	0.61	b
L104	0.204	0.02	1	ICP-MS	0.020	2.64	3.42	a
L108	0.125	0.004	3		0.001	-0.18	-0.61	b
L110	0.115	0.026	2	ICP-MS	0.013	-0.54	-0.98	a
L111	0.2	26	2	AAS	13.000	2.50	0.01	c
L113	0.134	0.013	2	ICP-MS	0.007	0.14	0.38	b
L115	0.141	0.061	2	ETAAS	0.031	0.39	0.35	c
L116	0.124	0.025	2	ETAAS	0.013	-0.22	-0.41	a
L117	0.1148	0.0694	2	ETAAS	0.035	-0.55	-0.43	c
L125	0.14	0.05	2	FAAS	0.025	0.36	0.38	a
L126	0.14	0.03	2	ICP-MS	0.015	0.36	0.58	a
N009	0.188	0.056	2	ETAAS	0.028	2.07	1.99	c
N010	0.141	0.021	2	ICP-MS	0.011	0.39	0.82	a
N016	0.7858	0.16	√3	ICP-MS	0.092	23.45	7.07	c
N017	0.165	0.033	2	ETAAS	0.017	1.25	1.90	a
N018	0.135	0.016	2	ICP-MS	0.008	0.18	0.43	b
N021	0.061	0.0057	2	AAS	0.003	-2.47	-8.00	b
N022	0.1307	0.019	2	ICP-MS	0.010	0.02	0.05	a
N023	0.141	0.012	2	ETAAS	0.006	0.39	1.08	b
N026	0.099	0.027	2	GF-AAS	0.014	-1.11	-1.97	a
N039	0.097	0.012	2	ICP-MS	0.006	-1.18	-3.27	b
N040	0.134	0.02	2	ICP-MS	0.010	0.14	0.31	a
N043	0.134	0.024	2	ICP-MS	0.012	0.14	0.27	a
N048	0.0889	0.0169	√3	ICP-MS	0.010	-1.47	-3.24	a
N091	0.115	0.013	2	FAAS	0.007	-0.54	-1.44	b
N122	0.119	0.005	2	ICP-MS	0.003	-0.40	-1.30	b

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

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

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


IMEP-118: Total Cadmium in in canned food (drained product)
Xref = 0.192; Uref (k=2) = 0.023 ; σ = 0.039 (mg kg-1)
Kernel Density Plot
Non-NRLs
Xref
Kernel Density Plot
NRLs
Xref
Mass fraction (mg kg-1)
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
L083 L114 L099 L123 L089 L077 L074 L096 L050 L064 L045 L107 L069 L084 L093 L120 L095 L087 L046 L066 L059 L034 L080 L101 L121 L070 L086 L037 L067 L078 L081 L055 L028 L079 L049 L052 L102 L112 L027 L036 L124 L127 L118 L109 L038
N020 N024 N012 N106 N011 N003 N008 N025 N002 N013 N019 N041 N007 N014 N015 N005 N004 N001 N006 N044 N030
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (Xref): solid black line; Reference interval (Xref ± Uref): dashed blue lines; Target interval (Xref ± 2σ): dotted red lines.


IMEP-118: Total Cadmium in canned food (solid/liquid composite)
X_{ref} = 0.130 ; U_{ref} (k=2) = 0.016 ; σ = 0.028 (mg kg^{-1})
Kernel Density Plot
Non-NRLs
Kernel Density Plot
NRLs
X_{ref}
(0.7858)
Mass fraction (mg kg^{-1})
Laboratory Code
L071 L100 L082 L098 L117 L051 L110 L088 L072 L075 L076 L094 L116 L108 L058 L065 L042 L033 L053 L060 L068 L092 L032 L085 L029 L113 L103 L062 L090 L056 L057 L061 L125 L126 L115 L054 L073 L111 L104 N021 N048 N039 N026 N091 N122 N022 N040 N043 N018 N010 N023 N017 N009 N016
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{ref}): solid black line; Reference interval (X_{ref} ± U_{ref}): dashed blue lines; Target interval (X_{ref} ± 2σ): dotted red lines.

Annex 12: Results for total Pb

Drained product

Assigned range: X_{ref} = 0.116; U_{ref} (*k=2*) = 0.019; σ = 0.025 (all values in mg kg^{-1})

Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L027	0.14	0.039	2	ICP-MS	0.020	0.96	1.13	a
L028	0.118	0.033	2	ICP-MS	0.017	0.10	0.13	a
L034	0.12	0.02	2	ICP-AES	0.010	0.18	0.32	a
L036	0.1374	0.01	2	ETAAS	0.005	0.86	2.04	b
L037	0.126	0.003	√3	ICP-MS	0.002	0.41	1.09	b
L038	0.361			ICP-AES	0	9.66	25.92	b
L045	0.133			ICP-MS	0	0.69	1.84	b
L046	0.125	0.009	2	ICP-MS	0.005	0.37	0.90	b
L049	0.1335	0.036	2	ICP-AES	0.018	0.71	0.88	a
L050	0.12	0.03	2	ICP-MS	0.015	0.18	0.25	a
L052	<0.5			ICP-AES				
L055	0.126	0.033	2	ICP-MS	0.017	0.41	0.55	a
L059	0.138	0.013	2	ICP-MS	0.007	0.88	1.95	b
L064	0.11	0.02	2	ICP-AES	0.010	-0.22	-0.40	a
L066	0.132	0.006	2	ICP-MS	0.003	0.65	1.66	b
L067	0.139	0.028	2	ICP-MS	0.014	0.92	1.39	a
L069	0.13	0.037	2	ICP-MS	0.019	0.57	0.70	a
L070	0.134	0.024	2	ICP-MS	0.012	0.73	1.21	a
L074	0.08	0.08	√3	AAS	0.046	-1.40	-0.75	c
L077	0.131			ICP-MS	0	0.61	1.63	b
L078	0.134			ICP-MS	0	0.73	1.95	b
L079	0.125	0.032	2	ICP-MS	0.016	0.37	0.51	a
L080	0.136	0.042	3	ICP-MS	0.014	0.80	1.21	a
L081	0.133	0.024	2	ICP-AES	0.012	0.69	1.14	a
L083	0.0371	25	√3	ICP-MS	14.434	-3.09	-0.01	c
L084	0.13	0.045	2	ETAAS	0.023	0.57	0.59	a
L086	0.104	40	√3	ICPMS	23.094	-0.45	0.00	c
L087	0.116	0.039	2	ICP-AES	0.020	0.02	0.02	a
L089	0.071	0.011	2	ETAAS	0.006	-1.75	-4.07	b
L093	0.12	0.02	2	ICP-MS	0.010	0.18	0.32	a
L095	0.135	10	√3	AAS	5.774	0.77	0.00	c
L096	0.133	0.028	2	AAS	0.014	0.69	1.03	a
L099	0.093	0.03	1	ETAAS	0.030	-0.89	-0.72	c
L101	<0.2			ICP-AES				
L102	0.128	0.017	2	ICP-MS	0.009	0.49	0.98	b
L107	0.114	32	2	ICP-MS	16.000	-0.06	0.00	c
L109	0.1	0.01	2	ETAAS	0.005	-0.61	-1.45	b
L112	0.12	0.012	2	ICP-MS	0.006	0.18	0.40	b
L114	0.14	0.028	2	ICP-AES	0.014	0.96	1.45	a
L118	0.039	0.005	2	ETAAS	0.003	-3.01	-7.82	b
L120	0.71	0.22	2	ICP-MS	0.110	23.38	5.38	c
L121	0.121	0.002	2	ICP-MS	0.001	0.21	0.57	b
L123	0.134	0.011	2	FAAS	0.006	0.73	1.68	b
L124	0.13	0.083	2	ICP-MS	0.042	0.57	0.34	c
L127	0.1	0.03	2	ETAAS	0.015	-0.61	-0.88	a
N001	0.15	0.02	2	ICP-MS	0.010	1.36	2.50	a
N002	0.095	0.03	2	ETAAS	0.015	-0.81	-1.16	a
N003	0.13	0.03	2	ICP-MS	0.015	0.41	0.59	a
N004	0.157	0.018	2	ICP-MS	0.009	1.63	3.17	b
N005	0.14	0.03	2	ICP-MS	0.015	0.96	1.38	a
N006	0.17	0.03	2	ICP-MS	0.015	2.14	3.07	a
N007	0.135	0.038	2	ICP-MS	0.019	0.77	0.92	a
N008	0.134	0.017	2		0.009	0.73	1.45	b
N011	0.11	0.03	2	ICP-MS	0.015	-0.22	-0.31	a
N012	0.11	0.028	2	ICP-MS	0.014	-0.22	-0.33	a
N013	0.12	0.011	2	ICP-MS	0.006	0.18	0.41	b
N014	0.119	0.024	2	ICP-MS	0.012	0.14	0.23	a
N015	0.161	0.032	2	ETAAS	0.016	1.79	2.44	a
N019	0.11	0.02	2	ICP-MS	0.010	-0.22	-0.40	a
N020	0.089	0.04	2	ICP-MS	0.020	-1.04	-1.20	a
N024	0.12	0.04	2	AAS	0.020	0.18	0.20	a
N025	0.112	0.036	2	ETAAS	0.018	-0.14	-0.17	a
N030	0.13	0.023	2	AAS	0.012	0.57	0.97	a
N041	0.13	0.031	2	ICP-MS	0.016	0.57	0.80	a
N044	0.14	0.071	2	ICP-MS	0.036	0.96	0.67	c
N106	0.13	0.041	2	ETAAS	0.021	0.57	0.64	a

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

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

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

Solid / liquid composite

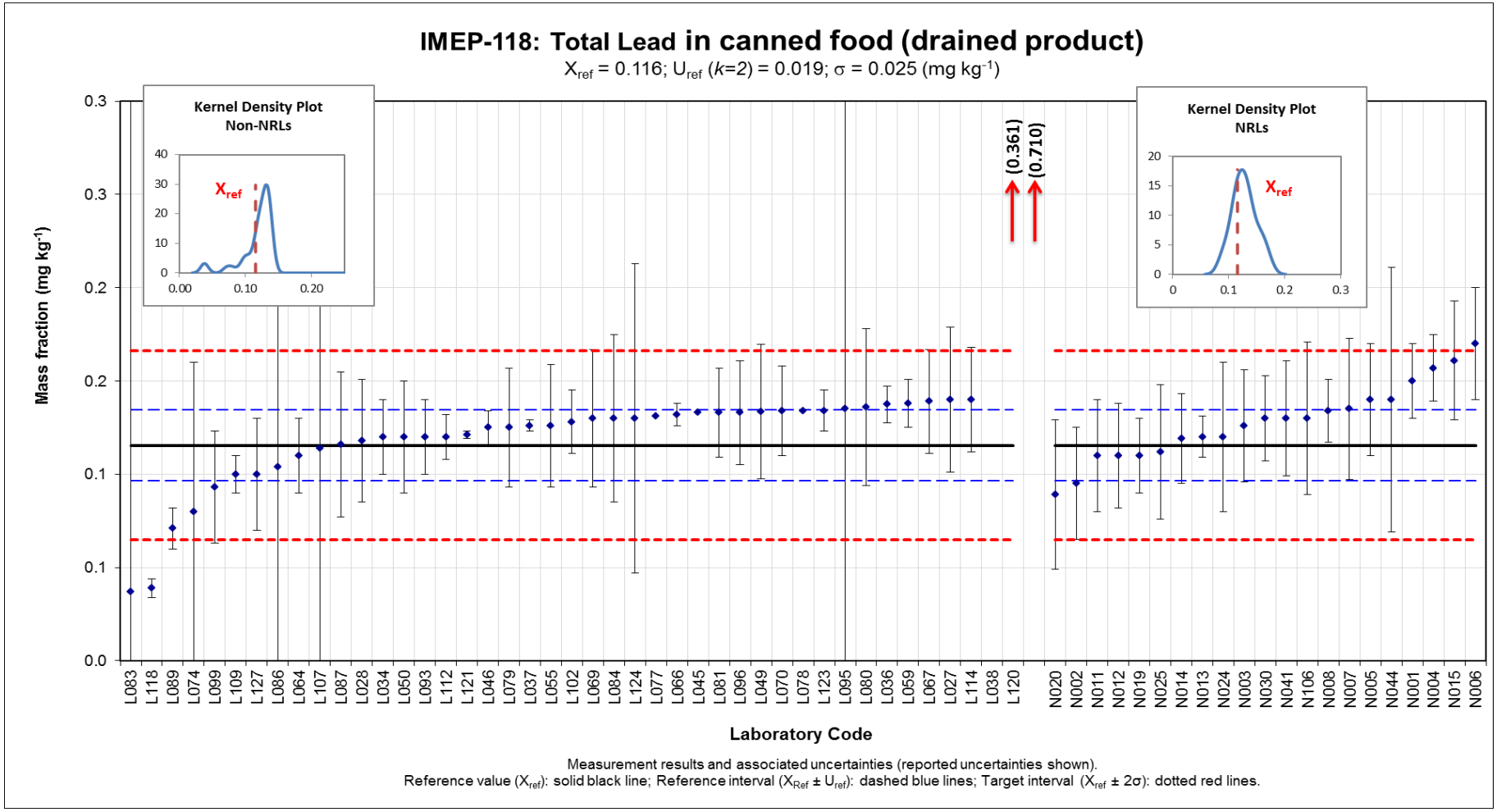
Assigned range: X_{ref} = 0.092; U_{ref} (k=2) = 0.012; σ = 0.020 (all values in mg kg^{-1})

