SLOVENSKI STANDARD
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Materiali in predmeti v stiku z živili – Polimerne prevleke na papirju in kartonu - Vodilo za izbiro pogojev in preskusnih metod za celotno migracijo

Materials and articles in contact with foodstuffs - Polymeric coatings on paper and board- Guide to the selection of conditions and test methods for overall migration

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Polymere Beschichtungen auf Papier und Pappe - Leitfaden für die Auswahl von Prüfbedingungen und Prüfverfahren für die Gesamtmigration

Matériaux et objets en contact avec les denrées alimentaires - Revetements polymeres sur papier et carton - Guide pour le choix des conditions et des méthodes d'essai en matiere de migration globale

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Materials and articles in contact with foodstuffs — Polymeric coatings on paper and board — Guide to the selection of conditions and test methods for overall migration

Matériaux et objets en contact avec les denrées alimentaires – Revêtements polymères sur papier et carton – Guide pour le choix des conditions et des méthodes d'essai en matière de migration globale

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Foreword

This document (CEN/TS 14234:2002) has been prepared by Technical Committee CEN/TC 194, "Utensils in contact with food", the secretariat of which is held by BSI.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For relationship with EU Directive(s), see informative annex ZA, which is an integral part of this document.

In this standard the Annex A is normative and Annex B is informative.

Guidance for migration testing of polymeric coatings on cellulosic substrates (paper and board) is given in the informative annex B.

At the time of preparation and publication of this Technical Specification the European Union legislation relating to resinous and polymeric coatings on paper and board has not been formulated.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This Technical Specification specifies test methods for 'alternative tests' and 'substitute tests' performed with volatile test media, iso-octane and a volume fraction of 95 % aqueous ethanol, for the determination of overall migration from polymeric coatings on paper and board intended to come into contact with fatty foodstuffs.

NOTE 1 It also includes guidance for the application of Council Directive 82/711/EEC [1], as last amended by Commission Directives 93/8/EC and 97/48/EC [2] [3] as regards the determination of the specific and overall migration into fatty food simulants (fat test) and includes a glossary to clarify the terminology used, see annex B.

NOTE 2 The iso-octane and a volume fraction of 95 % aqueous ethanol volatile test media used in these test methods are those specified for 'substitute tests' in Council Directive 82/711/EEC and its subsequent amendments. In addition to the use of iso-octane and 95 % v/v aqueous ethanol as test media for 'substitute tests', Council Directive 82/711/EEC and its subsequent amendments specifies the use of modified polyphenylene oxide as a test medium for use at temperatures of 100 °C and above. A test method for overall migration from polymeric coatings intended to come into contact with fatty foodstuffs using modified polyphenylene oxide (MPPO) is in preparation.

NOTE 3 These test methods can also be used for the 'alternative tests' described in Council Directive 82/711/EEC and its subsequent amendments, when the chosen volatile test media are iso-octane and a volume fraction of 95 % aqueous ethanol, see ENV 1186-1.

A suggested test scheme is given in Figure A.1. If the test result obtained by the total immersion method, described in clause 4 of this Technical Specification, exceeds the allowed overall migration limit the single-sided migration test using a cell, see clause 5, if technically feasible; may be used. If it is not possible to use the single sided cell method, e.g. because of pinholes, use adsorption by modified polyphenylene oxide, (MPPO).

Iso-octane is used as a test medium for test samples coated with non polar food contact layers, such as polyolefins. For test samples coated with polar food contact polymers such as polyamide and polyethylene terephthalate, a volume fraction of 95 % aqueous ethanol is used. For polystyrenes, plasticised polyvinyl chloride and other polymers where the identification or polarity of the polymer is not clear, two parallel tests shall be conducted using both of the proposed test media and taking the higher value obtained as the relevant result.

NOTE 4 The nature of the food contact layer determines the selection of the test medium(a).

NOTE 5 The overall migration limit for materials and articles of an all polymeric construction is specified in Commission Directive 90/128/EEC [4] and the conditions of test in Council Directive 82/711/EEC [1] and its subsequent amendments, [2],[3]. There is an expectation that the scope of this directive will be extended to cover polymeric coatings on paper and board.

