CHAPTER III

A. EXPERIMENTAL DETAILS
B. METHODS OF ISOLATION OF COMPLEXES
C. EXPERIMENTAL DATA
EXPERIMENTAL DETAILS

1. Analysis:

Metallic content of Cobalt (II) complexes was estimated by pyridine method after digesting the complexes with concentrated $\text{H}_2\text{SO}_4$. In Copper (II) complexes metallic content was estimated by precipitating copper as copper salicylaldoxime and cuprous thiocyanate. The complexes were digested with concentrated $\text{HCl}$ and $\text{H}_2\text{SO}_4$ respectively before precipitation of Cu. Nickel was estimated as Nickeldimethyl glyoxime. Carbon, hydrogen, and nitrogen were estimated by usual microanalytical methods.

2. Molecular weight measurements:

Molecular weights of the complexes were determined by noting the depression in freezing point using nitrobenzene and water as solvents (depending on the solubility of the complexes).

3. Conductivity measurements:

Measurements were made in different solvents like alcohol,
nitrobenzene, formamide (depending on the solubility of complexes). Measurements were made at concentration of $10^{-3}$M.

4. **Magnetic susceptibility measurements:**

All magnetic measurements were made at room temperature. Solid complexes were finely powdered and filled in a Gouy's tube by constant tapping. Change in mass was noted in each case. The Gouy's tube was calibrated with A.R. tetraaquocopper (II) sulphatemonohydrate.

5. **Visible spectrophotometric measurements:**

All measurements were made with the help of Beckmann DU-2 spectrophotometer. Solvents like chloroform, nitrobenzene, and formamide were used. The spectra were taken at approximate concentration M/500.

6. **Infrared measurements:**

The spectra were taken as potassium bromide pellets on Perkin-Elmer spectrophotometer and infracord model 137.

7. **Chemicals and solvents:**

All chemicals and solvents used were of A.R. quality.
METHODS OF ISOLATION OF COMPLEXES

Following two general methods have been used in the isolation of the complexes:

1. Shaking method
2. Refluxing method

1. Shaking method:

About 5 gms of starting material (e.g., Nickel (II) pyrrolidone copper (II) pyrrolidone, cobalt (II) pyrrolidone, copper (II) thiocyanate, cobalt (II) chromate, etc.) was suspended in 30 ml of acetone or in any other appropriate solvent (depending upon the compound used for complexation) in a 100 ml conical flask, and a calculated quantity of the amine was added dropwise. The contents were shaken for about 50 hrs. The completion of the reaction was marked by the uniformity of colour and size of the particles. The complexes were filtered in a crucible, washed repeatedly with acetone and amine mixture (100 : 0.5) and dried over phosphorus pentaoxide.

2. Refluxing method:

About 5 gms of the starting material (N-2 Pyrrolidone) was suspended in acetone or alcohol (30 ml). Calculated
quantity of ligand (e.g., 1:10 orthophenanthroline, 2,2'-Dipyridyl) was added to it. The contents refluxed on a water bath (60-70°C) for about 18-20 hrs. The solution develops characteristic colour due to formation of soluble complex. The insoluble ones were filtered, washed 10 times with acetone and dried (over P₂O₅). The soluble complexes were recovered by evaporating the solvent from the filtrate. The separated complexes are washed again with acetone, dried (over P₂O₅) and recrystallised.
EXPERIMENTAL DATA
SODIUM PYRROLIDONE

FORMULA: \( \text{Na} (\text{C}_4\text{H}_6\text{NO}) \)

COLOUR: White (Hygroscopic)

PREPARED BY\(^{181}\) —

ANALYSIS:

FOUND — Na, 21.29; C, 44.65; H, 05.66; N, 13.11; %

CALCULATED — Na, 21.21; C, 44.85; H, 05.60; N, 13.08; %

\(^{181}\) "Preparation of Lactames of Metals," C.A. 68, Feb. 5-12, 1968, page 29593 K.
Preparation of Co(II) Pyrrolidone:

The crude Sodium 2-pyrrolidone was washed several times with alcohol-benzene mixture (1:1), dried (over P_2O_5), powdered and then used for the preparation of Co(II) pyrrolidone. Co(II) pyrrolidone has been isolated by treatment of calculated quantity of Co(II) sulphate with Na-2-pyrrolidone in methyl alcohol. The contents of the flask were refluxed on a water bath (50-60°C) for four hours. The precipitated Co(II) pyrrolidone was washed several times with water and acetone to remove uncreated Co(II) sulphate and Na-pyrrolidone. The precipitate was dried filtered, dried (over P_2O_5) and analysed for Co(II), C, H and N by microanalytical methods. Co(II) pyrrolidonate has been used as the starting material for all the pyrrolidone complexes of Co(II). The analytical data have been reported separately.
1. COBALT (II) 2-PYRROLIDONE TETRAHYDRATE

FORMULA: \( \text{Co (C}_4\text{H}_6\text{NO})_2 \cdot 4\text{H}_2\text{O} \)

COLOUR: Brown

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Co, 19.20; C, 32.38; H, 04.05; N, 09.72; %

CALCULATED - Co, 19.18; C, 32.32; H, 04.04; N, 09.76; %

MOLECULAR WEIGHT:

FOUND - Insoluble.

CALCULATED - 294.94

MOLAR CONDUCTANCE: Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 4.71 \text{ B.M. Temp. } 300^\circ\text{A} \)

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig. 1):

300 sbr, 2900 sbr, 1670 sbr, 1580 sbr,
1450 ssp, 1450 ssp, 1355 ssp, 1285 msp,
1230 msp, 1105 sbr, 990 msp.
Fig. 1  I.R. : Cobalt (II) 2-Pyrrolidone tetrahydrate

Fig. 2  UV and Visible : Dipyrrrolidone ethylenediamine
Cobalt (II)
2. DIPYRROLIDONE ETHYLENE DIAmine COBALT (II)

FORMULA : \( \text{Co } (\text{CH}_2\text{NH}_2)_2 (\text{C}_4\text{H}_6\text{NO})_2 \)

COLOUR : Dark-brown

PREPARED BY : Shaking method

ANALYSIS :
FOUND - Co, 20.09; C, 41.87; H, 05.59; N, 19.51; %

CALCULATED - Co, 20.05; C, 41.80; H, 05.57;
N, 19.50; %

MOLECULAR WEIGHT :
FOUND (in nitrobenzene) - 286.94 ± 18

CALCULATED - 286.94

MOLAR CONDUCTANCE : Non-electrolyte

MAGNETIC SUSCEPTIBILITY :
\( \mu_{\text{eff}} = 5.12 \) B.M. Temp. 300°C

VISIBLE SPECTRAL BAND (Fig. 2) :
\[ \lambda_{\text{max}} (\text{m} \mu) \quad 430 \quad 600 \quad 835 \]
Frequency (cm\(^{-1}\)) 23,260 17,670 11,980
\( E_{430} \text{ m} \mu \) 14.64

INFRARED BANDS (Fig. 3) :
2960 sbr, 2550 mbr, 2000 wbr, 1590 sbr,
1460 ssp, 1370 ssp, 1280 sbr, 1250 sbr,
820 sbr.
Fig. 3  I.R. : Dipyrrolidone ethylenediamine Cobalt (II)

Fig. 4  UV and Visible : Dipyrrolidone propylenediamine Cobalt (II)
3. DIPYRROLIDONEPROPYLENEDIAMINE COBALT (II)

**FORMULA:** \[ \text{Co (NH}_2\text{.CH.CH}_3\text{CH}_2\text{NH}_2\text{)} (C}_4\text{H}_6\text{NO)}_2 \]

**COLOUR:** Brown

**PREPARED BY:** Shaking method

**ANALYSIS:**

**FOUND** - Co, 19.28; C, 43.72; H, 07.57; N, 09.34; %

**CALCULATED** - Co, 19.40; C, 43.85; H, 07.30; N, 09.30; %

**MOLECULAR WEIGHT:**

**FOUND (in nitrobenzene)** - 301.94 ± 8

**CALCULATED** - 301.94

**MOLAR CONDUCTANCE:** Non-electrolyte

**MAGNETIC SUSCEPTIBILITY:** \( \mu_{\text{eff}} = 5.14 \text{ B.M. Temp. 300}^\circ \text{A} \)

**VISIBLE SPECTRAL BANDS (Fig. 4):**

\[
\begin{array}{cccc}
\text{Frequency (cm}^{-1}) & 22,990 & 17,860 & 13,510 \\
\text{max (m u) } & 435 & 560 & 740 \\
\text{E}_{435 \text{ m u}} & 17.22 & & \\
\end{array}
\]

**INFRARED BANDS (Fig. 5):**

- 3620 wsp,
- 3375 sbr,
- 3250 sbr,
- 3150 msp,
- 2900 sbr,
- 2850 ssp,
- 1625 mbr,
- 1575 mbr,
- 1410 msp,
- 1385 msp,
- 1335 wsp,
- 1300 wsp,
- 1150 msp,
- 1050 msp,
- 106 w sp,
- 1037 asp,
- 1012 ssp,
- 925 wsp,
- 860 wsp.
Fig. 5  I.R. : Dipyrrolidone propylyenediamine Cobalt (II)

Fig. 6  UV and Visible : Dipyrrolidone bis 8-hydroxyquinoline Cobalt (II)
4. Dipyrrolidone Bis 8-Hydroxyquinoline Cobalt (II)

**FORMULA:**  \( \text{Co} \left( \text{C}_9\text{H}_7\text{ON} \right)_2 \left( \text{C}_4\text{H}_6\text{ON} \right)_2 \)

