



SLOVENSKI STANDARD
oSIST prEN 1431:2016
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Bitumen in bitumenska veziva - Določevanje veziva in oljnega destilata v bitumenskih emulzijah z metodo destilacije

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

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Destillationsrückstandes und des Öldestillates von Bitumenemulsionen mittels Destillation

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

<https://standards.iteh.ai/catalog/standards/sist/86b6a803-b1ad-4996-a622-bf2b787d0937/sist-en-1431-2018>

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

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

Bitumen und bitumenhaltige Bindemittel - Destillationsrückstands und des Oldestillates von Bitumenemulsionen mittels Destillation

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

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents	Page
European foreword.....	3
1 Scope	4
2 Normative references	4
3 Terms and definitions	4
4 Principle	4
5 Reagents and materials.....	5
6 Apparatus.....	5
7 Sampling.....	6
8 Procedure.....	6
9 Calculation	7
10 Expression of results.....	7
11 Precision.....	8
11.1 Repeatability.....	8
11.2 Reproducibility.....	8
12 Test report.....	8
Annex A (informative) Characteristics of thermometer.....	12
Bibliography.....	13

European foreword

This document (prEN 1431:2016) 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 CEN Enquiry.

This document will supersede EN 1431:2009.

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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, cut-back and fluxed bituminous binders.

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 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 metal plate and electroformed sheet — Nominal sizes of openings*

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EN ISO 4788, *Laboratory glassware - Graduated measuring cylinders (ISO 4788)*

3 Terms and definitions

For the purposes of this document, the following terms and 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 by distillation
residue from a bituminous emulsion after distillation of water and oil distillate

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, or other suitable basic solution.

6 Apparatus

6.1 Aluminium alloy still (see Figure 1) or iron still, (241 ± 5) mm in height by (101 ± 1) mm external diameter with a $(3,2 \pm 0,2)$ 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 temperature measuring devices (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 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.

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, metallic shield, and standard water cooled glass condenser tube with a metal or borosilicate glass jacket (see Figure 3).

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

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

6.4 Two temperature measuring devices (combining sensor and reading unit) which shall:

- have a range from at least 10 °C to 300 °C
- be readable to 1 °C or less and
- have an accuracy of 1 °C or better.

Solid stem mercury thermometer (which used to be the former reference thermometer as described in Annex A) are also allowed if national regulations permit their use.

The temperature measuring devices shall be calibrated regularly so as to provide the same readings as would be provided by the mercury stem thermometers, taking into account changed thermal response times compared with the mercury thermometers.

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 3500 g to an accuracy of 0,1 g.

6.6 Bunsen burner or equivalent means of heating.

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

prEN 1431:2016 (E)

6.8 Usual laboratory apparatus and glassware;

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 $(200,0 \pm 0,1)$ g, A_m , of the emulsion sample into the still (6.1) which has been previously tared (including lid, clamp, temperature measuring devices 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 temperature measuring device (6.4) through a cork or any other suitable material, in each of the two small holes provided in the cover. Adjust the temperature measuring device so that the measuring end of one is $(6,5 \pm 1,0)$ mm from the bottom of the still and the measuring end 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.

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 temperature measuring device reaches 215 °C, lower the ring burner until the reading on the temperature measuring device 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.

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 .

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

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

Retain the oil distillate for identification if required.

8.8 For further processing of the residual binder, reheat the still according to the prescription of EN 12594. Remove the cover from the still. Stir the residue, and pour it immediately through a $300 \mu\text{m}$ 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.

NOTE A too low reheating temperature creates difficulties in pouring an adequately homogeneous test sample

9 Calculation

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

$$r = \frac{B_m}{A_m} \times 100 \quad (1)$$

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 **as a volume percentage** (o_v), using the following formula:

$$o_v = \frac{D \cdot \rho_e}{A_m} \times 100 \quad (2)$$

where

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

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

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

Calculate the oil distillate **as a mass percentage** (o_m), using the following formula:

$$o_m = o_v \cdot \rho_o \quad (3)$$

where

ρ_o is the density of the distillate, in g/ml.

The density of the oil distillate may be determined in accordance with EN ISO 3838 or another suitable method. If the density cannot be determined in this way, a value of 0,850 may be assumed or, if known, the value given by the supplier of the oil distillate may be used. The way density has been determined is to be mentioned in the test report (see Clause 12).

Calculate the binder content of the emulsion as the mass percentage of the residual binder plus the mass percentage of oil distillate.

10 Expression of results

Express the residual binder as a mass percentage to the nearest 1 %. Express the oil distillate as a mass percentage to the nearest 0,1 %.

Express the binder content of the emulsion as a mass percentage to the nearest 1 %.

11 Precision

11.1 Repeatability

The difference between two successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operations of the test method, exceed the values given in Table 1 in only one case in 20.

11.2 Reproducibility

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would in the long run, in the normal and correct operations of the test method, exceed the values given in Table 1 in only one case in 20.

Table 1 — Precision

Residual binder mass %	Repeatability mass %	Reproducibility mass %
50 to 70	1,0	2,0

12 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) results of the test (see Clause 10);
 - mass % of residual binder
 - mass % of oil distillate
 - density of oil distillate and method used for its determination
 - binder content
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) date of the test.