SLOVENSKI STANDARD

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Materiali in predmeti v stiku z živili - Polimerni materiali - 15. del: Alternativne preskusne metode za migracijo v modelne raztopine za maščobna živila s hitro ekstrakcijo z izooktanom in/ali 95-odstotnim etanolom

Materials and articles in contact with foodstuffs - Plastics - Part 15: Alternative test methods to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 15: Alternative Prüfverfahren zur Migration in fettige Prüflebensmittel durch Schnellextraktion in Iso-Octan und/oder 95%iges Ethanol

Matériaux et objets en contact avec les denrées alimentaires - Plastiques - Partie 15 : Méthodes de remplacement pour la vérification de la migration dans les simulants gras par extraction rapide dans l'iso-octane et/ou l'éthanol aqueux a 95 %

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Materials and articles in contact with foodstuffs - Plastics - Part 15: Alternative test methods to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 15 : Méthodes d'essai alternatives pour la migration dans les simulants alimentaires gras par extraction rapide dans l'iso-octane et/ou l'éthanol à 95 %

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 15: Alternative Prüfverfahren zur Migration in fettige Prüflebensmittel durch Schnellextraktion in Iso-Octan und/oder 95%iges Ethanol

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 194.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Contents

Page

Foreword

This document (prEN 1186-15:2006) has been prepared by Technical Committee CEN/TC 194 “Utensils in contact with food”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

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.

This document will supersede EN 1186-15:2002.

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 volatilizing 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.

Read EN 1186-15 in conjunction with EN 1186-1.

Further Parts of this standard have been prepared concerned with the determination of overall migration from plastics materials into food simulants. Their titles are as follows:

EN 1186 *Materials and articles in contact with foodstuffs – Plastics –*

Part 1 *Guide to the selection of conditions and test methods for overall migration*

Part 2 *Test methods for overall migration into olive oil by total immersion*

Part 3 *Test methods for overall migration into aqueous food simulants by total immersion*

Part 4 *Test methods for overall migration into olive oil by cell*

Part 5 *Test methods for overall migration into aqueous food simulants by cell*

Part 6 *Test methods for overall migration into olive oil using a pouch*

Part 7 *Test methods for overall migration into aqueous food simulants using a pouch*

Part 8 *Test methods for overall migration into olive oil by article filling*

Part 9 *Test methods for overall migration into aqueous simulants by article filling*

Part 10 *Test methods for overall migration into olive oil (modified method for use in cases where incomplete extraction of olive oil occurs)*

Part 11 *Test methods for overall migration into mixtures of ^{14}C-labelled synthetic triglyceride*

Part 12 *Test methods for overall migration at low temperatures*

Part 13 *Test method for overall migration at high temperatures*

Part 14 *Test methods for 'substitute tests' for overall migration from plastics intended to come into contact with fatty foodstuffs using test media iso-octane and 95 % ethanol*

1 Scope

This European Standard specifies two alternative test methods, in the sense of an extraction test with a 'more severe' test character, for the assessment of the overall migration into fatty food simulants.

Method A is based on the determination of the extraction of migrateable substances from plastics 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 packaging material. According to results obtained by this method (see [1], [2], [3], [4], [5]) 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 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.

To ensure as complete as possible extraction of the potential migrants, a strong interaction, e.g. swelling, of the sample by the extraction solvent is necessary. For this purpose, iso-octane is used as an extraction solvent for plastics materials and articles containing non polar food contact layers, such as polyolefins. For test samples made from polar food contact plastics such as polyamide and polyethylene terephthalate, 95 % (v/v) aqueous ethanol is used. For polystyrenes, plasticized polyvinyl chloride and other polymers where the identification or polarity of the polymer is not clear, two parallel extraction tests should be conducted using both of the proposed extraction solvents and taking the higher value obtained as the relevant result.

NOTE 1 In case of multilayer structures such as plastics laminates and co-extruded plastics, the nature of the food contact layer determines the selection of the extraction solvent(s).

This test method should only be applied to flexible packagings which are less than 300 µm in thickness. When the result does not exceed the allowed overall migration limit then the material can be considered to be in compliance with EC regulations. If the test result exceeds the allowed overall migration limit the following options may be applied chronologically with respect to further migration testing:

1) single-sided extraction test using a cell, if technically feasible (see 15B of this standard);

2) conventional migration test using olive oil or other fatty food simulants;

NOTE 2 The overall migration limit is specified in Commission Directive 2002/72/EC [7] and the conditions of test in Council Directive 82/711/EEC [8] and its subsequent amendments, [9], [10].

