

Materials and articles in contact with foodstuffs — Plastics —

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

The European Standard EN 1186-14:2002 has the status of a British Standard

ICS 67.250

National foreword

This British Standard is the official English language version of EN 1186-14:2002. It supersedes DD ENV 1186-14:1999 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee CW/47, Materials and articles in contact with foodstuffs, to Subcommittee CW/47/1, Migration from plastics, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

This British Standard, having been prepared under the direction of the Consumer Products and Services Sector Policy and Strategy Committee, was published under the authority of the Standards Policy and Strategy Committee on 14 October 2002

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 20, an inside back cover and a back cover.

The BSI copyright date displayed in this document indicates when the document was last issued.

Amendments issued since publication

Amd. No.	Date	Comments

© BSI 14 October 2002

ISBN 0 580 40570 2

English version

Materials and articles in contact with foodstuffs - Plastics - 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

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 14: Méthodes d'essai pour des 'tests de substitution' pour la migration globale des matières destinées au contact avec les denrées alimentaires gras en utilisant des liquides simulateurs tels que l'isooctane et l'éthanol à 95 %

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 14: Prüfverfahren für "Ersatzprüfungen" für die Gesamtmigration aus Kunststoffen, die für den Kontakt mit fettigen Lebensmitteln bestimmt sind, unter Verwendung der Prüfmedien Iso-Octan und 95 %igem Ethanol

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.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

	page
Foreword.....	3
1 Scope	4
2 Normative references	4
3 Total immersion method	5
3.1 Principle	5
3.2 Reagents	5
3.3 Apparatus	5
3.4 Preparation of test specimens.....	6
3.4 Procedure	6
3.4.1 Exposure to test media	6
3.4.2 Determination of migrating substances	7
3.5 Expression of results	7
3.6 Test report	7
4 Cell method.....	8
4.1 Principle	8
4.2 Reagents	8
4.3 Apparatus	8
4.4 Preparation of test specimens.....	9
4.5 Procedure	9
4.5.1 Exposure to test media	9
4.5.2 Determination of migrating substances	10
4.6 Expression of results	10
4.7 Test report	10
5 Pouch method	11
5.1 Principle	11
5.2 Reagents	11
5.3 Apparatus	11
5.4 Preparation of test specimens.....	12
5.5 Procedure	12
5.5.1 Exposure to test media	12
5.5.2 Determination of migrating substances	13
5.6 Expression of results	13
5.7 Test report	13
6 Article filling method	14
6.1 Principle	14
6.2 Reagents	14
6.3 Apparatus	14
6.4 Preparation of test specimens.....	15
6.5 Procedure	15
6.5.1 Exposure to test media	15
6.5.2 Determination of migrating substances	16
6.6 Expression of results	16
6.7 Test report	16
Annex A (informative) Example of a pouch holder	17
Annex ZA (informative) Relationship of this European Standard with Council Directive 89/109/EEC and Commission Directive 90/128/EEC and associated Directives.....	18
Bibliography	20
2	

Foreword

This document EN 1186-14 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 1186-14:1999.

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

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

WARNING — Both iso-octane and ethanol are volatile flammable solvents. Take care to ensure that the test specimens are well stoppered, closed and covered to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator and generating an explosive mixture. Care should be taken at all times when handling these solvents to prevent contact with sources of ignition.

EN 1186-14 should be read in conjunction with EN 1186-1, EN 1186-3, EN 1186-5, EN 1886-7, and EN 1186-9.

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	3
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 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 13	Test methods for overall migration at high temperatures	

Part 15 Alternative test methods to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol

Annex A 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 'substitute tests' performed with volatile test media, iso-octane and 95 % v/v aqueous ethanol, for the determination of overall migration from plastics intended to come into contact with fatty foodstuffs at all temperatures and for any period of time.

These test methods are suitable for plastics samples in a wide variety of forms.

NOTE 1 The iso-octane and 95 % v/v aqueous ethanol volatile test media used in these test methods are those specified for 'substitute tests' in Commission Directive 82/711/EEC [3] and its subsequent amendments [4], [5]. In addition to the use of iso-octane and 95 % v/v aqueous ethanol as test media for 'substitute tests', Commission Directive 82/711/EEC and its subsequent amendments specifies the use of modified polyphenylene oxide as a test medium for use at temperatures of 100 °C and above. For a test method for overall migration from plastics intended to come into contact with fatty foodstuffs using modified polyphenylene oxide as a test medium, see EN 1186-13.

NOTE 2 These test methods can also be used for the 'alternative tests' described in Council Directive 82/711/EEC and its subsequent amendments, when the chosen volatile test media are iso-octane and 95 % aqueous ethanol, provided equivalence is shown with olive oil, see EN 1186-1:2002.

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).

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 1186-3:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 3: Test methods for overall migration into aqueous food simulants by total immersion.*

EN 1186-5:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 5: Test methods for overall migration into aqueous food simulants by cell.*

EN 1186-7:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 7: Test methods for overall migration into aqueous food simulants using a pouch.*

EN 1186-9:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 9: Test methods for overall migration into aqueous food simulants by article filling.*

EN 10088-1:1995, *Stainless steels - Part 1: List of stainless steels.*

ISO 648, *Laboratory glassware - One mark pipettes.*

ISO 4788, *Laboratory glassware - Graduated measuring cylinders.*

3 Total immersion method

3.1 Principle

The overall migration of non-volatile substances from a sample of the plastics material or article into the test medium is determined as the mass of non-volatile residue after evaporation of the test medium following immersion.

The selection of the conditions of test is determined by the conditions of use, see clauses 6 and 7 of EN 1186-1:2002.

