European Commission

JRC SCIENCE AND POLICY REPORTS

Training workshop "Safety of food contact materials: Technical Guidelines for Testing Migration under Regulation (EU) No 10/2011"

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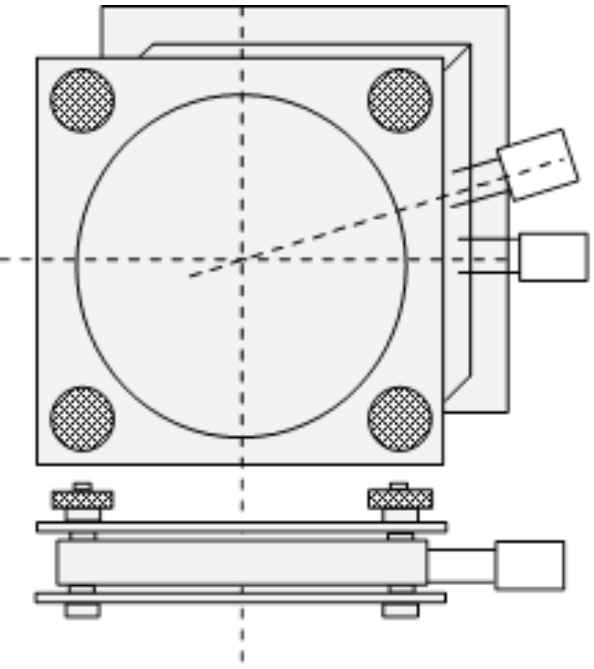
2014



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Abstract

This report outlines the results of the Workshop Technical Guidelines on Migration Testing under Regulation EU No 10/2011

Report of the Workshop Technical Guidelines on Migration Testing under Regulation EU No 10/2011

On the 8th of October 2014 the EURL organized a workshop on the progress of the drafting of the technical guidelines on compliance testing of plastic food contact materials under Regulation (EU) No 10/2011. This work also led to proposals for changing the Regulation. The meeting focused on participation by members of our EURL-NRL Network on food contact materials and by persons working on food contact materials in Enlargement and Integration countries. Other stakeholders in food contact materials could also participate. The agenda of the workshop is in Annex 1. Eddo Hoekstra (JRC) introduced the aim of the workshop and the background of the technical guidelines on compliance testing and its structure (Annex 2).

Oliver Kappenstein (BfR, NRL-DE) outlined the chapter on sampling of food contact materials differentiating between sampling for verification of compliance by official controls and compliance testing by industry (Annex 3). This chapter remains very general for official controls since many member states have specific guidelines for sampling. From the industry perspective, the business operator manufacturing a material or article may look for similarities in the composition and structure of their materials or articles to justify selecting one or more individual products out of a larger group, the "product family", as the worst case representative of that group. The justification for the decision to put products in one family and selecting a worst case representative of that group should be part of the supporting documents.

Emma Bradley (FERA, NRL-UK) explained the approach of compliance testing for already-packed food and in the case that food is used for testing migration (Annex 4). An open issue is how to test the migration into concentrated foods that need to be reconstituted before consumption. Can the migration result be corrected for dilution and under which conditions? A second question is if you can state a food compliant if the analysis has been done well before expiry date? Here several approaches are available depending on the material, contact conditions, equilibrium, period until expiry date, e.g. retest on expiry date. Another issue is the presence of other sources for the migrant then the packaging, e.g. phthalates.

Birgit Faust (CEFIC/PlasticEurope) presented the compliance testing of food contact materials using food simulants (Annex 5). It comprises the selection of the most severe contact conditions, i.e contact time, contact temperature, food simulant and surface-to-volume ratio, with food under worst foreseeable conditions of use of the food contact material. For most applications migration tests need to be performed using two or more food simulants, however, in specific cases testing may be reduced to a single food simulant, which is most severe for that particular combination of migrant and material, based scientific arguments. The specifications of vegetable oil are proposed to be changed into less than 1% of unsaponifiable matter. Under very specific conditions the food simulant vegetable oil may be replaced by a combination of iso-octane and ethanol 95% and for contact temperatures above 100°C food simulant E. Since iso-octane and ethanol 95% can only be used up to a contact temperature of 60°C and since they may extract more that vegetable oil separate contact time-temperature conditions are set. The highest result shall be compared with the overall or specific migration limit.

The contact temperature conditions above 175°C is proposed to split into two ranges: 175°C < T ≤ 200°C testing at 200°C and T > 200°C testing at 225°C. The hot fill conditions, i.e. articles filled with hot food which during a period of less than 15 minutes is at a temperature of 70°C<T≤100°C ('hot fill'), and which is not intended

for use at temperatures above 100°C, only the test at 2 hours and at 70°C shall be carried out, is proposed to be a derogation from the Table 1 and 2 of Annex V of the Regulation.
Roland Franz (IVV-Fraunhofer Institute) explained how mono-layer materials can be tested. For mono-layers testing can be done by immersion (Annex 6). However, the thickness of the test specimen determines whether the migration can be referred to the area of one side or two sides. For several materials the minimum thicknesses are tabled for which the migration can be referred to the area of two sides depending on the time-temperature conditions and the molecular mass of the migrant. The thicknesses in the table are based on the thickness were 99% of the migrant is still present and this thickness is multiplied by two. In a similar way the thickness where total mass transfer to food takes place (from 2-sides?) are based on the thickness were 99% of the migrant is still present and this thickness is divided by two. The functional barrier layer is the layer thicknesses were 100% of the migrant is still present.
Rainer Brandsch (MDCTec) presented the food simulant ranking for the different food characteristics (Annex 7). However for vegetable oil various organic solvents can be more severe from a solubility of the migrant point of view. If swelling occurs then contact time-temperature conditions need to be adapted. Most experience and experimental data are available for ethanol 95% and iso-octane and these solvents span a large polarity range of migrants. The recommendation on alternative food simulants selection is based on the rule "similar solves similar", i.e. the closer the polarity of the migrant and the simulant is, the better the solubility of the migrant will be in the simulant. As a measure of polarity the octanol to water partition coefficient ($K_{O/W}$) is used because plenty of scientific literature is available and numerous estimation procedures including software tools exist.
Eddo Hoekstra outlined the chapter on the analytical determination of migrants (Annex 8). There are no analytical methods in the legislation and there are few CEN methods available since most of them refer still to the old legislation. NRLs have validated some of their methods via the inter-laboratory comparison exercises in the framework of the EURL-NRL network. Furthermore, there are about 400 non-validated methods available from the substance applications to EFSA. There are few CEN methods available for the determination of a migrant in the plastic. Other relevant methods available are on the determination of the surface area of food contact materials and on the identification of plastics. There are also guidelines for the performance criteria and validation procedures of analytical methods. The work also comprised the rewriting of the methods for the determination of the overall migration to vegetable oil and aqueous food simulants and organic solvents.
Juana Bustos presented the way the migration test result should be reported (Annex 9). This includes the correction of the test result to the real surface-to-volume ratio, the correction by the food simulant D2 reduction factor compensating for the higher extraction power of food simulant D2 in comparison with certain fatty foods, the fat reduction factor for lipophilic substances compensating for the fact that the ingestion of fat is 200 g per day for an adult instead of 1 kg and the combination of them. Furthermore the choice of units is addressed and how migration results of caps and other items that have a very small surface-to-volume ratio need to be reported. Finally the minimum content of a test report is presented and how the results need to be interpreted for assessment of compliance.
After each presentation there was ample room for questions (Annex 10).
The customer satisfaction survey evaluated the events contents and the organisation and logistics (Annex 11). Out of 55 participants 36 responded to the survey. The results show that 89-100% of the participants rated the workshop of good quality of which 50-85% of the participants found the workshop very good. Industry noted that it is positive that they could participate in the workshop.

Annex 1

Workshop
Technical Guidelines on Migration Testing under Regulation EU No 10/2011

8TH OCTOBER 2014
ISPRA, ITALY

DRAFT AGENDA

09.00	Welcome
09.05	Introduction
09.15	Sampling
09:45	Discussion
10:00	Materials in contact with food and migration testing with food
10.30	Discussion
10.45	Coffee break
11.00	Migration testing with food simulants and residual content
12.30	Discussion
12.45	Lunch
14.15	Screening
15.45	Discussion
16.00	Coffee break
16.15	Analytical methods
16.45	Discussion
17.00	Reporting results
17.30	Discussion
18.00	End of the day

Annex 2 Introduction

Technical guidelines for migration testing

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

Workshop on Migration Testing, Ispra 8 October 2014

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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***

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Development of guidelines migration testing

Formation of a task force representing stakeholders

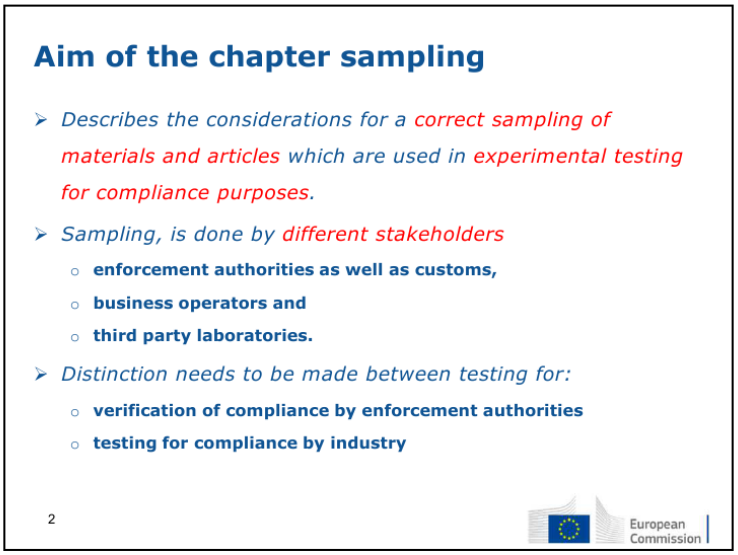
- *Industry/professional associations*
  - **CEFIC - PlasticsEurope - Flexipack Europe – EUPC**
- *Enforcement*
  - **3 NRLs – DE, ES, UK**
- *Individual experts*
  - **selected on expertise in migration testing**

- *Started end 2012 – 9 meetings*

Document structure and contents

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

Annex 3 Sampling



Common provisions for sampling

- *Sampling strategy*
  - **Should enable an appropriate and representative selection of samples that will be taken (e.g. the type, amount, size etc).**
- *Precautions should be taken to avoid any changes of the samples, which would affect:*
  - **the chemical composition of the material or article (residual content of a migrant, polymer structure).**
  - **the physical constitution, e.g. density.**
  - **the representativeness of the sample, e.g. scratches on the surface**
  - **the composition of food for already packed samples, e.g. microbiology, sensory properties and humidity**
  - **the organoleptic characteristics of the sample.**

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Common provisions for sampling

- *Labelling of samples*
  - **Relevant information permitting the sample to be identified unambiguously (sample ID etc.), should be marked on the sample or its packaging**
  - **Note: that this labelling of the sample shall not affect the migration testing.**
- *Packaging and transmission of samples*
  - **Wrap the sample in plain aluminium foil to prevent interaction with its surroundings during transport.**
  - **Or, it should be placed in a clean, inert container offering adequate protection from contamination and against damage during transport.**
  - **Precautions should be taken to avoid any change in or damage to the sample, which might arise during transportation or storage.**

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Sampling in the context of official controls - Scope

- *In the scope of Reg. (EU) No 10/2011 and in line with the related Union Guidelines (SANCO, 2014).*
- *Sampling for verification of compliance in the context of official controls shall follow Reg. (EC) No 882/2004 on official controls on feed and food.*
- *Samples obtained shall be considered as representative of the batch from which they are taken.*
- *Compliance laid down in Reg. (EC) No 10/2011 in articles, materials and foodstuffs shall be assessed on the basis of the levels determined in the laboratory samples.*

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Sampling in the context of official controls - Scope

- *Sampling can be performed*
  - **in all stages of the supply chain of food contact material**
  - **in the food industry**
  - **at the point of entry in the EU**
  - **at the point of distribution**
  - **at retailers.**
- *Member State's legal rules on sampling shall be respected.*

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Sampling in the context of official controls - Provisions

- *Personnel*
  - **Sampling should be performed by an authorised and/or instructed person.**
- *Material or article to be sampled*
  - **Each batch which is to be examined should be sampled separately. Large batches can be subdivided into sub-batches which can then be sampled separately.**
  - **The sample should always represent the worst case situation:**
  - For an already packed food commodity this means e.g. the worst place (highest expected storage temperature) or closed to the best before date.

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Sampling in the context of official controls - Provisions

- *Material or article to be sampled*
  - **The sample should always represent the worst case situation:**
  - If the sample is intended to represent a range of materials of different brands or grades, then it should be assured that material is selected that will represent the worst case situation in the migration testing (e.g. the highest concentration of additive).
  - If the substance is used in different kinds of polymers then, in principle, each type of polymer should be tested. However if it is properly argued only migration tests with the polymer representing worst case can be acceptable
- *When samples are taken from the manufacturer relevant DoC and SD shall be available on request.*

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Sampling in the context of official controls - Sampling protocol

- *Sealing of samples*
  - **Each sample taken for compliance analysis shall be sealed at the place of sampling and identified following the rules of the Member State.**
- *Sampling protocol*

  A detailed record shall be kept of each sample taken. As a minimum the following details should be recorded for each sample:
  - **Date and time of sampling**
  - **Place of sampling (i.e. full address of facility)**
  - **Spot of sampling (e.g. detailed description of the stage in the production batch)**
  - **Type of sample (e.g. material, article)**

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Sampling in the context of official controls - Sampling protocol

- **Labelling information according to Regulation (EC) No 1935/2004**
- **Number of samples taken**
- **Amount and/or size of each sample**
- **Sample identification: detailed description of sample (e.g. material type(s))**
- **Sample storage conditions from production up to and including the point of sampling (indicate whether or not lag-time or set-off could have occurred)**
- **Reason for sampling**
- **Name and signature of the responsible person and sampler**

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Sampling in the context of official controls - Sampling protocol

- *For each sampling of a batch, an appropriate sampling protocol form shall be prepared.*
- *This sampling protocol shall be issued to the relevant stakeholders according to national procedures. Examples of stakeholders are*
  - **the inspector,**
  - **the enforcement laboratory,**
  - **the business operator on sampling location**
  - **the producer of the corresponding FCM or article.**
- *The sampling protocol shall be forwarded to the business operator in order to be included into the supporting documents according to article 16 of the Regulation.*

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Sampling in the context of official controls - Quantity of material to be sampled

- *Test samples are taken for*
  - **enforcement (primary analysis),**
  - **dispute (in case of dispute the analysis should be repeated) and**
  - **reference (in case of lack of agreement after the analysis of the enforcement) purposes, unless such a procedure conflicts with Member States' rules for sampling and rights of the business operator.**

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Sampling in the context of official controls - Quantity of material to be sampled

- *FCM could be heterogeneously distributed, care must be taken to always have a representative quantity of test samples.*
- *For articles and sets of articles the recommended minimum amount of test samples per substance(group)/OM is 5, based on one combination of food/food simulant and time-temperature:*
  - **1 test sample for the identification of the polymer type**
  - **1 test sample for the surface area calculation**
  - **3 test samples for the migration test**

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Sampling in the context of official controls - Quantity of material to be sampled

- *This amount needs to be multiplied by 3 to obtain also the test samples necessary for dispute and reference. So in total a minimum of 15 test samples are necessary.*
- *Lids and caps form an exception; a total of 60 (3 x 20) has to be sampled for one test condition covering enforcement, dispute and reference.*
- *For materials such as foils, wraps, etc the recommended minimum amount of test sample for enforcement depends on the area necessary for sample identification and migration test in triplicate.*
- *Note: discarding the first layer of the bobbin of a foil/film may be necessary if changes or reactions of the foil or film occur in order to get a representative sample.*

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Sampling in the context of official controls - Quantity of material to be sampled

- *At the retail stage sampling of FCM/A and already packed foodstuffs and kitchen and tableware shall be done where possible in accordance with the above sampling provisions.*
- *Where this is not possible, other effective sampling procedures can be used provided that they ensure sufficient representativeness.*
- *Sampling of FCM/A as parts/components of industry production plants (e.g. flat conveyor; tubes, sealing) shall be done where possible in accordance with the above sampling provisions.*
  - **If needed, the corresponding food for testing shall be sampled in such a way as to guarantee both their legal and analytical validity.**

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Sampling for compliance testing by industry - Introduction

- *Considerations for sampling plastic materials and articles for screening of compliance with OML and SML.*
- *Ultimately sampling only has purpose if it is linked to testing, therefore the considerations made relate more to the reasons for testing (or for not testing) than the actual sampling.*
- *Depending on the purpose of the testing, a specific testing and sampling strategy can be devised, in which the testing that has been done previously can also be taken into account.*
- *The business operator should document his considerations on sampling and testing in his supporting documentation.*

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Sampling for compliance testing by industry - Basic aspects of sampling

- *To the extent that FCM/A are produced in accordance with the requirements on GMP (Reg. 2023/2006) and have a consistency in their properties and composition, any sample taken can be representative for any batch of that product irrespective of the number of production runs, until such time as a relevant change in product composition or its manufacturing parameters gives cause to re-examining its migration behaviour.*
- *If consistency is achieved and documented then re-testing can be done at lower frequency.*

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Sampling for compliance testing by industry - Basic aspects of sampling

