भारतीय मानक **IS 1350 (Part 4/Sec 1) : 2023**

Indian Standard

कोयला और कोक — परीक्षण पद्धतियाँ

भाग 4 अंतिम विश्लेषण

अनुभाग 1 कार्बन और हाइड्रोजन का निर्धारण

(दूसरा पुनरीक्षण)

Coal and Coke — Methods of Test

Part 4 Ultimate Analysis

Section 1 Determination of Carbon and Hydrogen

(Second Revision)

ICS 73.040; 75.160.10

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Solid Mineral Fuels and Solid Biofuels Sectional Committee, PCD 07

FOREWORD

This Indian Standard (Part 4/ Sec 1) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Solid Mineral Fuels and Solid Biofuels Sectional Committee has been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published as IS 1351 : 1959 ‘Methods of test for coal and coke — Ultimate analysis’ and subsequently revised as IS 1350 (Part 4/Sec 1) : 1974 ‘Methods of test for coal and coke: Part 4 Ultimate analysis, Section 1 Determination of carbon and hydrogen’.

In the light of the experience gained in the country and the ISO standard published on this subject, the committee responsible for the preparation of this standard, felt a need to revise IS 1351. Thus, the first revision was taken up to bifurcate IS 1351 : 1959 into two parts with modified IS No. and title as:

> IS 1350 (Part 4/Sec 1) : 1974 Methods of test for coal and coke: Part 4 Ultimate analysis, Section 1 Determination of carbon and hydrogen
>
> IS 1350 (Part 4/Sec 2) : 1975 Methods of test for coal and coke: Part 4 Ultimate analysis, Section 2 Determination of nitrogen

The 1974 version of this standard covered, the following three methods for determination of carbon and hydrogen:

a) Fentons method developed at Central Fuel Research Institute (CFRI), Dhanbad which is now CSIR - Central Fuel Research Institute, Dhanbad.
b) Liebig method, derived from ISO/R 625 : 1967 Determination of carbon and hydrogen in coal and coke by the Liebig method; and
c) High-temperature combustion method, derived from ISO/R 609 : 1967 Determination of carbon and hydrogen in coal and coke by the high-temperature combustion method.

Further, in 2011, the standard was amended to delete Liebig method and retained the other two methods. In this (*second*) revision, the reference of IS 1352 has been modified to IS 1350 (Part 5), as IS 1352 has been superseded by IS 1350 (Part 5). Also, the references have been updated.

The other series of this standard are:

Part 1 Proximate analysis
Part 2 Determination of gross calorific value
Part 3 Determination of total sulphur eschka method
Part 4 Ultimate analysis,
    Section 2 Determination of nitrogen
Part 5 Determination of special impurities (carbon present as carbonate, chlorine and phosphorus)

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

METHODS OF TEST FOR COAL AND COKE

PART 4 ULTIMATE ANALYSIS

SECTION 1 DETERMINATION OF CARBON AND HYDROGEN

(Second Revision)

1 SCOPE

This standard (Part 4/Sec 1) prescribes the methods of determination of carbon and hydrogen contents in coal.

NOTE — These methods are applicable equally to coke, brown coals and lignites.

2 REFERENCES

The standards listed below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibilities of applying the most recent edition of these standards:

IS No.	*Title*
IS 266 : 1993	Sulphuric acid — Specification (*third revision*)
IS 436 (Part 1/Sec 1) : 1964	Methods for sampling of coal and coke: Part 1 Sampling of coal, Section 1 Manual sampling (*first revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1350	Methods of test for coal and coke:
(Part 1) : 1984	Proximate analysis (*second revision*)
(Part 5) : 2017	Determination of special impurities (carbon present as carbonate, chlorine and phosphorus) (*second revision*)
IS 16143 (Part 5) : 2021/ ISO 13909-5 : 2016	Hard coal and coke — mechanical sampling: Part 5 Coke sampling from moving streams (*first revision*)

3 TERMINOLOGY

3.1 For the purpose of this standard, the following term shall apply.

3.1.1 *Ultimate Analysis* — The analysis of coal expressed in terms of its carbon, hydrogen, nitrogen, sulphur and oxygen contents.

4 QUALITY OF REAGENTS

Unless specified otherwise, ‘pure chemicals’ and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

5 SAMPLE

The method of preparing samples for carrying out tests shall be as prescribed in **10** of IS 436 (Part 1/Sec 1) and coke sample, in accordance with IS 16143 (Part 6).

6 DETERMINATION OF CARBON AND HYDROGEN

6.1 General

6.1.1 The Method A – Fentons method developed by Central Institute of Mining & Fuel Research (CIMFR) given in 6.2 shall be used as a routine method and Method B – High-temperature combustion method shall be used as referee method in case of a dispute.

