

**SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL STUDIES OF NI (II) COMPLEXES WITH SCHIFF BASE CO-LIGAND DERIVED FROM 5,6-DIAMINO-1,10-PHENANTHROLINE AND BENZENE-1,4-DICARBALDEHYDE**

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**ABSTRACT**

The Schiff base (PDB) was synthesized by refluxing ethanolic solution of 5,6-diamino-1,10-phenanthroline with Benzene-1,4-dicarbaldehyde for 3 hours. Similarly the complexes were synthesized by refluxing equimolar solution of PDB with [Ni(phen)<sub>2</sub>Cl<sub>2</sub>] and [Ni(bpy)<sub>2</sub>Cl<sub>2</sub>] for 5 hours each and characterized by FTIR, UV-visible, Mass and Elemental Analysis. The absorption band in IR spectrum of PDB at 1593 cm<sup>-1</sup> was assigned to C=N stretching frequency which was shifted to 1550 cm<sup>-1</sup> and 1549 cm<sup>-1</sup> in the spectra of [Ni(phen)<sub>2</sub>PDB](PF<sub>6</sub>)<sub>2</sub> and [Ni(bpy)<sub>2</sub>PDB](PF<sub>6</sub>)<sub>2</sub> respectively. The new absorption bands at 721 cm<sup>-1</sup> and 754 cm<sup>-1</sup> in the spectra of both complexes were due to M-N coordinate bond. The UV- visible absorption band at 390 nm was due to π→π\* transitions for azomethine. The low energy band at 450 nm for both complexes is due to MLCT Ni(dπ) →PDB(π\*) transition. The molecular ion peak (M)<sup>+</sup> in the mass spectra for PDB and the complexes were observed at 443 m/z, 984.75 m/z and 940.95 m/z respectively. The compounds were evaluated for antimicrobial activities, by well diffusion method against bacteria using agar nutrient as the medium and fungi using potato dextrose agar as medium. The free Schiff base has low activity compared to the complexes with inhibition zone between 4.0 mm to 6.6 mm whereas the complexes ranges from 7.5 mm to 11.2 mm.

**Keywords:** 5,6-diamino-1,10-phenanthroline, Antimicrobial activities, Nickel (II) complexes and Schiff base.

**INTRODUCTION**

Schiff bases are compounds containing imine or azomethine (C=N-) functional group. Which are produced through the condensation reaction of primary amines with carbonyl-

containing compounds (Fig.1) and they were firstly reported by Hugo Schiff (Khudheyer and Murad, 2018).

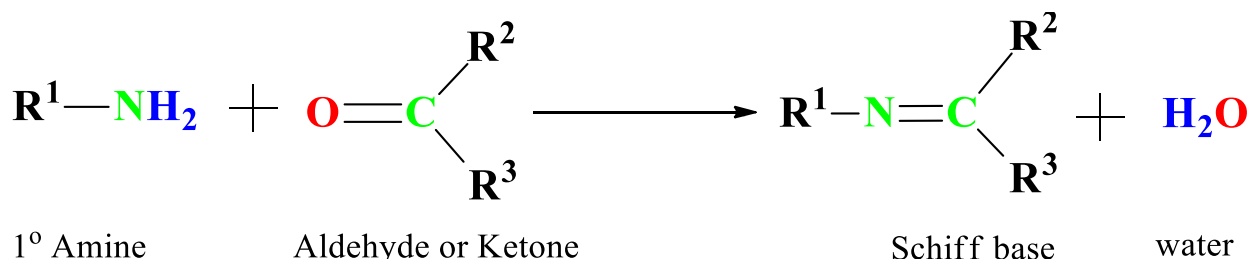


Fig. 1 - General scheme for formation of Schiff bases.

Schiff bases are considered as privileged ligands, because of their ability to form complexes with a wide range of metal ions resulting in the formation of stable complexes (Ajlouni *et al*, 2018). Schiff bases bearing aryl or heterocyclic groups have also been shown to exhibit a broad range of biological activities, including, antibacterial and antifungal properties (Lashanizadegan and Jamshidbeigi, 2011).

Transition metals have initiated the development of metal based drugs with promising pharmacological applications and offer unique therapeutic opportunities. Research has shown significant progress in utilization of transition metal complexes

as drugs to treat several human diseases like carcinomas, lymphomas, infections control, anti-inflammatory and neurological disorders. The recognition of Schiff base complexes as models for biologically active compounds has brought rapid advancement within the field of coordination and bio-inorganic chemistry and spawned extensive research on their synthesis and applications (Chohan and Sheazi, 1999). Metal complexes containing diimine ligands such as 1,10-phenanthroline and its derivatives have gained importance because of their versatile roles as building blocks for the synthesis of metallo-dendrimers and as molecular scaffolding for supramolecular assemblies, and in analytical chemistry, catalysis, electrochemistry, ring-opening metathesis polymerization and biochemistry,

(Mesut *et al.*, 2013)

Mesut *et al.*, 2013, reported the synthesis and characterization of new Co(II), Ni(II) and Cu(II) complexes with 1,10-Phenanthroline imidazole derivative, which is 2-p-tolyl-1H-imidazo[4,5-f][1,10] phenanthroline (L). Those compounds were screened for antibacterial activity against these bacterial strains; *A. hydrophila*, *S. aureus*, *K. pneumoniae*, *P. aeruginosa*, *S. marcescens*, *E. aerogenes*, *B. subtilis*, *E. coli* and *E. faecalis*. This research is aimed at the synthesis and characterization of Nickel (II) complexes derived from  $[\text{Ni}(\text{phen})_2\text{Cl}_2] \cdot 2\text{H}_2\text{O}$  and  $[\text{Ni}(\text{bpy})_2\text{Cl}_2] \cdot 2\text{H}_2\text{O}$  and to evaluate their antibacterial and antifungal activities. The proposed targets may result in the development of drugs with increased cytotoxicity compared to commercially available drugs.

