Experiment No. 4:-

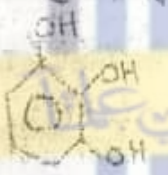
Determine the amount of Bi^{+3} gravimetrically.

Chemical Equation:-

Pyrogallol

white ppt.

Precipitating Agent:-



Pyrogallol

Observations & calculations:-

wt. of filter paper = w_1 = 0.9g

wt. of filter paper + Precipitates = w_2 = 1.2g

wt. of Precipitates = w_3 = 1.2 − 0.9

= 0.3g

Experiment No. 4:-

Determine the amount/L of Bi^{+3} gravimetrically.

Principle:-

it is a gravimetric analysis.

Chemistry of Bi^{+3}:-

Bismuth is the heaviest member of all naturally occuring elements. its atomic no. is 83 and atomic wt. is 208g/mol. It is post transition metal. it is belongs to the 6th period of the periodic table. the electronic configuration of bismuth is given as followed in the below:-

$$Bi = [Xe]\ 4f^{14}\ 5d^{10}\ 6s^{2}\ 6p^{3}$$

Bismuth is used in some pharmaceuticals. The medicine of Bismuth is Bismd which is used for acidity. Bismuth shows s-inert pair effect. its shows variable oxidation staten like as Bi^{+3} & Bi^{+5}, it is toxic.

Procedure:-

First of all 1% of $Bi(NO_3)_3 \cdot 5H_2O$

11

Amt of Sample contain Bi^{+3} = 0.1 mg

1 ml of Sample contain Bi^{+3} = $\frac{0.1}{20}$

1 ml of sample contain Bi^{+3} = $\frac{0.1}{20} \times 1000$

= 5 µg/L

Result:-

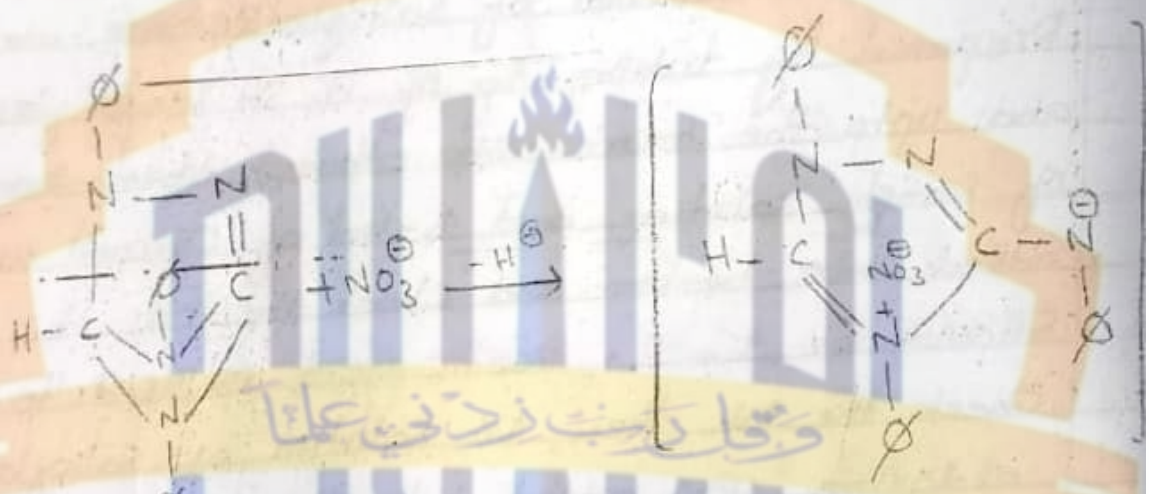
The amount of Bi^{+3} is 0.4 g

Solution was prepared by taking 1g of $Bi(NO_3)_3 \cdot 5H_2O$ in a flask and small quantity of HNO_3 was added in it, in order to dissolve it in water. Then it was marked upto 100ml with distilled water. 1% solution of Pyrogallol was Prepared by dissolving 1g of it in hot water and mark it upto 100ml with distilled water. kept the Pyrogallol Solution in cap bottle, it is reactive and explosive, then 20ml of metal Solution was added in 500ml beaker and Pyrogallol was added in it (7ml) till ppt. formed. If ppt. was not formed then added a few drops of liq. NH_3 and yellow coloured ppts. were formed, coagulate the ppt. by heating, filter it on dry filter Paper. Then dry it in oven and note down the weight.