**COLOUR:**  Yellow

**PREPARED BY:**  Reflux method

**ANALYSIS:**

**FOUND** - Co, 12.21; C, 61.93; H, 04.80; N, 11.21; %

**CALCULATED** - Co, 11.21; C, 61.31; H, 04.25;
N, 10.84; %

**MOLECULAR WEIGHT:**

**FOUND (in nitrobenzene)** - 517.94 ± 13

**CALCULATED** - 517.94

**MOLAR CONDUCTANCE:**  Non-electrolyte

**MAGNETIC SUSCEPTIBILITY:**  \( \mu_{\text{eff}} = 4.92 \text{ B.M. Temp. 300.1}^\circ\text{A} \)

**VISIBLE SPECTRAL BANDS (Fig. 6):**

\[
\begin{align*}
\lambda_{\text{max}} (\text{m }\mu) & \quad 415 \quad 545 \\
\text{Frequency (cm}^{-1}) & \quad 24,100 \quad 18,350 \\
E_{415} \text{ m }\mu & \quad 13.5
\end{align*}
\]

**INFRARED BANDS (Fig. 7):**

\[
\begin{align*}
2900 \text{ sbr,} & \quad 2840 \text{ sbr,} \quad 2060 \text{ msp,} \quad 1625 \text{ msp,} \\
1585 \text{ msp,} & \quad 1550 \text{ msp,} \quad 1450 \text{ ssp,} \quad 1410 \text{ msp,} \\
1375 \text{ msp,} & \quad 1300 \text{ ssp,} \quad 1275 \text{ ssp,} \quad 1140 \text{ msp,} \\
1090 \text{ msp,} & \quad 1020 \text{ msp,} \quad 875 \text{ msp,} \quad 825 \text{ msp,} \\
750 \text{ msp,} & \quad 720 \text{ msp.}
\end{align*}
\]
Fig. 7  I.R. : Dipyrrolidone bis 8-hydroxyquinoline
        Cobalt (II)

Fig. 8  UV and Visible : Dipyrrolidone bis
        2,2'dipyridyl Cobalt (II)
5. DIPYRROLIDONE BIS 2' 2' DIPYRIDYL COBALT (II)

FORMULA : Co ( C\(_{10}\)H\(_8\)N\(_2\)\(_2\) ( C\(_4\)H\(_6\)NO \)\(_2\)  

COLOUR : Black

PREPARED BY : Reflux method

ANALYSIS :

FOUND - Co, 10.52; C, 63.20; H, 05.18; N, 15.42; %

CALculated - Co, 10.90; C, 62.10; H, 05.17;

n, 15.52; %

MOLECULAR WEIGHT :

FOUND (in nitrobenzene) - 539.94 ± 17

CALCULATED - 539.94

MOLAR CONDUCTANCE :

Non-electrolyte

MAGNETIC SUSCEPTIBILITY :

\( \mu_{\text{eff}} = 4.91 \text{ B.M.} \)

Temp. 300.1°C

VISIBLE SPECTRAL BANDS (Fig. 8) :

\( \lambda_{\text{max (m } \mu} \) 522 700 915

Frequency (cm\(^{-1}\)) 19,160 14,290 10,930

\( E_{522 \text{ m }\mu} \) 17.23

INFRARED BANDS (Fig. 9) :

3510 ssp, 1815 mbr, 1790 msp, 1700 ssp,  
1610 msp, 1595 msp, 1570 msp, 1510 msp,  
1490 msp, 1400 msp, 1300 msp, 1090 wsh,  
890 wsp, 780 wsp.
Fig. 9  I.R. : Dipyrrolidone bis 2,2'dipyridyl Cobalt (II)

Fig. 10  UV and Visible : Dipyrrolidone bis 1:10 Orthophenanthroline Cobalt (II)
6. DIPYRROLIDONE BIS 1:10 ORTHOPHENANTHROLINE COBALT (II)

FORMULA: \( \text{Co} (\text{C}_{12}\text{H}_{8}\text{N}_2\cdot\text{H}_2\text{O})_2 (\text{C}_4\text{H}_6\text{NO})_2 \)

COLOUR: Black

PREPARED BY: Reflux method

ANALYSIS:

FIND - Co, 09.34; C, 61.72; H, 5.28; N, 13.84; %

CALCULATED - Co, 09.48; C, 61.63; H, 5.13; N, 13.90; %

MOLECULAR WEIGHT:

FOUND (in nitrobenzene) - 623 ± 0

CALCULATED - 623

MOLAR CONDUCTANCE:
Non-electrolyte

MAGNETIC SUSCEPTIBILITY:
\( \mu_{\text{eff}} = 4.92 \text{ B.M.} \)
Temp. 300.1°C

VISIBLE SPECTRAL BANDS (Fig. 10):

\( \lambda_{\text{max}} (\text{m} \mu) \)

<table>
<thead>
<tr>
<th>Frequency (cm(^{-1}))</th>
<th>450</th>
<th>620</th>
<th>750</th>
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<tr>
<td>( E_{450 \text{ m} \mu} )</td>
<td>17.62</td>
<td></td>
<td></td>
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</tbody>
</table>

