BRITISH STANDARD

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Methods for

Analysis and testing of coke —

Part 107: Caking and swelling properties of coal —

Section 107.3 Determination of swelling properties using a dilatometer

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Solid Mineral Fuels Standards Policy Committee (SFC/-) to Technical Committee SFC/3, upon which the following bodies were represented:

British Cement Association

British Coal Corporation

British Gas plc

British Steel Industry

Department of Trade and Industry (Standards and Quality Policy Unit, Quality, Design and Education Division)

Electricity Supply Industry in England and Wales

GAMBICA (BEAMA Ltd.)

Institute of British Foundrymen

Institute of Petroleum

Power Generation Contractors Association (BEAMA Ltd.)

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Contents

Foreword

This Section of BS 1016 has been prepared under the direction of the Solid Mineral Fuels Standards Policy Committee. It is a partial revision of BS 1016-12:1980 replacing clause **7** from which it differs principally in that a transducer/electrical signal recorder is specified as an alternative means of recording piston movement, and the alternative procedure whereby test pieces are tested in the same retort during independent furnace heating cycles is covered more fully. BS 1016-12:1980 will be withdrawn when Sections 107.1 and 107.2 are published.

This Part is one of the first to be renumbered under a scheme for rationalizing and restructuring BS 1016. The new series, when complete, will begin with Part 100, which will include a general introduction. The earlier series of Parts is as follows, with the new Part numbers (which will be given to revisions when they are published) in parentheses.

— *Part 1: Total moisture of coal (Part 101)*;

— *Part 2: Total moisture of coke (Part 102)*;

— *Part 3: Proximate analysis of coal (Part 104)*;

— *Part 4: Moisture, volatile matter and ash in the analysis sample of coke (Part 104)*;

— *Part 5: Gross calorific value of coal and coke (Part 105)*;

— *Part 6: Ultimate analysis of coal (Part 106)*;

— *Part 7: Ultimate analysis of coke (Part 106)*;

— *Part 8: Chlorine in coal and coke (Part 106)*;

— *Part 9: Phosphorus in coal and coke (Part 106)*;

— *Part 10: Arsenic in coal and coke (Part 106)*;

— *Part 11: Forms of sulphur in coal (Part 106)*;

— *Part 12: Caking and swelling properties of coal (Part 107)*;

— *Part 13: Tests special to coke (Part 108)*;

— *Part 14: Analysis of coal ash and coke ash (Part 114)*;

— *Part 15: Fusibility of coal ash and coke ash (Part 113)*;

— *Part 16: Methods for reporting results (Part 100)*;

— *Part 17: Size analysis of coal (Part 109)*;

— *Part 18: Size analysis of coke (Part 110)*;

— *Part 20: Determination of Hardgrove grindability index of hard coal (Part 112)*;

— *Part 21: Determination of moisture-holding capacity of hard coal (Part 103).*

The following Part in the new series has already been published.

— *Part 111: Determination of abrasion index of coal.*

It is intended to publish the following Sections of BS 1016-107.

— *Section 107.1: Determination of crucible swelling number*;

— *Section 107.2: Assessment of caking power by Gray-King coke test*;

— *Section 107.4: Determination of plastic properties using a constant-torque Gieseler plastometer.*

This Section of BS 1016 is based on ISO 8264:1989 "*Hard coal — Determination of the swelling properties using a dilatometer*", published by the International Organization for Standardization (ISO). The introduction in ISO 8264 explains how the Audibert-Arnu dilatometer test described in ISO 349 (for which there is no corresponding British Standard) will eventually be replaced internationally by the Ruhr dilatometer test described in ISO 8264. This text has been omitted from this Section. References to British Standards, which are not exactly technically equivalent to the references in ISO 8264 to international standards, have been substituted and certain other minor changes have been made. Subject to these changes, and for ease of production, the text of ISO 8264 has been used in this Section.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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1 Scope

This Section of BS 1016 describes a method for the measurement of the swelling of hard coal using a dilatometer.

2 References

The titles of the publications referred to in this standard are listed on the inside back cover.

3 Definitions

For the purpose of this British Standard, the following definitions apply.

3.1
softening temperature; temperature of initial contraction

the temperature at which the downward movement of the dilatometer piston is 0,5 mm

NOTE See θ_1 in Figure 3.

3.2
temperature of maximum contraction

the temperature at which the dilatometer piston reaches its lowest point

NOTE See θ_2 in Figure 3.

3.3
resolidification temperature; temperature of maximum dilatation

the temperature at which the dilatometer piston reaches its highest point

NOTE See θ_3 in Figure 3.

