



SLOVENSKI STANDARD
kSIST FprEN 13632:2010

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Bitumen in bitumenska veziva - Vizualni pregled razporeditve polimerov v bitumnih, modificiranih s polimeri

Bitumen and bituminous binders - Visualisation of polymer dispersion in polymer modified bitumen

Bitumen und bitumenhaltige Bindemittel - Visualisierung der Polymerverteilung in polymermodifiziertem Bitumen

Bitumes et liants bitumineux - Visualisation de la dispersion des polymères dans les bitumes modifiés par des polymères

Ta slovenski standard je istoveten z: FprEN 13632

ICS:

75.140	Voski, bitumni in drugi naftni proizvodi	Waxes, bituminous materials and other petroleum products
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Bitumen and bituminous binders - Visualisation of polymer dispersion in polymer modified bitumen

Bitumes et liants bitumineux - Visualisation de la dispersion des polymères dans les bitumes modifiés par des polymères

Bitumen und bitumenhaltige Bindemittel - Visualisierung der Polymerverteilung in polymermodifiziertem Bitumen

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Foreword

This document (FprEN 13632:2009) 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 Unique Acceptance Procedure.

This document will supersede EN 13632:2003.

Annex A is informative.

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1 Scope

This document specifies a method for visualisation of the polymer distribution in polymer modified bitumen by fluorescent microscopy.

The method is applicable for most of the commercially used polymers, but before the method is used it should be examined whether the test is applicable for the actual polymer.

The method should only be used for identification purposes, i.e. in connection with production control.

NOTE Sample preparation and treatment have an important influence on the test results and it is essential to follow strictly the method described to achieve comparable results.

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 sample of polymer modified bitumen is homogenised by gentle stirring and poured into a preheated mould. After a controlled cooling procedure to ambient temperature the sample is cooled below -20 °C for a minimum period of time. The bitumen layer is broken into small pieces and the freshly broken surface is viewed through a microscope with a magnification of 25 times to 500 times. The views can be stored photographically or electronically.

4 Apparatus

4.1 Epifluorescence microscope, (incident light excitation) with an appropriate light source and filter systems.

NOTE Examples of light source and filter system are given in Annex A.

4.2 Freezer or solid carbon dioxide

4.3 Sharp tool

Scissors shall not be used.

4.4 Aluminium basin, disposable, approximate height 35 mm, approximate diameter 70 mm

4.5 Porcelain evaporating basin, diameter 150 mm, height 63 mm (approximate volume: 600 ml)

4.6 Fine aggregate (sand)

4.7 Temperature regulated sand bath

5 Preparation of test samples

Take the sample in accordance with EN 58. Prepare it in accordance with EN 12594. Homogenise the sample by gentle hand stirring for at least 1 min and not more than 5 min. Pour the material into the aluminium basin placed in a sand bath (4.7) preheated to the same temperature as the sample during homogenising. The sand bath is an evaporating basin (4.5) containing sand (4.6). The aluminium basin (4.4) shall be totally surrounded by the sand and there shall be 20 mm of sand between the bottom of aluminium basin and the evaporating basin.

The sample is cooled to ambient temperature by switching off the heating of the sand bath. After cooling to ambient temperature, cool the sample to $-20\text{ }^{\circ}\text{C}$ or lower if the sample needs to become brittle. Using a freezer (4.2), a cooling time of 3 h minimum is required; using solid carbon dioxide (4.2) in a dewar, 10 min is sufficient.

NOTE The cooling procedure can be essential for the morphology. By cooling in the sand bath, a steady cooling rate is assured.

6 Procedure

Prepare small pieces of the frozen material by breaking or cutting with appropriate sharp tool (4.3). This preparation shall be done rapidly to avoid warming the sample. Inspect the freshly broken or cut surface within 1 h. The inspection can be done through a protecting glass or directly on the surface. Using a protecting glass turn the freshly broken surface towards a microscope cover glass and arrange for the freshly broken or cut surfaces to stick to this without disturbing the surface by moving. Turn the cover glass over and place it on the hole of the rigid support such that a perfectly horizontal and flat surface is observed through the glass.

Select the magnification according to the particle size and distribution; magnifications of 25 to 500 are suitable. Analyse each polymer modified bitumen on a minimum of 3 independently prepared surfaces scanning the entire surface before collecting the typical picture.

Provide a reference scale with the picture.

NOTE The picture can be obtained and stored by photographic or electronic systems.

7 Expression of results

Express the binder morphology either as a picture, a picture number (according to Annex A) or a combination of the characterisation letters shown in Annex A. Intermediate numbers are not allowed. If no fluorescent emission can be detected and the sample appears all black, note this as O.

NOTE Numbers are used as a simple tool for comparing pictures and neither assessing the quality, nor the performance of the binder.

8 Test report

The test report shall contain at least the following information:

- a) type and complete identification of the sample under test;
- b) reference to this European Standard;
- c) light source and filter system;

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- d) present the picture with a reference scale and if required a combination of the characterisation letters given in Annex A (see Clause 7);
- e) any deviation, by agreement or otherwise, from the procedure specified;
- f) date of the test.