Juni Khyat (जूनी खात) (UGC CARE Group I Listed Journal) SYNTHESIS, SPECTRAL CHARACTERIZATIONS AND CATALYTIC ACTIVITY OF M (II) COMPLEXES DERIVED FROM SCHIFF BASE LIGAND 5-DIETHYLAMINO-2-({[4-(DIMETHYLAMINO) PHENYL] IMINO} METHYL) PHENOL

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ABSTRACT:

A new series of metal complexes of the type $M(L)_2$ ([Cu(L)₂] (1), [Ni(L)₂] (2), ;Schiff base (L) derived has been synthesized by condensation in acidic medium and characterized by elemental analysis (C, N, O). The spectral (IR, UV, ¹H NMR and EPR) studies are also carried out. The molecular structures of the Schiff base ligand (L) is determined by the single crystal X-ray diffraction technique. The ligand (L) is crystallized into a monoclinic lattice with a space group of P2₁. The Schiff base ligand (L), $C_{19}H_{25}N_3O_3$, is approximately planar, with a dihedral angle of 9.03 (13)° between the planes of the aromatic rings, and has an E conformation about the N=C bond. In the IR spectral observations, the disappearance of $v_{(O-H)}$ and the lower frequency shift of $v_{(C=N)}$ of the ligand on complexation to the metal atom prove the bonding through imine nitrogen and deprotonate phenolic oxygen. The metal complexes 1 and 2 show strong Ligand to Metal Charge Transfer (LMCT) transition in the visible region (380-450 nm). The Schiff bases (L) behave as monobasic bidentate ligands in their complexes 1 and 2. The spectral data indicate that the ligand (L) coordinate through the phenolic oxygen and the azomethine nitrogen atoms. The observed A_{\parallel} values in the complexes 1 and 2 indicate a tetrahedrally distorted square planar structure. From the study of catalytic activity, the complex 1 of 0.80 equiv exhibits higher activity as catalyst in acylation reaction with 2.0 equiv of acetic anhydride at room temperature.

Keywords: Schiff base;metal complex; crystal structure; Intermolecular hydrogen bond; catalytic activity.

INTRODUCTION:

Schiff bases represent a versatile and diverse class of ligands for transition metal complexes. The ligands are typically prepared from the condensation reaction of either an aldehyde or ketone with a primary amine and, as such, it is possible to introduce a wide variety of steric and electronic features into their structure. Schiff bases could be bidentate, tridentate, tetradentate or poly-dentate ligand capable of forming very stable complexes with transition metals. They can only act as coordinating ligand if they bear a functional group, usually the hydroxyl, sufficiently near the site of condensation in such a way that a five or six member ring can be formed when reacting with a metal ion ^[1-4]. Traditional Schiff bases act as bidenatate N-O donors to a single metal centre, however, there are additional examples in which, the ligand may act as a tridentate N,O,O-, N,O,N-, N,O,S- or tetradentate N.N.O.O- donor ligand permitting the synthesis of multinuclear complexes, including one, two and three-dimensional metal organic frameworks. Therefore, Schiff base ligands have found widespread applications in catalysis, electrochemistry, nanotechnology and materials science as potential therapeutic agents ^[5-7]. Remarkably, the medicinal properties of transition metal complexes depend on the nature of the metal ions and the ligands ^[8-10]. The metal ions present in the complexes not only accelerate the drug activity, but also increase the effectiveness of the organic ligands [11-12].

