SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 17481:2020

01-april-2020

Bitumen in bitumenska veziva - Ugotavljanje deleža soli v bitumnu - Metoda električne prevodnosti

Bitumen and bituminous binders - Determination of salt content in bitumen - Electrical conductivity method

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Salzgehaltes in Bitumen - Verfahren durch Messung der elektrischen Leitfähigkeit

Bitumes et liants bitumineux - Détermination de la teneur en sel des bitumes - Méthode par mesure de conductivité

Ta slovenski standard je istoveten z: FprCEN/TS 17481

<u>ICS:</u>

75.140	Voski, bitumni in drugi naftni proizvodi	Waxes, bituminous materials and other petroleum products
91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

kSIST-TS FprCEN/TS 17481:2020 en,fr,de

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

FINAL DRAFT
FprCEN/TS 17481

February 2020

ICS 75.140; 91.100.50

English Version

Bitumen and bituminous binders - Determination of salt content in bitumen - Electrical conductivity method

Bitumes et liants bitumineux - Détermination de la teneur en sel des bitumes - Méthode par mesure de conductivité

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Salzgehaltes in Bitumen - Verfahren durch Messung der elektrischen Leitfähigkeit

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

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Ref. No. FprCEN/TS 17481:2020 E

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European foreword

This document (FprCEN/TS 17481:2020) 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.

Introduction

The proposed test method is an adaptation to bitumen of the ASTM D3230-13 applicable to crude oil.

In its principle, the method first extracts the salts contained in the bitumen sample by bringing the latter (previously dissolved in xylene) in contact with a hydro-alcoholic mixture. It is the water contained in this mixture which will dissolve those salts and give the mixture a certain electrical conductivity. The measuring of the intensity of the electrical current obtained when applying a given voltage allows then to derive an "equivalent NaCl content" of the bitumen solution by referring to a calibration curve plotted using standard solutions of which the NaCl content is known.

A few simplifications have been introduced in comparison to ASTM D3230-13 [1]. Those consist mainly in the replacement of the methanol of the hydro-alcoholic solution by denatured ethanol (less health hazardous) and in the replacement of the mixed salts reference solution (mixture of sodium, calcium and magnesium chlorides representative of sea water) by a single NaCl solution.

The critical points of the method can be listed as follows:

— to avoid the fouling of the electrodes, the amount of bitumen dissolved in the solution on which the measurement is performed is relatively small (3 g in 100 mL);

— since the amount of salts extracted from the bitumen sample may be quite small, it is important to limit as much as possible the incidence of the intrinsic conductivity of the water used for their extraction on the conductivity of the solution. This explains:

  — the small quantity of water in the composition of the hydro-alcoholic mixture;

  — the requirements relative to the purity of the used water (presence of conductive impurities);

  — the need to not keep the hydro-alcoholic mixture for more than one day after its manufacture. This is due to its hygroscopic nature (all the more so because the methanol prescribed in ASTM D3230-13 has been replaced by ethanol, which is more hygroscopic);

  — the need to systematically make a new « blank » measure each time a new hydro-alcoholic mixture is made;

— the need to generate and detect low intensity currents under good conditions makes it necessary to apply a relative high voltage and explains also the geometry which has been retained for the electrodes (important rectangular surface area and spacing). Measurements performed with different electrode geometries should therefore not be compared.

The proposed extraction method (which essentially takes over the procedure specified in ASTM D3230-13) may however be questioned when applied to bitumen.

— Under the retained operating conditions (small amount of water, only shaking the solution for one minute), the « transfer » to the water in the hydro-alcoholic mixture of the salts present in the product should be easier in the case of crude oil than for bitumen. It is very likely that only part of the salt present in the bitumen is transferred to the water and that the importance and the kinetics of this transfer depend on the nature of the bitumen. The efficiency and the reproducibility of the method may probably be enhanced through an optimization of the exchange conditions between the bitumen solution and the hydro-alcoholic phase.

In the frame of the 2nd Round Robin test of CEN/TC 336 on salt content [3], an increased amount of water in the hydro-alcoholic mixture (10 g instead of 3 g) and an extended contact time between the hydro-alcoholic mixture and the bitumen solution have been tested. Both lead to increased measured salt contents in comparison to the procedure described in this standard and which had been tested in the first

Round Robin [2]. Due to the low number of participating laboratories, the impact on repeatability and reproducibility could however not be established.

— Also the reason for the use of two different alcohols and for their respective proportions is not known and should be cleared up.

1 Scope

This document describes a method for determination of the salt content in bitumen, conventionally expressed in mg of sodium chloride (noted NaCl as from this point of this document) per kg of bitumen. This method is valid for "equivalent NaCl contents" between 20 mg/kg and 500 mg/kg.

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*

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

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
salt content in the bitumen
conventionally, sodium chloride, calcium chloride and magnesium chloride dissolved in the bitumen, other inorganic chlorides may also be present

Note 1 to entry: Other inorganic chlorides may also be present.

4 Principle

This test method consists in measuring the conductivity of a bitumen solution in a mixture of solvents in which are immersed two parallel stainless-steel plates constituting electrodes subjected to an alternating current. The intensity of the resulting current is measured on a milliammeter. An "equivalent NaCl content" of the bitumen solution is deduced by referring to a calibration curve plotted using standard solutions whose NaCl content is known.

5 Products and reagents

5.1 General

Only use reagents of a quality recognized for analysis.

5.2 Xylene RP (analytical grade).

5.3 Denatured anhydrous ethanol containing at least 95 % of ethanol.

5.4 Anhydrous n-butanol.

5.5 Demineralized water, conforming to a quality 3 or better as per ISO 3696.

5.6 Hydro-alcoholic mixture (3 g of water in a mixture of ethanol and n-butanol) prepared as described in (8.2).

5.7 Sodium chloride RP (analytical grade).

6 Apparatus

6.1 General

Usual laboratory apparatus and glassware, together with the following.

6.2 Conductivity meter equipped with:

— 2 stainless steel electrodes in the shape of rectangular plates (50 ± 1) mm long, (25 ± 1) mm wide, (1,5 ± 0,5) mm thick; the gap between the electrodes is (6,5 ± 0,5) mm; parallelism is achieved using washers in a non-conductive material; these electrodes are kept immersed in clean xylene (5.2) in a storage beaker (6.3);

— 1 adjustable potentiometer used to set the voltage on the electrode terminals at a constant value between 25 V and 250 V, e.g. **125 V**;

— 1 milliammeter allowing the measurement of electrical current intensity in the range going from 0 to 10 mA.

Some pieces of equipment are designed for directly displaying conductivity values (for example in micro-siemens per cm - µS/cm) or salt content values in mg/kg. If this is the case, the instructions given further down in this standard for displays in mA are to be applied to the displays given by the equipment used.

The equipment shall allow readings to be done to within 0,01 mA (or to within 0,1 µS/cm or to within 0,1 µS).

To avoid hydrolysis reactions in the measured solution, the electrical current generated between the electrodes should have a frequency of at least 500 Hz. This is normally the case for usual conductivity meters but it is recommended to check that this is indeed the case.

6.3 100 mL beakers, of a high shape, or any container specific to the conductivity meter, to cover the top of the electrodes by more than 5 mm for a solution volume of 100 mL.

6.4 Class A 10 mL two-line pipette, conforming to ISO 648.

6.5 Class A 25 mL burette, conforming to ISO 385.

6.6 Class A 100 mL graduated test tubes with ground stopper, conforming to ISO 4788.