SLOVENSKI STANDARD
SIST EN 1431:2009

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Bitumen in bitumenska veziva - Dolo ļ evanje veziva in oljnega destilata v bitumenskih emulzijah z metodo destilacije

Bitumen and bituminous binders - Determination of recovered binder and oil distillate from bitumen emulsions by distillation

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Destillationsr¿ckstandes und des ŕ ldestillates von Bitumenemulsionen mittels Destillation

Bitumes et liants bitumineux - Détermination par distillation du liant résiduel et du distillat d'huile dans les émulsions de bitume

Ta slovenski standard je istoveten z: **EN 1431:2009**

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

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Foreword

This document (EN 1431:2009) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by September 2009, and conflicting national standards shall be withdrawn at the latest by September 2009.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 1431:1999.

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1 Scope

This European Standard specifies a method for the quantitative determination of residual binder and oil distillate in bituminous emulsions.

The method can also be used to obtain residue and oil distillate for further testing.

NOTE The properties of the material recovered in the test are not necessarily the same as those of the original materials from which the emulsion was produced, especially for polymer modified bitumens.

WARNING — The use of this standard may 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

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 573-3, *Aluminium and aluminium alloys - Chemical composition and form of wrought products - Part 3: Chemical composition and form of products*

EN 1425*, Bitumen and bituminous binders – Characterization of perceptible properties*

EN 12594, *Bitumen and bituminous binders - Preparation of test samples*

ISO 565, *Test sieves - Metal wire cloth, perforated plate and electroformed sheet - Nominal sizes of openings*

3 Terms and definitions

For the purposes of this document, the following definitions apply.

3.1
oil distillate
hydrocarbon fraction which is distilled and collected in the graduated cylinder under conditions specified in this test

3.2
residual binder
residue from a bituminous emulsion after distillation of water and oil distillate

NOTE Although mentioned as "recovered binder content" in the EN 13808 specification standard, the residual binder by distillation is being considered in that case.
The EN 13808 specification standard specifies the "recovered binder content" according to EN 1431, although the appropriate denomination is "residual binder after distillation". Consequently, it is highly recommended that this correction should be considered when revising the EN 13808 standard.

4 Principle

Water and oil distillate are distilled from the bituminous emulsion, and separated in a graduated cylinder, leaving a residue of residual binder.

5 Reagents and materials

5.1 Cleaning agents, as used conventionally in a laboratory.

5.2 Sodium hydroxide solution, 40 g/l.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

NOTE For details of assembly of the distillation apparatus for the test, see Figure 3.

6.1 Aluminium alloy still (see Figure 1) or **iron still,** (241± 5) mm in height by (101 ± 1) mm external diameter with a (3,2 ± 0,2) mm thickness wall and a (125 ± 5) mm inside diameter ring burner, having holes on the inner circumference and having three appropriate spacers or guide pins, to ensure centring of burner around the still (see Figure 2).

Lid (see Figure 1) of suitable dimensions to allow two thermometers (6.4) to be inserted through a stopper and an outlet of suitable diameter to enable connection tube to be connected also through a stopper.

NOTE All tolerances in sub-clause 6.1 and shown on Figure 1 and Figure 2 are production tolerances for the manufactures of the test equipment. No calibration/verification concerning these tolerances is valid in Quality Control – apart from the thermometer.

The ring burner may be replaced by any alternative means of heating provided the same specified temperature and distillation conditions are achieved. In the event of a dispute, the reference ring burner as heating apparatus shall be used.

6.2 Connection apparatus, consisting of a glass connecting tube with a (12 ± 1) mm diameter, tin shield, and standard water cooled glass condenser tube with a metal or borosilicate glass jacket (see Figure 3).

6.3 Graduated cylinder, 100 ml, with graduation intervals of 0,5 ml, to comply with ISO 4788.

6.4 Thermometers, two low distillation thermometers, graduated in degrees Celsius, having a range from -2 °C to 300 °C. Thermometers conforming to the necessary requirements are specified in Annex A.

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, taking into account changed thermal response times compared with the mercury thermometer.

For this test method, in which increasing temperatures are read during the test, documented corrections shall be determined in advance and applied to the observed readings.

6.5 Balance, capable of weighing 3 500 g to an accuracy of 0,1 g.

6.6 Bunsen burner or equivalent means of heating.

6.7 Sieve, 300 µm, complying with R 40/3 of ISO 565.

7 Sampling

The laboratory sample shall be sampled in accordance with EN 58, and the test samples shall be prepared in accordance with EN 12594. Ensure that the laboratory sample is homogeneous and is not contaminated (see EN 1425).

8 Procedure

8.1 Weigh (200,0 ± 0,1) g, A_m , of the emulsion sample into the still (6.1) which has been previously tared (including lid, clamp, thermometers and gasket, if gasket is used).

8.2 Use a gasket of oiled paper between the still and its cover, or grind the joint to a tight fit. Securely clamp the cover on the still.

8.3 Insert a thermometer (6.4) through a cork, in each of the two small holes provided in the cover. Adjust the thermometers so that the end of the bulb of one is (6,5 ± 1,0) mm from the bottom of the still and the bulb of the other is (165 ± 2) mm from the bottom of the still.

8.4 Place the ring burner (6.1) at (152 ± 2) mm from the bottom of the still. Apply heat by lighting this burner and adjusting it to a low flame. Also apply sufficient heat from a Bunsen burner (6.6) to the connecting tube to prevent condensation of water in this tube.

NOTE The location of the flame of the ring burner at the start of the test is flexible. It can be raised to decrease the risk of foam-over or lowered to the middle of the still for emulsion containing no solvent. A sudden change in temperature reading of the upper thermometer indicates foam on the bulb and heating should be discontinued until foaming ceases.

8.5 Adjust the location of the ring burner from time to time so that a smooth constant distillation occurs throughout the whole procedure. When the reading on the lower thermometer reaches 215 °C, lower the ring burner until the reading on the thermometer is (260 ± 5) °C. Maintain the temperature at this level for 15 min.

Complete the total distillation in (60 ± 15) min from the first application of heat.

If the residual binder as mass percentage is not to be determined (see Clause 9), i.e. distillation has been performed only to get residual binder for further testing, cooling and weighing (8.6 and 8.7) are not necessary. Proceed directly to 8.8.

8.6 At the end of the heating period, allow the still and accessories to cool down before weighing. Weigh the still and accessories and determine the mass of residue after distillation, B_m. Reheat the still containing the sample up to (260 ± 5) °C for further processing of the residue (see 8.8)

8.7 Record the volume, *D*, of oil distillate in the graduated cylinder (6.3) to the nearest 0,5 ml.

NOTE To improve the separation of water and oil, 5 ml of sodium hydroxide solution (5.2) may be added to distillates from cationic emulsions.

8.8 Remove the cover from the still. Stir the residue, and pour it immediately through a 300 µm sieve (6.7) heated at the same temperature prior to use. Transfer sufficient quantities of the residue into suitable moulds and containers for carrying out any required further tests. Handle or condition moulds and containers for examination of the residue as described in EN 12594 and proceed as required by the appropriate EN test method from the steps that follow the pouring stage.

Retain the oil distillate for identification if required.

9 Calculation

Calculate the residual binder after distillation, r, as mass percentage, using the following equation:

$$r = \frac{B_m}{A_m} \text{ x } 100 \qquad (1)$$

where

A_m is the mass of emulsion sample, in grams (see 8.1);

B_m is the mass of the residue after distillation, in grams (see 8.6).

Calculate the oil distillate (o), **as a volume percentage**, using the following equation:

$$o = \frac{D \cdot \rho}{A_m} \times 100 \qquad (2)$$

where

D is the volume of oil distillate, in millilitres (see 8.7).

ρ is the density of the emulsion, in g/ml.

NOTE The density of the emulsion, ρ ,is assumed to be 1 000 kg/m³ (1 g/ml) at 15 °C.

10 Expression of results

Express the residual binder (see Note in 3.2) as a mass percentage to the nearest 1 %. Express the oil distillate as a volume percentage to the nearest 0,1 %.

The binder content of the emulsion determined by the distillation method described in this method shall be defined as the mass percentage of the residual binder plus the mass percentage of oil distillate.

NOTE Based on the volume percentage of oil distillate, the mass percentage of oil distillate m, may be determined on bulk quantities of flux following the determination of density, in accordance with EN ISO 3838. If the density cannot be determined in this way, a value of 0,850 may be assumed.