SLOVENSKI STANDARD
SIST EN 12607-2:2000

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Bitumen in bitumenska veziva - Določ evanje odpornosti proti strjevanju pod vplivom toplote in zraka - 2. del: Metoda TFOT

Bitumen and bituminous binders - Determination of the resistance to hardening under the influence of heat and air - Part 2: TFOT method

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Beständigkeit gegen Verhärtung unter Einfluß von Wärme and Luft - Teil 2: TFOT-Verfahren

Bitumes et liants bitumineux - Détermination de la résistance au durcissement sous l'effet de la chaleur et de l'air - Partie 2: Méthode TFOT

Ta slovenski standard je istoveten z: **EN 12607-2:1999**

ICS:

75.140	Voski, bitumni in drugi naftni proizvodi	Waxes, bituminous materials and other petroleum products
91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

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English version

Bitumen and bituminous binders - Determination of the resistance to hardening under the influence of heat and air - Part 2: TFOT Method

Bitumes et liants bitumineux - Détermination de la résistance au durcissement sous l'effet de la chaleur et de l'air - Partie 2: Méthode TFOT

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Beständigkeit gegen Verhärtung unter Einfluß von Wärme and Luft - Teil 2: TFOT-Verfahren

This European Standard was approved by CEN on 5 September 1999.

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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 Central Secretariat has the same status as the official versions.

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

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

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of an identical text or by endorsement, at the latest by April 2000, and conflicting national standards shall be withdrawn at the latest by April 2000.

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, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

This European Standard is based on ASTM D 1754-94.

This draft European standard EN 12607 consists of the following parts under the general title “Bitumen and bituminous binders – Determination of the resistance to hardening under the influence of heat and air”

Part 1: RTFOT method
Part 2: TFOT method
Part 3: RFT method

In this standard, annex A is normative.

1 Scope

This European Standard describes a method for measuring the combined effects of heat and air on a film of bitumen or bituminous binder, simulating the hardening which a bituminous binder undergoes during mixing in an asphalt mixing plant

The method is referred to as TFOT i.e. Thin Film Oven Test..

WARNING The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative References

This European Standard incorporates by dated or 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 or 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.

EN 58, *Sampling bituminous binders*.

EN 1425, *Bitumen and bituminous binders - Characterisation of perceptible properties*.

EN 12594, *Bitumen and bituminous binders - Preparation of test samples*.

EN 12595, *Bitumen and bituminous binders - Determination of kinematic viscosity*.

3 Principle

A film of bituminous binder is heated in an oven to a specified temperature for a given period of time.

The effects of heat and air are determined on the basis of the change in mass (expressed as a percentage) and in the change in the bituminous binder's properties such as kinematic viscosity (EN 12595) before and after the period in the oven.

NOTE : Penetration (EN 1426) and softening point (EN 1427) can be measured on harder grades before and after oven treatment at 163 °C.

4 Apparatus

Usual laboratory apparatus and glassware, together with the following :

4.1 Oven, electrically heated and conforming to the performance requirements indicated in table 1 for operating temperatures up to 180 °C.

The oven shall be rectangular with minimum interior height, width and depth of 330 mm. The front door shall be hinged and tightly fitted, and have a clear opening substantially of the same height and width as the interior of the oven. The window on the door shall at least be

100 mm by 100 mm, with two sheets of glass separated by an air space. The window shall permit an unobstructed view of the interior of the oven so that the control thermometer, located as specified in 6.1 may be read without opening the door.

NOTE 1 The oven can alternatively be provided with an inner glass door, through which the thermometer can be read on opening the outer door momentarily.

The oven shall be ventilated by convection currents of air. To this end, the oven shall have air inlets and outlets for the evacuation of hot gases, the requirements of table 1 being met.

NOTE 2 These inlets and outlets can be of different size and arrangement provided the requirements in table 1 are met.

Table 1 - Performance requirements for Thin Film Oven

Characteristic	Requirement
Deviation from specified test temperature throughout testing chamber during 24h period for the differential between ambient and test temperature, when more than 50 °C, maximum, % of differential	5
Time constant, maximum, s	720
Rate of ventilation of testing chamber, air changes per hour, minimum	10

NOTE 3 These performance requirements are extracted from ASTM E 145, type IB (gravity convection ventilated oven) *Specification for gravity-convection and force ventilated ovens*

The oven shall be provided with a metal circular shelf with minimum diameter of 250 mm. The shelf shall provide a flat surface for support of the containers without blocking all air circulation through the shelf when the containers are in place. The shelf shall be suspended by a vertical shaft and centred with respect to the horizontal interior of the oven. The shelf shall be mechanically driven to rotate at the rate of 5,5 min^{-1} ± 1,0 min^{-1}. The shelf shall be vertically located as close to the centre of the oven as allowed by the requirements of 6.1 regarding thermometer placement.

NOTE 4: Minimum size ovens require two containers. Larger ovens, having proportionally larger shelves to accommodate a greater number of containers can be suitable. Under no circumstances should more than one shelf, properly centred, be used in an oven.

4.2 Thermometers, solid stem, as specified in annex A.

Other temperature measuring devices may be used instead of mercury stem thermometers. However, the mercury stem thermometer is the reference device. Therefore any alternative device employed shall be calibrated so as to provide the same readings as would be provided by the mercury stem thermometer, recognising and allowing for the fact of changed thermal response times compared with the mercury thermometer.

NOTE When measuring and controlling nominally constant temperatures, as in this test method, alternative devices can indicate greater cyclic variations than mercury thermometers, to an extent depending on the cycle time of heating and the power of the controlled heat input.

