

*Laboratory Manuals*  
*for*  
*Undergraduate Students*



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## **List of experiments**

1. Proximate Analysis of Coal
2. Determination of strength of two acids in a mixture by conductometric titration
3. Estimation of Fe (II) in a given Solution
4. Estimation of Cu (II) in a given Solution
5. Estimation of Hardness of Water
6. Synthesis and Characterization of Tris(acetylacetonato) Manganese(III)
7. Aldol Condensation Reaction
8. Nitration Reaction
9. Diazotisation Reaction
10. Alkaline hydrolysis of Methyl benzoate to Benzoic acid

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## ***Experiment: 01***

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### **Aim:**

To determine moisture, volatile matter and ash content of a given sample of coal

### **Apparatus:**

Silica crucible with vented lid, electric oven, Muffle furnace, spatula, desiccator, pair of tongs, weighing balance, long legged tongs, etc.

### **Chemicals:**

Powdered coal sample

### **Theory:**

Coal is a primary, solid, fossil fuel. Coal sample has to be analyzed before using it in any field/industry to find out its quality and suitability. Moisture, volatile matter and ash content of coal are determined under proximate analysis. This method is simple and quick and is used primarily to determine the suitability of coal for cooking, power generation or for iron ore smelting in the manufacture of steel.

**Moisture:** Moisture held within the coal itself is known as inherent moisture and is analyzed. Moisture reduces the calorific value of coal and considerable amount of heat is wasted in evaporating it during combustion. Moisture content should be as low as possible.

**Volatile matter:** Volatile matter is usually a mixture of short and long chain hydrocarbons, aromatic hydrocarbons and some sulphur. Volatile matter of the coal is related to the length of the flame, smoke formation and ignition characteristics. High volatile matter coal gives long flame, high smoke and relatively low heating values. Volatile matter content should be low but minimum 20% is required for the ignition of coal.

**Ash:** Ash content of coal is the non-combustible residue left after coal is burnt. It consists of inorganic matter like silica, alumina, iron oxide, lime, magnesia etc. Ash reduces the heating value of coal, reduces air supply in furnaces and also requires labour (extra cost) for its regular disposal. Therefore, ash content of coal should be as low as possible.

**Fixed carbon:** The fixed carbon content of the coal is the carbon found in material which is left after volatile materials are driven off. More the fixed carbon content, higher will be the calorific value of coal.

## **Procedure:**

### ***A. Determination of Inherent Moisture***

Transfer about 1g (known quantity) of powdered air-dried coal sample into a previously weighed silica crucible. Place the open crucible with sample in an electric oven and heat it at about 105-110°C for an hour. Take out the crucible after one hour from the oven and cool it in a desiccator. Then weigh the crucible with sample and repeat the process of heating, cooling and weighing till constant weights obtained. Calculate the loss in weight.

### ***B. Determination of Volatile matter***

The dried sample of coal after determining moisture content is closed with a vented lid. The closed crucible is then heated in a Muffle furnace-maintained  $T\ 925 \pm 20^\circ\text{C}$  for exactly 7 minutes. The crucible is taken out from Muffle furnace carefully with the help of long-legged tongs. It is first cooled in air and then in desiccator. When the crucible attains room temperature it is weighed. Calculate the loss in weight.

### ***C. Determination of Ash***

Transfer about 1g (known quantity) of powdered air-dried coal sample into a previously weighed silica crucible. Place the open crucible with sample in Muffle furnace maintained at  $725 \pm 25^\circ\text{C}$  for about 40 minutes or till constant weight is obtained. Coal burns in open and the residue left is ash. Take out the crucible from muffle furnace carefully using long legged tongs. Cool the hot crucible first in air and then in desiccator. Weigh the crucible and find out the amount of unburnt residue left (ash).

### ***D. Determination of fixed carbon***

The percentage of fixed carbon is determined indirectly by subtracting the sum total percentages of moisture, volatile matter and ash from 100.

## **Observations and calculations:**

### ***A. For moisture***

- 1) Weight of empty crucible ( $W_1$ ) = \_\_\_\_\_g.
- 2) Weight of crucible + Coal sample ( $W_2$ ) = \_\_\_\_\_g.
- 3) Weight of coal sample before heating ( $W_3$ ) =  $W_2 - W_1$  = \_\_\_\_\_g.

- 4) Weight of crucible + Sample after heating for 1h at 105-110 °C ( $W_4$ ) = \_\_\_\_\_ g.
- 5) Weight of coal sample after heating ( $W_5$ ) =  $W_4 - W_1$  = \_\_\_\_\_ g.
- 6) Loss in weight of sample due to moisture ( $W_M$ ) =  $W_3 - W_5$  OR  $W_2 - W_4$  = \_\_\_\_\_ g (where  $W_M = v$ )
- 7) % Of moisture in coal =  $\frac{\text{Weight of moisture}}{\text{weight of coal before heating (W3)}} = \text{_____ \%}$

### ***B. For Volatile matter***

- 1) Weight of empty crucible ( $W_1$ ) ( $W_1$  from part A) = \_\_\_\_\_ g.
- 2) Weight of crucible + moisture free coal sample ( $W_2$ ) ( $W_4$  from part A) = \_\_\_\_\_ g.
- 3) Weight of moisture free sample (before heating) ( $W_3$ ) ( $W_5$  from part A) =  $W_2 - W_1$  = g.
- 4) Weight of crucible + Sample after heating for 7 mins. at  $925 \pm 20^\circ \text{C}$  ( $W_4$ ) = \_\_\_\_\_ g.
- 5) Weight of Sample after heating ( $W_5$ ) =  $W_4 - W_1$  = \_\_\_\_\_ g.
- 6) Loss in weight of sample due to volatile matter ( $W_{VM}$ ) =  $W_3 - W_5$  = \_\_\_\_\_ g.
- 7) % Of volatile matter in coal =  $\frac{\text{Weight of volatile matter}}{\text{Weight of coal (before heating) } W_3} \times 100 = \text{_____ \%}$

### ***C. For Ash***

- 1) Weight of empty crucible ( $W_1$ ) = \_\_\_\_\_ g.
- 2) Weight of crucible + Coal sample ( $W_2$ ) ( $W_4$  from part B) = \_\_\_\_\_ g.
- 3) Weight of Coal sample (before heating) ( $W_3$ ) =  $W_2 - W_1$  = \_\_\_\_\_ g.
- 4) Weight of crucible + Sample after heating for 40 mins. at  $725 \pm 25^\circ \text{C}$  ( $W_4$ ) = \_\_\_\_\_ g.
- 5) Weight of Ash formed ( $W_A$ ) =  $W_4 - W_1$  = \_\_\_\_\_ g.
- 6) % Of Ash in coal =  $\frac{\text{Weight of Ash}}{\text{Weight of coal (before heating) } W_3} \times 100 = \text{_____ \%}$

### ***D. For Fixed carbon (FC)***

% of Fixed Carbon =  $100 - (\% \text{ Moisture} + \% \text{ Volatile matter} + \% \text{ Ash}) = \text{_____ \%}$

**Results:**

The coal sample contains:      Moisture = \_\_\_\_\_ %  
   Volatile matter = \_\_\_\_\_ %  
   Ash = \_\_\_\_\_ %  
   Fixed Carbon = \_\_\_\_\_ %

**Conclusion:**

Quality of coal is good /Poor.

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## ***Experiment: 02***

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### **Aim:**

To determine the concentration of HCl (strong acid) and CH<sub>3</sub>COOH (weak acid) in a mixture by conductometric titration with NaOH.

### **Chemicals Required:**

Oxalic acid, HCl, NaOH, acetic acid, Phenolphthalein indicator.

### **Glassware and apparatus:**

(a) Instrument: Conductometer (b) Apparatus: Burette, pipette, conical flask, 100 mL volumetric flask, beaker, stand & clamp

### **Theory:**

The specific conductance,  $\kappa$ , of an electrolyte solution may be expressed as

$$\kappa = \text{constant} \times \sum_i c_i u_i \quad \dots(1)$$

where,  $c_i$  is the concentration of  $i$ -th ion (in g-ion L<sup>-1</sup>) and  $u_i$  is the corresponding ionic mobility. The constant includes absolute values of charges. Thus for a solution of HCl in water,

$$\kappa = \text{constant} [c_{H^+} \cdot u_{H^+} + c_{Cl^-} \cdot u_{Cl^-}] \quad \dots(2)$$

because water is practically un-ionized. Now, since  $c_{H^+} = c_{Cl^-} = c_{HCl}$

$$\kappa = \text{constant} \cdot c_{HCl} [u_{H^+} + u_{Cl^-}] \quad \dots(3)$$

As strong acids are completely ionized, the initial conductance of a mixture of HCl (strong acid) and CH<sub>3</sub>COOH (weak acid) will be entirely due to the strong acid, since the ionization of the weak acid will be suppressed by the common ion effect of H<sup>+</sup> ions. As the mobilities of ions are in the following order: H<sup>+</sup> > OH<sup>-</sup> > Na<sup>+</sup> > CH<sub>3</sub>COO<sup>-</sup>, in the absence of any base the specific conductance of a mixture of HCl and CH<sub>3</sub>COOH will be given by Eqn. (3).

When small amount (say  $x$  mol) of strong base (NaOH) is added, highly conducting H<sup>+</sup> ions are neutralized by OH<sup>-</sup> (to form unionized H<sub>2</sub>O) and replaced by Na<sup>+</sup> ions having much lower conductivity. Specific conductivity of such mixture of strong acid (HCl), its salt NaCl and weak acid (CH<sub>3</sub>COOH) will be given by

$$\begin{aligned} \kappa' &= \text{constant} [(c_{HCl} - x) \cdot u_{H^+} + c_{HCl} \cdot u_{Cl^-} + x \cdot u_{Na^+}] \\ &= \text{constant} [c_{HCl}(u_{H^+} + u_{Cl^-}) - x \cdot (u_{H^+} - u_{Na^+})] \end{aligned} \quad \dots(4)$$

$$\therefore \kappa' = \kappa - \text{constant} \cdot x(u_{H^+} - u_{Na^+}) \quad \dots(5)$$

Since  $u_{H^+} \gg u_{Na^+}$ , so  $(u_{H^+} - u_{Na^+})$  will be positive. Therefore the plot of  $\kappa'$  vs  $x$  will be a straight line (AB) with negative slope (Figure 1).

When neutralization of the strong acid is just complete, conductance of the mixture is at its minimum ( $\kappa_B$ ) and on further addition of NaOH, neutralization of the weak acid starts, producing the fully ionized salt, sodium acetate,  $CH_3COO^-Na^+$  and unionized  $H_2O$  molecules as before:



If  $y$  mol of the strong base (NaOH) is added after complete neutralization of HCl, then the specific conductance,  $\kappa'$ , will be given by

$$\kappa' = \kappa_B + \text{constant} \cdot y(u_{Na^+} + u_{CH_3COO^-}) \quad \dots(7)$$

which indicates the plot of  $\kappa'$  vs moles of NaOH ( $y$ ) will be a straight line (BC) with positive slope (Figure 1).

Due to the common ion effect by the  $CH_3COO^-$  ion the ionization equilibrium ( $CH_3COOH \rightleftharpoons CH_3COO^- + Na^+$ ) of acetic acid is repressed to some extent, and as a result, the conductance of the solution falls slightly, but soon increases because, the conductivity of the highly ionized salt,  $CH_3COO^-Na^+$ , exceeds that of the weak acid ( $CH_3COOH$ ) which it replaces.

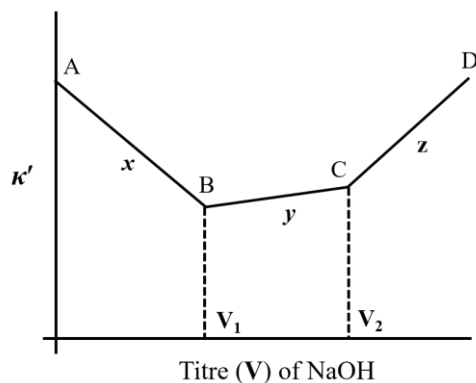
Immediately after the equivalence point, C, conductance ( $\kappa_C$ ) of the solution shows a steep rise (Figure 1), as the conductivity of  $OH^-$  ions from the excess NaOH is much higher than that of the acetate ( $CH_3COO^-$ ) ions. If  $z$  mol of the base is added after complete neutralization of the two acids, HCl and  $CH_3COOH$ , the solution will contain  $Na^+$ ,  $CH_3COO^-$ ,  $Cl^-$  and  $OH^-$  ions. Specific conductance ( $\kappa'$ ) of such a solution may be expressed as:

$$\kappa' = \kappa_C + \text{constant} \cdot z(u_{Na^+} + u_{OH^-}) \quad \dots(8)$$

which indicates the plot of  $\kappa'$  vs moles of NaOH ( $z$ ) will be a straight line (CD) with positive slope (Figure 1). Since  $u_{OH^-} \gg u_{CH_3COO^-}$ , the slope of the line CD will be higher than that of BC. Volume change during the titration will be almost negligible if the concentration of the titrant (NaOH) is 10 times as the concentration of the acids. Under this condition the plot of specific conductance ( $\kappa'$ ) vs volume ( $V$ ) of the titrant (NaOH) for a particular neutralization will be a straight line. The resulting plot will consist of straight line mutually intersecting at the equivalence points.



If the titre  $V_1$  of NaOH corresponds to the first neutralization point, i.e., of the strong acid (HCl) and the titre  $V_2$  corresponds to the second neutralization point, i.e., of the total quantity of strong and weak acid, then  $(V_2 - V_1)$  ml of NaOH is required to neutralize the weak acid ( $\text{CH}_3\text{COOH}$ ) only.



**Figure 1.** Conductometric Titration Curve

### **Procedure:**

1. Prepare  $\sim N/2$  NaOH solution of 100 ml volume.
2. Take 10 ml of mixed acid solution (total strength  $\sim N/10$ ) in 100 ml beaker and dip the conductivity cell into the solution and add just sufficient amount of distilled water to cover the electrodes of the cell with solution.
3. Stir the solution and measure the conductance.
4. Add 2-4 drops of the  $N/2$  NaOH solution from a 10 ml burette, stir thoroughly and then measure the conductance. Continue the process and each time record the number of drops of NaOH added and the conductance of the resulting solution in tabular form.
5. Plot the conductance against number of drops of NaOH added and draw the best straight lines through the experimental points and find the points of intersection.
6. Count the number of drops of alkali that is equivalent to 1 ml of NaOH solution. Using this calculate the volume of NaOH ( $V_1$  and  $V_2$ ) required for the neutralization of HCl and ( $\text{HCl} + \text{CH}_3\text{COOH}$ ) mixture.
7. Finally calculate the concentrations of the HCl and  $\text{CH}_3\text{COOH}$ .

## **Results:**

**Table 1. Conductometric titration**

Temperature of the experimental solution =

Strength of NaOH =

Volume of unknown acid solution = 10 ml

No. Of Obs.	No. of drops of NaOH solution added	Conductance

....etc

## **Calculation:**

Plot the conductance against number of drops of NaOH added and draw the best straight lines through the experimental points and find the points of intersection.

Let the no of drops per ml of NaOH sol<sup>n</sup> added = **n**. Then the titre value =  $1/n$ .

So, volume of NaOH solution for 1st neutralization pt.,  $V_1 = 1/n \times \text{no of drops at 1st intersection pt.}$  Also at neutralization point, number of moles of HCl = number of moles of NaOH.

So, using the law of chemical equivalence, i.e.,  $N_{\text{HCl}} V_{\text{HCl}} = N_{\text{NaOH}} V_1$ ,

strength of HCl in mixture ( $N_{\text{HCl}}$ ) = ----- N.

Similarly, volume of NaOH solution for 2nd neutralization pt.,  $V_2 = 1/n \times \text{no of drops at 2nd intersection pt.}$

Since, both acids are fully neutralized at 2nd nutralization pt, using the law of chemical equivalence,  $N_{\text{CH}_3\text{COOH}} V_{\text{CH}_3\text{COOH}} = N_{\text{NaOH}} (V_2 - V_1)$

strength of  $\text{CH}_3\text{COOH}$  in mixture ( $N_{\text{CH}_3\text{COOH}}$ ) = ----- N.

$\therefore$  Concentration of HCl in the mixture = ----- moles/L.

$\therefore$  Concentration of Acetic acid in the mixture = ----- moles/L.

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## *Experiment: 03*

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### **Aim:**

To determine the amount of Fe(II) in a given solution

### **Requirements:**

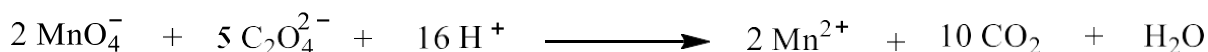
(a) **Apparatus:** Burette, pipette, conical flask, stand

(b) **Chemicals:** Fe(II) solution,  $\text{KMnO}_4$ , Oxalic acid,  $\text{H}_2\text{SO}_4$

### **Theory:**

$\text{KMnO}_4$  is a secondary standard since it contains traces of  $\text{MnO}_2$  as impurity; therefore, a solution of approximate normality is obtained when a definite weight of it is dissolved in definite volume of water. It is an oxidising agent and the exact strength of it can be calculated by titrating against oxalic acid solution of definite strength.

#### **Reaction involved:**

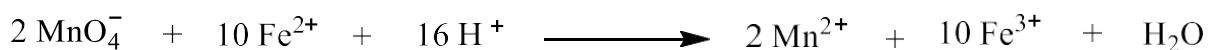


**Indicator:**  $\text{KMnO}_4$  acts as self-indicator.

**End Point:** Colourless to light pink

By knowing the strength of  $\text{KMnO}_4$ , the strength of Fe(II) solution can be calculated by redox titration between Fe(II) solution and  $\text{KMnO}_4$ .

#### **Reaction involved:**



**Indicator:**  $\text{KMnO}_4$  acts as self-indicator.

**End Point:** Colourless to light pink

### **Procedure:**

(a) **Standardization of  $\text{KMnO}_4$  by oxalic acid:** Fill the burette with  $\text{KMnO}_4$  solution. Pipette out 5 ml (N/20) oxalic acid solution in a conical flask. Add 1/3 test tube of dil.  $\text{H}_2\text{SO}_4$  to it and gently heat the solution to about 60- 80°C. Then titrate this heated mixture against  $\text{KMnO}_4$  to get the end point. Repeat and get the concordant reading.

**(b) Standardization of Fe(II) solution by  $\text{KMnO}_4$ :** Pipette out 5 ml Fe(II) solution in a conical flask. Add 1/3 test tube of dil.  $\text{H}_2\text{SO}_4$  and 4-5 drops of  $\text{H}_3\text{PO}_4$  to it and titrate (at room temperature) against  $\text{KMnO}_4$  taken in a burette to get the end point. Repeat and get the concordant reading.

### **Observation:**

**Table 1 for procedure (a)**

No. Of Obs.	Vol. Of oxalic acid(ml)	Burette Reading			Concordant Reading	Vol. Of $\text{KMnO}_4$ (ml)
		Initial	Final	Difference		

**Table 2 for procedure (b)**

No. Of Obs.	Vol. Of Fe (II) sol <sup>n</sup> (ml)	Burette Reading			Concordant Reading	Vol. Of $\text{KMnO}_4$ (ml)
		Initial	Final	Difference		

### **Calculation:**

From Table 1:  $N_1V_1$  of oxalic acid =  $N_2V_2$  of  $\text{KMnO}_4$

$$N_2 = \dots\dots\dots$$

From Table 2:  $N_1V_1$  of Fe(II) sol<sup>n</sup> =  $N_2V_2$  of  $\text{KMnO}_4$

$$N_1 = \dots\dots\dots$$

**Result:** Strength of Fe(II) solution =  $N_1 \times 56 \text{ g/L} = \dots\dots\dots \text{g/L}$

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## ***Experiment: 04***

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**Aim:** To determine the amount of Cu (II) in a given solution.

**Requirements:**

(a) **Apparatus:** Burette, pipette, conical flask, stand.

(b) **Chemicals:** Cu (II) solution, Na<sub>2</sub>CO<sub>3</sub> solution, Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution, dil. acetic acid, KI solution, starch solution, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution.

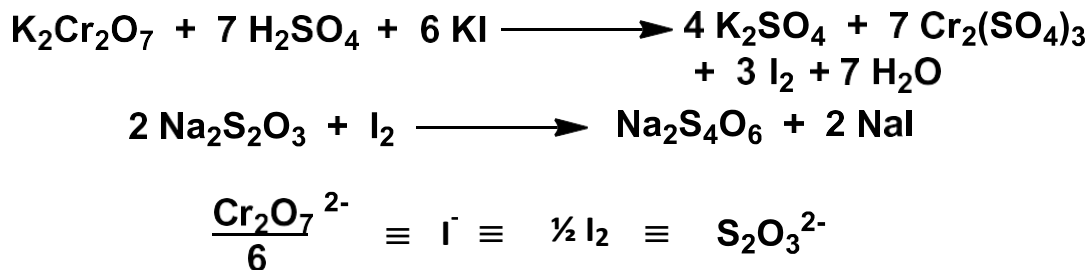
**Theory:**

This experiment comes under the category of Iodometry. Iodometry means the titration of liberated iodine with a standard solution of sodium thiosulfate. Sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O) is not a primary standard substance because its crystals are efflorescent, its aqueous solution is unstable and on standing turns turbid due to the separation of colloidal sulfur, which is caused by atmospheric CO<sub>2</sub>, or by bright sunlight and by bacteria. So, first the thiosulfate solution is required to be standardized by a primary standard solution.

