



# SLOVENSKI STANDARD

## SIST ENV 1186-4:1997

01-januar-1997

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### Materiali in predmeti v stiku z živilni - Plastične mase - 4. del: Preskusne metode za celotno migracijo v olivno olje z migracijsko celico

Materials and articles in contact with foodstuffs - Plastics - Part 4: Test methods for overall migration into olive oil by cell

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 4: Prüfverfahren der Gesamtmigration in Olivenöl mittels Zelle

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 4: Méthodes d'essai pour la migration globale dans l'huile d'olive en cellule

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#### **ICS:**

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

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English version

**Materials and articles in contact with foodstuffs -  
Plastics - Part 4 : Test methods for overall  
migration into olive oil by cell**

Matériaux et objets en contact avec les denrées  
alimentaires - Matière plastique - Partie 4 :  
Méthodes d'essai pour la migration globale dans  
l'huile d'olive en cellule

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Lebensmitteln - Kunststoffe - Teil 4 :  
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mittels Zelle



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REPUBLIKA SLOVENIJA  
MINISTRSTVO ZA ZNANOST IN TEHNOLOGIJO  
Urad RS za standardizacijo in meroslovje  
LJUBLJANA

SIST... ENV 1186-4  
PREVZET PO METODI RAZGLASITVE

-01- 1997

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# CEN

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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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 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 1186-5 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  
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- ENV 1186-7 Test methods for overall migration into aqueous food simulants using a pouch  
<https://standards.iteh.ai/catalog/standards/sist/b5eb6c76-3471-4b94-80eb-29-94557e1/sist-env-1186-4-1997>
- 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)

Further Parts in preparation are as follows:

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

ENV 1186-4 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.

## 1 Scope

This Part of this European Prestandard describes tests methods for the determination of the overall migration from one surface only of plastics in the form of sheet and film, which are intended to come into contact with fatty foodstuffs by exposure to olive oil in a standard cell or equivalent cell 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 films and sheets, but is particularly applicable to those materials consisting of more than one layer or of surfaces that differ in their migration characteristics, which must be tested with the food simulant in contact only with the surface which is intended to come into contact with foodstuffs.

The test method described is applicable to most types of plastics, although there are some plastics for which it is known not to be applicable, for example, owing to incomplete extraction of olive oil; for examples of these see 7.2 of ENV 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.

- [SIST ENV 1186-4:1997](http://standards.ohio.gov/standards/oh/b5eb6c76-3d71-4b94-80eb-291945553be1/sist-env-1186-4-1997)
- ISO 648:1977 [Laboratory glassware - One mark pipettes](http://standards.ohio.gov/standards/oh/b5eb6c76-3d71-4b94-80eb-291945553be1/sist-env-1186-4-1997)
- ISO 478:1980 Laboratory glassware - Graduated measuring cylinders
- ENV 1186-1 Guide to the selection of conditions and test methods for overall migration

## 3 Principle

The overall migration from a sample of the plastics is determined as the loss in mass per unit of surface area intended to come into contact with foodstuffs.

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

Test specimens of known mass are exposed in a standard cell to olive oil for 10 days, 24 h or 2 h at 40 °C or for 2 h at 70 °C then taken from the olive oil, blotted to remove oil adhering to the surface and reweighed.

The specimens will usually retain absorbed olive oil which is extracted and determined quantitatively by means of gas chromatography after conversion to methyl esters. Methylation is carried out by reacting a boron

trifluoride/methanol complex with fatty acids formed by hydrolysing the oil with potassium hydroxide. An internal standard, triheptadecanoin, is added prior to the extraction of the absorbed olive oil from the test specimens. This ensures that any active or extractable components of the plastics react with the internal standard, as well as with the extracted olive oil. The internal standard is also subjected to the hydrolysis and methylation reactions, providing compensation for any inefficiencies in the hydrolysis and methylation processes.

Migration into the olive oil is calculated by subtracting the mass of olive oil retained by the test specimen from the mass of the test specimen after removal from the olive oil, and then subtracting this mass from the initial mass of the specimen.

The total loss in mass is expressed in milligrams per square decimetre of surface area of the specimen and the overall migration is reported as the mean of a minimum of three determinations on separate test specimens.

To allow for inaccuracies which may arise during the procedure and which may be difficult to detect, due for example to contamination or loss of oil during the sample handling stages, quadruplicate determinations are carried out on the sample allowing for the result from one specimen to be discarded.

