BRITISH STANDARD

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Materials and articles in contact with foodstuffs — Plastics —

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

The European Standard EN 1186-15:2002 has the status of a British Standard

ICS 67.250

National foreword

This British Standard is the official English language version of EN 1186-15:2002.

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

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Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 15: Méthodes d'essai alternatives pour la migration dans les simulants alimentaires gras par extraction rapide dans l'iso-octane et/ou l'éthanol à 95 %

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 15: Alternative Prüfverfahren zur Migration in fettige Prüflebensmittel durch Schnellextraktion in Iso-Octan und/oder 95%iges 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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Contents

Foreword

This document EN 1186-15:2002 has been prepared by Technical Committee CEN/TC 194 “Utensils in contact with food”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2003, and conflicting national standards shall be withdrawn at the latest by March 2003.

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

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

At the time of preparation and publication of this standard the European Union legislation relating to plastics materials and articles intended to come into contact with foodstuffs is incomplete. Further Directives and amendments to existing Directives are expected which could change the legislative requirements which this standard supports. It is therefore strongly recommended that users of this standard refer to the latest relevant published Directive(s) before commencement of any of the test or tests described in this standard.

EN 1186-15 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 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 method 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

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 two alternative test methods, in the sense of an extraction test with a 'more severe' test character, for the assessment of the overall migration into fatty food simulants.

Method A is based on the determination of the extraction of migrateable substances from plastics which are intended to come into contact with foodstuffs, by total immersion in non-polar, iso-octane, and/or polar, ethanol, solvents depending on the polarity of the packaging material. According to results obtained by this method (see [1], [2], [3], [4], [5]) and taking physio-chemical considerations into account, the obtained extraction efficiency has, generally, been found to be equivalent to or higher than overall migration results obtained under the test conditions, 10 days at 40 °C, 2 h at 70 °C, 1 h at 100 °C, 30 min at 121 °C and 30 min at 130 °C.

To ensure as complete as possible extraction of the potential migrants, a strong interaction, e.g. swelling, of the sample by the extraction solvent is necessary. For this purpose, iso-octane is used as an extraction solvent for plastics materials and articles containing non polar food contact layers, such as polyolefins. For test samples made from polar food contact plastics such as polyamide and polyethylene terephthalate, 95 % (v/v) aqueous ethanol is used. For polystyrenes, plasticized polyvinyl chloride and other polymers where the identification or polarity of the polymer is not clear, two parallel extraction tests should be conducted using both of the proposed extraction solvents and taking the higher value obtained as the relevant result.

NOTE 1 In case of multilayer structures such as plastics laminates and co-extruded plastics, the nature of the food contact layer determines the selection of the extraction solvent(s).

This test method should only be applied to flexible packagings which are less than 300 µm in thickness. When the result does not exceed the allowed overall migration limit then the material can be considered to be in compliance with EC regulations. If the test result exceeds the allowed overall migration limit the following options may be applied chronologically with respect to further migration testing:

1) single-sided extraction test using a cell, if technically feasible (see clause 4 Method B of this standard);

2) conventional migration test using olive oil or other fatty food simulants;

NOTE 2 The overall migration limit is specified in Commission Directive 90/128/EEC [7] and the conditions of test in Council Directive 82/711/EEC [8] and its subsequent amendments, [9], [10].

Method B is applicable in those cases where the total immersion test, EN 1186-15 Method A, yields total extraction values that exceed the overall migration or may be technically unsuitable, i.e. in the case of multilayer structures, such as plastics laminates and co-extruded films. This test method should primarily only be applied to flexible packagings with a physical barrier layer (for instance of aluminium or other material to prevent penetrative loss of extraction solvent) and which have a thinner food contact layer than 300 µm. If the result does not exceed the allowed overall migration limit then the material can be considered to be in compliance with EC regulations. If the test result exceeds the allowed overall migration limit then the following option may be applied with respect to further migration testing:

- conventional migration test using olive oil or other fatty food simulants.

NOTE 3 Methods A and B are not applicable to test materials intended for applications over 130 °C.

NOTE 4 Test materials intended for applications over 70 °C should be checked for their physical suitability at the intended time and temperature of use.

