BRITISH STANDARD

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Materials and articles in contact with foodstuffs — Plastics —

Part 11: Test methods for overall migration into mixtures of C-labelled synthetic triglycerides

The European Standard EN 1186-11:2002 has the status of a British Standard

ICS 67.250

National foreword

This British Standard is the official English language version of EN 1186-11:2002. It supersedes DD ENV 1186-11:1995 which is withdrawn.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.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Materials and articles in contact with foodstuffs - Plastics - Part 11: Test methods for overall migration into mixtures of C-labelled synthetic triglycerides

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 11: Méthodes d'essai pour la migration globale dans des mélanges de triglycérides synthétiques marqués au C

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 11: Prüfverfahren für die Gesamtmigration in Mischungen aus 14C-markierten synthetischen Triglyceriden

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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
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Contents

Foreword

This document EN 1186-11:2002 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 1186-11:1995.

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.

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 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

EN 1186-11 should be read in conjunction with EN 1186-1.

The annexes A, B and C are normative. The annexes D, 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 European Standard specifies test methods for the determination of the overall migration into fatty food simulants from plastics materials and articles into a mixture of ^{14}C-labelled synthetic triglycerides at temperatures above 20 °C and up to, and including, 121 °C for selected times.

These methods are suitable for plastics in the form of films and sheets, a wide range of articles or containers from which test pieces of a suitable size can be cut and containers and articles that can be filled.

The test methods described are applicable to all plastics.

2 Normative references

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

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

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

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

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

3 Method A Total immersion

3.1 Principle

WARNING The use and disposal of ^{14}C labelled substances are subject to regulations which vary from country to country. Laboratories should ensure that they comply with local legislation requirements.

NOTE 1 This method is most suitable for plastics in the form of films and sheets, but can be applied to a wide range of articles or containers from which test pieces of a suitable size can be cut.

The 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 6 and 7 of EN 1186-1:2002.

Test specimens of known mass are immersed in a mixture of ^{14}C-labelled synthetic triglycerides for the exposure time at temperatures above 20 °C and, up to and including, 121 °C, then taken from the mixture of ^{14}C-labelled synthetic triglycerides, blotted to remove synthetic triglycerides adhering to the surface and reweighed.

The specimens will usually retain absorbed mixture of ^{14}C-labelled synthetic triglycerides which are extracted and determined quantitatively by means of liquid scintillation counting.

For some plastics the soxhlet extraction process does not achieve complete recovery of the absorbed mixture of ^{14}C-labelled synthetic triglycerides. In this method the mixture of ^{14}C-labelled synthetic triglycerides that remains after soxhlet extraction is released by dissolution or combustion. The combustion method is suitable for all plastics, the dissolution method is only suitable for polymers that are soluble in a suitable solvent, e.g. tetrahydrofuran.

NOTE 2 Good sensitivity can only be achieved for samples of very low mass, e.g. for thin films. The specific radioactivity of the mixture of ^{14}C-labelled synthetic triglycerides routinely used is approximately 200 dpm/mg. In routine tests the limit of determination in liquid scintillation counting is in the order of 20 dpm per sample, for combustion, and 10 dpm per sample, for dissolution. In combustion only aliquots up to approximately 50 mg can be used. Consequently the determination limit for retained simulant is in the order of 0,1 mg to 50 mg. It is apparent that for heavy test specimens the method gives only an estimation of the retained simulant. The dissolution method, which is generally preferred, results in similar figures. A higher specific radioactivity of the simulant would improve the determination limit.

Migration into the mixture of ^{14}C-labelled synthetic triglycerides is calculated by subtracting the mass of ^{14}C-labelled synthetic triglycerides retained by the test specimen from the mass of the test specimen after removal from the ^{14}C-labelled synthetic triglycerides, and 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 the ^{14}C-labelled synthetic triglycerides during the sample handling stages, quadruplicate 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 and to experienced laboratories.

3.2 Reagents

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

3.2.1 Mixture of ^{14}C-labelled synthetic triglycerides, simulant D as specified in 5.2 of EN 1186-1:2002.

NOTE Details of suppliers can be obtained from CEN.

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

3.2.2.1 Pentane 98 % (mixed isomers) boiling point 36 °C.

WARNING Pentane is a very volatile and highly flammable solvent. Take care when using and handling this solvent to prevent contact with sources of ignition. It is not recommended for extractions with this solvent to be left unattended, particularly overnight.

NOTE Due to low boiling point of the solvent, cooled condenser water can be used to prevent undue loss of the solvent from the condenser.

3.2.2.2 Other suitable solvent.

NOTE In previous methods for determining overall migration into ^{14}C-labelled synthetic triglycerides the extraction solvent used has been 1,1,2 trichlorotrifluoroethane. For environmental reasons the use of this solvent should be avoided where possible, see 10.1 of EN 1186-1:2002. Experience has shown that this solvent although effective for most plastics requires longer periods of extraction.

3.2.3 Liquid scintillation cocktail, suitable for scintillation counting of the ^{14}C-labelled synthetic triglycerides and in which the fat simulant is soluble.

3.2.4 Diethyl ether.

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

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

3.3 Apparatus

3.3.1 Cutting slab, clean smooth glass, metal or plastics slab of sufficient area to prepare test specimens, 250 mm $\times$ 250 mm is suitable.

3.3.2 Tweezers, stainless steel, blunt nosed.

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

3.3.4 Metal templates 100 mm ± 0,2 mm $\times$ 100 mm ± 0,2 mm (square).

3.3.5 Rule, 25 mm ± 1 mm wide.

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

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

3.3.8 Specimen supports, constructed of stainless steel with cross arms attached by welding or silver soldering. Stainless steel X4 CrNi 18 10 according to EN 10088-1 or of composition, chromium 17 %, nickel 9 %, carbon 0,04 %, is suitable. Before initial use thoroughly clean the steel supports. The use of a degreasing solvent and then dilute nitric acid has been found to be suitable.

NOTE For rigid samples, supports with a single cross arm can be used.

3.3.9 Gauze, pieces of fine stainless steel gauze, with a mesh size of 1 mm have been found to be suitable, approximately 25 mm x 100 mm for insertion between the test pieces on the supports. Before initial use thoroughly clean the gauze, first with a degreasing solvent and then with dilute nitric acid.

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 the 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.

3.3.10 Glass tubes, ground neck and stoppers, for retaining the ^{14}C-labelled synthetic triglycerides and test specimens. Tubes with an internal diameter of approximately 35 mm and length in the range of 120 mm to 200 mm, with a volume of not less than 120 ml, excluding the ground neck, see 8.2 of EN 1186-1:2002, have been found to be satisfactory.

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

3.3.12 Filter paper, lint-free.

3.3.13 Anti-bumping beads.

3.3.14 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 ^{14}C-labelled synthetic triglycerides from the test specimens can be used.

3.3.15 Water bath capable of holding the flasks of soxhlet type extractors (3.3.14).

3.3.16 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.

3.3.17 Steam bath or hot plate.

3.3.18 Measuring cylinders conforming to the minimum requirement of ISO 4788, 500 ml, 250 ml and 100 ml.

3.3.19 Glass beads, 2 mm to 3 mm diameter or glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long, see 8.2 of EN 1186-1:2002.

3.3.20 Liquid scintillation counter with integrated quench correction.

3.3.21 Liquid scintillation vials to fit into the liquid scintillation counter (3.3.20).

3.3.22 Vacuum oven or vacuum desiccator.

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

3.3.24 Device for combustion of ^{14}C-labelled materials for subsequent determination of radioactivity, e.g. Schöninger flask or automatic sample oxidizer.

3.3.25 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.

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

3.3.27 Syringes, disposable plastic, with luer fitting. 1 ml or 10 ml size.

3.3.28 Luer needles, wide gauge, 80 mm $\times$ 1,2 mm.

3.3.29 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.

3.4 Preparation of test specimens

3.4.1 General

It is essential that test specimens are clean and free from surface contamination (many plastics can readily attract dust due to static charges). Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint-free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use see 9.1 of EN 1186-1:2002. Minimise handling of the samples and, where necessary, wear cotton gloves.

To ensure that test pieces are well separated and that their surfaces are freely exposed to ^{14}C labelled synthetic triglycerides during the period of the test, for thin films insert a piece of fine stainless steel gauze (3.3.9) between the test pieces or for thick samples not placed on the supports, insert glass rods between the test pieces after immersion in the ^{14}C-labelled synthetic triglycerides. Where specimen supports are used, label the supports with a tag bearing the test specimen identification.

When preparing test specimens measure the surface area according to 9.3 of EN 1186-1:2002.

3.4.2 Number of test specimens

Six test specimens are required for samples, in the form of thin films, sheet, cut sections from containers or similar articles. Eight test specimens, similar dimensionally one to another, are required for samples of articles of irregular shape.

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) two test specimens for determination of the surface area, in the case of samples of irregular shape (see 3.4.5).

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

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

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.

3.4.3 Films and sheets

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

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

3.4.4 Containers and other articles

Cut sections from the walls of the container or article to give test specimens each of area approximately 1 dm². For articles with individual areas less than 1 dm², use a number of articles to provide each test specimen.

Measure the dimensions of each test specimen to the nearest 1 mm, using the rule, see 9.3 of EN 1186-1:2002.

Calculate the area of each test specimen to the nearest 0,01 dm² and record. If necessary, cut each test specimen into smaller pieces to enable them to fit into the glass tubes (3.3.11). The test specimens or pieces are placed on the specimen supports if these are appropriate or, if the test specimens or pieces are sufficiently rigid, they can be tested unsupported.

NOTE Cutting the test specimens into smaller pieces increases the area of cut edges. If the area of the cut edges exceeds 10 % of the test specimen area, than see 8.3 of EN 1186-1:2002.

3.4.5 Articles of irregular shape

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

3.5 Procedure

3.5.1 General

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

The mixture of ^{14}C-labelled synthetic triglycerides has a melting point of 28 °C to 30 °C. To ensure homogeneity of the simulant liquefy the contents of the storage bottle before use.

3.5.2 Initial weighing of test specimens

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

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

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

3.5.2.4 If the tests described in annex A show that conditioning is necessary, but constant mass cannot be achieved within 5 days then carry out the conditioning procedure described in B.3.1 or annex C.

NOTE 1 Long conditioning periods are not satisfactory due to oxidation of the ^{14}C-labelled synthetic triglycerides which can occur upon prolonged conditioning.

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

3.5.3 Exposure to food simulant

Take six of the glass tubes (3.3.10), mark them for identification purposes. Measure 100 ml ± 5 ml of ^{14}C-labelled synthetic triglycerides (3.2.1) into each tube by measuring cylinder and stopper the tube.

NOTE 1 If the procedure described in annex C is used, it can be necessary to dry all of the ^{14}C-labelled synthetic triglycerides used for the migration test, see C.3.2.

