BRITISH STANDARD

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Materials and articles in contact with foodstuffs — Plastics —

Part 12: Test methods for overall migration at low temperatures

The European Standard EN 1186-12:2002 has the status of a British Standard

ICS 67.250

National foreword

This British Standard is the official English language version of EN 1186-12:2002. It supersedes DD ENV 1186-12:1995 which is withdrawn.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 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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Contents

Foreword

This document EN 1186-12:2002 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 1186-12:1995.

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 1186-12 should be read in conjunction with EN 1186-1, EN 1186-2, EN 1186-4, EN 1186-6 and EN 1186-8.

Further Parts of this standard have been prepared concerned with the determination of overall migration from plastics materials into food simulants.

Their titles are as follows:

EN 1186 Materials and articles in contact with foodstuffs – Plastics -

Part 1	Guide to the selection of conditions and test methods for overall migration
Part 2	Test methods for overall migration into olive oil by total immersion
Part 3	Test methods for overall migration into aqueous food simulants by total immersion
Part 4	Test methods for overall migration into olive oil by cell
Part 5	Test methods for overall migration into aqueous food simulants by cell
Part 6	Test methods for overall migration into olive oil using a pouch
Part 7	Test methods for overall migration into aqueous food simulants using a pouch
Part 8	Test methods for overall migration into olive oil by article filling
Part 9	Test methods for overall migration into aqueous food simulants by article filling
Part 10	Test methods for overall migration into olive oil (modified method for use in cases where incomplete extraction of dewaxed sunflower oil occurs)

Part 11	Test methods for overall migration into mixtures of ^{14}C-labelled synthetic triglyceride
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

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 fatty food simulants from plastics materials and articles, by total immersion of test specimens in a fatty food simulant at temperatures from 5 °C, up to and including 20 °C, for selected 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 a suitable size can be cut.

The fatty food simulant used in these test methods is dewaxed sunflower oil since, unlike olive oil, remains liquid at the lower test temperature.

The test method described is applicable to most types of plastics, although there are some plastics for which it is known not to be applicable.

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 food – Plastics – Part 1: Guide to the selection of conditions and test methods for overall migration.*

EN 1186-2:2002, *Materials and articles in contact with food – Plastics – Part 2: Test methods for overall migration into olive oil by total immersion.*

EN 1186-4:2002, *Materials and articles in contact with food – Plastics – Part 4: Test methods for overall migration into olive oil by cell.*

EN 1186-6:2002, *Materials and articles in contact with food – Plastics – Part 6: Test methods for overall migration into olive oil using a pouch.*

EN 1186-8:2002, *Materials and articles in contact with food – Plastics – Part 8: Test methods for overall migration into olive oil by article filling.*

3 Overall migration into dewaxed sunflower oil by total immersion

3.1 Principle

The overall migration from a sample of the plastics is determined as the loss in mass per unit of surface area intended to come into contact with foodstuffs.

The selection of the conditions of test will be determined by the conditions of use, see clauses 4, 5 and 6 of EN 1186-1:2002.

Test specimens of known mass are immersed in dewaxed sunflower oil for the exposure time, at temperatures from 5 °C up to and including 20 °C, then taken from the dewaxed sunflower oil, blotted to remove oil adhering to the surface, and reweighed.

The specimens can usually retain absorbed dewaxed sunflower oil that is extracted and determined quantitatively by means of gas chromatography after conversion to methyl esters. Methylation is carried out by reacting a boron trifluoride/methanol complex with fatty acids formed by hydrolysing the oil with potassium hydroxide. An internal standard, triheptadecanoin, is added prior to the extraction of the absorbed dewaxed sunflower oil from the test specimens. This ensures that any active or extractable components of the plastics react with the internal standard, as well as with the extracted dewaxed sunflower oil. The internal standard is also subjected to the hydrolysis and methylation reactions, providing compensation for any inefficiencies in the hydrolysis and methylation processes.

Migration into the dewaxed sunflower oil is calculated by subtracting the mass of dewaxed sunflower oil retained by the test specimen from the mass of the test specimen after removal from the dewaxed sunflower oil, then subtracting this mass from the initial mass of the specimen.

The total loss in mass is expressed in milligrams per square decimetre of surface area of the specimen and the overall migration is reported as the mean of a minimum of three determinations on separate test specimens.

