SLOVENSKI STANDARD
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Materiali in predmeti v stiku z živili - Polimerni materiali - 10. del: Preskusne metode za celotno migracijo v olivno olje (modificirana metoda za uporabo pri nepopolni ekstrakciji 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 cases where incomplete extraction of olive oil occurs)

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

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

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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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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Foreword

This document EN 1186-10:2002 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of an identical text or by endorsement, at the latest by March 2003, and conflicting national standards shall be withdrawn at the latest by March 2003.

This document supersedes ENV 1186-10:1994.

This European Standard has been prepared as one of a series of methods of test for plastics materials and articles in contact with foodstuffs.

This Part of this European Standard has been prepared by a Subcommittee (SC1) of TC 194 'Utensils in contact with food' as 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.

For relationship with EU 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 1186-10 should be read in conjunction with EN 1186-1, EN 1186-2, EN 1186-4, EN 1186-6, EN 1186-8, prEN 1186-12 and EN 1186-13.

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 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 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 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

Annex A of this standard is normative where applicable. Annex B is 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 European Standard specifies 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 any temperatures above 5 °C up to and including 175 °C for selected times.

When some plastics are tested by the methods in EN 1186-2, EN 1186-4, EN 1186-6, EN 1186-8, prEN 1186-12 and EN 1186-13, the soxhlet extraction process does not achieve complete recovery of the absorbed olive oil from the test specimens. In this method, the olive oil is released from the plastics test specimens by dissolving them in chloroform, toluene, xylene or tetrahydrofuran.

This method is suitable for plastics when exposure to olive oil is by total immersion as described in EN 1186-2, in a cell, as described in EN 1186-4, in a pouch, as described in EN 1186-6, and by filling, as described in EN 1186-8 and to tests carried out at low and high temperature, as described in prEN 1186-12 and EN 1186-13.

This is provided the plastics are soluble in chloroform, toluene, xylene or tetrahydrofuran and insoluble in methanol and that whenever prEN 1186-2 is referred to in this method the appropriate clause of the relevant part of EN 1186 is substituted.

The method can also be suitable for plastics which are only partially soluble in chloroform, toluene, xylene or tetrahydrofuran and insoluble in methanol.

NOTE 1 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.

NOTE 2 If it has been established that the overall migration into olive oil from the plastics cannot be determined by use of either this method or the methods described in EN 1186-2, EN 1186-4, EN 1186-6 and EN 1186-8 then the use of substitute tests should be considered, see clause 6 of EN 1186-1:2001.

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 (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 1186-2:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 2: Test methods for overall migration into olive oil by total immersion.*

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.

The selection of the test conditions 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 exposed to olive oil for the exposure time, at temperatures varying from 5 °C to 175 °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 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.

In case the plastic does not dissolve completely, swelling of the plastic in the solvent should be such that the olive oil absorbed can be released from the plastic.

Depending on the type of plastic an appropriate organic solvent is selected in order to dissolve or swell the plastic.

For chloroform soluble plastics like polystyrene and polycarbonate, chloroform is used to release the olive oil absorbed.

For polyolefins, toluene and xylene are used as low density polyethylene shows good solubility in toluene and high density polyethylene and polypropylene dissolve or swell sufficiently in xylene.

For polyvinylchloride or polyvinylidene chloride, tetrahydrofuran can be applied.

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.

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 of EN 1186-2:2002. 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

The reagents shall be as described in clause 4 of EN 1186-2:2002, except that the extraction solvent (4.2) is not required and the following reagents are added to the list:

a) chloroform;

b) methanol;

c) tetrahydrofuran;

d) toluene;

e) xylene.

5 Apparatus

The apparatus shall be as described in clause 5 of EN 1186-2:2002, with the exception of the soxhlet extractors (5.15) which are not required and the addition of the following:

a) centrifuge;

b) centrifuge tubes 150 ml;

c) conical funnels, 100 mm diameter;

d) filter papers, 185 mm diameter.

6 Preparation of test specimens

Prepare the test specimens in accordance with clause 6 of EN 1186-2:2002.

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

Perform the initial weighing in accordance with 7.2 of EN 1186-2:2002.

7.3 Exposure to food simulant

Expose the test specimens in accordance with 7.3 of EN 1186-2:2002.

7.4 Final weighing of test specimens

Perform the final weighing in accordance with 7.4 of EN 1186-2:2002.

7.5 Extraction of absorbed olive oil

Take four flasks, 250 ml, to be used for the extraction, and place in each flask 10,0 ml of the internal standard cyclohexane solution of triheptadecanoin (4.3 of EN 1186-2:2002), using a pipette (5.21 of EN 1186-2:2002), or an alternative higher quantity if more than 100 mg of olive oil is present.

NOTE 1 If the test specimens have retained more than 100 mg of olive oil, 10,0 ml of the internal standard solution is not sufficient for optimum precision in the gas chromatography determination after extraction. Before commencing the operations in this clause an estimation of the quantity of olive oil retained in the test specimens should be obtained by comparing the final masses of the test specimens with their initial masses. If considered necessary the quantity of internal standard solution can be increased from 10 ml although it is essential that the same quantity is used for each test specimen, and that this quantity is also

used with the olive oil standards for the calibration graph. As a guide, approximately 0,5 mg of the internal standard is required for every milligramme of extracted olive oil.

Place the four test specimens in the flasks and add to each flask, by measuring cylinder, 50 ml to 60 ml of chloroform (4.a), tetrahydrofuran (4.c), toluene (4.d) or xylene (4.e) and a few anti-bump beads to control boiling. Carefully remove the specimens from the supports using tweezers. If necessary, carefully cut the test specimens in pieces of approximately 2 cm by 2 cm. Wash the tweezers and supports with 50 ml to 60 ml of the relevant solvent and transfer the washings to the flask. Couple each flask to a condenser, place on either a water bath or a steam bath and reflux for 30 min. Add slowly down the condenser, by measuring cylinder or syringe, 10 ml ± 0,2 ml of the potassium hydroxide solution (4.4 of EN 1186-2:2002) and continue refluxing for 15 min to 20 min. Add by measuring cylinder at least 50 ml ± 2 ml of methanol slowly down the condenser and continue refluxing for 5 min to 6 min.

NOTE 2 In such cases that the mass of the test sample is relatively high then the amount of the dissolving and precipitating solvent might need to be adapted to the mass of the test sample. In all cases a solution with reasonable viscosity should be obtained.

Remove the flasks from the water bath and allow to cool. Transfer the solution from each flask to individual 150 ml centrifuge tubes (5.25 of EN 1186-2:2002), washing out each flask with 5 ml to 10 ml of methanol into the tube. Centrifuge each solution until a clear supernatant liquid is obtained. Filter the supernatant solutions through a filter paper into 250 ml round bottom flasks. Evaporate the solutions to 15 ml to 20 ml, either using a rotary evaporator or a simple distillation apparatus. Transfer the solutions to individual 50 ml round bottom flasks, washing out with 5 ml to 7 ml of methanol, add a few anti-bumping beads. Evaporate each solution to dryness on a water bath.

NOTE 3 Oxidation of the olive oil should be avoided where possible. Therefore evaporation of the solvent to dryness should be carried out under mild conditions of temperature. In addition exposure of the olive oil to oxygen should be limited.

7.6 Determination of extracted olive oil

7.6.1 Preparation of fatty acid methyl esters

Add 10 ml ± 0,2 ml of n-heptane to each of the 50 ml flasks containing the dry residue as obtained in 7.5 by measuring cylinder (5.20 of EN 1186-2:2001), ensuring that the residues of olive oil and plastics extractables dissolve or are well dispersed by shaking, warming or by ultrasonic treatment.

NOTE 1 Unless the residues in the flasks are dissolved or well dispersed in the n-heptane, quantitative hydrolysis or methylation of the olive oil and of the internal standard might not be obtained under the conditions described particularly when these residues contain extractables from plastics in excess of 50 mg. The internal standard might not react with the plastics extractables to the same degree as does the olive oil and correct results for olive oil might not be obtained.

Add by measuring cylinder or graduated syringe, 10 ml ± 0,2 ml of methanol (4.b) and a few anti-bumping beads (5.14 of prEN 1186-2:2001). Connect a condenser to the flask and boil the mixture under reflux for 10 min ± 1,0 min.

Add through the condenser by measuring cylinder, or graduated syringe, 5,0 ml ± 0,2 ml of the methanol solution of boron trifluoride (4.5 of EN 1186-2:2002) and boil the mixture under reflux for 2 min ± 0,25 min.

Cool to room temperature and add, by measuring cylinder, 15 ml to 20 ml of saturated sodium sulfate solution (4.7.2 of EN 1186-2:2002) and shake well. Then add further sodium sulfate solution until the liquid level reaches the neck of the flask. Allow to stand until the phases have separated.

NOTE 2 The methyl esters for the subsequent gas chromatographic determination are in the upper, n-heptane, layer.

If there is a delay of more than 7 days in using a methyl ester solution for the gas chromatographic determinations, transfer the n-heptane layer to a small stoppered tube (5.24 of EN 1186-2:2002) containing solid anhydrous sodium sulfate (4.7.1 of EN 1186-2:2002) and store in a refrigerator.