

The European voice of the adhesive and sealant industry

Brussels, 27 May 2016

# Migration testing of adhesives intended for food contact materials

FEICA, the Association of the European Adhesive & Sealant Industry, is a multinational association representing the European adhesive and sealant industry. With the support of its national associations and several direct and affiliated members, FEICA coordinates, represents and advocates the common interests of our industry throughout Europe. In this regard, FEICA aims to establish a constructive dialogue with legislators in order to act as a reliable partner to resolve issues affecting the European adhesive and sealant industry.

## Introduction

This guidance paper forms part of a package on migration testing of non-plastic food contact materials developed by several sector associations from the packaging supply chain. This document outlines the specific guidelines for conformity testing of adhesives as components of food contact materials. To learn more about the guidance papers of the other sectors, refer to the information on the FEICA website at: <a href="http://www.feica.eu/our-priorities/key-projects/food-contact.aspx">http://www.feica.eu/our-priorities/key-projects/food-contact.aspx</a>

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# Format for Material-Specific Guidelines for Conformity Testing

# Chapter 2: Adhesives

# 1. Scope

This chapter outlines specific guidelines for conformity testing of adhesives as components of food contact materials. Adhesives typically make up less than 5% of the packaging and, for most adhesive applications, direct food contact is not intended<sup>1</sup>. Depending on the adhesive and application, contact may occur unintentionally at seams and edges, or via migration through the packaging or gas phase due to volatile compounds (vapour phase transfer). Consequently, specific migration of individual compounds is of greater interest than overall migration, as the latter is only a measure of inertness and has no toxicological meaning.

Following the requirements of Regulation (EU) No. 1935/2004, the finished food contact material has to be tested and / or evaluated under real use conditions. Testing the adhesive alone can only be considered as a screening tool and should only be used in cases where the worst-case calculation fails or cannot be carried out due to missing information. For more details of how to select a suitable adhesive for application in food contact materials, please consult the FEICA Guidance for a food contact status declaration for adhesives<sup>2</sup>

Tests according to the rules of the plastics regulation on the pure adhesive without substrate or the construction material will usually overestimate the migration of constituents into foodstuff, as contributing factors to real migration are not sufficiently considered. Contributing factors can be:

- Curing times and conditions
- Interaction of adhesive with other FCM layers
- Barrier properties of other FCM layers<sup>3</sup>
- Distribution of constituents within the FCM
- Ratio of adhesive amount to filling good

## 2. Typical adhesive systems used in food contact applications

Due to the wide range of applications and the complexity of the chemistry, no unified testing conditions can be defined for adhesives (see "References" for further details). Therefore, in many cases the conditions defined in Regulation 10/2011 for plastics cannot be applied to adhesives.

The following types of adhesives with their typical applications are defined<sup>4</sup>.

## 2.1. Reactive polyurethane (PU) adhesives

Reactive polyurethane adhesives are composed of one or two components (solvent-based, solvent-free or water-based) and are predominantly used for lamination of polymer films. In addition to pure polymer material, the layers of the finished laminate may consist of metallised films or other materials such as aluminium, paper, etc. Proper curing conditions according to the recommendations of the adhesive manufacturer have to be ensured.

<sup>&</sup>lt;sup>4</sup> Microwave and ovenable applications are not considered in this document.



<sup>&</sup>lt;sup>1</sup> With the exception of heat and cold seals and pressure sensitive for direct food labelling

<sup>&</sup>lt;sup>2</sup> <u>http://www.feica.eu/ehs-sustainability/food-contact</u>

<sup>&</sup>lt;sup>3</sup> The barrier properties of the different FCM layers are not within the scope of this guideline. More information on barrier properties of different materials can be found in the migration testing guideline for plastics.

The application ranges of the finished article encompasses all types of foodstuffs, including prolonged storage (> 6 months) and temperature conditions from cooled storage (below 0 °C) up to retort conditions (e.g. 135 °C).

