



# SLOVENSKI STANDARD

## SIST EN 1431:2018

01-junij-2018

Nadomešča:  
SIST EN 1431:2009

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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

Ta slovenski standard je istoveten z: **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

**SIST EN 1431:2018** **en,fr,de**

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 1431**

March 2018

ICS 75.140; 91.100.50

Supersedes EN 1431:2009

English Version

## 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 bitumes

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

This European Standard was approved by CEN on 6 November 2017.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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

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## European foreword

This document (EN 1431:2018) 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 2018, and conflicting national standards shall be withdrawn at the latest by September 2018.

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

This document supersedes EN 1431:2009.

The main technical changes are:

- Clause 3.2: since EN 13808:2013 now clearly refers to residual binder after distillation, the NOTE is no longer necessary and has thus been discarded.
- Clause 5.2: alternative basic solutions to sodium hydroxide are allowed.
- Clause 6.3: electric ring heater, which may be coupled to a temperature sensor, is described as another possible way for applying heat. This equipment may only be used if the purpose of the test is to determine residual binder and oil distillate content, without the determination of residual binder properties.
- Clause 6.6: mercury stem thermometers are replaced by temperature measuring devices allowing similar temperature determinations to be made. Annex A (characteristics of mercury stem thermometer) becomes informative.
- Clause 8.5: when electrical heating is used, the total distillation process may be extended to 120 min.
- Clauses 8.6 and 8.8: at the end of the distillation procedure, the residue is to be cooled down whatever the next steps. If the residual binder is to be further used for the preparation of test samples, it is to be reheated in accordance to EN 12594 (and not to 260 °C as in EN 1431:2009).
- Clause 9 and 10: as for residual binder content, oil distillate content is to be expressed as a mass percentage. Alternative methods to EN ISO 3838 or values provided by the supplier may be used for the determination of oil distillate density.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## EN 1431:2018 (E)

### 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 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 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*

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

EN ISO 3838, *Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary-stoppered pycnometer and graduated bicapillary pycnometer methods (ISO 3838)*

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.

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

**3.2****residual binder after distillation**

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

Note 1 to entry: The amount of the residual binder after distillation (% by mass) is referred to as the “residual binder content” of the emulsion.

**3.3****binder content of the emulsion**

sum of residual binder content and oil distillate content

**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**

Usual laboratory apparatus and glassware, together with the following:

**6.1** Aluminium alloy still (see Figure 1) or iron still, (241 ± 5) mm in height by (100 ± 2) mm external diameter with a (3,2 ± 0,2) mm thickness wall.

Lid (see Figure 1) of suitable dimensions to allow two temperature measuring devices (6.6) to be inserted through a stopper and an outlet of suitable diameter to enable connection tube to be connected also through a stopper.

**6.2** Heating system – Equipment needed when using a gas burner

Ring gas burner with a (125 ± 5) mm inside diameter, 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).

**6.3** Heating system – Equipment needed when using electrical heating

If the purpose of the test procedure is only to determine the residual binder and oil distillate content, without the determination of residual binder properties, the ring gas burner may be replaced by an electric ring heater with a capacity between 600 W and 1 000 W and a width of (50 ± 15) mm which can be tight-fitted around the aluminium alloy still (see Figure 3). The power supplied to the heating ring shall be controllable and preferably adjustable through a temperature control unit taking as a reference the temperature indicated by the lower temperature measuring device (see 8.3).

NOTE 1 The use of an electric ring heater is limited to the determination of the residual binder and oil distillate content since so far there is no evidence that both methods lead to the same residual binder properties.

NOTE 2 All tolerances in 6.1, 6.2 and 6.3 and shown on Figure 1 and Figure 2 are production tolerances for the manufacturers of the test equipment. No calibration/verification concerning these tolerances is necessary in Quality Control.

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**6.4** Connection apparatus, consisting of a glass connecting tube with a  $(12 \pm 1)$  mm outer diameter, metallic shield, and standard water cooled glass condenser tube with a metal or borosilicate glass jacket (see Figure 4).

NOTE For details of assembly of the distillation apparatus for the test, see Figure 4.

**6.5** Graduated cylinder, 100 ml, with graduation intervals of 1 ml, to comply with EN ISO 4788.

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

- have a range from at least  $(10\text{ °C to }300\text{ °C})$ ,
- be readable to  $1\text{ °C}$  or less and
- have an accuracy of  $\pm 1\text{ °C}$  or better.

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

The temperature measuring devices shall be calibrated regularly.

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.

**6.7** Balance, capable of weighing 3 500 g to an accuracy of  $\pm 0,1\text{ g}$ .

**6.8** Bunsen burner or electrical fan heater to prevent condensation of water in the connecting tube if necessary (see 8.4 or 8.5), or equivalent means of heating.

**6.9** Sieve, 300  $\mu\text{m}$ , complying with R 40/3 of ISO 565. <https://standards.iteh.ai/catalog/standards/sist/86b6a803-b1ad-4996-a622-bf2b787d0937/sist-en-1431-2018>

## 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, 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). Record the mass as  $A_m$ .

**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 (6.6) through a cork or any other suitable material, in each of the two small holes provided in the cover. Adjust the temperature measuring devices 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  $(165 \pm 2)$  mm from the bottom of the still.

**8.4** Procedure when using a gas ring burner



Place the ring burner (6.2) at  $(152 \pm 2)$  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 or equivalent (6.8) 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.

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  $215\text{ }^{\circ}\text{C}$ , lower the ring burner until the reading on the temperature measuring device is  $(260 \pm 5)\text{ }^{\circ}\text{C}$ . Maintain the temperature at this level for 15 min.

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

### 8.5 Procedure when using electrical heating

Place the heating ring (6.3) at  $(152 \pm 2)$  mm from the bottom of the still and apply heat by progressively increasing the supplied electric power. Also apply sufficient heat from an electrical fan heater or equivalent (6.8) to the connecting tube to prevent condensation of water in this tube.

A sudden change in temperature reading of the upper thermometer indicates foam on the bulb and heating should be discontinued until foaming ceases. Adjust the heating power of the electrical ring burner so that a smooth constant distillation occurs throughout the whole procedure. When the reading on the lower temperature measuring device reaches  $215\text{ }^{\circ}\text{C}$ , increase the electrical power until the reading on the temperature measuring device is  $(260 \pm 5)\text{ }^{\circ}\text{C}$ . Maintain the temperature at this level for 15 min.

Complete the total distillation in  $(60 \pm 15)$  min from the first application of heat. If, for certain type of emulsions, this time cannot be met, it is allowed to continue the procedure up to 120 min.

**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 (6.5) 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  $300\text{ }\mu\text{m}$  sieve (6.9) heated to 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 the residue as described in EN 12594 and proceed as required by the appropriate EN test method from the step that follows the pouring stage.

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