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IS 1350-1 (1984): Methods of Test for Coal and Coke, Part I: Proximate Analysis [PCD 7: Solid Mineral Fuels]



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“Knowledge is such a treasure which cannot be stolen”

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IS : 1350 (Part 1) - 1984
(Reaffirmed 2002)

Indian Standard
METHODS OF TEST FOR COAL AND COKE
PART I PROXIMATE ANALYSIS
(*Second Revision*)

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

Indian Standard

METHODS OF TEST FOR COAL AND COKE

PART 1 PROXIMATE ANALYSIS

(Second Revision)

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(Continued on page 2)

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IS : 1350 (Part 1) - 1984

(Continued from page 1)

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**AMENDMENT NO. 1 AUGUST 1992
TO
IS 1350 (Part 1) : 1984 METHODS OF TEST FOR COAL
AND COKE**

PART 1 PROXIMATE ANALYSIS

(Second Revision)

(Page 25, clause 8.2.1) — Insert the following after 8.2.1:

'8.2.1.1 For coals containing more than 4 percent carbon dioxide, the final temperature shall be $850 \pm 10^{\circ}\text{C}$.'

(PCD 7)

Indian Standard

METHODS OF TEST FOR COAL AND COKE

PART 1 PROXIMATE ANALYSIS

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Part 1) (Second Revision) was adopted by the Indian Standards Institution on 7 March 1984, after the draft finalized by the Solid Mineral Fuels Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was originally published in 1959 as IS : 1350 and was split into 3 parts during its first revision. Part 1 covering proximate analysis was issued in 1969. Later, with the perfection of a rapid method of determining moisture at 60 percent relative humidity and 40°C (*see 6.7.2*) by the Central Fuel Research Institute (CSIR), Dhanbad, the committee responsible for the preparation of the standard decided to revise it again. In the second revision, expression of the precision of results has been spelt out more clearly, and the basis for reporting results, evaluation of mineral matter and the format for reporting of test results have also been included to make the standard more comprehensive.

0.3 The methods of test for coal and coke are now covered in the following five parts:

Part 1 Proximate analysis

Part 2 Determination of calorific value

Part 3 Determination of sulphur

Part 4 Ultimate analysis:

Section 1 — Determination of carbon and hydrogen

Section 2 — Determination of nitrogen

Part 5 Special Impurities

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (*revised*).

IS : 1350 (Part 1) - 1984

1. SCOPE

1.1 This standard (Part 1) prescribes the methods of test for coal and coke relating to proximate analysis including determination of moisture under different conditions.

2. TERMINOLOGY

2.1 For the purpose of this standard the definitions given in IS : 3810 (Part 2)-1977* shall apply.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977†) shall be employed in these tests.

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

4. SAMPLING

4.1 Methods of Sampling — Methods of sampling shall be as prescribed in IS : 436 (Part 1/Section 1)-1964‡ for coal and IS : 436 (Part 2)-1965§ for coke.

4.2 Preparation of Sample for Test

4.2.0 General — It is expected that methods of sampling prescribed in IS : 436 (Part 1/Section 1)-1964‡ and IS : 436 (Part II)-1965§ would have been followed in the preparation of samples sent to the laboratory. Besides, the laboratory samples to be used for the analysis of coal and coke, special samples are recommended for moisture.

4.2.1 The samples prepared in accordance with IS : 436 (Part 1/Sec 1)-1964‡ and IS : 436 (Part 2)-1965§ shall be received in sealed containers and shall consist of about 300 g of coal or coke ground to pass 212-micron IS sieve.

4.2.2 Where air-drying has been adopted in the preparation of the samples, the percentage loss of moisture in this operation shall be recorded on the label together with the relevant Indian Standard method of sampling used.

4.2.3 Samples received in the laboratory if already ground to pass 212-micron IS sieve shall be re-sieved to verify that all the material passes

*Glossary of terms relating to solid mineral fuels: Part 2 Terms relating to coal sampling and analysis.

†Specification for water for general laboratory use (second revision).

‡Methods for sampling of coal and coke : Part 1 Sampling of coal Section 1 Manual sampling (revised).

§Methods for sampling of coal and coke : Part 2 Sampling of coke (revised).

through this sieve, and then air-dried for 24 hours and mixed and bottled as above. Should a sample of coal or coke be transported to any distance between the point of preparation and the laboratory under conditions in which it may be shaken down and may cause segregation to any degree, the entire contents of the bottle shall be re-mixed before analytical work is started. The normal method of mixing of coal in the laboratory is to roll it on a sheet of rubber cloth and turn it over at intervals with a large spatula or flat spoon. This method is not entirely satisfactory, since the movement of the coal tends to cause segregation. The difficulty may be overcome in part by spreading the mixed sample in a thin layer and filling the analysis bottle by means of small increments taken with the spoon from places spaced uniformly over the surface of the layer. This method of filling is particularly necessary when more than one bottle is being filled. A laboratory mechanical mixer is, however, preferable.

4.2.4 Certain coals may be found to give irregular results on analysis. In such cases, and for analytical work only, the analysis sample should be further ground to pass 125-micron IS sieve and re-mixed. This finer grinding is normally found to ensure adequate agreement between repeat determinations.

5. PROXIMATE ANALYSIS

5.1 General

5.1.1 Coals, especially those of low rank, are hygroscopic to various degrees, and absorb or lose moisture according to the humidity and temperature to which they are exposed. Coke is not so hygroscopic as some coals, but it absorbs moisture during grinding. Hence, in principle, any moisture determination which is carried out without reference to a standard humidity and temperature is, to a degree, unsatisfactory. A separate determination of moisture in the air-dried material shall be made at the same time as the ash and volatile matter are determined.

5.1.2 The results of proximate analysis are generally recorded as a percentage of the air-dried material. The ash may be expressed on 'dry' basis. Volatile matter and fixed carbon may be expressed on 'dry' basis, 'dry ash-free' basis or 'dry mineral free (dmf)' basis. Such figures are arrived at by calculation from the 'air-dried' basis results.

6. DETERMINATION OF MOISTURE

6.1 Forms and Conditions of Moisture — The moisture present in the laboratory sample of coal and coke is of importance both in proximate analysis and in calculating other analysis results. Moisture and free water may be defined as follows:

- a) *Total moisture* — The coal which has been exposed to contact with water in the seam or in a washery, or coal and coke wetted by rain, may carry free or visible water. This water plus the moisture within the material, is referred to as total moisture.

- b) *Moisture in coal equilibrated at 60 percent relative humidity and 40°C* — The moisture content of air-dried coal varies and depends upon the temperature and relative humidity of the air to which it is exposed. As such it is necessary to determine moisture content of different samples of coal under standard conditions. For this purpose, the coal is ground to pass 212-micron IS sieve and equilibrated in an atmosphere of 60 percent relative humidity and 40°C. The moisture determined under these conditions shall be taken as reference moisture for all purposes.
- c) *Moisture in coal equilibrated at 96 percent relative humidity and 40°C* — This is also termed as 'Near Saturation Moisture' or 'Bed Moisture'. It is exclusive of free or visible water and is determined after equilibrating coal in an atmosphere of 96 percent relative humidity and 40°C. This is a measure of the moisture holding capacity of a coal.
- d) *Free water or visible water* — Free water or visible water is that quantity of water which is physically adhering to coal. In essence, this is that quantity of water which is in excess of the moisture holding capacity of a coal.
- e) *Moisture in air-dried laboratory analysis sample of coal and coke* — It is the moisture in coal which has been air-dried under the laboratory atmospheric condition prior to analysis and is determined as a part of proximate analysis and also whenever portions of sample are weighed later for other analysis and tests. If all the portions for analysis are weighed under approximately the same conditions of humidity, one determination of moisture will suffice, but a check is desirable with high moisture coals.

6.2 Method — One of the following methods for determination of moisture shall be adopted:

- a) *Indirect method* — A known mass of the material is dried and the loss of mass calculated as moisture. The moisture may be determined either by drying in one stage at $108 \pm 2^\circ\text{C}$ or by a two-stage process in which the coal is first air-dried under atmospheric conditions and the remaining moisture removed by drying in an oven at $108 \pm 2^\circ\text{C}$. In the latter case the total moisture is calculated from the loss during air drying and that during oven drying. For the determination of moisture in coke, the minimum free-space oven method with a temperature of $200 \pm 5^\circ\text{C}$ and a heating period of 4 hours is employed.

The following are the indirect methods:

- i) Drying in air (one stage) — see 6.3.1 and 6.3.2;

- ii) Drying in air (two stage) — air-drying followed by oven-drying — see 6.4.1 and 6.4.2; and
 - iii) Drying in a minimum-free-space oven (for coke) — see 6.5.
- b) *Direct method* — A known mass of the material is heated in a glass tube in a current of dry nitrogen. The gas is passed through weighed moisture-absorption tubes. This method is applicable when free water is absent. This gravimetric method is covered in 6.6.

6.3 Drying in Air (One Stage) — Two different methods are employed.

6.3.1 Method I (At 200°C)

6.3.1.1 *Applicability* — This method is not applicable to coal, but it applies to a special moisture sample of 1 to 2 kg of coke, crushed to pass a round hole screen conforming to designation 16 mm PR of IS : 460-1978*, and delivered in a sealed non-corrodible container.

6.3.1.2 *Apparatus* — Ventilated air-oven, to take non-corrodible trays approximately 1 000 cm² in area and 5 cm deep. Alternatively a hot plate may be used.

6.3.1.3 *Procedure* — Weigh the sample together with the container to the nearest tenth of a gram. Empty the container on a dry weighed tray and spread the sample (about 1 to 2 kg) in an even layer not exceeding 40 mm in thickness. Dry the container in an air-oven or on hot plate maintained at about 200°C. Weigh the dry container and obtain the mass of the sample by difference. Place the tray with the sample in air-oven or hot plate maintained at the above temperature. Continue heating until the constant mass is reached. Constancy in mass is deemed to have been reached if the successive mass determinations do not differ by more than 0.05 percent from each other. Cool the tray on a metal plate and weigh while slightly warm. The initial mass of the tray with the sample is the sum of the mass of the tray and the mass of the sample as determined earlier. The difference between the mass of the tray with sample before and after heating gives the loss of moisture. Calculate on percent basis and report as moisture ' as received '.

6.3.2 Method II

6.3.2.1 *Applicability* — This method applies to laboratory samples of coal crushed to pass 212-micron IS sieve. The method is also used in determining the moisture for coal equilibrated at 60 percent relative humidity (see 6.7).

6.3.2.2 Apparatus

- a) *Air-oven* — Ventilated drying oven in which a constant and uniform temperature of 108 ± 2°C can be maintained.

*Specification for test sieves (second revision).

IS : 1350 (Part 1) - 1984

- b) *Weighing vessel* — Shallow vessel, approximately 10 cm² in area, made of silica or glass with ground edges and fitted with ground covers or of stainless steel.

6.3.2.3 Procedure — Heat the empty vessel at $108 \pm 2^\circ\text{C}$ and weigh after cooling for 20 minutes in a desiccator. Uniformly spread about 1.5 g of coal, passing 212-micron IS sieve, in the weighing vessel, so that there is not more than 0.15 g of the material per cm² area, and weigh again. Uncover the vessel in the drying oven and heat at a temperature of $108 \pm 2^\circ\text{C}$ until there is no further loss in mass. This normally takes 1 to 1.5 hours. Replace the cover, cool in a desiccator for 20 minutes and weigh.

6.3.2.4 Calculation and reporting of results — Express the loss in mass due to drying as a percentage of the total mass of the sample and report the result as the percentage moisture in the sample.

6.4 Drying in Air (Two Stage) — Air Drying Followed by Oven Drying.

6.4.1 Stage One (Air-Drying)

6.4.1.1 Applicability — This method applies to a special moisture sample of 1 kg of coal crushed to pass a square mesh screen conforming to designation 12.5 mm 'C' of IS : 460-1978* and delivered in a sealed non-corrodible container.

6.4.1.2 Apparatus — Non-corrodible trays approximately 1 000 cm² in area and large enough to hold the entire sample.

6.4.1.3 Procedure — Accurately weigh the sample and the container to the nearest 0.5 g (M). Transfer the sample from the container to the weighed metal tray and spread the material evenly on the tray. Dry the container in air and transfer to the tray any coal that was left behind adhering to the sides. Weigh the dry empty container (M_2). Record M_1 — M_2 as the mass of the sample taken for the test. Allow the material in the tray to air-dry at atmospheric temperature in a well ventilated place free from dust. Take the drying to be complete when the change in mass during an hour is less than 0.1 percent of the sample. Record this mass.

6.4.2 Stage Two (Oven-Drying)

6.4.2.1 Apparatus

- a) *Ventilated drying oven* — in which a constant and uniform temperature of $108 \pm 2^\circ\text{C}$ can be maintained.

*Specification for test sieves (*second revision*).

- b) *Weighing vessel* — shallow, approximately 40 cm² in area, made of silica, glass or stainless steel, with ground edges and fitted with ground cover.

6.4.2.2 Procedure — Heat the empty vessel at $108 \pm 2^\circ\text{C}$ and weigh after cooling for 20 minutes. Crush the air-dried material (see 6.4.1.3) to pass 2·90 mm IS sieve. Spread uniformly in the weighing vessel about 10 g of the crushed material and weigh. Heat the uncovered vessel in the drying oven at a temperature of $108 \pm 2^\circ\text{C}$ until there is no further loss in mass. This normally takes 1·5 to 3 hours. Replace the cover, cool in a desiccator for 20 minutes and weigh.

6.4.3 Calculation and Reporting of Results

6.4.3.1 Air-drying — Express the loss in mass during air-drying as a percentage of the original mass of the material. Represent this as (X).

