BRITISH STANDARD

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Materials and articles in contact with foodstuffs — Plastics —

Part 6: Test methods for overall migration into olive oil using a pouch

The European Standard EN 1186-6:2002 has the status of a British Standard

ICS 67.250

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National foreword

This British Standard is the official English language version of EN 1186-6:2002. It supersedes DD ENV 1186-6:1994 which is withdrawn.

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

— aid enquirers to understand the text;

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

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

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Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 6: Prüfverfahren für die Gesamtmigration in Olivenöl unter Verwendung eines Beutels

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Foreword

This document EN 1186-6:2002 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of an identical text or by endorsement, at the latest by October 2002, and conflicting national standards shall be withdrawn at the latest by October 2002.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 1186-6:1994.

This European Standard is one of a series of methods of test for plastics materials and articles in contact with foodstuffs.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EC Directive(s).

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

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

EN 1186-6:2002 should be read in conjunction with EN 1186-1:2002.

Further Parts of this standard have been prepared, and others are in preparation, 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 7	Test methods for overall migration into aqueous food simulants using a pouch
Part 8	Test methods for overall migration into olive oil by article filling
Part 9	Test methods for overall migration into aqueous food simulants by article filling
Part 10	Test methods for overall migration into olive oil (modified method for use in cases where incomplete extraction of olive oil occurs)
Part 11	Test methods for overall migration into mixtures of ^{14}C-labelled synthetic triglyceride

Part 12	Test methods for overall migration at low temperatures
Part 13	Test methods for overall migration at high temperatures
Part 14	Test methods for 'substitute tests' for overall migration from plastics intended to come into contact with fatty foodstuffs using test media iso-octane and 95 % ethanol
Part 15	Alternative test methods to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol

Annexes A, B, C and D to this standard are normative where applicable. Annexes E and F 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.

1 Scope

This Part of this European Standard specifies test methods for the determination of the overall migration into fatty food simulants from one surface only of plastics in the form of film or sheet, which is intended to come into contact with foodstuffs, by forming the plastics film or sheet into standard pouches and exposing them by filling at temperatures above 20 °C and up to, but not including, 100 °C for selected times.

A standard pouch is a pouch with dimensions as defined in 6.3.

This method is most suitable for plastics in the form of films and sheets, which are sealable by heat or pressure, but it is particularly applicable to those materials consisting of more than one layer, which has to be tested with the food simulant in contact only with the surface which is intended to come into contact with the foodstuffs.

NOTE This 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 will produce different chromatograms for the simulant methyl esters to those of the methyl esters of olive oil. Select suitable chromatogram peaks of the methyl esters of the other fatty food simulants for the quantitative determination of the simulant extracted from the test specimens.

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.*

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

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

3 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 and in the form of pouches are filled with olive oil for the exposure time, at temperatures above 20 °C and below 100 °C, then the olive oil is removed, the pouches cut open and the olive oil adhering to the surface is removed by blotting. The cut portion of each pouch is then reweighed,

The specimens will 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 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 olive oil extracted, see 7.1. 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.

4 Reagents

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

4.1 Olive oil, simulant D, as specified in 5.2 of EN 1186-1:2002.

4.2 Extraction solvent (see 10.1 of EN 1186-1:2002).

4.2.1 For non-polar plastics, such as polyethylene and polypropylene:

- Pentane 98 % boiling point 36 °C.

For polar plastics, such as polyamide and polyacetal:

- 95/5 by volume azeotropic mixture of pentane 98 % and ethanol 99 %.

NOTE 1 Pentane is a very volatile and highly flammable solvent. Care should therefore be taken when handling this solvent to prevent contact with sources of ignition. Ethanol is also a flammable solvent. It is not recommended that extractions with either pentane or the pentane/ethanol mixture be left unattended, particularly overnight.

NOTE 2 Due to the low boiling points of these solvents, cooled condenser water can be required to prevent undue loss of the solvent from the condenser.

or

4.2.2 Other suitable solvent.

NOTE 1 In previous methods for determining overall migration into olive oil the extraction solvent used has been 1,1,2-trichloro-trifluoroethane. For environmental reasons the use of this solvent should be avoided where possible, see 9.1 of EN 1186-1:2002. Experience has shown that this solvent, although effective for most plastics requires longer periods of extraction.

NOTE 2 Some solvents can contain non-volatile substances which, after hydrolysis and methylation processes, produce gas chromatography peaks with retention times similar to the retention times of olive oil methyl esters and methyl heptadecanoate from the internal standard. Solvents found to contain such substances should be redistilled before use.

4.3 Internal standard, triheptadecanoin (glyceryl trimargarate) CAS No. 2438-40-6[1)] of a quality such that the products from hydrolysis and methylation processes do not contain substances giving detectable gas chromatography peaks (see 9.3 of EN 1186-1:2002) with similar retention times to the olive oil methyl ester peaks. Prepared as a solution containing 2,0 mg/ml in cyclohexane.

4.4 Potassium hydroxide solution, 11,0 g/l in methanol.

4.5 Boron trifluoride, methanol complex, approximately 150 g/l BF_3.

4.6 n -Heptane.

4.7 Sodium sulfate.

4.7.1 Sodium sulfate, anhydrous, Na_2SO_4.

4.7.2 Sodium sulfate, saturated solution.

4.8 Diethyl ether.

4.9 Karl Fischer solvent, commercially prepared, methanol and chloroform based, water capacity of 5 mg/ml.

4.10 Karl Fischer titrant (for volumetric apparatus only), commercially prepared, water capacity of 2 mg/ml.

5 Apparatus

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

5.2 Tweezers, stainless steel, blunt nosed.

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

5.4 Metal templates (120 mm ± 1 mm) × (120 mm ± 1 mm) (square).

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

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

5.7 Pouch holder, as shown in Figure E.1 has been shown to be suitable, constructed from aluminium or other suitable material or an equivalent holder, plus clips to secure corners of pouches.

5.8 Conditioning containers, for conditioning test specimens at 50 % ± 5 % relative humidity and 80 % ± 5 % relative humidity at 20 °C ± 5 °C.

NOTE For 50 % relative humidity, 43 % w/v sulphuric acid solution in water is suitable and for 80 % relative humidity, 27 % w/v sulphuric acid solution is suitable. The solutions should be freshly prepared by adding a weighed amount of acid to a suitable volume of water, cooling to room temperature and making up to the required volume.

It is recommended that relative humidity and temperature be maintained during the conditioning period. Therefore the containers should be placed in a thermostatically controlled room or oven, at a temperature of approximately 20 °C, the set temperature should not vary by more than ± 1 °C.

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

5.9 Glass tubes, ground neck and stoppers, for retaining the olive oil and test specimens. Tubes with an internal diameter of approximately 35 mm and length in the range of 100 mm to 200 mm, excluding the ground neck, see 7.2 of EN 1186-1:2002, have been found to be satisfactory.

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

5.11 Filter paper, lint-free.

5.12 Anti-bumping beads.

5.13 Soxhlet type extractors, capable of holding test specimens on the supports, with 250 ml or 500 ml round bottom flasks to fit.

NOTE Alternative extractors capable of satisfactorily extracting absorbed olive oil from the test specimens can be used.

5.14 Water bath, capable of holding the flasks of soxhlet type extractors (5.15).

5.15 Rotary evaporator or distillation apparatus, for evaporation and collection of the extraction solvent.

NOTE Artificially cooled water can be necessary for efficient condensation of a low boiling point solvent.

