

Methods for

Analysis and testing of coal and coke —

Part 107: Caking and swelling
properties of coal —

Section 107.2 Assessment of caking
power by Gray-King coke test

Committees responsible for this British Standard

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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 United Kingdom
 GAMBICA (BEAMA Ltd)
 Institute of British Foundrymen
 Institute of Petroleum
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Foreword

This Section of BS 1016 has been prepared under the direction of the Solid Mineral Fuels Standards Policy Committee. Part 107 is a revision of the 1980 edition of BS 1016-12, which is superseded and withdrawn. This Section replaces clause 6 in BS 1016-12:1980, from which the principal change is that the standard anthracite is now specified as the only material for mixing with highly swelling coals (the electrode carbon having been omitted).

Part 107 is a further Part numbered 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 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 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 Parts in the new series have been published.

- *Part 104: Proximate analysis;*
- *Part 107: Caking and swelling properties of coal;*
- *Part 108: Tests special to coke;*
- *Part 111: Determination of abrasion index of coal.*

Part 107 is divided into four Sections as follows.

- *Section 107.1: Determination of crucible swelling number;*
- *Section 107.2: Assessment of caking power by Gray-King coke test;*
- *Section 107.3: Determination of swelling properties using a dilatometer;*
- *Section 107.4: Determination of plastic properties using a constant-torque Gieseler plastometer¹⁾.*

This Section is related to ISO 502:1982, published by the International Organization for Standardization (ISO). The principal difference is that ISO 502 specifies standard electrode carbon as the material for mixing with highly swelling coals (as well as allowing the use of equivalent materials).

WARNING NOTE. This British Standard does not necessarily detail all the precautions necessary to comply with the requirements of the Health and Safety at Work etc. Act 1974 or the Control of Substances Hazardous to Health Regulations 1988. Attention should be paid to any appropriate precautions and the method should be operated only by trained personnel.

¹⁾ In preparation.

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.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 8, 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.

1 Scope

This Section of BS 1016 describes a method of assessing the caking power of a coal by determining its Gray-King coke type.

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

2 Definitions

For the purposes of this Section of BS 1016, the definitions given in BS 1017-1 apply, together with the following.

Gray-King coke type

the type, denoted by a letter, with a subscript in certain instances, which defines, by reference to a series of standard profiles, the size and texture of the coke residue obtained when a specified mass of coal is heated in a retort tube under specified conditions

3 Principle

A test portion is heated in a retort tube to a final temperature of 600 °C. The coke residue obtained is classified by comparison with a photograph of a set of standard residues and a table of descriptions.

4 Reagents and materials

4.1 *Liquid paraffin*, laboratory grade, density at 20 °C of 0.83 g/mL to 0.86 g/mL.

4.2 *Standard anthracite*, complying with Table 1²⁾.

5 Apparatus

5.1 *Balance*, capable of weighing to the nearest 0.01 g.

5.2 *Furnace*, horizontal, electrically heated, designed either for separate determinations (single tube furnace) or for simultaneous determinations (multiple tube furnace). A single tube furnace shall have an internal diameter of 50 mm and a length of 300 mm, with one end closed and the other carrying a plug of insulating material which is bored centrally with a hole 25 mm in diameter. The winding of the furnace shall be such that the middle 200 mm is at a uniform temperature within ± 5 °C at both 300 °C and 600 °C.

Alternatively, the furnace may be constructed from an electrically-heated aluminium-bronze block, with one bore (single tube furnace) or several bores (multiple tube furnace) of diameter 25 mm. The furnace shall be thermally insulated and located in a cover of metal or other suitable material, and shall be equipped with a suitable thermocouple, lying above the retort tube, or tubes, when the latter is/are in position and with the junction at the centre of the furnace. An indicator shall be provided for showing the furnace temperature to an accuracy of ± 5 °C. A suitable means of controlling the energy input shall also be provided to achieve an increase in temperature at a rate of 5 K/min. The furnace may be of the fixed type or mounted on rails. Suitable furnaces are shown in Figure 1 and Figure 2.