- *To determine the appropriate timing for sampling, the business operator should take into account the presence of any material in his product that has not yet reached its definitive physical or chemical state immediately after production.*
  - **For example, inks may need to dry, two component adhesives are subject to a chemical curing process, or plastics can re-crystallise after extrusion. These processes should be allowed to come to equilibrium before taking the sample.**
- *The critical time for a final packaging material is when the article leaves the FCM producing company when it is for sale and can be used in contact with food.*
  - **This sets the lower limit for timing when to sample an FCM.**

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Sampling for compliance testing by industry - Basic aspects of sampling

- *The upper limit is set by the maximum age of the FCM at which it is still suitable for use. In between, consideration needs to be given to the following aspects (in particular for SML testing):*
  - **set-off can affect the amount of substance present on the food contact side of the FCM that has to be tested;**
  - **equilibration between the layers of a multilayer FCM can be addressed for example by**
    - waiting the material to come to equilibrium
  - **The previous two points would not be relevant for just-in-time deliveries where the user of the FCM does not store the FCM for any length of time.**

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Sampling for compliance testing by industry - Basic aspects of sampling

- *Notwithstanding any pre-conditioning, the sample should remain part of the product for as long as is practical.*
  - **For example if a normal production run involves producing stacks of cups that are kept in storage as stacks for a number of months.**
- *The physical place where the sample is taken out of the material produced, can be important in certain cases.*
  - **For example when producing a material that is wound on a reel, and sampling the outer winding of the reel to test for migration of a volatile substance, it can be expected that the substance has escaped**
- *Any similar aspects (e.g. set-off) should be given due consideration when deciding where and how to sample.*

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Sampling for compliance testing by industry - Basic aspects of sampling

- *In case of sampling plastic intermediates (granules etc.) it is best to transform the sampled intermediate material into a test specimen applying the appropriate processing conditions for the material.*
  - **The test specimen is typically a film or sheet or other article with defined thickness and shape.**
  - **Alternatively, for migration modelling or calculation of total transfer it is possible to determine a substance concentration directly in the sampled plastic intermediate.**
- *At the time of sampling, a record should be kept of the relevant points allowing to unambiguously link the sample taken to the production run of the product and the raw materials used.*
  - **And any other parameter considered relevant for the test and the interpretation of the results.**

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Sampling for compliance testing by industry - Family approach

- *If there is a need, the business operator can attempt to reduce the number of samples to be tested to a manageable number.*
- *The business operator manufacturing FCM/A will look for similarities in their composition and structure to justify selecting one or more individual products out of a larger group, the "product family", as the representative samples for that group.*
- *This justification for the decision to put products in one family should be part of the supporting documents.*
- *These considerations therefore are part of the manufacturer's supporting documentation.*
  - **The precise details of these considerations are impossible to describe in full detail in the guideline document.**

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Sampling for compliance testing by industry - Testing frequency

- *Compliance testing is part of the SD (Reg. Art. 16.2) needed to justify the information given in the DoC (Reg. Art. 15; Annex IV).*
- *Article 15.3 relates the interval at which the DoC needs to be renewed to substantial changes in the product's composition or production which change its migration characteristics.*
- *The test frequency shall be based on GMP and thus on the knowledge of the producer concerning the relation of the manufacturing parameters and the test results.*

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Sampling for compliance testing by industry - Testing frequency

- *The business operator should consider the statistical significance of a test result obtained on any given product, and how that affects his testing frequency.*
- *An overview of the historical track record of test results on that product or its product family will show whether or not there is sufficient consistency to conclude on sustained compliance.*
- *On the other hand a single isolated test implies greater uncertainty.*
  - **If the test result is not lower than the migration limit minus the analytical uncertainty additional tests for confirmation of compliance are needed.**

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Sampling for compliance testing by industry - Testing frequency

- *There are considerations that can be made on change control that are relevant to testing, e.g.:*
  - **Changes in the manufacturing process can be assessed by investigating the relevant properties of the material, or by investigating its migration properties, or both.**
  - **For SML compliance any change in composition that introduces a new substance with SML or substantially changes the amount present, would give rise to a new compliance assessment.**
  - **When following a "family approach" in establishing compliance for a group of products, any new or reformulated product may already fit within an existing product family definition and would then not require additional testing.**
- *Note: The testing may need to be repeated when the legal provisions on compliance testing change.*

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Sampling for compliance testing at/by industry - Alternatives to testing

- *For OML, there is no alternative to testing, but the provisions on family approach and on testing frequency fully apply.*
- *It needs to be noted that the Regulation does not require OML testing in every stage of the supply chain, nor does it require that only the finished material or article can be tested.*
- *The manufacturer of the final FCM/A has the legal obligation to confirm compliance with the OML.*
  - **This may be based on testing done by an upstream supplier if the manufacturer of the finished material or article can justify (in SD!) that there is no substantial difference in the migration characteristics of his finished material or article compared to the semi-finished material received from his supplier.**

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Sampling for compliance testing at/by industry - Alternatives to testing

- *For SML, there are alternatives to testing provided in the Regulation.*
- *There are certain business practices related to the exchange of information relevant for compliance with SML.*
  - **It is possible for a business operator to assess the compliance of an SML without testing – if his supplier has e.g. disclosed the concentration of the substance,**
  - **or has confirmed compliance on relevant samples,**
  - **or for certain use conditions or for certain layer thicknesses or blend concentrations, etc.**
  - **In these cases the only thing that remains to be done by the business operator receiving this information is to make sure that his use of the product received as well as the end uses in contact with food, are covered by the conditions described by his supplier.**
  - **Nevertheless self-monitoring of the business operator is also part of GMP.**

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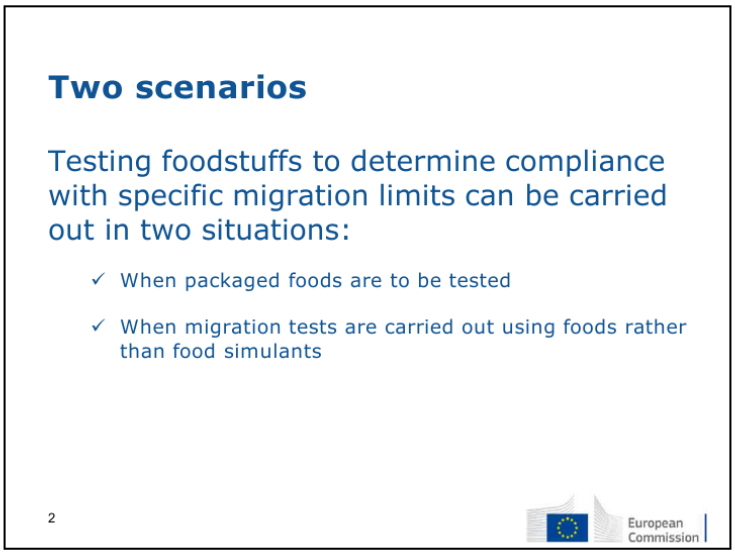
Annex 4 Materials in contact with food and migration testing with food



1

Packaged foodstuffs

If the food is already in contact with the material/article, determining the concentration of the substance in the foodstuff is the only way to assess compliance and non-compliance with specific migration limits

3

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Regulation (EU) No 10/2011

1.1 Sample preparation states:

- ✓ "The material or article shall be stored as indicated on the packaging label or under conditions adequate for the packaged food if no instructions are given. The food shall be removed from contact with the material or article before its expiration date or any date by which the manufacturer has indicated the product should be used for reasons of quality or safety."

4

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Regulation (EU) No 10/2011

1.2 Conditions of testing

- ✓ "The food shall be treated in accordance with the cooking instructions on the package if the food is to be cooked in the package. Parts of the food which are not intended to be eaten shall be removed and discarded. The remainder shall be homogenised and analysed for migration. The analytical results shall always be expressed on the basis of the food mass that is intended to be eaten, in contact with the food contact material."

5

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Sample preparation

- ✓ As detailed on-pack (e.g. heating in-pack)
  - ✓ More than one heating method defined?
  - ✓ Prepare following all instructions given
  - ✓ Use worst case (highest temperature at the interface – EN14233)
  - ✓ Accelerated conditions not recommended

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Sample preparation

- ✓ Remove non-edible parts
  - ✓ Bones
  - ✓ Liquid media
- ✓ Remove from the packaging as a consumer would do in the worst case (e.g. scraping food residues)

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Sample preparation

- ✓ Reconstitution/dilution of foods before analysis?
  - ✓ Powders, concentrated juices
  - ✓ Where does this stop?
    - ✓ Cooking food prior to analysis – e.g. dry pasta
    - ✓ Exposure versus migration
  - ✓ Can we resolve this in technical guidelines?
    - ✓ Substances that should be ND?
    - ✓ Foods intended for babies and infants?
  - ✓ Better resolved in the Regulation?

8

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Text in guidelines

- ✓ Examples
  - ✓ dilution with water
  - ✓ use as an ingredient
  - ✓ boiling in water (not in the packaging)
- ✓ "… concentration in the food should be determined on removal from the packaging and without further preparation"

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Text in guidelines

- ✓ The mass of the food used to determine the concentration is the mass of the packaged foodstuff
- ✓ Exceptions:
  - ✓ Foods which are reconstituted or diluted before being eaten
    - ✓ e.g. concentrated juices, milk powder, soup powder
- ✓ The mass of the food used to determine the concentration is the mass of the reconstituted or diluted food – following on-pack instructions

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Text in guidelines

- ✓ Regarding substances with a SML of "non-detectable" (ND), their specific migration should be ND in the concentrated form of the food, i.e. without application of a correction to the mass of the reconstituted food
  - ✓ Irrespective of the foodstuff

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Packaged foods

- ✓ Analysis of food before expiry date:
- ✓ Concentration in the foodstuff > SML
- ✓ Sample is non-compliant

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Packaged foods

- ✓ Analysis of food before expiry date:
- ✓ Concentration in the foodstuff < SML
- ✓ Can the sample be classed as compliant?
  - ✓ How long before the expiry date was the sample tested?
  - ✓ What was the polymer?
  - ✓ What were the contact conditions?
  - ✓ Equilibrium conditions?
  - ✓ Case-by-case basis

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Packaged foods

- ✓ Analysis of food before expiry date:
- ✓ Concentration in the foodstuff approaching the SML
  - ✓ Follow up with the manufacturer
  - ✓ Consideration of supporting documentation
  - ✓ Can migration modelling confirm the SML will not be exceeded?
  - ✓ Re-test on the expiry date

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Packaged foods - caution

- ✓ Concentration of a substance in the packaged foodstuff is the sum of all forms of contamination by this substance
  - ✓ Packaging used for the ingredients
  - ✓ Processing and preparation equipment
  - ✓ Final food packaging material
  - ✓ Ubiquitous in the environment
    - ✓ e.g. phthalates
    - ✓ is the substance present in the packaging?

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QUESTIONS ON SECTION 3.1 – CONSULTATION EXERCISE

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Packaged foods

How close does the migration value have to be to the SML to be considered to be approaching the value?

- ✓ *Analysis of food before expiry date:*
- ✓ *Concentration in the foodstuff approaching the SML*
  - ✓ Follow up with the manufacturer
  - ✓ Consideration of supporting documentation
  - ✓ Can migration modelling confirm the SML will not be exceeded?
  - ✓ Re-test on the expiry date

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Text in guidelines

- ✓ The mass of the food used to determine the concentration is the mass of the packaged foodstuff
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- ✓ The mass of the food used to determine the concentration is the mass of the reconstituted or diluted food – following on-pack instructions

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Disagreement with this statement - "The SML shall be verified in the food as packed in principle."

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Packaged foods - caution

- ✓ Concentration of a substance in the packaged foodstuff is the sum of all forms of contamination by this substance
  - ✓ Ubiquitous in the environment
    - ✓ e.g. phthalates
    - ✓ is the substance present in the packaging?

If phthalates are detected in the foodstuff and the DoC or analysis shows that phthalates are present in the packaging.

- Can we assume all of phthalates in the foodstuff result from migration from the final food packaging?

No. The supplier should be responsible to find the cause of the phthalates in the food

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DISCUSSION

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Materials and articles not yet in contact with foods

- ✓ Using foods in migration tests
- ✓ Regulation (EU) No 10/2011 Annex V, Chapter 2
  - ✓ Verification of compliance with a specific migration limit for a material or article can be demonstrated using food in a migration test rather than a food simulant

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When to test with food(s)?

- ✓ Where the foodstuff is water
- ✓ Where the representativeness of the food simulant is in doubt
- ✓ When a migration test into a food simulant fails, e.g. unacceptable quality assurance
- ✓ When testing with a food simulant is more analytically challenging than testing with the foodstuff itself

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When to test with food(s)?

- ✓ Where the material/article is intended to come into contact with a single and well defined foodstuff or a given food type for which a representative worst case foodstuff can be selected

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Regulation (EU) No 10/2011

Clause 2.1 of Annex V states:

- ✓ "Verification of compliance of migration into foods with the migration limits shall be carried out under the most extreme conditions of time and temperature foreseeable in actual use taking into account paragraphs 1.4, 2.1.1, 2.1.6 and 2.1.7."

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Exposure conditions

- ✓ *Representative material or article*
  - ✓ Only parts intended for contact with foods shall be used
- ✓ Expose to the foodstuff in the form it would come into contact with the material or article (e.g. don't reconstitute/dilute prior to the exposure)
- ✓ Actual or more severe contact times and temperatures
  - ✓ Should not alter the food properties and contact

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Exposure conditions

- ✓ *Materials/articles subjected to >1 combination of t/T*
  - ✓ Exposures on the same foods
- ✓ *Repeat use articles*
  - ✓ 3 successive exposures
  - ✓ Migration should not increase
  - ✓ The concentration derived from the third exposure should be compared to the migration limit
    - ✓ Except for ND substances

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Sample preparation

- ✓ Remove non-edible parts
- ✓ Remove all food residues from the material or article as a consumer would do in the worst case (e.g. scraping food residues)
- ✓ Test a portion of the food that has not been exposed to the material or article to confirm any substances present occur due to migration from the tested material or article
  - ✓ e.g. phthalates

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Using foods in migration tests

Article 18.6 of Regulation (EU) No 10/2011 states:

"The results of specific migration testing obtained in food shall prevail over the results obtained in food simulant and by screening tests."

- ✓ Migration results obtained are applicable to the specific foodstuff investigated and any other foods of the same type for which the food used in the migration test can be considered the worst case

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QUESTIONS ON SECTION 3.2 – CONSULTATION EXERCISE

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When to test with food(s)?

- ✓ Where the foodstuff is water → Clarify= Mineral or flavoured water
- ✓ Where the representativeness of the food simulant is in doubt
- ✓ When a migration test into a food simulant fails, e.g. unacceptable quality assurance
- ✓ When testing with a food simulant is more analytically challenging than testing with the foodstuff itself

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Exposure conditions

- ✓ *Materials/articles subjected to >1 combination of t/T*
  - ✓ Exposures on the same foods
- ✓ *Repeat use articles*
  - ✓ 3 successive exposures
  - ✓ Migration should not increase
  - ✓ The concentration derived from the third exposure should be compared to the migration limit
    - ✓ Except for ND substances

What about PAAs? Legislation to be amended

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DISCUSSION

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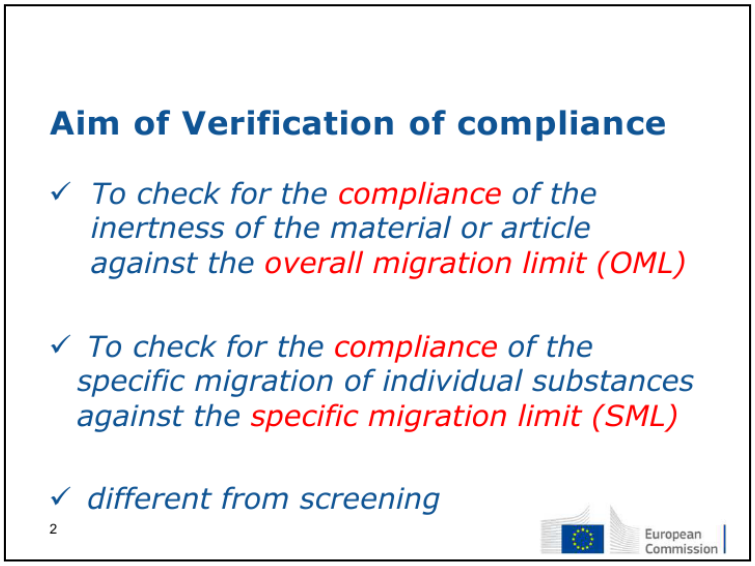
Annex 5 verification of compliance using food simulants



1

Scope of the chapter

guidance on selection of test conditions for most severe contact conditions of the material with food under foreseeable conditions of use

- *contact time*
- *contact temperature*
- *food simulant*
- *contact area*

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Food simulants

Food simulant	***Abbreviation***
Ethanol 10 % (v/v) (old: water)	*Food simulant A*
Acetic acid 3 % (w/v)	*Food simulant B*
Ethanol 20 % (v/v) (old: Ethanol 10 % (v/v)	*Food simulant C*
Ethanol 50 % (v/v)	*Food simulant D1*
Vegetable oil (old: olive oil)	*Food simulant D2*
poly(2,6-diphenyl-p-phenylene oxide), particle size 60-80 mesh, pore size 200 nm	*Food simulant E (TENAX, PPO or MPPO)*

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Food simulant E

Simulant for specific migration into dry food

chemical name *: Poly(2,6-diphenyl-p-phenylene oxide)*

trade name: *Tenax® TA or MPPO (modified polyphenylene oxide).*
Note that MPPO also refers to a blend of polyphenylene oxide and polystyrene.

issues with overall migration

- *incomplete extraction*
- *loss of migrants during evaporation*

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Food simulant E for OM

- *foods, for which only simulant E is prescribed by the Regulation, are not subject to OML testing*
  (ANNEX III, Table 2: dried or dehydrated vegetables whole, sliced or in the form of flour or powder)
- *if OM 7 (2h at 175°C, fatty food) is not feasible OM 8 or OM 9 should be applied*
- *OM 8 and OM 9 consists of two separate tests, 2 h at 175°C with simulant E (OM8 and OM9) and 2 h at 100°C (OM8) or 10 d at 40°C (OM9) with simulant D2. The analytical results of simulant E and D2 shall both comply with the OML.*

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Food simulant D2 - Specification

General

- *vegetable oil (e.g. olive oil, sun flower oil, corn oil)*
- *simulant for specific and overall migration into fatty food*

Specific migration

- *interferences shall not exceed a level 10% of the SML*

Overall migration

- *less than 1% of unsaponifiable matter (waxes and essential oils) to avoid wrong results*
- *interference of substances that can present in the polymer (e.g. GMS)*

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Situations where use of food simulant D2 is not feasible

Exceptions listed in Regulation

- ✓ *for food type 01.04 (undenaturated ethyl alcohol) use 95% ethanol*
- ✓ *Annex I Table 1 Column 11*

Exceptions due to other reasons

- ✓ *Reaction with the migrant*
- ✓ *great similarity in chemical and physical properties does not allow separation*

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Use of food simulant D2 is not feasible

Specific Migration

- ✓ *reaction of the substance with the simulant (e.g. primary amines react with oil)*
- ✓ *isolation of the substance from the oil is not possible due to physical or chemical properties (e.g. dimerised fatty acids, polymeric substances with SML of 0.05 mg/kg food, waxes (FCM 93) etc.)*
- ✓ *unavoidable interferences from the food simulant D2*
- ✓ *insufficient analytical detection limit of the substance in vegetable oil*

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Use of food simulant D2 is not feasible

Overall Migration

- ✓ *excessive absorption of oil (e.g. expanded polymers)*
- ✓ *difficulties to recover the absorbed oil with any of the known methods (see Annex EN 1186-part 10)*
- ✓ *presence of interfering substances in the recovery and determination of the absorbed oil*
- ✓ *difficulties 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)*
- ✓ *substitute test OM 8 and/or OM 9 are not relevant according to the selected test conditions*

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Use of food simulant D2 is not feasible

Alternative food simulants

- ***iso-octane, 95% ethanol and food simulant E***
- *maximum temperature applied to iso-octane and 95% ethanol is restricted to 60°C (safety reasons)*
- *temperatur above 100°C: no physical change (prove with vegetable oil)*
- *different time and temperature conditions*

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Use of food simulant D2 is not feasible

Conventional test conditions for polyolefines (only containing carbon and hydrogen) (LDPE, LLDPE, HDPE, PP (homo, random, rubbery), PS, SBS

Examples are given in Table 1 (Guidlines , Chapter 4)

simulant D2	ethanol 95%	iso-octane	simulant E
10 d at 5°C	same t/T conditions as for simulant D2	0.5 d at 5°C	no
10 d at 20°C	same t/T conditions as for simulant D2	1 d at 20°C	no
10 d at 40°C	same t/T conditions as for simulant D2	2 d at 20°C	no

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Use of food simulant D2 is not feasible

Conventional test conditions for non-polyolefines (containing also other atoms than carbon and hydrogen) (PET, PBT, PEN, PA6, PA66, PA12, PVC (rigid), PC, PMMA)

Examples are given in Table 2 (Guidlines , Chapter 4)

simulant D2	***ethanol 95%***	***iso-octane***	***simulant E***
10 d at 5°C	*0.5 d at 5°C*	*same t/T conditions as for simulant D2*	*no*
10 d at 20°C	*1 d at 20°C*	*same t/T conditions as for simulant D2*	*no*
10 d at 40°C	*2 d at 20°C*	*same t/T conditions as for simulant D2*	*no*

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DISCUSSION

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Selection of food simulants
(general rule given in Annex III)

Food simuant	food
Food simulant A	foods that have a hydrophilic character and are able to extract hydrophilic substances pH >4.5 (aqueous food)
Food simulant B	foods that have a hydrophilic character and are able to extract hydrophilic substances; pH < 4.5 (acidic food)
Food simulant C	alcoholic foods with an alcohol content of up to 20 % those foods which contain a relevant amount of organic ingredients that render the food more lipophilic
Food simulant D1	foods that have a lipophilic character and are able to extract lipophilic substances. Alcoholic foods with an alcohol content of above 20 % and oil in water emulsions (milk products)
Food simulant D2	foods that have a lipophilic character and are able to extract lipophilic substances. Foods which contain free fats at the surface (fatty food)
Food simulant E	testing specific migration into dry foods

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Selection of food simulants
(specific assignments given in Annex III table 2)

Specific migration

- ✓ if the specific food is not listed, select the closest food based on chemical-physical properties.
- ✓ for compliance for "all types of food" in general, select food simulants A, B and D2 (see section 2.1.2 of Annex V of the Regulation).
- ✓ for substances that do not react with acidic food simulant or with acidic foods, select food simulants A and D2 (see section 2.1.2 of Annex V of the Regulation).
- ✓ in specific cases testing may be reduced to a single food simulant , which is most severe for that particular substance and/or material (based scientific arguments)

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Selection of food simulants
(specific assignments given in Annex III table 2)

Overall migration

- ✓ for specific foods within a food category, select the simulant(s) indicated in Table 2 of Annex III
- ✓ non-specific foods, i.e. "aqueous", "acidic", "alcoholic" or "fatty" food types, select the food simulants according to section 4 of Annex III of the Regulation.
- ✓ for compliance for all types of food in general, select food simulants A, B and D2
- ✓ OM testing is not required for "dry" foods.
- ✓ in specific cases testing may be reduced to a single food simulant , which is the most severe for that particular material (based scientific arguments)

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Selection of Time and Temperature

General rules

- ✓ Separated conditions for overall migration (inertness) and specific migration (safety)
- ✓ the tests shall reflect the worst foreseeable conditions of use. If the result has to be expressed in mg/kg food, the highest foreseeable surface-to-volume needs to comply.
- ✓ Only if the conventional test conditions cause physical or other changes in the test specimen that do not occur under conditions of use, then worst case foreseeable real use test conditions may be applied that do not cause the changes in the test sample.

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Selection of Time and Temperature for Specific Migration

- *conditions are specified in Tables 1 and 2 of Annex V*
- *use of **Arrhenius equation** to shorten test time*

Temprature T1 (°C)	Temperature T2 (°C)	Acceleration Factor
5	20	7
5	40	67
5	60	503
20	40	10
20	60	73
40	60	7

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Selection of Time and Temperature for Specific Migration

- ✓ *the increased contact temperature in the test compared to the worst case contact temperature should not cause any physical changes such as phase transition*
- ✓ *for storage at room temperature testing time can be reduced to 10 days at 40 °C if there is scientific evidence that migration of the respective substance in the polymer has reached equilibration under this test condition*

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Special conditions for contact times above 30 days at room temperature and below (Specific Migration)

10 days at 20°C

- ✓ *any time at frozen condition*
- ✓ *Food packaged when frozen and defrosted outside the packaging*

10 days at 40°C

- ✓ *any time at refrigerated or frozen conditions including heating for maximum 2 h at 70°C and heating up to 100°C for maximum 15 minutes*
- ✓ *any time at room temperature provided it can be demonstrated that migration of a substance is at equilibrium after 10 days at 40°C.*

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Special conditions for contact times above 30 days at room temperature and below (Specific Migration)

10 days at 50°C

- ✓ *any time at refrigerated or frozen conditions including heating for maximum 2 h at 70°C and/ or heating up to 100°C for maximum 15 minutes*
- ✓ *storage times up to 6 months at room temperature*

10 days at 60°C

- ✓ *long term storage above 6 months at room temperature, including heating for maximum 2 h at 70°C and/or heating up to 100°C for maximum 15 minutes*

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Selection of Time and Temperature for Specific Migration

Example

Food is packed and stored for 2.5 months at ambient temperature

Test conditions

selected from Table 1 and section 2.1.4 of Annex V of Regulation (EU) No 10/2011

10 days at 50°C

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Selection of Time and Temperature for Specific Migration

Example

A cup is filled with hot soup (± 90°C). The temperature of the soup will decrease within 15 minutes to a temperature of ± 60°C.

Test conditions

Selected from Table 1 and 2 of Annex V of Regulation (EU) No 10/2011

0.5 h at 100°C

Remarks

- ✓ *material is not resistant to temperatures of 100°C but can be used for hot fill applications (initial temperature is 100°C and cools down in less than 15 minutes to a temperature of 70°C or below)*

2 h at 70°C

- ✓ *The actual use should be clear from the labelling of the material.*
- ✓ *material or article is intended to be used also for storage at room temperature or below*

10 days at a temperature depending on the storage period

(see special conditions)

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Selection of Time and Temperature for Specific Migration

Example

Ovenable packaging is filled with food and heated in an oven at 200°C for 25 minutes.

Test conditions

- ***contact time*** ***0.5 hour***
- ***actual contact temperature*** *is related to the composition of the food , not necessarily the same as applied temperatur*

a) foods containing a significant portion of water, will not exceed a temperature of 100°C (aqueous simulants (A, B, C or D1))

0.5 h at 100°C or under reflux

b) food contains a significant amount of oil or fat, starch or sugar at the surface then the temperature at the interface food/packaging may be significantly higher.

0.5 h at 200°C using food simulant E only

(Only if it can be demonstrated that the contact test temperature is up to or equal to 175°C the migration test can be performed at 175°C.)

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Combinations of Contact Times and Temperature

General rule

A material or article can also be subject to two or more successive time-temperature conditions. In such cases the test specimen shall undergo the same sequence of time-temperature conditions using the same portion of food simulant.

Example

A food is sterilised at 130°C for 2 h. After that it is stored for a maximum of 25 days at room temperature.

Test conditions

- *food simulant **D2** : 2 h @130°C followed by 10 d @40°C*
- *food simulants **A, B, C, D1**:*
  - *2 h @130°C under pressure followed by 10 d @40°C*
  - *8 h @100°C or reflux followed by 10 d @40°C*

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Combinations of Contact Times and Temperature

Alternatives using Arrhenius Equation

More severe test conditions can be established using the Arrhenius equation for a single migration test contact time, based on the highest contact temperature

Example

A food is sterilised at 130°C for 2 h. After that it is stored for a maximum of 25 days at room temperature.

Test conditions

- *food simulant D2:2h15min @130°C (15 min recalculated from 10 d @40°C)*
- *food simulants A, B, C or D1:*
  - *2h15min @130°C under pressure*
  - *9h45min @100°C or reflux (1h45min recalculated from 10 d @40°C)*

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DISCUSSION

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Overall migration test conditions

OM1 :10 d at 20°C

All storage of foodstuffs in fridges, at either frozen or refrigerated temperature, for any time

OM2 :10 d at 40°C

- ✓ *Long-term storage at room temperature of any food;*
- ✓ *Hot filling followed by cooling in the package and long-term storage, e.g. molten cheese, soups, tomatoes etc.;*
- ✓ *De-freezing and/or re-heating of food (e.g. ready meals) in microwave oven*
- ✓ *Flash pasteurization >70°C (time less than 15 min) or pasteurisation less than 70°C up to 2 hours, followed by long term storage at room temperature.*
- ✓ *Other short-time high-temperature treatment such as shrink of films.*

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Overall migration test conditions

OM3 :2h at 70°C

- *Hot filling for immediate consumption (e.g. coffee or tea cups; take away food)*
- *serving utensils and tableware intended to be used in hot food for 2 hours or less*
- *Articles intended for repeated usage in very short contact (< 5 minute) with food at room temperature; example: slicers, cutters, mincers.*

OM4 :1h at 100°C

- *Pasteurization in the packaging (time longer than 15 min at 100°C or longer than 2 hours at 70°C)*
- *Cooking of food (e.g., cooking of ham in moulds, pre-cooked seafood, boil-in-bag ready meals etc.) up to 1 hour.*
- *Cooking in microwave oven (time >15 min) when the temperature does not exceed 100°C.*
- *Reheating longer than 15 min at 100°C*

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Overall migration test conditions

OM5 :2h at 100°C or at reflux or 1h at 121°C

- ✓ *Cooking of food (e.g., cooking of ham in moulds, pre-cooked seafood, boil-in-bag ready meals etc.)*
- ✓ *Cooking in microwave oven when the temperature can exceed 100°C;*
- ✓ *Sterilization in the packaging, e.g. heat sterilization of broths and soups*
- ✓ *cooking utensils*

OM6 :4h at 100°C or at reflux

- ✓ *Cooking of food entailing long-term storage. It represents worst case conditions for simulants A, B, C and D1 in non-polyolefins.*

OM7 :2h at 175°C or at reflux

- ✓ *High temperature oven-ability, e.g. dual-ovenable packaging for fatty foods such as bread, and home cooking trays,*
- ✓ *Microwave susceptors*

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Overall migration test conditions

OM8 and OM9 *are substitute test conditions for OM7 in case the test in vegetable oil is not technically feasible*

OM 8 *Food simulant E for 2 hours at 175 °C and food simulant D2 for 2 hours at 100 °C*

High temperature applications only

covers OM1, OM3, OM4, OM5, and OM6

OM 9 *Food simulant E for 2 hours at 175 °C and food simulant D2 for 10 days at 40 °C*

High temperature applications including long term storage at room temperature

covers OM1, OM2, OM3, OM4, OM5 and OM6

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Repeated use articles

- ✓ *three successive contact periods, using a new portion of food simulant for each exposure period*
- ✓ *if it is known that migration will not increase in the second and third test **and the migration limit is not exceeded**, the successive tests may be omitted*
- ✓ *substances assigned with a specific migration limit of ND (not detectable) or substances that are not authorised because they are used behind a functional barrier shall not be detectable already in the first migration period*

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Repeated use articles

Determination of the migration from a repeated use article does not deviate from the procedure followed for single use articles

Example : *Conveyor belt for bakery products*

- ❖ *size of 60m x 0.6 m, life time of 3 years, speed 0.36 km/h*
- ❖ *cake (Ø 8 cm, 100 g) 90°C cooled down to 20°C*
- ❖ *approximately 10,000 cakes an hour*

Test conditions

*contact time : 10 minutes (60 m/360 m/h * 60 min).*
Temperature : from 90°C to 20°C within 15 min
contact area : one cake :5 dm²/kg food (0.4²·π dm²/0.10 kg)
total amount not clear: surface to volume ratio of 6 dm²/kg.

Three successive migration experiments of 2 h at 70 °C with simulant E or D2

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Contact areas

single sided contact

- *Preferably contact with the food simulant at one side (article fill, pouch, migration cell)*
- *filling can be done with any volume as long as the surface-to-volume is known and the migration result can be recalculated to to real surface-to-volume ratio*

double sided contact

- *Total immersion*
- *Taking both sides into account for calculation depends on material , thickness , molecular weight (specific migration)*
- *Experimental prove or calculation of layer thickness by migration modeling (examples in tables)*

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Verification by residual content

applicable only if

- ✓ *substances react with the food simulants*
- ✓ *an analytical method to determine the migration is not available*
- ✓ *use of QMA values.*
- ✓ *if no QMA given, than 100 % migration is assumed (thickness/surface/volume according actual conditions of use)*

Examples

- ✓ *Isocyanates*
- ✓ *Epoxy containing compound*
- ✓ *Phthalates*

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DISCUSSION

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Ad
4. Verification

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Ad 4. Verification

- *4.4.3 Contact conditions in migration testing [better: '**Contact modes** in……']*

This section deals with the question in which cases (at which film thickness) the migration evaluation should consider only one side or both sides when testing migration in the full immersion or one-sided contact mode.

['Historically', 500 µm was taken as a general limit, which is scientifically not longer justified]

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4.4.3 Contact modes in migra' testing

*If the thickness of the sample is equal to or higher than the layer thickness given in **Table 5** the migrating amount can be related to the area of both sides of the sample tested. Otherwise the migrating amount in [mg] will be related only to the area of one sample side.*

In the case of overall migration testing the thickness recommendations for the molecular mass range 501-750 g/mol apply.

