

norm**NEN-EN 1186-13**

Materialen en artikelen in contact met voedingsmiddelen - Kunststoffen - Deel 13: Beproevingmethoden voor totale migratie bij hoge temperaturen

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English version

**Materials and articles in contact with foodstuffs - Plastics - Part
13: Test methods for overall migration at high temperatures**

Matériaux et objets en contact avec les denrées
alimentaires - Matière plastique - Partie 13: Méthodes
d'essai pour la migration globale à hautes températures

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln
- Kunststoffe - Teil 13: Prüfverfahren für die
Gesamtmigration bei hohen Temperaturen

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 194 'Utensils in contact with food', the secretariat of which is held by BSI.

This document is currently submitted to the Unique Acceptance Procedure.

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 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 EU Directive(s).

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

This European Standard supersedes EN 1186-13:1999.

Read prEN 1186-13 Method A and Method B read in conjunction with prEN 1186-1. Also, read prEN 1186-13 Method A in conjunction with prEN 1186-2.

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.

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 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 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 and B are informative.

Introduction

Migration testing with olive oil at high temperatures introduces a number of analytical difficulties. Experience has shown that it is difficult to achieve reproducible results owing to different laboratories having different equipment which give rise to variations in the time taken to reach the exposure temperature. A method is described for determining overall migration by total immersion using an aluminium block with a consistent thermal capacity. Other analytical difficulties with olive oil include possible oxidation of oil at elevated temperatures and the hazard to personnel working with hot oil. Replacement of olive oil by an appropriate adsorbent material, in principal, can solve or reduce these problems and offers further experimental advantages.

1 Scope

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 from 100 °C up to and including, 175 °C for selected times. Also described is a procedure with a substitute test medium. In this substitute procedure the mass of components adsorbed on modified polyphenylene oxide (MPPO) is taken as a measure for the assessment of the overall migration into olive oil.

NOTE 1 The total immersion 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 A comparative migration test carried out with polypropylene and polyethylene terephthalate high temperature application containers as test samples at conditions 2 h at 100 °C and 2 h at 175 °C, respectively, in contact with ¹⁴C-labelled synthetic triglyceride and MPPO provide test results comparable within the analytical tolerance of the methods.

NOTE 3 To obtain reproducible and repeatable results using the MPPO method it may be necessary to measure the temperature of the test specimen before starting the migration period. An appropriate method for measuring the temperature of the test specimen needs to be established.

The described methods are most suitable for food contact articles in the form of sheets and films, but can also be applied to a wide range of articles and containers.

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).

prEN 1186-1:2001, *Guide to the selection of conditions and test methods for overall migration.*

prEN 1186-2:2001, *Test methods for overall migration into olive oil by total immersion.*

3 Method A - total immersion in olive oil

3.1 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 prEN 1186-1:2001.

Test specimens of known mass are immersed in olive oil for the exposure time, at temperatures from 100 °C up 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 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 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 9.3 of prEN 1186-1:2001. 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 prEN 1186-1:2001. 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 prEN 1186-1:2001.

3.2 Reagents

The reagents shall be as described in clause 4 of prEN 1186-2:2001.

3.3 Apparatus

The apparatus shall be as described in clause 5 of prEN 1186-2:2001, with the addition of:

Aluminium block or blocks with wells for holding up to ten glass tubes, see 5.11 of prEN 1186-2:2001, during the exposure time period in the oven or incubator.

NOTE A diagram of a suitable block is shown in figure A.1. The wells in the block should hold the tubes so that there is close contact between the tubes and the block. The block should be of sufficient depth that when the specimen is placed in the oil in the tubes, the level of the oil is lower than, or equal to the height of the block.

3.4 Preparation of test specimens

Prepare the test specimens in accordance with prEN 1186-2, except that an additional test specimen is required. This test specimen shall be placed in the tube in which the temperature is monitored.

3.5 Procedure

3.5.1 General

See 7.1 of prEN 1186-2:2001.

3.5.2 Initial weighing of test specimens

Perform the initial weighing in accordance with 7.2 of prEN 1186-2:2001.

3.5.3 Exposure to food simulant

Insert a thermocouple in the metal block (3.3). Place the metal block and thermocouple in the thermostatically controlled oven or incubator, set at the test temperature and leave for 24 h.

Observe the temperature of the metal block. Confirm that the temperature of the metal block reaches the test temperature, taking into account the tolerances specified in table B.1 of prEN 1186-1:2001.

NOTE 1 If the oven temperature is at the test temperature, but the temperature of the metal block is not, taking into account the tolerances specified in table B.1 of prEN 1186-1:2001, check the accuracy of the thermocouple and the temperature control of the oven. If necessary adjust the oven temperature, either by increasing or decreasing the oven set temperature, until the temperature of the metal block reaches the test temperature.

Take six of the glass tubes, mark them for identification purposes. Measure 100 ml \pm 5 ml of olive oil into each tube by measuring cylinder and stopper the tube.

NOTE 2 If the procedure described in annex D of prEN 1186-2:2001 is used, it may be necessary to dry all of the olive oil used for the migration test.

Alternatively mark the tubes for a volume of 100 ml and fill with olive oil to the mark. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Two extra tubes with a minimum of 50 ml of olive oil are required as blank simulant, if the procedure described in annex D of prEN 1186-2:2001 is used. Place the six or eight tubes, and two empty tubes, in the metal block or blocks (3.3) then put the metal block or blocks in the thermostatically controlled oven or incubator (5.12 of prEN 1186-2:2001) set at the test temperature. Leave until the olive oil has attained the test temperature, using the thermometer or thermocouple to monitor the temperature.

NOTE 3 More rapid temperature equilibration of the oil can be established by lifting the tubes from the block periodically and rotating gently before replacing rapidly or by stirring the oil in the tubes with a metal or glass rod, without removing from the block.

Place into four of the tubes containing olive oil, weighed test specimens prepared as in 3.4 and conditioned if necessary. Stopper the tubes. Ensure that the test specimens are totally immersed in olive oil; if they are not, then add either glass beads or glass rods (5.22 of prEN 1186-2:2001) to raise the level of the olive oil until total immersion is achieved.

WARNING 1 Take care when handling the hot metal block or blocks during removal from and replacement to the oven or incubator, to prevent skin burns. Take particular care when placing the test specimens in the hot olive oil to prevent splashing or spillage of the olive oil on to the skin. It is recommended that heat protective gloves and a face shield are worn.

NOTE 4 The olive oil in the fifth tube is used as a reference standard in constructing the calibration graph and if the procedure described in annex D of prEN 1186-2:2001 is used, as the third blank sample for Karl Fischer titrations. The olive oil in the sixth tube is used to check the temperature of the oil. If glass beads or glass rods have been used to raise the level of the olive oil to achieve total immersion, then similar glass beads or glass rods should be added to the sixth tube.

Place one test specimen in the sixth tube where the temperature of the oil is being monitored.

NOTE 5 If a test specimen is not added to the tube the temperature monitored will be high in comparison with temperature of the oil in the other tubes containing test specimens. For the determination of migration from thin films, which have little thermal capacity, it is not necessary to include a test specimen in the sixth tube, only the supports and gauzes as used to mount the test specimens. For thicker materials, which will have some thermal capacity, it should consist of an extra test specimen.

Place the remaining two test specimens into the empty tubes and stopper.

NOTE 6 These two test specimens are used to check whether the sample loses mass from the evaporation of volatiles, such as solvents, during the test period

Replace the block or blocks containing all eight or ten tubes 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 to prevent undue heat loss.

NOTE 7 Where the test time for migration test is between 0,5 h and 4 h, it is particularly important that the time period between placing the test specimens in the olive oil and the olive oil reaching the test temperature, be kept as short as possible. This time period can be minimized by placing the test specimens in the tubes without removing the tubes from the block and without removing the block from the oven or incubator. It may be necessary to raise the tubes from the block to check that the oil is above the level of the sample and that the test pieces remain separated. The tubes should be raised the minimum amount necessary to carry out these checks and then replaced as quickly as possible. The sixth tube, i.e. the tube in which the temperature is monitored, should be treated in a similar manner.

Observe the temperature of the thermostatically controlled oven or incubator or the olive oil in the sixth tube and leave the tubes for the selected test period, taking into account the tolerances specified in table B.1 of prEN 1186-1:2001, after the olive oil in the sixth tube has reached a temperature within the tolerance specified in table B.2 of prEN 1186-1:2001. For test temperatures between 100 °C and 150 °C the time between the immersion of the test specimens and the test temperature being regained shall be 10 min or less. For test temperatures between 151 °C and 175 °C the time shall be 15 min or less.

NOTE 7 It has been found that these times can be achieved by using silicone oil in the wells to increase thermal conductivity between the block and the tube.

NOTE 8 Annex B of prEN 1186-1:2001 includes tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant to this Part of the standard.

Take the metal block or blocks containing the tubes from the oven or incubator and immediately remove the test specimens from the tubes. Discard the test specimen that was in the sixth tube in which the temperature had been monitored. For the remaining specimens which have been in olive oil, allow the oil to drain.

WARNING 2 Take care when handling the metal block or blocks and removal of the test specimens from the olive oil at the end of the exposure period, to prevent skin burns. Use the protective wear recommended in WARNING 1.

Remove any adhering olive oil by gently pressing between filter papers (5.13 of prEN 1186-2:2001). Repeat the pressing procedure until the filter paper shows no spots of olive oil. For test specimens on supports, remove the individual test pieces from the supports to carry out this operation. Clean the supports of oil by washing with the extraction solvent and replace the test pieces on them.

3.5.4 Final weighing of test specimens

Perform the final weighing in accordance with 7.4 of prEN 1186-2:2001.

3.5.5 Extraction of absorbed olive oil

Extract the absorbed oil in accordance with 7.5 of prEN 1186-2:2001.

3.5.6 Determination of extracted olive oil

Determine the extracted olive oil in accordance with 7.6 of prEN 1186-2:2001.

3.6 Expression of results

3.6.1 Method of calculation

Express the results in accordance with 8.1 of prEN 1186-2:2001.

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