

EXPERIMENT - 01AIM :

To determine the effect of Temperature on the viscosity of the given lubricant using Redwood Viscometer &.

Requirements :

Redwood viscometer, Thermometer, Given sample of oil (lubricant), Stopwatch, 50ml standard narrow necked flask.

Theory :

The viscosity of a lubricating oil is defined as the relative fluidity or the force with which it passes through a capillary of standard size, overcoming the resistance due to its backward force during its flow. This force is expressed in terms of pressure per unit area and its unit is dyne/cm². In practical applications, the viscosity is measured in terms of time in seconds for the flow of the oil through a capillary. Mathematically the coefficient of viscosity (η) is represented by following equation :

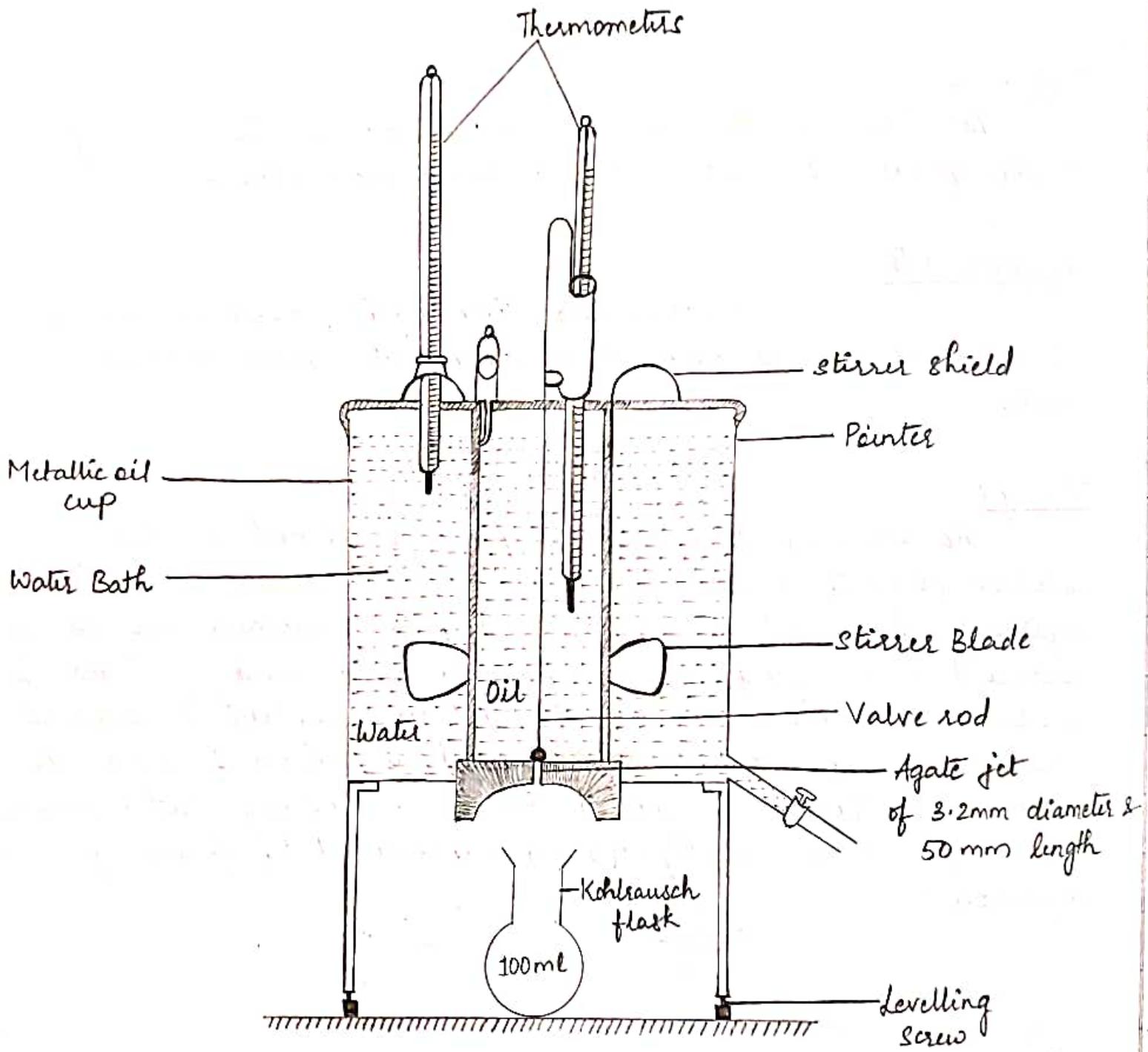
$$\eta = \frac{F \cdot d}{v}$$

where F = force per unit area

d = distance between two layers

v = velocity of flow of the liquid

Teacher's Signature _____



REDWOOD VISCOMETER - 2

In General, the viscosity of an oil is not measured in absolute units, instead it is measured in terms of time of its flow through an orifice of standard size at the prevailing temperature.

Viscosity is measured through an apparatus known as viscometer. There are three types of viscometers namely :- Saybolt's viscometer, Engler's viscometer and Redwood viscometer, which is being the most suitable viscometer in regard to India's weather conditions.

Viscosity of oil decreases invariably with the rise in temperature. This determination of viscosity at varying temperatures is necessary to provide efficient lubrication throughout the range of temperatures in which the machine operates.

Viscosity Index, the rate at which the oil changes its viscosity with temperature. It is measured in terms of relative viscosity index of different oils. A Good lubricant is the one which varies less with temperature, having low viscosity Index because it is able to provide efficient lubrication at variable temperature.

Description of Apparatus :

- 1) Oil cup : Oil cup of a standard size having a mark upto which oil has to be filled. The bottom of the oil cup is fitted with a jet made up of 'agate' and with the measurements of bore as 3.2mm diameter and 50mm length. The jet can be opened or closed by valve rod fitted with a brass ball. To record the

OBSERVATION TABLE

S.No.	Temperature ($^{\circ}\text{C}$)	Volume of oil collected (ml)	Time in Seconds (s)
1.	23	100	327
2.	30	100	203.
3.	36	100	152
4.	40	100	114

temperature of the oil, arrangement to fix the thermometer is provided.

2) Heating bath : The oil cup is surrounded by a cylindrical bath for containing the water. It is provided with an outlet to drain the water. A thermometer holder is provided to record the temperature of the water. A stirrer is provided in the water bath for maintaining uniformity of required temperature.

3) Receiver : A special type of receiver called Kohlrausch flask to collect 100 ml of the oil.

4) Spirit level : A spirit level used for levelling the apparatus vertically is also provided in the lid of the cup. The entire apparatus is supported on three legs, provided at the bottom with levelling screws.

Procedure :

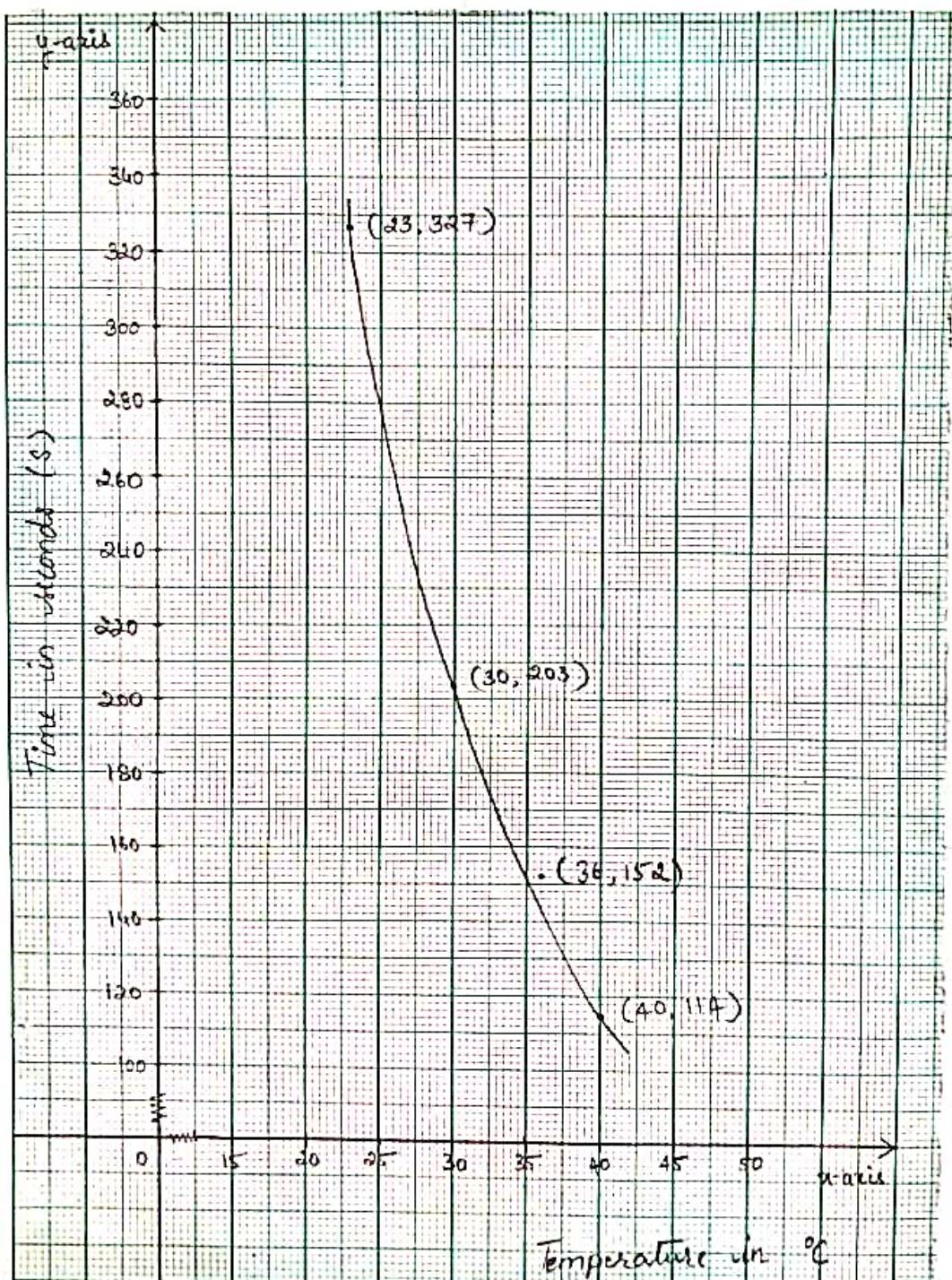
- 1) The given oil is filled upto the given mark in the oil cup.
- 2) Allow the oil to flow out through the orifice.
- 3) 100 ml of oil is collected through the orifice in a standard receiver at room temperature and the time of collection is noted down, with the help of a stop watch.
- 4) Same oil is filled again and ~~temperature of water bath is raised by about 5-6 °C and temperature of the oil is recorded. Maintain the uniformity in temperature of both oil & water, so that temperature of oil remains constant during the period of flow of oil.~~

TEMPERATURE VS TIME GRAPH

To plot the graph, temperature of the oil is taken on y-axis in $^{\circ}\text{C}$ and time is taken on x-axis in seconds (s).

