



**NRI INSTITUTE OF INFORMATION
SCIENCE & TECHNOLOGY BHOPAL (MP)**

Department of Basic Science

LAB MANUAL

Subject	Engg. Chemistry
Subject Code	BT-101
Scheme	CBGS
Class/Branch	I / II Semester

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EXPERIMENT NO. 1

AIM: Determination of total hardness of water by complexometric titration (EDTA method).

REAGENTS: Standard solution of EDTA, standard hard water sample, unknown hard water sample, Eriochrome Black – T indicator, Basic buffer solution of NH_4Cl and NH_4OH .

APPARATUS / GLASS WARE USED: Conical flask, Burette, Pipette, Beaker, Measuring flask etc.

THEORY:

Water which does not give readily and permanent lather with soap is called hard water. Presence of calcium and magnesium salts in the form of bicarbonate, chloride and sulphate in water makes water 'hard'. Water free from soluble salts of calcium and magnesium is called soft water. It gives lather with soap easily.

The property of water which restricts the lather formation with soap is called hardness.

Types of hardness:

- (a) Temporary hardness (b) Permanent hardness

Temporary Hardness: It is due to the presence of magnesium and calcium bicarbonates- $\text{Ca}(\text{HCO}_3)_2$ and $\text{Mg}(\text{HCO}_3)_2$.

Permanent Hardness:

It is due to the presence of soluble salts of magnesium and calcium in the form of chlorides and sulphates in water (CaCl_2 , CaSO_4 , MgCl_2 and MgSO_4). The unit used for expressing the hardness of water is parts per million (ppm).

Theory of complexometry: Eriochrome Black-T [EBT] is the indicator used in the determination of hardness by complexometric titration with EDTA. Here, Eriochrome Black-T is organic compound; sodium 1-[1-Hydroxynaphthylazo]-6-nitro-2-naphthol-4-sulfonate and EDTA is a hexadentate ligand [ethylenediaminetetraacetic acid]. When Eriochrome Black-T is added to the hard water at pH around 10, it gives wine red colored unstable complex with Ca^{2+} and Mg^{2+} ions of the sample water.



Now, when this wine red-colored solution is titrated against EDTA solution, EBT in the unstable complex is replaced by EDTA to form a stable metal-EDTA complex and liberates the free EBT. At this point, the color of the solution changes from wine-red to blue color which showing the end point of the titration.



EDTA

OBSERVATION TABLE

1. With standard water Sample:

S.No.	Volume of given water sample in ml	Burette reading		Volume of solution in ml EDTA
		Initial	Final	

2. With hard water sample-

S.No	Volume of give water sample in ml	Burette reading		Volume of solution in ml EDTA
		Initial	Final	

CALCULATIONS:

Standardization of EDTA solution. 1ml S.H.W = 1mg CaCO₃

$$V_1 \text{ ml of EDTA} = 20 \text{ ml SHW}$$

$$= 50 \text{ mg CaCO}_3$$

$$1 \text{ ml of EDTA} = 20/V_1 \text{ mg CaCO}_3$$

Total hardness

$$20 \text{ ml unknown hard water} = V_2 \text{ ml EDTA}$$

$$= V_2 \times 50/V_1 \text{ mg CaCO}_3$$

$$1 \text{ ml unknown hard water} = V_2 \times 50/V_1 \times 50 \text{ mg CaCO}_3$$

$$1000 \text{ ml unknown hard water} = V_2 \times 50/V_1 \times 1000/50 \text{ mg CaCO}_3$$

$$= V_2/V_1 \times 1000 \text{ CaCO}$$

$$\text{Total Hardness} = V_2/V_1 \times 1000$$

RESULT:

The total hardness of given water sample is ppm.

PRECAUTIONS

1. All solutions should be freshly prepared.
2. The glass wares should be rinsed with distilled water.
3. The end point should be checked carefully.

Viva questions

What is EDTA?