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4.4.3 Contact modes in migra' testing

***Table 5:** Layer thickness L (in µm) above which both side of the sample can be considered for calculation of migration if tested by full immersion at different contact conditions for four different molecular mass ranges*

Polymere:

- **Polyolefine**
- **Polyester**
- **PS, SBS**
- **PA's**
- **PVC**

Test conditions:

- **10d@60°C**
- **10d@40°C**
- **10d@20°C**
- **2h@100°C**

Polymer type molecular mass of migrant [g/mol]	Time/Temp	Layer thickness L in [µm] ≤500	501-750	751-1000
EPDM, PP rubbery	10 d at 60°C	none	none	5680	3040
	10 d at 40°C	none	11500	3680	1448
	10 d at 20°C	18008	3528	1280	488
	2h at 100°C	none	14000	4080	1928
HDPE	10 d at 60°C	1480	1480	1480	1480
	10 d at 40°C	11000	4000	1320	592
	10 d at 20°C	1200	1200	480	168
	2h at 100°C	none	8608	2640	1048
PP isotactic/homo PP random	10 d at 60°C	28008	6808	2080	848
	10 d at 40°C	5048	2208	680	288
	10 d at 20°C	1608	608	220	88
	2h at 100°C	11708	4328	1320	568
PET, PBT, PEN	10 d at 60°C	168	68	20	8
	10 d at 40°C	92	28	8	4
	10 d at 20°C	12	8	4	2
	2h at 100°C	108	48	12	8
PS	10 d at 60°C	828	84	88	12
	10 d at 40°C	88	48	20	8
	10 d at 20°C	28	12	8	4
	2h at 100°C	108	48	20	12
SBS	10 d at 60°C	5048	2168	1480	1008
	10 d at 40°C	none	9208	2680	1168
	10 d at 20°C	8408	3008	960	488
	2h at 100°C	none	12408	3680	1528
PA 6 (not micellen. eg. contact with D2, iso-octane, or any simulant not in direct contact, e.g. plastic multilayer)	10 d at 60°C	364	122	40	16
	10 d at 40°C	124	52	16	8
	10 d at 20°C	44	20	8	4
	2h at 100°C	176	68	24	12
PA 6,6 (not micellen. e.g. contact with D2, iso-octane, or any simulant not in direct contact, e.g. plastic multilayer)	10 d at 60°C	588	368	188	88
	10 d at 40°C	368	124	64	28
	10 d at 20°C	116	44	16	8
	2h at 100°C	488	188	56	24
PA 12 (not micellen. e.g. contact with D2, iso-octane, or any simulant not in direct contact, e.g. plastic multilayer)	10 d at 60°C	1328	508	168	68
	10 d at 40°C	528	188	56	28
	10 d at 20°C	168	68	20	12
	2h at 100°C	668	248	76	32
PVC, rigid	10 d at 60°C	828	84	88	12
	10 d at 40°C	88	48	20	8
	10 d at 20°C	28	12	8	4
	2h at 100°C	108	48	20	12

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4.4.3 Contact modes in migra' testing

***Table 5**: Layer thickness L (in µm) above which both side of the sample can be considered for calculation of migration if tested by full immersion at different contact conditions for four different molecular mass ranges*

Polymer type	time/Temp	layer thickness L in [µm] for molecular mass (g/mol) range			
molecular mass of migrant (g/mol)		100-250	251-500	501-750	751-1000
LDPE, PP rubbery	10 d at 60°C	none	none	9600	3840
	10 d at 40°C	none	12000	3680	1440
one < 500 µm	10 d at 20°C	10000	3520	1200	480
	2h at 100°C	none	16000	4880	1920
PS	10 d at 60°C	220	84	28	12
	10 d at 40°C	80	40	20	8
All < 500 µm	10 d at 20°C	28	12	8	4
	2h at 100°C	108	40	20	12

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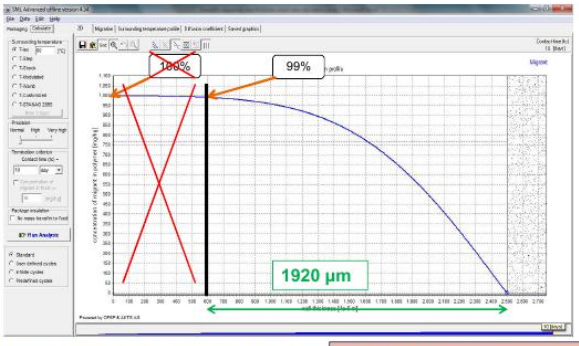
4.4.3 Contact modes in migra' testing

Case LDPE – m.w. range 750-1000 g/mol – Migrant ($C_{p,0}$ = 1000 ppm, 750g/mol) - 10 days@60°C



Layer thickness which is 'not affected': 1920 µm | *x 2 = 3840 µm => 2 sided evaluation*

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4.4.3 Contact modes in migra' testing

***Table 5**: Layer thickness L (in µm) above which both side of the sample can be considered for calculation of migration if tested by full immersion at different contact conditions for four different molecular mass ranges*

Polymer type	time/Temp	layer thickness L in [µm]			
molecular mass of migrant (g/mol)		100-250	251-500	**501-750**	751-1000
HDPE	10 d at 60°C	none	13700	**4200**	1680
	10 d at 40°C	11800	4800	**1320**	540
	10 d at 20°C	3200	1200	**400**	168
	2h at 100°C	none	8600	**2640**	1040
PS	10 d at 60°C	220	84	**28**	12
	10 d at 40°C	80	40	**20**	8
	10 d at 20°C	28	12	**8**	4
	2h at 100°C	108	40	**20**	12

Overall migration

European Commission

Ad
5. Screening

European Commission

Ad 5. Screening

- *5.2.3 Screening by residual content*

  .... is one of the four screening principles

This section deals with the approach of calculating the maximum possible migration via determination of the residual content of the migrant in the polymer.

['Historically', for non-polyolefins 250 µm was taken as a general limit for total mass transfer which leads in most cases to extreme and irrealistic 'migration values']

5.2.3 Screening by residual content

Based on migration modelling one can set conservatively the borderline thickness from which 'total mass transfer' may occur in dependency of

(i) the diffusion properties of the particular polymer
(ii) the molecular size or mass of the migrant.

Furthermore, whether or not it is exhaustive migration depends also on the time-temperature contact conditions.

=> Table 7

5.2.3 Screening by residual content

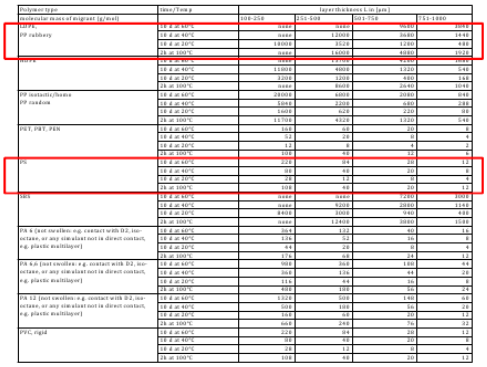
Table 7: Layer thickness L (in µm) for which total mass transfer assumption can be made at different contact conditions for four different molecular mass ranges

Polymere:

- **Polyolefine**
- **Polyester**
- **PS, SBS**
- **PA's**
- **PVC**

Test conditions:

- **10d@60°C**
- **10d@40°C**
- **10d@20°C**
- **2h@100°C**



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5.2.3 Screening by residual content

Table 7: Layer thickness L (in µm) for which total mass transfer assumption can be made at different contact conditions for four different molecular mass ranges

Polymer type	time/Temp	layer thickness L in [µm] for molecular mass (g/mol) range			
molecular mass of migrant (g/mol)		100-250	251-500	501-750	751-1000
LDPE, PP rubbery	10 d at 60°C	Full L	Full L	2400	960
	10 d at 40°C	Full L	3000	920	360
	10 d at 20°C	2500	880	300	120
	2h at 100°C	Full L	4000	1220	480
PS	10 d at 60°C	55	21	7	3
	10 d at 40°C	20	10	5	2
All < 250 µm	10 d at 20°C	7	3	2	1
	2h at 100°C	27	10	5	3

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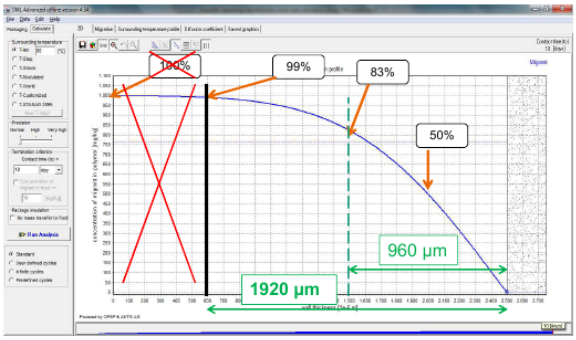
5.2.3 Screening by residual content

Case LDPE – m.w. range 750-1000 g/mol – Migrant ($C_{P,0}$ = 1000 ppm, 750g/mol) - 10 days@60°C



Layer thickness which is 'not affected': 1920 µm | *x 2 = 3840 µm => 2 sided evaluation*

Layer thickness for total mass transfer assumption: 0.5 x 99% layer = 960 µm

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5.2.7 Functional barrier considerations

…… Which materials at which thickness would exclude any permeation of potential migrants from outside into the food at any foreseeable contact condition for packed foods.
Here the following examples can be considered:

- *Glass of any thickness (not: SiO_x layers)*
- *Metal cans and lids*
- *Aluminium foils at thickness when pinholes or other damages can be excluded*
- *Plastic layers for substances in dependency of their molecular mass and in relation to defined time/temperature conditions* ***(=> Table 9)***

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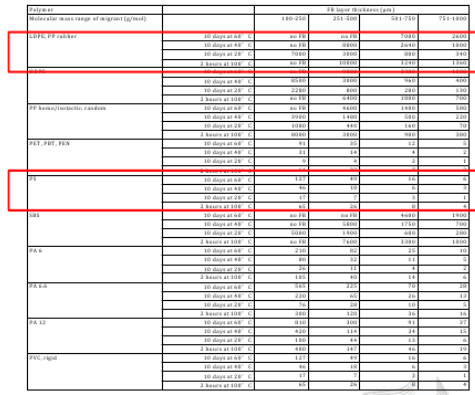
5.2.7 Functional barrier considerations

Table 9: Functional barrier layer thickness LFB (in µm) of various polymers through which no migration can be expected at different contact conditions for four different molecular mass ranges

Polymere:

- **Polyolefine**
- **Polyester**
- **PS, SBS**
- **PA's**
- **PVC**

Test conditions:
- **10d@60°C**
- **10d@40°C**
- **10d@20°C**
- **2h@100°C**



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5.2.7 Functional barrier considerations

Table 9: Functional barrier layer thickness LFB (in µm) of various polymers through which no migration can be expected at different contact conditions for four different molecular mass ranges

Polymer type	time/Temp	layer thickness L in [µm] for molecular mass (g/mol) range			
molecular mass of migrant (g/mol)		100-250	251-500	501-750	751-1000
LDPE, PP rubbery	10 d at 60°C	no FB	no FB	7000	2600
	10 d at 40°C	no FB	8800	2640	1000
	10 d at 20°C	7000	3000	800	340
	2h at 100°C	no FB	10000	3240	1360
PS	10 d at 60°C	127	49	16	6
	10 d at 40°C	46	18	6	3
	10 d at 20°C	17	7	3	1
	2h at 100°C	65	26	8	4

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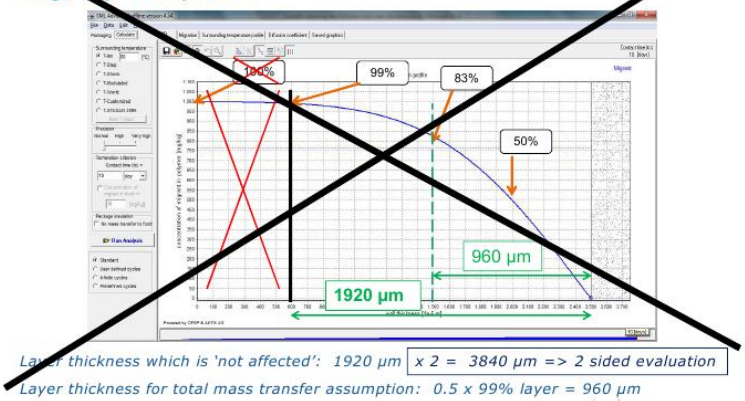
5.2.7 Functional barrier considerations

Case LDPE – m.w. range 750-1000 g/mol – Migrant ($C_{P,0}$ = 1000 ppm, 750g/mol) - 10 days@60°C



Layer thickness which is 'not affected': 1920 µm | *x 2 = 3840 µm => 2 sided evaluation*

Layer thickness for total mass transfer assumption: 0.5 x 99% layer = 960 µm

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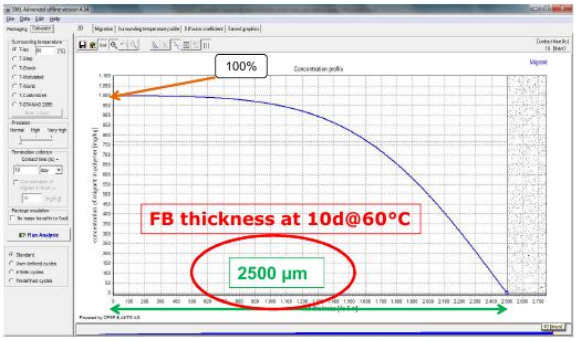
5.2.7 Functional barrier considerations

Case LDPE – m.w. range 750-1000 g/mol – Migrant ($C_{P,0}$ = 1000 ppm, 750g/mol) - 10 days@60°C



Layer thickness which acts as FB: 2500 µm
(100% of layer which is not affected)

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5.2.7 Functional barrier considerations

A higher degree of differentiation and further refinements can be achieved by migration modelling when the detailed parameters such as the exact molecular mass and concentration of the migrant in the releasing polymer layer and the structural specifications of the application are known or can reasonably be assumed. It can then be derived for a given migrant whether or not the barrier layer would be a functional barrier, i.e. would prevent migration from exceeding of the respective SML or another acceptable migration limit.

5.2.7 Functional barrier considerations

*Other materials than those mentioned in **Table 9** can act as functional barrier as long as it has been demonstrated at the worst case foreseeable conditions of use that the relevant potential migrants are not migrating above the limit of 0.01 mg/kg food.*

Besides the barrier properties achieved solely by a monolayer polymer other very thin barrier layers can be placed on usual carrier polymers to achieve excellent barrier effects by the whole composite, e.g. acrylic or PVDC coated PP films.

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5.2.7 Functional barrier considerations

Within a comprehensive research project (Fraunhofer IVV Report 'FPE Functional Barrier Project', 09.12.2011. Publication in preparation, see annex ?) the barrier properties of 24 different flexible packaging films were studied by permeation measurements across these films using

- *12 different test permeants with molecular masses between 90 and 400 g/mol*
- *at temperatures between 20°C and 80°C*
- *with core test conditions for all films of 40°C up to 47 days and 60°C up to 14 days.*

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5.2.7 FB - Fraunhofer IVV project

Overview test substances

Substance	CAS-No.	formula	m.w.	mp	bp
			(g/mol)	(°C)	(°C)
1-Methoxy-2-propanol	107-98-2	$C_4H_{10}O_2$	90,12	-97	119
1-Ethoxy-2-propanol	1569-02-4	$C_5H_{12}O_2$	104,14	-100	132
Toluene	108-88-3	C_7H_8	92,14	-95	111
Naphthalene	91-20-3	$C_{10}H_8$	128,17	80	218
Diphenyloxide	101-84-8	$C_{12}H_{10}O$	170,21	86	287
Hexadecane C16	544-76-3	$C_{16}H_{34}$	226,44	18,2	287
Benzophenone	119-61-9	$C_{13}H_{10}O$	182,22	48	305
N-Ethyl toluene sulphonamide (NETSA)	8047-99-2	$C_9H_{13}NO_2S$	199,26	66	324
Octadecane C18	593-45-3	$C_{18}H_{38}$	254,50	28,2	316
4-Methylbenzophenone	134-84-9	$C_{14}H_{12}O$	196,25	55	326
Dibutyl sebacate	109-43-3	$C_{18}H_{34}O_4$	314,47	-10	344
Acetyl tributyl citrate	77-90-7	$C_{20}H_{34}O_8$	402,47	-90	173 @ 1 hPa

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5.2.7 FB - Fraunhofer IVV project

Overview test films (24)

No.	Received from company	Sample specification	Base polymer	Barrier material
1	Company C	PE / EVOH / PE total 50 µm EVOH 5 µm	PE	EVOH 5 µm
2	Company C	36 µm CPET corona treated	PET	-
3	Company C	BOPP acrylic / PVOH 25 my (Mobil MB866)	PP	PVOH / acrylic
4	Company D	15 µm OPA (Filman BX 15)	PA	-
5	Company D	12 µm PET (PhD)	PET	-
6	Company D	12 µm PET metallized	PET	met
7	Company G	22 µm PVDC coated transparent OPP film	PP	PVDC
8	Company G	12 µm PVDC coated transparent Polyester film	PET	PVDC
9	Company G	18 µm metallised OPP film (18MM466)	PP	met
10	Company I	18µm metallised OPP-film (Taghleef ZSF)	PP	met
11	Company I	30 µm Acrylic coated OPP-film (Mobil MB 866)	PP	Acryl
12	Company A	6 µm aluminium		Aluminium
13	Company E	20 µm OPP-SiOx 60 nm (CPP002)	PP	SiOx
14	Company E	12 µm PET-SiOx 60 nm (CPT008)	PET	SiOx
15	Company E	12 µm PET-SiOx 60 nm Ormocer-Lacquer (CPT006)	PET	SiOx / Ormocer
16	Company H	60 g/m² Papier / 30 g/m² PVDC	paper	PVDC
17	Company H	12 µm metallisiertes PET	PET	met
18	Company H	12 µm PET / SiOx	PET	SiOx
19	Company B	PE / EVOH 3 µm / PE total 30 µm	PE	EVOH
20	Company M	12 µm PET / AlOx / adhesive / 30 µm PP	PP	PET-AlOx
22	Company F	75 µm PP	PP	-
23	Company G	30 µm OPP (TS5-Taghleef)	PP	-
24	Company G	50 µm LDPE LIS2-BR	PE	-
25	Company A	6 µm aluminium / PE	PE	Aluminium

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5.2.7 FB - Fraunhofer IVV project

Permeation cell

Aluminium

Total diameter:
20 cm

Testing diameter:
15.6 cm

Testing area:
1.91 dm²

Sealing rings:
Teflon or Viton

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5.2.7 FB - Fraunhofer IVV project

