



BS 1016 : Part 12 : 1980

UDC [622.66+662.74] : 662.66.016 : 539.388.8

Methods for

Analysis and testing of coal and coke

Part 12. Caking and swelling properties of coal

Méthodes d'analyse et d'essai du charbon et du coke
Partie 12. Pouvoir d'agglomération et gonflant du charbon

Verfahren für die Analyse und Prüfung von Kohle und Koks
Teil 12. Backfähigkeit und Quellbarkeit von Kohle

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BS 1016 : Part 12 : 1980

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Foreword

This British Standard is Part 12 of a series issued under the same number, BS 1016, dealing with methods for the analysis and testing of coal and coke; the other Parts are as follows:

- Part 1 Total moisture of coal
- Part 2 Total moisture of coke
- Part 3 Proximate analysis of coal
- Part 4 Moisture, volatile matter and ash in the analysis sample of coke
- Part 5 Gross calorific value of coal and coke
- Part 6 Ultimate analysis of coal
- Part 7 Ultimate analysis of coke
- Part 8 Chlorine in coal and coke
- Part 9 Phosphorus in coal and coke
- Part 10 Arsenic in coal and coke
- Part 11 Forms of sulphur in coal
- Part 13* Tests special to coke
- Part 14 Analysis of coal ash and coke ash
- Part 15 Fusibility of coal ash and coke ash

- Part 16* Reporting of results
- Part 17 Size analysis of coal
- Part 18† Size analysis of coke
- Part 19† Determination of abrasion index
- Part 20† Determination of the Hardgrove grindability index of hard coal

This revision of Part 12 has been undertaken to bring the presentation into line with earlier Parts: the technical content of the methods for determination of the crucible swelling number and the assessment of caking power (Gray-King coke test) remain unchanged from the previous edition but they have been generally aligned with the technically equivalent international standards ISO 501 and ISO 502. Full-size reproductions of Gray-King coke types and standard profiles are illustrated in figure 7, and replace supplement no. 1 (1961) to BS 1016 : Part 12 : 1959. A new method for measuring the swelling of coal using a dilatometer (dilatometer index) has been added; this has no ISO equivalent.

*A revision is in course of preparation.
†In course of preparation.

British Standard Methods for

Analysis and testing of coal and coke

Part 12. Caking and swelling properties of coal

1. Scope

This Part of BS 1016 specifies methods for determining the swelling properties of a coal when heated in a covered crucible (crucible swelling number), for the assessment of the caking properties of coal (by carbonizing under standard conditions) (Gray-King coke test) and for the measurement of the swelling of coal in a dilatometer.

2. References

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

3. Definitions

For the purposes of this Part of BS 1016 the following definitions apply.

3.1 crucible swelling number. The number which defines, by reference to a series of standard profiles, the size and shape of the residue obtained when a standard mass of coal is heated under the conditions specified.

3.2 Gray-King coke type. The type, denoted by a letter, which defines, by reference to a series of standard profiles, the size and texture of the coke residue obtained when a standard mass of coal is heated in a retort tube to a temperature of 600 °C under the conditions specified. For certain coals, subscripts applied to the letter G represent the minimum proportion, by mass, of electrode carbon required in admixture to obtain a coke residue of profile G.

3.3 softening temperature (temperature of initial contraction). The temperature at which the downward movement of the dilatometer piston is 0.5 mm.

NOTE. See 'T₁' in figure 13.

3.4 temperature of maximum contraction. The temperature at which the dilatometer piston reaches its lowest point.

NOTE. See 'T₂' in figure 13.

3.5 resolidification temperature (temperature of maximum dilatation). The temperature at which the dilatometer piston reaches its highest point.

NOTE. See 'T₃' in figure 13.

3.6 maximum contraction. The maximum downward movement of the dilatometer piston, measured from the zero point and expressed as a percentage of the initial pencil length.

NOTE. See 'c' in figures 13 and 14.

3.7 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 coal pencil length. The value can be either positive or negative (see figure 14).

NOTE. See 'd' in figures 13 and 14.

3.8 parallel dilatometer tests. Tests carried out upon two coal pencils prepared at the same time from a single coal sample, and treated in two retorts in the same furnace during a single heating cycle.

NOTE. This is the preferred procedure.