Ans. ethylenediaminetetraacetic acid.

Why the hardness determination titration called complexometric titration.

Ans. The hard water metal ions forms complex with EDTA.

Why NH_4Cl and NH_4OH are added during determination of hardness of water by EDTA ?

Ans. Because EDTA deprotonated at pH 10.

Why red colour is obtained when Eriochrome black T is added in hard water?

Ans. Red colour is obtained because EBT and metal ions (Ca^{2+} and Mg^{2+}) forms unstable complex in which ligand-to-ligand CT band takes place.

Why blue colour is obtained at end point.

Ans. At the end point EBT is discarded with EDTA and show blue colour.

EXPERIMENT NO. 2

AIM: To determine the sodium bicarbonate and sodium carbonate in a given alkaline mixture solution.

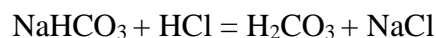
REAGENT: Standard N/20 HCl solution, alkaline mixture solution (Na_2CO_3 and NaHCO_3), phenolphthalein indicator, methyl orange indicator.

THEORY:

In double indicator method, alkali mixture solution is slowly titrated with standard HCl, using phenolphthalein indicator (pink). The end point corresponds to neutralization of Na_2CO_3 and NaHCO_3 stage



Then 2-3 drops of methyl orange indicator are added and the titration is continued till end point (pink color).



Thus, when a mixture of Na_2CO_3 and NaHCO_3 is titrated with HCl, the phenolphthalein end point (P) corresponds to neutralization of all the Na_2CO_3 to NaHCO_3 stage. While the methyl orange end point (M) corresponds to complete neutralization of Na_2CO_3 and NaHCO_3 present in the mixture solution.

$$P = \frac{1}{2} \text{Na}_2\text{CO}_3 \quad ; \quad (M) - 2(P) = \text{NaHCO}_3$$

$$M = \text{NaHCO}_3 + \text{Na}_2\text{CO}_3 \quad ; \quad 2(P) = \text{Na}_2\text{CO}_3$$

PROCEDURE :

Pipette out 20ml of given alkali mixture solution in a conical flask, add 2 drops of phenolphthalein indicator and titrated with N/20 HCl till the pink colour disappears. This is phenolphthalein end point. Now add 2 drops of methyl orange indicator to the same solution and continued the titration till yellow colour changes to pink colour. Note the total volume of HCl run down from the beginning of the experiment, as the methyl orange end point (M).

OBSERVATION:

S.No.	Vol. of alkali mixture taken	Volume of HCl runs down	
		Phenolphthalein end point (P)	Methyl orange end point (M)

(P) Average = ml

(M) Average =ml

CALCULATION :

For Phenolphthalein end point-

Water sample	HCl solution
$N_1 V_1$	$N_2 V_2$

$$N_1 = N_2 V_2 / V_1$$

$$[P] = N_1 \times \text{Equivalent Wt. of CaCO}_3 \times 1000$$

$$= N_1 \times 50 \times 1000$$

$$[P] = \text{.....mg/l or ppm}$$

For Methyl orange end point-

Water sample	HCl solution
$N_3 V_3$	$N_4 V_4$

$$N_3 = N_4 V_4 / V_3$$

$$[M] = N_3 \times \text{Equivalent Wt. of CaCO}_3 \times 1000$$

$$= N_3 \times 50 \times 1000$$

$$[M] = \text{.....mg/l or ppm}$$

Use theory table (from text book) to find the actual concentration and type of alkalinity in given water sample.

RESULTS:

Type and extent of alkalinity present in water sample mg/l

PRECAUTIONS:

1. The glass rod should be rinsed with distill water only.
2. All the solutions should be freshly prepared.

VIVA QUESTIONS

1. What is meant by alkalinity?

Ans. The increase of pH of water due to the presence of OH^- is called alkalinity.

2. What are the types of alkalinity present in water?

Ans. The radicals such as OH^- , CO_3^{2-} and HCO_3^- are responsible for the alkaline nature of water.

3. Name the indicators used in the determination of alkalinity?

Ans. Phenolphthalein and Methylorange.

EXPERIMENT NO. 3

AIM: Determination of viscosity of lubricating oil at different temperatures by Redwood Viscometer No.-1.

APPARATUS: Redwood Viscometer, Stop Watch, Lubricating Oil, 50ml flask, thermometer.

THEORY: Viscosity is a measure of the internal resistance to motion of a fluid and is mainly due to the forces of cohesion between the fluid molecules. It is one of the most important properties of lubricating oil. Viscosity is usually expressed in centipoises and usually determined with the help of Redwood Viscometer. In Redwood Viscometer, fixed volume of oil is made through to flow through a capillary tube of specified dimensions under a given set of conditions and the time of flow is measured at a particular temperature. The density of oil decreases with increase in temperature. Because of decrease in intermolecular attraction, the viscosity of the oil decreases with increase in temperature and oil takes less time to flow.

In internal combustion engines the lubricating oil used should be of such type that its viscosity varies minimum with temperature. In terms of viscosity index the oil should have high viscosity index i.e. change in viscosity must be less with temperature change.

DESCRIPTION OF THE APPARATUS: Redwood viscometer consists of the following parts:

- Oil Cup – It is a standard made by the brass and coated with silver from inside. It is 90 mm in height and 46.5 mm in diameter
- Agate Jet – The cup is fitted with an agate jet in the base the upper surface of the agate is ground to depression in which the brass ball can be placed in such a way that the channel is totally closed and no leakage of the oil from the cup through the orifice can take place.
- Orifice – It is fitted to the agate jet its diameter is 1.6 mm and capillary length is 10 mm.
- Brass Ball - It is a small ball made a brass and plated with silver it is attached to a stout wire.
- Pointer – The cup is provided with a pointer that indicates the level to which oil should be filled in the cup.
- Lid of the cup – The cup is open at the upper end and has a lid the lid is provided with an arrangement to fix a thermometer to measure oil temperature
- Water Bath – The oil cup is surrounded by a cylindrical copper vessel used to fill water which serves as a water bath and help to maintaining the desired oil temperature with the help of electric coils

It has an outlet to remove water and stirrer

- Stirrer – A stirrer with blades is provided in the water bath to maintain uniform temperature of water and oil and hence enabling uniform heating of the oil the stirrer contains a broad cured flange at the top to act as a shield for preventing any water from splashing into the oil cup
- Receiving Flask – It is calibrated flask used to receive the oil from agate discharge known as Kohlrausch flask
- Tripod Stand – The entire the apparatus rests on tripod stand provided with leveling screws at the bottom.

PROCEDURE:

Level the instrument and fill the water bath. Seal the orifice. Pour the oil in the cup up to the level. Keep 50 ml flask below the jet. Stir the oil and water and note the temperature. Raise the ball valve and start the stop watch. When the level of oil dropping reaches 50 ml, stop the watch and note the time in seconds. Replace the ball valve to seal the cup to prevent overflow of oil. Refill the oil cup to the mark. Repeat the experiments to get nearly reproducible result. Report the mean value as Redwood Viscometer at $T\text{ }^{\circ}\text{C} = t$ seconds. This is the viscosity at room temperature $T\text{ }^{\circ}\text{C}$. Repeat the experiment at five elevated temperatures say 35, 45, 55, 65 and 75 $^{\circ}\text{C}$ and note the respective times of efflux rate as described. A graph is plotted between temperature and time. The shape of the graph found is like hyperbola.



REDWOOD VISCOMETER

OBSERVATIONS:

S. No.	Volume of Oil (ml)	Temperature of oil Sample	Flow Time (in seconds)

RESULT: The graph shows that the time taken by the oil to flow through the standard orifice decreases with increase in temperature, which indicates that viscosity of the oil decreases with increases in temperature.