So, the experiment involves double titration. In the first titration the exact normality of a given thiosulfate solution is calculated by titrating against a given standard (known strength) solution of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. Then this thiosulfate solution of known normality is used in the second titration for calculating the normality of the given sample of Cu (II) of unknown strength.

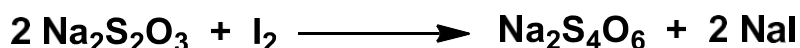
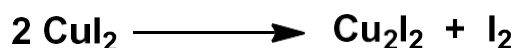
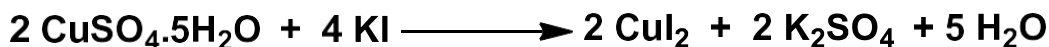
***First Titration: Standardization of thiosulfate***

In acid medium, Cr<sub>2</sub>O<sub>7</sub><sup>2-</sup> quantitatively oxidizes I<sup>-</sup> to I<sub>2</sub> and the liberated I<sub>2</sub> is titrated with thiosulfate solution using starch as indicator (End Point: Blue to Green)



### ***Second Titration: Estimation of the strength of Cu (II) solution***

Cu (II) reacts with iodide and the liberated I<sub>2</sub> is titrated with thiosulfate solution using starch as indicator (End Point: Blue to White)



From the above equation, it is evident



Eq. Wt. of Cu<sup>2+</sup> = (2 x 63.5)/2 = 63.5

From the normality equation, normality of Cu<sup>2+</sup> and hence the strength of Cu<sup>2+</sup> in hydrated CuSO<sub>4</sub> crystals is determined.

### **Procedure:**

**(a) Determination of strength of thiosulfate solution:** Fill the burette with thiosulfate solution. Pipette out 5 ml (N/20) K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution in a conical flask. Add 1/3 test tube of dil. H<sub>2</sub>SO<sub>4</sub> to it. Then add 5-6 drops of KI solution, cork the flask and leave in dark for 2-5 min. The solution in the titration flask will now become dark brown and is titrated against Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> till a pale brown colour is obtained. Add 2 drop starch. The solution turns blue. Now titrate it further to get the end point. Repeat and get the concordant reading.

**(b) Determination of strength of Cu(II) solution:** Pipette out 5 ml Cu(II) solution in a clean conical flask. Neutralise any free acid present by adding Na<sub>2</sub>CO<sub>3</sub> solution drop by drop till a faint permanent precipitate remains even on shaking. Add dil. Acetic acid drop wise until the precipitate just dissolves. Then add 10-15 drop of KI solution. Cork the flask and leave in dark for 2-5 min. The solution in the titration flask will now become dark brown and is titrated against Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> till a pale brown colour is obtained. Add 2 drop starch. The solution turns blue. Now titrate it further to get the end point. Repeat and get the concordant reading.

## **Observation:**

**Table 1 for Procedure (a)**

No. of Obs.	Vol. of $K_2Cr_2O_7$ (ml)	Burette Reading			Concordant Reading	Vol. Of thiosulfate (ml)
		Initial	Final	Difference		

**Table 2 for procedure (b)**

No. of Obs.	Vol. of Cu(II) Solution (ml)	Burette Reading			Concordant Reading	Vol. Of thiosulfate (ml)
		Initial	Final	Difference		

## **Calculation:**

From Table 1:  $N_1V_1$  of  $K_2Cr_2O_7 = N_2V_2$  of  $Na_2S_2O_3$

$$N_2 =$$

From Table 2:  $N_1V_1$  of Cu(II) solution =  $N_2V_2$  of  $Na_2S_2O_3$

$$N_1 =$$

**Result:** Strength of Cu(II) solution =  $N_1 \times 63.5$  g/L = ..... g/L

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## *Experiment: 05*

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### **Aim:**

To determine the hardness of given water sample by EDTA method

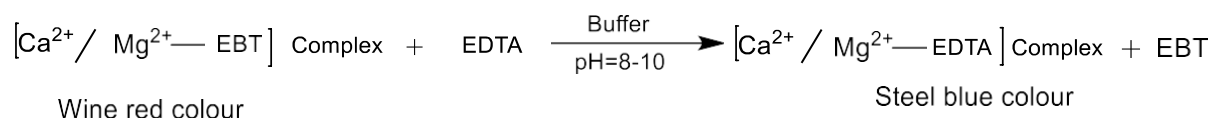
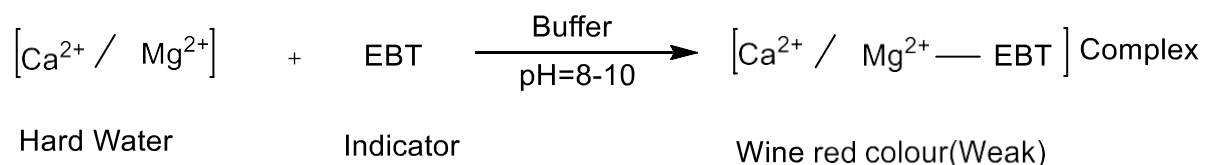
### **Requirements:**

(a) **Apparatus:** Burette, pipette, conical flask, stand

(b) **Chemicals:**  $\text{CaCl}_2$  solutions, buffer solution, tap water, Eriochrome Black-T (EBT) indicator, EDTA solution

### **Theory:**

EDTA is ethylene diamine tetra acetic acid. Since pure EDTA is not dissolved in water. Disodium salt of EDTA is taken for this experiment. It is a well-known complexing agent. It is used to estimate the hardness of given hard water containing the  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions. When the EDTA is added to hard water, it reacts with the calcium and magnesium present in hard water to form stable EDTA metal complexes. From the volume of EDTA consumed the hardness can be calculated. Eriochrome Black-T is used as an indicator. The indicator forms a weak complex with the metal ions present in hard water and gives wine red colour.



