# European Commission JRC activities

EFSA FCM Scientific Network meeting, 22/11/2022

# Content

- Kitchenware guideline on test conditions
- Options for checking compliance of plastic FCM articles for repeated use
- Mineral oil in food and FCM

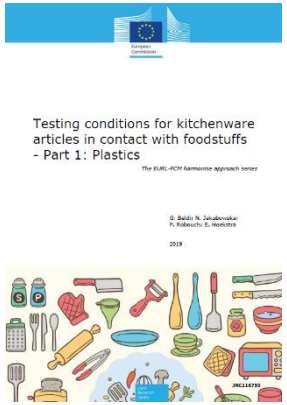
# Kitchenware guidelines

2019 – Guideline on "Testing conditions for kitchenware articles in contact with foodstuffs - Part 1: Plastics;

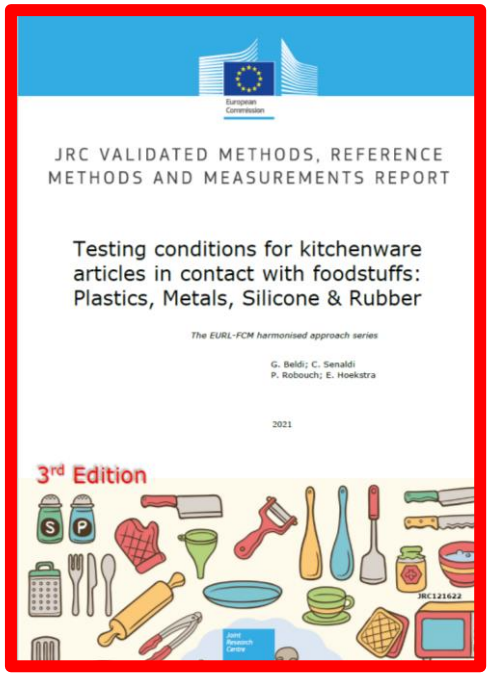
2020 – Guideline on "Testing conditions for kitchenware articles in contact with foodstuffs - Part 2: Plastics and Metals;

2021 – Guideline on "Testing conditions for kitchenware articles in contact with foodstuffs - Part 3: Plastics , Metals, Silicone & Rubber

2022 – paper & board next







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# Test conditions in Regulation (EU) No 10/2011

**Selection of test temperature**

| Worst foreseeable contact temperature | Contact temperature to be selected for testing |
|---|---|
| T ≤ 5 °C | 5 °C |
| 5 °C < T ≤ 20 °C | 20 °C |
| 20 °C < T ≤ 40 °C | 40 °C |
| 40 °C < T ≤ 70 °C | 70 °C |
| 70 °C < T ≤ 100 °C | 100 °C or reflux temperature |
| 100 °C < T ≤ 121 °C | 121 °C (*) |
| 121 °C < T ≤ 130 °C | 130 °C (*) |
| 130 °C < T ≤ 150 °C | 150 °C (*) |
| 150 °C < T < 175 °C | 175 °C (*) |
| 175 °C < T ≤ 200 °C | 200 °C (*) |
| T > 200 °C | 225 °C (*) |

(*) This temperature shall be used only for food simulants D2 and E. For applications heated under pressure, migration testing under pressure at the relevant temperature may be performed. For food simulants A, B, C or D1 the test may be replaced by a test at 100 °C or at reflux temperature for duration of four times the time selected according to the conditions in Table 1.

**Selection of test time**

| Contact time in worst foreseeable use | ►M7 Time to be selected for testing ◄ |
|---|---|
| t ≤ 5 min | 5 min |
| 5 min < t ≤ 0,5 hour | 0,5 hour |
| 0,5 hours < t ≤ 1 hour | 1 hour |
| 1 hour < t ≤ 2 hours | 2 hours |
| 2 hours < t ≤ 6 hours | 6 hours |
| 6 hours < t ≤ 24 hours | 24 hours |
| 1 day < t ≤ 3 days | 3 days |
| 3 days < t ≤ 30 days | 10 days |
| Above 30 days | See specific conditions |

+ Specific conditions for contact times above 30 days at room temperature and below

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# Test conditions

- **Plastics**: based on **expert judgement** on the “worst case” foreseeable conditions of use
  - not as the use intended by the producer
  - following principles of sections 2.1.3 and 2.1.4 (SM) and 3.1 (OM) of Annex V of Regulation (EU) No 10/2011
- Consumers use specific utensils **independently of the material**
  - Other materials: test conditions generally based on those for plastic FCM
- **Metals and alloys, silicone and rubber**: no material specific EU legislation
  - Except Art. 3 of Regulation (EC) No 1935/2004
  - **National legislation shall apply**
  - in absence, the test conditions in guidelines apply