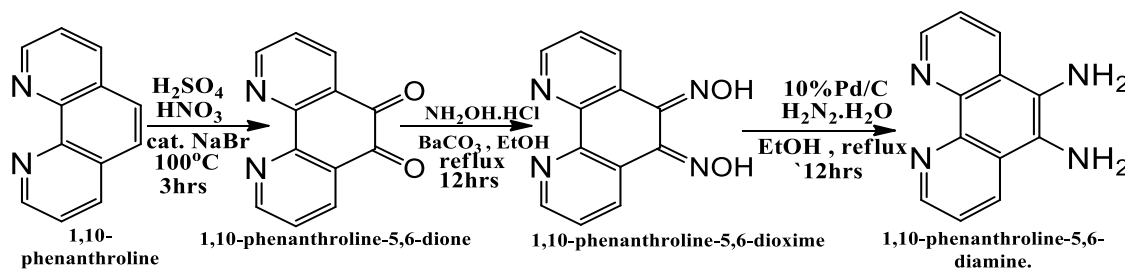
## EXPERIMENTAL

### MATERIALS AND METHODS

All chemicals were obtained from Sigma-Aldrich and used without purification. Tetrabutyl ammonium chloride (TBACl) and palladium on activated charcoal 10%Pd/C were purchased from E. Merck (India). All the reactions were monitored by checking TLC of the reaction mixture. The complexes were purified by column chromatography. The ligand and the complexes were characterized by standard analytical techniques (FT-IR, Mass, UV-visible spectroscopy and Elemental analysis).

### Preparation of the starting materials

The following precursor molecules that are necessary for the synthesis of new Schiff base used in this study have been prepared by adopting the published procedures.

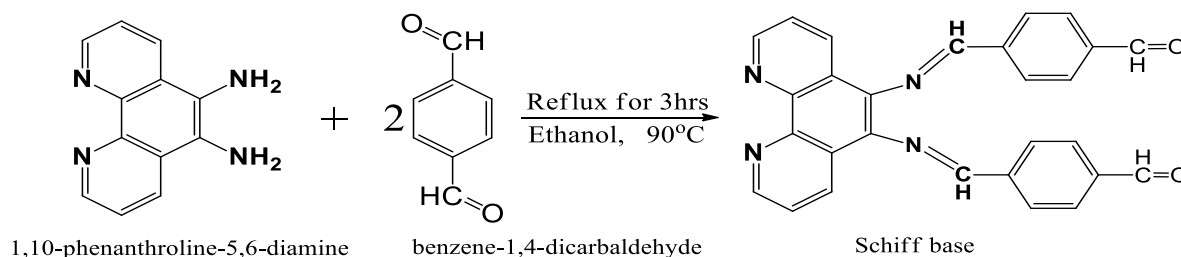


Scheme 1: Synthesis of 5,6-diamino-1,10-phenanthroline. Yamada *et al.*, (1992); Bodige and MacDonnell (1997)

### Synthesis of Schiff base ligand (PDB)

The Schiff base (PDB) was synthesized by adding Benzene-1,4-dicarbaldehyde (2 mmol) in 20 ml of ethanol to ethanolic solution of 5,6-diamino-1,10-phenanthroline (1 mmol). The mixture was refluxed for 3 hours. Then solution of the ligand

was kept for slow evaporation and coloured precipitate was collected and dried over  $\text{CaCl}_2$  for 2 days in desiccator. Yield: (78%). Anal. Calc. for  $\text{C}_{28}\text{H}_{18}\text{N}_4\text{O}_2$ : C, 76.02; H, 4.07; N, 12.67. Found: C, 76.10; H, 4.04; N, 12.46; FAB-MS (m/z): 443 ( $\text{M}^+$ ); UV-Vis., (nm);  $\text{CH}_3\text{CN} + \text{MeOH}$  (9:1): 246, 276, 390.



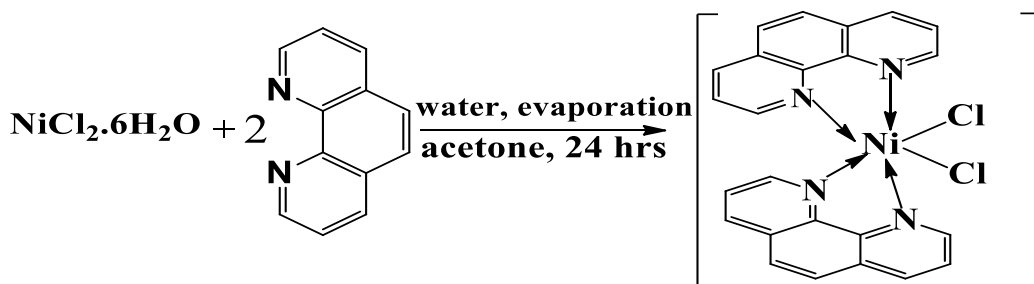
Scheme 2: Synthesis of Schiff base

### Preparation of Precursor Complexes

The following precursor complexes that are necessary for the synthesis of new complexes in this study have been prepared by adopting published procedures.

