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# Determination of melamine and formaldehyde (FA) in food contact material migration solutions.

*FCM-23/01 Proficiency Test Report*

BELAC 268-PT

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# Contents

## Abstract

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM) organised a proficiency testing round (FCM-23/01) for the determination of the mass fraction of melamine and formaldehyde in food simulant B solutions, in support to Commission Regulation (EU) 2020/1245. This proficiency testing exercise was open to EU National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs). The EURL dispatched two well-characterised test items for analysis, namely (i) a food simulant solution B spiked with the specified substances, and (ii) four bamboo/melamine mugs for the migration tests. Twenty-five NRLs from 24 countries, and 18 OCLs (from Belgium, Germany, Italy and Spain) reported results. Analysing the spiked solution proved relatively straightforward, with over 95 % of the participants reporting satisfactory results. However, the compliance assessment of the mugs was more challenging; only 6 laboratories assessed correctly the compliance of test item 2 for both melamine and formaldehyde in line with the observations of the EURL, while several others presented compliance statements in line with the EURL but inconsistent with their own experimental results. This report provides a detailed discussion about the outcomes of this proficiency testing exercise.

## Acknowledgements

The forty-three laboratories listed hereafter are kindly acknowledged for their participation in the PT.

| Organisation | Country |
|---|---|
| AGES, Gebrauchsgegenstände | Austria |
| Federal agency for the safety of the food chain (AFSCA-FAVV) | Belgium |
| Sciensano | Belgium |
| National Center of Public Health and Analyses (NCPHA) | Bulgaria |
| Croatian Institute of Public Health (HZJZ) | Croatia |
| State General Laboratory (SGL) | Cyprus |
| National Institute of Public Health (SZÚ) | Czech Republic |
| Technical University of Denmark (TUD) | Denmark |
| Danish Veterinary and Food Administration (DVFA) | Denmark |
| Finnish Customs (TULLI) | Finland |
| Laboratoire National de Metrologie et d'essais (LNE) | France |
| Service Commun des Laboratoires (SCL), Laboratoire de Bordeaux | France |
| Chemisches und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe (CVUA-MEL) | Germany |
| Thüringer Landesamt für Verbraucherschutz (TLV) | Germany |
| Landesbetrieb Hessisches Landeslabor (LHL), Hessen | Germany |
| German Federal Institute for Risk Assessment (BfR) | Germany |
| Chemisches- und Veterinäruntersuchungsamt (CVUA) Stuttgart (chemical & veterinary office) | Germany |
| LAVES, Institut für Bedarfsgegenstände Lüneburg (IfB) Lüneburg | Germany |
| Landeslabor Schleswig-Holstein (LSH) | Germany |
| General Chemical State Laboratory (GCSL) | Greece |
| National Food Chain Safety Office (NEBIH), Food Chain Safety Laboratory Directorate | Hungary |
| The Public Analyst's Laboratory | Ireland |
| Agenzia di Tutela della Salute (ATS)-Milano | Italy |
| Istituto Zooprofilattico Sperimentale della Lombardia e dell'Emilia Romagna (IZSLER), Chemical Department Bologna | Italy |
| Agenzia Regionale per la Protezione Ambientale delle Marche (ARPAM), Laboratorio Chimico | Italy |
| USL TOSCANA CENTRO, LABORATORIO DI SANITA PUBBLICA | Italy |
| Agenzia regionale per la protezione dell'ambiente Friuli Venezia Giulia (ARPA FVG), Laboratorio di Udine | Italy |
| Agenzia di Tutela della Salute (ATS) Insubria, Laboratorio di Prevenzione | Italy |
| Istituto Superiore di Sanità (ISS), Dipartimento Ambiente e salute (DAMSA) | Italy |
| National Public Health Surveillance Laboratory (NPHSL) | Lithuania |
| Laboratoire National de Santé (LNS) | Luxembourg |
| Netherlands Food and Consumer Product Safety Authority (NVWA) Product Safety Laboratory | Netherlands |
| Escola Superior de Biotecnologia - Universidade Católica Portuguesa | Portugal |
| National Institute of Public Health (INSP) | Romania |
| Regional Public Health Authority | Slovakia |
| National Laboratory of Health, Environment and Food (NLZOH) | Slovenia |
| Centro Analitico de Inspección y Control de Calidad de Comercio Exterior. SOIVRE | Spain |
| Centro Nacional Alimentacion (CNA) | Spain |
| Laboratorio de Salud Pública – Madrid Salud | Spain |
| Laboratorio de Salud Pública de Alicante | Spain |
| AINIA Centro Tecnologico | Spain |
| Swedish Food Agency | Sweden |
| Official Food Control Authority of the Canton of Zürich | Switzerland |


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This report has been authorised by Ursula Vincent, Head of Unit JRC F.5.

## Executive summary

The European Union Reference Laboratory for Food contact materials (EURL-FCM) organised a proficiency test (FCM-23/01) for the determination of melamine and formaldehyde to support the Commission Regulation (EU) 2020/1245 amending Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food. This proficiency test was open to National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs).

Two test items were prepared and dispatched to participants for analysis. Test item 1 consisted of a solution of melamine and formaldehyde in food simulant B (acetic acid 3 % w/v in water), while test item 2 consisted of four bamboo/melamine mugs. The homogeneity and stability of both test items were evaluated by the EURL while the assigned values for test item 1 were derived from the gravimetrical preparation of the solution. No assigned values were needed for test item 2.

Twenty-five NRLs from 24 countries and 18 OCLs registered to this PT round and reported results. The results for test item 1 were rated using $z$ and zeta ($\zeta$) scores in accordance with ISO 13528:2022. Relative standard deviations for proficiency assessment ($\sigma_{pt}$) were set to 15 % of the respective assigned values for the two analytes.

More than 95 % of the participating laboratories performed satisfactorily (according to the z score) for the analysis of melamine and formaldehyde in the solution. These results confirm the competence of the participants in effectively monitoring melamine and formaldehyde in food simulant B.

The assessment of participants' compliance statements for test item 2 presented significant difficulties. Minor variations in migration test conditions, though within the tolerable range had a significant impact on the test results. Only 28 % (12 out of 43) of the laboratories accurately assessed the compliance and the stability of migration for formaldehyde in test item 2, in line with the EURL conclusion. The assessment of melamine stability of migration and compliance with the specific migration limit (SML) for test item 2 emerged as notably arduous with only 18 % (7 out of 39) of the laboratories correctly assessing compliance and stability for melamine in test item 2, aligning with the EURL conclusions.

In conclusion, assessing the compliance of melamine-bamboo mugs remains challenging and requires further investigations.

# 1 Introduction

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM), hosted by the Joint Research Centre of the European Commission, organised a proficiency test (PT) for the determination of melamine and formaldehyde in food simulant B solution and their migration from melamine-bamboo ware to support the Commission Regulation (EU) 2020/1245 [1].

This PT was agreed upon with the Directorate General for Health and Food Safety (DG SANTE) as part of the EURL-FCM annual work programme 2023, thus complying with the mandate set in Regulation (EU) 2017/625 [2]. The PT was open to National Reference Laboratories (NRLs) and to Official Control Laboratories (OCLs) willing to participate.

This report summarises the outcome of the PT.

# 2 Scope

The present PT aims to assess the performance of NRLs and OCLs in the determination of melamine and formaldehyde in food simulant B and their migration from melamine-bamboo ware. The PT was mandatory for the NRLs active in the field of FCM, and open to OCLs. Participants were also asked to assess the compliance of the melamine-bamboo mugs (test item 2) with the specifications outlined in Regulations (EU) 2020/1245 [1].

This PT, organised in line with ISO 17043:2023 [3], is identified as "FCM-23/01".

# 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 [4] (certificate number BELAC 268-TEST)
- ISO/IEC 17043:2023 [3] (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 [3] 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 organisation of the PT FCM-23/01 exercise was announced during the EURL-FCM plenary meeting on October 12, 2022, and an additional email was sent in May 2023 (Annex 1a). An invitation letter was then sent to NRLs and OCLs on May 17, 2023 (Annex 1b). The registration deadline was set to June 12, 2023. Samples were sent to participants on June 21, 2023. The deadline for reporting of results was first set to August 25, 2023 (Annex 2), and later extended until September 6, 2023 upon request of some participants.

## 3.4 Distribution

Each participant received:

- One vial containing 20 mL of food simulant B (acetic acid 3 % w/v) solution spiked with melamine and formaldehyde (test item 1); and
- Four melamine-bamboo mugs (test item 2) for migration test of melamine and formaldehyde in food simulant B.

Samples were sent under normal transport conditions at ambient temperature.

## 3.5 Instructions to participants

Detailed instructions were given to participants in the "Instruction letter" (Annex 2). Participants were requested to inspect the vial and the mugs for damages upon arrival. They were also instructed to store test item 1 in the refrigerator at 4 °C ± 2 °C in the dark upon reception and until analysis, to minimise the risk of contamination and ensure stability. Analyses were to be conducted soon after reception.

For test item 1, participants were requested to determine the "**mass fraction of melamine and formaldehyde in the food simulant B solution**" and subsequently report the results and the corresponding expanded measurement uncertainties **in mg kg$^{-1}$**. This calculation was to be performed assuming a density of 1 kg m$^{-3}$ for the food simulant B solution. Results were to be reported using the same number of significant figures normally reported to customers.

Similarly, participants were requested:

- to treat test item 2 as routine samples;
- to perform three consecutive migration experiments (m1, m2 and m3) at 70 ± 2 °C for 120 ± 5 minutes, on three different mugs (MUG1, MUG2 and MUG3), following their routine procedures;
- to determine the "mass fraction of melamine and formaldehyde" migrated from the mugs into the food simulant B solution, and to assess the compliance of the mugs with the specifications outlined in Commission Regulation (EU) 2020/1245 [1].

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 3).

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

# 4 Test items

## 4.1 Preparation

Test item 1 consisted of a food simulant B solution spiked with melamine and formaldehyde.

Formaldehyde stock solution (LRAD2648 CRM 998 mg/L) was purchased from Sigma Aldrich, while the melamine stock solution was prepared by weighing ca. 50 mg of pure standard (Sigma Aldrich MKCP8204) in 100 mL volumetric flasks and filled to the mark with water. The bulk solution of test item 1 was prepared by transferring ca. 25 mL of formaldehyde and 10 mL of melamine stock solutions into a 2 L volumetric flask, subsequently filled to the mark with food simulant B solution. Individual test items were prepared by transferring 20 mL of the bulk solution into 22 mL glass vials, resulting in a total of 100 vials produced. Each vial was uniquely identified with a number and the PT identifier. The mass fractions of formaldehyde and melamine in both solutions were determined as described in Section 5.1.

A total of 250 melamine/bamboo mugs were purchased from a local market. Each participant received a set of four mugs (test item 2) to conduct the migration tests.

## 4.2 Homogeneity and stability

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

The melamine in food simulant B (acetic acid 3 % w/v) solution was determined by high performance liquid chromatography (HPLC) using UV detection at 220 nm. The formaldehyde analysis involved mixing food simulant B with 2,4-dinitrophenylhydrazine (DNPH) to form the corresponding hydrazine, followed by HPLC-UV detection at 360 nm.

Homogeneity assessment was performed after the preparation of the test items and before distribution to participants.

For test item 1, ten vials were randomly selected and analysed in duplicate. Results were evaluated according to ISO 13528:2022 [5]. The solution proved to be adequately homogeneous for the investigated analytes (Annex 4.A). The contribution from homogeneity ($u_{hom}$) to the standard uncertainty of the assigned value ($u(x_{pt})$) was calculated using SoftCRM [6].

The homogeneity test played a critical role for test item 2, due to the difficulty in finding a large batch of commercial mugs that was sufficiently homogeneous. The homogeneity study was conducted for three consecutive migrations (m1, m2 and m3) at 70 ± 2 °C for 120 ± 5 min, using 10 randomly selected mugs, and focusing on both melamine and formaldehyde. Due to the "destructive" nature of the test, no replicate analyses were feasible. Sufficient homogeneity was proven based on the reasonable relative standard deviations (below 10 %) observed in the migrations and the analyses of the selected mugs (Annex 4.B).

Stability assessment was conducted at two points: (i) immediately after the preparation of the test items (time 0) and (ii) after the reporting of results.

Three additional samples of the solution (test item 1) were analysed in duplicate after the reporting deadline. Results were then compared to those obtained from

the homogeneity study. This stability study confirmed that test item 1 was adequately stable (i) at room temperature (20°C) over the entire PT period (13 weeks, from the value assignment until the deadline for reporting results); (ii) for 1 week at 40 °C (simulating extreme conditions which may occur during transport); and (iii) at the reference temperature of 4 °C. Hence, the uncertainty contribution due to stability was set to zero ($u_{st}$ = 0) for all the investigated analytes (Annex 4.C).

No stability study was conducted for test item 2, as the mugs were assumed to be stable with the time and thus also the migration of melamine and formaldehyde from them.

# 5 Assigned values and corresponding uncertainties

## 5.1 Assigned values

The assigned values ($x_{pt}$) for melamine and formaldehyde in test item 1 were derived from formulation (Table 1).

## 5.2 Associated uncertainties

The associated standard uncertainties of the assigned values ($u(x_{pt})$) for melamine and formaldehyde in test item 1 were calculated following the law of uncertainty propagation, combining the standard measurement uncertainty of the characterisation ($u_{char}$) with the standard uncertainty contributions from homogeneity ($u_{hom}$) and stability ($u_{st}$), in compliance with ISO 13528:2022 [5]:

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

The uncertainty $u_{char}$ was estimated from the formulation following the law of uncertainty propagation, combining quadratically the standard deviations of the input quantities as recommended by the Guide of expression of uncertainty in measurement (GUM) [7].

## 5.3 Metrological traceability of the assigned value

The assigned values were obtained by gravimetric preparation from Sigma-Aldrich's standards. The SI-traceable calibration of balances and a thorough control of the weighing procedure ensure traceability of the weighing itself.

## 5.4 Standard deviation for proficiency assessment, $\sigma_{pt}$

The relative standard deviations for proficiency assessment ($\sigma_{pt,rel}$) of melamine and formaldehyde in test item 1 were set to 15 % of the respective assigned values, based on expert judgment (Table 1).

**Table 1:** Assigned values ($x_{pt}$) and standard deviation for the PT assessment ($\sigma_{pt}$) for melamine and formaldehyde in test item 1 (T1). All values (excluding last column) are expressed in mg kg$^{-1}$.

| Measurand | $x_{pt}$ | $u_{char}$ | $u_{hom}$ | $u(x_{pt})$ $k = 1$ | $\sigma_{pt}$ ($\sigma_{pt,rel}$) | $u(x_{pt})/\sigma_{pt}$ |
|---|---|---|---|---|---|---|
| Melamine in T1 | **2.528** | 0.0058 | 0.0037 | **0.0069** | 0.379 (15 %) | 0.02 |
| Formaldehyde in T1 | **12.48** | 0.21 | 0.01 | **0.21** | 1.87 (15 %) | 0.11 |

### 5.5 Compliance statement for test item 2

Commission Regulation (EU) 2020/1245 introduced the requirement for a plastic repeated-use article to be stable during three consecutive migrations. According to the note on "Options for checking compliance of plastic food contact material articles for repeated use - An EURL-FCM harmonised approach (Ares(2022)8554518)" sent by the EURL to DG SANTE, the sample shall be considered non-compliant for the migration of a substance considering the standard measurement uncertainty if:

$m3 > SML$, or

$m2 > m1 \quad OR \quad m3 > m2$

These criteria shall be evaluated as follows:

| | | |
|---|---|---|
| Crit.1: | **IF** $(\boldsymbol{m3} - \boldsymbol{SML})/[(\boldsymbol{u}(\boldsymbol{m3})] > \mathbf{1.64}$ | **THEN** $\boldsymbol{m3} > \boldsymbol{SML}$ |
| Crit.2: | **IF** $(\boldsymbol{mj} - \boldsymbol{mi})/[(\boldsymbol{u}(\boldsymbol{mj}) + \boldsymbol{u}(\boldsymbol{mi})] > \mathbf{1.64}$ | **THEN** $\boldsymbol{mj} > \boldsymbol{mi}$ |

For a harmonised approach the standard measurement uncertainty (u) is calculated by the modified Horwitz method.

The plastic mugs investigated are designed for repeated-use with hot-fill applications. As per Regulation (EU) No 10/2011 [10], the migration of substances from the mugs should be tested at 70 °C for 2 hours. Furthermore, the normative Annex C of the EN 13130-1:2004 standard [11] defines tolerance ranges for both contact temperature and time, of 70 ± 2 ˚C and 120 ± 5 min, respectively.

In a series of three consecutive migrations (m1, m2 and m3) from the same mug, performed by the EURL-FCM, a repeatable temperature profile (profile 1) was observed in the food simulant B (Annex 7), falling within the specified tolerance limits. This consistency ensures the accuracy of the migration results.

Compliance with the SML and the stability of test item 2 were assessed based on the homogeneity results (Annex 4B). The third migration of formaldehyde into food simulant B was significantly below the SML of 15 mg $kg^{-1}$, and the consistency observed across the three migrated levels confirmed the stability of the mugs (Annex 10A). Consequently, test item 2 is considered to be **compliant for formaldehyde**. On the contrary, the third migration of melamine into food simulant B exceeded significantly the SML of 2.5 mg $kg^{-1}$. Despite exhibiting stable behaviour throughout the three migrations (Annex 9A), the test item 2 is considered **non-compliant for melamine.**

# 6 Evaluation of results

### 6.1 Scores and evaluation criteria

The individual laboratory performance was expressed in terms of z and $\zeta$ scores according to ISO 13528:2022 [5]:

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

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

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;

$\sigma_{pt}$ is the standard deviation for proficiency test assessment.

The interpretation of the *z* and *ζ* scores is done according ISO 13528:2022 [5]:

| | | |
|---|---|---|
| \|score\| ≤ 2 | satisfactory performance | (green in Annexes 5-6) |
| 2 < \|score\| < 3 | questionable performance | (yellow in Annexes 5-6) |
| \|score\| ≥ 3 | unsatisfactory performance | (red in Annexes 5-6) |

The *z* scores compare the participant's deviation from the assigned value with the standard deviation for proficiency test assessment ($\sigma_{pt}$) 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 √3, as recommended by Eurachem [8].

Uncertainty estimation is not trivial, therefore an additional assessment was provided to each laboratory reporting measurement uncertainty, indicating how reasonable their measurement uncertainty estimation has been. The relative standard measurement uncertainty was calculated based on the absolute values of the assigned values [$u_{rel}(x_{pt}) = 100*(u(x_{pt})/x_{pt})$] and of the reported values [$u_{rel}(x_i) = 100*(u(x_i)/x_i)$].

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": $u_{min,rel} \leq u_{rel}(x_i) \leq u_{max,rel}$). $u_{min,rel}$ is set to the standard uncertainties of the assigned values $u_{rel}(x_{pt})$. It is unlikely that a laboratory carrying out the analysis on a routine basis would determine the measurand with a smaller measurement uncertainty than the expert laboratories chosen to establish the assigned value (ISO 13528:2022 §7.6) or, if applicable, by formulation (ISO 13528:2022 §7.3) or than the certified measurement uncertainty associated with a certified reference material property value (ISO 13528:2022 §7.4). $u_{max,rel}$ is set to the standard deviation accepted for the PT assessment, $\sigma_{pt}$ (expressed as a percentage of the assigned value). Consequently, case "a" becomes: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt,\%}$ .

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, relative measurement uncertainties smaller than $u_{rel}(x_{pt})$ are possible and plausible.

If $u_{rel}(x_i)$ is larger than $\sigma_{pt,\%}$ (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 $\zeta$ score, though the corresponding performance, expressed as a $z$ score, may be questionable or unsatisfactory.

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

## 6.2 General observations

Twenty-five NRLs from 24 Member States and Switzerland, and twenty OCLs (from Germany, Italy, Spain and Belgium) registered to the exercise. Two OCLs did not report results. The remaining 43 laboratories, represent most of the EU Member States (except Poland, Estonia and Malta). In addition, three NRLs and 2 OCLs reported results only for formaldehyde.

## 6.3 Laboratory results and scorings

### *6.3.1. Performances*

The laboratory performance for melamine and formaldehyde in test item 1 was evaluated using z and $\zeta$ (zeta) scores

Annexes 5 and 6 present the reported results as tables and graphs for both measurands. NRLs and OCLs are denoted as LNxx and LOxx, respectively.

Figure 1 summarises the performance scores obtained, with a total of 38 and 43 results scored for melamine and formaldehyde, respectively. Overall, a very good performance ("satisfactory" according to the z score) is observed for 95 % and 98 % of the results reported for melamine and formaldehyde, respectively. Similarly, the evaluation of the laboratory performance, according to the zeta scores, was satisfactory for over 70 % of the reported results. Two unsatisfactory (blunder) results were reported, one for melamine and one for formaldehyde, likely attributable to incorrect dilution factors used: a factor of 100 for LN19 for melamine, and a factor of 10 for LN15 for formaldehyde.

The robust statistics, calculated using the Excel add-in developed by the Statistical Subcommittee of the Analytical Methods Committee of the UK Royal Society of Chemistry [9], yielded the following results: 2.47 ± 0.13 (5 %) mg $kg^{-1}$ for melamine and 12.2 ± 0.7 (6 %) mg $kg^{-1}$ for formaldehyde. These values are in good agreement with the assigned values by the EURL and presented in Table 1. The low robust standard deviations observed suggest that the value of $\sigma_{pt}$ (set to 15 % in this PT) could be decreased for future PT assessments.

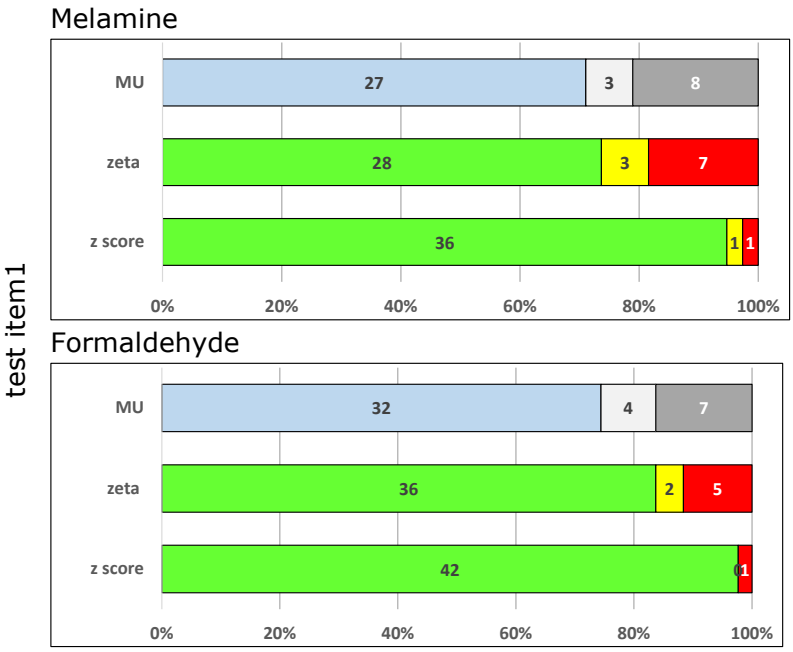



**Figure 1:**

Overview of laboratory performance per measurand according to *z* and *ζ* scores for formaldehyde and melamine in test item 1. Corresponding number of laboratories are included in the graph.