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*.

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

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

3 Method A
Alternative test method to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol by total immersion

3.1 Principle

The migrateable substances extracted from a sample of the plastics is determined as the mass of non-volatile residue after evaporation of the solvent following immersion. Test specimens of at least 1 dm² (single side considered) are immersed in the extraction solvent for 24 h at 40 °C or 50 °C and then removed. The extraction solvent 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 test specimen. The measured value is compared to the EC-official overall migration limit and taking the analytical tolerance of this method (± 1 mg/dm²) into account.

3.2 Reagents

NOTE For details of preparation and quality of these reagents, see clause 5 of EN 1186-1:2002.

3.2.1 Ethanol 95 % (v/v) in aqueous solution.

3.2.2 Iso-octane (2,2,4-trimethylpentane)

NOTE The extraction solvents given in 3.2.1 and 3.2.2 are selected according to the nature of the polymer test sample as given by Table 1, see 3.5.1.

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

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

3.3.7 Extraction containers; glass weighing jars with ground joints, tall form, of a capacity of approx. 60 ml.

3.3.8 Thermostatically controlled oven or incubator capable of maintaining a temperature within the range of + 40 °C to + 50 °C and meeting temperature tolerance values within those specified for the test temperature, see annex B of EN 1186-1:2002.

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

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

3.3.10 Steam bath, hot plate, distillation apparatus or rotary evaporator.

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

3.3.12 Measuring cylinder, 50 ml capacity, conforming to the minimum requirements of ISO 4788.

3.3.13 Round-bottom flask, 250 ml capacity [for distillation method (see 3.5.3.3) only].

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 9.1 of EN 1186-1:2002. Minimize 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 extractant during the period of the test, insert a piece of fine stainless steel gauze between the cut test pieces.

3.4.2 Number of test specimens

Three replicate test specimens are required.

3.4.3 Cutting and preparation of specimen

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 (3.3.5), that the dimensions of the specimen are within the specified tolerance (1 mm). Fold the test specimens into a fan-like shape or cut into strips approximately 2 cm wide and 5 cm long. Place in the extraction containers (3.3.7).

3.5 Procedure

3.5.1 Selection of extraction solvent

Select the appropriate extraction solvent(s) (see 3.2.1 and 3.2.2) according to the nature of the polymer test sample as given in Table 1.

Table 1 — Use of extraction solvents and test conditions – Method A

Polymer type of food contact layer	Extraction solvent to be applied	Extraction conditions to be applied
Polyolefines and copolymers	iso-octane	24 h at 40 °C
Polyamides	95 % ethanol	24 h at 40 °C
Polystyrene	iso-octane and 95 % ethanol	24 h at 40 °C
Polyethylene terephthalate	95 % ethanol	24 h at 50 °C
Polyvinyl chloride (plasticized)	iso-octane and 95 % ethanol	24 h at 40 °C
Polyvinyl chloride (rigid)	95 % ethanol	24 h at 50 °C

3.5.2 Exposure to solvent

Take three extraction containers or jars (3.3.7), measure by measuring cylinder (3.3.12) 50 ml of the solvent into each of these jars and immerse the test specimens in the solvent. Ensure that the test specimens are totally immersed in the solvent. If the evaporation method is to be used (3.5.3.2) measure into a further two jars by measuring cylinder the same amount of solvent, plus 10 ml ± 2 ml, to provide blanks. If the distillation method (3.5.3.3) is to be used measure into those further two jars by measuring cylinder the same amount of solvent in contact with the test specimens to provide blanks. Stopper the jars. Mark the jars for identification. Mark the liquid level on the outside of each jar with a suitable marker.

The extraction conditions are to be selected from Table 1 according to the nature of the polymer test samples.

