

# INSTRUCTION MANUAL FOR CHEMISTRY LABORATORY

(B. Tech. 1<sup>st</sup>/2<sup>nd</sup> Semester)

**Course Name** : Engineering Chemistry – II

**Course Code** : CYN11502/CYN12502

[Branch: Mechanical Engineering (ME), Production and Industrial Engineering (PIE), Engineering and Computational Mechanics (ECM), Civil Engineering (CE), Biotechnology (Biotech), Chemical Engineering (CHE)]



## Department of Chemistry

Motilal Nehru National Institute of Technology Allahabad  
Prayagraj, Uttar Pradesh – 211004, India

रसायन विभाग  
मोतीलाल नेहरू राष्ट्रीय प्रौद्योगिकी संस्थान इलाहाबाद,  
प्रयागराज, उत्तर प्रदेश - 211004, भारत

## Practical: List of Experiments

### Part A:

- (1) To determine the percentage of available chlorine in the supplied sample of bleaching powder.
- (2) To determine the total hardness,  $\text{Ca}^{2+}$  hardness, and  $\text{Mg}^{2+}$  hardness in the supplied water sample by titrating with standard EDTA solution.
- (3) To determine the alkalinity of the supplied water sample.
- (4) To determine the strength of supplied  $\text{K}_2\text{Cr}_2\text{O}_7$  solution using Ferrous Ammonium Sulphate solution as intermediate and Potassium Ferricyanide solution as an external indicator.
- (5) Preparation of Methyl Orange from sulfanilic acid using a diazonium coupling reaction.

### Part B:

- (6) Determination of flash point of oils by Abel's apparatus.
- (7) Determination of flash point of lubricating oil by Pensky-Martens closed cup tester.
- (8) To study the kinematic viscosity of the given lubricating oil at various temperatures using a Redwood viscometer.
- (9) To find out the aniline point of the given fuel samples.
- (10) Determination of viscosity average molecular weight of a polymer sample by Ostwald Viscometer.

### Part C:

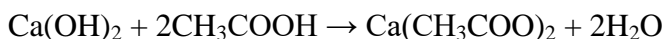
- (11) Preparation and characterization of biodiesel from waste cooking oils.

# Experiment: 1

To determine the percentage of available chlorine in the supplied sample of bleaching powder.

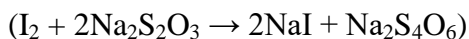
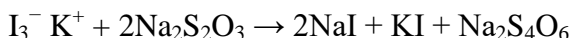
**Chemicals and glassware:** Sodium thiosulphate, potassium iodide, glacial acetic acid, starch solution, porcelain dish, test tube, 250 mL volumetric flask, funnel, burette, pipette, beaker, and conical flask.

**Reactions:** i)  $\text{CaOCl}_2 + \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2 + \text{Cl}_2$  (  $\text{Cl}_2 + \text{H}_2\text{O} \rightleftharpoons \text{HOCl} + \text{HCl}$  )



ii)  $\text{Cl}_2 + 2\text{KI} \rightarrow 2\text{KCl} + \text{I}_2$

iii)  $\text{I}_2 + \text{KI} \rightarrow \text{I}_3^- \text{K}^+$



**Procedure:** Weigh accurately about 1.5 g of bleaching powder in the porcelain dish, and rub it with the flat end of the test tube after adding a little amount of deionized water until a thick paste is formed. Transfer the contents of the dish completely into a 250 mL volumetric flask by adding more deionized water and dilute it upto the half of volumetric flask. Add 3 mL of glacial acetic acid and a little amount of deionized water to the dish, wash, and transfer it into the volumetric flask. Shake the volumetric flask after washing the funnel by adding around 20–30 mL of deionized water to get a clear solution and then, dilute it with deionized water to the mark. Pipette out 25 mL of the clear solution and transfer it into a conical flask. Add 2 mL of glacial acetic acid and 1 mL of KI solution resulting in a deep brown solution. Immediately, titrate the liberated iodine with standard Hypo solution (Sodium thiosulphate,  $\text{Na}_2\text{S}_2\text{O}_3$ ) taken in the burette till the faint yellow colour of the remaining iodine. Now, add about 10 drops of starch solution which gives a deep blue colour to the contents of the conical flask. Continue the titration with Hypo solution till the blue colour just disappears and becomes a colourless solution.

Repeat the process by taking 25 mL of beaching powder solution until get two concordant readings with Hypo solution.

(Eq. Wt. of Hypo = 248.19 g)

## Observations:

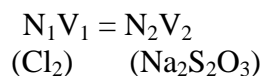
1. Weight of bleaching powder taken = W g = ..... g
2. Strength of Hypo solution (supplied) = ..... g/L

**Table: Volume of Hypo solution used for 25 mL of Bleaching Powder solution**

S. No.	Burette reading		Volume used, (V <sub>2</sub> -V <sub>1</sub> ) mL	Concordant Value, V mL
	Starting Point, V <sub>1</sub> mL	End Point, V <sub>2</sub> mL		
1				
2				
3				

**Calculations:**

Consider, X is the strength of available chlorine in normality (N), then



$$X \times 25 = \frac{V \times \text{Strength of Hypo solution (g/L)}}{\text{Eq. Wt. of Hypo}}$$

$$\therefore X = \frac{V \times \text{Strength of Hypo solution (g/L)}}{25 \times \text{Eq. Wt. of Hypo}} \text{ N}$$

i.e., X mole of chlorine is available in 1 L or 1000 mL of the bleaching powder dissolved solution.

∴ The strength of available chlorine (g) in 1000 mL of the bleaching powder dissolved solution,

$$Y = \text{Normality} \times \text{Eq. Wt. of chlorine (Cl}_2\text{)} = X \times 35.45 \text{ g}$$

i.e., Y g of chlorine is available in 1000 mL of the bleaching powder dissolved solution.

In 250 mL,

$$Z = \frac{Y \times 250}{1000}$$

∴ Z g of chlorine is available in 250 mL of the bleaching powder dissolved solution.

i.e., W g of bleaching powder contains Z g of available chlorine.

∴ The percentage of available chlorine in the supplied sample of bleaching powder is

$$= \frac{Z \times 100}{W} \%$$

**Result:** The % of available chlorine in the supplied sample of bleaching powder is .....

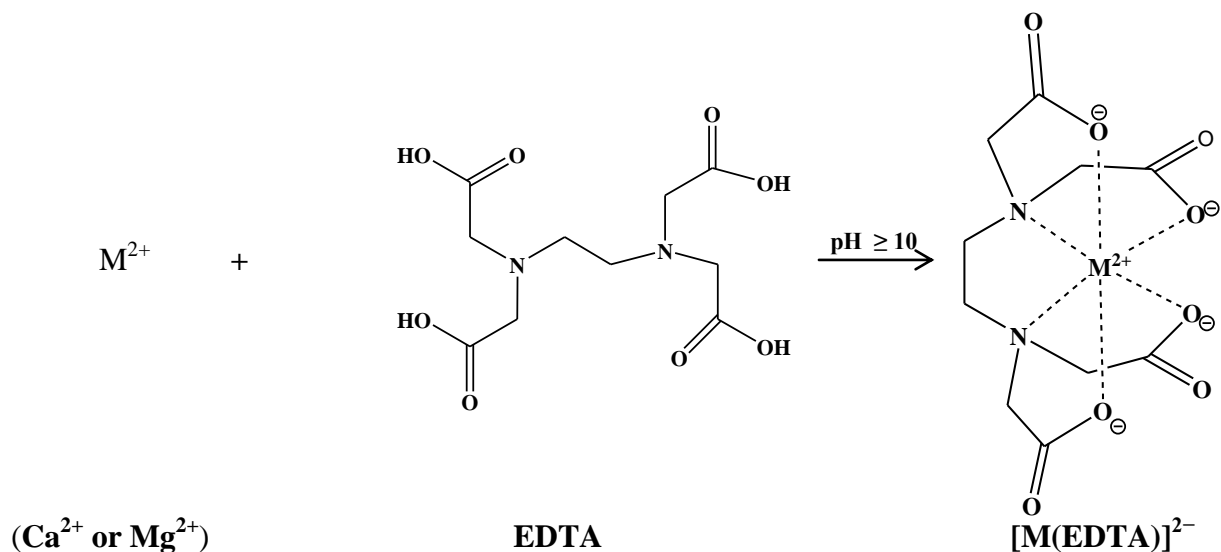
## Experiment: 2

To determine the total hardness,  $\text{Ca}^{2+}$  hardness, and  $\text{Mg}^{2+}$  hardness in the supplied water sample by titrating with standard EDTA solution.

