

SLOVENSKI STANDARD

SIST EN 1186-12:2002

01-september-2002

BUXca Yý U

SIST ENV 1186-12:1997

Materiali in predmeti v stiku z živili - Polimerni materiali - 12. del: Preskusne metode za celotno migracijo pri nizkih temperaturah

Materials and articles in contact with foodstuffs - Plastics - Part 12: Test methods for overall migration at low temperatures

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 12: Prüfverfahren für die Gesamtmigration bei tiefen Temperaturen

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 12: Méthodes d'essai pour la migration globale à basses températures

Ta slovenski standard je istoveten z: **EN 1186-12:2002**

ICS:

67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs
--------	--	---

SIST EN 1186-12:2002

en

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN 1186-12:2002

<https://standards.iteh.ai/catalog/standards/sist/71e9d725-2117-4081-94f6-1c4c72b64e5a/sist-en-1186-12-2002>

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 1186-12

April 2002

ICS 67.250

Supersedes ENV 1186-12:1995

English version

Materials and articles in contact with foodstuffs - Plastics - Part 12: Test methods for overall migration at low temperatures

Matériaux et objets en contact avec les denrées
alimentaires - Matière plastique - Partie 12: Méthodes
d'essai pour la migration globale à basses températures

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln
- Kunststoffe - Teil 12: Prüfverfahren für die
Gesamtmigration bei tiefen Temperaturen

This European Standard was approved by CEN on 4 January 2002.

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

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

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

SIST EN 1186-12:2002

<https://standards.iteh.ai/catalog/standards/sist/71e9d725-2117-4081-94f6-1c4c72b64e5a/sist-en-1186-12-2002>



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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

	page
Foreword.....	3
1 Scope	5
2 Normative references	5
3 Overall migration into dewaxed sunflower oil by total immersion	5
4 Overall migration into dewaxed sunflower oil by cell	8
5 Overall migration into dewaxed sunflower oil using a pouch.....	11
6 Overall migration into dewaxed sunflower oil by article filling.....	14
Annex ZA (informative) Relationship of this European Standard with Council Directive 89/109/EEC and Commission Directive 90/128/EEC and associated Directives.....	17
Bibliography	19

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN 1186-12:2002

<https://standards.iteh.ai/catalog/standards/sist/71e9d725-2117-4081-94f6-1c4c72b64e5a/sist-en-1186-12-2002>

Foreword

This document EN 1186-12:2002 has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2002, and conflicting national standards shall be withdrawn at the latest by October 2002.

This document supersedes ENV 1186-12:1995.

This European Standard is one of a series of methods of test for plastics materials and articles in contact with foodstuffs.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EC Directive(s).

For relationship with EC Directive(s), see informative annex ZA, which is an integral part of this document.

At the time of preparation and publication of this standard the European Union legislation relating to plastics materials and articles intended to come into contact with foodstuffs is incomplete. Further Directives and amendments to existing Directives are expected which could change the legislative requirements which this standard supports. It is therefore strongly recommended that users of this standard refer to the latest relevant published Directive(s) before commencement of any of the test or tests described in this standard.

EN 1186-12 should be read in conjunction with EN 1186-1, EN 1186-2, EN 1186-4, EN 1186-6 and EN 1186-8.

Further Parts of this standard have been prepared concerned with the determination of overall migration from plastics materials into food simulants.

Their titles are as follows:

EN 1186 Materials and articles in contact with foodstuffs – Plastics -

Part 1	Guide to the selection of conditions and test methods for overall migration
Part 2	Test methods for overall migration into olive oil by total immersion
Part 3	Test methods for overall migration into aqueous food simulants by total immersion
Part 4	Test methods for overall migration into olive oil by cell
Part 5	Test methods for overall migration into aqueous food simulants by cell
Part 6	Test methods for overall migration into olive oil using a pouch
Part 7	Test methods for overall migration into aqueous food simulants using a pouch
Part 8	Test methods for overall migration into olive oil by article filling
Part 9	Test methods for overall migration into aqueous food simulants by article filling
Part 10	Test methods for overall migration into olive oil (modified method for use in cases where incomplete extraction of dewaxed sunflower oil occurs)

EN 1186-12:2002 (E)

