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IMEP-34: Heavy Metals in Toys according to EN 71-3:1994

Interlaboratory Comparison Report

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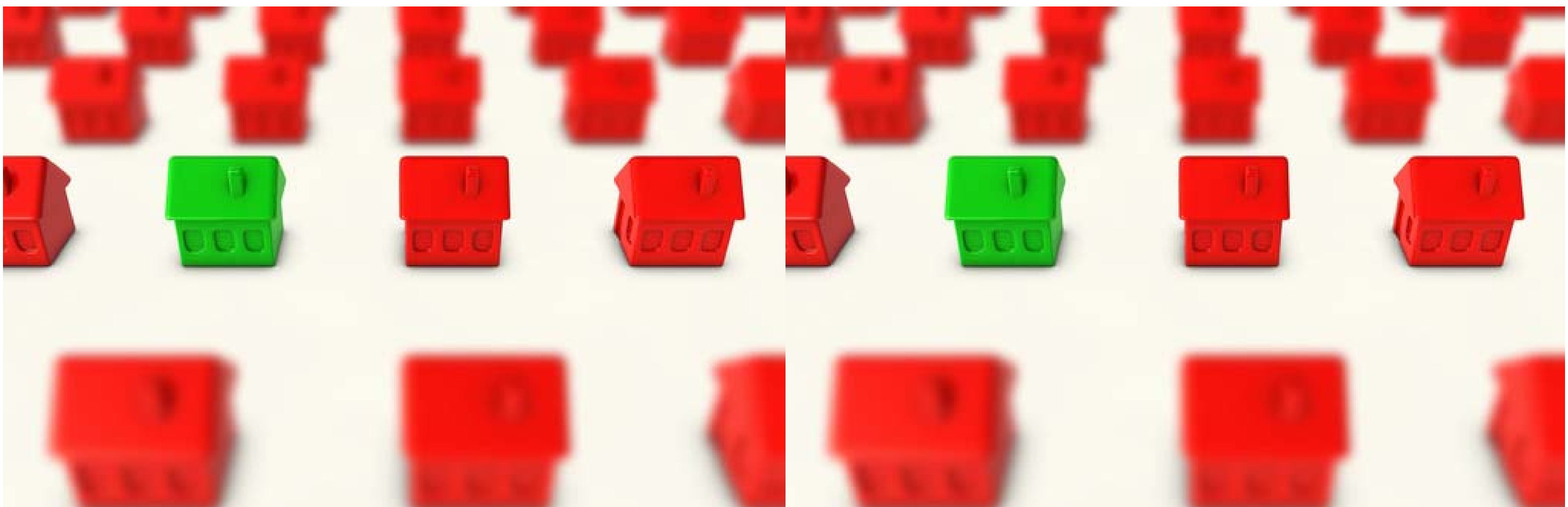

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Summary

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 soluble antimony (Sb), arsenic (As), barium (Ba), cadmium (Cd), chromium (Cr), lead (Pb), mercury (Hg), and selenium (Se) according to European Standard EN 71-3:1994.

The principle of the procedure in EN 71-3:1994 [1] consists in the extraction of soluble elements from toy material under the conditions simulating the material remaining in contact with stomach acid for a period of time after swallowing.

Fifty eight participants from twenty six countries registered to the exercise, of which 54 reported results for As, Sb, Ba, Se and Hg and 58 for Cr, Pb, and Cd, respectively.

The test item used was a certified reference material (CRM 623, comminuted paint flakes from alkyd resin paint), certified in 1998, which is not included anymore in the CRM catalogue. The validity of the certified values was assessed using some expert laboratories in the field. In most of the cases the results reported by the certifiers were not in agreement with the CRM reference values. The mean of the means reported by the expert laboratories was used as assigned value for the different measurands. The results reported by the expert laboratories for mercury were very scattered (RSD = 37.6 %). No assigned value could be attributed for mercury and therefore no scores were provided to the participants for this measurand.

The associated uncertainties of the assigned values were obtained following the ISO GUM [2]. Furthermore, participants were invited to report their measurement uncertainties. This was done by all laboratories having submitted results in this exercise.

Laboratory results were rated with *z*- and zeta (*ζ*-) scores in accordance with ISO 13528 [3]. The standard deviations for proficiency assessment were based on the analytical correction laid down in EN 71-3:1994.

The outcome of the exercise shows an improvement on the overall performance of the participants when compared to IMEP-24 [4] (a proficiency test for heavy metals in toys run in 2009 in which the same European standard was followed), particularly for cadmium, lead and to a lesser extent, for selenium and chromium. The share of satisfactory z-scores ranged from 65 to 79 %.

Contents

1 Introduction

Technological developments in the toys market and on the scientific knowledge have raised issues regarding the safety of toys. Increased concerns from consumers lead to a revision of the Directive 88/378/EEC [5]. The recently adopted Directive for the safety of toys (Directive 2009/48/EC, [6]) includes maximum migration limits for a number of trace elements (aluminium, antimony, arsenic, barium, boron, cadmium, chromium (III), chromium (VI), cobalt, copper, lead, manganese, mercury, nickel, selenium, tin, organic tin and zinc).

To allow toy manufacturers and other economic operators sufficient time to adapt to the requirements lay down by this Directive on chemical requirements, a transition period of four years is provided in which Part 3 of Annex II of Directive 88/378/EEC [5] relating to migration limits of elements is still applicable. The standard to be applied for the determination of extractable elements in toys is the European standard EN 71-3:1994 [1].

The requirements set up in the European standard EN 71-3:1994 are for the migration of trace elements from the following toy materials: coatings, polymeric and similar materials, paper and paper board, textiles, glass/ceramic/metallic materials, materials intended to leave a trace, pliable modelling materials, paints and other materials [1]. The material of interest for this interlaboratory comparison is a comminuted paint from alkyd resin paint, hence a powder-like toy material (as defined in Directive 2009/48/EC, [6]).

Concerned trace elements are antimony (Sb), arsenic (As), barium (Ba), cadmium (Cd), chromium (Cr), lead (Pb), mercury (Hg), and selenium (Se). Their migration from toys should comply with the limits listed in Table 1 when tested according to the procedure given in the European standard. An analytical correction is allowed for each element and is listed in the same table. The analytical result can be reduced by the given percentage when the analytical result equals or exceeds the set limit.

Table 1 summarises the maximum migrated limits (from toys or their components) as set in the European legislation.

Table 1 – Trace elements and their maximum limits (in mg kg^{-1}) as set in European legislation on toys (in dry, brittle, powder-like toy material)

Directive	Sb	As	Ba	Cd	Cr	Pb	Hg	Se
2009/48/EC [6]	45	3.8	4500	1.9	37.5[a]	13.5	7.5	17.5
EN 71-3:1994 [1]	60	25	1000	75	60	90	60	500
Analytical correction [%]	60	60	30	30	30	30	50	60

[a] *as Cr(III)*

IMEP-34 is to be considered as the follow-up exercise of the IMEP-24 [4] and aims to assess the performance of laboratories in measuring the above listed trace elements in toys.

2 IMEP support to EU policy

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

IMEP **disseminates metrology** from the highest level down to the field laboratories. These laboratories can benchmark their measurement result against the IMEP certified 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 results. IMEP integrates the estimate into the scoring, and provides assistance for the interpretation.

IMEP **supports EU policies** by organising interlaboratory comparisons in the frame of specific EU Directives, or on request of a specific Directorate-General. In the case of IMEP-34, it was realised in the context of the former Directive [5] applying the European Standard EN 71-3:1994 and in the context of the new toy safety Directive 2009/48/EC [6] for compliance assessment.

IMEP-34 provided specific **support to the European Co-operation for Accreditation (EA)** in the frame of a Memorandum of Understanding (MoU) on a number of metrological issues, including the organisation of interlaboratory comparisons. National accreditation bodies were invited to nominate a limited number of laboratories for free participation in IMEP-34. The Swedish Board for Accreditation and Conformity Assessment (SWEDAC) liaised between EA and IMEP for this ILC.

3 Scope and aim

Similarly to IMEP-24 [4], IMEP-34 enables laboratories performing tests on toy products to monitor their performance and to compare it with other laboratories from Europe and abroad. Another aim is to identify problems related to technique and methodology. This was particularly interesting in this exercise, since the sample preparation procedure to be applied is known to cause great spread of results. The observation of this spread in former interlaboratory trials actually led to the introduction of the analytical correction into the EN 71-3:1994 [1]. Furthermore, this ILC exercise aims to check if any significant improvement can be detected on the participant's performance since IMEP-24, and to assess the conformity compliance towards the new legislation [6].

4 Time frame

The project started in May 2011. Expert laboratories, which agreed on using their reported values for the establishment of the reference values, were invited to register (Annex 1). The EA coordinator Annika Norling informed the national accreditation bodies. The exercise was publicly announced on the IMEP webpage[1] in the middle of July 2011. In parallel, laboratories specialised in toy safety related analyses were contacted.

Interested laboratories could register till 19th September 2011. Samples were sent out to the laboratories on 10 and 11th October 2011. For all laboratories the deadline for reporting results was 18th November 2011.

5 Invitation, registration and distribution

Invitations for participation were sent to the EA coordinator (Annex 2) for distribution to nominated laboratories. Notified bodies from the NANDO list were sent an email (Annex 3) inviting them to take part in the exercise, after having retrieved their contact information from the NANDO webpage[2]. NANDO lists notified bodies fulfilling the relevant requirements and which can be designated to carry out conformity assessment according to a directive, which in this case is the Toy Safety Directive. Finally, a call for participation was also released on the IRMM website (Annex 4).

Instructions on measurands, sample storage and measurement procedure were sent to the participants in an accompanying letter together with the test items. The letter also contained the individual "code for access" to the result reporting website and the deadline for reporting (Annex 5). The reporting website included a questionnaire to collect additional information related to the experimental work (Annex 6).

5.1 Distribution

The test items were dispatched by IRMM on the 10-11 October 2011 to the certifying laboratories and to the participants. Each laboratory received one package containing the alkyd resin paint in powder form, the 'Confirmation of receipt' form (Annex 7) and an accompanying letter with instructions on sample handling, procedure and timelines (Annex 5).

The dispatch was followed by the courier's parcel tracking system on internet and in most of the cases the sample was delivered within a couple of days. Fifty eight laboratories registered out of which the majority submitted results for most of the measurands. Figure 1 represents the participating countries.

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

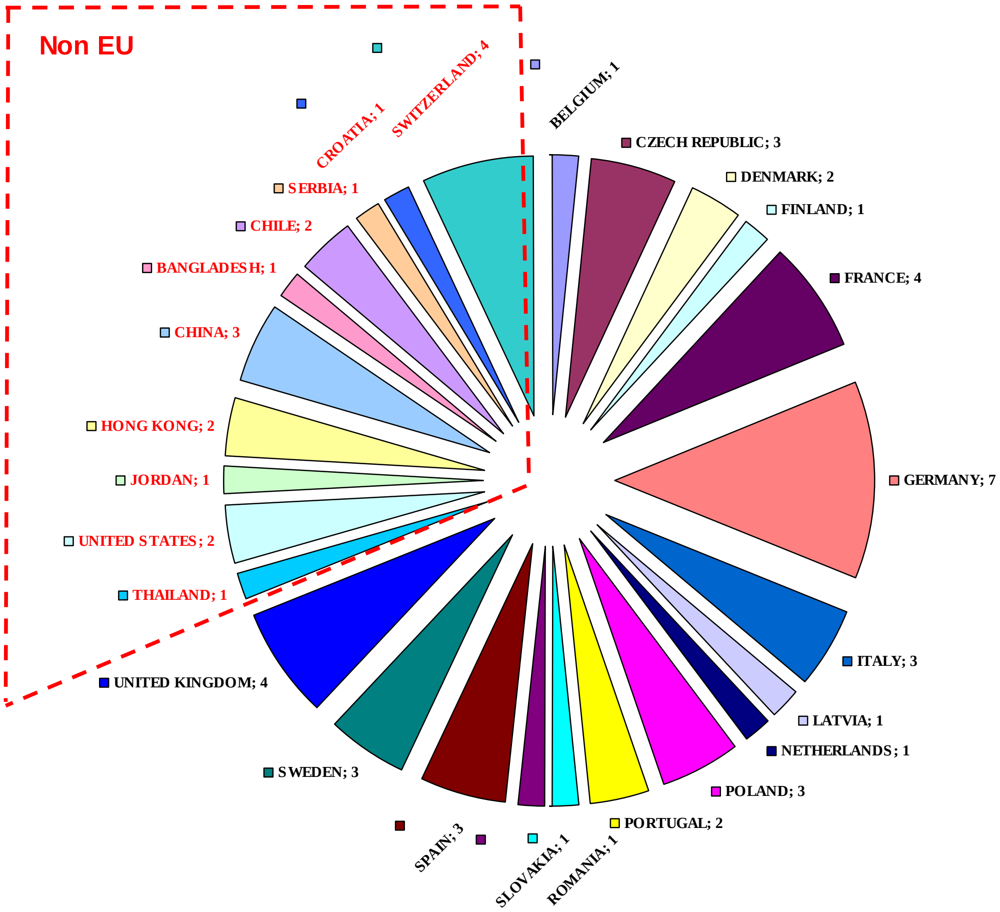

Fig. 1 – Participating countries, number of laboratories (non-EU countries in red)

5.2 Confidentiality

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."*

5.3 Procedure to apply

As this exercise was run to verify the performance of the laboratories when applying the EN 71-3:1994 [1], they were recommended to apply the corresponding procedure. Concerning the quantitative analysis of migrated elements, the standard recommends the use of methods having a detection limit of a maximum of 1/10 of the values to be determined.

[2] http://ec.europa.eu/enterprise/newapproach/nando/

6 Test item

The test item used for this exercise is the certified reference material CRM 623 which consists of 2 g of comminuted paint flakes from alkyd resin paint (in powder form) contained in an amber glass bottle. This material was certified in 1998 for levels of toxic element migration using the method specified in the EN 71-3:1994 [1]. All elements except mercury were certified. The CRM 623 was taken out of sales because of doubts of stability observed during monitoring analysis. The material was designed to be used without any further sieving or processing, hence, all analytical variability introduced by scrapping the paint off from each plate is avoided in the present ILC exercise (in contrast to IMEP-24).

The certification report is not available for the public since the material is not commercialised anymore. However, details about the certification are publically available [7, 8] and are summarised hereafter. The paint was ordered at a specialised paint manufacturing company Trimite Ltd (UK). It was adulterated with 8 toxic elements at concentrations sufficient to yield soluble element concentrations at or around the maximum permissible levels. The paint was produced using dark grey "base" paint and adding a series of "tinters" each containing one of the eight toxic elements. Auto Imagination Ltd (UK) was contacted to spray the completed paint batches onto mild steel panels and to produce the comminuted paint flakes. Mild steels were degreased and abraded on one side by sand blasting. The comminuted paint flakes were produced by spraying the alkyd resin paint onto sheets of plastics. Just before the paint was fully dry, the film of paint was scrapped off and left to dry. Flakes produced were gently comminuted using a water cooled analytical grinder and sieved through a 500 µm mesh size.

6.1 Homogeneity and stability studies

Since the material is withdrawn from the market it was decided to carry out a homogeneity study. Two certifying laboratories investigated the homogeneity of the test item using (i) neutron activation analysis with k_0-standardization (k_0-NAA) for the determination of total content of As, Ba, Cd, Hg, Sb and Se; (ii) inductively coupled plasma coupled with optical emission spectrometry (ICP-OES) for the determination of extractable lead, since k_0-NAA does not allow the determination of lead.

Both laboratories received 10 randomly chosen bottles from the sample set stored at 18 °C and analyses were performed in duplicate following, either the procedure given in EN 71-3:1994 [1] or their own method. Results were evaluated according to ISO 13528 [3] which describe tests to determine whether a ILC test item is adequately homogeneous for its purpose.

Assumption was made that, in case the test item is proven to be homogeneous for the total content, the corresponding soluble (extractable) content would be considered equally homogeneous. The homogeneity results can be found in Annex 8.

The test item used in this PT is similar to the CRM 620 used in the frame of the IMEP-24 project. As CRM 620 was proven to be stable, no additional short-term stability study was deemed necessary for the CRM 623 material.

7 Reference values and their uncertainties

7.1 Target values

By target values is meant the concentration of trace elements aimed at when producing the material. In this exercise they were set by the elements' concentrations of the material available. This material has been specifically produced for the toy safety norm for which the limits are set in EN 71-3:1994 [1] and target values were aimed at being close to these limits. Thus, the material was considered fit-for-purpose.

7.2 Establishing reference values and uncertainties (X_{ref}, U_{ref})

Five expert laboratories were contacted to perform accurate analysis so that their values could be used to either confirm the reference values from the expired certificate, or for the establishment of new reference values. Additionally, a reference value had to be determined for mercury, where no certified value was available. The five expert laboratories were:

- SGS CTS, Chemical Toys (Fr)
- LGC Ltd, Teddington (UK)
- SP Technical Research Institute of Sweden (SE)
- Finnish Customs Laboratory (FI)
- Istituto Italiano per la Sicurezza dei Giocattoli S.r.l., Cabiate – Co (IT)

One of the certifiers reported several "less than X" values (for Sb, As, Cr, Pb and Se), and submitted highly scattered Hg results. The advisory board decided to exclude the results of this certifier from the pool of results used to establish the various assigned values.

Annex 9 presents the results obtained by the remaining four expert laboratories and their expanded uncertainties. These results were generally in good agreement among them (except for Hg), but did not confirm the original certified values. For all the measurands, except mercury, the advisory board decided to set the assigned value (X_{ref}) as the average values derived from the results reported by the certifiers ($X_{Exp} \pm U_{Exp}$), instead of the original certified values. The associated combined uncertainty (u_{ref}) is calculated by propagating contributions (standard deviations) from characterisation (u_{Char}) and homogeneity (u_{Hom}) as follows [9]:

$$u_{ref} = \sqrt{u_{Char}^2 + u_{Hom}^2} \qquad \text{Eq. 1}$$

where the uncertainty of characterisation u_{Char} is calculated from the uncertainties reported by the expert laboratories (u_{Exp}) following the ISO GUM approach [2, 10]:

$$u_{Char} = \sqrt{\left(\sum_{i=1}^{n} u_{Exp}^2\right) \Big/ n} \qquad \text{Eq. 2}$$

where n refers to the number of accepted data sets.

No assigned value was established for Hg, and therefore no laboratory performance was evaluated for this element.

Table 2 – Assigned values, their associated uncertainties and $\hat{\sigma}$ for each element

Measurand	Certifier C 2		C 17		C 36		C 38		X_{ref}	u_{Char}	u_{Hom}	u_{ref}	U_{ref} (k=2)	$\hat{\sigma}$ (%)
	X_{Exp}	U_{Exp}	X_{Exp}	U_{Exp}	X_{Exp}	U_{Exp}	X_{Exp}	U_{Exp}						
Antimony (Sb)	12.36	1.0	9.29	1.0	8.4	2.5	8.3	2.0	9.6	0.4	0.2	0.5	1.0	30
Arsenic (As)	7.16	0.9	8.16	0.8	5.8	1.1	4.4	0.6	6.4	0.2	0.1	0.2	0.5	30
Barium (Ba)	96.11	3.5	94.8	17.0	92.7	15.0	84.3	8.0	92.0	3.1	2.7	4.1	8.2	15
Cadmium (Cd)	31.96	2.2	27.3	4.8	28.7	10.0	18.6	3.6	26.6	1.5	0.6	1.6	3.2	15
Chromium (Cr)	7.57	0.6	7.42	1.3	7.1	1.5	6.2	0.3	7.1	0.3	0.1	0.3	0.6	15
Lead (Pb)	14.32	1.0	12.17	2.1	11.6	3.0	9.1	2.0	11.8	0.5	0.6	0.8	1.6	17
Selenium (Se)	27.0	3.1	24.9	2.5	18.5	2.5	17.2	2.3	21.9	0.7	0.6	0.9	1.8	30
Mercury (Hg)	117.18	24.0	142.7	29.0	49.7	10.0	108.0	13.0	No scoring					

$\hat{\sigma}$ is expressed as a percentage of the respective X_{ref} value.

