EURL-FCM PT 23/01 – Migration of melamine and formaldehyde from bamboo/melamine ware

Unexpected observations

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EFSA FCM meeting hybrid, 22-24 Oct. 2024

European Commission

Joint Research Centre

PT 23/01 - Scope

- ✓ Confirm the analytical capability of the participant to quantify melamine and formaldehyde in acetic acid 3 % (food simulant B)
- ✓ Support the Commission Regulation (EU) 2020/1245, Section 2.1.6 of Annex V, related to the compliance rules for **“Repeated use materials and articles”** to ensure its harmonised implementation

PT 23/01 - Design

First exercise: analysis of melamine and formaldehyde in acetic acid 3 % w/v (food simulant B)

- Sample: spiked food simulant B
- Measurands: melamine, formaldehyde
- Reported values: average mass fraction and expanded uncertainty with coverage factor

Second exercise: migration from repeated use kitchenware (MUGS)

- Sample: 4 mugs (1 mug only for temperature optimisation and contact surface calculation)
- Food Simulant: B
- Test conditions: 70°C for 2 h (three consecutive migration experiments)
- Measurands: melamine and formaldehyde
- Reported values: individual mass fractions and expanded uncertainty (1st migration) with coverage factor

Measurement of the temperature of the food simulant during the migration using a certified calibrated thermometer

Test items

Test item1: one 22 mL vial containing a solution of melamine and formaldehyde in acetic acid 3 % w/v (food simulant B)

Test item 2: 4 melamine-bamboo mugs for migration test in food simulant B

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Time Frame

- Invitation for registration May 17, 2023
- Registration deadline: June 12, 2023
- Dispatch: June 21, 2023
- Reporting deadline: August 30, 2023 (September 6 – last reported data
- Draft report: October 2, 2023

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Participants

- 26 NRLs from 24 countries reported results
    - 1 NRL - 2 set of results for formaldehyde)
- 17 OCLs from Belgium, Germany, Italy and Spain;
- 2 participants did not reported results at all
- 4 participants did not report results for melamine
- 1 participant did not report results for formaldehyde

NRL, national reference laboratory
OCL, official control laboratory

First exercise : determination of the mass fraction of melamine and formaldehyde in food simulant B

Test Item 1: Food simulant B solution (3 % w/v acetic acid) spiked with melamine and formaldehyde certified standards

- Assessment of adequate homogeneity and stability (HPLC DAD at JRC)
- **Assigned value**

  x_{pt} ± U(x_{pt}) (k=2) by **formulation** (confirmed by HPLC DAD at JRC)

$$u(x_{pt}) = \sqrt{u_{char}^2 + u_{hom}^2 + u_{st}^2}$$

- **Standard deviation** for proficiency assessment – previous PT and expert judgment

  σ_{pt}: 15 % **Evaluation criteria:** z and ζ scores

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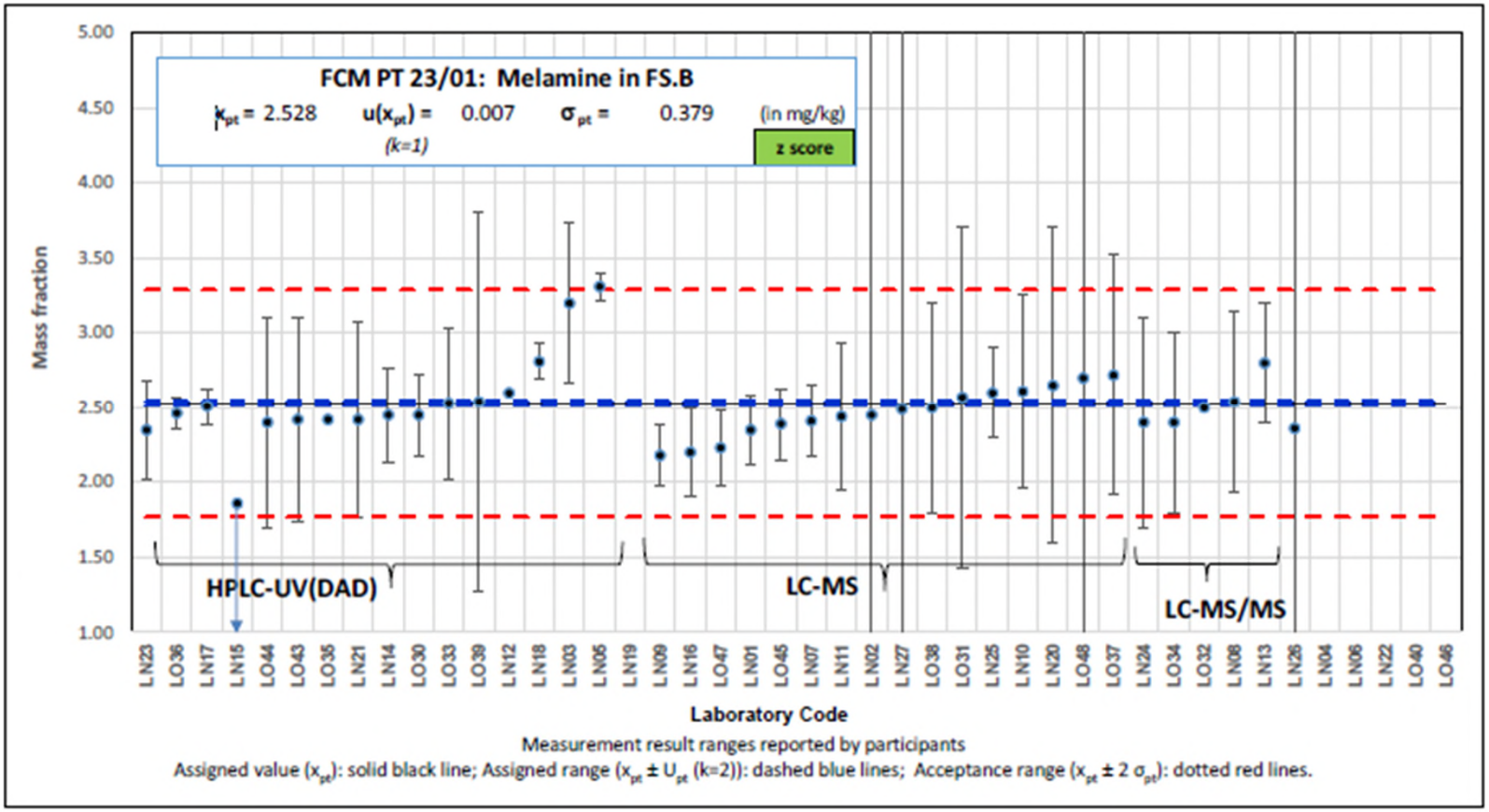
Results for melamine in food simulant B



Robust mean	**2.47 mg/kg**
Robust RSD	**5 %**

x_{pt} = 2.53 mg/kg
σ_{pt} = 15 %

RSD, relative standard deviation

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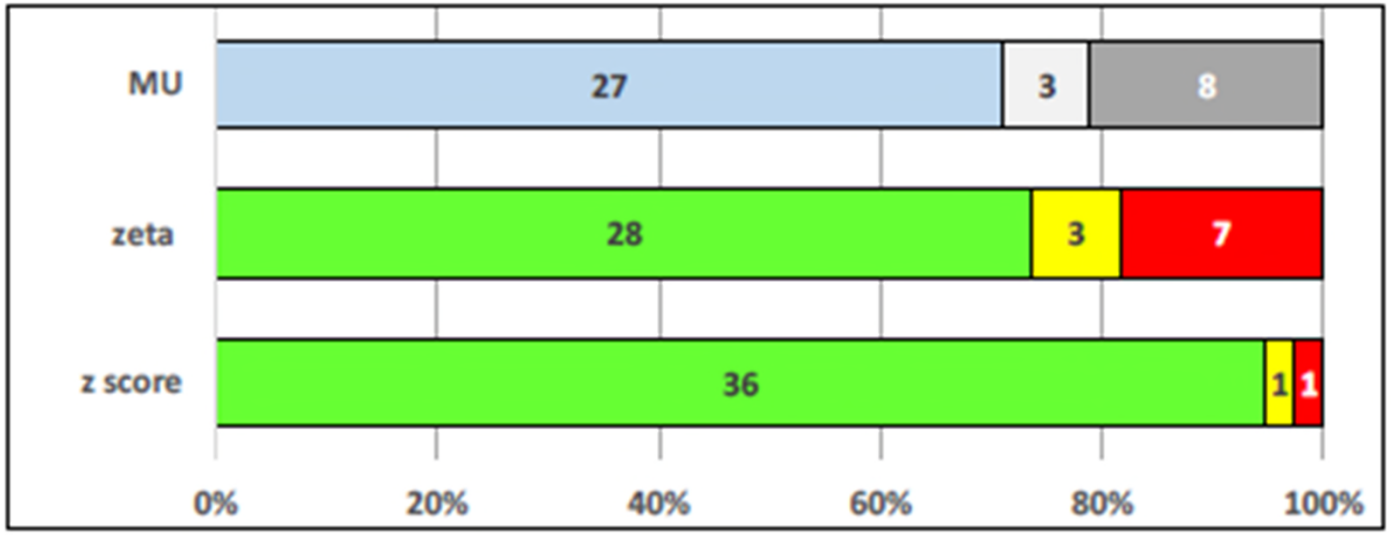
Results for melamine in food simulant B (2)



Satisfactory performance of **95 %** and above according to the **z score** and **70 %** and above according to **zeta score**

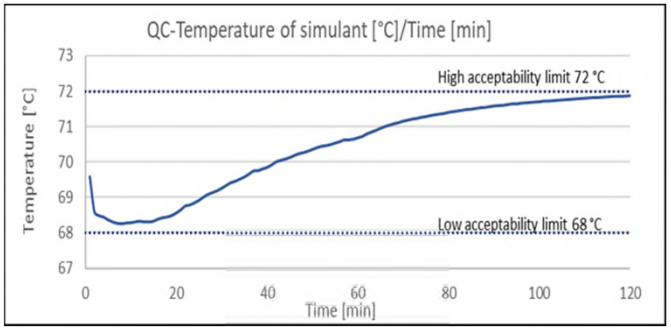
72 % of the participants reported a **realistic** measurement uncertainty evaluation (case "a": $u(x_{pt}) \leq u(x_i) \leq \sigma_{pt}$.

Second exercise : migration test

- Test Item 2: melamine-bamboo MUG
- Migration test: three consecutive migration (MIG, repeated use articles)
- Test condition : 70 ± 2 °C / 120 ± 5 min (control of food simulant temperature required)
- Reporting: 9 value per measurand

MUG1MIG1	MUG2MIG1	MUG3MIG1
MUG1MIG2	MUG2MIG2	MUG3MIG2
MUG1MIG3	MUG2MIG3	MUG3MIG3

- Evaluation: compliance statements justifications



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homogeneity assessment of MUGs for melamine

	Melamine		
	MIG1	MIG2	MIG3
Mug 1	3.36	3.53	3.79
Mug 2	3.41	3.34	3.19
Mug 3	3.34	3.67	3.97
Mug 4	3.08	3.49	3.65
Mug 5	3.99	3.81	3.76
Mug 6	3.85	3.05	3.62
Mug 7	3.35	3.67	3.82
Mug 8	3.17	3.06	3.20
Mug 9	3.70	3.18	3.40
Mug 10	3.20	3.39	3.66
Mean	3.45	3.42	3.61
RSD	8.8%	7.7%	7.3%

Set up of the exercise:

10 MUGs – 3 consecutive migration each;
Analyses for melamine and formaldehyde;
Evaluation of the RSD for sufficient homogeneity.

The set of 250 the mugs proved to be sufficiently homogeneous for each of the three migrations for the aim of the exercise

Mugs assumed to be stable with respect to the analytes over the time covered by the PT

Compliance criteria

Crit.1: **IF** $(m3 - SML)/[(u(m3)] > 1.64$ **THEN** $m3 > SML$

Crit.2: **IF** $(mj - mi)/[(u(mj) + u(mi)] > 1.64$ **THEN** $mj > mi$

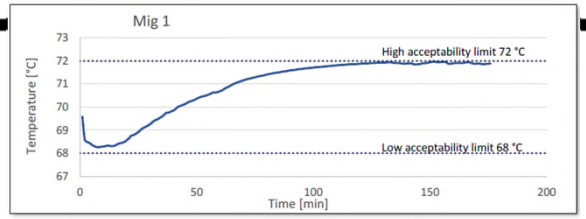
Compliance of test item 2

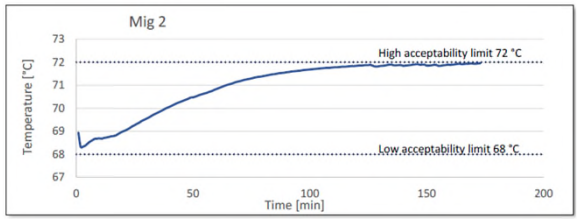
- Formaldehyde:
  - m3 ≤ SML (15 mg/kg)
  - m1 ≥ m2 ≥ m3 ➔ stable

