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Synthesis Spectral characterization and Biological Importance of N (2-hydroxybenzylidene) pyridine-2-amine and Its Metal Complexes With Co (II), Ni(II), Cu(II) and Zn(II)

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Abstract : The method of preparation of Schiff base from 2- hydroxybenzaldehyde and 2-aminopyridine and its complexes with Co, NI, Cu and Zn metals described. Obtained complexes were tested for IR, NMR, Elemental analysis and antimicrobial activity. Complexes with Co (II), Ni(II), Cu(II) are found to be high spin tetrahedral complexes and Zn(II) low spin with same geometry.

Key-words : Bacteria, Schiff base, Elemental Analysis, Aminopyridine.

Introduction:

Most substantial part include in coordination chemistry is designing ligand with different denticity¹⁻³ aldehyde and amines have number of donor atoms such as O, N³. The numerous amount of work have been performed on the complex behavior of O and N atoms.⁴ In recent years many efforts have been put in the study of Schiff bases and their metal complexes, they have probable biological interests⁵⁻⁷. The significance of complexes and metal atoms in biology is always been consideration. Schiff bases from salicylaldehyde and 2-aminopyridine is form by condensation reaction.⁸⁻¹¹ complexes of Co(II), NI(II), Cu(II) and Zn(II) with Schiff bases have significant medicinal applications and shows antibacterial activities.¹²⁻¹⁵. Schiff base and Their metal complexes displays biological activities.¹⁶⁻¹⁸ By keeping this fact in mind in the present work we tried to synthesize the Schiff base and its metal complexes of Co, Cu, Zn and Ni. All the compounds were tested for IR, NMR, Electronic spectra and antibacterial and antifungal activity.

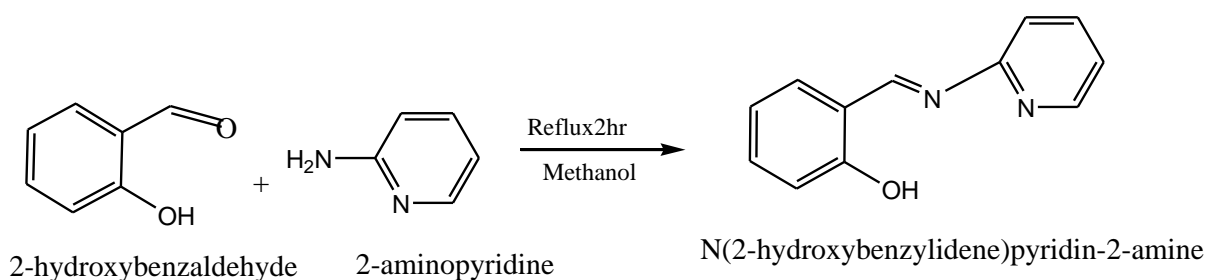
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All the chemicals and solvents used in this work were AR grade. Obtained from marketable sources and used as provided. UV spectra are taken in UV-visible spectrometer. Melting point were recorded by open capillary method. Elemental analysis was done on Eager 350 analyzer. $^1\text{H-NMR}$ spectra of metal complexes along with Schiff base recorded on Bruker 300Hzs spectrometer in DMSO from SPPU.

Synthesis of N-(2-hydroxybenzylidene) pyridine -2-amine

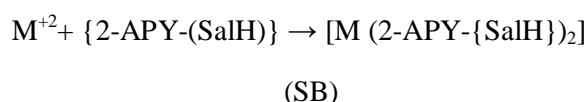
0.001M Salicylaldehyde (1.07ml) in 10 ml methanol was blended in with 0.001M 2-aminopyridine (0.94g) in 10 ml methanol under steady mixing for 1 hr. The subsequent yellow solution was expose to sunlight for 12hr and then refluxed for 2hr in round bottom flask furnished with an air cooled condenser. The yellow crystalline precipitate was obtained. The precipitate was filtered under Buckner funnel. Washed with ethanol recrystallized and dried. The reaction is given below.



Scheme 1

Synthesis of Schiff base complexes

1mmol of salt $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{CuCl}_2 \cdot 3\text{H}_2\text{O}$, $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{ZnCl}_2 \cdot 6\text{H}_2\text{O}$ were independently dissolved in 10ml of water. The Schiff base (SB) solution was added to these salts solution separately and stirred for 30 minute at 40-44°C temperature and allow to stand for half an hour. The precipitate was made as Schiff base complex compounds and were filtered off and dried over anhydrous CaCl_2 . The general reaction for synthesis of all the complexes is point out as:



Where, $\text{M}^{+2} = \text{Ni (II)}, \text{Co (II)}, \text{Cu (II)} \text{ and } \text{Zn (II)}$

Results and discussion

All the complexes are constant at room temperature and are insoluble in mutual organic solvents but are soluble in DMSO, DMF and CHCl_3 .

