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M.Sc. Final

Inorganic Chemistry.

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Experiment 1

Object: To prepare & study the IR - spectrum of copper formate.

Apparatus:
Round bottom flask burate assembly heating mantle.

Chemical Required:

(i) Cupric Chloride ($CuCl_2 \cdot 2H_2O$) = 3·5 gm
(ii) Sodium formate ($HCOONa$) = 2·76 gm.
(iii) Methanol = 60ml

Procedure:

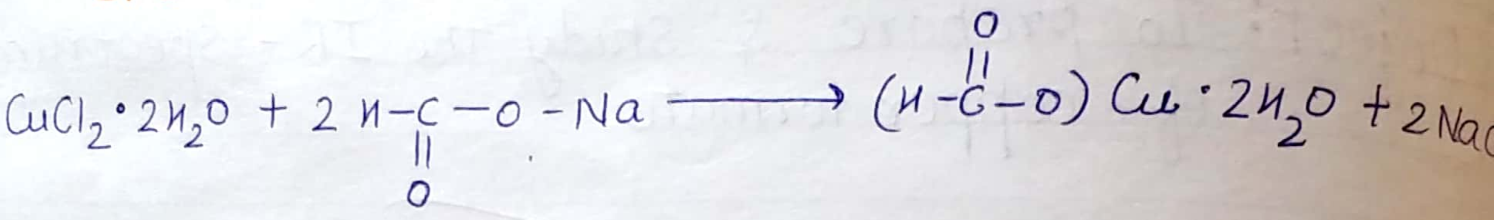
(i) First of all we dissolved 35 gm $CuCl_2 \cdot 2H_2O$ in minimum amount of CH_3OH & 2·76 gm of Sodium formate also dissolved in minimum amount of CH_3OH.

(ii) Mix bath the solution in round bottom flask make up. Sol^n with 60 ml of CH_3OH.

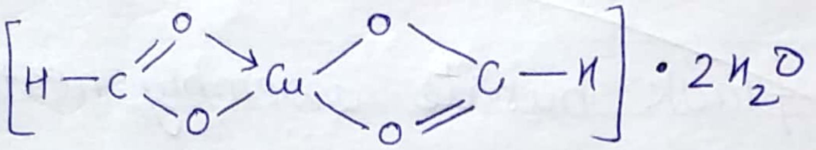
(iii) Reflux for about 2:30 hours & parrot green crystals are obtained.

(iv) wash with CH_3OH & acetone & dry the crystals.

Chemical Reaction :-



$$CuCl_2 \cdot 2H_2O + 2\,H-\underset{\underset{O}{\|}}{C}-O-Na \longrightarrow (H-\overset{\overset{O}{\|}}{C}-O)\,Cu \cdot 2H_2O + 2Na$$



$[Cu\,(HCOO)_2] \cdot 2H_2O$

$170gm$ of $CuCl_2 \cdot 2H_2O \longrightarrow 153$ gm of $(H(COO))_2 Cu \cdot 2H_2O$.

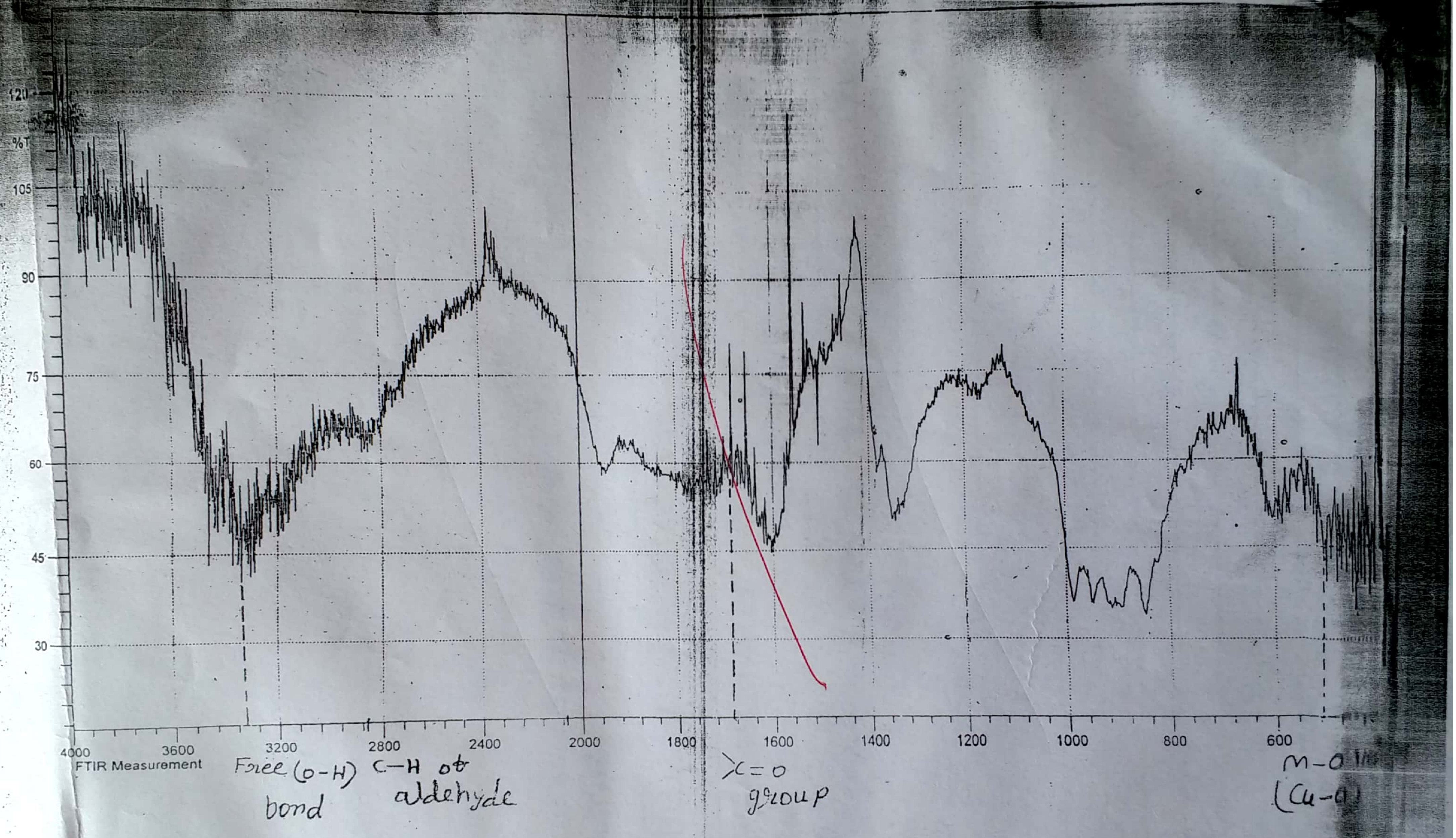
$1gm$ " " $\longrightarrow \frac{153}{170} gm$

3.5 gm " " $\longrightarrow \frac{153}{170} \times 3.5$ gm.

$= 3.15$ gm.

Expected yeild $= 3.15$ gm.
Observed yeild $= 2.70$ gm.

% yield $- \frac{2.70}{3.15} \times 100 = 85.71\%$.

120
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FTIR Measurement
Free (O-H) bond
C-H of aldehyde
>C=O group
M-O
(Cu-O)

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Yield:

2.70 gm of Parrot green dry of copper formate are obtained.

% yield = 85.71%

Precaution:

(i) Using the heating manthol carefully.

(ii) Dissolve $CuCl_2 \cdot 2H_2O$ & sodium formate properly.

Study of IR spectra:

Bond assignment	ν_{free}	$\nu_{cm^{-1}}$
ν_{O-H}	$3420 cm^{-1}$	$3400 cm^{-1}$
ν_{C-H} (Aldehyde)		$2800 cm^{-1}$
$\nu_{C=O}$ (carbonyl)	$1746 cm^{-1}$	$1700 cm^{-1}$
ν_{Cu-O}		510~~0~~ cm^{-1}

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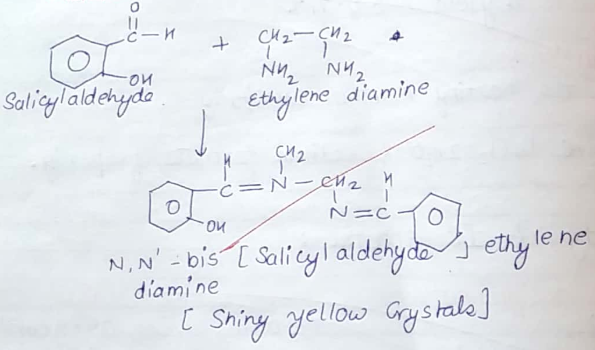
Chemical Rxn :



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Experiment - 2.

Object :

To prepare & Study the IR Spectra of the Cobalt sulfer complex (via N-N bisaliahdehyde. ethylene diamedimine are Sol^n.

Step 1: Preparation N,N bis (Salicylaldehyde) ethylene $ diamine or salenH_2

Chemical required -

Salicylaldehyde - 4·2 ml
Ethylene diamine - 1·3 ml
Ethyl Alcohol

Procedure -

(i) Took 4·2 ml of Salicylaldehyde in 50ml boiling 90gm ethanol

(ii) Added 1·3 ml of ethylene diamine to the above reaction mix with constsiring for about 3 to 4 min

(iii) $ Cooled the Sol^n in an ice bath.

(iv) Shiny yellow crystals of Solvent where seperated out.