3.4
maximum contraction

the maximum downward movement of the dilatometer piston, measured from the zero point and expressed as a percentage of the initial test piece length

NOTE See *c* in Figure 3 and Figure 4.

3.5
maximum dilatation

the maximum upward movement of the dilatometer piston after contraction, measured from the zero point and expressed as a percentage of the initial test piece length

NOTE See *d* in Figure 3 and Figure 4. The value can be either positive or negative.

3.6
repeatability

the maximum acceptable difference between two determinations which are carried out in the same laboratory, by the same operator with the same apparatus, on test pieces prepared from the same test sample and tested simultaneously in two different retorts during the same heating cycle or separately in the same retort during different heating cycles

3.7
reproducibility

the maximum acceptable difference between the means of two determinations which are carried out in each of two laboratories, on representative portions taken from the same gross sample, after the last stage of sample preparation

4 Principle

A test piece, in the form of a pencil, prepared from powdered coal is heated at a constant rate in a steel retort positioned in a furnace, the temperature monitoring system having been previously calibrated using two reference metals of known melting points. The change in level of a piston resting upon the test piece is observed continuously, and a record is produced which is characteristic of the swelling properties of the coal.

5 Materials

The following materials are required for temperature calibration (**7.1**).

5.1 Graphite pencils, 30 mm long, base diameter 7,4 mm, top diameter 6,8 mm, with a small cylindrical reservoir drilled in the narrow end of each pencil.

5.2 Metal balls, of the following reference metals:

a) lead, analytical reagent grade, assay (Pb) 99,98 % minimum, melting point 327,0 °C;

b) zinc, assay (Zn) 99,87 % minimum, melting point 419,3 °C.

5.3 Water-based blacking

6 Apparatus

6.1 *Mould and accessories*

6.1.1 *Mould* (see Figure 1), made from steel, case-hardened after machining. The bore shall be polished after hardening and the bore and uniformity of taper (i.e. 1 in 50) shall conform to the dimensions given in Table 1.

6.1.2 *Mallet*, plastics head, mass about 200 g.

6.1.3 *Ram* (d) (see Figure 1).

6.1.4 *Press* (see Figure 1).

6.1.5 *Load cell* (h) (see Figure 1), capable of registering a load of 0 to 15 kN.

6.1.6 *Test piece gauge* (i) (see Figure 1).

Table 1 — Dimensions of mould

Dimensions in millimetres

Distance from wide end	Bore (tolerance: – 0,00, + 0,005)
0	7,4
10	7,2
20	7,0
30	6,8
40	6,6
50	6,4
60	6,2
70	6,0

NOTE Information on suitable gauges for this purpose may be obtained from the British Coal Corporation, Coal Research Establishment, Stoke Orchard, Cheltenham, United Kingdom.

6.2 *Dilatometer*

A general arrangement of suitable dilatometer apparatus giving critical dimensions is shown in Figure 2.

6.3 *Dilatometer furnace*

A furnace capable of heating two or more retorts (**6.6**) to a temperature of 550 °C at a rate of 3 K/min. The furnace shall comply with the following operating conditions.

Heat the furnace at a rate of 3 K/min, and measure the temperature at the standard sensing point, i.e. at a position equivalent to that of the centre of a normally sited test piece 30 mm above the internal base of a retort. When the temperature has reached about 450 °C, measure the temperature over the lower 250 mm of the retort. The difference between the probe temperature and the mean temperature shown at the standard temperature sensing position shall be not more than:

2 K in the lower 120 mm;

5 K from 120 mm to 180 mm;

10 K from 180 mm to 250 mm.

NOTE The instrument used to measure the temperature may either be the recorder described in **6.5** or another of at least equal precision.

A suitable furnace (for heating three retorts) is illustrated in Figure 2 and consists of a casing fitted with a base and a top cover. The cover supports in a centre hole a cylindrical block of copper-aluminium alloy CA 104, complying with BS 2872, as manufactured (condition M), of 65 mm diameter and 460 mm long. The block has three holes of 380 mm minimum depth and 15,0 mm ± 0,1 mm diameter, drilled as shown in Figure 2. The top surface may be insulated by an appropriately shaped piece of board. The block is heated electrically by an insulated resistance winding, capable of raising the temperature of the block to 550 °C at a rate of 3 K/min. The space between the block and the casing is filled with a thermal insulating material. A suitable temperature sensor is positioned in the third retort in such away that the sensor tip lies centrally 30 mm above the internal base of the retort. The distance of 30 mm is established by using a graphite pencil (**5.1**) as a means of measurement.

6.4 *Temperature controller*

The temperature controller shall be a separate instrument from that used to record the rise of temperature during the test. It shall be of the automatic, programmed type capable of maintaining a mean rate of temperature rise of 3 K/min ± 0,05 K/min between 250 °C and 550 °C with a variation of not more than ± 1 K per 30 K rise in any 10 min period, with a precision of ± 1 K.

6.5 *Temperature recorder*

A suitable means of producing a complete record of the temperature variation during the test.

6.6 *Retort and piston*

A cylindrical retort of cold-drawn seamless tube of steel 150 M 19 complying with BS 970-1, fitted with a gas-tight threaded plug at its base and a collar at its top. When inserted in a hole in the furnace, the retort shall be supported only by the collar with the threaded plug clear of the bottom of the hole.

When new, the internal diameter of the retort shall be 8,00 mm ± 0,05 mm and the external diameter shall be 14,5 mm ± 0,1 mm. Check the internal diameter with a suitable ball gauge when new, and again after 100, 150, 200, etc., tests. If the internal diameter of the lower third of the retort has increased at any point to more than 8,075 mm, discard the retort.

The piston is machined from rod made of steel 070 M 55 complying with BS 970-1. Adjust the combined mass of the piston and pen assembly to 150 g ± 5 g by machining cut-out portions from the piston. The difference between the diameter of the piston and the internal diameter of the retort shall be 0,2 mm ± 0,05 mm on manufacture. If this difference exceeds 0,275 mm in use the piston shall be replaced. The piston shall slide freely in the retort.

A stand shall be provided to allow the retorts and pistons to cool in a vertical position after removal from the furnace.

6.7 *Means of recording piston movement*

A suitable means of recording piston movement versus time on a chart shall be used. The horizontal scale (time) shall be such that, when converted to temperature (see **7.3.3**), a range of 180 °C will occupy a length of at least 150 mm. On the vertical scale, 5 % expansion or contraction shall occupy at least 3 mm. This may be achieved either by a mechanical pen/chart system or a transducer/electrical signal recorder.

A simple mechanical system is illustrated in Figure 2. In this example two tests are recorded simultaneously on opposite sides of the chart by means of pens clipped firmly to the tops of the pistons. The chart is fixed to a cylinder which is rotated at uniform speed by either a clockwork or a synchronous motor and is mounted on a stand which is clamped to the top of the dilatometer outer casing.

6.8 *Cleaning instruments*

6.8.1 *Auger*, approximately 7,8 mm diameter and stem length 400 mm.

6.8.2 *Wire brush*, 8 mm diameter and stem length 400 mm.

NOTE A wear-resistant steel reamer, 7,95 mm diameter and stem length 400 mm may also be used.

7 Procedure

7.1 Temperature calibration

Carry out the following operations for each position in the furnace, other than the position used for the temperature sensor.

Coat the lower 30 mm of the internal wall and the screw thread and the sealing plug of the retort as well as the lower face of the piston (**6.6**) with a thin layer of blacking (**5.3**) prior to testing in order to prevent the molten reference metals adhering to the steel construction material. Dry by gentle warming. Place a lead ball [**5.2** a)] in the recess at the narrow end of a graphite pencil (**5.1**). Place the pencil in a retort, replace the screw plug and assemble the piston and recording mechanism. Insert the retort assembly into the furnace at a temperature of approximately 280 °C and determine the melting point of the lead using the procedure described in **7.3.3**, replacing the test piece (see **7.2.2**) by a prepared graphite pencil. Repeat this procedure using a zinc ball [**5.2** b)].

Before re-using graphite pencils heat the narrow end of each pencil in a bunsen flame for a few seconds and shake the molten metal from the cylindrical reservoir.

Repeat the calibration after 200 tests or after 3 months' use, whichever occurs first, or if any component is replaced.

If the difference between the standard and indicated temperatures is less than 7 K, establish a factor to correct the indicated temperatures. If the difference is greater than 7 K, check the sensor/indicator system by, for example, direct potentiometric calibration against a standard e.m.f.

7.2 Preparation of test sample and test pieces

7.2.1 ***Test sample***

7.2.1.1 *General*

Two alternative methods of preparing the test sample are described. If the determination is to be carried out immediately after preparation of the test sample, direct size reduction (**7.2.1.2**) may be used. If there is likely to be a delay between size reduction and testing, or if a laboratory sample with an upper particle size of 600 µm is required for other tests, the method described in **7.2.1.3** shall be used. In all cases the production of an excessive amount of fines shall be avoided.

NOTE Low-speed disc mills are suitable for carrying out such reductions.

