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Synthesis, Characterization and Magnetic Susceptibility of Novel Transition Metal Ion Complexes with (E)-2-((7H-Purin-6-ylimino)methyl)phenol and Its Antibacterial Efficiency

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ABSTRACT

The novel transition metal ion complexes were synthesized by refluxing the ethanolic solutions of metal acetates with Schiff base in 1:2 ratios. The structure and characterization of synthesized complexes of Mn (II), Co (II), Ni (II), Cu (II) and Zn (II) with Schiff base (E)-2-((7H-purin-6-ylimino)methyl)phenol were elucidated by using ^{1}H NMR, FT-IR and UV-Visible spectroscopic techniques. The synthesized compounds were also been screened against gram positive and gram-negative bacteria. The novel compounds were further carried out for the study of magnetic susceptibility.

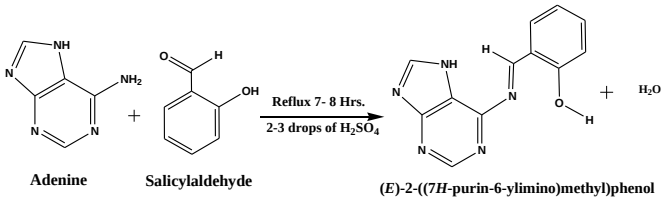
1. Introduction

Schiff-base macro ligands synthesized from reaction of dialdehydes and amino compounds [1-3] form stable complexes, perhaps selective to specific metallic ions with applications in electrochemistry, bioinorganic, antimicrobial activity, fluorescence properties, catalysis, metallic deactivators, separation processes, and environmental chemistry among others [3]. Preparation of new ligands is an important step in development of metal complexes, which exhibit unique properties and reactivity. For example, in asymmetric catalyst systems, small changes in donating ability of the ligand or the size of its substituents can have a dramatic effect on catalyst efficiency and enantioselectivities [3, 4]. The nitro group is a strong electron-withdrawing group and due to its steric effects, it has played an important role in affecting the reactivity and enantioselectivities in asymmetric cyclopropanation and allylic alkylation reactions [4]. In continuation of our research on preparation of Schiff bases [4, 5] and their complexes [6-9], it has been decided to prepare new Schiff bases containing electron withdrawing and donating substituents. This article describes the synthesis and spectroscopic characterization of several Schiff bases and their complexes with transition metal ions. The corresponding materials were characterized by spectroscopic (IR, UV-Vis, ^{1}H- NMR) and physical (melting point, magnetic susceptibility) data.

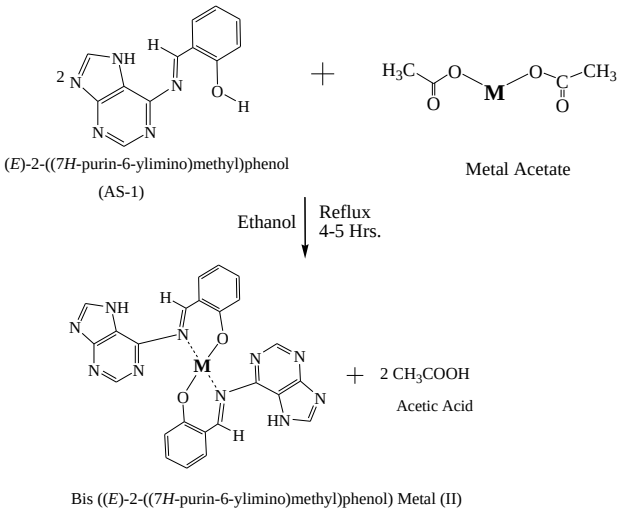
2. Experimental Methods

2.1 Synthesis of (E)-2-((7H-purin-6-ylimino) methyl)phenol (AS-1) – Schiff Base (L_1)

The Schiff base (ligand AS-1) was synthesized by taking adenine (0.01 M) and salicylaldehyde (0.01 M) in ethanolic medium followed by the addition of 2-3 drops of conc. sulphuric acid in a catalytic amount and poured it in a round bottom flask connected with a reflux condenser. The above given reaction mixture was refluxed for 7-8 hrs. Water formed during the reaction was collected through Deane Stark funnel. The solvent was removed under sunlight irradiation. The chemical reaction pathway represented in Scheme 1. The resulting pale yellowish solid was recrystallized from ethanol. Color - (Pale yellow), M.P.–107.2 °C, Yield-68%.



