TSC34 MIGRATION TESTING GUIDELINES

FOR RIGID METAL PACKAGING COATED WITH ORGANIC COATINGS INTENDED FOR DIRECT FOOD CONTACT

VERSION 6.3

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CONTENTS

1. SUMMARY

MIGRATION TESTING CONDITIONS FOR RIGID METAL PACKAGING COATED WITH ORGANIC COATINGS– SUMMARY AND CONCLUSIONS

These guidelines will be revised as and when necessary.

The guidelines have been prepared as part of an industry initiative to propose appropriate simulants and conditions for testing migration from rigid metal packaging coated with organic coatings to demonstrate compliance with Regulation (EC) No. 1935/2004 (1), in particular article 3. Industry believes that these guidelines are workable for all members of the supply chain. These guidelines only refer to demonstrating compliance of rigid metal packaging coated with organic coatings.

These guidelines have been formulated to help industry demonstrate compliance with (EC) 1935/2004, and it should not be assumed that they are suited to demonstrating non-compliance. If non-compliance has to be demonstrated e.g. by enforcement authorities, only the testing of the real food packed and stored under commercial conditions is appropriate.

These guidelines were developed by members of T-JIG[1] in response to the introduction to the draft JRC migration guidelines (2) which state that non-plastic food contact materials are not included in the scope and that individual guidelines will developed by professional bodies.

Although out of scope of the JRC migration guidelines, many of the principles have been adopted.

OVERALL MIGRATION

For overall migration the proposed simulants for all food stuffs, i.e. acidic, aqueous, alcoholic and fatty are 10% ethanol (1 hour @ 130°C), 95% Ethanol (3 hours @ 60°C) and isooctane (6 days @ 60°C). These represent all of the worst foreseeable conditions, in which no physical or other changes take place in the test specimen, for all food types. 95% ethanol and isooctane can be used instead of vegetable oil for the overall migration of fatty foods.

SPECIFIC MIGRATION

The simulants in the technical guidelines are appropriate with the exception of simulant D1 - 50% Ethanol, using the conditions in the guidelines (4.2.5.1 table 2) where alternatively 10% and 95% Ethanol can be used unless there are physical or other changes to the organic coating. Extractive solvents are a viable alternative to demonstrate compliance.

For products sterilised above 130°C the Arrhenius equation can be used as a practical means to allow compliance testing to take place using standard equipment. Alternatively, the actual conditions of processing can be used.

[1] EMPAC, CEPE, FDE, APEAL, EAA, CEFIC-FCA, PlasticsEurope

2. SCOPE

Rigid metal packaging for all types of foodstuffs encompasses the following articles which can be made from aluminium, electrolytic tinplate (ETP), tin-free steel (TFS) or black plate (3):

- Cans
  - Two piece cans
    - Single drawn
    - Drawn and redrawn (DRD)
    - Drawn and wall-ironed (DWI)
    - Bottle
  - Three-piece cans
- Can Ends
  - Classic Ends
  - Easy Open Ends
  - Peel Off Ends
  - Penny Lever Ends (PAL)
- Drums and Pails
- Aerosols
- Tubes
- Trays and foils
- Closures
  - Crowns
  - Vacuum closures
  - Bottle closures (Al)
- Slip Lids

In the majority of cases, but not all, the article is coated with organic coatings, either fully or partially. The scope of this document is to propose test conditions suited to direct food contact coatings on any of the above articles. In practice this is the food contact side of the article, irrespective of how many different coatings or layers of coating are applied to it.

The testing conditions and simulants specified in JRC Migration Testing Guidelines are not applicable to non-plastics as clearly stated in the introduction and in some cases are unsuitable for rigid metal packaging coated with organic coatings. For example, some of the simulants have chemical properties which are different from real foodstuffs and not relevant when they are used in contact with plastics but which may interfere with the migration test when applied to rigid metal packaging coated with organic coatings. The testing temperatures and times can be higher and longer than the packaging will ever be subjected to in its expected lifetime. This can lead to artificially high overall or specific migration results or damage caused to the packaging in a way which would not happen to commercially sold packaging.