Schiff base ligands have found their principal application in oxidation/reduction reactions as well as catalysts for hydrolysis. However, including hard donor ligands into the coordination sphere of soft metal centres raises the possibility of introducing unusual electronic features at a given metal and thus, imparting novel catalytic properties compared to more traditional ligands for low oxidation

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state metals such as phosphines, alkenes and carbon monoxide ^[13-15]. They can only act as a coordinating ligand, if they bear a functional group usually the hydroxyl sufficiently near the site of condensation in such a way that a five or six member ring can be formed, when reacting with a metal ion. Schiff bases derived from aromatic amines and aromatic aldehydes have a wide variety of applications in many fields, e.g. biological, inorganic and analytical chemistry. Applications of many new analytical devices require the presence of organic reagents as essential compounds of the measuring system ^[16]. In the present study, Schiff base ligand (L) and their metal complexes 1-2 has been synthesized and characterized by elemental analysis (C, N, and O) and spectral (IR, UV, ¹H NMR, EPR) studies. Also, the crystal structure determination of Schiff base ligand (L) and catalytic activity of complex 1 has been reported in this paper.

EXPERIMENTAL METHODS

The instruments used for various physical measurements are XRD, NMR, IR, UV-Vis, and EPR. *Preparation of 5-diethylamino-2-({[4-(dimethylamino) phenyl] imino} methyl)phenol (L)*

To an ethanol solution (10 ml) of 5-(diethylamino)-2-hydroxybenzaldehyde (96.5 mg, 0.5 mol) is added N^1,N^1 -dimethylbenzene-1,4-diamine (68 mg, 0.5 mol). The mixture is stirred and 2 to 3 drops of glacial acetic acid are added. Stirring is continued for 30 mins and then, the reaction mixture is refluxed for 2 hrs. On completion of the reaction, monitored by TLC, the mixture is allowed to cool to room temperature and the solid precipitate, formed is filtered, dried and recrystallized from DMF for giving colourless block-like crystals (**Scheme 1**).



Scheme 1

Synthesis of complexes 1 and 2

The complexes **1** and **2** are prepared using the methanolic solution (10 mL) of the Schiff base (1 mmol) and the metal acetate (0.5 mmol) (10 mL) is mixed thoroughly and boiled under reflux for 4-6 hrs. Then it is cooled to room temperature. The precipitate formed is filtered, dried and weighed (Scheme 2).



RESULTS AND DISCUSSION:

The analytical data of the **1-2** complexes indicate 2:1 ligand to metal stiochiometry. The present investigation has revealed that the ligand (L) readily form complexes with acetates of Cu(II) and Ni(II). The synthesized complexes are found to be sufficiently soluble in ethanol, chloroform, acetonitrile, acetone and hexane for spectral measurements. Analytical and physical data of all the metal complexes **1-2** are given in **Table 1** and they are in good agreement with the proposed composition.

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Com	Empirical formula	formul	Meltin	Yiel		Elemental analysis(found)			
pound		a weight	g Point (°C)	d (%)	Color	С	Н	Ν	0
L	C ₁₉ H ₂₅ N ₃ O	311.2	160	55	Colour less	73.2 8	8.09	13.49	5.14
						73.2 2	8.23	13.35	5.21
1	Cu(C ₁₉ H ₂₅ N ₃ O)	374.5	210	42	Brown	69.3 9	9.54	15.23	6.12
						69.1 2	9.32	15.21	6.17
2	Ni(C ₁₉ H ₂₅ N ₃ O)	369.7	220	36	-	73.1 8	8.21	13.45	5.19
					Green	73.1 3	8.29	13.21	5.24

 Table 1 Analytical and physical data of ligand (L) and complexes 1-2

CRYSTAL STRUCTURE OF SCHIFF BASE LIGAND (L):

Intensity data collection:

Accurate unit cell parameters and orientation matrix are obtained by a least-square fit of several high angle reflections in the ranges $2.4^{\circ} < \theta < 27.2^{\circ}$ for the title compound, using Mo K α radiation on Bruker Axs Kappa APEXII CCD diffractometer. The unit cell parameters are determined from 4428 reflections for the compound. The data reduction and cell refinement are carried out with the help of APEXII and SAINT (Bruker 2004) ^[17-19].