### Preparation of Bis (1,10-phenanthroline) dichloronickel (II)

$[\text{Ni}(\text{phen})_2\text{Cl}_2] \cdot \text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (1 mmol) and 1,10-phenanthroline (2 mmol) were dissolved in 20 ml water and the solution was evaporated to 5 ml. Acetone (20 ml) was added to this lilac solution which was then set aside in a closed vessel for 24 hrs. The crystals were filtered off, washed with acetone and dried under vacuum. Yield: ~ 65%. Haris and McKenzie (1967).

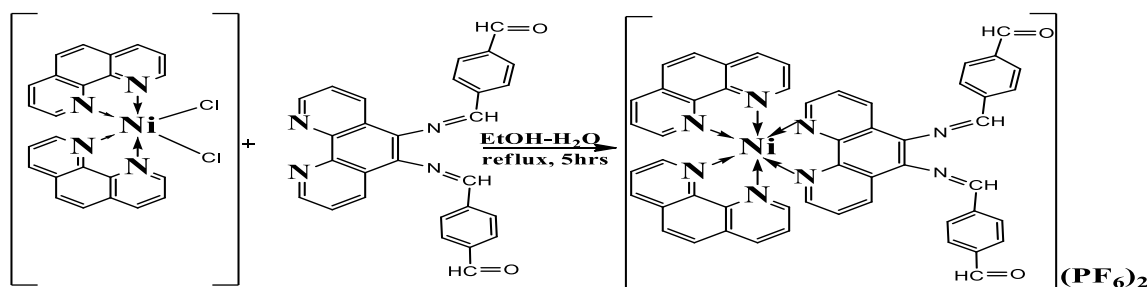


Similarly  $[\text{Ni}(\text{bpy})_2\text{Cl}_2]$  was prepared by adopting the same procedure reported by Haris and McKenzie

#### Synthesis of bis(1,10-phenanthroline)(PDB)nickel(II) hexafluorophosphate, $[\text{Ni}(\text{Phen})_2(\text{PDB})](\text{PF}_6)_2$

The complex was synthesized by refluxing equimolar solution of PDB and  $[\text{Ni}(\text{phen})_2\text{Cl}_2]$  in  $\text{C}_2\text{H}_5\text{OH}-\text{H}_2\text{O}$  (2:1, v/v; 225 ml) mixture for 5 hours. The red coloured crude complex was obtained on adding saturated solution of  $\text{NH}_4\text{PF}_6$ . It was purified by column chromatography (alumina,  $\text{CH}_3\text{CN}$  – Toluene (3:2, v/v) mixture) and was further recrystallized from

acetone-ether mixture (1:5, v/v). Yield = 0.67 g (71%). The chloride salt of  $[\text{Ni}(\text{phen})_2(\text{pdb})]^{2+}$  was obtained by dissolving the above hexafluorophosphate complex in minimum amount of acetone and precipitated out upon addition of a saturated solution of TBACl. Analytical data: Anal. Calc. for  $\text{C}_{52}\text{H}_{34}\text{N}_8\text{O}_2\text{NiCl}_2$ : C, 63.40; H, 3.45; N, 11.37. Found: C, 64.01; H, 3.42; N, 11.00. MS (FAB)  $m/z$  Calc.  $[\text{M}]^+$ , 984.75; Found:  $[\text{M}]^+$ , 984.05.



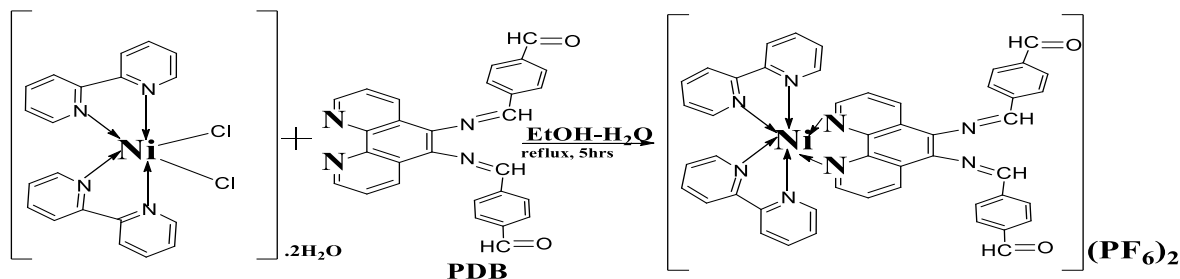
Scheme 5: Synthesis of  $[\text{Ni}(\text{phen})_2(\text{PDB})](\text{PF}_6)_2$