3.9 duplicate dilatometer tests. Tests carried out by the same operator using two coal pencils prepared from a single coal sample and treated in the same retort in independent furnace heating cycles.

3.10 repeatability. The maximum acceptable difference between two or more determinations, which are carried out simultaneously or at different times in the same laboratory by the same operator with the same apparatus on the same analysis sample.

3.11 reproducibility. The maximum acceptable difference between the means of two or more 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. Introduction

A knowledge of the caking properties of a coal is necessary in determining the use to which it may best be put, but there is no entirely satisfactory caking test which will enable industrial performance to be predicted accurately. While all the tests involve pyrolysis of the coal sample they differ radically in the conditions in which the coal is heated. Thus in the crucible swelling test the coal is shock-heated to above 800 °C at a rate greater than 300 °C/min, whereas in the Gray-King coke type test the coal is raised in temperature from 300 °C to 600 °C at the much slower rate of 5 °C/min. In the dilatometer test the coal is compressed into a pencil before heating at a rate of 3 °C/min.

NOTE. In this standard where the temperature is specified as a definite figure, this figure should be attained as closely as possible and should be subject only to the errors inherent in accurate measurement as defined in BS 1041. Where a range of temperature is given, the temperature may be anywhere in the range without detriment to the result; however the mean of the range should be aimed at in order that the inherent errors in measurement do not cause a temperature outside the specified range to be used inadvertently.

The tests in this Part of BS 1016 measure different phenomena but involve, to a greater or lesser extent, the phenomena of particle adherence (caking), softening, pyrolytic swelling and shrinkage, each of which could be considered as being a fundamental property of the coal itself which affects the formation of a satisfactory coke. Other factors such as the shape of the confining walls, the particle size of the coal and the nature of the surrounding atmosphere can also materially influence the course of caking, swelling and coking, whether it be in

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a laboratory test or in an industrial appliance. For these reasons, it is impossible to be precise regarding the application of the results from these tests to industrial practice, even though they do give a general indication of the caking and swelling properties of a coal.

The Gray-King coke type is used as a parameter in the classification of British coals and in the ECE 'International classification of hard coals by type' published in 1956.

5. Determination of the crucible swelling number

5.1 Principle. A standard mass of coal is heated in a covered crucible under standard conditions to a final temperature of $820 \pm 5^\circ\text{C}$. The coke button obtained is classified by comparison with the outlines of a set of standard profiles. The number of the profile most closely corresponding to the coke button obtained is the crucible swelling number.

Gas or electrical heating may be used but an electrically heated apparatus shall only be used if the results obtained are the same (within $\frac{1}{2}$ unit) as those obtained by the gas heating method.

5.2 Preparation of the sample. The coal used for the determination of the swelling number is the analysis sample ground to pass a 212 μm sieve (see BS 1017 : Part 1). Avoid both very fine grinding and undue exposure of the ground coal to the atmosphere since they are liable to lead to incorrect results. Prepare the coal on the same day that the test is to be carried out. With coals especially liable to oxidation, carry out the test as soon as possible and not more than 2 h after grinding, unless the sample can be stored in an inert atmosphere.

Before commencing the determination, mix the air-dried sample of coal thoroughly for at least 1 min, preferably by mechanical means.

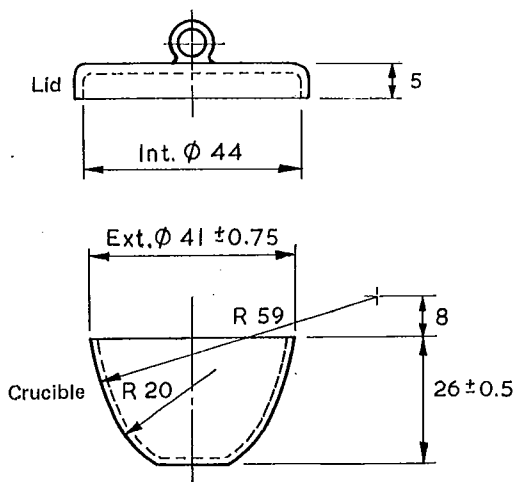
NOTE. The sample may be air-dried at a temperature not exceeding 30°C or dried in an inert atmosphere at a temperature not exceeding 45°C .

5.3 Gas heating method

5.3.1 Apparatus

5.3.1.1 Crucible and lid, (see figure 1) translucent silica crucible, with a flat base, squat form and silica lid with a

Flat top required



Internal diameter of base ≤ 11 .

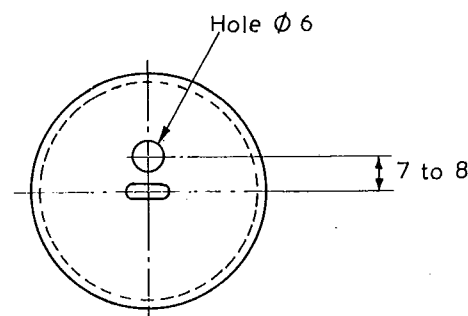
Dimensions are in millimetres

Figure 1. Crucible with lid for swelling test

ring handle. If the lower surface of the crucible lid is not flat, difficulty may be experienced in assessing the swelling number of the coal. A small mica plate shall then be inserted between the lid and crucible before carrying out the test. Crucibles and lids of other ceramic materials may be used provided that the results agree with those obtained with silica crucibles. The crucible shall conform to the following specification:

- external height 26 ± 0.5 mm
- external diameter at top 41 ± 0.75 mm
- minimum internal diameter at base 11 mm
- mass 11 g to 12.75 g
- capacity 16 ml to 17.5 ml

5.3.1.2 Pierced silica lid, (see figure 2) similar to the lid illustrated in figure 1 but with a 6 mm hole to accommodate the thermocouple.



Dimensions are in millimetres

Figure 2. Pierced crucible lid

5.3.1.3 Triangle, consisting of silica tubing of 6 mm to 6.5 mm external diameter mounted on nickel/chromium wire, the length of side being approximately 65 mm.

Unprotected nickel/chromium wire may be used provided it is sufficiently rigid.

5.3.1.4 Burner, capable of giving the required standard heating conditions. The following types have been found satisfactory:

- (a) a Teclu type burner with first family gas of Wobbe number 24.4 MJ/m^3 to 28.8 MJ/m^3 under standard reference conditions*; the internal diameter of the burner tube should be approximately 12.5 mm;
- (b) a Meker type burner with second family gas of Wobbe number 48.2 MJ/m^3 to 53.2 MJ/m^3 under standard reference conditions*; the external diameter of the burner grid should be approximately 30 mm.

5.3.1.5 Gauge, a gas pressure gauge.

5.3.1.6 Draught shield, made from refractory piping approximately 150 mm in length, 100 mm internal diameter and 110 mm external diameter. At one end the piping shall have three slots 25 mm deep in which the wire portions of the silica triangle rest (see figure 3).

5.3.1.7 Thermocouple, of fine wire of diameter not greater than 0.23 mm if made of platinum or 0.45 mm if made of base metal. The ends of the couple shall be in the form of a flattened loop.

5.3.1.8 Weight, 500 g.

5.3.1.9 Viewing tube, cylindrical tube 250 mm in length and 40 mm internal diameter (see figure 4).

*Metric standard reference conditions for gas, 15°C , 1013.25 mbar (1 bar = 10^5 Pa) and dry as defined by the International Gas Union (IGU).

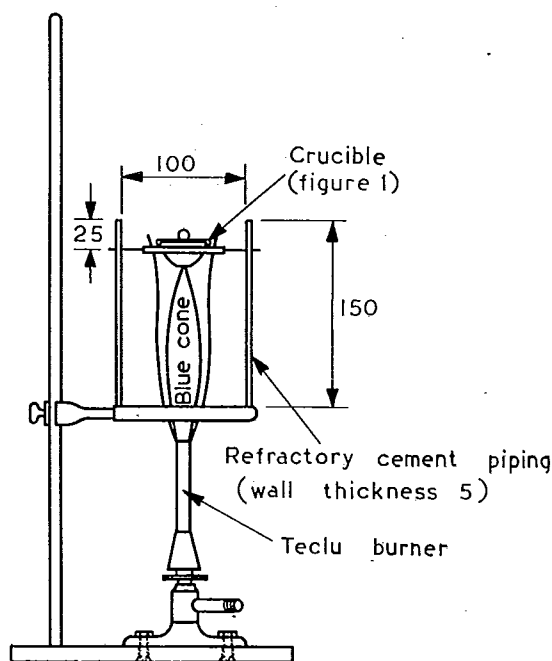
5.3.2 Preparation of apparatus. Assemble the apparatus as shown in figure 3. Place an empty crucible on the silica triangle and support it on the draught shield. Adjust the gas and air supplies to the burner so that the temperature of the inner surface of the base of the crucible reaches $800 \pm 10^\circ\text{C}$ in 1.5 min and $820 \pm 5^\circ\text{C}$ in 2.5 min from first igniting the gas.