8 Reported results

8.1 General observations

From the 58 laboratories that registered, all have submitted results together with their associated uncertainties. All except one have completed the associated questionnaire.

Laboratories which have reported "less than X" values were not given any scores. The majority of the participants reported measurement results for all eight elements. Only a very few obvious blunders were reported from one participant, including very low or very high values.

8.2 Measurement results

In IMEP-34, participants were asked to perform three independent results (one replicate from each of the bottles sent to each participant) and to report "the corrected mean". Unfortunately, this sentence seemly led to some confusion because it was understood by many participants as mandatory to correct their mean (using the respective AC as given in Table 1, as requested by EN 71-3) regardless on whether the material was compliant or not with the legislation. The use of the analytical correction (AC) depends on the concentration level found. If below the maximum tolerable limit (X_{EN}) the AC does not need to be applied since the material is already compliant. Hence the "Sample accompanying letter" (Annex 5) should have read in "Reporting of results: The result of each replicate and the corrected mean (if applicable, accordingly to EN 71-3)".

Participants were contacted by the PT coordinator to clarify whether the individual values reported for the three replicates have been corrected or not using the AC. Scores were then provided on the raw data (not corrected) taking the average of the three replicates.

All the results are shown in tables (Annex 10-17) including the reported averaged value, the uncertainty, the technique used, scorings, and the uncertainty evaluation (see below). Additionally Annexes 10 to 17 illustrate, in graphs, all the observed variability and include the Kernel density plots for each element.

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 [11, 12].

The results are generally normally distributed around the assigned value, or at least not much deviating from it. Some sub-populations can be observed in the Kernel plots mainly due to punctual very high or very low results.

8.3 Scores and evaluation criteria

Individual laboratory performance is expressed in terms of *z*- and (*ζ*-) zeta-scores in accordance with ISO 13528 [3] and the IUPAC International Harmonised Protocol [13]:

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

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 (accordingly to ISO 17043, [14]):

Satisfactory result when $|z\text{- or }\zeta\text{-score}| \leq 2$,

Questionable result when $2 < |z\text{- or }\zeta\text{-score}| < 3$ and,

Unsatisfactory result when $|z\text{- or }\zeta\text{-score}| \geq 3$

The *z*-score indicates whether a laboratory is able to perform the measurement in accordance with what can be considered as good practice within the EU. The standard deviation for proficiency testing $\hat{\sigma}$ is an estimate of the expected / required variability of the trial. It has to be determined for each ILC individually. In this exercise, it was established based on the analytical correction (AC) given in EN 71-3:1994. These were interpreted as expanded uncertainties. Thus, $\hat{\sigma}$ was set as half the AC (for each trace element, except for Pb, where it was set as 0.17 X_{ref}), assuming a confidence interval of 95 %. Table 2 summarises all reference values for the present PT exercise (X_{Exp}, X_{ref}, U_{ref}, $\hat{\sigma}$).

The IUPAC International Harmonised Protocol [13] suggests that participants can apply their own $\hat{\sigma}$ and recalculate the scores if the purpose of their measurements is different.

The *ζ*-score provides an indication of whether the estimate of uncertainty is consistent with the laboratory's deviation from the reference value [3, 13]. It is calculated only for those results that were accompanied by an uncertainty statement. The interpretation is similar to the interpretation of the *z*-score. An unsatisfactory *ζ*-score may be caused by an underestimated uncertainty or by a large deviation from the reference value.

The standard uncertainty of the laboratory (u_{lab}) was calculated as follows; if an expanded uncertainty was reported, it was divided by the coverage factor *k*. If no coverage factor was provided, the reported uncertainty was considered as the half-width of a rectangular distribution. The reported uncertainty was then divided by $\sqrt{3}$, in accordance with recommendations issued by Eurachem and CITAC [15].

Uncertainty estimation is not trivial; therefore an additional assessment was provided to each laboratory reporting uncertainty, indicating how reasonable their uncertainty estimate is. The standard uncertainty from the laboratory (u_{lab}) is most likely to fall in a range between a minimum uncertainty (u_{min}), and a maximum allowed (u_{max}), (case "a"). u_{min} is set to the standard uncertainty of the reference value ($u_{min} = u_{ref}$). It is unlikely that a laboratory carrying out the analysis on a routine basis would measure the trace element with a smaller uncertainty than the expert laboratories chosen to establish the assigned value. u_{max} is set to the target standard deviation ($\hat{\sigma}$) accepted for the PT ($u_{max} = \hat{\sigma}$). If u_{lab} is smaller than u_{ref} *(case "b")* the laboratory may have underestimated its uncertainty.

Such a statement has to be taken with care as each laboratory reported only measurement uncertainty, whereas the uncertainty of the reference value, generally, also includes contributions of homogeneity and stability (when applicable). If those are large, measurement uncertainties smaller than u_{ref} are possible and plausible. If $u_{lab} > \hat{\sigma}$ (case "c"), the laboratory may have overestimated the uncertainty. An evaluation of this statement can be made when looking at the difference of the reported value and the assigned value: if the difference is small and the uncertainty is large, then overestimation is likely. If, however, the deviation is large but is covered by the uncertainty, then the uncertainty is properly assessed but large. It should be pointed out that $\hat{\sigma}$ is only a normative criterion if set down by legislation.

8.4 Laboratory results and scorings

Scores were calculated with the raw data for all participants (taking the average of the three "non-corrected" replicates). Those having reported no value or a "less than" value were not included in any further statistical evaluation.

A large percentage of participants reported satisfactory measurement results (ranging from 65 to 79 % in *z*-score). Unsatisfactory *z*-scores ranged from 11 to 22 % (Figure 2).

This overall performance is more satisfactory than for IMEP-24. The percentage of satisfactory results in IMEP-24 was 44 % and 43 % for Cd and Pb, respectively. This comparison is valid as the same $\hat{\sigma}$ was used in both IMEP rounds.

The situation is slightly different for the *ζ*-scores (Figure 2). Only two elements (Ba and Cd) had equal or over 50 % of the participants getting satisfactory scores. That means that although the results reflected by the *z*-scores are generally good, there is an obvious problem with the estimation of the uncertainty for some elements, resulting in a high number of unsatisfactory *ζ*-scores. Annex 18 summarises all the scores per participant.

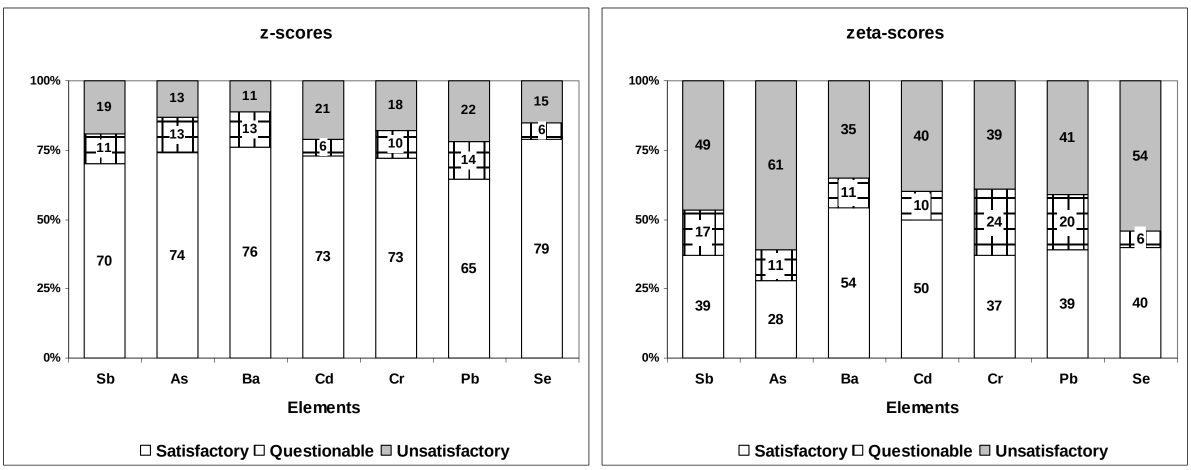

Fig. 2 – Overview of scores (in %)

All participants provided an uncertainty estimate, and most of these estimates were accompanied by a coverage factor. This is encouraging, but contrasts with the relatively modest proportion of results with a satisfactory ζ-score. Considering that only 23 % of the participants stated in the questionnaire that they usually report the uncertainty to their customers, one might think that this is the reason for the lack of experience in uncertainty estimation and reporting. When plotting the scores as a function of the reporting / non-reporting to customers, there is a trend for those reporting uncertainties to their customers to perform better (54 % of those who report uncertainty to their customers got a satisfactory ζ-score).

Uncertainty evaluation, for each element, is given in Annex 10 to 17. An overall evaluation is summarised in Table 3. Only a small percentage of participants have overestimated their uncertainty (case "c"). The percentages of participants who have estimated their uncertainty lower than the respective u_{ref} (case "b") ranges from 44 % (Se) to 67 % (Cd). It is worth mention that the contribution arising from the homogeneity is included in the estimation of u_{ref} but is not reflected in u_{lab}. The percentage of participants having reported an uncertainty value within u_{ref} and $\hat{\sigma}$ (case "a") ranges from 26 % (Cd) to 54 % (Se).

As conclusion, participants are advised to verify their ζ-scores, and review the principles of uncertainty estimation described in the ISO GUM [2] and in related guidance for the field of analytical chemistry, e.g. the EURACHEM / CITAC Guide [15].

Table 3 – Uncertainty evaluation for each element

	Uncertainty score (%)		
Measurand	**a**	**b**	**c**
Antimony (Sb)	**50**	**46**	**4**
Arsenic (As)	**44**	**52**	**4**
Barium (Ba)	**42**	**54**	**4**
Cadmium (Cd)	**26**	**67**	**7**
Chromium (Cr)	**47**	**45**	**8**
Lead (Pb)	**31**	**57**	**12**
Selenium (Se)	**54**	**44**	**2**

Where: "a": $u_{ref} \leq u_{lab} \leq \hat{\sigma}$; "b": $u_{lab} < u_{ref}$; "c": $u_{lab} > \hat{\sigma}$

8.4.1 Mercury

The analysis of Hg in the test item seems challenging. The Kernel density plot shows a bi-modal distribution of reported results (Annex 17). The same trend was observed by one of the certifiers when having four different analysts to perform their measurements on the three independent replicates, and in the results reported by the other four expert laboratories. The advisory board decided not to assign a reference value and not to perform any evaluation (scoring) for this element.

8.5 Conformity assessment according to the European legislation on toys

Participants were asked in the questionnaire whether they '*would accept or reject the entrance of the material on the market*' according to Directive 88/378/EEC and to the new toy safety Directive 2009/48/EC.

As for all the elements the assigned values are below the maximum limit (Table 1), the material is compliant with Directive 88/378/EEC (maximum migration limits as set by EN 71-3:1994). Twenty eight participants stated that the material is compliant to this Directive, while 20 stated the opposite; 4 participants did not reply to this question.

According to Directive 2009/48/EC this powder-like toy material should have been judged non compliant, since the assigned values (Table 2) are larger than the maximum migration limits for several trace elements (As, Cd and Se, see Table 1). Most of the reported results largely exceeded these limits. Nevertheless, 17 participants judged the test item as compliant while 26 considered it as non-compliant; 9 participants did not answer to this question.

In the sample accompanying letter (Annex 5) the sample matrix was defined as *"an alkyd resin paint in powder form"*. It is therefore surprising to see approximately 50 % of laboratories having used the wrong migration limits specified in Directive 2009/48/EC (scraped-off instead of powder-like), to assess the compliance of the test item, hence allowing placing on the market of a non-compliant toy.

Annex 19 presents the participant's answers regarding the conformity assessment to both toy safety Directives.

8.6 Further information extracted from the questionnaire

Almost all participants completed the questionnaire, although few of them skipped a large part of it. Since this exercise was carried out using the EN 71-3:1994, many questions were related to the sample preparation. All laboratories followed the EN 71-3:1994 for the required analysis; L27 deviated slightly from the standard and used a filter with different porosity.

Thirteen participants sieved the test sample. This experimental procedure increased the extraction efficiency and the recovery of all the elements.

The majority of the participants weighted 0.5 g of test sample per replicate, applied the recommended temperature of 37 °C during sample preparation and performed the analysis on the same day of sample processing.

For the uncertainty estimate, several participants gave various combinations of the given choices. Twenty-seven participants estimated their uncertainty from precision studies (replicates), 26 from in-house validation studies, 15 estimated their uncertainty following

ISO GUM approaches, 7 based on judgement, 3 from interlaboratory comparison data and finally 6 using a known uncertainty from the standard method.

It has to be emphasised that the latter should not be used on its own - the correct implementation of a standard method, in a laboratory, should always be verified by the laboratory applying it.

All except one have a quality system based on ISO 17025. Three have a quality system based on both ISO 17025 and ISO 9000 series and one based on ISO 9000 series. 93 % of the participants are accredited. 68 % of the participants declared to take part in an interlaboratory comparison on a regular basis.

Eighty nine percent of the participants carry out this type of analysis regularly. However, the number of samples analysed by the 52 laboratories who answered to this question varies as can be seen in Table 4 where the number of samples per year is reported.

Seventeen laboratories use a reference material (RM) for this type of analysis (30 %). All of them used the RM for the validation of their measurement protocol while 13 used it for the calibration of their instruments. The RMs used by the participants, are listed in Table 5.

Table 4 – Reported samples analysed per year (in %)

Number of samples per year	< 50	50 - 250	250 - 1000	>1000
Number of laboratories (% of total)	16 (39 %)	10 (19 %)	10 (19 %)	16 (31 %)

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

Lab ID	Which reference material?
C 2	In-house material for method for migration
C17	In-house quality control material is used.
L05	ex Toy test material round 43
L07	CRM Solution
L10	GBW(E)081536
L12	(mono-elemental standards are used for calibration of course)
L15	CRM- Certificate standard with a note concentration of metals
L16	PC-CR4 (in-house SRM)
L18	CRM solution
L23	Multielemental acid solution
L25	Titrisol for each of the eight trace elements (Merck)
L29	Solutions of known metals
L32	Spiked samples
L34	In-house made
L41	made in-house RM
L43	RM: ICP multi-element standard HC 945548, Merck ,CRM: TraceCERT, Fluka analytical (19 elements)
L44	Standard Reference Material for each metal (PANREAC)
L45	Certified reference material (CRM) from which are made internal standards to check the method
L50	not applicable
L51	In-house reference material

For the participants who have declared the use of standard solutions of the trace elements under investigation we wish to recall that standard solutions do not allow the trueness assessment of their method, only a matrix-matched reference material does.

Annex 20 provides a comprehensive list of experimental details stated by the participants.

9 Conclusion

The scatter of the results in IMEP-34 was smaller than in IMEP-24, showing a normal distribution around the reference values for all elements except mercury.

Similarly to IMEP-24, participants' results tend to be lower than X_{ref} in the case of arsenic and selenium, elements known to be difficult to analyse. The reason for these lower results could be attributed to the sample preparation, these elements being very volatile and easy to loose.

Conformity assessment to the two Directives was made. Half of the participants took the right decision regarding the compliance of the test item with legislation, even though about 50 % of the participants would have unduly allowed the test item to enter the European market according to Directive 2009/48/EC.

10 Acknowledgements

The author's wishes to acknowledge the Istituto Italiano per la Sicurezza dei Giocattoli S.r.l., LGC Ltd, SP Technical Research Institute of Sweden, Finnish Customs Laboratory, SGS CTS, Chemical Toys for performing high precision analyses on the test material for the establishment of the assigned values and SCK/CEN for measurements for the homogeneity and stability studies. Franz Ulberth is thanked for revising the manuscript.

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

Organisation	Country
SGS Bangladesh Limited	BANGLADESH
CTIB-TCHN	BELGIUM
Instituto de Investigaciones y Control	CHILE
CESMEC S A	CHILE
Specialized Technology Resources (Shanghai) Limited - Shenzhen Branch	CHINA
Specialized Technology Resources(Shanghai) Ltd.	CHINA
TUV Rhienland (Shanghai) Co., Ltd	CHINA
Institute of Public Health dr.Andrija Štampar	CROATIA
Institut pro testovani a certifikaci	CZECH REPUBLIC
Textilni zkusebni ustav	CZECH REPUBLIC
Technical and Test Institute for construction Prague	CZECH REPUBLIC
Eurofins Miljø A/S	DENMARK
Technological Institute	DENMARK
LNE nommé par EA	FRANCE
INTERTEK	FRANCE
BV CPS France	FRANCE
Hermes Hansecontrol	GERMANY
INDIKATOR GmbH	GERMANY
SLG Prüf- und Zertifizierungs GmbH	GERMANY
Dr. Graner & Partner GmbH	GERMANY
Intertek	GERMANY
PFI Pirmasens	GERMANY
Entwicklungs- und Prüflabor Holztechnologie GmbH (EPH)	GERMANY
Specialized Technology Resources (H.K.) Ltd.	HONG KONG
SGS Hong Kong Limited	HONG KONG
LABORATORIO DI ANALISI PROVE E RICERCHE TESSILI	ITALY
European Certifying Organization S.p.A.	ITALY
Royal Scientific Society	JORDAN
Ltd Latvian Certification Centre	LATVIA
nVWA region north / Nieuwe Voedsel en Waren Authoriteit	NETHERLANDS
Institute for Engineering of Polymer Materials and Dyes	POLAND
Office of Competition and Consumer Protection	POLAND
Polskie Centrum Badań i Certyfikacji S.A	POLAND
CATIM	PORTUGAL
CITEVE - Centro Tecnologico das Industrias Têxteis e Vestuario de Portugal	PORTUGAL
LAREX CNIEP	ROMANIA
Institute for public health Belgrade	SERBIA
VÚTCH-CHEMITEX spol.s r.o.	SLOVAKIA
Centro Analítico Inspección y Control de Calidad de Comercio Exterior (SOIVRE)	SPAIN
AIJU	SPAIN
LGAI TECHNOLOGICAL CENTER	SPAIN
ALS Scandinavia AB	SWEDEN
INNVENTIA AB	SWEDEN
SQTS - Swiss Quality Testing Services	SWITZERLAND
LABORATORIO CANTONALE	SWITZERLAND
Kantonales Laboratorium Baselland	SWITZERLAND
Kantonales Laboratorium Bern	SWITZERLAND
TUV Rheinland Thailand Ltd.	THAILAND
STR (UK) Ltd.	UNITED KINGDOM
City of Edinburgh Council	UNITED KINGDOM
Intertek	UNITED KINGDOM
SGS North America Inc., Consumer Testing Services	UNITED STATES
Consumer Testing Laboratories	UNITED STATES

Abbreviations

AAS	Atomic Absorption Spectroscopy
AC	Analytical Correction
AMC	Analytical Methods Committee of the Royal Society of Chemistry
CITAC	Co-operation for International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CVAAS	Cold Vapour Atomic Absorption Spectrometry
EA	European Co-operation for Accreditation
EC	European Commission
EN	European Standard
ETAAS	Electro Thermal Atomic Absorption Spectrometry
EU	European Union
EURACHEM	A focus for Analytical Chemistry in Europe
FAAS	Flame Atomic Absorption Spectroscopy
GUM	Guide to the Expression of Uncertainty in Measurement
ICP-MS	Inductively-Coupled Plasma Mass Spectrometry
ICP-OES	Inductively-Coupled Plasma Optical Emission 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
NANDO	New Approach Notified and Designated Organisations
MoU	Memorandum of Understanding
SP	Swedish National Testing and Research Institute
SWEDAC	Swedish Board for Accreditation and Conformity Assessment

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Annexes

Annex 1 : Invitation to expert laboratories

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements

Geel, 18 July 2011
JRC.DG.D6/IB/vsc/ARES(2011)/787479

(Name)
(Institution)
(Address)
(Address)
(Postal Code)
(Country)

Dear (Name),

Second intercomparison for trace metals in toys according to EN71-3:1994

You have agreed to participate in the above mentioned proficiency test and that your results could be used for the establishment of the reference value. Please be reminded that in that case a high precision analysis is expected of you and not a routine analysis. It goes without saying that your participation would be free of charge.