Test specimens of approximately 1 dm², see clause 9 of EN 1186-1:2002, are immersed in a test medium for set periods of time and at set temperatures. At the end of the test period, each test specimen is removed from the test medium. The test medium from each test is evaporated to dryness, the mass of the non-volatile residue is determined gravimetrically and expressed as milligrams per square decimetre of surface area of test specimen.

Overall migration is reported as the mean of three determinations on separate test specimens.

3.2 Reagents

3.2.1 Iso-octane, (2,2,4-trimethyl pentane), purity 98,5 % (v/v) or greater, CAS No. 540-84-1¹.

3.2.2 Ethanol, purity 96 % (v/v) or greater, 95 % (v/v) in aqueous solution.

WARNING — Both these solvents are flammable. Take care at all times when handling these solvents to prevent contact with sources of ignition.

3.3 Apparatus

3.3.1 Cutting slab, clean smooth glass, metal or plastics slab of suitable area to prepare test specimens, 250 mm × 250 mm is suitable.

3.3.2 Tweezers, stainless steel, blunt nosed.

3.3.3 Cutting implement, scalpel, scissors or sharp knife or other suitable device.

3.3.4 Metal template, 100 mm ± 0,2 mm x 100 mm ± 0,2 mm (square).

3.3.5 Rule or template, 25 mm ± 1 mm wide.

3.3.6 Rule, graduated in mm, and with an accuracy of 0,1 mm.

3.3.7 Analytical balance capable determining a change in mass of 0,1 mg.

3.3.8 Specimen supports, constructed of stainless steel with cross arms attached by welding or silver soldering, or of glass. Stainless steel X4 CrNi 18 10 according to EN 10088-1:1995 or of composition, chromium 17 %, nickel 9 %, carbon 0,04 %, is suitable. Before initial use thoroughly clean the stainless steel supports. The use of a degreasing solvent and then with dilute nitric acid has been found to be suitable.

NOTE The method has been written for the supports shown in Figure C.1 of prEN 1186-1:2002 which have been found to be suitable for holding thin film and sheet test pieces. However other supports can be used providing they are capable of

¹The source of this is the Chemical Abstracts published by the American Chemical Society

holding and keeping the test pieces apart and at the same time ensuring complete contact with the test medium. For rigid samples, supports with a single cross arm can be used.

3.3.9 Gauze, pieces of fine stainless steel gauze, with a mesh size of 1 mm have been found to be suitable, approximately 25 mm × 100 mm or, glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long for insertion between the test pieces. Before initial use thoroughly clean the gauze, first with a degreasing solvent and then with dilute nitric acid.

3.3.10 Glass tubes, ground neck with stoppers, for retaining the test medium and test specimens. Tubes with an internal diameter of approximately 35 mm and length in the range of 100 mm to 200 mm, excluding the ground neck, see 8.2 of EN 1186-1:2002, have been found to be satisfactory.

3.3.11 Glass beads, 2 mm to 3 mm diameter, or glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long, see 8.2 of EN 1186-1:2002.

3.3.12 Thermostatically controlled oven, incubator or refrigerator capable of maintaining a temperature within the range of 5 °C to 60 °C and within the tolerances specified in Table B.2 of EN 1186-1:2002.

WARNING — The interior/sample space of the oven, incubator or refrigerator should not have any exposed heating elements, to minimise safety hazards arising from any loss of the flammable test media from the tubes during the test period.

3.3.13 Dishes, stainless steel, nickel, platinum, platinum alloy, gold 50 mm to 90 mm diameter and maximum weight 100 g, for evaporation of test media and weighing of residues. Glass, glass ceramic or ceramic dishes may be used provided that the surface characteristics are such that the weights of the dishes after evaporation of any specified test media followed by conditioning in the desiccator used achieves a constancy of ± 0,5 mg.

3.3.14 Steam bath, hot plate, distillation apparatus or rotary evaporator for evaporation of test medium at the end of test period.

3.3.15 Desiccator with anhydrous calcium chloride or self indicating silica gel.

3.3.16 Measuring cylinder, 100 ml, complying with the minimum requirements of ISO 4788.

3.3.17 Thermometer or electronic temperature measuring instrument with thermocouple, capable of measuring temperature in the range 5 °C to 60 °C, with a precision of 0,1 °C.

3.4 Preparation of test specimens

Prepare the test specimens in accordance with 3.4 of EN 1186-3:2002.

3.4 Procedure

3.4.1 Exposure to test media

Take three of the glass tubes, for the test specimens and a further two to provide blanks, measure by measuring cylinder 100 ml ± 2 ml of the test medium into each tube and stopper the tube. If the evaporation method is to be used measure into a further two tubes, by measuring cylinder, 120 ml ± 2 ml of the test medium, to provide blanks. If the distillation method is to be used measure into those further two tubes by measuring cylinder 100 ml ± 2 ml of the test medium to provide blanks. Insert a thermometer or thermocouple in one of the tubes containing 100 ml of test medium, then stopper all five tubes. Place the five tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature, and leave until the test medium has attained the test temperature.

Place a test specimen into each of the three tubes containing 100 ml of test medium, re-insert the thermometer or thermocouple and re-stopper the tubes. Mark the tubes for identification. Ensure that the test specimens are totally immersed in the test medium; if they are not then add either glass beads or rods to raise the level of the test medium until total immersion is achieved. This part of the operation should be carried out in the minimum time to prevent undue heat loss from the test medium.

Mark the liquid level on the outside of each tube with a suitable marker.

Replace all of the tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature. Observe the temperature and leave the tubes for the selected test period, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002, after the temperature in the tube has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

WARNING 1 — Both iso-octane and ethanol are volatile flammable solvents. Take care to ensure that the tubes are well stoppered to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator and generating an explosive mixture.