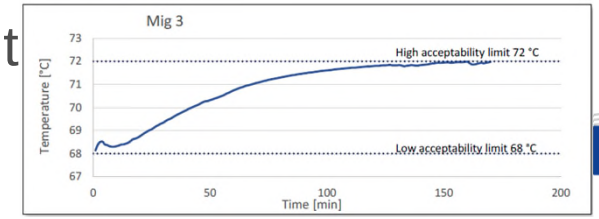
  } Compliant

- Melamine:
  - m3 > SML (2.5 mg/kg)
  - m1 ≥ m2 ≥ m3 ➔ stable

  } Non-compliant



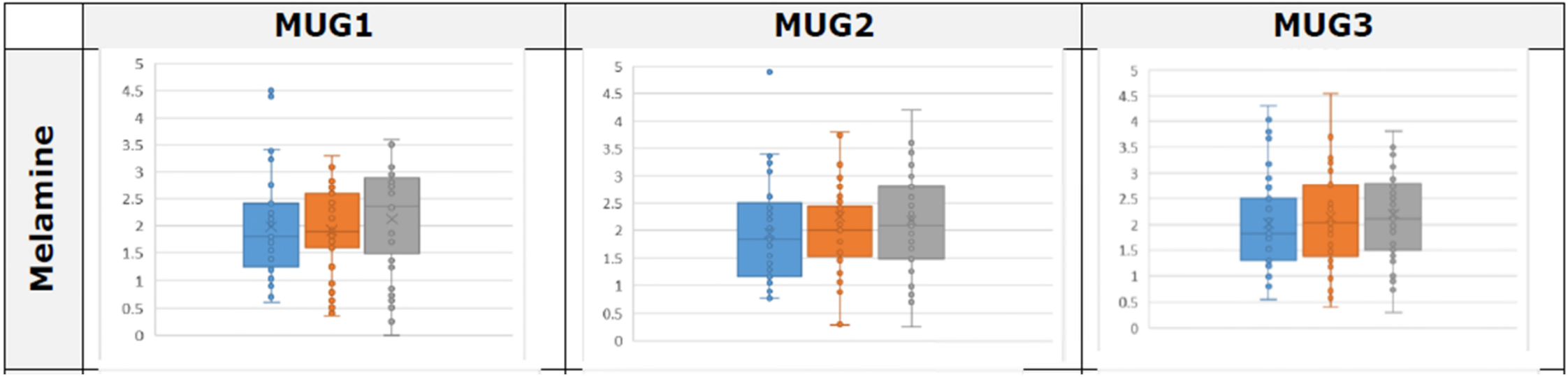




SML, specific migration limit

Outcome of the migration exercise

Melamine from bamboo/melamine ware



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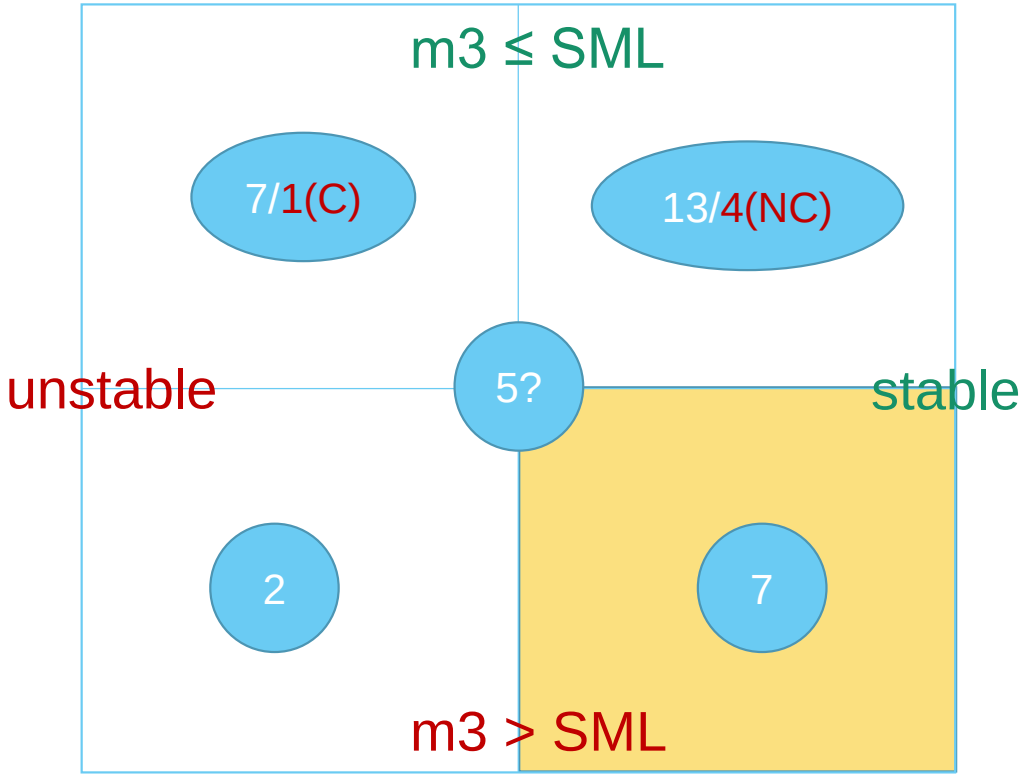
EURL assessment vs lab own assessment