Place the five jars in the thermostatically controlled oven or incubator (3.3.8), set at the test temperature and observe the temperature, leave the jars for the test period of 24 h after the air bath of the thermostatically controlled oven or incubator has reached the set temperature and taking the permitted time and temperature tolerances into account (see annex B of EN 1186-1:2002). Take the jars from the oven or incubator and allow them to cool down to room temperature. Check the level of solvent in each. If this has fallen to more than 5 mm below the mark, or has exposed any part of the test pieces, repeat the test using fresh test specimens. If the level of solvent in a jar is less than 5 mm below the mark, remove the test specimen from the jar, and allow the solvent adhering to the test specimen and support to drain back into the jar. Recover at least 90 % of the original volume of solvent, including the blanks, or repeat the test.

WARNING Both iso-octane and ethanol are volatile flammable solvents. Care should be taken to avoid any loss of solvent into the interior of the thermostatting device. Place the jars, if possible, in a drip container serving as a possible solvent reservoir in case of leakage. Do not allow the temperature to exceed 60 °C.

3.5.3 Determination of extracted substances

3.5.3.1 Preparation of dishes

Take five dishes (3.3.9), marked for identification, place the dishes in an oven maintained at 105 °C to 110 °C, for a period of 30 min ± 5 min, to dry. Remove the dishes from the oven, place in a desiccator 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.

3.5.3.2 Evaporation method

For each jar, including the two blank jars,containing the solvent, pour 20 ml to 25 ml into a prepared dish. By means of a steam bath, hot plate or other form of heating evaporate to a low volume (3.3.10), taking care to avoid loss of residue, in particular, by sputtering or overheating.

NOTE 1 The evaporation should be carried out in a fume cupboard.

When most of the solvent has evaporated, pour the remaining solvent from each of the jars into the respective dishes and continue the evaporation. Rinse each of the jars which had contained test specimens with two lots of 5 ml ± 1 ml of fresh solvent 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 solvent has almost completely evaporated, place the dish in an oven maintained at 105 °C to 110 °C, for a period of 30 min ± 5 min, to complete the evaporation and dry the residue. Remove the dishes from the oven, place in a desiccator (3.3.11) and allow to cool to ambient temperature. Weigh and record the individual masses of each 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 mass of the dish from the final mass of the dish and residue.

3.5.3.3 Distillation method

For each jar, transfer the contents to a round bottom flask (3.3.13). Rinse each jar twice, including the blank jars, with 20 ml ± 2 ml of fresh solvent, 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 solvents until approximately 15 ml to 25 ml remains in the flask. Transfer the remaining solvents to an evaporating dish. Rinse the flask with 10 ml ± 1 ml of fresh solvent and add the rinses to the appropriate dishes. Continue the evaporation of the solvent by means of a steam bath, hot plate or other form of heating, proceeding as in 3.5.3.2.

NOTE The evaporation should be carried out in a fume cupboard.

3.6 Expression of results

3.6.1 Method of calculation

Express the extraction 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:

$$M = \frac{(m_a - m_b)}{S} \qquad (1)$$

where

M is the total mass extracted into the solvent, 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 solvent in which it had been immersed, in milligrams;

m_b is the mass of residue from the solvent from blank only, in milligrams;

S is the surface area of the test specimen, in square decimetres, see clause 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.

In cases where both extraction solvents had to be applied (see Table 1), the higher value measured shall be the relevant one.

3.6.2 Precision

The repeatability (r) and the reproducibility (R) values are to be determined from collaborative trial results.

3.7 Test report

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

a) reference to this European Standard, to the Part and the method 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;

c) conditions of time and temperature of exposure to extractants;

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
Alternative test method to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol in the single side mode by cell

4.1 Principle

The total migrateable substances extracted from a sample of the plastics is determined as the mass of non-volatile residue after evaporation of the solvent following immersion.

One surface of the test specimen of at least 1 dm² (single side considered) is exposed in a cell to the extraction solvent for 24 h at 40 °C or 50 °C and then removed. The extraction solvent 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 test specimen. The measured value is compared to the EC-official overall migration limit and taking the analytical tolerance of this method (± 1 mg/dm²) into account.

4.2 Reagents

NOTE For details of preparation and quality of these reagents, see clause 5 of EN 1186-1:2002.

4.2.1 Ethanol 95 % (v/v) in aqueous solution.

4.2.2 Iso-octane (2,2,4-trimethylpentane)

The extraction solvents given in 4.2.1 and 4.2.2 are to be selected according to the nature of the polymer test sample as given in Table 2, see 4.5.1.

