

Technical guidelines for compliance testing in the framework of the plastic FCM Regulation (EU) No 10/2011

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Technical guidelines for compliance
testing

*In the framework of
Regulation (EU) No 10/2011
on plastic food contact
materials*

DRAFT FOR CONSULTATION

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Report EUR xxxxx xx

Guidelines supporting Regulation 10/2011

- **Technical guidelines on migration testing in support of the Regulation for plastics.**
 - ✓ **Understandable by non-specialist, Based on sound scientific data, Traceability, Fit for purpose**
- **Formation of a task force representing stakeholders**
 - ✓ **Industry/professional associations (CEFIC - PlasticsEurope - Flexipack Europe – EuPC)**
 - ✓ **Enforcement: 3 NRLs – DE, ES, UK**
 - ✓ **Individual experts: selected on expertise in migration testing**
- **Started end 2012 – 9 meetings**

- **Technical guidelines on migration modelling in support of the Regulation for plastics**

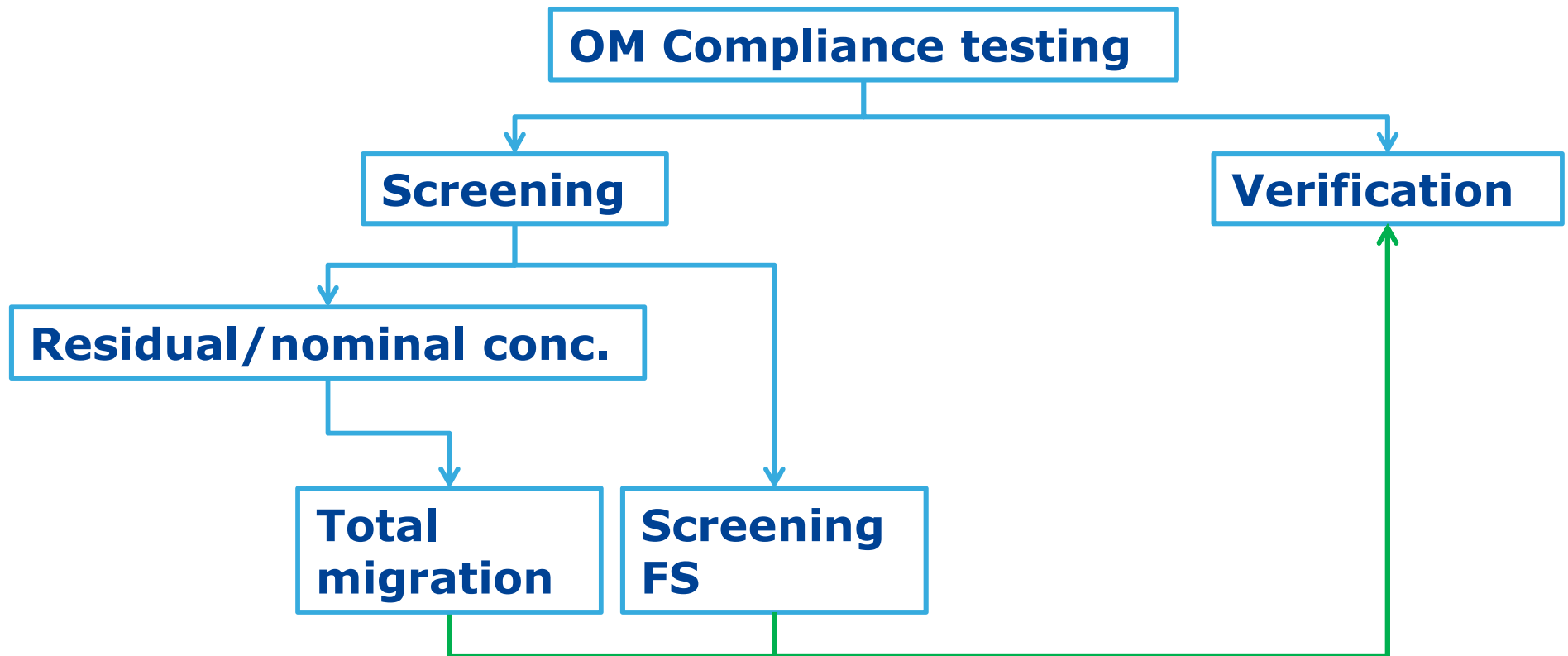
Document structure and contents

- **Sampling**
- **Materials and articles already in contact with food or using food as a simulant – testing for specific migration**
- **Verification of compliance with migration limits using food simulants**
- **Screening**
- **Analytical determination of migrants**
- **Reporting of the final migration test result**