Melamine

Including formaldehyde compliance:

6 laboratories in line with EURL

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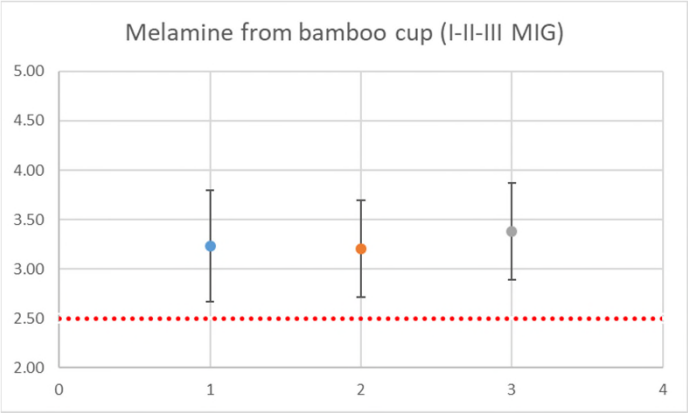
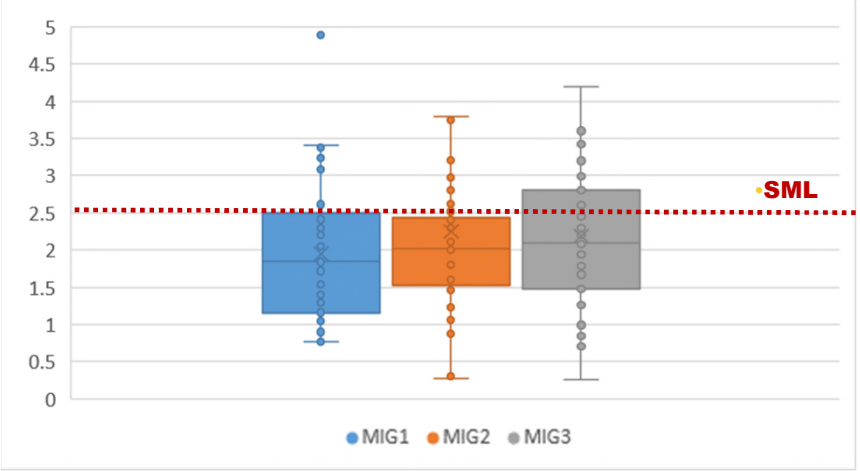
Melamine from bamboo/melamine ware





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	mig1	mig2	mig3
Ref. value, mg/kg	3.2	3.2	3.4
RSD, % (n=10)	8.8	7.7	7.3

PT participants

	mig1	mig2	mig3
Robust mean, mg/kg	1.9	2.0	2.2
Robust RSD, %	46.5	42.7	43.0

In food simulant
Robust RSD 5%

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Variations in the results from the migration tests

- **Uncontrolled/non-harmonised migration experiment ? - YES! Due to**

poor temperature (T) control over the food simulant during the migration test – profiles outside the 70±2°C, influenced by

