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Materiali in predmeti v stiku z živili - Polimerni materiali - 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 für die Gesamtmigration in Olivenöl durch völliges Eintauchen

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

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This European Standard was approved by CEN on 4 January 2002.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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Foreword

This document EN 1186-2:2002 has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2002, and conflicting national standards shall be withdrawn at the latest by October 2002.

This document supersedes ENV 1186-2:1994.

This European Standard is one of a series of methods of test for plastics materials and articles in contact with foodstuffs.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EC Directive(s).

For relationship with EC Directive(s), see informative annex ZA, which is an integral part of this document.

At the time of preparation and publication of this standard the European Union legislation relating to plastics materials and articles intended to come into contact with foodstuffs is incomplete. Further Directives and amendments to existing Directives are expected which could change the legislative requirements which this standard supports. It is therefore strongly recommended that users of this standard refer to the latest relevant published Directive(s) before commencement of any of the test or tests described in this standard.

EN 1186-2 should be read in conjunction with EN 1186-1.
Further Parts of this standard have been prepared concerned with the determination of overall migration from plastics materials into food simulants.

Their titles are as follows:

EN 1186 Materials and articles in contact with foodstuffs - Plastics –

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

Part 13	Test methods for overall migration at high temperatures
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
Part 15	Alternative test methods to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol

Annexes A, B, C and D to this standard are normative where applicable. The annexes E and F are informative.

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

1 Scope

This Part of this European Standard describes test methods for the determination of the overall migration into fatty food simulants from plastics materials and articles, by total immersion of test specimens in a fatty food simulant at temperatures above 20 °C and up to, but not including, 100 °C for selected times.

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.

NOTE This test method has been written for use with the fatty food simulant, olive oil. The test method can also be used with appropriate modifications with 'other fatty food simulants ' called simulant D - a synthetic mixture of triglycerides, sunflower oil and corn oil. These other fatty food simulants will produce different chromatograms for the simulant methyl esters to those of the methyl esters of olive oil. Select suitable chromatogram peaks of the methyl esters of the other fatty food simulants for the quantitative determination of the simulant extracted from the test specimens.

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.

2 Normative references

This European Standard 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 Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1186-1:2002, *Materials and articles in contact with foodstuffs - Plastics – Part 1:Guide to the selection of conditions and test methods for overall migration.*

EN 10088, *Stainless steels.*

EN ISO 8442-2:1997, *Materials and articles in contact with foodstuffs – Cutlery and table holloware – Part 2: Requirements for stainless steel and silver-plated cutlery (ISO 8442-2:1997)*.

ISO 648, *Laboratory glassware - One mark pipettes.*

ISO 4788, *Laboratory glassware - Graduated measuring cylinders.*

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 clauses 4, 5 and 6 of EN 1186-1:2002.

Test specimens of known mass are immersed in olive oil for the exposure time, at temperatures above 20 °C and below 100 °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 that 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, 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, four determinations are carried out on the sample allowing for the result from one specimen to be discarded.

This method includes variations that are applicable to certain plastics.

NOTE Before starting a migration exercise, the test sample should be examined for the presence of components interfering in the determination of the amount of olive oil extracted, see 7.1. If an unacceptable amount of interference is present then suitability of one of the 'other fatty food simulants' should be examined, see annex A and 9.3 and 9.5 of EN 1186-1:2002. If an interference is present which would interfere with the triheptadecanoin internal standard an alternative internal standard should be used, see annex A and 9.3 of EN 1186-1:2002.

4 Reagents

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

4.1 Olive oil, reference simulant D, as specified in 5.2 of EN 1186-1:2002.

4.2 Extraction solvent (see 10.1 of EN 1186-1:2002).

4.2.1 For non-polar plastics, such as polyethylene and polypropylene:

- Pentane 98 % boiling point 36 °C.

For polar plastics, such as polyamide and polyacetal:

- 95/5 by volume azeotropic mixture of pentane 98 % and ethanol 99 %.

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. Ethanol is also a flammable solvent. It is not recommended that extractions with either pentane or the pentane/ethanol mixture be left unattended, particularly overnight.

NOTE 2 Due to the low boiling points of these solvents, cooled condenser water can be required to prevent undue loss of the solvent from the condenser.

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 9.1 of EN 1186-1. Experience has shown that this solvent, although effective for most plastics requires longer periods of extraction.

NOTE 2 Some solvents can 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[1)] of a quality such that the products from hydrolysis and methylation processes do not contain substances giving detectable gas chromatography peaks (see 9.3 of EN 1186-1) with similar retention times to the olive oil methyl ester peaks. Prepared as a solution containing 2,0 mg/ml in cyclohexane.

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

4.5 Boron trifluoride, methanol complex, approximately 150 g/l BF_3.

4.6 n -Heptane.

4.7 Sodium sulfate.

4.7.1 Sodium sulfate, anhydrous, Na_2SO_4.

4.7.2 Sodium sulfate, saturated solution.

4.8 Diethyl ether.

4.9 Karl Fischer solvent, commercially prepared, methanol and chloroform based, water capacity of 5 mg/ml.

4.10 Karl Fischer titrant (for volumetric apparatus only), commercially prepared, water capacity of 2 mg/ml.

5 Apparatus

5.1 Cutting slab, clean smooth glass, metal or plastics slab of sufficient area to prepare test specimens, 250 mm × 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 ± 0,2 mm) × (100 mm ± 0,2 mm) (square).

