Irish Standard
I.S. EN 1186-2:2022

Version 2.00

Materials and articles in contact with foodstuffs - Plastics - Part 2: Test methods for overall migration in vegetable oils

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NSAI
1 Swift Square
Northwood, Santry
Dublin 9
D09 A0E4
+353 1 807 3800
standards@nsai.ie
NSAI.ie

Sales
+353 1 857 6730
Standards.ie

Údarás um Chaighdeáin Náisiúnta na hÉireann

National Foreword

I.S. EN 1186-2:2022 V2.00 is the version of the NSAI adopted European document EN 1186-2:2022, *Materials and articles in contact with foodstuffs - Plastics - Part 2: Test methods for overall migration in vegetable oils,* including any Corrections, Amendments etc. to EN 1186-2:2022.

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EUROPEAN STANDARD

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EN 1186-2

July 2022

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English Version

Materials and articles in contact with foodstuffs - Plastics - Part 2: Test methods for overall migration in vegetable oils

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 2 : Méthodes d'essai pour la migration globale dans les huiles végétales

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 2: Prüfverfahren für die Gesamtmigration in Pflanzenölen

This European Standard was approved by CEN on 20 June 2022.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 1186-2:2022) has been prepared by Technical Committee CEN/TC 194 “Utensils in contact with food”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by January 2023, and conflicting national standards shall be withdrawn at the latest by January 2023.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 1186-2:2002, EN 1186-4:2002, EN 1186-6:2002, EN 1186-8:2002, EN 1186-10:2002 and EN 1186-12:2002.

This document implements European Commission Regulation on plastic materials and articles intended to come into contact with food with regards to the determination of the overall migration in food simulants. This regulatory text is subject to change, it is therefore strongly recommended that users of this document refer to the latest relevant published regulatory texts before commencement of any of the test or tests described in this document, looking to the European Commission website.

In comparison with the previous editions, the following technical modifications have been made:

— removed the regulatory provisions put in the document to avoid the document being obsolete after an update of regulation;

— adapted the method of test to the new conditions defined in the regulation;

— gathered in one document the 6 previous standards that use vegetable oils as simulants and which are based on the same principle of measuring.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This document specifies methods for measuring overall migration of plastic materials and articles intended to come into contact with foodstuffs by contacting test specimens with vegetable oils at temperatures greater than or equal to 4 °C and less than or equal to 175 °C.

NOTE Some vegetable oils are not suitable for use below 20 °C.

The overall migration from a sample of the plastics is determined as the loss in mass of non-volatile substances expressed:

— per unit surface area; or

— per kg of food simulant; or

— per article

after contact with a food simulant under defined conditions.

According to the type of materials or shape of articles, contact with the food simulant is carried out on a single surface (pouch, cell, filling) or by immersion.

This document does not cover the interpretation of the results which is expected to account for regulatory requirements.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

3 Terms and definitions

For the purposes of this document, the following terms and definitions.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
ready-to-use article
article as sold that can be used with minimal if any preparation

3.2
sample
material or article under test

3.3
test specimen
part of the sample undergoing a measurement during the test

3.4
piece
portion of a test specimen

3.5
conventional oven
thermostatically controlled heat chamber where the air within is heated and this heat is then transferred to the food through the plastic as opposed to a microwave oven where the food itself is heated directly by microwave's irradiation

3.6
fillable pouch
receptacle of a defined size, manufactured in the film under test and which, once filled with food simulant, exposes the side of the film to be in contact with foodstuffs to such a food simulant or to a test medium

3.7
reverse pouch
pouch manufactured such that the surface to be in contact with foodstuffs is the outer surface

Note 1 to entry: All sides are sealed to prevent inner surfaces from coming into contact with the food simulant. The reverse pouch is to be completely immersed in the food simulant or in the test medium.

3.8
cell
device in which the film under test can be mounted and which, when assembled and filled with food simulant, exposes the side of the film to be in contact with foodstuffs to such a food simulant or to a test medium

3.9
food simulant
test medium imitating food, in its behaviour food simulant mimics migration for food contact materials

4 Test methods

4.1 Principle

4.1.1 General

The overall migration of a material or a ready-to-use article made of plastic in contact with fatty foods, for which vegetable oils must be used, is determined by putting test specimens in contact with a vegetable oil in test conditions chosen on the basis of the worst case scenario of use, by weighing test specimens before and after contact with the vegetable oil, by dosing the oil absorbed by the material by gas chromatography which is deduced from the obtained mass difference.

Test specimens of known mass are placed in contact with oil for the exposure time, at temperatures greater than or equal to 4 °C and below or equal to 175 °C, then taken from the oil, drained and wiped to remove oil adhering to the surface, and reweighed.

The specimens will usually retain absorbed oil that is extracted and determined quantitatively by means of gas chromatography after conversion to methyl esters. Methylation is carried out by reacting a boron trifluoride/methanol complex with fatty acids formed by hydrolyzing the oil with potassium hydroxide.

An internal standard, triheptadecanoin, is added prior to the extraction of the absorbed oil from the test specimens. This ensures that any active or extractable components of the plastics react with the internal standard, as well as with the extracted oil. The internal standard is also subjected to the hydrolysis and

methylation reactions, providing compensation for any inefficiencies in the hydrolysis and methylation processes.

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

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

According to the type of sample, the tests are conducted based on one of the following contacting methods at a temperature greater than or equal to 4 °C and less than or equal to 175 °C and for the specified exposure time.

NOTE In some vegetable oils, this method is also suitable for temperatures above 175 °C. The suitability of vegetable oils depends on their physical properties.

4.1.2 Method 1: total immersion

Test specimens of known mass and surface are immersed in oil; this method is most suitable for plastics in the form of films and sheets, but can also be applied to a wide range of articles or containers from which test specimens of a suitable size can be cut.

4.1.3 Method 2: cell

Test specimens of known mass are placed in contact with oil in a cell; this method is most suitable for plastics in the form of films or sheets in which only one surface is to be in contact with foodstuffs (printed, multi-layer materials, etc.).

4.1.4 Method 3: fillable pouch

Test specimens of known mass and in the form of pouches are filled with oil; this method is suitable for plastics in the form of films or sheets in which the surface to be in contact with foodstuffs (printed, multi-layer materials, etc.) can be sealed by applying heat or pressure, to form a pouch.

4.1.5 Method 4: reverse pouch

Test specimens of known mass and in the form of reverse pouches are immersed in oil; this method is suitable for plastics in the form of films or sheets (printed, multi-layer materials, etc.) in which both surfaces can be sealed by applying heat or pressure to form reverse pouches. This method, whenever possible, shall be preferred over those using fillable pouches when the oil temperature is above 70 °C due to the pressure from the oil that may damage the pouch seal at high temperatures.

4.1.6 Method 5: filling a container

Test specimens of known mass are filled with oil; this method is 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 provided they are not too large. If the article is large, to avoid handling and weighing problems or using excessive amounts of oil, it may be preferable to cut it paying attention not losing pieces of the material of test specimen.

4.2 Reagents

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

4.2.1 Oil. Vegetable oils used as simulant shall be rectified and contain less than 1 % of unsaponifiable matter (waxes and essential oils).

4.2.2 Extraction solvents.

4.2.2.1 Pentane is the solvent recommended for the first extraction for all type of plastic materials.

4.2.2.2 A 95/5 by volume azeotropic mixture of pentane 98 % and ethanol 96 % is the solvent recommended for polar plastics such as polyamide and polyacetal.

NOTE 1 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.

NOTE 2 The solvent can be recycled by redistilling it and removing fats.

4.2.3 Triheptadecanoin (glyceryl trimargarate) CAS n° 2438-40-6 solution, 2 mg/ml in cyclohexane.

NOTE Other internal standards can be used, such as methyl cinnamate (CAS n°: 103–26–4) or glyceryl trinonadecanoate (CAS n°26536–13–0).

4.2.4 Potassium hydroxide solution, 11g/l in methanol.

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

4.2.6 *n*-heptane.

4.2.7 Sodium sulfate, saturated solution.

4.3 Materials and apparatus

4.3.1 General

The constituent materials of the materials and apparatus used and the condition thereof shall make it possible to prevent contamination of the samples, reagents and solutions under analysis.

The materials and apparatus shall be suitably cleaned.

4.3.2 Common materials and apparatus for all methods

4.3.2.1 Analytical balance having a precision of at least 0,1 mg.

4.3.2.2 Conventional oven (thermostatically controlled oven, incubator, refrigerator, etc.) capable of maintaining the set temperature, within the tolerances specified in Annex A.

4.3.2.3 Steam bath, hot plate, distillation apparatus or rotary evaporator.

4.3.2.4 Desiccator with, for example, anhydrous calcium chloride or silica gel.

4.3.2.5 Lint-free cloth or soft brush or pure compressed air generator.

4.3.2.6 Conditioning containers, for conditioning test specimens at 50 % ± 5 % and 80 % ± 5 % relative humidity at 23 °C ± 2 °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 in water 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 conventional oven, at a temperature of approximately 20 °C, the set temperature should not vary by more than ± 1 °C.

4.3.2.7 Anti-bumping beads made of inert material, for example 2 mm to 3 mm in diameter made of glass or ceramic.

4.3.2.8 Soxhlet type extractors, capable of holding test specimens on the supports, with flasks of appropriate size.

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

4.3.2.9 Water bath or heating mantle, capable of holding and heating the flasks of Soxhlet type extractors.

4.3.2.10 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.

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

4.3.2.12 Gas chromatograph, with flame ionization detector or other suitable detector equipped with an appropriate column.

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

The following columns have been found to be suitable:

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

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

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

For other vegetable oils: The condition for separation of the main fatty acid esters should be checked.

4.3.2.13 Glass containers with stoppers, of a volume of approximately 10 ml, for storing the heptane layer if necessary.

4.3.2.14 Conventional oven under vacuum or vacuum desiccator, capable of maintaining a temperature of 60 °C ± 2 °C. The conventional oven or the 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 conventional oven under vacuum is not available, a vacuum desiccator placed in an oven at 60 °C can be used.

4.3.2.15 Disposable plastic syringes with luer fitting, 1 ml or 10 ml and wide gauge luer needles (80 mm ± 1,2 mm) or equivalent equipment (graduated cylinder, pipette, etc.).

4.3.2.16 Absorbent paper.

4.3.3 Common materials for methods other than filling a container

4.3.3.1 Cutting slab, clean smooth glass, metal or plastic slab of sufficient area to prepare test specimens.

4.3.3.2 Blunt-nosed tweezers, for example made of stainless steel.

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

4.3.3.4 Cutting templates measuring (100 mm ± 0,2 mm) X (100 mm ± 0,2 mm).

4.3.3.5 Tool for measuring length, having a precision of 1 mm.

4.3.3.6 Glass containers equipped with an inert sealing system (stopper, lid, etc.) for containing the oil and test specimens, for example test tubes, ground neck, with an internal diameter of approximately 35 mm and length in the range of 100 mm to 200 mm, excluding the ground neck.