5.16 Steam bath or water bath.

5.17 Flasks, 50 ml, long neck with condensers to fit, for methyl ester preparations.

5.18 Measuring cylinders, complying with the minimum requirements of ISO 4788, 500 ml, 250 ml, 100 ml, 25 ml, and 10 ml. A 10 ml graduated syringe may be used in place of the 10 ml measuring cylinder.

5.19 Pipettes, complying with the minimum requirements of ISO 648, 5 ml and 10 ml.

5.20 Glass beads, 2 mm to 3 mm in diameter or glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long (see 7.2 of EN 1186-1:2002).

5.21 Gas chromatograph, with flame ionisation detector equipped with an appropriate column. When using a polar column, the major peaks of olive oil, such as C16:0, methyl hexadecanoate (methyl palmitate), C16:1, methyl 9-hexadecenoate (methyl palmitoate), C18:0, methyl octadecanoate (methyl stearate), C18:1, methyl 9-octadecenoate (methyl oleate), C18:2, methyl 9,12-octadecadienoate (methyl linoleate) and the internal standard C17:0, methyl heptadecanoate (methyl margarate) shall demonstrate baseline separation. Optionally, a non-polar column can be used which shall give baseline separation of the methyl esters with 16 and 18 carbon numbers and the internal standard with 17 carbon number.

NOTE The following columns have been found to be suitable:

- Column 1, polar column, WCOT fused silica column, length 50 m, internal diameter 0,25 mm, coated with a 0,21 micrometre film of cyanopropyl silicone;

- Column 2, non polar column, BP1, length 25 m, internal diameter 0,32 mm, with a 1 micron film thickness;

- Column 3, polar column, stainless steel column 2 mm to 3 mm internal diameter and 2 m to 3 m length with a packing of 10 % to 20 % by mass of polyestersuccinate on a stationary phase of diatomaceous earth 80 mesh to 100 mesh.

5.22 Heat or pressure sealing device, for use in forming pouches

5.23 Glass tubes with ground glass necks and stoppers, of a volume of approximately 10 ml, for storing the heptane layer if necessary.

5.24 Vacuum oven or vacuum desiccator, capable of maintaining a temperature of 60 °C ± 2 °C. The vacuum oven or vacuum desiccator shall be equipped with or connected to a vacuum pump capable of achieving a vacuum

of 1,3 kPa or less. The vacuum pump shall be provided with a time controller to switch on the vacuum pump every hour for 15 min.

NOTE If a vacuum oven is not available, a vacuum desiccator placed in an oven at 60 °C can be used.

5.25 Desiccator containing self indicating silica gel or anhydrous calcium chloride.

5.26 Balance, capable of determining a change of mass of 10 mg.

5.27 Disposable plastic syringes with luer fitting. 1 ml or 10 ml size.

5.28 Wide gauge luer needles (80 mm × 1,2 mm).

5.29 Karl Fischer apparatus, either an automated volumetric titrator, or an automated coulometric titrator. The Karl Fischer titrator shall be capable of measuring the water content of the simulant with a precision (standard deviation) of 10 mg/kg or less (equivalent to 1 mg/dm^2 plastic). An automated volumetric or coulometric instrument shall be used. Manual titration procedures do not give the required accuracy or precision.

6 Preparation of test specimens

6.1 General

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

6.2 Number of test specimens

Seven test specimens are required. These test specimens are utilized as follows:

a) four test specimens for the migration test;

b) two test specimens to check for possible loss of volatiles;

c) one test specimen to determine the suitability of olive oil as the fatty food simulant and triheptadecanoin as the internal standard (see annex A);

If the conditioning test in annex C is used, one additional test specimen is required.

NOTE The two test specimens, b), are used to check whether the sample losses mass from the evaporation of volatiles, such as solvents, during the test period. If the vacuum drying procedure in annex C is used these test specimens are not required as during the vacuum drying any volatiles will have been removed from the test specimens.

If previous testing has established that interference in the gas chromatography procedure is unlikely and annex A is omitted, one fewer test specimen will be required.

A minimum of three valid test results is required to calculate the mean. Testing in triplicate is allowed but in this case if one test result is invalid repeat the entire procedure.

6.3 Cutting and preparation of test specimens

Lay the sample on the cutting slab (5.1) with the surface to be in contact with the olive oil uppermost and cut and cut test pieces using the 120 mm × 120 mm template (5.4). Two test pieces are required for each test specimen.

Place pairs of the test pieces together with the surfaces to be in contact with the olive oil facing. Using the heat or pressure sealer (5.22), join to form pouches with four seals parallel to all four edges, 10 mm from the edge. Measure the distances between the inner edges of the seals to the nearest 1 mm and calculate the total surface area of the test specimen which will be exposed to olive oil, to the nearest 0,01 dm^2. This shall be approximately 2 dm^2. Using the cutting implement, remove excess film from the sealed area (to reduce the area of film not directly exposed to olive oil) whilst leaving enough to withstand the test conditions without leaking.

Measure and record the surface area of the pouch which will be in contact with the simulant and the total external area of the pouch after trimming excess material.

Mark each pouch for identification. Cut off one corner of the pouch to leave a hole sufficiently large to insert a 100 ml pipette.

7 Procedure

7.1 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.

7.2 Initial weighing of test specimens

7.2.1 Determine the need for conditioning of the test specimens by carrying out the procedure described in annex B or in annex C. If prior tests have established that sample conditioning is not required then annex B and annex C may be omitted. If prior tests have established that the procedure described in annex D is applicable to the sample, then annex B or annex C may be omitted.

7.2.2 If the tests described in annex B or annex C show that conditioning is not necessary, determine and record the mass of each test specimen.

7.2.3 If the tests described in annex B or annex C show that conditioning is necessary, follow the directions in the relevant annex to determine the initial mass of the sample.

7.2.4 If the tests described in annex B show that conditioning is necessary, but constant mass cannot be achieved within five days then carry out the conditioning procedure described in C.3.1 or annex D.

NOTE 1 Long conditioning periods are not satisfactory due to oxidation of the olive oil which can occur upon prolonged conditioning.

NOTE 2 The conditioning procedures described in annex C and annex D can be used if it has been established that these procedures are more suited to the polymer type under test.

7.3 Exposure to food simulant

Take five of the glass tubes (5.9), measure 100 ml $\pm$ 5 ml of olive oil (4.1) into each tube by measuring cylinder (5.18) and stopper the tube.

NOTE 1 The pouch holder (5.7) should be cleaned before use, if necessary, using solvents, such as acetone and or detergents. For difficult to remove olive oil, use propriety solvent mixtures.

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 is used, it can be necessary to dry all of the olive oil used for the migration test, see D.3.2.

Alternatively mark the tubes for a volume of 100 ml and fill with olive oil to the mark. Two extra tubes with a minimum of 50 ml of olive oil are required as blank simulant, if the procedure in annex D is used. Place the five or seven tubes and the pouch holder, in the thermostatically controlled oven or incubator (5.10) 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 olive 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 olive oil from the oven and into four of the test specimen pouches pipette sufficient olive 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 olive oil in the fifth tube is used as a reference standard in constructing the calibration graph (see 7.6.2.2) and if the procedure in annex D 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 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 olive 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 olive 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 olive 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 olive oil from each test specimen and wipe any excess from the outside with filter paper (5.11). Take each of the four pouches in turn, lay on the cutting slab (5.1) and using the cutting implement (5.3.) 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 olive oil by gently pressing between filter papers (5.11). Repeat the pressing procedure until the filter paper shows no spots of olive oil.

NOTE 8 If the procedure in annex D is followed, retain the tubes containing the oil. The tubes should be capped to prevent further change in the moisture content of the oil and carry out the Karl Fischer determination of water content as soon as possible.

7.4 Final weighing of test specimens

7.4.1 For those specimens which did not require conditioning to obtain their initial masses (see 7.2.2), weigh all six test specimens i.e. the four that have been in olive oil and the two that were in the empty tubes and record the mass of each test specimen.

7.4.2 If conditioning of the test specimens was carried out using the procedure in annex B (see 7.2.3) then repeat the procedure.

7.4.3 If conditioning was carried out before the initial weighing using the procedure described in annex C (see 7.2.4) then carry out the procedure described in C.4.

7.4.4 If it was decided that the procedure described in annex D (see 7.2.4) was applicable to the test sample, then carry out that procedure.

7.4.5 If the final mass of each of the empty test specimens is less than their initial mass by more than 2,0 mg, then volatile substances have been lost and adjustment may be made, see 9.4 of EN 1186-1:2002, to the final mass for each test specimen such that the values obtained are a measure of the migration of non-volatile substances only.

7.5 Extraction of absorbed olive oil

Take four flasks, 250 ml or 500 ml, as appropriate to the size of the soxhlet type extractor (5.13) to be used for the extraction, and place in each flask 10,0 ml of the internal standard cyclohexane solution of triheptadecanoin (4.3), using a pipette (5.19), or an alternative higher quantity if more than 100 mg of olive oil is present.

NOTE 1 If the test specimens have retained more than 100 mg of olive oil, 10,0 ml of the internal standard solution will be insufficient for optimum precision in the gas chromatography determination after extraction. Before commencing the operations in this clause an estimation of the quantity of olive oil retained in the test specimens should be obtained by comparing the final masses of the test specimens with their initial masses. If considered necessary the quantity of internal standard solution can be increased from 10 ml although it is essential that the same quantity is used for each test specimen, and that this quantity is also used with the olive oil standards for the calibration graph (see 7.6.2.2). As a guide, approximately 0,5 mg of the internal standard is required for every mg of extracted olive oil.

Add sufficient extraction solvent (4.2) to allow cycling of the soxhlet type extractor (approximately 200 ml or 400 ml, according to the size of the flask) with anti-bumping beads (5.12) to control boiling.

Place the four test specimens which have been in contact with olive oil into four soxhlet type extractors. Couple each soxhlet to a flask containing the internal standard prepared as above. Using either a water bath or steam bath (5.16), extract for a period of $7\ ^{+1}_{0}$ h, with a minimum of six cycles per hour, ensuring that the test pieces are totally submerged in the solvent during each soxhlet cycle, and that they remain separated from each other.

Drain all of the solvent from the soxhlet type extractors, remove the flasks from the soxhlet type extractors and evaporate the solvent to approximately 10 ml using a rotary evaporator, or simple distillation apparatus (5.15). Transfer the solutions containing the extracted olive oil and internal standard to separate 50 ml flasks (5.17), and wash each flask with three portions of 5 ml of solvent. Add the three washings to the respective individual 50 ml flasks. Evaporate to dryness using a rotary evaporator or a waterbath (5.15 or 5.14).

NOTE 2 Oxidation of the olive oil is to be avoided where possible. Therefore evaporation of the solvent to dryness should be carried out under mild conditions of temperature. In addition exposure of the olive oil to oxygen should be limited.

NOTE 3 Some types of plastics are known to retain some of the absorbed olive oil. In these cases extraction of the olive oil is incomplete and a second extraction with a more polar solvent is required, see also 9.2 of EN 1186-1:2002.

Repeat the extraction of the test specimens for an additional $7\ ^{+1}_{0}$ h, with diethyl ether (4.8), adding a further quantity of the internal standard solution.

NOTE 4 The same quantity of internal standard solution is used as for the first 7 h extraction. This quantity might not be the optimum if the quantity of olive oil in the first 7 h extraction is high. Good precision is not required for the second 7 h

determinations since they are intended primarily as a check on the efficiency of the first 7 h extraction and using the same quantity of internal standard enables one calibration graph to be used.

If previous testing has established that all of the olive oil will be extracted from the test specimens during the first 7 h extraction then the second 7 h extraction may be omitted.

Isolate the residues in 50 ml flasks, using the procedure described above.

Determine the extracted olive oil in both the first 7 h and the second 7 h extraction by the procedure described in 7.6, but retain the test specimens in the soxhlet type extractors until the extracted olive oil has been determined for the second extraction.

7.6 Determination of extracted olive oil

7.6.1 Preparation of fatty acid methyl esters

Add 10 ml ± 0,2 ml of n-heptane to each of the 50 ml flasks containing the first 7 h extraction residue, by measuring cylinder (5.18), ensuring that the residues of olive oil and plastics extractables dissolve or are well dispersed by shaking, warming or by ultrasonic treatment.

NOTE 1 Unless the residues in the flasks are dissolved or well dispersed in the n-heptane, quantitative hydrolysis or methylation of the olive oil and of the internal standard might not be obtained under the conditions described particularly when these residues contain extractables from plastics in excess of 50 mg. The internal standard might not react with the plastics extractables to the same degree as does the olive oil and correct results for olive oil might not be obtained.

Add by measuring cylinder or graduated syringe (5.18), 10 ml ± 0,2 ml of the potassium hydroxide solution (4.4) and a few anti-bumping beads (5.12). Connect a condenser to the flask and boil the mixture under reflux for 10 min ± 1,0 min.

Add through the condenser by measuring cylinder, or graduated syringe (5.18), 5,0 ml ± 0,2 ml of the methanol solution of boron trifluoride (4.5) and boil the mixture under reflux for 2 min ± 0,25 min.

Cool to room temperature and add, by measuring cylinder (5.18), 15 ml to 20 ml of saturated sodium sulfate solution (4.7.2) and shake well. Then add further sodium sulfate solution until the liquid level reaches the neck of the flask. Allow to stand until the phases have separated.

NOTE 2 The methyl esters for the subsequent gas chromatographic determination are in the upper, n-heptane, layer.

Treat the residues from the second 7 h extraction as described above.

If there will be a delay of more than seven days in using a methyl ester solution for the gas chromatographic determinations, transfer the n-heptane layer to a small stoppered tube (5.23) containing solid anhydrous sodium sulfate (4.7.1) and store in a refrigerator.

7.6.2 Determination of fatty acid methyl esters

7.6.2.1 Instrument

Determine the methyl esters of the olive oil fatty acids using a gas chromatograph (5.21).

NOTE 1 For column 1 described in the note to 5.21 the following operating conditions have been found to be suitable:

carrier gas	helium at 2 ml/min
injector	split (ratio 40:1)
detector	flame ionisation

temperature programme	initially 1 min at 140 °C then ramped at 5 °C to 190 °C and maintained at 190 °C for 8 min.
injector temperature	220 °C
detector temperature	240 °C

For column 2 described in the note to 5.21 the following operating conditions have been found to be suitable

carrier gas	helium
oven temperature	250 °C isothermal
injector temperature	320 °C
detector temperature	320 °C

For column 3 described in the note to 5.21 the following operating conditions have been found to be suitable

carrier gas	nitrogen at 25 ml/min
oven temperature	185 °C to 195 °C
injector temperature	190 °C to 200 °C
detector temperature	190 °C to 200 °C

Use an integrator to measure the area of each of the olive oil peaks and the internal standard. Optionally a chart recorder may be used to record the chromatogram and the height of the various peaks is measured. In this case only the height of the internal standard and the major peak of olive oil (C18 or C18:1) shall be used for quantification of the amount of olive oil.