When the EDTA is added into the hard water, the metal ions form a stable complex with EDTA by leaving the indicator. When all the metal ions are taken by EDTA from the indicator metal ion complex, the wine-red colour changes into steel blue, which denotes the end point. The metal EDTA complex is stable at range of pH 8-10. This pH range can be maintained by adding buffer solution ( $\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$ ).



## **Procedure:**

- (a) ***Standardization of EDTA solution by  $\text{CaCl}_2$  solution:*** Fill the burette with EDTA solution. Pipette out 5ml (M/20)  $\text{CaCl}_2$  solution in a conical flask. Add  $\frac{1}{2}$  test tube of buffer solution and 2 drops of EBT indicator to it. Titrate against EDTA solution to get the end point (wine red to blue colour). Repeat and get the concordant reading.
- (b) ***Standardization of tap water by EDTA solution:*** Pipette out 25 ml tap water in a conical flask. Add  $\frac{1}{2}$  test tube of buffer solution and 2 drops of EBT indicator to it. Titrate against EDTA solution to get the end point (wine red to blue colour). Repeat and get the concordant reading.

## **Observation:**

**Table 1 for procedure (a)**

No. of obs.	Vol. of $\text{CaCl}_2$ solution (ml)	Burette Reading			Concordant Reading	Vol. of EDTA solution (ml)
		Initial	Final	Difference		
					$V_1$	$V_1$

**Table 2 for procedure (b)**

No. of obs.	Vol. of tap water (ml)	Burette Reading			Concordant Reading	Vol. of EDTA solution (ml)
		Initial	Final	Difference		
					$V_2$	$V_2$

## **Calculation:**

**From table 1:**

$$V_1 \text{ ml EDTA} = 5 \text{ ml (M/20) CaCl}_2$$

$$= 5 \text{ ml (M/20) CaCO}_3 = (100 \times 5 \times 1/20)/1000 \text{ g} = 1/40 \text{ g} = 25 \text{ mg CaCO}_3$$

$$1 \text{ ml EDTA} = 25/V_1 \text{ mg CaCO}_3$$

$$*W = (\text{Mol. Wt.} \times \text{Vol.} \times \text{Strength in molarity})/1000$$

**From table 2:**

$$25 \text{ ml tap water} = V_2 \text{ ml EDTA} = V_2 \times 25 / V_1 \text{ mg CaCO}_3$$

$$1000 \text{ ml tap water} = V_2 \times 25 / V_1 \times 1000 / 25 \text{ mg CaCO}_3 = \dots\dots\dots \text{ppm CaCO}_3$$

## **Result:**

Hardness of given water sample is.....ppm.

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## ***Experiment: 06***

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### **Aim:**

To synthesize Tris(acetylacetonato)Manganese(III).

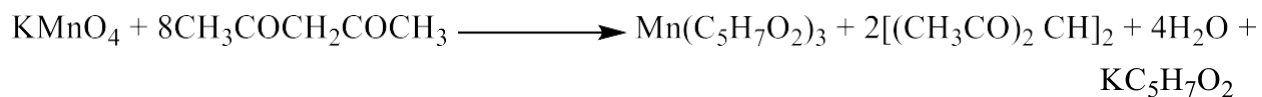
### **Requirement:**

**Apparatus:** 100 mL beaker, Buchner funnel setup, watch glass, 5 mL measuring cylinder, dropper, Spatula, Glass rod Capillary tube, Wash bottle, thermometer, melting point apparatus, filter paper

**Chemicals:** KMnO<sub>4</sub>, Acetyl acetone.

### **Theory:**

The complex Tris(acetylacetonato)Manganese(III) or Manganese tris(acetylacetonato) is synthesized by reacting KMnO<sub>4</sub> with acetylacetone.



The reaction is based on electron transfer between Mn(VII) and acacH. No any buffer is required. The compound is obtained as dark brown-black crystals. The Mn(acac)<sub>3</sub> is monomeric in nature. The oxidation state of Mn in the compound is determined iodometrically by reduction of a known amount of the compound with acidified potassium iodide solution followed by titration of liberated iodine with standard sodium thiosulphate solution. The redox titration can be also used for quantitative determination of Mn content in the compound.

### **Procedure:**

Take fine powdered of KMnO<sub>4</sub> (0.5 g 3.2 mmol) in 5 mL of distilled water into a 100 mL beaker. Dissolve it by slightly warming on water bath. To this solution add acetylacetone (2.3 mL 22 mmol) in dropwise and swirl time to time. Stir the reaction mixture for 5 min. Keep it again on water-bath until dark brown- black shiny crystals appear from the reaction mixture. Take out the reaction mixture from water-bath and allow mixture to cool for 20 min. Filter the reaction mixture with suction on Buchner funnel and wash the solid material with small amount of cold

acetyl acetone water mixture (1:1 v/v). Collect the dark brown black crystal and dry in vacuum oven. Weigh the solid material and calculate its yield. Find out the melting point of compound.

