SLOVENSKI STANDARD
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Bitumen in bitumenska veziva - Določ evanje viskoznosti bitumenskih emulzij z iztoč nim viskozimetrom - 2. del: Rezana in fluksirana bitumenska veziva

Bitumen and bituminous binders - Determination of the efflux time by the efflux viscometer - Part 2: Cut-back and fluxed bituminous binders

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Ausflusszeit mittels Ausflussviskosimeter - Teil 2: Verschnittene und gefluxte bitumenhaltige Bindemittel

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91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

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Bitumen and bituminous binders - Determination of the efflux time by the efflux viscometer - Part 2: Cut-back and fluxed bituminous binders

Bitumes et liants bitumineux - Détermination du temps d'écoulement à l'aide d'un viscosimètre à écoulement - Partie 2: Bitumes fluidifés et fluxés

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Ausflusszeit mittels Ausflussviskosimeter - Teil 2: Verschnittene und gefluxte bitumenhaltige Bindemittel

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 336.

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (prEN 12846-2:2008) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 13357:2002.

This European Standard EN 12846 consists of the following parts under the general title *Bitumen and bituminous binders – Determination of efflux time by efflux viscometer:*

Part 1 – Bituminous emulsions;

Part 2 – Cut-back and fluxed bituminous binders.

1 Scope

This European Standard specifies a method for the determination of the efflux time at 25 °C of petroleum cut-back and fluxed bituminous binders in seconds using an efflux viscometer. Alternative test temperatures are 40 °C, 50 °C and 60 °C.

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

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

EN 14896, *Bitumen and bituminous binders – Dynamic viscosity for bituminous emulsions, cut-back and fluxed bituminous binders – Rotating spindle viscometer method*

ISO 4788, *Laboratory glassware - Graduated measuring cylinders*

ISO 9722, *Nickel and nickel alloys- Composition and form 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.

4 Principle

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

Whatever temperatures or orifice diameters used, the efflux time shall not exceed 600 s. For highly viscous cut-back or fluxed bituminous binders, EN 14896 shall be used.

5 Reagents and materials

5.1 Light mineral oil

Light mineral oil having a viscosity equal or lower than 7cSt (7 mm^2/s) à 40 °C shall be used.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Efflux viscometer, (see an example of viscometer on 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 (10 mm and 4 mm). For other dimensions of the cup and the ball-valve, see Figure 3. The cup is a cylindrical brass tube with a dished phosphor-bronze bottom. The ball valve consists of 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.

The viscometer cups shall be provided with suitable corks for closing the orifices with the ball valve in position, and some means of covering the cups (e.g. lids) to prevent cooling.

The viscometer-cup holder shall be capable of:

— supporting one or more cups in a vertical position;

— providing a valve support to hold the valve (16 ± 1) mm vertically above the orifice of the cup during efflux of the test material.

NOTE The viscometer cup should be provided with a lid suitable for closing the upper end of the cup without touching the test material when the cup is filled. This lid is provided with a central hole through which the thermometer can pass and with a grove on one side through which the stem of the valve can pass.

6.2 Constant temperature water bath. A water bath, maintained at (25,0 ± 0,5) °C, in which one or more of the filled viscometer cups can be immersed up to the rim of the cup.

NOTE Alternative test temperatures are (40,0 ± 0,5) °C, (50,0 ± 0,5) °C and (60,0 ± 0,5) °C (see sub-clause 6.1, paragraph 3) and followings).

If a multiple-cup heating bath is used, the cups shall be separated from each other and from the walls of the bath by at least 55 mm.

A suitable support shall be provided to maintain the cup(s) in a vertical position.

6.3 Viscometer 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.

6.4 Thermometers, two, 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.

6.5 Receiver, consisting of a 100 ml cylinder with graduations at 20 ml, 25 ml and 75 ml, complying with the requirements of ISO 4788.

6.6 Timing device, capable of measuring the efflux time with an accuracy of ± 0,2 s.

7 Sampling

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.

The test shall be carried out in duplicate.

8 Procedure

8.1 General

Carry out the procedure under normal laboratory conditions.