To allow for inaccuracies which may arise during the procedure and which may be difficult to detect, due for example to contamination or loss of oil during the sample handling stages, four determinations are carried out on the sample allowing for the result from one specimen to be discarded.

This method includes variations that are applicable to certain plastics.

NOTE Before starting a migration exercise, the test sample should be examined for the presence of components interfering in the determination of the amount of dewaxed sunflower oil extracted, see 7.1 of EN 1186-2:2002. If an unacceptable amount of interference is present then suitability of one of the 'other fatty food simulants' should be examined, see annex A of EN 1186-2:2002 and 9.3 and 9.5 of EN 1186-1:2002. If an interference is present which would interfere with the triheptadecanoin internal standard an alternative internal standard should be used, see annex A of EN 1186-2:2002, and 9.3 of EN 1186-1:2002.

3.2 Reagents

The reagents shall be as described in clause 4 of EN 1186-2:2002, except that olive oil, reference simulant D, as specified in 4.1 of EN 1186-1:2002 is replaced by dewaxed sunflower oil, simulant D.

3.3 Apparatus

The apparatus shall be as described in clause 5 of EN 1186-2:2002.

3.4 Preparation of test specimens

The test specimens shall be prepared as described in clause 6 of EN 1186-2:2002.

3.5 Procedure

3.5.1 General

Determine the applicability of the method by carrying out the procedure described in annex A of EN 1186-2:2002. If prior tests have established that the method is applicable then annex A of EN 1186-2:2002 may be omitted.

Before weighing, discharge any build up of static electricity with an antistatic gun or other suitable means.

3.5.2 Initial weighing of test specimens

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

3.5.3 Exposure to food simulant

Take six of the glass tubes (5.11 of EN 1186-2:2002), mark them for identification purposes. Measure 100 ml ± 5 ml of dewaxed sunflower oil (4.1 of EN 1186-2:2002, see 3.2) into each tube by measuring cylinder and stopper the tube.

NOTE.1 If the procedure described in annex D of EN 1186-2:2002 is used, it can be necessary to dry all of the dewaxed sunflower oil used for the migration test, see D.3.2.of EN 1186-2:2002.

Alternatively mark the tubes for a volume of 100 ml and fill with dewaxed sunflower oil to the mark. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Two extra tubes with a minimum of 50 ml of dewaxed sunflower oil are required as blank simulant, if the procedure described in annex D of EN 1186-2:2002 is used. Place the six or eight tubes, and two empty tubes, in the thermostatically controlled oven or incubator (5.12 of EN 1186-2:2002) set at the test temperature. Leave until the dewaxed sunflower oil has attained the test temperature, using the thermometer or thermocouple to monitor the temperature. Take all tubes from the oven and place into four of the tubes containing dewaxed sunflower oil, weighed test specimens prepared as in clause 6 and conditioned if necessary. Stopper the tubes. Ensure that the test specimens are totally immersed in dewaxed sunflower oil; if they are not, then add either glass beads or glass rods (5.22 of EN 1186-2:2002) to raise the level of the dewaxed sunflower oil until total immersion is achieved.

NOTE 2 The dewaxed sunflower oil in the fifth tube is used as a reference standard in constructing the calibration graph and if the procedure described in annex D of EN 1186-2:2002 is used, as the third blank sample for Karl Fischer titrations. The dewaxed sunflower oil in the sixth tube is used to check the temperature of the oil. If glass beads or glass rods have been used to raise the level of the dewaxed sunflower oil to achieve total immersion, then similar glass beads or glass rods should be added to the sixth tube.

Place the remaining two test specimens into the empty tubes and stopper.

NOTE 3 These two test specimens are used to check whether the sample loses mass from the evaporation of volatiles, such as water, solvents and oligomers, during the test period. If the vacuum drying procedure described in annex C of EN 1186-2:2002 is applicable these test specimens are not required as during the vacuum drying volatiles will have been removed from the test specimens.

Replace all eight or ten tubes in the thermostatically controlled oven or incubator set at the test temperature. This part of the operation should be carried out in the minimum time possible to prevent undue heat loss. Observe the temperature of the thermostatically controlled oven or incubator or the dewaxed sunflower oil (see NOTE 5) in the sixth tube 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 dewaxed sunflower oil in the sixth tube has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

NOTE 4 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.

NOTE 5 For exposure times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

Take the tubes from the oven or incubator and immediately remove the test specimens from the tubes. For those specimens which have been in dewaxed sunflower oil, allow the oil to drain. Remove any adhering dewaxed sunflower oil by gently pressing between filter papers (5.13 of EN 1186-2:2002). Repeat the pressing procedure until the filter paper shows no spots of dewaxed sunflower oil. For test specimens on supports, remove the individual test pieces from the supports to carry out this operation. Clean the supports of oil by washing with the extraction solvent and replace the test pieces on them.

NOTE 6 If the procedure described in annex D of EN 1186-2:2002 is followed, the tubes containing the oil should be retained. The tubes should be capped to prevent further change in the moisture content of the oil and the Karl Fischer determination of water content should be carried out as soon as possible.

3.5.4 Final weighing of test specimens

Perform the final weighing in accordance with 7.4 of EN 1186-2:2002.

3.5.5 Extraction of absorbed dewaxed sunflower oil

Extract the dewaxed sunflower oil in accordance with 7.5 of EN 1186-2:2002.

3.5.6 Determination of extracted dewaxed sunflower oil

Determine the extracted dewaxed sunflower oil in accordance with 7.6 of EN 1186-2:2002.

3.6 Expression of results

3.6.1 Method of calculation

Express the results in accordance with 8.1 of EN 1186-2:2002.

3.6.2 Precision

See annex F of EN 1186-2:2002.

3.7 Test report

Prepare the test report in accordance with clause 9 of EN 1186-2:2002.

4 Overall migration into dewaxed sunflower oil by cell

4.1 Principle

The overall migration from a sample of the plastics is determined as the loss in mass per unit of surface area intended to come into contact with foodstuffs.

The selection of the conditions of test will be determined by the conditions of use, see clauses 3, 4 and 5 of EN 1186-1:2002.

Test specimens of known mass are exposed in a cell to dewaxed sunflower oil for the exposure time, at temperatures above 5 °C and below 20 °C, then taken from the cell, blotted to remove oil adhering to the surface, and reweighed.

The specimens will usually retain absorbed dewaxed sunflower oil that is extracted and determined quantitatively by means of gas chromatography after conversion to methyl esters. Methylation is carried out by reacting a boron trifluoride/methanol complex with fatty acids formed by hydrolysing the oil with potassium hydroxide. An internal standard, triheptadecanoin, is added prior to the extraction of the absorbed dewaxed sunflower oil from the test specimens. This ensures that any active or extractable components of the plastics react with the internal standard, as well as with the extracted dewaxed sunflower oil. The internal standard is also subjected to the hydrolysis and methylation reactions, providing compensation for any inefficiencies in the hydrolysis and methylation processes.

Migration into the dewaxed sunflower oil is calculated by subtracting the mass of dewaxed sunflower oil retained by the test specimen from the mass of the test specimen after removal from the dewaxed sunflower oil, then subtracting this mass from the initial mass of the specimen.

The total loss in mass is expressed in milligrams per square decimetre of surface area of the specimen and the overall migration is reported as the mean of a minimum of three determinations on separate test specimens.

To allow for inaccuracies which may arise during the procedure and which may be difficult to detect, due for example to contamination or loss of oil during the sample handling stages, four determinations are carried out on the sample allowing for the result from one specimen to be discarded.

This method includes variations which are applicable to certain plastics.

NOTE Before starting a migration exercise, the test sample should be examined for the presence of components interfering in the determination of the amount of dewaxed sunflower oil extracted, see 7.1 of EN 1186-2:2002. If an unacceptable amount of interference is present then suitability of one of the 'other fatty food simulants' should be examined, see annex A of EN 1186-4:2002 and 9.3 and 9.5 of EN 1186-1:2002. If an interference is present which would interfere with the triheptadecanoin internal standard an alternative internal standard should be used, see annex A of EN 1186-4:2002, and 9.3 of EN 1186-1:2002.

4.2 Reagents

The reagents shall be as described in clause 4 of EN 1186-4:2002, except that olive oil, reference simulant D, as specified in 4.1 of EN 1186-1:2002 is replaced by dewaxed sunflower oil, simulant D.

