LIST OF EXPERIMENTS

1. To determine the calorific value of coal using Bomb Calorimeter.
   Equipment/Materials: Different grades of coal, Kbr press and Bomb calorimeter

2. To determine the calorific value of coke using Bomb Calorimeter.
   Equipment/Materials: Different grades of coke, Kbr press and Bomb calorimeter

3. Proximate Analysis of coal and coke.
   Equipment/Materials: coal, coke and Muffle furnace

4. To determine flash point and fire point of diesel, Pensky-Martins Apparatus.
   Equipment/Materials: diesel and Pensky-Martins Apparatus

5. To determine flash point and fire point of petrol, Pensky-Martins Apparatus
   Equipment/Materials: petrol and Pensky-Martins Apparatus

6. To determine the effect of temperature on Kinematic Viscosity of glycerine by Redwood Viscometer.
   Equipment/Materials: glycerine and Redwood Viscometer
BOMB CALORIMETER

AIM OF THE EXPERIMENT
To determine the water equivalent of the calorimeter using the given sample of solid or liquid fuel of known calorific value (or) To determine the calorific value of the given solid or liquid fuel if the water equivalent of the calorimeter known.

APPARATUS
Bomb, water jacket, stirrer, calorimeter vessel, combined lid, sensitive thermometer, analytical balance with weight box, oxygen cylinder with pressure gauge, fuse wire, cotton thread, firing unit, regulating valve and crucible hand pellet press (KBr press).

PRINCIPLE OF OPERATION
A Bomb Calorimeter will measure the amount of heat generated when matter is burnt in a sealed chamber (Bomb) in an atmosphere of pure oxygen gas. A known amount of the sample of fuel is burnt in the sealed bomb, the air in the bomb being replaced by pure oxygen under pressure. The sample is ignited electrically. As the sample burns heat is produced and rises in the temperature. Since the amount of heat produced by burning the sample must be equal to the amount of heat absorbed by the calorimeter assembly, and rise in temperature enables the determination of heat of the combustion of the sample. If
\[ W = \text{Water equivalent of the calorimeter assembly in calories per degree centigrade.} \]
\[ T = \text{Rise in temperature (registered by a sensitive thermometer) in degrees centigrade.} \]
\[ H = \text{Heat of combustion of material in calories per gram.} \]
\[ M = \text{Mass of sample burnt in grams.} \]
Then \[ W \times T = H \times M \]
If the water equivalent of the calorimeter is to be determined, a substance like Benzoic acid has a stable calorific value can be burnt in the bomb. Assuming the calorific value of Benzoic acid and water equivalent can be determined.

CALORIFIC VALUE
**Gross or higher calorific value:** The total amount of heat produced when one unit mass of fuel has been burnt completely and the products of combustion have been cooled to room temperature.

**Net or Lower Calorific Value:** The net heat produced when unit mass of fuel is burnt completely and the products are permitted to escape.
\[ LCV = HCV – \text{Latent heat of water vapour formed} \]
DESCRIPTION

I. BOMB
The bomb consists of three parts i.e. bomb body, lid and the cap. Bomb Body and the lid are made of corrosion resistant stainless steel containing Chromium, Nickel and Molybdenum. The bomb body is cylindrical vessel having a capacity of 300 ml. The walls are strong enough to withstand the normal operating pressure (30 atm) to extreme high pressures (300 atm). During burning at high pressure the nitrogen and sulphur contents are oxidized to nitric acid and sulphuric acid respectively. The corrosion resistant nature of the bomb material protects it from corrosive vapors. The bomb has lid, which is provided with two terminals. The metallic rods pass through the terminals one of which are provided with a ring for placing the crucible with a small hook and the other with a groove. Each rod is also provided with a ring to press the fuse wire attached to it. The upper side of the lid also provided with a small hook rod lifting and with a Schrader valve for filling oxygen in the bomb.

II. WATER JACKET
The water jacket is made of copper and is highly chromium plated on the inside and outside to minimize radiative losses. The jacket is filled with water.

III. STIRRER UNIT
A stirrer is provided which is driven directly by an electric motor. The stirrer is immersed in the water. The water is continuously stirred during the experiment for uniform heat distribution.

IV. COMBINED LID
This is made of Borolite sheet and is provided with a hole for to keep the stirrer unit in fixed position and hole to insert the temperature sensor. It has also another hole to take out the connecting wires from the terminals on the bomb lid to firing unit.

