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Materiali in predmeti v stiku z živili - Polimerni materiali - 8. del: Preskusne metode za celotno migracijo v olivno olje, s katerim je napolnjen predmet

Materials and articles in contact with foodstuffs - Plastics - Part 8: Test methods for overall migration into olive oil by article filling

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 8: Prüfverfahren für die Gesamtmigration in Olivenöl durch Füllen des Gegenstandes

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Matériaux et objets en contact avec les denrées alimentaires - Matiere plastique - Partie 8: Méthodes d'essai pour la migration globale dans l'huile d'olive par remplissage

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67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs

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EUROPEAN STANDARD

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English version

Materials and articles in contact with foodstuffs - Plastics - Part 8: Test methods for overall migration into olive oil by article filling

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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 1186-8:2002 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of an identical text or by endorsement, at the latest by October 2002, and conflicting national standards shall be withdrawn at the latest by October 2002.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 1186-8:1994.

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.

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EN 1186-8 should be read in conjunction with EN 1186-1.

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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 2	Test methods for overall migration into olive oil by total immersion
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 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

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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

The annexes A to D are normative. 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.

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1 Scope

This Part of this European Standard specifies test methods for the determination of the overall migration into fatty food simulants from plastics materials and articles, by filling of test specimens with 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 containers and articles that can be filled.

Testing samples by this method enables testing of non-homogenous articles provided they are not too large.

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

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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).

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

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 filled with olive oil for the exposure time, at temperatures above 20 °C and below 100 °C, then emptied and 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.

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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 removing 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 which 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, simulant D, as specified in 4.2 of EN 1186-1:2002.

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4.2 Extraction solvent (see 9.1 of prEN 1186-1:2001).

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

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- 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.

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 9.1 of EN 1186-1:2002. 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

1) The source of this is the Chemical Abstracts published by the American Chemical Society.

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chromatography peaks (see 9.3 of EN 1186-1:2002) 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

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5.1 Tweezers, stainless steel, blunt nosed.

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

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

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

5.5 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.

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

5.7 Filter paper, lint-free.

5.8 Chromatography tank or any other airtight container for test sample storage.

5.9 Glass rods or metal gauze for use as spacers between test pieces during solvent extraction.

5.10 Antibumping beads.

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

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

5.12 Water bath, capable of holding the flasks of soxhlet type extractors (5.11)

5.13 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.

5.14 Steam bath or water bath.

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

5.16 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.

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

5.18 Lint-free cloth

5.19 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.

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NOTE The following columns have been found to be suitable:

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- 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.

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

5.21 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.

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

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

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

5.25 Wide gauge luer needles (80 mm × 1,2 mm).

5.26 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.

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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. Minimize handling of the samples and, where necessary, wear cotton gloves.

6.2 Number of test specimens

Nine test specimens are required for samples, in the form in which they are intended to be used.

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.

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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.

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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 Cutting test specimens

If the article is large, to avoid handling and weighing problems or using excessive amounts of olive oil it may be preferable to cut it so that the surface of the test specimen in contact with the olive oil does not exceed 3 dm².

If this is done, take care that olive oil does not come into contact with the cut edges of the test specimen. It is important that the area in contact with the oil is determined as it will be incorporated into the calculation later.

Scratch lightly an identification code on the external surface of each test specimen.

NOTE If only part of a specimen is tested, this part should be representative of the whole in terms of composition and wall or layer thickness.

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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.

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 or in annex C. If prior tests have established that sample conditioning is not required then annex B and annex C may be omitted. If prior tests have established that the procedure described in annex D is applicable to the sample, then annex B or annex C may be omitted.

7.2.2 If the tests described in annex B or annex C 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 or annex C show that conditioning is necessary, follow the directions in the relevant annex to determine the initial mass of the sample.

7.2.4 If the tests described in annex B show that conditioning is necessary, but constant mass cannot be achieved within five days then carry out the conditioning procedure described in C.3.1 or annex D.

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

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NOTE 2 The conditioning procedures described in annex C and annex D can be used if it has been established that these procedures are more suited to the polymer type under test.

7.3 Exposure to food simulant

Place a sufficient volume of olive oil in a beaker in the thermostatically controlled oven or incubator (5.6) which is set at the test temperature and leave until the test temperature has been attained.

Place each test specimen on a clean, oil free surface and fill four specimens with olive oil to within 0.5 cm of the top. If the container has a specified nominal volume of contents, see 8.2 of EN 1186-1:2002. Place into one of the filled test specimens a thermometer or thermocouple.

NOTE 1 If the procedure described in annex D is used, it can be necessary to dry all of the olive oil used for the migration test, see D.3.2.

NOTE 2 Care should be taken not to spill any oil on the external surfaces.

NOTE 3 The two remaining test specimens are used to check whether the sample losses mass from the evaporation of volatiles, such as water, solvents and oligomers, during the test period. If the vacuum drying procedure in annex C is applicable these test specimens are not required as during the vacuum drying volatiles will have been removed from the test specimens.

Place sufficient olive oil into a tube for use as reference standards in constructing the calibration graph (see 7.6.2.2) and if the procedure in annex D is used, as a third blank sample for Karl Fischer titrations, stopper the tube.

Place the four filled test specimens and the two empty test specimens and the reference oil in the tube in the thermostatically controlled oven or incubator set at the test temperature. This part of the operation should be carried out in the minimum time possible to prevent undue heat loss.