#### 2.2. Adhesives based on natural polymers

Adhesives based on natural polymers are water-soluble adhesives such as dextrins or starches and are mainly used for, but not limited to, the paper and cardboard packaging of dry foodstuff and for secondary / tertiary packaging. In combination with natural protein, starches and dextrins may be used for bottle labelling. They are also used as wet laminating adhesives in the construction of foil to paper for spiral wound tubes and for paper/foil lidding.

# 2.3. Dispersions / emulsions: Adhesives based on vinyl acetate polymers (PVAc) or ethylene vinyl acetate (EVA) copolymers

Vinyl acetate polymers (PVAc) and ethylene vinyl acetate (EVA) copolymers are applied as dispersions, emulsions or water-soluble adhesives. They are mainly used for, but not limited to, paper and cardboard packaging of dry foodstuff or for secondary / tertiary packaging. PVAc adhesives are also used for wet laminating of paper to foil, as are ethyl vinyl acetate EVA copolymer dispersions. Water-based EVA copolymer dispersions are used as a direct food contact layer for lidding applications on to foil, polyester, and various polyethylene and polypropylene substrates.

These materials can also be used in combination with acrylic emulsions to make synthetic cold seals for primary packaging for food.

In addition, they may be used for labelling and tapes, as well as tissue and towel applications.

# 2.4. Dispersions / emulsions: Adhesives based on acrylic polymers and copolymers, including styrene acrylate terpolymers and reactive systems

Acrylic polymers, copolymers or terpolymers are applied as dispersions, emulsions or water-soluble adhesives. They are mainly used for, but not limited to, packaging of dry foodstuff or for secondary / tertiary packaging, applied on different substrates such as paper, cardboard or polymer films.

Ethylene acrylic acid (EAA) and ethylene methyl acrylic acid (EMAA) dispersions are also used for wet laminating of paper to foil and direct lidding applications on to foil, polyester, and various polyethylene and polypropylene substrates. As pressure sensitive adhesives (PSA), they may be used for direct labelling of e.g. fruits, or for seal/reseal (reclose) applications of snack, meat and cheese packaging.

In most cases, the finished article can be expected to come into contact with foodstuff at or below room temperature. However, in special cases, use conditions may include 'boil-in-bag' and hot fill, in which case the adhesives supplier should be contacted.

Storage times depend on the shelf life of the respective foodstuff (a few days up to months).

#### 2.5. Cold seals

Natural rubber latex and synthetic rubber are applied as dispersions. They are typically used as a seam sealing application on film and paper, e.g. for chocolate, candy and ice cream. Intended direct contact with the foodstuff cannot be excluded. The filling good is expected to be dry or frozen, with contact conditions at or below room temperature. Storage times depend on the shelf life of the respective foodstuff (a few days up to months).



#### 2.6. Heat seals

Heat seals are synthetic resin based coatings for film (e.g. polyester, polyethylene, polypropylene) and foil substrates (including aluminium) used for tray and cup lids. Thus, intended direct contact with the foodstuff cannot be excluded.

The chemistries can also be polyester, acrylic and vinyl, and can also include some nitrocellulose. They can also contain hydrocarbon or rosin or modified rosin based materials as tackifiers plus natural and synthetic waxes.

#### 2.7. Hotmelt adhesives

Traditional hotmelts are based on high levels of waxes and low levels of polyolefin copolymers with low softening point<sup>5</sup>, such as EVAs and tackifiers. They are applied on paper and cardboard packaging of dry foodstuff (including bags and sacks) and for secondary / tertiary packaging (indirect food contact).

Newer (or alternative) types of hotmelts mainly consist of ethylene vinyl acetate (EVA) or polyolefin (PE, PP) copolymers and have a higher softening point. They still contain low levels of waxes and tackifiers. In addition, acrylic or styrene (co)polymers (e.g. SBS, SIS) might be added. These materials can also contain tackifiers along with various types of waxes. These types of materials could be used in lidding applications for direct food contact of either dry or wet food that possibly contains oils and fats.