Satisfactory, questionable and unsatisfactory performances (z and zeta scores) are indicated in green, yellow and red, respectively.

Realistic, underestimated and overestimated measurement uncertainties (a,b,c) are indicated in blue, white and grey, respectively.

### *6.3.2. Truncated values*

Laboratory LN15 reported an unrealistic truncated value (< 1.86 mg $kg^{-1}$) for melamine in test item 1, while the assigned value for this measurand is 2.528 mg $kg^{-1}$ (Table 1).

### *6.3.3. Measurement uncertainties*

The majority of laboratories reporting quantitative results provided expanded measurement uncertainties (MU) and coverage factors. However, two NRLs and two OCLs did not report MU for melamine, and one NRL and one OCL did not provide them for formaldehyde. Additionally, three participants did not report the coverage factor applied. Nevertheless, Figure 1 shows that over 70 % of the participants provided realistic evaluation of the measurement uncertainty.

### *6.3.4. Test item 2 compliance statement evaluation*

Participants were requested to perform three consecutive migration experiments (m1, m2 and m3) at 70 ± 2 °C for 120 ± 5 minutes, on three different mugs (MUG1, MUG2 and MUG3 – test item 2), following their routine procedures. They were then requested to determine the "mass fraction of melamine and formaldehyde" migrated from the mugs into the food simulant B solution, and to assess the compliance of the mugs with the specifications outlined in Commission Regulation (EU) 2020/1245 [1]. In addition, participants were requested to provide (i) information about their migration procedures in the questionnaire, and (ii) the temperature profile measured in the food simulant.

The final compliance statements should align with the participants' data and conform to the requirements outlined in Commission Regulation (EU) 2020/1245, specifically Section 2.1.6 of Annex V, which prescribes compliance rules for "Repeated use materials and articles." According to these requirements, evaluating the compliance of a repeated-use article involves two key assessments: (i) verifying compliance with the Specific Migration Limits (SML), and (ii) assessing the stability of the article by comparing results from two consecutive migrations ((m1,m2) and (m2,m3)).

Most of the laboratories submitted the following information for melamine and formaldehyde in three different mugs (MUG1, MUG2, MUG3):

- Results from three migration tests (m1, m2, m3);
- Associated expanded uncertainty for the 1st migration test, U(m1)*;
- Corresponding coverage factor used, k
- Compliance statement for test item 2; and
- Temperature profile used for the migration tests.

(*) Note: Although laboratories provided the expanded uncertainty for the first migration only, the EURL calculated the standard measurement uncertainty by dividing the expanded uncertainty by the coverage factor (u(m1) = U(m1)/k), and the corresponding relative standard deviation was calculated by dividing the standard measurement uncertainty by the migration value (RSD = u(m1)/m1), which was assumed to be constant for similar migration levels (RSD(m1) = RSD(m2) = RSD(m3).

The analytical results reported by the participants for three consecutive migration tests (m1, m2, m3) of melamine and formaldehyde from test items 2 are presented in Annexes 11 and 12. A summary overview is presented in Figure 2, grouped by mugs.

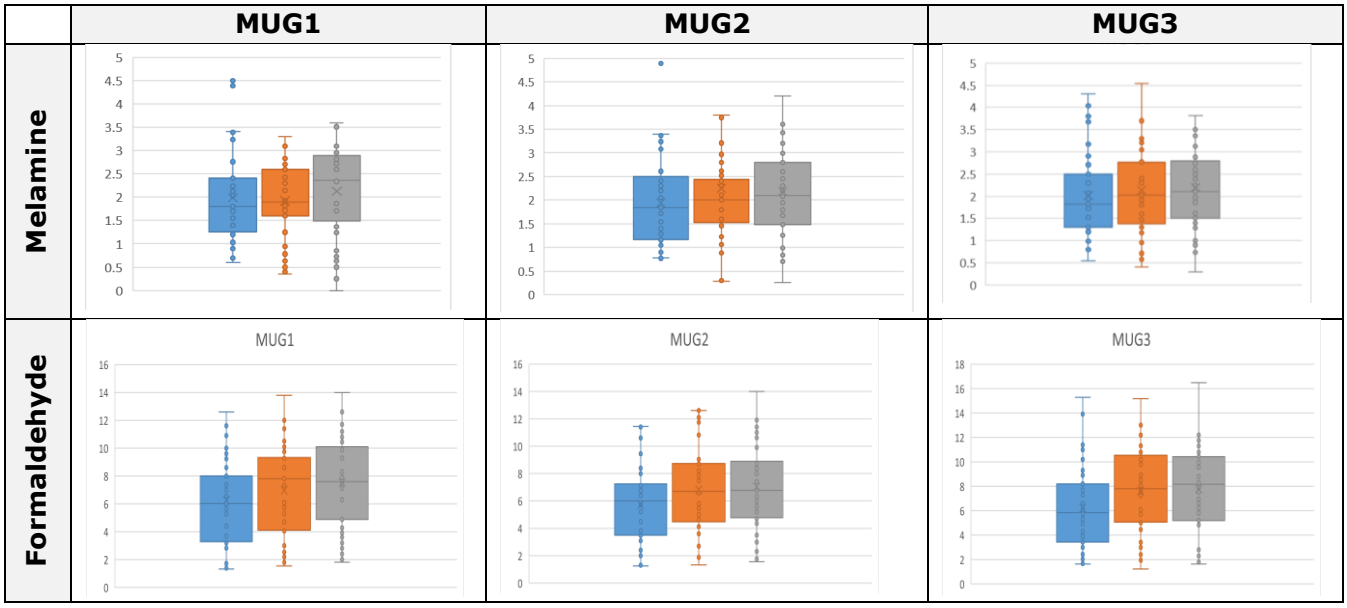



**Figure 2**: Box plots of the melamine and formaldehyde results reported by the participants for the three consecutive migrations ($m_1$, blue; $m_2$, orange; $m_3$, grey) from three mugs (MUG 1-3), (migration in mg $kg^{-1}$)

The EURL performed the following evaluation of the participants' data:

- Scrutinising all temperature profiles and categorised them as Satisfactory (S), Questionable (Q) and Unsatisfactory (U), as indicated in Table 2 and the last column of Annexes 11-12;
- Comparing each third migration result with the relevant SML (Example 1) and flagging values higher than SML with a pink cell in Annexes 11-12; and
- Examining consecutive migrations (m2, m1) and (m3,m2) (Example 2) to identify increases in migrated levels, and flagging any such increase with the symbol "↗".

These calculations were performed for all the reported results.

| **Example 1: Is a migrated level (m3) above SML? Crit.1** | |
|---|---|
| **Melamine** (Annex 11)<br>**SML** = 2.5 mg $kg^{-1}$<br>**LN01**, **MUG3**<br><br>m1 = 2.59 mg $kg^{-1}$<br>U(m1) = 0.259 mg $kg^{-1}$; k = 1.73<br>u(m1) = 0.259 / 1.73 = 0.15 mg $kg^{-1}$<br>RSu(m1)=RSu(m3)= 0.15 / 2.59 = 5.8 %<br><br>**m3** = 2.9 mg $kg^{-1}$<br>u(m3) = 0.058 * 2.9 = 0.17<br>**Crit.1**: (2.9 – 2.5) / 0.17 = 2.39 > 1.64<br>➔ **m3 > SML** | |

| **Example 2: Are the migrated levels stable?** | |
|---|---|
| **Melamine** (Annex 11)<br>**LN08**, **MUG2**<br><br>m1 = 1.4 mg $kg^{-1}$;<br>U(m1) = 0.34 mg $kg^{-1}$; k = 2<br>u(m1) = 0.34 / 2 = 0.17 mg $kg^{-1}$<br>RSu(m1)= 0.17 / 1.4 = 12.1 %<br>RSu(m1) = RSu(m2) = RSu(m3)<br><br>**m2** = 1.46 mg $kg^{-1}$<br>u(m2) = 1.46 * 0.121 = 0.18 mg $kg^{-1}$<br>**m3** = 2.45 mg $kg^{-1}$<br>u(m3) = 2.45 * 0.121 = 0.30 mg $kg^{-1}$<br><br>Crit.2: (2.45 – 1.46) / (0.18 + 0.30) =<br>= 2.1 > 1.64<br>➔ **m3 > m2**<br>Depicted in Annex 11 as 1.46 ↗ 2.45 | **Formaldehyde** (Annex 12)<br>**LN14, MUG3**<br>m1 = 9.3 mg $kg^{-1}$;<br><br>U(m1) = 2.12 mg $kg^{-1}$; k = 2<br>u(m1) = 2.12 / 2 = 1.06 mg $kg^{-1}$<br>RSu(m1)= 1.06 / 9.3 = 11.4 %<br>RSu(m1) = RSu(m2) = RSu(m3)<br><br>**m2** = 10.95 mg $kg^{-1}$<br>u(m2) = 10.95 * 0.114 = 1.25 mg $kg^{-1}$<br>**m3** = 11.75 mg $kg^{-1}$<br>u(m3) = 11.75 * 0.114 = 1.33 mg $kg^{-1}$<br><br>Crit.2: (11.75 – 10.95) / (1.25 + 1.33)<br>= 0.31 < 1.64<br>➔ **m3 ≤ m2** |

Note: Four laboratories (LN02, LN26, LN27, LO48) seem to have reported expanded uncertainty in %, rather than in mg $kg^{-1}$. For further calculations, the EURL-FCM considered these values as relative expanded uncertainties.

The EURL scrutinised the temperature profiles based on expert judgement. While Regulation (EU) No 10/2011 [10] specifies the test conditions (time and temperature) for plastic food contact material and articles, it does not provide tolerance intervals. The only document referring to tolerance ranges for time and temperature during the migration experiment is the normative Annex C of EN 13130-1:2004 [11], which establishes the following tolerance ranges for migration tests: 120 ± 5 min and 70 ± 2 °C, for contact time and temperature, respectively. These values were used for evaluation of the temperature profiles together with expert judgement. The result of this evaluation is reported in Table 2 using three quality categories: Satisfactory, Questionable and Unsatisfactory.

Annexes 11 and 12 present the evaluation conducted by the EURL based on the two criteria for the 9 results reported by the 43 laboratories for the three migrations in three mugs. The following assessment process was applied:

Criterion 1 (m3 ≤ SML) is considered fulfilled (yes) for each laboratory when the three results (M1m3, M2m3 and M3m3) are proven not to exceed the Specific Migration Limit (SML) taking the standard measurement uncertainty in consideration. When the value of m3 is at or below SML, assessing Criterion 1 is straightforward, even in cases where measurement uncertainties are not provided.

Criterion 2, addressing stability, is considered fulfilled (yes) when there is no observed increase in migration for any consecutive migrations. This criterion always requires knowledge of the laboratory's measurement uncertainty.

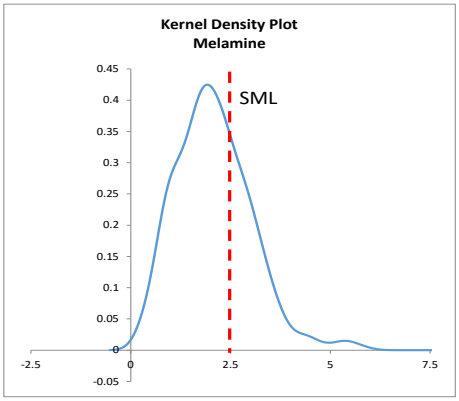
Consequently, samples with stable migration results and with a third migration result below the SML should be categorised as "Compliant." Similarly, non-stable migration results or third migration results above the SML should be labelled as "Non-Compliant." These logical conclusions should be compared with the assessments provided by the laboratories to ensure **coherence**. A compliance statement of a participant is regarded as coherent when supported by the data provided.

However, this consistency was not always observed. Incoherent statements were noted, such as laboratories declaring their samples (i) as "compliant" while presenting experimental evidence of instability or values above the SML, or (ii) as "non-compliant" when no extreme values or instability could be observed.

A detailed analysis of the reported results for test item 2 is presented hereafter.

**Melamine** (see Table 2, 3 & Annex 11)

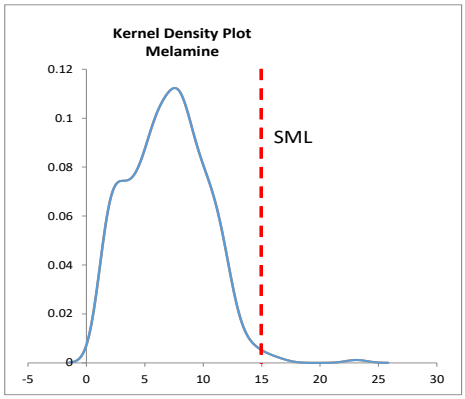
- Most of the laboratories (38 out of 44) reported quantitative results; 5 laboratories did not perform migration tests, while LN15 reported only truncated ("less than") values;
- The reported relative standard uncertainties (RSu) ranged from 3 to 27 %; while 6 laboratories did not provide their measurement uncertainties.
- Except one outlier (LO47 - MUG2,m2), all the 340 reported values ranged from 0.3 and 5.8 mg $kg^{-1}$, with a median value of 2.0, hence bracketing the SML of 2.5 mg $kg^{-1}$, as shown in the Kernel density plot [9] below.




- 5 laboratories issued an "incoherent" compliance statement that is contradicted by their experimental data.
- 5 laboratories gave a compliance statement without reporting uncertainty, thus making the evaluation impossible.
- 9 laboratories reported some third migration results that exceeded the SML whereas 28 other laboratories provided values equal to or smaller than SML. This confirms the challenge in determining the compliance for melamine, particularly when values are close to the SML.
- Results from 22 laboratories support the stability of the melamine migration from the test item 2 whereas 10 laboratories observed the opposite.
- 7 laboratories **presented experimental evidence indicating the non-compliance of melamine in the mugs due to stable successive migrations but with the 3rd migration above SML**, aligning with the EURL's findings; one of them (LN11) provided unsatisfactory temperature profile.

**Formaldehyde** (see Table 2, 3 & Annex 12)

- 43 laboratories reported results (excl. LO47);
- No truncated values were reported;
- The reported relative standard uncertainties (RSu) ranged from 2 to 26 %; while 6 laboratories did not provide their MU.
- Except one outlier (LN22 - MUG1,m1), all the 381 reported values ranged from 1.2 to 16.5 mg $kg^{-1}$, with a median value of 7.0 mg $kg^{-1}$; the third migration result never exceeded the SML of 15 mg $kg^{-1}$, when taking measurement uncertainties into account, thus complying with Criterion 1 (m3-u ≤ SML), as shown in the Kernel density plot [9] below.




- 9 laboratories issued an "incoherent" compliance statement that is contradicted by their experimental data.
- 7 laboratories gave a compliance statement without reporting uncertainty, thus making the evaluation impossible.
- All laboratories reported third migration results equal to or below the SML.
- Results from 21 laboratories demonstrated increasing levels in successive migrations, thus failing to comply with Criterion 2.
- 15 laboratories presented experimental evidence supporting the compliance and stability of formaldehyde in the mugs. However, three of them (LN15, LO23, LO39) erroneously assessed test item 2 as non-compliant. Therefore, only 12 of the laboratories provided a compliance statement in line with the EURL conclusions.

Finally, only 6 laboratories assessed correctly the compliance of test item 2 for both melamine and formaldehyde in line with the observations of the EURL.

**Table 2**: Comprehensive presentation detailing compliance statements of the participants ("statement") related to melamine (M) and formaldehyde (F), stability ("stable") and compliance with SML ("<SML") assessed by EURL, coherence of their statement to their data ("coherence"), the calculated relative standard uncertainty ("RSu (k=1)"), the evaluation of the temperature profile used for migration ("T-profile"; S, satisfactory; Q, questionable; U, unsatisfactory) and the overall agreement with the EURL conclusions ("OVERALL").

| | Melamine | | | | | | Formaldehyde | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | Statement | Stable | < SML | coherence | RSu (k=1) | Lab | Statement | Stable | < SML | coherence | RSu (k=1) | T profile | OVERALL |
| LN01 | NC | yes | no | yes | 6% | LN01 | C | yes | yes | yes | 12% | S | MF |
| LN02 | C | yes | yes | yes | 11% | LN02 | NC | no | yes | yes | 4% | Q | |
| LN03 | C | yes | yes | yes | 8% | LN03 | C | no | yes | no | 6% | U | |
| LN04 | not tested | | | | | LN04 | NC | no | yes | yes | 8% | U | |
| LN05 | NC | ? | ? | ? | | LN05 | NC | ? | yes | ? | | S | |
| LN06 | not tested | | | | | LN06 | NC | ? | yes | ? | | S | |
| LN07 | NC | no | yes | yes | 6% | LN07 | NC | no | yes | yes | 6% | S | |
| LN08 | NC | no | yes | yes | 12% | LN08 | NC | no | yes | yes | 6% | Q | |
| LN09 | C | no | yes | no | 3% | LN09 | C | no | yes | no | 6% | U | |
| LN10 | C | yes | yes | yes | 13% | LN10 | C | yes | yes | yes | 10% | U | F |
| LN11 | NC | yes | no | yes | 10% | LN11 | NC | no | yes | yes | 8% | U | M |
| LN12 | C | ? | yes | ? | | LN12 | C | ? | yes | ? | | Q | |
| LN13 | NC | yes | no | yes | 15% | LN13 | C | yes | yes | yes | 14% | S | MF |
| LN14 | NC | yes | no | yes | 14% | LN14 | C | yes | yes | yes | 12% | Q | MF |
| LN15 | NC | yes | yes | no | | LN15 | NC | yes | yes | no | 10% | S | F |
| LN16 | C | yes | yes | yes | 7% | LN16 | C | yes | yes | yes | 5% | | F |
| LN17 | NC | no | yes | yes | 3% | LN17 | NC | no | yes | yes | 7% | Q | |
| LN18 | NC | yes | no | yes | 4% | LN18 | C | yes | yes | yes | 10% | S | MF |
| LN19 | C | yes | yes | yes | 10% | LN19 | C | no | yes | no | 10% | | |
| LN20 | C | yes | yes | yes | 20% | LN20 | C | yes | yes | yes | 15% | S | F |
| LN21 | NC | yes | no | yes | 13% | LN21 | C | yes | yes | yes | 12% | U | MF |
| LN22 | not tested | | | | | LN22 | ? | no | yes | ? | 2% | U | |
| LN23 | NC | no | yes | yes | 7% | LN23 | NC | yes | yes | no | 19% | S | F |
| LN24 | C | yes | yes | yes | 17% | LN24 | NC | no | yes | yes | 12% | S | |
| LN25 | NC | yes | no | yes | 5% | LN25 | C | yes | yes | yes | 3% | S | MF |
| LN26 | NC | no | yes | yes | 6% | LN26 | NC | no | yes | yes | 2% | S | |
| LN27 | C | yes | yes | yes | 15% | LN27 | C | no | yes | no | 5% | S | |
| LO30 | NC | no | no | yes | 6% | LO30 | NC | no | yes | yes | 7% | S | |
| LO31 | C | yes | yes | yes | 27% | LO31 | C | yes | yes | yes | 26% | S | F |
| LO32 | NC | ? | ? | ? | | LO32 | C | ? | yes | ? | | S | |
| LO33 | C | ? | yes | ? | | LO33 | NC | ? | yes | ? | | S | |
| LO34 | C | yes | yes | yes | 14% | LO34 | C | yes | yes | yes | 7% | S | F |
| LO35 | C | ? | yes | ? | | LO35 | C | no | yes | no | 5% | Q | |
| LO36 | C | yes | yes | yes | 21% | LO36 | C | yes | yes | yes | 16% | U | F |
| LO37 | NC | yes | yes | no | 15% | LO37 | NC | ? | yes | ? | 17% | Q | |
| LO38 | C | yes | yes | yes | 17% | LO38 | NC | ? | yes | ? | | Q | |
| LO39 | NC | yes | yes | no | 25% | LO39 | NC | yes | yes | no | 13% | S | F |
| LO40 | not tested | | | | | LO40 | NC | no | yes | yes | 18% | Q | |
| LO43 | NC | no | yes | yes | 14% | LO43 | NC | no | yes | yes | 11% | S | |
| LO44 | NC | yes | yes | no | 17% | LO44 | NC | no | yes | yes | 10% | Q | |
| LO45 | C | yes | yes | yes | 5% | LO45 | NC | no | yes | yes | 5% | S | |
| LO46 | not tested | | | | | LO46 | C | no | yes | no | 11% | S | |
| LO47 | NC | no | no | yes | 5% | LO47 | not tested | | | | | | |
| LO48 | NC | no | yes | yes | 10% | LO48 | NC | no | yes | yes | 5% | S | |

**Table 3** Summarised overview of the compliance statements of the laboratories for melamine (non-compliant) and formaldehyde (compliant), reported stability, reported compliance with SML, corresponding number of laboratories, coherence of their statement, agreement with the EURL conclusions.