PRECAUTIONS:

1. Pour the oil carefully into the cup without spilling it in the water bath.
2. Take the reading when the temperature of oil and water are steady.

VIVA QUESTIONS:

1. Which flask is used to collect the flowing oil?

Ans.. Kohlarausch Flask.

2. Why viscosity of oil decreases with rise in temperature.

Ans. Viscosity of an oil decreases with increases of temperature because of decreases in intermolecular attraction due to expansion.

3. What is viscosity index?

Ans. The rate of change of viscosities with respect to temperature is known as viscosity index

$$V.I. = L - U / L - H * 100$$

5. What happens if the viscosity of oil is too low?

Ans. If the viscosity of the oil is too high excessive friction due to the shearing of oil its self would take place.

EXPERIMENT NO. 4

AIM: Determination of viscosity of lubricating oil at different temperatures by Redwood Viscometer No.- II.

APPARATUS: Redwood Viscometer, Stop Watch, Lubricating Oil, 50ml flask, thermometer.

THEORY: Viscosity is a measure of the internal resistance to motion of a fluid and is mainly due to the forces of cohesion between the fluid molecules. It is one of the most important properties of lubricating oil. Viscosity is usually expressed in centipoises and usually determined with the help of Redwood Viscometer. In Redwood Viscometer, fixed volume of oil is made through to flow through a capillary tube of specified dimensions under a given set of conditions and the time of flow is measured at a particular temperature. The density of oil decreases with increase in temperature. Because of decrease in intermolecular attraction, the viscosity of the oil decreases with increase in temperature and oil takes less time to flow.

In internal combustion engines the lubricating oil used should be of such type that its viscosity varies minimum with temperature. In terms of viscosity index the oil should have high viscosity index i.e. change in viscosity must be less with temperature change.

DESCRIPTION OF THE APPARATUS: Redwood viscometer consists of the following parts:

- Oil Cup – It is a standard made by the brass and coated with silver from inside. It has wider mouth than the cup of redwood viscometer-II.
- Agate Jet – The cup is fitted with an agate jet in the base the upper surface of the agate is ground to depression in which the brass ball can be placed in such a way that the channel is totally closed and no leakage of the oil from the cup through the orifice can take place.
- Orifice – It is fitted to the agate jet its diameter is 3.80 mm and capillary length is 50 mm.
- Brass Ball - It is a small ball made a brass and plated with silver it is attached to a stout wire.
- Pointer – The cup is provided with a pointer that indicates the level to which oil should be filled in the cup.
- Lid of the cup – The cup is open at the upper end and has a lid the lid is provided with a arrangement to fix a thermometer to measure oil temperature.
 - Water Bath – The oil cup is surrounded by a cylindrical copper vessel used to fill water which serves as a water bath and help to maintaining the desired oil temperature with the help of electric coils. It has an outlet to remove water and stirrer.
 - Stirrer – A stirrer with blades is provided in the water bath to maintain uniform temperature of water and oil and hence enabling uniform heating of the oil the stirrer

contains a broad cured flange at the top to act as a shield for preventing any water from splashing into the oil cup.

- Receiving Flask – It is calibrated flask used to receive the oil from agate discharge known as Kohlrausch flask.
- Tripod Stand – The entire the apparatus rests on tripod stand provided with leveling screws at the bottom.

PROCEDURE: Level the instrument and fill the water bath. Seal the orifice. Pour the oil in the cup up to the level. Keep 50 ml flask below the jet. Stir the oil and water and note the temperature. Raise the ball valve and start the stop watch. When the level of oil dropping reaches 50ml, stop the watch and note the time in seconds. Replace the ball valve to seal the cup to prevent overflow of oil. Refill the oil cup to the mark. Repeat the experiments to get nearly reproducible result. Report the mean value as Redwood Viscometer at $T\text{ }^{\circ}\text{C} = t$ seconds. This is the viscosity at room temperature $T\text{ }^{\circ}\text{C}$. Repeat the experiment at five elevated temperatures say 35, 45, 55, 65 and 75 $^{\circ}\text{C}$ and note the respective times of efflux as described. A graph is plotted between temperature and time. The shape of the graph found is like hyperbola.