The 'substitute tests' described in this Technical Specification are by total immersion, see clause 4 and in a migration cell, see clause 5.

NOTE 6 The test conditions are those described in Council Directive 82/711/EEC [1] and its subsequent amendments, [2],[3].

The 'alternative test' methods described here specifies a rapid extraction test with a 'more severe' test character, for the assessment of the overall migration into fatty food simulants. The method differs from the substitute test methods only in the combination of test time and test temperature. The method is based on the determination of the extraction of migrateable substances from polymeric coated paper or board, which are intended to come into contact with foodstuffs, by total immersion in non-polar, iso-octane, and/or polar, ethanol, solvents depending on the polarity of the polymeric coating. According to the results obtained by this method, see [5],[6],[7],[8],[9] and taking physio-chemical considerations into account, the obtained extraction efficiency has, generally, been found to be equivalent to or higher than overall migration results obtained using fat simulant D,under the test conditions, 10 days at 40 °C, 2 h at 70 °C, 1 h at 100 °C, 30 min at 121 °C and 30 min at 130 °C.

NOTE 7 This method is not applicable to test materials intended for applications over 130 °C. Test materials intended for applications over 70 °C have to be checked for their physical suitability at the intended time and temperature of use.

The test method should be applied to polymer coated packagings where the polymer layer is less than 300 µm in thickness. However, if this extraction test is applied to materials with coatings with higher thickness than 300 µm and the result does not exceed any allowed overall migration limit then the method is considered to be suitable for those materials.

In those cases where the rapid extraction test by total immersion, yields total extraction values that exceed the overall migration limit or may be technically unsuitable a cell test method using the same time and temperature conditions is applicable. This cell test method should primarily only be applied to packagings with a physical barrier layer (for instance of aluminium or other material to prevent penetrative loss of test medium) and which have a thinner food contact layer than 300 µm. This test method can not be applied if the polymeric coating is shown to have pinholes.

2 Normative references

This Technical Specification incorporates by dated and undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to and revisions of any of these publications apply to this Technical Specification only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

ENV 1186-1:1994	Materials and articles in contact with foodstuffs – Plastics - Guide to the selection of conditions and test methods for overall migration
ISO 648	Laboratory glassware – One-mark pipettes
ISO 4788	Laboratory glassware - Graduated measuring cylinders

3 Test conditions

3.1 ´Substitute test´ methods

For substitute tests, the conditions of test will be determined by the conditions of use, see clauses 4, 5 and 6 of ENV 1186-1:1994.

3.2 ´Alternative test' methods

For alternative tests, the test medium (a) and test conditions are to be selected according to the nature of the polymeric coating as given by Table 1.

Table 1 — Use of test media and test conditions for alternative tests

Polymer type of food contact layer	Test medium to be applied	Test conditions to be applied
Polyolefins and copolymers	iso-octane	24 h at 40 °C
Polyamides	95 % ethanol	24 h at 40 °C
Polystyrene	iso-octane and 95 % ethanol	24 h at 40 °C
Polyethylene terephthalate	95 % ethanol	24 h at 50 °C
Polyvinyl chloride (plasticised)	iso-octane and 95 % ethanol	24 h at 40 °C
Polyvinyl chloride (rigid)	95 % ethanol	24 h at 50 °C
In case of doubt or unknown	iso-octane and 95 % ethanol	24 h at 50 °C

4 Total immersion method

4.1 Principle

The migrateable substances extracted from a sample of the polymer coated paper or board is determined as the mass of non-volatile residue after evaporation of the solvent following immersion.

Test specimens of at least 1 dm² (single side considered) are immersed in the test medium for set periods of time and at set temperatures and then removed. The test medium is evaporated to dryness, the mass of the non-volatile residue is determined and expressed as milligrams per square decimetre of surface area of the test specimen.

4.2 Apparatus

4.2.1 Cutting slab, clean smooth glass, metal or plastics slab of suitable area to prepare test specimens, 250 mm x 250 mm is suitable.

