IS 1353 (1993): Methods of test for coal carbonization - caking index, swelling and Grey-King assay (L.T) coke types [PCD 7: Solid Mineral Fuels]

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(Reaffirmed 2000)

भारतीय मानक

कोयले के कार्बनीकरण की परीक्षण पद्धतियाँ — पिंड बनने का सूचकांक, फुल्लन अंक (एल टी) ग्रे किंग विश्लेषण

(पहला पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR COAL CARBONIZATION — CAKING INDEX, SWELLING NUMBER AND (LT) GRAY-KING ASSAY

(First Revision)

UDC 662·66 : 662·8·057·1

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 8

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Solid Mineral Fuels Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first issued in the year 1959 when it was prepared on the basis of limited investigational data available with Central Fuel Research Institute, Regional Coal Survey Stations and other centres of investigation and research in those days. The Committee at that time could derive benefit of studying various documents circulated by International Organization for Standardization (ISO) which were also in the formative stages. The Committee, responsible for the preparation of this standard had, therefore, envisaged to update the same as and when more data are made available.

In the present version use of transparent borosilicate glass retorts in addition to those of silica retorts has been prescribed on the basis of investigational work done and data made available. Besides, a simplified yet reliable method for determination of bulk density of finely crushed electrode carbon/coke breeze has been incorporated in place of the former cumbersome method. The details of various test methods have also been suitably updated.

In the preparation of this standard considerable assistance has been drawn from the following documents issued by the Technical Committee, namely, ISO/TC 27 — Solid Mineral Fuels of International Organization for Standardization (ISO):

i) ISO 501 : 1981 Coal — Determination of the crucible swelling number, and

ii) ISO 502 : 1982 Coal — Determination of caking power Gray-King coke test.

Owing to the varying characteristics of coal, tests prescribed in this standard are empirical and valid only if the conditions of test are rigidly observed.

For the purpose of deciding whether test results comply with the requirements of different standards of quality, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. Number of significant places retained in the rounded off value should be the same as that of the specified value in the appropriate standard.

Indian Standard

METHODS OF TEST FOR COAL CARBONIZATION — CAKING INDEX, SWELLING NUMBER AND (LT) GRAY-KING ASSAY

(First Revision)

1 SCOPE

This standard prescribes the methods of test for coal carbonization, namely, agglutinating or caking index, swelling number and low temperature (LT) Gray-King assay.

2 REFERENCES

Following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
436 (Part 1/ Sec 1) : 1964	Methods of sampling of coal and coke: Part 1 Sampling of coal, Section 1 Manual sampling (*first revision*)
1350 (Part 4/ Sec 2) : 1975	Methods of test for coal and coke : Part 4 Ultimate analysis, Section 2 Determination of nitrogen (*first revision*)

3 TERMINOLOGY

For the purpose of this standard the following definitions shall apply.

3.1 Agglutinating Index (Caking Index)

Maximum whole number ratio of sand to coal in a mixture of standard sand and coal which, after heating under specified conditions, gives a coherent mass, capable of supporting a 500 g mass and generating less than 5 percent of loose powder. It is necessary that specially prepared angular sand of specific graded size and quality should be used.

3.2 Crucible Swelling Number

Size index of the coke button produced when 1 g of coal is heated under specified conditions, as compared with a set of standard profiles.

3.3 Low Temperature (LT) Gray-King Assay

Size and nature of coke residue and also the yields of coke and various byproducts namely, tar, liquor, ammonia and gas produced by carbonizing 20 g of coal at 600°C in a transparent borosilicate glass or silica retort under specified conditions.

4 SAMPLING

4.1 Methods of Sampling

Methods of sampling shall be as prescribed in IS 436 (Part 1/Sec 1) : 1964.

5 CAKING AND SWELLING PROPERTIES OF COAL

5.1 The caking power of coal is the property or ability of coal to form a coherent mass when the finely powdered coal is heated under specified conditions in the laboratory. A number of methods have been evolved for the measurement of this property, but the evaluation of the results of those has differed because the property measured had not always been exactly the same. It is a matter of experience that for laboratory purposes, the measurement of one or other of these properties gives a fairly reliable indication of the coking class into which a given coal falls. Three methods are in use, namely:

a) Sand Method for determining Agglutination Index,
b) Crucible Swelling Number Test, and
c) Gray-King Assay (LT) Coke Type.

The sand method for determining stickiness or agglutinating power has gained general acceptance in India but there are many alternatives for the determination of caking power either quantitatively or qualitatively.

6 AGGLUTINATING INDEX OR CAKING INDEX

6.1 Outline of the Method

From a series of tests, the maximum ratio of sand to coal is obtained in a coal sand mixture which, after carbonization under specified conditions, gives a coherent mass capable of supporting a 500 g mass, at the same time the proportion of loose powder being less than 5 percent of the mass of sand and coal. This is the agglutinating index or caking index. It is understood that this method provides only an approximate measure of the tendency of a coal to agglutinate or to produce a lump from powder. To ensure reliable comparison, it is necessary that comparative tests between different operators should be carried out with supplies from the same batch of sand. In all cases the batch number should be stated.

6.2 Apparatus

a) *Weighing bottle* — stoppered, cylindrical, approximately 75 mm in height and 25 mm in diameter.

b) *Silica crucible* — translucent, with lid, of even taper, the inner surface being free from roughness. The crucible shall conform to the following dimensions:

Internal diameter at the top of crucible	38 ± 1 mm
External diameter at bottom	26 ± 1 mm
Thickness of walls:	
At top of crucible	2.25 ± 0.50 mm
At bottom	1.25 to 1.5 mm
Height of crucible	42 ± 0.75 mm
Radius of curvature, rounded edge of crucible bottom	3.5 mm
Minimum width of lid	46 mm
Maximum width, including extension to facilitate handling	60 to 62.5 mm
Thickness of lid	About 1.5 mm
Diameter of recessed part of lid	36 mm
Depth of recessed part of lid	3 to 4 mm

c) *Spatula* — made from sheet metal 0.7 mm (or 22 SWG)

d) *Silica triangle support* — of such dimensions that the crucible [*see* **6.2** (b)] is held upright with the base 10 mm above the level of the bench.

e) *Glazed paper*

f) *Muffle furnace* — gas or electrically heated, capable of maintaining a steady temperature of 900 ± 15°C, provided with a closely fitting door. The heat reserve of this furnace should be such that the temperature is regained before the end of the seven minutes heating period of the test. The temperature should be determined by means of a suitable insulated thermocouple and millivoltmeter, the hot junction of the former being placed away from the floor or sides of the furnace but in the position to be occupied subsequently by the crucible.

g) *Rubber bung* — solid, conforming to the following dimensions:

Diameter:	
Narrow end	35 mm
Wide end	41 mm
Height	32 mm

6.3 Sand

It is extremely important that the sand used in this test should be of uniform quality with regard to size, purity, sharpness, etc. The standard silica sand is not soluble in hot dilute hydrochloric acid to a greater extent than 0.5 percent. It consists mainly of angular particles of pure silica, and is free from impurities, such as clay, chalk, or iron carbonate. It is graded to pass IS Sieve 30 (296 microns) and to be retained on IS Sieve 20 (211 microns) containing not more than 5 percent of oversize and not more than 10 percent of undersize, the oversize and undersize material being not appreciably different from the specified screen sizes. The sand shall not break down on heating for three hours at 920°C to such an extent that the percentage of undersize material is increased by 2.5. Sand of this quality is obtainable from the Central Fuel Research Institute F.R.I., Bihar, India.