6.4.3.2 Oven-drying — Express the loss in mass on oven-drying the sample, as a percentage of the total mass of sample placed in the oven. In this case the figure obtained (Y) represents the percentage of moisture remaining in the air-dried material.

$$\begin{array}{l} \text{Total moisture content of the} \\ \text{original sample} \end{array} = X + Y \left(1 - \frac{X}{100} \right)$$

where

X = percentage loss in mass of the original sample in air-drying, and

Y = percentage loss in mass of the air-dried sample on oven-drying.

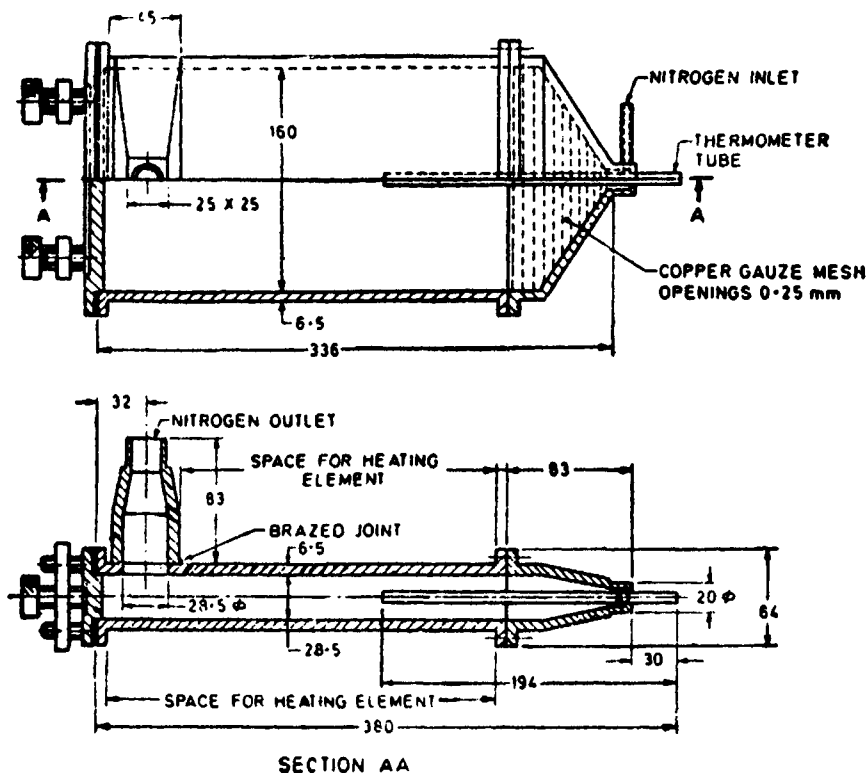
6.5 Drying in a Minimum-Free-Space Oven (for Coke)

6.5.1 Principle — The sample is heated to constant mass in a minimum-free-space oven in an atmosphere of nitrogen at 200°C and moisture calculated from the loss in mass of the coke.

6.5.2 Apparatus

6.5.2.1 Oven — A minimum-free-space oven capable of maintaining a temperature of $200 \pm 5^\circ\text{C}$ and with provision for passing nitrogen through it at a rate sufficient to change the atmosphere 15 times per hour. A suitable oven is illustrated in Fig. 1. Arrangements shall be made for measuring the flow of nitrogen through oven.

6.5.2.2 Drying tower — 250 ml capacity, packed with anhydrous calcium sulphate (- 200 to + 60 IS mesh) for drying nitrogen.



All dimensions in millimetres.
FIG. 1 MINIMUM-FREE-SPACE OVEN

6.5.2.3 Weighing vessel — a shallow vessel, of glass or silica with a ground-on cover or of corrosion-resistant metal with a well fitting cover, of such a size that the loading of the coke layer does not exceed 0.15 g per cm².

6.5.3 Reagents

6.5.3.1 Nitrogen — containing less than 10 ppm of oxygen.

6.5.3.2 Desiccant — self-indicating activated alumina or silica gel or anhydrous calcium sulphate (- 200 to + 60 IS mesh).

6.5.4 Procedure — Raise the oven to its working temperature while passing nitrogen through it at a specified rate. Weigh the clean dry empty

vessel with its cover to the nearest 0.1 mg and spread uniformly into it about 1 g of the coke sample. Weigh the covered vessel and its contents to determine the mass of sample taken. Place the cover in a desiccator and heat the uncovered vessel in an oven at a temperature of $200 \pm 5^\circ\text{C}$ until constant mass is obtained. Replace the cover, cool on a metal plate for not more than 5 minutes, transfer to a desiccator containing a metal plate and weigh when cold.

NOTE — Under the conditions described, drying should be complete in 4 hours. Constancy in mass (± 0.0003) should be established by reheating for a further period, following the same procedure for cooling and weighing.

6.5.5 Calculations — Calculate the moisture content in the analysed sample as follows:

$$\text{Percentage of moisture, } M = 100 \frac{(M_2 - M_3)}{(M_2 - M_1)}$$

where

M_2 = mass in g of the vessel plus cover and sample before heating,

M_3 = mass in g of the vessel plus cover and sample after heating, and

M_1 = mass in g of the vessel plus cover.

6.6 Gravimetric Determination

6.6.1 Applicability — This method applies to the analysis sample of 500 g of coal or coke, crushed to pass 212-micron IS sieve, delivered in an air-tight container. This method is recommended where the material is particularly liable to oxidation.

6.6.2 Apparatus — A diagrammatic arrangement of the apparatus is given in Fig. 2 with its various components as described below:

6.6.2.1 Heating device — a bath jacket, containing a solution of calcium chloride (relative density 1.245), fitted with a reflux condenser. The jacket encloses the glass retort tube and can be heated so as to give a fairly constant temperature. *Alternatively*, use an electrically heated thermostatically controlled aluminium oven.

6.6.2.2 Drying tower — of 250 ml capacity, packed with granular anhydrous calcium sulphate (– 200 to + 60 IS mesh) or other suitable desiccant. It is important that the same desiccant be used as in the absorption tubes (see 6.6.2.5) since the incoming stream of nitrogen shall be dried to exactly the same degree as the gas leaving the system.

6.6.2.3 Flowmeter — a suitable flowmeter, capable of measuring a flow rate of 100 ml per minute. If the pressure drop over a constriction is employed as a means of measuring flow rate, the manometers shall be filled with mercury or a non-volatile oil.

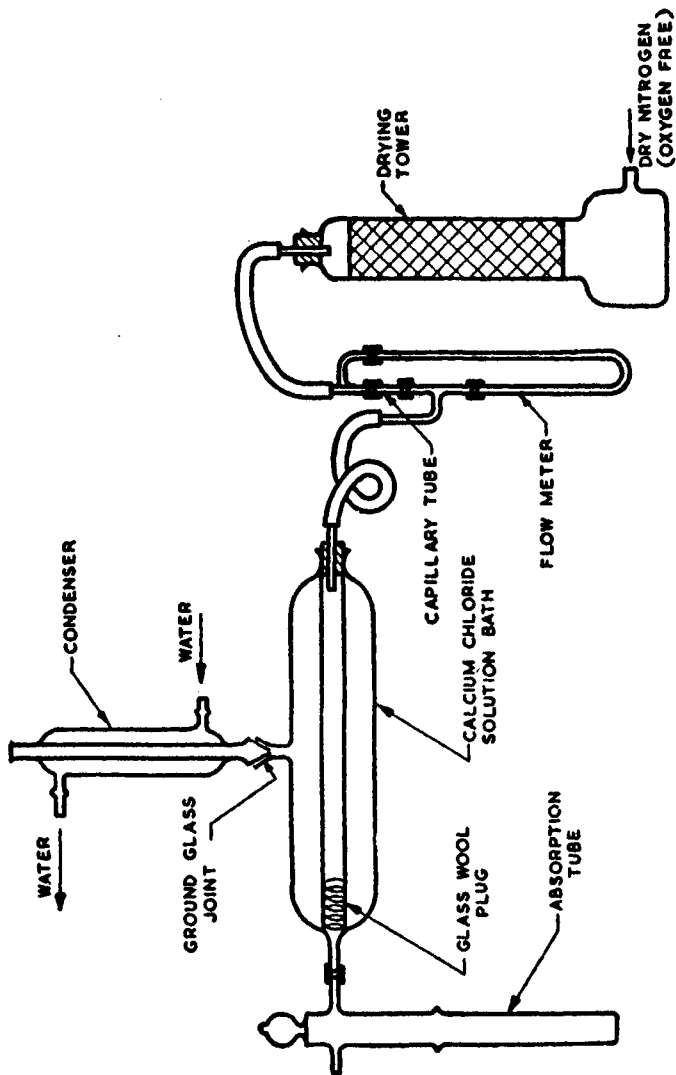


FIG. 2 APPARATUS FOR GRAVIMETRIC DETERMINATION OF MOISTURE

6.6.2.4 Retort tube — 250 × 15 mm internal diameter, made of glass, fitted with a one-holed cork for the inlet tube at one end, with other end constricted and drawn in the form of a narrow outlet tube. The inlet tube is connected to the flowmeter and terminates just inside the cork. The outlet is connected to the absorption tube. A plug of glass wool, 25 to 50 mm long is placed loosely near the constriction to entrap any solid particles of the powdered material that may be carried in the stream of nitrogen. The connections between the flowmeter and the tube are made with PVC tubing, and those between the glass to glass junctions of the retort tube and absorption tubes with rubber tubing.

6.6.2.5 Absorption tube — 200 mm in length and composed of two tubes, one inside the other (see Fig. 3). The centre tube carries plugs of glass wool at each end and the space between these is filled with desiccant (see 6.6.2.2).

6.6.2.6 Boat — made of porcelain of suitable size.

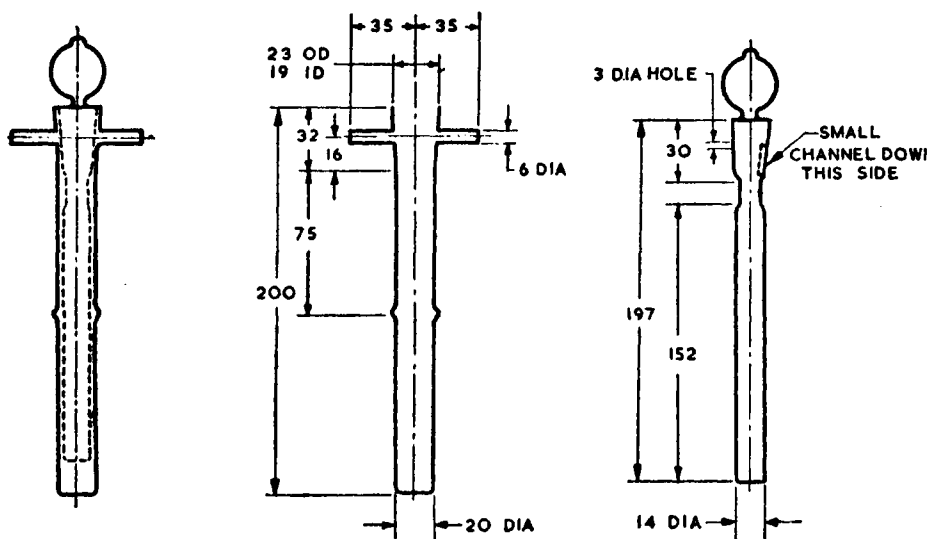


FIG. 3 ABSORPTION TUBE

6.6.3 Procedure

6.6.3.1 Pass nitrogen through the drying tower and flowmeter at 100 ml per minute. Connect the empty retort tube to an absorption tube and to the nitrogen stream from the flowmeter. With the absorption tube

tap closed, check for leaks by observing the flowmeter. Open the tap and adjust the nitrogen flow at 100 ml per minute.

6.6.3.2 Bring the calcium chloride solution in the bath jacket to boiling and continue the heating for 15 minutes for initial conditioning.

6.6.3.3 After 15 minutes, close the tap of the absorption tube, disconnect, wipe and allow to stand in balance room for 20 minutes to cool down to balance room temperature. On disconnecting the absorption tube, remove the sources of heat and allow the retort tube to cool with a stream of dry nitrogen flowing through. Weigh the absorption tube using a similar tube as a tare.

6.6.3.4 Accurately weigh about one gram of the material into a boat. Remove the bung of the glass tube to introduce the boat; replace the bung, connect the retort and absorption tube to the nitrogen stream, and check for leaks and flow rate as before.

6.6.3.5 Again bring the calcium chloride solution in the bath jacket to boiling and maintain the temperature at boiling point for 30 minutes.

6.6.3.6 Remove the absorption tube, close the taps, and condition for weighing as before. Weigh using as a tare the tube previously employed.

6.6.3.7 Carry out a blank determination once a day by passing the nitrogen through an empty retort tube and then through a weighed absorption tube, following exactly the same procedure as for a normal determination but omitting the material. In a blank determination, the increase in mass of the absorption tube shall not be more than 0.001 0 g. Deduct the amount of the blank from the mass of water obtained in each determination.

6.6.4 Calculation of Results

$$\text{Moisture, percent} = 100 \frac{(A - B)}{M}$$

where

A = increase in mass in g of absorption tube,

B = increase in mass in g during blank determination, and

M = mass in g of sample taken for the test.

6.6.5 Precision — The results of duplicate determinations shall agree within the following limits:

Moisture Percent	Precision	
	Repeatability	Reproducibility
Up to 3.0	0.1 units	0.2 units
Over 3.0	3.0 percent of the arithmetic mean of the duplicate values	6.0 percent of the arithmetic mean of the duplicate value

6.7 Moisture at 60 Percent Relative Humidity and 40°C — Either of the following two methods shall be used.

6.7.1 Reference Method — Take about 5 g of laboratory sample of coal, ground to pass 212-micron IS sieve, in a beaker or conical flask of 100 ml capacity. Pour about 20 ml of hot water on the sample and wet the coal by gently swirling the beaker or flask. Put the beaker or flask on an asbestos centered wire gauze and apply a small flame underneath, and allow the contents to boil very slowly for about 15 minutes. Filter off the water using a qualitative filter paper and then remove the visible water as far as possible by pressing the wet coal in between folds of blotting or filter paper. Take about 1.5 g of the wet coal in a previously heated, cooled and weighed silica or glass capsule with ground flange type lid and spread the coal in a uniformly thin layer so that the mass of coal per cm² in the capsule does not exceed 0.15 g. Place the uncovered capsule and the lid in a desiccator, the bottom portion of which is half filled with a solution of sulphuric acid in water to give a relative humidity of 60 percent at 40°C. Put the lid on the desiccator and leave the whole thing for 24 hours and then withdraw and break the lumps and spread the coal with a spreader, replace the lid and leave for further 24 hours. Withdraw the capsule after covering, cool it on metal plate for 20 minutes, and weigh. Note this mass and return the capsule with lid to the desiccator and leave it there for another 24 hours under the same conditions. Repeat the process of weighing and returning to desiccator till the change in mass does not exceed 0.001 g. Note the final mass. Put the weighed dishes in the air-oven maintained at $108 \pm 2^\circ\text{C}$ and determine the moisture as described in 6.3.2.3.

NOTE — The desiccator contains a mixture of one volume of concentrated sulphuric acid (relative density 1.84) and 2.9 volumes of water. The acid has a relative density of 1.284 at 30°C and yields in the desiccator a relative humidity of 60 ± 2 percent between 30°C and 40°C. Evacuate air from the desiccator to about 20 mmHg pressure during the period of equilibration. When not in use, the capsule with lid may be conveniently kept in the desiccator. The actual weighing of the coal on the balance shall be carried out quickly, if the humidity in the room differs markedly from 60 percent.

6.7.2 Rapid Method — Take about 4 g of the laboratory sample of coal ground to pass 212-micron IS sieve, in a small conical flask or pycnometer. Shake it thoroughly with about 15 ml of water and heat it to near boiling (around 80°C) on a water bath for about 30 minutes. Decant the mixture through filter paper. A mild suction with a water filter pump (or a vacuum pump) may be applied to hasten the process of filtration. Press the wet sample with pieces of filter paper to remove as much as possible of the free water, mix it thoroughly and spread evenly about 1.5 to 2 g in each of the two accurately weighed moisture dishes. Place the dishes with lids open into the humidity conditioner. Keep the conditioner in a constant temperature oven and plug its inlet and outlet for overnight. After the end of the period of about 17 to 18 hours remove the plugs and connect the conditioner in series with bubblers as shown in the diagram (see Fig. 4).

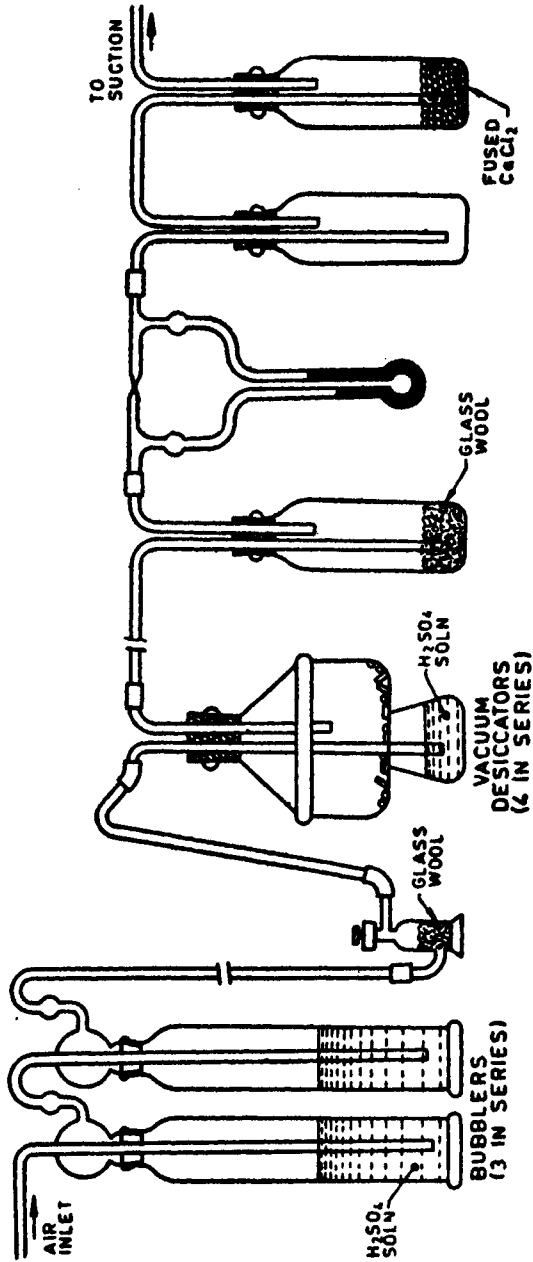


FIG. 4 APPARATUS FOR DETERMINATION OF MOISTURE BY MODIFIED RAPID METHOD

Draw a current of air through the system at the rate of 450 to 550 ml per minute for two hours. Stop the suction, close the lids of the dishes and weigh accurately. Put the weighed dishes in the air-oven maintained at $108 \pm 2^\circ\text{C}$ and determine the moisture as described in 6.3.2.3.

NOTE 1 — Humidity conditioner is a 15 cm diameter vacuum desiccator with inlet and outlet arrangements for passing air. The conditioner contains a solution having 39 percent of sulphuric acid by mass (relative density 1.277 g/cm^3 at 40°C and 1.284 g/cm^3 at 30°C). The same solution is also used in the three bubblers arranged in series. The conditioner and bubblers are kept in constant temperature oven. The solution in bubblers must be checked everyday whereas that in the conditioner may be checked twice a week by a hydrometer reading up to third place of decimal.

NOTE 2 — While applying suction during filtration polyethylene funnel should be used as ordinary glass funnels are likely to get cracked.

NOTE 3 — A set of 4 desiccators arranged in series can be used for simultaneous equilibration of 4 samples in duplicate. The experimental set-up is illustrated in Fig. 4.

6.7.3 Calculation

$$\text{Moisture in equilibrated sample, percent} = \frac{100 (M_2 - M_1)}{M_3 - M_1}$$

where

M_1 = mass in g of empty capsule plus lid,

M_2 = mass in g of capsule plus lid and equilibrated coal, and

M_3 = mass in g of capsule plus lid and dry coal.

6.7.4 Precision — shall be the same as in 6.6.5.

6.8 Moisture at 96 percent Relative Humidity and 40°C — The conditioner as used for equilibration is a desiccator type vessel containing saturated solution of potassium sulphate (with solid at the bottom). About 5-6 g laboratory coal sample is uniformly spread in a petridish or watch glass and placed in the conditioner for equilibration. The sample is equilibrated for 48 hours. The equilibrated sample is then transferred to a weighing bottle from which about 1-1.5 g coal is taken, through differential weighing of the bottle, in a moisture dish of known weight. The coal is uniformly spread in the dish so that there is not more than 0.15 g of the material per cm^2 area. Uncover the moisture dish in the drying oven and heat at a temperature of $108 \pm 2^\circ\text{C}$ until there is no further loss in mass. This normally takes about 1 to 1.5 hours. Replace the cover, cool the dish in a desiccator for about 20 minutes and weigh.

Calculate the moisture content as follows:

$$\begin{aligned} \text{Moisture, percent by mass} &= \frac{\text{Loss in mass on drying}}{\text{Mass of equilibrated sample taken in the dish}} \times 100 \\ &= \frac{M_1 - (M_2 - M_3)}{M_1} \times 100 \end{aligned}$$

where

M_1 = mass in g of the equilibrated sample taken in the dish,

M_2 = mass in g of the dish and sample after drying, and

M_3 = mass in g of the empty dish.

6.9 Free Water or Visible Water — Determine total moisture as prescribed under 6.4 and moisture at 96 percent relative humidity and 40°C (see 6.8) as *A* and *B* respectively.

$$\text{Free water or visible water, percent} = A - \frac{(100 - A) B}{(100 - B)} .$$

7. DETERMINATION OF VOLATILE MATTER

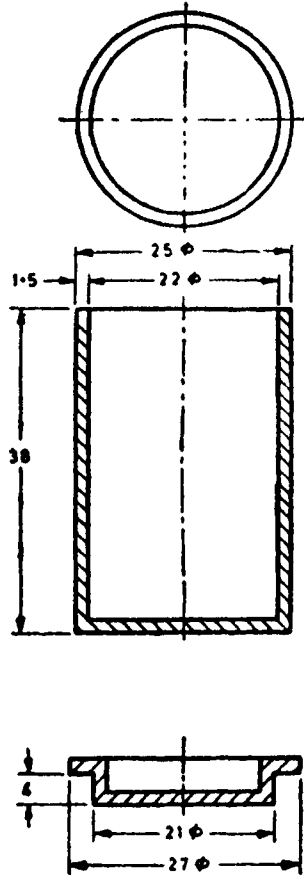
7.0 General — The volatile matter of a coal is of particular importance in assessing the use for which the coal is suitable. By itself or in conjunction with other characteristics the volatile matter on a dry mineral-free basis is used for coal classification. The volatile matter is the loss in mass less that due to moisture, when coal or coke is heated under standard conditions. The test is empirical and, in order to ensure accurate results, it is essential that the rate of heating, the final temperature and the overall duration of the test should be carefully controlled. It is also essential to exclude air from the crucible during heating to prevent oxidation and, therefore, the fit of the lid of the crucible is critical. The moisture content of the sample should be determined at the same time as the volatile matter so that the appropriate corrections could be made.

7.1 Method — The method consists of heating out of contact with air a weighed quantity of air-dried sample of coal or coke at the temperature of $900 \pm 10^\circ\text{C}$ for a period of seven minutes. Oxidation has to be avoided as far as possible; and when testing coke, 2 to 4 drops of benzene are added to the weighed material to secure a non-oxidizing atmosphere, as described in 7.4.2.

7.2 Apparatus

7.2.1 Crucible and Lid — Cylindrical crucible with a well fitting lid both of fused silica, or of other suitable ceramic material or platinum, provided that the latter two give the same result as that obtained with silica crucible.

The silica crucible and lid shall weigh 10 to 14 g and have dimensions approximating to those given in Fig. 5. The fit of the lid is a critical factor and the lid for a particular crucible shall be selected so that, when placed in position the maximum horizontal clearance at any point round the lid of the crucible is 0.5 mm. After selection each crucible and lid shall be given a distinguishing number or mark.



All dimensions in millimetres.

FIG. 5 SILICA CRUCIBLE AND LID

7.2.2 Stand — The stand on which the crucible is placed in the muffle furnace shall be such that the appropriate rate of heating can be achieved. For example it may consist of:

- a) *for single determinations* — a ring of heat-resistant steel wire as shown in Fig. 6A with two asbestos discs, 25 mm diameter and 1 mm thick, resting on the inner projection of its legs; or
- b) *for multiple determinations* — a tray of heat-resistant steel wire, as shown in Fig. 6B of appropriate size with an asbestos plate 2 mm thick, supporting the crucibles. Two determinations at a time are preferred.

7.2.3 Muffle Furnace — Gas or electrically heated in which a zone of constant and uniform temperature of $900 \pm 10^\circ\text{C}$ can be maintained. It shall be closed at the back, provided with a chimney and have at the front a well-fitting door, preferably hinged and of the plug type. The position for the crucible stand shall be chosen within the zone of uniform temperature, and this exact position shall be used in all tests. The heat capacity of the muffle furnace shall be such that, with an initial temperature of 900°C and with an empty crucible in position at least a temperature of 890°C is regained within three minutes when a charged crucible with stand is placed in its position. Normally, the furnace shall be designed specifically either for receiving one crucible and its stand at a time, or for multiple determinations using a number of crucibles (preferably two) in one stand.

NOTE — The temperature of 900°C is to be attained as closely as possible and the tolerance of $\pm 10^\circ\text{C}$ is specified so as to meet inherent errors in the temperature measurements and lack of uniformity in the temperature distribution.

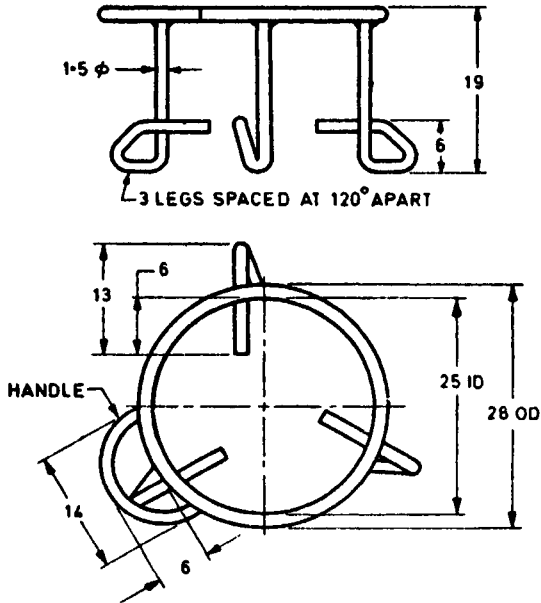
7.2.4 Pyrometer — The temperature of the working zone of the furnace are checked with an unsheathed thermocouple, of wire thicker than 1 mm. The thermojunction is inserted midway between the base of the crucible in its stand and the floor of the furnace. If the stand holds more than one crucible, then the temperature under each crucible is checked in the same manner. If desired, a sheathed thermocouple may be permanently installed in the furnace with its junction as close as possible to the centre of the zone of uniform temperature; in this case its temperature readings shall be calibrated at frequent intervals with those of unsheathed couple which is then only inserted when necessary (see Fig. 7).

NOTE — The temperature/emf relationship of a thermojunction maintained at elevated temperature gradually changes with time.

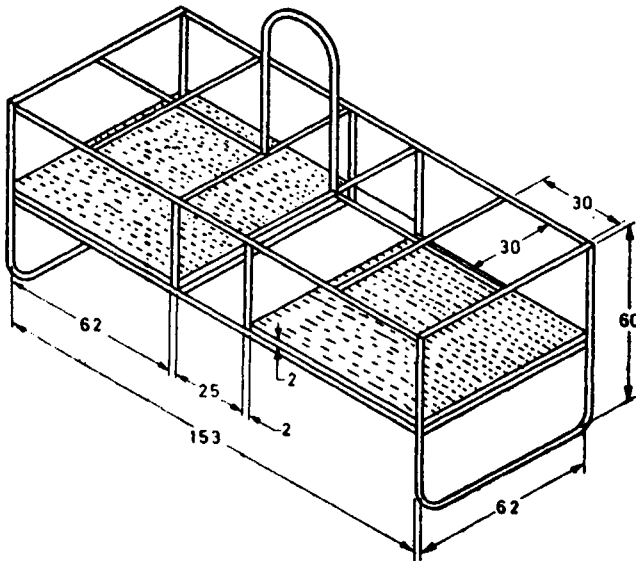
7.2.5 Asbestos Discs — 25 mm diameter, 1 mm thick.

7.3 Reagents

7.3.1 Desiccant — Fresh or freshly regenerated and self indicating silica gel. Other suitable desiccants are activated alumina or anhydrous calcium sulphate.



6A Suitable for Making One Determination at a Time



6B Suitable for Making Several Determinations at a Time
All dimensions in millimetres.

FIG. 6 CRUCIBLE STANDS

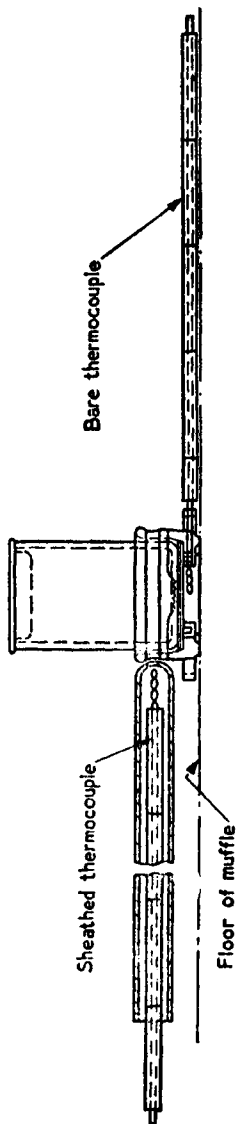


FIG. 7 LOCATION OF THERMOCOUPLE

7.3.2 Benzene — For use with coke sample, see IS : 1840-1961*.

7.4 Procedure

7.4.1 Use air-dried material passing through 212-micron IS sieve.

7.4.2 Depending on the furnace available, heat at $900 \pm 10^\circ\text{C}$ for 7 minutes either one crucible with lid or the requisite number of crucibles with lids to fill the stand. Remove from the furnace, cool the crucible(s) first on metal plate for 5 minutes and then in a desiccator for 10 minutes. Weigh each empty crucible with lid and weigh accurately into each crucible 1.00 to 1.01 g of sample. Replace the lid and tap the crucible on a clean hard surface until the sample forms a layer of even thickness.

7.4.2.1 Adjust the temperature of the zone in the muffle furnace, containing the stand and empty crucible(s) to $900 \pm 10^\circ\text{C}$ as indicated by the correctly located unsheathed thermocouple, or to the equivalent temperature as indicated by the sheathed thermocouple.

7.4.2.2 Remove the stand and empty crucible(s) and close the door of the muffle furnace to restore steady temperature conditions. If the sample is of coke or low volatile coal, remove the lid and add 2 drops of benzene and then replace the lid. Place the charged crucible(s) in another stand (see Note), transfer to the muffle furnace and leave for a period of exactly 7 minutes. Remove, cool and weigh the crucible(s) in the same manner as for the empty crucible(s). Note the nature of the residue left in the crucible(s).

NOTE — If multiple determinations are being made, any vacant places in the stand are to be filled with empty crucibles.

7.5 Calculation — Calculate the volatile matter of the sample on 'air-dried' basis as follows:

$$\text{Percent volatile matter, } V = \frac{100 (M_2 - M_3)}{M_2 - M_1} - M_0$$

where

M_0 = percentage of moisture in the sample on air dried basis;

M_1 = mass in g of empty crucible and lid;

M_2 = mass in g of crucible plus lid and sample before heating;
and

M_3 = mass in g of crucible plus lid and sample after heating.

*Specification for benzene, reagent grade.

7.6 Precision

7.6.1 The results of duplicate determination shall agree within the following limits:

Volatile Matter, percent	Precision	
	Repeatability	Reproducibility
For coals	1.0 percent of the arithmetic mean of the duplicate values	1.5 percent of the arithmetic mean of the duplicate determinations
For coke	0.2 units	0.4 units

8. DETERMINATION OF ASH

8.0 **Outline of the Method** — The sample is heated in air to 500°C in 30 minutes, from 500 to 815°C for a further 30 to 60 minutes and maintained at this temperature until constant in mass.

8.1 Apparatus

8.1.1 *Muffle Furnace* — Capable of giving a substantially uniform zone of 500°C in 30 minutes from cold, of being raised to $815 \pm 10^\circ\text{C}$ in a further 30 to 60 minutes and of maintaining this temperature up to the end of the run up period. The furnace should also be capable of being raised to a temperature of $850 \pm 10^\circ\text{C}$, if necessary. The ventilation shall be such as to give about five air changes per minute.

NOTE — The necessary rate of air change can be obtained by fitting a chimney about 20 cm high to the back of the muffle and leaving the front door of the muffle open by 1.25 cm. If the chimney cannot be fitted to the back of the muffle, the required ventilation can be obtained by fitting an insulating brick as a door, leaving a clearance of 12.5 cm³ as fair inlet, and carrying a silica tube passing to the back of the muffle to withdraw air, which is vented through a vertical light-alloy tube 2.5 cm in diameter and 50 cm in length, also fitted into the insulating brick. The ventilation rate may be checked by using a Pitet tube or provided that six 2-g samples of coal can be ashed completely in an hour.

8.1.2 *Dish* — of silica, porcelain or platinum, 10 to 15 mm deep, of such a size that, with the amount of sample used, the spread does not exceed 0.15 g/cm².

8.2 Procedure

8.2.1 Thoroughly mix for one minute the air-dried material, ground to pass through 212-micron IS sieve preferably with a laboratory mechanical mixer. Weigh a clean dry empty dish and lid. Into the dish weigh accurately about one to two grams of the material according to the size of the dish. Distribute the material so that the spread does not exceed 0.15 g per cm². Insert the uncovered dish into the muffle furnace at room

temperature, raise the temperature to 500°C in 30 minutes and to 815 ± 10°C in a further 30 to 60 minutes and maintain at this temperature for 60 minutes.

8.2.2 Cover the dish with its lid if the ash is light and fluffy, remove from the muffle furnace and allow to cool, first on a cold metal slab for ten minutes and finally in a desiccator placed at the side of the balance. Weigh after the dish has been in the desiccator for 15 minutes. Re-ignite at the same temperature until the change in mass of the ash is less than 0.001 g. Note the colour of ash. Brush out the ash and reweigh the empty dish. Obtain the mass of the ash by difference.

8.3 Calculation

$$\text{Ash, percent by mass} = 100 \times \frac{(M_3 - M_4)}{M_2 - M_1}$$

where

M_1 = mass in g of dish,

M_2 = mass in g of dish and sample,

M_3 = mass in g of dish and ash, and

M_4 = mass in g of dish after brushing out the ash and on reweighing.

8.4 Precision — The results of duplicate determinations shall agree within the following limits:

Ash, percent

Precision

	Precision	
	Repeatability	Reproducibility
Upto 10.0	0.2 units	0.4 units
Over 10.0	2.0 percent of the arithmetic mean of the duplicate values	3.0 percent of the arithmetic mean of the duplicate values

9. REPORTING OF RESULTS

9.0 General — The results of proximate analysis may be reported in the following manner making use of the symbols recommended.

9.1 On Air Dried Basis

9.1.1 The following symbols shall be used without any suffix:

M_0 = percentage of moisture in the sample,

A = percentage of ash in the sample,

V = percentage of volatile matter in the sample, and

F = percentage of fixed carbon in the sample.

9.1.2 It is to be noted that the volatile matter V is the total loss recorded during volatile matter determination minus the moisture given off on drying the coal sample.

9.1.3 Fixed carbon is arrived at after deducting from 100 the sum of moisture, ash and volatile matter contents expressed as percent, that is.

$$\text{Fixed carbon, } F = 100 - (M_o + A + V)$$

or

Fixed carbon = Residue after volatile matter test minus ash.

9.2 On 60 Percent Relative Humidity and 40°C Basis

9.2.1 The symbols indicated in 9.1.1 shall be used with suffix 1.

9.2.2 The results on 60 percent relative humidity basis are then obtained as follows:

$$A_1 = \frac{A \times (100 - M_{o_1})}{100 - M_o}$$

$$V_1 = \frac{V \times (100 - M_{o_1})}{100 - M_o}$$

$$F_1 = \frac{F \times (100 - M_{o_1})}{100 - M_o} \text{ or as a check } 100 - (M_{o_1} + A_1 + V_1)$$

9.3 On 'Dry' Basis

9.3.1 The symbols indicated in 9.1.1 shall be used with suffix d .

9.3.2 The results on 'dry basis' are then obtained as follows:

$$A_d = \frac{A \times 100}{100 - M_o}$$

$$V_d = \frac{V \times 100}{100 - M_o}$$

$$F_d = \frac{F \times 100}{100 - M_o} \text{ or as a check } 100 - (A_d + V_d)$$

9.4 On 'Dry Mineral Free' Basis (Formerly 'Dry Mineral-Matter-Free' Basis)

9.4.1 The symbols indicated in 9.1.1 shall be used with suffix dmf .

9.4.2 The results on 'dry mineral free' basis are then obtained as follows:

$$F_{dmf} = \frac{F \times 100}{100 - (\text{moisture} + \text{mineral matter})}$$

$$V_{dmf} = 100 - F_{dmf}$$

9.4.3 In conversion to V_{dmf} it is preferable first to evaluate F_{dmf} from F and then obtain V_{dmf} as $100 - F_{dmf}$. This is advisable as V contains water of hydration of the mineral matter and also carbon dioxide from carbonates etc, which calls for appropriate corrections in case of conversion to V_{dmf} direct from experimental value for volatiles.

9.5 Evaluation of Mineral Matter (MM)

9.5.1 For most of the Indian coals, containing low proportions of sulphur and ' carbonate ' minerals the general formula for the computation of mineral matter shall be as follows:

$$\text{mineral matter (MM)} = 1.1 \times A$$

where

A = represents percent ash content of a coal.

where the ' carbonate ' carbon dioxide content is more than 0.5 percent, the formula for mineral matter shall be as follows:

$$\text{mineral matter} = 1.1 A + 0.7 \text{ CO}_2$$

where

CO_2 = the percent ' carbonate ' carbon dioxide content in coal.

Usually the major carbonate mineral in coal is siderite associated often with some amounts of calcite, magnesite, ankerite, etc, for which the generalized factor 0.7 CO_2 holds good.

9.5.2 Where carbonate CO_2 exceeds one percent, the proportions of different forms of carbonates may be determined from analysis and the comprehensive factor worked out using 0.64 CO_2 for siderite, 0.87 CO_2 for calcite and 0.91 CO_2 for magnesite.

9.5.3 In case where sulphur is more than 1 percent, the formula for mineral matter shall be as follows:

$$\text{mineral matter} = 1.08 A + 0.55 S_{pyr}$$

where

S_{pyr} = percent sulphur occurring as pyrites.

While using this formula, F_{dmf} if calculated as follows:

$$F_{dmf} = \frac{F - 0.15 S_{pyr}}{100 - (\text{moisture} + \text{mineral matter})}$$

9.5.4 In case where both sulphur and ' carbonate ' carbon dioxide contents are high, the following formula shall be used:

$$\text{Mineral matter} = 1.08 A + 0.55 S_{pyr} + 0.70 \text{ CO}_2$$

Herein also F_{dmf} shall be calculated as in 9.5.3.

IS : 1350 (Part 1) - 1984

9.5.5 In case of coals where ash content is 5 percent or below the mineral matter may be taken to be numerically equal to the ash content. Although such a guideline may be largely correct, it is, however, safer to determine the mineral matter content by experimental method for a more precise evaluation.

9.6 Form for Reporting of Results

Ref. No
 Seam
 Location.....
 Sample No..... Character of sample.....
 Received on..... Size
 Prepared on..... Gross mass
 Nature of sample..... Sampling done by

Proximate Analysis	Air Dried Basis	60 Percent RH and 40°C Basis	Dry Basis	dmf Basis	Remarks		
					Clour of Ash	Nature of Residue After Volatile Matter Test	Mineral Matter Formula Used

Moisture,
percent

Ash,
percent

Volatile
matter,
percent

Fixed
carbon,
percent

Total	100·0	100·0	100·0	100·0			
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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 23230131, 23233375, 23239402

Fax: 91+011 23239399, 23239382

E - Mail : bis@vsnl.com

website : <http://www.bis.org.in>

Central Laboratory:

Plot No. 20/9, Site IV, Sahibabad Industrial Area, SAHIBABAD 201010

Telephone

27700 32

Regional Offices:

Central: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 2323 76 17

*Eastern: 1/14 CIT Scheme VII M, V.I.P. Road, Kankurgachi, KOLKATA 700054 2337 86 62

Northern: SCO 335-336, Sector 34-A, CHANDIGARH 160022 260 38 43

Southern: C.I.T. Campus, IV Cross Road, CHENNAI 600113 2254 19 84

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Branch Offices:

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SCO 21, Sector 12, Faridabad 121007 229 2175

Savitri Complex, 116 G.T. Road, GHAZIABAD 201001 286 1498

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Mahabir Bhavan, 1st Floor, Ropar Road, NALAGARH 174101 22 14 51

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"Sahajanand House" 3rd Floor, Bhaktinagar Circle, 80 Feet Road,
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