Scheme 1 Preparation of (E)-2-((7H-purin-6-ylimino)methyl)phenol (AS-1) from adenine and salicylaldehyde



M = Mn^{++} , Co^{++} , Ni^{++} , Cu^{++} , Zn^{++}

Scheme 2 Preparation of bis((E)-2-((7H-purin-6-ylimino)methyl)phenol)metal (II) complex

2.2 Synthesis of Transition Metal-Ligand Complexes of Schiff Base (L_1)

The metal-ligand mole ratio was taken in 1:2 proportions. Metal acetate salt (0.01 M) and (E)-2-((7H-purin-6-ylimino) methyl)phenol (AS-1) (0.02 M) was dissolved in 50 mL ethanolic solutions with vigorous stirring and warm it until the solution form turbid appearance. Then the solution was poured in round bottom flask equipped with refluxed condenser and refluxed it for 4-5 hrs. The solid complex with characteristic coloured was

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formed within few minutes after cooling at room temperature. The resultants filtered off by using whatman-41 filter paper and washed with ethanol. The product was dried and stored for further studies. The synthetic route of formation of complex is mentioned in Scheme 2.

2.3 Instrumentation

FTIR spectra in the range, 4000-400 cm^{-1}, were recorded using Agilent Technology Spectrophotometer; UV-visible spectra were measured by using Shimadzu 160 spectrophotometer in the range 200-800 nm. The magnetic susceptibility values of the prepared complexes were obtained at room temperature using Bruker Magnet B.M.6, The ^1H nuclear magnetic resonance spectra were recorded using BRUKER ADVANCED II 400 MHz spectrometer in DMSO as a solvent, relative to the internal standard tetramethylsilane (TMS).

3. Results and Discussion

3.1 NMR Spectroscopy

The data of ^1H NMR of the ligand (AS-1) and its complexes displayed good solubility in DMSO. The proton nuclear magnetic resonance spectral data gave additional support for the composition of the complexes. The observed changes are the evidences of complexation had happened because the chemical shift of a compound is heavily depended on its electronic environment. The ^1H NMR spectrum of the complexes confirmed the disappearance of O-H signal at δ 4.30 ppm in the free ligand. The δ 6.60-8.50 ppm resonance signal protons of the aromatic ring shifted to the higher field upon complexation. It is most likely that shift is due to the decrease of electron density at ^1H protons when oxygen is bonded to metal ion.

(E)-2-((7H-purin-6-ylimino)methyl)phenol (AS-1) (L_1)

Solid, M.P.: 123.1 °C; **UV (λ max) in ethanol:** 290 nm; **(IR) υ max (KBr/cm^{-1}):** 3625.05 (Ar-OH), 3291.59 (Ar-N-H), 3087.99 (Ar-C-H), 1669.93 (Ar-C=C), 1599.43 (Ar-C=N), 1305.24 (C-C), 1247.33 (C-N), 1119.14 (C-O), 937.49 (Trans disubstituted C=N), 764.79 (Ortho disubstituted aromatic); **^{1}H-NMR (δ-ppm):** 4.30 (s, 1H, Ar-O-H), 6.73-7.40 (m, 4H Ar-H), 7.80 (d, 1H, Ar-N=C-H), 8.70 (s, 1 H Ar-N=C-H), 9.60 (s, 1H, Ar-N=C-H), 10.50 (d, 1H, Ar-N-H).

Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol)manganese(II) Complex (ASM-1)

Solid, M.P.: 169.0 °C; **UV (λ max)** in ethanol: 280 nm; **(IR) υ max (KBr/cm^{-1}):** 3298.56 (Ar-N-H), 3097.26 (Ar-C-H), 1668.05 (Ar-C=C), 1550.08 (Ar-C=N), 1334.18 (C-C), 1250.29(C-N), 1092.03(C-O), 938.29 (Trans disubstituted C=N), 794.30 (Ortho disubstituted aromatic), 719.53 (Mn-O (Stretching)); **^{1}H-NMR (δ-ppm):** 6.00-6.80 (m, 4H Ar-H), 7.35 (d, 1H, Ar-N=C-H), 8.40 (s, 1 H Ar-N=C-H), 9.50 (s, 1H, Ar-N=C-H), 10.60 (d, 1H, Ar-N-H).

Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol)cobalt(II) Complex (ASC-2)

Solid, M.P.: 161.2 °C; **UV (λ max)** in ethanol: 283 nm, **(IR) υ max (KBr/cm^{-1}):** 3296.63 (Ar-N-H), 3117.96 (Ar-C-H), 1658.30 (Ar-C=C), 1565.25 (Ar-C=N), 1333.23 (C-C), 1245.25 (C-N), 1095.23 (C-O), 938.29 (Trans disubstituted C=N), 792.16 (Ortho disubstituted aromatic), 720.91 (Co-O (Stretching)); **^{1}H-NMR (δ-ppm):** 6.20-6.90 (m, 4H Ar-H), 7.40 (d, 1H, Ar-N=C-H), 8.60 (s, 1 H Ar-N=C-H), 9.40 (s, 1H, Ar-N=C-H), 10.30 (d, 1H, Ar-N-H).

Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol)nickel(II) Complex (ASN-3)

Solid, M.P.: 158.2 °C; **UV (λ max)** in ethanol: 285 nm; **(IR) υ max (KBr/cm^{-1}):** 3293.24 (Ar-N-H), 3115.74 (Ar-C-H), 1655.85 (Ar-C=C), 1563.90 (Ar-C=N), 1329.47 (C-C), 1246.92 (C-N), 1086.50 (C-O), 935.30 (Trans disubstituted C=N), 791.57 (Ortho disubstituted aromatic), 721.32 (Ni-O (Stretching)); **^{1}H-NMR (δ-ppm):** 6.00-6.80 (m, 4H Ar-H), 7.40 (d, 1H, Ar-N=C-H), 8.30 (s, 1 H Ar-N=C-H), 9.45 (s, 1H, Ar-N=C-H), 10.25 (d, 1H, Ar-N-H).

Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol)copper(II) Complex (ASC-4)

Solid, M.P.: 179.9 °C; **UV (λ max)** in ethanol: 290 nm; **(IR) υ max (KBr/cm^{-1}):** 3296.97 (Ar-N-H), 3123.85 (Ar-C-H), 1660.66 (Ar-C=C), 1583.58 (Ar-C=N), 1335.46 (C-C), 1304.34 (C-N), 1084.25 (C-O), 933.74 (Trans disubstituted C=N), 792.05 (Ortho disubstituted aromatic), 736.41 (Cu-O (Stretching)); **^{1}H-NMR (δ-ppm):** 5.70-7.00 (m, 4H Ar-H), 7.35 (d, 1H, Ar-N=C-H), 8.35 (s, 1 H Ar-N=C-H), 9.50 (s, 1H, Ar-N=C-H), 10.30 (d, 1H, Ar-N-H).

Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol)zinc(II) Complex (ASZ-5)

Solid, M.P.:165.8 °C; **UV (λ max)** in ethanol: 288 nm; **(IR) υ max (KBr/cm^{-1}):** 3294.35 (Ar-N-H), 3111.08 (Ar-C-H), 1669.85 (Ar-C=C), 1603.32 (Ar-C=N), 1325.41 (C-C), 1246.44 (C-N), 1069.60 (C-O), 932.82 (Trans disubstituted C=N), 790.22 (Ortho disubstituted aromatic), 722.69 (Zn-O (Stretching)); **^{1}H-NMR (δ-ppm):** 6.40-7.00 (m, 4H Ar-H), 7.20 (d, 1H, Ar-N=C-H), 8.20 (s, 1 H Ar-N=C-H), 9.50 (s, 1H, Ar-N=C-H), 10.40 (d, 1H, Ar-N-H).

3.2 Infra-Red Spectroscopy

The above given data of FT-IR spectrum of the ligand AS-1 shows the characteristic bands at 3625.05 cm^{-1}, 3291.59 cm^{-1} & 1625.32 cm^{-1} which are assigned to Ar-O-H, Ar-N-H and C=N stretching respectively. These bands reveal the formation of Schiff Base. The formation of complexes of Schiff Base with Mn (II), Co (II), Ni (II), Cu (II) and Zn (II) acetates was confirmed by the disappearance of –O-H band in the region of 3200-3600 cm^{-1} and occurrence of metal-oxide bands at 719.53 cm^{-1}, 720.91 cm^{-1}, 721.32 cm^{-1}, 736.41 cm^{-1} and 722.69 cm^{-1} for Mn-O, Co-O, Ni-O, Cu-O, Zn-O stretching respectively. In the free ligand, the band at 1599.43 cm^{-1} was assigned to the stretching of C=N bond. On complexation this band was shifted to a lower frequency region. This shift is due to the metal-ligand electron sharing effect.

