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# SYNTHESIS OF MACROCYCLIC LIGANDS AND THEIR COBALT(II) COMPLEXES SPECTRAL CHARACTRIZATION AND ANTIMICROBIAL STUDIES

Sulekh Chandra [*1] and Poonam Pipil [2] and Swati Agarwal [3]

Department of Chemistry [1],Zakir Husain Delhi Collage (University of Delhi) JLN-Marg, New Delhi, India
Department of Chemistry [2], Rajdhani College, University of Delhi, Delhi 110015, India
Department of Chemistry [3], Moti Lal Nehru College, Benito Juarez Marg, New Delhi 110021, India

**Keywords:**

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**Correspondence to Author:**
**Dr. Sulekh Chandra**

Department of Chemistry, Zakir Husain Delhi Collage (University of Delhi) JLN-Marg, New Delhi 110002 India

**E-mail**: www.poonampipil@gmail.com

**ABSTRACT:** Complexes of Co(II) with macrocylic ligands i.e., 2, 3, 9, 10 tetraphenyl – 1, 4, 8, 11 – tetraazacyclotetradeca – 1, 3, 8, 10 tetraene (BDP), 2, 4, 10, 12 – tetramethyl – 1, 5, 9, 13 tetraazacyclohexadeca 1, 4, 9, 12 tetraene (ADP), 2, 3, 9, 10 tetramethyl 1, 4, 8, 11 tatraazatetredeca – 1, 3, 8, 10 tetraene (DDP) have been synthesized. All these complexes were characterized by elemental analysis, molar conductance magnetic susceptibility measurement, electronic and epr spectral studies. On the basis of elemental analysis the complexes were found to have composition CoLX2. Molar conductance measured in DMF solution indicates that complexes are non-electrolytic in nature except Co(BDP)SO4 which are 1 : 2 electrolytes. Thus the complexes may be formulated as [CoCLX2]. Magnetic moments and the electronic spectra indicate six coordinated distorted octahedral geometry for the complexes. The ligand field parameters were calculated using various energy level diagrams. In vitro synthesized compounds and metal salts have also been tested against same species of plant pathogenic fungi and bacteria in order to assess their microbial properties.

**INTRODUCTION:** Azo schiff base complexes contain both azo and azomethine groups. The azo group possess excellent donor properties and is important in coordination chemistry [1-3] and some azo compounds have shown to possess good antibacterial activity [4-5]. Schiff bases are well known to have antifungal, antitumor and herbicidal activities [6-11]. Many metal complexes of naturally occuring porphyrins, corrins and phthalocyanines have been investigated because of their potential as dyestuffs or pigments [12]. A schiff base complexes are of great importance [13] in enehancing various industrial applications and in a number of biological processes such as photosynthesis and dioxygen transport [14].

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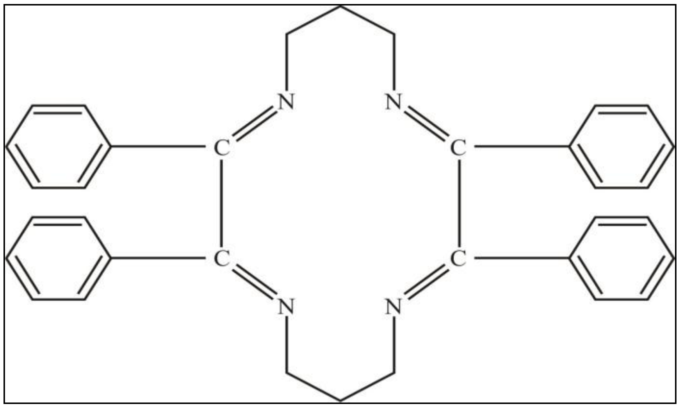
Cobalt complexes, besides their presence in several stereochemical dispositions, are oxygen carriers and oxygen activators [20-23] which interact with molecular oxygen and ultimately oxidize the complexes of Co(II) to Co(III). The best known biological function of cobalt is its involvement in the coenzyme related to vitamin $B_{12}$ [15-16].

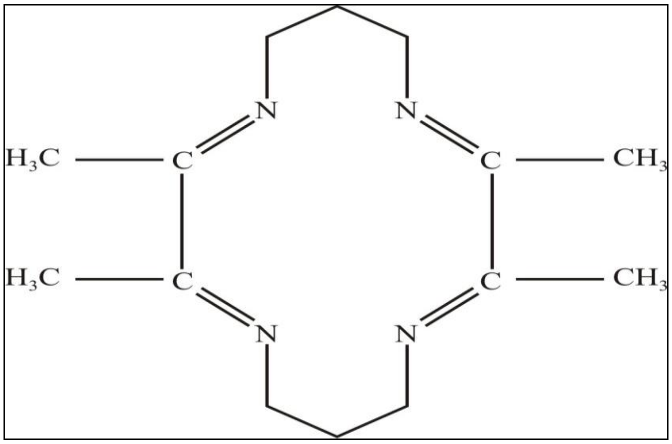
In the present paper we report the synthesis and characterization of Schiff base complexes of cobalt (II) with 2, 3, 9, 10 tetraphenyl – 1, 4, 8, 11 – tetraazacyclotetradeca – 1, 3, 8, 10 tetraene. (BDP) 2, 4, 10, 12 – tetramethyl – 1, 5, 9, 13 – tetraazacyclohexadeca – 1, 4, 9, 12 tetraene. (ADP) 2, 3, 9, 10 tetramethyl 1, 4, 8, 11 tetraazacyclohexadeca – 1, 3, 8, 10 tetraene (DDP). **Fig (1a-1c).**

## Experimental Section:

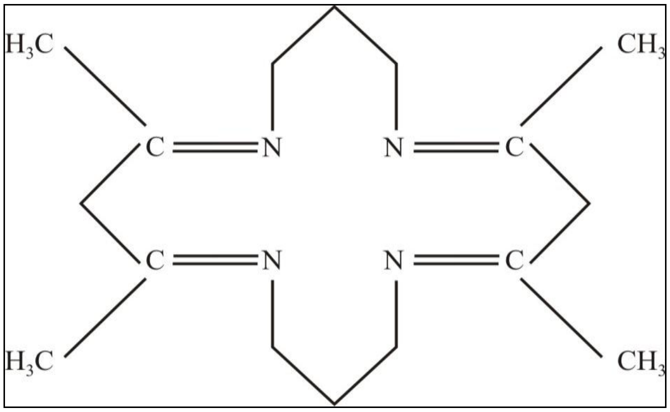
**Materials:** All the chemicals used in the present work of high purity, Anala R grade and purchased from Sigma-Aldrich. Metal salts were purchased from E. Merck and used as received. The Solvent used were either spectroscopic pure from SRL/BDH or purified by the recommended methods.



(FIG. 1A)



(FIG. 1B)



(FIG. 1C)

STRUCTURE OF LIGANDS (FIG 1A-1C)

**1(a)** 2, 3, 9, 10 tetraphenyl – 1, 4, 8, 11 – tetraazacyclotetradeca – 1, 3, 8, 10 tetraene (BDP)

**1(b)** 2, 4, 10, 12 – tetramethyl – 1, 5, 9, 13 tetraazacyclohexadeca 1, 4, 9, 12 tetraene (ADP),

**1(c)** 2, 3, 9, 10 tetramethyl 1, 4, 8, 11 tatraazatetredeca – 1, 3, 8, 10 tetraene (DDP)

### Preparation of ligands:

### Preparation of 2, 3, 9, 10 tetraphenyl – 1, 4, 8, 11 – tetraazacyclo tetradeca – 1, 3, 8, 10 tetraene. (BDP):

The ligand 2, 3, 9, 10 tetraphenyl – 1, 4, 8, 11 – tetraazacyclo tetradeca – 1, 3, 8, 10 tetraene. (BDP) was synthesized by refluxing an ethanolic solution of 1, 3 diaminopropane (0.5 mole) with an ethanolic solution of benzyl, (0.5 mole) in presence of ~3 mL of conc. HC1 for 3-4 hours. The resulting mixture was kept overnight, when an off-white coloured crystalline compound separated. This was then filtered, washed with ethanol, and dried over $P_4O_{10}$. The ligand is soluble in water and melted at $218^0$C.

### Preparation of 2, 4, 10, 12 – tetramethyl – 1, 5, 9, 13 – tetraazacyclohexadeca – 1, 4, 9, 12 tetraene. (ADP):

2, 4, 10, 12 – tetramethyl – 1, 5, 9, 13 – tetraazacyclohexadeca – 1, 4, 9, 12 tetraene. (ADP) was prepared by adding an ethanolic solution of acetylacetone (0.5 mole) to an ethanolic solution of 1,3 diaminopropane (0.5 mole) in presence of ~3 mL conc. HC1 and the resulting solution was refluxed for 4 hours and kept overnight. A white crystalline compound separated on filtration which was washed with ethanol and then dried over $P_4O_{10}$. The compound was soluble in most organic solvents, and it’s melting point was recorded as $226^0$C.

### Preparation of 2, 3, 9, 10 tetramethyl 1, 4, 8, 11 tatraazatetredeea – 1, 3, 8, 10 tetraene (DDP):

2, 3, 9, 10 tetramethyl 1, 4, 8, 11 tatraazatetredeea – 1, 3, 8, 10 tetraene (DDP) ligand was synthesized by diacetyl (0.5 mole) to an ethanolic solution of 1, 3 diaminopropane (0.5 mole) in presence of ~3 mL conc. HC1 and the resulting solution was refluxed for 4 hours and then kept overnight. Light-yellow crystals separated on filteration which were washed with ethanol and then dried over $P_4O_{10}$ The ligend is water soluble & melts at $221^0$C

### Preparation of Co(II) complexes with BDP, ADP and DDP ligands:

The complexes of the ligands BDP, ADP and DDP ligands have been synthesized by template method because the yield of the complexes was low when the ligands were created with metal salt to form complexes.