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5.) filtered the bright yellow crystals and washed with a small vol^m of ethar.

6) Re crystalised with alcohol and dreied in air

Yield:-

4·25gm of soleh H_2

IR spectra :-

There was a sharp intense peak at about 360 cm^{-1} due to OH group. ethane was a wide intense peak the b/w 3030 – 360 cm^{-1} which the presence of AV-H. group.

there was a sharp as well as intense peak b/w 1485 – 1440 cm^{-1} which show the presence of an asymmetric deformation of $-CH_2$ group.

- A wide peek was absored at 745 – 44^{-1} due to presence of disubstitited (artho) aromatic hydro carbon.

- (x = H) group peak absorved at about 3300 cm^{-1}

theoretically:- 2.68 gm of solen H_2 gives the cobalt + solen complex = 325 gm.

∴ 1 gm of solen H_2 gives the cobalt comp. = $\frac{325}{268}$

∴ 2.34 gm sol solen H_2 gives the cobalt solen complex $= \frac{325}{268} \times 2.34 = 2.83$ gm.

observed yield of the complex = 2.35 gm.

∴ % yield $= \frac{\text{observed yield}}{\text{Theoritically yield}} \times 100$

$= \frac{2.35}{2.83} \times 100 = 83.03\%$.

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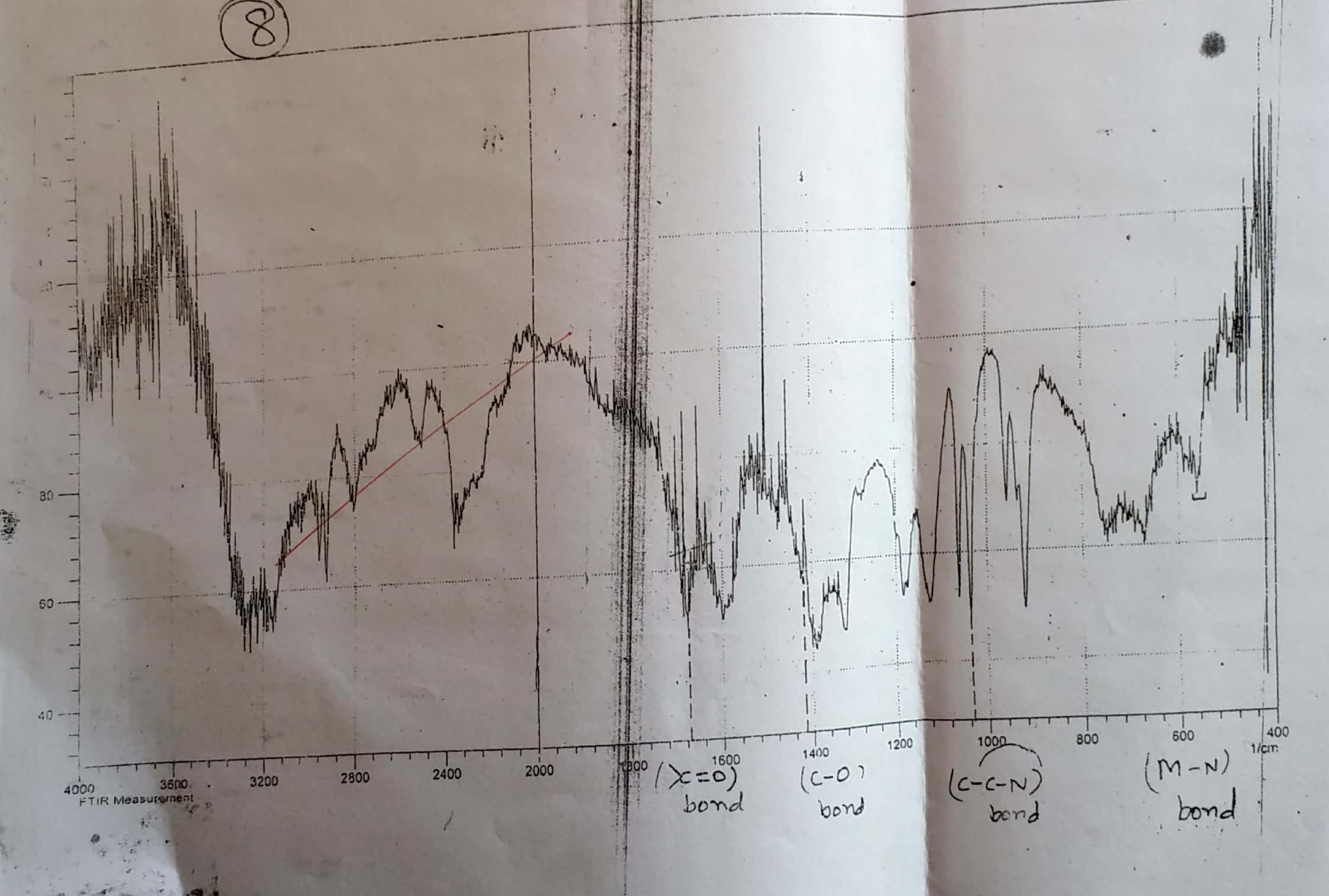
IR spectra of cis[Cu(glycinato)$_2$]·H_2O

⑧



Step-II^{nd}:-

Preparation of cobalt solen. complex seeb:- N, N' bis(solicylod ehyde) ethylene di'amine (Cobalt II)

Chemical Required:-

Solen H_2 - 2.34 gm.
$cacl_2$ crystals - 2.66 gm
Distil water - 50 ml
$NaoH$ pallet - 4 gm.

Procedure:-

1.) 4 gm of $NaoH$ Pallet was taken and dissolved in water (30 ml of distill water and make a clear sol^n.

2.) 2.34 gm of finely powdered solen H_2 then added to the above sol^n.

3.) prepared a $cacl_2$ sol^n in anathen beaker by dissolving 2.66 gm of $cacl_2$ crystal in 50 ml of distill water.

4.) then the $cocl_2$ sol^n was added to the above sol^n with constant stirring

5.) A brown geletions precipitat was sepreted out

6.) filtered the brown colour precipitate and recrystallsed it with chloroform.

Yield - 2.35 gm of cobat solen complex

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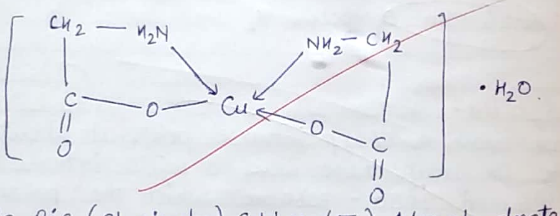

Cis-Bis (glycinato) Copper (II) Monohydrate

Experiment 3

Object:

To prepare & Study of IR-Spectra of the geometrical isomer of a cordination complex cis & trans bis (glycinato) copper II monohydrated.

1. Cis $[Cu(glycinato)_2] \cdot H_2O$
2. Trans $[Cu(glycinato)_2] \cdot H_2O$.

Chemical Required:

(i) Copper (II) acetate monohydrate - 20gm.
(ii) Ethanol - 25ml
(iii) Glycine - 15gm.

Chemical Rxn:

$$Cu(CH_3COO)_2 \cdot H_2O + 2\,NH_2-CH_2-COOH + C_2H_5$$

$$\downarrow$$

$$cis\ [Cu(glycinato)_2] \cdot H_2O + 2\,CH_3COOH.$$

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Procedure:

(i) 2 gm of Copper (II) acetate mono hydrate dissolved in 25 ~~gm~~ ml of hot water than 25 ml of hot ethanol was added to this.

(ii) In other beaker dissolved 1·5 gm glycine in water

(iii) Then heat the copper (II) acetate solⁿ (temperature) should be min to 70°c.
Then glycine solⁿ was added to it with constant stirring.

(iv) Left the above solⁿ 3·5 min. than it was cooled in an ice bath

(v) Blue Coloured crystals of cis-bis (glycinato) copper (II) monohydrated Complex were obtained.

