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IMEP-28: Total arsenic, cadmium, lead and mercury in food supplements

Interlaboratory Comparison Report

Ines Baer, Beatriz de la Calle, Lane Sander, Stephen Long, Steven Christopher, Russell Day, Karen Murphy, Inge Verbist, Danny Vendelbo, Håkan Emteborg, Philip Taylor

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European Commission
Joint Research Centre
Institute for Reference Materials and Measurements

Contact information
Ms. Beatriz de la Calle
European Commission
Joint Research Centre
Institute for Reference Materials and Measurements
Retieseweg 111
2440 Geel, Belgium

E-mail: maria.de-la-calle@ec.europa.eu

Tel.: +32 (0) 14 571252
Fax: +32 (0) 14 571865

http://irmm.jrc.ec.europa.eu/
http://www.jrc.ec.europa.eu/

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Interlaboratory Comparison Report

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Ines Baer (*a*)
Beatriz de la Calle (*b,c*)
Lane Sander *(c)*
Stephen Long *(c)*
Steven Christopher *(c)*
Russell Day *(c)*
Karen Murphy *(c)*
Inge Verbist (*d*)
Danny Vendelbo *(d)*
Håkan Emteborg *(c)*
Philip Taylor (*e*)

(a) ILC coordinator, (b) IMEP programme coordinator,
(c) technical / scientific support, (d) administrative support,
(e) ILC conception

IMEP®

Contents

1 Summary

The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre (JRC), a Directorate-General of the European Commission, operates the International Measurement Evaluation Programme IMEP. It organises interlaboratory comparisons (ILC's) in support to EU policies. This report presents the results of an ILC which focussed on the determination of total As, Cd, Pb and Hg in food supplements relying on Commission Regulations 333/2007 [1] and 1881/2006 [2].

The test material used in this exercise was the candidate standard reference material (SRM) SRM 3256, of the National Institute of Standards and Technology (NIST), the matrix being green tea food supplement. The material was labeled at IRMM and dispatched to the participants on the first week of June 2009. Each participant received approximately 5 g of test material. Sixty-two participants from twenty countries registered to the exercise of which 46 reported results for total Cd, 50 for total Pb, 42 for total As and 40 for total Hg. The assigned values were the reference values as provided by NIST.

The uncertainties, u_{ref}, of the respective assigned values were also provided by NIST. Participants were invited to report the uncertainty on their measurements. This was done by 49 of the 58 laboratories having submitted results in this exercise.

Laboratory results were rated with z and zeta-scores in accordance with ISO 13528 [3]. Standard deviation for proficiency assessment (also called target standard deviation) was fixed to 15 % for total Cd, Pb and As by the advisory board of this ILC, on the basis of the outcome of previous ILC's, and 22% for mercury based on the modified Horwitz equation.

The outcome of the exercise was altogether positive, with over 60 % of the participants reaching satisfactory scores for both types of scorings for almost all elements.

2 IMEP support to EU policy

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

IMEP **distributes metrology** from the highest level down to the field laboratories. These laboratories can benchmark their measurement result against the IMEP reference value. This value is established according to metrological best practice.

IMEP helps laboratories to assess their estimate of **measurement uncertainty**. The participants are invited to report the uncertainty on their measurement result. IMEP integrates the estimate into the scoring, and provides assistance for the interpretation.

IMEP supports EU policies by organising intercomparisons in the frame of specific EU Directives, or on request of a specific Directorate-General. IMEP-28 provided specific support to the following stakeholders:

- To the European Co-operation for Accreditation (EA) in the frame of a formal collaboration on a number of metrological issues, including the organisation of intercomparisons. National accreditation bodies were invited to nominate a limited number of laboratories for free participation in IMEP-28. Mrs Annika Norling from the Swedish Board for Accreditation and Conformity Assessment (SWEDAC) liaised between EA and IMEP for this intercomparison. This report does not discern the EA nominees from the other participants. Their results are however summarised in a separate report to EA.

- To the Community Reference Laboratory for Heavy Metals in Feed and Food (CRL-HM) in the frame of the support to the National Reference Laboratories (NRLs). The exercise was announced to the network of NRLs and they were invited to distribute the information between routine laboratories in their country. The results gathered in IMEP-28 represent the state of the art of the official control laboratories involved in analysis of food supplements in Europe.

3 Introduction

According to Directive 2002/46/EC of the European Parliament and of the Council on the approximation of the laws of the Member States relating to food supplements [4], *"'Food supplements' means foodstuffs the purpose of which is to supplement the normal diet and which are concentrated sources of nutrients or other substances with a nutritional or physiological effect alone or in combination, marketed in dose form, namely forms such as capsules, pastilles, tablets, pills and other similar forms, sachets of powder, ampoules of liquids, drop dispensing bottles and other similar forms of liquids and powders designed to be taken in measured small unit quantities"*.

High levels of Pb, Cd and Hg have been found in certain food supplements and were notified through the Rapid Alert System for Food and Feed (RASFF). It has been shown that these food supplements can contribute significantly to human exposure to the mentioned metals. In order to protect public health, it was therefore considered appropriate to set maximum levels for Pb, Cd and Hg. Maximum levels for Pb, Cd and Hg in food supplements have been introduced by Regulation (EC) No. 629/2008 [5] of 2 July 2008 and are applicable since 1 July 2009. These maximum levels are 3.0 mg/kg for lead

for all food supplements, 1.0 mg/kg for cadmium for all food supplements excluding supplements consisting exclusively or mainly of dried seaweed or of products derived from seaweed, for which a maximum level of 3.0 mg/kg applies, and 0.1 mg/kg for mercury for all food supplements.

For arsenic no maximum level is yet established at European level but it is anticipated that limits will be set for arsenic in the near future as the methodology for the determination of arsenic improves. Data from a recent SCOOP report (EU Scientific Cooperation Task, 2004) on exposure of the European population to heavy metals in their diet showed that with the exception of seafood and animal offal, the concentration of arsenic is generally less than 250 ug/kg [6]. It seems however that high levels of arsenic are frequently found in different food supplements and have been subject to Raid Alert Notifications in recent years. Products often notified are clays, mineral drinks, products on basis of seaweed and ayurvedic food supplements.

IMEP organised a proficiency test (PT) exercise for the determination of total As, Cd, Pb and Hg in food supplements. This exercise was open to all laboratories involved in this type of analysis and it was carried out in parallel with a PT organised by the CRL-HM for its network of NRLs (IMEP-106). The same test material was used in both exercises.

4 Scope

The scope of this ILC is to test the competence of the participating laboratories to determine the total concentration of Cd, Pb, As and Hg in food supplements. The assessment of the measurement results is undertaken on the basis of requirements laid down in legislation [1, 2] and follows the administrative and logistics procedures of IMEP, the International Measurement and Evaluation Programme, of IRMM. This programme is accredited according to ISO Guide 43-1.

5 Time frame

This interlaboratory comparison was agreed upon by the NRLs network at the third CRL-HM workshop held on 25-26 September 2008. The ILC was announced to the EA coordinator on 4 May 2009, who would forward it then to the national accreditation bodies in order to nominate laboratories. The exercise was publicly announced on the IMEP webpage [7] during the first half of May 2009. Finally, on 12 May 2009 NRLs involved in IMEP-106 were informed about this parallel exercise to give them the opportunity to invite laboratories from their respective countries.

Interested laboratories had time until Friday 29 May 2009 to register. Samples were sent out to the laboratories on 3 June 2009. For all laboratories the deadline for reporting results was the 3 July 2009.

This deadline was extended for two laboratories by one week, after getting confirmation that they would be able to submit results in time.

6 Invitation, registration and distribution

Invitations for participation were sent to the EA coordinator (Annex 1) for distribution to nominated and interested laboratories. NRLs were informed via email (Annex 2). And a call for participation was also released on the IRMM website (Annex 3).

Instructions on measurands, sample storage, reconstitution and measurement were sent to the participants together with the samples. The letter also contained the individual code for access to the result reporting website and further details on the envisaged time frame (Annex 4).

The participants who had submitted a result received the reference value two weeks after the reporting deadline. Fig 1 shows the participating countries and the number of participants having reported results.

6.1 Confidentiality and participation fees

EA was invited to nominate laboratories for participation. The following confidentiality statement was made to EA: "Confidentiality of the participants and their results towards third parties is guaranteed. However, IMEP will disclose details of the participants that have been nominated by EA to the EA working group for ILCs in Testing. The EA accreditation bodies may wish to inform the nominees of this disclosure."

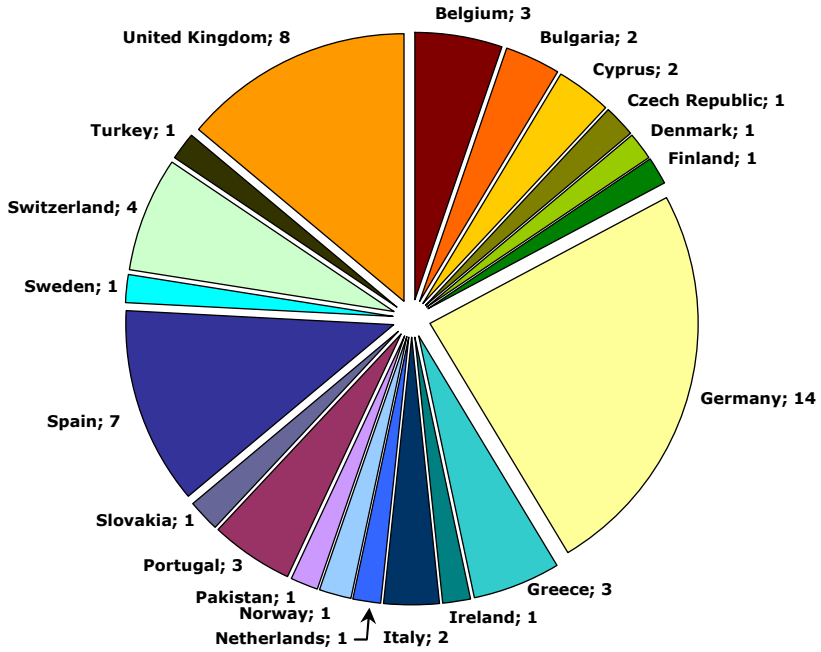
Laboratories nominated by EA were exempt of charge on the basis of a collaboration agreement between EA and IRMM. The participation fee for other laboratories was € 160.

6.2 Distribution

The ILC sample was dispatched by IRMM on 3 June 2009 to the participants. Each participant received two sachets containing approximately 2.5 g of test material, an accompanying letter with instructions on sample handling and reporting (Annex 4) and a form that had to be sent after receipt of the test material to confirm its arrival (Annex 5).

The dispatch was followed by the messenger's parcel tracking system on internet and in most of the cases the sample was delivered within a couple of days. In one case, the dispatch took longer than the one-week period. It was however assumed that the parcel was not submitted to high enough temperatures or long enough time to have an impact on the samples' stability.

Fig 1- Country distribution in IMEP-28 based on number of participants having submitted results



6.3 Procedure to apply

Details on this intercomparison were discussed with the NRLs at the third workshop organised by the CRL-HM, held in Geel on 25-26 September 2008. Concrete instructions were given to all participants in a letter that accompanied the test material. The measurands and matrix were defined as "Total Cd, Pb, As and Hg in food supplements".

Laboratories were asked to perform two or three independent measurements and report them, together with the mean of the results and its associated uncertainty. The measurement results were to be corrected for moisture following a procedure described in the accompanying letter (the procedure has been optimised at IRMM by the Reference Materials Unit) and for recovery. Participants were asked to follow their routine procedures. The results were to be reported in the same manner (eg. number of significant figures) as those normally reported to customers.

The results were to be reported in a special on-line form for which each participant received an individual access code. A special questionnaire was attached to this on-line form. The questionnaire was intended to provide further information on the measurements and the laboratories. A copy of the questionnaire is presented in Annex 6.

7 Test material

7.1 Preparation

Processing of the test material used in this exercise was made by NIST. SRM 3256 is a blend of 4 different green tea-containing formulations obtained from commercial sources. The tablets were ground and capsules were opened, then the materials were sieved, and blended prior to packaging. The material was packed in portions of 2.6 g ± 0.1 g in heat-sealed 4 mL polyethylene bags. These bags were heat sealed in aluminized plastic bags along with 2 packets of silica gel.

NIST dispatched 386 sachets at room temperature by courier to IRMM.

7.2 Homogeneity and stability

The homogeneity tests were conducted by NIST. No short term stability test was performed because according to the experience of the SRM producer the measurands covered in this exercise are stable at room temperature in this type of matrix.

8 Reference values and their uncertainties

NIST provided reference values for all the measurands included in this study. Certification of SRM 3526 was carried out in the same period of time that IMEP-28 was organised and conducted. The reference values as determined by NIST using ICP-MS were used as assigned values (X_{ref}) for this ILC.