As a reminder, the interlaboratory comparison concerns the determination of the eight trace metals whose safety limits are set out by the EU Toy Safety Directive (88/378/EEC) and specified in the harmonised European Standard EN71-3:1994. The elements are antimony, arsenic, barium, cadmium, chromium, lead, mercury and selenium. The test material is comminuted paint from alkyd resin paint.

The registration interface for the exercise was opened and laboratories can register until **16 September 2011**. Distribution of the samples is foreseen for the second half of September 2011, and the foreseen result reporting deadline is **28 October 2011**.

I therefore kindly invite you to register using the following link :

https://irmm.jrc.ec.europa.eu/ilc/ilcRegistration.do?selComparison=740

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

Annex 2 : Invitation to EA to nominate laboratories

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements

Geel, 19 July 2011
JRC.DG.D6/IBa/vsc/ARES(2011)/783627

SWEDAC
Annika Norling
Box 2231
10315 Stockholm
SWEDEN

Dear Annika,

Second intercomparison for trace metals in toys according to EN71-3:1994

The Institute for Reference Materials and Measurements (IRMM) organises a second interlaboratory comparison for the determination of the eight trace metals whose safety limits are set out by the EU Toy Safety Directive (88/378/EEC) and specified in the harmonised European Standard EN71-3:1994. The concerned elements are antimony, arsenic, barium, cadmium, chromium, lead, mercury and selenium. The test material is comminuted paint from alkyd resin paint.
In the frame of the EA-IRMM collaboration agreement, IRMM kindly invites EA to nominate laboratories for free participation. These laboratories must be involved in toy safety evaluation and be familiar with the above mentioned standard, since it will be the method to be applied to the sample. They also 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. The number of nominees should not exceed 2-3 laboratories per country.
Confidentiality of the participants and their results towards third parties is guaranteed. However, IMEP will disclose details of the participants that have been nominated by EA to the EA working group for ILCs in Testing. The EA accreditation bodies may wish to inform the nominees of this disclosure.
Registration of participants is open until **16 September 2011**. Distribution of the samples is foreseen for the second half of September 2011, and the foreseen result reporting deadline is **28 October 2011**.

In order to register, laboratories must:

1. **Enter** their details online:

https://irmm.jrc.ec.europa.eu/ilc/ilcRegistration.do?selComparison=740

2. **Print** the completed form when the system asks to do so and clearly indicate on the printed form that you have been appointed by the European Cooperation for Accreditation to take part in this exercise **otherwise your laboratory will be invoiced 400 EUR for participation** normally applied for non-appointed laboratories.

3. **Send** the printout to both the IMEP-34 and the EA-IMEP-34 coordinators:

IMEP-34 coordinator	**EA-IMEP-34 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-34 Coordinator

Annex 3: Invitation to notified bodies from NANDO list

KORTSEN KONRAD Bibi (JRC-GEEL)

From: BAER Ines (JRC-GEEL)
Sent: 20 July 2011 09:43
To: JRC IRMM IMEP
Subject: IMEP-34 - interlaboratory comparison on trace metals in toys according to EN71-3:1994

Importance: High

To whom it may concern

My name is Ines Baer and I am working at the European Commission - Institute for Reference Materials and Measurements (IRMM), more specifically on the organisation of interlaboratory comparisons (ILC) in the frame of IMEP, the International Measurement Evaluation Programme.

We are currently organising IMEP-34, an ILC for the determination of the eight trace metals whose safety limits are set out by the EU Toy Safety Directive (88/378/EEC) and specified in the harmonised European Standard EN71-3:1994. The exercise may be of particular interest to you as your institute is listed under the Toy Safety Directive as being responsible for this type of examination.

For more information on the exercise and for registration please go to
http://irmm.jrc.ec.europa.eu/interlaboratory_comparisons/imep/Imep-34/Pages/IMEP-34.aspx

Registration deadline is 16 September 2011.

FYI, IMEP has carried out a similar exercise two years ago called IMEP-24 and the outcome was met with great interest by laboratories and authorities. You can find the Final Report on our website http://irmm.jrc.ec.europa.eu/interlaboratory_comparisons/imep/imep-24/Pages/index.aspx .

Feel free to contact me in case of any further questions.

Looking forward to welcoming you in our exercise.

Kind regards

Ines Baer
International Measurement Evaluation Programme - IMEP
EC-JRC-IRMM
Tel: +32 (0)14 57 16 82
Fax: +32 (0)14 57 18 65
jrc-irmm-imep@ec.europa.eu
http://irmm.jrc.ec.europa.eu

1

Annex 4: Publication on IRMM website

IMEP-34 Trace metals in toys II according to EN71-3:1994

Privacy statement | Legal notice

European Commission
Joint Research Centre
Institute for Reference Materials and Measurements

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Font Size: A A A A

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- Activities
- Reference materials
- EU Reference Laboratories
- Interlaboratory comparisons
- Job opportunities
- Events
- Training
- Calls
- Publications

IMEP-34 Trace metals in toys II according to EN71-3:1994

IMEP-34 was an interlaboratory comparison for the determination of the eight trace metals whose safety limits are set out by the EU Toy Safety Directive (88/378/EEC) and specified in the harmonised European Standard EN71-3:1994.

The exercise was open to all laboratories.

The cost of this interlaboratory comparison was **EUR 400** per registration.

Test material and analytes

The measurands are antimony, arsenic, barium, cadmium, chromium, lead, mercury and selenium. The test material is comminuted paint from alkyd resin paint and comes in bottles of 2g.

General outline of the exercise

Each participant will receive three bottles of the test material. Participants are requested to perform 3 independent analyses (one replicate per bottle) and to report the mean, its expanded uncertainty and coverage factor k. The laboratories are asked to apply the sample preparation method described in EN71-3:1994, but otherwise to follow their routine procedure. Detailed instructions will be sent together with the sample.

Schedule

Registration	Sample dispatch	Reporting of results	Report to participants
Deadline 16/09/2011	Second half September 2011	Deadline 18/11/2011	April 2012

Latest update 26 October, 2011

News archive

- Environmental analysis
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http://irmm.jrc.ec.europa.eu/interlaboratory_comparisons/imep/Imep-34/Pages/IMEP-34.aspx[16/02/2012 11:32:26]

Annex 5: Sample accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for reference materials and measurements
Food Safety & Quality

Geel, 6 October 2011
JRC.DG.D6/IBa/bk/ARES(2011)/

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

Participation in IMEP-34, a proficiency test exercise for the determination of eight trace elements in toys according to EN71-3:1994

Dear «TITLE» «SURNAME»,

Thank you for participating in the IMEP-34 proficiency test for the determination of eight trace elements specified in the harmonised European Standard EN71-3:1994, and whose safety limits were set out by the EU toy safety directive 88/378/EEC and which are still included in the current toy safety directive 2009/48/EC. **Please keep this letter**, you need it for reporting your results.

This parcel contains:
a) Three bottles containing approximately 2 g of the test material each
b) A "Confirmation of Receipt" form
c) A summary of the questionnaire to be answered on-line after reporting your results.
d) This accompanying letter

Please check whether the bottles 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 place at ≤18 °C until analysis.

Measurands and procedure to apply
Measurands are the migrated concentrations of arsenic, antimony, barium, cadmium, chromium, lead, mercury and selenium to be determined as described in EN71-3:1994. The sample matrix is an alkyd resin paint in powder form.

«Part_key»

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

1/4

One measurement per bottle is to be performed, meaning in total 3 replicates. Perform the measurements as you use to in routine sample analysis. A minimum sample intake of 0.5 g is recommended.

Reporting of results
The reporting website is https://irmm.jrc.ec.europa.eu/ilc/ilcReporting.do
Please report:
- the result for **each replicate** and the **corrected mean** (mg kg^{-1})
- the associated expanded **uncertainty** (mg kg^{-1}),
- the **coverage factor** and
- the **technique** you used.

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

To access the webpage you need a personal password key, which is: **«Part_key»**. The system will guide you through the reporting procedure. **Check your results carefully** for any errors before submission, since your results cannot be changed after we have received them.

Please also complete the relating online-questionnaire. A summary of the questions was sent with this letter. Do not forget to save and submit when required.

For final submission please:
- press "Confirm results and questionnaire"
- **print** the completed report form
- **sign** the paper version and
- **send** it to IRMM by fax or by e-mail.

The **deadline** for submission of results is **18/11/2011**.

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

Fernando Cordeiro Raposo

Dr. Fernando Cordeiro Raposo
IMEP-34 Co-ordinator

Enclosures: 1) three bottles containing the test material; 2) confirmation of receipt form; 3) Summary IMEP-34 questionnaire; 4) Accompanying letter.

2/4 «Part_key»

Annex 6: Questionnaire

Milc questionnaire

Comparison for IMEP-34

Please complete the questionnaire.

Submission Form

1. **Please answer following questions regarding EN71-3:1994.**

1.1. Please specify which procedure you have followed (which chapter) in EN71-3 :

1.2. Have you sieved the sample ?

- No
- Yes

1.2.1. If yes, what sieve/mesh size have you used ?

1.3. State the sample amount used per replicate :

1.4. What shaking device have you used ?

1.5. Have you applied the temperature recommendation of 37 C ?

- No
- Yes

1.5.1. If not, which temperature was applied ?

1.6. What was the final pH ?

1.7. Specify the type and porosity of the membrane filter used:

1.8. Was a centrifugation step necessary ?

- No
- Yes

1.9. Did you analyse the samples on the day of processing ?

- No
- Yes

1.9.1. If not :

1.9.1.1. How did you store the samples until analysis ?

1.9.1.2. How long have you stored the samples ?

2. **If you have deviated from the EN71-3 protocol, please describe briefly how :**

3. **What are your detection limits (LoD, mg/kg) for :**

3.1. Antimony :

3.2. Arsenic :

3.3. Barium :

3.4. Cadmium :

3.5. Chromium :

3.6. Lead :

3.7. Mercury :

3.8. Selenium :

4. **What is the level of confidence reflected by coverage factor k reported with your results ? (in %)**

5. **What is the basis of your uncertainty estimate ? (multiple answers possible)**

- [ ] a) uncertainty budget according to ISO-GUM
- [ ] b) known uncertainty of the standard method
- [ ] c) uncertainty of the method as determined during in-house validation
- [ ] d) measurement of replicates (i.e. precision)
- [ ] e) estimation based on judgement
- [ ] f) use of intercomparison data
- [ ] g) other

5.1. If other, please specify :

____

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

- ( ) No
- ( ) Yes

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

- ( ) No
- ( ) Yes

7.1. If yes, which one ?

- [ ] ISO 17025
- [ ] ISO 9000 series
- [ ] Other

7.1.1. If other, please specify :

____

7.2. Are you accredited ?

- ( ) No
- ( ) Yes

7.2.1. If yes, by which accreditation body ?

____

8. **Does your laboratory carry out this type of analysis on a regular basis ?**

- ( ) No
- ( ) Yes

8.1. If yes, please estimate the number of samples :

- ( ) a) 0-50 samples per year
- ( ) b) 50-250 samples per year
- ( ) c) 250-1000 samples per year
- ( ) d) more than 1000 samples per year

9. **Does your laboratory take part in similar interlaboratory comparisons on a regular basis ?**

- ( ) No
- ( ) Yes

9.1. Which ILC scheme(s) ?

____

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

- ( ) No
- ( ) Yes

10.1. If yes, which one ?

____

10.2. Is the material used for the validation of procedures ?

- ( ) No
- ( ) Yes

10.3. Is the material used for the calibration of instruments ?

- ( ) No
- ( ) Yes

11. **Concerning your reported results, have you applied the analytical correction (EN71-3, Ch. 4.2) ?**

- ( ) No
- ( ) Yes

11.1. If yes, for which elements ?

____

12. **Would you accept the material on the European market according to**

12.1. Toy Safety Directive 88/378/EEC ?

- No
- Yes

12.1.1. Explain, why :

12.2. Toy Safety Directive 2009/48/EC ?

- No
- Yes

12.2.1. Explain, why :

12.3. Did you base your decision on

- raw results
- results corrected by analytical correction

13. **How have you heard about this exercise ?**

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

Annex 7: 'Confirmation of receipt' form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
Food Safety & Quality

Annex to JRC.DG.D6/IBa/bk/ARES(2011)/

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

IMEP-34

Trace metals in toys II

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 Fernando Cordeiro Raposo

IMEP-34 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

Annex 8: Homogeneity study

Homogeneity	Sb		As		Ba		Cd		Cr		Pb		Hg		Se	
Sample	R1	R2	R1	R2	R1	R2	R1	R2	R1	R2	R1	R2	R1	R2	R1	R2
	626.9	626.9	150.8	146.0	511.4	528.3	11988.9	11612.7	90.0	87.6	12.7	12.3	3559.9	3265.0	1057.4	1028.0
	627.3	646.5	147.9	142.6	535.8	571.4	12008.7	12276.0	88.8	91.7	10.7	11.0	3235.0	3302.9	1035.9	1138.8
	661.8	664.3	151.3	146.8	557.3	561.4	12978.9	12513.6	93.4	93.2	12.0	11.6	2477.4	2513.3	1087.8	1165.2
	654.1	664.8	152.1	145.6	570.9	534.9	12949.2	12335.4	101.7	92.7	12.1	12.1	2447.3	2459.0	1070.2	1162.3
	639.6	656.8	143.6	148.7	569.4	548.1	12276.0	12870.0	89.9	90.8	12.6	11.9	3462.9	3466.8	1041.7	1142.7
	675.2	647.9	151.3	151.4	593.2	577.6	12860.1	12553.2	95.5	92.7	12.1	11.5	2451.2	2356.1	1186.8	1064.3
	675.7	654.1	148.8	155.1	567.4	576.2	12800.7	12939.3	95.2	91.8	13.4	13.1	2577.3	2486.1	1190.7	1076.0
	655.1	679.8	154.2	150.2	586.8	583.1	12978.9	12830.4	91.5	96.1	12.0	11.3	2826.6	2918.7	1067.2	1184.8
	653.3	645.7	154.4	142.1	551.9	534.8	13008.6	12097.8	90.2	91.2	11.6	11.2	3480.4	3450.3	1162.3	1120.1
	657.4	685.7	148.8	151.1	536.7	567.7	12780.9	13295.7	90.4	97.6			3009.9	3098.2	1065.3	1178.0
Mean	654.9		149.1		558.2		12597.8		92.6		12.0		2942.2		1111.3	
Half Anal Corr [%]	30		30		15		15		15				15		15	
$\hat{\sigma}$ [mg kg^{-1}]	196.5		44.7		83.7		1889.7		13.9		2.0		441.3		166.7	
Homogeneity test according to ISO 13528 (mg kg^{-1})																
$0.3\hat{\sigma}$	58.95		13.42		25.12		566.90		4.17		0.61		132.40		50.01	
s_x	14.33		2.32		19.49		371.69		2.51		0.64		445.90		29.45	
s_w	13.16		4.19		15.66		345.62		3.08		0.30		79.86		68.04	
s_s	10.89		0.00		16.04		280.05		1.24		0.60		442.31		0.00	
$s_s \le 0.3\hat{\sigma}$?	Yes		Yes		Yes		Yes		Yes		Yes		No		Yes	
Test	**Passed**		**Passed**		**Passed**		**Passed**		**Passed**		**Passed**		**Failed**		**Passed**	

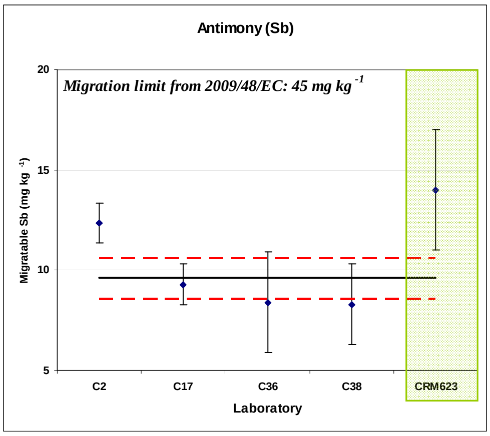
Where: $\hat{\sigma}$ is the standard deviation for the PT assessment,

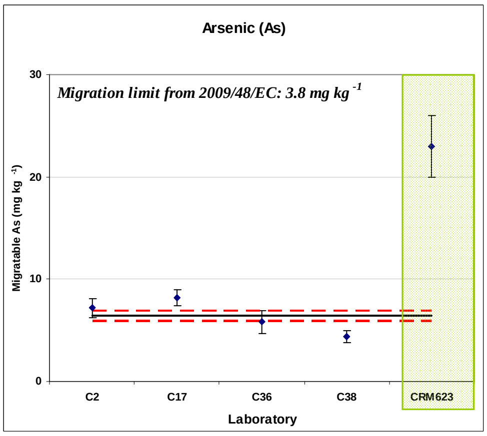
S_x is the standard deviation of the samples averages,

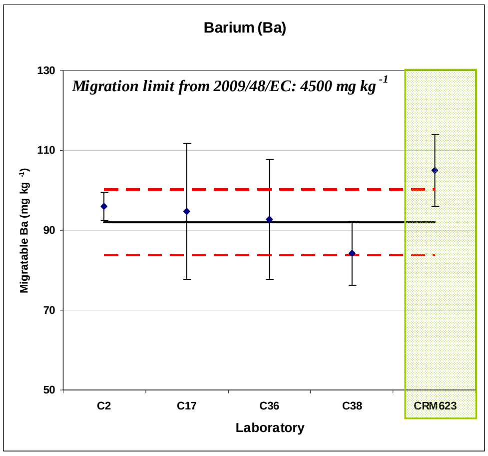
S_w is the within-samples standard deviation,

