European Commission

JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

EURL-FCM-01-2017
Proficiency test report

Quantification of migration of bisphenol A from can coatings by article filling

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2018

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JRC 111695

Luxembourg: Publications Office of the European Union, 2018

How to cite this report: S. Valzacchi, J.L. Vicente, F. Cordeiro, P. Robouch and E.J. Hoekstra, "*EURL-FCM-01-2017 proficiency test report: Quantification of migration of bisphenol A from can coatings by article filling".* JRC Technical Reports, 2018; JRC111695

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Quantification of migration of bisphenol A from can coatings by article filling

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

Abstract

In view of the upcoming European Regulation on the use of Bisphenol A (2,2-bis(4-hydroxyphenyl)propane or BPA) in varnishes and coatings for food metal cans, the European Union Reference Laboratory for Food Contact Materials (EURL-FCM) organised a proficiency test (PT) to assess the analytical capabilities of the EU National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs) on the determination of the migration of BPA from coated cans at the proposed specific migration limit (SML) of 0.05 mg kg^{-1} food. Representatives of the Association of Southeast Asian Nations (ASEAN) were also invited to participate in the frame of international collaboration with the European Commission.

The tailored epoxy resins coated metal cans used as PT test specimens were provided by the Ardha Group (Sutton-in-Ashfield, UK). The established migration conditions were 70 °C for 2 hours, using food simulant D1 (ethanol 50 %, v/v). Two additional solutions had to be analysed: (i) a food simulant D1 obtained from a migration test spiked with BPA (Solution 1); and (ii) a freshly prepared food simulant D1 spiked with BPA (Solution 2).

Fifty-one laboratories (26 NRLs, 18 OCLs and 7 ASEAN laboratories) participated in this PT.

All assigned values were determined by the EURL-FCM using a single-laboratory validated method based on High-Performance Liquid Chromatography with Fluorescence detection (HPLC-FLD). The relative standard deviations for proficiency assessment (σ_{pt}) were set to 25 % (of the assigned value) for the migration from cans, and to 15 % for Solution 1 and Solution 2. Laboratory results were assessed using z and ζ scores according to ISO 13528:2015.

More than 80 % of the participants obtained satisfactory z scores, thus **confirming the analytical capability of most of the participating NRLs and OCLs to enforce the upcoming SML for BPA**. However, roughly half of the participants reported unrealistic measurement uncertainties that need to be reviewed. Hence, a dedicated training on measurement uncertainty will be organised by the EURL-FCM in 2018.

List of abbreviations and definitions

ASEAN	Association of Southeast Asian Nations
BPA	2,2-Bis(4-hydroxyphenyl)propane; Bisphenol A
DG SANTE	Directorate General for Health and Food Safety
EURL-FCM	European Union Reference Laboratory for Food Contact Materials
GC-MS	Gas chromatography with mass spectrometry
HPLC-FLD	High-performance liquid chromatography with fluorescence detection
HPLC-UV	High-performance liquid chromatography with ultraviolet detection
JRC	Joint Research Centre
LC-MS	Liquid chromatography with mass spectrometry
LC-MS/MS	Liquid chromatography with tandem mass spectrometry
LC-TOF/MS	Liquid chromatography time-of-flight mass spectrometry
LOQ	Limit of quantification
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PT	Proficiency test
SML	Specific migration limit
UPLC-MS	Ultra-performance liquid chromatography with mass spectrometry
UPLC-MS/MS	Ultra-performance liquid chromatography with tandem mass spectrometry

List of symbols and definitions

k	coverage factor
σ_{pt}	standard deviation for proficiency test assessment
$u(x_i)$	calculated standard measurement uncertainty (of 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	reported mean value by participant "i"
x_{pt}	assigned value
z	z score
ζ	zeta score

Contents

1 Introduction

The use of certain epoxy resins as coating material of metal cans for food is debated due to the suspected endocrine disruptor activity of Bisphenol A (2,2-Bis(4-hydroxyphenyl)propane or BPA). In the absence of comprehensive European legislation on coatings for food contact, the European Commission drafted a measure proposing a specific migration limit (SML) of 0.05 mg/kg food for the migration of BPA from varnishes and coatings.

In view of the upcoming measure, the European Union Reference Laboratory for Food Contact Materials (EURL-FCM), hosted by the Joint Research Centre (JRC), organised a proficiency test (PT) for the determination of the mass fraction of BPA in food simulant D1.

This PT was agreed with the Directorate General for Health and Food Safety (DG SANTE) as part of the EURL-FCM annual work program for 2016-2017. The PT was open to National Reference Laboratories (NRLs) and to Official Control Laboratories (OCLs). In addition, several laboratories from ASEAN countries were invited to participate in the frame of the international training programme of the EURL-FCM.

2 Scope

One of the core duties of the EURL-FCM, established by Commission Regulation (EC) No 882/2004 [1], is to organise inter-laboratory comparison exercises for the benefit of NRLs and OCLs.

At first this PT aims to assess the performance of the NRLs and OCLs in the determination of the mass fraction of BPA migrating from metal cans coated with epoxy resins (containing BPA) by article filling. The migration conditions were set to 70 °C for 2 hours, using food simulant D1 (ethanol 50% v/v), a conventional food simulant for foods containing more than 20 % of alcohol and oil in water emulsions.

In addition, participants were requested to determine the mass fraction of BPA in two solutions: (i) food simulant D1 solution spiked with BPA after migration of BPA from cans (Solution 1); (ii) pure food simulant D1 solution spiked with BPA (Solution 2). The results for Solution 2 would allow evaluating the efficiency of the instrumental analysis; while the comparison of results obtained for Solution 1 and Solution 2 may identify potential matrix effects.

Participants were also requested to record and report the temperature of the food simulant during the migration step and any decrease in (loss of) food simulant volume.

3 Set up of the exercise

3.1 Time frame

The organisation of the PT exercise EURL-FCM ILC 01-2017 was agreed by the EURL-NRL-FCM network during its Plenary meeting held in Berlin on June 6-7, 2016. The PT was officially announced during the networks' Plenary meeting held in Ispra on May 16-17, 2017. Invitation letters were sent via e-emails to NRLs and OCLs on June 7, 2017 (Annex 1). The registration deadline was set to June 16, 2017. Samples were sent to participants on June 26, 2017. The dispatch was monitored by the EURL-FCM using the messenger's parcel tracking system on the internet. The original deadline for reporting of results was set to July 31, 2017 (Annex 1), and postponed to August 31, 2017, to compensate for some late sample delivery due to customs delays.

3.2 Confidentiality

The procedures used for the organisation of PTs are accredited according to ISO 17043:2010 [2] and guarantee that the identity of the participants and the information provided by them is treated

as confidential. However, the "lab codes" of the NRLs that have been appointed in line with Regulation (EC) No 882/2004 [1] may be disclosed to DG SANTE upon request for the purpose of an assessment of their (long-term) performance.

3.3 **Distribution of samples**

Each participant received:

- The "Accompanying letter" (Annex 2);
- Five metal cans coated with epoxy resins (3 to perform the migration experiments, 1 for the temperature control, and 1 spare);
- Two bottles labelled "Solution 1: EURL FCM 01/2017 BPA" and "Solution 2: EURL FCM 01/2017 BPA" (each containing 50 mL); and
- A “Confirmation of receipt” form to be sent back to the JRC after receipt of the test specimens (Annex 3).

3.4 **Instructions to participants**

Detailed instructions were given to participants in the "Accompanying letter" mentioned above (Annex 2). The measurand was defined as "the mass fraction of BPA in food simulant D1".

Participants were asked to perform three independent migration experiments, determine the mass fraction of BPA migrated from cans, and report the mean value (x_i) together with the corresponding expanded uncertainty ($U(x_i)$) and coverage factor (k). Similarly, participants were requested to determine the mass fraction of BPA in Solution 1 and Solution 2, and report the respective $x_i \pm U(x_i)$ (k).

The "migration from cans" experiment had to be performed using the food simulant D1 (ethanol 50% v/v), at 70 °C for 2 hours. Participants had to report the temperature of the food simulant during the migration step, at time intervals of 5 minutes, using a calibrated and certified data-logger for temperature equipped with an immersion probe (when available).

Results had to be reported in "mg kg^{-1}" with two significant figures, assuming a density of the food simulant of 1 g cm^{-3}. Participants were requested to use their routine methods for the analysis.

Participants received an individual code to report online their measurement results and to complete a dedicated questionnaire designed to collect additional information related to measurements and laboratories (Annex 4). The laboratory codes were given randomly and communicated to the participants by e-mail.

4 Test specimen

4.1 Preparation

The tailor-made cans used in the PT were provided to the EURL-FCM by the Ardagh Group (Sutton-in-Ashfield, UK), a European company producing cans for the food industry. A dedicated batch of cans was produced with a reduced curing time of the epoxy resin coating to obtain a release of BPA around the proposed specific migration limit (SML) of 0.05 mg kg^{-1}.

At first the EURL-FCM tested some cans for BPA migration at 70 °C for two hours, using the three food simulants listed Regulation (EU) No 10/2011 [3], namely: acetic acid 3 % w/v ("B"); ethanol 50 % v/v ("D1"); and sunflower oil ("D2"). While no BPA migration could be detected in food simulants B and D2, the level of migrated BPA in food simulant D1 was found to be around the target value of 0.05 mg kg^{-1}. It was therefore decided to use the "food simulant D1" to assess the competence of participants in performing specific migration tests of BPA from can coatings.

The EURL-FCM prepared Solution 1 by spiking a migration solution of food simulant D1 resulting from a migration experiment with the epoxy resin coated cans at 70 °C for 2 h with BPA. The procedure consisted of the preparation of a stock solution of BPA in methanol by adding 90 mg of BPA (with a declared minimum purity of 99%) in a 100 mL flask. Next, a five litres volumetric flask was spiked with 0.2 mL of the stock solution (corresponding to 180 µg of BPA) and filled to the mark with the migration solution. Then aliquots of 50 mL were transferred into 100 mL glass bottles and sealed with Teflon lined crimp caps. The same protocol was used to prepare Solution 2 using a freshly prepared food simulant D1 to fill to the 5 L mark instead.

4.2 Homogeneity and stability

The EURL-FCM performed the homogeneity and stability studies according to ISO 13528:2015 [4], using a single-laboratory validated method based on High-Performance Liquid Chromatography with Fluorescence detection (HPLC-FLD).

