IS 1350-2 (1975): Methods of Test for Coal and Coke, Part II: Determination of Calorific Value [PCD 7: Solid Mineral Fuels]

BLANK PAGE

IS : 1350 (Part II) - 1970
(Reaffirmed 2000)

Indian Standard REAFFIRMED

METHODS OF TEST FOR COAL AND COKE

PART II DETERMINATION OF CALORIFIC VALUE

(First Revision)

Ninth Reprint MARCH 2001
(Incorporating Amendment No.1)

UDC 662.74 : 536.6.081

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

Gr 7

April 1971

Indian Standard

METHODS OF TEST FOR COAL AND COKE

PART II DETERMINATION OF CALORIFIC VALUE

(First Revision)

Solid Mineral Fuels Sectional Committee, CDC 14

Chairman	*Representing*
Dr A. Lahiri	Central Fuel Research Institute (CSIR), Jealgora
Vice-Chairman	
Shri A. K. Moitra	Central Fuel Research Institute (CSIR), Jealgora
Members	
Shri A. Banerjee	Coal Controller, Calcutta
Shri K. L. Banerjee	National Test House, Calcutta
Shri R. N. Roy (*Alternate*)	
Shri S. K. Bose	National Coal Development Corporation Ltd, Ranchi
Chief Mining Adviser, Railway Board, Dhanbad	Ministry of Railways
Joint Director, Mechanical Engineering (Fuel), Railway Board, New Delhi (*Alternate*)	
Dr A. N. Chowdhury	Geological Survey of India, Calcutta
Shri A. R. Das Gupta	Bird & Co (Private) Ltd, Calcutta
Shri L. D. Hughes	Joint Working Committee of Indian Mining Association, Indian Mining Federation, Indian Colliery Owners Association, and Madhya Pradesh & Vidarbha Mining Association, Calcutta
Shri R. R. Khanna (*Alternate*)	
Shri J. K. Jain	Coal Consumers' Association of India, Calcutta
Shri S. K. Mandal	Hindustan Steel Ltd, Ranchi
Shri S. N. Wazir (*Alternate*)	
Shri A. K. Mitra	Coal Board, Calcutta
Shri U. N. Jha (*Alternate*)	
Shri K. G. Rangrez	Regional Research Laboratory (CSIR), Hyderabad
Shri K. Seshagiri Rao (*Alternate*)	

(*Continued on page* 2)

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

(*Continued from page* 1)

Members	*Representing*
Dr C. V. S. Ratnam	Neyveli Lignite Corporation Ltd, Neyveli
Shri N. Selvarajan (*Alternate*)	
Shri B. C. Roy Ghatak	The Indian Iron and Steel Co Ltd, Calcutta
Shri S. B. Sarkar	Institute of Fuels (Indian Section), Calcutta
Shri F. S. Watcha	The Tata Iron and Steel Co Ltd, Jamshedpur
Shri L. Wilsing	Coke Oven Managers' Association (Indian Section), Burnpur
Shri D. Das Gupta, Director (Chem)	Director General, ISI (*Ex-officio Member*)

Secretary

Shri S. Subrahmanyan
Deputy Director (Chem), ISI

Methods of Test for Coal and Coke Subcommittee, CDC 14 : 2

Convener	
Dr A. N. Chowdhury	Geological Survey of India, Calcutta
Members	
Shri N. G. Banerjee	Central Fuel Research Institute (CSIR), Jealgora
Shri N. N. Chatterjee (*Alternate*)	
Shri D. K. Basu	Hindustan Steel Ltd, Ranchi
Dr U. N. Bhrany	The Indian Iron & Steel Co Ltd, Calcutta
Shri S. K. Dikshit (*Alternate*)	
Chemist and Metallurgist, South Central Railway, Secunderabad	Ministry of Railways
Assistant Director (Met) RDSO, Chittaranjan (*Alternate*)	
Shri A. K. Das Gupta	Institute of Fuels (Indian Section), Calcutta
Shri A. R. Das Gupta	Bird & Co (Private) Ltd, Calcutta
Dr G. V. L. N. Murty	The Tata Iron and Steel Co Ltd, Jamshedpur
Dr M. P. Gupta (*Alternate*)	
Shri R. N. Roy	National Test House, Calcutta
Shri S. B. Sarkar	Coal Board, Calcutta
Shri N. Selvarajan	Neyveli Lignite Corporation Ltd, Neyveli
Shri K. Seshagiri Rao	Regional Research Laboratory (CSIR), Hyderbad

Panel for Reviewing Indian Standards on Methods of Test for Coal and Coke, CDC 14 : 2 : 2

Convener	
Shri N. G. Banerjee	Central Fuel Research Institute (CSIR), Jealgora
Members	
Shri S. P. Dhar	Durgapur Projects Ltd, Durgapur
Dr S. R. Ghose	Hindustan Steel Ltd, Ranchi
Shri A. R. Panikar (*Alternate*)	

(*Continued on page* 28)

Indian Standard

METHODS OF TEST FOR COAL AND COKE

PART II DETERMINATION OF CALORIFIC VALUE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part II) (First Revision) was adopted by the Indian Standards Institution on 16 March 1970, after the draft finalized by the Solid Mineral Fuels Sectional Committee had been approved by the Chemical Division Council.

0.2 The ' Indian Standard methods of test for coal and coke — Proximate analysis, total sulphur and calorific value ' was first published in 1959. Since then, the experience gained with those methods in the country and a study of the various recommendations made by the Technical Committee, TC 27, ' Solid Mineral Fuels ' of the International Organization for Standardization (ISO) necessitated the present revision. While taking up this revision the concerned Sectional Committee decided that the standard should be issued in three parts as follows:

Part I Proximate analysis,
Part II Determination of calorific value, and
Part III Determination of sulphur.

0.3 The methods covered in this part are based largely on the documents under consideration by TC 27 ISO. In the case of the isothermal bomb method, the conventional method of stirring by electric motor is prescribed for stirring the water in the jacket. However, for a more convenient and accurate determination, reference may be made to the following published article:

WHITAKER (JW), GHOSH (AK) and CHAKRAVORTY (RN). Air stirring in bomb calorimetry. *Fuel* XXXIV; No. 3 (1955).

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*.

1. SCOPE

1.1 This standard (Part II) prescribes the methods of test for coal and coke relating to the determination of calorific value.

2. TERMINOLOGY

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

2.1 Air-Dried Coal — The sample of coal, which for the purposes of analysis, is exposed to the atmosphere of the laboratory to bring it in equilibrium with the humidity conditions prevailing there, so that the sample does not lose or gain weight during weighing.

2.2 Units of Heat

a) *Absolute Joule* — The basic unit, equivalent to 10^7 ergs.

b) *Mean Gram Calorie* — One hundredth part of the amount of heat required to raise the temperature of one gram of water from 0° to 100°C. It is equal to 4·189 7 joules.

c) *Gram Calorie (cal)* — Amount of heat required to raise the temperature of one gram of water from 14·5 to 15·5°C, used as an international standard for calorimetry. It is equal to 4·185 5 joules and it differs from the mean calorie by only one part in 1 000, which is not significant enough to require correction for practical purposes.

d) *International Steam Table Calorie* — Equal to 4·186 8 joules (exactly) and differs from gram calorie by approximately 0·3 part in 1 000.

