

# Migration tests on repeated use materials and articles

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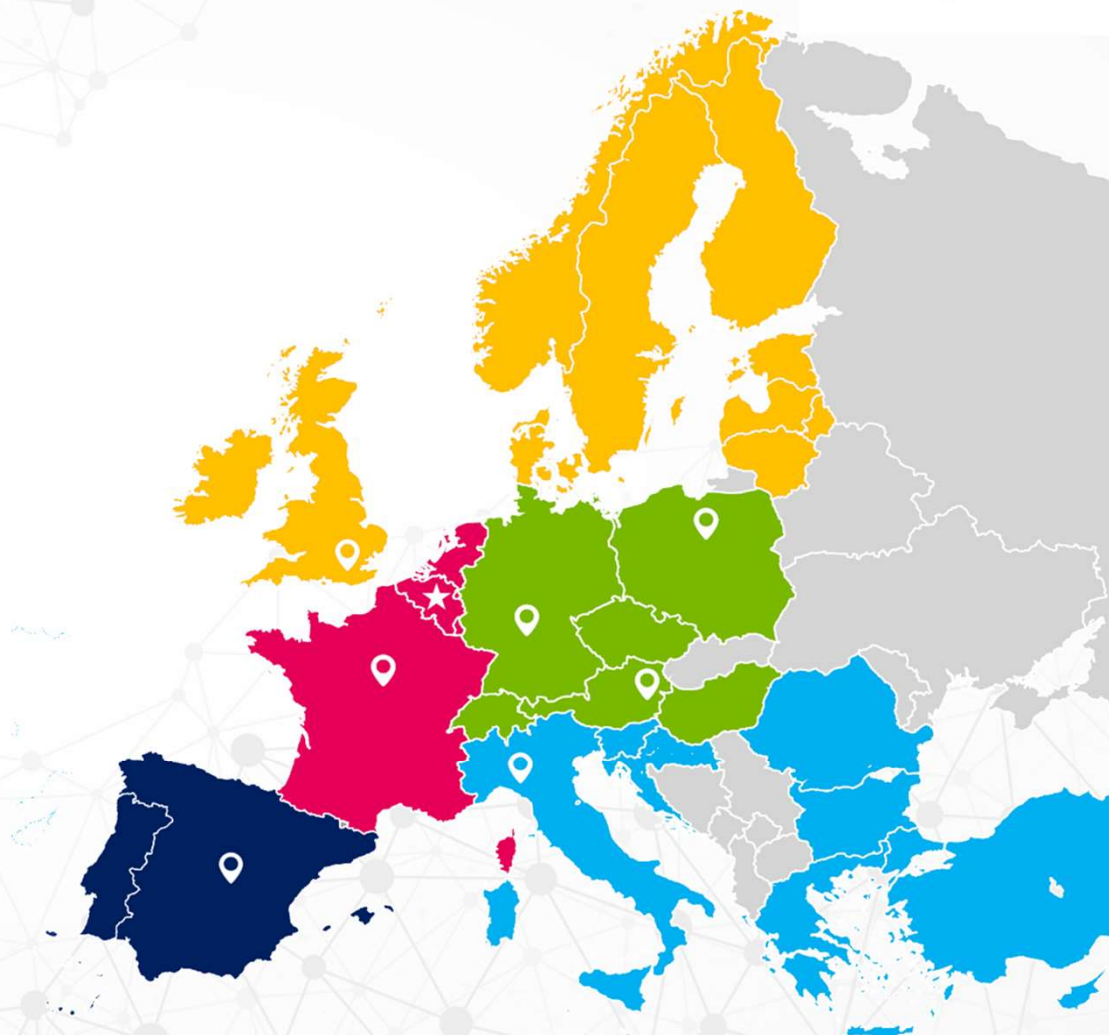
# Brief introduction on PlasticsEurope