Method B is applicable in those cases where the total immersion test, EN 1186-15 Method 15A, yields total extraction values that exceed the overall migration or may be technically unsuitable, i.e. in the case of multilayer structures, such as plastics laminates and co-extruded films. This test method should primarily only be applied to flexible packagings with a physical barrier layer (for instance of aluminium or other material to prevent penetrative loss of extraction solvent) and which have a thinner food contact layer than 300 µm. If the result does not exceed the allowed overall migration limit then the material can be considered to be in compliance with EC regulations. If the test result exceeds the allowed overall migration limit then the following option may be applied with respect to further migration testing:

- conventional migration test using olive oil or other fatty food simulants.

NOTE 3 Methods A and B are not applicable to test materials intended for applications over 130 °C.

NOTE 4 Test materials intended for applications over 70 °C should be checked for their physical suitability at the intended time and temperature of use.

2 Normative references

This European Standard 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 European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1186-1:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 1: 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 Method A
Alternative test method to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol by total immersion

3.1 Principle

The migrateable substances extracted from a sample of the plastics 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 extraction solvent for 24 h at 40 °C or 50 °C 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 (± 1 mg/dm²) into account.

3.2 Reagents

NOTE For details of preparation and quality of these reagents, see clause 4 of EN 1186-1:2002.

3.2.1 Ethanol 95 % (v/v) in aqueous solution.

3.2.2 Iso-octane (2,2,4-trimethylpentane)

NOTE The extraction solvents given in 3.2.1 and 3.2.2 are selected according to the nature of the polymer test sample as given by Table 1, see 3.5.1.

3.3 Apparatus

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

3.3.2 Tweezers, stainless steel, blunt nosed.

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

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

3.3.5 Rule, graduated in mm, and with an accuracy of 0,1 mm.

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

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

3.3.8 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 EN 1186-1:2002.

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.

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

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

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

3.3.12 Measuring cylinder, 50 ml capacity, conforming to the minimum requirements of ISO 4788.

3.3.13 Round-bottom flask, 250 ml capacity [for distillation method (see 3.5.3.3) only].

3.4 Preparation of test specimens

3.4.1 General

It is essential that test specimens are clean and free from surface contamination (many plastics 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 EN 1186-1:2002. 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.

3.4.2 Number of test specimens

Three replicate test specimens are required.

3.4.3 Cutting and preparation of specimen

Lay the sample on the cutting slab (3.3.1) and cut the test specimens of 1 dm² (see 8.3 of EN 1186-1:2002), using the 100 mm x 100 mm template (3.3.4). Check, using the rule (3.3.5), that the dimensions of the specimen are within the specified tolerance (1 mm). 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 (3.3.7).

3.5 Procedure

3.5.1 Selection of extraction solvent

Select the appropriate extraction solvent(s) (see 3.2.1 and 3.2.2) according to the nature of the polymer test sample as given in Table 1.

Table 1 — Use of extraction solvents and test conditions – Method A

Polymer type of food contact layer	Extraction solvent to be applied	Extraction conditions to be applied
Polyolefines 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 (plasticized)	iso-octane and 95 % ethanol	24 h at 40 °C
Polyvinyl chloride (rigid)	95 % ethanol	24 h at 50 °C

3.5.2 Exposure to solvent

Take three extraction containers or jars (3.3.7), measure by measuring cylinder (3.3.12) 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 (3.5.3.2) 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 (3.5.3.3) 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 according to the nature of the polymer test samples.

Place the five jars in the thermostatically controlled oven or incubator (3.3.8), set at the test temperature and observe the temperature, leave the jars for the test period of 24 h 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 EN 1186-1:2002). 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 test specimens. 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 jar. Recover at least 90 % of the original volume of solvent, including the blanks, or repeat the test.

WARNING Both iso-octane and ethanol are volatile flammable solvents. Care should be taken to avoid any loss of solvent into the interior of the thermostatting 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.

3.5.3 Determination of extracted substances

3.5.3.1 Preparation of dishes

Take five dishes (3.3.9), 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 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.

3.5.3.2 Evaporation method

For each jar, including the two blank jars, 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 (3.3.10), taking care to avoid loss of residue, in particular, by sputtering or overheating.

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. Rinse each of the jars 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 (3.3.11) 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.

3.5.3.3 Distillation method

For each jar, transfer the contents to a round bottom flask (3.3.13). 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