SLOVENSKI STANDARD
SIST EN 14481:2003

01-september-2003

Materiali in predmeti v stiku z ģivili - Polimerni materiali ï Preskusne metode za ugotavljanje stika z maģļ obo

Materials and articles in contact with foodstuffs - Plastics - Test methods for the determination of fatty contact

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunstoffe - Prüfverfahren zur Bestimmung des fettigen Kontaktes

Matériaux et articles en contact avec les denrées alimentaires - Plastiques - Méthodes d'essai pour la détermination d'un contact gras

Ta slovenski standard je istoveten z: **EN 14481:2003**

ICS:

67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs
83.080.01	Polimerni materiali na splošno	Plastics in general

SIST EN 14481:2003 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14481

May 2003

ICS 67.250; 83.080.01

English version

Materials and articles in contact with foodstuffs - Plastics - Test methods for the determination of fatty contact

Matériaux et articles en contact avec les denrées alimentaires - Plastiques - Méthodes d'essai pour la détermination d'un contact gras

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Kunstoffe - Prüfverfahren zur Bestimmung des fettigen Kontaktes

This European Standard was approved by CEN on 9 January 2003.

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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.

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

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

Ref. No. EN 14481:2003 E

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Foreword

This document (EN 14481:2003) 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of an identical text or by endorsement, at the latest by November 2003, and conflicting national standards shall be withdrawn at the latest by November 2003.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This European Standard contains an informative Annex A.

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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

For certain specified food types, if it can be demonstrated, by means of an appropriate test, that there is no 'fatty contact' with the plastic, the migration test with simulant D (olive oil or alternatives) may be dispensed with. The method described here can be used to determine whether a food makes fatty contact with plastics.

NOTE 1 The food types are specified in Council Directive 85/572/EEC [1].

NOTE 2 Guidance on the selection of conditions and test methods for overall migration, and the test methods for the specific migration of substances from plastics into food and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants is given in EN 1186-1 and EN 13130-1 respectively

1 Scope

This Part of this European Standard specifies a test method to determine whether there is fatty contact and is applicable to all foods. Testing some foods can require modifications to the method. The method is applicable to contact situations from -20 °C to 100 °C.

2 Principle

Fatty contact between a food and a plastic material or article is determined by placing food in contact with a polyethylene film containing a fat-soluble fluorescent dye. After exposure, the dye is extracted from the food and determined by high performance liquid chromatography (HPLC) with fluorescence detection. The method employs 1,6-diphenyl-1,3,5-hexatriene as internal standard. The degree of transfer is related to the extent of fatty contact made, and is used to determine whether simulant D be employed or not in subsequent tests of plastics intended to come into contact with that food.

The food used in the test should be the same as, or similar in composition to, the food which will come into contact with the plastic in actual use. To allow for possible difficulties encountered in obtaining reproducible film to food contact, the test is carried out on five replicate samples of the food.

3 Reagents

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

3.1 Low density polyethylene film containing 1,4-diphenyl-1,3-butadiene, fluorescent dye, at 17,3 μg/dm^2 ± 0,9 μg/dm^2, in the form of a lay-flat tube of width 20 cm, e.g. the certified reference material BCR.593, Plastic film E, certified on the basis of the mass fraction of DPBD (12,58 mg/kg ± 0,63 mg/kg) and on the film thickness (149,7 μm ± 0,7 μm).

3.2 1,4-diphenyl-1,3-butadiene (DPBD)

3.3 1,6-diphenyl-1,3,5-hexatriene (DPHT)

NOTE As DPBD and DPHT are slightly light sensitive, the film and standards should be stored with the exclusion of light.

3.4 n-Hexane, C_6H_{14}, HPLC grade

NOTE As n-hexane is considered to be harmful by inhalation, ingestion and through skin contact, correct safety procedures should be followed at all times when using this solvent.

3.5 Dichloromethane, CH_2Cl_2, HPLC grade

NOTE As dichloromethane is considered to be harmful by inhalation, ingestion and through skin contact and as a potential carcinogen, correct safety procedures should be followed at all times when using this solvent.

4 Apparatus

4.1 Heat sealer suitable for sealing the polyethylene film (3.1)

4.2 Dispersion homogenizer

4.3 Centrifuge, bench-top

4.4 Centrifuge tubes, 10 ml to 20 ml capacity

4.5 Filter paper, 15,0 cm diameter circles, Whatman Grade 2 or equivalent

4.6 Volumetric flasks, 25 ml, 50 ml and 100 ml capacity

4.7 Glass vials, 2 ml and 20 ml capacity with screw or crimp cap closures and polytetrafluoroethylene (PTFE) lined sealing disks

4.8 Pasteur pipettes

4.9 Syringes, glass-barrelled, with needles, 50 µl, 100 µl, 500 µl, 1 ml, 2,5 ml and 10 ml capacity

4.10 Filter funnels, glass

4.11 HPLC equipment

NOTE With a 20 µl injection loop and a pumping system capable of generating a gradient at 0,5 ml/min of n-hexane (held for 7 min) switched to dichloromethane over 1min (held for 5 min), returned to n-hexane over 1 min and then equilibrated for 6 min.

