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भारतीय मानक मसौदा
पेट्रोलियम और उसके उत्पाद — परीक्षण पद्धतियाँ
भाग 6 बम कैलोरीमीटर द्वारा तरल हाइड्रोकार्बन ईंधन की दहन ऊष्मा का निर्धारण
(IS 1448 भाग 6 का दूसरा पुनरीक्षण)

Draft Indian Standard

**PETROLEUM AND ITS PRODUCTS — METHODS OF TEST
PART 6 DETERMINATION OF HEAT OF COMBUSTION OF LIQUID
HYDROCARBON FUELS BY BOMB CALORIMETER**

[Second Revision of IS 1448 (Part 6)]

(ICS 75.080)

Methods of Sampling and Test for Petroleum and
related Products of Natural or Synthetic Origin
(excluding bitumen) Sectional Committee PCD 01

Last date for receipt of comment is
27 January 2025

FOREWORD

(Formal clause will be added later)

This standard was first revised in 1984 and was published on based Institute of Petroleum Standard IP 12/73 (1980). The IP standard has been revised as IP 12/79 (2001).

This revision has been brought out to keep pace with the latest technological developments and international practices. In this revision following major changes have been made:

- a) References Clause has been incorporated.
- b) Specification of calorimeter bomb has been specified in Annex A.

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

1 SCOPE

This draft standard (Part 6) describes a procedure for the determination of the gross heat of combustion and net heat of combustion of liquid hydrocarbon fuels.

2 REFERENCES

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

<i>IS No./ Other Standards</i>	<i>Title</i>
IS 1448	Methods of test for petroleum and its products
(Part 33): 2021	Sulphur by High Pressure Decomposition Device Method (<i>third revision</i>)
(Part 34): 1979 PCD/01/22406 (F)	Determination of Sulphur in Petroleum Products by Lamp method (<i>second revision</i>)
(Part 161): 2017/ ISO 13032: 2012	Determination of Low concentration of sulphur in Automotive fuels — Energy-Dispersive X-Ray Fluorescence Spectrometric Method
(Part 160): 2017/ ISO 20846: 2011	Determination of Sulphur content of Automotive Fuels — Ultraviolet Fluorescence.
(Part 153): 2012/ ISO 20847: 2004	Determination of Sulphur content of Automotive Fuels —Energy-Dispersive X-Ray Fluorescence Spectrometry
(Part 159): 2018/ ISO 20884: 2011	Determination of Sulphur content of Automotive Fuels —Wavelength dispersive X-Ray Fluorescence Spectrometry.

3 SUMMARY OF METHOD

3.1 A weighed quantity of the sample is burned in the presence of oxygen in a bomb calorimeter under controlled conditions. The Gross heat of combustion at constant volume is calculated from the mass of the sample and the rise in temperature. Bomb calorimeter should have proper allowance for transfer of heat and for the formation of nitric and sulphuric acids in the bomb and no account being taken for other minor products formed. The value so obtained is the gross heat of combustion at constant volume.

4 TERMINOLOGY

For the purpose of Standard, the following terminology shall apply.

4.1 Gross Heat of Combustion

The quantity of energy released when a unit mass of fuel is burned in oxygen in a constant volume enclosure, with the products being gaseous, other than water that is condensed to the liquid state. The resultant materials in the bomb are considered as being gaseous oxygen, carbon dioxide, sulphur dioxide, nitrogen, and liquid water in equilibrium with its vapour and saturated with carbon dioxide, other compounds in solution, and solid ash. In this procedure, 25 °C is the initial temperature of the fuel and the oxygen, and the final temperature of the products of combustion. The gross heat of combustion is represented by the symbol Q_g .

4.2 Net Heat of Combustion

The quantity of energy released when a unit mass of fuel is burned at constant pressure, with all the products, including water, being gaseous. In this procedure, the combustion takes place at a constant pressure of 101.325 kPa (1 atm), and 25 °C is the initial temperature of the fuel and the oxygen, and the final temperature of the products of combustion. The net heat of combustion is represented by the symbol Q_n .

4.3 Unit of Measurement

The unit of measurement in this method is the joule. To convert calories per gram (Cal/g) to Joules, the following conversion shall be used:

$$1 \text{ Cal/g} = 4.1868 \text{ J/g}$$

5 APPARATUS

5.1 The room in which the bomb calorimeter is located shall be free from draughts and sudden temperature changes. The apparatus shall not keep a place where direct rays of sun hit the jacket or thermometers. The room shall have adequate lighting and ventilation, preferably with thermostatic control of room temperature and relative humidity.

5.2 Calorimeter Bomb — Conforming to the specification given in Annex A-1.

5.3 Calorimeter Vessel

Metal vessel polished on the outside, preferably at least 75 mm higher than the bomb, preferably of such cross-sectional area that 2 litres of water just cover the bomb.

5.3.1 Stirrer

A mechanical device for stirring the water in the calorimeter vessel at a uniform rate. The stirring shall not so rapid as to cause appreciable heating of the water. Continuous stirring of the water in the calorimeter vessel of the isothermal jacketed apparatus for 10 min after thermal equilibrium

has been established shall not cause an increase in temperature of more than 0.01 °C. With the adiabatic jacketed apparatus continuous stirring of the water in the calorimeter vessel for 10 min after thermal equilibrium has been established shall not cause an increase in temperature of more than 0.002 °C. The portion of the stirrer immersed in the calorimeter vessel shall be connected to the outside portion with material of low heat conductivity.