NOTE "Normal laboratory conditions" means that the range of temperature is 18 °C to 28 °C.

8.2 Preparation of apparatus

Clean the viscometer cup (6.1) with a suitable solvent, enabling to remove any mark of binder, 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.

8.3 Measurement

8.3.1 General

If the pseudoviscosity is unknown, measure it at 25 °C with the 10 mm orifice viscometer cup. According to the efflux time obtained, 5 main cases are possible. Choose the diameter of the orifice of the cup and the temperature of the test as follows.

1) If the efflux time is lower than 15 s, perform another determination at 25 °C with 4 mm orifice.

2) If the efflux time is between 15 s and 600 s, no change in the experimental conditions (25 °C with 10 mm orifice) is necessary.

3) If the efflux time reaches 600 s, perform another determination at 40 °C with the 10 mm orifice.

4) If the efflux time determined at 40 °C is greater than 600 s, perform another determination at 50 °C with the 10 mm orifice.

5) If the efflux time determined at 50 °C is greater than 600 s, perform another determination at 60 °C with the 10 mm orifice.

Table 1 — Temperature up to 40 °C and diameter of the cup

25 °C 10 mm

If t < 15 s	If 15 s < t < 600 s	If t > 600 s
T = 25 °C Ø = 4 mm	T = 25 °C Ø = 10 mm	T = 40 °C (see 4) and 5) if necessary) Ø = 10 mm

For highly viscous cut-back or fluxed bituminous binders having efflux time at 60 °C with the 10 mm orifice higher than 600 s, test shall be performed by means of dynamic viscosity measurement (EN 14896).

8.3.2 Close the lower part of the orifice with a cork or a cap and place the ball valve on top of the orifice. Carefully 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. Cover the top of the cup for example with a suitable lid. It shall be provided with a central hole and a groove on one side through which the stem of the valve (Figure 3, Key 7) may be passed into the upper end of the cup, and pass the thermometer (6.4) through the central hole so that its bulb is approximately at the geometric centre of the sample.

8.3.3 Suspend the cup up to its rim in the water bath (6.2) maintained within ± 0,5 °C of the test temperature for a period of (90 ± 10) min.

8.3.4 Condition the viscometer water-bath (6.3), by stirring the water in the bath with the relevant device and check that the temperature is at the required value for the test, maintained within ± 0,5 °C.

8.3.5 Remove the fill cup from the water bath and remove the cork or stopper.

8.3.6 Place the filled cup into the viscometer cup holder.

8.3.7 Check that the sample has reached the required temperature. Remove any excess sample while removing the thermometer so that the final level of the binder is on the centre line of the levelling peg when the valve is in a vertical position.

8.3.8 Pour light mineral oil into the receiver up to the 20 ml graduation mark and place the receiver (6.5) 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 (6.6) when the liquid in the receiver reaches the 25 ml graduation mark and stop it when the liquid reaches the 75 ml graduation mark.

Record the time of efflux to the nearest 0,2 s.

8.3.9 Repeat Sampling (Clause 7) and Procedure (Clause 8) steps on a second test sample.

9 Expression of results

Express results as the arithmetic mean of the two results obtained in accordance with Clause 8, to the nearest second, provided that individual results do not differ by more than 10 % of the arithmetic mean.

If the two results differ by more than 10 %, repeat the whole procedure.

Report the diameter of the orifice (4 mm or 10 mm) used and the test temperature(s) (25 °C, and possibly 40 °C, 50 °C and/or 60 °C).

10 Precision

10.1 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 2 only in one case in twenty.

10.2 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 2 only in one case in twenty.

Table 2 — Precision

Efflux time s	Repeatability	Reproducibility
below 20	1 s	2 s
20 to 40	2 s	10 % of the mean
above 40	5 % of the mean	10 % of the mean

11 Test report

The test report shall contain at least the following information:

a) type and complete identification of the sample under test (including date of sampling and date of sample preparation);

b) reference to this European Standard;

c) test temperature;

d) diameter of the orifice;

e) result of the test in seconds (see Clause 9);

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

g) date of the test.