SLOVENSKI STANDARD
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Materiali in predmeti v stiku z živili - Polimerni materiali - 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 - Matiere plastique - Partie 8: 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 in vegetable oils

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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (prEN 1186-2:2020) has been prepared by Technical Committee CEN/TC 194 “Utensils in contact with food”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

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.

This document will supersede 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.

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 up 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 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.

EN ISO 8442-2:1997, *Materials and articles in contact with foodstuffs — Cutlery and table hollowware — Part 2: Requirements for gold-plated cutlery (ISO 8442-2;1997)*

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

3 Terms and definitions

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

Note 1 to entry: Only terms not included in European Commission regulation on plastic materials are defined herein.

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

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

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

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

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 above 4 °C and below 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 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) 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; **t**his method is suitable for plastics in the form of films or sheets 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

4.2 Reagents

4.2.1 General

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

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

4.2.3 Extraction solvents

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

4.2.3.2 Diethylether

4.2.3.3 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.

4.2.4 Triheptadecanoin (glyceryl trimargarate) CAS No. 2438-40-6 solution, 2 mg/ml in cyclohexane

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

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

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

4.2.7 *n*-heptane

4.2.8 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 20°C ± 5°C

NOTE For 50 % relative humidity, 43 % *w/v* sulphuric acid solution in water is suitable and for 80 % relative humidity, 27 % *w/v* sulphuric acid solution is suitable.

The solutions should be freshly prepared by adding a weighed amount of acid to a suitable volume of water, cooling to room temperature and making up to the required volume. It is recommended that relative humidity and temperature be maintained during the conditioning period. Therefore, the containers should be placed in a 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.

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, capable of holding 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 micron film of cyanopropyl silicone;

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

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

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, 50 ml and 100 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 0,5 dm^2 of contact area for the test specimen

— Minimum of 1 dm^2/ 50 ml of surface to volume ratio for the test specimen

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

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

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 (see 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.

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 two 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 required 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 1dm^2 and a volume of 100 ml. Deviations should be recorded and explained. If necessary, a test specimen could be cut in pieces.