5.3 *Retort tube* (see Figure 3), of heat-resistant glass or transparent silica, internal diameter 20 mm and length 300 mm, closed at one end, with a side arm, internal diameter 8 mm and length 50 mm, at a distance of 25 mm from the open end. The tube shall be smooth and have either uniform bore or a slight taper (19 mm to 21 mm), such that the open end is the larger.

5.4 *Positioning rod*, with a flat disc, of diameter 19 mm, at one end to assist in the packing of the coal and marked to indicate the correct position of the free end of the test portion in the retort tube.

5.5 Means of disposing of the gas and tar

NOTE For a single tube furnace, this may be a glass vessel of adequate size, suitably supported and attached to the side arm of the retort tube, fitted with an outlet tube leading to the atmosphere or to a piece of small bore silica tubing at the end of which the gas leaving the receiver can be burned or conducted to a fume extraction system. The receiver may conveniently be a U-tube which can be immersed in water.

For a multiple tube furnace, the side arms of the retort tubes may be connected to a manifold and the tar collected in bulk, the crude gas being burnt in a mains gas flame (see Figure 2).

²⁾ For information on the availability of a suitable material, apply to Customer Information, BSI, Linford Wood, Milton Keynes MK14 6LE.

Table 1 — Standard anthracite

Property	Test method	Requirement
Ash	BS 1016-104.4	lower than 5
Volatile matter content	BS 1016-104.3	lower than 8
Size distribution	BS 1016-17	
Passing 212 μm test sieve		100
Passing 125 μm test sieve		90 to 95
Passing 63 μm test sieve		65 to 75

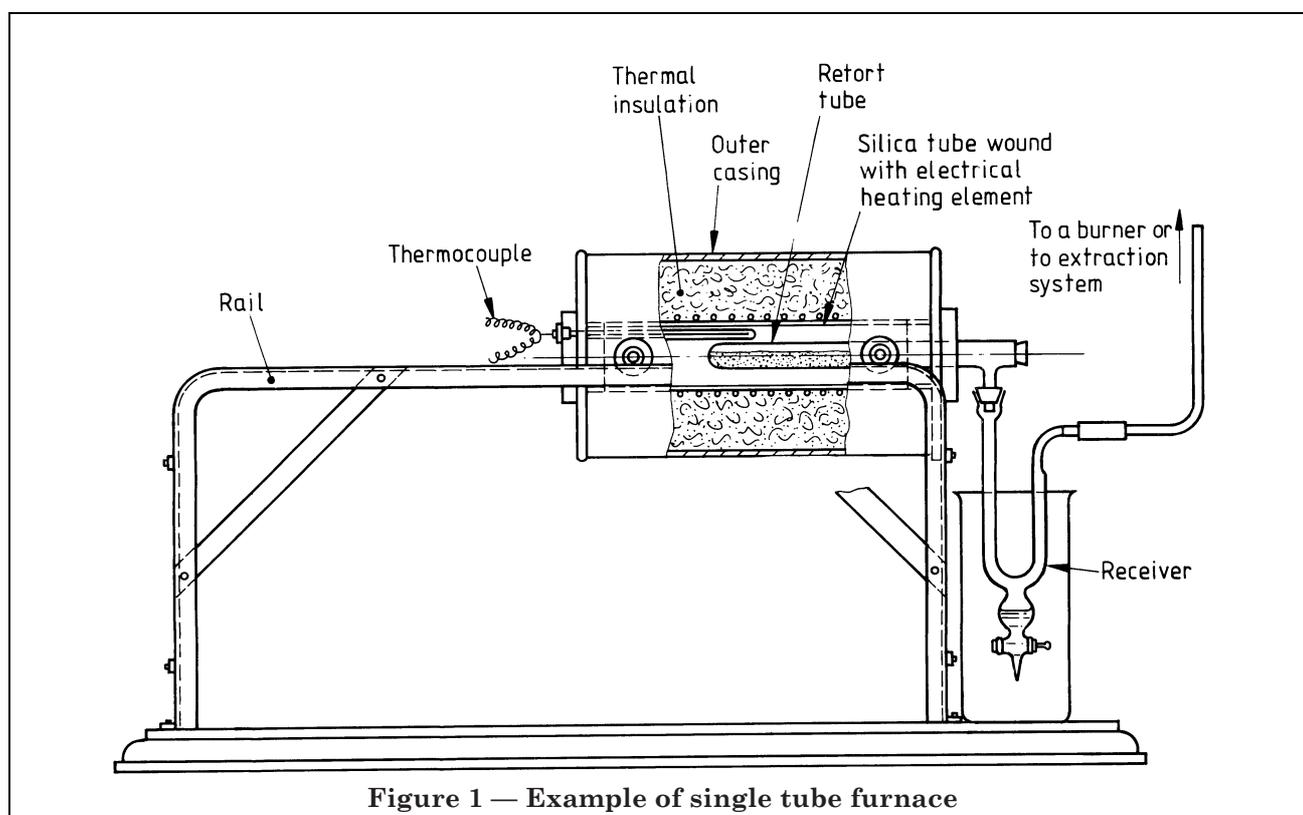


Figure 1 — Example of single tube furnace

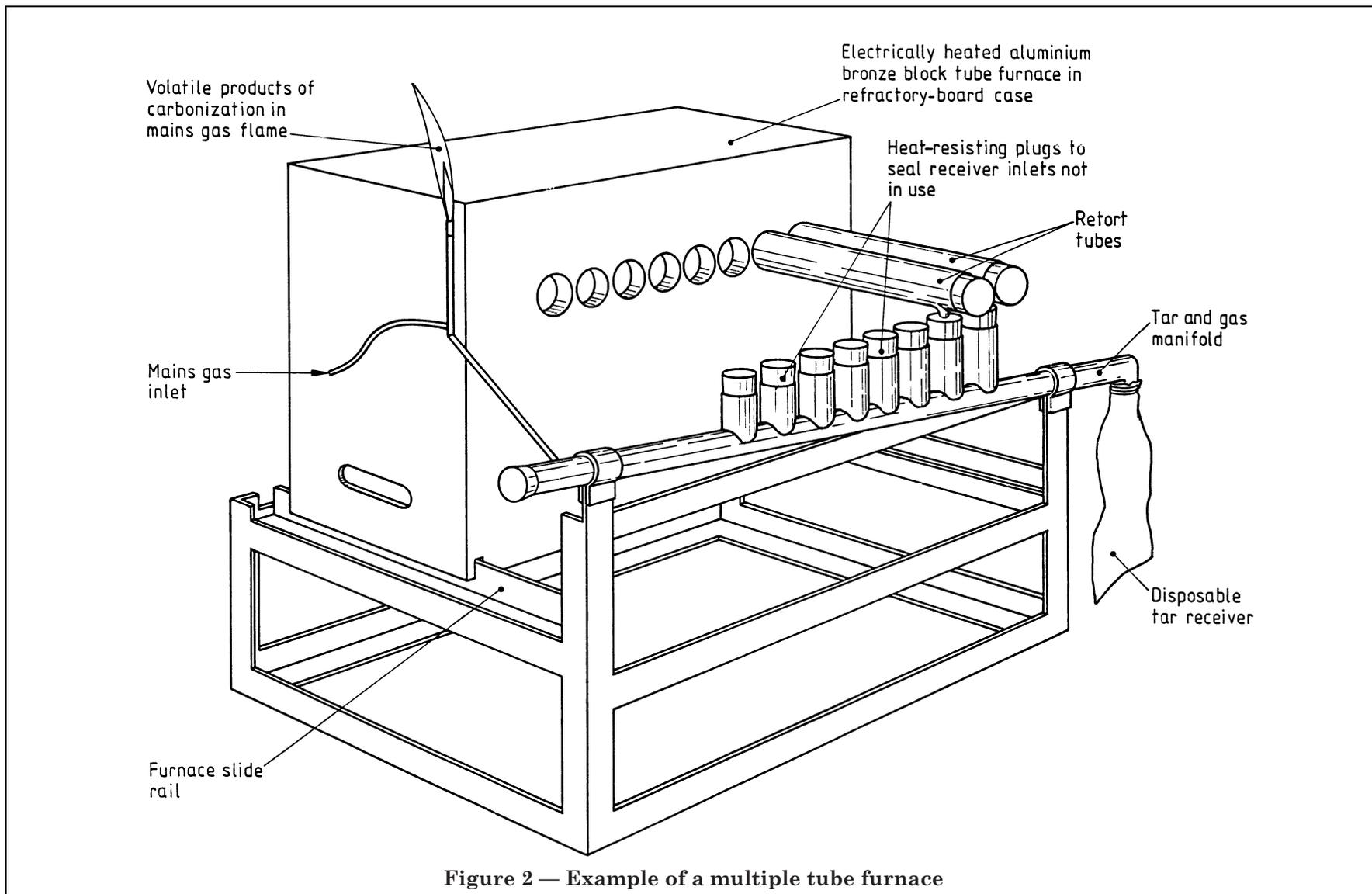


Figure 2 — Example of a multiple tube furnace

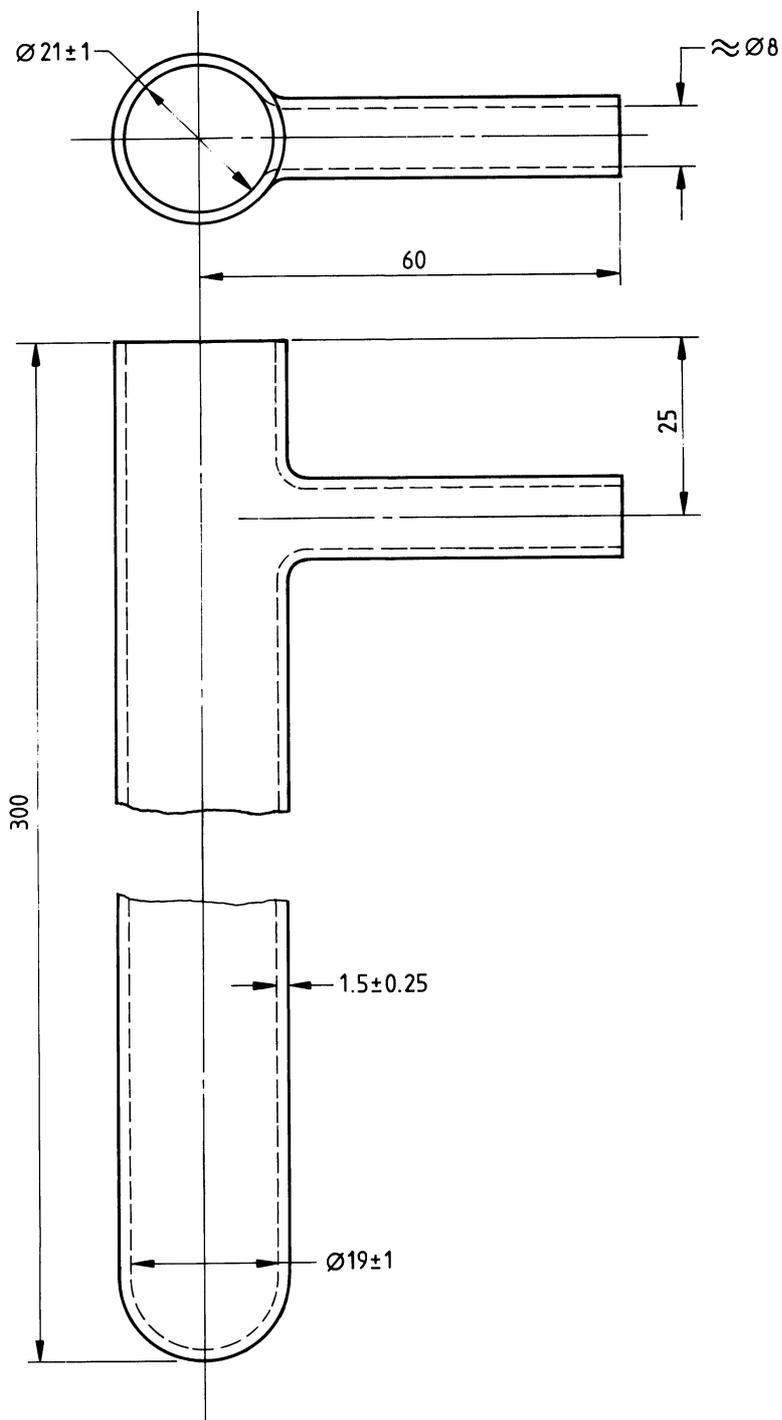


Figure 3 — Retort tube

6 Preparation of test sample

The coal used for the determination of the Gray-King coke type is the general analysis sample, ground to pass a sieve of 212 μm aperture (see clause 22 of BS 1017-1:1989). Expose the sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere. Prepare the test sample on the same day that the test is carried out.

Immediately before weighing out the test portion (see 7.2 or 7.6), mix the test sample for at least 1 min, preferably by mechanical means.

7 Procedure

7.1 General

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 standard anthracite. For these highly swelling coals the Gray-King coke type (designated G_x) is defined by the minimum amount of standard anthracite (x g) required to produce a strong hard coke residue of the same volume as the original coal and standard anthracite mixture.

NOTE Although there is no precise relationship between the crucible swelling number, determined by the method described in BS 1016-107.1, and the Gray-King coke type, Table 2 shows the broad relationship to be expected. This