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Determination of total As, Cd, Pb, Hg and inorganic As in chocolate

EURL-HM-20 Proficiency test Report

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Executive summary

The European Union Reference Laboratory for Heavy Metals in Feed and Food (EURL-HM) organised a proficiency test (EURL-HM-20) for the determination of total As, Cd, Pb, Hg and inorganic As (iAs) in chocolate in support to Commission Regulation (EC) 1881/2006 setting maximum levels for certain contaminants in foodstuffs. This PT was open to National Reference Laboratories (NRLs), official control laboratories (OCLs) and other interested laboratories.

One hundred and six participants from 42 countries registered to the exercise. Only five participants did not report results. Thirty two NRLs (out of the 33 that registered) reported results.

The material used as test item was commercially available chocolate which, after appropriate processing, was bottled, labelled and dispatched to the participants during the first half of May 2015. Seven laboratories with demonstrated measurement capabilities in the field provided results to establish the assigned values. The standard uncertainties associated to the assigned values were calculated according to ISO Guide 35.

Laboratory results were rated using *z*- and zeta (*ζ*-) scores in accordance with ISO 13528:2005. The relative standard deviation for proficiency assessment was set to 19% for total Cd, to 22 % for total As and Pb and to 25% for iAs. The expert laboratories reported "less than" values for the total Hg mass fraction; therefore no scoring was provided for this measurand.

All NRLs (100%) and 94 % of the other laboratories performed satisfactorily for the determination of the total Cd mass fraction in chocolate demonstrating that the recently amended European Regulation (EC) No 1881/2006 setting MLs for cadmium in cocoa and chocolate can be implemented The percentage of satisfactory scores decreased to 61, 67 and 64%, (63, 77 and 78% for NRLs) due to the low mass fractions of total As, Pb and iAs in the chocolate. Several laboratories reported "less than" values. Only 27% of the participants (50% of the NRLs) reported results for iAs, half of which were "less than" values.

In all cases, the percentage of satisfactory ζ-scores was lower than the corresponding one for z-scores indicating that several laboratories should improve their estimate of measurement uncertainty.

Acknowledgements

The authors wish to thank colleagues from the IRMM for their valuable contributions they made during preparation and testing of the proficiency test matrix.

The hundred and one laboratories having participating in this exercise, listed in Annex 1, are kindly acknowledged.

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 proportionally with 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].

The European Food Safety Authority (EFSA) carried out in 2012 an in-depth evaluation of the dietary exposure to cadmium (Cd) via different food commodities, over specific groups of population [2]. Data indicated that high levels of Cd were found (among others) in cocoa-based products. According to previous EFSA opinions published in 2009 and 2011 certain population groups (such as children, vegetarians and people living in highly contaminated areas) can easily exceed by a factor of two the tolerable weekly Cd intake of 2.5 µg kg^{-1} body weight. Cocoa powder and cocoa-based products are critical food commodities, especially for children due to high consumption, their lower body weight and their higher digestive absorption of metals. Following these findings, the European Commission published an amendment to Regulation (EC) No 1881/2006 setting maximum levels (MLs) for certain contaminants in food, in order to include MLs for Cd in cocoa and chocolate [3-5]. The following limits are effective from 1 January 2019:

- 0.10 mg kg^{-1} for milk chocolate with < 30 % total dry cocoa solids;
- 0.3 mg kg^{-1} for chocolate with < 50 % total dry cocoa solids; and milk chocolate with ≥ 30 % total dry cocoa solids, and
- 0.8 mg kg^{-1} for chocolate with ≥ 50 % total dry cocoa solids.

The European Union Reference Laboratory for Heavy Metals in Feed and Food (EURL-HM) organised the proficiency test (EURL-HM-20) to assess the performance of National Reference Laboratories (NRLs) and other food control laboratories in the determination of total arsenic (As), cadmium (Cd), lead (Pb), mercury (Hg) and inorganic Arsenic (iAs) mass fractions in chocolate, as agreed with the Directorate General for Health and Food Safety (DG SANTE) in the annual work programme of the EURL-HM. This report summarises the outcome of this PT.

2. Scope and aim

As stated in Regulation (EC) No 882/2004 one of the core duties of the European Union Reference Laboratories is to organise interlaboratory comparisons for the benefit of NRLs [6].

The present proficiency test (PT) aims to assess the performance of NRLs and other interested laboratories in the determination of total As, Cd, Pb, Hg and iAs mass fractions in chocolate.

The assessment of measurement results follows the administrative and logistic procedures of the EC-JRC-IRMM for the organisation of PTs which is accredited according to ISO 17043:2010 [7].

This PT is identified as EURL-HM-20.

3. Set up of the exercise

3.1 Time frame

The organisation of the EUR-HM-20 exercise was agreed upon by the NRL network at the 8th EURL-HM Workshop held in Brussels on September 24, 2013. The exercise was announced on the JRC webpage on February 25, 2015 (Annex 2). Invitation letters were sent to NRLs as well as to the European Cooperation for Accreditation (EA), to the Asian Pacific Laboratory Accreditation Cooperation (APLAC), to Inter-American Accreditation Cooperation (IAAC) and to African Accreditation Cooperation (AFRAC) on March 4, 2015 (Annex 3-7). The registration deadline was set to April 10, 2015. The reporting deadline was set to June 12, 2015. Dispatch was monitored by the PT coordinator using the messenger's parcel tracking system on the internet.

3.2 Confidentiality

The following confidentiality statement was made to the EA, APLAC, IAAC and AFRAC: *"Confidentiality of the participants and their results towards third parties is guaranteed.*

In the case of EA and NRLs having appointed OCLs to participate in the PT an additional statement of disclosure was added (Annex 3,4): *"The organisers will disclose to you the details of the participants that have been nominates by you"*.

3.3 Distribution

Test items were dispatched to participants during the first half of May (4-13 May). Each participant received:

- One pack of six vials containing the test item (approx. 0.5 g / bottle);
- A "Test item accompanying letter" (Annex 8); and
- A "Confirmation of receipt form" to be sent back to IRMM after receipt of the test item (Annex 9).

3.4 Instructions to participants

Detailed instructions were given to participants in the "Test item accompanying letter" mentioned above. Measurands were defined as "the mass fractions of total As, Cd, Pb, Hg and iAs in chocolate".

Participants were asked to perform two or three independent measurements, to report their calculated mean (X_{lab}) and the associated expanded measurement uncertainty (U_{lab}).

Participants received an individual code to access the on-line reporting interface, to report their measurement results and to complete the related questionnaire. A dedicated questionnaire was used to gather additional information related to measurements and laboratories (Annex 9).

Participants were informed that the procedure used for the analysis should resemble as closely as possible their routine procedures for this type of matrix/analytes and concentration levels.

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

4.Test item

4.1 Preparation

Dark south-American chocolate (1kg of 64% cocoa content) was purchased in a local market. Its origin and the batch number was clearly stated on the package. Chocolate from four different batch numbers were screened for cadmium, lead and arsenic contents using an Agilent 7500 series ICP-MS (Diegem, BE) after digestion. The batches showed high content of cadmium and lower amounts of lead and arsenic. All results were within the legal limits set by the amended European Regulation (EC) 1881:2006.

In order to provide test items that could be easily removed from their containers, it was decided to supply single units of 0.5 g chocolate pellets in acid washed 10-mL vial. Six vials were placed in an aluminised sachet, resulting in a kit of six pellets. At the time of analysis, pellets were to be accurately weighed and placed directly in the proper digestion vessel. Due to the relatively low mass of the pellets, the fat content and the amount of other organic material were not expected to produce over-pressure conditions during digestion with strong mineral acids. A total of 250 sachets (1,500 pellets) were prepared for the EURL-HM-20 project.

The vials (10-mL) and rubber lyo-inserts were first acid washed for 30 minutes with nitric acid 10 % in a three dimensional mixer (Dynamix CM-200, WAB, Basel, CH) and subsequently rinsed with Type I water. Vials were then soaked in 10 % nitric acid for 24 h and rinsed three times with Type I water. Thereafter the glass vials and rubber inserts were left to dry on acid washed nylon meshes placed in a clean cell, flushed with a HEPA filtered air. The cleaned vials were then placed in plastic crates awaiting filling with the chocolate pellets.

Having contacted the chocolate industry at Barry Callebaut Services, (Lebbeke-Wieze, BE), it was decided to use polycarbonate moulds to produce the 0.5-g chocolate pellets. Each mould consisted of 88 uniform volume pits where melted chocolate would solidify into pellets. The necessary moulds were prepared by the IRMM workshop using computerised milling equipment. The pits were made conical to simplify removal of the pellets after cooling down.

For the production of pellets, IRMM processing staff assisted the industry experts. The day before production the chocolate was melted using a Hermes JKV-30 equipment (JKV, Gilze, NL) at 45 °C. The chocolate was allowed to recirculate over-night at about 5 L min^{-1} in order to thoroughly homogenize the bulk. On the day of production the temperature of chocolate was set to 32 °C (optimal temperature to work with dark chocolate) and chocolate was allowed to recirculate for another 30 min. After the moulds were filled, chocolate solidified in a fridge and was later transferred into properly labelled polyethylene bags. All parts of the machine that were in contact with the chocolate were made of AISI 304-grade stainless steel which is not expected to contaminate chocolate with cadmium, lead or arsenic.

Finally, the plastic bags were emptied on an acid washed plastic tray and each pellet was manually introduced into one vial using acid washed Teflon tweezers. All operations were performed inside a clean cell flushed with HEPA filtered air. Once the vials were filled, the rubber inserts were placed in the neck of the vial and placed in a Martin Christ Epsilon 2-100D freeze dryer (Osterode, DE). Air was then removed from the freeze drying chamber and replaced by argon. The shelves of the freeze dryer were used to press down the inserts firmly into the necks of the vials resulting in chocolate pellets sealed under oxygen free atmosphere. Subsequent capping and labelling according to fill order took place using the Bausch und Ströbel (Ilshofen, DE) and BBK (Beerfelden, DE) equipments.

4.2 Homogeneity and stability

Measurements for the homogeneity and stability studies were performed by the Centro de Salud Pública de Alicante (CSPA, Alicante, Spain). Inductively coupled plasma mass spectrometry (ICP-MS) was used, after microwave digestion (using 0.50 g of chocolate sample and 5 ml of a mixture of HNO_3/H_2O_2 1:1).

An elemental mercury analyser (EMA) was used to quantify the total Hg mass fraction, using approximately 60 mg of chocolate per analysis.

The statistical treatment of data was performed by the EURL-HM.

Homogeneity was evaluated according to ISO 13528:2005 [8]. The test item proved to be adequately homogeneous for all the investigated measurands.

The stability study was conducted applying the isochronous design [9, 10]. The test item proved to be adequately stable for all measurands at 18°C during the 6 weeks that elapsed between the dispatch of the samples and the deadline for reporting.

From previous experience (IMEP-107, IMEP-118), it was assumed that the homogeneity and stability of the total As mass fraction are representative of those of iAs.

The contribution from homogeneity (u_{bb}) and stability (u_{st}) to the standard measurement uncertainty of the assigned value (u_{ref}) was calculated using SoftCRM [11]. The analytical results reported by the expert laboratories and the statistical evaluation of the homogeneity and stability studies are presented in Table 1 and in Annex 10.

5. Assigned values and their uncertainties

5.1 Assigned value, X_{ref}

The assigned values for the five measurands (total As, Cd, Pb, Hg and iAs in chocolate) were determined by seven laboratories, all selected on the basis of on their demonstrated measurement capabilities (later referred to as expert laboratories):

- *ALS Scandinavia AB (Luleå, Sweden);*
- *SCK-CEN - Studiecentrum voor Kernenergie (Mol, Belgium);*
- *Umweltbundesamt GmbH (Vienna, Austria);*
- *CSPA - Centro de Salud Pública de Alicante (Alicante, Spain);*
- *VITO - Vlaamse Instelling voor Technologisch Onderzoek (Mol, Belgium);*
- *IRMM* – Institute for Reference Materials and Measurements (*Geel, Belgium*); and
- *Institut für Chemie, Bereich Analytische Chemie, University of Graz (Graz, Austria)*

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

- ALS Scandinavia used inductively coupled plasma sector field mass spectrometry (ICP-SFMS) after closed microwave digestion of the sample (approx.. 0.5 g in closed Teflon containers) using HNO_3, H_2O_2 and HF. Analyses were made according to the modified EPA 200.8 method. ALS reported results for the total As, Cd, Pb and Hg mass fractions.

- SCK-CEN applied instrumental neutron activation analysis (k_0-INAA) for the determination of total As, Cd and Hg mass fractions. Three samples of (approx. 0.5 g) were transferred in standard high-density polyethylene vials and weighed. Samples were irradiated for seven hours in the BR1 reactor under a thermal flux of 3 10^{11} n s^{-1} cm^2 together with six IRMM-530 (Al-0.1%Au alloy) neutron flux monitors, and several reference materials for validation (SMELS II; SMELS III; BCR 176 - fly ash; and BCR 278 - mussel tissue). Two spectra per sample were then collected (after 3 and 14 days) on a k_0-calibrated HPGe detector. No additional sample treatment was applied.

- Umweltbundesamt GmbH used microwave assisted digestion with 5 ml HNO_3 + 2 ml H_2O_2 using the total content of each bottle (approx. 0.5 g). The determination of total As, Cd and Pb mass fractions was done by ICP-MS applying EN ISO 17294-2 and of total Hg by CV-AAS applying EN ISO 12846.

- CSPA used ICP-MS after microwave digestion for the total As, Cd and Pb mass fractions, while elemental mercury analysis (EMA) was used for the total Hg mass fraction. All samples (approx. 0.5 g) were weighted in a quartz digestion vessels and 5 mL of HNO_3:H_2O 1:1 were added in a fume hood. The mixture was left to react approximately an hour till the end of the gas generation process. Samples were placed in the microwave digestion system and a two steps digestion approach was applied.

- VITO used high resolution ICP-MS after digestion for the determination of total As, Cd and Pb mass fractions and CV-AFS for total Hg. The test item (approx. 0.5 g) was weighed accurately into a PTFE vessel, and 6 ml of ultrapur nitric acid were added together with 2 ml of ultrapure hydrogen peroxide. The vessels were closed and the samples were digested.

- IRMM used isotope dilution ICP-MS for the determination of the total mass fractions of Cd and Pb. The chocolate pellets were accurately weighed and spiked with the appropriate isotopic CRM. After spiking 5 mL of 60 % ultra-pure nitric acid, 0.5 ml of supra pure H_20_2 was added and the samples were left for one hour to allow for isotopic equilibration before microwave digestion. The obtained sample digests were properly diluted with H_2O and analysed using ICP-MS.

- Institut für Chemie of the University of Graz used microwave digestion with concentrated nitric acid for the mineralisation of the sample (0.5 g of chocolate) combined with ICP-MS for the determination of total As mass fraction. For iAs, samples were heated with a solution of CF_3COOH/H_2O_2 (95°C for 60 min) and analysed by HPLC-ICP-MS.

For this PT, the mean of the means reported by the expert laboratories was used to derive the assigned values (X_{ref}) according to ISO Guide 35:2006 [12].

5.2 Associated uncertainty, u_{ref}

The associated standard uncertainties (u_{ref}) of the assigned values were calculated following the law of uncertainty propagation, combining the standard measurement uncertainty of the characterization (u_{char}) with the standard uncertainty contributions from homogeneity (u_{bb}) and stability (u_{st}), in compliance with ISO Guide 35:2006 [12].

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

In all cases expert laboratories reported values with overlapping expanded measurement uncertainties (Table 1 and Figure 1), hence u_{char} was calculated according to ISO 13528:2005 [8]:

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

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

For iAs, results were requested from one expert laboratory only; his measurement uncertainty was used to set the corresponding u_{char}.

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

5.3 Standard deviation of the proficiency test assessment, σ

The relative standard deviation for PT assessment (σ, in %) was derived from the Horwitz equation modified by Thompson [13] and was set to 19 % for total Cd, 22 % for total As and Pb. Since the mass fraction of iAs in the test item was low , the scientific board of the PT has set the σ to 25% for iAs.

For total Hg mass fractions all expert laboratories stated that their measurement results were below their limit of quantification; therefore the performance of participants for total Hg determination was not scored.

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

	tot-As	tot-Cd	tot-Pb	tot-Hg	iAs
Expert 1	0.0175 ± 0.0023	0.274 ± 0.008	0.0255 ± 0.0013	<0.005	
Expert 2	0.01552 ± 0.0024	0.347 ± 0.06		<0.030	
Expert 3	0.0163 ± 0.002	0.302 ± 0.042	0.0242 ± 0.003	<0.010	
Expert 4	0.015 ± 0.0018				0.0114 ± 0.0028
Expert 5	0.01647 ± 0.0013	0.286 ± 0.02	0.0325 ± 0.0061	<0.001	
Expert 6		0.3017 ± 0.0046	0.02391 ± 0.00061		
Expert 7	<0.040	0.31 ± 0.061	0.029 ± 0.006		
X_{Ref}	**0.0162**	**0.303**	**0.0270**		**0.0114**
u_{char}	0.0006	0.010	0.0011		0.0014
u_{hom}	0.0002	0.002	0.0012		0.0002
u_{st}	0.0008	0.003	0.0005		0.0006
u_{ref}	0.0010	0.011	0.0017		0.0015
U_{ref} (*)	**0.0020**	**0.021**	**0.0030**		**0.0030**
σ	**0.0040**	**0.058**	**0.0060**		**0.0029**
σ (%)	22.0%	19.0%	22.0%		25.0%

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

Note: Expert laboratories do not correspond to the order they are presented in the text.

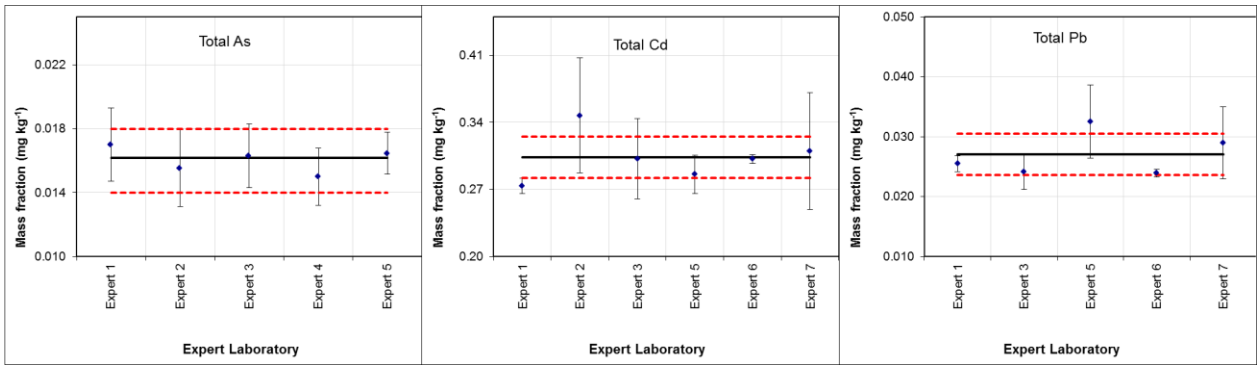

Figure 1: *The assigned values of the PT for the chocolate test item. The rhombuses represent the reported values from the expert laboratories (± U_{cert}); Black solid line represents the assigned value (X_{ref}); the red dashed lines represent the expanded assigned uncertainty interval (X_{ref} ± U_{ref}).*

6. Evaluation of results

6.1 Scores and evaluation criteria

Individual laboratory performance was expressed in terms of z- and ζ -scores in accordance with ISO 13528:2005 [8]:

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

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

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

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

X_{ref} is the assigned value;

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

σ is the standard deviation for proficiency test assessment.

The interpretation of the z- and ζ-score is done according ISO 17043:2010 [7]:

|score| ≤ 2	satisfactory performance	(green in Annexes 11 to 15)
2 < |score| < 3	questionable performance	(yellow in Annexes 11 to 15)
|score| ≥ 3	unsatisfactory performance	(red in Annexes 11 to 15)

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

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

The standard measurement uncertainty of the laboratory (u_{lab}) was obtained by dividing the reported expanded measurement uncertainty by the reported coverage factor, *k*. When no uncertainty was reported, it was set to zero (u_{lab} = 0). When *k* was not specified, the reported expanded measurement uncertainty was considered as the half-width of a rectangular distribution; u_{lab} was then calculated by dividing this half-width by √3, as recommended by Eurachem and CITAC [14].

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

The standard measurement uncertainty from the laboratory (u_{lab}) is most likely to fall in a range between a minimum and a maximum allowed uncertainty (Case "a": $u_{min} \leq u_{lab} \leq u_{max}$). The minimum allowed uncertainty (u_{min}) is set to the standard uncertainties of the assigned values (u_{ref}). It is unlikely that a laboratory carrying out the analysis on a routine basis would determine the measurand with a smaller measurement

uncertainty than the expert laboratories chosen to establish the assigned value. The maximum allowed uncertainty is set to the standard deviation accepted for the PT assessment (σ). Consequently, Case "a" becomes: $u_{ref} \leq u_{lab} \leq \sigma$.

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

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

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

6.2 General observations

One hundred and six participants from 42 countries of which 33 NRLs, registered to the exercise (Fig 2). The Estonian and Luxemburg NRLs did not participate in the PT. Five laboratories did not report results

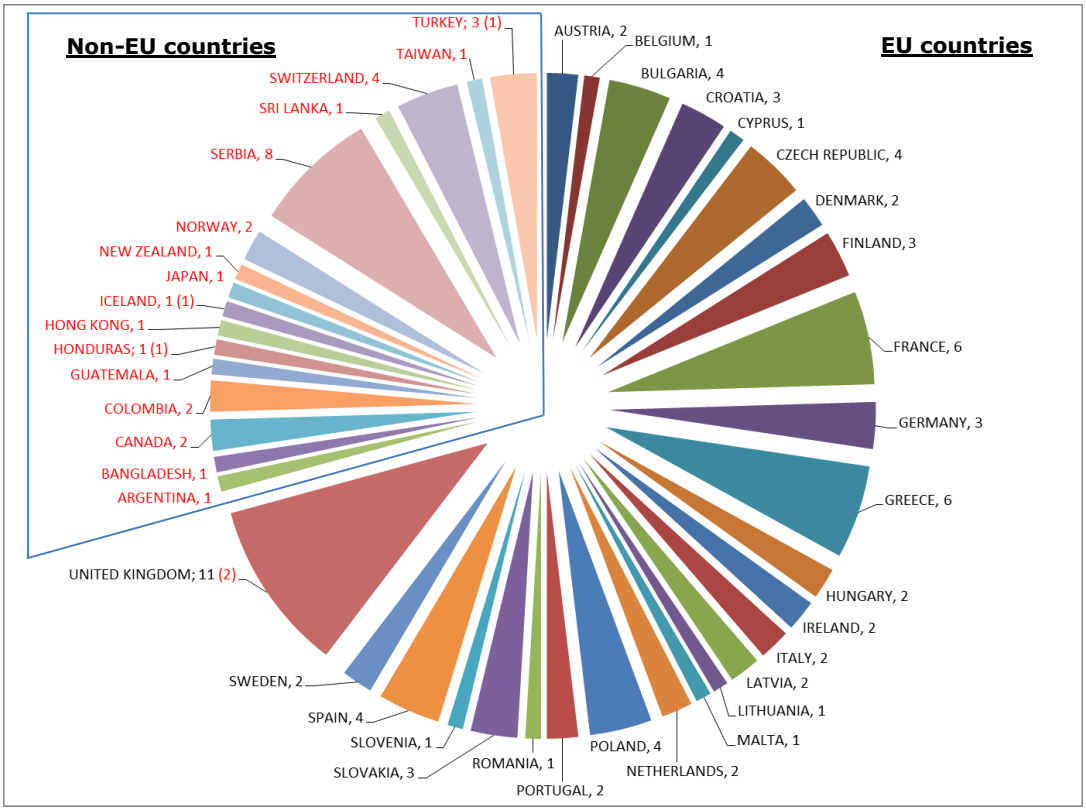

Figure 2: Countries having registered in EURL-HM-20 from the European Union and the rest of the world. 106 laboratories registered of which 101 reported. The number of laboratories that did not return results is indicated in parentheses.

6.3 Laboratory results and scorings

6.3.1 Performances

Annexes 12 to 16 present the reported results as tables and graphs for each measurand, where NRLs and non-NRLs, are denoted as NXXX and LXXX, respectively. 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 [15] are also included.

Figure 3 presents the reporting and performance statistics of the PT, expressed as z- and ζ-scores for the whole population, for NRLs and non-NRLs sub-populations. Participants performed satisfactorily for the determination of the total Cd mass fraction (96%) while poorer performances were recorded for the total As, Pb and iAs mass fractions, where 61, 67 and 64% of satisfactory performances were observed.

No direct correlations could be found between the analytical methods used by the participants and the quality of their reported results. The main observations are summarised hereafter.

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. When the reported limit value was lower than the corresponding $X_{ref} - U_{ref}$, this statement was considered incorrect (flagged in red in Annexes 12 - 15), since the laboratory should have detected the corresponding element. Three laboratories reported incorrect "less than" values: N020 (0.005 mg kg^{-1}) and N021 (0.01 mg kg^{-1}) for the total As mass fraction for which "X_{ref} - U_{ref}" = 0.014 mg kg^{-1}; and N009 (0.02 mg kg^{-1}) for the total mass fraction of Pb for which "$X_{ref} - U_{ref}$" = 0.02 mg kg^{-1}.

For the total As mass fraction the low percentage of satisfactory performances (61%) could be attributed to the relatively low concentration of the measurand (0.0162 ± 0.0020 mg kg^{-1}). This hypothesis is further confirmed by the 33 out of 87 laboratories having reported "less than" values. Questionable or unsatisfactory performances were due to overestimated values which may be attributed to contamination at low total As concentration.

The same was observed for the even lower concentration of iAs mass fraction. Only 27 laboratories reported results (16 NRLs) half of which (13) where "less than" values. For the remaining 14 laboratories, 64 % of them (78 % of the NRLs) performed satisfactorily.

For the total Cd mass fraction all participants except two non-NRLs, reported results with an overall satisfactory performance of 96% (100% for the NRLs).

For the total Pb mass fraction where the assigned value was relatively low (0.0270 ± 0.0030 mg kg^{-1}) 67% of the participants performed satisfactorily (77% for NRLs). Twenty one (6 NRLs) laboratories reported "less than" values. Most of the unsatisfactory performances (22 out of 25) were due to overestimation. From the 96 laboratories that reported results for total Pb, 30 (9 NRLs) used AAS based techniques for their analysis, from which 11 reported "less than" and 12 questionable/unsatisfactory results. As for the ICP based techniques 42 out of the 65 participant using them, performed satisfactorily.

A total of twenty-three participants (14 NRLs) reported results for all measurands, but only seven laboratories performed satisfactorily for total As, Cd, Pb and iAs.

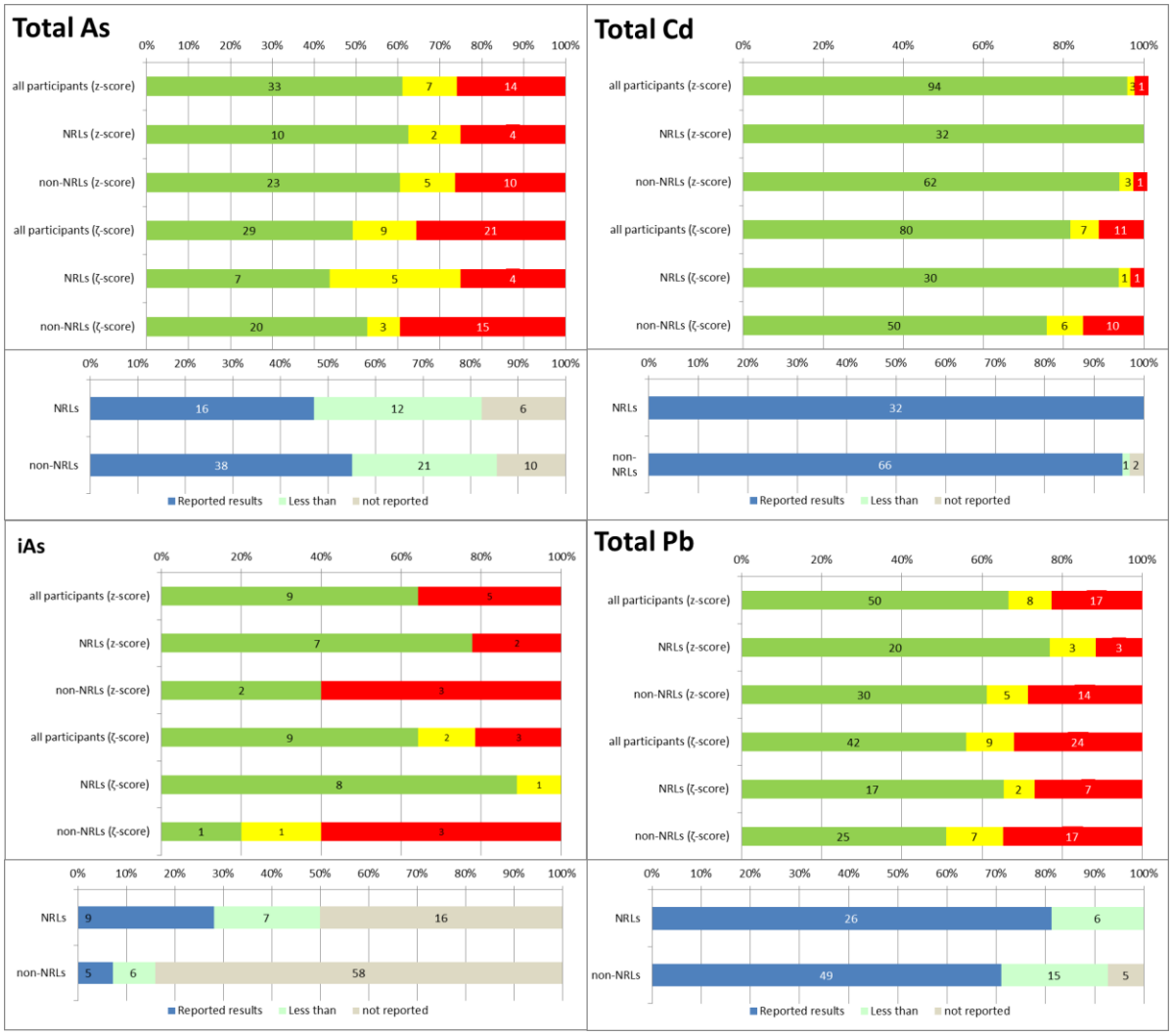

Figure 3: *Number of laboratories with satisfactory (green), questionable (yellow) and unsatisfactory performance (red) together with the respective number of participants that reported results, less than values or did not report at all for each measurand.*

Table 2 - *Uncertainty assessment per analyte*

	$u_{ref} \leq u_{lab} \leq \sigma$		$u_{lab} < u_{ref}$		$u_{lab} > \sigma$	
	NRLs	**Non-NRLs**	**NRLs**	**Non-NRLs**	**NRLs**	**Non-NRLs**
Tot-As	11(69%)	16(42%)	3(19%)	13(14%)	2(12%)	9(24%)
Tot-Cd	26(82%)	36(54%)	3(9%)	9(14%)	3(9%)	21(32%)
Tot-Pb	17(65%)	18(37%)	5(20%)	15(30%)	4(15%)	16(33%)

For the total Hg mass fraction the expert laboratories reported "less than" values ranging from 0.001 to 0.03 mg kg^{-1}. However, Annex 15 shows that 41 participants (14 NRLs) reported values for total Hg, from which only one was lower than 0.001 mg kg^{-1}. In five cases (L052, L059, L083, L102, N002, N027) the values reported were higher than 0.03 mg kg^{-1}. Three laboratories (L052, L074 and N018) reported values lower than their respective LODs.

In all cases, the percentage of satisfactory ζ-scores was lower than the corresponding one for z-scores, indicating that that several laboratories should improve their estimate of measurement uncertainty.

In general NRLs performed better than non-NRLs, not only in terms of z- and ζ- scores but also for their reasonable measurement uncertainty statements. Most of the NRLs reported realistic measurement uncertainties (case "a" $u_{ref} \leq u_{lab} \leq \sigma$, cf. Table 2 and Annexes 12-15): 69%/42% for total As; 82%/54% for total Cd; and 65%/37% for total Pb (NRLs/non-NRLs). Table 2 does not include data for iAs due to the low number of reported results.

6.3.2 Analysis of the information extracted from the questionnaire

The questionnaire was answered by 93 (out of 101) participants. Several approaches were used to evaluate measurement uncertainties (Table 3). The majority of the NRLs (30 out of 32) report uncertainty to their customers, 26 out of the 61 non-NRLs do the same. A total of 152 out of 229 results were assessed with satisfactory ζ-score, from which 87 corresponded to realistic uncertainty estimates (case "a"). 71% of the latter were obtained by laboratories reporting regularly measurement uncertainty to their customers.

Laboratories were asked to report the LODs of the methods used for the determination of the six measurands. LODs, the respective techniques and the general experimental conditions used are summarised in Annex 17. Large discrepancies in reported LODs are observed even among laboratories using the same technique.

Sixty laboratories determined recovery factors for their analyses ranging from 25 to 132 %. NRLs reported recoveries in the range of 74 - 111 %. 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. Several approaches for the determination of recovery were used by the participants, as shown in Table 4.

Table 3 - *Approaches used by the participants in EURL-HM-20 to estimate the uncertainty of their measurements. Multiple selections were possible.*

Approach followed for uncertainty calculation	**Number of labs.**
Uncertainty budget (ISO-GUM), validation	32
Known uncertainty of the standard method (ISO 21748)	2
Uncertainty of the method (in-house)	65
Measurement of replicates (precision)	31
Estimation based on judgment	2
Use of intercomparison data	17
Other: Calculation based on guidelines of NORDTEST: 1 lab Quantifying Uncertainty in Analytical Measurement-Eurachem: 1 lab	2

Table 4 - *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)	42
Using a certified reference material	34
Other: - "Using internal standard or RM"	5

Seventy nine participants (30 NRLs) stated that they are accredited for one or more of the investigated measurands, according to ISO/IEC 17025. Slightly better perfomances were observed for the accredited laboratories (accredited/non-accredited): 70%/61% for total As; 97%/84% for total Cd; 69%/57% for total Pb; and 57%/60% for iAs.

The majority of the laboratories (86) regularly take part in PTs.

No correlation between performance and experience (evaluated as number of analyses per year) on the specific analysis could be identified for all measurands.

Conclusion

The overall performance of the participating laboratories on the determination of the total mass fraction of Cd in the chocolate test items, was satisfactory. (96% for non-NRLs, 100% for the NRLs). This clearly confirms the analytical capabilities of the participating laboratories to enforce the newly amended European Regulation (EC) No 1881/2006 setting MLs for cadmium in cocoa and chocolate.

For low natural concentrations of total As, Pb and iAs (ranging from 0.011 to 0.027 mg kg^{-1}) the laboratories performed satisfactorily (from 61 to 67 %; from 63 to 89% for NRLs). These concentrations were below the LODs of several laboratories. In the case of iAs only 27% of the participants reported results.

In general NRLs performed better than non-NRLs, when referring to z- and ζ-scores as well as for their reasonable measurement uncertainty statements. However, the percentage of satisfactory ζ-scores was lower than the corresponding one for z-scores, indicating that several laboratories should improve their estimate of measurement uncertainty. Measurement uncertainty is of paramount importance in case of litigations and the capability of control laboratories to estimate it correctly is a fundamental requirement.

Another area of improvement relates to the proper determination and/or declaration of limits of detection and quantification. Significant discrepancies were observed for the LODs reported even for similar analytical methods, which may be attributed to the confusion between the LOD of an analytical method and the instrumental LOD.

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List of abbreviations and definitions

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 SANTE	Directorate General for Health and Food Safety
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
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

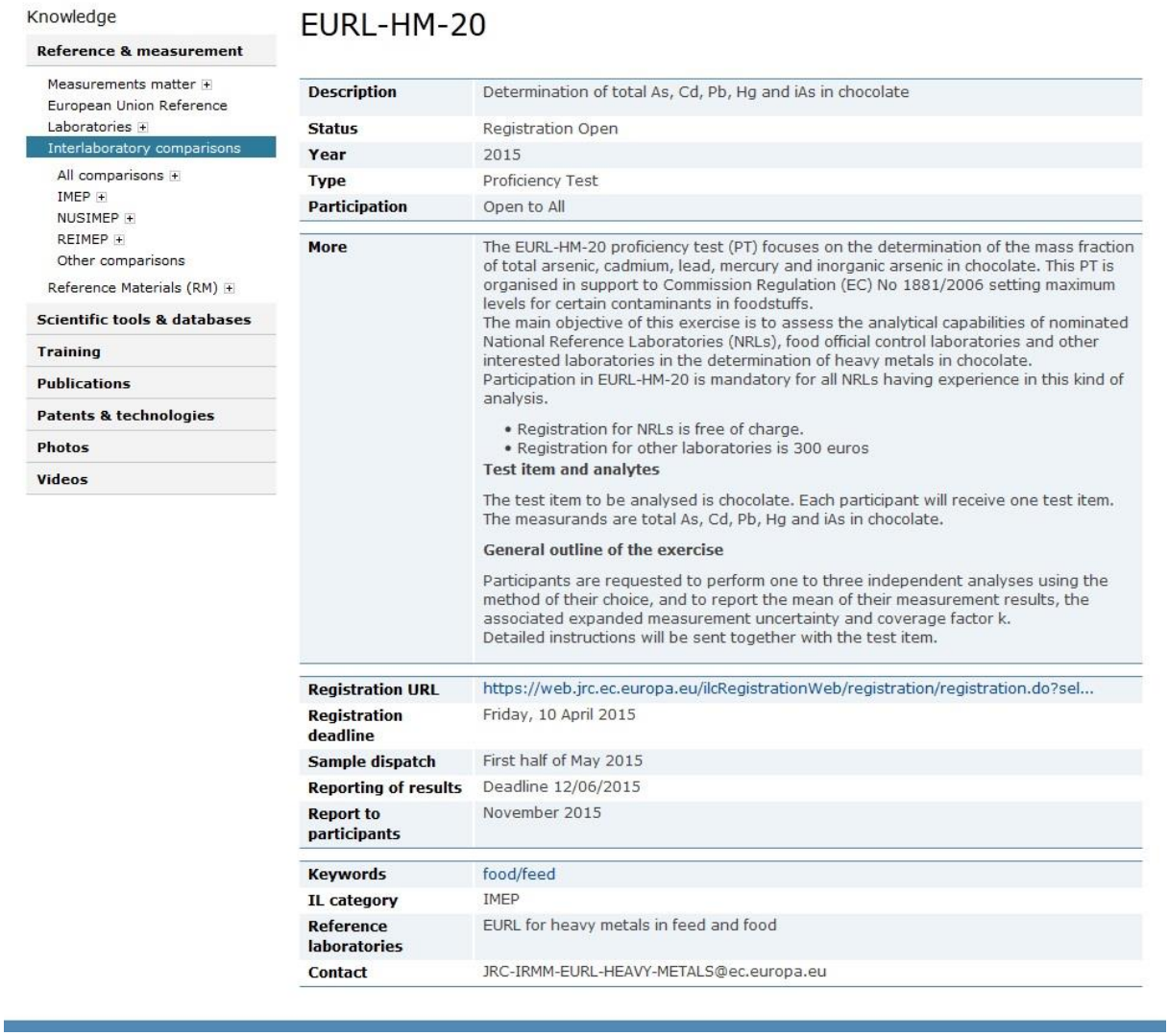
Annexes

Annex 1: List of Participants

Organisation	***Country***
JLA Argentina S.A	*ARGENTINA*
AGES GmbH	*AUSTRIA*
ANALYTEC GmbH	*AUSTRIA*
Bangladesh Atomic Energy Commission	*BANGLADESH*
CODA-CERVA	*BELGIUM*
Central Laboratory for Chemical Testing and Control	*BULGARIA*
ACLT Komihris	*BULGARIA*
Di and Vi Consult Ltd	*BULGARIA*
SGS Bulgaria Ltd	*BULGARIA*
University of Guelph, Laboratory Services	*CANADA*
SGS Canada Inc	*CANADA*
Laboratorio Microbiológico Barranquilla S.A.S	*COLOMBIA*
Tecnimicro Laboratorio de Análisis S.A.S.	*COLOMBIA*
Croatian National Institute of Public Health	*CROATIA*
Faculty of Food Technology and Biotechnology	*CROATIA*
Teaching Institute of Public Health	*CROATIA*
STATE GENERAL LABORATORY	*CYPRUS*
State Veterinary Institute Olomouc	*CZECH REPUBLIC*
CISTA	*CZECH REPUBLIC*
Statni veterinarni ustav Praha	*CZECH REPUBLIC*
Laborator M O R A V A s.r.o.	*CZECH REPUBLIC*
DTU Food	*DENMARK*
Veterinary and Food Administration	*DENMARK*
Finnish Food Safety Authority Evira	*FINLAND*
Finnish Customs Laboratory	*FINLAND*
MetropoliLab Oy	*FINLAND*
Frenche Agency for food, Environmental and Occupational Health and Safety	*FRANCE*
LABORATOIRE SCL DE BORDEAUX	*FRANCE*
Laboratoire Phytocontrol	*FRANCE*
CAMP66	*FRANCE*
La Drôme laboratoire	*FRANCE*
Nestlé NQAC Cergy	*FRANCE*
Federal Office for Consumer Protection and Food Safety (BVL)	*GERMANY*
Landesbetrieb Hessisches Landeslabor	*GERMANY*
Lebensmittel- und Veterinärinstitut Oldenburg (LVIOL/LAVES)	*GERMANY*
REGIONAL CENTER OF PLANT PROTECTION AND QUALITY CONTROL OF MAGNISSIA	*GREECE*
GENERAL CHEMICAL STATE LABORATORY	*GREECE*
GENERAL CHEMICAL STATE LABORATORY	*GREECE*
General Chemical State Laboratory,	*GREECE*
AGROLAB-RDS	*GREECE*
A. TSAKALIDIS INC	*GREECE*
Universidad Mariano Gálvez	*GUATEMALA*
Enviro Labs Limited	*HONG KONG*
Corvinus University of Budapest- Dept. Applied Chem.	*HUNGARY*
National Food Chain Office Food and Feed Safety Directorate	*HUNGARY*
Health Service Executive	*IRELAND*
Public Analyst's Laboratory Dublin	*IRELAND*
Istituto Superiore di Sanità	*ITALY*
ISTITUTO ZOOPROFILATTICO SPERIMENTALE DEL PIEMONTE, LIGURIA E VALLE D'AOSTA	*ITALY*
JAPAN FROZEN FOODS INSPECTION CORPORATION	*JAPAN*
Institute of Food Safety, Animal Health and Environment	*LATVIA*
Latvian Certification Centre Ltd	*LATVIA*
National Food and Veterinary Risk Assessment Institute	*LITHUANIA*
Environmental Health Directorate	*MALTA*

RIKILT	*NETHERLANDS*
Food & Consumer Products Safety Authority	*NETHERLANDS*
AsureQuality Auckland Laboratory	*NEW ZEALAND*
NIFES	*NORWAY*
Trondheim kommune	*NORWAY*
National Institute of Public Health - National Institute of Hygiene (NIPH - NIH)	*POLAND*
SGS Polska sp z o.o.	*POLAND*
Wojewodzka Stacja Sanitarno-Epidemiologiczna we Wroclawiu	*POLAND*
Wojewódzka Stacja Sanitarno-Epidemiologiczna	*POLAND*
ASAE	*PORTUGAL*
ISQ	*PORTUGAL*
Sanitary Veterinary and Food Safety Laboratory Bucharest	*ROMANIA*
Jugoinspekt Beograd AD	*SERBIA*
A BIO TECH LAB d.o.o.	*SERBIA*
Faculty of Technology, University of Novi Sad	*SERBIA*
MP BIO d.o.o., MP LAB Laboratory testing	*SERBIA*
Institute of Public Health of Vojvodina	*SERBIA*
Institute of Public Health Kraljevo	*SERBIA*
Center for Food Analysis	*SERBIA*
Institute of public health Kragujevac	*SERBIA*
Veterinary and food institute in Košice	*SLOVAKIA*
State Veterinary and Food Institute	*SLOVAKIA*
Regional Public Health Institute in Žilina	*SLOVAKIA*
National Laboratory for Health, Environment and Food - Maribor	*SLOVENIA*
MAGRAMA	*SPAIN*
LABORATORIO DE SALUD PUBLICA (MADRID SALUD) AYUNTAMIENTO DE MADRID	*SPAIN*
SILLIKER IBERICA	*SPAIN*
PUBLIC HEALTH LABORATORY OF BARCELONA	*SPAIN*
Industrial Technology Institute	*SRI LANKA*
National Food Agency	*SWEDEN*
ALS Scandinavia AB	*SWEDEN*
Coop	*SWITZERLAND*
Laboratoire cantonal du Jura	*SWITZERLAND*
UFAG Laboratorien AG	*SWITZERLAND*
Kantonales Labor Zürich	*SWITZERLAND*
Intertek Testing Services Taiwan Ltd.	*TAIWAN*
ACIBADEM LABVITAL FOOD CONTROL LABORATORY	*TURKEY*
Ege Chelab Gıda ve Endüstriyel Analiz Laboratuvarları A.Ş.	*TURKEY*
Fera	*UNITED KINGDOM*
Reading Scientific Services Ltd	*UNITED KINGDOM*
Covance Laboratories Limited	*UNITED KINGDOM*
Hampshire Scientific Service	*UNITED KINGDOM*
Worcestershire Scientific Services	*UNITED KINGDOM*
Public Analyst Scientific Services Limited	*UNITED KINGDOM*
Stafordshire County Council	*UNITED KINGDOM*
Kent County Council	*UNITED KINGDOM*
City of Edinburgh Council	*UNITED KINGDOM*

Annex 2: JRC web announcement



Knowledge

Reference & measurement

- Measurements matter
- European Union Reference Laboratories
- Interlaboratory comparisons
  - All comparisons
  - IMEP
  - NUSIMEP
  - REIMEP
  - Other comparisons
- Reference Materials (RM)

Scientific tools & databases

Training

Publications

Patents & technologies

Photos

Videos

EURL-HM-20

Description	Determination of total As, Cd, Pb, Hg and iAs in chocolate
Status	Registration Open
Year	2015
Type	Proficiency Test
Participation	Open to All

More	The EURL-HM-20 proficiency test (PT) focuses on the determination of the mass fraction of total arsenic, cadmium, lead, mercury and inorganic arsenic in chocolate. This PT is organised in support to Commission Regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs. The main objective of this exercise is to assess the analytical capabilities of nominated National Reference Laboratories (NRLs), food official control laboratories and other interested laboratories in the determination of heavy metals in chocolate. Participation in EURL-HM-20 is mandatory for all NRLs having experience in this kind of analysis. • Registration for NRLs is free of charge. • Registration for other laboratories is 300 euros **Test item and analytes** The test item to be analysed is chocolate. Each participant will receive one test item. The measurands are total As, Cd, Pb, Hg and iAs in chocolate. **General outline of the exercise** Participants are requested to perform one to three independent analyses using the method of their choice, and to report the mean of their measurement results, the associated expanded measurement uncertainty and coverage factor k. Detailed instructions will be sent together with the test item.

Registration URL	https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?sel...
Registration deadline	Friday, 10 April 2015
Sample dispatch	First half of May 2015
Reporting of results	Deadline 12/06/2015
Report to participants	November 2015

Keywords	food/feed
IL category	IMEP
Reference laboratories	EURL for heavy metals in feed and food
Contact	JRC-IRMM-EURL-HEAVY-METALS@ec.europa.eu

Annex 3: Invitation letter to NRLs

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

Ref. Ares(2015)835746 - 26/02/2015

Geel, 26 February 2015
JRC.D.5/PRO/IF/acs/ARES

Sent by e-mail

Subject: Proficiency testing for the determination of total As, Cd, Pb, Hg and iAs in chocolate (EURL-HM-20)

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 EURL-HM-20 for the **"Determination of total As, Cd, Pb, Hg and iAs in chocolate"**.

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

Your participation is free of charge.

Please register using the following link:
https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1402

Once you submitted your registration, copy the confirmation page that will appear and send it to JRC-IRMM-EURL-HEAVY-METALS@ec.europa.eu. This e-mail will be the confirmation of your participation.

If you know a laboratory interested in participating in the EURL-HM-20 exercise, please forward this link: https://ec.europa.eu/jrc/en/interlaboratory-comparison/eurl-hm-20?search&form-return

In case you plan to pay for the participation of official food control laboratories belonging to your national network, please inform them that their identity will be disclosed to you.

The **deadline for registration is 10 April 2015**. Samples will be sent to participants during the first half of May 2015. The deadline for submission of results is **12 June 2015**.

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

Yours sincerely

Dr. Ioannis Fiamegkos
EURL-HM-20 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: https://ec.europa.eu/jrc/en/eurl/heavy-metals

Ref. Ares(2015)969096 - 04/03/2015

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
Standards for Food Bioscience Unit

Mr. Baran Bozoğlu,
TÜRKAK
Esat Cad. No. 41
Küçükesat - Ankara
TURKEY

EURL-HM-20: Interlaboratory comparison for the determination of total As, Cd, Pb, Hg and iAs in chocolate

Dear Mr. Bozoğlu,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"EURL-HM-20: Determination of total As, Cd, Pb, Hg and iAs in chocolate"** 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, the organizers will disclose details of the participants that have been nominated by EA to you. The EA accreditation bodies may wish to inform the nominees of this disclosure.

The registration page for laboratories appointed by EA is open until the **10 April 2015**. Distribution of the test items is foreseen for the first half of May 2015. The deadline for submission of results is the **5 June 2015**.
More information about this PT following the link:

https://ec.europa.eu/jrc/en/interlaboratory-comparison/eurl-hm-20?search&form-return

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-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=1402

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

2. **Clearly indicate on the printed form that they have been appointed by the European Cooperation for Accreditation** to take part in this exercise **otherwise the laboratory will be invoiced 300 € for participation** as charged to the non-appointed laboratories.

3. **Send** the printout to both the EURL-HM-20 and the EA coordinators:

EURL-HM-20 coordinator Dr. Ioannis Fiamegkos	**EA coordinator** Mr. Baran Bozoğlu
E-mail: jrc-irmm-imep@ec.europa.eu	E-mail: bbozoglu@turkak.org.tr

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

With kind regards

Ioannis Fiamegkos
EURL-HM-20 Coordinator

Annex 5: Invitation letter to Asian Pacific Laboratory Accreditation Cooperation (APLAC)

Ref. Ares(2015)969066 - 04/03/2015

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
Standards for Food Bioscience Unit

To: Ms Cynthia Chen
APLAC PT Committee

EURL-HM-20: Interlaboratory comparison for the determination of total As, Cd, Pb, Hg and iAs in chocolate

Dear Ms Chen,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"EURL-HM-20: Determination of total As, Cd, Pb, Hg and iAs in chocolate".**

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 **EURL-HM-20** paying a registration fee of 300 €.

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

Registration of participants is open until **10 April 2015**. Distribution of the test items is foreseen for the first half of May 2015, and the deadline for submission of results is **05 June 2015**.
More information about this PT following the link:
https://ec.europa.eu/jrc/en/interlaboratory-comparison/eurl-hm-20?search&form-return

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=1402

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 300 € for participation** normally applied for non-appointed laboratories.
4. **Send** the printout to both the **EURL-HM-20** and the APLAC coordinators:

EURL-HM-20 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
EURL-HM-20 Coordinator

Annex 6: Invitation letter to Inter-American Accreditation Cooperation (IAAC)

Ref. Ares(2015)969129 - 04/03/2015

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
Standards for Food Bioscience Unit

To: Barbara Belzer
IAAC Lab Committee

EURL-HM-20: Interlaboratory comparison for the determination of total As, Cd, Pb, Hg and iAs in chocolate

Dear Mrs. Belzer,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"EURL-HM-20: Determination of total As, Cd, Pb, Hg and iAs in chocolate".**

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 EURL-HM-20 paying a registration fee of 300 €.

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

Registration of participants is open until the **10 April 2015**. Distribution of the test items is foreseen for the first half of May 2015, and the deadline for submission of results is the **5 June 2015**.
More information about this PT following the link:

https://ec.europa.eu/jrc/en/interlaboratory-comparison/eurl-hm-20?search&form-return

In order to register, laboratories must:

1. **Enter** their details online:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1402

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

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 300 € for participation** normally applied for non-appointed laboratories.

4. **Send** the printout to both the **EURL-HM-20** and the IAAC coordinators:

EURL-HM-20 coordinator	**IAAC coordinator**
Ioannis Fiamegkos (*Ph.D*)	Barbara Belzer
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
EURL-HM-20 Coordinator

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Ref. Ares(2015)969018 - 04/03/2015

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
Standards for Food Bioscience Unit

To: Ms Nonhlanhla Halimana

African Accreditation Cooperation

DTI Campus

77 Meintjies Street

Block G, Ground Floor

Sunnyside, Pretoria 0132
South Africa

EURL-HM-20: Interlaboratory comparison for the determination of total As, Cd, Pb, Hg and iAs in chocolate

Dear Ms Halimana,

The Institute for Reference Materials and Measurements (IRMM) organises a proficiency test named **"EURL-HM-20: Determination of total As, Cd, Pb, Hg and iAs in chocolate".**

IRMM kindly invites AFRAC 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 EURL-HM-20 paying a registration fee of 300 €.

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

Registration of participants is open until **10 April 2015**. Distribution of the test items is foreseen for the first half of May 2015, and the deadline for submission of results is **05 June 2015**.
More information about this PT following the link:
https://ec.europa.eu/jrc/en/interlaboratory-comparison/eurl-hm-20?search&form-return

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=1402

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 AFRAC** to take part in this exercise **otherwise the laboratory will be invoiced 300 € for participation** normally applied for non-appointed laboratories.

4. **Send** the printout to both the EURL-HM-20 and the AFRAC coordinators:

EURL-HM-20 coordinator	**AFRAC coordinator**
Ioannis Fiamegkos	Nonhlanhla Halimana
Fax +32 14 571 865	Fax +27 12 394 4788
E-mail: jrc-irmm-imep@ec.europa.eu	E-mail: nonhlanhlah@sanas.co.za

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

With kind regards

Dr. Ioannis Fiamegkos
EURL-HM-20 Coordinator

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

Geel, 21 April 2015
JRC.D5/IF/acs/Ares(2015)1689149

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

Subject: Participation in EURL-HM-20

Dear «Title» «Surname»,

Thank you for participating in the EURL-HM-20 proficiency test for the determination of total As, Cd, Pb, Hg and iAs in chocolate. This proficiency test (PT) 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 pack of six bottles containing the test item (approx. 0.5 g / bottle) for the determination of the of total As, Cd, Pb, Hg and iAs.

(b) A "Confirmation of receipt" letter,

Please check whether the bottles containing the test item are 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). The test item is to be kept in a dark place at 4°C until analysis.

The measurands are total As, Cd, Pb, Hg, and iAs in chocolate.

The procedure used for the analyses should resemble as closely as possible the one that you use in routine analyses. **The content of each bottle is to be fully used as test portion for the analysis**, no sub-sampling within a bottle is allowed because homogeneity could not be warranted for aliquots smaller than 0.5 g.

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:

- 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 **12/06/2015**.

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)
EURL-HM-20 Coordinator

Cc: F. Ulberth (SFB HoU), P. Robouch

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 9: Confirmation of receipt form

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

Geel, 21 April 2015
JRC.D5/IF/acs/Ares(2015)1689149

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

EURL-HM-20

Heavy Metals in chocolate

Confirmation of receipt of the samples

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

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

Date of package arrival .....

Signature .....

Please return this form to:

Dr Ioannis Fiamegkos

EURL-HM-20 Coordinator
EC-JRC-IRMM
Retieseweg 111
B-2440 GEEL, Belgium

Fax : +32-14-571865

e-mail : JRC-IRMM-EURL-HEAVY-METALS@ec.europa.eu

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211
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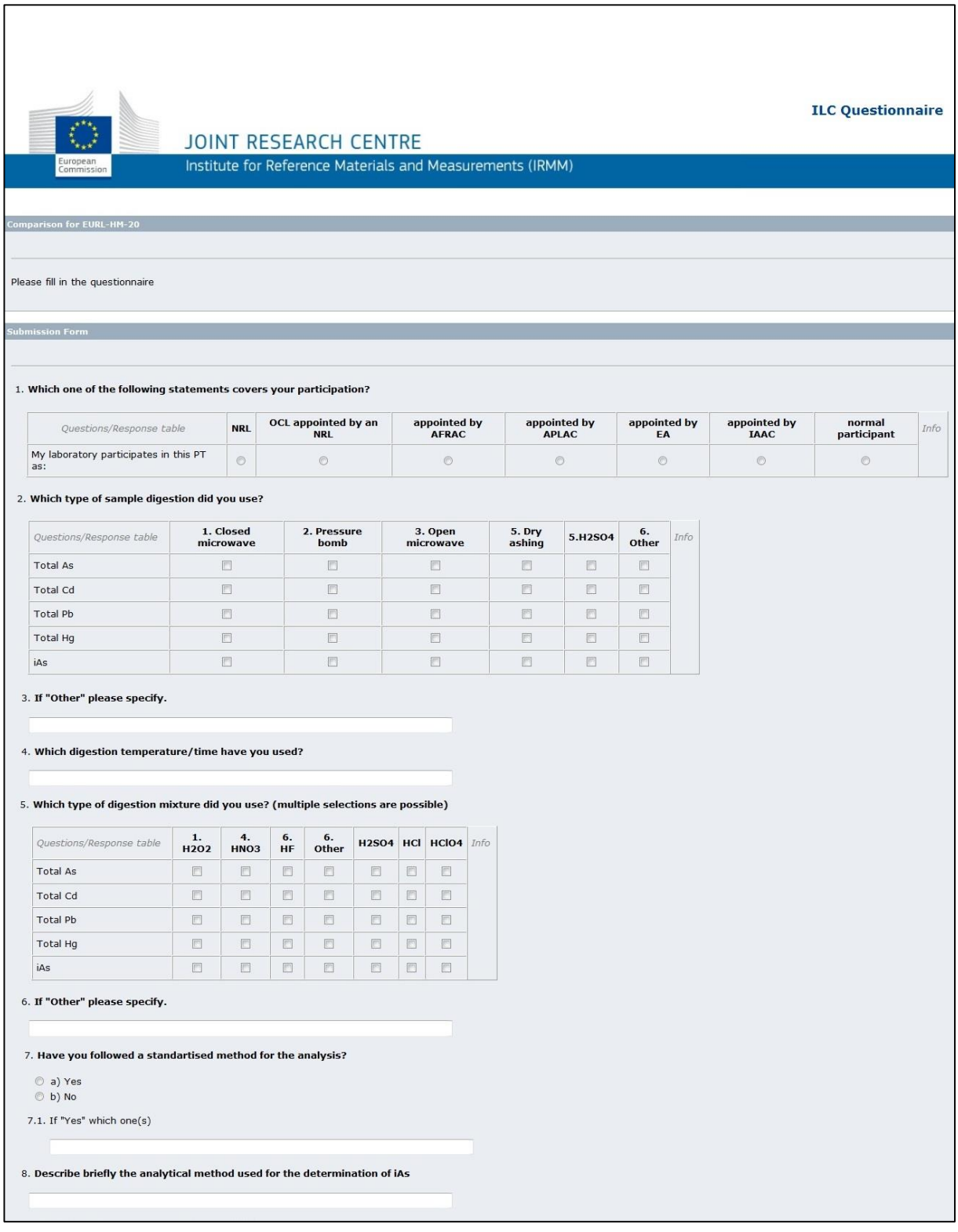
E-mail: JRC-IRMM-EURL-HEAVY-METALS@ec.europa.eu
Web site: http://irmm.jrc.ec.europa.eu

Annex 10: Questionnaire



ILC Questionnaire

JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements (IRMM)

Comparison for EURL-HM-20

Please fill in the questionnaire

Submission Form

1. **Which one of the following statements covers your participation?**

Questions/Response table	NRL	OCL appointed by an NRL	appointed by AFRAC	appointed by APLAC	appointed by EA	appointed by IAAC	normal participant	*Info*
My laboratory participates in this PT as:	○	○	○	○	○	○	○	

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

Questions/Response table	1. Closed microwave	2. Pressure bomb	3. Open microwave	5. Dry ashing	5.H2SO4	6. Other	*Info*
Total As	☐	☐	☐	☐	☐	☐	
Total Cd	☐	☐	☐	☐	☐	☐	
Total Pb	☐	☐	☐	☐	☐	☐	
Total Hg	☐	☐	☐	☐	☐	☐	
iAs	☐	☐	☐	☐	☐	☐	

3. **If "Other" please specify.**

4. **Which digestion temperature/time have you used?**

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

Questions/Response table	1. H2O2	4. HNO3	6. HF	6. Other	H2SO4	HCl	HClO4	*Info*
Total As	☐	☐	☐	☐	☐	☐	☐	
Total Cd	☐	☐	☐	☐	☐	☐	☐	
Total Pb	☐	☐	☐	☐	☐	☐	☐	
Total Hg	☐	☐	☐	☐	☐	☐	☐	
iAs	☐	☐	☐	☐	☐	☐	☐	

6. **If "Other" please specify.**

7. **Have you followed a standartised method for the analysis?**

○ a) Yes
○ b) No

7.1. If "Yes" which one(s)

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

10. **Additional remarks/comments regarding the method of analysis.**

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

(Certified) reference materials

Questions/Response table	Total As	Total Cd	Total Pb	Total Hg	iAs
Method validation (Recovery)					
Instrument calibration					

12. **Are you accredited for the determination of this analyte?**

Questions/Response table	**Total As**	**Total Cd**	**Total Hg**	**Total Pb**	**iAs**	*Info*
Accredited for:	☐	☐	☐	☐	☐	

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

Questions/Response table	**0-50**	**251-1000**	**51-250**	**>1000**	**Never**	*Info*
Total As	☐	☐	☐	☐	☐	
Total Cd	☐	☐	☐	☐	☐	
Total Pb	☐	☐	☐	☐	☐	
Total Hg	☐	☐	☐	☐	☐	
iAs	☐	☐	☐	☐	☐	

14. **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 judgemnt
- ☐ f) From interlaboratory comparison data
- ☐ g) Other

14.1. If "Other" please specify.

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

- ○ a) Yes
- ○ b) No

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

- ○ a) Yes
- ○ b) No

16.1. If "Yes", which:

- ☐ a) ISO 17025:2005
- ☐ b) ISO 9000 series
- ☐ c) Other

16.1.1. If "Other" please specify.

17. **Does your laboratory take part in proficiency tests (PTs) for this type of analysis?**

- ○ a) Yes
- ○ b) No

17.1. If "Yes" which one(s)?

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

Annex 11: Homogeneity and stability studies

11.1 Homogeneity studies (all values in mg kg^{-1})

	As		Cd		Pb	
Bottle ID	*R1*	*R2*	*R1*	*R2*	*R1*	*R2*
142	0.017	0.017	0.305	0.308	0.023	0.025
99	0.018	0.017	0.311	0.303	0.025	0.025
10	0.017	0.017	0.305	0.308	0.028	0.024
72	0.018	0.017	0.303	0.299	0.025	0.024
15	0.017	0.017	0.301	0.302	0.025	0.024
180	0.017	0.017	0.312	0.306	0.032	0.024
56	0.017	0.017	0.305	0.305	0.028	0.025
32	0.017	0.016	0.305	0.303	0.024	0.024
123	0.018	0.017	0.303	0.306	0.023	0.024
190	0.017	0.016	0.304	0.306	0.043 (*)	0.029
Mean	0.016915		0.305		0.02610	
s_p	0.003555		0.058		0.00594	
0.3* s_p	0.001066		0.017		0.00178	
Critical value	0.000001		0.0002		0.00001	
s_x	0.000325		0.002		0.00152	
s_w	0.000377		0.003		0.00219	
s_s	0.000185		0.001		0.00000	
$s_s \leq 0.3 * \sigma$ (ISO 13528)	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,

(*) flagged as Grubbs outlier and excluded from the calculations

11.2 Stability studies (all values in mg kg^{-1})

	Time in Weeks				
	0	**3**	**5**	**8**	**u_{st}**
As	0.0174	0.0159		0.017	
	0.0183	0.0154	0.0161	0.0158	**5.0%**
Cd	0	3	5	8	
	0.283	0.285	0.286	0.277	
	0.278	0.283	0.28	0.275	**1.0%**
Pb	0	3	5	8	
	0.0225	0.0246	0.0245	0.0235	
	0.0239	0.0236	0.0236	0.0236	**2.0%**

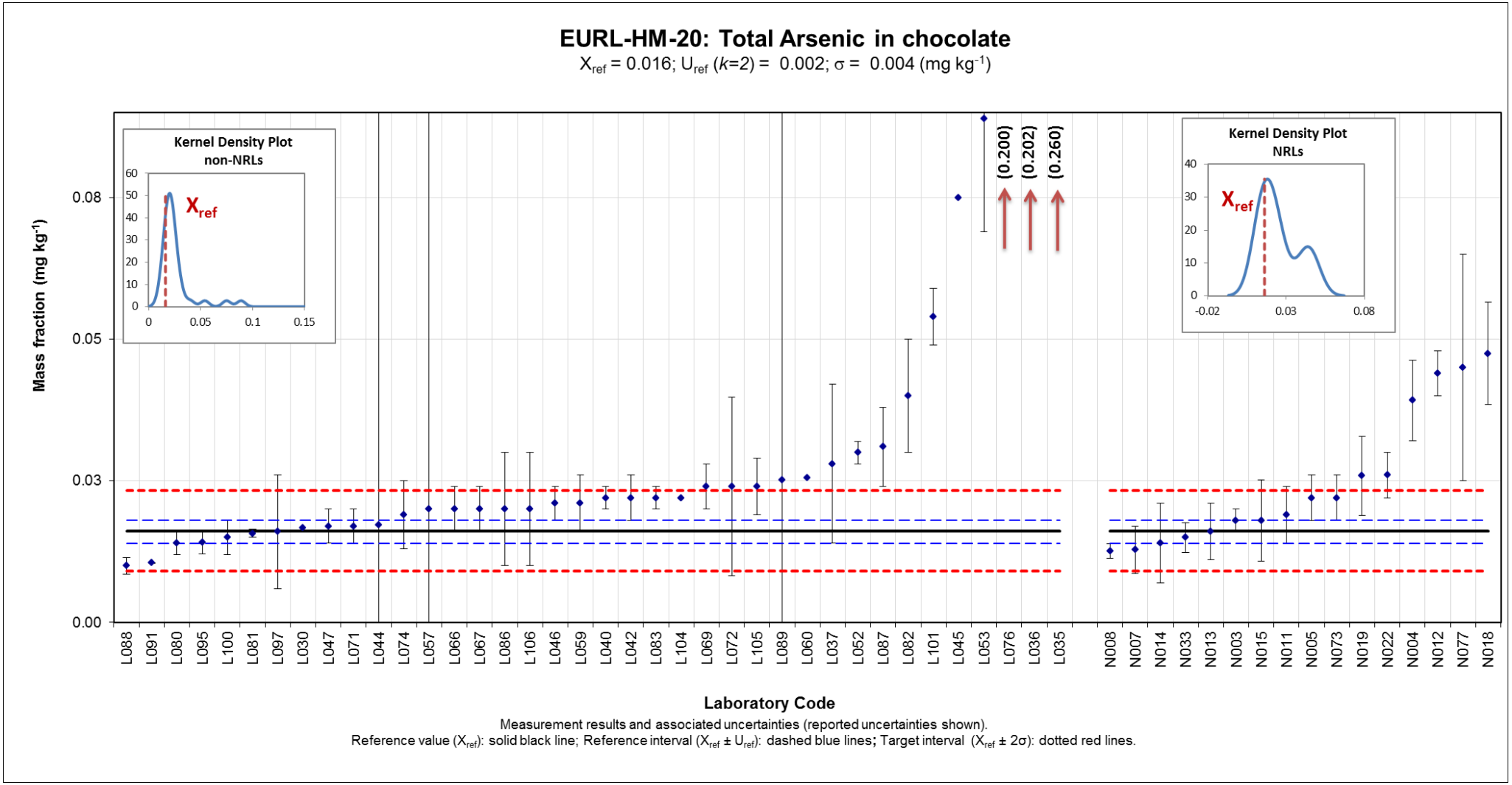
Where: u_{st} is the standard measurement uncertainty due to stability (6 weeks, expressed as a %)

Annex 12: Results for total As

Assigned range: X_{ref} = 0.016; U_{ref} (k=2) = 0.002; σ = 0.004 (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]
N001	< 0.1000		v3	AAS				
N002	< 0.0670		v3	AAS				
N003	0.018	0.002	2	ICP-MS	0.001	0.52	1.3	b
N004	0.0392	0.0071	2	ICP-MS	0.0036	6.48	6.24	a
N005	0.022	0.004	2	ICP-MS	0.002	1.64	2.61	a
N007	0.0128	0.0042	2	ICP-MS	0.0021	-0.94	-1.44	a
N008	0.0126	0.0013	2	ICP-MS	0.0006	-1	-2.97	b
N009	< 0.1000			HG-AAS				
N011	0.019	0.005	2	ICP-MS	0.0025	0.8	1.05	a
N012	0.044	0.004	2	ICP-MS	0.002	7.83	12.43	a
N013	0.016	0.005	2	ICP-MS	0.0025	-0.04	-0.06	a
N014	0.014	0.007	2	HG-AAS	0.0035	-0.61	-0.59	a
N015	0.018	0.0072	2	ICP-MS	0.0036	0.52	0.49	c
N016	< 0.0250			ICP-MS				
N017	< 0.0200			ICP-MS				
N018	0.0475	0.009	v3	ICP-MS	0.0052	8.82	5.91	c
N019	0.0259	0.007	2	ETAAS	0.0035	2.74	2.67	a
N020	< 0.0050			ICP-MS				
N021	< 0.0100			ICP-MS				
N022	0.026	0.004	2	ICP-MS	0.002	2.77	4.39	a
N025	< 0.0750			HG-AAS				
N026	< 0.2000			ICP-MS				
N027	< 0.0200			ICP-MS				
N033	0.015	0.0026	2	ICP-MS	0.0013	-0.33	-0.7	a
N034	< 0.0400			AAS				
N038	< 0.0300			ICP-MS				
N073	0.022	0.004	2	ICP-MS	0.002	1.64	2.61	a
N077	0.045	0.02	2	ICP-MS	0.01	8.11	2.87	c
L029	< 0.0300			ICP-MS				
L030	0.0167			ICP-MS	0	0.15	0.54	b
L032	< 0.1000			ICP-OES				
L035	0.26	0.07	2	ICP-MS	0.035	68.6	6.96	c
L036	0.2024	0.0023	2	ICP-OES	0.0012	52.39	120.89	a
L037	0.028	0.014	2	ICP-MS	0.007	3.33	1.67	c
L040	0.022	0.002	v3	ICP-MS	0.0012	1.64	3.81	a
L041	< 0.0280		2	AAS-GF				
L042	0.022	0.004	2	ICP-MS	0.002	1.64	2.61	a
L044	0.0172	20	2	ICP-MS	10	0.29	0	c
L045	0.075			H-AAS	0	16.55	58.38	b
L046	0.021	0.003	2	ICP-MS	0.0015	1.36	2.68	a
L047	0.017	0.003	2	FIAS.	0.0015	0.24	0.47	a
L049	< 0.0400		2.94	ICP-MS				
L050	< 0.0500			ICP-MS				
L051	< 2.5000			ICP-OES				
L052	0.03	0.002	0	HG-AAS	0.5	3.89	0.03	c
L053	0.089	0.02	2	HG-AAS	0.01	20.49	7.25	c
L055	< 1.0000			ICP-OES				
L056	< 0.2400			HG-AAS				
L057	0.02	16	2	AAS-VGA	8	1.08	0	c
L059	0.021	0.005	2	ICP-MS	0.0025	1.36	1.8	a
L060	0.0255		v3	ICP-MS	0	2.63	9.27	b
L061	< 0.1000			ICP-MS				
L063	< 0.1000			AAS				
L065	< 0.0300			ICP-MS				
L066	0.02	0.004	2	ICP-MS	0.002	1.08	1.72	a
L067	0.02	0.004	2	ICP-MS	0.002	1.08	1.72	a
L068	< 0.1000			ICP-MS				
L069	0.024	0.004	v3	ICP-MS	0.0023	2	3.11	a
L071	0.017	0.003	2	SEM-ICP-MS	0.0015	0.24	0.47	a
L072	0.024	0.0157	2	ICP-MS	0.0078	2	0.99	c
L074	0.019	0.006	2	ICP-MS	0.003	0.8	0.9	a
L075	< 0.0700			HG-AAS				
L076	0.2	0.01	2	ICP-OES	0.005	51.72	36.04	c
L078	< 0.2000			AAS				
L079	< 0.5000			AAS				
L080	0.014	0.002	2	ICP-MS	0.001	-0.61	-1.52	b
L081	0.0157	0.0007	1.96	HG-AAS	0.0004	-0.13	-0.43	b
L082	0.04	0.01	2	HG-AAS	0.005	6.71	4.67	c
L083	0.022	0.002	2	ICP-MS	0.001	1.64	4.11	b
L084	< 0.0200			HG-AAS				
L085	< 0.1000			AAS				
L086	0.02	0.01	2	ICP-MS	0.005	1.08	0.75	c
L087	0.031	0.007	2	ICP-MS	0.0035	4.18	4.07	a
L088	0.01	0.0015	2	HG-AAS	0.0008	-1.73	-4.9	b
L089	0.0252	0.245	2	ICP-MS	0.1225	2.54	0.07	c
L091	0.0105	0.0001	v3	ICP-MS	0.0001	-1.59	-5.6	b
L092	< 0.0200			ICP-MS				
L094	< 0.0700			ICP-MS				
L095	0.0142	0.0021	2	ICP-MS	0.0011	-0.55	-1.34	a
L097	0.016	0.01	2	HG-AAS	0.005	-0.04	-0.03	c
L099	< 0.0500		100	AAS				
L100	0.015	0.003	2	ICP-OES	0.0015	-0.33	-0.64	a
L101	0.054	0.005	2	AFS	0.0025	10.65	14.04	a
L102	< 0.1000			HG-AAS				
L104	0.022		v3	ICP-MS	0	1.64	5.8	b
L105	0.024	0.005	2	ICP-MS	0.0025	2	2.91	a
L106	0.02	0.01	2	ICP-MS	0.005	1.08	0.75	c

[a] √3 is set by the ILC coordinator when no expansion factor k is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3,
[b] performance: satisfactory, questionable, unsatisfactory,
[c] a : $u_{min}(u_{ref}) \leq u_{lab} \leq u_{max}(\sigma)$; b : $u_{lab} < u_{min}$; and c : $u_{lab} > u_{max}$



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

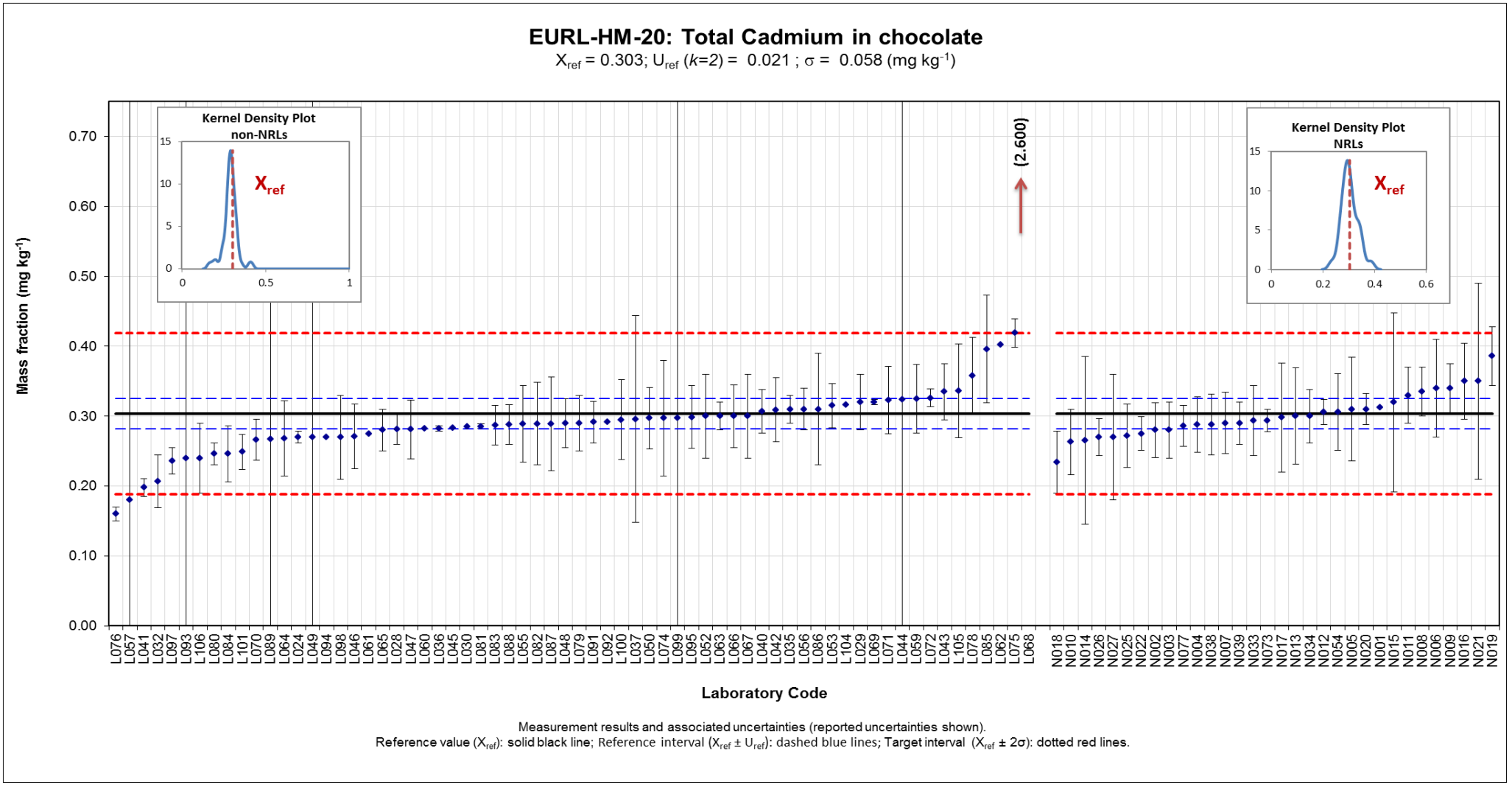
Annex 13: Results for total Cd

Assigned range: X_{ref} = 0.303; U_{ref} (k=2) = 0.021; σ = 0.058 (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]
N001	0.313			AAS	0	0.17	0.89	b
N002	0.28	0.039	2	AAS	0.0195	-0.41	-1.05	a
N003	0.28	0.04	2	ICP-MS	0.02	-0.41	-1.03	a
N004	0.288	0.04	2	ICP-MS	0.02	-0.27	-0.68	a
N005	0.31	0.074	2	ICP-MS	0.037	0.11	0.17	a
N006	0.34	0.07	2	ICP-MS	0.035	0.63	1	a
N007	0.29	0.044	2	ICP-MS	0.022	-0.23	-0.55	a
N008	0.335	0.035	2	ICP-MS	0.0175	0.55	1.54	a
N009	0.34	0.035	2	AAS	0.0175	0.63	1.78	a
N010	0.263	0.047	2	GF-AAS	0.0235	-0.70	-1.57	a
N011	0.33	0.04	2	ICP-MS	0.02	0.46	1.17	a
N012	0.306	0.018	2	ICP-MS	0.009	0.04	0.18	b
N013	0.3	0.069	2	ICP-MS	0.0345	-0.06	-0.1	a
N014	0.265	0.12	2	ET-AAS	0.06	-0.67	-0.63	c
N015	0.32	0.128	2	ICP-MS	0.064	0.29	0.26	c
N016	0.35	0.054	2	ICP-MS	0.027	0.81	1.6	a
N017	0.298	0.078	2	ICP-MS	0.039	-0.09	-0.13	a
N018	0.2344	0.0445	v3	ICP-MS	0.0257	-1.2	-2.48	a
N019	0.386	0.042	2	ETAAS	0.021	1.43	3.5	a
N020	0.31	0.022	2	ICP-MS	0.011	0.11	0.43	a
N021	0.35	0.14	2	ICP-MS	0.07	0.81	0.66	c
N022	0.275	0.024	2	ICP-MS	0.012	-0.49	-1.77	a
N025	0.272	0.045	2	AAS	0.0225	-0.55	-1.26	a
N026	0.27	0.0265	2	ICP-MS	0.0132	-0.58	-1.96	a
N027	0.27	0.09	2	ICP-MS	0.045	-0.58	-0.72	a
N033	0.294	0.05	2	ICP-MS	0.025	-0.16	-0.35	a
N034	0.3	0.038	2	AAS	0.019	-0.06	-0.16	a
N038	0.288	0.043	2	ICP-MS	0.0215	-0.27	-0.64	a
N039	0.29	0.03	2	ICP-MS	0.015	-0.23	-0.73	a
N054	0.306	0.055	2	AAS	0.0275	0.04	0.09	a
N073	0.294	0.016	2	ICP-MS	0.008	-0.16	-0.71	b
N077	0.286	0.029	2	ICP-MS	0.0145	-0.30	-0.97	a
L024	0.27	0.0089	v3	GFAAS	0.0051	-0.58	-2.81	b
L028	0.281	0.021	2	AAS	0.0105	-0.39	-1.49	b
L029	0.32	0.04	2	ICP-MS	0.02	0.29	0.73	a
L030	0.285			ICP-MS	0	-0.32	-1.72	b
L032	0.207	0.038	v3	ICP-OES	0.0219	-1.67	-3.95	a
L035	0.31	0.02	2	ICP-MS	0.01	0.11	0.45	b
L036	0.2822	0.0036	2	ICP-OES	0.0018	-0.37	-1.95	b
L037	0.296	0.148	2	ICP-MS	0.074	-0.13	-0.1	c
L040	0.307	0.031	v3	ICP-MS	0.0179	0.06	0.17	a
L041	0.198	0.013	2	GF-AAS	0.0065	-1.83	-8.4	b
L042	0.309	0.046	2	ICP-MS	0.023	0.1	0.22	a
L043	0.335	0.04	v3	ICP-MS	0.0231	0.55	1.24	a
L044	0.324	20	2	ICP-MS	10	0.36	0.00	c
L045	0.283			GFAAS	0	-0.35	-1.9	b
L046	0.271	0.046	2	ICP-MS	0.023	-0.56	-1.28	a
L047	0.281	0.042	2	ET-AAS	0.021	-0.39	-0.95	a
L048	0.29	0.035	2	AAS	0.0175	-0.23	-0.66	a
L049	0.27	7	3.5	ICP-MS	2	-0.58	-0.02	c
L050	0.297	0.044	2	ICP-MS	0.022	-0.11	-0.26	a
L051	< 0.5000		v3	ICP-OES				
L052	0.3	0.06	0.12	GF-AAS	0.5	-0.06	-0.01	c
L053	0.315	0.032	2	AAS	0.016	0.2	0.6	a
L055	0.289	0.055	2	ICP-OES	0.0275	-0.25	-0.49	a
L056	0.31	0.03	v3	ICP-AES	0.0173	0.11	0.32	a
L057	0.18	17	2	AAS-GTA	8.5	-2.14	-0.01	c
L059	0.325	0.049	2	ICP-MS	0.0245	0.37	0.81	a
L060	0.282			ICP-MS	0	-0.37	-2	b
L061	0.275			ICP-MS	0	-0.49	-2.65	b
L062	0.402			CV-AAS	0	1.71	9.18	b
L063	0.3	0.02	2	AAS	0.01	-0.06	-0.24	b
L064	0.268	0.054	2	ET-AAS	0.027	-0.61	-1.22	a
L065	0.28	0.03	v3	ICP-MS	0.0173	-0.41	-1.15	a
L066	0.3	0.045	2	ICP-MS	0.0225	-0.06	-0.14	a
L067	0.3	0.06	2	ICP-MS	0.03	-0.06	-0.11	a
L068	2.6	0.3	v3	ICP-MS	0.1732	39.83	13.23	c
L069	0.32	0.004	v3	ICP-MS	0.0023	0.29	1.51	b
L070	0.2664	0.0293	2	GF-AAS	0.0146	-0.64	-2.04	a
L071	0.323	0.048	2	SEM-ICP-MS	0.024	0.34	0.74	a
L072	0.326	0.0126	2	ICP-MS	0.0063	0.39	1.81	b
L074	0.297	0.083	2	ICP-MS	0.0415	-0.11	-0.15	a
L075	0.419	0.02	2	ET-AAS	0.01	2	7.87	b
L076	0.16	0.01	2	ICP-OES	0.005	-2.49	-12.11	b
L078	0.358	0.055	2	AAS	0.0275	0.95	1.85	a
L079	0.29	0.04	2	AAS	0.02	-0.23	-0.59	a
L080	0.246	0.016	2	ICP-MS	0.008	-1	-4.29	b
L081	0.2853	0.0034	1.96	ICP-MS	0.0017	-0.31	-1.67	b
L082	0.289	0.059	2	FAAS	0.0295	-0.25	-0.46	a
L083	0.287	0.028	2	ICP-MS	0.014	-0.29	-0.93	a
L084	0.246	0.04	2	AAS	0.02	-1.00	-2.53	a
L085	0.396	0.077	2	AAS	0.0385	1.61	2.32	a
L086	0.31	0.08	2	ICP-MS	0.04	0.11	0.16	a
L087	0.289	0.067	2	ICP-MS	0.0335	-0.25	-0.41	a
L088	0.288	0.0288	2	ICP-MS	0.0144	-0.27	-0.86	a
L089	0.2676	0.282	2	ICP-MS	0.141	-0.62	-0.25	c
L091	0.2917	0.0297	v3	ICP-MS	0.0171	-0.2	-0.58	a
L092	0.292			ICP-MS	0	-0.2	-1.07	b
L093	0.24	0.6	2	FAAS	0.3	-1.1	-0.21	c
L094	0.27			ICP-MS	0	-0.58	-3.11	b
L095	0.2986	0.0448	2	ICP-MS	0.0224	-0.08	-0.2	a
L097	0.236	0.019	2	GF-AAS	0.0095	-1.17	-4.7	b
L098	0.27	0.06	2	AAS	0.03	-0.58	-1.05	a
L099	0.297	25	100	AAS	0.25	-0.11	-0.03	c
L100	0.295	0.057	2	ICP-OES	0.0285	-0.15	-0.28	a
L101	0.249	0.025	2	ICP-AES	0.0125	-0.94	-3.3	a
L104	0.316			ICP-MS	0	0.22	1.17	b
L105	0.336	0.067	2	ICP-MS	0.0335	0.56	0.93	a
L106	0.24	0.05	2	ICP-MS	0.025	-1.10	-2.33	a

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

[b] performance: satisfactory, questionable, unsatisfactory, [c] a : $u_{min}(u_{ref}) \leq u_{lab} \leq u_{max}(\sigma)$; b : $u_{lab} < u_{min}$; and c : $u_{lab} > u_{max}$



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

Annex 14: Results for total Pb

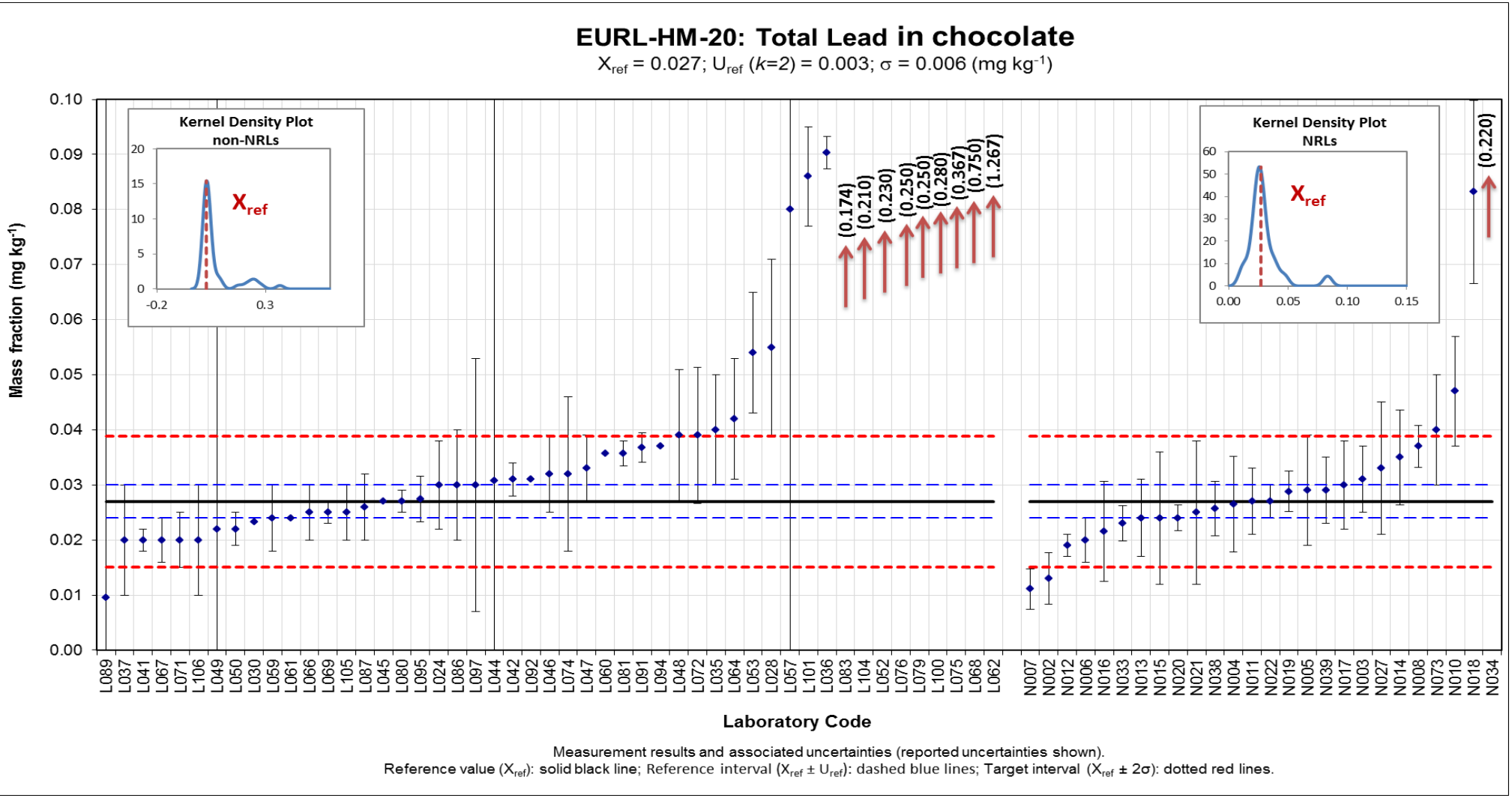
Assigned range: X_{ref} = 0.027; U_{ref} (*k*=2) = 0.003; σ = 0.006 (all values in mg kg^{-1})

Lab code	X_{lab}	U_{lab}	K[a]	technique	u_{lab}	z-score[b]	ζ-score[b]	Uncert.[c]
N001	< 0.0500		v3	AAS				
N002	0.013	0.0047	2	AAS	0.0024	-2.36	-4.81	a
N003	0.031	0.006	2	ICP-MS	0.0030	0.67	1.15	a
N004	0.0265	0.0087	2	ICP-MS	0.0043	-0.09	-0.11	a
N005	0.029	0.01	2	ICP-MS	0.0050	0.33	0.37	a
N006	0.02	0.004	2	ICP-MS	0.0020	-1.18	-2.66	a
N007	0.0111	0.0037	2	ICP-MS	0.0019	-2.68	-6.30	a
N008	0.037	0.0038	2	ICP-MS	0.0019	1.68	3.89	a
N009	< 0.0200			AAS				
N010	0.047	0.01	2	GF-AAS	0.0050	3.36	3.78	a
N011	0.027	0.006	2	ICP-MS	0.0030	0.00	-0.01	a
N012	0.019	0.002	2	ICP-MS	0.0010	-1.35	-4.03	b
N013	0.024	0.007	2	ICP-MS	0.0035	-0.51	-0.77	a
N014	0.035	0.0086	2	ET-AAS	0.0043	1.34	1.72	a
N015	0.024	0.012	2	ICP-MS	0.0060	-0.51	-0.48	c
N016	0.0216	0.0091	2	ICP-MS	0.0046	-0.91	-1.11	a
N017	0.03	0.008	2	ICP-MS	0.0040	0.50	0.68	a
N018	0.0832	0.0166	v3	ICP-MS	0.0096	9.45	5.76	c
N019	0.0288	0.0037	2	ETAAS	0.0019	0.30	0.70	a
N020	0.024	0.0023	2	ICP-MS	0.0011	-0.51	-1.46	b
N021	0.025	0.013	2	ICP-MS	0.0065	-0.34	-0.30	c
N022	0.027	0.003	2	ICP-MS	0.0015	0.00	-0.01	b
N025	< 0.0500			AAS				
N026	< 0.1200			ICP-MS				
N027	0.033	0.012	2	ICP-MS	0.0060	1.01	0.96	c
N033	0.023	0.0032	2	ICP-MS	0.0016	-0.68	-1.71	b
N034	0.22	0.07	2	AAS	0.0350	32.46	5.51	c
N038	0.0257	0.0049	2	ICP-MS	0.0024	-0.22	-0.44	a
N039	0.029	0.006	2	ICP-MS	0.0030	0.33	0.57	a
N054	< 0.5000			AAS				
N073	0.04	0.01	2	ICP-MS	0.0050	2.18	2.45	a
N077	< 0.3000			ICP-MS				
L024	0.03	0.008	v3	GFAAS	0.0046	0.50	0.60	a
L028	0.055	0.016	2	AAS	0.0080	4.71	3.42	c
L029	< 0.0400			ICP-MS				
L030	0.0233	0		ICP-MS	0	-0.63	-2.16	b
L032	< 0.2000			ICP-OES				
L035	0.04	0.01	2	ICP-MS	0.0050	2.18	2.45	a
L036	0.0903	0.0029	2	ICP-OES	0.0015	10.64	28.01	b
L037	0.02	0.01	2	ICP-MS	0.0050	-1.18	-1.33	a
L040	< 0.0500			ICP-MS				
L041	0.02	0.002	2	GF-AAS	0.0010	-1.18	-3.53	b
L042	0.031	0.003	2	ICP-MS	0.0015	0.67	1.74	b
L044	0.0308	20	2	ICP-MS	10.0000	0.64	0.00	c
L045	0.027	0	v3	GFAAS	0	0.00	-0.01	b
L046	0.032	0.007	2	ICP-MS	0.0035	0.84	1.28	a
L047	0.033	0.006	2	ET-AAS	0.0030	1.01	1.73	a
L048	0.039	0.012	2	AAS	0.0060	2.01	1.92	c
L049	0.022	0.51	0.26	ICP-MS	1.9615	-0.84	0.00	c
L050	0.022	0.003	2	ICP-MS	0.0015	-0.84	-2.20	b
L051	< 1.0000			ICP-OES				
L052	0.23	0.01	0.02	GF-AAS	0.5000	34.14	0.41	c
L053	0.054	0.011	2	AAS	0.0055	4.54	4.68	a
L055	< 0.5000			ICP-OES				
L056	< 0.3000			ICP-AES				
L057	0.08	15	2	GF-AAS	7.5000	8.91	0.01	c
L059	0.024	0.006	2	ICP-MS	0.0030	-0.51	-0.87	a
L060	0.0357	0	v3	ICP-MS	0	1.46	5.04	b
L061	0.024	0	v3	ICP-MS	0	-0.51	-1.76	b
L062	1.267	0	v3	CV-AAS	0	208.58	720.85	b
L063	< 0.0500			AAS				
L064	0.042	0.011	2	ET AAS	0.0055	2.52	2.60	a
L065	< 0.0500			ICP-MS				
L066	0.025	0.005	2	ICP-MS	0.0025	-0.34	-0.67	a
L067	0.02	0.004	2	ICP-MS	0.0020	-1.18	-2.66	a
L068	0.75	0.08	v3	ICP-MS	0.0462	121.61	15.64	c
L069	0.025	0.002	v3	ICP-MS	0.0012	-0.34	-0.98	b
L070	< 0.0500			GF-AAS				
L071	0.02	0.005	2	SEM-ICP-MS	0.0025	-1.18	-2.31	a
L072	0.039	0.0124	2	ICP-MS	0.0062	2.01	1.86	c
L074	0.032	0.014	2	ICP-MS	0.0070	0.84	0.69	c
L075	0.367	0.03	2	EET-AAS	0.0150	57.19	22.52	c
L076	0.25	0.01	2	ICP-AES	0.0050	37.51	42.17	a
L078	< 0.1000			AAS				
L079	0.25	0.06	2	AAS	0.0300	37.51	7.42	c
L080	0.027	0.002	2	ICP-MS	0.0010	0.00	-0.01	b
L081	0.0357	0.0023	1.96	AAS	0.0012	1.46	4.17	b
L083	0.174	0.017	2	ICP-MS	0.0085	24.72	16.95	c
L084	< 0.1000			AAS				
L085	< 0.1200			AAS				
L086	0.03	0.01	2	ICP-MS	0.0050	0.50	0.56	a
L087	0.026	0.006	2	ICP-MS	0.0030	-0.17	-0.30	a
L088	< 0.1000			ICP-MS				
L089	0.0096	0.0924	2	ICP-MS	0.0462	-2.93	-0.38	c
L091	0.0368	0.0027	v3	ICP-MS	0.0016	1.64	4.21	b
L092	0.031	0	v3	ICP-MS	0	0.67	2.31	b
L094	0.037	0	v3	ICP-MS	0	1.68	5.80	b
L095	0.0274	0.0041	2	ICP-MS	0.0021	0.07	0.15	a
L097	0.03	0.023	2	GF-AAS	0.0115	0.50	0.26	c
L098	< 0.0800			AAS				
L099	< 0.0500		100	AAS				
L100	0.28	0.055	2	ICP-OES	0.0275	42.55	9.18	c
L101	0.086	0.009	2	ICP-AES	0.0045	9.92	12.24	a
L102			v3					
L104	0.21	0	v3	ICP-MS	0	30.78	106.37	b
L105	0.025	0.005	2	ICP-MS	0.0025	-0.34	-0.67	a
L106	0.02	0.01	2	ICP-MS	0.0050	-1.18	-1.33	a

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

[b] performance: satisfactory, questionable, unsatisfactory,

[c] a : $u_{min}(u_{ref}) \leq u_{lab} \leq u_{max}(\sigma)$; b : $u_{lab} < u_{min}$; and c : $u_{lab} > u_{max}$



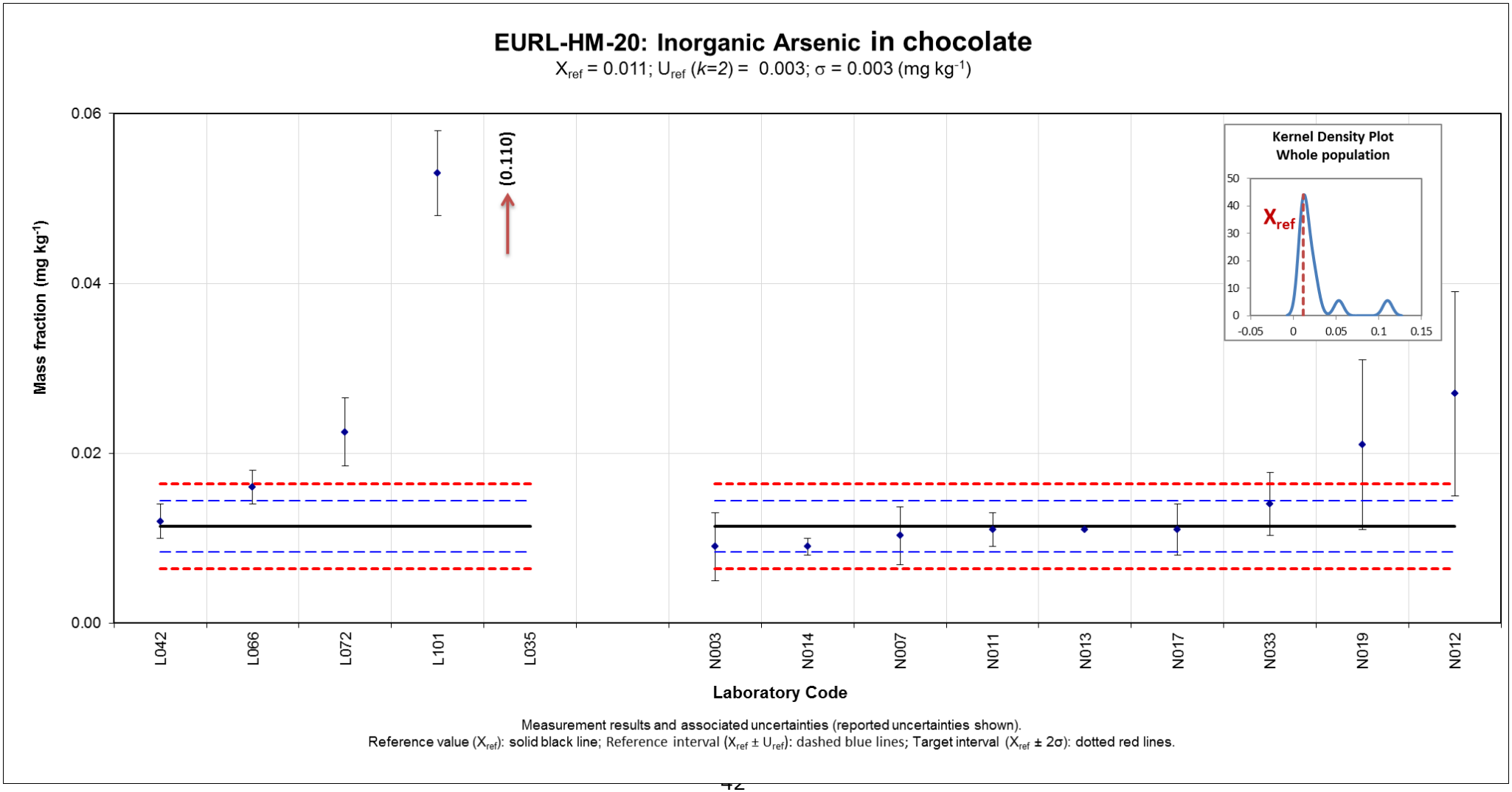
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (X_{ref}): solid black line; Reference interval ($X_{ref} \pm U_{ref}$): dashed blue lines; Target interval ($X_{ref} \pm 2\sigma$): dotted red lines.

Annex 15: Results for inorganic arsenic, iAs

Assigned range: X_{ref}= 0.011; U_{ref} (k=2) = 0.004; σ = 0.003
(all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k	technique	u_{lab}	z-score	ζ-score	uncert.
N001	<0.2			LC-ICP-MS				
N003	0.009	0.004	2	HPLC-ICP-MS	0.002	-0.84	-0.96	a
N004	<0.05							
N007	0.0103	0.0034	2	HPLC-ICP-MS	0.002	-0.39	-0.48	a
N011	0.011	0.002	2	HPLC-ICP-MS	0.001	-0.14	-0.22	b
N012	0.027	0.012	2	HPLC-ICP-MS	0.006	5.47	2.52	c
N013	0.011			LC-ICP-MS	0	-0.14	-0.26	b
N014	0.009	0.001	2	HG-AAS	0.001	-0.84	-1.50	b
N016	<0.025			LC-ICP-MS				
N017	0.011	0.003	2	LC-ICP-MS	0.002	-0.14	-0.19	b
N019	0.021	0.01	2	HG-AAS	0.005	3.37	1.84	c
N020	<0.0084			LC-ICP-MS				
N025	<0.065			HG-AAS				
N027	<0.020			HPLC-ICP-MS				
N033	0.014	0.0037	2	ICP-MS	0.002	0.91	1.09	a
N077	<0.035			LC-ICP-MS				
L029	<0.05			ICP-MS				
L031	<0.1			HPLC-ICP-MS				
L032	<0.1			ICP-OES				
L035	0.110	0.03	2	HPLC-ICP-MS	0.015	34.60	6.54	c
L042	0.012	0.002	2	HPLC-ICP-MS	0.001	0.21	0.33	b
L051	<3.3			ICP-OES				
L066	0.016	0.002	2	LC-ICP-MS	0.001	1.61	2.53	b
L072	0.023	0.004	2	LC-ICP-MS	0.002	3.89	4.42	a
L081	<0.05			AAS				
L101	0.053	0.005	2	AFS	0.003	14.60	14.22	a
L102	<0.1			HG-AAS				

[a] √3 is set by the ILC coordinator when no expansion factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k*=√3,
[b]performance: satisfactory, questionable, unsatisfactory,
[c]a : $u_{min}(u_{ref}) \leq u_{lab} \leq u_{max}(\sigma)$; b : $u_{lab}<u_{min}$; and c : $u_{lab}>u_{max}$


EURL-HM-20: Inorganic Arsenic in chocolate
Xref = 0.011; Uref (k=2) = 0.003; σ = 0.003 (mg kg-1)
Mass fraction (mg kg-1)
0.06
0.04
0.02
0.00
(0.110)
Kernel Density Plot
Whole population
Xref
50
40
30
20
10
0
-0.05
0
0.05
0.1
0.15
L042
L066
L072
L101
L035
N003
N014
N007
N011
N013
N017
N033
N019
N012
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.

Annex 16: Results for total Hg

Lab Code	X_{lab}	U_{lab}	k	technique
N001	0.005			CV-AFS
N002	0.08	0.018	2	HG-AAS
N003	0.02	0.002	2	ICP-MS
N004	<0.005			CV-AAS
N005	0.0013	0.00031	2	AAS
N006	<0.006			FIMS
N007	<0.0075			ICP-MS
N008	<0.02			CV-AAS
N009	<0.1			CV-AAS
N011	0.0012	0.0003	2	Direct Mercury Analysis
N012	0.0005	0.0002	2	Direct mercury analysis
N013	<0.01			Autoanalyser
N014	<0.05			CV-AAS
N015	0.007	0.0028	2	ICP-MS
N016	<0.004			CV-AFS
N017	<0.04			ICP-IDMS
N018	0.00114	0.000228		AAS
N019	0.0011	0.0005	2	Mercury Analyser, AMA 254
N020	<0.001			ICP-MS
N021	0.013	0.005	2	ICP-MS
N022	<0.006			ICP-MS
N025	0.002	0.0003	2	CV-AAS
N026	<0.05			ICP-MS
N027	0.103	0.031	2	thermal decomposition-amalgamation-AAS (AMA 254)
N033	<0.001			ICP-MS
N034	<0.05			CV-AAS
N038	0.0101	0.0026	2	ICP-MS
N054	<0.1			CV-AAS
N073	<0.02			ICP-MS
N077	0.002	0.001	2	direct mercury analysis
.				
L024	0.001	0.0001		AAS
L029	<0.005			ICP-MS
L030	<0.002			ICP-MS
L032	<0.05			ICP-OES
L035	<0.03			ICP-MS
L036	<0.01			ICP-OES
L037	<0.017			ICP-MS
L040	<0.02			ICP-MS
L042	0.012	0.001	2	ICP-MS
L043	<0.01			CV-AAS
L044	0.00792	20	2	ICP-MS
L045	<0.02			CV-AAS
L047	0.029	0.005	2	FIAS
L049	<0.02	2.33	1.16	ICP-MS
L050	<0.003			FIMS
L051	<2.5			ICP-OES
L052	0.03	0.003	0.006	CV-AAS
L053	0.0177	0.002	2	AAS
L055	<0.5			ICP-OES
L057	<0.05			CV-AAS
L059	0.03	0.011	2	ICP-MS
L060	<0.01			ICP-MS
L061	<0.05			ICP-MS
L063	<0.05			CV-AAS
L065	0.0018	0.0002		CV-AAS
L066	<0.004			ICP-MS
L067	0.003	0.0002	2	ICP-MS
L068	0.0058	0.0006		CV-AAS
L069	0.004			ICP-MS
L070	<0.0004			Advanced mercury atomizer AMA 254
L072	<0.02			ICP-MS
L074	0.005	0.004	2	ICP-MS
L075	<0.2			CV-AAS
L076	0.0025	0.0001	2	DMA-80 Millestone
L078	<0.008			H-AAS
L079	<0.05			CV-AAS
L080	0.0046	0.0005	2	ICP-MS
L081	<0.005			HG-AAS
L083	0.1	0.02	2	ICP-MS
L084	<0.0007			CV-AAS
L086	0.011	0.002	2	ICP-MS
L087	0.003	0.001	2	ICP-MS
L088	0.001	0.00024	2	Atomic absorption spectroscopy – AMA 254
L089	<0.0043			ICP-MS
L091	0.0035	0.0003		ICP-MS
L092	0.005			ICP-MS
L093	<0.004			DMA
L095	0.01255	0.00188	2	ICP-MS
L097	0.025	0.018	2	CV-AAS
L098	<0.05			HG-AAS
L099	<0.001	13	100	AAS
L100	0.029	0.007	2	ICP-OES
L101	0.011	0.001	2	LECO AMA
L102	0.13	0.03	2	CV-AAS
L104	<0.01			ICP-MS
L105	0.015	0.003	2	ICP-MS
L106	0			ICP-MS

Annex 17: Experimental details

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
L024	As			HRN EN 14084:2005, EPA method 7473	X	X		not analysed			
L024	Cd		Merck 1.19777		Closed microwave	H2O2, HNO3			93.2	0.001	GFAAS
L024	Hg		Fluka 16482		Mercury analyzer	X				0.0001	AAS
L024	iAs				X	X					
L024	Pb		Merck 1.19776		Closed microwave	H2O2, HNO3			75.4	0.01	GFAAS
L028	As			true	X	X	500-800W/30min				
L028	Cd				Closed microwave	H2O2, HNO3			100		AAS
L028	Hg				X	X					
L028	iAs				X	X					
L028	Pb				Closed microwave	H2O2, HNO3			100		AAS
L029	As			No	Dry ashing	HNO3	90			0.015	ICP-MS
L029	Cd									0.01	ICP-MS
L029	Hg									0.0025	ICP-MS
L029	iAs									0.025	ICP-MS
L029	Pb									0.02	ICP-MS
L030	As			US EPA 200.8	Ultraclave	HNO3, HF	200ºC, 25 min.				ICP-MS
L030	Cd										ICP-MS
L030	Hg										ICP-MS
L030	iAs				X	X					
L030	Pb				Ultraclave	HNO3, HF					ICP-MS
L031	As			No	X	X	1 hour at 60 then 2 hours at 80.	Arsenic species are extracted with TMAOH, neutralised, centrifuged separated by IC determined by ICP-MS.			
L031	Cd				X	X					
L031	Hg				X	X					
L031	iAs	No	No		TMAOH	25% TMAOH aq			106	0.03	HPLC-ICP-MS
L031	Pb				X	X					
L032	As										ICP-OES
L032	Cd										ICP-OES
L032	Hg										ICP-OES
L032	iAs										ICP-OES
L032	Pb										ICP-OES
L035	As			EN 15763:2009; EN 13805:2002; EPA Method 6020A:2007; EN 13804:2013	Closed microwave	H2O2, HNO3, HCl	for total element concentration: 20 min up to 250 psi, 15 min hold at 250 psi	Extraction with diluted (1%) nitric acid and H2O2 (3%), HPLC-ICP-MS analysis with a SAX column, pH 8.9, (NH4)2CO3 buffer as eluent			ICP-MS
L035	Cd										ICP-MS
L035	Hg										ICP-MS
L035	iAs	ERM-BC211			closed vessel, 95 C degree, 1 hour	H2O2, HNO3			97	0.02	HPLC-ICP-MS
L035	Pb				Closed microwave	H2O2, HNO3, HCl					ICP-MS
L036	As	IRMM	JT Baker	EPA 6010C, EPA 3052	Closed microwave	H2O2, HNO3	180 degrees of Celsius for half an hour		107	0.001	ICP-OES
L036	Cd								97	0.001	ICP-OES
L036	Hg								87	0.001	ICP-OES
L036	iAs				X	X					
L036	Pb	IRMM	JT Baker		Closed microwave	H2O2, HNO3			89	0.001	ICP-OES
L037	As			method ANSES Cime 8 and 12	Pressure bomb	HNO3	100°C			0.002	ICP-MS
L037	Cd									0.002	ICP-MS
L037	Hg									0.002	ICP-MS
L037	iAs				X	X					
L037	Pb				Pressure bomb	HNO3				0.002	ICP-MS
L040	As			EN ISO 15763	Pressure bomb	HNO3	240°C/1h			0.001	ICP-MS
L040	Cd									0.0004	ICP-MS
L040	Hg									0.01	ICP-MS
L040	iAs				X	X					

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
L040	Pb				Pressure bomb	HNO3				0.0025	ICP-MS
L041	As	Atomic Absorption standar	Atomic Absorption standard	No	Closed microwave	H2O2, HNO3	200 Celsius degrees/30 min	-	66.48	0.028	GF-AAS
L041	Cd							-	76.03	0.01	GF-AAS
L041	Hg				X	X		-			
L041	iAs				X	X		-			
L041	Pb	Atomic Absorption standar	Atomic Absorption standar		Closed microwave	H2O2, HNO3		-	70.14	0.005	GF-AAS
L042	As	NCS ZC73013		NF EN 17852 and PR NF EN 16802	Closed microwave	HNO3	20 min at 200°C	PR NF EN 16802	103	0.01	ICP-MS
L042	Cd								94	0.01	ICP-MS
L042	Hg								104	0.01	ICP-MS
L042	iAs					H2O2, HNO3			60	0.01	HPLC-ICP-MS
L042	Pb	NCS ZC7301				HNO3			82	0.02	ICP-MS
L043	As										
L043	Cd										ICP-MS
L043	Hg										CV-AAS
L043	iAs										
L043	Pb										
L044	As			In-house developed and validated method	Closed microwave	H2O2, HNO3	250/1hr	N/A	111	0.00001	ICP-MS
L044	Cd								102	0.000004	ICP-MS
L044	Hg								96	0.000003	ICP-MS
L044	iAs					Followed by addition of HCl (post-digestion)					
L044	Pb				Closed microwave	H2O2, HNO3			96	0.000005	ICP-MS
L045	As			Yes	Closed microwave	H2O2, HNO3	200/30 min				H-AAS
L045	Cd										GFAAS
L045	Hg										CV-AAS
L045	iAs				X	X					
L045	Pb				Closed microwave	H2O2, HNO3					GFAAS
L046	As			SRPS EN 13805:2008	Closed microwave	H2O2, HNO3	Temperature 210 degree Celsius/25 minutes		91.5	0.01	ICP-MS
L046	Cd								100.5	0.01	ICP-MS
L046	Hg				X	X					
L046	iAs				X	X					
L046	Pb				Closed microwave	H2O2, HNO3			100.2	0.015	ICP-MS
L047	As			No	Closed microwave	H2O2, HNO3	180ºC/120min		80	0.005	FIAS
L047	Cd								92	0.005	ET-AAS
L047	Hg								80	0.005	FIAS
L047	iAs				X	X			/	/	
L047	Pb				Closed microwave	H2O2, HNO3			88	0.01	ET-AAS
L048	As			No	X	X	200oC/15min				
L048	Cd		FAPAS T07143		Closed microwave	H2O2, HNO3				0.001	AAS
L048	Hg				X	X					
L048	iAs				X	X					
L048	Pb		FAPAS T07143		Closed microwave	H2O2, HNO3				0.006	AAS
L049	As	NA	NA	No	Hot Acid Digestion	HNO3, HF	100 degrees celsius for 1 Hour	NA	NA	<0.04	ICP-MS
L049	Cd								NA	<0.002	ICP-MS
L049	Hg								NA	<0.02	ICP-MS
L049	iAs				X	X			NA	NA	
L049	Pb				Hot Acid Digestion	HNO3, HF			NA	<0.02	ICP-MS
L050	As	DOLT4, GBW		NS-EN 17294-2 (basis), NS-EN 1483 (basis)	Closed microwave, Pressure bomb	H2O2, HNO3	170 °C			0.05	ICP-MS
L050	Cd									0.005	ICP-MS
L050	Hg									0.003	FIMS
L050	iAs				X	X					

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
L050	Pb	DOLT4, GBW			Closed microwave, Pressure bomb	H2O2, HNO3				0.01	ICP-MS
L051	As	GB CRM	GB CRM	No	Closed microwave	HNO3	175Celsius/40min	By calculation according to As content	99	2.5	ICP-OES
L051	Cd								94	0.5	ICP-OES
L051	Hg								96	2.5	ICP-OES
L051	iAs								99	3.3	ICP-OES
L051	Pb								101	1	ICP-OES
L052	As			No	Closed microwave	HNO3	120 degree celcious		105	0.02	HG-AAS
L052	Cd								107	0.01	GF-AAS
L052	Hg								101	0.03	CV-AAS
L052	iAs				X	X					
L052	Pb				Closed microwave	HNO3			98.4	0.05	GF-AAS
L053	As			No	Pressure bomb	HNO3	185/6		100	0.05	HG-AAS
L053	Cd								100	0.005	AAS
L053	Hg				X	X			100	0.0002	AAS
L053	iAs				X	X					
L053	Pb				Pressure bomb	HNO3			100	0.03	AAS
L055	As			No	Closed microwave	HNO3	255°C/45min.		none	1	ICP-OES
L055	Cd								none	0.05	ICP-OES
L055	Hg								none	0.5	ICP-OES
L055	iAs				X	X					
L055	Pb				Closed microwave	HNO3			none	0.5	ICP-OES
L056	As		Panreac 313171.1208	No	Digestion with Magnesium Nitrate, Magnesium Oxide and Acid Nitric	HNO3, Magnesium Nitrate and Magnesium Oxide	For Cd and Pb 25 min at 190ºC; For As 48 h at 450ºC	none	1	0.24	HG-AAS
L056	Cd		VHG-ACDN-100	b) No	Closed microwave	H2O2, HNO3			1.02	0.02	ICP-AES
L056	Hg			b) No	X	X					
L056	iAs			b) No	X	X					
L056	Pb		VHG-APBN-100	b) No	Closed microwave	H2O2, HNO3			0.97	0.3	ICP-AES
L057	As		NIST Traceable spectrosol	AOAC 986.15 (2012),AOAC 999.10(2012)	Closed microwave, Dry ashing	1. H2O2, 4. HNO3	172 0C for 20 minutes-microwave digestion and 500 0C for 3 hours-dry ashing	Not analysed			atomic absorption spectrophotometry-vapour generation accessory(VGA)
L057	Cd				Closed microwave	H2O2, HNO3					atomic absorption spectrophotometry-GTA
L057	Hg										CV-AAS
L057	iAs				X	X					
L057	Pb		NIST Traceable spectrosol		Closed microwave	H2O2, HNO3					atomic absorption spectrophotometry-GTA
L059	As			No	Pressure bomb	HNO3	180 °C/60 minutes	not analysed	25	0.009	ICP-MS
L059	Cd								25	0.002	ICP-MS
L059	Hg								35	0.007	ICP-MS
L059	iAs					Other					
L059	Pb				Pressure bomb	HNO3			25	0.005	ICP-MS
L060	As		Yes	a) Yes	1. Closed microwave	4. HNO3	20 min Ramp to 200'c Hold 20 min			0.01	ICP-MS
L060	Cd		Yes	a) Yes	1. Closed microwave	4. HNO3	20 min Ramp to 200'c Hold 20 min			0.01	ICP-MS
L060	Hg		Yes	a) Yes	1. Closed microwave	4. HNO3	20 min Ramp to 200'c Hold 20 min			0.01	ICP-MS
L060	iAs			a) Yes		X	20 min Ramp to 200'c Hold 20 min				
L060	Pb		Yes	a) Yes	1. Closed microwave	4. HNO3	20 min Ramp to 200'c Hold 20 min			0.005	ICP-MS

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
L061	As										ICP-MS
L061	Cd										ICP-MS
L061	Hg										ICP-MS
L061	iAs										
L061	Pb										ICP-MS
L062	As				X	X					
L062	Cd				Dry ashing	HNO3, HCl			100	0.075	CV-AAS
L062	Hg			No	X	X	450°C 16hours				
L062	iAs				X	X					
L062	Pb				Dry ashing	HNO3, HCl			100	0.5	CV-AAS
L063	As	BCR-186		No	Closed microwave	H2O2, HNO3	200ºC/10 minutes	not tested	100	0.1	AAS
L063	Cd								103	0.05	AAS
L063	Hg								114	0.05	CV-AAS
L063	iAs				X	X					
L063	Pb	BCR-186			Closed microwave	H2O2, HNO3			101	0.05	AAS
L064	As			EN 14084	X	X					
L064	Cd	CRM	CRM		Closed microwave	HNO3			90	0.01	Electrothermal AAS
L064	Hg				X	X					
L064	iAs				X	X					
L064	Pb	CRM	CRM		Closed microwave	HNO3			85	0.02	Electrothermal AAS
L065	As										ICP-MS
L065	Cd										ICP-MS
L065	Hg										CV-AAS
L065	iAs										
L065	Pb										ICP-MS
L066	As	YES	YES	No	Closed microwave	H2O2, HNO3	200ºC/20 min for digestion of As, Cd, Pb, Hg. And up to 95 ºC/20 min for iAs	We have used 2 different methods with same result. Same microwave extraction for both (0,25 g of sample + 10 mL of a mix H2O2/HNO3 0,1M). Measuring with LC-ICP-MS and SPE-ICP-MS too.	100	0.004	ICP-MS
L066	Cd	YES	YES						100	0.002	ICP-MS
L066	Hg	YES	YES			H2O2, HNO3, HCl			100	0.002	ICP-MS
L066	iAs	YES	YES			H2O2, HNO3, diluted nitric acid for iAs instead of concentrated as for As,Cd,Hg,Pb			100	0.002	LC-ICP-MS
L066	Pb	YES	YES			H2O2, HNO3			100	0.004	ICP-MS
L067	As	NCS DC 73349		Yes	Closed microwave	H2O2, HNO3	In total 6 min (4 min ramping + 2 min holding) in 200 degrees.	No determination and results		0.00001	ICP-MS
L067	Cd	INCT-MPH-2								0.000002	ICP-MS
L067	Hg	CRM Dolt 4 Fish Liver				H2O2, HNO3, HCl				3E-07	ICP-MS
L067	iAs				X	X					
L067	Pb	INCT-MPH-2			Closed microwave	H2O2, HNO3				0.00001	ICP-MS
L068	As			No	Pressure bomb	HNO3	200°C, 30 minutes	We measured only total As			ICP-MS
L068	Cd										ICP-MS
L068	Hg										CV-AAS
L068	iAs										
L068	Pb										ICP-MS
L069	As			No	Closed microwave	H2O2, HNO3					ICP-MS
L069	Cd										ICP-MS
L069	Hg										ICP-MS
L069	iAs				X	X					
L069	Pb				Closed microwave	H2O2, HNO3					ICP-MS
L070	As			No	X	X	Cd, Pb - 520 temperature/15 ours, Hg - 550 temperature/ 8 min.				
L070	Cd	Tea, White Cabbege	RM Cd		Dry ashing	HNO3			86.3	0.0002	ET-AAS
L070	Hg	Milk Powder	RM Hg			Dry ashing			97	0.0004	AMA 254
L070	iAs				X	X					

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
L070	Pb	Tea, White Cabbage	RM Pb		Dry ashing	HNO3			99.9	0.01	ET-AAS
L071	As			No	Open acid digestion	H2O2, HNO3	110°C/230min		112.5	0.002	SEM-ICP-MS
L071	Cd								106.3	0.002	SEM-ICP-MS
L071	Hg				X	X					
L071	iAs				X	X					
L071	Pb				Open acid digestion	H2O2, HNO3			99.5	0.005	SEM-ICP-MS
L072	As	Yes		Official test method in Taiwan (TFDA).	Closed microwave	HNO3	100 C 15 min to 160 C 15 min	Sample was cuted into small pieces. Add 10 ml 1% HNO3 into sample tube. Sample was extracted by ultrasonic device and followed by LC-ICP/MS analysis.	105	0.02	ICP-MS
L072	Cd	Yes							102.6	0.02	ICP-MS
L072	Hg	Yes							94.9	0.02	ICP-MS
L072	iAs	Yes			Sample was extracted with 1% HNO3 and analysis by LC-ICP/MS.				114.9	0.02	LC-ICP-MS
L072	Pb	Yes			Closed microwave				93.5	0.02	ICP-MS
L074	As	NIST-1570a	NIST-1570a	AOAC 2013.06	Closed microwave	H2O2, HNO3	Maximum: T=180ºC / Total time: 45min		107	0.01	ICP-MS
L074	Cd								90	0.01	ICP-MS
L074	Hg								82	0.005	ICP-MS
L074	iAs				X	X					
L074	Pb	NIST-1570a	NIST-1570a		Closed microwave	H2O2, HNO3			84	0.01	ICP-MS
L075	As	yes	yes	As: AOAC Ed 19 (2012) 986.15; Cd and Pb: AOAC Ed 19(2012) 999.11; Hg: Chemical Methods Manual for fish and seafoods- Canadian Food Inspection. Agency, Amed 4, 1999	Dry ashing	HNO3	As: 2 h at 150 C, drying at 375 C, 0.5 h at 450 C; Cd and Pb: 4 h at 450 C; Hg: 2 h at 60 C		95	0.001	HG-AAS
L075	Cd	yes	yes			HNO3, HCl			96	0.002	ET-AAS
L075	Hg	yes	yes			HNO3, H2SO4			93	0.003	CV-AAS
L075	iAs				X	X					
L075	Pb	yes	yes		Dry ashing	HNO3, HCl			97	0.015	ET-AAS
L076	As	X		No	Closed microwave	H2O2, HNO3	180ºC/ 10 MINUTES		99.75	0.001	ICP-OES
L076	Cd	X							99.88	0.001	ICP-OES
L076	Hg	X							99.79	0.00005	DMA-80 Millestone
L076	iAs				X	X					
L076	Pb	X			Closed microwave	H2O2, HNO3			99.56	0.001	ICP-AES
L078	As										AAS
L078	Cd										AAS
L078	Hg										H-AAS
L078	iAs										
L078	Pb										AAS
L079	As			No	Closed microwave	H2O2, HNO3	15min untill 180C , 10 min 180C	Not Analysed			AAS
L079	Cd										AAS
L079	Hg										CV-AAS
L079	iAs				X	X					
L079	Pb				Closed microwave	H2O2, HNO3					AAS
L080	As		1000 mg/l As Certipur	EN ISO 15763:2010	Closed microwave	H2O2, HNO3				0.002	ICP-MS
L080	Cd	ERM-BD151	1000 mg/l Cd Certipur							0.0001	ICP-MS
L080	Hg		1000 mg/l Hg Certipur							0.00005	ICP-MS
L080	iAs				X	X					
L080	Pb	ERM-BD151	1000 mg/ Pb Certipur		Closed microwave	H2O2, HNO3				0.0004	ICP-MS
L081	As	many	many	As-DIN EN ISO11969:1996-11 , Cd-	Pressure bomb	H2O2, HNO3	240 °C for 30 min	Hydrid-AAS measurement of acicic extracted sample.		0.01	HG-AAS
L081	Cd	many	many							0.025	ICP-MS

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
L081	Hg	many	many	DIN EN ISO 17294-2:2005-02, Pb-DIN EN ISO 14083:2003-07, Hg-DIN EN ISO 12846:2012-08, iAs-§ 64 LFGB L 15.06-2						0.005	HG-AAS
L081	iAs	rice flour NRI JCRM7503-a	rice flour NRI JCRM7503-a		thermal preparation 95 °C for 90min	HNO3				0.05	AAS
L081	Pb	many	many		Pressure bomb	H2O2, HNO3				0.02	AAS
L082	As			No	Dry ashing	HNO3, HCl	for As: max 400°C / 8 h for Cd: max 450°C / 8 h		105	0.02	HG-AAS
L082	Cd					HNO3			98	0.023	FAAS
L082	Hg				X	X					
L082	iAs				X	X					
L082	Pb				X	X					
L083	As			No	Pressure bomb	H2O2, HNO3					ICP-MS
L083	Cd										ICP-MS
L083	Hg										ICP-MS
L083	iAs				X	X					
L083	Pb				Pressure bomb	H2O2, HNO3					ICP-MS
L084	As	MIXED HERBS INCT-MPH-2		No	wet digestion,	HNO3, HClO4	Pb, Cd- 240°C/45min, Hg-max 850°C/ 5min, As- max 300°C/2days	iAs is no tested in our laboratory	95.9	0.008	HG-AAS
L084	Cd				Closed microwave	HNO3			92.5	0.005	AAS
L084	Hg				automatic mercury analyzer (MA-2000 System)	Al2O3, mixture of NaCO3+Ca(OH)2			97	0.0004	CV-AAS
L084	iAs				X	X					
L084	Pb	MIXED HERBS INCT-MPH-2			Closed microwave	HNO3			102.4	0.05	AAS
L085	As	rice flour	standard solution	FDA	Closed microwave	H2O2, HNO3	200 °C/15 min	digestion closed microwave with H2O2/HNO3 mixture, read by GFAAS	78	0.024	AAS
L085	Cd	peach leaves							68	0.006	AAS
L085	Hg				X	X					
L085	iAs				X	X					
L085	Pb	peach leaves	standard solution		1. Closed microwave	H2O2, HNO3			41	0.028	AAS
L086	As			No	Closed microwave	H2O2, HNO3	200°C/20 min			0.006	ICP-MS
L086	Cd									0.001	ICP-MS
L086	Hg									0.002	ICP-MS
L086	iAs				X	X					
L086	Pb				Closed microwave	H2O2, HNO3				0.003	ICP-MS
L087	As	Yes		ICP-MS 010 in house developed method	Closed microwave	HNO3	180C / 20 minutes	n/a	91.97	0.0004	ICP-MS
L087	Cd	Yes							94.15	0.0002	ICP-MS
L087	Hg	Yes							101.74	0.0002	ICP-MS
L087	iAs	n/a			X	X			n/a	n/a	
L087	Pb	Yes			Closed microwave	HNO3			102.36	0.001	ICP-MS
L088	As	SRM1568b	Titrisol Arsenic standard	No	Dry ashing	As: Mg(NO3)2					HG-AAS
L088	Cd		CertiPUR ICP		Closed microwave	H2O2, HNO3					ICP-MS
L088	Hg	IAEA-V-10 Hay powder0			Untreated sample was directly introduced to the AMA 254.	X					Atomic absorption spectroscopy – Advanced Mercury Analyser 254
L088	iAs				X	X					
L088	Pb	BCR191	CertiPUR ICP		Closed microwave	H2O2, HNO3					ICP-MS
L089	As	AA03N-10X-20ML	AA03N-10X-20ML	NMKL 186	Closed microwave	H2O2, HNO3	145°C /5 min , 190°C 15 min		0.8623	0.0039	ICP-MS
L089	Cd								0.8875	0.004	ICP-MS
L089	Hg								0.9156	0.0043	ICP-MS
L089	iAs				X	X					
L089	Pb	AA29N-10X-20ML	AA29N-10X-20ML		Closed microwave	H2O2, 4. HNO3			0.8079	0.004	ICP-MS
L091	As										ICP-MS

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
L091	Cd										ICP-MS
L091	Hg										ICP-MS
L091	iAs										
L091	Pb										ICP-MS
L092	As			No	Closed microwave	HNO3, HCl	180 degrees Celcius for 15 minutes			0.02	ICP-MS
L092	Cd									0.0024	ICP-MS
L092	Hg									0.0004	ICP-MS
L092	iAs				X	X					
L092	Pb				Closed microwave	HNO3, HCl				0.004	ICP-MS
L093	As			No	X	X	400ºC >24h				
L093	Cd				Dry ashing	X				0.07	FAAS
L093	Hg				DMA	X				0.004	DMA
L093	iAs				X	X					
L093	Pb				X	X					
L094	As										ICP-AES
L094	Cd										ICP-AES
L094	Hg										
L094	iAs										
L094	Pb										ICP-AES
L095	As			internal SOP	Closed microwave	HNO3, HClO4	250°C / 20 min			0.013	ICP-MS
L095	Cd									0.002	ICP-MS
L095	Hg									0.006	ICP-MS
L095	iAs					X				n.a.	
L095	Pb				Closed microwave	HNO3, HClO4				0.003	ICP-MS
L097	As			NMKL and ISO methods	Closed microwave	H2O2, HNO3	180°C/15min; 220°C/10min; 240°C/15min			0.01	HG-AAS
L097	Cd									0.004	GF_AAS
L097	Hg									0.02	CV-AAS
L097	iAs				X	X					
L097	Pb				Closed microwave	H2O2, HNO3				0.08	GF_AAS
L098	As			true	X	X					
L098	Cd				Closed microwave	H2O2, HNO3					AAS
L098	Hg										HG-AAS
L098	iAs				X	X					
L098	Pb				Closed microwave	H2O2, HNO3					AAS
L099	As										AAS
L099	Cd										AAS
L099	Hg										AAS
L099	iAs										
L099	Pb										AAS
L100	As	FAPAS_07190 - Arsenic (to	J/8003/05		Closed microwave	H2O2, HNO3	30-150°C/40min		86.25	0.0076	ICP-OES
L100	Cd								89.5	0.0061	ICP-OES
L100	Hg								84.83	0.0095	ICP-OES
L100	iAs				X	X					ICP-OES
L100	Pb	FAPAS_07190 - Arsenic (to	J/8035/05		Closed microwave	H2O2, HNO3			90.5	0.0074	ICP-OES
L101	As				Arsenic - oxidation and acid digestion	HCl	450 4h	Acid digestion - flurescence spectrocopy.	98.3	0.06	AFS
L101	Cd				Dry ashing	HNO3			88	0.0004	ICP-AES
L101	Hg				Mercury analyser (LECO)	Hg - no preparation			105.6	0.0025	LECO AMA
L101	iAs				Arsenic - oxidation and acid digestion	HCl			102.1	0.06	AFS
L101	Pb				Dry ashing	HNO3			81	0.005	ICP-AES
L102	As				Dry ashing	HCl	1 hour/approx 70°C	digest in HCl, add hydrobromic acid and hydrazine and extract	97	0.1	HG-AAS
L102	Cd				X	X					