**Melamine, assumed "Non-Compliant**

| Lab.statement | Stable? | < SML? | Nr | coherent with experiment? | aligned with EURL | Labs |
|---|---|---|---|---|---|---|
| NC | yes | no | 7 | yes | Yes | LN01; LN11; LN13; LN14; LN18; LN21; LN25 |
| NC | yes | yes | 4 | no | | LN15; LO37; LO39; LO44 |
| NC | no | no | 2 | yes | | LO30; LO47 |
| NC | no | yes | 7 | yes | | LN07; LN08; LN17; LN23; LN26; LO43; LO48 |
| NC | ? | ? | 2 | | | LN05; LO32 |
| C | yes | yes | 13 | yes | | LN02; 03; 10; 16; 19; 20; 24; 27; LO31; 34; 36; 38; 45 |
| C | no | yes | 1 | no | | LN09 |
| C | ? | yes | 3 | | | LN12; LO33; LO35 |
| not tested | -- | -- | 5 | | | LN04; LN06; LN22; LO40; LO46 |
| | (?): no MU | total | 44 | | | |

**Formaldehyde, assumed "Compliant"**

| Lab.statement | Stable? | < SML? | Nr | coherent with experiment? | aligned with EURL | Labs |
|---|---|---|---|---|---|---|
| C | yes | yes | 12 | yes | Yes | LN01; 10; 13; 14; 16; 18;20; 21; 25; LO31; 34; 36 |
| C | no | yes | 6 | no | | LN03; LN09; LN19; LN27; LO35; LO46 |
| C | ? | yes | 2 | | | LN12; LO32 |
| NC | yes | yes | 3 | no | | LN15; LN23; LO39 |
| NC | no | yes | 14 | yes | | LN02; 04; 07; 08; 11; 17; 24; 26; LO30; 40; 43; 44; 45; 48 |
| NC | ? | yes | 5 | | | LN05; LN06; LO33; LO37 |
| -- | no | yes | 1 | | | LN22 |
| not tested | -- | -- | 1 | | | LO47 |
| | (?): no MU | total | 44 | | | |

### *6.3.5. Observations on the compliance assessment of test item 2*

Given the significant variations observed in compliance statements across laboratories, the novelty of compliance assessment for NRLs and OCIs, and the large variety of temperature profiles reported by the laboratories, in addition to migrated levels of melamine in food simulant B close to the SML, the EURL-FCM conducted an additional experiment. This experiment aimed to assess the impact of temperature tolerance in the food simulant on the compliance of test item 2. Initially, a migration test was conducted on three different mugs, using slightly different oven settings while ensuring that the temperature profiles remained within the tolerance range of 68 – 72 °C (see Annex 8).

Annex 7 demonstrates the EURL's ability to replicate the temperature profile for consecutive migrations for test item 2. This is important for obtaining repeatable migration results (Annexes 9A and 10A).

Annex 8 shows that different temperature profiles can occur in the food simulant during migration, with ranges 68 - 72 °C, 67.8 - 69.5 °C, and 67.3 - 68.3 °C, the latter exceeding the tolerance range for 15 minutes. Annexes 9B and 10B show that the migration levels increase with higher "average" temperature of the food simulant. Specifically for melamine (Annex 9B), migrations with (lower) temperature profiles outside the tolerance range exhibit significantly lower levels compared to those within the tolerance range. A similar trend is observed for formaldehyde (Annex 10B).

In the case of melamine, migration levels significantly below the SML indicate compliance when the temperature profile is lower, whereas the migration is significantly above the SML, indicating non-compliance, with the highest temperature profile. This experiment highlights the sensitivity of both melamine and formaldehyde migration to minor variations in the contact temperature profile, even within the tolerable range. Therefore, to accurately assess the stability of an article, it is crucial to conduct **three consecutive migrations with similar temperature profiles**. This is essential to avoid erroneously declarations of instability for an article that is in fact stable.

Challenges arise in maintaining consistent compliance statements concerning the SML, as narrowing the tolerance range of the temperature in the food simulant may be required. It should be noted that these observations may or may not be applicable to other materials, articles, or migration conditions.

The EURL-FCM emphasises the critical importance of conducting migration tests strictly under the required contact time and at the correct contact temperature of the food simulant (not that of the oven). Based on appropriate analytical results and despite acknowledging the complications to follow, the EURL challenges the tolerable range (70 ± 2 °C) for the contact temperature laid down in the normative Annex C of EN 13130-01:2004 [11], considering it too broad for some analytes (as melamine and formaldehyde) under certain conditions (2 h at 70 °C) depending on the kinetic of the migration process and thus insufficient to ensure comparable migration (Annex 9-10).

For future proficiency testing exercises involving a migration step, participants will be requested to use exclusively calibrated data loggers capable of measuring the contact temperature of the food simulant within the tested article at high frequency (30-60 seconds). Achieving a satisfactory profile requires thorough optimisation before the actual migration experiment, using the spare article provided. When testing articles repeatedly, laboratories must guarantee repeatable temperature profiles for the first, second, and third migrations to ensure a proper assessment of migration stability. This involves maintaining identical initial temperatures of the articles at the moment of filling with the food simulant pre-heated to the same temperature.

Other factors that could impact migration, as demonstrated in the previous PTs, include food simulant losses (up to 25 ml [13-15]) and their compensations, uniformity of the contact temperature profile within the oven; timing and location of the food simulant filling, accurate correlation between the measured volume and calculated contact surface, etc. As a general guideline, the volume of the food simulant should be measured at room temperature, while the contact surface should be calculated using a mug filled with pre-heated food simulant.

### *6.3.6. Additional information extracted from the questionnaire*

All participants completed the questionnaire, providing valuable information about their laboratories, operational methodologies, and analytical methods. Annex 13 summarises the experimental details, the technique used and additional information requested.

The majority of the participants (26 out of 43) were accredited according ISO 17025 for the analyses of both melamine and formaldehyde in food simulants, while 10 participants are accredited only for formaldehyde and two exclusively for melamine. Six

participants declared that they do not have migration procedures within the scope of their accreditation, and an additional five were not accredited for the scope of this PT.

***Instrumental techniques applied***

Analytical techniques for the analyses of melamine and formaldehyde in food simulant B are presented in Table 4, while the additional experimental details are provided in Annex 13. The use of different instrumental techniques for both measurands did not impact the performance of the laboratories in analysing test item 1, as shown in Annexes 5 and 6.

**Table 4** Analytical techniques used to determine melamine (MEL) and formaldehyde (FA) in test item 1.

| Technique | MEL | FA |
|---|---|---|
| HPLC-DAD | 3 | 1 |
| HPLC-UV | 13 | 7 |
| LC-MS | 17 | 1 |
| LC-MS/MS | 5 | 1 |
| Spectrophotometry | -- | 23 |

The main difference among the instrumental techniques relates to the achievable Limits of Quantification (LOQs). For melamine, reported LOQs start at 1 $\mu$g kg$^{-1}$ and higher for LC-MS/MS, while for HPLC-UV they range from 1 to 1.5 mg kg$^{-1}$, resembling reporting limits more than LOQs. However, the majority of participants reported a reasonable LOQ range of 0.05-0.5 mg kg$^{-1}$. For formaldehyde, the LOQ range is much narrower (0.1-3 mg kg$^{-1}$), and there is no significant difference observed based on the sample preparation technique, whether using 2,4-dinitrophenylhydrazin, acetyl acetone or chromotropic acid.

# 7 Conclusion

The proficiency test FCM-23/01 was organised to assess (i) the analytical capabilities of EU National Reference Laboratories and Official Control Laboratories in determining the mass fractions of melamine and formaldehyde in food simulant B and (ii) the ability to assess the compliance of a repeated-use article against the new requirements outlined in Commission Regulation (EU) 2020/1245.

The participants demonstrated a highly satisfactory overall performance in determining these substances in food simulant B (3% acetic acid), with approximately 95 % of the results achieving z scores below 2 (satisfactory) against the 15 % standard deviation for proficiency assessment set by the EURL-FCM. This suggests that the $\sigma_{pt}$ could potentially be further reduced in the future.

Most of the participants reported realistic measurement uncertainty estimations.

The task of evaluating participants' compliance statements regarding test item 2 posed notable challenges as even minor variations in migration test conditions, while within acceptable limits, significantly influenced the test results. Only 28% of the laboratories accurately assessed compliance and stability for formaldehyde in test item 2, aligning with the conclusions drawn by the European Reference Laboratory (EURL).

Assessing the stability of melamine and its compliance with the specific migration limit (SML) for test item 2 proved to be particularly difficult as the migration was close to the

SML. Less than 20% of the laboratories correctly evaluated compliance and stability for melamine in test item 2, consistent with the EURL conclusions.

In conclusion, the assessment of compliance for melamine-bamboo mugs remains a challenging task that requires further investigation.

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## List of abbreviations and symbols

| | |
|---|---|
| DAD | Diode Array Detector |
| DG SANTE | Directorate General for Health and Food Safety |
| EC | European Commission |
| EFSA | European Food Safety Authority |
| EU | European Union |
| EURL | European Union Reference Laboratory |
| FCM | Food Contact Materials |
| GUM | Guide for the Expression of Uncertainty in Measurement |
| GC-MS(MS/MS) | Gas chromatography-mass spectrometry or tandem mass spectrometry |
| HPLC-FLD | High performance liquid chromatography-fluorescence detection |
| HPLC-UV(DAD) | High performance liquid chromatography-ultraviolet (diode array detection) |
| ILC | Interlaboratory Comparison |
| ISO | International Organization for Standardization |
| JRC | Joint Research Centre |
| LOD | Limit of Detection |
| LOQ | Limit of Quantification |
| NRL | National Reference Laboratory |
| OCL | Official Control Laboratory |
| PT | Proficiency test |
| SOP | Standard operating procedure |
| $k$ | coverage factor |
| $\sigma_{pt}$ | 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 |
| $z$ (or $z'$) | z (or z') score |
| $\zeta$ | zeta score |

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## Annex 1: A. Announcement of the PT round "FCM-23/01"

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

Ispra/Geel, 31 January 2024

**Subject: Announcement of a Proficiency Testing round "FCM-23/01"**

Dear NRL colleagues,

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM) will organise a proficiency test round (FCM-23/01) for the "Determination of melamine and formaldehyde migrated from bamboo/melamine kitchenware". This PT is organised to support the sections 2.1.6 and 3.3.2 of Annex V of the Commission Regulation (EU) 2020/1245, related to the repeated use materials and article.

It is your duty as NRL to participate in the PTs organised by the EURL-FCM as agreed in the work program. There is no charge for participation.

Two test items will be provided:

- One solution of melamine and formaldehyde in food simulant B;
- 4 mugs for migration test in food simulant B

This PT will be organised under ISO 17043 accreditation.

The following time schedule is foreseen:

| | |
|---|---|
| Invitation for registration: | end of May 2023 |
| Deadline for registration: | mid-June 2023 |
| Sample dispatch: | end June 2023 |
| Deadline for reporting: | end August 2023 |
| Draft PT report: | September 2023 |

Please forward this invitation to the Official Control Laboratories (OCLs) in your network that would be interested in participating. As the test items are limited, the OCLs registration will be accepted on first come first serve base depending on sample availability.
Soon you will receive an e-mail with a link for registration.
Do not hesitate to contact us if you have further questions.

Stefanka Bratinova
PT coordinator

Commission européenne/Europese Commissie, Retieseweg 111, 2440 Geel, BELGIQUE/BELGIË - Tel. +32 14571705
Office: 010 L01/103B - Tel. direct line +32 14571-1234

Commissione europea, Via Enrico Fermi 2749, 21027 Ispra VA, ITALIA - Tel. +39 332789111

Eddo.hoekstra@ec.europa.eu

## Annex 1: B. Invitation letter

Ref. Ares(2023)3591361 - 24/05/2023

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Directorate F - Health, Consumers & Reference Materials
**European Union Reference Laboratory for Food Contact Materials**

EURL European Union Reference Laboratory for Food Contact Materials

Ispra/Geel, 17 May 2023

**Subject:** **Invitation to participate in Proficiency Testing round "FCM-23/01"**

Dear Colleague,

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM), managed by the Joint Research Centre (JRC) of the European Commission (EC), invites you to participate in the Proficiency Testing round **FCM-23/01** for the " **Determination of melamine and formaldehyde in bamboo/melamine kitchenware**".

It is your duty as NRL to participate in the PTs organised by the EURL-FCM as agreed in the work programme. There is no charge for participation.

The following test items will be dispatched:

- test item 1 - One solution of melamine and formaldehyde in clean food simulant B;
- test item 2 - 4 mugs for migration test in food simulant B.

Performance scoring (z-scores) for melamine and FA will be assigned to the participants for test item 1. For test item 2, the compliance statement and its justification will be evaluated, though all results from the 3 consecutive migration tests should be reported to support the statement.

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, laboratory codes of National Reference Laboratories (NRLs) - appointed in line with Regulation (EU) 2017/625 - will be disclosed to DG SANTE upon request for (long-term) performance assessment. Similarly, laboratory codes of appointed Official Control Laboratories (OCLs) may be disclosed to their NRL upon request.

If you intend to participate, register electronically as soon as possible by using the link below and following the instructions provided.
https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=2881

The **deadline for registration is set to 12 June 2023**.
Samples will be dispatched by the end-June.
The deadline for submission of results will be end August.

Do not hesitate to contact us if you have any question.

Kind regards

/signed electronically in Ares/
Stefanka Bratinova
FCM-23/01 PT Coordinator

/signed electronically in Ares/
Eddo J. Hoekstra
Operating Manager EURL-FCM

Cc: U.Vincent, Head of Unit, JRC.F.5 Food and Feed Compliance

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## Annex 2: Instructions for participants



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate F – Health and Food
**Food and Feed Compliance**

Geel, 19 June 2023
JRC.F.5/SPB/mt 23-043

**Subject:** **Instructions to participants in Proficiency Testing round "FCM-23/01"**

Dear Ms./Mr.,

Thank you for participating in the FCM-23/01 proficiency test (PT) **"Determination of melamine and formaldehyde in bamboo/melamine kitchenware"**.

The parcels are dispatch today. Each parcel contains:

(1) test item 1 - One 20 mL vial containing a solution of melamine and formaldehyde in food simulant B;
(2) test item 2 - 4 mugs for migration test in food simulant B.

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

Test item 1 should be stored refrigerated until analysis and should be analysed for formaldehyde and melamine content according to your routine procedure. Test results +/- MU should be expressed in mg/kg food simulant assuming the density of food simulant B 1 kg·m−3. The laboratory performance will be z and ζ-scored for evaluation.

Test item 2 should be treated as routine samples for which the compliance have to be assessed following the requirements of the Commission Regulation (EU) 2020/1245 of 2 September 2020 amending and correcting Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food and more specific the Section 2.1.6 of Annex V, related to the compliance rules for "Repeated use materials and articles".

Three consecutive migration experiments should be performed (MIG1, MIG2 and MIG3) at 70 °C for 2 h and results for formaldehyde and melamine have to be reported for three mugs (MUG1, MUG2 and MUG3). The results should be reported in mg/kg food simulant in the same format (e.g. number of significant figures) as you normally report to customers.

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Office: 210 L00/ 032 – Tel. direct line +32 14571-1800

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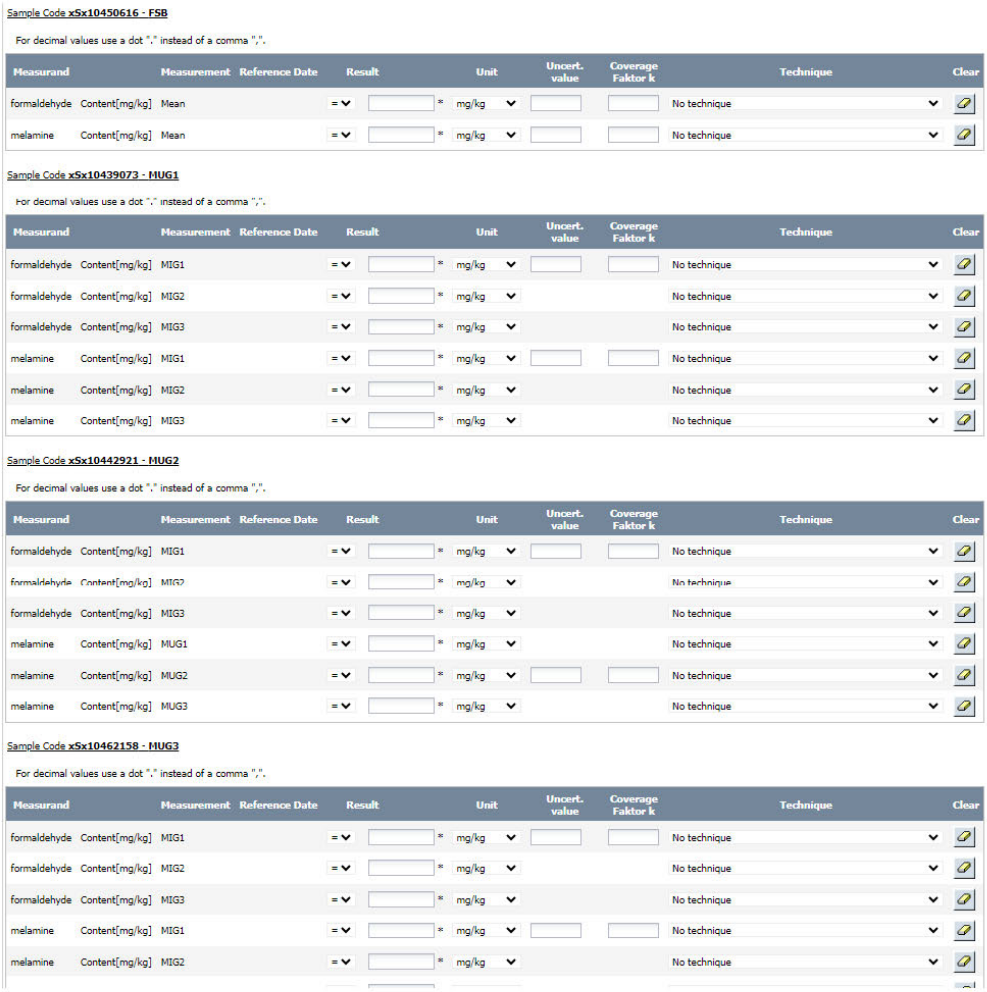
Stefanka-Petkova.BRATINOVA@ec.europa.eu

Three consecutive migration experiments should be performed (MIG1, MIG2 and MIG3) at 70 °C for 2 h and results for formaldehyde and melamine have to be reported for three mugs (MUG1, MUG2 and MUG3). The results should be reported in mg/kg food simulant in the same format (e.g. number of significant figures) as you normally report to customers.

Example of the reporting table:



Sample Code **xSx10450616 - FSB**

For decimal values use a dot "." instead of a comma ",".

| Measurand | | Measurement | Reference Date | Result | | Unit | Uncert. value | Coverage Faktor k | Technique | Clear |
|---|---|---|---|---|---|---|---|---|---|---|
| formaldehyde | Content[mg/kg] | Mean | | = | * | mg/kg | | | No technique | |
| melamine | Content[mg/kg] | Mean | | = | * | mg/kg | | | No technique | |

Sample Code **xSx10439073 - MUG1**

For decimal values use a dot "." instead of a comma ",".

| Measurand | | Measurement | Reference Date | Result | | Unit | Uncert. value | Coverage Faktor k | Technique | Clear |
|---|---|---|---|---|---|---|---|---|---|---|
| formaldehyde | Content[mg/kg] | MIG1 | | = | * | mg/kg | | | No technique | |
| formaldehyde | Content[mg/kg] | MIG2 | | = | * | mg/kg | | | No technique | |
| formaldehyde | Content[mg/kg] | MIG3 | | = | * | mg/kg | | | No technique | |
| melamine | Content[mg/kg] | MIG1 | | = | * | mg/kg | | | No technique | |
| melamine | Content[mg/kg] | MIG2 | | = | * | mg/kg | | | No technique | |
| melamine | Content[mg/kg] | MIG3 | | = | * | mg/kg | | | No technique | |

Sample Code **xSx10442921 - MUG2**

For decimal values use a dot "." instead of a comma ",".

| Measurand | | Measurement | Reference Date | Result | | Unit | Uncert. value | Coverage Faktor k | Technique | Clear |
|---|---|---|---|---|---|---|---|---|---|---|
| formaldehyde | Content[mg/kg] | MIG1 | | = | * | mg/kg | | | No technique | |
| formaldehyde | Content[mg/kg] | MIG2 | | = | * | mg/kg | | | No technique | |
| formaldehyde | Content[mg/kg] | MIG3 | | = | * | mg/kg | | | No technique | |
| melamine | Content[mg/kg] | MUG1 | | = | * | mg/kg | | | No technique | |
| melamine | Content[mg/kg] | MUG2 | | = | * | mg/kg | | | No technique | |
| melamine | Content[mg/kg] | MUG3 | | = | * | mg/kg | | | No technique | |

Sample Code **xSx10462158 - MUG3**

For decimal values use a dot "." instead of a comma ",".

| Measurand | | Measurement | Reference Date | Result | | Unit | Uncert. value | Coverage Faktor k | Technique | Clear |
|---|---|---|---|---|---|---|---|---|---|---|
| formaldehyde | Content[mg/kg] | MIG1 | | = | * | mg/kg | | | No technique | |
| formaldehyde | Content[mg/kg] | MIG2 | | = | * | mg/kg | | | No technique | |
| formaldehyde | Content[mg/kg] | MIG3 | | = | * | mg/kg | | | No technique | |
| melamine | Content[mg/kg] | MIG1 | | = | * | mg/kg | | | No technique | |
| melamine | Content[mg/kg] | MIG2 | | = | * | mg/kg | | | No technique | |

The results from the migration experiment will be not scored with z- and □ scores, though all the results will be published and commented. Instead, only the correctness of the compliance statements justifications will be evaluated. For that purpose, please be as detailed as possible in supporting your statements with explanations. This information should be filled in the questionnaire part of the same interface

For evaluation of the migration experimental conditions, the temperature profiles of the food simulant during the migration test and the volumes of the food simulants should be provided as well. As the reporting interface does not support yet the file uploads, *you have to send separate files via e-mail with downloads from the data loggers readings or an excel*

*table filled in with the T readings of the simulant every 5 min during the 120 min migration experiment.*

The homogeneity study for test item 1 was performed with a sample intake of 1 mL

You can find the MILC reporting website at https://web.jrc.ec.europa.eu/ilcReportingWeb. You need first to login with your EU login account (see detailed guideline) and then enter the personal password. Your unique password is indicated above in the box under your address data. The system will guide you through the reporting procedure. Do not forget to submit and confirm when required.

Directly after submitting your results and the questionnaire online, you will be requested to print the completed report form. Please check carefully this report form and send it back via mail to us. In case mistakes are detected, contact the PT coordinator as soon as possible before the reporting deadline.

**The deadline for submission of results is 25/08/2023**. It will not be possible to submit your results after the deadline.

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. Lab codes of appointed Official Control Laboratories may be disclosed to their National Reference Laboratory upon request.

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.

Your participation in this PT is greatly appreciated. Please be aware of the existence of an appeal procedure in case you disagree with your scores.

Do not hesitate to contact me for further information.