will be found useful for indicating the necessity for blending with standard anthracite and the probable amount required. The above relationship is known to be applicable to United Kingdom coals and is intended only as a general guide.

Table 2 — Relationship between crucible swelling number and Gray-King coke type

Crucible swelling number	Gray-King coke type
0 to 0.5	A to B
1 to 4	C to G_2
4.5 to 6	F to G_4
6.5 to 8	G_3 to G_9
8.5 to 9	G_7 or above

Raise the temperature of the furnace until it is steady at 325 ± 5 °C. Then proceed as described in 7.2 to 7.5 or, if the coal is expected to produce a swollen, hard and strong coke, as described in 7.6.

7.2 Test portion

Using the balance (5.1) weigh, to the nearest 0.01 g, 20.00 g to 20.01 g of the test sample and transfer it to the retort tube (5.3), held in such a manner that the coal cannot enter the side arm, allowing the coal to fall to the far end of the retort tube. Hold the tube horizontally with the side arm vertically downwards, insert the positioning rod (5.4) so that the face of the disc is 150 mm from the closed end of the retort tube and spread the coal into a layer of uniform depth by shaking and turning. Withdraw the positioning rod and insert a flattened pad of heat-resistant material to retain the coal in position. Without disturbing the position of the coal, close the open end of the retort tube with a heat-resisting stopper.

7.3 Preparation of the coke

Insert the retort tube containing the test portion (see 7.2) in position in the furnace (5.2), so that the centre of the coal layer coincides with the centre of the furnace, with the side arm connected to the means of disposing of the gas and tar (5.5). If the furnace is mounted on rails, clamp the retort tube in a horizontal position and run the furnace into position.

Raise the energy input to the furnace in such a manner that the temperature of 325 ± 5 °C is regained in 3 min to 7 min and maintain a uniform rate of rise of 5 K/min thereafter until a temperature of 600 ± 5 °C is reached. Maintain this temperature for 15 min.

Withdraw the retort tube (or retract the furnace) and allow it to cool. Detach the side arm connection, remove the stopper and slide the coke residue out for examination.

7.4 Visual assessment of coke type

Determine the coke type by comparison with the standard residues in Figure 4 and by reference to the descriptions in Table 3. If necessary, determine the volume of the coke as described in 7.5.

If the coke is assessed as being type G_x (types G_1 and G_2 are shown in Figure 4 as examples), proceed as described in 7.6.