6.1.2 The results obtained include the carbon in the carbonates and the hydrogen combined in the moisture and in the water of hydration of silicates. A determination of carbon dioxide may be carried out in accordance with **5** of IS 1350 (Part 5) and the total carbon value corrected for the presence of mineral carbonates. Similarly, determination of moisture is carried out at the same time in accordance with **6** of IS 1350 (Part 1) and appropriate correction is applied to the hydrogen value obtained by combustion.

6.2 Method A – Fentons Method developed by Central Institute of Mining & Fuel Research (CIMFR)

6.2.1 *Principle*

The sample is burnt in a current of oxygen. All the hydrogen is converted into water and all the carbon to carbon dioxide. The products are absorbed by suitable reagents and determined gravimetrically. Oxides of sulphur are retained by lead chromate, chlorine by silver gauze and oxides of nitrogen by red lead.

6.2.2 *Apparatus*

An assembly of the apparatus is shown in Fig. 1. Details are given below.

6.2.2.1 *Metering and purification train* — for regulating the flow of oxygen and absorbing water vapour and carbon dioxide present in oxygen. Assemble the train in the order stated:

a) Calibrated U-tube type flowmeter, for measuring gas flow which is about 200 ml to 250 ml per minute;
b) Bubbler containing concentrated sulphuric acid (*see* IS 266), for absorbing water;
c) Bubbler, empty. For retaining any acid which may come from (b) above;
d) U-tube containing soda-asbestos, for absorbing carbon dioxide; and
e) U-tube containing anhydrous magnesium perchlorate (anhydrone), for absorbing the water evolved in the reaction between carbon dioxide and soda-asbestos.

NOTE — The purification train should be large enough to render frequent charging unnecessary even with continuous use.

6.2.2.2 *T-piece inlet* — made of fused silica or hard glass

6.2.2.3 *Pusher rod* — made of fused silica with a disc-end 12 mm in diameter for pushing the boat into the furnace

6.2.2.4 *Combustion device* — consisting of the following and arranged as shown in Fig. 2:

a) *Furnaces*

The combustion device is heated by two furnaces, the lengths of which are given below for a combustion tube 80 cm in length. These are constructed by winding nichrome wire of diameter 0.38 mm on silica tube of internal diameter 25 mm and wall thickness 1 mm.

1) Furnace No. 1 — 35 cm in length, to heat the boat and its contents and the platinum gauze (*see* Fig. 2) to a temperature of 800 ºC to 900 ºC .

2) Furnace No. 2 — 23 cm in length, to heat the copper gauze, lead chromate, red lead and silver gauze (*see* Fig. 2) to a temperature of 550 °C up to a length of 9 cm and providing a temperature gradient from 550 ºC to 250 °C along the remaining 14 cm such that in the last 9 cm of length temperature is in the range of 350 ºC to 250 ºC .

NOTE — If the supply voltage is 230 V, 50 cps (cycle per second), the control of temperature is conveniently affected by 1 kW energy regulator.

b) *Combustion tube*

It is made of fused silica. For a combustion tube 80 cm long and 19 mm in internal diameter, the following arrangement of packing (*see* Fig. 2) beginning at the end connected to the purification train is suitable.

Arrangement of Packing	*Length*, cm
(1)	(2)
Space for stopper	1
Blank space	30
Plug of platinum gauze	6
Blank space	15
Roll of copper gauze	1
Packing of fused granular lead chromate	8
Roll of copper gauze	1
Packing of red lead	9
Packing of ignited asbestos wool	1
Roll of silver gauze	7
Space for stopper	1
Total	80

c) *Combustion boat*

Made of unglazed porcelain or fused silica, approximately 8 cm long.