6.4 Procedure

a) Air-dry the coal and grind it so that the material just passes through IS Sieve 20 (211 micron). In preparing the coal to this size, it is essential that very fine grinding should be avoided since the results obtained are to some extent dependent upon the amount of very fine material in the sample.

b) Weigh into the weighing bottle, the requisite amounts of the coal and the standard sand to give 25 g of the mixture, containing the two ingredients in the desired proportions. Mix the sand and coal by rotating the tube between the finger and thumb, until the mixture is of uniform appearance to the eye. Pour the mixture into the silica crucible, the inner surface of which having been covered with a layer of graphitic carbon from a previous test.

c) As some segregation of the ingredients may occur during the transfer to the crucible complete the mixing by rotating the crucible, resting on its base, with the left hand in a counter-clockwise direction. At the same time, hold the spatula with the narrower end in the right hand slightly inclined to the vertical away from the operator with the blade of the spatula facing the direction of rotation of the crucible, and with its broader end immersed in the mixture. Repeatedly raise and lower the spatula while the crucible is rotated, continue the mixing for two minutes, and withdraw the spatula. Level the surface of the mixture by pressing gently with the narrower end of the rubber bung, care being taken during this operation that the crucible is not jarred or the contents tamped.

d) Cover the crucible with its lid and transfer to the silica triangle support. Place the crucible and the support, in an electric or gas-fired muffle, previously raised to a steady temperature of 900°C ± 15°C and provided

with a closely fitting door. Replace the muffle door. At the end of seven minutes withdraw the crucible and the support from the furnace and allow to cool slowly to room temperature while standing on an asbestos board. At the end of 30 to 40 minutes remove the crucible lid, withdraw the crucible from the support and place it on its base on a sheet of glazed paper.

e) Place the rubber bung with the narrower end resting on the surface of the contents of the crucible. Invert the crucible and rubber bung by holding the rim of the crucible and the rubber bung, on opposite sides, between the thumb and second finger of one hand. Carry out this operation over the sheet of glazed paper so that any particles falling from the crucible and adhering to the finger and thumb may be collected. Raise the crucible vertically away from the carbonized residue of sand and coal. Lift the residual cake from the bung, holding it gently between the finger and thumb (care being taken to produce no powder) and place it with its broad end on a porcelain tile. Gently lower a 500 g mass until its base rests upon the carbonized residue and not whether the mass is supported without crumbling. Weigh the loose powder on the glazed paper, together with that from the top of the bung.

f) After the test, the inner side of the crucible will be found to be covered with a bright layer of carbon. This should be retained from test to test and should not be burnt off.

g) Repeat the test with increasing ratios of sand to coal, the mass of the total mixture always being 25 g and report as agglutinating index the maximum ratio which supports a mass of 500 g with the proportion of loose powder being less than 5 percent of the mass of the mixture.

7 CRUCIBLE SWELLING NUMBER

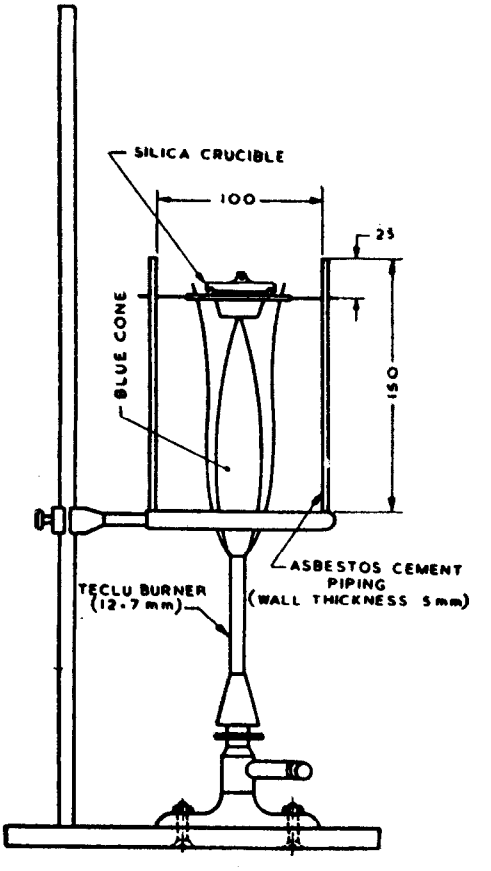
7.1 General

The swelling number of coal, as determined by the crucible swelling number test, described below, is intended solely to give some comparative measure of the swelling properties of coals. From a consideration of the average error, it has been ascertained that the mean result of four tests on the same coal sample is correct to within ± 1 unit in 99 out of 100 cases and within ± 0.5 unit in 80 out of 100 cases, there is thus some assurance that different investigators can closely reproduce results on the same coal. The source of heat used in the test is a gas burner or electrically heated furnace. The International Organization for Standardization (ISO) has recommended both the heating methods.

7.2 Gas Heating Method

7.2.1 *Apparatus*

The assembly of the apparatus is shown in Fig. 1. It consists of the following components.

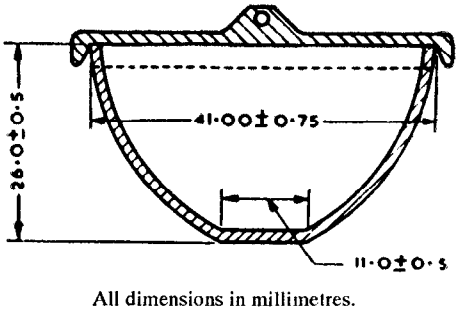


All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE CRUCIBLE SWELLING NUMBER TEST

7.2.1.1 *Silica*

Translucent, squat shape, having a silica lid with ring handle weighing not more than 12.75 g or less than 11 g with capacity approximately 17 ml and with the dimensions given in Fig. 2.



All dimensions in millimetres.

FIG. 2 CRUCIBLE AND LID FOR CRUCIBLE SWELLING NUMBER

In case where the lower surface of the crucible lid is not flat, difficulty may be experienced in assessing the swelling number of the coal. To overcome this, it is suggested that a small mica plate should be inserted between the lid and the crucible before heating the coal.

7.2.1.2 *Silica triangle*

Consisting of translucent silica tubing, 6 to 6.5 mm external diameter, mounted on chrome-nickel wire the length of the side being 63 to 64 mm, the diameter of the inscribed circle being approximately 32 mm.

7.2.1.3 *Teclu burner*

12.7 mm bore. Any other suitable type of burner may also be used.

7.2.1.4 *Draught shield*

Made from asbestos cement piping, approximately 150 mm long with 100 mm internal and 110 mm external diameters. The piping has three slots at one end, 25 mm deep, in which the wire portions of the silica triangle rest (*see* Fig. 1).

7.2.2 *Conditions of Heating*

The gas pressure and the gas and air supplies for the burner shall be adjusted by enlarging the size of the gas jet so that the flame is approximately 300 mm long. With the burner so adjusted, the position of the crucible, resting in the silica triangle and supported in the draught shield, shall be so arranged that the flame envelops the crucible and the temperature of the inner surface of the bottom of the crucible reaches 800°C ± 10°C in 1.5 minutes and 820°C ± 5°C in 2.5 minutes from the time the gas is ignited first. With these conditions, it will generally be found that the base of the crucible is just above the tip of the blue cone. These conditions apply particularly to coal gas of about 4 500 K-Cal/nm^3. For gas of much higher or much lower calorific value a different length of flame may be required. The use of four crucibles so that the test can be carried out in rapid sequence, following a blank test to warm the draught shield, is helpful when difficulty is found in attaining the standard rate of heating. These conditions of heating shall be checked at frequent intervals by means of a fine wire thermocouple inserted through a pierced lid, and having its unprotected junction in contact with the centre of the base of the empty crucible. This couple should be made of wires not heavier than 0.22 mm platinum or 0.45 mm base metal, and the end of the couple should be in the form of flattened loop so that the junction and a portion of each wire rest on the bottom of the crucible during a temperature measurement. The conditions for attaining the correct heating having been ascertained, the apparatus may conveniently remain permanently erected by fixing the draught shield on a suitable support, the burner remaining centred and being adjusted *in situ*.

7.2.3 *Grinding the Coal*

Air-dry the coal to be tested and grind it so that it passes IS Sieve 20 (211 microns) [*see* **6.4** (a)]. The grinding should be done not more than two hours before testing coal of weak swelling characteristics to prevent error due to oxidation.

7.2.4 *Procedure*

Weigh 1.00 to 1.01 g of the freshly ground coal into a crucible and lightly tap the crucible 12 times on the bench to level the surface of the coal. Cover the crucible with the lid and place it upright in the silica triangle supported in the draught shield. Light the gas and heat for such time as is required for the flame of the burning volatile matter to die out, and in any case for not less than 2.5 minutes. Allow the crucible to cool and carefully remove the coke button. Repeat the test until four buttons have been obtained. In some cases it may be necessary to prepare 5 buttons. After each test remove the carbon residue in the crucible and wipe the interior of the crucible clean.

7.2.5 *Examination*

Compare the coke button with the standard numbered outlines in Fig. 3. For the comparison, rotate the button about its axis so that the largest profile is presented to view. A method of viewing which excludes the effect of parallax is shown in Fig. 4. Place the drawing with which the button is to be compared exactly in the centre of the field of vision from the top of the tube. Arrange the button so that the maximum cross section is in line with the drawing when viewed with one eye placed immediately over the top of the tube.

7.2.6 *Report*

The swelling index of a button is the number inscribed in the outline that its largest profile most nearly matches. Report the mean swelling number of the series, expressed to the nearest half number.

7.2.7 For non-swollen button, the number '0' is used to describe coals which give a powder residue. The number '½' describes coals which give a coherent residue that will not bear a 500 g mass. The number '1' describes coals which give a coherent residue that cracks into two or three hard piece when the 500 g mass is applied.

7.3 Electrical Heating Method

7.3.1 *Apparatus*

7.3.1.1 *Furnace*

A suitable type of electrically heated furnace is shown in Fig. 5, although other types of furnaces may also be used, provided that the results obtained are the same (within ½ unit) as those obtained with the gas heating method.

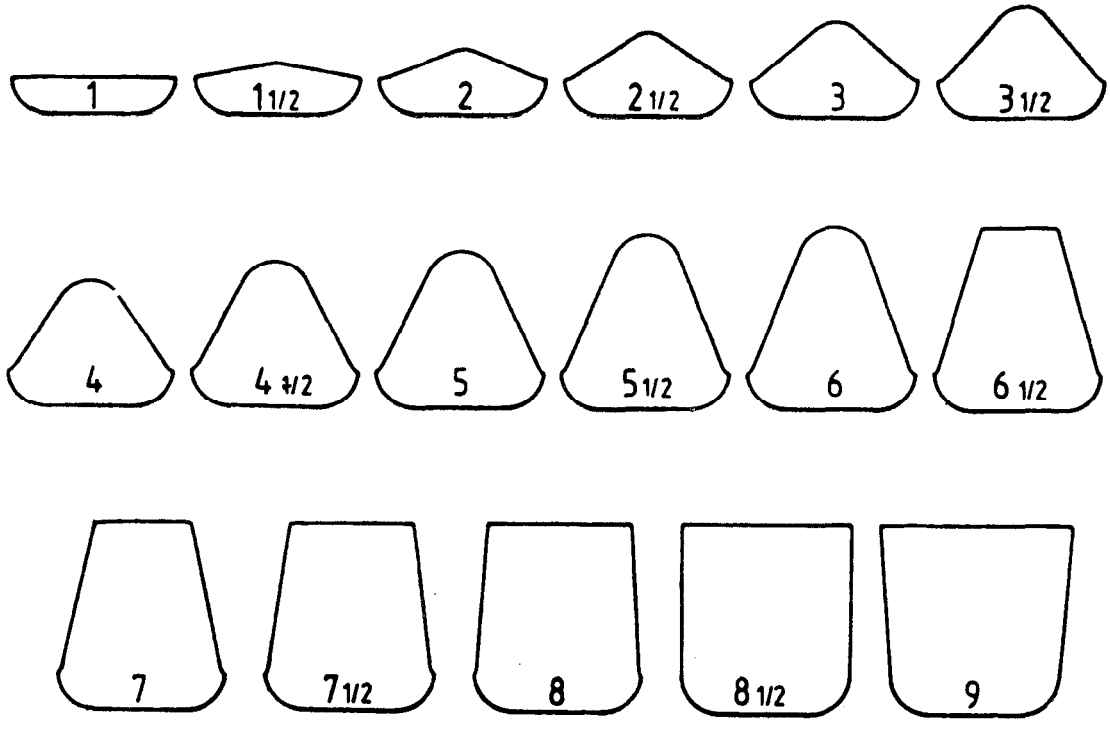


FIG. 3 STANDARD PROFILES AND CORRESPONDING CRUCIBLE SWELLING NUMBERS

The furnace illustrated consists of a groove refractory plate approximately 100 mm in diameter and 20 mm thick, carrying the heating element, which can be spiralled metallic coil. An inverted silica dish of 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 of approximately 225 mm diameter, bored to a depth of about 50 mm with a 100 mm diameter hole. The cylinder is fitted with a refractory lid 75 mm thick, a hole 50 mm in diameter is bored in the centre of the refractory lid to allow for insertion of the crucible. The whole furnace is placed in an aluminium or mild steel case.

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

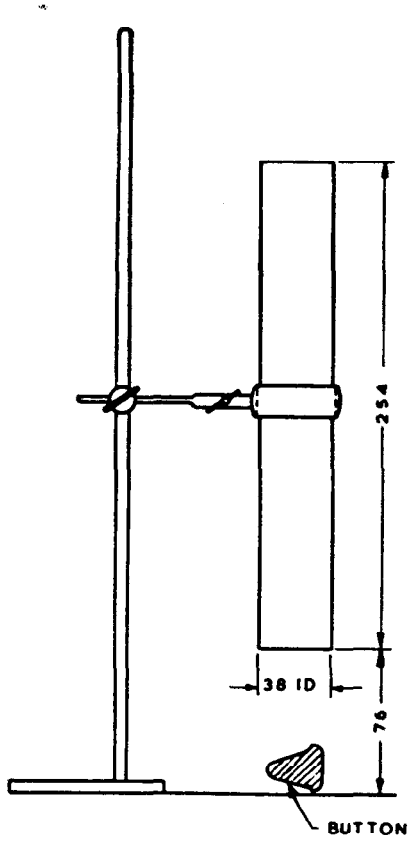
The furnace shall be equipped with a suitable auto-transformer meter and suitable temperature measuring device.

7.3.1.2 *Crucible and lid*

Same as described under **7.2.1.1**.

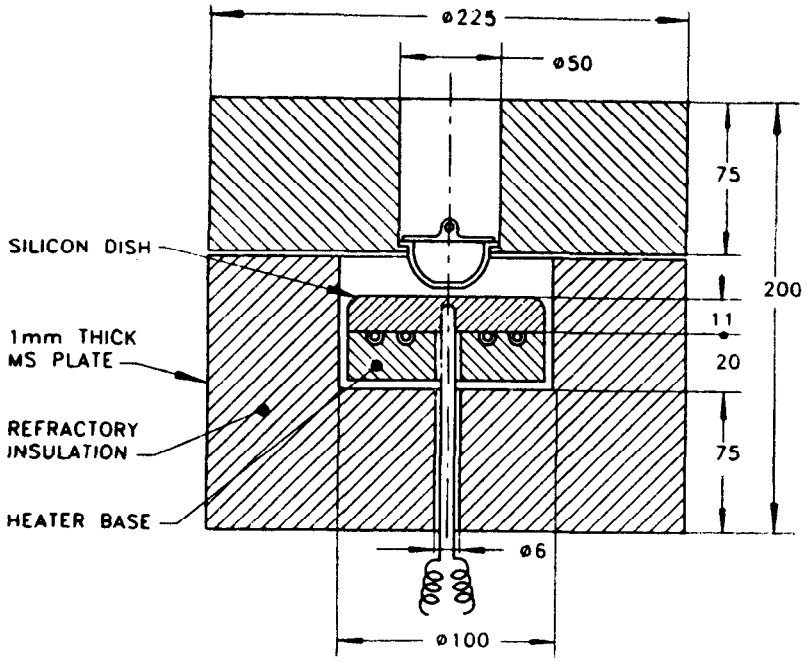
7.3.1.3 *Pierced silica lid*

Similar to that described in **7.2.1.1** but with a 6 mm hole to accommodate the thermocouple (Fig. 6).



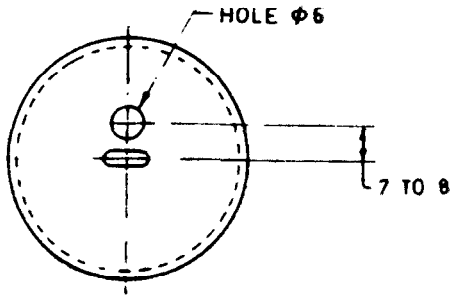
All dimensions in millimetres.

FIG. 4 APPARATUS FOR VIEWING THE BUTTON



All dimensions in millimetres.

FIG. 5 DETAILS OF SWELLING INDEX FURNACE



All dimensions in millimetres.

FIG. 6 PIERCED CRUCIBLE LID

7.3.1.4 *Thermocouples*

Of fine wire of diameter not greater than 0.5 mm if made of chromel alumel. The ends of the couple shall be in the form of a flattened loop.

7.3.2 *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 the crucible resting on the silica plate. 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 crucible. Ascertain that the standard heating conditions of 800 ± 10°C in 1.5 minute and 820 ± 5°C in 2.5 minute 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 serving as a datum.

7.3.3 *Procedure*

Weigh 1.00 to 1.01 g of the freshly ground sample 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 specified in **7.2.5**.

Carry out five tests in succession, replacing one crucible with the next to avoid heat losses through the top of the furnace.

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

7.4 Report

Report the swelling number of the coal as specified in **7.2.6** and **7.2.7**.

8 DETERMINATION OF LOW TEMPERATURE (LT) GRAY-KING ASSAY

8.1 General

The purpose of the test is to assess the caking properties of a coal or a blend of coals and also the yield of various byproducts by carbonising in a laboratory under standard conditions at a maximum temperature of 600°C. The coke residue from the carbonization of finely ground coal at 600°C is

classified by comparison with a series of described coke types. For strongly swelling coals, the coal is blended with electrode carbon or high temperature coke breeze in a proportion which gives on carbonization, a strong, hard coke of the same volume as the original coal and electrode carbon/coke breeze mixture.

8.2 Apparatus

Figure 7 shows the assembly of apparatus.

8.2.1 *Furnace*

'*A*' is a 300 mm long tubular electrically heated type capable of giving a uniformly heated space (within 10°C any temperature between 300°C and 600°C) in the middle 20 cm portion. The wheels fitted to the furnace facilitate its easy movement over the retort (*B*). The energy regulator of the furnace should be capable of allowing temperature rise at the rate of 5°C per minute. The well fitting ends of insulated material provided prevent undue cooling of the retort by circulation of air.

8.2.2 *Thermocouple*

A thermocouple is fitted into a refractory or stainless steel sheeth passing through the rear end of the furnace and positioned so that the tip of the thermocouple is midway along the length of the charge and the sheath is almost in contact with the retort when the latter is in the furnace. The thermocouple is connected with an instrument for measuring the emf or equivalent temperature difference, the combination being sensitive to 5°C.

8.2.3 *Retort*

Preferably of transparent borosilicate glass/silica, consisting of a tube, 300 mm in length closed at one end, with a side-arm near the open end and with dimensions and tolerances as given in Fig. 8. There should be a slight taper in the bore of the tube so that it is little wider at the open end than at the closed end.

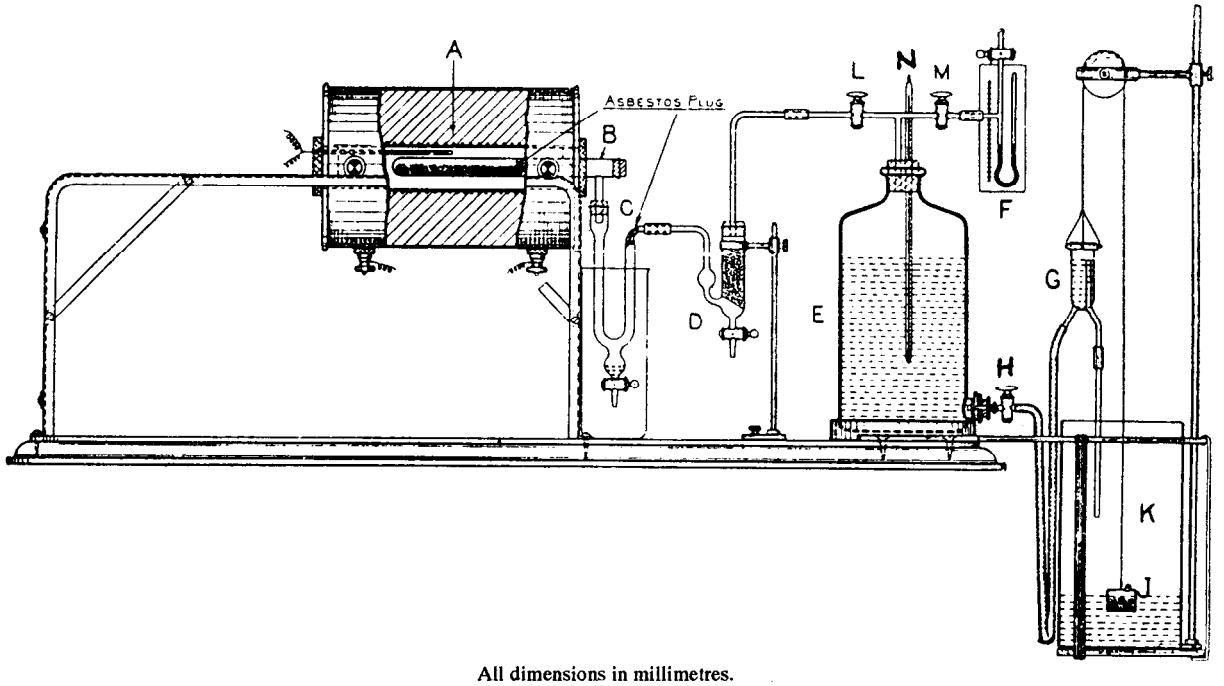
8.2.4 *Receivers*

Tar cooler *C* is a U-shaped hard glass tube (*see* Fig. 9) with its inlet limb closely fitting the rubber bung attached to the side arm of the retort and outlet limb extended and bent to allow for connection with the ammonia scrubber *D* downstream. It is provided with a bulb (at the bottom) of capacity of at least 5 ml for receiving the condensed products and a stopcock which allows their removal when required. The tar cooler is supported suitably and cooled externally by immersing in a tall glass beaker/jar containing cold water or ice.

NOTES

1 Glass beaker/jar is preferable as it allows for the observation of tar cooler, progress of condensation of products, leakage, if any, of the stopcock, etc, from outside enabling the operator to take the required precautionary measures.

2 If it is required to determine the (LT) Gray-King Coke Type only the outlet limb of the tar cooler is fitted with a suitable extension of rubber and silica tubings leading to the atmosphere or with a burner at the end of which the gas can be burnt. The type of furnace shown in Fig. 10 allows for multiple determinations of coke type simultaneously.



All dimensions in millimetres.

FIG. 7 LOW TEMPERATURE GRAY-KING ASSAY APPARATUS

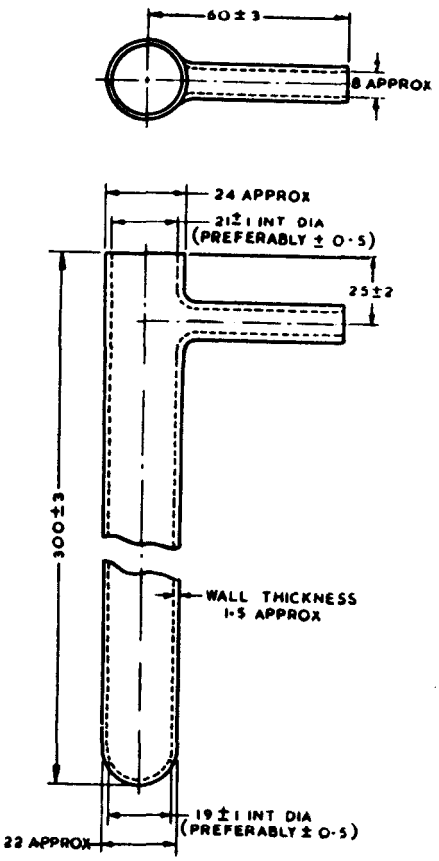


FIG. 8 RETORT FOR GRAY-KING TEST

8.2.5 *Distance Rod*

To gauge the length over which the sample is spread in the retort tube. It may be in the form of a brass piston or of a rubber bung mounted on a glass rod. The end fits loosely in the bore of the retort.

8.2.6 *Electrode Carbon*

To comply with the following requirements:

	Retained	*Percent*
a)	Screen analysis:	
	On IS Sieve 20	Nil
	On IS Sieve 12 and passing through IS Sieve 20, *Max*	26
	On IS Sieve 6 and passing through IS Sieve 20	10 to 40
	Passing through IS Sieve 6	50 to 85
b)	Moisture, air-dried basis	1.0
c)	Volatile matter, less moisture air-dried basis, *Max*	1.5
d)	Ash, *Max*	5.0
e)	Bulk density (determined by the method described under **8.6**)	1.00 to 1.03 g/ml at 25°C
f)	Specific gravity	2.05 to 2.09

NOTES

1 True specific gravity or density is determined by the normal method using the density bottle. To ensure complete wetting of the electrode carbon, a one percent solution of a sodium alkyl sulphate type wetting agent solution is used and the density bottle containing the electrode carbon and the wetting agent solution is evacuated to pressure of 60 mm of mercury in a vacuum desiccator. This vacuum is maintained for 10 minutes before transferring the density bottle to a water-bath thermostatically controlled at 25°C.

2 In place of electrode carbon, high temperature coke breeze of normal metallurgical coke crushed 100 percent through 15 IS Sieve with about 50 percent passing through 6 IS Sieve may be used.

8.2.7 *Ammonia Scrubber*

D is a hard glass tube fitted with a stopcock at bottom and an inlet limb as shown in detail in Fig. 9. The scrubber is charged with dilute sulphuric acid (about 6 N) to absorb ammonia gas. The closely fitting rubber bung at the top of the scrubber is provided with a bent glass outlet tube which in turn is connected to the gas-holder through the stopcock *L*.

NOTE — A few glass beads are to be immersed in H_2SO_4 solution to increase the surface area.

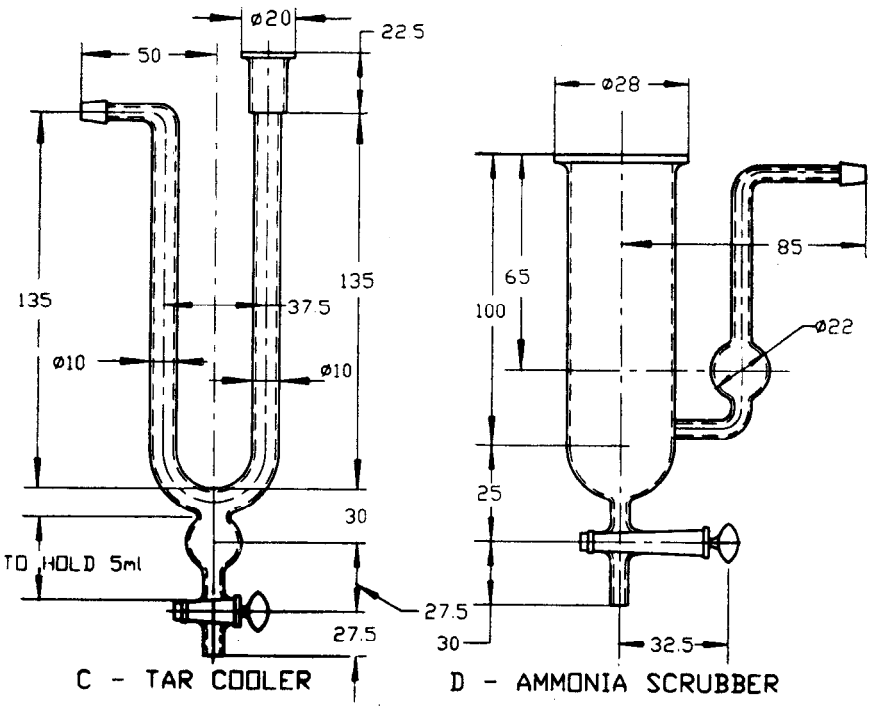
8.2.8 *Gas Holder*

E is a 5 litre aspirator bottle, filled with a mixture of glycerine and water (50 : 50 by volume) and a few ml H_2SO_4 (specific gravity of the mixture 1.15 approx). The gas holder is connected through the stopcock *H* by rubber tubing to a freely suspended glass reservoir *G* using a pulley as shown in Fig. 7. A glass vessel *J* containing lead block counterbalances the reservoir.

NOTE — Addition of a few ml of H_2SO_4 prevents scum formation.

When gas enters the gas holder through the stopcock *L* the displaced liquid flows into the glass reservoir *G*, from where the overflow liquid falls into the glass container *K*. If the internal diameter of the container *K* is equal to that of the gas holder *E*, any fall in the liquid level in *E* causes an equal rise in the liquid level in the container *K*. The counterpoise *J* floating on the liquid in the container *K* rises with the liquid level. Consequently, the reservoir is lowered to the extent of the rise of counterpoise *J*, thereby maintaining a constant pressure automatically throughout in the gas holder *E*. A mild suction required for the easy flow of gas may be maintained by suitably adjusting the height of the reservoir.

The stopcocks *L* and *M* on both sides of the gas holder serve to isolate it from the system and thereby contained gas at the end of the experiment. The water level in the limbs of manometer *F* shows the difference in the pressures of gas in gas holder *E* and that of the atmosphere. The thermometer *N*, fitted to the gas holder *E*, serves to measure the temperature at which the gas is collected.



All dimensions in millimetres.

FIG. 9 TAR COOLER AND AMMONIA SCRUBBER

8.2.9 *Barometer*

To measure the atmospheric pressure prevailing at the time of the experiment.

8.2.10 *Clock or Watch*

Indicating both minutes and seconds.

8.3 Preparation of Coal Sample

The coal is air-dried and ground to pass IS Sieve 20 (21 microns) preferably with a laboratory mechanical mixer.

8.3.1 *Precautions for Rapidly Deteriorating Coking Coals or Oxidising Coals*

The coking properties/propensities of some coals deteriorate rapidly when they are exposed to air, and in all strictly comparative work, precaution should be taken to obviate this deterioration. Storage in sealed containers under water or with the air replaced by nitrogen, are recognised methods. Since certain coals are very liable to oxidation it is important that the coal should not be heated above normal room temperature at any stage in the preparation of the sample.

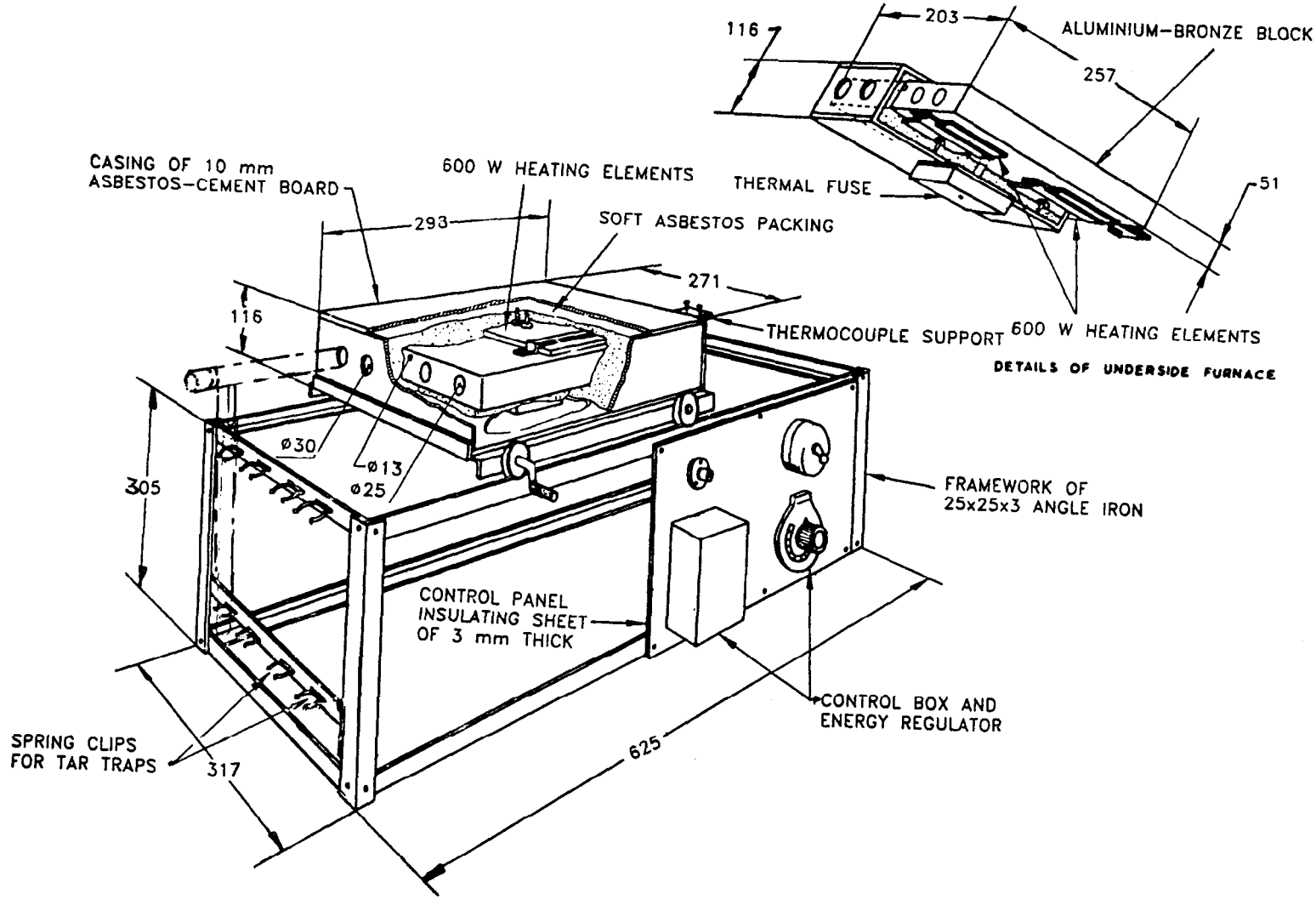
8.4 Procedure

Two techniques are followed depending upon the types of coal under investigation.