5.3.2 Water Jacket

A double walled metal vessel containing water, fitted with a lid and leaving an air gap of preferably 10 mm all around the calorimetric vessel.

5.3.2.1 The isothermal jacket shall be arranged to remain at a uniform temperature during the test and temperature variation should not cross 0.05 °C.

5.3.2.2 Adiabatic calorimeters shall be capable of circulating water continuously through both jacket and lid. It should be fitted with heating and cooling devices such that the temperature of the jacket water is rapidly adjusted to that of the water in the calorimeter vessel with a sensitivity sufficiently good to satisfy the requirements.

5.4 Fuel Cups

A range of fuel cups that may be used are illustrated in Annex A-2 and fuel cup assembly is illustrated in Fig. 1.

5.5 Ignition System

The ignition current should be drawn either from a step-down transformer connected to an alternating current line or from a storage battery. The firing voltage shall not exceed 24 volts and should be held to the lowest practicable level recommended by the manufacturer. An ammeter or a pilot light may be used in the circuit to indicate when current is flowing, and the circuit shall be controlled by a double-pole, momentary contact switch which remains normally open, except when held closed by the operator. The switch shall be held in the closed position not more than 2 s.

5.6 Thermometers

Temperatures be measured with thermometers conforming to any of the following types:

5.6.1 Bomb calorimeter thermometers

Having a 6 °C range, subdivided graduations not greater than 0.01 °C. Thermometers shall have been tested for accuracy at intervals of 0.5 °C or less over the entire graduated scale. Corrections shall be reported to 0.002 °C or better for each test point.

5.6.2 Platinum resistance, quartz crystal or thermistor thermometers

With elements enclosed in a metal sheath and connected to suitable electrical measuring equipment may be used provided the temperature measuring system meets the following requirements:

5.6.2.1 Readability — The last digit shall be readable to 0.001 °C.

5.6.2.2 Repeatability — In temperature rise measurements over the same 3 °C range ± 0.001 °C

5.6.2.3 Absolute accuracy — The error in any single temperature reading shall not exceed ± 0.150 °C.

5.6.3 Thermometer reading eyepiece — Magnifying type with adjustable focus.

5.7 Oxygen Charging Equipment — Conforming to the specification given in Annex A-3

6 REAGENTS AND MATERIALS

6.1 Water — Distilled or de-mineralized reagent water (*see* IS 1070).

6.2 Oxygen — Under pressure in cylinders, free from combustible material.

6.3 Ignition Wire

(0.1 ± 0.03) mm diameter platinum wire. The same diameter and length of wire shall be used in the calibration and determination.

6.4 Firing Cotton

A strand of wick as specified for use in IS 1448 (Part 34), dried at 100 °C to 105°C and stored over silica gel until required. The heat of combustion of each batch of firing cotton (if required) should be determined.

6.5 Sodium Hydroxide Standard Solution — 0.1 N.

6.6 Benzoic Acid

Powder or pellet form, of thermo-chemical standard of purity and of a heat of combustion certified by the National Physical Laboratory (NPL) or the National Institute of Standards and Technology (NIST). Powdered material shall be compressed into pellets.

6.7 Cellulose Adhesive Tape

Plain-transparent only cellulose based film of width not less than 25 mm and thickness 0.05 mm to 0.075 mm. The heat of combustion of each batch of tape shall be determined.

6.8 Indicator — The following alternatives should be used.

6.8.1 Methyl Purple Solution — 0.1 percent aqueous solution,

6.8.2 Methyl Red Solution — 60 percent alcohol-aqueous solution containing 0.1 percent indicator.

7 SAFETY PRECAUTIONS

7.1 The heat developed during the combustion shall in no case exceed 90 J/ml bomb capacity and the total weight of sample, together with any combustible material such as cellulose tape, shall be calculated in advance, so that the given limit is not exceeded.

7.2 Ensure that the oxygen filling pressure does not exceed the safety limit of 40 bar.

NOTE — The operator shall not go near the calorimeter until at least 20 seconds have elapsed from the time of firing.

7.3 The bomb shall be completely submerged in water during firing and shall not be ignited if there is any evidence of gas leakage.

7.4 Because of the risk of explosion, it is strongly recommended that the firing point and the oxygen cylinder be situated outside the room or safety enclosure containing the calorimeter. But in any case firing shall be by remote control in order to minimize the risk of injury to the operator (*see 5.5*).

8 SAMPLE

8.1 Collect liquid fuels of flash point less than 38 °C in a 200 ml capacity container completely as possible, close store samples in a refrigerator at below 4 °C until required. The fuel shall be transferred to the fuel cup at a temperature as near as possible to the storage temperature.

8.2 If results are required on a dry basis, determine the water content independently and correct the result accordingly.

9 STANDARDIZATION

9.1 Determination of Energy Equivalent (Effective Heat Capacity or Water Equivalent) of the Calorimeter

Calculate approximate weight of benzoic acid in g from the relation:

$$m = \frac{90 \times \text{Bomb capacity, ml}}{26500}$$

where

m = approximate mass of benzoic acid in g.