V. HAND PELLET PRESS
It is used for pressing the powder into a pellet.

VI. CRUCIBLE
It is made of stainless steel. The fuel to be burnt is weighed in this crucible.

VII. IGNITION WIRE
It is recommended that platinum wire used but an alternative nichrome wire is also being offered.

VIII. FIRING UNIT
It consists of the firing key, provision to give power to the stirrer motor, a switch for operating the stirrer motor, two indicating lamps. When the circuit is completed the indicating lamp
glows. After the firing key is closed on, the fuse wire burns, the indicating lamp stops glowing indicating the burning of the fuse wire.

**PROCEDURE**

- About 0.5 to 1 grm of finely ground benzoic acid (Preferably compressed into a pellet) is accurately weighed and taken into crucible.
- Place the bomb lid on the stand provided and stretch pieces of fuse wire across the electrodes (metal rods) provided in the lid tie about 5 cm of sewing cotton round the wire.
- Place the crucible in position and arrange the loose end of the cotton thread to contact the Benzoic acid pellet in the crucible.
- About 10 ml of distilled water are introduced into the bomb to absorb vapors of sulphuric acid and nitric acids formed during the combustion and lid of the bomb is screwed.
- Charge the bomb slowly with oxygen from the oxygen cylinder to a pressure of 25 atm. close the value and detach the bomb from the oxygen supply. Fill the calorimeter vessel with sufficient water to submerge the cap of the bomb to a depth of at least 2mm leaving the terminals projecting lower the bomb carefully in the calorimeter vessel and after ascertaining that it is gas tight, connect the terminals to the ignition circuit.
- Adjust the stirrer and place the temperature sensor and cover in position. Start the stirring mechanism, which must be kept in continuous operation during the experiment after stirring for 5 minutes note the temperature reading of the calorimeter. Close the circuit momentarily to fire the charge and continue the observations of the temperature at an interval of one minute till the rate of change of temperature becomes constant.
- Afterwards stop the stirrer and remove the power supply to the firing unit. Remove the bomb from the calorimeter and relax the pressure by opening the value. Verify that the combustion is complete and washout the contents of the bomb clean and dry.
- Calculate the calorific value of the fuel or water equivalent of the calorimeter.

**OBSERVATIONS:**

Weight of the empty crucible \( W_1 \) = \( gm \)

Weight of the empty crucible + Benzoic acid pellet \( W_2 \) = \( gm \)

Weight of the benzoic acid pellet \( W_2 - W_1 \) = \( gm \)

Weight of water taken in the calorimeter \( W_3 \) = \( gm \)

Temperature of the water just before firing \( t_1 \) = \( ^0C \)
Temperature of the water after firing \( t_3 = 0 \)°C

**CALCULATIONS**

Heat produced by burning of benzoic acid + Heat produced by burning of fuse wire and cotton wire etc = Heat absorbed by calorimeter.

\[
(W_2 - W_1) \times C_v = (W_3 - W_0)(t_2 - t_1)
\]

**PRECAUTIONS**

Sample should not exceed 1 gms.

Don’t charge with more oxygen than is necessary.

Don’t fire the bomb if gas bubbles are leaking from the bomb when it is submerged in water.

**RESULT**

Water equivalent of calorimeter \( W_E = \) gm

Calorific value of sample \( C_v = \) gm

![Bomb Calorimeter](image)
PROXIMATE ANALYSIS OF SOLID FUEL
AIM
To determine the proximate analysis and calorific value of coal and coke

PROXIMATE ANALYSIS

The proximate analysis of coal and coke 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 chemisorptions 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 vapors, nitrogen, ammonia, hydrogen sulphide etc. The pyrolysis temperature of coal may lie in the range from 600–800°C. 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°C. 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±5°C 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:**

(1) Heat a clean crucible and its lid at 900±15°C for 7 minutes in muffle furnace.
(2) Allow the crucible and lid to cool on a metal plate for a minute and in dessicator 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 900°C 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.

**OBSERVATION:**

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Amount Moisture(gm)</th>
<th>Volatile Matter (gm)</th>
<th>Ash (gm)</th>
<th>Fixed carbon (gm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coke</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**CONCLUSION:**
REDWOOD VISCOMETER - I

REDWOOD VISCOMETER I
AIM OF THE EXPERIMENT
To determine the viscosity in Redwood seconds of the given sample of oil and to plot the variation of Redwood seconds, kinematic and dynamic viscosity with temperature.

APPARATUS
- Redwood viscometer-I,
- Stopwatch,
- Thermometer (0-1100°C)
- Measuring flask. (50 c.c.)

THEORY
The viscosity of given oil is determined as the time of flow in Redwood seconds. The viscosity of a fluid indicates the resistance offered to shear under laminar condition. Dynamic viscosity of a fluid is the tangential force on unit area of either of two parallel planes at unit distance apart when the space between the plates is filled with the fluid and one of the plate’s moves relative to the other with unit velocity in its own plane. The unit of dynamic viscosity is dyne-sec/cm^2. Kinematic viscosity of a fluid is equal to the ratio of the dynamic viscosity and density of the fluid. The unit of kinematic viscosity is sec cm^2.

