SLOVENSKI STANDARD
SIST ENV 1186-13:2000

01-april-2000

Materiali in predmeti v stiku z živili - Plastiļne mase - 13. del: Preskusne metode za celotno migracijo pri visokih temperaturah

Materials and articles in contact with foodstuffs - Plastics - Part 13: Test method for overall migration at high temperatures

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 13: Prüfverfahren für die Gesamtmigration bei hohen Temperaturen

Matériaux et objets en contact avec les denrées alimentaires - Matiere plastique - Partie 13: Méthodes d'essai pour la migration globale a hautes températures

Ta slovenski standard je istoveten z: **ENV 1186-13:1999**

ICS:

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

SIST ENV 1186-13:2000 **en**

EUROPEAN PRESTANDARD
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ENV 1186-13

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English version

Materials and articles in contact with foodstuffs - Plastics - Part 13: Test method for overall migration at high temperatures

Matériaux et objets en contact avec les denrées alimentaires - Matière plastique - Partie 13: Méthodes d'essai pour la migration globale à hautes températures

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunststoffe - Teil 13: Prüfverfahren für die Gesamtmigration bei hohen Temperaturen

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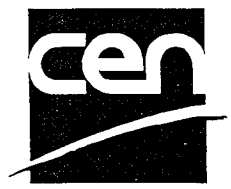

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

Further Parts of this prestandard have been prepared, and others are in preparation, concerned with the determination of overall migration from plastics materials into food simulants

Their titles are as follows:

ENV 1186-1	Guide to the selection of conditions and test methods for overall migration
ENV 1186-2	Test methods for overall migration into olive oil by total immersion
ENV 1186-3	Test methods for overall migration into aqueous food simulants by total immersion
ENV 1186-4	Test methods for overall migration into olive oil by cell
ENV 1186-5	Test methods for overall migration into aqueous food simulants by cell
ENV 1186-6	Test methods for overall migration into olive oil using a pouch
ENV 1186-7	Test methods for overall migration into aqueous food simulants using a pouch
ENV 1186-8	Test methods for overall migration into olive oil by article filling
ENV 1186-9	Test methods for overall migration into aqueous food simulants by article filling
ENV 1186-10	Test methods for overall migration into olive oil (modified method for use in cases where incomplete extraction of olive oil occurs)
ENV 1186-11	Test methods for overall migration into mixtures of ^{14}C-labelled synthetic triglyceride
ENV 1186-12	Test methods for overall migration at low temperatures

Further Parts in preparation are as follows:

ENV 1186-14	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

ENV 1186-13 should be read in conjunction with EN 1186-1.

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

0 Introduction

Migration testing with olive oil at high temperatures introduces a number of analytical difficulties. These include possible oxidation of oil at elevated temperatures and the hazard to personnel working with hot oil. Replacement of olive oil by an appropriate adsorbent material, in principal, can solve or reduce these problems and offers further experimental advantages.

1 Scope

This Part of this European Prestandard describes a substitute procedure for the determination of overall migration into olive oil from articles which are intended to come into contact with foodstuffs at elevated temperatures from 100 °C up to and including 175 °C. In order to circumvent a number of analytical difficulties caused by the food simulant olive oil at high temperatures the mass of components adsorbed on modified polyphenylene oxide (MPPO) is taken as a measure for the assessment of the overall migration into olive oil.

NOTE 1: A comparative migration test carried out with polypropylene and polyethylene terephthalate high temperature application containers as test samples at conditions 2 h at 100 °C and 2 h at 175 °C, respectively, in contact with ^{14}C-labelled synthetic triglyceride and MPPO provided test results comparable within the analytical tolerance of the methods.

NOTE 2: To obtain reproducible and repeatable results it may be necessary to measure the temperature of the test specimen before starting the migration period. An appropriate method for measuring the temperature of the test specimen has to be established.

The described method is most suitable for food contact articles in the form of sheets and films, but can also be applied to a wide range of articles and containers. The method has been used on plastic cookware and trays for microwave as well as conventional oven heating, and plastics sheets and films as well as laminated and coated papers and cartons at temperatures up to 175 °C.

2 Normative references

This European Prestandard 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 Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 1186-1:1994 Guide to the selection of conditions and test methods for overall migration

3 Principle

The surface of the article to be tested is covered with modified polyphenylene oxide and held at the desired time-temperature test conditions where the maximum temperature applicable is 175 °C. The heating takes place principally in a conventional oven even when the samples are for use in a microwave oven. The exposure is followed by a extraction of the adsorbent using diethyl ether. Finally, the extract is concentrated to dryness using a nitrogen stream and the residue remaining is determined gravimetrically. Modified polyphenylene oxide is a porous polymer with a high molecular weight, 500 000 to 1 000 000, a very high temperature stability (Tmax = 350 °C), a high surface area and a low specific mass (0,23 g/cm^3).

4 Reagents

All reagents shall be of recognized analytical quality, unless otherwise specified.

4.1 Diethylether of 99,8 % purity and stabilized with 0,0005 % 2,6-di-*tert.* butyl-p-cresol (BHT)

4.2 Modified polyphenylene oxide (MPPO), 60 mesh to 80 mesh

Gas chromatograms obtained from extracts of new commercial MPPO material has shown that unacceptable high levels of impurities may be already present.

Therefore, Soxhlet extraction using diethylether is obligatory for complete purification of the MPPO prior to the first use in this test procedure. The extraction is carried as follows: Place MPPO in a soxhlet cartridge and extract for 6 h with diethylether. Spread the MPPO in a Petri dish of suitable diameter and place the dish in a fumehood. Allow the ether to evaporate while frequently mixing with a glass rod. Then place the dish in an oven at 160 °C for 6 h. After heating store the MPPO, if not needed immediately, in a closed conical flask.