A foothold across Europe



**Headquartered in Brussels,**  
PlasticsEurope has regional  
offices in 7 countries and national  
representation teams in the  
remaining EU 20, as well as in  
Norway, Switzerland, Turkey, and  
the UK



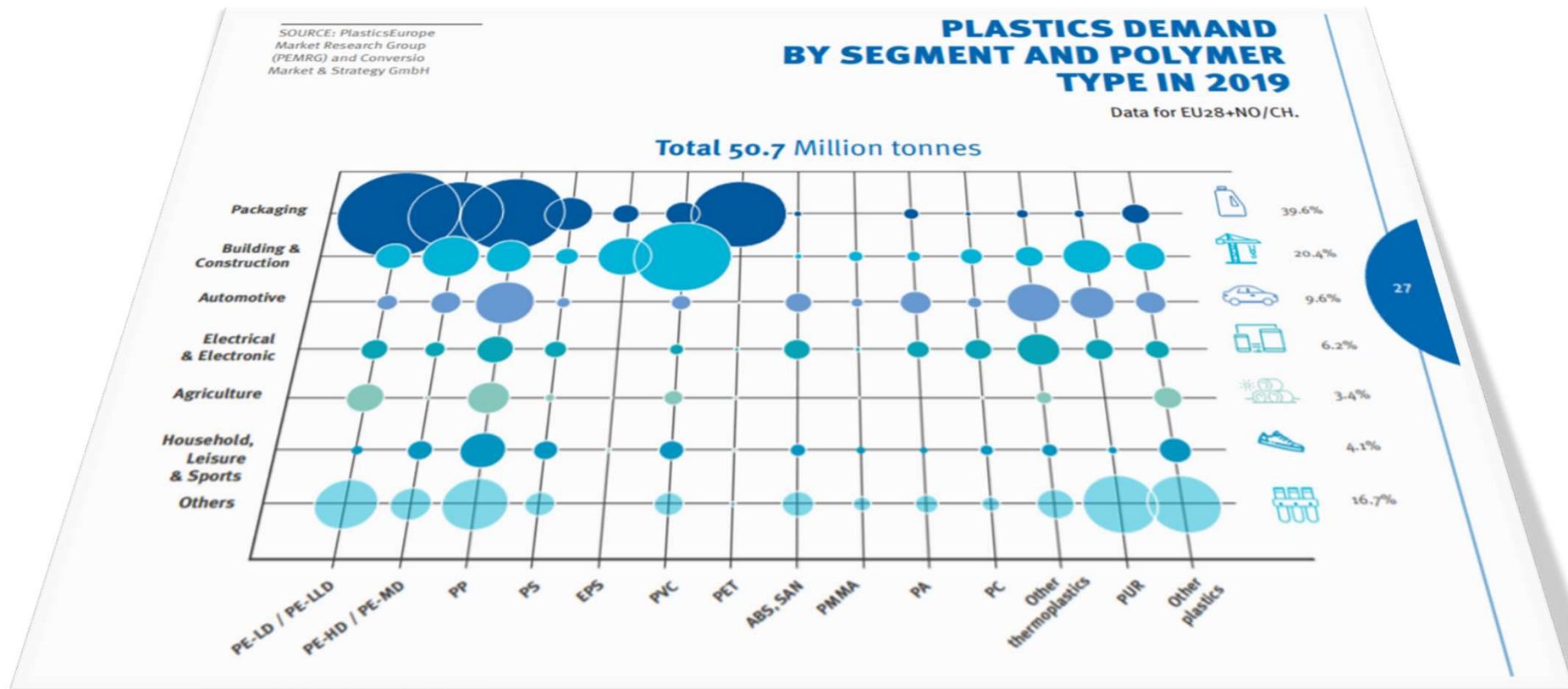


# Our purpose and strategic focus

PlasticsEurope is a **catalyst for the plastics industry**, to deliver Sustainable, safe and circular solutions valued by society.