OBSERVATIONS:

S. No.	Volume of oil (ml)	Temperature of oil sample	Flow time (in seconds)

RESULT: The graph shows that the time taken by the oil to flow through the standard orifice decreases with increase in temperature, which indicates that viscosity of the oil decreases with increases in temperature.

PRECAUTIONS:

1. Pour the oil carefully into the cup without spilling it in the water bath.
2. Take the reading when the temperature of oil and water are steady.
3. Note the time and temperature carefully.
4. Operate the apparatus carefully.

VIVA QUESTIONS:

1. What are the uses of low viscosity oil?

Ans. Low viscosity oil is used in bearings for high speed equipment such as turbines spindles and centrifuges.

2. What are the uses of high viscosity oils?

Ans. High viscosity oils are used in bearings of low speed equipment

3. What is the diameter of the jet and length of the jet?

Ans. The diameter of the jet is 3.80 mm and length of the jet is 50 mm.

4. What is the viscosity value for water?

Ans. Viscosity of water at 20 °C is about 1centipoises.

5. What is absolute viscosity?

Ans. Absolute viscosity may be defined as the tangential force per unit area which is required to maintain a unit velocity gradient between two parallel layers it is denoted by symbol η .

EXPERIMENT NO. - 5

AIM: Determination of Flash and Fire Point of the given lubricating oil by Pensky – Martins closed cup apparatus.

APPARATUS: Pensky – Martins Closed Cup Apparatus, oil sample, thermometer etc.

THEORY: The flash point of oil is the lowest temperature at which the oil gives off sufficient vapors to ignite momentarily when a flame of standard dimension is brought near the surface of the oil. Fire point of oil is the minimum temperature at which the vapors of the oil burns continuously for at least 5 seconds when the flame is brought near the oil surface. In majority of case the fire point is about 5 to 40 °C higher than its flash point. The lubricating oil selected should have a flash point which is reasonably above its working temperature. This ensures safety against fire hazards during the storage, transport and use of the lubricating oil.

DESCRIPTION:

1. Oil Cup – It is about 5 cm is diameter and 5.5 cm deep, the level to which oil is to be filled is marked inside the Cup.
2. The lid of the cup – The cup lid is provided with four openings of standard size to insert thermometer, stirrer and sliding shutter.
3. Sliding Shutter – It is a lever mechanism, provided at the top of the cup. By moving the shutter, lid opens & flame is dipped into this opening to bring the flame over the oil surface.
4. Air Bath – Oil cup supported by an air bath which is heated by an electrical coil.
5. Flames Exposure Device – It is a tiny flame cemented to the shutter by a lever mechanism.
6. Plot Burner – As the flame is introduced in the opening, it gets extinguished but when the test flame is returned to its original position it is automatically lightened by the fire burn.

PROCEDURE: The oil sample is taken into the oil cup up to the specified mark and placed inside the apparatus. After ensuring that oil cup, thermometer and heater are placed correctly, heating is started. Observations taken by bringing tiny flame near the opening of mouth of cup. Flash and fire are reported. Once getting fire, immediately switched off the heater and observation recorded in decreasing temperature.



PENSKY MARTIN'S APPARATUS

OBSERVATIONS

Table 1: Flash and Fire point during heating-

S.No.	Temperature	Observation	Inference

Table 2: Flash and Fire point during cooling-

S.No.	Temperature	Observation	Inference

RESULT

Flash point of the given lubricating oil is.....

Fire point of the given lubricating oil is.....

PRECAUTIONS:

1. Breathing over the surface of the oil should be avoided.
2. Heating device must be switched off after getting fire point.
3. The bulb of the thermometer should dip inside the oil.