4.2.2 Tweezers, stainless steel, blunt nosed.

4.2.3 Cutting implement, scalpel, scissors or sharp knife or other suitable device.

4.2.4 Metal template, 100 mm ± 0,2 mm x 100 mm ± 0,2 mm (square).

4.2.5 Rule, graduated in mm, and readable to the nearest 0,1 mm.

4.2.6 Analytical balance, capable of determining a change in mass of 0,1 mg.

4.2.7 Extraction containers; glass weighing jars with ground joints, tall form, of a capacity of approximately 60 ml.

4.2.8 Thermostatically controlled oven or incubator capable of maintaining a temperature within the range of + 40 °C to + 50 °C and meeting temperature tolerance values within those specified for the test temperature, see annex B of ENV 1186-1: 1994.

WARNING The interior/sample space of the oven or incubator should not have any exposed heating elements, to minimise safety hazards arising from any loss of flammable test media during the test period.

4.2.9 Dishes, stainless steel, nickel, platinum, platinum alloy or gold, 50 mm to 90 mm diameter and maximum weight 100 g, for evaporation of solvents and weighing of residues. Glass, glass ceramic, ceramic or aluminium dishes may be used provided that the surface characteristics are such that the weights of the dishes after evaporation of any specified solvent followed by conditioning in the desiccator used achieves a constancy of 0,5 mg.

4.2.10 Steam bath, hot plate, distillation apparatus or rotary evaporator.

4.2.11 Desiccator with anhydrous calcium chloride or self indicating silica gel.

4.2.12 Measuring cylinder, of a capacity of 50 ml, complying with the minimum requirements of ISO 4788.

4.3 Reagents

4.3.1 Iso-octane, (2,2,4-trimethyl pentane), a volume fraction of 98,5 % purity or greater, CAS No. 540-84-1[1])

4.3.2 Ethanol [a volume fraction of 95 % in aqueous solution], a volume fraction of 96 % purity or greater,

WARNING Both iso-octane and ethanol are volatile flammable solvents. Take care to ensure that the test specimens are well stoppered, closed and covered to prevent solvent volatilising into the interior of the oven, incubator or refrigerator and generating an explosive mixture. Care should be taken at all times when handling these solvents to prevent contact with sources of ignition

[1]) The source of this is the Chemical Abstracts published by the American Chemical Society

4.4 Preparation of test specimens

4.4.1 General

It is essential that test specimens are clean and free from surface contamination (many polymeric coatings can readily attract dust due to static charges). Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use see 8.1 of ENV 1186-1: 1994. Minimize handling of the samples and where necessary, wear cotton gloves.

To ensure that test pieces are well separated and that the surfaces are freely exposed to the extractant during the period of the test, insert a piece of fine stainless steel gauze between the cut test pieces.

4.4.2 Number of test specimens

Three replicate test specimens are required.

4.4.3 Cutting and preparation of specimen

Lay the sample on the cutting slab (4.2.1) and cut the test specimens of 1 dm² (see 8.3 of ENV 1186-1:1994), using the 100 mm x 100 mm template (4.2.4). Check, using the rule (4.2.5), that the dimensions of the specimen are within the specified tolerance (1mm). Fold the test specimens into a fan-like shape or cut into strips approximately 2 cm wide and 5 cm long. Place in the extraction containers (4.2.7).

4.5 Procedure

4.5.1 Exposure to solvent

Take three extraction containers or jars (4.2.7), measure by measuring cylinder (4.2.12) 50 ml of the solvent into each of these jars and immerse the test specimens in the solvent. Ensure that the test specimens are totally immersed in the solvent. If the evaporation method is to be used (4.5.2.2) measure into a further two jars by measuring cylinder the same amount of solvent, plus 10 ml ± 2 ml, to provide blanks. If the distillation method (4.5.2.3) is to be used measure into those further two jars by measuring cylinder the same amount of solvent in contact with the test specimens to provide blanks. Stopper the jars. Mark the jars for identification. Mark the liquid level on the outside of each jar with a suitable marker.

The extraction conditions are to be selected from table 1 (clause 3.1) according to the nature of the polymeric coating of the samples, or according to clauses 4, 5 and 6 of ENV 1186-1: 1994.