If a Tecflu type burner is used it will generally be found that a flame approximately 300 mm long, with the crucible positioned just above the tip of the blue cone, will give the standard temperature conditions.

If a Meker type burner is used, place the crucible approximately 10 mm above the burner grid and adjust the flame so that the standard temperature conditions are attained.

Measure the temperature by means of the thermocouple inserted through the pierced lid and having its unprotected junction and a portion of each wire in contact with the centre of the base of the empty crucible.

Check the apparatus at frequent intervals to ensure that the standard conditions apply.



Dimensions are in millimetres

Figure 3. Apparatus for the gas heating method

5.3.3 Procedure. Weigh 1.00 g to 1.01 g of the freshly ground coal into a dry crucible and lightly tap the crucible about 12 times on the bench to level the surface of the coal. Cover the crucible with the unpierced lid and place it upright on the silica triangle supported in the draught shield. Light the gas and heat at the predetermined rate for such time as is required for the flame of the burning volatile matter to die out and in any case for at least 2.5 min. Turn off the gas and allow the crucible to cool.

Remove the residue carefully from the crucible.

If the residue is non-coherent, i.e. mostly loose powder, no further examination is needed and this result shall be designated swelling number 0.

If the residue is a coherent coke button but is not swollen, place it on a flat surface and rest the 500 g weight on it. If the button supports the 500 g weight without breaking into more than two or three hard pieces, designate it swelling number 1; if it crumbles, or disintegrates, then designate it swelling number ½.

If the coke button is swollen compare it with the standard numbered profiles shown in figure 5. Rotate the button about its axis so that its largest profile is presented for comparison; designate the result by matching the residue to the most similar outline of the swelling numbers given in figure 5.

NOTE. A method of viewing to avoid the effect of parallax is shown in figure 4.

Carry out five tests; after each test burn off the carbon residue and wipe the crucible with a clean cloth.

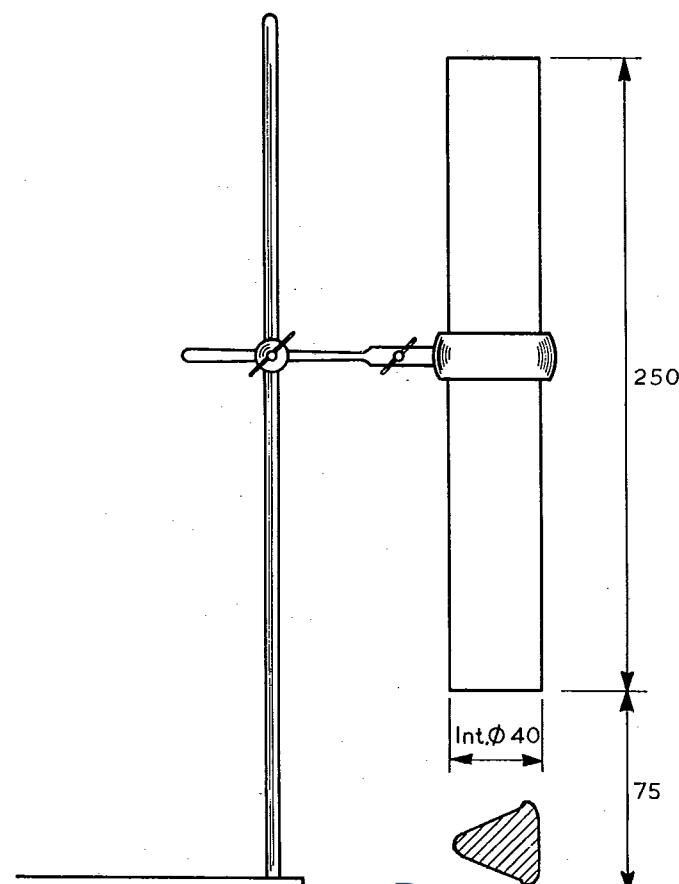
5.4 Electrical heating method

5.4.1 Apparatus

5.4.1.1 Furnace, a suitable type of electrically heated furnace is shown in figure 6, although other types of furnace may be used provided that the results obtained are the same (within ½ unit) as those obtained with the gas heating method.