2.3 Calorific Value

a) *Gross Calorific Value* — Number of heat units liberated when a unit mass of the fuel is burnt at constant volume in oxygen saturated with water vapour, the original material and final products being at approximately 25°C. The residual products are taken as carbon dioxide, sulphur dioxide, nitrogen and water; the residual

---

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

water other than that originally present as vapour, being in the liquid state.

NOTE 1 — The gross calorific value at constant volume is the one usually used in coal technology. It is assumed that all the heat produced is available, including the heat of condensation of any steam, resulting from the combustion of hydrogen of the fuel, to water at room temperature.

NOTE 2 — To convert gross calorific value to net calorific value, employ the formula:

$$Nc = Gc - 53\ H$$

where

Nc = net calorific value in Kcal/kg,
Gc = gross calorific value in Kcal/kg, and
H = percentage of hydrogen present in the coal sample, including hydrogen of moisture and of water of constitution.

b) *Net Calorific Value* — Number of heat units liberated when a unit mass of the fuel is burnt at constant volume in oxygen saturated with water vapour, the original and final materials being at approximately 25°C. The residual products are taken as carbon dioxide, sulphur dioxide, nitrogen and water vapour.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS: 1070-1960*) shall be employed.

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 I)-1964† for coal and IS: 436(Part II)-1965‡ for coke.

4.2 Preparation of Samples for Test

4.2.0 *General* — It is expected that methods of sampling prescribed in IS: 436(Part I)-1964† and IS: 436(Part II)-1965‡ shall 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.

---

*Specification for water, distilled quality (*revised*).

†Methods for sampling of coal and coke: Part I Sampling of coal (*revised*).

‡Methods for sampling of coal and coke: Part II Sampling of coke (*revised*).

4.2.1 The samples prepared in accordance with IS: 436(Part I)-1964* and IS: 436(Part II)-1965† shall be received in sealed containers and shall consist of the following:

a) Analysis sample of about 300 g of air-dried coal or coke, ground to pass 212-micron IS Sieve (*see* IS: 460-1962‡); and

b) Special moisture sample of 1 kg of coal or 2·5 kg of coke, crushed to pass 12·5-mm square-mesh screen (*see* IS: 460-1962‡), to be sent in duplicate.

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 required to be recorded on the label together with the 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 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 remixed before analytical work is started. The use of mechanical devices is recommended.

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 remixed. This final grinding will normally be found to ensure reasonable agreement between repeat determinations. The normal method of mixing 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.

5. CALORIFIC VALUE OF COAL AND COKE

5.0 General — Two methods have been described to determine the calorific value of coal or coke. They are: (a) making use of calorimetric bomb immersed in a static or isothermal water jacket (*see* **5.4**), and (b) making use of calorimetric bomb immersed in an adiabatic jacket (*see* **5.5**).

---

*Methods for sampling of coal and coke: Part I Sampling of coal (*revised*).

†Methods for sampling of coal and coke: Part II Sampling of coke (*revised*).

‡Specification for test sieves (*revised*).

5.0.1 The calorific value as determined in these methods is the gross calorific value of coal and coke at constant volume expressed in calories per gram.

5.0.2 *Temperature Dependence of Calorific Value* — The calorific value of coal and coke decreases with increase of temperature. The magnitude of dependence varies between 0·1 and 0·3 cal/g°C, the lower value being for anthracites; correction for variation in temperature of determination is, therefore, usually negligible.

5.1 Principle — The coal or coke is burned in a bomb calorimeter of known heat capacity. The principal observation is that of a temperature rise which, when corrected for the errors of the thermometer and multiplied by the effective heat capacity at the mean temperature of the chief period, gives the heat release. Further, allowance is necessary for (a) the cooling loss; (b) the heat gain due to the heat released by the ignition system; and (c) the heats of formation of sulphuric and nitric acids from sulphur dioxide and nitrogen.

5.1.1 This method is applicable to all bituminous coals and anthracites provided sufficient excess of oxygen is present to ensure complete combustion. With bombs near the upper limit of size, an initial oxygen pressure of 25 atmospheres is sufficient when using one gram of sample but for smaller capacity bombs it may be necessary to increase the initial oxygen pressure to 30 atmospheres or to reduce the weight of the sample to 0·8 g. A redetermination of the heat capacity (*see* **5.6**) for the use of the higher pressure is not necessary. The heat capacity varies slightly with the crucible in use; adjustments may be based on the calculated heat capacities (mass × specific heat) of the different crucibles used on the basis of the following specific heats:

Platinum	0·03 cal/g°C
Nickel-chromium alloy	0·11 cal/g°C
Silica	0·18 cal/g°C

5.2 Apparatus

5.2.1 *Combustion Bomb* — A high pressure bomb, preferably of the three-piece Berthelot type with separate cup, cap and union nut. The cup and cap are machined from rolled bar of an austenitic chromium-nickel-molybdenum steel resistant to intercrystalline corrosion. The union nut, which does not come into contact with acidic gases, should be made of free-cutting austenitic steel or of rolled aluminium bronze with a 0·5 percent proof stress of not less than 2 000 kg/cm^2. This ensures that the bomb will be capable of (a) burning completely one gram of coal using an initial oxygen pressure of 30 atmospheres and (b) withstanding, with an adequate safety factor but without excessive mass, the maximum pressure likely to be produced. The union nut shall not be made of the same type of austenitic steel as that used for the cup and cap; this is important to prevent damage due to seizing of the threads; aluminium bronze is given as an alternative to low-molybdenum (free-cutting) austenitic steel and, if used, may be plated to improve the finish.

The bomb shall also be:

a) of 250 to 350 ml capacity (existing bombs of up to 500 ml capacity, if resistant to intergranular corrosion, may be used but their replacement is advised);

b) of not more than 3·25 kg weight; and

c) capable of withstanding under test, for ten minutes and without permanent deformation, an internal pressure of 210 kg/cm^2.

5.2.2 *Calorimeter Vessel* — The calorimeter vessel for containing the bomb shall be of plated copper or brass, polished on the outside and fitted with a cover. It shall hold two litres of water with the bomb covered with water, to a depth of at least two centimetres above the lid of the bomb.

5.2.3 *Water-Jacket* — A water-jacket should be provided for containing the vessel and this may be of the static, isothermal, or adiabatic type. The vessel should be insulated from the jacket by being supported on the minimum amount of a non-conducting material, preferably on a three-point support. The air space between the vessel and the jacket should be approximately 1 cm to eliminate convection currents. The water-jacket should be provided with a cover.

5.2.3.1 The static water-jacket should contain at least 12·5 litres of water to eliminate the effect of external temperature change on the temperature in the calorimeter.

5.2.3.2 The isothermal jacket should be provided with means of keeping its temperature constant throughout the determination to within ± 0·1°C.

5.2.3.3 The adiabatic jacket is fitted with either electrode or immersion heaters capable of supplying energy at a rate sufficient to heat the water contained in the jacket so that its temperature is maintained within 0·1°C of the calorimeter vessel temperature after the bomb has been fired. The sensitivity of the temperature control system is such that the temperature drift of the calorimeter vessel, when in balance at 20°C, does not exceed 0·000 5°C.

5.2.4 *Stirring Arrangement* (*see* **0.3**) — The water in the calorimeter shall be efficiently stirred by a constant speed motor and the rate of stirring, which shall be constant, shall be such that the temperature readings plotted against time shall approximate to a smooth curve which in the preliminary and the after periods, shall be as straight line. To ensure this, the stirrer shall operate at a speed slightly greater than the minimum necessary. The stirrer shaft shall contain, at or above the level of the cover of the calorimeter vessel, a non-conducting section to prevent transmission of heat to or from the calorimeter.

5.2.5 *Thermometer* — The thermometer shall be of 6-degree range and duly certified by the National Physical Laboratory or any other Institution recognized by the Indian Standards Institution. It shall be graduated to 0·01°C and used with certified corrections which shall be given to within 0 002°C at intervals of not more than 0·5°C. The thermometer shall be tapped before each reading in order to prevent the mercury thread from sticking. If a thermometer of the Beckmann type is used, and the certificate of testing does not show the changing value of the degree, due to the variable amount of mercury in the bulb, determinations of the water equivalent shall be done to various temperatures, say, when the zero of the thermometer corresponds to 10°C, 15°C, 20°C (about), etc, and the appropriate value, found by interpolation, shall be used when calorimetric determinations are made. A solid stem thermometer is preferable.

5.2.6 *Thermometer Viewer* — A viewer with a magnification ×5 shall be used to permit the reading of temperatures by estimation to 0·001°C. A cathetometer may be used to avoid the possibility of parallax error; with a single lens in a tube, parallax errors shall be avoided by ensuring that the operator's eye is at the correct level and by illuminating the scale through a circular hole in the tube and not a cut-away aperture at the end of the tube.

5.2.7 *Crucible* — While a variety of shapes of crucibles are suitable, it may be desirable, in order to avoid ejection of ash during the determination, to use a crucible with the walls vertical or tapering inwards and upwards. Alternatively, the coal briquette may be covered by a few silica chips approximately 10×5×1 mm to prevent ejection of the combustible material or ash. The crucibles may be of silica, nickel-chromium or platinum having flat base with a diameter of about 25 mm and a height of not more than 20 mm. Silica crucibles should be about 1·5 mm thick and metal crucibles about 0·5 mm thick. To reduce any error from incomplete combustion when testing high ash coals, a shallow crucible of nickel-chromium foil about 0·25 mm thick is recommended.

5.2.7.1 The production of carbon smears when determining effective heat capacity with benzoic acid may be avoided by the use of a smaller nickel-chromium crucible, about 0·25 mm thick, 15 mm in diameter and 7 mm deep.

5.2.8 *Ignition Circuit* — An electrical supply of 6 to 12 volts, for igniting the sample with the controls mounted on a wall or bench and not on the calorimeter shall be used. An ammeter may be included in the circuit to indicate breakage of the firing wire. The circuit may conveniently consist of a six-volt battery connected through an open key. If the supply is provided by means of a transformer, a 5-ampere fuse shall be fitted in the secondary circuit.

CAUTION — The firing switch should not be mounted on the calorimeter.

5.2.9 *Timer* — A timer, reading in minutes and seconds, shall be fixed in a convenient position. A stop-watch may be used.

5.2.10 *Pressure Regulator and Pressure Gauge* — A regulator shall be provided in control filling of the bomb and a pressure gauge [0 to 5×10^6 N/m^2 (50 atm)] fitted to indicate the pressure in the bomb. To avoid overfilling of the bomb, a relief valve or bursting disc operating at $3{\cdot}5 \times 10^6$ N/m^2 (35 atm) may be installed in the filling line.

CAUTION — Equipment for high-pressure oxygen must be kept free from oil and grease.

5.3 Reagents

5.3.1 *Benzoic Acid* - pure, preferably of thermochemical quality.

5.3.2 *Oxygen* — free from combustible matter and hydrogen and available at a pressure of not less than 25 atmospheres. Oxygen prepared by the electrolytic process may contain a small percentage of hydrogen, which invalidates its use.

5.3.3 *Firing Wire* — platinum wire of about 0·08 mm diameter, or nickel-chromium wire of about 0·10 mm diameter.

5.3.4 *Cotton Thread* — white sewing cotton thread.

5.3.5 *Standard Sodium Hydroxide Solution* — 0·1 N.

5.3.6 *Standard Hydrochloric Acid Solution* — 0·1 N.

5.3.7 *Methyl Orange Indicator* — 1 percent (*w/v*) in water.

5.3.8 *Phenolphthalein Indicator* — 0·1 percent (*w/v*) ethyl alcohol.

5.3.9 *Concentrated Hydrochloric Acid* — sp gr 1·16 (*see* IS: 265-1962*).

5.3.10 *Standard Barium Hydroxide Solution* — 0·1 N.

5.3.11 *Standard Sodium Carbonate Solution* — 0·1 N.

---

*Specification for hydrochloric acid (*revised*).

5.4 Isothermal Calorimeter Method

5.4.1 Procedure

5.4.1.0 Coal used for the determination of calorific value is the analysis sample ground to pass through 212-micron IS Sieve. The sample is exposed in a thin layer for the minimum time necessary for the moisture content to reach equilibrium with the laboratory atmosphere.

5.4.1.1 Weigh the crucible to the nearest 0·1 mg and introduce into it sufficient quantity of the sample to cause a temperature rise of 2° to 3°C. Weigh the crucible and contents to determine the weight of sample taken. Also determine the moisture content of the sample at the same time.

NOTE — Normally 1 g of coal will be required. For high ash coals, the use of 0·75 g of the sample and the shallow crucible of nickel-chromium foil should reduce errors of incomplete combustion. If considered desirable, the sample may be previously compressed into a cylindrical pellet before weighing. Bituminous coals can often be burnt with more certainty when compressed, whereas anthracite cannot be compressed sufficiently. Anthracite also cannot be briquetted satisfactorily and is, frequently, incompletely burned if weighed into the crucible in the ordinary way. It is best weighed on a thin disc of mica or a layer of fine ignited sand or silica, placed on the bottom of the crucible and a known weight, say 0·01 to 0·02 g of benzoic acid. With a silica crucible the use of mica is unnecessary. Coals containing a high percentage of inorganic matter give a fused residue which may enclose a considerable proportion of unburnt carbon. In such cases, combustion in a crucible, the bottom of which is covered with a thin layer of coarsely powdered quartz, is recommended. The quartz breaks up the fused ash into minute globules, thus minimizing the proportion of unburnt carbon; it also facilitates the removal of the residue if it should appear desirable to determine the quantity of unburnt matter. (*see* **5.4.2.5** and **5.5.2.4**).

5.4.1.2 Connect a piece of firing wire tautly across the terminals of the bomb. Tie a known weight of cotton to the firing wire and arrange the ends of the cotton so that they touch the sample.

