BRITISH STANDARD

BS EN 12593:2007
BS 2000-80:2007

Bitumen and bituminous binders — Determination of the Fraass breaking point

The European Standard EN 12593:2007 has the status of a British Standard

ICS 74.140; 91.100.50

BSI British Standards

National foreword

This British Standard was published by BSI. It is the UK implementation of EN 12593:2007. It supersedes BS EN 12593:2000/IP which is withdrawn.

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 31 May 2007

ISBN 978 0 580 50768 7

Amendments issued since publication

Amd. No.	Date	Comments

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 12593

March 2007

ICS 75.140; 91.100.50

Supersedes EN 12593:1999

English Version

Bitumen and bituminous binders - Determination of the Fraass breaking point

Bitumes et liants bitumineux - Détermination du point de fragilité Fraass

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Brechpunktes nach Fraaß

This European Standard was approved by CEN on 3 February 2007.

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 responsibility 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: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 12593:2007: E

Contents

Page

Foreword

This document (EN 12593:2007) 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 2007, and conflicting national standards shall be withdrawn at the latest by September 2007.

This document supersedes EN 12593:1999.

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 method for determining the Fraass breaking point which measures the brittleness of bitumen and bituminous binders at low temperatures.

WARNING — Use of this European Standard can involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced standards 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 standard (including any amendments) applies.

EN 58, *Bitumen and bituminous binders - Sampling bituminous binders*

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

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 term and definition applies.

3.1
Fraass breaking point
temperature, expressed in degrees Celsius, at which a film of bituminous binder of a specified and uniform thickness will break under defined loading conditions

4 Principle

A sample of bituminous binder is applied to a metal plate at an even thickness. This plate is submitted to a constant cooling rate and flexed repeatedly until the binder layer breaks; the temperature at which the first crack appears is reported as the Fraass breaking point

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Plates, made of tempered spring steel with the following dimensions: (41,00 ± 0,05) mm long, (20,0 ± 0,2) mm wide and (0,15 ± 0,02) mm thick. The plates shall be kept flat and protected from corrosion when not in use. Any plate that becomes visibly curved or corroded shall be discarded.

5.2 Plate preparation equipment, used for application of the melted sample, and including:

5.2.1 Magnet block with a flat and smooth surface (Figure 1) holding one to three plates with a suitable cover (Figure 2).

5.2.2 Metal support with two distinct zones: one temperature regulated and controlled, the other one cooled by water circulation. The support shall be horizontal and include an air bubble level and level adjustment screws.

5.3 Fraass breaking apparatus, as shown in Figure 3, consisting of the parts described in 5.3.1 to 5.3.3.

NOTE Manual apparatus can be replaced by semi automatic or automatic apparatus reproducing the same conditions.

5.3.1 Bending apparatus, as shown in Figure 4. The clearance between the two tubes, when assembled so that one can move longitudinally within the other, shall not exceed 1 mm. The tubes shall be made of a material that is of low thermal expansion (linear expansion coefficient: $< 40 \times 10^{-6}$ 1/K) and a poor conductor of heat (thermal conductivity: < 0,3 W/K x m).

The plate shall be held by two steel clips as shown in Figure 5, the upper clip being attached to the bottom end of the outer tube, and the lower clip being attached to the inner tube by means of a metal connecting piece. The clips shall be coplanar, parallel to the axis of the tube, and secured against twisting. The thermometer shall be mounted in such a way that the connecting piece does not act as a shield between the thermometer bulb and ambient temperature and that the reservoir of the thermometer is the same distance between the wall of the inner tube and the middle of the pre-bended test plate when at rest.

By rotating the crank handle (see Figure 3), which operates a mechanism consisting of a cone of hardened metal, as shown in Figure 6, and a setting screw, the inner tube may be moved up and down relative to the outer tube. Eleven rotations of the handle shall permit the initial distance between the upper and lower clip of (40,0 ± 0,1) mm to be steadily reduced by (3,5 ± 0,1) mm.

A steel strut may be used to fix the initial bending of the steel test plate. The height of the strut is such that, when in place, the initial distance between the upper and the lower clips is (40,0 ± 0,1) mm.

Use of a semi automatic bending apparatus, from which the raising and lowering of the inner tube is controlled, for example, by a motor-driven cam disc, or of fully automatic apparatus, in which the reduction in temperature is controlled and the breaking point is automatically indicated, is permitted, provided that the test conditions specified in this standard are complied with.

5.3.2 Cooling apparatus, as shown in Figure 3, and comprising the inner test tube (5), the outer test tube (4) and the glass cylinder (1). The bungs (6), (7) and (8) shall be made of either rubber or cork. The bore (9) in the bung (7) may be used for introducing solid carbon dioxide. The cylinder (1) and the inner test tube (5) shall contain a small amount of drying agent. A transparent Dewar vessel having an inside diameter of (75 ± 5) mm may be substituted for the outer test tube (4) and the cylinder (1).

NOTE 1 Care should be taken to ensure that all elements of the apparatus are vertical.

NOTE 2 Suitable bath liquids for the Dewar vessel are alcohols like ethanol, 1-propanol or 2-propanol.

5.3.3 Thermometer, solid stem, as 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 and 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 changed thermal response times compared with the mercury thermometer.

NOTE For this test method, in which decreasing temperatures are read during the test procedure, documented corrections should be determined in advance and applied to the observed readings.

5.4 Press, consisting of a base plate, a frame and two metal pressure blocks, measuring 100 mm x 72 mm x 25 mm (see Figure 7). The lower pressure block shall be mounted on an intermediate disk made of non-conducting material (see 5.5) and having the same dimensions as the pressure block.

A recess measuring 72 mm x 60 mm x 0,7 mm shall have been let into the pressure surface of the lower block. A metal plate shall be fixed at the vertex of the upper block with non-conducting material being used to separate the two. The vertex plate shall be fitted with a spindle that is turned by a handle. The thread of the spindle shall pass through the frame.

The depth of the notch shall be between 0,2 mm and 0,5 mm.

Electrical heating elements shall be fitted between the pressure blocks and the non-conducting material (see 5.5), and connected to a control device, by means of which the temperature of the pressure blocks can be varied. The pressure blocks shall be provided with bore holes into which the sensors of temperature measuring instruments shall be inserted.

5.5 Separating films, heat-proof, such as films made of cellulose hydrate, 0,05 mm thick, or silicone-coated paper.

5.6 Gripping pliers, for inserting the test plates between the clips. The ends of the gripping arms shall not be more than 8 mm wide and a block shall be used to prevent the ends approaching each other closer than 35 mm, to prevent excessive flexing of the test plates during insertion.

5.7 Balance, accurate to ± 5 mg.

6 Sampling and sample preparation

6.1 General

Take the test sample in accordance with EN 58, taking all necessary safety precautions and ensuring that the test sample is representative of the laboratory sample from which it is taken. Ensure that the laboratory sample is homogeneous and is not contaminated (see EN 12594 and EN 1425).

6.2 Coating test plates

Prepare three plates (5.1) by cleaning with a suitable and appropriate degreasing solvent, then drying and weighing to the nearest 0,01 g or tare with the same accuracy. For the repeatability and reproducibility of test results, it is essential that all tests are carried out using binder films of uniform thickness.

NOTE For instance solvents such as acetone, cyclohexane or methylene chloride can be used.

6.3 Melt application of sample

The plate shall be coated manually.

Weigh (410 ± 10) mg of bituminous binder on to the plate and place the plate on the magnet block (5.2.1).

NOTE 1 The sample can be in a heated (liquid) or unheated (normally solid) form as preferred.

The magnet block shall then be placed on the heating metal support (5.2.2) which is controlled at a temperature not exceeding the Ring and Ball softening point of the bituminous binder (see EN 1427) by more than 80 ºC. For polymer modified bitumen, the temperature may not exceed 200 °C irrespective to the softening point.

As soon as the fluidity of the bituminous binder is sufficient, ensure a uniform distribution of the bituminous binder by manipulating the heating support.

NOTE 2 If necessary, a thin bladed instrument can be used (e.g. a rejected penetration needle) to assist in obtaining a uniform distribution.

If the remaining amount of bituminous binder is less than required, prepare a new plate. Allow the plate to rest for 1 min to 2 min to ensure that the entire plate is evenly coated with the bituminous binder and the surface is flat.

By carefully fanning with a flame, dispel any small air bubbles, which may have become entrapped, avoiding local overheating.

Move the magnet block supporting the plate to the cooling side of the support with the help of the cover.

NOTE 3 The total plate preparation should not take more than 10 min.

Leave the plates to lie horizontally on the cooled support at ambient temperature protected by a cover.

6.4 Press-application of sample

For binders with a Ring and Ball softening point (determined according to EN 1427) greater than 100 ºC, the plate shall be laid centrally on a separating film measuring 100 mm x 100 mm. Approximately 2 g of binder shall be placed in the centre of the plate and covered with another separating film. The plate thus prepared shall be laid in the recess of the lower pressure block of the press, which has been heated in advance to about 20 ºC above the softening point of sample under test (see Figure 8).

The upper pressure block shall be lowered by means of the threaded spindle until it rests on the lower block, and left in this position for 1 min. Then the upper block shall be raised again, and the coated test plate, after being left to cool to ambient temperature, shall be placed in ice water, with the separating films still in position. After approximately 2 min, the separating films shall be removed, and the sample trimmed to the edge of the metal plate with help of a sharp blade. The coated test plate shall then be weighed.

The binder quantity pressed on shall be (410 ± 10) mg. If the coated test plate fails the weight check, then another metal plate shall be coated according to the same procedure.

7 Procedure

7.1 Test conditions

Test the coated test plate 30 min to 240 min after coating starting at a temperature of at least 15 ºC above the expected breaking point. Cool at 1 ºC/min and bend at every degree Celsius starting at least 8 ºC and not more than 12 ºC above the anticipated breaking point.

NOTE If necessary, for a relatively high Fraass breaking point, the coated test plate can have a temperature above ambient to allow enough time to stabilize the cooling rate at 1 ºC/min.

7.2 Measurement

Insert the coated test plate between the clips with the help of the gripping pliers (5.6). Take care when inserting the test plate to ensure that it bends gently enough for the binder film not to crack at this stage of the test. Should a crack in the film still occur, replace with another coated plate.

Mount the bending apparatus in the inner test tube of the cooling apparatus, and introduce the thermometer so that its bulb is located centrally behind the test plate inserted between the clips. Commence cooling at a rate of 1 ºC/min. To achieve this, the space between the inner and the outer test tube shall be filled to a level of at least 100 mm with bath liquid (alcohol or any similar bath liquid), the temperature of which has been

adjusted to match the test plate temperature, the fall in temperature being produced by the addition of small quantities of solid carbon dioxide. The first 3 min are used to establish the fall in temperature at the rate specified. After an initial fall of 3 ºC, the temperature shall continue to fall by 1 ºC every (60 ± 5) s. No variation shall exceed this maximum permissible variation of ± 5 s, nor shall the variation be averaged over the period of the test.

Commence flexure of the test plate at a temperature of (10 ± 2) ºC above the expected breaking point.

Bend and stretch the test plate by turning the handle at a uniform rate of 1 r/s until it is arrested, examine the binder film for the appearance of the first crack and record the temperature at which it occurred to the nearest 1 ºC, then, without pause, turn the handle backwards at the same speed.

Between the periodic bending stresses, the bending apparatus is maintained for (38 ± 5) s in the initial position.

If the bending apparatus has been removed from the inner test tube during the test, even for examination, the test shall be discontinued.

7.3 Determination of the Fraass breaking point

The first plate tested allows a determination of the approximate value of the breaking point: T_j. with the first flexing carried out at a temperature higher than T_j by 8 ºC to 12 ºC.

If this condition is satisfied for the first measurement, then $T_j = T_0$ and the test shall be repeated to determine T_1.

If this condition is not satisfied for the first measurement, then two further determinations shall be carried out (T_2, T_3).

7.4 Maximum range of the valid determinations

The two determinations (T_0, T_1) or (T_2, T_3) are acceptable provided their difference is less than or equal to 3 ºC. If the difference is greater than 3 ºC, carry out two other additional measurements (T_4, T_5). If the difference between T_4 and T_5 is less than or equal to 3 ºC, these values are valid.

If the difference between T_4 and T_5 is again greater than 3 ºC, calculate the average of the 4 values T_0, T_1, T_4 and T_5 or T_2, T_3, T_4 and T_5 and indicate the range of the results.

8 Expression of results

Express the Fraass breaking point in degrees Celsius as the average of two (or four) valid determinations (see 7.4), rounded to the nearest whole number.

9 Precision

9.1 Repeatability

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

9.2 Reproducibility

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

Table 1 — Precision limits

	Repeatability, **r** °C	Reproducibility, **R** °C
Fraass Breaking Point	3	6

NOTE 1 Values are the same for manual, semi-automatic and automatic apparatus.

NOTE 2 These precision data are not automatically applicable to modified bitumen and for modified bitumen they should only be used for guidance, until criteria data are available. These precision data state the best available data at the moment. Further Round Robin Tests should be carried out.

10 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) reference to the type of apparatus used (manual, semi-automatic or automatic);

d) result of the test (see Clause 8);

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

f) date of the test.

Dimensions in millimetres (indicative of the suitable apparatus)

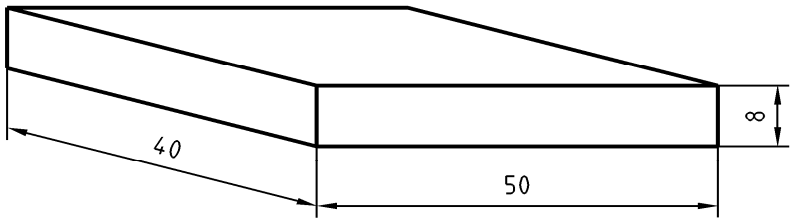


Figure 1 — Magnet block

Dimensions in millimetres

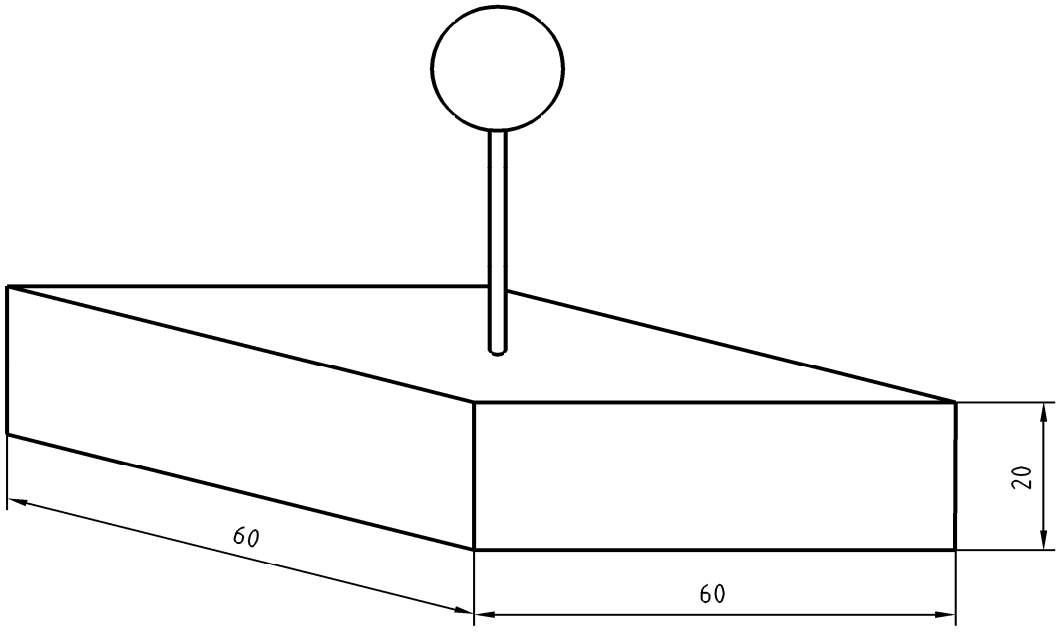
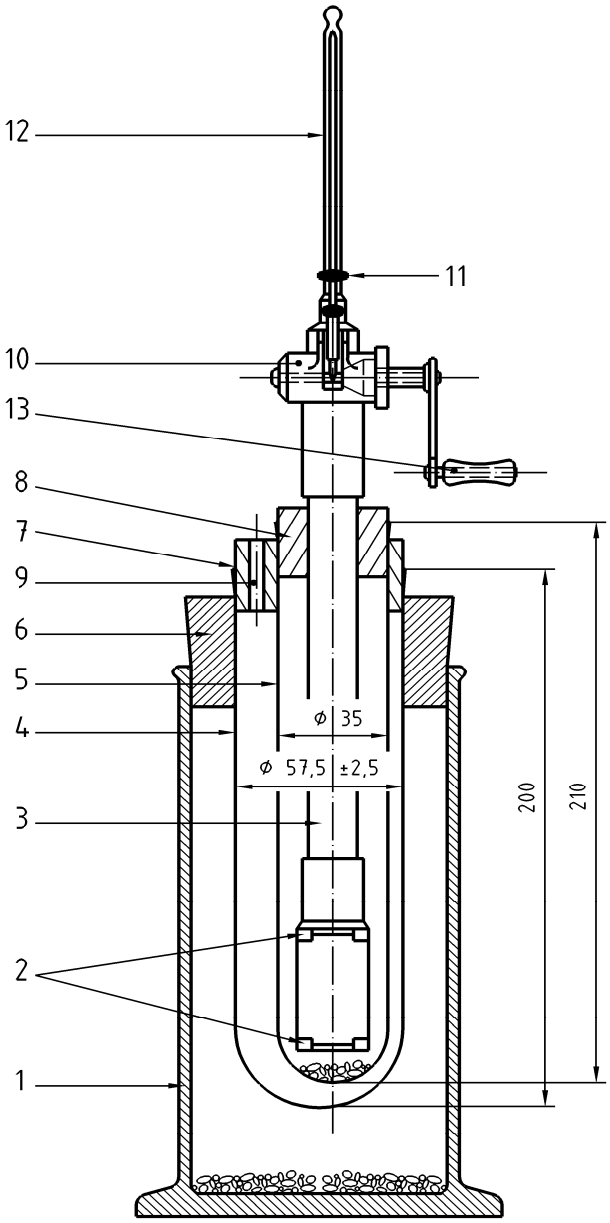


Figure 2 — Metal cover (1,5 mm thickness)

Dimensions in millimetres

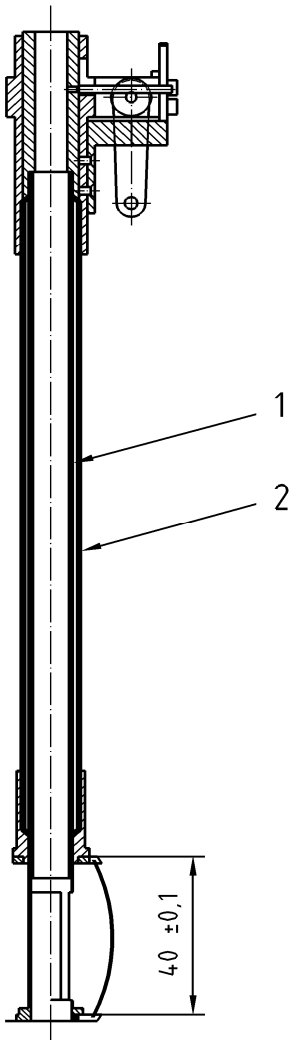


Key

1. Cylinder
2. Upper and lower clips
3. Outer tube of the bending apparatus
4. Outer test tube
5. Inner test tube
6. Bung for cylinder (1), holding the outer test tube (4)
7. Bung for outer test tube (4), holding the inner test tube (5)
8. Bung for inner test tube (5), holding the bending apparatus
9. Bore
10. Cone
11. Setting screw
12. Thermometer
13. Crank handle

Figure 3 — Example of a Fraass breaking point apparatus (the dimensions specified are mandatory)

Dimensions in millimetres



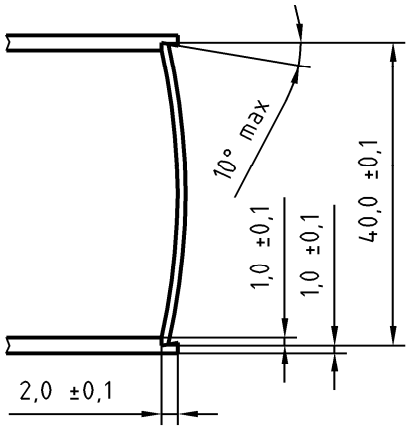
Key

1) Inner tube of the bending apparatus - External diameter : 11,5, internal diameter: 7,5

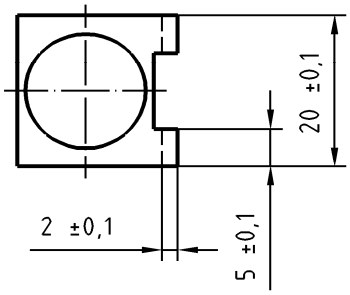
2) Outer tube of the bending apparatus - External diameter : 16,5, internal diameter: 12,5

Figure 4 — Lower part of the bending apparatus with test plate in position

Dimensions in millimetres



(side view with test plate in initial position)



(plan view shown without test plate)

Figure 5 — Clips

Dimensions in millimetres

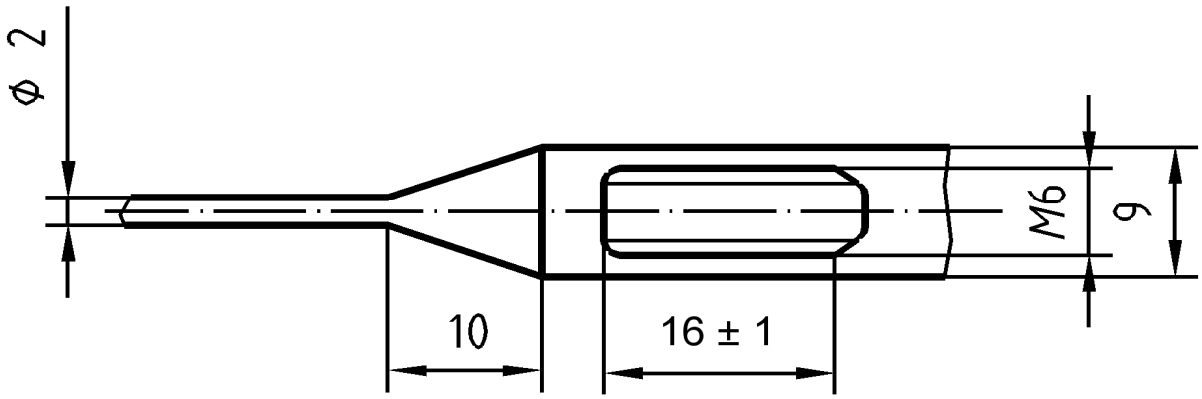
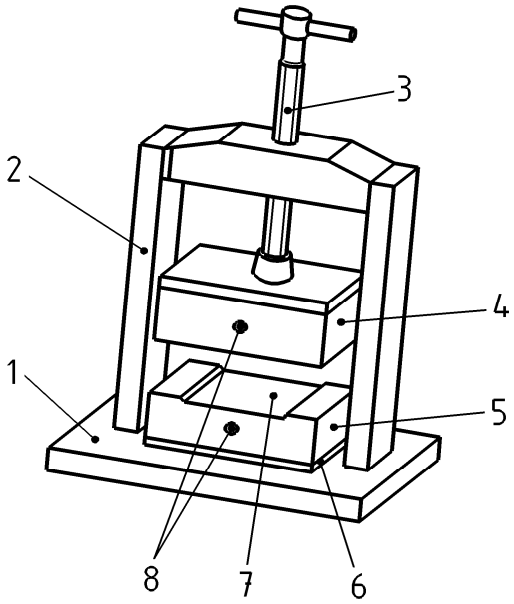


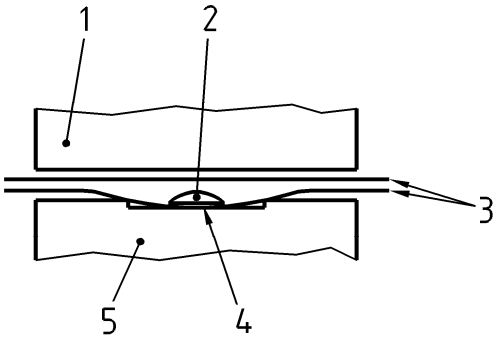
Figure 6 — Cone



Key

1) Base
2) Frame
3) Stem with handle
4) Upper pressure block
5) Lower pressure block
6) Intermediate disk
7) Slot
8) Holes for temperature measurement

Figure 7 — Press



Key

1) Upper pressure block
2) Sample
3) Separation film
4) Test plate
5) Lower pressure block

Figure 8 — Scheme of sample press-application

Annex A
(normative)

Characteristics of the thermometer

Temperature range	°C	-38 to +30
Scale marks		
Subdivision	°C	0,5
Long lines at each	°C	1 and 5
Numbers at each:	°C	5
Maximum line width	mm	0,15
Maximum scale error	°C	0,5
Immersion	mm	250
Expansion chamber : permits heating to	°C	80
Total length	mm	360 to 380
Stem length	mm	> 60
Stem outside diameter	mm	6,0 to 7,0
Bulb length	mm	10 to 16
Bulb outside diameter	mm	< stem diameter
Scale location		
Distance between bottom of bulb to line at -38 °C	mm	271 approximately
Length of scale	mm	72 approximately

NOTE 1 The thermometer IP 42C has been found suitable.

NOTE 2 Thermocouple thermometers can be used instead of solid stem thermometers, provided that they are calibrated regularly and give the same results.

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