### **Observation:**

Weight of compound prepared: -

Melting point of the compound: -

### **Calculation:**

Calculate the theoretical yield and find out percentage yield of product.

### **Results:**

Percentage yield -----

Melting point -----

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## ***Experiment: 07***

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### **Aim:**

Preparation of dibenzalacetone by aldol condensation reaction

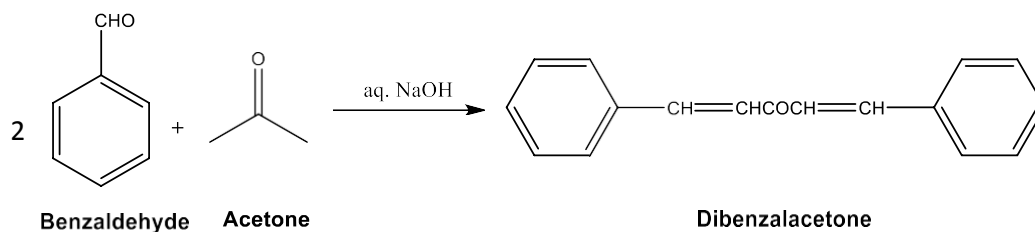
### **Chemicals and Apparatus required:**

- |                 |                                |                         |
|-----------------|--------------------------------|-------------------------|
| 1) Benzaldehyde | 2) A.R. Acetone                | 3) Sodium hydroxide     |
| 4) Ethanol      | 5) Rectified spirit            | 6) 250 ml Conical flask |
| 7) Test tube    | 8) Suction filtering apparatus | 9) Thermometer          |

### **Procedure:**

Prepare a cold solution of 500 mg of sodium hydroxide in 5 ml of water and 4 ml of ethanol in a 100 ml conical flask. In a dry test tube, prepare a mixture of 0.5 ml of benzaldehyde and 1 ml of A. R. acetone. Add the mixture from the test tube into the cold solution in the conical flask. Shake frequently and keep the temperature at 20°C - 25°C for 15 minutes by immersing the flask in a bath of ice-cold water. Collect the precipitated dibenzalacetone by filtration at the pump, wash with cold water to remove the alkali and dry the solid at room temperature upon the filter paper. Recrystallize the crude product from hot rectified spirit. Dibenzalacetone appears as pale yellow crystalline needles. Check the melting point of the product. Calculate the yield and percent yield of the product.

### ***Reactions Involved:***



**Inference:** The melting point of the product formed \_\_\_\_\_  
The yield of the product formed \_\_\_\_\_  
The percent yield of the product formed \_\_\_\_\_

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## Experiment: 08

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### Aim:

Preparation of 1-nitronaphthalene by nitration

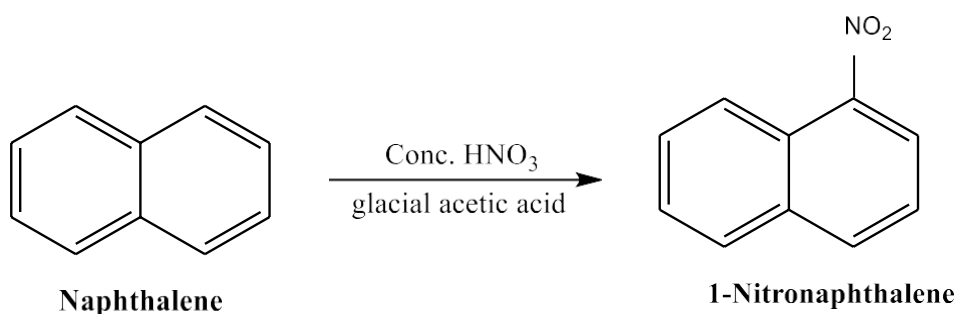
### Chemicals and Apparatus required:

- |                      |                                     |                         |
|----------------------|-------------------------------------|-------------------------|
| 1) Naphthalene       | 2) Glacial Acetic acid              | 3) Conc. $\text{HNO}_3$ |
| 4) Ethanol           | 5) 100 ml 24/29 Joint Conical flask | 6) Air condenser        |
| 7) Boiling chip      | 8) Suction filtering apparatus      | 9) Hot plate            |
| 10) Crystalline dish | 11) Thermometer                     |                         |

### Procedure:

Dissolve 500 mg of naphthalene in 2.5 ml glacial acetic acid by warming in a 100 ml conical flask. Cool the solution and add dropwise 0.5 ml of concentrated nitric acid keeping the temperature of the reaction mixture below  $40^\circ\text{C}$ . Heat the reaction mixture on a hot water bath at  $\sim 45^\circ\text{C}$  -  $50^\circ\text{C}$  for 10 minutes. Pour the solution on to  $\sim 10$  g crushed ice with stirring. When ice has melted, filter the solid under suction and wash thoroughly with cold water. Recrystallize the crude product from ethanol. 1-Nitronaphthalene separates as, pale yellow needles. Check the melting point of the product. Calculate the yield and percent yield of the product.