4.3 Apparatus

The apparatus shall be as described in clause 5 of EN 1186-4:2002.

4.4 Preparation of test specimens

The test specimens shall be prepared as described in clause 6 of EN 1186-4:2002.

4.5 Procedure

4.5.1 General

Determine the applicability of the method by carrying out the procedure described in annex A of EN 1186-4:2002. If prior tests have established that the method is applicable then annex A of EN 1186-4:2002 may be omitted.

Before weighing, discharge any build up of static electricity with an antistatic gun or other suitable means.

4.5.2 Initial weighing of test specimens

Perform the initial weighing in accordance with 7.2 of EN 1186-4:2002.

4.5.3 Exposure to food simulant

Take four type A cells (5.6 of EN 1186-4:2002), mark them for identification purposes. Place in the thermostatically controlled oven or incubator (5.9 of EN 1186-4:2002), which is set at the test temperature and leave until the test temperature has been attained.

Take five glass tubes (5.8 of EN 1186-4:2002), measure 125 ml ± 5 ml of dewaxed sunflower oil (4.1 of EN 1186-4:2002) into each tube by measuring cylinder and stopper the tubes.

NOTE 1 If the procedure described in annex D of EN 1186-4:2002 is used, it can be necessary to dry all of the dewaxed sunflower oil used for the migration test, see D.3.2 of EN 1186-4:2002.

Alternatively mark the tubes for a volume of 125 ml and fill with dewaxed sunflower oil to the mark. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Two extra tubes with a minimum of 50 ml of dewaxed sunflower oil are required as blank simulant, if the procedure in annex D of EN 1186-4:2002 is used. Place the five or seven tubes, and two empty tubes, in the thermostatically controlled oven or incubator (5.9 of EN 1186-4:2002) set at the test temperature. Leave until the dewaxed sunflower oil has attained the test temperature, using the thermometer or thermocouple to monitor the temperature.

Remove the cells from the thermostatically controlled oven or incubator, dismantle the cells 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 four tubes containing 125 ml of dewaxed sunflower oil from the thermostatically controlled oven or incubator or refrigerator and transfer the dewaxed sunflower oil from each tube to each of the cells through the filler

hole. Remove the thermometer or thermocouple from the tube and insert, if applicable see NOTE 5, in one of the cells and replace the filler plugs.

NOTE 2 The dewaxed sunflower oil in the fifth tube is used as a reference standard in constructing the calibration graph (see 7.6.6 of EN 1186-4:2002) and if the procedure in annex D of EN 1186-4:2002 is used, as the third blank sample for Karl Fischer titrations. The dewaxed sunflower oil in the sixth tube is used to check the temperature of the oil. If glass beads or glass rods have been used to raise the level of the dewaxed sunflower oil to achieve total immersion, then similar glass beads or glass rods should be added to the sixth tube.

Remove the two empty tubes from the thermostatically controlled oven or incubator or refrigerator and place in each tube one of the remaining two test specimens and stopper.

NOTE 3 These two test specimens are used to check whether the sample loses mass from the evaporation of volatiles, such as water, solvents and oligomers, during the test period. If the vacuum drying procedure in annex C of EN 1186-4:2002 is applicable these test specimens are not required as during the vacuum drying volatiles will have been removed from the test specimens.

Replace the four cells and the two tubes in the thermostatically controlled oven or incubator set at the test temperature. This part of the operation should be carried out in the minimum time to prevent undue heat loss from the cells and dewaxed sunflower oil. Observe the temperature of the thermostatically controlled oven or incubator or the dewaxed sunflower oil (see NOTE 5) in the one of the cells 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 dewaxed sunflower oil in the cell has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

NOTE 4 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.

NOTE 5 For exposure times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

Take the cells and tubes from the oven or incubator and immediately remove the test specimens from the cells. For those specimens which have been in dewaxed sunflower oil, allow the oil to drain. Remove any adhering dewaxed sunflower oil by gently pressing between filter papers (5.10 of EN 1186-4:2002). Repeat the pressing procedure until the filter paper shows no spots of dewaxed sunflower oil.

NOTE 6 If the procedure in annex D of EN 1186-4:2002 is followed, the tubes containing the oil should be retained. The tubes should be capped to prevent further change in the moisture content of the oil and the Karl Fischer determination of water content should be carried out as soon as possible.

4.5.3 Final weighing of test specimens

Perform the final weighing according to 7.4 of EN 1186-4:2002.

4.5.4 Extraction of absorbed dewaxed sunflower oil

Extract the dewaxed sunflower oil in accordance with 7.5 of EN 1186-4:2002.

4.5.5 Determination of extracted dewaxed sunflower oil

Determine the extracted dewaxed sunflower oil in accordance with 7.6 of EN 1186-4:2002.

4.6 Expression of results

4.6.1 Method of calculation

Express the results in accordance with 8.1 of EN 1186-4:2002.

4.6.2 Precision

See annex F of EN 1186-4:2002.

4.7 Test report

Prepare the test report in accordance with clause 9 of EN 1186-4:2002.

5 Overall migration into dewaxed sunflower oil using a pouch

5.1 Principle

The overall migration from a sample of the plastics is determined as the loss in mass per unit of surface area intended to come into contact with foodstuffs.

The selection of the conditions of test will be determined by the conditions of use, see clauses 3, 4 and 5 of EN 1186-1:2002.

Test specimens of known mass and in the form of pouches are filled with dewaxed sunflower oil for the exposure time, at temperatures from 5 °C up to and including 20 °C, then the dewaxed sunflower oil is removed, the pouches cut open and the dewaxed sunflower oil adhering to the surface is removed by blotting. The cut portion of each pouch is then reweighed,

The specimens will usually retain absorbed dewaxed sunflower oil that is extracted and determined quantitatively by means of gas chromatography after conversion to methyl esters. Methylation is carried out by reacting a boron trifluoride/methanol complex with fatty acids formed by hydrolysing the oil with potassium hydroxide. An internal standard, triheptadecanoin, is added prior to the extraction of the absorbed dewaxed sunflower oil from the test specimens. This ensures that any active or extractable components of the plastics react with the internal standard, as well as with the extracted dewaxed sunflower oil. The internal standard is also subjected to the hydrolysis and methylation reactions, providing compensation for any inefficiencies in the hydrolysis and methylation processes.

Migration into the dewaxed sunflower oil is calculated by subtracting the mass of dewaxed sunflower oil retained by the test specimen from the mass of the test specimen after removal from the dewaxed sunflower oil, then subtracting this mass from the initial mass of the specimen.

The total loss in mass is expressed in milligrams per square decimetre of surface area of the specimen and the overall migration is reported as the mean of a minimum of three determinations on separate test specimens.

To allow for inaccuracies which may arise during the procedure and which may be difficult to detect, due for example to contamination or loss of oil during the sample handling stages, four determinations are carried out on the sample allowing for the result from one specimen to be discarded.

This method includes variations which are applicable to certain plastics.

NOTE Before starting a migration exercise, the test sample should be examined for the presence of components interfering in the determination of the amount of dewaxed sunflower oil extracted, see 7.1 of EN 1186-6:2002. If an unacceptable amount of interference is present then suitability of one of the 'other fatty food simulants' should be examined, see annex A of EN 1186-6:2002 and 9.3 and 9.5 of EN 1186-1:2002. If an interference is present which would interfere with the triheptadecanoin internal standard an alternative internal standard should be used, see annex A of EN 1186-6:2002, and 9.3 of EN 1186-1:2002.

5.2 Reagents

The reagents shall be as described in clause 4 of EN 1186-6:2002, except that olive oil, reference simulant D, as specified in 4.1 of EN 1186-1:2002 is replaced by dewaxed sunflower oil, simulant D.

5.3 Apparatus

The apparatus shall be as described in EN 1186-6:2002.

5.4 Preparation of test specimens

The test specimens shall be prepared as described in clause 6 of EN 1186-6:2002.

5.5 Procedure

5.5.1 General

Determine the applicability of the method by carrying out the procedure described in annex A of EN 1186-6:2002. If prior tests have established that the method is applicable then annex A of EN 1186-6:2002 may be omitted.

Before weighing, discharge any build up of static electricity with an antistatic gun or other suitable means.

5.5.2 Initial weighing of test specimens

Perform the initial weighing in accordance with 7.2 of EN 1186-6:2002.

5.5.3 Exposure to food simulant

Take five of the glass tubes (5.9 of EN 1186-6:2002), measure 100 ml ± 5 ml of dewaxed sunflower oil (4.1 of EN 1186-6:2002, see 5.2) into each tube by measuring cylinder (5.18 of EN 1186-6:2002) and stopper the tube.

NOTE 1 The pouch holder (5.7 of EN 1186-6:2002) should be cleaned before use, if necessary, using solvents, such as acetone and or detergents. For difficult to remove dewaxed sunflower oil, propriety solvent mixtures can be used.

WARNING Proprietary solvent mixtures usually contain caustic substances and also volatile solvents. Handle with care, using protective gloves and eye protection, in a fume cupboard. Information regarding sources of the proprietary mixtures specified in this European Standard are available from National Standards Bodies.

NOTE 2 If the procedure described in annex D of EN 1186-6:2002 is used, it can be necessary to dry all of the dewaxed sunflower oil used for the migration test, see D.3.2 of EN 1186-6:2002.

Alternatively mark the tubes for a volume of 100 ml and fill with dewaxed sunflower oil to the mark. Two extra tubes with a minimum of 50 ml of dewaxed sunflower oil are required as blank simulant, if the procedure in of annex D of EN 1186-6:2002 is used. Place the five or seven tubes and the pouch holder, in the thermostatically controlled oven or incubator (5.10 of EN 1186-6:2002) set at the test temperature.

NOTE 3 Leakage can occur from the pouches and it is advisable to have a drip tray in the oven.

Leave until the dewaxed sunflower oil has attained the test temperature, using the thermometer or thermocouple to monitor the temperature.

Remove the pouch holder from the thermostatically controlled oven or incubator and place the test specimens between the spacers.

Remove four of the tubes containing dewaxed sunflower oil from the oven and into four of the test specimen pouches pipette sufficient dewaxed sunflower oil to just fill the pouch. This shall be approximately 100 ml, but for thick/semirigid materials the quantity will be less. Place a thermocouple in one pouch and close the open corners with a clip.

NOTE 4 The dewaxed sunflower oil in the fifth tube is used as a reference standard in constructing the calibration graph (see 7.6.2.2 of EN 1186-6:2002) and if the procedure in annex D of EN 1186-6:2002 is used, as the third blank sample for Karl Fischer titrations.

Place the remaining two test specimens pouches into the pouch holder.

NOTE 5 These two test specimens are used to check whether the sample losses mass from the evaporation of volatiles, such as water, solvents and oligomers, during the test period. If the vacuum drying procedure in annex C of EN 1186-6:2002 is applicable these test specimens are not required as during the vacuum drying volatiles will have been removed from the test specimens.

Replace the pouch holder, containing the six test specimen pouches, in the thermostatically controlled oven or incubator set at the test temperature. This part of the operation should be carried out in the minimum time possible to prevent undue heat loss. Observe the temperature of the thermostatically controlled oven or incubator or the dewaxed sunflower oil (see NOTE 7) in the pouch 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 dewaxed sunflower oil in the sixth tube has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

NOTE 6 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.

NOTE 7 For exposure times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

Take the pouch holder and the tubes containing dewaxed sunflower oil from the thermostatically controlled oven or incubator.

If an evident leak has occurred with more than one pouch the test is invalid and shall be repeated.

If no evident leaks have occurred in at least three pouches, then remove the test specimen pouches from the holder.

Pour the dewaxed sunflower oil from each test specimen and wipe any excess from the outside with filter paper (5.11 of EN 1186-6:2002). Take each of the four pouches in turn, lay on the cutting slab (5.1 of EN 1186-6:2002) and using the cutting implement (5.3 of EN 1186-6:2002) carefully open by cutting through one layer along the inner edges of the seals.

Take the two portions of each test specimen and remove adhering dewaxed sunflower oil by gently pressing between filter papers (5.11 of EN 1186-6:2002). Repeat the pressing procedure until the filter paper shows no spots of dewaxed sunflower oil.

NOTE 8 If the procedure in annex D of EN 1186-6:2002 is followed, the tubes containing the oil should be retained. The tubes should be capped to prevent further change in the moisture content of the oil and the Karl Fischer determination of water content should be carried out as soon as possible.

