

SLOVENSKI STANDARD SIST EN 12607-2:2007

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Nadomešča:

SIST EN 12607-2:2000

Bitumen in bitumenska veziva - Določanje odpornosti proti otrjevanju 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 und Luft - Teil 2: TFOT-Verfahren

Bitumes et liants bitumineux - Détermination de la résistance au durcissement sous l'effet de la chaleur étide l'airud Partie 2 lo Méthode TFO Toe-b687-47bb-bc38-539bcec7b4a4/sist-en-12607-2-2007

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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 und Luft - Teil 2: TFOT-Verfahren

This European Standard was approved by CEN on 3 February 2007.

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 CEN 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 CEN 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

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Coi	ntents	Page
Fore	eword	3
1	Scope	
2	Normative references	4
3	Principle	4
4	Apparatus	4
5	Sampling	6
6	Procedure	
7	Calculation	7
8	Expression of results	7
9	Precision	8
10	Test report	8
	Annex A (normative) Characteristics of thermometer	
	(standards.iteh.ai)	

<u>SIST EN 12607-2:2007</u> https://standards.iteh.ai/catalog/standards/sist/f2bf940e-b687-47bb-bc38-539bcec7b4a4/sist-en-12607-2-2007

Foreword

This document (EN 12607-2:2007) has been prepared by Technical Committee CEN/TC 336 "Bituminous binders", the secretariat of which is held by AFNOR.

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 September 2007, and conflicting national standards shall be withdrawn at the latest by September 2007.

This document supersedes EN 12607-2:1999.

This 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

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

SIST EN 12607-2:2007

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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 that 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 — 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 to determine the applicability of regulatory limitations prior to use.

If there is a likelihood of volatile components being present in a binder, this procedure should not be used. It should not be used for cutback bitumen or bituminous emulsions before these products have been stabilised, e.g. in accordance with EN 14895.

2 Normative references

The following referenced documents are indispensable for the application 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 58, Bitumen and bituminous binders - Sampling bituminous binders

EN 1425, Bitumen and bituminous binders - Characterization of perceptible properties

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

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EN 12595, Bitumen and bituminous binders Determination of kinematic Viscosity

3 Principle

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

The effects of heat and air are determined based on the change in mass (expressed as a percentage) and on 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 $163\,^{\circ}$ 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 a minimum interior height, width and depth of 330 mm.

The front door shall be hinged and tightly fitted, and have a clear opening of the same height and width as the interior of the oven. The window on the door shall be at least 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 upon opening the outer door momentarily.

The oven shall be ventilated by convection currents of air; the oven shall have air inlets and outlets evacuating hot gases meeting the requirements of Table 1.

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

Characteristic	Requirement
Deviation from a specified test temperature throughout the testing chamber during a 24 h 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 h, minimum	10

Table 1 — Performance requirements for Thin Film Oven

NOTE 3 These performance requirements are extracted from ASTM E 145, type IB (gravity convection ventilated oven).

The oven shall be provided with a metal circular shelf with a minimum diameter of 250 mm. The shelf shall provide a flat surface to support 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 $(5,5\pm1,0)$ r/min. 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 allow the use of 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. 539bccc7b4a4/sist-en-12607-2-2007

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 to provide the same readings as would be provided by the mercury stem thermometer, recognising and allowing for 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 to an extent greater cyclic variations than mercury thermometers 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 ± 1) mm in inside diameter and (9.5 ± 0.5) mm deep with a flat bottom, stainless steel or aluminium, with a thickness of (0.6 ± 0.1) mm to (1.0 ± 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 significantly affect results, it is advisable to eliminate damaged pans.

4.4 Balance, accurate to ± 10 mg.

5 Sampling

5.1 General

Make sure that the laboratory sample is homogeneous and is not contaminated (see EN 1425). 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). The laboratory sample shall be taken in accordance with EN 58.

5.2 Test sample preparation

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

The sample shall be free of water. Heat the sample in an oven in its container with a loosely fitted cover 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. If special bituminous binders, modified binders or bituminous binders with a high softening point are tested, it may be necessary to prepare the sample at a higher temperature. In this case, heat the sample as described above and in accordance with EN 12594. For polymer modified bitumens, the temperature may not exceed 200 °C, irrespective of the softening point.

5.3 Initial characteristics

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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 a Cl. (EN:12595) (V:1b687-47bb-bc38-

539bcec7b4a4/sist-en-12607-2-2007

6 Procedure

6.1 Test conditions

Ensure that the oven (4.1) is level so that the shelf rotates on 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 centre 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 ± 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 ± 1) °C for harder bitumen grades.

6.2 Determination

Weigh separately metal containers (4.3) to the nearest 1 mg (M_0).

Pour (50.0 ± 0.5) g of the sample into each of two or more of the containers. Allow the samples to cool to room temperature for approximately 30 min, and weigh them separately to the nearest 1 mg (M_1) .

With the oven at the test temperature \pm 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 for approximately 30 min, 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 cannot 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 (e.g. the kinematic viscosity of the residue at 60 °C (EN 12595), v_2) within 72 h. Avoid reheating the sample more than once.

NOTE Excessively reheating the sample can influence the test results obtained.

7 Calculation

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

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$$\Delta v$$
, ratio of kinematic viscosities at 60 °C $\frac{v_1}{v_2}$ teh.ai) (1)

$$- \Delta M, \text{ percentage of change in mass} = 100 \times 100$$

where

- v₁ is the kinematic viscosity of the sample before hardening, in millimetres squared per second;
- v_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 a loss in mass as a negative percentage change and a gain in mass as a positive percentage change.

The results of the percentage change in mass for two containers are considered valid if the difference of the two measurements is equal to or less than 0,05 % mass fraction absolute.

Express the percentage change in mass as the average of a minimum 2 valid determinations to the nearest 0.01 %.

NOTE If the value of a physical property is measured on the sample after TFOT, e.g. kinematic viscosity, express the ratio of viscosities to the nearest 0,1. Report the values of initial properties and residue properties.