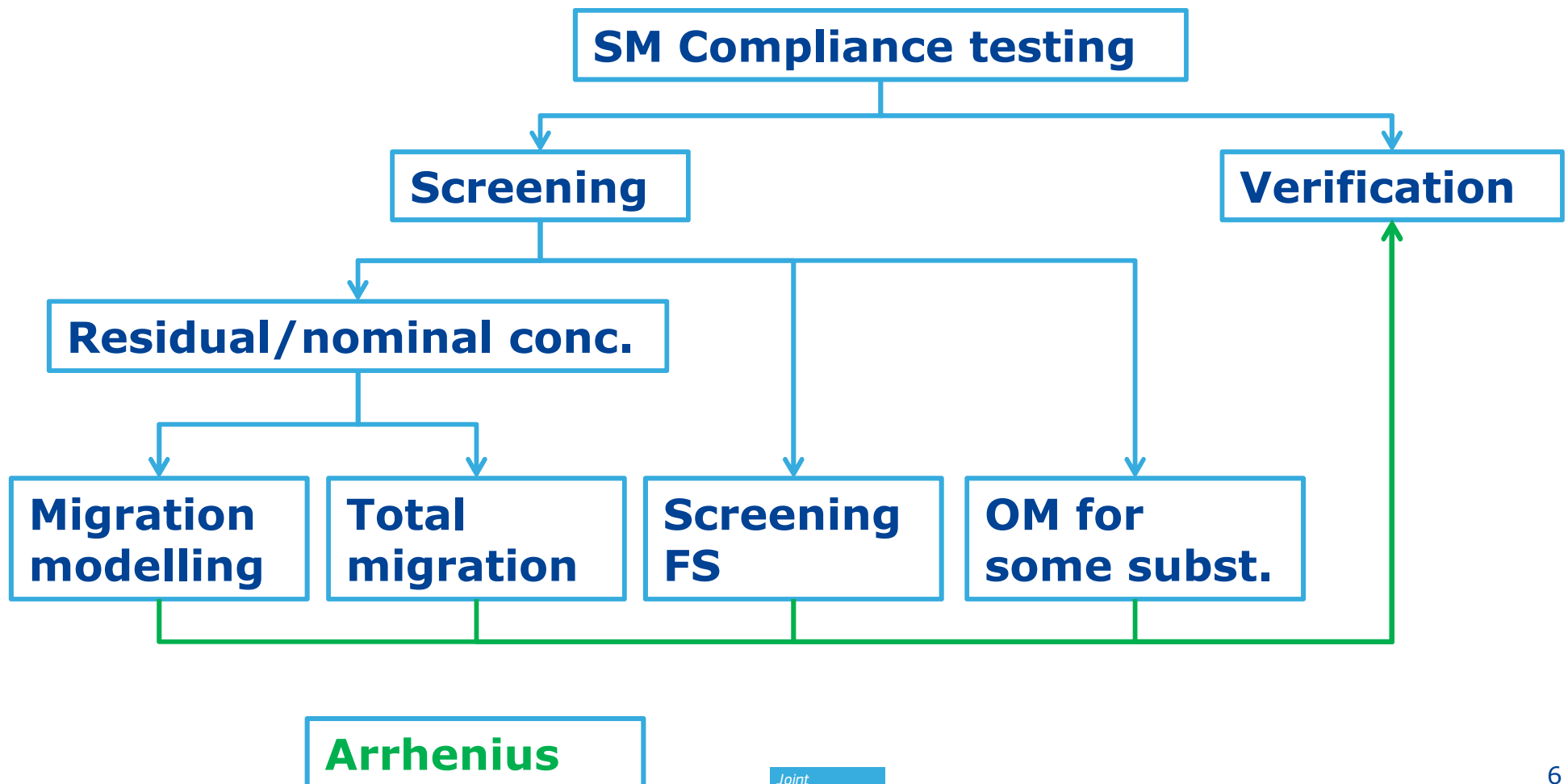
Annexes

- **Example of sampling protocol**
- **Abstract Fraunhofer IVV Report 'FPE Functional Barrier Project', 09.12.2011. Publication in preparation**
- **Cleaning procedures for food simulant E**
- **Background Table 5, 7 and 9**
- **Section 5.2.1 – Replacing specific migration by overall migration**
- **Section 5.2.5 background document**
- **Test method for overall migration into vegetable oil**
- **Test method for overall migration into water, aqueous food simulants, isooctane and ethanol 95%**
 - ✓ **CEN TC194 WG8 had first meeting**

OM compliance testing



SM compliance testing



Screening

- **AT LEAST AS SEVERE AS verification of compliance**
- **Screening approaches for specific migration (SM)**
 - ✓ **Migration modelling: multi-layers**
 - ✓ **Total mass transfer: thickness for known polymers**
 - ✓ **Screening food simulants: test conditions EtOH 95%, isooctane, FS E**
 - ✓ **Replacing specific migration by overall migration: generic SML; non-volatiles; volatiles**
 - ✓ **Use of Arrhenius equation**
 - ✓ **Functional barrier considerations: thickness for known polymers**
- **Screening approaches for OM testing**
 - ✓ **Total mass transfer: Residual/nominal concentration**
 - ✓ **Screening food simulants**

Screening – replacing SM by OM

OM in aqueous food simulants

- All non-volatile substances with generic SML
- All substances with a SML > OM analytical tolerance

OM in oil

- All non-volatile substances with generic SML (applying vacuum treatment)
- All substances with generic SML (no vacuum treatment)
 - ✓ bp < 300°C recovery requirements from FS
- All substances with a SML > OM analytical tolerance

Verification of compliance

Selection of

- **Food or food simulants**
- **Worst case contact time**
- **Worst case foreseeable contact temperature**

FCM already in contact with food

Preamble (35)

- **Substances found in food already in contact with FCM may originate from other sources, including other FCM**
- **Amount of a substance present in the food not originating from the tested FCM should not be taken into account for compliance with the Regulation**

Annex V section 1.4

- **"... the test results shall be corrected for the amount of that substance originating from the other source or sources before comparing the test results to the applicable specific migration limit"**