**Chemicals and glassware:** Calcium carbonate, hydrochloric acid, EDTA solution, buffer solution (pH 10), Eriochrome Black T, Murexide, sodium hydroxide, weighing tube, 250 mL volumetric flask, funnel, glass rod, conical flask, pipette, burette, and beaker.

**Theory:** For determining Calcium and Magnesium hardness separately, the total hardness is being determined first by titration against Ethylenediaminetetraacetic acid (EDTA) using the buffer of pH 10 and Eriochrome Black T (EBT) indicator. In the next step, Magnesium ions are precipitated in the form of  $\text{Mg}(\text{OH})_2$  by adding  $\text{NaOH}$  which rises pH of the solution to 12.5. Calcium hardness left in the water can be determined by titration using Murexide indicator.

### Reaction:



### Procedure:

#### Standardization of EDTA solution

Prepare a standard solution of  $\text{CaCO}_3$  by dissolving 0.250 g of  $\text{CaCO}_3$  in minimum amount of dil.  $\text{HCl}$  by boiling. Cool and make the solution in a 250 ml volumetric flask using deionized water. Take 25 mL of standard solution in a conical flask and add 2 mL buffer solution of pH 10 (supplied). Now, add 2–3 drops of the EBT indicator to the solution- a wine red colour appears. Titrate against EDTA solution till a blue colour appears. Note the observation for two concordant readings ( $V_s$  mL) in Table 1.

**To determine the total hardness of the water sample**, take 25 mL of water sample in a conical flask, add 2 mL of buffer solution of pH 10, and 2–3 drops of the EBT indicator. Titrate against standard EDTA solution and record the readings as  $V_t$  in Table 2.

**To determine the calcium hardness**, Transfer 25 mL of water sample into a conical flask with the help of a pipette. Add 5 mL of NaOH and 3–4 drops of Murexide indicator. Shake the flask and titrate it against standard EDTA solution until the colour changes from slight reddish to slight pink. Repeat the titration until two concordant readings are obtained. Record the observations as  $V_c$  in Table 3.

**Observations:**

1. Weight of empty weighing tube =  $W_1$  g = ..... g
2. Weight of tube +  $\text{CaCO}_3$  powder =  $W_2$  g = ..... g
3. Weight of tube after transferring the substance to volumetric flask =  $W_3$  g = .....g
4. Weight of  $\text{CaCO}_3$  powder actually taken  $W = (W_2 - W_3)$  g = .....g
5. Observation for standardisation of EDTA ( $V_s$ ) (Table 1) = ..... mL
6. Observation for Titration with EBT ( $V_t$ ) for total hardness (Table 2) = ..... mL
7. Observation for Titration with Murexide ( $V_c$ ) (Table 3) = ..... mL

**Table 1: Titration value for standardization of EDTA solution against standard  $\text{CaCO}_3$  solution.**

S. No.	Burette reading		Volume used, ( $V_2 - V_1$ ) mL	Concordant Value, $V_s$ mL
	Starting Point, $V_1$ mL	End Point, $V_2$ mL		
1.				
2.				
3.				

**Table 2: Titration value for total hardness of unknown water sample**

S. No.	Burette reading		Volume used, ( $V_4 - V_3$ ) mL	Concordant Value, $V_t$ mL
	Starting Point, $V_3$ mL	End Point, $V_4$ mL		
1.				
2.				
3.				

**Table 3: Titration value for Calcium hardness of unknown water sample**

S. No.	Burette reading		Volume used, (V <sub>6</sub> -V <sub>5</sub> ) mL	Concordant Value, V <sub>c</sub> mL
	Starting Point, V <sub>5</sub> mL	End Point, V <sub>6</sub> mL		
1.				
2.				
3.				

**Calculations:**

**1. Standardization of EDTA solution**

Weight of CaCO<sub>3</sub> in 250 ml = W g

∴ Weight of CaCO<sub>3</sub> per litre = 4W g

Normality of CaCO<sub>3</sub> solution =  $\frac{4W}{50} = N_2$  (say)

Then,

$N_1 \times V_s = N_2 \times V_2$  where N<sub>1</sub> is the normality of EDTA solution.  
(EDTA) (CaCO<sub>3</sub>)

$$N_1 = \frac{4W}{50} \times \frac{25}{V_s} = \dots\dots\dots N$$

**2. Total hardness of a given water sample**

$$N_1 \times V_t = N_2 \times 25$$

(EDTA) (Water sample)

$$N_2 = \frac{N_1 \times V_t}{25}$$

Total hardness of the water sample in ppm =  $N_2 \times \text{gram eq. wt. of CaCO}_3 \text{ g/1000 mL}$   
 $= \frac{N_1 \times V_t \times 50 \times 1000}{25} \text{ mg/1000 mL or ppm}$

**3. Calcium hardness of a given water sample**

Calcium hardness of the water sample in ppm =  $\frac{N_1 \times V_c \times 50 \times 1000}{25}$  ppm

**Results:**

- (I) Total hardness of a given water sample = ..... ppm
- (II) Ca-hardness of a given water sample = ..... ppm
- (III) Mg-hardness of a given water sample = (Total hardness – Ca-hardness)  
 = ..... ppm

## Experiment: 3

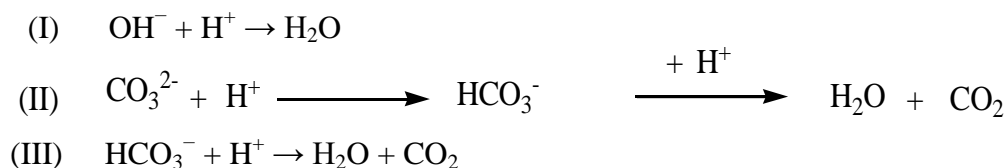
**To determine the alkalinity of the supplied water sample.**

**Chemicals and glassware:** Sodium carbonate, acid solution, phenolphthalein, methyl orange, 250 mL volumetric flask, funnel, glass rod, weighing tube, conical flask, pipette, and burette.

**Theory:** In a water sample, the alkalinity is generally due to the presence of hydroxide, carbonate and / or bicarbonate ions. Alkalinity can be expressed as follows:

$$\text{Alkalinity (mol/L)} = [\text{HCO}_3^-] + 2 [\text{CO}_3^{2-}] + [\text{OH}^-] - [\text{H}^+]$$

It is determined by the titration with the standard acid solution. Titration is performed in the presence of two indicators i.e. phenolphthalein and methyl orange to characterize the type of alkalinity present and the composition of alkalinity.



### **Procedure:**

#### **Standardization of acid solution:**

Weigh accurately 0.265 g of Na<sub>2</sub>CO<sub>3</sub> (dried) A. R. grade, transfer it in a 250 mL volumetric flask. Dissolve in about 100 mL deionized water and make it up to the mark by diluting with more deionized water. Pipette out 25 mL of this solution in a conical flask add 2–4 drops of methyl orange indicator and titrate with acid solution. Note down the end point when light yellow colour changes to red. Repeat the titration for two concordant readings. Note down the readings as V<sub>s</sub> as given below in the Table 1.

#### **Titration of unknown water sample:**

Pipette out 25 mL of water sample in a clean conical flask, add 2–4 drops of phenolphthalein indicator and titrate with standard acid solution till pink colour disappears. Record the volume of acid used as V<sub>p</sub> mL. To the same solution, add 2–3 drops of methyl orange indicator, and continue the titration with same acid solution. At the end point, light yellow colour sharply changes to red. Record the reading as V<sub>m</sub> mL. Repeat the titration by taking 25 mL of water sample until get two concordant observations as given below in the Tables 2 and 3.



