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Guidance document on fat reduction factor, functional barrier concept, phthalates and primary aromatic amines

Hoekstra, Eddo J.; Petersen, Jens Højslev; Bustos, Juana

Link to article, DOI:
[10.2788/24439](https://doi.org/10.2788/24439)

Publication date:
2011

Document Version
Publisher's PDF, also known as Version of record

[Link back to DTU Orbit](#)

Citation (APA):
Hoekstra, E. J., Petersen, J. H., & Bustos, J. (2011). Guidance document on fat reduction factor, functional barrier concept, phthalates and primary aromatic amines. Italy: European Commission. (JRC; No. 68007). (E U R; No. 25112). DOI: 10.2788/24439

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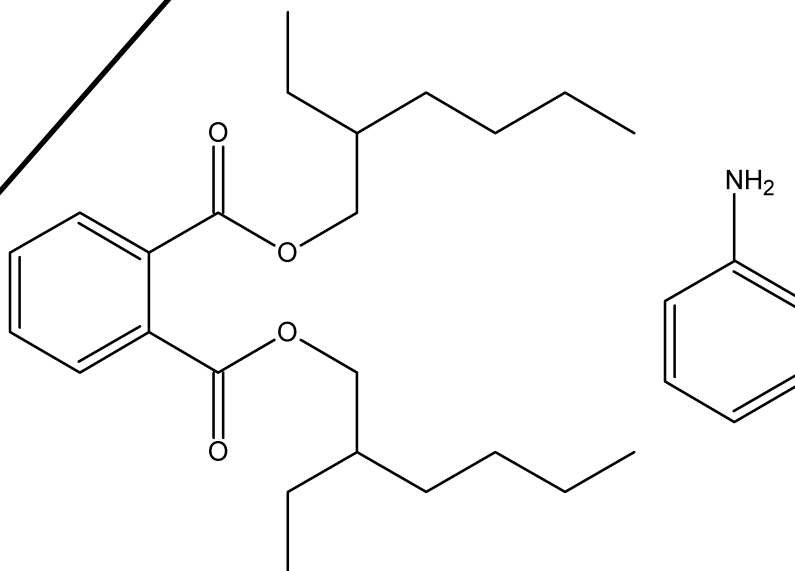
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Guidance document on fat reduction factor, functional barrier concept, phthalates and primary aromatic amines

Eddo J. Hoekstra, Jens H. Petersen and Juana Bustos



EUR 25112 EN - 2011

Guidance document on fat reduction factor, functional barrier concept, phthalates and primary aromatic amines

Eddo J. Hoekstra¹, Jens H. Petersen² and Juana Bustos³

1 Joint Research Centre of the European Commission

2 National Food Institute, Technical University of Denmark

3 National Centre for Food, Spanish Agency for Food Safety and Nutrition

The mission of the JRC-IHCP is to protect the interests and health of the consumer in the framework of EU legislation on chemicals, food, and consumer products by providing scientific and technical support including risk-benefit assessment and analysis of traceability.

Photo cover page: J.H. Petersen

European Commission
Joint Research Centre
Institute for Health and Consumer Protection

Contact information

Address: Via E. Fermi 2749, 21027 Ispra (VA), Italy

E-mail: eddo.hoekstra@jrc.ec.europa.eu

Tel.: +39-0332-785319

Fax: +39-0332-785707

http://ihcp.jrc.ec.europa.eu/our_labs/eurl_food_c_m

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JRC 68007

EUR 25112 EN

ISBN 978-92-79-22336-5

ISSN 1831-9424

doi:10.2788/24439

Luxembourg: Publications Office of the European Union

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Abstract

Commission Regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food contains four issues for which food inspectors and enforcement laboratories need further guidance. These issues are the concept of the fat reduction factor and the functional barrier, and the restrictions for certain phthalates and primary aromatic amines. The Regulation applies from 1 May 2011.

The network of the European Union Reference Laboratory and the National Reference Laboratories for food contact materials created a Task Force in order to give guidance on these issues.

Acknowledgment:

This work was performed under the EURL mandate:

SANCO/2008/foodsafety/047-food contact materials

SANCO/2008/foodsafety/055-food contact materials

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1. Introduction

The Regulation (EU) No 10/2011 on plastic materials and articles intended to come in contact with food (EU, 2011) contains five issues for which food inspectors and enforcement laboratories need further guidance. These issues are the concept of the fat reduction factor and the functional barrier, and the restrictions for certain phthalates and primary aromatic amines:

1. The migration of lipophilic substances, listed in column (7) of Table 1 in Annex I of the Regulation, into food with a fat content of more than 20% and into fatty food simulants (simulant D2) can be corrected under certain conditions with a Fat Reduction Factor (FRF). Chapter 4 of Annex V of the Regulation introduces the FRF as a correction. Guidance on the necessary calculations is given and a spreadsheet is developed in order to facilitate the calculations.
2. The Regulation specifies rules for plastic multi-layer materials or articles. One of these layers can act as a functional barrier for the migration of substances from the outer layers to food. Layers behind the plastic functional barrier may not comply with the restrictions and specifications as set in the Regulation or may be produced with substances other than those listed in the Regulation. The migration of non-listed substances behind the plastic functional barrier should be not detectable with a detection limit of 0.01 mg/kg food. The non-listed substances shall not be classified as proved or suspect carcinogenic, mutagenic or toxic to reproduction or substances in nanoform. Guidance is given to the food packaging industry, the food inspector and the enforcement laboratory.
3. The Regulation introduced rules for multi-material multi-layer materials and articles. The mayor difference with plastic multi-layer materials is that there are no restrictions to the migration of substances from the plastic layers both in contact with food and not in contact with food. Regulation of the migration of these substances is left for national legislation. Vinyl chloride, substances that are classified as proved or suspect carcinogenic, mutagenic or toxic to reproduction, and substances in nanoform are exceptions.
4. The Regulation lists five phthalates in Table 1 of Annex I. Each phthalate has its own restrictions and specifications. A table clarifying the critical parameters to control is given.
5. The Regulation specifies rules for the release of primary aromatic amines in Section 2 of Annex II. These guidelines show the differences in the procedures used to assess compliance compared to Commission Directive 2002/72/EC.

2. Fat Reduction Factor and other correction factors

The aim of this chapter is to give technical background information on the spreadsheet¹ that has been developed for the purpose of enforcement laboratories, for correcting the experimental specific migration of a substance from a plastic material into a food or food simulant for various correction factors before comparing them with the migration limit. The spreadsheet can also be useful for other stakeholders in plastic food contact materials. This chapter explains how the legislative texts are translated into the formulas used in the spreadsheet. This chapter is not applicable for substances behind a functional barrier or for substances migrating from a cap, gasket, stopper or similar sealing article, for which the intended use is unknown.

2.1. Legislation

The Regulation (EU) No 10/2011 contains three correction factors that should be applied to the experimental specific migration of a substance before the result is compared to the Specific Migration Limit (SML):

1. correction for the difference of surface-to-volume ratio between the experiment and the real food contact (Art. 17).
2. correction for the simulant D2 Reduction Factor (DRF) for defined foods (Section 4.2 of Annex V)
3. correction for the Fat Reduction Factor (FRF) for lipophilic substances migrating into food with a fat content of more than 20% (Section 4.1 of Annex V)

The correction factors were already introduced in the previous legislation of plastic food contact materials, Directive 2002/72/EC. The concept of the FRF remains unchanged in Regulation (EU) No 10/2011. Based on the fact that the FRF is a correction factor applied to migration of lipophilic substances it is evident that it only is relevant for correcting specific migration into food simulant D2, even though this is not explicitly mentioned in the Regulation.

The Directive explicitly stated that the FRF is not applied to substances for which the specific migration should be not detectable. Even though not explicitly stated, this concept is also applied in Regulation (EU) No 10/2011.

The Regulation differs from the Directive on the following aspects:

- The specific migration limit is always expressed in mg/kg food. The value of specific migration obtained when testing materials and articles that have a volume of less than 500 ml or more than 10 l or that are films or sheets now always has to be recalculated and expressed in mg/kg.
- The DRF can now be applied to specific migration of lipophilic substances from materials where the total mass of the substance migrated into food simulant D is higher than 80% of the mass of the substance in the finished materials or article. This aspect will be clarified in the coming Guidance to the Regulation.
- The Regulation no longer provides for the option previously mentioned in the Directive to check compliance of the specific migration of lipophilic substances from materials or articles, having a smaller volume than 500 ml or higher volume than 10 l or from films and sheets, expressed in either mg/kg or mg/dm².

¹ http://ihcp.jrc.ec.europa.eu/our_labs/eurl_food_c_m/publications/publications#technical-guidelines

2.2. Legislation in formula's

A flowchart (Figure 1) was developed for mapping the complexity of applying the three correction factors to the experimental specific migration values before checking compliance with the SML. The figure consists of three main parts. The first part (green) consists of several questions in order to evaluate whether the FRF can be applied or not. The second part (yellow) deals with all situations where the FRF is not applicable, whereas the third part (orange) deals with the situations in which the FRF may be applicable.