## 3. Material-specific properties to be considered when testing this class of FCM

#### 3.1. Reactive polyurethane (PU) adhesives

Polyurethane adhesives bond the different layers of a multilayer material. The finished material may be used as a flexible packaging for foodstuff, typically being subject to the plastics regulation (EU) No. 10/2011. Thus, as a general requirement, the finished material has to be tested according to the plastics regulation, including the contribution of the polyurethane adhesive to overall and specific migration.

Direct testing of the adhesives is not possible due to their reactivity. The tests can only be performed on the final structure (to be defined by the user) or on model systems prepared for that purpose. This implies a thorough understanding of the expected conditions of use (e.g.: type of foodstuff, temperatures, time, ratio of surface to amount of filling good). In addition, the monolayers held together by the adhesives should be investigated separately to clearly distinguish between the contribution of the adhesives compared to the other layers of the finished material. Moreover, the recommended curing conditions specified by the adhesive manufacturer have to be ensured, as the conversion conditions might affect the migration properties. The following material-specific properties have to be considered when testing laminates for the adhesive contribution to migration:

<sup>&</sup>lt;sup>5</sup> BfR recommendation XXV Hard Paraffins, Microcrystalline Waxes and Mixtures of these with Waxes, Resins and Plastics



#### 3.1.1.Reaction with food simulants

Based on reactive isocyanate chemistry, it has to be ensured that the food simulant used does not react with constituents of the adhesive. For example the food simulant ethanol will react with isocyanates to form carbamates. As a consequence, the residual isocyanate content cannot be determined. Most food simulants (e.g. 10% ethanol, 3% acetic acid, 20% ethanol and 95% ethanol, etc.) will react with unreacted isocyanate and/or unreacted isocyanate prepolymers; this reaction does not limit or negate the use of these food simulants but rather necessitates the proper identification of the potential by-products.

As a consequence, determination of primary aromatic amines (PAA) from hydrolysis of isocyanates would not be successful in ethanolic food simulants (formation of carbamates is favoured), nor would it be possible to determine residual isocyanate monomer content from any of the listed aqueous or ethanolic simulants.

## 3.1.2. Weakening of the laminate structure

The testing of most laminates under hot-fill, boil-in-bag, or retort conditions will exhibit some decrease of bond strength with most food simulants (e.g. 3% acetic acid, 10% ethanol or 95% ethanol). However this decrease in bond strength does not negate the need for testing and evaluation with these food simulants. In case of doubt, the actual application needs to be tested.

## 3.1.3. Accelerated tests at elevated temperature

According to the plastics regulation, 60°C for 10 days is applied as an accelerated test for prolonged storage (> 6 months). However, for some cases it is known that a change of physical properties will take place at 60°C. The observed migration at 60°C will be different from real migration at room temperature or even at 40°C. In these cases, the migration results obtained may not be valid, and will be too high in the worst-case scenario.

## 3.1.4.False-positive results from extracted constituents

Some species can be created from other components in the extract during analysis. Therefore, it is essential to consider the type of adhesive during selection of analytical method and to verify positive results.

## 3.2. Adhesives based on natural polymers

For vegetable adhesives and paper/cardboard applications, direct contact is not intended but cannot be excluded on seams or edges. Migration via gas phase should be considered in case the substrate offers no barrier properties (vapour phase transfer).

In contrast, bottle labelling with substrates like glass is not expected to contribute to migration, as glass will act as an absolute barrier.

The major limitation of the testing guidelines based on plastics regulation (EU) No. 10/2011 is the predominant use of liquid simulants such as 3% acetic acid or ethanolic solutions. Liquid simulants may re-dissolve adhesives based on natural polymers, leading to total extraction rather than migration. As a result, adhesives based on natural polymers, especially in paper/cardboard applications, cannot be tested for migration with liquid simulants.



# 3.3. Dispersions / Emulsions: Adhesives based on vinyl acetate polymers (PVAc) or ethylene vinyl acetate (EVA) copolymers

Adhesives based on vinyl acetate polymers (PVAc) and ethylene vinyl acetate copolymers are used for paper/cardboard applications, where direct contact is not intended but cannot be excluded on seams or edges. Migration via gas phase (vapour phase transfer) should be considered in case there are only weak barrier properties or no barrier properties at all.