NOTE — For convenience, a measured length of cotton of known weight per unit length may be used; the length used in each determination of calorific value should be the same as used in the determination of effective heat capacity of the system.

5.4.1.3 Put 1 ml of distilled water in the bomb. Assemble the bomb and charge it slowly with oxygen to a pressure of $3{\cdot}0 \times 10^6$ N/m^2 (30 atm) without displacing the original air. Put sufficient water in the calorimeter vessel to cover the flat upper surface of the bomb cap. This quantity of water should be the same, within 1 g, as that used in determining the mean effective heat capacity. The temperature of the water shall be about 2·5°C lower than that of the water jacket.

NOTE — Use of one millilitre is recommended. Any other quantity up to 5 millilitres may be used provided the same quantity is used in all determinations of both heat capacity and calorific value.

5.4.1.4 Transfer the calorimeter vessel to the water-jacket; lower the bomb into the calorimeter vessel and check that the bomb is gas-tight. If gas escapes from the bomb, discard the test.

5.4.1.5 Assemble, start up the apparatus and keep the stirrer and the circulation arrangements in continuous operation throughout the determination. Use a constant rate of stirring. After an interval of not less than ten minutes, read the temperature to 0·001°C and continue the readings for five minutes, that is, the preliminary period, at equal intervals of not more than one minute, tapping the thermometer lightly during 10 seconds prior to each reading. If, over a period of five minutes, the average deviation of the individual values of the rate of change of temperature is less than 0·000 72°C per minute, close the battery circuit momentarily to fire the charge and continue the observations of the temperature at intervals of similar duration to those of the preliminary period. If the rate of change of temperature is not constant within this limit, extend the preliminary period until it is constant.

5.4.1.6 In the chief period, which extends from the instant of firing until the time after which the rate of change of temperature again becomes constant, take the earlier readings to the nearest 0·01°C since it will not be possible to take the earlier readings to 0·001°C. Resume the readings to this precision as soon as possible. Determine the rate of change in the after period (which follows the chief period) by taking readings at 1 minute intervals for at least five, preferably ten minutes.

NOTE — It is desirable to keep the jacket temperature and the room temperature as close to the calorimeter temperature as possible. Hence the jacket and room temperatures should be recorded.

5.4.1.7 Remove the bomb from the calorimeter vessel, release the pressure and dismantle the bomb. Examine the bomb interior and discard the test if unburnt sample or sooty deposits are found.

5.4.1.8 Wash the contents of the bomb into a beaker with distilled water. Wash the underside of the bomb cap and the outside of the crucible with distilled water; add the washings to the beaker. Dilute to approximately 100 ml and boil to expel carbon dioxide. While still hot, titrate with standard barium hydroxide solution using phenolphthalein solution as indicator. Add 20 ml of sodium carbonate solution, warm, filter and wash the precipitate with distilled water. When cold, titrate the filtrate with the hydrochloric acid solution, using the methyl orange solution as indicator. ignoring the phenolphthalein colour change.

NOTE — These titrations may be omitted if the sulphur content of the coal and the nitric acid correction are known.

5.4.2 *Corrections* — The following corrections are made to the experimental observations.

5.4.2.1 *Thermometer corrections* — If a mercury-in-glass thermometer is used the corrections prescribed in the certificate issued with the thermometer are applied to the observed firing temperature t_0 and the final temperature t_n.

5.4.2.2 *Cooling correction* — The cooling correction includes the effects of the heat interchange between the vessel and the jacket due to conduction, convection and radiation, and of the heats of stirring and evaporation. This is minimized by having (a) the temperature of the water in the vessel below that of the water in the jacket at the time of firing and within ±1·0°C, preferably ±0·5°C, of it at the end of the chief period; (b) a low heat of stirring; and (c) a low loss by evaporation. The correction shall be determined as follows by the use of the Regnault-Pfaundler formula or the Whitaker formula which gives, for a representative series of coals, a mean correction differing by not more than one part in 1 000 from that given by the Regnault-Pfaundler formula.

a) *The Regnault-Pfaundler (R-P) correction*

$$\begin{aligned}\text{Correction} &= nv' + \frac{v''-v'}{t''-t'}\{\Sigma_1^{n-1}(t) + \tfrac{1}{2}(t_0 + t_n) - nt\} \\ &= nv' + kS\end{aligned}$$

where

n = number of minutes in the chief period (5 to 10 minutes);

v' = rate of fall of temperature per minute in the preliminary period. (If the temperature is rising during the preliminary period, then v' is negative);

v'' = rate of fall of temperature per minute in the after period;

t'' = average temperature during after period;

t' = average temperature during preliminary period;

t_0 = firing temperature;

$\Sigma_1^{n-1}(t)$ = sum of the temperatures (t_1, t_2, t_3, ..., $t_n - 1$) recorded during the chief period;

t_1, t_2, t_3, ..., t_n = successive temperatures recorded during the chief period. The final temperature, t_n, is the first temperature after which the rate of change is constant;

$\frac{1}{2}(t_0 + t_n)$ = mean of firing temperature t_0 and temperature t_n at the end of the chief period after which the rate of fall becomes constant again;

$k = \frac{v''-v'}{t''-t'}$ is the 'cooling canstant' of the calorimeter system which shall be determined for each set of conditions; and

S = the expression within the brackets.

From the nature of the formula it is seen that the temperature difference (t_n-t_0) to which this correction is added to obtain the corrected temperature rise is not necessarily the maximum observed rise. In fact, within reasonable limits, the corrected temperature rise should be the same irrespective of how far t_n is taken along the cooling curve, provided that the period of uniform fall of temperature has been reached.

b) *Whitaker correction* — In place of the Ragnault-Pfaundler cooling correction, which is cumbersome, the Whitaker correction may be adopted, provided that it has also been employed to find the water equivalent or the heat capacity of the apparatus. The heat capacity will be found by the Whitaker correction to be about 0·25 to 0·3 percent lower than the value given when *R-P* correction is used — a difference of 7 to 9 in 3 000.

To employ the Whitaker correction ten temperature readings are taken in the after period in order to obtain a more reliable value of the cooling rate per minute. If this rate be denoted by dt and the time from the instant of firing to any chosen temperature reading in the steady cooling period be x minutes, then the cooling correction is simply $(x \times dt)$ °C. The 'observed rise' is the difference between the temperature at the instant of firing and the temperature taken x minutes later (as above) in the cooling range. It is not necessary, nor is it desirable, to take the actual maximum temperature reading. Generally, a temperature taken 2 or 3 minutes later in the steady range of cooling forms a more reliable and definite 'observed maximum'. The temperature value at x, of course, depends upon the magnitude of x, but the same corrected temperature rise is obtained provided that dt is constant.

NOTE — Of the two corrections, the Whitaker formula is preferable. It is simpler; it is equally accurate and there is far less chance of arithmetical error in the calculations.