The experimental specific migration (M_{test}) is first corrected in case the surface-to-volume ratio in the test is different from the surface-to-volume ratio that will come in contact with real food ($M_{S/V} = M_{\text{test}} (S/V)_{\text{real}} / (S/V)_{\text{test}}$). The second question is whether experimental specific migration was obtained by testing with 10% aqueous ethanol (simulant A), 3% of aqueous acetic acid (simulant B), 20% of aqueous ethanol (simulant C) or 50% of aqueous ethanol (simulant D1) or poly(2,6-diphenyl-p-phenylene oxide) (simulant E). The FRF is not applicable to results obtained by those simulants. The third question is whether column (7) of Table 1 in Annex I indicates that the FRF is applicable for that substance. The fourth question concerns the fat content of the food that will be in contact with the material tested. The FRF may only be applied for foods with a fat content of more than 20%. The fifth question relates to the (intended) contact with food for infants (<12 month) and young children (1-3 years). The FRF is not applicable for contact with infant food and food for young children. The sixth question relates to materials and articles for which the surface-to-volume ratio is impractical to estimate. The FRF is not applicable to those materials.

If on the basis of the paragraph above it is concluded that the FRF is not applicable, then there are two main flows depending if the test was carried out with food or with food simulants (upper right part of Figure 1). This takes into account that the specific migration into food shall not exceed 60 mg/kg. It also takes into account that for simulant D2 the DRF may be applicable ($M_{\text{DRF}} = M_{S/V} / \text{DRF}$). The DRF cannot be applied for other food simulants or when the SML is "not detectable". For fillable articles having a volume in the range of 500 ml to 10 l, the M_{DRF} can directly be compared with the SML. For fillable articles outside this range or non-fillable articles such as sheets and films, or articles, for which the surface-to-volume ratio is impractical to estimate, a real surface-to-volume ratio of 6 dm²/kg food is assumed ($M = M_{\text{DRF}} * 6 / (S/V)_{\text{real}}$), except for articles that are intended for children. For substances for which the Regulation does not introduce a specific SML in column (8) or (9) of Table 1 of Annex I, the SML is 60 mg/kg.

If the initial questions in the green section suggest that the FRF may be applicable on the experimental specific migration, there are again two flows depending if the test was carried out with food or with food simulants (lower part of Figure 1). In the case of test results in food obtained from fillable articles in the range of 500 ml to 10 l, the first step is to check whether the surface-to-volume corrected specific migration ($M_{S/V}$) is below 60 mg/kg. If that is true then the FRF is calculated according to g fat/kg food/200 or %fat x 5/100: the value of the FRF is in the range of 1-5. The specific migration corrected for the FRF ($M_{\text{FRF}} = M_{S/V} / \text{FRF}$) should comply with the SML. In the case of testing with food non-fillable articles (films) and fillable articles smaller than 500 ml or larger than 10 l, a surface-to-volume ratio of 6 dm²/kg food is assumed ($M = M_{S/V} * 6 / (S/V)_{\text{real}}$).

If the initial questions in the green section suggest that the FRF may be applicable on the experimental specific migration and if food simulant D2 was used in the migration test, one selects the DRF, calculates the FRF as above and calculates the total reduction factor ($\text{TRF} = \text{DRF} * \text{FRF}$). If the TRF is above five, the TRF is set at five. The specific migration is corrected for the TRF ($M_{\text{TRF}} = M_{S/V} / \text{TRF}$). For fillable articles in the range of 500 ml to 10 l the specific migration corrected by the TRF is compared with the SML. For other materials or articles a surface-to-volume ratio of 6 dm²/kg food is assumed ($M = M_{\text{TRF}} * 6 / (S/V)_{\text{real}}$).

Example

The scheme of Figure 1 is illustrated with a concrete example. A polypropylene bottle of 510.4 ml (diameter = 10 cm; height = 6.5 cm; $S/V = 7.1 \text{ dm}^2/\text{kg}$; thickness = 2 mm; density = 0.91 kg/l; mass of polymer in contact = 0.066 kg; $c_p = 600 \text{ mg/kg}$) was tested for the release of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (Irganox 1076; CAS No. 2082-79-3; Ref. No. 68320; FRF applicable (Annex I, Table 1, column 7), SML = 6 mg/kg) for its application of packing liquid soup (08.03.B of Regulation) having a pH above 4.5. The concentration in the food simulant water was 0.52 mg/kg and in olive oil 16 mg/kg. Figure 2 and Figure 3 show the scheme for the test in water and olive oil respectively. If an olive oil producer would like to use this bottle (05.01 of Regulation) the situation is pictured in Figure 4. The producer of the polypropene bottles still has some sheets available with a concentration of Irganox 1076 of 1500 mg/kg. The migration is 33.5 mg/kg ($S/V = 6 \text{ dm}^2/\text{kg}$) and Figure 5 shows whether these sheets can be used for food contact with oily products or not.

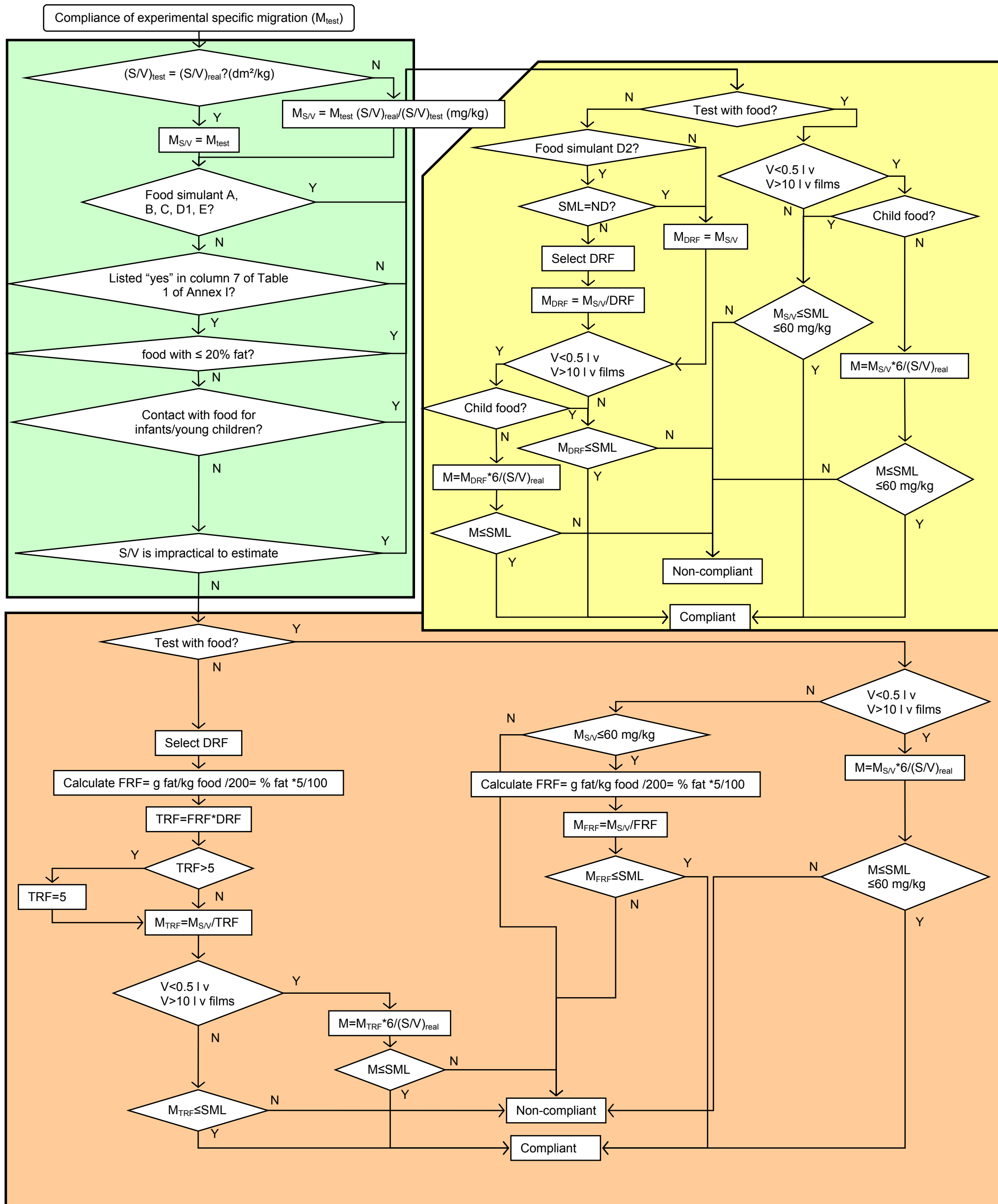
Figure 1 is the basis for the calculation spreadsheet that has been developed for the purpose of enforcement laboratories, for correcting the experimental specific migration of a substance from a plastic material into a food or food simulant for various correction factors.

The [spreadsheet](#)

- calculates the maximum acceptable concentration in the test based on the SML of the substance, dimensions of article, type of food and test conditions as defined by the input of the user. This is useful for test design.
- calculates the corrected experimental specific migration that should be compared with the SML.
- checks whether the article is in compliance or not.

The formula's used in the spreadsheet are reported in the Annex – formula's used in the spreadsheet.

Figure 1 Decision flowchart for compliance check of an experimentally obtained specific migration value according to the correction factors applicable in FCM Regulation on plastics (No 10/2011)



DRF, simulant D2 reduction factor; FRF, fat reduction factor; M_{test} , specific migration determined in test; (mg/kg); $M_{S/V}$, specific migration corrected for Annex I 2 (not 2a and 2 b.); M_{TRF} , migration corrected for S/V-ratio and TRF; M_{DRF} , migration corrected for S/V-ratio and DRF; M_{FRF} , migration corrected for S/V-ratio and FRF; M specific migration (mg/kg); ND, not detectable; S, contact surface (dm²); $(S/V)_{real}$, surface-to-volume ratio of food in contact with material/article; $(S/V)_{test}$, surface-to-volume ratio of food simulant in test; SML, specific migration limit (mg/kg); TRF, total reduction factor (DRF * FRF ≤ 5); V, volume of article (litre).