#### Synthesis of bis (2,2-bipyridine) (PDB) nickel (II) hexafluorophosphate, $[\text{Ni}(\text{bpy})_2(\text{PDB})](\text{PF}_6)_2$

This complex was synthesized by refluxing equimolar solution of PDB with  $[\text{Ni}(\text{bpy})_2\text{Cl}_2]$  in  $\text{C}_2\text{H}_5\text{OH} - \text{H}_2\text{O}$  (2:1, v/v; 225 ml) mixture for 5 hours. The orange crude complex was obtained on adding saturated solution of  $\text{NH}_4\text{PF}_6$ . It was purified by column chromatography (alumina,  $\text{CH}_3\text{CN}$  – Toluene (3:2, v/v) mixture) and was further recrystallized from acetone-ether

mixture (1:5, v/v). Yield = 0.58 g (69%). The chloride salt of  $[\text{Ni}(\text{phen})_2(\text{pdb})]^{2+}$  was obtained by dissolving the above hexafluorophosphate complex in minimum amount of acetone and precipitated out upon addition of a saturated solution of TBACl.

Analytical data: Anal. Calc. for  $\text{C}_{48}\text{H}_{24}\text{N}_8\text{O}_2\text{NiCl}_2$ : C, 61.20; H, 2.55; N, 11.90. Found: C, 61.81; H, 2.42; N, 12.07. MS (FAB)  $m/z$  Calc.  $[\text{M}]^+$ , 941; Found:  $[\text{M}]^+$ , 940.95.



Scheme 6: Synthesis of  $[\text{Ni}(\text{bpy})_2(\text{PDB})](\text{PF}_6)_2$

#### Molar Conductivity Measurements

The molar conductance of the complexes was measured at room temperature in DMF using  $0.001 \text{ molL}^{-1}$  solution of both complexes.

#### Antimicrobial activity

The ligand PDB and its Ni(II) complexes were evaluated for antimicrobial activity by the well diffusion method against the bacteria *Salmonella typhi*, *Pseudomonas aeruginosa*,

*Escherichia coli* and *Staphylococcus aureus* using agar nutrient as the medium and antifungal activities against the fungi *Aspergillus niger*, *Aspergillus flavus* and *Rhizoctonia bataicola* cultured on potato dextrose agar as medium. The stock solution ( $10^{-3}$  molL $^{-1}$ ) was prepared by dissolving the compounds in DMSO. The antimicrobial activities were performed in triplicate and the average was taken as the final reading. The well was made on the agar medium inoculated with microorganisms and filled with the test solution. The plate was incubated for 24 hours for bacteria and 72 hours for fungi at 35 °C. During this period,

the test solution was diffused and the growths of the inoculated microorganisms were affected. The inhibition zone was developed and it was measured in mm. Zone of inhibition of the investigated compounds against the bacteria and fungi are summarized in Table 4 and 5. Streptomycin and Ketoconazole were used as standard reference compounds for antibacterial and antifungal studies respectively (Sheikh *et al.*, 2004).

## RESULT AND DISCUSSION

**Table 1: Percentage yield and some physical properties of the Schiff base and Ni(II) complexes**

Properties	(PDB) Ligand	[Ni(phen) <sub>2</sub> PDB]Cl <sub>2</sub>	[Ni(bpy) <sub>2</sub> PDB]Cl <sub>2</sub>
% yield	78%	71%	69%
Colour	greenish-yellow	Red	Orange
Appearance	Powder	Crystalline powder	Crystalline powder
Melting point	>350 °C	-	-
Decomposition temperature	-	225 °C	242 °C
Molar conductivity	-	97 Ω <sup>-1</sup> cm <sup>2</sup> mol <sup>-1</sup>	89 Ω <sup>-1</sup> cm <sup>2</sup> mol <sup>-1</sup>

The Schiff base has a percentage yield of 78%, whereas the two red and orange crystalline powdered complexes were obtained in appreciable yield of 71% and 69% respectively. Both two complexes [Ni(phen)<sub>2</sub>PDB]Cl<sub>2</sub> and [Ni(bpy)<sub>2</sub>PDB]Cl<sub>2</sub> are electrolytic in nature having molar conductance values of 97 Ω<sup>-1</sup>cm<sup>2</sup>mol<sup>-1</sup> and 89 Ω<sup>-1</sup>cm<sup>2</sup>mol<sup>-1</sup> respectively, which are in good agreement with similar complexes reported in literature.

**Table 2: Infrared spectroscopic data for the Schiff base and Ni(II) complexes**

Compounds	IR data(cm <sup>-1</sup> )				
	$\nu$ (C=N)	$\nu$ (C=O)	$\nu$ (C=C) <sub>aro</sub>	$\nu$ (C-H <sub>ben</sub> ) <sub>aro</sub>	$\nu$ (M-N)
PDB (Schiff base)	1593	1642	1557	992	-
[Ni(phen) <sub>2</sub> PDB](PF <sub>6</sub> ) <sub>2</sub>	1550	1629	1535	842	721
[Ni(bpy) <sub>2</sub> PDB](PF <sub>6</sub> ) <sub>2</sub>	1549	1630	1533	843	754