5.4.2.3 *Heat of ignition* — The heat release from the cotton and firing wire is subtracted from the total heat release. The heat release from the cotton is calculated from the weight, after drying at 105°C, of a known length of cotton thread, and using the calorific value of cellulose (4180 cal/g). Determine the weight of a piece of wire equal in length to the distance between the poles of the bomb, and calculate the heat release by allowing 335 cal/g for nickel-chromium wire, or 100 cal/g for platinum wire.

5.4.2.4 *Heat of formation of acids* — The heat gain due to the formation of sulphuric acid and nitric acid is subtracted from the total heat released. These corrections amount to 3·60 cal/ml of 0·1 N sulphuric acid and 1·43 cal/ml of 0·1 N nitric acid present in the bomb washings and are calculated as follows:

Sulphuric acid correction $= 3{\cdot}60\ (a + b - 20)$ cal; and
Nitric acid correction $= 1{\cdot}43\ (20 - a)$ cal

where

a = vol in ml of 0·1 N hydrochloric acid used (*see* **5.4.1.8**), and
b = vol in ml of 0·1 N barium hydroxide used (*see* **5.4.1.8**).

NOTE — The sulphuric acid correction may also be computed using the sulphur content determined by the Eschka method using the relationship that 0·01 g of sulphur (that is, one percent sulphur content on one gram of fuel) amounts to 22·5 cal.

5.4.2.5 *Correction for unburned carbon* (*see* **5.4.1.1**) — If unburned carbon is suspected, its heat equivalent, on the basis of 1 mg of carbon equals 8 calories shall be added to the determined heat release. Unburned carbon is determined as the loss in weight on ignition of the residue from the crucible.

5.4.3 *Calculation* — The following example illustrates the method of calculation using the *R-P* and the Whitaker corrections respectively:

a) *Calculation using R-P correction*

Weight of crucible + thread = 5·298 4 g
Weight of crucible + thread + coal = 6·301 7 g
Weight of air-dried coal = 1·003 3 g
Mean calorimeter temperature = 26·5°C
Effective mean heat capacity at 26·5°C = 2 871 cal/°C
Correction for sulphur (0·71 percent) in coal = 16 cal
Temperature of calorimeter jacket = 26·7°C
Temperature of thermometer setting = 24·6°C
Room temperature = 26·6°C
Moisture in coal = 1·90 percent
Ash = 12·1 percent

Time	*Minutes*	*Temperature* °C	*Correction Using Regnault-Pfaundler Formula*
Preliminary period	0	1·881	$t' = 1{\cdot}883$
	1	1·882	$v' = -0{\cdot}001$
	2	1·883	$n = 8$
	3	1·884	$nv' = -0{\cdot}008$
	4	1·885	
	5	1·886(t_0)	
Chief period	6	3·250(t_1)	
	7	4·160(t_2)	
	8	4·315(t_3)	$S = \{\Sigma_1^{n-1}(t) + \frac{1}{2}(t_0 + t_n) - nt'\}$
	9	4·362(t_4)	$= 29{\cdot}212 + 3{\cdot}126 - 15{\cdot}064$
	10	4·376(t_5)	$= +\ 17{\cdot}274$
	11	4·377(t_6)	
	12	4·372(t_7)	
	13	4·366($t_8 = t_n$)	
After period	14	4·360	
	15	4·354	
	16	4·349	$t'' = 4{\cdot}330$
	17	4·343	$v'' = 0{\cdot}006$
	18	4·336	$k = \dfrac{v'' - v'}{t'' - t'}$
	19	4·330	
	20	4·324	$= \dfrac{0{\cdot}007}{2{\cdot}447}$
	21	4·318	
	22	4·312	$= 0{\cdot}002\,861$
	23	4·306	
	24	4·300	

Cooling correction $nv' + ks$
$= 0{\cdot}049\,4 - 0{\cdot}008 = 0{\cdot}041\,4$

Uncorrected temperature rise ($t_n - t_0$)	= 2·480°C
Add cooling correction	= 0·041 4°C
Add thermometer correction	= 0·014°C
Corrected temperature rise	= 2·535 4°C
Heat liberated	= 2 871 × 2·535 4 = 7 279 cal
Subtract nitric acid correction	= 8 cal
Subtract correction for heat of ignition	= 21 cal
Heat from 1·003 3 g coal	= 7 250 cal
Heat from 1·000 0 g coal	= 7 226 cal
Subtract sulphur correction	= 16 cal
Calorific value of air-dried coal	= 7 210 cal/g

b) ***Calculation using Whitaker correction***

Weight of crucible + thread = 5·298 4 g
Weight of crucible + thread + coal = 6·301 7 g
Weight of air-dried coal = 1·003 3 g
Mean calorimeter temperature = 26·5°C
Effective mean heat capacity at 26·5°C = 2 863 cal/°C
Correction for sulphur (0·71 percent) in coal = 16 cal
Temperature of calorimeter jacket = 26·7°C
Temperature of thermometer setting = 24·6°C
Room temperature = 26·6°C
Moisture in coal = 1·90 percent
Ash = 12·1 percent

Time	*Minutes*	*Temperature* °C	*Correction Using Whitaker Formula*
Preliminary period	0	1·881	$dt_1 = -0{\cdot}001$ $n = 8$
	1	1·882	
	2	1·883	
	3	1·884	
	4	1·885	
	5	1·886(t_0)	
Chief period	6	3·250(t)	
	7	4·160	
	8	4·315	
	9	4·362	
	10	4·376	
	11	4·377	
	12	4·372	
	13	4·366(t_n)	
After period	14	4·360	$dt_2 = 0{\cdot}006$ $n \times dt_2 = 0{\cdot}048$
	15	4·354	
	16	4·349	
	17	4·343	
	18	4·336	
	19	4·330	
	20	4·324	
	21	4·318	
	22	4·312	
	23	4·306	
	24	4·300	

Uncorrected temperature rise ($t_n - t_0$) = 2·480°C
Add cooling correction = 0·048°C
Add thermometer correction = 0·014°C
Corrected temperature rise = 2·542°C
Heat liberated = 2 863 × 2·542
= 7 278 cal
Subtract nitric acid correction = 8 cal
Subtract correction for heat of ignition = 21 cal
Heat from 1·003 3 g coal = 7 249 cal
Heat from 1·000 0 g coal = 7 225 cal
Subtract sulphur correction = 16 cal
Calorific value of air-dried coal = 7 209 cal/g

5.4.3.1 The calorific value recorded above, namely, 7 209 cal/g, is that of the air-dried coal. The calorific value may be required of the coal as received. If such coal has, say, m percent moisture, the calorific value becomes 7 209 $(100-m)/(100-1{\cdot}9)$, as there is 1·90 percent moisture in the air-dried coal.

If it is desired to know the calorific value of the dry coal, the figure becomes $7\,209 \times 100/(100-1{\cdot}9)$. The calorific value of the pure dry mineral-matter-free coal is given by $7\,209 \times 100/(100-1{\cdot}9 + 1{\cdot}1$ times ash)

$$= 7\,209 \times \frac{100}{100-(1{\cdot}9 + 1{\cdot}1 \times 12{\cdot}1)}$$

$$= 7\,209 \times \frac{100}{84{\cdot}79}$$

$$= 8\,502 \text{ cal/g}$$

5.4.3.2 *Rounding off* — Round off the mean of the values or the single value to the nearest 10 cal/g.

5.4.3.3 *Precision* — The results of duplicate determinations in one laboratory should agree to within 25 cal/g, and those between different laboratory to within 50 cal/g.

5.5 Adiabatic Calorimeter Method

5.5.1 *Procedure*

5.5.1.1 The coal used for the determination of calorific value is the analysis sample ground to pass through 212-microns IS Sieve. The sample is exposed in a thin layer for the minimum time necessary for the moisture content to reach equilibrium with the laboratory atmosphere.

5.5.1.2 Weigh the crucible to the nearest 0·1 mg and introduce into it sufficient quantity of the sample to cause a temperature rise of 2° to 3°C. Weigh the crucible and contents to determine the weight of sample taken. Also determine the moisture content of the sample at the same time.

NOTE — Normally 1 g of coal will be required. For high ash coals, the use of 0·75 g of the sample and the shallow crucible of nickel-chromium foil should reduce errors of incomplete combustion.

5.5.1.3 Connect a piece of firing wire tautly across the terminals of the bomb. Tie a known weight of cotton to the firing wire; arrange the ends of the cotton so that they touch the sample.

NOTE — For convenience, a measured length of cotton of known weight per unit length may be used; the length used in each determination of calorific value should be the same as used in the determination of effective heat capacity of the system.

5.5.1.4 Put 1 ml of distilled water in the bomb. Assemble the bomb and charge it slowly with oxygen to a pressure of $3{\cdot}0 \times 10^6$ N/m^2 (30 atm) without displacing the original air. Put sufficient water in the calorimeter vessel to cover the flat upper surface of the bomb cap. This quantity of water should be the same, within 1 g, as that used in determining the mean effective heat capacity. The temperature of the water should be such that the final temperature reached in the determination is within a degree of the standard reference temperature, 27°C.

NOTE — Use of one millilitre is recommended. Any other quantity up to 5 millilitres may be used provided the same quantity is used in all determinations of both heat capacity and calorific value.

5.5.1.5 Transfer the calorimeter vessel to the water-jacket, lower the bomb into the calorimeter vessel and check that the bomb is gas-tight. If gas escapes from the bomb, discard the test.

5.5.1.6 Assemble, start up the apparatus and keep the stirrer and the circulation arrangements in continuous operation throughout the determination. Use a constant rate of stirring such that the length of the predetermined interval does not exceed 10 minutes. Select the setting of the bridge circuit that will result in the minimum drift in the temperature of the calorimeter vessel at the final temperature. After 10 minutes tap the thermometer lightly for 10 seconds and read it to 0·001°C (the 'firing temperature', t_0). Fire the charge; hold the switch closed long enough to ignite the fuse.

5.5.1.7 After the predetermined interval, established when the mean effective heat capacity of the system is determined, tap the thermometer again for 10 seconds and read it to 0·001°C (the 'final temperature', t_n). The observer should take care to avoid parallax errors when using the magnifying viewer to read mercury-in-glass thermometer.

5.5.1.8 Remove the bomb from the calorimeter vessel, release the pressure and dismantle the bomb. Examine the bomb interior and discard the test if unburnt sample or sooty deposits are found.

5.5.1.9 Wash the contents of the bomb into a beaker with distilled water. Wash the underside of the bomb cap and the outside of the crucible with distilled water; add the washing to the beaker. Dilute to approximately 100 ml and boil to expel carbon dioxide. While still hot, titrate with standard barium hydroxide solution using the phenolphthalein solution as indicater. Add 20 ml of sodium carbonate solution, warm, filter and wash the precipitate with distilled water. When cold, titrate the filtrate with the hydrochloric acid solution using the methyl orange solution as indicator, ignoring the phenolphthalein colour change.

NOTE — These titrations may be omitted if the sulphur content of the coal and the nitric acid correction are known.

5.5.2 *Corrections* — The following corrections are made to the experimental observations.

5.5.2.1 *Thermometer corrections* — If a mercury-in-glass thermometer is used the corrections prescribed in the certificate issued with the thermometer are applied to the observed firing temperature, t_0 and the final temperature t_n.

5.5.2.2 *Heat of ignition* — The heat release from the cotton and firing wire is subtracted from the total heat release. The heat release from the cotton is calculated from the weight after drying at 105°C a known length of cotton thread, and using the calorific value of cellulose (4 180 cal/g). Determine the weight of a piece of wire equal in length to the distance between the poles of the bomb, and calculate the heat release by allowing 335 cal/g for nickel-chromium wire or 100 cal/g for platinum wire.

5.5.2.3 *Heat of formation of acids* — The heat gain due to the formation of sulphuric acid and nitric acid is subtracted from the total heat released. These corrections amount to 3·60 cal/ml of 0·1 N sulphuric acid and 1·43 cal/ml of 0·1 N nitric acid present in the bomb washings and are calculated as follows:

Sulphuric acid correction $= 3\cdot60\ (a + b - 20)$ cal: and
Nitric acid correction $= 1\cdot43\ (20 - a)$ cal

where

a = volume in ml of 0·1 N hydrochloric acid used (*see* **5.4.1.8**),
and b = volume in ml of 0·1 N barium hydroxide used (*see* **5.4.1.8**).

NOTE — The sulphuric acid correction may also be computed using the sulphur content determined by the Eschka method using the relationship that 0·01 g of sulphur (that is, one percent sulphur content on one gram of fuel) amounts to 22·5 cal.

5.5.2.4 *Correction for unburned carbon* (*see* **5.4.1.1**) — If unburned carbon is suspected, its heat equivalent on the basis of 1 mg of carbon equals to 8 calories shall be added to the determined heat release. Unburned carbon is determined as the loss in weight on ignition of the residue from the crucible.

5.5.3 *Calculation* — The following example illustrates the method of calculation:

Weight of crucible + thread = 5·298 4 g
Weight of crucible + thread + coal = 6·301 7 g
Weight of air-dried coal = 1·003 3 g
Effective heat capacity at 26·5°C = 2 871 cal/°C
Correction for sulphur (0·71 percent) in coal = 16 cal
Room temperature = 26·6°C
Moisture in coal = 1·90 percent
Ash = 12·1 percent
Uncorrected temperature rise $(t_n - t_0)$ = 2·521°C
Add thermometer correction = 0·014°C
Corrected temperature rise = 2·535°C
Heat liberated = 2 871 × 2·535 = 7 279 cal
Subtract nitric acid correction = 8 cal
Subtract correction for heat of ignition = 21 cal
Heat from 1·003 3 g coal = 7 250 cal
Heat from 1·000 0 g coal = 7 226 cal
Subtract sulphur correction = 16 cal
Calorific value of air-dried coal = 7 210 cal/g

5.5.3.1 *Rounding off* — Round off the mean of the values or the single value to the nearest 10 cal/g.

5.5.3.2 *Precision* — The results of duplicate determinations in one laboratory should agree to within 30 cal/g, and those between different laboratories to within 65 cal/g.

5.6 Mean Effective Heat Capacity

5.6.0 *General* - The effective heat capacity is the heat required to effect unit temperature rise in the system under the conditions of a calorimetric determination. It has a temperature dependence since the specific heats of the constituent parts of the system vary with temperature; and 25°C has been chosen as the reference temperature because of its use in thermo-chemical calculations, and because the specific heat of water in the range of 25° to 40°C is constant within ± 0·002 cal/g°C.

5.6.1 *Principle* — A known weight of benzoic acid of certified calorific value is burned in oxygen in the bomb calorimeter, which is to be used eventually in the determinations of calorific value of coal and coke. To the heat of combustion of benzoic acid are added the heats of combustion of the firing wire and cotton thread, and also the heat of formation of nitric acid. At least five complete determinations of the effective heat capacity should be carried out and their mean calculated and reported.

5.6.2 *Procedure*

5.6.2.1 *Preparation of briquette* — Dry the benzoic acid carefully by heating to not more than 5°C above its melting point (121°C), which will cause fusion. Compress it in the form of a briquette weighing approximately 1·2 g.

NOTE — It will be necessary to use 1·2 g of benzoic acid in order to maintain a heat release of about 7 500 cal. By keeping the heat release constant at approximately this same level, the necessity for correcting for the sources of constant heat gain (namely the heats of ignition from the firing wire and the cotton thread) may be eliminated.

Difficulty is sometimes experienced in burning 1·2 g of benzoic acid without leaving smears of residual carbon; if these persist even when the distance between the crucible and the bomb cap has been increased to the maximum possible, then the special crucible (*see* **5.2.7.1**) shall be used.

5.6.2.2 Proceed as described in **5.4.1** or **5.5.1** for the determination of calorific value, depending on the type of calorimeter used; but read the temperature at 1 minute intervals over a period of 10 minutes, commencing 5 minutes after the firing of the charge.

5.6.2.3 Dilute the bomb washings to approximately 50 ml with distilled water. Titrate the nitric acid obtained with standard sodium hydroxide solution using methyl orange solution as indicator.

5.6.2.4 Carry out at least five complete determinations of effective heat capacity.

5.6.3 *Corrections* — The following corrections are to be applied to the experimental observations, as applicable.

5.6.3.1 *Thermometer corrections* — If a mercury-in-glass thermometer is used the corrections prescribed in the certificate issued with the thermometer are applied to the observed firing temperature t_0 and the final temperature t_n.

5.6.3.2 *Cooling correction* — The cooling correction determined as prescribed in **5.4.2.2** should be added to the measured temperature rise, in the case of an isothermal or static calorimeter.

5.6.3.3 *Heat of ignition* — The heat gain on account of the burning of firing wire and cotton thread is to be added to the heat of combustion of benzoic acid.

5.6.3.4 *Nitric acid correction* — The heat gain due to the formation of nitric acid is added to the heat of combustion of benzoic acid. It amounts to 1·43 cal/ml of 0·1 N sodium hydroxide solution used in the titration of bomb washings (*see* **5.5.2.3**).

5.6.4 *Calculation* — The following example illustrates the method of calculation:

Weight of crucible + thread = 5·234 8 g
Weight of crucible + thread + Benzoic acid = 6·429 0 g
Weight of benzoic acid = 1·194 2 g
Volume of 0·1 N sodium hydroxide solution = 6·8 ml
Corrected temperature rise (including cooling correction in the case of the isothermal type) = 2·641°C
Final calorimeter temperature = 25·5°C
Calorific value of benzoic acid at temperature (*see* Note) = 6 321·6 cal/g
Heat liberated, 1·194 2 × 6 321·6 = 7 548·1 cal
Add nitric acid correction, 1·43 × 6·8 = 9·7 cal
Add correction for heat of ignition = 21 cal
Total heat release = 7 578·8 cal
Effective heat capacity, 7 578·8/2·641 = 2 870 cal/°C

NOTE — The calorific value of pure benzoic acid, weighed in air, for the combustion of 1·2 g in a bomb of 300 ml capacity containing 1 g of water and using an initial oxygen pressure of 25 atmospheres is recorded below for different values of the temperature to which the products of combustion are cooled:

Final Calorimeter Temperature °C	*Calorific Value* (cal/g)	*Final Calorimeter Temperature* °C	*Calorific Value* (cal/g)
40	6 316·0	25	6 320·1
35	6 317·3	20	6 321·5
30	6 318·7	15	6 322·9

For bombs of 250 ml capacity, add 0·3 cal/g.
For bombs of 350 ml capacity, subtract 0·2 cal/g.
If 1 g of benzoic acid is used, subtract 0·2 cal/g.

5.6.4.1 *Variation with temperature* — The correction to 25°C of an effective heat capacity determined at t°C, the mean temperature during the chief period being $\frac{t_0 + t_n}{2}$, where t_0 is the temperature on firing and t_n is the temperature at the end of the chief period, may be expressed as follows:

$$C_{25} = C_t - D$$

where

C_{25} = effective heat capacity at 25°C;

C_t = the value of effective heat capacity determined at t°C;

$D = AW_m - B(W_s + W_0/4)$ cal/°C;

W_m = weight in kg of water in the system;

W_s = weight in kg of the stainless steel parts of the system;
W_b = weight in kg of the bronze and brass parts of the system (calorimeter vessel, stirrer and possibly nut); and

A and B for various temperatures are given below:

(°C)	A	B	t(°C)	A	B
10	3·01	1·50	27·5	−0·17	−0·25
12·5	2·16	1·25	30	−0·26	−0·50
15	1·48	1·00	32·5	−0·32	−0·75
17·5	0·95	0·75	35	−0·35	−1·00
20	0·55	0·50	37·5	−0·38	−1·25
22·5	0·24	0·25	40	−0·40	−1·50
25	0·00	0·00			

In the determination of the calorific value of a fuel, the appropriate heat capacity is found by adding the correct value of D to the mean value of C_{25}. Correction shall only be made in whole numbers. The temperature ranges over which particular integral corrections shall apply may be determined by calculating the value of D for a series of temperatures (covering the likely working range and plotting the data; for example, D is taken as 2 when it lies between 1·5 and 2·5).

5.6.4.2 *Reporting the result of mean effective heat capacity* — The result shall be reported at 25°C as the mean of at least 5 (preferably 10) values with a range not exceeding 0·3 percent of the mean value; it will then be correct to within 3 cal/°C.

5.6.5 *Predetermined Interval* — The period between the firing of the charge and the reading of the final temperature is the predetermined interval. It is calculated from the temperature readings taken at intervals of 1 minute in each determination of effective heat capacity.

5.6.5.1 From the recorded observations of each determination note the shortest time in minutes from the firing of the charge to reaching the second of three consecutive readings which do not differ by more than 0·001°C. Calculate the mean of the five values to the nearest whole minute to obtain the length of the predetermined interval; this should not exceed 10 minutes.

5.6.5.2 Use the predetermined interval for all determinations of calorific value until a new value is established. It is re-established when commissioning a new calorimeter and checked after changing any component.