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# Food simulants

- Plastics: Regulation (EU) No 10/2011
- Metals and alloys, silicone and rubber: follow national legislation and in absence
  - Use other guidance, e.g. the practical guideline of the Council of Europe or recommendations and in absence of those
  - Food simulants for plastics
  - Metals: if tested with a food simulant for acidic foods (pH ≤ 4.5), additional testing in artificial tap water is not required.
- If for any reason the indicated food simulants are not appropriate, testing with food should be considered
- NOTE: results in food prevail over the results obtained in food simulant.

# How to select the test conditions (1)

1. Select the main class and subclass of the kitchenware article

Example

| | | |
|---|---|---|
| Food Serving Utensils for Cold/Ambient or Hot use | FSU/CAH1 | Cup, Glass, Drinkware |
| | FSU/CAH2 | Open flask, Carafe, Can, Jug |
| | FSU/CAH3 | Bottle |
| | FSU/CAH4 | Baby bottle, Teats |
| | FSU/CAH5 | Tableware, Plate, Dishware, Serving stand |
| | FSU/CAH6 | Food tray, Serving board, French fries box, Finger food bag, Snack box, Popcorn box |
| | FSU/CAH7 | Thermos flask, Isothermic drinking beaker |

# How to select the test conditions (2)

Embossed; 260°C

2. If a **permanent label on the article** (e.g. embossed or engraved) defining limiting conditions of use or providing operating instructions, then adapt the test conditions accordingly **BUT** …
3. If instructions are **ONLY** on the packaging of the article (can be discharged) or not present at all, then select the test condition for that type of article from this guide.
4. When this guideline assigns several possible test conditions for the same type of article, then select the **most severe** test conditions appropriate for the specific article.


Not fixed; 450°F (232°C)

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# Selection of most severe test conditions

Table 2 for plastic:

| Subclass | Use | | | | Sample prep | | | Test type | | | | Food/Food simulant | | | | | | | SM Conditions (only food simulants) | | | S/V | | | | OM conditions | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | cold (< 20 °C) | Room Temperature | hot (> 40 °C) | storage (in months) | cut test specimen | intact article | part of it | actual use | article fill | migration cell | (total) immersion | food | A | B | C | D1 | D2 | E | time | Temp (°C) | label/instructions | Real | Real (infant/young) | 6 (V < 0.5L or V > 10L) | 6 impractical s/v | | |
| FSU/CAH1 | x | x | | | | y | x | | y ; x | | x | x | x | x | | | x | | 24 h | 40 | | x | x | x | | 2 | |
| | | x | x | | | y | x | | y ; x | | x | x | x | x | | | x | | 2 h | 70 | | x | x | x | | 3 | followed by 24 h at 40 °C, if used for storage [OM2] |
| FSU/CAH2 | x | x | | | | x | | | x | | | x | | x | | x | | | 24 h | 40 | | x | | x | | 2 | |
| | | x | x | | | x | | | x | | | x | | x | | x | | | 2 h | 70 | | x | | x | | 3 | followed by 24 h at 40 °C, if used for storage [OM2] |

Cup, glass, drinkware

Open flask, carafe, can, jug,

Most severe

2 h 70 x x x 3 followed by 24 h at 40 °C, if used for storage [OM2]

# How to select the test conditions (3)

5. If the prescribed test conditions may physically damage the test specimen, the migration tests shall be carried out under the “worst foreseeable conditions of use” to avoid such changes.
6. If a food simulant causes changes to the test specimen, e.g. swelling, that does not occur with food, this food simulant is not suitable.
   - Perform the migration test using food or another equivalent food simulant not causing such changes
7. For articles used only under specific time, temperature conditions and/or for specific foods the selected test conditions and food simulants should comply with those specific conditions of use.

# Paper & board

Coated/treated paper v.s. uncoated/untreated paper

Regulation (EU) No 10/2011

CEN “extraction” standards?