8.4.1 *Technique I —Applicable to Non-swelling and Moderately Swelling Coals Giving Cokes Up to and Including Type* G_2

a) Determine the moisture content of the air-dried coal sample. Weigh 20.0 ± 0.1 g of coal on a scoop and transfer without fouling the side-arm, into the main body of the retort which is held almost vertically. Brush into the retort with a small soft brush any coal adhering to the scoop. Turn the retort into a horizontal position with the side arm downwards and the distance rod inserted so that the face of the piston is 150 mm from the closed end of the retort. Hold the open end of the retort and the positioned distance rod in one hand and the closed end of the retort in the other. By careful shaking, spread the coal into an even layer over the 150 mm length, and then lightly consolidate by tapping the retortion the bench. Carefully push back any coal which may have crept past the distance rod, on to the main body of the coal by careful strokes of the distance rod itself. Re-level, consolidate again and withdraw the distance rod.

Alternatively, a plug of ignited asbestos wool is inserted into the retort (after dropping coal into the retort and levelling the same) at 150 mm from the closed end. Gentle tapping help smoothen the coal layer. Close the open end of the retort by means of a soft rubber or neoprene bung.



All dimensions in millimetres.

FIG. 10 FOUR-IN-ONE GRAY-KING FURNACE

Clean and dry the tar cooler, keep a small wad of burnt asbestos wool in its outlet limb (the wad of asbestos swool prevents the escape of tar vapours) weigh and attach to the side arm of the retort.

Fill the ammonia scrubber *D* with dilute sulphuric acid (6 N) till the glass beads are completely drenched.

Fill the gas holder *E* completely with glycerine/water mixture. Adjust the level of reservoir *G* so that top of the overflow tube is slightly below the level of the liquid in the gas holder *E* to develop mild suction.

Weigh the container *K* and place it in position.

Clamp the assembly, namely, retort *B*, tar cooler *C*, ammonia scrubber *D*, gas holder *E*, gas reservoir *G*, container *K* into position as shown in Fig. 7.

b) The furnace, previously raised to a steady temperature of 325°C, rests on the frame behind the retort and is screened from it by a piece of asbestos board. Remove the asbestos board and draw the furnace smoothly and quickly over the retort. Start the timing from this instant, and increase the heating current by a pre-determined amount to give, as nearly as possible, a regular rise in temperature of 5°C per minute.

c) The tolerances for the temperature control are as follows:

Heating rate	5°C ± 1°C per minute
Temperature at any instant	300°C + 5*t* ± 10°C where *t* is the time in minutes from the start of heating of the coal

d) When the furnace is drawn over the retort, the temperature drops to about 300°C and then starts to rise at approximately the required rate of 5°C per minute. At the end of the fifth minute it may not be exactly 325°C, but this temperature must be attained within a range of 3 to 7 minutes from the start.

e) Consider the re-attainment of 325°C as the 5 minutes datum line and reset the clock accordingly. Thereafter maintain the regular increase in temperature of 5°C per minute by small increase of current at approximately regular intervals of time, the temperature being observed every two or three minutes.

f) Observe and note down the 'gas point' and 'oil point', that is, the temperatures at which the evolution of gas commences and oil vapours first appear respectively.

When the temperature reaches about 590°C reduce the current to that required to maintain the furnace at 600°C. The 'thermal inertia' of the apparatus is usually sufficient to carry the temperature from 590°C to 600°C in the final two minutes. Hold the furnace at this temperature for further 1 hour always cautiously maintaining the pressure level in the manometer.

NOTE — The rate of evolution of gas at the end of 1 hour at the final temperature falls so low that further heating gives a negligible addition to the volume.

g) Pipette out some liquid from the container *K* and allow to flow into the glass reservoir *G* raising it slowly till the top of the inlet is at the same level to that of the liquid in the gas holder *E* and the manometer *F* is levelled. Return the unused liquid to the container *K*. Record the temperature (T_1) as shown by thermometer *N* and the pressure (P_1) as shown by the barometer.

Close stopcocks *L* and *M*, withdraw the furnace, detach retort *B* from the tar cooler *C* and allow the retort to cool.

h) *Coke type and yield* — When the retort is cool enough to handle, weigh it along with the rubber bung. Remove traces of tar sticking to the mouth and side arm of the retort and the rubber bung by burning off in a blowpipe flame or by solvents. Record the mass of tar removed from retort. Also record the yield of coke residue.

j) Gently slide carbonised residue out of the retort and compare it with standard coke types as shown in Fig. 11 and assign appropriate coke types.

If coke type obtained is above G_2 repeat the assay by following technique II applicable to swelling coals.

k) *Tar yield* — Clean and weigh tar cooler *C*. The increase in mass represents the combined yield of tar, liquor and moisture from coal.

In order to obtain yields of tar and liquor separately wash down contents of the tar cooler with benzene into a 10 ml measuring cylinder. Measure the volume of the aqueous layer and convert to grams assuming specific gravity of liquor to be 1.0. Record this mass as mass of liquor plus moisture from coal. Deduct the mass so obtained from the combined mass of tar liquor and moisture from coal to get the yield of tar and then add the mass of tar removed from retort to get the total yield of tar.

m) *Liquor yield* — The yield of liquor is obtained by deducting 1/5th of the percentage moisture of coal from the combined mass of liquor plus moisture from coal.

n) *Ammonia yield* — Wash down contents of ammonia scrubber with distilled water and take it along with the aqueous layer from tar cooler into a distillation flask and make up the volume with sufficient distilled water.

Clamp the flask into the distillation apparatus, make the contents alkaline by adding 100 ml of sodium hydroxide solution (40 percent, *m/v*), distil and estimate ammonia as given in IS 1350 (Part 4/Sec 2):1975. A single determination will suffice.

p) *Gas yield* — Weigh container *K* along with the collected liquid. The increase in mass represents the mass of liquid displaced by the gas collected in gasholder *E*. Measure the specific gravity of liquid with a hydrometer. Calculate the volume of gas. (Alternatively, the gas volume can also be estimated by measuring the volume of the displaced liquid itself). Record temperature of gas and atmospheric pressure. Note the vapour pressure at that temperature (*see* Table 1) and subtract this to arrive at the correct pressure. Calculate the volume to NTP (saturated) that is, 0°C and 760 mm Hg (saturated).

q) An example for calculation of yield of products is illustrated in Annex A.

r) The cooling of the furnace after the test can be hastened by placing in it a suitable iron rod or a water cooled tube. After the removal of tar and coke from the retort, it is only necessary to wipe out the inside of the retort with a cloth to clean it ready for the next test.

8.4.2 *Technique II —Applicable to Swelling Coals, Giving Cokes Above Type* G_2 (*see* **8.5**)

a) A different technique is used for coals giving a coke that is swollen to a volume much greater than that of the original coal. Here the classification is based on the minimum proportion of electrode carbon or coke breeze which is necessary to add to the coal to control the swelling.

b) A first rough guide to the correct blend is given by the following table relating crucible swelling numbers and (LT) Gray-King Assay coke types although it must be appreciated that there are many and striking exceptions:

Crucible Swelling Number	*(LT) Gray-King Assay Coke Type*
0 — ½	A — B
1 — 4	C — G_2
4½ — 6	F — G_4
6½ — 8	G_3 — G_9
8½ — 9	G_7 or above

In this table, the subscript figure to the letter *G* gives the number of parts of electrode carbon or coke breeze in 20 parts of that coal-electrode carbon or coke breeze mixture which gives a coke of type G. In all the trials carried out by this technique, 20 ± 0.2 g of the mixture are carbonised, consisting of a quantity of electrode carbon or coke breeze, say x ± 0.1 g and (20 – x) ± 0.1 g of the coal. The quantities are weighed separately and thoroughly mixed by shaking and rotating for 5 minutes in a screw capped jar of about 100 ml capacity. The whole of the 20 g mixture is then transferred to the retort and carbonised as described for non-swelling coals (*see* **8.4.1**).

c) Two or more assays on different blends are usually necessary to make a precise assessment of a swelling coal.

8.4.3 Conduct the assay in duplicate and take average of the corresponding yields of the products. Carry out blank determination using electrode carbon or coke breeze, other procedure of the experiment remaining the same. Deduct the volume of gas (that is, displaced air converted to NTP) from the volume of gas obtained at NTP.

8.4.4 *Tolerance*

Degree of accuracy of the method is ± 0.2 percent of the coal for the yields of coke, tar and liquor and ± 125 ml per 100 g of coal for the gas volume.