5.5 Rule or template, 25 mm ± 1 mm wide.

5.6 Rule, graduated in millimetres, 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 EN 10088 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 C.1 of EN 1186-1:2002 which have been found to be suitable for holding thin film and sheet test pieces. However other supports can 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 can 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 × 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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1) The source of this is the Chemical Abstracts published by the American Chemical Society.

5.10 Conditioning containers, for conditioning test specimens at 50 % ± 5 % relative humidity and 80 % ± 5 % relative humidity at 20 °C ± 5 °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. It is recommended that relative humidity and temperature be maintained during the conditioning period. Therefore the containers should be placed in a thermostatically controlled room or oven, at a temperature of approximately 20 °C, the set temperature should not vary by more than ± 1 °C.

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 7.2 of EN 1186-1:2002, have been found to be satisfactory.

5.12 Thermostatically controlled oven or incubator capable of maintaining the set temperature, within the tolerances specified in Table B.2 of EN 1186-1:2002.

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 can 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 can be necessary for efficient condensation of a low boiling point solvent.

5.18 Steam bath or water bath.

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 7.2 of EN 1186-1:2002).

5.23 Gas chromatograph, with flame ionisation detector equipped with an appropriate column. When using a polar column, the major peaks of olive oil, such as C16:0, methyl hexadecanoate (methyl palmitate), C16:1, methyl 9-hexadecenoate (methyl palmitoate), C18:0, methyl octadecanoate (methyl stearate), C18:1, methyl 9-octadecenoate (methyl oleate), C18:2, methyl 9,12-octadecadienoate (methyl linoleate) and the internal standard C17:0, methyl heptadecanoate (methyl margarate) shall demonstrate baseline separation. Optionally, a non-polar column can be used which shall give baseline separation of the methyl esters with 16 and 18 carbon numbers and the internal standard with 17 carbon number.

NOTE The following columns have been found to be suitable:

- Column 1, polar column, WCOT fused silica column, length 50 m, internal diameter 0,25 mm, coated with a 0,21 micrometre film of cyanopropyl silicone;

- Column 2, non polar column, BP1, length 25 m, internal diameter 0,32 mm, with a 1 micron film thickness;

- Column 3, polar column, 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 mass 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, capable of maintaining a temperature of 60 °C ± 2 °C. The vacuum oven or vacuum desiccator shall be equipped with or connected to a vacuum pump capable of achieving a vacuum of 1,3 kPa or less. The vacuum pump shall be provided with a time controller to switch on the vacuum pump every hour for 15 min.

NOTE If a vacuum oven is not available, a vacuum desiccator placed in an oven at 60 °C can be used.

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

5.27 Balance, capable of determining a change of mass of 10 mg.

5.28 Disposable plastic syringes with luer fitting. 1 ml or 10 ml size.

5.29 Wide gauge luer needles (80 mm × 1.2 mm).

5.30 Karl Fischer apparatus, either an automated volumetric titrator, or an automated coulometric titrator. The Karl Fischer titrator shall be capable of measuring the water content of the simulant with a precision (standard deviation) of 10 mg/kg or less (equivalent to 1 mg/dm^2 plastic). An automated volumetric or coulometric instrument shall be used. Manual titration procedures do not give the required accuracy or precision.

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 8.1 of EN 1186-1:2002. 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.

When preparing test specimens measure the surface area according to 8.3 of EN 1186-1:2002.

6.2 Number of test specimens

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

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 the conditioning test in annex C is used, one additional test specimen is required.

NOTE The two test specimens, b), are used to check whether the sample losses mass from the evaporation of volatiles, such as solvents, during the test period. If the vacuum drying procedure in annex C is used these test specimens are not required as during the vacuum drying any volatiles will have been removed from the test specimens.

If previous testing has established that interference in the gas chromatography procedure is unlikely and annex A is omitted, one fewer test specimen will be required.

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 Films and sheets

Lay the sample on the cutting slab (5.1) and cut test specimens of 1 dm², see 8.3 of EN 1186-1:2002, using the 100 mm × 100 mm template (5.4). Check, using the rule (5.6), that the dimensions of the test specimen are within the specified deviation (± 1 mm).

Cut each test specimen into four test pieces 25 mm × 100 mm using the rule (5.5). Assemble one test specimen onto the support by piercing suitable holes in the test pieces and placing two test pieces on each side of the cross arms of the support. Repeat this procedure for all remaining test specimens.

6.4 Containers and other articles

Cut sections from the walls of the container or article to give test specimens each of area approximately 1 dm². For articles with individual areas less than 1 dm², use a number of articles to provide each test specimen.

Measure the dimensions of each test specimen to the nearest 1 mm, using the rule, see 8.3 of EN 1186-1:2002.

Calculate the area of each test specimen to the nearest 0,01 dm² and record. If necessary, cut each test specimen into smaller pieces to enable them to fit into the glass tubes (5.11). The test specimens or pieces are placed on the specimen supports if these are appropriate or, if the test specimens or pieces are sufficiently rigid, they can be tested unsupported.

NOTE Cutting the test specimens into smaller pieces will increase the area of cut edges, so that the area of cut edges exceeds 10 % of the of the test specimen area. In this case see 8.3 of EN 1186-1:2002.

6.5 Articles of irregular shape

Select representative portions of the article, or multiples of the article for small articles, to give nine dimensionally similar test specimens each with a known total surface area of at least 1 dm². Measure only the surface area intended to come into contact with foodstuffs of two of these test specimens to the nearest 0,05 dm² using the Schlegel Method, as described in EN ISO 8442-2:1997, annex B, or any other suitable method. Record the surface area of each test specimen.

7 Procedure

7.1 General

Determine the applicability of the method by carrying out the procedure described in annex A. If prior tests 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.