Alternatively mark the tubes for a volume of 100 ml and fill with ^{14}C-labelled synthetic triglycerides 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 ^{14}C-labelled synthetic triglycerides are required as blank simulant, if the procedure in annex C is used. Place the six or eight tubes, and two empty tubes, in the thermostatically controlled oven or incubator (3.3.11) set at the test temperature. Leave until the ^{14}C-labelled synthetic triglycerides have attained the test temperature, using the thermometer or thermocouple to monitor the temperature. Take all tubes from the oven and place into four of the tubes containing ^{14}C-labelled synthetic triglycerides, weighed test specimens prepared as in 3.4 and conditioned if necessary. Stopper the tubes. Ensure that the test specimens are totally immersed in ^{14}C-labelled synthetic triglycerides; if they are not, then add either glass beads or glass rods (3.3.19) to raise the level of the ^{14}C-labelled synthetic triglycerides until total immersion is achieved.

NOTE 2 If the procedure in annex C is used, the ^{14}C-labelled synthetic triglycerides in the fifth tube is used as the third blank sample for Karl Fischer titrations. The ^{14}C-labelled synthetic triglycerides in the sixth tube are used to check the temperature of the triglycerides. If glass beads or glass rods have been used to raise the level of the ^{14}C-labelled synthetic triglycerides to achieve total immersion, then similar glass beads or glass rods should be added to the sixth tube.

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

NOTE 3 These two test specimens are used to check whether the sample loses mass from the evaporation of volatiles, such as water, solvents and oligomers, during the test period. If the vacuum drying procedure in annex B is applicable these test specimens are not required as during the vacuum drying volatiles are removed from the test specimens.

NOTE 4 Experience has shown that it is not necessary to check the contribution of extracts from the test specimens not exposed to the fat simulant to the level of radioactivity in liquid scintillation counting.

Replace all eight or ten tubes in the thermostatically controlled oven or incubator set at the test temperature. Carry out this part of the operation in the minimum time possible to prevent undue heat loss. Observe the temperature of the thermostatically controlled oven or incubator or the ^{14}C-labelled synthetic triglycerides (see NOTE 6) 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 ^{14}C-labelled synthetic triglycerides in the sixth tube has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

NOTE 5 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 6 For exposure times of 24 h or more it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

Take the tubes from the oven or incubator and immediately remove the test specimens from the tubes. For those specimens which have been in ^{14}C-labelled synthetic triglycerides, allow the triglycerides to drain. Remove any adhering ^{14}C-labelled synthetic triglycerides by gently pressing between filter papers (3.3.12). Repeat the pressing procedure until the filter paper shows no spots of ^{14}C-labelled synthetic triglycerides. For test specimens on supports, remove the individual test pieces from the supports to carry out this operation. Clean the supports of triglycerides by washing with the extraction solvent and replace the test pieces on them.

NOTE 7 If the procedure in annex C is followed, retain the tubes containing the triglycerides. The tubes should be capped to prevent further change in the moisture content of the triglycerides and the Karl Fischer determination of water content should be carried out as soon as possible.

3.5.4 Final weighing of test specimens

3.5.4.1 For those specimens which did not require conditioning to obtain their initial masses (see 3.5.2.2), weigh all six test specimens, i.e. the four that have been in ^{14}C-labelled synthetic triglycerides and the two that were in the empty tubes and record the mass of each test specimen.

3.5.4.2 If conditioning of the test specimens was carried out using the procedure in annex A then repeat the procedure.

3.5.4.3 If conditioning was carried out before the initial weighing using the procedure described in annex B then carry out the procedure described in B.4.

3.5.4.4 If it was decided that the procedure described in annex C was applicable to the test sample, then carry out that procedure.

3.5.4.5 If the final mass of each of the test specimens which have been in empty tubes is less than their initial mass by more than 2,0 mg, then volatile substances have been lost and adjustment may be made, see 10.4 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.

3.5.5 Extraction of absorbed ^{14}C-labelled synthetic triglycerides

NOTE Some types of plastics are known to retain some of the absorbed ^{14}C-labelled synthetic triglycerides. In these cases extraction of the ^{14}C-labelled synthetic triglycerides is incomplete and a second extraction with a more polar solvent is required, see also 10.2 of EN 1186-1:2002.

Take four flasks, 250 ml or 500 ml as appropriate to the size of the soxhlet type extractor (3.3.14) to be used for the extraction, and add sufficient extraction solvent (3.2.2) 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 (3.3.13) to control boiling.

Place the four test specimens which have been in contact with ^{14}C-labelled synthetic triglycerides into four soxhlet type extractors. Couple each soxhlet to a flask containing the extraction solvent. Using either a water bath (3.3.15) or steam bath (3.3.17), extract for a period of $7\ ^{+1}_{0}$ h, with a minimum of 6 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 almost to dryness using a rotary evaporator, or simple distillation apparatus (3.3.16). Transfer the remaining solutions containing the extracted ^{14}C-labelled synthetic triglycerides to separate liquid scintillation vials, and wash each flask with three portions of liquid scintillation cocktail. Add the three washings to the respective individual vials.

Repeat the extraction of the test specimens for an additional $7\ ^{+1}_{0}$ h, with diethyl ether (3.2.4).

If previous testing has established that all of the ^{14}C-labelled synthetic triglycerides 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 scintillation vials, using the procedure described above.

Determine the extracted ^{14}C-labelled synthetic triglycerides in both the first 7 h and the second 7 h extraction by the procedure described in 3.5.6, but retain the test specimens in the soxhlet type extractors until the extracted ^{14}C-labelled synthetic triglycerides has been determined for the second extraction. If more than 2,0 mg per test specimen is found in the second extract, then determine the retained ^{14}C-labelled synthetic triglycerides via liquid scintillation after combustion or dissolution of the test sample.

3.5.6 Determination of extracted mixture of ^{14}C - labelled synthetic triglycerides

3.5.6.1 Standard and background samples

Take five scintillation vials and add the ^{14}C-labelled synthetic triglycerides from the same batch as used for the migration test, the amounts being from 50 mg to 250 mg. Weigh to the nearest 0,1 mg and add liquid scintillation cocktail in the required amount. Take three scintillation vials and fill with cocktail only.

3.5.6.2 Liquid scintillation counting

Transfer the samples prepared according to 3.5.5 and 3.5.6.1 into the liquid scintillation counter (3.3.21) and determine the radioactivity in the sample. Make sure that the instrument has been set to the correct parameters for determination of carbon-14, including the correct quench curve.

3.5.6.3 Calculation of extracted mixture of ^{14}C-labelled synthetic triglycerides

Calculate the specific radioactivity, s_A, of the ^{14}C-labelled synthetic triglycerides with consideration of the background value as follows:

$$s_A = \frac{R_S - R_0}{w} \qquad (1)$$

where

s_A is the specific radioactivity, in disintegrations per minute per milligram;

R_S is the measuring rate, in disintegrations per minute, of the standard sample (see 3.5.6.1);

R_0 is the measuring rate, in disintegrations per minute, of the background sample (see 3.5.6.1);

w is the mass of the standard sample, in milligrams.

Calculate the amount of extracted ^{14}C-labelled synthetic triglycerides as follows:

$$m_c = \frac{R_M - R_0}{s_A \times 1000} \qquad (2)$$

where

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams;

R_M is the measuring rate, in disintegrations per minute, of the sample;

R_0 is the measuring rate, in disintegrations per minute, of the background sample as prepared in 3.5.6.1;

s_A is the specific radioactivity, in disintegrations per minute per milligram.

3.5.4.1 Determination of retained ^{14}C-labelled synthetic triglycerides

3.5.4.1.1 General

If the amount of ^{14}C-labelled synthetic triglycerides in the second extraction is less than 2 mg, but measurable, add this to the amount determined from the first extraction and record the total mass to the nearest milligram of extracted ^{14}C-labelled synthetic triglycerides for each test specimen in grams.

If the amount of ^{14}C-labelled synthetic triglycerides of more than one of the test specimens in the second extraction is greater than 2 mg for each test specimen, add the quantity of ^{14}C-labelled synthetic triglycerides from the second extraction to the quantity determined in the first extraction and determine the amount of ^{14}C-labelled synthetic triglycerides retained after combustion or dissolution of the extracted sample. The combustion method (see 3.5.6.4.2) is suitable for all plastics. The dissolution method (see 3.5.6.4.3) is only suitable for plastics that are soluble in a suitable solvent, e.g. tetrahydrofuran.

3.5.4.1.2 Combustion method

Dry the extracted test specimen and weigh as described in 3.5.4.1; cut five small pieces of about 50 mg from each test specimen, weigh to the nearest 0,1 mg and combust in a sample oxidizer (3.3.24). Determine the radioactivity in the samples obtained, as described in 3.5.6.2, and calculate the amount of ^{14}C-labelled synthetic triglycerides retained, according to 3.5.6.3, taking into account the aliquot of the extracted test specimen used for combustion. Add this quantity of ^{14}C-labelled synthetic triglycerides to that found by the extraction for each test specimen.

3.5.4.1.3 Dissolution method

Transfer the extracted test specimen into a beaker (100 ml, 250 ml or 500 ml as appropriate), dissolve in a minimum amount of tetrahydrofuran, transfer the solution into a volumetric flask (250 ml, 500 ml or 1 000 ml as appropriate) and make up to the mark. Take three aliquots of 2 ml each into scintillation vials, add liquid scintillation cocktail in the required amount and determine the radioactivity in the samples as described in 3.5.6.2. Calculate the amount of ^{14}C-labelled synthetic triglycerides retained according to 3.5.6.3.

Add this quantity of ^{14}C-labelled synthetic triglycerides to that found by the extraction for each test specimen.

3.6 Expression of results

3.6.1 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 ^{14}C-labelled synthetic triglycerides, 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 ^{14}C-labelled synthetic triglycerides, in grams (see 3.5.2.2 or 3.5.2.3 or 3.5.2.4 as appropriate);

m_b is the mass of the test specimen after contact with ^{14}C-labelled synthetic triglycerides, in grams. (see 3.5.4) or corrected mass (see equation (4)) where the loss of volatiles is greater than 2 mg per test specimen (see 3.5.4.4);

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams (see 3.5.6.3 or 3.5.6.4);

S is the surface area of the test specimen intended to come into contact with foodstuffs in square decimetres. Conventionally, if the thickness exceeds 0,5 mm, the whole of the surface area is taken into account in determining the migration value, see 9.3 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 12.3 of EN 1186-1:2002, for directions to determine whether the results are valid.

The corrected mass is calculated using the formula:

$$m_b = m_b^{\prime} + m_d \qquad (4)$$

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;

$m_b^{\prime}$ is the mass of the test specimen after contact with the ^{14}C-labelled synthetic triglycerides, 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 ^{14}C-labelled synthetic triglycerides equates to the mean loss of volatile substances from the two test specimens in the empty tubes.

If the procedure described in annex C 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 ^{14}C-labelled synthetic triglycerides, 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 C;

m_a is the initial mass of the test specimen, before contact with the ^{14}C-labelled synthetic triglycerides, in grams;

m_b is the mass of the test specimen after contact with ^{14}C-labelled synthetic triglycerides, in grams or corrected mass (see equation (4)) where the loss of volatiles is greater than 2 mg per test specimen;

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams;

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

S is the surface area of the test specimen intended to come into contact with foodstuffs in square decimetres. Conventionally, if the thickness exceeds 0,5 mm, the whole of the surface area is taken into account in determining the migration value, see 9.3 of EN 1186-1:2002.

3.6.2 Precision

See annex E.

3.7 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 12.2 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;

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

4 Method B Cell method

4.1 Principle

WARNING The use and disposal of ^{14}C labelled substances are subject to regulations which vary from country to country. Laboratories should ensure that they comply with local legislation requirements.

NOTE 1 This method is most suitable for plastics in the form of films and sheets, but is particularly applicable to those materials consisting of more than one layer or of surfaces that differ in their migration characteristics, which should be tested with the food simulant in contact only with the surface which is intended to come into contact with foodstuffs.

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 6 and 7 of EN 1186-1:2002.

Test specimens of known mass are exposed in a cell to ^{14}C-labelled synthetic triglycerides for the exposure time, at temperatures above 20 °C and up to and including 121 °C, then taken from the cell, blotted to remove triglycerides adhering to the surface, and reweighed.

The specimens will usually retain absorbed ^{14}C-labelled synthetic triglycerides which are extracted and determined quantitatively by means of liquid scintillation counting.

For some plastics the soxhlet extraction process does not achieve complete recovery of the absorbed mixture of ^{14}C-labelled synthetic triglycerides. In this method the mixture of ^{14}C-labelled synthetic triglycerides that remains after soxhlet extraction is released by dissolution or combustion. The combustion method is suitable for all plastics, the dissolution method is only suitable for polymers that are soluble in a suitable solvent, e.g. tetrahydrofuran.

NOTE 2 Good sensitivity can only be achieved for samples of very low mass, e.g. for thin films. The specific radioactivity of the mixture of ^{14}C-labelled synthetic triglycerides routinely used is approximately 200 dpm/mg. In routine tests the limit of determination in liquid scintillation counting is in the order of 20 dpm per sample, for combustion, and 10 dpm per sample, for dissolution. In combustion only aliquots up to approximately 50 mg can be used. Consequently the determination limit for retained simulant is in the order of 0,1 mg to 50 mg. It is apparent that for heavy test specimens the method gives only an estimation of the retained simulant. The dissolution method which is generally preferred, results in similar figures. A higher specific radioactivity of the simulant would improve the determination limit.

Migration into the ^{14}C-labelled synthetic triglycerides is calculated by subtracting the mass of ^{14}C-labelled synthetic triglycerides retained by the test specimen from the mass of the test specimen after removal from the ^{14}C-labelled synthetic triglycerides, 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 triglycerides 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.

4.2 Reagents

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

4.2.1 ^{14}C-labelled synthetic triglycerides, reference simulant D, as specified in 5.2 of EN 1186-1:2002.

NOTE Details of suppliers can be obtained from CEN.

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

4.2.2.1 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 pentane 98 % and ethanol 99 %.

WARNING Pentane is a very volatile and highly flammable solvent. Take care 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 Due to the low boiling points of these solvents, cooled condenser water may be required to prevent undue loss of the solvent from the condenser.

4.2.2.2 Other suitable solvent.

NOTE In previous methods for determining overall migration into ^{14}C-labelled synthetic triglycerides 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 10.1 of EN 1186-1:2002. Experience has shown that this solvent, although effective for most plastics requires longer periods of extraction.

4.2.3 Liquid scintillation cocktail, suitable for scintillation counting of ^{14}C-labelled synthetic triglycerides and in which the fat simulant is soluble.

4.2.4 Diethyl ether.

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

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

4.3 Apparatus

4.3.1 Cutting slab, clean smooth glass, metal or plastics slab of sufficient area to prepare test specimens, 250 mm $\times$ 250 mm is suitable.

4.3.2 Tweezers, stainless steel, blunt nosed.

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

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

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

4.3.6 Cells, type A as shown in Figure C.3 of EN 1186-1:2002 or equivalent cells.

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

4.3.7 Conditioning containers, for conditioning test specimens at 50 % $\pm$ 5 % relative humidity and 80 % $\pm$ 5 % relative humidity at 20 °C $\pm$ 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 $\pm$ 1 °C.

4.3.8 Glass tubes, ground neck and stoppers, for retaining the ^{14}C-labelled synthetic triglycerides. 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 8.2 of EN 1186-1:2002, have been found to be satisfactory.

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

4.3.10 Filter paper, lint-free.

4.3.11 Anti-bumping beads.

4.3.12 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 ^{14} C-labelled synthetic triglycerides from the test specimens may be used.

4.3.13 Water bath, capable of holding the flasks of soxhlet type extractors (4.3.12).

4.3.14 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.

4.3.15 Steam bath or water bath.

4.3.16 Measuring cylinders, conforming to 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.

4.3.17 Liquid scintillation counter with integrated quench correction.

4.3.18 Liquid scintillation vials to fit into the liquid scintillation counter (4.3.17).

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

4.3.20 Device for combustion of ^{14}C-labelled materials for subsequent determination of radioactivity, e.g. Schöninger flask or automatic sample oxidizer.

4.3.21 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.

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

4.3.23 Syringes, disposable plastic with luer fitting. 1 ml or 10 ml size.

4.3.24 Luer needles, wide gauge, 80 mm x 1,2 mm.

4.3.25 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.

4.4 Preparation of test specimens

4.4.1 General

NOTE The procedures described in 4.4 and 4.5 refer to the use of cell type A, when using equivalent cells (see 8.3 of EN 1186-1:2002) some modifications can be necessary.

It is essential that test specimens are clean and free from surface contamination (many plastics can readily attract dust due to static charges). Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint-free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use see 9.1 of EN 1186-1:2002. Minimise handling of the samples and, where necessary, wear cotton gloves.

When preparing test specimens measure the surface area according to 9.3 of EN 1186-1:2002.

4.4.2 Number of test specimens

Six test specimens are required for samples, in the form of thin films, sheet and flat sections cut from containers or similar articles.

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.

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

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

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.

4.4.3 Cutting test specimens

Lay the sample on the cutting slab (4.3.1) with the surface to be in contact with the ^{14}C-labelled synthetic triglycerides uppermost. Take the ring from the cell type A (4.3.6) and place on the test sample. Cut out the test specimen by cutting round the outer edge of the ring, using the cutting implement (4.3.3)

4.5 Procedure

4.5.1 General

NOTE The procedures described in 4.4 and 4.5 refer to the use of cell type A, when using equivalent cells (see 8.3 of EN 1186-1:2002) some modifications can be necessary.

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

The mixture of ^{14}C-labelled synthetic triglycerides has a melting point of 28 °C to 30 °C. To ensure homogeneity of the simulant liquefy the contents of the storage bottle before use.

4.5.2 Initial weighing of test specimens

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

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

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

4.5.2.4 If the tests described in annex A show that conditioning is necessary, but constant mass cannot be achieved within 5 days then carry out the conditioning procedure described in B.3.1 or annex C.

NOTE 1 Long conditioning periods are not satisfactory due to oxidation of the ^{14}C-labelled synthetic triglycerides which can occur upon prolonged conditioning.

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

4.5.3 Exposure to food simulant

Take four type A cells (4.3.6), mark them for identification purposes. Place in the thermostatically controlled oven or incubator (4.3.9), which is set at the test temperature and leave until the test temperature has been attained.

Take five glass tubes (4.3.8), measure 125 ml $\pm$ 5 ml of ^{14}C-labelled synthetic triglycerides (4.2.1) into each tube by measuring cylinder and stopper the tubes.

NOTE 1 If the procedure described in annex C is used, it may be necessary to dry all of the ^{14}C-labelled synthetic triglycerides used for the migration test, see C.3.2.

Alternatively mark the tubes for a volume of 125 ml and fill with ^{14}C-labelled synthetic triglycerides 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 ^{14}C-labelled synthetic triglycerides are required as blank simulant, if the procedure in annex C is used. Place the five or seven tubes, and two empty tubes, in the thermostatically controlled oven or incubator (4.3.9) set at the test temperature. Leave until the ^{14}C-labelled synthetic triglycerides have attained the test temperature, using the thermometer or thermocouple to monitor the temperature.

Remove the cells from the thermostatically controlled oven or incubator, dismantle the cells and place on the base of each cell one of the test specimens. Reassemble the cells, ensuring that the clamping screw wheel is well tightened down.

Remove four tubes containing 125 ml of ^{14}C-labelled synthetic triglycerides from the thermostatically controlled oven or incubator or refrigerator and transfer the ^{14}C-labelled synthetic triglycerides from each tube to each of the cells through the filler hole. Remove the thermometer or thermocouple from the tube and insert, if applicable see NOTE 4, in one of the cells and replace the filler plugs.

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

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

Replace the four cells and the two tubes in the thermostatically controlled oven or incubator set at the test temperature. Carry out this part of the operation in the minimum time to prevent undue heat loss from the cells and ^{14}C-labelled synthetic triglycerides. Observe the temperature of the thermostatically controlled oven or incubator or the ^{14}C-labelled synthetic triglycerides (see NOTE 4) in one of the cells and leave the cells and tubes for the selected test period, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002, after the ^{14}C-labelled synthetic triglycerides in the cell has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

NOTE 3 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 4 For exposure times of 24 h or more it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

Take the cells and tubes from the oven or incubator and immediately remove the test specimens from the cells. For those specimens which have been in ^{14}C-labelled synthetic triglycerides, allow the triglycerides to drain. Remove any adhering ^{14}C-labelled synthetic triglycerides by gently pressing between filter papers (4.3.10). Repeat the pressing procedure until the filter paper shows no spots of ^{14}C-labelled synthetic triglycerides.

If the procedure in annex C is followed, retain the tubes containing the triglycerides. Cap the tubes to prevent further change in the moisture content of the triglycerides and carry out the Karl Fischer determination of water content as soon as possible.

4.5.4 Final weighing of test specimens

4.5.4.1 For those specimens which did not require conditioning to obtain their initial masses (see 4.5.2.2), weigh all six test specimens i.e. the four that have been in ^{14}C-labelled synthetic triglycerides and the two that were in the empty tubes and record the mass of each test specimen.

4.5.4.2 If conditioning of the test specimens was carried out using the procedure in annex A (see 4.5.2.3) then repeat the procedure.

4.5.4.3 If conditioning was carried out before the initial weighing using the procedure described in annex B (see 4.5.2.4) then carry out the procedure described in B.4.