NOTE 2 The use of an integrator and measurement of the peak area is the preferred method.

7.6.2.2 Calibration graph

Weigh a range of quantities of the blank reference olive oil which has been subjected to the same test conditions as the test specimens into 50 ml flasks (5.17). Weigh a range of olive oil quantities spanning the quantities of olive oil in the first 7 h extractions, taking no fewer than four standards.

Add 10,0 ml of the internal standard cyclohexane solution of triheptadecanoin (4.3) to each flask using a pipette (5.19), or the alternative quantity which has been added to the extraction flasks in 7.5. Remove the cyclohexane using a rotary evaporator or water bath (5.15 or 5.14). Subject the olive oil quantities, with the added internal standard, to the methyl ester preparation procedure described in 7.6.1.

Inject each of the n-heptane methyl ester solutions in duplicate, as a minimum, into the gas chromatographic column.

NOTE 1 Typical chromatograms generated using columns 1 and 2 are shown respectively in Figures E.2 and E.3.

Construct a calibration graph, plotting the ratios of olive oil methyl esters to the internal standard peak on the y-axis and against the weighed quantities of olive oil on the x-axis.

Various methods for the construction of a calibration graph are suitable and the choice of method depends on the equipment and chromatographic column used. The following methods are acceptable:

Method 1 Peak height method

Measure the peak height of the internal standard peak and of the methyl oleate (C18:1) peak, when a polar column has been employed. In the case where a non-polar column has been used for the separation of the methyl ester, then measure the internal standard peak and the C18 peak of the olive oil. Calculate the ratio of the measured C18 peaks to the internal standard peak and plot the ratios versus the weighed quantities of olive oil.

Method 2 Peak area method

Measure the peak area of the internal standard peak and of each of the methyl esters originating from the olive oil. Add together the peak areas of the C16 and C18 peaks if a non-polar column was employed. If a polar column was used, sum the areas of all the peaks (C16:0, C16:1, C18:0, C18:1 and C18:2) originating from the olive oil. Calculate the ratio of the combined areas of the measured peaks to the area of the internal standard peak and plot the ratio versus the weighed quantities of olive oil.

Method 3 Peak area method in the case of interference from the test sample

In the event that the analysis of a blank test sample, see annex A, has revealed an interference with one or more of the olive oil methyl esters, but not all of the peaks, then this peak or peaks shall be excluded from the calculation of the total area of the olive oil methyl esters. Calculate the ratio of the total area of the methyl esters originating from olive oil and which are free from interference and the area of the internal standard and plot the ratios versus the weighed quantities of oil.

NOTE 2 A typical calibration graph is shown in Figure E.4.

Calculate from each calibration standard chromatogram the C18:1/C16:0 ratio if a polar column was used or C18/C16 ratio in the case of a non polar column. Determine the mean ratio value from the duplicate or multiple injections for comparison with the same ratio obtained from the test specimen extracts, see 7.6.2.3.

7.6.2.3 Determination of olive oil absorbed by test specimens

Inject into the gas chromatograph (5.21) a suitable quantity from each of the n-heptane methyl ester solutions prepared from the residues containing the extracted olive oil (see 7.6.1). Inject in duplicate, as a minimum.

For each chromatogram, measure the height or area of the olive oil methyl ester peak or peaks and the internal standard peak using the same peaks and method as used in the construction of the calibration graph, see 7.6.2.2. Calculate the ratio of the relevant peaks to the internal standard peak for each chromatogram and for each solution determine the mean ratio value from the duplicate or multiple injections.

Calculate the amount of olive oil extracted from the test specimen as follows:

Graphical method

Read the amount of olive oil extracted from the calibration graph (7.6.2.2) using the calculated ratio of the relevant olive oil peak or peaks to the internal standard peak.

Calculation from regression parameters

If the regression line equation is

$$y = a\,x + b \qquad (1)$$

then:

$$m_{oo} = \frac{(y - b)}{a} \qquad (2)$$

where

m_{oo} is the mass of olive oil extracted from the sample, in milligrams;

a is the slope of the calibration graph;

b is the intercept of the calibration graph;

x is the mass of olive oil in the standard, in milligrams;

y is the ratio of olive oil methyl esters to internal standard.

Both procedures yield directly the amount of olive oil extracted from the test specimen, in milligrams.

NOTE 1 The method applying calculation from the regression parameters is the preferred method.

If olive oil is found in the second extract from more than one of the test specimens and the amount is less than 10 mg, but measurable, add this to the amount determined from the first 7 h extraction and record the total mass of extracted olive oil for each test specimen in grams.

If more than 10 mg of olive oil is found in the second extract or the ratio C18 to the C16 peaks has changed, see 9.2 and 9.6 of EN 1186-1:2002.

For each chromatogram from the first 7 h extractions, calculate the ratio of the height or area of the C18 peak to the height or area of the C16 peak. Determine the mean value of these ratios and compare to the similar ratio determined in 7.6.2.2 from the olive oil calibration chromatograms. Establish whether the difference between the two ratios values is acceptable, see 9.6 of EN 1186-1:2002.

NOTE 2 A change in the C18/C16 ratio for extracted olive oil samples compared with the same ratio for olive oil used for the calibration graph indicates that some reaction or fractionation of the olive oil has occurred, either during the test period or during extraction of the test specimens. Such changes will have an adverse effect on the overall migration result.

8 Expression of results

8.1 Method of calculation

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

$$M = \frac{[m_a - (m_b - m_c)] \times 1000}{S} \qquad (3)$$

where

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

m_a is the initial mass of the test specimen, before contact with the olive oil, in grams (see 7.2.2 or 7.2.3 or 7.2.4 as appropriate);

m_b is the mass of the test specimen after contact with olive oil, in grams. (see 7.4) or corrected mass (see equation (4)) where the loss of volatiles is greater than 2 mg per test specimen (see 7.4.5);

m_c is the mass of olive oil absorbed by test specimen, in grams (see 7.6.2.3);

S is the surface area of the test specimen in contact with food simulant in square decimetres. See 8.5 of EN 1186-1:2002.

Calculate the result for each test specimen to the nearest 0,1 mg/dm² and the mean of the valid test results, to the nearest milligrams per square decimetre.

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

The corrected mass is calculated using the formula:

$$m_b = m_b' + m_d \qquad (4)$$

where

m_b is the corrected mass of the test specimens allowing for loss of volatiles in empty tubes, in grams;

m_d is the mean loss in mass of the test specimens in the empty tubes, in grams;

m_b' is the mass of the test specimen after contact with the olive oil, in grams.

NOTE This allowance for loss of volatile substances during the exposure period of the test specimens assumes the quantities of volatile substances lost from the test specimens immersed in the olive oil equates to the mean loss of volatile substances from the two test specimens in the empty tubes.