The intensities of a total of 13009 reflections are collected, resulting in 3824 independent reflections. Of which, 2438 reflections have satisfied the condition $I > 2\sigma$ (I) for the compound and this is considered as observed. The intensities of the compound are corrected for Lorentz and polarization effects and also for absorption using SADABS software (Sheldrick 2008).

STRUCTURE SOLUTION AND REFINEMENT:

The crystal structure of the Schiff base ligand (L) is solved by direct method procedure using SHELEXS97 (Sheldrick 2008) program. The E-map estimated for the best phase set generated by the program has revealed the positions of all the non-hydrogen atoms of the compound. The residual factor calculated on the point atom model (R_E) is 0.205. The trial structure, thus obtained is refined by full matrix least square technique on F² using SHELXL97 (Sheldrick 2008) for the title compound. In the initial stages of refinement, thermal parameters are assigned a value of 0.05 for each atom and the refinement was carried out. The H atoms are positioned geometrically and refined using riding model: O—H = 0.82 Å, C—H = 0.93-0.97 Å with U_{iso} (H) = 1.5 U_{eq} (O,C) for the hydroxyl and methyl H atoms and 1.2 U_{eq} (C) for other H atoms. The components of the anisotropic displacement parameters of the atoms in bonds N3—C16, N3—C18 and N1—C2 are restrained to be equal within an effective standard deviation of 0.001 using the DELU command, and the C16—C17 bond distance was restrained to 1.54 (1) Å ^[20].

Inter and Intra molecular Interactions:

In crystalline state, the molecules are stabilized by intramolecular and intermolecular interactions like hydrogen bonds, van der Waal's forces and possibly some short contacts between the two atoms. Hydrogen bonding is the specific type of non-bonded interaction

between two electronegative atoms (donor and acceptor) where the hydrogen atom is bonded to them.



Fig. 1 The molecular structure of title compound showing 30% probability displacement ellipsoids



Fig. 1a Crystal structure and unit cell packing diagram for Schiff

base(L)

The usual convention for the representation of the hydrogen bond is D-H...A where D is the donor and A is the acceptor. In the title compound, the crystal structure is stabilized by weak intramolecular O—H...N hydrogen bond. The crystal structure is influenced by weak C—H... π interactions to form sheets parallel to the bc plane.

Fig. 1 shows ORTEP plot of the molecular structure of title compound with the atom–numbering scheme. Displacement of ellipsoids is drawn at 30% probability level. H atoms are presented as small spheres of arbitrary radius. **Fig. 1a** shows crystal structure and unit cell packing diagram of Schiff base (**L**). The intramolecular hydrogen bond is depicted by a dashed line. The geometric parameters are comparable to those reported for similar structures. The dihedral angle between the benzene rings (C3–C8) and (C10–C15) is 9.03 (13)°. The molecular structure is stabilized by an intramolecular O–H…N hydrogen bond, with an S(6) ring motif. The title Schiff base compound, $C_{19}H_{25}N_3O$, is approximately planar, with a dihedral angle of 9.03 (13)° between the planes of the aromatic rings and it has an E conformation about the N= C bond. **Table 2** summarizes the crystal data, intensity data collection and refinement details of the compound.

Table 2 Summary of crystal data, intensity data collection and refinement details for the Schiff base ligand (L)

Parameter	Compound
Empirical formula	$C_{19}H_{25}N_{3}O$
Formula weight	311.42
Temperature	295 K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, $P2_1$
	Hall symbol: P 2yb
	a = 8.8201 (7)Å
Unit cell dimensions	b = 7.8850 (7)Å
	c = 13.0639 (10)Å
	$\beta = 108.407 (3)^{\circ}$
Z	2
Volume	$V = 862.06 (12)A^3$
	1 200 Ma == -3
Absorption coefficient	1.200 Mg m ⁻¹
Absorption coefficient	$\mu = 0.08 \text{ mm} - 1$
F(000)	330
Crystal size	$0.26 \times 0.22 \times 0.20 \text{ mm}$
Theta range for datacollection	24.272°
č	2.4-21.2°
T • •• • •	$h = -11 \rightarrow 10$
Limiting indices	$k = -10 \rightarrow 10$