Permeation measurement system

Cells in Cabinet

(0 to 100 °C)

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5.2.7 FB - Fraunhofer IVV project

Permeation measurement system

Whole system

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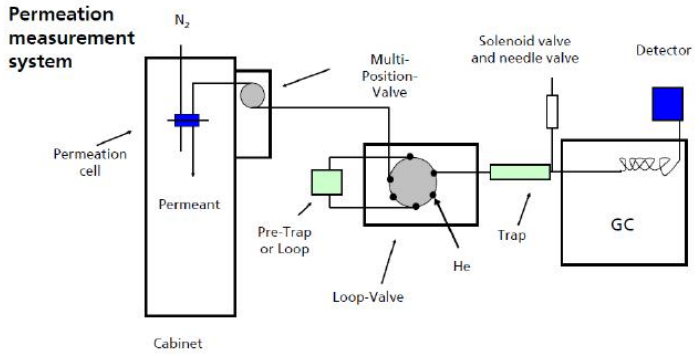
13

5.2.7 FB - Fraunhofer IVV project



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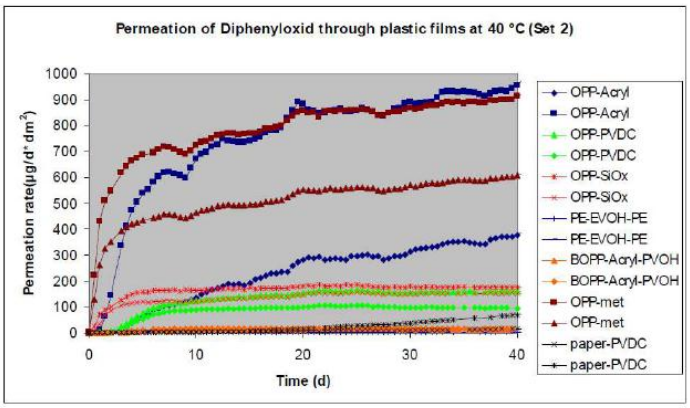
5.2.7 FB - Fraunhofer IVV project



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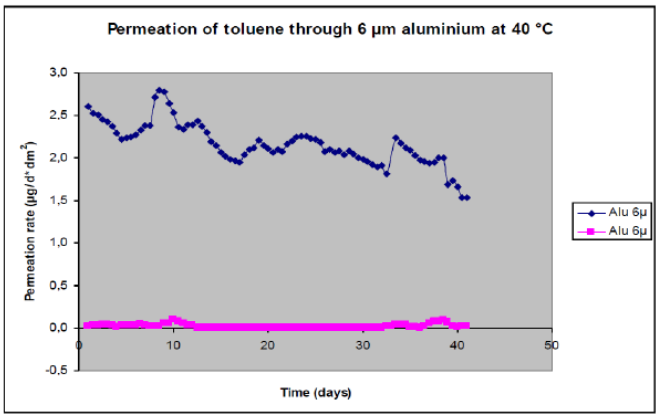
5.2.7 FB - Fraunhofer IVV project



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5.2.7 Functional barrier considerations

*Specific migration testing for potential migrants present behind the films, for instance from other outer layers such as polymers, adhesives, printing inks, coatings, paper and board or secondary packaging is not needed at all for the general functional barriers (=> **Table 10**: 10d@60°C case) unless the migration target value would be much lower than 10 ppb or an issue with set-off has been identified.*

For higher temperature (than 60°C) applications the barrier properties have to be checked and verified.

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5.2.7 Functional barrier considerations

***Table 10**: Barrier films which act as a general FB in reducing any migration down to levels below of 10 ppb at test contact conditions of 10 d @ 60°C.*

Film structure	Base polymer	Barrier material
36 µm O-PET corona treated	PET	PET
12 µm PET metallised	PET	metallisation
12 µm PET-SiOx 80 nm	PET	SiOx
12 µm PET-SiOx 50 nm Ormocer-Laquer	PET	SiOx / Ormocer
12 µm metallised PET (different producer)	PET	metallisation
12 µm PET / SiOx	PET	SiOx
12 µm PET / AlOx / adhesive / 30 µm PP	PP	PET-AlOx
6 µm aluminium)*		*Aluminium*
6 µm aluminium) / PE*	*PE*	*Aluminium*

**) This is only the case when no pinholes or other damages are present.*

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5.2.7 Functional barrier considerations

*The analogous conclusion can be made for barrier films listed in **Table 11** for long term storage at room temperature contact applications.*

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5.2.7 Functional barrier considerations

***Table 11**: Barrier films which act as a FB in reducing any migration down to levels below of 10 ppb when used for long term storage at room temperature.*

Film structure	Base polymer	Barrier material
15 µm OPA*)	PA	PA
12 µm PET	PET	PET
12 µm PVDC coated transparent Polyester film	PET	PVDC
PE / EVOH 3 µm / PE total 30 µm	PE	EVOH

**) This efficiency is only ensured when no swelling occurs*

5.2.7 Functional barrier considerations

Another understanding of absolute barriers is related to the non-permeability of plastics for particular migrants or groups of migrants.

Polymers used in food packaging are impermeable for
- inorganic pigments,
- inorganic salts or
- nanoparticles.

Annex 7 Screening

Introduction to:
Technical guidelines for migration testing

- *1. Introduction*
- *2. Sampling*
- *3. Testing in Food (real use conditions)*
- *4. Testing with Food Simulants (conventional cond.)*
- ***5. Screening Approaches***
- *6. Analytical determination of migrants*
- *7. Reporting*

5. Screening Approaches
Annex V to Regulation (EU) No 10/2011

- *5.1 Introduction*
- *5.2 Screening approaches for specific migration*
  - *Replacing specific migration by overall migration*
  - *Screening by residual content*
  - *Screening by migration modelling*
  - ***Screening food simulants***
  - *Functional barrier considerations*
- *5.3 Screening approaches for overall migration*

1

Screening by migration modelling
=> systematic approach supporting set up of the testing guideline

- *applicable to plastic materials for which migration behaviour is well known based on systematic investigations and scientific literature (PE, PP, PS, PET, PA, ...)*
- *includes best availalbe knowledge regarding migration behaviour of basic plastic materials*
- *migration typically obeys the law of diffusion*
- *real life is more complex and sometimes does not account for boundary conditions of the model*

Regulated food simulants

- *Foods with hydrophilic and acidic character with pH<4.5*
  => acetic acid 3%
- *Foods with hydrophilic and acidic character with pH≥4.5*
  => ethanol 10%; ethanol 20%
- *Foods with hydrophilic character that contain relevant amounts of organic ingredients that render the food more lipophilic*
  => ethanol 20%; ethanol 50%
- *Foods with hydrophilic and alcoholic character*
  => ethanol 20%; ethanol 50%; (ethanol 95%)
- *Foods with lipophilic character, oil-in-water emulsion character*
  => ethanol 50%
- *Foods with lipophilic character, free fat character at the contact surface*
  => vegetable oil
- *Foods with dry character*
  => food simulant E

Severity ranking of food simulants

- *Annex III to Regulation (EU) No 10/2011 Food simulant assignment for testing overall migration*

To demonstrate compliance with the overall migration limit for all type of foods testing in distilled water or water of equivalent quality or food simulant A and food simulant B and simulant D2 shall be performed.

To demonstrate compliance with the overall migration limit for all aqueous, acidic and alcoholic foods and milk products testing in food simulant D1 and food simulant B shall be performed.

>>> what about testing for all type of foods with B + D1 + D2 ???

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Ranking for regulated food simulants

For screening, a food simulant that is considered more severe than the above assigned regular food simulants can be selected per food category if desired.

Food category	More severe regulated food simulant	Most severe screening food simulant
hydrophilic, pH<4.5	acetic acid 3%	Acetic acid 3%
hydrophilic, pH≥4.5	ethanol 20%	Ethanol 50%
hydrophilic, organic character	ethanol 50%	Ethanol 95%
hydrophilic, alcoholic character	ethanol 50%	Ethanol 95%
lipophilic, oil in water emulsion character	ethanol 50%	Ethanol 95%, Vegetable oil
lipophilic, free fat character	vegetable oil	Vegetable oil*
dry character	food simulant E	Vegetable oil *

**) additional simulants under discussion*

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Screening food simulants
Section 2.2.4. of Annex V (specific migration)

To screen for specific migration, regulated food simulants can be replaced by screening food simulants if:

- it is based on scientific evidence that

- the screening food simulants overestimate migration compared to the regulated food simulants

Screening food simulants
=> for lipophilic foods

This section intends to give scientific guidance in selection of screening food simulants for vegetable oil taking into account the legal requirement that the result of specific migration tests with the screening food simulant must be at least as high as compared to the test results with vegetable oils, i.e. the migration test with the screening food simulant is at least as severe as compared to that with vegetable oils.

>>> what is scientifically available ???

Solubility considerations

This implies that the solubility of the migrants in the screening food simulant is at a minimum as high as in vegetable oils.

In combination with the use of conventional time and temperature conditions as applicable for vegetable oil, in general migration test results will be obtained that are at least as severe as the verification method.

Swelling effects

If the selected screening food simulant will cause swelling of the polymer, i.e. accelerate migration, it might be feasible to deviate from the conventional time and temperature conditions based on scientific evidence, i.e. select shorter times and/or lower temperatures for testing, to account for the swelling effect.

>>> because of limited knowledge, no generally applicable recommendation can be given for selection of adequate time/temperature conditions

General recommendation
(for specific & overall migration)

According to the scientific considerations (Feigenbaum et al., 2000) esters built from C2 to C8 acids with C2 to C8 alcohols and mixtures of these with aliphatic hydrocarbons with C6 to C8 carbon atoms can generally be recommended as screening food simulants for migration testing (specific and overall), which most likely will satisfy the requirement that the solubility of the migrants in the screening food simulant is as high as in vegetable oils, due to similar polarity of the screening food simulant with vegetable oil.

Other screening food simulants

In some cases this general approach may not work due to swelling of the polymer.

This is the reason that other screening food simulants may be used provided the solubility of the migrant in the screening food simulant is still as high as in simulant D2.

Most experience and experimental data available for ethanol 95% and iso-octane

Recommendation based on ratio K

The recommendation on alternative food simulants selection is based on the rule "similar solves similar", i.e. the closer the polarity of the migrant and the simulant is, the better the solubility of the migrant will be in the simulant.

As a measure of polarity the octanol to water partition coefficient ($K_{O/W}$) is used because plenty of scientific literature is in place and numerous estimation procedures including software tools exist.

$K_{O/W}$ for vegetable oil

Starting point is a specific migration experiment for a migrant from a plastic with vegetable oil as required by legislation. The migrant will exhibit an octanol to water partition coefficient of $K_{O/W}$(mig) and the vegetable oil will exhibit an octanol to water partition coefficient of $K_{O/W}$(oil).

Octanol to water partition coefficients for vegetable oils are in the range of 20 to 30, e.g. for:

- *Tripalmitoylglycerol $K_{O/W}$(oil) = 21.90*
- *Tristearoylglycerol $K_{O/W}$(oli) = 25.11.*

$K_{O/W}$ for solvents

When substituting the vegetable oil with an alternative food simulant this would exhibit an octanol to water partition coefficient of $K_{O/W}(sim)$.

The following systematic approach is based on the alternative food simulants ethanol and iso-octane, because they represent the two extremes from polarity point of view and substantial experience has been gained in the past published in the scientific literature.

- *$K_{O/W}(ethanol) \sim 0$ (depending on the estimation tool)*
- *$K_{O/W}(iso\text{-}octane) = 4.1$ (estimated with EPI suite)*

$K_{O/W}$ corrected by molecular weight

Because of the significantly lower molecular weight of alternative food simulants compared to vegetable oil the octanol to water partition coefficient $K_{O/W}(sim)$ is corrected by the ratio of the molecular weight between vegetable oil and the alternative food simulant.

- *$K_{O/W}^{corr}(ethanol) = 861.44 / 46 * K_{O/W}(ethanol) \sim 0$*
- *$K_{O/W}^{corr}(iso\text{-}octane) = 861.44 / 114 * K_{O/W}(iso\text{-}octane) = 31$*

>>> a polarity scale from 0 (polar) to 31 (non-polar) results

8

Condition for the selection of the alternative food simulant

$ratio^K = [K_{O/W}(sim) - K_{O/W}(mig)] / [K_{O/W}(oli) - K_{O/W}(mig)]$

$$-1 < ratio^K < 1$$

If the $ratio^K$ is above -1 and below 1 the alternative food simulant can be considered to be an alternative for vegetable oil.

If the above $ratio^K$ is between -1.5 and -1 respectively 1 and 1.5 the food simulant may be a reasonable alternative for vegetable oil, but a certain risk of underestimation exists.

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What about ethanol 95% and iso-octane ?

Polarity scale based on octanol to water partition coefficients:

Based on the above considerations it can be considered that in most of the cases specific migration testing can be performed with the alternative food simulants ethanol 95% for substances with an octanol to water partition coefficient log $K_{O/W}$ < 14 and with iso-octane for substances with an octanol to water partition coefficient log $K_{O/W}$ > 26.

There is a gap for substances exhibiting a polarity similar to vegetable oil which is too far from that of ethanol 95% but not close enough to that iso-octane.

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Example for laurolactam $K_{O/W}$=3.6
(monomer in PA12)

The calculation for ethanol is:

$$ratio^K = [0 - 3.6] / [22.74 - 3.6] = -0.19$$

$$\mathbf{-1 < ratio^K < 1}$$

*For testing the specific migration of laurolactam ethanol **is** a suitable alternative food simulant for vegetable oil because laurolactam is better soluble in ethanol than in vegetable oil due to the fact that $K_{O/W}$(laurolactem) is closer to $K_{O/W}$(ethanol) than to $K_{O/W}$(oil).*

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Example for laurolactam $K_{O/W}$=3.6
(monomer in PA12)

The calculation for iso-octan is:

$$ratio^K = [31 - 3.6] / [22.74 - 3.6] \mid = 1.43$$

$$\mathbf{< 1\ ratio^K < 1.5}$$

*For testing the specific migration of laurolactam iso-octane **may be** a suitable alternative food simulant for vegetable oil because laurolactam is less soluble in ethanol than in vegetable oil due to the fact that $K_{O/W}$(laurolactem) is closer to $K_{O/W}$(oil) than to $K_{O/W}$(iso-octane).*

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Example for Irganox 1076 $K_{O/W}$=13.4
(antioxidant in PP)

The calculation for ethanol is:

$ratio^K = [0 - 13.4] / [22.74 - 13.4] = -1.43$

$\mathbf{-1.5 < ratio^K < -1}$

*For testing the specific migration of Irganox 1076 ethanol **may be** a suitable alternative food simulant for vegetable oil because Irganox 1076 is less soluble in ethanol than in vegetable oil due to the fact that $K_{O/W}$(Irganox 1076) is closer to $K_{O/W}$(oil) than to $K_{O/W}$(ethanol).*

European Commission

Example for Irganox 1076 $K_{O/W}$=13.4
(antioxidant in PP)

The calculation for iso-octan is:

$ratio^K = [31 - 13.4] / [22.74 - 13.4] = 1.88$

$\mathbf{ratio^K > 1.5}$

*For testing the specific migration of Irganox 1076 iso-octane **is not** expected to be a suitable alternative food simulant for vegetable oil because Irganox 1076 is much less soluble in iso-octane than in vegetable oil due to the fact that $K_{O/W}$(Irganox 1076) is much closer to $K_{O/W}$(oil) than to $K_{O/W}$(iso-octane).*

European Commission

Validation by literature

G. Beldì, S. Pastorelli, F. Franchini, C. Simoneau; "Time-and temperature-dependent migration studies of Irganox 1076 from plastics into foods and food simulants."; Food Additives and Contaminants, Vol. 29, No. 5, May 2012, 836–845

For Irganox 1076 a systematic migration study from LDPE in various foods and olive oil compared to ethanol 95% and iso-octane exists which demonstrates that ethanol 95% is still a suitable alternative simulant compared to olive oil.

The systematic migration study also indicates that iso-octane might be a suitable alternative food simulant as well. Due to strong swelling of the LDPE film by iso-octane and sufficient solubility for the amount of Irganox 1076 present in the LDPE film in iso-octane the migration into iso-octane is higher compared to olive oil.

European Commission

Remark 1

Finally solubility of substances in liquid media strongly depends on temperature. Because most of the literature data cited are at or below 60°C it is considered that the recommendation made above are valid up to a maximum temperature of 70°C. Above 70°C the use of alternative food simulants in many cases will induces physical changes of the materials investigated and from laboratory point of view their use above 70°C is dangerous due to their flammability.

European Commission

Remark 2

The closer the polarity of the alternative food simulant to the polarity of the plastic is, the higher is the risk of interaction between polymer and simulant, i.e. swelling of the polymer by uptake of alternative food simulant. Depending on (a) the amount of simulant taken up by the polymer and (b) the extent of the plasticising effect related to the amount of simulant taken up, an increase of the migration rate compared to testing with vegetable oil can be expected.

Remark 3

The swelling effect may open the possibility for deviations from the conventional time and temperature testing conditions for vegetable oil when testing with alternative food simulants.

At the moment there is limited scientific background available to make a general recommendation regarding selection of time and temperature conditions for migration testing with respect to the possible combinations of plastic materials and alternative food simulants.