# European plastics demand by segments and polymer types



**2019 EUROPEAN Plastics converter demand: ~51 millions Tonnes/y**  
**Food Contact Application: > 10-12 millions Tonnes/y (~50% packaging +...)**

# Commission Regulation (EU) 2020/1245



# Regulation (EU) 2020/1245

- Published on 2 September 2020
  - Also known as the 15<sup>th</sup> amendment of Commission Regulation (EU) No 10/2011 of 14 January 2011

- Contains a.o.

- Restrictions (SML's) for metals in Annex II
- Requirements for genotoxic substances in Annex IV (6)
- Transition period

[https://www.plasticseurope.org/application/files/8416/2125/8683/15th\\_amendment\\_interpretation\\_1\\_1.\\_May\\_2021\\_clear\\_version\\_17.05.2021.pdf](https://www.plasticseurope.org/application/files/8416/2125/8683/15th_amendment_interpretation_1_1._May_2021_clear_version_17.05.2021.pdf)

- Standardised conditions for OM testing (Table 3)
- **Revised requirements on repeated use materials and articles**



## Definition Repeated use materials and articles

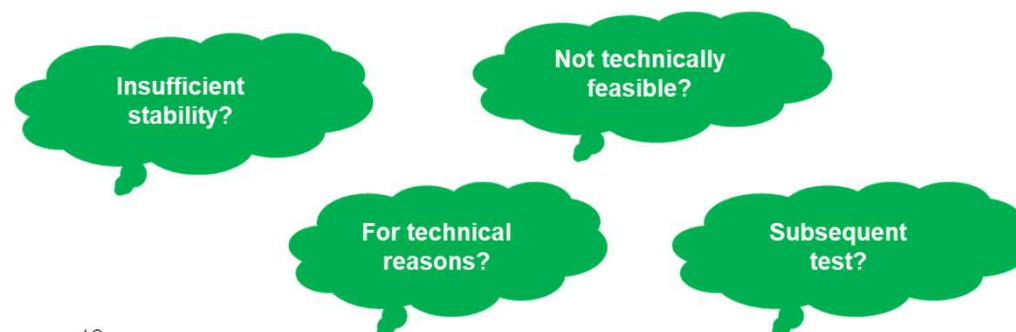
- Union Guidelines on Regulation (EU) No 10/2011 (Section 2.2)

“An article intended to be used several times that comes into contact with different portions of foods during its lifetime. For example, kitchenware, reusable containers or components of packaging machinery”



## Regulation (EU) No. 2020/1245

- Points 2.1.6 and 3.3.2 of Annex V of Regulation (EU) No. 10/2011 have been completely replaced by Regulation (EU) No. 2020/1245 (15th Amendment)
  - Main reason: the legal text of point 2.1.6 and 3.3.2 were not in line
  - A.o. the second subparagraph of point 2.1.6 did not contain the requirement that the migration should not increase between the subsequent tests as stated in the first paragraph of point 2.1.6
- More complicated as thought before
- The ‘the devil is in the details’
- To draw a compliance conclusion you always have to view the full picture
- Lacking definitions/guidance documents



# Regulation (EU) No. 2020/1245

- Point 2.1.6 Repeated use materials and articles

*“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 **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.*

Equal or lower

*Compliance of the material or article shall then 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. 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 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. 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.*

*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.’*

## Point 2.1.6 Repeated use materials and articles

- 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.
  - The result of the 3rd migration test is normative for compliance
- 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.
  - If migration increases between the 1st and the 3rd test, the material is considered to be instable



- In 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.
  - In case of an instable material, SM compliance shall not be established, even if  $SM < SML$
- 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.

