CHAPTER IV

Experimental
(A) EXPERIMENTAL DETAILS

[a] Sovents and Chemicals

Nalidixic acid, Norfloxacin, Ciprofloxacin. [S.A. Pharmacy and Medi
Deals, Sagar] and Cloxacillin Sodium [Lapaz pharmaceutical, Sagar] were obtained
in pure form their concerns [Mentioned in brackets] and were used as such.

Metallic Salts and other chemical used were of Analytical R / E. Merck/
BDH grade.

[b] Analysis

Estimation of Carbon, Hydrogen, Nitrogen and Sulpher were carried
out by Carlo-erba analyser at Regional Sophisticated Instrumentation Centre,
Central Drug Research Institute Lucknow (U.P.) and I.I.T. Kanpur. Mn, Fe, Ni, Cu
and Zn of the complexes were estimated after decomposing the complexes with
aquaregia by EDTA titration method using Solochrome black - T, Variamine blue,
Murexide, Fast sulphone black, Eriochrome black - T as indicators¹, while nickel
(II) is also estimated as bis glyoximato nickel (II)². Chloride ion were estimated
by the standard methods.³⁴

[c] Molar Conductance

The molar conductance were determined in dimethyl formamide and
methanol using 10⁻³ M solutions of the complexes using CM-82T conductivity
bridge.

[d] Calculation of Magnetic Moment

Magnetic susceptibility were measured on Gouy's balance at room
temperature using standard CuSO₄·5H₂O as the calibrant. The diamagnetic
correction were applied using pascal's constants.⁵⁶
[e] Spectral Measurements

The electronic spectra of solution of the complexes in Dimethyl formamide and methanol were recorded on Schimadzu UV-210 A spectrophotometer in the range 350 to 1000 nm.

[f] Infrared Measurements

The infrared spectra of ligand and complexes were recorded on Shimadzu IR-470. Infrared spectrophotometer in KBr medium. Between the range 400-4000 cm⁻¹.

[g] Biological Activities

Antimicrobial activity (Antibacterial and Antifungal) were performed at Department of Botany, Dr. H.S. Gour University, Sagar (M.P.). Using paper disc method on gram + ve and gram - ve bacteria, like Escherichia coli, Bacillus subtilis, Salmonella typhosa, Shigella flexneri, Bacillus pyocyaneus and Staphylococcus aureus bacteria and Aspergillus niger, Aspergillus flavus, Candida albicans, Crysosporium panicale and Tricoderma viride fungus.

(B) METHOD OF ISOLATION OF COMPLEXES

Complexes were isolated using refluxing method. Two types of solvents were used to isolated the complexes by this method.

Water Solvent

Complexes of Ciprofloxacin and Cloxacillin Sodium were isolated from water. Concentrated aqueous solution of the ligand [L] in slight excess over stoichiometric M : L :: 1 : 1 or M : L :: 1 : 2 ratio was added slowly with constant stirring to an aqueous solution of metal salt. pH of the the solution was so adjusted
as to get the indication of complex formation by colour change or by precipitation. Elico digital pH metar [Model L1-120] was used to adjust the pH of reactants. All the complexes prepared were obtained in pH range 4 to 6.5. After adjusting the pH the content was refluxed over steam bath for about an hour. In some cases it was observed that granular and well defined complex was obtained after keeping the refluxed mixture at 5-15°C over night. Precipitate so obtained was filtered and washed many times with not distilled water. It was dried in an oven at about 110°C and stored in a desiccat over anhydrous CaCl₂.

**Organic Solvents**

Norfloxacin and Nalidixic acid complexes were isolated form glacial acetic acid and chloroform. To a hot solution of the ligand in the same solvent in ratio 1 : 1 :: M : L or 1 : 2 :: M : L were added. The resulting mixture was refluxed on a water bath for 10-12 hrs. The precipitated complex was filtered, washed, dried in a oven at about 110°C and stored in a desiccat over anhydrous CaCl₂. Stoichiometric composition and adjustment of pH for various complexes are listed below:

**Nalidixic acid :** [1-ethyl - 7 - methyl - 4 - oxo - 1, 4 - dihydro - 1, 8 - napthrydine - 3 - carboxylic acid] complexes.

(I) \([\text{Mn} \left( C_{12}H_{11}N_2O_3 \right) \text{L}_2 (H_2O)_2] \)

(II) \([\text{Fe} \left( C_{12}H_{11}N_2O_3 \right) \text{L}_2 (\text{Cl}) (H_2O)] \)

(III) \([\text{Ni} \left( C_{12}H_{11}N_2O_3 \right) \text{L}_2 (H_2O)_2] \)

(IV) \([\text{Cu} \left( C_{12}H_{11}N_2O_3 \right) (\text{OH}) (H_2O)_3] \)

(V) \([\text{Zn} \left( C_{12}H_{11}N_2O_3 \right) (\text{OH}) (H_2O)_3] \)
The complexes were prepared by refluxing the mixture of the concentrated (Non-equeous) solution of the drug and metal salt in the ratio 1 : 1 or 1 : 2 for 1 hour at pH 4-5.5 and then keeping the reactants overnight.