The furnace illustrated in figure 6 consists of a grooved refractory plate (1), approximately 88 mm in diameter and 13 mm thick, carrying the heating element, which may be a spiralled metallic coil. The plate is supported by a second grooved plate (2). An inverted silica dish (3) 1 mm wall thickness, 10 mm high and approximately 85 mm external diameter, is placed over the windings and acts as a support for the crucible.

The plates are surrounded by a refractory cylinder (4) of approximately 140 mm diameter, bored to a depth of about 60 mm with a 90 mm diameter hole. The cylinder is fitted with a refractory top (5), 20 mm thick; a hole 50 mm diameter is bored in the centre of the refractory top to allow for insertion of the crucible. A refractory lid (8) is



Dimensions are in millimetres

Figure 4. Viewing tube

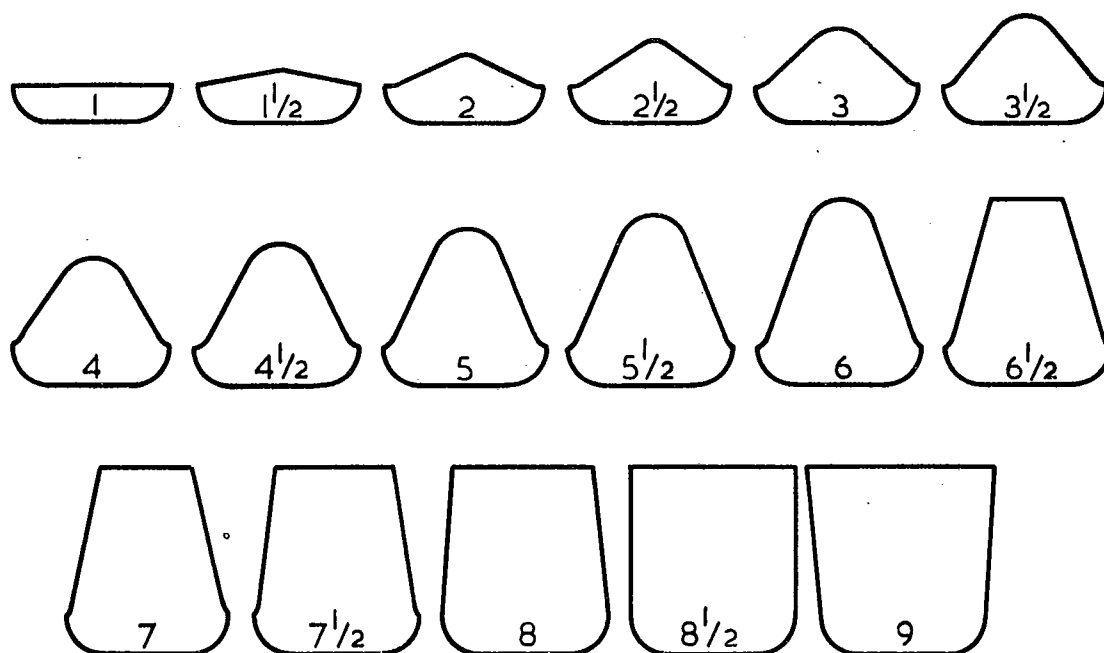


Figure 5. Determination of crucible swelling number: standard profiles and corresponding swelling numbers

used to conserve heat losses from the furnace between determinations. The refractory cylinder rests on a refractory sheet and is surrounded by a 10 mm thick insulation of alumina-silica wool (6). The whole furnace is placed in an aluminium case (7).

A hole bored through the base of the furnace enables a thermocouple to be placed in contact with the underside of the silica dish.

The furnace shall be equipped with a suitable energy regulator and ammeter.

