



# SLOVENSKI STANDARD SIST ENV 1186-14:2000

01-april-2000

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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

**iTeh STANDARD PREVIEW**

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 Isooctan und 95 % Ethanol

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

**Ta slovenski standard je istoveten z: ENV 1186-14:1999**

**ICS:**

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

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EUROPEAN PRESTANDARD  
PRÉNORME EUROPÉENNE  
EUROPÄISCHE VORNORM

**ENV 1186-14**

March 1999

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 - Matières plastiques - 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 %

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This European Prestandard (ENV) was approved by CEN on 18 February 1999 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, 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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REPUBLIKA SLOVENIJA  
AGENCIJA REPUBLIKE SLOVENIJE  
ZA KAZENSKO PRAMENIŠTVO  
IN VARNOST  
REPUBLIKA SLOVENIJA  
AGENCIJA REPUBLIKE SLOVENIJE  
ZA KAZENSKO PRAMENIŠTVO  
IN VARNOST

## Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

Further Parts of this prestandard have been prepared, and others are in preparation, concerned with the determination of overall migration from plastics materials into food simulants.

Their titles are as follows:

- ENV 1186-1 Guide to the selection of conditions and test methods for overall migration
- ENV 1186-2 Test methods for overall migration into olive oil by total immersion
- ENV 1186-3 Test methods for overall migration into aqueous food simulants by total immersion
- ENV 1186-4 Test methods for overall migration into olive oil by cell
- ENV 1186-5 Test methods for overall migration into aqueous food simulants by cell
- ENV 1186-6 Test methods for overall migration into olive oil using a pouch
- ENV 1186-7 Test methods for overall migration into aqueous food simulants using a pouch
- ENV 1186-8 Test methods for overall migration into olive oil by article filling
- ENV 1186-9 Test methods for overall migration into aqueous simulants by article filling
- ENV 1186-10 Test methods for overall migration into olive oil (modified method for use in cases where incomplete extraction of olive oil occurs)
- ENV 1186-11 Test methods for overall migration into mixtures of <sup>14</sup>C-labelled synthetic triglyceride
- ENV 1186-12 Test methods for overall migration at low temperatures

Further Parts in preparation are as follows:

- ENV 1186-13 Test methods for overall migration at high temperatures

Annex A to this prestandard is normative where applicable.

ENV 1186-14 should be read in conjunction with EN 1186-1.

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

## 1 Scope

This Part of this European Prestandard describes 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.

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 and its subsequent amendments.

The permitted use of 'substitute tests' with volatile test media iso-octane and 95 % v/v aqueous ethanol is specified in Commission Directive 82/711/EEC and its subsequent amendments.

NOTE 1: 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 ENV 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.

## 2 Normative references

This European Prestandard 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 Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 648:1977 Laboratory glassware - One mark pipettes

ISO 4788:1980 Laboratory glassware - Graduated measuring cylinders

ISO 8442:1988 Stainless steel and silver plated table cutlery <sup>1</sup>

EN 1186-1:1994 Guide to the selection of conditions and test methods for overall migration

ENV 1186-3:1994 Test methods for overall migration into aqueous food simulants by total immersion

ENV 1186-5:1994 Test methods for overall migration into aqueous food simulants by cell

ENV 1186-7:1994 Test methods for overall migration into aqueous food simulants using a pouch

ENV 1186-9:1994 Test methods for overall migration into aqueous food simulants by article filling

prEN 10088-4 Stainless steels - Technical delivery conditions for sheet/plate and strip for boilers and pressure vessels

<sup>1</sup>

A European Standard for stainless steel and silver plated cutlery is in the course of preparation

### 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 will be determined by the conditions of use, see clause 4, 5 and 6 of EN 1186-1:1994.

Test specimens of approximately 1 dm<sup>2</sup>, see clause 9 of EN 1186-1:1994, are immersed in a test medium for periods of time and at temperatures specified in Council Directive 82/711/EEC, and its subsequent amendments. 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>2</sup>

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

WARNING: Both these solvents are flammable. Care should be taken at all times when handling these solvents to prevent contact with sources of ignition.

#### 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 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 prEN 10088-4 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 D.1 of EN 1186-1 which have been found to be suitable for holding thin film and sheet test pieces. However other supports may be used providing they are capable of 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 may 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 x 100 mm or, glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long for

<sup>2</sup>

The source of this is the Chemical Abstracts published by the American Chemical Society

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 1186-1:1994, 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 1186-1:1994.

3.3.12 Thermostatically controlled oven, incubator or refrigerator capable of maintaining a temperature within the range of 5 °C to 60 °C and meeting temperature tolerance values within those specified for the test temperature, see annex A.

**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  $\pm 0,5$  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 clause 6 of ENV 1186-3:1994.

### 3.5 Procedure

#### 3.5.1 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  $\pm$  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  $\pm$  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  $\pm$  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 period of time after the temperature in



the tube has reached a temperature within the permitted tolerance for the test temperature, see annex A for permitted tolerances on test times and temperatures.

**WARNING 1:** Both iso-octane and ethanol are volatile flammable solvents. Care should be taken 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. On no account should tests be conducted 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.

### 3.5.2 Determination of migrating substances

Determine the migrating substances in accordance with 7.2 of ENV 1186-3:1994.

**WARNING:** Both iso-octane and ethanol are volatile and flammable solvents. Care should be taken 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.

## 3.6 Expression of results

Calculate the results in accordance with clause 8 of ENV 1186-3:1994.

## 3.7 Test report

The test report shall include the following, see clause 12 of EN 1186-1:1994:

- a) reference to this European Prestandard 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.

## 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 will be determined by the conditions of use, see clause 4,5 and 6 of EN 1186-1:1994.

One surface of the test specimen is exposed in a cell to a test medium for periods of time and at temperatures specified in Council Directive 82/711/EEC and its subsequent amendments. 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. Care should be taken 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 x 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 D.3 of EN 1186-1, 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  $\pm$  0,1 mm, to give an area of the test specimen exposed to the test medium of 2,5 dm<sup>2</sup>. It will be 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 1186-1:1994.

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 1186-1:1994, 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 meeting temperature tolerance values within those specified for the test temperatures, see annex A.

**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 ENV 1186-5:1994.

#### 4.5 Procedure

##### 4.5.1 Exposure to test media

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 for further tests.

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