Experiment No. 6:-

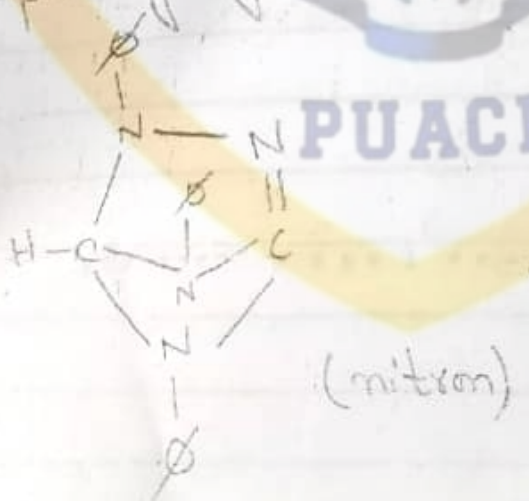
Determine the amount/L of $NO_3^{\ominus}$ ion gravimetrically.

Chemical Equation:-



(Nitron) (nitrate - nitron Complex)

Precipitating Agent:-



(nitron)

Experiment No. 6:-

Determine the amount/L of NO_3^- ion gravimetrically:-

Principle:-

It is a gravimetric analysis.

Chemistry of NO_3^- ion:-

Nitrate is polyatomic ion with molecular formula $NO_3^{\ominus}$ and a molecular weight of 62.0049g/mol. they are mainly Produced for used as fertilizers. because of their high solubility and biodegradibility, the main nitrates are ammonium, Potassium, Sodium and calcium salts. To treat acidic soil in Pakistan fertilizers like ammonium nitrate and potassium nitrate are used. the second major application of nitrates is as an oxidizing agent.

Sodium nitrate is used to remove air bubbles from molten glass and some ceramics. they oxidizes the Fe atom in heamoglobin from ferrous Iron(+2) to ferric iron (+3) make it unable to carry O_2. Excessive $NO_3^{\ominus}$ are runoff with rain H_2O where it causes algal bloom due to which death of the Phytoplankton and the Zooplankton.

Observations and calculations:-

wt. of filter Paper = w_1 g = 0.9g

wt. of Paper + ppt. = w_2 g = 2.23g

wt. of Precipitates = w_3 g = $w_2 - w_1$ = 2.23 − 0.9

= 1.33g

374g of complex contain $NO_3^{\ominus}$ = 62g

1g of complex contain $NO_3^{\ominus}$ = $\frac{62}{374}$ g

1.33g of complex contain $NO_3^{\ominus}$ = $\frac{62}{374} \times 1.33$

= 0.22g

20ml of Sample contain $NO_3^{\ominus}$ = 0.22g

1ml of Sample contain $NO_3^{\ominus}$ = $\frac{0.22}{20}$ g

1000ml of Sample Contain $NO_3^{\ominus}$ = $\frac{0.22}{20} \times 1000$

= 11g/L

Result:-

The amount/L of nitrate ion is 11g/L.