7.2.1.2 *Direct size reduction*

Air dry the sample of coal and reduce to an upper particle size of 212 µm to yield a 225 g test sample, as described in BS 1017-1, avoiding oxidation. The size distribution of the test sample shall comply with the following:

passing 212 µm test sieve 100 %

passing 125 µm test sieve 70 % to 60 %

passing 63 µm test sieve 40 % to 30 %

Commence the determination as soon as possible after the preparation of the test sample.

7.2.1.3 *Size reduction via a 600 µm laboratory sample*

If coal of a maximum particle size of 600 µm is required for other analyses, air dry the sample and reduce to an upper particle size of 600 µm, avoiding an excessive amount of fines, to yield a 225 g laboratory sample. For preparation of the dilatometer test pieces crush a 20 g sub-sample to yield a test sample with a maximum particle size of 212 µm and a size distribution as in **7.2.1.2**. Commence the final reduction as soon as possible after reduction to 600 µm and the determination as soon as possible after reduction to 212 µm.

7.2.1.4 *Storage of sample*

If necessary, store the test sample in an inert atmosphere in a sealed glass phial.

7.2.2 ***Test piece***

Place 10 g of the test sample (**7.2.1**) in a small glass beaker; add approximately 1 ml of water and mix thoroughly for 2 min to 5 min using a glass stirring rod. The quantity of the water shall be such that the coal just holds together when pressed between the fingers.

Mount the mould (**6.1.1**), with its plug in position and resting on the base holder, on a firm surface. Place approximately 0,5 g of the moistened sample in the mould and place the ram (**6.1.3**) on top of the coal. Consolidate the sample by three or four sharp blows from the mallet (**6.1.2**). Add at least five further increments to the mould and consolidate to fill the barrel of the mould. After the last portion has been inserted and consolidated, compress the test piece further in the screw press (**6.1.4**) by applying continuously a load to a maximum of 15 kN. Release the load as soon as 15 kN is reached.

Remove the base and plug from the mould barrel. Trim the wide end of the test piece free from irregularities. This is conveniently carried out by scraping the end of the test piece using a metal straight-edge of an appropriate width to fit into the recess at the base of the mould barrel. A piece of hacksaw blade suitably ground with a square edge is satisfactory for this purpose.

Expel the formed test piece by suspending the mould on the carrier arm of the press, and screwing the plunger down on to the compressed sample surface. Reduce the length of the test piece to 60 mm ± 0,5 mm by removing material from the narrow end, using, for example a sharp knife, so that the test piece conforms to the length of a gauge 60 mm long (see Figure 1).

Two test pieces are required.

7.3 Determination

7.3.1 ***Number of tests***

Carry out the determination in duplicate using test pieces prepared from the same test sample and tested either in two retorts in the same furnace during a single heating cycle or in the same retort during independent furnace heating cycles.

7.3.2 ***Inspection of apparatus***

It is essential that the test is carried out with the retorts freely suspended in the furnace and with the dilatometer piston and retort scrupulously clean. Clean as described in **7.3.4** after each test.

7.3.3 ***Determination of dilatation***

Place a test piece (**7.2.2**) narrow end uppermost in the dilatometer retort and insert the piston into the retort so that it rests on the test piece.

Stabilize the furnace temperature at 30 °C below the expected softening temperature or, if this is unknown, at the appropriate temperature given in Table 2 or, if the volatile matter content is unknown, at 265 °C.

Table 2 — Furnace temperature

Volatile matter content of coal (dry, ash-free basis) % (*m*/*m*)	Stabilized charging temperature °C
38,1 and above	265
28,1 to 38,0	295
18,1 to 28,0	325
18,0 and below	355

Insert the retort containing the test piece and piston in the appropriate hole in the furnace block and allow 10 min for the system to regain the stabilized charging temperature. Attach the recording mechanism to the piston during this period and adjust at the zero location.

NOTE A slight offset from zero is recommended to facilitate subsequent reading of the chart.

Commence the heating programme and, after a further period of 10 min, start the recorder and index the furnace temperature, as indicated by the temperature recorder, upon the chart.

Conclude the test when no further dilatation has occurred for 5 min or when no dilatation has occurred up to 500 °C. In the 5 min interval following completion of the dilatation process, or at about 500 °C when no dilatation has occurred, make a second indexing of furnace temperature from the temperature recorder on the dilatation chart. If, in the period between the indexed temperatures, the temperature recorder shows a rate of temperature rise which differs from 3 K/min, i.e. 30 °C ± 1 °C in any 10 min interval, the test is not valid.

At the end of the test, detach the recording mechanism and withdraw the retort and piston assembly from the furnace. The piston may be removed from the retort and independently cooled by suspending in air in a suitable stand.