5.5.4 Final weighing of test specimens

Perform the final weighing in accordance with 7.4 of EN 1186-6:2002.

5.5.5 Extraction of absorbed dewaxed sunflower oil

Extract the dewaxed sunflower oil in accordance with 7.5 of EN 1186-6:2002.

5.5.6 Determination of extracted dewaxed sunflower oil

Determine the extracted dewaxed sunflower oil in accordance with 7.6 of EN 1186-6:2002.

5.6 Expression of results

5.6.1 Method of calculation

Express the results in accordance with 8.1 of EN 1186-6:2002.

5.6.2 Precision

See annex F of EN 1186-6:2002.

5.7 Test report

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

6 Overall migration into dewaxed sunflower oil by article filling

6.1 Principle

The overall migration from a sample of the plastics is determined as the loss in mass per unit of surface area intended to come into contact with foodstuffs.

The selection of the conditions of test will be determined by the conditions of use, see clauses 3, 4 and 5 of EN 1186-1:2002.

Test specimens of known mass are filled with dewaxed sunflower oil for the exposure time, at temperatures from 5 °C up to and including 20 °C, then emptied and blotted to remove oil adhering to the surface, and reweighed.

The specimens will usually retain absorbed dewaxed sunflower oil that is extracted and determined quantitatively by means of gas chromatography after conversion to methyl esters. Methylation is carried out by reacting a boron trifluoride/methanol complex with fatty acids formed by hydrolysing the oil with potassium hydroxide. An internal standard, triheptadecanoin, is added prior to the extraction of the absorbed dewaxed sunflower oil from the test specimens. This ensures that any active or extractable components of the plastics react with the internal standard, as well as with the extracted dewaxed sunflower oil. The internal standard is also subjected to the hydrolysis and methylation reactions, providing compensation for any inefficiencies in the hydrolysis and methylation processes.

Migration into the dewaxed sunflower oil is calculated by subtracting the mass of dewaxed sunflower oil retained by the test specimen from the mass of the test specimen after removing the dewaxed sunflower oil, then subtracting this mass from the initial mass of the specimen.

The total loss in mass is expressed in milligrams per square decimetre of surface area of the specimen and the overall migration is reported as the mean of a minimum of three determinations on separate test specimens.

To allow for inaccuracies which may arise during the procedure and which may be difficult to detect, due for example to contamination or loss of oil during the sample handling stages, four determinations are carried out on the sample allowing for the result from one specimen to be discarded.

This method includes variations which are applicable to certain plastics.

NOTE Before starting a migration exercise, the test sample should be examined for the presence of components interfering in the determination of the amount of dewaxed sunflower oil extracted, see 7.1 of EN 1186-8:2002. If an unacceptable amount of interference is present then suitability of one of the 'other fatty food simulants' should be examined, see annex A of EN 1186-8:2002 and 9.3 and 9.5 of EN 1186-1:2002. If an interference is present which would interfere with the triheptadecanoin internal standard an alternative internal standard should be used, see annex A of EN 1186-8:2002, and 9.3 of EN 1186-1:2002.

6.2 Reagents

The reagents shall be as described in clause 4 of EN 1186-8:2002, except that dewaxed sunflower oil, reference simulant D, as specified in 4.2 of EN 1186-1:2002 is replaced by dewaxed sunflower oil, simulant D.

6.3 Apparatus

The apparatus shall be as described in clause 5 of EN 1186-8:2002.

6.4 Preparation of test specimens

The test specimens shall be as described in clause 6 of EN 1186-8:2002.

6.5 Procedure

6.5.1 General

Determine the applicability of the method by carrying out the procedure described in annex A. If prior tests have established that the method is applicable then annex A may be omitted.

Before weighing, discharge any build up of static electricity with an antistatic gun or other suitable means.

6.5.2 Initial weighing of test specimens

Perform the initial weighing in accordance with 7.2 of EN 1186-8:200.

6.5.3 Exposure to food simulant

Place a sufficient volume of dewaxed sunflower oil in a beaker in the thermostatically controlled oven or incubator (5.6 of EN 1186-8:2002) which is set at the test temperature and leave until the test temperature has been attained.