4.3.3.7 Ground-necked flasks of suitable size.

4.3.3.8 Pipettes, 5 ml and 10 ml, conforming to the requirements of ISO 648 Class B or automatic pipettes of equivalent performances.

4.3.4 Materials for method 1 (total immersion)

4.3.4.1 Specimen supports, for example made of stainless steel, capable of holding and keeping the test pieces apart and at the same time ensuring complete contact with oil.

4.3.4.2 Gauze, for example, fine stainless steel gauze, mesh size 1 mm, approximately 25 mm x 100 mm in size.

4.3.4.3 Glass rods, for example 2 mm to 3 mm in diameter and approximately 100 mm long, for insertion between the test pieces.

4.3.4.4 Glass beads, for example 2 mm to 3 mm in diameter.

4.3.5 Materials for method 2 (cell)

4.3.5.1 Migration cell:

— minimum of 0,4 dm^2 of contact area for the test specimen;

— surface to volume ratio shall be between (1 to 2) cm^{-1} for the test specimen;

— blank value shall be less than 5 mg/l.

4.3.6 Materials for method 3 (fillable pouches holder) and for the method 4 (reverse pouches holder)

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

4.3.7 Materials for method 5 (filling a container)

Glass containers.

4.4 Preparation of test specimens

4.4.1 General

The test specimens shall be 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, or with a compressed air stream.

As a general rule, do not wash the sample with water or solvent. However, if the articles are accompanied by instructions for use intended for the user advising cleaning before use, these instructions should be followed for the test, unless they advise rubbing the article with oil: in this case, the instructions should not be followed insofar as the oil would be included in the overall migration.

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

Seven test specimens are required for samples, in the form of thin films, sheets, cut sections from containers or similar articles. Nine test specimens, similar dimensionally to one 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) one test specimen to determine the suitability of the oil as the fatty food simulant and triheptadecanoin as the internal standard (according to Annex B);

c) a minimum of one test specimens to check for possible loss of volatiles if the sample does not undergo a stage in a vacuum oven;

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

In case of repeated used articles, the numbers of test specimen are utilized as follows:

e) four test specimen for each migration test: twelves (three times four);

f) one test specimen to determine the suitability of the oil as the fatty food simulant and triheptadecanoin as the internal standard (according to Annex B);

g) one test specimen for each migration test for possible loss of volatiles: three (three times one);

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

If the conditioning test in Annex D is used, one additional test specimen is required.

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

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

Determine the area of each test specimen to the nearest 0,01 dm^2 and record.

If the result is to be expressed in mg/kg, determine the contact surface area and the volume of the article and record.

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

NOTE The test specimens c) 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 D is used, these test specimens are not needed as during the vacuum drying any volatiles will have been removed from the test specimens.

4.4.2 Preparation of test specimens and determination of the area in contact

4.4.2.1 General

It is recommended to use surface of 1 dm^2 and a volume of 100 ml. Deviations should be recorded and explained. If necessary, a test specimen could be cut in pieces.

The test specimens shall be clean and free from surface contamination (many plastics can readily attract dust due to static charges).

4.4.2.2 Method 1: total immersion

4.4.2.2.1 General

Cut out test specimens measuring 1 dm^2 ± 0,01 dm^2 using the tool of 4.3.3.4. If this is not possible, cut out pieces suitable for obtaining test specimens having a surface area of approximately 1 dm^2.

Ensure that the test pieces are well separated and that their surfaces are freely exposed to the oil during the test, using gauze, glass rods and/or specimen holders as required.

If the area of the edges of the test specimen exceeds 10 % of the area of the test specimen measured, this is incorporated in the area calculation.

4.4.2.2.2 Films and sheets

Cut each test specimen into four pieces 25 mm x 100 mm using the tool of 4.3.3.5.

4.4.2.2.3 Containers and other articles

Cut sections from the walls of the container or article to give test specimens each of area approximately 1 dm^2. For articles with individual areas less than 1 dm^2, use a sufficient number of articles to provide each test specimen. Measure the dimensions of each test specimen to the nearest 1 mm, using a suitable measuring tool.

Calculate the area of each test specimen to the nearest 0,01 dm^2 and record. If necessary, cut each test specimen into smaller pieces to enable them to fit into the glass containers. The test specimens or pieces are placed on the specimen supports or, if the test specimens or pieces are sufficiently rigid, they can be tested unsupported.

The number of cuttings should be limited.

4.4.2.2.4 Articles of irregular shape

Select representative portions of the article, or multiples of the article for small articles, to give sufficient area of material to obtain test specimen (s). Surface area of each test specimen should be at least 1 dm^2. Measure only the surface area intended to come into contact with foodstuffs of two of these test specimens to the nearest 0,05 dm^2 using the Schlegel Method [5], the method described by Mieth and Hoekstra [6], or any other suitable method [7]. Record the surface area of each test specimen.

4.4.2.3 Method 2: cell

Lay the sample on the cutting slab with the surface to be in contact with the oil uppermost.

Cut out the test specimen of suitable dimensions for the cell using the cutting implement.

4.4.2.4 Method 3: fillable pouch

Prepare a pouch suitable for obtaining an internal contact area of 2 dm^2 and containing a suitable volume of oil.

For example, fold over a sheet measuring 120 mm by 240 mm, with the surface under test facing inward, and seal the 3 other sides such that the area inside the seals defines a square measuring 100 mm by 100 mm, then cut one corner of the pouch in order to be able to fill it with oil (after filling, the corner of the pouch may be sealed or closed with a clamp).