NOTE 1: Heating of MPPO saturated with diethylether may be explosive. Therefore, ensure that diethylether is completely evaporated before drying at 160 °C.

NOTE 2: MPPO cleaned in this way can be used repeatedly.

NOTE 3: MPPO is powdery and lightweight and is readily blown about by air currents. When drying MPPO or carrying out the exposure in a forced air oven, set the oven to low and cover dishes to prevent the MPPO from blowing about.

4.3 Nitrogen, purity 99,999 %

5 Apparatus

5.1 Cutting implement, scalpel, scissors, sharp knife or other suitable device

5.2 Rule, graduated in mm, with an accuracy of 0,1 mm

5.3 Brush

5.4 Analytical balance capable of determining a change in mass of 0,1 mg.

5.5 Oven or incubator, thermostatically controlled and capable of maintaining the set temperature within the tolerances specified in annex A

NOTE: In case of an oven with a ventilating system the ventilation rate should be switched to low.

5.6 Glass Petri dishes with an internal diameter of 140 mm, heat resistant

5.7 Glass rings, with an internal diameter of 125 mm and an external diameter of approximately 130 mm

5.8 Erlenmeyer flasks, glass-stoppered, capacity of 300 ml.

5.9 Glass filter funnels.

5.10 Folded filter, with a diameter of 125 mm.

5.11 Glass vials, capacities 10 ml and 100 ml.

5.12 Dropping pipettes with dropper teats.

5.13 Lint-free cloth.

5.14 Glass plates, to cover the dishes or trays.

5.15 Erlenmeyer flask, glass-stoppered to wash the MPPO

NOTE 2: The size of the flask depends on the mass of MPPO that is used (see table 1).

5.16 Apparatus to blow off solvent with nitrogen.

6 Sample preparation

6.1 General

It is essential that test specimens are clean and free from surface contamination (many plastics can readily attract dust due to static charges). Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use see 9.1 of EN 1186-1:1994. Minimise handling of the samples and where necessary, wear cotton gloves.

6.2 Number of test specimens

Three specimens are required for the test.

6.3 Films and sheets

Lay the sample on a cutting slab. Take the glass ring (5.7) and place on the surface of the sample. Cut out the test specimen by cutting round the outside of the glass ring, using the cutting implement.

NOTE: Taking the inner diameter of the glass ring into account the effective contact area obtained in this way is 1,22 dm²

6.4 Containers and other articles

Articles do not have to be cut if it is possible to cover these samples with pieces of glass. In this case, determine the flat bottom area of the article to find the required mass of adsorbent (see 6.5).

6.5 Preparation of MPPO

To cover the food contact surface sufficiently, 4 g MPPO per square decimetre of surface area of the test specimen is required.

7 Procedure

7.1 Exposure to MPPO

For flexible thin film and sheet materials (6.3), take three Petri dishes (5.6) and place a prepared test specimen (1,22 dm² effective contact area) into each dish, stabilize the test specimen with a glass ring (5.7) and place 4,8 g MPPO evenly on the surface of each test specimen, inside the glass ring. Close the Petri dishes.

For rigid containers and other articles (6.4) which are to be tested whole, cover the flat bottom of the article with the required amount of MPPO . Calculate the required amount of MPPO according to the flat bottom food contact surface area which can be covered with MPPO, taking 4 g MPPO per square decimetre of surface area. Weigh the appropriate mass of MPPO with an accuracy of ± 0,1 g and place it on the flat bottom area of the test

specimen. Cover each of the three articles with a glass plate (5.14).

NOTE 1: When the test sample, prepared as described, is placed in the oven the time required to reach the intended temperature may be significant when compared to the intended exposure time and the allowed tolerances on contact time and contact temperature. Therefore it may be necessary to include a procedure for the control of the time and temperature of the contact of the test specimens with the MPPO, in order to achieve reproducible and repeatable results

NOTE 2: In case of articles of irregular geometry and with no flat areas, a corresponding way has to be found to expose the food contact surface to MPPO. Possible solutions are to cut appropriate parts from the article and cover or mix them with MPPO, using the conventional mass of MPPO to food contact area ratio (6.5). If necessary, higher amounts of MPPO should be used to ensure complete contact between the test specimen and MPPO.

For the blank determination, take an empty Petri dish and place in it the same mass of MPPO as was placed on each test specimen and cover the dish. Set the oven (5.5) at the required test temperature and observe the temperature. When the oven has reached the test temperature place the prepared test specimens in the oven. Observe the temperature and leave the test samples for the selected period of time after the temperature of the oven has reached a temperature within the permitted tolerance for the test temperature, see annex A for permitted tolerances on the test times and temperatures .

Remove the test specimens from the oven and allow them to cool to room temperature without removing the glass covers.

NOTE 3: Cooling to room temperature takes approximately half an hour.

7.2 Determination of the migrating substances

Transfer the MPPO adsorbent into the Erlenmeyer flask (5.8) with the aid of a funnel (5.9). If necessary use the brush (5.3) for complete transfer of the MPPO.

Calculate, by reference to table 1, the volume of diethylether needed for extraction of the MPPO adsorbent.

Table 1: Volumes of diethylether needed for the extraction of MPPO

Mass of MPPO absorbent grams	Volume of diethylether for the 1st extraction ml	Volume of diethylether for the 2nd extraction ml	Volume of diethylether for the 3rd extraction ml
1,0	20	30	30
2,0	30	30	30
3,0	35	30	30
4,0	45	30	30
5,0	50	30	30
6,0	55	30	30
7,0	60	30	30
8,0	70	30	30
9,0	80	40	40
10,0	90	40	40
15,0	120	50	50
20,0	160	60	60