**Norfloracin**: [3-quinolinecarboxylic acid, 1-ethyl-6-fluoro-1, 4-dihydro-4-oxo-7-(1-piperazinyl) -] complexes.

(I) \[\text{Mn} \ (\text{C}_{16} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3}) \ (\text{Cl}) \ (\text{H}_{2} \text{O})_{3}\]

(II) \[\text{Fe} \ (\text{C}_{16} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3}) \ (\text{Cl})_{2} \ (\text{H}_{2} \text{O})_{2}\]

(III) \[\text{Ni} \ (\text{C}_{16} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3}) \ (\text{CH}_{3} \text{COO}) \ (\text{H}_{2} \text{O})_{3}\]

(IV) \[\text{Cu} \ (\text{C}_{16} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3})_{2} (\text{H}_{2} \text{O})_{2}\]

(V) \[\text{Zn} \ (\text{C}_{16} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3}) \ (\text{CH}_{3} \text{COO}) (\text{H}_{2} \text{O})_{3}\]

The complexes were prepared by refluxing the mixture of aqueous solution of metal salt and drug in the stoichiometric ratio \(\text{M} : \text{L} \rightarrow 1 : 1\) and \(\text{M} : \text{L} \rightarrow 1 : 2\) at pH 5-6.5 for 1 hour.

**Ciprofloracin**: [3-quinolinecarboxylic acid, 1 - cyclopropyl - 6 - fluoro -, 4 dihydro - 4 - oxo - 7 - (1 - piperazinyl) -] complexes.

(I) \[\text{Mn} \ (\text{C}_{17} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3}) \ (\text{Cl}) \ (\text{H}_{2} \text{O})_{3}\]

(II) \[\text{Fe} \ (\text{C}_{17} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3})_{2} (\text{OH}) (\text{H}_{2} \text{O})\]

(III) \[\text{Ni} \ (\text{C}_{17} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3}) \ (\text{CH}_{3} \text{COO}) (\text{H}_{2} \text{O})_{3}\]

(IV) \[\text{Cu} \ (\text{C}_{17} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3})_{2} (\text{H}_{2} \text{O})_{2}\]

(V) \[\text{Zn} \ (\text{C}_{17} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3}) \ (\text{CH}_{3} \text{COO}) (\text{H}_{2} \text{O})_{3}\]

Metal to drug ratio was \(\text{M} : \text{L} \rightarrow 1 : 1\) and \(\text{M} : \text{L} \rightarrow 1 : 2\) pH adjusted to
5-6 with the help of dilute NH₃ solution. After refluxing the solution for 1 hour
the content was kept 5-15°C over night.

**Cloxacillin Sodium**: [2S - (2α, 5α, 6β)] - 4 - Thio - 1- azabicyclo [3, 2, 0]
heptane - 2 - carboxylic acid, 6- [[ {3 - (2 - chlorophenyl) - 5 methyl - 4 - isoxazolyl]
carbonyl] amino] - 3, 3 - dimethyl-7 - oxo -, monosodium salt, monohydrated.

(I) \[\text{Mn} (C_{19}H_{19}Cl N_3O_6S) (Cl) (H_2O)_3] \]

(II) \[\text{Fe} (C_{19}H_{19}Cl N_3O_6S) (SO_4) (H_2O)_2] \]

(III) \[\text{Ni} (C_{19}H_{19}Cl N_3O_6S) (OH) (H_2O)_3] \]

(IV) \[\text{Cu} (C_{19}H_{19}Cl N_3O_6S) (OH) (H_2O)_3] \]

(V) \[\text{Zn} (C_{19}H_{19}Cl N_3O_6S) (OH) (H_2O)_3] \]

The complexes were prepared by refluxing the mixture of
non - aqueous solution of metal salt and drug in the stoichiometric ratio M : L : :
1 : 1 at pH 4.5 - 5.5 for 1 hour. After refluxing the mixture was cooled at 5-15°C
over night.
1 - Nalidixic acid - Mn (II) complex :-

Molecular Formula - \([\text{Mn} (\text{C}_{12}\text{H}_{11}\text{N}_{2}\text{O}_{3})_2(\text{H}_2\text{O})_2]\)

Calculated Molecular Weight - 553.41

Colour - Light Brown

Decomposition Temperature - 308°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Mn = 10.95, C = 53.38, H = 4.49, N = 10.32

Calculated (%) Mn = 9.92, C = 52.04, H = 4.69, N = 10.11

Molar Conductance - Insoluble

Magnetic Measurement - Temperature = 300°K

- Magnetic Susceptibility = 26.0024\times10^{-6}

- \(\mu_{\text{eff}} = 5.88\ \text{BM}\)

Electronic Spectral Data - Insoluble

Infrared Bands - [Fig : 3.A]

\[
3685\text{w} \quad 3650\text{w} \quad 1620\text{sp} \quad 1540\text{mw} \quad 1440\text{mw} \quad 1330\text{mw} \\
1225\text{mw} \quad 1220\text{br} \quad 880\text{br} \quad 845\text{sp} \quad 771\text{w} \quad 702\text{mw} \\
540\text{mw} \quad 450\text{mw}
\]
FIG:3-A. INFRARED SPECTRUM OF NALIDIXIC ACID Mn(II) COMPLEX.
2 - Nalidixic acid - Fe (III) complex :-

Molecular Formula - [Fe(C₁₂H₁₁N₂O₃)₂(Cl)(H₂O)]

Calculated Molecular Weight - 571.82

Colour - Dark Brown

Decomposition Temperature - 318°C

Prepared by - Reflux Method

Elemental Analysis -

  Observed (%) Fe = 10.10, C=49.08, H = 4.92, N = 10.12, Cl = 7.05

  Calculated (%) Fe = 9.76, C = 50.36, H = 4.19, N = 9.79, Cl = 6.20

Molar Conductance - \( \Lambda \ 17.05 \ \text{ohm}^{-1} \ \text{cm}^2 \ \text{mole}^{-1} \)

Magnetic Measurement - Temperature = 300°C

- Magnetic Susceptibility=23.6481x10⁻⁶

- \( \mu_{\text{eff}} = 5.7 \ \text{BM} \)

Electronic Spectral Data - [Fig : 3.1]

Principal Bands Position [cm⁻¹] - 23866, 17361, 13404

Ligand Field Parameters - \( D_q = 613.8 \ \text{cm}^{-1} \)

- \( B = 558 \ \text{cm}^{-1} ; \beta = 0.5497 \)

Infrared Bands - [Fig : 3.2]

\begin{align*}
3695w & \quad 3670w & \quad 1617m \ \text{br} & \quad 1560 \ w & \quad 1443w & \quad 1323w \\
1253m \ w & \quad 1227w & \quad 874 \ w & \quad 846w & \quad 805sp & \quad 777m \ w \\
706w & \quad 617sp & \quad 539w & \quad 454w & \quad 421m \ w
\end{align*}
FIG: 3.1. ELECTRONIC SPECTRUM OF NALIDIXIC ACID Fe(III) COMPLEX.
FIG:3-B. INFRARED SPECTRUM OF NALIDIXIC ACID Fe(III) COMPLEX.
3 - Nalidixic acid - Ni (II) complex:-

Molecular Formula - \([\text{Ni}(\text{C}_{12}\text{H}_{11}\text{N}_{2}\text{O}_{3})_2(\text{H}_2\text{O})_2]\)

Calculated Molecular Weight - 557.17

Colour - Light Green

Decomposition Temperature - 291°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Ni = 11.00, C = 52.55, H = 5.01, N = 11.05
Calculated (%) Ni = 10.53, C = 51.68, H = 4.66, N = 10.05

Molar Conductance - \(\Lambda = 15.07 \, \text{ohm}^{-1} \, \text{cm}^2 \, \text{mole}^{-1}\)

Magnetic Measurement - Temperature = 300K

- Magnetic Susceptibility = 9.1506 x 10^-4
- \(\mu_{\text{eff}} = 3.5 \, \text{BM}\)

Electronic Spectral Data - [Fig : 3.2]

Principal Bands Position [cm\(^{-1}\)] - 25575, 19120, 12610

Ligand Field Parameters - \(D_q = 1261 \, \text{cm}^{-1}\)

- \(B = 457 \, \text{cm}^{-1} ; \beta = 0.4436\)

Infrared Bands - [Fig : 3.C]

3680w  3645w  1618m  1555mw  1450sp  1338sp
1225msp  1219w  870mw  840sp  782mw  695w
535m  448m
FIG: 3.2. ELECTRONIC SPECTRUM OF NALIDIXIC ACID Ni(II) COMPLEX.
FIG:3-C. INFRARED SPECTRUM OF NALIDIXIC ACID Ni(II) COMPLEX.
4 - Nalidixic acid - Cu (II) complex :-

Molecular Formula - \([\text{Cu} \left( \text{C}_{12}\text{H}_{11}\text{N}_{2}\text{O}_{3} \right)_2 \text{(OH)} \left( \text{H}_2\text{O} \right)_3] \]

Calculated Molecular Weight - 365.78

Colour - Light Blue

Decomposition Temperature - 301°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Cu = 16.99, C = 40.15, H = 5.12, N = 8.95

Calculated (%) Cu = 17.37, C = 39.36, H = 4.92, N = 7.65

Molar Conductance - \(\Lambda \ 20.15 \text{ ohm}^{-1} \text{ cm}^2 \text{ mole}^{-1}\)