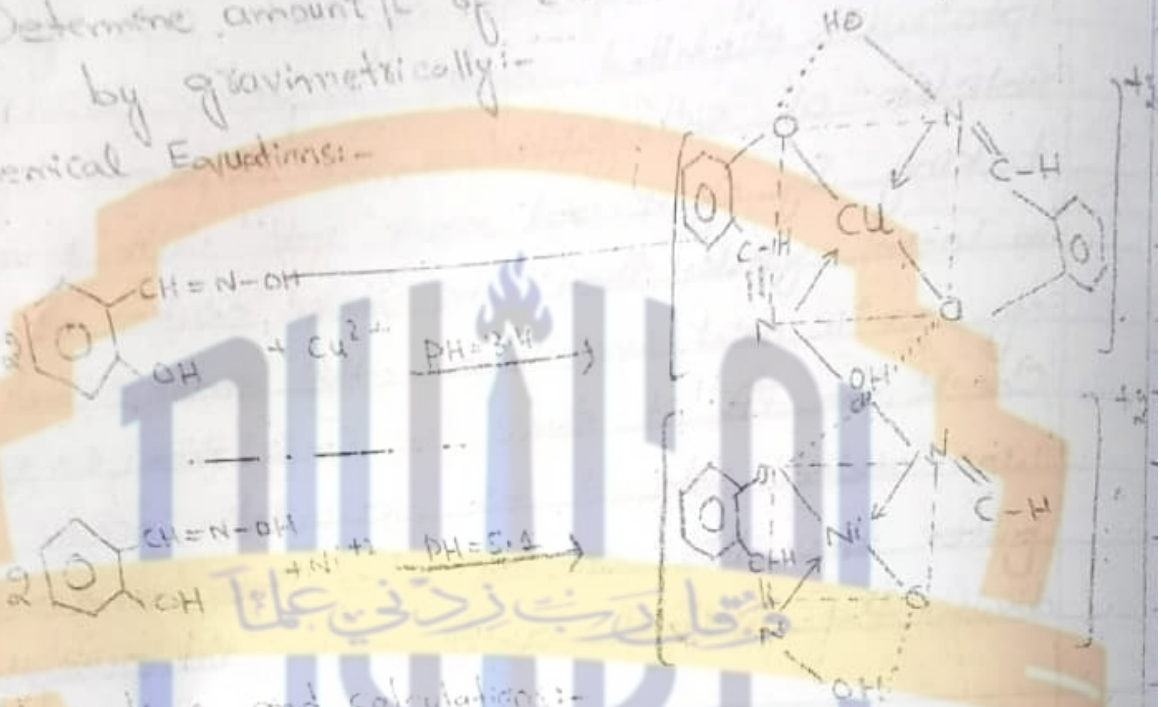
Procedure:-

First of all, 5% solution of CH_3COOH was Prepared. In lab 100% acetic acid is available, So according to $C_1V_1 = C_2V_2$, 5ml of CH_3COOH was taken in a 100ml flask. 10-15ml of ethanol was added into it and upto mark with distilled water. then 1% solution of nitron was prepared by adding 1g of nitron in 100ml flask and upto mark with 5% solution of acetic acid with ethanol. if not dissolve then heat it on water bath. then 20ml of Sample solution was taken in beaker. 1% solution of $NO_2^{\ominus}$ ion was pre-pared by dissolving 1g of $NaNO_3$ in 100ml flask and mark upto with distilled H_2O. then 20ml Sample solution + 1ml of CH_3COOH was add in beaker, then nitron solution was added into it till precipitation occur. then Coagulate the ppt. by boiling, cool, filter it on dry filter paper and note down weight.

Experiment No. 8:-

Determine amount/L of Cu^{+2} and Ni^{2+} in give mix. by gravimetrically:-

Chemical Equations:-



Observations and calculations:-

Wt. of filter paper = W_1 g

Wt. of Paper + ppt = W_2 g

Wt. of Precipitates = W_3 g = $W_2 - W_1$ = A g

335.5 g of Cu-salicyaldoxime complex contain

Cu^{2+} = 63.54 g

1 g of Cu-Salicyaldoxime complex contain

$Cu^{+2} = \frac{63.54}{335.5}$ g

A g of Cu-Salicyaldoxime complex contain

$Cu^{2+} = \frac{63.54}{335.5}$ g x A = 'B' g

Experiment No. 8:-

Determine the amount/L of Cu^{+2} and Ni^{+2} in given mixture by gravimetrically:-

Principle:-

This is gravimetric analysis.

Theory:-

Gravimetry is non-instrumental technique and gravimetric analysis describes a set of methods in analytical chemistry for the qualitative determination of analyte based on mass.

When based on instrumental, then it is called thermal gravimetry. it is used as qualitative as well as for the quantitative analysis. two types of errors come.

* Positive error.
* Negative error.

Positive error is due to impurity or due to deposition of organic solvents. Negative error comes when organic solvent used in large quantity and complex dissolve in it.

20ml of sample contain Cu^{2+} = B g

1.0ml of sample contain Cu^{2+} = $\frac{B}{20}$ g

1000ml of sample contain Cu^{2+} = $\frac{B}{20} \times 1000$ = 'C' g/L

Wt. of filter paper = W_4 g

wt. of Paper + ppt = W_5 g

wt. of ppt. = W_6 g = $W_5 - W_4$ = 'D' g

330.7 g of Ni-salicyaldoxime complex contain Ni^{2+} = 58.00 g

1000 g of Ni-Salicyaldoxime complex contain Ni^{+2} = $\frac{58}{330.7}$

D g of Ni-Salicyaldoxime complex contain Ni^{+2} = $\frac{58}{330.7}$ g $\times D$ = 'E' g

20ml of Sample Solution contains Ni^{2+} = E g

1.0ml of Sample Solution contains Ni^{2+} = $\frac{E}{20}$ g

1000ml of Sample Solution contains Ni^{2+} = $\frac{E}{20} \times 1000$ = 'F' g/L

Total amount of mixture = G

% age of Cu^{2+} = $\frac{C}{G} \times 100$ = 'x' %

% age of Ni^{2+} = $\frac{F}{G} \times 100$ 'y' %

Result:-

The amount/L of Cu^{2+} is 'c' g/L and of Ni^{2+} is 'F' g/L in given mixture.

