
6]li a Yb`]b`V]li a Ybg_Uj Yn]j U!`8 c`c Yj Ub`Y`j Yn]j U]b`c``bY[UXYgh]`UHj
V]li a Ybg_] `Ya i `n]`U `n`a YfcXc`XYgh]`UWY

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

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Destillationsrückstands 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

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Ta slovenski standard je istoveten z: EN 1431:1999

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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en

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 1431

October 1999

ICS 91.100.50

English version

Bitumen and bituminous binders - Determination of recovered
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 - Bestimmung des
Destillationsrückstands und des Öldestillates von
Bitumenemulsionen mittels Destillation

This European Standard was approved by CEN on 5 September 1999.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

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 April 2000, and conflicting national standards shall be withdrawn at the latest by June 2002.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

This European Standard is based on ASTM D244-95 (clauses 11 to 15) [1].

This draft is part of a package including 15 standards : EN(WI 00019104), EN 1428, EN 1429, EN 1430, EN 1431, EN 12846, EN 12847, EN 12848, EN 12849, EN 12850, EN 13074, EN 13075-1, EN 13075-2, EN 13614-1, EN 13614-2.

In this standard annex A is normative and annex B is informative.

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1 Scope

This European Standard specifies a method for the quantitative determination of recovered binder and oil distillate in bitumen emulsions composed principally of a semisolid or liquid bituminous base, water and an emulsifying agent.

The method can be used for quantitative determination of recovered binder and oil distillate in bitumen emulsions for specification acceptance, service evaluation, control and research. The method can also be used to obtain residue and oil distillate for further testing.

NOTE 1 : 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.

NOTE 2 : The method described recovers only oil distillate as defined in 3.1.

NOTE 3 : In the case of delay of distillation during testing of polymer modified bitumen emulsions, the test method should be used in accordance with prEN 13074:1997 [2].

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.

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2 Normative references

This European Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies.

EN 58, *Sampling bituminous binders*

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

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

EN 1426, *Bitumen and bituminous binders - Determination of needle penetration*

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

EN 12595, *Bitumen and bituminous binders - Determination of kinematic viscosity*

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

3 Definitions

For the purposes of this standard, the following definitions apply :

3.1

oil distillate

hydrocarbon solvent with a boiling point of less than 260 °C.

3.2

recovered binder

residue from a bitumen emulsion after distillation of water and oil distillate.

4 Principle

Water and oil distillate are distilled from the bitumen emulsion, and separated in a graduated cylinder, leaving a residue of recovered 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 mm \pm 2 mm in height by 94 mm \pm 2 mm internal diameter with a 121 mm \pm 2 mm, or 127 mm \pm 2 mm, inside diameter ring burner, having holes on the inner circumference and having three spacers, to ensure centring of burner under the still (see figure 2).

6.2 **Connection apparatus**, consisting of a glass connecting tube, tin shield, and water cooled glass condenser tube with a metal or borosilicate glass jacket.

6.3 **Graduated cylinder**, 100 ml, with graduation intervals of 1,0 ml.

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, recognising and allowing for the fact of changed thermal response times compared with the mercury thermometer.

For this test method, in which increasing temperatures are read during the test procedure, 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 g \pm 0,1 g, A_m of the emulsion sample into the previously weighed still (6.1) (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 mm from the bottom of the still and the bulb of the other is 165 mm from the bottom of the still.
- 8.4** Place the ring burner (6.1) at least 150 mm up 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 $^{\circ}\text{C}$, lower the ring burner until the reading on the thermometer is 260 $^{\circ}\text{C} \pm 5$ $^{\circ}\text{C}$. Maintain the temperature at this level for 15 min.

NOTE : The time taken to raise the temperature from 215 $^{\circ}\text{C}$ to 260 $^{\circ}\text{C}$, should be kept between 30 min and 60 min. The temperature and time for the last 15 min of the procedure are very important for the precision of the method.

- 8.6** At the end of the heating period, weigh immediately the still and accessories as in 8.1. The aluminium alloy still at room temperature weighs 1,5 g more than at 260 $^{\circ}\text{C}$. Correct for this by adding 1,5 g to the gross mass obtained prior to determining the mass of residue after distillation, B_m . 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) can be added to distillates from cationic emulsions.

- 8.7** Remove the cover from the still, stir the residue, and pour it immediately through a 300 μm sieve (6.7). 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 method (for example EN 1426 or EN 12595) from the points that follow the pouring stage.

Retain the oil distillate for identification if required.

9 Calculation

Calculate the recovered binder (r), as a percentage by mass, using the following equation :

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

where :

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

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

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

$$o = \frac{D}{A_m} \times 100 \quad \dots(2)$$

where :

D is the volume of oil distillate, in millilitres (see 8.6).

NOTE : The density of the emulsion is assumed to be 1 000 kg/m³ at 15 °C.
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10 Expression of results

Express the recovered binder as a percentage by mass to the nearest 1 %. Express the volume of oil distillate as a percentage by volume to the nearest 0,1 %.

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, differ by more than the value given in the below table 1 in only one case in twenty.

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, differ by more than the value given in the below table 1 in only one case in twenty.

Table 1 : Precision

Residue by distillation % (<i>m/m</i>)	Repeatability % (<i>m/m</i>)	Reproducibility % (<i>m/m</i>)
50 to 70	1,0	2,0

12 Test report

The test report shall contain at least the following information :

- a) the type and complete identification of the sample under test ;
- b) a reference to this European Standard ;
- c) the result of the test (see clause 10) ;
- d) any deviation, by agreement or otherwise, from the procedure specified ;
- e) the date of the test.

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