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An Unique Novel Ni(II) Octahedral Complex Derived from Potential Tetradentate Schiff Base (H_4L) Of N,N’-Bis-(2-Amino-Ethyl)-Ethane-1,2-Diamine and 1-(5-Chloro-2-Hydroxy-Phenyl)-Ethanone – Synthesis, Characterization and Magneto Structural Co-Relationship

Dhrubajyoti Majumdar*

Department of Chemistry, Tamralipta Mahavidyalaya Tamluk, Tamluk – 721 636, West Bengal, India.

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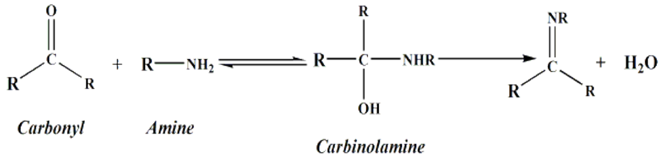
ABSTRACT

The work in this paper presents the detail syntheses, characterization and magneto structural co-relationship of one unique novel Ni(II) octahedral complex [$Ni(C_{22}H_{26}N_4O_2Cl_2)$] (1), derived from azo-linked potentially tetradentate compartmental Schiff base (H_4L). The compartmental tertadentate Schiff base (H_4L) was synthesized by normal 1:2 condensation reaction between N,N’-Bis-(2-amino-ethyl)-ethane-1,2-diamine and 1-(5-chloro-2-hydroxy-phenyl)-ethanone. The new mononuclear Ni(II) complex was characterized by different microanalytical methods like elemental analyses, FT-IR, ^1H NMR, UV-Vis spectra, mass spectra & molar conductance. Molar conductance study confirmed complex (1) is non-electrolyte in nature. Inside the cavity of compartmental Schiff base (H_4L), Ni(II) metal center was connected by bridging imine nitrogen (N_4 binding core) and deprotonating (phenolato oxygen), thus Schiff base(H_4L) embedded complete octahedral geometrical arrangement. Complex (1) does not exhibit any kind of hydrogen bonding and thus $\pi \rightarrow \pi^*$ interactions never lead to formation of supramolecular architectures. Finally in a comparative approach magnetic moment of respective Ni(II) complex have been investigated thoroughly with theoretical some Ni(II) octahedral complexes.

1. Introduction

Thanks to Prof. Sir Hugo Joseph Schiff [1] in 1864 year the beautiful discovery of novel Schiff base and thus opened an extensive research area in the field of coordination chemistry basically for inorganic chemists. After his novel discovery many inquisitive chemists got interested in the same field of research and till now research in the Schiff base coordination chemistry would continued without ending the page. According to Prof. Hugo Schiff, it is a compound that contains only azomethine functional group [2] attached to an aryl/alkyl having no hydrogen. Owing to its easily synthetic routes and product isolation it is also referred as ‘privileged ligands’ [3]. Generally the formation of Schiff base is a combination of elimination followed by addition according to Scheme 1 [4] which further reflects that during Schiff base formation C-N double bond formation common.



