SLOVENSKI STANDARD
SIST EN 13357:2003

01-december-2003

Bitumen in bitumenska veziva ï Doloļ anje viskoznosti s petrolejem rezanega in fluksiranega bitumna, metoda z iztoļ nim viskozimetrom

Bitumen and bituminous binders - Determination of the efflux time of petroleum cut-back and fluxed bitumens

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Ausflusszeit von Mineralölverschnittbitumen und -fluxbitumen

Bitumes et liants bitumineux - Détermination du temps d'écoulement des bitumes fluidifiés et fluxés

Ta slovenski standard je istoveten z: **EN 13357:2002**

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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English version

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This European Standard was approved by CEN on 23 October 2002.

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Foreword

This document (EN 13357:2002) 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of an identical text or by endorsement, at the latest by June 2003, and conflicting national standards shall be withdrawn at the latest by June 2003.

This European Standard is based on IP 72-86.

Annex A is normative.

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

1 Scope

This European Standard specifies a method for the determination of the efflux time of petroleum cut-back and fluxed bitumens in seconds using an efflux viscometer.

WARNING – The use of this European Standard may 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 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 (including amendments).

EN 58[1)] , *Sampling bituminous binders*.

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

ISO 9722, *Nickel and nickel alloys – Composition and forms of wrought products.*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
viscosity
internal resistance of a fluid to flow

3.2
efflux time
time needed for a specified volume of a material to flow through a specified orifice at a specified temperature

NOTE The efflux time is an indication of the pseudoviscosity, which is defined as the internal resistance to flow of fluids.

4 Principle

The viscosity of petroleum cut-back and fluxed bitumens is determined using an efflux viscometer known as the Standard Tar Viscometer (STV) which determines the time of efflux of 50 ml of the sample through a 4 mm or a 10 mm orifice.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Viscometer, (see the example of viscometer in Figure 1) consisting essentially of a cup with an orifice in the centre of the base which may be closed by a ball-and-socket valve (see Figure 2). Two forms are required, differing only in the size of the orifice (4 mm and 10 mm). For other dimensions, see Figure 3 (10 mm orifice) and Figure 4 (4 mm orifice). The cup is a cylindrical brass tube with a dished phosphor-bronze bottom. The ball valve consists of

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1) In course of revision.

a phosphor-bronze ball on a rod of wrought nickel alloy with copper or rod of metals NiCu30 in accordance with ISO 9722, with an oil-level peg attached to the rod and a hemispherical top by means of which the valve may be supported in a vertical position.

Choose the diameter of the orifice of the cup as follows:

— A viscometer cup with a 10 mm orifice is used at 25 °C for materials the efflux time of which at that temperature and from that cup exceeds 15 s and at 40 °C for materials the efflux time of which at 25 °C exceeds 500 s.

— A viscometer cup with a 4 mm orifice is used at a temperature of 25 °C for materials the efflux time of which is less than 15 s from the 10 mm orifice container at 25 °C.

— If the pseudoviscosity is unknown, measure it at 25 °C with the 10 mm orifice viscometer cup. According to the efflux time obtained, 3 cases are possible:

25 °C 10 mm

If t ≤ 15 s	If 15 s < t < 500 s	If t ≥ 500 s
T = 25 °C Ø = 4 mm	T = 25 °C Ø = 10 mm	T = 40 °C Ø = 10 mm

NOTE The viscometer cup should be provided with:

— For the 10 mm orifice viscometer cup, a cork suitable for closing the 10 mm orifice with the ball valve in position;

— A cork suitable for closing the upper end of the cup without touching the test material when the cup is filled. This cork should be provided with a central hole through which the thermometer can pass.

5.2 Water-bath, constant temperature for maintaining the test temperature to within ± 0,5 °C. A typical elevation and a plan of assembled viscometer is given in Figure 1.

5.3 Thermometer, conforming to the requirements described 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.

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.

5.4 Receiver, consisting of a 100 ml cylinder with graduations at 20 ml, 25 ml and 75 ml.

5.5 Stopwatch, capable of measuring the efflux time with an accuracy of ± 0,2 s.

6 Procedure

6.1 Preparation of sample

The material under test shall be sampled in accordance with EN 58 and prepared in accordance with EN 12594.

NOTE A viscometer cup with hot bitumen should not be plunged into cool water as this shock cooling will radically effect the viscosity result.

6.2 Preparation of apparatus

Clean the viscometer cup (5.1) with a suitable solvent and thoroughly dry it to remove all traces of solvent. If necessary, rub the interior of the cup and/or clean the orifice. Use soft tissue-paper or some similar material that will not leave particles behind or abrade the metal.

When cleaning, care shall be taken not to damage the orifice.

6.3 Procedure

6.3.1 Close the bottom orifice with the ball valve. Fill the cup with the prepared sample to such a height that the levelling peg on the valve is just immersed when the latter is vertical. Insert a large plug, to close the top of the cup. It shall be provided with a central hole and a groove on one side through which the stem of the valve may be passed into the upper end of the cup, and pass the thermometer (5.3) through the central hole so that its bulb is approximately at the geometric centre of the sample. Suspend the cup up to its rim in a water bath (5.2) maintained within ± 0,5 °C of the test temperature for a period of 90 min ± 10 min.

6.3.2 Set up the viscometer (5.1) in a level position, fill the jacket with water and adjust its temperature to within ± 0,5 °C of the test temperature. Throughout the test, maintain the water jacket temperature at this value, stirring frequently.

6.3.3 Check that the sample has reached the required temperature, and remove the thermometer and plugs. Remove any excess sample so that its final level is on the centre line of the levelling peg when the valve is in a vertical position. Pour light mineral oil into the receiver up to the 20 ml graduation mark and place the receiver (5.4) directly under the orifice of the cup. Lift the valve and suspend it on the valve support such that the peg is levelled with the upper edge of the cup of at least 16 mm. Start the time-recording device (5.5) when the liquid in the receiver reaches the 25 ml graduation mark and stop it when the liquid reaches the 75 ml graduation mark.

7 Expression of results

Report the time of efflux in seconds, the size of the orifice (4 mm or 10 mm) and the temperature of the test (25 °C or 40 °C).

8 Precision

8.1 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 value given in Table 1 only in one case in twenty.

8.2 Reproducibility

The difference between two single and independent test 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 value given in Table 1 only in one case in twenty.

Table 1 — Precision

Efflux time s	Repeatability	Reproducibility
< 20	2 s	2 s
20 to 40	2 s	10 % of the mean
> 40	5 % of the mean	10 % of the mean

9 Test report

The test report shall contain at least the following information:

a) the type and complete identification of the sample under test;

b) reference to this test method;

c) the result of the test (see Clause 7);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.