JRC TECHNICAL REPORT

Determination of the mass fraction of Cd and Pb released from ceramic bowls

Proficiency Testing Report
FCM-20/02 (Part 1)

Fernando Cordeiro, Silvia García-Ruiz, James Snell, Geert Van Britsom, Pieter Dehouck, Aneta Cizek-Stroh, Piotr Robouch, Eddo Hoekstra

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Joint Research Centre

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268-PT Accredited by the
Belgian Accreditation Body (BELAC)

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Executive summary

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM) organised a proficiency test (FCM-20/02 Part 1) for the determination of the mass fraction of cadmium (Cd) and lead (Pb) released from ceramic bowls. This PT round was organised in the frame of a possible revision of the Ceramic Directive 84/500/EEC in which the limits for cadmium and lead mass fractions migrating from ceramic artefacts are to be lowered, and to recommend the use of three successive migrations. This proficiency test was open to National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs).

The test item consisted of ceramic bowls. The homogeneity of the test item was evaluated and the assigned values were derived from the results determined by the EURL-FCM.

Twenty-four NRLs from 23 Member States and one OCL from Switzerland registered to the exercise. All laboratories reported their results.

Laboratory results were rated using (z) and zeta (ζ) scores in accordance with ISO 13528:2015. A relative standard deviation for proficiency assessment (σ_{pt}) of 30 % of the respective assigned values was set for the two measurands based on the opinion of experts.

Eighty percent of the participating laboratories performed satisfactorily (according to the z score) for the analysis of the mass fractions of Cd and Pb after the first migration. These performances confirm that most NRLs are able to monitor the mass fractions of Cd and Pb in the frame of Directive 84/500/EEC.

List of abbreviations and symbols

AAS	Atomic Absorption Spectrometry
DG SANTE	Directorate General for Health and Food Safety
EC	European Commission
EU	European Union
EURL	European Union Reference Laboratory
FCM	Food Contact Materials
FAAS	Flame Atomic Absorption Spectrometry
GUM	Guide for the Expression of Uncertainty in Measurement
GF-AAS	Graphite Furnace - Atomic Absorption Spectrometry
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
ICP-OES	Inductively Coupled Plasma – Optical Emission Spectrometry
ILC	Interlaboratory Comparison
ISO	International Organization for Standardization
JRC	Joint Research Centre
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PT	Proficiency Test
k	coverage factor
σ_{pt}	standard deviation for proficiency assessment
$u(x_i)$	standard measurement uncertainty reported by participant "i"
$u(x_{pt})$	standard uncertainty of the assigned value
u_{char}	(standard) uncertainty contribution due to characterisation
u_{hom}	(standard) uncertainty contribution due to homogeneity
u_{st}	(standard) uncertainty contribution due to stability
$U(x_i)$	reported expanded uncertainty by participant "i"
$U(x_{pt})$	expanded uncertainty of the assigned value
x_i	mean value reported by participant "i"
x_{pt}	assigned value
z	z score
ζ	zeta score

1 Introduction

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM), hosted by the Joint Research Centre (JRC) of the European Commission, organised a proficiency testing (PT) round for the determination of the mass fraction of cadmium (Cd) and lead (Pb) migrating from ceramic bowls into 4 % v/v acetic acid solution (food simulant solution) under controlled conditions. This PT round was organised in support to the Ceramic Directive 84/500/EEC [1] to lower the legal limits of Cd and Pb and to recommend the use of three successive migrations.

This PT was agreed with the Directorate General for Health and Food Safety (DG SANTE) as part of the EURL-FCM annual work programme 2020, thus complying with the mandate set in Regulation (EU) 2017/625 [2]. The PT round was open to National Reference Laboratories (NRLs) and to Official Control Laboratories (OCLs) willing to participate.

This report summarises the outcome of this PT round.

2 Scope

The present PT aims to assess the performance of NRLs and OCLs in the determination of the mass fractions of Cd and Pb migrating from ceramic bowls under controlled conditions. The PT was mandatory for the NRLs and open to OCLs (under certain conditions). Participants were asked to provide a compliance statement for the test item in relation to Council Directive 84/500/EEC [1].

This PT organised in line with ISO 17043:2010 [3] is identified as "FCM-20/02 (Part 1)".

3 Set up of the exercise

3.1 Quality assurance

The JRC Unit hosting the EURL FCM is accredited according to:

- ISO/IEC 17025:2017 (certificate number: BELAC 268-TEST); and
- ISO/IEC 17043:2010 (certificate number: BELAC 268-PT, proficiency test provider)

The reported results were evaluated following the relevant administrative and logistic procedures.

3.2 Confidentiality

The procedures used for the organisation of PTs guarantee that the identity of the participants and the information provided by them is treated as confidential. The participants in this PT received a unique laboratory code used throughout this report. However, the laboratory codes of NRLs appointed in line with Regulation (EU) 2017/625 [2] may be disclosed to DG SANTE upon request for the purpose of an assessment of their (long-term) performance. Similarly, laboratory codes of appointed OCLs may be disclosed to their respective NRL upon request.

3.3 Time frame

The FCM-20/02 (Part 1) PT round was announced by invitation letters to NRLs and OCLs on March 2, 2020 (Annex 1). The registration deadline was set to March 16, 2020. Samples were sent to participants on October 12, 2020. Due to the coronavirus pandemics, the deadline for reporting of results was extended until January 29, 2021.

3.4 Distribution

Each participant received:

- The test item consisting of four ceramic bowls;
- The "Test item accompanying letter" (Annex 2); and
- A "Confirmation of receipt form" to be sent back to the PT coordinator after receipt of the test item (Annex 3).

Samples were sent under normal transport conditions. No cooling was needed during dispatch.

3.5 Instructions to participants

Detailed instructions were given to participants in the "Test item accompanying letter" mentioned above.

The measurands were defined as the mass fractions of Cd and Pb released from the ceramic bowls.

Every participant was requested:

- to perform all migration experiments following the procedure described in Council Directive 84/500/EEC Annex 1;
- to use freshly prepared food simulant solution for each migration experiment;
- to perform the migration experiments on at least three different ceramic bowls;
- to carry out three consecutive migrations at 22 ± 2 °C for 24 ± 0.5 h for each ceramic bowl,
- to determine the Cd and Pb mass fractions after each migration (1st (M1), 2nd (M2) and 3rd (M3) migration);
- to assume the density of the food simulant solution to be equal to 1 kg dm^{-3}.

Participants were asked (i) to check whether the bowls were undamaged after transport, and (ii) to return the "Confirmation of receipt" form within 3 days after receipt of the samples.

Participants were instructed to store test items at room temperature and away of any possible contamination.

Participants were asked to report, for every migration, the average total mass fractions of Cd and Pb (x_i), the associated expanded measurement uncertainty ($U(x_i)$) together with the coverage factor (k), and the analytical technique used for the analysis.

Results had to be reported in the same format (e.g. number of significant figures) as normally reported to customers.

Participants were informed that the procedure used for the analysis should resemble as closely as possible their routine procedures.

Participants received an individual code to access the on-line reporting interface, to report their measurement results and to complete the related questionnaire. The latter was designed to gather additional information related to measurements and laboratories (Annex 4).

Random laboratory codes were attributed and communicated to participants by e-mail.

4 Test item

4.1 Preparation

Innovarcilla, the Spanish technology centre of ceramics (www.innovarcilla.es) has manufactured, under control conditions, a homogeneous batch of ceramic bowls complying with the technical specifications (Cd and Pb spiking levels) provided by the EURL-FCM. A total of 200 ceramic bowls were delivered to the JRC in Geel. These bowls were dispatched to the participants without any further treatment.

4.2 Homogeneity and stability

Measurements for the homogeneity study and the statistical treatment of data were performed by the EURL-FCM.

The assessment of homogeneity was performed before the sample distribution to participants. Ten ceramic bowls were randomly selected. The first, second and third migrations (performed as prescribed at 22 ± 2 °C for 24 ± 0.5 h) were carried out for each ceramic bowl. After each migration, the mass fractions of Cd and Pb in the food simulant solutions were determined applying the single-laboratory validated method based on inductively coupled plasma – mass spectrometry (ICP-MS). The results were evaluated according to ISO 13528:2015 [4].

Due to the specificity of such measurements, whereby replicated migrations cannot be performed on the same bowl, the experimental standard deviation among ceramic bowls was taken as the standard deviation due to a potential inhomogeneity (u_{hom}), according to ISO 13528:2015 Annex B [4]. A standard deviation (s_r) was estimated for Cd and Pb measurements by ICP-MS (independently of this homogeneity study) under analytical repeatability conditions on the food simulant solution. The analytical variance (s_r^2) was subtracted from the overall experimental variance (s_{bb}^2, between-bowl variance) to derive the variance due to potential inhomogeneity, and consequently the standard uncertainty on the inhomogeneity (u_{hom}).

Both measurement targets proved to be adequately homogeneously distributed (Annex 5).

The ceramic bowls were assumed to be sufficiently stable over the whole period of this PT round. Hence, the corresponding uncertainty contribution due to stability was set to zero (u_{st} = 0) for Cd and Pb in line with ISO 13528 [4].

5 Assigned values and corresponding uncertainties

5.1 Assigned values

The results obtained in the frame of the homogeneity study were used to derive the assigned value (x_{pt}) for Cd and Pb after the first, second and third migrations performed on ten bowls (see mean values in Annex 5).

5.2 Associated uncertainties

The associated standard uncertainties of the assigned values ($u(x_{pt})$) were calculated following the law of uncertainty propagation, combining the standard measurement uncertainty of the characterization (u_{char}) with the standard uncertainty contribution from homogeneity (u_{hom}), in compliance with ISO 13528:2015 [4]:

$$u\left(x_{pt}\right) = \sqrt{u_{char}^2 + u_{hom}^2} \qquad \text{Eq. 1}$$

The uncertainty u_{char} was estimated according to the Guide to the Expression of Uncertainty in Measurement [5]. The following sources of uncertainty were taken into account: calibration standards, background correction, procedural blank, and the density of the food simulant solution. The calculated values are presented in Table 1.

5.3 Standard deviation for proficiency assessment, σ_{pt}

Based on the expert opinion and the knowledge acquired in a previous PT round, all the relative standard deviations for proficiency assessment (σ_{pt}) were set to 30 % of the respective assigned values for the mass fraction of Cd and Pb in the food simulant solution for the three migrations investigated (Table 1).

Table 1: Assigned values (x_{pt}), associated expanded measurement uncertainties ($U(x_{pt})$, k = 2), standard deviation for the PT assessment (σ_{pt}) and other relevant parameters for the assessment of results related to the determination of Cd and Pb migrated from ceramic bowls. M1, M2, M3 for the 1st, 2nd and 3rd migration, respectively.

Element	x_{pt} mg kg^{-1}	u_{char} mg kg^{-1}	u_{hom} mg kg^{-1}	$u(x_{pt})$ mg kg^{-1}	σ_{pt} mg kg^{-1} (%)	$u(x_{pt})/\sigma_{pt}$
Cd (M1)	0.0508	0.0005	0.0045	0.0045	0.0152 (30 %)	0.29
Cd (M2)	0.0417	0.0004	0.0013	0.0013	0.0125 (30 %)	0.10
Cd (M3)	0.0240	0.0002	0.0015	0.0015	0.0072 (30 %)	0.21
Pb (M1)	0.467	0.0115	0.032	0.034	0.140 (30 %)	0.24
Pb (M2)	0.375	0.009	0.015	0.017	0.113 (30 %)	0.15
Pb (M3)	0.218	0.005	0.011	0.012	0.065 (30 %)	0.18

6 Evaluation of results

6.1 Scores and evaluation criteria

The individual laboratory performances were expressed in terms of z and ζ scores according to ISO 13528:2015 [4]:

$$z = \frac{x_i - x_{pt}}{\sigma_{pt}} \quad \text{Eq. 2}$$

$$\zeta = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}} \quad \text{Eq. 3}$$

where: x_i is the measurement result reported by a participant;
$u(x_i)$ is the standard measurement uncertainty reported by a participant;
x_{pt} is the assigned value;
$u(x_{pt})$ is the standard measurement uncertainty of the assigned value;
σ_{pt} is the standard deviation for proficiency assessment.

The interpretation of the z and ζ performance scores is done according ISO 13528:2015 [4]:

$\lvert score \rvert \leq 2$	satisfactory performance	(green in Annexes 6 - 12)
$2 < \lvert score \rvert < 3$	questionable performance	(yellow in Annexes 6 - 12)
$\lvert score \rvert \geq 3$	unsatisfactory performance	(red in Annexes 6 - 12)

The z scores compare the participant's deviation from the assigned value with the standard deviation for proficiency test assessment (σ_{pt}) used as common quality criterion.

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

The standard measurement uncertainty of the laboratory $u(x_i)$ was obtained by dividing the reported expanded measurement uncertainty by the reported coverage factor, k. When no uncertainty was reported, it was set to zero ($u(x_i) = 0$) by the PT coordinator. When k was not specified, the reported expanded measurement uncertainty was considered by the PT coordinator as the half-width of a rectangular distribution; $u(x_i)$ was then calculated by dividing this half-width by $\sqrt{3}$, as recommended by Eurachem [6].

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

The relative standard measurement uncertainty from the laboratory $u_{rel}(x_i)$ is most likely to fall in a range between a minimum and a maximum allowed uncertainty (case "a": $u_{rel,min} \leq u_{rel}(x_i) \leq u_{rel,max}$). $u_{rel,min}$ is set to the relative standard uncertainty of the assigned value $u_{rel}(x_{pt})$.

It is unlikely that, a laboratory carrying out the analysis on a routine basis, would determine the measurand with a smaller measurement uncertainty than; (i) the uncertainty of the assigned value established by expert laboratories (ISO 13528:2015 §7.6 [4]); (ii) the one estimated by formulation (ISO 13528:2015 §7.3 [4]); (iii) the certified measurement uncertainty associated with a certified reference material property value (ISO 13528:2015 §7.4 [4]).

$u_{rel,max}$ is set to the relative standard deviation accepted for the PT assessment ($\sigma_{pt,rel} = \sigma_{pt}/x_{pt}$). Consequently, case "a" becomes: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt,rel}$.

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

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

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

6.2 General observations

Twenty-four NRLs from 23 EU Member States and one OCL (from Switzerland) registered to the exercise. All participating laboratories reported their values for the two measurands. Laboratory L13 reported the mass fractions of Cd and Pb released from ceramic after the first migration only.

Most of the participants applied ICP-MS (64 %), or ICP coupled with optical emission spectrometry (ICP-OES, 16 %). The remaining participants (20 %) used atomic absorption spectrometry (AAS), coupled with a graphite furnace (GF-AAS) or a flame (FAAS).

Annex 12 is providing all experimental details.

6.3 Laboratory results and scorings

6.3.1 Performances

Annexes 6 to 11 present the reported results as tables and graphs for each measurand.

The corresponding Kernel density plots were produced using the Excel add-in available from the Statistical Subcommittee of the Analytical Methods Committee of the UK Royal Society of Chemistry [7].

Figure 1 presents the overall performance of the participating laboratories expressed as z or ζ scores.

Several months after the closing date for reporting, L26 acknowledged having inverted the Cd results for the 1st and 2nd migration.

Most of the laboratories performed satisfactorily the three migrations for Pb (above 80 %) and Cd (above 70 %) according to the z score (Figure 1). L15 and L19 reported the lowest values, while L06, L08 and L12 reported the highest ones for the mass fractions of Cd and Pb in the migration solutions.

6.3.2 Measurement uncertainties

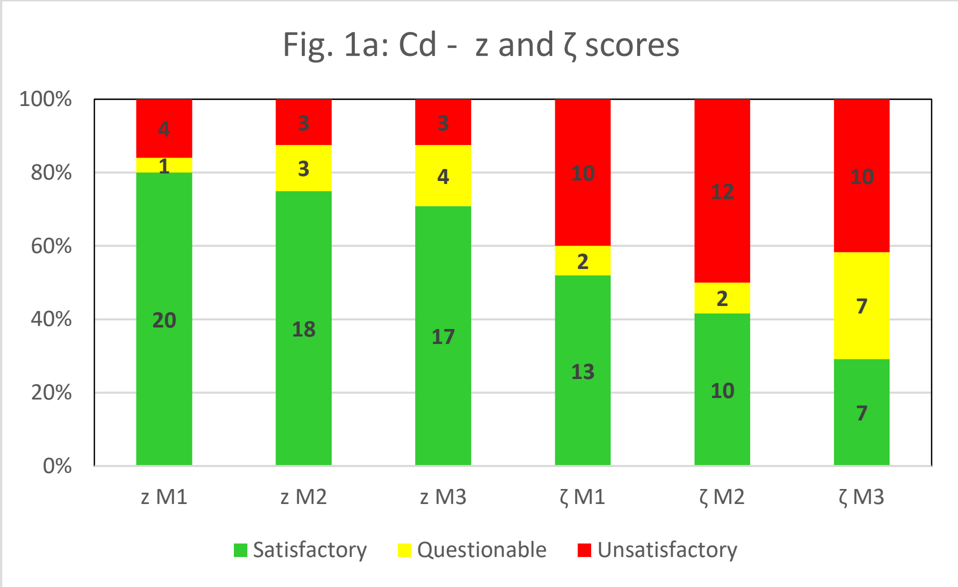
The majority of the participants (20 out of 24) is routinely reporting uncertainties to their customers for this type of analysis.

Most of the laboratories provided expanded measurement uncertainties and their associated coverage factors.

Thirteen laboratories (out of 24) reported realistic uncertainties for the six measurands investigated, while ten laboratories reported underestimated relative measurement uncertainties below 7 % (case "b"). L14 erroneously reported a relative measurement uncertainty of 15 % instead of a value in the requested units for measurement uncertainty (in mg kg^{-1}).

Similarly, L20 acknowledged having reported the confidence interval (97 %) instead of the requested coverage factor k. This did not influence L20's performance when expressed as a z score, but led to an unsatisfactory ζ score.

Table 2 summarises the approaches used for a measurement uncertainty evaluation by the different laboratories.



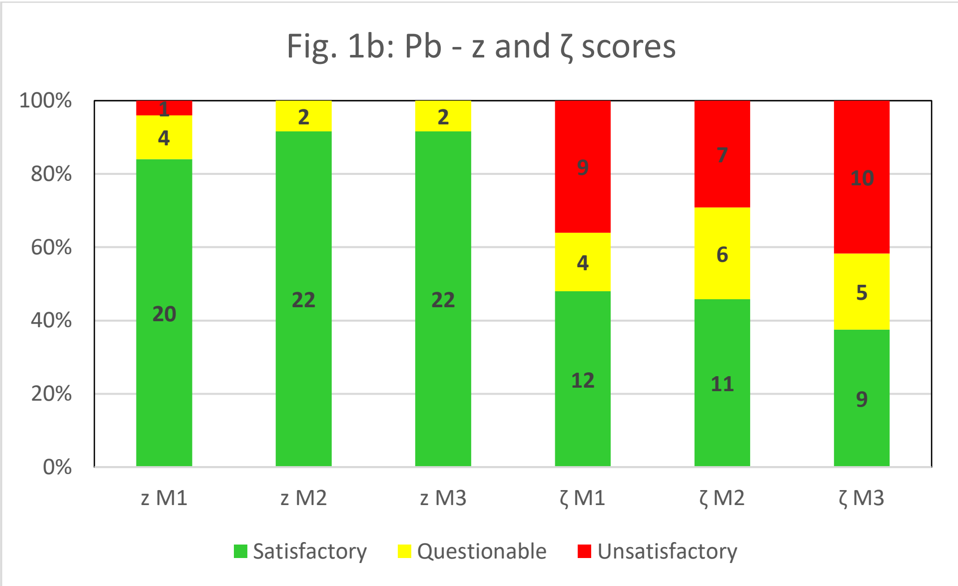


Figure 1: Overview of laboratory performance per measurand according to *z* and *ζ* scores, for (1a) Cd and (1b) Pb for the three migrations (M1, M2, M3). Corresponding number of laboratories included in the graph. Satisfactory, questionable and unsatisfactory performances indicated in green, yellow and red, respectively.

Table 2: Overview of the approaches used to estimate measurement uncertainty (multiple selections were possible).