9.1.1 Weigh the fuel cup with Benzoic acid and its contents to the nearest 0.1 mg. Pipette 1ml of water into the bomb (to saturate the oxygen used for combustion with water vapour). Place the fuel cup in the circular ring support attached to the terminals of the bomb.

9.1.1.1 Attach a measured length of ignition wire (100 mm, *see 6.3*) across the bomb terminals so as to form a U-shape loop, the central portion of the loop is in contact with the benzoic acid.

OR

9.1.1.2 Attach a measured length of ignition wire (100 mm, *see 6.3*) across the bomb terminals under light tension. Weigh a suitable length of dry firing cotton and tie it at the centre of the ignition wire. Insert the free end of the firing cotton into the fuel cup so that it is in contact with the benzoic acid.

9.1.2 Close the bomb securely.

9.1.3 Fill oxygen slowly to a gauge pressure of 30 bar.

9.1.4 Determine the effective heat capacity of the system by making minimum 5 combustions of the certified benzoic acid using the standard quantity of water. The standard quantity of water is such that the cover of the bomb (but not terminals) is immersed and the effective heat capacity of the system is approximately 10500 joules/ °C. If necessary, make preliminary combustions of benzoic acid to establish the correct quantity of water. Once established this shall be the standard quantity of water, weighed to within 0.5 g, and used in all subsequent determinations.

9.1.5 When using isothermal apparatus follow procedure described in **10.4**. When using adiabatic apparatus follow the procedure described in **10.5**. Calculate the effective heat capacity as described in **11.1** to **11.4**

10 PROCEDURE

For liquid fuels of flash point below 38 °C follow as in **10.1.1** or **10.1.2** and continue the determination as in **10.3**, **10.4**, **10.6** and **10.7** if using isothermal apparatus or as in **10.3**, **10.5**, **10.6** and **10.7** if using adiabatic apparatus. For liquid fuels of flash point above 38 °C follow as in **10.2.1** or **10.2.2** and continue the determination as in **10.3**, **10.4**, and **10.7** if using isothermal apparatus or as in **10.3**, **10.5**, and **10.7** if using adiabatic apparatus.

10.1 For Liquid Fuels of Flash Point less than 38 °C

10.1.1 *Fuel Cup Type 1 or 4 Annex A2 Fig. 3*

Weigh the fuel cup to nearest 0.1 mg. Cover the cup with the cellulose adhesive tape, trim around the edge with a razor blade, and seal tightly. Make a hole on the tape with a large needle having opening somewhat larger than the diameter of the hypodermic syringe needle. Place a 4 mm × 12 mm strip of tape, creased in the middle and sealed along one side, in the centre of the tape disk to give a flap arrangement which will cover the hole when finally sealed down, as shown in Fig.1. Weigh the cup and tape, and remove from the balance with forceps. Add the required weight (*see Note*) of sample with a hypodermic syringe through the hole in the tape. Seal the flap down over the hole by pressing lightly with a metal spatula. Allow the temperature to reach equilibrium (*see 8.1*) and reweigh the cup, tape and sample. Avoid contacting the tape or cup with bare fingers. Place the fuel cup in the circular ring support attached to the terminals of the bomb and attach a measured length of ignition wire (*see 6.3*) across the bomb terminals such that the central portion of the loop presses down on the centre of the tape disk. Pipette 1 ml of water into the bomb and immediately close the bomb securely.

OR

10.1.2 *Fuel Cup Type 1 or 4 of Annex A2 Fig. 3*

Weigh the fuel cup to the nearest 0.1 mg. Cover the fuel cup top with two pieces of cellulose adhesive tape, so that they slightly overlap. Fix one end of a weighed length of firing cotton into the overlap, the other end being free or attachment to the ignition wire. Weigh the fuel cup and the attached tape and firing cotton to the nearest 0.1 mg. Lift the lip of the sealing tape and introduce the required weight (*see Note 1*) of liquid fuel with a hypodermic syringe. Replace the tape to seal the fuel cup, allow the temperature to reach equilibrium (*see 8.1*). Weigh the fuel cup and its contents to the nearest 0.1 mg. Pipette 1 ml of water into the bomb. Place the fuel cup in the circular ring support attached to the terminals of the bomb. Attach a measured length of ignition wire (*see 6.3*) across the bomb terminals under light tension. Tie the free end of the firing cotton to the centre of the ignition wire. With a pin make about six small holes in the tape sealing the mouth of the fuel cup. Immediately close the bomb securely.

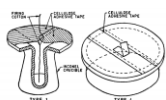


FIG. 1 SEALED FUEL CUP ASSEMBLIES

10.2 Liquid Fuels of Flash Point greater than 38 °C, (see Note 2)

10.2.1 Fuel Cup Type 2, 3 or 4 of Annex A2 Fig. 3

Place the required weight (see **Note 1**) of liquid fuel in the weighed fuel cup. Weigh the fuel cup and its contents to the nearest 0.1 mg. Pipette 1 ml of water into the bomb. Place the fuel cup in the circular ring support attached to the terminals of the bomb. Attach a measured length of ignition wire across the bomb terminals under light tension. Weigh a suitable length of dry firing cotton and tie it at the centre of the ignition wire. Dip the free end of the firing cotton into the contents of the fuel cup and immediately close the bomb securely.