The absorption bands at 1593 cm<sup>-1</sup>, 1550 cm<sup>-1</sup> and 1549 cm<sup>-1</sup> on the spectra of PDB ligand [Ni(phen)<sub>2</sub>PDB](PF<sub>6</sub>)<sub>2</sub> and [Ni(bpy)<sub>2</sub>PDB](PF<sub>6</sub>)<sub>2</sub> respectively are attributed to C=N stretching frequencies. Similarly, the absorption bands at 1642 cm<sup>-1</sup>, 1629 cm<sup>-1</sup> and 1630 cm<sup>-1</sup> are assigned to C=O. The absorption bands at 992 cm<sup>-1</sup>, 842 cm<sup>-1</sup> and 843 cm<sup>-1</sup> in the

spectra are given to (C-H) (aromatic) bending vibrational frequencies. Whereas the new absorption bands at 721 cm<sup>-1</sup> and 754 cm<sup>-1</sup> in the spectra of both complexes are assigned to M-N coordinate bond as reported by Arounaguiria *et al.*, (2000).

**Table 3: UV-Visible and Mass spectral data for the Schiff base and Ni(II) Complexes**

Compounds	UV-visible $\lambda$ .max (nm) (log $\epsilon$ )	Mass
PDB (Schiff base)	390 (3.0), 246 (3.1), 276 (3.3)	443 (M) <sup>+</sup> , base peak 211
[Ni(phen) <sub>2</sub> PDB](PF <sub>6</sub> ) <sub>2</sub>	450 (2.7), 280 (3.3)	984.05 (M) <sup>+</sup> , base-peak 317.50
[Ni(bpy) <sub>2</sub> PDB](PF <sub>6</sub> ) <sub>2</sub>	450 (2.6), 275 (3.3)	940.95 (M) <sup>+</sup> , base-peak 341.65

\*Spectra were measured in acetonitrile

low energy absorption band due to  $\pi \rightarrow \pi^*$  transitions at 390

### Absorption spectra for PDB and Ni(II) Complexes

As seen, the spectra of PDB is characterized by low intensity,

nm. This band is assigned to the azomethine chromophore

attached to the phenanthroline moiety. The high-energy bands (PDB: 246 nm and 276 nm) are attributed to the  $\pi \rightarrow \pi^*$  transitions corresponding to the phenanthroline moiety of the ligand. The low energy band at 450 nm for both complexes is due to MLCT Ni(d $\pi$ )  $\rightarrow$  PDB( $\pi^*$ ) transition. The band centered at 280 nm and 275 nm for [Ni(phen)<sub>2</sub>(PDB)](PF<sub>6</sub>)<sub>2</sub> and [Ni(bpy)<sub>2</sub>(PDB)](PF<sub>6</sub>)<sub>2</sub> are attributed to intra-ligand  $\pi \rightarrow \pi^*$  transitions. All the electronic transitions were found to be similar to those reported by Suma *et al.*, (2012).

#### Mass Spectra for Schiff base and Ni(II) Complexes

The mass spectrum of the Schiff base showed its molecular ion peak at 443 m/z also a base-peak at 211 m/z is observed due to C<sub>12</sub>H<sub>6</sub>N<sub>4</sub>(M - C<sub>14</sub>H<sub>10</sub>O<sub>2</sub>). In the case of corresponding mixed-ligand Ni(II) complexes the molecular ion peak for the complex([Ni(phen)<sub>2</sub>(PDB)]PF<sub>6</sub>)<sup>+</sup> were seen at 984.75 m/z (M)<sup>+</sup>. Similarly the molecular ion peak for the complex ([Ni(bpy)<sub>2</sub>(PDB)]PF<sub>6</sub>)<sup>+</sup> was obtained at 940.95 m/z (M)<sup>+</sup> which are in good agreement with the complexes obtained and reported by Suma *et al.*, (2012).

**Table 4. Antibacterial activity of PDB ligand and its Ni(II) complexes**

Compounds	Zone of inhibition (mm)			
	<i>S. typhi</i>	<i>S. aureus</i>	<i>E. coli</i>	<i>P. aeruginosa</i>
PDB	5.0	5.6	6.2	6.6
[Ni(phen) <sub>2</sub> PDB]Cl <sub>2</sub>	7.0	8.5	12	11.0
[Ni(bpy) <sub>2</sub> PDB]Cl <sub>2</sub>	8.7	10.6	9.7	11.2
Streptomycin	13.0	16.0	15.0	15.0

**Table 5. Antifungal activity of PDB ligand and its Ni(II) complexes**

Compounds	Zone of inhibition (mm)		
	<i>Aspergillus niger</i>	<i>Aspergillus flavus</i>	<i>Rhizoctonia bataicola</i>
PDB	4.0	4.1	5.2
[Ni(phen) <sub>2</sub> PDB]Cl <sub>2</sub>	8.0	7.5	10
[Ni(bpy) <sub>2</sub> PDB]Cl <sub>2</sub>	7.5	8.8	9.0
Ketoconazole	12.0	11.0	14.0

\*The test was done using 10<sup>-3</sup>M concentration of Ni(II) complexes by well diffusion technique. The values are mean of three replications.

The complex [Ni(phen)<sub>2</sub>PDB]Cl<sub>2</sub> show more activity against *E. coli*, whereas [Ni(bpy)<sub>2</sub>PDB]Cl<sub>2</sub> has higher activity against *P. Aeruginosa* and both two complexes show good activities against *Rhizoctonia bataicola*. Generally, the results indicate that the complexes are more potent when compared to the ligand but, they have lower activity than the control as reported by Sheikh *et al.*, 2004.

#### CONCLUSION

We have successfully synthesized novel Ni(II) complexes with coordinating Schiff base which were characterized by elemental analysis, Infrared spectroscopy, UV-visible and mass spectral analysis. The data of molar conductance values in dimethyl sulphoxide (DMSO) solutions shows the electrolytic behavior of both complexes in 1:2 ratios suggesting the

presence of chloride ions in the outer sphere of complex structures. Octahedral geometry for [Ni(phen)<sub>2</sub>PDB](Cl)<sub>2</sub> and [Ni(bpy)<sub>2</sub>PDB](Cl)<sub>2</sub> were tentatively proposed, based on the data obtained from molar conductivity, elemental and spectral analyses. The antimicrobial activities of the ligand and the complexes were screened against four bacterial and three fungal species, both complexes showed higher activity against microbes when compared to the free Schiff base, hence the obtained inhibition zones data indicate the possibility of their applications in the treatment of diseases.

#### ACKNOWLEDGEMENT

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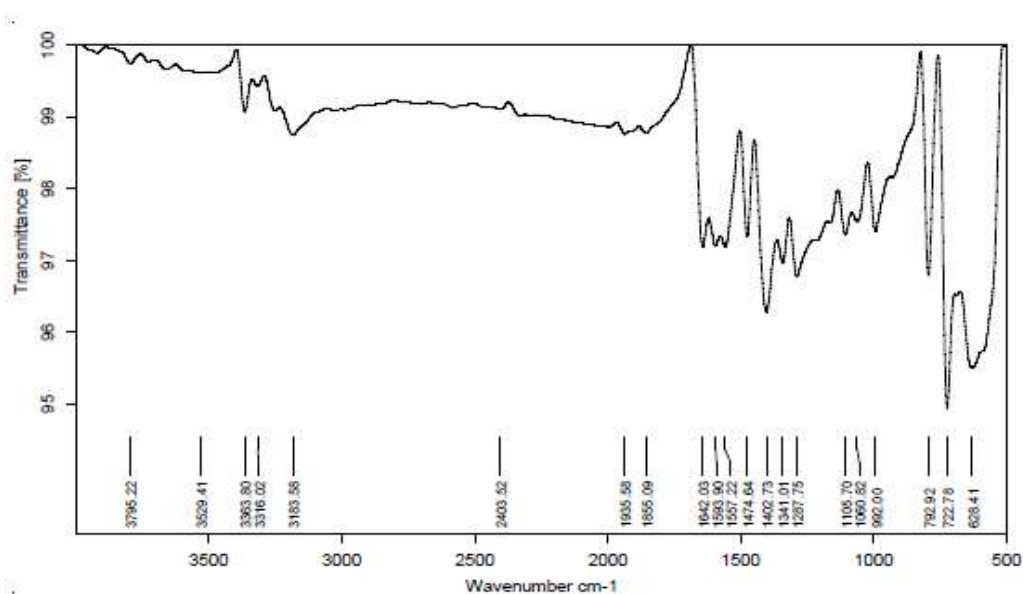


Fig.1. Infrared Spectrum of the Schiff Base (PDB)

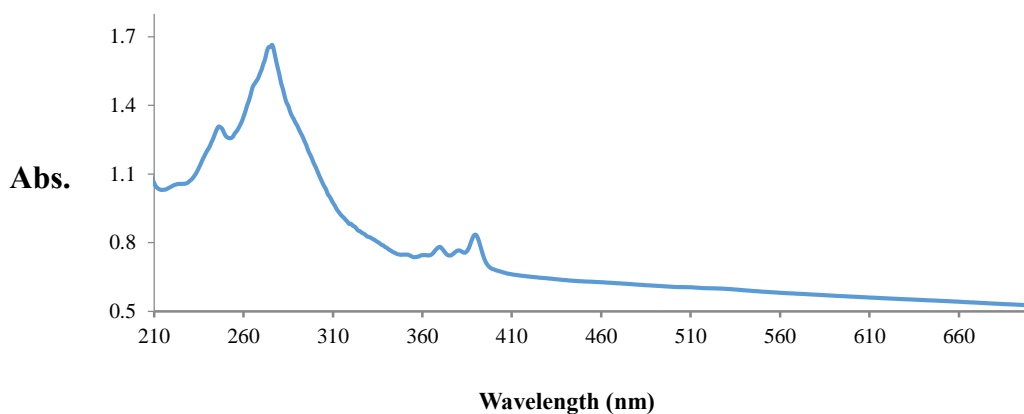


Fig.2. UV-Vis Spectrum of the Schiff Base (PDB)

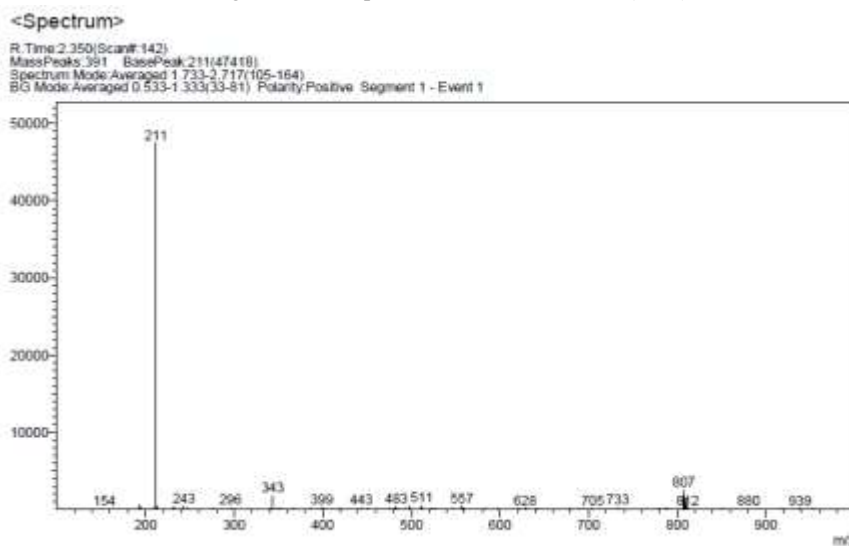
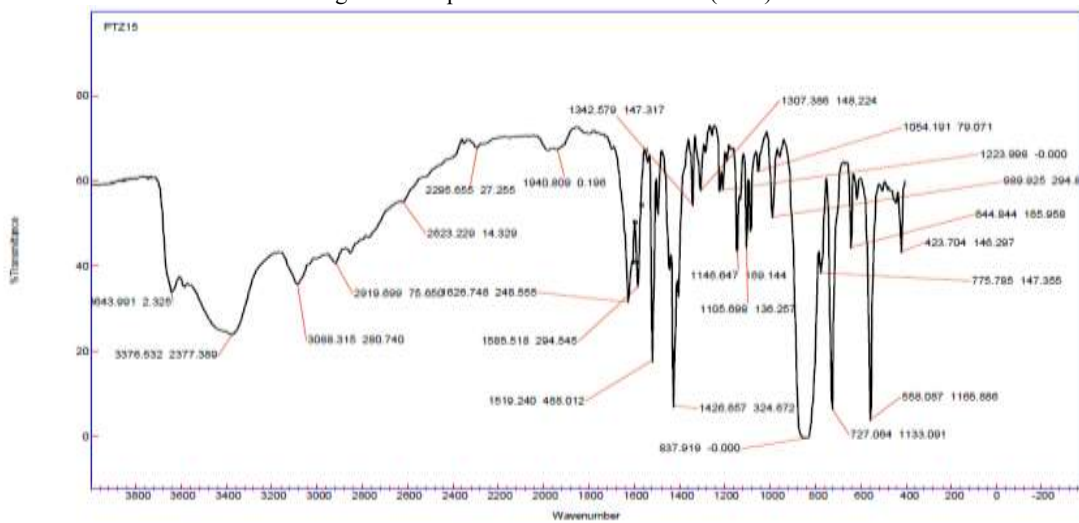
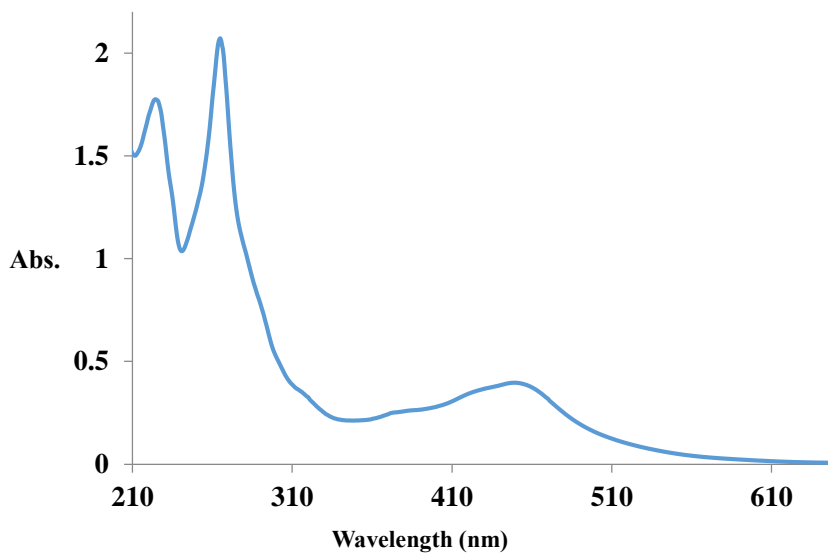


Fig.3. Mass Spectrum of the Schiff Base (PDB)

Fig. 4. Infrared Spectrum of  $[\text{Ni}(\text{phen})_2\text{PDB}](\text{PF}_6)_2$ Fig. 5. UV-Vis Spectrum of  $[\text{Ni}(\text{phen})_2\text{PDB}](\text{PF}_6)_2$