This method includes variations which are applicable to certain plastics and to experienced laboratories.

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### 4 Reagents (standards.iteh.ai)

All reagents shall be of recognized analytical quality, unless otherwise specified. <https://standards.iteh.ai/catalog/standards/sist/b5eb6c76-3471-4b94-80eb-291945553e1/sist-env-1186-4-1997>

4.1 Olive oil, Simulant D as specified in 4.2. of ENV 1186-1.

4.2 Extraction solvent (see 7.1 of ENV 1186-1).

4.2.1 Pentane 98 % (mixed isomers) boiling point 36 °C

NOTE 1: Pentane is a very volatile and highly flammable solvent. Care should therefore be taken when handling this solvent to prevent contact with sources of ignition. It is not recommended that extractions with this solvent be left unattended, particularly overnight.

NOTE 2: Due to the low boiling point of this solvent, cooled condenser water may be required to prevent undue loss of the solvent from the condenser.

OR

4.2.2 Other suitable solvent.

NOTE 1: In previous methods for determining overall migration into olive oil the extraction solvent used has been 1,1,2 trichloro

trifluoroethane. For environmental reasons the use of this solvent should be avoided where possible, see 7.1. of ENV 1186-1 Experience has shown that this solvent although effective for most plastics requires longer periods of extraction.

NOTE 2: Some solvents may contain non-volatile substances which, after hydrolysis and methylation processes, produce gas chromatography peaks with retention times similar to the retention times of olive oil methyl esters and methyl heptadecanoate from the internal standard. Solvents found to contain such substances should be redistilled before use.

4.3 Internal standard, triheptadecanoin (glyceryl trimargarate) CAS No. 2438-40-6 of a quality such that the products from hydrolysis and methylation processes do not contain substances giving detectable gas chromatography peaks (see 7.3 of ENV 1186-1) with similar retention times to the olive oil methyl ester peaks. Prepared as a solution containing 2,0 mg/ml in n-heptane.

4.4 Potassium hydroxide solution, 11,0 g/l in methanol.

4.5 Boron trifluoride, methanol complex, approximately 150 g/l  $\text{BF}_3$ .

4.6 n-Heptane.

4.7 Sodium sulphate.

4.7.1 Sodium sulphate, anhydrous,  $\text{Na}_2\text{SO}_4$ .

4.7.2 Sodium sulphate, saturated solution.

<https://standards.iteh.ai/catalog/standards/sist/b5eb6c76-3471-4b94-80eb-291945553e1/sist-env-1186-4-1997>

## 5 Apparatus

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

5.2 Tweezers, stainless steel, blunt nosed.

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

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

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

5.6 Standard cells, type A as shown in figure B3 of ENV 1186-1. The internal diameter of the rib of the sealing ring should be  $178,4 \text{ mm} \pm 0,1 \text{ mm}$ , to give an area of the test specimen exposed to the food simulant of  $2,5 \text{ dm}^2$ .

For details of equivalent cells see 6.5 of ENV 1186-1.

5.7 Conditioning containers, for conditioning test specimens at  $50 \% \pm 5 \%$  relative humidity and  $80 \% \pm 5 \%$  relative humidity at  $20 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$ .

\*) The source of this is Chemical Abstracts, published by the American Chemical Society



NOTE: For 50 % relative humidity, 43 % w/v sulphuric acid solution in water is suitable and for 80 % relative humidity, 27 % w/v sulphuric acid solution are suitable. The solutions should be freshly prepared by adding the weighed amount of acid to a suitable volume of water, cooling to room temperature and making up to the required volume.

5.8 Glass tubes, ground neck and stoppers, for retaining the olive oil and test specimens. Tubes with an internal diameter of approximately 35 mm and length in the range 100 mm to 200 mm, excluding the ground neck (see 5.2 of ENV 1186-1) have been found to be satisfactory.

5.9 Thermostatically controlled oven or incubator capable of maintaining a temperature of  $40\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$  and  $70\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ .

5.10 Filter paper, lint-free.

5.11 Anti-bumping beads.

5.12 Soxhlet type extractors, capable of holding test specimens on the supports, with 250 ml or 500 ml round bottom flasks to fit.

NOTE: Alternative extractors capable of satisfactorily extracting absorbed olive oil from the test specimens may be used.