Figure 2 migration of irganox 1076 from a 510 ml polypropene bottle into water (liquid soup)

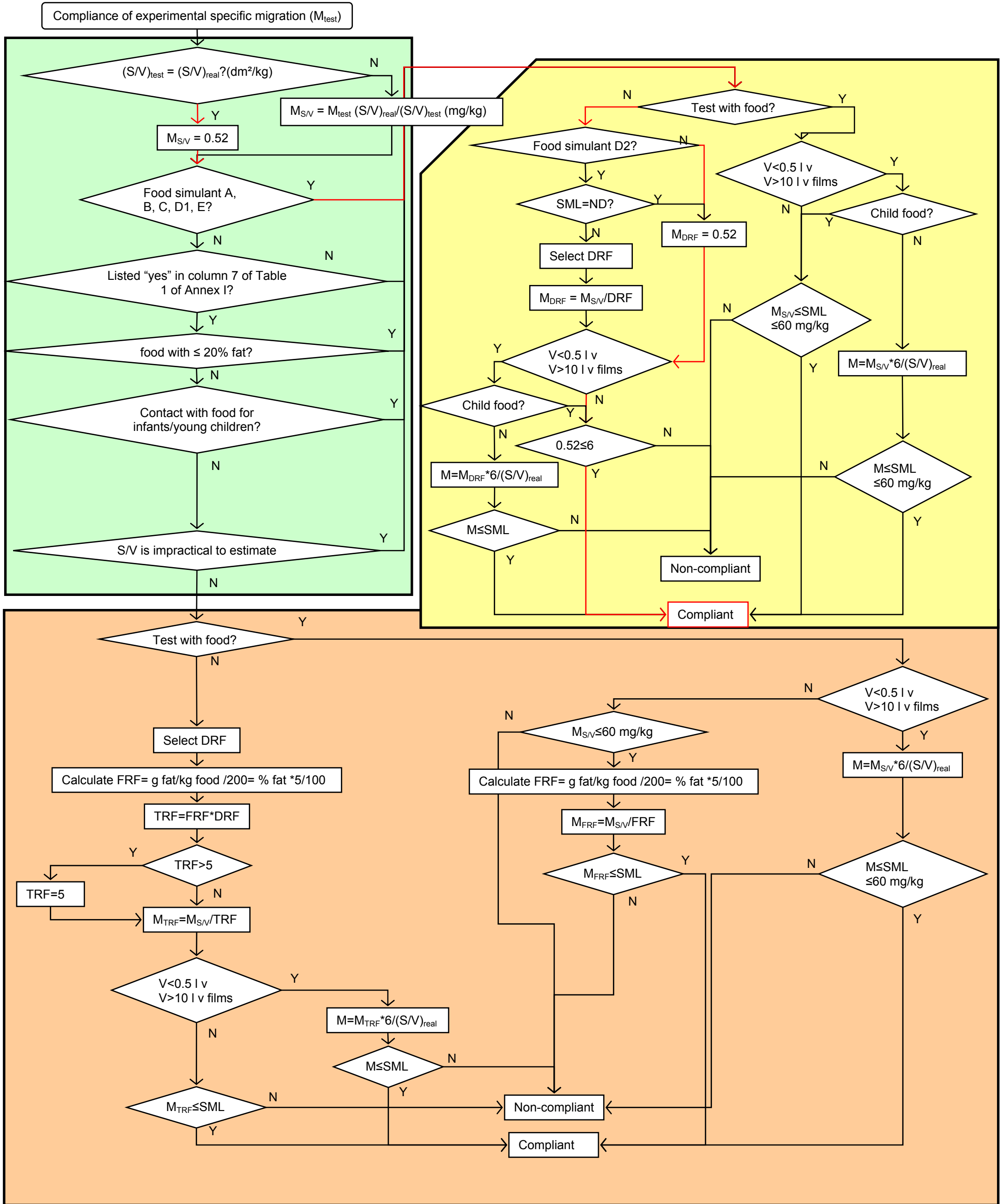


Figure 3 migration of irganox 1076 from a 510 ml polypropene bottle into olive oil (liquid soup)

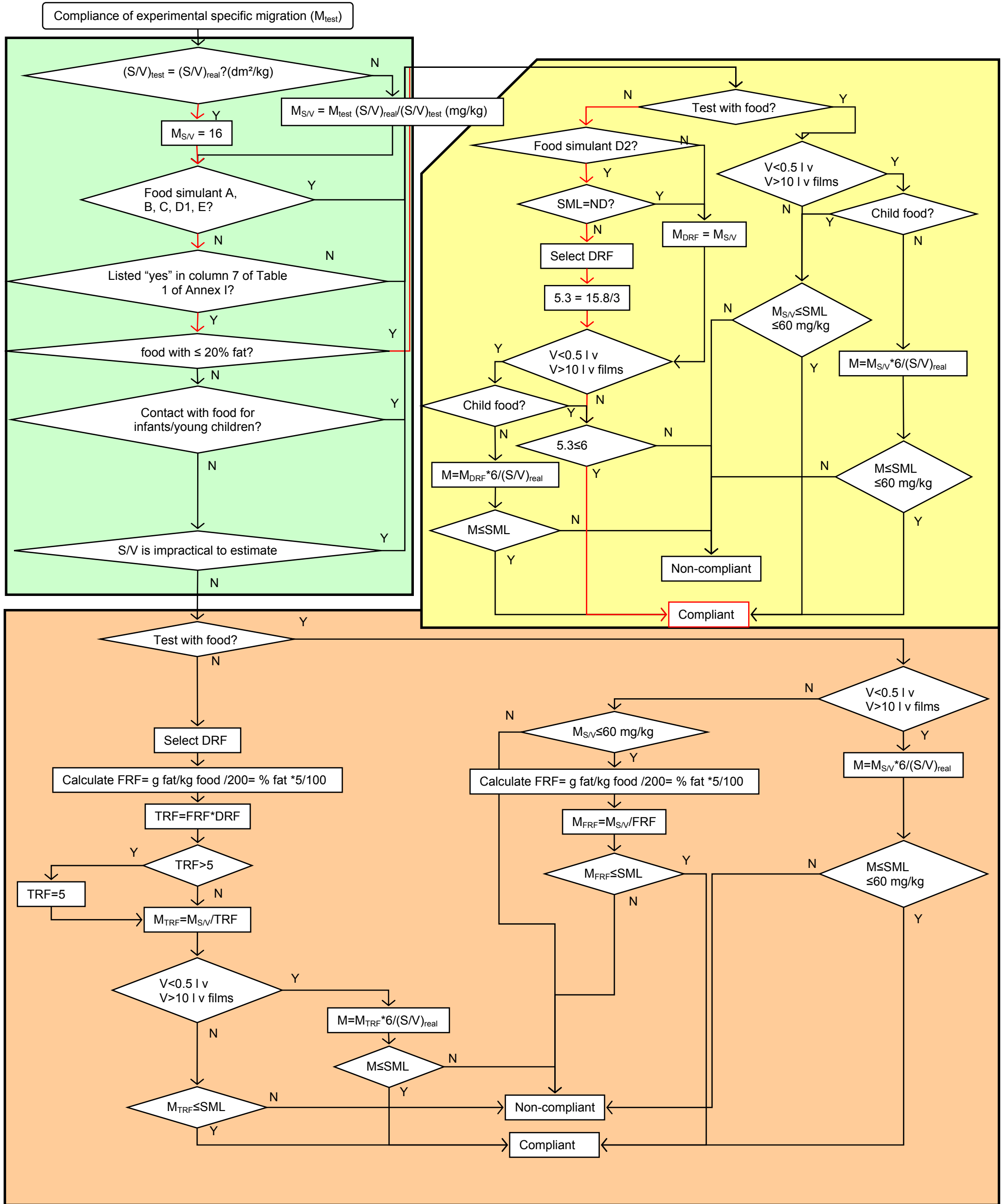


Figure 4 migration of irganox 1076 from a 510 ml polypropene bottle into olive oil (olive oil)

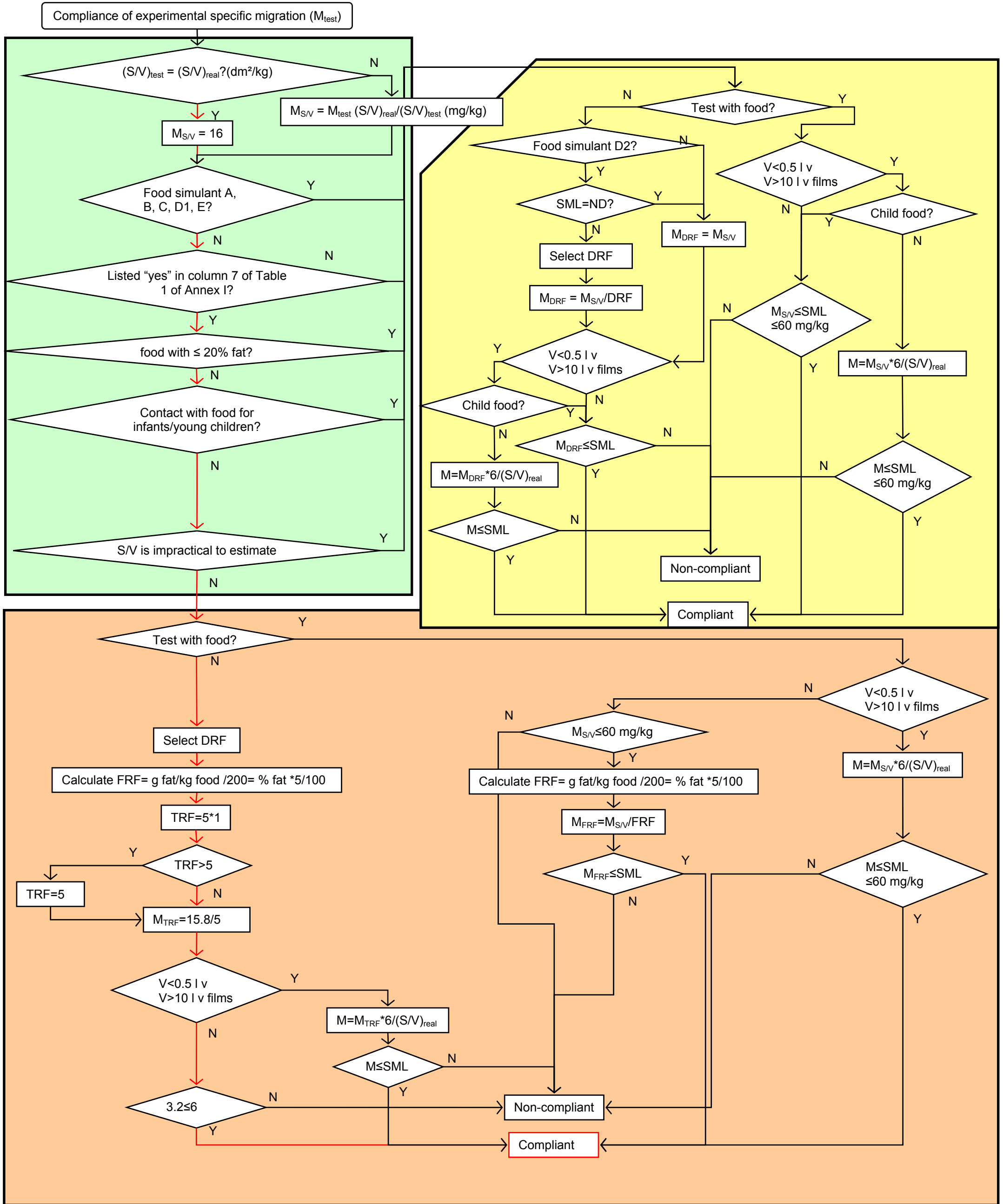
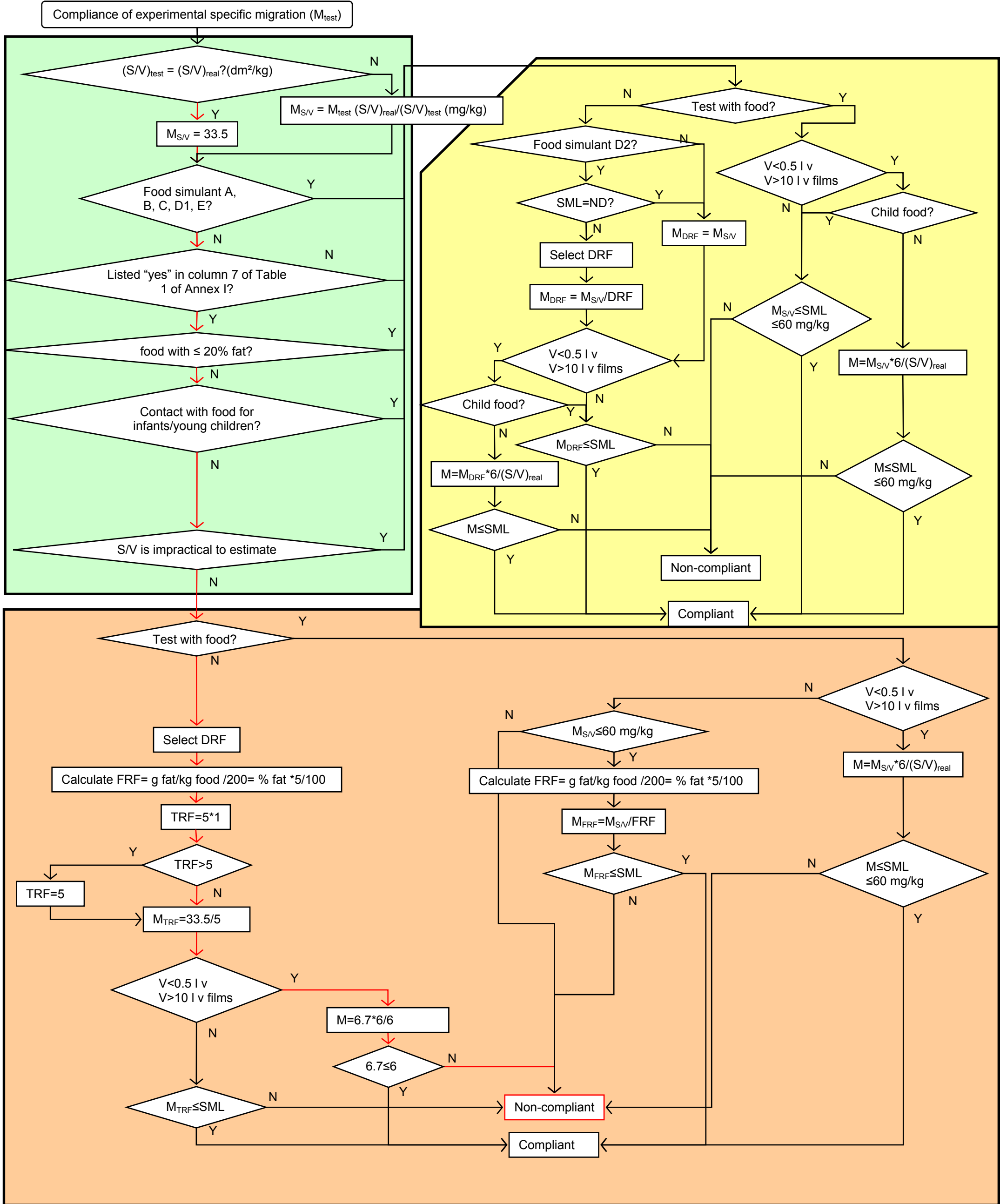


Figure 5 migration of irganox 1076 from polypropene sheet for olive oil application (olive oil)



3. Functional barrier

3.1. Introduction

Section 3.2 is a short guidance document detailing the requirements for the producer, guiding the food inspector and describing the role of the enforcement laboratory in relation to functional barriers. It should be read in conjunction with Art. 13 and 14 of Regulation (EU) No 10/2011. The requirements mentioned are of relevance when a producer of a multilayer plastic material claims that the final product contains a plastic functional barrier, which ensures that the migration from behind this barrier of any non-authorised substance found in the material is not detectable with a detection limit of 0.01 mg/kg food. This guidance is also relevant when a producer of a multi-material multi-layer material claims that their functional barrier prevents migration of non-authorised substances.

Box 1 Legislation citations on functional barriers

Legislative text of Art. 13 of Regulation (EC) No 10/2011 on plastic functional barriers includes one major addition concerning nanoparticles compared to Directive 2002/72/EC:

1. In a plastic multi-layer material or article, the composition of each plastic layer shall comply with this Regulation.
2. By derogation from paragraph 1, a plastic layer which is not in direct contact with food and is separated from the food by a functional barrier, may:
 - (a) not comply with the restrictions and specifications set out in this Regulation except for vinyl chloride monomer as provided in Annex I; and/or
 - (b) be manufactured with substances not listed in the Union list or in the provisional list.
3. The migration of the substances under paragraph 2(b) into food or food simulant shall not be detectable measured with statistical certainty by a method of analysis set out in Article 11 of Regulation (EC) No 882/2004 with a limit of detection of 0.01 mg/kg. That limit shall always be expressed as concentration in foods or food simulants. That limit shall apply to a group of compounds, if they are structurally and toxicologically related, in particular isomers or compounds with the same relevant functional group, and shall include possible set-off transfer.
4. The substances not listed in the Union list or provisional list referred to in paragraph 2(b) shall not belong to either of the following categories:
 - (a) substances classified as 'mutagenic', 'carcinogenic' or 'toxic to reproduction' in accordance with the criteria set out in sections 3.5, 3.6. and 3.7 of Annex I to Regulation (EC) No 1272/2008 of the European Parliament and the Council
 - (b) substances in nanoform.
5. The final plastic multi-layer material or article shall comply with the specific migration limits set out in Article 11 and the overall migration limit set out in Article 12 of this Regulation.

Legislative text of Art. 14 of Regulation (EC) No 10/2011 on multi-material multi-layer materials is new; the concept of plastic functional barrier is extended to non-plastic functional barriers in multi-material multi-layers. The main difference with Art. 13 is that restrictions to the migration of substances (except Art 14.3 and 14.5) from the plastic layer both in contact with food and not in contact with food (Art. 14.4) remain covered by national law:

1. In a multi-material multi-layer material or article, the composition of each plastic layer shall comply with this Regulation.
2. By derogation from paragraph 1, in a multi-material multi-layer material or article a plastic layer which is not in direct contact with food and is separated from the food by a functional barrier, may be manufactured with substances not listed in the Union list or the provisional list.
3. The substances not listed in the Union list or provisional list referred to in paragraph 2 shall not belong to either of the following categories:
 - (a) substances classified as 'mutagenic', 'carcinogenic' or 'toxic to reproduction' in accordance with the criteria set out in sections 3.5, 3.6. and 3.7 of Annex I to Regulation (EC) No 1272/2008;
 - (b) substances in nanoform.
4. By derogation from paragraph 1, Articles 11 and 12 of this Regulation do not apply to plastic layers in multi-material multi-layer materials and articles.
5. The plastic layers in a multi-material multi-layer material or article shall always comply with the restrictions for vinyl chloride monomer laid down in Annex I to this Regulation.
6. In a multi-material multi-layer material or article, specific and overall migration limits for plastic layers and for the final material or article may be established by national law.