VIVA QUESTIONS:

1. What type of oil is used in Pensky Marten's Apparatus?

Ans. Lubricating thick oil.

2. What significance is of flash and fire point?

Ans. Lubricating oil selected for a job should have a flash point which is reasonably above its working temperature this insures safety against fire hazards during the storage transport and use of the lubricating oil. .

3. How many openings are there in lid of Pensky Martens Apparatus?

Ans. Four openings are there in the lid.

4. Which type of Jacket is presenting of Pensky Martens apparatus?

Ans. Air Jacket.

5. Name any two solid lubricants.

Ans. Graphite (C) and molybdenum disulphite (MoS_2).

EXPERIMENT NO. – 6

AIM: Determination of Flash and Fire Point of the given lubricating oil by Abel's closed cup apparatus.

APPARATUS: Abel's Closed Cup Apparatus, given oil sample, thermometer etc.

THEORY:

Flash Point- The flash point of oil is the lowest temperature at which the oil gives off sufficient vapors to ignite momentarily when a flame of standard dimension is brought near the surface of the oil.

Fire Point- Fire point of oil is the minimum temperature at which the vapors of the oil burns continuously for at least 5 seconds when the flame is brought near the oil surface.

The lubricating oil selected should have a flash point which is reasonably above its working temperature and the difference between flash and fire point should be more than 20-25 °C for good lubricating oil. This ensures safety against fire hazards during the storage, transport and use of the lubricating oil.

DESCRIPTION: Abel's apparatus consists of following parts:

1. Oil cup - It is a standard cylindrical cup made of brass.
2. Pointer - The oil cup is provided with a pointer to indicate the level up to which oil should be filled in the cup.
3. Water Bath - The oil cup is surrounded by a double jacketed cylindrical copper vessel containing water which serves as a water bath for maintaining the desired oil temperature with the help of electric heating coils.
4. Air Bath - The space between the water bath and the oil cup form an air bath by mean of which the oil can be heated uniformly.
5. Tripod Stand -The entire apparatus stand on a tripod stand

PROCEDURE:

The oil cup is filled with oil which is to be tested up to the point and it is covered with lid. Now oil cup is placed into the apparatus, the paddle stirrer and the standard thermometer, assembled with its bulb dipping into the oil at their respective places provided in the apparatus. The water bath filled with water and is heated with electrical coil. The rate of heating adjusted in such a way that the temperature of the oil increases 1 to 1.5 °C per minute. At every degree rise of temperature the sliding shutter is opened and the test flame is introduced over the oil surface through the central opening to see whether the oil gives a flash/fire. The minimum temperature at

which a distinct flash/fire appears are recorded. Procedure is repeated in cooling of the oil and results by comparing both observations.



ABEL'S APPARATUS

OBSERVATIONS

Table 1: Flash and Fire point during heating-

S.No.	Temperature (°C)	Observation	Inference

Table 2: Flash and Fire point during cooling-

S.No.	Temperature (°C)	Observation	Inference

RESULT

Flash point of the given lubricating oil is.....

Fire point of the given lubricating oil is.....

PRECAUTIONS:

1. Breathing over the surface of the oil should be avoided.
2. Heating device must be switched off after getting fire point.
3. The bulb of the thermometer should dip inside the oil.

VIVA QUESTIONS:

2. What is lubrication?

Ans. Lubrication is the process, or technique employed to reduce wear of one or both surfaces in close proximity, and moving relative to each other, by interposing a substance called lubricant between the surfaces to carry or to help carry the load (pressure generated) between the opposing surfaces.

3. Which oil is filled in the oil cup of Abel's apparatus?

Ans. Mineral oil

4. Define Flash and Fire point.

Ans. The flash point of oil is minimum temperature at which oil gives of sufficient vapors to ignite momentarily when a flame of standard dimensions is brought near the surface of the

oil and the fire point of an oil is the lowest the temperature at which the vapors of the oil burns continuously for at least 5 seconds when the standard flame is brought near the surface of the oil.

