



# SLOVENSKI STANDARD SIST EN 1186-14:2003

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Materials and articles in contact with foodstuffs - Plastics - 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

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Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 14: Prüfverfahren für "Ersatzprüfungen" für die Gesamtmigration aus Kunststoffen, die für den Kontakt mit fettigen Lebensmitteln bestimmt sind, unter Verwendung der Prüfmedien iso-Octan und 95 % Ethanol

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 14: Méthodes de remplacement pour la vérification de la migration globale des matières plastiques en contact avec des denrées alimentaires grasses dans l'iso-octane et l'éthanol a 95 %

**Ta slovenski standard je istoveten z: EN 1186-14:2002**

**ICS:**

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

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
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**EN 1186-14**

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ICS 67.250

English version

**Materials and articles in contact with foodstuffs - Plastics - 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**

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 14: Méthodes d'essai pour des 'tests de substitution' pour la migration globale des matières destinées au contact avec les denrées alimentaires gras en utilisant des liquides simulateurs tels que l'isooctane et l'éthanol à 95 %

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 14: Prüfverfahren für "Ersatzprüfungen" für die Gesamtmigration aus Kunststoffen, die für den Kontakt mit fettigen Lebensmitteln bestimmt sind, unter Verwendung der Prüfmedien Iso-Octan und 95 %igem 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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



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

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## Foreword

This document EN 1186-14 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 document supersedes ENV 1186-14:1999.

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

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

**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.**

EN 1186-14 should be read in conjunction with EN 1186-1, EN 1186-3, EN 1186-5, EN 1886-7, and EN 1186-9.

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 <sup>14</sup> C-labelled synthetic triglyceride                                  |
| Part 12 | Test methods for overall migration at low temperatures   |
| Part 13 | Test methods for overall migration at high temperatures  |

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Part 15 Alternative test methods to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol

Annex A is informative.

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.

**1 Scope**

This European Standard specifies test methods for 'substitute tests' performed with volatile test media, iso-octane and 95 % v/v aqueous ethanol, for the determination of overall migration from plastics intended to come into contact with fatty foodstuffs at all temperatures and for any period of time.

These test methods are suitable for plastics samples in a wide variety of forms.

NOTE 1 The iso-octane and 95 % v/v aqueous ethanol volatile test media used in these test methods are those specified for 'substitute tests' in Commission Directive 82/711/EEC [3] and its subsequent amendments [4], [5]. In addition to the use of iso-octane and 95 % v/v aqueous ethanol as test media for 'substitute tests', Commission 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. For a test method for overall migration from plastics intended to come into contact with fatty foodstuffs using modified polyphenylene oxide as a test medium, see EN 1186-13.

NOTE 2 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 95 % aqueous ethanol, provided equivalence is shown with olive oil, see EN 1186-1:2002.

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**2 Normative references** <https://standards.iteh.ai/catalog/standards/sist/c91130ac-8eac-420c-ac4b-07d4f7d7ccd4/sist-en-1186-14-2003>

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.*

EN 1186-3:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 3: Test methods for overall migration into aqueous food simulants by total immersion.*

EN 1186-5:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 5: Test methods for overall migration into aqueous food simulants by cell.*

EN 1186-7:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 7: Test methods for overall migration into aqueous food simulants using a pouch.*

EN 1186-9:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 9: Test methods for overall migration into aqueous food simulants by article filling.*

EN 10088-1:1995, *Stainless steels - Part 1: List of stainless steels.*

ISO 648, *Laboratory glassware - One mark pipettes.*

ISO 4788, *Laboratory glassware - Graduated measuring cylinders.*

### 3 Total immersion method

#### 3.1 Principle

The overall migration of non-volatile substances from a sample of the plastics material or article into the test medium is determined as the mass of non-volatile residue after evaporation of the test medium following immersion.

The selection of the conditions of test is determined by the conditions of use, see clauses 6 and 7 of EN 1186-1:2002.