Place the five jars in the thermostatically controlled oven or incubator (4.2.8), set at the test temperature and observe the temperature, leave the jars for the set test period after the air bath of the thermostatically controlled oven or incubator has reached the set temperature and taking the permitted time and temperature tolerances into account, see annex B of ENV 1186-1: 1994. Take the jars from the oven or incubator and allow them to cool down to room temperature. Check the level of solvent in each. If this has fallen to more than 5 mm below the mark, or has exposed any part of the test pieces, repeat the test using fresh samples. If the level of solvent in a jar is less than 5 mm below the mark, remove the test specimen from the jar, and allow the solvent adhering to the test specimen and support to drain back into the tube. Recover at least 90 % of the original volume of solvent or repeat the test.

WARNING Both iso-octane and ethanol are volatile flammable solvents. Take care to avoid any loss of solvent into the interior of the thermostatted device. Place the jars, if possible, in a drip container serving as a possible solvent reservoir in case of leakage. Do not allow the temperature to exceed 60 °C.

4.5.2 Determination of extracted substances

4.5.2.1 Preparation of dishes

Take five dishes (4.2.9), marked for identification, place the dishes in an oven maintained at 105 °C to 110 °C, for a period of 30 min ± 5 min, to dry. Remove the dishes from the oven, place in a desiccator (4.2.11) and allow to cool to ambient temperature. Weigh and record the individual masses of each dish. Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until individual consecutive masses differ by not more than 0,5 mg. Record their final masses.

4.5.2.2 Evaporation method

For each jar containing the solvent, pour 20 ml to 25 ml into a prepared dish. By means of a steam bath, hot plate or other form of heating evaporate to a low volume (4.2.10), taking care to avoid loss, in particular, by sputtering or overheating of the residues.

NOTE 1 The evaporation should be carried out in a fume cupboard.

When most of the solvent has evaporated, pour the remaining solvent from each of the jars into the respective dishes and continue the evaporation. Wash out each of the tubes which had contained test specimens with two lots of 5 ml ± 1 ml of fresh solvent and pour these washings into the respective dishes. Continue the evaporation.

NOTE 2 A stream of nitrogen may be used to facilitate evaporation.

When the solvent has almost completely evaporated, place the dish in an oven maintained at 105 °C to 110 °C, for a period of 30 min ± 5 min, to complete the evaporation and dry the residue. Remove the dishes from the oven, place in a desiccator (4.2.11) and allow to cool to ambient temperature. Weigh and record the individual masses of each dish and residue. Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until individual consecutive masses differ by not more than 0,5 mg. Determine the mass of the residue by subtracting the original mass of the dish from the final mass of the dish and residue.

4.5.3 Distillation method

For each jar, transfer the contents to a round bottom flask (250 ml is suitable). Rinse each jar twice, including the blank jars, with 20 ml ± 2 ml of fresh solvent, add these rinses to the respective flasks. Place the flasks in an electric heating mantle and connect to a side arm distillation arrangement or use a rotary evaporator. Distil off the solvent until approximately 15 ml to 25 ml remains in the flask. Transfer the remaining solvents to an evaporating dish. Rinse the flask with 10 ml ± 1 ml of fresh solvent and add the rinses to the appropriate dishes. Continue the evaporation of the solvent by means of a steam bath, hot plate or other form of heating, proceeding as in 4.5.2.2.

NOTE The evaporation should be carried out in a fume cupboard.

Continue with clauses 6 and 7.

5 Cell method

5.1 Principle

The total migrateable substances extracted from a sample of the polymeric coating is determined as the mass of non-volatile residue after evaporation of the solvent following immersion.

One surface of the test specimen of at least 1 dm² (single side considered) is exposed in a cell to the extraction solvent for set periods of time and at set temperatures and then removed. The extraction solvent is evaporated to dryness, the mass of the non-volatile residue is determined and expressed as milligrams per square decimetre of surface area of the test specimen. The measured value is compared to the EC-official overall migration limit and taking the analytical tolerance of this method (± 1mg/dm²) into account.