Scale :- on x-axis : 1 unit = 5°C (temperature)

on y-axis : 1 unit = 20 seconds (time)



- 5) Again, 100ml of oil is collected in the receiver using same procedure and the time of collection is noted.
- 6) Repeat the same procedure by varying the temperature by 5-6 °C and the time of collection is noted, while ensuring that the time difference is not too much.
- 7) Plot the Temperature vs Time graph according to the observations noted and study the nature of the curve by which the Viscosity Index of an oil at a particular temperature can be calculated.

Result:

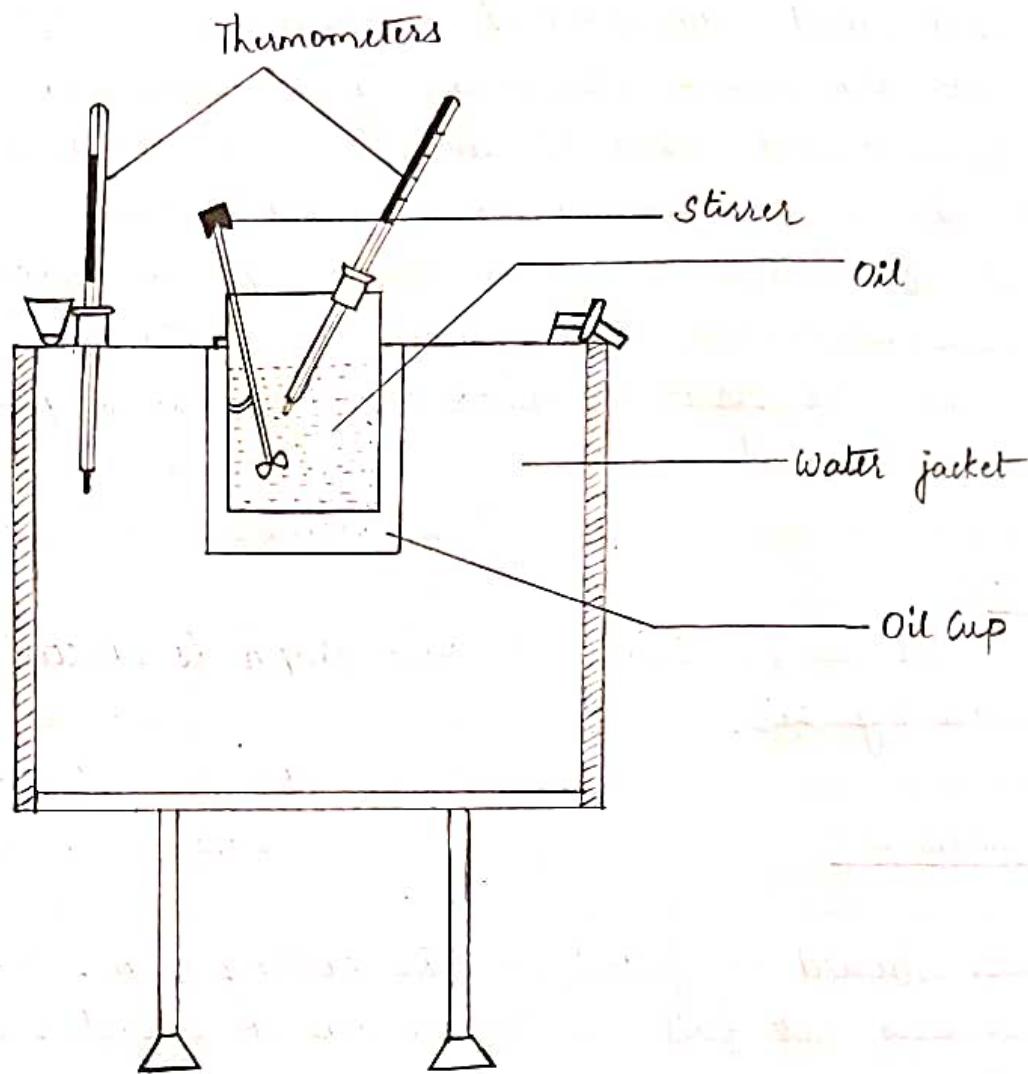
The Temperature vs time graph is plotted for the given lubricating oil.

Precautions:

- 1) Water should be filled in the heating bath, taking care that water does not fall in the oil cup to avoid creation of an emulsion.
- 2) The temperature of water and oil should be almost at same value, which can be ensured by careful stirring.
- 3) The oil flowing from the oil cup should fall clear in the receiver and should not slide down through the walls of the receiver.

Result:

Graph shows that by increasing temperature, time of collection of oil decreased, which shows fall of viscosity with increase in temperature.



ABEL'S CLOSED CUP APPARATUS

EXPERIMENT - 02Aim:

To determine the flash point and fire point of a given lubricating oil by Abel's Apparatus.

Requirements:

Abel's closed cup, combustion liquid or oil, thermometer, measuring flask, spirit lamp, small wooden sticks.

Theory:

Flash point and Fire point are two of the important characteristics of a lubricating oil though they do not effect the lubricating properties of an oil, but are required in cases where the oil is exposed to high temperature, then the oil used should have higher flash and fire points than the temperature at which it is likely to be used.

Flash point: It is the lowest temperature at which a lubricating oil produces enough vapours, which on combining with atmospheric oxygen form an explosive mixture of such a nature that when a flame comes in contact with it, the mixture ignites for a moment giving a momentary flash. (like the flash of a photographic camera).

Fire point: It is the lowest temperature at which a lubricating oil produces enough vapours, which on combining with atmospheric oxygen form an explosive mixture of such a nature that when a

flame comes in contact with it, the mixture burns for few seconds continuously depending upon the volatility of the oil.

Generally, the fire point of a lubricating oil is about 5° to 20°C higher than its flash point depending upon the nature of the oil. Flash and fire points are determined with the help of the following flash and fire point apparatus:

- Abul's apparatus
- Pensky-Martin's apparatus
- Cleveland's apparatus

In the study of the properties of lubricating oils, the flash point and fire point are to be determined when the oil is likely to be heated during its use, without causing burn outs and fire accidents. This is also necessary for safety reasons in many applications, such as the transformer oils and the mobile used for heavy duty switch gears and heat engines. The information of flash and fire points give an idea of the risk of fire accidents, involving the apparatus and the life of attendants both. Thus the safety limits of the oils have been standardized during their uses and applications.

Description of Apparatus:

- Oil cup: oil cup of standard size having a mark upto which the oil has to be filled.
- Lid of the oil cup: It consists of four parts or openings, one used for inserting the thermometer, other for carrying two copper blades for stirring, third one for the entry of the air and the fourth is a shutter (sliding slit) to introduce the test flame.

Observation table while heating

S.NO.	Temperature (°C)	Observation
1	40	No observation
2	42	Flash observed
3	43	No observation
4	44	Flash observed
5	45	No observation
6	46	Flash observed
7	48	Flash observed
8	50	Flash observed
9	53	Flash observed
10	55	Flash observed
11	57	Flash observed
12	58	Fire observed

3) Water Jacket :

The oil cup is surrounded by a cylindrical water jacket in which the hot water is filled to heat the oil, which is volatile and inflammable and cannot be heated directly.

4) Thermometer : It is used in the measurement of the temperature of given liquid.

Procedure :

- 1) The combustion liquid is poured upto the marked level in the cup. The lid is covered and the thermometer is inserted.
- 2) Close the sliding shutter after the water bath is filled. Make sure to drain a small portion of water after filling the water bath completely to allow free heating of water.
- 3) Start heating the water bath.
- 4) After the oil is heated upto 40°C , introduce the test flame above the oil, as it starts to produce vapours.
- 5) Check for spark in the liquid through the liquid opening slide after every 1°C increase in temperature.
- 6) The temperature of oil at which the spark is noticed is noted as the flash point. Here, the spark is less than 5 seconds.
- 7) The temperature at which the spark comes out and remains for more than 5 seconds is known as fire point.
- 8) After observation of fire, turn off the heat supply and remove the oil cup from the heat bath and place it aside for cooling the liquid.

Observation table while cooling

S.No.	Temperature (°C)	Observation
1	57	Flash observed
2	56	Flash observed
3	55	Flash observed
4	54	No observation
5	53	Flash observed
6	51	Flash observed
7	49	No observation
8	48	Flash observed
9	47	No observation
10	46	Flash observed
11	45	No observation
12	44	Flash observed
13	43	Flash observed
14	42	Flash observed
15	41	Flash observed.
16	40	No observation

- 9) Repeat the same experiment for the decrease in temperature by 1°C after the heat is stopped and cooling has begun.
- 10) The observations of flash point and fire point are tabulated.

Result:

The flash point of the given lubricating oil by use of Abel's apparatus is found to be 41°C and the fire point is found to be 58°C .

Precautions:

- 1) The lid of the apparatus must be opened only when the flame is being introduced above it.
- 2) The thermometer kept inside the sample cup should not touch the metallic cup.
- 3) The stirrer should not be hitting the thermometer.

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EXPERIMENT-03Aim:

To determine the flash point and fire point of the given oil by Pensky-Martin's closed cup apparatus.