The major limitation of the testing guidelines based on plastics regulation (EU) No. 10/2011 is the predominant use of liquid simulants such as 3% acetic acid or ethanolic solutions. Liquid simulants may re-dissolve the adhesive, leading to total extraction rather than migration. As a result, the adhesive cannot be tested for migration with liquid simulants, particularly in paper/cardboard applications.

# 3.4. Dispersions / emulsions: Adhesives based on acrylic polymers and copolymers, including styrene acrylate terpolymers and reactive systems

Adhesives based on acrylic polymers and copolymers, including styrene acrylate terpolymers and reactive systems, are used for paper/cardboard applications, where direct contact is not intended but cannot be excluded on seams or edges (for direct labelling see below). Migration via gas phase (vapour phase transfer) should be considered in case there are only weak barrier properties or no barrier properties at all.

The major limitation of the testing guidelines based on plastics regulation (EU) No. 10/2011 is the predominant use of liquid simulants such as 3% acetic acid or ethanolic solutions. Liquid simulants may re-dissolve the adhesive, leading to total extraction rather than migration. As a result, the adhesive cannot be tested for migration with liquid simulants, particularly in paper/cardboard applications.

Special attention has to be paid to direct labelling of foodstuff in the case of pressure-sensitive adhesives (PSA). Plastics regulation (EU) No. 10/2011 refers to liquid simulants anytime free fats may be present on the surface of dry foodstuff. In these cases, it has to be ensured that the migration test is performed in a way that comes closest to the actual application. Migration results are not valid when the adhesive is dissolved in the liquid food simulant or removed from the substrate.

# 3.5. Cold seals

Natural rubber and synthetic latex are used for paper and film applications in the packaging of dry foodstuff. Direct food contact is not intended but cannot be excluded on seams or edges.

The major limitation of the testing guidelines based on plastics regulation (EU) No. 10/2011 is the predominant use of liquid simulants such as 3% acetic acid or ethanolic solutions. Liquid simulants may re-dissolve the latex, leading to total extraction rather than migration. As a result, the dispersion cannot be tested for migration with liquid simulants, particularly in paper applications.

## 3.6. Heat seals

Typical lid applications for cups and trays include direct food contact. Migration with liquid food simulants according to plastics regulation (EU) No. 10/2011 may lead to re-dissolving of the heat seal, especially using lipophilic simulants with high ethanol content. Moreover, when applied on aluminium, 3% acetic acid as a food simulant for acid foodstuff will cause corrosion of the aluminium layer, followed by aluminium release into the simulant.



In these cases, migration results have to be considered as non-valid, because the actual foodstuff would not re-dissolve the heat seals nor corrode the aluminium layer.

## 3.7. Hotmelt adhesives

For hotmelt adhesives and paper/cardboard applications, direct contact is not intended but cannot be excluded on seams or edges, including window applications in paper bags. Migration via gas phase (vapour phase transfer) should be considered in case the substrate offers no barrier properties. Depending on the type of hotmelt, it may be used as an extrudable heat seal coating to replace a water- or solvent-based coating or a sealant film.

The major limitation of the testing guidelines based on plastics regulation (EU) No. 10/2011 is the predominant use of liquid food simulants such as ethanolic solutions. Liquid simulants with ethanol as an organic solvent may re-dissolve large parts of adhesives (e.g. tackifiers), which the packaged food would not do, resulting in extraction rather than migration. In addition, elevated temperatures above 40°C are likely to cause a softening of the hotmelt due to the low molecular weight fraction (e.g. waxes)<sup>6</sup>.

In general, food simulants and conditions in Regulation (EU) No. 10/2011 are not appropriate for this category of materials. The detailed testing conditions should be defined on a case-by-case basis, depending on the adhesive and application types.