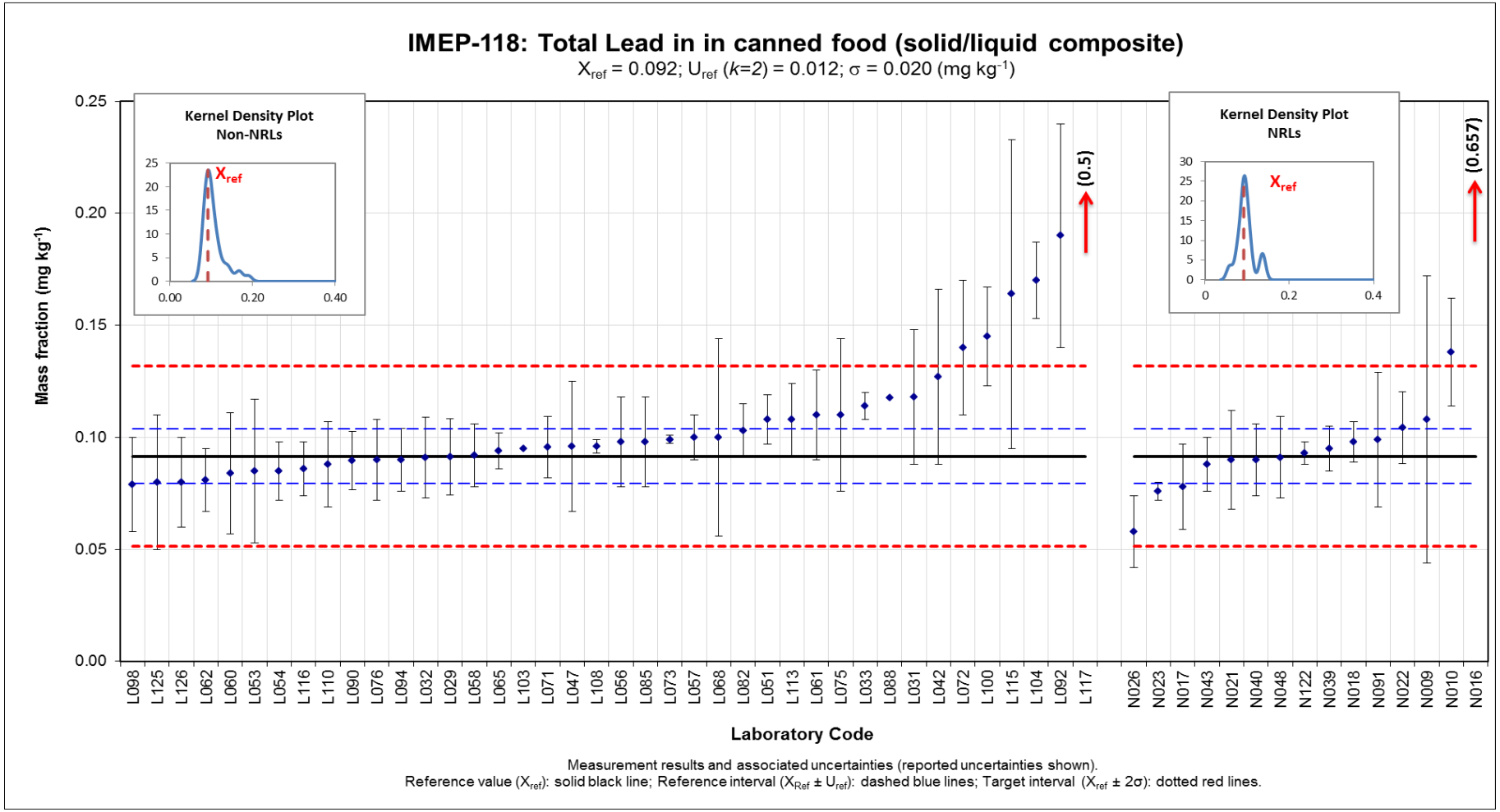
Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L029	0.0914	0.017	2	SFICP-MS	0.009	-0.01	-0.01	a
L031	0.118	0.03	2	ICP-MS	0.015	1.31	1.64	a
L032	0.091	0.018	2	ICP-MS	0.009	-0.03	-0.05	a
L033	0.114	0.006	2	ICP-MS	0.003	1.11	3.33	b
L042	0.127	0.039	2	ICPMS	0.020	1.75	1.74	a
L047	0.096	0.029	2	ETAAS	0.015	0.22	0.28	a
L051	0.108	0.011	2	ICP-MS	0.006	0.81	2.01	b
L053	0.085	0.032	2	ETAAS	0.016	-0.32	-0.38	a
L054	0.085	0.013	2	ETAAS	0.007	-0.32	-0.74	a
L056	0.098	0.02	2	ICP-MS	0.010	0.32	0.55	a
L057	0.1	0.01	√3	ICP-MS	0.006	0.42	1.01	b
L058	0.092	0.014	2	ICP-MS	0.007	0.02	0.05	a
L060	0.084	0.027	2	G-AAS	0.014	-0.37	-0.51	a
L061	0.11	0.02	2	ICP-MS	0.010	0.91	1.58	a
L062	0.081	0.014	2	ICP-MS	0.007	-0.52	-1.14	a
L065	0.094	0.008	√3	AAS	0.005	0.12	0.32	b
L068	0.1	0.044	2	HGA-AA	0.022	0.42	0.37	c
L071	0.0956	0.0138	2	GF-AAS	0.007	0.20	0.44	a
L072	0.14	0.03	2	ICP-AES	0.015	2.39	3.00	a
L073	0.0991	0.0019	90	ETAAS	0.000	0.37	1.25	b
L075	0.11	0.034	2	ICP-MS	0.017	0.91	1.02	a
L076	0.09	0.018	2	AAS	0.009	-0.08	-0.14	a
L082	0.103	0.012	√3	AAS	0.007	0.57	1.25	a
L085	0.098	0.02	2	SFICP-MS	0.010	0.32	0.55	a
L088	0.118			ICP-AES	0	1.28	4.29	b
L090	0.0895	0.013	2	ICP-MS	0.007	-0.10	-0.23	a
L092	0.19	0.05	2	ETAAS	0.025	4.87	3.83	c
L094	0.09	0.014	2	ICP-MS	0.007	-0.08	-0.17	a
L098	0.079	0.021	2	ICP-OES	0.011	-0.62	-1.04	a
L100	0.145	0.022	1	ICP-AES	0.022	2.64	2.34	c
L103	0.095			AAS	0	0.17	0.57	b
L104	0.17	0.017	1	ICP-MS	0.017	3.88	4.35	a
L108	0.096	0.003	3		0.001	0.22	0.73	b
L110	0.088	0.019	2	ICP-MS	0.010	-0.18	-0.32	a
L113	0.108	0.016	2	ICP-MS	0.008	0.81	1.64	a
L115	0.164	0.069	2	ETAAS	0.035	3.58	2.07	c
L116	0.086	0.012	2	ETAAS	0.006	-0.27	-0.65	b
L117	0.5	0.245	2	ETAAS	0.123	20.19	3.33	c
L125	0.08	0.03	2	FAAS	0.015	-0.57	-0.71	a
L126	0.08	0.02	2	ICP-MS	0.010	-0.57	-0.99	a
N009	0.108	0.064	2	ETAAS	0.032	0.81	0.51	c
N010	0.14	0.024	2	ICP-MS	0.012	2.30	3.46	a
N016	0.6507	0.13	√3	ICP-MS	0.075	27.64	7.43	c
N017	0.078	0.019	2	ETAAS	0.010	-0.67	-1.20	a
N018	0.098	0.009	2	ICP-MS	0.005	0.32	0.86	b
N021	0.09	0.022	2	AAS	0.011	-0.08	-0.12	a
N022	0.1043	0.016	2	ICP-MS	0.008	0.63	1.27	a
N023	0.076	0.004	2	ETAAS	0.002	-0.77	-2.44	b
N026	0.058	0.016	2	GF-AAS	0.008	-1.66	-3.35	a
N039	0.095	0.01	2	ICP-MS	0.005	0.17	0.44	b
N040	0.09	0.016	2	ICP-MS	0.008	-0.08	-0.15	a
N043	0.088	0.012	2	ICP-MS	0.006	-0.18	-0.42	b
N048	0.09	0.02	√3	ICP-MS	0.011	-0.03	-0.05	a
N091	0.099	0.03	2	FAAS	0.015	0.37	0.46	a
N122	0.093	0.005	2	ICP-MS	0.003	0.07	0.22	b

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

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

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


IMEP-118: Total Lead in canned food (drained product)
X_{ref} = 0.116; U_{ref} (*k*=2) = 0.019; σ = 0.025 (mg kg^{-1})
Kernel Density Plot
Non-NRLs
X_{ref}
Kernel Density Plot
NRLs
X_{ref}
(0.361)
(0.710)
Mass fraction (mg kg^{-1})
0.0
0.1
0.1
0.2
0.2
0.3
0.3
L083 L118 L089 L074 L099 L109 L127 L086 L064 L107 L087 L028 L034 L050 L093 L112 L121 L046 L079 L037 L055 L102 L069 L084 L124 L077 L066 L045 L081 L096 L049 L070 L078 L123 L095 L080 L036 L059 L067 L027 L114 L038 L120 N020 N002 N011 N012 N019 N025 N014 N013 N024 N003 N030 N041 N106 N008 N007 N005 N044 N001 N004 N015 N006
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{ref}): solid black line; Reference interval ($X_{Ref} \pm U_{ref}$): dashed blue lines; Target interval ($X_{ref} \pm 2\sigma$): dotted red lines.


IMEP-118: Total Lead in in canned food (solid/liquid composite)
X_{ref} = 0.092; U_{ref} (*k*=2) = 0.012; σ = 0.020 (mg kg^{-1})
Kernel Density Plot
Non-NRLs
X_{ref}
Kernel Density Plot
NRLs
X_{ref}
(0.5)
(0.657)
Mass fraction (mg kg^{-1})
0.25
0.20
0.15
0.10
0.05
0.00
L098 L125 L126 L062 L060 L053 L054 L116 L110 L090 L076 L094 L032 L029 L058 L065 L103 L071 L047 L108 L056 L085 L073 L057 L068 L082 L051 L113 L061 L075 L033 L088 L031 L042 L072 L100 L115 L104 L092 L117
N026 N023 N017 N043 N021 N040 N048 N122 N039 N018 N091 N022 N009 N010 N016
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{ref}): solid black line; Reference interval (X_{Ref} ± U_{ref}): dashed blue lines; Target interval (X_{ref} ± 2σ): dotted red lines.

Annex 13: Results for total Hg

Drained product

Lab Code	x_{lab}	U_{lab}	k	technique
L027	<0.01			DMA
L028	<0.01			FIMS
L034	<0.15			ICP-AES
L037	<0.01			ICP-MS
L038	<0.1			ICP-AES
L045	<0.05			FIMS
L046	<0.001			DMA
L049	<0.006			CV-AAS
L050	<0.1			DMA
L052	<1			ICP-AES
L055	0.00073	0.00008	2	DMA
L059	<0.0005			AFS
L066	<0.001			ICP-MS
L067	<0.02			CV-AFS
L069	0.03	0.041	2	ICP-MS
L070	<0.01			DMA
L074	<0.03			AAS
L077	<0.1			FIMS
L078	<0.005			ICP-MS
L079	<0.01			ICP-MS
L081	<0.019			ICP-AES
L083	<0.03			CV-AAS
L086	<0.005			DMA-80
L089	<0.06			CV-AAS
L093	<0.03			ICP-MS
L095	<0.001	10		AAS
L096	<0.005			DMA
L099	<0.001			AAS
L101	<0.2			ICP-AES
L102	<0.0043			ICP-MS
L105	<0.05			CV-AAS
L107	<0.005			ICP-MS
L109	0.13	0.02	2	thermal decomposition, gold amalgamation, and atomic absorption spectroscopy
L112	<0.02			ICP-MS
L114	0.032	0.006	2	FAAS-MHS
L118	0.09	0.017	2	CV-AAS
L121	<0.002			ICP-MS
L123	<0.005			CV-AAS
L127	<0.02			CV-ETA or FIAS)
N001	<0.01			ICP-MS
N002	<0.005			CV-AFS
N003	0.0002	0.00006	2	DMA
N004	<0.005			CV-AAS
N005	<0.05			ICP-MS
N006	<0.034			DMA
N007	<0.007			ICP-MS
N008	<0.0005			
N011	0			ICP-MS
N012	<0.01			DMA
N013	<0.0004			ICP-MS
N014	<0.0038			ICP-MS
N015	0.0005	0.0001	2	DMA
N019	<0.01			DMA
N020	0.001	0.001	2	ICP-MS
N024	<0.01			CV-AAS
N025	0.009	0.001	2	CV-AAS
N030	<0.009			CV-AAS
N041	<0.006			FIMS
N044	<0.005			ICP-MS

Solid / liquid composite

Lab Code	x_{lab}	U_{lab}	k	technique
L029	<0.005			SFICP-MS
L031	<0.005			ICP-MS
L032	<0.1			ICP-MS
L033	<0.0004			DMA
L042	<0.002			ICPMS
L051	0.0199	0.0029	2	ICP-MS
L053	0.01	0.004	2	CV-AAS
L054	<0.0005			DMA
L056	<0.01			CV-AAS
L057	<0.002			ICP-MS
L058	<0.001			CV-AAS
L060	<0.0034			KD-AAS
L061	<0.001			CV-AAS
L062	0.002			ICP-MS
L065	<0.004			FIMS
L068	<0.01			HG-AAS
L072	0.01	0.002	2	CV-AAS
L073	<0.003			CV-AAS
L075	0.0014	0.002	2	DMA
L076	<0.003			CV-AAS
L082	<0.01			CV-AAS
L085	<0.002			DMA
L088	0.0011			CV-AAS
L090	<0.0006			AFS
L092	0.0047	0.0014	2	CV-AAS
L094	<0.01			CV-AAS
L098	<0.003			ICP-OES
L100	0.0042	0.00067	1	DMA
L103	<0.02			CV-AAS
L104	0.00012	0.00001	1	FIMS
L108	<0.005			

Lab Code	x_{lab}	U_{lab}	k	technique
L110	<0.005			ICP-MS
L111	<0.06			DMA
L113	<0.005			ICP-MS
L116	<0.003			CV-AAS
L117	0.0007	0.0001	2	CV-AAS
L125	<0.03			HG-AAS
L126	0.01	0.002	2	ICP-MS
N009	<0.01			DMA
N010	0.013	0.003	2	AMA 254 Altec Ltd, Automated Mercury Analyser
N016	<0.0066			CV-AFS
N017	<0.01			CV-AAS
N018	<0.004			ICP-MS
N022	<0.001		2	DMA
N023	<0.1			CV-AAS
N026	<0.05			HG-AAS
N039	0.0004	0.0001	2	DMA
N040	<0.001			CV-AFS
N043	<0.0005			ICP-MS
N048	0.0004	0.000021		DMA

Annex 14: Results for total Sn

Drained product

Assigned range: X_{ref} = 275.5; U_{ref} (*k=2*) = 22.3; σ = 33.1 (al values in mg kg^{-1})

Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L027	254	50.89	2	ICP-AES	25.4	-0.64	-0.76	a
L034	396	79	2	ICP-AES	39.5	3.64	2.94	c
L038	178			ICP-AES	0	-2.94	-8.73	b
L045	38			ICP-AES	0	-7.18	-21.33	b
L046	327	21	2	ICP-MS	10.5	1.56	3.36	b
L050	300	48	2	ICP-MS	24	0.74	0.92	a
L052	370	37	2	ICP-AES	18.5	2.86	4.37	a
L055	304	46	2	ICP-MS	23	0.86	1.11	a
L059	329	18	2	ICP-MS	9	1.62	3.73	b
L064	330	25	2	FAAS	12.5	1.65	3.25	a
L066	316	9	2	ICP-MS	4.5	1.22	3.37	b
L067	294	44	2	ICP-MS	22	0.56	0.75	a
L069	2	0.4	2	ICP-MS	0	-8.27	-24.57	b
L070	304	38	2	ICP-MS	19	0.86	1.29	a
L077	250			ICP-AES	0	-0.77	-2.29	b
L078	86			ICP-MS	0	-5.74	-17.05	b
L079	302	19	2	ICP-MS	9.5	0.80	1.81	b
L081	282	19	2	ICP-AES	9.5	0.20	0.44	b
L083	287	20	√3	ICP-MS	11.5	0.34	0.71	a
L084	266	53.2	2.00	ETAAS	26.6	-0.29	-0.33	a
L086	280	20	7.6	ICP AES	2.6	0.14	0.40	b
L087	276	50	2	ICP-AES	25	0.01	0.02	a
L089	209	31.29	2	ICP-AES	15.6	-2.02	-3.49	a
L093	261	21	2	ICP-MS	10.5	-0.44	-0.95	b
L096	290	10	2	ICP-MS	5	0.44	1.18	b
L099	205	36.8	1	FAAS	36.8	-2.15	-1.85	c
L101	283	19	1	ICP-AES	19	0.23	0.35	a
L102	301	36	2	ICP-MS	18	0.77	1.20	a
L105	283	34	2	FAAS	17	0.23	0.37	a
L107	22	7	2	ICP-MS	3.5	-7.68	-21.75	b
L112	185	18.6	2	ICP-MS	9.3	-2.73	-6.21	b
L114	221	44	2	ICP-AES	22	-1.65	-2.21	a
L121	297	5.5	2	ICP-MS	2.75	0.65	1.87	b
L123	319	21	2	FAAS	10.5	1.31	2.84	b
L127	312	47	2	ICP-AES	23.5	1.10	1.40	a
N005	337	39	2	ICP-MS	19.5	1.86	2.74	a
N006	343	61.8	2	ETAAS	30.9	2.04	2.06	a
N007	307	61	2	ICP-MS	30.5	0.95	0.97	a
N008	364	15	2			2.7	7.9	b
N011	293	53	2	FAAS	26.5	0.53	0.61	a
N012	292	61	2	ICP-MS	30.5	0.50	0.51	a
N013	277	6	2	ICP-AES	3	0.04	0.13	b
N014	290	58	2	ICP-AES	29	0.44	0.47	a
N015	306	49	2	FAAS	24.5	0.92	1.13	a
N019	300	50	2	ICP-MS	25	0.74	0.89	a
N020	120	48	2	ICP-MS	24	-4.70	-5.88	a
N025	287	20.6	2	FAAS	10.3	0.33	0.72	b
N030	336	47	2	ICP-AES	23.5	1.83	2.32	a
N044	340	61	2	ICP-MS	30.5	1.95	1.99	a
N106	285	29	2	ETAAS	14.5	0.29	0.52	a

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

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

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

Solid / liquid composite

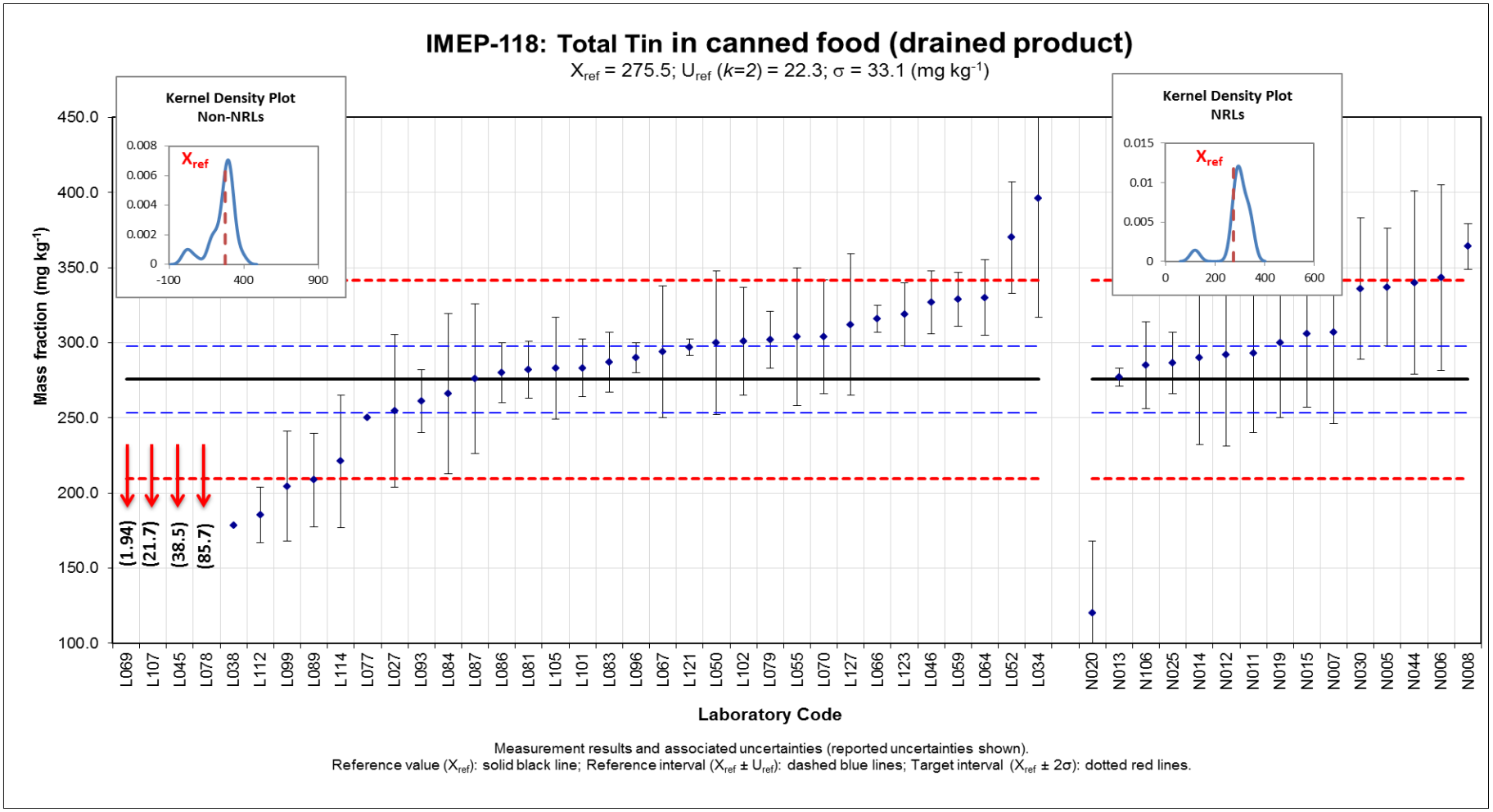
Assigned range: X_{ref} = 201.2; U_{ref} (k=2) = 16.2; σ = 24.1 (al values in mg kg^{-1})

