SLOVENSKI STANDARD
SIST EN 12606-1:2000

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Bitumen in bitumenska veziva - Določanje 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

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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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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

This European Standard was approved by CEN on 5 September 1999.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

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of an identical text or by endorsement, at the latest by April 2000, and conflicting national standards shall be withdrawn at the latest by April 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

This draft European standard is based upon DIN 52015 :1980.

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

1 Scope

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

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

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 58, *Sampling bituminous binders*

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

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

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

ISO 2207, *Petroleum waxes- Determination of congealing point*

3 Terms and definitions

For the purposes of this European Standard, the following definition applies.

3.1
paraffin wax
mixture of hydrocarbons crystallizing in an ether/ethanol 50 % *(V/V)* mixture at temperatures down to -20 °C, obtained by a specified process and having a range of melting 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 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 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, mixture of ether/ethanol 50 % (*V/V*).

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Oven, capable of maintaining a temperature of 125 °C ± 5 °C;

6.2 Thermometers, referred to in this standard as:

6.2.1 Sample thermometer: a solid stem thermometer, in the range -38 °C to 50 °C, and with a subdivision every 1 °C; total length 360 mm ± 5 mm, immersion 180 mm ± 5 mm, stem outside diameter 10 mm ± 0,5 mm.

6.2.2 Bath thermometer: a solid stem thermometer, in the range -30 °C to 50 °C, and with a subdivision every 0,5 °C, total length 220 mm ± 5 mm, immersion 50 mm ± 5 mm, stem outside diameter 8 mm ± 0,5 mm.

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.

NOTE 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.

6.3 Distillation flask as shown in figure 1, fitted with cork stopper;

6.4 Sheet metal guard ring of about 18 mm inside diameter and approximately 65 mm outside diameter;

6.5 Test tubes, dimensions of which are given in figure 2, fitted with sput and bored cork stopper;

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

6.7 Erlenmeyer flask, 100 ml, to be used as distillation receiver fitted with a bored cork stopper continously 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 figure 2);

6.11 Funnel (in cooling bath), diameter 80 mm, total length 200 mm;

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

6.13 Balance, accurate to ± 5 mg;

6.14 Balance, accurate to ± 0,5 mg;

6.15 Laboratory burner (see figure 3);

6.16 Desiccator;

6.17 Porcelain casserole, diameter 80 mm;

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

6.19 Timer, accurate to the nearest 0,1 s;

6.20 Pincers.

7 Sampling

Laboratory samples shall be taken in accordance with EN 58 and/or in accordance with the requirements of national standards or regulations. The national requirements for sampling shall be set out in detail of shall be referred to by a reference in a national annex to this European Standard.
The test shall be carried out on two portions each of 25 g ± 1 g prepared in accordance with EN 12594.

8 Procedure

Melt each test portion in a porcelain crucible in accordance with EN 12594, pour 25 g ± 1 g into the distillation flask (6.3) and weight 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 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 adjustment of 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 commencement of distillation.

Continue heating for a further minute 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 in a desiccator to ambient temperature 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 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 ml ± 1 ml of ether (5.1) and add 25 ml ± 1 ml of ethanol (5.2).

Close the test tube with a stopper fitted with a sample thermometer (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 maintenance of the sample temperature of - 20 °C that will be required later, lower the bath temperature to -22 °C ± 1 °C. Transfer 20 ml ± 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 °C ± 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 °C ± 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 °C ± 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 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 thermometer 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 min ± 1 min at 125 °C ± 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 °C ± 0,5 °C and separate by filtering the paraffin waxes that crystallize out. Wash the evaporating basin, the thermometer and the filter several times with acetone brought to 15 °C ± 0,5 °C from a wash bottle ensuring that the total volume of washing liquid is 30 ml ± 1 ml.

Dissolve the paraffin waxes purified in this wax 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.

Dry the crystallised paraffin waxes obtained in this way for 15 min ± 1 min at 125 °C ± 5 °C in the oven and, after cooling in the desiccator, determine the mass to the nearest 0,5 mg (mass m_A).

If the final mass is outside the range 65 mg to 85 mg, reject the result and repeat the test with an appropriately adjusted initial mass of the same distillate (mass m_E).

If the initial mass, which is required to give a final mass between 65 mg and 85 mg, is below 2 g or above 4 g, record this in the test report.

NOTE For cooling purposes an automatic cooling device can be used provided the same results are obtained.

9 Calculation

Calculate the paraffin wax content C_p, for each test portion, expressed as a percentage by mass, using the following equation:

$$C_p = \frac{m_D \times m_A}{m_B \times m_E} \times 100$$

where

m_B is the initial mass of bitumen, in grams;

m_D is the mass of distillate, in grams;

m_E is the initial mass of distillate, in grams;

m_A is the final mass of paraffin wax, in grams.