A hot ethanolic solution of benzil/ acetylacetone / diacetyl (0.01 mole) was added to an ethanolic solution of 1, 3 diaminopropane (0.01 mole) and the resulting solution was refluxed for half and hour at ~ $40^0$C. A solution of $CoX_2.nH_2O$(0.005 mole, n (0-6) (X=$Cl^-$, $NO_3^-$, $NCS^-$ in ethanol and X = ½ $SO_4^{2-}$ in water) was then added to the above solution and refluxing continued for a further four to six hours. On cooling the solution, pink/brown crystalline compounds separated out. They were filtered, washed with ethanol, and dried under vacuum over $P_4O_{10}$.

**Physical Measurement:**

The C, H and N were analysed in Carlo-Erba 1106 elemental analyzer. Molar conductance was measured on an ELICO (CM82T) conductivity bridge. Magnetic susceptibility was measured at room temperature, on CAHN-2000 magnetic susceptibility balance using $CuSO_4.5H_2O$ as a calibrant. Infrared spectra of ligands and complexes were recorded as KBr pelletson a Pekin-Elmer 1310 spectrophotometer. The electronic spectra of complexes were recorded in DMSO, on a shimadzu UV mini-1240 spectrophotometer. EPR spectra of complexes were recorded on JEOL, JES, FE3XG, EPR spectrometer. The spectra were recorded in solid as polycrystalline sample at room temperature on $E_4$-EPR spectrometer using the DPPH as the g-marker.

## RESULTS AND DISCUSSION:

On the basis of elemental analyses **(Table 1)** all the complexes have the composition $CoLX_2$. Molar conductance of the complexes indicates that the complexes are non-electrolytic in nature, except complexes $Co(BDP)SO_4$. Which are 1: 2 electrloytes. Thus the complexes may be formulated as $[CoLX_2]$ and [CoL] $SO_4$ respectively. Magnetic moments and the electronic spectra of the complexes discussed in succeeding paragraphs suggest six coordinated distorted octahedral geometry for the complexes.

**TABLE 1: ELEMENTAL ANALYSES AND MOLAR CONDUCTANCE DATA OF COBALT(II) COMPLEXES**

| Complex | Colour | Molar Conductance | Elemental Analysis, % Found/(Calc) | | | | % Yield | M.P.$^0$C |
|---|---|---|---|---|---|---|---|---|
| | | ($\Omega^{-1}cm^{-1}mol^{-1}$) | C | H | N | Co | | |
| [Co(BDP)$C1_2$] | Mauve Pink | 18.3 | 65.20 | 5.10 | 8.58 | 9.10 | 55 | 270 |
| | | | (65.36) | (5.12) | (8.89) | (9.35) | | |
| [Co(BDP)$(NO_3)_2$] | Blue | 14.6 | 60.84 | 5.00 | 12.64 | 8.12 | 60 | 240 |
| | | | (60.29) | (4.72) | (12.31) | (8.63) | | |
| [Co(BDP)$SO_4$ | Blue | 185 | 62.03 | 4.88 | 8.78 | 8.35 | 62 | 220 |
| | | | (62.87) | (4.93) | (8.55) | (8.99) | | |
| [Co(BDP)$(NCS)_2$] | Light brown | 12.8 | 64.21 | 4.23 | 12.19 | 8.48 | 58 | 233 |
| | | | (64.56) | (4.78) | (12.45) | (8.72) | | |
| [Co(ADP)$C1_2$] | Brown | 13 | 48.84 | 7.00 | 13.88 | 14.66 | 64 | 222 |
| | | | (47.50) | (6.92) | (13.74) | (14.44) | | |
| [Co(ADP)$(NO_3)_2$] | Dull Green | 12.8 | 41.97 | 5.98 | 17.90 | 12.42 | 66 | 242 |
| | | | (42.04) | (6.12) | (18.24) | (12.78) | | |
| [Co(ADP)$SO_4$] | Pink | 13 | 44.88 | 6.62 | 12.63 | 13.29 | 59 | 256 |
| | | | (44.76) | (6.52) | (12.94) | (13.60) | | |
| [Co(ADP)$(NCS)_2$] | Greyish Green | 14.2 | 48.42 | 6.10 | 18.98 | 12.68 | 62 | 230 |
| | | | (48.09) | (6.23) | (18.54) | (12.99) | | |
| [Co(DDP)$C1_2$] | Green | 13.1 | 44.74 | 6.25 | 14.62 | 15.92 | 65 | 234 |
| | | | (44.66) | (6.37) | (14.76) | (15.51) | | |
| [Co(DDP)$(NO_3)_2$] | Yellowish Pink | 12.2 | 39.39 | 5.48 | 19.72 | 13.88 | 55 | 236 |
| | | | (39.19) | (5.59) | (19.43) | (13.61) | | |
| [Co(DDP)$SO_4$] | Light Blue | 11.8 | 41.64 | 5.72 | 13.98 | 14.42 | 60 | 242 |
| | | | (41.89) | (5.98) | (13.84) | (14.55) | | |
| [Co(DDP)$(NCS)_2$] | Whitish Brown | 15.4 | 45.63 | 5.81 | 19.10 | 13.14 | 61 | 262 |
| | | | (45.59) | (5.69) | (19.77) | (13.86) | | |