OR

10.2.2 Fuel Cup Type 2, 3 or 4 of Annex A2 Fig. 3

Place the required weight (see **Note 1**) of sample in the weighed fuel cup. Weigh the cup and sample to nearest 0.1 mg. Place the fuel in the circular ring support attached to the terminals of the bomb. Attach a measured length of ignition wire so that the central portion of the loop just dips into the contents of the cup. Pipette 1 ml of water into the bomb. Immediately close the bomb securely.

NOTE

- 1) The weight of the sample controlled such that:

$$M = \frac{90 \times \text{Bomb capacity, ml}}{Q_g}$$

where

- $M =$ is the required weight of sample in g;
 $Q_g =$ is approximate heat of combustion of the sample in J/g

- 2) Liquid fuels having flash point in the range 38 °C to 60 °C may tend to lose a small proportion of their more volatile components on exposure to the atmosphere. Weigh the sample into the fuel cup and seal immediately in the bomb to avoid exposure to draughts.

10.3 Fill oxygen slowly to a gauge pressure of 30 bar.

10.4 Isothermal Jacket Calorimeters

Fill the water jacket with water at a temperature within 3 °C of room temperature and ensure that it remains constant throughout. Fill the calorimeter vessel with the standard quantity of water (see **9.1.4**) weighed to within 0.5 g. The initial temperature of the bomb and calorimeter shall be:

- i) in the range 15 °C to 32 °C;

- ii) above the dew point;
- iii) approximately 2.5 °C below the temperature of the water jacket and preferably within 2 °C of the temperature of standardization.

10.4.1 Place the prepared bomb, in the calorimeter vessel. Ensure that there is no leakage of oxygen, confirm that the firing leads are dead, and make the appropriate electrical connections. Put the covers in position, arrange the thermometer and stirrer so that they do not touch the bomb or vessel, and start the stirrer.

10.4.2 When the temperature is rising steadily and at least 5 min after starting the stirrer, record a series of temperature readings (*see Note 1*) at 1 min intervals for 5 min from which the temperature at the end of the sixth min that is, time of firing will later be found by extrapolation. The rate of temperature-rise in the 5 min period before firing shall be constant (*see Note 2*).

NOTE

- 1) Temperatures should be read to 0.001 °C. This will not be possible during the rapid period, and the most accurate readings which are practicable at this time should be recorded. A convenient method of effecting this is to mount a small buzzer directly on top of the thermometer, and operate it for a few moments before each reading. Use an eyepiece to measure thermometer reading.
- 2) The rate of temperature change with time shall be considered constant if the variation in the rate does not exceed 0.001 °C/min. The rate of change of temperature shall be calculated as the mean of the relevant recordings.

10.4.3 At the end of the 6 min fire the charge by closing the firing circuit for not longer than 2 s.

10.4.4 Record the temperatures at 45, 60, 75, 90 and 105 seconds after the time of firing. By interpolation between these readings the time at which the temperature-rise reached 60 percent of its final value will later be found (*see Note*).

NOTE — When the approximate heat of combustion of the fuel is known to within 1500 joule/g, the expected temperature rise may be determined from the relation:

$$t = \frac{MQg}{W}$$

where

Qg = approximate gross heat combustion, in I/g;

M = mass in g of sampling g; and

W = effective heat capacity of calorimeter in J/ °C.

Calculate the temperature at the 60 percent point by adding to the temperature at the time of firing, 60 percent of the expected temperature rise. Start an additional stopwatch at the time of the first reading of the series of temperature recordings made before firing, Stop the stopwatch when the calculated 60 percent temperature rise is attained and record the time.

10.5 Adiabatic Calorimeters

Fill the calorimeter vessel with the standard quantity of water, weighed to within 0.5 g (*see 9.1.4*). The initial temperature of the bomb and calorimeter shall be in the range of 15 °C to 32 °C, and above the dew point and preferably within 2 °C of the temperature of standardization. Ensure that the jacket is filled with water at a temperature close to, and preferably equal to or slightly below that of the calorimeter vessel. Place the prepared bomb in the calorimeter vessel. Ensure that there is no leakage of oxygen, confirm that the firing leads are dead and make the appropriate electrical connections. Place the covers in position, arrange the stirrer, thermometer, and temperature regulating device in position so that they do not touch the bomb or calorimeter walls. Switch on the calorimeter stirrer, jacket stirrer, heaters, and cooling coil and run the apparatus for 5 min to attain equilibrium.

10.5.1 If, at the start, the bomb is markedly different in temperature from the calorimeter vessel more than 5 min may be required to reach equilibrium.

10.5.2 Check that the apparatus is at equilibrium by recording the temperature to the nearest 0.001 °C at 1 min intervals. Successive readings shall not differ by more than 0.001 °C and shall not show any progressive tendency. When four successive readings have complied with the requirements consider the apparatus to be at equilibrium and record the temperature to the nearest 0.001 °C as the firing temperature (t_a). Fire the charge by closing the firing circuit for not longer than 2 s.

10.5.3 Record the temperature to the nearest 0.001 °C at 1 min intervals until the same temperature is recorded for three successive readings. Record this value as the final temperature t_f (*see 11.3*) (*see Note*).

NOTE — If a constant temperature is not obtained within 15 minutes of firing the apparatus should be considered suspect.