3.2. EURL-NRL Guidance on functional barrier-containing materials as defined in the plastics legislation

3.2.1. Requirements to the producer, converter and importer

The producer/converter/importer claiming the use of a **plastic** functional barrier in plastic materials and articles must know the identity of the barrier layer. Its efficiency in reducing the migration of unauthorised substances must be demonstrated in documentation supporting the Declaration of Compliance. This means that:

- Each business operator in the chain of the production of a materials or article that come into contact with food must ensure that he receives from his supplier of his starting material or article a Declaration of Compliance. The supplier issuing the Declaration of compliance is obliged to keep supporting documentation and make it available to enforcement authorities on request.
- Each business operator in the chain of the production of a material or article that come into contact with food must issue a Declaration of Compliance for his products on the basis of his in-house supporting documentation. This means that the business operator should know the identity of each monomer and additive that he used in his plastic multilayer material or product and that he should have in house supporting documentation including the compliance declarations from his suppliers that can be made available to the food inspection. He should provide to his customer all information that is necessary to ensure the compliance of his (final) product.
- The identity of those substances which are **not** authorised and **not** in the EU positive list must be known². Sufficient information and documentation must show that these substances are **not** classified as
 - 'mutagenic', 'carcinogenic' or 'toxic to reproduction'³.
 - Substances in nanoform⁴

² Article 13.2.b and 14.2 (EU/10/2011)

³ Article 13.4(a) and 14.3(a) (EU/10/2011)

⁴ Article 13.4(b) and 14.3(b) (EU/10/2011)

- Migration of each non-authorized monomer, starting substance or additive in a plastic layer behind a **plastic** functional barrier must not be detectable with a detection limit of 0.01 mg/kg food or food simulant. Compliance can be verified using a tiered approach: firstly by simple calculations of the complete migration of the substance or by “worst-case” testing with solvents; secondly by migration into food/food simulants. In all migration experiments, a certain lag-time for migration of substances from outer layers must be taken in consideration.
 - for substances that are structurally and toxicologically related the limit applies to the sum of concentrations.
 - If an authorised substance is present in the above mentioned layers the Specific Migration Limit will apply.
- For some production processes of multi-layers, e.g. during co-extrusion, the temperature is high and substances from the different layers may diffuse into the barrier layer to a different extent than during use. The business operator should have knowledge to what extent this diffusion takes place.
- If a substance, not authorised in plastics, migrates in detectable amounts from a surface coating, a layer of epoxy resin, an adhesive or printing ink, the manufacturer should be able to provide evidence that the substance is not used as monomer/other starting substance or additive in the plastic layers. National legislations may govern substances of these layers and the general requirements of the framework Regulation on food contact materials⁵ apply.

For multi-material multilayer materials only a limited specific harmonised regulation is in place⁶. When the presence of a functional barrier is claimed then plastic layers behind this functional barrier may contain non-authorized monomers, starting substances or additives with exception of those that are mutagenic, carcinogenic, toxic to reproduction or are in nanoform. When plastic layers in multi-material multi layers contain residual vinyl-chloride monomer (VCM), the amount in the plastic layer should be below 1 mg/kg in any plastic layers. The migration of VCM should not be detectable.

3.2.2. Guidance for the food inspector

The efficiency of the **plastic** functional barrier in reducing the migration of unauthorised substances should be investigated. The food inspector checks the in-house control system at the producer/converter/importer and inspects the Declaration of Compliance eventually including supporting documentation. The food inspector is advised to clarify in advance if the inspection is supported by analytical expertise from an enforcement laboratory in order to choose between document control alone or utilising the possibility of taking samples for analytical control.

Questions to be addressed for all plastic multilayer materials/articles:

- Control if reliable supporting documentation in section 3.2.1 (Requirements to the producer, converter and importer) is available:
 - If available, check the “fit for use” of the plastic functional barrier according to the criteria summarised in section 3.2.1 and conclude if plastic functional barrier is efficient or not. If yes, the material is compliant.
 - If documentation is insufficient or not satisfactory, collect as much as possible of relevant information. When backed up by laboratory assistance, collect samples for testing. If not, order the manufacturer to provide relevant documentation before a given deadline.
 - If documentation is not available, the material/article is deemed not to be compliant.

⁵ Article 3 of Regulation (EC) No 1935/2004

⁶ Article 14 (EU/10/2011),

Questions to be addressed when the layers behind the plastic functional barrier contain recycled plastic materials:

- Until the date of establishment of an EU list of authorised recycling processes: Is the recycling process of the recycled plastic material or the material itself authorised at national level?
- As from the date of establishment of an EU list of authorised recycling processes: Is the recycling process included in the EU list of authorised recycling processes?
 - If yes, this layer should be treated like any other plastic material for food contact.
 - If not, the following questions are relevant:
- Do any documents indicate that the composition of the recycled material is in control?

Questions to be addressed in case a multi-material multilayer material claimed to have a functional barrier contain any plastic layers:

- Could any of the layers contain residual vinyl chloride monomer, e.g. if one of the layers are PVC-based?
- Is it likely that the functional barrier is capable of reducing the migration of any unauthorised monomer, starting substance or additive in the plastic layers to a level, that brings the material in compliance with the framework Regulation⁷ or any relevant national legislation.

3.2.3. Role of the enforcement laboratory

Inspection and evaluation of supporting documentation collected by the food inspection. Ask for up-stream compliance supporting documentation if needed.

In case of plastic multilayer materials:

- Is it likely that the claimed functional barrier layer is suitable for its purpose?
 - Yes: stop
 - No: go for analysis
- Conduct tests on samples collected:
 - Verify identity of the material used for the barrier layer
 - Screening test with solvents and determination of residual concentration in the material. Diffusion modelling could be applied. Test or modelling is followed by:
 - Acceptance of the material or
 - Formal testing with official food simulants or foodstuffs

For multilayer materials care must be taken, as far as possible, to follow generally accepted test procedures.

⁷ Article 3 of Regulation (EC) No 1935/2004

4. Phthalates

In order to facilitate the work of the enforcement authorities concerning the restrictions of phthalates, Table 1 has been developed specifying the parameters, i.e. specific migration limit or maximum permitted quantity of the residual substance in the material relevant to these substances.

Box 2 Legislation citations on phthalates

Regulation (EC) No 10/2011 provides a set of restrictions and specifications for the use of a number of phthalates in food contact materials (Table 1 of Annex I):

Phthalic acid, benzyl butyl ester (FCM substance no 159; ref. no. 74560; CAS no. 000085-68-7) to be used only as:

- (a) plasticizer in repeated use materials and articles;
- (b) plasticizer in single-use materials and articles contacting non-fatty foods except for infant formulae and follow-on formulae as defined by Directive 2006/141/EC) and processed cereal-based foods and baby foods for infants and young children as defined by Directive 2006/125/EC;
- (c) technical support agent in concentrations up to 0.1% in the final product.

SML = 30 mg/kg food simulant.

Phthalic acid, bis (2-ethylhexyl) ester (FCM substance no 283; ref. no. 74640; CAS no. 000117-81-7) to be used only as:

- (a) plasticizer in repeated use materials and articles contacting non-fatty foods;
- (b) technical support agent in concentrations up to 0.1% in the final product.

SML = 1.5 mg/kg food simulant.

Phthalic acid, dibutyl ester (FCM substance no 157; ref. no. 74880; CAS no. 000084-74-2) to be used only as:

- (a) plasticizer in repeated use materials and articles contacting non-fatty foods;
- (b) technical support agent in polyolefines in concentrations up to 0.05% in the final product.

SML = 0.3 mg/kg food simulant.

Phthalic acid, diesters with primary, saturated C8-C10 branched alcohols, more than 60% C9 (FCM substance no 728; ref. no. 75100; CAS no. 068515-48-0 and 028553-12-0) to be used only as:

- (a) plasticizer in repeated use materials and articles;
- (b) plasticizer in single-use materials and articles contacting non-fatty foods except for infant formulae and follow-on formulae as defined by Directive 2006/141/EC) and processed cereal-based foods and baby foods for infants and young children as defined by Directive 2006/125/EC;
- (c) technical support agent in concentrations up to 0.1% in the final product.

SML(T) = 9 mg/kg food simulant (sum of FCM substance no. 728 and 729.).

Phthalic acid, diesters with primary, saturated C9-C11 alcohols more than 90% C10 (FCM substance no 729; ref no. 75105; CAS no. 068515-49-1 and 026761-40-0) to be used only as:

- (a) plasticizer in repeated use materials and articles;
- (b) plasticizer in single-use materials and articles contacting non-fatty foods except for infant formulae and follow-on formulae as defined by Directive 2006/141/EC) and processed cereal-based foods and baby foods for infants and young children as defined by Directive 2006/125/EC;
- (c) technical support agent in concentrations up to 0.1% in the final product.

SML(T) = 9 mg/kg food simulant (sum of FCM substance no. [728](#) and [729](#)).