Table 3 — Identification of Gray-King coke type

A, B and C			D, E and F			G	G _x
retains initial cross section			shrunken			retains initial volume	swollen
Examine for strength			Examine for strength			Examine for strength	Examine for strength
non-coherent	barely coherent	coherent	moderately hard and shrunken	hard and very shrunken	hard, strong and shrunken	hard and strong	hard and strong
Usually in powder form but may contain some pieces which, however, cannot be handled without breaking	In several pieces and some loose powder. Pieces can be picked up but break into powder on handling	Usually in one piece but easily broken; may be in two or three pieces with practically no loose powder; very friable and dull	May be fissured but can be scratched with fingernail and stains the fingers on rubbing the curved surface vigorously; usually dull and black and appearing fritted rather than fused	Volume below 16.0 cm ³ . Usually very fissured; moderate metallic ring; does not stain the fingers on rubbing; grey or black with slight lustre	Volume within the range 16.0 cm ³ to 22.9 cm ³ . May be fissured; moderate metallic ring; does not stain the fingers on rubbing. Cross section well fused and greyish	Volume within the range 23.0 cm ³ to 25.9 cm ³ . Well fused with a good metallic ring when tapped on a hard wooden surface	Volume greater than 26.0 cm ³ . Well fused with a good metallic ring when tapped on a hard wooden surface
A	B	C	D	E	F	G	G _x

7.5 Measurement of coke volume

If the coke is not in one piece, glue it together using a suitable adhesive.

Completely immerse the coke in the liquid paraffin (4.1) in a glass tube, 180 mm to 200 mm long, for 15 min.

NOTE 1 For swollen cokes that float, it is convenient to hold the coke below the surface of the paraffin using a piece of wire fixed into a cork pushed into the top of the tube.

After immersion, remove the coke and allow it to drain for 30 min whilst lying in a horizontal position and supported on two narrow strips of wood or equivalent material.

After draining, completely immerse the coke in water contained in a tube fitted with a side arm. Collect the displaced water and determine its volume, and hence that of the coke, to the nearest 0.1 cm³.

NOTE 2 The volume of the displaced water can be determined conveniently by weighing it and then calculating the volume from its density.

7.6 Examination of coals which produce swollen, hard and strong cokes

Using the balance (5.1) weigh, to the nearest 0.01 g, y g of the standard anthracite (4.2), where y is an integer (being an estimate of the subscript of the coke type of the coal under test, see 7.1), into a weighing bottle and add $(20.00 - y)$ g, weighed to the nearest 0.01 g, of the test sample. Insert the stopper and mix the contents thoroughly.

Treating the mixture as the test portion, transfer it to the retort tube and proceed exactly as described in 7.2 and 7.3.

Determine the coke type as described in 7.4.

If the coke is assessed as being type A to G, repeat the process using 1 g less of the standard anthracite, and 1 g more of the test sample, until a swollen coke (i.e. type G₁) is obtained. Record the result as type G _{x} , where x is the number of grams of standard anthracite used for the penultimate determination in this series.

If the first coke is swollen, repeat the process using 1 g more of the standard anthracite, and 1 g less of the test sample, until a type G coke is obtained. Record the result as type G _{x} , where x is the number of grams of standard anthracite used for the final determination in this series.

8 Expression of results

Express the result as the Gray-King coke type of the original coal, including the subscript number if any.

9 Precision

9.1 Repeatability

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator using the same apparatus on representative portions taken from the same test sample, should not differ by more than the value given in Table 4.

Table 4 — Precision

Type of coke	Repeatability	Reproducibility
A to G	One letter	One letter
G _{x}	One unit in the subscript	One unit in the subscript