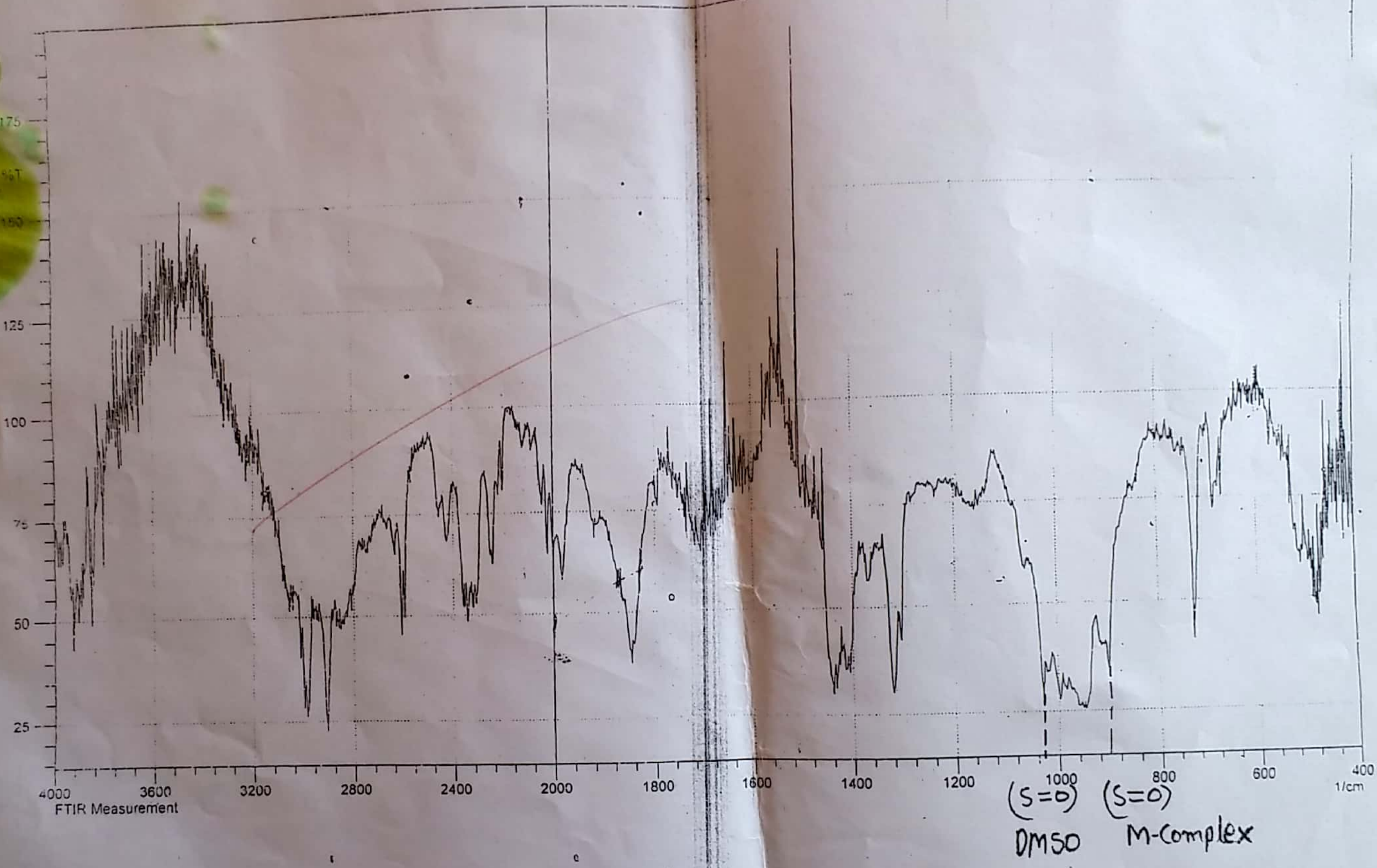
(vi) filtered & washed with water & ethanol and drided it

yield : 2·35 gm.

Study of IR Spectra:

ν_{O-H} = H_2O — 3484 cm^{-1}
$\nu_{C=O}$ bond — 1650 cm^{-1}
ν_{C-O} bond — 1405 cm^{-1}

$[CuCl_2(DMSO)_2]$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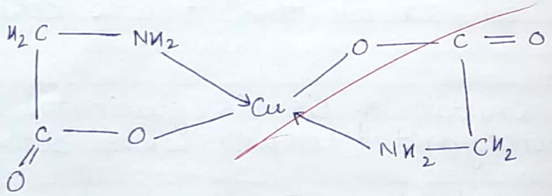
(S=O)
M-Complex



Trans - bis [glycinato] copper -(II) monohydrate

ν_{M-N} bond (cis)	485 cm^{-1}
ν_{M-O} bond (cis)	342 cm^{-1}
ν_{C-C-N} bond	1038 cm^{-1}
ν_{NH_2}	3152 cm^{-1}

→ Preparation of trans-bis (glycinato) Copper Monohydrated Complex –

Chemical Required :

(i) Cis [Cu (glycinato)$_2$] · H_2O

(ii) glycine – 1·0 gm.

(iii) filterate of cis [Cu (glycinato)] · H_2O – 15 ml

Procedure :

(i) 1·5 gm of ~~is~~ cis - copper (glycinato) · H_2O was taken in about 15 ml cis [Cu (glycenato] H_2O.

(ii) When 1 gm of glycine was added to it.

(iii) Above solⁿ was refulxed for 1 hr.

(iv) Shiny blue crystals of trans-bis [Cu (glycinato)$_2$] · H_2O was obtained.

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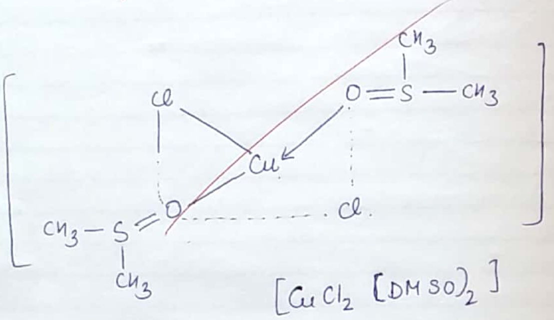
(iv) Filtered & than the crystals was air dried.

Yield :

1·05 gm.

Structure formula-



$[CuCl_2 [DMSO)_2]$

Experiment 4

Object:

Prepare the dimethyl Sulphur oxide complex compound of Copper (II) or $[CuCl_2(DMSO)_2]$ It's IR Spectra Study.

Chemical Required:

(i) $CuCl_2$ - 1·34gm
(ii) DMSO Sol^n - 2ml
(iii) Ethanol - 10ml

Procedure -

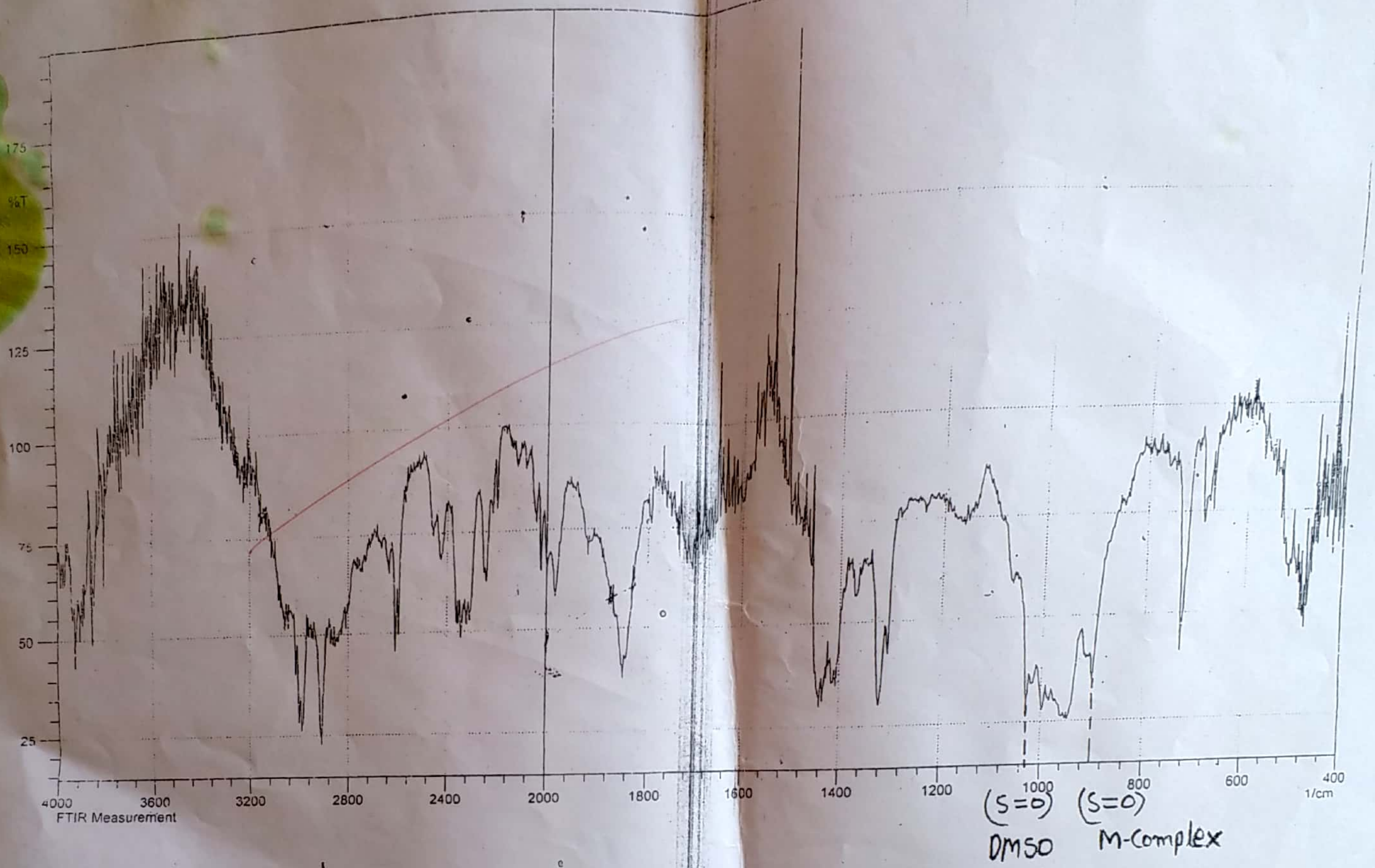
(i) Took 1·34 gm crystals of $CuCl_2$ and added 10ml ethanol to it.

(ii) Than added 2ml of DMSO sol^n to the above Sol^n & Stirred continously.

(iii) An exothermic Rxn accured & a light green coloured ppt. was obtained on cooling & constant stirring with glass rod

(iv) Filtered the ppt. with cold ethanol and recrystallised it with not ethanol.

$[CuCl_2(DMSO)_2]$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1/cm
FTIR Measurement
(S=O)
DMSO
(S=O)
M-complex

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Yield :

2·45 gm.

