

SLOVENSKI STANDARD

SIST ENV 1186-9:1997

01-januar-1997

Materiali in predmeti v stiku z živilni - Plastične mase - 9. del: Preskusne metode za celotno migracijo v modelno vodno raztopino, s katero je napolnjen predmet

Materials and articles in contact with foodstuffs - Plastics - Part 9: Test methods for overall migration into aqueous food simulants by article filling

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 9: Prüfverfahren der Gesamtmigration in wäßrige Prüflebensmitteln durch Füllen des Gegenstandes

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Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 9: Méthodes d'essai pour la migration globale dans les liquides simulateurs aqueux par remplissage

Ta slovenski standard je istoveten z: **ENV 1186-9:1994**

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

ENV 1186-9

PRÉNORME EUROPÉENNE

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Descriptors: plastic products, materials, plastics, food products, food-container contact, determination, migrations, tests, simulation, liquids, filling

English version

**Materials end articles in contact with foodstuffs -
Plastics - Part 9 : Test methods for overall
migration into aqueous food simulants by article
filling**

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Matériaux et objets en contact avec les denrées
alimentaires - Matière plastique - Partie 9 :
Méthodes d'essai pour la migration globale dans
les liquides simulateurs aqueux par remplissage

Werkstoffe und Gegenstände in Kontakt mit
Lebensmitteln - Kunststoffe - Teil 9 :
Prüfverfahren der Gesamtmigration in wässrigen
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REPUBLIKA SLOVENIJA
MINISTRSTVO ZA ZNANOST IN TEHNOLOGIJO
Urad RS za standardizacijo in meroslovje
LJUBLJANA

SIST... ENV 1186-9

PREVZET PO METODI RAZGLASITVE

-01- 1997

This European Prestandard (ENV) was approved by CEN on 1993-03-09 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 an European Standard (EN).

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Foreword

This Part of this European Prestandard has been prepared by a Subcommittee (SC1) of TC194 'Utensils in contact with food' as one of a series of methods of test for plastics materials and articles in contact with foodstuffs.

Further Parts of this prestandard have been prepared, and 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
<https://standards.iteh.ai/catalog/standards/sist/d5a05149-f803-4d0b-b737-806665816a82/sist-env-1186-9-1997>
- 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 1186-10 Test methods for overall migration into olive oil (modified method for use in cases where incomplete extraction of olive oil occurs)

Further Parts in preparation are as follows;

- ENV 1186-11 Test methods for overall migration into mixtures of ¹⁴C-labelled synthetic triglyceride
- ENV 1186-12 Test methods for overall migration at low temperatures
- ENV 1186-13 Test methods for overall migration at high temperatures

ENV 1186-9 should be read in conjunction with ENV 1186-1.

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

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1 Scope

This Part of this European Prestandard describes a method of test for the determination of the overall migration into aqueous based food simulants from plastics which are intended to come into contact with foodstuffs, by filling articles with aqueous food simulant for 10 days, 24 h or 2 h at 40 °C or for 2 h at 70 °C.

This method is most suitable for plastics in the form of containers and articles that can be filled.

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

ENV 1186-1 Guide to the selection of conditions and test methods for overall migration

3 Principle

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The overall migration of non-volatile substances from a sample of the plastics is determined as the mass of non-volatile residue after evaporation of the food simulants following filling the test specimen.

The selection of the conditions of test will be determined by the conditions of use, see clause 3 of ENV 1186-1.

Test specimens are filled with the food simulant for either 10 d, 24 h or 2 h at 40 °C or for 2 h at 70 °C. After exposure to the simulant the test specimen is emptied and the food simulant evaporated to dryness. The mass of the non-volatile residue is determined and expressed as milligrams per square decimetre of surface area exposed to the food simulant.

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

4 Reagents

For details of the preparation and purity of these reagents see clause 4 of ENV 1186-1.

4.1 Distilled water or water of equivalent quality (simulant A)

- 4.2 Acetic acid 3 % (w/v) in aqueous solution (simulant B)
- 4.3 Ethanol 15 % (v/v) in aqueous solution (= simulant C)
- 4.4 Alcoholic simulants for liquids or beverages of an alcoholic strength exceeding 15 % v/v.

NOTE: In the case of materials and articles intended to come into contact with liquids or beverages of an alcoholic strength exceeding 15 % v/v, the test may be carried out with aqueous solutions of ethanol of a similar strength.

5 Apparatus

- 5.1 Analytical balance capable of determining a change in mass of 0,1 mg.
- 5.2 Lint-free cloth.
- 5.3 Beaker, 2 l.
- 5.4 Glass beads, 2 mm to 3 mm diameter.
- 5.5 Thermostatically controlled oven or incubator capable of maintaining a temperature of $40\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ and $70\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.
- 5.6 Dishes, stainless steel, nickel, platinum, platinum alloy, gold 50 mm to 90 mm diameter and maximum weight 100 g, for evaporation of food simulants 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 food simulants followed by conditioning in the desiccator used, achieves a constancy of $\pm 0,5$ mg. Stainless steel and nickel dishes are suitable only for distilled water and ethanol solutions. Glass, glass ceramic, glazed ceramic, platinum, platinum alloy or gold dishes are suitable for all three simulants.
- 5.7 Steam bath, hot plate, distillation apparatus or rotary evaporator for evaporation of food simulant at the end of test period.
- 5.8 Desiccator with anhydrous calcium chloride or self indicating silica gel.
- 5.9 Beakers, 250 ml.
- 5.10 Pipette, 200 ml, complying with the minimum requirements of ISO 648.

6 Preparation of test specimens

6.1 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 6.1 of ENV 1186-1. Minimise handling of the samples and where necessary, wear cotton gloves.

6.2 Number of test specimens

6.2.1 Volume of articles

Determine and record the volume of food simulant required to fill an article, see 6.2 of ENV 1186-1.

6.2.2 Articles with a nominal volume of more than 200 ml

Five articles are required to provide five test specimens. These test specimens are utilized as follows:

- a) Three test specimens for the migration test;
- b) Two test specimens for the determination of surface area.

6.2.3 Articles with a nominal volume of less than 200 ml

The number of articles required to provide a test specimen is dependant on their volume. A test specimen shall be made up of sufficient articles to contain 200 ml of the food simulant.

Five test specimens are required. These test specimens are utilized as follows:

- a) Three test specimens for the migration test;
- b) Two test specimens for the determination of surface area.

Record the number of articles used to provide the test specimen.

6.3 Determine and record the surface area of the test specimen exposed to food simulant when it is filled to its nominal volume.

NOTE: In the case of articles with a volume of less than 200 ml this will be the surface area of one article multiplied by the number of articles used to provide a test specimen.

6.4 Articles with a capacity of not less than 500 ml and not more than 10 l.

It is not necessary to determine the volume of these articles since the migration must be expressed in mg/kg of food simulant,

7 Procedure

7.1 Exposure to food simulant

Mark each of the articles making up each test specimen with an identification

code.

Place, in a beaker, a sufficient volume of the food simulant to fill the three test specimens prewarmed if necessary and to provide two blanks in the thermostatically controlled oven or incubator, set at the test temperature and leave until the test temperature has been attained.

Remove the beaker containing the food simulant from the thermostatically controlled oven or incubator. Fill the three test specimens with simulant to within 0,5 cm of the top. If the container has a specified nominal volume of contents see 6.2 of ENV 1186-1. Cover the test specimens and the remaining simulant with an inert material to prevent evaporation, eg glass. This part of the operation should be carried out in the minimum time to prevent undue heat loss from the simulant.

Place the test specimens and food simulant in the thermostatically controlled oven or incubator set at the test temperature, leave for a test period of
 $+5$ $+0,5$ $+5$
 240 0 h or 24 0 h or 120 0 min, after the air bath of the thermostatically controlled oven or incubator has reached a temperature within 1 °C of the set temperature.

NOTE: Were the surface of simulant is large a check should be made to ensure that excessive loss of simulant by evaporation does not occur.

7.2 Determination of migrating substances

7.2.1 Preparation of dishes

Take five dishes (5.6) marked for identification, place the dishes in an oven maintained at 105 °C to 110 °C, for a period of 30 min \pm 5 min, to dry.

Remove the dishes from the oven, place in a desiccator (5.8) 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 no more than 0,5 mg, record their masses.

7.2.2 Evaporation method

Take the test specimens and measure, by pipette, 200 ml of the simulant into separate 250 ml beakers, ensuring that the simulant is mixed.

Measure aliquots of 200 ml of the food simulant into two more beakers to provide blanks, which are handled in the same way as simulants incontact with the test specimens.

By means of a steam bath, hot plate or other form of heating (5.7) evaporate to a low volume, taking care to ensure that only mild boiling occurs to avoid loss, in particular by sputtering, or overheating of the residues.