4.11.1 Fluorescence detector, with excitation wavelength of 346 nm and emission wavelength of 374 nm and linked to a chromatographic recorder and integrator

4.11.2 HPLC column

NOTE The following column has been found suitable. Stainless steel, 100 mm x 3 mm internal diameter, silica, 5µm particle size.

4.12 Vacuum line with attached needle

4.13 Beakers, glass, tall form, 400 ml capacity

4.14 Thermostatically controlled cabinet(s) capable of maintaining a temperature of -20 °C ± 2 °C, 5 °C ± 1 °C, 25 °C ± 1 °C and 70 °C ± 2 °C.

4.15 Food processor

4.16 Volumetric glass pipette, 5 ml capacity

5 Preparation of standards and samples

5.1 Preparation of standards

5.1.1 Stock solution of DPBD at 1mg/ml

Weigh 0,050 g ± 0,001 g of DPBD (3.2) into a 50 ml volumetric flask and make to the mark with n-hexane (3.4).

5.1.2 Working standard solution of DPBD in n-hexane at 1 µg/ml

Place approximately 50 ml of n-hexane into a 100 ml volumetric flask. Add, by syringe, 100 µl of the stock solution (5.1.1). Make to the mark with n-hexane.

5.1.3 Stock solution of DPHT at 1mg/ml

Weigh 0,050 g ± 0,001 g of DPHT (3.3) into a 50 ml volumetric flask and make to the mark with dichloromethane (3.5).

5.1.4 Calibration solution of DPHT in n-hexane at 100 µg/ml

Place approximately 30 ml of n-hexane into a 50 ml volumetric flask. Add, by pipette (4.16), 5 ml of the stock solution (5.1.3). Make to the mark with n-hexane.

NOTE As DPBD and DPHT are sensitive to light, standard solutions should be stored with the exclusion of light.

5.2 Preparation of samples

5.2.1 The reference film is in the form of a lay-flat polyethylene tube wound on a cardboard core, wrapped in aluminium foil and then heat-sealed in a barrier pack. Unpack the film. Unroll the outer turn from the reel, cut off and discard. Cut a 220 mm length of the film. Use the heat sealer (4.1) to close one of the cut edges. Place the food sample in the pouch so formed and then seal the opening to give a 200 mm x 200 mm pouch.

Prepare a further four replicate pouches containing food.

NOTE The quantity of food used and the plastic-to-food contact, should be representative of that in actual use. Some foods can need modification to give representative contact e.g. for non-homogeneous foods with differing surfaces, it could be necessary to cut the food such that only the food surface which will contact the plastic in actual use comes into contact with the polyethylene film. Alternatively, the non-representative surface(s) can be shielded from contact by introducing an interlayer of n-hexane-washed aluminium foil between the surface(s) and the film.

Where a food is non-homogeneous and an actual use-size portion will not fit in the pouch, it is suggested that the food sample be modified by cutting or otherwise reducing in size, ensuring that the portion taken for exposure is representative of the food as a whole. For example, for a cut of fish, the ratio of skin to lean flesh in the cut portion should be the same as that for the whole portion. Caution should be taken to ensure that cut edges do not modify the nature of the food. If necessary, they may be shielded from contact by the use of aluminium foil.

For homogeneous foods such as powders and liquids, a 100 mm x 100 mm pouch can be used and the quantity of food reduced accordingly.

5.2.2 After sealing the food in the bag, insert the vacuum needle (4.12) at one corner and use vacuum suction to remove the trapped air. When the majority of the air is removed and the food sample makes good contact with the film, withdraw the needle slightly and reseal the pouch across the corner without allowing ingress of air.

NOTE The contact between the food and the test plastic should be similar to that which occurs in actual use. Samples should not be evacuated so strongly that the food is crushed and abnormal contact is obtained.

5.2.3 Using a pen mark the outline of the food on both walls of the pouch. Measure the area within this outline. This is the contact area.

5.2.4 Place the filled pouches in the thermostatically controlled cabinet (4.14). Expose for the required time period and temperature chosen according to Table 1.

Table 1 — Test times and temperatures

Intended application	**Test time and temperature**
Contact up to 100 °C	30 $^{+1}_{0}$ min at 70 °C ± 2 °C
Ambient storage	30 $^{+1}_{0}$ d at 25 °C ± 1 °C
Refrigerated storage	168 $^{+6}_{0}$ h at 5 °C ± 1 °C
Frozen storage	28 $^{+1}_{0}$ d at -20 °C ± 2 °C

NOTE 1 Indications only. For specific applications, the time and temperature of the test can be varied from those shown in table 1, to correspond more closely with those expected for that application, where known.

NOTE 2 As DPBD is slightly light sensitive, the filled pouches should be tested in subdued light. Subsequent extraction procedures should be conducted away from direct sunlight and the final extracts should be stored either in amber vials or with the exclusion of light.