The assigned values and their respective estimated uncertainties, expanded and not expanded, the degrees of freedom and the expansion factors are summarised in Table 1.

Table 1 - Assigned values and their associated expanded uncertainties for the measurands of this ILC as provided by NIST.

Measurand	X_{ref} (mg kg $^{-1}$)	U_{ref} (mg kg^{-1})	df	k	u_{ref} (mg kg^{-1})
Total As	0.278	0.022	5.02	2.57	0.009
Total Cd	0.0264	0.0012	5.07	2.56	0.0005
Total Pb	0.314	0.069	5	2.57	0.027
Total Hg	0.0129	0.0026	6.6	2.39	0.0011

X_{ref} is the reference value and u_{ref} is the corresponding associated standard uncertainty; U_{ref} is the estimated associated expanded uncertainty.

The uncertainty in the reference concentration values is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide 35. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the effect of within-laboratory components of uncertainty. The uncertainty for mercury incorporates an inhomogeneity component and is in the form of a prediction interval. The coverage factor, k, is determined from the Student's t-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte.

9 Evaluation of results

9.1 General observations

From the 62 laboratories that registered for participation, 58 submitted their results and completed the associated questionnaire. Concerning the four non-submissions, 1 laboratory cancelled its participation for technical reasons, 1 informed us of not being able to realise the required analyses with such small quantities, 1 notified us 2 weeks after submission deadline that all their results were below the detection limit, and the last one did not provide us with information or justification.

Results reported as "less than" were treated as not reporting. Unfortunately, this was the case for quite a number of laboratories – 6 participants reported this expression for one element, 13 for two and one for three elements. Half of the participants submitted values for 1, 2 or 3 of the elements, and only 28 laboratories reported values for all 4 elements.

No obvious wrong result reporting was observed, except one participant who reported a result for Hg that seems to be a factor 1000 higher than expected. However, since the other results from the same participant were correctly reported, the value was used as reported. Two participants submitted results with unreasonable high uncertainties, and one participant did the same for one element but not for the other reported results.

9.2 Uncertainties and coverage factor

All except eight participants reported an uncertainty associated to their results, which is a very satisfying observation (> 86%). Three participants reported uncertainties for only some of their results.

The laboratories were asked to perform 1 to 3 replicates, and to report them together with the mean, its associated uncertainty and the expansion factor. Fourteen laboratories reported an uncertainty with each single replicate result. Of these, two laboratories seem to have derived the uncertainties of the means by averaging the uncertainties of the single measurements, which is fundamentally incorrect.

Another 5 laboratories reported different uncertainties with the measurements, but none with the result's mean, and so the mean uncertainty had to be taken for the results evaluation.

Concerning the factor k, only 33 participants gave a value, which seems to reflect a lack of understanding of what k means. This situation should be improved.

9.3 Scores and evaluation criteria

Individual laboratory performance is expressed in terms of z and zeta-scores in accordance with ISO 13528 [3] and the International Harmonised Protocol [8].

$$z = \frac{x_{lab} - X_{ref}}{\hat{\sigma}} \qquad \text{and} \qquad zeta = \frac{x_{lab} - X_{ref}}{\sqrt{u_{ref}^2 + u_{lab}^2}}$$

where:

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

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

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

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

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

Both scores can be interpreted as: satisfactory result for $|score| \leq 2$, questionable result for $2 < |score| \leq 3$ and unsatisfactory result for $|score| > 3$.

z-score

The z-score compares the participant's deviation from the reference value with the standard deviation accepted for the proficiency test, $\hat{\sigma}$. In this exercise $\hat{\sigma}$ was fixed to 15 % for the four measurands by the advisory board of this ILC, on the basis of the outcome of previous ILC's on heavy metal determinations in food, organised by IMEP.

Should participants feel that these $\hat{\sigma}$ values are not fit for their purpose they can recalculate their scorings with a standard deviation matching their requirements. x_{lab} is the mean of the individual measurement results calculated by the ILC organiser.

zeta-score

The zeta-score states if the laboratory result agrees with the assigned value within the respective uncertainties. An unsatisfactory zeta-score might be due to an underestimation of the uncertainty, or to a large error causing a large deviation from the reference value, or to a combination of the two factors. A laboratory with an unsatisfactory zeta-score has an estimation of the uncertainty of its measurements

which is not consistent with the laboratory's deviation from the reference value. Laboratories reporting a u_{lab} which is higher than $\hat{\sigma}$, have an analytical system in place which is not up to the "state-of-the-art".

The standard uncertainty of the laboratory (u_{lab}) was calculated by dividing the reported expanded uncertainty by the reported coverage factor (*k*). When *k* was not specified, the reported expanded uncertainty was considered as the half-width of a rectangular distribution; u_{lab} was then calculated by dividing this half-width by √3, as recommended by Eurachem and CITAC [9]. Laboratories that did not report any uncertainty, did not receive a zeta-score.

9.4 Laboratory results and scorings

The results as reported by the participants are summarised in Annex 7 - 10 for total As, total Cd, total Pb and total Hg, respectively. A table of the results together with the z- and zeta-scores and their graphical representation are provided. Laboratory codes were given randomly.

The results were also evaluated using Kernel density plots, useful to highlight sub-populations. These plots can be found in Annex 11. The software used to calculate Kernel densities was provided by the Statistical Subcommittee of the Analytical Methods Committee (AMC) of the Royal Society of Chemistry [10].

Regarding the z- and zeta-scores, the results are summarised in Table 2. The laboratories' performance appears to be good for total As, total Pb, and total Hg – the percentage of satisfying scores range between 63 and 76 %, for z- and zeta-scores. For total Cd this can only be said for the z-score, with less than half of laboratories having a good zeta-score. Also when looking at the number of laboratories having a satisfying z- and zeta-score, shown in Table 3, the number is much lower for Cd. This indicates a problem with the laboratories' estimation of the appropriate uncertainty for this element. The larger u_{ref} provided by NIST for Pb made that the problem of underestimation of uncertainties look less severe for Pb.

Table 2 - Overview of scores with S(atisfactory), Q(uestionable) and U(nsatisfactory)

	z-scores					zeta-scores			
	As	**Cd**	**Pb**	**Hg**		**As**	**Cd**	**Pb**	**Hg**
n	42	46	50	40	**n**	33	39	44	36
S (#)	32	31	33	27	**S (#)**	22	17	27	24
S(%)	76%	67%	66%	68%	**S(%)**	65%	44%	63%	67%
Q(#)	4	6	6	3	**Q(#)**	2	4	4	3
Q(%)	10%	13%	12%	8%	**Q(%)**	6%	10%	9%	8%
U(#)	6	9	11	10	**U(#)**	10	18	12	9
U(%)	14%	20%	22%	25%	**U(%)**	29%	46%	28%	25%

- number of laboratories

Table 3 – Number of laboratories having satisfying z- and zeta-score

	As	Cd	Pb	Hg
Both scores S (#)	22	13	27	22
n	33	39	44	36

It is interesting however to observe that the results' distribution for total Cd around the assigned value and its uncertainty is good, whereas for total Hg there is a tendency for higher results, probably due to contaminations, interference problems or errors in calibration of low concentrations. Annex 12 summarises all scorings per lab and element.

9.5 Further information extracted from the questionnaire

Additional information was gathered from the questionnaire that participants were asked to fill in (Annex 6). For uncertainty estimates, various combinations of one or more options (Q3 in Annex 6) were given. Thirty-one laboratories use the uncertainty as calculated during the in-house validation of the method, twenty-seven laboratories use the uncertainty obtained by measuring replicates (i.e precision). Seven participants applied a bottom-up approach following the ISO-GUM. Six laboratories used the known uncertainty of the standard method. Four laboratories made use of intercomparison data and one used the expert guesstimate (which corresponds to "estimation based on judgment", as defined in the Eurachem/CITAC guide on Quantifying Uncertainty in Analytical Measurements [9]). One laboratory stated the comparison to a CRM as a third method to base their uncertainty on. Twenty-four laboratories provide an uncertainty statement to their customers and thirty-three do not.

All participants but six corrected their results for the water content. Four out of the six gave an answer when asked for the reason and they are listed in Table 4. The way in which the moisture content of the test material was to be calculated was described in detail in the sample accompanying letter.

Table 4 – Water correction

Part Nr	Reason for missing water correction
2756	We perform correction only in order to referring analytical data to raw foodstuff (Reg. CE/1881/2006)
2772	All below limit of detection, hence not possible to apply factor
2830	it was stated not to use for heavy metal determination.
2834	water content not relevant (3.49%)

Thirty-four laboratories analysed the test material following an official method. Two participants did not answer to this question. The information reported by the laboratories about their method of analysis is summarised in Annex 13. Forty-eight participants carry out this type of analysis (as regards the measurands, matrix and methods) on a routine basis, and ten do not.

All participants but five have a quality system in place based on ISO 17025, two have a quality system based on ISO 9000, one based on a national accreditation system, one has no quality system in place and one did not answer. The number of non-accredited laboratories is twelve for the determination of Cd (three did not provide any answer), thirteen for the determination of Pb (two did not provide any answer), twenty-one for the determination of As (five did not provide any answer), and sixteen for the determination of Hg (one did not provide any answer). Forty-seven laboratories participate regularly in ILC's for this type of analysis, ten do not.

Forty-one participants use a reference material for this type of analysis: all of them use it for the validation procedure and fourteen for calibration of the instrument. Table 5 summarises the reference materials used for the validation of the methods as reported by the participants.

Table 5 – Reference materials used by the participants as reported in the questionnaire

Part Nr	Use of reference Material?	Used for validation?	Used for calibration?	Which reference material?
2754	yes	yes		AAFCO, FAPAS
2736	yes	yes	yes	BCR
2896	yes	yes	yes	several CRM, SRM, local RM
2756	yes	yes	no	BCR185R - NRCC DORM3
2753	yes	yes	yes	NIST 1515; NIST 1575a
2770	yes	yes	no	
2772	yes	yes	no	ERM 278 (Mussell Tissue)
2738	yes	yes	no	BCR 279
2830	yes	yes	yes	different
2879	yes	yes	yes	BCR
2834	yes	yes	no	various
2952	yes	yes	yes	NCS ZC80003 Brassica Oleracea; BCR-679 White cabbbage; Traceable CRM for As (No 39436), Cd (No 51994), Hg (No 16482) and Pb (No 16595) Fluka
2790	yes	yes	yes	BRC, NIST
2833	yes	yes	no	Bovine Liver (NBS), Oyster Tissue (NBS), Tomato Leaves (NIST)
2939	yes	yes	yes	Hg
2893	yes	yes	no	TORT-2
2795	yes	yes	no	BCR-482
2881	yes	yes	yes	interlaboratory comparison material
2882	yes		yes	peach leaves, dolt
2651	yes	yes	no	NIST 1547
2791	yes	yes	yes	ICP-AES, Amagamation-AAS
2670	yes	yes	no	
2936	yes	yes	no	Apple leaves from NIST, Lobster from the National Research Council Canada
3150	yes	yes	yes	
2755	no	yes	no	We use remaining quantities of proficiency test.
2953	yes	yes	no	SRM1568a
2915	yes	yes	no	ERM CE 278
2910	yes	yes	yes	
2885	yes	yes	no	BCR 186, NIST SRM 1575, NIST 1643e
2752	yes	yes	no	Fapas
2892	yes	yes	no	Different materials (IRMM, BCR, LGC, ...)

Part Nr	Use of reference Material?	Used for validation?	Used for calibration?	Which reference material?
2916	yes	yes	no	Dogfish muscle (DORM-2;NRC-CNRC)
2889	yes		no	BCR-151
2914	yes	yes	no	
2890	yes	yes	no	
2971	yes	yes	no	
2938	yes	yes	no	
2835	yes	yes	no	IAEA V-10 Hay Powder, SRM 1570a-Spinach Leaves
2796	yes	yes	no	
2891	yes	yes	no	IAEA 407
2956	yes	yes	yes	CTA-OTL-1; CertiPUR Merck for Pb, Cd, As, HG
2950	yes	yes	no	BCR63

Several laboratories indicated that the amount of test material distributed was too small. According to the International Harmonised Protocol for Proficiency Testing of Analytical Chemistry Laboratories [8] *"The quantity of material in a distribution unit must be sufficient for the analysis required, including any reanalysis where permitted by the scheme protocol"*. In the case of this ILC the material was distributed as produced by the SRM producer in sachets containing about 2.5 g of test material. Being aware that this amount was not enough for the 2 to 3 replicates required plus the material needed for the moisture content determination (1.5 g in total), two sachets were sent to every participant. Every participant received then around 5 g of test material which was enough for three replicates (assuming an average sample intake for every replicate of 0.5 g) and the moisture determination. About 2 g would still remain for further replicates in case accidental spillage occurred. Furthermore, extra material was sent to the participants when requested if a reasonable justification was given, which was the case for two laboratories (15 g and 25 g, sent additionally).