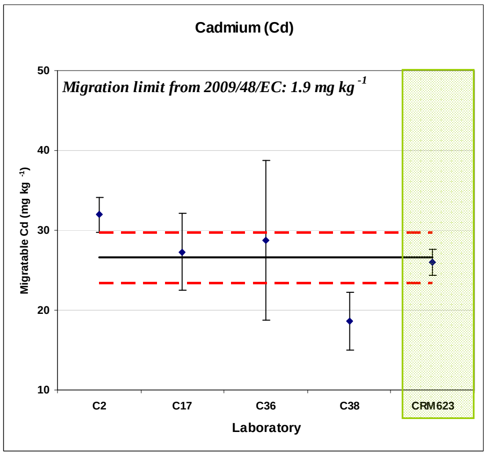
S_S is the between-samples standard deviation

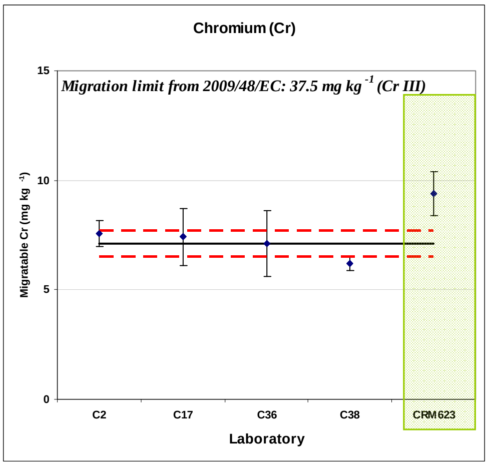
Annex 9: Reference values and their associated uncertainties

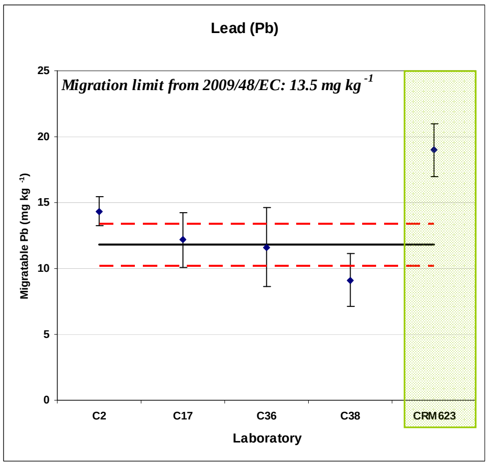


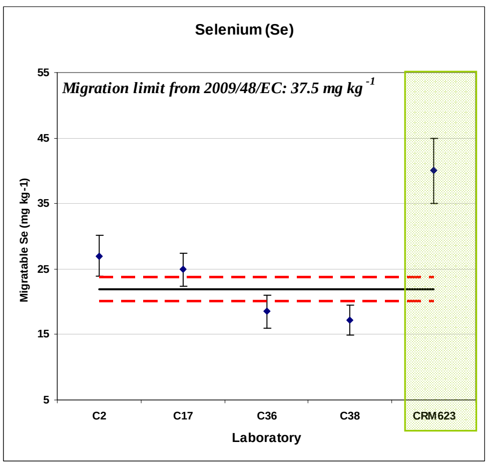


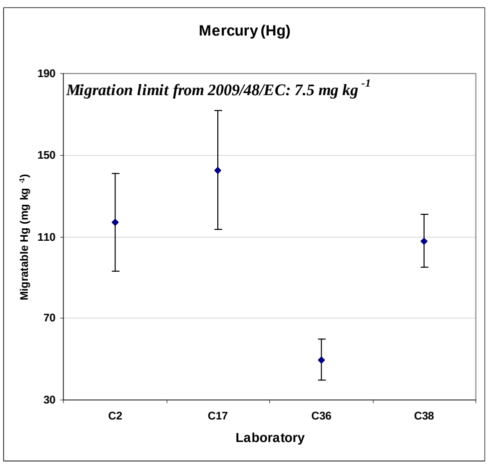












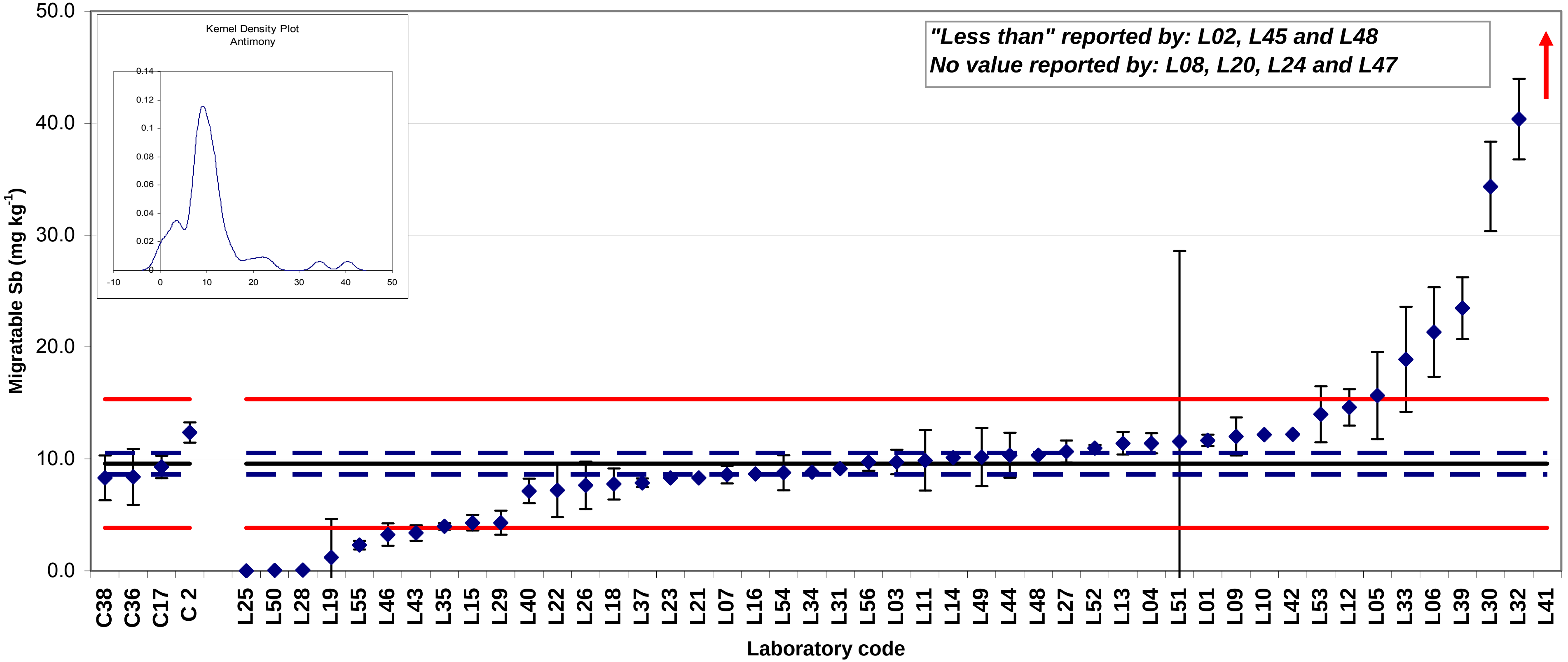
Annex 10: Results for Antimony

X_{ref} = 9.6 and U_{ref} = 1.0; all values are given in (mg kg^{-1})

Lab ID	X_{mean}	U_{lab}	k[a]	u_{lab}	Technique	z-score[b]	ζ-score[b]	U[c]
C 1	< 10	0	√3	0.00	ICP-OES			b
C 2	12.36	0.89	√3	0.51	ICP-MS			a
C17	9.29	1	2	0.50	ICP-OES			a
C36	8.40	2.5	2	1.25	ICP-OES			a
C38	8.30	2	2	1.00	ICP-OES			a
L01	11.67	0.5	2	0.25	ICP-OES	0.7	3.8	b
L02	< 15	0	√3	0.00	FAAS			b
L03	9.73	1.1	2	0.55	ICP-MS	0.1	0.2	a
L04	11.40	0.9	2	0.45	ICP-MS	0.6	2.8	b
L05	15.67	3.9	2	1.95	ICP-OES	2.1	3.0	a
L06	21.33	4	2	2.00	ICP-MS	4.1	5.7	a
L07	8.60	0.79	2	0.40	ICP-OES	-0.3	-1.6	b
L09	12.00	1.7	2	0.85	ICP-MS	0.8	2.5	a
L10	12.17	0	√3	0.00	ICP-OES	0.9	5.4	b
L11	9.87	2.7	2	1.35	ICP-OES	0.1	0.2	a
L12	14.61	1.63	2	0.82	ICP-OES	1.7	5.3	a
L13	11.40	1	2	0.50	ICP-OES	0.6	2.6	a
L14	10.12	0	1.96	0.00	ICP-OES	0.2	1.1	b
L15	4.30	0.7	2	0.35	ICP-OES	-1.8	-8.9	b
L16	8.67	0	√3	0.00	ICP-OES	-0.3	-1.9	b
L18	7.77	1.4	2	0.70	ICP-OES	-0.6	-2.1	a
L19	1.20	3.46	2	1.73	ETAAS	-2.9	-4.7	a
L21	8.31	0	√3	0.00	ICP-OES	-0.4	-2.7	b
L22	7.20	2.41	2	1.21	ICP-OES	-0.8	-1.8	a
L23	8.30	0	√3	0.00	ETAAS	-0.4	-2.7	b
L25	0.00	0	√3	0.00		-3.3	-20.0	b
L26	7.64	2.12	2	1.06	ICP-OES	-0.7	-1.7	a
L27	10.67	1	2	0.50	ICP-OES	0.4	1.6	a
L28	0.06	0.0122	2	0.01	ICP-MS	-3.3	-19.9	b
L29	4.30	1.07	2	0.54	ICP-MS	-1.8	-7.4	a
L30	34.33	4	2	2.00	ICP-OES	8.6	12.0	a
L31	9.12	0	√3	0.00	ICP-OES	-0.2	-1.0	b
L32	40.37	3.6	2	1.80	ICP-MS	10.7	16.5	a
L33	18.90	4.7	2	2.35	ICP-OES	3.2	3.9	a
L34	8.83	0	√3	0.00	ICP-MS	-0.3	-1.6	b
L35	3.98	0.288	60	0.00	ICP-OES	-1.9	-11.7	b
L37	7.87	0.39	2	0.20	ICP-OES	-0.6	-3.3	b
L39	23.47	2.77	2	1.39	ETAAS	4.8	9.5	a
L40	7.13	1.1	√3	0.64	FAAS	-0.9	-3.1	a
L41	5824.00	36	2	18.00	CV-AAS	2021.3	322.9	c
L42	12.19	0	√3	0.00	ICP-OES	0.9	5.4	b
L43	3.39	0.7	2	0.35	ETAAS	-2.2	-10.4	b
L44	10.33	2	2	1.00	ICP-OES	0.3	0.7	a
L45	< 38.1	0	√3	0.00	FAAS			b
L46	3.24	1	2	0.50	ICP-MS	-2.2	-9.2	a
L48	10.33	0	√3	0.00	ICP-OES	0.3	1.6	b
L49	10.17	2.6	2	1.30	ICP-OES	0.2	0.4	a
L50	0.06	0.011	2	0.01	ICP-OES	-3.3	-19.9	b
L51	11.57	17	2	8.50	ICP-OES	0.7	0.2	c
L52	10.97	0.3	3	0.10	ICP-MS	0.5	2.8	b
L53	14.00	2.5	√3	1.44	ICP-OES	1.5	2.9	a
L54	8.77	1.58	2	0.79	FAAS	-0.3	-0.9	a
L55	2.30	0.4	2	0.20	ICP-MS	-2.5	-14.0	b
L56	9.73	0.8	2	0.40	ICP-OES	0.1	0.2	b

[a] √3 is set by the ILC coordinator when no expansion factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k*=√3.
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u_{ref} \leq u_{lab} \leq \hat{\sigma}$; "b": $u_{lab} < u_{ref}$; "c": $u_{lab} > \hat{\sigma}$

IMEP-34 (Trace elements in toys): Antimony
Assigned value: X_{ref} = 9.6 mg kg^{-1}; U_{ref} = 1.0 mg kg^{-1} (k = 2)



This graph displays the averaged value of the three replicates with their associated uncertainties. The uncertainties are shown as reported. The thick blak line corresponds to $\mathbf{X}_{ref}$, the blue lines to the boundaries of X_{ref} ($\mathbf{X}_{ref} \pm 2\mathbf{u}_{ref}$) the red lines to the acceptance interval ($\mathbf{X}_{ref} \pm 2\sigma$).

Annex 11: Results for Arsenic

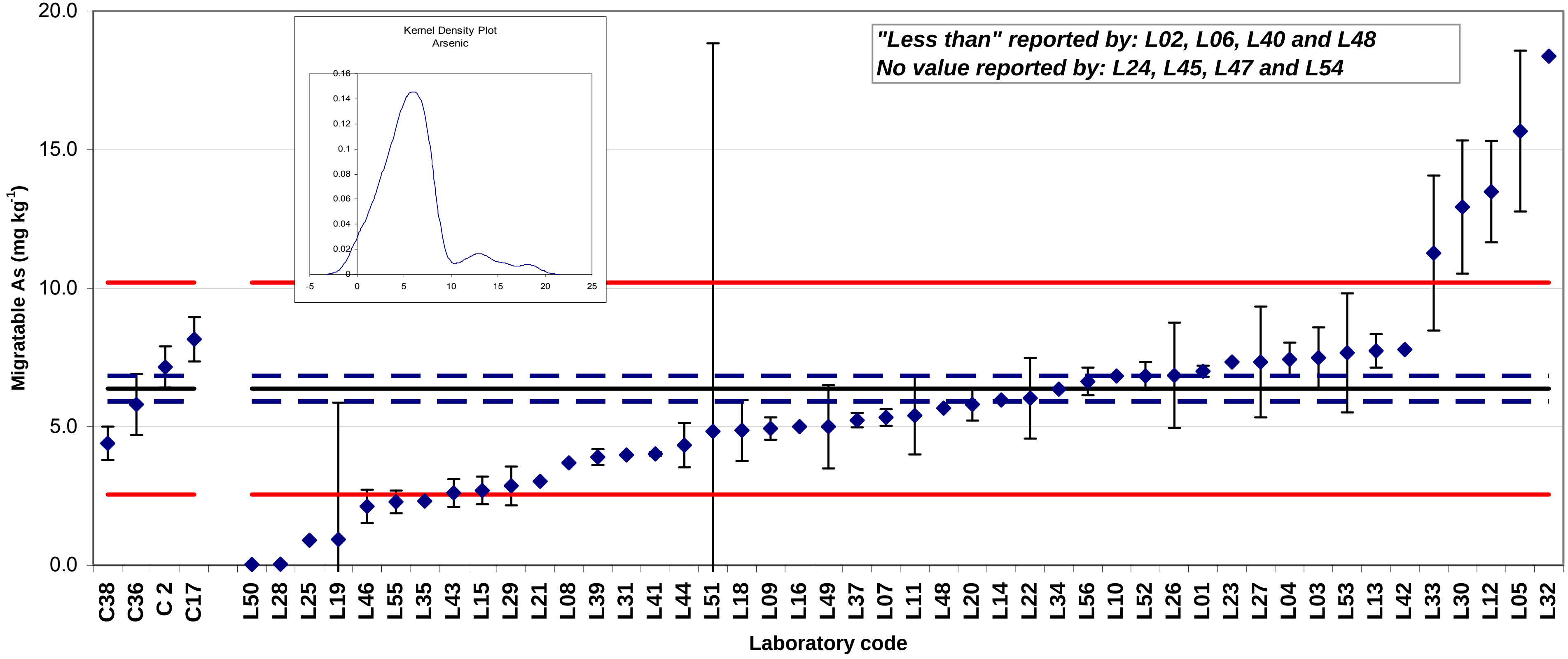
X_{ref} = 6.4 and U_{ref} = 0.5; all values are given in (mg kg^{-1})

Lab ID	X_{mean}	U_{lab}	k[a]	u_{lab}	Technique	z-score[b]	ζ-score[b]	U[c]
C 1	<10	0	√3	0.00	ICP-OES			b
C 2	7.16	0.74	√3	0.43	ICP-MS			a
C17	8.16	0.8	2	0.40	ICP-OES			a
C36	5.80	1.1	2	0.55	ICP-OES			a
C38	4.40	0.6	2	0.30	ICP-OES			a
L01	7.00	0.2	2	0.10	ICP-OES	0.3	2.5	b
L02	<5	0	√3	0.00	FAAS			b
L03	7.49	1.1	2	0.55	ICP-MS	0.6	1.9	a
L04	7.43	0.6	2	0.30	ICP-MS	0.6	2.8	a
L05	15.67	2.9	2	1.45	ICP-OES	4.9	6.3	a
L06	<0.5	0	√3	0.00	ICP-MS			b
L07	5.33	0.3	2	0.15	ICP-OES	-0.5	-3.8	b
L08	3.70	0	√3	0.00	HG-AAS	-1.4	-11.7	b
L09	4.93	0.4	2	0.20	ICP-MS	-0.8	-4.7	b
L10	6.83	0	√3	0.00	ICP-OES	0.2	2.0	b
L11	5.40	1.4	2	0.70	ICP-OES	-0.5	-1.3	a
L12	13.48	1.83	2	0.92	ICP-OES	3.7	7.5	a
L13	7.73	0.6	2	0.30	ICP-OES	0.7	3.6	a
L14	5.96	0	1.96	0.00	ICP-OES	-0.2	-1.8	b
L15	2.70	0.5	2	0.25	ICP-OES	-1.9	-10.8	a
L16	5.00	0	√3	0.00	ICP-OES	-0.7	-6.0	b
L18	4.87	1.1	2	0.55	ICP-OES	-0.8	-2.5	a
L19	0.93	4.94	2	2.47	ETAAS	-2.8	-2.2	c
L20	5.80	0.58	500	0.00	HG-AAS	-0.3	-2.5	b
L21	3.03	0	√3	0.00	ICP-OES	-1.8	-14.6	b
L22	6.03	1.46	2	0.73	ICP-MS	-0.2	-0.5	a
L23	7.33	0	√3	0.00	ETAAS	0.5	4.2	b
L24								
L25	0.90	0.006	√3	0.00	HG-AAS	-2.9	-23.8	b
L26	6.85	1.9	2	0.95	ICP-OES	0.2	0.5	a
L27	7.33	2	2	1.00	ICP-OES	0.5	0.9	a
L28	0.04	0.008	2	0.00	ICP-MS	-3.3	-27.6	b
L29	2.87	0.7	2	0.35	ICP-MS	-1.8	-8.4	a
L30	12.93	2.4	2	1.20	ICP-OES	3.4	5.4	a
L31	3.98	0	√3	0.00	ICP-OES	-1.3	-10.4	b
L32	18.37	0	√3	0.00	ICP-MS	6.3	52.2	b
L33	11.27	2.8	2	1.40	ICP-OES	2.6	3.4	a
L34	6.36	0	√3	0.00	ICP-MS	0.0	-0.1	b
L35	2.31	0.02	60	0.00	ICP-OES	-2.1	-17.7	b
L37	5.23	0.26	2	0.13	ICP-OES	-0.6	-4.3	b
L39	3.91	0.29	2	0.15	ETAAS	-1.3	-9.1	b
L40	<10	0	√3	0.00	FAAS			b
L41	4.02	0.07	2	0.04	CV-AAS	-1.2	-10.1	b
L42	7.79	0	√3	0.00	ICP-OES	0.7	6.1	b
L43	2.61	0.5	2	0.25	ETAAS	-2.0	-11.1	a
L44	4.33	0.8	2	0.40	ICP-OES	-1.1	-4.4	a
L45								
L46	2.13	0.6	2	0.30	ICP-MS	-2.2	-11.3	a
L47								
L48	5.67	0	√3	0.00	ICP-OES	-0.4	-3.1	b
L49	5.00	1.5	2	0.75	ICP-OES	-0.7	-1.8	a
L50	0.03	0.01	2	0.01	ICP-OES	-3.3	-27.6	b
L51	4.83	14	2	7.00	ICP-OES	-0.8	-0.2	c
L52	6.83	0.5	3	0.17	ICP-MS	0.2	1.6	b
L53	7.67	2.15	√3	1.24	ICP-OES	0.7	1.0	a
L54								
L55	2.28	0.41	2	0.21	ICP-MS	-2.1	-13.3	b
L56	6.63	0.5	2	0.25	ICP-OES	0.1	0.8	a

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

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u_{ref} \leq u_{lab} \leq \hat{\sigma}$; "b": $u_{lab} < u_{ref}$; "c": $u_{lab} > \hat{\sigma}$



This graph displays the averaged value of the three replicates with their associated uncertainties. The uncertainties are shown as reported. The thick blak line corresponds to $\mathbf{X_{ref}}$, the blue lines to the boundaries of X_{ref} ($\mathbf{X_{ref} \pm 2u_{ref}}$) the red lines to the acceptance interval ($\mathbf{X_{ref} \pm 2\sigma}$).