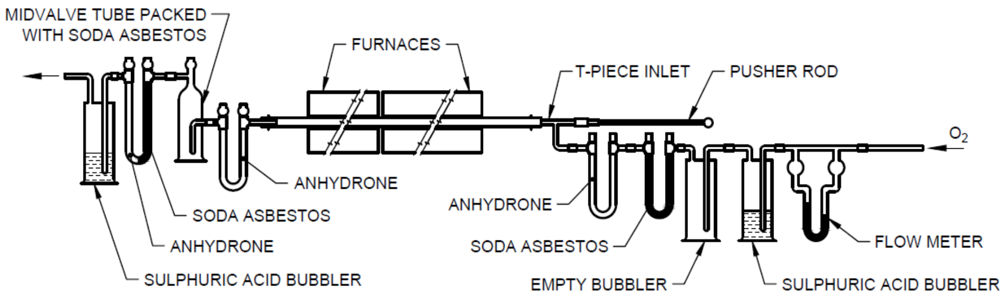


FIG. 1 ASSEMBLY OF APPARATUS

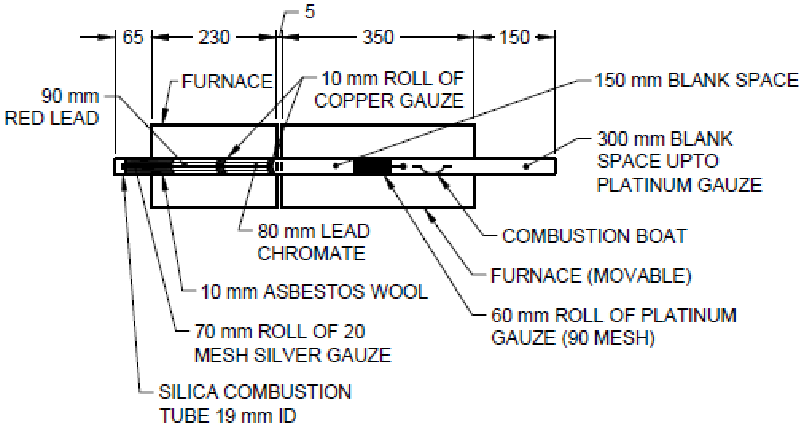


FIG. 2 DETAILS OF FURNACES AND COMBUSTION TUBE

6.2.2.5 *Absorption train* — for absorbing the water and carbon dioxide evolved by the combustion of the sample. A typical absorption train with details of the packing is shown in Fig. 1. Assemble the train using the following reagents in the order stated:

a) Anhydrous magnesium perchlorate (anhydrone), in a U-tube for absorbing the water evolved during combustion;

b) Soda-asbestos, in a midvale tube for absorbing carbon dioxide;

c) Soda-asbestos and anhydrous magnesium perchlorate (anhydrone), packed separately in two arms of a U-tube, to absorb carbon dioxide and the water produced in the reaction between carbon dioxide and soda-asbestos; and

d) Concentrated sulphuric acid, contained in a bubbler to indicate the flow of oxygen and to prevent any back flow of air to the absorbents.

6.2.2.6 *Balance* — sensitive to 0.1 mg

6.2.3 *Reagents*

6.2.3.1 *Concentrated sulphuric acid* — of relative density 1.84 (*see* IS 266)

6.2.3.2 *Soda-asbestos* — size range 0.7 mm to 1.2 mm, preferably of self-indicating type

6.2.3.3 *Anhydrous magnesium perchlorate (anhydrone)* — free from dust and within the size range 0.4 mm to 2.4 mm, preferably within the size range 0.7 mm to 1.2 mm.

CAUTION — Regeneration of magnesium perchlorate should not be attempted owing to the risk of explosion. When exhausted, the magnesium perchlorate should be washed down the sink with a current of water.

6.2.3.4 *Platinum gauze* — made of platinum or platinum-rhodium alloy, 180 micron

6.2.3.5 *Copper gauze* — 425 micron

6.2.3.6 *Pure silver gauze* — approximately 710 micron

6.2.3.7 *Oxygen* — hydrogen-free. Oxygen shall be prepared preferably from liquid air and not by electrolysis. Electrolytically prepared oxygen shall be passed over red-hot copper oxide before use to remove any trace of hydrogen.

6.2.3.8 *Lead chromate* — fused, size range 0.7 mm to 1.2 mm

6.2.3.9 *Red lead* — prepared by making a paste of lead oxide with water, flattening it out on a clock-glass, drying at 110 °C, cutting it into small cubical pieces and then heating at 400 °C till fully covered with red lead.

6.2.4 *Setting up the Apparatus and Blank Determination*

6.2.4.1 *General*

In setting up the assembly, use glass to-glass joints and, as far as possible, a minimum of rubber connections to avoid leakage of gas. Grease used for lubricating should have low vapour pressure.

6.2.4.2 Before starting a determination with a newly packed combustion tube, heat the tube throughout its length as stated in **6.2.2.4(a)**, while passing a current of oxygen at a rate of about 200 ml per minute, for a few minutes.

6.2.4.3 *Blank determinations*

For a blank determination connect the absorption train to the apparatus and purge it with oxygen for

20 min at the rate of 200 ml per minute with the furnaces at their working temperatures. Disconnect the absorbers from the train, stopper the side arms and close the taps and allow them to cool to the balance room temperature. Wipe each absorber with a clean cloth, remove the stoppers from the side arms and weigh. Re-connect the absorption train to the combustion tube and reopen the taps, particular care being taken to ensure that the inlet of the water-absorption U-tube of the absorption train is flush with the inner end of the rubber stopper in the combustion tube and that all joints between the component parts of the absorption train are made by bringing the ends of tubes close together using the rubber tubing merely as a seal. After the absorption train has remained attached to the apparatus for 15 min, stop the oxygen flow, remove and weigh the absorbers as before, observing the same precautions as in the initial weighing. The difference in the mass of the anhydrone tube gives the blank value for moisture for 15 min (at 200 ml per minute flow of oxygen); and the difference in the masses of the midvale tube and the guard tube added together gives the blank value for carbon dioxide.

6.2.5 *Procedure*

When starting the first determination on each day, raise the temperatures of the furnaces as given in 6.2.2.4(a) while passing oxygen at the rate of 200 ml per minute. Connect the absorption train and purge for 20 min. Stop the oxygen flow, disconnect the train and weigh absorbers as described in 6.2.4.3. Before commencing the determination, mix thoroughly for at least 1 min preferably by mechanical means the air-dried sample of coal, ground to pass 212 micron IS sieve. Re-expose a small portion of the sample to the air for a few hours and again mix before weighing out the quantity required for the determination.

> **IMPORTANT** — A determination of moisture shall be made immediately on the remainder of this small sample.

6.2.5.1 Weigh accurately 0.2 g to 0.25 g of the sample and spread it evenly over the bottom of a previously ignited boat. Cover it with folded platinum gauze. Connect the weighed absorption train to the combustion tube as described above and transfer the boat quickly to the combustion tube. Turn on the oxygen supply slowly. After 30 s to 40 s increase the flow to 200 ml per minute and push the boat forward slowly by the pusher rod so that the other end of the boat just enters the high temperature furnace, the sample catches fire. After 2 min to 3 min of combustion, push the boat further ahead by stages so that in 6 min to 7 min, the rear end of the boat just enters the furnace. In about 30 s, the boat is pushed into the central and the hottest part. Though the combustion should be complete by this time, allow another 7 min to ensure decomposition of carbonates and removal of the products of combustion to the absorbers. Turn off the oxygen supply, detach the absorbers from the train, allow to cool to the balance room temperature and weigh as stated in **6.2.4.3**.

> NOTE — On cooling, the pressure of oxygen inside the absorbers falls below atmospheric pressure. To equalize this, connect the train to the oxygen supply to fill the absorbers with oxygen. Alternatively, open the absorbers to air momentarily. This introduces to measurable error.

6.2.5.2 Carefully inspect the ash in the boat for unburnt carbon; if it is present, it shall invalidate the determination which shall then be repeated.

6.2.6 *Calculation*

Calculate the percentage of carbon and hydrogen by mass, as follows:

a) $C = 27.29 \times \frac{m_2}{m_1}$; and

$$C_1 = C - (0.273\ CO_2)$$

b) $H = 11.19 \times \frac{m_3}{m_1}$; and

$$H_1 = H - (0.111\,9 \times M) - (0.1\,A \times 0.111\,9)$$

where

C = total carbon in the sample as analyzed, expressed as a percentage;

m_2 = increase in mass, in g, of the soda-asbestos tube;

m_1 = mass, in g, of the sample taken for the test;

C_1 = total carbon less that present as carbon dioxide, expressed as a percentage;

CO_2 = carbon dioxide in the sample (*see* **6.1.1**), expressed as a percentage;

H = total hydrogen in the sample as analyzed, expressed as a percentage;

m_3 = increase in mass, in g, of the magnesium perchlorate tube;

H_1 = total hydrogen less that present as moisture, expressed as a percentage;

M = moisture in the sample as analyzed, expressed as a percentage; and

A = percentage of ash in coal sample as fired.

> NOTE — The factor 0.1 *A* is the correction for the water of hydration associated with mineral matter in coal. This,

however, varies from coal to coal; the average proportion being 10 percent of ash in the case of Indian coal. For precise estimation it is necessary to determine water of hydration, particularly in the case of high ash coals.

6.3 High Temperature Combustion Method

6.3.1 *Principle*

The sample is burnt in a rapid current of oxygen at a high temperature in an impervious tube, all the hydrogen is converted to water and all the carbon to carbon dioxide. These products are absorbed by suitable reagents and determined gravimetrically. Oxides of sulphur and chlorine are retained by a silver gauze roll at the outlet end of the tube.

6.3.2 *Apparatus*

6.3.2.1 *Purification trains* — two, first for absorbing water vapour and carbon dioxide from the oxygen used for the combustion and the other for similarly treating the air used for sweeping out the absorption train before and after a determination. Assemble each train as mentioned below:

a) *Anhydrous magnesium perchlorate* (*anhydrone*) — for absorbing water;
b) *Soda-asbestos* — for absorbing carbon dioxide; and
c) *Anhydrous magnesium perchlorate* (*anhydrone*) — for absorbing the water evolved in the reaction between carbon dioxide and soda-asbestos.