**IR Spectra:**

IR Spectra of all the complexes do not contain any bands that can be assigned to C=O or N-H groups. Characteristic IR bands due to phenyl groups are present in the spectra of complexes **Fig (2a-2b)** BDP in the region of 700-770 $cm^{-1}$. Strong bands appearing as doublets in the spectra of all the complexes around 1590-1620 $cm^{-1}$ may be assigned to $v_{C=N}$ vibrations. The absence of absorptions around 3400 $cm^{-1}$ show that amino group of the diamine have reacted with the di ketone.

The phenyl ring absorptions also appear in the 1400-1600 $cm^{-1}$ region. The presence of new bands at *ca.* 1272, *ca.* 1190 and *ca.* 1030 $cm^{-1}$ assignable to $v(C\text{-}CH_3)+v(C=C)$, $\delta(CH)+v(C\text{-}CH_3)$ and $\rho_r(CH_3)$ show that the diketone moiety is present in the complexes. The changes in the position $v_{C=N}$ vibrations indicate coordination through this site. The bands at *ca.* 1640 and 840 $cm^{-1}$ may be assigned to NH deformation coupled with NH out of plane bending and may be due to the presence of this group in these complexes. Thus in presence of metal salts, a quadridentate macrocycle is formed which coordinate through azomethine nitrogens while pyridine nitrogen does not take part in coordination[17-18].

**Bands due to anions:**

**Co(ADP(NCS)$_2$] Complex:**

The IR spectrum of [Co(ADP)(NCS)$_2$] shows a strong band at 2075 $cm^{-1}$ thereby indicating that the thiocyanate group is N-coordinated. The absence of a band around 2140 $cm^{-1}$ shows that there is no coordination through S so both the thiocyanate groups are N-coordinated. A single shap band at 474 $cm^{-1}$ also confirms that the complex is N-bonded.

**Co(DDP)SO$_4$:**

The IR spectra Co(DDP)SO$_4$ shows the splitting of $v_3$ into three bands around 1200, 23 and 1080 $cm^{-1}$ and at 669 and 660 $cm^{-1}$ indicating the bidenate nature of the sulphate group. Similar is the case with Co(DDP)SO$_4$ while for the other complexes Co(BDP)SO$_4$, sulphate does not show coordination with the metal ion as only one strong broad band is observed around 1100 $cm^{-1}$.

**Co(BDP(NO$_3$)$_2$ and Co(ADP)(NO$_3$)$_2$:**

The appearance of new bands at *ca.* 1405($v_1$), 1187($v_5$) and 825 $cm^{-1}$ ($v_6$) in the spectra of nitrato complex of BDP indicates the monodentate nature of the nitrate group, since the separation between $v_1$ and $v_5$ is 218 $cm^{-1}$ conforming to the unidentate group, while the presence of a single band at *ca.* 1383 $cm^{-1}$ in the nitrato complex of ADP indicates that the nitrate group is uncoordinated.