Selection of food simulants (SM)

- **One or more specific foods**
 - ✓ food simulant(s) in Table 2 of Annex III
- **Broad categories of non-specific foods, e.g. “acidic”**
 - ✓ food simulants according to section 2 of Annex III
- **“All types of food” in general**
 - ✓ food simulants A, B, D2
- **All types of foods and for substances not reacting with acidic food simulant or with acidic foods**
 - ✓ food simulants A and D2
- **Based on scientific arguments, in specific cases**
 - ✓ One most severe food simulant for that particular substance and/or material (e.g. PAA in FS B)

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Food simulants

- **A: ethanol 10%** (hydrophilic food)
- **B: acetic acid 3%** (hydrophilic food pH<4.5)
- **C: ethanol 20%** (alcoholic more lipophilic food)
- **D1: ethanol 50%** (lipophilic/alcoholic food; oil in water emulsion)
- **D2: vegetable oil;** (lipophilic food; surface free fats)
 - ✓ **unsaponifiable matter**
- **E: poly (2,6-diphenyl-p-phenylene oxide)** (dry food)
 - ✓ **Tenax®, MPPO**
- **Alternatives to food simulants:**
 - ✓ **technical reasons only;**
 - ✓ **Ethanol 95%, isooctane, FS E (≥100°C)**
 - ✓ **Conventional test conditions**

When use of FS D2 is not feasible

Overall migration

- Excessive absorption of oil
- Difficulty to recover the absorbed oil
- Presence of interfering substances in the recovery and determination of the absorbed oil
- Difficulty to determine of the accurate weight of the sample before and after contact with the oil
- Physical changes in the test sample (e.g. delamination)

Specific migration

- reaction of the substance with the simulant (e.g. PAA with oil)
- isolation of the substance from the oil not possible (e.g. waxes)
- unavoidable interferences from D2
- Insufficient analytical detection limit in D2

Contact time

1. Table 1, Annex V

2. $t > 30$ d & $T \leq RT \rightarrow$ Arrhenius (long term)

➤ 10 d @ 20°C

- all storage times at frozen condition
- including the freezing and defrosting processes if labelling or other instructions ensure that 20°C is not exceeded and
- the total time above -15°C does not exceed 1 day in total during the foreseeable intended use of the material or article.