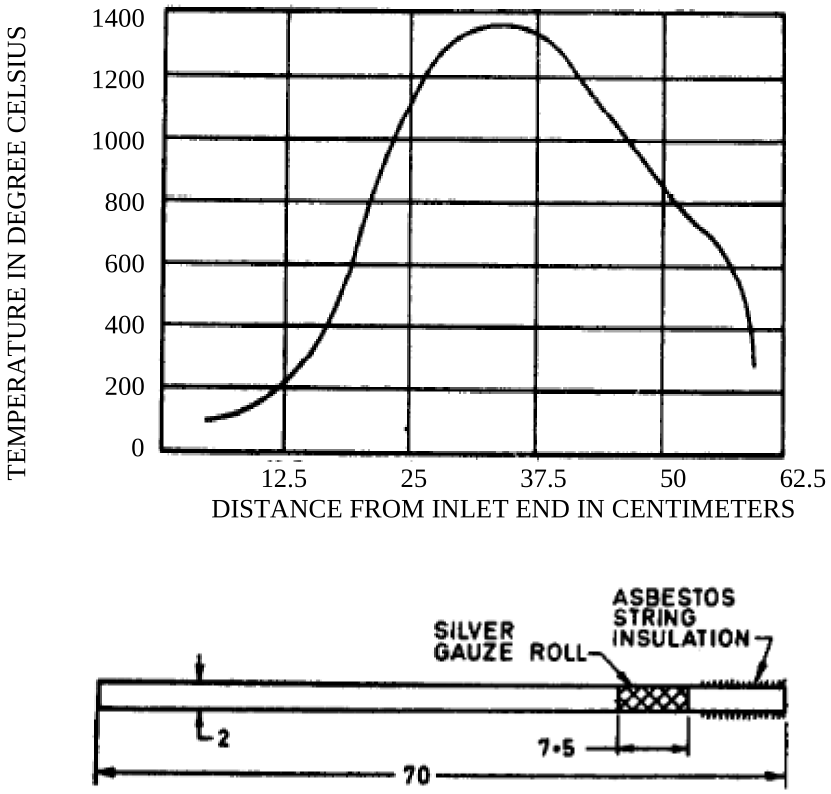
NOTE — The purification train should be large enough to render frequent recharging unnecessary even with continuous use.

6.3.2.2 *Combustion device* — consisting of the following:

a) *Furnace* — capable of carrying a tube approximately 25 mm outside diameter and heating it over a length of approximately 12.5 cm to over 1 250 °C and up to a maximum of l 350 °C. Suitable furnaces are as follows:
    1) molybdenum or tungsten wire wound;
    2) platinium or platinum-rhodium wire wound; and
    3) heated by silicon carbide rods.

A temperature-distribution curve for a typical furnace is shown in Fig. 3.

NOTE — Furnace of the type normally used for the determination of carbon or sulphur in steel are not suitable because of the absence of the auxiliary section to maintain the silver gauze roll at the correct temperature.



COMBUSTION TUBE

All dimensions in the millimeters.

FIG. 3 TYPICAL TEMPERATURE — DISTRIBUTION CURVE FOR FURNACE

b) *Combustion tube* — approximately 20 mm internal diameter and approximately 70 cm long, made of refractory aluminous porcelain which is not permeable to gases at 1 400 °C.

c) *Combustion boat* — of iron-free unglazed porcelain approximately 6 cm long and 12.5 mm wide and 10 mm deep, capable of withstanding a temperature of 1 350 °C.

NOTE — Boats should not blister, discolour or change in mass on heating in oxygen at 1 350 °C for 3 h. A suitable boat may last for about 10 to 20 determinations and should then be discarded because of the accumulation of fused ash. For high ash coals, it may be convenient to line the boat with alumina before adding the sample, in order to prevent fusion of the ash to the boat.

6.3.2.3 *Absorption train* — for absorbing the water and carbon dioxide evolved by the combustion of the sample. A typical absorption train, with details of packing is shown in Fig. 4. Midvale tubes (*see* Fig. 5), which provide a large area of reaction, are used to reduce the back pressure in the apparatus and to avoid the danger of leakage through the rubber sleeve carrying the pusher. Assemble the train (*see* Fig. 4) using the following reagents in the order stated:

a) *Anhydrous magnesium perchlorate* (*anhydrone*) — for absorbing the water evolved during the combustion (*see A* in Fig. 4);

b) *Soda-asbestos* — for absorbing carbon dioxide (*see* B and C in Fig. 4); and

c) *Anhydrous magnesium perchlorate* (*anhydrone*) — for absorbing the water evolved in the reaction between carbon dioxide and soda-asbestos (*see C* in Fig. 4).

NOTE — Cotton wool is placed above the absorbents to prevent carryover of dust by the rapid flow of oxygen and the cracking of the soda-asbestos tube by the heat of reaction. *A* is the absorber for water, *B* and *C* are the absorbers for carbon dioxide, *C* serving as a control to indicate when the packing in *B* is in need of replacement. Any water produced in *B* by the reaction between soda-asbestos and carbon dioxide is absorbed in *C*.

6.3.2.4 *Flow meters* — two, first capable of measuring rates of flow up to 300 ml per minute and the other to 250 ml per minute.