If the procedure described in annex D has been followed express the overall migration as milligrams lost per square decimetre of surface of the sample which is intended to come into contact with foodstuffs, calculated for each test specimen using the following formula:

$$M_D = \frac{[m_a - (m_b - m_c + M_w)] \times 1000}{S} \qquad (5)$$

where

M_D is the overall migration into olive oil, in milligrams per square decimetre of the surface area of sample intended to come into contact with the foodstuff obtained by following the procedure described in annex D;

m_a is the initial mass of the test specimen, before contact with the olive oil, in grams (see 7.2.2 or 7.2.3 or 7.2.4 as appropriate);

m_b is the mass of the test specimen after contact with olive oil, in grams. (see 7.4) or corrected mass (see equation (4)) where the loss of volatiles is greater than 2 mg per test specimen (see 7.4.4);

m_c is the mass of olive oil absorbed by test specimen, in grams (see 7.6.2.3);

M_w is the mass of water lost or gained from the migration test specimens, in milligrams;

S is the surface area of the test specimen in contact with the food simulant in square decimetres. See 8.5 of EN 1186-1:2002.

8.2 Precision

See annex F.

9 Test report

Where the plastics material is intended for use in contact with fatty foods for which reduction factors are permitted then these factors shall be taken into account when reporting the results, see 11.2 of EN 1186-1:2002.

The test report shall include the following:

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

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

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

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

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

f) reference to the procedure used for determining the mass of moisture sensitive samples and the reason for selecting that procedure;

g) any adjustment made for loss of volatile substances from the test specimens;

h) relevant comments on the test results; including the area of the pouch in contact with the simulant, and the total external area of the pouch after trimming excess material;

i) reference to any reduction factor used in calculating migration.

Annex A
(normative)

Determination of the suitability of olive oil as the fatty food simulant and of triheptadecanoin as the internal standard

A.1 Principle

This procedure is carried out to verify that olive oil is suitable as the fatty food simulant, and that triheptadecanoin is suitable for use as an internal standard for the gas chromatographic determination of olive oil as its methyl esters.

A.2 Procedure

A.2.1 Weigh 45 mg to 55 mg of olive oil (4.1) into a 50 ml flask (5.17) and add 10,0 ml of the cyclohexane solution of triheptadecanoin (4.3) by pipette (5.19). Remove the cyclohexane using a rotary evaporator or water bath (5.15 or 5.14) and add, by measuring cylinder or graduated syringe (5.18), 10 ml ± 0,2 ml of n-heptane (4.6). Ensure the residue of olive oil is well dispersed by shaking, warming or by ultrasonic treatment.

A.2.2 Add by measuring cylinder or graduated syringe (5.18), 10 ml ± 0,2 ml of the potassium hydroxide solution (4.4) and a few anti-bumping beads (5.12). Connect a condenser to the flask and boil the mixture under reflux for 10 min ± 0,5 min.

Add through the condenser by measuring cylinder, or graduated syringe (5.18), 5 ml ± 0,2 ml of the methanol solution of boron trifluoride (4.5) and boil the mixture under reflux for 2 min ± 0,25 min.

Cool to room temperature and add, by measuring cylinder (5.18), 15 ml to 20 ml of saturated sodium sulfate solution (4.7.2) and shake well. Then add further sodium sulfate solution until the liquid level reaches the neck of the flask. Allow to stand until the phases have separated.

If there will be a delay of more than seven days in using a methyl ester solution for the gas chromatographic determinations, transfer the n-heptane layer to a small stoppered tube (5.23) containing solid anhydrous sodium sulfate (4.7.1) and store in a refrigerator.

NOTE The methyl esters for the subsequent gas chromatographic determination are in the upper, n-heptane, layer.

A.2.3 Inject the methyl ester solution into the gas chromatograph (5.21).

NOTE A volume of 1 µl to 3 µL has been found suitable for the columns described in the note to 5.21.

Retain the chromatogram for comparison.

A.2.4 Take one of the test specimens, as prepared in clause 6, and place it in a soxhlet type extractor (5.13). Take a 250 ml or 500 ml flask (5.17) and add 10 ml of cyclohexane without the internal standard and sufficient extraction solvent (4.2) to allow cycling of the soxhlet type extractor (approximately 200 ml or 400 ml, according to the size of the flask) with anti-bumping beads (5.12)) to control boiling. Using either a water bath or steam bath (5.16) extract for a period of $7\ ^{+1}_{0}$ h, with not less than six extraction cycles per hour, ensuring that the test pieces are totally submerged in the solvent during each soxhlet cycle, and that they remain separated from each other.

A.2.5 Drain all of the solvent from the soxhlet type extractor into the flask, remove the flask from the soxhlet type extractor and evaporate the solvent to a volume of approximately 10 ml using a rotary evaporator or simple distillation apparatus (5.15). Transfer the solution of the test specimen extract to a separate 50 ml flask (5.17) and

wash the flask with three portions of 5 ml of solvent. Add the washings to the 50 ml flask. Evaporate to dryness using a rotary evaporator or water bath (5.15 or 5.14).

A.2.6 Add 10,0 ml ± 0,2 ml of the n-heptane to the 50 ml flask. Ensure that the test specimen extract is well dispersed by shaking, warming or by ultrasonic treatment. Then subject the contents of the flask to the methyl ester preparation procedure, described in A.2.2 and inject the same volume of the resulting solution as used in A.2.3 into the gas chromatograph (5.21). Retain the chromatogram.

A.3 Conclusions

Compare the chromatogram of the methyl esters produced from the olive oil and internal standard in the procedure in A.2.3 with the chromatogram of the preparation from the test specimen extract produced in procedure A.2.6. If peaks are present in the chromatogram of the extract with similar retention times to those of the peaks of olive oil methyl esters, and equate to 2 mg or more of olive oil, then the method is unsuitable for the material under examination and 9.3 of EN 1186-1:2002 has to be consulted. If a polar column has been used and interferences are observed on the peaks of the C18:0 and/or C18:2 peak, but not on other olive oil methyl ester peaks, then olive oil may be considered to be a suitable fat simulant, following method 3 given in 7.6.2.2.

If a peak is present in the chromatogram of the extract with similar retention time to that of the peak for methyl heptadecanoate, originating from triheptadecanoin, the internal standard, and is more than 1 % of the height or area of that peak, then consider an alternative internal standard.

NOTE 1 A suitable alternative internal standard is trinonadecanoin or hydrocinnamic acid, ethyl ester, see 9.3 of EN 1186:2002.

NOTE 2 Figures E.1 and E.2 show typical chromatograms of the methyl esters of olive oil and triheptadecanoin using columns 1 and 2 respectively.

Annex B
(normative)

Determination of the need for sample conditioning

B.1 Principle

The procedures described in B.2 and B.3 are carried out to determine whether the conditioning of test specimens with respect to moisture content will be required. The procedures described in B.4 and B.5 are carried out to determine the masses of test specimens, which have been shown to be moisture sensitive.

B.2 Procedure

B.2.1 Take one test specimen, as prepared in clause 6 and place in a container (5.8) maintained at 80 % relative humidity for 24 h ± 4 h. Remove the test specimen and weigh as quickly as possible after its removal from the controlled environment, to minimise loss of moisture and change in mass.

B.2.2 Place the same test specimen in a container (5.8) maintained at 50 % relative humidity for 24 h ± 4 h. Remove the test specimen and weigh , taking the same precautions as in B.2.1.

B.3 Conclusions

If the difference between the masses of the test specimen as determined in B.2.1 and B.2.2 is greater than 2 mg/dm², then conditioning of the test specimens will be necessary before each weighing operation in the test procedure.

