SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 17482:2020

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Bitumen in bitumenska veziva - Določevanje kislinskega števila bitumna - Potenciometrijska metoda

Bitumen and bituminous binders - Determination of acid number of bitumen - Potentiometric method

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Säurezahl von Bitumen - Potentiometrisches Verfahren

Bitumes et liants bitumineux - Détermination de l'indice d'acide d'un bitume - Méthode potentiométrique

Ta slovenski standard je istoveten z: **FprCEN/TS 17482**

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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TECHNICAL SPECIFICATION

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ICS 75.140; 91.100.50

English Version

Bitumen and bituminous binders - Determination of acid number of bitumen - Potentiometric method

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Bitumen und bitumenhaltige Bindemittel - Bestimmung der Säurezahl von Bitumen - Potentiometrisches Verfahren

This draft Technical Specification is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 336.

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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 (FprCEN/TS 17482:2020) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

This document is currently submitted to the Vote on TS.

1 Scope

This document describes a method for the determination of the free acidic constituents present in bitumen, conventionally known as acid number.

WARNING — The use of this document may involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and to 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*

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
acid number
number of milligrams of potassium hydroxide necessary for the neutralization of the free acids contained in one gram of bitumen

3.2
equivalence point
stage of the titration procedure at which the added volume (equivalent volume) of titration reagent has allowed to neutralize the acid compounds of the test sample

Note 1 to entry: In potentiometric titrations, this point corresponds to the inflexion point of the potential curve.

4 Principle

The bitumen is dissolved in a solvent-alcohol mixture. The acidic constituents are titrated using a solution of alcoholic potassium hydroxide. The titration is monitored by potentiometry.

5 Products and reagents

5.1 General

Only use reagents of a quality recognized for analysis.

5.2 Potassium hydroxide RP (analytical grade), in pellets.

5.3 Propan-2-ol (isopropanol) **RP** (analytical grade).

5.4 Solvent RP (analytical grade), xylene or methyl benzene (toluene).

5.5 Benzoic acid RP (analytical grade).

5.6 Solution of alcoholic potassium hydroxide, 0,1 mol/L.

Dissolve (6 ± 0,2) g of potassium hydroxide pellets (5.2) in 1 L of pure propan-2-ol (5.3).

A ready-for-use, off-the-shelf solution can also be used. Only a titration certificate coming from an authorized supplier can avoid the titration procedure (9.1). The alcoholic potassium hydroxide solution shall be kept in brown glass containers and stored in the dark.

6 Apparatus

6.1 General

Usual laboratory equipment, together with the following.

6.2 Balance of adequate capacity for weight with precision of ± 1 mg.

6.3 Potentiometry apparatus, for automatic or manual titration:

— apparatus for automatic titration, including a titration device with sensitivity of at least 2 mV, equipped with an automatic dosage unit with a capacity of 5 mL to 20 mL. Most of these pieces of equipment offer a program for potentiometric detection with dynamic equivalence point titration. It means that increment is slowed down near the equivalence point;

— apparatus for manual titration, including a potentiometer with a sensitivity of at least 2 mV and a burette with a pointed spout.

The equipment shall allow titration steps to be performed at an accuracy of at least ± 0,05 mL in volume.

6.4 Glass combination electrode – Ag/AgCl.

6.4.1 A pH glass electrode for non-aqueous media and a reference electrode of Ag/AgCl (kept in its inner reference electrolyte).

6.4.2 Combined pH electrode for non-aqueous media instead of the two electrodes cell of 6.4.1.

6.5 Glass beakers, or any glass titration container, specific for the automatic potentiometric titration device (6.3).

The volume of beakers shall be appropriate for immersing the membrane of electrodes into the tested solution and shall allow increasing this volume during titration.

The level of solution in the beaker shall be below the filling hole of the electrodes but has to be high enough to prevent the stirring bar (if it is necessary to use it) from hitting the membranes if the electrodes have to be close to the bottom of the beaker.

6.6 Magnetic stirrer, with polytetrafluoroethylene (PTFE)-coated bar.

7 Sampling

Take the sample in accordance with EN 58. Prepare the test samples in accordance with EN 12594.

8 General instructions

8.1 Preparation of the potentiometry apparatus and electrodes

The titration device shall be prepared following manufacturer instructions.

Special attention shall be paid to the storing and maintenance of the electrodes:

— before using, the inner body shall be filled with the electrolyte. If necessary, refill and let the electrode to rest for at least one hour immersed in the reference electrolyte;

— before each titration, let the electrode to rest for at least 5 min immersed in the reference electrolyte;

— when the test is finished, the electrodes shall be cleaned with the solvent (5.4), then rinsed with water, and finally stored according to the recommendations of the supplier.

8.2 Titration of the solution of alcoholic potassium hydroxide and blank test

The solution of alcoholic potassium hydroxide used is to be titrated in accordance with the procedure described in 9.1 before starting the titration of bitumen. This titration can only be avoided when using a certified ready to use solution (5.6). In any case, it is recommended to titrate the solutions when they are used after storage and to verify this titration on a weekly basis.