➤ 10 d @ 40°C

- All storage times at refrigerated or frozen conditions including hot-fill conditions and/or heating up to $70^{\circ}\text{C} \leq T \leq 100^{\circ}\text{C}$ for maximum $t = 120/2^{((T-70)/10)}$ minutes
- any time at RT provided it can be demonstrated that migration of a substance is at equilibrium after 10 days at 40°C

Contact time

1. Table 1, Annex V

2. $t > 30$ d & $T \leq RT \rightarrow$ Arrhenius (long term)

➤ 10 d @ 50°C

- all storage times of up to 6 months at room temperature, including hot-fill conditions and/or heating up to $70^{\circ}\text{C} \leq T \leq 100^{\circ}\text{C}$ for maximum $t = 120/2^{((T-70)/10)}$ minutes

➤ 10 d @ 60°C

- storage above 6 months at room temperature and below, including hot-fill conditions and/or heating up to $70^{\circ}\text{C} \leq T \leq 100^{\circ}\text{C}$ for maximum $t = 120/2^{((T-70)/10)}$ minutes

Contact temperature – Table 2, Annex V

Contact temperature	test contact temperature
$121^{\circ}\text{C} < T \leq 130^{\circ}\text{C}$	$130^{\circ}\text{C} (*)$
$130^{\circ}\text{C} < T \leq 150^{\circ}\text{C}$	$150^{\circ}\text{C} (*)$
$150^{\circ}\text{C} < T \leq 175^{\circ}\text{C}$	$175^{\circ}\text{C} (*)$
$175^{\circ}\text{C} < T \leq 200^{\circ}\text{C}$	$200^{\circ}\text{C} (*)$
$T > 200^{\circ}\text{C}$	$225^{\circ}\text{C} (*)$

(*) as in Regulation (EU) No 10/2011

Hot fill derogation

Definition

'hot-fill' means the filling of any article with a food with a temperature not exceeding 100°C at the moment of filling, after which the food cools down to 50°C or below within 60 minutes, or to 30°C or below within 150 minutes.

➤ **Test 2 h @ 70°C.**

Including storage at room temperature

- **test conditions according to Table 1 and 2 or**
- **section 2.1.4 of Annex V of Regulation (EU) No 10/2011 depending on the duration of storage.**

Alternative FS test conditions

- **Physical changes under conventional test conditions → worst foreseeable conditions**
- **FS D2 is not feasible under worst foreseeable conditions of use → isooctane + ethanol 95% (+ FS E)**

Selected conventional test conditions for e.g. PET

Food simulant D2	isooctane	ethanol 95%	simulant E
10 d at 50° C	as D2	5 d at 20° C	no
10 d at 60° C	as D2	10 d at 20° C	no
2 h at 70° C	4 h at 60° C	0.5 h at 40° C	no
0.5 h at 100° C	12 h at 60° C	0.5 h at 60° C	as D2
1 h at 100° C	1 d at 60° C	1 h at 60° C	as D2

OM test conditions

- OM2 10 d at 40°C** Any long term storage at room temperature or below, including when packaged under hot-fill conditions, and/or heating up to a temperature T where $70^{\circ}\text{C} \leq T \leq 100^{\circ}\text{C}$ for a maximum of $t = 120/2^{((T-70)/10)}$ minutes.
- OM3 2 h at 70°C** Any food contact conditions that include hot-fill and/or heating up to a temperature T where $70^{\circ}\text{C} \leq T \leq 100^{\circ}\text{C}$ for maximum of $t = 120/2^{((T-70)/10)}$ minutes, which are not followed by long term room temperature or refrigerated storage.
- OM6 4 h at 100°C or at reflux** Any food contact conditions at a temperature exceeding 40°C , and with foods for which point 4 of Annex III assigns simulants A, B, C or D1.