Testing specific migration with alternative food simulants

- *Selection of alternative food simulants for specific migration testing should follow the criteria defined for ratio^{K} described above.*
- *As a first recommendation time/temperature conditions for specific migration testing with alternative food simulants should be used as those to be used with vegetable oil.*

European Commission

Time/temperature conditions

If based on ratio^{K} ethanol or iso-octane are recommended

plastic	vegetable oil	ethanol 95%	iso-octane	simulant E
LDPE, LLDPE PP random PP rubbery	@ > 100°C			same t/T as for oil
	10d @ 60°C	10d @ 60°C	1d @ 60°C	
	10d @ 40°C	10d @ 40°C	1d @ 40°C	
	10d @ 20°C	10d @ 20°C	1d @ 20°C	
HDPE	@ > 100°C			same t/T as for oil
	10d @ 60°C	10d @ 60°C	2d @ 60°C	
	10d @ 40°C	10d @ 40°C	2d @ 40°C	
	10d @ 20°C	10d @ 20°C	2d @ 20°C	
PP isotactic	@ > 100°C			same t/T as for oil
	10d @ 60°C	10d @ 60°C	2d @ 60°C	
	10d @ 40°C	10d @ 40°C	2d @ 40°C	
	10d @ 20°C	10d @ 20°C	2d @ 20°C	
PS	@ > 100°C			same t/T as for oil
	10d @ 60°C	2d @ 60°C	2d @ 60°C	
	10d @ 40°C	2d @ 40°C	2d @ 40°C	
	10d @20°C	2d @ 20°C	2d @ 20°C	

European Commission

Time/temperature conditions

If based on ratio[K] ethanol or iso-octane are recommended

plastic	vegetable oil	ethanol 95%	iso-octane	simulant E
PET, PBT, PEN	@ > 100° C			same t/T as for oil
	10d @ 60° C	1d @ 60° C	10d @ 60° C	
	10d @ 40° C	1d @ 40° C	10d @ 40° C	
	10d @20° C	1d @ 20° C	10d @ 20°	
PA 6, PA 6.6	@ > 100° C			same t/T as for oil
	10d @ 60° C	1d @ 60° C	10d @ 60° C	
	10d @ 40° C	1d @ 40° C	10d @ 40° C	
	10d @20° C	1d @ 20° C	10d @ 20°	
PA 12	@ > 100° C			same t/T as for oil
	10d @ 60° C	1d @ 60° C	10d @ 60° C	
	10d @ 40° C	1d @ 40° C	10d @ 40° C	
	10d @20° C	1d @ 20° C	10d @ 20°	
PVC, rigid	@ > 100° C			same t/T as for oil
	10d @ 60° C	1d @ 60° C	10d @ 60° C	
	10d @ 40° C	1d @ 40° C	10d @ 40° C	
	10d @20° C	1d @ 20° C	10d @ 20°	

European Commission

Arrhenius calculator

calculation of other t/T-conditions

For contact times above 30 days at room temperature and below the specimen shall be tested in an accelerated test at elevated temperature for a maximum of 10 days at 60 °C. Testing time and temperature conditions shall be based on the following formula.

$$t2 = t1 * \mathrm{Exp}\left((-Ea/R) * (1/T1 - 1/T2)\right)$$

E_A - worst case activation energy
R - gas constant 8.31 J/K/mol
t1 - real contact time
t2 - testing time
T1 - real contact temperature
T2 - testing temperautre

European Commission

Arrhenius calculator
calculation of other t/T-conditions

- *can only be used for plastics where the migration is controlled by diffusion.*
- *if hydrolysis of a plastic, e.g. melamine or polycarbonate takes place at the foreseen conditions of use, the Arrhenius equation cannot be used.*
- *may be helpful to calculate other t/T-conditions not covered in the tables*
- *extend of use still under discussion*

Screening food simulants
Section 3.4.2. of Annex V (overall migration)

To screen for overall migration, regulated food simulants can be replaced by screening food simulants if:

- it is based on scientific evidence that

- the screening food simulants overestimate migration compared to the regulated food simulants

Alternative food simulants
(ethanol 95% + iso-octane)

The two solvents ethanol 95% and iso-octane span the polarity range of migrants from plastics encountered in practice.

Substituting the overall migration test with vegetable oil requires testing with both solvents under consideration of the highest result for compliance evaluation.

Recommended time/temperature conditions for overall migration testing account for swelling.

European Commission

plastic	vegetable oil	ethanol 95%	iso-octane
LDPE, LLDPE PP random PP rubbery	OM2	10d@40°C	2d@20°C 1d@40°C
	OM1	2d@40°C	1d@20°C
HDPE	OM2	2d@60°C	1d@40°C
	OM1	2d@40°C	1d@20°C
PP isotactic	OM2	2d@60°C	1d@40°C
	OM1	2d@40°C	1d@20°C
PET, PBT, PEN	OM2	1d@40°C 1d@50°C	10d@40°C
	OM1	1d@20°C	2d@40°C
PS	OM2	1d@40°C	1d@40°C
	OM1	1d@20°C	1d@20°C
SBS	OM2	2d@60°C	2d@20°C 1d@40°C
	OM1	2d@40°C	1d@20°C
PA 6, PA 6.6	OM2	1d@40°C	2d@60°C
	OM1	1d@20°C	2d@40°C
PA 12	OM2	1d@40°C	2d@60°C
	OM1	1d@20°C	2d@40°C
PVC, rigid	OM2	1d@40°C	2d@60°C
	OM1	1d@20°C	2d@40°C

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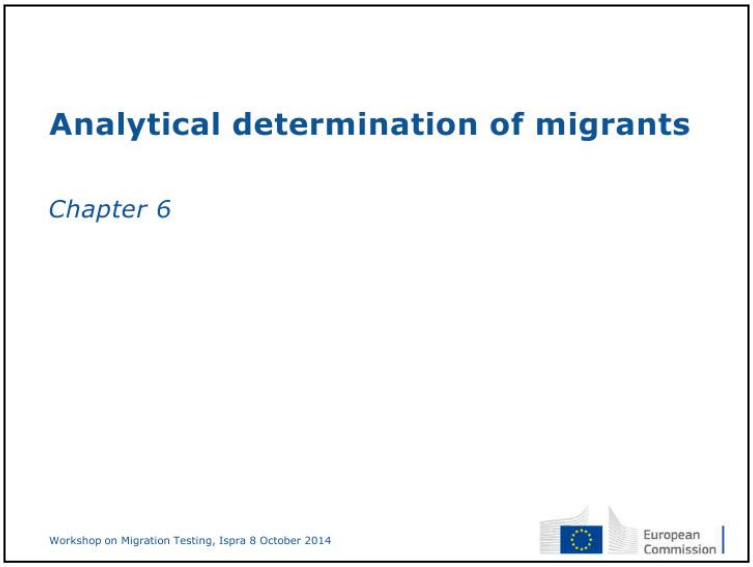
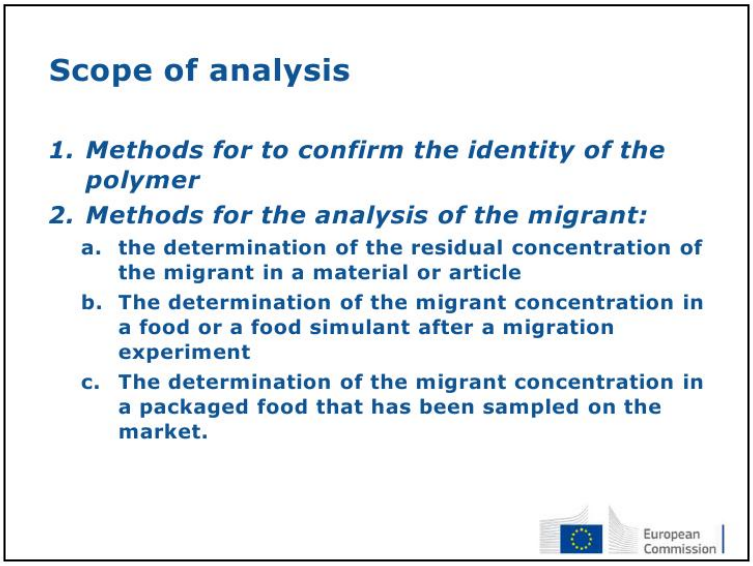
Annex 8 Analytical determination of migrants





1

Hierarchy of methods

1. ***Methods in EU legislation***
2. ***CEN methods***
   - **Present OM and SM methods refer to old legislation**
   - **Not valid for Regulation (EU) No 10/2011 except methods for residual content**
3. ***Other validated methods***
   - **Determination of Di-isodecyl phthalate (DIDP) in sunflower oil**
   - **Determination of butyl hydroxytoluene, benzophenone, bis(2-ethylhexyl)adipade, diisobutylphtalate and 1,2-cyclohexanedicarboxylic acid diisononyl ester in FS E**

European Commission

Content of method of analysis

1. ***Scope***
2. ***Principle***
3. ***Sampling***
4. ***Reagents (Safety precautions)***
5. ***Apparatus***
6. ***Procedure***
7. ***Confirmation***
8. ***Measurement uncertainty***
9. ***Test report***

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Choice of analytical method (1)

1. ***Substance: volatility, polarity, functional groups***
2. ***Food (simulant)***
3. ***Concentration***

Type of substance	Example	Predominant technique
Volatile organics (bp< 150°C)	Monomers, solvent residues (e.g. styrene)	Headspace, SPME, purge & trap and GC, with mostly FID or MS
Semi-volatile organics (bp <300°C)	Plasticisers, glycols, additives, MW <400-500 amu (e.g. phthalates)	Liquid injection (split, splitless, PTV, on-column etc) and GC with FID or MS
Non-volatile organics	Antioxidants, polymeric plasticisers, additives with M_w >400-500 amu (e.g. perfluorotelomers)	LC in majority reverse phase, with diode array, fluorescence or MS detection
elements	Ba, Co, Cu, Fe, Li, Mn, Zn	ICP-MS

Choice of analytical method (2)

1. ***Extraction/digestion***
2. ***Clean-up***
3. ***Determination***

±400 non-validated methods available at EURL-FCM

Characterisation of materials (1)

- ***Identity***
  1. **Identification of materials of both sides of FCM**
     - **e.g. wavelength range 600-4000 cm^{-1} by FTIR**
     - **Libraries available**
  2. **Preparation microtome cross-section of the sample**
  3. **Identify different layers using transmitted light microscope using polarised light**
  4. **Separate different layers by peeling or solvents**
  5. **Identify all separated layers**
- ***Residual concentration***
  - **Dissolution of polymer in solvent**
  - **Digestion**

Characterisation of materials (2)

- ***EN 13130-4:2004 Determination of 1,3-butadiene in plastics***
- ***EN 13130-6:2004 Determination of vinylidene chloride in plastics***
- ***EN 13130-8:2004 Determination of isocyanates in plastics***
- ***CEN/TS 13130-17:2005 Determination of carbonyl chloride in plastics***
- ***CEN/TS 13130-20:2005 Determination of epichlorohydrin in plastics***
- ***CEN/TS 13130-22:2005 Determination of ethylene oxide and propylene oxide in plastics***

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Determination of surface area

- *Calculation of the surface area using mathematical formulas*
- *Wrapping the sample in paper,*
- *Wrapping the sample in aluminium foil*
- *Drawing the sample outline on paper*
- *3D-scanner*

European Commission

Calibrants

- *Known purity (>95%)*
- *Store at low temperature*
- *Exclude degradation: light and moisture*
- *Check purity beyond expiry date*
  - ✓ Check detector response old vs. new (n≥3)
  - ✓ Deviation ≤10%
- *Use CAS no. from Declaration of Compliance*
  - ✓ No info → use both and compare
- *Lower quality (≤95%)*
  - Check interferences using more columns

  For GC-FID: $M = m\ A_{calibrant}/A_{total}$

European Commission

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Quantification

- ***Calibration of every batch of analysis***
  - ✓ **Internal standards preferred**
- ***If possible***
  - ✓ **Isotope-labelled standards**
  - ✓ **Injection standard for recovery**

European Commission

Quality assurance

- ***Quality assurance samples should be included in each batch, i.e.***
  - ✓ **solvent blanks**
  - ✓ **procedural blanks**
  - ✓ **certified or other well characterised reference materials and/or spiked sample**
- ***Participation in inter-laboratory comparison exercises***

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Overall migration – vegetable oil

- ***temperature range of 20-100°C***
- ***temperature range of 5-20°C***
  - de-waxed sunflower oil
- ***temperature range of 100-175°C***
  - Vegetable oil 100-175°C
  - FS E > 175°C
- ***in case of incomplete extraction of vegetable oil (5-175°C)***
  - dissolve plastic in chloroform, toluene, xylene or tetrahydrofuran (pentane (non-polar) or 95/5 v/v pentane-ethanol (polar)

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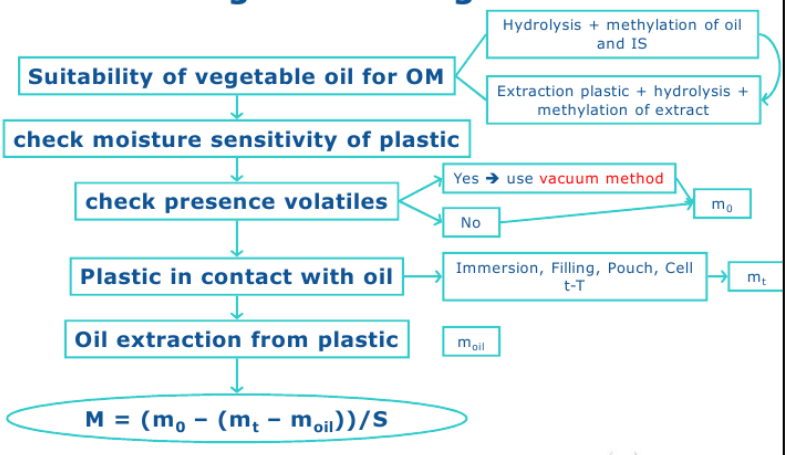
Overall migration – vegetable oil



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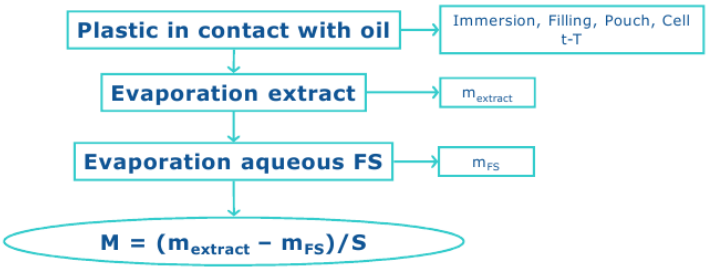
Overall migration – aqueous FS + D1



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Method performance (1)

- ***Overall migration***
  - **Aqueous FS + D1**
    - ✓ Analytical tolerance of 2 mg/dm² (12 mg/kg food)
    - ✓ Results valid if $|x_n - \bar{x}| \leq 2\ mg/dm^2$ for all n=3
    - ✓ If not, repeat OM test
  - **Vegetable oil – single use articles**
    - ✓ Analytical tolerance of 3 mg/dm² (20 mg/kg food)
    - ✓ Results > 10 mg/dm² valid if $x_n - \bar{x} \leq 3.3\ mg/dm^2$ for all n=3

      n=4:
    - ✓ Results valid if $|x_n - \bar{x}| \leq 3\ mg/dm^2$ for all n=4
    - ✓ If one results > $3\ mg/dm^2$ ➔ reject and calculate new $\bar{x}$
    - ✓ If two results > $3\ mg/dm^2$ ➔ reject result with largest difference and calculate new $\bar{x}$; check $|x_n - \bar{x}| \leq 3\ mg/dm^2$ for remaining n=3

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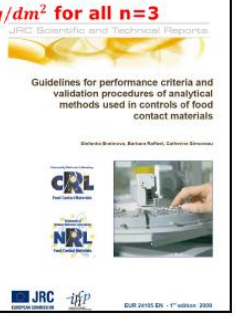
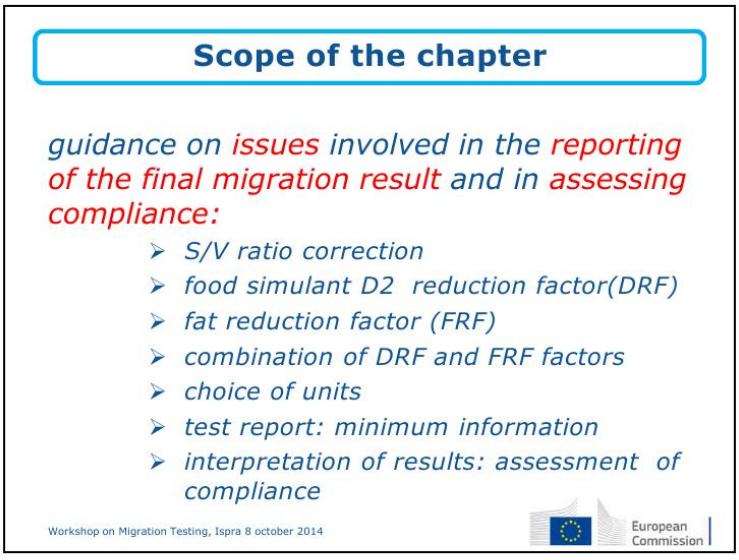
Method performance (2)

- ***Overall migration***
  - **Vegetable oil – repeated use articles**
    - ✓ **Analytical tolerance of 3 mg/dm² (20 mg/kg food)**