10.6 15 min after the ignition, remove the bomb from the calorimeter, dry the outside of the bomb and release the pressure uniformly at such a rate that the operation takes at least 1 min. Open the bomb and examine the inside for traces of unburned oil or soot; if either is found, discard the determination (*see Note*).

NOTE — The combustion of highly leaded fuels may cause appreciable corrosion inside a stainless steel bomb, but this is not to be taken as reason for rejecting the test result.

10.6.1 Rinse out the interior of the bomb, including the fuel cup, with a fine jet of distilled water, and collect the washings, which shall not exceed a volume of 350 ml, in a beaker. Add a few drops of methyl red indicator and titrate with the sodium hydroxide solution to determine the quantity of nitric and sulphuric acids formed in the combustion.

10.7 When the sulphur content of the sample is unknown, filter the neutralized washings through a medium filter paper such as a Whatman No. 40, and determine the sulphur content as prescribed in IS 1448 (Part 33)

11 Calculation and Report

11.1 Correct all temperatures for thermometer errors according to the certificate.

11.2 Temperature rise in Isothermal Jacket calorimeter.

Using data obtained in section **10.4** compute the temperature rise, Δt , in an isothermal jacket calorimeter as follows:

$$\Delta t = t_c - t_a - r_1 (b-a) - r_2 (c-b)$$

where

- Δt = Corrected temperature rise, in °C
- a = Time for firing, in min;
- b = Time (to the nearest 0.1 min) when the temperature rise reaches 60 percent of the total, in min;
- c = Time at beginning of period in which the rate of temperature change with time has become constant (after combustion), in min;
- t_a = Temperature at time of firing, in °C;
- t_c = Temperature at time, c , in °C;
- r_1 = Rate at which the temperature was rising during the 6 min period before firing, in °C/min;
- r_2 = Rate at which the temperature was rising during the 5 min period after time c in °C/min. If the temperature was falling r_2 is negative and the quantity $[- r_2 (c - b)]$ is positive.

11.3 Temperature rise in adiabatic Jacket calorimeter.

Using data obtained as prescribed in **10.5** and **11.1** compute the temperature rise, Δt in an adiabatic jacket calorimeter as follows:

$$\Delta t = t_f - t_a$$

where

- Δt = is the corrected temperature rise, in °C;
- t_a = temperature at time of firing, in °C; and
- t_f = final maximum recorded temperature, in °C.

11.4 Effective Heat capacity of the calorimeter.

Calculate the effective heat capacity of the calorimeter, W in joules °C as follows:

$$W = \frac{[Q_B + 1.2(25 - t_f)]m + e_1 + e_2}{\Delta t} - D_T$$

where

Q_B = Certified calorific value of the benzoic acid in J/g (see section 6.6);

m = Weight of benzoic acid, in g;

Δt = Corrected temperature rise, °C;

e_1 = Correction for the heat of formation of acid in J (1 ml of 0.1N NaOH = 5.9J);

e_2 = Correction for heat of combustion of firing cotton in J (if used)

D_T = Correction for test temperature variation [see note] in J/°C. (Apply this correction in whole numbers.)

NOTE

- 1) The term $[Q_B + 1.2(25 - t_f)]$ relates the certified heat of combustion of benzoic acid at 25 °C to the final temperature of the calorimeter.
- 2) The term D_T is an approximation which applied to adjust the effective heat capacity of the system at the mean temperature, T , of the determination to the reference temperature of 25 °C and is only necessary when the mean temperature of the bomb standardisation and the determination differ by more than 3 °C. It is calculated as follows:

$$D_T = \alpha M_w - \beta [M_s + (M_B/4)] J/°C$$

where

M_w = Weight of water in the system, kg;

M_s = Weight of stainless steel in system, in kg;

M_B = Weight of bronze and brass in system, in kg.

The values α and β are obtained from the graph in Fig. 4. The mean temperature T in °C is given by the relation:

$$T = \frac{t_a + t_f}{2}$$

where

t_a = Temperature at the time of firing in °C;

t_f = Final maximum recorded temperature in °C

Plot the calibrated values of D_T in J/ °C against values of T between 15 °C and 35 °C.

11.5 Gross Heat of Combustion

Calculate the gross heat of combustion a constant volume and at 25°C, in J/g from the equation:

$$Q_g = \frac{\Delta t \times (W + D_T) - e_1 - e_2 - e_3 - e_4}{M}$$

where

W = Effective heat capacity of the system at 25°C, in J/°C;

D_T = Correction at the mean temperature, T obtained from the data plotted as described in , in J/°C.

Δt = Corrected temperature rise, in °C;

e_1 = Correction for the heat of formation of acids, in joules. (1 ml 0.1 N NaOH = 5.9 J);

e_2 = Correction for firing cotton (if used) in joules (heat of combustion of batch X mass of cotton used);

e_3 = Supplementary correction for the sulphur content of the sample, in joules (58.6 x percent sulphur x mass of sample, in g);

e_4 = Correction for cellulose tape, if used, in joules (heat of combustion of batch x mass of tape used); and

M = Mass of sample, in g.

11.6 Net Heat of Combustion

Calculate the net heat of combustion (Q_n) in joules/g from the following equation:

$$Q_n = Q_g - 212.19H$$

where

H = Hydrogen content of the sample in percent by mass.

