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

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Preview

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Will supersede EN 1186-13:2002

English Version

**Materials and articles in contact with foodstuffs - Plastics - Part
13: Test method 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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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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Foreword

This document (prEN 1186-13:2006) 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 CEN Enquiry.

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 document will supersede EN 1186-13:2002.

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

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 1186-13 Method A and Method B should be read in conjunction with EN 1186-1. Also, EN 1186-13 Method A should be read in conjunction with EN 1186-2.

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 case where incomplete extraction of olive oil occurs)*
- Part 11 *Test methods for overall migration into mixtures of 14C-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*

Preview
prEN 1186-13:2006

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.

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Preview

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 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 provided 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 or 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 or 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 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 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 EN 1186-1:2002.

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 EN 1186-1:2002. 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.

3.2 Reagents

The reagents shall be as described in clause 4 of EN 1186-2:2002.

3.3 Apparatus

The apparatus shall be as described in clause 5 of EN 1186-2:2002, with the addition of:

Aluminium block or blocks with wells for holding up to ten glass tubes, see 5.11 of EN 1186-2:2002, 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 EN 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 EN 1186-2:2002.

3.5.2 Initial weighing of test specimens

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

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