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Bitumen and bituminous binders — Determination of efflux time of bituminous emulsions using the Redwood No. II Viscometer

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Bitumen and bituminous binders - Determination of efflux time of bituminous emulsions using the Redwood No. II Viscometer

Bitumes et liants bitumineux - Mesure du temps d'écoulement des émulsions bitumineuses avec le Viscosimètre Redwood No. II

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Ausflusszeit von Bitumenemulsionen mit dem Redwood Nr. II-Viskosimeter

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Foreword

This document (EN 16345:2012) 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of an identical text or by endorsement, at the latest by January 2013, and conflicting national standards shall be withdrawn at the latest by May 2014.

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1 Scope

This European Standard specifies a method for the determination of the efflux time (in seconds) of a bituminous emulsion at 85 °C using the Redwood No. II Viscometer.

WARNING – The use of this European Standard can 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 to determine the applicability of regulatory limitations prior to use.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

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 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 the Redwood No. II Viscometer which determines the time of efflux of a 50 ml sample through a 3,8 mm orifice at 85 °C.

5 Reagents and materials

5.1 Spirit level.

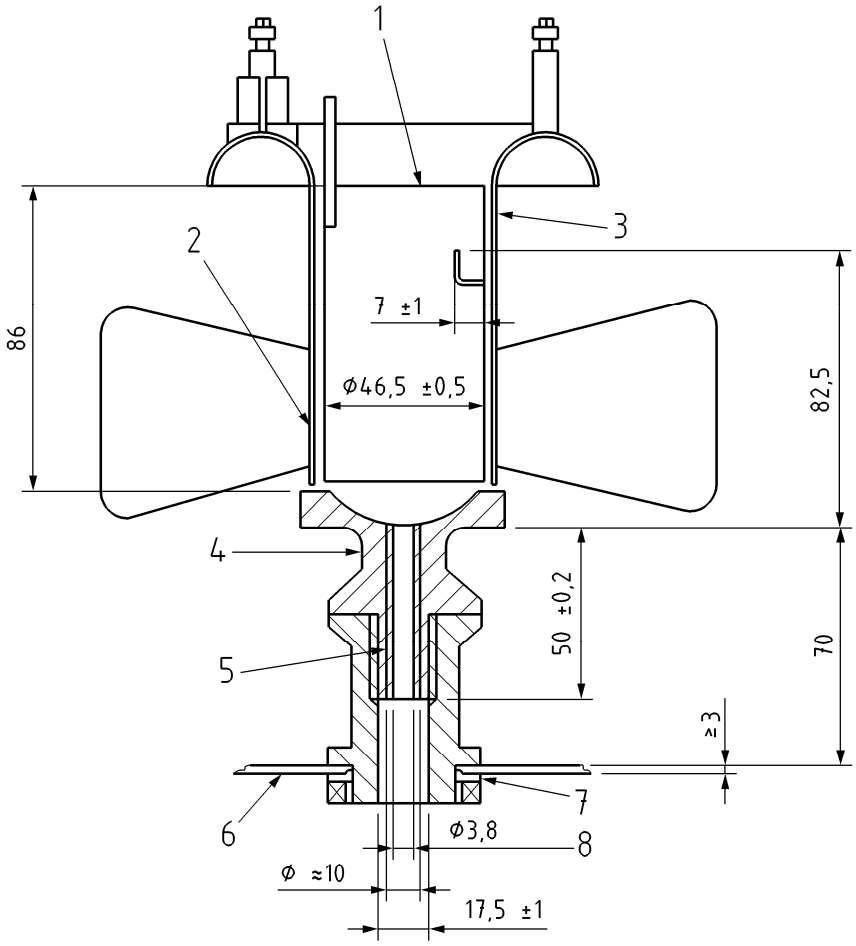
5.2 Soft tissue paper, or other material suitable for drying the oil cup, which will not leave any residue.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Standard Redwood No. II Viscometer, of the dimensions given in Figure 1, complete with ball-valve, oil cup cover, thermometer clip, stand and screen.

Dimensions in millimetres



Key

1 oil cup
2 stirrer body cut away between vanes
3 stirrer
4 brass tube
5 agate jet
6 bottom of heating bath
7 washer
8 minimum bore

Figure 1 — Redwood No. II Viscometer: section of cup

6.2 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.