Requirements:

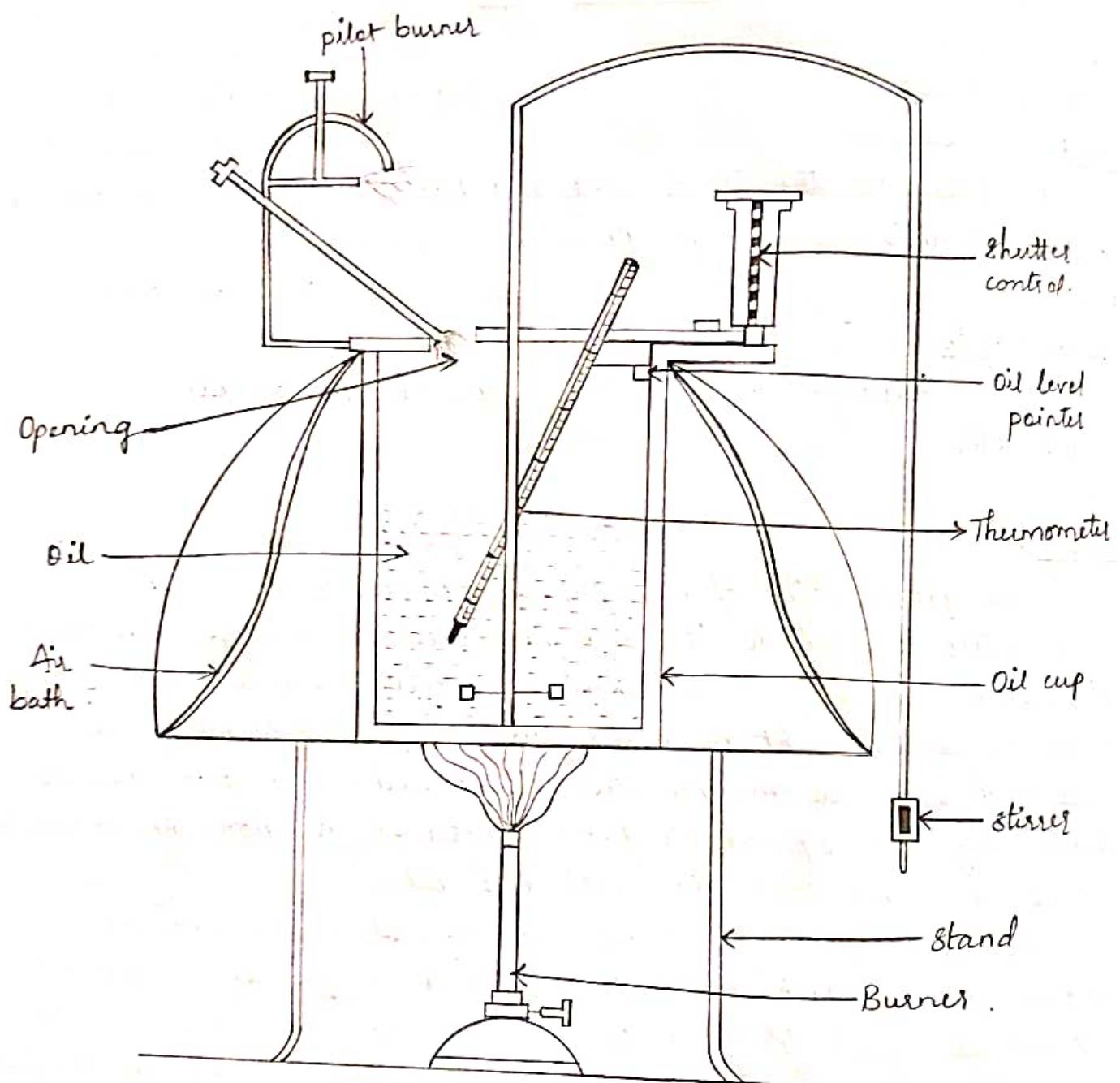
Pensky-Martin's closed cup, combustion oil, Thermometer

Theory:

The flash point of oil may be defined as the minimum temperature at which it must be heated to give off sufficient vapours to ignite for more than 5 seconds when a flame of standard dimensions is brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions. This is detected by the appearance of momentary flash upon the application of small flame over the surface of oil.

The fire point of oil may be defined as the maximum temperature to which it must be heated to give off sufficient vapours to ignite for more than 5 seconds when a flame of standard temperature and dimensions is brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions.

PENSKY - MARTIN'S FLASH POINT APPARATUS.



Pensky - Martin Closed Cup Apparatus

Description of Apparatus:

A Pensky-Martin apparatus consists of the following parts:-

- 1) Sample cup: It is a cylindrical vessel, made up of brass with a filling mark grooved inside near the top. It is provided with a lid.
- 2) Lid: The lid is equipped with the following parts
 - (i) Stirrer: The stirrer consists of a vertical steel shaft mounted in the centre of the cup and carrying two-bladed brass propellers.
 - (ii) Cover: It has four openings, one for thermometer and the rest for oxygen entry & exposure of vapours to the test flame.
 - (iii) Shutter: The lid is equipped with a brass shutter operating on the plane of the upper surface of the cover. The shutter is so shaped and mounted on the lid that when in one position, the holes are completely closed and when in the other, these orifices are completely opened.
 - (iv) The flame exposure device: The lid is equipped with a pilot lamp with such a mechanism that its flame operates simultaneously with the shutter. When the shutter is in the open position, the tip is lowered down in the centre of the central orifice.
- 3) Heater: The cup is heated by the means of the burner or it is electrically heated. The air bath has cylindrical interior about 4cm deep and can be heated by a direct flame or an electrical resistance element. The top plate is made up of metal and mounted with an air gap between it and air bath.

Observation table while heating.

S.No.	Temperature (°C)	Observation
1.	100	No observation
2.	102	No observation
3.	104	Flash observed
4.	106	Flash observed
5.	108	No observation
6.	110	No observation
7.	112	Flash observed
8.	114	No observation
9.	118	Flash observed
10.	120	Flash observed
11.	124	Flash observed
12.	128	No observation
13.	130	No observation
14.	134	No observation
15.	138	Flash observed
16.	140	Flash observed
17.	144	Flash observed.
18.	146	Flash observed
19.	150	Flash observed.
20.	154	No observation
21.	158	No observation
22.	164	Flash observed
23.	168	Flash observed
24.	172	Flash observed.
25.	178	No observation
26.	188	Fire observed

Procedure :

- 1) The cup and its accessories are well cleaned before the test is started.
- 2) Now the cup is filled with the oil to be tested up to the level indicated by the filling mark and covered with the lid.
- 3) The stirring device, thermometer and flame exposure device is fixed on the top of the cover.
- 4) The cup is now set in the apparatus properly and the thermometer is inserted.
- 5) The test flame is lighted and adjusted until it is the size of a bead.
- 6) The apparatus is heated so that the heating rate is maintained, with the help of a rheostat, at $5-6^{\circ}\text{C}$ per minute and stirring rate at 1-2 stirs per second.
- 7) Once the heating starts, the test flame is applied after each 2°C rise of temperature nearer to the sample in the cup by opening the shutter and check the appearance of flash.
- 8) On observing a fire, stop the heating process and allow the temperature to decrease, check the occurrence of flash at every 1°C drop in temperature.
- 9) Record the lowest temperature at which the flash is observed as flash point of the sample & the lowest temperature at lowest temperature at which the fire is observed as the fire point of the sample.
- 10) Repeat the experiment and record the temperatures at which the flash is observed.

Observation table while cooling

S.No.	Temperature (°C)	Observation
1.	184	Fire observed
2.	180	Flash observed
3.	178	Flash observed
4.	172	No observation
5.	170	Flash observed
6.	162	Flash observed
7.	158	Flash observed
8.	152	Flash observed
9.	146	Flash observed
10.	142	No observation
11.	138	Flash observed
12.	134	No observation
13.	130	Flash observed
14.	126	Flash observed
15	122	No observation
16.	120	Flash observed
17.	116	No observation.
18.	112	Flash observed
19.	110	Flash observed
20.	108	No observation
21.	106	No observation
22.	102	No observation
23.	100	No observation

Result:

The flash point and fire point of the given oil sample determined by Pensky-Martin's closed cup apparatus is found to be 108°C and 184°C .

Precautions:

- 1) Do not stir the sample while applying test.
- 2) Do not test the sample after fire point is determined.
- 3) Thermometer must not touch the bottom of the metal cup.

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EXPERIMENT - 04

Aim:

To check the flash point and fire point of given lubricating oil by using Cleveland open cup apparatus.

Requirements:

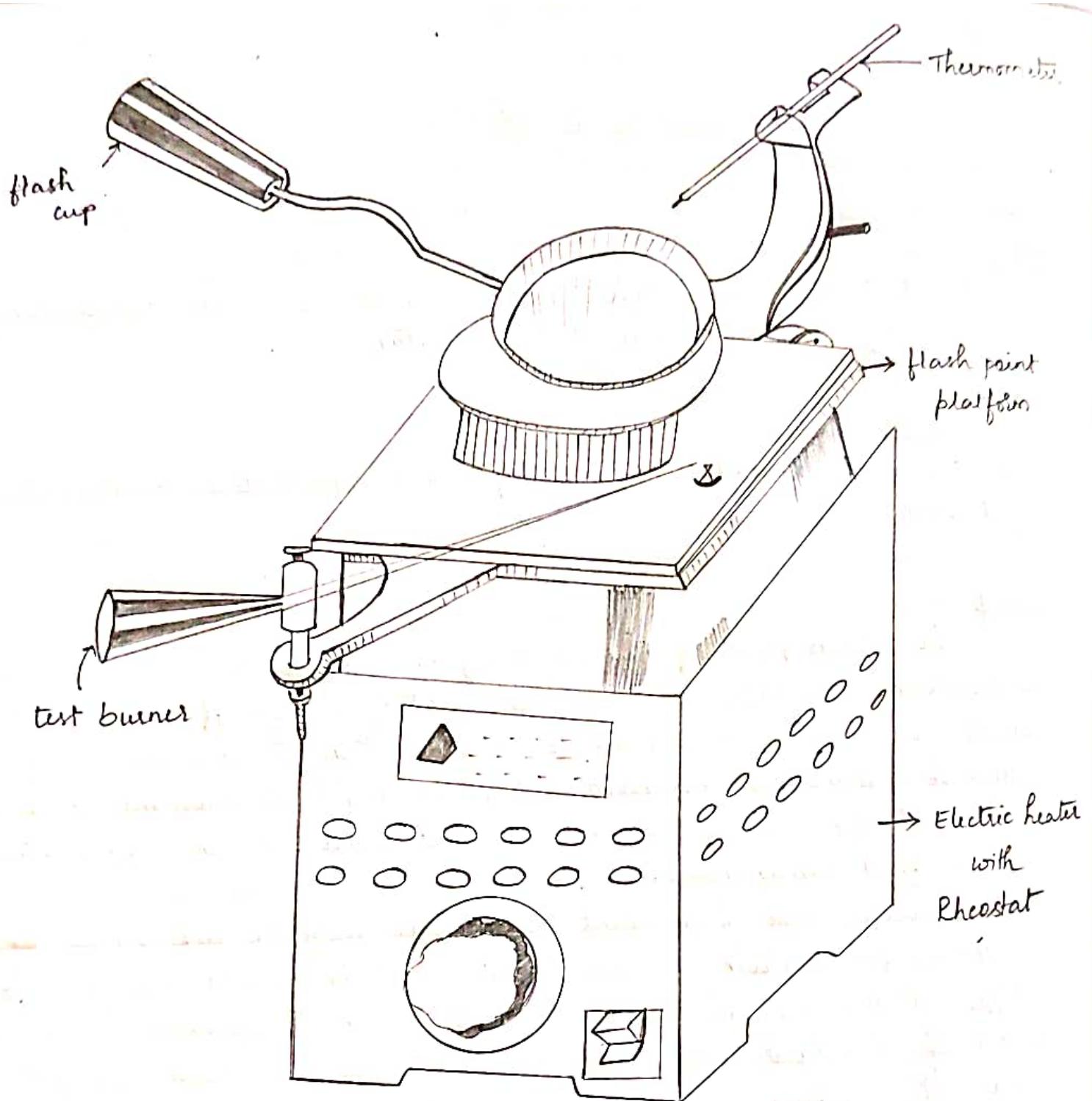
Oil sample, Cleveland open cup apparatus, Thermometer, spirit lamp.