Elemental analysis and Conductivity Measurement

Micro analysis and some physical properties like melting point, color, yield of the complexes are given in Table 1 and Table 2. The data for micro analysis were in good arrangement with the suggested empirical formulation of the complexes. All compounds were obtained in good yield and color. The conductance values of the complexes revealed that all the complexes are non-electrolyte in nature¹⁶. The obtained values imply that no anions are present outside the coordination sphere in all the complexes¹⁸.

Table 1 Elemental analysis of the ligand and complexes

Compounds	Molecular Weight g/mol	% of Element		
		C	H	N
(2-APY-{SalH}) (SB)	198.21	72.60 (72.73)	4.96 (5.04)	14.02 (14.14)
Ni(C ₁₂ H ₁₀ N ₂ O) ₂	455.10	62.42 (63.33)	4.39 (4.13)	11.75 (12.31)
Co(C ₁₂ H ₁₀ N ₂ O) ₂	455.34	62.48 (63.30)	3.80 (4.39)	11.52 (12.30)
Cu(C ₁₂ H ₁₀ N ₂ O) ₂	459.96	61.52 (62.67)	4.04 (4.35)	11.86 (12.18)
Zn(C ₁₂ H ₁₀ N ₂ O) ₂	461.82	56.06 (56.67)	3.12 (3.93)	10.50 (11.02)

Table 2 Physical properties of the ligand and complexes.

compounds	Color weight	Yield %	Melting pt. °C	Molar Conductance (Ω ⁻² cm ² mol ⁻¹)
(2-APY-{SalH}) (SB)	Orange red	96	65	-
Ni(C ₁₂ H ₁₀ N ₂ O) ₂	Green	62	283	15.4
Co(C ₁₂ H ₁₀ N ₂ O) ₂	Brown	63		17.2
Cu(C ₁₂ H ₁₀ N ₂ O) ₂	black	59	150	15.6
Zn(C ₁₂ H ₁₀ N ₂ O) ₂	yellow	65	188	14.3

Table 3 Magnetic Moment Of complexes

Complexes	Co(C ₁₂ H ₁₀ N ₂ O) ₂	Ni(C ₁₂ H ₁₀ N ₂ O) ₂	Cu(C ₁₂ H ₁₀ N ₂ O) ₂	Zn(C ₁₂ H ₁₀ N ₂ O) ₂
Magnetic Moment (B.M)	3.73	2.85	1.83	0

Value of magnetic moment was found to be 3.7, 2.85 and 1.83 B.M. for Co (II), Ni(II) and Cu(II) respectively indicates the high spin tetrahedral complexes. The value for and Zn(II) was observed to be zero indicates diamagnetic nature, while other three are paramagnetic in nature

IR Spectral Studies

The Infrared spectral data of the ligand and complexes were listed in table 3. The spectral data of the ligand showed a strong absorption band at 1580-1640 cm⁻¹ due to ν(C=N) stretching^{17,18}. Band at 3020-3165 cm⁻¹ due to aromatic ν(C-H) stretching¹¹⁻¹⁵. The band at 500-600 cm⁻¹ shows coordination of ligand to metal ion through N atom. Band at 440-500 cm⁻¹ due to ν(M-O) stretching, which indicated the complexation have taken place to the metal through the deprotonated O atom of phenolic OH moiety. Similar type of observation was found by Md. Motahar Hossain et al 2018.

Table 4 IR spectral data of ligand and complexes

Compounds	$\nu(\text{O-H}) \text{ cm}^{-1}$	$\nu(\text{C=N}) \text{ cm}^{-1}$	$\nu(\text{C-H})$ of aromatic cm^{-1}	$\nu(\text{M-O}) \text{ cm}^{-1}$	$\nu(\text{M-N}) \text{ cm}^{-1}$
(2-APY-{SalH}) (SB)	3415.35	1623	3015	-	-
Ni(C ₁₂ H ₁₀ N ₂ O) ₂	-	1609	3070	447	557
Co(C ₁₂ H ₁₀ N ₂ O) ₂	-	1622	3013	448	520
Cu(C ₁₂ H ₁₀ N ₂ O) ₂	-	1614	3062	441	555
Zn(C ₁₂ H ₁₀ N ₂ O) ₂	-	1622	3009	444	541