WARNING 2 — If possible place the tubes in a drip container capable of holding the total volume of volatile simulant in case of accident.

WARNING 3 — To minimise hazards arising due to the volatile and flammable nature of the two test media the maximum test temperature is 60 °C. Do not conduct the tests at temperatures above 60 °C.

Take the tubes from the oven, incubator or refrigerator and check the level of test medium in each, if this has fallen to more than 10 mm below the mark, or has exposed any part of the test pieces, repeat the test using fresh test specimens.

NOTE For exposure times of more than 24 h it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator, instead of the temperature of the simulant.

If the level of test medium in a tube is less than 10 mm below the mark, remove the test specimen from the tube, and allow the test medium adhering to the test specimen and support to drain back into the tube. Recover at least 90 % of the original volume of test medium or repeat the test.

3.4.2 Determination of migrating substances

Determine the migrating substances in accordance with 3.5 of EN 1186-3:2002.

WARNING — Both iso-octane and ethanol are volatile and flammable solvents. Take care when evaporating these test media to prevent vapours contacting sources of ignition, particularly when using a hot plate to carry out the evaporation. The evaporation should be carried out in a fume cupboard.

3.5 Expression of results

Calculate the results in accordance with 3.6 of EN 1186-3:2002.

3.6 Test report

The test report shall include the following, see clause 12 of EN 1186-1:2002:

- a) reference to this European Standard and to the Part used for the test procedure;
- b) all information necessary for complete identification of the sample such as chemical type, supplier, trade mark, grade, batch number, thicknesses;
- c) conditions of time and temperature of exposure to test media;
- d) departures from the specified procedure, and reasons for these;
- e) individual test results, and the mean of these, expressed as milligrams of residue per square decimetre of sample;
- f) relevant comments on the test results.

4 Cell method

4.1 Principle

The overall migration of non-volatile substances from a sample of the plastics material or article into the test medium, is determined as the mass of non-volatile residue after evaporation of the test medium.

The selection of the conditions of test are determined by the conditions of use, see clauses 6 and 7 of EN 1186-1:2002.

One surface of the test specimen is exposed in a cell to a test medium for set periods of time and at set temperatures. At the end of the test period, each test specimen is removed from contact with the test medium. The test medium from each test is evaporated to dryness, the mass of the non-volatile residue is determined gravimetrically and expressed as milligrams per square decimetre of surface area of test specimen, which had been in contact with the test medium.

Overall migration is reported as the mean of three determinations on separate test specimens.

4.2 Reagents

4.2.1 Iso-octane (2,2,4-trimethyl pentane), purity 98,5 % (v/v) or greater, CAS No. 540-84-1.

4.2.2 Ethanol, purity 96 % (v/v) or greater, 95 % (v/v) in aqueous solution.

WARNING — Both these solvents are flammable. Take care at all times when handling these solvents to prevent contact with sources of ignition.

4.3 Apparatus

4.3.1 Cutting slab, clean smooth glass, metal or plastics slab of suitable area to prepare test specimens, 250 mm × 250 mm is suitable.

4.3.2 Tweezers, stainless steel, blunt nosed.

4.3.3 Cutting implement, scalpel, scissors or sharp knife or other suitable device.

4.3.4 Rule, graduated in mm, and with an accuracy of 0,1 mm.

4.3.5 Analytical balance capable of determining a change in mass of 0,1 mg.

4.3.6 Cell type A, as shown in Figure C.3 of EN 1186-1:2002, either the all aluminium (anodised) cells or the cells with the stainless steel (316 grade) lids and rings. The internal diameter of the rib of the sealing ring shall be 178,4 mm ± 0,1 mm, to give an area of the test specimen exposed to the test medium of 2,5 dm². It is necessary to modify the filler plug on one of the cells to allow a thermometer or thermocouple to be inserted into the test medium, whilst maintaining an effective seal to prevent loss of test medium during the test period.

NOTE The cell type A is constructed with a rubber mat in the base plate. It is advised that a disc of aluminium foil is placed on the mat before inserting the test specimen. The use of these discs will prevent any substances from the mat influencing the migration result.

For details of equivalent cells see 8.3 of EN 1186-1:2002.

4.3.7 Pipettes, complying with the minimum requirements of ISO 648, 50 ml and 100 ml.

4.3.8 Glass tubes, ground neck, and stoppers, for retaining the test medium. Tubes with an internal diameter of approximately 35 mm and length of approximately 100 mm to 200 mm, excluding the ground neck, see 8.2 of EN 1186-1:2002, have been found to be satisfactory.

4.3.9 Thermostatically controlled oven, incubator or refrigerator capable of maintaining a temperature within the range 5 °C to 60 °C and within the tolerances specified in Table B.1 of EN 1186-1:2002.

WARNING — The interior/sample space of the oven, incubator or refrigerator should not have any exposed heating elements, to minimise safety hazards arising from any loss of the flammable test media from the tubes and cells.

4.3.10 Dishes, stainless steel, nickel, platinum, platinum alloy, gold, 50 mm to 90 mm diameter and maximum weight 100 g, for evaporation of test media and weighing of residues. Glass, glass ceramic or ceramic dishes may be used provided that the surface characteristics are such that the weights of the dishes after evaporation of any specified test media followed by conditioning in the desiccator used achieves a constancy of $\pm 0,5$ mg.

4.3.11 Steam bath, hot plate, distillation apparatus or rotary evaporator for evaporation of test medium at the end of test period.

4.3.12 Desiccator with anhydrous calcium chloride or self indicating silica gel.

4.3.13 Measuring cylinders, 250 ml, complying with the minimum requirements of ISO 4788.

4.3.14 Thermometer or electronic temperature measuring instrument, with thermocouple, capable of measuring temperature in the range 5 °C to 60 °C, with precision of 0,1 °C.

4.4 Preparation of test specimens

Prepare the test specimens in accordance with clause 6 of EN 1186-5:2002.

4.5 Procedure

4.5.1 Exposure to test media

Take three cells, mark these for identification purposes. Place in the thermostatically controlled oven, incubator or refrigerator, which is set at the selected test temperature and leave until the test temperature has been attained.

Take three glass tubes, measure by measuring cylinder 125 ml \pm 2 ml of the test medium into each tube, stopper the tube and mark the level of the liquid on the outside, together with a mark for identification. If the evaporation method is to be used measure into a further two tubes by measuring cylinder 185 ml \pm 2 ml of the test medium, to provide blanks. If the distillation method is to be used measure into a further two tubes by measuring cylinder 125 ml \pm 2 ml of the test medium to provide blanks.

Mark the liquid level on the outside of each tube with a suitable marker. Insert a thermometer or thermocouple in one of the tubes containing 125 ml of test medium, then stopper all five tubes. Place the five tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature, and leave until the test medium has attained the test temperature.

Remove the cells from the thermostatically controlled oven, incubator or refrigerator, dismantle and place on the base of each cell one of the test specimens. Reassemble the cells, ensuring that the clamping screw wheel is well tightened down.

Remove three tubes containing 125 ml of test medium from the thermostatically controlled oven, incubator or refrigerator and transfer the test medium from each tube to each of the test cells through the filler hole.

Replace the filler plugs and in one of the cells insert the thermometer or thermocouple. This part of the operation should be carried out in the minimum of time to prevent undue heat loss from the test medium. Carefully check each cell for any leakage of simulant. If any leakage is observed reject that cell from further tests.

WARNING 1 — Never place a leaking cell in the oven.

Return the test cells to the thermostatically controlled oven, incubator or refrigerator, set at the test temperature. Observe the temperature and leave the cells and tubes for the selected test period, taking into account the

tolerances specified in Table B.1 of EN 1186-1:2002 after the test medium in the cell has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

WARNING 2 — Both iso-octane and ethanol are volatile flammable solvents. Take care to ensure that the tubes are well stoppered to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator and generating an explosive mixture and that the cells do not leak.

WARNING 3 — If possible place the tubes in a drip container capable of holding the total volume of volatile simulant in case of accident.

WARNING 4 — To minimise hazards arising due to the volatile and flammable nature of the two solvents, the maximum test temperature is 60 °C. Do not conduct the tests at temperatures above 60 °C.

NOTE For exposure times of more than 24 h it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator, instead of the temperature of the simulant.

Take the cells and the two tubes containing the blank test media from the thermostatically controlled oven, incubator or refrigerator.

Transfer by a 50 ml or 100 ml pipette the test medium from each of the three cells into the three tubes, check the level of test medium in each, if this has fallen to more than 10 mm below the mark, repeat the test with fresh test pieces. 90 % of the original volume of test medium has to be recovered.

Rinse each cell twice with 20 ml \pm 2 ml of test medium, add these rinses to the respective tubes.

4.5.2 Determination of migrating substances

Determine the migrating substances in accordance with clause 8 of EN 1186-5:2002.

WARNING — Both iso-octane and ethanol are volatile and flammable solvents. Take care when evaporating these test media to prevent vapours contacting sources of ignition, particularly when using a hot plate to carry out the evaporation. The evaporation should be carried out in a fume cupboard.

4.6 Expression of results

Calculate the results in accordance with clause 9 of EN 1186-5:2002.

4.7 Test report

The test report shall include the following:

- a) reference to this European Standard and to the Part used for the test procedure;
- b) all information necessary for complete identification of the sample such as chemical type, supplier, trade mark, grade, batch number(s), thickness;
- c) conditions of time and temperature of exposure to test media;
- d) departures from the specified procedure and reasons for these;
- e) individual test results and the mean of these expressed as milligrams lost per square decimetre of sample;
- f) relevant comments on the test results.

5 Pouch method

5.1 Principle

The overall migration of non-volatile substances from a sample of the plastics material or article into the test medium, is determined as the mass of non-volatile residue after evaporation of the test medium.

The selection of the conditions of test is determined by the conditions of use, see clauses 6 and 7 of EN 1186-1:2002.

Test specimens in the form of pouches are filled with a test medium and stored for set periods of time and at set temperatures. At the end of the test period the test medium is removed from each pouch. The test medium from each test is evaporated to dryness, the mass of the non-volatile residue is determined gravimetrically and expressed as milligrams per square decimetre of test specimen surface area which had been in contact with the test medium.

Overall migration is reported as the mean of three determinations on separate test specimens.

5.2 Reagents

5.2.1 Iso-octane (2,2,4-trimethyl pentane), purity 98,5 % (v/v) or greater, CAS No. 540-84-1.

5.2.2 Ethanol, purity 96 % (v/v) or greater, 95 % (v/v) in aqueous solution.

WARNING — Both these solvents are flammable. Take care at all times, when handling these solvents, to prevent contact with sources of ignition.

5.3 Apparatus

5.3.1 Cutting slab, clean smooth glass, metal or plastics slab of suitable area to prepare test specimens, 250 mm × 250 mm is suitable.

5.3.2 Tweezers, stainless steel, blunt nosed.

5.3.3 Cutting implement, scalpel, scissors or sharp knife or other suitable device.

5.3.4 Rule, graduated in mm, and with an accuracy of 0,1 mm.

5.3.5 Metal template (120 mm ± 1 mm) x (120 mm ± 1 mm) (square).

5.3.6 Analytical balance capable of determining a change in mass of 0,1 mg.

5.3.7 Pouch holder, the example shown in Figure A.1 has been shown to be suitable, constructed from aluminium or other suitable material or an equivalent holder, plus clips to secure corners of pouches.

5.3.8 Pipettes, complying with the minimum requirements of ISO 648, 50 ml and 100 ml.

5.3.9 Glass tubes, ground neck, and stoppers, for retaining the test medium and test specimens. Tubes with an internal diameter of approximately 35 mm and length in the range of 100 mm to 200 mm, excluding the ground neck, see 8.2 of EN 1186-1:2002, have been found to be satisfactory.

5.3.10 Thermostatically controlled oven, incubator or refrigerator capable of maintaining a temperature in the range of 5 °C to 60 °C and within the tolerances specified in Table B.2 of EN 1186-1:2002.

WARNING — The interior/sample space of the oven, incubator or refrigerator should not have any exposed heating elements, to minimise safety hazards arising from any loss of the flammable test media from the tubes and cells.

5.3.11 Dishes, stainless steel, nickel, platinum, platinum alloy, gold 50 mm to 90 mm diameter and maximum weight 100 g, for evaporation of test media and weighing of residues. Glass, glass ceramic or ceramic dishes may be used provided that the surface characteristics are such that the weights of the dishes after evaporation of any specified test media followed by conditioning in the desiccator used achieves a constancy of $\pm 0,5$ mg.

5.3.12 Steam bath, hot plate, distillation apparatus or rotary evaporator for evaporation of test medium at the end of test period.

5.3.13 Desiccator with anhydrous calcium chloride or self indicating silica gel.

5.3.14 Heat or pressure sealing device, for use in forming pouches.

5.3.15 Measuring cylinders, 100 ml, complying with the minimum requirements of ISO 4788.

5.3.16 Thermometer or electronic temperature measuring instrument with thermocouple, capable of measuring temperature in the range 5 °C to 60 °C, with a precision of 0,1 °C.

5.4 Preparation of test specimens

Prepare the test specimens in accordance with clause 6 of EN 1186-7:2002.

5.5 Procedure

NOTE Before starting a migration experiment, using the pouch method, a check should be made on whether the test material is sufficiently resistant to the volatile simulant. A pouch should be prepared, filled with the simulant and the weight of the pouch before and after filling determined. The pouch should be stored at room temperature in a fumehood and, after 48 h, the pouch and its contents reweighed. If the loss of weight is more than 1 % of the initial weight of the volatile simulant in the pouch then, for safety reasons, the pouch method should not be applied.

5.5.1 Exposure to test media

Take three glass tubes, measure by measuring cylinder 100 ml \pm 2 ml of the test medium into each tube and stopper the tubes. If the evaporation method is to be used measure into a further two tubes by measuring cylinder 120 ml \pm 2 ml of the test medium, to provide blanks. If the distillation method is to be used, measure into a further two tubes by measuring cylinder 100 ml \pm 2 ml of the test medium to provide blanks. Insert a thermometer or thermocouple in one of the tubes containing 120 ml of test medium, then stopper all five tubes. Place the five tubes and the pouch holder in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature and leave until the test medium has attained the test temperature.

Remove the pouch holder from the thermostatically controlled oven, incubator or refrigerator and place between the spacers the test specimen pouches.

Remove the three tubes containing the 100 ml of test medium from the thermostatically controlled oven, incubator or refrigerator and into the three test specimens pouches pipette sufficient test medium to just fill the pouch. This shall be about 100 ml, but for thick/semi-rigid materials the quantity can be less. Insert the thermometer or thermocouple into the open corner of one of the pouches and secure the open corners of all three with clips. If all of the test medium is not used to fill the pouch, retain the tube and residual contents. Measure and record the volume of the residual test medium. Measure and record the area of the pouch in contact with the test medium and the total external area of the pouch after trimming excess material. This part of the operation should be carried out in the minimum time to prevent undue heat loss. Carefully check the pouches for leakage before placing the pouches in the oven.

Replace the pouch holder, containing the test specimens pouches, in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature. Observe the temperature and leave the pouches and tubes for the selected test period, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002, after the test medium in the pouch has reached a temperature within the tolerances specified in Table B.2 of EN 1186-1:2002.

WARNING 1 — Both iso-octane and ethanol are volatile flammable solvents. Take care to ensure that the tubes are well stoppered and pouches are carefully closed at the corner to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator and generating an explosive mixture.

WARNING 2 — For safety reasons do not load an oven with more than the test specimens of one test sample.

WARNING 3 — Place the pouches and tubes in a drip container capable of holding the total volume of volatile simulant in case of an accident.

WARNING 4 — To minimise hazards arising due to the volatile and flammable nature of the two solvents, the maximum test temperature is 60 °C. Do not conduct the tests at temperatures above 60 °C.

NOTE For exposure times of more than 24 h it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator, instead of the temperature of the simulant.

Take the pouch holder and the tubes containing the blank test medium from the thermostatically controlled oven, incubator or refrigerator.

If an evident leak has occurred with one of the pouches, the test is invalid and shall be repeated using fresh pouches.

If no evident leaks have occurred in any of the three pouches, then proceed with 5.5.2.

5.5.2 Determination of migrating substances

Determine the migrating substances in accordance with 7.2 of EN 1186-7:2002.

WARNING — Both iso-octane and ethanol are volatile and flammable solvents. Take care when evaporating these test media to prevent vapours contacting sources of ignition, particularly when using a hot plate to carry out the evaporation. The evaporation should be carried out in a fume cupboard.