Magnetic Measurement - Temperature = 300°K

- Magnetic Susceptibility = 4.2383x10^{-6}

- \(\mu_{\text{eff}} = 1.93 \text{ BM}\)

Electronic Spectral Data - [Fig : 3.3]

Principal Bands Position [cm\(^{-1}\)] - Broad assymetrical band near 14285-12500

Infrared Bands - [Fig : 3.D]

3689w 3648w 1624m 1558mw 1488m 1317w

1264sp 1229m w 1113m br 875w 857mw 811sp

786sp 673m 617sp 550m 463w
FIG:3.3. ELECTRONIC SPECTRUM OF NALIDIXIC ACID Cu-II COMPLEX.
FIG 3-D. INFRARED SPECTRUM OF NALIDIXIC ACID-Cu(II) COMPLEX.
5 - Nalidixic acid - Zn (II) complex :-

Molecular Formula - \([\text{Zn} (\text{C}_{12} \text{H}_{11} \text{N}_{2} \text{O}_3) \text{(OH)} \text{(H}_2\text{O})_3]\)

Calculated Molecular Weight - 367.62

Colour - White

Decomposition Temperature - 295°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (\%) Zn = 17.02, C = 40.01, H = 4.80, N = 7.05

Calculated (\%) Zn = 17.78, C = 39.17, H = 4.89, N = 7.60

Molar Conductance - 19.2 ohm\(^{-1}\) cm\(^2\) mole\(^{-1}\)

Magnetic Measurement - Diamagnetic

Infrared Bands - [Fig: 3.E]

3691w 3677w 1622m 1577m 1473mw 1317w
1261m w 1229sp 1132m w 891m w 875w 807sp
789w 775w 633m w 545w 447mw
FIG. 3-E. INFRARED SPECTRUM OF NALIDIXIC ACID Zn(II) COMPLEX.
1. Norfloxacirn - Mn (II) Complex :-

Molecular Formula - \[\text{Mn (C}_{16}\text{H}_{17}\text{F N}_{3}\text{O}_{3})\text{ (Cl) (H}_{2}\text{O})_{3}]\]

Calculated Molecular Weight - 462.72

Colour - Brownish

Decomposition Temperature - 333°C

Prepared by - Reflux Method

Elemental Analysis -

\[
\text{Observed (\%)} \quad \text{Mn} = 11.50, \text{ C} = 42.48, \text{ H} = 4.50, \text{ N} = 8.93, \text{ Cl} = 7.66
\]

\[
\text{Calculated (\%)} \quad \text{Mn} = 11.87, \text{ C} = 41.53, \text{ H} = 4.97, \text{ N} = 9.07, \text{ Cl} = 6.98
\]

Molar Conductance - \(\Lambda = 12.5 \text{ ohm}^{-1} \text{ cm}^{2} \text{ mole}^{-1}\)

Magnetic Measurement - Temperature = 300°K

- Magnetic Susceptibility = 30.2582x10^{-6}

- \(\mu_{\text{eff}} = 5.80 \text{ BM}\)

Electronic Spectral Data - [Fig : 3.4]

Principal Bands Position [cm^{-1}] - Broad assymetric band near 19200-13800

Infrared Bands - [Fig : 3.F]

3870 mw, 3735 mw, 3238 mw, 1624 sp, 1581 ms, 1390 ms

1259 s, 1190 msp, 1134 msp, 817 mw, 744 m, 623 sh, 511 w

482 sh, 416 w.
FIG. 3.4. ELECTRONIC SPECTRUM OF NORFLOXACIN Mn(II) COMPLEX.
FIG:3-F. INFRARED SPECTRUM OF NORFLOXACIN Mn(II) COMPLEX.
2. Norfloxacin - Fe (III) Complex :-

Molecular Formula - \[ \text{[Fe} (\text{C}_{16} \text{H}_{17} \text{F} \text{N}_{3} \text{O}_{3}) (\text{Cl})_{2} (\text{H}_{2} \text{O})_{2}] \]

Calculated Molecular Weight - 481.13

Colour - Brown

Decomposition Temperature - 245°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Fe = 11.69, C = 38.61, H = 5.10, N = 8.79, Cl = 13.76

Calculated (%) Fe = 11.60, C = 39.90, H = 4.46, N = 8.72, Cl = 14.75

Molar Conductance - \[ \Lambda = 16.20 \text{ ohm}^{-1} \text{ cm}^{2} \text{ mole}^{-1} \]

Magnetic Measurement - Temperature = 300°K

- Magnetic Susceptibility = 30.4321 x 10⁻⁶

- \( \mu_{\text{eff}} = 5.8 \) BM

Electronic Spectral Data - [Fig: 3.5]

