SLOVENSKI STANDARD
SIST EN 12606-1:2015

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SIST EN 12606-1:2007

Bitumen in bitumenska veziva - Določevanje parafina - 1. del: Metoda destilacije

Bitumen and bituminous binders - Determination of the paraffin wax content - Part 1: Method by distillation

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Paraffingehaltes - Teil 1: Destillationsverfahren

Bitumes et liants bitumineux - Détermination de la teneur en paraffines - Partie 1 : Méthode par distillation

Ta slovenski standard je istoveten z: **EN 12606-1:2015**

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 the paraffin wax content - Part 1: Method by distillation

Bitumes et liants bitumineux - Détermination de la teneur en paraffines - Partie 1 : Méthode par distillation

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Paraffingehaltes - Teil 1: Destillationsverfahren

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Page

Foreword

This document (EN 12606-1:2015) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

This document supersedes EN 12606-1:2007.

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of an identical text or by endorsement, at the latest by January 2016, and conflicting national standards shall be withdrawn at the latest by January 2016.

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.

The standard has been revised due to a normative reference to mercury thermometers. The changes involve mainly the text in 6.2. The terms “sample thermometer” and “bath thermometer” have been changed, as a consequence, to “sample temperature transducer” and “bath temperature transducer” respectively in some subparagraphs of Clause 8.

This draft European standard EN 12606 consists of the following parts under the general title *Bitumen and bituminous binders – Determination of the paraffin wax content*

— Part 1: Method by distillation;

— Part 2: Method by extraction.

The changes introduced mainly involve the revision of the text in 6.2 relating to the mentioning of mercury thermometers as reference thermometers.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a procedure for determining the paraffin wax content of bitumen and bituminous binder by the DIN method.

Aqueous bituminous binders, fluxed or cut-back anhydrous binders, and modified binders, whatever their consistency, are not within the scope of the present test method.

WARNING — 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, 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 1425, *Bitumen and bituminous binders - Characterization of perceptible properties*

EN 12594, *Bitumen and bituminous binders - Preparation of test samples*

EN ISO 3696:1995, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

ISO 383, *Laboratory glassware — Interchangeable conical ground joints*

3 Terms and definitions

For the purposes of this document, the following term and definition applies.

3.1
paraffin wax
mixture of hydrocarbons crystallising in a 50 % volume fraction mixture of ether/ethanol at temperatures down to - 20 °C, obtained by a specified process and having a melting range of above 25 °C

4 Principle

Paraffin wax present in bitumen is determined in the distillate obtained from a specified distillation process.

5 Reagents and materials

5.1 General

Use only reagents of recognized analytical grade and water conforming to grade 3 of EN ISO 3696:1995 unless otherwise specified.

5.2 Ethoxyethane (Diethylether), anhydrous, referred to in this standard as ether.

5.3 Ethanol, absolute.

5.4 Ethanol, technical grade.

5.5 Petroleum spirit, with density of approximately 645 kg/m³ at 15 °C, and a distillation range of approximately 30 °C to 75 °C.

5.6 Acetone, reagent grade.

5.7 Carbon dioxide, solid, finely ground.

NOTE A cryostat with a cooling effect equivalent to that of solid carbon dioxide can be used.

5.8 Ice, finely ground.

5.9 Washing liquid, a 50 % volume fraction mixture of ether/ethanol.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following: (Dimensions in this clause are approximate unless tolerances are stated).

6.1 Oven, capable of maintaining (125 ± 5) °C.

6.2 Temperature measuring devices, (combining reading device and transducers) referred to in this standard as:

6.2.1 Sample temperature transducer: a sensor which together with a reading device shall have a measuring range of –38 °C to 50 °C, be readable to the nearest 1,0 °C with an accuracy of 0,5 °C. The sensor shall be embedded in a glass rod with total length (360 ± 5) mm and outside diameter (10,0 ± 0,5) mm.

6.2.2 Bath temperature transducer: a sensor which together with a reading device shall have a measuring range of –30 °C to 50 °C, be readable to the nearest 0,5 °C with an accuracy of 0,5 °C. The sensor shall be embedded in a glass rod with total length (220 ± 5) mm and outside diameter (8,0 ± 0,5) mm.

The temperature measuring devices employed shall be calibrated regularly.

6.3 Distillation flask as shown in Figure 1, fitted with cork stopper.

6.4 Sheet metal guard ring with an approximate 18 mm inside diameter and a 65 mm outside diameter.

6.5 Test tubes, fitted with a spout and bored cork stopper; dimensions are given in Figure 2.

6.6 Test tubes, fitted with a 29/32 ground socket and a wash bottle fitted with a 29/32 ground cone according to ISO 383; dimensions are given in Figure 2.

6.7 Erlenmeyer flask, 100 ml, to be used as distillation receiver fitted with a bored cork stopper continuously vertically notched on the outer surface.

6.8 Filter flask, 500 ml, with vacuum unit.

6.9 Glass wash bottle, 500 ml.

6.10 Cooling bath (see an example on Figure 2), a different cooling bath with the same isolation effect can also be used.

6.11 Funnel (in cooling bath), with a diameter of 72 mm and a total length of 200 mm. A jacketed funnel with similar dimensions, connected to the cooling bath, is also allowed.

