BRITISH STANDARD

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Materials and articles in contact with foodstuffs — Plastics —

Part 13: Test methods for overall migration at high temperatures

The European Standard EN 1186-13:2002 has the status of a British Standard

ICS 67.250

National foreword

This British Standard is the official English language version of EN 1186-13:2002. It supersedes DD ENV 1186-13:1999 which is withdrawn.

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

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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

Cross-references

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

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

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 13: Méthodes d'essai pour la migration globale à hautes températures

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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 1186-13:2002 has been prepared by Technical Committee CEN/TC 194 “Utensils in contact with food”, the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by March 2003, and conflicting national standards shall be withdrawn at the latest by March 2003.

This document supersedes ENV 1186-13:1999.

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

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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

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

EN 1186-13 Method A and Method B should be read in conjunction with EN 1186-1. Also, EN 1186-13 Method A should be read in conjunction with EN 1186-2.

Further Parts of this standard have been prepared concerned with the determination of overall migration from plastics materials into food simulants. Their titles are as follows:

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

Part 1	Guide to the selection of conditions and test methods for overall migration
Part 2	Test methods for overall migration into olive oil by total immersion
Part 3	Test methods for overall migration into aqueous food simulants by total immersion
Part 4	Test methods for overall migration into olive oil by cell
Part 5	Test methods for overall migration into aqueous food simulants by cell
Part 6	Test methods for overall migration into olive oil using a pouch
Part 7	Test methods for overall migration into aqueous food simulants using a pouch
Part 8	Test methods for overall migration into olive oil by article filling
Part 9	Test methods for overall migration into aqueous food simulants by article filling
Part 10	Test methods for overall migration into olive oil (modified method for use in cases where incomplete extraction of olive oil occurs)
Part 11	Test methods for overall migration into mixtures of ^{14}C-labelled synthetic triglyceride
Part 12	Test methods for overall migration at low temperatures
Part 14	Test methods for 'substitute tests' for overall migration from plastics intended to come into contact with fatty foodstuffs using test media iso-octane and 95 % ethanol

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

The annexes A and B are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

Migration testing with olive oil at high temperatures introduces a number of analytical difficulties. Experience has shown that it is difficult to achieve reproducible results owing to different laboratories having different equipment which give rise to variations in the time taken to reach the exposure temperature. A method is described for determining overall migration by total immersion using an aluminium block with a consistent thermal capacity. Other analytical difficulties with olive oil include possible oxidation of oil at elevated temperatures and the hazard to personnel working with hot oil. Replacement of olive oil by an appropriate adsorbent material, in principal, can solve or reduce these problems and offers further experimental advantages.

1 Scope

This European Standard specifies test methods for the determination of the overall migration into fatty food simulants from plastics materials and articles, by total immersion of test specimens in a fatty food simulant at temperatures from 100 °C up to and including, 175 °C for selected times. Also described is a procedure with a substitute test medium. In this substitute procedure the mass of components adsorbed on modified polyphenylene oxide (MPPO) is taken as a measure for the assessment of the overall migration into olive oil.

NOTE 1 The total immersion test method has been written for use with the fatty food simulant, olive oil. The test method can also be used with appropriate modifications with 'other fatty food simulants' called simulant D - a synthetic mixture of triglycerides, sunflower oil and corn oil. These other fatty food simulants produce different chromatograms for the simulant methyl esters to those of the methyl esters of olive oil. Suitable chromatogram peaks of the methyl esters of the other fatty food simulants should be selected for the quantitative determination of the simulant extracted from the test specimens.

NOTE 2 A comparative migration test carried out with polypropylene and polyethylene terephthalate high temperature application containers as test samples at conditions 2 h at 100 °C and 2 h at 175 °C, respectively, in contact with ^{14}C-labelled synthetic triglyceride and MPPO provided test results comparable within the analytical tolerance of the methods.

NOTE 3 To obtain reproducible and repeatable results using the MPPO method it may be necessary to measure the temperature of the test specimen before starting the migration period. An appropriate method for measuring the temperature of the test specimen needs to be established.

The described methods are most suitable for food contact articles in the form of sheets and films, but can also be applied to a wide range of articles and containers.

2 Normative references

This European Standard incorporates by dated and undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to and revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies, including amendments (including amendments).