Infrared spectra of Schiff base and metal complexes of Ni and Cu is given in figure A, B and C below respectively

Figure 1 IR spectra of Schiff base ligand

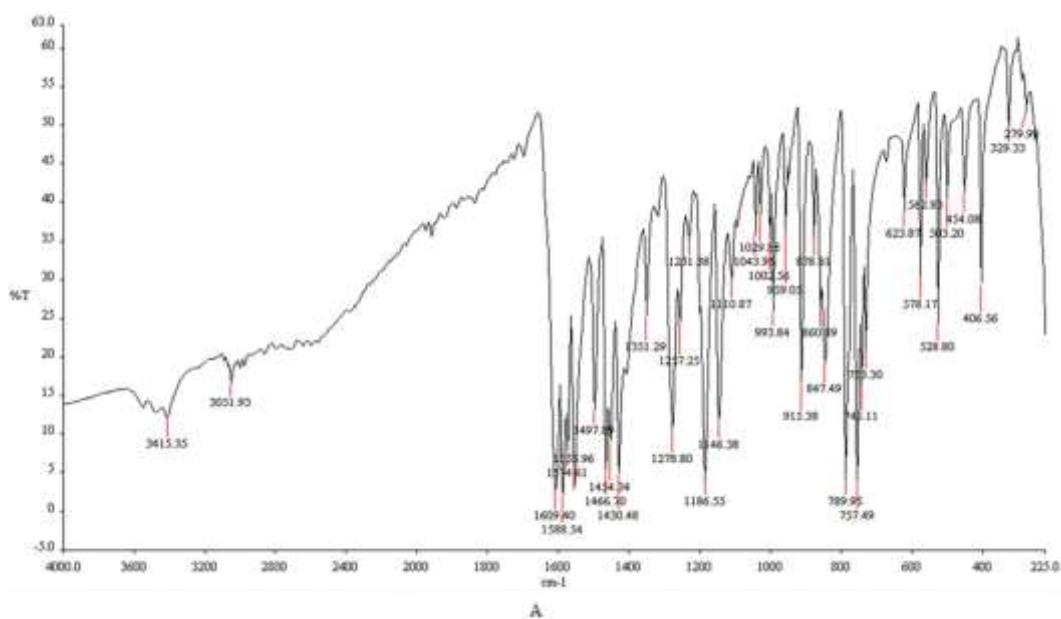
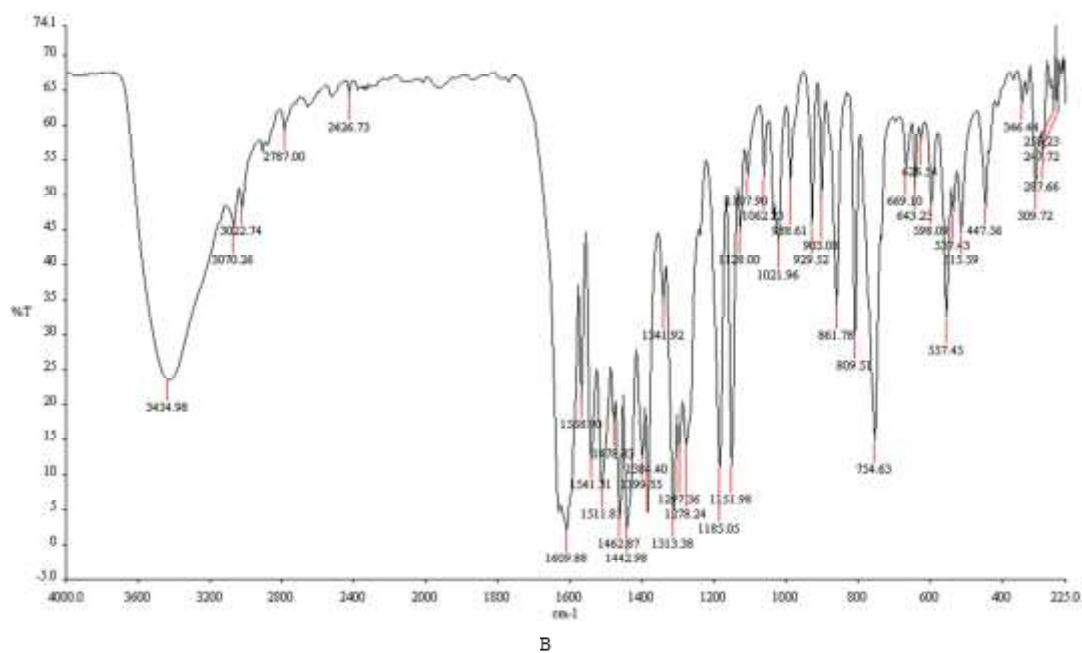
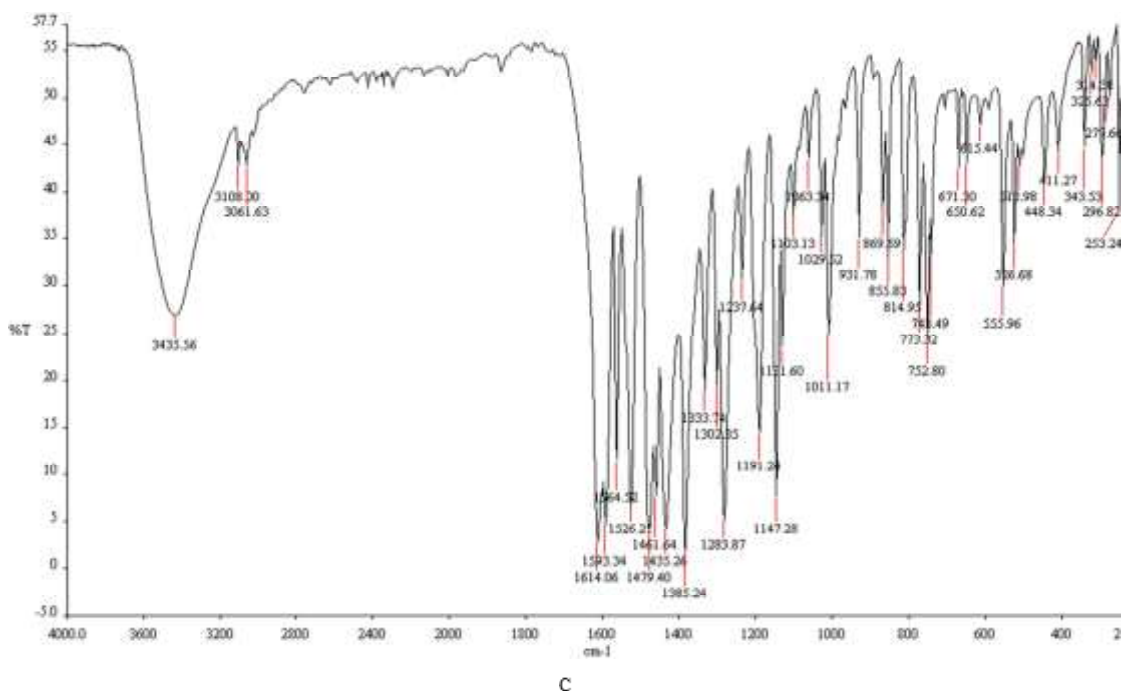