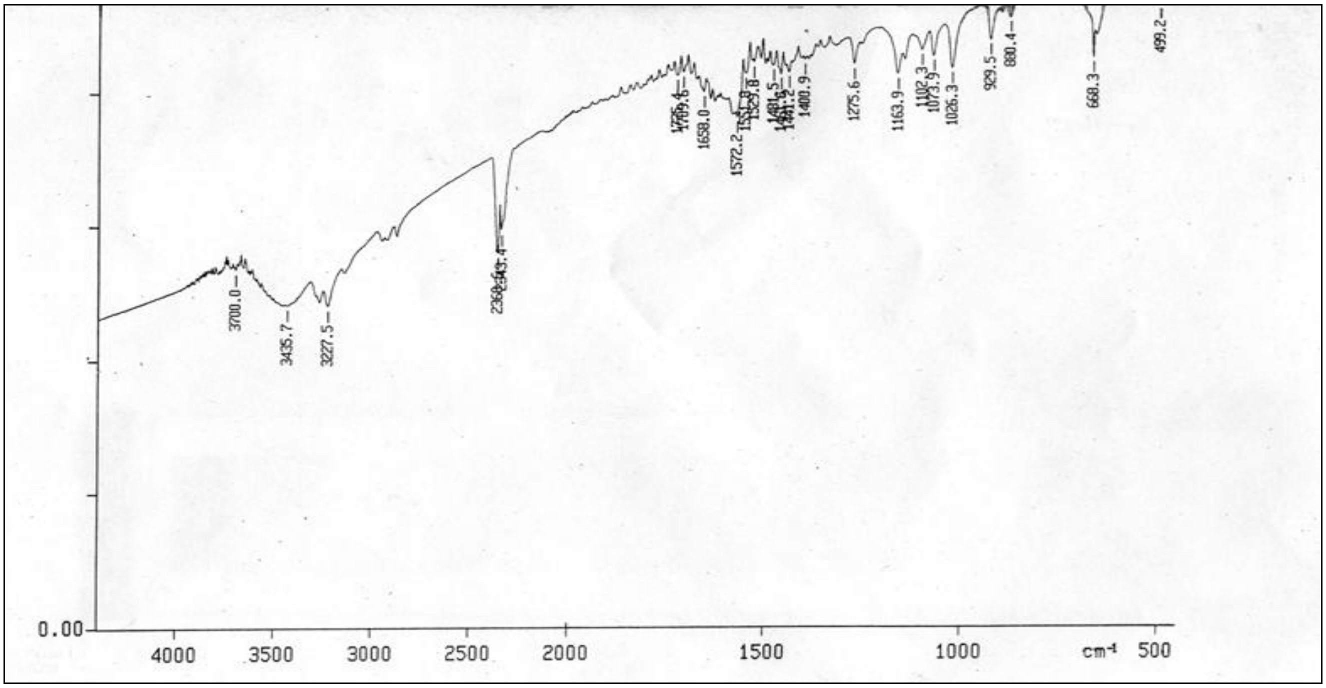


FIG 2a: IR SPECTRUM OF [Co (BDP) (NO$_3$)$_2$] COMPLEX

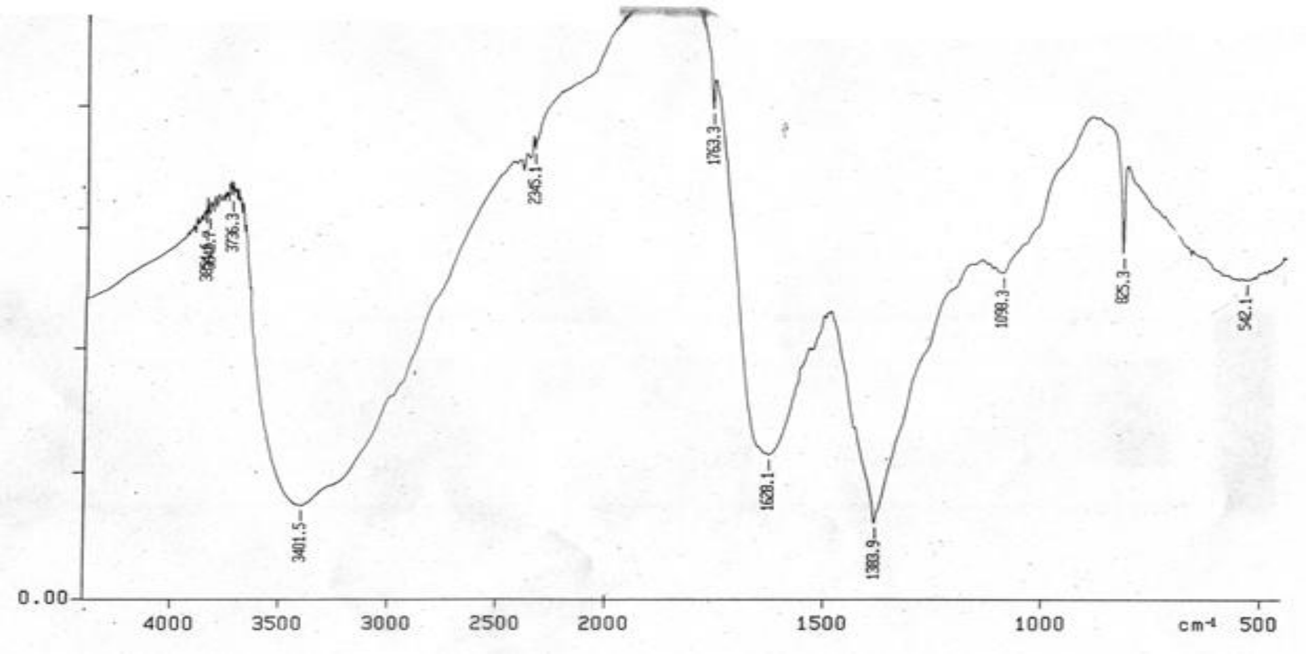


**FIG 2b: IR SPECTRUM OF [Co (DDP) $(NO_3)_2$] COMPLEX**

## Magnetic Moments:

Experimental magnetic moment in literature [19-21] lie in the range 4.42 to 4.7 B.M. Magnetic moments of the complexes under study have been determined at room temperature and lie in the range of 4.95-5.04 B.M. indicating a spin quartet six coordinate octahedral $Co^{2+}$ complexes and 4.49-4.68 B.M. for sulphato complexes indicating four coordinate geometry **(Table 2).**

**TABLE 2: MAGNETIC MOMENTS AND ELECTRONIC SPECTRAL BANDS OF COBALT (II)**

| Complex | $\mu_{eff}$(B.M.) | $v_1$ ($cm^{-1}$) | $v_2$ ($cm^{-1}$) | $v_3$ ($cm^{-1}$) |
|---|---|---|---|---|
| [Co(BDP)$C1_2$] | 4.98 | 8970 | 14410 | 20110 |
| [Co(BDP)$(NO_3)_2$] | 5.01 | 9722 | 14278 | 20456 |
| [Co(BDP)]$SO_4$ | 4.68 | | 14040 | 20768 |
| [Co(BDP)$(NCS)_2$] | 4.97 | 9088 | 14788 | 20367 |
| [Co(ADP)$C1_2$] | 4.98 | 8827 | 14476 | 21055 |
| [Co(ADP)$(NO_3)_2$] | 5.02 | 8693 | 14541 | 18656 |
| [Co(ADP)$SO_4$] | 5.04 | 9040 | 14409 | 21114 |
| [Co(ADP)$(NCS)_2$] | 5.00 | 8450 | 14098 | 20248 |
| [Co(DDP)$C1_2$] | 5.01 | 8765 | 14748 | 21691 |
| [Co(DDP)$(NO_3)_2$] | 4.99 | 8563 | 14211 | 22123 |
| [Co(DDP)$SO_4$] | 4.98 | 9142 | 14468 | 21900 |
| [Co(DDP)$(NCS)_2$] | 5.04 | 9079 | 14223 | 20168 |

## Electronic Spectra:

Electronic spectra of the complexes under study disply two well defined spectral bands at 8700-9600 $cm^{-1}$ and 20200-21000 $cm^{-1}$ and a shoulder at 14000-14800 $cm^{-1}$ to $^4T_{1g} \rightarrow {}^4T_{2g}$ (F) ($v_1$), $^4T_{1g} \rightarrow {}^4T_{1g}$ (P) ($v_3$) and $^4T_{1g} \rightarrow {}^4T_{2g}$ (F) ($v_2$) transitions respectively, characteristic of octahedral geometry of cobalt(II) complexes. The electronic spectral bands indicates that these macrocyclic complexes have distorted octahedral geometries and might be possessing $D_{4th}$ symmetr [22-24]

## Co(ADP$(NCS)_2$] Complex:

Electronic spectra of the complexes under study disply two well defined spectral bands at 8700-9600 $cm^{-1}$ d and 20200-21000 $cm^{-1}$ and a shoulder at 14000-14800 $cm^{-1}$ to $^4T_{1g} \rightarrow {}^4T_{2g}$ (F) ($v_1$), $^4T_{1g} \rightarrow {}^4T_{1g}$ (P) ($v_3$) and $^4T_{1g} \rightarrow {}^4T_{2g}$ (F) ($v_2$) transitions respectively, characteristic of octahedral geometry of cobalt(II) complexes. The electronic spectral bands indicates that these macrocyclic complexes have distorted octahedral geometries and might be possessing $D_{4th}$ symmetry [22-24]

## Co(DDP)$SO_4$ :

The three electronic transitions occur from the ground state. $^4$ $A_2$. The first $v_1$ is rarely observed while $v_2$ is usually wide and apeears in the near IR region and $v_3$ is intense and broad occurring in the region 15000–21000 $cm^{-1}$.

## Co(BDP$(NO_3)_2$ and Co(ADP)$(NO_3)_2$:

The electronic spectra of all the nitroto complexes show three bands around 8700, 14000, and 20500 $cm^{-1}$, which are characteristic of octahedral cobalt(II) complexes.

## Ligand Field Parameters:

Various ligand field parameters Dq, B and β have been calculated and given in **Table 3**. Dq values have been evaluated by using Orgel energy level diagram[31] Nephelauxetic parameters β has been calculated by using equation.

$$\beta = \frac{B(\text{Complex})}{B(\text{free ion})}$$

Where B (free ion) [25] is 1120 $cm^{-1}$. The value of β in the present study indicates appreciable covalent character.

**TABLE 3: LIGAND FIELD PARAMETERS AND ESR SPECTRAL DATA OF COBALT(II) COMPLEXES**

| Complex | Dq($cm^{-1}$) | B($cm^{-1}$) | β | $g_{\|}$ | $g_{\perp}$ |
|---|---|---|---|---|---|
| [Co(BDP)$Cl_2$] | 1050.14 | 1093.90 | 0.98 | 2.13 | 1.92 |
| [Co(BDP)$(NO_3)_2$] | 1014.44 | 1056.71 | 0.94 | 2.10 | 1.93 |
| [Co(BDP)]$SO_4$ | | | | 2.39 | 2.00 |
| [Co(BDP)$(NCS)_2$] | 1063.96 | 1108.29 | 0.99 | 2.06 | 1.95 |
| [Co(ADP)$Cl_2$] | 1033.40 | 1076.46 | 0.96 | 2.05 | 1.99 |
| [Co(ADP)$(NO_3)_2$] | 1017.72 | 1060.12 | 0.95 | 2.03 | 2.00 |
| [Co(ADP)$SO_4$] | 1058.34 | 1102.44 | 0.98 | 2.08 | 1.93 |
| [Co(ADP)$(NCS)_2$] | 989.65 | 1030.89 | 0.92 | 2.11 | 1.92 |
| [Co(DDP)$Cl_2$] | 1026.14 | 1068.90 | 0.95 | 2.04 | 1.99 |
| [Co(DDP)$(NO_3)_2$] | 1006.01 | 1047.93 | 0.94 | 2.06 | 2.00 |
| [Co(DDP)$SO_4$] | 1014.32 | 1056.59 | 0.94 | 2.07 | 1.88 |
| [Co(DDP)$(NCS)_2$] | 1062.91 | 1107.20 | 0.99 | 2.15 | 1.98 |

## EPR Spectra:

EPR spectra of the polycrystalline complexes under study [25] were recorded at liquid nitrogen temperature **Fig (3a).** Since, the rapid spin lattice relaxation of $Co^{2+}$ broadens the lines at higher temperatures, g-values are presented in **Table 3**. The large deviation of the g values from the spin only value (g = 2.0023) is due to the large angular momentum contribution, this result accords with the magnetic susceptibilities and electronic spectra as discussed earlier.

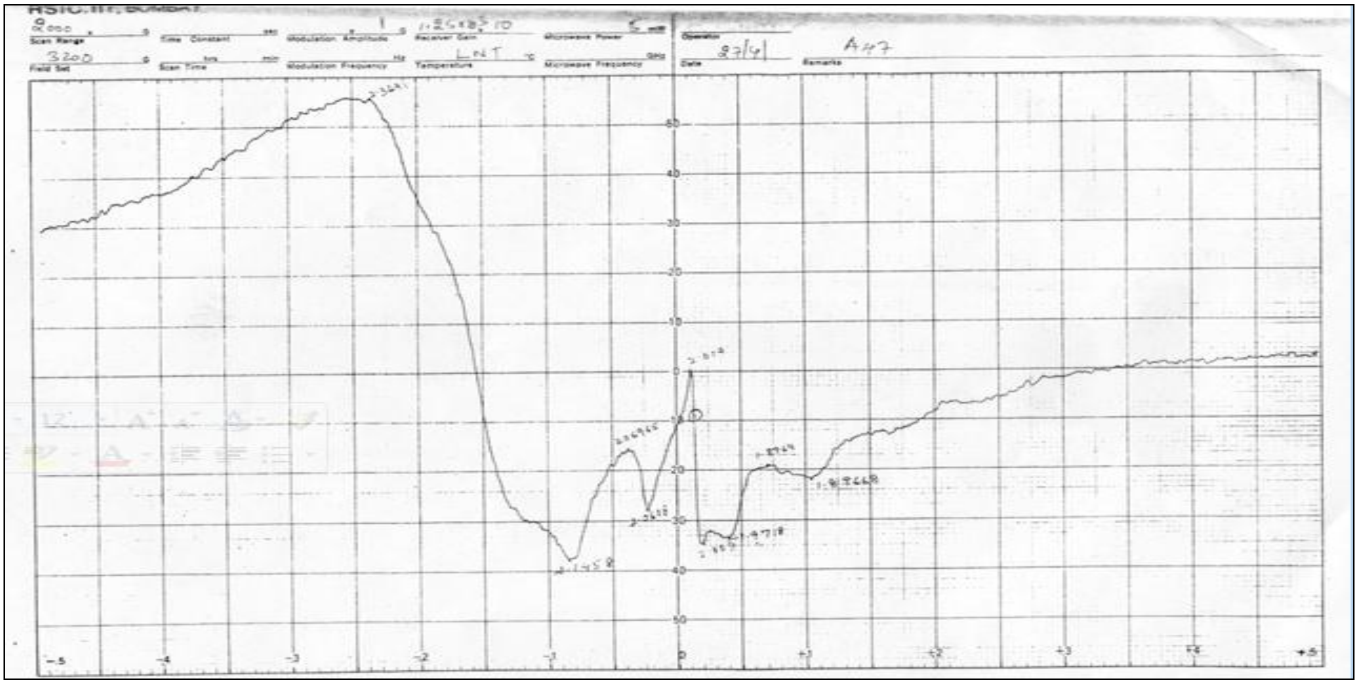


**FIG 3a: ESR SPECTRUM OF [Co (DDP) $(SO_4)_2$] COMPLEX**

Thus, on the basis of magnetic susceptibility measurement, molar conductance measureent, IR, electronic and EPR spectral studies and the subsequent discussion for the complexes given above, the following structure may be proposed for the 6-coordinate complexes **(Fig. 4a-4c)** while for the 4-coordinate complexes the structure are proposed as **(Fig. 4d)**.