- Temperature distribution in the oven
- Initial T of the oven
- If relevant gradient maintaining the T of the food simulant as constant as possible (closest to 70°C)
- Initial T of the food simulant
- Initial T of the article before the migration test - article preheating
- Measurements of the
  - contact surface @test T
  - volume of the food simulant @ room temperature)
  - control for losses and compensations

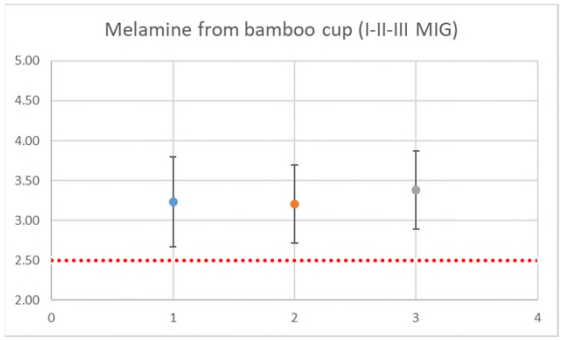
All pre-heat the food simulant:
15 – at 70°C;
24 – at 71-75°C;
3 – at 80°C;

13 pre-heat the article (7 NRLs)
30 do not pre-heat the article

2/13 - in different days;
11/13 - consecutively in 1 day

11/30 - the 3 migrations one after another 5-15 min apart;
7/30 - the 3 migration in 3 different consecutive days;
11/30 - the 3 migrations in 2 days

Melamine – T profile impact on the results



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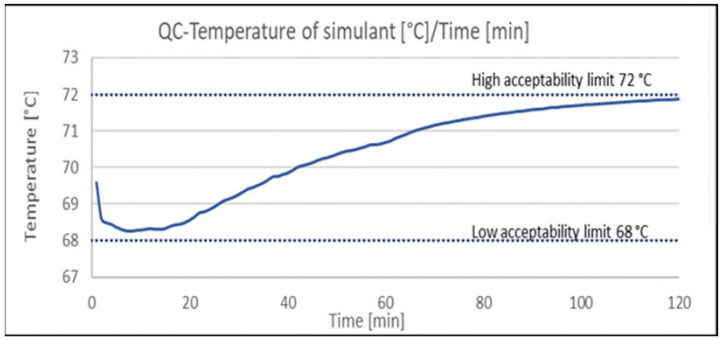

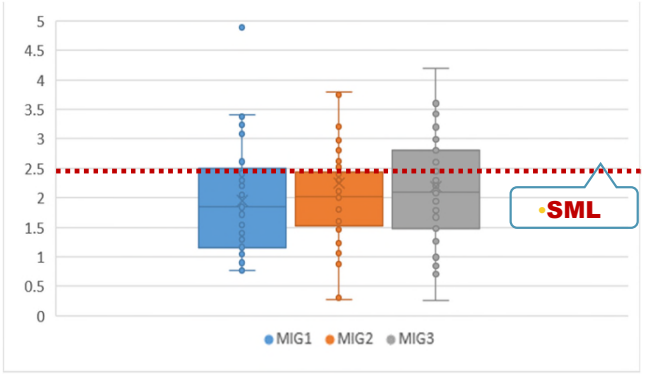

PT participants

Very different temperature profiles

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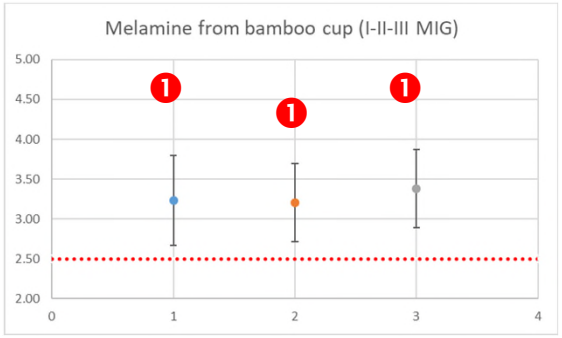
Melamine – T profile impact on the results