    For each migration
    - ✓ **Results >10 mg/dm² valid if $x_n - \bar{x} \leq 3.3\ mg/dm^2$ for all n=3**
    - ✓ **Results <10 mg/dm² valid if $|x_n - \bar{x}| \leq 3\ mg/dm^2$ for all n=3**
    - ✓ **If not, repeat OM test**
- ***Specific migration***
  - **Follow "standard level" validation scheme of Bratinova et al. (2009)**
  - **x ±U where U = k u_c**



Annex 9 Reporting of migration test results



1

Surface-to-Volume ratio correction

- *Applies for results expressed in mg/kg when S/V ratio in test conditions differs from:*
  - **a) *worst case foreseeable or actual S/V ratio***
    - ✧ *materials/articles intended for children (≤ 3 years)*
    - ✧ *articles with V from 500 mL to 10 L*

$$M_{S/V} = (M_{test} \times S/V_{actual}) / S/V_{test}$$

where:

$M_{S/V}$	*migration in mg/kg*
M_{test}	*migration in mg/kg food or simulant released in the migration test*
S/V_{actual}	*surface to area ratio (dm²/kg food) under real conditions of use*
S/V_{test}	*surface to area ratio (dm²/kg food) in the migration test*

Surface-to-Volume ratio correction

b) conventional S/V ratio

- ✧ *articles with V less than 500 mL or higher 10 L*
- ✧ *articles/materials with unknown S/V in actual use*

$$M_{S/V} = (M_{test} \times 6) / S/V_{test}$$

where:

$M_{S/V}$	*migration in mg/kg*
M_{test}	*migration in mg/kg food or simulant in the migration test*
S/V_{test}	*surface to area ratio (dm²/kg food) in the migration test*

Food simulant D2 reduction factor

- *compensates the higher extraction power of simulant D2 in comparison with certain fatty foods*
- *also applicable to substitute simulants of D2*
- *applies to OM and SM*
- *Not applicable for substances:*
  - *behind a functional barrier*
  - *with a SML "not detectable"*
  - *cap, gasket, stopper or similar sealing article, for which the intended used is unknown*

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Food simulant D2 reduction factor

- *DRF factor get values between 1 and 5 (sub-column D2, table 2, annex 3)*

(1)	(2)	(3)					
Reference number	Description of food	Food simulants					
		A	B	C	D1	D2	E
08.06	Sandwiches, toasted bread pizza and the like containing any kind of foodstuff						
	A. With fatty substances on the surface	X				X/5	
	B. Other						X
08.07	Ice-creams			X			

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Food simulant D2 reduction factor

- *the migration result shall be divided by the DRF before comparison with the SML:*

$$M_{DRF} = M_{S/V} / DRF$$

where:

M_{DRF}	*overall or specific migration corrected by the DRF in mg/dm² or mg/kg food*
$M_{S/V}$	*experimental determined migration, corrected to the S/V ratio if applicable.*

Fat Reduction Factor

- *based on the fact that the ingestion of fat per day by an adult is 200 g, instead of 1 kg*
- *applies to the SM of certain lipophilic substances*
- *Conditions required for the application of FRF:*
  - *substance indicated as "yes" in column 7, table 1, annex 1*
  - *simulants D1, D2 or food*
  - *fat content of food intended for contact: ≥ 20 %*
  - *food intended for contact: not for children ≤ 3 years*

Fat Reduction Factor

- ✧ *not article/material for which the S/V ratio in real use cannot be estimated (art. 17.2(b))*
- ✧ *the S/V corrected migration value is not >60 mg/kg*

FRF= (g fat in food/kg food)/200 = (% fat x 5)/100

- ➢ *FRF values in the range 1-5*
- ➢ the migration result shall be divided by the FRF before comparison with the SML :

$$M_{FRF} = M_{S/V} / FRF$$

where:

M_{FRF}	*specific migration corrected by the FRF in mg/kg food*
$M_{S/V}$	*experimental determined migration, corrected to the S/V ratio if applicable.*

Total Reduction Factor: combination of DRF and FRF

- ➢ *The combination applies in specific migration*

$$TRF = DRF \times FRF$$

where:

DRF	*correction factor for simulant D2*
FRF	*fat reduction factor*

- ➢ *the specific migration result shall be divided by the TRF before comparison with the SML :*

$$M_{TRF} = M_{S/V} / TRF$$

where:

M_{TRF}	*specific migration corrected by the TRF in mg/kg food*
$M_{S/V}$	*experimental determined migration, corrected to the S/V ratio if applicable.*

Total Reduction Factor: combination of DRF and FRF

- *Further information on the application of reduction factors: Guidance document on fat reduction factor, functional barrier concept, phthalates and primary aromatic amines*

http://ihcp.jrc.ec.europa.eu/our_labs/eurl_food_c_m/publications/publications#technical-guidelines

Choice of units for migration test results

- ***Considerations in selecting units:***
  - *OM or SM result?*
  - *cap, gasket or similar sealing article?(art.17.3, 17.4)*
  - *known intended use of the article?*
  - *intended use for food for children ≤ 3 years?*

Choice of units for migration test results

Overall migration

- *measure of inertness of the material*

units ⟶ *mg/dm²*

if intended use is food for children ≤ 3, and are not caps, gaskets or similar sealing articles

units ⟶ *mg/kg food, (actual S/V)*

Choice of units for migration test results

Overall migration

- ***special case**: caps, gaskets, or similar sealing materials:*
  - *a) intended used is known: mg/dm² applying the total contact surface: container + sealing article.*
    Note: it applies independently of the material of the container

cap migration= A mg
cap internal contact surface = S1 dm²

internal bottle contact surface = S2 dm²

OM_{cap} = A / (S1 + S2) = C mg/dm²

Choice of units for migration test results

→ ***Compliance of combined articles:*** *cap + container:*

OM_{cap} = C mg/dm²
OM_{bottle} = D mg/dm²
$OM_{cap+bottle}$ = (C + D) mg/dm²…….≤ 10 mg/dm²

Note: if the combined article is for children ≤ 3 years, to assess compliance, the result is expressed in mg/kg for the actual content:

$mg/kg = mg/dm^2 \times S/V_{actual}$…….≤ 60 mg/kg

b) intended used is unknown: mg/article

cap *migration= A mg*
OM_{cap} = A mg/article

→ ***End user of the cap: verification of compliance of combined articles:*** *cap + container (as above)*

Choice of units for migration test results

Specific migration

➢ *units mg/kg food (Art. 17.1 and 17.2)*

✧ *applying actual in use surface to volume ratio:*
- *articles with volume in the range 500 mL- 10L*
- *articles intended to contact food for children ≤ 3 years*

✧ *applying the conventional ratio of 6 dm²/kg food*
- *articles with volume < 500 mL or > 10 L*
- *films, sheets*
- *unknown/ unestimable S/V in real use*

Choice of units for migration test results

Specific migration

- ***special case**: caps, gaskets, or similar sealing materials:*
  - *a) intended used is known*
    - *for articles with V = 500 mL – 10 L and any article intended for children ≤ 3 years:*
      mg/kg food, applying actual content
    - *for articles with V < 500 mL or >10 L and articles with unknown/unestimable S/V ratio in actual use:*
      mg/kg food, applying S/V ratio of 6 dm^2/kg food

      calculate mg/dm^2, applying the total contact surface: container + sealing article

      → *mg/kg food = 6 x mg/dm^2 total contact surface*

Choice of units for migration test results

Specific migration

→ *cap migration= A mg*
cap internal contact surface = S1 dm^2

300 mL
internal bottle contact surface = S2 dm^2

***SM_{cap}** = A / (S1 + S2) = C mg/dm^2*
…….for comparison with SML:
*C mg/dm^2 x 6 = **SM_{cap} (mg/kg)***

500 mL

***SM_{cap} (mg/kg)** = A mg / 0,5 kg*

→ ***Compliance of combined articles:***

$SM_{cap} + SM_{bottle} \leq SML$

Choice of units for migration test results

Specific migration

b) intended used is unknown: mg/article

cap *migration= A mg*
SM_{cap} = A mg/article

→ ***End user of the cap: verification of compliance of combined articles:*** *cap + container (as above)*

Minimum information in the test report

guidance:

- *identification of the sample (consignment, lot, sample number)*
- *info for complete description of the sample (eg. chemical type, trade mark, dimensions, pictures advisable)*
- *date and method of sampling*
- *analyte/type of test, reference to method(s) used*
- *testing conditions (T, t, contact, number exposures)*
- *surface/volume ratio in migration testing*
- *name of the laboratory, person responsible of the analysis and date of report*

Interpretation of the results. Assessment of compliance with migration limits

enforcement point of view:

if

analytical result – expanded uncertainty > legal limit

sample not-compliant

European Commission

DISCUSSION

European Commission

Annex 10 Questions

Sampling

OK: General remark on sampling - Due to the fact, that we finally (Chapter: Results) assess the compliance on each sample, the question could be raised, why taking so many samples. Therefore, the amount of samples taken should be more a recommendation than a fixed rule. This should be discussed.

NRL EL: For repeated use testing for OM you need more samples since you perform the test differently compared to SM testing

- Action EH: check OM test for amount needed it is for test with olive oil only
- OK: It is more an exception, than the rule. I prefer, that we mentioned somewhere, that on request of the official control laboratory for such a OM test the corresponding needed amount shall be sampled.

RV: What to do with very large articles (e.g. >10 L)

- Proposal: this depends on the size of the article. E.g. 1) take indicated amount of samples of 15 but this may be sufficient for testing all parameters and test condition since you may cut piece from the article. Of course with a plastic multilayer the test specimen should be flat or sealable (pouch).
- OK: See general remark above. If it is a recommendation, than an adaption for sampling such large articles will be easy.

NRL NL: Why sampling such a huge amount of lids?

- Proposal: in Ch4 we need to give directions how many lids you need to test in order to cover the inhomogeneity of plastisols in lids.
- OK: See general remark above.
- OK: From my point of view official control laboratories do not have the time for testing such a huge amount of samples. Take into consideration, that they assess each sample for compliance!

NRL-AT: It may not be possible to take 15 samples when sampling at the retailer as there may not be that many available

- Proposal: as 882/2004 allows exceptions then these should be allowed in the guidelines here.

NRL EL: did you consider aging of materials. The example was of an article that was produced and only tested after 1 year. It did not comply whereas an article that was tested directly complied.

- Proposal: mention 1-2 lines on this issue in the Introduction: The MT guidelines do not cover this issue since it is considered as part of GMP.
- OK: In Germany, the official control laboratories have to test and assess samples within a certain time frame (mostly within 6 weeks). Therefore, they have the state their expert opinion for the sample as taking from the market at the corresponding time. If it is indicated, that a sample will be above an SML after a certain, or at the end of the shelf-life, than the SD and DoC should be requested from the responsible company. Yes, it will be a matter of DoC, SD and GMP!
- BS said that if ageing is important this should be taken up at a legislative level and not as part of the guidelines.

- RF also commented that it was the responsibility of the producer to demonstrate the technical suitability of the product as the example provided by NRL-Greece was one of technical suitability rather than, for example, the effect of a functional barrier.

Food and food as simulant

NRL PT: Who is responsible if e.g. phthalates are exceeding the SML in food?

- Proposal: the food is not compliant. It is up to the retailer to find out where the source of the phthalates is. It is up to the MS Competent Authority to take a risk management decision (there was also the example of Fe migration).
- OK: The example on Fe should not been taken too seriously. It is really a case to case decision, taking into account which phthalate (different toxicological end points!!) at which concentration is present in the actual food sample. In the context of FCM risk assessment should be taken into consideration. Furthermore, Reg. (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs could also be used for the assessment of the food.

NRL AT: to prevent food waste, food producers tend to avoid labelling with expiry dates. How to decide on compliance?

- Proposal: Supporting documents to see the exposure conditions that the producers considered to be the worst case?

NRL UK: How should reconstitution/dilution be dealt with?

- Proposal: For foodstuffs that are diluted with water prior to consumption compliance should be checked by comparing the concentration in the diluted food/drink with the SML. If this approach is agreed should the guidelines indicate which foods/drinks this is applicable too? It was also noted that the concentrated product can be tested and the measured concentration corrected for the dilution prior to comparison with the SML.
- See Oliver's related comment later for an alternative viewpoint.

RV: the text states that if the concentration in the food is greater than the SML then the product is non-compliant but it is possible that the contamination of the food is not from that source.

- Proposal: Tone down the wording to state that it should be investigated further rather than is definitely non-compliant.

Food simulants verification of compliance

RV: how to test laminates with polyolefin as food contact layer and non-polyolefin in other layers regarding substitute test for food simulant D2?

- Proposal the material, which comes into contact with the food should be used to select the conditions.

RV: what to do when OM 8 or OM 9 are also not feasible?

- Proposal these tests are only an escape for some high temperature applications. We should think if it is useful to have the tests in the legislation.

RV: How are volatiles in OM test defined?

- By evaporation at 105°C of aqueous FS
- By vacuum drying for Simulant D – to demonstrate non-compliance is it necessary to carry out the vacuum drying step to ensure no volatiles are included in the overall migration?

- By evaporation when taking vials to constant weight for the alternative simulants

NRL PT: there was a discussion on whether tables 1-2 can be used for screening (in addition to table 7 in 5.2.4.2 or 11 in 5.3.2).

- It appeared that when tables 1-2 are used for testing that nobody identified non-compliances.
- In the meeting Roland proposed that the text below table 2 should be changed to "NOTE: The simulants and test conditions listed in the Tables are not necessarily applicable for screening purposes. For screening tests the conditions given in Chapter 5 shall be applied."

RV: 10d@40°C and 10d@50°C in section 2.1.4 and OM3 in Annex V of Regulation shall also mention cooling in addition to heating

- Action EH: OK. Agreed that the text should be consistent with that in the Regulation.

NRL-PT: When selecting the alternative simulant for a multi-layer material does it matter in which layer the migrant is

- Proposal: In the meeting Birgit replied that the food contact layer should be considered when selecting the simulant as this is the one in contact with the food and so has the greatest interaction with the food/simulant. Fatima asked for better explanation to be given in the text.

NRL-DK: If Table 1 and 2 of chapter 4 are the product of migration modelling and expert judgement then will the guidelines be constantly updated?

- Proposal: the guidelines will be updated whenever there is scientific or legislative reasoning to do so and that due to translation requirements it may only be possible to change when legislation changes. The Commission will need to find a means of providing updates.
- the conditions given in Tables 1 and 2 are based on the existing knowledge, scientific data and migration test results and so are a good step forward compared to previous guidance.

RV: Can simulant derived for OM be used to determine SM for less severe conditions?

- Proposal: Yes for simulant D2 (not the alternative simulants) as long as it can be demonstrated that the migration will occur by diffusion and not hydrolysis and equivalence or worst case can be demonstrated applying the Arrhenius calculator.

NRL AT: Is it possible to use OM test conditions other than those defined in the legislation?

- Proposal: No. OM is a measure of the inertness of the material and as conditions are defined in the Regulation these must be adhered to.

NRL SI: OM repeat use procedure is not described in the guideline

- Proposal: Add guidance?

NRL-Greece: asked how the toxicity of mixtures of migrants could be considered

- Proposal: It can't as insufficient data is available and as there are different toxicological end points you can't simply add all migration results together. SML are established conservatively.

Testing repeated use articles with a functional barrier. Substances behind FB migrate in retardation so how are we sure that we capture those substances by 3 successive migration tests and that their migration values are decreasing from 1st to 3rd migration?

- Proposal Part of GMP/supporting documentation. we have to prove that the functional barrier is 100% (e.g. modelling)

RV: examples of OM5 and 6 are not clear

- Proposal who rewrite?

NRL CY: how to test articles used in microwave?

- Proposal: EN 14233 gives a procedure to measure the interface temperature in the microwave and then you can apply this temperature in an conventional oven translate microwave conditions to conventional oven test conditions

NRL AT: For OM4 (1 hour at 100C) is it necessary to use a pressure cooker to reach the temperature as OM4 does not allow for reflux?

- Proposal: Yes as reflux is not included in the Regulation to reach the required temperature exposure in a pressure cooker will be necessary.

Screening

NRL PT: proposal to put the Ko/w equation into the main text

- Proposal RB?

RV some substances have a high molecular mass than 1000.

- Proposal: extend mass range from 750-1000 to 750-1500. For all tables in our guidelines? If table is for MW 751 then will be appropriate for any masses above this and so can be extended to 1500.

RV: perfluoro substances are relative more volatile considering their mass. Needs consideration.

- OK: Yes, the atomic radius of fluor is considerably smaller compared to hydrogen. Therefore, molecules based on fluor atoms instead of hydrogen atoms got a higher molecular mass without changing the corresponding molecular volume. Furthermore the per fluorination, as already mentioned, increased volatility of such substances. The assumption, that substances with a molecular weight > 1000 Dalton would not been absorbed by the gastrointestinal tract and therefore, have not any toxicological effect, could not assigned to those kind of substances. Therefore, for such perfluorinated substance with a molecular distribution above a molecular mass of 1000 Dalton should also be considered.

Methods

Turkey: For oil extraction from plastics soxhlet is defined in the CEN methods. Can this be replaced by ASE?

- Proposal: Not for inclusion here but may be considered by future work of CEN. For accreditation would need to demonstrate that the same results are obtained using the two extraction techniques. To be considered as a standard method then the equipment needed must be accessible to all labs. This may not yet be the case for ASE.

Turkey: What is the analytical tolerance for overall migration?

- Proposal: Check correct analytical tolerance of 2 mg/dm^2 for aqueous simulants is included in the guidelines.

NRL FR: Will the methods in the Annex be kept after CEN have updated 1186?