5.4.1.2 Crucible and lid, as specified in 5.3.1.1.

5.4.1.3 Pierced silica lid, as specified in 5.3.1.2.

5.4.1.4 Thermocouples

- (a) a fine wire thermocouple, as specified in 5.3.1.7;
- (b) a suitable base metal thermocouple to measure the temperature of the underside of the silica dish.

5.4.1.5 Weight, 500 g.

5.4.2 Preparation of apparatus. Switch on the furnace and adjust the energy input so that a steady temperature of about 850 °C is maintained at the base of a crucible resting on the silica dish. Remove the crucible and insert a cold crucible, covered with a pierced lid through which passes the fine wire thermocouple held so that its unprotected junction and a portion of each wire rests on the base of the crucible. Ascertain that the standard heating conditions of 800 ± 10 °C in 1.5 min and 820 ± 5 °C in 2.5 min are attained from the time of inserting the crucible. If these conditions are not attained adjust the furnace temperature until the specified conditions are attained. Record the furnace temperature as indicated by the thermocouple at the underside of the silica dish; this temperature then serves as a datum.

5.4.3 Procedure. Weigh 1.00 g to 1.01 g of the freshly ground coal into a dry crucible and lightly tap the crucible about 12 times on the bench to level the surface of the coal. Cover the crucible with the unpierced lid and place it centrally in the furnace on the silica dish. Heat until the volatile matter ceases to be evolved and in any case for at least 2.5 min.

Remove the crucible from the furnace and allow it to cool.

Examine the residue as described in 5.3.3.

Carry out five tests in succession replacing one crucible with the next to avoid heat losses through the top of the furnace. Alternatively, a lid of refractory material (8) as shown in figure 6 may be used to maintain the furnace temperature between tests.

After each test burn off the carbon residue and wipe the crucible with a clean cloth.

5.5 Reporting of results. Record the swelling number of each crucible as follows:

- swelling number 0 : non-coherent residue;
- swelling number ½ : non-swollen coke button that disintegrates under the 500 g weight;
- swelling number 1 : non-swollen coke button that supports the 500 g weight without breaking into more than two or three hard coherent pieces;
- swelling number 1½ to 9 : number of the profile in figure 5 that a swollen coke button most nearly matches.

Report the mean swelling number of the five tests to the nearest ½ unit as the crucible swelling number of the coal sample.

5.6 Precision of the method. When the methods specified in this Part of BS 1016 are operated satisfactorily the numerical values for repeatability and reproducibility (see clause 3) should not exceed those given in the table below; otherwise reference should be made to 4.4 in BS 1016 : Part 16 : 1971.

5.6.1 Repeatability. The range of the results of the five tests carried out in the same laboratory by the same operator with the same apparatus on the same analysis sample shall not exceed the value shown in the table below.

5.6.2 Reproducibility. The means of the five results obtained in each of two different laboratories on representative portions taken from the same sample after the last stage of sample preparation shall not differ by more than the value shown in the table below.