IR spectra :

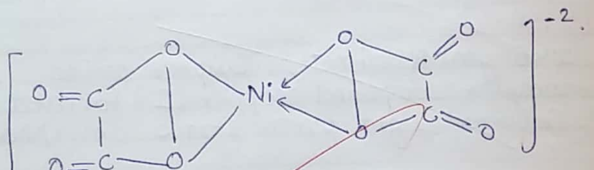
$\nu_{S=O}$ in DMSO = 1050 cm^{-1}

$\nu_{S=O}$ in complex = 923 cm^{-1}

Result :

2·45 gm of dimethyl Sulpher oxide complex compound copper (II) formed Coloured light green states amorphous.

Structural formula -



yield - 237 gm $NiCl_2 \cdot 6H_2O$ — 281 gm.

1 gm " " — $\frac{281}{237}$ gm.

1.189 gm " " $= \frac{281}{237} \times 1.189$.

$= 1.41$ gm.

Expected yield = 1.41 gm.

Observed yield = 1.30 gm.

% yield $= \frac{1.30}{1.41} \times 100$

$= 92.19\%$.

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Experiment - 5

Object:

To prepare & IR spectra study of bis oxalato nickel~~ate~~(II) anion.

Chemical Required:

(i) Nickel Chloride = 1.18 gm
(ii) Methanol = 40 ml
(iii) Sodium oxalate = 1.34 gm

Chemical Reaction -

$$NiCl_2 + 2Na_2C_2O_4 \longrightarrow Na_2[Ni(C_2O_4)] + 2NaCl.$$

$$Na_2[Ni(C_2O_4)_2] \longrightarrow 2Na^+ + [Ni(C_2O_4)_2]$$

Procedure:

(i) 1.189 gm of $NiCl_2 \cdot 6H_2O$ was taken in a round bottom flask & dissolved it 40 ml methanol

(ii) Then 1.34 gm of Sodium oxolate was added to the above Sol^n^ & refluxed the contents for about 2½ hours.

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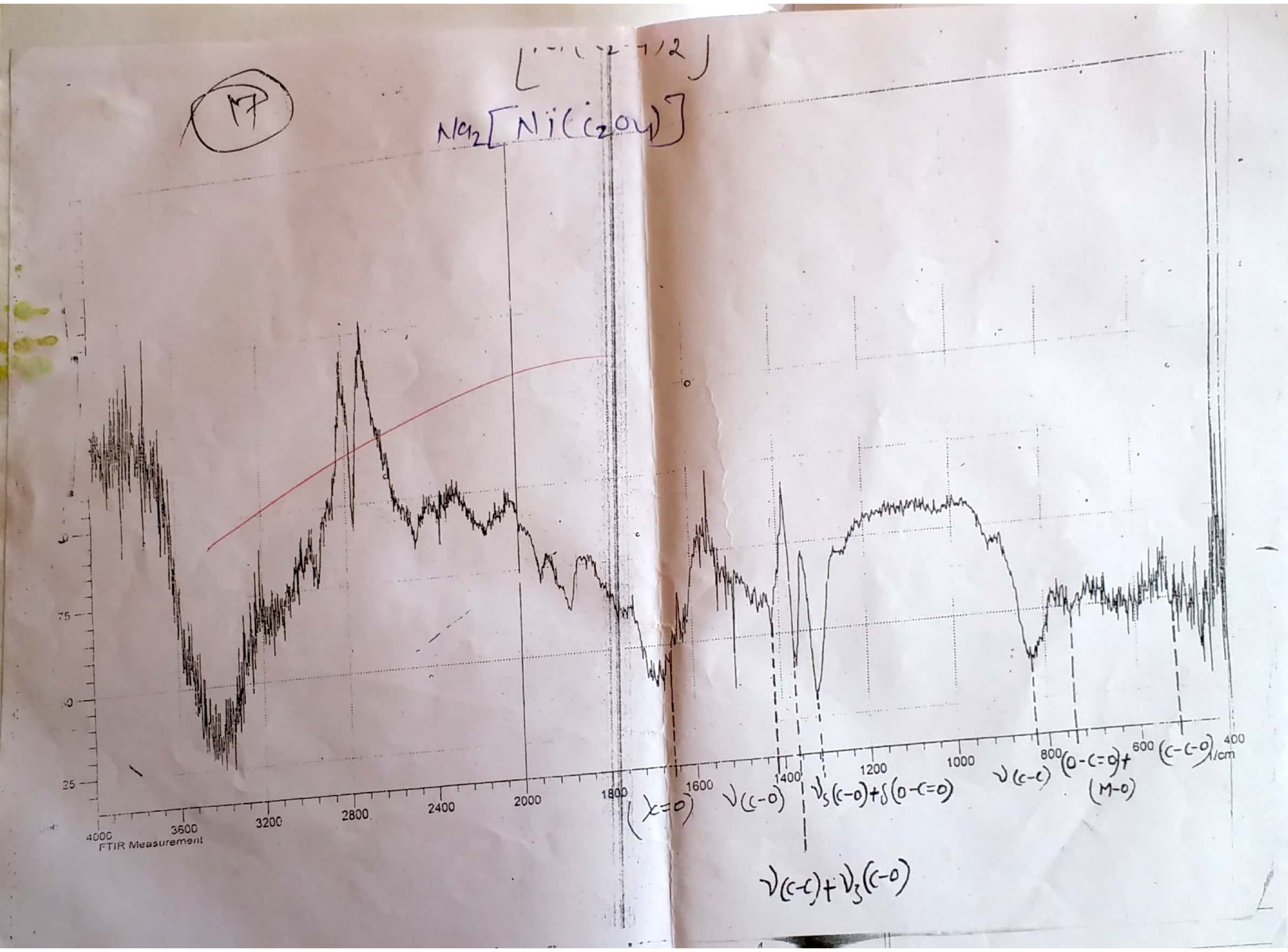17
[Ni(C_2O_4)_2]
Na_2[Ni(C_2O_4)]
25
50
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4000
3600
3200
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400
1/cm
FTIR Measurement
(C=O)
ν(C-O)
ν_s(C-O)+δ(O-C=O)
ν(C-C)+ν_3(C-O)
ν(C-C)
(O-C=O)+
(M-O)
(C-C-O)

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iii) Light green coloured crystals of bis (oxalato) nickleate (II) anion were reported out.

iv) Filtered the light green participate washed bath methanol & acetone.

Yield:

30 gm of Sodium Salt of bis (oxalato) Nickalate (II) Complex compound

IR Spectra:

Bond assignment	$\nu_{cm^{-1}}$ (free)	$\nu_{cm^{-1}}$ (comp.)
ν_{OH}	3420 cm^{-1}	3420 cm^{-1}
$\nu_{as}(C=O)$	1630 cm^{-1}	1630 cm^{-1}
$\nu_s(C=O)$	1630 cm^{-1}	1420 cm^{-1}
ν_{C-C-H} $\nu_s(C-O)$	1335 cm^{-1}	1380 cm^{-1}
$\nu_s(C-O) + \nu(O=C=O)$	1016 cm^{-1}	1320 cm^{-1}
ν_{C-C}.		840 cm^{-1}
$\nu_{(C-C-O)}$		540 cm^{-1}
$\nu_{C-C} + \nu_{M-O}$		510 cm^{-1}
$\nu_{(C-C-O)} + \nu_{(M-O)}$	518 cm^{-1}	472 cm^{-1}
$\nu_{m(O-C=O)} + \nu_{(M-O)}$	768 cm^{-1}	760 cm^{-1}

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Result :

1·30 gm of Sodium salt of bis (oxalato) Nicklate (II) (cis) formed.

Colour- Cd - light green.

28/4/18

Experiment 6

Object:

To prepare & Study ~~of~~ IR spectra of bis(oxalato) Cuprate (II) anion.

Chemical Requied:

(i) $CuCl_2 \cdot 2H_2O$ — 0·85gm
(ii) Methanol — 40ml
(iii) Sodium Oxalate — 1·34 gm.

Chemical Reaction :

$$CuCl_2 + 2NaC_2O_4 \longrightarrow Na_2[Cu(C_2O_4)_2] + 2NaCl$$

$$Na_2[Cu(C_2O_4)_2] \longrightarrow 2Na^+ + [Cu(C_2O_4)_2]$$

Procedure :

(i) 0·085 gm of $CaCl_2 \cdot H_2O$ was taken in a round bottom flask & dissolved it into 40ml of methanol.

(ii) Then 1·34 gm of Sodium oxalate we added to the above Solution & refluxed the contents for above 2½ hours.

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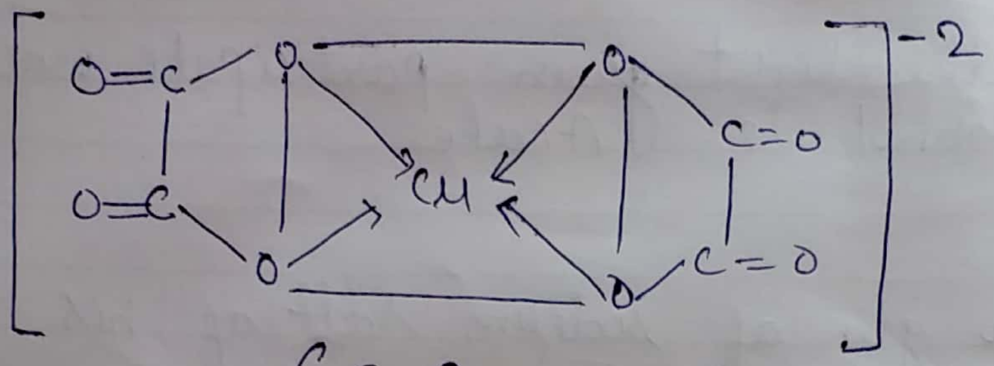
Structure formula.



$[Cu(C_2O_4)]^{-2}$

Yield :-

170 gm $CuCl_2 \cdot 2H_2O$ → 285.54 gm of $Na_2[Cu(C_2O_4)_2]$

1 gm " " → $\frac{285.54}{170}$ gm

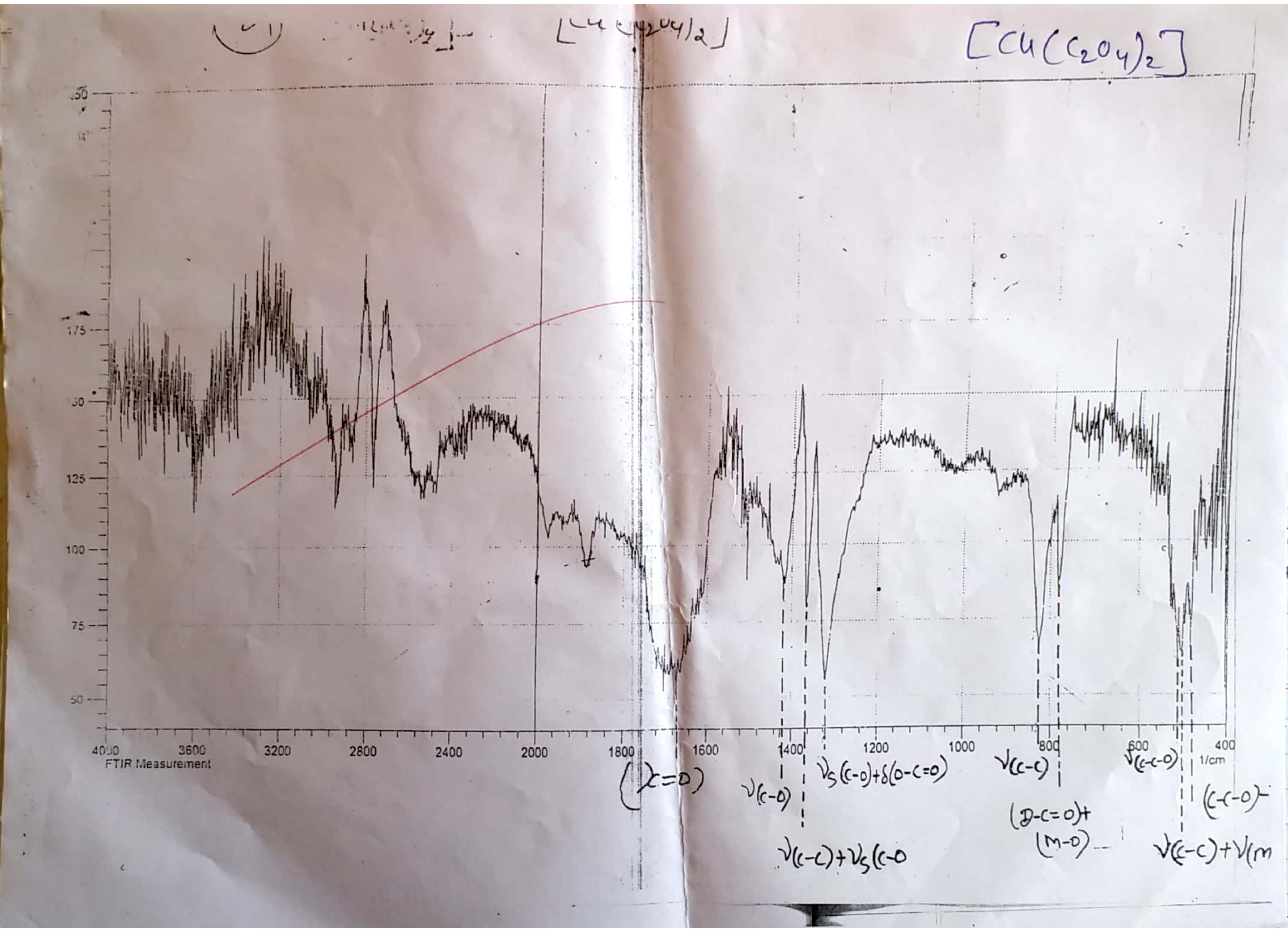
85 gm " $\frac{285.54}{170} \times 0.85$ gm

Expected yield . 1.42 gm

observed yield - 1.42 gm

% yield = $\frac{1.25}{1.42} \times 100 = 88.02\%$

$[Cu(C_2O_4)_2]$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FTIR Measurement
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ν(C-O)
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ν(C-C)+νs(C-O
ν(C-C)
(δ-C=O)+
(M-O)
δ(C-C-O)
(C-C-O)
ν(C-C)+ν(M

(iii) Light blue coloured crystals of bis (oxalato) Cuprate(II) anion we separated our.

(iv) Piltered the light blue green ppt. washed with the methanol & acetone.

Yield:

1·25 gm of Sodium Salt bis (oxalato) Cuprate(II)

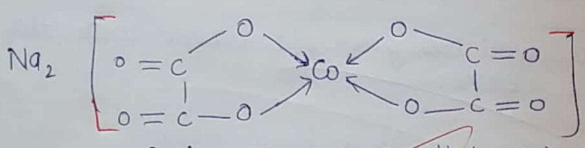
IR - Spectra -

Bond Assignment	$\nu_{cm^{-1}}$ free	$\nu_{cm^{-1}}$ (comp.)
ν_{OH}.		3400 cm^{-1}
$\nu_{as}(C=O)$	1630	1635 cm^{-1}
$\nu_s(C-O)$		1415 cm^{-1}
$\nu_{C-C} + \nu_s(C-O)$	1335 cm^{-1}	1360 cm^{-1}
$\nu_s(C-O) + \delta(O-C=O)$	1316 cm^{-1}	1312 cm^{-1}
ν_{C-C}	–	880 cm^{-1}
$\delta_{an}(O-C=O) + \nu_{M-O}$	76·8 cm^{-1}	785 cm^{-1}
$\delta_a(C-C-O)$	–	530 cm^{-1}
$\nu_{C-C} + \nu_{M-O}$	–	510 cm^{-1}
$\delta_s(C-C-O) + \nu_{(M,O)}$	518	485 cm^{-1}

Result:

1·2 gm of Sodium Salt of the bis (oxalato) Cuprate (II)

Structural formula :⇒



Sodium dioxalato Cobaltate (II) Complex.

Yield :⇒

238 gm $CoCl_2 \cdot 6H_2O$ ⟶ 281 gm of $Na_2[Co(C_2O_4)_2]$

1 gm " " —— $\frac{281}{238} \times 1{\cdot}14$ gm

1 gm " " —— $= 1{\cdot}14$ gm.

Expected yield – 1·14 gm.
Observed yield – 1·32 gm.

% yield $= \frac{1{\cdot}32}{1{\cdot}41} \times 100$.

$= 92{\cdot}96$ %.

Experiment 7

Object -

To prepare and study of IR spectra of Sodium dioxalato cobaltate (II) Complex compound.

Chemical Required:

(i) Cobalt Chloride – 1·19 gm.
(ii) Sodium Oxalate – 1·34 gm.
(iii) Methanol – 60 ml

Chemical Rxn:

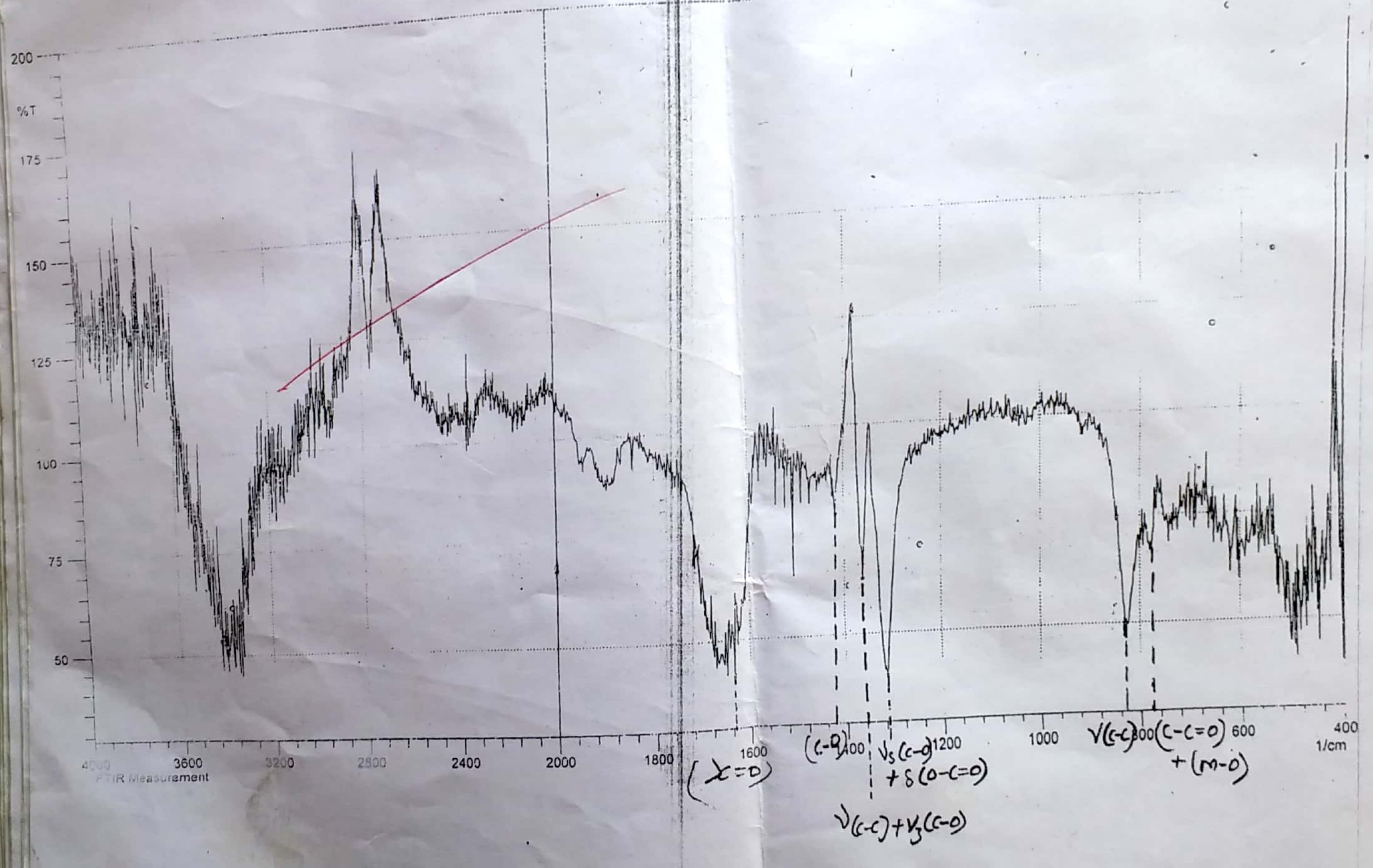
$$CoCl_2 \cdot 6H_2O + \begin{array}{l} OC - ONa \\ | \\ C - ONa \end{array} \longrightarrow Na_2[Co(C_2O_4)_2] + 2NaCl + 2H_2O$$