**Observations:**

1. Weight of empty weighing tube =  $W_1$  g = ..... g
2. Weight of tube +  $\text{Na}_2\text{CO}_3$  powder =  $W_2$  g = ..... g
3. Weight of tube after transferring the substance to volumetric flask =  $W_3$  g = .....g
4. Weight of  $\text{Na}_2\text{CO}_3$  powder actually taken  $W = (W_2 - W_3)$  g = .....g
5. Volume of the acid used for standardisation with  $\text{Na}_2\text{CO}_3$  =  $V_s$  mL = ..... mL

**Table 1: Titration value for standardization of acid solution**

S. No.	Burette reading		Volume used, ( $V_2 - V_1$ ) mL	Concordant Value, $V_s$ mL
	Starting Point, $V_1$ mL	End Point, $V_2$ mL		
1.				
2.				
3.				

**Titration values for unknown water sample****Table 2: Using phenolphthalein indicator**

S. No.	Burette reading		Volume used, ( $V_4 - V_3$ ) mL	Concordant Value, $V_p$ mL
	Starting Point, $V_3$ mL	End Point, $V_4$ mL		
1.				
2.				
3.				

**Table 3: Using methyl orange indicator**

S. No.	Burette reading		Volume used, ( $V_5 - V_3$ ) mL	Concordant Value, $V_m$ mL
	Starting Point, $V_3$ mL	End Point, $V_5$ mL		
1.				
2.				
3.				

**Calculations:**

**(a) Standardization of acid by  $\text{Na}_2\text{CO}_3$**

$N_1$  is the normality of acid solution.

$$\begin{array}{cc} N_1 V_1 = N_2 V_2 \\ \text{(Acid)} & \text{(Na}_2\text{CO}_3\text{)} \end{array}$$

$$N_1 \times V_s = \frac{W}{53} \times \frac{1000}{250} \times 25$$

$$N_1 \times V_s = \frac{4W}{53} \times 25$$

Therefore, 
$$N_1 = \frac{4W}{53} \times \frac{25}{V_s} = \dots\dots\dots N$$

**(b) Determination of the alkalinity of the supplied water sample**

$$\begin{array}{cc} N_1 \times V = N_2 \times 25 & \text{where } N_2 \text{ is the normality of alkaline water (CaCO}_3\text{).} \\ \text{(Acid)} & \text{(Water sample)} \end{array}$$

$$N_2 = \frac{N_1 \times V}{25}$$

$$\begin{aligned} \text{Alkalinity of the water sample in ppm} &= N_2 \times \text{gram eq. wt. of CaCO}_3 && \text{g/1000 mL} \\ &= \frac{N_1 \times V}{25} \times 50 \times 1000 && \text{mg/1000 mL or ppm} \end{aligned}$$

Therefore,

**Alkalinity up to phenolphthalein end point (in ppm),**

$$P = \frac{N_1 \times V_p \times 50 \times 1000}{25} \text{ ppm}$$

**Similarly, alkalinity up to methyl orange end point (in ppm),**

$$M = \frac{N_1 \times V_m \times 50 \times 1000}{25} \text{ ppm}$$

The type of alkalinity can be obtained from the following table (interpreting by P and M)

S. No.	Condition	$\text{OH}^-$ in ppm	$\text{CO}_3^{2-}$ in ppm	$\text{HCO}_3^-$ in ppm
(I)	$P = 0$	Nil	Nil	M
(II)	$P = M$	P	Nil	Nil
(III)	$P = \frac{1}{2} M$	Nil	2P	Nil
(IV)	$P > \frac{1}{2} M$	$2P - M$	$2(M - P)$	Nil
(V)	$P < \frac{1}{2} M$	Nil	2P	$M - 2P$

**Results:**

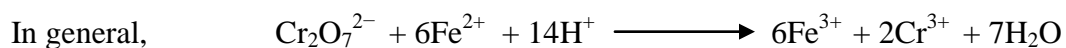
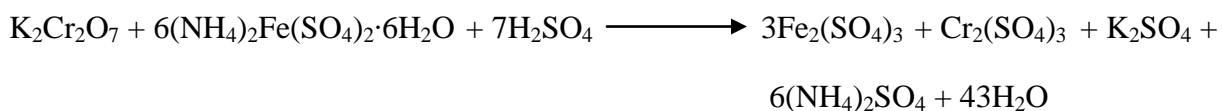
- (I)  $\text{OH}^-$  alkalinity = ..... ppm
- (II)  $\text{CO}_3^{2-}$  alkalinity = ..... ppm
- (III)  $\text{HCO}_3^-$  alkalinity = ..... ppm

## Experiment: 4

To determine the strength of supplied  $K_2Cr_2O_7$  solution using Ferrous Ammonium Sulphate (FAS) solution as an intermediate and Potassium Ferricyanide solution as an external indicator.

**Chemicals and glassware:** Potassium dichromate, ferrous ammonium sulphate, potassium ferricyanide, dil.  $H_2SO_4$ , 250 mL volumetric flask, conical flask, pipette, burette, funnel, glass rod, weighing tube, and white tile.

### Reactions:



### Procedure:

#### Standardization of FAS solution:

Weigh out exactly about 0.6129 g of  $K_2Cr_2O_7$  crystals in weighing tube. Transfer it into a 250 mL volumetric flask, dissolve it with deionized water, and make the solution up to the mark. Fill the burette with prepared  $K_2Cr_2O_7$  solution. Pipette out 25 mL of Ferrous Ammonium Sulphate solution (FAS, supplied) in the conical flask and add 5 mL of dil.  $H_2SO_4$  (supplied). Put a number of drops of freshly prepared Potassium Ferricyanide solution (supplied) on a clean dry white tile with the help of a glass rod. Now, add  $K_2Cr_2O_7$  solution gradually 1 mL at a time from the burette. After adding of each mL, shake the contents of the conical flask and put a drop of the solution over one drop of the indicator on the tile with the help of a glass rod. After touching the drop, wash the rod thoroughly in running (tap) water follow by deionized water and dry it. The end point is the disappearance of the blue color when a drop of the solution is in contact with the indicator placed on a tile. Suppose, the drop gets coloured at 15 mL of  $K_2Cr_2O_7$  solution and remains unchanged at 16 mL. This indicates that the end point lies between 15 mL and 16 mL of  $K_2Cr_2O_7$  solution. Again, take 25 mL of FAS solution in the conical flask, add 5 mL of dil.  $H_2SO_4$  and titrate upto 15 mL of  $K_2Cr_2O_7$  solution directly from the burette. Later, add  $K_2Cr_2O_7$  solution gradually 0.1 mL at a time and test the solution as before. Find the end point at which color unchanged. Repeat the procedure until to get atleast two concordant readings.

#### Titration of supplied unknown $K_2Cr_2O_7$ solution:

Fill the burette with unknown  $K_2Cr_2O_7$  solution, pipette out 25 mL of FAS solution in a conical flask, and follow the same procedure until get two concordant readings.

$$(\text{Eq. Wt. of } K_2Cr_2O_7 = 49.03)$$

**Observations:**

1. Weight of empty weighing tube =  $W_1$  g = ..... g
2. Weight of tube +  $K_2Cr_2O_7$  crystal =  $W_2$  g = ..... g
3. Weight of tube after transferring the substance to volumetric flask =  $W_3$  g = ..... g
4. Weight of  $K_2Cr_2O_7$  crystal actually taken,  $W = (W_2 - W_3)$  g = ..... g

**Table 1: Titration values to determine the concentration of supplied FAS solution**

Volume of FAS solution for standardization,  $V = 25$  mL

S. No.	Burette reading		Volume used, ( $V_2 - V_1$ ) mL	Concordant Value, $V_s$ mL
	Starting Point, $V_1$ mL	End Point, $V_2$ mL		
1.				
2.				
3.				

**Table 2: Titration value for unknown  $K_2Cr_2O_7$  solution**

Volume of FAS solution for unknown  $K_2Cr_2O_7$  solution,  $V = 25$  mL

S. No.	Burette reading		Volume used, ( $V_4 - V_3$ ) mL	Concordant Value, $V_u$ mL
	Starting Point, $V_3$ mL	End Point, $V_4$ mL		
1.				
2.				
3.				