## 4. Test procedures and evaluation of test results

Numerous test procedures are available for the determination of defined compounds in various matrices. Partly, they have been subject to standardisation, and are published as test guidelines at the European level, e.g. DIN EN 13130 test method series.

However, due to the wide range of applications concerning food contact materials, the preparation and analysis of test solutions greatly impacts the final assessment.

The preparation of test solutions involves the following steps:

- Anticipation of a real food contact scenario and transformation into a migration test setup.
- Migration of the material under pre-defined conditions (choice of food simulant, temperature and time).
- Transfer of migration / extraction solution into a "ready-to-analyse" test solution<sup>7</sup> or test substance (extractions, etc.).

In each of these steps, decisions have to be made that will influence the analytical result of the applied test procedure. Thus, separate analyses of identical food contact materials according to the same test method might reveal different test results.

The following subchapters list recommendations on how to test adhesives, taking into account their material-specific properties, in contrast to pure plastic materials. This includes consideration of all parts of the migration study, from setting up the migration test to final evaluation of the test results.

#### Annotation 1:

Besides migration, residual content measurements (QM) according to the plastics regulation may be required. In these measurements, strong solvents such as dichloromethane are used to achieve

<sup>&</sup>lt;sup>7</sup> As injected in the analytical system (GCMS, LCMS, etc.)



<sup>&</sup>lt;sup>6</sup> BfR recommendation XXV Hard Paraffins, Microcrystalline Waxes and Mixtures of these with Waxes, Resins and Plastics

total extraction. Under these severe conditions, monomers, polymers and other constituents may be re-dissolved. Depending on the analytical technique, oligomers and polymers are prone to cleavage, giving false-positive monomer results. For example, the thermal impact on the extraction solution while being injected into a hot GC (gas chromatograph) injection port will release monomeric isocyanates from polyurethanes (see chapter 3.1.4).

#### Annotation 2:

Whenever an adhesive is applied on a substrate for migration analysis (e.g. on paper or polymer film), the substrate itself needs to be investigated thoroughly. Migration from the substrate has to be evaluated separately in order to differentiate the contribution to migration (generation of a "blank value" without adhesive).

#### Annotation 3:

As already outlined in chapter 2 "<u>Typical adhesive systems used in food contact applications</u>" and chapter 3 "<u>Material-specific properties to be considered</u>", the complexity of adhesive formulations needs to be considered when performing migration tests (e.g. applicability of analytical procedures, dissolving of raw materials and formulations in food simulants, softening/melting of ingredients at elevated temperatures). See chapter 5 "<u>References</u>" for further details on specific adhesive properties.

#### 4.1. Reactive polyurethane (PU) adhesives

#### 4.1.1.Reaction with food simulants

Determination of residual isocyanates should be performed using inert solvents such as dichloromethane to prevent any degradation or formation of by-products. It has to be noted that inert solvents may contain impurities or residual moisture, so it is recommended to use solvents with high purity. Even with a specified residual water content, the actual amount will vary from batch to batch, and increase after being opened. Consequently, solvents with the same specification may result in different isocyanate values.

Furthermore, performing a total extraction on the laminate (e.g. via cutting the material for total immersion) may resolve parts of the polyurethane polymer, which may interfere in the further course of the investigations (see chapter 3.1.4).

If an inert solvent cannot be used, e.g. special food simulants like ethanol are mandatory or significant residual impurities cannot be avoided, a reaction between the solvent and the adhesive system has to be expected. In that case, the applied analytical method has to be capable of detecting and determining the amount of the unavoidable by-products. For example, determination of residual isocyanates in ethanolic solutions is achieved via their carbamates, i.e. their ethyl adduct.

It has to be noted that reference substances (= "standards") are typically required for unequivocal identification and quantification. When determining reactive adhesive ingredients via their by-products, the availability of suitable standards to ensure a reliable determination may be limited. In this case, semi-quantitative determination using internal standards may be appropriate.

