

Materials and articles in contact with foodstuffs — Plastics —

Part 3: Test methods for overall migration into aqueous food simulants by total immersion

The European Standard EN 1186-3:2002 has the status of a
British Standard

ICS 67.250

National foreword

This British Standard is the official English language version of EN 1186-3:2002. It supersedes DD ENV 1186-3:1994 which is withdrawn.

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

- aid enquirers to understand the text;
- present to the responsible 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 Standards Catalogue under the section entitled “International Standards Correspondence Index”, or by using the “Find” facility of the BSI Standards Electronic Catalogue.

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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 21 May 2002

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

Matériaux et objets en contact avec les denrées
alimentaires - Matière plastique - Partie 3: Méthodes
d'essai pour la migration globale dans l'huile d'olive par
immersion totale

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln
- Kunststoffe - Teil 3: Prüfverfahren für die
Gesamtmigration in wässrige Prüflebensmittel durch
völliges Eintauchen

This European Standard was approved by CEN on 4 January 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.

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Foreword

This document EN 1186-3:2002 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 October 2002, and conflicting national standards shall be withdrawn at the latest by October 2002.

This document supersedes ENV 1186-3:1994.

This European Standard is 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 EC Directive(s).

For relationship with EC 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 1186-3 should be read in conjunction with EN 1186-1.

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 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 13	Test methods for overall migration at high 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

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 Part of this European Standard specifies test methods for the determination of the overall migration into aqueous based food simulants from plastics which are intended to come into contact with foodstuffs, by total immersion of test specimens in a selected food simulant at test temperatures up to reflux for selected test times.

This method is most suitable for plastics in the form of films and sheets, but can be applied to a wide range of articles or containers from which test pieces of suitable size can be cut.

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

EN ISO 8442-2:1997, *Materials and articles in contact with foodstuffs – Cutlery and table holloware – Part 2: Requirements for stainless steel and silver-plated cutlery (ISO 8442-2:1997)*.

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

3 Method A - overall migration into aqueous food simulants by total immersion in an oven or incubator or refrigerator

3.1 Principle

The overall migration of non-volatile substances from a sample of the plastics is determined as the mass of non-volatile residue after evaporation of the food simulant following immersion.

The selection of the conditions of test and the food simulant(s) shall be determined by the conditions of use; see clauses 4, 5 and 6 of EN 1186-1:2002.

Test specimens of approximately 1 dm², see clause 8 of EN 1186-1:2002, are immersed in the food simulant for the exposure time at temperatures up to and including the temperature of reflux. At the end of the test period, each test specimen is removed from the food simulant. The food simulant from each test specimen 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

NOTE For details of preparation and quality of these reagents, see clause 4 of EN 1186-1:2002.

3.2.1 Distilled water or water of equivalent quality (simulant A)

3.2.2 Acetic acid 3 % (w/v) in aqueous solution (simulant B)

3.2.3 Ethanol 10 % (v/v) in aqueous solution (simulant C)

3.2.4 Alcoholic simulants for liquids or beverages of an alcoholic strength exceeding 10 % (v/v).

NOTE In the case of materials and articles intended to come into contact with liquids or beverages of an alcoholic strength exceeding 10 % (v/v) the test is carried out with aqueous solutions of ethanol of a similar strength.

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) × (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 dilute nitric acid has been found to be suitable. For the aqueous acetic acid food simulant, use supports constructed out of glass, as there is a tendency for the acetic acid to corrode stainless steel supports, particularly if the joints are silver soldered.

NOTE 1 However stainless steel supports can be used for acetic acid if it can be demonstrated that when immersed on their own in simulant, for the test period, at the test temperature, the residue after evaporating the simulant to dryness and drying in an oven or incubator or refrigerator to constant mass at 105 °C to 110 °C is less than 5mg/l, see 4.1 of EN 1186-1:2002.

