

SYNTHESIS, SPECTRAL AND XRD STUDIES OF SCHIFF-BASE LIGAND AND A SERIES OF 1,10-PHENANTHROLINE TRANSITION METAL COMPLEXES

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Abstract : 1,10-phenanthroline transition metal complexes of the type $[M(\text{phen})_2(L)](\text{OAc})_2 \cdot n\text{H}_2\text{O}$ containing Schiff base ligands derived from 2-chloro ethyl amine and 2-hydroxy-3,5-diiodo benzaldehyde were synthesized and characterized by elemental analysis, Powder XRD, UV-vis, IR, ¹H NMR, spectral methods. Elemental analysis data revealed that the complexes have a 1:2:1 molar ratio among the metal and ligands. The IR and ¹H NMR, spectral data confirmed that the ligands coordinate with the metal ions in a bidentate fashion through azomethine nitrogen and phenolic oxygen to form complexes. Powder XRD shows the sharp crystalline peaks indicating the crystalline state of the complexes.

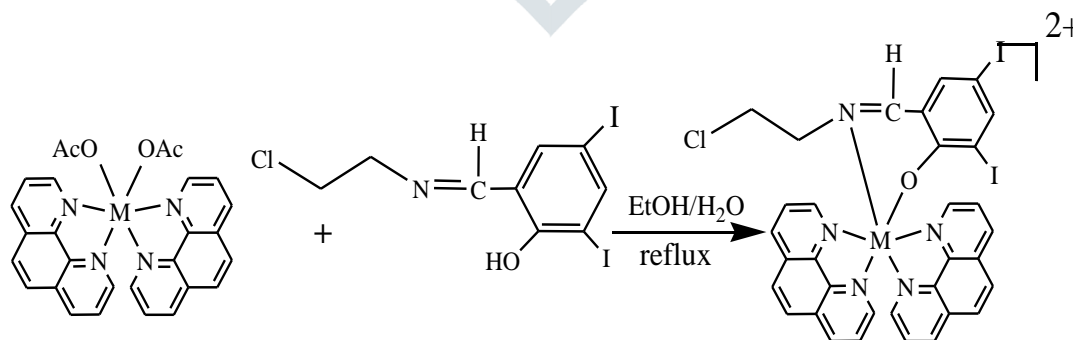
Keywords : 1,10 phenanthroline transition metal complexes, 2-chloro ethyl amine,

I. INTRODUCTION

1, 10-phenanthroline and 2, 2-bipyridine transition metal complexes of Schiff bases play an important role in coordination chemistry for new therapeutic agents, various crystallographic features, catalytic activity, photochromic properties and in petroleum refining.¹⁻³ A large number of Schiff bases and their complexes have been investigated for great details in various crystallographic, structural and magnetic features also interaction of these complexes with DNA. Transition metal Schiff base complexes are used in various fields, such as medicine, agriculture industries involved in oxygen metabolism. They have been extensively studied in great details for analytical, physical, and biochemical purposes. The present work deals with the synthesis of 1,10-phenanthroline transition metal complexes of Schiff base ligand derived from 2-chloro ethyl amine. They were characterized using analytical and various spectral techniques.

II RESEARCH METHODOLOGY

Synthesis of complexes: The complexes were prepared by refluxing a solution of $[M(\text{phen})_2](\text{OAc})_2 \cdot n\text{H}_2\text{O}$ and ligands (1 mmols) in aqueous ethanol (20 ml) for 4h. The solid obtained were filtered, washed with ethanol and then dried as shown in (Scheme 1)⁴.



Scheme 1. Synthesis of complex

RESULTS AND DISCUSSION

Elemental Analysis:

Elemental analysis data confirmed that the complexes have a 1:2:1 molar ratio between the metal and ligands. i.e. one mole of metal acetate reacted with two moles of 1,10-phenanthroline and one mole of ligands to give the corresponding complexes. All the complexes show the analytical results close to the theoretical values indicating the presence of two types of ligands.

IR Spectra:

The IR spectra of free Schiff base ligand showed the band at 1360 cm^{-1} attributed to the phenolic C–O stretching vibrations of the free ligands was shifted to 1426 cm^{-1} upon complexation. The broad band at 3400 cm^{-1} were due to stretching vibrations of phenolic OH. This bands was absent in the complexes, indicating deprotonation on coordination. The imine (C=N) functional group of the free ligands was observed at 1660 cm^{-1} was shifted to 1620 cm^{-1} in the spectra of the complexes, indicating coordination of azomethine nitrogen of the Schiff base ligands to metal ion⁵. The mode of coordination of the Schiff base ligands was supported by the appearance of two new weak bands in the lower frequency region at 565 cm^{-1} and 425 cm^{-1} . These bands were assigned to the M–N and M–O stretching vibrations.