Procedure:-

0.1g of Ni salt and 0.1g of Cu salt was taken in 100ml flask and marks upto the distilled water. it is 0.1% of sample solution of mixture. 0.1% of Salicyaldoxime in ethanol was prepared by taking the 0.1g of it and dissolve it into 100ml of ethanol. 2ml of sample was taken and then added the fresh glacial acetic acid to maintain PH 2 or 3, then added Salicyaldoxime till the ppt. formed. for Coagulation heat the ppt, then filtered and dried it. these are Cu^{+2} ppt. then added dilute NH_3 soln. in the above filterate and adjust PH 6 and then added the Salicyaldoxime till the precipitates formed, then heat for coagulation, filtered it and dried the ppt. in oven. these are Ni^{+2} Precipitates

Experiment No: 26

Determine the amount of Iron Colorimetrically in given Sample:-

→ Preparation of Solution:-

($FeSO_4 \cdot 7H_2O$)

Mol. wt. = 178.01 = 371 = 0.371 / 100mL

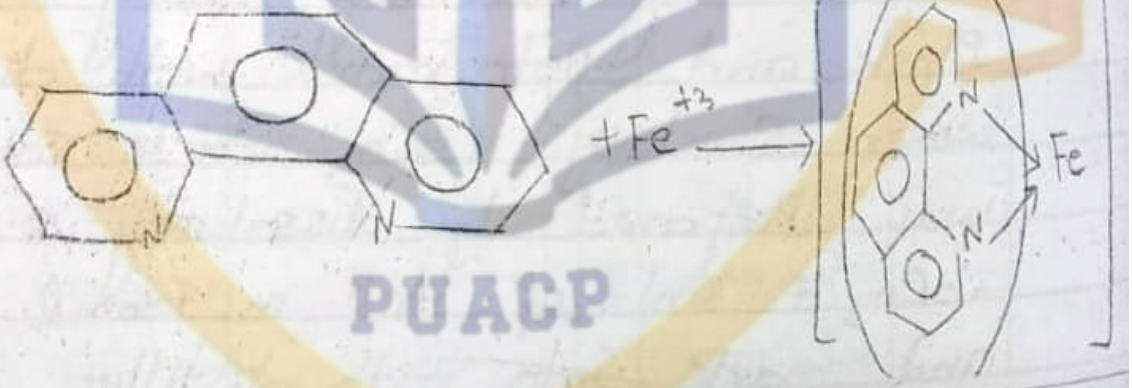
molar mass 56 10

→ 0.25% 1,10-phenanthroline solution:-

0.25g in 100mL flask.

λ = 515nm

→ Chemical Equations:-



→ Buffer of PH 4.5:-

→ 0.1M CH_3COOH:-

Given : Required

$C_1V_1 = C_2V_2$

$17 \times V_1 = 0.1 \times 100$

$V_1 = 0.58mL/100mL$

→ 0.1M CH_3COONa

mol. wt = 82g

$M = \frac{mass}{m.mass} \times \frac{100}{100}$

$0.1 = \frac{x}{82} \times 10$

$x = \frac{0.1 \times 82}{}$

= 0.82g/Pmol

Experiment No: 26

Determine the amount/L of Iron Colori-metrically in given Sample:-

Principle:-

This is a calorimetric analysis of iron and fall in visible spectroscopy.

Theory:-

Spectroscopy is interaction of radiations with matter. it can be classified on the basis of wavelength.

These wavelengths are following like X-ray, U.V, Visible, I.R, microwave Spectroscopy and γ-ray Spectroscopy.

On the basis of Specics,

Atomic Spectroscopy.

- Flame-photometery
- AAS
- Icp-Analysis

Molecular Spectroscopy.

- U.V
- Visible

→ For Buffer preparation:-

Mix 65 mL of CH_3COOH and 35mL of 0.1m of CH_3COONa.

→ 7% Hydroquinone:-

1g hydroquinone in 100mL of buffer. to dissolve hydroquinone, 5-6mL of C_2H_5OH was added into it

→ For standard solution preparation:-

In a test tube 10mL of Standard solution + 2mL of hydroquinone + 5mL of orthophenanthrolein was added.

→ For blank solution:-

10ml of dis. H_2O + 2mL of Hydroquinone + 5mL of orthophenanthrolein was added into it.

Visible Spectroscopy is called colorimetry.

Phenomena

Emission Spectroscopy | Absorption Spectroscopy | Fluorescene | Phosphorescen

Source of Radiation

Flame photometery | Arc spectroscopy | Plasma Spectroscopy

Atomizer is flame | current is used | plasma as a source.

Basic Requirements of colorimetric analysis:-

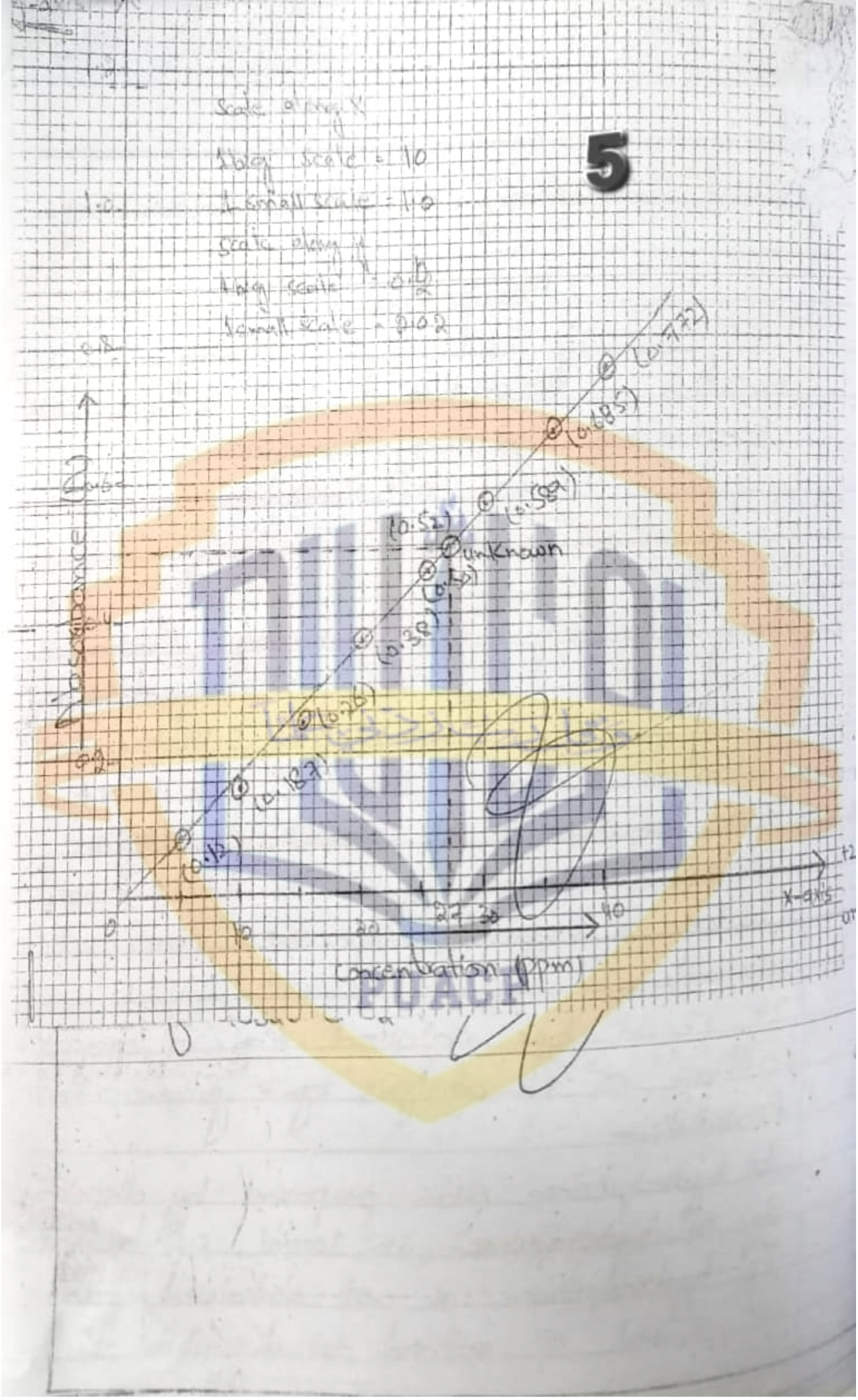
Species would be soluble in either solvent (water or organic), suspensions are not allowed.

It should be coloured eg. in organic Caffiene or Ni-analysis give yellow colour.

Procedure:-

1% hydroquinone was prepared by dissolving 1g of hydroquinone in 100ml of R buffer. If hydroquinone is not dissolved, then add 5-6ml of ethanol to dissolve the hydroquinone.

5


(0·732)
(0·685)
(0·584)
(0·52)
unknown
(0·38)
(0·26)
(0·187)
(0·12)
Concentration (ppm)
X-axis

5-50ppm solution were prepared. 5ppm solution was prepared by dissolving 1oml Standard solution. 2ml of hydroquinone and 5ml of phenenthrolein in test tube. Similarly othere solutions were Prepared.

Reading was taken with the help of the electronic. it was set at 0.0. with the help of blank solution. And blank solution was prepared by dissolving 1oml of dist. H_2O + 2ml of hydroquinone + 5ml of the orthophenanthrolein. Instrument was set at 515nm and absorbance was noted and graph was plotted b/w absorbance and concentration.

Experiment No: 07

Determine the amount/L of Ni^{2+} by colorimetrically:-

→ Preparation of Solutions:-

($NiSO_4 \cdot 6H_2O$)

$$\frac{\text{Mol. wt.}}{\text{m. mass}} = \frac{263}{58} \cdot \frac{4.53}{10} = 0.453\text{g}/100\text{mL}$$

→ 1% DMG solution:-

0.1g of DMG in ethanol

→ 0.5% NH_3 soln:-

Given : Required

$$C_1V_1 = C_2V_2$$

$$33 \times V_1 = 5 \times 100$$

$$V_1 = 15\text{mL in } 100\text{mL flask}$$

→ For standard solution preparation:-

In a seperating funnel 10mL $CHCl_3$ + 10mL standard solution + 1 drop of NH_3 + 10mL DMG.

Lower organic layer seperate out.

Experiment No:07

Aim:- Determine the amount/L of Ni^{+2} by colorimetrically in a given sample.

Principle:-

This is calorimetric analysis of Nickel.

Theory:-

Spectroscopy is interaction of radiations with matter. It can be classified on the basis of wavelength and the species. Visible Spectroscopy is colorimetery.

Basic requirements of colorimetric analysis:

Species should be soluble in either solvent. Suspensions are not to be allowed.

it should be coloured or form coloured complex.

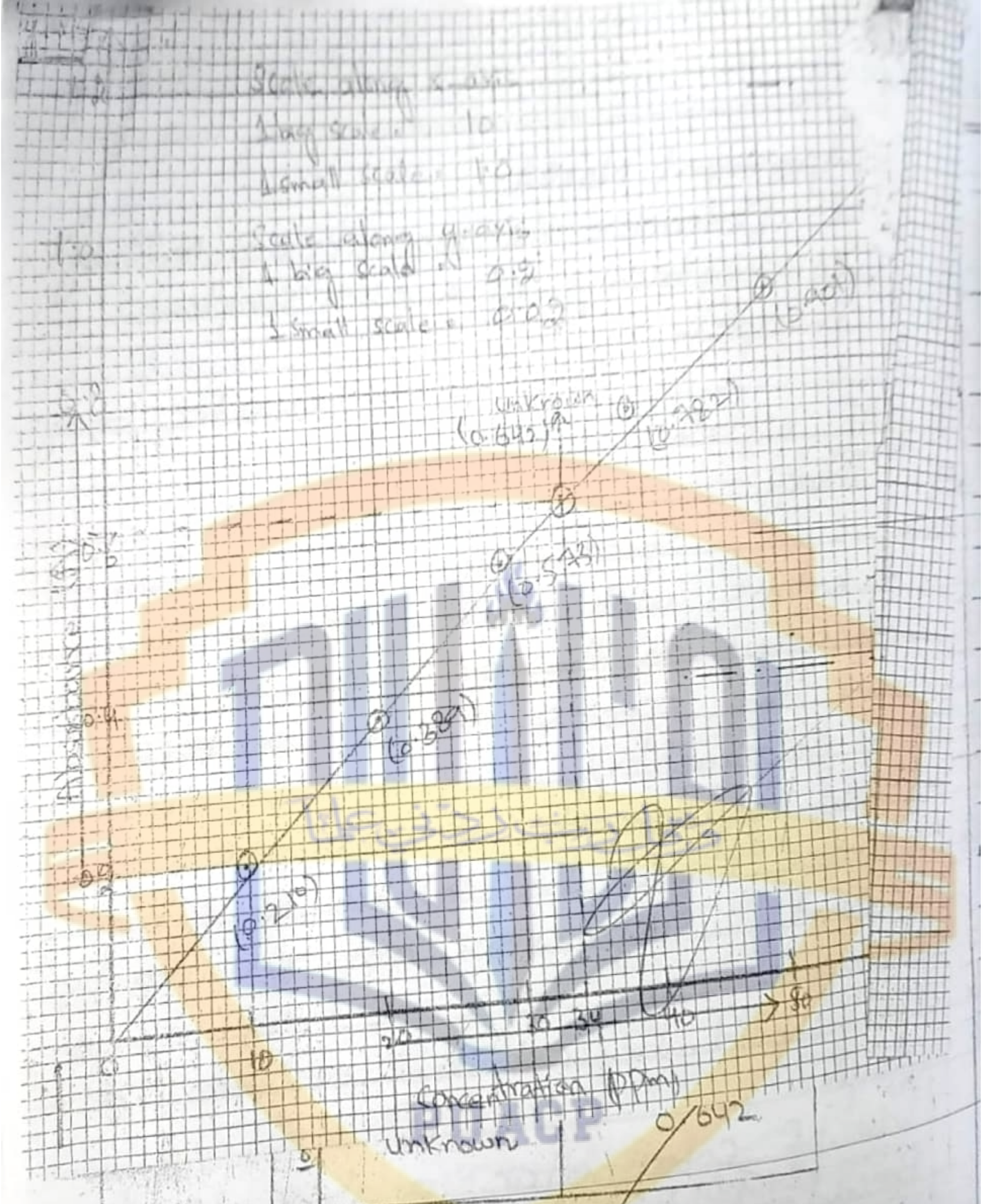
Law of colorimetric analysis is the Beer-Lambert law;

$$A \propto Cl$$

$$A = \varepsilon Cl$$

Procedure:-

First of all preparation of following solh. are done.