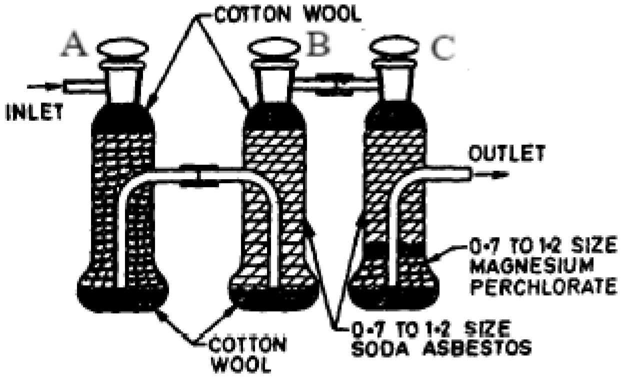
6.3.2.5 *U-gauze* — to measure the resistance of the system.

NOTE — The normal back pressure on the system is 50 mm to 79 mm H_2O.

6.3.2.6 *Heat-resisting stopper* — made of acrylonitrile or chloroprene for connecting the absorption train to the combustion tube.

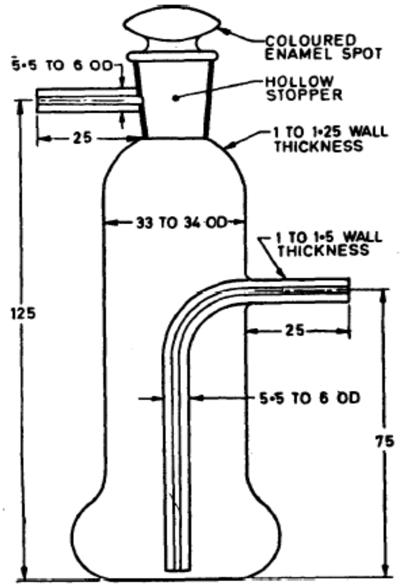
6.3.2.7 *A silica pusher* — with a disc end 12 mm in diameter for pushing the boat into the furnace. It is about 45 cm long, made from 6 mm diameter silica rod flattened at one end, or from a 6 mm diameter silica tube sealed and flattened similarly. The pusher passes loosely through a glass T-piece, one end of which fits into the rubber stopper which closes the inlet end of the combustion tube, the other being sealed with a rubber sleeve through which the pusher slides. Oxygen is admitted through the limb of the T-piece. The pusher is marked from the disc-end for convenience in ascertaining the position of the boat in the combustion tube during pushing.

NOTE — This sleeve should be changed periodically to avoid loss by leakage.



All dimensions in the millimeters.

FIG. 4 ABSORPTION TRAIN



All dimensions in the millimeters.

FIG. 5 MIDVALE TUBE

6.3.2.8 *Balance* — sensitive to 0.1 mg

6.3.3 *Reagents*

6.3.3.1 *Anhydrous magnesium perchlorate* (*anhydrone*) — same as in **6.2.3.3**

6.3.3.2 *Soda-asbestos* — same as in **6.2.3.2**

6.3.3.3 *Alumina* — finely divided approximately 0.1 mm in size

6.3.3.4 *Sodium borate solution* — 0.05 N approximately

6.3.3.5 *Hydrogen peroxide* — 1 percent (*v*/*v*) neutral solution

6.3.3.6 *Pure silver gauze* — same as in **6.2.3.6**

6.3.3.7 *Oxygen* — same as in **6.2.3.7**

6.3.4 *Preparation of the Apparatus*

6.3.4.1 *Preparation of combustion tube*

Insert the combustion tube into the furnace so that it projects 7.5 cm at the exit end. Wrap this projecting portion with asbestos string to prevent condensation of water in the tube. Insert the rubber stopper carrying the silica pusher at the inlet end of the combustion tube and connect the purified oxygen supply to the limb of the glass T-piece.

6.3.4.2 *Preparation and location of silver gauze*

The roll of silver gauze through which passes a stout silver wire provided with a loop for convenience of withdrawal, should be 7.5 cm long and of sufficient diameter to ensure a close sliding fit in the combustion tube. Insert the silver gauze roll into the exit end of the combustion tube to a distance of about 7.5 cm. With the furnace at its working temperature of 1 350 °C, connect a bubbler containing solution of the hydrogen peroxide to the exit end of the combustion tube by means of a heat-resisting stopper. Burn 0.5 g of coal containing 1 percent to 2 percent of sulphur by the procedure described below. Determine the amount of oxides of sulphur passing the silver gauze roll by titration of the hydrogen peroxide against the sodium borate solution. Move the silver gauze roll in 5 mm stages and repeat the test until a position is found where the volume of the sodium borate solution corresponding to the end point is not more than 0.2 ml, which will represent at least 99 percent retention of the oxides of sulphur by the silver gauze roll. Note its exact location.

NOTE — The silver gauze roll can absorb sulphur equivalent to 7 percent of the sample, for higher sulphur contents, the mass of the coal sample should be proportionately reduced. The roll should be periodically cleaned by boiling in water (to remove silver sulphate), in concentrated ammonia solution (to remove silver chloride) and finally in water, after which it should be dried. The total sulphur content of the samples examined between two cleanings of the roll should not exceed 0.035 g.