4.3 Container, (in which the sample of bituminous binder is subjected to the test), cylindrical pan 140 mm ± 1 mm in inside diameter and 9,5 mm ± 0,5 mm deep with a flat bottom, stainless steel or aluminium, thickness 0,6 mm ± 0,1 mm to 1,0 mm ± 0,1 mm.

NOTE 1 50 ml of sample in this container will give a film thickness of approximately 3,2 mm.

NOTE 2 Containers have a tendency to become warped or bent with use. Although a small amount of warping does not significally affect results, it is advisable to eliminate damaged pans.

4.4 Balance, accurate to ± 10 mg, readable to 1 mg.

5 Sampling

5.1 General

Take all necessary safety precautions and ensure that the test sample is representative of the laboratory sample from which it is taken (see EN 58). Ensure that the laboratory sample is homogeneous and is not contaminated (see EN 1425).

5.2 Test sample preparation

Prepare the test sample in accordance with EN 12594. Remove sufficient quantity of the laboratory sample to perform tests to establish the characteristics to be measured on the bituminous binder before and after the TFOT hardening test, if necessary using a warmed knife, and transfer it to a suitable container.

The sample shall be free of water. Heat the sample in its container with a loosely fitted cover in an oven to a fluid condition not exceeding 10 °C below the test temperature, for the minimum time necessary to ensure that the sample is completely fluid. Homogenize the sample by stirring.

5.3 Initial characteristics

Determine the initial characteristics of the bituminous binder using an appropriate test method, according to the grade in question, e.g. the kinematic viscosity at 60°C (EN 12595), ν_1.

6 Procedure

6.1 Test conditions

Ensure that the oven (4.1) is level so that the shelf rotates in a horizontal plane with a maximum tilt during rotation of not more than 3° from the horizontal. Determine the temperature of the oven with the specified thermometer (4.2) in a vertical position at a point equidistant from the center and the outer edge of the shelf and the bottom of the thermometer bulb approximately 6 mm above the top of the shelf.

NOTE The reference temperature of the test is 120 °C ± 1 °C, corresponding to the mixing temperature of soft paving grade bitumens. However, it is possible to perform the test at other temperatures e.g. 163 °C ± 1 °C for harder bitumen grades.

6.2 Determination

Weigh separately metal containers (4.3) to the nearest 1 mg reading (M_0).
Pour 50,0 g ± 0,5 g of the sample into each of two or more of the containers. Allow the samples to cool to room temperature and weigh them separately to the nearest 1 mg (M_1).

With the oven at the test temperature ± 1 °C, rapidly place the containers with the sample on the circular shelf, close the oven, and commence rotating the shelf. Maintain the sample in the oven for 5 h from the time the test temperature reaches 1 °C below the test temperature. Ensure that the total time that a sample is in the oven is not more than 5 h 15 min. At the end of the heating period, remove the samples from the oven.

Cool to room temperature, weigh to the nearest 1 mg, (M_2), and calculate the change in mass of the bituminous binder in each container.

Do not test different samples in the same oven at the same time.

NOTE When a complete test can not be completed on the same day, weigh the residues and store them overnight before reheating.

After weighing the samples, place them back on the shelf in the oven at the test temperature. Close the oven and rotate the shelf for 15 min. Remove the samples and transfer the material from each pan into a 250 ml container preheated to the test temperature. Stir the combined residues thoroughly, heating the container if necessary.

6.3 Measurement of properties

Measure the properties chosen in 5.3 within 72 h, and avoid reheating the residue more than once e.g. the kinematic viscosity of the residue at 60 °C (EN 12595) ν_2.

NOTE Excessive reheating of the sample can influence the test results obtained.

7 Calculation

Calculate the change in physical properties or characteristics after the hardening procedure as follows:

. $\Delta\nu$, ratio of kinematic viscosities at 60°C = $\frac{\nu_2}{\nu_1}$

. ΔM, percentage of change in mass = $100 \times \frac{(M_2 - M_1)}{(M_1 - M_0)}$

where

ν_1 is the kinematic viscosity of the sample before hardening, in millimetres squared per second;
ν_2 is the kinematic viscosity of the residue, in millimetres squared per second;
M_0 is the mass of the empty container, in grams;
M_1 is the mass of the container with the sample before the test, in grams;
M_2 is the mass of the container with the sample after the test, in grams.

8 Expression of results

Express the ratio of kinematic viscosities to the nearest 0,01. Report the values of initial properties and residue properties.

Express a loss in mass as a negative percentage change and a gain in mass as a positive percentage change.

Express the percentage change in mass as the average of minimum 2 valid determinations to the nearest 0,01%.

9 Precision

9.1 Repeatability

The difference between two test results, obtained by the same operator, with the same apparatus, under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in table 2 in only one case in twenty.

9.2 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in table 2 in only one case in twenty.

Table 2 - Precision

Characteristics	Repeatability *r*	Reproducibility *R*
Change in mass TFOT 120 °C [1)]		
≤ 0,1% (*m/m*) absolute	0,02 % (*m/m*)	0,14 % (*m/m*)
> 0,1% (*m/m*) absolute	8,0 % of mean	38 % of mean
Ratio of dynamic viscosity at 60 °C [1)]		
ratio < 1,5	6 % of mean	16 % of mean

1) These figures are based on analysis of data from 16 laboratories on three samples of soft paving grade bitumens with viscosity ratio < 1,5

NOTE These figures are not automatically applicable at other conditions and for modified bitumens and for industrial bitumens. For modified bitumens they should only be used for guidance, until criteria data are available.

10 Test report

The test report shall contain at least the following information :

a) the type and complete identification of the sample under test;
b) a reference to this part of the European Standard ;
c) the test temperature;
d) the results obtained (see clause 8) ;
e) any deviation, by agreement or otherwise, from the procedure specified ;
f) the date of the test.