## Point 2.1.6 Repeated use materials and articles

- Applies to SM testing
- SM result third test  $\leq$  second test  $\leq$  first test
- SM of the third test is normative for compliance
- The material must be stable during the SM tests
- If on the basis of scientific evidence, SM decreases in the second and third tests and if SM does not exceed the SML in the first test, the first test alone will be sufficient
- Does not apply to substances subject to Article 11(4), if in the first test the SM  $>$  Non-DL



## Regulation (EU) No. 2020/1245

### ■ Point 3.3.2 Repeated use materials and articles

*“The applicable overall migration test shall be carried out **three times** on a single sample using **another portion** of food simulant on each occasion. The migration shall be determined using an analytical method in accordance with the requirements of Article 34 of Regulation (EU) 2017/625 of the European Parliament and of the Council. The **overall migration** in the second test shall be **lower than** in the first test, and the overall migration in the third test shall be **lower than** in the second test. Compliance with the overall migration limit shall be verified on the basis of the level of the overall migration found in the third test*



*If it is not technically feasible to test the same sample three times, such as when testing in vegetable oil, the overall migration test can be carried out by testing different samples for three different periods of time lasting one, two and three times the applicable contact test time. The difference between the third and the second test results shall be considered to represent the overall migration. Compliance shall be verified on the basis of this difference, which shall not exceed the overall migration limit. In addition, the difference between the second and the first test results shall be lower than the first test results and the difference between the third and the second test results shall be lower than the difference between the second and the first test results.*

*By derogation from the first paragraph, if, on the basis of scientific evidence, it is established that for the material or article being tested the overall migration decreases in the second and third tests and if the overall migration limit is not exceeded in the first test, the first test alone shall be sufficient”*

# Regulation (EU) No. 2020/1245



- If it is not technically feasible to test the *same sample three times*, such as when testing in *vegetable oil*, the overall migration test can be carried out by testing different samples for three different periods of time lasting one, two and three times the applicable contact test time.
  - e.g. 10 days ► 20 days ► 30 days
- The difference between the *third* and the *second test results* shall be considered to represent the overall migration. Compliance shall be verified on the basis of *this difference*, which shall not exceed the overall migration limit.
- In addition, the difference between the *second* and the *first test results* shall be lower than the first test results and the difference between the *third* and the *second test results* shall be lower than the difference between the *second* and the *first test results*.



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## Point 3.3.2 Repeated use materials and articles

- Applies to OM testing
- OM result third test < second test < first test
- OM of the third test is normative for compliance
- An analytical method acc. to Article 34 of Regulation (EU) No. 2017/625 should be used
  - CEN standards
- The material must be stable during the OM tests
- If is not technically feasible to carry out a OM test in food simulant D2, an OM test should be carried out at different contact times, lasting one, two and three times the applicable contact time
  - The difference between the third and the second test results shall be considered to represent the OM  
Compliance shall be verified on the basis of this difference, which shall not exceed the OML
  - The difference in OM result second and first test < result first test and the difference in OM result third and second test < second and first test
- If on the basis of scientific evidence, OM decreases in the second and third tests and if OM does not exceed the OML in the first test, the first test alone will be sufficient and the OM with different contact times (see above) can be omitted

## Regulation (EU) No. 2020/1245

- Recital (29)

Following in time,  
order, or place

*“Point 2.1.6 of Annex V to the Regulation requires three subsequent tests for articles and materials that are placed in repeated contact with food.*

➤ Subsequent or consecutive? At least you have to do it consistently

Following one after  
the other in order

*“[...] However, in some instances, such as when migration is low relative to the **measurement error**, it may be difficult to establish a decreasing trend analytically and it would require complex rules. Therefore it is appropriate to only require that a the migration established in a subsequent test does not exceed that of the previous test, to clarify this principle in the Regulation, and to establish that a material that shows increasing migration over the subsequent tests should never be considered compliant.”*

➤ The analytical tolerance of the migration method is an important factor which you have to take into account