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	$l = -16 \rightarrow 16$
Reflections collected	29557
Independent reflections	$3825[R_{(int)} = 0.027]$
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3825/6/214
Goodness-of-fit on F ²	1.03
R-indices (all data)	$R_1 = 0.052, wR_2 = 0.159$
Largest diff. peak and hole	0.29 and -0.16e Å ⁻³

H¹NMR Spectra

The ¹H NMR spectral data of the Schiff base ligands (L) are listed in the **Table 3**. The ¹H NMR spectra of the ligand is recorded in CDCl₃ solvent. The ¹H NMR spectra of the Schiff base ligand (L) is shown in the Fig. 2. Singlet peaks observed in the ¹H NMR spectra of the ligand (L) at 13.979 ppm are due to phenolic (-OH). The characteristic signal at 8.632 ppm are assigned to-HC=N for ligand (L). The ¹H NMR spectra of the Schiff base indicate the presence of the aliphatic protons in the range of 2.49-3.36 ppm as singlets. The aromatic proton of free ligand is observed as multiplets in the aromatic region (6.32-7.27 ppm).



Fig. 2 ¹H NMR spectrum of Schiff base (L) in CDCl₃ Table 3 ¹H NMR spectral data of the ligand (L) (δ in ppm)

Compound	U (О-Н)	U(CH=N)	Aromatic	Aliphatic
\mathbf{L}	13.979	8.632	6.038-6.761(m,4H)	1.107-2.501(m,7H)
			7.208-7.273(m,3H)	2.501-3.632(m,7H)

Infrared spectra

The IR spectral data of the ligand (L) and their metal complexes 1-2 are given in Table 4 and Fig. 3 **a-c**, respectively. IR spectra of the ligand (L) show absorption around 3444 cm⁻¹ with medium intensity due to $v_{(OH)}$. The IR spectra of the Schiff base free ligand (L) show strong band at 1623 cm⁻ ¹, due to $v_{(C=N)}$ of imine, which indicates that the Schiff base ligand has been obtained. In metal complexes 1-2, azomethine band $v_{(C=N)}$ is shifted to lower frequencies (1608-1612 cm⁻¹) after complexation ^[21-24]. The strong bands at 1272-1290 cm⁻¹ in the free schiff base ligand (L) is due to the phenolic C-O stretchings. On complex formation, these bands shift to lower frequencies (1164-1182 cm⁻¹) by indicating the coordination through phenolic oxygen. Bands due to M-N appear in the 644-657 cm⁻¹ and M-O appears in the region 530-561 cm⁻¹ [25-27].





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Fig. 3a IR spectrum of ligand (L)

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Fig. 3b IR spectrum of complex 1



Table 4 IR data of ligand (L) and its complexes 1-2

Compound	D(C-M)	U (C-	υ	U (М-
compound	O(C=N)	O)	(M-N)	O)
\mathbf{L}	1623	1290	-	-
1	1608	1164	644	530
2	1612	1182	648	561

Fig. 3c IR spectrum of complex 2

Electronic spectra

The UV-Vis spectra are recorded in CHCl₃ solutions in the range of 800-200 nm for the Schiff base and their complexes 1-2 (Fig. 4(a-b)). The molar absorbtivities for ligand (L) at 350-335 nm may be assigned to $n-\pi^*$ transition between the lone-pair electrons of the p orbitals of the N atoms in the azomethine (HC=N) groups and the π bonds of the aromatic rings [24, 28]. The peaks at 263 and 280 nm are assigned to the $\pi-\pi^*$ and $n-\pi^*$ transitions of the Schiff base (L). The lower intensity absorption bands in the range 360-450 nm are assigned to ligand–metal charge transfer for complexes 1-2 (LMCT) [29]. The ligand field spectra of the complexes 1 and 2 in CHCl₃ show a band around 655-700 nm, due to a combination of ${}^{2}B_{1g} \rightarrow {}^{2}A_{1g}$ and ${}^{2}B_{1g} \rightarrow {}^{2}B_{2g}$ transition reveals square planar geometry [^{30-33]}.