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

4.3.5 Cell, type A as shown in Figure C.3 of EN 1186-1:2002, either the aluminium (anodized) cells or the cells with the stainless steel (316 grade) lids and rings, are suitable for the extraction solvents or equivalent cells.

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

4.3.6 Glass tubes, ground neck, and stoppers, for retaining the food simulant.

4.3.7 Pipettes, conforming to the minimum requirements of ISO 648, 50 ml and 100 ml.

4.3.8 Thermostatically controlled oven or incubator capable of maintaining a temperature within the range of + 40 °C to + 50 °C and meeting temperature tolerance values within those specified for the test temperature, see annex B of EN 1186-1:2002.

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

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

4.3.10 Steam bath, hot plate, distillation apparatus or rotary evaporator.

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

4.3.12 Measuring cylinder, 50 ml capacity, conforming to the minimum requirements of ISO 4788.

4.3.13 Round-bottom flask, 250 ml capacity [for distillation method (see 4.5.3.3) only].

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 9.1 of EN 1186-1:2002. Minimize handling of the samples and where necessary, wear cotton gloves.

4.4.2 Number of test specimens

Three replicate test specimens are required.

4.4.3 Cutting test specimens

Lay the sample on the cutting slab (4.3.1) with the surface to be in contact with the food simulant uppermost. Take the ring from the cell (4.3.5) and place on the surface of the sample. Cut out the test specimen by cutting round the outer edge of the ring, using the cutting implement (4.3.3).

4.5 Procedure

4.5.1 Selection of extraction solvent

Select the appropriate extraction solvent(s) (see 4.2.1 and 4.2.2) according to the nature of the polymer test sample as given in Table 2.

Table 2 — Use of extraction solvents and test conditions- Method B

Polymer type of food contact layer	Extraction solvent to be applied	Extraction conditions to be applied
Polyolefines and copolymers	iso-octane	24 h at 40 °C
Polyamides	95 % ethanol	24 h at 40 °C
Polystyrene	iso-octane and 95 % ethanol	24 h at 40 °C
Polyethylene terephthalate	95 % ethanol	24 h at 50 °C
Polyvinyl chloride (plasticized)	iso-octane and 95 % ethanol	24 h at 40 °C
Polyvinyl chloride (rigid)	95% ethanol	24 h at 50 °C

4.5.2 Exposure to solvent

NOTE The cells type A are constructed with a rubber mat in the base plate. When using the cells it is advised that a disc of aluminium foil is placed on the mat before inserting the test specimen. The use of this disc prevents any substance from the mat influencing the extraction result. For details of equivalent cells see clause 8 of EN 1186-1:2002.

Take three cells (4.3.5), mark these for identification purposes. Dismantle and place on the base of each cell one test specimens. Reassemble the cells, ensuring that the clamping screw wheel is well tightened down.

Measure by measuring cylinder an appropriate amount of the solvent into each cell through the filler hole and replace the filler plug. The ratio between the area in square decimetres exposed to the amount of food simulant in millilitres shall be a maximum 1/50. If the evaporation method is to be used (4.5.3.2) measure into two tubes (4.3.6) by measuring cylinder the same amount of solvent as measured into each cell, plus 10 ml ± 2 ml, to provide blanks. If the distillation method (4.5.3.3) is to be used measure into those two tubes by measuring cylinder the same amount of solvent in contact with the test specimens to provide blanks. Stopper the tubes. Mark the tubes for identification. Mark the liquid level on the outside of each tube with a suitable marker.

The extraction conditions are to be selected from Table 2 according to the nature of the polymer test samples.

Place the three cells and the two tubes in the thermostatically controlled oven or incubator (4.3.8), set at the test temperature and observe the temperature. Leave the cells and tubes for the test period of 24 h after the air bath of the thermostatically controlled oven or incubator has reached the set temperature and taking the permitted time and temperature tolerances into account (see annex B of EN 1186-1:2002). Take the cells and the two tubes containing the blank simulants from the thermostatically controlled oven or incubator and let the cell and contents cool to room temperature. Transfer by a 50 ml or 100 ml pipette (4.3.7) the solvent from each of the three cells into three tubes, measure amount of simulant in each. Recover at least 90 % of the original volume of solvent or repeat the test with fresh test specimens. Rinse each cell twice with 20 ml ± 2 ml of simulant, add these rinses to the respective tubes.