Theory:

The flash point of an oil may be defined as the minimum temperature to which it must be heated to give off sufficient vapour to ignite momentarily or for less than 5 seconds when a flame of standard dimensions (approx 4mm) is brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions.

Whereas, the fire point of all oils may be defined as the minimum temperature to which it must be heated to give off sufficient vapours to ignite for more than 5 seconds when a flame of standard dimensions (approx 4mm) is about brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions.

Flash point is detected by the appearance of momentary flash upon the application of small flame over the surface of oil. Its detection is important as it helps in detection and safety against the fire hazards during storage, transport and use.



Cleveland's Open Cup Apparatus

Description of Apparatus:

The cleveland open cup apparatus consists of :

- 1) Test Cup : made without any lid and is equipped with a handle. The cup is supported by a metal plate known as heating plate. The cup may be heated by an electric heater mounted below the cup in the apparatus itself.
- 2) Metal Plate : has an extension for mounting the testing flame and thermometer support. The test flame is mounted in such a manner as to permit automatic duplication of the sweep of the test flame over the sample cup. The size of flame can be adjusted with respect to the dimension of metal bead (4mm).

Procedure:

- 1) The apparatus is thoroughly cleaned and the thermometer is suspended in such a way that the bottom of the thermometer bulb just stays above the bottom of Sample cup.
- 2) The cup is now filled with lubricating oil to the filling mark grooved on the inner side of the cup taking care that the surface of the sample is free from bubbles and there is no oil above the filling mark.
- 3) Now move the test flame over the sample cup and check the appearance of flash over the sample inside the cup.
- 4) If no flash is observed, increase the temperature of the sample taken and take a flame over the cup after every 2°C increases. On observing a flash, we need to check the fire point after reaching the fire point, stop the heating process and allow the temperature to decrease.

OBSERVATION TABLES.

Observation Table while heating.

S.No	Temperature (°C)	Observation	S.No	Temperature (°C)	Observation
1.	100	No observation	14.	190	Flash observed
2.	110	No observation	15.	210	Flash observed
3.	118	No observation	16.	220	No observation
4.	120	Flash observed	17.	228	Flash observed
5.	124	Flash observed	18.	238	No observation
6.	130	Flash observed	19.	242	No observation
7.	136	No observation	20.	254.	Fire observed.
8.	140	Flash observed			
9.	144	Flash observed			
10.	150	No observation			
11.	164	Flash observed			
12.	170	Flash observed			
13.	178	No observation			

Observation Table while cooling.

S.No	Temperature (°C)	Observation	S.No	Temperature (°C)	Observation
1.	240	Fire observed	12.	152	Flash observed
2.	230	No observation	13.	164	Flash observed
3.	224	Fire observed	14.	140	Flash observed
4.	216	Flash observed	15.	132	No observation
5.	208	No observation	16.	126	Flash observed
6.	200	Flash observed	17.	120	No observation
7.	192	Flash observed	18.	116	No observation
8.	180	No observation	19.	110	No observation
9.	174	No observation	20.	104	No observation
10.	164	Flash observed	21.	100	No observation
11.	160	Flash observed			

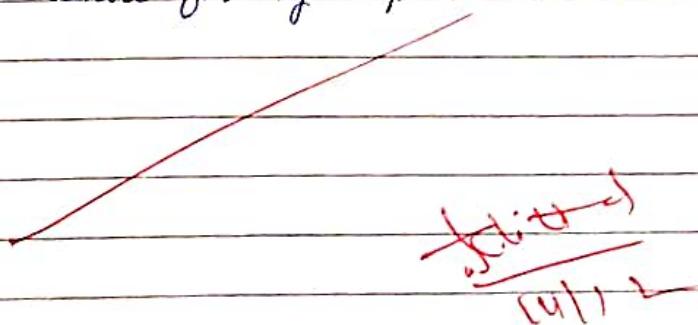
5) Check the occurrence of a flash at every 1°C drop in temperature. Record the latest temperature, the lowest one at which the flash is observed as the flash point of the sample.

Result:

- 1) The flash point of given lubricating oil determined by cleveland's apparatus is found to be 120°C .
- 2) The fire point of given lubricating oil determined by cleveland's apparatus is found to be 224°C .

Precautions:

- 1) Do not touch cup or oil unless fully cooled.
- 2) Suspend thermometer properly to avoid contact with base and cup.
- 3) When flash point is found, roughly scale down temperature difference to 2°C for more precise reading.
- 4) Avoid bubble formation while filling cup to avoid errors.



EXPERIMENT-05Aim:

To determine the effect of temperature on the viscosity of the given oil using Redwood viscometer.

Requirements:

Redwood viscometer, Thermometer, given sample of oil, stop watch, 50ml standard narrow necked flask.

Theory:

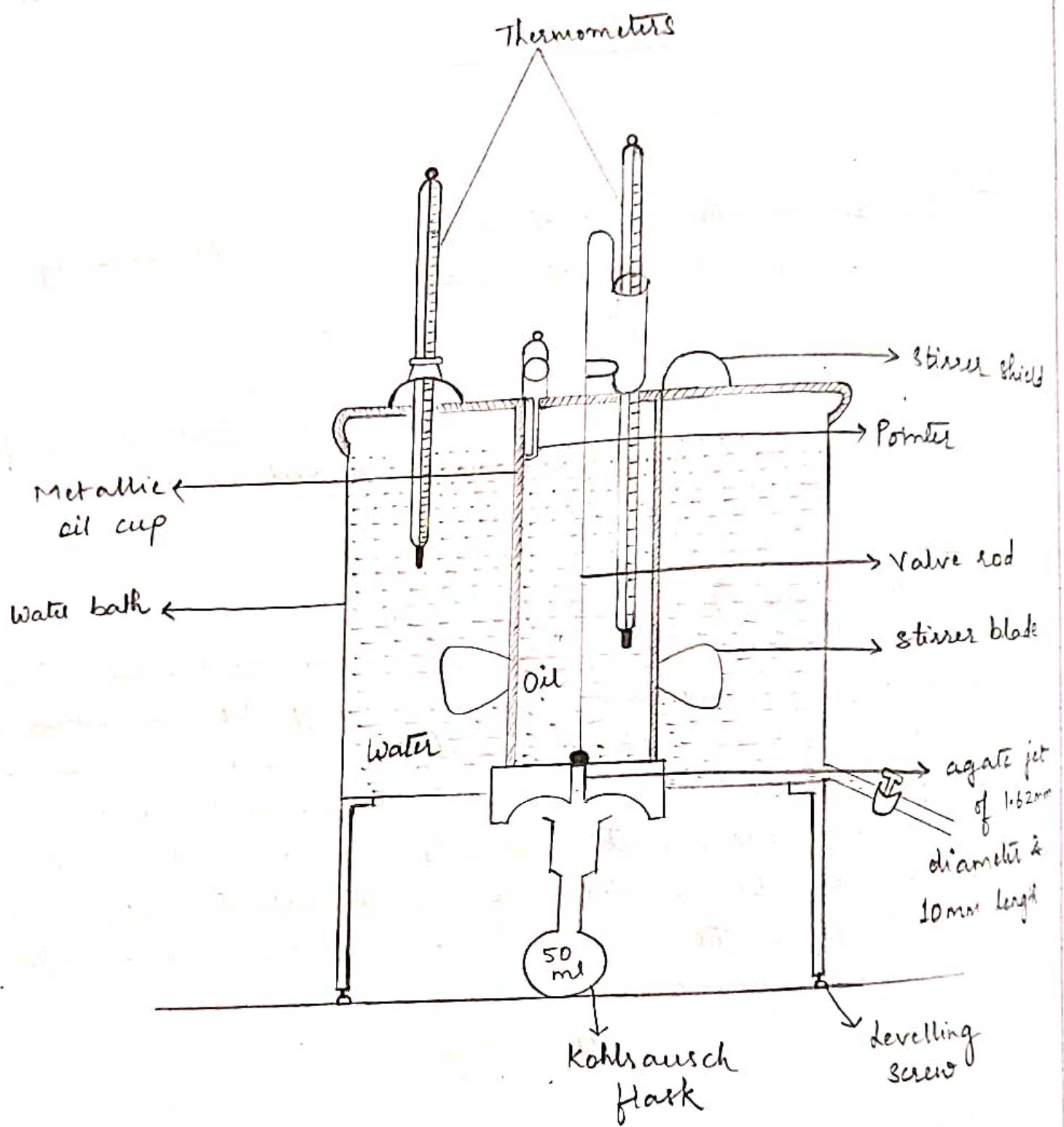
The viscosity of a lubricating oil is defined as the relative fluidity or the force with which it passes through a capillary of standard size, overcoming the resistance due to its backward force during its flow. This force is expressed in terms of pressure per unit area and its unit is dyne/cm². In practical applications, the viscosity is measured in terms of time in seconds for the flow of the oil through a capillary. Mathematically, the coefficient of viscosity (η) is represented by following equation:

$$\eta = \frac{F \cdot d}{v}$$

where, F = Force per unit area

d = distance between two layers

v = velocity of flow of the liquid



REDWOOD VISCOMETER - 1

Generally, the viscosity of the oil is not measured in the absolute units, instead it is measured in terms of time of its flow through an orifice of standard size at the prevailing temperature.