Therefore, the migration testing recommended in this guideline more closely simulates the action of real foodstuffs with the coating components of actual commercially filled rigid metal packaging coated with organic coatings than the conditions and simulants specified in JRC Migration Testing Guidelines for plastic FCM's. This guidance defines appropriate

simulants and test conditions for overall and specific migration testing to allow reproducible, relevant and practicable testing of rigid metal packaging coated with organic coatings. This will allow the sector to adequately demonstrate that the correctly applied and cured coatings are suitable for contact with foodstuffs.

These guidelines are for demonstrating compliance of the organic food contact coatings on rigid light metal packaging, and are not intended to assess metal release from the packaging substrate. In order for enforcement authorities to demonstrate non-compliance it is necessary to test real foods packed and stored under commercial conditions

The proposal of the testing parameters for rigid metal packaging coated with organic coatings also allows the sector to have more confidence that the migration testing at each stage of the supply chain will adequately model the performance of the product in its end use, and ensure the safety of their current products and help with development of new products.

3. DEFINITIONS

Definitions specific to metal packaging

ORGANIC COATINGS FOR RIGID METAL PACKAGING, PAILS AND DRUMS

With few exceptions organic coatings for rigid metal packaging are transformed into the food contact layer at elevated temperatures. Most organic coatings for rigid metal packaging are thermoset in nature, although some thermoplastic ones are used. Organic coatings can be applied to the fabricated rigid metal packaging article or most likely at some stage during the manufacture of the rigid metal packaging article. Organic coatings are mainly prepared from one or more mainly organic substances (raw materials), which in their finished state do not form a self-supporting layer or film, but when applied onto a substrate form a partial or an integral layer, which has certain intended technological effects in the material or article. Organic coatings are typically applied in a liquid (solution or dispersion) or powder state and need to dry, cure or solidify to reach their finished state. Inorganic coatings largely based on inorganic materials including metals and oxides are excluded from this definition. Further details are available in the ILSI Monograph on Metal Packaging for Foodstuffs (3).

APPLIED COATING

The dried film formed from the organic coating once it has been applied to the substrate and dried, cured or solidified. This includes pigmented organic coatings. Metal surface treatments are currently out of the scope of this document.

METAL

For rigid metal packaging, the metals used are aluminium, tin-free steel (TFS) electrolytic tinplate (ETP) and black plate.

4. MATERIAL SPECIFIC PROPERTIES TO BE CONSIDERED WHEN TESTING THIS CLASS OF FOOD CONTACT MATERIAL

OVERVIEW

For rigid metal packaging coated with organic coatings, it is found that certain simulants are more severe than the foodstuffs they represent. Simulant B, 3% acetic acid, causes corrosion of the metal substrate, which significantly interferes with overall migration testing. As this degree of metal corrosion is not seen in commercial packaging containing acidic food then the sole use of 3% acetic acid does not accurately simulate the food, primarily due to the fact that the presence of oxygen cannot be excluded when testing with simulants and natural buffering effects of some foodstuffs are not reproduced. Compared to actual food, simulant D1, 50% Ethanol, for the simulation of dairy foods may cause physical changes to metal packaging coated with organic coatings which may give false results.

A key principle of the JRC Migration Testing Guidelines is that testing should not cause physical or other changes in the test specimen and recommends that migration tests shall be carried out under the worst foreseeable conditions of use in which these physical or other changes do not take place. This principle requires that appropriate simulants and testing conditions are established for the specific food contact material and application.

Furthermore, JRC Migration Testing Guidelines gives a generally accepted principle that a test can always be replaced by another test which is at least as severe and, based on scientific arguments, in specific cases testing may be reduced to a single food simulant which is known to be the most severe for that particular substance and/or material.

Another key principle established in JRC Migration Testing Guideline is that in cases where the actual most extreme conditions of processing FCM's are generally known and are strictly controlled (for microbiological reasons) the actual conditions can be used. This principle should apply to organic coatings for rigid metal packaging rather than using the OM bands in JRC Migration Testing Guidelines and confirmed in Regulation (EU) No. 2016/1416 (4), which are for more general cases and only applicable for plastic FCM's.

