SLOVENSKI STANDARD
SIST EN 12607-3:2000

01-julij-2000

Bitumen in bitumenska veziva - Določ evanje odpornosti proti strjevanju pod vplivom toplote in zraka - 3. del: Metoda RFT

Bitumen and bituminous binders - Determination of the resistance to hardening under the 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

Ta slovenski standard je istoveten z: **EN 12607-3:1999**

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

SIST EN 12607-3:2000 **en**

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 12607-3

October 1999

ICS 75.140

English version

Bitumen and bituminous binders - Determination of the resistance to hardening under the 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

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

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 12607-3:1999 E

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 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 specifies a method for measuring the combined effects of heat and air on a moving thin 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 RFT i.e. Rotating Flask 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 - Determination of perceptible properties.*

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

EN 1427, *Bitumen and bituminous binders - Determination of 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.*

3 Principle

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

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

The effects of heat and air are determined on the basis of the change in mass (expressed as a percentage) or as a change in the bituminous binders' characteristics such as penetration (EN 1426), softening point ring and ball (EN 1427) or dynamic viscosity (EN 12596) before and after hardening.

4 Apparatus

Usual laboratory apparatus and glassware, together with the following:

4.1 Rotary evaporator, capable of maintaining 20 min^{-1} ± 5 min^{-1}, 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 ml/min ± 10 ml/min at ambient temperature.

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

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 Thermometer, 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.

4.5 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 °C ± 1 °C.

4.8 Oven, capable of achieving temperatures up to not less than 120 °C.

4.9 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 for 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

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 RFT hardening test, if necessary using a warmed knife, and transfer it to a suitable container according to EN 12594.

5.3 Measurement

The test sample shall weigh 100 g ± 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 sample 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, the penetration at 25 °C (EN 1426),
- T_1, the softening point ring and ball (EN 1427),
- η_1, the dynamic viscosity at 60 °C (EN 12596).

6 Procedure

6.1 Test with the determination of change in mass

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

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 mm ± 2 mm between the lower end of the pipe and the bottom of the flask.

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 min^{-1} ± 5 min^{-1}.
After 10 min ± 1 min, switch on the air supply at a flow rate of 500 ml/min ± 10 ml/min.

Ensure that the air supply is at a temperature of 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 min ± 1 min, measured from the time when 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 a temperature of 110 °C ± 5°C and keep it there for 30 min ± 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 min ± 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.

Bring the flask carefully to a temperature of 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 subsequent tests.
If two or more samples of a bitumen are subjected to thermal stressing, 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 (80 °C to 90 °C above the expected ring and ball softening point) and thoroughly mix with the thermometer (4.4). 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 the permitted delay (see 6.1) in accordance with the various methods of tests. Avoid reheating of the samples. 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.

7 Calculation

Calculate the percentage of change in mass of the sample for procedure 6.1 as follows:

$$\Delta m, \text{change in mass} = 100 \times \frac{m_A - m_E}{m_E}$$

where

m_E is the mass of the sample, in grams, before the hardening;

m_A is the mass of the sample, in grams, after the hardening.

Calculate the changes in the physical characteristics after the hardening procedure as follows:

- percentage of retained penetration at 25 °C = $100 \times \frac{P_2}{P_1}$

- increase in softening point ring and ball = T_2 - T_1 (in °C)

- ratio of dynamic viscosities at 60 °C = $\frac{\eta_2}{\eta_1}$

8 Expression of results

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

Two results of the percentage of change in mass are considered valid if they do not differ by more than 0,05 % (*m/m*) absolute.
Express the percentage of change in mass value as the average of the two valid determinations, to the nearest 0,01 % (*m/m*) absolute.

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 1 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 1 in only one case in twenty.

Table 1 - Precision

Characteristic	Repeatability *r*	Reproducibility *R*
% change in mass (% (*m/m*) absolute) (> 0,3 % and < 0,80 %)	0,15	0,20
% retained penetration at 25 °C (% (*m/m*) absolute)	7	10
Increase in softening point ring and ball (°C) - values < 6,5 °C - values ≥ 6,5 °C	 1,5 3,0	 2,0 4,0
Ratio of dynamic viscosity at 60 °C (% of mean)	10	20

NOTE These precision data 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: