# DBT-STAR COLLEGE SCHEME

# Revised Practical Protocol Manual

## Volume 2: CHEMISTRY

Since 1951


**JAMAL MOHAMED COLLEGE (Autonomous)**

**College with Potential for Excellence**

**Accredited (3rd cycle) with "A" Grade by NAAC**

**DBT Star Scheme & DST-FIST Funded College**

**(Affiliated to Bharathidasan University)**

**TIRUCHIRAPPALLI-620 020**

## 1. ESTIMATION OF OXALIC ACID BY $KMNO_4$ USING A STANDARD OXALIC ACID

### Aim

Estimate the amount of oxalic acid present in the whole of the given solution, being supplied with oxalic acid and approximately N/20 $KMnO_4$ solution.

### Principle

Estimation is based on the reaction between $KMnO_4$ and Oxalic acid. $KMnO_4$ oxidizes oxalic acid in the presence of acid and while hot.

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$5(COOH)_2 + 5[O] \rightarrow 5H_2O + 10CO_2\uparrow$$

Equivalent weight of oxalic acid = 63

### Procedure

**Titration 1: Standardization of Oxalic acid**

Approximately 0.8g of oxalic acid is weighed and transferred into a 250 mL standard flask and made up to the mark. 20 mL of the standard oxalic acid solution is pipetted out into a clean conical flask and 20 mL of dil. $H_2SO_4$ is added. It is then heated to 60-80$^oC$ and the hot solution is titrated against the $KMnO_4$ solution taken in the burette. The end point is the appearance of the pale pink colour. The titrations are repeated for concordant values.

**Titration 2: Estimation of Oxalic acid**

The given oxalic acid solution is made up to 100 mL in a standard flask. 20 mL of the solution is pipetted out into a clean conical flask and added with about 20 ml of dil.$H_2SO_4$. It is then heated to 60-80$^oC$ and the hot solution is titrated against $KMnO_4$ solution is taken in a burette. The end point is the appearance of pink colour. The titration is repeated for concordant values.

### RESULT

The amount of Oxalic acid present in whole of the given solution is=…….g.

**ESTIMATION OF OXALIC ACID BY $KMNO_4$ USING A STANDARD OXALIC ACID**

Strength of Oxalic acid = $\frac{\text{Weight /lit.}}{\text{Eq.Wt.}}$

Strength of Oxalic acid = ----------N.

**Titration 1:** Standardization of Oxalic acid

Std. Oxalic acid Vs $KMnO_4$ Indicator: Self

| S. No. | Volume of Oxalic acid (mL) | Burette Reading | | Volume of $KMnO_4$ (mL) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (mL) | Final (mL) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

**Calculation:**

Volume of Oxalic acid (V1) = ……..mL

Strength of Oxalic acid (N1) = ……..N

Volume of $KMnO_4$ (V2) = …….mL

Strength of $KMnO_4$ (N2) = V1xN1/V2

The strength of $KMnO_4$ = ……N.

**Titration 2:** Estimation of Oxalic acid

Given Oxalic acid Vs $KMnO_4$ Indicator: Self

| S. No. | Volume of Oxalic acid (mL) | Burette Reading | | Volume of $KMnO_4$ (mL) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (mL) | Final (mL) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

**Calculation:**

Volume of $KMnO_4$ (V1) = ……mL

Strength of $KMnO_4$ (N1) = ……N

Volume of oxalic acid (V2) = ……...mL

Strength of oxalic acid (N2) = V1xN1/V2 = ……N

Amount of Oxalic Acid present in the whole of the given solution

$$= \frac{\text{Strength of Oxalic acid X Eq.wt.of oxalic acid}}{10}$$

= ----------g.

## 2. ESTIMATION OF FERROUS SULPHATE

**Aim**

Estimate the amount of Ferrous Sulphate present in whole of the given solution, being supplied with oxalic acid crystals and approximately N/20 $KMnO_4$ solution.

**Principle**

Estimation is based on the reaction between $KMnO_4$ and $FeSO_4$. $FeSO_4$ in the acid medium is oxidized by permanent to Ferric Sulphate as shown in the following equation.

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_20 + 5\,[0]$$
$$FeSO_4 + H_2SO_4 + [0] \longrightarrow Fe_2(SO_4)_3 + H_2O$$
$$H_2C_2O_4 + (O) \longrightarrow H_2O + 2CO_2$$

Equivalent weight of oxalic acid = 63
Equivalent weight of Ferrous Sulphate = 278

**Procedure**

**Titration 1: Standardization of $KMnO_4$**

Approximately 0.8 g oxalic acid is weighed and transferred into a 250 mL standard flask. It is then dissolved in distilled water and made up to the mark. 20 mLof the standard oxalic acid solution is pipetted out into a clean conical flask and 20 mL of dil.$H_2SO_4$ is added and the mixture is heated to 60-80$^oC$ on a wire gauze. This solution is titrated against the $KMnO_4$ solution taken in the burette. The end point is the appearance of the pale pink colour. The titration is repeated for concordant values.

**Titration 2: Estimation of Ferrous Sulphate**

The given Ferrous Sulphate solution is made up to 100 mL standard flask. 20 mL of the solution is pipetted out into a clean conical flask and then added with about 20 mL of dil.$H_2SO_4$. It is titrated against $KMnO_4$ solution taken in the burette. The end point is the appearance of pink colour. The titration is repeated for concordant values.

**RESULT**

The amount of Ferrous Sulphate present in whole of the given solution is =…….g.

## ESTIMATION OF FERROUS SULPHATE

Strength of Oxalic acid = $\frac{\text{Weight /lit.}}{\text{Eq.Wt.}}$

Strength of Oxalic acid = ---------- N.

Titration 1: Standardization of $KMnO_4$

Std. Oxalic acid Vs $KMnO_4$ Indicator: Self

| S. No. | Volume of Oxalic acid (mL) | Burette Reading | | Volume of $KMnO_4$ (mL) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (mL) | Final (mL) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

Calculation:

Volume of Oxalic acid (V1) = ……..mL

Strength of Oxalic acid (N1) = …….N

Volume of $KMnO_4$ (V2) = ……mL

Strength of $KMnO_4$ (N2) = V1xN1/V2

The strength of $KMnO_4$ = ----------N.

Titration 2: Estimation of Ferrous Sulphate

Given Ferrous Sulphate Vs $KMnO_4$ Indicator: Self

| S. No. | Volume of Ferrous Sulphate (mL) | Burette Reading | | Volume of $KMnO_4$ (mL) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (mL) | Final (mL) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

Calculation:

Volume of $KMnO_4$ (V1) = ……mL

Strength of $KMnO_4$ (N1) = ……N

Volume of $FeSO_4$ (V2) = ……mL

Strength of $FeSO_4$ (N2) = V1xN1/V2

The strength of $FeSO_4$ = ……..N

The amount of Ferrous sulphate present in the whole of the given solution

$$= \frac{\text{Strength of } FeSO_4 \text{ X Eq.wt.of } FeSO_4}{10}$$

= ---------- g

## 3. ESTIMATION OF OXALIC ACID

**Aim**

Estimate the amount of oxalic acid present in the whole of the given solution, being supplied with Ferrous Sulphate crystals and approximately N/20 $KMnO_4$ solution.