Test specimens of approximately 1 dm<sup>2</sup>, see clause 9 of EN 1186-1:2002, are immersed in a test medium for set periods of time and at set temperatures. At the end of the test period, each test specimen is removed from the test medium. The test medium from each test is evaporated to dryness, the mass of the non-volatile residue is determined gravimetrically and expressed as milligrams per square decimetre of surface area of test specimen.

Overall migration is reported as the mean of three determinations on separate test specimens.

#### 3.2 Reagents

**3.2.1** Iso-octane, (2,2,4-trimethyl pentane), purity 98,5 % (v/v) or greater, CAS No. 540-84-1<sup>1</sup>.

**3.2.2** Ethanol, purity 96 % (v/v) or greater, 95 % (v/v) in aqueous solution.

**WARNING** — Both these solvents are flammable. Take care at all times when handling these solvents to prevent contact with sources of ignition.

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#### 3.3 Apparatus

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**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 x 100 mm ± 0,2 mm (square).

**3.3.5** Rule or template, 25 mm ± 1 mm wide.

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

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

**3.3.8** Specimen supports, constructed of stainless steel with cross arms attached by welding or silver soldering, or of glass. Stainless steel X4 CrNi 18 10 according to EN 10088-1:1995 or of composition, chromium 17 %, nickel 9 %, carbon 0,04 %, is suitable. Before initial use thoroughly clean the stainless steel supports. The use of a degreasing solvent and then with dilute nitric acid has been found to be suitable.

**NOTE** The method has been written for the supports shown in Figure C.1 of prEN 1186-1:2002 which have been found to be suitable for holding thin film and sheet test pieces. However other supports can be used providing they are capable of

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<sup>1</sup>The source of this is the Chemical Abstracts published by the American Chemical Society

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holding and keeping the test pieces apart and at the same time ensuring complete contact with the test medium. For rigid samples, supports with a single cross arm can be used.

**3.3.9** Gauze, pieces of fine stainless steel gauze, with a mesh size of 1 mm have been found to be suitable, approximately 25 mm × 100 mm or, glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long for insertion between the test pieces. Before initial use thoroughly clean the gauze, first with a degreasing solvent and then with dilute nitric acid.

**3.3.10** Glass tubes, ground neck with stoppers, for retaining the test medium and test specimens. Tubes with an internal diameter of approximately 35 mm and length in the range of 100 mm to 200 mm, excluding the ground neck, see 8.2 of EN 1186-1:2002, have been found to be satisfactory.

**3.3.11** Glass beads, 2 mm to 3 mm diameter, or glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long, see 8.2 of EN 1186-1:2002.

**3.3.12** Thermostatically controlled oven, incubator or refrigerator capable of maintaining a temperature within the range of 5 °C to 60 °C and within the tolerances specified in Table B.2 of EN 1186-1:2002.

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

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

**3.3.14** Steam bath, hot plate, distillation apparatus or rotary evaporator for evaporation of test medium at the end of test period.

**3.3.15** Desiccator with anhydrous calcium chloride or self indicating silica gel.

**3.3.16** Measuring cylinder, 100 ml, complying with the minimum requirements of ISO 4788.

**3.3.17** Thermometer or electronic temperature measuring instrument with thermocouple, capable of measuring temperature in the range 5 °C to 60 °C, with a precision of 0,1 °C.

### **3.4 Preparation of test specimens**

Prepare the test specimens in accordance with 3.4 of EN 1186-3:2002.

#### **3.4 Procedure**

##### **3.4.1 Exposure to test media**

Take three of the glass tubes, for the test specimens and a further two to provide blanks, measure by measuring cylinder 100 ml ± 2 ml of the test medium into each tube and stopper the tube. If the evaporation method is to be used measure into a further two tubes, by measuring cylinder, 120 ml ± 2 ml of the test medium, to provide blanks. If the distillation method is to be used measure into those further two tubes by measuring cylinder 100 ml ± 2 ml of the test medium to provide blanks. Insert a thermometer or thermocouple in one of the tubes containing 100 ml of test medium, then stopper all five tubes. Place the five tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature, and leave until the test medium has attained the test temperature.