If the difference between the masses of the test specimen as determined in B.2.1 and B.2.2 is less than 2 mg/dm², then conditioning of the test specimens will not be necessary before each weighing operation in the test procedure.

B.4 Initial weighing of test specimens

Place the test specimens in the container maintained at 50 % relative humidity, weigh at intervals of about 24 h, until the change in mass between consecutive weighings of each test specimen is less than 2 mg/dm² and record the eventual mass of each test specimen.

B.5 Final weighing of test specimens

Replace the test specimens in the container maintained at 50 % relative humidity, weigh at intervals of about 24 h, until the change in mass between consecutive weighings of each test specimen is less than 2 mg/dm² and record the eventual mass of each test specimen.

Annex C
(normative)

Determination of the need for sample conditioning and determination of the mass of moisture sensitive test specimens, by vacuum drying

C.1 Principle

The procedure described in C.2 is carried out to establish whether the conditioning of test specimens with respect to moisture content will be required. The procedures described in C.3 and C.4 are carried out to determine the masses of test specimens, which have been shown to be moisture sensitive.

C.2 Establishing the need for conditioning of test specimens

C.2.1 Procedure

Take one test specimen, as prepared in clause 6 and determine the mass to the nearest milligramme. Place the test specimen in a vacuum oven (5.24) at 60 °C ± 5 °C. Reduce the pressure in the oven to 1,3 kPa or less. Leave the test specimen in the oven for 60 min ± 10 min. Release the pressure and transfer the test specimen from the vacuum oven to a desiccator (5.25) containing self indicating silica gel or anhydrous calcium chloride. Determine, after cooling for 60 min ± 10 min the mass of the test specimen. Calculate the difference between the mass of the test specimen before and after the one hour vacuum conditioning. Discard the test specimen.

C.2.2 Conclusions

If the difference between the masses of the test specimen is greater than 2 mg/dm², then conditioning of the test specimens to be used in the test will be necessary before each weighing operation in the test procedure (C.3). If the difference between the masses of the test specimen is less than 2 mg/dm², then conditioning of the test specimens to be used in the test will not be necessary before each weighing operation in the test procedure.

C.3 Initial weighing of test specimens

C.3.1 Conditioning of test specimens

Weigh the four test specimens, as prepared in clause 6, then transfer to a vacuum oven (5.24) at 60 °C ± 5 °C and reduce the pressure to approximately 1,3 kPa using a high vacuum pump. The vacuum pump can be turned off provided the pressure is maintained. Turn on the vacuum pump every hour for a period of 10 min to 15 min to remove moisture from the oven and to refresh the vacuum. Leave the test specimens under this condition in the vacuum oven for a period of 24 h ± 2 h. Transfer the test specimens from the vacuum oven to a desiccator (5.25) containing self indicating silica gel or anhydrous calcium chloride. Determine, after cooling for 60 min ± 10 min, the mass of the test specimen. Repeat the conditioning procedure until the change in mass between two consecutive weighings is less than 2 mg/dm². Record the final mass of each test specimen.

C.3.2 Reconditioning of the test specimens

Place the test specimens at ambient humidity or in a container (5.8) maintained at 80 % relative humidity, until the test specimens have regained at least 70 % of the mass lost during vacuum drying, see 9.9 of EN 1186-1:2002. The test specimens are now ready to be brought into contact with the olive oil.

C.4 Final weighing of test specimens

After the exposure period, the test specimens are placed in the vacuum oven for 24 h periods as above, until constant mass has been achieved. Record the eventual mass of each test specimen. The test specimens can now be extracted to recover the olive oil.

Annex D
(normative)

Determination of change in moisture content of test specimens by measurement of the transfer of water to, or from olive oil, by Karl Fischer titration

D.1 Principle

Simulant from the migration experiment is stored in sealed containers protected from atmospheric moisture prior to analysis for water. The Karl Fischer titration employs dedicated volumetric or coulometric apparatus and is specific for water. The water content of fresh and used simulant is determined and the water loss (or gain) from the test specimen is thus calculated from the difference. This value is then used to compensate for water loss (or gain) in the gravimetric overall migration procedure.

D.2 Reagents

D.2.1 Samples of olive oil simulant obtained from each of the four (or three) tubes which contained the test specimens immediately following the migration testing (50 ml).

D.2.2 Three samples of the blank olive oil simulant carried through an equivalent migration procedure but with no test specimen present (50 ml).

D.2.3 Sample of olive oil (4.1) simulant intended for use in migration tests.

D.3 Procedure

D.3.1 Assessment of the simulant

Take replicate (n=5) portions of simulant intended for use in the migration tests (D.2.3) and determine the performance of the Karl Fischer apparatus employed (5.29). The precision (standard deviation) of this determination shall be 10 mg/kg or less in order to achieve the required precision of 1mg/dm^2 equivalence in the final result. If this precision cannot be achieved because the background level of water in the simulant is high, dry the simulant according to D.3.2.

NOTE If the precision remains inadequate, an alternative Karl Fischer apparatus should be employed.

D.3.2 Drying the simulant

Take sufficient simulant for the migration tests and blanks and hold for 4 h at 150 °C whilst purging with dry nitrogen at 15 ml/min to 20 ml/min. Assess the simulant according to D.3.1. Store the dried simulant in a sealed container.

NOTE The water content of the simulant should typically be 50 mg/kg or less after this procedure.

D.3.3 Preparation of samples for testing

When using this procedure determine the exact mass of simulant employed by mass difference (M_m). Expose three blank portions of simulant (simulant but no test specimen) in parallel with the exposed test specimens in order to provide simulant blanks.

D.3.4 Karl Fischer titration of simulant samples

D.3.4.1 Calibrate the Karl Fischer titrator as recommended by the manufacturer.

D.3.4.2 Take triplicate subsamples of simulant from each of the four portions of simulant which have been in contact with the test specimens and from each of the three simulant blanks. Determine the water content according to D.3.4.3.

D.3.4.3 Introduce an aliquot of simulant into the titrator.

NOTE For typical coulometric instruments a smaller sample of 1 g is adequate. Approximately 10 g is required for volumetric apparatus.

Using a tared syringe and needle (5.27 and 5.28) determine the exact mass added (M_o) to a precision of 10 mg by back-weighing the empty syringe. Allow time for the sample to dissolve (typically 1 min to 2 min) before commencing the titration.

D.3.4.4 The Karl Fischer apparatus will typically output directly the mass of water found (Q_W). Calculate the water content of the simulant, W_c, in milligrams per kilogram as $W_C = Q_W / M_O$.

D.3.5 Precision of the water determination

The results W_c for the triplicate subsamples should agree to within ± 10 mg/kg. If this criterion is satisfied for each of the simulant samples, calculate the mean of the triplicate results for each of the four migration samples to give W_{S1}, W_{S2}, W_{S3}, and W_{S4} and for the three blank simulants to give W_{b1}, W_{b2} and W_{b3}. If a variation in excess of ± 10 mg/kg is found, examine the Karl Fischer procedure (D.3.4) and remove the source of variation.

D.3.6 Reproducibility of simulant blank experiments

If the results W_{b1}, W_{b2}, and W_{b3} for the three simulant blank samples agree to within ± 10 mg/kg, calculate the mean to give W_b. If a variation in excess of ±10 mg/kg is seen, examine the procedure and remove the source of variation.