6.3.5 *Procedure*

6.3.5.1 Raise the furnace to its working temperature of 1 350 °C and confirm that the silver gauze roll is in its correct position. Admit oxygen to the apparatus about 30 min before the temperature reaches 1 350 °C and adjust the rate of flow to 300 ml/min. Before commencing the determination, mix thoroughly for at least 1 min, preferably by mechanical means the air-dried sample of coal, ground to pass 212-micron IS sieve. A small portion of the sample should be re-exposed to air for a few hours and again mixed before weighing the quantity required for the determination.

> **IMPORTANT** — A determination of moisture shall be made immediately on the remainder of this small sample.

6.3.5.2 Spread about 0.5 g of the sample evenly over the bottom of a dried boat and weigh accurately. Cover with about 0.5 g of alumina (with care 0.5 g of alumina can be made to blanket completely the sample in the boat). Connect a previously weighed absorption train and insert the charged boat into the combustion tube to a position such that its center is 22.5 cm from the center of the hottest zone. With the silica pusher fully withdrawn, replace the rubber stopper and continue to pass oxygen at a rate of 300 ml/min. At the end of each of the next six one-minute periods push the boat forward about 3.75 cm, withdrawing the silica pusher each time to prevent distortion, after the last push the center of the boat should be at the center of the hottest zone.

NOTES

1 Certain coals which liberate volatile matter at a high rate in the early stages of heating may give a carry over of carbon particles. For such coals, the rate of pushing should be reduced according to the following procedure:

Insert the charged boat so that its center is 22.5 cm from the center of the hottest zone. At the end of the first minute, move the boat forward 38 mm. At the end of each of the next 8 min move the boat forward by 19 mm, and at the end of tenth minute move the boat forward by 38 mm. Allow the boat to remain in the hottest zone for a further 4 min.

2 For unreactive cokes, that is, those with less than 0.5 percent of volatile matter, the rate of pushing should also be reduced as described above.

6.3.5.3 Allow the boat to remain in the hottest zone for a further 4 min. Disconnect the absorption train, connect it to the purification train and purge it for 10 min with purified air at the rate of 200 ml/min to 250 ml/min. Disconnect the absorbers *A*, *B* and *C*, close the taps and stopper the central side arms and allow to cool.

NOTE — Midvale tubes used for the absorption of carbon dioxide cool slowly and 60 min should be allowed before weighing.

6.3.5.4 Wipe and remove the guard stoppers and weigh the absorbers.

NOTE — When not connected to the apparatus, the midvale tubes should be protected from atmospheric contamination by closing the taps and fitting guard stoppers of plugged rubber tubing to the open limbs. It is usual to weigh the absorption tubes without guard stoppers, after wiping.

6.3.5.5 A bent nickel-chromium wire may be used to withdraw the heated boat, which is conveniently pulled on to a thick sheet of asbestos.

NOTE — The total moisture in the alumina is determined by heating 1 g to 1 350 ºC in the apparatus and absorbing the water in magnesium perchlorate. Drying of alumina at 108 °C is unsuitable, since the water m the alumina is not liberated below 1 000 °C. Only one or two determinations of moisture need be carried out on each bottle of alumina.

6.3.6 *Calculation*

Calculate the percentage of carbon and hydrogen by mass, as follows:

a) $C = 27.29 \times \frac{m_2}{m_1}$; and

$C_1 = C - 0.273\ CO_2$

b) $H = 11.19 \times \frac{m_3}{m_1}$; and

$H_1 = H - (0.111\,9 \times M) - (0.1\ A \times 0.111\,9)$

where

C = total carbon in the sample is analyzed, expressed as a percentage;

m_2 = increase in mass, in g, of absorbers *B* and *C*;

m_1 = mass, in g, of the sample taken for the test;

C_1 = total carbon less that present as a carbon dioxide, expressed as a percentage;

CO_2 = carbon dioxide in sample (*see* **6.1.1**) expressed as a percentage;

H = total hydrogen in the sample as analyzed, expressed as a percentage;

m_3 = increase in mass, in g, of absorber *A*, less the moisture in the alumina used;

H_1 = total hydrogen less that present as moisture, expressed as a percentage;

M = moisture in the sample as analyzed, expressed as a percentage; and

A = percentage of ash in coal sample as burnt (*see* **6.2.6**).

7 EXPRESSION OF RESULTS AND PRECISION OF DETERMINATION

7.1 Expression of Results

The results, preferably the mean of duplicate determinations, shall be reported to the nearest 0.1 percent for the carbon determination and to the nearest 0.01 percent for the hydrogen determination.

7.2 Precision of Determination

7.2.1 *Repeatability*

The maximum acceptable difference between the results of duplicate determinations carried out at different times in the same laboratory by the same operator with the same apparatus on representative portions taken from the same analysis sample shall not differ by more than as follows:

For carbon ± 0.25 percent absolute, and
For hydrogen ± 0.12 percent absolute.