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
L102	Hg				H2SO4	HNO3, H2SO4, HCl		in chloroform. back extract with 1M HCl. Add ashing aid and HNO3 and digest, evapourate and ash then disolve in CHCL, reduce with KI/ascorbic and read by AAS hydride generation	88	0.03	CV-AAS
L102	iAs				Dry ashing	HCl			79	0.1	HG-AAS
L102	Pb				X	X					
L104	As	FAPAS TO7192			Closed microwave	HNO3	200C / 20 min	n/a	132.2	0.01	ICP-MS
L104	Cd								111.3	0.1	ICP-MS
L104	Hg								115.2	0.01	ICP-MS
L104	iAs				X	X					
L104	Pb	NCS ZC73013			Closed microwave	HNO3			108.7	0.1	ICP-MS
L105	As	DORM-3 (104.5%)			Closed microwave	H2O2, HNO3	260 °C for 20 min		112	0.01	ICP-MS
L105	Cd	DORM-3 (103.4%)							116	0.005	ICP-MS
L105	Hg	DORM-3 (112.5%)							86	0.01	ICP-MS
L105	iAs	-			X	X			-	-	
L105	Pb	DORM-3 (89.5%)			Closed microwave	H2O2, HNO3			81	0.01	ICP-MS
L106	As	yes			Closed microwave	HNO3	200 deg C, 2 min	not tested	100	0.02	ICP-MS
L106	Cd	yes							100	0.03	ICP-MS
L106	Hg	yes							100	0.02	ICP-MS
L106	iAs	no							0	0	
L106	Pb	yes			Closed microwave	HNO3			100	0.02	ICP-MS
N001	As	BCR 482			Closed microwave	HNO3	200 C, 90 min	extraction 90C with dilluted HCl (0.07 M) + peroxide			AAS
N001	Cd										AAS
N001	Hg										CV-AFS
N001	iAs				extraction, 90C	extraction 90C with dilluted HCl					LC-ICP-MS
N001	Pb				Closed microwave	HNO3					AAS
N002	As	IMEP119		AOAC 999.10	Closed microwave	H2O2, HNO3	180 C for 30 min		80-110	0.067	AAS
N002	Cd								80-110	0.0033	AAS
N002	Hg	IMEP103							80-110	0.016	HG-AAS
N002	iAs				X	X					
N002	Pb	IMEP119			Closed microwave	H2O2, HNO3			80-110	0.0033	AAS
N003	As			EN15763:2009 (for total element analysis) and prEN16802 for iAs	Closed microwave	HNO3	approx 200°C and 20 min	Waterbath extraction at 90°C with dilute HNO3 and H2O2 followed by anion-exchange HPLC-ICPMS determinaton using matrix matched external calibration.	111	0.001	ICP-MS
N003	Cd								94	0.001	ICP-MS
N003	Hg								107	0.01	ICP-MS
N003	iAs				Waterbath assisted extraction with dilute acid	0,1 M HNO3 in 3% H2O2			91	0.003	HPLC-ICP-MS
N003	Pb				Closed microwave	HNO3			101	0.012	ICP-MS
N004	As	SRM 3256 Green Tea	-	SIST EN 15763 and EPA 7473	Closed microwave	H2O2, HNO3	15 min. to 200oC and 20 min. on 200oC	-	108	0.02	ICP-MS
N004	Cd	BCR 150 Skim milk powder	-					-	94	0.001	ICP-MS
N004	Hg		-		For total Hg we used direct mercury analyser.	X		-	88	0.005	CV-AAS
N004	iAs	SRM 3256 Green Tea	-		X	X		-	-	0.05	
N004	Pb	BCR 063R Skim milk powder	-		Closed microwave	H2O2, HNO3		-	98	0.01	ICP-MS
N005	As	BCR 185R			Closed microwave	H2O2, HNO3	120 C/20min	iAs was not determined		0.005	ICP-MS
N005	Cd									0.005	ICP-MS

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
N005	Hg	SRM 2974a			No digestion.	No digestion				0.0005	AAS
N005	iAs				X	X					
N005	Pb	BCR185 R			Closed microwave	H2O2, HNO3				0.005	ICP-MS
N006	As				X	X					
N006	Cd	MR 1 g/l	MR 1 g/l	EN 15763:2009; EN 13806:2002.	Closed microwave	H2O2, HNO3	Main step 180°C 30 min		97	0.0001	ICP-MS
N006	Hg								96.7	0.003	FIMS
N006	iAs				X	X					
N006	Pb	MR 1 g/l	MR 1 g/l		Closed microwave	H2O2, HNO3			104	0.0005	ICP-MS
N007	As	NIST 1547		EN 15763	Closed microwave	4. HNO3	230	Extraction with HNO3 + H2O2, determination with HPLC-ICPMS - EN 16802	100	0.013	ICP-MS
N007	Cd					HNO3			100	0.0036	ICP-MS
N007	Hg								100	0.0075	ICP-MS
N007	iAs				Extraction with HNO3 and H2O2	H2O2, HNO3			100	0.01	HPLC-ICP-MS
N007	Pb	NIST 1547			Closed microwave	HNO3			100	0.011	ICP-MS
N008	As	IMEP-118	Sigma-Aldrich		Closed microwave	H2O2, HNO3	For As, Cd, Pb: first stage: ramp 20 min, hold 40 min, temperature 150 ºC; second stage: ramp 20 min, hold 40 min, temperature 180 ºC		100	0.005	ICP-MS
N008	Cd								100	0.005	ICP-MS
N008	Hg				H2SO4				100	0.02	CV-AAS
N008	iAs				X	X					
N008	Pb	IMEP-118	Sigma-Aldrich		Closed microwave	H2O2, HNO3			100	0.01	ICP-MS
N009	As	Y	Y	EN 14084:2003	Closed microwave	H2O2, HNO3	165oC / 15min		1.1	0.03	HG-AAS
N009	Cd	Y	Y						0.856	0.005	AAS
N009	Hg	Y	Y						0.993	0.03	CV-AAS
N009	iAs				X	X					
N009	Pb	Y	Y		Closed microwave	H2O2, HNO3			0.91	0.02	AAS
N010	As				X	X	200oC/ 25min, cooling/20min				
N010	Cd	BCR 191	BCR 610		1. Closed microwave	4. HNO3			100	0.003	GF-AAS
N010	Hg				X	X					
N010	iAs				X	X					
N010	Pb	BCR 191	BCR 713		1. Closed microwave	4. HNO3			100	0.008	GF-AAS
N011	As	IRMM-804			Closed microwave	HNO3	Total As, Cd, Pb: 180°C, 30 minutes; iAs: 90°C, 60 minutes	0.5 g sample + 9 ml HNO3 0.11M + 1 ml H2O2 30%; MAE at 90°C for 60 minutes with constant stirring		0.0012	ICP-MS
N011	Cd									0.0003	ICP-MS
N011	Hg	BCR-150			No sample digestion, direct mercury analysis	X				0.0001	Direct Mercury Analysis
N011	iAs	NMIJ-7503a, NMIJ-7532a			Closed microwave	H2O2, HNO3				0.0006	HPLC-ICP-MS
N011	Pb	IRMM-804				HNO3				0.0018	ICP-MS
N012	As	DORM-4			Closed microwave	H2O2, HNO3	We have used 150C / 20 min and 180C/ 10min.	Determination by HPLC-ICP-MS after microwave assisted extraction.	102	0.0003	ICP-MS
N012	Cd					H2O2, HNO3			102	0.0001	ICP-MS
N012	Hg	IAEA-336			direct mercury analyser without digestion	X			99.8	0.0001	Direct mercury analysis
N012	iAs	IMEP32-7			for iAs microwave assisted extraction.	H2O2, HCl			88	0.008	HPLC-ICP-MS
N012	Pb	DORM-4			Closed microwave	H2O2, HNO3			100	0.002	ICP-MS
N013	As				Closed microwave	HNO3	200ºc y 20´	HPLC-ICP-MS			ICP-MS
N013	Cd										ICP-MS
N013	Hg										Autoanalyser
N013	iAs				X	X					LC-ICP-MS
N013	Pb				Closed microwave	HNO3					ICP-MS
N014	As			Total Arsenic - EN 14546:2005	Dry ashing	HNO3, Magnesium nitrate hexahydrate and magnesium oxide ashing aid mixture, HCl	Closed microwave - 200C / 20min, Dry Ashing - 450C / 24 to 48hours	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-		0.006	HG-AAS

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
N014	Cd					HNO3		AAS.		0.001	ET-AAS
N014	Hg				Closed microwave	HNO3				0.025	CV-AAS
N014	iAs				Dry ashing	HNO3, Magnesium nitrate hexahydrate and magnesium oxide ashing aid mixture, HCl				0.003	HG-AAS
N014	Pb					HNO3				0.006	ET-AAS
N015	As	Oyster Tissue		NMKL procedure nr 186 2007	Closed microwave	HNO3					ICP-MS
N015	Cd										ICP-MS
N015	Hg										ICP-MS
N015	iAs				X	X					
N015	Pb	Oyster Tissue			Closed microwave	HNO3					ICP-MS
N016	As	Cocoa PT material			Closed microwave	HNO3	200°C	for inorganic arsenic extraction according to CEN-mandate method	106	0.008	ICP-MS
N016	Cd								110	0.002	ICP-MS
N016	Hg								98	0.0015	CV-AFS
N016	iAs				for inorganic arsenic extraction according to CEN-mandate method	extraction according to CEN-mandate method				0.01	LC-ICP-MS
N016	Pb	cacoa PT material			Closed microwave	HNO3			105	0.005	ICP-MS
N017	As	NIST 1570a		EN 15763:2009 and prEN 16802	Closed microwave	HNO3, HCl	190 degrees	A representative test portion of the sample is treated with a diluted nitric acid and hydrogen peroxide solution in a heated waterbath. Hereby the arsenic species are extracted into solution and As(III) is oxidised to As(V). The inorganic arsenic is selectively separated from other arsenic compounds using anion exchange HPLC (High Performance Liquid Chromatography) coupled on-line to the element-s	100	0.01	ICP-MS
N017	Cd	NIST 1570a							100	0.003	ICP-MS
N017	Hg								100	0.02	ICP-IDMS
N017	iAs	BRL PT Cocoa			Water bath 90 degrees for iAs	H2O2, HNO3			100	0.002	LC-ICP-MS
N017	Pb	NIST 1570a			Closed microwave				100	0.004	ICP-MS
N018	As			STN EN 15763	Closed microwave	H2O2	210/45 min.		87	0.00231	ICP-MS
N018	Cd								89	0.0016	ICP-MS
N018	Hg								95	0.00373	AAS
N018	iAs				X	X					
N018	Pb				Closed microwave	H2O2			87	0.00135	ICP-MS
N019	As	FAPAS 752, (98%)		EN 14084:2003	Closed microwave	H2O2, HNO3	200 °C, 30 min	CEN/TS 16731:2014	98	0.01	ETAAS
N019	Cd	BCR 191 (99%)				H2O2, HNO3			99	0.006	ETAAS
N019	Hg	BCR 278 (99%)			direct, withaut pre-tretment	X			99	0.0005	Mercury Analyser, AMA 254 Altec
N019	iAs				1 g sample + 10 ml HNO3 (0,28m) 90min at 95°C	HNO3					HG-AAS
N019	Pb	BCR 191 (102%)			Closed microwave	H2O2, HNO3			98	0.02	ETAAS
N020	As	Rice flour 1568a +IRMM804	std curve		Closed microwave	HNO3	200 °c , 20 minutes	Extraction on waterbath with dilutes nitric acid and hydrogen peroxide. Measurement using anion exchange HPLC coupled on-line to an ICP-MS	99	0.005	ICP-MS
N020	Cd								102	0.0014	ICP-MS
N020	Hg	Rice Flour 1568a							106	0.001	ICP-MS

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
N020	iAs	Rice flour ERM BC 211			Waterbath	H2O2, HNO3			not done	0.0084	LC-ICP-MS
N020	Pb	Rice flour IRMM804			Closed microwave	4. HNO3			97	0.0033	ICP-MS
N021	As	DORM-3		EN 15763 (modified)	Closed microwave	H2O2, HNO3	180 C/ 10 minutes		87	0.005	ICP-MS
N021	Cd	BCR-191							83	0.0003	ICP-MS
N021	Hg	DORM-3							88	0.001	ICP-MS
N021	iAs				X	X					
N021	Pb	BCR-191			Closed microwave	H2O2, HNO3			87	0.0015	ICP-MS
N022	As	ERM 278k	standard solution		Closed microwave	HNO3	200°C / 25 min			0.006	ICP-MS
N022	Cd	NIST 2384								0.001	ICP-MS
N022	Hg	ERM 278k								0.006	ICP-MS
N022	iAs				X	X					
N022	Pb	NIST 2384	standard solution		Closed microwave	HNO3				0.003	ICP-MS
N025	As	NIST 1566b		EN 14083:2003,. EN 14546:2005.	Dry ashing	HNO3	according to the instruction of the producer of the microwave digestion system	Sample was hydrolysed using concentrated hydrochloric acid. After reduction by hydrobromic acid and htdrazine sulfate, the inorganic arsenic was extracted into chloroform, then back-extracted into 1M HCl, dry-ashed and quantified by HG-AAS	95	0.025	HG-AAS
N025	Cd	NIST 1566b, CTA-OTL-1			Closed microwave	H2O2, 4. HNO3			102	0.003	AAS
N025	Hg	NIST 1566b, BCR-422,1568a			in case of mercury direct determination was performed without any digestion mixture (AMA 254)	X			100	0.0002	CV-AAS
N025	iAs	control material(after PT			Dry ashing	HNO3			74	0.04	HG-AAS
N025	Pb	CTA-OTL-1, 1566b			Closed microwave	H2O2, 4. HNO3			87	0.025	AAS
N026	As	LGC 7162	As Stds		Closed microwave	H2O2, 4. HNO3	22 Minutes	The laboratory does not carry out iAs analyses	78.8	0.05	ICP-MS
N026	Cd		Cd Stds.						107.8	0.003	ICP-MS
N026	Hg	TORT 3	Hg Stds						93.1	0.01	ICP-MS
N026	iAs				X	X					
N026	Pb	LGC 7162	Pb Stds		Closed microwave	H2O2, 4. HNO3			99.9	0.03	ICP-MS
N027	As	CRM - ERM	CRM - ERM		Closed microwave	HNO3	For Cd, Pb, As : 5 minutes at 140°C then 20 minutes at 200°C - For iAs : 4 minutes at 80°C		100	0.02	ICP-MS
N027	Cd	ERM	ERM						100	0.01	ICP-MS
N027	Hg				thermal decomposition (AAS-gold amalgamation)	no digestion mixture			100	0.01	thermal decomposition-amalgamation-AAS (AMA254)
N027	iAs				Closed microwave	H2O			100	0.05	HPLC-ICP-MS
N027	Pb					HNO3			100	0.01	ICP-MS
N033	As	BCR185R			Closed microwave	4. HNO3, HCl	ramp to 220C over 20 minutes, held 220C for 15 minutes	hydrochloric acid solubilization, reduction, chloroform extraction & back-extraction into hydrochloric acid	97	0.001	ICP-MS
N033	Cd					4. HNO3, HCl			95	0.001	ICP-MS
N033	Hg					4. HNO3, HCl			91	0.001	ICP-MS
N033	iAs	IMEP107			room temperature acid solubilization	HCl			83	0.004	ICP-MS
N033	Pb	BCR185R			Closed microwave	4. HNO3, HCl			95	0.003	ICP-MS
N034	As			AOAC, 974.14 (2005), AOAC, 999.10: (2010)	Closed microwave	H2O2, 4. HNO3	200 0 C/40 minutes			0.012	AAS
N034	Cd								91.6		AAS
N034	Hg									0.016	CV-AAS
N034	iAs				X	X					
N034	Pb				Closed microwave	H2O2, 4. HNO3			83.1		AAS
N038	As			LST EN 15763:2010	Closed microwave	H2O2, 4. HNO3	200 degres of Celsium, 30 min.			0.017	ICP-MS
N038	Cd									0.0033	ICP-MS
N038	Hg									0.0017	ICP-MS
N038	iAs				X	X					

Part. key	Measurand	CRM - method validation	CRM - instrument calibration	Standard Method Used	Digestion type	Digestion mixture	Digestion temperature	Analytical method for the iAs determination	Recovery (%)	LODs	Technique
N038	Pb				Closed microwave	H2O2, 4. HNO3				0.0033	ICP-MS
N039	As				X	X	110°C for 10 min; 200°C for 18 min				
N039	Cd	dolt4;soya fleur	linearcalibr.1-5-20-50ppb		Closed microwave	H2O2, 4. HNO3			102.11	0.007	ICP-MS
N039	Hg				X	X					
N039	iAs				X	X					
N039	Pb	brownbreadbcr 191;lichen	linearcalibr.1-5-20-50ppb		Closed microwave	H2O2, 4. HNO3			90.91	0.008	ICP-MS
N054	As			SR EN 13805, SR EN 13806	X	X	180 degree C				
N054	Cd	IRMM-805	NIST 1640a		Closed microwave	H2O2, 4. HNO3			99	0.025	AAS
N054	Hg	BCR-463							100	0.05	CV-AAS
N054	iAs				X	X					
N054	Pb	IRMM-805	NIST 1640a		Closed microwave	H2O2, 4. HNO3			98	0.25	AAS
N073	As			Yes	Closed microwave	H2O2, 4. HNO3	180 °C		98	0.01	ICP-MS
N073	Cd								93	0.002	ICP-MS
N073	Hg								90	0.01	ICP-MS
N073	iAs				X	X					
N073	Pb				Closed microwave	H2O2, 4. HNO3			95	0.01	ICP-MS
N077	As	GBW 7604	CZ9003(1N)	EN15763	Open microwave	H2O2, 4. HNO3	190 degrees / 10 minutes	closed MW extraction with temperature 90 degrees 20 minutes, LC -ICP-MS analysis	100	0.006	ICP-MS
N077	Cd								100	0.006	ICP-MS
N077	Hg				Hg-direct combustion in an oxygen in Advanced Mercury Analyzer (AMA 254)	Hg-dry ashing, combustion in an oxygen,without acids			100	0.0003	direct mercury analysis
N077	iAs				Closed microwave	H2O2, HCl					LC-ICP-MS
N077	Pb	GBW 7604	CZ 9041(N)		Open microwave				100	0.09	ICP-MS

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JRC Mission

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Working in close cooperation with policy Directorates-General, the JRC addresses key societal challenges while stimulating innovation through developing new methods, tools and standards, and sharing its know-how with the Member States, the scientific community and international partners.

Serving society
Stimulating innovation
Supporting legislation