Procedure :-

(i) Took 1·19 gm $CoCl_2 \cdot 6H_2O$ in round bottom flask & dissolved it in bottom flask & dissolved it in 40 ml methanol properly.

(ii) Then 1·34 gm sodium oxalate was added in it.

$Na_2[Co(C_2O_4)_2]$ / 25



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ii) It was shake well & their refluxed for 2·30 hr.

v) After refluxing it was filtered & separated.

v) Wash it with methanol & a ethier dried it.

i) Pink colour Crystals are obtained.

Yield :

1·32 gm.

IR Spectra

Bond assigment	$\nu_{cm^{-1}}$ complex.
ν_{OH}	3410 cm^{-1}
$\nu_{as}(C=O)$	1625 cm^{-1}
$\nu_{s}(C-O)$	1410 cm^{-1}
$\nu_{C-C} + \nu_{s}(C-O)$	1380 cm^{-1}
$\nu_{s}(C-O) + \delta\nu(O-C=O)$	1320 cm^{-1}
ν_{C-C}	860 cm^{-1}
$\nu_{aN}(O-C=O)$	780 cm^{-1}
$\nu_{a}(C-C-O)$	545 cm^{-1}
$\nu_{(C-C)} + \nu(M-O)$	510 cm^{-1}
$\nu_{s}(C-C-O) + \nu(M-O)$	495 cm^{-1}

Result:⇒

Yield :⇒ ~~[illegible]~~ 1·32 gm of Sodium dioxalato Cobalt II Complen formed

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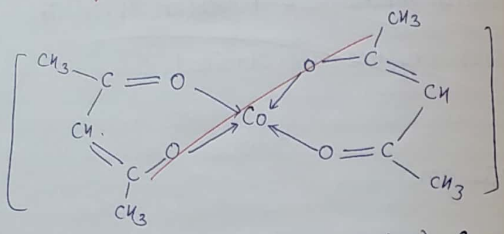
Structural formula -



[Bis-(acetyl acetonato) Cobalt (II) Complex]

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Experiment 8

Object:

To prepare bis (acetylacetonato) Cobalt (II) Complex compound & Study of IR-Spectra.

Chemical Required:

(i) $CoCl_2 \cdot 6H_2O$ — 1·19 gm
(ii) Acetylacetone — 2·2 ml
(iii) Methanol — 40 ml
(iv) ~~Anhy~~ Aqueous NH_3 Sol^n — as required.

Procedure -

(i) Dissolved 1·19 gm of $CoCl_2 \cdot 6H_2O$ in 40 ml of methanol

(ii) Then 2·2 ml Cu· actylaccetone ~~in 40~~ was added to the colour solution with constant stirring for about three to four miniutes

(iii) Reflux the above Sol^n for above 3½ hours the solution about 5·6 ml by adding 1:4 ppt. was not observed.

(iv) Filtered the ppt. & washed with methanol & Acetone.

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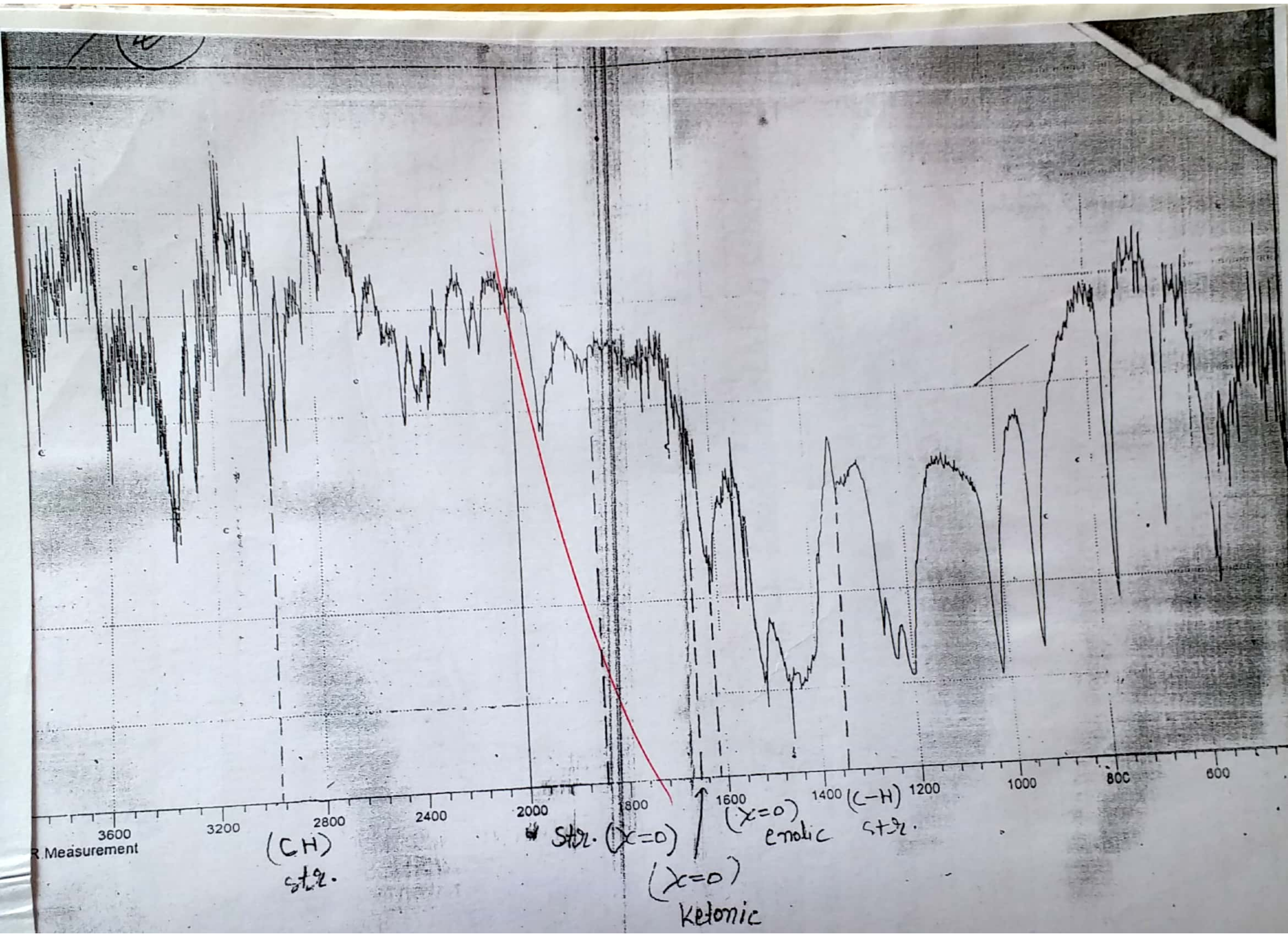
3600
3200
2800
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R Measurement
(CH) str.
str. (C=O)
(C=O) ketonic
(C=O) enolic
(C-H) str.

Yield:

2·01 gm of Bis (acetyl acetonato) cobalt(II) complex compound.

IR Spectrum:

Bond assignment	$\nu_{cm^{-1}}$ complex
C-H Streching	2960 - 2850 cm^{-1}
$>C=O$ streaching	1850 cm^{-1}
keton $>C=O$S	1725 cm^{-1}
enolic $>C=O$S	1630 cm^{-1}
C-H Streching	1390 - 1370 cm^{-1}

Result:

2·01 gm of Bis (acetyl acetonato) Cobalt(II) Complex compound formed.

Colour-

Pink.

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Dissolved the tetrathionate into minimum quantity of water 35°C temp. & resprecipitate it by adding ethanol.

filter the product & dry it

Yield:

2·8 gm.