## Analytical tolerances of OM tests

- Analytical tolerances of the **overall migration tests** are no longer included in Regulation (EU) No.10/2011 or its guidance documents.
  - They were part of Commission Directive 2002/72/EC, which has been repealed by Regulation (EU) No.10/2011
  - The analytical tolerances can be found in
    - CEN standard series 1186 (These standards have not been updated since 2002)
    - JRC Guidelines on testing conditions for articles in contact with foodstuffs 1<sup>st</sup> Edition (2009)
- The following analytical tolerances are allowed:
  - *12 mg/kg food or 2 mg/dm<sup>2</sup> for all aqueous food simulants (and food simulant D1)*
  - *20 mg/kg food or 3 mg/dm<sup>2</sup> for all fatty food simulants and substitute test media*

An example

	1 <sup>st</sup> test	2 <sup>nd</sup> test	3 <sup>rd</sup> test
OM test in food simulant D2 (10 d 40°C)	7 mg/dm <sup>2</sup>	8 mg/dm <sup>2</sup>	6 mg/dm <sup>2</sup>
OM test in food simulant A (10 d 40°C)	<1 mg/dm <sup>2</sup>	2 mg/dm <sup>2</sup>	<1 mg/dm <sup>2</sup>



# Regulation (EU) No. 10/2011

- Annex V Chapter 3 point 3.1 (Standardised testing conditions)

- Applies to standardised testing conditions for OM

*“If it is found that carrying out the tests under the contact conditions specified in Table 3 causes **physical or other changes** in the test specimen which do not occur under worst foreseeable conditions of use of the material or article under examination, the migration tests shall be carried out under the worst foreseeable conditions of use in which these physical or other changes do not take place”*

- The material should be stable during migration tests

Instable or not  
technical  
feasible?

- If physical or other changes occur under used migration conditions, is the material or article suitable for the intended use?



(Smudge marking see post wiping with Kimwipe paper )



(Post molding surface irregularities )

## Regulation (EU) No. 10/2011

- Annex V, Chapter 3, section 3.2. (Substitute overall migration tests for tests with food simulant D2)  
*“If it is **not technically feasible** to perform one or more of the tests OM0 to OM6 in food simulant D2, migration tests shall be done using ethanol 95 % and isooctane. In addition a test shall be done using food simulant E in case the worst foreseeable conditions of use exceed 100 °C. The test that results in the highest overall migration shall be used to establish compliance with the Regulation”*
- Annex V, Chapter 3, section 3.4.2. (Food simulant substitute)  
*“To screen for overall migration, food simulants can be replaced if based on scientific evidence the **substitute food simulants** result in migration that is at least as severe as migration that would be obtained using the food simulants specified in Annex III.”*



Regulation (EU) No. 10/2011 does not contain test conditions for the use of ethanol 95 % and isooctane, and Commission Directive 97/48/EC has been finally repealed by Regulation( EU) No. 10/2011. We advise you to follow the test conditions in table 4 of the JRC Guidelines on testing conditions for articles in contact with foodstuffs (2009)



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# Regulation (EU) No. 10/2011

- A similar paragraph exists for SM testing (Annex V, Chapter 2, section 2.1.3(iv))
- Annex V, Chapter 2, section 2.2.4. (Food simulant substitutes)

*“To screen for specific migration, food simulants can be replaced by substitute food simulants if it is based on scientific evidence that the substitute food simulants result in migration that is at least as severe as migration that would be obtained using the food simulants specified in Section 2.1.2”.*



## 2.1.2. Choice of food simulant

Materials and articles intended for contact with all types of food shall be tested with food simulant A, B and D2. However, if substances that may react with acidic food simulant or foods are not present testing in food simulant B can be omitted.