4.5.4.4 If it was decided that the procedure described in annex C (see 4.5.2.4) was applicable to the test sample, then carry out that procedure.

4.5.4.5 If the final mass of each of the test specimens which have been in empty tubes is less than their initial mass by more than 2,0 mg, then volatile substances have been lost and adjustment may be made, see 10.4 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.

4.5.5 Extraction of absorbed ^{14}C-labelled synthetic triglycerides

Take four flasks, 250 ml or 500 ml as appropriate to the size of the soxhlet type extractor (4.3.12) and add sufficient extraction solvent (4.2.2) 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 (4.3.11) to control boiling.

Place the four test specimens which have been in contact with ^{14}C-labelled synthetic triglycerides into four soxhlet type extractors. Couple each soxhlet to a flask containing the extraction solvent. Using either a water bath or steam bath (4.3.15), extract for a period of 7 $^{+1}_{0}$ h, with a minimum of 6 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 almost to dryness using a rotary evaporator, or simple distillation apparatus (4.3.14). Transfer the solutions containing the extracted ^{14}C-labelled synthetic triglycerides

to separate scintillation vials (4.3.18), and wash each flask with three portions of liquid scintillation cocktail. Add the three washings to the respective individual vials.

NOTE Some types of plastics are known to retain some of the absorbed ^{14}C-labelled synthetic triglycerides. In these cases extraction of the ^{14}C-labelled synthetic triglycerides is incomplete and a second extraction with a more polar solvent is required, see also 10.2 of EN 1186-1:2002.

Repeat the extraction of the test specimens for an additional $7\,^{+1}_{0}$ h, with diethyl ether (4.2.4).

If previous testing has established that all of the ^{14}C-labelled synthetic triglycerides 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 scintillation vials, using the procedure described above.

Determine the extracted ^{14}C-labelled synthetic triglycerides in both the first 7 h and the second 7 h extractions by the procedure described in 4.5.6, but retain the test specimens in the soxhlet type extractors until the extracted ^{14}C-labelled synthetic triglycerides has been determined for the second 7 h extractions. If more than 5 mg per test specimen is found in the second extract, then determine the retained ^{14}C-labelled synthetic triglycerides via liquid scintillation counting after combustion or dissolution of the test specimens.

4.5.6 Determination of extracted mixture of ^{14}C-labelled synthetic triglycerides

4.5.6.1 Standard and background samples

Take five scintillation vials (4.3.18) and add ^{14}C-labelled synthetic triglycerides from the same batch as used for the migration test, the amounts being from 50 mg to 250 mg. Weigh to the nearest 0,1 mg and add liquid scintillation cocktail in the required amount. Take three vials and fill with cocktail only.

4.5.6.2 Liquid scintillation counting

Transfer the samples prepared according to 4.5.5 and 4.5.6.1 into the liquid scintillation counter (4.3.17) and determine the radioactivity in the sample. Make sure that the instrument has been set to the correct parameters for determination of carbon-14, including the correct quench curve.

4.5.6.3 Calculation of extracted mixture of ^{14}C-labelled synthetic triglycerides

Calculate the specific radioactivity *sA* of the ^{14}C-labelled synthetic triglycerides with consideration of the background value

$$sA = \frac{R_S - R_O}{w} \qquad (6)$$

where

sA is the specific radioactivity, in disintegrations per minute per milligram;

R_s is the measuring rate, in disintegrations per minute, of the standard sample (4.5.6.1);

R_O is the measuring rate, in disintegrations per minute, of the background sample (4.5.6.1);

w is the mass of the standard sample, in milligrams;

Calculate the amount of extracted ^{14}C-labelled synthetic triglycerides as follows:

$$m_c = \frac{R_M - R_o}{sA \times 1000} \qquad (7)$$

where

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams;

R_M is the measuring rate, in disintegrations per minute, of the sample;

R_O is the measuring rate, in disintegrations per minute, of the background sample as prepared in 4.5.6.1.

sA is the specific radioactivity, in disintegrations per minute per milligram.

4.5.6.4 Determination of retained ^{14}C-labelled synthetic triglycerides

4.5.6.4.1 General

If the amount of ^{14}C-labelled synthetic triglycerides in the second extraction is less than 5 mg, but measurable, add this to the amount determined from the first extraction and record the total mass to the nearest milligram of extracted ^{14}C-labelled synthetic triglycerides for each test specimen in grams.

If the amount of ^{14}C-labelled synthetic triglycerides of more than one of the test specimens in the second extraction is greater than 5 mg for each test specimen, add the quantity of ^{14}C-labelled synthetic triglycerides from the second extraction to the quantity determined in the first extraction and determine the amount of ^{14}C-labelled synthetic triglycerides retained after combustion or dissolution of the extracted sample. The combustion method (see 4.5.6.4.2) is suitable for all plastics. The dissolution method (see 4.5.6.4.3) is only suitable for plastics that are soluble in a suitable solvent, e.g. tetrahydrofuran.

4.5.6.4.2 Combustion method

Dry the extracted test specimen and weigh as described in 4.5.4.1; cut five small pieces of about 50 mg from each test specimen, weigh to the nearest 0,1 mg and combust in a sample oxidizer (4.3.20). Determine the radioactivity in the samples obtained, as described in 4.5.6.2, and calculate the amount of ^{14}C-labelled synthetic triglycerides retained, according to 4.5.6.3, taking into account the aliquot of the extracted test specimen used for combustion. Add this quantity of mixture of ^{14}C-labelled synthetic triglycerides to that found by the extraction for each test specimen.

4.5.6.4.3 Dissolution method

Transfer the extracted test specimen into a beaker (100 ml, 250 ml or 500 ml as appropriate), dissolve in a minimum amount of tetrahydrofuran, transfer the solution into a volumetric flask (250 ml, 500 ml or 1 000 ml as appropriate) and make up to the mark. Take three aliquots of 2 ml each into scintillation vials, add liquid scintillation cocktail in the required amount and determine the radioactivity in the samples as described in 4.5.6.2. Calculate the amount of ^{14}C-labelled synthetic triglycerides retained according to 4.5.6.3. Add this quantity of ^{14}C-labelled synthetic triglycerides to that found by the extraction for each test specimen.

4.6 Expression of results

4.6.1 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:

$$M = \frac{m_a - (m_b - m_c) \times 1000}{S} \qquad (8)$$

where

M is the overall migration into ^{14}C-labelled synthetic triglycerides, in grams per square decimetre of 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 ^{14}C-labelled synthetic triglycerides, in grams (see 4.5.2.2 or 4.5.2.3 or 4.5.2.4 as appropriate);

m_b is the mass of the test specimen after contact with ^{14}C-labelled synthetic triglycerides in grams. (see 4.5.4) or corrected mass (see formula 8), where the loss of volatiles is greater than 5 mg per test specimen (see 4.5.4.4);

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams (see 4.5.6.3 or 4.5.6.4);

S is the surface area of the test specimen in contact with the food simulant in the cell, that is 2,5 dm² in the standard cell. See 9.4 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 milligram per square decimetre. See 12.3 of EN 1186-1:2002, for directions to determine whether the results are valid.

The corrected mass is calculated using the formula:

$$m_b = m_b' + m_d \qquad (9)$$

where

m_b is the corrected loss in mass of the test specimens in the 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 ^{14}C-labelled synthetic triglycerides, in grams.

NOTE This adjustment assumes that the same quantity of volatiles is lost from a test specimen in contact with ^{14}C-labelled synthetic triglycerides, as is lost from a test specimen in an empty tube.

If the procedure described in annex C 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:

$$M_D = \frac{[m_a - (m_b - m_c + M_W)] \times 1000}{S} \qquad (10)$$

where

M_D is the overall migration into ^{14}C-labelled synthetic triglycerides, 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 C;

m_a is the initial mass of the test specimen, before contact with the ^{14}C-labelled synthetic triglycerides, in grams;

m_b is the mass of the test specimen after contact with ^{14}C-labelled synthetic triglycerides, in grams. or corrected mass (see equation (4)) where the loss of volatiles is greater than 2 mg per test specimen;

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams;

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 intended to come into contact with foodstuffs in square decimetres. See 8.4 of EN 1186-1:2002. Conventionally, if the thickness exceeds 0,5 mm, the whole of the surface area is taken into account in determining the migration value, see 9.3 of EN 1186-1:2002.

4.6.2 Precision

See annex E.

4.7 Test report

Where the plastics 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 12.2 of EN 1186-1:2002).

The test report shall include the following:

a) reference to this European Standard and the Part of it used in 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 standard procedure and reasons therefore;

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

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

g) relevant comments on the test results; if an equivalent cell was used, supply the details;

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

5 Method C Pouch

5.1 Principle

WARNING The use and disposal of ^{14}C labelled substances are subject to regulations which vary from country to country. Laboratories should ensure that they comply with local legislation requirements.

NOTE 1 This method is most suitable for plastics in the form of films and sheets which are sealable by heat or pressure, but is particularly applicable to those materials consisting of more than one layer, which have to be tested with the food sumulant in contact only with the surface which is intended to come into contact with the foodstuffs.

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 6 and 7 of EN 1186-1:2002.

Test specimens of known mass and in the form of pouches are filled with ^{14}C-labelled synthetic triglycerides for the exposure time, at temperatures above 20 °C and up to and including 121 °C, then the ^{14}C-labelled synthetic triglycerides are removed, the pouches cut open and the ^{14}C-labelled synthetic triglycerides adhering to the surface removed by blotting. The cut portion of each pouch is then reweighed.

The specimens usually retain absorbed ^{14}C-labelled synthetic triglycerides which is extracted and determined quantitatively by means of liquid scintillation counting.

For some plastics the soxhlet extraction process does not achieve complete recovery of the absorbed mixture of ^{14}C-labelled synthetic triglycerides. In this method the mixture of ^{14}C-labelled synthetic triglycerides that remains after soxhlet extraction is released by dissolution or combustion. The combustion method is suitable for all plastics, the dissolution method is only suitable for polymers that are soluble in a suitable solvent, e.g. tetrahydrofuran.

NOTE 2 Good sensitivity can only be achieved for samples of very low mass, e.g. for thin films. The specific radioactivity of the mixture of ^{14}C-labelled synthetic triglycerides routinely used is approximately 200 dpm/mg. In routine tests the limit of determination in liquid scintillation counting is in the order of 20 dpm per sample, for combustion, and 10 dpm per sample, for dissolution. In combustion only aliquots up to approximately 50 mg can be used. Consequently the determination limit for retained simulant is in the order of 0,1 mg to 50 mg. It is apparent that for heavy test specimens the method gives only an estimation of the retained simulant. The dissolution method which is generally preferred, results in similar figures. A higher specific radioactivity of the simulant would improve the determination limit.

Migration into the ^{14}C-labelled synthetic triglycerides is calculated by subtracting the mass of ^{14}C-labelled synthetic triglycerides retained by the test specimen from the mass of the test specimen after removal from the ^{14}C-labelled synthetic triglycerides, 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 triglycerides 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.