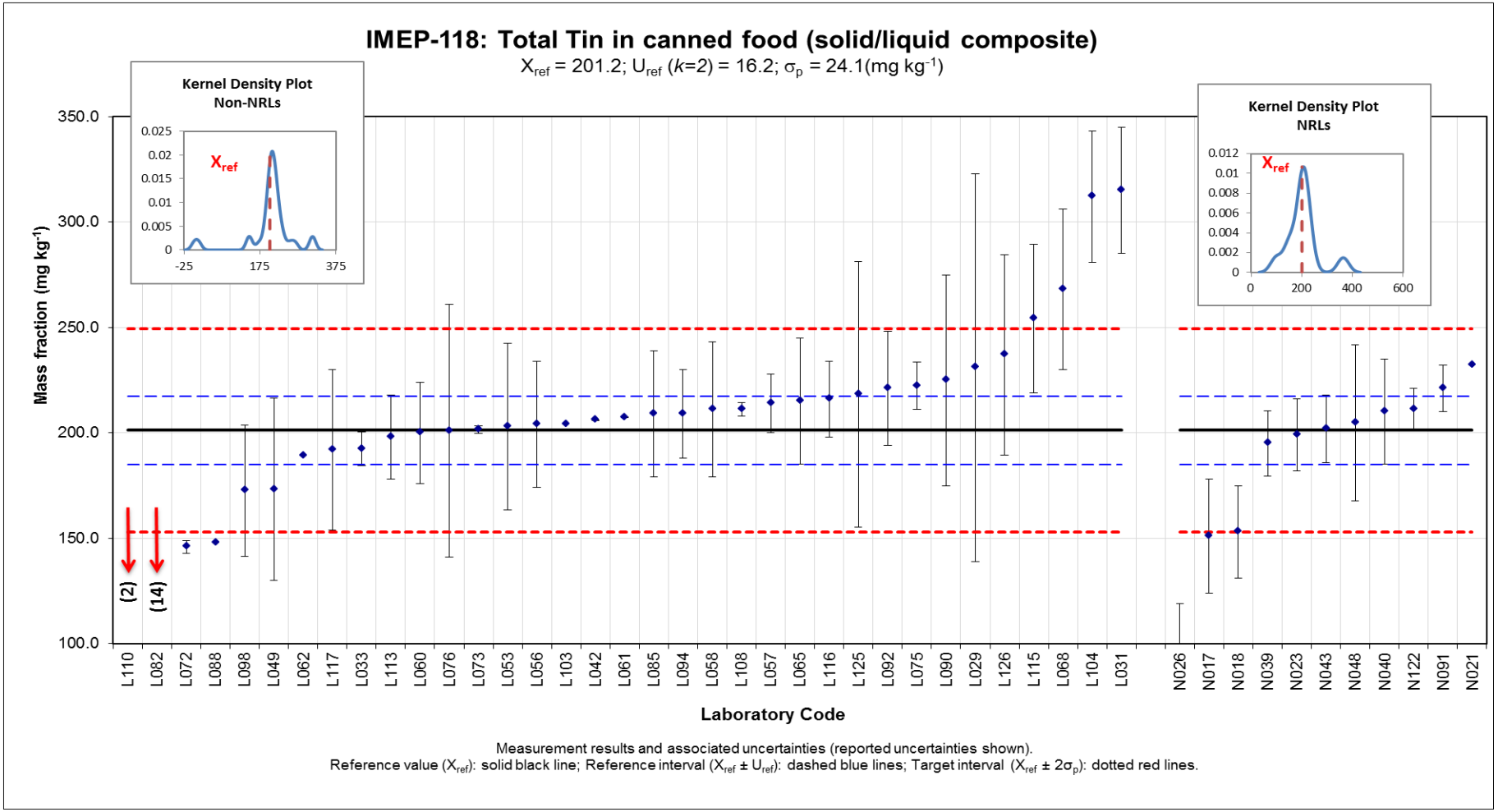
Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L029	231	92	2	SFICP-MS	46	1.23	0.64	c
L031	315	30	2	ICP-MS	15	4.71	6.67	a
L033	192	7.9	2	ICP-MS	3.95	-0.36	-0.98	b
L042	206	0.15	2	ICP-AES	0.075	0.20	0.59	b
L049	173.2	43.3	2	AAS	21.7	-1.16	-1.21	a
L053	203	39.6	2	ICP-AES	19.8	0.07	0.08	a
L056	204	30	2	ICP-MS	15	0.12	0.16	a
L057	214	14	1.732	ICP-MS	8.1	0.53	1.12	b
L058	211	32	2	ICP-MS	16	0.41	0.55	a
L060	200	24	2	ICP-OES	12	-0.05	-0.08	a
L061	207	0.3	2	ICP-AES	0.15	0.26	0.76	b
L062	189			ICP-MS	0	-0.51	-1.51	b
L065	215	30	1.732	FAAS	17.3	0.57	0.72	a
L068	268	38	2	HGA-AA	19	2.77	3.23	a
L072	146	3	2	ICP-AES	1.5	-2.29	-6.70	b
L073	202	1.87	100	ICP-MS	0.02	0.02	0.05	b
L075	222	11.1	2	ICP-MS	5.55	0.87	2.15	b
L076	201	60	2	AAS	30	-0.01	-0.01	c
L082	14	1.2	1.732	AAS	0.69	-7.75	-23.01	b
L085	209	30	2	SFICP-MS	15	0.32	0.46	a
L088	148			ICP-AES	0	-2.21	-6.57	b
L090	225	50	2	ICP-AES	25	0.99	0.91	c
L092	221	27	2	ICP-OES	13.5	0.82	1.26	a
L094	209	21	2	ICP-MS	10.5	0.32	0.59	a
L098	173	31.1	2	ICP-OES	15.6	-1.18	-1.63	a
L100	<0.5			ICP-AES				
L103	204			FAAS	0	0.12	0.34	b
L104	312	31.2	1	ICP-MS	31.2	4.59	3.44	c
L108	211	3.2	3		1.07	0.42	1.23	b
L110	2	0.61	2	ICP-MS	0.31	-8.24	-24.53	b
L113	198	20	2	ICP-MS	10	-0.13	-0.25	a
L115	254	35.3	2	ETAAS	17.7	2.19	2.73	a
L116	216	18	2	ETAAS	9	0.61	1.22	a
L117	192	38	2	FAAS	19	-0.38	-0.45	a
L125	218	63	2	FAAS	31.5	0.70	0.52	c
L126	237	47.4	2	ICP-MS	23.7	1.48	1.43	a
N017	151	27	2	ICP-AES	13.5	-2.08	-3.19	a
N018	153	22	2	ICP-MS	11	-2.00	-3.53	a
N021	232			AAS	0	1.28	3.80	b
N023	199	17	2	ETAAS	8.5	-0.09	-0.19	a
N026	99	19.85	2	ICP-AES	9.925	-4.22	-7.96	a
N039	195	15.6	2	ICP-MS	7.8	-0.26	-0.55	b
N040	210	25	2	ICP-MS	12.5	0.36	0.59	a
N043	202	16	2	ICP-MS	8	0.03	0.07	b
N048	205	36.85	1.732	ICP-MS	21.3	0.15	0.15	a
N091	221	11	2	FAAS	5.5	0.82	2.02	b
N122	211	10	2	ICP-MS	5	0.41	1.03	b

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

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

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


IMEP-118: Total Tin in canned food (drained product)
Xref = 275.5; Uref (k=2) = 22.3; σ = 33.1 (mg kg-1)
Kernel Density Plot
Non-NRLs
Xref
Kernel Density Plot
NRLs
Xref
Mass fraction (mg kg-1)
450.0
400.0
350.0
300.0
250.0
200.0
150.0
100.0
(1.94)
(21.7)
(38.5)
(85.7)
L069 L107 L045 L078 L038 L112 L099 L089 L114 L077 L027 L093 L084 L087 L086 L081 L105 L101 L083 L096 L067 L121 L050 L102 L079 L055 L070 L127 L066 L123 L046 L059 L064 L052 L034
N020 N013 N106 N025 N014 N012 N011 N019 N015 N007 N030 N005 N044 N006 N008
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (Xref): solid black line; Reference interval (Xref ± Uref): dashed blue lines; Target interval (Xref ± 2σ): dotted red lines.


IMEP-118: Total Tin in canned food (solid/liquid composite)
X_{ref} = 201.2; U_{ref} (*k*=2) = 16.2; σ_p = 24.1(mg kg^{-1})
Kernel Density Plot
Non-NRLs
X_{ref}
Kernel Density Plot
NRLs
X_{ref}
Mass fraction (mg kg^{-1})
350.0
300.0
250.0
200.0
150.0
100.0
(2)
(14)
L110 L082 L072 L088 L098 L049 L062 L117 L033 L113 L060 L076 L073 L053 L056 L103 L042 L061 L085 L094 L058 L108 L057 L065 L116 L125 L092 L075 L090 L029 L126 L115 L068 L104 L031 N026 N017 N018 N039 N023 N043 N048 N040 N122 N091 N021
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{ref}): solid black line; Reference interval ($X_{ref} \pm U_{ref}$): dashed blue lines; Target interval ($X_{ref} \pm 2\sigma_p$): dotted red lines.

Annex 15: Results for inorganic As

Drained product

Assigned range: X_{ref} = 0.098; U_{ref} (*k=2*) = 0.020; σ = 0.022 (al values in mg kg^{-1})

Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L027	0.105	0.026	2	HPLC-ICP-MS	0.013	0.32	0.42	a
L028	0.077	0.025	2	ICP-MS	0.013	-0.97	-1.31	a
L038	0.316				0	10.11	21.54	b
L052	<3.3			ICP-AES				
L055	0.081	0.015	2	HPLC-ICP-MS	0.008	-0.79	-1.35	b
L059	0.076	0.005	2	LC-ICP-MS	0.003	-1.02	-2.11	b
L070	0.093	0.019	2	ICP-MS	0.010	-0.23	-0.36	b
L087	<0.06			AFS				
L107	0.099	28	2	ICP-MS	14.0	0.05	0.00	c
N001	0.13	0.02	2	HPLC-ICP-MS	0.010	1.48	2.25	b
N002	0.07	0.03	2	HPLC-ICP-MS	0.015	-1.30	-1.55	a
N003	0.083	0.022	2	HPLC-ICP-MS	0.011	-0.70	-1.00	a
N007	0.154	0.04	2	LC-ICP-MS	0.020	2.60	2.50	a
N008	0.00365	0.00015	2		0.0001	-4.38	-9.32	b
N012	0.098	0.016	2	HPLC-ICP-MS	0.008	0.00	0.00	b
N013	0.093	0.015	2	LC-ICP-MS	0.008	-0.23	-0.40	b
N015	0.098	0.022	2	HG-AAS	0.011	0.00	0.00	a
N019	0.11	0.03	2	ICP-MS	0.015	0.56	0.66	a
N020	0.1	0.04	2	ICP-MS	0.020	0.09	0.09	a
N025	0.0770	0.013	2	HG-AAS	0.007	-0.97	-1.75	b
N106	0.11	0.011	2	HG-AAS	0.006	0.56	1.04	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] **Satisfactory, Questionable, Unsatisfactory**

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

Solid / liquid

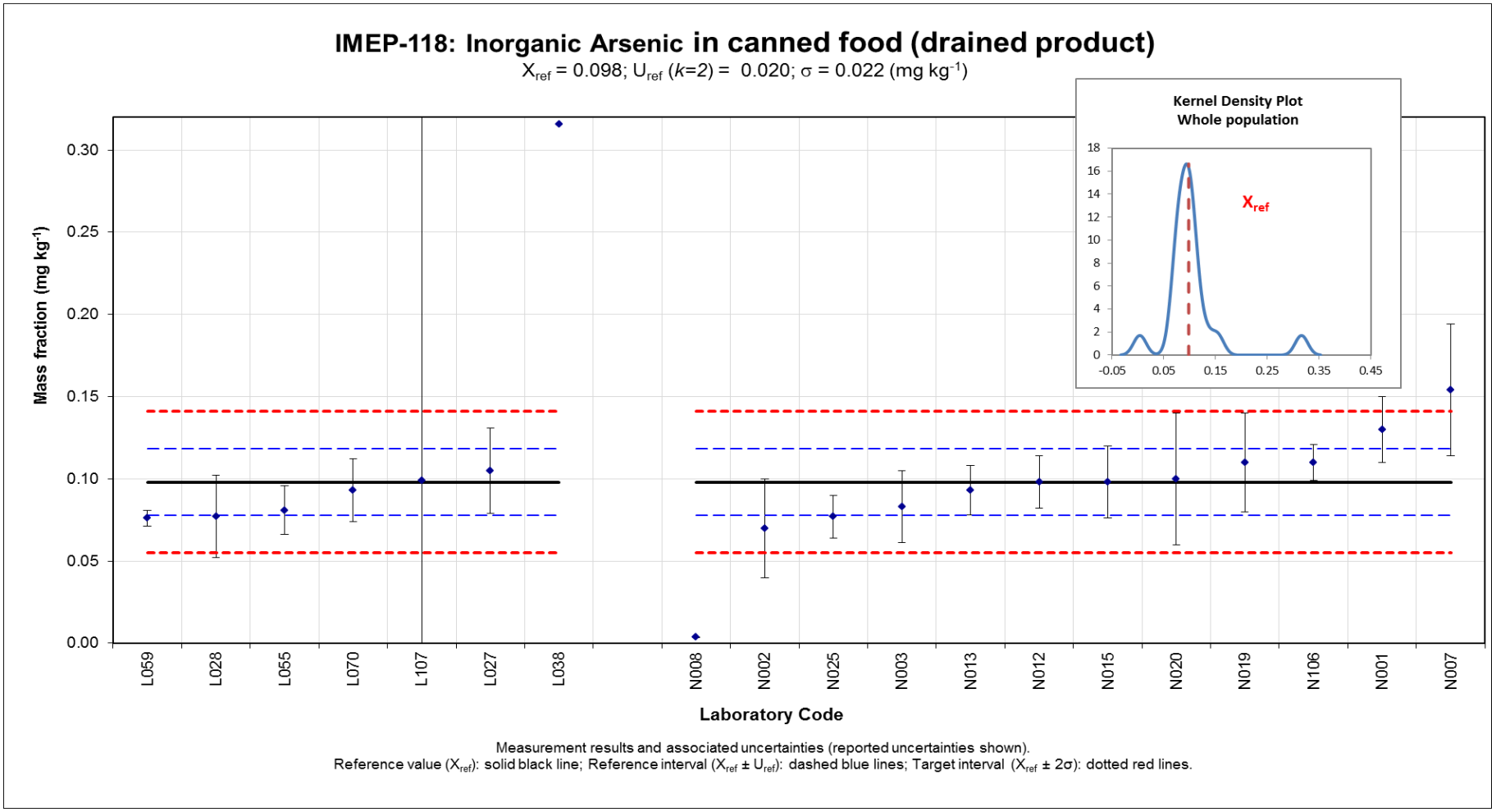
Assigned range: X_{ref} = 0.082; U_{ref} (*k=2*) = 0.008; σ = 0.018 (al values in mg kg^{-1})