The analyses were performed with a Hewlett-Packard HPLC 1100 system equipped with a C18 column (150 mm x 3 mm; 5 µm) with a water/methanol mobile phase and gradient elution. The following performance characteristics were derived from the validation study: repeatability and intermediate-precision relative standard deviations of 1.6 % and 2.7 %, respectively; a limit of quantification (LOQ) of 0.003 mg kg^{-1}; and a standard measurement uncertainty of 5 %.

The homogeneity experiment consisted of duplicate analysis on 12 samples randomly selected. The analyses were performed in random order. All three materials were rated sufficiently homogeneous at a sample intake of 2 mL (Annex 5). The contributions from homogeneity (u_{hom}, Table 1) to the standard uncertainty of the assigned value ($u(x_{pt})$) for Solution 1 and Solution 2 were calculated using SoftCRM [5]. Since replicate measurement could not be conducted for the "migration from cans" samples (destructive test), u_{hom}(cans) was calculated as the between-sample standard deviation.

The stability study was performed at time 0 and after 16 weeks, at 20 and 40 °C. A test was also performed at 60 °C for 2.5 days (short term stability) to simulate a shipment at extreme high temperature. Samples were analysed in duplicate under repeatability conditions. No significant differences in BPA concentrations were found, thus confirming the stability of the investigated samples during the period of the exercise (Annex 6). Hence, the uncertainty contribution due to stability was set to zero (u_{st} = 0) for the three types of samples (Table 1).

5 Assigned values and corresponding uncertainties

5.1 Assigned values

The assigned values (x_{pt}) and the standard measurement uncertainties due to characterisation (u_{char}) were determined for the three test specimens.

The known amount of BPA spiked in Solution 2 (0.0362 mg L^{-1}) was experimentally confirmed by HPLC-FLD in the frame of the homogeneity study (Annex 5); this formulation/nominal value was then set as the assigned value for Solution 2. The law of uncertainty propagation was applied to calculate u_{char}(Sol.2) (Table 1).

The concentrations of (i) BPA "migrated from cans" and (ii) BPA in Solution 1 were determined experimentally by the EURL-FCM - in the frame of the homogeneity study using the HPLC-FLD method (Annex 5). Consequently, the averages (of all the experimental results) were set as assigned values (x_{pt}) for the two test specimens, while a u_{char} of 5 % was derived from the validation study for the two test specimen (Table 1)..

5.2 Uncertainty of the assigned value

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 contributions from homogeneity (u_{hom}), and stability (u_{st}), in compliance with ISO 13528:2015 [4]:

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

5.3 Standard deviation for proficiency assessment

The relative standard deviation for PT assessment (σ_{pt}, in %), based on the expert opinion ("perception", see Clause 8.2 of ISO 13528:2015 [4]), were set to 15 % of the assigned value for Solutions 1 and 2, and to 25 % for the BPA after "Migration from Cans" (Table 1).

Table1: Assigned values (x_{pt}, $u(x_{pt})$ and $U(x_{pt}, k=2)$); standard uncertainties (u_{char}, u_{hom}, u_{st}); and standard deviations for PT assessment σ_{pt}.

	Migration from cans	Solution 1	Solution 2
$x_{pt}{}^{*}$	0.0443	0.0873	0.0362
$u_{char}{}^{*}$	0.0022	0.0044	0.0002
$u_{hom}{}^{*}$	0.0029	0.0009	0.0005
$u_{st}{}^{*}$	0	0	0
$u(x_{pt})^{*}$	0.0037	0.0044	0.0006
	8.3%	5.1%	1.6%
$U(x_{pt})^{*}$, k=2	0.0073	0.0089	0.0012
$\sigma_{pt}{}^{*}$	0.0111	0.0131	0.0054
$\sigma_{pt}\,(\%)$	25%	15%	15%
$u(x_{pt})/\sigma_{pt}$	0.33	0.34	0.11

(*) Values are expressed in mg kg^{-1} of food simulant (assuming a density of 1 g cm^3).

6 Evaluation of results

The results reported by the laboratories were assessed following the administrative and logistic procedures of the JRC Unit in charge of the EURL-FCM, which is accredited for the organisation of PTs according to ISO/IEC 17043:2010 [2].

6.1 Scores and evaluation criteria

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

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

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

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 test assessment.

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

|score| ≤ 2	satisfactory performance	(green in Annexes 7 – 9)
2 < |score| < 3	questionable performance	(yellow in Annexes 7 – 9)
|score| ≥ 3	unsatisfactory performance	(orange in Annexes 7 – 9)

The *z* scores compare the participant's deviation from the assigned value with the standard deviation for proficiency test asessment (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 $(u(x_i) = 0)$. When k was not specified, the reported expanded measurement uncertainty was considered as the half-width of a rectangular distribution; $u(x_i)$ was then calculated by dividing this half-width by √3, as recommended by Eurachem [7].

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

The standard measurement uncertainty from the laboratory $u(x_i)$ is most likely to fall in a range between a minimum and a maximum allowed uncertainty (case a": $u_{min} \leq u_i \leq u_{max}$). u_{min} is set to the standard uncertainties of the assigned values $u(x_{pt})$. It is unlikely that a laboratory carrying out the analysis on a routine basis would determine the measurand with a smaller measurement uncertainty than the expert laboratories chosen to establish the assigned value. u_{max} is set to the standard deviation accepted for the PT assessment (σ_{pt}). Consequently, case "a" becomes: $u(x_{pt}) \leq u(x_i) \leq \sigma_{pt}$.

If $u(x_i)$ is smaller than $u(x_{pt})$ (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 specimen. If those are large, measurement uncertainties smaller than $u(x_{pt})$ are possible and plausible.

If $u(x_i)$ is larger than σ_{pt} (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.

6.2 General observations

Twenty-nine NRLs from twenty-seven countries (including Switzerland) registered to the exercise and twenty-six reported results. The NRLs of Malta and The Netherlands did not register and the NRLs of Czech Republic and Romania, and one NRL of France did not report results. Twenty OCLs registered to the exercise (six from Germany; three from Italy, Poland, Spain and United Kingdom; one from Belgium and Czech Republic) and two of them did not report results. Seven laboratories from six countries of ASEAN (Indonesia, Myanmar, Philippines, Singapore, Thailand and two from Vietnam) registered to the exercise and all reported results.

6.3 Participants results and scoring

6.3.1 Performances

Annexes 7 to 9 present the results reported as tables and graphs for each measurand, where laboratories are denoted as "N-XXX", "O-XXX" and "A-XXX" for NRLs, OCLs and ASEAN laboratories respectively. The z and ζ scores and the corresponding graphs were calculated and plotted by the EURL-FCM, while the "kernel density plots" were obtained using the software developed by the Statistical Subcommittee of the Analytical Method Committee of the UK Royal Society of Chemistry [6].

The laboratory performances for the determination of the mass fraction of BPA migrated from cans, in Solution 1, and in Solution 2 were assessed using the z and ζ scores (Figure 1). According to the z scores, **fourty two or more participants (out of 51, above 85 %) reported satisfactory results for the three measurands** (Figure 1) with BPA concentrations ranging from 0.0362 ± 0.0012 mg kg^{-1} to 0.0873 ± 0.0089 mg kg^{-1} (Table 1). The decrease in satisfactory results observed for ζ scores in Figure 1 (around 65 %) may be due to the "apparent under-estimated" measurement uncertainties reported (see discussion in the next section).

The mode of the reported results for "Cans" and "Solution 2" are in good agreement with the assigned values derived from the EURL-FCM measurements (see Kernel distribution plots Annexes 7 and 9). The BPA concentration in "Solution 1" measured by the EURL-FCM was in agreement with the sum of the BPA concentration in the migration solution used for the preparation and the concentration of the spike. The similar scatter of results of ca. 12 % observed for Solution 1 and 2 indicates the absence of possible interferences from other migrating substances. This scatter could be attributed to the variability of the instrumental analysis.

Laboratories O-060; O-081 and A-501 reported significantly over-estimated results and should review their calculations to identify possible "multiplication/dilution" factor mistakes.

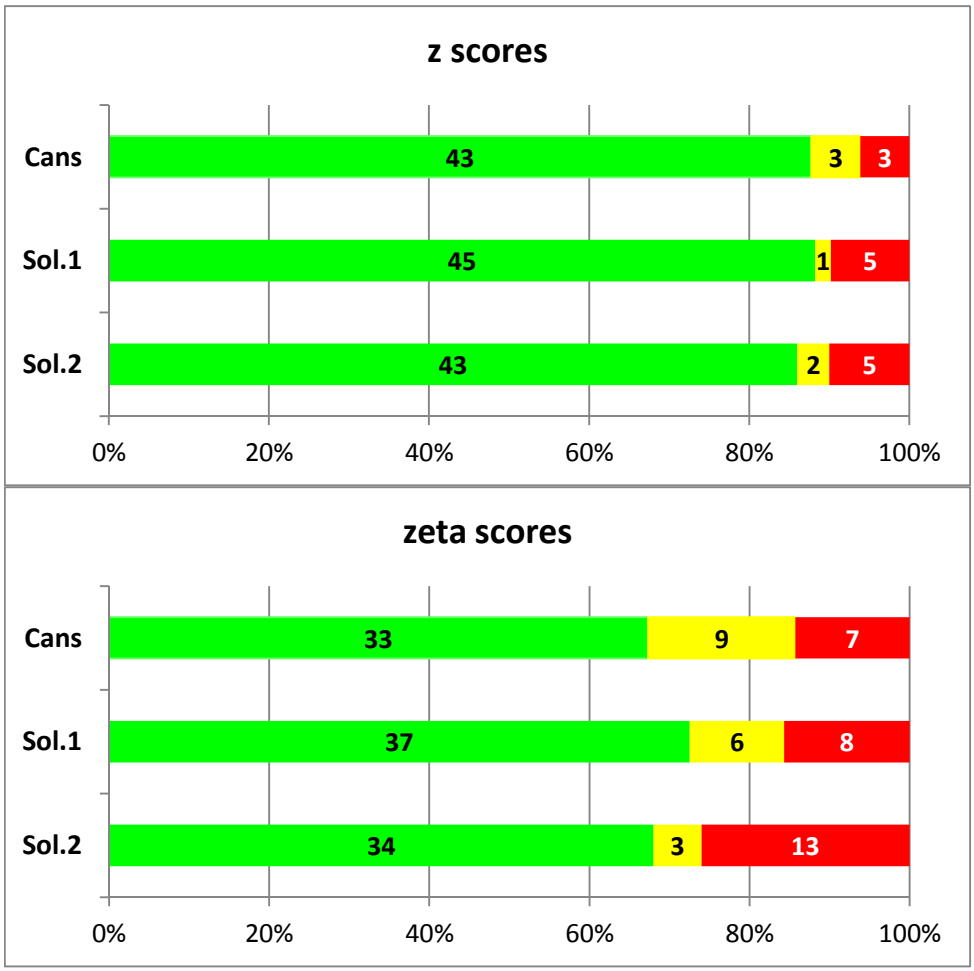


Figure 1: Overview of laboratory performance expressed as z and ζ scores for the mass fraction of BPA migrated from cans (CAN); BPA in Solution 1 (Sol.1); and BPA in Solution 2 (Sol.2). Satisfactory, questionable and unsatisfactory performances indicated in green, yellow and red, respectively. The corresponding numbers of participants are indicated in the columns.