5.2 Reagents

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

5.2.1 Mixture of ^{14}C-labelled synthetic triglycerides, simulant D as specified in 5.2 of EN 1186-1:2002.

NOTE Details of suppliers can be obtained from CEN.

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

5.2.2.1 Pentane 98 % (mixed isomers) boiling point 36 °C

WARNING Pentane is a very volatile and highly flammable solvent. Take care when using and handling this solvent to prevent contact with sources of ignition. It is not recommended for extractions with this solvent to be left unattended, particularly overnight.

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

5.2.2.2 Other suitable solvent.

NOTE In previous methods for determining overall migration in ^{14}C-labelled synthetic triglycerides the extraction solvent used has been 1,1,2 trichlorotrifluoroethane. For environmental reasons the use of this solvent should be avoided where possible, see 10.1 of EN 1186-1:2002. Experience has shown that this solvent although effective for most plastics requires longer periods of extraction.

5.2.3 Liquid scintillation cocktail, suitable for scintillation counting of ^{14}C-labelled synthetic triglycerides and in which the fat simulant is soluble.

5.2.4 Diethyl ether.

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

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

5.3 Apparatus

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

5.3.2 Tweezers, stainless steel, blunt nosed.

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

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

5.3.5 Metal template 120 mm × 120 mm × 1 mm.

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

5.3.7 Pouch holder as shown in Figure D.1 constructed from aluminium or other suitable material or an equivalent holder, plus clips to secure corners of pouches.

5.3.8 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 the 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.

5.3.9 Glass tubes, ground neck and stoppers, for retaining the ^{14}C-labelled synthetic triglycerides and test specimens. Tubes with an internal diameter of approximately 35 mm and length in the range 120 mm to 200 mm with a volume of not less than 120 ml, excluding the ground neck (see 8.2 of EN 1186-1:2002).

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

5.3.11 Filter paper, lint-free.

5.3.12 Anti-bumping beads.

5.3.13 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 the absorbed ^{14}C-labelled synthetic triglycerides from the test specimens can be used.

5.3.14 Water bath capable of holding the flasks of soxhlet type extractors (5.3.13).

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

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

5.3.16 Steam bath or hot plate.

5.3.17 Measuring cylinders, conforming to the minimum requirements of ISO 4788, 500 ml, 250 ml and 100 ml.

5.3.18 Pipettes, conforming to the minimum requirements of ISO 648, 100 ml.

5.3.19 Glass rods, 2 mm to 3 mm in diameter, for use as spacers between test pieces during solvent extraction, see 8.2 of EN 1186-1:2002.

5.3.20 Heat or pressure sealing device, for use in forming pouches.

5.3.21 Liquid scintillation counter with integrated quench correction.

5.3.22 Liquid scintillation vials to fit into the liquid scintillation counter.

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

5.3.24 Device for combustion of ^{14}C-labelled materials for subsequent determination of radioactivity, e.g. Schöninger flask or automatic sample oxidizer.

5.3.25 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.

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

5.3.27 Syringes, disposable plastic with luer fitting. 1 ml or 10 ml size.

5.3.28 Luer needles, wide gauge, 80 mm × 1,2 mm.

5.3.29 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.

5.4 Preparation of test specimens

5.4.1 General

It is essential that test specimens are clean and free from surface contamination (many plastics can readily attract dust due to static charges). Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint-free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use see 9.1 of EN 1186-1:2002. Minimise handling of the samples and, where necessary, wear cotton gloves.

5.4.2 Number of test specimens

Six 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;

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

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

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.

5.4.3 Cutting and preparation of test specimens

Lay the sample on the cutting slab (5.3.1) with the surface to be in contact with the ^{14}C-labelled synthetic triglycerides uppermost and cut test pieces using the 120 mm × 120 mm template (5.3.5). 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 ^{14}C-labelled synthetic triglycerides facing. Using the heat or pressure sealer (5.3.20), 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 ^{14}C-labelled synthetic triglycerides, to the nearest 0,01 dm². This shall be approximately 2 dm². Using the cutting implement, remove excess film from the sealed area (to reduce the area of film not directly exposed to the ^{14}C-labelled synthetic triglycerides) 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.

5.5 Procedure

5.5.1 General

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

The mixture of ^{14}C-labelled synthetic triglycerides has a melting point of 28 °C to 30 °C. To ensure homogeneity of the simulant liquefy the contents of the storage bottle before use.

5.5.2 Initial weighing of test specimens

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

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

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

5.5.2.4 If the tests described in annex A show that conditioning is necessary, but constant mass cannot be achieved within 5 days then carry out the conditioning procedure described in B.3.1 or annex C.

NOTE 1 Long conditioning periods are not satisfactory due to oxidation of the ^{14}C-labelled synthetic triglycerides which may occur upon prolonged conditioning.

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

5.5.3 Exposure to food simulant

Take five of the glass tubes (5.3.9), measure 100 ml ± 5 ml of ^{14}C-labelled synthetic triglycerides (5.2.1) into each tube by measuring cylinder (5.3.17) and stopper the tube.

NOTE 1 The pouch holder (5.3.7) should be cleaned before use, if necessary, using solvents, such as acetone and or detergents. For difficult to remove ^{14}C-labelled synthetic triglycerides, propriety solvent mixtures should be used.

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

NOTE 2 If the procedure described in annex C is used, it can be necessary to dry all of the ^{14}C-labelled synthetic triglycerides used for the migration test, see C.3.2.

Alternatively mark the tubes for a volume of 100 ml and fill with ^{14}C-labelled synthetic triglycerides to the mark. Two extra tubes with a minimum of 50 ml of ^{14}C-labelled synthetic triglycerides are required as blank simulant, if the procedure in annex C is used. Place the five or seven tubes and the pouch holder, in the thermostatically controlled oven or incubator (5.3.10) 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 ^{14}C-labelled synthetic triglycerides have 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 ^{14}C-labelled synthetic triglycerides from the oven and into four of the test specimen pouches pipette sufficient ^{14}C-labelled synthetic triglycerides 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 ^{14}C-labelled synthetic triglycerides in the fifth tube is used if the procedure in annex C 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 loses mass from the evaporation of volatiles, such as water, solvents and oligomers, during the test period. If the vacuum drying procedure in annex B is applicable these test specimens are not required as during the vacuum drying volatiles are 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. Carry out this part of the operation in the minimum time possible to prevent undue heat loss. Observe the temperature of the thermostatically controlled oven or incubator or the ^{14}C-labelled synthetic triglycerides (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 ^{14}C-labelled synthetic triglycerides 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 airbath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

Take the pouch holder and the tubes containing ^{14}C-labelled synthetic triglycerides 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 ^{14}C-labelled synthetic triglycerides from each test specimen and wipe any excess from the outside with filter paper (5.3.11). Take each of the four pouches in turn, lay on the cutting slab (5.3.1) and using the cutting implement (5.3.3) 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 ^{14}C-labelled synthetic triglycerides by gently pressing between filter papers (5.3.11). Repeat the pressing procedure until the filter paper shows no spots of ^{14}C-labelled synthetic triglycerides.

If the procedure in annex C is followed, retain the tubes containing the triglycerides. Cap the tubes to prevent further change in the moisture content of the triglycerides and carry out the Karl Fischer determination of water content as soon as possible.

5.5.4 Final weighing of test specimens

5.5.4.1 For those specimens which did not require conditioning to obtain their initial masses (see 5.5.2.2), weigh all six test specimens i.e. the four that have been in ^{14}C-labelled synthetic triglycerides and the two that were in the empty tubes and record the mass of each test specimen.

5.5.4.2 If conditioning of the test specimens was carried out using the procedure in annex A (see 5.5.2.3) then repeat the procedure.

5.5.4.3 If conditioning was carried out before the initial weighing using the procedure described in annex B (see 5.5.2.4) then carry out the procedure described in B.4.

5.5.4.4 If it was decided that the procedure described in annex C (see 5.5.2.4) was applicable to the test sample, then carry out that procedure.

5.5.4.5 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 10.4 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.

5.5.5 Extraction of absorbed ^{14}C-labelled synthetic triglycerides

Take four flasks, 250 ml or 500 ml, as appropriate to the size of the soxhlet type extractor (5.3.13) to be used for the extraction, and add sufficient extraction solvent (5.2.2) 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 (5.3.12) to control boiling.

Place the four test specimens which have been in contact with ^{14}C-labelled synthetic triglycerides into four soxhlet type extractors. Couple each soxhlet to a flask containing the extraction solvent. Using either a water bath or steam bath (5.3.16), extract for a period of 7 $^{+1}_{0}$ h, with a minimum of 6 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 almost to dryness using a rotary evaporator, or simple distillation apparatus (5.3.15). Transfer the remaining solutions containing the extracted ^{14}C labelled synthetic triglycerides to separate scintillation vials (5.3.22), and wash each flask with three portions of liquid scintillation cocktail. Add the three washings to the respective individual vials

NOTE 1 Oxidation of the ^{14}C-labelled synthetic triglycerides should 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 ^{14}C-labelled synthetic triglycerides to oxygen should be limited.

NOTE 2 Some types of plastics are known to retain some of the absorbed ^{14}C-labelled synthetic triglycerides. In these cases extraction of the ^{14}C-labelled synthetic triglycerides is incomplete and a second extraction with a more polar solvent is required, see also 10.2 of EN 1186-1:2002.

Repeat the extraction of the test specimens for an additional 7 $^{+1}_{0}$ h, with diethyl ether (5.2.4)

If previous testing has established that all of the ^{14}C-labelled synthetic triglycerides are extracted from the test specimens during the first 7 h extraction then the second 7 h extraction may be omitted.

Isolate the residues in scintillation vials, using the procedure described above.

Determine the extracted ^{14}C-labelled synthetic triglycerides in both the first 7 h and the second 7 h extraction by the procedure described in 5.5.6, but retain the test specimens in the soxhlet type extractors until the extracted ^{14}C-labelled synthetic triglycerides has been determined for the second extraction. If more than 4 mg per test specimen is found in the second extract, then determine the

retained ^{14}C-labelled synthetic triglycerides via liquid scintillation counting after combustion or dissolution of the test specimen.

5.5.6 Determination of extracted mixture of ^{14}C-labelled synthetic triglycerides

5.5.6.1 Standard and background samples

Take five scintillation vials and add ^{14}C-labelled synthetic triglycerides from the same batch as used for the migration test, the amounts being from 50 mg to 250 mg. Weigh to the nearest 0,1 mg and add liquid scintillation cocktail in the required amount. Take three scintillation vials and fill with cocktail only.

5.5.6.2 Liquid scintillation counting

Transfer the samples prepared according to 5.5.5 and 5.5.6.1 into the liquid scintillation counter (5.3.21) and determine the radioactivity in the sample. Make sure that the instrument has been set to the correct parameters for determination of carbon-14, including the correct quench curve.