Four participants commented about eventual interferences affecting measurements, particularly those of As, due to matrix effects (e.g. presence of other metals – Fe).

10 Conclusion

The main conclusion that can be made is that it was a rather successful exercise. Over 60 % of the participants gave satisfactory scores and this even for those heavy metals present at very low concentration in the test material. This is particularly satisfying in the case of mercury, an element known to be difficult to analyse.

An extra effort is still needed in the evaluation of the uncertainties associated with the results. Laboratories must take into account that the uncertainty of a measurement frequently depends on the concentration range, so that when analysing trace elements present at low concentrations, as it was the case of Cd in this exercise, the uncertainty is higher.

It should also be said that the small quantities and low concentrations apparently caused some difficulties for the analysis resulting in missing results (or reporting of "less than") especially in the case of mercury and cadmium, and to a lesser extent for arsenic.

11 Acknowledgements

The organiser of the IMEP-28 thanks NIST for the provision of the candidate Standard Reference Material used as test material in this exercise. The Reference Material Unit of IRMM is acknowledged for their support in the optimisation of a method for the determination of the moisture content of the material. Anne-Mette Jensen is thanked for revising the manuscript.

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

Organisation	Country
Eurofins Belgium - Site Oostkamp	BELGIUM
Laboratorium ECCA NV	BELGIUM
CODA-CERVA	BELGIUM
Central Laboratory for Chemical Testing and Control	BULGARIA
Testing Center Laborex	BULGARIA
PANCHRIS ANIMAL PREMIX LTD	CYPRUS
cp.FOODLAB LTD	CYPRUS
Institut pro testování a certifikaci, a.s.	CZECH REPUBLIC
Danish Veterinary and Food Administration, Region North	DENMARK
MetropoliLab	FINLAND
Landesbetrieb Hessisches Landeslabor	GERMANY
Chemisches und Veterinäruntersuchungsamt Ostwestfalen-Lippe	GERMANY
Chemisches und Veterinäruntersuchungsamt	GERMANY
Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit	GERMANY
Landeslabor Berlin-Brandenburg	GERMANY
CVUA-RRW	GERMANY
Landeslabor Schleswig-Holstein	GERMANY
GALAB Laboratories GmbH	GERMANY
Amt für Verbraucherschutz Kreis Mettmann	GERMANY
Chemisches und Veterinäruntersuchungsamt Sigmaringen	GERMANY
LUA Sachsen	GERMANY
Landesamt für Verbraucherschutz des Landes Sachsen-Anhalt	GERMANY
Landeslabor Berlin-Brandenburg	GERMANY
muva kempten	GERMANY
Chemical Service of Ioannina, General Chemical State Laboratory	GREECE
AGROLAB	GREECE
General Chemical State Laboratory, B' Division of Chemical Services of Thessaloniki	GREECE
Public Analysts Laboratory	IRELAND
Istituto Zooprofilattico Sperimentale delle Venezie	ITALY
Istituto Zooprofilattico Sperimentale della Puglia e della Basilicata	ITALY
Food and Consumer Product Safety Authority (VWA)	NETHERLANDS
National Veterinary Institute	NORWAY
Qarshi Research International Pvt. Ltd.	PAKISTAN
ASAE	PORTUGAL

Organisation	Country
Silliker Portugal, S.A	PORTUGAL
Univ. Católica Portuguesa - Esc. Sup. Biotecnologia	PORTUGAL
BEL/NOVAMANN, International, Ltd	SLOVAKIA
Laboratorio Agroalimentario y de Sanidad Animal	SPAIN
Gobierno Vasco	SPAIN
Laboratorio Agroalimentario de la Generalitat Valenciana	SPAIN
LABORATORIO AGROALIMENTARIO.CORDOBA	SPAIN
Laboratorio Agrario Regional. Junta de Castilla y León	SPAIN
CENTRO DE SALUD PUBLICA DE ALICANTE	SPAIN
FUNDACIÓN AZTI	SPAIN
ALS Scandinavia AB	SWEDEN
Laboratorium der Urkantone	SWITZERLAND
Laboratorio cantonale	SWITZERLAND
SQTS	SWITZERLAND
Kantonales Labor Zürich	SWITZERLAND
MSM Food Control Laboratories Inc	TURKEY
Hampshire Scientific Service	UNITED KINGDOM
Premier Analytical Services	UNITED KINGDOM
Worcestershire Scientific Services	UNITED KINGDOM
Staffordshire County Council	UNITED KINGDOM
Tayside Scientific Services	UNITED KINGDOM
Minton Treharne & Davies Ltd	UNITED KINGDOM
Eurofins Laboratories	UNITED KINGDOM
Lancashire County Laboratory	UNITED KINGDOM

Abbreviations

AMC	Analytical Methods Committee of the Royal Society of Chemistry
CRL-HM	Community Reference Laboratory for Heavy Metals in Feed and Food
CITAC	Co-operation for International Traceability in Analytical Chemistry
EA	European Co-operation for Accreditation
EC	European Commission
EU	European Union
EURACHEM	A focus for Analytical Chemistry in Europe
GUM	Guide to the Expression of Uncertainty in Measurement
ICP-MS	Inductively-Coupled Plasma Mass Spectrometry
ILC	Interlaboratory Comparison
IMEP	International Measurement Evaluation Programme
IRMM	Institute for Reference Materials and Measurements
ISO	International Organisation for Standardisation
IUPAC	International Union for Pure and Applied Chemistry
JRC	Joint Research Centre
NIST	National Institute of Standards and Technology
NRL	National Reference Laboratory
PT	Proficiency Test
RASFF	Rapid Alert System for Food and Feed
SCOOP	EU Scientific Cooperation Task
SRM	Standard Reference Material
SWEDAC	Swedish Board for Accreditation and Conformity Assessment

References

[1] Commission Regulation (EC) No 333/2007 of 28 March 2007 laying down the methods of sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs (2007), issued by European Commission, Official Journal of the European Union, L 88/29

[2] Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs (2006), issued by European Commission, Official Journal of the European Union, L 364/5

[3] Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons (2005), issued by International Organisation for Standardisation, No ISO 13528

[4] Directive 2002/46/EC of the European Parliament and of the Council of 10 June 2002 on the approximation of the laws of the Member States relating to food supplements (2002), issued by European Commission, Official Journal of the European Union, L 183/51

[5] Commission Regulation (EC) No 629/2008 of 2 July 2008 amending Regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs (2008), issued by European Commission, Official Journal of the European Union, L 173/6

[6] Mercury, Lead, Cadmium, Tin and Arsenic in Food (2009), Food Safety Authority of Ireland, Toxicology Factsheets Series, (Issue No 1 / May 2009)

[7] http://irmm.jrc.ec.europa.eu/html/interlaboratory_comparisons/

[8] Thompson M, Ellison SLR, Wood R (2006) The International Harmonized Protocol for the proficiency testing of analytical chemistry laboratories: (IUPAC technical report). Pure and Applied Chemistry 78(1): 145-196

[9] Quantifying Uncertainty in Analytical Measurement (2000), Eurachem/CITAC, http://www.eurachem.org

[10] Representing data distributions with Kernel density estimates (2006). AMC Technical Brief issued by the Statistical Subcommittee of the Analytical Methods Committee (AMC) of the Royal Society of Chemistry

Annexes

Annex 1 : Invitation to EA to nominate laboratories

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements

IMEP

Geel, 4 May 2009
JRC.D04/IBa/ive/ARES(2009)/85756

SWEDAC
Annika Norling
Box 2231
10315 Stockholm
SWEDEN

Dear Annika,

Intercomparison for heavy metals in food supplements

The Institute for Reference Materials and Measurements (IRMM) organises an interlaboratory comparison for the determination of the total amount of four heavy metals in food supplements. The concerned elements are lead, arsenic, mercury, and cadmium.

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 their consideration. There is a maximum number of 80 samples at your disposal, i.e. ca. 2-3 nominees per country.

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

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 715. Fax: (32-14) 571 865.
E-mail: jrc-irmm-imep@ec.europa.eu

Registration of participants is open until 29 May 2009. Distribution of the samples is foreseen for the first half of June 2009. In order to register, laboratories must:

1. **Enter** their details online:

   https://irmm.jrc.ec.europa.eu/ilc/ilcRegistration.do?selComparison=280

2. **Print** the completed form when the system asks to do so
   Do not forget to clearly indicate on the printed form that you have been appointed by the European Cooperation for Accreditation to take part in this exercise.

3. **Send** the printout to both the IMEP-28 and the EA-IMEP-28 coordinators:

IMEP-28 coordinator	**EA-IMEP-28 coordinator**
Ms Ines Baer	Mrs Annika Norling
Fax +32 14 571865	Fax +46 0 791 89 29
E-mail: jrc-irmm-imep@ec.europa.eu	E.Mail: Annika.norling@swedac.se

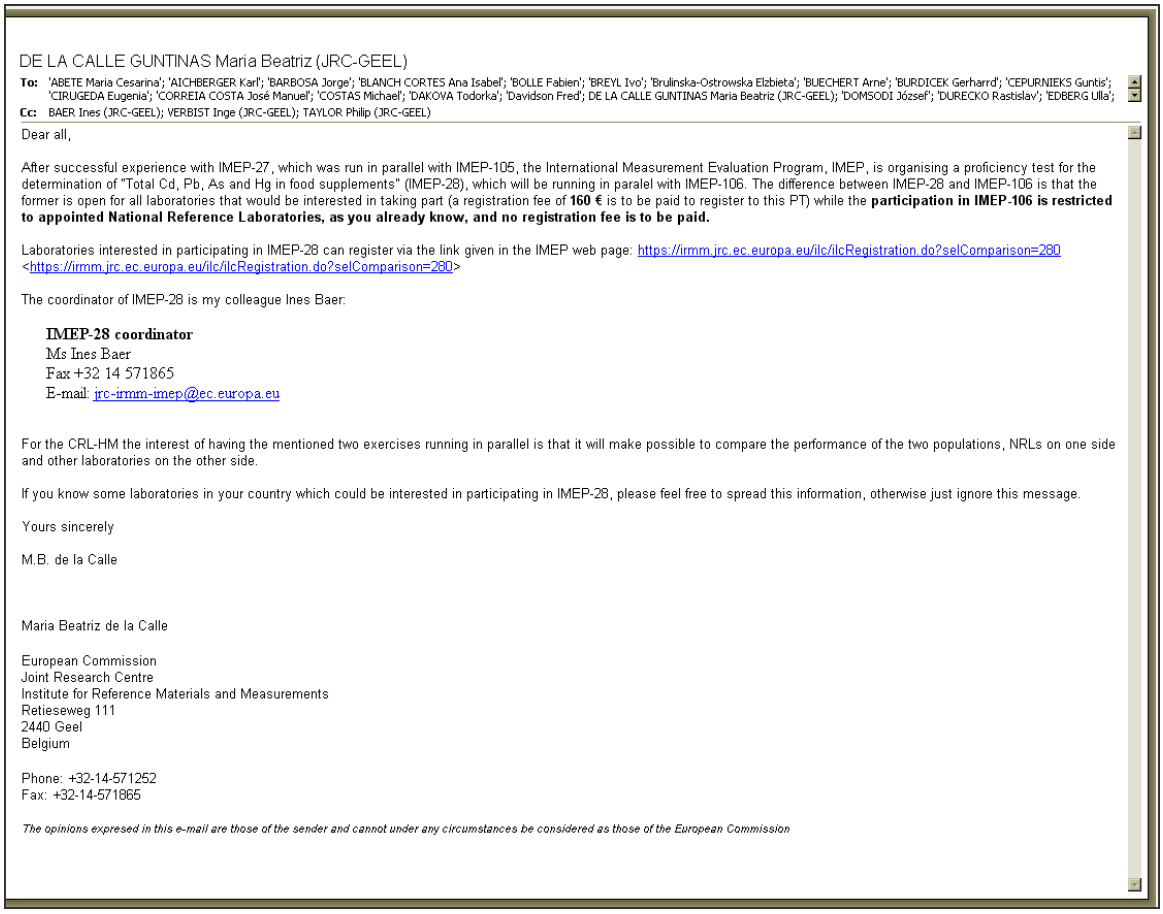
Please contact me if you have any questions or comments. We are looking forward to our cooperation!