Approach	N° of labs
According to ISO-GUM	5
From known uncertainty of a standard method	0
Derived from a single-laboratory validation study	11
Measurement of replicates (precision)	6
Estimation based on judgment	5
Derived from interlaboratory comparison data	0
From Horwitz model	0

6.3.3 Compliance statement

Participants were asked to report their compliance statement relative to the settings in Directive 84/500/EEC [1] where maximum limits are fixed at 0.3 mg L^{-1} and 4.0 mg L^{-1}, for Cd and Pb, respectively. Since all the assigned values (Table 1) are below the maximum limits set by the legislation, the investigated ceramic bowls shall be considered as compliant. This was further confirmed by all the participants having assessed the compliance of the investigated artefact (20 out of 24).

6.3.4 Additional information extracted from the questionnaire

Twenty-four participants have answered to the questionnaire giving valuable information on the laboratories, their way of working and their analytical methods. Annex 12 summarises the experimental details used by the laboratories. All participants stated to be accredited according to ISO/IEC 17025 [8] for this type of measurements.

Fourteen laboratories applied an accredited method for the determination of the mass fraction of Cd and Pb. Nine laboratories followed a validated method. Five laboratories followed a standard method of analysis.

The majority of participants (20 out of 24) have experience with the analysis of Cd and Pb released from ceramic kitchenware. All of them stated that they are participating in similar migration related proficiency testing schemes.

Annex 12 presents additional experimental details provided by the laboratories in the dedicated questionnaire. Most of the laboratories controlled the temperature of the food simulant solution during each migration experiment, using a calibrated thermometer or a calibrated data logger. Despite the fact that two laboratories did not preheat the ceramic bowls before each migration, their results led to satisfactory performances.

No instructions were given to the participants regarding the time interval between each migration. This time interval ranged from 10 to 95 minutes, without any noticeable influence on the participant's performance.

7 Conclusion

The proficiency testing round "FCM-20/02 (Part 1)" was organised to assess the analytical capabilities of EU NRLs and OCLs to determine the mass fractions of Cd and Pb having migrated from ceramic bowls under specified conditions.

All the laboratories filled in the questionnaire. 20 out of 24 laboratories made a correct conformity statement regarding the test item compliance.

Thirteen of them (out of 24) reported realistic measurement uncertainties for the six measurands investigated, while ten others reported underestimated relative measurement uncertainties below 7 % (case "b").

The overall performance of the participants for the determination of the mass fractions of Cd and Pb migrating from the ceramic bowls at prescribed conditions was satisfactory. This confirms the analytical capabilities of the NRLs to enforce Directive 84/500/EEC.

Acknowledgements

The laboratories listed hereafter are kindly acknowledged for their participation in this PT round.

Organisation	Country
AGES - Austrian Agency for Health & Food Safety	Austria
Sciensano	Belgium
National Center of Public Health and Analyses	Bulgaria
Croatian Institute of Public Health	Croatia
Ministry of Health, State General Laboratory	Cyprus
National Institute of Public Health	Czech Republic
The Danish Veterinary and Food Administration	Denmark
Health Board	Estonia
Finnish Customs Laboratory	Finland
Laboratoire National d'Essais	France
Service Commun des Laboratoires - Laboratoire de Bordeaux	France
German Federal Institute for Risk Assessment	Germany
General Chemical State Laboratory	Greece
National Food Chain Safety Office Food Chain Safety Laboratory Directorate	Hungary
Public Analyst's Laboratory	Ireland
Laboratoire National de Santé	Luxembourg
The Netherlands Food and Consumer Product Safety Authority (NVWA)	Netherlands
National Institute of Public Health - National Institute of Hygiene	Poland
Escola Superior de Biotecnologia - Universidade Católica Portuguesa	Portugal
Regional Public Health Authority	Slovakia
National Laboratory of Health, Environment and Food	Slovenia
CENTRO NACIONAL ALIMENTACION (CNA)-AESAN	Spain
Swedish Food Agency	Sweden
Lebensmittelkontrolle Solothurn	Switzerland
Fera Science Ltd.	United Kingdom

References

[1] Council Directive 84/500/EEC on the approximation of the laws of the Member States relating to ceramic articles intended to come into contact with foodstuffs, Off. J. Eur. Communities, L 277/12 (1984).

[2] Commission Regulation, (EU) No 2017/625 of The European Parliament and of The Council of 15 march 2017 on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products, Off. J. Eur. Communities. L 95/1 (2017).

[3] ISO/IEC 17043:2010 *"Conformity assessment – General requirements for proficiency testing"*, issued by ISO-Geneva (CH), International Organisation for Standardization.

[4] ISO 13528:2015 *"Statistical methods for use in proficiency testing by interlaboratory comparisons"*, issued by ISO-Geneva (CH), International Organisation for Standardization.

[5] ISO/IEC Guide 98-3:2008 "Guide to the expression of uncertainty in measurement", International Organisation for Standardization.

[6] S L R Ellison and A Williams (Eds). Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, Third edition, (2012) ISBN 978-0-948926-30-3. Available from www.eurachem.org.

[7] Analytical Methods Committee Technical Brief - *"Representing data distributions with kernel density estimates"*, AMC Tech. Br. 4 (2006). http://www.rsc.org/images/brief4_tcm18-25925.pdf.

Software for calculating kernel densities (Excell add-in) available from : https://www.rsc.org/membership-and-community/connect-with-others/through-interests/divisions/analytical/amc/software/

[8] ISO/IEC 17025:2017, "General requirements for the competence of testing and calibration laboratories", International Organisation for Standardization.

Annex 1: Invitation letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate F - Health, Consumers and Reference Materials (Geel)
Food and Feed Compliance

EURL
European Union Reference Laboratory
for Food Contact Materials

Geel, 20th October 2020
JRC.F.5/FCR/AS/Ares(2020)5671905

Subject: **Participation in FCM-20/02 – "Determination of the mass fractions of i) Cd and Pb migrated from ceramics (test item 1, T1) and of ii) As, Cd, Cr, Pb, Eu, La, Gd, Hg and Tb in food simulant B solution (test item 2, T2)"**

Dear FCM-20/02 PT Participant,

Thank you for participating in the FCM-20/02 proficiency test (PT). This PT is organised in support to possible revision of the ceramic Directive 84/500/EEC (test item 1, T1) and in the frame of the 15th amendment of Regulation (EU) 10/2011 (test item 2, T2).

The measurands are mass fractions (mg kg^{-1}) of Cd and Pb migrated from ceramic bowls (T1) into a 4 % v/v ethanoic acid solution and of As, Cd, Cr, Pb, Eu, La, Gd, Hg and Tb in food simulant B solution (T2).

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

The parcel you received contains,

- Four ceramic bowls (T1),
- A glass bottle with Teflon-lined caps (100 ml) containing food simulant B solution (3 % m/v ethanoic acid) spiked with the nine elements described above (T2).

Upon arrival of this parcel, please check whether the test items are undamaged after transport.

May I kindly ask you to send us or email the "**Confirmation of receipt**" form within 3 days after receipt of the test items.

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

- Prepare a food simulant solution (4 % v/v ethanoic acid, p.a. > 99 %),

For each ceramic bowl (T1, and for at least three ceramic bowls):

Retieseweg 111, B-2440 Geel - Belgium

European Commission, Via Enrico Fermi 2749, 21027 Ispra (Varese) - Italy. Telephone: +39 0332 78-9111
Office: 26 00/005 - Tel. direct line +39 332 78 5319

Fernando.CORDEIRO-RAPOSO@ec.europa.eu

Annex 2: Test item accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate F - Health, Consumers and Reference Materials (Geel)
Food and Feed Compliance

EURL
European Union Reference Laboratory
for Food Contact Materials

Geel, 20th October 2020
JRC.F.5/FCR/AS/Ares(2020)5671905

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Dear FCM-20/02 PT Participant,

Thank you for participating in the FCM-20/02 proficiency test (PT). This PT is organised in support to possible revision of the ceramic Directive 84/500/EEC (test item 1, T1) and in the frame of the 15th amendment of Regulation (EU) 10/2011 (test item 2, T2).

The measurands are mass fractions (mg kg^{-1}) of Cd and Pb migrated from ceramic bowls (T1) into a 4 % v/v ethanoic acid solution and of As, Cd, Cr, Pb, Eu, La, Gd, Hg and Tb in food simulant B solution (T2).

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

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Retieseweg 111, B-2440 Geel - Belgium

European Commission, Via Enrico Fermi 2749, 21027 Ispra (Varese) - Italy. Telephone: +39 0332 78-9111
Office: 26 00/005 - Tel. direct line +39 332 78 5319

Fernando.CORDEIRO-RAPOSO@ec.europa.eu

- Carry out a 1st migration (<u>at 22 ± 2 °C for 24 ± 0.5h</u>) on each ceramic bowl,
- Carry out a 2nd migration (<u>at 22 ± 2 °C for 24 ± 0.5h</u>) on each ceramic bowl,
- Carry out a 3rd migration (<u>at 22 ± 2 °C for 24 ± 0.5h</u>) on each ceramic bowl.

Use freshly prepared food simulant solution for each migration and follow the procedure as described in Council Directive 84/500/EEC Annex 1. Assume the density of the food simulant solution to be equal to the unity.

For T2:

- Perform **two or three independent replicates** on the determination of the mass fractions of As, Cd, Cr, Pb, Eu, La, Gd, Hg and Tb in food simulant solution (T2).

Please report separately for each test item, the following:

For T1:

- the **mean** of at least **three mass fractions of Cd and Pb after the 1st migration (in mg kg^{-1})**;
- the **mean** of at least **three mass fractions of Cd and Pb after the 2nd migration (in mg kg^{-1})**;
- the **mean** of at least **three mass fractions of Cd and Pb after the 3rd migration (in mg kg^{-1})**;
- the associated **expanded uncertainty for the 1st migration (in mg kg^{-1});**
- the **coverage factor;** and
- the analytical technique used,

The mean of the three mass fractions of Cd and Pb after the 1st migration shall be used for compliance assessment of test item T1 towards Council Directive 84/500/EEC.

For T2:

- The **mean** of the **mass fractions of As, Cd, Cr, Pb, Eu, La, Gd, Hg and Tb in food simulant B solution (T2, in mg kg^{-1})**,
- the associated **expanded uncertainty (in mg kg^{-1});**
- the **coverage factor;** and
- the analytical technique used.

The results shall be reported in the same format (e.g. number of significant figures) as you normally report to customers.

The deadline for submission of results is the **29th January 2021**.