Table 1 Clarification of the critical parameter of “classical” phthalates for control in enforcement work

Ref. no.	Substance	SML	QM	Parameter to control in <i>single use</i> Food Contact Material			Parameter to control in <i>repeated use</i> Food Contact Material			Limit in fatty food simulant [@]	
				Fatty food	Infant food	Non-fatty food	Fatty food	Non-fatty food	Infant food (non-fatty)		
159 74560	Phthalic acid, benzyl butyl ester (BBP)	30	0.1	QM		SML	SML			30-150	
283 74640	Phthalic acid, bis(2-ethylhexyl) ester (DEHP)	1.5	0.1	QM			QM	SML			n.r.
157 74880	Phthalic acid, dibutyl ester (DBP)	0.3	0.05 [#]	QM			QM	SML			n.r.
728 75100	Phthalic acid, diester with C8-C10 (DiNP)	9 ^{\$}	0.1	QM		SML	SML			9-45	
729 75105	Phthalic acid, diester with C9-C11 (DiDP)	9 ^{\$}	0.1	QM		SML	SML			9-45	

* Packaging made from glasses with lid containing a plasticized gasket is usually considered as a single use material; [@] Taking the simulant D reduction factor into consideration. When simulant D is 50% ethanol, no reduction factor is of relevance; [#] only permitted in polyolefins; ^{\$} SML(T) is sum of DiNP and DiDP

n.r., not relevant; QM, maximum permitted quantity of the residual substance in the material; SML, specific migration limit

5. Primary Aromatic Amines

Since 2002, Primary Aromatic Amines (PAA) shall not be released from food contact materials into food into a detectable quantity (Directive 2002/72/EC and Annex II.2. of Regulation (EC) No 10/2011). With the fourth amendment of the Directive the detection limit was no longer expressed with analytical tolerance included, the procedure of the summation of the concentrations changed and the way the measurement uncertainty was included, was changed.

Box 3 Legislation citations on primary aromatic amines

The original version of Directive 2002/72/EC states:

The material and article manufactured by using aromatic isocyanates or colorants prepared by diazo-coupling, shall not release primary aromatic amines (expressed as aniline) in a detectable quantity (DL = 0.02 mg/kg of food or food simulant, analytical tolerance included). However, the migration value of the primary aromatic amines listed in this Directive are excluded from this restriction.

The Regulation (EC) No 10/2011 states:

Plastic materials and articles shall not release primary aromatic amines, excluding those appearing in Table 1 of Annex I, in a detectable quantity into food or food simulant. The detection limit is 0,01 mg of substance per kg of food or food simulant. The detection limit applies to the sum of primary aromatic amines released.

The detection limit in the original Directive 2002/72/EC was 0.02 mg/kg expressed as aniline equivalent (eq.), meaning that an analytical result plus the analytical uncertainty should be below this limit before the sample could be deemed compliant.

The new detection limit has been established at 0.01 mg/kg, what in practice means that an analytical result minus the analytical uncertainty must be above 0.01 mg/kg before the sample can be deemed non-compliant. Furthermore, the analytical result should be expressed as the sum of concentrations of the individual PAA and it is not required any more to convert the concentration of each PAA to aniline equivalents. Consequently, PAA other than aniline will contribute relatively more to the final result because of their higher molecular weight.

It should be noted that PAA listed in Table 1 of Annex I of the Regulation remain excluded from the restriction of Annex II.2. and shall continue to be checked individually against their specific migration limit.

An example illustrates the new specifications:

In a migration test you find 0.005 mg/kg of aniline and 0.010 mg/kg of 4,4'-methylenedianiline (MDA) and each substance was determined with a standard measurement uncertainty (u) of 0.001 mg/kg. The expanded uncertainty (U) assuming a approximate 95% confidence interval (conventional covering factor, $k=2$) is calculated from the standard measurement uncertainty (Bratinova *et al.*, 2009):

$$U = k \sqrt{u_{ANL}^2 + u_{4,4'MDA}^2}$$

The lower limit of the 95% confidence interval of the result is calculated as:

- 0.005 mg/kg of aniline
- + 0.010 mg/kg of MDA
- the expanded uncertainty of the sum of both PAA calculated as:

$$U = 2 \sqrt{0.001^2 + 0.001^2} = 0.0028 \text{ mg/kg PAA}$$

- = 0.012 mg/kg of PAA to be compared with the legislative limit of 0.01 mg/kg of PAA. In this case, the sample must be deemed non-compliant.

6. References

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EC. 2002. Commission Directive 2002/72/EC of 6 August 2002 relating to plastic materials and articles intended to come into contact with foodstuffs (and its amendments). Official Journal of the European Communities. L220:18-58. Available from:

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=CELEX:32002L0072:EN:NOT>

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<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=CELEX:32007L0019:EN:NOT>

EU, 2011. Commission Regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food. Official Journal of the European Union. L12:1-89. Available from:

<http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=CELEX:32011R0010:EN:NOT>

7. Annex – formula's used in the spreadsheet

FRF = IF("FRF applicable?" = "yes", (IF(%fat*5/100<=1, 1, %fat*5/100)), 1)

TRF = IF(DRF*FRF>=5, 5, DRF*FRF)

C_{test,max} (mg/kg) = IF(AND(FRF="yes", "child food"="yes"), "FRF not for child food", IF(OR("food simulant"="A", "food simulant"="B", "food simulant"="C", "food simulant"="D1", "food simulant"="E"), IF("V<500 or V>10000"="yes", IF("child food"="yes", SML*(S/V)_{TEST}/(S/V)_{REAL}, SML*(S/V)_{TEST}/6), SML*(S/V)_{TEST}/(S/V)_{REAL}), IF(FRF="no", IF("food simulant"="D2", IF("V<500 or V>10000"="yes", IF("child food"="yes", IF("ND"="yes", SML*(S/V)_{TEST}/(S/V)_{REAL}, SML*(S/V)_{TEST}/6*DRF), IF("ND"="yes", SML*(S/V)_{TEST}/(S/V)_{REAL}*DRF), IF("V<500 or V>10000"="yes", IF("child food"="yes", IF(SML*(S/V)_{TEST}/(S/V)_{REAL}>60, 60, SML*(S/V)_{TEST}/(S/V)_{REAL}), IF(SML*(S/V)_{TEST}/6>60, 60, SML*(S/V)_{TEST}/6)), IF(SML*(S/V)_{TEST}/(S/V)_{REAL}>60, 60, SML*(S/V)_{TEST}/(S/V)_{REAL}))), IF("food simulant"="D2", IF("V<500 or V>10000"="yes", SML*(S/V)_{TEST}/6*TRF, SML*(S/V)_{TEST}/(S/V)_{REAL}*TRF), IF("V<500 or V>10000"="yes", IF(SML*(S/V)_{TEST}/6>60, 60, SML*(S/V)_{TEST}/6), IF(SML*(S/V)_{TEST}/(S/V)_{REAL}<=60, SML*(S/V)_{TEST}/(S/V)_{REAL}*FRF, 60)))))) (see Figure 6)

C_{test,max} (mg/dm²) = IF(AND(FRF="yes", "child food"="yes"), "FRF not for child food", IF(OR("food simulant"="A", "food simulant"="B", "food simulant"="C", "food simulant"="D1", "food simulant"="E"), IF("V<500 or V>10000"="yes", IF("child food"="yes", SML/(S/V)_{REAL}, SML/(S/V)_{REAL}), IF(FRF="no", IF("food simulant"="D2", IF("V<500 or V>10000"="yes", IF("child food"="yes", IF("ND"="yes", SML/(S/V)_{REAL}, SML/(S/V)_{REAL}*DRF), IF("ND"="yes", SML/6, SML/6*DRF), IF("ND"="yes", SML/(S/V)_{REAL}, SML/(S/V)_{REAL}*DRF)), IF("V<500 or V>10000"="yes", IF("child food"="yes", IF(SML*(S/V)_{TEST}/(S/V)_{REAL}>60, 60/(S/V)_{TEST}, SML/(S/V)_{REAL}), IF(SML*(S/V)_{TEST}/6>60, 60/(S/V)_{TEST}, SML/6), IF(SML*(S/V)_{TEST}/(S/V)_{REAL}>60, 60/(S/V)_{TEST}, SML/(S/V)_{REAL}))), IF("food simulant"="D2", IF("V<500 or V>10000"="yes", SML/6*TRF, SML/(S/V)_{REAL}*TRF), IF("V<500 or V>10000"="yes", IF(SML*(S/V)_{TEST}/6>60, 60/(S/V)_{TEST}, SML/6, IF(SML*(S/V)_{TEST}/(S/V)_{REAL}<=60, SML/(S/V)_{REAL}*FRF, 60/C30)))))) (see Figure 7)