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 Sample ID : PTZ-7  
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 Vial# : 1  
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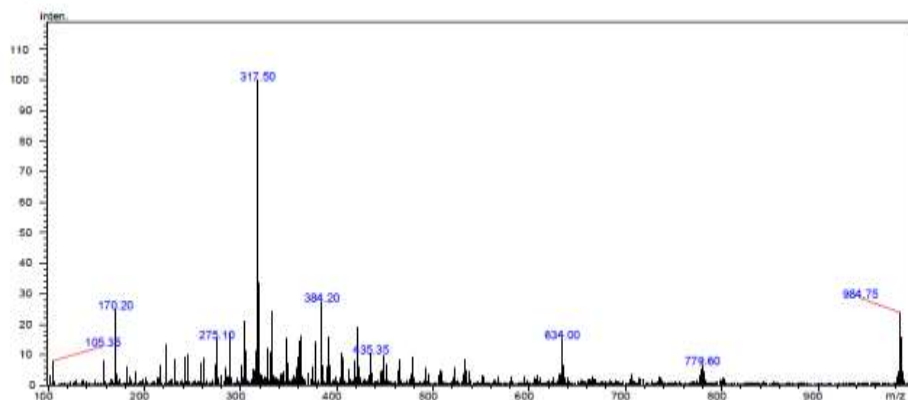
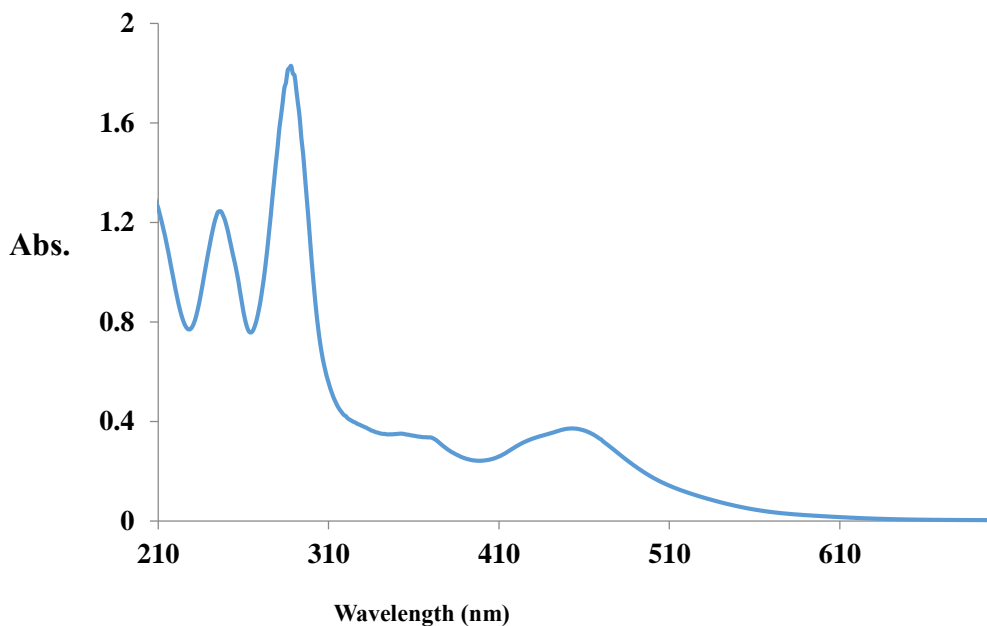


Fig. 6. Mass Spectrum of  $[\text{Ni}(\text{phen})_2\text{PDB}](\text{PF}_6)_2$

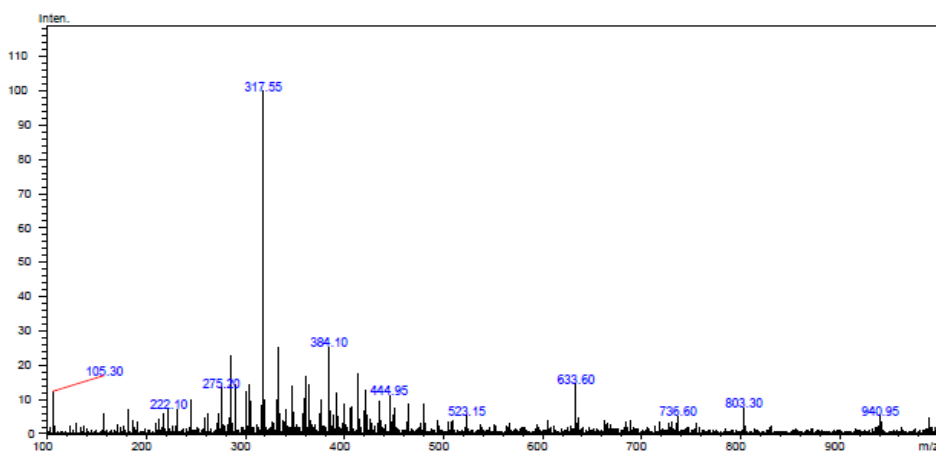


Fig. 7. Infrared Spectrum of  $[\text{Ni}(\text{bpy})_2\text{PDB}](\text{PF}_6)_2$



Fig. 8. UV-Vis spectrum of  $[\text{Ni}(\text{bpy})_2(\text{PDB})](\text{PF}_6)_2$ 

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 Sample ID : PTZ-8  
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Fig. 9. Mass spectrum of  $[\text{Ni}(\text{bpy})_2\text{PDB}](\text{PF}_6)_2$ 

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