7.2.2 *Reproducibility*

The maximum acceptable difference between the mean of the results of duplicate determinations carried out in one laboratory and the mean of the results of duplicate determinations carried out in any other laboratory on representative portions from the same analysis sample shall not differ by more than as follows:

For carbon ± 0.6 percent absolute, and
For hydrogen ± 0.25 percent absolute.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Solid Mineral Fuels and Solid Biofuels Sectional Committee, PCD 07

Organization	*Representative(s)*
CSIR - Central Institute for Mining and Fuel Research, Dhanbad	Prof Arvind Kumar Mishra **(*Chairperson*)**
Bharat Coking Coal Limited, Dhanbad	Shri Samir Kumar Kundu Shri G. Venugopal (*Alternate*)
Bharat Heavy Electricals Limited, Tiruchirappalli	Dr Sivaji Seepana Shri Manda Teja Tarakesh (*Alternate* I) Shri Aman Seraphim Surin (*Alternate* II)
Cement Manufacturers Association, New Delhi	Shri Raju Goyal Shri Vipul Singh (*Alternate*)
Central Electricity Authority, New Delhi	Shri S. K. Thakur Shri L. D. Papney (*Alternate*)
Central Mine Planning and Design Institute Limited, Ranchi	Shri N. Ramakrishna Ms Zeba Imam (*Alternate*)
Central Pollution Control Board, New Delhi	Shri Nazimuddin
Central Revenue Control Laboratory, New Delhi	Shri V. Suresh Dr Mahesh Kumar (*Alternate*)
Coal Controller Organization, Kolkata	Shri Anjani Kumar
Coal India Limited, Kolkata	Shri Subhasis Sahu
CSIR - Central Institute for Mining and Fuel Research, Dhanbad	Dr Ashok Kumar Singh Dr Sujan Saha (*Alternate*)
CSIR - Institute of Minerals and Materials Technology, Bhubaneswar	Dr Suddhasatwa Basu Dr D. S. Rao (*Alternate* I) Dr Manish Kumar (*Alternate* II)
CSIR - National Metallurgical Laboratory, Jamshedpur	Dr Sanchita Chakravarty Ms Rupa Das Biswas (*Alternate*)
Geological Survey of India, Kolkata	Dr Sudip Bhattacharyya
Hindalco Industries Limited, Ranchi	Shri Mirajul Haque Dr Rahul Mitra (*Alternate*)
Indian Council of Agricultural Research, New Delhi	Dr Panna Lal Singh
Indian Institute of Technology (ISM), Dhanbad	Dr S. Bhattacharya
Indian Metals and Ferro Alloys Limited, Bhubaneswar	Shri Dinesh Kumar Mohanty (*Alternate*)

Organization	*Representative(s)*
Mahanadi Coal Field Limited, Burla	Shri Ravikesh Kumar Raju Shri Neeraj Kalla (*Alternate*)
Ministry of Coal, New Delhi	Shri Anindya Sinha Shri Piyush (*Alternate*)
Ministry of Environment Forest and Climate Change, New Delhi	Shri N. Subrahmanyam
Ministry of New and Renewable Energy, New Delhi	Shri Hiren Chandra Borah Shri Vikram Dhaka (*Alternate*)
National Council for Cement and Building Materials, Faridabad	Shri Ankur Mittal Shri Gaurav Bhatnagar (*Alternate*)
National Test House, Kolkata	Shri S. K. Laha Shri Ratul Bezbaruah (*Alternate*)
NLC India Limited, Chennai	Shri R. Shanmugasundaram Shri G. Thirumurugan (*Alternate*)
Northern Coalfields Limited, Singrauli	Shri Satish Jha
Steel Authority of India Limited (SAIL), Research & Development Centre for Iron & Steel, Ranchi	Shri K. K. Manjhi Shri B. Chakraborty (*Alternate*)
Tata Power Limited, Mumbai	Shri Pravash Sinha
Tata Steel Limited, Kolkata	Shri B. K. Singh Shri H. N. Prasad (*Alternate*)
The Energy and Resources Institute, New Delhi	Shri Sunil Dhingra Shri N. K. Ram (*Alternate*)
The Singareni Collieries Company Limited, Kothagudem	Shri Syed Habeeb Hussain
In Personal Capacity (*A-304, 1st Floor, Defence Colony, New Delhi*)	Shri S. K. Grover
In Personal Capacity (*687, Baishnabghata Patuli, Ghoshpara Xing On E M Bypass, Kolkata*)	Dr Kalyan Sen
BIS Directorate General	Shrimati Meenal Passi, Scientist 'F'/ Senior Director and Head (Petroleum Coal and Related Products) [Representing Director General (*Ex-officio*)]

Member Secretary
Ms Aditi Choudhary
Scientist 'B'/Assistant Director
(Petroleum, Coal and Related Products), BIS

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