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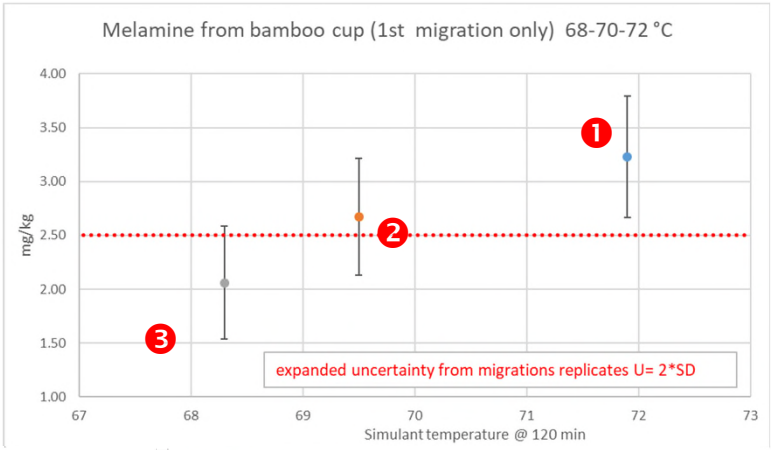

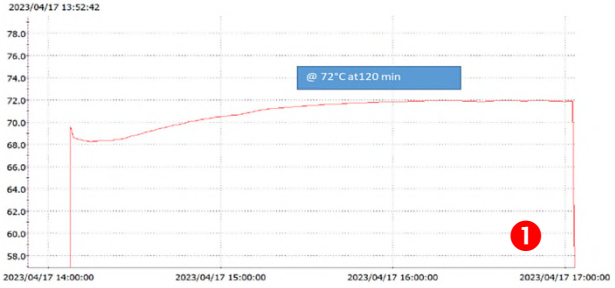

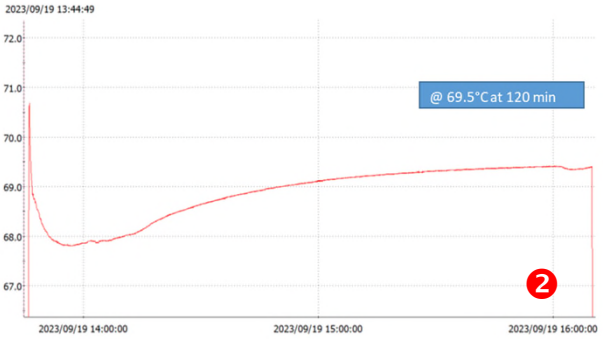

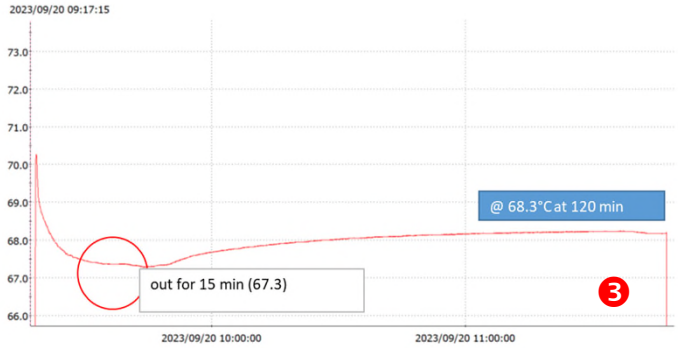

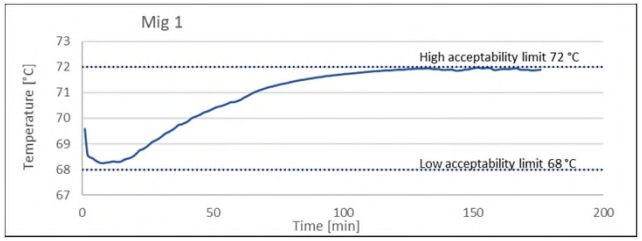

Conclusion …

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Finding

Strictly following all the requirements in the legislation and the kitchenware guidance, an homogeneous batch of articles could be correctly evaluated as compliant and as non-compliant with respect to the SML, only due to the allowed tolerance in the T profiles during the migration experiment

What has to be strictly harmonised?

- Mandatory data logger with readings at least every minute !
- Position of the data logger probe – in the food simulant filled in the test item (NOT OTHER item);
- Homogeneity of the T profiles at different point in the oven (ventilation oven needed)
- Initial T of the food simulant – pre-heating T needs optimization depending on the test items
- Initial T of the article before filling – should be evaluated on effect on migration, e.g. multilayers
- Ensuring the same T profile during the three successive migrations for repeated use articles – time between the consecutive migrations enough for the article to cool down;
- Measurements of the
  - contact surface @test T
  - volume of the food simulant @ room temperature
  - control for losses and compensations

Thank you

https://joint-research-centre.ec.europa.eu/eurl-food-contact-materials_en

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