SLOVENSKI STANDARD
oSIST prEN 13358:2018

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Bitumen in bitumenska veziva - Določevanje destilacijskih značilnosti rezanih in fluksiranih bitumenskih veziv

Bitumen and bituminous binders - Determination of the distillation characteristics of cut-back and fluxed bituminous binders made with mineral fluxes

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Destillationsverlaufes von mit Mineralölfluxmitteln verschnittenen oder gefluxten bitumenhaltigen Bindemitteln

Bitumes et liants bitumineux - Détermination des caractéristiques de distillation des bitumes fluidifiés et fluxés par des fluxants d'origine minérale

Ta slovenski standard je istoveten z: **prEN 13358**

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

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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 13358:2018) has been prepared by Technical Committee CEN/TC 336 “Bitumen and bituminous binders”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 13358:2010.

The main technical changes are:

— Clause 5: an electric heating plate or heating mantle may be used as alternatives to the gas burner;

— Clause 5: mercury stem thermometers are replaced by temperature measuring devices allowing similar temperature determinations to be made. Annex A (characteristics of mercury stem thermometer) becomes informative;

— Clause 6.4.2: requirements on distillation rate (drops per minute) may be replaced by a minimum rate of temperature increase in cases where the distillation process has a very low yield;

— Clause 6.4.3: volumes of distillate are to be recorded at least at the reference corrected temperatures mentioned in EN 15322;

— Clauses 6.4.3 and 10: the possibility to record volumes of distillate to a precision of 0,1 ml is abandoned.

This European Standard is based on ASTM D 402-97.

1 Scope

This document specifies a method for the determination of the distillation characteristics of cut-back and fluxed bituminous binders made with mineral fluxes.

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 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 15322, *Bitumen and bituminous binders - Framework for specifying cut-back and fluxed bituminous binders*

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 http://www.electropedia.org/

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

3.1
distillation
process of evaporation and condensation of a liquid

3.2
mineral flux
flux which may be of carbochemical or petrochemical origin or a mixture of both

4 Principle

Petroleum cut-back and fluxed bituminous binders are distilled at a controlled rate until the temperature of the liquid reaches 360 °C. The volumes of distillate and residue obtained over specified temperature ranges are measured. The residue from the distillation and also the distillate may be tested as required.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Distillation flask, 500 ml and with side-arm, of dimensions given in Figure 1.

5.2 Heating system - Equipment needed when using a gas burner or equivalent (see Figure 3).

5.2.1 Shield, of steel, lined with 3 mm of suitable thermal insulation material and fitted with transparent mica windows of the form and dimensions shown in Figure 2, which is used to protect the flask from air currents and to reduce radiation.

5.2.2 Shield cover (top), consisting of two parts of fire resistant millboard of 6 mm minimum thickness.

5.2.3 Support for shield and flask, two sheets 150 mm × 150 mm of a mesh of approximately 1 mm wire gauze on a tripod or ring.

5.2.4 Heat source, adjustable Tirrill gas burner or equivalent.

NOTE 1 It is advised to protect the gas burner flame using a chimney, as shown in Figure 3.

NOTE 2 Tirrill is an example of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product.

5.2.5 Height adjustable platform, to support the gas burner.

5.3 Heating system - Equipment needed when using an electric heater.

5.3.1 Shield and shield cover, as described under 5.2.1 and 5.2.2.

5.3.2 Electric heater (replacing items 6, 7 and 8 in Figure 3), with a heating plate adequately surfaced to support the distillation flask and designed so as to efficiently transfer the heat. The heating plate shall be of sufficient dimensions to support the shield over its entire base. The heater shall be equipped with a transformer capable of controlling from 0 to 750 W.

5.3.3 Height adjustable platform, to support the electric heater.

5.4 Heating system - Equipment needed when using an electric heating mantle.

5.4.1 Electric heating mantle (replacing items 4, 6, 7 and 8 in Figure 3), of adequate inner size to fit the 500 ml flask and completely embed the 200 ml product sample once filled into the flask (see 6.3.1). The heating mantle shall be completed with a removable insulation collar to cover the part of the distillation flask emerging from the heating mantle. The heating mantle shall be equipped with a transformer capable of controlling from 0 to 750 W.

5.4.2 Height adjustable platform, to support the electric heating mantle

5.5 Condenser, standard glass-jacketed of nominal jacket length from 200 mm to 300 mm and overall tube length of (450 ± 10) mm (see Figure 3).

5.6 Adapter, made with glass of approximately 1 mm thickness, with reinforced top, having an angle of approximately 105°. The inside diameter shall be (18 ± 2) mm at the wide end and not less than 5 mm at the narrow end. The lower surface of the adaptor shall be on a smooth descending curve from the wider end to the narrower. The inside line of the outlet end shall be vertical, and the outlet shall be cut or ground (not fire-polished) at an angle of (45 ± 5)° (see Figure 3).

In the equipment described in the above paragraphs and in Figure 1 and Figure 3, cork stoppers are used, however, equipment with ground glass joints may also be used.

5.7 Receivers, standard 25, 50 or 100 ml graduated cylinders (depending on expected amount of distillate) conforming to the dimensions given in Figure 4.

5.8 A temperature measuring device (combining sensor and reading unit) which shall:

— have a range from at least 10 °C to 400 °C;

— be readable to 1 °C or less;

— have an accuracy of 1 °C or better.

Sensors based on platinum resistance thermometers have been found suitable but other principles are also allowed. A solid stem mercury thermometer (which used to be the former reference thermometer as described in Annex A) is also allowed if national regulations permit its use.

The temperature measuring device shall be calibrated regularly.