Scheme 1 Synthesis Schiff base

Schiff base have different donor centers like oxygen, nitrogen & sulphur hence acting as a well behaved potential chelating ligands. Due to this reason Schiff bases are capable to form vast number of mono, di & polynuclear chelate complexes with nitrogen or oxygen donor atom [5-7]. Salen-type tetradentate Schiff base complexes with N_2O_2 donors form ‘ligand complexes’ and thereby it prefer to combine with other metals through their O,O donors and acting as precursors for new polynuclear homo/hetero-metallic complexes leading to enrich coordination chemistry [8]. Thus Salen-type Schiff base constitute a unique class for their structural richness and wide range of applications e.g Ni(II) Salen-type N,O-donor Schiff base act as important models for enzyme like urease [9]. Schiff base with N,O-donor and ortho position blocked by –OMe/-OEt/-OH also exhibit wide range of photo & thermochromic properties in solution and solid state , thus it opens up a new gate of opportunity for photochemical applications of formed Schiff base complexes [10-11]. Moreover Salen-type dinuclear transition metal complexes are very significant in magneto-chemistry to understand the nature of exchange interactions involved between metals present in the complex and ligand. In the current research scenario, Schiff base metal complexes (mono/di/polynuclear) have extensive applications, such as, in the treatment of cancer [12], as antibactericide agents [13-14], as antivirus agents [15-17], as fungicide agents [18-19], and in the field of common biological field [20-22]. Apart from other well behaved applications of Schiff base complexes are in chemical analysis [23], absorption and transport of oxygen, in pesticides [24-25], and homo and heterogeneous catalysis for oxidation and polymerization of organic compounds [26]. Potential Schiff base having imine nitrogen and phenolato oxygen are excellent in catalysis and biological replication owing to their strong coordinating binding ability with transition metal ions [27]. In this paper we communicate the synthesis, characterization by different microanalytical methods of one new mononuclear Ni(II) complex [$Ni(C_{22}H_{26}N_4O_2Cl_2)$] (1), derived from the compartmental Schiff base (H_4L), obtained by condensation of N,N’-Bis-(2-Amino-Ethyl)-Ethane-1,2-Diamine and 1-(5-chloro-2-hydroxy-phenyl)-Ethanone. Finally in a very lucid manner respective Ni(II) complex magnetic moment data have been compared with literature survey selected some Ni(II) octahedral complexes.

2. Experimental Methods

2.1 Materials

All chemicals were of reagent grade and used as received without further purification. All solvents like methanol or ethanol and other reagents used in this research work were obtained from commercial sources. 5-chloro-2-hydroxyacetophenone was purchased from Sigma Aldrich Company, USA. Triethylenetetramine, $NiCl_2.6H_2O$ both are

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*Corresponding Author
Email Address: dmajumdar30@gmail.com (Dhrubajyoti Majumdar)

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purchased from Merck chemical Company. All synthetic reactions and work-up were done in open air.

2.2 Physical Measurement

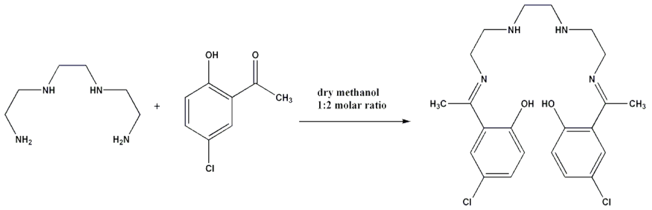
Elemental analyses (carbon, hydrogen and nitrogen) of compartmental Schiff base (H_4L) and the Ni(II) complex (I) were determined with the help of a Perkin–Elmer CHN analyzer 2400. Mass spectra were done with a JEOLJMS-AX 500 mass spectrometer. IR spectra (KBr, 400–4000 cm^{-1}) were recorded on a Perkin–Elmer model 883 infrared spectrophotometer. The electronic spectra of title complex (1) in organic solvent were recorded on a Hitachi model U-3501 spectrophometer. The ^1H NMR spectra of compartmental Schiff base (H_4L) were recorded on Bruker 300 MHz FT-NMR spectrophotometer using trimethylsilane as internal standard in $CDCl_3$ solvent. The magnetic susceptibility value of Ni(II) complex (1) was determined by Gouy method. Correction for diamagnetism of the constituent atoms was calculated using common Pascal's constant. The effective magnetic moments values μ_{eff}, of respective Ni(II) complex was calculated using the given equation:

$$\mu_{eff} = 2.83\ (\chi_M T)^{1/2}$$

2.3 Synthesis

2.3.1 Synthesis of Compartmental Schiff Base (H_4L)

To a dry methanol solution (50 mL) of 5-chloro-2-hydroxy acetophenone (1.024 g, 6 mmol) was added with triethylenetetramine (0.439 g, 3 mmol) in the same methanol solvent (5 mL) with constant stirring for 1 hr. The colour of the overall solution turned to yellow and it was then additionally reflux for 7 hours. The solution was then heated on a steam bath to reduce the volume 15 mL and overall solution was cooled on an ice bath. At this moment a deep yellow product was separated out, collected by filtration under suction, washed with cold methanol. Then the yellow solid was dried in vacuum over fused $CaCl_2$ desicator (Scheme 2).



