

SLOVENSKI STANDARD oSIST prEN 12846-1:2009

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Bitumen and bituminous binders - Determination of efflux time by the efflux viscometer - Part 1: Bituminous emulsions

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Ausflusszeit mittels Ausflussviskosimeter - Teil 1: Bitumenemulsionen

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ICS:

75.140 Voski, bitumni in drugi naftni Waxes, bituminous materials

proizvodi and other petroleum products

91.100.50 Veziva. Tesnilni materiali Binders. Sealing materials

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Bitumen and bituminous binders - Determination of efflux time by the efflux viscometer - Part 1: Bituminous emulsions

Bitumes et liants bitumineux - Détermination du temps d'écoulement à l'aide d'un viscosimètre à écoulement -Partie 1: Emulsions de bitume Bitumen und bitumenhaltige Bindemittel - Bestimmung der Ausflusszeit mittels Ausflussviskosimeter - Teil 1: Bitumenemulsionen

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

If this draft becomes a European Standard, 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.

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

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Foreword

This document (prEN 12846-1:2008) 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 12846:2002.

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.

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1 Scope

This European Standard specifies a method for the determination of the efflux time at 40 °C of bituminous emulsions in seconds using an efflux viscometer. Alternative test temperature is 50 °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 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 indication of the pseudoviscosity, which is defined as the internal resistance to flow.

4 Principle

The viscosity of a bituminous emulsion 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 10 mm or a 4 mm or a 2 mm orifice at a specified temperature.

Whatever temperatures or orifice diameters used, the efflux time shall not exceed 600 s. For highly viscous emulsions, EN 14896 shall be used.

5 Reagents and materials

5.1 Solution Sa

Aqueous solution with a nominal 1 % mass of laurylsulfate sodium, or in default aqueous solution with a nominal 1 % mass of sodium oleate shall be used for testing anionic emulsions. Those solutions shall be added with 1 % mass of sodium hydroxide (NaOH). The actual soap solution may be used if available on site, in case of routine tests or for simplicity.

NOTE S_a stands for "anionic solution" as this solution is used for testing anionic emulsions.

5.2 Solution S_c

Aqueous solution with a nominal 1 % mass of alkyltrimethylammonium chloride, or in default aqueous solution with a nominal 1 % mass of cetyltrimethylammonium bromide shall be used for testing cationic emulsions. Those solutions shall be added with 1 % mass of hydrochloric acid (HCI) (HCI concentration shall be approximately equal to 1 mol/l). The actual aqueous phase may be used if available on site, in case of routine tests or for simplicity.

NOTE S_C stands for "cationic solution" as this solution is used for testing cationic emulsions.

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). Three forms are required, differing only in the size of the orifice (10 mm, 4 mm and 2 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 a 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 equipped 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 evaporation of water.

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, maintained at (40.0 ± 0.5) °C, in which one or more of the viscometer cups can be immersed up to the rim of the cup.

NOTE Alternative test temperatures is (50.0 ± 0.5) °C.

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.

An appropriate climatic chamber may be used for low breaking index emulsions (see 8.3.2).

- **6.3 Viscometer water-bath**, constant temperature for maintaining the test temperature to within \pm 0,5 °C. A typical elevation and a plan of assembled viscometer are 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.

The test shall be carried out in duplicate.

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8 Procedure https://standards.iteh.ai/catalog/standards/sist/ed7b4029-c70d-4173-a70c-

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 pseudo-viscosity is unknown, measure it at 40 °C with the 4 mm orifice viscometer cup.

According to the efflux time obtained, 3 cases are possible. Choose the diameter of the orifice of the cup as follows.

1) If the efflux time is lower than 5 s, perform another determination at 40 °C with 2 mm orifice.

- 2) If the efflux time is greater than 5 s and still continuous flow, report the value obtained at 40 °C with 4 mm orifice.
- 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.

Orifice size, mm	Efflux time, s		
	Minimum	Maximum	
10 and 4 and 2	5	Non continuous flow or 600 s	

Table 1 — Diameter of the orifice of the cup

For highly viscous emulsions having efflux time at 40 °C with the 10 mm orifice higher than 600 s, test shall be performed at higher temperature (50 °C) or 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. 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 \pm 10) min.

For low breaking index emulsions, preconditioning during (90 \pm 10) min in the water bath (6.2) may be avoided. Preconditioning of the emulsion which has just been sampled and sieved (in accordance with EN 12594, sub-clause 7.3.3) in an appropriate receiver may be performed directly by placing this receiver in the climatic chamber (6.2). In that case, the viscometer-cup which is empty shall be heated in the conditions.

- **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 \pm 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 emulsion sample while removing the thermometer so that the final level of the binder is on the centre line of the levelling peg when the stem of the valve is in a vertical position.
- **8.3.8** Pour solution S_a (5.1) or solution S_c (5.2) 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 efflux time to the nearest 0.2 s.

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

9 Expression of results

Express the result 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 used (10 mm, 4 mm or 2 mm) and the test temperature (40 °C, 50 °C).

10 Precision

NOTE The precision of the method was evaluated with 4 mm and 10 mm cups only. With the 2 mm cup, the precision is not available.

10.1 Repeatability

The difference between two successive 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 following values in only one case in twenty.

10.2 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 following values in only one case in twenty.

 Viscosity
 Repeatability
 Reproducibility

 below 20
 73ac 7a12ca7/s11 s_n-12846
 -2011 2 s

 20 to 40
 2 s
 10 % of the mean

 above 40
 5 % of the mean
 10 % of the mean

Table 2 — Precision

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 the sampling and date of the 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.