NOTE 2 The method has been written for the supports shown in Figure C.1 of EN 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 holding and keeping the test pieces apart and at the same time ensuring complete contact with the simulant. For rigid samples, supports with a single cross arm may 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 to be used with the acetic acid food simulant, 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, for retaining the food simulant and test specimens. Tubes with an internal diameter of approximately 35 mm and length of between 100 mm and 200 mm, excluding the ground neck (see 7.2 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 7.2 of EN 1186-1:2002).

3.3.12 Thermostatically controlled oven or incubator or refrigerator capable of maintaining the set temperature within the tolerances specified in Table B.2 of EN 1186-1:2002.

3.3.13 Dishes, stainless steel, nickel, platinum, platinum alloy, gold 50 mm to 90 mm diameter and maximum mass 100 g, for evaporation of food simulants and weighing of residues. Glass, glass ceramic or ceramic dishes may be used provided that the surface characteristics are such that the masses of the dishes after evaporation of any specified food simulants followed by conditioning in the desiccator used achieves a constancy of ± 0,5 mg. Stainless steel and nickel dishes are suitable only for distilled water and ethanol solutions. Glass, glass ceramic, glazed ceramic, platinum or, platinum alloy or gold dishes are suitable for all three simulants.

3.3.14 Steam bath, hot plate, distillation apparatus or rotary evaporator for evaporation of food simulant 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.

3.4 Preparation of test specimens

3.4.1 General

It is essential that test specimens are clean and free from surface contamination (many plastics can readily attract dust due to static charges). Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use see 8.1 of EN 1186-1:2002. Minimise handling of the samples and where necessary, wear cotton gloves.

To ensure that test pieces are well separated and that the surfaces are freely exposed to the food simulant during the period of the test, for thin films, insert a piece of fine stainless steel gauze, or glass rods with the acetic acid simulant, between the test pieces or for thick samples not placed on the supports, insert glass rods between the test pieces after immersion in the food simulant. Where specimen supports are used, label the supports with a tag bearing the test specimen identification.

When preparing test specimens measure the surface area according to 8.3 of EN 1186-1:2002.

3.4.2 Number of test specimens

Three test specimens are required for samples, in the form of thin films, sheets, cut sections from containers or similar articles. Five test specimens, similar dimensionally one to another, are required for samples of articles of irregular shape. These test specimens are utilized as follows:

- a) three test specimens for the migration test;
- b) two test specimens for determination of the surface area, in the case of samples of irregular shape (3.4.5).

3.4.3 Films and sheets

Lay the sample on the cutting slab (3.3.1) and cut the test specimens of 1 dm² (see 9.3 of EN 1186-1:2002), using the 100 mm × 100 mm template (3.3.4). Check, using the rule (3.3.6), that the dimensions of the specimen are within the specified tolerance (± 1 mm).

Cut each test specimen into four test pieces 25 mm × 100 mm using the rule (3.3.5). Assemble one test specimen onto the support (3.3.8) by piercing suitable holes in the test pieces and placing two test pieces on each side of the cross arms of the support. Repeat this procedure for all remaining test specimens.

3.4.4 Containers and other articles

Cut sections from the walls of the container or article to give test specimens each of area approximately 1 dm². For articles with individual areas less than 1 dm², use a number of articles to provide each test specimen. Measure the dimensions of each test specimen to the nearest 1 mm, using the rule. Calculate only the surface area of the sample which is intended to come into contact with foodstuffs, see 8.3 of EN 1186-1:2002.

Calculate the area of each test specimen to the nearest 0,01 dm² and record. If necessary, cut each test specimen into smaller pieces to enable them to fit into the glass tubes (3.3.10). The test specimens or pieces are placed on the specimen supports if these are appropriate or, if the test specimens or pieces are sufficiently rigid, they can be tested unsupported.

3.4.5 Articles of irregular shape

Select representative portions of the article, or multiples of the article for small articles, to give five dimensionally similar test specimens each with a known total surface area of at least 1 dm². Measure only the surface area intended to come into contact with foodstuffs of two of these test specimens to the nearest 0,05 dm² using the Schlegel Method, as described in EN ISO 8442-2:1997, annex B, or any other suitable method. Record the surface area of each test specimen.

3.5 Procedure

3.5.1 Exposure to food simulant

Take three of the glass tubes, for the test specimens and a further two to provide blanks, measure by measuring cylinder 100 ml \pm 2 ml of the food simulant into each tube and stopper the tube. Insert a thermometer or thermocouple, if applicable see NOTE 2, in one of the test specimen tubes, and 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 simulant has attained the test temperature.

Place a test specimen into each of the three tubes containing 100 ml of simulant, re-insert the thermometer or thermocouple and stopper the tubes. Mark the tubes for identification. Ensure that the test specimens are totally immersed in the simulant; if they are not then add either glass beads or rods to raise the level of the simulant 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 simulant.

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 period of time after the temperature of the simulant has reached a temperature within the permitted tolerance for the test temperature, see Tables B.1 and B.2 of EN 1186-1:2002 for permitted tolerances on test times and temperature.

NOTE 1 Annex B of EN 1186-1:2002 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 tubes from the oven or incubator or refrigerator and check the level of simulant 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.

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

NOTE 2 For exposure times of 24 h or more it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

3.5.2 Determination of migrating substances

3.5.2.1 Preparation of dishes

Take five dishes (3.3.13), marked for identification, place the dishes in an oven maintained at 105 °C to 110 °C, for a period of 30 min \pm 5 min, to dry.

Remove the dishes from the oven, place in a desiccator (3.3.15) and allow to cool to ambient temperature. Weigh and record the individual masses of each dish.

Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until individual consecutive masses differ by not more than 0,5 mg, record their final masses.

3.5.2.2 Evaporation method

Take the tubes containing the simulant and pour 40 ml to 50 ml from each into separate dishes. By means of a steam bath, hot plate or other form of heating (3.3.14) evaporate to a low volume, taking care to avoid loss, in particular, by sputtering or overheating of the residues.

NOTE 1 The evaporation of acetic acid and ethanol should be carried out in a fume cupboard.

When most of the simulant has evaporated, pour the remaining simulant from each of the tubes into the respective dishes and continue the evaporation. Wash out each of the tubes, including the blank tubes with two lots of 10 ml \pm 1 ml of unused simulant and pour these washings into the respective dishes. Continue the evaporation.

NOTE 2 A stream of nitrogen can be used to facilitate evaporation.

When the simulant has almost completely evaporated, place the dish in an oven maintained at 105 °C to 110 °C, for a period of 30 min \pm 5 min, to complete the evaporation and dry the residue.

Remove the dishes from the oven, place in a desiccator (3.3.15) and allow to cool to ambient temperature. Weigh and record the individual masses of a dish and residue.

Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until individual consecutive masses differ by not more than 0,5 mg.

Determine the mass of the residue by subtracting the original stable mass of the dish (3.5.2.1) from the stable mass of the dish and residue.

3.5.2.3 Distillation method

Transfer the simulants to individual round bottom flasks (250 ml are suitable). Wash out each of the tubes, including the blank tubes, with two lots of 10 ml \pm 1 ml of unused simulant, add these rinses to the respective flasks. Place the flasks in an electric heating mantle and connect to a side arm distillation arrangement or rotary evaporator. Distil off the simulants until approximately 30 ml to 50 ml remains in each flask. Transfer the remaining simulants from each flask to individual evaporating dishes (3.3.13). Rinse each flask with two lots of 10 ml \pm 1 ml of fresh simulant and add the rinses to the appropriate dishes. Continue the evaporation of the simulant by means of a steam bath, hot plate or other form of heating, proceeding as in 3.5.2.2.

NOTE The evaporation of acetic acid and ethanol should be carried out in a fume cupboard.

3.6 Expression of results

3.6.1 Method of calculation

Express the overall migration as milligrams of residue per square decimetre of the surface of the sample which is intended to come into contact with foodstuffs, calculated for each test specimen using the following formula:

$$M = \frac{(m_a - m_b) \times 1000}{S}$$

where

M is the overall migration into the simulant, in milligrams per square decimetre of surface area of sample intended to come into contact with foodstuffs;

m_a is the mass of the residue from the test specimen after evaporation of the simulant in which it had been immersed, in grams;

m_b is the mass of residue from the blank simulant, in grams;

S is the surface area of the test specimen intended to come into contact with foodstuff, in square decimetres, see 6.1 and 9 of EN 1186-1:2002.