An alternative method consists of only sealing two sides and sealing the final side after filling with oil.

Another alternative method consists of using two sheets measuring 120 mm by 120 mm and creating four seals.

If required, remove any air bubbles liable to be present in the oil before sealing the filled pouch for the final time.

Measure the internal dimensions of the pouches prepared and calculate the exposed areas to the nearest 0,01 dm^2. These measurements may be made after exposure to oil.

Remove excess film from the sealed area (to reduce the area of film not directly exposed to oil), whilst leaving enough to withstand the test conditions without leaking.

For multilayer films in which the external side is sealable, the reverse pouch method may be used to test the internal surface by immersion.

Pouches of dimensions other than 100 mm x 100 mm can be used for this test. These pouches shall have, wherever possible, a contact area greater than or equal to 1dm^2.

4.4.2.5 Method 4: reverse pouch

Prepare pouches such that the surface in contact with the oil is on the outside. These pouches shall have a contact surface area of 2 dm^2, for example by folding over a sheet measuring 120 mm by 240 mm, with the surface under test facing inward, by sealing 2 sides such that the area inside the seals defines a square measuring 100 mm by 100 mm. Then reverse the pouch, and seal the 4th side.

An alternative method consists of taking two sheets measuring 120 mm by 120 mm, creating 3 seals, reversing the pouch and sealing the 4th side.

Measure the external dimensions of the pouches prepared and calculate the exposed areas to the nearest 0,01 dm^2.

Any leaks can be viewed by placing a piece of filter paper inside each reversed pouch, before sealing for the final time; the filter paper will absorb the oil, thus indicating the leak.

4.4.2.6 Method 5: filling a container

If the article is large, to avoid handling and weighing problems or using excessive amounts of oil, it may be preferable to cut it in two pieces and use the bottom of the test specimen so that the surface in contact with the oil does not exceed 3 dm^2.

If this is done, take care that oil does not come into contact with the cut edges of the test specimen. It is important that the area in contact with the oil is determined as it will be incorporated into the calculation later.

Lightly mark an identification code on the external surface of each test specimen.

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.

If the migration result is to be expressed in mg/kg, the test specimen shall be made up of sufficient articles to contain a minimum of 100 ml of the oil.

If the migration result is to be expressed in mg/dm^2, the test specimen shall be made up of sufficient articles to represent a minimum surface area of 1 dm^2.

Determine the surface area to be in contact with the oil when filled to the nominal volume, if known, or when filled to 5 mm from the top of the test specimen if the nominal volume is not known.

In the case of articles with a volume of less than 100 ml or surface area less than 1 dm^2, this should be the surface area of one article multiplied by the number of articles used to provide a test specimen.

4.5 Procedure

4.5.1 General

Determine the applicability of the method by carrying out the procedure according to Annex B. If prior tests have established that the method is applicable, then Annex B may be omitted.

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

4.5.2 Initial weighing of test specimens

Determine the need for conditioning of the test specimens:

— due to volatiles content above 2 mg/dm^2 follow flow chart 1 (See Figure 1) by carrying out the procedure according to Annex D;

— due to moisture sensitivity follow flow chart 2 (See Figure 2) by carrying out the procedure according to Annex C;

— due to volatiles content and moisture sensitivity above 2 mg/dm^2, follow the flow chart 3 (See Figure 3).

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

NOTE 2 The procedure for conditioning can be chosen according to the type of material.