C_{test,corrected} (mg/kg) = IF(AND(C34>0, C35>0), "fill in only one test result, IF(C34>0, IF(AND(FRF="yes", "child food"="yes"), "FRF not for child food", IF(OR("food simulant"="A", "food simulant"="B", "food simulant"="C", "food simulant"="D1", "food simulant"="E"), IF("V<500 or V>10000"="yes", IF("child food"="yes", C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}, C_{test}(mg/kg)*6/(S/V)_{TEST}), C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}), IF(FRF="no", IF("food simulant"="D2", IF("V<500 or V>10000"="yes", IF("child food"="yes", IF("ND"="yes", C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}, C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}/DRF), IF("ND"="yes", C_{test}(mg/kg)*6/(S/V)_{TEST}, C_{test}(mg/kg)*6/(S/V)_{TEST}/DRF)), IF("ND"="yes", C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}, C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}/DRF)), IF("V<500 or V>10000"="yes", IF("child food"="yes", C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}, C_{test}(mg/kg)*6/(S/V)_{TEST}), C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}), IF("food simulant"="D2", IF("V<500 or V>10000"="yes", C_{test}(mg/kg)*6/(S/V)_{TEST}/TRF, C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}/TRF), IF("V<500 or V>10000"="yes", C_{test}(mg/kg)*6/(S/V)_{TEST}, IF(C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}<=60, C_{test}(mg/kg)*(S/V)_{REAL}/(S/V)_{TEST}/FRF, "not compliant")))), IF(AND(FRF="yes", "child food"="yes"), "FRF not for child food", IF(OR("food simulant"="A", "food simulant"="B", "food simulant"="C", "food simulant"="D1", "food simulant"="E"), IF("V<500 or V>10000"="yes", IF("child food"="yes", C_{test}(mg/dm²)*(S/V)_{REAL}, C_{test}(mg/dm²)*6), C_{test}(mg/dm²)*(S/V)_{REAL}), IF(FRF="no", IF("food simulant"="D2", IF("V<500 or V>10000"="yes", IF("child food"="yes", IF("ND"="yes", C_{test}(mg/dm²)*(S/V)_{REAL}, C_{test}(mg/dm²)*(S/V)_{REAL}/DRF), IF("ND"="yes", C_{test}(mg/dm²)*6/DRF), IF("ND"="yes", C_{test}(mg/dm²)*(S/V)_{REAL}, C_{test}(mg/dm²)*(S/V)_{REAL}/DRF)), IF("V<500 or V>10000"="yes", IF("child food"="yes", C_{test}(mg/dm²)*(S/V)_{REAL}, C_{test}(mg/dm²)*6), C_{test}(mg/dm²)*(S/V)_{REAL}), IF("child food"="D2", IF("V<500 or V>10000"="yes", C_{test}(mg/dm²)*6/TRF, C_{test}(mg/dm²)*(S/V)_{REAL}/TRF), IF("V<500 or V>10000"="yes", C_{test}(mg/dm²)*6, IF(C_{test}(mg/dm²)*(S/V)_{REAL}<=60, C_{test}(mg/dm²)*(S/V)_{REAL}/FRF, "not compliant")))))))) (see Figure 8 and Figure 9)

compliance = IF(OR(C_{test,corrected}=0, SML=0), "no", IF(C_{test,corrected}>SML, "no", "yes"))

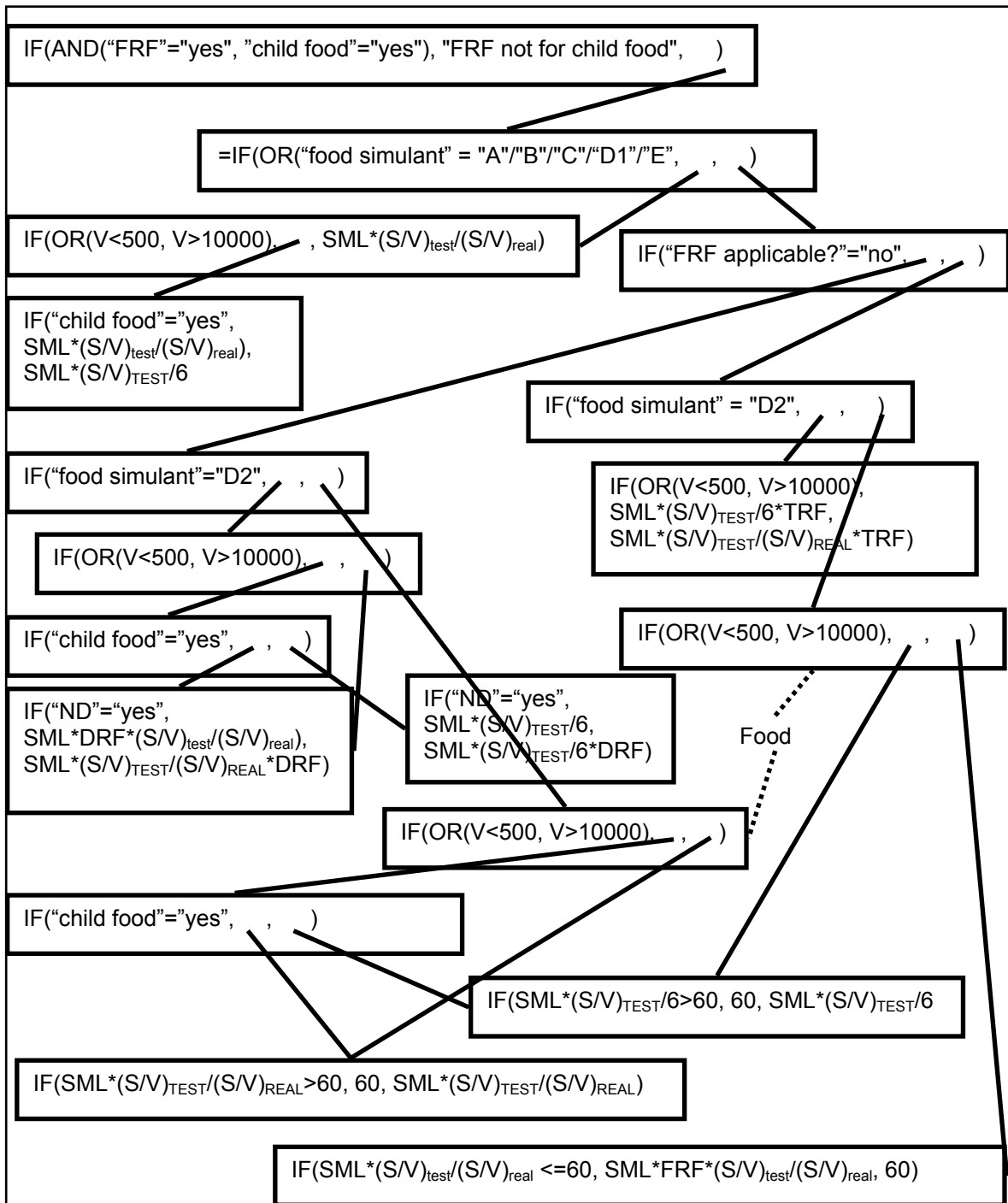


Figure 6 Calculation scheme of the maximum acceptable specific migration (mg/kg) in the test

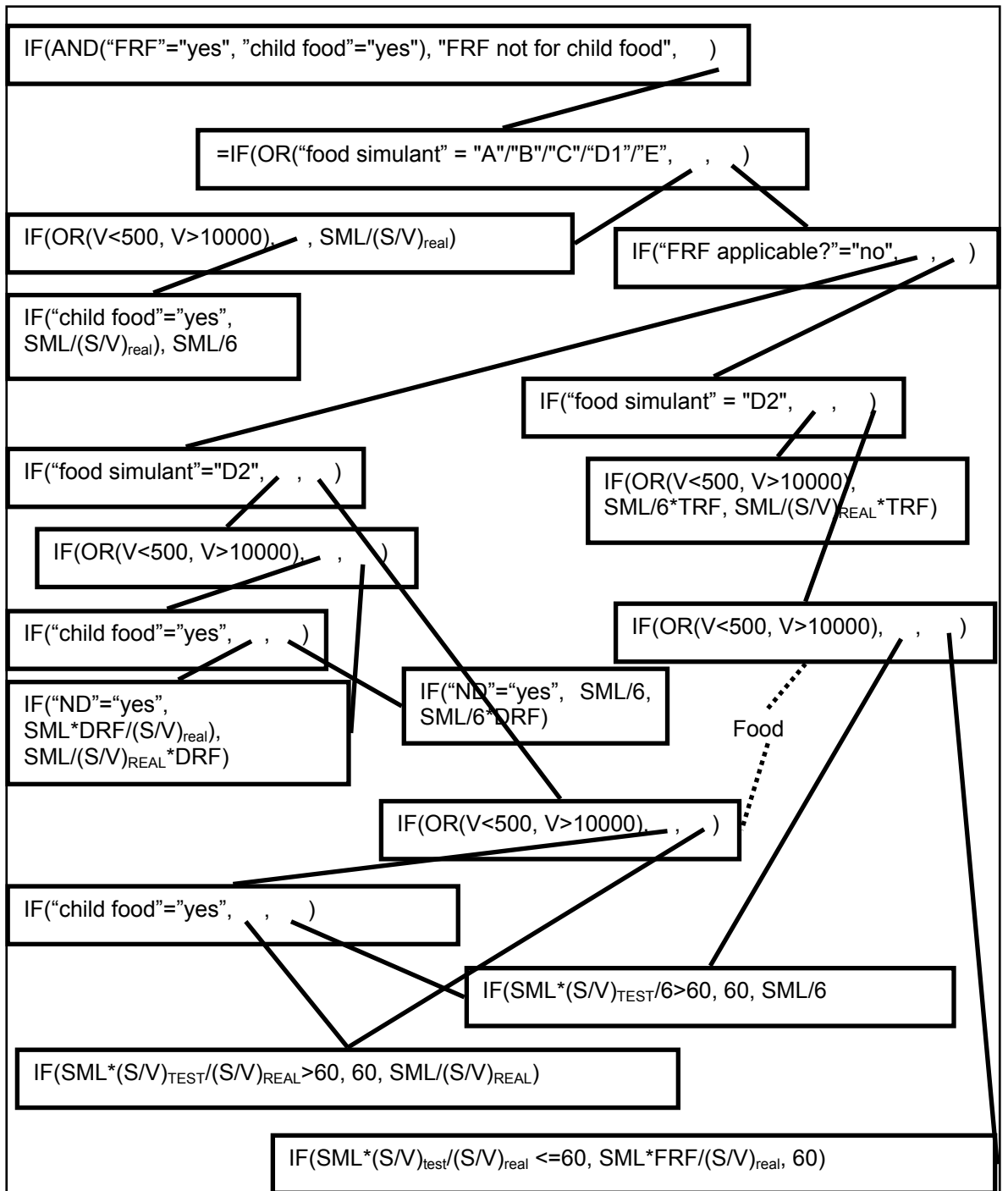


Figure 7 Calculation scheme of the maximum acceptable specific migration (mg/dm²) in the test