Place each test specimen on a clean, oil free surface and fill four specimens with dewaxed sunflower oil to within 0,5 cm of the top. If the container has a specified nominal volume of contents, see 8.2 of EN 1186-1:2002. Place into one of the filled test specimens a thermometer or thermocouple.

NOTE 1 If the procedure described in annex D of EN 1186-8:2002 is used, it may be necessary to dry all of the dewaxed sunflower oil used for the migration test, see D.3.2 of EN 1186-8:2002.

NOTE 2 Care should be taken not to spill any oil on the external surfaces.

NOTE 3 The two remaining test specimens are used to check whether the sample losses mass from the evaporation of volatiles, such as water, solvents and oligomers, during the test period. If the vacuum drying procedure in annex C of EN 1186-8:2002 is applicable these test specimens are not required as during the vacuum drying volatiles will have been removed from the test specimens.

Place sufficient dewaxed sunflower oil into a tube for use as reference standards in constructing the calibration graph, see 7.6.2 of EN 1186-8:2002, and if the procedure in annex D of EN 1186-8:2002 is used, as a third blank sample for Karl Fischer titrations, stopper the tube.

Place the four filled test specimens and the two empty test specimens and the reference oil in the tube in the thermostatically controlled oven or incubator set at the test temperature. This part of the operation should be carried out in the minimum time possible to prevent undue heat loss.

If the procedure in annex D of EN 1186-8:2002 is followed the test specimens filled with dewaxed sunflower oil and the tube containing dewaxed sunflower oil have to be sealed.

Observe the temperature of the thermostatically controlled oven or incubator or the dewaxed sunflower oil (see NOTE 5) in the filled article and leave the test specimens for the selected test period, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002, after the dewaxed sunflower oil in the test specimen has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

NOTE 4 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.

NOTE 5 For exposure times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

NOTE 6 In this method the outer surfaces of the specimens in the thermostatically controlled oven or incubator are exposed to the oven temperature and hence can be effected by humidity changes in the thermostatically controlled oven or incubator.

For some plastics materials these humidity changes can cause large mass variations which add to analysis time during sample conditioning. These variations can be reduced by putting all test specimens into an airtight container before placing in the thermostatically controlled oven or incubator.

Remove the test specimens and the tube from the thermostatically controlled oven or incubator and immediately empty the test specimens that contained dewaxed sunflower oil and allow the oil to drain. Remove any adhering dewaxed sunflower oil by gently pressing between filter papers (5.7 of EN 1186-8:2001). Repeat the pressing procedure until the filter paper shows no spots of dewaxed sunflower oil.

If the procedure in annex D of EN 1186-8:2002 is followed, transfer the olive from the test specimens into tubes and seal the tubes to prevent further change in the moisture content of the oil, seal the tube containing reference dewaxed sunflower oil and carry out the Karl Fischer determination of water content as soon as possible.

6.5.4 Final weighing of test specimens

Perform the final weighing in accordance with 7.4 of EN 1186-8:2002.

6.5.5 Extraction of absorbed dewaxed sunflower oil

Extract the dewaxed sunflower oil in accordance with 7.5 of EN 1186-8:2002.

6.5.6 Determination of extracted dewaxed sunflower oil

Determine the extracted sunflower oil in accordance with 7.6 of EN 1186-8:2002.

6.6 Expression of results

6.6.1 Method of calculation

Express the results in accordance with 8.1 of EN 1186-8:2002.

6.6.2 Precision

See annex F of EN 1186-8:2002.

6.7 Test report

Prepare the test report in accordance with clause 9 of EN 1186-8:2002.

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 dewaxed sunflower 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 a test method for the measurement of overall migration from plastics materials into dewaxed sunflower oil by total immersion, 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. It should be borne in mind that the test methods for overall migration described in this standard, in general, measure the migration of non volatile substances.

Commission Directive 90/128/EEC also specifies that the migration tests using rectified dewaxed sunflower oil or substitutes shall not be carried out to check compliance with the overall migration limit in cases were there is conclusive proof that the specified analytical method is inadequate from the technical standpoint.

In any such case, for 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. L349, p26. Corrigendum of the previous publication, Official Journal of the European Communities, 21 March 1990, no. L 75. p19.

[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, p14.

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