3.3 Ultraviolet-Visible Spectroscopy

The ultraviolet visible electronic spectrums of the compounds were recorded in DMSO solvent. The bands at wavelengths 290, 280, 283, 285, 290 and 288 nm are attributed to π→π* electronic transition [11]. The electronic spectra of complexes showed, as expected, different absorptions from that of the free ligand. In the complexes these bands were shifted to different wavelength than the corresponding bands in the ligand as shown above, which appears in the wavelength range between 280-300 nm. The ligand field electronic transitions between the metal d orbital's appear in Ni (II) and Cu (II) bands located in the visible region at 460 nm for Ni $(L)_2$ assigned to the transition $^3A_2 \rightarrow {}^3T_1$ (p) and 610 nm for $Cu(L)_2$ assigned to the transitions $^2B_1g \rightarrow {}^2A_1g$. The other complexes were diamagnetic as expected for d^{10} ions; so that no (d-d) transition can be expected in the visible region.

3.4 Magnetic Susceptibility and Conductivity Measurements

The experimental magnetic moments for metal complexes are listed in Table 1. Magnetic measurements are widely used in studying transition metal complexes. The magnetic properties are due to the presence of unpaired electrons in the partially filled d-orbital in the outer shell of these elements. This magnetic measurement gives an idea about the electronic state of the metal ion in the complexes.

Table 1 Magnetic moment, conductivity measurements in DMF solvent

Symbol	Name	Conductivity ohm^{-1}cm^2 mol^{-1}	Magnetic moment (B.M.)	Suggested structure
(AS-1) (L_1)	(E)-2-((7H-purin-6-ylimino) methyl)phenol	-	-	-
(ASM-1)	Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol) manganese(II) complex	15	4.25	Tetrahedral
(ASC-2)	Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol) cobalt(II) complex	13	4.20	Tetrahedral
(ASN-3)	Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol) nickel(II) complex	16	4.30	Tetrahedral
(ASC-4)	Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol) copper(II) complex	25	1.88	Square planner
(ASZ-5)	Bis((E)-2-((7H-purin-6-ylimino)methyl)phenol) zinc(II) complex	16	3.90	Tetrahedral

The magnetic moment for Ni (II) in any complex is approximately 3.11 B.M., refers to a high spin tetrahedral structure, while the value of Cu (II) is approximately 1.44 led to suggest the square planar structure which can become in a good agreement with the data of electronic transitions. Other complexes have no magnetic moment because of diamagnetic nature. Molar conductivity measurement in DMF solvent at 25 °C showed that the complexes were non-electrolyte [11].

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3.5 Antibacterial Activity

The titled compounds were screened for their antibacterial activity using disc diffusion method. The bacterial organisms used included both gram positive and gram-negative strains like *Staphylococcus aureus*, *Escherichia coli*, *Salmonella enteric Ser paratyphi*, *Klebsiella pneumonia* and *Pseudomons aeruginosa*. For antibacterial susceptibility testing of title compounds, the sterile disc of 6 mm diameter (SD067, Hi-Media, Mumbai) was loaded with 20 µL of title compound solution (1000 µg/mL) in DMF. The discs were then placed at centre on the Mueller-Hinton agar seeded with bacterial inoculums approximately 106 CFU/ mL, incubated at 37 °C for 24 hrs and growth inhibition zone formed around disc was measured. Test was done in triplicate and mean value was considered as inhibition zone. Solvents were used as controls and showed no inhibitions in preliminary studies. All the synthesized complexes exhibited moderate to good activity against the test organisms [7].

Table 2 Antimicrobial activity

Compound	Gram positive bacteria	Gram negative bacteria			
	Staphylococcus aureus	*Salmonella enterica Ser Paratyphi*	*Escherichia Coli*	*Klebsiella Pneumonia*	*Pseudomons aeruginosa*
AS-1(L1)	+	++	+	++	+
ASM-1	-	++	-	++	+
ASC-2	++	+	+	+	++
ASN-3	+++	++	+	+	+++
ASC-4	++	++	++	++	++
ASZ-5	++	+++	+++	++	++

+++ = Zone size 16-22 mm; ++ = Zone size 9-15 mm; + = Zone size 6-8 mm; — = No inhibition

4. Conclusion

The ligand (E)-2-((7H-purin-6-ylimino)methyl)phenol (AS-1) was successfully synthesized by the condensation method. The ligand (AS-1) was treated with Mn(II), Co(II), Ni(II), Cu(II), Zn(II) metal acetate salts to afford the corresponding complexes. The characterization data of ^{1}H NMR, IR and UV-Vis reveals the successful formation of ligand and their complexes. The magnetic susceptibility data was attributed to the square planar geometry of Cu (II) complex and tetrahedral geometry of other complexes. The antibacterial study reveals that Ni (II), Cu (II) and Zn (II) metal ion complexes showed best inhibition activity towards all the strains of bacteria where as other complexes showed good activity against the gram positive and gram-negative bacteria.

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