With kind regards,

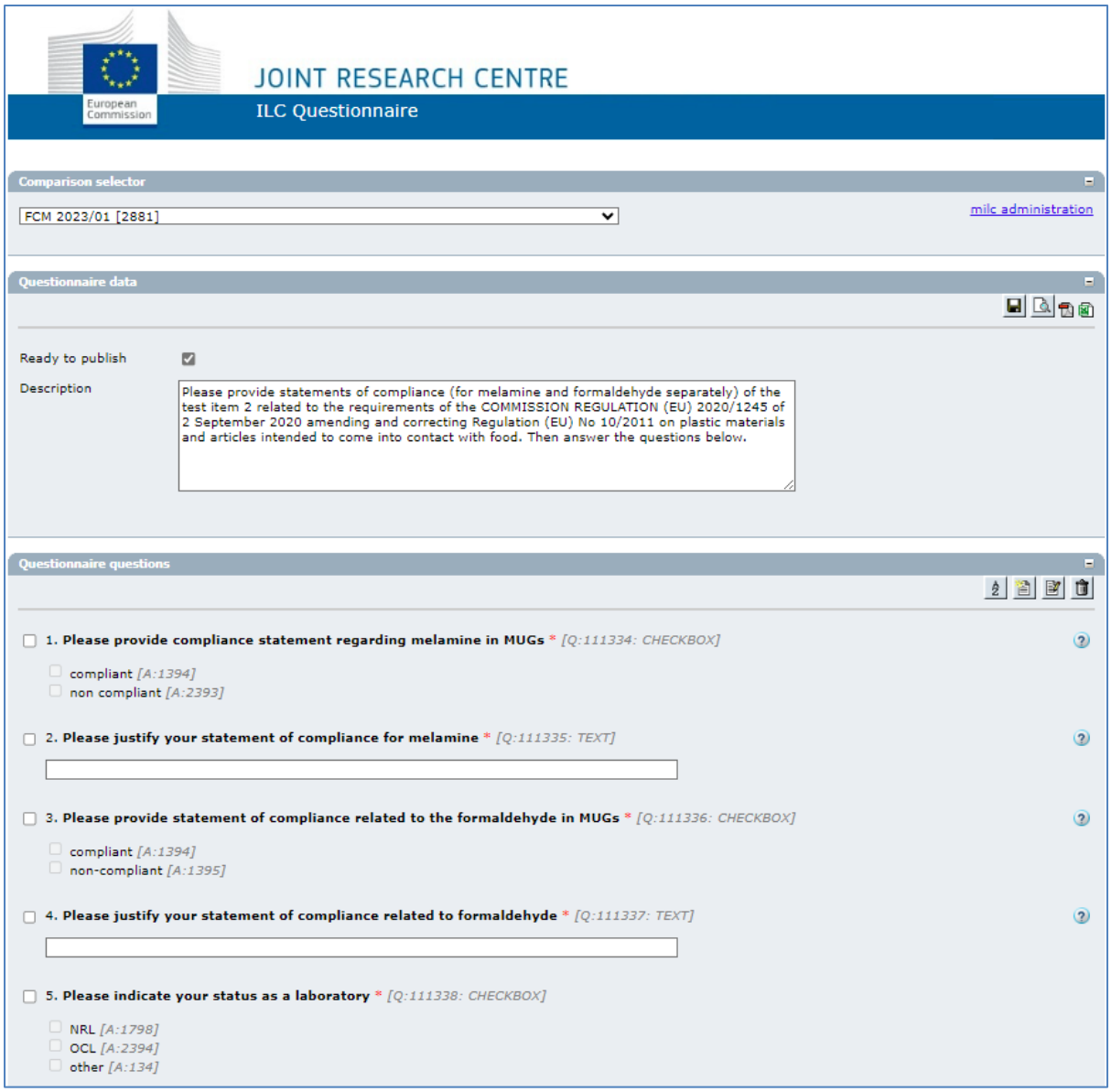
*/signed electronically in Ares/*

Stefanka Bratinova
PT Coordinator

*/signed electronically in Ares/*

Giorgia Beldi
Deputy PT Coordinator

# Annex 3: Questionnaire



JOINT RESEARCH CENTRE

ILC Questionnaire

**Comparison selector**

FCM 2023/01 [2881]

milc administration

**Questionnaire data**

Ready to publish ☑

Description: Please provide statements of compliance (for melamine and formaldehyde separately) of the test item 2 related to the requirements of the COMMISSION REGULATION (EU) 2020/1245 of 2 September 2020 amending and correcting Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food. Then answer the questions below.

**Questionnaire questions**

☐ 1. **Please provide compliance statement regarding melamine in MUGs** * *[Q:111334: CHECKBOX]*

- ☐ compliant *[A:1394]*
- ☐ non compliant *[A:2393]*

☐ 2. **Please justify your statement of compliance for melamine** * *[Q:111335: TEXT]*

☐ 3. **Please provide statement of compliance related to the formaldehyde in MUGs** * *[Q:111336: CHECKBOX]*

- ☐ compliant *[A:1394]*
- ☐ non-compliant *[A:1395]*

☐ 4. **Please justify your statement of compliance related to formaldehyde** * *[Q:111337: TEXT]*

☐ 5. **Please indicate your status as a laboratory** * *[Q:111338: CHECKBOX]*

- ☐ NRL *[A:1798]*
- ☐ OCL *[A:2394]*
- ☐ other *[A:134]*

6. **Are you accredited for the analyses of melamine and formaldehyde in food simulant B?** * *[Q:111339: CHECKBOX]*

- No *[A:396]*
- Other (please specify) *[A:1235]*
- Yes, for FA *[A:2396]*
- Yes, for MEL; *[A:2395]*
- Yes, for both *[A:2397]*

7. **Are you accredited for the migration procedure?** * *[Q:111403: CHECKBOX]*

- no *[A:179]*
- other, please specify below *[A:384]*
- yes *[A:124]*

7.1. Please specify *[Q:111404: TEXT]*

8. **Did you pre-heat the simulant before filling the article?** * *[Q:111396: RADIO]*

- NO *[A:299]*
- Other *[A:1437]*
- YES *[A:298]*

8.1. Temperature of the food simulant just before the filling of the test items * if parent A:298 checked *[Q:111398: TEXT]*

9. **How did you control the temperature of the food simulant during the migration experiments** * *[Q:111343: CHECKBOX]*

- Other (please specify) *[A:1235]*
- calibrated datalogger *[A:2400]*
- calibrated thermometer *[A:2399]*
- non-calibrated thermometer/datalogger *[A:2401]*
- not controlled *[A:2402]*

10. **Did you preheat the mugs before migration?** * *[Q:111342: RADIO]*

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

11. **Volume of the food simulant used for the migration experiments** * *[Q:111340: TEXT]*

12. **Temperature of the food simulant during the migration experiment (in oC) - Please send by mail a file from datalogger or a table with the FS temperature any 5 minutes of the migration experiment** *[Q:111341: TEXT]*

13. **Did you measure the food simulant losses after each migration?** * *[Q:111344: RADIO]*

- No *[A:1236]*
- Yes *[A:397]*

14. **If yes, please report the volume of the losses in ml** *[Q:111345: TEXT]*

☐ 15. **Analytical techniques for melamine in food simulant?** * *[Q:111346: CHECKBOX]*

- ☐ HPLC-DAD *[A:2404]*
- ☐ HPLC-UV *[A:1389]*
- ☐ LC-MS *[A:632]*
- ☐ LC-MS/MS *[A:633]*
- ☐ other, please specify below *[A:384]*

☐ 15.1. Please specify *[Q:111408: TEXT]*

☐ 16. **LOQ for melamine in food simulant B?** * *[Q:111405: TEXT]*

☐ 17. **Sample preparation for formaldehyde?** * *[Q:111347: CHECKBOX]*

- ☐ 2,4-dinitrophenylhydrazine *[A:2412]*
- ☐ acetyl acetone (AA) *[A:2405]*
- ☐ chromotropic acid *[A:2407]*
- ☐ other, please specify below *[A:384]*

☐ 17.1. Please specify *[Q:111409: TEXT]*

☐ 18. **Analytical techniques for formaldehyde** * *[Q:111397: RADIO]*

- ○ HPLC-UV *[A:1389]*
- ○ HPLC/DAD *[A:2403]*
- ○ Other (please specify) *[A:1235]*
- ○ Spectrophotometry *[A:2413]*

☐ 18.1. Please specify * if parent A:1235 checked *[Q:111410: TEXT]*

☐ 19. **LOQ for formaldehyde in food simulant B?** * *[Q:111406: TEXT]*

☐ 20. **Time (in hours/days) between the migration experiments (MIG1, MIG2 and MIG3)** * *[Q:111348: TEXT]*

☐ 21. **surface to volume ratio applied to the test result** * *[Q:111407: TEXT]*

☐ 22. **Any problem encountered?** * *[Q:111349: TEXT]*

version: 2.2.8 (01/07/2021) [GEEL_01]

## Annex 4: Homogeneity and stability results

### A. Homogeneity study for test item 1, all values in mg kg$^{-1}$

| | test item 1 | | | |
|---|---|---|---|---|
| | Melamine | | Formaldehyde | |
| 1 | 2.52 | 2.53 | 12.51 | 12.53 |
| 2 | 2.52 | 2.51 | 12.55 | 12.52 |
| 3 | 2.53 | 2.52 | 12.52 | 12.50 |
| 4 | 2.54 | 2.52 | 12.44 | 12.52 |
| 5 | 2.52 | 2.52 | 12.50 | 12.46 |
| 6 | 2.52 | 2.53 | 12.55 | 12.53 |
| 7 | 2.53 | 2.52 | 12.49 | 12.56 |
| 8 | 2.53 | 2.52 | 12.52 | 12.51 |
| 9 | 2.52 | 2.51 | 12.54 | 12.55 |
| 10 | 2.52 | 2.51 | 12.54 | 12.52 |
| Mean | 2.52 | | 12.52 | |
| $u_{hom}$ | 0.12 % | | 0.11 % | |
| $\sigma_{pt}$ | 15 % | | 15 % | |
| 0.3 $\sigma_{pt}$ | 4.5 % | | 4.5 % | |
| $u_{hom}$ < 0.3 $\sigma_{pt}$ | passed | | passed | |

Where: $\sigma_{pt}$ is the standard deviation for the PT assessment,
$u_{hom}$ is the standard uncertainty contribution due to homogeneity

### B. Homogeneity study for test item 2, all values in mg kg$^{-1}$

| | Melamine | | | Formaldehyde | | |
|---|---|---|---|---|---|---|
| | MIG1 | MIG2 | MIG3 | MIG1 | MIG2 | MIG3 |
| **Mug 1** | 3.36 | 3.53 | 3.79 | 9.51 | 10.20 | 10.99 |
| **Mug 2** | 3.41 | 3.34 | 3.19 | 9.29 | 10.10 | 10.86 |
| **Mug 3** | 3.34 | 3.67 | 3.97 | 9.13 | 10.89 | 12.06 |
| **Mug 4** | 3.08 | 3.49 | 3.65 | 8.84 | 10.19 | 10.97 |
| **Mug 5** | 3.99 | 3.81 | 3.76 | 8.42 | 9.34 | 9.42 |
| **Mug 6** | 3.85 | 3.05 | 3.62 | 8.35 | 7.86 | 9.37 |
| **Mug 7** | 3.35 | 3.67 | 3.82 | 9.92 | 11.13 | 11.80 |
| **Mug 8** | 3.17 | 3.06 | 3.20 | 7.80 | 8.91 | 9.63 |
| **Mug 9** | 3.70 | 3.18 | 3.40 | 10.02 | 10.62 | 10.91 |
| **Mug 10** | 3.20 | 3.39 | 3.66 | 8.59 | 10.12 | 10.86 |
| Mean | 3.45 | 3.42 | 3.61 | 8.99 | 9.94 | 10.69 |
| RSD | 8.8% | 7.7% | 7.3% | 8.0% | 9.9% | 8.7% |

**C. Stability study at 40°C, 20°C and 4°C (RT) for one and/or 13 weeks** all values **in mg kg$^{-1}$ (REF, reference)**

| Melamine | | | | |
|---|---|---|---|---|
| T (°C) | **4 °C (Ref.)** | **40 °C** | **20 °C** | **4 °C** |
| weeks | **$T_0$** | **1** | **13** | **13** |
| | 2.52 | 2.53 | 2.52 | 2.53 |
| | 2.52 | 2.52 | 2.53 | 2.53 |
| | 2.53 | 2.51 | 2.53 | 2.53 |
| | 2.53 | | | |
| | 2.52 | | | |
| | 2.53 | | | |
| | 2.52 | | | |
| | 2.53 | | | |
| | 2.51 | | | |
| | 2.52 | | | |
| Mean | 2.52 | 2.52 | 2.53 | 2.53 |
| $\lvert Y_0-Y_{end}\rvert$ | | 0.001 | 0.003 | 0.007 |
| 0.3 $\sigma_{pt}$ | | 0.114 | | |
| Stable[a]? | | yes | yes | yes |

| Formaldehyde | | | | |
|---|---|---|---|---|
| T (°C) | **4 °C (Ref.)** | **40 °C** | **20 °C** | **4 °C** |
| weeks | **$T_0$** | **1** | **13** | **13** |
| | 12.52 | 12.52 | 12.65 | 12.54 |
| | 12.54 | 12.48 | 12.64 | 12.57 |
| | 12.50 | 12.50 | 12.66 | 12.59 |
| | 12.48 | | | |
| | 12.48 | | | |
| | 12.54 | | | |
| | 12.52 | | | |
| | 12.52 | | | |
| | 12.54 | | | |
| | 12.53 | | | |
| Mean | 12.52 | 12.50 | 12.65 | 12.56 |
| $\lvert Y_0-Y_{end}\rvert$ | | 0.019 | -0.132 | -0.046 |
| 0.3 $\sigma_{pt}$ | | 0.563 | | |
| Stable[a]? | | yes | yes | yes |

[a] Stability criteria according to ISO 13528:2022 § B.5. ($\lvert Y_0-Y_{end}\rvert < 0.3\ \sigma_{pt}$)

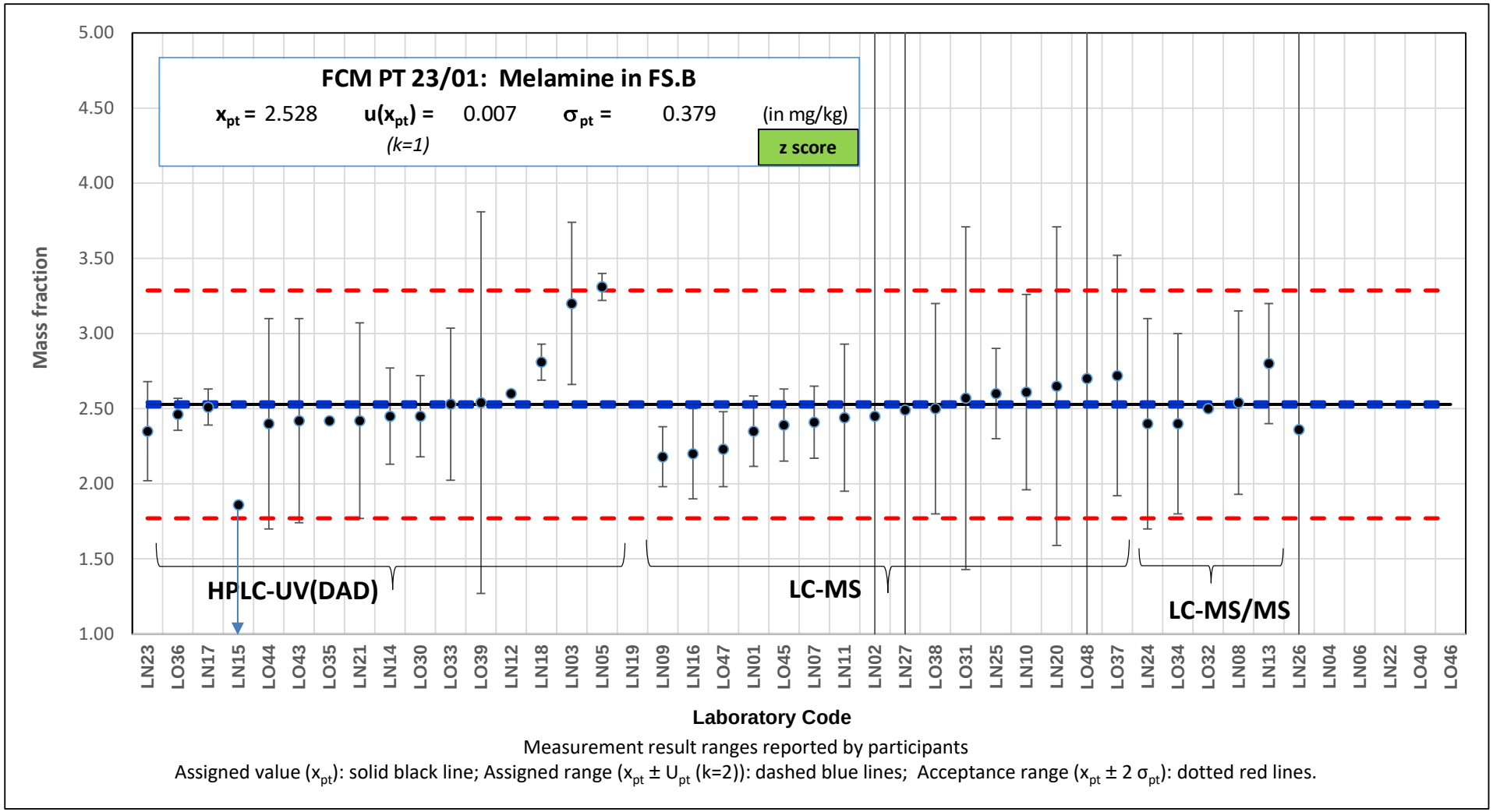
## Annex 5: Results for melamine in FS.B (test item 1)

Assigned range: $x_{pt}$ = 2.528 ± *0.0069 (k = 2 )*; $\sigma_{pt}$ = 0.379 , all values in mg kg$^{-1}$

| LabCode | $x_i$ | ± | $k$ | Technique | z score | ζ score | MU. |
|---|---|---|---|---|---|---|---|
| LN01 | 2.35 | 0.235 | 1.73 | LC-MS | -0.47 | -1.31 | a |
| LN02 | 2.45 | 21 | 2 | LC-MS | -0.21 | -0.01 | c |
| LN03 | 3.2 | 0.54 | 2 | HPLC-UV | 1.77 | 2.49 | a |
| LN04 | | | | | | | |
| LN05 | 3.31 | 0.09 | 2 | HPLC-UV | 2.06 | 17.18 | a |
| LN06 | | | | | | | |
| LN07 | 2.41 | 0.24 | 2 | LC-MS | -0.31 | -0.98 | a |
| LN08 | 2.54 | 0.61 | 2 | LC-MS/MS | 0.03 | 0.04 | a |
| LN09 | 2.18 | 0.2 | 2 | LC-MS | -0.92 | -3.47 | a |
| LN10 | 2.61 | 0.65 | 2 | LC-MS | 0.22 | 0.25 | a |
| LN11 | 2.44 | 0.49 | 2 | LC-MS | -0.23 | -0.36 | a |
| LN12 | 2.6 | | | HPLC-UV | 0.19 | 10.51 | np |
| LN13 | 2.8 | 0.4 | 2 | LC-MS/MS | 0.72 | 1.36 | a |
| LN14 | 2.45 | 0.32 | 2 | HPLC-UV | -0.21 | -0.49 | a |
| LN15 | < 1.86 | | | HPLC-UV | | | |
| LN16 | 2.2 | 0.3 | 2 | LC-MS | -0.86 | -2.18 | a |
| LN17 | 2.51 | 0.12 | 2 | HPLC-DAD | -0.05 | -0.30 | a |
| LN18 | 2.81 | 0.12 | 2 | HPLC-UV | 0.74 | 4.67 | a |
| LN19 | 0.026 | 0.0005 | 2 | LC-MS | -6.60 | -364.40 | a |
| LN20 | 2.65 | 1.06 | 2 | LC-MS | 0.32 | 0.23 | c |
| LN21 | 2.42 | 0.65 | 1.73 | HPLC-UV | -0.28 | -0.29 | c |
| LN22 | | | | | | | |
| LN23 | 2.35 | 0.33 | 2 | HPLC-DAD | -0.47 | -1.08 | a |
| LN24 | 2.4 | 0.7 | 2 | LC-MS/MS | -0.34 | -0.37 | a |
| LN25 | 2.6 | 0.3 | 2 | LC-MS | 0.19 | 0.48 | a |
| LN26 | 2.36 | 21 | 1.73 | | -0.44 | -0.01 | c |
| LN27 | 2.49 | 30 | 2 | LC-MS | -0.10 | 0.00 | c |
| LO30 | 2.45 | 0.27 | 2 | HPLC-UV | -0.21 | -0.58 | a |
| LO31 | 2.57 | 1.14 | 1.64 | LC-MS | 0.11 | 0.06 | c |
| LO32 | 2.5 | | | LC-MS/MS | -0.07 | -4.07 | np |
| LO33 | 2.53 | 0.506 | 2 | HPLC-UV | 0.01 | 0.01 | a |
| LO34 | 2.4 | 0.6 | 2 | LC-MS/MS | -0.34 | -0.43 | a |
| LO35 | 2.42 | | | HPLC-UV | -0.28 | -15.73 | np |
| LO36 | 2.462 | 0.106 | 2 | HPLC-DAD | -0.17 | -1.23 | a |
| LO37 | 2.72 | 0.8 | 2 | LC-MS | 0.51 | 0.48 | a |
| LO38 | 2.5 | 0.7 | 2 | LC-MS | -0.07 | -0.08 | a |
| LO39 | 2.54 | 1.27 | 2 | HPLC-UV | 0.03 | 0.02 | c |
| LO40 | | | | | | | |
| LO43 | 2.42 | 0.68 | 2 | HPLC-UV | -0.28 | -0.32 | a |
| LO44 | 2.4 | 0.7 | 2 | HPLC-UV | -0.34 | -0.37 | a |
| LO45 | 2.39 | 0.24 | 2 | LC-MS | -0.36 | -1.15 | a |
| LO46 | | | | | | | |
| LO47 | 2.23 | 0.25 | 2 | LC-MS | -0.79 | -2.38 | a |
| LO48 | 2.7 | 20 | 2 | LC-MS | 0.45 | 0.02 | c |

Performance (z, ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU – "a" $u(x_{pt,rel}) \leq u(x_{i,rel}) \leq \sigma_{pt,rel}$ ; "b" $u(x_{i,rel}) < u(x_{pt,rel})$; "c" $u(x_{i,rel}) > \sigma_{pt,rel}$;"np" not provided




Measurement result ranges 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.

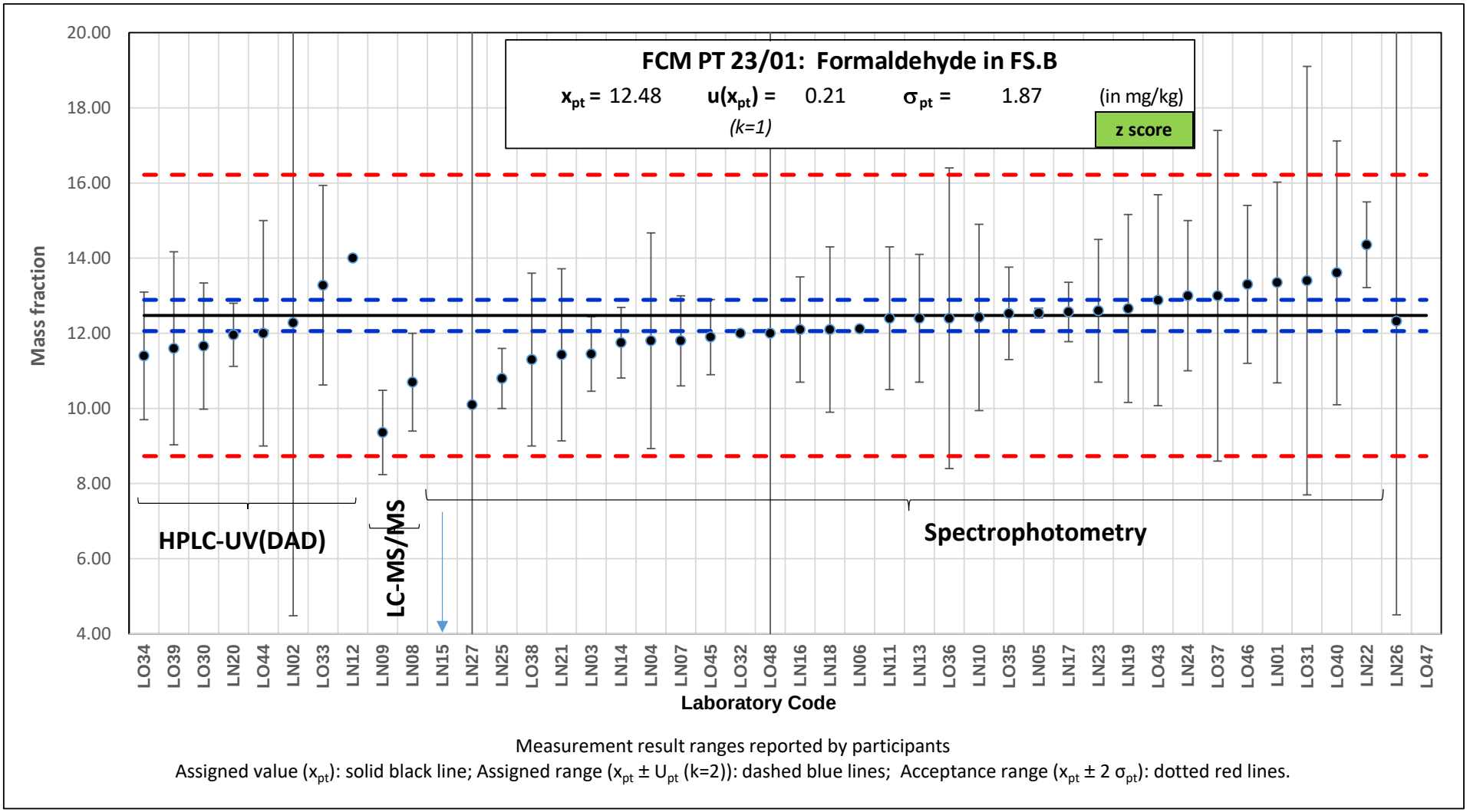
## Annex 6: Results for formaldehyde in FS.B (test item 1)

Assigned range: $x_{pt}$ = 12.48 ± 0.41 ($k$ = 2); $\sigma_{pt}$ = 1.87 (all values in mg kg$^{-1}$)

| LabCode | $x_i$ | ± | $k$ | Technique | z score | ζ score | MU. |
|---|---|---|---|---|---|---|---|
| LN01 | 13.35 | 2.67 | 1.73 | Spectroph. | 0.47 | 0.56 | a |
| LN02 | 12.28 | 7.8 | 2 | HPLC-UV | -0.10 | -0.05 | c |
| LN03 | 11.45 | 0.99 | 2 | Spectroph. | -0.55 | -1.91 | a |
| LN04 | 11.8 | 2.87 | 2 | Spectroph. | -0.36 | -0.47 | a |
| LN05 | 12.54 | 0.13 | 2 | Spectroph. | 0.03 | 0.30 | b |
| LN06 | 12.12 | 0.03 | 2 | Spectroph. | -0.19 | -1.71 | b |
| LN07 | 11.8 | 1.2 | 2 | Spectroph. | -0.36 | -1.06 | a |
| LN08 | 10.7 | 1.3 | 2 | LC-MS/MS | -0.95 | -2.60 | a |
| LN09 | 9.36 | 1.12 | 2 | LC-MS | -1.66 | -5.22 | a |
| LN10 | 12.42 | 2.48 | 2 | Spectroph. | -0.03 | -0.04 | a |
| LN11 | 12.4 | 1.9 | 2 | Spectroph. | -0.04 | -0.08 | a |
| LN12 | 14 | | | HPLC-UV | 0.81 | 7.37 | np |
| LN13 | 12.4 | 1.7 | 2 | Spectroph. | -0.04 | -0.09 | a |
| LN14 | 11.75 | 0.94 | 2 | Spectroph. | -0.39 | -1.41 | a |
| LN15 | 1.15 | 0.23 | 2 | Spectroph. | -6.05 | -47.85 | a |
| LN16 | 12.1 | 1.4 | 2 | Spectroph. | -0.20 | -0.51 | a |
| LN17 | 12.57 | 0.79 | 2 | Spectroph. | 0.05 | 0.21 | a |
| LN18 | 12.1 | 2.2 | 2 | Spectroph. | -0.20 | -0.34 | a |
| LN19 | 12.66 | 2.5 | 2 | Spectroph. | 0.10 | 0.15 | a |
| LN20 | 11.96 | 0.84 | 2 | HPLC-UV | -0.28 | -1.10 | a |
| LN21 | 11.43 | 2.29 | 1.73 | Spectroph. | -0.56 | -0.78 | a |
| LN22 | 14.357 | 1.14 | 2 | Spectroph. | 1.01 | 3.10 | a |
| LN23 | 12.6 | 1.9 | 2 | Spectroph. | 0.07 | 0.13 | a |
| LN24 | 13 | 2 | 2 | Spectroph. | 0.28 | 0.51 | a |
| LN25 | 10.8 | 0.8 | 2 | Spectroph. | -0.90 | -3.72 | a |
| LN26 | 12.32 | 7.81 | 1.73 | | -0.08 | -0.03 | c |
| LN27 | 10.1 | 10 | 2 | Spectroph. | -1.27 | -0.47 | c |
| LO30 | 11.66 | 1.68 | 2 | HPLC-UV | -0.44 | -0.94 | a |
| LO31 | 13.4 | 5.7 | 1.64 | Spectroph. | 0.49 | 0.27 | c |
| LO32 | 12 | | | Spectroph. | -0.25 | -2.30 | np |
| LO33 | 13.28 | 2.656 | 2 | HPLC-UV | 0.43 | 0.60 | a |
| LO34 | 11.4 | 1.7 | 2 | HPLC-DAD | -0.57 | -1.23 | a |
| LO35 | 12.53 | 1.23 | 2 | Spectroph. | 0.03 | 0.08 | a |
| LO36 | 12.4 | 4 | 2 | Spectroph. | -0.04 | -0.04 | c |
| LO37 | 13 | 4.4 | 2 | Spectroph. | 0.28 | 0.24 | c |
| LO38 | 11.3 | 2.3 | 2 | Spectroph. | -0.63 | -1.01 | a |
| LO39 | 11.6 | 2.57 | 2 | HPLC-UV | -0.47 | -0.67 | a |
| LO40 | 13.61 | 3.51 | 2 | Spectroph. | 0.61 | 0.64 | a |
| LO43 | 12.88 | 2.81 | 2 | Spectroph. | 0.22 | 0.29 | a |
| LO44 | 12 | 3 | 2 | HPLC-UV | -0.25 | -0.31 | a |
| LO45 | 11.9 | 1 | 2 | Spectroph. | -0.31 | -1.06 | a |
| LO46 | 13.3 | 2.1 | 2 | Spectroph. | 0.44 | 0.77 | a |
| LO47 | | | | | | | |
| LO48 | 12 | 10.3 | 2 | Spectroph. | -0.25 | -0.09 | c |

Performance (z, ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU – "a" $u(x_{pt,rel}) \leq u(x_{i,rel}) \leq \sigma_{pt,rel}$; "b" $u(x_{i,rel}) < u(x_{pt,rel})$; "c" $u(x_{i,rel}) > \sigma_{pt,rel}$; "np" not provided

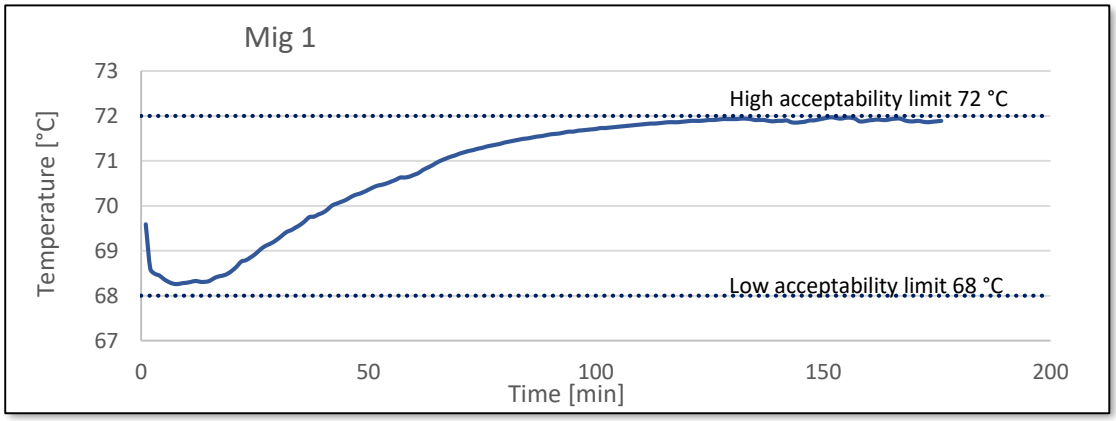


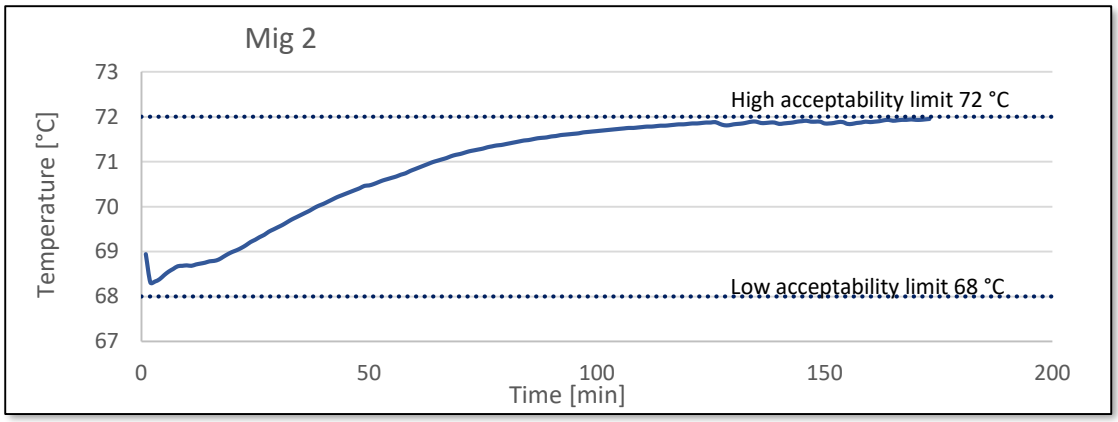

Measurement result ranges reported by participants

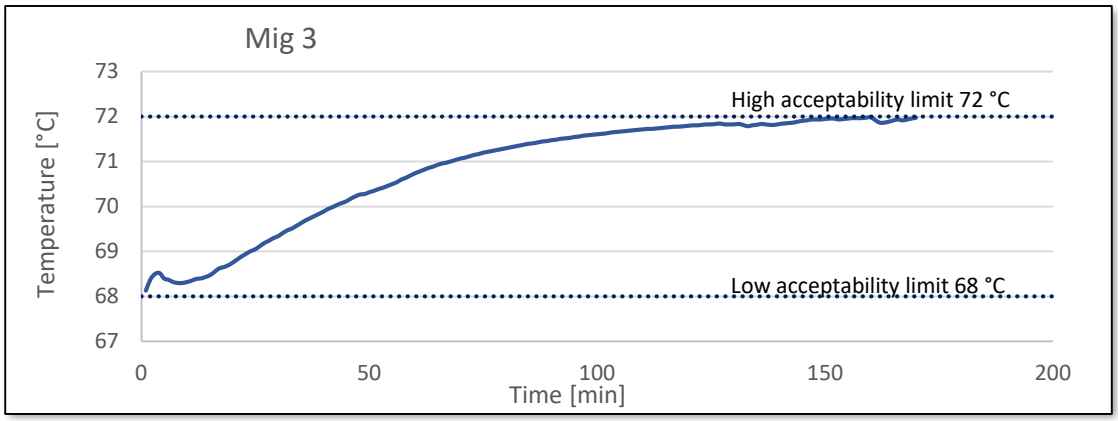
Assigned value ($x_{pt}$): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

## Annex 7: Repeated temperature profiles

(3 consecutive migrations between 68 and 72 °C (Profile 1) performed by the EURL on a mug (test item 2)

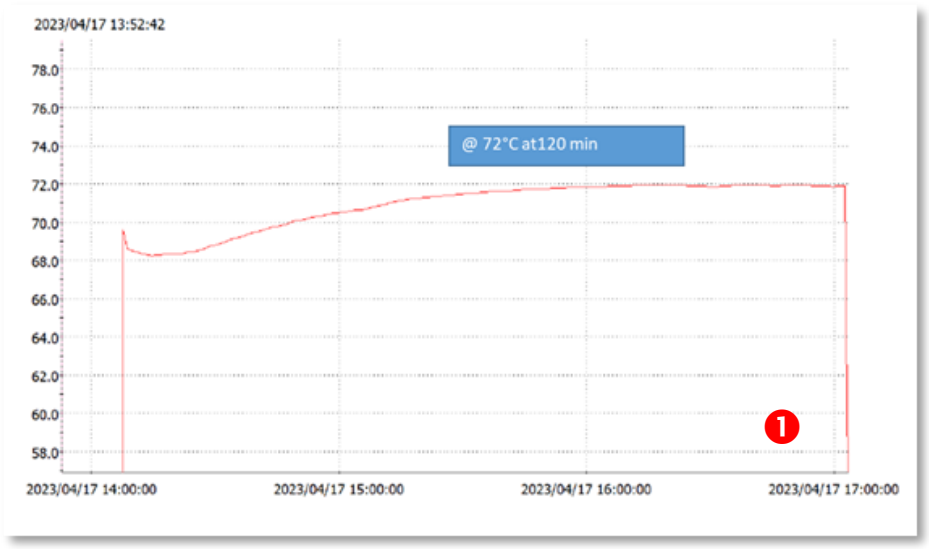
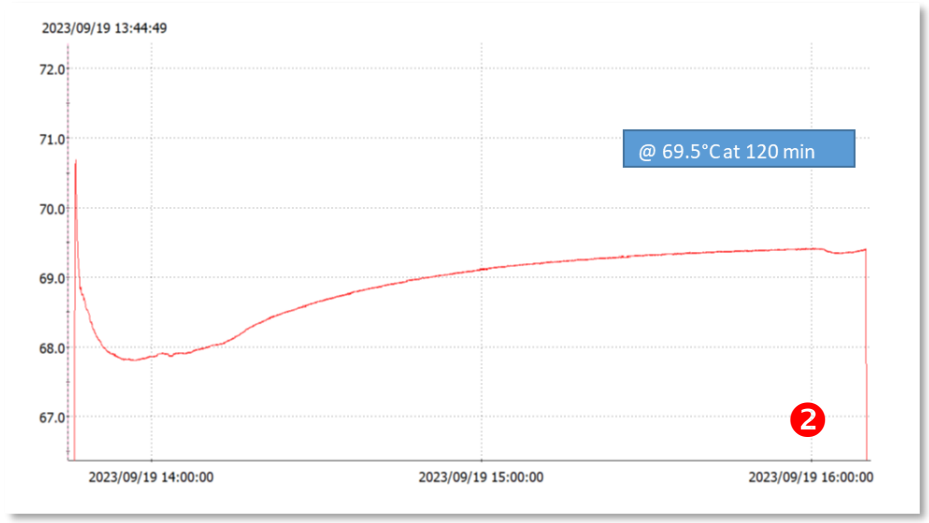
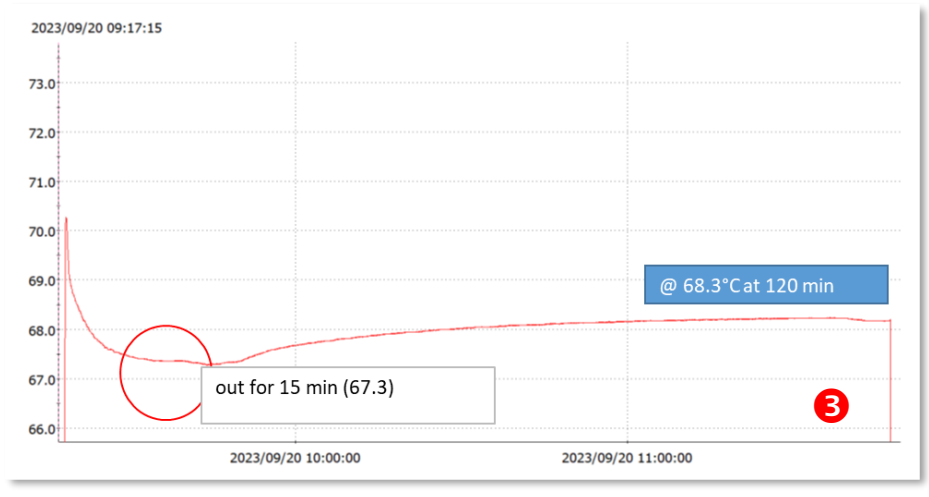


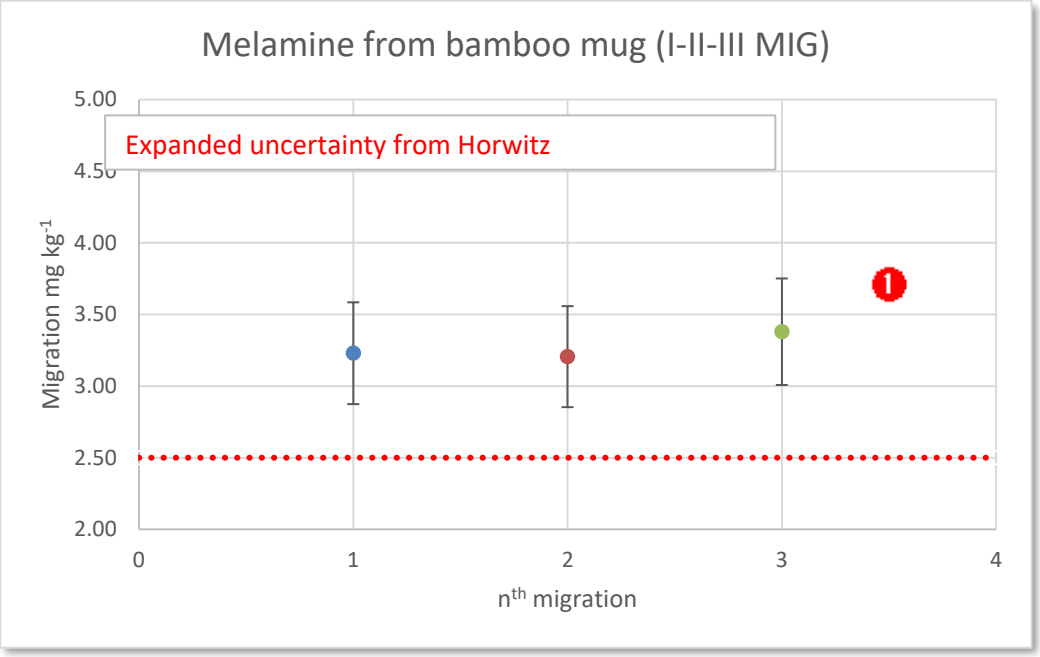
## Annex 8: Three different temperature profiles

within the recommended temperature range of 70 ± 2 °C, obtained by the EURL for different mugs (test item 2)

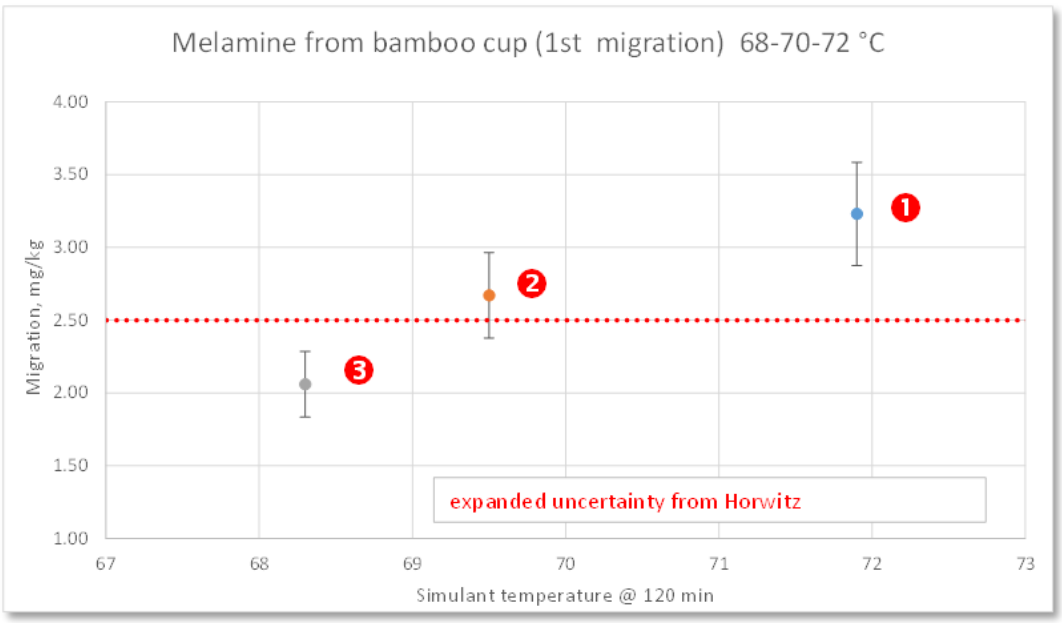
| | |
|---|---|
|  | **68 ≤ T ≤ 72 °C** |
|  | **67.8 ≤ T ≤ 69.5 °C** |
|  | **67.5 ≤ T ≤ 68.3 °C** |

## Annex 9: Melamine results obtained by the EURL

**A.** Three consecutive migrations performed applying the same temperature profile (see Annex 8, Profile 1). All migrations are above the SML; while the reported ranges have overlapping uncertainties, demonstrating the stability of the test item.