5. What is difference between Abel's apparatus and Pensky Martin apparatus?

Ans. Abel's Flash Point Apparatus: This determines the closed cup flash points and fire point of petroleum products and mixtures to ascertain whether they give off inflammable vapor below 100 °C.

Pensky Martens Apparatus: This is widely used for determination of Flash Point and Fire point of oil, cut back asphalts, other viscous material and suspension of solids having a flash point and fire point above 150 °C.

6. How many openings present in Abel's apparatus?

Ans. In Abel's apparatus three openings are present.

7. Which type of jacket present in Abel's apparatus?

Ans. Water jacket.

EXPERIMENT NO. – 7

AIM - To determine the Flash point and Fire point of given oil by using Cleveland Open Cup apparatus.

APPARATUS - Cleveland open-cup apparatus, oil sample, thermometer etc.

THEORY: The flash point of oil is the lowest temperature at which the oil gives off sufficient vapors to ignite momentarily when a flame of standard dimension is brought near the surface of the oil. Fire point of oil is the minimum temperature at which the vapors of the oil burns continuously for at least 5 seconds when the flame is brought near the oil surface. In majority of case the fire point is about 5 to 40 °C higher than its flash point. The lubricating oil selected should have a flash point which is reasonably above its working temperature. This ensures safety against fire hazards during the storage, transport and use of the lubricating oil.

DESCRIPTION OF THE APPARATUS -

Oil cup – The apparatus consists of a brass cup with a mark showing the level up to which the level up to which the oil should be filled.

Base of the cup – The cup is supported by a metal place which has a circular opening at the center, of enough size to fit the cup in position.

Heating Device – An electrical heating device is provided in such a way that the cup is uniformly in such a way that the cup is uniformly heated.

Clamps – A clamp is provided to hold the thermometer.

Thermometer – Thermometer of range 0 – 300 °C used with the apparatus.

Procedure – The cup is filled with oil under test in such a way that the oil level is exactly up to the mark. The thermometer is held vertically by means of the clamp such that the bottom of the bulb is not touch the bottom of the cup or sides of the cup. Heating is started by electrical device and the rise in temperature observed with the help of thermometer. At every degree, rise in temperature observed with the help of thermometer. At every degree, rise in temperature the test flame is brought near the mouth of the opening of the oil cup and observed whether a flash / fire appear on oil surface. The minimum temperature at which a distinct flash / fire appears on the surface of the oil in the cup is recorded as the flash / fire of the oil under test. Procedure is repeated by cooling the oil and results are reported by comparing both observation tables.



Cleveland's Apparatus

OBSERVATIONS – Table 1: Flash and Fire point during heating-

S.No.	Temperature	Observation	Inference

Table 2: Flash and Fire point during cooling-

S.No.	Temperature	Observation	Inference

RESULT – Flash point of the given lubricating oil is

Fire point of the given lubricating oil is

PRECAUTIONS –

1. Heating's device must be switched off after observe the fire point.
2. Breathing over the surface of the oil should be avoided.

VIVA QUESTIONS:

1. Which type of Jacket is presenting of Cleveland apparatus.

Ans. Open Cup.

2. In which oil oiliness is least?

Ans. Mineral oil.

3. Which class of oil posses higher oiliness?

Ans. Vegetable oil and Animal oil.

4. What is oiliness?

Ans. Oiliness of a lubricant is a measure of its capacity to stick on to the surface of machine parts under conditions of heavy pressure or load.

5. Name different types of a lubricant

Ans. (a). Lubricating oil (b) Greases or semi solid lubricant

(c) Solid Lubricant (d) Synthetic lubricants

EXPERIMENT NO. - 8

AIM: To determine moisture, volatile matter in a given coal sample by Proximate Analysis.