Part 11	Test methods for overall migration into mixtures of ¹⁴ C-labelled synthetic triglyceride
Part 13	Test methods for overall migration at high temperatures
Part 14	Test methods for 'substitute tests' for overall migration from plastics intended to come into contact with fatty foodstuffs using test media iso-octane and 95 % ethanol
Part 15	Alternative test methods to migration into fatty food simulants by rapid extraction into iso-octane and/or 95 % ethanol

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

iTeh STANDARD PREVIEW **(standards.iteh.ai)**

SIST EN 1186-12:2002

<https://standards.iteh.ai/catalog/standards/sist/71e9d725-2117-4081-94f6-1c4c72b64e5a/sist-en-1186-12-2002>

1 Scope

This Part of this European Standard specifies test methods for the determination of the overall migration into fatty food simulants from plastics materials and articles, by total immersion of test specimens in a fatty food simulant at temperatures from 5 °C, up to and including 20 °C, for selected times.

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

The fatty food simulant used in these test methods is dewaxed sunflower oil since, unlike olive oil, remains liquid at the lower test temperature.

The test method described is applicable to most types of plastics, although there are some plastics for which it is known not to be applicable.

2 Normative references

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

EN 1186-1:2002, *Materials and articles in contact with food – Plastics – Part 1: Guide to the selection of conditions and test methods for overall migration.* [SIST EN 1186-12:2002](https://standards.iteh.ai/catalog/standards/sist/71e9d725-2117-4081-94f6-187723292)

<https://standards.iteh.ai/catalog/standards/sist/71e9d725-2117-4081-94f6-187723292>

EN 1186-2:2002, *Materials and articles in contact with food – Plastics – Part 2: Test methods for overall migration into olive oil by total immersion.*

EN 1186-4:2002, *Materials and articles in contact with food – Plastics – Part 4: Test methods for overall migration into olive oil by cell.*

EN 1186-6:2002, *Materials and articles in contact with food – Plastics – Part 6: Test methods for overall migration into olive oil using a pouch.*

EN 1186-8:2002, *Materials and articles in contact with food – Plastics – Part 8: Test methods for overall migration into olive oil by article filling.*

3 Overall migration into dewaxed sunflower oil by total immersion

3.1 Principle

The overall migration from a sample of the plastics is determined as the loss in mass per unit of surface area intended to come into contact with foodstuffs.

The selection of the conditions of test will be determined by the conditions of use, see clauses 4, 5 and 6 of EN 1186-1:2002.

Test specimens of known mass are immersed in dewaxed sunflower oil for the exposure time, at temperatures from 5 °C up to and including 20 °C, then taken from the dewaxed sunflower oil, blotted to remove oil adhering to the surface, and reweighed.

EN 1186-12:2002 (E)

The specimens can usually retain absorbed dewaxed sunflower 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 hydrolysing the oil with potassium hydroxide. An internal standard, triheptadecanoin, is added prior to the extraction of the absorbed dewaxed sunflower 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 dewaxed sunflower 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 dewaxed sunflower oil is calculated by subtracting the mass of dewaxed sunflower oil retained by the test specimen from the mass of the test specimen after removal from the dewaxed sunflower 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 specimen and the overall migration is reported as the mean of a minimum of three determinations on separate test specimens.

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

This method includes variations that are applicable to certain plastics.

NOTE Before starting a migration exercise, the test sample should be examined for the presence of components interfering in the determination of the amount of dewaxed sunflower oil extracted, see 7.1 of EN 1186-2:2002. If an unacceptable amount of interference is present then suitability of one of the 'other fatty food simulants' should be examined, see annex A of EN 1186-2:2002 and 9.3 and 9.5 of EN 1186-1:2002. If an interference is present which would interfere with the triheptadecanoin internal standard an alternative internal standard should be used, see annex A of EN 1186-2:2002, and 9.3 of EN 1186-1:2002.

3.2 Reagents

SIST EN 1186-12:2002

[https://standards.iteh.ai/catalog/standards/sist/71e9d725-2117-4081-94f6-](https://standards.iteh.ai/catalog/standards/sist/71e9d725-2117-4081-94f6-1e4c7216c15a/en-1186-12:2002)

The reagents shall be as described in clause 4 of EN 1186-2:2002, except that olive oil, reference simulant D, as specified in 4.1 of EN 1186-1:2002 is replaced by dewaxed sunflower oil, simulant D.

3.3 Apparatus

The apparatus shall be as described in clause 5 of EN 1186-2:2002.

3.4 Preparation of test specimens

The test specimens shall be prepared as described in clause 6 of EN 1186-2:2002.

3.5 Procedure**3.5.1 General**

Determine the applicability of the method by carrying out the procedure described in annex A of EN 1186-2:2002. If prior tests have established that the method is applicable then annex A of EN 1186-2:2002 may be omitted.

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

3.5.2 Initial weighing of test specimens

Perform the initial weighing in accordance with 7.2 of EN 1186-2:2002.

3.5.3 Exposure to food simulant

Take six of the glass tubes (5.11 of EN 1186-2:2002), mark them for identification purposes. Measure 100 ml \pm 5 ml of dewaxed sunflower oil (4.1 of EN 1186-2:2002, see 3.2) into each tube by measuring cylinder and stopper the tube.

NOTE.1 If the procedure described in annex D of EN 1186-2:2002 is used, it can be necessary to dry all of the dewaxed sunflower oil used for the migration test, see D.3.2. of EN 1186-2:2002.

Alternatively mark the tubes for a volume of 100 ml and fill with dewaxed sunflower oil to the mark. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Two extra tubes with a minimum of 50 ml of dewaxed sunflower oil are required as blank simulant, if the procedure described in annex D of EN 1186-2:2002 is used. Place the six or eight tubes, and two empty tubes, in the thermostatically controlled oven or incubator (5.12 of EN 1186-2:2002) set at the test temperature. Leave until the dewaxed sunflower oil has attained the test temperature, using the thermometer or thermocouple to monitor the temperature. Take all tubes from the oven and place into four of the tubes containing dewaxed sunflower oil, weighed test specimens prepared as in clause 6 and conditioned if necessary. Stopper the tubes. Ensure that the test specimens are totally immersed in dewaxed sunflower oil; if they are not, then add either glass beads or glass rods (5.22 of EN 1186-2:2002) to raise the level of the dewaxed sunflower oil until total immersion is achieved.

NOTE 2 The dewaxed sunflower oil in the fifth tube is used as a reference standard in constructing the calibration graph and if the procedure described in annex D of EN 1186-2:2002 is used, as the third blank sample for Karl Fischer titrations. The dewaxed sunflower oil in the sixth tube is used to check the temperature of the oil. If glass beads or glass rods have been used to raise the level of the dewaxed sunflower oil to achieve total immersion, then similar glass beads or glass rods should be added to the sixth tube.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

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

NOTE 3 These two test specimens are used to check whether the sample loses mass from the evaporation of volatiles, such as water, solvents and oligomers, during the test period. If the vacuum drying procedure described in annex C of EN 1186-2:2002 is applicable these test specimens are not required as during the vacuum drying volatiles will have been removed from the test specimens.

<https://standards.iteh.ai/catalog/standards/sist/71e9d725-2117-4081-94f6-1c4c72b64e5a/sist-en-1186-12-2002>

Replace all eight or ten tubes in the thermostatically controlled oven or incubator set at the test temperature. This part of the operation should be carried out in the minimum time possible to prevent undue heat loss. Observe the temperature of the thermostatically controlled oven or incubator or the dewaxed sunflower oil (see NOTE 5) in the sixth tube and leave the tubes for the selected test period, taking into account the tolerances specified in Table B.1 of EN 1186-1:2002, after the dewaxed sunflower oil in the sixth tube has reached a temperature within the tolerance specified in Table B.2 of EN 1186-1:2002.

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

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

Take the tubes from the oven or incubator and immediately remove the test specimens from the tubes. For those specimens which have been in dewaxed sunflower oil, allow the oil to drain. Remove any adhering dewaxed sunflower oil by gently pressing between filter papers (5.13 of EN 1186-2:2002). Repeat the pressing procedure until the filter paper shows no spots of dewaxed sunflower 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 replace the test pieces on them.

NOTE 6 If the procedure described in annex D of EN 1186-2:2002 is followed, the tubes containing the oil should be retained. The tubes should be capped to prevent further change in the moisture content of the oil and the Karl Fischer determination of water content should be carried out as soon as possible.

3.5.4 Final weighing of test specimens

Perform the final weighing in accordance with 7.4 of EN 1186-2:2002.

EN 1186-12:2002 (E)

3.5.5 Extraction of absorbed dewaxed sunflower oil

Extract the dewaxed sunflower oil in accordance with 7.5 of EN 1186-2:2002.

