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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

iTeh STANDARD PREVIEW

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

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Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - 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 %

Ta slovenski standard je istoveten z: EN 1186-15:2002

ICS:

67.250 Materiali in predmeti v stiku z živili Materials and articles in contact with foodstuffs

SIST EN 1186-15:2003**en**

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 1186-15

September 2002

ICS 67.250

English version

**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 European Standard was approved by CEN on 29 April 2002.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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

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Foreword

This document EN 1186-15:2002 has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2003, and conflicting national standards shall be withdrawn at the latest by March 2003.

This European Standard has been prepared as one of a series of methods of test for plastics materials and articles in contact with foodstuffs.

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

At the time of preparation and publication of this standard the European Union legislation relating to plastics materials and articles intended to come into contact with foodstuffs is incomplete. Further Directives and amendments to existing Directives are expected which could change the legislative requirements which this standard supports. It is therefore strongly recommended that users of this standard refer to the latest relevant published Directive(s) before commencement of any of the test or tests described in this standard.

EN 1186-15 should be read 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 |

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

EN 1186-15:2002 (E)**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 clause 4 Method B 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 90/128/EEC [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 A, 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.

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3.2 Reagents

NOTE For details of preparation and quality of these reagents, see clause 5 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.

EN 1186-15:2002 (E)**3.3 Apparatus**

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

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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 9.1 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 9.3 of EN 1186-1:2002), using the 100 mm × 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 <u>and</u> 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.