## EFSA Note for guidance for petitioners

- Paragraph 2.8.3.1 of chapter IV EFSA Note for guidance ("Note for guidance for petitioners presenting an application for the safety assessment of a substance to be used in food contact materials prior to its authorisation") of July 2008 (repealed by the Note of Guidance of March 2017) states:

*"[...] that values obtained with 95% (v/v) ethanol tend to give slightly lower results compared to olive oil for most polyolefins"*

*"[.....] it should be noted that some special types of polyolefins may give migration values with isooctane higher of those expected in the real use [...]"*

*"Also polystyrene containing more than 6.5% of polybutadiene and/or mineral oil may give high results, while polyamides may give low results"*

## Draft JRC document on compliance testing\*

- *“Only in cases where it is demonstrated that for **technical reasons** the verification of compliance with an SML is not feasible in any vegetable oil within the specification of food simulant D2 the approach below can be applied to perform verification of compliance for fatty foods”*
  - *In the determination of the **OM***
    - *Excessive absorption of oil*
    - *Difficulties to recover the absorbed oil with any of the known methods. This may occur in some high temperature applications.*
    - *Presence of interfering substances in the recovery and determination of the absorbed oil*
    - *Difficulties to determine of the accurate mass of the sample before and after contact with the oil*
    - *Physical changes in the test sample (e.g. delamination)*
    - *Substitute test OM 8 and/or OM 9 are not suitable according to the selected test conditions*
  - *In the determination of the **specific migration***
    - *Reaction of the substance with the food simulant (e.g. primary amines with oil)*
    - *Physical or chemical properties of the substance that prevent isolation of the substance from the oil.*
    - *Unavoidable interferences from the food simulant D2*
    - *Insufficient analytical detection limit of the substance in food simulant D2”*



## OM in food simulant D2

- The revised Point 3.3.2 prescribes a method in case it is not **technically feasible** possible to carry an OM in food simulant D2
- Different CEN standards 1186 such as 1186-2, -4 or -6 describe the OM in food simulant D2
  - “The specimens will usually retain absorbed vegetable oil that is extracted and determined quantitatively by means of GC after conversion to methyl esters”
  - “Migration into the vegetable oil is calculated by subtracting the mass of vegetable oil retained by the test specimen from the mass of the test specimen after removal from the vegetable oil, then subtracting this mass from the initial mass of the specimen”



**Not possible to carry out OM  
repeated use tests on the same test  
sample**

**Not technically  
feasible**



- > After this, the test material cannot longer be used for a subsequent migration test or becomes unreliable
- > Why has the COM not aligned Point 3.3.2 with Annex V, Chapter 3 section 3.2?

## Reliability migration data

- In contrast to SM, OM can not be spiked with a reference substance, calculated or modelled
- Round-robin migration tests have shown, that the determined OM of a standard material can strongly vary between the various participants (accredited laboratories)

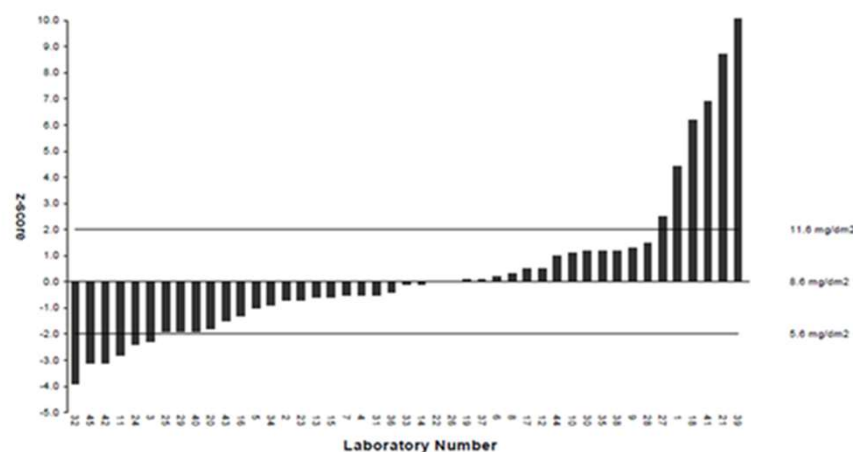


Figure 1: z-Scores for Overall Migration of Plastic Film into Olive Oil (8.6 mg/dm<sup>2</sup>)