Similarly, the temperature and time conditions chosen for migration testing need to take account of the limitations of the testing equipment used. Standard equipment used operates with a maximum temperature of 130 °C. Given this limitation, it should be possible to use the Arrhenius conversion recommended in JRC Migration Testing Guideline to conduct the tests at 130 °C with an appropriate adjustment of the duration of the test to match the actual process conditions.

The contributors to this guideline believe that the migration testing conditions in JRC Migration Testing Guidelines may be appropriate to demonstrate compliance but they are not appropriate for demonstrating non- compliance. There are a number of real cases that can be generalised. Some examples follow:

a) ALCOHOLIC BEVERAGES

It is very important to take into account that the simulants and time/temperature bands set out in JRC Migration Testing Guidelines are not always appropriate for metal coated with organic coatings. For example, alcoholic beverages are often pasteurised in the packaging. The processing conditions are typically around 30 minutes at ~ 70°C. In practice, for production reasons, conditions could easily slightly exceed this time and temperature. Under the conditions in JRC Migration Testing Guidelines this would require testing for 1 hour at 100°C with simulant C (20% ethanol/water). The test time and temperature alone would (according to the Arrhenius equation) be 8 times more severe than the real conditions and with a simulant having typically 4 times the ethanol concentration, huge overestimation of migration is likely. This is not a theoretical example.

Testing for a specified monomer in canned beverages gives less than 10% of the value compared to testing for the same monomer, in pasteurised beverages, using simulant and conditions according to JRC Migration Testing Guidelines. Whilst it is reasonable that testing with simulants should not underestimate real migration, it has to be realistic, which for metal packaging coated with organic coatings will not always be the case if JRC Migration Testing Guidelines conditions are applied.

b) CLOUDY FRUIT-BASED SOFT DRINKS

Cloudy fruit-based drinks are sometimes pasteurised in the same way as alcoholic beverages, so a similar issue will arise. However this is exacerbated by the use of simulant D1 (50% ethanol/ water) which will give even higher migration results and in some cases lead to destruction of the organic coating under simulation conditions. Again using the same monomer for comparison, in actual cloudy fruit-based soft drinks the level is less than 10% of that found in the simulant.

c) ACIDIC FOODSTUFFS

It is well known that 3% acetic acid (AA) is problematic for overall migration (OM) testing of organic coatings on aluminium, tinplate or ECCS (electrolytically chromium coated steel) due to corrosion. Corrosion will often occur in testing metal coated with organic coatings with AA where it does not occur with acidic foods (even if they have the same acidity or pH). This is due to buffering and passivation effects of food constituents, as well as oxygen in the packaged foodstuff being removed, unlike testing in simulants. In addition, the corrosion product is only partly from the package (the cation). Corrosion should not directly influence specific migration although if the corrosion is gross, the level of corrosion product may interfere with the sample work up, etc. causing the need for filtration. Of course if the testing causes complete failure of the organic coating, leading to detachment, it would be reasonable to assume that this does not represent a realistic test, not least because you would then be extracting from both surfaces of the coating and if you had a two-coat system you would be directly extracting from the base coat which would not happen in reality. If the test with 3% acetic acid leads to visible failure and/or any detachment of the organic coating it should be considered invalid and tests should be carried out under the worst foreseeable conditions of use in which these physical or other changes do not take place.

d) LARGE FOOD CANS

A large food can (5kg), used for catering packs may undergo processing in reality for 3 hours but the time specified in JRC Migration Testing Guidelines is 6 hours. Regulation (EU) No. 2016/1416 (4) allows actual time / temperature conditions to be used.

Organic coatings are applied to a substrate forming a film having a typical thickness of 3-20µm. In contrast to plastics, organic coatings do not form a self-supporting film. Although the extended testing times may work for plastics, as the quantity of material which is being tested in an organic coating film is much smaller than for a plastic then the conditions which are used should more closely match the actual conditions of the organic coating in a commercial application.