Retieseweg 111, B-2440 Geel - Belgium

European Commission, Via Enrico Fermi 2749, 21027 Ispra (Varese) - Italy. Telephone: +39 0332 78-9111
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The procedures used for the organisation of PTs are accredited according to ISO/IEC 17043:2010 and guarantee that the identity of the participants and the information provided by them is treated as confidential. However, lab codes of National Reference Laboratories appointed in line with Regulation (EU) 2017/625 will be disclosed to DG SANTE upon request for (long-term) performance assessment.

Remember 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.

Please be aware of the existence of an appeal procedure in case you disagree with your performance scores.

Your participation in this project is greatly appreciated.

Do not hesitate to contact us for further information.

With kind regards,

/signed electronically in Ares/

Dr. Fernando Cordeiro / Dr. James Snell
FCM-20/02 PT Coordinator / Deputy

Cc: H. Emons (Head of Unit, Food & Feed Compliance, F.5),
E. Hoekstra (Operating Manager EURL-FCM)

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Annex 3: Confirmation of receipt form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate F - Health, Consumers and Reference Materials (Geel)
Food and Feed Compliance

EURL
European Union Reference Laboratory
for Food Contact Materials

Geel, 20th October 2020
JRC.F.5/FCR/AS/Ares(2020)5671905

Subject: "Confirmation receipt" form - FCM-20/02 – "Determination of the mass fractions of i) Cd and Pb migrated from ceramics (test item 1) and of ii) As, Cd, Cr, Pb, Eu, La, Gd, Hg and Tb in food simulant B solution (test item 2)"

Please return this form at your earliest convenience, to confirm that the package arrived well to your laboratory. If samples are damaged, please mention it below and contact us as soon as possible.

Lab Code (see email):

____

Name of your organisation:

____

Date of package arrival:

____

Were the samples damaged? ☐ YES ☐ NO

Remarks:

____

____

Signature:

Thank you for returning this form by email to:

Dr. F. Cordeiro/ S. García-Ruiz
FCM-20/02 Coordinator/Deputy
e-mail : jrc-eurl-fcm@ec.europa.eu

Retieseweg 111, B-2440 Geel - Belgium

European Commission, Via Enrico Fermi 2749, 21027 Ispra (Varese) - Italy. Telephone: +39 0332 78-9111
Office: 26 00/005 - Tel. direct line +39 332 78 5319

Fernando.CORDEIRO-RAPOSO@ec.europa.eu

Annex 4: Questionnaire

Milc questionnaire

Comparison for EURL-FCM-20/02

This questionnaire is meant to collect additional information about your laboratory and experimental details about your analytical method. Your answers will be used in the evaluation of the proficiency test FCM-20/02. Please enter the information related to the method used for the determination of the mass fractions of the selected analytes. Please do so comprehensively, in order to allow appropriate evaluation and relevant discussion of the results.

Submission Form

1. **Are you a National Reference Laboratory?**

☐ a) Yes

☐ b) No

2. **Compliance statement**

See table **Compliance to the relevant European legislation** at bottom

2.1. If "Not in compliance" please specify why.

3. **Does your laboratory have a quality management system?**

☐ a) Yes

☐ b) No

3.1. If "Yes" based on which standard?

☐ ISO 17025

☐ ISO 9001

☐ Other

4. **What was the basis for your measurement uncertainty evaluation?**

- ☐ a) Uncertainty budget (ISO GUM)
- ☐ b) Known uncertainty of standard method (ISO 21748)
- ☐ c) In-house method validation
- ☐ d) Measurement of replicates (precision)
- ☐ e) From expert judgment

5. **Do you provide an uncertainty statement to your customers?**

- ☐ a) Yes
- ☐ b) No

6. **Provide your experience in the analysis of elements migrated from FCM**

See table **Experience (samples per year)** at bottom

7. **Test item 1 (Cd & Pb migrated from ceramics)**

- ☐ a) Standard method
- ☐ b) Validated method
- ☐ c) Accredited method

8. **What was the volume (in mL) of food simulant solution used for the migration experiment?**

9. **Please provide the temperature (in C) of the oven used for the migration experiment.**

- Page 2 of 6 -

9.1. Did you control the food simulant solution temperature during migration?

☐ a) Yes

☐ b) No

9.1.1. If yes, please specify how you controlled the temperature.

○ a) Calibrated termometer

○ b) Calibrated datalogger

○ c) Non calibrated device

○ d) Other

10. **Did you preheat the ceramic bowls before the migration?**

☐ a) Yes

☐ b) No

10.1. If "Yes" please provide the exact temperature and time

10.2. How long was the time interval (minutes) between finishing the first migration step and starting the second?

10.3. Were the bowls washed between successive migrations, and if so, with what?

11. **Test item 2 (trace elments in food simulant B)**

- ☐ a) Standard method
- ☐ b) Validated method
- ☐ c) Accredited method

12. **Do you participate in PT scheme for this type of analysis?**

- ☐ a) Cd & Pb in ceramics
- ☐ b) Trace elements in plastic

13. **Do you have any additional comment. Let us know!**

Compliance to the relevant European legislation

Directive 84/500/EEC for T1 (ceramics) / Regulation (EU) 10/2011 for T2 (plastic)

Questions/Response table	*In compliance*	*Not in compliance*
Cd & Pb in ceramic		
Trace elments in plastic		

Experience (samples per year)

Indicate your experience with "X"

Questions/Response table	*None*	*1-50*	*51-250*	*251-1000*
Cd & Pb in ceramics				
Trace elements in plastic				

Annex 5: Homogeneity study (all values in mg L^{-1})

	1st migration		2nd migration		3rd migration	
Bowl	Cd	Pb	Cd	Pb	Cd	Pb
1	0.0598		0.04244	0.397	0.02464	0.2360
2	0.0528	0.433	0.04429	0.342	0.02478	0.1921
3	0.0491	0.460	0.04090	0.368	0.02230	0.2060
4	0.0464	0.436	0.04129	0.357	0.02280	0.2120
5	0.0425	0.412	0.03762	0.337	0.02136	0.1988
6	0.0545	0.540	0.04505	0.428	0.02731	0.2490
7	0.0496	0.470	0.04250	0.389	0.02374	0.2270
8	0.0551	0.528	0.04070	0.383	0.02296	0.2150
9	0.0491	0.464	0.04050	0.371	0.02610	0.2210
10	0.0488	0.456	0.04170	0.378	0.02430	0.2210
mean	0.0508	0.467	0.0417	0.375	0.0240	0.218
s_{bb}	0.0049	0.042	0.0021	0.027	0.0018	0.017
s_r	0.0020	0.028	0.0017	0.023	0.0010	0.013
u_{hom}	0.0045	0.032	0.0012	0.015	0.0015	0.011
σ_{pt} (30 %)	0.0152	0.140	0.0125	0.113	0.0072	0.065
0.3 σ_{pt}	0.0046	0.042	0.0038	0.034	0.0022	0.020
u_{hom} < 0.3 σ_{pt}	passed	passed	passed	passed	passed	passed

Where: s_{bb} is the between-bowl standard deviation,
s_r is the analytical standard deviation under repeatability conditions,
u_{hom} is the standard deviation due to inhomogeneity,
σ_{pt} is the standard deviation for performance assessment.

Annex 6: Results for Cd after the 1st migration

Assigned range (in mg kg^{-1}): $x_{pt} \pm U$ = 0.0508 ± 0.0090 (k = 2) and σ_{pt} = 0.0152

Lab	x_i	$U(x_i)$	k [a]	Technique	$u(x_i)$	z score [b]	ζ score [b]	MU [c]
L01	0.0763	0.011	2	ICP-MS	0.006	1.68	3.60	b
L02	0.059	0.018	2	ICP-MS	0.009	0.54	0.82	a
L04	0.079	0.0039	1	ICP-MS	0.004	1.85	4.75	b
L05	0.0514	0.0098	1.73	AAS	0.006	0.04	0.09	a
L06	0.099	0.022	2	ICP-MS	0.011	3.17	4.06	a
L07	0.0457	0.0079	2	ICP-MS	0.004	-0.33	-0.85	b
L08	0.11	0.01	2	ICP-OES	0.005	3.89	8.81	b
L09	0.065	0.02	2	AAS	0.010	0.93	1.30	a
L10	0.047	0.005	2	ICP-MS	0.003	-0.25	-0.73	b
L11	0.0624	0.01001	2	ICP-MS	0.005	0.76	1.73	b
L12	0.103	0.019	2	EAA-GF	0.010	3.43	4.97	a
L13	0.026	0.004	2	AAS	0.002	-1.63	-5.04	b
L14	0.0629	15*	2	ICP-MS	7.500	0.80	0.00	c
L15	0.003	0.002	1	ICP-MS	0.002	-3.14	-9.71	c
L16	0.046	0.005	3.18	ICP-MS	0.002	-0.31	-1.00	b
L17	0.0705	0.0045	2	GF-AAS	0.002	1.30	3.93	b
L18	0.049	0.015	2	ICP-OES	0.008	-0.12	-0.20	a
L19	0.027	0.006	2	ICP-OES	0.003	-1.56	-4.40	a
L20	0.0828	0.034	97.6**	ICP-MS	0.000	2.10	7.11	b
L21	0.05265	0.000863	2	ICP-OES	0.000	0.12	0.42	b
L22	0.0645	0.0071	2	ICP-MS	0.004	0.90	2.40	b
L23	0.05	0.005	2	FAAS	0.003	-0.05	-0.15	b
L24	0.070	0.014	2	ICP-MS	0.007	1.26	2.31	a
L25	0.05045	0.00383	2	ICP-MS	0.002	-0.02	-0.06	b
L26	0.060***	0.016	2	ICP-MS	0.013	0.61	1.01	a