→ Result:-

The conc. of unknown sample is at absorbance of 0.642 is 34 ppm.

Metal ion solution i.e Ni^{2+} 0.45g of $NiSO_4 \cdot 6H_2O$ in 100mL flask and upto mark it with distilled H_2O.
0.1% DMG solution was prepared in ethanol. 5% NH_3 solution was Prepared according to dilution formula.

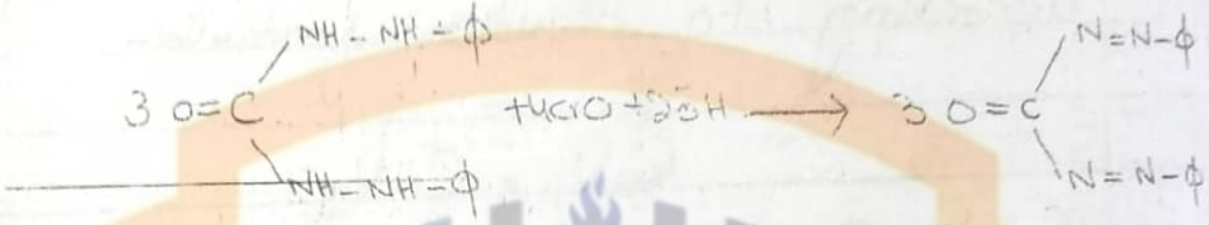
$$C_1V_1 = C_2V_2$$

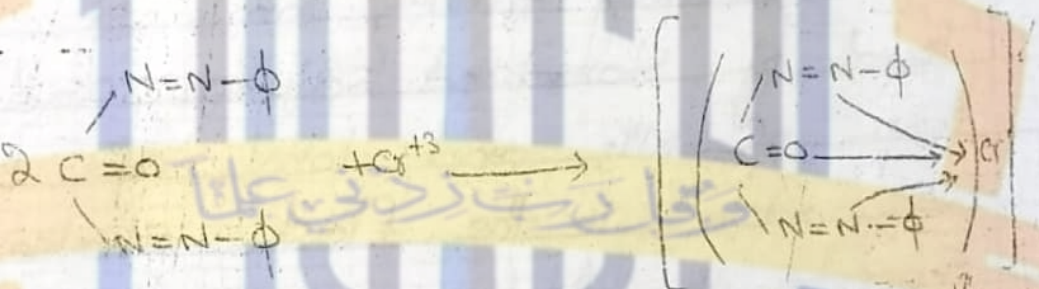
15 ml of NH_3 dissolved in 100mL flask In Seperating funnel, 10mL of $CHCl_3$, 10 ml of standard solution and 1 drop of NH_3 and 10mL of DMG, was added in the flask. Shaked and Seperate the lower organic layer, which is of yellow colour.
Spectronic was set at 0 by Putting the chloroform as blank solution.
Readings were taken with the help of Spectronic 20. The graph was plotted b/w concentration and absorbance which is straight line.

Experiment No: 28

Determine the amount/L of Cr^{+6} by using diphenyl Carbazide Colorimetrically.

→ Chemical Equation:-





Red-violet complex

→ ($K_2Cr_2O_7$)

$$\frac{\text{Mol.wt.}}{\text{in. mass}} = \frac{294.18}{52\times2} = \frac{294.18}{104} = \frac{2.82}{10} = 0.28\text{g/l}$$

→ 0.5% DFC Solution:-

0.25g of DFC in 100mL acetone.

→ 6M H_2SO_4

33mL of H_2SO_4 in 100mL flask.

Experiment No: 28

Determine the amount/L of Cr^{3+} by using Diphenyl carbazide colorimetrically:-

Principle:-

This is colorimetric analysis of Chromium.

Theory:-

Spectroscopy is interaction of radiations with matter. It can be classified on the basis of wavelength and species. Visible spectroscopy is the colorimetry.

Basic requirements of the colorimetric analysis.