With kind regards

Ines Baer
IMEP-28 Coordinator

2

Annex 2 : Invitation sent to NRLs



DE LA CALLE GUNTINAS Maria Beatriz (JRC-GEEL)

To: 'ABETE Maria Cesarina'; 'AICHBERGER Karl'; 'BARBOSA Jorge'; 'BLANCH CORTES Ana Isabel'; 'BOLLE Fabien'; 'BREYL Ivo'; 'Brulinska-Ostrowska Elzbieta'; 'BUECHERT Arne'; 'BURDICEK Gerharrd'; 'CEPURNIEKS Guntis'; 'CIRUGEDA Eugenia'; 'CORREIA COSTA José Manuel'; 'COSTAS Michael'; 'DAKOWA Todorka'; 'Davidson Fred'; DE LA CALLE GUNTINAS Maria Beatriz (JRC-GEEL); 'DOMSODI József'; 'DURECKO Rastislav'; 'EDBERG Ulla';

Cc: BAER Ines (JRC-GEEL); VERBIST Inge (JRC-GEEL); TAYLOR Philip (JRC-GEEL)

Dear all,

After successful experience with IMEP-27, which was run in parallel with IMEP-105, the International Measurement Evaluation Program, IMEP, is organising a proficiency test for the determination of "Total Cd, Pb, As and Hg in food supplements" (IMEP-28), which will be running in paralel with IMEP-106. The difference between IMEP-28 and IMEP-106 is that the former is open for all laboratories that would be interested in taking part (a registration fee of **160 €** is to be paid to register to this PT) while the **participation in IMEP-106 is restricted to appointed National Reference Laboratories, as you already know, and no registration fee is to be paid.**

Laboratories interested in participating in IMEP-28 can register via the link given in the IMEP web page: https://irmm.jrc.ec.europa.eu/ilc/ilcRegistration.do?selComparison=280 <https://irmm.jrc.ec.europa.eu/ilc/ilcRegistration.do?selComparison=280>

The coordinator of IMEP-28 is my colleague Ines Baer:

IMEP-28 coordinator
Ms Ines Baer
Fax +32 14 571865
E-mail: jrc-irmm-imep@ec.europa.eu

For the CRL-HM the interest of having the mentioned two exercises running in parallel is that it will make possible to compare the performance of the two populations, NRLs on one side and other laboratories on the other side.

If you know some laboratories in your country which could be interested in participating in IMEP-28, please feel free to spread this information, otherwise just ignore this message.

Yours sincerely

M.B. de la Calle

Maria Beatriz de la Calle

European Commission
Joint Research Centre
Institute for Reference Materials and Measurements
Retieseweg 111
2440 Geel
Belgium

Phone: +32-14-571252
Fax: +32-14-571865

The opinions expresed in this e-mail are those of the sender and cannot under any circumstances be considered as those of the European Commission

Annex 3 : Announcement on IRMM website

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
Isotope measurements

IMEP®

Geel, 04 June 2009
JRC.D04/IBa/ive/ARES(2009)/115822

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

Participation to IMEP-28, a proficiency test exercise for the determination of total Cd, Pb, As and Hg in food supplements

Dear «TITLE» «SURNAME»,

Thank you for participating in the IMEP-28 intercomparison for the determination of **total** Cd, Pb, As and Hg in food supplements.

This parcel contains:
a) Two sachets containing each approximately 2.5 g of the test material
b) A "Confirmation of Receipt" form
c) This accompanying letter

Please check whether the sachets containing the test material remained undamaged during transport. Then, please send the "Confirmation of receipt" form back (fax: +32-14-571865, e-mail: jrc-irmm-imep@ec.europa.eu). You should store the samples in a dark and cold place (not more than 18 °C) until analysis.

The measurands are: **total** Cd, Pb, As and Hg in food supplements. The procedure used for the analyses should resemble as closely as possible the one that you use in routine sample analysis.

Please perform two or three independent measurements per measurand. Correct the measurement results for recovery, and report the corrected values, plus their mean on the reporting website. The results should be reported in the same form (e.g., number of significant figures) as those normally reported to the customer.

The results are to be reported referring to dry mass and thus corrected for humidity. To calculate the water content in the test material, please apply the following procedure:

«PARTKEY»

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 682. Fax: (32-14) 571 865.

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

1. *Weigh accurately 0.5 g of test material in a glass container of 5-7 cm diameter, Preferably with a lid because when the prescribed drying time has passed, the glass container must cool down about 30 minutes in a desiccator before weighing.*
2. *Place it in an oven for 120 ± 5 min at 80 ± 2 °C.*
3. *Place the glass container covered with a lid in a desiccator and wait 30 min before weighing the test material again.*

Note 1: perform the measurements of the water content in triplicate.
Note 2: ***do not use for the heavy metal determinations the aliquots of test material that you have used for the water content determination!***

You can find the reporting website at https://irmm.jrc.ec.europa.eu/ilc/ilcReporting.do
To access this webpage you need a personal password key, which is: **«PARTKEY»**. The system will guide you through the reporting procedure. Please enter for each parameter the two or three measurement results plus the technique you used, but do not report the uncertainty for each individual measurement. In addition, please report the mean of the results with technique and with uncertainty information in the allocated space for "measurement 4". After entering all results, please also complete the relating questionnaire. Do not forget to save, submit and confirm always when required.

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

The deadline for submission of results is 03/07/2009.

Please keep in mind that collusion is contrary to professional scientific conduct and serves only to nullify the benefits of proficiency tests to customers, accreditation bodies and analysts alike.

Your participation in this project is greatly appreciated. If you have any remaining questions, please contact me by e-mail:
JRC-IRMM-IMEP@ec.europa.eu

With kind regards

Dr. Ines Baer
IMEP-28 Co-ordinator

Enclosures: 1) two sachets containing the test material; 2) confirmation of receipt form; 3) Accompanying letter.

Cc: P. Taylor

«PARTKEY»

Annex 5 : 'Confirmation of receipt' form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
Isotope measurements

Annex to JRC.D04/IBa/ive/ARES(2009)/115822

«TITLE» «FIRSTNAME» «SURNAME»
«ORGANISATION»
«DEPARTMENT»
«ADDRESS»
«ADDRESS2»
«ADDRESS3»
«Address4»
«ZIP» «TOWN»
«COUNTRY»

IMEP-28

Total Cd, Pb, As and Hg in food supplements

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 Ines Baer

IMEP-28 Coordinator
EC-JRC-IRMM
Retieseweg 111
B-2440 GEEL, Belgium

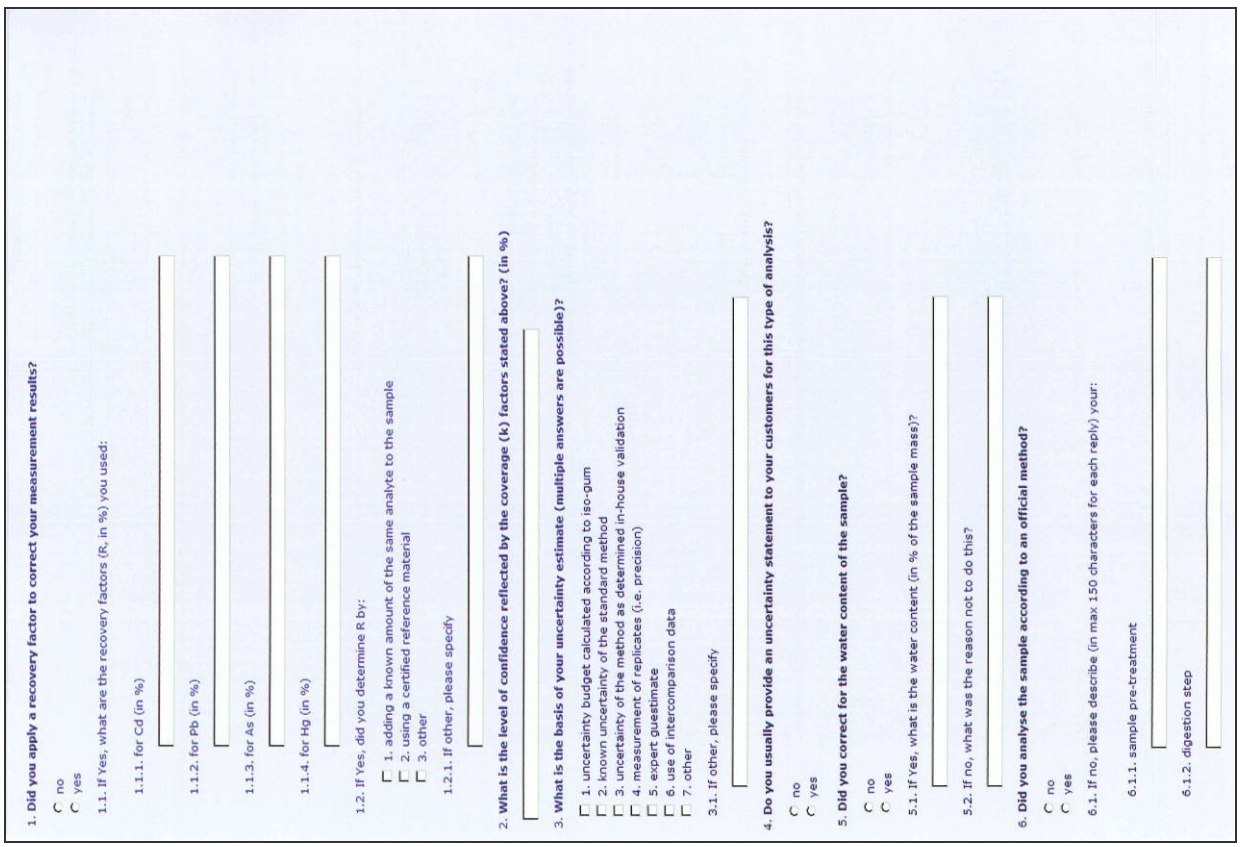
Fax : +32-14-571865
e-mail : jrc-irmm-imep@ec.europa.eu

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 682. Fax: (32-14) 571 865.

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

IMEP®

Annex 6 : Questionnaire



1. **Did you apply a recovery factor to correct your measurement results?**
   - ○ no
   - ○ yes

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

1.1.1. for Cd (in %)

1.1.2. for Pb (in %)

1.1.3. for As (in %)

1.1.4. for Hg (in %)

1.2. If Yes, did you determine R by:
- ☐ 1. adding a known amount of the same analyte to the sample
- ☐ 2. using a certified reference material
- ☐ 3. other

1.2.1. If other, please specify

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

3. **What is the basis of your uncertainty estimate (multiple answers are possible)?**
- ☐ 1. uncertainty budget calculated according to iso-gum
- ☐ 2. known uncertainty of the standard method
- ☐ 3. uncertainty of the method as determined in-house validation
- ☐ 4. measurement of replicates (i.e. precision)
- ☐ 5. expert guestimate
- ☐ 6. use of intercomparison data
- ☐ 7. other

3.1. If other, please specify

4. **Do you usually provide an uncertainty statement to your customers for this type of analysis?**
   - ○ no
   - ○ yes

5. **Did you correct for the water content of the sample?**
   - ○ no
   - ○ yes

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

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

6. **Did you analyse the sample according to an official method?**
   - ○ no
   - ○ yes

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

6.1.1. sample pre-treatment

6.1.2. digestion step

6.1.3. extraction / separation step

6.1.4. instrument calibration step

6.2. If yes, which:

7. **Does your laboratory carry out this type of analysis (as regards the measurands, matrix and methods) on a routine basis?**
   - ○ no
   - ○ yes

7.1. If Yes, please estimate the number of samples (Cd, Hg, Pb measurements together):
   - ○ a) 0-50 samples per year
   - ○ b) 50-250 samples per year
   - ○ c) 250- 1000 samples per year
   - ○ d) more than 1000 samples per year

8. **Does your laboratory have a quality system in place?**
   - ○ no
   - ○ yes

8.1. If yes, which:
- ☐ iso 9000 series
- ☐ iso/iec 17025
- ☐ other

8.1.1. If other, please specify

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

Questions/Response table	No	Yes	*Info*
Total Hg	○	○	
Total As	○	○	
Total Pb	○	○	
Total Cd	○	○	

10. **If you are accredited, by which Accreditation Body?**

11. **Does your laboratory take part in an interlaboratory comparison for this type of analysis on a regular basis?**
    - ○ no
    - ○ yes

11.1. If yes, which one(s):

12. **Does your laboratory use a reference material for this type of analysis?**
    - ○ no
    - ○ yes