5.5.6.3 Calculation of extracted ^{14}C-labelled synthetic triglycerides

Calculate the specific radioactivity, *sA*, of the ^{14}C-labelled synthetic triglycerides with consideration of the background value.

$$sA = \frac{R_S - R_O}{w} \qquad (11)$$

where

sA is the specific radioactivity, in disintegrations per minute per milligram;

R_S is the measuring rate, in disintegrations per minute, of the standard sample (5.5.6.1);

R_O is the measuring rate, in disintegrations per minute, of the background sample (5.5.6.1);

w is the mass of the standard sample, in milligrams.

Calculate the amount of extracted ^{14}C-labelled synthetic triglycerides as follows:

$$m_c = \frac{R_M - R_O}{sA \times 1000} \qquad (12)$$

where

m_c is the mass of mixture of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams;

R_M is the measuring rate, in disintegrations per minute, of the sample (5.5.6.2);

R_O is the measuring rate, in disintegrations per minute, of the background sample as prepared in 5.5.6.1;

sA is the specific radioactivity, in disintegrations per minute per milligram.

5.5.6.4 Determination of retained ^{14}C-labelled synthetic triglycerides

5.5.6.4.1 General

If the amount of mixture of ^{14}C-labelled synthetic triglycerides in the second extraction is less than 4 mg, but measurable, add this to the amount determined from the first extraction and record the total mass to the nearest milligram of extracted ^{14}C-labelled synthetic triglycerides for each test specimen in grams.

If the amount of mixture of ^{14}C-labelled synthetic triglycerides of more than one of the test specimens in the second extraction is greater than 4 mg for each test specimen, add the quantity of ^{14} C-labelled synthetic triglycerides in the second extraction to the quantity determined in the first extraction and determine the amount mixture of ^{14}C-labelled synthetic triglyceridesretained after combustion or dissolution of the extracted sample. The combustion method (see 5.5.6.4.2) is suitable for all plastics. The dissolution method (see 5.5.6.4.3) is only suitable for plastics that are soluble in a suitable solvent, e.g. tetrahydrofuran.

5.5.6.4.2 Combustion method

Dry the extracted test specimen and weigh as described in 5.5.4.1; cut five small pieces of about 50 mg from each test specimen, weigh to the nearest 0,1 mg and combust in a sample oxidizer (5.3.24). Determine the radioactivity in the samples obtained, as described in 5.5.6.2, and calculate the amount of ^{14}C-labelled synthetic triglycerides retained, according to 5.5.6.3, taking into account the aliquot of the extracted test specimen used for combustion. Add this quantity of ^{14}C-labelled synthetic triglycerides to that found by the extraction for each test specimen.

5.5.6.4.3 Dissolution method

Transfer the extracted test specimen into a beaker (100 ml, 250 ml or 500 ml as appropriate), dissolve in a minimum amount of tetrahydrofuran, transfer the solution into a volumetric flask (250 ml, 500 ml or 1 000 ml as appropriate) and make up to the mark. Take three aliquots of 2 ml each into scintillation vials, add liquid scintillation cocktail in the required amount and determine the radioactivity in the samples as described in 5.5.6.2. Calculate the amount of ^{14}C-labelled synthetic triglycerides retained according to 5.5.6.3. Add this quantity of ^{14}C-labelled synthetic triglycerides to that found by the extraction for each test specimen.

5.6 Expression of results

5.6.1 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:

$$M = \frac{[\, m_a - (m_b - m_c)] \times 1000}{S} \qquad (13)$$

where

M is the overall migration into ^{14}C-labelled synthetic triglycerides in milligrams per square decimetre of 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 ^{14}C-labelled synthetic triglycerides, in grams (see 5.5.2.2 or 5.5.2.3 or 5.5.2.4 as appropriate);

m_b is the mass of the test specimen after contact with ^{14}C-labelled synthetic triglycerides, in grams (see 5.5.4) or corrected mass (see formula 14) where the loss of volatiles is greater than 4 mg per test specimen (see 5.5.4.4);

m_c is the mass of mixture of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams (see 5.5.6.3 or 5.5.6.4);

S is the surface area of the test specimen in contact with the food simulant, in square decimetres. See 9.5 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 milligram per square decimetre. See 12.3 of EN 1186-1:2002, for directions to determine whether the results are valid.

The corrected mass is calculated using the formula:

$$m_b = m_b' + m_d \qquad (14)$$

where

m_b is the corrected loss in mass of the test specimens in the 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 ^{14}C-labelled synthetic triglycerides, in grams.

NOTE This adjustment assumes that the same quantity of volatiles is lost from a test specimen exposed to ^{14}C-labelled synthetic triglycerides, as is lost from a test specimen not exposed to ^{14}C-labelled synthetic triglycerides.

If the procedure described in annex C 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:

$$M_D = \frac{[m_a - (m_b - m_c + M_W)] \times 1000}{S} \qquad (15)$$

where

M_D is the overall migration into ^{14}C-labelled synthetic triglycerides, 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 C;

m_a is the initial mass of the test specimen, before contact with the ^{14}C-labelled synthetic triglycerides, in grams;

m_b is the mass of the test specimen after contact with ^{14}C-labelled synthetic triglycerides, in grams, or corrected mass (see formula (14)) where the loss of volatiles is greater than 2 mg per test specimen;

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams;

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

S is the surface area of the test specimen intended to come into contact with foodstuffs in square decimetres. See 9.5 of EN 1186-1:2002.

5.6.2 Precision

See annex E.

5.7 Test report

Where the plastics 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 12.2 of EN 1186-1:2002).

The test report shall include the following:

a) reference to this European Standard and 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 therefore;

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

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

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

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

6 Method D - Article filling method

6.1 Principle

WARNING: The use and disposal of ^{14}C labelled substances are subject to regulations which vary from country to country. Laboratories should ensure that they comply with local legislation requirements.

NOTE 1 This method is most suitable for plastics in the form of containers and articles that can be filled. Testing samples by this method enables testing of non-homogeneous articles providing that they are not too large.

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 6 and 7 of EN 1186-1:2002.

Test specimens of known mass are filled with mixture of ^{14}C-labelled synthetic triglycerides for the exposure time, at temperatures above 20 °C and up to and including 121 °C, then emptied and blotted to remove oil adhering to the surface, and reweighed.

The specimens usually retain absorbed mixture of ^{14}C-labelled synthetic triglycerides which are extracted and determined quantitatively by means of liquid scintillation counting.

For some plastics the soxhlet extraction process does not achieve complete recovery of the absorbed mixture of ^{14}C-labelled synthetic triglycerides. In this method the mixture of ^{14}C-labelled synthetic triglycerides that remains after soxhlet extraction is released by dissolution or combustion. The

combustion method is suitable for all plastics, the dissolution method is only suitable for polymers that are soluble in a suitable solvent, e.g. tetrahydrofuran.

NOTE 2 Good sensitivity can only be achieved for samples of very low mass, e.g. for thin films. The specific radioactivity of the mixture of ^{14}C-labelled synthetic triglycerides routinely used is approximately 200 dpm/mg. In routine tests the limit of determination in liquid scintillation counting is in the order of 20 dpm per sample, for combustion, and 10 dpm per sample, for dissolution. In combustion only aliquots up to approximately 50 mg can be used. Consequently the determination limit for retained simulant is in the order of 0,1 mg to 50 mg. It is apparent that for heavy test specimens the method gives only an estimation of the retained simulant. The dissolution method which is generally preferred, results in similar figures. A higher specific radioactivity of the simulant would improve the determination limit.

Migration into the mixture of ^{14}C-labelled synthetic triglycerides is calculated by subtracting the mass of mixture of ^{14}C-labelled synthetic triglycerides retained by the test specimen from the mass of the test specimen after removal from the mixture of ^{14}C-labelled synthetic triglycerides, 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 triglycerides 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.

6.2 Reagents

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

6.2.1 mixture of ^{14}C-labelled synthetic triglycerides, Simulant D, as specified in 5.2 of EN 1186-1:2002.

NOTE Details of suppliers can be obtained from CEN.

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

6.2.2.1 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 %.

WARNING Pentane is a very volatile and highly flammable solvent. Take care 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 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.

6.2.2.2 Other suitable solvent.

NOTE In previous methods for determining overall migration into ^{14}C-labelled synthetic triglycerides 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 10.1 of EN 1186-1:2002. Experience has shown that this solvent, although effective for most plastics requires longer periods of extraction.

6.2.3 Diethyl ether.

6.2.4 Liquid scintillation cocktail, suitable for scintillation counting of ^{14}C-labelled synthetic triglycerides and in which the fat simulant is soluble.

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

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

6.3 Apparatus

6.3.1 Tweezers, stainless steel, blunt nosed.

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

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

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

6.3.5 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.

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

6.3.7 Filter paper, lint-free.

6.3.8 Chromatography tank or any other airtight container for test sample storage.

6.3.9 Glass rods or metal gauze for use as spacers between test pieces during solvent extraction.

6.3.10 Antibumping beads.

6.3.11 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 ^{14}C-labelled synthetic triglycerides from the test specimens can be used.

6.3.12 Water bath, capable of holding the flasks of soxhlet type extractors (6.3.11)

6.3.13 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.

6.3.14 Steam bath or water bath.

6.3.15 Measuring cylinders, conforming to 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.

6.3.16 Pipettes, conforming to the minimum requirements of ISO 648, 5 ml and 10 ml.

6.3.17 Lint-free cloth.

6.3.18 Liquid scintillation counter with integrated quench correction.

6.3.19 Liquid scintillation vials to fit into the liquid scintillation counter (6.3.18).

6.3.20 Device for combustion of ^{14}C-labelled materials for subsequent determination of radioactivity, e.g. Schöninger flask or automatic sample oxidizer.

6.3.21 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.

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

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

6.3.24 Syringes, disposable plastic with luer fitting. 1 ml or 10 ml size.

6.3.25 Luer needles, wide gauge, 80 mm x 1,2 mm.

6.3.26 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/dm² plastic). An automated volumetric or coulometric instrument shall be used. Manual titration procedures do not give the required accuracy or precision.

6.4 Preparation of test specimens

6.4.1 General

It is essential that test specimens are clean and free from surface contamination (many plastics can readily attract dust due to static charges). Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint-free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use see 9.1 of EN 1186-1:2002. Minimize handling of the samples and, where necessary, wear cotton gloves.

6.4.2 Number of test specimens

Eight test specimens are required for samples, in the form in which they are intended to be used.

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) two test specimens for determination of the surface area.

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

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

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.

6.4.3 Cutting test specimens

If the article is large, to avoid handling and weighing problems or using excessive amounts of ^{14}C-labelled synthetic triglycerides it may be preferable to cut it so that the surface of the test specimen in contact with the ^{14}C-labelled synthetic triglycerides does not exceed 3 dm².

If this is done, take care that ^{14}C-labelled synthetic triglycerides does not come into contact with the cut edges of the test specimen. It is important that the area in contact with the ^{14}C-labelled synthetic triglycerides is determined as it is incorporated into the calculation later.

Scratch lightly an identification code on the external surface of each test specimen.

NOTE If only part of a specimen is tested, this part should be representative of the whole in terms of composition and wall or layer thickness.

6.5 Procedure

6.5.1 General

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

The mixture of ^{14}C-labelled synthetic triglycerides has a melting point of 28 °C to 30 °C. To ensure homogeneity of the simulant liquefy the contents of the storage bottle before use.

6.5.2 Initial weighing of test specimens

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

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

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

6.5.2.4 If the tests described in annex A show that conditioning is necessary, but constant mass cannot be achieved within 5 days then carry out the conditioning procedure described in B.3.1 or annex C.

NOTE 1 Long conditioning periods are not satisfactory due to oxidation of the ^{14}C-labelled synthetic triglycerides which can occur upon prolonged conditioning.

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

6.5.3 Exposure to food simulant

Place a sufficient volume of ^{14}C-labelled synthetic triglycerides in a beaker in the thermostatically controlled oven or incubator (6.3.6) which is set at the test temperature and leave until the test temperature has been attained.

Place each test specimen on a clean, oil free surface and fill four specimens with ^{14}C-labelled synthetic triglycerides to within 0,5 cm of the top. If the container has a specified nominal volume of contents, see 9.2 of EN 1186-1:2002. Place into one of the filled test specimens a thermometer or thermocouple.

If the procedure in annex C is used place sufficient ^{14}C-labelled synthetic triglycerides, as a blank sample for Karl Fischer titrations, in a tube and stopper the tube.

NOTE 1 If the procedure described in annex C is used, it can be necessary to dry all of the ^{14}C-labelled synthetic triglycerides used for the migration test, see C.3.2.

NOTE 2 Care should be taken not to spill any with ^{14}C-labelled synthetic triglycerides on the external surfaces.

NOTE 3 The two remaining test specimens are used to check whether the sample loses mass from the evaporation of volatiles, such as water, solvents and oligomers, during the test period. If the vacuum drying procedure in annex B is applicable these test specimens are not required as during the vacuum drying volatiles are removed from the test specimens.

Place the four filled test specimens, and the two empty test specimens and, if following annex C, the tube containing ^{14}C-labelled synthetic triglycerides, in the thermostatically controlled oven or incubator set at the test temperature. Carry out this part of the operation in the minimum time possible to prevent undue heat loss.

If the procedure in annex C is followed the test specimens filled with ^{14}C-labelled synthetic triglycerides and the tube containing ^{14}C-labelled synthetic triglycerides have to be sealed.

Observe the temperature of the thermostatically controlled oven or incubator or the ^{14}C-labelled synthetic triglycerides (see NOTE 5) in the filled article and leave the test specimens for the selected test period, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002, after the ^{14}C-labelled synthetic triglycerides in the test specimen has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

NOTE 4 Annex B of EN 1186-1:2002 includes tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant to this Part of the standard.

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

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

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

Remove the test specimens and the tube, if used, from the thermostatically controlled oven or incubator and immediately empty the test specimens that contained ^{14}C-labelled synthetic triglycerides and allow the oil to drain. Remove any adhering ^{14}C-labelled synthetic triglycerides by gently pressing between filter papers (6.3.7). Repeat the pressing procedure until the filter paper shows no spots of ^{14}C-labelled synthetic triglycerides

If the procedure in annex C is followed, transfer the ^{14}C-labelled synthetic triglycerides from the test specimens into tubes and seal the tubes to prevent further change in the moisture content of the oil, seal the tube containing reference ^{14}C-labelled synthetic triglycerides and carry out the Karl Fischer determination of water content as soon as possible.

6.5.4 Final weighing of test specimens

6.5.4.1 For those specimens which did not require conditioning to obtain their initial masses (see 6.5.2.2), weigh all six test specimens i.e. the four that have been in ^{14}C-labelled synthetic triglycerides and the two that were in the empty tubes and record the mass of each test specimen.

6.5.4.2 If conditioning of the test specimens was carried out using the procedure in annex A (see 6.5.2.3) then repeat the procedure.

6.5.4.3 If conditioning was carried out before the initial weighing using the procedure described in annex B (see 6.5.2.4) then carry out the procedure described in B.4.

6.5.4.4 If it was decided that the procedure described in annex C (see 6.5.2.4) was applicable to the test sample, then carry out that procedure.

6.5.4.5 If the final mass of each of the 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 10.4 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.

6.5.5 Extraction of absorbed ^{14}C-labelled synthetic triglycerides

Cut into suitable sized strips, not wider than 30 mm and of correct length such that the strips shall be totally immersed during the soxhlet cycle.

NOTE 1 Care should be taken when carrying out the cutting operations to ensure that slivers are not produced and lost.

Take four flasks, 250 ml or 500 ml as appropriate to the size of the soxhlet type extractor (6.3.11) to be used for the extraction and add sufficient extraction solvent (6.2.2) 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 (6.3.10) to control boiling.

Place the four test specimens which have been in contact with ^{14}C-labelled synthetic triglycerides into four soxhlet type extractors. Couple each soxhlet to a flask containing the extraction solvent. Using either a water bath or steam bath (6.3.14), extract for a period of $7\,^{+1}_{0}$ h with a minimum of 6 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 almost dryness using a rotary evaporator, or simple distillation apparatus (6.3.13). Transfer the solutions containing the extracted ^{14}C-labelled synthetic triglycerides to separate scintillation vials (6.3.19), and wash each flask with three portions of liquid scintillation cocktail. Add the three washings to the respective individual vials.

NOTE 2 Some types of plastics are known to retain some of the absorbed ^{14}C-labelled synthetic triglycerides. In these cases extraction of the ^{14}C-labelled synthetic triglycerides is incomplete and a second extraction with a more polar solvent is required, see also 10.2 of EN 1186-1:2002.

Repeat the extraction of the test specimens for an additional 7 $^{+1}_{0}$ h, with diethyl ether (6.2.3).

If previous testing has established that all of the ^{14}C-labelled synthetic triglycerides are extracted from the test specimens during the first 7 h extraction then the second 7 h extraction may be omitted.

Isolate the residues in scintillation vials, using the procedure described above.

Determine the extracted ^{14}C-labelled synthetic triglycerides in both the first 7 h and the second 7 h extraction by the procedure described in 6.5.6, but retain the test specimens in the soxhlet type extractors until the extracted ^{14}C-labelled synthetic triglycerides has been determined for the second extraction. If more than 2 mg of ^{14}C-labelled synthetic triglycerides per square decimetre of the surface exposed to ^{14}C-labelled synthetic triglycerides is found in the second extract, then determine the retained ^{14}C-labelled synthetic triglycerides via liquid scintillation counting after combustion or dissolution of the test specimen.

6.5.6 Determination of extracted ^{14}C-labelled synthetic triglycerides

6.5.6.1 Standard and background samples

Take five scintillation vials and add ^{14}C-labelled synthetic triglycerides from the same batch as used for the migration test, the amounts being from 50 mg to 250 mg. Weigh to the nearest 0,1 mg and add liquid scintillation cocktail in the required amount. Take three scintillation vials and fill with cocktail only.

6.5.6.2 Liquid scintillation counting

Transfer the samples prepared according to 6.5.5 and 6.5.6.1 into the liquid scintillation counter (6.3.18) and determine the radioactivity in the sample. Make sure that the instrument has been set to the correct parameters for determination of carbon-14, including the correct quench curve.

6.5.6.3 Calculation of extracted ^{14}C-labelled synthetic triglycerides

Calculate the specific radioactivity, *sA*, of the mixture of ^{14}C-labelled synthetic triglycerides with consideration of the background value

$$sA = \frac{R_S - R_0}{w} \qquad (16)$$

where

sA is the specific radioactivity, in disintegrations per minute per milligram;

R_S is the measuring rate, in disintegrations per minute, of the standard sample (6.5.6.1);

R_0 is the measuring rate, in disintegrations per minute, of the background sample (6.5.6.1);

w is the mass of the standard sample, in milligrams.

Calculate the amount of extracted ^{14}C-labelled synthetic triglycerides as follows:

$$m_c = \frac{R_M - R_0}{sA \times 1000} \qquad (17)$$

where

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams;

R_M is the measuring rate, in disintegrations per minute, of the sample (6.5.6.2);

R_0 is the measuring rate, in disintegrations per minute, of the background sample as prepared in 6.5.6.1;

sA is the specific radioactivity, in disintegrations per minute per milligram.

6.5.6.4 Determination of retained ^{14}C-labelled synthetic triglycerides

6.5.6.4.1 General

If the amount of ^{14}C-labelled synthetic triglycerides in the second extraction is less than 4 mg, but measurable, add this to the amount determined from the first extraction and record the total mass to the nearest milligram of extracted ^{14}C-labelled synthetic triglycerides for each test specimen in grams.

If the amount of ^{14}C-labelled synthetic triglycerides of more than one of the test specimens in the second extraction is greater than 4 mg for each test specimen, add the quantity of ^{14}C-labelled synthetic triglycerides from the second extraction to the quantity determined in the first extraction and determine the amount of ^{14}C-labelled synthetic triglycerides retained after combustion or dissolution of the extracted sample. The combustion method (see 6.5.6.4.2) is suitable for all plastics. The dissolution method (see 6.5.6.4.3) is only suitable for plastics that are soluble in a suitable solvent, e.g. tetrahydrofuran.

6.5.6.4.2 Combustion method

Dry the extracted test specimen and weigh as described in 6.5.4.1; cut five small pieces of about 50 mg from each test specimen, weigh to the nearest 0,1 mg and combust in a sample oxidizer (6.3.20). Determine the radioactivity in the samples obtained, as described in 6.5.6.2, and calculate the amount of ^{14}C-labelled synthetic triglycerides retained, according to 6.5.6.3, taking into account the aliquot of the extracted test specimen used for combustion. Add this quantity of ^{14}C-labelled synthetic triglycerides to that found by the extraction for each test specimen.

6.5.6.4.3 Dissolution method

Transfer the extracted test specimen into a beaker (100 ml, 250 ml or 500 ml as appropriate), dissolve in a minimum amount of tetrahydrofuran, transfer the solution into a volumetric flask (250 ml, 500 ml or 1 000 ml as appropriate) and make up to the mark. Take three aliquots of 2 ml each into scintillation vials, add liquid scintillation cocktail in the required amount and determine the radioactivity in the samples as described in 6.5.6.2. Calculate the amount of ^{14}C-labelled synthetic triglycerides retained according to 6.5.6.3. Add this quantity of ^{14}C-labelled synthetic triglycerides to that found by the extraction for each test specimen.

6.6 Expression of results

6.6.1 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:

$$M = \frac{[\, m_a - (m_b - m_c)\,] \times 1000}{S} \qquad (18)$$

where

M is the overall migration into ^{14}C-labelled synthetic triglycerides, in milligrams per square decimetre of 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 ^{14}C-labelled synthetic triglycerides in grams (see 6.5.2.2 or 6.5.2.3 or 6.5.2.4 as appropriate);

m_b is the mass of the test specimen after contact with ^{14}C-labelled synthetic triglycerides in grams (see 6.5.4) or corrected mass (see formula 20) where the loss of volatiles is greater than 4 mg per test specimen (see 6.5.4.4);

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams (see 6.5.6.3 or 6.5.6.4);

S is the surface area of the test specimen in contact with the food simulant, in square decimetres. See 9.7 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 milligram per square decimetre. See 12.3 of EN 1186-1:2002, for directions to determine whether the results are valid.

However the overall migration shall be expressed in milligrams lost 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.

The overall migration shall be calculated for case a) and for case b) for each test specimen using the following formula:

$$ML = \frac{[m_a - (m_b - m_c)] \times 1000}{V} \qquad (19)$$

where

ML is the overall migration into the simulant, in milligrams per kilogram of food simulant;

m_a is the initial mass of the test specimen, before contact with the ^{14}C-labelled synthetic triglycerides, in grams (see 6.5.2.2 or 6.5.2.3 or 6.5.2.4 as appropriate);

m_b is the mass of the test specimen after contact with ^{14}C-labelled synthetic triglycerides in grams (see 6.5.4) or corrected mass (see formula 17) where the loss of volatiles is greater than 2 mg/dm² of surface exposed to ^{14}C-labelled synthetic triglycerides per test specimen (see 6.5.4.4);

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams (see 6.5.6.3);

V is the volume of the food simulant in contact with the test specimen, in litres, since the specific gravity of the ^{14}C-labelled synthetic triglycerides is conventionally assumed to be 1.

Calculate the result for each specimen to the nearest 0,1 mg/kg and the mean of the valid test results, to the nearest milligram per kilogram. See 12.3 of EN 1186-1:2002, for directions to determine whether the results are valid.

The corrected mass is calculated using the formula:

$$m_b = m_b' + m_d \qquad (20)$$

where

m_b is the corrected loss in mass of the test specimens in the 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 ^{14}C-labelled synthetic triglycerides, in grams.

NOTE This adjustment assumes that the same quantity of volatiles is lost from a test specimen exposed to ^{14}C-labelled synthetic triglycerides, as is lost from a test specimen not exposed to ^{14}C-labelled synthetic triglycerides.

If the procedure described in annex C 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:

$$M_D = \frac{[m_a - (m_b - m_c + M_w)] \times 1000}{S} \qquad (21)$$

where

M_D is the overall migration into ^{14}C-labelled synthetic triglycerides, 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 C;

m_a is the initial mass of the test specimen, before contact with the ^{14}C-labelled synthetic triglycerides, in grams;

m_b is the mass of the test specimen after contact with ^{14}C-labelled synthetic triglycerides, in grams. or corrected mass (see equation (4)) where the loss of volatiles is greater than 2 mg per test specimen;

m_c is the mass of ^{14}C-labelled synthetic triglycerides absorbed by test specimen, in grams;

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

S is the surface area of the test specimen intended to come into contact with foodstuffs in square decimetres.

6.6.2 Precision

See annex E.

6.7 Test report

Where the plastics 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 12.2 of EN 1186-1:2002).

The test report shall include the following:

a) reference to this European Standard and 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 therefore;

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

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

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

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

Annex A
(normative)

Determination of the need for sample conditioning

A.1 Principle

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

A.2 Procedure

A.2.1 Take one test specimen, prepared as required by the individual methods and place in a container 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.

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

A.3 Conclusions

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

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

A.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.

A.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 B
(normative)

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

B.1 Principle

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

B.2 Establishing the need for conditioning of test specimens

B.2.1 Procedure

Take one test specimen, prepared as required by the individual methods and determine the mass to the nearest mg. Place the test specimen in a vacuum oven 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 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.

B.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 (B.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 is not necessary before each weighing operation in the test procedure.

B.3 Initial weighing of test specimens

B.3.1 Conditioning of test specimens

Weigh the four test specimens, prepared as required by the individual methods, then transfer to a vacuum oven 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 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.

B.3.2 Reconditioning of the test specimens

Place the test specimens at ambient humidity or in a container maintained at 80 % relative humidity, until the test specimens have regained at least 70 % of the mass lost during vacuum drying, see 10.9 of EN 1186-1:2002. The test specimens are now ready to be brought into contact with the ^{14}C-labelled synthetic triglycerides.

B.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 ^{14}C-labelled synthetic triglycerides.

Annex C
(normative)

Determination of change in moisture content of test specimens by measurement of the transfer of water to, or from mixture of ^{14}C-labelled synthetic triglycerides, by Karl Fischer titration

C.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.

C.2 Reagents

C.2.1 Samples of ^{14}C-labelled synthetic triglycerides simulant obtained from each of the 4 (or 3) tubes which contained the test specimens immediately following the migration testing (50 ml).

C.2.2 Three samples of the blank ^{14}C-labelled synthetic triglycerides simulant carried through an equivalent migration procedure but with no test specimen present (50 ml).

C.2.3 Sample of ^{14}C-labelled synthetic triglycerides (5.2 of EN 1186-1:2002) simulant intended for use in migration tests.

C.3 Procedure

C.3.1 Assessment of the simulant

Take replicate (n = 5) portions of simulant intended for use in the migration tests (C.2.3) and determine the performance of the Karl Fischer apparatus employed. The precision (standard deviation) of this determination shall be 10 mg/kg or less in order to achieve the required precision of 1 mg/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 C.3.2.

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

C.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 C.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.

C.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.

C.3.4 Karl Fischer titration of simulant samples

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

C.3.4.2 Take triplicate sub-samples 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 C.3.4.3.

C.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 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 1-2 min) before commencing the titration.

C.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 $W_C = Q_W / M_O$.

C.3.5 Precision of the water determination

The results W_C for the triplicate sub-samples have to 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 (C.3.4) and remove the source of variation.

C.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.

C.4 Expression of results

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

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

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 D
(informative)

Example of a pouch holder

Dimensions in millimetres

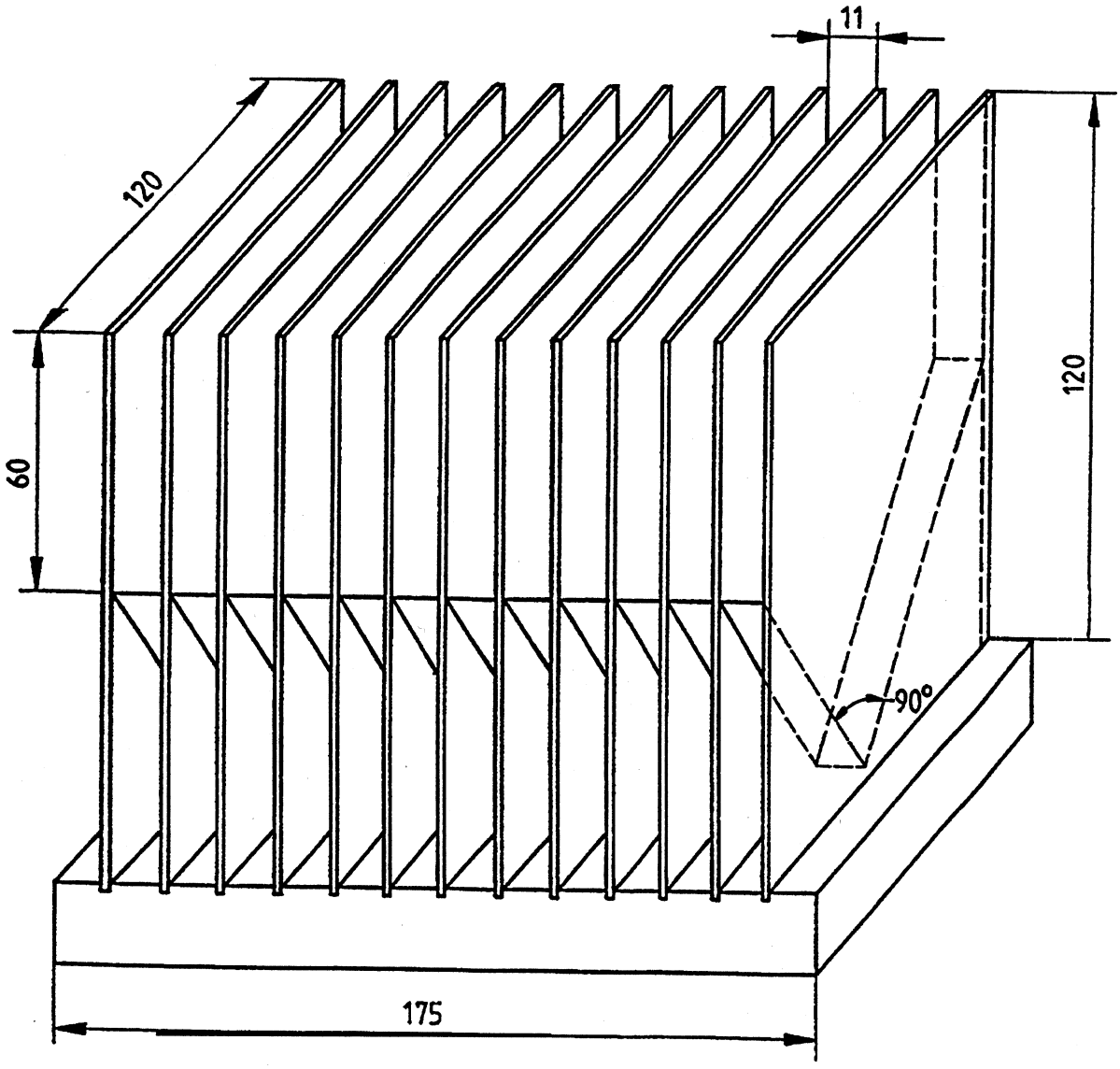

Figure D.1 — Example of a pouch holder

Annex E
(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 ^{14}C-labelled synthetic triglycerides 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 olive oil or other fatty food simulants e.g. ^{14}C-labelled synthetic triglycerides 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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[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. L75. p 19.

[3] Commission of the European Communities, Council Directive of 18 October 1982 laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs (82/711/EEC), Official Journal of the European Communities, 23 October 1982, no. L 297, p 26.

[4] Commission of the European Communities, Commission Directive of 15 March 1993 amending Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs (93/8/EEC), Official Journal of the European Communities, 14 April 1993, no. L 90, p 22.

[5] Commission of the European Communities, Commission Directive 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.

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[7] EN ISO 8442-2, *Stainless steel and silver plated table cutlery*.

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