11.6.1 The following equation may be used for the calculation of net heat of combustion of aviation gasoline and turbine fuel when the hydrogen content of the sample is not known:

$$Q_n = 10023 + 0.7195 Q_g \text{ (see Note).}$$

NOTE — This equation is only recommended if the percentage of hydrogen is not known. It is based on the previous equation and an empirical relationship between Q_n and the percentage of hydrogen in aviation gasoline and turbine fuels, developed from data by R. S. Jessup and C. S. Gragoe. 'Heat of Combustion of AN-F-28 Aviation Gasoline', National Advisory Committee of Aeronautics, Tech. Note 996. June 1945, and J. A. Cogliano and R. S. Jessup'. 'Relationship between Net Heat of Combustion and Aniline Gravity Product of Aircraft Fuels, National Bureau of Standards Report 2348, March 1953.

11.7 To obtain the gross or net heat of combustion in cal/g divide Q_g or Q_n by the appropriate factor given in section 4.

11.8 Report the result as the Gross or Net heat of combustion to the nearest 20J/g or to the nearest 5 cal_{IT}/g.

12 Precision

The following criteria should be used for judging the acceptability of results (95 percent confidence).

12.1 Repeatability

Duplicate results by the same operator should be considered suspect if they differ by more than 276 J/g (66 cal/g).

12.2 Reproducibility

The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than 773 J/g (185 cal/g).

ANNEX A
(Clauses, 5.2, 5.4, 5.7, 10.1.1, 10.1.2, 10.2.1 and 10.2.2)
APPARATUS FOR DETERMINATION OF THE HEAT OF COMBUSTION OF
LIQUID FUELS

A-1 CALORIMETER BOMB SPECIFICATION

A-1.1 General

This specification covers the essential constructional features and materials, and testing of bombs to be used for determining the heat of combustion of liquid fuels, or their Sulphur content, in accordance with this test method.

A-1.2 General Design

The proportions of the bomb shall conform to Fig.2

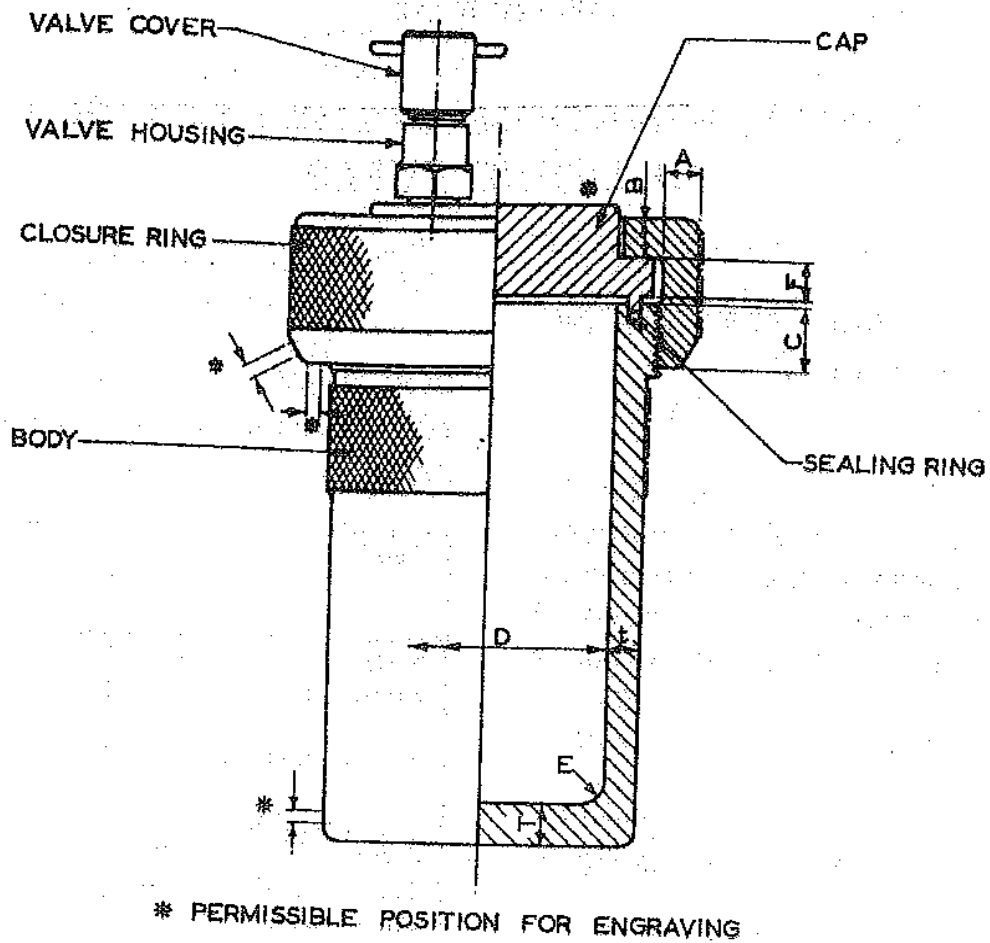


FIG. 2 TYPICAL BOMB FOR HEAT OF COMBUSTION

Internal diameter of bomb shall not be less than 55 mm and a capacity of (325 ± 25) ml. The weight of the bomb shall not exceed 3.25 kg not should be leak proof and can be easily drained. All parts enclosing the gas space should be constructed with materials which are not affected sufficiently by the combustion process. The electrodes shall be made of heat and corrosion resistant material, e.g. Platinum or its alloys, suitable steels or nickel chrome. The electrodes are placed such that when the fuel cup is in position its base shall be not less than 90 mm from the bottom of the bomb lid. The closure of the bomb shall be so designed that in normal operation the bomb can be sealed and opened by hand without the use of tools of any type. The calorimeter bomb seal shall be so designed that after hand-tightening an increase of pressure in the bomb will be tighten the seal and prevent leakage.