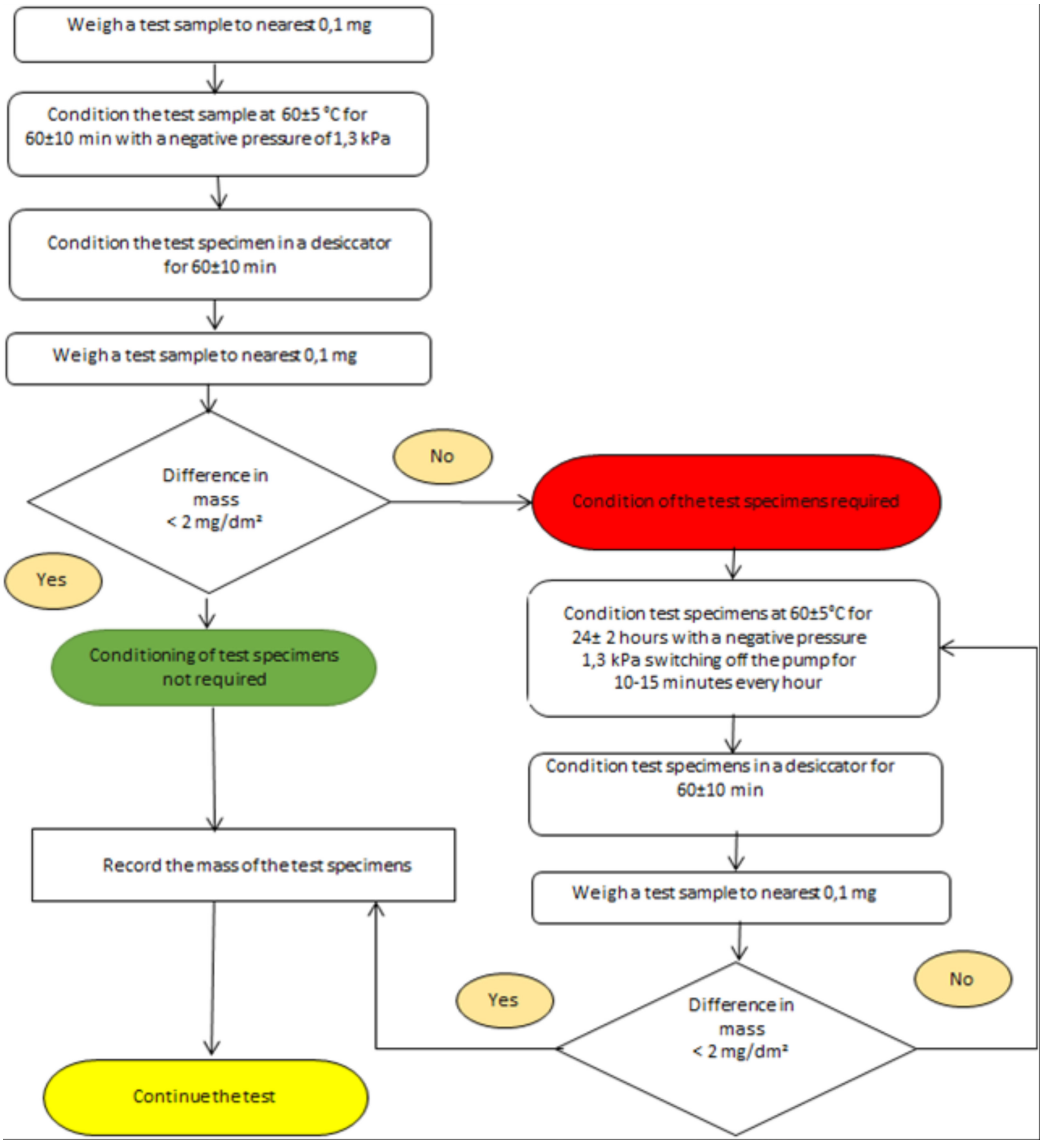

Figure 1 — Test for presence of volatiles and determination of mass of non-moisture-sensitive test specimens

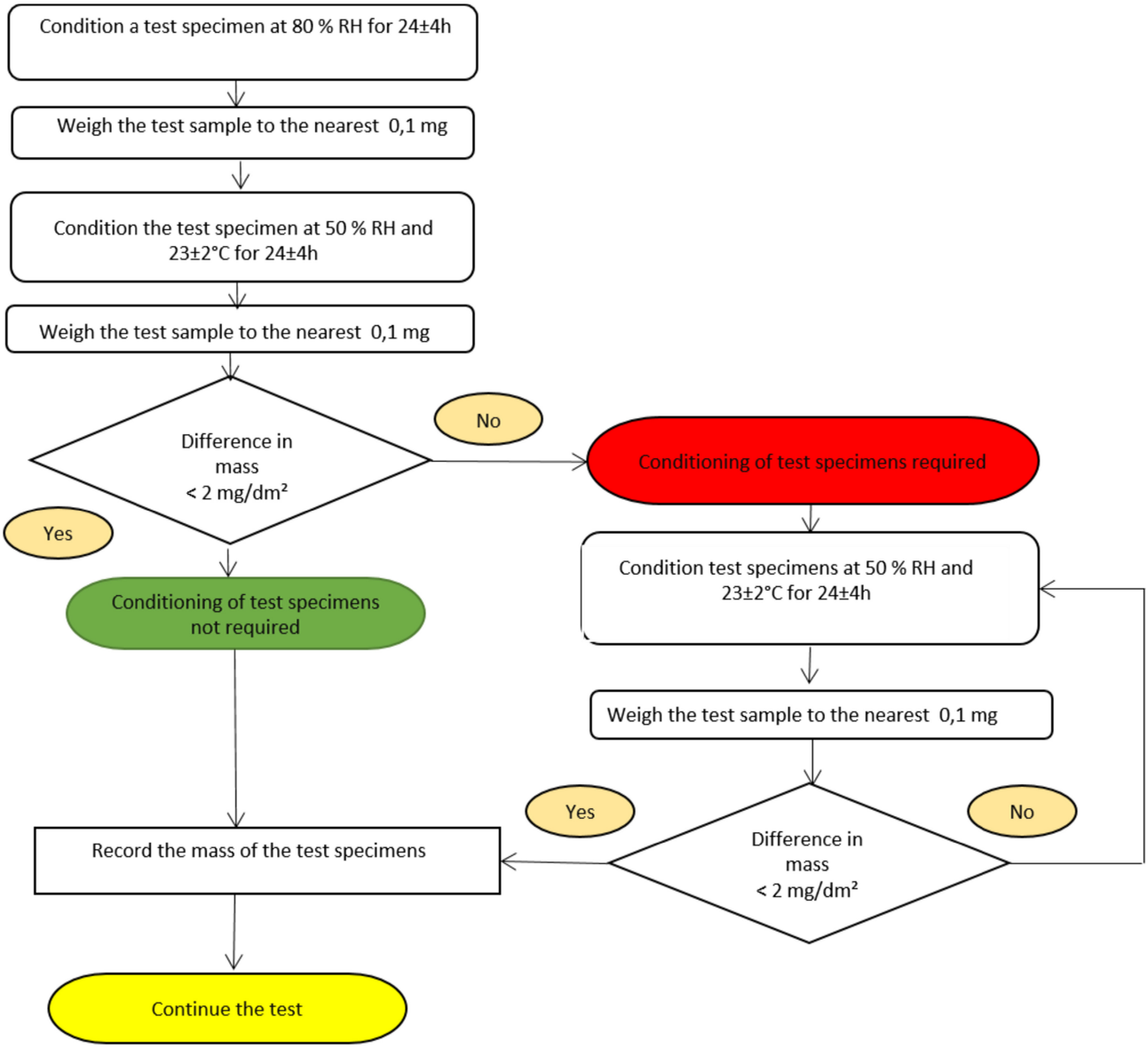

Figure 2 — Test of moisture-sensitivity of test specimens and determination of mass of test specimens in the absence of volatiles