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

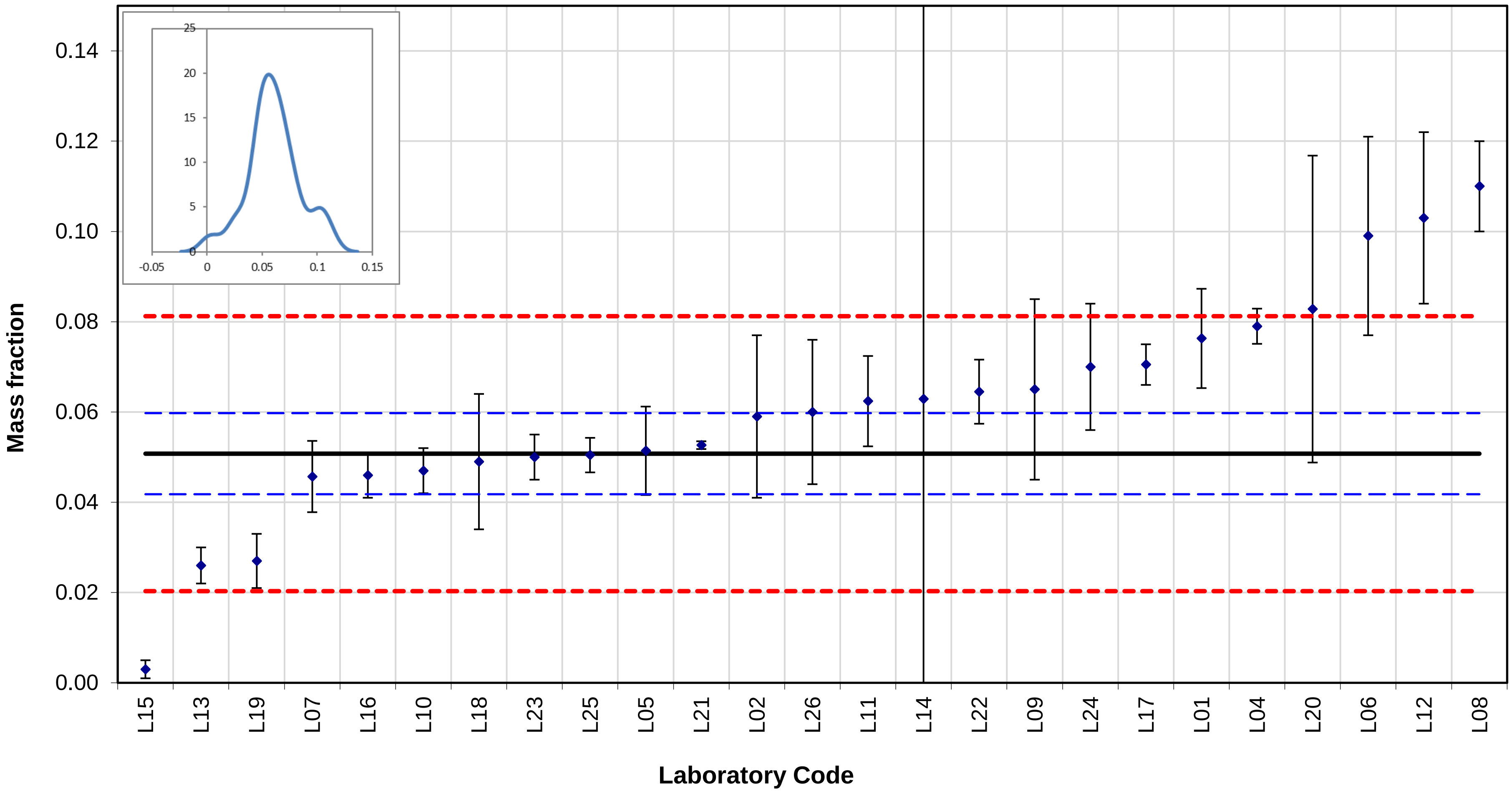
[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt,rel}$; b: $u_{rel}(x_i) < u_{rel}(x_{pt})$ and c: $u_{rel}(x_i) > \sigma_{pt,rel}$

* Measurement uncertainty reported in %, instead of mg kg^{-1}.

** Percent confidence interval reported instead of the coverage factor.

*** The laboratory inverted the results of the 1st and 2nd migrations.



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 7: Results for Cd after the 2nd migration

Assigned range (in mg kg^{-1}) : $x_{pt} \pm U$ = 0.0417 ± 0.0026 (k = 2) and σ_{pt} = 0.0125

Lab	x_i	$U(x_i)$	k [a]	Technique	$u(x_i)$	z score [b]	ζ score [b]	MU [c]
L01	0.0625	0.0094	2	ICP-MS	0.005	1.66	4.26	a
L02	0.047	0.014	2	ICP-MS	0.007	0.42	0.74	a
L04	0.047		1	ICP-MS	0.000	0.42	4.04	b
L05	0.0472	0.0090	1.73	AAS	0.005	0.44	1.03	a
L06	0.083	0.016	2	ICP-MS	0.008	3.30	5.09	a
L07	0.0430	0.0074	2	ICP-MS	0.004	0.10	0.33	a
L08	0.10	0.02	2	ICP-MS	0.010	4.66	5.78	a
L09	0.060	0.01	2	AAS	0.005	1.46	3.54	a
L10	0.043	0.004	2	ICP-MS	0.002	0.10	0.54	a
L11	0.0383	0.00597	2	ICP-MS	0.003	-0.27	-1.04	a
L12	0.073	0.006	2	EAA-GF	0.003	2.50	9.56	a
L13								
L14	0.0637	15*	2	ICP-MS	7.500	1.76	0.00	c
L15	0.007	0.004	1	ICP-MS	0.004	-2.77	-8.24	c
L16	0.041	0.005	3.18	ICP-MS	0.002	- 0.06	- 0.34	a
L17	0.0523	0.0034	2	GF-AAS	0.002	0.85	4.93	a
L18	0.043	0.013	2	ICP-OES	0.007	0.10	0.20	a
L19	0.020			ICP-OES	0.000	-1.73	-16.52	b
L20	0.0575	0.0054	97.6**	ICP-MS	0.000	1.26	12.02	b
L21	0.04164	0.000863	2	ICP-OES	0.000	0.00	-0.04	b
L22	0.0498	0.0055	2	ICP-MS	0.003	0.65	2.66	a
L23	0.04	0.01	2	FAAS	0.005	-0.14	-0.33	a
L24	0.068	0.018	2	ICP-MS	0.009	2.10	2.89	a
L25	0.04891	0.00372	2	ICP-MS	0.002	0.58	3.17	a
L26	0.099***	0.026	2	ICP-MS	0.013	4.58	4.39	a

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

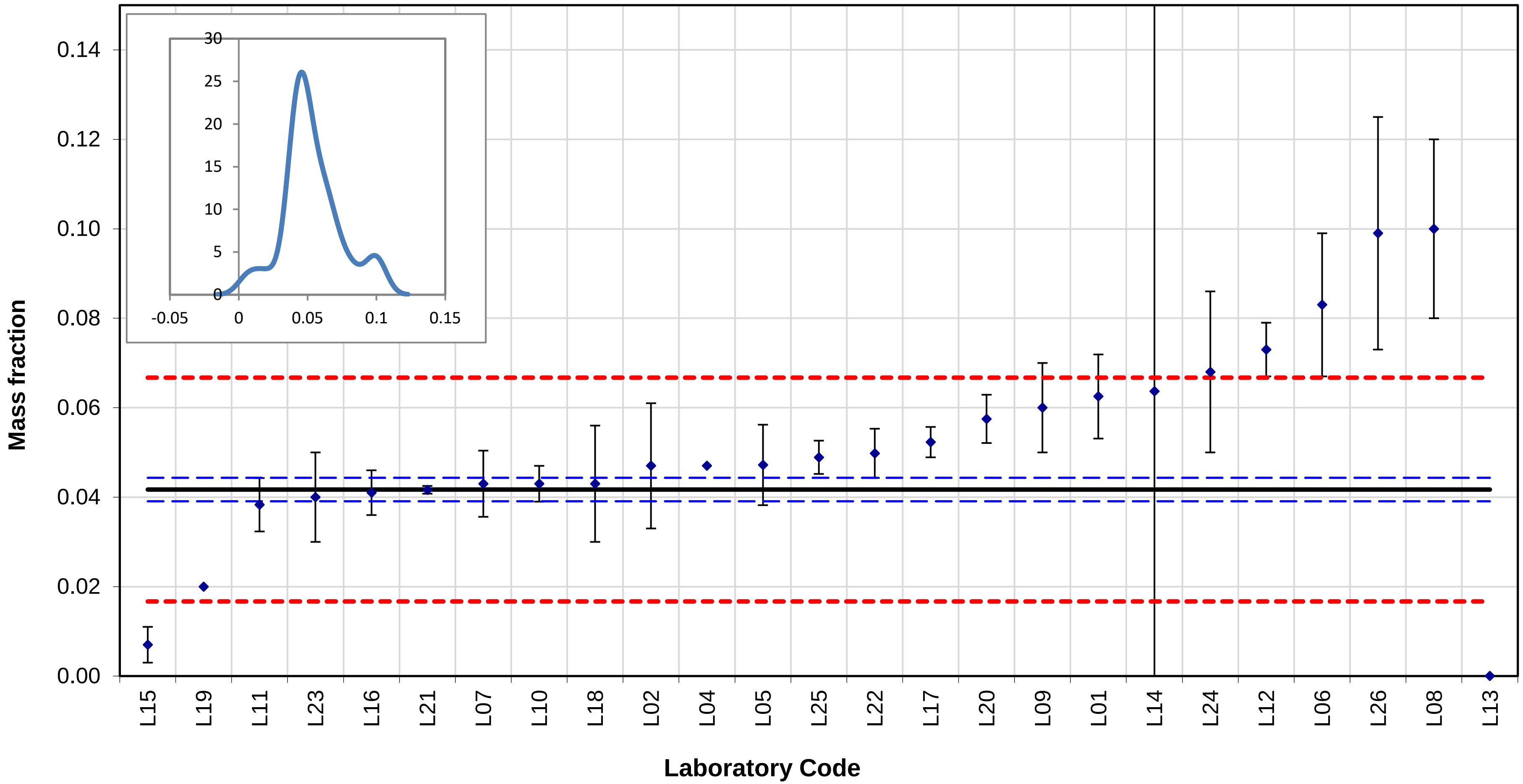
[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt,rel}$; b: $u_{rel}(x_i) < u_{rel}(x_{pt})$ and c: $u_{rel}(x_i) > \sigma_{pt,rel}$

* Measurement uncertainty reported in %, instead of mg kg^{-1}.