INFRARED BANDS (Fig. 11):

3110 msp, 1600 wsp, 1510 msp, 1460 msp,
1200 mbr, 1120 mbr, 1030 mbr, 970 ssp,
880 msp, 810 msp, 760 msp.
Fig. 11  I.R.: Dipyrrolidone bis 1:10 Orthophenanthroline Cobalt (II)

Fig. 12  I.R.: Dipyrrolidone dimethylglyoxime Cobalt (II)
7. DIPYRROLIDONE DIMETHYLGLYOXIME COBALT (II)

FORMULA: \( \text{Co} (\text{CH}_3\text{CON})_2 (\text{C}_4\text{H}_6\text{ON})_2 \)

COLOUR: Tea colour

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Co, 17.28; C, 42.30; H, 05.29; N, 16.43; %

CALCULATED - Co, 17.20; C, 41.98; H, 05.24;
N, 16.32; %

MOLECULAR WEIGHT:

FOUND

CALCULATED - 340.94

MOLAR CONDUCTANCE:

Non-electrolyte

MAGNETIC SUSCEPTIBILITY:

\( \mu_{\text{eff}} = 4.97 \text{ B.M.} \)

Temp. 300.1\(^\circ\)A

VISIBLE SPECTRAL BANDS:

Insoluble

INFRARED BANDS (Fig. 12):

2970 ss, 2610 ss, 1580 m, 1495 m,
1460 m, 1370 m, 1050 m, 910 m,
820 w, 760 w.
8. DITHIOCYNATOETHYLENEDIAMINE COBALT (II)

FORMULA: \[ \text{Co} \left( CH_{2}NH_{2}NH_{2}CH_{2} \right) \left( \text{SON} \right)_{2} \]

COLOUR: Light green

PREPARED BY: Shaking method

ANALYSIS:

FOUND - Co, 20.22; C, 24.58; H, 05.38; (SCN), 38.97; %

CALculated - Co, 20.00; C, 24.40; H, 05.42; (SCN), 39.32; %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 234.94

MOLAR CONDUCTANCE

Insoluble

MAGNETIC SUSCEPTIBILITY \[ \mu_{\text{eff}} = 5.2 \text{ B.M.} \]

Temp. 300°A

VISIBLE SPECTRAL BANDS:

Insoluble

INFRARED BANDS (Fig. 13):

3200 wsp, 3100 wbr, 2900 ssp, 2840 ssp,
2120 msp, 2050 msp, 1575 wbr, 1450 ssp,
1375 msp, 1100 svbr, 980 wsp.
Fig. 13  I.R. : Dithiocynatoethylenediamine Cobalt (II)

Fig. 14  I.R. : Dithiocynatopropylenediamine Cobalt (II)
9. DITHIOCYNATOPROPYLENEDIAMINE COBALT (II)

FORMULA: \[ \text{Co} (\text{NH}_2\text{CH} \cdot \text{CH}_2\text{CH}_2\text{NH}_2) (\text{SON})_2 \]

COLOUR: Green

PREPARED BY: Shaking method

ANALYSIS:

FOUND - Co, 23.43; C, 22.98; H, 04.72; (SCN), 49.88; %

CALCULATED - Co, 23.71; C, 24.09; H, 04.34;
(SCN), 49.79; %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 248.94

MOLAR CONDUCTANCE:

Insoluble

MAGNETIC SUSCEPTIBILITY:

\( \mu_{\text{eff}} = 5.02 \text{ B.M.} \)

Temp. 300\(^\circ\text{C}\)

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig. 14):

3560 ssp, 3100 sbr, 2960-2940 msp sh.,
2860 msp, 2350 mbr, 1600 mbr, 1470-1450 ssp sh,
1400 msp, sh., 1370 ssp, 1340 ssp, 1300 ssp,
1130 ssp, 1065 ssp, 1030 ssp, 980 ssp,
960 msp, 850-860 msp, sh., 700 ssp.
10. BIS 8-HYDROXYQUINOLINE COBALT (II) PERCHLORATE

FORMULA: \( \text{Co}\left(\text{C}_9\text{H}_7\text{ON}\right)_2\left(\text{ClO}_4\right)_2 \)

COLOUR: Deep yellow

PREPARED BY: Reflux method

ANALYSIS:

FOUND: Co, 11.49; C, 40.08; H, 02.79; (\text{ClO}_4), 36.32; N, 05.04; %

CALCULATED: Co, 10.74; C, 39.34; H, 02.55;
\( (\text{ClO}_4), 36.24; \) N, 05.10; %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 549 ± 8

MOLAR CONDUCTANCE: Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 4.81 \text{ B.M.} \)
Temp. 300.2°A

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig. 15):