Determination of primary aromatic amines (PAA) should preferably be performed from acetic acid solutions, e.g. 3% acetic acid as a food simulant. For laminates used under ambient conditions, it is generally accepted that 70°C for 2 hours ensures hydrolysis of available aromatic isocyanates. However, when laminates are subjected to elevated temperatures in use, e.g. boil-in-bag, there is the possibility of a thermal degradation of the adhesive, releasing isocyanate and hence PAA. For such applications, the actual conditions of use should form the basis of the testing conditions.

Measurement of PAAs does not ensure that limits for NCOs (i.e.: QM(T) 1 mg/kg in the final product expressed as isocyanate moiety) are met. However, it may be possible to demonstrate a correlation between PAA and NCO content. In such cases, PAA testing may be used as a practical method of demonstrating an adequate cure of the adhesive.



It should be noted that aliphatic diisocyanates do not generate PAAs; hence only the limits for NCOs apply.

#### 4.1.2. Weakening of the laminate structure

It should be confirmed that the production and curing of the laminate complies with the recommendations of the adhesive manufacturer. The sample to be migrated and analysed should be representative for the laminate.

If the laminate fails under aggressive testing, then the results need to be noted and reported. If there is degradation of the polymer network, the resulting by-products should then be analysed and reported. It needs to be noted that these results may or may not indicate that this adhesive system is not suitable for the desired application, but it could also be an indication that the testing conditions simply need to be adjusted.

In case of doubt, the real application needs to be tested.

#### 4.1.3. Accelerated tests at elevated temperature

Migration conditions defined by regulation (EU) No. 10/2011 may include elevated temperatures (e.g. 60°C) that do not reflect the real conditions of use. Anytime these standardised test conditions lead to a change of the physical properties of the laminate compared to the properties of the actual food contact scenario, this should be noted and reported. In case there is doubt concerning the relevance of the migration test, the test should be repeated using the actual contact temperature, time and/or foodstuff.

#### 4.1.4.False-positive results from extracted constituents

Special attention has to be paid to migration solutions that contain extracted polymers or oligomers. Organic solvents used as food simulants may extract polymeric parts of the food contact material. When analysed via gas chromatography (GC), an injected aliquot of the test solution is heated up to more than 200 °C, which leads to a decomposition of the polymer and the detection of monomers. This finding has to be evaluated as an analytical artefact, because the monomers are not present in the migration solution, but being generated artificially in the analytical test system. Consequently, the test results are not valid.

The following polymers may be prone to thermal decomposition (examples):

- Polyurethanes: detection of residual isocyanates
- Polyesters: detection of residual glycols
- Polyacrylates: detection of residual acrylates
- etc.

The understanding of thermal decomposition plays a pivotal role in the evaluation of migration test results, as GC(MS) screenings are an established way to obtain a quick overview of the migration properties of food contact materials. Doing so, the analyst has to be aware of possible false-positive results with respect to residual monomer contents. Adequate countermeasures have to be taken and reported.

#### Annotation 1: Critical temperatures for thermal decomposition

Due to the wide variety of polymeric content in food contact materials and adhesive systems, it is not possible to define one critical temperature that needs to be exceeded to result in thermal decomposition. However, thermal impact close to and exceeding 100 °C in the analytical test system (e.g. hot injection port of GC systems) should be evaluated carefully.



#### Annotation 2: Analytical alternatives in case of thermal decomposition

Gas chromatography causes thermal impact on the injected test solution. However, there are also injection techniques in place that offer injection at lower temperatures (e.g. on-column injection). Liquid chromatography (e.g. HPLC = high pressure liquid chromatography) generally involves no thermal impact at all, and should be considered as first choice for the evaluation of residual monomers.

#### Annotation 3: Verification of residual monomer results

When using established GCMS screenings for a quick overview, any finding of residual monomers could be verified by changing the temperatures within the analytical test system. E.g. changing the temperature of the injection port should not change the true amount of residual monomers. Any correlation between temperature and monomer content may be an indication for thermal decomposition during analysis.