** Percent confidence interval reported instead of the coverage factor.

*** The laboratory inverted the results of the 1st and 2nd migrations.



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 8: Results for Cd after the 3rd migration

Assigned range (in mg kg^{-1}): $x_{pt} \pm U$ = 0.0240 ± 0.0030 (k = 2) and σ_{pt} = 0.0072

Lab	x_i	$U(x_i)$	k [a]	Technique	$u(x_i)$	z score [b]	ζ score [b]	MU [c]
L01	0.0375	0.0056	2	ICP-MS	0.003	1.87	4.22	a
L02	0.031	0.009	2	ICP-MS	0.005	0.97	1.47	a
L04	0.028		1	ICP-MS	0.000	0.55	2.59	b
L05	0.0231	0.0044	1.73	AAS	0.003	-0.13	-0.31	a
L06	0.052	0.014	2	ICP-MS	0.007	3.88	3.90	a
L07	0.0320	0.0055	2	ICP-MS	0.003	1.11	2.53	a
L08	0.07	0.01	2	ICP-MS	0.005	6.38	8.79	a
L09	0.038	0.01	2	AAS	0.005	1.94	2.67	a
L10	0.031	0.003	2	ICP-MS	0.002	0.97	3.25	b
L11	0.0258	0.00265	2	ICP-MS	0.001	0.25	0.87	b
L12	0.043	0.004	2	EAA-GF	0.002	2.63	7.53	b
L13								
L14	0.0419	15*	2	ICP-MS	7.500	2.48	0.00	c
L15	0.008	0.001	1	ICP-MS	0.001	-2.22	-8.76	a
L16	0.031	0.002	3.18	ICP-MS	0.001	0.97	4.21	b
L17	0.0273	0.0017	2	GF-AAS	0.001	0.45	1.87	b
L18	0.035	0.011	2	ICP-OES	0.006	1.52	1.92	a
L19	0.020			ICP-OES	0.000	-0.56	-2.63	b
L20	0.0392	0.00286	97.6**	ICP-MS	0.000	2.10	9.91	b
L21	0.02795	0.000863	2	ICP-OES	0.000	0.54	2.46	b
L22	0.0360	0.0040	2	ICP-MS	0.002	1.66	4.75	b
L23	0.02	0.005	2	FAAS	0.003	-0.56	-1.37	a
L24	0.048	0.015	2	ICP-MS	0.008	3.33	3.13	a
L25	0.02916	0.00222	2	ICP-MS	0.001	0.71	2.71	b
L26	0.034	0.009	2	ICP-MS	0.005	1.38	2.10	a

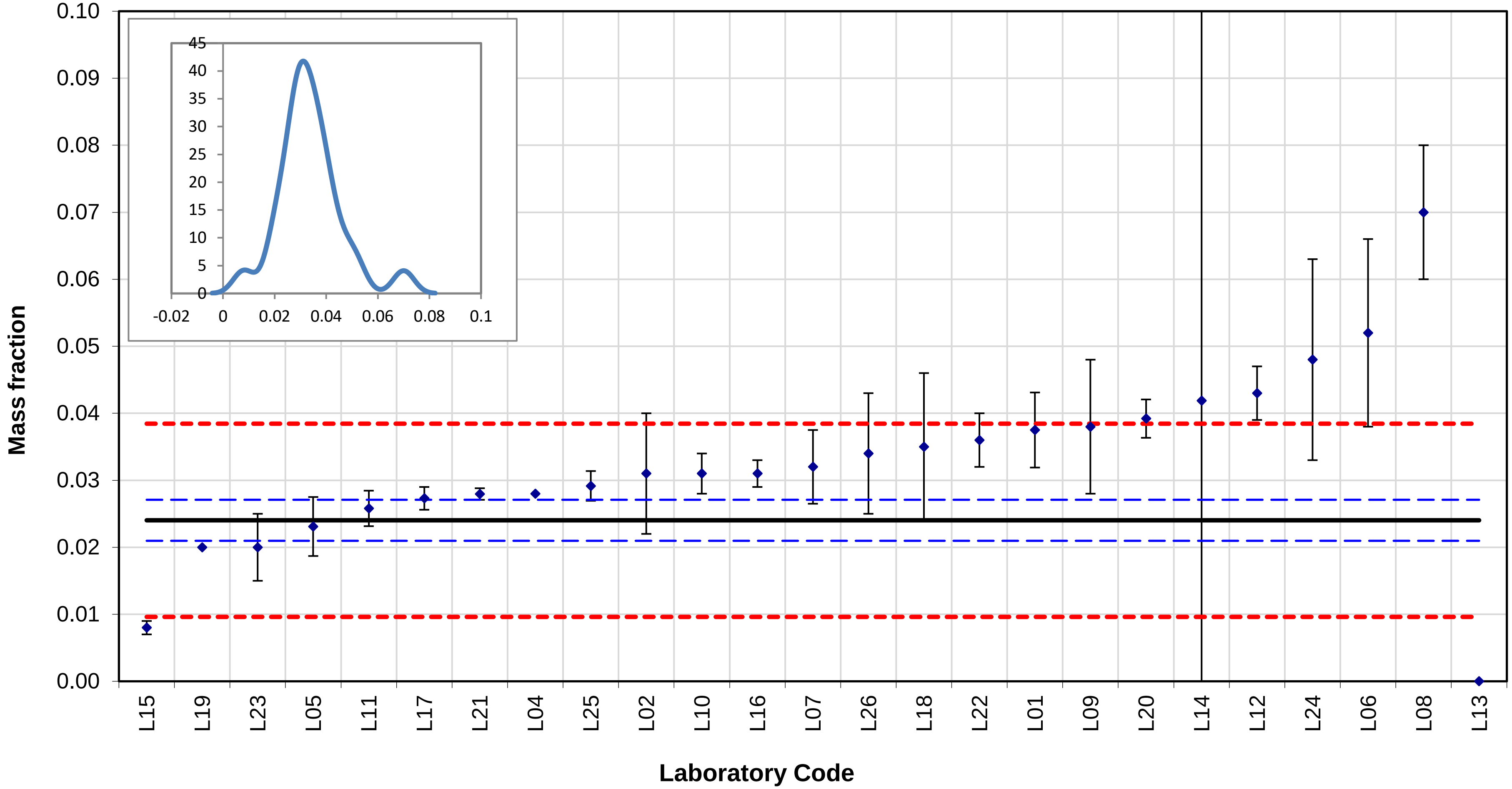
[a] √3 is set by the PT coordinator when no coverage factor k is reported. The reported uncertainty was assumed to have a rectangular distribution with k = √3,

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt,rel}$; b: $u_{rel}(x_i) < u_{rel}(x_{pt})$ and c: $u_{rel}(x_i) > \sigma_{pt,rel}$

* Measurement uncertainty reported in %, instead of mg kg^{-1}.

** Percent confidence interval reported instead of the coverage factor.



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 9: Results for Pb after the 1st migration

Assigned range (in mg kg^{-1}): $x_{pt} \pm U$ = 0.467 ± 0.068 (k = 2) and σ_{pt} = 0.140

Lab	x_i	$U(x_i)$	k [a]	Technique	$u(x_i)$	z score [b]	ζ score [b]	MU [c]
L01	0.592	0.089	2	ICP-MS	0.045	0.90	2.25	a
L02	0.506	0.152	2	ICP-MS	0.076	0.28	0.47	a
L04	0.737	0.046	1	ICP-MS	0.046	1.93	4.74	b
L05	0.466	0.056	1.73	AAS	0.032	0.00	-0.01	b
L06	0.78	0.33	2	ICP-MS	0.165	2.24	1.86	a
L07	0.3254	0.0562	2	ICP-MS	0.028	-1.01	-3.21	a
L08	0.74	0.04	2	ICP-MS	0.020	1.95	6.97	b
L09	0.42	0.04	2	AAS	0.020	-0.33	-1.19	b
L10	0.43	0.04	2	ICP-MS	0.020	-0.26	-0.93	b
L11	0.438	0.09309	2	ICP-MS	0.047	-0.20	-0.50	a
L12	0.865	0.069	2	EAA-GF	0.035	2.85	8.26	b
L13	0.29	0.04	2	AAS	0.020	-1.26	-4.50	b
L14	0.5448	13*	2	ICP-MS	6.500	0.56	0.01	c
L15	0.171	0.046	1	ICP-MS	0.046	-2.11	-5.18	a
L16	0.408	0.065	3.18	ICP-MS	0.020	-0.42	-1.48	b
L17	0.569	0.042	2	FAAS	0.021	0.73	2.58	b
L18	0.415	0.104	2	ICP-OES	0.052	-0.37	-0.83	a
L19	0.18	0.04	2	ICP-OES	0.020	-2.05	-7.31	a
L20	0.744	0.503	97.6**	ICP-MS	0.005	1.98	8.13	b
L21	0.55643	0.006234	2	ICP-OES	0.003	0.64	2.65	b
L22	0.574	0.074	2	ICP-MS	0.037	0.77	2.15	b
L23	0.42	0.06	2	FAAS	0.030	-0.33	-1.03	b
L24	0.60	0.15	2	ICP-MS	0.075	0.95	1.62	a
L25	0.4384	0.0324	2	ICP-MS	0.016	-0.20	-0.75	b
L26	1.014	0.241	2	ICP-MS	0.121	3.91	4.37	a