**Principle**

Estimation is based on the reaction between $KMnO_4$ and Oxalic acid. $KMnO_4$ oxidizes oxalic acid in the presence of acid and while hot.

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_20 + 5\,[0]$$
$$FeSO_4 + H_2SO_4 + [0] \longrightarrow Fe_2(SO_4)_3 + H_2O$$
$$H_2C_2O_4 + (O) \longrightarrow H_2O + 2CO_2$$

Equivalent weight of oxalic acid = 63
Equivalent weight of Ferrous Sulphate = 278

**Procedure**

**Titration 1: Standardization of $KMnO_4$**

Approximately 3.5 g of Ferrous Sulphate is weighed and transferred into a 250 mL standard flask. It is then dissolved in distilled water to which 10 mL of dil.$H_2SO_4$ has been added and made up to the mark. 20 mL of the standard Ferrous Sulphate solution is pipetted out into a clean conical flask and 20 mL of dil. $H_2SO_4$ is added. This solution is titrated against the $KMnO_4$ solution taken in the burette. The end point is the appearance of the pale pink colour. The titration is repeated for concordant values.

**Titration 2: Estimation of Oxalic acid**

The given Oxalic acid solution is made up to 100 mL in a standard flask. 20 mL of the solution is pipetted out into a clean conical flask and added with about 20 mL of dil.$H_2SO_4$. It is then heated to 60-80$^oC$ and the hot solution is titrated against $KMnO_4$ solution is taken in a burette. The end point is the appearance of pink colour. The titration is repeated for concordant values.

**RESULT**

Amount of Oxalic acid present in the whole of the given solution is=…….g.

## ESTIMATION OF OXALIC ACID

Strength of $FeSO_4$ = $\frac{\text{Weight /lit.}}{\text{Equ.Wt.}}$

Strength of $FeSO_4$ = ----------N

Titration 1: Standardization of $KMnO_4$

Std. $FeSO_4$ Vs $KMnO_4$ Indicator: Self

| S.No | Volume of Ferrous Sulphate (mL) | Burette Reading | | Volume of $KMnO_4$ (mL) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (mL) | Final (mL) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

Calculation:

Volume of $FeSO_4$ (V1) = 20.0 mL

Strength of $FeSO_4$ (N1) = …….N

Volume of $KMnO_4$ (V2) = ……mL

Strength of $KMnO_4$ (N2) = V1xN1/V2

The strength of $KMnO_4$ = ……..N.

Titration 2: Estimation of Oxalic acid

Given Oxalic acid Vs $KMnO_4$ Indicator: Self

| S.No | Volume of Oxalic acid (ml) | Burette Reading | | volume of $KMnO_4$ (ml) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (ml) | Final (ml) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

Calculation:

Volume of $KMnO_4$ (V1) = ……mL

Strength of $KMnO_4$ (N1) = ……..N

Volume of Oxalic acid (V2) = 20.0 mL

Strength of Oxalic acid (N2) = V1xN1/V2 --------- -= N

The amount of Oxalic acid present in the whole of the given solution

$= \frac{\text{Strength of Oxalic acid X Eq.wt.of Oxalic acid}}{10}$

= ----------g.

## 4. ESTIMATION OF MAGNESIUM

**Aim**

Estimate the amount of magnesium present in the whole of the given solution, being supplied with Zinc sulphate crystals and approximately N/20 EDTA solution.

**Principle**

$Mg^{2+}$ ion is forming complex with EDTA Eriochrome Black-T (EBT) as indicator. The reaction involved in this titration is as follows:

$Mg^{2+}$ + EBT $\longrightarrow$ (EBT- $Mg^{2+}$)

(EBT- $Mg^{2+}$) + EDTA $\longrightarrow$ (EDTA - $Mg^{2+}$) + EBT

Molecular weight of Magnesium Sulphate = 123.23g
Equivalent weight of Zinc Sulphate = 143.8g

**Procedure**

**Titration 1: Standardization of EDTA**

Approximately 0.8g of $ZnSO_4$ is weighed and transferred into a 250 mL standard flask. It is then dissolved in distilled water and made up to the mark. 20 mL of the standard solution of $ZnSO_4$ is pipette out into a clean conical flask and 2 mL of buffer solution of pH 10 is added followed by 2 drops of EBT as indicator.This solution is titrated against the EDTA solution. The end point is change of colour from wine-red to blue. The titration is repeated for concordant value.

**Titration 2 : Estimation of Magnesium**

The given $MgSO_4$ solution is made up to 100 mL. 20 mL of the made up solution is pipette out into a clean conical flask. 2 ml of buffer solution of pH 10 is added followed by 4 drops of EBT as indicator. The solution is warmed to 40$^oC$ and titrated against EDTA solution. The end point is change of colour from wine-red to blue. The titration is repeated for concordant value.

**RESULT**

Amount of magnesium present in the whole of the given solution =…….g.

## ESTIMATION OF MAGNESIUM

Strength of $ZnSO_4$ = Weight /lit.
Equ.Wt.

Strength of $ZnSO_4$ = ----------N.

Titration 1: Standardization of EDTA

Std. Zinc Sulphate Vs EDTA Indicator: EBT

| S.No | Volume of Zinc Sulphate (mL) | Burette Reading | | Volume of EDTA (mL) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (mL) | Final (mL) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

Calculation:

Volume of $ZnSO_4$ (V1) = 20.0 mL

Strength of $ZnSO_4$ (N1) = ……N

Volume of EDTA (V2) = ……mL

Strength of EDTA (N2) = V1xN1/V2

The strength of EDTA = ----------N.

Titration 2: Estimation of $MgSO_4$

Given Magnesium Sulphate Vs EDTA Indicator: EBT

| S.No | Volume of Magnesium Sulphate (mL) | Burette Reading | | Volume of EDTA (mL) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (mL) | Final (mL) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

Calculation:

Volume of EDTA (V1) = ……..mL

Strength of EDTA (N1) = ……N

Volume of $MgSO_4$ (V2) = 20.0 mL

Strength of $MgSO_4$ (N2) = V1xN1/V2

The strength of Magnesium sulphate = ……..N.

The amount of $MgSO_4$ present in the whole of the given solution

= Strength of $MgSO_4$ X Eq.wt.of $MgSO_4$
10

= ----------g.

## 5. ESTIMATION OF POTASSIUM DICHROMATE

**Aim**

Estimate the amount of $K_2Cr_2O_7$ present in the whole of the given solution, being supplied with $K_2Cr_2O_7$ crystals and approximately N/20 Sodium thiosulphate solution.

**Principle**

$$Cr_2O_7^{2-} + 14H^+ + 6I^- \longrightarrow 2Cr^{3+} + 7H_2O + 3H_2$$

$$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$$

Equivalent weight of $K_2Cr_2O_7$ = 49g

**Procedure**

**Titration 1: Standardization of Thio**

Approximately 0.6125g of $K_2Cr_2O_7$ is weighed and transferred into a 250 mL standard flask. It is dissolved in distilled water and made up to the mark. 20 mL of the stranded solution of $K_2Cr_2O_7$ is pipette out into a clean conical flask. About 5 mL of Conc. HCl is added, followed by 10 mL of 10% aq.KI solution. The liberated iodine is immediately titrated against thio sulphate solution taken in the burette.When the solution becomes pale yellow in color, 1 mL of freshly prepared starch solution is added, and the titration is continued, adding thio in dropwise, with constant shaking. The end point is the change of colour from blue to green (due to $Cr^{3+}$). The titration is repeated to get concordant values.

**Titration 2: Estimation of Potassium di chromate**

The given $K_2Cr_2O_7$ solution is made up to 100 mL. 20 mL of the made up solution is pipette out into a clean conical flask. About 5 mL of conc. HCl is added, followed by 10 mL of 10% aq.KI solution. The liberated iodine is immediately titrated against thio sulphate solution taken in the burette. When the solution becomes pale yellow in colour, 1 mL of freshly prepared starch solution is added and the titration is continued, adding thio in dropwise, with constant shaking. The end point is the change of colour from blue to green (due to $Cr^{3+}$). The titration is repeated to get concordant values.

**RESULT**

The amount of $K_2Cr_2O_7$ present in whole of the given solution =…….g.

## ESTIMATION OF POTASSIUM DICHROMATE

Strength of $K_2Cr_2O_7$ = Weight /lit.
Equ.Wt.

Strength of $K_2Cr_2O_7$ = ----------N.

Titration 1: Standardization of Thio

Std. $K_2Cr_2O_7$ Vs Thio Indicator: Starch

| S.No | Volume of $K_2Cr_2O_7$ (mL) | Burette Reading | | Volume of Thio (mL) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (mL) | Final (mL) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

Calculation:

Volume of $K_2Cr_2O_7$ (V1) = 20.0 mL

Strength of $K_2Cr_2O_7$ (N1) = ……N

Volume of Thio (V2) = ……..mL

Strength of Thio (N2) = V1xN1/V2

The strength of Thio = ----------N.

Titration 2: Estimation of $K_2Cr_2O_7$

Given $K_2Cr_2O_7$ Vs Thio Indicator: Starch

| S.No | Volume of $K_2Cr_2O_7$ (mL) | Burette Reading | | Volume of Thio (mL) | Concordant value |
|---|---|---|---|---|---|
| | | Initial (mL) | Final (mL) | | |
| 1 | 20.0 | 0.0 | | | |
| 2 | 20.0 | 0.0 | | | |

Calculation:

Volume of Thio (V1) = ……..mL

Strength of Thio (N1) = ……N

Volume of $K_2Cr_2O_7$ (V2) = 20.0 mL

Strength of $K_2Cr_2O_7$ (N2) = V1xN1/V2

The strength of $K_2Cr_2O_7$ = ……..N.

The amount of $K_2Cr_2O_7$ present in the whole of the given solution

= Strength of $K_2Cr_2O_7$ X Eq.wt.of $K_2Cr_2O_7$
10

= ----------g.

## SPECTROPHOTOMETRIC ANALYSIS

## 1. ESTIMATION OF COMMERCIAL ASPIRIN

**Aim:**

To analyse the aspirin content in commercial Aspirin tablet by spectrophotometrically

**Chemicals Required:**

Acetylsalicylic acid

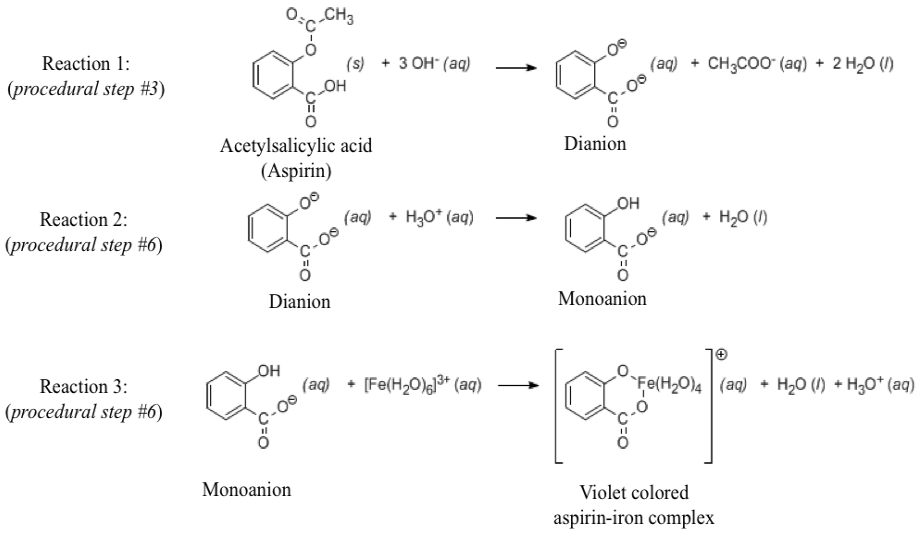
1 M sodium hydroxide

0.02 M iron(III) chloride

Commercial aspirin tablet

## Principle:

Acetylsalicylic acid, commonly known as aspirin, absorbs -light in the UV region of the electromagnetic spectrum. 324. The Spectronic 200 operates in the visible region. Therefore, we must perform a series of chemical reactions to convert acetylsalicylic acid to a colored complex, A base (e.g., sodium hydroxide) hydrolyzes acetylsalicylic acid to yield salicylate dianion. Acidification converts the dianion to a monoanion, which complexes with iron (III) to produce a violet-colored complex.




**Procedure:**

**Preparation of standard solution**

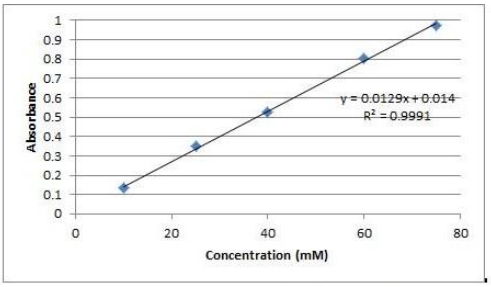
Weigh approximately 0.16 g of acetylsalicylic acid in a 125 mL Erlenmeyer flask. Add 5 mL of 1 M sodium hydroxide and heat the mixture for complete dissolution. Allow this solution to cool, and then completely transfer it into a 100 mL volumetric flask. Dilute the solution with deionized water to the 100 mL mark on the flask (*Note: This solution is label as stock solution).* pipette out 0.5 mL stock solution into a 10.0 mL volumetric flask then add 0.02 M iron(III) chloride that is buffered to pH 1.6 and dilute this solution to the 10.0 mL mark. This solution in a test tube labeled as A. In a similar fashion, prepare solutions labeled B, C, D, and E by using 0.40, 0.30, 0.20, and 0.10 mL aliquots of the sodium salicylate solution, diluting to 10.0 mL with iron(III) chloride solution.

**Preparation of sample**

A commercial aspirin tablet is crushed and divided into two equal halves. Record the exact mass of each portion .Transfer one portion of the crushed aspirin into 100 mL Erlenmeyer flasks. Add 5 mL of 1 M sodium hydroxide and heat the mixture until all solid dissolves. Allow this solution to cool and then transfer into two 100.0 mL volumetric flasks, using a glass funnel to ensure a quantitative transfer. Dilute these solutions to the 100.0 mL mark on the flasks and label these flasks Sample 1 and dublicate is made as Sample 2. Using a 1 mL graduated pipette, transfer a 0.3 mL sample of each solution into two 10.0 mL volumetric flasks and dilute to the 10.0 mL mark with 0.02 M iron(III) chloride. Record the absorbance for each standard solution and commercial sample solution using spectrophotometer. Plot Absorbance versus concentration of Fe(III)-salicylate complex. From the plot, concentration of commercial aspirin is determined.

**Table:**

| S. No. | Solution | Concentration | Absorbance |
|---|---|---|---|
| 01 | A | | |
| 02 | B | | |
| 03 | C | | |
| 04 | D | | |
| 05 | E | | |
| 06 | Sample1 | | |
| 07 | Sample2 | | |




Absorbance vs Concentration of Fe(III)-salicylate complex.

**Result:** The amount of aspirin present in commercial aspirin tablet is----------

## 2. ESTIMATION OF TRACE CHROMIUM CONTENT IN FOOD SAMPLE

**Aim:**

To analyse the chromium content in food samples by spectro photometrically

***Reagent required:***

$5.94 \times 10^{-6}$ M and $9.6 \times 10^{-5}$ M of standard chromium (VI)
1, 5-diphenylcarbazide
50 grams of each canned fruit juice sample
Phenol

### Principle:

Hexavalent chromium reacts with 1, 5-diphenylcarbazide to produce a reddish purple color in acidic solution and quantified by measuring its absorbance at its wavelength of maximum absorption.

**Procedure:**

**Determination of Absorption of Chromium (VI) - 1, 5-diphenylcarbazide (DPC) Complex:**

4 mL of $9.6 \times 10^{-5}$ M of standard chromium (VI) was pipetted out into 10 mL volumetric flask containing 4 mL of 0.01% 1,5-diphenylcarbazide. It was then diluted to mark with 0.2N sulfuric acid and mixed. The absorbance was then taken from 200 to 800 nanometer (nm) using a solution of 0.2N sulfuric acid as reference.

**1,5-Diphenylcarbazide Adherence to Beer's Law:**

Using a burette, 0, 2 ,4, 6 and 8 mL of $5.94 \times 10^{-5}$M standard chromium (VI) was transferred to each of five 25 mL volumetric flask containing 15 mL of 0.01% 1,5-diphenylcarbazide solution. The solution was mixed and diluted to mark with 0.2N sulfuric acid. After 30 minutes, the absorbance of the solution is recorded at maximum wavelength using ***UV-***VIS spectrophotometer and reference solution made by diluting 15 mL of 0.01% DPC to 25 mL with 0.2N sulfuric acid.

**Preparation of the sample*:***

About 50 g of each canned fruit juice sample in three replicates was separately placed in previously weighed empty crucible. It was then evaporated to dryness with low flame to avoid spattering of the sample followed by charring of the sample. The crucible was then placed in the muffle furnace and heated at 550$^{o}$C for one hour until the colour of the ash turned white. The crucible was removed from the furnace, transferred to a desiccator, cooled and weighed. The percentage of ash was then calculated. After ashing, 1 mL of HCl was added, rotated to wet all the ash and 2 mL of $HNO_3$ was added, transferred to 100 mL beaker and evaporated to dryness. The removal of the acid at this point must be fairly complete so that the subsequent addition of empirically established amount of bromine-sodium solution and sulfuric acid will bring the pH of the final solution within the range for colour development. Approximately 5 mL of distilled water was delivered into the sides of the beaker using a very fine stream of water. The solution was evaporated to dryness again. It was then removed from the hot plate and the residue was added with approximately 12 mL distilled water and 2 mL of bromine-sodium hydroxide oxidizing solution. This should precipitate all the iron and make the

solution definitely alkaline. It was then evaporated to a volume of approximately 4 mL with occasional stirring to ensure complete contact of the oxidizing solution. The mixture was allowed to room temperature and centrifuged to separate unwanted precipitate. It was decanted into a 50 mL volumetric flask. To the flask, 0.5 mL of 25% $H_2SO_4$ was added to make the solution 0.2 to 0.3N. Acidification produced the yellow brown color of the free bromine which was removed by the addition of 0.5 mL phenol and diluted to mark with distilled water.

Treatment with 0.1% 1,5-diphenylcarbazide *(DPC): Standard Addition Method*

Using a burette, 4 mL of the test solution was dropped into two 10 mL volumetric flask containing a 4 mL of DPC. One volumetric flask was added with 2 mL of the $5.94 \times 10^{-6}$ M standard Cr (VI) solution. The contents were mixed and diluted to mark with distilled water. The absorbance was measured at 543 nm against a reference solution made by diluting 4 mL of DPC with distilled water to 10 mL.

Ash content

The ash content can be a general measure of the quality of the product. It is an indication of the inorganic mineral content left after the oxidation of the samples. Various group of food vary in their ash content. Most fresh food can rarely have ash greater than 5%. Pure fats and oils have zero or little ash while processed food like bacon can have as high as 11.6%. Dairy products may vary from 0.5 to 5.1 % while fruits and fruit juice contain 0.2 to 0.6% ash.

High ash content suggests the presence of an adulterant. Figure 2 shows that sample 1 juice drink had the highest percentage ash content of 0.35%. The results of ash content analysis for all juice samples were within the average standard value, an implication that no adulterant was added to the fruit juice samples.

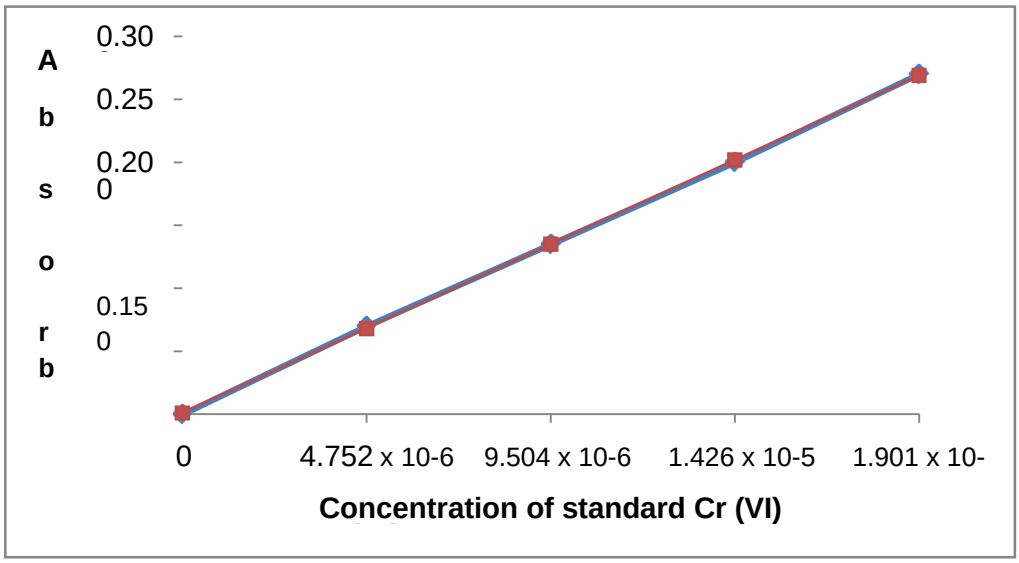
Chromium (VI) Content by Standard addition method

Standard addition method was used to determine the Cr (VI) content at 543 nm. Table 2 showed that pineapple orange flavor sample had the highest level of Cr (VI) concentration of 0.714 ppm. This is followed by orange flavor sample number 4, 0.450 ppm; pineapple sample 2, 0.426 ppm; orange flavor sample 5, 0.400 ppm and pineapple sample 3, 0.362 ppm. The range for Cr (VI) concentration for all samples is 0.362 ppm to 0.714 ppm. All of these values were beyond the permissible limit for Cr (VI) as set by the United States Environmental Protection Agency in drinking water which is 0.1 ppm

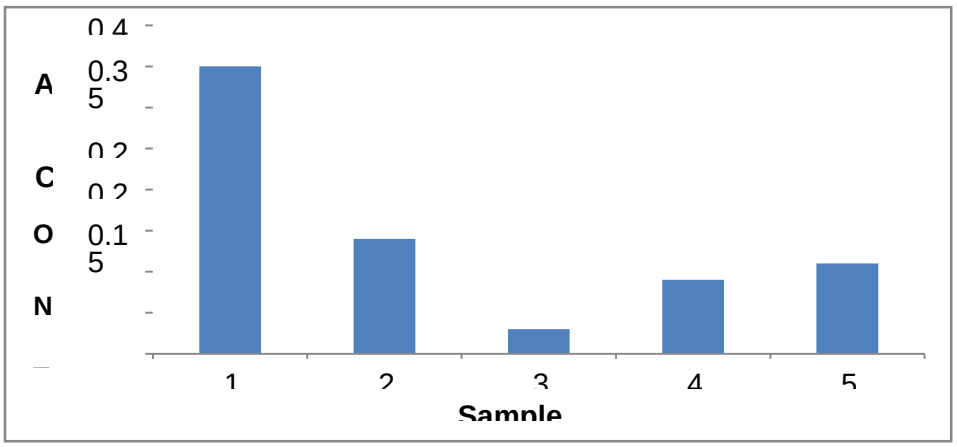
**Model Table 1.Different concentrations of standard Cr(VI) solution and its corresponding absorbance.**

| Run No. | Cr (VI) concentration, M | Absorbance |
|---|---|---|
| **1** | 0 | |
| **2** | $4.752 \times 10^{-6}$ | |
| **3** | $9.504 \times 10^{-6}$ | |
| **4** | $1.426 \times 10^{-5}$ | |
| **5** | $1.901 \times 10^{-5}$ | |

**Model Graph 1:**




**Model Graph 2:**

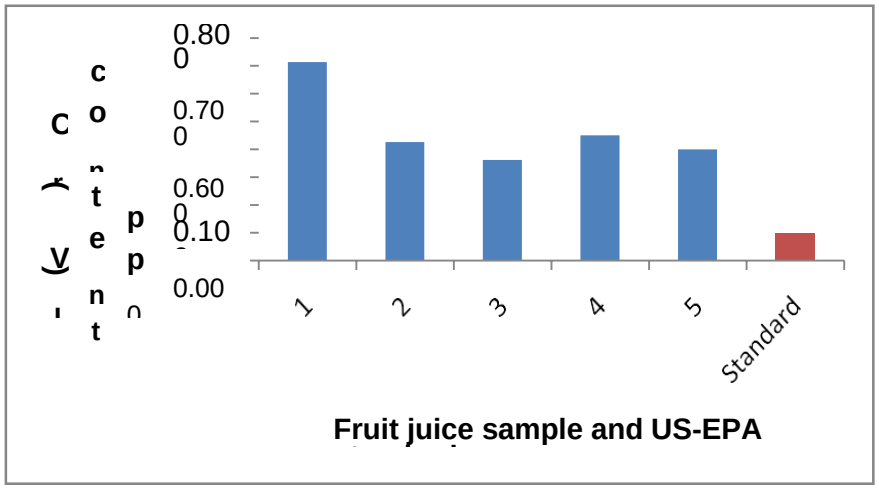



Ash content of different canned fruit juices.

**Model Table 2.Chromium (VI) Content in parts per million (ppm) for canned fruit juice samples.**

| Sample | Chromium (VI) Content, ppm |
|---|---|
| **A. Pineapple orange flavor** | |
| Sample 1 | 0.714 |
| **B. Pineapple Flavor** | |
| Sample 2 | 0.426 |
| Sample 3 | 0.362 |
| **C. Orange Flavor** | |
| Sample 4 | 0.450 |
| Sample 5 | 0.400 |

**Model Graph 3:**




Chromium (VI) content of canned fruit juices as compared to the US-EPA standard of Cr (VI) for drinking water.

**Result:**

The amount of Cr (VI) present in commercially available canned fruit juice is ----------

## 3. ESTIMATION OF IRON CONTENT IN FOOD ITEMS
## [Vitamin tablet, Flour and Tea samples]

**Aim**

To estimate the amount of Iron present in the whole of the given food sample (Vitamin tablet, Flour and Tea).

**Principle**

Iron is an important mineral in our diets. Iron in foods is in the form of either iron (II) or iron (III). The test for the iron (III) ion is done in solution and is based on the following reaction:

$$Fe^{3+}(aq) + SCN^{-}(aq) \rightarrow Fe(SCN)^{2+}(aq)$$

The deep read colour of the iron (III) thiocyanate ion is directly related to the concentration of iron (III) originally present in the solution. In this test, all iron in the original sample is converted to iron (III) ions or is not determined through the thiocyanate test.

**Materials/Equipment:**

$Fe(NO_3)_3$ (0.001 M) solution in 0.1 M HCl

KSCN (0.1 M)

HCl (0.1 M, 2.0M)

Spectrophotometer or UV-VIS, cuvettes

various food items (raisins, cereals, peas—cooked/uncooked, etc.)

**Procedure**

**Preparation of the Standards:**

Prepare the following solutions in five test tubes. Thoroughly mix each with a stirring rod. Add 2.5 mL of 0.1 M KSCN to each test tube. Mix well. A red color should result from the formation of the $FeSCN^{2+}$ ion.

| Test Tube | 0.001 M $Fe(NO_3)_3$ (mL) | $H_2O$ (mL) | Concentration (mM/L) |
|---|---|---|---|
| 1 | 0 | 20 mL **0.1 M HCl** | 0.00 |
| 2 | 5 | 15 | 0.25 |
| 3 | 10 | 10 | 0.50 |
| 4 | 15 | 5 | 0.75 |
| 5 | 20 | 0 | 1.00 |

**Preparation of the Food Samples:**

Weigh about 2.5 g of the solid food and place in a crucible. Heat the crucible until the food sample has turned to ash (approximately 5-20 minutes depending on the food sample used). Cool the ash and transfer into a small beaker. Add 10 mL of 2.0 M HCl and 10 mL distilled water; stir and Filter the mixture; collect the filtrate. Add 2.5 mL of 0.1 M KSCN. Mix well.

**Absorbance measurement:**

Use a UV-VIS spectrophotometer at a wavelength of 458 nm. Place standard solution and food solution into a separate cuvette. Record the absorbance of each solution. Prepare a standard curve (Beer's Law) of the standard concentrations vs. absorbance. Find out the concentration of the food samples Using standard curve of iron (III).

**Result:**

The amount of Fe(III) present in food sample----------

## 1. COPPER(II) – EDTA COMPLEX

**Aim:**

To find out the ratio of metal ion and ligand of the Copper(II) – EDTA complex.

**Chemicals Required**: $CuSO_4.5H_2O$, EDTA

**Instrument:** UV- Visible Spectrophotometer

**Procedure:**

0.005 M solution of $CuSO_4.5H_2O$ in 250 mL of distilled water is prepared by dissolving 0.3120 g pure crystals of $CuSO_4$. Similarly 0.005 M solution of EDTA is prepared. Instrument is standardized using water as reference to measure absorbance as zero at the wavelength of 740 nm. 3 mL of $CuSO_4$ and 27 mL of EDTA are mixed in a small beaker and used as sample no. 1. The solution is taken in cuvette and absorbance is measured at 740 nm. Similarly, absorbance readings for nine different concentrations varying from 3:27 to 27:3 with respect to $CuSO_4$ solution at the same wavelength has been measured. The absorbance obtained is plotted against the volume of solution used in the complex formation. The plot shows a parabolic graph and using the maximum absorbance value from the graph and the corresponding volume of reactant, value of 'n' is calculated.

| Solution A (mL) | Solution B (mL) | Mole fraction of reactant B | Absorbance value |
|---|---|---|---|
| 3 | 27 | 0.9 | |
| 6 | 24 | 0.8 | |
| 9 | 21 | 0.7 | |
| 12 | 18 | 0.6 | |
| 15 | 15 | 0.5 | |
| 18 | 12 | 0.4 | |
| 21 | 9 | 0.3 | |
| 24 | 6 | 0.2 | |
| 27 | 3 | 0.1 | |

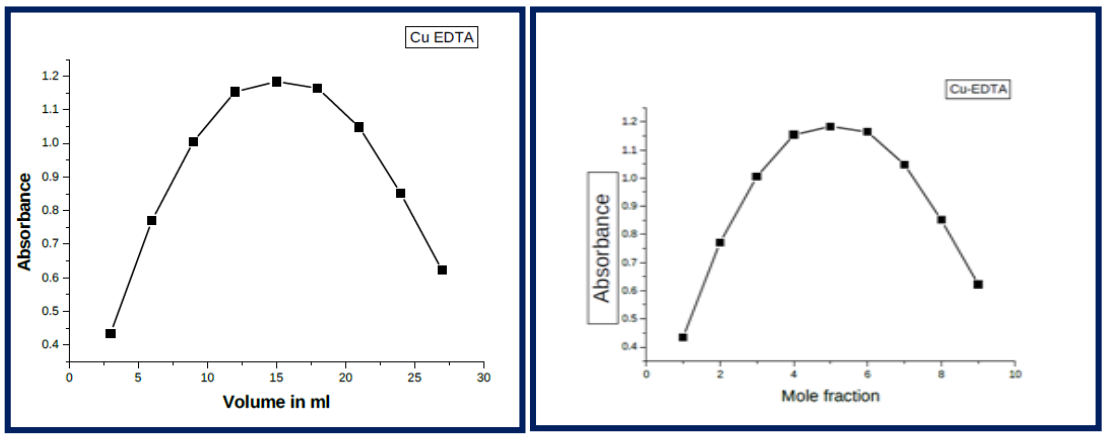
$$X_L = \frac{V_L}{V_L + V_M^\circ}$$

Where $V_L$- the volume of the titrant added at each;

$V_{MO}$- the initial volume of metal titrant.

The graph 1a & 1b shows the photometric curve at 740 nm by titrating solution A with solution B, the graphs are given below.

**Model graph:**




**Report:**

The plot exhibits at $X_{Cu^{2+}}$= 0.5, it indicates the formulation of 1:1complex.

**Note:** If XL (n) = 15/30=0.5

The value of n=0.5 indicates that in the $CuSO_4.5H_2O$ & EDTA complex the metal ligand ratio is 1:1

## 2. COBALT – HYDRAZIDO COMPLEX

Aim:To find out the ratio of metal ion and ligand of the Cobalt – Hydrazido complex

Chemicals Required: $CoCl_2.6H_2O$, $NH_2NH_2 \cdot H_2O$

**Instrument:** UV- Visible Spectrophotometer.

**Procedure:**

0.005 M solution of $CoCl_2.6H_2O$ in 250 mL of distilled water is prepared by dissolving 0.2967 g pure crystals of $CoCl_2$. Similarly 0.005 M solution of $NH_2$ $NH_2.H_2O$ is prepared. Instrument is standardized using water as reference to measure absorbance as zero at the wavelength of 635 nm. 3 mL of $CoCl_2.6H_2O$ and 27 mL of $NH_2NH_2 \cdot H_2O$ are mixed in a small beaker and used as sample no. 1. Solution and is used as sample no. 1. The solution is taken in cuvette and absorbance is measured at 635 nm. Similarly the absorbance readings for nine different concentration ratio varying from 3:27 to 27:3 with respect to $CoCl_2.6H_2O$ solution at the same wavelength. The absorbance obtained is plotted vs volume of an each solution used in the complex formation. The plot shows a parabolic graph and using the maximum absorbance value from the graph and its corresponding volume of reactant, value of 'n' is calculated.

| Solution A (mL) | Solution B (mL) | Mole fraction of reactant B | Absorbance value |
|---|---|---|---|
| 3 | 27 | 0.9 | |
| 6 | 24 | 0.8 | |
| 9 | 21 | 0.7 | |
| 12 | 18 | 0.6 | |
| 15 | 15 | 0.5 | |
| 18 | 12 | 0.4 | |
| 21 | 9 | 0.3 | |
| 24 | 6 | 0.2 | |
| 27 | 3 | 0.1 | |

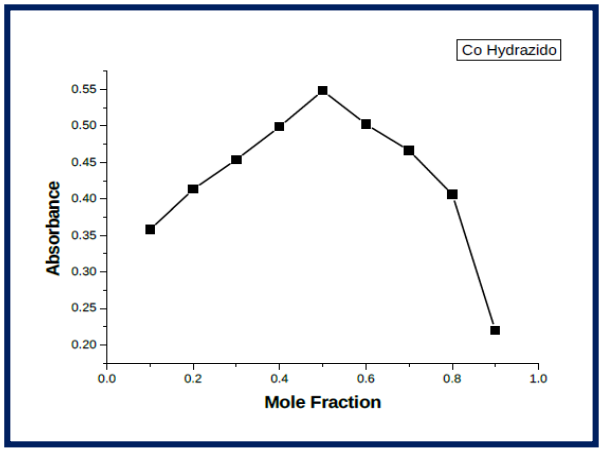
**Calculations:**

$$X_L = \frac{V_L}{V_L + V_M^{\circ}}$$

Where $V_L$ - is the volume of the titrant added at each

$V_M^{o}$ - is the initial volume of metal titrant.

The graph 2a & 2b shows the photometric curve at 635nm by titrating solution A with solution B, the graphs are given below.




**Report:**

The plot exhibit at $X_{Co^{2+}}$ = , it indicates the formulation of 1:1 complex.

## 3. ZINC – EDTA COMPLEX

**Aim:** To find out the ratio of metal ion and ligand of the Zn – EDTA Complex

**Chemicals Required:** $ZnSO_4 .7H_2O$, EDTA

**Instrument:** UV- Visible Spectrophotometer

**Procedure:**

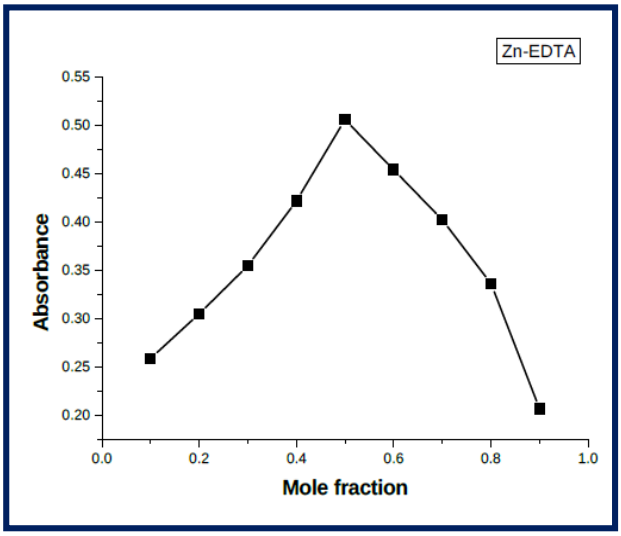
0.005 M solution of $ZnSO_4 .7H_2O$ in 250 mL of distilled water is prepared by dissolving 0.3344 g pure crystals of $ZnSO_4$. Similarly 0.005 M solution of EDTA is prepared. Instrument is standardised using water as reference to measure absorbance as zero at the wavelength of 240 nm. 3 ml of $ZnSO_4$ and 27 Ml of EDTA are mixed in a beaker and is used as sample no. 1. The solution is taken in cuvette and absorbance is measured at 740 nm. Similarly the absorbance readings for nine different concentration ratios varying from 3:27 to 27:3 with respect to $ZnSO_4$ solution at the same wavelength are measured. The absorbance obtained is plotted against the volume of either solution used in the complex formation. The plot shows a parabolic graph and using the maximum absorbance value from the graph and the corresponding volume of reactant, value of 'n' is calculated.

| Solution A (mL) | Solution B (mL) | Mole fraction of reactant B | Absorbance value |
|---|---|---|---|
| 3 | 27 | 0.9 | |
| 6 | 24 | 0.8 | |
| 9 | 21 | 0.7 | |
| 12 | 18 | 0.6 | |
| 15 | 15 | 0.5 | |
| 18 | 12 | 0.4 | |
| 21 | 9 | 0.3 | |
| 24 | 6 | 0.2 | |
| 27 | 3 | 0.1 | |

$$X_L = \frac{V_L}{V_L + V_M^o}$$

Where VL - is the volume of the titrant added at each

$V_M^o$ - is the initial volume of metaltitrant.




**Report:**

The plot exhibit at $X_{Zn^{2+}}$= ------, it indicates the formulation of ---:--complex

## 4. CHLORO CUPRATE COMPLEX

**Aim:** To find out the ratio of metal ion and ligand of the Chloro Cuprate complex

**Chemicals Required:** $CuSO_4 .5H_2O$,KCl

**Instrument:** UV- Visible Spectrophotometer

**Procedure:**

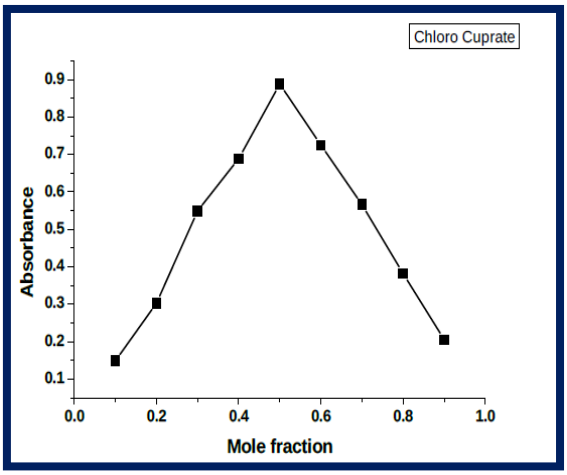
0.005 M solution of $CuSO_4 .5H_2O$ in 250 mL of distilled water is prepared by dissolving 0.4653 g pure crystals of $CuSO_4$.Similarly 0.005 M solution of KCl is prepared. Instrument is standardized using water as reference to measure absorbance as zero at the wavelength of 275 nm.3 mL of $CuSO_4$ and 27 Ml of KCl are mixed in a small beaker and used as sample no.1. The solution is taken in a cuvette and absorbance is measured at 275 nm. Similarly the absorbance readings for nine different concentration ratios varying from 3:27 to 27:3 with respect to $CuSO_4$ solution at the same wavelength are measured. The absorbance obtained is plotted against the volume of either solution used in the complex formation. The plot shows a parabolic graph and using the maximum absorbance value from the graph and the corresponding volume of reactant, value of 'n' is calculated.

| Solution A (mL) | Solution B (mL) | Mole fraction of reactant B | Absorbance value |
|---|---|---|---|
| 3 | 27 | 0.9 | |
| 6 | 24 | 0.8 | |
| 9 | 21 | 0.7 | |
| 12 | 18 | 0.6 | |
| 15 | 15 | 0.5 | |
| 18 | 12 | 0.4 | |
| 21 | 9 | 0.3 | |
| 24 | 6 | 0.2 | |
| 27 | 3 | 0.1 | |

$$X_L = \frac{V_L}{V_L + V_M^{\circ}}$$

Where VL - is the volume of the titrant added at each

$V_M^{o}$ - is the initial volume of metal titrant.




**Report:**

The plot exhibit at $X_{Cu^{2+}}$= ----, it indicates the formulation of 1:1complex.

## DETERMINATION OF MOLECULAR WEIGHT OF A POLYMER BY OSTWALD VISCOMETER

### AIM

To determine the molecular weight of a polymer by viscosity average method.

### PRINCIPLE

Viscosity average method is based on the flow behaviour of the polymer solutions . According to Mark – Hawnik equation, the intrinsic viscosity of a polymer is given as

$$[\eta]_{int} = KM^a$$

Where,

M = molecular weight of the polymer

K & a are constants for a particular polymer – solvent system

$\eta$ = Intrinsic viscosity = $[\eta_{sp}/C]_{C=0} = [\eta_r/C]_{C=0}$

$\eta_{sp}$= specific viscosity = $\eta_r - 1$

$\eta_r$ = relative viscosity = $\eta/\eta_0 = t / t_0$

Since accurate measurement of absolute viscosity is a difficult task, relative viscosity is taken into account.

$\eta$ = Viscosity of the polymer solution

$\eta_0$ = Viscosity of the pure solvent

t = flow time of the polymer solution

$t_0$ = flow time of the pure solvent

The flow time of the polymer solution (t) and that of the pure solvent ($t_0$) are found experimentally and substituted to get $\eta_{sp}$ , $\eta_r$ and thus $[\eta]_{int}$.

Knowing K & a, molecular weight of the polymer solution is calculated.

DP = M / m (M= mol.wt of polymer, m = mol. wt of monomer)

**PROCEDURE**

Accurately 1g of polyvinyl pyrrolidone is weighed, dissolved in water and made up to 100 mL (1dl) in a standard flask. From the bulk, solutions of conc. 0.1 g/dl, 0.2 g/dl, 0.3 g/dl. 0.4 g/dl and 0.5g/dl are prepared using the relation $V_1N_1 = V_2N_2$

[E.g. X * 1g / dl = 0.2g / dl * 100ml,

Where X = volume of bulk solution to be taken for preparing 100ml of 0.2g/dl Polymer solution]

A well cleaned Ostwald viscometer is rinsed with water and filled with 10 mL of distilled water, Water in the viscometer is sucked into the upper bulb using a rubber bulb. The time taken for water to flow from the upper mark to the lower mark is measured with a stop clock and noted as $t_0$.

Water from the viscometer is drained completely and 10 mL of the polymer solution of conc. 0.1 g/dl is poured in the viscometer. The flow time of the polymer solution is found and noted as t. The procedure is repeated with the other solutions of the polymer.

From the values of t and $t_0$ ,$\eta_r$ / C and $\eta_{sp}$ / C are calculated and graphs with $\eta_{sp}$ / C Vs C and ln$\eta_r$ / C Vs C are drawn. The straight lines obtained are extrapolated to zero concentration. The intercept values are equal to $[\eta]_{int.}$ From $[\eta]_{int}$ molecular weight of the polymer (M) is calculated using the formula $[\eta]_{int} = KM^a$ and the table.

From the values of M and m , degree of polymerization can be calculated.

Solvent used = Water

K of the polymer solvent system = ----------

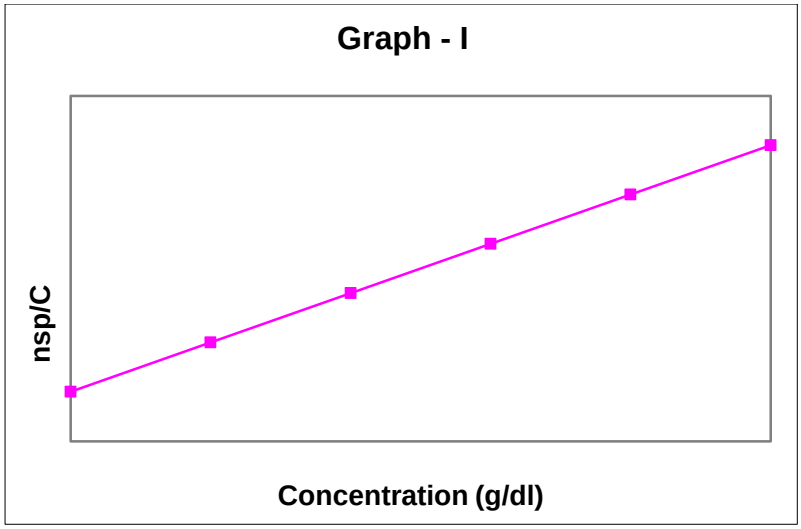
a of the polymer-solvent system = ----------

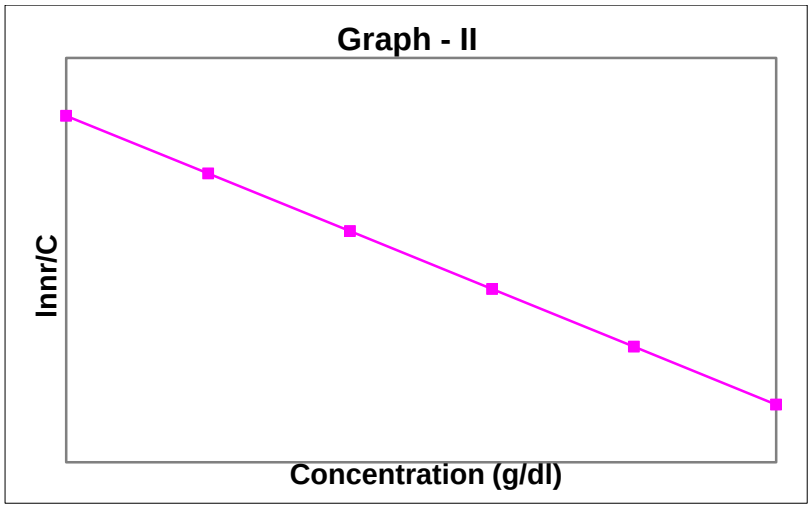
volume of liquid taken for finding the flow time = 10 ml

flow time of the solvent ( $t_0$ ) = ----------

| s.no | Conc. g/dl (%) | Flow time (t)sec | $\eta_r = t / t_0$ | $\ln\eta_r$ | $\ln\eta_r / C$ | $\eta_{sp}=\eta_r -1$ | $\eta_{sp}/ C$ |
|---|---|---|---|---|---|---|---|
| | | | | | | | |

| S. No. | Polymer | Solvent | $K*10^{-5}$ (g/ml) | a |
|---|---|---|---|---|
| 1. | Polyvinyl alcohol | Water | 45.3 | 0.64 |
| 2. | Polyvinyl pyrrolidone | Water | 39.3 | 0.59 |
| 3. | Polystyrene (atactic) | Benzene | 11.5 | 0.73 |
| 4. | Polystyrene (isotactic) | Benzene | 10.6 | 0.735 |




**RESULT:**

The molecular weight of the given polymer =