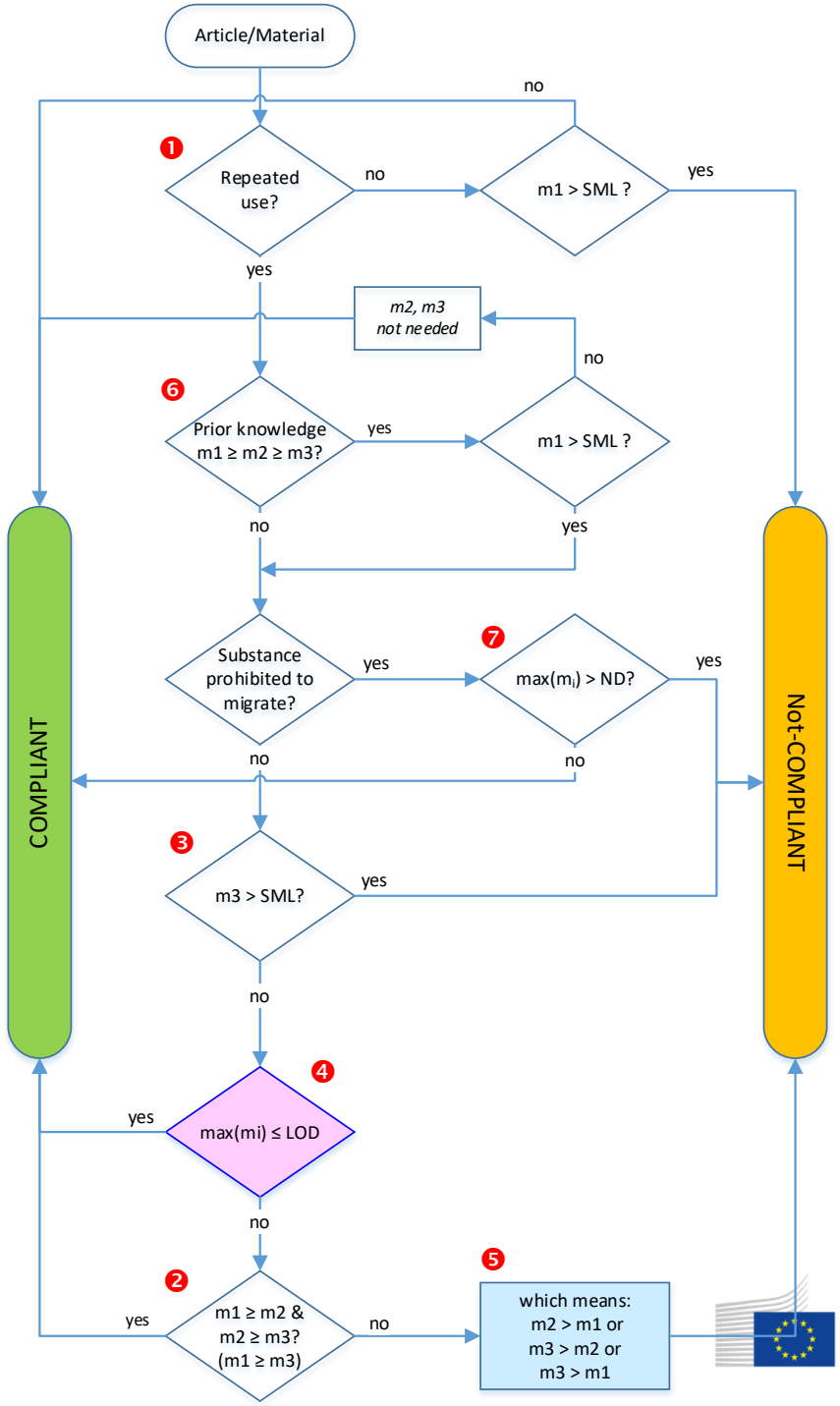
| Food in contact with | Food Simulant/extraction media | Test Conditions | Ref |
|---|---|---|---|
| **Dry, non fatty food** | E | Real conditions of use | EN 14338 |
| **Aqueous food in contact with coffee filter, tea bags and cooking bags** | Deionized water | 2h @ 80°C “hot water extraction” | EN 647 |
| **Moist/Aqueous food and beverages (other app.)** | Deionized water | 24h @ 23°C “cold water extraction” | EN 645 |
| **All kind of food (baking application)** | Deionized water | 2h @ 80°C “hot water extraction” | EN 647 |
| | E | 2h @ 175°C [oven use]<br>0.5h @ 150°C [microwave use] | EN 14338 |
| **Fatty food** | OS (95 % EtOH or Isooctane) | 2h @ 20°C [short contact]<br>24h @ 20°C [long contact]<br>2h @ 60°C [cooking and baking application for any time contact ] | EN 15519 |
| | Deionized water | 24h @ 23°C “cold water extraction” | EN 645 |