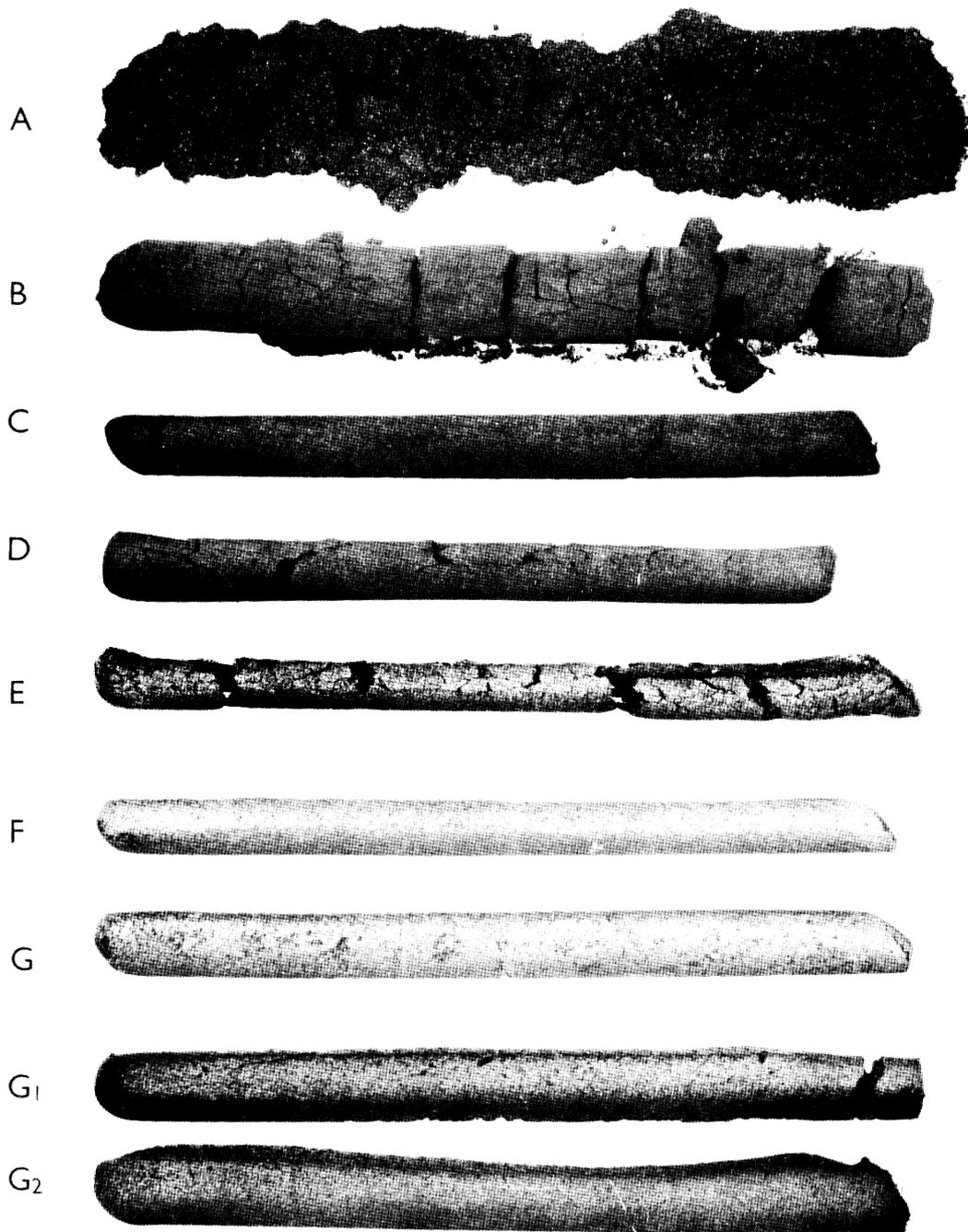
9.2 Reproducibility

The means of the results of duplicate determinations, carried out in each of two different laboratories on representative portions taken from the same sample after the final stage of sample preparation, should not differ by more than the value given in Table 4.

10 Test report

The test report shall include the following:

- a complete identification of the sample;
- the reference to the method used, i.e. BS 1016-107.2:1991;
- the results expressed in accordance with clause 8;
- any unusual features noted during the determination;
- any operations not included in this standard or regarded as optional.



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Figure 4 — Gray-King coke type

Publication(s) referred to

BS 1016, *Methods for analysis and testing of coal and coke.*

BS 1016-17, *Size analysis of coal.*

BS 1016-104, *Proximate analysis.*

BS 1016-104.3, *Determination of volatile matter content.*

BS 1016-104.4, *Determination of ash.*

BS 1016-107, *Caking and swelling properties of coal.*

BS 1016-107.1, *Determination of crucible swelling number.*

BS 1017, *Sampling of coal and coke.*

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

ISO 502, *Coal — Determination of caking power — Gray-King coke test³⁾.*

³⁾ Referred to in the foreword only.

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