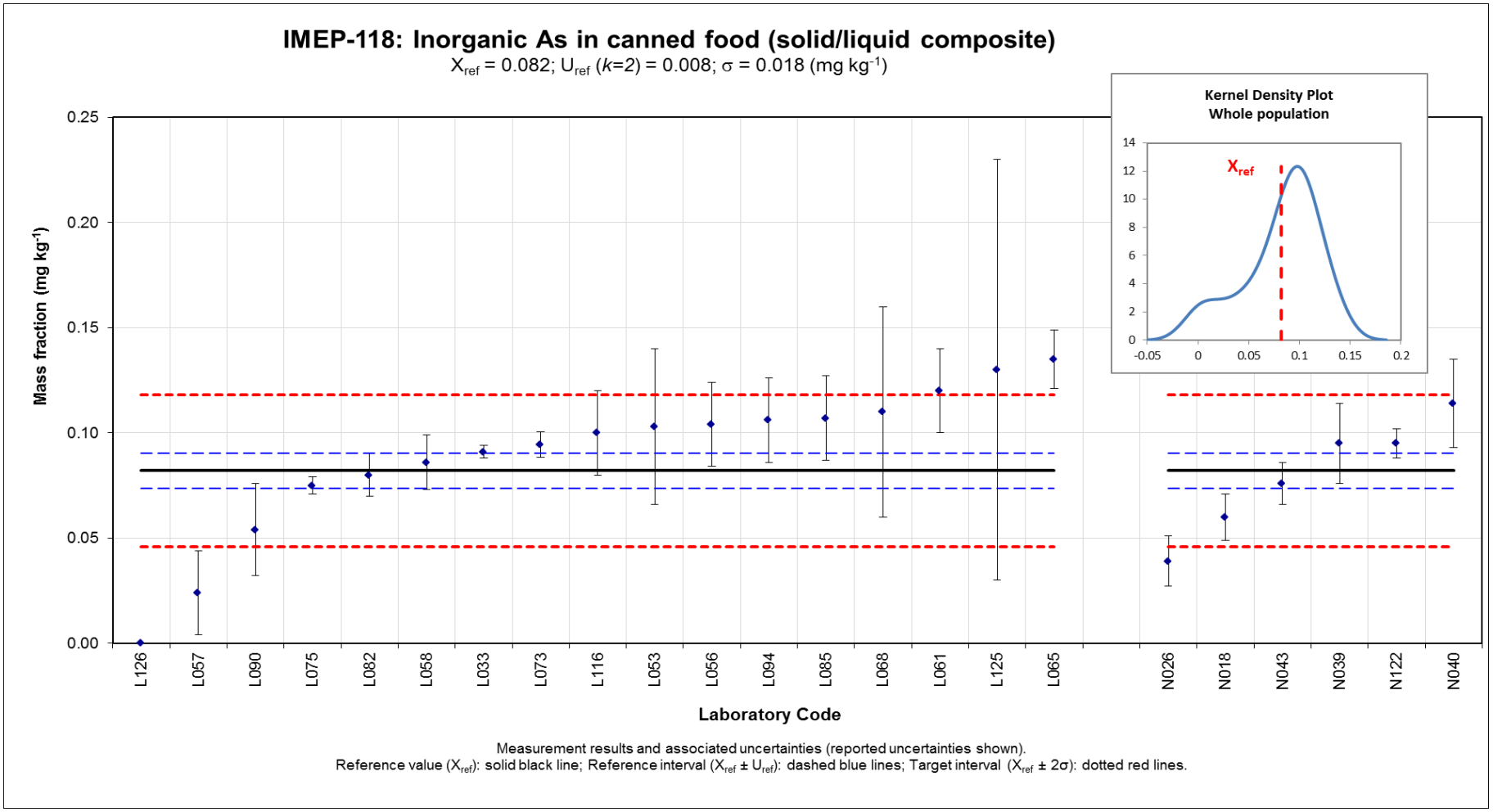
Lab Code	x_{lab}	U_{lab}	k[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	uncert.[c]
L033	0.091	0.003	2	HPLC-ICP-MS	0.002	0.50	2.00	b
L053	0.103	0.037	2	HG-AAS	0.019	1.17	1.11	c
L056	0.1040	0.02	2	LC-ICP-MS	0.010	1.22	2.03	a
L057	0.024	0.02	√3	HG-AAS	0.012	-3.22	-4.72	a
L058	0.086	0.013	2	LC-ICP-MS	0.007	0.22	0.52	a
L060	<0.031			H-AAS				
L061	0.120	0.02	2	AAS	0.010	2.11	3.50	a
L065	0.135	0.014	√3	H-AAS	0.008	2.94	5.81	a
L068	0.11	0.05	2	Hydride-ICP	0.025	1.56	1.10	c
L073	0.0944	0.0059	100	HG-AAS	0.000	0.69	2.93	b
L075	0.075	0.004	2	LC-ICP-MS	0.002	-0.39	-1.49	b
L082	0.08	0.01	√3	AAS	0.006	-0.11	-0.28	a
L085	0.107	0.02	2	IC-ICP-MS	0.010	1.39	2.30	a
L090	0.054	0.022	2	H-AAS	0.011	-1.56	-2.38	a
L094	0.106	0.02	2	HG-ICP-MS	0.010	1.33	2.21	a
L100	<0.1			ICP-AES				
L116	0.1	0.02	2	HG-AAS	0.010	1.00	1.66	a
L125	0.13	0.1	2	HG-AAS	0.050	2.7	0.96	c
L126	0	0	0					
N009	<0.1			HG-AAS				
N018	0.060	0.011	2	HPLC-ICP-MS	0.006	-1.22	-3.17	a
N026	0.039	0.012	2	HG-AAS	0.006	-2.39	-5.86	a
N039	0.095	0.019	2	HPLC-ICP-MS	0.010	0.72	1.25	a
N040	0.114	0.021	2	LC-ICP-MS	0.011	1.78	2.83	a
N043	0.076	0.01	2	ICP-MS	0.005	-0.33	-0.92	a
N122	0.095	0.007	2	HG-AAS	0.004	0.72	2.37	b

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

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

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


IMEP-118: Inorganic Arsenic in canned food (drained product)
Xref = 0.098; Uref (k=2) = 0.020; σ = 0.022 (mg kg-1)
Kernel Density Plot
Whole population
Xref
Mass fraction (mg kg-1)
0.00
0.05
0.10
0.15
0.20
0.25
0.30
L059
L028
L055
L070
L107
L027
L038
N008
N002
N025
N003
N013
N012
N015
N020
N019
N106
N001
N007
Laboratory Code
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (Xref): solid black line; Reference interval (Xref ± Uref): dashed blue lines; Target interval (Xref ± 2σ): dotted red lines.


IMEP-118: Inorganic As in canned food (solid/liquid composite)
X_{ref} = 0.082; U_{ref} (*k*=2) = 0.008; σ = 0.018 (mg kg^{-1})
Mass fraction (mg kg^{-1})
0.25
0.20
0.15
0.10
0.05
0.00
L126
L057
L090
L075
L082
L058
L033
L073
L116
L053
L056
L094
L085
L068
L061
L125
L065
N026
N018
N043
N039
N122
N040
Laboratory Code
Kernel Density Plot
Whole population
X_{ref}
14
12
10
8
6
4
2
0
-0.05
0
0.05
0.1
0.15
0.2
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{ref}): solid black line; Reference interval (X_{ref} ± U_{ref}): dashed blue lines; Target interval (X_{ref} ± 2σ): dotted red lines.

Annex 16: Experimental details and scoring

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
N001	D	The can was opened and the product was drained and dried at room temperature and then freezedried prior to analysis.	yes	Closed microwave	HNO_3	114	0.001	ICP-MS	b) No	As
N001	D		yes	Closed microwave	HNO_3	107	0.001	ICP-MS	b) No	Cd
N001	D		yes	Closed microwave	HNO_3	99	0.01	ICP-MS	b) No	Hg
N001	D		yes	Extraction with dilute HNO_3 and H_2O_2 in water-bath and analysis by anion-exchange HPLC-ICPMS with external calibration.		120	0.01	HPLC-ICP-MS	b) No	iAs
N001	D		yes	Closed microwave	HNO_3	98	0.012	ICP-MS	b) No	Pb
N002	D	Analyzing the product without the brine.		Closed microwave	HNO_3	97	0.03	ETAAS	b) No	As
N002	D			Closed microwave	HNO_3	98	0.003	ETAAS	b) No	Cd
N002	D			Closed microwave	HNO_3	103	0.005	CV-AFS	b) No	Hg
N002	D			HPLC/ICPMS		87	0.01	HPLC-ICP-MS	b) No	iAs
N002	D			Closed microwave	HNO_3	97	0.03	ETAAS	b) No	Pb
N003	D	the whole sample was drained in a colander, softly shaken to remove remaining fluid, and then mixed in a blender	IRMM 804	Closed microwave	HNO_3	114	0.0006	ICP-MS	b) No	As
N003	D		IRMM 804	Closed microwave	HNO_3	100	0.00015	ICP-MS	b) No	Cd
N003	D		BCR 150	X	X	106	0.000051	DMA	b) No	Hg
N003	D		NMIJ 7503a	9 ml HNO3 0.11M and 1 ml H2O2 30% were added to about 1 g of homogenized sample in a microwave tube. Under constant stirring, the sample is extracted at 90°C (7+20 min). After cooling, the solution is centrifuged (10 min, 12500 g). The supernatans is filtered over a 0.45 µm filter and then analysed by HPLC-ICP-MS		95	0.0006	HPLC-ICP-MS	b) No	iAs
N003	D		IRMM 806	Closed microwave	HNO_3	102	0.0009	ICP-MS	b) No	Pb
N004	D	Carefully take out of liquid and homogenize by blender	IMEP-114	Closed microwave	H_2O_2, HNO_3	95	0.01	ICP-MS	b) No	As
N004	D		IMEP-114	Closed microwave	H_2O_2, HNO_3	97	0.005	ICP-MS	b) No	Cd
N004	D		-	Open wet	H_2SO_4, HNO_3	-	0.005	CV-AAS	b) No	Hg
N004	D		IMEP-114	Closed microwave	H_2O_2, HNO_3	94	0.01	ICP-MS	b) No	Pb

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
N005	D	Drained contents of glass jar through a sieve. The peas were homogenised in a food blender and the slurry was used for the analyses.	LGC7162	Closed microwave	$H2O_2$, HNO_3	86.1	0.05	ICP-MS	b) No	As
N005	D		LGC7162	Closed microwave	H2O2, HNO3	87.5	0.003	ICP-MS	b) No	Cd
N005	D		TORT-2	Closed microwave	H2O2, HNO3	90.5	0.01	ICP-MS	b) No	Hg
N005	D		LGC7162	Closed microwave	H2O2, HNO3	88.3	0.03	ICP-MS	b) No	Pb
N005	D		NIST1548A	Closed microwave	HCL, HNO3	84.5	5	ICP-MS	b) No	Sn
N006	D		BCR482	Closed microwave	H2O2, HF, HNO3	99.14	0.18	ETAAS	b) No	As
N006	D			Closed microwave	H2O2, HF, HNO3	104.29	0.007	ICP-MS	b) No	Cd
N006	D		NIST1573A	X	X	93.3	0.01	DMA	b) No	Hg
N006	D		BCR482	Closed microwave	H2O2, HF, HNO3	104.93	0.008	ICP-MS	b) No	Pb
N006	D			Closed microwave	HCL, HNO3	111.75	2	ETAAS	b) No	Sn
N007	D	The contents were poured in a sieve and the liquid was discarded. Peas were then homogenised.	Spinach 1570a NIST	Closed microwave	HCL, HNO3	100	0.002	ICP-MS	b) No	As
N007	D		Spinach 1570a NIST	Closed microwave	HCL, HNO3	100	0.0005	ICP-MS	b) No	Cd
N007	D		Spinach 1570a NIST	Closed microwave	HCL, HNO3	100	0.004	ICP-MS	b) No	Hg
N007	D		IMEP-112, NMIJ 7503-a	0.2 gram of sample is extracted by 10 ml of 0.1 M HNO3 and 3% H2O2 at 90 degrees C for one hour. After centrifugation and filtering the solution is analysed by strong anion exchange chromatography ICP-MS		100	0.003	LC-ICP-MS	b) No	iAs
N007	D		Spinach 1570a NIST	Closed microwave	HCL, HNO3	100	0.001	ICP-MS	b) No	Pb
N007	D		Spinach 1570a NIST	Closed microwave	HCL, HNO3	100	0.015	ICP-MS	b) No	Sn
N008	D	EN 13804:2013	BCR 186 - RF standard	Closed microwave	H2O2, HNO3	99	0.008		a) Yes	As
N008	D		ERM-BC084a - RF standard	Closed microwave	H2O2, HNO3	101	0.006		a) Yes	Cd
N008	D		LGC-Phytas 016 - RF standard	X	X	98	0.0005		a) Yes	Hg
N008	D		RF standard	Protocol for determination of iAs in food samples (IMEP-41)		105	0.01		a) Yes	iAs
N008	D		ERM-BC084a - RF standard	Closed microwave	H2O2, HNO3	95	0.02		a) Yes	Pb
N008	D		ERM-BC084a - RF standard	Open wet	HCL	92	5		a) Yes	Sn

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
N009	S/L	knife mill - three independent measurements	internal ref.	Closed microwave	HNO3		0.03	ETAAS	a) Yes	As
N009	S/L		internal ref.	Closed microwave	HNO3		0.002	ETAAS	a) Yes	Cd
N009	S/L		External Ref. Mat (BIPEA) - internal ref.	X	X		0.003	DMA	a) Yes	Hg
N009	S/L		External Ref. Mat (BIPEA) - internal ref.	hydride generation atomic absorption spectrometry (HGAAS) after acid extraction			0.03	HG-AAS	a) Yes	iAs
N009	S/L		internal ref.	Closed microwave	HNO3		0.015	ETAAS	a) Yes	Pb
N010	S/L	stored in the fridge at 4°C till homogenization then digested with HNO3 - H2O2	Dolt3	Open wet	H2O2, HNO3	90.9	0.000006	ICP-MS	a) Yes	As
N010	S/L		EURL CEFAO 18th PT	Open wet	H2O2, HNO3	98.6	0.000006	ICP-MS	a) Yes	Cd
N010	S/L		Dolt3	Open wet	H2O2, HNO3	100.6	7.23E-05	AMA 254 Altec Ltd, Automated Mercury Analyser	a) Yes	Hg
N010	S/L		EURL CEFAO 18th PT	Open wet	H2O2, HNO3	98.9	1.87E-05	ICP-MS	a) Yes	Pb
N011	D	We seperated peas from brine. Then mixed only peas and take a portion for digestion.		Closed microwave	H2O2, HNO3	111	0.01	ICP-MS	a) Yes	As
N011	D		Yes	Closed microwave	H2O2, HNO3	98	0.003	ICP-MS	a) Yes	Cd
N011	D			Closed microwave	H2O2, HNO3	92	0.003	ICP-MS	a) Yes	Hg
N011	D		Yes	Closed microwave	H2O2, HNO3	98	0.003	ICP-MS	a) Yes	Pb
N011	D		Yes	Open wet	HCL, HNO3	109	6	FAAS	a) Yes	Sn
N012	D	Draining for 3 minutes at room temperature on a mesh screen 3mm and then homogenization.		Closed microwave	HNO3	95		ICP-MS	b) No	As
N012	D			Closed microwave	HNO3	101		ICP-MS	b) No	Cd
N012	D			X	X	98		DMA	b) No	Hg
N012	D			HPLC-ICP-MS				HPLC-ICP-MS	b) No	iAs
N012	D			Closed microwave	HNO3	101		ICP-MS	b) No	Pb
N012	D			Open wet	HCL	97		ICP-MS	b) No	Sn
N013	D	After draining we freeze dried the sample before decomposition	Peach Leaves SRM 1547 - Std. curve	Closed microwave	HNO3	104	0.002	ICP-MS	b) No	As
N013	D		Peach Leaves SRM 1547 - Std. curve	Closed microwave	HNO3	105	0.0005	ICP-MS	b) No	Cd
N013	D		Peach Leaves SRM 1547 - Std. curve	Closed microwave	HNO3	104	0.0004	ICP-MS	b) No	Hg
N013	D		Rice ERM BC211 - std. curve	HPLC-ICP-MS		104	0.03	LC-ICP-MS	b) No	iAs

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
N013	D		Peach Leaves SRM 1547 - Std. curve	Closed microwave	HNO3	99	0.001	ICP-MS	b) No	Pb
N013	D		Fapas T0758 - std. curve	Closed microwave	HCL, HNO3	104	0.6	ICP-AES	b) No	Sn
N014	D	Open can, let the peas drain (through a sieve), wash the peas with water, let them drain again (through a sieve), homogenize them	NIST 1547	Closed microwave	HNO3		0.0075	ICP-MS	b) No	As
N014	D		NIST 1547	Closed microwave	HNO3		0.0018	ICP-MS	b) No	Cd
N014	D		NIST 1547	Closed microwave	HNO3		0.0038	ICP-MS	b) No	Hg
N014	D		NIST 1547	Closed microwave	HNO3		0.006	ICP-MS	b) No	Pb
N014	D			Closed microwave	HCL, HNO3		7.5	ICP-AES	b) No	Sn
N015	D	Brine was removed by draining then drained peas was homogenised	NIST 1566b	Dry ashing	HNO3	95	0.025	HG-AAS	b) No	As
N015	D		NIST 1566b, CTA-OTL-1	Closed microwave	H2O2, HNO3	102	0.002	ETAAS	b) No	Cd
N015	D		1566b, 1568a, BCR-422	X	X	100	0.0002	DMA	b) No	Hg
N015	D		control matrial (after PT	Sample was hydrolysed using concentrated hydrochloric acid. After reduction by hydrobromic acid and hydrazine sulfate, the inorganic arsenic was extracted into chloroform, then back-extracted into 1M HCl, dry-ashed, and quantified by HG-AAS		74	0,027	HG-AAS	b) No	iAs
N015	D		CTA-OTL-1,1566b	Closed microwave	H2O2, HNO3	87	0.012	ETAAS	b) No	Pb
N015	D		control material (PT)	Closed microwave	HCL, HNO3	101	3.5	FAAS	b) No	Sn
N016	S/L	Mixed all the solid and liquid in the can with a hand blender.		Closed microwave	H2O2, HNO3			ICP-MS	X	As
N016	S/L			Closed microwave	H2O2, HNO3			ICP-MS	X	Cd
N016	S/L			Closed microwave	H2O2, HNO3			CV-AFS	X	Hg
N016	S/L			Closed microwave	H2O2, HNO3			ICP-MS	X	Pb
N017	S/L	all sample homogenized	SRM1568a	Dry ashing	Other		0.05	HG-AAS	a) Yes	As
N017	S/L		SRM1568a	Closed microwave	H2O2, HNO3		0.01	ETAAS	a) Yes	Cd
N017	S/L		IMEP-110	Closed microwave	H2O2, HNO3		0.01	CV-AAS	a) Yes	Hg
N017	S/L		IMEP110	Closed microwave	H2O2, HNO3		0.01	ETAAS	a) Yes	Pb
N017	S/L		FAPAS07116	Closed microwave	HCL, HNO3		5	ICP-AES	a) Yes	Sn
N018	S/L	mixing the sample with the water of the jar until homogenization	NIST 2976 - Standard	Closed microwave	HNO3		0.001	ICP-MS	b) No	As
N018	S/L		NIST 2976 - Standard	Closed microwave	HNO3		0.0003	ICP-MS	b) No	Cd