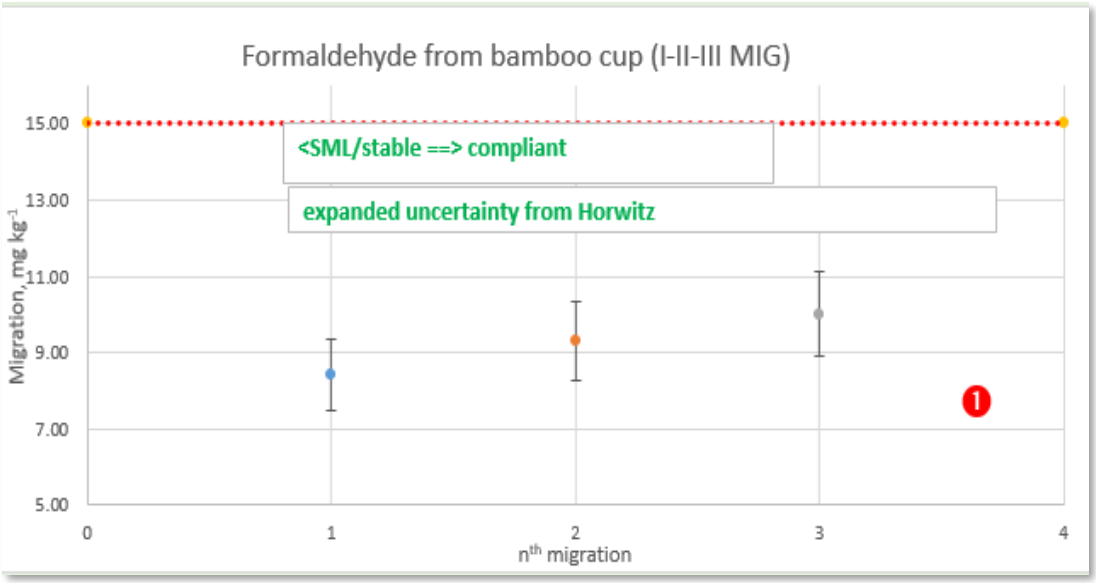



**B.** First migrations of three different mugs performed at three different temperature profiles (see Annex 8, Profiles 1, 2 and 3). Migrated levels increase with increasing temperature ranges; those obtained for temperatures ranging from 67.5 and 68.3 °C, are below the SML

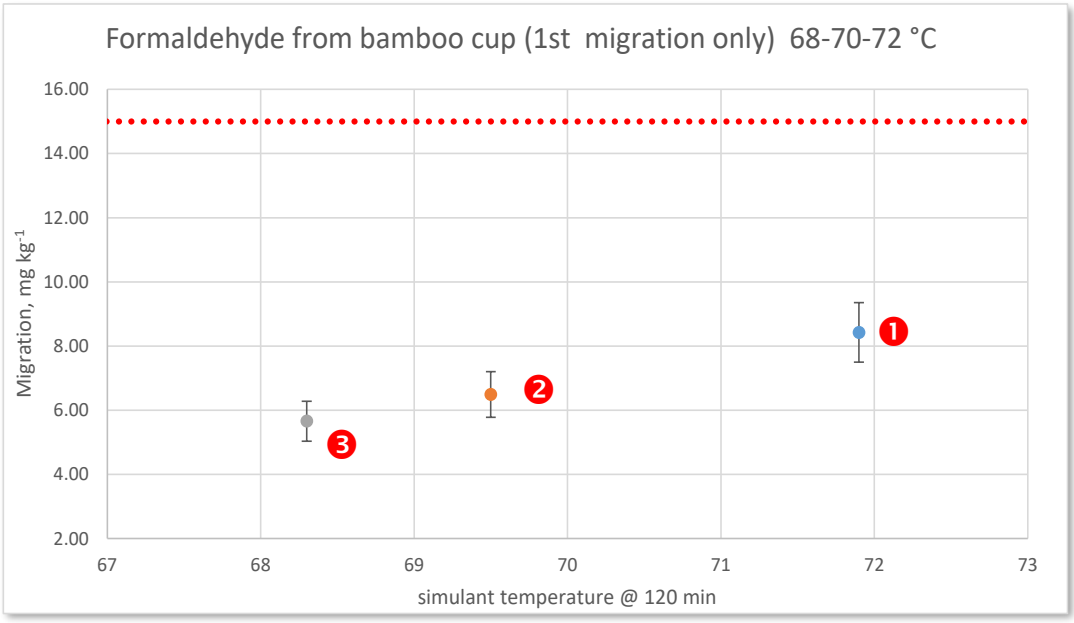


## Annex 10: Formaldehyde migration results obtained by the EURL

**A.** Three consecutive migrations performed applying the same temperature profile (see Annex 7, Profile 1). All migrations are below the SML; and the reported ranges have overlapping uncertainties, demonstrating the stability of the test item.




**B.** First migrations of three different mugs performed at three different temperature profiles (see Annex 8, Profiles 1, 2 and 3). Migrated levels increase with increasing temperature ranges.



## Annex 11: Reported results for three consecutive migrations (m1, m2, m3) of melamine in food simulant B from test item 2 (MUG), expressed in mg kg$^{-1}$

| | MUG 1 | | | | | | MUG 2 | | | | | | MUG 3 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | U(m1) | | Rsu | | | | U(m1) | | Rsu | | | | U(m1) | | RSu | | | | |
| Lab | m1 | m2 | m3 | k=2 | k | k=1 | m1 | m2 | m3 | k=2 | k | k=1 | m1 | m2 | m3 | k=2 | k | k=1 | Lab statement | Stable | < SML | coherent | T profile |
| LN01 | 2.41 | 2.43 | 2.72 | 0.241 | 1.73 | 5.8% | 2.05 | 2.11 | 2.26 | 0.205 | 1.73 | 5.8% | 2.59 | 2.83 | 2.9 | 0.259 | 1.73 | 5.8% | NC | yes | no | yes | S |
| LN02 | 2.24 | 2.31 | 2.37 | 0.235 | 1 | 10.5% | 2.41 | 2.41 | 2.2 | 0.253 | 1 | 10.5% | 1.9 | 2.3 | 2.39 | 0.200 | 1 | 10.5% | C | yes | yes | yes | Q |
| LN03 | 1.09 | 1 | 0.85 | 0.17 | 2 | 7.8% | 1.16 | 1.53 | 1.07 | 0.21 | 2 | 9.1% | 1.05 | 1.18 | 0.91 | 0.17 | 2 | 8.1% | C | yes | yes | yes | U |
| LN04 | -- | | | | | | | | | | | | | | | | | | not tested | | | | U |
| LN05 | 2.803 | 2.826 | 3.528 | np | | | 3.086 | 3.748 | 3.424 | np | | | 4.037 | 3.047 | 3.822 | np | | | NC | ? | ? | ? | S |
| LN06 | -- | | | | | | | | | | | | | | | | | | not tested | | | | S |
| LN07 | 2.13 ↗ | 2.63 | 2.6 | 0.26 | 2 | 6.1% | 2.05 | 2.2 | 2.01 | 0.25 | 2 | 6.1% | 1.95 | 2.33 | 2.01 | 0.23 | 2 | 5.9% | NC | no | yes | yes | S |
| LN08 | 2.07 | 1.88 ↗ | 2.96 | 0.5 | 2 | 12.1% | 1.4 | 1.46 ↗ | 2.45 | 0.34 | 2 | 12.1% | 1.82 | 2.02 | 2.8 | 0.44 | 2 | 12.1% | NC | no | yes | yes | Q |
| LN09 | 1.25 | 0.63 ↗ | 0.73 | 0.08 | 2 | 3.2% | 1.07 | 0.88 | 0.99 | 0.09 | 2 | 4.2% | 0.99 | 0.71 ↗ | 0.9 | 0.08 | 2 | 4.0% | C | no | yes | no | U |
| LN10 | 1.6 | 1.6 | 1.9 | 0.41 | 2 | 12.8% | 2.3 | 2 | 2.3 | 0.58 | 2 | 12.6% | 1.6 | 1.3 | 1.5 | 0.4 | 2 | 12.5% | C | yes | yes | yes | U |
| LN11 | 2.12 | 2.62 | 3 | 0.43 | 2 | 10.1% | 2.61 | 2.62 | 3.2 | 0.53 | 2 | 10.2% | 1.82 | 1.8 | 2.13 | 0.37 | 2 | 10.2% | NC | yes | no | yes | U |
| LN12 | 1.4 | 2.2 | 1.8 | np | | | 1.6 | 1.6 | 2.1 | np | | | 1.9 | 1.6 | 2 | np | | | C | ? | yes | ? | Q |
| LN13 | 3.4 | 3.3 | 3.6 | 1 | 2 | 15% | 3.1 | 2.7 | 3.2 | 0.9 | 2 | 15% | 3.8 | 3.3 | 3.5 | 1.1 | 2 | 14.5% | NC | yes | no | yes | S |
| LN14 | 2.76 | 2.43 | 2.82 | 0.75 | 2 | 13.6% | 3.24 | 2.97 | 3.22 | 0.86 | 2 | 13.3% | 3.17 | 3.1 | 3.36 | 0.85 | 2 | 13.4% | NC | yes | no | yes | Q |
| LN15 | < 1.86 | < 1.86 | < 1.86 | np | | | < 1.86 | < 1.86 | < 1.86 | np | | | < 1.86 | < 1.86 | < 1.86 | np | | | NC | yes | yes | no | S |
| LN16 | 1.4 | 0.4 | < 0.25 | 0.2 | 2 | 7.1% | 0.9 | 0.3 | < 0.25 | 0.1 | 2 | 5.6% | 1 | 0.4 | 0.3 | 0.1 | 2 | 5.0% | C | yes | yes | yes | |
| LN17 | 0.95 | 0.35 ↗ | 1.37 | 0.05 | 2 | 2.6% | 1.05 | 0.28 ↗ | 1.48 | 0.06 | 2 | 2.9% | 0.83 | 0.58 ↗ | 1.28 | 0.05 | 2 | 3.0% | NC | no | yes | yes | Q |
| LN18 | 3.39 | 3.17 | 2.89 | 0.3 | 2 | 4.4% | 3.37 | 3.21 | 2.81 | 0.29 | 2 | 4.3% | 3.68 | 3.07 | 2.74 | 0.32 | 2 | 4.3% | NC | yes | no | yes | S |
| LN19 | 0.92 | 0.78 | 0.63 | 0.19 | 2 | 10.3% | 0.77 | 1.06 | 0.84 | 0.15 | 2 | 9.7% | 1.27 | 0.96 | 0.74 | 0.25 | 2 | 9.8% | C | yes | yes | yes | |
| LN20 | 1.05 | 1.25 | 1.24 | 0.42 | 2 | 20% | 1.84 | 2.15 | 2.09 | 0.74 | 2 | 20% | 1.72 | 1.91 | 1.99 | 0.69 | 2 | 20% | C | yes | yes | yes | S |
| LN21 | 4.39 | 2.85 | 3.51 | 0.97 | 1.73 | 12.8% | 3.11 | 2.44 | 4.2 | 0.88 | 1.73 | 16.4% | 5.36 | 4.54 | 5.45 | 1.38 | 1.73 | 15% | NC | yes | no | yes | U |
| LN22 | -- | | | | | | | | | | | | | | | | | | not tested | | | | |
| LN23 | 1.85 | 1.61 ↗ | 2.38 | 0.26 | 2 | 7.0% | 1.41 | 1.84 | 1.83 | 0.28 | 2 | 9.9% | 1.86 | 2.1 ↗ | 2.8 | 0.3 | 2 | 8.1% | NC | no | yes | yes | S |
| LN24 | 1.8 | 1.6 | 2.4 | 0.6 | 2 | 17% | 2.5 | 2.8 | 2.6 | 0.9 | 2 | 18% | 1.3 | 2.3 | 1.9 | 0.5 | 2 | 19% | C | yes | yes | yes | S |
| LN25 | 2.8 | 2.5 | 2.9 | 0.3 | 2 | 5.4% | 2.5 | 2.4 | 2.6 | 0.2 | 2 | 4.0% | 2.9 | 2.3 | 2.1 | 0.3 | 2 | 5.2% | NC | yes | no | yes | S |
| LN26 | 0.69 ↗ | 2.16 | 2.34 | 0.07 | 1.73 | 6.1% | 0.77 ↗ | 1.89 ↗ | 2.63 | 0.08 | 1.73 | 6.1% | 0.54 ↗ | 2.43 | 2.43 | 0.06 | 1.73 | 6.1% | NC | no | yes | yes | S |
| LN27 | 3.23 | 2.88 | 2.95 | 0.969 | 2 | 15% | 1.75 | 2.08 | 2.08 | 0.525 | 2 | 15.0% | 2.71 | 3.2 | 3.12 | 0.813 | 2 | 15.0% | C | yes | yes | yes | S |
| LO30 | 1.63 ↗ | 2.71 | 2.87 | 0.18 | 2 | 5.5% | 1.09 ↗ | 2.02 | 2.01 | 0.12 | 2 | 5.5% | 2.02 ↗ | 2.77 | 2.88 | 0.22 | 2 | 5.4% | NC | no | no | yes | S |
| LO31 | 1.7 | 1.9 | 2.42 | 0.75 | 1.64 | 27% | 2.31 | 2.52 | 0.92 | 1.03 | 1.64 | 27% | 2.07 | 1.55 | 1.61 | 0.92 | 1.64 | 27% | C | yes | yes | yes | S |
| LO32 | 3.3 | 2.3 | 2.9 | np | | | 3.4 | 3.8 | 3.6 | np | | | 2.3 | 3.7 | 3.5 | np | | | NC | ? | ? | ? | S |
| LO33 | 1.75 | 2.15 | 1.71 | np | | | 1.54 | 2.01 | 1.5 | np | | | 1.03 | 1.47 | 1.64 | np | | | C | ? | yes | ? | S |
| LO34 | 2.2 | 2.6 | 3.1 | 0.6 | 2 | 13.6% | 2.2 | 2.3 | 2.8 | 0.6 | 2 | 13.6% | 2.5 | 2.4 | 2.6 | 0.6 | 2 | 12.0% | C | yes | yes | yes | S |
| LO35 | 1.55 | 1.82 | 1.76 | np | | | 1.86 | 1.64 | 1.94 | np | | | 1.52 | 1.32 | 1.39 | np | | | C | ? | yes | ? | Q |
| LO36 | 1.559 | 1.717 | 1.919 | 0.67 | 2 | 21% | 1.718 | 1.553 | 1.67 | 0.739 | 2 | 22% | 1.557 | 1.67 | 1.869 | 0.67 | 2 | 22% | C | yes | yes | yes | U |
| LO37 | 2.03 | 1.73 | 2.35 | 0.6 | 2 | 15% | 2.22 | 2.2 | 2.99 | 0.65 | 2 | 15% | 1.52 | 1.68 | 2.24 | 0.45 | 2 | 15% | NC | yes | yes | no | Q |
| LO38 | 0.6 | 0.5 | 0.5 | 0.2 | 2 | 17% | 0.8 | 0.9 | 0.7 | 0.3 | 2 | 19% | 1.2 | 1 | 0.9 | 0.4 | 2 | 17% | C | yes | yes | yes | Q |
| LO39 | 1.03 | 0.94 | 1.42 | 0.52 | 2 | 25% | 1.18 | 1.12 | 1.26 | 0.59 | 2 | 25% | 1.72 | 1.38 | 1.48 | 0.86 | 2 | 25% | NC | yes | yes | no | S |
| LO40 | -- | | | | | | | | | | | | | | | | | | not tested | | | | Q |
| LO43 | 2.21 | 1.93 | 1.88 | 0.63 | 2 | 14.3% | 1.56 | 1.88 | 1.79 | 0.47 | 2 | 15% | 1.32 ↗ | 2.39 | 2.27 | 0.41 | 2 | 16% | NC | no | yes | yes | S |
| LO44 | 0.9 | 0.8 | 1.3 | 0.3 | 2 | 17% | 0.9 | 0.9 | 1.3 | 0.3 | 2 | 17% | 0.8 | 1 | 1 | 0.2 | 2 | 12.5% | NC | yes | yes | no | Q |
| LO45 | 1.77 | 1.8 | 1.8 | 0.18 | 2 | 5.1% | 1.29 | 1.23 | 1.34 | 0.13 | 2 | 5.0% | 1.8 | 1.83 | 1.97 | 0.19 | 2 | 5.3% | C | yes | yes | yes | S |
| LO46 | -- | | | | | | | | | | | | | | | | | | not tested | | | | |
| LO47 | 4.5 | 3.09 | 2.76 | 0.49 | 2 | 5.4% | 4.89 ↗ | 13.39 | 5.26 | 0.54 | 2 | 5.5% | 4.3 ↗ | 5.77 | 2.5 | 0.47 | 2 | 5.5% | NC | no | no | yes | S |
| LO48 | 1.2 | 1.6 ↗ | 2.9 | 0.24 | 2 | 10.0% | 1.1 ↗ | 1.8 | 2.5 | 0.22 | 2 | 10.0% | 1.3 ↗ | 2.3 | 2.8 | 0.26 | 2 | 10.0% | NC | no | yes | yes | S |

## Annex 12: Reported results for three consecutive migrations (m1, m2, m3) of formaldehyde in food simulant B from test item 2 (MUG), expressed in mg kg$^{-1}$