12.1. If yes, is the material used for the validation of procedures?
    - ○ no
    - ○ yes

12.2. If yes, is the material used for calibration of instrument?
    - ○ no
    - ○ yes

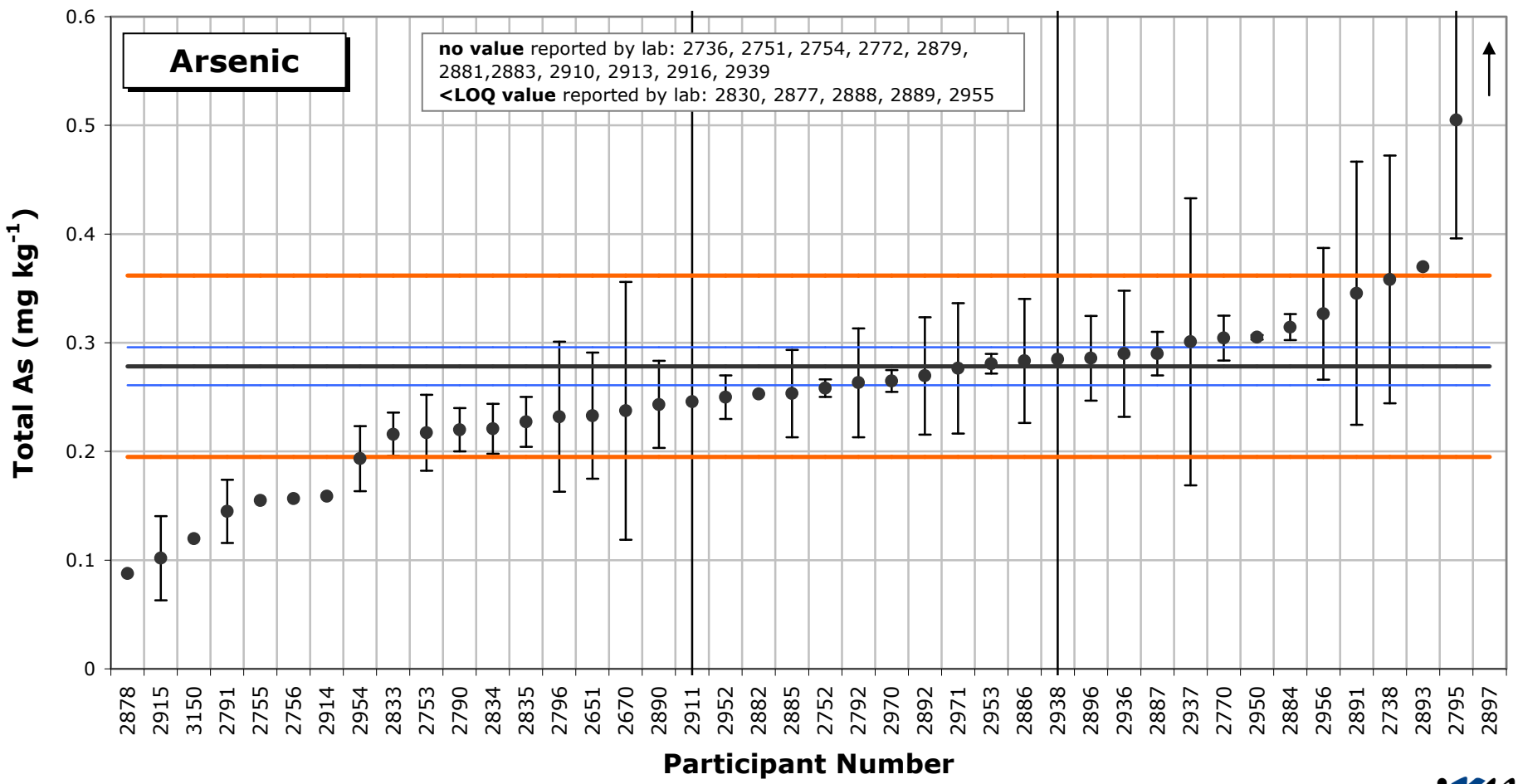
12.3. If yes, which one(s):

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

Annex 7 : Results for Arsenic

X_{ref} = 0.278 and U_{ref} = 0.022; all values are given in (mg kg^{-1})

Part Nr	x1	x2	x3	x4	Ulab	k	Mean	ulab	Technique	z	zeta
2651	0.241	0.222	0.236		0.058	2	0.233	0.029	ICP-MS	-1.1	-1.5
2670	0.272	0.203			0.119	2	0.238	0.059	ICP-AES	-1.0	-0.7
2738	0.354	0.371	0.350		0.114	2	0.358	0.057	HG-AAS	1.9	1.4
2752	0.257	0.261	0.257		0.008	0.926	0.258	0.009	ICP-MS	-0.5	-1.6
2753	0.210	0.230	0.212		0.035	2	0.217	0.018	HG-AAS	-1.5	-3.1
2755	0.16	0.15					0.16		FAFS	-3.0	
2756	0.15	0.16	0.16				0.16		ETAAS	-2.9	
2770	0.28	0.32	0.313		0.021	1.96	0.304	0.011	ICP-MS	0.6	1.9
2790	0.22	0.23	0.21		0.02	√3	0.22	0.01	FIAS	-1.4	-4.0
2791	0.145	0.145			0.029	2	0.145	0.015	ICP-AES	-3.2	-7.9
2792	0.28	0.24	0.27		0.05	2	0.26	0.03	HG-AAS	-0.4	-0.6
2795	0.50	0.51			0.109	2	0.51	0.055	ICP-MS	5.4	4.1
2796	0.233	0.231			0.069	2	0.232	0.035	HG-AAS	-1.1	-1.3
2830	<0.05	<0.05	<0.05	<0.05					ICP-MS		
2833	0.211	0.221			0.02	√3	0.216	0.01	HG-AAS	-1.5	-4.3
2834					0.023	2	0.221	0.012	ICP-MS	-1.4	-4.0
2835	0.231	0.223	0.228		0.023	√3	0.227	0.013	ICP-MS	-1.2	-3.2
2877	<0.1	<0.1	0.1						ETAAS		
2878	0.092	0.091	0.080				0.088		HG-AAS	-4.6	
2882	0.254	0.252					0.253		ICP-MS	-0.6	
2884	0.315	0.314			0.012	2	0.315	0.006	ICP-MS	0.9	3.4
2885	0.27	0.25	0.24		0.04	2	0.25	0.02	ICP-MS	-0.6	-1.2
2886	0.280	0.287			0.057	2	0.284	0.029	ICP-MS	0.1	0.2
2887	0.28	0.30			0.02	√3	0.29	0.01	ICP-MS	0.3	0.8
2888	<1								ETAAS		
2889	<0.1	<0.1							ETAAS		
2890	0.27	0.21	0.25		0.04	2	0.24	0.02	ICP-MS	-0.8	-1.6
2891	0.348	0.345	0.344		0.121	2	0.346	0.061	ICP-MS	1.6	1.1
2892	0.258	0.274	0.277		0.054	2	0.270	0.027	ICP-MS	-0.2	-0.3
2893							0.37		HG-AAS	2.2	
2896	0.3025	0.2716	0.2835		0.039	2	0.286	0.020	ICP-MS	0.2	0.3
2897	0.96	0.95	0.94				0.95		ICP-MS, ICP-AES	16.1	
2911	0.2367	0.2840	0.217		16.3	2	0.2459	8.2	ICP-MS	-0.8	0.0
2914	0.162	0.156					0.159		ETAAS	-2.9	
2915	0.087	0.095	0.110	0.115	0.039	2	0.102	0.019	ETAAS	-4.2	-8.3
2936	0.29	0.29			0.058	√3	0.29	0.033	ICP-MS	0.3	0.3
2937	0.329	0.304	0.270		0.132	2	0.301	0.066	ICP-MS	0.5	0.3
2938	0.24	0.25	0.32	0.33	20	2	0.285	10	ICP-MS	0.2	0.0
2950	0.30	0.31			0.002	√3	0.31	0.001	ICP-MS	0.6	3.0
2952	0.23	0.24	0.26	0.27	0.02	2	0.25	0.01	HG-AAS	-0.7	-2.1
2953	0.281	0.276	0.285		0.009	2	0.281	0.005	ICP-MS	0.1	0.2
2954	0.1850	0.2209	0.1746		0.030	√3	0.1935	0.017	ICP-OES	-2.0	-4.4
2955	<0.06	<0.06	<0.06	<0.06					HG-AAS		
2956	0.3435	0.3099			0.0606	0.8983	0.3267	0.0675	ETAAS	1.2	0.7
2970	0.261	0.268	0.266		0.01	2	0.265	0.01	ICP-MS	-0.3	-1.3
2971	0.27	0.28	0.28		0.06	2	0.28	0.03	HG-AAS	0.0	-0.1
3150							0.120		ICP-OES	-3.8	

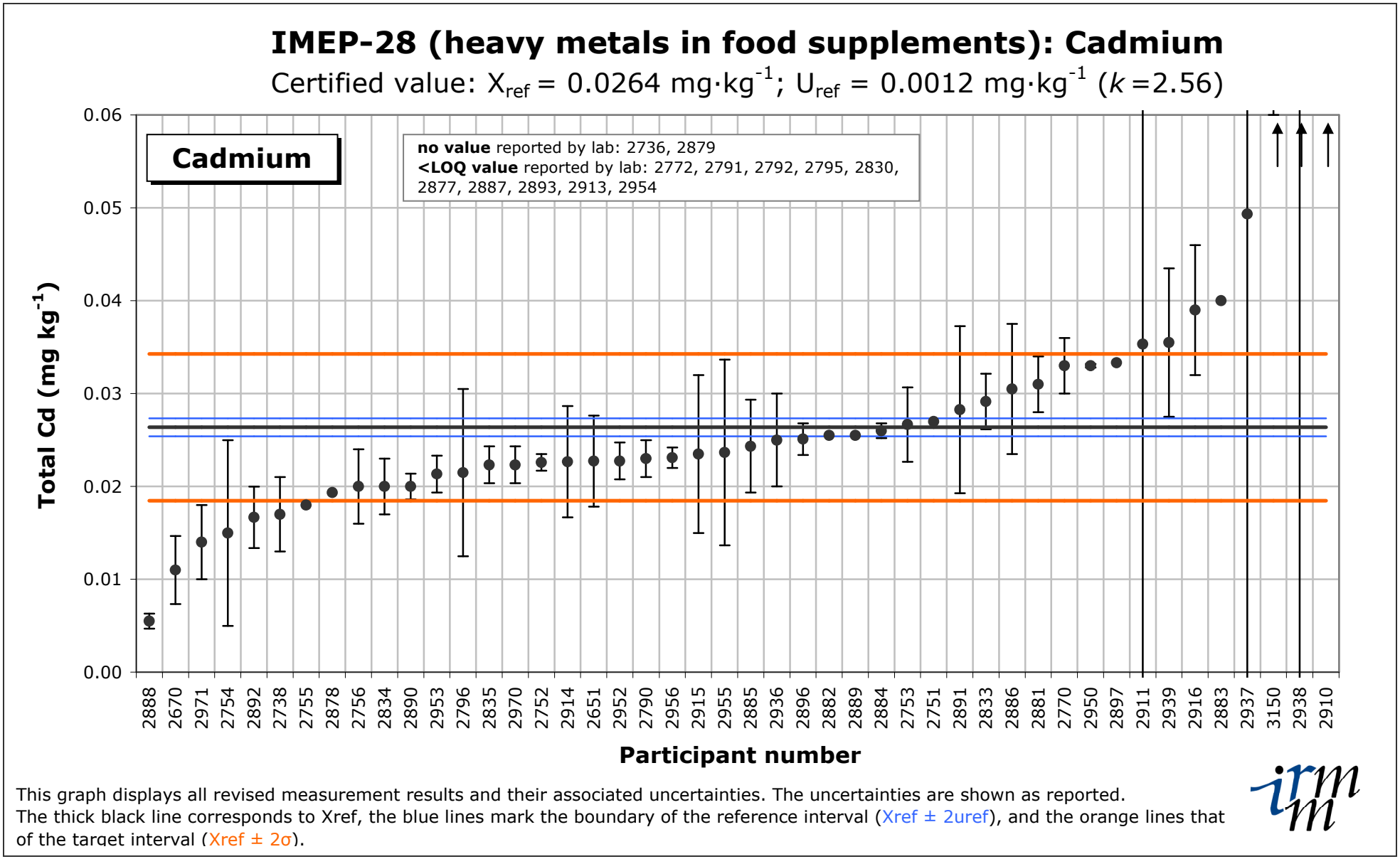


This graph displays all revised measurement results and their associated uncertainties. The uncertainties are shown as reported.
The thick black line corresponds to Xref, the blue lines mark the boundary of the reference interval (Xref ± 2uref), and the orange lines that of the target interval (Xref ± 2σ).