### *Reactions Involved:*



**Inference:** The melting point of the product formed \_\_\_\_\_  
The yield of the product formed \_\_\_\_\_  
The percent yield of the product formed \_\_\_\_\_

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## Experiment: 09

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### Aim:

Preparation of *p*-iodonitrobenzene via diazotisation

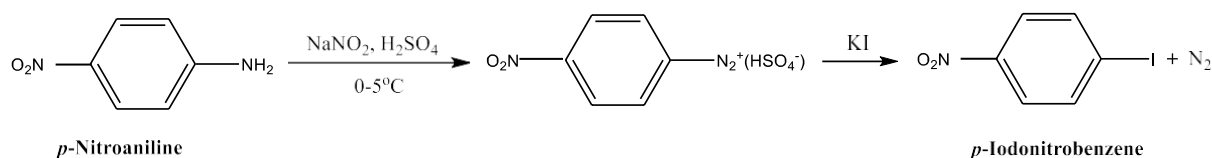
### Chemicals and Apparatus required:

- |                           |                                     |
|---------------------------|-------------------------------------|
| 1) <i>p</i> -Nitroaniline | 7) Suction filtering apparatus      |
| 2) Sodium nitrite         | 8) Magnetic stirrer bar             |
| 3) Potassium iodide       | 9) 100 ml Beaker                    |
| 4) Conc. Sulphuric acid   | 10) Magnetic stirrer with hot plate |
| 5) Sodium thiosulphate    | 11) Thermometer                     |
| 6) Ethanol                |                                     |

### Procedure:

Place 1.5 g of *p*-nitroaniline, 3 ml of water and 1.35 ml conc. sulphuric acid in a 100 ml beaker. Stir for 20 minutes. Place a thermometer in the beaker and cool the reaction mixture to 0°-5°C (ice-salt bath). Add dropwise with stirring a solution of 0.25g of NaNO<sub>2</sub> in 1 ml of water keeping the temperature below 5°C. Filter the cold solution and add the filtrate with stirring to a solution of 1g of KI in 3 ml of water taken in a 100 ml beaker. Collect the precipitated solid by filtration under suction and then shake it with sodium thiosulphate solution to remove the excess of iodine. Filter off the product, wash with water, drain well and dry by suction, then recrystallize from ethanol. Check the melting point of the product. Calculate the yield and percent yield of the product.

### Reactions Involved:



**Inference:** The melting point of the product formed \_\_\_\_\_

The yield of the product formed \_\_\_\_\_

The percent yield of the product formed \_\_\_\_\_

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## Experiment: 10

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### Aim:

Alkaline hydrolysis of methyl benzoate to benzoic acid

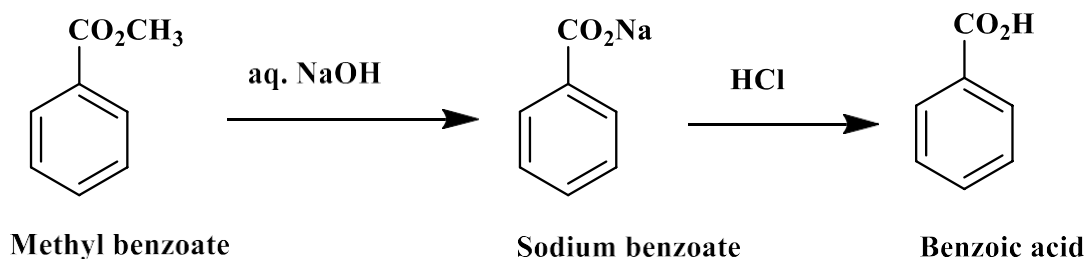
### Chemicals and Apparatus required:

- |                       |                                |                     |
|-----------------------|--------------------------------|---------------------|
| 1) Methyl benzoate    | 2) Sodium hydroxide (20 %)     | 3) Conc. HCl        |
| 4) 150 ml R. B. flask | 5) Reflux condenser            | 6) Boiling chip     |
| 7) Hot plate          | 8) Suction filtering apparatus | 9) Crystalline dish |
| 10) Thermometer       | 11) TLC Plate                  | 12) Silica gel      |
| 13) DCM               |                                |                     |

### Theory:

Esters are hydrolyzed either by an acid or a base. Alkaline hydrolysis of ester is irreversible while acid hydrolysis of ester is a reversible reaction. Acid hydrolysis of esters can occur by more than one type of mechanism, the common mechanism is the alkaline hydrolysis, which occurs through a nucleophilic acyl substitution. Here  $\text{OH}^-$  ion of sodium hydroxide act as a nucleophile. Methyl benzoate react faster with aq. NaOH to give sodium benzoate and methanol. This process is called saponification. Sodium benzoate is non-volatile, so remain in solution. On treating this solution with HCl, we get benzoic acid which can be recrystallized to get white crystalline solid.

### *Reactions:*



### Procedure:

Place 0.5 ml of methyl benzoate and 5 ml of 20 % sodium hydroxide solution in a 50 ml round-bottom flask equipped with a reflux condenser. Add some boiling chips. Boil the mixture under reflux for 30 minutes or until the ester layer disappears. Cool and acidify with



conc. HCl and then allow to stand at room temperature. Filter the crude benzoic acid under suction, wash with cold water and drain well. Recrystallize the crude acid from hot water. Pure benzoic acid separates as colourless crystals. Check the melting point of the product. Calculate the yield and percent yield of the product.

### **Calculation:**

Molecular formula of methyl benzoate =  $C_8H_8O_2$

Molecular formula of benzoic acid =  $C_7H_6O_2$

Weight of methyl benzoate = .....g/mol

Weight of benzoic acid = .....g/mol

### ***Theoretical yield:***

136.15g of methyl benzoate gives 112g of benzoic acid

0.5g of methyl benzoate gives benzoic acid = .....g

Experimental yield = .....g

$$\% \text{ Yield} = \frac{\text{Experimental yield}}{\text{Theoretical yield}} \times 100$$

### **Result:**

The yield of the product formed \_\_\_\_\_

The percent yield of the product formed \_\_\_\_\_