| | MUG 1 | | | | | | MUG 2 | | | | | | MUG 3 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | m1 | m2 | m3 | U(m1) k=2 | k | RSD k=1 | m1 | m2 | m3 | U(m1) k=2 | k | RSD k=1 | m1 | m2 | m3 | U(m1) k=2 | k | RSD k=1 | Lab statement | Stable | < SML | coherent | T profile |
| LN01 | 8 | 9.28 | 10.12 | 1.6 | 1.73 | 11.6% | 6.77 | 7.68 | 8.64 | 1.35 | 1.73 | 11.5% | 7.44 | 8.92 | 9.57 | 1.49 | 1.73 | 11.6% | C | yes | yes | yes | S |
| LN02 | 6.54 ↗ | 7.9 | 7.9 | 0.26 | 1 | 3.9% | 9.45 ↗ | 10.81 | 10.62 | 0.37 | 1 | 3.9% | 7.99 ↗ | 9.78 | 10.43 | 0.31 | 1 | 3.9% | NC | no | yes | yes | Q |
| LN03 | 3.22 | 2.48 | 2.4 | 0.39 | 2 | 6.1% | 2.62 ↗ | 4.21 | 2.58 | 0.46 | 2 | 8.8% | 2.6 ↗ | 3.47 | 1.79 | 0.38 | 2 | 7.3% | C | no | yes | no | U |
| LN04 | 1.686 | 1.548 ↗ | 2.014 | 0.257 | 2 | 7.6% | 1.329 | 1.334 | 1.765 | 0.257 | 2 | 9.7% | 1.828 | 1.231 ↗ | 1.889 | 0.257 | 2 | 7.0% | NC | no | yes | yes | U |
| LN05 | 3.686 | 7.334 | 7.131 np | | | | 5.826 | 11.744 | 9.904 np | | | | 6.892 | 11.563 | 9.941 np | | | | NC | ? | yes | ? | S |
| LN06 | 3.945 | 7.423 | 7.357 np | | | | 5.716 | 12.61 | 10.026 np | | | | 6.177 | 10.966 | 10.639 np | | | | NC | ? | yes | ? | S |
| LN07 | 6.2 ↗ | 8.7 | 9.3 | 0.8 | 2 | 6.5% | 6 ↗ | 7.9 | 8 | 0.8 | 2 | 6.7% | 5.5 ↗ | 7.6 | 7.5 | 0.7 | 2 | 6.4% | NC | no | yes | yes | S |
| LN08 | 6.71 | 7.81 ↗ | 11.2 | 0.8 | 2 | 6.0% | 3.52 ↗ | 4.33 ↗ | 6.43 | 0.42 | 2 | 6.0% | 6.28 | 7.47 ↗ | 9.62 | 0.75 | 2 | 6.0% | NC | no | yes | yes | Q |
| LN09 | 3.29 | 2.24 ↗ | 3.92 | 0.38 | 2 | 5.8% | 3.48 | 3.71 | 3.5 | 0.43 | 2 | 6.2% | 2.34 | 2.41 | 2.8 | 0.3 | 2 | 6.4% | C | no | yes | no | U |
| LN10 | 5.7 | 6 | 3.6 | 1.1 | 2 | 9.6% | 6.9 | 6.7 | 4.4 | 1.4 | 2 | 10.1% | 7.7 | 7.3 | 5 | 1.5 | 2 | 9.7% | C | yes | yes | yes | U |
| LN11 | 7.4 ↗ | 10.1 | 10.8 | 1.2 | 2 | 8.1% | 8 | 9.2 | 6.3 | 1.2 | 2 | 7.5% | 4.6 | 5.7 ↗ | 9.8 | 0.7 | 2 | 7.6% | NC | no | yes | yes | U |
| LN12 | 3.2 | 5.3 | 6.6 np | | | | 5.2 | 5.7 | 6 np | | | | 6.1 | 5.9 | 6.8 np | | | | C | ? | yes | | Q |
| LN13 | 10.9 | 11.4 | 11.7 | 3.1 | 2 | 14% | 8.1 | 8.3 | 8.4 | 2.3 | 2 | 14% | 11.4 | 12.2 | 12.2 | 3.3 | 2 | 14% | C | yes | yes | yes | S |
| LN14 | 8.2 | 9.75 | 10.8 | 1.9 | 2 | 11.6% | 7.12 | 7.86 | 8.69 | 1.69 | 2 | 11.9% | 9.3 | 10.95 | 11.75 | 2.12 | 2 | 11.4% | C | yes | yes | yes | Q |
| LN15 | 1.73 | 1.8 | 1.82 | 0.36 | 2 | 10.4% | 1.62 | 1.89 | 1.56 | 0.34 | 2 | 10.5% | 1.64 | 1.95 | 1.64 | 0.35 | 2 | 10.7% | NC | yes | yes | no | S |
| LN16 | 4.6 | < 3 | < 3 | 0.5 | 2 | 5.4% | 3.1 | < 3 | < 3 | 0.4 | 2 | 6.5% | < 3 | < 3 | < 3 | | | | C | yes | yes | yes | |
| LN17 | 3.9 | 2.35 ↗ | 6.38 | 0.53 | 2 | 6.8% | 3.9 | 1.88 ↗ | 6.53 | 0.53 | 2 | 6.8% | 3.43 | 3.59 ↗ | 6.22 | 0.53 | 2 | 7.7% | NC | no | yes | yes | Q |
| LN18 | 12.6 | 13.8 | 12.6 | 2.5 | 2 | 9.9% | 11.4 | 12.6 | 11.4 | 2.3 | 2 | 10.1% | 9.6 | 10.8 | 9.6 | 1.9 | 2 | 9.9% | C | yes | yes | yes | S |
| LN19 | 1.78 ↗ | 2.54 | 2.81 | 0.35 | 2 | 9.8% | 2.66 ↗ | 5.45 | 3.51 | 0.52 | 2 | 9.8% | 2.44 ↗ | 4.46 | 2.89 | 0.48 | 2 | 9.8% | C | no | yes | no | |
| LN20 | 2.83 | 4.11 | 4.28 | 0.85 | 2 | 15% | 6.44 | 8.77 | 8.72 | 1.93 | 2 | 15% | 5.85 | 8.49 | 8.16 | 1.76 | 2 | 15% | C | yes | yes | yes | S |
| LN21 | 9.33 | 8.69 | 9.89 | 1.86 | 1.73 | 11.5% | 6.44 | 7.48 | 11.2 | 1.67 | 1.73 | 15.0% | 13.91 | 15.18 | 16.49 | 3.04 | 1.73 | 12.6% | C | yes | yes | yes | U |
| LN22 | 23.10 | 11.44 | 8.51 | 1.14 | 2 | 2.5% | 11.44 | 4.81 ↗ | 8.38 | 1.14 | 2 | 5.0% | 15.28 | 11.31 | 8.63 | 1.14 | 2 | 3.7% | -- | no | yes | | U |
| LN23 | 9.6 | 10.5 | 11.3 | 3.6 | 2 | 19% | 7.4 | 8.2 | 8.8 | 3.5 | 2 | 24% | 10.2 | 10.9 | 12.4 | 4.6 | 2 | 23% | NC | yes | yes | no | S |
| LN24 | 4.6 | 5.8 | 4.9 | 1.1 | 2 | 12.0% | 8.1 ↗ | 12 | 11 | 1.9 | 2 | 11.7% | 3 ↗ | 7.8 | 5.8 | 0.72 | 2 | 12.0% | NC | no | yes | yes | S |
| LN25 | 8.6 | 8.6 | 9.4 | 0.6 | 2 | 3.5% | 6.4 | 6.6 | 7.4 | 0.6 | 2 | 4.7% | 9 | 8.8 | 8.8 | 0.7 | 2 | 3.9% | C | yes | yes | yes | S |
| LN26 | 1.33 ↗ | 6.01 ↗ | 7.4 | 0.05 | 1.73 | 2.3% | 1.31 ↗ | 4.62 ↗ | 6.32 | 0.05 | 1.73 | 2.3% | 1.67 ↗ | 7.24 ↗ | 7.9 | 0.07 | 1.73 | 2.3% | NC | no | yes | yes | S |
| LN27 | 6.56 ↗ | 7.85 | 7.9 | 0.656 | 2 | 5.0% | 4.69 ↗ | 5.8 | 5.37 | 0.469 | 2 | 5.0% | 8.2 ↗ | 10.2 | 9.88 | 0.82 | 2 | 5.0% | C | no | yes | no | S |
| LO30 | 5.67 ↗ | 9.33 | 9.57 | 0.82 | 2 | 7.2% | 3.41 ↗ | 5.91 | 6.77 | 0.49 | 2 | 7.2% | 4.94 ↗ | 10.44 | 10.81 | 0.71 | 2 | 7.2% | NC | no | yes | yes | S |
| LO31 | 6.64 | 8.7 | 7.46 | 2.86 | 1.64 | 26% | 7.37 | 9.04 | 4.35 | 3.17 | 1.64 | 26% | 7.29 | 8.48 | 6.95 | 3.13 | 1.64 | 26% | C | yes | yes | yes | S |
| LO32 | 11.6 | 9.4 | 10.8 np | | | | 10.6 | 12.1 | 11.9 np | | | | 7.8 | 11.6 | 11.3 np | | | | C | ? | yes | ? | S |
| LO33 | 6.029 | 7.86 | 7.346 np | | | | 5.467 | 6.633 | 5.441 np | | | | 4.49 | 5.07 | 7.057 np | | | | NC | ? | yes | ? | S |
| LO34 | 7.6 | 9.5 | 9.6 | 1.1 | 2 | 7.2% | 8.4 | 8.7 | 10.2 | 1.3 | 2 | 7.7% | 8.9 | 9.8 | 10.8 | 1.3 | 2 | 7.3% | C | yes | yes | yes | S |
| LO35 | 5.88 ↗ | 7.49 | 7.12 | 0.6 | 2 | 5.1% | 3.82 ↗ | 4.92 | 5.62 | 0.4 | 2 | 5.2% | 3.92 ↗ | 5.01 | 5.35 | 0.4 | 2 | 5.1% | C | no | yes | no | Q |
| LO36 | 6.6 | 8 | 8.3 | 2.1 | 2 | 16% | 6.6 | 7.9 | 8.4 | 2.1 | 2 | 16% | 6.6 | 7.9 | 8.3 | 2.1 | 2 | 16% | C | yes | yes | yes | U |
| LO37 | 7 | 8 | 10 | 2.4 | 2 | 17% | 7 | 8 | 10 | 2.4 | 2 | 17% | 5 | 6 | 7 | 1.68 | 2 | 17% | NC | ? | yes | ? | Q |
| LO38 | 2 | 2.2 | 3.2 np | | | | 2 | 2.7 | 3.3 np | | | | 2 | 2 | 2.4 np | | | | NC | ? | yes | ? | Q |
| LO39 | 3.26 | 4.06 | 4.88 | 0.87 | 2 | 13.3% | 3.59 | 4.12 | 4.85 | 0.95 | 2 | 13.2% | 5.31 | 5.98 | 4.84 | 1.32 | 2 | 12.4% | NC | yes | yes | no | S |
| LO40 | 1.39 ↗ | 3.27 | 2.45 | 0.5 | 2 | 18% | 1.25 ↗ | 2.9 | 2.31 | 0.46 | 2 | 18% | 2.05 | 3.41 | 2.29 | 0.66 | 2 | 16% | NC | no | yes | yes | Q |
| LO43 | 9.24 | 10.01 | 10.44 | 2.1 | 2 | 11.4% | 6.41 | 8.38 | 8.21 | 1.55 | 2 | 12.1% | 5.74 ↗ | 10.54 | 10.12 | 1.41 | 2 | 12.3% | NC | no | yes | yes | S |
| LO44 | 4.4 | 4.7 ↗ | 7.6 | 0.9 | 2 | 10.2% | 2.4 ↗ | 3.6 | 4.7 | 0.5 | 2 | 10.4% | 4.3 | 5.2 | 5.2 | 0.9 | 2 | 10.5% | NC | no | yes | yes | Q |
| LO45 | 5.3 | 6.1 | 6.3 | 0.5 | 2 | 4.7% | 4.5 | 5 | 5.2 | 0.4 | 2 | 4.4% | 5.2 ↗ | 6.1 | 6.6 | 0.5 | 2 | 4.8% | NC | no | yes | yes | S |
| LO46 | 7.58 | 5.74 ↗ | 10.1 | 1.7 | 2 | 11.2% | 11.4 | 8.88 ↗ | 14 | 2.4 | 2 | 10.5% | 11 | 8.05 ↗ | 12.5 | 2.1 | 2 | 9.5% | C | no | yes | no | |
| LO47 | -- | | | | | | -- | | | | | | -- | | | | | | not tested | | | | |
| LO48 | 10 ↗ | 12 | 14 | 1.03 | 2 | 5.2% | 7 ↗ | 9 | 9 | 0.721 | 2 | 5.2% | 9 ↗ | 13 | 12 | 0.927 | 2 | 5.2% | NC | no | yes | yes | S |

# Annex 13: Results of the questionnaire

| LC Code | Laboratory status | Compliance statement regarding melamine | Justification | Compliance statement regarding formaldehyde | Justification | Accreditation for melamine and formaldehyde in food simulant B? |
|---|---|---|---|---|---|---|
| LN01 | NRL | non compliant | The third test of mug 3 gave a result of 2,90 ± 0,290 mg/kg melamine (FCM No. 239) and is above the specific migration limit of 2,5 mg/kg food or simulant given in the union list. Therefore, the whole test item 2 is non-compliant. | compliant | The apparent increase in formaldehyde migration from the first to the second and third migration result in all mugs is insufficiently verifiable with the given measurement uncertainty. Therefore, for official control, non-compliance cannot be proven based on these results. However, from the point of view of the BO, compliance cannot be proven either! | Yes, for both |
| LN02 | NRL | compliant | M1>M2; M2>M3 and M1>M3 ; M3<SML | non-compliant | M2>M1 and M3>M1; | Yes, for both |
| LN03 | NRL | compliant | (1) The mean levels of migration from test Item 2 (from all 3 mugs) in mg/kg are as follows: MIG1 = 1.10 ± 0.18; MIG2 = 1.24 ± 0.21; MIG3 = 1.02 ± 0.17. All of them are below the SML (2.5 mg/kg). (2) Migration is above the LOD in any of the three migration tests; MIG1 < MIG2 > MIG3; MIG2 is not significantly higher than MIG1 (zeta < 2); the stability of the material is sufficient. | compliant | (1) The mean levels of migration from test Item 2 (from all 3 mugs) in mg/kg are as follows: MIG1 = 2.81 ± 0.41; MIG2 = 3.39 ± 0.0.50; MIG3 = 2.26 ± 0.32. All of them are below the SML (15 mg/kg). (2) Migration is above the LOD in any of the three migration tests; MIG1 < MIG2 > MIG3; MIG2 is not significantly higher than MIG1 (zeta < 2); the stability of the material is sufficient. | Yes, for both |
| LN04 | NRL | compliant | Melamine is not measured. | non-compliant | Migration 3 is bigger than migration 2. | Yes, for FA |
| LN05 | NRL, OCL | non compliant | MIG3>SML and "Insufficient Stability" e.g. MIG2>MIG1 or/and MIG3>MIG2 or/and MIG3>MIG1 | non-compliant | Although MIG3<SML the Stability of the Material is Insufficient. MIG2>MIG1 and MIG1<MIG3<MIG2 | Yes, for FA |
| LN06 | NRL, OCL | non compliant | MIG3>SML and "Insufficient Stability" e.g. MIG2>MIG1 or/and MIG3>MIG2 or/and MIG3>MIG1 | non-compliant | Although MIG3<SML the Stability of the Material is Insufficient. MIG2>MIG1 and MIG1<MIG3<MIG2 | Yes, for FA |
| LN07 | NRL | non compliant | The stability of the material shall be considered insufficient. The second migration test exceeds the level observed in the first test - MIG1 < MIG2. | non-compliant | The stability of the material shall be considered insufficient. The second migration test exceeds the level observed in the first test - MIG1 < MIG2. The third migration test is approx. at the same level observed in the second test. | Yes, for MEL; |
| LN08 | NRL | non compliant | Compliant with SML but non compliant with requirement of stability of material. Migration test of cup 2 resulted in non compliance with stability. See used rule under point 22. | non-compliant | Compliant with SML but non compliant with requirement of stability of material. Migration tests of all three cups resulted in non compliance with stability. See used rule under point 22. | Yes, for both |
| LN09 | NRL, OCL | compliant | Result below SML. Stability OK. M3>M2 but not significantly when taking MU into account | compliant | Result below SML. Stability OK. M3>M2 but not significantly when taking MU into account | Yes, for FA |
| LN10 | NRL | compliant | the amount of migration and stability are ok | compliant | the amount of migration and stability are ok | Yes, for both |
| LN11 | NRL | non compliant | The third migration for the second mug is over the SML of 2.5 mg/kg (3.20 +/- 0.64 mg/kg) | non-compliant | The stability is insufficient for mugs 1 and 3 : the migration increases between the first and third tests (mug 1: 7.4 +/- 1.2 < 10.8 +/- 1.7 - mug 3: 4.6 +/- 0.7 < 9.0 +/- 1.4) | Yes, for FA |
| LN12 | NRL | compliant | the values are stable and under the specific migration limit | compliant | the values are stable and under the specific migration limit | Yes, for both |
| LN13 | NRL | non compliant | MIG3>SML for 2 out of 3 mugs -> non-compliant. Compliant acc. to stability rule. Applying lab's MU instead of Horwitz would result in compliant mugs. | compliant | MIG3<SML and compliant acc. to stability rule. | Yes, for both |
| LN14 | NRL | non compliant | The migration of melamine in the 3rd migration test was found to exceed the SML of 2.5 mg/kg for 2 out of the 3 subsamples tested. The relevant criterion for non-compliance is: (m3 - SML )/[(u(m3)] > 1.64. The Horwitz equation was used to calculate the measurement uncertainty. | compliant | For all the subsamples tested: The migration of formaldehyde in the 3rd migration test was lower than the SML of 15 mg/kg. The migration from the first to the third migration test did not increase significantly. Regarding the stability of the material, the criterion for compliance is: (mj - mi )/[(u(mj) + u(mi)] < 1.64. The Horwitz equation was used to calculate measurement uncertainties. | Yes, for both |
| LN15 | NRL | non compliant | The result is below our LOQ value, LOQ is1,86mg/kg. Formaldehyde leaching stability of the sample: M1>M2<M3 | non-compliant | M1>M2<M3 | Yes, for both |

| LC Code | Laboratory status | Compliance statement regarding melamine | Justification | Compliance statement regarding formaldehyde | Justification | Accreditation for melamine and formaldehyde in food simulant B? |
|---|---|---|---|---|---|---|
| **LN16** | **NRL** | **compliant** | **Sample complies with Commission Regulation (EU) 2020/1245 as their is no increase between extractions** | **compliant** | **Sample complies with Commission Regulation (EU) 2020/1245 as their is no increase between extractions** | **Yes, for both** |
| **LN17** | **NRL** | **non compliant** | **According to Reg (EU) 2020/1245 the samples are not compliant because MIG3 is higher than MIG2 and MIG1** | **non-compliant** | **According to Reg (EU) 2020/1245 the samples are not compliant because MIG3 is higher than MIG2 and MIG1** | **Yes, for both** |
| LN18 | NRL | non compliant | The stability of the material is sufficient because in any of the three migration tests, the migration does not increase from the first migration test to the third migration test (mx+1 – mx > 1.64 ( x+1+ x) → (m(x+1) - m(x)) / (u(x+1)+u(x)) < 1.64), and the specific migration limit is exceeded in the third test (m3 – 1.64 u3) > ML →((m3 – ML) / u3 > 1.64). $u$ $u$ | compliant | The stability of the material is sufficient because in any of the three migration tests, the migration does not increase from the first migration test to the third migration test (mx+1 – mx > 1.64 ( x+1+ x) → (m(x+1) - m(x)) / (u(x+1)+u(x)) < 1.64), and the specific migration limit is not exceeded in the third test (m3 – 1.64 u3) > ML → ((m3 – ML) / u3 < 1.64). $u$ $u$ | Yes, for FA |
| LN19 | NRL | compliant | below MRL and no increase | compliant | below MRL and no increase | Yes, for both |
| LN20 | NRL | compliant | All migrations for all 3 mugs are below 2.5 mg/kg | compliant | All migrations are below 15 mg/kg | Yes, for FA |
| LN21 | NRL | non compliant | Because the average of the 3 MUGs for MIG3 > SML + U | compliant | Because the average of the 3 MUGs for MIG3 < or = SML+U; also there are no statistically differences between averages of MIG1 and MIG2, MIG2 and MIG3, MIG1 and MIG3 | No |
| LN22 | | | | | | |
| LN23 | NRL | non compliant | Item 2 doesn´t meet requirements according Commission Regulation (EU) 2020/1245 - Annex V Chapter 2 - 2.1.6. | non-compliant | Item 2 doesn´t meet requirements according Commission Regulation (EU) 2020/1245 - Annex V Chapter 2 - 2.1.6. | Yes, for FA |
| LN24 | NRL | compliant | m3 < SML (Mug 1 and 3) and m3 < SML for Mug 2 taking into account our own laboratory expanded MU (2.6±0.9mg/kg) or using the statistical tool from draft JRC document ''Options for checking compliance ..2022'', point 3.3 a). Stability rule: m1 > m2 and m2 > m3 and m1 > m3, taking into account our own laboratory expanded MU or using the statistical tool from draft JRC document, point 3.3.b). | non-compliant | m3 < SML, but INSUFFICIENT STABILITY: Mug 1: m3 > m1 and m3 > m2, Mug 3: m3 >m1 and m2 > m1 using the statistical tool from draft JRC 2022 document, point 3.3.b). Using our own laboratory expanded MU: Mug 1: m3 > m1, Mug 3: m3 >m1 and m2 > m1; slightly different interpretation but still not compliant sample. | No |
| LN25 | NRL | non compliant | Result in MUG1 MIG3-U (2.6 mg/kg) > SML (2.5 mg/kg). | compliant | Results in MUG1, MUG2 and MUG3 MIG3-U <= SML (15 mg/kg) and stability rule is compliant. | Yes, for both |
| LN26 | NRL | non compliant | Taking into account cup 2-4 since cup 1 cracked during migration. Cup 2: M2>M1, M3>M1; Cup 3: M2>M1, M3>M1; Cup 4: M3>SML, M2>M1, M3>M1 | non-compliant | Taking into account cup 2-4 since cup 1 cracked during migration. M2>M1, M3>M2, M3>M1 | No |
| LN27 | NRL | compliant | all 3 mugs tested: non-significant increase because all values for migration 1, 2 and 3 within measurement uncertainty (±30 %). all 3 mugs tested: SML not exceeded considering the measurement uncertainty of ±30 % | compliant | mug 2 and mug 3: significant increase between migration 1 and 2, but non-significant increase for mug 1, increase remains within measurement uncertainty (±30 %) | Yes, for both |
| LO30 | OCL | non compliant | According to the stability rule none of them are compliant. Based on the MIG3, only MUG2 is compliant. MUG1 is about 2.56 mg/kg (considering the Uc) and MUG3 about 2.57 | non-compliant | Not compliant neither according to the stability rule but compliant if only the 15 mg/kg SML on the MIG3 is considered. | Yes, for both |
| LO31 | OCL | compliant | Taking into account the measurement uncertainty, the values are below the limit value or there is no increase in migration. | compliant | Taking into account the measurement uncertainty, the values are below the limit value or there is no increase in migration. | Yes, for both |
| LO32 | other | non compliant | migrationlimit for melamine according to regulation (EU) No 10/2011 exceeded. Partially increase in migration from first to third test but not consistent. Significance questionable, stability is classified as compliant. | compliant | migrationlimit for melamine according to regulation (EU) No 10/2011 not exceeded, partially increase in migration from first to third test but not consistent. Significance questionable, stability is classified as compliant. | Yes, for both |

| LC Code | Laboratory status | Compliance statement regarding melamine | Justification | Compliance statement regarding formaldehyde | Justification | Accreditation for melamine and formaldehyde in food simulant B? |
|---|---|---|---|---|---|---|
| **LO33** | **other** | **compliant** | **below the SML; stability of the material pass** | **non-compliant** | **Below the SML but - Stability test of the materilas fail.** | **Yes, for both** |
| **LO34** | **OCL** | **compliant** | **SML: Migration values are exceeding the SML of 2.5 mg/kg in the 3rd migration test of each mug. However, the exceedance is not statistically significant due to the measurement uncertainty of the method (25 %) and the criteria by Horwitz; Stability rule: Migration values are generally increasing for each mug. However, the increase is** | **compliant** | **SML: Migration values are below the SML of 15 mg/kg in the 3rd migration test of each mug; Stability rule: Migration values are generally increasing for each mug. However, the increase is not statistically significant due to the criteria by Horwitz.** | **Yes, for both** |
| **LO35** | **OCL** | **compliant** | **result MIG3 below SML 2,5 mg/kg** | **compliant** | **result MIG3 below SML 15 mg/kg** | **Yes, for both** |
| **LO36** | **OCL** | **compliant** | **compliant taking into account the expanded measurement uncertainty (including measurement uncertainty of the migration)** | **compliant** | **compliant taking into account the expanded measurement uncertainty** | **Yes, for both** |
| LO37 | other | non compliant | insufficient stability: compliance of the material is not be established even the specific migration limit is not exceeded in any of the three tests (value of migration for third test of MUG2 is compliant with the uncertainty). | non-compliant | insufficient stability: compliance of the material is not be established even the specific migration limit is not exceeded in any of the three tests | Yes, for both |
| LO38 | OCL | compliant | | non-compliant | There is a significant increase in formaldehyde concentration in the three attacks | Yes, for both |
| LO39 | OCL | non compliant | The requirement reported on Reg EU 10/2010 (point 2.1.6) is not satisfied. For MUG1, MUG2 and MUG3: MIG3 level exceeds MIG2 level. | non-compliant | The requirement reported on Reg EU 10/2010 (point 2.1.6) is not satisfied. For MUG1 e MUG2: MIG2 level exceeds MIG1 level and MIG3 level exceeds MIG2 level. For MUG3: MIG2 level exceeds MIG1 level. | Yes, for both |
| LO40 | other | non compliant | test not performed | non-compliant | Specific Migration (MS) is less than LMS sed the sample is not stable: MIG3<LMS, MIG3<MIG2 e MIG2>MIG1 | No |
| LO43 | OCL | non compliant | Even if the specific migration limit is not exceeded in any of the three tests, the result is not compliant because an increase from the first migration test to the third migration test is observed, indicating an insufficient stability of the material. | non-compliant | Even if the specific migration limit is not exceeded in any of the three tests, the result is not compliant because an increase from the first migration test to the third migration test is observed, indicating an insufficient stability of the material. | No |
| LO44 | other | non compliant | sample unstable | non-compliant | sample unstable | Yes, for both |
| LO45 | OCL | compliant | The value at the third incubation for all mugs are <LMR and all the mugs are stable | non-compliant | The value at the third incubation for all mugs are <LMR but the mug 2 is not stable for second incubation compare to the first incubation | Yes, for both |
| LO46 | OCL | non compliant | We not test the item | compliant | All determinations have a value less than 15 mg/kg | Yes, for FA |
| LO47 | OCL | non compliant | Concentration for 3 mugs over SML | non-compliant | No analyzed | Yes, for MEL; |
| LO48 | other | non compliant | MIG 3 is >SML (MUG 1 and MUG 3), and the samples are not stable as migration increases with contacts (the rule (mj-mi)/[MU(mj)+MU(mi)]>1.64 (uncertainty K=1) has been used as a criterion for stability assessment). | non-compliant | Although the results obtained are <SML, the sample is not stable as migration increases with contacts (the rule (mj-mi)/[MU(mj)+MU(mi)]>1.64 (uncertainty K=1) has been used as a criterion for stability assessment). | Yes, for FA |