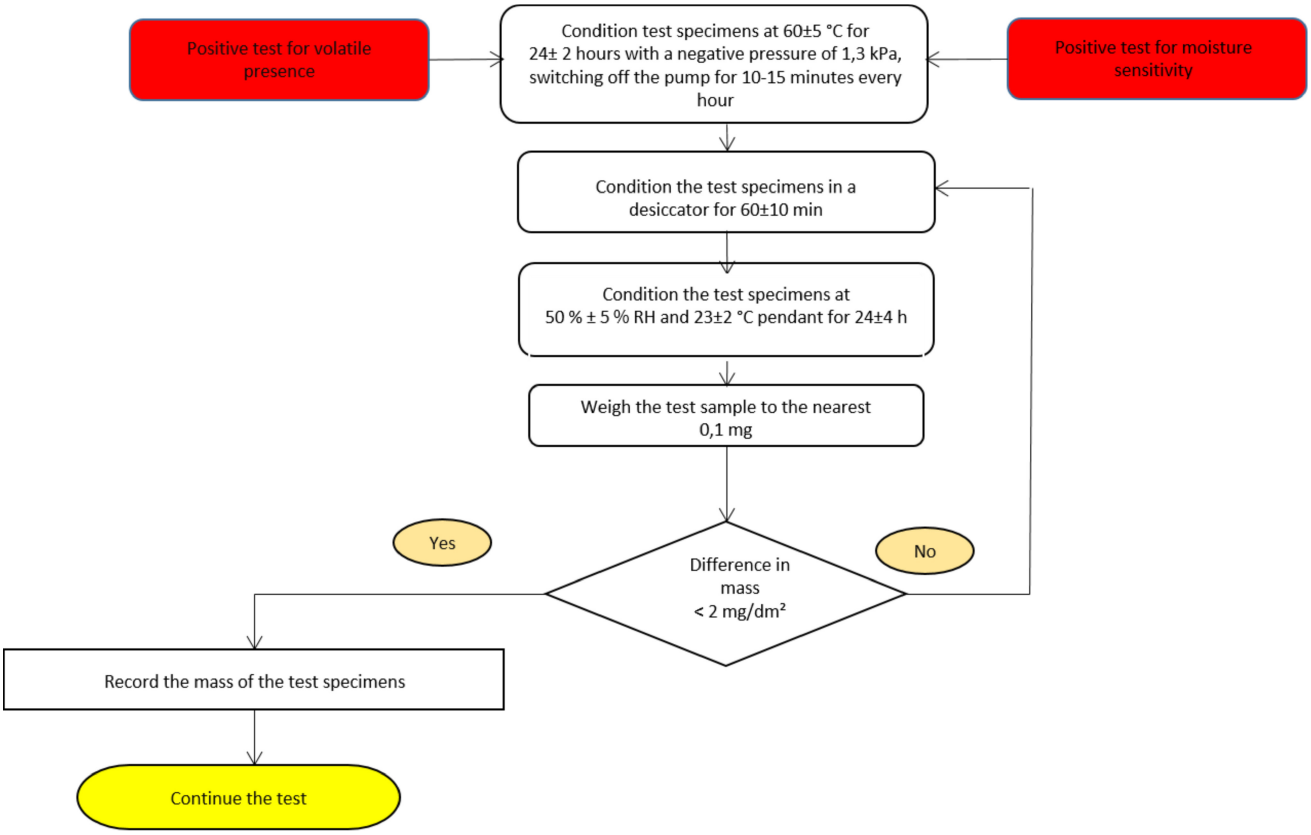

Figure 3 — Determination of mass of moisture-sensitive test specimens with volatiles

4.5.3 Exposure to oil

4.5.3.1 General

General requirements for all exposure methods are to determine:

— the amount of volatiles lost during the exposure;

— the possible interferences of substances extracted from test specimens during the measurement of absorbed oil.

For this purposes, two test specimens shall be placed under the test conditions without exposure to oil. Afterward, these test specimens shall follow the same procedure as the test specimens exposed to oil. If the sample needs vacuum drying, only one test specimen will be used for interferences test.

4.5.3.2 Method 1: Total Immersion and Method 4: Reverse pouch

Prepare eight glass containers. Take six of the glass containers, mark them for identification purposes. Measure the suitable amount of oil into each glass container by measuring cylinder and stopper the glass container.

Alternatively, mark the glass container for a suitable amount of oil and fill with oil to the mark. Place into one of the glass containers a thermometer or a thermocouple and stopper the containers. Place the six glass containers, plus two empty glass containers, in the conventional oven set at the test temperature. Leave until the oil has attained the test temperature, using the thermometer or the thermocouple to monitor the temperature. Take all glass containers from the conventional oven and place into four of the glass containers containing oil, weighed test specimens prepared as in 4.1.1 and conditioned if necessary.

Stopper the glass containers. Ensure that the test specimens are totally immersed in oil. If they are not, then add either glass beads or glass rods to raise the level of the oil until total immersion is achieved.

The oil in the fifth glass container is used as a reference to plot the calibration graph (see 4.5.6.2.2). The oil in the sixth glass container is used to check the temperature of the oil. If glass beads or glass rods have been used to raise the level of the oil to achieve total immersion, then similar glass beads or glass rods should be added to the sixth glass container.