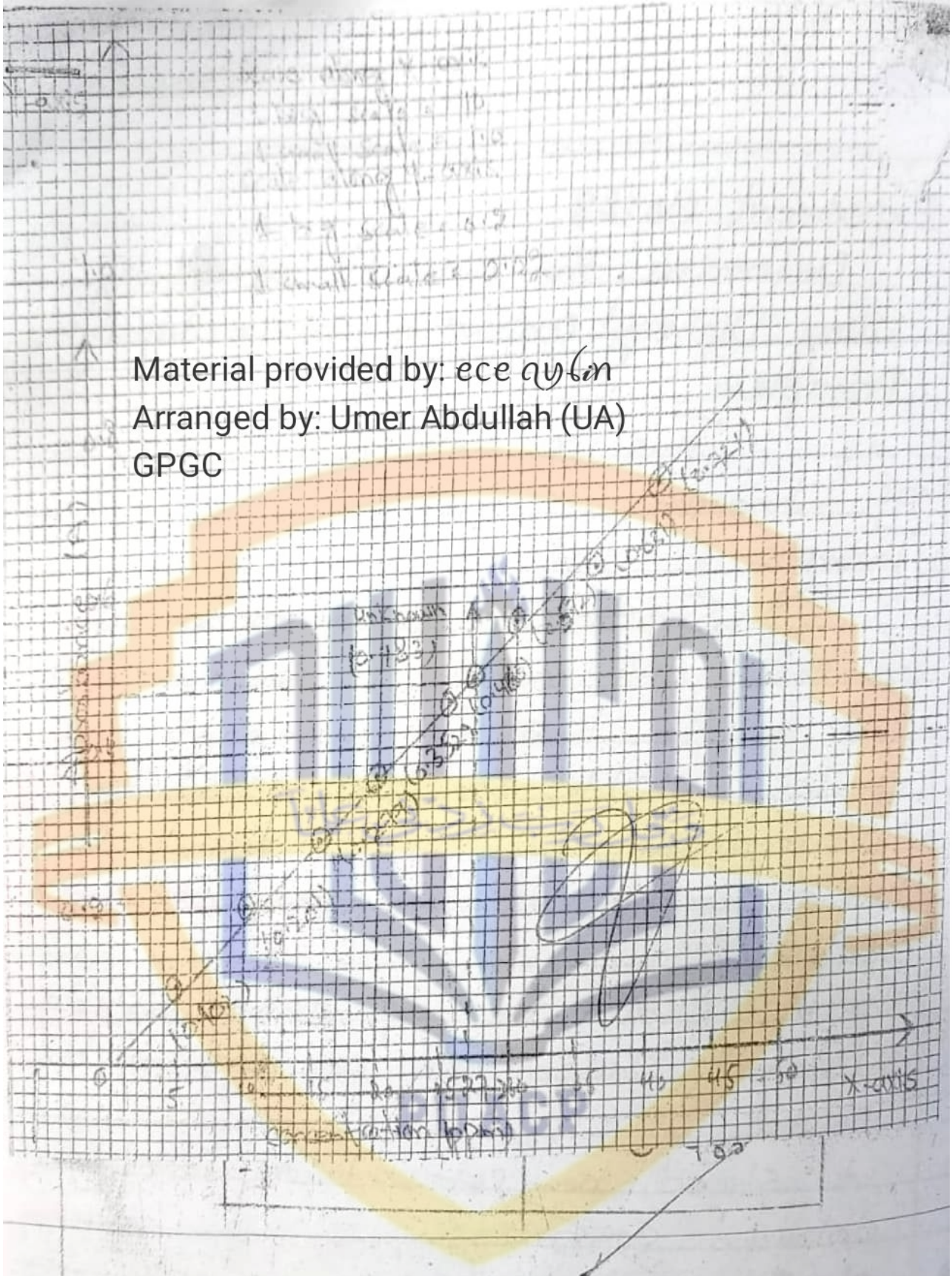
Species should be of either soluble in solvent. Suspensions are not to be allowed.

It should be coloured or form the coloured complexes.

Chemistry of Chromium:-

It has atomic no. 24 and group 6th d-block. It belongs to period 4th and transition series. Its atmoic wt. is 52 g/mol. Its electronic configuration is,

Material provided by: ece aylan
Arranged by: Umer Abdullah (UA)
GPGC



→ Result:-

The conc. of Cr^{+3} in unknown sample is 89 ppm.

$Cr = [Ar]\ 3d^5\ 4s^1$

It is steel grey lustrous hard and brittle metal. In larger amount it can be toxic and carcinogenic. The most prominent sample of Cr is the hexavalent Cr^{+6}.

Cr III salts are used for the tanning of leather.

Procedure:-

First of all metal salt soln. of $K_2Cr_2O_7$ in which Cr^{+6} is in +6 O. state was prepared by dissolving 0.28g in 100ml flask and upto mark with distilled H_2O.

0.25% of solution of diphenyl carbazide was prepared by dissolving 0.25g in 100ml acetone and upto mark with distilled H_2O.

Solution was taken in spectronic and reading was taken and graph was plotted b/w absorbance and concentration.