**Calculations:**

**(a) Standardization of FAS by known  $K_2Cr_2O_7$  solution**

$$N_1 V_1 = N_2 V_2$$

(FAS)    ( $K_2Cr_2O_7$ )

$$N_1 \times V = N_2 \times V_s$$

$$N_1 \times 25 = \frac{W}{49.03} \times \frac{1000}{250} \times V_s$$

$$\text{Normality of FAS, } N_1 = \frac{4W}{49.03} \times \frac{V_s}{25} = \dots\dots\dots N$$

**(b) Determination of the strength of a supplied unknown  $K_2Cr_2O_7$  solution**

$$\begin{array}{l} N_1 V_1 = N_2 V_2 \\ \text{(FAS) (K}_2\text{Cr}_2\text{O}_7\text{)} \\ N_1 \times V = N_2 \times V_u \quad \text{where } N_2 \text{ is the normality of unknown } K_2Cr_2O_7 \text{ solution.} \end{array}$$

Therefore, 
$$N_2 = \frac{N_1 \times 25}{V_u} = \dots\dots\dots N$$

The strength of supplied  $K_2Cr_2O_7$  solution =  $N_2 \times 49.03 = \dots\dots\dots \text{ g/L}$

**Result:**

The strength of supplied  $K_2Cr_2O_7$  solution is  $\dots\dots\dots \text{ g/L}$ .

## Experiment: 5

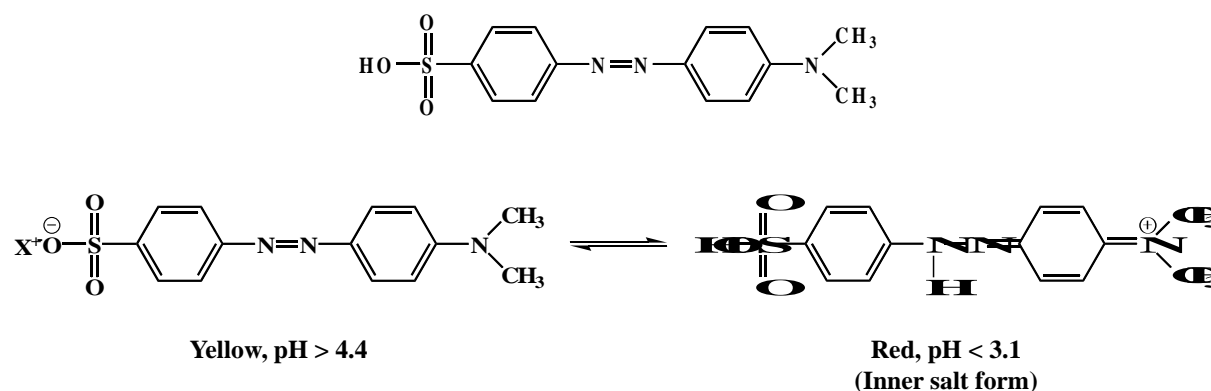
**Preparation of Methyl Orange from sulfanilic acid using a diazonium coupling reaction.**

**Chemicals and glassware:** Sulfanilic acid, anhydrous sodium carbonate, sodium nitrite, Conc. HCl, *N,N*-dimethylaniline, glacial acetic acid, sodium hydroxide, sodium chloride, 250 mL beaker,

**Theory:**

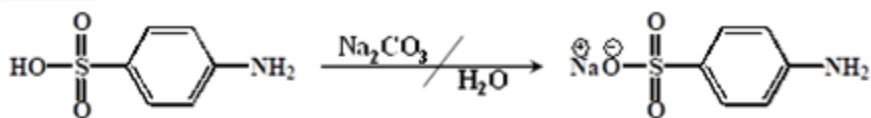
Methyl orange is an azo dye that forms beautiful orange crystals and is used as an acid-base indicator. The anion form is yellow and the acid form is red.

Structure of Methyl Orange



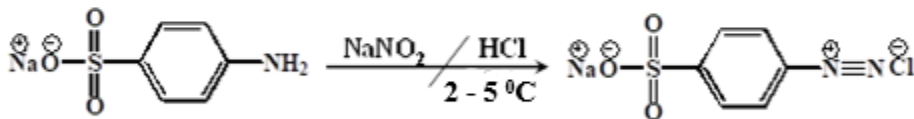
**Reactions:**

**Step-I**

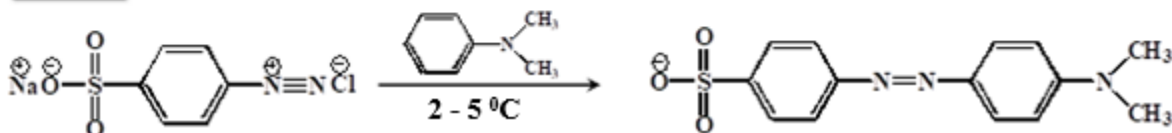


Sulfanilic acid

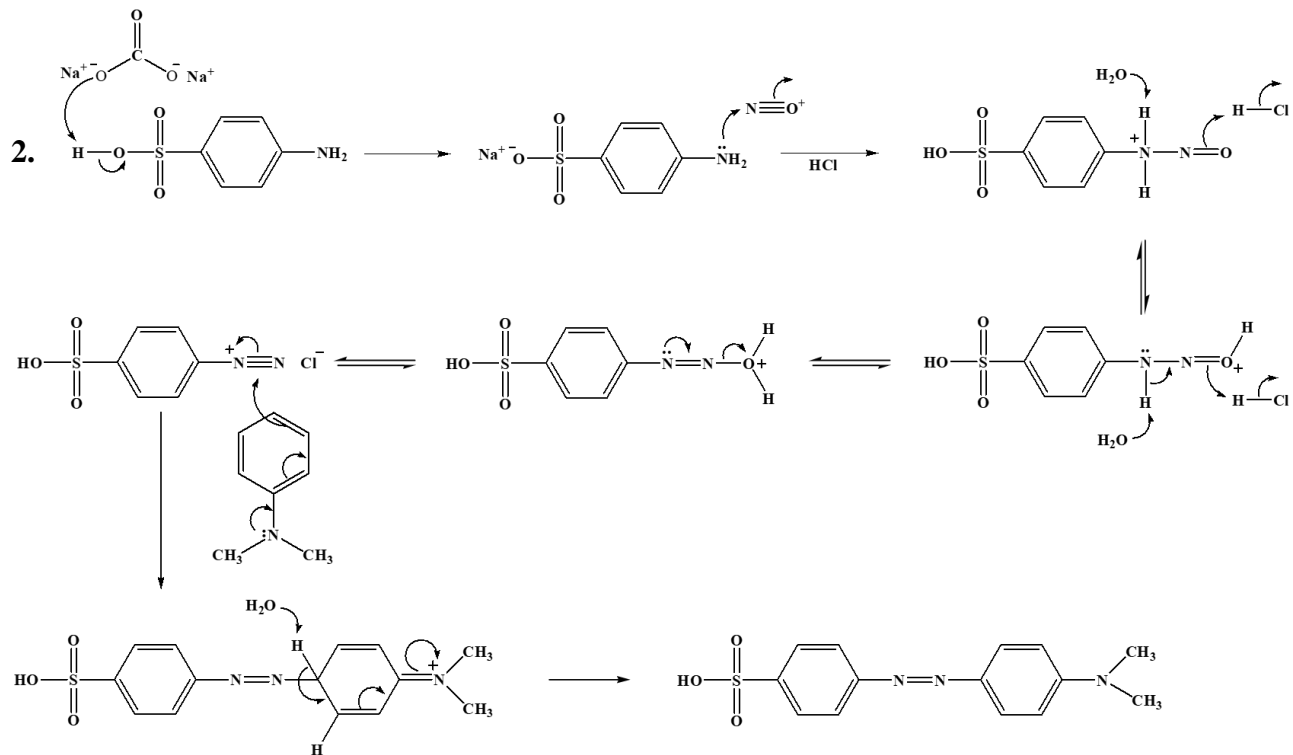
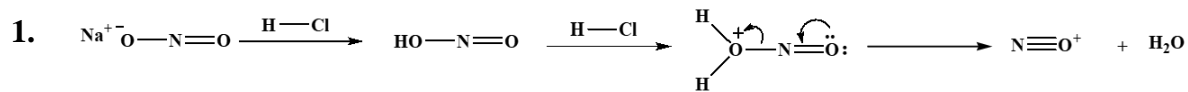
**Step-II**