5.6 Expression of results

Calculate the results in accordance with clause 8 of EN 1186-7:2002.

5.7 Test report

The test report shall include the following, see clause 12 of EN 1186-1:2002:

- a) reference to this European Standard and to the Part used for the test procedure;
- b) all information necessary for complete identification of the sample such as chemical type, supplier, trade mark, grade, batch number(s), thickness;
- c) conditions of time and temperature of exposure to test media;
- d) departures from the specified procedure and reasons for these;
- e) individual test results and the mean of these expressed as milligrams lost per square decimetre of sample;
- f) relevant comments on the test results; including the area of the pouch in contact with the test medium, the total external area of the pouch after trimming excess material and the volume of the residual test medium.

6 Article filling method

6.1 Principle

The overall migration of non-volatile substances from a sample of the plastics material or article into the test medium is determined as the mass of non-volatile residue after evaporation of the test medium.

The selection of the conditions of test is determined by the conditions of use, see clauses 6 and 7 of EN 1186-1:2002.

Test specimens are filled with a test medium and stored for set periods of time and at set temperatures. At the end of the test period the test medium is removed from each test specimen. The test medium is evaporated to dryness, the mass of the non-volatile residue is determined gravimetrically and expressed as milligrams per square decimetre of test specimen surface area exposed to the test medium, or milligrams lost per kilogram of test medium.

Overall migration is reported as the mean of three determinations on separate test specimens.

6.2 Reagents

6.2.1 Iso-octane (2,2,4-trimethyl pentane), purity 98,5 % or greater, CAS No. 540-84-1.

6.2.2 Ethanol, purity 96 % or greater, 95 % (v/v) in aqueous solution.

WARNING — Both these solvents are flammable. Take care, at all times when handling these solvents, to prevent contact with sources of ignition.

6.3 Apparatus

6.3.1 Analytical balance capable of determining a change in mass of 0,1 mg.

6.3.2 Lint-free cloth.

6.3.3 Stoppered flask, 2 l.

6.3.4 Glass beads, 2 mm to 3 mm diameter.

6.3.5 Thermostatically controlled oven, incubator or refrigerator capable of maintaining a temperature within the range of 5 °C to 60 °C and within the tolerances specified in Table B.2 of EN 1186-1:2002.

WARNING — The interior/sample space of the oven, incubator or refrigerator should not have any exposed heating elements, to minimise safety hazards arising from any loss of the flammable test media from the flask and test specimens.

6.3.6 Dishes, stainless steel, nickel, platinum, platinum alloy, gold 50 mm to 90 mm diameter and maximum weight 100 g, for evaporation of test media and weighing of residues. Glass, glass ceramic or ceramic dishes may be used provided that the surface characteristics are such that the weights of the dishes after evaporation of any specified test media followed by conditioning in the desiccator used, achieves a constancy of $\pm 0,5$ mg.

6.3.7 Steam bath, hot plate, distillation apparatus or rotary evaporator for evaporation of test medium at the end of test period.

6.3.8 Desiccator with anhydrous calcium chloride or self indicating silica gel.

6.3.9 Beakers, 250 ml.

6.3.10 Pipette, 200 ml, complying with the minimum requirements of ISO 648.

6.3.11 Thermometer or electronic temperature measuring instrument with thermocouple, capable of measuring temperature in the range 5 °C to 60 °C, with a precision of 0,1 °C.

6.4 Preparation of test specimens

Prepare the test specimens in accordance with clause 6 of EN 1186-9:2002.

6.5 Procedure

NOTE Before starting a migration experiment, using the fill method, a check should be made on whether the test material is sufficiently resistant to the volatile simulant. For this purpose an article is filled with the simulant and then placed in a glass container, e.g. a desiccator, equilibrated at the required temperature and stored for twice the intended exposure time, up to a maximum of 48 hours. Observe the article or container for deformations. If no deformation is observed, then the exposure procedure is carried out using fresh articles.

6.5.1 Exposure to test media

Mark each of the articles making up each test specimen with an identification code.

Place, in a stoppered flask, a sufficient volume of the test medium to fill the three test specimens, prewarmed if necessary, and to provide two blanks, in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature. Insert a thermometer or thermocouple into the test medium and leave until the test temperature has been attained.

Remove the flask containing the test medium from the thermostatically controlled oven, incubator or refrigerator. Fill the three test specimens with test medium to within 0,5 cm of the top. If the container has a specified nominal volume of contents, see 9.2 of EN 1186-1:2002. Insert the thermometer or thermocouple into the test medium in one of the test specimens, cover and seal all test specimens with an inert material to prevent evaporation. Stopper the remaining test medium in the flask. This part of the operation should be carried out in the minimum time to prevent undue heat loss from the test medium.

WARNING 1 — Covering of filled articles is very important in respect to safety. Due to the variety of fillable articles it is impossible to prescribe one method for covering the filling opening. In general aluminium foil has appeared to be suitable in many cases. Also, a combination of glass plates with aluminium foil can be useful. Containers, like bottles and cups, are easily closed by carefully wrapping aluminium foil over the filling opening. Articles with large open areas, such as trays or dishes, should be covered with a glass plate of an appropriate size. The article and cover should then be placed on a sheet of aluminium foil, which is then folded around and over the article and glass plate. In this way a pouch is made that prevents evaporation of the simulant or reduces it to an acceptable level. To insert a thermocouple the glass plate should be provided with a hole that fits the thermocouple or that is large enough to accept a polytetrafluoroethylene stopper with the thermocouple.

WARNING 2 — For safety reasons do not load an oven with more than the test specimens of one test sample. In case were the capacity of the article is large, then the test specimens should be placed in the oven one at a time.