For this test method, in which increasing temperatures are read during the test procedure, documented corrections shall be determined in advance and applied to the observed readings.

5.9 Residue container, of approximately 250 ml capacity. A seamless metal container, with a slip-on cover of (75 ± 5) mm diameter and (55 ± 5) mm height, or similar, is suitable.

5.10 Balance with a reading accuracy of at least 0,1 g.

6 Procedure

6.1 General

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

6.2 Preparation of test samples

6.2.1 Samples shall be prepared in accordance with the provisions detailed in EN 12594, especially those regarding cut-back and fluxed bituminous binders. In particular, stir the sample thoroughly, warming if necessary, to ensure homogeneity before removal of a representative portion for analysis.

6.2.2 If sufficient water is present to cause foaming or bumping, dehydrate a sample of not less than 250 ml by heating in a distillation flask sufficiently large to prevent foaming over into the side-arm. When foaming has ceased, stop the heating. If any light oil has distilled over, separate and pour back into the flask when the contents have cooled down sufficiently to prevent loss of volatile oil. Mix the contents of the flask thoroughly before removal for analysis.

6.3 Preparation of apparatus

6.3.1 Calculate the mass of 200 ml of the sample from the density of the material at 15 °C. Weigh this amount with an accuracy of ± 0,5 g into the 500 ml distillation flask (5.1).

NOTE Sufficient warming of the sample is necessary to allow the pouring into the flask.

6.2.3 Place the flask in the shield (5.2.1 and 5.2.2) on its support (5.2.3), or on the electrical heater (5.3.2 and 5.3.3) or place the flask into the heating mantle (5.4.1 and 5.4.2). Connect the condenser tube (5.5) to the side-arm of the flask with a cork stopper or equivalent, keeping the centre of its neck vertical. Adjust the adapter (5.6) over the end of the condenser tube so that the distance from the neck of the distillation flask to the adapter outlet is (650 ± 50) mm.

6.3.3 Insert the temperature measuring device through a tight-fitting cork in the neck of the distillation flask so that its end rests on the bottom of the flask. Raise the temperature measuring device (6,5 ± 1,0) mm from the bottom of the flask using the top of the cork as reference.

6.3.4 Place the receiver (5.7) so that the adapter extends into it by at least 25 mm but not to below the upper level mark. Cover the neck of the receiver with a piece of suitably weighted blotting paper, or similar material, cut to fit the adapter snugly.

6.3.5 Ensure that the flask, condenser, adapter and receiver are clean and dry. Place the residue container and its cover in an area free from drafts.

6.3.6 Pass cold water through the condenser jacket by reflux. Use warm water if necessary to prevent the formation of solid condensate in the condenser tube.

6.4 Test procedure

6.4.1 If the barometric pressure, in Pascals, at the time of the test is known, correct the temperature according to Table 1. Do not correct for the emergent stem of the thermometer. If the barometric pressure is not known, and the elevation of the laboratory is more than 150 m above sea level, correct the temperature according to Table 2.

NOTE 1 Only one correction is used each time.

NOTE 2 Standard pressure is 1 013 hPa.

Table 1 — Factors for calculating temperature corrections

Nominal temperature (°C)	Correction [a] per 13,33 hPa difference in pressure
160	0,514
175	0,531
190	0,549
225	0,591
250	0,620
260	0,632
275	0,650
300	0,680
316	0,698
325	0,709
360	0,751
[a] To be subtracted when the barometric pressure is below 1 013 hPa; to be added when the barometric pressure is above 1 013 hPa.	

NOTE 2 For the temperature correction, it is convenient to take into account that experimental temperatures can only be read at the nearest 1 °C.

Table 2 — Corrected fractionation temperature for various altitudes

Elevation above sea level (m)	Fractionation temperature for various altitudes (°C)				
- 305	192	227	263	318	362
- 152	191	226	261	317	361
0	190	225	260	316	360
152	189	224	259	315	359
305	189	224	258	314	358
457	188	223	258	313	357
610	187	222	257	312	356
762	186	221	256	312	355
914	186	220	255	311	354
1 067	185	220	254	310	353
1 219	184	219	254	309	352
1 372	184	218	253	308	351
1 524	183	218	252	307	350
1 676	182	217	251	306	349
1 829	182	216	250	305	349
1 981	181	215	250	305	348
2 134	180	214	249	304	347
2 286	180	214	248	303	346
2 438	179	213	248	302	345

6.4.2 Apply heat such that the first drop of distillate falls from the end of the distillation flask side-arm within 5 min to 15 min. Conduct the distillation so as to maintain the following drop rates, at the adapter tip (all temperatures refer to corrected temperatures):

— up to 260 °C, 50 drops per minute to 70 drops per minute;

— from 260 °C to 316 °C, 20 drops per minute to 70 drops per minute;

— from 316 °C to 360 °C, complete the distillation within 10 min.

Some cut-back and fluxed bituminous binders yield either no distillate or very little distillate over portions of the temperature range to 316 °C. In this case, it becomes impractical to maintain the above distillation rates. For such cases, the intent of the method shall be met if the rate of rise of temperature exceeds 5 °C/min.

NOTE The addition of some pumice-stone into the distillation flask prior to the introduction of the test sample usually improves the smoothness of the distillation process.

6.4.3 Record the volumes of distillate to the nearest 0,5 ml in the receiver at the corrected temperatures. The reference corrected temperatures to be considered are 190 °C, 225 °C, 260 °C, and 316 °C but additional temperatures may be considered as well

6.4.4 When the temperature reaches the corrected temperature of 360 °C, extinguish the flame or electric heating device and remove the flask and temperature measuring device. With the flask in a pouring position, remove the temperature measuring device and then pour the contents into the residue