FUEL TECHNOLOGY 1
LAB MANUAL

LIST OF EXPERIMENTS

1. To determine the calorific value and water equivalent of a supplied sample by using Bomb calorimeter (manually)
2. To determine the calorific value and water equivalent of a supplied sample by using Bomb calorimeter (Automatically)
3. To determine the proximate analysis of coal sample
4. To determine the ultimate analysis of the coal sample

EXPERIMENT NO-1

AIM OF THE EXPERIMENT: To determine the calorific value and water equivalent of a supplied sample by using Bomb calorimeter (manually)

APPARATUS REQUIRED:

1. Bomb calorimeter
2. Weighing machine
3. Cotton thread and nichrome wire

SAMPLE REQUIRED: Powered sample (1gm)

THEORY:

A bomb calorimeter is an apparatus used for measuring heats of combustion. Digital bomb calorimeter provides a simple, inexpensive and an accurate method for determination of both heat of combustion and sulphur contents of all types of liquid and solid hydrocarbons. Bomb calorimeter is widely used in industries in diversified fields for quality control, testing laboratories for research & development and in teaching establishment. The accessories supplied are complete for analysis as per method recommended by the Indian standards institutions (IS1350-1966), British standards institutions (BSI 1016 part 5-1967) and the institute of petroleum (IP 12/63 T). Each component of the package has been finished and tested according to the specifications by this institution.

PRINCIPLE OF OPERATION

A sample is weighed and places in a heavy duty stainless steel cylinder referred to as "Bomb". The bomb is then sealed with oxygen and the sample is ignited electrically. The complete oxidation of the compound release heat and this is measured through the temperature change of the water bath surrounding the bomb. A digital sensor measures the rise in temperature. The heat of combustion at SEROLOGICAL volume can be calculated from the resulting rise in temperature.

DESCRIPTION OF COMPONENTS BOMB:

The bomb is a stainless steel cylindrical vessel having capacity of 300 ml. The volume of the bomb does not change during a reaction and the walls are strong enough to easily bear the normal operating pressure of 26 kg/cm². The bomb is also tested at 300 kg/cm² hydraulic pressure. Burning of samples at high pressure, the nitrogen and sulphur contents are oxidized to nitric and sulphuric acids respectively. The corrosion resistant nature of the bomb protects it from the corrosive vapours. The lid is provided with two terminals for electrical connections. Each terminal is connected to an electrode with small grooves for attaching the

fuse. One of the electrodes is provided with a crucible holder. The caps on the electrodes compress the fuse wire. The lid is also provided with a small hook for lifting purposes and a "valve body" with fitted Schrader valve for filling oxygen in the bomb.

BOMB: It is tested for its performances as per the requirements of the Institute of petroleum (IP 12/63 T). The test is conducted at 300 kg/cm² hydraulic pressure and the pressure is maintained for a period of 10 minutes without any sign of leakage.

WATER/PUFF INSULATED JACKET: It is made of stainless steel and is highly polished on the inside and outside to minimize radioactive losses. A pole is provided on the top of the jacket to hold the stirrer unit and an inlet through which is added. A screw is located at the bottom for draining the water in the jacket. There is no need to add water to puff insulated jacket. Two terminals for electrical connections from firing unit to bomb are provided on the jacket.

OFFSET STIRRER: Stirrer is driven at a serological speed of 800 RPM by a motor through a heat insulated rubber belt. The motor unit is kept at sufficient distance from the vessel to eliminate heating. This arrangement does not raise the temperature of water by even 0.1°C in 10 minutes. Thus easily meet the specific requirements by the Indian institution, the British Standard Institution and the Institute of Petroleum.

CALORIMETER VESSEL: The bomb is placed in the vessel during experiment. It is made of copper and is brightly polished.

COMBINED LID: It is used as a cover for calorimeter vessel and jacket.

PRESSURE GAUGE ON STAND: An accurate pressure gauge is supplied for measurement of pressure of oxygen in the bomb. The dial is graduated from 0 to56 kg/cm² (0 to about 800 lb/in^2). Normally the oxygen is filled in the bomb at the pressure of 26 kg/cm².

SAFETY DEVICE: Accidentally if more oxygen is filled in the bomb than a plain disk in each safety device will burst and excess oxygen will lead to ensure safety of the bomb.

OXYGEN CONTROL VALVE: It is attached to the oxygen cylinder to provide fine adjusting for filling oxygen in the bomb.

GAS RELEASING PIN: It is used to release the pressure from the bomb.

PELLET PRESS: It is necessary that solid samples are air dried and grounded to powder from until all particles pass through a 60 mesh screen. The particle size is imported because the combustion reaction proceeds to completion within a few seconds and if any of the individual particles are too large or small they will not burn completely. Pellet press offers a possible solution to this problem since pellets are easier to handle than loose samples.

CRUSIBLE: Stainless steel crucible is offered as a standard with instrument. Platinum quartz and nickel crucibles are also being offered as accessories at extra cost.

IGNITION WIRE/COTTON THREAD: Nichrome wire and cotton thread of known calorific values are used as combustion aid.

PROCEDURE:

1. ATTACHING IGNITION WIRE:

Cut a single length of fuse wire 6 cm long. Insert the wire in the slots on the electrodes and firmly compress it by the adjustable caps. Ignition process requires 10 cm cotton thread to carry the flame to the samples.

2.WATER IN THE BOMB:

Place 1-2 ml of distilled water in the bomb from a pipette for a saturated atmosphere.

3. FILLING OXYGEN IN THE BOMB:

Connect the oxygen filling assembly. Connect copper tubes from oxygen control valve to pressure gauge to safety device to bomb. Make sure that the connections are proper to ensure that there is no sign of leakage. Slowly rotate the oxygen valve in the direction pointing "on" observe the gauge and allow the pressure to rise until the desired point is reached about 26 kg/cm^2 simultaneously close the valve.

4. ADDING WATER TO CALORIMETER VESSEL:

Place the bomb carefully in the calorimeter vessel. Add distilled water until the upper surface of closers ring of the bomb is at a depth of 1-2 cm. Stop adding water where the SR. NO. engraved on the bomb is covered with water, record the quantity of water added. Another method of adding water is by weight. On a balance determine the weight of completely dried calorimeter vessel and add (1750 ±1) grams of distilled water. Now place the vessel in the jacket and lower the bomb carefully. Make water adjustment until the upper surface of closure ring of the bomb is at a depth of 102 cm. Now knowing the weight of water in the vessel (1750 ±1 grams), calculate the amount of water required for the experiment.

5. STANDARDIZING THE CALORMETER

Bomb calorimeter is standardized by igniting a pellet of a supplied sample weighing one gram or less of known calorific value in oxygen sealed bomb. By igniting supplied sample, water Equivalent (W) is evaluated which is the weight of water equivalent in effective heat capacity to the entire system (calorimeter vessel containing a specified weight of water, bomb charged with oxygen and combustion aid). It will vary from one apparatus to the next, because the precise mass of each component will very slightly. You need to know this value

in order to determine how much heat is generated by combustion of your sample. Once water equivalent (W) is evaluated the same value of W is substituted in the equation for determining the calorific value of the samples (CVs). However, precautions listed below must be followed. When performing the experiment to determine water Equivalent (W) using a supplied sample. The same volume of distilled water must be used for all experiments. (Follow the procedure above for adding water to calorimeter vessel). Use the same length of ignition wire and cotton thread as recommended 6 cm wire and 10 cm thread. It is recommended to perform the experiment of water Equivalent 3 to 5 times and the mean of the reading should be taken as W. It is recommended to calibrate the system using of a supplied sample after every 25 tests. For precise results, substitute the mass of the sample to the third place of the decimal (.001) in the formula.

TABULATION

Sl. No	Temperature (^oC)	Time (sec)

CALCULATION:

The following formula is the amended formula to calculate the W.E of the Bomb Calorimeter taking the Fuse Wire into consideration. It is to be noted that the calorific value for Nichrome Fuse Wire is known and is 333.68 Cal/gm.

$$W.E = \frac{(\mathrm{M})(\mathrm{H})+(\mathrm{Ew})+(\mathrm{Et})}{\Delta \mathrm{T}}$$

Where,

W. E=water equivalent=2548.43 Cal/gm

M=mass of the sample i.e., 1 gm

H=heat capacity or the calorific value of the taste sample

ΔT= rise in temperature

E_w=correction of heat of combustion for Nichrome fuse wire =1.6684 ca

E_t= correction of heat of combustion for cotton thread=76.912 cal

Heat capacity or calorific value= $H = \frac{\mathrm{WE}*\Delta\mathrm{T}}{\mathrm{M}}$ cal/gm

RESULT:

The water equivalent and the heat capacity of a supplied sample is found to be _____ and _____.

SAFETY PRECAUTIONS:

The operator must follow the following basic points in order to operate the Oxygen Bomb Calorimeter safely:

1. Do not use too much sample. The bomb cannot be expected to withstand the effects of combustible charges which liberate more than 10,000 calories. This generally limits the total weight of combustible material (sample + gelatine, firing oil or any combustible aid) to not more than 1.10 gm. Do not charge with more oxygen than is necessary and do not fire the bomb if an overcharge of oxygen should accidentally be admitted.
2. Keep all parts of the bomb, especially the insulated electrode assembly in good repair at all times. Do not fire the bomb if gas bubbles are leaking from the bomb when it is submerged in water.
3. Stand back from the calorimeter for at least 15 seconds after firing and above all, keep clear of the top of the calorimeter. If the bomb explodes, it is most likely that the force of explosion will be directed upwards.
4. Proceed with caution and use only a fraction of the allowable maximum sample when testing new materials which burn rapidly or which have explosive characteristics.
5. The bomb should be kept dry and clean and free from dust.

BOMB CALORIMETER (MANUAL)

EXPERIMENT NO - 2

AIM OF THE EXPERIMENT: To determine the calorific value and water equivalent of a given supplied sample by using Bomb calorimeter (Automatically)

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SAMPLE REQUIRED: Powered sample (1gm)

THEORY:

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4. ADDING WATER TO CALORIMETER VESSEL:

Place the bomb carefully in the calorimeter vessel. Add distilled water until the upper surface of closers ring of the bomb is at a depth of 1-2 cm. Stop adding water where the SR. NO. engraved on the bomb is covered with water, record the quantity of water added. Another method of adding water is by weight. On a balance determine the weight of completely dried calorimeter vessel and add (1750 ±1) grams of distilled water. Now place the vessel in the jacket and lower the bomb carefully. Make water adjustment until the upper surface of closure ring of the bomb is at a depth of 102 cm. now knowing the weight of water in the vessel (1750 ±1 grams) calculate the amount of water required for the experiment.

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TABULATION:

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Heat capacity or calorific value= $H = \frac{WE * \Delta T}{M}$ cal/gm

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2. Keep all parts of the bomb, especially the insulated electrode assembly in good repair at all times. Do not fire the bomb if gas bubbles are leaking from the bomb when it is submerged in water.

3. Stand back from the calorimeter for at least 15 seconds after firing and above all, keep clear of the top of the calorimeter. If the bomb explodes, it is most likely that the force of explosion will be directed upwards.

4. Proceed with caution and use only a fraction of the allowable maximum sample when testing new materials which burn rapidly, or which have explosive characteristics.

5. The bomb should be kept dry and clean and free from dust.

BOMB CALORIMETER (AUTOMATIC)

EXPERIMENT NO: 3

AIM OF THE EXPERIMENT: To determine the proximate analysis of coal

APPARATUS REQUIRED:

1. Silica crucible with lid
2. weight balance
3. Muffle Furnace
4. Oven

SAMPLE REQUIRED: Powered coal sample (1gm)

THEORY:

PROXIMATE ANALYSIS: The proximate analysis of coal comprises of determination of the moisture, ash, volatile matter and fixed carbon.

MOISTURE: Free water may exist in the coal as adsorbed on the surface, condensed inside fine capillary network and as bound to the coal molecule by chemisorption and hydrogen bonding.

VOLATILE MATTER: A volatile product obtained by the pyrolysis of coal in the absence of air is known as volatile matter. The product may contain hydrogen, methane, carbon monoxide, carbon dioxide, higher hydrocarbons, tar, water vapours, nitrogen, ammonia, hydrogen sulphide etc. The pyrolysis temperature of coal may lie in the range from 600-800 ℃. The yield of volatile can be taken as a measure of its rank. Volatile matter will be much less in coke than that of coal as pyrolysis had occurred during coking at around 1000 ℃. Volatile matter does not contain the moisture of coal but it contains water that is formed from the hydrogen and oxygen of coal during the decomposition.