Table2: Overview of performance expressed as z scores - per laboratory category - for the mass fraction of BPA migrated from cans; BPA in Solution 1; and BPA in Solution 2.

	NRLs (26)			OCLs (18)			ASEAN (7)		
	S	Q	U	S	Q	U	S	Q	U
Cans	26	0	0	12	2	2	4	1	2
Solution 1	25	1	0	14	0	4	6	0	1
Solution 2	26	0	0	11	0	6	6	0	1

Satisfactory (S), Questionable (Q) and Unsatisfactory (U) performances

6.3.2 Measurement uncertainties

All participants reported their measurement uncertainty (MU) estimates for the three measurands. Figure 3 presents the corresponding uncertainty assessment. It seems that the majority of laboratories have underestimated their standard MUs (i.e. 25 or 29 out of 51 for "cans" or Solution 1" - "Case b"; $u(x_i) < u(x_{pt})$), while only 15 or 20 of them submitted realistic MUs ("Case a"; $u(x_{pt}) < u(x_i) < \sigma_{pt}$). This may be due to the fact that 17 laboratories reported for "Cans" relative MUs ranging from 5 to 8 % (strictly below 8.2 %, even though comparable), and 9 laboratories reported for "Solution 1" relative MUs ranging from 3.5 to 5 % (idem, strictly below 5.2 %). Only few laboratories reported "unreasonable" relative standard MUs below 1 % (certainly underestimated) or above 45 % (certaintly overestimated); the latter may have reported in % instead of mg kg^{-1} (e.g N-016, 0-026, A-501).

Several approaches were used to estimate measurement uncertainty (Table 3). Most of the laboratories derived their uncertainty estimates from their single-laboratory validation study or from measurement of replicates (cf. precision), the latter clearly resulting in underestimated statements.

Having observed a significant scatter of reported MUs, the EURL-FCM committed to organise in 2018 a dedicated training on "*How to estimate my measurement uncertainty*".

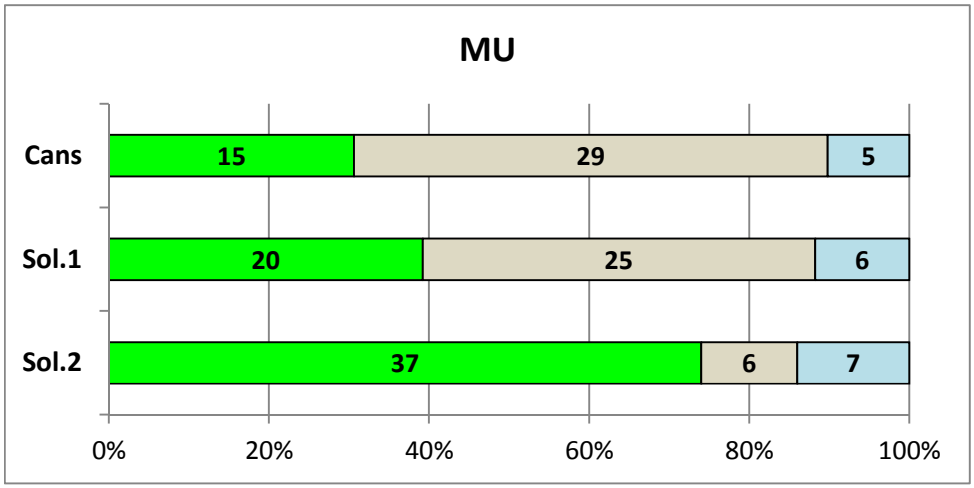


Figure 3: Review of uncertainties reported per measurand. Corresponding number of laboratories indicated in the graph. Case "a": $u(x_{pt}) \leq u(x_i) \leq \sigma_{pt}$ (green); Case "b": $u(x_i) < u(x_{pt})$ (light brown); and Case "c": $u(x_i) > \sigma_{pt}$ (blue).

Table 3: Overview of the approaches used to estimate the uncertainty of measurement (multiple options could be chosen by participants).

Approach	No of labs cans	No of labs solutions
According to GUM	2	5
Known uncertainty of standard method	0	0
Obtained from in-house validation study	13	14
Measurements of replicates (precision)	11	8
Estimation based on judgment	1	1
Horwitz	2	3
According to Nordtest	1	0
Obtained from proficiency test data	1	1

6.4 **Additional information from the questionnaire**

The majority of the participants stated to be experienced in BPA analysis (94 %), to have a validated method for BPA analysis (74 %), and to be accredited according to ISO/IEC 17025 for this type of analysis (67 %).

Participants were requested to register the temperature of the food simulant during the migration from cans, to specify if a certified thermometer was used, and to report the loss in volume of food simulant lost observed at the end of the the migration experiment (Annex 10).

Most of the participants (90 %) obtained a temperature of the food simulant within the expected tolerance range (70 ± 2 °C) required by EN 13130-1:2004 [8]. Five laboratories (2 NRLs, 1 OCL and 2 ASEAN) reported temperature ranges below the tolerance limit (Figure 4) without any significant effect on their migration results (four satisfactory and one questionable z scores).

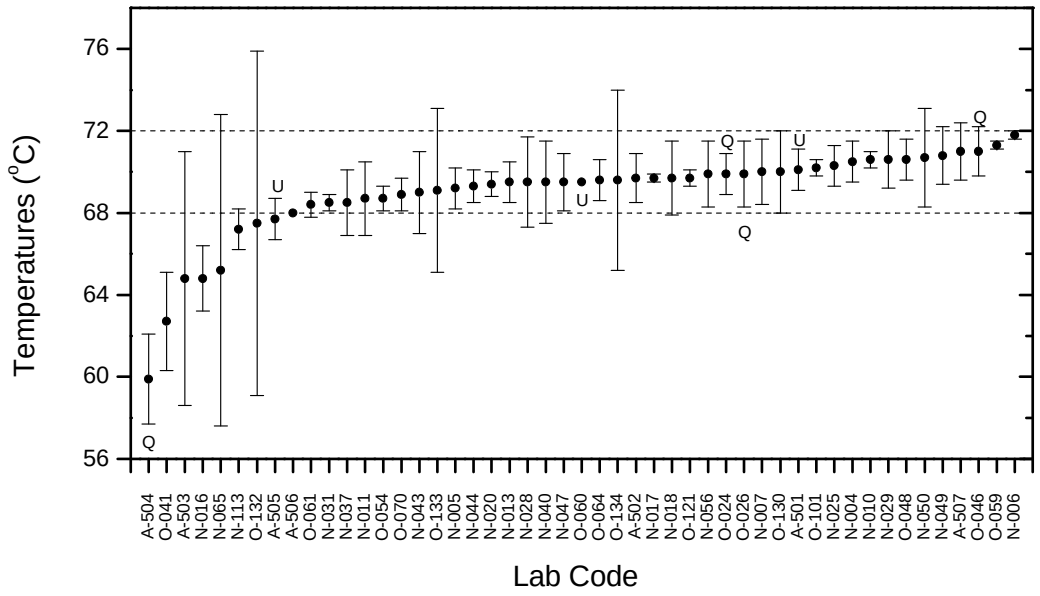


Figure 4: Temperature ranges recorded by participants in the food simulant D1 during the BPA migration from cans

Ten laboratories (out of 49) – of which five are accredited laboratories - did not use a certified thermometer to monitor the temperature in the food simulant during the migration test, which constitutes a major infringement to the requirement set by the standard EN 13130-1:2004 [8].

Half of the participants reported a decrease in volume of food simulant (loss) after the migration test below 11 mL, while one participant reported a loss up to 43 mL (equivalent to 10 % of the total volume of food simulant contained in the can). Although no clear correlation could be established between the loss of simulant and poor performance, the EURL-FCM recommends covering the can during the migration experiment with a watch glass - kept in place by a vinyl covered lead ring - to collect back the condensed simulant .

Several analytical techniques were applied by the participants for the determination of BPA (Annex 8), with the samples analysed directly, possibly after filtration. Most of them used high-performance liquid chromatography with fluorescence detection (HPLC-FLD, 61 %), followed by liquid chromatography with mass spectrometry (LC-MS, 31 %); while three participants used HPLC with ultraviolet detection (HPLC-UV). Only one participant used gas chromatography with mass spectrometry (GC-MS) with liquid-liquid extraction of BPA from the food simulant. No direct correlation could be found between the analytical methods used by the participants and the quality of the reported results.

7 Conclusions

The EURL-FCM organised a PT to assess the analytical capabilities of the EU NRLs and OCLs to determine the mass fractions of BPA migrated from food cans coated with epoxy resins and/or in solutions of a food simulant.

The overall performance of the NRLs was satisfactory for the three measurands of interest, with only two questionable z scores (out of 78 reported results). This confirms the analytical capabilities of the NRLs to enforce the requirement of the foreseen level of 0.05 mg kg^{-1} for the migration of BPA from food cans. Similarly, most of the OCLs scored satisfactorly with twelve unsatisfactory results over the three measurands (out of 51 reported results).

Less than half of the participants estimated realistic measurement uncertainties. The EURL-FCM will therefore organise in 2018 a dedicated trainign on "*How to estimate my measurement uncertainty*".

