SLOVENSKI STANDARD
oSIST prEN 12846-1:2021

01-oktober-2021

Bitumen in bitumenska veziva - Določanje viskoznosti z iztočnim viskozimetrom - 1. del: Bitumenske emulzije

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

Bitumes et liants bitumineux - Détermination du temps d'écoulement à l'aide d'un viscosimètre à écoulement - Partie 1 : Émulsions bitumineuses

Ta slovenski standard je istoveten z: **prEN 12846-1**

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

oSIST prEN 12846-1:2021 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 12846-1

August 2021

ICS 91.100.50

Will supersede EN 12846-1:2011

English Version

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 : Émulsions bitumineuses

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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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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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European foreword

This document (prEN 12846-1:2021) 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 12846-1:2011.

The main technical changes are:

— possibility to use a vegetal oil, requirement on density replaces requirement on viscosity (5.1);

— the use of Sa and Sc solutions has been abandoned (5.2, 5.3 and 8.3.9);

— Figures 1 and 2 have been modified to be in line with the evolution of test equipment (6.1 and 6.2);

— possibility to use alternate working apparatus that meet the requirements of this document, such as for example an integrated block viscometer (6.1);

— mercury stem thermometers are no longer specified as reference thermometers (6.4) and Annex A has been removed;

— emulsion samples, viscometer cups, valves and lids are preconditioned at test temperature to minimize time of presence of the emulsion sample in the viscometer cup (8.3.3 and 8.3.4);

— more accurate description of measurement procedure (8.3);

— precision data have been re-evaluated based on more comprehensive operational experience (10).

The EN 12846 series 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.*

1 Scope

This document 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.

NOTE The procedure described in this document can also be followed to determine efflux time at other temperatures such as 25 °C.

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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)*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at https://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

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 1 to entry: The efflux time is an indirect measure of the viscosity and is also referred to as “pseudo-viscosity”.

4 Principle

The efflux time 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 13302 shall be used.

5 Reagents and materials

5.1 Light mineral or vegetal oil.

Light mineral or vegetal oil having a density lower or equal to 900 kg/m^3 at 15 °C.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following.

6.1 Efflux viscometer (example of assembled 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 (Figure 3).

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 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 equipped 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 prevent evaporation of water and minimize surface cooling effects. This lid shall not touch the test material when the cup is filled. It is provided with a central hole through which the temperature measuring device can pass and with a groove on one side through which the rod of the valve can pass.

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 (Figure 2).

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 [1] are possible materials for the rod of the ball valve.

It is allowed to use working apparatus with other technical solutions that meet the requirements of this document, such as for example an integrated block viscometer, which is a combination of a specific efflux viscometer, thermostating element and temperature controller.

6.2 Viscometer water-bath, capable of maintaining the test temperature of 40 °C or 50 °C (8.3.1) constant to within ± 0,5 °C. Cups shall be separated from each other and from the walls of the bath by at least 55 mm.

6.3 Temperature controlled water bath, capable of maintaining the test temperature (6.2) constant to within ± 0,5 °C.

If the water bath is to be used for the preconditioning of the empty cups (8.3.4) or the conditioning of the filled cups (8.3.6), a suitable support shall be provided to maintain the cup(s) in a vertical position. Cups shall be separated from each other and from the walls of the bath by at least 55 mm.

For the preconditioning of emulsion samples and viscometer cups (8.3.3), the water bath may be replaced by an appropriate climatic chamber capable of maintaining the test temperature constant to ± 1 °C.

6.4 Temperature measuring devices (combining sensor and reading unit), two, which shall:

— have a range from at least 10 °C to 60 °C;

— be readable to 0,2 °C or less;

— have an accuracy of ± 0,2 °C or better;

— have a suitable length so that the measuring tip may be immersed at mid-height of the viscometer-bath, water-bath and viscometer-cup (8.3.5).

Sensors based on platinum resistance thermometers have been found suitable but other principles are also allowed. A solid stem mercury thermometer thermometer (such as IP 8C, ASTM 19C and ASTM 17C which used to be the former reference thermometers) is also allowed if national regulations permit its use.

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 ± 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.

8 Procedure

8.1 General

Carry out the procedure in laboratory at room temperature between 18 °C to 28 °C.

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 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 or equal to 5 s and lower than or equal to 600 s with still a 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 50 °C with the 4 mm orifice or at 40 °C with the 10 mm orifice or by means of dynamic viscosity measurement (EN 13302).

Table 1 — Diameter of the orifice of the cup

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

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 ± 0,5 °C.

8.3.3 Precondition the emulsion which has just been sampled and sieved (in accordance with EN 12594) in an appropriate receiver by directly placing this receiver in the water bath or into a climatic chamber set to the test temperature (6.3) for a period of time sufficient to reach the test temperature.

8.3.4 Precondition the empty viscometer-cup, ball valve and lid in either the viscometer water-bath (6.2) or the separate water bath or climatic chamber (6.3) for a period of time sufficient to reach the test temperature. If the viscometer-cup is to be filled and maintained within the water bath prior to its transfer into the viscometer (8.3.6), close the lower part of the cup orifice with a cork or cap prior to it's placing into the water bath or climatic chamber.

8.3.5 If not already done, suspend the cup up to its rim in the viscometer water bath and start the procedure by placing 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. Pass the temperature measuring device (6.4) through the central hole so that its measuring tip is approximately at the geometric centre of the sample. Continue the procedure as from 8.3.7.

8.3.6 Apply the same procedure as in 8.3.5 to additional viscometer-cups which are to be maintained at test temperature in the separate water-bath (6.3) in the wait for being tested. Once ready for testing, remove the filled cup from the water bath and place it into the viscometer cup holder.

8.3.7 Check that the sample is still at the required temperature. If not, wait for the minimum time necessary to get again into the tolerance range (±0,5 °C) around the targeted test temperature.

8.3.8 Remove any excess emulsion sample while removing the temperature measuring device so that the final level of the binder is on the centre line of the levelling peg when the rod of the valve is in a vertical position. Remove the cork or stopper if used.

8.3.9 Pour the light mineral or vegetal oil (5.1) into the receiver (6.5) up to the 20 ml graduation mark and place the receiver 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 efflux time to the nearest 0,2 s.

8.3.10 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 the value for repeatability given in Table 2 under (Clause 10).

If the two results differ by more than the above specified values, repeat the whole procedure.

10 Precision

10.1 General

The precision of the method has been re-evaluated by TC 336/WG 2 in 2018/2019 with 2 mm and 4 mm cups [2]. With the 10 mm cup, precision is not available.

10.2 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 value given in Table 2 in only one case in twenty.

10.3 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.

Table 2 — Precision

Repeatability	Reproducibility
15 % of the mean	40 % of the mean