Place a test specimen into each of the three tubes containing 100 ml of test medium, re-insert the thermometer or thermocouple and re-stopper the tubes. Mark the tubes for identification. Ensure that the test specimens are totally immersed in the test medium; if they are not then add either glass beads or rods to raise the level of the test medium until total immersion is achieved. This part of the operation should be carried out in the minimum time to prevent undue heat loss from the test medium.



Mark the liquid level on the outside of each tube with a suitable marker.

Replace all of the tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature. Observe the temperature and leave the tubes for the selected test period, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002, after the temperature in the tube has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

**WARNING 1 — Both iso-octane and ethanol are volatile flammable solvents. Take care to ensure that the tubes are well stoppered to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator and generating an explosive mixture.**

**WARNING 2 — If possible place the tubes in a drip container capable of holding the total volume of volatile simulant in case of accident.**

**WARNING 3 — To minimise hazards arising due to the volatile and flammable nature of the two test media the maximum test temperature is 60 °C. Do not conduct the tests at temperatures above 60 °C.**

Take the tubes from the oven, incubator or refrigerator and check the level of test medium in each, if this has fallen to more than 10 mm below the mark, or has exposed any part of the test pieces, repeat the test using fresh test specimens.

NOTE For exposure times of more than 24 h it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator, instead of the temperature of the simulant.

If the level of test medium in a tube is less than 10 mm below the mark, remove the test specimen from the tube, and allow the test medium adhering to the test specimen and support to drain back into the tube. Recover at least 90 % of the original volume of test medium or repeat the test.

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### 3.4.2 Determination of migrating substances

Determine the migrating substances in accordance with 3.5 of EN 1186-3:2002.

**WARNING — Both iso-octane and ethanol are volatile and flammable solvents. Take care when evaporating these test media to prevent vapours contacting sources of ignition, particularly when using a hot plate to carry out the evaporation. The evaporation should be carried out in a fume cupboard.**

### 3.5 Expression of results

Calculate the results in accordance with 3.6 of EN 1186-3:2002.

### 3.6 Test report

The test report shall include the following, see clause 12 of EN 1186-1:2002:

- a) reference to this European Standard and to the Part used for the test procedure;
- b) all information necessary for complete identification of the sample such as chemical type, supplier, trade mark, grade, batch number, thicknesses;
- c) conditions of time and temperature of exposure to test media;
- d) departures from the specified procedure, and reasons for these;
- e) individual test results, and the mean of these, expressed as milligrams of residue per square decimetre of sample;
- f) relevant comments on the test results.

**EN 1186-14:2002 (E)****4 Cell method****4.1 Principle**

The overall migration of non-volatile substances from a sample of the plastics material or article into the test medium, is determined as the mass of non-volatile residue after evaporation of the test medium.

The selection of the conditions of test are determined by the conditions of use, see clauses 6 and 7 of EN 1186-1:2002.

One surface of the test specimen is exposed in a cell to a test medium for set periods of time and at set temperatures. At the end of the test period, each test specimen is removed from contact with the test medium. The test medium from each test is evaporated to dryness, the mass of the non-volatile residue is determined gravimetrically and expressed as milligrams per square decimetre of surface area of test specimen, which had been in contact with the test medium.

Overall migration is reported as the mean of three determinations on separate test specimens.

**4.2 Reagents**

**4.2.1** Iso-octane (2,2,4-trimethyl pentane), purity 98,5 % (v/v) or greater, CAS No. 540-84-1.

**4.2.2** Ethanol, purity 96 % (v/v) or greater, 95 % (v/v) in aqueous solution.

**WARNING** — Both these solvents are flammable. Take care at all times when handling these solvents to prevent contact with sources of ignition.

