SLOVENSKI STANDARD
SIST ENV 1186-8:1997

01-januar-1997

Materiali in predmeti v stiku z živili - Plastične mase - 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 der 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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EUROPEAN PRESTANDARD

PRÉNORME EUROPÉENNE

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Descriptors: plastic products, materials, plastics, food products, food-container contact, determination, migrations, tests, olive oil, filling

English version

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

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

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REPUBLIKA SLOVENIJA
MINISTRSTVO ZA ZNANOST IN TEHNOLOGIJO
Urad RS za standardizacijo in meroslovje
LJUBLJANA
SIST ENV 1186-8
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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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

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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

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ENV 1186-6 Test methods for overall migration into olive oil using a pouch

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ENV 1186-7 Test methods for overall migration into aqueous food simulants using a pouch

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 ^{14}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 1186-8 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 from one surface only of the plastics in the form of finished articles, which are intended to come into contact with a fatty foodstuff, by filling the finished article with olive oil and leaving for 10 days, 24 h or 2 h at 40 °C or for 2 h at 70 °C.

Testing samples by this method enables testing of non-homogeneous articles providing that they are not too large.

This method is most suitable for plastics in the form of containers and articles that can be filled.

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.

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ISO 648:1977 Laboratory glassware - One mark pipettes

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ISO 4788:1980 Laboratory glassware - Graduated measuring cylinders

ENV 1186-1 Guide to the selection of conditions and test methods for overall migration

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 filled with olive oil and stored for 10 days, 24 h or 2 h at 40 °C or 2 h at 70 °C, then emptied, blotted to remove olive 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

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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 removing 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.

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4 Reagents

All reagents shall be recognised analytical quality, unless otherwise specified.

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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

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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, triheptadecanoin (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 BF_3.

4.6 n-Heptane.

4.7 Sodium sulphate.

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4.7.1 Sodium sulphate, anhydrous, Na_2SO_4.

4.7.2 Sodium sulphate, saturated solution.

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5 Apparatus

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.

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.

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.

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

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5.6 Thermostatically controlled oven or incubator capable of maintaining a temperature of 40 °C ± 1°C and 70 °C ± 2 °C.

5.7 Filter paper, lint-free.

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

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

5.10 Anti-bumping 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.

NOTE: Alternative extractors capable of satisfactorily extracting absorbed olive oil from the test specimens may 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 may be necessary for efficient condensation of a low boiling point solvent.

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5.14 Steam bath or hot plate.

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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, 10 ml, 50 ml, and 100 ml.

5.18 Lint-free cloth or brush.

5.19 Gas chromatograph, with flame ionisation detector equipped with an appropriate column capable of giving baseline resolution of the C_{17} methyl ester internal standard (methyl heptadecenoate) from C_{18} methyl 9-octadecenoate (methyl oleate) and the C_{16} methyl hexadecenoate (methyl palmitate) components derived from olive oil.

NOTE: The following arrangements have been found to be suitable:

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- Column 1, WCOT fused silica column, length 50 m, internal diameter 0,25 mm, coated with a 0,21 micrometre thick 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.

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

5.21 Vacuum oven or vacuum desiccator

5.22 Desiccator containing self indicating silica gel or anhydrous calcium chloride

6 Preparation of test articles

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.

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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 utilised 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 the determination of the surface area.

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

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reduced if it is known that the loss of volatiles from test specimens during the test is less than 2 mg per square decimetre of surface of test specimen in contact with olive oil.

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 dm^2.

If this is done, care must be taken that olive oil does not come into contact with the cut edge of the test specimen. It is important that the area in contact with the olive 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 a part of an 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

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

7.2.2 If the tests described in annex B 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 show that conditioning is necessary, replace the test specimen in the container maintained at 50% relative humidity, weigh at intervals of about 24 h, until the change in mass between