EN 1186-1:2002, *Materials and articles in contact with foodstuffs – Plastics – Part 1: Guide to the selection of conditions and test methods for overall migration.*

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

3 Method A - total immersion in olive oil

3.1 Principle

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

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

Test specimens of known mass are immersed in olive oil for the exposure time, at temperatures from 100 °C up to 175 °C, then taken from the olive oil, blotted to remove oil adhering to the surface, and reweighed.

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

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

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

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

This method includes variations which are applicable to certain plastics.

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

3.2 Reagents

The reagents shall be as described in clause 4 of EN 1186-2:2002.

3.3 Apparatus

The apparatus shall be as described in clause 5 of EN 1186-2:2002, with the addition of:

Aluminium block or blocks with wells for holding up to ten glass tubes, see 5.11 of EN 1186-2:2002, during the exposure time period in the oven or incubator.

NOTE A diagram of a suitable block is shown in Figure A.1. The wells in the block should hold the tubes so that there is close contact between the tubes and the block. The block should be of sufficient depth that when the specimen is placed in the oil in the tubes, the level of the oil is lower than, or equal to the height of the block.

3.4 Preparation of test specimens

Prepare the test specimens in accordance with EN 1186-2, except that an additional test specimen is required. This test specimen shall be placed in the tube in which the temperature is monitored.

3.5 Procedure

3.5.1 General

See 7.1 of EN 1186-2:2002.

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

Insert a thermocouple in the metal block (3.3). Place the metal block and thermocouple in the thermostatically controlled oven or incubator, set at the test temperature and leave for 24 h.

Observe the temperature of the metal block. Confirm that the temperature of the metal block reaches the test temperature, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002.

NOTE 1 If the oven temperature is at the test temperature, but the temperature of the metal block is not, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002, the accuracy of the thermocouple should be checked and the temperature control of the oven. The oven temperature should be adjusted, if necessary, either by increasing or decreasing the oven set temperature, until the temperature of the metal block reaches the test temperature.

Take six of the glass tubes, mark them for identification purposes. Measure 100 ml ± 5 ml of olive oil into each tube by measuring cylinder and stopper the tube.

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

Alternatively mark the tubes for a volume of 100 ml and fill with olive 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 olive 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 metal block or blocks (3.3) then put the metal block or blocks in the thermostatically controlled oven or incubator (5.12 of EN 1186-2:2002) set at the test temperature. Leave until the olive oil has attained the test temperature, using the thermometer or thermocouple to monitor the temperature.

NOTE 3 More rapid temperature equilibration of the oil can be established by lifting the tubes from the block periodically and rotating gently before replacing rapidly or by stirring the oil in the tubes with a metal or glass rod, without removing from the block.

Place into four of the tubes containing olive oil, weighed test specimens prepared as in 3.4 and conditioned if necessary. Stopper the tubes. Ensure that the test specimens are totally immersed in olive 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 olive oil until total immersion

is achieved.

WARNING 1 Take care when handling the hot metal block or blocks during removal from and replacement to the oven or incubator, to prevent skin burns. Take particular care when placing the test specimens in the hot olive oil to prevent splashing or spillage of the olive oil on to the skin. It is recommended that heat protective gloves and a face shield are worn.

NOTE 4 The olive 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 olive 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 olive oil to achieve total immersion, then similar glass beads or glass rods should be added to the sixth tube.

Place one test specimen in the sixth tube where the temperature of the oil is being monitored.

NOTE 5 If a test specimen is not added to the tube the temperature monitored can be high in comparison with the temperature of the oil in the other tubes containing test specimens. For the determination of migration from thin films, which have little thermal capacity, it is not necessary to include a test specimen in the sixth tube, only the supports and gauzes as used to mount the test specimens. For thicker materials, which have some thermal capacity, it should consist of an extra test specimen.

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

NOTE 6 These two test specimens are used to check whether the sample loses mass from the evaporation of volatiles, such as solvents, during the test period.

Replace the block or blocks containing 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 to prevent undue heat loss.

NOTE 7 Where the test time for migration test is between 0,5 h and 4 h, it is particularly important that the time period between placing the test specimens in the olive oil and the olive oil reaching the test temperature, be kept as short as possible. This time period can be minimized by placing the test specimens in the tubes without removing the tubes from the block and without removing the block from the oven or incubator. It can be necessary to raise the tubes from the block to check that the oil is above the level of the sample and that the test pieces remain separated. The tubes should be raised the minimum amount necessary to carry out these checks and then replaced as quickly as possible. The sixth tube, i.e. the tube in which the temperature is monitored, should be treated in a similar manner.