Scheme 2 Route for synthesis of Schiff base (H_4L)

For novel Schiff base H_4L (Yield: 85 %), M.Wt.(451.39), Exact Mass: 450.16, m/e: 451, Anal. Calc. for $C_{22}H_{28}N_4O_2Cl_2$: C, 58.54; H, 6.25; Cl, 15.71; N, 12.41; O, 7.09 Found: C, 58.6; H, 6.22; Cl, 15.7; N, 12.4; O, 7.00 IR(KBr, ν_{max}/cm^{-1}): ν(O-H) =2800-2920; ν(C=N) 1616.07; ν(C=O) 1700-1800. UV-Vis spectrum, $\lambda_{max}(CH_3OH)$: 272 nm, 333 nm and 402 nm.

2.3.2 Synthesis of Novel Mononuclear Complex (I) [$Ni(C_{22}H_{26}N_4O_2Cl_2)$]

To the solution of $NiCl_2.6H_2O$ (0.23771 g, 1 mmol) in methanol (25 mL), a solution of the ligand (H_4L) (0.451 g, 1 mmol) in same solvent (25 mL) was added dropwise, the colour of the solution was turned into deep red. The whole assembly was stirred for 2.5 hrs. Immediately a red coloured ppt. appeared, filtered under suction and was dried in vacuum over fused $CaCl_2$ desicator.

For complex (1) [$Ni(C_{22}H_{26}N_4O_2Cl_2)$], (Yield: 85 %), M.Wt. (508.07), Exact Mass: 506.08, m/e: 506.08, Anal. Calc. for complex (1): C, 52.01; H, 5.16; Cl, 13.6; N, 11.03; O,6.30, Ni, 11.55 Found: C, 52.00; H, 5.14; Cl, 13.5; N, 11.00; O, 6.31, Ni, 11.53 IR(KBr, ν_{max}/cm^{-1}): 1570.93(m), 1451.78(s), 1323.67(s), 1406.25(s), 2347.77(s), 2341.35(s), 670.40(s), 823.20(s), 1236.41(m). UV-Vis spectrum, $\lambda_{max}(CH_3OH)$: 339 nm and 401 nm.

3. Results and Discussion

3.1 Synthesis

Compartmental Schiff base (H_4L) and one new mononuclear Ni(II) complex (1) have been synthesized successfully in our laboratory. Both are characterized by different physicochemical techniques like elemental analyses, molar conductance study, IR, UV-Vis, mass (m/e), and ^1H NMR spectroscopic studies. Additionally magnetic moment was determined for newly synthesized Ni(II) mononuclear complex.

3.2 Characterization of Compartmental Novel Schiff Base (H_4L) & Mononuclear Ni(II) Complex (1)

3.2.1 Characterization of Compartmental Schiff Base (H_4L)

From IR studies, the presence of IR band near 1616.06 cm^{-1} provides the evidence of formation of azomethine functional group (C=N) in the synthesized novel Schiff base. The absence of C=O at around 1700-1800 cm^{-1} confirmed the Schiff base (C=N) double bond formation according to Scheme 1. The very week band near 2800-2920 cm^{-1} indicates the presence of intramolecular hydrogen bond –OH (Fig. 1). ^1H NMR different spectroscopic data was further confirmed the formation of novel Schiff base (Fig. 2). The UV-Vis spectrum in methanol of compartmental Schiff base reflects three main peaks at 272 nm, 333 nm and 400 nm. The first two UV-Vis spectrum are assigned due to benzene $\pi \rightarrow \pi^*$ and imino $\pi \rightarrow \pi^*$ transitions. Additional peak 400 nm is due to $n \rightarrow \pi^*$ transition (Fig. 3).