5. ALTERNATIVE TESTING CONDITIONS SPECIFIC TO RIGID METAL PACKAGING COATED WITH ORGANIC COATINGS

The following sections describe how each issue has been addressed and the proposals for alternative conditions:

a) ALTERNATIVE APPROACH FOR OVERALL MIGRATION IN 3% ACETIC ACID;

The testing of overall migration with 3% acetic acid in the presence of oxygen and absence of buffering agents can result in misleading results. Therefore 3% acetic acid is not recommended for overall migration. Overall migration of organic constituents can be demonstrated by testing in 10% (or 20%) and 95% ethanol (and/or isooctane). It should be noted that 3% acetic acid can be used for specific migration.

b) ALTERNATIVE SIMULANT FOR 50% ETHANOL FOR SPECIFIC MIGRATION;

The use of 50% ethanol (D1) as a simulant for dairy food and fruit juice pulps, under sterilisation conditions combined with long term storage can be more severe than the actual foodstuff. This can manifest as delamination of the organic coating from the substrate. According to JRC Migration Testing Guidelines 4.3.3, materials and articles intended for contact with all types of food shall be tested with food simulants 10% ethanol (A), 3% acetic acid (B) and vegetable oil (D2). As simulant A simulates aqueous food and simulant D2 simulates fatty foods, then by using both of these the extreme characteristics of dairy food are being tested.

c) EVIDENCE THAT ADDITIONAL LONG TERM STORAGE MAY NOT BE REQUIRED FOR OM TESTING OF STERILISED PRODUCTS.

Due to the low film thickness and chemistry of the organic coatings, the bulk of the overall migration occurs during the sterilisation stage of 1 hour @ 121°C or 30 minutes @ 130°C. In some cases it can be demonstrated that there is no migration after the sterilisation stage in which case the 10 day storage period is not required.

d) EVIDENCE THAT TEMPERATURE CONDITION OF 135°C COULD BE CARRIED OUT AT LOWER TEMPERATURES FOR A LONGER TIME.

Although industrial food cans may exceptionally be sterilised at temperatures up to 135°C, laboratory retorts are not generally able to operate reliably at 135°C. Therefore instead of carrying out sterilisation of 30 mins @ 135°C, using the Arrhenius equation the sterilisation could be carried out for example at 40 mins @ 130°C or 70 mins @ 121°C.

e) ACTUAL CONDITIONS VERSUS THOSE SPECIFIED IN THE JRC MIGRATION GUIDELINES.

Unlike some FCMs, the actual most extreme conditions of processing of organic coatings for rigid metal packaging are generally known and are strictly controlled for microbiological reasons, therefore rather than using the OM bands in JRC Migration Testing Guidelines and Regulation (EU) No. 2016/1416 (4), which are for more general cases, the actual conditions can be used in preference.