Viscosity can be measured through apparatus known as viscometers. There are three types of viscometers namely :- Saybolt's viscometer, Engler's viscometer and Redwood viscometer, which is being the most suitable as per India's weather conditions.

Viscosity of the oil decreases invariably with the rise in temperature. This determination of viscosity at varying temperature is necessary to provide efficient lubrication throughout the range of temperature of operation of the machine part.

Viscosity Index, rate at which the oil changes its viscosity with temperature. It is measured in terms of relative viscosity index of different oils. A good lubricant (oil) is the one which varies less with temperature. Low viscosity index because it is able to provide efficient lubrication at variable temperature.

Description of Instruments:

- 1) Oil cup of standard size having a mark upto which the oil has to be filled. The bottom of the oil cup is fitted with a jet made up of material 'agate'. The jet can be opened or closed by valve rod fitted with a brass ball. To record the temperature of the oil, arrangement to fix the thermometer is provided.

OBSERVATION TABLE

S. No.	Temperature (°C)	Volume of oil collected (ml)	Time (seconds)
1.	20°C	50	222
2.	32°C	50	85
3.	40°C	50	65
4.	48°C	50	44

2) Heating Bath :

The oil cup is surrounded by a cylindrical bath for containing the water. It is provided with an outlet for taking out the water from it. It consists of a thermometer holder and a stirrer. They record the temperature and also maintains the uniformity of desired temperature respectively.

3) Receivers :

A special type of receiver called Kohlrausch's flask to collect 50ml of oil.

4) Spirit Level :

It is used for levelling the apparatus vertically is also provided in the lid of the cup. The entire apparatus is supported on three legs, provided at the bottom with levelling screws.

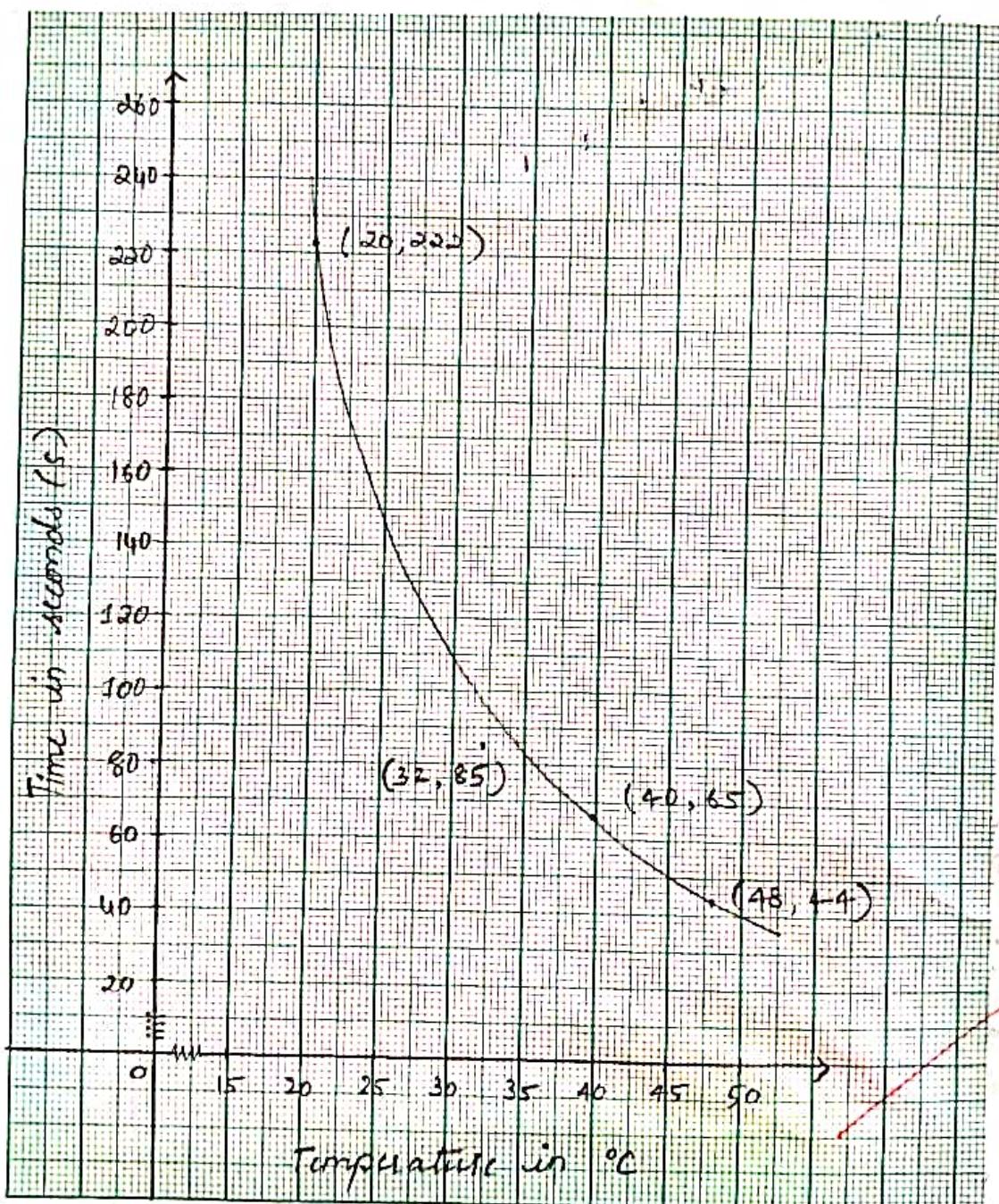
Procedure :

- 1) The given oil is filled upto the given mark in the oil cup.
- 2) Allow the oil to flow out through the surface orifice.
- 3) Exactly 50ml of oil is collected in a standard receiver, firstly at the room temperature & time of collection is noted down, with the help of a stop watch.
- 4) Same oil is filled again & temperature of waterbath is raised by about 5-6°C and temperature of the oil is recorded.
~~Maintain the uniformity of temperature of both oil and water so that temperature of oil remains constant during the period of flow of oil.~~

TEMPERATURE V/S TIME GRAPH

To plot the graph, temperature of the oil is taken on x-axis, in $^{\circ}\text{C}$ and time is taken on y-axis in seconds (s)

Scale: x-axis : 1 unit = 5°C (Temperature)
 y-axis : 1 unit = 20 seconds (time)



- 5) once again, 50ml of oil is collected again using same procedure and time of collection is noted.
- 6) Repeat the same procedure by varying 5-6 °C temperature and note the time of collection until the time difference is not too much.
- 7) Plot the graph accordingly and study the nature of the curve which leads to the calculation of the viscosity index of oil at a particular temperature in the numerical terms.
- 8) Compare the viscosity index of different lubricating oils at a particular temperature.

Result:

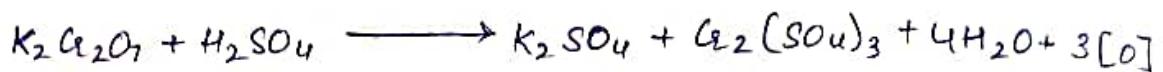
The temperature vs. time graph is plotted for the given lubricating oil. Graph shows that by increasing temperature, time of collection of oil decreases, which shows fall of viscosity with increase in temperature.

Precautions:

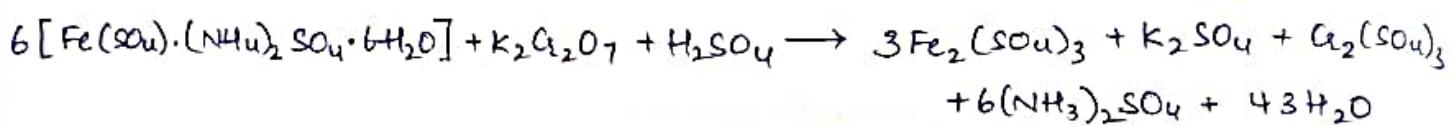
- 1) The oil cup and the receiver should be properly aligned, such that the oil flowing from the oil cup should fall clear in the receiver and should not slide down through the walls of the receiver.
- 2) The temperature of water and oil should be almost at the same value, which can be ensured by careful stirring.
- 3) Water should be filled in the heating bath, taking care that water does not fall in the oil cup to avoid creation of an emulsion.

Chemical Reactions:

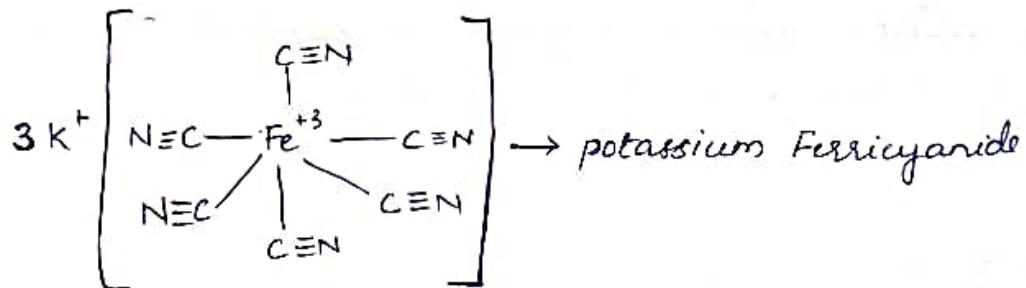
$K_2Cr_2O_7$ is a good oxidising agent and in the presence of dilute H_2SO_4 , it liberates three atoms of oxygen.



The liberated nascent oxygen oxidised Fe^{+2} ions in Ferrous Ammonium Sulphate to Fe^{+3} ions as expressed.



When the reaction is completed or all the Fe^{+2} ions get oxidised to Fe^{+3} ions, then they will not form any complex with indicator as they contain Fe^{+3} ions.



Formation of furo-ferricyanate



EXPERIMENT - 06

Aim:

To determine the strength of unknown FAS by titrating it against intermediate $K_2Cr_2O_7$ using potassium ferricyanide as an external indicator.

The normality of known FAS is $N/40$.

Requirements:

Apparatus: Burette, Pipette, beaker, conical flask, funnel, dropper, dish, porcelain dish plate, glass rod

Chemicals: a known solution of FAS ($\frac{N}{40}$), an unknown solution of potassium dichromate, external indicator $K_3[Fe(CN)_6]$

Theory:

The reaction in which oxidation as well as reduction of elements occur simultaneously is known as redox reactions.

$FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ is a stable double salt of $FeSO_4$, being its active constituent, acidic potassium dichromate is a strong oxidising agent and is rapidly reduced by Ferrous ions at the oxidising temperature to a green chromic salt when added to Mohr's salt.

$[FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O]$ solution containing dilute H_2SO_4 . In this reaction, ferrous sulphate is oxidised to ferric sulphate.

The solution will turn yellow with addition of dichromate ions and ferric ions are produced, they will also impart yellow colour to the solution, so there is no colour change during the reaction and hence another indicator is required.

Observation Tables

Titration of known FAS against $K_2Cr_2O_7$

S.No.	Volume of FAS (in ml)	Burette reading (in ml)		Final titrate reading
		initial	final	
1.	20.0	0.0	15.0	
2.	20.0	0.0	14.2	
3.	20.0	0.0	14.2	

Titration of unknown FAS against $K_2Cr_2O_7$

S.No.	Volume of FAS (in ml)	Burette reading (in ml)		Final titrate reading
		initial	final	
1.	20.0	0.0	14.0	
2.	20.0	0.0	13.8	
3.	20.0	0.0	13.8	

Procedure:

- 1) Wash the burette and rinse it with $K_2Cr_2O_7$ solution. Then, fill the burette with $K_2Cr_2O_7$ upto the initial mark.
- 2) Pipette out 20ml of Ferrous Ammonium Sulphate into the 50ml conical flask.
- 3) Acidify the $K_2Cr_2O_7$ solution with dilute H_2SO_4 .
- 4) The solution mixture of flask is then titrated with constant stirring against dichromate solution taken in the burette.
- 5) With the help of dropper, the indicator is taken in the porcelain plate.
- 6) After adding potassium dichromate. Test the titration mixture with indicator with the help of glass rod, then prussian blue colour is observed.
- 7) Repeat the above step for every 1 ml of dichromate solution until colourless solution is observed which indicates the completion of the titration.
- 8) Note down the readings and repeat the process three or more times until two concordant values are observed.

Calculation:

We know that in redox reaction, $NV = \text{constant}$

The strength of known FAS, $N_1 = \frac{N}{40}$

Let N_2 be the strength of $\text{K}_2\text{Cr}_2\text{O}_7$

Volume of FAS, $V_1 = 20 \text{ ml}$

Volume of $\text{K}_2\text{Cr}_2\text{O}_7 = V_2$.

$$N_1 V_1 = N_2 V_2$$

$$\Rightarrow N_2 = \frac{N_1 V_1}{V_2} = \frac{1}{40} \times \frac{20}{14.2}$$

$$N_2 = 0.035 N$$

Let N_3 be the normality of unknown FAS and

V_3 be the volume of unknown FAS, $V_3 = 20 \text{ ml}$.

$$N_2 V_2 = N_3 V_3$$

$$(0.035)(14.2) = N_3 (20)$$

$$\Rightarrow N_3 = 0.025 N$$

Strength = normality (N_3) \times equivalent weight of FAS

$$= 0.025 \times 392.2$$

$$= 9.8 \text{ gm/L}$$

Result:

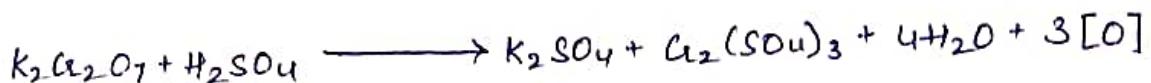
The strength of the given Ferrous Ammonium Sulphate is 9.8 gm/L.

Precautions:

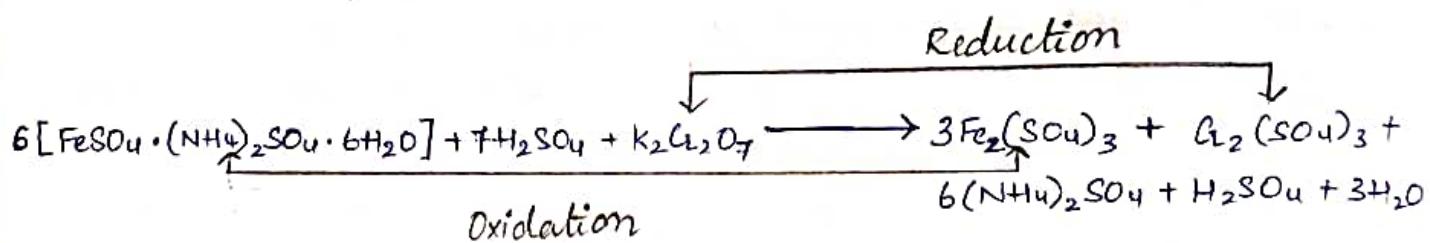
- 1) Dichromate is toxic to the environment. After the experiment, excess dichromate and the analyte are to be discarded in the designated bottles located on the reagent bench.
- 2) Dichromate solution should be freshly prepared.
- 3) Burette and pipette should be rinsed with the solution to be taken in it.
- 4) There should not be any leakage in the burette.
- 5) Concentrated sulphuric acid waste must be placed in the plastic labelled acid waste container. (Alternatively, it may be neutralized with sodium bicarbonate safety solution)

Chemical Reactions:

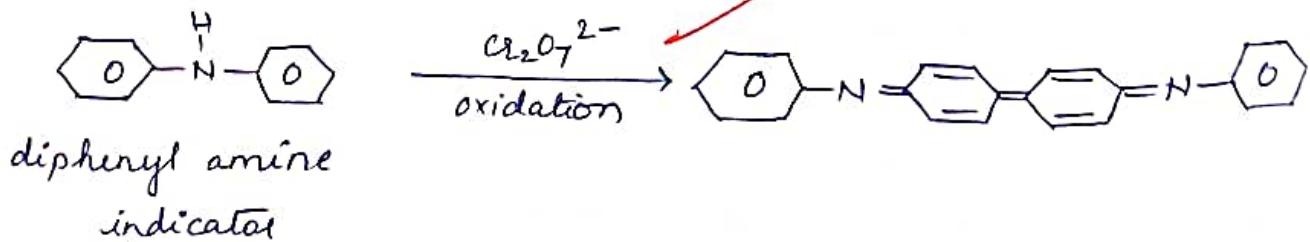
$K_2Cr_2O_7$ is a strong oxidising agent and in the presence of sulphuric acid, it liberates three atoms of oxygen.



The liberated nascent oxygen oxidises Fe^{+2} ions in Ferrous ammonium sulphate to Fe^{+3} ions.



On titration, diphenyl amine is used as internal indicator. At the end point, all the ferrous ions gets oxidised to ferric ions by chromate ions and as soon as a slight excess of dichromate is added, it will bring about the oxidation of diphenylamine and results in the formation of a blue-coloured complex. This indicates the end point of titration.



EXPERIMENT - 07Aim:

To determine the strength (in gm/L) of unknown Ferrous ammonium sulphate by titrating it against the standard potassium dichromate using diphenyl amine as an internal indicator.

Requirements:

Apparatus: Burette, pipette, conical flask, beaker, funnel, dropper.

Chemicals: Diphenyl amine indicator, potassium dichromate, Ferrous ammonium sulphate.

Theory:

The reaction in which oxidation as well as reduction of elements occur simultaneously is known as redox reactions. Ferrous ammonium sulphate or Mohr's salt is a stable double salt of Ferrous sulphate (FeSO_4) and ammonium sulphate $[(\text{NH}_4)_2\text{SO}_4]$ with FeSO_4 being its active constituent. In the presence of sulphuric acid, potassium dichromate oxidises ferrous sulphate to ferric sulphate, while ammonium sulphate remains unreacted. An internal indicator is added to the titration flask for detecting the end point of a redox reaction. The colour of diphenyl amine indicator changes from greenish to blue or purple colour at the end point of reaction. As the colour intensity is quite deep hence sometimes it is difficult to judge the end point. Therefore, to avoid this problem, the solution is diluted by adding 100 ml. of water. Now, the

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Observation Tables:

Titration of known FAS against $K_2Cr_2O_7$

S.N.	Volume of known FAS (ml)	Burette reading (ml)		Final titrate reading <i>wf</i>
		initial	final	
1.	25.0	0.0	20.5	
2.	25.0	0.0	19.1	
3.	25.0	0.0	19.1	

Titration of unknown FAS against $K_2Cr_2O_7$

S.No.	Volume of unknown FAS (ml)	Burette reading (ml)		Final titrate reading <i>wf</i>
		initial	final	
1.	25.0	0.0	18.5	
2.	25.0	0.0	18.2	
3.	25.0	0.0	18.2	

end point would be light blue or purple colour.

Procedure :

- 1) Wash the burette and rinse it with $K_2Cr_2O_7$ solution. Then, fill the burette with $K_2Cr_2O_7$ upto the initial mark.
- 2) Pipette out 25ml of ferrous ammonium sulphate into a clean 250ml conical flask. Add 100ml of water to the solution to dilute in order to get correct end point using measuring cylinder.
- 3) Add 5ml of sulphuric acid (H_2SO_4) solution to the mixture taken in conical flask. Then add 2-3 drops of diphenyl amine into the solution as an indicator using a dropper.
- 4) The solution mixture of flask is then titrated with constant stirring against potassium dichromate solution taken in burette until the colour of the solution changes from greenish to purple by a single drop addition.
- 5) Repeat this titration several times until concordant readings of burette are obtained.

Calculation:

1) Let N_1 and N_2 be the normalities of known FAS and potassium dichromate. Let V_1 and V_2 be the volumes of known FAS and potassium dichromate. Given that the normality of known FAS is $N/40$.