- Proposal: This will need to be considered. If they are included now and then removed as the guidelines are publically available and CEN standards must be purchased.

Results

Apply FRF on dry food or on reconstituted food

- FRF applies to the reconstituted/diluted food if it is finally accepted that for those foods labelled to be reconstituted or diluted (e.g. powder soups, concentrated juices), the result is referred to the reconstituted/diluted food.
- OK: From my point of view, the topic on reconstituted food / food preparation is such an essential question in the context of all FCM, that it shall be discussed more generally at a other level. It is not adequate to implement such an exception within a guideline to a regulation for one material! Therefore the corresponding paragraph should be deleted. Nowadays there is a questionnaire forwarded from the commission to the MS asking for FAQ in the context of Reg 1935/2004. Maybe, this is the right place for the discussions.

RV: Clarify the child- adult issue: sometimes the article is compliant for use by children and non-compliant for use by adults.

- In my view this case would only be possible with articles with volume > 10 L and a S/V ratio < 6 or articles with volume < 0,5 L and S/V <6. Are such articles, intended for adults + children <3, very frequent in the market?

Verification of compliance (official control). It was asked what would be the decision if one article out of three fails? Would the sample be considered not-compliant or the mean of the three articles should compared to the limit? It was suggested to indicate this in the guidelines.

- For SM, if we are consistent with former taken decisions, such as in *EU guidelines for the import of polyamide and melamine kitchenware from China and Hong Kong* (section 6.4), then if one article fails the sample would be not compliant. This would not be the case for OM (why?).

<u>General</u>

Put background documents as annex or as separate documents? I would prefer in the annex

- OK: Due to the fact, that the guidelines are already a huge document, I really put it in the background/separate document.

Annex 11 Customer satisfaction survey

Customer Satisfaction Survey

Workshop "Guidelines on migration testing of food contact Materials in support of Regulation (EU) No 10/2011"

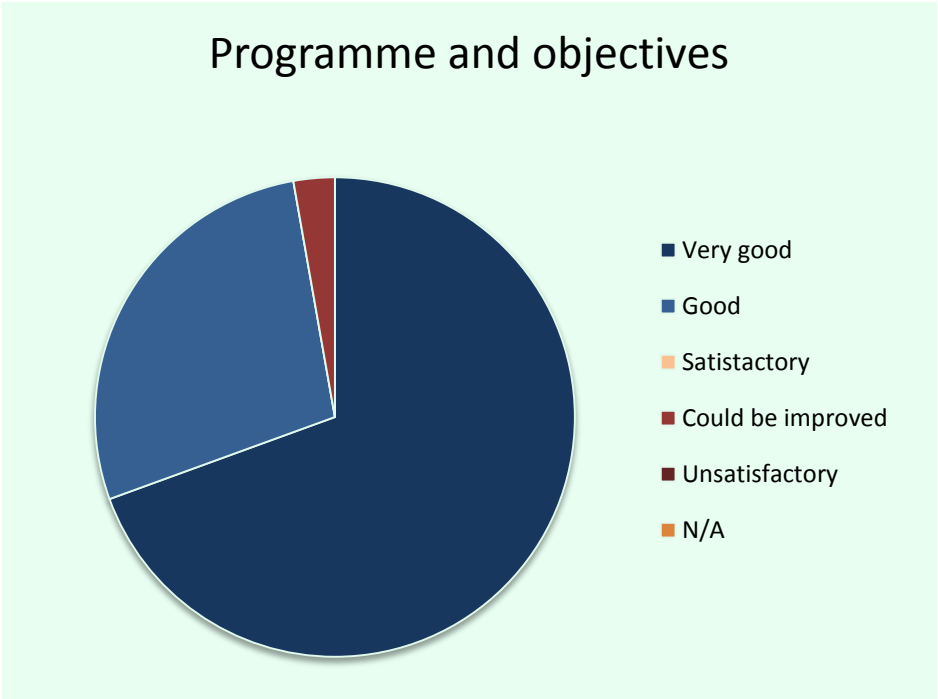
08 October 2014, JRC Ispra

Dear participant,

We hope that you enjoyed this workshop. As part of our own improvement efforts and in order to help us to assess how well this event met your expectations, please take a few minutes to fill out this feedback form. Many thanks for your contribution.

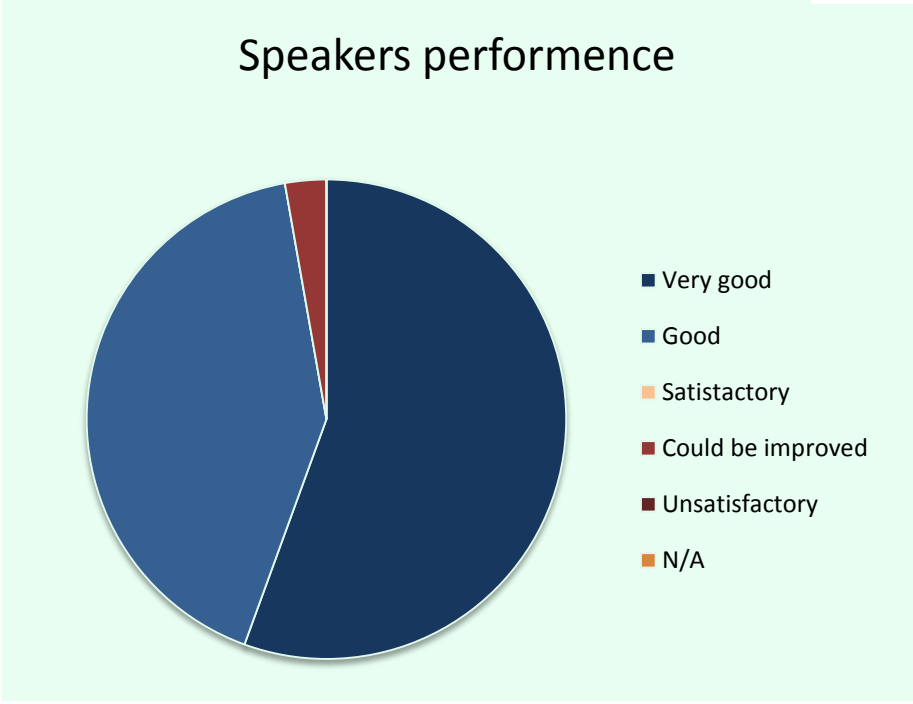
Event's contents	Very good	Good	Satisfactory	Could be improved	Unsatisfactory	N/A
Programme and objectives	☐	☐	☐	☐	☐	☐
Contents, quality of presentations	☐	☐	☐	☐	☐	☐
Speakers competence	☐	☐	☐	☐	☐	☐
Speakers performance	☐	☐	☐	☐	☐	☐
Balance between sessions	☐	☐	☐	☐	☐	☐
Interactions with speakers	☐	☐	☐	☐	☐	☐
Supporting material	☐	☐	☐	☐	☐	☐
Overall evaluation	☐	☐	☐	☐	☐	☐
Comments/Suggestions:						

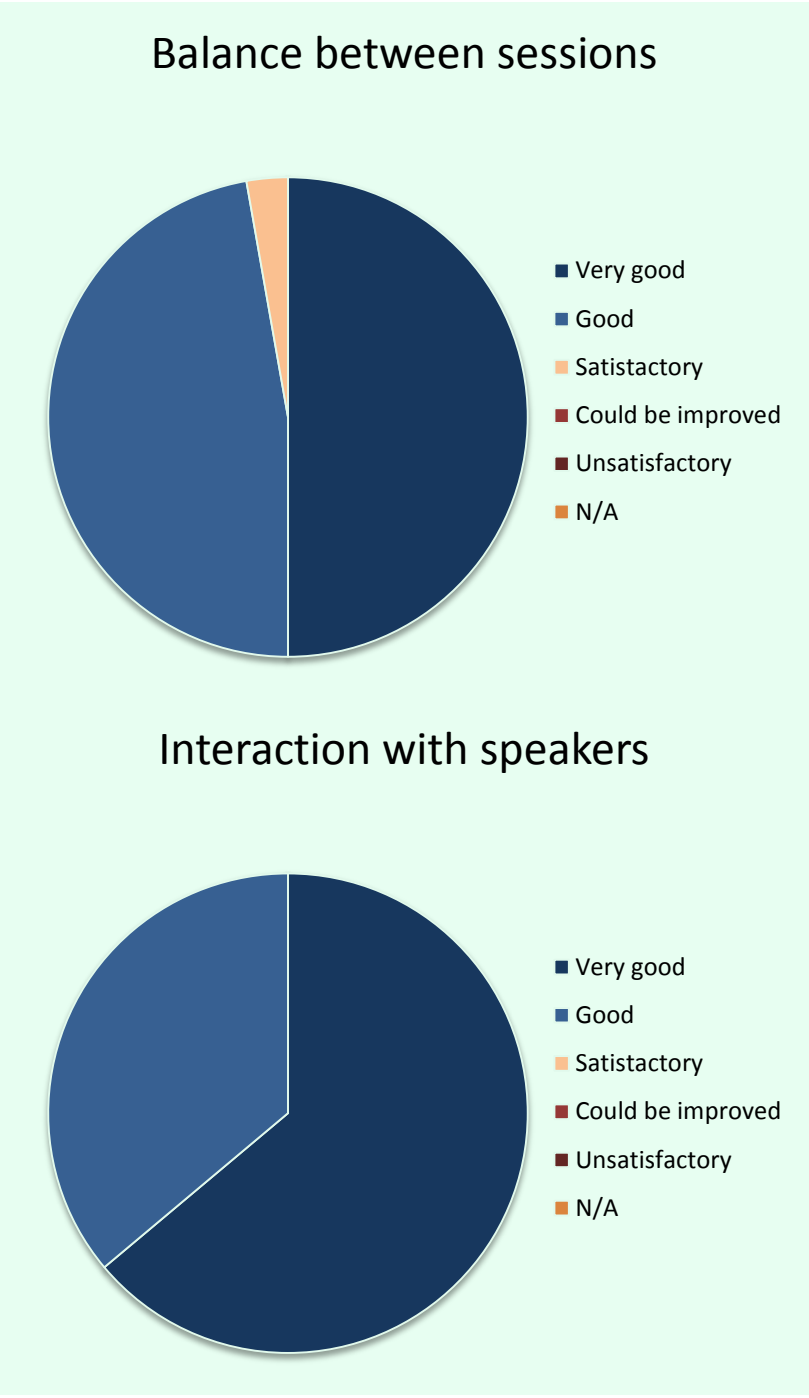
Organisation and Logistics	Very good	Good	Satisfactory	Could be improved	Unsatisfactory	N/A
Online registration	☐	☐	☐	☐	☐	☐
Transport and venue	☐	☐	☐	☐	☐	☐
Comments/Suggestions:						

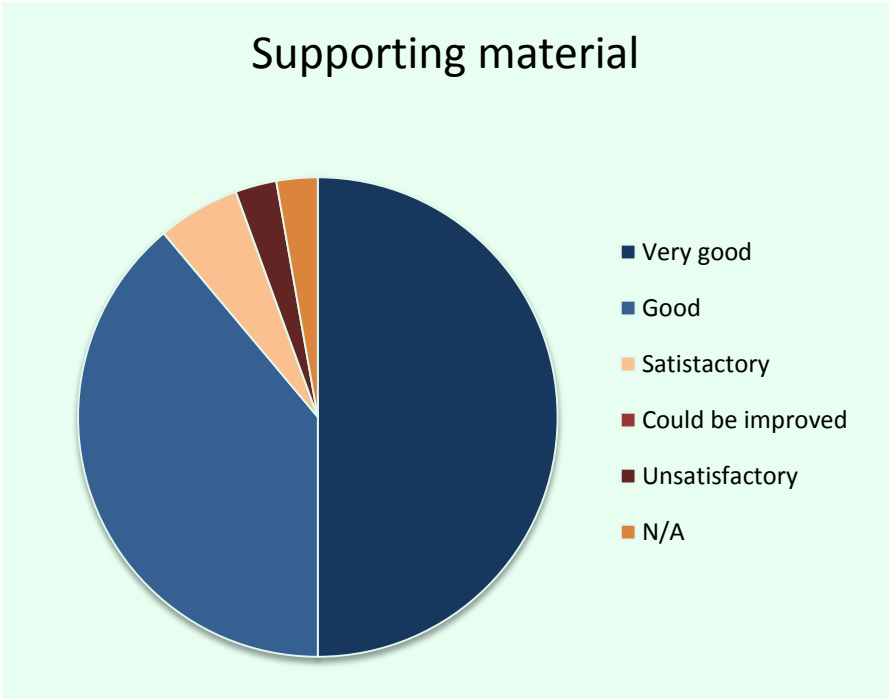

Programme and objectives
Very good
Good
Satistactory
Could be improved
Unsatisfactory
N/A

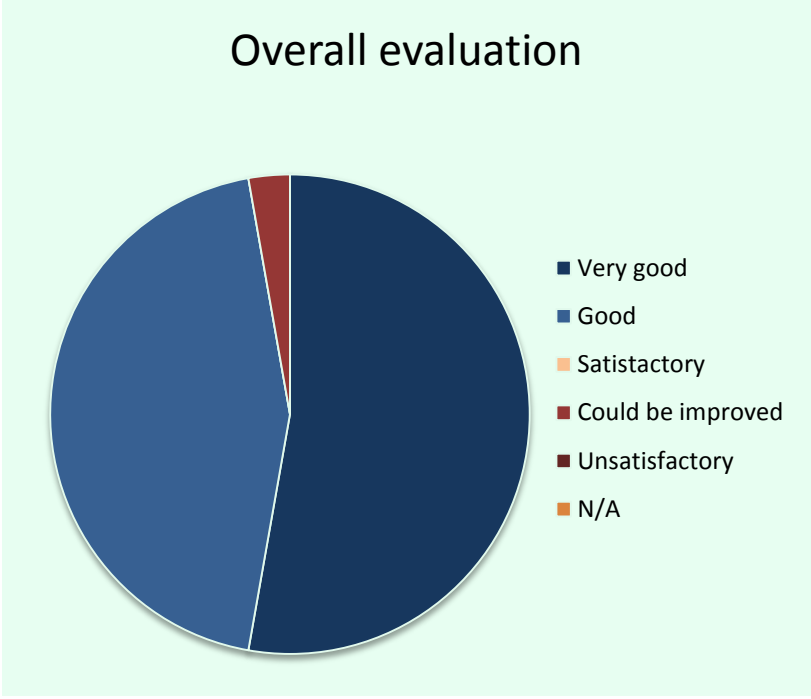

Contents, quality of presentation
Very good
Good
Satistactory
Could be improved
Unsatisfactory
N/A


Speakers competance
Very good
Good
Satistactory
Could be improved
Unsatisfactory
N/A

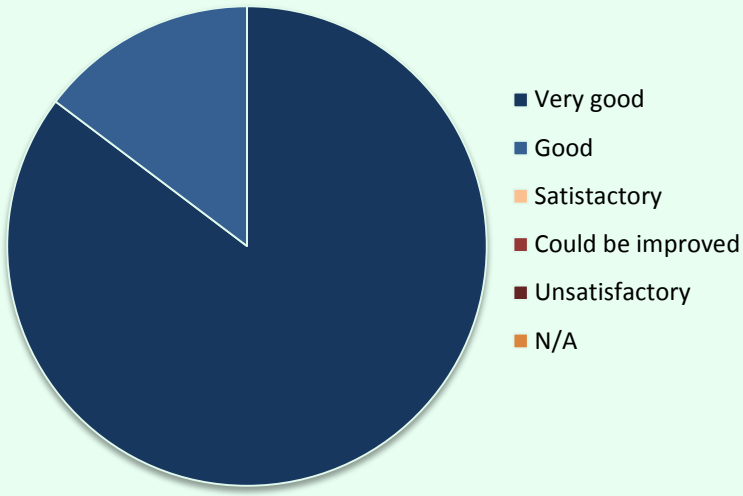

Speakers performence
Very good
Good
Satistactory
Could be improved
Unsatisfactory
N/A


Balance between sessions
Very good
Good
Satistactory
Could be improved
Unsatisfactory
N/A
Interaction with speakers
Very good
Good
Satistactory
Could be improved
Unsatisfactory
N/A

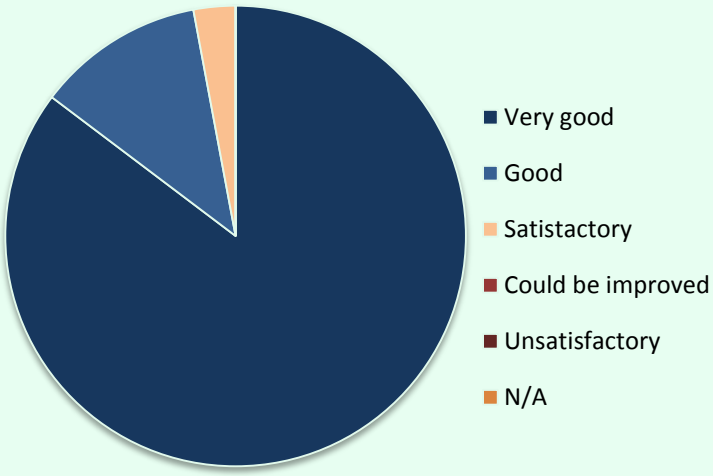

Supporting material
Very good
Good
Satistactory
Could be improved
Unsatisfactory
N/A


Overall evaluation
Very good
Good
Satistactory
Could be improved
Unsatisfactory
N/A

Online registration



Transport and venue



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