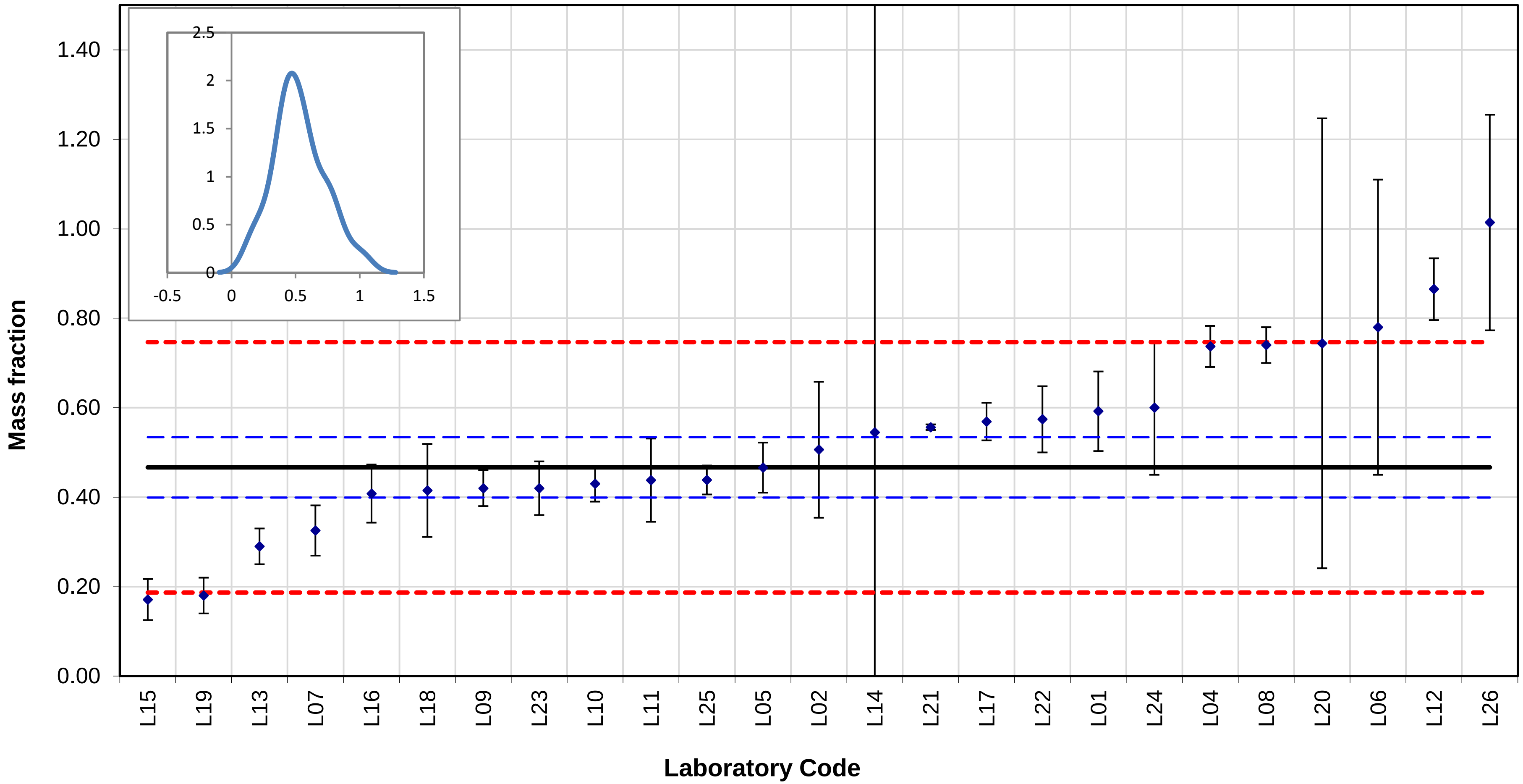
[a] √3 is set by the PT coordinator when no coverage factor k is reported. The reported uncertainty was assumed to have a rectangular distribution with k = √3,

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt,rel}$; b: $u_{rel}(x_i) < u_{rel}(x_{pt})$ and c: $u_{rel}(x_i) > \sigma_{pt,rel}$

* Measurement uncertainty reported in %, instead of mg kg^{-1}.

** Percent confidence interval reported instead of the coverage factor.



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 10: Results for Pb after the 2nd migration

Assigned range (in mg kg^{-1}): $x_{pt} \pm U = 0.375 \pm 0.034$ (k = 2) and $\sigma_{pt} = 0.113$

Lab	x_i	$U(x_i)$	k [a]	Technique	$u(x_i)$	z score [b]	ζ score [b]	MU [c]
L01	0.399	0.060	2	ICP-MS	0.030	0.21	0.69	a
L02	0.385	0.115	2	ICP-MS	0.058	0.09	0.17	a
L04	0.422		1	ICP-MS	0.000	0.42	2.74	b
L05	0.360	0.054	1.73	AAS	0.031	-0.13	-0.42	a
L06	0.59	0.14	2	ICP-MS	0.070	1.91	2.98	a
L07	0.2553	0.0441	2	ICP-MS	0.022	-1.06	-4.28	a
L08	0.55	0.04	2	ICP-MS	0.020	1.56	6.64	b
L09	0.38	0.05	2	AAS	0.025	0.04	0.16	a
L10	0.36	0.04	2	ICP-MS	0.020	-0.13	-0.57	a
L11	0.330	0.05116	2	ICP-MS	0.026	-0.40	-1.46	a
L12	0.566	0.037	2	EAA-GF	0.019	1.70	7.57	b
L13								
L14	0.5340	13*	2	ICP-MS	6.500	1.41	0.02	c
L15	0.121	0.012	1	ICP-MS	0.012	-2.26	-12.12	a
L16	0.356	0.057	3.18	ICP-MS	0.018	-0.17	-0.77	a
L17	0.397	0.043	2	FAAS	0.022	0.20	0.80	a
L18	0.373	0.093	2	ICP-OES	0.047	-0.02	-0.04	a
L19	0.13			ICP-OES	0.000	-2.18	-14.27	b
L20	0.466	0.194	97.6**	ICP-MS	0.002	0.81	5.26	b
L21	0.41485	0.006234	2	ICP-OES	0.003	0.35	2.28	b
L22	0.433	0.056	2	ICP-MS	0.028	0.52	1.77	a
L23	0.32	0.04	2	FAAS	0.020	-0.49	-2.09	a
L24	0.57	0.12	2	ICP-MS	0.060	1.73	3.12	a
L25	0.3147	0.0233	2	ICP-MS	0.012	-0.54	-2.91	b
L26	0.592	0.141	2	ICP-MS	0.071	1.93	2.99	a

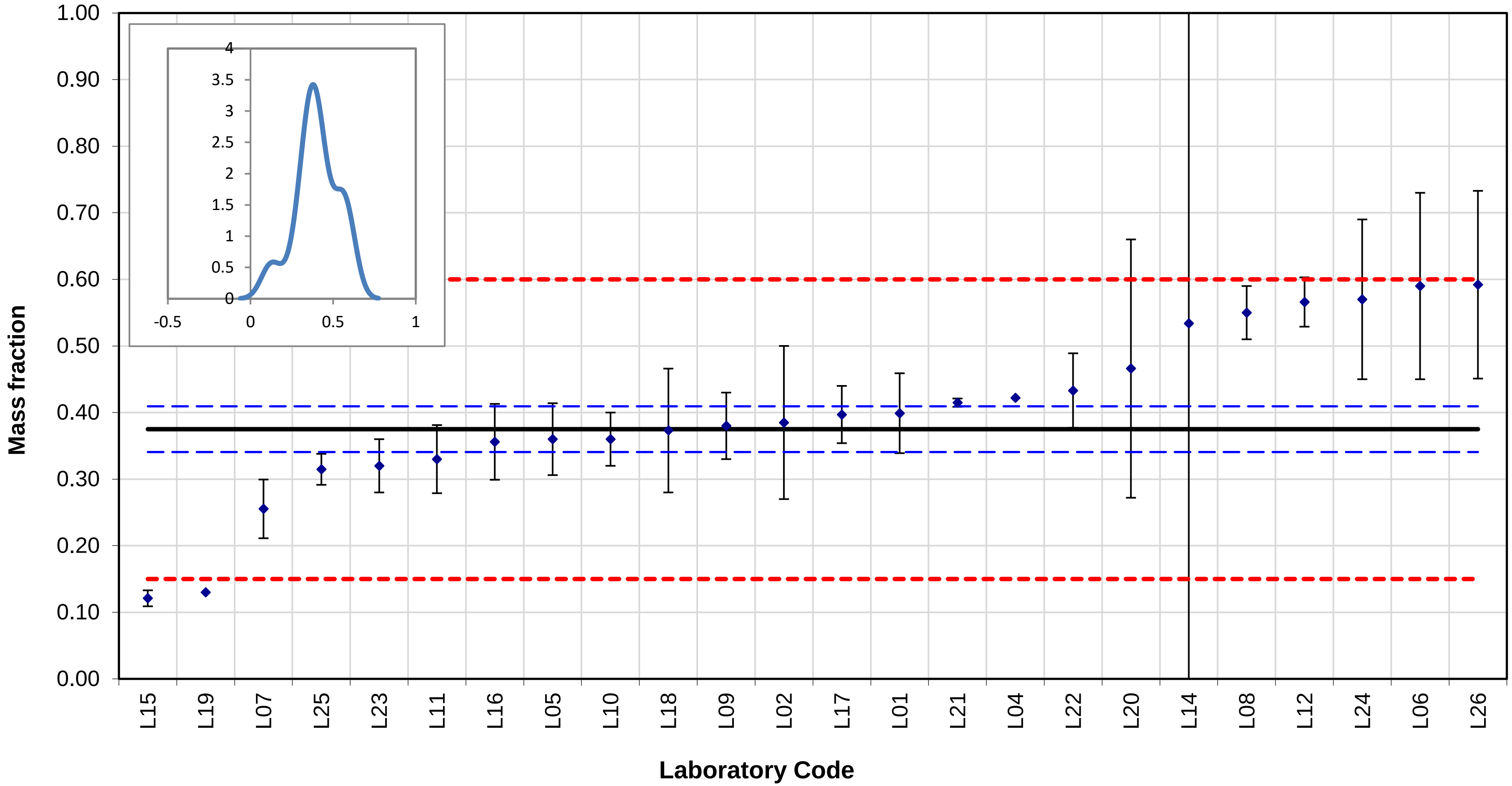
[a] √3 is set by the PT coordinator when no coverage factor k is reported. The reported uncertainty was assumed to have a rectangular distribution with $k = \sqrt{3}$,

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt,rel}$; b: $u_{rel}(x_i) < u_{rel}(x_{pt})$ and c: $u_{rel}(x_i) > \sigma_{pt,rel}$

* Measurement uncertainty reported in %, instead of mg kg^{-1}.