APPARATUS: Coal sample, Analytical balance, Electric Oven, Desiccators, Silica crucible, Tongs, Muffle Furnace.

THEORY: Varying amount of moisture is associated with different coal samples. Higher percentage of moisture is undesirable as it decreases calorific value of coal sample. Moisture increases the cost of coal.

Volatile matter consists of a mixture of gases and liquid. Volatile matter in coal may be in the form of combustible gases (CH_4 , H_2 , CO and others) or non-combustible gases like CO_2 and N_2) presence of non-combustible gases is undesirable.

PROCEDURE: The clean crucible is weighed. Approx 1gm of air dried coal sample is taken in it. The crucible is weighed again with coal before heating. It is heated in oven at 110°C for 1 hour. Crucible is taken out from oven and allowed to cool in desiccators. The process of heating and cooling repeated till the weight of crucible with coal become constant.

Above left coal is taken and weighed in silica crucible. Keep the crucible with lid in a muffle furnace at $925^\circ\text{C} \pm 15^\circ\text{C}$ and close the door. Heat for 7 min. and take out and cool in a desiccators and weight loss in amount of volatile matter and report in percentage of volatile matter.

OBSERVATION:

For Moisture:

Weight of the crucible = W gm

Weight of crucible + coal =W1 gm

(Before heating)

Weight of coal taken= $W1 - W = \dots\dots\dots$ gm

Weight of crucible + coal =W2 gm

(After heating)

$$\text{Loss in weight of coal} = W1 - W2$$

For volatile matter:

$$\text{Weight of the crucible} = \dots\dots\dots W3\text{gm}$$

$$\text{Weight of crucible + coal} = \dots\dots\dots W4 \text{ gm}$$

(Before heating)

$$\text{Weight of coal taken} = W4 - W3 = \dots\dots\dots \text{ gm}$$

$$\text{Weight of crucible + coal} = \dots\dots\dots W5 \text{ gm}$$

(After heating)

$$\text{Loss in weight of coal} = W4 - W5$$

CALCULATIONS:

Percentage of Moisture

$$\begin{aligned} &= \frac{\text{Loss in weight of coal} * 100}{\text{Weight of coal sample taken}} \\ &= \frac{(W1 - W2) * 100}{(W1 - W)} \end{aligned}$$

Percentage of Volatile Matter

$$\begin{aligned} &= \frac{\text{Loss in weight of coal} * 100}{\text{Weight of coal sample taken}} \\ &= \frac{(W4 - W5) * 100}{(W4 - W3)} \end{aligned}$$

PRECAUTIONS:

1. Weighing should be done carefully

2. Crucible should be properly cleaned.
3. Lid of the desiccators should be air – tight.

VIVA QUESTIONS:

What is proximate analysis?

Ans. The "proximate" analysis gives moisture content, volatile content, consisting of gases and vapors driven off during pyrolysis (when heated to 950 °C), the fixed carbon and the ash, the inorganic residue remaining after combustion in the sample and the high heating value based on the complete combustion of the sample to carbon dioxide and liquid water. Proximate analysis is the most often used analysis for characterizing coals in connection with their utilization.

What is the method of coal analysis?

Ans. Its methods are- 1. Proximate analysis 2. Ultimate analysis.

What is the significance of moisture content in the analysis of coal?

Ans. The significance of moisture content in coal because moisture content gives the valuable information about water molecules associate with coal which decreases the calorific value.

What is the purpose to determine the percentage of volatile matter in the coal?

Ans. The purpose to determine the volatile matter in coal is that the volatile matter decreases the calorific value of the coal.

What are permissible limits of moisture content for a good quality of coal?

Ans. 0.5 to 10%

What is the disadvantage of high percentage of moisture in coal?

Ans. The disadvantage of high percentage of moisture in coal is it increases heat loss due to evaporation and superheating of vapor.

which is the most widely used

solid fuel? Ans. Wood