Annex 8 : Results for Cadmium

X_{ref} = 0.0264 and U_{ref} = 0.0012; all values are given in (mg kg^{-1})

Part Nr	x1	x2	x3	x4	Ulab	k	Mean	ulab	Technique	z	zeta
2651	0.0236	0.0225	0.0221		0.0049	2	0.0227	0.0025	ICP-MS	-0.9	-1.5
2670	0.012	0.012	0.009		0.004	√3	0.011	0.002	ICP-AES	-3.9	-7.1
2738	0.018	0.017	0.016		0.004	2	0.017	0.002	ETAAS	-2.4	-4.6
2751	0.029	0.025	0.029	0.025			0.027		Grafite Furnance A. Atómic	0.2	
2752	0.0225	0.0226	0.0227		0.0009	0.952	0.0226	0.0009	ICP-MS	-1.0	-3.6
2753	0.023	0.031	0.026		0.004	2	0.027	0.002	ETAAS	0.1	0.1
2754	0.019	0.011			0.010	2	0.015	0.005	ICP-AES	-2.9	-2.3
2755	0.017	0.019					0.018		ETAAS	-2.1	
2756	0.02	0.02	0.02		0.004	2.31	0.02	0.002	ETAAS	-1.6	-3.5
2770	0.030	0.035	0.034		0.003	1.96	0.033	0.002	ICP-MS	1.7	4.1
2772	<0.02	<0.02	<0.02	<0.02					GFAAS		
2790	0.023	0.022	0.024		0.002	√3	0.023	0.001	HG-AAS	-0.9	-2.7
2791	<0.25	<0.25	<0.25	<0.25					ICP-AES		
2792	<0.1	<0.1	<0.1	<0.1					FAAS		
2795	<0.025	<0.025	<0.025						ICP-MS		
2796	0.021	0.022			0.009	2	0.022	0.005	ETAAS	-1.2	-1.1
2830	<0.01	<0.01	<0.01	<0.01					ICP-MS		
2833	0.0288	0.0295			0.003	√3	0.0292	0.002	ETAAS	0.7	1.5
2834					0.003	2	0.020	0.002	ICP-MS	-1.6	-4.0
2835	0.023	0.022	0.022		0.002	√3	0.022	0.001	ICP-MS	-1.0	-3.2
2877	<0.25	<0.25	<0.25						ICP-AES		
2878	0.020	0.019	0.019				0.019		ETAAS	-1.8	
2881	0.030	0.030	0.033		0.003	√3	0.031	0.002	AAS graphit	1.2	2.6
2882	0.025	0.026					0.026		ICP-MS	-0.2	
2883							0.04		ETAAS	3.4	
2884	0.026	0.026			0.0008	2	0.026	0.0004	ICP-MS	-0.1	-0.6
2885	0.023	0.024	0.026		0.005	2	0.024	0.003	ICP-MS	-0.5	-0.8
2886	0.031	0.030			0.007	2	0.031	0.004	ICP-MS	1.0	1.2
2887	<0.05	<0.05	<0.05						ICP-MS		
2888					0.0008	√3	0.0055	0.0005	ETAAS	-5.3	-31.3
2889	0.028	0.023					0.026			-0.2	
2890	0.02	0.02	0.02		0.0014	2	0.020	0.0007	ICP-MS	-1.6	-7.5
2891	0.0294	0.0281	0.0273		0.009	2	0.0283	0.005	ICP-MS	0.5	0.4
2892	0.017	0.017	0.016		0.0033	2	0.017	0.0017	ICP-MS	-2.5	-5.6
2893	<0.18								FAAS		
2896	0.0247	0.0247	0.0259		0.0017	2	0.0251	0.0009	ETAAS	-0.3	-1.3
2897	0.03	0.04	0.03				0.03		ICP-MS	1.8	
2910	0.0797	0.0897	0.0827		0.009	√3	0.0840	0.005	ICP-OES	14.6	11.1
2911	0.041	0.031	0.034		21.3	2	0.035	10.65	ICP-MS	2.3	0.0
2913	<0.1	<0.1	<0.1	<0.1					FAAS		
2914	0.024	0.019	0.025		0.006	2	0.023	0.003	ETAAS	-0.9	-1.2
2915	0.021	0.023	0.025	0.025	0.009	2	0.024	0.004	ETAAS	-0.7	-0.7
2916	0.04	0.038			0.007	2	0.039	0.004	ETAAS	3.2	3.6
2936	0.025	0.025			0.005	√3	0.025	0.003	ICP-MS	-0.3	-0.5
2937	0.038	0.062	0.048		0.09	2	0.049	0.05	ICP-MS	5.8	0.5
2938	0.09	0.09	0.07		20	√3	0.08	12	ICP-MS	14.4	0.0
2939	0.031	0.040			0.008	√3	0.036	0.005	ICP-AES	2.3	2.0
2950	0.035	0.031			0.0002	√3	0.033	0.0001	ICP-MS	1.7	13.4
2952	0.024	0.022	0.023	0.022	0.002	2	0.023	0.001	ETAAS	-0.9	-3.3
2953	0.023	0.020	0.021		0.002	2	0.021	0.001	ICP-MS	-1.3	-4.5
2954	<0.04	<0.04	<0.04	<0.04					ICP-OES		
2955	0.021	0.024	<0.023	0.026	0.010	√3	0.024	0.006	Graphit-AAS	-0.7	-0.5
2956	0.0236	0.0226			0.0011	0.9412	0.0231	0.0012	ETAAS, GF-AAS	-0.8	-2.6
2970	0.024	0.021	0.022		0.002	2	0.022	0.001	ICP-MS	-1.0	-3.6
2971	0.014	0.014	0.014		0.004	2	0.014	0.002	ETAAS	-3.1	-6.0
3150					0.006	√3	0.066	0.003	ICP-OES	10.0	11.3

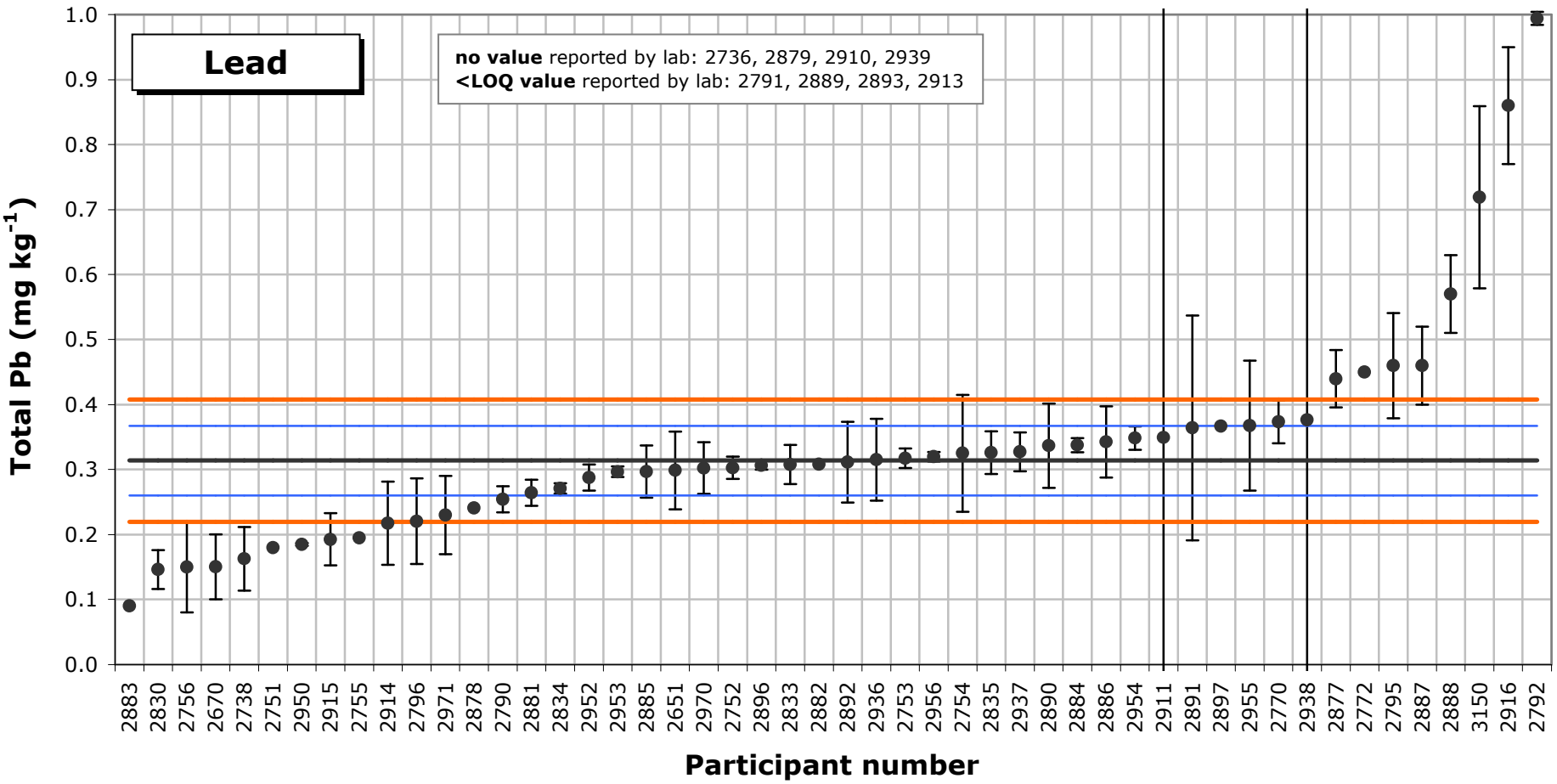


This graph displays all revised measurement results and their associated uncertainties. The uncertainties are shown as reported.
The thick black line corresponds to Xref, the blue lines mark the boundary of the reference interval (Xref ± 2uref), and the orange lines that of the target interval (Xref ± 2σ).

Annex 9 : Results for Lead

X_{ref} = 0.314 and U_{ref} = 0.069; all values are given in (mg kg^{-1})

Part Nr	x1	x2	x3	x4	Ulab	k	Mean	ulab	Technique	z	zeta
2651	0.307	0.302	0.287		0.060	√3	0.299	0.035	ICP-MS	-0.3	-0.3
2670	0.188	0.142	0.116	0.155	0.050	2	0.150	0.025	ICP-AES	-3.5	-4.4
2738	0.151	0.169	0.168		0.049	2	0.163	0.025	ICP-AES	-3.2	-4.2
2751	0.173	0.181	0.177	0.189			0.180		Graphit Furnace AAS	-2.8	
2752	0.301	0.302	0.305		0.017	0.990	0.303	0.017	ICP-MS	-0.2	-0.3
2753	0.317	0.325	0.310		0.015	2	0.317	0.008	ETAAS	0.1	0.1
2754	0.32	0.33			0.09	2	0.33	0.05	ICP-AES	0.2	0.2
2755	0.19	0.20					0.20		ETAAS	-2.5	
2756	0.14	0.16	0.15		0.07	2.13	0.15	0.03	ETAAS	-3.5	-3.9
2770	0.38	0.38	0.36		0.03	1.96	0.37	0.02	ICP-MS	1.3	1.9
2772	<0.2	<0.2	0.2	0.7			0.45		GFAAS	2.9	
2790	0.254	0.260	0.248		0.02	√3	0.254	0.01	HG-AAS	-1.3	-2.0
2791	<2	<2	<2	<2					ICP-AES		
2792	1.014	0.975	0.993		0.01	2	0.994	0.01	FAAS	14.5	24.9
2795	0.47	0.45			0.081	2	0.46	0.041	ICP-MS	3.1	3.0
2796	0.237	0.204			0.066	2	0.221	0.033	ETAAS	-2.0	-2.2
2830	0.145	0.146	0.131	0.162	0.03	√3	0.146	0.02	ICP-MS	-3.6	-5.3
2833	0.301	0.314			0.03	√3	0.308	0.02	ETAAS	-0.1	-0.2
2834					0.008	2	0.271	0.004	ICP-MS	-0.9	-1.6
2835	0.325	0.327			0.033	√3	0.326	0.019	ICP-MS	0.3	0.4
2877	0.332	0.547	0.440		0.044	√3	0.440	0.025	ICP-AES	2.7	3.4
2878	0.242	0.236	0.244				0.241		ETAAS	-1.6	
2881	0.265	0.261	0.267		0.02	√3	0.264	0.01	AAS graphit	-1.0	-1.7
2882	0.305	0.311					0.308		ICP-MS	-0.1	
2883	0.09						0.09		ETAAS	-4.8	
2884	0.339	0.336			0.011	2	0.338	0.006	ICP-MS	0.5	0.9
2885	0.32	0.28	0.29		0.04	2	0.30	0.02	ICP-MS	-0.4	-0.5
2886	0.359	0.326			0.055	2	0.343	0.028	ICP-MS	0.6	0.8
2887	0.46	0.47			0.06	√3	0.46	0.03	ICP-MS	3.1	3.3
2888	0.57				0.06	√3	0.57	0.03	ETAAS	5.4	5.9
2889	<0.10	<0.10							ETAAS		
2890	0.31	0.35	0.35		0.065	2	0.34	0.033	ICP-MS	0.5	0.5
2891	0.372	0.354	0.367		0.173	2	0.364	0.087	ICP-MS	1.1	0.6
2892	0.280	0.319	0.335		0.062	2	0.311	0.031	ICP-MS	0.0	-0.1
2893	<0.58								FAAS		
2896	0.3070	0.3034	0.3091		0.007	2	0.307	0.004	ICP-MS	-0.2	-0.3
2897	0.39	0.36	0.35				0.37		ICP-MS	1.1	
2911	0.3672	0.3206	0.3604		23	2	0.3494	12		0.8	0.0
2913	<0.1	<0.1	<0.1	<0.1					FAAS		
2914	0.202	0.253	0.197		0.064	2	0.217	0.032	ETAAS	-2.0	-2.3
2915	0.205	0.200	0.195	0.170	0.040	2	0.193	0.020	ETAAS	-2.6	-3.6
2916	0.94	0.78			0.09	2	0.86	0.05	ETAAS	11.6	10.4
2936	0.32	0.31			0.063	√3	0.315	0.036	ICP-MS	0.0	0.0
2937	0.332	0.327	0.323		0.03	2	0.327	0.02	ICP-MS	0.3	0.4
2938	0.37	0.39	0.37		20	√3	0.38	12	ICP-MS	1.3	0.0
2950	0.18	0.19			0.002	√3	0.19	0.001	ICP-MS	-2.7	-4.8
2952	0.32	0.27	0.28	0.28	0.02	2	0.29	0.01	ETAAS	-0.6	-0.9
2953	0.301	0.293	0.296	0.296	0.008	2	0.297	0.004	ICP-MS	-0.4	-0.6
2954	0.3669	0.3429	0.3351		0.018	√3	0.3483	0.010	ICP-OES	0.7	1.2
2955	0.39	0.34	0.35	0.39	0.10	√3	0.37	0.06	Graphit-AAS	1.1	0.8
2956	0.3196	0.3195			0.0073	1.089	0.3196	0.0067	ETAAS	0.1	0.2
2970	0.278	0.331	0.298		0.04	2	0.302	0.02	ICP-MS	-0.2	-0.3
2971	0.23	0.23	0.23		0.06	2	0.23	0.03	ETAAS	-1.8	-2.1
3150	0.719				0.140	√3	0.719	0.081	ICP-OES	8.6	4.8