We know that,

$$N_1 V_1 = N_2 V_2$$

$$\frac{N}{40} \times 25 = N_2 \times (19.1)$$

$$\Rightarrow N_2 = \frac{25}{40 \times (19.1)}$$

$$\Rightarrow N_2 = 0.032 N$$

2) Let N_3, V_3 be the normality and volume of the unknown FAS.

$$N_2 V_2 = N_3 V_3$$

$$0.032 N \times (19.1) = N_3 \times 25$$

$$\Rightarrow N_3 = \frac{0.032 \times 19.1}{25}$$

$$\Rightarrow N_3 = 0.024 N$$

Strength = Normality of unknown FAS (N_3) \times equivalent weight of FAS
= 0.024×392.2
= 9.4 gm/L

Result:

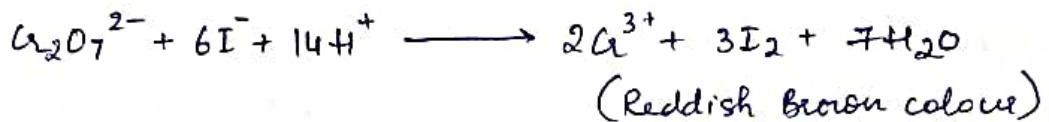
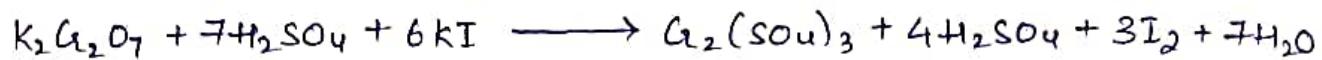
The strength of the given Ferrous ammonium sulphate is 9.4 gm/L

Precautions:

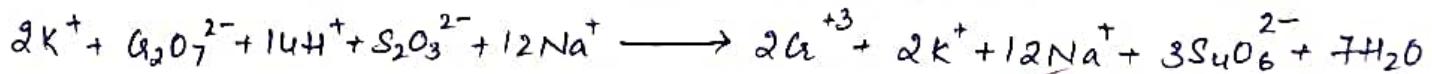
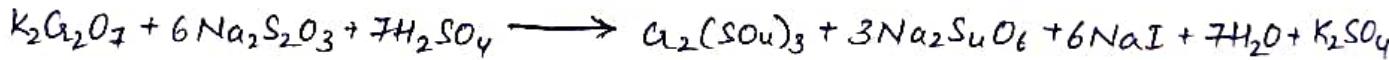
- 1) For clear observation, addition of titrant must be dropwise.
- 2) Use funnel to pour the solution in the burette
- 3) The burette should be checked properly and must be maintained without any leakage.
- 4) Do not blow into the pipette to make it completely empty.
- 5) Rinse all the glassware properly after using them and also before using them

Chemical Reactions:

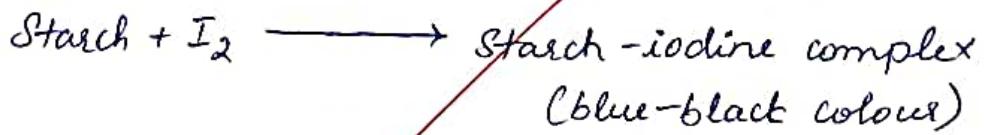
Iodometric reaction :



Net reaction :



Indicator reaction :



EXPERIMENT-08

Aim:

To determine the strength of unknown potassium dichromate ($K_2Cr_2O_7$) solution by titrating it against intermediate solution of sodium thio Sulphate (hypo) ($Na_2S_2O_3$) using Potassium Iodide (KI) as a source of Iodine and starch as an absorption indicator.

The normality of known $K_2Cr_2O_7$ is $N/40$.

Apparatus Required :

Burette, pipette, burette stand, dropper,
beaker, conical flask, measuring cylinder

Chemical Required :

Hypo solution, KI solution, $K_2Cr_2O_7$ solution with known strength ($N/40$), $K_2Cr_2O_7$ solution with unknown solution strength, freshly prepared starch.

Theory:

Iodometric titration is a method of volumetric analysis. A redox reaction where appearance and disappearance of elementary iodine indicates the end point. It involves indirect titration of iodine liberated by reaction with analyte.

For the determination of $K_2Cr_2O_7$ solution KI is added to acidify $K_2Cr_2O_7$ solution. It is given an equivalent amount of free iodine (I_2) which is then titrated against standard solution of sodium thio sulphate (hypo) using starch as indicator.

The iodine is reduced to sodium iodide (NaI) and hypo converts it into tetra thionate. Iodine also reacts with starch from blue colour.

Observation Tables:

Titration of known $K_2Cr_2O_7$ against $Na_2S_2O_3$

S.No.	Volume of known $K_2Cr_2O_7$ in ml	Burette reading in ml		Final titrate Reading(ml)
		initial	final	
1.	20.0	0.0	23.5	
2.	20.0	0.0	22.0	22.0
3.	20.0	0.0	22.0	

Titration of unknown $K_2Cr_2O_7$ against $Na_2S_2O_3$

S.No.	Volume of unknown $K_2Cr_2O_7$ in ml	Burette reading in ml		Final titrate Reading(ml)
		initial	final	
1.	20.0	0.0	20.5	
2.	20.0	0.0	20	
3.	20.0	0.0	20	20.0

iodo-starch complex. Acidified dichromate with iodine gives chromium ion.

$$\text{I}^- \longrightarrow \text{Cr}^{3+} + \text{I}_2$$

Now, I_2 dissolves in I^- solution to give I_3^- ions which are dark brown in colour.

The tri-iodide solution gives back I^- ion adding hypo solution. Starch used as indicator absorbs iodine and forms dark blue complex. This complex forms colourless near end point and gives light green colour of Cr^{3+} ion which is formed in the first step, this is imparted into reaction mixture. It should be noted that starch is added in a midway to prevent hydrolysis and titration should be carried out at low temperatures. At the end point, we get oceanic blue colour.

Procedure:

- 1) Wash and rinse the burette with water and hypo solution. Then, fill the burette with hypo solution upto the initial mark.
- 2) Pipette out 20 ml of known solution of $\text{K}_2\text{Cr}_2\text{O}_7$ in a 250ml conical flask.
- 3) Now add 5ml of KI solution to the conical flask and mix it well. so that the solution becomes brownish red due to formation of KI_3 complex with titration of I_2 .
- A) Titrate the liberated I_2 with intermediate solution of $\text{Na}_2\text{S}_2\text{O}_3$. The dark brown colour fades away slowly on addition of hypo solution.
- 5) Now add 2-3 drops of starch solution.
- 6) The solution now becomes ocean blue in colour on titrating the hypo solution, due to formation of iodo-starch complex.
- 7) When the solution turns into ocean blue, it is the end point of titration.

Calculation:

Let N_1, N_2, N_3 be the normalities of known K_2CrO_7 , $Na_2S_2O_3$ and unknown K_2CrO_7 . And v_1, v_2, v_3 be the volumes of known K_2CrO_7 , $Na_2S_2O_3$ and unknown K_2CrO_7 respectively.

$$(i) \quad N_1 v_1 = N_2 v_2$$

$$\frac{N}{20} \times 20 = N_2 \times 22$$

$$N_2 = \frac{N}{2(22)}$$

$$\Rightarrow N_2 = 0.023 N$$

$$(ii) \quad N_2 v_2 = N_3 v_3$$

$$(0.023)(20) = N_3 (20)$$

$$\Rightarrow N_3 = 0.023 N$$

Strength = normality of unknown K_2CrO_7 (N_3) \times equivalent weight of K_2CrO_7

$$= 0.023 \times 49$$

$$= 1.127 \text{ gm/L}$$

Ques. No. 3) Repeat the same procedure in case of unknown solution.

Result:

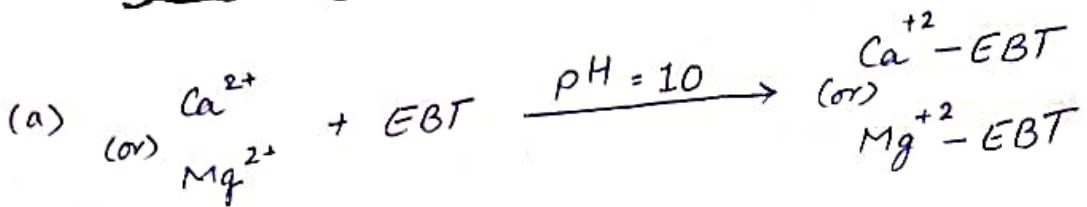
The strength of unknown K_2CrO_7 solution is 1.127 g/L.

Precautions:

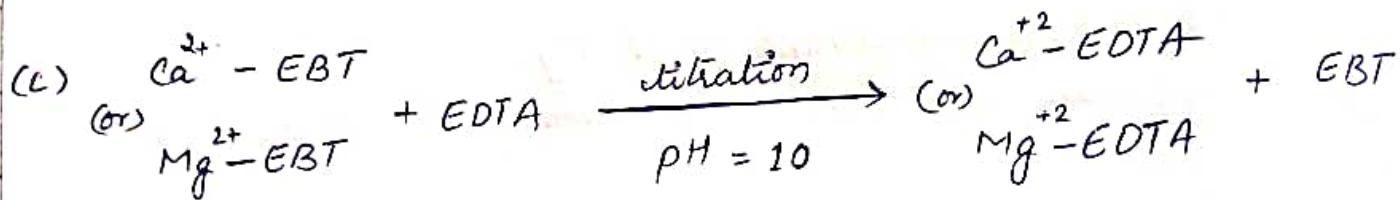
- 1) Rinse all the glassware properly before and after use.
- 2) Use funnel to pour solution in the burette.
- 3) Always take care of the meniscus.
- 4) Take the observation readings carefully.
- 5) Do not blow into the pipette to empty it completely.

(Q)

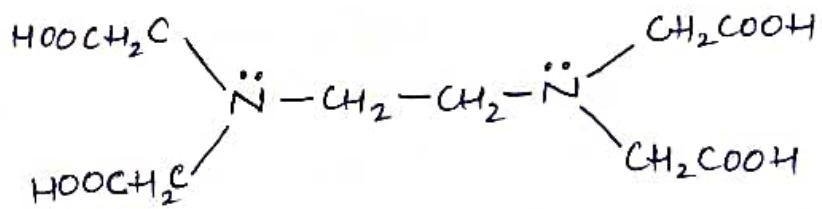
Chemical Reaction:



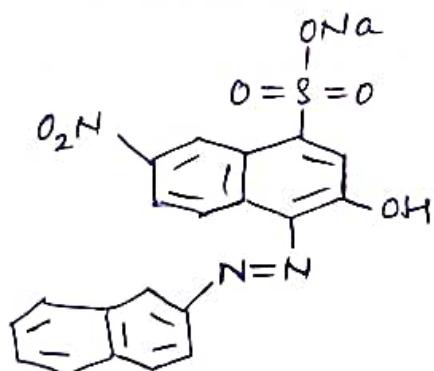
(b) colourless metal-EDTA stable complex formation



EDTA complex \Rightarrow



EBT \rightarrow



sodium 1-[1-hydroxy naphthylazo]-6-nitro-2-naphthol-4-sulphonate

EXPERIMENT - 09

Aim: To determine the total hardness of given water sample by titrating it against EDTA as titrimate and EBT as an indicator.