A-1.3 Materials

A-1.3.1 The bomb cap and body shall be machined from solid or hollow forgings or bar. They shall not be fabricated from components welded or brazed together.

A-1.3.2 For a bomb designed the material shall have the following mechanical properties given in Table 1 and the minimum dimensions and dimensional ratios given in Table 2.

Table 1 Mechanical properties of the material.

(Clause A-1.3.2)

Sl. No (1)	Mechanical Properties (2)	Specifies Requirement (3)
1.	Tensile strength	Shall be not less than 550 NM/m ²
2.	Yield stress	Shall be not less than 210 NM/m ²
3.	Elongation	Shall not break less than 30 percent
4.	Izod impact	Shall be not less than 68 NM

Table 2 Dimensions of bomb body, closure ring and screw thread.

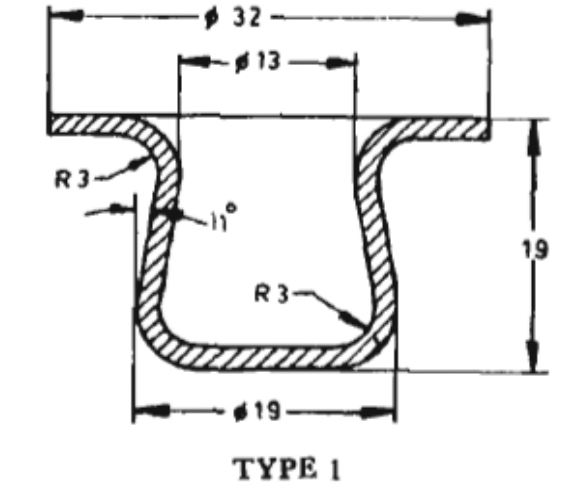
(Clause A-1.3.2)

Sl. No (1)	Detail (2)	Symbol (See Fig.2) (3)	Requirement (4)
1.	Body		
i)	External diameter		To be uniform within 0.03 mm
ii)	Internal diameter	D	Shall be not less than 55 mm
iii)	Capacity of the Combustion chamber		(325 ± 25) ml

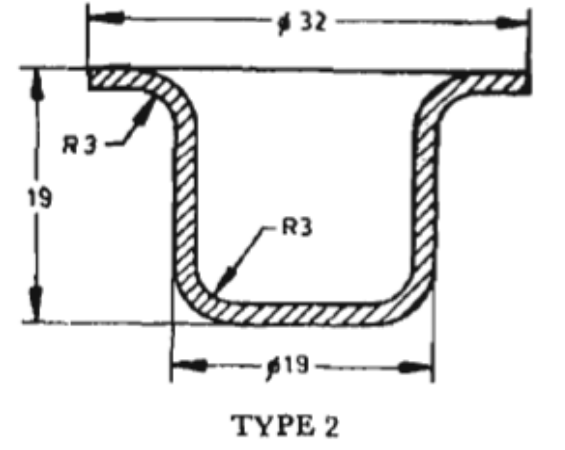
iv)	Thickness of side wall	t	Not less than 0.10D
v)	Thickness of base	T	Not less than 0.165 D
vi)	Internal radius	E	Not less than 't'
2.	Closure Ring		
i)	Dimensions of closure ring when made of aluminum bronze:		
a)	Thickness of wall	A	Not less than 0.14 D
b)	Thickness of flange	B	Not less than 0.17 D
c)	Screw Thread		
d)	Normal pitch thread form, and fit		ISO metric threads 1.5 mm or 2 mm pitch shall be used with a 6H/6g (medium) fit complying with BS 3643. ISO inch (unified) threads

A-2 FUEL CUPS

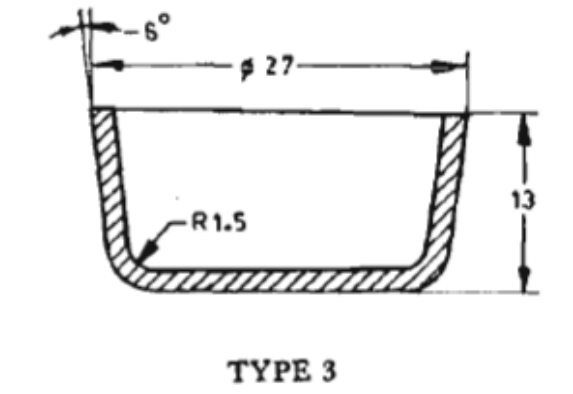
A-2.1 The fuel cups illustrated in Fig. 3 are suitable for use in this method.



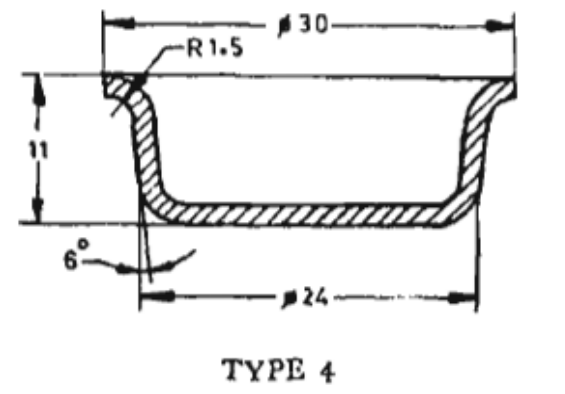
MATERIAL	THICKNESS	APPROX MASS
Inconel	0.46	8.3 g



MATERIAL	THICKNESS	APPROX MASS
Inconel	0.46	8.3 g



MATERIAL	THICKNESS	APPROX MASS
Type 310 Stainless Steel	0.8	9 g
Hastalloy C	0.7	9 g
Platinum 3½ percent Rh	0.35	11 g



MATERIAL	THICKNESS	APPROX MASS
Platinum 3½ percent Rh	0.35	11 g

All dimensions in millimetres.

FIG 3 FUEL CUPS

A-3 OXYGEN CHARGING EQUIPMENT

A-3.1 The valves, gauges, filling tube and fittings used in the oxygen charging system must meet industry safety codes and must be rated for use at input pressure up to 206 bar (3000psig) and discharge pressure up to 55 bar (800 psig). Separate gauges should be provided to show the supply pressure and the bomb pressure. The bomb gauge should be not less than 75 mm in diameter and preferably graduated from 0 to 55 bar in 1 bar sub-divisions. Both gauges must be absolutely oil-free and must never be tested in a hydraulic system containing oil. The charging equipment should include either a pressure reducing valve which will limit the discharge pressure to a maximum of 40 bar (600 psig) or relief valve set to discharge at 40 bar (600 psig) in case the bomb should accidentally be overcharged. Means should also be provided for releasing the residual pressure in the filling tube after the bomb valve has been closed.

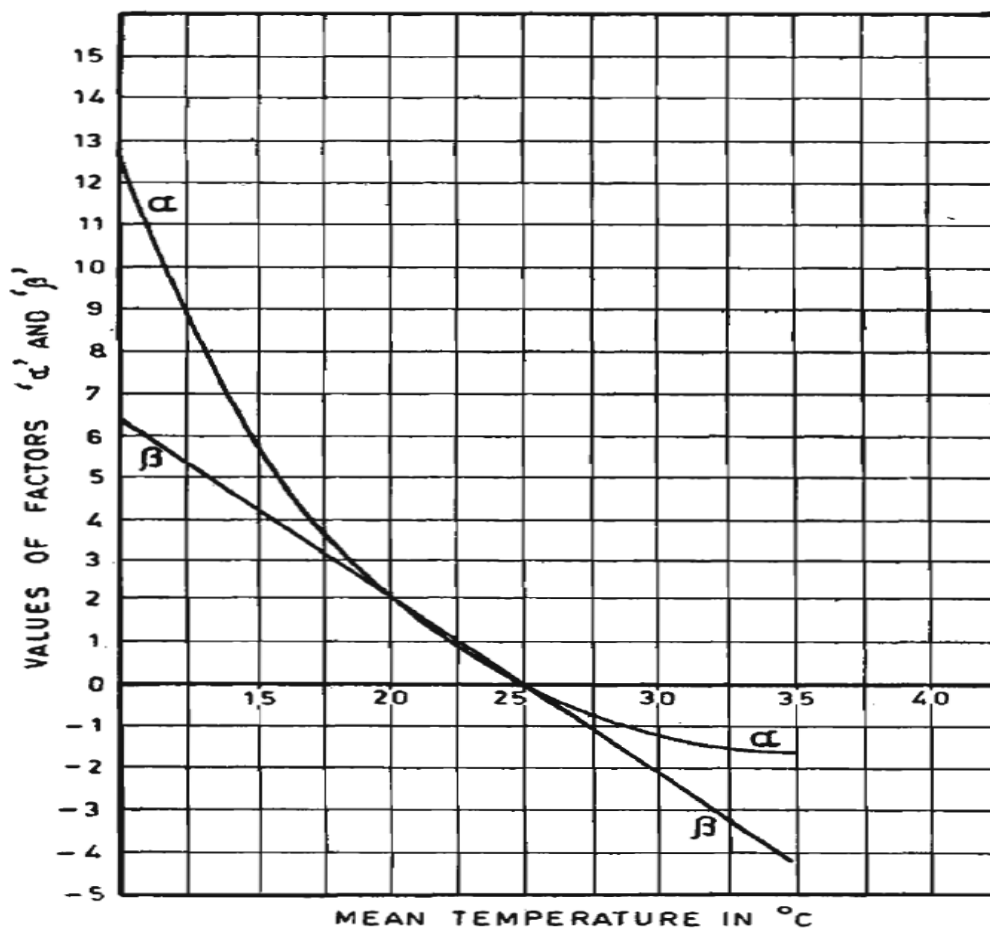


FIG 4 VALUE OF α AND β FOR CALCULATING D_T