**Step-III**



### Reaction mechanism:



### Procedure:

Take 5 g of sulfanilic acid, 1.5 g of anhydrous sodium carbonate, and 50 mL of deionized water in a 250 mL beaker and stir the mixture to obtain a clear solution (warm if necessary). Cool the resulting solution in ice-salt freezing mixture. To this cold solution, first add sodium nitrite solution (2 g of Sodium Nitrite dissolved in 15 mL of ice-cold water) and then, add ice-cold hydrochloric acid solution (5 mL of Conc. HCl in 25 mL of ice-cold water) slowly with continuous stirring. During this addition, the temperature of the overall solution should be maintained between 2–5 °C. Test for free nitrous acid with potassium iodide starch paper. Fine crystals of Diazobenzene sulphonate will separate after 15 min. **Do not filter as this will be dissolved during next stage of preparation.**



In a 100 mL beaker, mix 3.1 mL of *N,N*-dimethylaniline and 1.5 mL of glacial acetic acid in 8 mL of deionized water. Cool the mixture in an ice bath and then, add this solution to the diazotized solution slowly with vigorous stirring and maintain the temperature between 2–5 °C. Allow the mixture to stand for 15 minutes at the temperature of 2–5 °C and separate the red orange form of the methyl orange. Then, make the resulting solution alkaline by dropwise adding 40 mL of 20% NaOH solution with continuous stirring. Heat the reaction mixture nearly to boil, add 15 g of sodium chloride, and stir it in hot condition until a clear solution is obtained. Cool the solution in an ice bath to separate the solid product, filter, and wash with ice-cold water. Recrystallize the solid product from hot water, filter, and then, dry in air.

**Results:**

Yield of the product, methyl orange = ..... g

Color = .....

## **Experiment: 6**

### **Determination of flash point of oils by Abel's apparatus.**

#### **Object:**

The flash point of oil may be defined as the minimum temperature to which it must be heated to give off sufficient vapour to form an inflammable mixture with air. This is deduced by the appearance of momentary flash upon the application of small flame over the surface of oil. The flash point is defined as closed or opens accordingly as the apparatus is provided with a cover over the oil cup or not.

This test is of immense importance for illuminating and lubricating oils. This helps in detecting the highly volatile at ordinary temperatures, the issuing vapours may cause the fire hazardous. So, to ensure safety, certain minimum temperatures are laid down for fuels and lubricating oils below which they should not give off adequate vapours to make them burn.

#### **Outline of the method:**

This method applies to oils having a flash point less than 49 °C.

The sample is placed in the oil cup of the apparatus and heated at a slow uniform rate. A small test flame is directed into the cup at regular intervals, and the lowest temperature at which application of the test flame causes the vapour above the sample to ignite, with a distinct flash inside the cup, is recorded as Abel's flash point.

#### **Apparatus:**

The Abel's apparatus is depicted in the sectional diagram; it consists of the following essential parts:

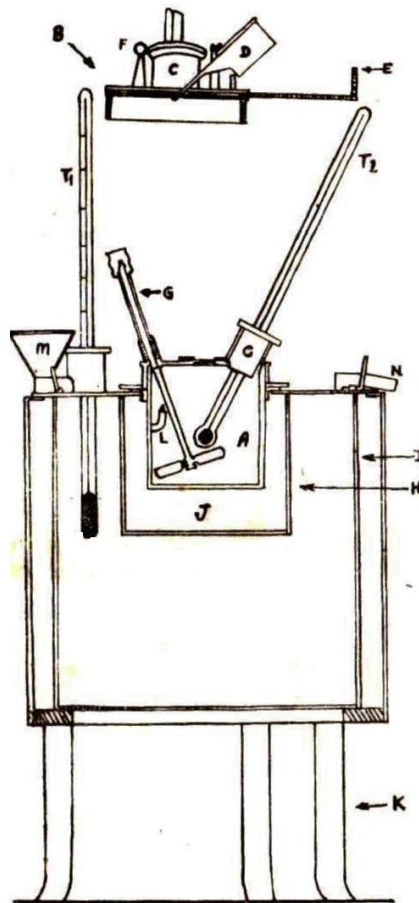
Oil cup (A) is a cylindrical vessel open at the top and fitted on the outside with a flat circular flange projecting at right angles. It is made of brass or gunmetal and within the cup, near the top, there is a level mark (L).

The cup is provided with a close-fitting cover (B). The cover is provided with a thermometer socket (C), turn ions to support an oil test lamp (D), a pair of guides in which a slide (E) moves, and a white bead (F). The top of the cover is pierced by three rectangular holes which are covered or uncovered by moving the slide which has two perforations allow air to enter the oil cup and bring contact between vapours of the oil and flame of the test- lamp when in 'open' position.

A white bead, the dimensions of which represent the size of test flame, is mounted in a visible position on the cover. In a modified apparatus the cover is also provided with a stirrer (G) consisting of a round stem having four blades at one end and projects into the oil cup.

The heating vessel consists of two flat bottomed cylindrical copper vessels (H) and (I) placed coaxially, one inside the other, and soldered at their tops. The space between the two vessels is thus enclosed and is used as a water jacket. When the oil cup is placed into the hole at the top of the vessel, it fits into it and leaves an air bath (J) and provides proper heating of the oil.

The vessel rests upon a tripod stand (K) and can be heated electrically or with a burner or spirit lamp. Two thermometers are provided with the apparatus, the one ( $T_1$ ) for measuring the temperature of the bath, the other ( $T_2$ ) for determining the flash point. The heating vessel is provided with a funnel (M) for introducing water outlet (N) tube.



### Procedure:

Following two methods may be used for the closed flash point of oils by Abel's apparatus method A is for oils flashing below 32 °C, and method B for oils which flash between 32 °C and 49 °C inclusive.

### Method A

The heating vessel is filled to overflowing with warm water and its temperature is adjusted to 55 °C at the beginning of the test. The test flame is adjusted to a bead approximately four mm in diameter and compared with the projecting white bead mounted on the cover, to maintain its size constant.

The temperature of the sample is adjusted to approximately 15 °C and filled in the already washed and dried oil cup until the liquid just reaches the level mark. The oil cup is fitted with the cover, with the slide closed, and placed in the heating vessel. The thermometers are so inserted that if cup is properly placed the thermometers scale faces the operator.

The test flame is set up and when the temperature of oil reaches 20 °C test-flame is applied, by slowly opening the slide after every 0.5 °C rise in temperature and closed immediately, until a distinct flash occurs in the interior of the cup. This temperature is recorded as flash point. The vessel is not heated during the test.

### Method B

The method is same as described above expect that in this case the air chamber is filled, to a depth of four cm with cold water and the heating vessel is filled with cold water. The vessel is heated at a rate which gives a temperature rise of 1 °C per minute. The testing in this case is started when the temperature has risen to 25 °C.