The blank test (9.1) is to be done on a daily basis on the same solvent and propan-2-ol samples as those used for the titration of bitumen.

8.3 Operating procedure

This document concerns the preferential use of an apparatus for automatic titration. In the case of use of an apparatus for manual titration, the method described in Annex A can be referred to, as a replacement to 9.1.4 to 9.1.6 and 9.2.5 to 9.2.7 in this document.

The titration procedure described in 9.1.6, 9.2.7 and Annex A considers the usual case where the potential curve presents a single inflexion point (equivalence point). The possible presence of several acid compounds with different strengths could however lead to the existence of more than one equivalence point. To be sure that total neutralization has been achieved, titration shall therefore be continued until the evolution of the measured potential with the added volume of titrant becomes nil or presents a fairly constant slope. If several equivalence points are detected, this needs to be confirmed by a second test and the corresponding acid numbers shall be calculated as per Clause 10 and reported in the test report.

9 Operating procedure

9.1 Titration of the solution of alcoholic potassium hydroxide and blank test

9.1.1 Using the balance (6.2), weigh approximately 122 mg of benzoic acid (5.5) in a beaker (6.5). Write down the weight, m_1, to within 1 mg. If the dosage unit is of low capacity (typically less than 10 mL), the amount of benzoic acid may be reduced provided however that this quantity is known with a precision of at least ± 1 %.

9.1.2 Add into the beaker (50 ± 1) mL of solvent (5.4) and (50 ± 1) mL of propan-2-ol (5.3). Depending on the titration containers used or to ensure proper immersion of the electrodes, it may be necessary to adjust the total amount of solvent and propan-2-ol. This shall be done while maintaining the 1/1 ratio of solvent to propan-2-ol.

9.1.3 Place the bar of the magnetic stirrer in the beaker (6.5), then place the beaker on the stirrer (6.6) and switch the stirrer on, to achieve complete dissolution of the benzoic acid.

9.1.4 Connect the electrode (6.4) to the potentiometric titration device (6.3). Immerse the electrode into the beaker.

9.1.5 Connect the automatic dosage unit of the potentiometric titration device to the container holding the solution of alcoholic potassium hydroxide (5.6).

9.1.6 Perform the titration, while stirring, as per the operating procedure given by the manufacturer of the potentiometric titration device (the rate at which the alcoholic potassium hydroxide is added into the beaker shall not exceed 0,1 mL/min near the equivalence point). The equivalence point corresponds to the volume of alcoholic potassium hydroxide leading to the highest potential step.

The equivalent volume of alcoholic potassium hydroxide, as determined by calculation by the titration device, is written as v_1 to two decimal places.

9.1.7 Carry out a blank test by performing every one of steps 9.1.2 to 9.1.6. For step 9.1.6, the equivalent volume of alcoholic potassium hydroxide corresponding to the blank test is written as v_0, to two decimal places.

When performing the blank test, the equivalent volume is likely to be very small. It may then be preferable to switch from dynamic to monotonic mode (equal increments) of titrant addition, so as to avoid missing the equivalence point.

9.1.8 Calculate the titre (N) of the alcoholic potassium hydroxide in mol/L, according to the following Formula (1), and express it to three decimal places:

$$N = \frac{m_1}{122,12 \times (v_1 - v_0)} \quad (1)$$

where

m_1 is the mass of benzoic acid, expressed in mg;

v_1 is the equivalent volume of alcoholic potassium hydroxide, poured for the test, expressed in mL;

v_0 is the equivalent volume of alcoholic potassium hydroxide, poured for the blank test, expressed in mL.

9.2 Titration of the bitumen

9.2.1 Using a spatula, homogenize the still warm sample of bitumen, prepared beforehand as per standard EN 12594. Pour (5 ± 1) g of the bitumen sample into a beaker (6.5). Write down, m_2, to within 0,01 g.

9.2.2 Add (50 ± 1) mL of solvent (5.4) and the bar of the magnetic stirrer (6.6) into the beaker (6.5).

9.2.3 Place the beaker, covered by a watch glass, on the stirrer, and switch the stirrer on. Continue stirring until the bitumen has fully dissolved. The process can be accelerated by gently heating the solution and by using an ultrasonic device.

9.2.4 After the bitumen has fully dissolved, add (50 ± 1) mL of propan-2-ol (5.3) into the beaker. Keep stirring for 5 min minimum.

Depending on the titration containers used or to ensure proper immersion of the electrodes, it may be necessary to adjust the total amount of solvent, propan-2-ol and bitumen. This shall be done while maintaining the 1/1 ratio of solvent to propan-2-ol. The amount of bitumen shall be adjusted while maintaining a minimum ratio of 4 g of bitumen for 50 mL of solvent (e.g. 8 g of bitumen in 100 mL of