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
N018	S/L		NIST 2976 - Standard	Closed microwave	HNO3		0.004	ICP-MS	b) No	Hg
N018	S/L		BC-211 - Standard	microwave assisted extraction with water			0.001	HPLC-ICP-MS	b) No	iAs
N018	S/L		NIST 2976 - Standard	Closed microwave	HNO3		0.001	ICP-MS	b) No	Pb
N018	S/L		NIST 1548a - Standard	Closed microwave	HNO3		0.042	ICP-MS	b) No	Sn
N019	D	we drained the liquid through the plastic sieve, than we homogenised the sample in ceramic homogenizer	CRM ZC 73012	Closed microwave	H2O2, HNO3	97	0.02	ICP-MS	b) No	As
N019	D		SRM ZC 73012	Closed microwave	H2O2, HNO3	100	0.005	ICP-MS	b) No	Cd
N019	D		SRM ZC 73012	X	X	99	0.005	DMA	b) No	Hg
N019	D		IMEP 116	we used the modified method EN 16278:2012 (ICP-MS determination)		99	0.05	ICP-MS	b) No	iAs
N019	D		SRM ZC 73012	Closed microwave	H2O2, HNO3	96	0.01	ICP-MS	b) No	Pb
N019	D		-	Closed microwave	H2O2, HCL, HNO3		0.05	ICP-MS	b) No	Sn
N020	D	dry freezing	Oyster tissue	Closed microwave	HNO3			ICP-MS	X	As
N020	D		oyster tissue	Closed microwave	HNO3			ICP-MS	X	Cd
N020	D		oyster tissue	Closed microwave	HNO3			ICP-MS	X	Hg
N020	D		oyster tissue					ICP-MS	X	iAs
N020	D		oyster tissue	Closed microwave	HNO3			ICP-MS	X	Pb
N020	D		oyster tissue	X	HNO3			ICP-MS	X	Sn
N021	S/L	HOMOGENISATION OF THE WHOLE SAMPLE	IMEP111, IMEP 117	Closed microwave	H2O2, HNO3	80-110	0.001	AAS	b) No	Cd
N021	S/L		IMEP111, IMEP 114	Closed microwave	H2O2, HNO3	80-110	0.0033	AAS	b) No	Pb
N021	S/L			Closed microwave	H2O2, HNO3			AAS	b) No	Sn
N022	S/L		SRM 1643e - VAR-CAL-2 INorg. Vent.	Closed microwave	H2O2, HNO3	94.3	0.0005	ICP-MS	b) No	As
N022	S/L		SRM 1643e - VAR-CAL-2 INorg. Vent.	Closed microwave	H2O2, HNO3	101.5	0.0005	ICP-MS	b) No	Cd
N022	S/L		TORT 2	Dry ashing	X	116	0.001	DMA	b) No	Hg
N022	S/L		SRM 1643e - VAR-CAL-2 INorg. Vent.	Closed microwave	H2O2, HNO3	94.3	0.005	ICP-MS	b) No	Pb
N023	S/L	All content is mixed by a blender to produce a homogeneous mixture. From this mixture 0.2 -	RM WEPAL	Closed microwave	H2O2, HNO3	100	0.1	HG-AAS	b) No	As
N023	S/L		RM WEPAL	Closed microwave	H2O2, HNO3	95.4	0.01	ETAAS	b) No	Cd

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
N023	S/L	1.0g are taken for digestion.		Closed microwave	HNO3	95	0.1	CV-AAS	b) No	Hg
N023	S/L		RM WEPAL	Closed microwave	H2O2, HNO3	91.2	0.02	ETAAS	b) No	Pb
N023	S/L			Closed microwave	HCL, HF, HNO3	91	1	ETAAS	b) No	Sn
N024	D							AAS		As
N024	D							AAS		Cd
N024	D							CV-AAS		Hg
N024	D							AAS		Pb
N025	D	Brine was drained. The peas were transferred to a stomacher bag and homogenised using a stomacher until the sample became homogegous.		Dry ashing	HCL, HNO3		0.016	HG-AAS	b) No	As
N025	D		Past PT material	Open wet	H2O2, HNO3		0.004	ETAAS	b) No	Cd
N025	D			Open wet	H2O2, HNO3		0.005	CV-AAS	b) No	Hg
N025	D		Past PT material	1. Hydrolysis step using HCl. 2. Reduction and chloroform extraction. 3. Clean-up step. 4. Back extraction in 1M HCl. 5. Dry ashing and quantification by HG-AAS.			0.006	HG-AAS	b) No	iAs
N025	D		Past PT material	Open wet	H2O2, HNO3		0.03	ETAAS	b) No	Pb
N025	D		Past PT material	Open wet	HCL		25	FAAS	b) No	Sn
N026	S/L	Mixed and homogenised all amount of the jar in the original vessel with a hand blender.Weighed out three parallel from this solid matrix and lyophilized the remainder part.We also analised three parallel fom the lyophilized material.	IMEP112 - Merck	Closed microwave	H2O2, HNO3	88	0.04	HG-AAS	X	As
N026	S/L		IMEP117 - Merck	Closed microwave	H2O2, HNO3	120	0.04	GF-AAS	X	Cd
N026	S/L		IMEP117 - CaPurAn	Closed microwave	H2O2, HNO3		0.05	HG-AAS	X	Hg
N026	S/L		IMEP112	The known chloroform extraction method followed muffle furnace mineralization at 425C and HG-AAS measurament .			0.04	HG-AAS	X	iAs
N026	S/L		IMEP114 - Merck	Closed microwave	H2O2, HNO3	72	0.04	GF-AAS	X	Pb
N026	S/L		IMEP114 - SCPScience	Closed microwave	H2O2, HNO3		0.1	ICP-AES	X	Sn
L027	D	decant the liquid and homogenized the solid	bipea, fapas samples, CRM - Custom made solution	Closed microwave	H2O2, HNO3	100	0.05	ICP-MS	b) No	As
L027	D		bipea, fapas samples, CRM - Custom made solution	Closed microwave	H2O2, HNO3	100	0.01	ICP-MS	b) No	Cd

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L027	D		bipea, fapas samples, CRM - Custom made solution	Closed microwave	H2O2, HNO3	100	0.01	DMA	b) No	Hg
L027	D		bipea, fapas samples, CRM - Custom made solution	Jens Sloth proposed EN method: acid extraction in heated waterbath - HPLC-ICPMS		100	0.02	HPLC-ICP-MS	b) No	iAs
L027	D		bipea, fapas samples, CRM - Custom made solution	Closed microwave	H2O2, HNO3	100	0.02	ICP-MS	b) No	Pb
L027	D		bipea, fapas samples, CRM - Custom made solution	Closed microwave	H2O2, HF, HNO3	100	2.5	ICP-AES	b) No	Sn
L028	D	Wash, leak and mix the sample	NIST 1570a Spinach leaves	Closed microwave	H2O2, HNO3	110.54	0.05	ICP-MS	b) No	As
L028	D		IRMM 804 Rice	Closed microwave	H2O2, HNO3	105.2	0.01	ICP-MS	b) No	Cd
L028	D		NIST 1570a Spinach leaves	Closed microwave	H2O2, HNO3	89.39	0.01	FIMS	b) No	Hg
L028	D			Determination of inorganic arsenic by ICP-MS after microwave extraction and separation by solid phase extraction (SPE)		95.32	0.05	ICP-MS	b) No	iAs
L028	D		IRMM 804 Rice	Closed microwave	H2O2, HNO3	103.81	0.02	ICP-MS	b) No	Pb
L029	S/L		CRM, prim stand.	Closed microwave	HF, HNO3		0.005	SFICP-MS	b) No	As
L029	S/L		CRM, prim stand.	Closed microwave	HF, HNO3		0.002	SFICP-MS	b) No	Cd
L029	S/L		CRM, prim stand.	Closed microwave	HF, HNO3		0.005	SFICP-MS	b) No	Hg
L029	S/L		CRM, prim stand.	Closed microwave	HF, HNO3		0.01	SFICP-MS	b) No	Pb
L029	S/L		CRM, prim stand.	Closed microwave	HF, HNO3		0.02	SFICP-MS	b) No	Sn
N030	D	We separated the peas from the brine and analysed the drained peas		Closed microwave	H2O2, HNO3	101	0.02	AAS	b) No	As
N030	D			Closed microwave	H2O2, HNO3	107.3	0.0008	AAS	b) No	Cd
N030	D			Closed microwave	H2O2, HNO3		0.009	CV-AAS	b) No	Hg
N030	D			Closed microwave	H2O2, HNO3	99.9	0.008	AAS	b) No	Pb
N030	D			Closed microwave	H2O2, HNO3	83	1.5	ICP-AES	b) No	Sn
L031	S/L	homogenisation, mixing	NIST8436	Closed microwave	H2O2, HCL, HNO3		0.1	ICP-MS	X	As
L031	S/L		NIST8436	Closed microwave	H2O2, HCL, HNO3		0.01	ICP-MS	X	Cd

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L031	S/L		NIST8436	Closed microwave	H2O2, HCL, HNO3		0.005	ICP-MS	X	Hg
L031	S/L		NIST8436	Closed microwave	H2O2, HCL, HNO3		0.05	ICP-MS	X	Pb
L031	S/L			Closed microwave	H2O2, HCL, HNO3		0.1	ICP-MS	X	Sn
L032	S/L	Homogénéisé et broyé la totalité de l'échantillon - Prise d'essai de 1 g	ERM CE278K	Open wet	HNO3		0.1	ICP-MS	b) No	As
L032	S/L		ERM CE278K	Open wet	HNO3		0.003	ICP-MS	b) No	Cd
L032	S/L		ERM CE278K	Open wet	HNO3		0.033	ICP-MS	b) No	Hg
L032	S/L		ERM CE278K	Open wet	HNO3		0.007	ICP-MS	b) No	Pb
L033	S/L	All samples were mixed by the homogenizer	NIST1568A	Closed microwave	H2O2, HNO3	103	0.001	ICP-MS	a) Yes	As
L033	S/L		NIST1568A,Fapas07205	Closed microwave	H2O2, HNO3	98	0.001	ICP-MS	a) Yes	Cd
L033	S/L		GBW10010	X	X	98	0.00008	DMA	a) Yes	Hg
L033	S/L		IST1568A	The sample is extracted by 0.15 mol/L nitric acid at 90 ℃ for 3 h , centrifuged at 9000 rpm for 10min and filtered by 0.45 um membrane		100	0.005	HPLC-ICP-MS	a) Yes	iAs
L033	S/L		Fapas07205	Closed microwave	H2O2, HNO3	97	0.001	ICP-MS	a) Yes	Pb
L033	S/L		Fapas07205	Closed microwave	H2O2, HNO3	90	0.008	ICP-MS	a) Yes	Sn
L034	D	is allowed to drain through a sieve for 3 minutes		Open wet	H2O2, HNO3	95	0.15	ICP-AES	b) No	As
L034	D			Dry ashing	HCL	97	0.02	ICP-AES	b) No	Cd
L034	D			Open wet	H2O2, HNO3	95	0.15	ICP-AES	b) No	Hg
L034	D			Dry ashing	HCL	91	0.05	ICP-AES	b) No	Pb
L034	D			Open wet	HCL, HNO3	90	0.3	ICP-AES	b) No	Sn
L036	D	BAS EN ISO 13804 Performance criteria, general considerations and sample preparation- (Processed food -canned food, frozen food) Remove the sauce, brine or other medium which is normaly not eaten, by draning		Dry ashing	HNO3	96.3	0.01	H-AAS	b) No	As
L036	D			Closed microwave	H2O2, HNO3	101.2	0.001	ETAAS	b) No	Cd
L036	D			Closed microwave	H2O2, HNO3	96.3	0.01	ETAAS	b) No	Pb
L037	D	We analysed both the solid and the liquid composite, we reported only the solid product	yes	Closed microwave	H2O2, HNO3	125	0.066	ICP-MS	b) No	As
L037	D		yes	Closed microwave	H2O2, HNO3	103.5	0.005	ICP-MS	b) No	Cd
L037	D		yes	Closed microwave	H2O2, HNO3	96.3	0.01	ICP-MS	b) No	Hg

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L037	D		yes	Closed microwave	H2O2, HNO3	86.6	0.008	ICP-MS	b) No	Pb
L038	D	dried, ashed and diluted in 2N nitric acid solution		Dry ashing	HNO3	70.9		ICP-AES	a) Yes	As
L038	D			Dry ashing	HNO3	95.3		ICP-AES	a) Yes	Cd
L038	D			Dry ashing	HNO3	60.2		ICP-AES	a) Yes	Hg
L038	D								a) Yes	iAs
L038	D			Dry ashing	HNO3	98.8		ICP-AES	a) Yes	Pb
L038	D			Dry ashing	HNO3	101.7		ICP-AES	a) Yes	Sn
N039	S/L	We have mixed all sample (pea with liquid).	IAEA-336	Closed microwave	H2O2, HNO3	102	0.0002	ICP-MS	b) No	As
N039	S/L		DORM-4	Closed microwave	H2O2, HNO3	101	0.0001	ICP-MS	b) No	Cd
N039	S/L		CZ9024	X	Other	99	0.0001	DMA	b) No	Hg
N039	S/L		IMEP32-7	Determination by HPLC-ICP-MS after microwave assisted extraction.		86	0.008	HPLC-ICP-MS	b) No	iAs
N039	S/L		IAEA-336	Closed microwave	H2O2, HNO3	97	0.0007	ICP-MS	b) No	Pb
N039	S/L		DORM-4	Closed microwave	H2O2, HNO3		0.0004	ICP-MS	b) No	Sn
N040	S/L	homogenisation by Ultra-Turrax	NIST1570a	Closed microwave	HNO3	102	0.013	ICP-MS	b) No	As
N040	S/L		NIST1570a	Closed microwave	HNO3	99	0.003	ICP-MS	b) No	Cd
N040	S/L		NIST1570a	Closed microwave	HNO3	93	0.001	CV-AFS	b) No	Hg
N040	S/L		NIST1570a	Extraction by acids and 3% H2O2, Filtration		92	0.013	LC-ICP-MS	b) No	iAs
N040	S/L		NIST1570a	Closed microwave	HNO3	100	0.01	ICP-MS	b) No	Pb
N040	S/L			Closed microwave	HCL, HNO3	94	5	ICP-MS	b) No	Sn
N041	D	Jar has been well shaken; sample has been homogenized directly in the can using plastic tools	MR 1g/l	Closed microwave	H2O2, HNO3			ETAAS	b) No	Cd
N041	D		MR 1g/l	Closed microwave	H2O2, HNO3	96.7	0.003	FIMS	b) No	Hg
N041	D		MR 1g/l	Closed microwave	H2O2, HNO3	104	0.52	ICP-MS	b) No	Pb
L042	S/L	Blended all contents together in a mixer.	Romil	Closed microwave	HNO3	130	0.07	ICPMS	a) Yes	As
L042	S/L		Romil	Closed microwave	HNO3	102	0.007	ICPMS	a) Yes	Cd
L042	S/L		Romil	Closed microwave	HNO3	108	0.007	ICPMS	a) Yes	Hg
L042	S/L		Romil	Closed microwave	HNO3	99	0.07	ICPMS	a) Yes	Pb
L042	S/L		Romil	Closed microwave	HCL, HNO3	109	10	ICP-AES	a) Yes	Sn

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
N043	S/L	We analysed the drained product and liquid separately, reporting a composite value. This proficiency was a conundrum for us. Regulations did not specifically state to drain off liquid.COMMISSION REGULATION (EC) No 333/2007, PART B, SAMPLING METHODS, B.2. SAMPLING PLANS, Table 4 has the comment "The maximum levels for inorganic tin apply to the contents of each can" so we have reported composite.*	NIST1548a, CE278K	Closed microwave	HCL, HNO3	99	0.001	ICP-MS	a) Yes	As
N043	S/L		NIST1548a, CE278K	Closed microwave	HCL, HNO3	97	0.0005	ICP-MS	a) Yes	Cd
N043	S/L		NIST1548a, CE278K	Closed microwave	HCL, HNO3	93	0.0005	ICP-MS	a) Yes	Hg
N043	S/L		IMEP-107, NMIJ7503a	Solubilisation in concentrated HCl, reduced and extracted into CCl4, back extracted into dilute HCl		77	0.005	ICP-MS	a) Yes	iAs
N043	S/L		NIST1548a, CE278K	Closed microwave	HCL, HNO3	98	0.005	ICP-MS	a) Yes	Pb
N043	S/L		NIST1548a, CE278K	Closed microwave	HCL, HNO3	101	0.01	ICP-MS	a) Yes	Sn
N044	D	We drained the product using a strainer and then homogenised and analysed the peas.	DORM-3	Closed microwave	H2O2, HNO3	86	0.0005	ICP-MS	b) No	As
N044	D		BCR-191	Closed microwave	H2O2, HNO3	81	0.0003	ICP-MS	b) No	Cd
N044	D		DORM-3	Closed microwave	H2O2, HNO3	88	0.001	ICP-MS	b) No	Hg
N044	D		BCR-191	Closed microwave	H2O2, HNO3	84	0.0015	ICP-MS	b) No	Pb
N044	D		T07150QC	Closed microwave	H2O2, HCL, HNO3	101	0.03	ICP-MS	b) No	Sn
L045	D			Closed microwave	X			ICP-MS	X	As
L045	D			Closed microwave	X			ICP-MS	X	Cd
L045	D			X	X			FIMS	X	Hg
L045	D			Closed microwave	X			ICP-MS	X	Pb
L045	D			X	X			ICP-AES	X	Sn
L046	D	Remove brine by draining and smash beans by grinding mill according to BS EN 13804:2002	GBW10015/GBW10021 - GSB G 62022-90	Open wet, Pressure bomb	HNO3	106.1	0.006	ICP-MS	b) No	As
L046	D		GBW10015/GBW10021 - GSB G 62022-90	Open wet, Pressure bomb	HNO3	95.4	0.0005	ICP-MS	b) No	Cd
L046	D		GBW10015/GBW10021 - GSB G 62022-90	X	X	106.7	0.001	DMA	b) No	Hg
L046	D		GBW10015/GBW10021 - GSB G 62022-90	Open wet, Pressure bomb	HNO3	101	0.005	ICP-MS	b) No	Pb
L046	D		GBW10015/GBW10021 - GSB G 62022-90	Open wet, Pressure bomb	HCL, HNO3	94.7	0.07	ICP-MS	b) No	Sn
L047	S/L		BCR 189	Pressure bomb	HNO3			ETAAS	a) Yes	Cd
L047	S/L		BCR 189	Pressure bomb	HNO3			ETAAS	a) Yes	Pb
N048	S/L	We homogenized the whole content of the can.		Closed microwave	H2O2, HNO3	95	0.00069	ICP-MS	b) No	As
N048	S/L			Closed microwave	H2O2, HNO3	94	0.00048	ICP-MS	b) No	Cd

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
N048	S/L			X	X	98	0.00002	DMA	b) No	Hg
N048	S/L			Closed microwave	H2O2, HNO3	90	0.0004	ICP-MS	b) No	Pb
N048	S/L			Closed microwave	H2O2, HNO3	89	0.006	ICP-MS	b) No	Sn
L049	D	separation of the peas from the liquid by decantation, weight of the peas and total weigh by difference:the ratio peas on total is 0.61425, Sn is analysed on the whole product (solid and liquiud)	yes	Dry ashing	HCL, HNO3	85	0.05	H-AAS	b) No	As
L049	D		yes	Closed microwave	H2O2, HNO3	110	0.002	ICP-AES	b) No	Cd
L049	D		yes	Closed microwave	H2O2, HNO3	85	0.006	CV-AAS	b) No	Hg
L049	D		yesyes	Closed microwave	H2O2, HNO3	120	0.04	ICP-AES	b) No	Pb
L049	S/L		yes	Dry ashing	H2SO4, HNO3	90	10	AAS	b) No	Sn
L050	D		DORM-3 - 1000 mg/L	Closed microwave	H2O2, HNO3	85-115	0.0003	ICP-MS	X	Cd
L050	D		DORM-3 - 1000 mg/L	X	X	80-120	0.002	DMA	X	Hg
L050	D		DORM-3 - 1000 mg/L	Closed microwave	H2O2, HNO3	85-115	0.005	ICP-MS	X	Pb
L050	D		1000 mg/L	Closed microwave	H2O2, HCL, HNO3	90-110	0.3	ICP-MS	X	Sn
L051	S/L	We homogenised total content of jar and take sub-samples for analysis.		Closed microwave	H2O2, HNO3	106.5	0.003	ICP-MS	b) No	As
L051	S/L		ERM-BC084a,T07150QC	Closed microwave	H2O2, HNO3	109.3	0.003	ICP-MS	b) No	Cd
L051	S/L			Closed microwave	H2O2, HNO3	78.3	0.006	ICP-MS	b) No	Hg
L051	S/L		ERM-BC084a,T07150QC	Closed microwave	H2O2, HNO3	97.4	0.011	ICP-MS	b) No	Pb
L052	D		Yes	Closed microwave	H2O2, HNO3	103	2.5	ICP-AES	a) Yes	As
L052	D		Yes	Closed microwave	H2O2, HNO3	104	0.05	ICP-AES	a) Yes	Cd
L052	D		Yes	Closed microwave	H2O2, HNO3	109	1	ICP-AES	a) Yes	Hg
L052	D		Yes	By calculation, convert Arsenic to Arsenic (III) oxide		103	3.3	ICP-AES	a) Yes	iAs
L052	D		Yes	Closed microwave	H2O2, HNO3	105	0.5	ICP-AES	a) Yes	Pb
L052	D		Yes	Closed microwave	H2O2, HNO3	108	75	ICP-AES	a) Yes	Sn
L053	S/L	The whole content of the jar was homogenated in a Grindomix GM200 (RÉTSCH). Sample weight for digestion and extraction 1 g.	BVL-LVU 2012 Rote Bete	Closed microwave	H2O2, HNO3	100	0,005	HG-AAS	a) Yes	As
L053	S/L		BVL-LVU 2012 Rote Bete	Closed microwave	H2O2, HNO3	100	0,001	ICP-AES	a) Yes	Cd
L053	S/L		BVL-LVU 2012 Rote Bete	Closed microwave	H2O2, HNO3	100	0,0005	CV-AAS	a) Yes	Hg
L053	S/L		NRL-LVU 2011 iAs in rice	Extraction with 0,28 m HNO3, Filtration, Hydride Generation Atomic Absorption Spectroscopy		100	0,003		a) Yes	iAs

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L053	S/L		BVL-LVU 2012 Rote Bete	Closed microwave	H2O2, HNO3	100	0,008	ETAAS	a) Yes	Pb
L053	S/L		LGC BV6221/2013	Closed microwave	H2O2, HNO3	100	25	ICP-AES	a) Yes	Sn
L054	S/L	We treated separately peas and brine, analysed each and the weight ratio was reflected in the final result	As CRM SMI - As Ultra Sc. Anal. Solut.	Dry ashing	H2O2, HNO3	110	0.006	FIAS-AAS	a) Yes	As
L054	S/L		Cd CRM SMI - Cd Ultra Sc. Anal. Solut.	Dry ashing	H2O2, HNO3	106	0.003	ETAAS	a) Yes	Cd
L054	S/L		Hg CRM SMI - Hg Ultra Sc. Anal. Solut.	X	X	110	0.0005	DMA	a) Yes	Hg
L054	S/L		Pb CRM SMI - Pb Ultra Sc. Anal. Solut.	Dry ashing	H2O2, HNO3	105	0.012	ETAAS	a) Yes	Pb
L055	D	Sample was sieved (in order to separate the covering liquid) following homogenization by a grinder.	Several (ERM, BCR, IRMM..	Closed microwave	HNO3	102.4	0.0017	ICP-MS	b) No	As
L055	D		Several (ERM, BCR, IRMM..	Closed microwave	HNO3	96.9	0.0017	ICP-MS	b) No	Cd
L055	D		Several (ERM, BCR, IRMM..	X	Other	98.2	0.0017	DMA	b) No	Hg
L055	D		ERM-BC211	0.25-g of the test material were weighed in Quartz vessels and then extracted by adding 10 mL of 0.2 % (w/v) HNO3 and 1 % (w/v) H2O2 solution in a microwave digestion system. A three steps extraction program was aplied 55°C (10 min), 75 °C (10 min) and 95 °C (30 min). Samples were centrifuged and the supernatant was filtered through PET filters (0.45 µm). iAs was determined by HPLC & ICP-MS.		94.5	0.0033	HPLC-ICP-MS	b) No	iAs
L055	D		Several (ERM, BCR, IRMM..	Closed microwave	HNO3	101.6	0.0017	ICP-MS	b) No	Pb
L055	D		Interlab remaining sample	Closed microwave	H2O2, HCL, HNO3	100.1	0.33	ICP-MS	b) No	Sn
L056	S/L	Homogenisation of the whole content of the can.	yes	Closed microwave	HCL, HNO3	104	0.01	ICP-MS	b) No	As
L056	S/L		yes	Closed microwave	HCL, HNO3	106	0.01	ICP-MS	b) No	Cd
L056	S/L		yes	Closed microwave	HCL, HNO3	93	0.005	CV-AAS	b) No	Hg
L056	S/L		yes	Extraction with 0.28 m HNO3 1 h on a boiling waterbath for 1 h, chromatography with an anion-exchange column (PRP-X100) and measurement of AsIII and AsV with		100	0.02	LC-ICP-MS	b) No	iAs

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
				ICP-MS.						
L056	S/L		yes	Closed microwave	HCL, HNO3	106	0.02	ICP-MS	b) No	Pb
L056	S/L		yes	Closed microwave	HCL, HNO3	103	0.1	ICP-MS	b) No	Sn
L057	S/L			Closed microwave	H2O2, HNO3		0.009	ICP-MS	b) No	As
L057	S/L			Closed microwave	H2O2, HNO3		0.001	ICP-MS	b) No	Cd
L057	S/L			Closed microwave	H2O2, HNO3		0.002	ICP-MS	b) No	Hg
L057	S/L							HG-AAS	b) No	iAs
L057	S/L			Closed microwave	H2O2, HNO3		0.001	ICP-MS	b) No	Pb
L057	S/L			Closed microwave	H2O2, HNO3			ICP-MS	b) No	Sn
L058	S/L	whole jar content has been homogenized	SRM 1570a-Spinach Leaves - Merck VI	Closed microwave	H2O2, HNO3	100	0.05	ICP-MS	b) No	As
L058	S/L		SRM 1570a-Spinach Leaves - Merck VI	Closed microwave	H2O2, HNO3	100	0.002	ICP-MS	b) No	Cd
L058	S/L		SRM 1570a-Spinach Leaves - Bernd Kraft AAS-Standard	Closed microwave	H2O2, HNO3	100	0.001	CV-AAS	b) No	Hg
L058	S/L		NMIJ CRM 7503-a White Ric - Bernd Kraft ICP-Standard	Extraktion with 0,2% acetic acid in 3% H2O2-solution at 95°C during 90 min, centrifugation at 8000 rpm, membrane filtration on 0,45 µm		100	0.04	LC-ICP-MS	b) No	iAs
L058	S/L		SRM 1570a-Spinach Leaves - Merck VI	Closed microwave	H2O2, HNO3	100	0.001	ICP-MS	b) No	Pb
L058	S/L		SRM1548a Typical Diet - Bernd Kraft ICP-Standard	Closed microwave	H2O2, HCL, HNO3	100	2	ICP-MS	b) No	Sn
L059	D	Remove the sauce by draining	GBW10020 ,GBW 10014	Closed microwave	HCL, HNO3	96	0.0002	ICP-MS	a) Yes	As
L059	D		GBW10020 ,GBW 10014	Closed microwave	HCL, HNO3	105	0.0001	ICP-MS	a) Yes	Cd
L059	D		GBW10020	Closed microwave	HCL, HNO3	105	0.0005	AFS	a) Yes	Hg
L059	D		NIST 1568b	The extraction conditions we chose was 1% nitric acid, heat-assistant extraction 3h, 90℃. Then the components were separated by an anion exchange column and detected by LC-ICP-MS.		84	0.01	LC-ICP-MS	a) Yes	iAs
L059	D		GBW10020 ,GBW 10014	Closed microwave	HCL, HNO3	113	0.00005	ICP-MS	a) Yes	Pb

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L059	D		GBW10020	Closed microwave	HCL, HNO3	103	0.0006	ICP-MS	a) Yes	Sn
L060	S/L	mixture of whole material in B400	CRM278 - Elementstandard	Closed microwave	HCL, HNO3	103	0.09	G-AAS	a) Yes	As
L060	S/L		NIST1640e - Elementstandard	Closed microwave	HCL, HNO3	98.1	0.008	G-AAS	a) Yes	Cd
L060	S/L		CRM278 - Elementstandard	Closed microwave	HCL, HNO3	95.3	0.0034	KD-AAS	a) Yes	Hg
L060	S/L		inhouse-Material - Elementstandard	ASU §64 LFGB L 15.06-2 / 2013		113	0.031	H-AAS	a) Yes	iAs
L060	S/L		CRM278 - Elementstandard	Closed microwave	HCL, HNO3	99.4	0.046	G-AAS	a) Yes	Pb
L060	S/L		LVU-Material (Bohne) - Elementstandard	Closed microwave	HCL, HNO3	102	1.2	ICP-OES	a) Yes	Sn
L061	S/L		0.38	Closed microwave	H2O2, HNO3	96.8	0.004	ICP-MS	a) Yes	As
L061	S/L		0.110	Closed microwave	H2O2, HNO3	100	0.002	AAS	a) Yes	Cd
L061	S/L		0.04	Closed microwave	H2O2, HNO3	99	0.001	CV-AAS	a) Yes	Hg
L061	S/L		0.38	AAS-Hydride Technique		98	0.01	AAS	a) Yes	iAs
L061	S/L		0.24	Closed microwave	H2O2, HNO3	102	0.01	ICP-MS	a) Yes	Pb
L061	S/L		5.0	Closed microwave	H2O2, HNO3	99.7	0.01	ICP-AES	a) Yes	Sn
L062	S/L	Mixing the whole content of the jar with hand blender	NCS ZC 73012	Closed microwave	H2O2, HNO3	94.4	0.000046	ICP-MS	X	As
L062	S/L		NCS ZC73012	Closed microwave	H2O2, HNO3	115.9	0.003	ICP-MS	X	Cd
L062	S/L		NIST 1515	Closed microwave	H2O2, HNO3	100.6		ICP-MS	X	Hg
L062	S/L		NCS ZC73012	Closed microwave	H2O2, HNO3	105.6	0.018	ICP-MS	X	Pb
L062	S/L			Closed microwave	H2O2, HNO3	99.2		ICP-MS	X	Sn
L064	D	Drained peas from brine; Macerated pea portion.	TORT-3	Open wet	HCL, HNO3	104	0.05	FAAS-MHS	b) No	As
L064	D			Dry ashing	HNO3	114	0.01	ICP-AES	b) No	Cd
L064	D			Dry ashing	HNO3	106	0.01	ICP-AES	b) No	Pb
L064	D			Open wet	HCL, HNO3	83	20	FAAS	b) No	Sn
L065	S/L	sample preparation: total sample (peas in brine) was homogenized, and the total homogenate was analyzed.		Closed microwave	HNO3			AAS	X	As
L065	S/L			Closed microwave	HNO3			AAS	X	Cd
L065	S/L			Closed microwave	HNO3			FIMS	X	Hg
L065	S/L							H-AAS	X	iAs

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L065	S/L			Closed microwave	HNO3			AAS	X	Pb
L065	S/L			Dry ashing	HNO3			FAAS	X	Sn
L066	D	Remove the brine ,by draning. The sample(canned peas) have homogenised by IKA.	GBW10021 GBW10020 - GBW08611	Pressure bomb	HNO3	94	0.005	ICP-MS	b) No	As
L066	D		GBW10020 GBW10021 - GBW08612	Pressure bomb	HNO3	96	0.001	ICP-MS	b) No	Cd
L066	D		GBW10020 GBW10021 - GBW08617	Pressure bomb	HNO3	103	0.001	ICP-MS	b) No	Hg
L066	D		GBW10020 GBW10021 - GBW08619	Pressure bomb	HNO3	102	0.01	ICP-MS	b) No	Pb
L066	D		GBW10021 GBW10020 - GSB 04-1753-2004	Pressure bomb	HCL, HNO3	98	0.02	ICP-MS	b) No	Sn
L067	D	The total sample was drained and homogenised before sampling to three separate determinations.	no	Closed microwave	H2O2, HNO3		0.05	ICP-MS	X	As
L067	D		no	Closed microwave	H2O2, HNO3		0.01	ICP-MS	X	Cd
L067	D		no	Open wet	H2O2, HCL, HNO3		0.02	CV-AFS	X	Hg
L067	D		no	Closed microwave	H2O2, HNO3		0.02	ICP-MS	X	Pb
L067	D		no	Open wet	HCL		10	ICP-MS	X	Sn
L068	S/L	Alimenti di origine vegetale e marina		Closed microwave	H2O2, HCL, HNO3	103	0.05	HGA-AA	b) No	As
L068	S/L			Closed microwave	H2O2, HNO3	106	0.005	HGA-AA	b) No	Cd
L068	S/L			Closed microwave	H2O2, HNO3	98	0.01	HG-AAS	b) No	Hg
L068	S/L			Mineralizzazione a microonde Analisi con Idruri- ICP		100	0.05	Hydride-ICP	b) No	iAs
L068	S/L			Closed microwave	H2O2, HNO3	91	0.02	HGA-AA	b) No	Pb
L068	S/L			Closed microwave	H2O2, HCL, HNO3	101	0.1	HGA-AA	b) No	Sn
L069	D	Sample was drained in a plastic seive which had been acid soaked, rinsed and dried. The drained portion (peas) was homogenised and this portion was analysed.	TNRL03	Open wet	HNO3	89.8	0.02	ICP-MS	b) No	As
L069	D		TNRL03	Open wet	HNO3	91.7	0.02	ICP-MS	b) No	Cd
L069	D		TRNL03	Open wet	HNO3	87.7	0.02	ICP-MS	b) No	Hg
L069	D		TRNL03	Open wet	HNO3	85.6	0.02	ICP-MS	b) No	Pb
L069	D		FAPAS0747	Open wet	HCL, HNO3	91.3	0.11	ICP-MS	b) No	Sn
L070	D	As described in UNE EN-13804, sample was drained to separate the liquid.	TORT-2 - Certipur refmat trac-NIST	Closed microwave	H2O2, HNO3	103	0.005	ICP-MS	b) No	As
L070	D		TORT-2 - CCertipur	Closed microwave	H2O2, HNO3	100	0.002	ICP-MS	b) No	Cd

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
			refmat trac-NIS							
L070	D		TORT-2 - Certipur refmat trac-NIST	X	Other	101	0.003	DMA	b) No	Hg
L070	D		ERM-BC211 - Certipur refmat trac-NIST	2 different methods has been used, with same result. First; extracction with CHCl3 and HCl 0,1 N, and final measure by ICP-MS. Second method it is a microwave extraction with CL-ICP-MS		95	0.005	ICP-MS	b) No	iAs
L070	D		TORT-2 - Certipur refmat trac-NIST	Closed microwave	H2O2, HNO3	102	0.005	ICP-MS	b) No	Pb
L070	D		Certipur refmat trac-NIST	Closed microwave	H2O2, HCL, HNO3	102	0.2	ICP-MS	b) No	Sn
L071	S/L	Solid and liquid mix ,0.5g~1.0g + 6mL HNO3,microwave digestion,dil to 20ml, GFAAS test	0.049±0.004 - 0.045	Closed microwave	HNO3	99.74	0.01	ETAAS	a) Yes	As
L071	S/L		1.61±0.07 - 1.64	Closed microwave	HNO3	96.71	0.001	ETAAS	a) Yes	Cd
L071	S/L		0.42±0.07 - 0.38	Closed microwave	HNO3	98.49	0.01	ETAAS	a) Yes	Pb
L072	S/L	Mixed thoroughly to ensure homogeneity of sample.		Dry ashing, Open wet	HCL, HNO3	98.8	0.0005	AFS	a) Yes	As
L072	S/L			Dry ashing, Open wet	HCL, HNO3	102.6	0.05	ICP-AES	a) Yes	Cd
L072	S/L			Dry ashing, Open wet	HCL, HNO3	100.6	0.01	CV-AAS	a) Yes	Hg
L072	S/L			Dry ashing	HCL, HNO3	100.8	0.1	ICP-AES	a) Yes	Pb
L072	S/L			Dry ashing, Open wet	HCL, HNO3	95.5	0.5	ICP-AES	a) Yes	Sn
L073	S/L	homogenisation and microwave digestion with HNO3/H2O2/HCl	NBS 1569 A - NBS 1569 A	Closed microwave	H2O2, HNO3	95	0.01	HG-AAS	b) No	As
L073	S/L		NIST 1515 - NIST 1515	Closed microwave	H2O2, HNO3	90	0.004	ETAAS	b) No	Cd
L073	S/L		Dorm_3 - Dorm_3	Closed microwave	H2O2, HNO3	100	0.003	CV-AAS	b) No	Hg
L073	S/L		NBS 1569 A - NBS 1569 A	DIN EN 16278 (Solid Phase Extraction after Digestion)		100	0.01	HG-AAS	b) No	iAs
L073	S/L		NIST 1515 - NIST 1515	Closed microwave	H2O2, HNO3	90	0.02	ETAAS	b) No	Pb
L073	S/L		TM 15.2 - TM 15.2	Closed microwave	H2O2, HCL, HNO3	100	0.25	ICP-MS	b) No	Sn
L074	D	Upon arrival, the sample is coded and a number is assigned. Then, it is prepared and packaged in neutral jar. Thus, the sample is anonymous during its passage in analysis.	Flour of fish	Closed microwave	HNO3			AAS	b) No	As
L074	D		Flour of fish	Closed microwave	HNO3			AAS	b) No	Cd
L074	D		Flour of fish	Closed microwave	HNO3			AAS	b) No	Hg
L074	D		Flour of fish	Closed microwave	HNO3			AAS	b) No	Pb

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L075	S/L	the whole content of the can was homogenized	Nist 1548a - Standard Merck	Closed microwave	H2O2, HNO3	100	0.0003	ICP-MS	b) No	As
L075	S/L		Nist01548a - Standard Merck	Closed microwave	H2O2, HNO3	106	0.0017	ICP-MS	b) No	Cd
L075	S/L		NIST 1575 - NIST 1547	X	Other	89	0.0002	DMA	b) No	Hg
L075	S/L		BVL LVU Reis 2011 - Standard Spex Certiprep	Extraction with H2O2+acetic acid, 95 degrees, 120 min		124	0.01	LC-ICP-MS	b) No	iAs
L075	S/L		BVL LVU Grünkohl 2012 - Standard Merck	Closed microwave	H2O2, HNO3	100	0.0037	ICP-MS	b) No	Pb
L075	S/L		Nist 1548a - Standard Merck	Closed microwave	H2O2, HNO3	90	0.004	ICP-MS	b) No	Sn
L076	S/L			Dry ashing	HNO3	100	0.005	HG-AAS	b) No	As
L076	S/L			Dry ashing	H2SO4	100	0.005	AAS	b) No	Cd
L076	S/L			Closed microwave	HCL, HNO3	100	0.003	CV-AAS	b) No	Hg
L076	S/L			Dry ashing	H2SO4	100	0.02	AAS	b) No	Pb
L076	S/L			Dry ashing	H2SO4	100	1	AAS	b) No	Sn
L077	D	we have sieved the sample and minced mechanical		Closed microwave	H2O2, HNO3			ICP-MS	a) Yes	As
L077	D			Closed microwave	H2O2, HNO3			ICP-MS	a) Yes	Cd
L077	D			Closed microwave	H2O2, HNO3			FIMS	a) Yes	Hg
L077	D			Closed microwave	H2O2, HNO3			ICP-MS	a) Yes	Pb
L077	D			Closed microwave	H2O2, HNO3			ICP-AES	a) Yes	Sn
L078	D			Closed microwave	HNO3			ICP-MS	X	As
L078	D			Closed microwave	HNO3			ICP-MS	X	Cd
L078	D			Closed microwave	HNO3			ICP-MS	X	Hg
L078	D			Closed microwave	HNO3			ICP-MS	X	Pb
L078	D			Closed microwave	HNO3			ICP-MS	X	Sn
L079	D	peas and brine analysed seperately, values provided are from peas analysis (fresh weight!); brine see point 16	NRC-CNRC Tort-2 - SRM1643e	Closed microwave	HNO3	94	0.04	ICP-MS	b) No	As
L079	D		ERM-BC 084a - SRM1643e	Closed microwave	HNO3	99	0.01	ICP-MS	b) No	Cd
L079	D		NRC-CNRC Tort-2 - AAS-Standard	Closed microwave	HNO3	102	0.01	ICP-MS	b) No	Hg
L079	D		ERM-BC 084a - SRM1643e	Closed microwave	HNO3	98	0.01	ICP-MS	b) No	Pb

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L079	D		ERM-BC 084a - CRM TMRAIN-04	Closed microwave	HCL, HNO3	104	13	ICP-MS	b) No	Sn
L080	D		NIST 1568b	Closed microwave	HNO3	20	0.002	ICP-MS	a) Yes	As
L080	D		NIST 1568b	Closed microwave	HNO3	20	0.002	ICP-MS	a) Yes	Cd
L080	D		NIST 1568b	Closed microwave	HNO3	20	0.002	ICP-MS	a) Yes	Pb
L081	D							ICP-AES		As
L081	D							ICP-AES		Hg
L081	D							ICP-AES		Pb
L081	D							ICP-AES		Sn
L082	S/L	homogenisation		Closed microwave	H2O2, HNO3	92	0.02	AAS	b) No	As
L082	S/L			Closed microwave	H2O2, HNO3	98	0.005	AAS	b) No	Cd
L082	S/L			Closed microwave	H2O2, HNO3	87	0.01	CV-AAS	b) No	Hg
L082	S/L			extraction with 6 M HCl by wet digestion				AAS	b) No	iAs
L082	S/L			Closed microwave	H2O2, HNO3	115	0.05	AAS	b) No	Pb
L082	S/L			Closed microwave	HCL, HNO3	91	0.1	AAS	b) No	Sn
L083	D	Drained the liquid from the peas.	SPS-SW1 / TM-23.4	Closed microwave	H2O2, HNO3			ICP-MS	b) No	As
L083	D		SPS-SW1 / TM-23.4	Closed microwave	H2O2, HNO3			ICP-MS	b) No	Cd
L083	D			Closed microwave	H2O2, HNO3			CV-AAS	b) No	Hg
L083	D		SPS-SW1 / TM-23.4	Closed microwave	H2O2, HNO3			ICP-MS	b) No	Pb
L083	D		TM-23.4	Closed microwave	HCL, HNO3			ICP-MS	b) No	Sn
L084	D	The jar content was drained and the vegetable was homogenised by mixer	Schema 2203	Closed microwave	H2O2, HNO3	98-102	0.03	ETAAS	b) No	As
L084	D		fapas 7188	Closed microwave	H2O2, HNO3	98-102	0.01	ETAAS	b) No	Cd
L084	D		fapas 7188	Closed microwave	H2O2, HNO3	98-102	0.016	ETAAS	b) No	Pb
L084	D		fapas 7188	Closed microwave	H2O2, HCL, HNO3	98-102	5	ETAAS	b) No	Sn
L085	S/L	homogenisation of the whole content of the can	Yes	Closed microwave	HNO3	98	0.01	SFICP-MS	b) No	As
L085	S/L		Yes	Closed microwave	HNO3	96	0.001	SFICP-MS	b) No	Cd
L085	S/L		Yes	Dry ashing	Other	95	0.002	DMA	b) No	Hg
L085	S/L		Yes			95	0.002	IC-ICP-MS	b) No	iAs

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L085	S/L		Yes	Closed microwave	HNO3	87	0.004	SFICP-MS	b) No	Pb
L085	S/L		Yes	Closed microwave	HCL, HNO3	93	0.05	SFICP-MS	b) No	Sn
L086	D							ICPMS		As
L086	D							ICPMS		Cd
L086	D							DMA-80		Hg
L086	D							ICPMS		Pb
L086	D							ICP AES		Sn
L087	D	Sample tested for drained weight using documented sieving procedure - Drained Weight 62.25%		Open microwave	HCL, HNO3	119.6		ICP-AES	b) No	Cd
L087	D			Digestion on digiblock - use of AFS.				AFS	b) No	iAs
L087	D			Open microwave	HCL, HNO3	103.9		ICP-AES	b) No	Pb
L087	D			Open wet	HCL	100		ICP-AES	b) No	Sn
L088	S/L							ICP-AES		As
L088	S/L							ICP-AES		Cd
L088	S/L							CV-AAS		Hg
L088	S/L							ICP-AES		Pb
L088	S/L							ICP-AES		Sn
L089	D		interne	Dry ashing	HCL, Other	82	0.015	HG-AAS	b) No	As
L089	D		BCR2976 - TM15-2	Dry ashing	H2SO4	113	0.001	ETAAS	b) No	Cd
L089	D		BCRDOLT4	Open wet	H2SO4, HNO3	92	0.015	CV-AAS	b) No	Hg
L089	D		BCR2976 - TM15-2	Dry ashing	H2SO4	93	0.005	ETAAS	b) No	Pb
L089	D		TM15-2	Closed microwave	H2O2, HCL, HNO3		0.5	ICP-AES	b) No	Sn
L090	S/L							ICP-MS		As
L090	S/L							ICP-MS		Cd
L090	S/L							AFS		Hg
L090	S/L							H-AAS		iAs
L090	S/L							ICP-MS		Pb
L090	S/L							ICP-AES		Sn
N091	S/L		standard solution	Dry ashing	HCL, HNO3	80	0.001	HG-AAS	b) No	As

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
N091	S/L		standard solution	Dry ashing	HNO3	84	0.005	FAAS	b) No	Cd
N091	S/L		standard solution	Dry ashing	HNO3	101	0.05	FAAS	b) No	Pb
N091	S/L		standard solution	Dry ashing	HCL, HNO3	101	5	FAAS	b) No	Sn
L092	S/L		CRM - Merck	Closed microwave	HCL, HNO3	85	0.001	HG-AAS	a) Yes	As
L092	S/L		CRM - Merck	Closed microwave	HCL, HNO3	90	0.005	ETAAS	a) Yes	Cd
L092	S/L		CRM - Merck	Closed microwave	HCL, HNO3	85	0.001	CV-AAS	a) Yes	Hg
L092	S/L		CRM - Merck	Closed microwave	HCL, HNO3	85	0.01	ETAAS	a) Yes	Pb
L092	S/L		CRM - Merck	Closed microwave	HCL, HNO3		0.05	ICP-OES	a) Yes	Sn
L093	D	Discard the liquid and then blend the solid		Closed microwave	HCL, HNO3			ICP-MS	a) Yes	As
L093	D			Closed microwave	HCL, HNO3			ICP-MS	a) Yes	Cd
L093	D			Closed microwave	HCL, HNO3			ICP-MS	a) Yes	Hg
L093	D			Closed microwave	HCL, HNO3			ICP-MS	a) Yes	Pb
L093	D			Closed microwave	HCL, HNO3			ICP-MS	a) Yes	Sn
L094	S/L	Homogenisation with Titan-cutter, complete	Proficiency Test Material - LGC certified	Closed microwave	HNO3		0.03	ICP-MS	b) No	As
L094	S/L		Proficiency Test Material - LGC certified	Closed microwave	HNO3		0.03	ICP-MS	b) No	Cd
L094	S/L		Proficiency Test Material - LGC certified	Closed microwave	HNO3		0.01	CV-AAS	b) No	Hg
L094	S/L		Proficiency Test Material - LGC certified				0.03	HG-ICP-MS	b) No	iAs
L094	S/L		Proficiency Test Material - LGC certified	Closed microwave	HNO3		0.05	ICP-MS	b) No	Pb
L094	S/L		Proficiency Test Material - LGC certified	Closed microwave	HCL, HNO3		0.1	ICP-MS	b) No	Sn
L095	D							AAS		Cd
L095	D							AAS		Hg
L095	D							AAS		Pb
L096	D	I've separated the liquid from the peas and I've homogenized the whole	BCR N° 279 - Ultrascientific As 1 g/L	Closed microwave	HNO3	103.5	0.01	ICP-MS	b) No	As
L096	D		NRC-MC GBW10016 - Ultrascientific Cd 1g/L	Closed microwave	HNO3	100	0.01	AAS	b) No	Cd
L096	D		NRC-CNRC DORM-4 - Ultrascientific Hg 1 g/L	Open wet	Other	95.6	0.05	DMA	b) No	Hg

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L096	D		NRC-MC GBW10016 - PE Lead (II oxide) 1 g/L	Closed microwave	HNO3	93.3	0.02	AAS	b) No	Pb
L096	D		NIST SRM-1548 - Ultrascientific Sn 1 g/L	Closed microwave	HNO3	79.6	0.08	ICP-MS	b) No	Sn
L098	S/L	The sample was blended and prepare for analyse with H2O2 and HNO3.	merck	Closed microwave	H2O2, HNO3	98	0.015	ICP-OES	a) Yes	As
L098	S/L		merck	Closed microwave	H2O2, HNO3	101	0.003	ICP-OES	a) Yes	Cd
L098	S/L		merck	Closed microwave	H2O2, HNO3	109	0.003	ICP-OES	a) Yes	Hg
L098	S/L		merck	Closed microwave	H2O2, HNO3	94	0.019	ICP-OES	a) Yes	Pb
L098	S/L		merck	Closed microwave	H2O2, HNO3	101	0.15	ICP-OES	a) Yes	Sn
L099	D	It was homogenized.	SRM1568a	Dry ashing	HCL, Other		0.005	HG-AAS	a) Yes	As
L099	D		SRM1568a	Closed microwave	H2O2, HNO3		0.001	ETAAS	a) Yes	Cd
L099	D		IAEA-V-10	Dry ashing	X		0.0005	AAS	a) Yes	Hg
L099	D		BCR191	Closed microwave	H2O2, HNO3		0.005	ETAAS	a) Yes	Pb
L099	D		FapasTM07188	Closed microwave	H2O2, HCL, HNO3		5	FAAS	a) Yes	Sn
L100	S/L							ICP-AES		As
L100	S/L			Closed microwave	H2O2, HNO3			ICP-AES		Cd
L100	S/L							DMA		Hg
L100	S/L							ICP-AES		iAs
L100	S/L							ICP-AES		Pb
L100	S/L							ICP-AES		Sn
L101	D	liquid was drained		Closed microwave	HCL, HNO3	95-124	0.5	ICP-AES	b) No	As
L101	D			Closed microwave	HCL, HNO3	100-122	0.05	ICP-AES	b) No	Cd
L101	D			Closed microwave	HCL, HNO3	95-114	0.2	ICP-AES	b) No	Hg
L101	D			Closed microwave	HCL, HNO3	95-100	0.2	ICP-AES	b) No	Pb
L101	D			Closed microwave	HCL, HNO3	88-99	1	ICP-AES	b) No	Sn
L102	D	Separated the solid from liquid part. The solid part was homogenized and anlyzed	No one - Yes	Closed microwave	H2O2, HNO3	100	0.0071	ICP-MS	b) No	As
L102	D		No one - Yes	Closed microwave	H2O2, HNO3	100	0.0011	ICP-MS	b) No	Cd
L102	D		No one - Yes	Closed microwave	H2O2, HNO3	100	0.0011	ICP-MS	b) No	Hg
L102	D		No one - Yes	Closed microwave	H2O2, HNO3	100	0.003	ICP-MS	b) No	Pb