Annex 12: Results for Barium

X_{ref} = 92.0 and U_{ref} = 8.2; all values are given in (mg kg^{-1})

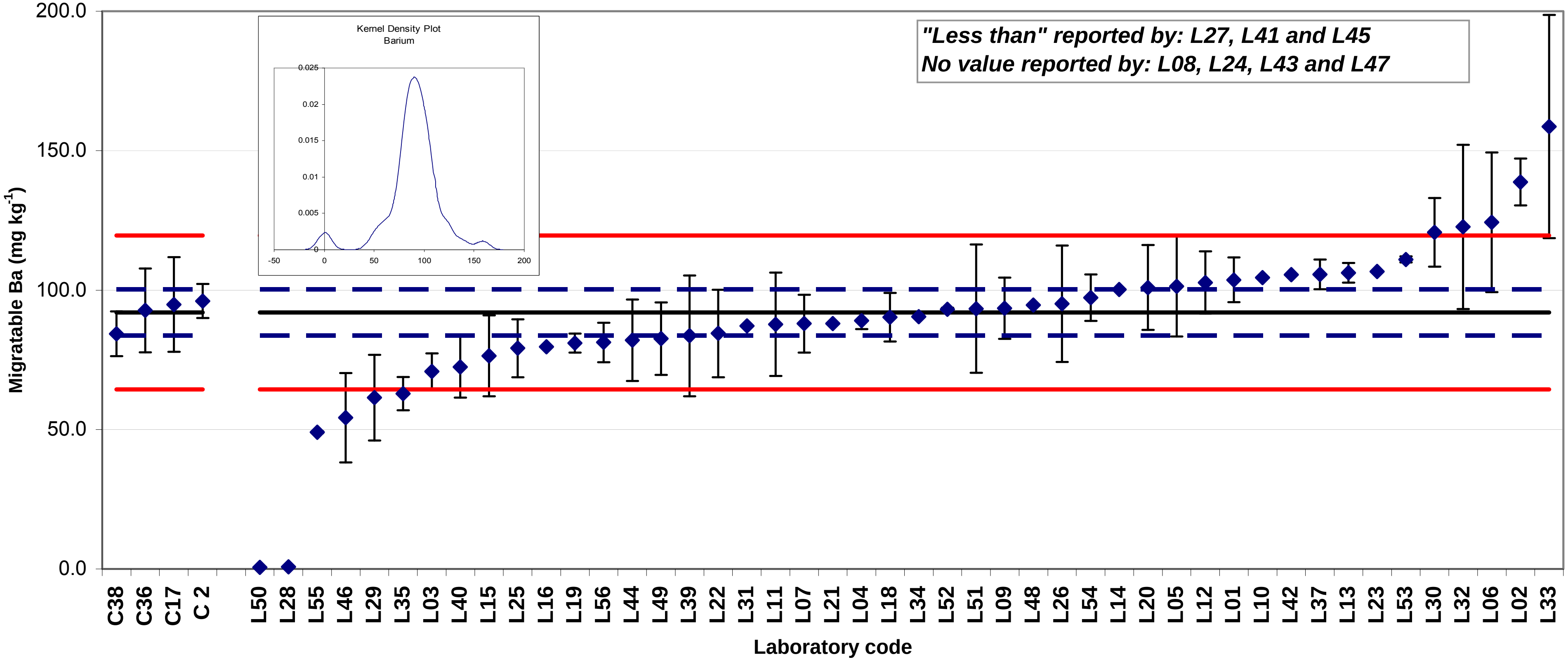
Lab ID	X_{mean}	U_{lab}	k[a]	u_{lab}	Technique	z-score[b]	ζ-score[b]	U[c]
C 1	80.37	0	√3	0.00	ICP-OES			b
C 2	96.11	6.11	√3	3.53	ICP-MS			b
C17	94.83	17	2	8.50	ICP-OES			a
C36	92.70	15	2	7.50	ICP-OES			a
C38	84.33	8	2	4.00	ICP-OES			b
L01	103.67	8	2	4.00	ICP-OES	0.8	2.0	b
L02	138.77	8.4	√3	4.85	FAAS	3.4	7.4	a
L03	70.82	6.5	2	3.25	ICP-MS	-1.5	-4.0	b
L04	89.00	3	2	1.50	ICP-MS	-0.2	-0.7	b
L05	101.33	18	2	9.00	ICP-OES	0.7	0.9	a
L06	124.33	25	2	12.50	ICP-MS	2.3	2.5	a
L07	87.97	10.4	2	5.20	ICP-OES	-0.3	-0.6	a
L08								
L09	93.53	11	2	5.50	ICP-MS	0.1	0.2	a
L10	104.50	0	√3	0.00	ICP-OES	0.9	3.0	b
L11	87.77	18.5	2	9.25	ICP-OES	-0.3	-0.4	a
L12	102.72	11.16	2	5.58	ICP-OES	0.8	1.5	a
L13	106.23	3.5	2	1.75	ICP-OES	1.0	3.2	b
L14	100.21	0	1.96	0.00	ICP-OES	0.6	2.0	b
L15	76.47	14.5	2	7.25	ICP-OES	-1.1	-1.9	a
L16	79.67	0	√3	0.00	ICP-OES	-0.9	-3.0	b
L18	90.27	8.7	2	4.35	ICP-OES	-0.1	-0.3	a
L19	81.00	3.4	2	1.70	ETAAS	-0.8	-2.5	b
L20	100.93	15.2	500	0.03	ICP-MS	0.6	2.2	b
L21	88.01	0	√3	0.00	ICP-OES	-0.3	-1.0	b
L22	84.47	15.7	2	7.85	ICP-OES	-0.5	-0.8	a
L23	106.67	0	√3	0.00	ETAAS	1.1	3.6	b
L24								
L25	79.16	10.38	√3	5.99	FAAS	-0.9	-1.8	a
L26	95.14	20.87	2	10.44	ICP-OES	0.2	0.3	a
L27	<100	0	√3	0.00	ICP-OES			b
L28	0.77	0.1532	2	0.08	ICP-MS	-6.6	-22.2	b
L29	61.43	15.37	2	7.69	ICP-MS	-2.2	-3.5	a
L30	120.73	12.3	2	6.15	ICP-OES	2.1	3.9	a
L31	87.13	0	√3	0.00	ICP-OES	-0.4	-1.2	b
L32	122.67	29.4	2	14.70	ICP-MS	2.2	2.0	c
L33	158.67	40	2	20.00	ICP-OES	4.8	3.3	c
L34	90.44	0	√3	0.00	ICP-MS	-0.1	-0.4	b
L35	62.87	6	30	0.20	ICP-OES	-2.1	-7.1	b
L37	105.67	5.3	2	2.65	ICP-OES	1.0	2.8	b
L39	83.60	21.69	2	10.85	ETAAS	-0.6	-0.7	a
L40	72.47	11	√3	6.35	FAAS	-1.4	-2.6	a
L41	<8	0	√3	0.00	FAAS			b
L42	105.56	0	√3	0.00	ICP-OES	1.0	3.3	b
L43								
L44	82.00	14.6	2	7.30	ICP-OES	-0.7	-1.2	a
L45	<157	0	√3	0.00	FAAS			b
L46	54.23	16	2	8.00	ICP-MS	-2.7	-4.2	a
L47								
L48	94.67	0	√3	0.00	ICP-OES	0.2	0.6	b
L49	82.57	13	2	6.50	ICP-OES	-0.7	-1.2	a
L50	0.60	0.113	2	0.06	ICP-OES	-6.6	-22.2	b
L51	93.33	23	2	11.50	ICP-OES	0.1	0.1	a
L52	93.17	0.5	3	0.17	ICP-MS	0.1	0.3	b
L53	111.00	1.05	√3	0.61	ICP-OES	1.4	4.6	b
L54	97.33	8.34	2	4.17	FAAS	0.4	0.9	a
L55	49.05	0.38	2	0.19	ICP-MS	-3.1	-10.4	b
L56	81.23	7.1	2	3.55	ICP-OES	-0.8	-2.0	b

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

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u_{ref} \leq u_{lab} \leq \hat{\sigma}$; "b": $u_{lab} < u_{ref}$; "c": $u_{lab} > \hat{\sigma}$

IMEP-34 (Trace elements in toys): Barium
Assigned value: X_{ref} = 92.0 mg kg^{-1}; U_{ref} = 8.2 mg kg^{-1} (k = 2)



This graph displays the averaged value of the three replicates with their associated uncertainties. The uncertainties are shown as reported. The thick blak line corresponds to $\mathbf{X_{ref}}$, the blue lines to the boundaries of X_{ref} ($\mathbf{X_{ref} \pm 2u_{ref}}$) the red lines to the acceptance interval ($\mathbf{X_{ref} \pm 2\sigma}$).

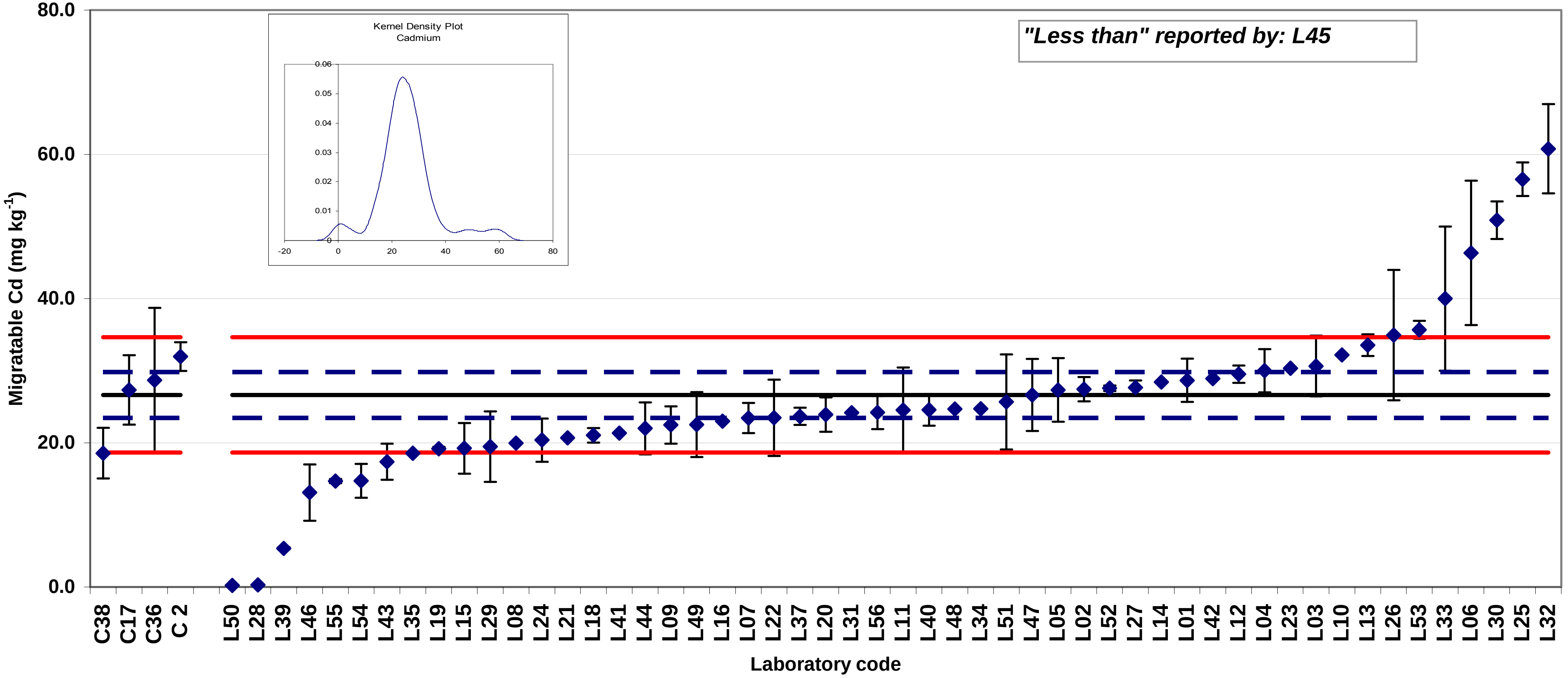
Annex 13: Results for Cadmium

X_{ref} = 26.6 and U_{ref} = 3.2; all values are given in (mg kg^{-1})

Lab ID	X_{mean}	U_{lab}	k[a]	u_{lab}	Technique	z-score[b]	ζ-score[b]	U[c]
C 1	25.20	0	√3	0.00	ICP-OES			b
C 2	31.96	1.98	√3	1.14	ICP-MS			b
C17	27.33	4.8	2	2.40	ICP-OES			a
C36	28.70	10	2	5.00	ICP-OES			c
C38	18.57	3.5	2	1.75	ICP-OES			a
L01	28.67	3	2	1.50	ICP-OES	0.5	0.9	b
L02	27.43	1.7	√3	0.98	FAAS	0.2	0.4	b
L03	30.64	4.2	2	2.10	ICP-MS	1.0	1.5	a
L04	30.00	3	2	1.50	ICP-MS	0.8	1.5	b
L05	27.33	4.4	2	2.20	ICP-OES	0.2	0.3	a
L06	46.33	10	2	5.00	ICP-MS	4.9	3.8	c
L07	23.43	2.1	2	1.05	ICP-OES	-0.8	-1.7	b
L08	19.93	0	√3	0.00	FAAS	-1.7	-4.2	b
L09	22.47	2.6	2	1.30	ICP-MS	-1.0	-2.0	b
L10	32.17	0	√3	0.00	ICP-OES	1.4	3.5	b
L11	24.53	5.9	2	2.95	ICP-OES	-0.5	-0.6	a
L12	29.53	1.19	2	0.60	ICP-OES	0.7	1.7	b
L13	33.53	1.5	2	0.75	ICP-OES	1.7	3.9	b
L14	28.42	0	1.96	0.00	ICP-OES	0.4	1.1	b
L15	19.23	3.5	2	1.75	ICP-OES	-1.9	-3.1	a
L16	23.00	0	√3	0.00	ICP-OES	-0.9	-2.3	b
L18	21.03	1	2	0.50	ICP-OES	-1.4	-3.4	b
L19	19.17	0.28	2	0.14	ETAAS	-1.9	-4.7	b
L20	23.90	2.39	500	0.00	ICP-MS	-0.7	-1.7	b
L21	20.68	0	√3	0.00	ICP-OES	-1.5	-3.7	b
L22	23.47	5.3	2	2.65	ICP-OES	-0.8	-1.0	a
L23	30.33	0	√3	0.00	ETAAS	0.9	2.3	b
L24	20.37	3	√3	1.73	ETAAS	-1.6	-2.7	a
L25	56.54	2.337	√3	1.35	FAAS	7.5	14.3	b
L26	34.92	9.04	2	4.52	ICP-OES	2.1	1.7	c
L27	27.67	1	2	0.50	ICP-OES	0.3	0.6	b
L28	0.28	0.0568	2	0.03	ICP-MS	-6.6	-16.5	b
L29	19.47	4.9	2	2.45	ICP-MS	-1.8	-2.5	a
L30	50.87	2.6	2	1.30	ICP-OES	6.1	11.8	b
L31	24.17	0	√3	0.00	ICP-OES	-0.6	-1.6	b
L32	60.77	6.2	2	3.10	ICP-MS	8.5	9.8	a
L33	40.00	10	2	5.00	ICP-OES	3.3	2.5	c
L34	24.70	0	√3	0.00	ICP-MS	-0.5	-1.2	b
L35	18.55	0.004	30	0.00	ICP-OES	-2.0	-5.1	b
L37	23.67	1.2	2	0.60	ICP-OES	-0.7	-1.7	b
L39	5.38	0.16	2	0.08	ETAAS	-5.3	-13.3	b
L40	24.57	2.2	√3	1.27	FAAS	-0.5	-1.0	b
L41	21.36	0.01	2	0.01	FAAS	-1.3	-3.3	b
L42	28.89	0	√3	0.00	ICP-OES	0.6	1.4	b
L43	17.38	2.5	2	1.25	ETAAS	-2.3	-4.6	b
L44	22.00	3.6	2	1.80	ICP-OES	-1.2	-1.9	a
L45	<55.5	0	√3	0.00	FAAS			b
L46	13.10	3.9	2	1.95	ICP-MS	-3.4	-5.4	a
L47	26.63	5	2	2.50	ETAAS	0.0	0.0	a
L48	24.67	0	√3	0.00	ICP-OES	-0.5	-1.2	b
L49	22.53	4.5	2	2.25	ICP-OES	-1.0	-1.5	a
L50	0.21	0.045	2	0.02	ICP-OES	-6.6	-16.6	b
L51	25.67	6.6	2	3.30	ICP-OES	-0.2	-0.3	a
L52	27.57	0.4	3	0.13	ICP-MS	0.2	0.6	b
L53	35.67	1.25	√3	0.72	ICP-OES	2.3	5.2	b
L54	14.73	2.36	2	1.18	FAAS	-3.0	-6.0	b
L55	14.69	0.31	2	0.16	ICP-MS	-3.0	-7.5	b
L56	24.20	2.3	2	1.15	ICP-OES	-0.6	-1.2	b

[a] √3 is set by the ILC coordinator when no expansion factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k*=√3.
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u_{ref} \leq u_{lab} \leq \hat{\sigma}$; "b": $u_{lab} < u_{ref}$; "c": $u_{lab} > \hat{\sigma}$

IMEP-34 (Trace elements in toys): Cadmium
Assigned value: X_{ref} = 26.6 mg kg^{-1}; U_{ref} = 3.2 mg kg^{-1} (k = 2)