- Laboratories conducting migration tests must have experience with migration testing, especially in food simulant D2. OM in food simulant D2 requires 'Fingerspitzen Gefühl' (skills)
- Migration tests have always be carried out in triplicate (see CEN standards)

# Actions

- The revised legal text of Points 2.1.6 and 3.3.2 needs further clarification from the COM, due to lacking guidance or requirements
  - Clarification on definitions will be needed
  - Alignment/harmonization with other paragraphs in Regulation (EC) No.10/2011 is strictly needed
- We also ask the COM to publish the draft JRC document on compliance testing from 2016
  - This document it is crucial to carry out compliance tests adequately



**It leads to confusion/  
contradiction**

**>** PlasticsEurope and 6 other plastic value associations have send a letter to JRC

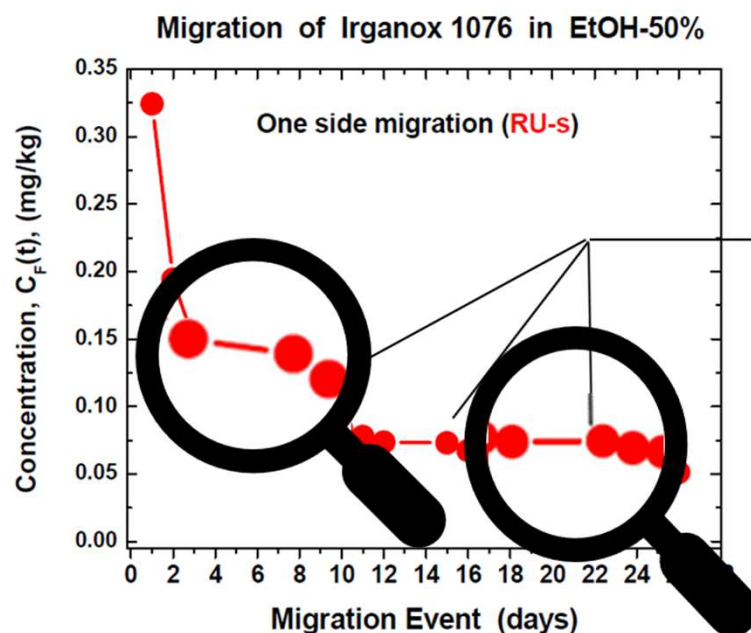
## Recommendations

- Be always critical on the obtained migration results
- If migration is borderline (close to DL or to ML), the risk of drawing the wrong conclusion increases
  - Check the differences in the triplicate results
  - Check if the obtained migration results fit with your expectations (based on previous results or comparable materials)
  - Ask questions at the used laboratory, manufacturing (or supplier) if the material has been changed, that something has been changed in production or during the migration test
  - Know the score of the used laboratory in a round-robin testing program
  - In case of doubt, retest the material (use always a new fresh sample)

	1 <sup>st</sup> test	2 <sup>nd</sup> test	3 <sup>rd</sup> test
OM in 3% HAc (2h 70°C)	2,9 mg/dm <sup>2</sup>	3,7mg/dm <sup>2</sup>	
OM in 95% EtOH (2h 60 °C)	8 mg/dm <sup>2</sup>	4 mg/dm <sup>2</sup>	4 mg/dm <sup>2</sup>
OM in iso-octane	6,7 mg/dm <sup>2</sup>	3,9 mg/dm <sup>2</sup>	4,9 mg/dm <sup>2</sup>
OM in water (10days 40°C)	3 mg/dm <sup>2</sup>	6 mg/dm <sup>2</sup>	3 mg/dm <sup>2</sup>

## Recommendation

- Since there is no information about the time between the subsequent migration trials, a suggestion is to wait 24 h between migration experiments to establish the equilibrium within the test sample
  - It represents more the use of a repeated use article than in a consecutive migration test




 Leads to an underestimation !