6.12 Evaporating basin, diameter 80 mm, mark at 15 mm.

6.13 Balance, accurate to ± 10 mg, readable to 1 mg.

6.14 Balance, accurate to ± 2,0 mg, readable to 0,1 mg.

6.15 Laboratory burner (see Figure 3).

6.16 Desiccator

6.17 Porcelain crucible, with a diameter of 80 mm.

6.18 Round filter, with a diameter of 110 mm, for quantitative analysis, transmission: middle close or middle rapid flowing.

6.19 Timer

6.20 Pincers

7 Sampling

Make sure that the laboratory sample is homogeneous and is not contaminated (see EN 1425). Take all necessary safety precautions and ensure that the test sample is representative of the laboratory sample from which it is taken (see EN 58). The laboratory sample shall be taken in accordance with EN 58.

The test shall be carried out on two portions each of (25 ± 1) g. Prepare the test sample in accordance with EN 12594.

8 Procedure

Melt each test portion in a porcelain crucible for the minimum time necessary to ensure that the sample is completely fluid. Pour (25 ± 1) g into the distillation flask (6.3) and weigh to the nearest 10 mg (mass m_B).

Heat the distillation flask with a laboratory burner with a soft flame, approximately 150 mm high, (without a flame cone) that has just ceased to be luminous in such a way that the first distillate drop falls down after 3 min to 4 min. Fit the sheet metal guard ring (see 6.4) loosely on the distillation flask to prevent possible burning of the cork stopper.

Ensure that the vapours produced during distillation are being condensed by weighing, to the nearest 10 mg, the Erlenmeyer flask (6.7) into which the lower bent end of the outlet tube projects to its full length

(see Figure 2), and immerse this as far as possible into a mixture of finely ground ice and water. The rate of distillation shall remain visible and capable of being checked.

Adjust the distillation rate so that (15 ± 5) drops fall from the end of the outlet tube into the distillation receiver every 10 s.

Continue heating without adjusting the laboratory burner flame, until the distillation rate slows and no drop falls from the outlet tube over a period of 10 s or after 14 min from the start of distillation.

Continue heating for a further min with a completely non-luminous roaring flame until the flask glows red.

Complete the distillation in a maximum of 15 min. Do not transfer the condensate left in the outlet tube after distillation to the distillation receiver.

Mix the distillate thoroughly by gently warming it whilst at the same time, carefully swirling the receiver.

Cool the receiver to ambient temperature in a desiccator and weigh the distillate contained in the receiver to the nearest 10 mg (mass m_D). Depending on the expected paraffin wax content, add 2 g to 4 g of the distillate into a test tube (6.5) and weigh to the nearest 5 mg (mass m_E).

NOTE 1 If the paraffin wax content cannot be estimated in advance, an initial mass of distillate of approximately 3 g is recommended.

Dissolve the weighed mass of distillate in (25 ± 1) ml of ether (5.2) and add (25 ± 1) ml of ethanol (5.3).

Close the test tube with a stopper fitted with a sample temperature transducer (6.2.1) extending down into the liquid and place the test tube in a cooling bath. Cool the bath liquid by adding finely crushed solid carbon dioxide or with a cryostat (see note to 5.7). To ensure the sample temperature is maintained at - 20 °C, which will be required later, lower the bath temperature to (- 22 ± 1) °C. Transfer (20 ± 1) ml of the washing liquid (5.9) into the test tube fitted with the wash bottle head and cool in the cooling bath (6.10) to (- 20,0 ± 0,5)°C, maintain this temperature until filtration is complete.

Place the round filter (6.18) in the funnel standing in the cooling bath and connect it to the filter flask placed below the cooling bath. Quickly transfer the slurry of crystals produced at (- 20,0 ± 0,5) °C to the filter. Rinse the test tube with the cooled washing liquid. Re-adjust the temperature of the washing liquid to (- 20,0 ± 0,5) °C and use it again for rinsing the crystal slurry into the filter. Distribute the washing liquid as uniformly as possible between the three washing operations.

Support the filtration by a gentle vacuum process during which the pressure does not fall below 5 kPa. As soon as filtration is complete, lift off the round filter using pincers and place it in the funnel situated over the evaporating basin or Erlenmeyer Flask, which has been weighed previously to the nearest 0,5 mg. Dissolve the crude paraffin wax residue by carefully spraying heated petroleum spirit over it. Dissolve in the same way any paraffin wax that may be adhering to the temperature transducer or to the test tube. Evaporate the mixed filtrates in the evaporating basin over the water bath. To prevent liquid creeping over the rim, carry out the evaporation in a weak air stream. Dry the residue for (15 ± 1) min at (125 ± 5) °C in the oven and allow it to cool. When the previously purified paraffin waxes have cooled down but have not quite solidified, add approximately 15 ml of acetone.

Dissolve the paraffin waxes by gently heating and carefully swirling the evaporating basin. Make up any acetone lost by evaporation. Cool the acetone/paraffin wax solution in a water bath to (15,0 ± 0,5) °C and separate by filtering the paraffin waxes that crystallize out. Wash the evaporating basin, the temperature transducer and the filter several times with acetone brought to (15,0 ± 0,5) °C from a wash bottle ensuring that the total volume of washing liquid is (30 ± 1) ml. The washing liquid shall be discarded.

Dissolve the paraffin waxes purified in this way by carefully spraying them with heated petroleum spirit and collect them again in the same evaporating basin. Evaporate the petroleum spirit/paraffin wax solution in a weak air stream over the water bath.