
Materiali in predmeti v stiku z živilni - Plastične mase - 10. del: Preskusne metode za celotno migracijo v olivno olje (modificirana metoda za uporabo v primeru nepopolne ekstrakcije olivnega olja)

Materials and articles in contact with foodstuffs - Plastics - Part 10: Test methods for overall migration into olive oil (modified method for use in case where incomplete extraction of olive oil occurs)

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 10: Prüfverfahren der Gesamtmigration in Olivenöl (Modifiziertes Verfahren für die Anwendung bei unvollständiger Extraktion von Olivenöl)

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Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 10: Méthodes d'essai pour la migration globale dans l'huile d'olive (méthode modifiée à utiliser en cas d'extraction incomplète de l'huile d'olive)

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English version

**Materials and articles in contact with foodstuffs -
Plastics - Part 10 : Test methods for overall into
olive oil (modified method for use in case where
incomplete extraction of olive oil occurs)**

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 10 : Méthodes d'essai pour la migration globale dans l'huile d'olive (méthode modifiée à utiliser en cas d'extraction incomplète de l'huile d'olive)

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 10 : Prüfverfahren der Gesamtmigration in Olivenöl (Modifiziertes Verfahren für die Anwendung bei unvollständiger Extraktion von Olivenöl)

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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
- 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

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 1186-10 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 a fatty food simulant from polystyrene based plastics intended to come into contact with foodstuffs, by total immersion in olive oil for 10 days, 24 h or 2 h at 40 °C or for 2 h at 70 °C.

When some plastics are tested by the methods in Parts 2, 4, 6, 8, the soxhlet extraction process does not achieve complete recovery of the absorbed olive oil from the test specimens (see ENV 1186-1). In this method, the olive oil is released from the plastics test specimens by dissolving them in chloroform.

This method is suitable for plastics when exposure to olive oil is by total immersion as described in ENV 1186-2, provided they are soluble in chloroform and insoluble in methanol.

The method is also suitable for plastics when exposure to olive oil is in a Tice cell, as described in ENV 1186-4. This is provided the plastics are soluble in chloroform and insoluble in methanol and that whenever ENV 1186-2 is referred to in this method the appropriate clause of ENV 1186-4 is substituted.

Also, this method is suitable for plastics when exposure to olive oil is in a standard pouch, as described in ENV 1186-6. This is provided the plastics are soluble in chloroform and insoluble in methanol and that whenever ENV 1186-2 is referred to in this method the appropriate clause of ENV 1186-6 is substituted.

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Similarly, the method is suitable for plastics when exposure to olive oil is by filling, as described in ENV 1186-8. This is provided the plastics are soluble in chloroform and insoluble in methanol and that whenever ENV 1186-2 is referred to in this method the appropriate clause of ENV 1186-8 is substituted.

The method may also be suitable for plastics which are only partially soluble in chloroform and insoluble in methanol (see ENV 1186-1). The olive oil is not fully extracted but this is compensated for by preparing the methyl esters of both the olive oil and the internal standard in situ.

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 ¹⁾
- 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 1186-4 Test methods for overall migration into olive oil by cell
- ENV 1186-6 Test methods for overall migration into olive oil using a pouch
- ENV 1186-8 Test methods for overall migration into olive oil by article filling
- prEN 10088 Stainless steels ²⁾

3 Principle

The overall migration from a sample of the plastic, such as polystyrene, is determined as the loss in mass of specimens after immersion in olive oil.

Test specimens of known mass are immersed in 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 selection of the test conditions will be determined by the conditions of use, see clause 3 of ENV 1186-1.

The specimens will usually retain absorbed olive oil which is extracted by a dissolution and precipitation procedure and determined quantitatively by 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.

Overall 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 test specimen.

The total loss in mass is expressed in milligrams per square decimetre of surface area of the test specimen intended to come into contact with foodstuffs 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 sampling handling stages, quadruplicate determinations are carried out on the sample allowing for the result from one specimen to be discarded.

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

2) In course of preparation

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during the sampling handling stages, quadruplicate determinations are carried out on the sample allowing for the result from one specimen to be discarded.

4 Reagents

The reagents shall be as in ENV 1186-2,

plus:

4.2 Extraction solvent not required

4.8 Chloroform;

4.9 Methanol;

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 Metal templates 100 mm \pm 0,2 mm x 100 mm \pm 0,2 mm (square).

5.5 Rule, 25 mm \pm 1 mm wide. [SIST ENV 1186-10:1997](https://standards.iteh.ai/catalog/standards/sist/47da6864-e84c-449e-9cdb-1496970c1011/sist-env-1186-10-1997)
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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 10088 or of 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 with dilute nitric acid has been found to be suitable.

NOTE: The method has been written for the supports shown in figure B.1 of ENV 1186-1, 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.

5.10 Conditioning containers, for conditioning test specimens at 50 % \pm 5 % relative humidity and 80 % \pm 5 % relative humidity.

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.

5.11 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 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, not required.

5.16 Water bath capable of holding 250 ml flasks (5.26), and condensers.

5.17 Rotary evaporator or distillation apparatus, for evaporation and collection of the extraction 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 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 C₁₇ methyl ester internal standard (methyl heptadecenoate) from C₁₈ methyl 9-octadecenoate (methyl oleate) and the C₁₆ methyl hexadecenoate (methyl palmitate) components derived from olive oil.