**Observation:** (Illustration)

#### Barometric pressure ..... mm Hg

S. No.	Temperature °C	Interferences
1	20	No flash
2	21	"
3	.....	"
4	....	"
..	....	"
..	.....	"
x	< 49	Flash observed

**Result:** The ‘closed’ Abel’s flash point of the oil is found to be ..... °C at barometric pressure ..... mm Hg.

## Experiment: 7

**Determination of flash point of lubricating oil by Pensky-Martens 'closed' tester.**

### **Object:**

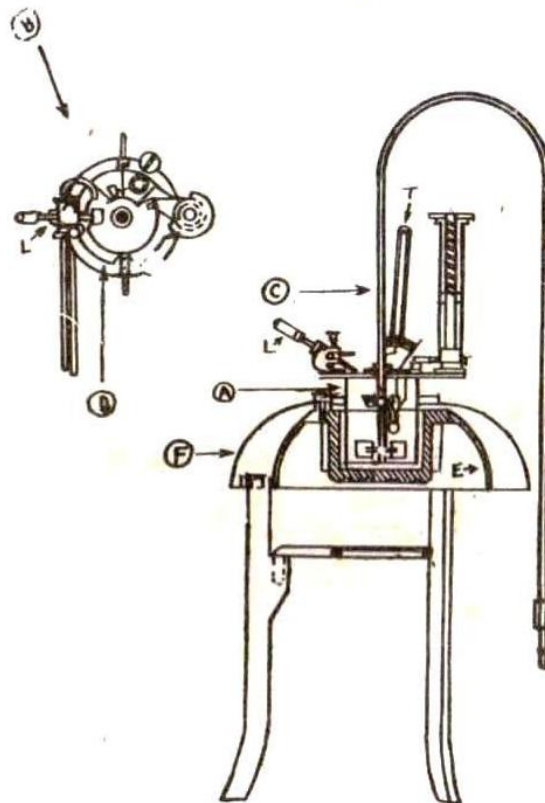
The flash point of oil may be defined as the minimum temperature to which it must be heated to give off sufficient vapour to form an inflammable mixture with air. This is deduced by the appearance of momentary flash upon the application of small flame over the surface of oil. The flash point is defined as closed or opens accordingly as the apparatus is provided with a cover over the oil cup or not.

This test is of immense importance for illuminating and lubricating oils. This helps in detecting the highly volatile at ordinary temperatures, the issuing vapours may cause the fire hazardous. So, to ensure safety, certain minimum temperatures are laid down for fuels and lubricating oils below which they should not give off adequate vapours to make them burn.

### **Apparatus:**

This apparatus is used for the oil flashing between 49 °C and 370 °C.

A Pensky-Martens apparatus consists of the following major parts:



Oil cup (A) is a cylindrical vessel, made of brass, with a filling mark grooved inside near the top. The inside of the cup is of slightly larger diameter above the filling mark and is covered with a lid.

Lid (B) is equipped with the following parts:

**Stirrer:** The stirring device consists of a vertical steel shaft (C) mounted in the centre of the cup and carrying two two-bladed brass propellers.

**Cover proper:** It is made up of brass having a rim projecting downward and fitting the outside of the cup closely. It has four openings: one for thermometer (T); and the rest for the oxygen entry and exposure of vapours to the test flame.

**Shutter:** The lid is equipped with a brass shutter (D) operating on the plane of the upper surface of the cover proper. The shutter is so shaped and mounted on the lid that when in one position, the holes are completely closed when in the other, these orifices are completely opened.

**The flame exposure device:** The lid is equipped with a pilot-lamp (L) 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.

The cup is heated by means of a properly stove which consists of an 'air bath' (E) and a top-plate (F). The air-bath has cylindrical interior about 4 cm deep and can be heated by a direct flame or an electric resistance element. The top-plate is also made of metal and mounted with an air gap between it and air bath.

### **Procedure:**

The cup and its accessories are well cleaned and dried before the test is started. 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. The cup is now set in the stove properly and the thermometer inserted, the test flame is lit and the heating rate is maintained, with the help of rheostat, at 5–6 °C per minute and stirring rate at 100 to 120 rpm.

Once the heating is started, the test flame is applied after each 2 °C rise of temperature up to a temperature of 105 °C. After that the test flame is applied after each 5 °C rise of temperature. The first application of the test flame is made at least 10 °C below the actual flash point, which can be determined in a rough experiment. The application of the test is made by operating the device controlling the shutters and test-flame burner so that the flame is lowered in 0.5 second, left in its lowered positions for one second, and quickly raised to its position.

The temperature at which the flame application causes a distinct flash in the interior of the cup is recorded as the flash point.

**Observation: (Illustration)**

**Barometric pressure ..... mm Hg**

S. No.	Temperature °C	Observations
1	90	No flash
2	91	"
3	92	"
4	93	"
5	94	"
6	95	"
7	96	Flash observed

**Result:** The Pensky-Martens 'closed' flash point of the given sample of oil comes out to be ..... °C at a pressure of..... mm Hg.

## Experiment: 8

**To study the viscosity of the given sample of lubricating oil with a Redwood viscometer and to study the viscosity at various temperatures.**

### **Object:**

Viscosity is one of the most important properties of a lubricant which tells about its suitability for lubricating purpose. A lubricant reduces friction between different moving parts of a machine by avoiding direct metal to metal contact. The thin film of the oil, formed between the moving surfaces, keeps the apart and thus the frictional resistance is entirely on account of the shearing of the liquid. The viscosity of the fluid measures the amount of this internal friction.

The object of this determination, therefore, is to ascertain whether the oil is sufficiently viscous, under the higher pressure and temperature of the machine, to adhere to the bearing. The variation of viscosity with temperature is of great importance to the engineer in all problems of fluid flow and fluid friction and also in pumps, fans and all types of bearings.

### **Theory:**

Viscosity is the property of a homogeneous fluid which causes it to offer frictional resistance to motion. The property may be measured in several ways, one of which is determining the time required for the liquid to flow through a capillary. Such method includes the use Saybolt, Engler and Redwood viscometer.

The viscosity may be expressed as dynamic (or absolute) viscosity, kinematic viscosity or the viscosity called after the name of the apparatus used for its determination.

### **Dynamic viscosity:**

Dynamic viscosity is the tangential force on unit area of either of the two parallel planes, at unit distance apart, when the space between the planes is filled with the fluid and one of the planes moves relatively to the other with unit velocity in its own plane. It is defined as the ratio of the shearing stress to the rate of shearing strain. If  $F$  is the force required to keep moving an article, of surface area  $A$  in contact with the fluid, separated from stationary surface by a thickness  $d$ , with a velocity  $V$ , then,

$$\text{Shearing stress} = F/A; \text{Rate of shearing strain} = V/d$$

$$\text{Therefore, absolute viscosity, } \mu = \frac{Fd}{AV} \quad \dots\dots\dots (1)$$



The numerical value of the  $\mu$  depends upon the units used in Eq. (1). In metric system the unit is poise ( $F = 1$  dyne,  $A = 1$  sq. cm,  $V = 1$  cm per second). A smaller unit, centipoises is more often used. Absolute viscosity is also referred to as “coefficient of viscosity”.

**Kinematic viscosity:**

This is absolute viscosity divided by the density of the fluid. The metric units are stoke and centistokes. Thus, if  $\rho$  is the density then,

$$\text{Kinematic viscosity } \nu = \frac{\mu}{\rho} \text{ Stokes} \dots\dots\dots (2)$$

Since the rate, at which a fluid will flow through an aperture, increases as the internal friction of the fluid decreases, the rate of flow through an orifice or short tube may be used as a means for measuring viscosity. This is the principle involved in the Redwood viscometer which is an English standard and is one of the several short tube viscometers used for oils.

The time in seconds required for 50 mL of oil to gravitate through the Redwood viscometer at a given temperature is expressed as its viscosity in Redwood seconds at the temperature.