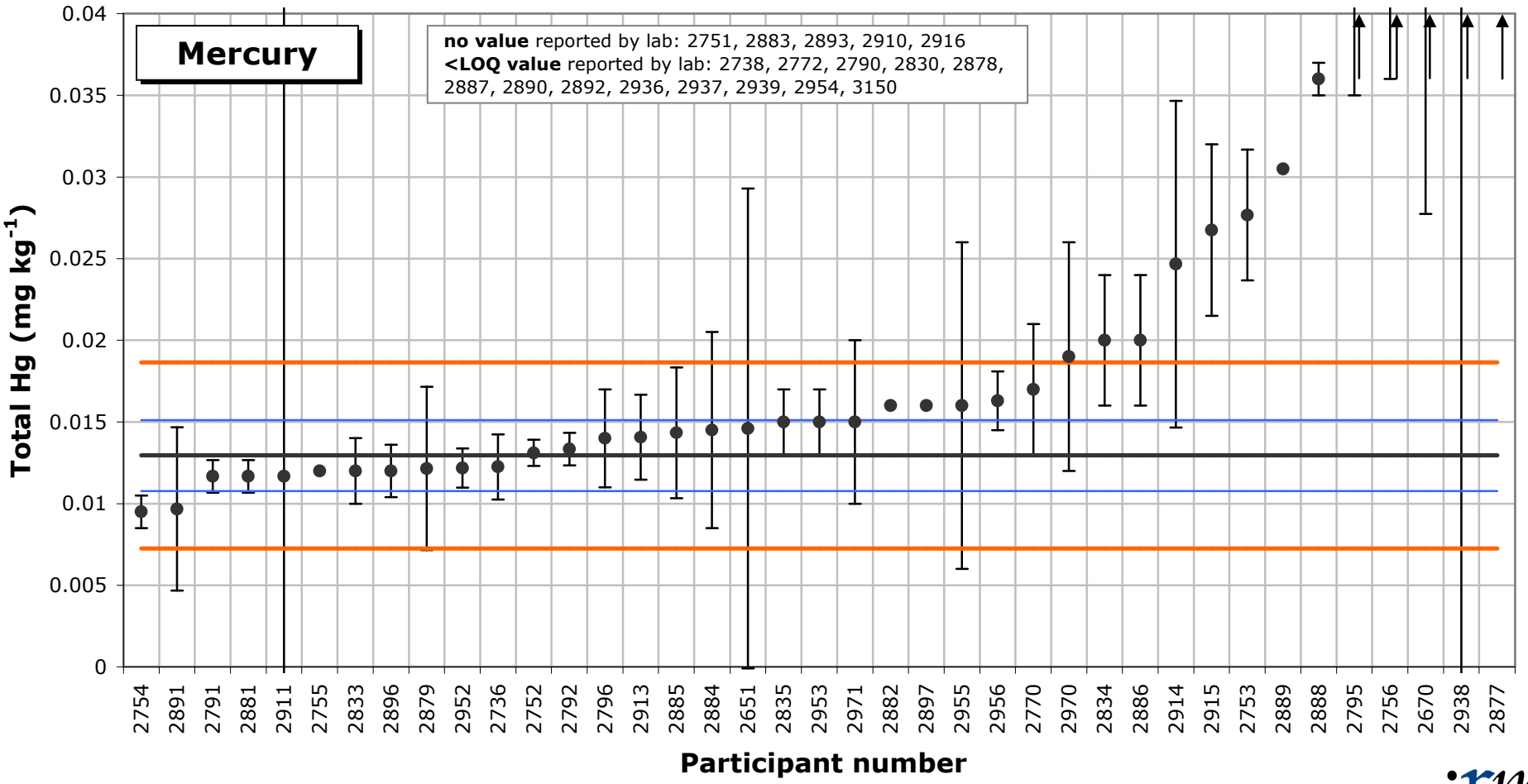


This graph displays all revised measurement results and their associated uncertainties. The uncertainties are shown as reported.
The thick black line corresponds to Xref, the blue lines mark the boundary of the reference interval (Xref ± 2uref), and the orange lines that of the target interval (Xref ± 2σ).

Annex 10 : Results for Mercury

X_{ref} = 0.0129 and U_{ref} = 0.0026; all values are given in (mg kg^{-1})

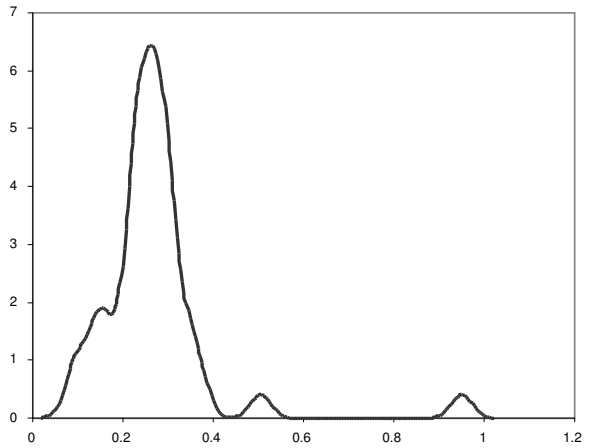
Part Nr	x1	x2	x3	x4	Ulab	k	Mean	ulab	Technique	z	zeta
2651	0.0153	0.0145	0.0140		0.0147	2	0.0146	0.0074	ICP-MS	0.6	0.2
2670	0.065	0.065	0.048	0.046	0.028	2	0.056	0.014	ICP-AES	15.1	3.0
2736	0.012	0.011	0.013	0.013	0.002	2	0.012	0.001	Combustión. CV-AA Amalgama	-0.2	-0.5
2738	<0.03	<0.03	<0.03	<0.03					CV-AAS		
2752	0.0133	0.0132	0.0130		0.0008	0.990	0.0131	0.0008	ICP-MS	0.1	0.1
2753	0.026	0.033	0.024		0.004	2	0.028	0.002	CV-AAS	5.2	6.5
2754	0.009	0.010			0.001	2	0.010	0.001	Mercury autoanalyzer	-1.2	-2.9
2755	0.013	0.012					0.012		Direct Mercury analysis	-0.3	
2756	0.045	0.046	0.044		0.009	2.31	0.045	0.004	TDA-AAS (Automatic Hg analyzer)	11.3	7.9
2770	0.017				0.004	1.96	0.017	0.002	ICP-MS	1.4	1.8
2772	<0.5	<0.5							HG-AAS		
2790	0	0	0	0					FIAS, HG-AAS		
2791	0.012	0.011	0.012		0.001	2	0.012	0.001	Amalgamation-AAS	-0.4	-1.1
2792	0.01	0.02	0.01		0.001	2	0.01	0.001	CV-AAS	0.1	0.3
2795	0.065	0.024			0.009	2	0.044	0.005	ICP-MS	10.9	6.7
2796	0.013	0.015			0.003	2	0.014	0.002	TDA-AAS	0.4	0.6
2830	<0.01	<0.01	<0.01	<0.01					CV-AAS		
2833	0.0124	0.0116			0.002	√3	0.0120	0.001	CV-AAS	-0.3	-0.6
2834					0.004	2	0.020	0.002	HG-AAS	2.5	3.1
2835	0.015	0.015			0.002	√3	0.015	0.001	HG-AAS	0.7	1.3
2877	14.195	14.007	14.001		2.115	√3	14.068	1.221	CV-AAS	4934.4	11.5
2878	<0.001	<0.001	<0.001	<0.001					CV-AAS		
2879	0.0126	0.0117			0.005	2	0.012	0.003	DMA-80	-0.3	-0.3
2881	0.012	0.012	0.011		0.001	√3	0.012	0.001	ICP-AES	-0.4	-1.0
2882	0.017	0.015					0.016		ICP-MS	1.1	
2884	0.015	0.014			0.006	2	0.015	0.003	ICP-MS	0.5	0.5
2885	0.014	0.015	0.014		0.004	2	0.014	0.002	CV-AAS	0.5	0.6
2886	0.021	0.019			0.004	2	0.020	0.002	FIMS	2.5	3.1
2887	<0.05	<0.05	<0.05						ICP-MS		
2888	0.036				0.001	√3	0.036	0.001	HG-AAS	8.1	18.7
2889	0.030	0.031					0.031		CV-AAS	6.2	
2890	<0.015	<0.015	<0.015	<0.015					ICP-MS		
2891	0.00963	0.00982	0.00957		0.005	2	0.00967	0.003	AMA	-1.1	-1.2
2892	<0.07	<0.07	<0.07	<0.07					CV-AAS		
2896	0.0127	0.0118	0.0115		0.0016	2	0.0120	0.0008	CV-AAS	-0.3	-0.7
2897	0.014	0.020	0.014				0.016		CV-AAS	1.1	
2911	0.013	0.011	0.011		11.4	2	0.012	5.7	ICP-MS	-0.4	0.0
2913	0.0138	0.0151	0.0133		0.0026	2	0.0141	0.0013	LECO AMA 254 Analyser	0.4	0.7
2914	0.028	0.026	0.020		0.01	2	0.025	0.01	HG-AAS	4.1	2.3
2915	0.027	0.030	0.022	0.028	0.005	2	0.027	0.003	HG-AAS	4.8	4.9
2936				<0.01					ICP-MS		
2937	<0.05	<0.05	<0.05	<0.05					ICP-MS		
2938	0.11	0.11	0.03	0.04	25	√3	0.07	14	CV-AAS	20.9	0.0
2939	<0.05	<0.05							LECO Mercury Analyser		
2950	0.010	0.007	0.010		0.0003	√3	0.009	0.00014	Direct Mercury Analyzer (DMA)	-1.4	-3.6
2952	0.0115	0.0131	0.0120	0.0121	0.0012	2	0.0122	0.0006	Direct Mercury Analyzer	-0.3	-0.6
2953	0.016	0.014	0.015		0.002	2	0.015	0.001	ICP-MS	0.7	1.4
2954	<0.06	<0.06	<0.06	<0.06					ICP-OES		
2955	0.016	0.015	0.018	0.015	0.01	√3	0.016	0.01	FIMS-AAS	1.1	0.5
2956	0.0158	0.0168			0.0018	0.9072	0.0163	0.0020	CV-AFS	1.2	1.5
2970	0.019	0.023	0.018		0.007	2	0.019	0.004	CV-AAS	2.1	1.7
2971	0.017	0.014	0.014		0.005	2	0.015	0.003	AAS AMA-254	0.7	0.8
3150	<0.3								ICP-OES		



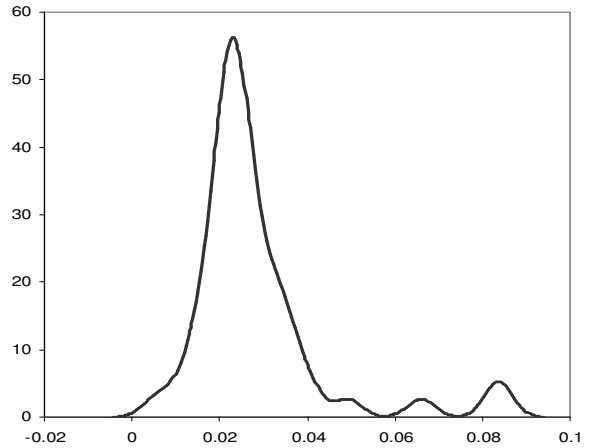
This graph displays all revised measurement results and their associated uncertainties. The uncertainties are shown as reported.
The thick black line corresponds to Xref, the blue lines mark the boundary of the reference interval (Xref ± 2uref), and the orange lines that of the target interval (Xref ± 2σ).