3.5.6 Determination of extracted dewaxed sunflower oil

Determine the extracted dewaxed sunflower oil in accordance with 7.6 of EN 1186-2:2002.

3.6 Expression of results

3.6.1 Method of calculation

Express the results in accordance with 8.1 of EN 1186-2:2002.

3.6.2 Precision

See annex F of EN 1186-2:2002.

3.7 Test report

Prepare the test report in accordance with clause 9 of EN 1186-2:2002.

4 Overall migration into dewaxed sunflower oil by cell

4.1 Principle

The overall migration from a sample of the plastics is determined as the loss in mass per unit of surface area intended to come into contact with foodstuffs.

The selection of the conditions of test will be determined by the conditions of use, see clauses 3, 4 and 5 of EN 1186-1:2002.

Test specimens of known mass are exposed in a cell to dewaxed sunflower oil for the exposure time, at temperatures above 5 °C and below 20 °C, then taken from the cell, blotted to remove oil adhering to the surface, and reweighed.

The specimens will usually retain absorbed dewaxed sunflower 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 hydrolysing the oil with potassium hydroxide. An internal standard, triheptadecanoin, is added prior to the extraction of the absorbed dewaxed sunflower 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 dewaxed sunflower 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 dewaxed sunflower oil is calculated by subtracting the mass of dewaxed sunflower oil retained by the test specimen from the mass of the test specimen after removal from the dewaxed sunflower 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 specimen and the overall migration is reported as the mean of a minimum of three determinations on separate test specimens.

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

This method includes variations which are applicable to certain plastics.

NOTE Before starting a migration exercise, the test sample should be examined for the presence of components interfering in the determination of the amount of dewaxed sunflower oil extracted, see 7.1 of EN 1186-2:2002. If an unacceptable amount of interference is present then suitability of one of the 'other fatty food simulants' should be examined, see annex A of EN 1186-4:2002 and 9.3 and 9.5 of EN 1186-1:2002. If an interference is present which would interfere with the triheptadecanoin internal standard an alternative internal standard should be used, see annex A of EN 1186-4:2002, and 9.3 of EN 1186-1:2002.

4.2 Reagents

The reagents shall be as described in clause 4 of EN 1186-4:2002, except that olive oil, reference simulant D, as specified in 4.1 of EN 1186-1:2002 is replaced by dewaxed sunflower oil, simulant D.

4.3 Apparatus

The apparatus shall be as described in clause 5 of EN 1186-4:2002.

4.4 Preparation of test specimens

The test specimens shall be prepared as described in clause 6 of EN 1186-4:2002.

4.5 Procedure

4.5.1 General

Determine the applicability of the method by carrying out the procedure described in annex A of EN 1186-4:2002. If prior tests have established that the method is applicable then annex A of EN 1186-4:2002 may be omitted.

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

4.5.2 Initial weighing of test specimens

Perform the initial weighing in accordance with 7.2 of EN 1186-4:2002.

4.5.3 Exposure to food simulant

Take four type A cells (5.6 of EN 1186-4:2002), mark them for identification purposes. Place in the thermostatically controlled oven or incubator (5.9 of EN 1186-4:2002), which is set at the test temperature and leave until the test temperature has been attained.

Take five glass tubes (5.8 of EN 1186-4:2002), measure 125 ml \pm 5 ml of dewaxed sunflower oil (4.1 of EN 1186-4:2002) into each tube by measuring cylinder and stopper the tubes.

NOTE 1 If the procedure described in annex D of EN 1186-4:2002 is used, it can be necessary to dry all of the dewaxed sunflower oil used for the migration test, see D.3.2 of EN 1186-4:2002.

Alternatively mark the tubes for a volume of 125 ml and fill with dewaxed sunflower oil to the mark. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Two extra tubes with a minimum of 50 ml of dewaxed sunflower oil are required as blank simulant, if the procedure in annex D of EN 1186-4:2002 is used. Place the five or seven tubes, and two empty tubes, in the thermostatically controlled oven or incubator (5.9 of EN 1186-4:2002) set at the test temperature. Leave until the dewaxed sunflower oil has attained the test temperature, using the thermometer or thermocouple to monitor the temperature.

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

Remove four tubes containing 125 ml of dewaxed sunflower oil from the thermostatically controlled oven or incubator or refrigerator and transfer the dewaxed sunflower oil from each tube to each of the cells through the filler