Acknowledgements

The EURL-FCM thanks the Ardagh Group (Sutton-in-Ashfield, UK) for providing the coated cans used as PT test specimens. Authors wish to thank Giorgia Beldì and Natalia Jakubowska (EURL-FCM) for their valuable contributions to this project. The fifty-one laboratories listed below are kindly acknowledged for their participation in this PT.

European National Reference Laboratories

Austria	Austrian Agency for Health and Food Safety (AGES), Institut für Lebensmittelsicherheit
Belgium	Institute of Public Health, ISSP-LP
Bulgaria	National Centre of Public Health & Analysis
Cyprus	State General Laboratory
Croatia	Croatian National Institute of Public Health, Laboratory of Common Goods Items
Denmark	National Food Institute, Technical University of Denmark
Denmark	Danish Veterinary & Food Administration, Laboratory
Estonia	Central Laboratory of Chemistry
Finland	Finnish Customs Laboratory
France	SCL Laboratoire de Bordeaux-Pessac
Germany	German Federal Institute for Risk Assessment (BfR)
Greece	General Chemical State Laboratory, Laboratory of Articles and Materials in Contact with Foodstuffs
Hungary	National Food Chain Safety Office, Food and Feed Safety Directorate
Ireland	Public Analyst's Laboratory
Italy	Istituto Superiore di Sanità, Laboratorio Esposizione e Rischio da Materiali
Latvia	Institute of Food Safety, Animal Health and Environment "BIOR"
Lithuania	National Public Health Surveillance Laboratory, Laboratory of Chemistry
Luxembourg	National Health Laboratory, Food Laboratory
Poland	National Institute of Public Health, Department of Food Safety
Portugal	Catholic University, Faculty of Biotechnology, Packaging Department
Slovak Republic	Regional Public Health Authority in Poprad, National Reference Centre and Laboratory for Material and Articles Intended to Come into Contact with Food
Slovenia	National Laboratory of Health, Centre for Environment and Health
Spain	Spanish Agency for Consumer Affairs, Food Safety and Nutrition
Sweden	National Food Administration, Chemistry Division
Switzerland	Official Food Control Authority of the Canton of Zurich
United Kingdom	Food and Environment Research Agency

Official Control Laboratories

Belgium	Laboratoire Fédéral pour la Sécurité Alimentaire (LFSAL)
Czech Republic	Public Health Institute of Ústí nad Labem
Germany	Landeslabor Schleswig-Holstein
Germany	Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen Sachsen
Germany	Chemisches und Veterinäruntersuchungsamt, Münsterland-Emscher-Lippe
Germany	Chemisches und Veterinäruntersuchungsamt, Stuttgart
Germany	LAVES, Institut für Bedarfsgegenstände, Lüneburg
Germany	Thüringer Landesamt für Verbraucherschutz
Italy	Istituto Zooprofilattico Sperimentale della Lombardia ed Emilia Romagna, Reparto Chimico degli Alimenti
Italy	Agenzia Regionale per la Protezione Ambientale del Piemonte
Italy	Agenzia Provinciale per la Protezione dell'Ambiente, Provincia Autonoma di Trento
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna, Katowice
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna, Białystok
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna, Rzeszów
Spain	Laboratorio de Salud Pública de Valencia
Spain	University of Zaragoza, Department of Analytical Chemistry
United Kingdom	Scientific Services, Worcestershire County Council
United Kingdom	Staffordshire Scientific Services

Laboratories from ASEAN Countries

Indonesia	National Agency of Drug and Food, Food division, National Quality Control Laboratory of Drug and Food
Myanmar	Food and Drug Administration, Food Chemical Laboratory
Philippines	Food and Drug Administration, Toxicology Section, Common Service Laboratory
Singapore	Health Science Authority, Food Safety Division, Applied Sciences Group
Thailand	Division of Food Products and Food Contact Materials, Department of Science Service
Vietnam	National Institute for Food Control
Vietnam	Quality Assurance and Testing Center 3, Consumer Laboratory

References

[1] Commission Regulation (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules, OJ L 165, 30.4.2004, p. 1–141.

[2] ISO/IEC 17043:2010 "Conformity assessment – General requirements for proficiency testing", International Organization for Standardization, Geneva, Switzerland.

[3] Commission Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food, OJ L 12, 15.1.2011, p. 1–89.

[4] ISO 13528:2015 "Statistical methods for use in proficiency testing by interlaboratory comparisons", International Organization for Standardization, Geneva, Switzerland.

[5] SoftCRM, software for the development of Certified Reference Materials (CRMs). www.eie.gr/iopc/softcrm

[6] Analytical Method Committee, "Representing data distributions with kernel density estimates", AMC Tech. Br. 4 (2006); www.rsc.org/images/brief4_tcm18-25925.pdf

[7] 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 .

[8] EN 13130-1:2004 "Materials and articles in contact with foodstuffs. Plastics substances subject to limitation. Guide to test methods for the specific migration of substances from plastics to foods and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants", European Committee for Standardization (CEN).

Annexes

Annex 1. Invitation e-mail

Ref. Ares(2017)2849267 - 07/06/2017

Cc: HOEKSTRA Eddo (JRC-ISPRA); JRC EURL FCM
Subject: EURL-FCM ILC01-2017 - Registration

Dear participant,

On behalf of the EURL for Food Contact Materials, we would like to invite you to participate in the Proficiency Test for Bisphenol A in Can Coatings 2017.

Please register electronically by using the link below and following the instructions on screen.
https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1782

Once you have submitted your registration electronically, you will have to:

1) Print your registration form, as indicated on screen
2) Sign it, date it and send it to us by e-mail (JRC-EURL-FCM@ec.europa.eu) or fax (+39 033278 5707)

Only upon receipt of the registration signed form, we will accept you as a participant. Please **register yourselves by Friday the 16th of June**.

Samples and further instructions will be **sent to you before the 23rd of June**. The deadline for **submission of results is the 31st of July**.

You will be sent 5 cans for the migration, a food simulant D1 solution spiked with BPA and a migration solution spiked with BPA for analysis.

If you are NRL, you are kindly reminded that, according to Regulation (EC) 882/2004 it is your duty to participate in the PTs organised by the EURL-FCM.
Your participation is free of charge.

Do not hesitate to contact us if you have any further questions.

Kind regards,

The EURL-FCM Team

Joana Lobo Vicente, Ph.D
Project Officer

European Commission
Directorate General Joint Research Centre
Directorate F – Health, Consumers & Reference Materials
Food and Feed Compliance Unit

Via Enrico Fermi, 2749, TP260
I-21027 Ispra (VA), Italy
Tel: +39 033278 6679

Annex 2. Accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Directorate F – Health, Consumers and Reference Materials
European Union Reference Laboratory for Food Contact Materials

Ispra, 23rd June 2017
Ares(2017)3165411

Subject: Participation in EURL FCM 01/2017 BPA in can coating Proficiency Test

To whom it may concern,

Thank you for participating in the EURL FCM ILC01 2017 BPA in can coating Proficiency Test. This is organised in support of EC No 882/2004 on official controls.
The parcel you received contains, in addition to this letter:

- 5 cans (3 to perform the migration with D1 simulant, 1 for the temperature control, and an extra one as spare);
- Two vials labelled "Solution 1 EURL FCM 01/2017 BPA" and "Solution 2 EURL FCM 01/2017 BPA" (approx. 50 mL each);
- the "Confirmation of receipt" form.

Upon arrival of this parcel, please check whether the test items are undamaged after transport, and send us by email the "Confirmation of receipt" form, to JRC-EURL-FCM@ec.europa.eu .

Samples can be stored at room temperature.

The mandatory measurements are:

- **the mass fraction of BPA in mg kg^{-1} in the first migration from the cans,**
- **the mass fraction of BPA in mg kg^{-1} in Solution 1**
- **the mass fraction of BPA in mg kg^{-1} in Solution 2**

The migration test shall be performed in food simulant D1, at 70±2 °C for 2 hours (the cans should be filled up to 5 mm from the rim).

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

Perform the measurements and report:

- the **mean** of your results (in mg kg^{-1}) using 2 significant figures (e.g. 0.073 mg kg^{-1}), assuming that the density of food simulant D1 is 1 g cm^{-3}
- the associated expanded **uncertainty** (in mg kg^{-1}) using 2 significant figures,
- **the coverage factor for the calculation of expanded uncertainty.**

Via Enrico Fermi 2749, TP 260, 21027 Ispra (VA) – Italy Tel.: +39 033278 5293
https://ec.europa.eu/jrc/en/eurl/food-contact-materials ; E-mail: JRC-EURL-FCM@ec.europa.eu

The reporting is done using the software MILC (the same that was used for registration): https://web.jrc.ec.europa.eu/ilcReportingWeb

We will provide you with a personal password key by e-mail to access the software MILC to report your results, together with instructions on how to submit them. The system will guide you through the reporting procedure. You will also need to complete the corresponding questionnaire.

You are also asked to **report the temperature of the food simulant D1 during the migration test** (Table in the Questionnaire in MILC). The results from the temperature control experiment will be used to support the results of the can migration and they will not be used to assess your performance. You shall register the temperature using a thermometer immersed in the simulant during the migration test (you can use one of the cans only for the measurement of the temperature). The temperatures shall be registered with a certified calibrated thermometer.

You are also kindly asked to measure the loss of simulant (mL) during migration and insert the value in the appropriate section in the Questionnaire.

Do not forget to confirm your results after submission using MILC.

Please check carefully your report. If mistakes are detected, please contact the PT coordinator as soon as possible before the reporting deadline.

The deadline for submission of results is **31/08/2017**.

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.

Do not hesitate to contact me for further information.