Absolute and Kinematic viscosities can also be determined from the Redwood values by means of following formula:

$$\text{Absolute viscosity} = (At - B/t) \times \rho \dots\dots\dots (3)$$

$$\text{Kinematic viscosities} = \frac{\mu}{\rho} = At - B/t \dots\dots\dots (4)$$

where A and B are constants known as instrument factors, t is Redwood viscosity, and  $\rho$  is the density of fluid. The values of A and B for Redwood viscometer No. 1 are as follows:

Viscosity range in R. I. second	A	B
34–100	1.79	0.00260
Above 100	0.59	0.00247

**Apparatus:**

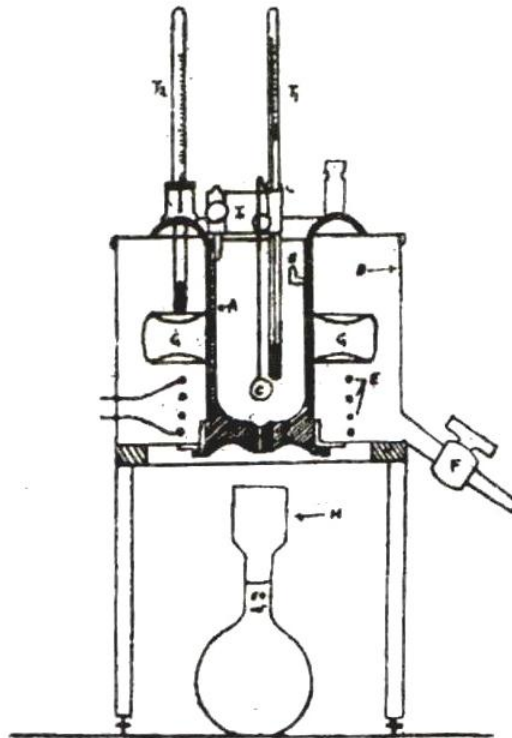
The Redwood viscometer is available in two types, no. I and II. Both are suitable for use with oils having flow time, at the temperature of the test, of not less than 30 seconds. When the flow time with no. I apparatus exceed 2000 seconds the use is made of no. II viscometer. The only main difference between the two viscometers is in the diameter of the discharge jets, the no. I jet having a diameter of 1062 mm and length of 10 mm, while the no. II jet is 3.88 mm in diameter and 50 mm in length.

### Viscometers:

The viscometer consists of an oil cup (A) which is a cylindrical vessel made of brass. The upper end of the cup is open while the bottom of the cup is concave internally, to allow complete drainage of the liquid with which it may be fitted. The base of the cup has a tapering central hole in which the jet (J) is fitted. A stout wire, turned upwards at right angles, is fixed into the side of the oil cup to indicate the level (B) to which the oil is to be filled.

The jet, constructed of agate, is provided with a concave depression which can be covered with a ball valve (C) for starting or stopping the flow of oil.

The cup is surrounded with a cylindrical heating bath, the contents of which can be heated by an electrically heated coil (E) placed in it. The bath is provided with a tap (F) for emptying. Stirring of the bath is affected by means of a cylinder surrounding the oil cup, provided with four vanes (G) the upper and lower portions of which are turned in opposite directions.



The apparatus is elevated on legs or tripod stands so that a receiving flask (H) can be placed beneath the jet to catch and measure the oil flowing from the tube. The flask is of special type, graduated with a 50 mL mark at the neck and with a flaring mouth. The tripod stand has levelling screws at the bottom and the thermometer ( $T_1$ ) is suspended in the oil by means of a spring clip (I) sliding on the vertical rod fixed on the top of the cup. The cover of the tin cup has provisions

for the insertion of thermometer and the ball-valve projects outside the cover through a hole. Another thermometer ( $T_2$ ) measures the temperature of the bath.

**Procedure:**

The apparatus is prepared first by cleaning it and then levelling by the screws provided for this purpose.

When marking the viscosity determinations, the bath is heated to a few degrees above the desired test temperature. The test sample is adjusted until the sample in the cup is at the test temperature, stirring the contents of the bath the cup during this process. When the temperature of the sample has become quite steady at the desired value, the liquid level is adjusted by allowing the sample to flow, until the surface of the sample touches the filling mark. The oil cup is covered and the thermometers inserted. The clean, dry, standard 50 mL flask is placed centrally below the jet properly. The ball value is lifted and the stopwatch is started simultaneously. The valve is not removed completely, but kept immersed in the oil by supporting it by the hook provided for the purpose. The receiving flask is positioned so that the oil strikes the flared mouth and does not drop directly into the opening which would cause foaming. The stopwatch is stopped when the level of the oil reaches 50 mL mark in the neck of the flask.

The time elapsed in seconds is recorded as Redwood viscosity at the test temperature, observed before the flow was started.

Following this procedure, the viscosities are determined at one or more of the following temperatures: 70 °F (21.1 °C); 100 °F (37.8 °C); 122 °F (50 °C); 140 °F (60 °C); 200 °F (121 °C); 300 °F (149 °C); and 400 °F (204 °C).

**Table 1:**

S. No.	Temperature (°C)	Kinematic viscosity
1.		
2.		
3.		
..		
n		

Calculate the kinematic viscosities at different temperatures using equation (4) and plot a graph of kinematic viscosity versus temperature.

**Result:** .....

## Experiment: 9

**To find out the aniline point of the given sample of fuel.**

### **Object:**

The approximate method of testing the ignition quality of oils is based on measuring the proportion of paraffin hydrocarbons in the fuel. In the case of petroleum fuel oils, the high paraffin content to better ignition quality.

The aromatic hydrocarbons mix readily with aniline at room temperature but paraffins mix only at relatively higher temperatures. A high aniline point indicates a high paraffin content and higher ignition quality.

Moreover, aromatic hydrocarbons dissolve natural rubber and some synthetic rubbers also. This means a higher aromatic content in a lubricating oil may not be desirable as otherwise it may damage the oil sealing or packing which are usually made of rubber and used in the machines.

### **Definition:**

The aniline point of an oil is the lowest temperature at which the oil is completely miscible with an equal volume of aniline.

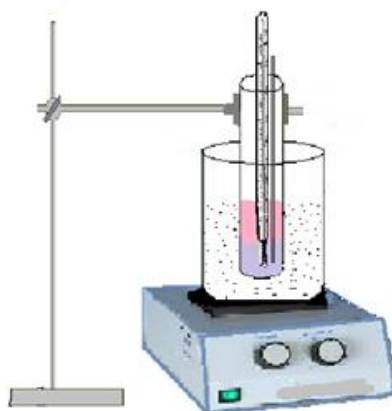
### **Procedure:**

Equal volume of aniline and oil are heated to effect complete dissolution and then cooled under controlled conditions. The temperature at which the entire mixture becomes cloudy throughout is reported as the aniline-point of the lubricating oil.

The apparatus used for the determination of aniline-point of a lubricating oil is shown in given fig.



(A)



(B)

The apparatus is thoroughly cleaned and dried at 110 °C before starting the experiment. About 10 mL of pure and dry aniline (dried over pure KOH pellets, filtered and freshly distilled) are taken in a heat resistant pyrex or corning test tube (2.5 cm – 15 cm) and an equal volume of the lubricating oil is added. The tube is fitted with a cork which also holds an electrically operated stirrer and a thermometer in such a way that the bulb of the thermometer is about 5 mm above the bottom of the test tube. The tube is inserted into an outer jacket (4 cm-17.5 cm). The stirrer is started and the aniline-oil mixture is observed to find whether the miscibility is complete at room temperature itself. If so, the jacket holding the tube is immersed in a non-aqueous cooling bath. Cooling is continued slowly at a rate of 0.5 to 1 °C per minute, until the entire aniline-oil mixture suddenly becomes cloudy throughout. This temperature is recorded as the aniline-point of the lubricating oil.

If the aniline-lubricating oil mixture is not completely miscible in the tube at the room temperature, then slowly and carefully heat the jacket, holding the tube either directly or by immersing in a hot bath. Stirring is continued while slowly raising the temperature, until the aniline-oil mixture is completely miscible. Then the jacket holding the tube is withdrawn from the burner or the hot bath, as the case may be. Now, while the stirring is continued, the temperature is allowed to fall at a rate of 1<sup>0</sup>C per minute. A cold bath may be used for this purpose, if necessary. The temperature at which the entire aniline-oil mixture becomes cloudy or hazy throughout and the outline of the thermometer bulb is observed is taken as the aniline-point of the oil sample.