WARNING 3 — If possible place the article in a drip container capable of holding the total volume of volatile simulant in case of an accident.

Place the test specimens and test medium in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature. Observe the temperature and leave the test specimens and flask of test medium for the test period, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002, after the test medium in the test specimen has reached a temperature within the tolerances specified in Table B.2 of EN 1186-1:2002.

WARNING 4 — To minimise hazards arising due to the volatile and flammable nature of the two solvents, the maximum test temperature is 60 °C. Do not conduct the tests at temperatures above 60 °C.

NOTE For exposure times of more than 24 h it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator, instead of the temperature of the simulant.

6.5.2 Determination of migrating substances

Determine the migrating substances in accordance with 7.2 of EN 1186-9:2002.

WARNING — Both iso-octane and ethanol are volatile and flammable solvents. Take care when evaporating these test media to prevent vapours contacting sources of ignition, particularly when using a hot plate to carry out the evaporation. The evaporation should be carried out in a fume cupboard.

6.6 Expression of results

Calculate the results in accordance with clause 8 of EN 1186-9:2002.

6.7 Test report

The test report shall include the following, see clause 12 of EN 1186-1:2002:

- a) reference to this European Standard and to the Part used for the test procedure;
- b) all information necessary for complete identification of the sample such as chemical type, supplier, trade mark, grade, batch number, thickness, geometry of sample tested;
- c) conditions of time and temperature of exposure to test media;
- d) departures from the standard procedure, and reasons therefore;
- e) individual test results, and the mean of these, expressed as milligrams of residue per square decimetre of sample or milligrams lost per kilogram of test medium;
- f) relevant comments on the test results.

Annex A (informative)

Example of a pouch holder

Dimensions in millimetres

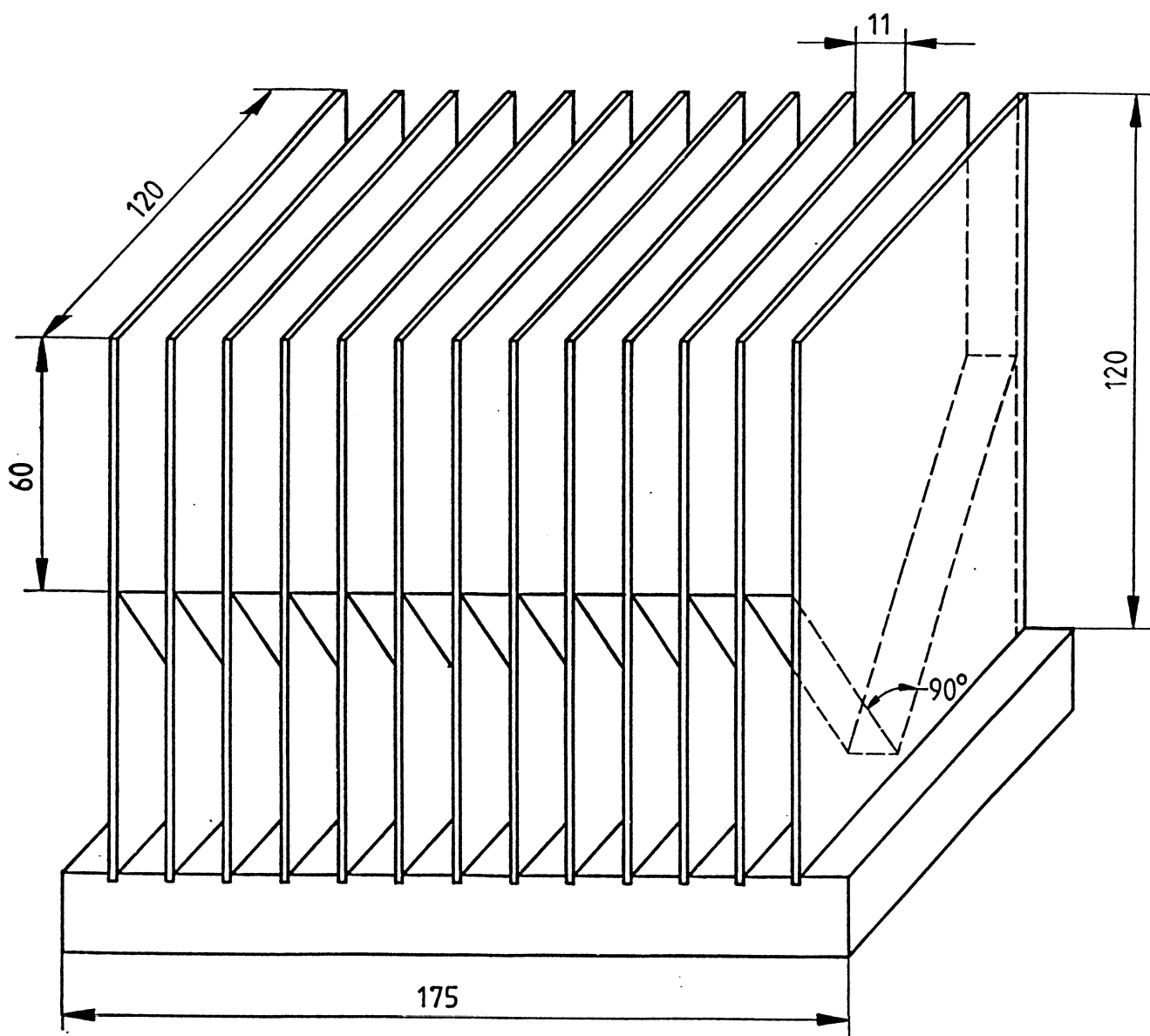


Figure A.1 — Pouch holder

Annex ZA (informative)

Relationship of this European Standard with Council Directive 89/109/EEC and Commission Directive 90/128/EEC and associated Directives

NOTE Other requirements and other EU Directives may be applicable to products falling within the scope of this standard.