Electronic Spectra:

The electronic spectra of the ligand and their metal complexes were carried out in DMSO. The absorption band observed around 270–350 nm range respectively were assigned to $\pi\text{-}\pi^*$ and $n\text{-}\pi^*$ transitions⁶. The bands observed at 350 nm was assigned to the $\pi\text{-}\pi^*$ transitions of the azomethine, which were shifted to 425 nm in the electronic spectra of complex⁷. The bands observed at 270 nm were assigned to the $\pi\text{-}\pi^*$ transitions of the phenol indicating the schiff base ligand is coordinating via phenolic O and the azomethine N.

Powder XRD:

Powder XRD patterns of complexes show the sharp crystalline peaks indicating their crystalline phase^{8,9}. The diffraction pattern of complexes is measured in the range ($2\theta = 0\text{-}80^\circ$) are shown in (Figure 1).

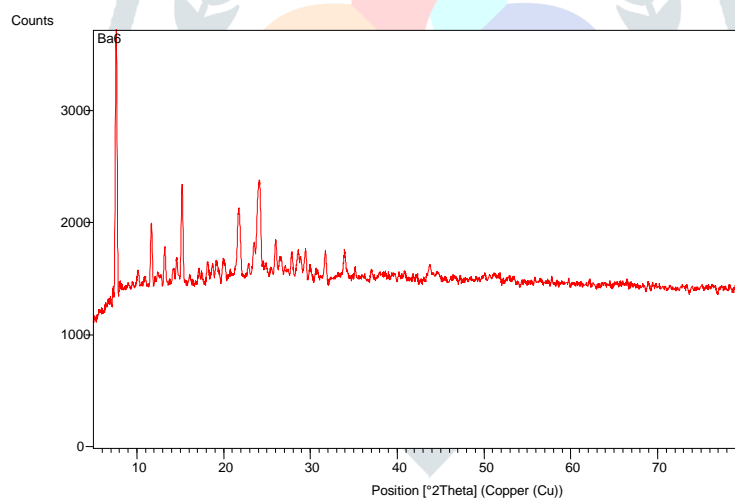


Figure 1. Powder XRD pattern of complex.

III.CONCLUSIONS:

The transition metal complexes of the type $[M(\text{phen})_2(L)](\text{OAc})_2 \cdot (n\text{H}_2\text{O})$ containing Schiff base ligands derived from 2-chloro ethyl amine and 2-hydroxy-3,5-diiodo benzaldehyde were synthesized and characterized. Based on the above observations of the elemental analysis, UV-Vis., IR, $^1\text{H-NMR}$ spectral data and Powder XRD it is possible to determine the type of coordination of the ligands in their complexes. Powder XRD indicates the crystalline state of the complexes.

REFERENCES

1. H. Naeimi, M. Moradian, *J. Coord. Chem.*, **63**, 156, (2010).
2. R. K. Dubey, U. K. Dubey, S. K. Mishra, *J. Coord. Chem.*, **64**, 2292, (2011)..
3. D. Gardenghi, *Inorg. Chem.*, **515**, 1, (2007).
4. B. P. Sullivan, D. J. Salmon, T. J. Meyer, *Inorg. Chem.*, **17**, 3334, (1978).
5. S. Khaled, Z. A. Mohammed, G. K. Firdous, K. A. Shaikh, *Int. J. Inorg. Chem.*, 212435, (2013).
6. M. Siwy, D. Sek, B. Kaczmarczyk, J. Wietrzyk, A. Nasulewicz, Opolski, *AnticancerResearch*, **27**, 1553, (2007).
7. S. V. Amosovo, N. A. Makhaeva, A. V. Martinov, V. A. Potapov, B. A. Steele, I. D. Kostas, *Synthesis*, **10**, 1641, (2005).
8. Prasad, Surendra, *Trans. Met. Chem.*, **32**, 143, (2007).
9. D. Kannan, M. N. Arumugham, *Int. J. Inorg. and Bioinorg. Chem.*, **2**, 50, (2012).