D.4 Expression of results

Calculate the mass of water (M_W) lost from or gained by each migration test specimen as follows:

$$M_W = (W_S - W_b) \times M_m \qquad (6)$$

where

M_W is the mass of water lost from or gained by the migration test specimens, in milligrams;

W_S is the water content of migration simulant, in milligrams per kilogram;

W_b is the average water content of blank simulants in milligrams per kilogram;

M_m is the mass of simulant used for migration test, in kilograms;

Annex E
(informative)

Pouch holder, typical chromatograms and calibration graph

Dimensions in millimetres

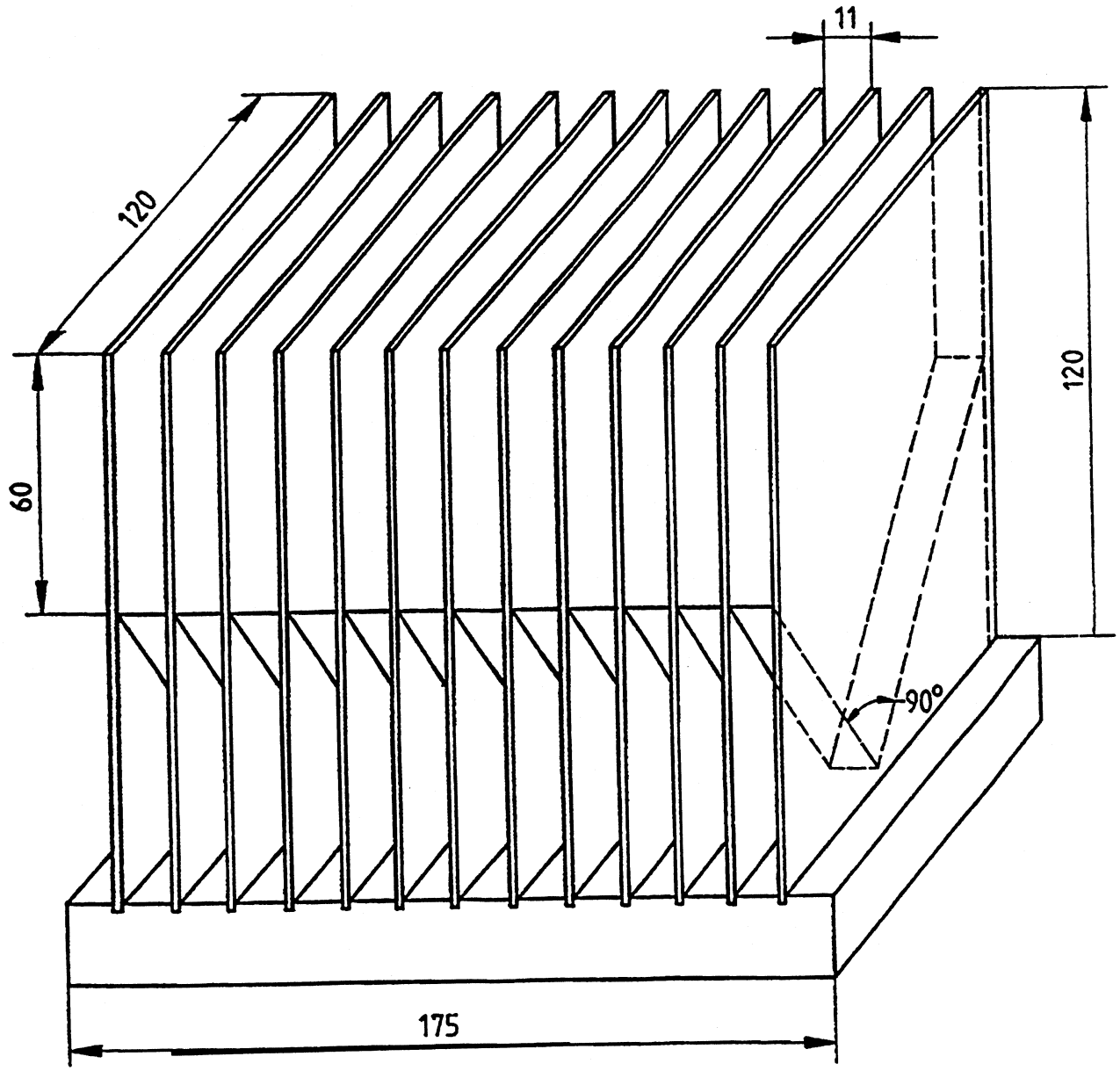
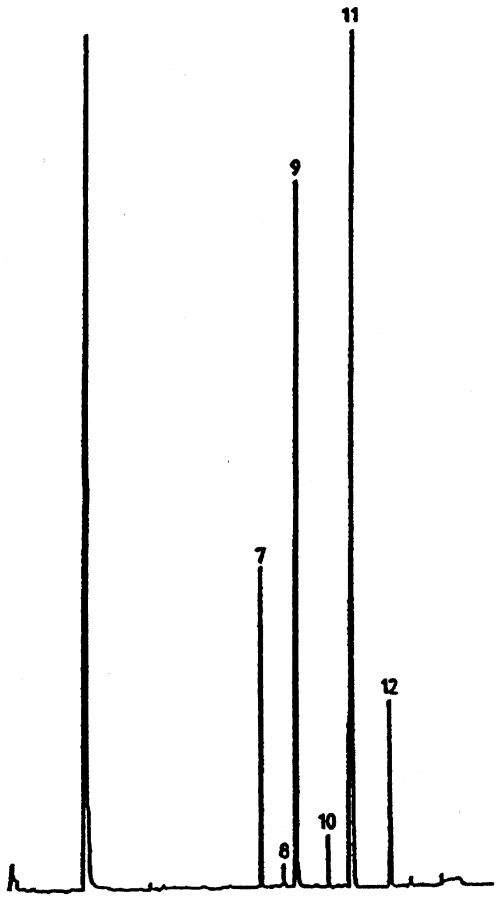
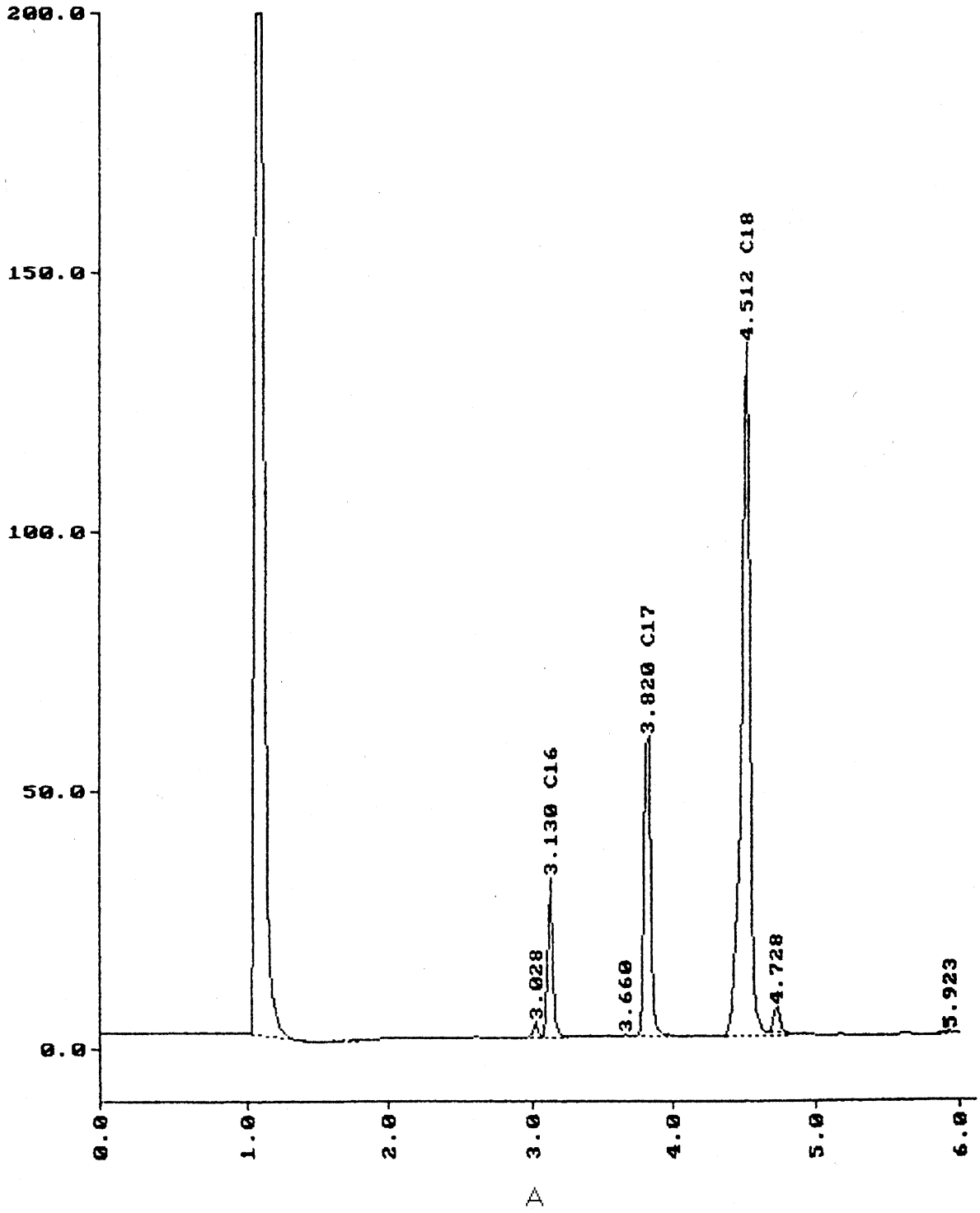


