SLOVENSKI STANDARD
SIST EN 12607-3:2014

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Nadomešča:
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Bitumen in bitumenska veziva - Določanje odpornosti proti otrjevanju pod vplivom toplote in zraka - 3. del: Metoda RFT

Bitumen and bituminous binders - Determination of the resistance to hardening under influence of heat and air - Part 3: RFT method

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Beständigkeit gegen Verhärtung unter Einfluss von Wärme und Luft - Teil 3: RFT-Verfahren

Bitumes et liants bitumineux - Détermination de la résistance au durcissement sous l'effet de la chaleur et de l'air - Partie 3: Méthode RFT

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ICS:

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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 influence of heat and air - Part 3: RFT method

Bitumes et liants bitumineux - Détermination de la résistance au durcissement sous l'effet de la chaleur et de l'air - Partie 3: Méthode RFT

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Beständigkeit gegen Verhärtung unter Einfluss von Wärme und Luft - Teil 3: RFT-Verfahren

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Foreword

This document (EN 12607-3:2014) 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of an identical text or by endorsement, at the latest by May 2015 and conflicting national standards shall be withdrawn at the latest by May 2015.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 12607-3:2007.

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 12607-3:2007, the following significant changes have been made:

— changed/added wording of the Warning in the Scope;

— EN 13302, *Bitumen and bituminous binders - Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus* has been added to Clause 2;

— the reference to mercury thermometer has been deleted (see subclause 4.4) and Annex A is informative;

— subclause 5.2: reference to RFT instead of RTFOT.

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, 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.

1 Scope

This part of EN 12607 specifies a method for measuring the combined effects of heat and air on a thin moving 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.

The method is referred to as RFT, i.e. Rotating Flask 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.

2 Normative references

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 1426, *Bitumen and bituminous binders - Determination of needle penetration*

EN 1427, *Bitumen and bituminous binders - Determination of the softening point - Ring and Ball method*

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

EN 12596, *Bitumen and bituminous binders - Determination of dynamic viscosity by vacuum capillary*

EN 13302, *Bitumen and bituminous binders - Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus*

3 Principle

A moving film of bituminous binder is heated in a rotating flask of a rotary evaporator at a specified temperature for a given period of time.

The effect of rotation is that material forming on the surface of the sample in the flask is constantly replaced, preventing the formation of a skin.

The effects of heat and air are determined based on the change in mass (expressed as a percentage) or as a change in the bituminous binders' characteristics such as penetration according to EN 1426, softening point

ring and ball according to EN 1427 or dynamic viscosity according to EN 12596 or EN 13302, before and after hardening.

4 Apparatus

Usual laboratory apparatus and glassware, together with the following:

4.1 Rotary evaporator, capable of maintaining a rotational speed of (20 ± 5) r/min, used in conjunction with a 1 000 ml round bottom flask with a 29/32 ground cone socket.

NOTE Cooler and receiver are not required.

4.2 Flow control device, capable of maintaining an air flow rate of (500 ± 10) ml/min at ambient temperature.

NOTE To eliminate the effects of oxidation reactions, the air can be replaced with inert gases such as nitrogen.

4.3 Flowmeter, capable of measuring the airflow at a rate of 500 ml/min with a maximum indication error of ± 5 ml/min.

4.4 Temperature measuring device

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

— have a range from at least 30 °C to 200 °C,

— be readable to 0,1 °C or less, and

— have an accuracy of 0,5 °C or better.

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 experiences.

4.5 Glass air inlet pipe, approximately 400 mm long and with an inside diameter of 7 mm, mounted along the axis of rotation of the flask, as illustrated in Figure 1.

4.6 Compressor, or compressed air cylinder, fitted with a reducing valve.

4.7 Thermostatically controlled oil bath, regulated to (165 ± 1) °C.

4.8 Oven, capable of achieving temperatures up to no less than 120 °C.

4.9 Balance, accurate to ± 10 mg.

5 Sampling

5.1 General

Ensure 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 RFT 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 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 bitumen, the temperature may not exceed 200 °C, irrespective of the softening point.

5.3 Measurement

The test sample shall weigh (100 ± 1) g.

If this quantity of sample is not sufficient for the determination of the properties that are to be subsequently measured (see Table 1), further samples shall be separately subjected to the same test procedure.