Result:

2·8gm of Sodium tetrathionate formed.

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chemical rexn

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

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Object →

To prepare sodium tetrathianato [$Na_2S_4O_6$]

Chemical required →

Sodium thio sulphato - 4 gm
Iodine - 4 gm
ethanal - 40 ml

procedure →

mix sodium thiosulphate (4 gm.) & Iodine (4 gm) into a paste withe water 2m

→ Add ethanol in a above solⁿ and leave the mixture for 16-hours at reum temperature

→ filter and add the sodium tetrathionalte in a beaker and wash the prosedure product with ethanol to removes and erressa of Iodine

→ Dissolue the tetrathionate in the minimum aquantitiy of warm water (35°C)

→ and repprecipitate it by adding ethand

→ Filter the product and dry it

Yield - 2·8 gm

Result → 2·8 gm of sodium tetra thionate formed

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Object →

To seprate the mixture [Cu^{+2} & cd^{+2}] in to individual components using paper chromatography and determinting Rf value

Theory →

Paper chromatography in based on the difference in the rate ~~on the~~ at which the components of a mixture move through midium called stalinry phase under the influence of some solvent called moving phase. In this technique a drop of metal soln is applid as some small spot one what mam filter paper & dried the spot.

The spot should be near the edge of the filter paper then the paper is placed into a devloping solvent the cation containing in mixture are moved by solvent has moves there cation to a Soluble.

hight (15 = 20mit)

then the paper is dried up and the various spot are visudised by suitable reagents called visualisiving reagent the movement of cation realative to the solvent is expressed in terms of Rf value

Distance travelled by the substance from

The origion line

R.F Distance traveled by the solvent from origin line

R.f → Retention factor

Salt sol^{n} →

1% sol^{n} is prepased in the distilled water using cupric chloride ($CuCl_2$) cadmium chloride ($CdCl_2$) & acidified their sol^{n} with con: H_2SO_4

Developing solvent →

M Butanal saturated with 3N HCl prepare the solvent by shaking up equal volume of the two components in a seprating funnl reject the lower aqous layer and use the paper organic phase for chromatography.

visualising sol^{n} →

0.05% dithiozone sol^{n} take 0.05 gm dithiozone and mixed in 100 ml ~~ethanol~~ chloroform

→ seperated and dried whatman paper no-2 to visualised the colour at moved contains.

calculation for R.f value →

for cu^{+2} R.f = $\frac{2}{13}$ = 0.15

for cd^{+2} R.f = $\frac{9}{13}$ = 0.69.

Procedures →

→ All the first take a whatman no.1 filter paper strip 25 cm ~~75~~ 7cm

→ A line is drown strip by a led pencial

→ .This line should be about 2cm. from. & end of the filter ~~to~~ location of the spot is marked with xcm thin line with pencil

→ After draying the spots filter paper

→ strip is fixed vartically in chromatography chamber.

→ The paper should be diped in 1cm into the developing solved when solvent is travelled a reasonable height (15–20 cm) the filter paper is taken out and allowe to dry.

→ The spots are were sparcayed with 0.05%. dithiozone in CHCl3 the various spots aquied the following colours

cu^{2+} → purple brown
cd^{+2} → purple pink

The distance travelled by various contain from origion line are followed

Cu^{2+} → purple brown
Cd^{+2} → purple pink

→ The distance travelled by the solvent is
Cu^{+2} = 2cm
Cd^{+2} = 9cm

Result →
The various cation are separated by paper chromatography and R.F value for various cation are

Cu^{+2} → 0.15
Cd^{+2} → 0.69

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Object →

To determine nickle and zink gravimatrically and also determine the strength of copper volumetrically.

Sol^n prepared →

→ N/25 $CuSO_4 \cdot 5H_2O$ Sol^n.

→ Sodium thio sulphate (hypo) $Na_2S_2O_3 \cdot 5H_2O$ Sol^n

→ Stark sol^n of $CuSO_4 \cdot 5H_2O$

→ Nickel ammonium sulphate $Ni(NH_3)SO_4 \cdot 6H_2O$

→ zink sol^n.

→ take 40 ml of each above solution in 250ml valumetric flask. and is made up by adding. distilled water

→ 50 ml of the sol^n. is taken for gravimetric. estrimation of copper

1) Standaredization of hypo.sol^n. →

1) This is done by tritation of hyposolen aganist N/25 $CuSO_4$ sol^n (Know

Observation table

S.No.	volume of $CuSO_4 \cdot 5H_2O$	volume of ~~Hypo~~ Hypo.Sol^n		
		Initial	final	co.n.
1	10 ml	0	11.3	11.3
2	10 ml	0	11.2	11.2
3	10 ml	0	11.2	11.2

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1) **Principle. Reaction ⇒**

$2CuSO_4 \cdot 5H_2O + 4KI \rightarrow K_2SO_4 + Cu_2I_2 + I_2 + 5H_2O$

$2Cu^{+2} + 4I^- \longrightarrow Cu_2I_2 + I_2$

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

$I_2 + 2S_2O_3^{-2} \longrightarrow 2I^- + S_4O_6^{-2}$

calculation ⇒

(1) **known standard $CuSO_4$ V/s Hypo**

$\frac{N}{25}$

$N_1V_1 = N_2V_2$

N_1 = Normality $CuSO_4$ soln

V_1 = volume of $CuSO_4$ soln $= 10$

$\frac{1}{25} \times 10 = N_2 \times 11.3$

N_2 = Normality of Hypo $= ?$

V_2 = volume of Hypo $= 11.2$

$N_2 = \frac{10 \times N}{25 \times 11.3} = 0.0353982N$

(2) **unknown (given) $CuSO_4$ V/s Hypo**

$N_3V_3 = N_4V_4$

N_3 ⇒ Normality of Hypo

V_3 ⇒ volume of Hypo

$N_4 = 0.038938N$

N_4 ⇒ Normality of unknown s

for Cu^{+2} ion

V_4 ⇒ volume of $CuSO_4 = 10$ml

3) **Strength of Cu^{+2} ions ⇒**

strength = Normality × liquid

$= 0.038980 \times 63.5$

$= 2.47444$ gm/Lit

Strength of Cu^{+2} ions in 1000 ml Sol^{n} = $\frac{2.4744}{1000}$.

" " " " " 250ml " = 0.6188 ml.

Amount of Cu^{+2} ions in 40 ml = 0.618861.

In " " " " 1000 ml = $\frac{0.618811 \times 1000}{40}$

So, strength of Cu^{+2} volumetrically = 15.4535.

For $CuSO_4$:

Strength of (8 gm/Litre) = ~~Normality × Equi.~~

~~= 0.389802 × 249.71~~

= 9.71 gm/Litre

Strength of $CuSO_4$ in 1000 ml prepared = 9.71 gm/litre

" " " " 25 ml " = $\frac{9.71}{1000} \times 25$.

= 2.4289 gm/litre.

$CuSO_4$ present in 250 ml (prepared Sol^{n}) $CuSO_4$

" " " 40 ml (original)

Amount of $CuSO_4$ in 40 ml = 2.4289 gms.

" " " " 1000 ml = $\frac{2.4289}{40} \times 1000$

= 60.6875 gm/Litre.

③ Volumetric analysis of $CuSO_4$ (Unknown) from the solution titrate the solution against standarlised Hypo solⁿ.

S.No.	Vol. of $CuSO_4 \cdot 5H_2O$	Hypo (ml)
1	10 ml	11·0 ml
2	10 ml	11·0 ml
3	10 ml	11·0 ml

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Titration of $CuSo_4$ v/s Hypo:

Took 10ml of $CuSo_4$ in the titration flask & hypo sol^n in the burate.

Now add dropwise drak yellow coluration add 5 ml of 10% KI Sol^n up to dark yellow coluration add hypo from the burate, fill till light yellow colouration is obtained.

Now we added starch as an indicator & again titrate till it becomes colourless note the total reading from burte.

Removal of Copper in the Solution -

Took 50ml of unknown solution in a 500ml beaker

Now add 2·5ml of conc. HCl, 30-40ml of the freshly prepared sulphours acid sol^n.

Dilute the solution upto 100 ml by adding water.

Heat to boiling, cool at the room temp. than 10% ammonium thio cynate Sol^n above (30ml) slowly with stirring.

After cooling the solution digest the ppt. by heating the beaker on a water bath by adding one or two drops of ammonium thio cynate sol^n pressure the filterate for estimation of Nickle.

Estimation of Ni Gravimatrically –

Wt. of empty crucible = 28·854 gram.
Wt. of Crucible with ppt. = 29·089 gm
Net Wt. = 0·235 gm.

Calculation for Ni –

Wt. of Ni (DMG) = 288·91.

28891 Nickel DMG = 58·69 gm.

0·235 " " $= \frac{58·69}{288·91} \times 0·235$

Ni 50 ml Sol^n = 0·04977386

" 250 ml " $= \frac{0·0477386}{50} \times 250$

= 0·238893.

- 250ml of Sol^n = 40ml of Sol^n

In 40ml Sol^n Ni present = 0·238693

In 100ml of Sol^n Ni $= \frac{0·23869}{40} \times 100$

= 5·967325 gm.

For Nickel Ammonium Sulphate –

288·91 NiDMG contains = 394·69 of Ni solⁿ

0·235 " " " = $\frac{394·69}{288·91} \times 0·235$

= 0·3210417.

Ni Ammonium Sulphate 50 ml = 0·3210417

" " in 250 ml = $\frac{0·3210417}{50} \times 250$

= 1·6052084.

