BRITISH STANDARD

BS EN 15326:2007 +A1:2009
BS 2000-549:2007

Bitumen and bituminous binders — Measurement of density and specific gravity — Capillary-stoppered pyknometer method

ICS 75.140; 91.100.50

BSI British Standards

National foreword

This British Standard is the UK implementation of EN 15326:2007+A1:2009. It supersedes BS EN 15326:2007, which is withdrawn.

The start and finish of text introduced or altered by amendment is indicated in the text by tags. Tags indicating changes to CEN text carry the number of the CEN amendment. For example, text altered by CEN amendment A1 is indicated by [A1] ⟨A1⟩.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum testing and terminology.

A list of organizations represented on this committee can be obtained on request to its secretary.

Energy Institute, under the brand of IP, publishes and sells all Parts of BS 2000, and all BS EN petroleum test methods that would be Part of BS 2000, both in its annual publication "Standard methods for analysis and testing of petroleum and related products and British Standard 2000 Parts" and individually.

Further information is available from: Energy Institute, 61 New Cavendish Street, London W1G 7AR. Tel: 020 7467 7100. Fax: 020 7255 1472.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 April 2007

ISBN 978 0 580 64493 1

Amendments/corrigenda issued since publication

Date	Comments
31 August 2009	Implementation of CEN amendment A1:2009

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15326:2007+A1

May 2009

ICS 75.140; 91.100.50

Supersedes EN 15326:2007

English Version

Bitumen and bituminous binders - Measurement of density and specific gravity - Capillary-stoppered pyknometer method

Bitumes et liants bitumineux - Mesure de la masse volumique et de la densité - Méthode du pycnomètre à bouchon capillaire

Bitumen und bitumenhaltige Bindemittel - Messung der Dichte und der relativen Dichte - Pyknometerverfahren mit Kapillarstopfen

This European Standard was approved by CEN on 10 February 2007 and includes Amendment 1 approved by CEN on 5 April 2009.

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 CEN Management Centre 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 respons bility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 15326:2007+A1:2009: E

Contents

Page

Foreword

This document (EN 15326:2007+A1:2009) 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 November 2009, and conflicting national standards shall be withdrawn at the latest by November 2009.

This document includes Amendment 1 approved by CEN on 2009-04-05.

This document supersedes EN 15326:2007.

The start and finish of text introduced or altered by amendment is indicated in the text by tags 〚A1〉 〈A1〛.

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

1 Scope

This European standard specifies a procedure for determining the specific gravity and density of bituminous binders at (25,0 ± 0,2) °C using the capillary-stoppered pyknometer method. Emulsions are excluded from the scope of this method.

NOTE 1 This method can also be used for other hydrocarbon binders, for bituminous binders from other sources, e.g. recovered from asphalt or after hardening and for bituminous binders containing filler. However, a lower precision is to be expected.

NOTE 2 This test method may be performed at other temperatures (i.e. 15 °C). When doing so, the density values of water and isopropanol should be determined.

The pyknometer method shall be used as a reference method for determining the specific gravity and density of bituminous binders.

WARNING — 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 1427, *Bitumen and bituminous binders – Determination of the softening point – Ring and Ball method*

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

3 Terms and definitions

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

3.1
density (*ρ*)
mass per unit of volume of a material, measured in unit of kg/m^3

NOTE 1 Standard temperature for determining the density of bituminous binders is (25,0 ± 0,2) °C. Density, ρ, is calculated from specific gravity, d, of the bituminous binder.

NOTE 2 Density is used for converting volumes to mass as required by other standards, applications and in sale transactions.

3.2
specific gravity (*d*)
ratio of density of the bituminous binder, ρ, and density of a test liquid, ρ_T, measured under the same conditions, at 25,0 °C

$$d_{25/25} = \frac{\rho}{\rho_T} \quad (1)$$

4 Principle

Masses of equal volumes of bituminous sample and of test liquid are compared. Equal volumes are ensured by the pyknometer being filled so as to overflow when placed in a bath at the test temperature until thermal equilibrium is reached (approximately 30 min).

The calibrated pyknometer is filled approximately three quarters of its capacity with the bituminous binder to be tested and weighed with the stopper. The pyknometer is filled with a test liquid and weighed again. From the different masses, specific gravity and density shall be calculated.

5 Reagents

5.1 Test liquids

Freshly boiled and cooled distilled water or deionized water shall be used to fill the pyknometer, the low-form beaker (6.6) and the waterbath.

For testing petroleum cut-back and fluxed bitumen for which specific gravity at 25 °C is assumed to be lower than 1, isopropanol (propan-2-ol) with a density of (782,7 ± 0,1) kg/m^3 at 25 °C shall be used.

5.2 Solvents

Appropriate solvents are required to clean the equipment used.

6 Apparatus

6.1 Pyknometer

Glass, consisting of a conical or cylindrical vessel carefully ground to receive an accurately fitting glass stopper. The stoppered pyknometer shall have a capacity of (25 ± 2) cm^3, and shall weigh not more than 40 g. The wide-mouth (Hubbard) form of pyknometer may be used as detailed in ISO 3507. In case larger samples are available, a larger Hubard pyknometer with a 50 ml capacity may be used also detailed in ISO 3507; masses should be determined with the same accuracy.

The upper part of the stopper shall be smooth and perfectly flat. Its lower part shall be concave to allow air bubbles to easily escape through the opening. The concave part shall have a height of (5 ± 1) mm in the middle.

An example of suitable pyknometer is illustrated in Figure 1.

Dimensions in millimetres

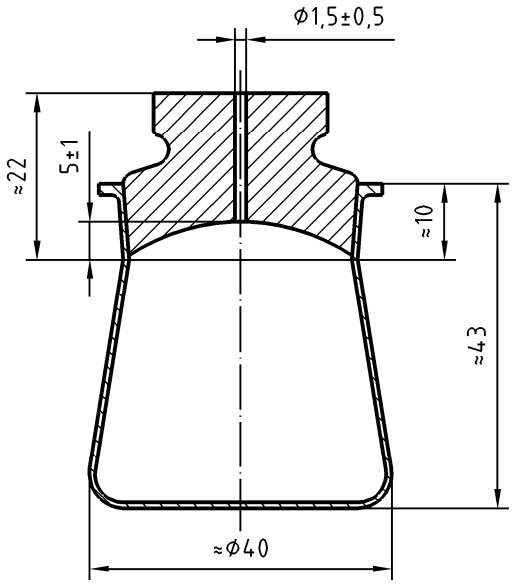

Figure 1 — Example of a capillary-stoppered pyknometer

6.2 Constant-temperature water bath, having a depth greater than that of the pyknometer, capable of being maintained within (± 0,2) °C of the desired temperature.

6.3 Bath thermometer

Thermometer shall be readable to (± 0,1) °C and shall be linked to the agreed reference standards.

6.4 Balance

Laboratory balance capable to reach the required precision.

6.5 Appropriate oven

For temperatures up to 220 °C, in order to melt any kind of bituminous binder. The appropriate temperature shall be adapted to the type of binder tested.

6.6 Low-form beaker

Capacity of 600 ml is convenient.

7 Procedure

7.1 General

Samples shall be taken and prepared according to EN 58 and EN 12594.

7.2 Preparation of equipment

7.2.1 Preparation of the water bath

Fill the water bath with distilled water or deionized water and maintain the temperature of the water bath within (25 ± 0,2) °C.

7.2.2 Conditioning of the low –form beaker

Partially fill the 600 ml low-form beaker (6.6) with freshly boiled and cooled distilled or deionized water to a level that will allow the pyknometer to be immersed to a depth of not less than approximately 40 mm.

Partially immerse the beaker in the water bath to a depth sufficient to allow the bottom of the beaker to be immersed to a depth of not less than approximately 100 mm, while the top of the beaker is above the water level of the bath. Clamp the beaker in place for approximately 30 min.

7.3 〈A1〉 Calibration of the pyknometer

7.3.1 General

The calibration shall be carried out at least once a year in accordance with the procedure detailed below. Such a calibration is valid only for the temperature at which it is carried out.

In this standard, except if mentioned explicitly, the reference to the pyknometer specifies the pyknometer body and its stopper.

During the year or in case of doubt about any result, a new determination of the mass of the empty pyknometer and the mass of the pyknometer filled with the test liquid shall be carried out.

The calibrated pyknometers shall be preserved from dust and from stains.

When first using a new pyknometer, register the pyknometer body and its stopper with a permanent readable marking that may not change the mass of the pyknometer through time (typically engraved glass). Mention clearly that the pyknometer body and its stopper are not dissociable.

7.3.2 Calibrations of the mass of the empty pyknometer and the mass of the pyknometer filled with the test liquid

7.3.2.1 Determination of the mass of the empty pyknometer and the mass of the pyknometer with the test liquid by 2 operators

When evaluating the mass of the empty pyknometer (A) and the mass of the pyknometer filled with the test liquid (B), the measurements shall be carried out by at least 2 operators, if possible on the same day.

If only one operator carries out the 2 determinations, i.e. the determinations of the mass of the empty pyknometer (*A*) and the mass of the pyknometer filled with the test liquid (*B*), each of those two determinations shall carried out twice within at least 24 h in between but no more than 72 h.

7.3.2.2 Calibration of the mass of the empty pyknometer (A)

Thoroughly clean the pyknometer with solvent and remove the solvent completely by storing the pyknometer in an oven at approximately 10 °C above the boiling point of the solvent used. Allow the pyknometer to cool to ambient temperature (18 °C to 28 °C) and clean it with care to prevent electrostatic charge.

Weigh the pyknometer to the nearest mg without handling it with fingers but with grippers. Designate this mass as *A*.

7.3.2.3 Referencing the pyknometer and its empty mass (A)

Report in the appropriate document, the pyknometer identification mark (see 7.3.1) and its empty mass (A), as the average of the independent results determined by one or two operators. This mass evaluated at least once a year, will be considered for the determination of the specific gravity or the density as detailed in this standard under Clause 8.

7.3.2.4 Calibration of the mass of the pyknometer filled with the test liquid (B)

Fill the pyknometer with the test-liquid at close to 25 °C, place the stopper on loosely. Place the pyknometer in the low-form beaker (7.2.2) and press the stopper firmly in place. If the test liquid is water, the water surface shall overflow the capillary stopper of the pyknometer; if the test liquid is isopropanol the water surface shall be in the middle of the capillary stopper.

Allow the pyknometer to remain in the beaker for not less than 30 min. Remove the pyknometer from the bath, immediately dry the top of the stopper with one stroke of a dry towel and then quickly dry the remaining outside area of the pyknometer. The top of the stopper shall not be re-dried even if a small droplet of water forms when removing the pyknometer from the beaker, the proper mass of the contents at the test temperature is thus recorded. If moisture condenses on the pyknometer during weighing, quickly redry the outside of the pyknometer (excluding the top) before recording the mass.

Weigh to the nearest mg. Designate the mass of the pyknometer plus test liquid as *B*.

Empty the pyknometer and dry it in the oven at (110 ± 5) °C.

7.3.2.5 Referencing the pyknometer and its mass determined with the test liquid (B)

Report in an appropriate document, the pyknometer identification mark and its mass determined with the test liquid (B), as the average of the independent results determined by one or two operators. This mass evaluated at least once a year, will be considered for the determination of the specific gravity or the density as detailed in this standard under Clause 8. 〈A1〉

7.4 〈A1〉 Performing the routine test

7.4.1 Determination of the empty mass of the pyknometer

Before any determination of the specific gravity or the density of a bituminous binder, the empty pyknometer shall be weighed and its mass shall be compared to the mass (A) determined when calibrating (see 7.3.2.2 and 7.3.2.3). In case those 2 masses are in no way different from more than 5 mg, the masses (A) and (B) evaluated when calibrating the pynometer shall be used for the calculations detailed under Clause 8. On the contrary, a new calibration shall be performed as detailed under sub-clause 7.3.

7.4.2 Weighing of the pyknometer filled with bituminous binder sample

Pour enough sample into the clean, dry, warmed pyknometer (within the range 50 °C to 80 °C) to fill three quarters of its capacity. Take precautions to keep the material from touching the sides of the pyknometer above the final level and to prevent the inclusion of air bubbles. Except when testing fluxed or cut-back bitumen, in case bubbles are visible on the surface and/or on the walls of the pyknometer, place the filled pyknometer (without stopper) in an oven, for (20 ± 2) min., at a temperature of 80 °C to 90 °C above the softening point, determined by Ring and Ball method (EN 1427) to allow air bubbles to disappear.

Allow the pyknometer and its content to cool to ambient temperature for a period of not less than 40 min, protecting it from dust. Weigh the pyknometer with the stopper to the nearest 1 mg. Designate the mass of the pyknometer plus sample as *C*. 〈A1〉

7.5 Weighing of the pyknometer filled with bituminous binder sample added with test liquid

Add test liquid to the pyknometer already containing the bituminous binder sample, place the stopper loosely, place the pyknometer in the beaker at (25 ± 0, 2)°C and press the stopper firmly in place. If the test-liquid is water, the water surface shall overflow the capillary stopper of the pyknometer. If the test-liquid is isopropanol, the liquid surface shall be in the middle of the stopper. Allow the pyknometer to remain in the beaker for a period of no less than 30 min. Remove the pyknometer from the beaker. Dry and weigh using the same technique and timing as employed in 7.3.3. Designate the mass of the pyknometer plus sample plus test liquid as *D*.

8 Calculations

8.1 Symbols

The following symbols are used in the calculations:

A is the mass (g) of the pyknometer including weight of the stopper;

B is the mass (g) of the pyknometer filled with test liquid (water or isopropanol);

C is the mass (g) of the pyknometer partially filled with bituminous binder sample;

D is the mass (g) of the pyknometer plus bituminous binder sample plus test liquid;

$d_{25/25}$ is the specific gravity of bituminous binder at 25 °C;

ρ_T is the density of the test liquid.

In case water is used as the test liquid, density shall be considered equal to 997,0 kg/m^3 at 25,0 °C. For testing cut-back and fluxed bitumen where isopropanol is required, its density shall be considered equal to 782,7 kg/ m^3 at 25 °C.

NOTE Water density value at 25,0 °C mentioned above is taken from a table reported in EN ISO 3838 [2]. In case the test method is performed at any other temperature, water density at this temperature should be found out from the same table if possible.

8.2 Calculations of specific gravity and density

Calculate specific gravity, *d*, with 3 decimal places, and density of the bituminous binder, *ρ*, to the nearest 1kg/m^3 as follows:

$$d_{25/25} = \frac{C - A}{(B - A) - (D - C)} \quad (2)$$

and

$$\rho = \frac{C - A}{(B - A) - (D - C)} \times \rho_T \quad (3)$$

Two determinations minimum are necessary, starting each determination from sub-clause 7.1. The result is the average of the two first determinations not differing more than 2 kg/m^3.

NOTE For the binders from other procedures, e.g. recovered from asphalt, after hardening or for binders containing filler, a lower precision is to be expected.

9 Precision

9.1 Repeatability

The difference between two 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 operation of the test method, exceed the values given in Table 1 only in one case in twenty.

9.2 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 operation of the test method, exceed the value given in Table 1 only in one case in twenty.

The following criteria shall be used for judging the acceptability of results

Table 1 — Precision values on density

Binder	Repeatability kg/m^3	Reproducibility kg/m^3
Bitumen	3	5
Fluxed bitumen	3	6
Other binders	3	7
NOTE Precision values of specific gravity are 1/1 000 of the values mentioned in Table 1 dealing with density.		

10 Report

The test report shall contain at least the following information:

a) type and complete identification of the sample under test;

b) reference of this European standard;

c) reference to the type of pyknometer used;

d) test liquid used and bath temperature;

e) results of specific gravity and density (to the nearest kg/m^3);

f) any deviation, by agreement or otherwise, from the specified procedure;

g) date of the test.

Bibliography

[1] EN 12597, *Bitumen and bituminous binders – Terminology.*

[2] EN ISO 3838, *Crude petroleum and liquid or solid petroleum products – Determination of density or relative density – Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods (ISO 3838:2004).*

[3] ASTM D70-03, *Standard Test Method for Density of Semi-Solid Bituminous Materials (Pycnometer Method).*

[4] ISO 3507, *Laboratory glassware – Pyknometers*

blank

BS 2000 series

Energy Institute

Buying Parts of BS 2000

Orders for BS 2000 publications should be addressed to either:

Energy Institute - Library and Information Service
61 New Cavendish Street
London
W1G 7AR

Tel:+44 (0)20 7467 7100
Fax: +44 (0)20 7255 1472
www.energyinst.org.uk

Order standards securely via:
www.energyinstpubs.org.uk

or:

British Standards Institution - Customer Services
389 Chiswick High Road
London
W4 4AL

Tel:+44 (0)20 8996 9001
Fax: +44 (0)20 8996 7001
www.bsi-global.com

Order hard copy standards securely via:
www.bsi-global.com/bsonline

Copyright