WARNING Both iso-octane and ethanol are volatile flammable solvents. Care should be taken to avoid any loss of solvent into the interior of the thermostatting device. Place the cells and tubes, if possible, in a drip container serving as a possible solvent reservoir in case of leakage. Do not allow the temperature to exceed 60°C.

4.5.3 Determination of extracted substances

NOTE The methods given in 4.5.3.2 and 4.5.3.3 are alternatives.

4.5.3.1 Preparation of dishes

Take five dishes (4.3.9), marked for identification, place the dishes in an oven maintained at 105 °C to 110 °C, for a period of 30 min ± 5 min, to dry. Remove the dishes from the oven, place in a desiccator 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.

4.5.3.2 Evaporation method

Take the tubes containing the solvent and pour 20 ml to 25 ml from each into separate dishes. By means of a steam bath, hot plate or other form of heating (4.3.10) evaporate to a low volume, taking care to avoid loss, in particular, by sputtering or overheating of the residues.

NOTE 1 The evaporation should be carried out in a fume cupboard.

When most of the solvent has evaporated, pour the remaining solvent from each of the tubes into the respective dishes and continue the evaporation. Wash out each of the tubes which had contained the solvent exposed to the test specimens in the cells with two lots of 5 ml ± 1 ml of unused solvent 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 solvent has almost completely evaporated, place the dish in an oven maintained at 105 °C to 110 °C, for a period of 30 min ± 5 min, to complete the evaporation and dry the residue. Remove the dishes from the oven, place in a desiccator (4.3.11) 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 mass of the dish from the final mass of the dish and residue.

4.5.3.3 Distillation method

Transfer the solvents to individual round bottom flasks (4.3.13). Rinse each tube twice, including the blank tubes, with 20 m ± 2 ml of fresh solvent, 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 solvents until approximately 15 ml to 25 ml remains in the flask. Transfer the remaining solvents to an evaporating dish. Rinse the flask with 10 ml ± 1 ml of fresh solvent and add the rinses to the appropriate dishes. Continue the evaporation of the solvent by means of a steam bath, hot plate or other form of heating, proceeding as in 4.5.3.2.

NOTE The evaporation should be carried out in a fume cupboard.

4.6 Expression of results

4.6.1 Method of calculation

Express the extraction 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:

$$M = \frac{(m_a - m_b)}{S} \qquad (2)$$

where

M is the total mass extract into the solvent, 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 solvent with which it was in contact, in milligrams;

m_b is the mass of residue from the solvent blank only, in milligrams;

S is the surface area of the test specimen, in square decimetres, see clause 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.

4.6.2 In cases where both extraction solvents had to be applied (see Table 2), the higher value measured shall be the relevant one.Precision

The repeatability (r) and the reproducibility (R) values are to be determined from collaborative trial results.

4.7 Test report

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

a) reference to this European Standard, to the Part and the method 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;

c) conditions of time and temperature of exposure to extractants;

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.

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 [6], 90/128/EEC [7], 82/711/EEC [8] and its amendments 93/8/EEC [9] and 97/48/EC [10], and 85/572/EEC [11].

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, [7], 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 [8], and the subsequent amendments (Directives 93/8/EEC [9] and 97/48/EC [10]), 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 [11] has a Table in the annex which contains a non-exhaustive list of foodstuffs and which identifies the simulants to be used in migration tests on those plastics materials and articles intended to come into contact with a particular foodstuff or group of foodstuffs.

This standard specifies alternative test methods, in the sense of an extraction test with a 'more severe' test character, for the assessment of the overall migration into fatty food simulants. The method is based on the determination of the extraction of migrateable substances from plastics which are intended to come into contact with foodstuffs, by total immersion and single surface contact in non-polar, iso-octane, and/or polar, ethanol, solvents depending on the polarity of the packaging material.

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