In 40 ml Solⁿ of Ammonium sulphate

= 1·6052084

1000 ml Solⁿ of Ammonium Sulphate = $\frac{1·6052084.}{40.}$

= 40·13021.

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Gravimetric estimation of Nickel :

Transfer the filterate in a 500 ml beaker & add 20 ml concⁿ HCl & 30 ml concⁿ HNO_3 Evaporate the Solⁿ to 100 ml, cool it & dilute it up to 50 ml by adding distilled water.

Add 2-3 drops of distilled water. Now add 30 ml of 1·1 DMG with constant stirring. Now go on adding ammonia solution dropwise ~~th~~ till this solution become alkaline, digest the ppt. on a water bath for 30 min cool it & filter it through & dry it & weight it. Presence the filtrate for the estimation zinc

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Estimation of Zn (gravimetrically)

Wt. of empty Craible = 28·711 gm
Wt of crucible ppt. = 28·954. gm.
Net wt. = 0·243 gm.

M. Wt. of $Zn NH_4 PO_4$ = 178·3992.
178·3992 Zn & $H_4 PO_4$ contains = 81·38 ZnO.

0·243 gm " $= \frac{81 \cdot 38}{178 \cdot 39} \times 0 \cdot 243$

$= \cdot 1108492$ gm.

ZnO in ml = 0·1108492 gm.
ZnO in 50ml = 0·1108492 gm.
" " 250ml $= \frac{0 \cdot 1108492}{50} \times 250$.

$= 0 \cdot 554246$.

In 40 ml sol^n Zinc Oxide = 0·554246.

In 1000 ml Zinc oxide $= \frac{0 \cdot 554246}{40} \times 1000$

= 13·85 gm/litre.

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Gravimetric estimation of Zinc :

Transfer the filterate into beaker & add 35ml of HNO_3 & 15ml of HCl & dry the sol^n almost to dry.
Dissolve the residue in distilled water & diltute the sol^n upto 150ml & add one ml of methyl red indicator.
The colour of the sol^n becomes red.
Add dilute NH_3 sol^n very slowly untill the sol^n becomes just yellow.

To this Solution add 1gm of Solid. Ammonium acetate & 5gm of NH_4Cl, heat the sol^n to boiling & to it add 30ml of 10% diammonium hydrogen phosphate sol^n with constant Stirring.

Digest the ppt. for 30 min., this stage colour of the Sol^n should be yellow.
Filter ppt. through a G·V crucible wash it with 1% ammonium hydrogen phosphate.

After that wash it with 50% alcohols dry it & weight it.

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Experiment 19

Object:

To determine the amount of from present in the given Solution solution using Fe(II) 1·10 Phenonthroline complex.

Theory:

Iron (II) react with 1·10 Phenonthrdine from an red complex.

$$Fe^{+2} + 3(C_{12}H_8N_2) \longrightarrow \left[[C_{12}H_8N_2] Fe\right]^{+2}$$

The colour intensity is independent of the acidity in the pH range 2·9 and it stable for a long time Fe(III) may be reduced with hydrogylamine hydro Chloride adsorption is measured at 515 nm against a reagent blank using a digital spectrometer.

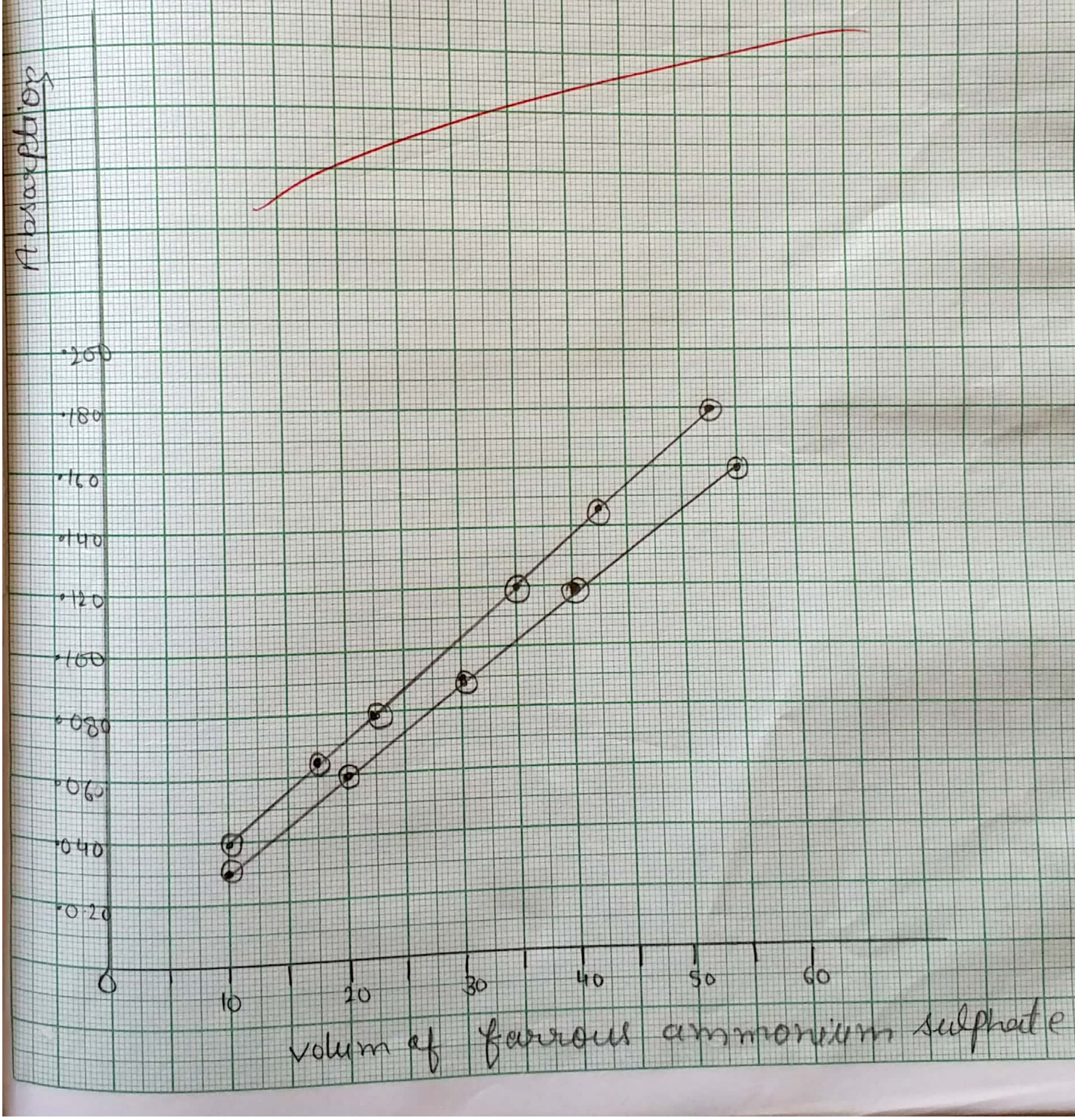
Chemical Required:

(i) Conc. H_2SO_4
(ii) Ferrous ammonium sulphate
(iii) 1,10 Phenanthroline
(iv) Hydroxylamine hydro Chloride
(v) Sodium acetate.

Scale

on σx axisis 1cm = 5ml

on σY axisis 1cm = ·0200 b carbon



Procedure (Preparation of the solution) :

(i) Dissolved 0·25 gm of ferrous ammonium sulphate in about 1·2 ml conc. H_2SO_4 & diluted it to the 250 ml with distilled water.

(ii) Prepared 0·25% , 1-10 Phenanthroline solution by dissolving 0·625 gm of the Salt in 250 ml at distilled water.

(iii) Prepared 0·2% sodium acetate solution by the dissolving 0·2 gm of CH_3COONa in 100 ml of the distilled water.

(iv) Dissolving 70gm of $NH_4OH \cdot HCl$ in 100 ml of the distilled water 10% Hydroxylamine hydrochloride Solution was prepared.

Calibration Curve for Iron :

(i) Took 10, 20, 30, 40, 50 ml & unknown ml of 10 PPm ferrous ammonium sulphate solution by '5' different really memembered 100 ml by measuring flask.

(ii) Added 5ml of hydroxylamine hydrochloride solution of reach of the measuring flask and added 5ml of sodium acetate buffer Sol^n also.

2.4

(iii) Then 5 ml of 1.10 Phenanthroline solution to each of measuring flask and diluted it up to 50 ml distilled water.

(iv) Measured absorbance of each solution at 510 nm using a digital spectrometer.

(v) Same procedure was done for unknown. As solution & absorptionce was measured for it.

(vi) Plot a graph of Absorbance (optical activity) against volume of 10 PPm (as soln).

(vii) Determine the amount of Iron present in the given unknown solution with the help of it's absorbance.

Observation table :

S.No.	Volume of ferrous A.S (ml)	Volume of hydr. HCl sol (ml)	Vol. of Sod. ac.	Vol. of 1,10 Phen.	Absor. (O·D)
1	10	5	5	5	0·030
2	20	5	5	5	0·060
3	30	5	5	5	0·96.

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		2.5			
4	40	5	5	5	0.120
5	50	5	5	5	0.160
6	Unknown	5	5	5	0.170

Observation :

Standard Calebration curve of the absorbance against concentration of standard solution was a straight line and determine the concentration at iron present in the unknown solution from it's absorbance.

Result :

The concentration of ferrous ion in a given sample (unknown) solution using the spectro Photometer by 1,0 Phenanthroline method is 53.

Surendra
28/4/18