Figure E.1 — Pouch holder



7 = C16:0 8 = C16:1 9 = C17:0 10 = C18:0 11 = C18:1 12 = C18:2

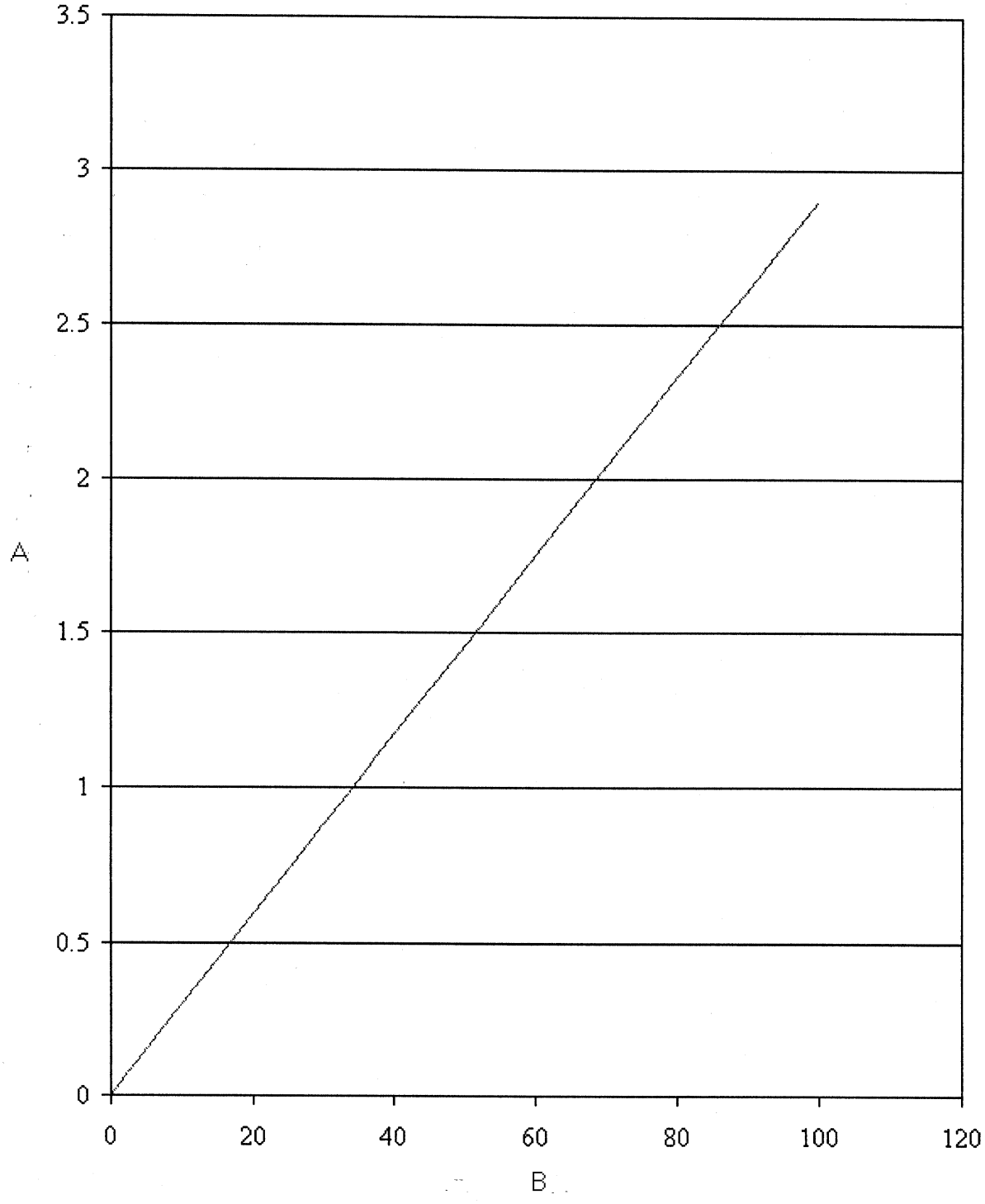
Figure E.2 — Typical chromatogram - Column 1



Key

A Retention time in minutes

Figure E.3 — Typical chromatogram - Column 2



Key

A Peak area ratio (C18 + C18)/C17

B Milligrams of olive oil

Figure E.4 — Typical calibration graph

Annex F
(informative)

Precision data

Evaluation of the results of a collaborative trial with a plastic film having a mean overall migration of 6,6 mg/dm², determined by the total immersion method, has given the following values for '*r*' and '*R*':

- repeatability, r = 2,0 mg/dm²;

- reproducibility, R = 2,9 mg/dm².

The precision data were determined from an experiment conducted in 1996 involving 11 laboratories and six replicates.

Evaluation of the results of a further collaborative trial with a plastic film having a mean overall migration of 8,3 mg/dm² and determined by the total immersion method, has given the following values for '*r*' and '*R*':

- repeatability, r = 1,8 mg/dm²;

- reproducibility, R = 3,7 mg/dm².

The precision data were determined from an experiment conducted in 1997 involving eight laboratories and six replicates.

As such they are intended to be indicative of the results to be expected using the other methods, for which the precision data are at present unavailable.

NOTE It is anticipated that more complete precision data, for all of the methods will be incorporated in a future revision of EN 1186-1.

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.

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. L372, p14.

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