Figure 2 IR spectra of Ni complex

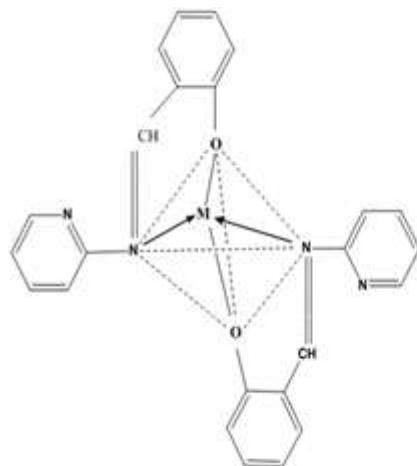


IR spectra of Cu complex



Conclusion

Magnetic susceptibility measurement shows that Ni(II) Co(II) and Cu(II) complexes are paramagnetic in nature and Zn(II) is diamagnetic in nature. The IR data showed that all the metals are coordinated through two atoms of Schiff base that is nitrogen of azo group and O of hydroxyl group. Magnetic moment data revealed that complexes of Co(II), Ni(II) and Cu(II) with Schiff base are tetrahedral high spin complexes whereas Zn(II) had tetrahedral low spin geometry. The electronic spectral study were definite that all the complexes shows tetrahedral geometry. Based on the above results and facts, the structure of the complexes have been proposed in figure 4.



M- Co(II), Ni(II), Cu(II) and Zn(II)

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