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Materiali in predmeti v stiku z ġivili - Polimerni materiali - 11. del: Preskusne metode za celotno migracijo v zmes sinteţiļnih trigliceridov, oznaļenih s 14C

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

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

Matériaux et objets en contact avec les denrées alimentaires - Matières plastiques - Partie 11: Méthodes d'essai pour la migration globale dans des mélanges de triglycérides synthétiques marqués au 14C

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This European Standard was approved by CEN on 29 April 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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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of an identical text or by endorsement, at the latest by March 2003, and conflicting national standards shall be withdrawn at the latest by March 2003.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 × 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 × 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 × 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 20 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);