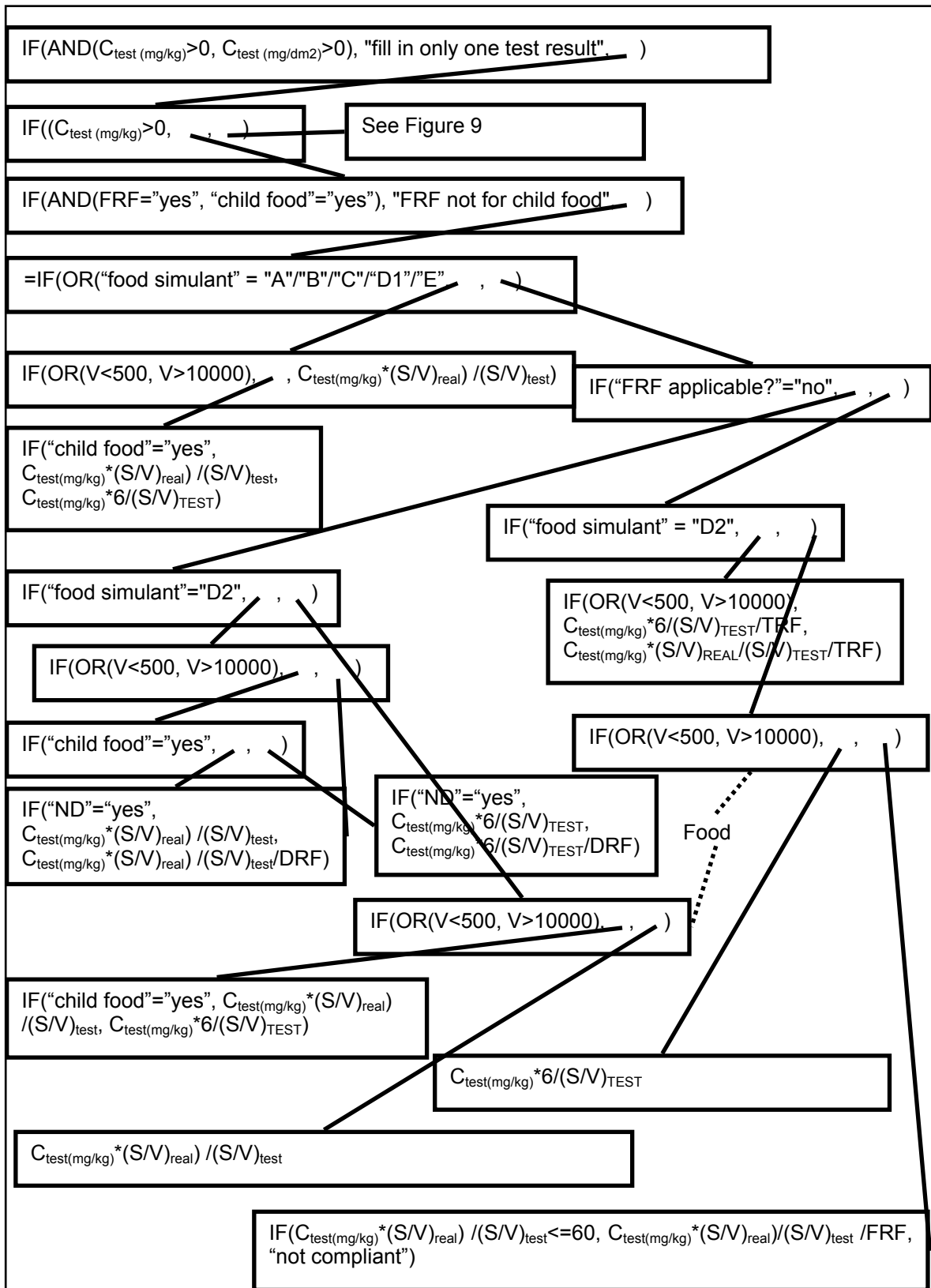


Figure 8 Calculation scheme of the corrected experimental specific migration (mg/kg)

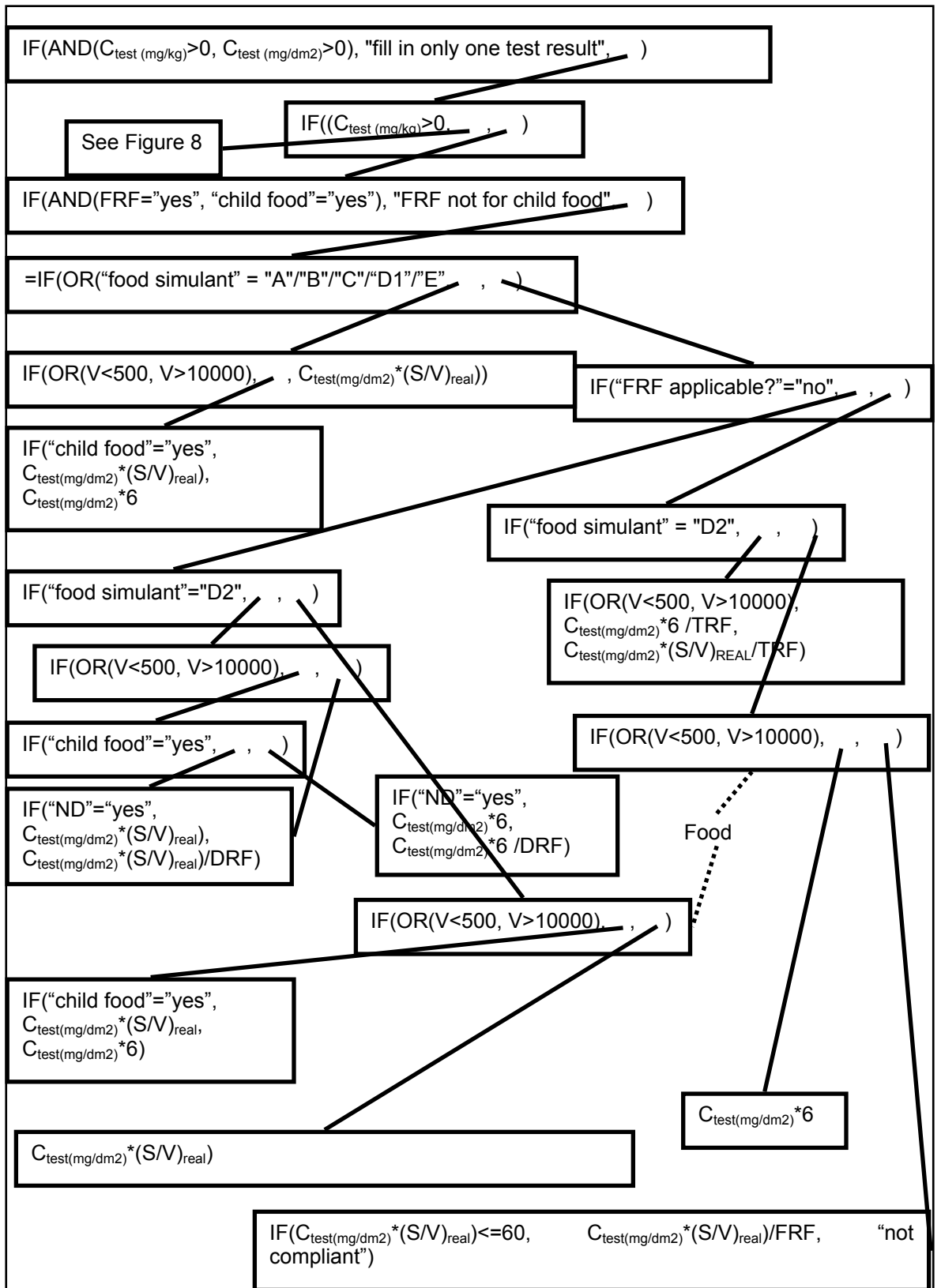


Figure 9 Calculation scheme of the corrected experimental specific migration (mg/kg)

European Commission

EUR 25112 EN – Joint Research Centre – Institute for Health and Consumer Protection

Title: Guidance document on fat reduction factor, functional barrier concept, phthalates and primary aromatic amines

Author(s): E.J. Hoekstra, J.H. Petersen, J. Bustos

Luxembourg: Publications Office of the European Union

2011 – 36 pp. – 29.7 x 21 cm

EUR – Scientific and Technical Research series – ISSN 1831-9424 (online), ISSN 1018-5593 (print)

ISBN 978-92-79-22336-5

doi:10.2788/24439

Abstract

Commission Regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food contains four issues for which food inspectors and enforcement laboratories need further guidance. These issues are the concept of the fat reduction factor and the functional barrier, and the restrictions for certain phthalates and primary aromatic amines. The Regulation applies from 1 May 2011.

The network of the European Union Reference Laboratory and the National Reference Laboratories for food contact materials created a Task Force in order to give guidance on these issues.

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LB-NA-25112-EN-N



ISBN 978-92-79-22336-5