With kind regards,

/signed electronically in Ares/

Dr. Sandro VALZACCHI
EURL-FCM ILC01-2017 Coordinator

Cc: H. Emons (Head of Unit, Food & Feed Compliance unit)
E. Hoekstra (Team Leader Food Contact Materials)

Via Enrico Fermi 2749, TP 260, 21027 Ispra (VA) – Italy Tel.: +39 033278 5293
https://ec.europa.eu/jrc/en/eurl/food-contact-materials ; E-mail: JRC-EURL-FCM@ec.europa.eu

Annex 3. Confirmation of receipt form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Directorate F – Health, Consumers and Reference Materials
European Union Reference Laboratory for Food Contact Materials

Ispra, 22nd June 2017
Ares(2017)2273549

To Whom it May Concern,

Subject: "Confirmation receipt" form
EURL-FCM ILC01-2017 BPA in can coating Proficiency Test

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

Date of package arrival _____

Remarks _____

_____

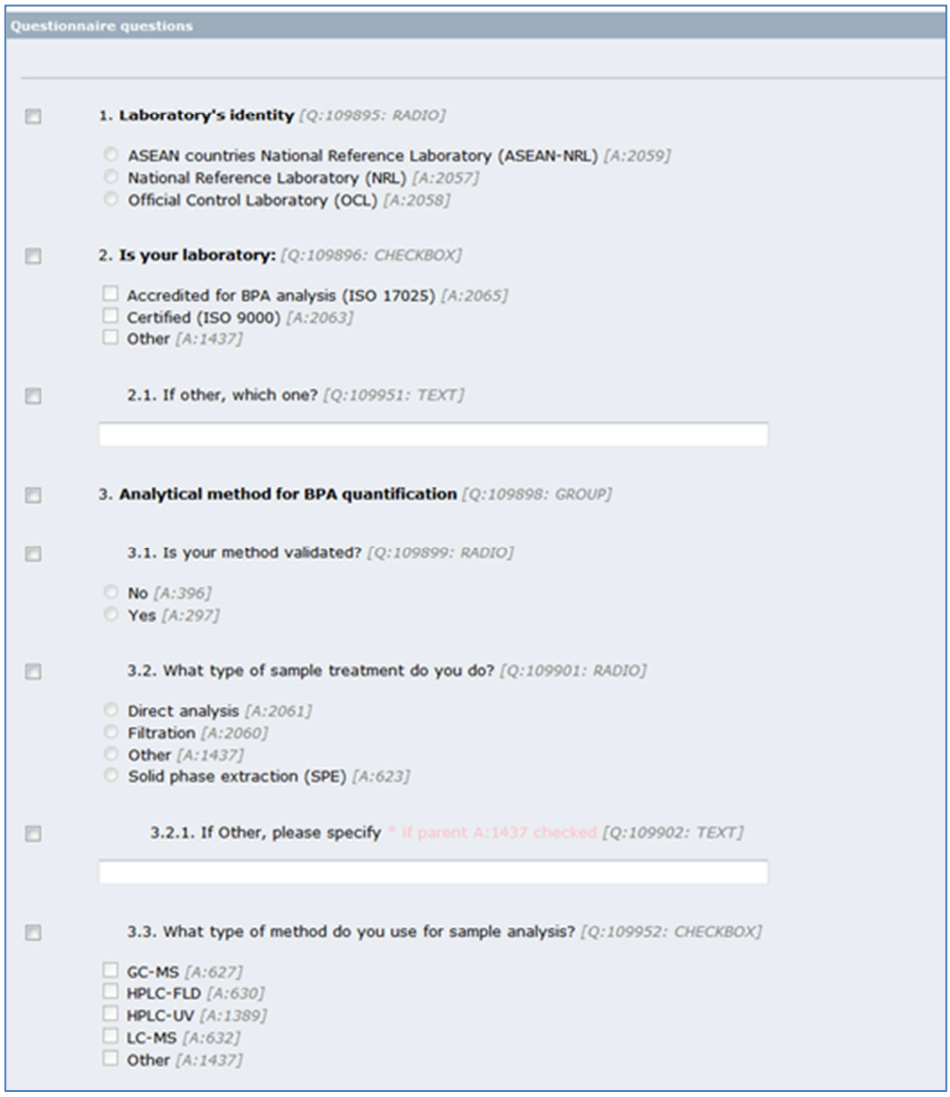
Signature _____

Thank you for returning this form by email to:

Dr. S. Valzacchi
EURL-FCM ILC01-2017 Coordinator
E-mail : JRC-EURL-FCM@ec.europa.eu

Via Enrico Fermi 2749, TP260, 21027 Ispra (VA) – Italy Tel.: +39 033278 5293.
https://ec.europa.eu/jrc/en/eurl/food-contact-materials ; E-mail: JRC-EURL-FCM@ec.europa.eu

Annex 4. Questionnaire



Questionnaire questions

1. **Laboratory's identity** *[Q:109895: RADIO]*

- ASEAN countries National Reference Laboratory (ASEAN-NRL) *[A:2059]*
- National Reference Laboratory (NRL) *[A:2057]*
- Official Control Laboratory (OCL) *[A:2058]*

2. **Is your laboratory:** *[Q:109896: CHECKBOX]*

- Accredited for BPA analysis (ISO 17025) *[A:2065]*
- Certified (ISO 9000) *[A:2063]*
- Other *[A:1437]*

2.1. If other, which one? *[Q:109951: TEXT]*

3. **Analytical method for BPA quantification** *[Q:109898: GROUP]*

3.1. Is your method validated? *[Q:109899: RADIO]*

- No *[A:396]*
- Yes *[A:297]*

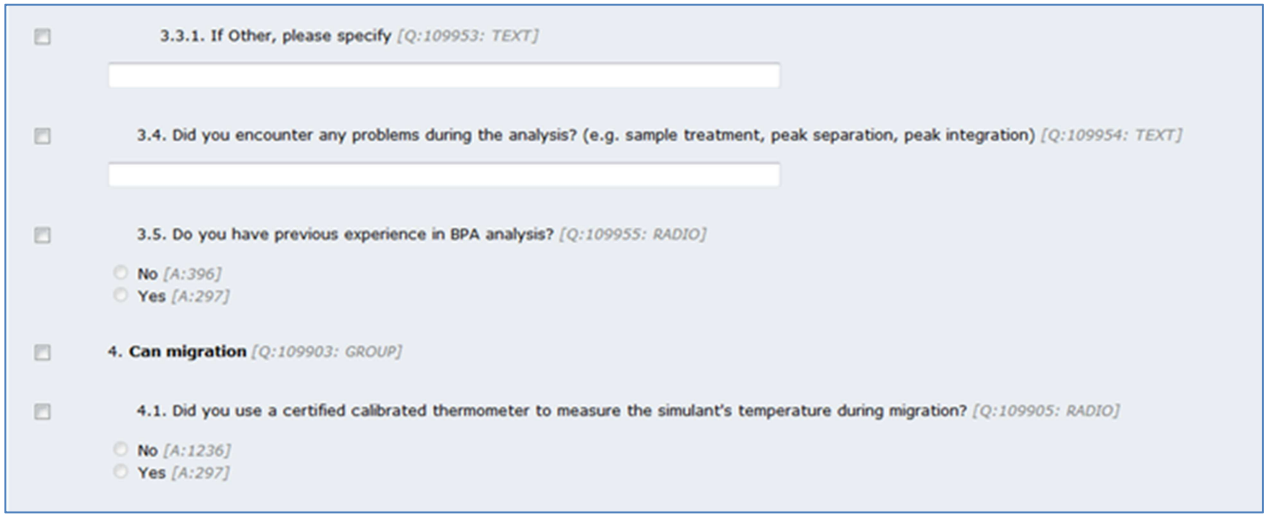
3.2. What type of sample treatment do you do? *[Q:109901: RADIO]*

- Direct analysis *[A:2061]*
- Filtration *[A:2060]*
- Other *[A:1437]*
- Solid phase extraction (SPE) *[A:623]*

3.2.1. If Other, please specify * if parent A:1437 checked *[Q:109902: TEXT]*

3.3. What type of method do you use for sample analysis? *[Q:109952: CHECKBOX]*

- GC-MS *[A:627]*
- HPLC-FLD *[A:630]*
- HPLC-UV *[A:1389]*
- LC-MS *[A:632]*
- Other *[A:1437]*



3.3.1. If Other, please specify *[Q:109953: TEXT]*

3.4. Did you encounter any problems during the analysis? (e.g. sample treatment, peak separation, peak integration) *[Q:109954: TEXT]*

3.5. Do you have previous experience in BPA analysis? *[Q:109955: RADIO]*

- No *[A:396]*
- Yes *[A:297]*

4. **Can migration** *[Q:109903: GROUP]*

4.1. Did you use a certified calibrated thermometer to measure the simulant's temperature during migration? *[Q:109905: RADIO]*

- No *[A:1236]*
- Yes *[A:297]*

4.2. Temperature control *[Q:109906: CUSTOM TABLE]*

Temperature Control

Please, insert the simulant's registered temperature every 5 minutes.

Questions/Response table	T simulant (ºC)
t (min) 5	
10	
15	
20	
25	
30	
35	
40	
45	
50	
55	
60	
65	
70	
75	
80	
85	
90	
95	
100	
105	
110	
115	
120	

4.3. Did you encounter any problems during the migration process? *[Q:109957: TEXT]*

4.4. Please report the loss of food simulant (if any) in mL after migration. *[Q:109958: TEXT]*

Annex 5. Homogeneity study

	Cans	**Solution 1**		**Solution 2**	
Sample ID	R1	R1	R2	R1	R2
1	0.042	0.086	0.088	0,038	0,036
2	0.045	0.085	0.089	0,036	0,039
3	0.038	0.083	0.087	0,038	0,038
4	0.045	0.087	0.087	0,038	0,036
5	0.048	0.084	0.086	0,038	0,037
6	0.044	0.089	0.090	0,038	0,037
7	0.048	0.086	0.089	0,036	0,037
8	0.046	0.088	0.089	0,038	0,037
9	0.044	0.086	0.087	0,035	0,036
10	0.043	0.088	0.087	0,038	0,035
11	-	0.089	0.089	0,036	0,036
12	-	0.087	0.088	0,038	0,035
Mean	0.044	0.0872		0.0370	
S_x	0.0029	0.0014		0.0006	
S_w	-	0.0015		0.0012	
S_s	-	0.0009		0	
u_{hom}	0.0029	0.0009		0.0005	
σ_{pt}	0.01064	0.013		0.0054	
$0.3*\sigma_{pt}$	0.003	0.004		0.0016	
$S_s \leq 0.3*\sigma_{pt}$	**passed**	**passed**		**passed**	

(all values expressed in mg kg^{-1})

σ_{pt} : standard deviation for PT assessment;
s_x : standard deviation of the sample averages;
s_w : within-sample standard deviation;
s_s : between-sample standard deviation.

