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**Materiali in predmeti v stiku z živilni - Plastične mase - 2. del: Preskusne metode za celotno migracijo v olivno olje s popolno potopitvijo**

Materials and articles in contact with foodstuffs - Plastics - Part 2: Test methods for overall migration into olive oil by total immersion

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 2: Prüfverfahren der Gesamtmigration in Olivenöl durch völliges Eintauchen

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 2: Méthodes d'essai pour la migration globale dans l'huile d'olive par immersion totale

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English version

**Materials and articles in contact with foodstuffs -  
Plastics - Part 2 : Test methods for overall  
migration into olive oil by total immersion**

iTeh STANDARD PREVIEW

Matériaux et objets en contact avec les denrées  
alimentaires - Matière plastique - Partie 2 :  
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REPUBLIKA SLOVENIJA  
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SIST. ENV 1186-2

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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**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 1186-3 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)

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 1186-2 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 test methods for the determination of the overall migration from plastics intended to come into contact with a fatty foodstuff, by total immersion in olive oil for 10 days, 24 h or for 2 h at 40 °C or 2 h at 70 °C.

This method is most suitable for plastics in the form of films and sheets, but can be applied to a wide range of articles or containers from which test pieces of a suitable size can be cut.

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, see 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.

- ISO 648:1977 Laboratory glassware - One mark pipettes
- ISO 4788:1980 Laboratory glassware - Graduated measuring cylinders
- ISO 8442:1988 <http://standards.iteh.ai/SIST ENV 1186-2:1997> Stainless steel and silver plated table cutlery 1)
- ENV 1186-1 Guide to the selection of conditions and test methods for overall migration
- prEN 10088 Stainless steels 2)

- 1) A European Standard for stainless steel and silver plated cutlery is in course of preparation.
- 2) In course of preparation

### 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 immersed in olive oil for 10 days, 24 h or 2 h at 40 °C or 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.

### 4 Reagents

All reagents shall be of recognized analytical quality, unless otherwise specified.

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, triheptadecanoic (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.

## 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.

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



- 5.3 Cutting implement, scalpel, scissors, sharp knife or other suitable device.
- 5.4 Metal templates  $(100 \text{ mm} \pm 0,2 \text{ mm}) \times (100 \text{ mm} \pm 0,2 \text{ mm})$  (square).
- 5.5 Rule, 25 mm  $\pm$  1 mm wide.
- 5.6 Rule, graduated in mm, and with an accuracy of 0,1 mm.
- 5.7 Analytical balance capable of determining a change in mass of 0,1 mg.
- 5.8 Specimen supports, constructed of stainless steel with cross arms attached by welding or silver soldering. Stainless steel X4 CrNi 18 10 according to prEN 10 088 or of composition, chromium 17 %, nickel 9 %, carbon 0,04 %, is suitable. Before initial use thoroughly clean the steel supports. The use of a degreasing solvent and then dilute nitric acid has been found to be suitable.

NOTE: The method has been written for the supports shown in figure B1 of ENV 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 simulant. For rigid samples, supports with a single cross arm may be used.

- 5.9 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 for insertion between the test pieces on the supports. Before initial use thoroughly clean the gauze, first with a degreasing solvent and then with dilute nitric acid.

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- 5.10 Conditioning containers, for conditioning test specimens at 50 %  $\pm$  5 % relative humidity and 80 %  $\pm$  5 % relative humidity at 20 °C  $\pm$  3 °C.

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 is suitable. The solutions should be freshly prepared by adding a weighed amount of acid to a suitable volume of water, cooling to room temperature and making up to the required volume.

- 5.11 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 of 100 mm to 200 mm, excluding the ground neck (see 5.2 of ENV 1186-1) have been found to be satisfactory.
- 5.12 Thermostatically controlled oven or incubator capable of maintaining a temperature of 40 °C  $\pm$  1 °C and 70 °C  $\pm$  2 °C.
- 5.13 Filter paper, lint-free.
- 5.14 Anti-bumping beads.
- 5.15 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.

5.16 Water bath, capable of holding the flasks of soxhlet type extractors (5.15).

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

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

5.18 Steam bath or hot plate.

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

5.20 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.

5.21 Pipettes, complying with the minimum requirements of ISO 648, 5 ml and 10 ml.

5.22 Glass beads, 2 mm to 3 mm in diameter or glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long (see 5.2 of ENV 1186-1).

5.23 Gas chromatograph, with flame ionisation detector equipped with an appropriate column capable of giving baseline resolution of the C<sub>17</sub> methyl ester internal standard (methyl heptadecenoate) from C<sub>18</sub> methyl 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 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.

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

5.25 Vacuum oven or vacuum desiccator

5.26 Desiccator containing self indicating silica gel or anhydrous calcium chloride

## 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.

To ensure that test pieces are well separated and that their surfaces are freely exposed to olive oil during the period of the test, for thin films insert a piece of fine stainless steel gauze (5.9) between the test pieces or for thick samples not placed on the supports, insert glass rods between the test pieces after immersion in the olive oil. Where specimen supports are used, label the supports with a tag bearing the test specimen identification.

### 6.2 Number of test specimens

Seven test specimens are required for samples, in the form of thin films, sheet, containers or similar articles. Nine test specimens, similar dimensionally one to another, are required for samples of articles of irregular shape.

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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 triheptadecanoin as the internal standard (see annex A);
- d) Two test specimens for determination of the surface area, in the case of samples of irregular shape (see 6.5).

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 2 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.

### 6.3 Thin films and sheet materials