Observe the temperature of the thermostatically controlled oven or incubator or the olive oil 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 olive oil in the sixth tube has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002. For test temperatures between 100 °C and 150 °C the time between the immersion of the test specimens and the test temperature being regained shall be 10 min or less. For test temperatures between 151 °C and 175 °C the time shall be 15 min or less.

NOTE 7 It has been found that these times can be achieved by using silicone oil in the wells to increase thermal conductivity between the block and the tube.

NOTE 8 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 metal block or blocks containing the tubes from the oven or incubator and immediately remove the test specimens from the tubes. Discard the test specimen that was in the sixth tube in which the temperature had been monitored. For the remaining specimens which have been in olive oil, allow the oil to drain.

WARNING 2 Take care when handling the metal block or blocks and removal of the test specimens from the olive oil at the end of the exposure period, to prevent skin burns. Use the protective wear recommended in WARNING 1.

Remove any adhering olive 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 olive 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.

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 olive oil

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

3.5.6 Determination of extracted olive oil

Determine the extracted olive 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 B.

3.7 Test report

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

4 Method B -adsorption by modified polyphenylene oxide

4.1 Principle

The surface of the article to be tested is covered with modified polyphenylene oxide and held at the selected time-temperature test conditions where the maximum temperature applicable is 175 °C. The heating takes place in a conventional oven even if the samples are for use in a microwave oven. The exposure is followed by extraction of the adsorbent using diethyl ether. Finally, the extract is evaporated to dryness using a nitrogen stream and the residue remaining is determined gravimetrically. Modified polyphenylene oxide is a porous polymer with a high molecular weight, 500 000 to 1 000 000, a very high temperature stability (T_{max} = 350 °C), a high surface area and a low specific mass (0,23 g/cm^3).

4.2 Reagents

All reagents shall be of recognized analytical quality, unless otherwise specified.

4.2.1 Diethylether of 99,8 % purity and stabilized with 1,5 % to 2,5 % ethanol.

4.2.2 Modified polyphenylene oxide (MPPO), 60 mesh to 80 mesh

Gas chromatograms obtained from extracts of new commercial MPPO have shown that unacceptably high levels of impurities may be present. Therefore, prior to its first use in this test procedure, the MPPO shall be purified by soxhlet extraction, using diethylether. The extraction is carried as follows : place the MPPO in a soxhlet cartridge and extract for 6 h with diethylether. Spread the MPPO in a Petri dish of a suitable diameter and place the dish in a fumehood. Allow the ether to evaporate while frequently mixing with a glass rod. Then place the dish in an oven at 160 °C for 6 h. After heating store the MPPO, if not needed immediately, in a closed conical flask.

NOTE 1 Heating of MPPO saturated with diethylether can be explosive. Therefore, it should be ensured that diethylether is completely evaporated before drying at 160 °C.

NOTE 2 MPPO cleaned in this way can be used repeatedly.

NOTE 3 MPPO is powdery and lightweight and is readily blown about by air currents. When drying MPPO or carrying out the exposure in a forced air oven, the oven should be set to low and cover dishes to prevent the MPPO from blowing about.

4.2.3 Nitrogen, purity 99,999 %.

4.3 Apparatus

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

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

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

4.3.4 Oven or incubator, thermostatically controlled and capable of maintaining the set temperature within the tolerances specified in Table B.2 of EN 1186-1:2002.

NOTE In case of an oven with a ventilating system the ventilation rate should be switched to low.

4.3.5 Petri dishes, glass with an internal diameter of 140 mm, heat resistant.

4.3.6 Rings, glass with an internal diameter of 125 mm and an external diameter of approximately 130 mm.

4.3.7 Erlenmeyer flasks, glass-stoppered, capacity of 300 ml.

4.3.8 Filter funnels, glass.

4.3.9 Filter, folded, with a diameter of 125 mm.

4.3.10 Vials, glass with capacities of 10 ml and 100 ml.

4.3.11 Dropping pipettes with dropper teats.

4.3.12 Lint-free cloth.

4.3.13 Plates, glass, to cover the dishes or trays.

4.3.14 Erlenmeyer flask, glass-stoppered to wash the MPPO.

NOTE The size of the flask depends on the mass of MPPO that is used (see Table 1).

4.3.15 Apparatus to blow off solvent with nitrogen

4.4 Sample preparation

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. Minimize handling of the samples and where necessary, wear cotton gloves.

4.4.2 Number of test specimens

Three specimens are required for the test.

4.4.3 Films and sheets

Lay the sample on a cutting slab. Take the glass ring (4.3.7) and place on the surface of the sample. Cut out the test specimen by cutting round the outside of the glass ring, using the cutting implement.

NOTE Taking the inner diameter of the glass ring into account the effective contact area obtained in this way is 1,22 dm².

4.4.4 Containers and other articles

Articles do not have to be cut if it is possible to cover these samples with pieces of glass. In this case, determine the flat bottom area of the article to find the required mass of adsorbent (see 4.4.5).

4.4.5 Preparation of MPPO

To cover the food contact surface sufficiently, 4 g MPPO per square decimetre of surface area of the test specimen is required.

4.5 Procedure

4.5.1 Exposure to MPPO

For flexible thin film and sheet materials (4.4.3), take three Petri dishes (4.3.6) and place a prepared test specimen (1,22 dm² effective contact area) into each dish. Stabilize the test specimen with a glass ring (4.3.7) and place 4,8 g MPPO evenly on the surface of each test specimen, inside the glass ring. Close the Petri dishes.

For rigid containers and other articles (4.4.4) which are to be tested whole, cover the flat bottom of the article with the required amount of MPPO. Calculate the required amount of MPPO according to the flat bottom food contact surface area which can be covered with MPPO, taking 4 g MPPO per square decimetre of surface area. Weigh the appropriate mass of MPPO with an accuracy of ± 0,1 g and place it on the flat bottom area of the test specimen. Cover each of the three articles with a glass plate (4.3.14).

NOTE 1 When the test sample, prepared as described, is placed in the oven the time required to reach the intended temperature can be significant when compared to the intended exposure time and the allowed tolerances on contact time and contact temperature. Therefore it can be necessary to include a procedure for the control of the time and temperature of the contact of the test specimens with the MPPO, in order to achieve reproducible and repeatable results.

NOTE 2 In the case of articles of irregular geometry and with no flat areas, a corresponding way needs to be found to expose the food contact surface to MPPO. Possible solutions are to cut appropriate parts from the article and cover or mix them with MPPO, using the conventional mass of MPPO to food contact area ratio (4.4.5). If necessary, higher amounts of MPPO should be used to ensure complete contact between the test specimen and MPPO.

For the blank determination, take an empty Petri dish and place in it the same mass of MPPO as was placed on each test specimen and cover the dish. Set the oven (4.3.5) at the required test temperature and observe the temperature. When the oven has reached the test temperature place the prepared test specimens in the oven.

Observe the temperature and leave the test samples for the selected period of time after the temperature of the oven has reached a temperature within the permitted tolerance for the test temperature, see annex B of EN 1186-1:2002 for tolerances on time and temperature.

Remove the test specimens from the oven and allow them to cool to room temperature without removing the glass covers.

NOTE 3 Cooling to room temperature takes approximately half an hour.

4.5.2 Determination of the migrating substances

Transfer the MPPO into the Erlenmeyer flask (4.3.8) with the aid of a funnel (4.3.9). If necessary use the brush (4.3.3) for complete transfer of the MPPO.

Calculate, by reference to Table 1, the volume of diethylether needed for extraction of the MPPO.

Table 1 — Volumes of diethylether needed for the extraction of MPPO

Mass of MPPO adsorbent g	Volume of diethylether for the 1st extraction ml	Volume of diethylether for the 2nd extraction ml	Volume of diethylether for the 3rd extraction ml
1,0	20	30	30
2,0	30	30	30
3,0	35	30	30
4,0	45	30	30
5,0	50	30	30
6,0	55	30	30
7,0	60	30	30
8,0	70	30	30
9,0	80	40	40
10,0	90	40	40
15,0	120	50	50
20,0	160	60	60

NOTE During the extraction of MPPO the solvent is decanted from the extracted MPPO at each washing step. Considerable amounts of solvent remain on the MPPO after each washing step which allows the use of a 100 ml volume capacity vial. If necessary, a second 100 ml vial should be used.

Pour the diethylether through the funnel (4.3.9) into the Erlenmeyer flask and shake it manually for 1 minute. Allow the Erlenmeyer flask and its contents to stand for 1 minute, without shaking.

Place a folded filter (4.3.10) into the funnel and insert the funnel into the 100 ml vial (4.3.11). Decant the diethylether from the MPPO extraction through the filter into the vial.

Repeat this extraction procedure twice, using the diethylether volumes given in Table 1.

Rinse the filter with 10 ml diethylether and concentrate the combined diethylether solutions to approximately 5 ml, first by using a rotary evaporator and finally with the aid of a gentle nitrogen flow (4.3.16).

Weigh, with an accuracy of ± 0,1 mg, a 10 ml vial (4.3.11) for each test specimen and for the blank determination.

Transfer each of the concentrated extracts, quantitatively, into the prepared 10 ml vials using a dropping pipette (4.3.12) and include a rinsing step using 5 ml of diethylether.

Evaporate the concentrates to dryness, using a stream of nitrogen, until constant weight has been achieved in the following way; evaporate to dryness (which takes approximately 30 minutes) and remove the condensed water formed at the outside of the glass vial. Under a stream of nitrogen monitor the mass of the residue remaining in the glass vial every 5 minutes. Constant weight has been achieved when, after the second weighing, the weight difference is equal to or smaller than 0,5 mg.

Determine the mass of residue by subtracting the original mass of the vial from the stable mass of the vial and residue.

4.6 Expression of results

4.6.1 Method of calculation

Express the amount of material adsorbed onto the MPPO as milligrams lost per square decimetre of the test specimen taking only that area into account which was covered by the MPPO. Calculate the adsorbed amount, *M*, for each specimen according to the following formula:

$$M = \frac{m_a - m_b}{s}$$

where

M is the mass of migrated substances adsorbed onto MPPO from the test specimen, in milligrams per square decimetre;

m_a is the mass of residue from the MPPO that had been in contact with the test specimen, in milligrams;

m_b is the mass of residue from the MPPO that had not been in contact with the test specimen, in milligrams;

S is the surface area of the test specimen that was in contact with the MPPO, in square decimetres.

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.

4.6.2 Precision

See annex B.

From a large number of tests, the described procedure provided a maximum repeatability value r = 1 mg/dm².

4.7 Test report

The test report shall contain:

a) reference to this European Standard and to the Part used for the test procedure;

b) all information necessary for complete identification of the sample such as chemical type, supplier, trade mark, grade, batch number, thickness;

c) conditions of time and temperature of exposure to the MPPO;

d) departures from the specified procedure, and reasons for these;

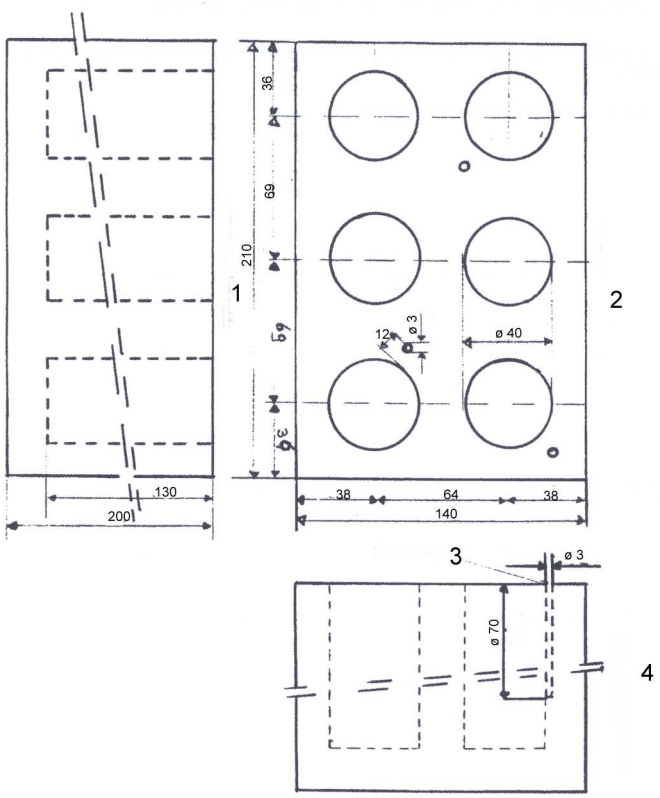
e) individual test results, and mean of these, expressed as milligrams residue per square decimetre of sample;

f) relevant comments on the test results.

Annex A
(informative)

Aluminium block

Dimensions in millimetres



Key

1 Cross section long side
2 Top view
3 Thermocouple
4 Cross section short side

Figure A.1 — Aluminium block

Annex B
(informative)

Precision data

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

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 a test method for the measurement of overall migration from plastics materials into olive 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 olive 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.

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