5.4 Measurement of initial characteristics

Determine the initial characteristics of the bituminous binder, e.g.:

— P_1, penetration at 25 °C according to EN 1426;

— T_1, softening point ring and ball according to EN 1427;

— η_1, dynamic viscosity at 60 °C according to EN 12596 or EN 13302.

6 Procedure

6.1 Test with the determination of change in mass

Weigh the clean and dry flask of the rotary evaporator (4.1) to the nearest 5 mg and record the mass m_0. This value will be needed to determine the mass of the sample before and after hardening.

Weigh (100 ± 1) g of the test sample into the flask of the rotary evaporator (4.1). Allow to cool in a desiccator to a temperature between 18 °C and 28 °C, and weigh the flask to the nearest 5 mg and determine the mass m_E of the sample.

Heat the oil bath (4.7) to the test temperature ± 1 °C and mount the flask containing the sample in the bath with the axis of rotation of the flask lying at an angle of 45° to the perpendicular and the spherical body of the flask being completely immersed in the bath liquid (see Figure 1). Insert the air-inlet pipe (4.5) along the axis of rotation of the flask with a clearance of (40 ± 2) mm between the lower end of the pipe and the bottom of

the flask. As there is a danger that the flask may detach from the rotary evaporator during the test, ensure that the flask is secured, either by an integral clamp on the rotary evaporator or by a separate joint clip of suitable material (e.g. PTFE).

NOTE The reference temperature of the test is 165 °C, however, it is possible to perform the test at other temperatures.

Heat the sample without supplying additional air while the flask rotates at (20 ± 5) r/min. After (10 ± 1) min, switch on the air supply at a flow rate of (500 ± 10) ml/min.

Ensure that the air supply is between 18 °C and 28 °C when entering the air-inlet pipe and that throughout the test, the temperature of the bath liquid is maintained at the test temperature ± 1 °C.

After (150 ± 1) min, measured from the time when the air was first admitted, switch off the rotation mechanism and air supply and remove the flask immediately from the bath liquid. When the flask has cooled slightly, wipe off the oil adhering to its outer surface with a cloth saturated with a suitable volatile solvent.

Immediately place the flask in the oven (4.8), set at (110 ± 5) °C and keep it there for (30 ± 1) min to permit the sample dispersed over the inner wall during rotation, to collect at the bottom of the flask and to allow remaining solvent to be released.

Cool the flask in the desiccator for (90 ± 5) min to an ambient temperature between 18 °C and 28 °C and weigh the content of the flask to the nearest 5 mg. Calculate the mass, m_A of the sample after hardening.

Carefully bring the flask to 80 °C to 90 °C above the expected ring and ball softening point (EN 1427), and pour the contents immediately from the flask into the various vessels and moulds required for the subsequent tests. For polymer modified bitumen, the temperature may not exceed 200 °C, irrespective of the softening point.

If two or more samples of a bitumen are subjected to this procedure, pour each sample firstly into a collecting vessel maintained at an ambient temperature of between 18 °C and 28 °C.

When the last sample has been poured into this collecting vessel, gently heat the contents to the pouring temperature, i.e. 80 °C to 90 °C above the expected ring and ball softening point (see EN 1427) and stir thoroughly. For polymer modified bitumen, the temperature may not exceed 200 °C, irrespective to the softening point. When thoroughly mixed, pour the contents immediately into the vessels and moulds required for subsequent tests.

Measure the characteristics of the hardened binder as described in 6.3.

6.2 Test without the determination of change in mass

Carry out the procedure described in 6.1, but without determining the mass of the sample.

After the sample has been submitted to the hardening test, bring the contents of the flask to the pouring temperature (see 6.1), and fill the vessels and moulds necessary for subsequent tests.

If two or more samples are required, carry out the procedure as described in 6.1.

Measure the characteristics of the hardened binder as described in 6.3.

6.3 Measurement of binder characteristics after hardening of the binder

Measure the characteristics of the hardened binder within 72 h in accordance with the various methods of tests. Avoid reheating the samples more than once. P_2 is the penetration at 25 °C, T_2 is the softening point ring and ball and η_2 is the dynamic viscosity at 60 °C, of the hardened binder.