Annex 6. Stability study

At 20, 40 and 60 °C, for 1, 4 and 16 weeks; all values expressed in mg kg^{-1}

temperature	**20 °C**	**20 °C**	**20 °C**	**40 °C**	**40 °C**	**60 °C**	Significant
time	0	**4 w**	**16 w**	**4 w**	**16 w**	**1 w**	Slope? (#)
Cans	0.044 *	0.044	0.044	0.048	0.044	0.049	
		0.051	0.043	0.047	0.046	0.047	No
		0.043	0.046	0.050	0.044	0.046	
		0.041	0.041	0.043	0.043	0.045	Stable
Sol.1	0.087 *	0.086	0.083	0.085	0.087	0.083	
		0.085	0.083	0.085	0.087	0.086	No
		0.084	0.084	0.086	0.087	0.083	
		0.084	0.085	0.085	0.087	0.084	Stable
Sol.2	0.037 *	0.035	0.036	0.036	0.036	0.037	
		0.036	0.036	0.036	0.037	0.037	No
		0.036	0.036	0.037	0.037	0.037	
		0.036	0.037	0.037	0.037	0.037	Stable

*** Homogeneity data**

(#) Is the slope of the linear regression significantly different from "0" at a 95 % level?

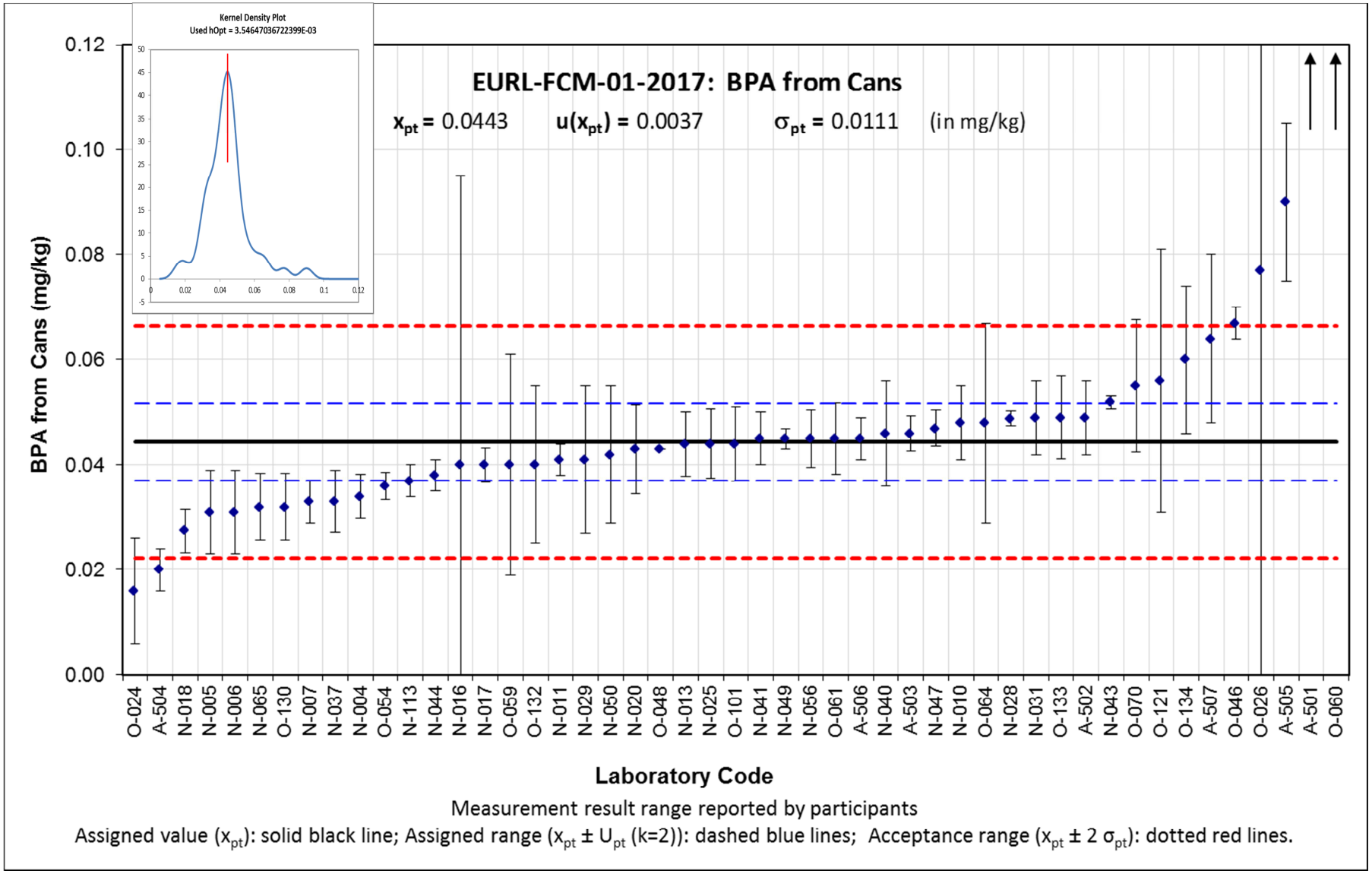
Annex 7. Results for migration of **BPA from cans**

Assigned range: x_{pt} = 0.0443; $u(x_{pt})$ = 0.0037; σ_{pt} = 0.0111 (* values in mg kg^{-1})

Lab Code	x_i (*)	± (*)	k	method	u_i	z	zeta	unc.
N-004	0.034	0.0042	2	HPLC-FLD	0.002	-0.93	-2.45	b
N-005	0.031	0.008	2	HPLC-FLD	0.004	-1.20	-2.46	a
N-006	0.031	0.008	2	HPLC-FLD	0.004	-1.20	-2.46	a
N-007	0.033	0.004	2	HPLC-FLD	0.002	-1.02	-2.72	b
N-010	0.048	0.007	2	HPLC-FLD	0.004	0.33	0.72	b
N-011	0.041	0.003	2	HPLC-FLD	0.002	-0.30	-0.85	b
N-013	0.044	0.0061	2	HPLC-FLD	0.003	-0.03	-0.07	b
N-016	0.040	0.055	2	HPLC-FLD	0.028	-0.39	-0.16	c
N-017	0.040	0.0032	2	HPLC-FLD	0.002	-0.39	-1.09	b
N-018	0.0274	0.0041	1.73	UPLC-MS/MS	0.002	-1.53	-3.89	b
N-020	0.043	0.0084	2	HPLC-FLD	0.004	-0.12	-0.24	a
O-024	0.016	0.01	2	HPLC-FLD	0.005	-2.56	-4.57	a
N-025	0.044	0.0066	2	HPLC-FLD	0.003	-0.03	-0.07	b
O-026	0.077	0.1	2	LC-MS/MS	0.050	2.94	0.65	c
N-028	0.0488	0.0014	2	LC-MS/MS	0.001	0.40	1.19	b
N-029	0.041	0.014	3.18	HPLC-FLD	0.004	-0.30	-0.58	a
N-031	0.049	0.007	2	HPLC-FLD	0.004	0.42	0.92	b
N-037	0.033	0.0059	2	HPLC-FLD	0.003	-1.02	-2.41	b
N-040	0.046	0.01	2	LC-MS	0.005	0.15	0.27	a
N-041	0.045	0.005	2	LC-MS	0.003	0.06	0.15	b
N-043	0.0519	0.0012	2	LC-MS/MS	0.001	0.68	2.04	b
N-044	0.038	0.003	1.73	LC-TOF/MS	0.002	-0.57	-1.57	b
O-046	0.067	0.003	1.73	HPLC-FLD	0.002	2.04	5.59	b
N-047	0.047	0.0034	2	LC-MS/MS	0.002	0.24	0.66	b
O-048	0.043			LC-MS	0.000	-0.12	-0.37	b
N-049	0.045	0.002	1.73	HPLC-FLD	0.001	0.06	0.17	b
N-050	0.042	0.013	2	GC-MS	0.007	-0.21	-0.31	a
O-054	0.036	0.0026	1.96	LC-MS	0.001	-0.75	-2.14	b
N-056	0.045	0.0054	2	HPLC-FLD	0.003	0.06	0.14	b
O-059	0.040	0.021	2	HPLC-FLD	0.011	-0.39	-0.39	a
O-060	0.410	0.04	2	HPLC-FLD	0.020	32.98	17.98	c
O-061	0.045	0.0068	2	LC-MS	0.003	0.06	0.13	b
O-064	0.048	0.019	2	LC-MS	0.010	0.33	0.36	a
N-065	0.032	0.0064	2	HPLC-FLD	0.003	-1.11	-2.54	b
O-070	0.055	0.0126	2	HPLC-FLD	0.006	0.96	1.46	a
O-101	0.044	0.007	2	HPLC-FLD	0.004	-0.03	-0.07	b
N-113	0.037	0.003	2	HPLC-FLD	0.002	-0.66	-1.86	b
O-121	0.056	0.025	2	HPLC-UV	0.013	1.05	0.89	c
O-130	0.032	0.0064	2	HPLC-UV	0.003	-1.11	-2.54	b
O-132	0.040	0.015	2	HPLC-FLD	0.008	-0.39	-0.52	a
O-133	0.049	0.0079	2	HPLC-FLD	0.004	0.42	0.86	a
O-134	0.060	0.014	1.96	LC-MS	0.007	1.41	1.95	a
A-501	0.390	0.08	2	LC-MS	0.040	31.18	8.61	c
A-502	0.049	0.007	2	HPLC-FLD	0.004	0.42	0.92	b
A-503	0.046	0.0033	2	LC-MS	0.002	0.15	0.41	b
A-504	0.020	0.004	2	HPLC-FLD	0.002	-2.20	-5.84	b
A-505	0.090	0.0151	2	HPLC-UV	0.008	4.12	5.44	a
A-506	0.045	0.004	2	HPLC-FLD	0.002	0.06	0.16	b
A-507	0.064	0.016	2	HPLC-FLD	0.008	1.77	2.23	a

[#] performance: satisfactory, questionable, unsatisfactory

[@] uncertainty; a: $u(x_{pt}) \leq u_i \leq \sigma_{pt}$; b: $u_i < u(x_{pt})$; c: $u_i > \sigma_{pt}$



Measurement result range reported by participants

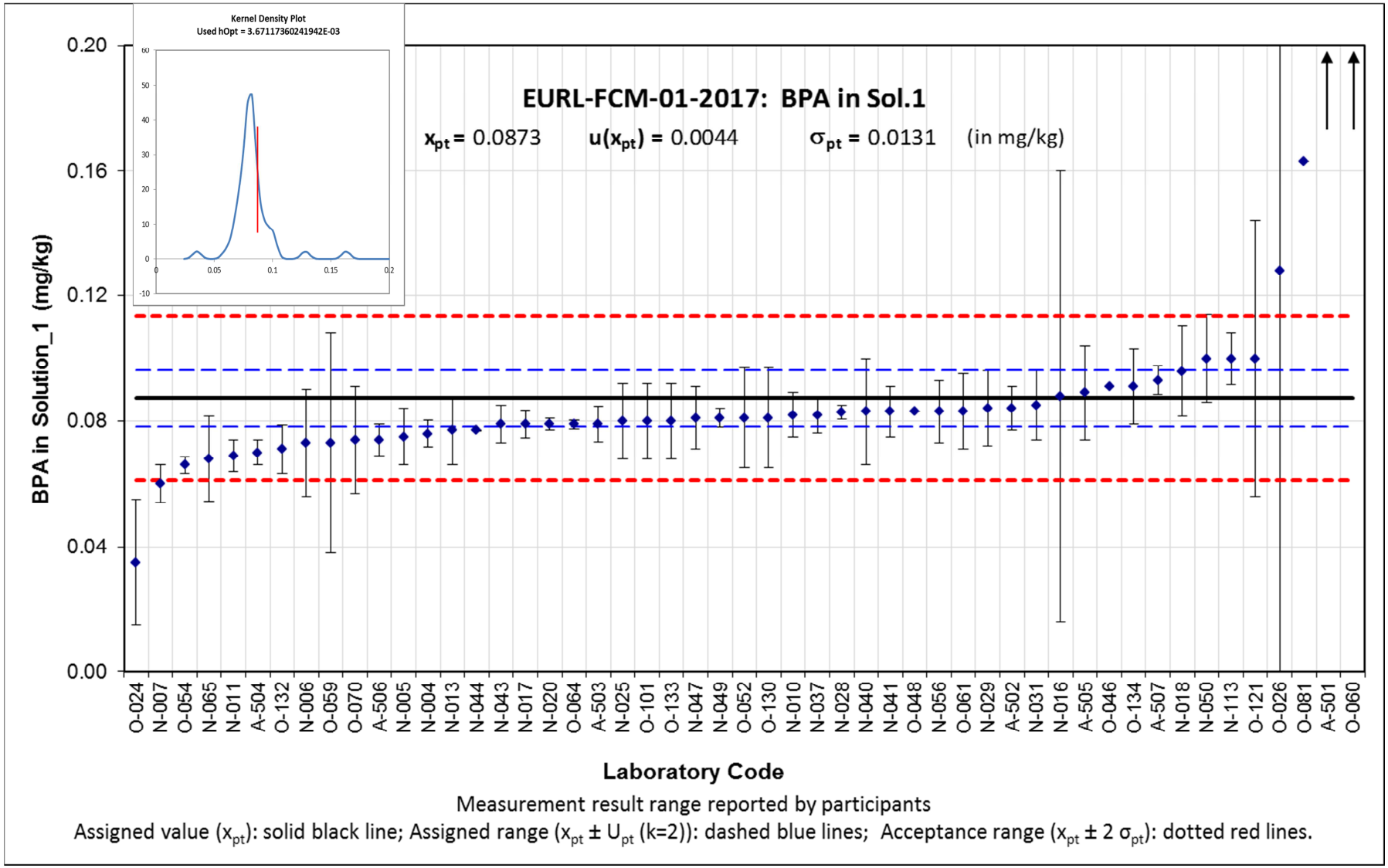
Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 8. Results for **BPA in Solution 1**

Assigned range: x_{pt} = 0.0873; $u(x_{pt})$ = 0.0044; σ_{pt} = 0.0131 (* values in mg kg^{-1})

Lab Code	x_i (*)	± (*)	k	method	u_i	z	zeta	unc.
N-004	0.076	0.0042	2	HPLC-FLD	0.002	-0.86	-2.29	b
N-005	0.075	0.009	2	HPLC-FLD	0.005	-0.94	-1.94	a
N-006	0.073	0.017	2	HPLC-FLD	0.009	-1.09	-1.49	a
N-007	0.06	0.006	2	HPLC-FLD	0.003	-2.08	-5.08	b
N-010	0.082	0.007	2	HPLC-FLD	0.004	-0.40	-0.93	b
N-011	0.069	0.005	2	HPLC-FLD	0.003	-1.39	-3.58	b
N-013	0.077	0.011	2	HPLC-FLD	0.006	-0.78	-1.45	a
N-016	0.088	0.072	2	HPLC-FLD	0.036	0.06	0.02	c
N-017	0.079	0.0043	2	HPLC-FLD	0.002	-0.63	-1.67	b
N-018	0.0958	0.0144	1.73	UPLC-MS/MS	0.008	0.65	0.91	a
N-020	0.079	0.0018	2	HPLC-FLD	0.001	-0.63	-1.82	b
O-024	0.035	0.02	2	HPLC-FLD	0.010	-3.99	-4.77	a
N-025	0.08	0.012	2	HPLC-FLD	0.006	-0.55	-0.97	a
O-026	0.128	0.166	2	LC-MS/MS	0.083	3.11	0.49	c
N-028	0.0829	0.0022	2	LC-MS/MS	0.001	-0.33	-0.95	b
N-029	0.084	0.012	3.18	HPLC-FLD	0.004	-0.25	-0.56	b
N-031	0.085	0.011	2	HPLC-FLD	0.006	-0.17	-0.32	a
N-037	0.082	0.0057	2	HPLC-FLD	0.003	-0.40	-0.99	b
N-040	0.083	0.017	2	LC-MS	0.009	-0.32	-0.44	a
N-041	0.083	0.008	2	LC-MS	0.004	-0.32	-0.71	b
N-043	0.0789	0.006	2	LC-MS/MS	0.003	-0.64	-1.56	b
N-044	0.077	0.0002	1.73	LC-TOF/MS	0.000	-0.78	-2.30	b
O-046	0.091			HPLC-FLD	0.000	0.29	0.84	b
N-047	0.081	0.01	2	LC-MS/MS	0.005	-0.48	-0.93	a
O-048	0.083			LC-MS	0.000	-0.32	-0.96	b
N-049	0.081	0.003	1.73	HPLC-FLD	0.002	-0.48	-1.31	b
N-050	0.100	0.014	2	GC-MS	0.007	0.97	1.54	a
O-052	0.081	0.016	1.73		0.009	-0.48	-0.61	a
O-054	0.066	0.0026	1.96	LC-MS	0.001	-1.62	-4.58	b
N-056	0.083	0.0099	2	HPLC-FLD	0.005	-0.32	-0.64	a
O-059	0.073	0.035	2	HPLC-FLD	0.018	-1.09	-0.79	c
O-060	0.940	0.09	2	HPLC-FLD	0.045	65.16	18.86	c
O-061	0.083	0.012	2	LC-MS	0.006	-0.32	-0.57	a
O-064	0.079	0.0014	2	LC-MS	0.001	-0.63	-1.83	b
N-065	0.068	0.0136	2	HPLC-FLD	0.007	-1.47	-2.37	a
O-070	0.074	0.0171	2	HPLC-FLD	0.009	-1.01	-1.37	a
O-081	0.163				0.000	5.79	17.03	b
O-101	0.080	0.012	2	HPLC-FLD	0.006	-0.55	-0.97	a
N-113	0.100	0.0082	2	HPLC-FLD	0.004	0.97	2.11	b
O-121	0.100	0.044	2	HPLC-UV	0.022	0.97	0.57	c
O-130	0.081	0.016	2	HPLC-UV	0.008	-0.48	-0.68	a
O-132	0.071	0.0076	2	HPLC-FLD	0.004	-1.24	-2.78	b
O-133	0.080	0.012	2	HPLC-FLD	0.006	-0.55	-0.97	a
O-134	0.091	0.012	1.96	LC-MS	0.006	0.29	0.50	a
A-501	0.400	0.04	2	LC-MS	0.020	23.90	15.26	c
A-502	0.084	0.007	2	HPLC-FLD	0.004	-0.25	-0.57	b
A-503	0.079	0.0056	2	LC-MS	0.003	-0.63	-1.57	b
A-504	0.070	0.004	2	HPLC-FLD	0.002	-1.32	-3.54	b
A-505	0.089	0.015	2	HPLC-UV	0.008	0.13	0.20	a
A-506	0.074	0.005	2	HPLC-FLD	0.003	-1.01	-2.60	b
A-507	0.093	0.0046	2	HPLC-FLD	0.002	0.44	1.15	b