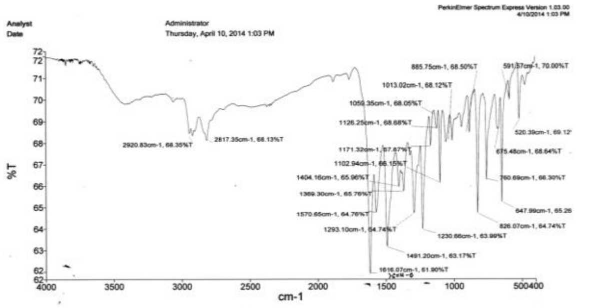


Fig. 1 IR spectrum of compartmental Schiff base (H_4L)

IR (KBr, ν_{max}/cm^{-1}): 1616.07 (s), 1491.20 (s), 1230.66 (s), 826.07 (s), 1570.65 (m), 1293.10 (m), 1102.94 (m), 1570.65 (m), 2920.83 (w), 2817.35 (w).

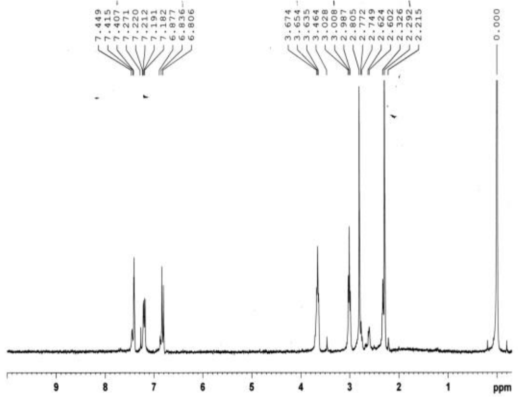


Fig. 2 ^1H NMR spectrum of Schiff base (H_4L)

^{1}HNMR (300 MHz, $CDCl_3$) δ/ppm 2.292 (6H, $-CH_3$), 2.772 (4H, $-CH_2$ alpha to NH2), 3.008 (4H, $-CH_2$ beta to N=C), 3.654 (4H, $-CH_2$ alpha to N=C), 6.806- 7.449 (aromatic ring H).

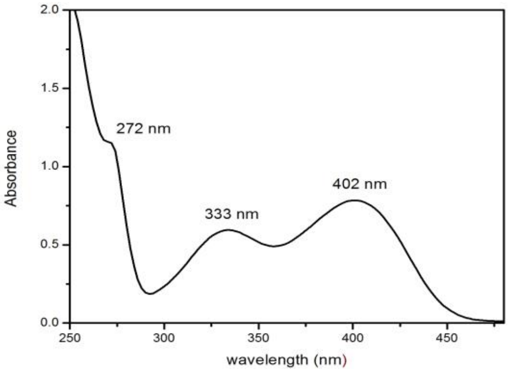


Fig. 3 UV-Vis spectrum of Schiff base(H_4L)

3.2.2 Characterization of Mononuclear Ni(II) Complex(1)

Respective Ni(II) complex (1), from structural view point we observed that here Ni(II) metal center are coordinated with imine nitrogen (N_4 binding core) & two deprotonated (phenolato oxygen). Thus the above complexing situation is confirmed by the shift of characteristic frequency of azomethine (C=N) to lower wavenumber (1406.25 cm^{-1}) (Fig. 4).The IR frequency of free compartmental Schiff base (2800-2920 cm^{-1}) was completely removed during complex formation and this was further confirmed that the free –OH must be strongly coordinated with Ni(II) metal center. Complex (1) shows very clearly 339 nm and 401 nm UV-Visible absorption peak (Fig. 5). This is merely the modification of compartmental Schiff base ligand π-π* and n-π* transition. The shift of spectral peak takes place mainly metal coordination with ligand imine (N$_4$ binding core) and two deprotonated (phenolato oxygen). d-d transition does not appear due to very low intensity.

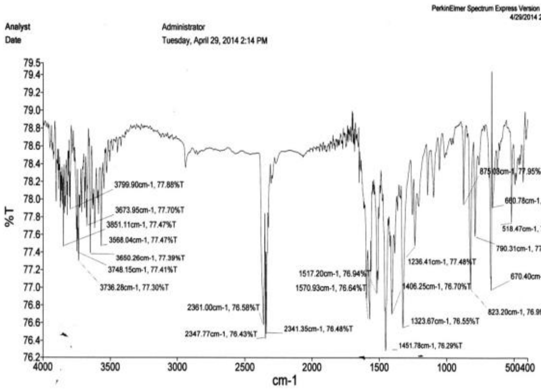


Fig. 4 IR spectrum of synthesised complex (1)

IR (KBr, ν_{max}/cm^{-1}): 1570.93(m), 1451.78(s), 1323.67(s), 1406.25(s), 2347.77(s), 2341.35(s), 670.40(s), 823.20(s), 1236.41(m).

