



SLOVENSKI STANDARD

SIST EN 12846-2:2011

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Nadomešča:
SIST EN 13357:2003

Bitumen in bitumenska veziva - Določevanje viskoznosti bitumenskih emulzij z iztočnim 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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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

Bitumen and bituminous binders - Determination of 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 fluidifiés et fluxés

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

This European Standard was approved by CEN on 22 January 2011.

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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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Contents

Page

Foreword.....	3
1 Scope	4
2 Normative references	4
3 Terms and definitions	4
4 Principle.....	4
5 Reagents and materials	5
6 Apparatus	5
7 Sampling.....	6
8 Procedure	6
8.1 General.....	6
8.2 Preparation of apparatus	6
8.3 Measurement.....	6
9 Expression of results	8
10 Precision.....	8
10.1 Repeatability.....	8
10.2 Reproducibility.....	8
11 Test report	8
Annex A (normative) Specifications of thermometer.....	11
Bibliography.....	12

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(standards.iteh.ai)

SIST EN 12846-2:2011

<https://standards.iteh.ai/catalog/standards/sist/9cbb246-d13d-41bd-9a4d-b3698c5a4491/sist-en-12846-2-2011>

Foreword

This document (EN 12846-2:2011) 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 September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

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 13357:2002.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive 89/106/EEC.

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

— Part 1: Bituminous emulsions,

— Part 2: Cut-back and fluxed bituminous binders.

EN 12846-2 has been created as the result of the merging of EN 12846:2002 and EN 13357:2002 under a single EN 12846 reference (two different parts), since both standards describe very similar procedures with identical equipment. The two different parts have been made as consistent as possible by eliminating all existing minor differences between both methods.

Compared with EN 13357:2002, test temperatures were detailed in the scope, a restriction was added in the principle, tolerances were specified or modified in the apparatus dimensions, details were added in the test procedure and measurements made and the precision data were reviewed.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

EN 12846-2:2011 (E)**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 13302, *Bitumen and bituminous binders — Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus*

EN ISO 4788, *Laboratory glassware — Graduated measuring cylinders (ISO 4788:2005)*

3 Terms and definitions

SIST EN 12846-2:2011

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For the purposes of this document, 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 indirect measure for viscosity and is referred to as “pseudo-viscosity”.

4 Principle

The efflux time 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 13302 shall be used.

5 Reagents and materials

5.1 Light mineral oil.

Light mineral oil having a viscosity equal or lower than $7 \text{ mm}^2/\text{s}$ at $40 \text{ }^\circ\text{C}$ 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 cylinder shall be made of brass. The ball valve should be made of corrosion-resistant metal, with a ball on a rod, a levelling 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 or caps for closing the orifices with the ball valve in position, and some means of covering the cups (e.g. lids) to minimize surface cooling effects.

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 at least 16 mm vertically above the orifice of the cup during efflux of the test material.

SIST EN 12846-2:2011

NOTE 1 To enhance resistance to wear and corrosion of the ball and socket valve, the bottom of the cup may be made from a different, corrosion resistant, material and screwed to the brass tube. It is then advised to use the same material, such as for instance phosphor-bronze, for the cup bottom and the ball valve. Wrought nickel alloy with copper or metals NiCu30 in accordance with ISO 9722 are possible materials for the rod of the ball valve.

NOTE 2 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 groove on one side through which the rod of the valve can pass.

6.2 Viscometer water-bath, constant temperature for maintaining the test temperature to within $\pm 0,5 \text{ }^\circ\text{C}$. A typical elevation and a plan of assembled viscometer is given in Figure 1.

6.3 Temperature controlled water bath. A water bath, maintained at $(25,0 \pm 0,5) \text{ }^\circ\text{C}$, in which one or more of the filled viscometer cups can be immersed up to the rim of the cup.

NOTE 1 Alternative test temperatures are $(40,0 \pm 0,5) \text{ }^\circ\text{C}$, $(50,0 \pm 0,5) \text{ }^\circ\text{C}$ and $(60,0 \pm 0,5) \text{ }^\circ\text{C}$ (see 8.3.1, 3), 4) and 5)).

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.

NOTE 2 The viscometer water bath (6.2) may also be used to directly condition the test sample in the cup.

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

EN 12846-2:2011 (E)

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 EN ISO 4788.

6.6 Timing device, capable of measuring the efflux time with an accuracy of $\pm 0,2$ 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

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8.1 General

Carry out the procedure in laboratory at room temperature between 18 °C to 28 °C
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8.2 Preparation of apparatus

Clean the viscometer cup (6.1) with a suitable solvent, 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 If the efflux time 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 5 s, perform another determination at 25 °C with 4 mm orifice.
- 2) If the efflux time is between 5 s and 600 s with still a continuous flow, no change in the experimental conditions (25 °C with 10 mm orifice) is necessary.
- 3) In case of a non continuous flow or if the efflux time is greater than 600 s, perform another determination at 40 °C with the 10 mm orifice.
- 4) In case of a non continuous flow or 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) In case of a non continuous flow or 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

<div style="border: 1px solid black; padding: 5px; width: fit-content; margin: 0 auto;"> 25 °C 10 mm </div>		
↙	↓	↘
If $t < 5$ s	If $5 \text{ s} \leq t \leq 600$ s and continuous flow	If $t > 600$ s or non-continuous flow
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 13302).

8.3.2 Condition the viscometer water bath (6.2) and, if used, the 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 $\pm 0,5$ °C.

8.3.3 Close the lower part of the cup 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 rod of the valve (Figure 3, Key element 2) 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.

8.3.4 Suspend the cup up to its rim in the water bath (6.3) or directly into the viscometer water bath (6.2) maintained within $\pm 0,5$ °C of the test temperature for a period of time sufficient to reach the test temperature.

8.3.5 If a separate water bath is used (6.3), remove the filled cup from the water bath and place it into the viscometer cup holder. Check that the sample has maintained the required temperature. If not, wait till equilibrium is reached again.

8.3.6 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. Remove the cork or stopper.

8.3.7 Pour light mineral oil into the receiver (6.5) 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 timing 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.

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