7.3.4 ***Cleaning of the furnace, retort and piston***

Check and clean the equipment at the end of each test as follows.

7.3.4.1 *Furnace*

Check that each retort can be freely suspended from the collar. If not, clean out the holes in the furnace block.

7.3.4.2 *Retort and plug*

Remove the plug, crush the semi-coke and remove as much of it as possible with the auger (**6.8.1**). Complete the cleaning of the retort using the wire brush (**6.8.2**) and a reamer if necessary. Clean the plug end with very fine emery paper, taking care, as far as is possible, not to abrade the metal. Solvents, such as pyridine or dimethylformamide can be used for this purpose, provided all necessary requirements of national health and safety regulations are observed.

7.3.4.3 *Piston*

Clean the piston including its base with wire wool and very fine emery paper taking care not to round the edges. Check that the piston slides freely in the cleaned retort; the required tolerances between the piston and the retort are given in **6.6**.

8 Expression of results

Report the following five basic parameters obtained from the charts after completion of the test (see Figure 3 and Figure 4):

— softening temperature, θ_1 (**3.1**), in degrees Celsius;

— temperature of maximum contraction, θ_2 (**3.2**), in degrees Celsius;

— resolidification temperature, θ_3 (**3.3**), in degrees Celsius;

— maximum contraction, c (**3.4**), as a percentage;

— maximum dilatation, d (**3.5**), as a percentage.

The dilatation is positive if the final line of maximum dilatation is above the zero line [(see Figure 4 a)], and negative if below it [see Figure 4 b)]. If the dilatometer curve does not rise after the initial contraction, the dilatation behaviour is noted as "contraction only" [see Figure 4 c)]. If the final trace of the curve is not truly horizontal but slopes downward [see Figure 4 d)], report the contraction as the value observed at 500 °C.

If the maximum dilatation, d, is greater than 300 %, report as $d > 300$.

Average the results of acceptable duplicate determinations (see **9.1**), and round to the nearest whole number for temperature, contraction and dilatation.

9 Precision

When the method specified in this British Standard is operated satisfactorily the numerical values for repeatability (**3.6**) and reproducibility (**3.7**) shall not exceed those given in Table 3.

Fractional dilatation tolerances shall be rounded to the next higher whole number.

9.1 Repeatability

The results of duplicate determinations carried out in the same laboratory by the same operator using the same dilatometer on test pieces prepared from the same test sample, shall not differ by more than the values shown in Table 3.

9.2 Reproducibility

The means of acceptable duplicate determinations carried out in two different laboratories on test pieces prepared from representative samples, which are taken from the same gross sample after reduction to a maximum coal particle size of 2,8 mm, shall not differ by more than the values shown in Table 3. If differences persistently greater than these limits are observed, the need for further investigation into the methods of sampling and testing is indicated.

Table 3 — Precision

Property	Repeatability	Reproducibility
Temperature parameters	7 K	15 K
Contraction, c	5 units	8 units
Negative dilatation, d	5 units	8 units
Positive dilatation, d	$5\left(1 + \frac{d}{100}\right)$	$5\left(2 + \frac{d}{100}\right)$

10 Test report

The test report shall contain the following information:

a) the reference of the method used;

b) the results and the methods of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this British Standard, or regarded as optional.

