

SLOVENSKI STANDARD

SIST EN 12607-2:2014

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Nadomešča:

SIST EN 12607-2:2007

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 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 et de l'air - Partie 2: Méthode TFOT

Ta slovenski standard je istoveten z: **EN 12607-2:2014**

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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EUROPEAN STANDARD

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Bitumen and bituminous binders - Determination of the resistance to hardening under 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 16 August 2014.

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-CENELEC 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-CENELEC 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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 12607-2:2014) 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 May 2015 and conflicting national standards shall be withdrawn at the latest by May 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 12607-2:2007.

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

In comparison with EN 12607-2:2007, the following significant changes have been made:

- changed/added wording of the Warning in the Scope;
- EN 12595, *Bitumen and bituminous binders — Determination of kinematic viscosity* has been added to Clause 2;
- the reference to mercury thermometer has been deleted (see subclause 4.2) and Annex A is informative.

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*.

The scope of this standard has been enlarged by adding oxidized bitumens and hard industrial bitumens that are not used for paving applications. The reason is to facilitate that EN 13303, *Bitumen and bituminous binders – Determination of the loss of mass after heating of industrial bitumen* can be replaced in product standards by EN 12607-2 at the next revision of EN 13303.

It is to be noted that this – if so decided – will change the property from loss in mass into change in mass in EN 13304, *Bitumen and bituminous binders – Framework for specification of oxidized bitumens* and in EN 13305, *Bitumen and bituminous binders – Framework for specification of hard industrial bitumens*.

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, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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1 Scope

This part of EN 12607 specifies a method for measuring the combined effects of heat and air on a film of bitumen or bituminous binder, simulating the hardening which most bituminous binders undergo during mixing in an asphalt mixing plant. The method is suitable for other bituminous binders than paving grade bitumen, but the reference temperature might give excessive hardening that does not resemble real conditions during mixing at the plant. The method may not represent the hardening that occurs during mixing of warm mix binders.

Additionally, this part of EN 12607 specifies a method for the determination of the change in mass of oxidized bitumens and hard industrial bitumens after heating. The method is used to detect volatile components.

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

WARNING — Use of this European Standard can involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to identify the hazards and assess the risks involved in performing this test method and to implement sufficient control measures to protect individual operators (and the environment). This includes appropriate safety and health practices and determination of 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 cut-back bitumen or bituminous emulsions before these products have been stabilized, e.g. in accordance with EN 13074-2.

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2 Normative references

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The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

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.

For bituminous binders for paving applications, 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 according to EN 12595, before and after the period in the oven.

For bituminous binders for industrial applications, the combined effects of heat and air and loss of volatiles are determined based on the change in mass (expressed as a percentage). This will be the value from this conditioning method that will be utilized in EN 13304 and EN 13305 as loss in mass (change in sign).

NOTE Penetration according to EN 1426 and softening point according to 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 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.

Table 1 — Performance requirements for Thin Film Oven

Characteristics	Requirements
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

NOTE 3 These performance requirements are extracted from ASTM E145, 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 $(5,5 \pm 1,0)$ 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.

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.

4.2 Temperature measuring device.

A temperature measuring device (combining sensor and reading unit) shall

- have a range from at least 110 °C to 170 °C,
- be readable to 0,1 °C or less and,
- have an accuracy of 0,5 °C or better.

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Sensors based on platinum resistance thermometers have been found suitable but other principles are also allowed. The thermal response time of the sensor shall be comparable with the former used reference (see informative Annex A). The temperature measuring device shall be calibrated regularly.

A solid stem mercury thermometer (which used to be the former reference thermometer as described in Annex A) is also allowed if national regulations permit its use.

When measuring and controlling nominally constant temperatures as in this test method, the thermal response time can be rather high (e.g. slow response to a change in temperature). Care shall be taken to consider this aspect since low thermal response times of the sensor can indicate greater cyclic variations than the bituminous material in practise.

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

5 Sampling

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5.1 General

Make sure that the laboratory sample is homogeneous and is not contaminated according to 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

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), v_1 .

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.

For bituminous binders for paving applications, the reference temperature of the test is (120 ± 1) °C, corresponding to the mixing temperature of soft paving grade bitumens.

NOTE However, it is possible to perform the test at other temperatures, e.g. (163 ± 1) °C for harder paving bitumen grades.

For bituminous binders for industrial applications, the reference temperature of the test is (163 ± 1) °C.

6.2 Determination

Weigh separately containers (4.3) to the nearest 1 mg (m_0).

Pour $(50,0 \pm 0,5)$ 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 ± 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.

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: