

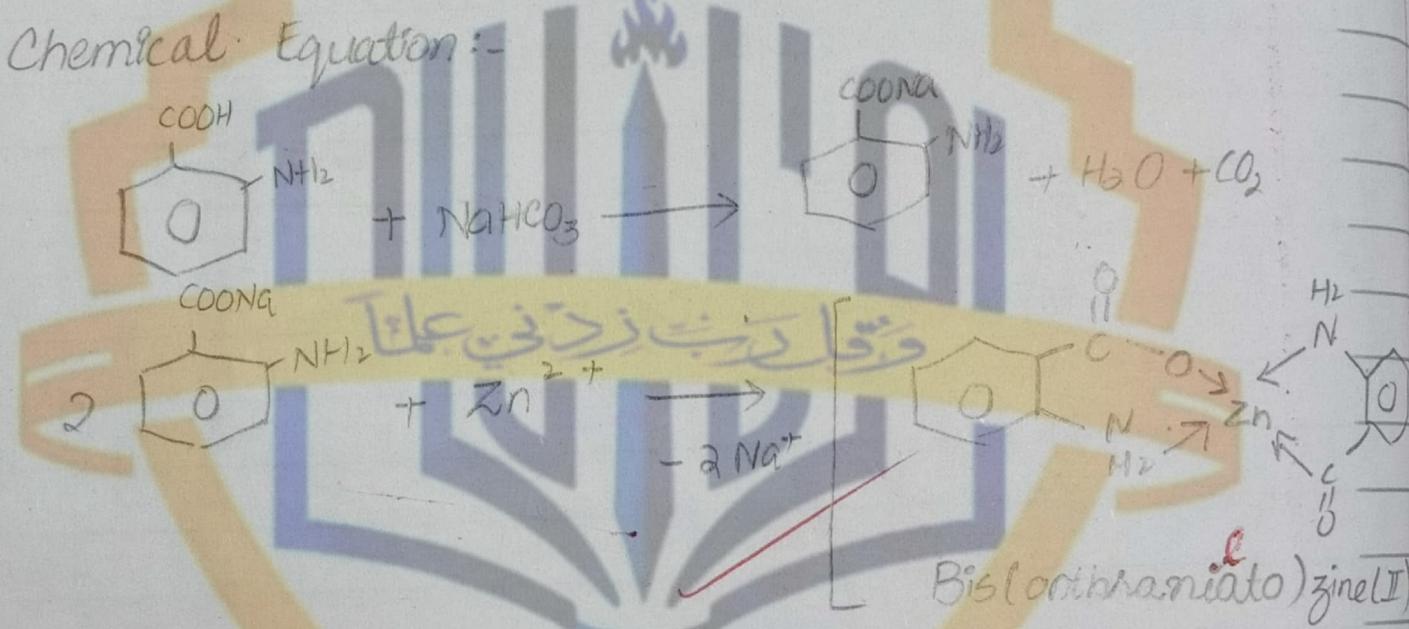
## Experiment # 01

Determine the amount per  $\text{dm}^3$  of  $\text{Zn}^{2+}$  ions in the sample gravimetrically using anthranilic acid.

### Apparatus:-

Filter paper, Beaker, Funnel, pipette, Measuring Balance, stirrer, Measuring flask.

### Chemical Equation :-



### Chemicals and Solutions:

i) 3% Sodium anthranilic acid : 5g of  $\text{NaHCO}_3$

was dissolved in water in  $100\text{cm}^3$  beaker.

Then 3g of anthranilic acid was dissolved in  $250\text{cm}^3$  beaker.

# Experiment # 01

Determine

The amount per dm<sup>3</sup> of Zn<sup>2+</sup> ions in the sample gravimetrically using anthranilic acid.

## Theory :-

### Gravimetric analysis:-

Gravimetry is the Greek word "gravi" means a high molecular weight insoluble species "meter" to measure.

### Definition:-

It is a process of isolating and weighing a definite compound of an element in as pure form as possible. The weight of an element or metal ion calculated from molecular formula of a compound and the atomic weight of the constituent element.

### Solubility of metal complex:-

The Solubility of metal complex in a given organic reagent is primarily dependent upon the solubility of its metal complex. If the ligand is highly soluble in water and the complex

ii) 1% Metal ion Solution : 1g of  $ZnSO_4$  was dissolved in  $100\text{ cm}^3$  of water. The 1% metal ion solution was prepared.

$$\text{Weight of filter paper } (W_1) = 1.42\text{ g}$$

$$\text{Weight of filter paper + precipitates } (W_2) = 1.66\text{ g}$$

$$\text{Weight of precipitates } (W') = W_2 - W_1$$

$$= (1.66 - 1.42)\text{ g}$$

$$= 0.24\text{ g}$$

According to formula :-

$337.39\text{ g of zinc anthranilate complex contain } \frac{65.37}{337.39}\text{ g Zinc}$

$1\text{ g of zinc anthranilate complex contain } \frac{65.37}{337.39}\text{ g Zinc}$

$0.24\text{ g of zinc anthranilate complex contain } \frac{65.37 \times 0.24}{337.39}\text{ g Zinc}$

Amount per  $\text{dm}^3$  :  $= 0.046\text{ g}$

So,

$20.0\text{ cm}^3$  of solution contain  $= 0.046\text{ g Zinc}$

Obtain is insoluble in<sup>\*</sup> than the amount of organic reagent adsorbed on precipitates will be less and easily removed by washing. Some of organic reagents are practically soluble in water. So their  $\text{Na}^+$ ,  $\text{K}^+$  or  $\text{NH}_4^+$  salts are used.

### Types :-

There are four types of gravimetric analysis.

- Physical gravimetry.
- Thermogravimetry.
- Precipitative gravimetric analysis.
- Electro deposition.

### Physical gravimetry:-

It involves the physical separation and classification of matter in environmental samples based on volatility and particle size.

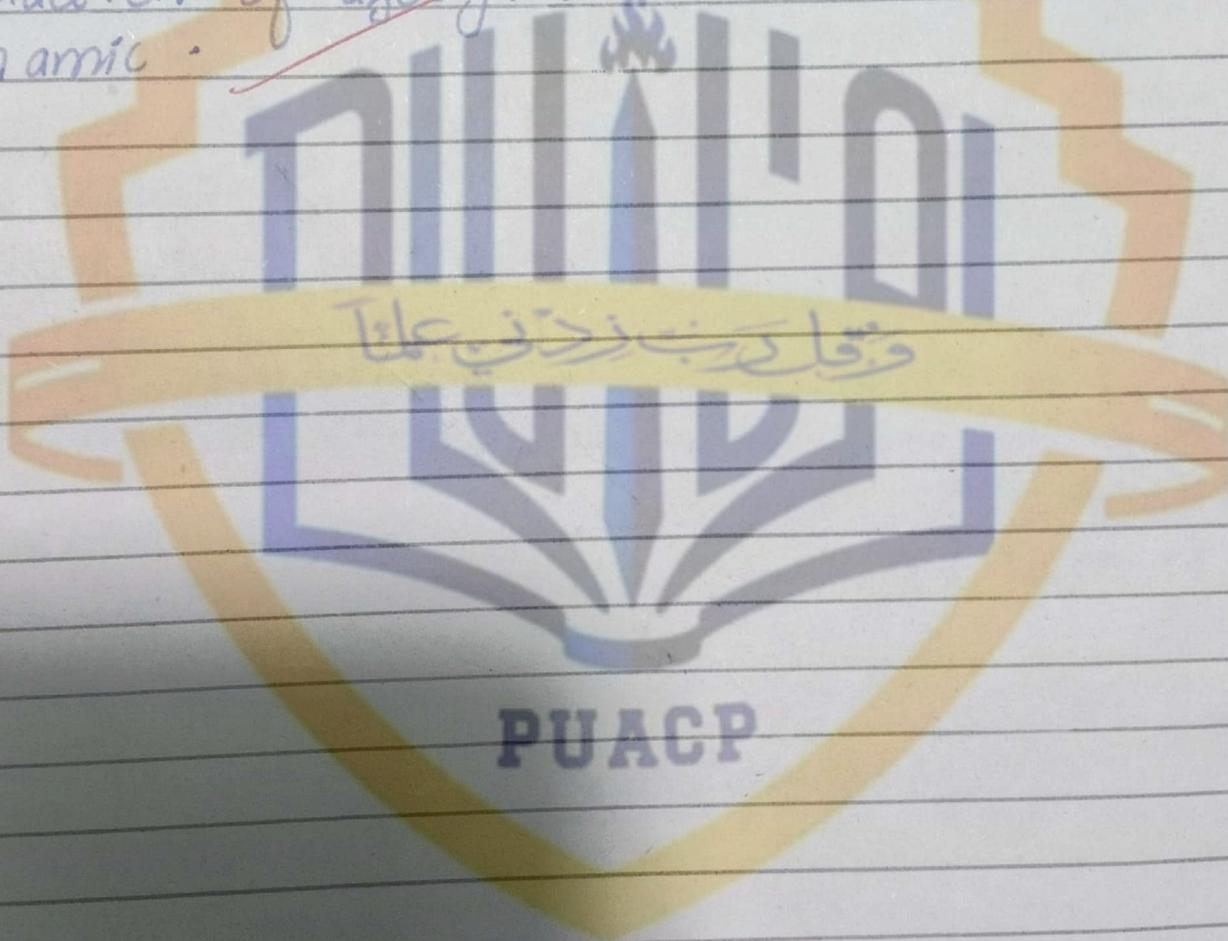
e.g., total suspended solids in benzene.

- It is odourless.
- Its solubility in water is  $0.572 \text{ g/100 mL}$  (25°C)

## Uses :-

It is used for gravimetric analysis ( $\text{Zn}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Co}^{2+}$ ). It is intern ediate in the production of azodynes and saccharin.

Fenamic.



$1\text{cm}^3$  of solution contain zinc =  $\frac{0.046}{20}$

$$1000 \text{ cm}^3 \text{ of solution contain zinc} = \frac{0.046 \times 1000}{90} \\ = 2.3 \text{ g/l dm}^3$$

Results:-

The amount of per  $\text{dm}^3$  of  $\text{Zn}^{2+}$  in the given sample is  $2.3 \text{ g/l dm}^3$ .

PUACP

ii)

## Thermogravimetry :-

The samples are heated and the changes in sample mass are recorded. Volatile solid analysis is an important example of gravimetric analysis.

iii)

## Precipitative gravimetry :-

The chemical precipitation of an analyte occurs in the precipitative gravimetry. The important application of this technique in environmental field is the analysis of Sulphite.

iv)

## Electro deposition :-

It involves the electrochemical reduction of metal ions at a cathode and simultaneous deposition of the ions on the cathode.

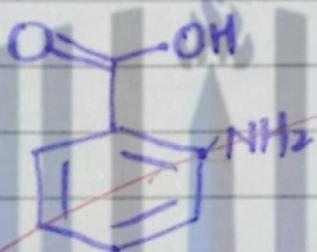
## Steps in gravimetric analysis:-

The steps commonly followed in gravimetric analysis are:

- i) Preparation of a solution containing a known weight of sample.

- ii) Separation of the desired constituent.
- iii) Weighing the isolated constituent.
- iv) Computation of amount of the particular constituent in the sample from the observed weight of sample & the isolated substance

## Anthranilic acid :-



Molecular formula : C<sub>7</sub>H<sub>7</sub>NO<sub>2</sub>

Boiling point : 200°C

Melting point : 146-148°C

Density : 1.412 g/cm<sup>3</sup>

## Properties :-

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- Anthranilic acid is a white solid when pure although appears yellowish commercially.
- It is soluble in ethanol, ether, ethyl ether and Slightly soluble in Benzene.
- It is odourless

• Its solubility in water is  $0.572\text{ g/l}$   $100\text{ mL}$  ( $25^\circ\text{C}$ )

## Uses :-

It is used for gravimetric analysis ( $\text{Zn}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Co}^{2+}$ ) It is intermediate in the production of azodyes and saccharin.

Fenamic acid is a derivative of anthranilic acid which in turn is a nitrogen iso stereoisomer of Salicylic acid, which is the active metabolite of aspirin.

## Zinc ions-

Zinc ion or Zinc cation has molecular formula of  $\text{Zn}^{2+}$ .

Its molecular weight is 65.4.

$\text{Zn}^{2+}$  is a divalent metal cation a zinc cation and a monatomic dication.

$\text{Zn}^{2+}$  is required for catalytic activity as well for structural stability of enzyme.

## Procedure:-

- In the first step, 3% sodium anthranilic acid was prepared.
- The 1% Zinc ion  $Zn^{2+}$  solution was prepared in next step.
- ~~20 cm<sup>3</sup> solution of  $Zn^{2+}$  was taken and 3% anthranilic acid was added till the formation of precipitates.~~
- It was warmed to avoid Co-agulation of precipitate.
- Then the above solution was cooled.
- When it was cooled, it was filtered on the filter paper.
- The precipitate formed was washed by water. Finally, the precipitates were dried in oven at  $110^{\circ}C$  till weight becomes constant.

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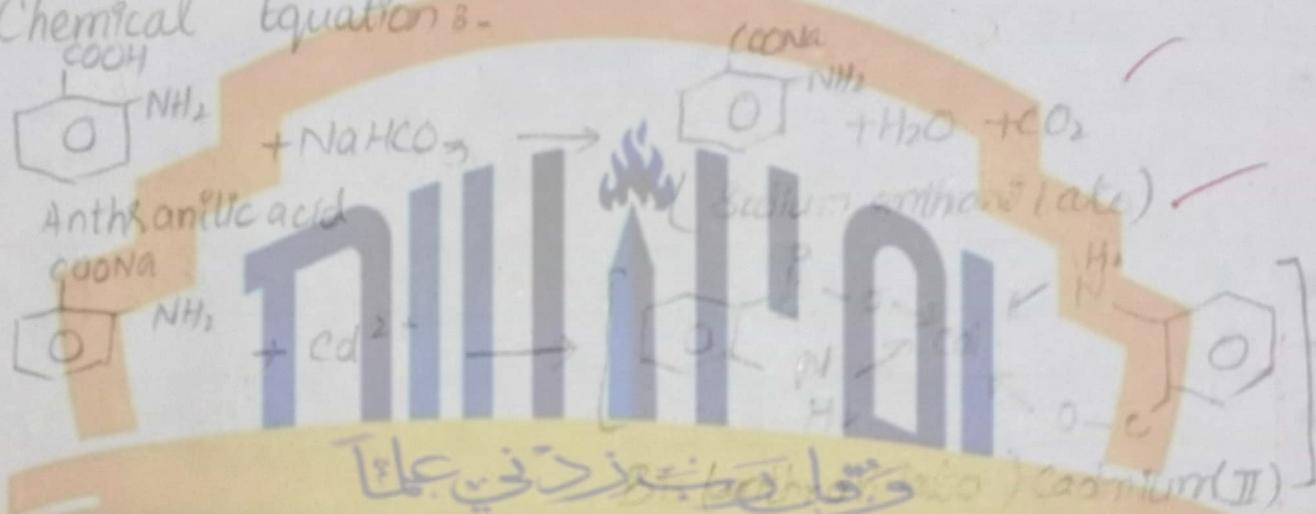
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## Experiment no 2 :-

### Apparatus :-

Filter paper, Beaker, Funnel, Pipette, Measuring Balance, Stirrer, Measuring flask.

### Chemical Equation :-



### Chemicals and solutions:

i) 3% Sodium orthanilate solution.

ii) 1%  $\text{CdCl}_2$  1/10 solution.

### Calculations:

Weight of filter paper =  $W_1 = 0.94\text{g}$

Weight of filter paper =  $W_2 = 1.75\text{g}$

Precipitates

Weight of precipitates =  $W' = W_2 - W_1$

$$= 1.75 - 0.94$$

$$W' = 0.81\text{g}$$

## Experiment # 02

Determine the amount per  $\text{dm}^3$  of  $\text{Cd}^{2+}$  ion in the sample gravimetrically by using antharilic acid.

### Theory :-

Theory has already given in experiment no 10.

### Cadmium ion :-

$\text{Cd}^{2+}$  is a ~~trivalent~~ divalent metal cation.

Its molecular formula is  $\text{Cd}^{2+}$ .

It has a role as cofactor.

It is solid at room temperature.

The melting point of  $\text{Cd}^{2+}$  is  $321^\circ\text{C}$ .

The molecular weight of  $\text{Cd}^{2+}$  is  $1112.41 \text{ g/mol}$ .

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### Uses :-

Cadmium combines with heavy metals to yield alloys.

Cadmium used in batteries as anode

So,

384.41 g of Cd anthranilate complex contain cadmium

1 g of Cd anthranilate complex contain cadmium =  $\frac{112.4}{384.41}$

0.81 g of Cd anthranilate complex contain cadmium =  $\frac{112.4}{384.41} \times 0.81$

So,

20 cm<sup>3</sup> of sample solution contain Cd<sup>2+</sup> = 0.236 g  
of Cd<sup>2+</sup> ions.

1 cm<sup>3</sup> of sample solution contain Cd<sup>2+</sup> =  $\frac{0.236}{20}$

1000 cm<sup>3</sup> of sample solution contain Cd<sup>2+</sup> ions =

$$\frac{0.236}{20} \times 1000$$

$$= 11.8 \text{ g/dm}^3$$

Result:-

Cd<sup>2+</sup> in

The amount per dm<sup>3</sup> of given sample is 11.8 g/dm<sup>3</sup>.

*Graphical*  
*Method*  
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## Procedure :-

- i) In first step , 3% sodium anthranilic acid was prepared by adding 5g of  $\text{NaHCO}_3$  in 100mL of water and adding 3g of anthranilic acid in 250 mL of beaker.
- ii) 1%  $\text{CdCl}_2$  solution was prepared by adding 1g of  $\text{CdCl}_2$  in 100 ml of distilled water .
- iii) 20ml of  $\text{CdCl}_2$  solution was taken in 250mL beaker.
- iv) 3% sodium anthranilic acid sol was added in the beaker  ~~Till the complete formation of precipitates -~~
- v) The precipitates was dried at  $210^\circ\text{C}$  in oven.
- vi) The amount of  $\text{Cd}^{+2}$  ions in the sample was calculated.

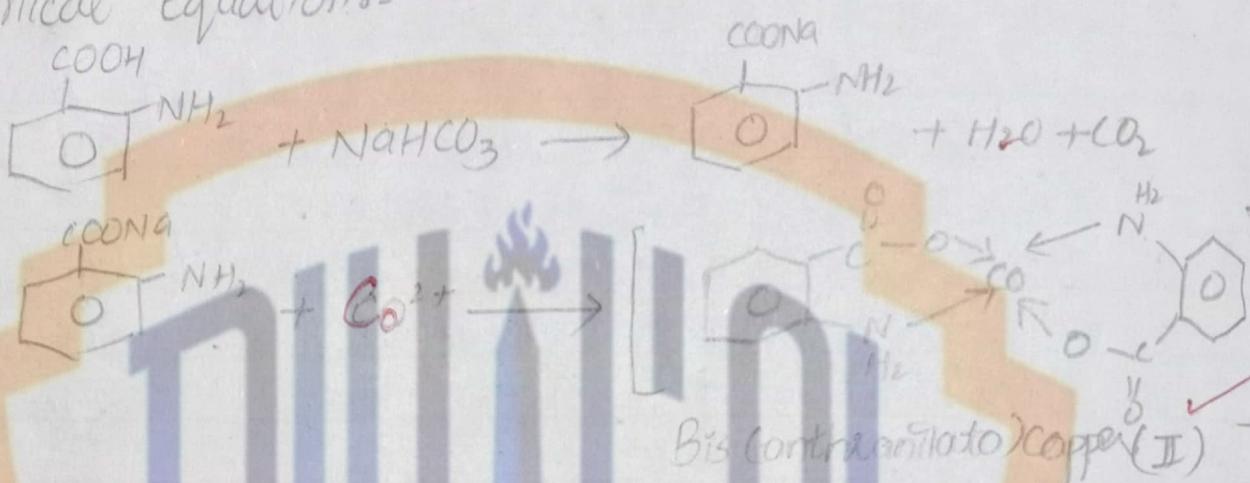
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## Experiment #3 :-

### Apparatus :-

Filter paper, Beakers, Funnel, Pipette,  
Measuring Balance, Measuring flask, Stirrer-

### Chemical Equations :-



### Chemicals and solutions:-

- i) 3% Na anthranilate solution: 5g of  $\text{NaHCO}_3$  was dissolved in  $100 \text{ cm}^3$  of water. Then 3g of anthranilic acid was added in  $250 \text{ cm}^3$  beaker. ✓
- ii) 2% Metal ion solution: 1g of  $\text{CoCl}_2$  was dissolved in 100ml of  $\text{H}_2\text{O}$ . ✓

### Calculations :-

### **PUACP**

Weight of filter paper =  $H_1 = 0.94\text{g}$   
 weight of filter paper + precipitates =  $H_2 = 1.16\text{g}$

## Experiment # 03

Determine the amount per dm<sup>3</sup> of Co<sup>2+</sup> in the given sample gravimetrically using anthranilic acid.

### Theory :-

no 1.

Theory has already given in experiment

### Cobalt<sup>2+</sup>

The molecular formula is Co<sup>2+</sup>. Cobalt<sup>2+</sup> is a divalent metal cation a cobalt cation and a monoatomic dication.

The melting point of Co<sup>2+</sup> is 1495 °C.

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Weight of precipitates =  $W_1 = 0.229$

331 g of co-anthranilate complex  
contain  $\text{CO}^{2+}$  = 59 g

1g of co-anthranilate complex contain

$$\text{CO}^{2+} = \frac{59}{331}$$

0.22 g of Co anthranilate complex contain  $\text{CO}^{2+} = \frac{39}{\frac{59}{331}} \times 0.22$

$$0.0399$$



20  $\text{cm}^3$  of sample solution contain  $\text{CO}^{2+} = 0.0399$

1  $\text{cm}^3$  of sample solution contain  $\text{CO}^{2+} = 0.039$

20.

1000  $\text{cm}^3$  of sample solution contain  $\text{CO}^{2+} = \frac{0.039}{20} \times 1000$

20

$$1.93 \text{ g/dm}^3$$

Result:

The amount of  $\text{CO}^{2+}$  per  $\text{dm}^3$  of  $\text{Co}^{2+}$  ion in the given sample is

$$1.93 \text{ g/dm}^3$$

Prepared by  
2021

## Procedure:-

- In the first step, 20mL  $\text{CO}_2$  solution was taken.
- 3% sodium anthranilate solution was added in the solution of  $\text{CaCl}_2$ .
- Addition of  $\text{CO}_2$  solution was continued till the formation of precipitates.
- Preheated filter paper was used for the filtration of precipitates.
- Washed the precipitates using water.
- Precipitates were dried.
- The dried precipitates were weighed and calculations was taken after conducting data.

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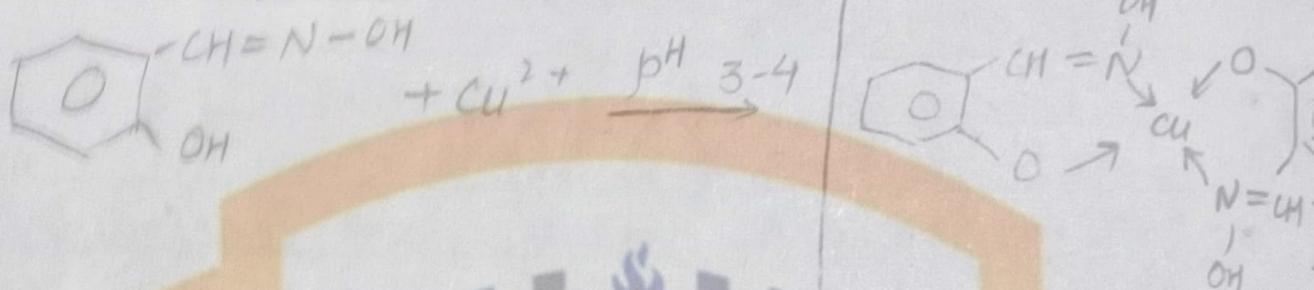
**PUACP**

## Experiment #4

### Apparatus

Filter paper, Beaker, Funnel, Pipette,  
Measuring balance, Measuring flask, Stirrer.

### Chemical Equation



### Chemicals and Solutions:-

- i) 1% Salicylaldoxime solution: 1g of salicylaldoxime was dissolved in 15cm<sup>3</sup> of ethanol and dilute upto 10cm<sup>3</sup> measuring flask with distilled water.
- ii) 1% Cu<sup>2+</sup> ion solution: was prepared by dissolving 1g of CuCl<sub>2</sub> in 100ml of distilled water.

### Calculations:-

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$$\text{Weight of filter paper } W_1 = 0.93\text{g}$$

$$\text{Weight of filter paper + Precipitate} = W_2 = 1.22\text{g}$$

$$\begin{aligned} \text{Weight of precipitate} &= W' = W_2 - W_1 \\ &= 0.29\text{g} \end{aligned}$$

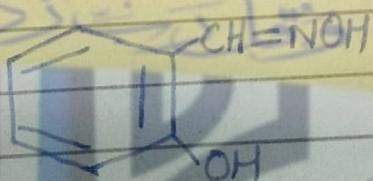
## Experiment # 04

Determine the percentage of  $\text{Cu}^{2+}$  ion in the given sample gravimetrically using Salicyaldoxime.

### Theory :-

~~Theory has already given in experiment no 1.~~

### Salicyaldoxime :-



Molecular Weight = 137.14 g/mol  
 Melting point = 570 °C  
 Molecular formula =  $\text{C}_7\text{H}_7\text{NO}_2$

### Properties :-

- It exists as white crystalline solid.
- It is sparingly soluble in water.
- It is freely soluble in  $\text{CaH}_5\text{OH}$ , diethyl ether and acetone.

So,

335.54 g of copper Salicylaldoxime = 63.5 g  
complex contain  $\text{Cu}^{2+}$

1g of Cu-Salicylaldoxime complex =  $\frac{63.5}{335.4}$  g  
contain  $\text{Cu}^{2+}$

0.2 g of Cu-Salicylaldoxime =  $\frac{63.5}{335.4} \times 0.29$   
contain copper ( $\text{Cu}^{2+}$ ) ion

So,

20cm<sup>3</sup> of sample solution contain  $\text{Cu}^{2+}$  = 0.05 g

10cm<sup>3</sup> of sample solution contain =  $\frac{0.05}{20}$

100cm<sup>3</sup> of sample solution contain  $\text{Cu}^{2+}$

$$= \frac{0.05 \times 100}{20}$$

$$= \boxed{25\%}$$

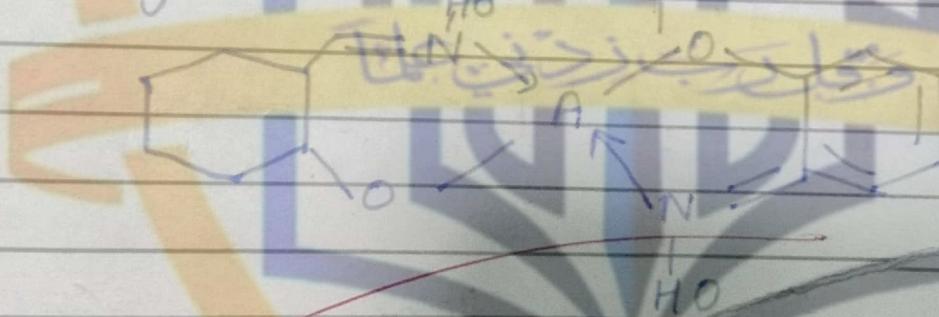
It is readily decompose in solution form (so its freely prepared solution is used).

## Uses :-

It reacts with many metals and gives intense colored complex.

With  $\text{Cu}^{2+}$  it gives green yellow precipitates in the presence of  $\text{CH}_3\text{COOH}$  at  $\text{pH} = 2.6$ .

With  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$  and  $\text{Pd}^{2+}$  it forms the complex having the structural formula -



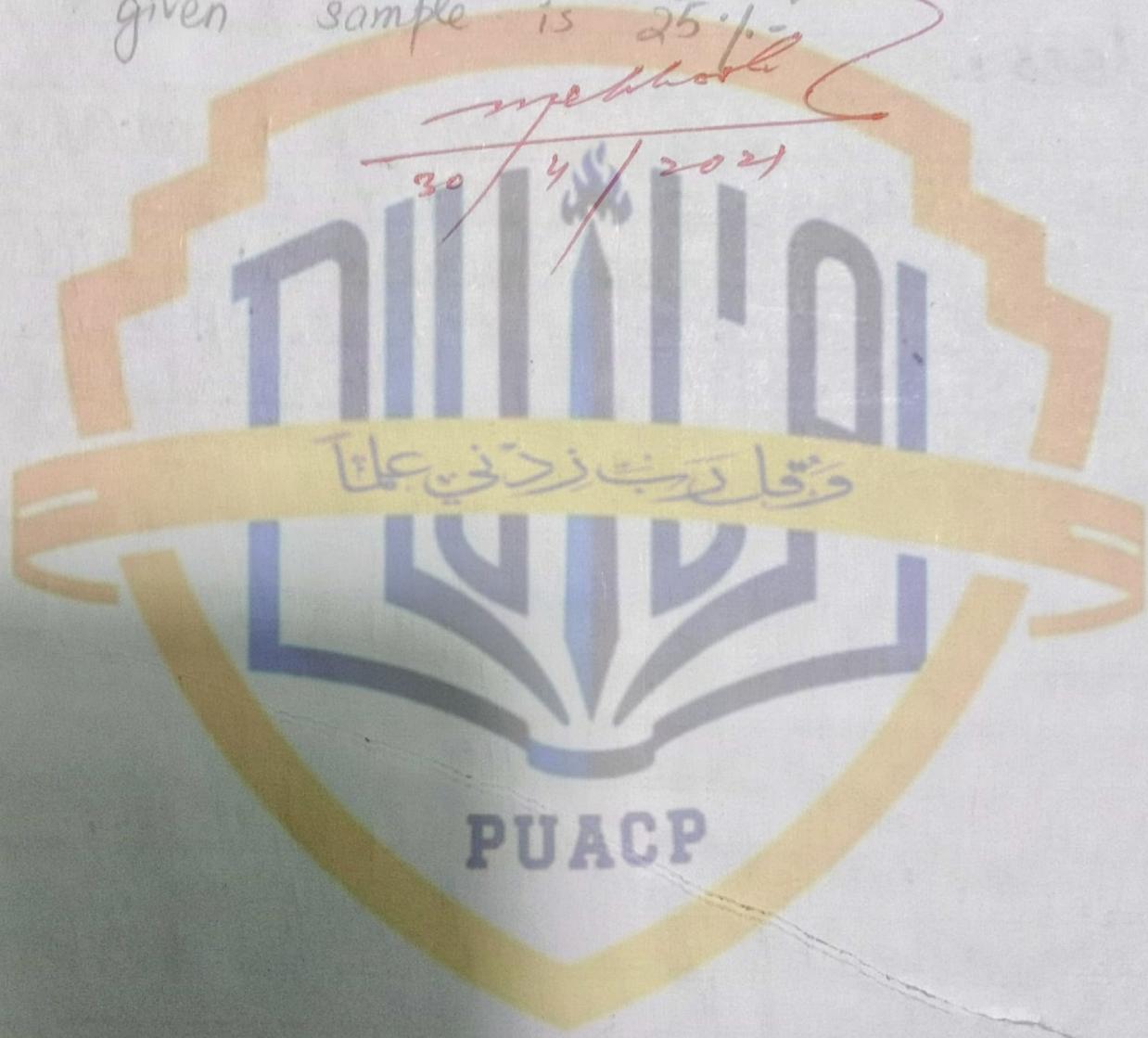
With  $\text{Pd}^{2+}$  it forms yellow precipitates.

With  $\text{Ni}^{2+}$  it forms green complex.

For volumetric and amphoteric determination of  $\text{Cu}^{2+}$  and  $\text{Pd}^{2+}$ .

Result :

The percentage of  $\text{Cu}^{2+}$  in the  
given sample is 25%.



## Procedure :-

20ml of metal ion  $\text{Cu}^{2+}$  ion solution was taken in  $250 \text{ cm}^3$  beaker.

The pH was adjusted between 3 to 4 by using Acetic acid.

~~Salicylaldoxime~~ solution was added in above solution where pH seted up, and shaken well-till the formation of complete precipitates.

Precipitates were filtered by using filter paper.

Precipitates were dried using oven with temperature ~~at  $70-80^\circ\text{C}$  - 15 hours~~

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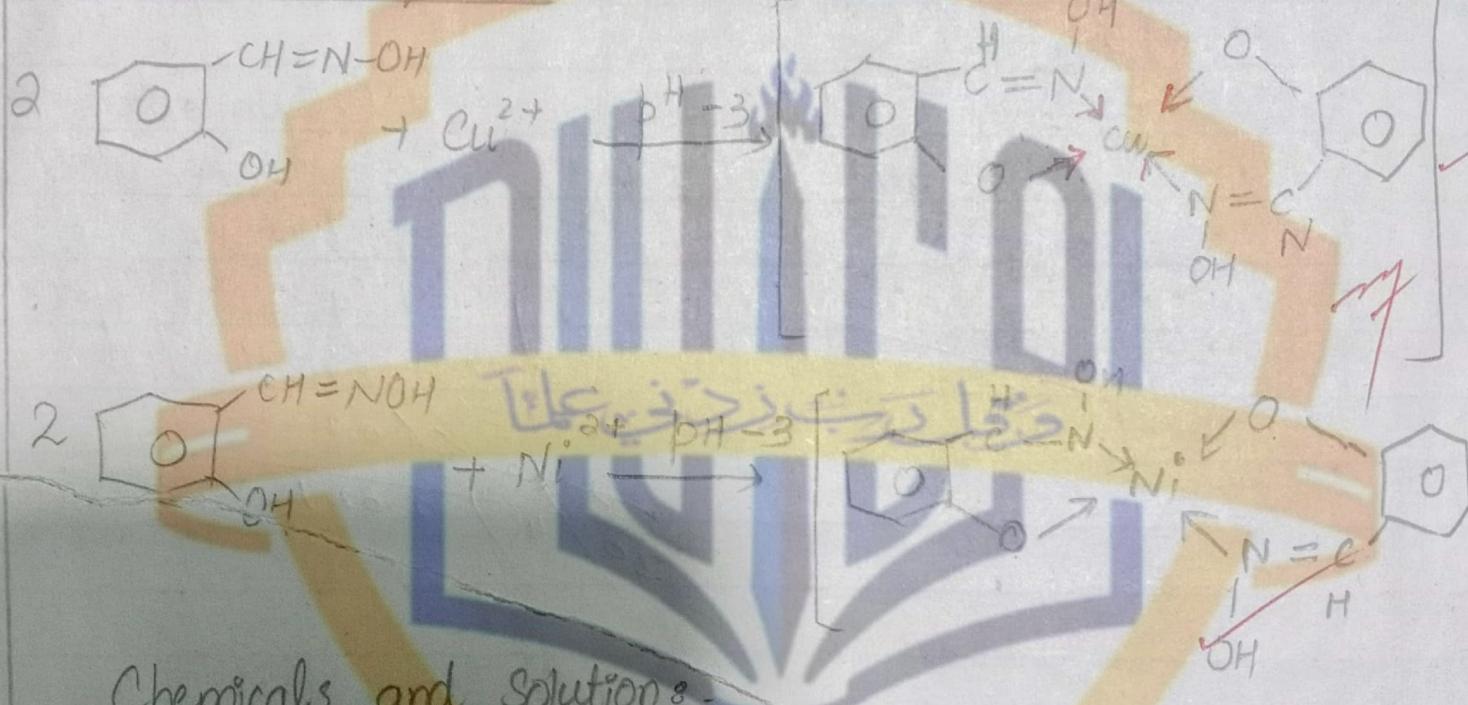
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## Experiment # 5

### Apparatus:-

Filter paper, Beakers, (100cm<sup>3</sup>, 25cm<sup>3</sup>)  
Funnel, Pipette, Measuring balance, Measuring flask.

### Chemical Equation:-



### Chemicals and Solutions:-

- (i) 1g of Salicylaldoxime was dissolved in 15cm<sup>3</sup> of ethanol, then distilled it upto 100cm<sup>3</sup> distilled water - ✓
- (ii) 1% Cu<sup>2+</sup> ion Solution: 0.1 g of NiSO<sub>4</sub> · 7H<sub>2</sub>O and 0.2 g of CuSO<sub>4</sub> · 5H<sub>2</sub>O was mixed and dissolve in 100cm<sup>3</sup> water - ✓

# Experiment #5

08

Determine the percentage  
of  $\text{Cu}^{2+}$  and  $\text{Ni}^{2+}$  in the given sample  
gravimetrically using Salicylaldoxime.

## Theory :-

Theory has already given in experiment

#1.

## Procedure:-

- In the first step,  $20\text{cm}^3$  of sample solution was taken.
- The pH 5-6 was adjusted by using  $\text{NH}_3$ .
- Salicylaldoxime solution was added till the complete formation of precipitates.
- Preheated filter paper was used for the filtration of precipitates.
- Precipitates was dried in an oven.

## For copper:-

- $20\text{cm}^3$  of  $\text{Cu}^{2+}$  solution was taken

concentrations.

For  $\text{Ni}^{2+}$ :

330.7 g of Ni complex contain  $\text{Ni}^{2+} = 58.79 \text{ g}$

1 g of sample contain  $\text{Ni}^{2+} = \frac{58.79}{330.7}$

0.04 g of sample contain  $\text{Ni}^{2+} = \frac{58.79}{330.7} \times 0.04 = 0.007 \text{ g}$

20 cm<sup>3</sup> of sample solution contain  $\text{Ni}^{2+} = 0.007 \text{ g}$

1 cm<sup>3</sup> of sample solution contain  $\text{Ni}^{2+} = 0.007$

100 cm<sup>3</sup> of sample solution contain  $\text{Ni}^{2+} = \frac{0.007}{20} \times 100 = 0.035 \text{ g}$

So,

0.1 g of sample contain  $\text{Ni}^{2+} = 0.035 \text{ g}$

1 g of sample contain  $\text{Ni}^{2+} = 0.035$

10 g of sample contain  $\text{Ni}^{2+} = \frac{0.035}{10} \times 100 = 35\%$

For copper:

weight of filter paper =  $w_1 = 0.94$

weight of filter paper + residue =  $w_2 = 1.04 \text{ g}$

So, 335.54 g of Co-complex contain  $\text{Cu}^{2+} = 63.54 \text{ g}$

1 g of Co-complex contain  $\text{Cu}^{2+} = 63.54$

0.1 g of Co-complex contain  $\text{Cu}^{2+} = \frac{63.54}{335.54} \times 0.1 = 0.018 \text{ g}$

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20 cm<sup>3</sup> of solution contain  $\text{Cu}^{2+} = 0.018 \text{ g}$

100 cm<sup>3</sup> of solution contain  $\text{Cu}^{2+} = \frac{0.018}{20}$

1000 cm<sup>3</sup> of solution contain  $\text{Cu}^{2+} = \frac{0.018 \times 1000}{20} = 0.9 \text{ g}$

0.2 g of sample contain  $\text{Cu}^{2+} = 0.9 \text{ g}$

100 g of sample contain  $\text{Cu}^{2+} = \frac{0.9}{0.2} \times 100 = 45\%$

pH was adjusted to 3 by using acetic acid.

Salicylaldoxime was added in above solution till the complete formation of precipitates.

Prewighed filter paper was used for filtration.

Precipitates were dried in oven.

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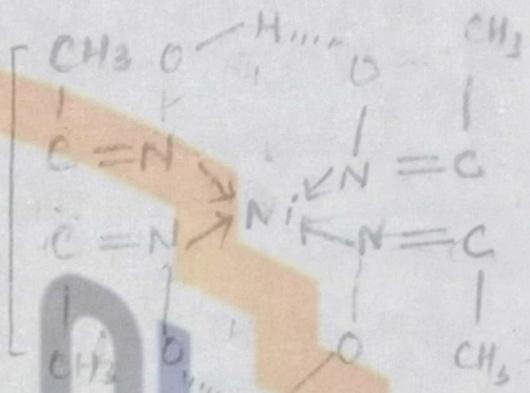
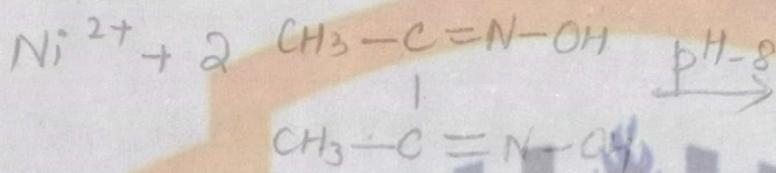
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## Experiment #6

### Apparatus :-

Filter paper, Beakers, Funnel, Pipettes,  
Measuring Balance, Measuring cylinders, Stirrer-

### Chemical Equation:-



### Chemicals and Solutions:-

- (i) 0.1% DMG in Ethanol: 0.1 g of DMG was (II) dissolve in 100 cm<sup>3</sup> ethanol
- (ii) 0.1% Ni<sup>2+</sup> ion: 0.1 g of NiSO<sub>4</sub> · 6H<sub>2</sub>O was dissolve in 100 cm<sup>3</sup> of distilled water.

### Calculations:-

$$\begin{aligned} \text{Weight of filter paper} &= w_1 = 0.889 \\ \text{Weight of filter paper} + w_2 &= 1.049 \\ \text{Weight of precipitate} &= w' = 0.159 \end{aligned}$$

$$\text{Weight of precipitates} - w' = 0.159$$

2.98g [Ni(DMG)<sub>2</sub>] complex contain Ni<sup>2+</sup> = 5.99

1g [Ni(DMG)<sub>2</sub>] complex contain Ni<sup>2+</sup> =  $\frac{5.99}{2.98}$

## Experiment no. 6

Determine the amount per  $\text{dm}^3$  Ni ion in given sample by gravimetrically.

### Theory:-

Theory has already been discussed in experiment no 1.

### DMG:-



Molecular weight = 116.12 g/mol

Melting point = ~~237.5°C~~

Molecular formula =  $\text{C}_4\text{H}_8\text{N}_2\text{O}_2$

### Properties:-

- It exist as white powdery substance.

$$0.16 \text{ g} \quad " \quad \therefore \quad \frac{59}{298} \times 0.16 \\ = 0.039$$

$20 \text{ cm}^3$  of Sample solution contain  $\text{Ni}^{2+} = 0.039$

$1 \text{ cm}^3$  of Sample solution contain  $\text{Ni}^{2+} = \frac{0.03}{20}$

$1000 \text{ cm}^3$  of sample solution contain  $\text{Ni}^{2+} = \frac{0.03}{20} \times 1000$   
 $= 1.5 \text{ g/dm}^3$

### Result:

The amount per  $\text{dm}^3$  of  $\text{Ni}^{2+}$  in a given sample is  $1.5 \text{ g/dm}^3$ .

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It is sparingly soluble in water.

It is soluble in ethyl alcohol, ether and acetone.

It decomposes at its melting point.

### Uses:-

- It forms colored water soluble complexes with  $\text{Fe}^{2+}$ ,  $\text{Co}^{2+}$ , and  $\text{Cu}^{2+}$ .
- It is used from polarographic determination of  $\text{Ni}^{2+}$  and  $\text{Co}^{2+}$ .
- It is used as locating agent for group IV basic radicals  $\text{Ni}^{2+}$ ,  $\text{Co}^{2+}$ .

### Procedure :-

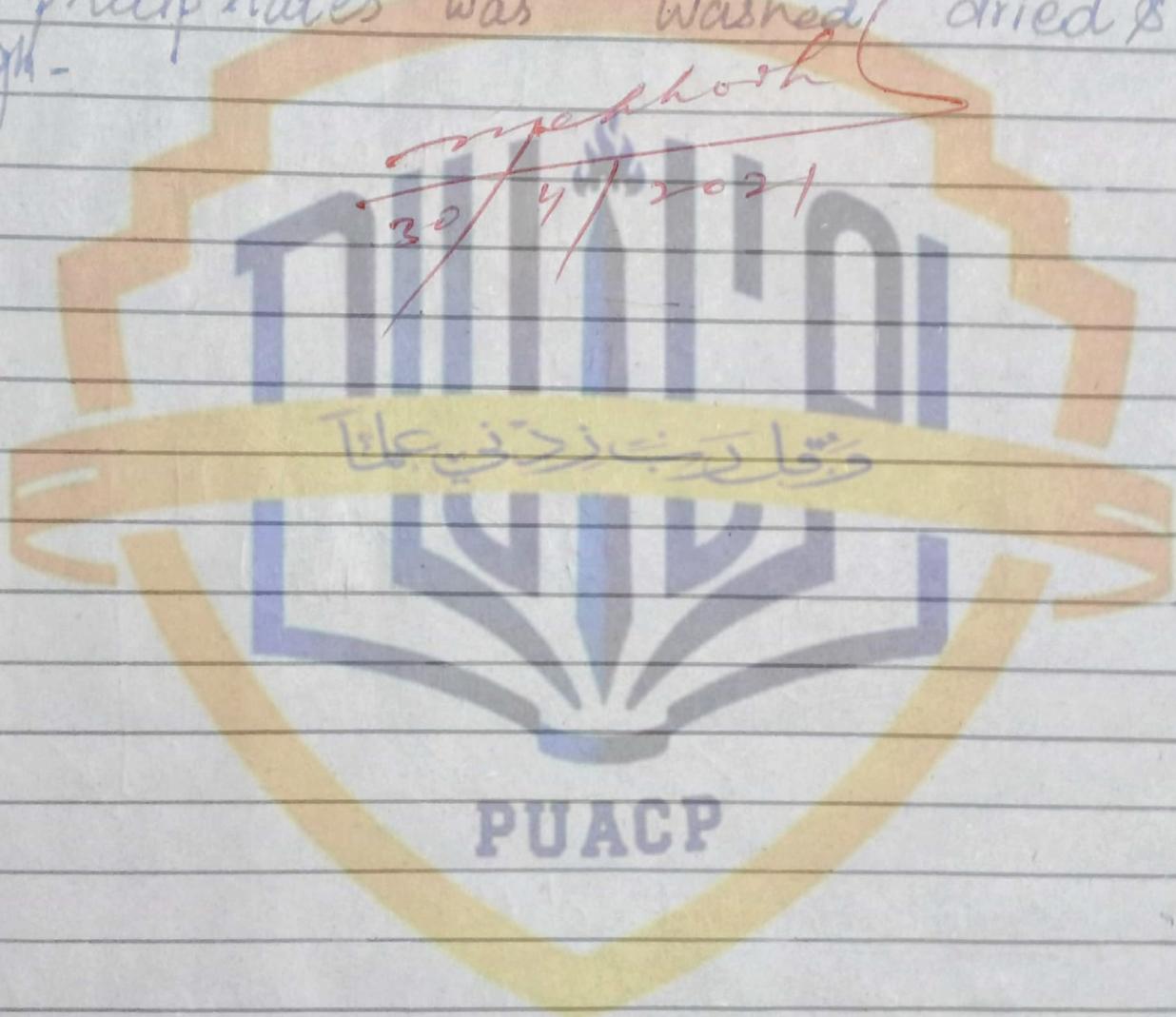
20.0  $\text{cm}^3$  of  $\text{Ni}^{2+}$  sample solution was taken. The pH of the solution was maintained at 8 using few drops of dil. HCl.

The dimethylglyoxime (CDMG) was added to above  $\text{Ni}^{2+}$  solution.

The rose red precipitate was immediately formed.

The rose red precipitate was collected as residue over the weight filter paper.

The precipitates was washed & dried & weighed.

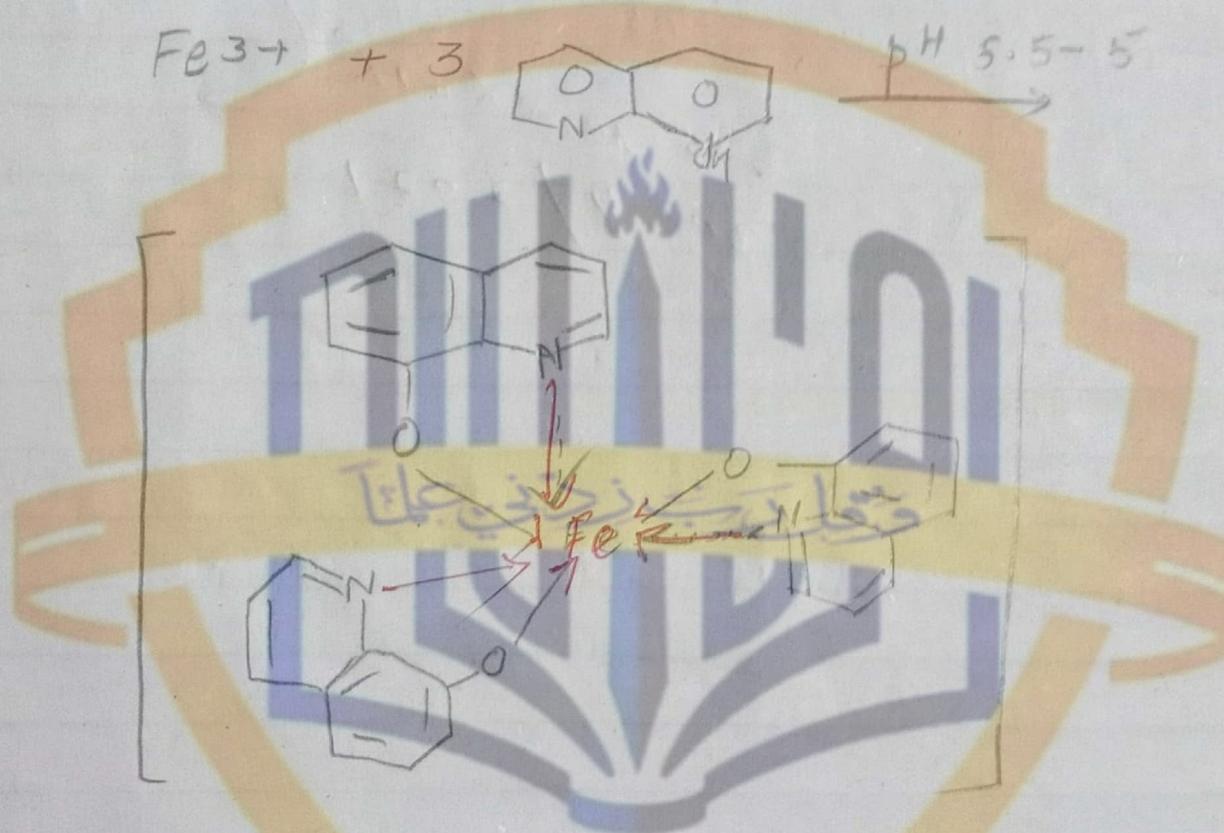


## Experiment #7

### Apparatus:

Filter paper, Beaker, funnel, pipette, measuring balance, Measuring flask, stirrer -

### Chemical Equation



### i) Chemicals and FUMOP

1M acetic acid solution: 1.5 ml a.a dissolved in 250 mL of flask and diluted it upto the mark by dis.H<sub>2</sub>O -

ii) 1M acetic acid: 15 ml of a.a is dissolved in 250 mL of flask and diluted it upto the mark by distilled water -

No.

## Experiment no. 7

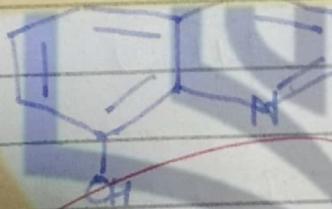
no.

Determine the amount per dm<sup>3</sup> of Fe<sup>3+</sup> gravimetrically by using 8-Hydroxy Quinoline.

### Theory :-

~~Theory has already given in experiment no 1~~

### 8-Hydroxy Quinoline



Molecular weight	:	145.75 g/mol
Melting point	:	74 - 76 °C
Boiling point	:	267 °C
Molecular formula	:	C <sub>9</sub> H <sub>7</sub> NO

### Properties :-

- It exists as white crystalline solid.

iii) 0.1M  $\text{CH}_3\text{COONa}$  solution: 0.82 g of sodium acetate is diluted up to 100 ml water distilled water.

iv) Buffer solution

5 ml of 0.1M acetic acid and 35 ml of 0.1M sodium acetate is added in 100 ml of flask. ✓

v) 2% 8-Hydroxyquinoline

2g of 8-hydroxyquinoline was added in 100 ml of flask and 10-15 ml of ethanol was also added and diluted it upto the mark with 1M acetic acid. ✓

vi) Sample solution:  $\frac{1}{2}$  g Ferrie alum  $(\text{NH}_4)_2\text{SO}_4 \cdot \text{Fe}(\text{SO}_4)_2 \cdot 2\text{H}_2\text{O}$  was added in 100 ml of flask and diluted it upto marks with dis.  $\text{H}_2\text{O}$ . ✓

Calculations

**PUACP**

Weight of filter paper = 0.87 g

Weight of filter paper + precipitate = 1.02 g

Weight of ppt = 1.02 - 0.87

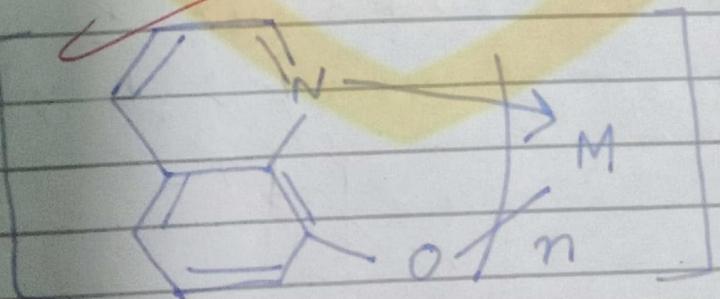
= 0.15 g ✓

- It is insoluble in water and diethyl ether.
- Soluble in ethanol, acetone, chloroform, benzene, mineral acids (like  $\text{HCl}$ ,  $\text{H}_2\text{SO}_4$  or  $\text{HNO}_3$ )

### Uses :-

- It is used for gravimetric analysis of  $\text{Al}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Bi}^{3+}$  and  $\text{W}^{6+}$ .
- It is used for solvent extraction and gravimetric determination of  $\text{Cu}^{2+}$  and  $\text{V}^{3+}$  in  $\text{CHCl}_3$  solution.
- It is used for fluorimetric determination of  $\text{Al}^{3+}$ ,  $\text{Ga}^{3+}$ ,  $\text{In}^{3+}$ .
- $\text{Zn}^{2+}$  which forms fluorescent metal oxanates in  $\text{CHCl}_3$ .

### Complex Structure :-



$$n = 2 \text{ or } 3$$

488 g of complex contain  $\text{Fe}^{3+}$  = 56 g

1g of complex contain  $\text{Fe}^{3+}$  =  $\frac{56}{488}$

$$0.15 \text{ g of complex contain } \text{Fe}^{3+} = \frac{56}{488} \times 0.15 \\ = 0.017 \text{ g}$$

20 ml of sample contain  $\text{Fe}^{3+}$  = 0.017 g

1 ml , , , , , =  $0.017 / 20$

$$1000 \text{ ml of sample contain } \text{Fe}^{3+} = \frac{0.017}{20} \times 1000 \\ = 0.85 \text{ g/dm}^3$$

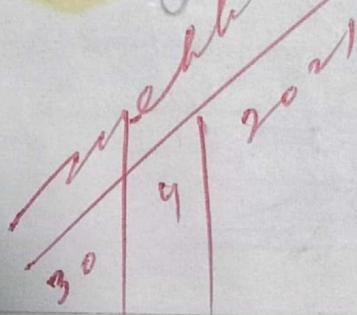
100 ml of ~~sample solution~~ contain  $\text{Fe}^{3+}$

$$\text{Fe}^{3+} = \frac{0.017}{20} \times 100$$

$$= 0.085 \text{ g}$$

Result:

The amount of  $\text{Fe}^{3+}$  per liter is 0.085 g *(per liter)*



## Procedure:-

- 20 cm<sup>3</sup> of solution was taken in beaker.
- 2-3 cm<sup>3</sup> of buffer was added and PH was maintained at 4-5.
- 8-Hydroxyquinoline was added to that till the formation of precipitate.
- Above mixture was heated so paste was formed.
- The solution was cooled and filtered.
- Oven was used for dried of precipitate.
- Precipitate was heated.

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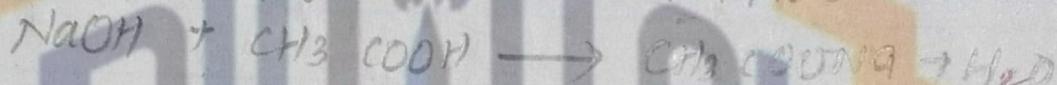
## Experiment no 08:-

Determine the Strength of HCl and CH<sub>3</sub>COOH in the given solution by Conductometric method. You are provided with 0.1 M NaOH.

### Principle:

It is an acid base titration.

Chemical equations:-



Observation and calculations.

0.1 M NaOH

$$\text{Amount of NaOH in } \frac{\text{g}}{\text{dm}^3} = \frac{\text{Molarity}}{\text{dm}^3} \times \text{molar mass} \quad (\text{mol})$$
$$= 0.1 \times 40.0 \quad (\text{mol})$$
$$= 4.0 \text{ g/dm}^3$$

0.05 M HCl

Given      **PURPOSE**

$$M_1 V_1 = M_2 V_2$$

$$12.1 \times V_1 = 0.05 \times 1000$$

$$V_1 = \frac{0.05 \times 1000}{12}$$

$$V_1 = 3.12 \text{ cm}^3$$

Experiment #08: Determine the Strength of HCl and  $\text{CH}_3\text{COOH}$  in the given solution by conductometric method. You are provided with 0.1 M NaOH.

## Theory

### Conductance:

The degree to which an object conducts electricity.

OR.

The reciprocal of resistance is called conductance

### Units:

It is Siemens(s) or mhos ( $\text{mho}$ )  $(\text{S})$   $(\text{A} \cdot \text{V}^{-1})$

### Conductometric titration:

It is a type of titration in which electrolytic conductivity of the reaction mixture is continuously monitored as one reactant is added.

### Equivalence Points

0.05 M  $\text{CH}_3\text{COOH}$  :-

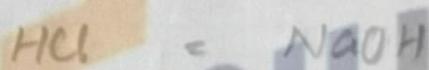
$$M_1 V_1 = M_2 V_2$$

$$17 \times V_1 = 0.05 \times 1000$$

$$V_1 = \frac{0.05 \times 1000}{17}$$

$$V_1 = 2.94 \text{ cm}^3$$

Calculations from Graph



$$\frac{M_1 V_1}{n_1} = \frac{M_2 V_2}{n_2}$$

$$\frac{M_1 \times 25}{1} = \frac{0.1 \times 5}{1}$$

$$M_1 = 0.03 \text{ M}$$



$$\frac{M_1 \times 25}{1} = \frac{0.1 \times 12}{1}$$

$$M_1 = 0.04 \text{ M}$$

Amount per  $\text{dm}^3$  = Molarity  $\times$  Molarmass

Amount  $/ \text{dm}^3$  of HCl  $= 36 \times 0.03 = 1.08 \text{ g/dm}^3$

Amount  $/ \text{dm}^3$  of  $\text{CH}_3\text{COOH} = 60 \times 0.04 = 2.4 \text{ g/dm}^3$

It is the point at which conductivity undergo a sudden change.

## Ohm's Law

The current flowing in a conductor is directly proportional to the electromotive force and inversely proportional to resistance.

$$I \propto E \\ I = \frac{E}{R}$$

## Principle:-

Conductometric titration based on the strength of conductance of electric current through electrolyte solution similar to metallic conductor.

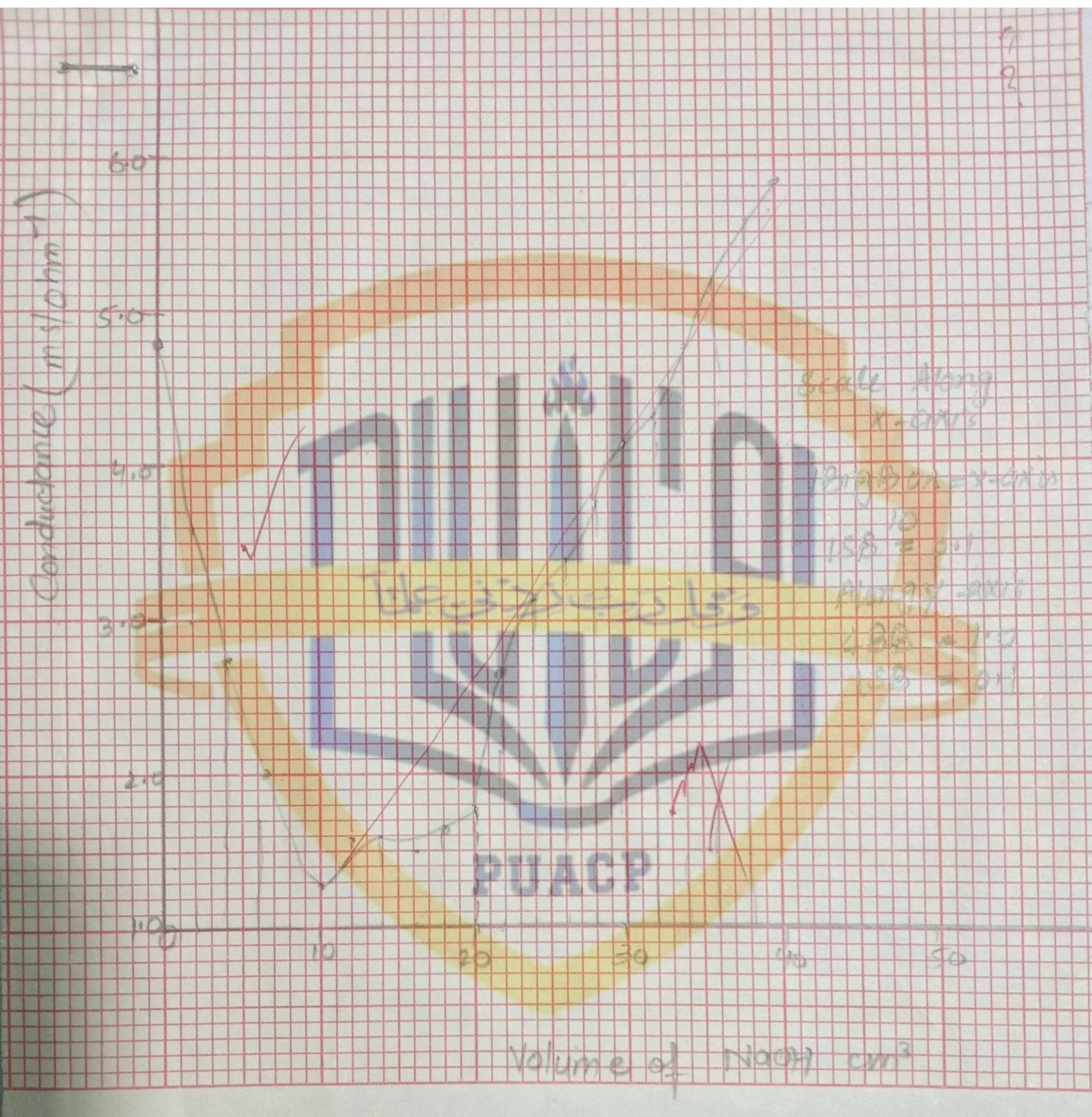
## Advantages:-

Does not required indicator since change in conductance is measured by conductometer.

Suitable for colored solutions  
End point is measured by graphical means,  
accurate result obtained by minimum error.

## Observations:-

Volume of NaOH ( $\text{cm}^3$ )	Conductance ( $\mu\text{S}/\text{cm}$ )
0	4.81
2	3.61
4	2.75
6	1.46
8	1.14
10	1.24
12	1.34
14	1.43
16	1.56
18	1.64
20	1.73
22	2.66
24	3.02
26	3.38
28	3.74
30	4.02
32	4.32
34	4.88
36	5.19
38	5.57
40	5.88



## Disadvantages:-

Increasing level of salts in solution masks the conductivity changes, Accurate reading is not obtained. Its application in redox titration is limited.

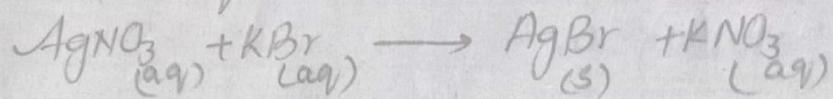
## Procedure:-

- ~~25 cm<sup>3</sup> of 0.05M acetic acid was taken in a beaker. Add 25 cm<sup>3</sup> of 0.05 M HNO<sub>3</sub> solution in same beaker.~~ ~~→~~ 0.1M NaOH solution was added in burette.
- Beaker was placed under burette and 0.1M NaOH added dropwise.
- Conductance was measured after addition of 20cm<sup>3</sup> NaOH in beaker every time.
- The conductance was increased at V<sub>1</sub> which shows the volume of CH<sub>3</sub>COOH and V<sub>3</sub>-V<sub>1</sub> showed volume of HNO<sub>3</sub>.
- The graph showed on decreasing due to decrease in H<sup>+</sup> ions and increases due to increase in H<sup>+</sup> ion concentration.

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## Experiment 09 :-

Chemical Equation :



### Preparations:

i) 0.05M  $AgNO_3$  solution:

$$n = \text{Molarity} \times \text{Molar mass}$$

$$= 0.05 \times 170$$

$$= 8.5 / 10$$

$$= 0.858$$

ii) 0.025M of  $KBr$  in  $100\text{ cm}^3$

$$n = \text{Molarity} \times \text{Molar mass}$$

$$= 0.025 \times 119$$

$$= 2.97 / 10$$

$$= 0.298$$

### Calculations

$KBr$

$$\frac{M_1 V_1}{n_1}$$

$AgNO_3$

$$\frac{PU}{M_2}$$

$$\frac{M_1 \times 25}{1} = \frac{0.05 \times 18}{1}$$

$$M_1 = 0.03M$$

Amount /  $\text{dm}^3$  of  $Br^-$  ion = Molarity  $\times$  Molar weight

$$= 0.03 \times 80$$

$$= 2.4 \text{ g/dm}^3$$

## Experiment 09:

Determine the amount per  $\text{dm}^3$  of  $\text{Br}^-$  using 0.05 M  $\text{AgNO}_3$  solution.

### Theory:

Theory has already given in experiment no 8.

### Procedure:

- 20  $\text{cm}^3$  of solution was taken in the beaker.
  - 0.05 M acetic acid  $\text{AgNO}_3$  solution was added in burette.
  - 2  $\text{cm}^3$  of  $\text{AgNO}_3$  was added in the beaker dropwise.
  - After every 2  $\text{cm}^3$  addition, conductance was measured by conductometer.
  - Conductance was noted.
- Unknown conductance was measured by graph.

PUACP

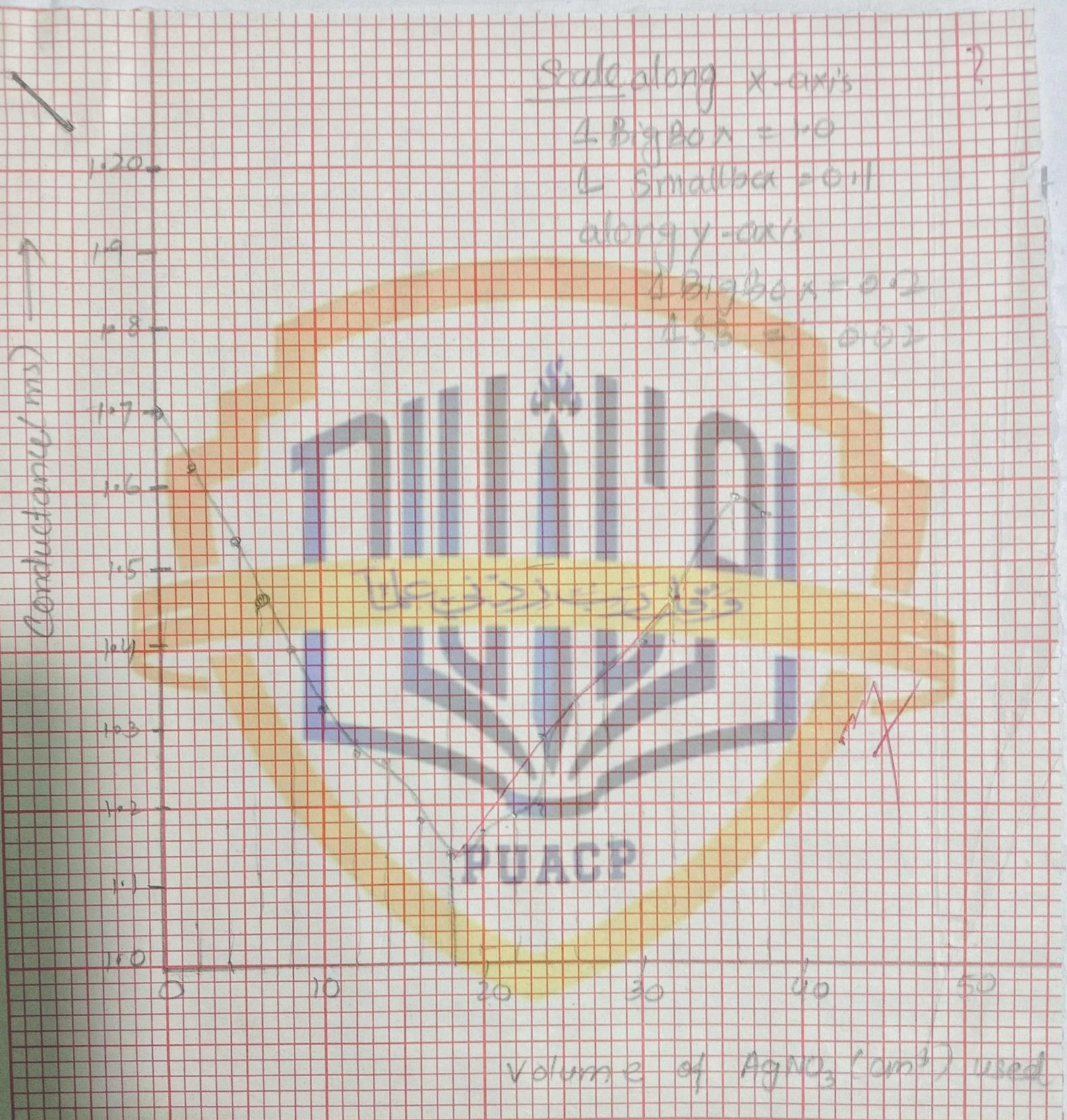
Observations

Volume of $\text{AgNO}_3$ used	Conductance (m.s/ohm)
0	1.70
2	1.61
4	1.54
6	1.46
8	1.40
10	1.33
12	1.27
14	1.24
16	1.19
18	1.14
20	1.16
22	1.19
24	1.28
26	1.32
28	1.37
30	1.40
32	1.44
34	1.48
36	1.58
38	1.57

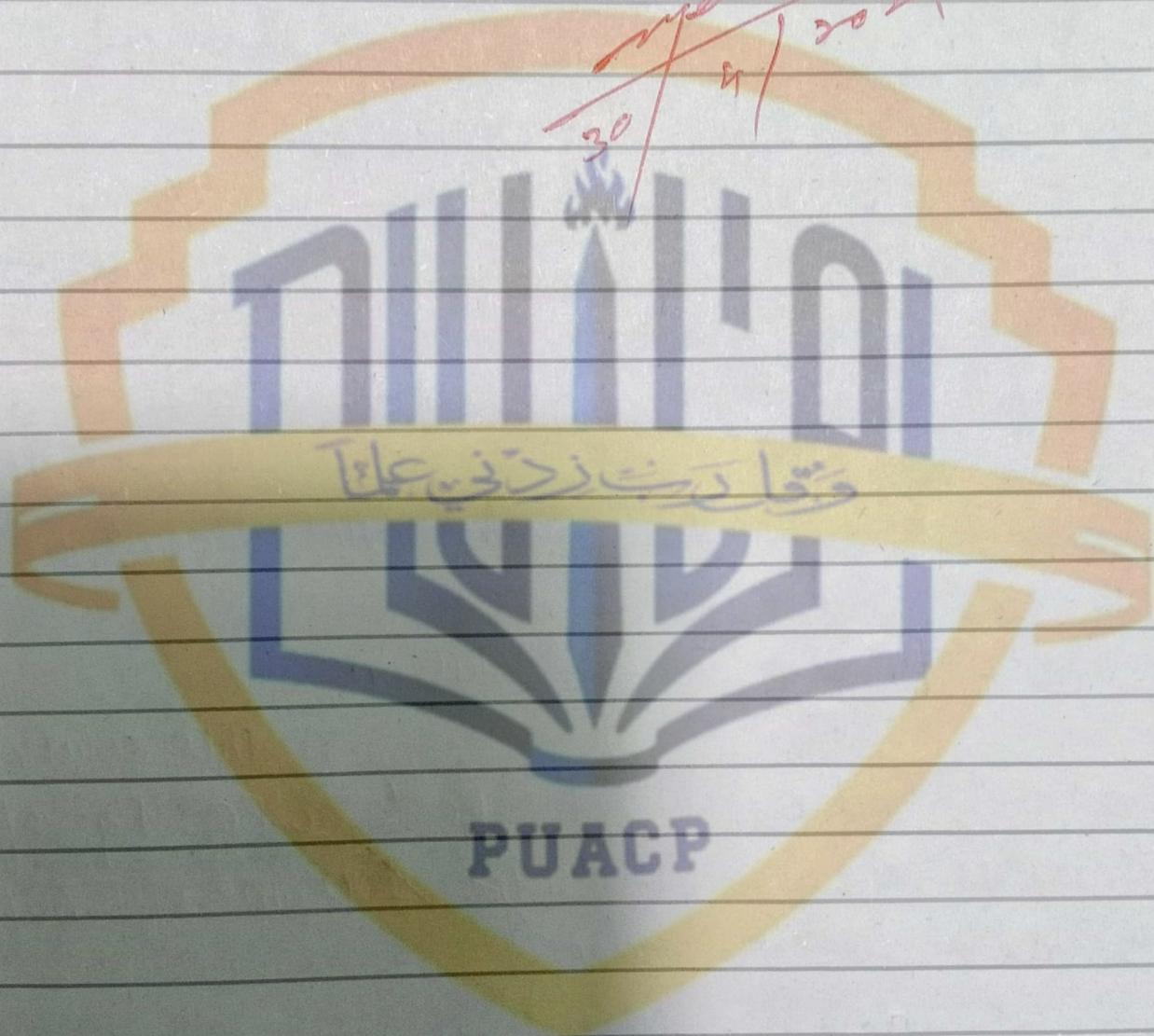
Result :

In the given amount per  $\text{dm}^3$  of  $\text{Br}^-$  ion  
 solution is  $2.49 \text{ dm}^{-3}$  -

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~~Result were concluded.~~



## Experiment no 10

Determination of  $\text{Fe}^{2+}$  spectro-  
photometrically by 1-10 phenanthroline.

Chemicals Required:-

Buffer solution ( $\text{pH} = 4.5$ )

1% Hydroquinone

0.1% Bipyridyl solution

Preparation of Chemicals:-

1) Buffer solution of  $\text{pH} = 4.5$ -

12 cm<sup>3</sup> of acetic acid + 8.3 g sodium acetate,  
dilute it upto the mark with 100 cm<sup>3</sup> of  
distilled water.

2) 1% Hydroquinone solution:-

This solution used  
for the reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  1g of  
hydroquinone add into 100 cm<sup>3</sup> of buffer  
heated it to dissolve.

3) 0.1% Bipyridyl solution:-

0.1g bipyridyl + 15 cm<sup>3</sup>  
of and dilute it upto the mark  
with H<sub>2</sub>O in 100 cm<sup>3</sup> flask.

## Experiment no 10

Determination  
of  $\text{Fe}^{2+}$  spectrophotometrically by 1-10  
phenanthroline.

### Theory :-

#### Spectrophotometry :-

It is the quantitative measurement of the reflection or transmission property of materials as a function by wavelength ( $\lambda$ )

#### Spectrophotometer :-

These techniques are used to measure the concentration of solutes in solution by measuring the amount of the light that is absorbed by the solution in a cuvette placed in spectrophotometer. This technique is used to measure light intensity as a function of wavelength.

→ Spectrometry is an interaction of radiation with matter, that are various classified as,

→ Standard solution of  $\text{Fe}^{2+}$   
Mohr's salt + Iron sulphate  
 $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{FeSO}_4 \cdot 6\text{H}_2\text{O}$

$$\frac{\text{Molecular weight}}{\text{Atomic weight}} = \frac{392}{56} = 7g$$

$$\frac{1}{10} = 0.7g$$

→ 0.7g dissolved in 100 mL  $\text{H}_2\text{O}$  to form  
1000 ppm solution.

Preparation of PPM solutions -

FOR 100 ppm :-

$$\frac{\text{Given}}{M_1 V_1} = \frac{\text{Required}}{M_2 V_2}$$

$$\frac{1000 \times V_1}{V_1} = \frac{100 \times 100}{100 \text{ cm}^3}$$

FOR 2 ppm :-

Given                  Required /

$$M_1 V_1 = M_2 V_2$$

$$100 \times V_1 = 2 \times 100$$

$$V_1 = 2 \text{ cm}^3$$

# Types of Spectroscopy :-

## Atomic Spectroscopy :-

It is based on absorption, emission, or fluorescence by atoms or elementary ions. It can be divided into

- i) Atomic absorption spectroscopy - (AAS)
- ii) Atomic emission spectroscopy - (AES)
- iii) Fluorescence spectroscopy - (AFS)

## Ultra-Violet and visible spectroscopy :-

UV and visible spectroscopy analysis compounds using the electromagnetic radiation spectrum from 10 nm to 700 nm. Many atoms are able to emit or absorb visible light.

## Infrared Spectroscopy :-

This analysis used infrared spectrum which can split into following.

- near IR.
- Mid IR.
- Far IR.

FOR 4ppm:-

Given

$$M_1 V_1 = M_2 V_2$$

$$100 \times V_1 = 4 \times 100$$

$$V_1 = 4 \text{ cm}^3$$

Required

FOR 6ppm:-

Given

$$M_1 V_1 = M_2 V_2$$

$$100 \times V_1 = 6 \times 100$$

$$V_1 = 6 \text{ cm}^3$$

Required

FOR 8ppm:-

Given

Required

$$M_1 V_1$$

$$= M_2 V_2$$

$$100 \times V_1 = 8 \times 100$$

$$V_1 = 8 \text{ cm}^3$$

i) Near IR has the greatest energy <sup>and</sup> can penetrate much deeper, It is less sensitive.

## Raman spectroscopy :-

It is similar to IR in that It is a vibrational spectroscopy technique but It is less in elastic scattering.

## Nuclear Magnetic Resonance :-

It uses resonance Spectroscopy and nuclear spin state for spectroscopic analysis.

## Molecular Spectroscopy :-

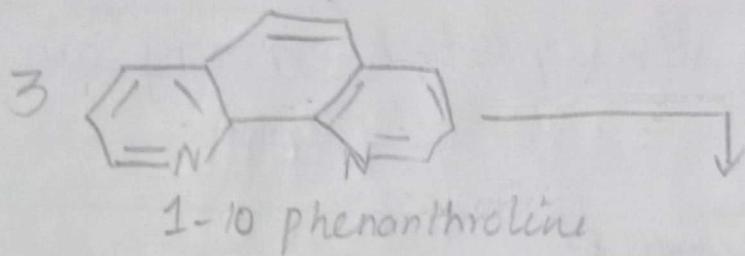
UV, IR, NMR,

mass spectrometry -

~~i) On the basis of Emission and spark~~ FES, ICPAES, AES are and ~~spark~~ used as a source of radiation rather flame -

~~ii) IR, NMR -  
iii) Absorption AAS -~~

Chemical equation :-



Concentration (ppm)	Absorbance
0	0.00
2	0.68
4	1.12
6	1.63
8	2.33
Unknown	1.35

## Spectrum diagram:-

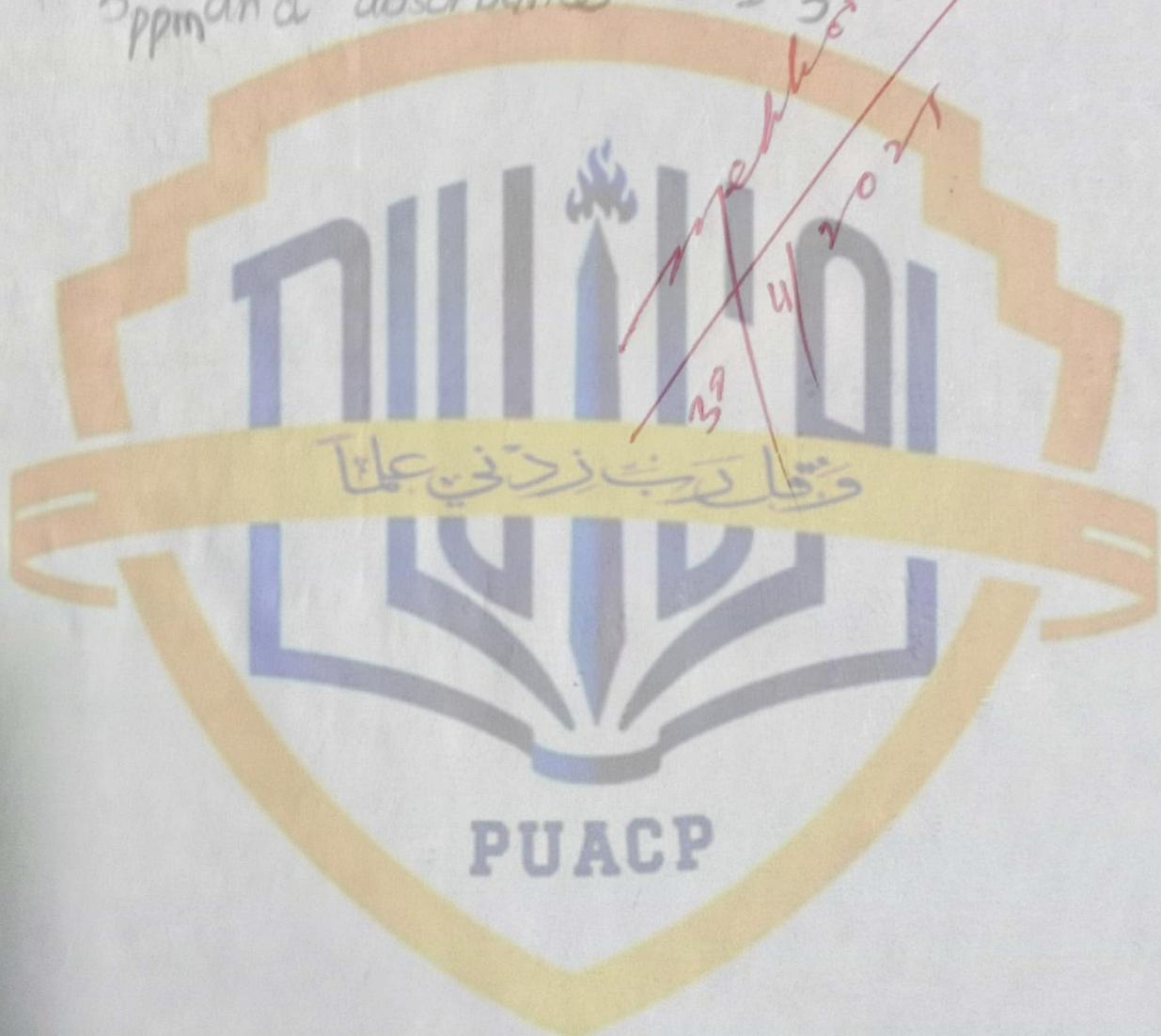
Spectral region: Cosmic rays, Gamma, X-rays, UV, IR, micro-wavelength  
 $10^{-4} \text{ Å}$ ,  $10^{-3} \text{ Å}$ ,  $10^{-1} \text{ } 100 \text{ Å}$ ,  $400 \text{ nm}$   $800$ ,  $10^3$   
Energy:  $2 \times 10^{-11}$ ,  $2 \times 10^{-12}$ ,  $2 \times 10^{-4}$ ,  $2 \times 10^{-17}$ ,  $5 \times 10^{-8}$   
 $2 \times 10^{-19}$ ,  $2 \times 10^{-22}$ ,  $2 \times 10^{-25}$

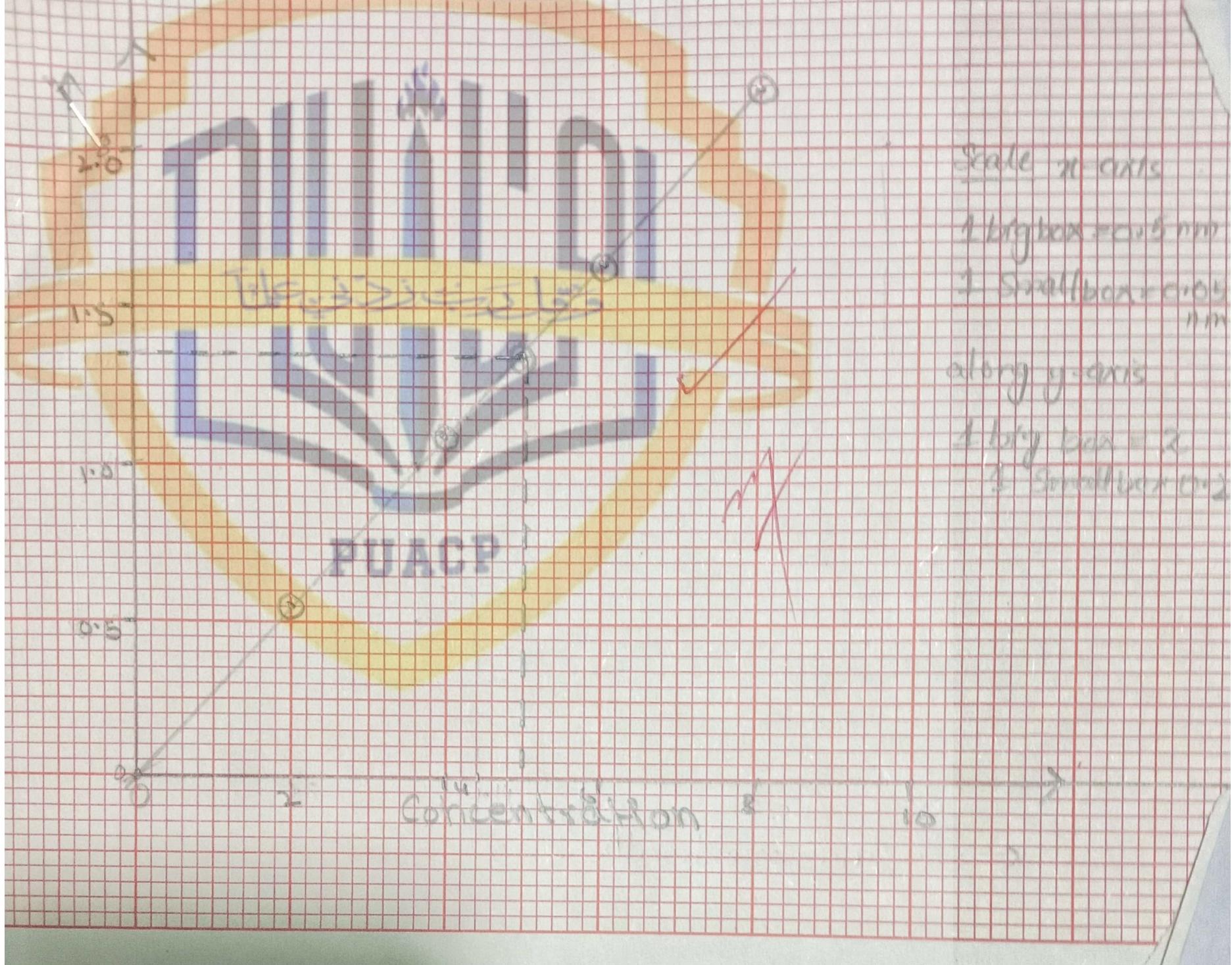
## Procedure:-

- Stock solution of  $\text{Fe}^{2+}$  is formed by dissolving  $0.3 \text{ g}$  of Mohr's salt in flask, upto the mark with  $100 \text{ cm}^3$  of distilled water -
- This is  $1000 \text{ ppm}$  stock solution.
- Standard solution is formed from  $1000 \text{ ppm}$  stock solution  $5-30 \text{ ppm}$  standard solution was formed and unknown solution of  $13 \text{ ppm}$  was formed from  $1000 \text{ ppm}$  stock solution by applying dilution formula.  
$$M_1 V_1 = M_2 V_2$$
- $5 \text{ cm}^3$  of standard solution of  $5 \text{ ppm}$  was separately taken in test tube and  $2 \text{ cm}^3$  hydroquinoline solution was added, which act as reducing agent and reduce  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  and  $5 \text{ cm}^3$

Results-

The concentration of unknown sample  
is 5 ppm and absorbance is 1.34 nm-





O-phenanthroline solution was added-

- Absorbance of these solution was noted by keeping the  $\lambda_{max}$  at 515 nm.
- This procedure was repeated for all the standard solutions.
- Calibration curve was drawn by taking the concentration at x-axis and absorbance at y-axis.

PUACE  
Experiment No. 1  
Date: 30/04/2021