#### 4.2. Adhesives other than reactive polyurethane (PU) adhesives

This chapter summarises recommendations on how to test adhesives other than reactive PU adhesives and includes the following adhesive types:

- Adhesives based on natural polymers
- Dispersions / emulsions: Adhesives based on vinyl acetate polymers (PVAc) or ethylene vinyl acetate (EVA) copolymers
- Dispersions / emulsions: Adhesives based on acrylic polymers and copolymers, including styrene acrylate terpolymers and reactive systems
- Cold seals
- Heat seals
- Hotmelts

#### 4.2.1 General recommendations

For their typical applications in paper and cardboard packaging, adhesive ingredients will migrate via the gas phase into the foodstuff (vapour phase transition). This migration path should be simulated using Tenax<sup>®</sup> as a food simulant. Migration time and temperature shall be chosen according to the expected conditions of use, considering material specific properties (e.g. change of physical properties due to softening of waxes).

Migration should be performed on the finished good, considering the influence of other parts/layers of the food contact material. In case the finished good is not available, the adhesive may be applied on a suitable substrate that most closely approximates to the real application. For example, the adhesive may be applied as a film on paper and, after drying, this coated paper may be used for migration testing. Headspace analysis of the adhesive may act as a first screening tool for determination of volatile components and their risk assessment.

Typically, Tenax<sup>®</sup> is not transferred directly onto the adhesive layer, but stored within a certain distance for vapour phase transition. However, when direct contact of the adhesive with the foodstuff cannot be excluded in the actual application, migration may also be performed in direct contact, giving worst-case values.

In the case of direct contact with liquid food simulants, the dried adhesive may be re-dissolved. The results obtained from such tests cannot be evaluated as migration results, as the physical properties of the test items clearly change. Anytime a re-dissolving of the adhesive film in liquid simulants is observed, this should be noted and reported. These results may be used for worst-case considerations, but clearly overestimate migration onto dry foodstuff. In addition, false-positive results from extracted constituents have to be considered as described for reactive PU adhesives (see chapter 3.1.4.: polymers from the adhesive formulation may be cleaved during analysis, indicating false-positive residual monomer contents.



#### 4.2.2 Special recommendations for pressure-sensitive adhesives (PSA)

Dispersions and emulsions based on acrylic polymers (see chapter 3.4.), as well as hotmelts (see chapter 3.7), may include pressure-sensitive adhesives (PSA). After application, they will maintain their tacky properties, so that the dry food simulant Tenax<sup>®</sup> will stick to the adhesive and will not be able to be removed after migration. However, should direct food contact need to be simulated, e.g. direct labelling of foodstuff, it should be considered to apply the PSA on a substrate with low or no barrier properties, e.g. paper or thin PE film. Migration then can be performed from the paper or PE side, preventing Tenax<sup>®</sup> from sticking to the adhesive.

In this migration setup, the substrate should only help to separate the food simulant from the PSA. It should not influence migration. In case of doubt, the actual application needs to be tested.

## 4.2.3 Special recommendations for cold seal and heat seal applications

Cold seals and heat seals may be applied on aluminium as a substrate (e.g. lidding applications). It has to be considered that acid food simulants such as 3% acetic acid cannot be used in these cases. However, just for testing purposes, it is possible to select alternative substrates. Besides the possibility of re-dissolving the cold seal or heat seal (as discussed in preceding chapters), acid liquids will also dissolve the aluminium substrate, leading to the total destruction of the food contact material. The results of such migration tests are not valid.

In case of doubt, and where the food contact scenario includes contact with acid foodstuff, the actual application needs to be tested.

## 5. References

- MIGRESIVES, Research Programme on Migration from Adhesives in Food Packaging, Materials in Support of European Legislation and Standardisation, Collective Research Programme, COLL-CT-2006-030309, Final Activity Report 2011
- Adhesives in Food Contact Materials and Articles, Proceedings from a Nordic Seminar (June 2001), Svensson et al., TemaNord 20021551© Nordic Council of Ministers, Copenhagen 2002, ISBN 92-893-0808-7

# 6. Contact

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