	Repeatability	Reproducibility
Crucible swelling number	½ unit	1 unit

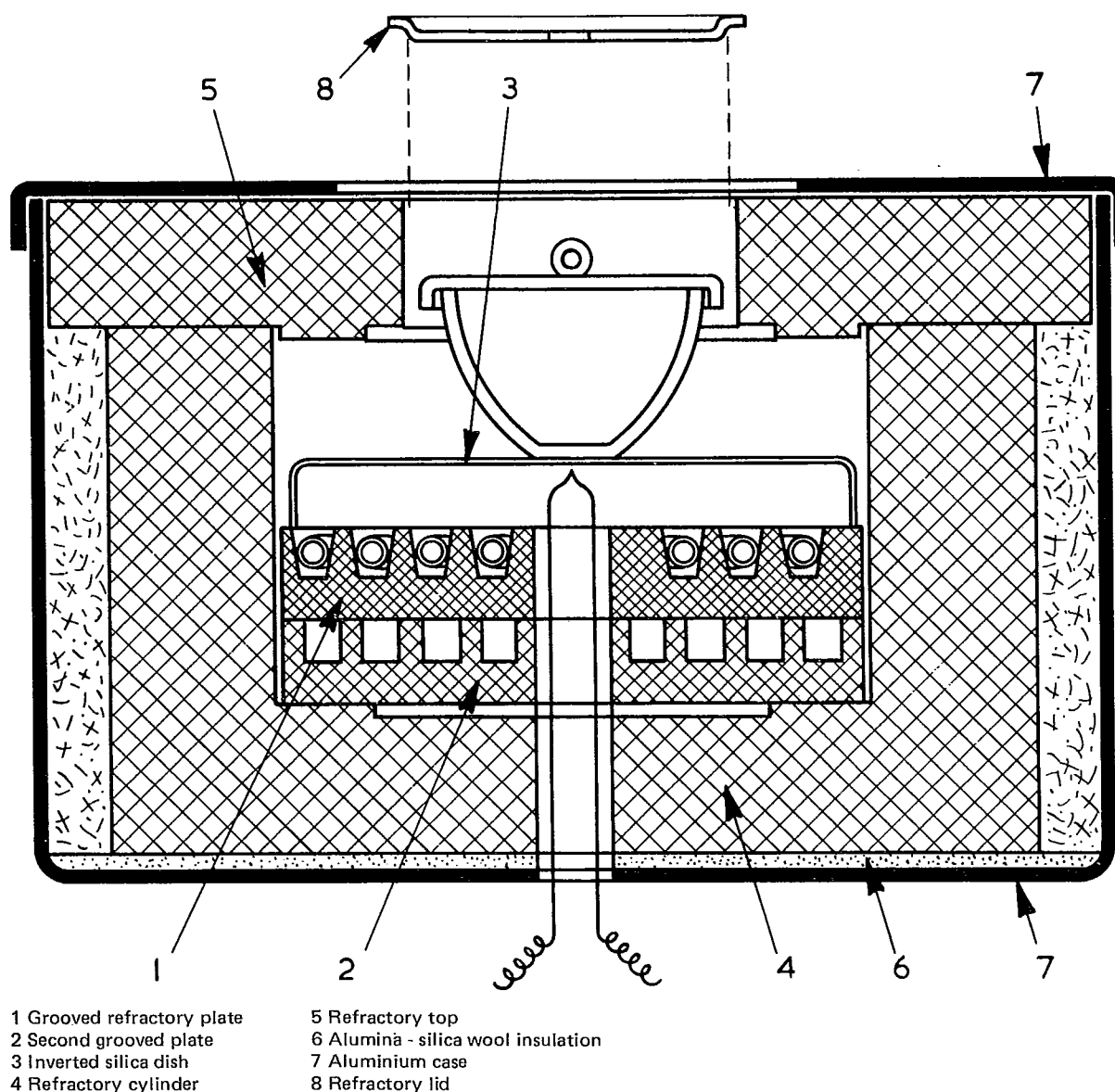


Figure 6. Electrically heated furnace

6. Assessment of caking power : Gray-King coke test

6.1 Introduction. The purpose of the Gray-King coke test, which is one of the parameters adopted for the International Classification of Hard Coal by Type by the United Nations Economic Commission for Europe, is to assess the caking properties of a type of coal or a blend of coals by carbonizing under standard conditions.

6.2 Principle. The coal is heated under standard conditions to a final temperature of 600 °C. The coke residue obtained is classified by reference to a series of standard residues (see figure 7). If the coke residue produced is so swollen that it fills the cross section of the retort tube, the determination is repeated with the coal admixed with a suitable quantity of electrode carbon or equivalent material (see note 1 to 6.3). For these highly swelling coals the Gray-King coke type is defined by the minimum amount of electrode carbon required to produce a strong hard coke residue of the same volume as the original coal and electrode carbon mixture.

6.3 Reagent. The reagent shall be high temperature electrode carbon (or equivalent material, see note 1) complying with the following requirements :

Moisture	less than 1 %
Volatile matter	less than 1.5 %
Ash	less than 5 %

Bulk density at 25 °C
this shall be determined
by the method given in
appendix A

1.00 g/cm³ to 1.05 g/cm³

Relative density at 25 °C 2.05 to 2.09

Size analysis (sieve sizes shown are nominal aperture sizes in accordance with the requirements of BS 410):

Retained on 212 µm test sieve	nil
Through 212 µm test sieve, retained on 125 µm test sieve	less than 20 %