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Materiali in predmeti v stiku z živil - Plastika - 2. del: Preskusne metode za celotno migracijo v olivno olje

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

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

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

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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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Contents	Page
European foreword.....	3
1 Scope.....	4
2 Normative references.....	4
3 Terms and definitions	4
4 Test methods	5
4.1 Principle	5
4.2 Reagents	6
4.3 Materials and apparatus.....	7
4.4 Preparation of test specimens.....	10
4.5 Procedure.....	13
4.6 Expression of results.....	23
5 Test report.....	25
Annex A (normative) Contact time and temperature tolerances.....	27
Annex B (normative) Determination of the suitability of oil as the fatty food simulant and of triheptadecanoin as the internal standard.....	29
Annex C (normative) Determination of the need for sample conditioning.....	31
Annex D (normative) Determination of the need for sample conditioning and determination of the mass of moisture sensitive and/or with presence of volatiles specimens , by vacuum drying.....	32
Annex E (informative) Typical chromatograms and calibration graph	34
Annex F (informative) Precision data.....	37
Bibliography.....	38

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.

EN 1186-2:2022 (E)**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

EN 1186-2:2022 (E)

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 BF₃.

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

EN 1186-2:2022 (E)

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 $60\text{ °C} \pm 2\text{ °C}$. 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 dm² 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.

EN 1186-2:2022 (E)**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: twelve (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 dm² 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 dm² 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 dm² ± 0,01 dm² 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 dm².

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 dm². For articles with individual areas less than 1 dm², 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 dm² 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 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 [5], the method described by Mieth and Hoekstra [6], or any other suitable method [7]. Record the surface area of each test specimen.