Calculate the result for each test specimen to the nearest 0,1 mg/dm² and the mean of the individual test results, to the nearest 0,1 mg/dm².

See 11.3 of EN 1186-1:2002 for the directions to determine whether the results are valid.

3.6.2 Precision

See annex A.

3.7 Test report

The test report shall include the following (see clause 11 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 simulants;
- 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 Method B - overall migration into aqueous food simulants by total immersion at reflux

4.1 Principle

The overall migration of non-volatile substances from a sample of the plastics is determined as the mass of non-volatile residue after evaporation of the food simulant following immersion.

The selection of the conditions of test and the food simulant(s) shall be determined by the conditions of use, see clauses 3, 4 and 5 of EN 1186-1:2002.

Test specimens of approximately 1 dm², see clause 8 of EN 1186-1:2002, are immersed in the food simulant for the exposure time at reflux then removed. The food simulant is evaporated to dryness, the mass of the non-volatile residue is determined and expressed as milligrams per square decimetre of surface area of the specimen.

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

4.2 Reagents

The reagents shall be in accordance with 3.2

4.3 Apparatus

The apparatus shall be in accordance with 3.3, but include:

- a) flasks, 250 ml, suitable for attaching to reflux condensers;
- b) condensers to fit the flasks;
- c) heating mantle for maintaining the simulants at reflux temperature during the exposure;
- d) glass filter of porosity G1

and the following are not required:

3.3.8 Specimen supports;

3.3.9 Pieces of stainless steel gauze and lengths of glass rod;

3.3.10 Glass tubes.

4.4 Preparation of test specimens

4.4.1 General

It is essential that test specimens are clean and free from surface contamination (many plastics can readily attract dust due to static charges). Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use, see 8.1 of EN 1186-1:2002. Minimise handling of the samples and where necessary, wear cotton gloves.

When preparing test specimens calculate the surface area according to 8.3 of EN 1186-1:2002.

4.4.2 Number of test specimens

Three test specimens are required for samples, in the form of thin films, sheet, cut sections from containers or similar articles. Five test specimens, similar dimensionally one to another, are required for samples of articles of irregular shape. These test specimens are utilized as follows:

- a) three test specimens for the migration test;
- b) two test specimens for determination of the surface area, in the case of samples of irregular shape (4.4.5).

4.4.3 Films and sheet

Lay the sample on the cutting slab (3.3.1) and cut the test specimens of 1 dm² (see 8.3 of EN 1186-1:2002), using the 100 mm × 100 mm template (3.3.4). Check, using the rule (3.3.6), that the dimensions of the specimen are within the specified tolerance (± 1 mm). Cut each test specimen into sixteen test pieces 25 mm × 25 mm using the rule. Repeat this procedure for all remaining test specimens.

4.4.4 Containers and other articles

Cut sections from the walls of the container or article to give test specimens each of area approximately 1 dm². For articles with individual areas less than 1 dm², use a number of articles to provide each test specimen. Measure the dimensions of each test specimen to the nearest 1 mm, using the rule. Calculate for each test specimen only the surface area that is intended to come into contact with foodstuffs, see 8.3 of EN 1186-1:2002, to the nearest 0,01 dm² and record.

Cut each test specimen into test pieces 25 mm × 25 mm.

4.4.5 Articles of irregular shape

Select representative portions of the article, or multiples of the article for small articles, to give five dimensionally similar test specimens each with a known total surface area of at least 1 dm². Measure only the surface area intended to come into contact with foodstuffs of two of these test specimens to the nearest 0,05 dm² using the Schlegel Method, as described in EN ISO 8442-2:1997, annex B, or any other suitable method.

Record the surface area of each test specimen.

4.5 Procedure

4.5.1 Exposure to food simulants

Take three flasks (4.3 a) for the test specimens and a further two to provide blanks, measure by measuring cylinder 100 ml \pm 2 ml of food simulant into each flask.