** Percent confidence interval reported instead of the coverage factor.



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 11: Results for Pb after the 3rd migration

Assigned range (in mg kg^{-1}): $x_{pt} \pm U$ = 0.218 ± 0.024 (k = 2) and σ_{pt} = 0.065

Lab	x_i	$U(x_i)$	k [a]	Technique	$u(x_i)$	z score [b]	ζ score [b]	MU [c]
L01	0.210	0.032	2	ICP-MS	0.016	-0.12	-0.40	a
L02	0.261	0.078	2	ICP-MS	0.039	0.66	1.05	a
L04	0.247		1	ICP-MS	0.000	0.44	2.42	b
L05	0.195	0.029	1.73	AAS	0.017	-0.35	-1.12	a
L06	0.32	0.09	2	ICP-MS	0.045	1.56	2.19	a
L07	0.1676	0.0290	2	ICP-MS	0.015	-0.77	-2.68	a
L08	0.36	0.05	2	ICP-MS	0.025	2.17	5.12	a
L09	0.23	0.04	2	AAS	0.020	0.18	0.51	a
L10	0.26	0.03	2	ICP-MS	0.015	0.64	2.19	a
L11	0.224	0.02579	2	ICP-MS	0.013	0.09	0.34	a
L12	0.335	0.026	2	EAA-GF	0.013	1.79	6.62	b
L13								
L14	0.3254	13*	2	ICP-MS	6.500	1.64	0.02	c
L15	0.107	0.007	1	ICP-MS	0.007	-1.70	-7.99	a
L16	0.271	0.039	3.18	ICP-MS	0.012	0.81	3.09	b
L17	0.227	0.016	2	FAAS	0.008	0.14	0.62	b
L18	0.309	0.077	2	ICP-OES	0.039	1.39	2.26	a
L19	0.13			ICP-OES	0.000	-1.35	-7.34	b
L20	0.284	0.139	97.6**	ICP-MS	0.001	1.01	5.47	b
L21	0.26618	0.006234	2	ICP-OES	0.003	0.74	3.89	b
L22	0.309	0.040	2	ICP-MS	0.020	1.39	3.90	a
L23	0.19	0.03	2	FAAS	0.015	-0.43	-1.46	a
L24	0.36	0.08	2	ICP-MS	0.040	2.17	3.40	a
L25	0.2217	0.0164	2	ICP-MS	0.008	0.06	0.25	b
L26	0.348	0.083	2	ICP-MS	0.042	1.99	3.01	a

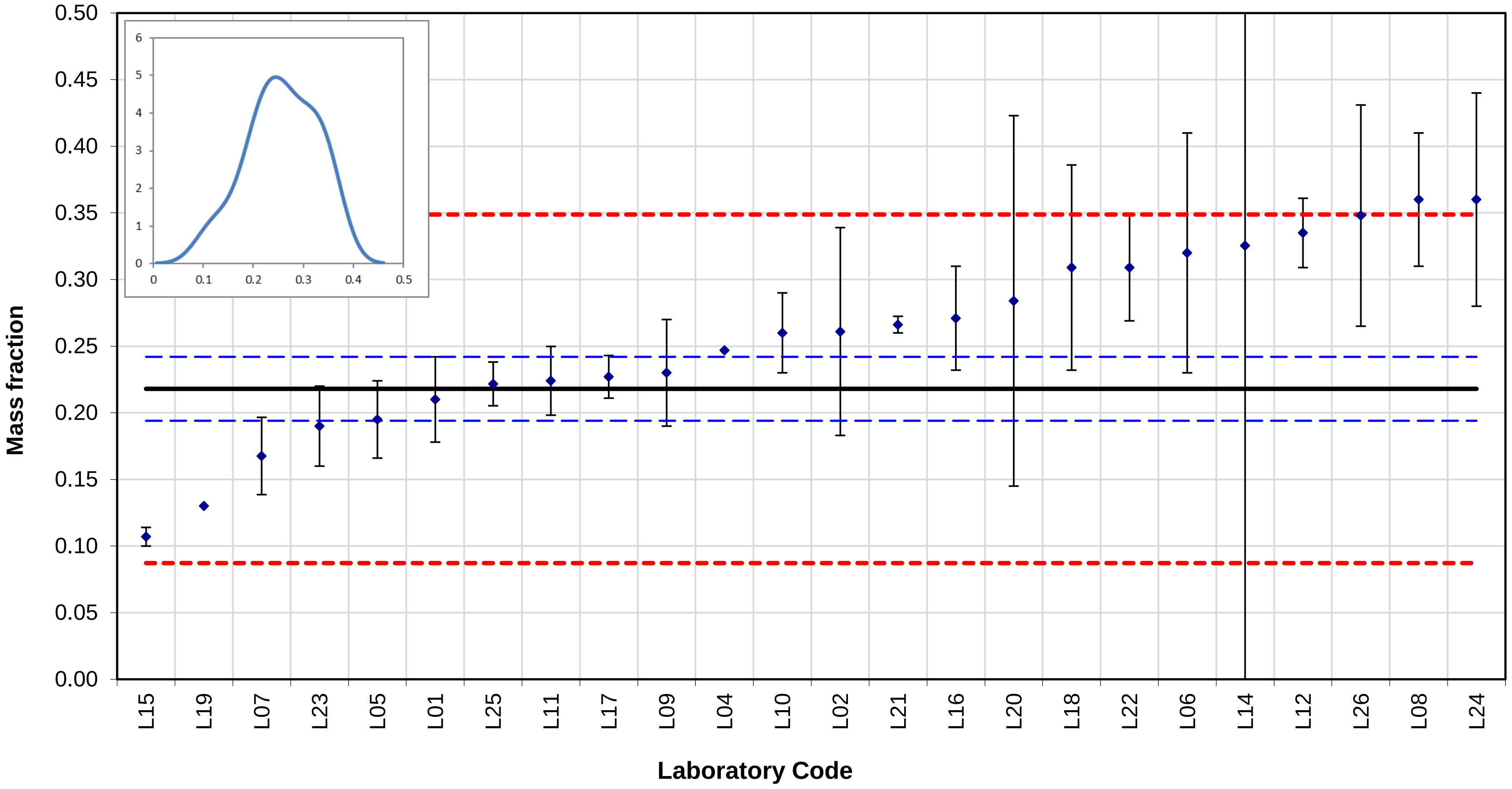
[a] √3 is set by the PT coordinator when no coverage factor k is reported. The reported uncertainty was assumed to have a rectangular distribution with k = √3,

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt,rel}$; b: $u_{rel}(x_i) < u_{rel}(x_{pt})$ and c: $u_{rel}(x_i) > \sigma_{pt,rel}$

* Measurement uncertainty reported in %, instead of mg kg^{-1}.

** Percent confidence interval reported instead of the coverage factor.



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\,\sigma_{pt}$): dotted red lines.

Annex 12: Information extracted from the questionnaire

Lab Code	Cd M1	Cd M2	Pb M1	Pb M2	Did you follow an accredited, validated or standard method?	Volume of food simulant solution used for each migration?	Temperature of the oven during the migration (°C)	Did you control the food simulant solution temperature during migration?		Did you preheat the ceramic bowls before the migration?	How long was the time interval (minutes) between finishing the first migration step and starting the second?	Were the bowls washed between successive migrations, and if so, with what?	PT participation?
L01	S	S	S	S	Accredited method	160 mL	22 ± 2	Yes	Calibrated termometer	Yes	Max. 10 minutes	Rinse with distilled water	Yes
L02	S	S	S	S	Validated method	160	22	Yes	Calibrated datalogger	Yes	30	Yes (Milli-Q water)	Yes
L04	S	S	S	S	Accredited method	Not determined, is not important	22	Yes	Calibrated termometer	No			Yes
L05	S	S	S	S	Accredited method	150	22 ± 2	No		No	max 1 hour	Yes before 1st migration test, not between 2nd and 3rd migration tests	Yes
L06	U	U	Q	S	Validated method	120	22 ± 2	Yes	Calibrated datalogger	No	40	No	Yes
L07	S	S	S	S	Standard method	180ml for each bowl	22 ± 2°C	Yes	Calibrated datalogger	No	15 minutes	Bowls were washed between successive migrations with distilled water and dried.	Yes
L08	U	U	S	S	Standard method	150	22	No		No			Yes
L09	S	S	S	S	Standard, Validated, Accredited method	150 ml	Oven wasn't used. Migration at room temperature 21-21.5 C. Room temperature was	No		No	It was approximately 10 minutes.	Yes. Before the 1st migration bowls were gently washed with detergent and rinsed with distilled water. After 1st and 2nd migration bowls were only rinsed with distilled water.	Yes
L10	S	S	S	S	Standard method	182 ml/bowl	around 22	No		No	15 min.	Yes, with Milli-Q water	Yes
L11	S	S	S	S	Accredited method	155 ml		No		No	About 45 minutes.	The bowls washed with deionized water between successive migrations.	Yes
L12	U	Q	Q	S	Standard method	100 mL	24 C	No		No	Aprox. 15 min	Yes, rinsed with deonised water	
L13	S		S		Standard, Validated, Accredited method	170	22 ± 2 °C	Yes	Calibrated termometer	No	-	The bowls were washed before first migration	Yes
L14	S	S	S	S	Accredited method	150 ml added, 100 - 110 ml recovered	22 degC	Yes	Calibrated datalogger	No	10 minutes	No	
L16	S	S	S	S	Standard, Accredited method	150	22	Yes	Non calibrated device	No	30	yes, high purity water	Yes
L17	S	S	S	S	Validated method	145 ml	22 oC	Yes	Other	No	80 min	Yes, with deionized water	Yes
L18	S	S	S	S	Accredited method	160 ml	Room temperature 21 °C	No		No	95 min	Rinsed with water	Yes
L19	S	S	Q	Q	Validated method	150 mL	22 ± 0,6 C	No		No	60 minutes	The bowls were washed with deionised water	Yes
L20	Q	S	S	S	Validated method	150	20	Yes	Calibrated termometer	No	30	Water nanopur and dried	
L21	S	S	S	S	Accredited method	155 ml	22 Celcius	No		No	1 day	No	Yes
L22	S	S	S	S	Standard, Validated method	150mL	22 ± 1°C	Yes	Calibrated datalogger	No	30 minutes	Yes, the bowls were washed before first and between sucessive migrations with liquid detergent, rinsed with tapwater, deionised water, drained and wiped dry with filter paper (we followed washing procedure from EN1388-1).	Yes
L23	S	S	S	S	Accredited method	170	22	No		No	20	NO	Yes
L24	S	Q	S	S	Accredited method	175	22	Yes	Calibrated termometer	No	25 min	They were rinsed with distilled water in between successive migrations.	Yes
L25	S	S	S	S	Validated method	145	22	No		No			
L26	S	U	U	S	Accredited method	145	22	Yes	Calibrated datalogger	No	30 minutes	No	Yes

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