SLOVENSKI STANDARD
SIST EN 12606-2:2000

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Bitumen in bitumenska veziva - Določ evanje parafina - 2. del: Metoda ekstrakcije

Bitumen and bituminous binders - Determination of the paraffin wax content - Part 2: Method of extraction

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Paraffingehaltes - Teil 2: Extraktionsverfahren

Bitumes et liants bitumineux - Détermination de la teneur en paraffines - Partie 2: Méthode par extraction

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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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EUROPEAN STANDARD

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English version

Bitumen and bituminous binders - Determination of the paraffin wax content - Part 2: Method by extraction

Bitumes et liants bitumineux - Détermination de la teneur en paraffines - Partie 2: Méthode par extraction

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Paraffingehaltes - Teil 2: Extraktionsverfahren

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.

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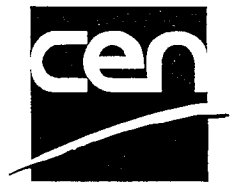

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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

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of an identical text or by endorsement, at the latest by April 2000, and conflicting national standards shall be withdrawn at the latest by April 2000.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 AFNOR NF T 66-015:1984.

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 binders by the AFNOR 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 4793, *Laboratory sintered (fritted) filters - Porosity grading, classification and designation*

ISO 5272, *Toluene for industrial use - Specifications.*

3 Terms and definitions

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

3.1
paraffin wax
hydrocarbons crystallising 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 after the extraction of asphaltenes with petroleum spirit and the extraction of most aromatic components with oleum. The extracted sample is dissolved in a mixture of ethanol and ether of sufficient volume for all of the oily components to remain in solution when it is later cooled to crystallize the paraffin wax. The solution is cooled to -20 °C ± 0,5 °C and the crystallized paraffin wax filtered off and collected in a filtering crucible, itself cooled to -20 °C ± 0,5 °C. The paraffin wax crystals are re-dissolved in warm toluene, which is evaporated and the paraffin wax residue weighed.

5 Reagents and materials

Use only reagents of recognized analytical grade and water conforming to grade 3 of EN ISO 3696.

5.1 Toluene conforming to ISO 5272;

5.2 Sulphuric acid at 96% concentration (density at 20°C = 1 835 kg/m³);

5.3 Oleum, 60 % SO_3 (*V/V*) in H_2SO_4;

5.4 Ethanol, 99 % *(V/V)*;

5.5 Ethoxyethane (diethyl ether), anhydrous;

5.6 Sodium hydroxide, pellets;

5.7 Phenolphthalein, solution of 1 % phenolphthalein in 99 % ethanol;

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

NOTE The product referred to as petroleum ether is suitable.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Erlenmeyer flasks, 150 ml stoppered ;

6.2 Filtering crucible with a calcined glass plate, having a porosity of P16 (see ISO 4793);

6.3 Vacuum flask, 500 ml, and pumping system;

6.4 Separating funnel, 500 ml;

6.5 Crystallizer (approximately 50 mm diameter);

6.6 Balance accurate to ± 2 mg, readable to 0,1 mg;

6.7 Insulated receptacle containing a refrigerating liquid maintained at a temperature of -20 °C ± 0,5 °C by a suitable device;

6.8 Water-bath, regulated to 30 °C – 40 °C, for evaporating the solutions ;

6.9 Oven regulated to 100 °C – 110 °C.

6.10 Thermometers, referred to in this standard as:

6.10.1 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.

6.10.2 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.

7 Sampling

Laboratory samples shall be taken in accordance with EN 58. Any national requirements for sampling shall be set out in detail or shall be referred to by a reference in a national annex to this European Standard. Prepare the sample in accordance with EN 12594.

8 Procedure

Using the balance (6.6), weigh approximately 1 g of bitumen, to the nearest milligram, into each of the Erlenmeyer flasks (6.1), dried and weighed with their stoppers, to give masses m_1 and m'_1 for the two samples respectively.

Add 70 ml ± 2 ml of petroleum spirit (5.8) to each of the bitumen samples and stir until they have completely dissolved.

Place the stoppered flasks in an inclined position and leave for 48 h away from direct light.

Filter the mixture under vacuum using the crucible (6.2), and wash the precipitate (containing the asphaltenes) with petroleum spirit until a practically colourless filtrate is obtained (70 ml ± 2 ml).

Pour the solution of petroleum spirit (containing the maltenes and the paraffin) into a separating funnel (6.4).

Add 30 ml ± 2 ml of an acid mixture composed of 2/3 sulphuric acid (5.2) and 1/3 oleum (5.3).
Shake slowly to prevent rapid reaction and the formation of a stable emulsion, then shake vigorously, taking care to hold the funnel at an angle in order to be able to open the tap to release petroleum spirit vapours that form as the result of exothermic reaction.

Allow to settle 12 h ± 4 h, then decant the acid layer. Add 30 ml ± 2 ml of sulphuric acid (5.2), shake as before, and decant again after 4 h ± 2 h. If the acid layer is coloured, repeat the sulphuric acid treatment.

Rinse the solution left in the separating funnel, firstly with water, then with an alcoholic solution of 5 % sodium hydroxide (50 ml ± 2 ml water, 50 ml ± 2 ml ethanol (5.4) and 5 g ± 0,1 g sodium hydroxide (5.6)) and repeatedly with water, until there is no alkaline reaction of the water layer to phenolphthalein indicator (5.7).

Pour the solution into an Erlenmeyer flask and evaporate the petroleum spirit (5.8).

Dissolve the residue in 50 ml ± 2 ml of ethoxyethane (5.5) then add 50 ml ± 2 ml of ethanol (5.4).

Leave for 1 hour at -20 °C ± 0,5 °C in the insulated receptacle (6.7).

At the same time, cool an Erlenmeyer flask containing 60 ml ± 2 ml of a mixture of ethanol/ethoxyethane (in equal amounts).

Using a filtering crucible that has been cooled to -20 °C ± 0,5 °C, filter the mixture under vacuum in less than 5 min. If the solution does not filter within this time, the paraffin, which crystallizes at -20 °C ± 0,5 °C, will re-dissolve and the test shall be invalidated. Rinse three times with 20 ml ± 2 ml of the ethanol/ethoxyethane mixture cooled to -20 °C ± 0,5 °C until the oily components are eliminated.

Leave the filtering crucible for approximately 1 hour in an oven heated to 100 °C to 110 °C, then place in a desiccator.

Using as little quantity of warm toluene as possible, dissolve the paraffin wax and collect the solution in a weighed crystallizer.
Evaporate the solvent and weigh the paraffin wax to the nearest milligram. This gives masses m_2 and m'_2, for the two samples respectively.

9 Calculation

Calculate the paraffin wax contents of the two samples (C_p, C'_p) as percentages by mass using the equations :

$$C_p = 100 \times m_2 / m_1$$

$$C_p = 100 \times m'_2 / m'_1$$

10 Expression of results

Report the paraffin wax content rounded to the nearest half percent; the two results are considered valid if they do not differ by more than 1 %.

11 Precision

11.1 Repeatability

The difference between two 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 1 only in one case in twenty.