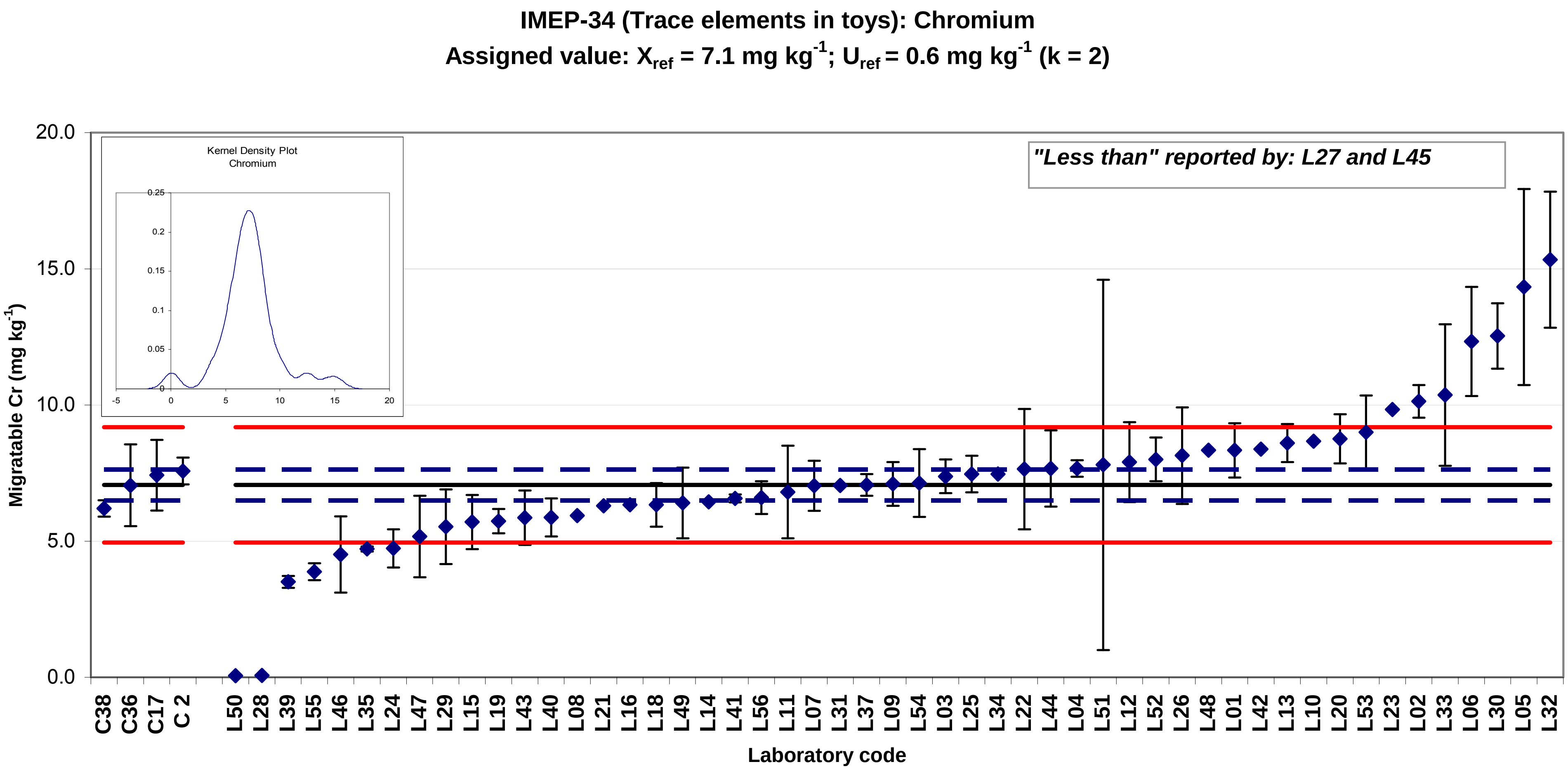
This graph displays the averaged value of the three replicates with their associated uncertainties. The uncertainties are shown as reported. The thick blak line corresponds to $\mathbf{X_{ref}}$, the blue lines to the boundaries of X_{ref} ($\mathbf{X_{ref} \pm 2u_{ref}}$) the red lines to the acceptance interval ($\mathbf{X_{ref} \pm 2\sigma}$).

Annex 14: Results for Chromium

X_{ref} = 7.1 and U_{ref} = 0.6; all values are given in (mg kg^{-1})

Lab ID	X_{mean}	U_{lab}	k[a]	u_{lab}	Technique	z-score[b]	ζ-score[b]	U[c]
C 1	<10	0	√3	0.00	ICP-OES			b
C 2	7.57	0.49	√3	0.28	ICP-MS			b
C17	7.42	1.3	2	0.65	ICP-OES			a
C36	7.05	1.5	2	0.75	ICP-OES			a
C38	6.20	0.3	2	0.15	ICP-OES			b
L01	8.33	1	2	0.50	ICP-OES	1.2	2.2	a
L02	10.13	0.6	√3	0.35	FAAS	2.9	6.9	a
L03	7.38	0.62	2	0.31	ICP-MS	0.3	0.8	a
L04	7.67	0.3	2	0.15	ICP-MS	0.6	1.9	b
L05	14.33	3.6	2	1.80	ICP-OES	6.9	4.0	c
L06	12.33	2	2	1.00	ICP-MS	5.0	5.1	a
L07	7.03	0.92	2	0.46	ICP-OES	0.0	-0.1	a
L08	5.93	0	√3	0.00	FAAS	-1.1	-4.0	b
L09	7.10	0.8	2	0.40	ICP-MS	0.0	0.1	a
L10	8.67	0	√3	0.00	ICP-OES	1.5	5.7	b
L11	6.80	1.7	2	0.85	ICP-OES	-0.2	-0.3	a
L12	7.90	1.47	2	0.74	ICP-OES	0.8	1.1	a
L13	8.60	0.7	2	0.35	ICP-OES	1.5	3.4	a
L14	6.44	0	1.96	0.00	ICP-OES	-0.6	-2.2	b
L15	5.70	1	2	0.50	ICP-OES	-1.3	-2.4	a
L16	6.33	0	√3	0.00	ICP-OES	-0.7	-2.6	b
L18	6.33	0.8	2	0.40	ICP-OES	-0.7	-1.5	a
L19	5.73	0.449	2	0.22	ETAAS	-1.3	-3.7	b
L20	8.76	0.9	500	0.00	ICP-MS	1.6	6.0	b
L21	6.30	0	√3	0.00	ICP-OES	-0.7	-2.7	b
L22	7.65	2.21	2	1.11	ICP-OES	0.6	0.5	c
L23	9.83	0	√3	0.00	ETAAS	2.6	9.8	b
L24	4.73	0.7	√3	0.40	ETAAS	-2.2	-4.7	a
L25	7.46	0.67	√3	0.39	FAAS	0.4	0.8	a
L26	8.14	1.77	2	0.89	ICP-OES	1.0	1.2	a
L27	<10	0	√3	0.00	ICP-OES			b
L28	0.07	0.015	2	0.01	ICP-MS	-6.6	-24.6	b
L29	5.53	1.37	2	0.69	ICP-MS	-1.4	-2.1	a
L30	12.53	1.2	2	0.60	ICP-OES	5.2	8.2	a
L31	7.04	0	√3	0.00	ICP-OES	0.0	-0.1	b
L32	15.33	2.5	2	1.25	ICP-MS	7.8	6.5	c
L33	10.37	2.6	2	1.30	ICP-OES	3.1	2.5	c
L34	7.47	0	√3	0.00	ICP-MS	0.4	1.4	b
L35	4.71	0.094	30	0.00	ICP-OES	-2.2	-8.3	b
L37	7.07	0.4	2	0.20	ICP-OES	0.0	0.0	b
L39	3.51	0.22	2	0.11	ETAAS	-3.4	-11.7	b
L40	5.87	0.7	√3	0.40	FAAS	-1.1	-2.4	a
L41	6.57	0.142	2	0.07	FAAS	-0.5	-1.7	b
L42	8.38	0	√3	0.00	ICP-OES	1.2	4.6	b
L43	5.86	1	2	0.50	ETAAS	-1.1	-2.1	a
L44	7.67	1.4	2	0.70	ICP-OES	0.6	0.8	a
L45	<44.4	0	√3	0.00	FAAS			b
L46	4.51	1.4	2	0.70	ICP-MS	-2.4	-3.4	a
L47	5.17	1.5	2	0.75	ETAAS	-1.8	-2.4	a
L48	8.33	0	√3	0.00	ICP-OES	1.2	4.5	b
L49	6.40	1.3	2	0.65	ICP-OES	-0.6	-0.9	a
L50	0.06	0.009	2	0.00	ICP-OES	-6.6	-24.6	b
L51	7.80	6.8	2	3.40	ICP-OES	0.7	0.2	c
L52	8.00	0.8	3	0.27	ICP-MS	0.9	2.4	b
L53	9.00	1.35	√3	0.78	ICP-OES	1.8	2.3	a
L54	7.13	1.24	2	0.62	FAAS	0.1	0.1	a
L55	3.88	0.31	2	0.16	ICP-MS	-3.0	-9.8	b
L56	6.60	0.6	2	0.30	ICP-OES	-0.4	-1.1	a

[a] √3 is set by the ILC coordinator when no expansion factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k*=√3.
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u_{ref} \leq u_{lab} \leq \hat{\sigma}$; "b": $u_{lab} < u_{ref}$; "c": $u_{lab} > \hat{\sigma}$



This graph displays the averaged value of the three replicates with their associated uncertainties. The uncertainties are shown as reported. The thick blak line corresponds to $\mathbf{X_{ref}}$, the blue lines to the boundaries of X_{ref} ($\mathbf{X_{ref} \pm 2u_{ref}}$) the red lines to the acceptance interval ($\mathbf{X_{ref} \pm 2\sigma}$).

Annex 15: Results for Lead

X_{ref} = 11.8 and U_{ref} = 1.6; all values are given in (mg kg^{-1})

Lab ID	X_{mean}	U_{lab}	k[a]	u_{lab}	Technique	z-score[b]	ζ-score[b]	U[c]
C 1	7.77	0	√3	0.00	ICP-OES			b
C 2	14.32	0.94	√3	0.54	ICP-MS			b
C17	12.17	2.1	2	1.05	ICP-OES			a
C36	11.60	3	2	1.50	ICP-OES			a
C38	9.10	2	2	1.00	ICP-OES			a
L01	14.67	6	2	3.00	ICP-OES	1.4	1.7	c
L02	19.80	1.2	√3	0.69	FAAS	4.0	9.2	b
L03	12.92	1.3	2	0.65	ICP-MS	0.6	1.3	b
L04	13.00	1	2	0.50	ICP-MS	0.6	1.4	b
L05	13.00	2.6	2	1.30	ICP-OES	0.6	1.2	a
L06	23.33	4	2	2.00	ICP-MS	5.8	9.0	a
L07	10.20	1	2	0.50	ICP-OES	-0.8	-1.9	b
L08	6.63	0	√3	0.00	FAAS	-2.6	-6.5	b
L09	10.97	2.6	2	1.30	ICP-MS	-0.4	-0.8	a
L10	14.83	0	√3	0.00	ICP-OES	1.5	3.8	b
L11	11.60	3.1	2	1.55	ICP-OES	-0.1	-0.2	a
L12	19.40	6.25	2	3.13	ICP-OES	3.8	4.3	c
L13	15.20	1.5	2	0.75	ICP-OES	1.7	3.9	b
L14	12.41	0	1.96	0.00	ICP-OES	0.3	0.8	b
L15	9.77	1.9	2	0.95	ICP-OES	-1.0	-2.2	a
L16	10.33	0	√3	0.00	ICP-OES	-0.7	-1.8	b
L18	9.70	1	2	0.50	ICP-OES	-1.0	-2.5	b
L19	13.90	1.79	2	0.90	ETAAS	1.0	2.3	a
L20	12.40	1.24	500	0.00	ICP-MS	0.3	0.8	b
L21	9.65	0	√3	0.00	ICP-OES	-1.1	-2.7	b
L22	9.17	2.55	2	1.28	ICP-OES	-1.3	-2.6	a
L23	17.00	0	√3	0.00	ETAAS	2.6	6.5	b
L24	9.51	0.7	√3	0.40	ETAAS	-1.1	-2.8	b
L25	40.20	7.053	√3	4.07	FAAS	14.2	13.0	c
L26	14.53	4.21	2	2.11	ICP-OES	1.4	2.1	c
L27	12.67	1	2	0.50	ICP-OES	0.4	1.0	b
L28	0.12	0.0244	2	0.01	ICP-MS	-5.8	-14.6	b
L29	9.07	2.23	2	1.12	ICP-MS	-1.4	-2.8	a
L30	30.27	0.6	2	0.30	ICP-OES	9.2	22.7	b
L31	11.10	0	√3	0.00	ICP-OES	-0.3	-0.9	b
L32	44.40	5.7	2	2.85	ICP-MS	16.3	20.0	c
L33	21.43	5.4	2	2.70	ICP-OES	4.8	6.1	c
L34	11.28	0	√3	0.00	ICP-MS	-0.3	-0.6	b
L35	7.63	0.034	30	0.00	ICP-OES	-2.1	-5.2	b
L37	10.10	0.5	2	0.25	ICP-OES	-0.8	-2.1	b
L39	1.03	0.35	2	0.18	ETAAS	-5.4	-13.4	b
L40	11.73	2.1	√3	1.21	FAAS	0.0	-0.1	a
L41	12.65	0.075	2	0.04	FAAS	0.4	1.1	b
L42	13.58	0	√3	0.00	ICP-OES	0.9	2.2	b
L43	8.50	3	2	1.50	ETAAS	-1.6	-3.0	a
L44	17.33	3.1	2	1.55	ICP-OES	2.8	5.0	a
L45	<44.4	0	√3	0.00	FAAS			b
L46	6.90	2.1	2	1.05	ICP-MS	-2.4	-5.1	a
L47	16.13	3.5	2	1.75	ETAAS	2.2	3.7	a
L48	12.00	0	√3	0.00	ICP-OES	0.1	0.3	b
L49	10.63	2.1	2	1.05	ICP-OES	-0.6	-1.2	a
L50	0.13	0.021	2	0.01	ICP-OES	-5.8	-14.6	b
L51	11.67	13	2	6.50	ICP-OES	-0.1	0.0	c
L52	12.43	1.4	3	0.47	ICP-MS	0.3	0.8	b
L53	16.00	1.55	√3	0.89	ICP-OES	2.1	4.6	a
L54	<20	0	√3	0.00	FAAS			b
L55	5.16	0.4	2	0.20	ICP-MS	-3.3	-8.2	b
L56	11.23	1	2	0.50	ICP-OES	-0.3	-0.7	b

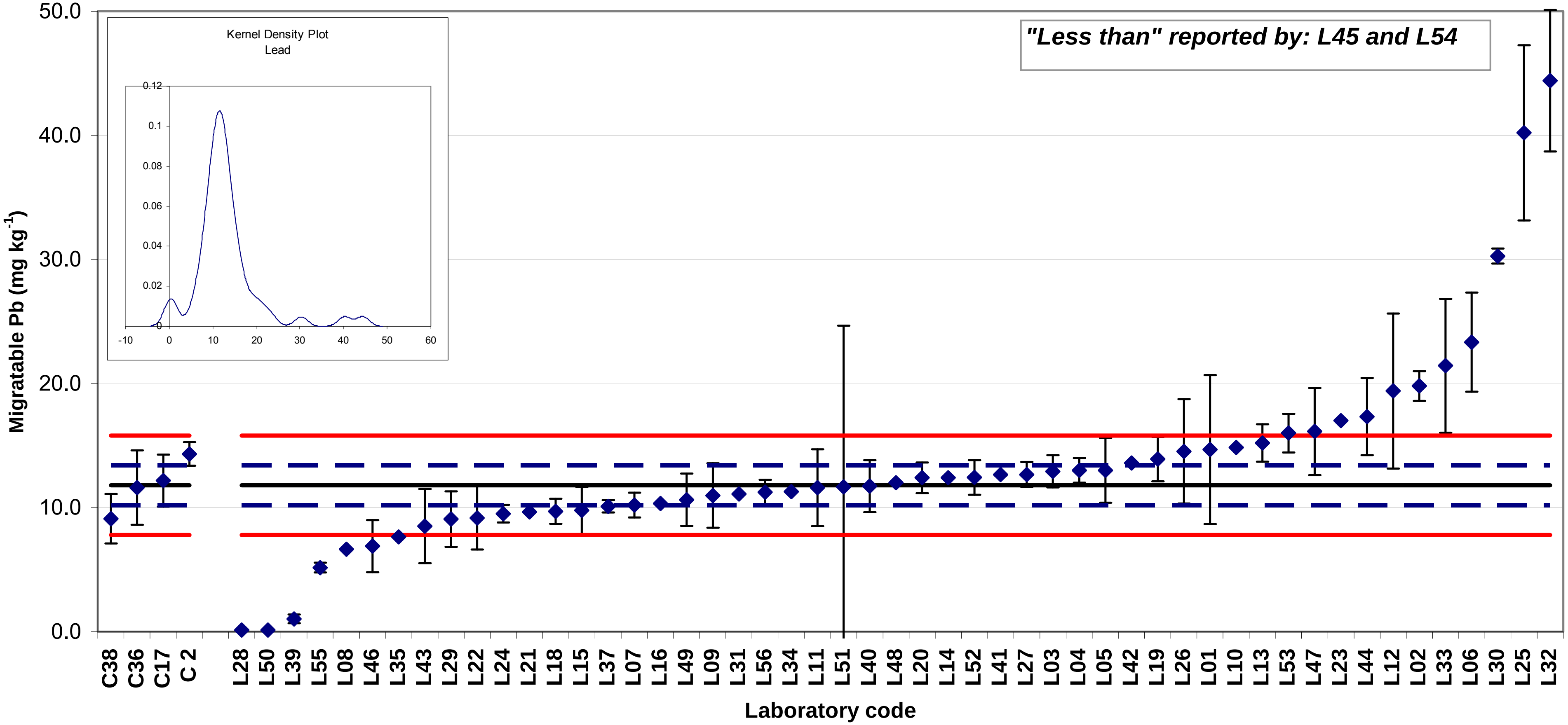
[a] √3 is set by the ILC coordinator when no expansion factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k*=√3.

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u_{ref} \leq u_{lab} \leq \hat{\sigma}$; "b": $u_{lab} < u_{ref}$; "c": $u_{lab} > \hat{\sigma}$

IMEP-34 (Trace elements in toys): Lead

Assigned value: X_{ref} = 11.8 mg kg^{-1}; U_{ref} = 1.6 mg kg^{-1} (k = 2)



This graph displays the averaged value of the three replicates with their associated uncertainties. The uncertainties are shown as reported. The thick blak line corresponds to $\mathbf{X_{ref}}$, the blue lines to the boundaries of X_{ref} ($\mathbf{X_{ref} \pm 2u_{ref}}$) the red lines to the acceptance interval ($\mathbf{X_{ref} \pm 2\sigma}$).

Annex 16: Results for Selenium

X_{ref} = 21.9 and U_{ref} = 1.8; all values are given in (mg kg^{-1})

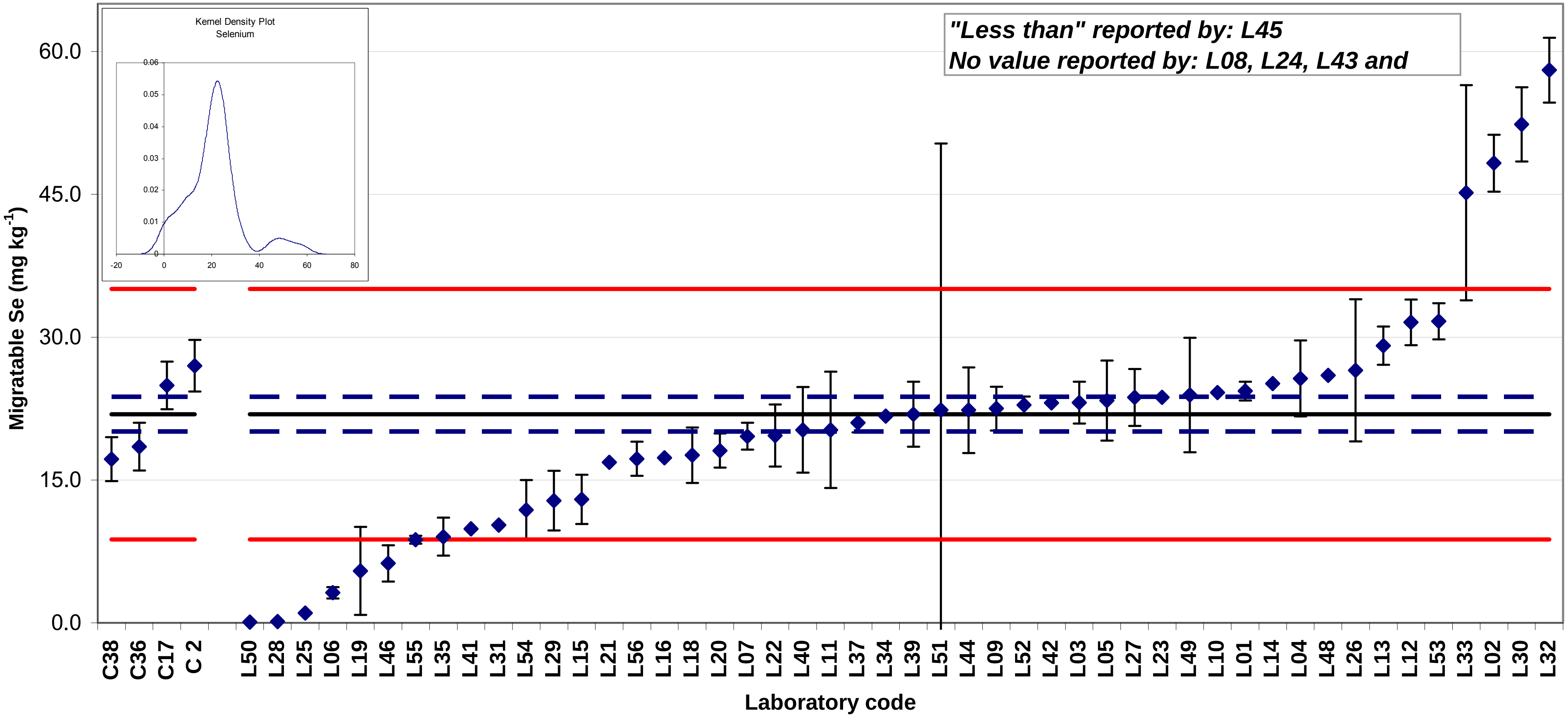
Lab ID	X_{mean}	U_{lab}	k[a]	u_{lab}	Technique	z-score[b]	ζ-score[b]	U[c]
C 1	<10	0	√3	0.00	ICP-OES			b
C 2	27.00	2.72	√3	1.57	ICP-MS			a
C17	24.93	2.5	2	1.25	ICP-OES			a
C36	18.50	2.5	2	1.25	ICP-OES			a
C38	17.20	2.3	2	1.15	ICP-OES			a
L01	24.33	1	2	0.50	ICP-OES	0.4	2.3	b
L02	48.27	3	√3	1.73	FAAS	4.0	13.5	a
L03	23.13	2.2	2	1.10	ICP-MS	0.2	0.9	a
L04	25.67	4	2	2.00	ICP-MS	0.6	1.7	a
L05	23.33	4.2	2	2.10	ICP-OES	0.2	0.6	a
L06	3.17	0.6	2	0.30	HG-AAS	-2.9	-19.5	b
L07	19.60	1.4	2	0.70	ICP-OES	-0.4	-2.0	b
L08								
L09	22.50	2.3	2	1.15	ICP-MS	0.1	0.4	a
L10	24.17	0	√3	0.00	ICP-OES	0.3	2.5	b
L11	20.27	6.1	2	3.05	ICP-OES	-0.2	-0.5	a
L12	31.55	2.39	2	1.20	ICP-OES	1.5	6.4	a
L13	29.10	2	2	1.00	ICP-OES	1.1	5.3	a
L14	25.13	0	1.96	0.00	ICP-OES	0.5	3.5	b
L15	12.97	2.6	2	1.30	ICP-OES	-1.4	-5.6	a
L16	17.33	0	√3	0.00	ICP-OES	-0.7	-5.0	b
L18	17.60	2.9	2	1.45	ICP-OES	-0.7	-2.5	a
L19	5.47	4.62	2	2.31	ETAAS	-2.5	-6.6	a
L20	18.10	1.8	500	0.00	HG-AAS	-0.6	-4.2	b
L21	16.85	0	√3	0.00	ICP-OES	-0.8	-5.5	b
L22	19.67	3.25	√3	1.88	ICP-OES	-0.3	-1.1	a
L23	23.67	0	√3	0.00	ETAAS	0.3	1.9	b
L24								
L25	1.04	0.003	√3	0.00	HG-AAS	-3.2	-22.8	b
L26	26.51	7.46	2	3.73	ICP-OES	0.7	1.2	a
L27	23.67	3	2	1.50	ICP-OES	0.3	1.0	a
L28	0.14	0.0274	2	0.01	ICP-MS	-3.3	-23.8	b
L29	12.83	3.13	2	1.57	ICP-MS	-1.4	-5.0	a
L30	52.33	3.9	2	1.95	ICP-OES	4.6	14.1	a
L31	10.27	0	√3	0.00	ICP-OES	-1.8	-12.7	b
L32	58.03	3.4	2	1.70	ICP-MS	5.5	18.7	a
L33	45.17	11.3	2	5.65	ICP-OES	3.5	4.1	a
L34	21.71	0	√3	0.00	ICP-MS	0.0	-0.2	b
L35	9.06	2	60	0.03	ICP-OES	-2.0	-14.1	b
L37	21.00	1	2	0.50	ICP-OES	-0.1	-0.9	b
L39	21.90	3.41	2	1.71	ETAAS	0.0	0.0	a
L40	20.27	4.5	√3	2.60	FAAS	-0.2	-0.6	a
L41	9.89	0.014	2	0.01	CV-AAS	-1.8	-13.2	b
L42	23.10	0	√3	0.00	ICP-OES	0.2	1.3	b
L43								
L44	22.33	4.5	2	2.25	ICP-OES	0.1	0.2	a
L45	<253.8	0	√3	0.00	FAAS			b
L46	6.25	1.9	2	0.95	ICP-MS	-2.4	-11.9	a
L47								
L48	26.00	0	√3	0.00	ICP-OES	0.6	4.5	b
L49	23.93	6	2	3.00	ICP-OES	0.3	0.6	a
L50	0.09	0.031	2	0.02	ICP-OES	-3.3	-23.9	b
L51	22.33	28	2	14.00	ICP-OES	0.1	0.0	c
L52	22.87	0.9	3	0.30	ICP-MS	0.1	1.0	b
L53	31.67	1.9	√3	1.10	ICP-OES	1.5	6.8	a
L54	11.87	3.12	2	1.56	FAAS	-1.5	-5.6	a
L55	8.73	0.41	2	0.21	ICP-MS	-2.0	-14.1	b
L56	17.23	1.8	2	0.90	ICP-OES	-0.7	-3.6	b

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

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u_{ref} \leq u_{lab} \leq \hat{\sigma}$; "b": $u_{lab} < u_{ref}$; "c": $u_{lab} > \hat{\sigma}$

IMEP-34 (Trace elements in toys): Selenium
Assigned value: X_{ref} = 21.9 mg kg^{-1}; U_{ref} = 1.8 mg kg^{-1} (k = 2)



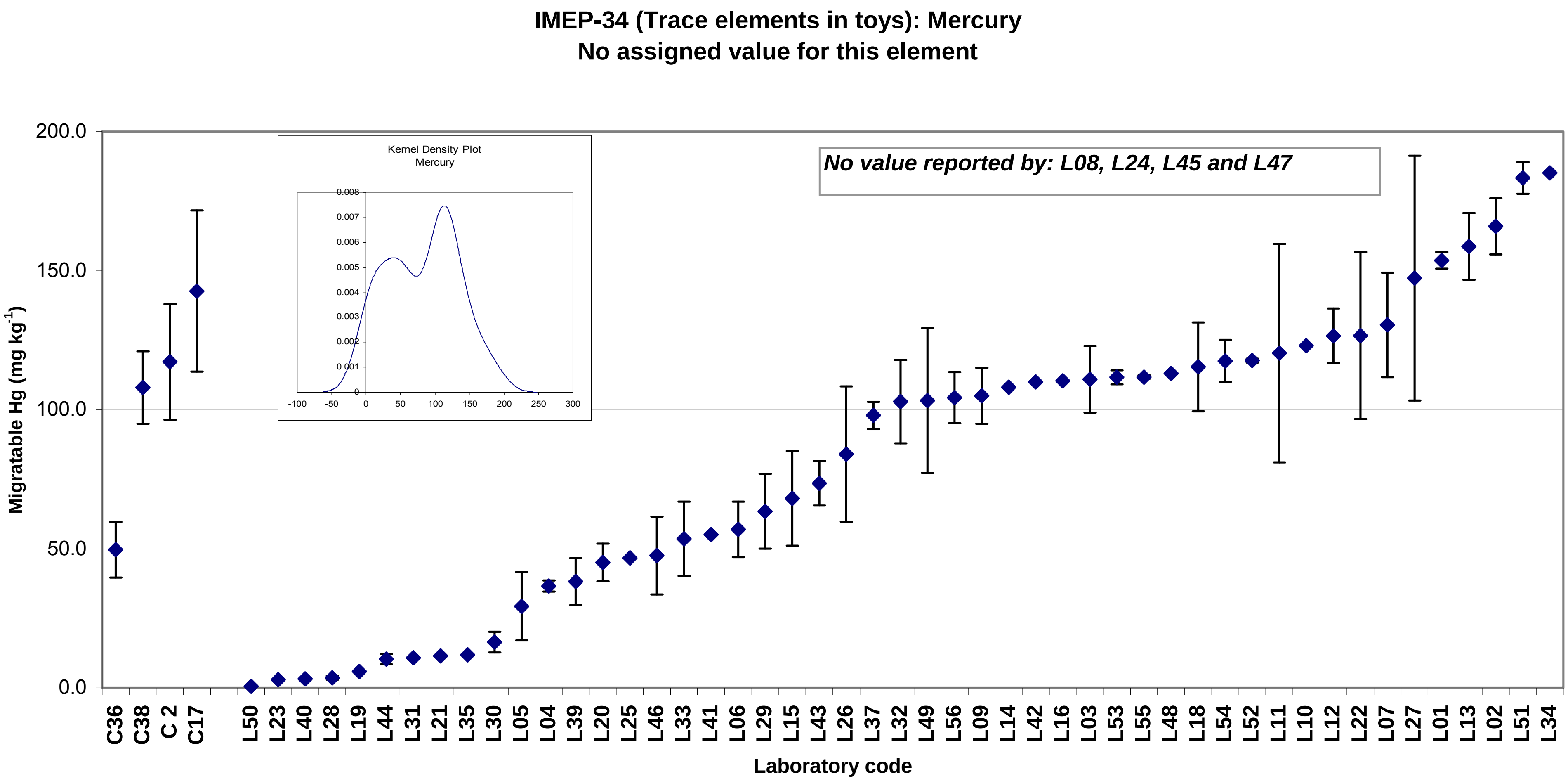
This graph displays the averaged value of the three replicates with their associated uncertainties. The uncertainties are shown as reported. The thick blak line corresponds to $\mathbf{X_{ref}}$, the blue lines to the boundaries of X_{ref} ($\mathbf{X_{ref} \pm 2u_{ref}}$) the red lines to the acceptance interval ($\mathbf{X_{ref} \pm 2\sigma}$).

Annex 17: Results for Mercury

X_{ref} = No scoring; all values are given in (mg kg^{-1})

Lab ID	X_{mean}	U_{lab}	k[a]	u_{lab}	Technique
C 1	61.60	0	√3	0.00	ICP-OES
C 2	117.18	20.81	√3	12.01	ICP-MS
C17	142.67	29	2	14.50	ICP-OES
C36	49.70	10	2	5.00	ICP-OES
C38	108.00	13	2	6.50	ICP-OES
L01	153.67	3	2	1.50	ICP-OES
L02	165.93	10.1	√3	5.83	FAAS
L03	110.96	12	2	6.00	ICP-MS
L04	36.67	2	2	1.00	FIMS
L05	29.33	12.3	2	6.15	ICP-OES
L06	57.00	10	2	5.00	CV-AAS
L07	130.53	18.8	2	9.40	ICP-OES
L09	105.00	10	2	5.00	ICP-MS
L10	123.00	0	√3	0.00	ICP-OES
L11	120.37	39.3	2	19.65	ICP-OES
L12	126.58	9.83	2	4.92	ICP-OES
L13	158.73	12	2	6.00	ICP-OES
L14	108.08	0	1.96	0.00	ICP-OES
L15	68.13	17	2	8.50	ICP-OES
L16	110.33	0	√3	0.00	ICP-OES
L18	115.40	16	2	8.00	ICP-OES
L19	5.90	0.26	2	0.13	FIMS
L20	45.10	6.77	500	0.01	FIMS
L21	11.49	0	√3	0.00	FIMS
L22	126.67	30	2	15.00	ICP-OES
L23	2.93	0	√3	0.00	CV-AAS
L25	46.68	0.087	√3	0.05	HG-AAS
L26	84.03	24.31	2	12.16	ICP-OES
L27	147.33	44	2	22.00	ICP-OES
L28	3.65	0.7304	2	0.37	ICP-MS
L29	63.50	13.43	2	6.72	ICP-MS
L30	16.47	3.7	2	1.85	CV-AAS
L31	10.82	0	√3	0.00	HG-AAS
L32	102.93	15	2	7.50	ICP-MS
L33	53.60	13.4	2	6.70	FIMS
L34	185.12	0	√3	0.00	ICP-MS
L35	11.93	0.004	50	0.00	Hydride generation-ICP-OES
L37	98.00	4.9	2	2.45	ICP-OES
L39	38.26	8.49	2	4.25	ETAAS
L40	3.23	0.3	√3	0.17	FIMS
L41	55.08	0.055	2	0.03	CV-AAS
L42	110.00	0	√3	0.00	ICP-OES
L43	73.57	8	2	4.00	AAS - mercury analyse
L44	10.33	1.9	2	0.95	ICP-OES
L46	47.57	14	2	7.00	ICP-MS
L48	113.00	0	√3	0.00	ICP-OES
L49	103.30	26	2	13.00	ICP-OES
L50	0.54	0.287	2	0.14	ICP-OES
L51	183.33	5.7	2	2.85	ICP-OES
L52	117.67	0.7	3	0.23	ICP-MS
L53	111.67	2.5	√3	1.44	ICP-OES
L54	117.53	7.58	2	3.79	CV-AAS
L55	111.74	0.59	2	0.30	ICP-MS
L56	104.33	9.2	2	4.60	ICP-OES

[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.



This graph displays the averaged value of the three replicates with their associated uncertainties. The uncertainties are shown as reported.