# Options for checking compliance of plastic FCM articles for repeated use

# Flowchart of legal text Regulation 10/2011

2.1.6. Repeated use materials and articles

1. If the material or article is intended to come into repeated contact with foods, the migration test(s) shall be carried out three times on a single sample using another portion of food simulant on each occasion. The
2. specific migration in the second test shall not exceed the level observed in the first test, and the specific migration in the third test shall not exceed the level observed in the second test.
3. Compliance of the material or article shall than be verified on the basis of the level of the migration found in the third test and on the basis of the stability of the material or article from the first to the third migration test.
4. The stability of the material shall be considered insufficient if migration is observed above the level of detection in any of the three migration tests, and increases from the first migration test to the third migration test. In
5. case of insufficient stability, compliance of the material shall not be established even in case the specific migration limit is not exceeded in any of the three tests.
6. However, if there is conclusive scientific proof that the level of the migration decreases in the second and third tests and if the migration limits are not exceeded on the first test, no further test is necessary.
7. Irrespective of the above rules, a material or article shall never be considered to comply with this Regulation if in the first test a substance that is prohibited from migrating or from being released in detectable quantities under Article 11(4) is detected.



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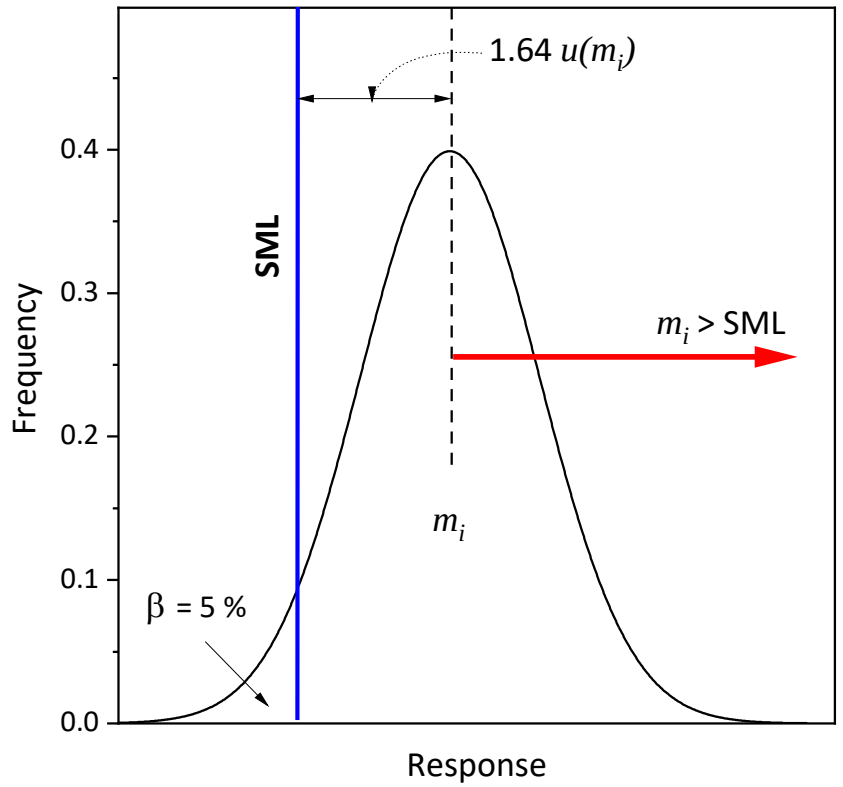
# Three ingredients

1. **Concentration** level of the migrating substance;
2. Associated measurement **uncertainty**;
3. **Statistical** rules used for comparison.

# Stats: $m_i$ > SML ?




According to **Reg. 2021/808**, $m_j$ **exceed** $SML$ *without any reasonable doubt* when:

$$m_i - 1.64\, u(m_j) > SML$$

which is equivalent to:

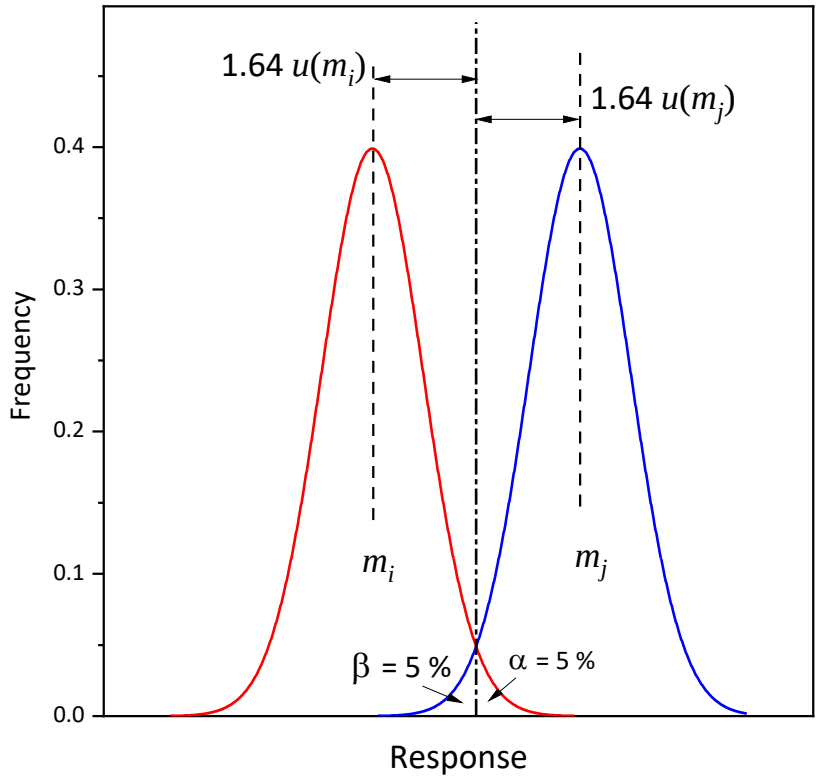
$$(m_i - SML)/u(m_i) > 1.64$$

where $u(m_i)$ is the **standard measurement uncertainties** (k = 1)



- Commission Implementing Regulation (EU) 2021/808 of 22 March 2021 on the performance of analytical methods for residues of pharmacologically active substances used in food-producing animals and on the interpretation of results …

# Stats: $m_i > m_j$ ?




According to **Reg. 2021/808**, $m_j$ **exceed** $m_i$ *without any reasonable doubt* when:

$$m_j - 1.64\,u(m_j) > m_i + 1.64\,u(m_i)$$

which is equivalent to:

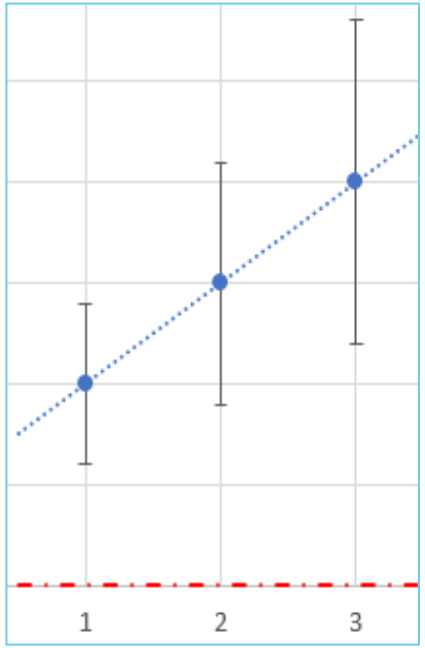
$$(m_j - m_i)/[u(m_j) + u(m_i)] > 1.64$$

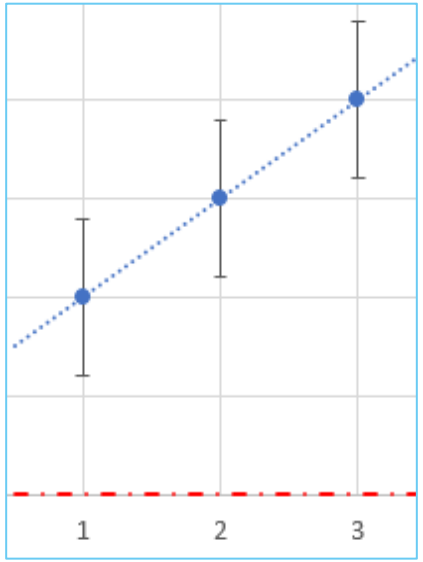
where $u(m_i)$ and $u(m_j)$ are the **standard measurement uncertainties** (k = 1)

# Measurement uncertainty

- Fitness for purpose approach (Reg (EC) No 333/2007)
- Modified Horwitz equations (CIR (EU) 2019/2093)
- QUAM (www.eurachem.org) ISBN 978-0-948926-30-3