Materials Required:

Burette, burette stand, conical flask, dropper, beaker, EBT indicator, measuring cylinder of 100ml capacity and 10ml capacity, $\frac{N}{50}$ sample of standard hard water, tap water of 100ml each, EDTA, EBT.

EDTA - Ethylene Diamine Tetraacetic acid

EBT - Eriochrome Black - T

Theory:

The hardness of water is basically due to dissolved contents Ca^{2+} and Mg^{2+} ions. EBT binds with metal ions loosely while EDTA binds with metal ions strongly.

So, when all metal ions are bound to EDTA, indicator EBT remains free in the sample and the solution turns blue in colour. When EBT binds with free metal ion in water, it forms a red wine complex. EDTA has strong affinity for the metal ions than EBT. So, when EDTA is added, it replaces EBT and EBT returns to its blue colour.

Role of $\text{NH}_4\text{OH}-\text{NH}_4\text{Cl}$ (Buffer solution) in determination of hardness of water by EDTA. Following important reaction requires basic medium ($\text{pH} = 10$), maintained by addition of $\text{NH}_4\text{OH}-\text{NH}_4\text{Cl}$ buffer.

Observation Table:

1) Titration of standard hard water against EDTA

S.No	Volume of standard hard water (ml)	Burette Reading (ml)		Final titrate reading (ml)
		initial	final	
1.	100	0.0	7.5	
2.	100	0.0	6.0	
3.	100	0.0	6.0	6.0

2) Titration of tap water against EDTA

S.No	Volume of tap water (ml)	Burette Reading (ml)		Final titrate Reading (ml)
		initial	final	
1.	100	0.0	6.2	
2.	100	0.0	5.8	
3.	100	0.0	5.8	5.8

Calculation:

Let N_1, N_2, N_3 be the normalities of standard hard water, EDTA and tap water respectively. Let V_1, V_2, V_3 be the volumes of standard hard water, EDTA and tap water respectively.

$$1) \quad N_1 V_1 = N_2 V_2$$

$$\frac{N}{50} \times 100 = N_2 \times 6 \Rightarrow N_2 = 0.34 N$$

$$2) \quad N_2 V_2 = N_3 V_3$$

$$(0.34) (5.8) \cdot N_3 (100) \Rightarrow N_3 = 0.019 N$$

Strength of Tap water: Normality of tap water (N_3) \times equivalent weight
 $= 0.019 \times 50 \times 1000 \text{ mg/L}$
 $= 950 \text{ mg/L}$

Procedure:

- 1) Clean and rinse all the apparatus before starting experiment.
Wash the burette thoroughly with tap water and rinse with EDTA.
- 2) Take 100 ml of standard hard water into a conical flask. After that, add 5 ml of buffer solution and 2-3 drops of EBT into the conical flask and stir well.
- 3) The indicator is originally blue which acquires a wine red colour.
- 4) Titrate the solution with EDTA solution taken in a burette till the wine red colour turns blue.
- 5) When the solution turns to blue colour, it is the end point of reaction. Now, take the readings of burette.
- 6) Repeat the same experiment procedure in case of tap water.

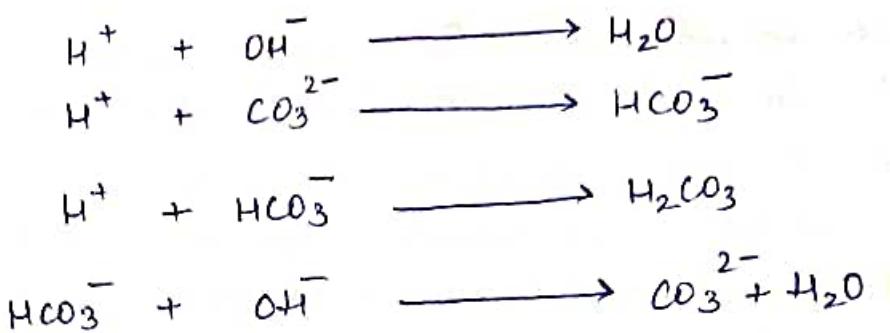
Result:

The total hardness of given tap water is 950 mg/l

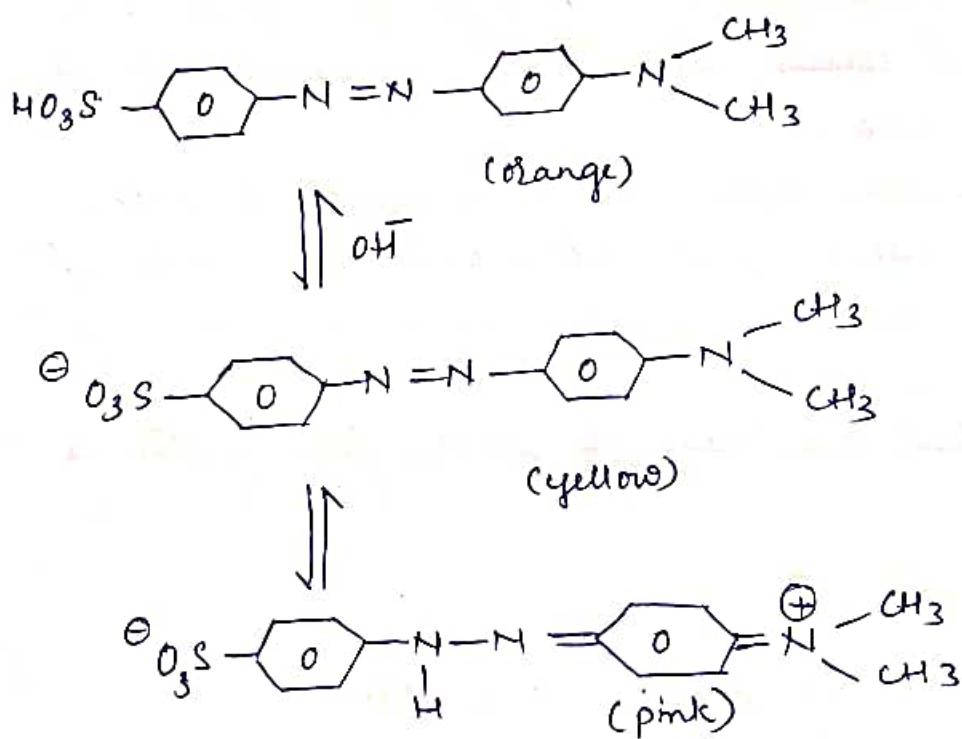
Precautions:

- 1) Rinse the equipment before using them.
- 2) Remove the funnel after filling the burette.
- 3) Use funnel to pour the solution into burette.
- 4) Always check the knob of burette before pouring the solution.
- 5) Always mix the reaction mixture by rotating it in circular motion.

Chemical Reactions:



Methyl Orange :



S.No	Result of Titration	Alkalinity		
		Hydroxide (OH^-)	Carbonate (CO_3^{2-})	Bicarbonate (HCO_3^-)
1.	$P = 0$	nil	nil	nil
2.	$P = M$	$P (\delta) M$	nil	nil
3.	$P = \frac{1}{2}M$	nil	$2P$	nil
4.	$P > \frac{1}{2}M$	$2P - M$	$2(M - P)$	nil
5.	$P < \frac{1}{2}M$	nil	$2P$	$M - 2P$

where
 P = phenolphthalein

M = methyl orange

EXPERIMENT - 10Aim:

To determine the alkalinity of water sample by titrating against H_2SO_4 (using methyl orange as indicator.)

Materials Required:

Burette, burette stand, conical flask, dropper, flask, measuring cylinder of 100 ml capacity, H_2SO_4 of normality $\frac{N}{50}$, 100 ml sample of tap water, 100 ml sample of hard water.

Theory:

The alkalinity of water is basically the amount of acid required to bring the sample at the pH of 4.2, so that the pH of alkalinity causing compounds in the water sample get used up. The total alkalinity of given water sample is reported in ppm or mg/L.

Procedure :

- 1) Rinse the burette with tap water to clear it. Then, fill it with H_2SO_4 solution using funnel.
- 2) Now, take the sample hard water of 100 ml in a conical flask.
- 3) Add 1-2 drops of methyl orange to the flask and mix well.
- 4) After mixing the solution, the orange colour turns yellow.
- 5) Now, titrate the water solution with H_2SO_4 solution, taken in the burette until the yellow colour turns to light pink colour. This is the end point.

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Observation Tables :

1) Titration of given 100ml of sample water (hard) against H_2SO_4

S.No	volume of std. hard water (ml)	Burette Readings (ml)		Final titrate Reading
		initial	final	
1.	100	0.0	7.4	
2.	100	0.0	6.5	
3.	100	0.0	6.5	6.5

2) Titration of 100ml of tap water against H_2SO_4

S.No	volume of tap water (ml)	Burette Readings (ml)		Final titrate Reading
		initial	final	
1.	100	0.0	2.8	
2.	100	0.0	2.5	
3.	100	0.0	2.5	2.5

Calculation :

Let N_1, N_2, N_3 be the normalities of standard hard water, H_2SO_4 and tap water respectively. Let V_1, V_2, V_3 be the volumes of standard hard water, H_2SO_4 and tap water respectively.

$$1) \quad N_1 V_1 = N_2 V_2$$

$$\frac{N}{50} \times 100 = N_2 \times (6.5)$$

$$N_2 = \frac{100}{6.5 \times 50} N \Rightarrow N_2 = 0.307 N$$

$$2) \quad N_2 V_2 = N_3 V_3$$

$$(0.307)(2.5) = N_3 \times 100$$

$$N_3 = \frac{0.307 \times 2.5}{100} \Rightarrow N_3 = 0.0076 N$$

$$\begin{aligned} \text{Strength of tap water} &= \text{normality of tap water} \times \text{equivalent weight of } \text{Ca}^{(2)} \\ &= 0.0076 \times 50 \times 1000 \\ &= 380 \text{ mg/L} \end{aligned}$$

- 6) Note the readings of burette carefully.
- 7) Repeat the same experiment three times.
- 8) Repeat the same procedure by taking 100ml of tapwater.

Result:

Alkalinity of Tapwater is 380 mg/l

Precautions:

- 1) Rinse all the glassware before and after use.
- 2) Always ensure that the knot of the burette is tightened.
- 3) Use a funnel to pour solution into the burette.
- 4) Remove funnel from burette after pouring solution in it for accurate measurement.
- 5) Take care while handling chemicals and glassware.