Principal Bands Position [cm⁻¹] - 23807, 17331, 13368

Ligand Field Parameters - Dq = 612.7 cm⁻¹

- B = 557 cm⁻¹, β = 0.5487

Infrared Bands - [Fig: 3.G]

3805 mw, 3398 br, 3207sh, 1631 sp

1564 sp, 1402 msp, 1272 s, 1190 m

1118 ms, 813 w, 750 m, 621m, 516 mw,

455 w, 466 w.
FIG. 3.5. ELECTRONIC SPECTRUM OF NORFLOXACIN Fe(III) COMPLEX.
FIG3-G: INFRARED SPECTRUM OF NORFLOXACIN Fe(III) COMPLEX.
3. Norfloxacin - Ni (II) Complex

Molecular Formula - [Ni \( (\text{C}_{16}\text{H}_{17}\text{F}\text{N}_{3}\text{O}_{3})\text{(CH}_{3}\text{COO}\text{)}\text{(H}_{2}\text{O})_{2} \)]

Calculated Molecular Weight - 490.05

Colour - Green

Decomposition Temperature - 345°C

Prepared by - Reflux Method

Elemental Analysis

   Observed (%)   Ni = 10.97, C = 44.14, H = 5.18, N = 8.43
   Calculated (%) Ni = 11.97, C = 44.11, H = 5.30, N = 8.57

Molar Conductance - \( \Lambda 10.50 \text{ ohm}^{-1} \text{ cm}^{2} \text{ mole}^{-1} \)

Magnetic Measurement

   Temperature = 300°K
   Magnetic Susceptibility = 8.6969 \times 10^{-6}
   \( \mu_{\text{eff}} = 3.20 \text{ BM} \)

Electronic Spectral Data - [Fig : 3.6]

Principal Bands Position [cm\(^{-1}\)] - 25500, 19000, 12550

Ligand Field Parameters

   Dq = 1255 \text{ cm}^{-1}
   B = 456 \text{ cm}^{-1}; \beta = 0.4427

Infrared Bands

   3830 \text{ mw}, 3745 \text{ mw}, 3417 \text{ br}, 3076 \text{ sh}, 1631 \text{ ms}
   1568 \text{ shm}, 1494 \text{ m}, 1467 \text{ sh}, 1407 \text{ m}, 1342 \text{ mw}
   1267 \text{ m}, 1188 \text{ w}, 1120 \text{ w}, 820 \text{ w}, 746 \text{ shw} 626 \text{ shw}
   522 \text{ w}, 478 \text{ w}.
FIG:3.6 ELECTRONIC SPECTRUM OF NORFLOXACIN Ni(II) COMPLEX.
FIG:3-H.INFRARED SPECTRUM OF NORFLOXACIN Ni(II) COMPLEX.
4. Norfloxacin -Cu (II) Complex :-

Molecular Formula - [Cu (C₁₆H₁₇FN₃O₃)$_₂$(H₂O)$_₂$]

Calculated Molecular Weight - 736.28

Colour - Light Green

Decomposition Temperature - 232°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Cu = 7.71, C = 50.19, H = 5.0, N = 10.49

Calculated (%) Cu = 8.63, C = 52.20, H = 5.16, N = 11.40

Molar Conductance - A 13.10 ohm$^{-1}$ cm$^2$ mole$^{-1}$

Magnetic Measurement - Temperature = 300°K

- Magnetic Susceptibility = 1.8315x10$^{-6}$

- $\mu_{\text{eff}} = 1.80$ BM

Electronic Spectral Data - [Fig : 3.7]

Principal Bands Position [cm$^{-1}$] - Broad assymetric band near 13650-12030

Infrared Bands - [Fig : 3.1]

3884mw, 3749mw, 3381br, 3003w, 1627s

1579sp, 1390sp, 1390sp, 1259sp, 1191mw

1134mw, 830mw, 752mw, 624w, 516w, 484sh
FIG:3.7. ELECTRONIC SPECTRUM OF NORFLOXACIN Cu(II) COMPLEX.
FIG:3-I. INFRARED SPECTRUM OF NORFLOXACIN Cu(II) COMPLEX.
5. Norfloxacin -Zn (II) Complex :-

Molecular Formula - [Zn(C_{16}H_{17}F_{2}N_{2}O_{3})(CH_{3}COO)(H_{2}O)_{3}]

Calculated Molecular Weight - 496.75

Colour - White

Decomposition Temperature - 332°C

Prepared by - Reflux Method

Elemental Analysis

Observed (%) Zn = 13.33, C = 45.68, H = 5.10, N = 9.51

Calculated (%) Zn = 13.16, C = 43.52, H = 5.23, N = 8.45

Molar Conductance - \( \Lambda \) 15.08 ohm\(^{-1}\) cm\(^2\) mole\(^{-1}\)

Magnetic Measurement - Diamagnetic

Infrared Bands - [Fig: 3.J]

3550mw, 3257brsp, 1622m, 1577mw, 1492 mw, 1448sh, 1396m, 1338m, 1253shm, 1188mw,
1134shm, 819mw, 746sh, 623w, 505w, 482shw
FIG B-J. INFRARED SPECTRUM OF NORFLOXACIN Zn(II) COMPLEX.
1. Ciprofloxacin - Mn (II) Complex :

Molecular Formula - [Mn (C\(_{17}H_{17}F\)N\(_3\)O\(_3\))(Cl) (H\(_2\)O)\(_3\)]]

Calculated Molecular Weight - 474.80

Colour - Brownish Yellow

Decomposition Temperature - 318\(^\circ\)C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Mn = 11.83, C = 41.05, H = 5.01, N = 8.13, Cl = 8.05

Calculated (%) Mn = 11.56, C = 42.96, H = 4.84, N = 8.84, Cl = 7.47

Molar Conductance - \(\Lambda\) 18.20 ohm\(^{-1}\) cm\(^2\) mole\(^{-1}\)

Magnetic Measurement - Temperature = 300\(^\circ\)K

- Magnetic Susceptibility=23.7029x10\(^{-6}\)

- \(\mu_{eff} = 5.2\) BM

Electronic Spectral Data - [Fig : 3.8]

Principal Bands Position [cm\(^{-1}\)] - Broad asymmetric band near 19305-14044

Infrared Bands - [Fig : 3.K]

3892w, 3840w, 3791mw, 3735m, 3377br

3263sh, 1625sp, 1577m, 1386m, 1255m

1120w, 748w, 623w, 480w, 416w
FIG 3.8 ELECTRONIC SPECTRUM OF CIPROFLOXACIN Mn(II) COMPLEX.
FIG:3-K. INFRARED SPECTRUM OF CIPROFLOXACIN Mn(II) COMPLEX.
2. Ciprofloxacin - Fe (III) Complex :-

Molecular Formula - \([Fe(C_{17}H_{17}F\,N_{3}O_{3})_2(\OH)(\H_{2}O)]\)

Calculated Molecular Weight - 751.58

Colour - Dark Brown

Decomposition Temperature - 286°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Fe = 8.15, C = 55.32, H = 5.10, N = 11.60

Calculated (%) Fe = 7.42, C = 54.28, H = 4.92, N = 11.17

Molar Conductance - \(\Lambda 23.15 \ \text{ohm}^{-1} \ \text{cm}^2 \ \text{mole}^{-1}\)

Magnetic Measurement - Temperature = 300°K

- Magnetic Susceptibility=17.3662x10^{-6}

- \(\mu_{\text{eff}} = 5.6 \ \text{BM}\)

Electronic Spectral Data - [Fig : 3.9 ]

Principal Bands Position [cm⁻¹] - Broad assymetric band near 23923-17421

Infrared Bands - [Fig : 3.L]

3810w, 3750w, 3386br, 1622sp, 1585w, 1375sp

1292mw, 1263w, 1178mw, 1143mw, 860w, 827w, 785w, 725mw, 621w, 545br, 499w
FIG: 3.9. ELECTRONIC SPECTRUM OF CIPROFLOXACIN Fe(III) COMPLEX.
FIG: 3-L. INFRARED SPECTRUM OF CIPROFLOXACIN Fe(III) COMPLEX.
3. Ciprofloxacin - Ni (III) Complex

Molecular Formula - [Ni (C_{17}H_{17}F N_{3}O_{3})(CH_{3}COO) (H_{2}O)_{3}]

Calculated Molecular Weight - 502.06

Colour - Light Green

Decomposition Temperature - 330°C

Prepared by - Reflux Method

Elemental Analysis

Observed (%) Ni = 11.92, C = 45.36, H = 5.70, N = 8.22

Calculated (%) Ni = 11.68, C = 45.41, H = 5.17, N = 8.36

Molar Conductance - Λ 11.3 ohm^{-1} cm^{2} mole^{-1}

Magnetic Measurement

Temperature = 300°K

Magnetic Susceptibility = 8.4888 \times 10^{-6}

μeff = 3.20 BM

Electronic Spectral Data - [Fig : 3.10]

Principal Bands Position [cm^{-1}] - 25445, 18867, 12531

Ligand Field Parameters - Dq = 1253 cm^{-1}

- B = 447 cm^{-1}; β = 0.4339

Infrared Bands - [Fig : 3.M]

3805w, 3710w, 3315br sh, 1625sp, 1577sp, 1485sp, 1452w, 1392spm, 1307 msp, 1255sp, 1184mw, 1120w, 891w, 862w, 754mw, 626w, 545w, 426w
FIG 3.10. ELECTRONIC SPECTRUM OF CIPROFLOXACIN NI(II) COMPLEX.
FIG. 3.14. INFRARED SPECTRUM OF CIPROFLOXACIN Ni(II) COMPLEX.
4. Ciprofloxacin - Cu (II) Complex :-

Molecular Formula - \([\text{Cu}(\text{C}_{17}\text{H}_{12}\text{F}\text{N}_{2}\text{O}_{3})_{2}(\text{H}_{2}\text{O})_{2}]\)

Calculated Molecular Weight - 760.28

Colour - Green

Decomposition Temperature - 239°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Cu = 8.97, C = 50.82, H = 4.96, N = 10.15

Calculated (%) Cu = 8.35, C = 53.66, H = 4.73, N = 11.04

Molar Conductance - \(\Lambda 15.25 \text{ ohm}^{-1} \text{ cm}^2 \text{ mole}^{-1}\)

Magnetic Measurement - Temperature = 300°K

- Magnetic Susceptibility = \(1.4366 \times 10^{-6}\)

- \(\mu_{\text{eff}} = 1.62 \text{ BM}\)

Electronic Spectral Data - [Fig : 3.11]

Principal Bands Position [cm\(^{-1}\)] - Broad assymetric band near 13703-12150

Infrared Bands - [Fig : 3.N]

3840w, 3791, 3730w, 3388br sh, 3298sh, 1624w,

1581w, 1384m, 1257m, 1184mw, 1118w, 891w,

790w, 756m, 626w, 551w, 416w
FIG. 3.11. ELECTRONIC SPECTRUM OF CIPROFLOXACIN Cu(II) COMPLEX.
FIG:3-N. INFRARED SPECTRUM OF CIPROFLOXACIN Cu(II) COMPLEX.
5. Ciprofloxacin - Zn (II) Complex :-

Molecular Formula - [Zn(C_{17}H_{17}F_{N_{3}}O_{3})(CH_{3}COO)(H_{2}O),]

Calculated Molecular Weight - 508.75

Colour - White

Decomposition Temperature - 298°C

Prepared by - Reflux Method

Elemental Analysis -

   Observed (%) Zn = 13.02, C = 44.84, H = 5.42, N = 8.12

   Calculated (%) Zn = 12.85, C = 44.81, H = 5.11, N = 8.25

Molar Conductance - \( \Lambda 9.5 \text{ ohm}^{-1} \text{ cm}^{2} \text{ mole}^{-1} \)

Magnetic Measurement - Diamagnetic

Infrared Bands - [Fig : 3.0]

3830w, 3807w, 3745w, 3714w, 3398br sh,
1625sp, 1579m, 1485m, 1388mw, 1309m,
1259m, 1184mw, 1114w, 752w, 513w, 470w
FIG:3.0. INFRARED SPECTRUM OF CIPROFLOXACIN Zn(II) COMPLEX.
1. Cloxacillin - Mn (II) Complex :

Molecular Formula - [Mn (C₁₉H₁₉ClN₂O₆S)(Cl) (H₂O)₂]

Calculated Molecular Weight - 597.27

Colour - Light Yellow

Decomposition Temperature - 228°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Mn = 9.37, C = 37.50, H = 4.30, N = 6.82, S = 2.27

Calculated (%) Mn = 9.19, C = 38.17, H = 4.18, N = 7.03, S = 5.35

Molar Conductance - \( \Lambda \) 22.10 ohm\(^{-1}\) cm\(^2\) mole\(^{-1}\)

Magnetic Measurement - Temperature = 298°C

- Magnetic Susceptibility = 1.68434 x 10\(^{-6}\)
- \( \mu \text{eff} = 4.90 \) BM

Electronic Spectral Data - [Fig: 3.12]

Principal Bands Position [cm\(^{-1}\)] - 26215, 23510, 17320

Ligand Field Parameters - Dq = 793.8 cm\(^{-1}\)

- B = 721 cm\(^{-1}\); \( \beta = 0.8391 \)

Infrared Bands - [Fig: 3.P]

3649w 3564w 3548w 3266w 1775mw

1670br 1631mbr 1423w 1345m 898w

587w 454mw 420w
FIG:312. ELECTRONIC SPECTRUM OF CLOXACILLIN Mn(II) COMPLEX.
FIG. 3: P. INFRARED SPECTRUM OF CLOxacillin Mn (II) COMPLEX
2. Cloxacillin - Fe (III) Complex:

Molecular Formula: 
\[ \text{Fe} \left( \text{C}_{19}\text{H}_{19}\text{Cl N}_3\text{O}_6\text{S} \right) \left( \text{SO}_4 \right) \left( \text{H}_2\text{O} \right) \]

Calculated Molecular Weight: 640.73

Colour: Brown

Decomposition Temperature: 190°C

Prepared by: Reﬂux Method

Elemental Analysis:

Observed (%): Fe = 9.10, C = 34.26, H = 4.61, N = 8.65, S = 9.34

Calculated (%): Fe = 8.91, C = 35.58, H = 3.58, N = 6.55, S = 9.98

Molar Conductance: \( \Lambda \; 17.05 \; \text{ohm}^{-1} \; \text{cm}^2 \; \text{mole}^{-1} \)