The clauses of this standard are likely to support Directives 89/109/EEC [1], 90/128/EEC [2], 82/711/EEC [3] and its amendments 93/8/EEC [4] and 97/48/EC [5], and 85/572/EEC [6].

Compliance with this standard provides one means of conforming to the overall migration requirements of the Directive concerned and associated EFTA regulations.

European Commission Directive 90/128/EEC relating to plastics materials and articles intended to come into contact with foodstuffs, [2], specifies in article 2:

"Plastics materials and articles shall not transfer their constituents to foodstuffs in quantities exceeding 10 milligrams per square decimetre of surface area of materials or articles (overall migration limit). However this limit shall be 60 milligrams of constituents released per kilogram of foodstuff in the following cases:

- a) articles which are containers or are comparable to containers or which can be filled, with a capacity of not less than 500 ml and not more than 10 l;
- b) articles which can be filled and for which it is impracticable to estimate the surface area in contact with foodstuffs;
- c) caps, gaskets, stoppers or similar devices for sealing."

European Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs [3], and the subsequent amendments (Directives 93/8/EEC [4] and 97/48/EC [5]), recognizes that there are difficulties in the determination of the migration in food products and allows use of food simulants with conventional test conditions, which reproduce, as far as possible, the migration phenomena which may occur with contact between the article and foodstuffs. There are four food simulants:

- simulant A, distilled water or water of equivalent quality;
- simulant B, 3 % acetic acid (w/v) in aqueous solution;
- simulant C, 10 % ethanol (v/v) in aqueous solution;
- simulant D, rectified olive oil, or other fatty food simulants.

European Directive 82/711/EEC and the subsequent amendments also contain the conventional test conditions (time and temperature) for migration tests with food simulants. European Commission Directive 97/48/EC, the second amendment to European Council Directive 82/711/EEC, also contains test media and conventional test conditions for 'substitute tests', provision for alternative tests and more severe tests. Substitute tests may be performed in place of migration tests with simulant D, if it has been shown that for technical reasons connected with the method of analysis it is not feasible to obtain a valid test result in a migration test with simulant D.

European Council Directive 85/572/EEC laying down the list of simulants to be used for testing of constituents of plastics materials and articles intended to come into contact with foodstuffs [6] has a Table in the annex which contains a non-exhaustive list of foodstuffs and which identify the simulants to be used in migration tests on those plastic materials and articles intended to come into contact with a particular foodstuff or group of foodstuffs.

This standard describes test methods for 'substitute tests' performed with volatile test media, iso-octane and 95 % v/v aqueous ethanol, for the determination of overall migration from plastics intended to come into contact with fatty foodstuffs at all temperatures and for any period of time.

© British Standards Institution 2002

Bibliography

[1] Commission of the European Communities, Council Directive of 21 December 1988 on the approximation of the laws of the Member States relating to materials and articles intended to come into contact with foodstuff (89/109/EEC), Official Journal of the European Communities, 11 February 1989, no. L 40, p 3 8.

[2] Commission of the European Communities, Commission Directive of 23 February 1990 relating to plastics materials and articles intended to come into contact with foodstuffs (90/128/EEC), Official Journal of the European Communities, 13 December 1990, no. L 349, p26. Corrigendum of the previous publication, Official Journal of the European Communities, 21 March 1990, no. L 75. p 19.

[3] Commission of the European Communities, Council Directive of 18 October 1982 laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs (82/711/EEC), Official Journal of the European Communities, 23 October 1982, no. L 297, p 26.

[4] Commission of the European Communities, Commission Directive of 15 March 1993 amending Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs (93/8/EEC), Official Journal of the European Communities, 14 April 1993, no. L 90, p 22.

[5] Commission of the European Communities, Commission Directive 97/48/EC of 29 July 1997 amending Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs , Official Journal of the European Communities, 12 August 1997, no. L 222, p 10.

[6] Commission of the European Communities, Council Directive of 19 December 1985 laying down the list of simulants to be used for testing migration of constituents of plastics materials and articles intended to come into contact with foodstuffs (85/572/EEC), Official Journal of the European Communities, 31 December 1985, no. L 372, p 14.

Copyright British Standards Institution

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: +44 (0)20 8996 9000. Fax: +44 (0)20 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: +44 (0)20 8996 9001. Fax: +44 (0)20 8996 7001. Email: orders@bsi-global.com. Standards are also available from the BSI website at <http://www.bsi-global.com>.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: +44 (0)20 8996 7111. Fax: +44 (0)20 8996 7048. Email: info@bsi-global.com.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: +44 (0)20 8996 7002. Fax: +44 (0)20 8996 7001. Email: membership@bsi-global.com.

Information regarding online access to British Standards via British Standards Online can be found at <http://www.bsi-global.com/bsonline>.

Further information about BSI is available on the BSI website at <http://www.bsi-global.com>.

Copyright

Copyright subsists in all BSI publications. BSI also holds the copyright, in the UK, of the publications of the international standardization bodies. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI.

This does not preclude the free use, in the course of implementing the standard, of necessary details such as symbols, and size, type or grade designations. If these details are to be used for any other purpose than implementation then the prior written permission of BSI must be obtained.

Details and advice can be obtained from the Copyright & Licensing Manager. Tel: +44 (0)20 8996 7070. Fax: +44 (0)20 8996 7553. Email: copyright@bsi-global.com.

BSI
389 Chiswick High Road
London
W4 4AL