[#] performance: satisfactory, questionable, unsatisfactory

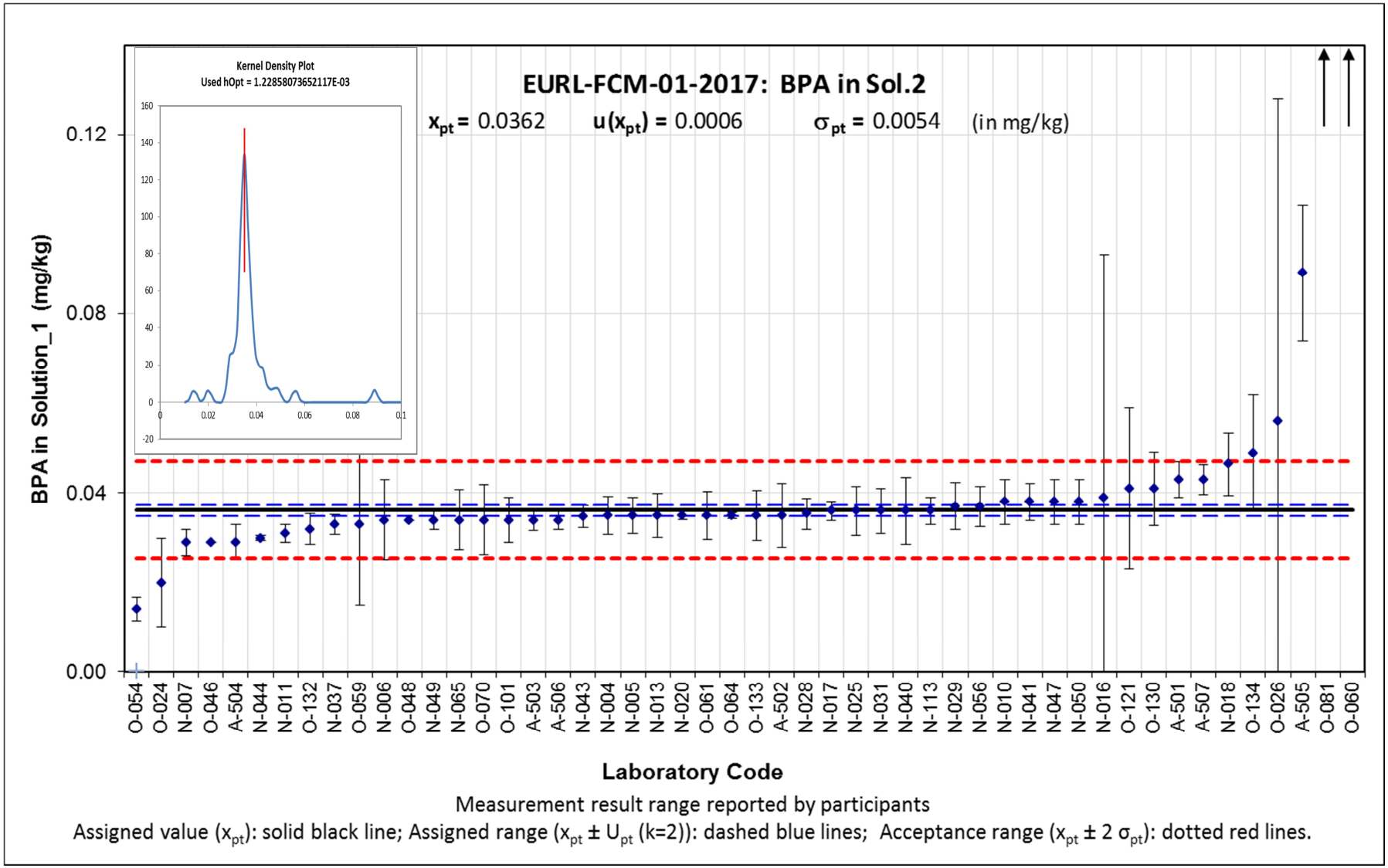

Kernel Density Plot
Used hOpt = 3.67117360241942E-03
EURL-FCM-01-2017: BPA in Sol.1
x_{pt} = 0.0873 $u(x_{pt})$ = 0.0044 σ_{pt} = 0.0131 (in mg/kg)
BPA in Solution_1 (mg/kg)
0.20
0.16
0.12
0.08
0.04
0.00
O-024 N-007 O-054 N-065 N-011 A-504 O-132 N-006 O-059 O-070 A-506 N-005 N-004 N-013 N-044 N-043 N-017 N-020 O-064 A-503 N-025 O-101 O-133 N-047 N-049 O-052 O-130 N-010 N-037 N-028 N-040 N-041 O-048 N-056 O-061 N-029 A-502 N-031 N-016 A-505 O-046 O-134 A-507 N-018 N-050 N-113 O-121 O-026 O-081 A-501 O-060
Laboratory Code
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 **BPA in Solution 2**

Assigned range: x_{pt} = 0.0362; $u(x_{pt})$ = 0.0006; σ_{pt} = 0.0054 (all values in mg kg^{-1})

Lab Code	x_i (*)	± (*)	k	method	u_i	z	zeta	unc.
N-004	0.035	0.0042	2	HPLC-FLD	0.002	-0.21	-0.53	a
N-005	0.035	0.004	2	HPLC-FLD	0.002	-0.21	-0.56	a
N-006	0.034	0.009	2	HPLC-FLD	0.005	-0.40	-0.48	a
N-007	0.029	0.003	2	HPLC-FLD	0.002	-1.32	-4.45	a
N-010	0.038	0.005	2	HPLC-FLD	0.003	0.34	0.72	a
N-011	0.031	0.002	2	HPLC-FLD	0.001	-0.95	-4.46	a
N-013	0.035	0.0049	2	HPLC-FLD	0.002	-0.21	-0.46	a
N-016	0.039	0.054	2	HPLC-FLD	0.027	0.52	0.11	c
N-017	0.036	0.002	2	HPLC-FLD	0.001	-0.03	-0.14	a
N-018	0.0464	0.007	1.73	UPLC-MS/MS	0.004	1.89	2.50	a
N-020	0.035	0.00093	2	HPLC-FLD	0.000	-0.21	-1.56	b
O-024	0.02	0.01	2	HPLC-FLD	0.005	-2.98	-3.21	a
N-025	0.036	0.0054	2	HPLC-FLD	0.003	-0.03	-0.06	a
O-026	0.056	0.072	2	LC-MS/MS	0.036	3.66	0.55	c
N-028	0.0354	0.0034	2	LC-MS/MS	0.002	-0.14	-0.42	a
N-029	0.037	0.0052	3.18	HPLC-FLD	0.002	0.15	0.48	a
N-031	0.036	0.005	2	HPLC-FLD	0.003	-0.03	-0.06	a
N-037	0.033	0.0023	2	HPLC-FLD	0.001	-0.58	-2.45	a
N-040	0.036	0.0073	2	LC-MS	0.004	-0.03	-0.04	a
N-041	0.038	0.004	2	LC-MS	0.002	0.34	0.88	a
N-043	0.0347	0.0024	2	LC-MS/MS	0.001	-0.27	-1.09	a
N-044	0.03	0.0006	1.73	LC-TOF/MS	0.000	-1.14	-9.09	b
O-046	0.029			HPLC-FLD	0.000	-1.32	-12.29	b
N-047	0.038	0.0049	2	LC-MS/MS	0.002	0.34	0.73	a
O-048	0.034			LC-MS	0.000	-0.40	-3.71	b
N-049	0.034	0.002	1.73	HPLC-FLD	0.001	-0.40	-1.67	a
N-050	0.038	0.005	2	GC-MS	0.003	0.34	0.72	a
O-054	0.014	0.0026	1.96	LC-MS	0.001	-4.09	-15.30	a
N-056	0.037	0.0044	2	HPLC-FLD	0.002	0.15	0.37	a
O-059	0.033	0.018	2	HPLC-FLD	0.009	-0.58	-0.35	c
O-060	0.400	0.04	2	HPLC-FLD	0.020	67.08	18.18	c
O-061	0.035	0.0053	2	LC-MS	0.003	-0.21	-0.43	a
O-064	0.035	0.0006	2	LC-MS	0.000	-0.21	-1.77	b
N-065	0.034	0.0068	2	HPLC-FLD	0.003	-0.40	-0.63	a
O-070	0.034	0.0079	2	HPLC-FLD	0.004	-0.40	-0.54	a
O-081	0.276				0.000	44.22	411.79	b
O-101	0.034	0.005	2	HPLC-FLD	0.003	-0.40	-0.84	a
N-113	0.036	0.0029	2	HPLC-FLD	0.001	-0.03	-0.10	a
O-121	0.041	0.018	2	HPLC-UV	0.009	0.89	0.54	c
O-130	0.041	0.0082	2	HPLC-UV	0.004	0.89	1.17	a
O-132	0.032	0.0034	2	HPLC-FLD	0.002	-0.77	-2.31	a
O-133	0.035	0.0055	2	HPLC-FLD	0.003	-0.21	-0.41	a
O-134	0.049	0.013	1.96	LC-MS	0.007	2.37	1.93	c
A-501	0.043	0.004	2	LC-MS	0.002	1.26	3.28	a
A-502	0.035	0.007	2	HPLC-FLD	0.004	-0.21	-0.33	a
A-503	0.034	0.0024	2	LC-MS	0.001	-0.40	-1.62	a
A-504	0.029	0.004	2	HPLC-FLD	0.002	-1.32	-3.44	a
A-505	0.089	0.0151	2	HPLC-UV	0.008	9.74	6.98	c
A-506	0.034	0.002	2	HPLC-FLD	0.001	-0.40	-1.87	a
A-507	0.043	0.0033	2	HPLC-FLD	0.002	1.26	3.91	a

[#] performance: satisfactory, questionable, unsatisfactory



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. Temperature of the simulant and volume of simulant loss in migration from cans

Lab	T mean (°C)	T stdev (°C)	Certified thermometer	Simulant loss (mL)
LC0004	70.5	0.5	Y	15
LC0005	69.2	0.5	Y	20
LC0006	71.8	0.1	Y	43
LC0007	70.0	0.8	N	20
LC0010	70.6	0.2	N	15
LC0011	68.7	0.9	N	13
LC0013	69.5	0.5	Y	13
LC0016	64.8	0.8	Y	0
LC0017	69.7	0.1	Y	10
LC0018	69.7	0.9	Y	15
LC0020	69.4	0.3	Y	15
LC0024	69.9	0.5	Y	0
LC0025	70.3	0.5	Y	0
LC0026	69.9	0.8	Y	nd
LC0028	69.5	1.1	Y	3
LC0029	70.6	0.7	Y	5
LC0031	68.5	0.2	Y	0
LC0037	68.5	0.8	N	0
LC0040	69.5	1.0	Y	19
LC0041	62.7	1.2	Y	10
LC0043	69.0	1.0	Y	3
LC0044	69.3	0.4	N	0
LC0046	71.0	0.6	Y	5
LC0047	69.5	0.7	Y	14
LC0048	70.6	0.5	N	5
LC0049	70.8	0.7	Y	25
LC0050	70.7	1.2	Y	0
LC0052	nd			
LC0054	68.7	0.3	Y	4
LC0056	69.9	0.8	Y	30
LC0059	71.3	0.1	Y	20
LC0060	69.5	0.0	N	0
LC0061	68.4	0.3	Y	0
LC0064	69.6	0.5	Y	13
LC0065	65.2	3.8	Y	0
LC0070	68.9	0.4	Y	10
LC0081	nd			
LC0101	70.2	0.2	Y	0
LC0113	67.2	0.5	Y	13
LC0121	69.7	0.2	Y	35
LC0130	70.0	1.0	Y	15
LC0132	67.5	4.2	Y	0
LC0133	69.1	2.0	Y	20
LC0134	69.6	2.2	N	42
LC0501	70.1	0.5	Y	0
LC0502	69.7	0.6	Y	0
LC0503	64.8	3.1	Y	22
LC0504	59.9	1.1	N	20
LC0505	67.7	0.5	Y	30
LC0506	68.0	0.0	Y	0
LC0507	71.0	0.7	N	5

nd: not determined

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