

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

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

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN 1186-13:2003 sisaldab Euroopa standardi EN 1186-13:2002 ingliskeelset teksti.	This Estonian standard EVS-EN 1186-13:2003 consists of the English text of the European standard EN 1186-13:2002.
Käesolev dokument on jõustatud 18.02.2003 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.	This document is endorsed on 18.02.2003 with the notification being published in the official publication of the Estonian national standardisation organisation.
Standard on kättesaadav Eesti standardiorganisatsioonist.	The standard is available from Estonian standardisation organisation.

<b>Käsitlusala:</b> 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	<b>Scope:</b> 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
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ICS 67.250

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

This European Standard was approved by CEN on 29 April 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



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## Foreword

This document EN 1186-13:2002 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 1186-13:1999.

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.

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 1186-13 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 cases where incomplete extraction of olive oil occurs)
Part 11	Test methods for overall migration into mixtures of <sup>14</sup> 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.

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.

## 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 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 produce different chromatograms for the simulant methyl esters to those of the methyl esters of olive oil. Suitable chromatogram peaks of the methyl esters of the other fatty food simulants should be selected 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 <sup>14</sup>C-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 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 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 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.