OM test repeated use

- **Test shall be carried out three times on a single sample**
- **Fresh portion of food simulant each test**
- **OM (3rd) < OM (2nd) < OM (1st)**
- **Compliance OM (3rd) < OML**
- **Oil: OM (3t) – OM(2t) < OM(2t) – OM(t) < OM(t) < OML**

Contact conditions

- **Mono-layers**
 - ✓ **Known plastic 1-/2-sided**
 - ✓ **Unknown plastic**
 - **immersion and refer to one area**
 - **Food contact side test**
- **Multi-layers**
 - ✓ **1-side test**
- **Multi-material multi-layer**
 - ✓ **Absence of VCM migration**
 - ✓ **Not covered by 10/2011**

Polymer type	time/Temp	layer thickness L in [μm] for			
		molecular mass of migrant (g/mol)	100-250	251-500	501-750
LDPE, PP rubbery	10 d at 60° C	none	none	9600	3840
	10 d at 40° C	none	12000	3680	1440
	10 d at 20° C	10000	3520	1200	480
	2h at 100° C	none	16000	4880	1920
PET, PBT, PEN	10 d at 60° C	160	60	20	8
	10 d at 40° C	52	20	8	4
	10 d at 20° C	12	8	4	2
	2h at 100° C	100	40	12	6

Contact conditions

➤ **Multi-component/assembled articles**

✓ **Test individual components**

✓ **Test assembled article**

✓ **Preamble (37)**

- **Business operators use precisely controlled t-T food processing**

- **operated in accordance with good manufacturing practice.**

- **The exact worst foreseeable processing conditions will be representative for the actual migration**

- **The standardised testing conditions set out in Table 1 and 2 of Annex V may significantly over-estimate migration**

- **Consequently unreasonable burden on business operators**

✓ **Annex V section 2.1.3: if the material or article during its intended use is subjected only to precisely controlled time and temperature conditions in food processing equipment, either as part of food packaging or as part of the processing equipment itself, testing may be done using the worst foreseeable contact conditions**

Foreseeable use vs. labelling

Verification test conditions need to take into account conditions of use specified for the material

- **FCM shall be properly labelled to assure food safety**
- **Labelling should be in conformity with**
 - ✓ **the claimed use**
 - ✓ **the foreseeable use related to the functionality of the FCM**