2900 ssp, 2850 ssp, 1625 wbr, 1600 wsp,
1575 msp, 1495 msp, 1465 ssp, 1420 msp,
1375 ssp, 1325 ssp, 1275 msp, 1237 msp,
1100 msp, 1065 wsp, 1025 wsp, 905 wsp,
850 sv sp., 820 ssp, 800 w sp., 780 m sp.,
740 w sp.
Fig. 15  I.R. : Bis 8-hydroxyquinoline Cobalt (II) perchlorate

Fig. 16  I.R. : Cobalt (II) chromatedihydrate
11. **Cobalt (II) Chromatedihydrate**

**Formula:** $\text{Co} \left( \text{CrO}_4 \right) \cdot 2\text{H}_2\text{O}

**Colour:** Deep reddish brown

**Prepared by:** Reflux method

**Analysis:**

**Found -** Co, 26.65; (CrO$_4$), 52.73; %

**Calculated -** Co, 26.69; (CrO$_4$), 52.49; %

**Molecular Weight:**

**Found** - Insoluble

**Calculated** - 210.95

**Molar Conductance:**

Insoluble

**Magnetic Susceptibility:** $\mu_{\text{eff}} = 4.72$ B.M.

Temp. 300.2°C

**Visible Spectral Band:** Insoluble

**Infrared Bands:** (Fig. 16)

3430 mvbr, 1600 wvbr, 950 ssp, 890 ssp, 860 ssp,

790 ssp, 665 sp, 650 ssp.
12. BIS DIMETHYLGLYOXIME CO(II) CHROMATE

FORMULA: \( \text{Co} \left( \text{C}_4\text{H}_6\text{N}_2\text{O}_2 \right)_2 \text{CrO}_4 \)

COLOUR: Tea colour

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Co, 14.42; C, 23.78; H, 02.36; N, 14.20; (CrO₄), 28.99; %

CALCULATED - Co, 14.21; C, 23.94; H, 02.99; N, 13.96; (CrO₄), 28.92; %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 402.95

MOLAR CONDUCTANCE:

Insoluble

MAGNETIC SUSCEPTIBILITY:

\( \mu_{\text{eff}} = 4.94 \text{ B.M.} \)

Temp. 300⁰A

VISIBLE SPECTRAL BANDS:

Insoluble

INFRARED BANDS (Fig. 17):

3400 sbr, v; 1630 mbr, 1580 ssp, 1440 mbr,
1380 msp, 1210 ssp, 1080 ssp, 975 msp, 985 ssp,
880 sbr, 850 ssp, 780 ssp, 750 ssp, 660 ssp,
640 ssp.
Fig. 17 I.R. : Bis dimethylglyoxime Cobalt (II) chromate

Fig. 18 I.R. : Thiocarbazide Cobalt (II) perchlorate
13-METHIOCARBAZIDE CO(II) PERCHLORATE

FORMULA: \( \text{Co} (\text{NH}_2 \cdot \text{CS} \cdot \text{NH} \cdot \text{NH}_2) \text{ClO}_4 \)

COLOUR: Yellow

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Co, 23.72; C, 4.81; H, 1.98; N, 16.96; (ClO\(_4\)), 37.65; %

CALCULATED - Co, 23.85; C, 4.87; H, 1.88; N, 16.72; (ClO\(_4\)), 37.89; %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 249.44

MOLAR CONDUCTANCE: Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 4.87 \) B.M.

Temp. 300°A

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig. 18):

3200 mbr v, 1580 msp, 1535 ssp, 1430 mbr,
1365 msp, 1320 msp, 1210 ssp, 1080 sbr,
1060 spr, 980 msp, 960 ssp, 900 msp,
860 mbr, 815 mbr, 720 ssp, 700 mbr,
665 msp, 650 msp.
NICKEL (II) COMPLEXES
14. NICKEL (II) 2-PYRROLIDONETETRAHYDRATE

FORMULA: \( \text{Ni} \left( \text{C}_4\text{H}_6\text{NO} \right)_2 \cdot 4 \text{H}_2\text{O} \)

COLOUR: Green

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Ni, 24.11; C, 38.93; H, 4.98; N, 11.61 %

CALculated - Ni, 24.07; C, 38.89; H, 4.97; N, 11.62 %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 241.69

MOLAR CONDUCTANCE:

Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 2.89 \, \text{B.M.} \) Temp. \( 300.1^\circ\text{A} \)

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig.19):

3320 sbr v, 1630 sbr v, 1600 sbr v, 14360 msp sh.,
1400 msp sh., 1280 msp, 1060 wsp, 980 wsp,
840 msp, 600 mbr, 550 msp.
Fig. 19  
I.R. : Nickel (II) 2-Pyrrolidone tetrahydrate

Fig. 20  
UV and Visible : Dipyrrolidone ethylenediamine
Nickel (II)
15. DIPYRROLIDONE ETHYLENEDIAMINE NICKEL (II)

FORMULA: \( \text{Ni} \ (\text{CH}_2\text{NH}_2)_2 \ (\text{C}_4\text{H}_6\text{NO}_2) \)

COLOUR: Light blue

PREPARED BY: Shaking method

ANALYSIS:

FOUND - Ni, 20.51; C, 41.08; H, 06.22; N, 19.98 %

CALCULATED - Ni, 20.27; C, 41.95; H, 06.99; N, 19.58 %

MOLECULAR WEIGHT:

FOUND (in nitrobenzene) - 286.69 ± 11.4

CALCULATED - 286.69

MOLAR CONDUCTANCE: Non-electrolyte

MAGNETIC SUSCEPTIBILITY: \( \kappa_{\text{eff}} \) 0.73 B.M. Temp. 300.1°A

VISIBLE SPECTRAL BANDS (Fig. 20):

\( \lambda_{\text{max}} \) (m \( \mu \)) 360 515 625

Frequency (cm\(^{-1}\)) 27,780 19,420 16,000

\( E_{360} \text{ m u} \) 18.5

\( \nu_2/\nu_1 \) 1.73

INFRARED BANDS (Fig. 21):

3440 mbr, 3260 sbr, 3140 sbr, 2100 wbr, 1635 wsp,

1590-1580 msp sh., 1320 msp, 1270 msp, 1145 msp,

1080 wsp, 1065 msp, 1005 ssp, 975 msp, 715 msp,

650msp.
Fig. 21  I.R.: Dipyrrolidone ethylenediamine Nickel (II)

Fig. 22  UV and Visible: Dipyrrolidone propylenediamine Nickel (II)
16. DIPYRROLIDONEPROPYLENEDIAMINE NICKEL (II)

FORMULA: \( \text{Ni}(\text{NH}_2\cdot\text{CH} \cdot \text{CH}_3 \cdot \text{CH}_2 \cdot \text{NH}_2)(\text{C}_4\text{H}_6\text{NO})_2 \)

COLOUR: Light green

PREPARED BY: Shaking method

ANALYSIS:

FOUND - Ni, 19.42; C, 44.6; H, 07.39; N, 18.74 %

CALCULATED - Ni, 18.74; C, 44.00; H, 07.33;
N, 18.66 %

MOLECULAR WEIGHT:

FOUND (in nitrobenzene) - 300 + 19.9

CALCULATED - 300

MOLAR CONDUCTANCE: Non-electrolyte

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 0.52 \text{ B.M. Temp. 300.1}^\circ\text{A} \)

VISIBLE SPECTRAL BANDS (Fig. 22):

\[ \lambda_{\text{max}}(\text{m} \mu) \]

<table>
<thead>
<tr>
<th>365</th>
<th>475</th>
<th>653</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frequency (cm(^{-1}))</td>
<td>27,400</td>
<td>21,050</td>
</tr>
<tr>
<td>( E_{365} \text{ m u} )</td>
<td>16.2</td>
<td></td>
</tr>
<tr>
<td>( v_2/v_1 )</td>
<td>1.79</td>
<td></td>
</tr>
</tbody>
</table>

INFRARED BANDS (Fig. 5):

| 3620 wsp. | 3375 abr. | 3150 mbr. | 1625 msp. | 1575 wbr. |
| 1385 msp. | 1335 msp. | 1300 wsp. | 1150 msp. | 1065 msp. |
| 1037 ssp. | 1012 ssp. | 925 wsp. | 860 v sp. |
17. DIPYRROLIDONE BIS 8-HYDROXYQUINOLINE NICKEL (II)

FORMULA: \( \text{Ni} \left( \text{C}_9\text{H}_7\text{ON} \right)_2 \left( \text{C}_4\text{H}_6\text{NO} \right)_2 \)

COLOUR: Lemon yellow

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Ni, 11.31; C, 60.42; H, 05.41; N, 10.92 %
CALCULATED - Ni, 11.24; C, 60.46; H, 05.39; N, 10.85 %

MOLECULAR WEIGHT:

FOUND (in nitrobenzene) - 516.94 \pm 86
CALCULATED - 516.94

MOLAR CONDUCTANCE: Non-electrolyte

MAGNETIC MEASUREMENT: \( \mu_{\text{eff}} = 0.57 \) B.M. Temp. 300\(^\circ\)A

VISIBLE SPECTRAL BANDS (Fig. 23):

\[ \lambda_{\text{max}} \text{ (m } \mu) \quad 400 \quad 505 \quad 785 \]

Frequency (cm\(^{-1}\)) 25,000 19,800 12,740
\( \varepsilon_{540} \text{ m} \mu \) 48.4
\( \nu_3/\nu_1 \) 1.96

INFRARED BANDS (Fig. 24):

3300 mbr v, 3030 msp, 1590 msp, 1580 ssp, 1565 ssp,
1490 ssp, 1460 ssp, 1380 ssp, 1360 ssp, 1320 ssp,
1280 ssp, 1220 msp, 1200 msp, 1170 msp, 1130 msp,
1100 ssp, 890 msp, 8020 ssp, 790 ssp, 780 ssp,
760 msp, 740 ssp, 725 ssp, 650 ssp.
Fig. 23  UV and Visible: Dipyrrolidone bis 8-hydroxyquinoline Nickel (II)

Fig. 24  I.R.: Dipyrrolidone bis 8-hydroxyquinoline Nickel (II)
18. DIPYRROLIDONE BIS 2 2' DIPYRIDYL NICKEL (II)

FORMULA: \( \text{Ni}_x(\text{C}_{10}\text{H}_8\text{N}_2)_2(\text{C}_4\text{H}_6\text{NO})_2 \)

COLOUR: Pink

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Ni, 10.29; C, 62.95; H, 05.29; N, 15.69 %
CALCULATED - Ni, 10.09; C, 62.45; H, 05.28;
N, 15.61 %

MOLECULAR WEIGHT:

FOUND - 538 ± 11.5
CALCULATED - 538

MOLAR CONDUCTANCE: Non-electrolyte

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 2.97 \) B.M. Temp. 300° A

VISIBLE SPECTRAL BANDS (Fig. 25):

<table>
<thead>
<tr>
<th>( \lambda ) (m ( \mu ))</th>
<th>460</th>
<th>595</th>
<th>805</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frequency (cm(^{-1}))</td>
<td>21,740</td>
<td>16,810</td>
<td>12,426</td>
</tr>
<tr>
<td>( E_{460} ) m ( \mu )</td>
<td>18.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \nu_3/\nu_1 )</td>
<td>1.72</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

INFRARED BANDS (Fig. 9):

3500 ssp, 1800 msp, 1810 msp, 1690 msp,
1595 msp, 1580 msp, 1570 msp, 1505 msp,
1395 msp, 1350 msp, 1240 msp, 1090 msp,
1080 msp, 880 ssp, 780 msp.
19. DIPYRROLIDONE BIS 1,10 ORTHOPHENANTHROLINE NICKEL (II)

FORMULA: \( \text{Ni} \left( \text{C}_{12}\text{H}_8\text{N}_2\cdot\text{H}_2\text{O} \right)_2 \left( \text{C}_{4}\text{H}_6\text{NO} \right) \)

COLOUR: Pink

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Ni, 9.41; C, 62.04; H, 05.29; N, 13.37 %

CALCULATED - Ni, 9.32; C, 61.17; H, 05.14;

N, 13.50 %

MOLECULAR WEIGHT:

FOUND - 622.3

CALCULATED - 622.3 ± 8.8

MOLAR CONDUCTANCE: Non-electrolyte

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 3.10 \) B.M. Temp. 300° A

VISIBLE SPECTRAL BANDS (Fig. 26):

\[ \lambda_{\text{max}} (\text{m} \mu) \quad 410 \quad 570 \quad 775 \]

Frequency (cm\(^{-1}\)) 24,390 17,540 12,900

\[ \text{E}_{410} \text{ m} \mu \quad 19.2 \]

\[ \frac{\nu_2}{\nu_1} \quad 1.88 \]

INFRARED BANDS (Fig. 11):

3110 msp, 1595 mbr, 1505 msp, 1490 msp,
1210 mbr, 1110 mbr, 1050 mbr, 960 ssp,
870 msp, 805 msp, 760 mbr.
Fig. 25  UV and Visible : Dipyrrolidone bis 2 2' dipyridyl Nickel (II)

Fig. 26  UV and Visible : Dipyrrolidone bis 1:10 Orthophenanthroline Nickel (II)
20. DIPYRROLIDONEDIMETHYLGLYOXIME NICKEL (II)

FORMULA: \( \text{Ni} (\text{CH}_3\text{CNO})_2 (\text{C}_4\text{H}_6\text{NO})_2 \)

COLOUR: reddish brown

PREPARED BY: reflux method

ANALYSIS:

FOUND - \( \text{Ni}, 16.40; \text{C}, 42.31; \text{H}, 05.29; \text{N}, 16.40 \% \)
CALCULATED - \( \text{Ni}, 16.94; \text{C}, 42.10; \text{H}, 05.26; \text{N}, 16.37 \% \)

MOLECULAR WEIGHT:

FOUND - Insoluble
CALCULATED - 337.69

MOLAR CONDUCTANCE: Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 0.64 \text{ B.M. Temp. } 300^\circ \text{A} \)

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig. 12):

2895 ssp, 2830 ssp, 1575 wsp, 1525 mbr,
1450 msp, 1375 msp, 1075 mbr, v, 965 msp,
900 mbr, v, 700 mbr.
COPPER (II) COMPLEXES
21. COPPER 2-PYRROLIDONE DIHYDRATE

FORMULA: \( \text{Cu} \left( \text{C}_4\text{H}_6\text{NO} \right)_2 \cdot \text{2H}_2\text{O} \)

COLOUR: Blue

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Cu, 25.20; C, 38.62; H, 6.90; N, 11.23; %