Magnetic Measurement:
- Temperature = 298°C
- Magnetic Susceptibility = 17.0088 x 10^{-6}
- \( \mu_{\text{eff}} = 5.10 \; \text{BM} \)

Electronic Spectral Data:
- [Fig : 3.13]

Principal Bands Position [cm^{-1}]: 23980, 17513, 13774

Ligand Field Parameters:
- \( D_q = 630.3 \; \text{cm}^{-1} \)
- \( B = 573 \; \text{cm}^{-1}; \beta = 0.5645 \)

Infrared Bands:
- [Fig : 3.Q]

3652w 3210mw 1782m 1657mw 1604w
1401mbr 1324w 1112m 1035w 895w
643sp 580w 483w 462w
FIG3.13.ELECTRONIC SPECTRUM OF CLOXACILLIN Fe(III) COMPLEX.
FIG:3-Q. INFRARED SPECTRUM OF CLOxacillin Fe(III) COMPLEX
3. Cloxacillin - Ni (II) Complex ::-

Molecular Formula  -  \([\text{Ni} (\text{C}_{19}\text{H}_{19}\text{Cl N}_{3}\text{O}_{6}\text{S}) (\text{OH}) (\text{H}_{2}\text{O})_{3}]\)

Calculated Molecular Weight  -  582.58

Colour  -  Light Green

Decomposition Temperature  -  229°C

Prepared by  -  Reflux Method

Elemental Analysis  -

  Observed (%)  Ni = 10.90, C = 39.47, H = 4.94, N = 8.63, S = 4.09

  Calculated (%)  Ni = 10.07, C = 39.13, H = 4.46, N = 7.20, S = 5.49

Molar Conductance  -  Insoluble

Magnetic Measurement  -  Temperature = 298°C

  Magnetic Susceptibility = 6.9116\times10^{-6}

  \(\mu_{\text{eff}} = 3.10\) BM

Infrared Bands  -  [Fig: 3.R]

  3510w  3490w  3215w  1715w  1695mw
  1620mw  1402mbr  1383w  1127m  898w
  574w  461w
FIG. 3-R. INFRARED SPECTRUM OF CLOXACILLIN Ni (II) COMPLEX
4. Cloxacillin - Cu (II) Complex :

Molecular Formula  - [Cu (C₁₅H₁₉ClN₃O₆S)(OH) (H₂O)₃]
Calculated Molecular Weight  - 587.43
Colour  - Grey
Decomposition Temperature  - 182°C
Prepared by  - Reflux Method
Elemental Analysis

Observed (%)  Cu = 11.20, C = 37.82, H = 3.79, N = 7.21, S = 4.19
Calculated (%) Cu = 10.81, C = 38.18, H = 4.42, N = 7.14, S = 5.44

Molar Conductance  - Λ 14.60 ohm⁻¹ cm² mole⁻¹

Magnetic Measurement  - Temperature = 298⁰K  
                        - Magnetic Susceptibility=2.1101x10⁻⁶  
                        - μeff = 1.72 BM

Electronic Spectral Data  - [Fig : 3.14]

Principal Bands Position [cm⁻¹]  - Broad assymetric band near 13793- 11764

Infrared Bands  - [Fig : 3.5]

3498br  3479br  3444br  3289wbr  1726w  
1643sh  1627sh  1408mw  1344w  1112br  
898w  574w  465w
FIG 3.14. ELECTRONIC SPECTRUM OF CLOXACILLIN Cu(II) COMPLEX.
FIG. 3 - INFRARED SPECTRUM OF CLOXACILLIN Cu(II) COMPLEX
5. Cloxacillin - Zn (II) Complex :-

Molecular Formula - [Zn (C_{19}H_{15}Cl N_{3}O_{6}S)(OH) (H_{2}O)_{3}]

Calculated Molecular Weight - 589.27

Colour - White

Decomposition Temperature - 221°C

Prepared by - Reflux Method

Elemental Analysis -

Observed (%) Zn = 11.50, C = 37.60, H = 4.59, N = 7.72, S = 4.89

Calculated (%) Zn = 11.09, C = 38.69, H = 4.41, N = 7.12, S = 5.43

Molar Conductance - \( \Lambda \) 17.10 ohm\(^{-1}\) cm\(^{2}\) mole\(^{-1}\)

Magnetic Measurement - Diamagnetic

Infrared Bands - [Fig: 3.T]

3520w 3495w 3225br 1710mw 1685mw
1635w 1402mw 1350w 1113m 899m
570w 462w
FIG: 3-T. INFRARED SPECTRUM OF CLOXACILLIN Zn (II) COMPLEX
REFERENCES


2. Ibid., P. 26.

3. Ibid., P. 433.

4. Ibid., P. 335.