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**4.3 Apparatus**

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

**4.3.2** Tweezers, stainless steel, blunt nosed.

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

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

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

**4.3.6** Cell type A, as shown in Figure C.3 of EN 1186-1:2002, either the all aluminium (anodised) cells or the cells with the stainless steel (316 grade) lids and rings. The internal diameter of the rib of the sealing ring shall be 178,4 mm ± 0,1 mm, to give an area of the test specimen exposed to the test medium of 2,5 dm<sup>2</sup>. It is necessary to modify the filler plug on one of the cells to allow a thermometer or thermocouple to be inserted into the test medium, whilst maintaining an effective seal to prevent loss of test medium during the test period.

**NOTE** The cell type A is constructed with a rubber mat in the base plate. It is advised that a disc of aluminium foil is placed on the mat before inserting the test specimen. The use of these discs will prevent any substances from the mat influencing the migration result.

For details of equivalent cells see 8.3 of EN 1186-1:2002.

**4.3.7** Pipettes, complying with the minimum requirements of ISO 648, 50 ml and 100 ml.

**4.3.8** Glass tubes, ground neck, and stoppers, for retaining the test medium. Tubes with an internal diameter of approximately 35 mm and length of approximately 100 mm to 200 mm, excluding the ground neck, see 8.2 of EN 1186-1:2002, have been found to be satisfactory.

**4.3.9** Thermostatically controlled oven, incubator or refrigerator capable of maintaining a temperature within the range 5 °C to 60 °C and within the tolerances specified in Table B.1 of EN 1186-1:2002.

**WARNING — The interior/sample space of the oven, incubator or refrigerator should not have any exposed heating elements, to minimise safety hazards arising from any loss of the flammable test media from the tubes and cells.**

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

**4.3.11** Steam bath, hot plate, distillation apparatus or rotary evaporator for evaporation of test medium at the end of test period.

**4.3.12** Desiccator with anhydrous calcium chloride or self indicating silica gel.

**4.3.13** Measuring cylinders, 250 ml, complying with the minimum requirements of ISO 4788.

**4.3.14** Thermometer or electronic temperature measuring instrument, with thermocouple, capable of measuring temperature in the range 5 °C to 60 °C, with precision of 0,1 °C.

#### 4.4 Preparation of test specimens

Prepare the test specimens in accordance with clause 6 of EN 1186-5:2002.

#### 4.5 Procedure

##### 4.5.1 Exposure to test media

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Take three cells, mark these for identification purposes. Place in the thermostatically controlled oven, incubator or refrigerator, which is set at the selected test temperature and leave until the test temperature has been attained.

Take three glass tubes, measure by measuring cylinder 125 ml  $\pm$  2 ml of the test medium into each tube, stopper the tube and mark the level of the liquid on the outside, together with a mark for identification. If the evaporation method is to be used measure into a further two tubes by measuring cylinder 185 ml  $\pm$  2 ml of the test medium, to provide blanks. If the distillation method is to be used measure into a further two tubes by measuring cylinder 125 ml  $\pm$  2 ml of the test medium to provide blanks.

Mark the liquid level on the outside of each tube with a suitable marker. Insert a thermometer or thermocouple in one of the tubes containing 125 ml of test medium, then stopper all five tubes. Place the five tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature, and leave until the test medium has attained the test temperature.

Remove the cells from the thermostatically controlled oven, incubator or refrigerator, dismantle and place on the base of each cell one of the test specimens. Reassemble the cells, ensuring that the clamping screw wheel is well tightened down.

Remove three tubes containing 125 ml of test medium from the thermostatically controlled oven, incubator or refrigerator and transfer the test medium from each tube to each of the test cells through the filler hole.

Replace the filler plugs and in one of the cells insert the thermometer or thermocouple. This part of the operation should be carried out in the minimum of time to prevent undue heat loss from the test medium. Carefully check each cell for any leakage of simulant. If any leakage is observed reject that cell from further tests.

**WARNING 1 — Never place a leaking cell in the oven.**

Return the test cells to the thermostatically controlled oven, incubator or refrigerator, set at the test temperature. Observe the temperature and leave the cells and tubes for the selected test period, taking into account the