5.6.6 *Redetermination of the Mean Effective Heat Capacity* — When any part of the system is changed, the mean effective heat capacity is redetermined at intervals not longer than six months. Where a change to the system is not involved, the redetermined mean should be within 5 cal/°C of that previously determined. If the difference is greater than 5 cal/°C experimental procedures should be examined and carefully checked.

6. REPORTING OF RESULTS

6.0 The results of calorific value may be reported in the following manner making use of the symbols recommended.

6.1 Air-Dried Basis

6.1.1 The following symbols shall be used without any suffix:

M = percentage of moisture in the sample,
A = percentage of ash in the sample,
V = percentage of volatile matter in the sample,
F = percentage of fixed carbon in the sample,
Q = calorific value of the sample, and
S = percentage of total sulphur in the sample.

6.1.2 It is to be noted that volatile matter V is the total loss recorded during volatile matter determination minus the moisture given off on drying at 105° to 110°C.

Fixed carbon = $F = 100 \quad (M + A + V)$
Fixed carbon = coke residue after volatile matter test *minus* ash.

6.2 As-Received Basis

6.2.1 The symbols indicated in **6.1.1** shall be used with suffix 1.

6.2.2 The results on the as-received basis are then obtained as follows:

Moisture = M_1

$$A_1 = \frac{A \times (100 - M_1)}{100 - M}$$

$$V_1 = \frac{V \times (100 - M_1)}{100 - M}$$

$$F_1 = \frac{F \times (100 - M_1)}{100 - M} \quad \textit{or, as a check, } 100 - (M_1 + A_1 + V_1)$$

$$Q_1 = \frac{Q \times (100 - M_1)}{100 - M}$$

$$S_1 = \frac{S \times (100 - M_1)}{100 - M}$$

6.3 Air-Dry Basis (Sample Equilibrated at 40°C and 60 Percent Relative Humidity)

6.3.1 The symbols indicated in **6.1.1** shall be used with suffix 2.

6.3.2 The results on the air-dry basis are then obtained as follows:

Moisture $= M_2$

$$A_2 = \frac{A \times (100 - M_2)}{100 - M}$$

$$V_2 = \frac{V \times (100 - M_2)}{100 - M}$$

$$F_2 = \frac{F \times (100 - M_2)}{100 - M} \quad \textit{or, as a check,}\ 100 - (M_2 + A_2 + V_2)$$

$$Q_2 = \frac{Q \times (100 - M_2)}{100 - M}$$

$$S_2 = \frac{S \times (100 - M_2)}{100 - M}$$

6.4 Dry Basis

6.4.1 The symbols indicated in **6.1.1** shall be used with suffix *d*.

6.4.2 The results on the dry basis are then obtained as follows:

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

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

$$F_d = \frac{F \times 100}{100 - M} \quad \textit{or, as a check,}\ 100 - (A_d + V_d)$$

$$Q_d = \frac{Q \times 100}{100 - M}$$

$$S_d = \frac{S \times 100}{100 - M}$$

6.5 Dry Mineral-Free Basis

6.5.1 The symbols indicated in **6.1.1** shall be used with suffix *dmf*. ' Mineral-matter-free ' is conveniently shortened to ' mineral-free '

6.5.2 The results of the dry mineral-free basis are then obtained as follows:

$$F_{dmf} = \frac{F \times 100}{100 - (M + 1 \cdot 1\ A)}$$

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

6.5.3 The following points may be noted while making calculations on the ***dmf*** basis:

a) The above calculation assumes that mineral matter is equal to 1·1 ash, which will hold good for the majority of Indian coals. When the coal, however, contains more than one percent of sulphur and 0·5 percent of carbon dioxide, the calculation of mineral matter is preferably made in accordance with the formula given in **2·6** of IS: 770-1964*.

b) 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 (roughly to the extent of 1/10 of the ash) and also carbon dioxide of any carbonates.

c) In obtaining S_{dmf}, allowance shall be made for sulphur percent as FeS_2 and sulphates (that is for sulphur present in the mineral matter). It is only the organic sulphur which can be expressed on a *dmf* basis.

d) In obtaining C_{dmf} (that is total carbon), it is necessary again to ensure that in the total carbon figure given from ultimate analysis the carbon present as carbonate has been deducted.

e) In evaluating H_{dmf} (that is, hydrogen) similarly the allowance of 1/10 ash is necessary for the water of hydration. This reduces to 1/9 of 1/10 ash for the hydrogen correction.

f) In converting calorific value to dry mineral-matter-free basis the heat evolved due to the combustion of pyrites in burning to sulphur dioxide should be subtracted from the calorific value as analyzed. The value to be deducted on this score is $27·8\ Sp$ cal/g where Sp is the percentage of sulphur as pyrites. As the pyrites content in most Indian coals is low (less than 0·5 percent) this correction is seldom necessary.

6.6 The analysis may be reported in the form indicated in Table 1.

---

*General classification of coal (*revised*).

TABLE 1 FORM FOR REPORTING OF RESULTS

(*Clause* 6.6)

Ref No. ....

Seam ....

Location ....

Sample No. .... Character of Sample ....

Received on .... Size ....

Prepared on .... Gross Weight ....

Nature of Sample .... Sampling Done by ....

PROXIMATE ANALYSIS	AS ANALYSED BASIS	AS RECEIVED BASIS	AS EQUILIBRATED AT 40°C AND 60 PERCENT RH BASIS	DRY BASIS	*dmf* BASIS	REMARKS (The colour of ash and the nature of coke bead to be recorded in this column)
Moisture, percent						
Ash, percent						
Volatile matter, percent						
Fixed carbon, percent						
Calorific value (gross) by bomb, Kcal/g						
Sulphur, total, percent						

(*Continued from page* 2)

Members	*Representing*
SHRI A. K. MITRA SHRI S. B. SARKAR (*Alternate*)	Coal Board, Calcutta
SHRI H. P. SAMANTA	The Tata Iron & Steel Co Ltd, Jamshedpur
SHRI SOMAR SEN GUPTA SHRI BANAMALI SEN (*Alternate*)	Coke Oven Managers' Association (Indian Section), Burnpur

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones: 323 0131, 323 3375, 323 9402
Fax : 91 11 3234062, 91 11 3239399, 91 11 3239382

Telegrams : Manaksanstha
(Common to all Offices)

Central Laboratory:	Telephone
Plot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010	8-77 00 32

Regional Offices:	
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	323 76 17
*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	337 86 62
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9, Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	832 92 95

Branch Offices:	
'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	550 13 48
‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003	55 40 21
Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28 88 01
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	8-71 19 96
53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	20 10 83
E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001	37 29 25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
Patliputra Industrial Estate, PATNA 800013	26 23 05
Institution of Engineers (India) Building 1332 Shivaji Nagar, PUNE 411005	32 36 35
T.C. No. 14/1421, University P.O. Palayam, THIRUVANANTHAPURAM 695034	6 21 17

---

*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, CALCUTTA 700072	27 10 85
†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	309 65 28
‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002	222 39 71

Printed at Simco Printing Press, Delhi