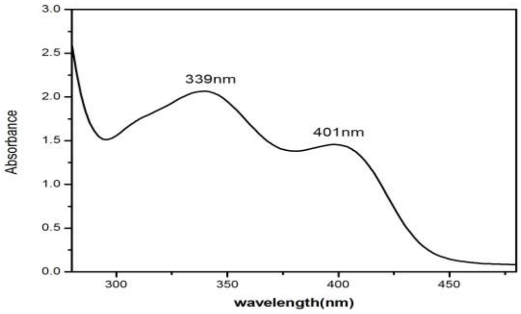


Fig. 5 UV spectrum of synthesised complex (1)

3.3 Magnetic Moment Study

Ni(II) metal has well known d^8 configuration and thus probable stereochemical configurations are octahedral, square planar/tetrahedral(CN=4) & five coordinated square pyramidal /TBP. The peculiarity of Ni(II) chemistry is that complex of one configuration is readily converted to other configuration. This structural convertibility implies that the free energy difference between different stereochemical configurations is very small. For all regular Ni(II) octahedral complexes magnetic moment range 2.80-3.40 BM, but on account of spin orbit coupling and contribution from $^3A_{2g}$ and next higher $^3T_{2g}$ give somewhat higher value than spin only moment [2.83 BM]. Our new synthesized Ni(II) complex (1) shows μ_{eff} = 2.9 BM, that consistent with the octahedral geometry (Fig. 6). During our research work, we have critically survey of selected some Ni(II) octahedral complexes magnetic moment data (Table 1) and observed that μ_{eff} value of our synthesized new complex very close to literature survey magnetic moment values of Ni(II) octahedral complexes (Table 1).

Table 1 Details theoretical Survey of Ni(II) octahedral Complexes magnetic moment data

Octahedral complexes of Ni(II)	μ_{eff} data (B.M)	References
1. $C_{21}H_{22}Cl_2NiN_6O_{10}$	2.86	28
2.$C_{22}H_{24}Cl_2NiN_6O_{10}$	2.97	29
3.$[Ni(GHL1)_2](OAc)_2.4H_2O$	2.82	30
4.$C_{17}H_{20}N_2O_2Ni$	2.50	31
5.$[NiL_2].2H_2O$	2.87	32
6.$[NiL_2(H_2O)_2]$	2.80	33
7. $C_{40}H_{40}N_6Ni_3O_4S_2$	2.87	34

N.B. 1. L & GHL1→ Notations are particular Schiff base as per references

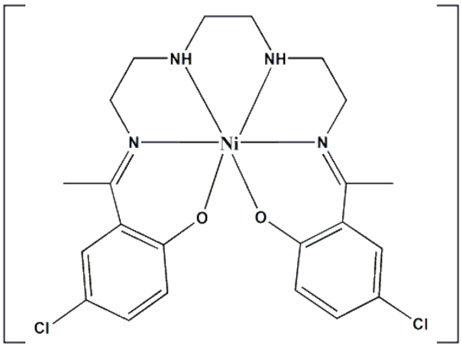


Fig. 6 probable expected structure of complex (1)

4. Conclusion

We have successfully synthesized in our laboratory symmetrical azo-linked tetradentate novel Schiff base (H_4L), and one new mononuclear Ni(II) complex(1). Compartmental Schiff base (H_4L) and Ni(II) complex(1) have been characterized by elemental analyses, molar conductance, FT-IR, UV-Vis, mass, and ^{1}H NMR spectroscopic study. Additionally magnetic moment study will be conducted to confirmed the expected probable geometry of synthesized Ni(II) complex. A complete literature survey of magnetic moment data of some selected purely octahedral Ni(II) complexes have been compared with our synthesized Ni(II) complex and concluded that the respective complex (1) must be fulfilled octahedral geometry.

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