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L102	D		No one - Yes	Closed microwave	H2O2, HNO3	100	0.01	ICP-MS	b) No	Sn
L103	S/L							CV-AAS		As
L103	S/L							AAS		Cd
L103	S/L							CV-AAS		Hg
L103	S/L							AAS		Pb
L103	S/L							FAAS		Sn
L104	S/L		Merck 1.70303.0100	Closed microwave	H2O2, HNO3		0.000133	ICP-MS	a) Yes	As
L104	S/L		Merck 1.70309.0100	Closed microwave	H2O2, HNO3		0.000133	ICP-MS	a) Yes	Cd
L104	S/L		Merck 1.70333.0100	Closed microwave	H2O2, HNO3		0.0001	FIMS	a) Yes	Hg
L104	S/L		Merck 1.70328.0100	Closed microwave	H2O2, HNO3		0.00007	ICP-MS	a) Yes	Pb
L104	S/L		Merck 1.70362.0100	Closed microwave	H2O2, HCL, HNO3		0.00027	ICP-MS	a) Yes	Sn
L105	D		panreac313171	Dry ashing	HCL, Other	98.9	0.06	HG-AAS	b) No	As
L105	D		FAPAST07170QC - Panreac313186	Closed microwave	H2O2, HNO3	95.3	0.05	CV-AAS	b) No	Hg
L105	D		FAPAST07170QC - ScharlauES0061	Closed microwave	H2O2, HNO3	100.8	10	FAAS	b) No	Sn
N106	D	Drain peas from brine, homogenise peas with Buchi Mixer B-400	FAPAS-Rice test material - Standard	Closed microwave	HNO3	100	0.03	ETAAS	b) No	As
N106	D		BCR-191 - BCR-610	Closed microwave	HNO3	100	0.003	ETAAS	b) No	Cd
N106	D		FAPAS-Rice test material - Standard solution	Protocol from IMEP-41		100	0.008	HG-AAS	b) No	iAs
N106	D		BCR-191 - BCR-713	Closed microwave	HNO3	100	0.008	ETAAS	b) No	Pb
N106	D		Spike - FAPAS-Water test material	Closed microwave	HNO3	100	0.2	ETAAS	b) No	Sn
L107	D	we reported the results of only peas:we analyse also liquid (if you want we could send the results)		Open wet	HNO3			ICP-MS	a) Yes	As
L107	D			Open wet	HNO3	>80	0.003	ICP-MS	a) Yes	Cd
L107	D			Open wet	HNO3	>80	0.003	ICP-MS	a) Yes	Hg
L107	D					>80	0.003	ICP-MS	a) Yes	iAs
L107	D			Open wet	HNO3	>80	0.003	ICP-MS	a) Yes	Pb
L107	D			Open wet	HNO3	>80	0.003	ICP-MS	a) Yes	Sn
L108	S/L	we mixed and crushed the complete content		Closed microwave	HCL, HNO3		0.003		a) Yes	As

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L108	S/L	of the jar		Closed microwave	HCL, HNO3		0.0007		a) Yes	Cd
L108	S/L			Closed microwave	HCL, HNO3				a) Yes	Hg
L108	S/L			Closed microwave	HCL, HNO3		0.003		a) Yes	Pb
L108	S/L			Closed microwave	HCL, HNO3		0.003		a) Yes	Sn
L109	D	The content of the sample was drained in a sieve, homogenized in a blender and an aliquot taken for analysis		Dry ashing	HNO3	80-113	0.004	ETAAS	b) No	Cd
L109	D			X	X	81-113	0.02	thermal decomposition, gold amalgamation, and atomic absorption spectroscopy	b) No	Hg
L109	D			Dry ashing	HNO3	82-117	0.03	ETAAS	b) No	Pb
L110	S/L	crushing, mineralisation,analysis	spiking	Pressure bomb	HNO3		0.002	ICP-MS	b) No	As
L110	S/L		spiking	Pressure bomb	HNO3		0.002	ICP-MS	b) No	Cd
L110	S/L		spiking	Pressure bomb	HNO3		0.002	ICP-MS	b) No	Hg
L110	S/L		spiking	Pressure bomb	HNO3		0.002	ICP-MS	b) No	Pb
L110	S/L			Pressure bomb	HNO3		0.02	ICP-MS	b) No	Sn
L111	S/L		Reference Material - Certified Ref. Material	Closed microwave	HNO3	-	0.007	AAS	b) No	Cd
L111	S/L		Reference Material - Certified Ref. Material	X	Other	-	0.06	DMA	b) No	Hg
L112	D	The paes were drained before weighed for mineralization	LGC 7162 / NCS-ZC73013	Closed microwave	HNO3		0.007	ICP-MS	b) No	As
L112	D		LGC 7162 / NCS-ZC73013	Closed microwave	HNO3		0.007	ICP-MS	b) No	Cd
L112	D		LGC 7162 / NCS-ZC73013	Closed microwave	HNO3		0.007	ICP-MS	b) No	Hg
L112	D		LGC 7162 / NCS-ZC73013	Closed microwave	HNO3		0.007	ICP-MS	b) No	Pb
L112	D			Closed microwave	HNO3		0.007	ICP-MS	b) No	Sn
L113	S/L	Grinding of all of the sample (pea + juice)	DORM	Closed microwave	H2O2, HNO3	90-110	0.002	ICP-MS	b) No	As
L113	S/L		DORM	Closed microwave	H2O2, HNO3	90-110	0.002	ICP-MS	b) No	Cd
L113	S/L		DORM	Closed microwave	H2O2, HNO3	90-110	0.003	ICP-MS	b) No	Hg
L113	S/L		DORM	Closed microwave	H2O2, HNO3	90-110	0.002	ICP-MS	b) No	Pb
L113	S/L		DORM	Closed microwave	H2O2, HNO3	90-110	0.005	ICP-MS	b) No	Sn

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L114	D	Peas drained from the brine and mixed in laboratory homogeniser. Weight of peas and liquid recorded.		Dry ashing	HCL, HNO3	112	0.01	ICP-AES	a) Yes	As
L114	D			Dry ashing	HCL, HNO3	74.4	0.01	ICP-AES	a) Yes	Cd
L114	D			Dry ashing	H2O2, HNO3	100.7	0.01	FAAS-MHS	a) Yes	Hg
L114	D			Dry ashing	HCL, HNO3	91.6	0.01	ICP-AES	a) Yes	Pb
L114	D			Open wet	H2O2, HNO3	-	0.1	ICP-AES	a) Yes	Sn
L115	S/L		LGC7162 - perkin elmer	Closed microwave	H2O2, HNO3	117.8	0.009	ETAAS	b) No	As
L115	S/L		LGC7162 - perkin elmer	Closed microwave	H2O2, HNO3	105.3	0.0005	ETAAS	b) No	Cd
L115	S/L		LGC7162 - perkin elmer	Closed microwave	H2O2, HNO3	89.8	0.0182	ETAAS	b) No	Pb
L115	S/L		FAPAS T07210 - perkin el	Closed microwave	H2O2, HCL, HNO3	113	0.32	ETAAS	b) No	Sn
L116	S/L	Homogenized the entire contents of the can (peas and floods).	NCS ZC 85006 Tomato - Arsenic ICP Standard1000	Dry ashing	HCL, HNO3, Other	89.7	0.02	HG-AAS	b) No	As
L116	S/L		NCS ZC 85006 Tomato - ICP-08N-1	Closed microwave	HNO3	113	0.003	ETAAS	b) No	Cd
L116	S/L		NCS ZC 85006 Tomato - Mercury Standard Solution	Open wet	H2SO4, HNO3, Other	97.3	0.0011	CV-AAS	b) No	Hg
L116	S/L		NCS ZC 85006 Tomato - Arsenic ICP Standard 1000	Hydrolysis with HCl, extraction into chloroform, reextraction into HCl, dry mineralization the same like in total As, analysis technique Hydride generation - atomic absorption spectroscopy				HG-AAS	b) No	iAs
L116	S/L		NCS ZC 85006 Tomato - ICP-29N-1	Closed microwave	HNO3	112	0.016	ETAAS	b) No	Pb
L116	S/L		ERM-BC084a Tomato Pasta - Tin ICP Standard1000mg/l	Closed microwave	HCL, HNO3	103.9	0.15	ETAAS	b) No	Sn
L117	S/L	homogenization, mineralization	tobacco leaves - Central Office of Maeasure	Closed microwave	HNO3, Other	92.57	0.0096	HG-AAS	b) No	As
L117	S/L		tobacco leaves - Central Office of Maeasure	Dry ashing	HNO3	73.57	0.0007	ETAAS	b) No	Cd
L117	S/L		tobacco leaves - Central Office of Maeasure	Dry ashing	X	93.29	0.0002	CV-AAS	b) No	Hg
L117	S/L		tobacco leaves - Central Office of Maeasure	Dry ashing	HNO3	91.12	0.008	ETAAS	b) No	Pb
L117	S/L			Open wet	HCL	93.72	5	FAAS	b) No	Sn

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L118	D	Only solid part was analysed	PT material	Closed microwave	H2O2, HNO3	93.2	0.01	CV-AAS	b) No	As
L118	D		BCR-191	Closed microwave	H2O2, HNO3	95.7	0.001	ETAAS	b) No	Cd
L118	D		BCR-191	Closed microwave	H2O2, HNO3	99.4	0.005	CV-AAS	b) No	Hg
L118	D		BCR-191	Closed microwave	H2O2, HNO3	99.5	0.01	ETAAS	b) No	Pb
L120	D	Liquid drainedfrom jar. Peas blended.	Dorm 4	Closed microwave	H2O2, HNO3		0.02	ICP-MS	a) Yes	As
L120	D		Dorm 4	Closed microwave	H2O2, HNO3		0.006	ICP-MS	a) Yes	Cd
L120	D		Dorm 4	Closed microwave	H2O2, HNO3		0.02	ICP-MS	a) Yes	Pb
L121	D	The peas were seperated from the liquid using a sieve. The peas were grinded using a miller.	SRM	Open wet	HNO3	101	0.015	ICP-MS	a) Yes	As
L121	D			Open wet	HNO3	97	0.001	ICP-MS	a) Yes	Cd
L121	D			Open wet	HNO3	94	0.002	ICP-MS	a) Yes	Hg
L121	D			Open wet	HNO3	97	0.02	ICP-MS	a) Yes	Pb
L121	D			X	HCL, HNO3	104	0.05	ICP-MS	a) Yes	Sn
N122	S/L		std solution	Closed microwave	H2O2, HNO3	96	0.01	ICP-MS	b) No	As
N122	S/L		std solution	Closed microwave	H2O2, HNO3	98	0.002	ICP-MS	b) No	Cd
N122	S/L		std solution				0.01	HG-AAS	b) No	iAs
N122	S/L		std solution	Closed microwave	H2O2, HNO3	98	0.005	ICP-MS	b) No	Pb
N122	S/L		std solution	Closed microwave	H2O2, HCL, HNO3		0.4	ICP-MS	b) No	Sn
L123	D	Brine was removed, peas was drying and then peas was homogenizated	1 g/l	Dry ashing, Open wet	HNO3	96.4	0.004	HG-AAS	a) Yes	As
L123	D		1 g/l	Dry ashing	X	98.38	0.011	FAAS	a) Yes	Cd
L123	D		1 g/l	Open wet	H2SO4, HNO3	98.75	0.002	CV-AAS	a) Yes	Hg
L123	D		1 g/l	Dry ashing	X	98.25	0.022	FAAS	a) Yes	Pb
L123	D		1 g/l	Open wet	HCL	99	4.8	FAAS	a) Yes	Sn
L124	D							ICP-MS		Cd
L124	D							ICP-MS		Pb
L125	S/L	prepared whole in mascerator	yes	Dry ashing	HCL, HNO3	94	0.025	HG-AAS	b) No	As
L125	S/L		yes	Dry ashing	HCL	97	0.004	FAAS	b) No	Cd
L125	S/L		yes	Open wet	H2SO4, HCL, HNO3	92	0.02	HG-AAS	b) No	Hg

Lab. code	Sample preparation	Question 5.1: Treatment of the sample	CRM -Method Validation or Instrument calibration	Digestion type - iAs analysis Method	Digestion Mix	Recovery	LODs	Technique	Compliant or not	z-score
L125	S/L		yes	digest sample in HCl, ad hyrobromic acid and hydrazine sulphate. liquid/liquid extraction into chloroform.Back extyraction into HCl. Add magnesium nitrate and HNO3, ash, reduce and hydride AAS		79	0.06	HG-AAS	b) No	iAs
L125	S/L		yes	Dry ashing	HCL	86	0.037	FAAS	b) No	Pb
L125	S/L		yes	Open wet	HCL	98	1.4	FAAS	b) No	Sn
L126	S/L		CRM Dolt-4 - in house standard	Closed microwave	HNO3		0.3	ICP-MS	a) Yes	As
L126	S/L		CRM Dolt-4 - in house standard	Closed microwave	HNO3		0.03	ICP-MS	a) Yes	Cd
L126	S/L		CRM Dolt-4 - in house standard	Open wet	Other		0.05	ICP-MS	a) Yes	Hg
L126	S/L			N/A					a) Yes	iAs
L126	S/L		CRM Dolt-4 - in house standard	Closed microwave	HNO3		0.03	ICP-MS	a) Yes	Pb
L126	S/L		CRM No.27 - in house standard	Open wet	Other		0.5	ICP-MS	a) Yes	Sn
L127	D	we sampled the drained product, weighed, omogenized and digested.	DORM-3 - Absolute Standard	Closed microwave	H2O2, HNO3	111	0.03	ETAAS	b) No	Cd
L127	D		DORM-3 - Absolute Standard	Closed microwave	H2O2, HCL, HNO3	112	0.01	CV-ETA (Cold Vapour-ETA or FIAS-FURNACE)	b) No	Hg
L127	D		DORM-3 - Absolute Standard	Closed microwave	H2O2, HNO3	98	0.03	ETAAS	b) No	Pb
L127	D		FAPAS - Absolute Standard	Closed microwave	H2O2, HCL, HNO3	102	1	ICP-AES	b) No	Sn

D: drained product; S/L: solid / liquid composite.

Annex 17: Comments of the laboratories participating in IMEP-118

Lab code	Do you have any comments? Please let us know…
N001	CRM used was NIST1572 Citrus leaves
N005	More information on sample preparation (prior to acid digestion) should have been recommended such as reporting results on a wet weight or dry weight basis - in order to achieve as much consistency as possible for all participants
N010	Pea is a legume and whereas for Lead there is a clear distinction between vegetables and legumes, for Cadmium there is no category where pea would adequately fit.
N011	Detected concentration of Lead is lower than MRL, but cadmium concentration is bigger than MRL. Consequently we decided that sample is not acceptable.
N025	If available, we would like to receive further sample for future reference.
L027	we discussed the preparation of the sample: since a majority did not 'eat' the liquid, it was decanted.
L029	Not a reliable values for Sn, Sn seems to be very unstable, and is probably higher.
N030	For the analysis of Hg in this PT we could not detect any amount and the reported value is our LOD. For the analysis of Sn according to EN 13804:2002 we report the results of the drained samples.
L031	LOD for Cd is raised because of interference on Cd from high content of Sn
L042	Accepted tin as the level of uncertainty present could mean the result is actually lower than the prescribed limit.
N043	* We clarify with the customer what is to be measured if it is not regulatory work.
N044	Our instrument was a bit insensitive when the analyses were carried out. The results are not corrected for dry matter.
L049	the legislation is confused for Sn (expressed on the whole product) comparing with Cd an Pb both on the drained product
L070	Mercury should not have been set for analysis if it cannot be evaluated. No instructions for sample preparation (drained or not) have been done so, comparison can be difficult (and z-score feasibility too). Although the objective is interesting, National Accreditation Bodies have asked some laboratories to participate and evaluate them with this IMEP, that may give bizarre results due to sample preparation.
L079	Contents of above metals in brine are relatively high. Total weight of sample 175.3g, brine: 68.1g. Content of metals in brine [mg/kg]: As 0,164; Cd 0.033; Pb 0.059; Sn 69.0; Hg <0.008
L083	The drained liquid was highly contaminated, e.g. Sn. Hg, Sn, Pb, As and Cd were detectable.
L087	Website keeps crashing
L096	We couldn't indicate the technique for Sn, wich is ICP-MS
L108	According to the ml of Pb and cd a conclusion whether the sample is accepted or not cannot be taken as the sample was homogenized completely for tin. As only one jar of test item was sent the amount of water in the jar is unknown. So the manufacturing factor according to Art. 2 VO 1881/2006 cannot be considered acceptably. Furthermore the source of cadmium is not clear (peas, water or material of
L111	It is not of same matrix and have not been checked if the digestion is optimised for this matrix.
L114	The sample was difficult to homogenise
L125	jar marked IMEP-41
L126	iAS is not in place in our laboratory. This is commercial laboratory and method development is upon market demand.

European Commission
EUR 27145 **EN – Joint Research Centre – Institute for Reference Materials and Measurements**

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Author(s): I. Fiamegkos, B. de la Calle, H. Emteborg, J. Seghers, M.-F. Tumba, M. Vahcic, F. Cordeiro, A. Cizek-Stroh, P. Robouch

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