Place the flasks in the heating mantle, connect the condensers.

Turn on the water supply to the condensers.

Switch on the heating mantle and heat the simulant in each of the flasks to boiling. Turn off the heating, allow the flasks to cool for 2 min to 3 min, remove the condensers from three flasks containing 100 ml of simulant and place a test specimen in each flask. Ensure that the test specimens are totally immersed in the food simulant.

WARNING: Hot fumes can emit from the flasks when the lids are removed.

Replace the condensers and switch on the heating mantles, and heat so that reflux is achieved within 5 min.

Observe the food simulant in the flask, following the onset of reflux, leave for the test time, taking into account the tolerances in Table B.1 of EN 1186-1:2002. Turn off the heating mantle, turn off the water to the condenser and remove the flask from the mantle.

NOTE Annex B of EN 1186-1:2002 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.

To separate the simulant from the test specimens, pour off the hot simulant through a glass filter (4.3 d), collecting the filtrate in a clean container. Rinse each of the flasks and the test specimens in the flasks with two portions of $10\text{ ml} \pm 1\text{ ml}$ of unused simulant and pour these washings through the filter.

WARNING: Danger hot flasks and contents.

4.5.2 Determination of migrating substances

The determination of the migration substances shall be in accordance with 3.5, except that the food simulant is contained in flasks.

4.6 Expression of results

The expression of results shall be in accordance with 3.6.

4.7 Test report

The test report shall be in accordance with 3.7

Annex A (informative)

Precision data

The precision data were determined for a polyamide sample under the test conditions of 24 h at 40 °C with simulants A, B and C.

The precision data for simulant A were determined from the BSI/DTI trial conducted in 1991 (Pira Report No.SP91/2- January 1992) involving 13 laboratories and one sample.

The precision data for simulant B were determined from the BSI/DTI trial conducted in 1991 (Pira Report No.SP91/2- January 1992) involving 10 laboratories and one sample.

The precision data for simulant C were determined from the BSI/DTI trial conducted in 1991 (Pira Report No.SP91/2- January 1992) involving 13 laboratories and one sample.

Table A.1 — Precision data

Level	Repeatability (<i>r</i>)	Reproducibility (<i>R</i>)
Simulant A 6,9 mg/dm ²	1,3 mg/dm ²	2,6 mg/dm ²
Simulant B 10,7 mg/dm ²	1,1 mg/dm ²	2,3 mg/dm ²
Simulant C 11,9 mg/dm ²	1,1 mg/dm ²	2,9 mg/dm ²

The difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval can exceed the repeatability value *r* on average not more than once in 20 cases in the normal and correct operation of the method.

Single results on identical test material reported by two laboratories can differ by more than the reproducibility value *R* on average not more than once in 20 cases in the normal and correct operation of the method.

Annex ZA (informative)

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

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association (EFTA).

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'. 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 contains the test methods for the measurement of overall migration from plastics materials to food simulants, or test media, using conventional contact test conditions of time and temperature, to determine compliance with the legislative overall migration limit specified in article 2 of European Commission Directive 90/128/EEC.

These test methods may also be used for the verification of compliance with the specific migration limits provided for in paragraph 1 of Commission Directive 90/128/EEC, if it can be established that compliance with the overall migration limit laid down in Article 2 of Commission Directive 90/128/EEC implies that the specific migration limits are not exceeded.

Commission Directive also specifies that the migration tests using rectified olive oil or substitutes shall not be carried out to check compliance with the overall migration limit in cases where there is conclusive proof that the specified analytical method is inadequate from the technical standpoint.

In any such case, substances exempt from specific migration limits or other restrictions in the list provided in Annex II of Commission Directive 90/128/EEC, a generic specific migration limit of 60 mg/kg or 10 mg/dm², according to the case, is applied. However, Commission Directive 90/128/EEC requires that the sum of all specific migrations determined shall not exceed the overall migration limit.

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 38.
- [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, p 26. Corrigendum of the previous publication, Official Journal of the European Communities, 21 March 1990, no. 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 of 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.

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