ASH: Coals are associated with certain mineral or inorganic matter, which gets deposited along with vegetable matter or gets into coal by subsequent infiltration. The ash consists mainly of silica, alumina, iron oxide and lime. When heated, coal as does not melt sharply at any temperature, but begins to soften at much lower temperature than that required melting. The ash content in coke is much higher than in coal.

FIXED CARBON: Fixed carbon is obtained by deducting the sum of moisture, ash and volatile matter percentage from 100.

PROCEDURE:

MOISTURE DETERMINATION:

1. Dry the silica dish in an oven and weigh.
2. Spread out about 1 gm of 20 mesh coal sample on the dish.

3. Weigh the dish again to find the exact mass of the sample.
4. Heat the dish without any cover in the oven at about 105-110 ℃ for 1 hour.
5. Take out the dish from the oven, cover it with the lid and cool.
6. Weigh the dish to find the loss in weight of coal due to presence of moisture.

VOLATILE MATTER DETERMINATION:

1. Heat a clean crucible and its lid at 950 ℃ for 7 minutes in muffle furnace.
2. Allow the crucible and lid to cool on a metal plate for a minute and in desiccators for 10 minutes.
3. Weigh the crucible and lid together.
4. Put near 1 gm sample and weigh again to know the exact mass.
5. Insert the crucible with the lid on it into the furnace at 950 ℃ and keep there for a period of 7 minutes.
6. Weigh the crucible with the lid to know the weight loss due to expulsion of volatile matter.

ASH DETERMINATION:

1. Follow the steps 1 to 3 of moisture determination.
2. Insert the open dish in the furnace at 750 ℃ for an hour.
3. Remove the dish; allow it to cool for 10 minutes on the slab and 15 minutes in the desiccators.
4. Weigh the dish to find the mass left which is the ash content of the coal.

FIXED CARBON DETERMINATION:

1. The difference between the mass of coal taken and sum of volatile, ash, and moisture content give the fixed carbon content.

TABULATION:

Proximate analysis of coal samples

Sl. No	Temp (℃)	Time (min)	Dish wt.	Dish + sample wt.	Dish + Residue wt.

CALCULATION:

% of moisture in coal = (Loss in wt. of coal /wt. of coal initially taken) *100

% of volatile matter = (Loss in wt. of moisture free coal / wt. of moisture free coal) *100

% of Ash in coal = (wt. of residue ash formed / wt. of coal initially taken) *100

% of fixed carbon =100 - (% of moisture + % of volatile matter + % of Ash content)

CONCLUSION:

From the above experiment we have to find out the % of moisture content, % of volatile matter content, % of ash content and % of fixed carbon content of a given supplied coal.

EXPERIMENT NO - 4

AIM OF THE EXPERIMENT: To determine the ultimate analysis of the coal sample.

SAMPLE REQUIRED: Powered coal sample (1gm).

THEORY:

1. Coal is composed primarily of carbon along with variable qualities of other elements, like hydrogen, sulphur, oxygen, nitrogen.
2. Ultimate analysis of coal, also known as elemental analysis, is the method of determination of its total carbon, oxygen, nitrogen, sulphur and ash content in the given sample of coal.
3. Ultimate analysis is required as it is essential to understand the properties of bio mass material to evaluate their utility.
4. As chemical food stock and also design suitable gasifier systems, the analysis is carried out to fine oxygen required for combustion for efficient production of energy.
5. It gives analysis in terms of elementary constituents and is helpful in combustion calculation for design of furnaces and its auxiliaries.

PROCEDURE:

From the proximate analysis of coal, we have found that:

% of moisture = 2 % (M)

% of volatile matter = 30 % (VM)

% of ash content=16 % (A)

% of fixed carbon content = 52 % (FC)

By using the above data of proximate analysis, the ultimate analysis of the supplied coal sample i.e. the percentage of hydrogen, nitrogen, sulphur, oxygen and carbon can be calculated.

CALCULATION:

1. % of carbon content = 0.97(FC) +0.7(VM-0.1A)-M (0.6-0.01M)
2. % of hydrogen content = 0.036 (FC) + 0.086(VM-0.1A)-0.035M^2(1-0.02M)
3. % of Nitrogen content = 2.10-0.020(VM)

4. % of Sulphur content = 0.8%
5. % of oxygen content =100- (% C +% H+% N+% S)

CONCLUSION:

From the above experiment we have to find out the % of carbon content, % of hydrogen content, % of nitrogen content, % of sulphur content and the % of oxygen content of a given coal sample.