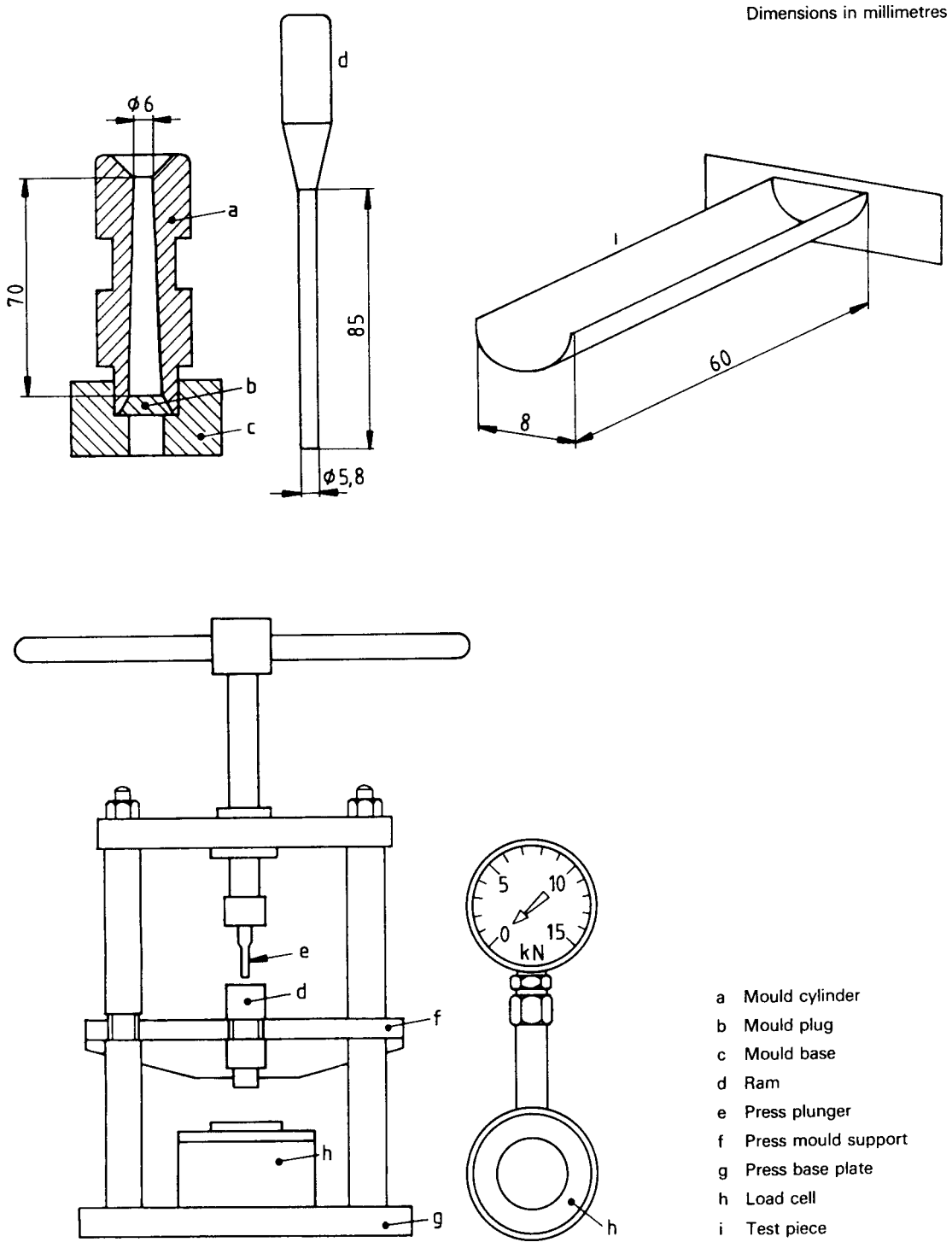

Figure 1 — Mould, press and accessories

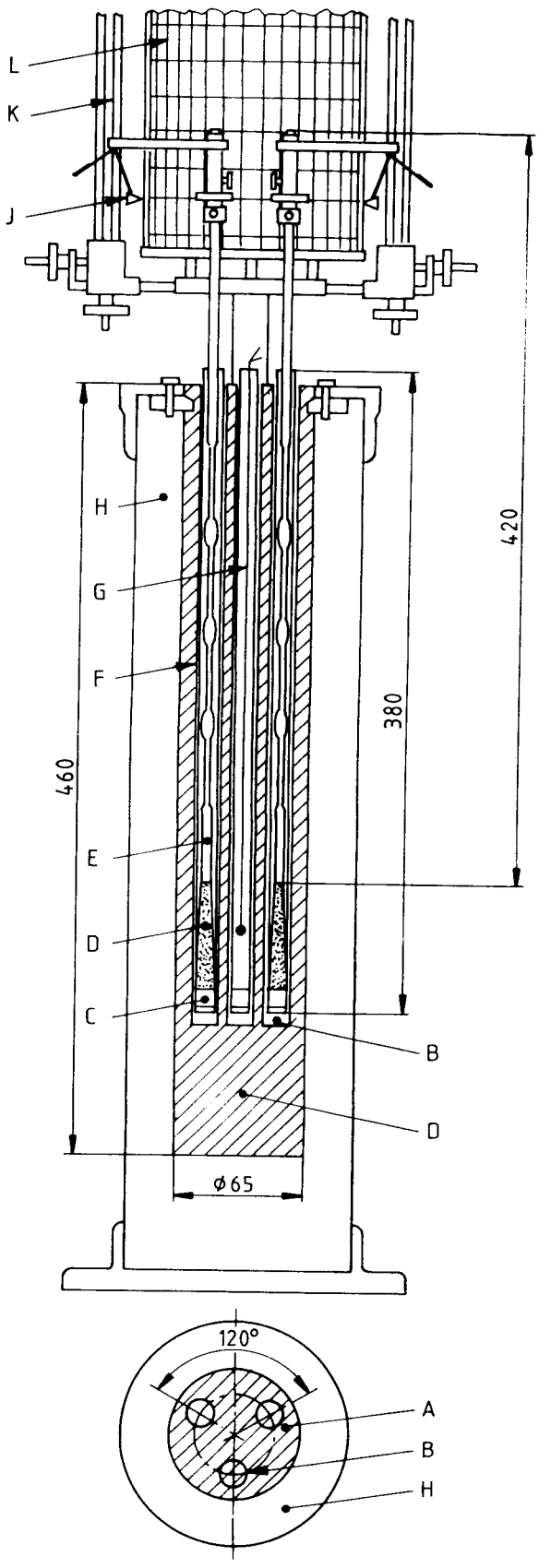

Figure 2 — Typical dilatometer apparatus

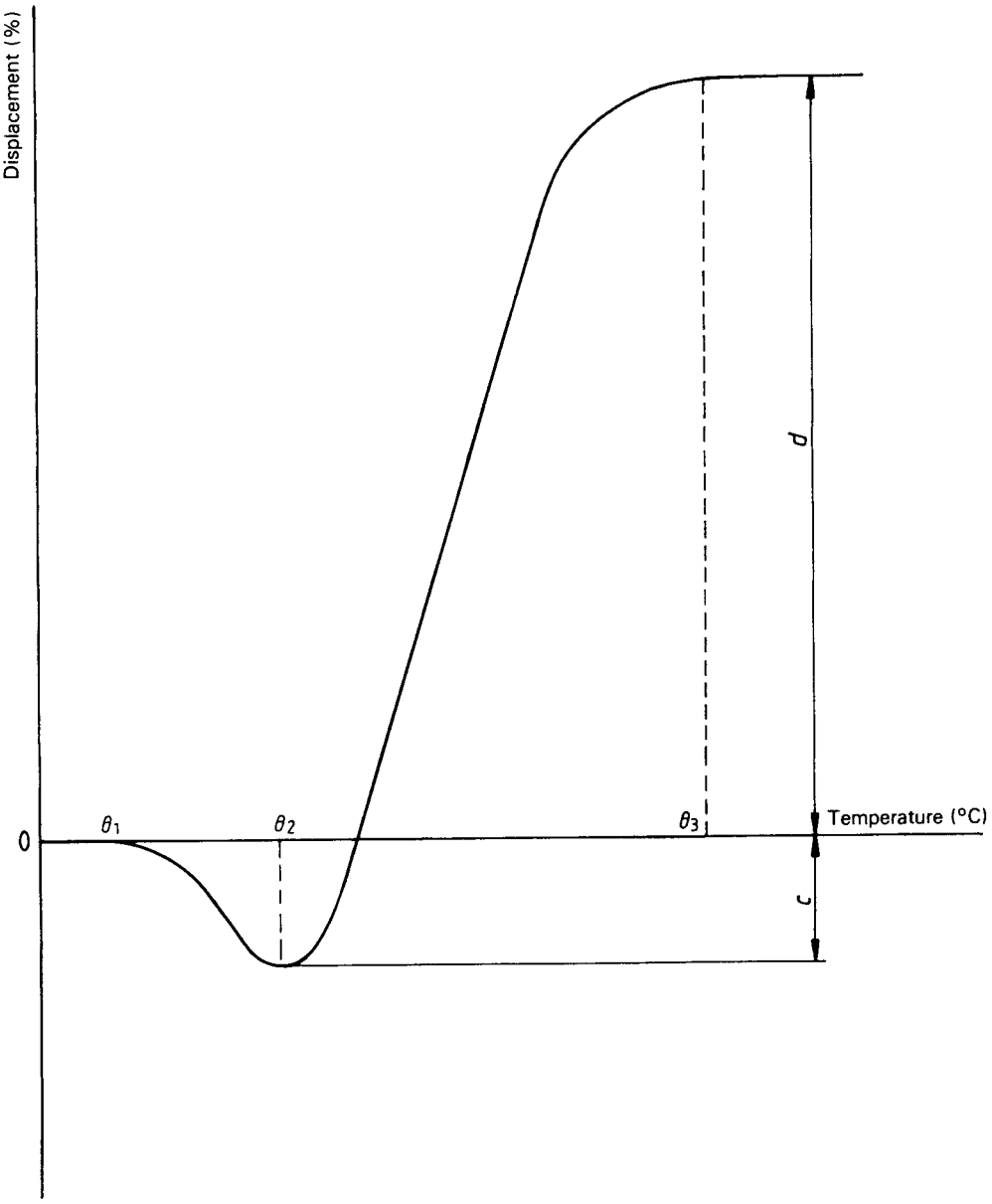

Figure 3 — Characteristic dilatation curve

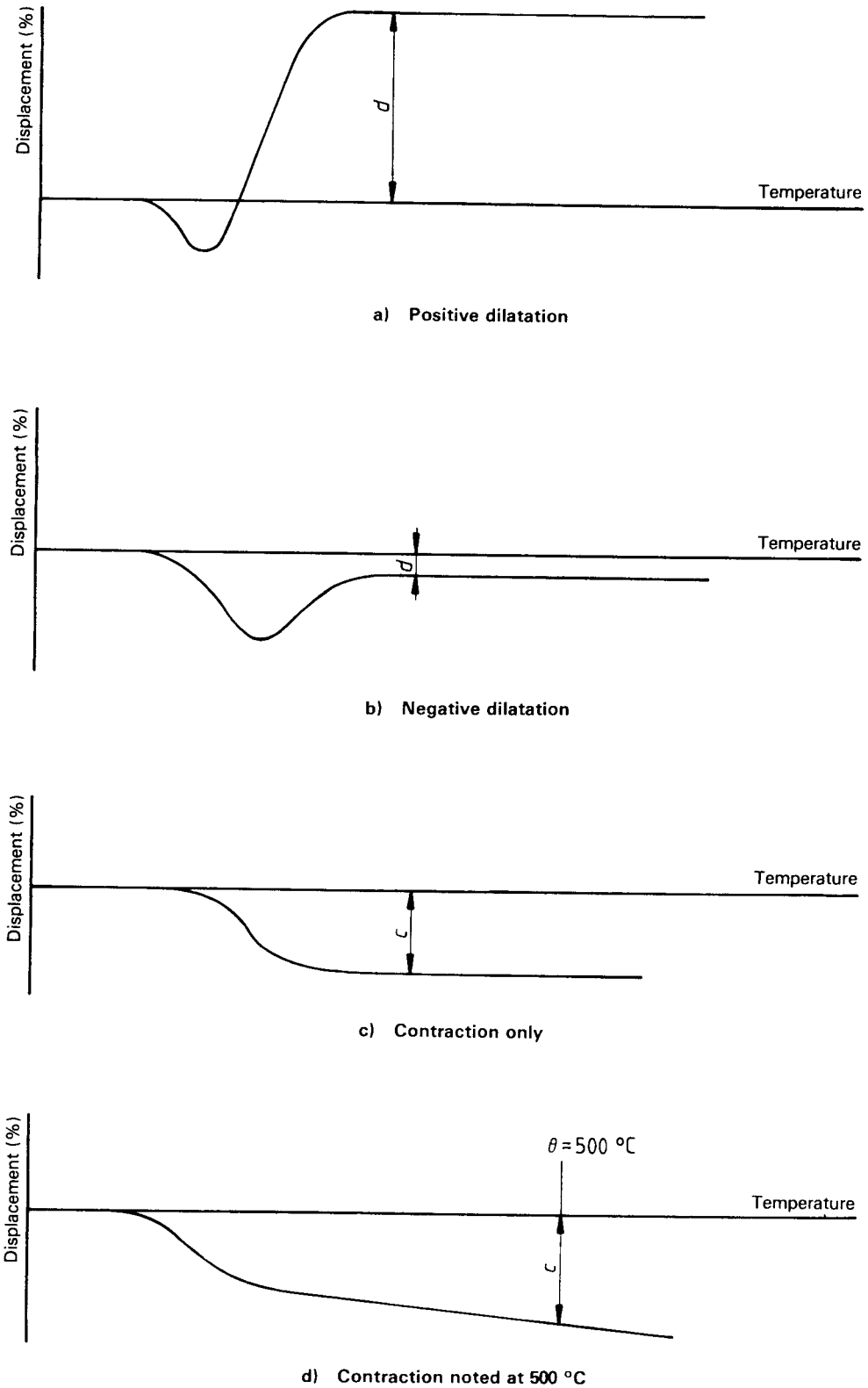

Figure 4 — Types of dilatation curve

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Publications referred to

BS 970, *Specification for wrought steels for mechanical and allied engineering purposes.*

BS 970-1, *General inspection and testing procedures and specific requirements for carbon, carbon manganese, alloy and stainless steels.*

BS 1017, *Sampling of coal and coke.*

BS 1017-1, *Methods for sampling of coal.*

BS 2872, *Specification for copper and copper alloy forging stock and forgings.*

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