Place the remaining two test specimens into the empty glass containers and stopper.

Return the eight glass containers to the conventional oven set at the test temperature.

These operations should be carried out in the minimum time to prevent undue heat loss.

At the end of the exposure time, remove the glass containers from the conventional oven and remove the test specimens immediately from the glass containers. For those specimens which have been in oil, allow the oil to drain. Remove any adhering oil by gently pressing between filter papers. Repeat the pressing procedure until the filter paper shows no spots of oil. For test specimens on supports, remove the individual test pieces from the supports to carry out this operation. Clean the supports of oil by washing with the extraction solvent and put the test pieces back.

4.5.3.3 Method 2: Cell

Heat a suitable amount of oil in a glass container in a conventional oven, monitoring the temperature with a thermometer or a thermocouple. Leave until the oil has attained the test temperature. When the targeted temperature is reached, remove the glass container containing the suitable amount of oil from the conventional oven. Remove the thermometer or the thermocouple from the glass container.

Place the test specimens in the migration cells. Add the hot oil and place the migration cells in the preheated oven. Preheat the cells and the food simulant at a temperature as close as possible to the testing temperature. In addition, two glass containers with oil and one glass container with two test specimens are placed in the oven. The oil in the first glass container is used as a reference to plot the calibration graph. The oil in the second glass container is used to check the temperature of the oil. Both of the test specimens in the third glass container are used to measure the volatiles and one of the two test specimens is used to determine the interferences during the chromatography analysis.

NOTE Preheating of the migration cells and glass containers can be necessary.

These operations should be carried out in the minimum time to prevent undue heat loss.

At the end of the exposure time, take the migration cells and the glass containers from the conventional oven, empty the oil and immediately remove the test specimens from the cells. For those test specimens which have been exposed, allow the oil to drain. Remove any adhering oil by gently pressing between filter papers. Repeat the pressing procedure until the filter paper shows no spots of oil. Keep all the test specimens in a controlled temperature and moisture atmosphere.

4.5.3.4 Method 3: Fillable Pouch

Take five of the glass containers. Measure the suitable amount of oil into each glass container by measuring with a graduated cylinder and stopper.

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

Alternatively mark the glass container for a suitable amount of oil and fill with oil to the mark. Place the five glass containers and the pouch holder in the conventional oven set at the test temperature. Leakage can occur from the pouches and it is advisable to have a drip tray in the conventional oven.

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

Remove the pouch holder from the conventional oven and position the test specimens between the spacers.

Remove four of the tubes containing oil from the conventional oven and, into four of the test specimen pouches, pipette sufficient oil to fill the pouch. This shall be approximately 100 ml, but for thick/semi-rigid materials the quantity will be less and then follow the process decribed in 4.4.2.4 to seal or clamp the pouches.

The oil in the fifth glass container is used as a reference to plot the calibration graph.

Place the remaining two pouches into the pouch holder.

Replace the pouch holder, containing the six pouches, in the conventional oven set at the test temperature.

These operations should be carried out in the minimum time to prevent undue heat loss.

At the end of the exposure time, remove the pouch holder and the glass containers containing the oil from the conventional oven.

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 oil from each pouch and wipe any excess from the outside with filter paper. Take each of the four pouches in turn, lay on the cutting slab and, using the cutting implement, 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 oil by gently pressing between filter papers. Repeat the pressing procedure until the filter paper shows no spots of oil.

4.5.3.5 Method 5: Filling a container

Place a sufficient volume of oil in a glass container in the conventional oven 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 oil to within 0,5 cm of the top or to the nominal volume, if known. Place into one of the filled test specimens a thermometer or thermocouple.

Place sufficient oil into a glass container for use as reference standards in constructing the calibration graph.

Place the four filled test specimens and the two empty test specimens and the reference oil in the conventional oven set at the test temperature.

This part of the operation should be carried out in the minimum time possible to prevent undue heat loss.

At the end of the exposure time, remove the test specimens and the glass container from the conventional oven and immediately empty the test specimens that contained oil and allow the oil to drain. Remove any adhering oil by gently pressing between filter papers. Repeat the pressing procedure until the filter paper shows no spots of oil.

NOTE For exposure times of 24 h or more, it is acceptable to monitor the temperature of the air bath of the conventional oven, instead of the temperature of the oil.

4.5.3.6 Repeated exposure

In the case of articles to be in repeated contact with foodstuffs, the procedure consisting of exposing the same sample repeatedly to a fresh oil is not feasible, as this method requires solvent extraction to remove fats. For this reason, the test is conducted on three series of similar test specimens from the sample of material or article. One of the series of test specimens undergoes the suitable test for articles intended

for single use according to the reference procedure and the mean result is recorded (M1). The second and third series undergo exposure in exactly the same manner as the first, except that the exposure time is different. Indeed, the second series of test specimens is exposed for a time that is two times longer than the first (M2), and the third for a time that is three times longer than the first (M3).

The migration as a result of the second or third period is calculated as follows:

— migration caused by first period = M1;

— migration caused by the second period = M2 - M1;

— migration caused by the third period = M3 - M2.

No increase in migration into fatty food simulant is deemed to have occurred if the results (M3 - M2) and (M2 - M1) do not exceed M1 by more than the analytical tolerance.

The true values for M1, M2 or M3 are subject to uncertainty owing to the lack of precision inherent in the method. Systematic errors in the determination of the overall migration are likely to occur equally in the determination of M1, M2 or M3 and therefore need not be allowed for. Random errors do need to be recognized and allowed for.