CALCULATED - Cu, 25.30; C, 38.65; H, 6.91; N, 11.25%

MOLECULAR WEIGHT:

FOUND - Insoluble
CALCULATED - 231.57

MOLAR CONDUCTANCE: Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 1.82 \) B.M.
Temp. 300\(^\circ\)A

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig. 27):

\( 3550 \text{ msp}, \ 1650 \text{ mbr}, \ 1470 \text{ mbr}, \ 1410 \text{ msp} \)
\( 1375 \text{ mbr}, \ 1110 \text{ msp}, \ 835 \text{ mbr}, \ 890 \text{ mbr} \)
\( 670 \text{ msp}, \ 650 \text{ msp} \)
Fig. 27 I.R. : Copper (II) 2-pyrrolidone dihydrate

Fig. 28 I.R. : Dipyrrrolidone propylenediamine Copper (II)
22. DIPYRROLIDONE 1,10 ORTHOPHENANTHROLINE COPPER (II)

FORMULA: \( \text{Cu (C}_{12}\text{H}_8\text{N}_2\cdot\text{H}_2\text{O}) (\text{C}_{4}\text{H}_6\text{NO})_2 \)

COLOUR: Green

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Cu, 14.62; C, 55.63; H, 05.22; N, 12.92 %

CALCULATED - Cu, 14.82; C, 55.93; H, 05.12; N, 13.05 %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 439.57

MOLAR CONDUCTANCE: Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 1.76 \) B.M. Temp. 300°C

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig. 11):

3110 ssp, 1605 mbr, 1510 msp, 1490 msp,

1210 mbr, 1170 msp, 980 ssp, 880 msp,

820 ssp, 710 mbr.
23. DIPYRROLIDONEDIMETHYLGLYOXIME COPPER (II)

FORMULA: \( \text{Cu} \left( \text{CH}_3\text{CNO} \right)_2 \left( \text{C}_4\text{H}_6\text{NO} \right)_2 \)

COLOUR: Dark brown

PREPARED BY: Reflux method

ANALYSIS:

FOUND - Co, 18.73; C, 41.22; H, 05.17; N, 16.31 %

CALculated - Co, 18.15; C, 41.15; H, 05.18;

N, 16.24 %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 347.57

MOLAR CONDUCTANCE:

Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 1.81 \text{ B.M. Temp.} 300.1^\circ\text{A} \)

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig. 12):

2900 ssp, 2830 ssp, 1575 msp, 1525 mbr,

1450 mbr, 1370 msp, 1075 mbr v, 975 msp,

875 mbr v, 720 mbr.
24. DIPYRROLIDONE ETHYLENEDIAMINE COPPER (II)

FORMULA: \[ \text{Cu} \left( \text{CH}_2\text{NH}_2 \right)_2 \left( \text{C}_4\text{H}_6\text{NO} \right)_2 \]

COLOUR: Blue

PREPARED BY: Shaking method

ANALYSIS:

FOUND - Cu, 21.63; C, 41.41; H, 06.73; N, 19.22 %

CALCULATED - Cu, 21.65; C, 41.23; H, 06.86; N, 19.24 %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 291.57

MOLAR CONDUCTANCE:

Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 1.91 \) B.M. Temp. 300.1°C

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS (Fig. 21):

3440 mbr, 3260 sbr v, 3140 sbr, 2100 wbr,
1635 msp, 1590-1580 msp sh., 1400 ssp,
1375 ssp, 1320 msp, 1270 msp, 1145 msp,
1100 msp, 1080 msp, 1065 msp, 1005 ssp,
975 msp, 715 msp.
25. DIPYRROLOIDONE PROPYLENEDIAMINE COPPER (II)

FORMULA: \( \text{Cu} \left( \text{NH}_2 \cdot \text{CH} \cdot \text{CH}_3 \cdot \text{CH}_2 \text{NH}_2 \right) \left( \text{C}_4\text{H}_6\text{NO} \right)_2 \)

COLOUR: Dark blue

PREPARED BY: Shaking method

ANALYSIS:

FOUND - Cu, 20.58; C, 43.29; H, 07.31; N, 18.44 %

CALCULATED - Cu, 20.65; C, 43.27; H, 07.21; N, 18.36 %

MOLECULAR WEIGHT:

FOUND - Insoluble

CALCULATED - 305.57

MOLAR CONDUCTANCE: Insoluble

MAGNETIC SUSCEPTIBILITY: \( \mu_{\text{eff}} = 1.83 \text{ B.M. Temp. 300.1}^\circ\text{A} \)

VISIBLE SPECTRAL BANDS: Insoluble

INFRARED BANDS: Fig 28

3550 ssp, 3250 s, br, v, 3090 mbr, 2920 msp,
2880 mbr, 2850 msp, 1550 msp, 1420 msp,
1410 msp, 1390 msp, 1350 msp, 1310-1290 msp sh.,
1060 ssp, 1020 ssp, 920 msp, 880 msp,
800 mbr, 690 mbr, 610 msp.