Problem: claimed use is less stringent than foreseeable use

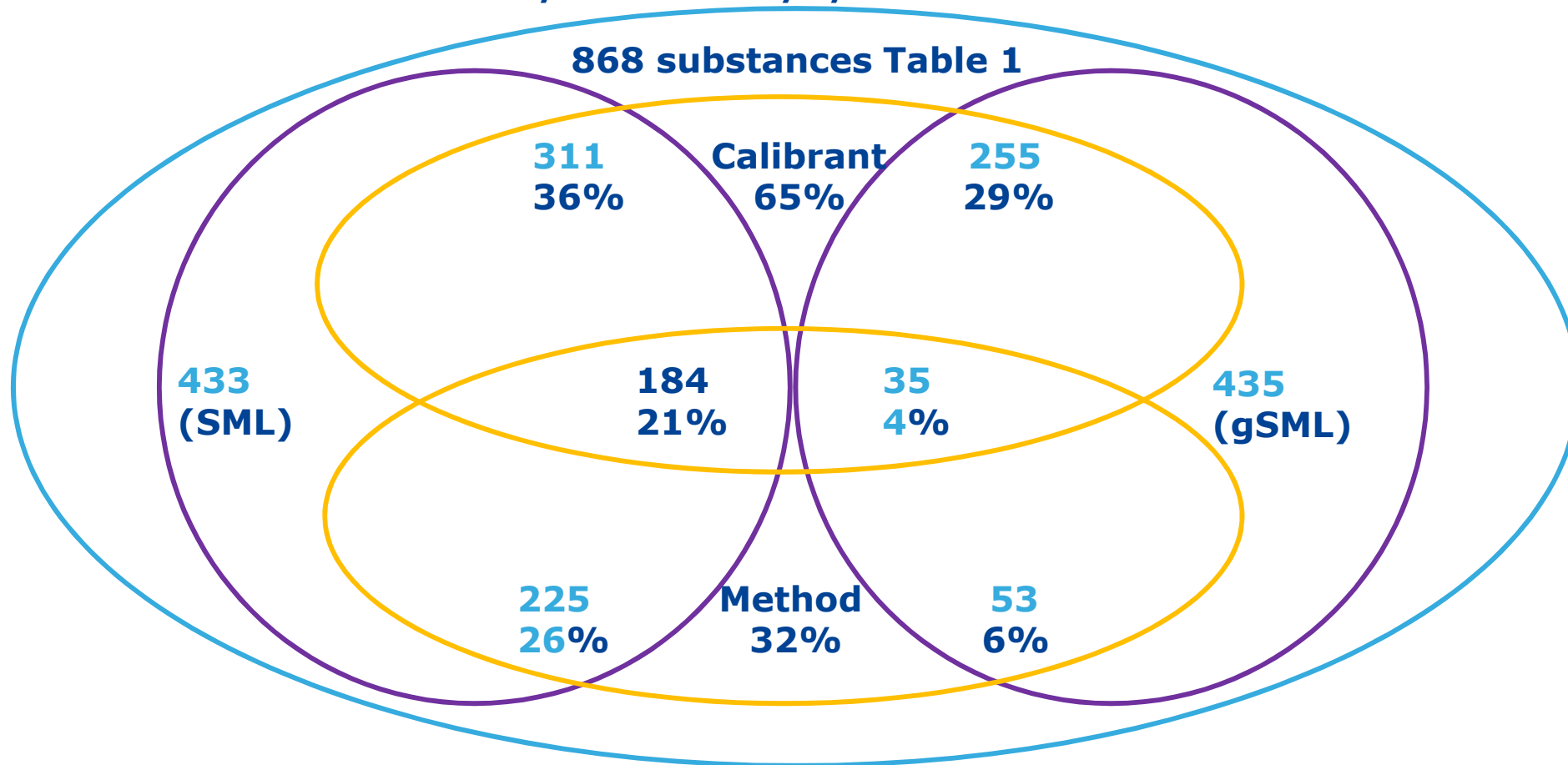
Solution: foreseeable use shall prevail over labelled use conditions in case of problem

Family approach

- **attempt to reduce the number of samples to be tested to a more manageable number**
- **selecting one or more individual products out of a larger group, the “product family” based on:**
 - ✓ **similarities in their composition and**
 - ✓ **structure**
- **justification for the decision shall be part of the supporting documents**

Available analytical methods and calibrants

Consolidated version of 10/2011 on 26/2/2015



Reporting of the final migration test results

- **Correction of the migration test result for the surface-to-volume ratio from experimental to actual contact**
- **Food simulant D2 reduction factor**
- **Fat Reduction Factor**
 - ✓ **SM in food or food simulant shall not exceed 60 mg/kg food before application of the FRF."**
- **Combination of correction factors, DRF and FRF, in specific migration**
 - ✓ **"When testing is performed in food simulant D2 or E and when the test results are corrected in application of the correction factor laid down in Table 2 of Annex III this correction may be applied in combination with the FRF by multiplying both factors. The combined correction factor shall not exceed 5, unless the correction factor laid down in Table 2 of Annex III exceeds 5"**
- **Choice of units for migration test results: caps**
- **Minimum information in the report**
- **Interpretation of results and assessment of compliance with limits**

Assessment of compliance with migration limits

Enforcement point of view:

if

analytical result – expanded measurement uncertainty > legal limit



sample is not compliant

Planning

- **To do**
 - **Update current draft of technical guidelines**

- **Forward cycle**
 - **Inter-service consultation 9/2016**
 - **Endorsement Standing Committee**
 - **Target Q4 2015 for publication EN version together with 6th amendment of 10/2011**
 - **Translation in DE, FR, ES and Chinese**



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*Thank you
for your attention*



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