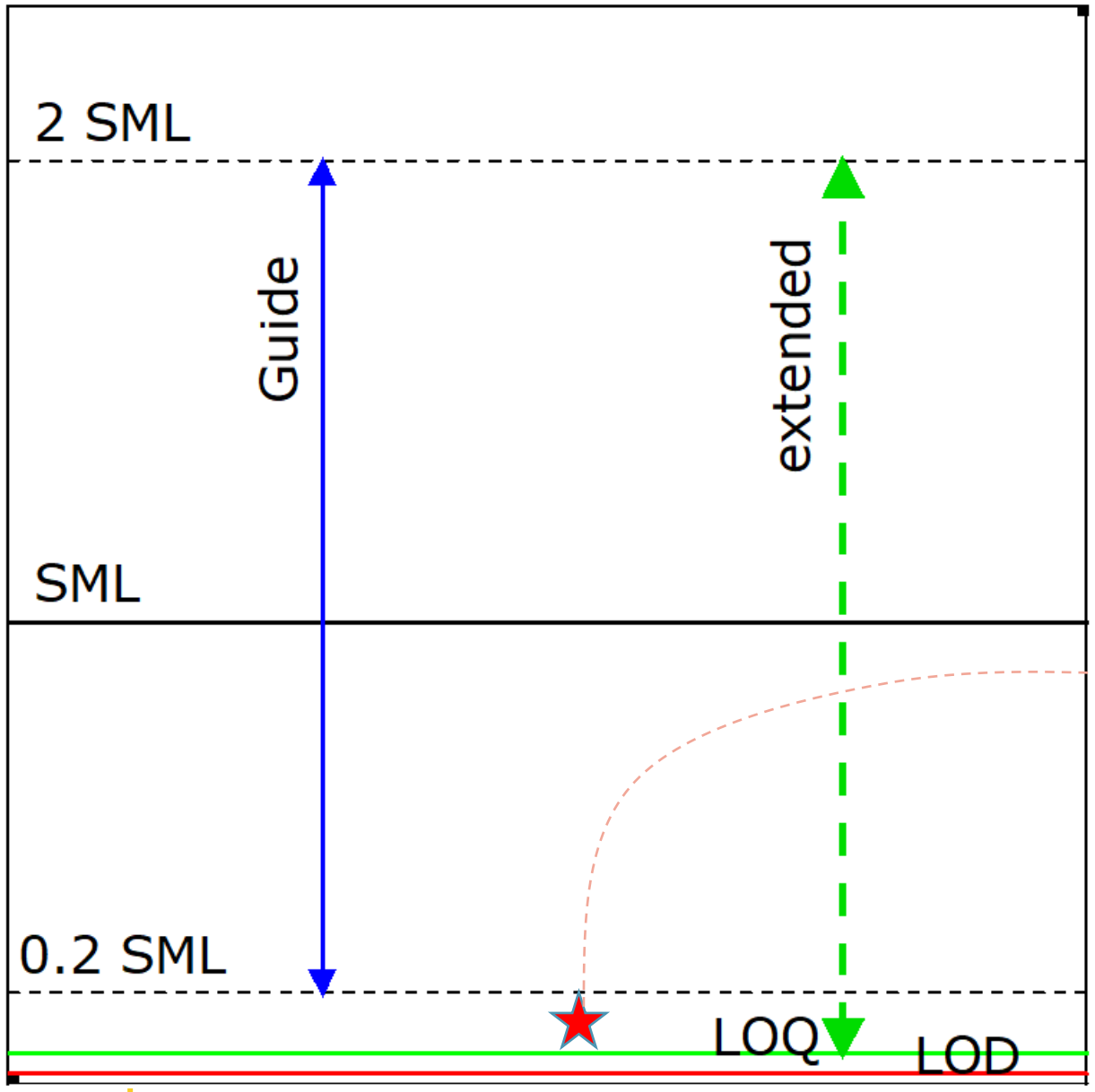
Realistic MU
$m_1 = m_2 = m_3$
Compliant




Underestimated MU
$m_3 > m_1$
Non-compliant

# Concentration levels




Comments from NRLs

- No increase expected for plastics where migration is driven by diffusion.
- (significant) increase from well-below SML within lifetime of FCM realistic?
- Define concentration range for establishing stability.
- Reporting range ⇔ Validation range

  * EURL Guide [0.2 SML; 2 SML]

    $m_i$ < 0.2 SML ➔ how to assess increase?

  * extend range down to LOD/LOQ (when method allows)

    $m_i \pm u(m_i)$ - even if below 0.2 SML

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# Stability rule

The Regulation states:

"*The specific migration in the second test* ***shall not exceed*** *the level observed in the first test, and the specific migration in the third test* ***shall not exceed*** *the level observed in the second test."*

So sufficient stability is proven when:

"$m_1 \geq m_2$" **AND** "$m_2 \geq m_3$" [**AND** "$m_1 \geq m_3$"],

the negation of which should read:

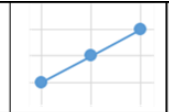
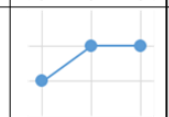
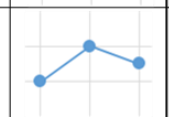
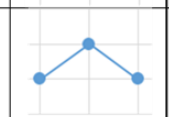
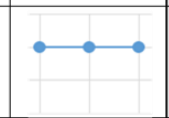
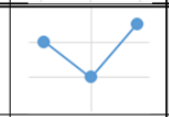
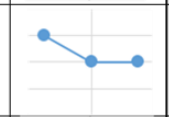
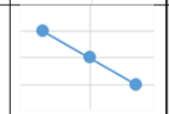
"$m_1 < m_2$" **OR** "$m_2 < m_3$" [**OR** "$m_1 < m_3$"]

which is equivalent to say **"*any increase in migration" may indicate insufficient stability*.**

This "logical" conclusion was questioned by many NRLs

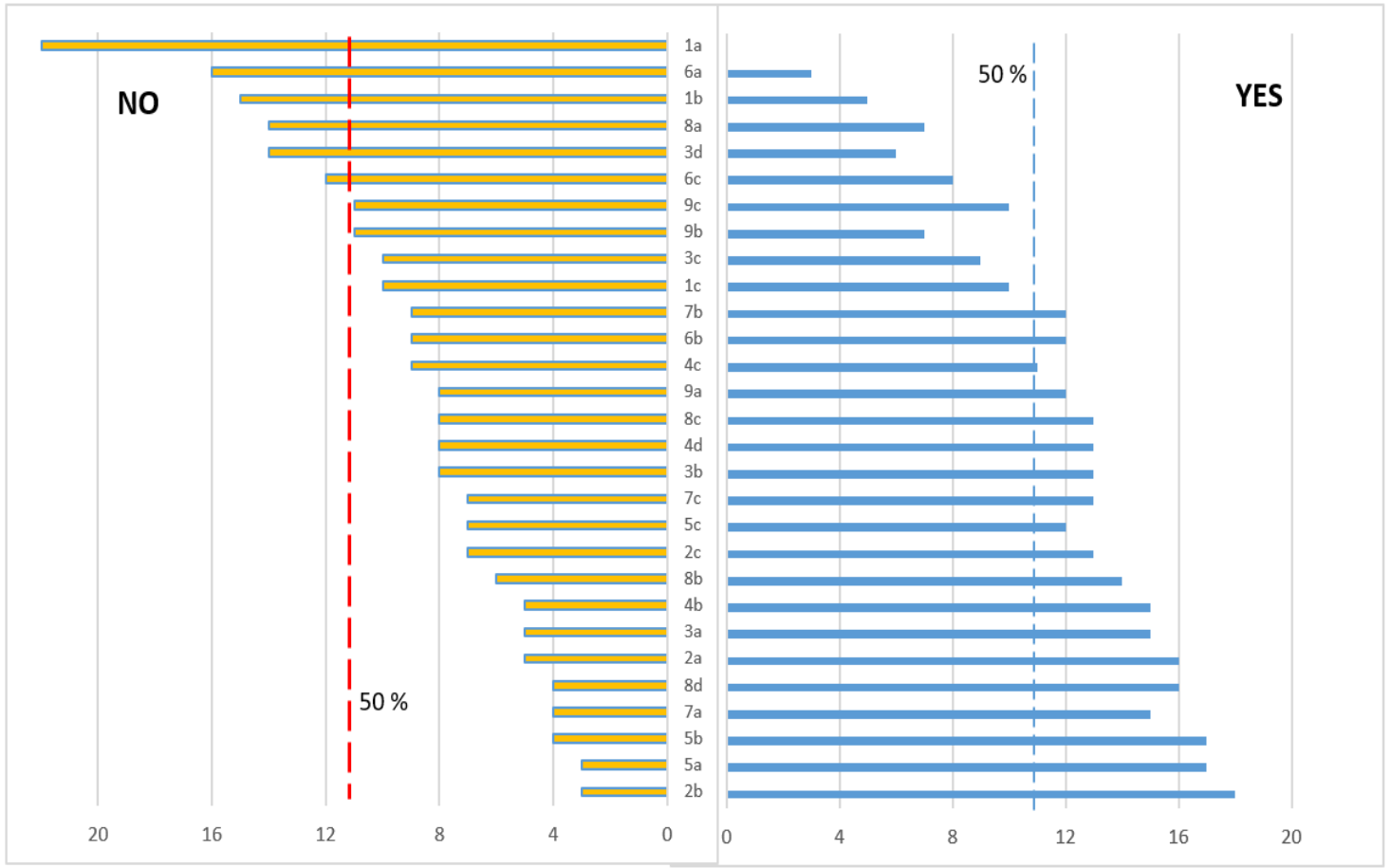
# The survey

- 29 cases using 13 trends
- Comparison of migration test results
- Harmonisation of measurement uncertainty calculation
- Stability evaluation below 20 % SML
- “Instability” criteria

| | | | | |
|---|---|---|---|---|
| m1 < m2 | m1 < m2 < m3 | |  | 1 |
| | m1 < m2 = m3 | |  | 2 |
| | m1 < m2 > m3 | m1 < m3 |  | 3 |
| | | m1 = m3 |  | 4 |
| | | m1 > m3 |  | 5 |
| m1 = m2 | m1 = m2 < m3 | |  | 6 |
| | m1 = m2 = m3 | |  | 7 |
| | m1 = m2 > m3 | |  | 8 |
| m1 > m2 | m1 > m2 < m3 | m1 < m3 |  | 9 |
| | | m1 = m3 |  | 10 |
| | | m1 > m3 |  | 11 |
| | m1 > m2 = m3 | |  | 12 |
| | m1 > m2 > m3 | |  | 13 |

# Survey Results

- 28 non-compliant cases
- 21 participants




- 70% agree with one-sided comparison of migration
- 50% welcome harmonised approach MU estimation
- 80% agrees migration below 20 % SML to be reported as “< 20 % SML”
- $m_2 > m_1$ often disregarded



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# To be (agreed &) recommended

- Harmonised MU: Horwitz or Uf or other? (Select one)
- Suggested decision rules:
  IF $(m_j - m_i) / [u(m_j) + u(m_i)] > 1.64$ THEN $m_j > m_i$
  IF $(m_i - \mathrm{SML}) / u(m_i) > 1.64$ THEN $m_i > \mathrm{SML}$
- Concentration range (validation):
  **from 0.2 SML** to 2 SML ➔ $m_i$ < 0.2 SML (even if detected)
  OR **from LOD** to 2 SML (extended) ➔ report $m_i \pm u(m_i)$ ➔ assessment
- Criteria for non-compliance: ***"shall not exceed"*** ➔ cf. ***"any increase"***

  - "$m_1 < m_2$" **OR** "$m_2 < m_3$" **OR** "$m_1 < m_3$" ?
    according to Section 2.1.6 of Annex V of Reg. (EU) No 10/2011; or
  - "$m_1 < m_2$" **AND** "$m_2 < m_3$" (hence "$m_1 < m_3$")? or
  - "$m_1 < m_2$" **OR** "$m_2 < m_3$" **AND** "$m_1 < m_3$"?
    - $m_2 < m_3$;
    - Or perform additional successive migrations

# Thank you

National Reference Laboratories

JRC FCM team





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# Mineral oil in food and FCM

# Mineral oil

***Major deliverables***:

- ✓ Guidance document for the determination of mineral oil hydrocarbons (cf. report EUR 29666 EN); update in preparation
- ✓ Two interlaboratory comparison rounds were organised for the determination of MOAH in IF (cf. reports JRC121915EN and JRC125669EN)
- ✓ The reference material SN500* was characterised for the MOSH/MOAH contents (report EUR 30990 EN)
- ✓ A virtual proficiency test is carried out assessing the quality of MOSH and MOAH chromatogram integration by expert laboratories (report EUR 31101 EN)
- ✓ Development and fine-tuning of an analytical method for the determination of mineral oil aromatic hydrocarbons (MOAH) in infant formula (IF); Standard Operating Procedure published and validation report in preparation

# Mineral oil

***Additional deliverables***:

- ✓ Procedure for the determination of the limit of quantification (in prepararation);
- ✓ EURL-FCM PT 2020/01 for determination of MOSH/MOAH in olive oil in paper and muesli (EUR 30787 EN)
- ✓ EURL-FCM PT 2022/01 for determination of MOSH/MOAH in olive oil; report in preparation
- ✓ Distribution of a Quality Control kit including a set of well-characterised samples (e.g., several mineral oils, spiked olive oils and 15 infant formulas.