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Bitumen and bituminous binders — Determination of storage stability of modified bitumen

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May 2010

ICS 75.140; 91.100.50

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English Version

Bitumen and bituminous binders - Determination of storage stability of modified bitumen

Bitumes et liants bitumineux - Détermination de la stabilité au stockage des bitumes modifiés

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Lagerbeständigkeit von modifiziertem Bitumen

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Foreword

This document (EN 13399:2010) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR/BNPé.

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of an identical text or by endorsement, at the latest by November 2010, and conflicting national standards shall be withdrawn at the latest by November 2010.

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1 Scope

This European Standard specifies a method for measuring the storage stability at high temperatures.

NOTE Modified bitumen and, in particular, polymer-modified bitumen, which consist of mainly bitumen and at least one additional agent, are known to display phase separation under certain conditions.

WARNING — The 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 referenced documents are indispensable for the application 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 Principle

A homogeneous sample of modified bitumen is maintained in a vertical vessel at 180 °C, or at a temperature specified by the producer, for 3 days. After the sample has cooled down, it is cut into three equal parts. The two ends (top and bottom) are analysed further to evaluate possible differences in characteristics.

If the chosen temperature differs from 180 °C, it shall be mentioned in the test report.

4 Apparatus

Usual laboratory apparatus and glassware, together with the following:

4.1 Tube, of thin unvarnished aluminium, of height 160 mm minimum and of diameter between 25 and 40 mm, closed at one end and typically "toothpaste tube".

4.2 Oven, maintained at a temperature of (180 ± 5) °C for three consecutive days.

4.3 Tins, one to be marked "top" and one to be marked "bottom" (see 5.5), each of sufficient capacity to contain the combined upper and lower parts of different tubes.

5 Procedure

5.1 General

Ensure that the laboratory sample is representative of the modified bitumen to be analysed, in accordance with EN 58. Ensure that the laboratory sample is homogeneous and non-contaminated.

Remove at least 150 g of the laboratory sample in accordance to EN 12594 and place it in a suitable container.

5.2 Filling of the tube

Heat the sample according to EN 12594 and re-homogenize using a stirrer.

Pour the homogeneous liquid sample into the tube to a height of 100 mm to 120 mm, taking care to avoid incorporation of air bubbles.

5.3 Closing of the tube

Ensure that the tube is tightly closed to avoid any action of air during high-temperature storage.

A possibility for thermal expansion of the test material is necessary, and is created by squeezing the tube jacket slightly flat. After pouring the test material into the tube, and before the test material has completely cooled, press the open end of the tube together at a short distance below the test material surface. Ensure that no air remains by squeezing part of the test material from the tube. Press the end of the tube flat and fold the tube tightly several times.

5.4 Test conditions

Within 1 h after filling, place the tube in the preheated oven in a vertical position.

Ensure that the tube is maintained in a vertical position with an accuracy of ± 5 °C at the chosen temperature for (72 ± 1) h.

5.5 Recovery of the sample

Remove the tube from the oven and let it cool down to room temperature, still in the same vertical position.

Peel the aluminium tube to recover the modified bitumen sample.

NOTE 1 For this operation it is recommended that the tube is placed in a refrigerating chamber, e.g. for 30 min at - 20 °C, and that the aluminium foil is then quickly removed.

Place the recovered cylindrical sample horizontally on a clean flat surface and cut it into three equal parts with a heated knife.

Place the top and bottom parts separately in the respective tins marked "top" and "bottom". These parts are now ready for further testing.

The middle part is of no use and should be eliminated.

NOTE 2 For characterisation tests (e.g. softening point EN 1427 [1]; needle penetration, EN 1426 [2] and others) it can be necessary to use more than one tube. In such cases, two or more tubes of the same sample should be tested simultaneously.

6 Test report

The test report shall contain at least the following information:

- a) type and identification of the sample under test;
- b) reference to this European Standard;
- c) measured values for the top and the bottom part according to the utilised European standards;
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) date of the test.

Bibliography

- [1] EN 1427, *Bitumen and bituminous binders — Determination of softening point — Ring and Ball method*
- [2] EN 1426, *Bitumen and bituminous binders — Determination of needle penetration*

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