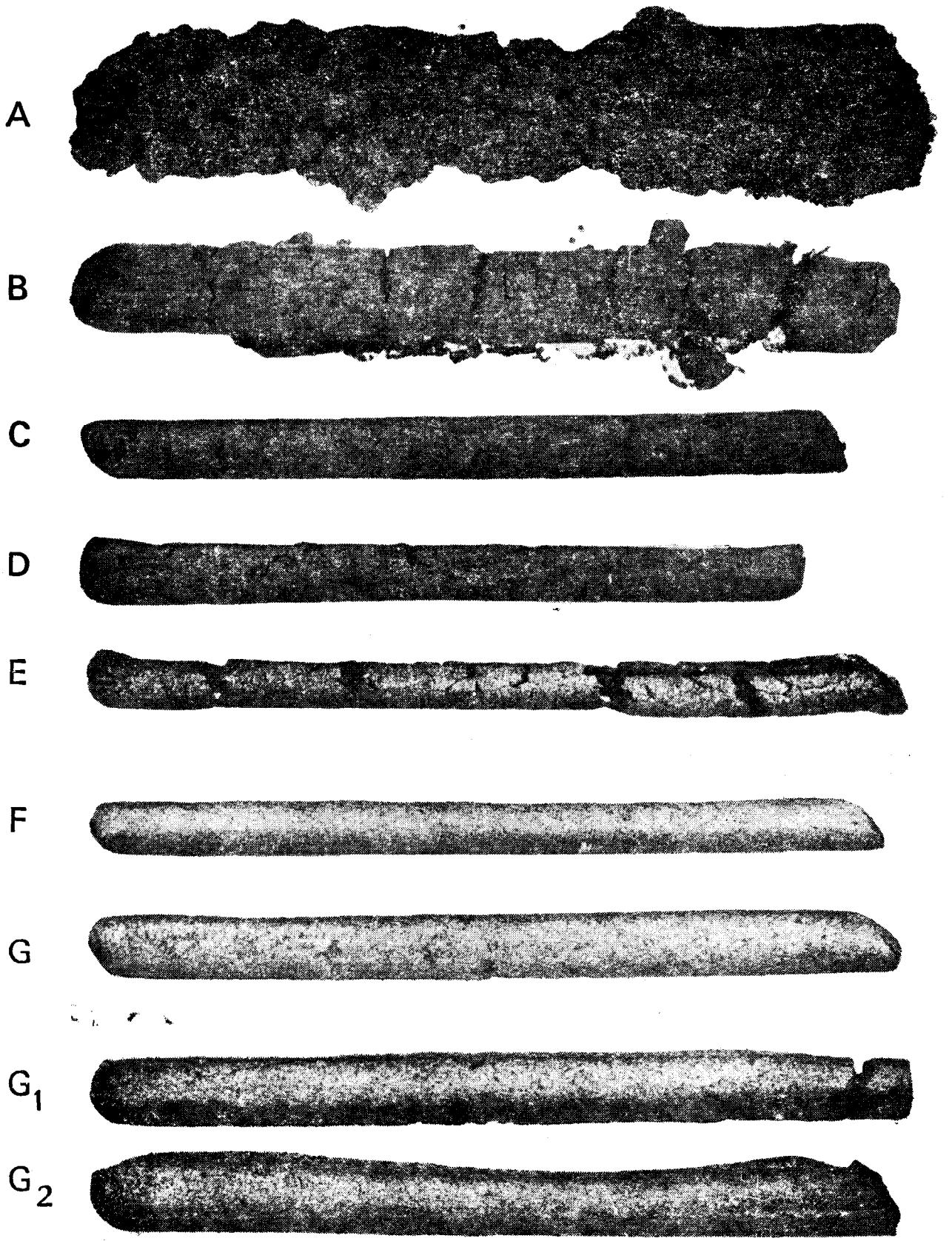


FIG. 11 TYPES OF COKE FROM GRAY-KING ASSAY

Table 1 Vapour Pressure of Liquid Water

[*Clause* 8.4.1(p)]

mm Hg

Temp, °C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
5	6.343	6.589	6.635	6.681	6.728	6.775	6.822	6.869	6.917	6.965
6	7.013	7.062	7.111	7.160	7.209	7.259	7.309	7.360	7.411	7.462
7	7.513	7.565	7.617	7.669	7.722	7.775	7.828	7.882	7.936	7.990
8	8.045	8.100	8.155	8.211	8.267	8.323	8.380	8.437	8.494	8.504
9	8.609	8.658	8.727	8.786	8.845	8.905	8.965	9.025	9.086	9.147
10	9.209	9.271	9.333	9.395	9.458	9.521	9.585	9.649	9.714	9.779
11	9.844	9.910	9.976	10.042	10.109	10.176	10.244	10.312	10.380	10.449
12	10.518	10.588	10.658	10.728	10.799	10.870	10.941	11.013	11.085	11.158
13	11.231	11.305	11.379	11.453	11.526	11.604	11.680	11.755	11.833	11.910
14	11.987	12.065	12.144	12.223	12.302	12.382	12.462	12.543	12.624	12.706
15	12.788	12.870	12.953	13.037	13.121	13.205	13.290	13.375	13.461	13.457
16	13.634	13.721	13.809	13.898	13.987	14.076	14.166	14.256	14.347	14.438
17	14.530	14.622	14.715	14.809	14.903	14.997	15.092	15.188	15.284	15.380
18	15.477	15.575	15.673	15.772	15.871	15.971	16.071	16.171	16.272	16.374
19	16.477	16.581	16.685	16.789	16.894	16.999	17.105	17.212	17.319	17.427
20	17.535	17.664	17.753	17.863	17.974	18.085	18.197	18.309	18.422	18.536
21	18.650	18.765	18.880	18.996	19.113	19.231	19.349	19.468	19.587	19.707
22	19.827	19.948	20.070	20.193	20.316	20.440	20.565	20.690	20.815	20.941
23	21.068	21.196	21.324	21.453	21.583	21.714	21.845	21.977	22.110	22.243
24	22.377	22.512	22.648	22.785	22.922	23.060	23.198	23.337	23.476	23.616
25	23.756	23.897	24.039	24.182	24.326	24.471	24.617	24.764	24.912	25.060
26	25.209	25.359	25.509	25.660	25.812	25.964	26.117	26.271	26.426	26.582
27	26.739	26.897	27.055	27.214	27.374	27.535	27.696	27.858	28.021	28.185
28	28.349	28.514	28.680	28.847	29.015	29.184	29.354	29.525	29.697	29.870
29	30.043	30.217	30.392	30.568	30.745	30.923	31.102	31.281	31.461	31.042
30	31.824	32.007	32.191	32.376	32.561	32.747	32.934	33.122	33.312	33.503
31	33.695	33.888	34.082	34.276	34.471	34.667	34.864	35.062	35.261	35.462
32	35.663	35.865	36.068	36.272	36.477	36.683	36.891	37.099	37.308	37.518
33	37.729	37.942	38.155	38.369	38.584	38.801	39.018	39.237	39.457	39.677
34	39.898	40.121	40.344	40.569	40.796	41.023	41.251	41.480	41.710	41.942
35	42.175	42.409	42.644	42.880	43.117	43.355	43.595	43.836	43.078	44.320
36	44.563	44.808	45.054	45.301	45.549	45.799	46.050	46.302	46.556	46.811
37	47.067	47.324	47.582	47.841	48.102	48.364	48.627	48.891	49.157	49.424
38	49.692	49.961	50.231	50.502	50.774	51.048	51.323	51.600	51.879	52.160
39	52.442	52.725	53.009	53.294	53.580	53.867	54.156	54.446	54.737	55.030
40	55.324	55.61	55.91	56.21	56.51	56.81	57.11	57.41	57.72	58.03
41	58.34	58.65	58.96	59.27	59.58	59.90	60.22	60.54	60.86	61.18
42	61.50	61.82	62.14	62.47	62.80	63.13	63.46	63.79	64.12	64.46
43	64.80	65.14	65.48	65.82	66.16	66.51	66.86	67.21	67.56	67.91
44	68.26	68.61	68.97	69.33	69.69	70.05	70.41	70.77	71.14	71.51
45	71.88	72.25	72.62	72.99	73.36	73.74	74.12	74.50	74.88	75.25
46	75.65	76.04	76.43	76.82	77.21	77.60	78.00	78.40	78.80	79.20
47	79.60	80.00	80.41	80.82	81.23	81.64	82.05	82.46	82.87	83.29
48	83.71	84.13	84.56	84.99	85.42	85.85	86.28	86.71	87.14	87.58
49	88.02	88.46	88.90	89.34	89.79	90.24	90.69	91.14	91.59	92.05
50	92.51									

Products	*Difference Between the Duplicate in 20 g of Dry Coal*
Coke	0.04 g
Tar	0.04 g
Liquor	0.04 g
Gas	25 ml (at NTP)

8.5 Assessment of Coke Type and Classification of the Coal

8.5.1 The description of the coke is made in standard terms, photographs of the types of standard coke have been reproduced in Fig. 11 and a systematic scheme for deciding the coke type is shown in Table 2. The photographs indicate the character and degree of swelling of the cokes. The letters A to G have been used to designate cokes from coals that are non-caking up to those that gives a hard strong coke of the same volume as the original coal (Type *G*). For coals of greater swelling power than *G*, the terms G_1, G_2, G_3 etc, are used, the subscript figure being the number of parts of electrode carbon or coke breeze added, as explained above. Coke types G_1 and G_2 should be judged from the swelling of the coke, without admixture of electrode carbon or coke breeze — coke types G_3 and above are assessed by the blending procedure. When assessing the 'G' number, it is essential to work from the direction of low electrode carbon or coke bronze content, that is, from swollen cokes. The result that is reported is the minimum number of parts of electrode carbon or coke breeze which will result in the production of a coke of standard G type. Surmises from blends giving cokes more shrunken than G or greater than G_1 are unreliable.