5.13 Water bath, capable of holding the flasks of soxhlet type extractors (5.12).

5.14 Rotary evaporator or distillation apparatus, for evaporation and collection of the extraction solvent.

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NOTE: Artificially cooled water may be necessary for efficient condensation of a low boiling point solvent.

5.15 Steam bath or hot plate.

5.16 Flasks, 50 ml, long neck with condensers to fit, for methyl ester preparations.

5.17 Measuring cylinders, complying with the minimum requirements of ISO 4788, 500 ml, 250 ml, 100 ml, 25 ml, and 10 ml. A 10 ml graduated syringe may be used in place of the 10 ml measuring cylinder.

5.18 Pipettes, complying with the minimum requirements of ISO 648, 5 ml, 10 ml, 50 ml and 100 ml.

5.19 Glass rods, 2mm to 3mm in diameter, for use as spacers between test pieces during solvent extraction, see 5.2 of ENV 1186-1.

5.20 Gas chromatograph, with flame ionisation detector equipped with an appropriate column capable of giving baseline resolution of the  $\text{C}_{17}$  methyl ester internal standard (methyl heptadecenoate) from  $\text{C}_{18}$  methyl

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9-octadecenoate (methyl oleate) and the C<sub>16</sub> methyl hexadecenoate (methyl palmitate) components derived from olive oil.

NOTE: The following arrangements have been found to be suitable:

- Column 1, WCOT fused silica column, length 50 m, internal diameter 0,25 mm, coated with a 0,21 micrometre thick film of cyanopropyl silicone;
- Column 2, fused silica capillary column, length 12 m, internal diameter 0,32 mm, liquid phase dimethyl siloxane;
- Column 3, stainless steel column 2 mm to 3 mm internal diameter and 2 m to 3 m length with a packing of 10 % to 20 % by weight of polyestersuccinate on a stationary phase of diatomaceous earth 80 mesh to 100 mesh.

5.21 Glass tubes with ground glass necks and stoppers, of a volume of about 10 ml, for storing the heptane layer if necessary.

5.22 Vacuum oven or vacuum desiccator.

5.23 Desiccator containing self indicating silica gel or anhydrous calcium chloride.

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### 6 Preparation of test specimens [standards.iteh.ai](https://standards.iteh.ai)

#### 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

Seven test specimens are required for samples in the form of thin films, sheet, and flat sections cut from containers or similar articles.

These test specimens are utilized as follows:

- a) Four test specimens for the migration test;
- b) Two test specimens to check for possible loss of volatiles;
- c) One test specimen to determine the suitability of olive oil as the fatty food simulant and triheptadecanoic acid as the internal standard (see annex A).

If previous testing has established that interference in the gas chromatography procedure is unlikely and annex A is omitted, one less test specimen will be required. The number of test specimens can be further reduced if it is known that the loss of volatiles from test specimens during the test is less than 5 mg per test specimen.

A minimum of three valid test results is required to calculate the mean. Testing in triplicate is allowed but in this case if one test result is invalid repeat the entire procedure.

## 6.2 Cutting test specimens

Lay the sample on the cutting slab (5.1) with the surface to be in contact with the olive oil uppermost. Take the ring from cell type A (5.6) and place on the surface of the sample. Cut out the test specimen by cutting round the outer edge of the ring, using the cutting implement (5.3).

## 7 Procedure

### 7.1 General

Determine the applicability of the method by carrying out the procedure described in annex A. If prior test have established that the method is applicable then annex A may be omitted.

Before weighing, discharge any build up of static electricity with an antistatic gun or other suitable means,

### 7.2 Initial weighing of test specimens

7.2.1 Determine the need for conditioning of the test specimens by carrying out the procedure described in annex B. If prior tests have established that sample conditioning is not required then annex B may be omitted. If prior tests have established that the conditioning procedure described in annex C is applicable to the sample then annex B may be omitted.

7.2.2 If the tests described in annex B show that conditioning is not necessary, determine and record the mass of each test specimen.

7.2.3 If the tests described in annex B show that conditioning is necessary, replace the test specimens in the container maintained at 50 % relative humidity, weigh at intervals of about 24 h, until the change in mass between consecutive weighings of each test specimen is less than 5 mg and record the eventual mass of each test specimen.

7.2.4 If the tests described in annex B show that conditioning is necessary, but constant weight cannot be achieved within 5 days then carry out the conditioning procedure described in C.2.1.

NOTE 1: Long conditioning periods are not satisfactory due to oxidation of the olive oil which may occur upon prolonged conditioning.