6.3 Receiver, that is a Kohlrausch type flask, of (50 ± 0,5) ml capacity at 20 °C or a 100 ml cylinder with graduations at 25 ml and 75 ml.

6.4 Timing device / stopwatch, capable of reading to 0,2 s or less and accurate to 0,1 % over a period of not less than 15 min.

6.5 Loosely stoppered container, of at least 200 ml capacity.

6.6 Water/oil bath.

7 Sampling and preparation of sample

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

Place approximately 200 ml of the specimen in a suitable, clean, loosely stoppered container and bring to a temperature a little over the test temperature (85 °C) by immersing in a hot water or oil bath. Do not heat the specimen over a flame or by immersing hot bodies in it.

8 Procedure

8.1 General

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

8.2 Preparation of apparatus

Clean the oil cup with suitable solvents and dry thoroughly with soft tissue paper or other material which will not leave any residue.

Set up the viscometer, level by using a spirit level and fill the bath with water or oil to not less than 10 mm below the rim of the oil cup at the test temperature.

8.3 Measurement

Heat the viscometer bath to a few degrees above the test temperature. Homogenize the specimen by stirring and pour the prepared specimen into the oil cup. Adjust the temperature of the bath until the specimen in the cup is maintained at the test temperature of 85 °C, stirring the contents of the bath and cup during the process. Stir the specimen with the thermometer during the preliminary period. Do not stir the specimen during the actual determination.

When the temperature of the specimen has become steady at the test temperature, adjust the liquid level, if necessary, by removing any excess until the surface of the specimen touches the filling point. This should be achieved within 15 min of pouring the specimen into the oil cup in order to minimize loss of water by evaporation.

Slightly warm the oil cup cover, place it in position on the oil cup and swing the oil cup thermometer towards the closed end of the curved slot in the cover. Place the clean, dry standard 50 ml flask or 100 ml cylinder containing 25 ml of water below the jet with the neck a few millimetres from the bottom of the jet. Do not insulate the flask in any way.

Lift the ball-valve and simultaneously start the time recorder. Suspend the valve from the clip, supporting the oil cup thermometer by means of the hook in the wire stem. Stop the timing device at the instant the specimen reaches the 50 ml graduation mark on the flask or the 75 ml graduation on the cylinder and note the final reading of the oil cup thermometer.

Reject any determination if the temperature of the specimen in the oil cup varies during the run by more than 0,3 °C.

9 Expression of results

Express the efflux time in Redwood No. II seconds to the nearest 0,5 s.

10 Precision

10.1 Repeatability

In the long run and in normal and correct operation of the test method, the difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would exceed the value given in Table 1 in only one case in twenty.

10.2 Reproducibility

In the long run and in normal and correct operation of the test method, the difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would exceed the value given in Table 1 in only one case in twenty.

Table 1 — Repeatability and reproducibility values

Efflux time	Repeatability	Reproducibility
< 20	2 s	2 s
20-50	2 s	10 % of the mean
> 50	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 the sampling and date of the sample preparation);

b) reference to this European Standard;

c) test temperature (if different to the reference test temperature of 85 °C);

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.

Annex A
(normative)

Specifications of thermometer

a)	Temperature range	76 °C to + 122 °C
b)	Immersion	65 mm
c)	Scale marks:	
	1) Subdivisions	0,2 °C
	2) Long lines at each	1°C + 5 °C
	3) Numbers at each	5 °C
	4) Maximum line width	0,15 mm
	5) Scale error, maximum	0,2 °C
d)	Expansion chamber permitting heating to	Required
e)	Total length	330 mm to 350 mm
f)	Stem outside diameter	5,5 mm to 8,0 mm
g)	Bulb length	10 mm to 16 mm
h)	Bulb outside diameter	Not greater than stem
i)	Scale location:	
	1) Distance between bottom of bulb and line at 76 °C	115 mm minimum
	2) Length of scale range	150 mm to 190 mm

NOTE The thermometer IP-10C has been found to be suitable.

Bibliography

[1] EN 13302:2010, *Bitumen and bituminous binders — Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus*

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

[3] BS 593:1989, *Specification for laboratory thermometers*

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