4.5.4 Final weighing of test specimens

4.5.4.1 Weigh all six test specimens, i.e. the four that have been in contact with oil and record the mass of each test specimen.

4.5.4.2 If conditioning was carried out due to moisture sensitivity before the initial weighing, use the procedure described in D.4 for the determination of the final weight.

4.5.5 Extraction of absorbed oil

Take flasks appropriate to the size of the soxhlet type extractor to be used for the extraction, and place in each flask 10 ml of the internal standard solution of triheptadecanoin, using a pipette, or an alternative higher quantity if more than 100 mg of oil is present.

If the test specimens have retained more than 100 mg of oil, 10,0 ml of the internal standard solution is not sufficient for optimum precision in the gas chromatography determination after extraction. Before commencing the operations in this Clause, an estimation of the quantity of oil retained in the test specimens should be obtained by comparing the final masses of the test specimens with their initial masses. If considered necessary, the quantity of internal standard solution can be increased from 10 ml, although it is essential that the same quantity is used for each test specimen, and that this quantity is also used with the oil standards for the calibration graph (see 4.5.6.2.2). As a guide, approximately 0,5 mg of the internal standard is required for every milligram of extracted oil.

Pentane is the solvent recommended for the first extraction for all type of plastic materials.

Add sufficient extraction solvent to allow cycling of the soxhlet type extractor with anti-bumping beads to control boiling.

Place the test specimens which have been in contact with oil into four soxhlet type extractors. Couple each soxhlet to a flask containing the internal standard prepared as above. Using either a water bath or steam bath or heating mantles, 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.

If using manual soxhlet type extractors, drain all of the solvent, remove the flasks from the soxhlet type extractors and evaporate the solvent to approximately 10 ml using a rotary evaporator, or simple distillation apparatus. Transfer the remaining solvent and the residue containing the extracted oil and

internal standard to separate 50 ml flasks, and wash each flask with 3 portions of 5 ml of solvent. Add the three washings to the respective individual 50 ml flasks. Evaporate to dryness using a steam bath or a water bath.

NOTE 1 Evaporation of the solvent to dryness can be carried out under mild conditions of temperature, in order to avoid the oxidation of the oil where possible.

Perform a second extraction of the test specimens for an additional 7 h, adding a further quantity of the internal standard solution. If necessary, use a more polar solvent to complete the extraction of oil.

NOTE 2 The same quantity of internal standard solution is used as for the first 7 h extraction. This quantity might not be the optimum if the quantity of oil in the first 7 h extraction is high. Good precision is not essential for the second 7 h determinations since they are intended primarily as a check on the efficiency of the first 7 h extraction and using the same quantity of internal standard enables one calibration graph to be used.

NOTE 3 If previous testing or scientific evidences has established that all of the oil will be extracted from the test specimens during the first extraction then the second extraction can be omitted.

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

After the first extraction, retain the test specimens in the soxhlet type extractors for the second extraction. Determine the extracted oil in both the first and the second extraction by the procedure described in 4.5.6.

NOTE 4 Other extractions methods (equipment, solvent, conditions) can be applied if demonstrated the capability of the extraction and further transesterification show equivalent results.

4.5.6 Determination of extracted oil

4.5.6.1 Preparation of fatty acid methyl esters

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

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

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

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

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

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

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

If there will be a delay of more than 7 days in using a methyl ester solution for the gas chromatographic determinations, transfer the *n*-heptane layer to a small stoppered glass container containing solid anhydrous sodium sulfate and store in a refrigerator.

NOTE 3 Other transesterification method could be applied if results are equivalent.

4.5.6.2 Determination of fatty acid methyl esters

4.5.6.2.1 Instrument

Determine the methyl esters of the oil fatty acids using a gas chromatograph (4.3.2.12). Example of typical chromatograms and calibration graph are given in Annex E.

4.5.6.2.2 Calibration graph

Weigh a range of quantities of the blank reference oil which has been subjected to the same test conditions as the test specimens into 50 ml flasks. Weigh a range of oil quantities spanning the quantities of oil in the first 7 h extractions, taking no fewer than 4 standards.

Add 10,0 ml of the internal standard solution (triheptadecanoin) to each flask using a pipette, or the alternative quantity which has been added to the extraction flasks in 4.5.5. Remove the cyclohexane using a rotary evaporator or water bath. Subject these oil samples to the methyl ester preparation procedure described in 4.5.6.1.

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

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

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

Various methods for the construction of a calibration graph are suitable and the choice of method depends on the equipment and chromatographic column used. The following methods are acceptable for olive oil and the methods should be adapted if other vegetable oils are used.

Method A — Peak area method

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

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

In the event that the analysis of a blank test sample, see Annex B, has revealed an interference with one or more of the oil methyl esters, but not all of the peaks, then this peak or peaks shall be excluded from the calculation of the total area of the oil methyl esters. Calculate the ratio of the total area of the methyl esters originating from the oil using the peaks without interference and the area of the internal standard. Plot the ratios versus the weighed quantities of oil.

NOTE 2 A typical calibration graph is shown in Figure E.3.

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

For other vegetable oils, the main fatty acid methyl esters shall be determined and evaluated in a comparable way as above.

4.5.6.2.3 Determination of oil absorbed by test specimens

Inject into the gas chromatograph a suitable quantity from each of the n-heptane methyl ester solutions prepared from the residues containing the extracted oil (see 4.5.6.1). Inject in duplicate, as a minimum.