Annex 11 : Kernel densities

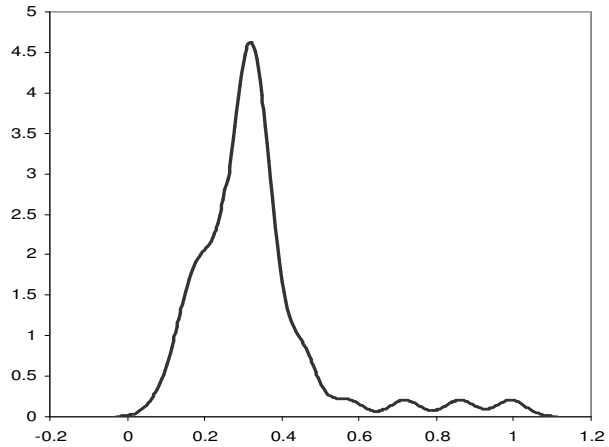
Kernel densities [mg kg^{-1}]



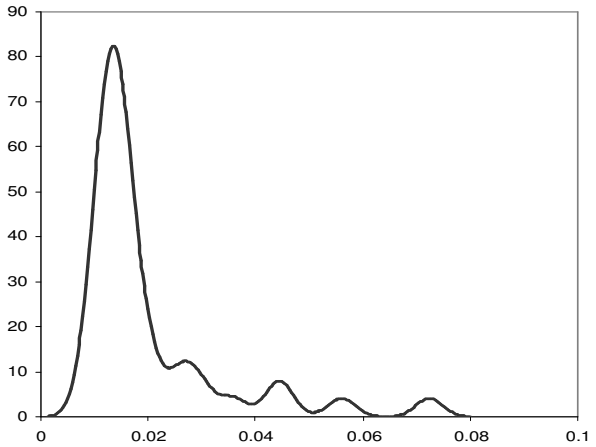
Arsenic



Cadmium



Lead



Mercury

Annex 12 : Summary of scorings

Part Nr	Arsenic		Cadmium		Lead		Mercury	
	z	zeta	z	zeta	z	zeta	z	zeta
2651	-1.1	-1.5	-0.9	-1.5	-0.3	-0.3	0.6	0.2
2670	-1.0	-0.7	-3.9	-7.1	-3.5	-4.4	15.1	3.0
2736							-0.2	-0.5
2738	1.9	1.4	-2.4	-4.6	-3.2	-4.2		
2751			0.2		-2.8			
2752	-0.5	-1.6	-1.0	-3.6	-0.2	-0.3	0.1	0.1
2753	-1.5	-3.1	0.1	0.1	0.1	0.1	5.2	6.5
2754			-2.9	-2.3	0.2	0.2	-1.2	-2.9
2755	-3.0		-2.1		-2.5		-0.3	
2756	-2.9		-1.6	-3.5	-3.5	-3.9	11.3	7.9
2770	0.6	1.9	1.7	4.1	1.3	1.9	1.4	1.8
2772					2.9			
2790	-1.4	-4.0	-0.9	-2.7	-1.3	-2.0		
2791	-3.2	-7.9					-0.4	-1.1
2792	-0.4	-0.6			14.5	24.9	0.1	0.3
2795	5.4	4.1			3.1	3.0	10.9	6.7
2796	-1.1	-1.3	-1.2	-1.1	-2.0	-2.2	0.4	0.6
2830					-3.6	-5.3		
2833	-1.5	-4.3	0.7	1.5	-0.1	-0.2	-0.3	-0.6
2834	-1.4	-4.0	-1.6	-4.0	-0.9	-1.6	2.5	3.1
2835	-1.2	-3.2	-1.0	-3.2	0.3	0.4	0.7	1.3
2877					2.7	3.4	4934.4	11.5
2878	-4.6		-1.8		-1.6			
2879							-0.3	-0.3
2881			1.2	2.6	-1.0	-1.7	-0.4	-1.0
2882	-0.6		-0.2		-0.1		1.1	
2883			3.4		-4.8			
2884	0.9	3.4	-0.1	-0.6	0.5	0.9	0.5	0.5
2885	-0.6	-1.2	-0.5	-0.8	-0.4	-0.5	0.5	0.6
2886	0.1	0.2	1.0	1.2	0.6	0.8	2.5	3.1
2887	0.3	0.8			3.1	3.3		
2888			-5.3	-31.3	5.4	5.9	8.1	18.7
2889			-0.2				6.2	
2890	-0.8	-1.6	-1.6	-7.5	0.5	0.5		
2891	1.6	1.1	0.5	0.4	1.1	0.6	-1.1	-1.2
2892	-0.2	-0.3	-2.5	-5.6	0.0	-0.1		
2893	2.2							
2896	0.2	0.3	-0.3	-1.3	-0.2	-0.3	-0.3	-0.7
2897	16.1		1.8		1.1		1.1	
2910			14.6	11.1				
2911	-0.8	0.0	2.3	0.0	0.8	0.0	-0.4	0.0
2913							0.4	0.7
2914	-2.9		-0.9	-1.2	-2.0	-2.3	4.1	2.3
2915	-4.2	-8.3	-0.7	-0.7	-2.6	-3.6	4.8	4.9
2916			3.2	3.6	11.6	10.4		
2936	0.3	0.3	-0.3	-0.5	0.0	0.0		
2937	0.5	0.3	5.8	0.5	0.3	0.4		
2938	0.2	0.0	14.4	0.0	1.3	0.0	20.9	0.0
2939			2.3	2.0				
2950	0.6	3.0	1.7	13.4	-2.7	-4.8	-1.4	-3.6
2952	-0.7	-2.1	-0.9	-3.3	-0.6	-0.9	-0.3	-0.6
2953	0.1	0.2	-1.3	-4.5	-0.4	-0.6	0.7	1.4
2954	-2.0	-4.4			0.7	1.2		
2955			-0.7	-0.5	1.1	0.8	1.1	0.5
2956	1.2	0.7	-0.8	-2.6	0.1	0.2	1.2	1.5
2970	-0.3	-1.3	-1.0	-3.6	-0.2	-0.3	2.1	1.7
2971	0.0	-0.1	-3.1	-6.0	-1.8	-2.1	0.7	0.8
3150	-3.8		10.0	11.3	8.6	4.8		

Annex 13 : Experimental details

Part Nr	Official Method?	Which one?	Sample pretreatment	Digestion step	Extraction / separation step	Instrument calibration
2651	yes	EN 13805 mod (5 ml HNO3 instead of 3ml)				
2670	yes	EPA 6010C				
2736	no		no	combustion		
2738	yes					
2751	no		cenizas			
2752	yes					
2753	yes	Cd,Pb -EN 14084; As -EN 14546; Hg -EN 13806				
2754	no		None	Microwave (nitric acid + hydrogen peroxide)	None	Blank + 4 calibrators
2755	yes					
2756	no		Homogenisation	Nitric Acid/idrogen peroxide - Microwave Oven		Calibration curve obtained by means of certified analytical standard
2770	no		Acid digestion in mix (5ml of Nitric Acid, 1.5ml of hydrogen peroxide and 1.5 ml of ultrapure water) at atmosferic pressure	mineralization in Acid solution assisted by micro-wave in closed-teflon vessel. Mineralization cycle is about 60 minutes and the mineralization solution is the same used for pre-treatment step.	Not applicable	5 standard solution in 2% Nitric acid (included blank solution) of appropriate concentration.
2772	no		None	Microwave digestion in nitric acid and hydrogen peroxide	None	Standards calibration
2790	yes					
2791	no					
2792	yes	AOAC (1990) 15th Ed. pp,42, 84, 237, 312, 498, 708.& AAS cook book section1.2000				
2795	no		homogenise	microwave digestion	n.a.	calibration using certified standard solutions
2796	yes	EN-ISO 14084				
2830	yes	EN 1483 / EN-ISO 17294-2				
2833	yes	methods published by § 64 LFGB (Germany)				
2834	yes	DIN EN 13805:Juni 2002; DIN EN ISO 17 294-2 2005; DIN EN 1483:2007				
2835	yes					
2877	yes	SLMB-Method (Swiss Food Authority)				

Part Nr	Official Method?	Which one?	Sample pretreatment	Digestion step	Extraction / separation step	Instrument calibration
2878	no		No	0.5 g of sample in 10 ml of NO3H hiperpure + 1ml of H2O2	no	calibration with blank and standard solutions
2879	no		No	No	No	calibration curve
2881	yes	Cd = EN ISO 5961, PB = DIN 38406-6, Hg = EN 1483				
2882	yes					
2883	yes	EN 14082				
2884	yes	ASU § 64 LFGB L-00.00 19/1; DIN EN ISO 17294 Part 1 and 2 (E 36, E29)				
2885	yes	§64 LFGB: L 00.00-19/4 (2003) und DIN EN ISO 17294 (2007)				
2886	yes	afgeleid van NBN EN 14084				
2887	no		none	microwave with aqua regia	made to volume only	External Standard Calibration + Internal Standard addition to samples
2888	no		Drying	microwave with acid mixture	no	external calibration
2889	yes	Modified based on 999.10 AOAC 18th edition				
2890	no			digest with concentrated nitric acid temperature programmed digestion block	dilute to known volume including internal standard	5 calibration standards
2891	yes	linear	mixing	micro wave assisted	nitric acid	yes
2892	no		Homogenation	microwave digestion		external calibration
2893	yes	Pb/Cd (EC 152/2009; ANNEX IV; METHOD C)				
2896	yes	§ 64 of the German Food and Feed Code (LFGB)				
2897	no	ISO 8070				
2910	yes					
2911	no					
2913	no		Hg - none, Pb and Cd sample ashed at 425±25°C	Hg - none. Pb and Cd taken up in hydrochloric and nitric acid and made to volume.	Hg - none	Hg - carried out by AMA analyser. For Pb and Cd calibrated with standards prepared from 1000mg/l Spectrosol solutions.
2914	no			microwave digestion with nitric acid and hydrogen peroxide		calibration curve
2915	no		None	Microwave digestion with HNO3 and H2O2	None	acqueous standards and QC samples
2916	yes	EN 14082:2003				

Part Nr	Official Method?	Which one?	Sample pretreatment	Digestion step	Extraction / separation step	Instrument calibration
2936	no		250mg sample + HNO3 + H202 + H20	Microwave ramp to- and hold at 200℃ / 1h	Dilution in H20	4 point
2937			None	Pb, Cd, As Microwave digestion using nitric acid. Hg block digest	none	5 point calibration
2938	yes	SFS-EN ISO 17294:2005				
2939						
2950	yes					
2952	yes	EN 14083 (Pb&Cd); EPA 7473 (Hg); EN 14627 (As)				
2953	yes	Digestion: §64 LFGB L 00.00-19/1; Measurement: EN ISO 17294-2				
2954	no		Hand blend and weighed out into digestion vessels.	Microwave oven digestion with conc. HNO3. H2O2 added for mercury samples.		ICP-OES calibrated for As, Cd, Hg, Pb 0 - 100 ppb.
2955	yes					
2956	yes	LFGB § 64 L00.00-19/1; 19/3; 19/4				
2970	no			Microwave with HNO3/H2O2		external calibration
2971	yes	EN 14546, EN14084, Manual AMA 254				
3150	yes					

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Abstract
The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre (JRC), a Directorate-General of the European Commission, operates the International Measurement Evaluation Programme IMEP. It organises interlaboratory comparisons (ILC's) in support to EU policies. This report presents the results of an ILC which focussed on the determination of total As, Cd, Pb and Hg in food supplements relying on Commission Regulations 333/2007 and 1881/2006.

The test material used in this exercise was the candidate standard reference material (SRM) SRM 3256, of the National Institute of Standards and Technology (NIST), the matrix being green tea food supplement. The material was labeled at IRMM and dispatched to the participants on the first week of June 2009. Each participant received two sachets containing approximately 2.5 g of test material each. Sixty-two participants from twenty countries registered to the exercise of which 46 reported results for total Cd, 50 for total Pb, 42 for total As and 40 for total Hg. The assigned values were the reference values as provided by NIST.

The uncertainties, u_{ref}, of the respective assigned values were also provided by NIST. Participants were invited to report the uncertainty on their measurements. This was done by 49 of the 58 laboratories having submitted results in this exercise.

Laboratory results were rated with z and zeta scores in accordance with ISO 13528. Standard deviation for proficiency assessment (also called target standard deviation) was fixed to 15 % for total Cd, Pb and As by the advisory board of this ILC, on the basis of the outcome of previous ILC's, and 22% for mercury based on the modified Horwitz equation.

The outcome of the exercise was altogether positive, with over 60 % of the participants reaching satisfactory scores for both types of scorings for almost all elements.

The mission of the JRC is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.

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