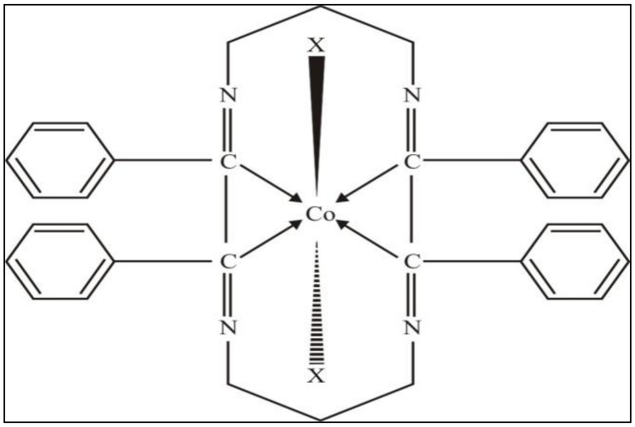


**FIG. 4a: [Co(BDP)$X_2$]**

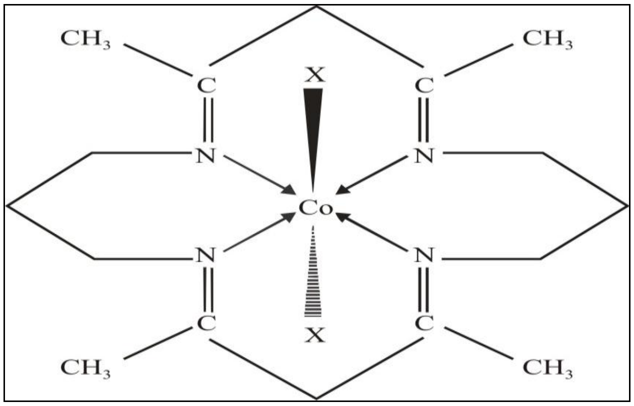


**FIG. 4b [Co(ADP)$X_2$]**

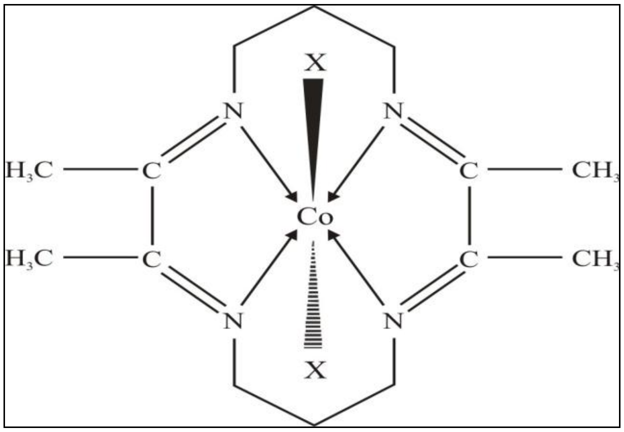


**FIG. 4c [Co(DDP)$X_2$]**

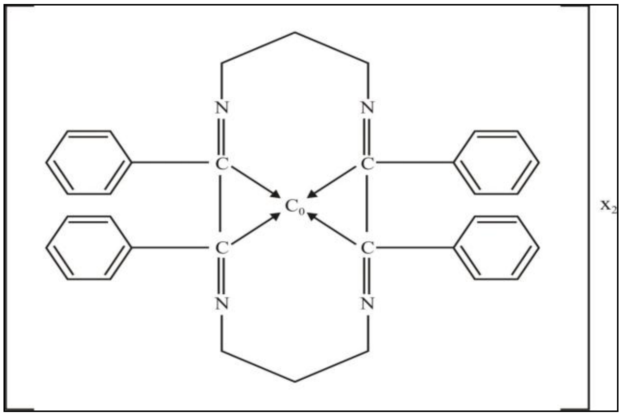


**FIG. 4d [Co(BDP)]$SO_4$**

**Biological Study:**

The antimicrobial screeniung data show that the metal chelates exhibit a higher inibitory effect than the free ligand and metal salts. The increased activity of the metal chelates can be explained based on the chelation theory. The chelation reduces the polarity of the metal atom mainly because of the partial sharing of its positive charge with the donor groups and possible p electron delocalization within the whole chelation ring.

The chelation ring increases the lipophillic nature of the central atom which subsequently favours its permeation through the lipid layer of the cell memberane. The enhanced activity of the complexes can also be explained on the basis of their high solubility fineness of the particles, size of the metal ion and the presence of bulkier organic moieties. The mode of action may involve the formation of a hydrogen bond through the azomethane nitrogen atom with the active centers of the cell constituents, resulting in interference with the normal cell prosess. The variation in the effectiveness of different compounds against different organisms depends either on the impermeability of the cells of the microbes or difference in ribosomes of microbial cells. It has also been proposed that concentration plays a vital role in increasing the degree of inhibition; as the concentration increases, the activity increases [26-30]. The results of fungicidal screening **(Figure 3)** show that all the complexes are highly active as compared to free ligand and metal salt against all the fungal species.

**CONCLUSION:** The present study reveled six coordinated octahedral geometry for the Co(II) complexes. All the ligands act as a tetradentatc manner coordinating through four nitrogens of the azomethine groups in N N N N fashion moreover, the fungicial data reveal that the complexes were superior to the free ligand in the inhibition of the tested fungi it is proposed that concentration plays a vital role in increasing the degree of inhibition, the activity increased with increasing concentration of the complexes.

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