| LC Code | Accreditation for the migration procedure? | Please specify | Did you pre-heat the simulant before filling the artide? | Temperature of the food simulant before filling of the test items | How did you control the temperature of the food simulant during the migration experiments | Did you preheat the mugs before migration? | Volume of the food simulant used for the migration experiments | Temperature of the food simulant during the migration experiment (in oC) | Did you measure the food simulant losses | If yes, please report the volume of the losses in ml | Analytical techniquecs for melamine |
|---|---|---|---|---|---|---|---|---|---|---|---|
| LN01 | no | | YES | 80 °C | calibrated datalogger | No | 410 ml | will be sent via mail | Yes | MUG 1: 8.3, 7.8, 7.0 ml \|\| MUG 2: 7.8, 7.5, 5.3 ml \|\| MUG 3: 5.5, 6.1, 7.0 ml | LC-MS/MS |
| LN02 | yes | | YES | 75 °C | calibrated datalogger | No | 420 ml | See excel file attached | Yes | No loss | HPLC-UV |
| LN03 | yes | EN 1186-3:2022 | YES | 72 °C | non-calibrated thermometer/datalogger | No | 400 ml | 70.1 +/- 1.0 oC | Yes | 5 ml | HPLC-UV |
| LN04 | no | | YES | 75 °C | calibrated thermometer | Yes | 400 ml | | No | | HPLC-UV |
| LN05 | yes | | YES | 85 °C | non-calibrated thermometer/datalogger | No | 430 ml | 70.1-71.9 oC | No | | HPLC-UV |
| LN06 | yes | | YES | 85 °C | non-calibrated thermometer/datalogger | No | 430 ml | 70.1-71.9 oC | No | | HPLC-UV |
| LN07 | yes | | YES | 73 °C | non-calibrated thermometer/datalogger | Yes | 470 ml | | Yes | 10 ml | LC-MS/MS |
| LN08 | yes | | YES | 72 °C | calibrated thermometer | No | 450 ml | Sent by email | Yes | Weight of simulant was measured after migration and used for further calculation of result | LC-MS/MS |
| LN09 | no | We have validated the procedure | YES | 70 °C | calibrated datalogger | No | 445 ml | | Yes | 1 | LC-MS/MS |
| LN10 | yes | | YES | 73,5 °C | calibrated datalogger | No | 470 ml | | No | | LC-MS/MS |
| LN11 | yes | | YES | 78 °C | calibrated datalogger | No | 470 ml | | No | | LC-MS/MS |
| LN12 | yes | yes by the COFRAC | YES | 70°C | calibrated datalogger | No | 400 ml | | No | | HPLC-UV |
| LN13 | no | - | YES | 72.3 | calibrated datalogger | Yes | 430 ml | MIG1: 69.7-71.2 °C / MIG2: 69.8-71.3 °C / MIG3: 69.1-70.8 °C | Yes | 7-12 mL | LC-MS/MS |
| LN14 | yes | | YES | 72°C | calibrated thermometer | No | 450 ml | Temperature range: 67.5°C - 70.0°C | No | | HPLC-DAD |
| LN15 | yes | Melamine: CEN/TS 13130-27:2005, Formaldehyde: MSZ EN ISO 4614-2000(B) | YES | 70°C | calibrated thermometer | No | 450 ml | send by mail | Yes | 3,5mL | HPLC-UV |

| LC Code | Accreditation for the migration procedure? | Please specify | Did you pre-heat the simulant before filling the artide? | Temperature of the food simulant before filling of the test items | How did you control the temperature of the food simulant during the migration experiments | Did you preheat the mugs before migration? | Volume of the food simulant used for the migration experiments | Temperature of the food simulant during the migration experiment (in oC) | Did you measure the food simulant losses | If yes, please report the volume of the losses in ml | Analytical techniquecs for melamine |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **LN16** | **yes** | | **YES** | **70 oC** | **Other (please specify)** | **No** | **475 ml** | **70oC** | **No** | | **LC-MS/MS** |
| **LN17** | **no** | | **YES** | **70 °C** | **calibrated datalogger** | **No** | **450 ml** | **70 °C** | **No** | | **HPLC-DAD** |
| LN18 | yes | | YES | 72 °C | calibrated datalogger | No | 453 ml | 70±2 °C | Yes | 2 mL | HPLC-UV |
| LN19 | yes | | YES | / | not controlled | Yes | 400 ml | 70°C | Yes | between 4 and 14 ml | LC-MS/MS |
| LN20 | yes | | YES | 71 | calibrated thermometer | Yes | 463 ml | 68.1 - 70.0 | No | | LC-MS/MS |
| LN21 | other, please specify below | Migration step is accredited together with the analite determination; we do not have accreditation for the migration step by it self. | YES | 73 C | calibrated thermometer | No | 400 ml | | Yes | average of 5 mL | HPLC-UV |
| LN22 | | | | | | | | | | | |
| LN23 | yes | accreditation of procedure is part of the method | YES | 73°C | calibrated thermometer | Yes | 460 ml | | Yes | | HPLC-DAD |
| LN24 | no | | YES | 72°C | calibrated datalogger | No | 400 ml | Excel file sent by e-mail. | Yes | 1 ml (1st migration test), 0 ml (2nd and 3rd migration test) | LC-MS/MS |
| LN25 | yes | European Standard CEN-EN 13130-1 and Regulation (EU) No 10/2011 | YES | 74 ºC | calibrated datalogger | No | 450 ml | 70 ºC (with an tolerance range of 2 ºC) | Yes | Looses in MIG1=5 mL, MIG2=5 mL and MIG3=10 mL | LC-MS/MS |
| LN26 | no | | YES | 75 °C | calibrated datalogger | No | 420 ml | | Yes | No losses | LC-MS/MS |
| LN27 | yes | | YES | 70 °C | calibrated datalogger | Yes | 400 ml | 68-71 °C | Yes | 2.0-2.8 mL | LC-MS/MS |
| LO30 | yes | | YES | in an oven at 75 °C | calibrated datalogger | No | 430 ml | Around 70.5°C (see the datalogger) | No | | HPLC-DAD |
| LO31 | yes | | YES | 75 °C | calibrated thermometer | Yes | 420 ml | 70 °C | No | | LC-MS/MS |
| LO32 | yes | we are flexibly accredited in the field of migration studies | YES | 71 °C | calibrated thermometer | Yes | mug 1/2/3:460 ml, mug 4: 450 ml | separate Excelsheet (Mail) | Yes | 10 | LC-MS/MS |

| LC Code | Accreditation for the migration procedure? | Please specify | Did you pre-heat the simulant before filling the artide? | Temperature of the food simulant before filling of the test items | How did you control the temperature of the food simulant during the migration experiments | Did you preheat the mugs before migration? | Volume of the food simulant used for the migration experiments | Temperature of the food simulant during the migration experiment (in oC) | Did you measure the food simulant losses | If yes, please report the volume of the losses in ml | Analytical techniquecs for melamine |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **LO33** | **yes** | | **YES** | **70 °C** | **calibrated datalogger** | **No** | **400 ml** | **68,0 - 69,9 °C** | **No** | | **HPLC-DAD** |
| **LO34** | **other, please specify below** | **The migration procedure is part of the accredited methods for melamine and formaldehyde.** | **YES** | **75 °C** | **calibrated datalogger** | **Yes** | **440 ml** | | **No** | | **LC-MS/MS** |
| **LO35** | **yes** | | **YES** | **70 °C** | **calibrated datalogger** | **No** | **for each experiment 450 ml** | **the temperature is controlled in a preheated beaker** | **No** | | **HPLC-DAD** |
| **LO36** | **yes** | | **YES** | **70 °C** | **calibrated thermometer** | **No** | **400 ml** | **please see excelsheet by separate mail** | **No** | | **HPLC-DAD** |
| LO37 | yes | | YES | 73 °C | calibrated thermometer | No | 455 ml | 70°C | No | | LC-MS/MS |
| LO38 | no | | YES | 70 °C | calibrated thermometer | No | 450 ml | 70°C | No | | LC-MS/MS |
| LO39 | yes | | YES | 70 °C | calibrated datalogger | No | 400 ml | Please see attachment | No | | HPLC-DAD |
| LO40 | no | | YES | 70 °C | calibrated thermometer | Yes | 460 ml | 69.4°C | No | | other, please specify below |
| LO43 | no | | YES | 70 °C | calibrated datalogger | No | 470 ml | | No | | HPLC-UV |
| LO44 | other, please specify below | accreditating since october 2023 | YES | 70 °C | calibrated datalogger | No | 410 ml | 70°C | No | | HPLC-DAD |
| LO45 | yes | | YES | 72 ºC | calibrated thermometer | Yes | 450 ml | 70 ºC | No | | LC-MS/MS |
| LO46 | yes | | YES | 73 ºC | calibrated datalogger | No | 466 ml | | No | | other, please specify below |
| LO47 | yes | | YES | 70 ºC | calibrated datalogger | No | 450 ml | | Yes | 25 ml ca. | LC-MS/MS |
| LO48 | yes | UNE-EN 13130.1 | YES | 73-74**ºC** | calibrated datalogger | Yes | 450 ml | MIG 1 (Media: 70.1ºC) // MIG 2 (Media: 69.8ºC) // MIG 3 (Media: 70.4ºC) | Yes | In case of loss of simulant, it is replaced up to the original starting volume. | LC-MS/MS |

| LC Code | LOQ for melamine in food simulant B? | Sample preparation for formaldehyde? | Analytical techniques for formaldehyde | LOQ for fo rmaldehyde in food simulant B' | Time (in hours/days) between the migration experiments (MIG1, MIG2 and MIG3) | surface to volume ratio applied to the test result | Any problem encountered? |
|---|---|---|---|---|---|---|---|
| 1 | 19 | 20 | 21 | 22 | 23 | 24 | 25 |
| LN01 | 0.1 mg/kg | acetyl acetone (AA) | Spectrophotometry | 2.5 mg/kg | 1 day (same starting time) | concentration per area calculated using c * 0.41 l / 2.78 dm² and then 6 dm² / 1 kg applied acc. article 17(2) | Measurement uncertainty could only be entered for the first migration each, but unfortunately only as decimal, not in percent. Therefore, it does not apply to second and third migrations. The field length for Q2 and Q4 was way too short to be "as detailed as possible", as was requested in the instructions. |
| LN02 | 0.001 mg/kg | acetyl acetone (AA) | HPLC-UV | 1 mg/kg | MIG 1 to MIG 2 : 10min ; MIG 2 to MIG 3 : 20 min | Yes, Volume : 0.42L ; surface 2.78 dm² | On of the 4 cups cracked during experiments. The results of the 3 other cups was taken into account |
| LN03 | 1.0 mg/kg | chromotropic acid | Spectrophotometry | 1.5 mg/kg | 0.25 h (15 min) after compleating of the previous migration | 2.58 : 0.4 = 6.45 | No |
| LN04 | - | chromotropic acid | Spectrophotometry | 0,92 mg/L | All experiment were made in 2 working days. | No ratio applied. | No. |
| LN05 | 0.25 mg/Kg | acetyl acetone (AA) | Spectrophotometry | 1.5 mg/Kg | 1hr between MIG1/MIG2 and 18h between MIG2/MIG3 | not applicable | No |
| LN06 | 0.25 mg/Kg | chromotropic acid | Spectrophotometry | 1.5 mg/Kg | 1hr between MIG1/MIG2 and 18h between MIG2/MIG3 | not applicable | No |
| LN07 | 0.00007 mg/kg | acetyl acetone (AA) | Spectrophotometry | 0.1 mg/kg | 1 hour | 3.07 dm2 : 0.47 l | |
| LN08 | 0.25 mg/kg | acetyl acetone (AA) | LC-MS/MS | 1,5 mg/kg | 0.1 hour | 6 | Not enough space for commenting on question 1 and 3: "Although no common agreement has been reached on harmonised procedure for checking stability we are reporting according to: Each migration result has uncertainty based on our analytical method validation and a coverage factor of 2. Non compliant if m1<m2 or m2<m3 or m1<m3 (result >= |
| LN09 | 0.25 mg/kg | acetyl acetone (AA) | LC-MS/MS | 1.5 mg/kg | 1 | 6 | no |
| LN10 | 0,5 mg/kg | acetyl acetone (AA) | Spectrophotometry | 3 mg/kg | mig1 -> 18 h -> mig2 -> 2 h -> mig3 | 6 dm2/l | no |
| LN11 | 0.1 mg/kg | acetyl acetone (AA) | Spectrophotometry | 1 mg/kg | 22 h (MIG1 - MIG2), 0.5 h (MIG2 - MIG3) | 2.96 dm² / 0.47 L | datalogger failure (migration 1 and 2) + evaporation in the mug used for the temperature monitoring (temperature underestimated, migration 3) |
| LN12 | 0,5 mg/L | 2,4-dinitrophenylhydrazine | HPLC-UV | 1 mg/L | 4 hours between mig 1 and mig 2 and 24h between mig 2 and mig 3 | real surface volume ratio | no |
| LN13 | 5 µg/L | acetyl acetone (AA) | Spectrophotometry | 0.5 mg/L | MIG1 -> MIG2: 38 min / MIG2 -> MIG3: 37 min | Filling volume and calculated surface of mugs (2.62 dm^2) were applied to the raw migration results. Results were converted to kg using a surface to volume ratio of 6 dm^2/kg. | Dispatch of ILC test items was delayed over the weekend. |
| LN14 | 0.25 mg/L | acetyl acetone (AA) | Spectrophotometry | 1,5 mg/L | 1 day (MIG1, MIG2 and MIG3 were performed in 3 consecutive days) | 6 dm2/kg | No |
| LN15 | 1,86mg/kg | acetyl acetone (AA) | Spectrophotometry | 0,33mg/kg | 2 hours | 1:2 | no |
| LN16 | 0.25mg/kg | other, please specify below | Spectrophotometry | 0.3 mg/kg | 20 mins | Yes | No |
| LN17 | 0.25 mg/Kg | chromotropic acid | Spectrophotometry | 2 mg/Kg | Tests are carried out in the same time | - | - |
| LN20 | 1.0 µg/L | acetyl acetone (AA) | HPLC-UV | 0.03 mg/L | 0.5 hours | 6:1 | No |

| LC Code | LOQ for melamine in food simulant B? | Sample preparation for formaldehyde? | Analytical techniques for formaldehyde | LOQ for fo rmaldehyde in food simulant B' | Time (in hours/days) between the migration experiments (MIG1, MIG2 and MIG3) | surface to volume ratio applied to the test result | Any problem encountered? |
|---|---|---|---|---|---|---|---|
| 1 | 19 | 20 | 21 | 22 | 23 | 24 | 25 |
| LN18 | 1,6 mg/kg | acetyl acetone (AA) | Spectrophotometry | 1.5 mg/kg | 24 hours | 6 dm2/kg | None |
| LN19 | 0.23 mg/kg | other, please specify below | Spectrophotometry | 2 mg/kg | 1 day at each time | 0.001 | none |
| LN20 | 1.0 µg/L | acetyl acetone (AA) | HPLC-UV | 0.03 mg/L | 0.5 hours | 6:1 | No |
| LN21 | 0.23 mg/kg | acetyl acetone (AA) | Spectrophotometry | 1.75 mg/kg | 0.25 hours | Real ratio A/V | Time to reach the test temperature due to the type of article and the volume of the test. The mugs seemed to be made with isolated material turning the transference of the temperature thru the mugs wall very difficult. The control of the 2nd test temperature was facilitated because the mugs were already warm from the 1st test. |
| LN22 | | | | | | | |
| LN23 | 0.6 mg/kg | acetyl acetone (AA) | Spectrophotometry | 3.00 mg/kg | MIG1 - MIG2 (15 min); MIG2-MIG3 (19 min), total time between MIG1-MIG3 (2 hours and 34 min) | 2.79 square dm / 460 ml | no |
| LN24 | 0.25 mg/kg | chromotropic acid | Spectrophotometry | 3 mg/kg | 3 minutes | Surface to volume ratio 6 dm2 / 1000 ml was applied to the test results (because volume at migration test was 400ml, surface of the sample was 2.43dm2). | no |
| LN25 | 0.5 mg/kg | chromotropic acid | Spectrophotometry | 1.4 mg/kg | 24 hours between MIG1 and MIG3 (Last migration process). | S/V (dm2/kg)=6 | Accidental loss of food simulant during the filling of MUG3 in MIG3. |
| LN26 | 0,001 mg/kg | acetyl acetone (AA) | HPLC-UV | 1 mg/kg | Between MIG 1 and 2: 13 min. Between MIG 2 and 3: 18 h. | Yes. Surface 2,78 dm2 Volume 0,42 l | One of the cups cracked during migrations. |
| LN27 | 0.1 mg/L | acetyl acetone (AA) | Spectrophotometry | 0.1 mg/L | <0.25 h | 2.336 dm2/400 mL | We suspect hexamethylenetetramine (HMTA) and 1,4-butanediol formal – if present – to be decomposed and forming formaldehyde during migration testing (acidic, 70 °C). An issue to be investigated in the future. |
| LO30 | 0,5 mg/kg | 2,4-dinitrophenylhydrazine | HPLC/DAD | 3 mg/kg | 17h between MIG1&2 (consecutively performed) and MIG3. See datalogger | No because could be used by children < 3 yo. The Surface in contact is 2.94 dm² and the filling volume 430 mL | No |
| LO31 | 0,25 mg/kg | acetyl acetone (AA) | Spectrophotometry | 0,13 mg/kg | MIG1 Day 1, MIG 2 and MIG 3 day 2, intervall 1 h | 6 mg/dm² (results are normalised) | MUG 4 was broken, MUG 5 was also analyzed, results are much higher, see attached file via mail. |
| LO32 | 0,03 mg/l | acetyl acetone (AA) | Spectrophotometry | 0,9 µg/25ml | all experiments in one day, ca. 45 min between tests | mug 1-3: 6 dm²/l, mug 4: 6,2 dm²/l | one of the cups had significantly lower migration values for melamine and formaldehyde and was therefore not included in the data submission. |
| LO33 | 1,22 mg/kg | 2,4-dinitrophenylhydrazine | HPLC/DAD | 0,70 mg/kg | MIG1 to MIG2: 5 min; MIG2 to MIG3: 6 min (time between the migration tests) | volume: 400 mL; contact surface: 2,92 dm² / and surface to volume ratio 6 dm² per kg of food | no |
| LO34 | 0.03 mg/kg | 2,4-dinitrophenylhydrazine | HPLC/DAD | 1 mg/kg | MIG1, MIG2 and MIG3 were performed in direct succession with a few minutes in between. | 6.0 dm²/L | During migration a little piece of plastic broke from the inner bottom of one of the 4 mugs. Consequently, this mug was not considered for the reported migration values. |
| LO35 | 0,14 mg/kg | acetyl acetone (AA) | Spectrophotometry | 0,6 mg/kg | only few minutes, all migrations within one day | 6.47 | no |
| LO36 | 0.25 mg/kg | acetyl acetone (AA) | Spectrophotometry | 0.9 mg/kg | time between the migration experiments under 5 minutes | 6 dm2/kg | no |

| LC Code | LOQ for melamine in food simulant B? | Sample preparation for formaldehyde? | Analytical techniques for formaldehyde | LOQ for fo rmaldehyde in food simulant B' | Time (in hours/days) between the migration experiments (MIG1, MIG2 and MIG3) | surface to volume ratio applied to the test result | Any problem encountered? |
|---|---|---|---|---|---|---|---|
| 1 | 19 | 20 | 21 | 22 | 23 | 24 | 25 |
| LO37 | 0.25 mg/kg | chromotropic acid | Spectrophotometry | 1.5 mg/kg | the three mugs were analyzed simultaneously | 0.6 | It's not clear to which values apply the uncertainty of the method in the 3 tests. It was decided to apply it to the first test for each mug |
| LO38 | 0.3 mg/Kg | chromotropic acid | Spectrophotometry | 2.0 mg/Kg | 8 days for Formaldehyde; 16 days for Melamine | 0,6 | |
| LO39 | 0.1 mg/Kg | 2,4-dinitrophenylhydrazine | HPLC/DAD | 2 mg/Kg | 1 day | 3.12 dm2 | no |
| LO40 | 0 | chromotropic acid | Spectrophotometry | 1.50 mg/kg | MIG1-MIG2 30 minutes; MIG2-MIG3 two days | YES | NO |
| LO43 | 0.5 mg/Kg | chromotropic acid | Spectrophotometry | 3 mg/Kg | 5 minutes | 6 dm2/L | No problems encountered |
| LO44 | 0,1 mg/kg | 2,4-dinitrophenylhydrazine | HPLC/DAD | 1,5 mg/kg | 10 minutes | not applied | no |
| LO45 | 0.2 mg/Kg | chromotropic acid | Spectrophotometry | 3 mg/Kg | 0 hours, in successive incubations, one after the other on the same day. | 6 (dm2/Kg) /6.3 (dm2/Kg) = 0.95 | NO |
| LO46 | 0,0 | chromotropic acid | Spectrophotometry | 3 mg/kg | 3 days | 6 dm2/kg | No |
| LO47 | 0.05 mg/kg | 2,4-dinitrophenylhydrazine | HPLC-UV | . | 2 days | 0.66 | Some difficulties for maintaining mug temperature |
| LO48 | LOQ = 0.1 mg/Kg | chromotropic acid | Spectrophotometry | LOQ = 3 mg/Kg | MIG 1 (25/07/23, 13:30-15:30) // MIG 2 (26/07/23, 9:00-11:00) // MIG 3 (26/07/23, 12:40-14:40) | 6.0 dm2/Kg (according Reg 10/2011, article 17, articles less than 500 millilitres | No |

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