**Result:** .....

### **Precautions**

1. The whole apparatus should be perfectly dry, because even traces of moisture may give erroneously high results.
2. Aniline used should be perfectly pure and dry.
3. Aniline is hygroscopic and hence water should not be used even in cold and hot baths. Non-aqueous, non-volatile and transparent liquids should be used for the cold and hot baths.
4. Aniline is highly toxic and hence it should not be sucked into the pipette by mouth. Pipette provided with a rubber suction bulb or an aspirator should be used for this purpose.
5. Stirring should be done at such a rate that undue splashing of the liquid or formations of air bubbles are avoided.
6. If the expected aniline-point is below the dew point at the atmosphere, the space above the aniline-oil mixture in the tube should be filled with dry N<sub>2</sub>.
7. The true aniline point is characterised by a turbidity which increases sharply as the temperature is lowered. The minimum equilibrium solution temperature, i.e. the temperature at which the entire aniline-oil mixture suddenly becomes cloudy throughout is taken as the aniline-point.

## Experiment: 10

**Determination of viscosity average molecular weight of a polymer sample by Viscometer.**

**Chemicals and glassware:** Starch, 100 ml volumetric flask, 100 mL beaker, pipette, Ostwald viscometer, and stopwatch.

### **Theory:**

Addition of even a very small amount of a polymer to a solvent of low viscosity causes a sharp increase in its viscosity. The magnitude of increase in viscosity depends in addition, to other factors such as concentration, shape and size of the solute molecules, on the molecular weight of the polymers.

The ratio of the coefficient of viscosity of the solution ( $\eta_s$ ) to the coefficient of viscosity of the pure solvent ( $\eta_o$ ) at the same temperature is known as viscosity ratio or relative viscosity ( $\eta_r$ ) which can be written as:

$$\eta_r = \frac{\eta_s}{\eta_o} = \frac{\rho_s t_s}{\rho_o t_o}$$

where,  $\rho_s$  and  $\rho_o$  are respective densities of polymer solution and pure solvent;  $t_s$  and  $t_o$  are the corresponding efflux time. For a dilute polymer solution ( $\approx 0.5$  g/ 100 mL) the two densities can be taken as equal. Then the equation becomes:

$$\eta_r = \frac{t_s}{t_o}$$

$$\eta_{sp} (\text{specific}) = \frac{\eta_s - \eta_o}{\eta_o} = \frac{\eta_s}{\eta_o} - 1 = \eta_r - 1 = \frac{t_s}{t_o} - 1$$

The ratio of the specific viscosity of the solution to its concentration C, is called viscosity number or reduced specific viscosity.

$$\eta_{red} = \frac{\eta_{sp}}{C}$$

C = Concentration in g/ 100 mL

The plot of  $\eta_{red}$  versus C is a straight line is extrapolated to zero conc. (i.e., cut into the Y-axis,  $\eta_{red}$ ). The intercept on the ordinate is the viscosity at zero conc. or the limiting viscosity and termed as limiting viscosity number or intrinsic viscosity,  $[\eta]$ , which is related to molecular weight  $\overline{M}_v$ , by an empirical equation.

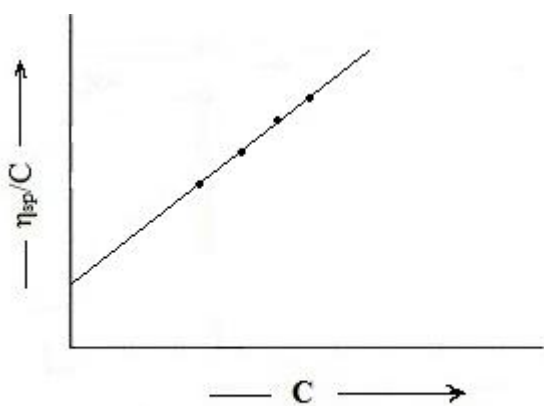
$$[\eta] = K \overline{Mv}^\alpha$$

where 'K' and 'α' are constants for a particular polymer/ solvent/ temperature system and can be obtained from the books.

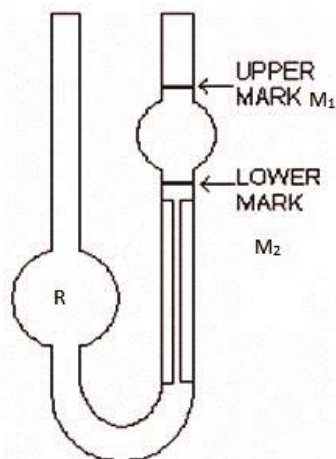
### Procedure:

**Preparation of polymer solution:** Weigh accurately 500 to 100 mg of the well dried powdered polymer (whatever concentration you need to prepare) and transfer to a 100 mL volumetric flask. Add 80–90 mL of the solvent (in which polymer is soluble) stopper the flask and suspend in a thermostatic water bath at 25 °C, shake occasionally to dissolve the sample and then fill the solvent up to the mark.

**Measurement of flow time with Ostwald Viscometer:** Wash the viscometer with chromic-sulphuric acid mixture and keep for drying in air oven. Pipette 20 mL of the solvent into the wide arm (reservoir R) and affix in a thermostatic water bath. Allow 5–10 minutes for attainment of temperature. Using a rubber ball, force air into the wide arm so as to raise the level of liquid into the capillary arm above the higher mark  $M_1$ . Then release the pressure and with a timer measure the time in which the liquid meniscus moves from the upper mark  $M_1$  to the lower mark  $M_2$  and use the flow time as  $t_0$ .



Plot for calculation of  $[\eta]$



Ostwald Viscometer

Pour out the liquid from the wide arm as completely as possible. Clean the viscometer, dry it and fill it with 20–25 mL of the polymer solution and determine the flow time in the similar condition ( $t_s$ ). Similarly determine the flow times for solutions of minimum three different concentrations.





# Experiment: 11

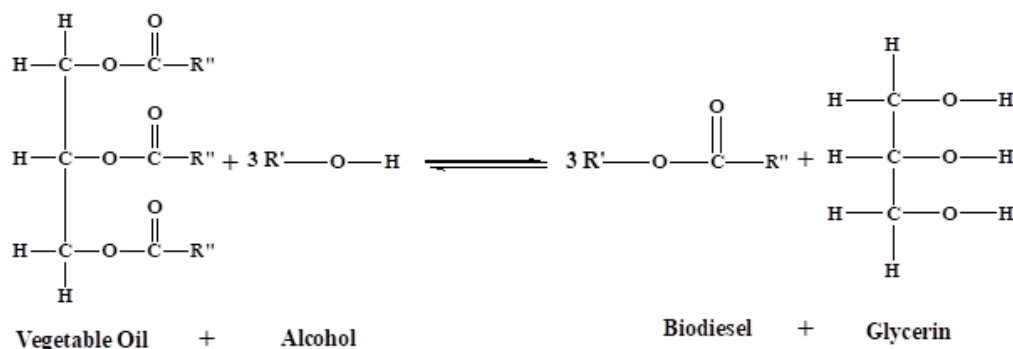
## Preparation and characterization of biodiesel from waste cooking oils.

**Chemicals and glassware:** Methanol, KOH, vegetable oil, conical flask, and separating funnel.

### Introduction:

Biodiesel is a renewable, biodegradable fuel manufactured domestically from vegetable oils, or animal fats. Due to its biodegradable nature, it is used as a replacement for fossil diesel fuel. It can also be mixed with petroleum diesel fuel in any proportion. The production of biodiesel involves a chemical reaction, which is known as transesterification. It is the conversion of one ester into another one by exchanging the alkoxy (–OR) group under basic or acidic conditions. In biodiesel synthesis, vegetable oil or animal fat reacts with alcohol in the presence of a base catalyst to yield an ester and glycerol (glycerin).

### Reaction:



Where R'' = fatty chain of the triglyceride (this will vary for each type of vegetable oil)  
R' = in the case of methanol, this will be methyl group (CH<sub>3</sub>)

### Procedure:

Pour 20 mL of methanol into a conical flask and mix with 0.85 g of crushed KOH pellets for 10 min. Add 100 mL of vegetable oil to the conical flask and heat at 50 °C for 25 min with continuous stirring. Transfer the resultant products into a separating funnel and keep it for two weeks. After two weeks, discard the bottom layer containing glycerol and collect the top layer containing biodiesel. Measure the flash point of prepared biodiesel using Pensky-Martens 'closed' tester.

- Results:**
1. Yield of the product, biodiesel = .....
  2. Flash point = ..... °C
  3. Color = .....