6. ANNEXES

ANNEX 1: EXAMPLE OF A TINPLATE PANEL COATED WITH AN ORGANIC COATING AFTER 3% ACETIC ACID MIGRATION TESTING

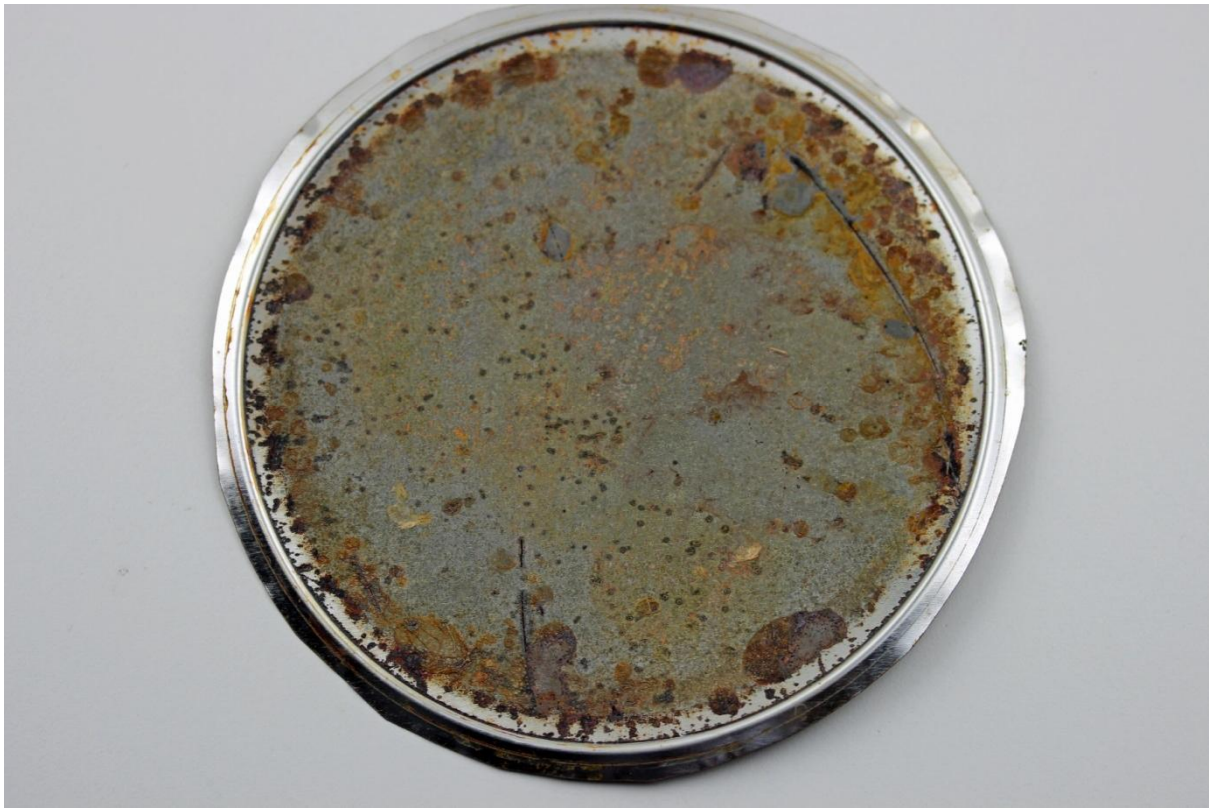

Figure 1 - Tinplate panel coated with an organic coating after OM testing in simulant B, 1 hour @ 130°C + 10 days @ 60°C

ANNEX 2: TYPES OF RIGID METAL PACKAGING COATED WITH ORGANIC COATINGS

- Cans
  - Two piece cans

Single drawn

Drawn and redrawn (DRD)

Bottle

  - Drawn and wall-ironed (DWI)

Drawn and ironed (DI)

  - Three-piece cans

- Can Ends

Classic Ends

Easy Open Ends

Peel Off Ends

- Drums and Pails

· Aerosols

· Tubes

· Trays and foils

· Closures

Crowns

Vacuum closures

Bottle closures (Al)

· Slip Lids

ANNEX 3: REFERENCES

1. Regulation (EC) No. 1935/2004 Of The European Parliament And Of The Council On Materials And Articles Intended To Come Into Contact With Food And Repealing Directives 80/590/EEC and 89/109/EEC. *Official Journal Of The European Union.* Strasbourg : s.n., 27th October 2004. Vol. L 338/4.

2. Joint Research Centre. Technical Guidelines For Compliance Testing, DRAFT for stakeholder consultation, In The Framework Of Regulation (EU) No. 10/2011 On Plastic Food Contact Materials. *Science and Policy Report.* s.l. : European Union, 2016.

3. Oldring, P K and Nehring , Ulrich. *ILSI Europe Report Series - Packaging Materials 7. Metal Packaging For Foodstuffs.* 2007.

4. COMMISSION REGULATION (EU) No. 2016/1416 amending and correcting Regulation (EU) No. 10/2011 on plastic materials and articles intended to come into contact with food. *Official Journal of the European Union.* 24th August 2016. Vol. L230/22.