DESCRIPTION
Redwood viscometer-I consists of a water bath and oil bath, both provided with two thermometers inside them. There is a ball valve, which is located at center of oil bath to flow of oil through the orifice. A heater with regulator is fixed for heating purpose.

PROCEDURE
1. Clean the oil cup with a suitable solvent thoroughly and dry it using soft tissue paper.
2. Keep the ball valve in its position so as to keep the orifice closed.
3. The water is taken into the water bath and the oil whose viscosity is to be determined is taken into the oil cup up to the mark.
4. Note down the time taken in Redwood seconds for a collection of 50 cc of oil with a stopwatch at the room temperature without supply of electric supply.
5. Heat the bath and continuously stir it taking care to see that heating of the bath is done in a careful and controlled manner.
6. When the desired temperature is reached, place the cleaned 50 c.c. Flask below the orifice in position.

7. Remove the ball valve and simultaneously start a stopwatch. Note the time of collection of oil up to the 50 c.c. Mark.

8. During the collection of oil don’t stir the bath. Repeat the process at various temperatures.

**OBSERVATION:**

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Oil Temperature°C</th>
<th>Time for collecting 50c.c of oil sec</th>
<th>Kinematic viscosity $V = (A \times t) - (B/t)$ cm²/sec</th>
<th>Density($\rho$) gm/sec</th>
<th>Absolute Viscosity $\mu = v \times \rho$</th>
</tr>
</thead>
</table>

Where

$A = 0.0026\ 2cm^2/sec$

$B = 1.72\ cm^2$

**GRAPHS TO BE DRAWN**

1. Redwood secondsVs .temperature
2. Kinematic ViscosityVs .temperature
3. Absolute ViscosityVs .temperature
PRECAUTIONS
1. Stir the water continuously so that the temperature of the oil and water are equal.
2. Before collecting the oil at a temperature, check whether the oil is up to the Indicator in the oil cup.
3. Always take the readings at a stable temperature
4. Ensure proper setting of the ball valve to avoid leakage

RESULT
Variation of Redwood seconds, absolute viscosity and Kinematic viscosity with temperature, were observed and found to be decreasing with temperature.

CONCLUSION:
REDWOOD VISCOMETER - I
PENSKY MARTEN’S FLASH AND FIRE POINT TEST
PENSKY MARTEN’S FLASH AND FIRE POINT TEST

AIM OF THE EXPERIMENT
To determine the flash and fire point of the given sample of oil using Pensky Marten’s apparatus by both open and closed cup methods.

APPARATUS
1. Pensky Marten’s apparatus,
2. Thermometer (0-1100°C).

THEORY
This method determines the closed cup and open cup flash and fire points of petroleum products and mixtures to ascertain whether they give off inflammable vapours below a certain temperature.

Flash Point: It is the lowest temperatures of the oil at which application of test flame causes the vapour above the sample to ignite with a distinct flash inside the cup.

Fire point: It is the lowest temperature of the oil, at which, application of test flame causes burning for a period of about five seconds.

DESCRIPTION
The apparatus consists of a brass cup and cover fitted with shutter mechanism without shutter mechanism (open cup), test flame arrangement, hand stirrer (closed cup), thermometer socket, etc., heated with energy regulator, a thermometer socket made of copper.

PROCEDURE
1. Clean the oil cup thoroughly and fill the oil cup with the sample oil to be tested up to the mark.
2. Insert the thermometer into the oil cup through a provision, which measures the rise of oil temperature.
3. Using the Energy regulator, control the power supply given to the heater and rate of heating
4. The oil is heated slowly when temperature of oil rises, it is checked for the flash point for every one degree rise in temperature.
5. After determining the flash point, the heating shall be further continued. The temperature at which time of flame application which causes burning for a period at least 5 seconds shall be recorded as the fire point.
6. Repeat the experiment 2 or 3 times with fresh sample of the same oil
7. Take the average value of flash and fire points.

PRECAUTIONS
1. Stir the oil bath continuously to maintain the uniform temperature of sample oil.
2. The bluish halo that some time surrounds the test flame should not be confused with true flash.

**OBSERVATIONS**

<table>
<thead>
<tr>
<th>Sample oil</th>
<th>Flash Point, °C</th>
<th>Fire Point, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**RESULT**

The flash point is observed at .................. °C

The fire point is observed at ..................... °C

**CONCLUSION:**
PENSKY MARTEN’S FLASH AND FIRE POINT TEST