Annex 18: Summary of scorings

	Arsenic (As)		Antimony (Sb)		Barium (Ba)		Cadmium (Cd)		Chromium (Cr)		Lead (Pb)		Selenium (Se)	
Lab ID	z-score	ζ-score	z-score	ζ-score	z-score	ζ-score	z-score	ζ-score	z-score	ζ-score	z-score	ζ-score	z-score	ζ-score
L01	0.3	2.5	0.7	3.8	0.8	2.0	0.5	0.9	1.2	2.2	1.4	1.7	0.4	2.3
L02					3.4	7.4	0.2	0.4	2.9	6.9	4.0	9.2	4.0	13.5
L03	0.6	1.9	0.1	0.2	-1.5	-4.0	1.0	1.5	0.3	0.8	0.6	1.3	0.2	0.9
L04	0.6	2.8	0.6	2.8	-0.2	-0.7	0.8	1.5	0.6	1.9	0.6	1.4	0.6	1.7
L05	4.9	6.3	2.1	3.0	0.7	0.9	0.2	0.3	6.9	4.0	0.6	1.2	0.2	0.6
L06			4.1	5.7	2.3	2.5	4.9	3.8	5.0	5.1	5.8	9.0	-2.9	-19.5
L07	-0.5	-3.8	-0.3	-1.6	-0.3	-0.6	-0.8	-1.7	0.0	-0.1	-0.8	-1.9	-0.4	-2.0
L08	-1.4	-11.7					-1.7	-4.2	-1.1	-4.0	-2.6	-6.5		
L09	-0.8	-4.7	0.8	2.5	0.1	0.2	-1.0	-2.0	0.0	0.1	-0.4	-0.8	0.1	0.4
L10	0.2	2.0	0.9	5.4	0.9	3.0	1.4	3.5	1.5	5.7	1.5	3.8	0.3	2.5
L11	-0.5	-1.3	0.1	0.2	-0.3	-0.4	-0.5	-0.6	-0.2	-0.3	-0.1	-0.2	-0.2	-0.5
L12	3.7	7.5	1.7	5.3	0.8	1.5	0.7	1.7	0.8	1.1	3.8	4.3	1.5	6.4
L13	0.7	3.6	0.6	2.6	1.0	3.2	1.7	3.9	1.5	3.4	1.7	3.9	1.1	5.3
L14	-0.2	-1.8	0.2	1.1	0.6	2.0	0.4	1.1	-0.6	-2.2	0.3	0.8	0.5	3.5
L15	-1.9	-10.8	-1.8	-8.9	-1.1	-1.9	-1.9	-3.1	-1.3	-2.4	-1.0	-2.2	-1.4	-5.6
L16	-0.7	-6.0	-0.3	-1.9	-0.9	-3.0	-0.9	-2.3	-0.7	-2.6	-0.7	-1.8	-0.7	-5.0
L18	-0.8	-2.5	-0.6	-2.1	-0.1	-0.3	-1.4	-3.4	-0.7	-1.5	-1.0	-2.5	-0.7	-2.5
L19	-2.8	-2.2	-2.9	-4.7	-0.8	-2.5	-1.9	-4.7	-1.3	-3.7	1.0	2.3	-2.5	-6.6
L20	-0.3	-2.5			0.6	2.2	-0.7	-1.7	1.6	6.0	0.3	0.8	-0.6	-4.2
L21	-1.8	-14.6	-0.4	-2.7	-0.3	-1.0	-1.5	-3.7	-0.7	-2.7	-1.1	-2.7	-0.8	-5.5
L22	-0.2	-0.5	-0.8	-1.8	-0.5	-0.8	-0.8	-1.0	0.6	0.5	-1.3	-2.6	-0.3	-1.1
L23	0.5	4.2	-0.4	-2.7	1.1	3.6	0.9	2.3	2.6	9.8	2.6	6.5	0.3	1.9
L24							-1.6	-2.7	-2.2	-4.7	-1.1	-2.8		
L25	-2.9	-23.8	-3.3	-20.0	-0.9	-1.8	7.5	14.3	0.4	0.8	14.2	13.0	-3.2	-22.8
L26	0.2	0.5	-0.7	-1.7	0.2	0.3	2.1	1.7	1.0	1.2	1.4	2.1	0.7	1.2
L27	0.5	0.9	0.4	1.6			0.3	0.6			0.4	1.0	0.3	1.0
L28	-3.3	-27.6	-3.3	-19.9	-6.6	-22.2	-6.6	-16.5	-6.6	-24.6	-5.8	-14.6	-3.3	-23.8
L29	-1.8	-8.4	-1.8	-7.4	-2.2	-3.5	-1.8	-2.5	-1.4	-2.1	-1.4	-2.8	-1.4	-5.0
L30	3.4	5.4	8.6	12.0	2.1	3.9	6.1	11.8	5.2	8.2	9.2	22.7	4.6	14.1
L31	-1.3	-10.4	-0.2	-1.0	-0.4	-1.2	-0.6	-1.6	0.0	-0.1	-0.3	-0.9	-1.8	-12.7
L32	6.3	52.2	10.7	16.5	2.2	2.0	8.5	9.8	7.8	6.5	16.3	20.0	5.5	18.7
L33	2.6	3.4	3.2	3.9	4.8	3.3	3.3	2.5	3.1	2.5	4.8	6.1	3.5	4.1
L34	0.0	-0.1	-0.3	-1.6	-0.1	-0.4	-0.5	-1.2	0.4	1.4	-0.3	-0.6	0.0	-0.2
L35	-2.1	-17.7	-1.9	-11.7	-2.1	-7.1	-2.0	-5.1	-2.2	-8.3	-2.1	-5.2	-2.0	-14.1
L37	-0.6	-4.3	-0.6	-3.3	1.0	2.8	-0.7	-1.7	0.0	0.0	-0.8	-2.1	-0.1	-0.9
L39	-1.3	-9.1	4.8	9.5	-0.6	-0.7	-5.3	-13.3	-3.4	-11.7	-5.4	-13.4	0.0	0.0
L40			-0.9	-3.1	-1.4	-2.6	-0.5	-1.0	-1.1	-2.4	0.0	-0.1	-0.2	-0.6
L41	-1.2	-10.1	2021.3	322.9			-1.3	-3.3	-0.5	-1.7	0.4	1.1	-1.8	-13.2
L42	0.7	6.1	0.9	5.4	1.0	3.3	0.6	1.4	1.2	4.6	0.9	2.2	0.2	1.3
L43	-2.0	-11.1	-2.2	-10.4			-2.3	-4.6	-1.1	-2.1	-1.6	-3.0		
L44	-1.1	-4.4	0.3	0.7	-0.7	-1.2	-1.2	-1.9	0.6	0.8	2.8	5.0	0.1	0.2
L45														
L46	-2.2	-11.3	-2.2	-9.2	-2.7	-4.2	-3.4	-5.4	-2.4	-3.4	-2.4	-5.1	-2.4	-11.9
L47							0.0	0.0	-1.8	-2.4	2.2	3.7		
L48	-0.4	-3.1	0.3	1.6	0.2	0.6	-0.5	-1.2	1.2	4.5	0.1	0.3	0.6	4.5
L49	-0.7	-1.8	0.2	0.4	-0.7	-1.2	-1.0	-1.5	-0.6	-0.9	-0.6	-1.2	0.3	0.6
L50	-3.3	-27.6	-3.3	-19.9	-6.6	-22.2	-6.6	-16.6	-6.6	-24.6	-5.8	-14.6	-3.3	-23.9
L51	-0.8	-0.2	0.7	0.2	0.1	0.1	-0.2	-0.3	0.7	0.2	-0.1	0.0	0.1	0.0
L52	0.2	1.6	0.5	2.8	0.1	0.3	0.2	0.6	0.9	2.4	0.3	0.8	0.1	1.0
L53	0.7	1.0	1.5	2.9	1.4	4.6	2.3	5.2	1.8	2.3	2.1	4.6	1.5	6.8
L54			-0.3	-0.9	0.4	0.9	-3.0	-6.0	0.1	0.1			-1.5	-5.6
L55	-2.1	-13.3	-2.5	-14.0	-3.1	-10.4	-3.0	-7.5	-3.0	-9.8	-3.3	-8.2	-2.0	-14.1
L56	0.1	0.8	0.1	0.2	-0.8	-2.0	-0.6	-1.2	-0.4	-1.1	-0.3	-0.7	-0.7	-3.6

Annex 19 A: Compliance assessment to Directive 88/378/EEC

LCode	Directive 88/378/EEC	
		Explain why:
C 1	Yes	
C17	No	Migration of mercury (with analytical correction) is over the limit of 60 mg/kg.
C36	Yes	
C38	Yes	-
L02	No	The concentration of the metals analysed is out of the specification given on the EN 71-3.
L05	Yes	All results below max permitted.
L06	Yes	in accordance with EN-71/3:2005
L07	No	Corrected Mercury value is 65.25 mg/kg. Limit after correction is 60 mg/kg
L08	Yes	
L09	No	the corrected value for Mercury is above the limit (60 mg/kg)
L10	No	The soluble mercury content of the material has exceeded the Toy Safety Directive 88/378/EEC limit.
L11	No	Adjusted result of Hg exceeds the limit of 60 mg/kg.
L12	No	the limit for mercury is exceeded
L13	Yes	
L15	No	mercury (Hg) content is too high
L16	No	High Mercury, uncertain even if 50% analytical correction was applied
L18	Yes	Affer applying correction factor, all results are below limits of EN 71 Part 3:1994 + A1:2000/AC:2002
L19	Yes	all elements keep the limits
L20	No	
L21		
L22	No	Hg > 60mg/kg
L23	Yes	All elements are < migration limit before correction
L24		
L25	No	
L26	Yes	
L27	No	The concentration of Hg exceed the limit in the standard
L28	Yes	Below the limits of element migration (EN71-3:1994)
L29	No	Because of the high level of migration of Mercury
L30		
L31	No	
L32	No	In this directive only the total amount of metals per day is stated not the maximum levels in mg/kg as in EN71-3. Whit that information you can not decide if the material is safe on the market.
L33	Yes	Measured values below limits
L34	No	migration limit Pb to high
L35	No	no opinion
L37	Yes	
L39	Yes	Directive corresponds to the limit values of EN 71-3. All limit values are met by the sample.
L40	Yes	
L41	Yes	in case of results below the limit in accordance EN 71-3
L42	Yes	According to 88/378/CE directive, the EN 71-3 (december 1994) + A1 April 2000 standard gives presumption of conformity to the essential safety requirements given in Annex II - II - 3. 2 biodisponibility.The corrected analytical results show that for all the elements the amount of heavy metals quantified are under the limits given in EN 71-3 (december 1994) + A1 April 2000 - clause 4.1 - table 1.
L43	Yes	normative document for EU member States for migration EN 71-3
L44	Yes	Because all the results are below the maximum allowed limits
L45	Yes	Received values the migrated concentrations of Sb, Ba, Cd, Cr, Pb, Se don't exceed safety limits specified in the harmonised European Standard EN 71-3:1994
L46		This judgement is not done by our laboratory, but by the costumers themself
L47	Yes	
L48	Yes	the results are under the limits stated in the EN71/3
L49	No	Hg
L50	Yes	it is very importat for health of children
L51	Yes	
L52	No	Several elements with applied correction are above the limits (based on a 0.1 g sample) (i.e. As, Cd, Sb, Hg)
L53	Yes	Every values except Hg are below limits. For Hg (112 mg/kg) we apply AC 50% and the new result (56mg/kg) is below the limit too.
L55	Yes	We still use the test method of EN 71-3 and requirement from this direction.
L56	Yes	All results are passed

Annex 19 B: Compliance assessment to Directive 2009/48/EC

LCode	Directive 2009/48/EC	
		Explain why:
C 1	No	
C17	Yes	Based on the limits of **scraped-off** toy material this test material would agree with the limits of the toy safety directive when analytical corrections from 71-1:1994 are used.
C36	No	Not all elements have been determined
C38	Yes	the positive evaluation is based only on the elements requested and if the actual analytical tollerance for Hg will be confirmed by the NEW EN 71-3 and does not consider the Cr VI requirement due to there is not a validated method
L02	No	The concentration of the metals analysed is out of the specification given on the EN 71-3.
L05	Yes	All results below max permitted.
L06	No	No results for Cr VI and org. tin compounds. Pb,Hg above the limit
L07	No	**Uncorrected values for (Cd 23.4 mg/kg, Hg 130.5 mg/kg) are over limit (Cd 23 mg/kg, Hg 94 mg/kg)**
L08	Yes	
L09		I don't know because we don't have a standard for all the metals descrived in this directive and if the correction factor remains the same for the elements.
L10	No	The soluble mercury content of the material has exceeded the Toy Safety Directive 2009/48/EC limit.
L11	Yes	**All 8 adjusted results are less than the limits of "scraped-off toy material".**
L12	No	the limits for arsenic, mercury, lead and cadmium are exceeded (considering the limits for powder-like material)
L13		
L15	No	Cadmium (Cd)-Mercury(Hg) content are too high
L16	No	**Scrapeable Material contains excess mercury. Cadmium is on the limit.**
L18	No	Result exceed regulatory limit (Decision based on tested 8 elements). No analytical correction factor was mentioned in 2009/48/EC.
L19	No	not all elements claimed in 2009/48/EC were tested
L20		
L21		
L22	No	As > 3.8, Cd > 1.9, Hg > 7.5 mg/kg
L23	No	Cd >migration limit (1,9 mg/kg) after correction
L24		
L25	Yes	In Chile there is no legislation to control toys, this is only done when they are exported, no control is performed for toys importand is why it is very interesting work, implement and test the toys under the Directive 2009/48/EC on the safety of toys
L26		
L27	No	The concentration of Hg exceed the limit in the standard
L28	Yes	**Below the limits of element migration (EN71-3:1994)**
L29	No	There isn't an harmonized standard for 2009/48/EC yet
L30	No	Because the values of lead, cadmium, mercury, selenium and arsenic are exceeded the migration limits from the Directive.
L31		
L32	No	The As, Cd, Pb and Hg level exceeds the maximum level allowed in toys according to 2009/48/EEC. See Annex II, III Chemical properties, part 13 in column 1 (in dry, brittle, powder-like or pliable toy materials) in the table.
L33	Yes	Measured values below limits
L34	Yes	complies all limits
L35	No	no opinion
L37	Yes	
L39	No	Limit values for Cadmium and Mercury are exeeded even by the corrected mean values. Arsenic is exeeded by the raw value.
L40	No	
L41	Yes	in case of results below the limit in accordance EN 71-3
L42	No	The new directive 2009/48/EC deals with 19 elements and has differents limits againts the nature of the material (powder, liquid, etc....). The current EN 71-3 (december 1994)+A1 April 2000 deals with only 8 elements. This standard is under revision to update the list of elements and tests methods. For this reason we can not conclude on the conformity in regards of the 2009/48/CE directive.
L43	Yes	normative document for EU member States
L44	Yes	
L45	Yes	Received values the migrated concentrations of Sb,Pb, Se, Ba don't exceed safety limits specified in the harmonised European Standard EN 71-3:1994. For elements Cr, Cd we can't state it.
L46		This judgement is not done by our laboratory, but by the costumers themself
L47	No	the limits for metals are too permisive
L48		
L49	No	Hg, As, Cd
L50	No	it is not nesesary at this time
L51	Yes	
L52	Yes	**The higher requirement limits for material 'scraped off toys' allows a passing rating for all elements**
L53	No	**For Cd the limit is 23 mg/kg and our result is 36 and for Hg limit is 94 and our result is 112 mg/kg.Our results are only based of the result of 8 heavy metals out of 17.**
L55	Yes	New chemical requirement is not enforced yet.
L56	No	Cd>1.9 mg/kg, Hg>7.5 mg/kg, As>3.8 mg/kg

Annex 20: Experimental details extracted from the questionnaire

Lab ID	Sieved sample?	Mesh size	Sample amount	Shaking device	37 °C used?	Centri-fugation	Analyse on day of preparation?
C 1	No		0,2g	multi magnetic stirrers plancha	Yes	No	No
C 2	No		0.50 g	Shaking water bath	Yes	No	Yes
C17	No		0,5 g	water bath with a shaker	Yes	No	Yes
C36	No		0.2 g	Thermostatted waterbath with shaking	Yes	No	Yes
C38	Yes	0.5 mm	200 mg	orbital shaker	Yes	No	Yes
L01							
L02	Yes	0,05	Rep2: 2.0407g ; Rep3: 1.9758g	a water-bath with a shaking device	Yes	No	Yes
L03	No		0.5 g	magnetic stirring	Yes	No	No
L04	No		0.5 g	Magnetrührer	Yes	No	Yes
L05	Yes	0.5 mm	0.2g	A shaking thermostated water bath.	Yes	No	No
L06	No		0.5	magnetic stirring bar	Yes	No	Yes
L07	Yes	0.5 mm	0.5 g	Orbital Shaker	Yes	No	Yes
L08	No		0.5 g	incubating shaker	Yes	No	No
L09	No		0,5 g	water bath with linear agitation (150 rpm)	Yes	No	Yes
L10	No	N/A	0,5 g	Shaking water bath	Yes	No	Yes
L11	Yes	a metal sieve with an aperture of 0,5 mm	0.2 g	Constant Temperature Water Bath Shaker	Yes	No	Yes
L12	No		Rep1 = 0.5016g ; Rep2 = 0.5016g ; Rep3 = 0.5023g	manual stirring	Yes	No	No
L13	No		0.5 g	Thermostatic Shake bath	Yes	No	Yes
L14	Yes	whatman 41	0.31 g	shaking water bath	Yes	No	Yes
L15	No	-	solid	shaking water bath dub0ff	Yes	No	Yes
L16	No		100 mg	Reciprocating (shaking) water bath (Grant SS40)	Yes	No	Yes
L18	Yes	0.5mm	0.5g	shaking water bath	Yes	No	Yes
L19	No		1 g	heated water bath shaker	Yes	No	Yes
L20					Yes		No
L21	No		2.5 ml		Yes	No	No
L22	No		0.5 g	orbital shaker	Yes	No	No
L23	Yes	0,5 mm	0,5 g	Lateral oscillating bath	Yes	No	No
L24							
L25	No		0.5 grams	HEAT-STIR/STUART/SERIAL:R 00106763	Yes	No	No
L26	No		0.5 g	shaked thermostatic bath	Yes	Yes	Yes
L27	No		0.5011 gr; 0.5011 gr; 0.5025 gr	Magnetic	Yes	No	Yes
L28	No		50mg	Magnetic stirrer	Yes	No	Yes
L29	No		1 g	automatic shaker OXYTOP	Yes	No	No
L30	No		Rep1:1.0045g; rep2:1.0034g; rep3:1.0032g	magnetic stirrer	No	No	Yes
L31	No		0.5 g in 25 ml 0.07N HCl	forwards and backwards movement	Yes	No	No

Lab ID	Sieved sample?	Mesh size	Sample amount	Shaking device	37 °C used?	Centri-fugation	Analyse on day of preparation?
L32	No		0.5 g	Shaking Water bath.	Yes	No	Yes
L33	No		0.5 g		Yes	No	No
L34	No		500 mg	waterbath	Yes	No	Yes
L35	No		0,5 g	magnetic stirrer	Yes	No	Yes
L37	No		500 mg	Shaking waterbath	Yes	No	Yes
L39	No	-	500 mg	waterbath with shaking device for bottles, drying oven	Yes	No	Yes
L40	Yes	0.5 mm	0.6 g	shaking device Julabo SW-20C	Yes	No	Yes
L41	No		0,5 g	shaker laboratory equipment	Yes	No	No
L42	No		200 mg	Orbital shaker	Yes	No	Yes
L43	No	-	0,5 g	shaking device LT-2	Yes	No	No
L44	No		1 gram	swinging shaker	Yes	No	Yes
L45	No	-	0,5 g	water bath with shaking device Type WB-14, Memmert GmbH + CO. KG, Germany	Yes	No	No
L46	No		0.5 g	shaking table	Yes	No	Yes
L47	No		0.05 g	ultrasonic method	Yes	No	Yes
L48	Yes	mesh size: 0.5mm	rep.1: 0.5013 g, rep.2: 0.5179g, rep. 3 : 0.5146g	water shaker bath 150rpm	Yes	No	Yes
L49	No		0.5 g	Enviromental Shaker ES 20	Yes	No	Yes
L50	No	Not applicable	at least 0.5g	Thermoshake Gerhardt	Yes	No	Yes
L51	No		0.5 g	end over end shaker	Yes	No	Yes
L52	Yes	500 µm	0.10 g	shaking water bath	Yes	No	Yes
L53	Yes	0.5 mm	0.15g	shaking water bath	Yes	No	Yes
L54	No		0.5 g	Magnetic stirred with heating	Yes	No	Yes
L55	No		0.5 g	Water Shaker bath	Yes	No	Yes
L56	Yes	500 µm	0.2g	water bath with shaking	Yes	No	Yes

European Commission

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Title: **Heavy metals in toys according to EN 71-3:1994**

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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 soluble antimony (Sb), arsenic (As), barium (Ba), cadmium (Cd), chromium (Cr), lead (Pb), mercury (Hg), and selenium (Se) according to European Standard EN 71-3:1994.

The principle of the procedure in EN 71-3:1994 [1] consists in the extraction of soluble elements from toy material under the conditions simulating the material remaining in contact with stomach acid for a period of time after swallowing.

Fifty eight participants from twenty six countries registered to the exercise, of which 54 reported results for As, Sb, Ba, Se and Hg and 58 for Cr, Pb, and Cd, respectively.

The test item used was a certified reference material (CRM 623, comminuted paint flakes from alkyd resin paint), certified in 1998, which is not included anymore in the CRM catalogue. The validity of the certified values was assessed using some expert laboratories in the field. In most of the cases the results reported by the certifiers were not in agreement with the CRM reference values. The mean of the means reported by the expert laboratories was used as assigned value for the different measurands. The results reported by the expert laboratories for mercury were very scattered (RSD = 37.6 %). No assigned value could be attributed for mercury and therefore no scores were provided to the participants for this measurand.

The associated uncertainties of the assigned values were obtained following the ISO GUM. Furthermore, participants were invited to report their measurement uncertainties. This was done by all laboratories having submitted results in this exercise.

Laboratory results were rated with *z*- and zeta (*ζ*-) scores in accordance with ISO 13528. The standard deviations for proficiency assessment were based on the analytical correction laid down in EN 71-3:1994.

The outcome of the exercise shows an improvement on the overall performance of the participants when compared to IMEP-24 (a proficiency test for heavy metals in toys run in 2009 in which the same European standard was followed), particularly for cadmium, lead and to a lesser extent, for selenium and chromium. The share of satisfactory *z*-scores ranged from 65 to 79 %.

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As the Commission's in-house science service, the Joint Research Centre's mission is to provide EU policies with independent, evidence-based scientific and technical support throughout the whole policy cycle.

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