8.6 Determination of Bulk Density of Electrode Carbon

8.6.1 Weigh accurately about 50 g (*M*) of the electrode carbon of specified size (**8.2.6**) and transfer it completely into a graduated glass cylinder measuring 100 ml (each sub-division of 1 ml graduation). Raise the cylinder to a height of about 25 mm above the level of the work-bench and gently tap on a rubber pad placed on the workbench, for 50 times and read the volume in ml (*V*).

8.6.2 *Calculation*

$$\text{Bulk density } (D)_n \text{ g/ml} = \frac{M}{V}$$

where

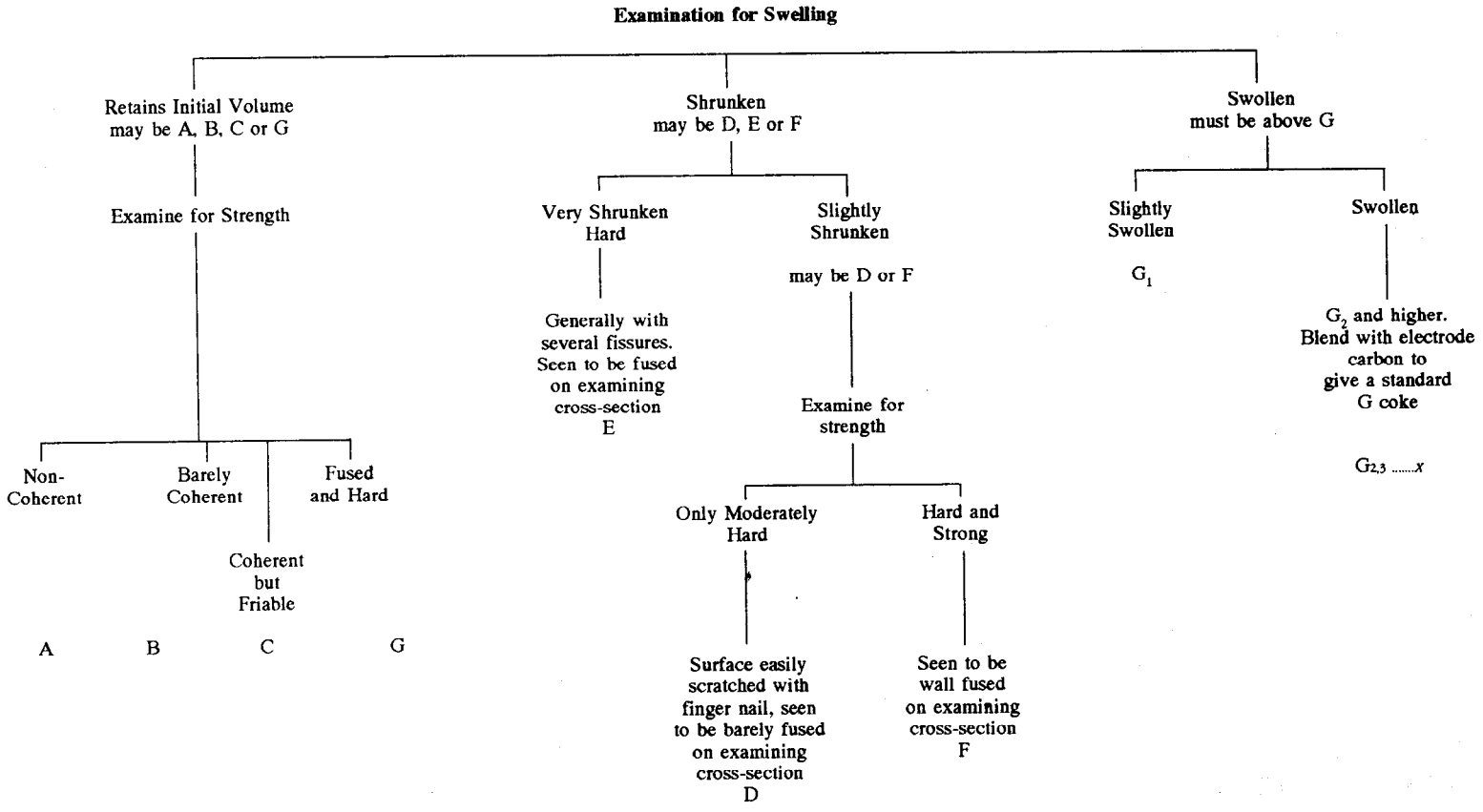
M = mass in g of electrode carbon taken, and

V = volume in ml of electrode carbon after taping (**8.6.1**).

8.6.3 Repeat the operation two times more and take the mean of the three values to arrive at the bulk density of the material.

Table 2 Scheme for Examination and Classification of (LT) Gray-King Assay

(*Clause* 8.5.1)



ANNEX A

[*Clause* 8.4.1 (q)]

EXAMPLE OF CALCULATION OF YIELD OF PRODUCTS OF (LT) GRAY-KING ASSAY

Moisture of the coal sample (M)	1.3%
Mass of the retort	92.86 g
Mass of the retort + coal	112.86 g
Mass of the retort + coal + plug	113.55 g
Mass of the retort + plug (a)	93.55 g
Mass of the retort + coke + tar + plug	110.54 g
Mass of the retort + coke + plug (b)	110.52 g
Mass of the tar in the retort (t_1)	0.02 g
Mass of the coke ($b - a$)	16.97 g
Mass of tar cooler	75.704 g
Mass of tar cooler + tar + liquor + moisture from coal	77.574 g
Mass of tar + liquor + moisture from coal	1.870 g
Mass of liquor + moisture from coal (Sp. Gr-I)	0.700 g
Mass of tar in the tar cooler (t_2)	1.170 g
Mass of coal tar ($t_1 + t_2$)	1.190 g
Mass of moisture due to coal ($M/5$)	0.260 g
Mass of liquor	0.44 g
Mass of container	760 g
Mass of container + displaced liquid	3946 g
Mass of displaced liquid (W)	3186 g
Relative density of the liquid (d)	1.15 g/ml
Volume of gas W/d	2770 ml
Temperature of gas	25°C
Pressure of gas (P)	748.00 mm
Vapour pressure at 25°C (Ref Table 1) (p)	23.8 mm
Corrected pressure ($P - p$)	724.2 mm
Volume of gas at NTP (0°C & 760 mm) (V)	2417 ml
Blank for the retort (u)	37 ml
Correct volume of gas ($v - u$)	2380 ml
Ammonia ($X \times S \times 0.001\ 7$) = ($13.8 \times 0.870\ 1 \times 0.001\ 7$)	0.02 g

X = Vol of sulphuric acid

S = Strength of N/10 sulphuric acid

Details of calculation of gas volume at NTP

Volume (V) at NTP is given by

$$\frac{760 \times V}{273} \times \frac{724.2 \times 2770}{(273 + 25)}$$

or V = 2 417 ml

Reporting of Results

Particulars	*Yield per 20 g (Air-Dried Coal)*	*Yield per 100 g (Air-Dried Coal)*	*Yield per 100 g (Dry Coal)*	*Yield per tonne (Dry Coal)*
Coke	16.97 g	84.65 g	85.97 g	859.7 kg
Tar	1.19 g	5.95 g	6.03 g	60.3 kg
Liquor	0.44 g	2.20 g	2.23 g	22.3 kg
Gas at (NTP)	2 380 ml	11.900 ml	12.057 ml	120.6 m^3
Ammonia	0.02 g	0.10 g	0.10 g	1.0 kg

Oil point — 355°C

Gas point — 380°C

Coke type — E

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