Fig. 4a Electronic spectrum of complex 1 in CHCl₃ Fig. 4b Electronic spectrum

of complex 2 in CHCl₃



EPR Spectra

The X-band EPR spectra of complex **1** is recorded in acetonitrile solution at liquid nitrogen temperature. The EPR spectral parameters derived from their respective spectra are presented in **Table 5**. A representative EPR spectrum of complex **1** is given in **Fig. 5**. The EPR spectra of complex in solution exhibit a set of four well resolved peaks in the high field region. The "g" tensor values of the complex can be used to derive the ground state ^[34-36]. The g_{\parallel} values observed are in the range (2.2522 - 2.2545) for complexes **1** whereas $g_{\perp are}$ (2.0751 - 2.0820) shows $g_{\parallel} > g_{\perp}$. This indicates that the unpaired electron occupies the $d_{x^2-y^2}$ orbital with ²B_{1g} ground state which leads to axially elongated square planar structure. However, in a compressed octahedron, the unpaired electron lies predominantly in the $d_{x^2-y^2}$ orbital. The quotient $g_{\parallel} / A_{\parallel}$ is empirically treated as a measure of the distortion from planarity, the observed values of (155–160) for copper(II) complexe **1**, clearly indicates a slight tetrahedral distortion in their structure.

		Compounds	gli	g⊥	A (x10 ⁻ ⁴ cm ⁻ ¹)	A⊥ (x10 ⁻ ⁴ cm ⁻ ¹)	g∥ /A∥
NV.	Fig. 5 EPR	1	2.2522	2.0751	140	75	160
e na V sobo sobo sobo ebi Magnaric field	Spectrum of						

Table 5 EPR spectral data of complex 1

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CATALYTIC ACTIVITY:

Acylation is usually carried out by treating alcohols or thiols with acetic anhydride or acetyl chloride in the presence of acid or base catalyst in suitable organic solvent. Acetic anhydride is most commonly used due to low toxicity ^{[37]a-c}. The most efficient base catalysts for acylation of alcohols are 4-(dimethylamino) pyridine (DMAP) and phosphines. Various acidic catalysts employed include CoCl₂, RuCl₃, nitrobenzenboronic acid, Sc(OTf)₃, Al(OTf)₃, Cu(OTf)₂, LiClO₄, Cu(ClO₄)₂, and N-heterocyclic carbine [38]a-k. Apart from some operational disadvantages such as moisturesensitivity and high cost of catalyst, numerous methods employ perchlorates which are explosive ^[39] or toxic metal derivatives for the catalyst. Most of the work takes the advantage of chlorinated hydrocarbon as solvent. Accordingly simple, mild and efficient catalytic method without the help of metal and solvent is desirable.

ACYLATION OF BENZYL ALCOHOL WITH ACETIC ANHYDRIDE BY COMPLEX 1

In order to establish the optimized reaction condition, the present study has been carried out using benzyl alcohol as a model substrate in the presence of various amount of complex 1 (Table 6) and it is shown in (Scheme 3). Initially, 0.20 equiv of metal complex 1 and acetic anhydride give only 40% of product within 120 min, while 0.80 equiv of metal complex 1 yields 94 % in 30 min reaction time. Neither increasing metal complex 1 amount from 0.40 to 0.80 equiv nor decreasing the reaction temperature to 0 °C affects the catalyst performance. The use of 1.0 equiv of acetic anhydride gives somewhat lower yield of 40 % in quite longer reaction time (40 min). Accordingly, the catalyst loading of 0.80 equiv with 2.0 equiv of acetic anhydride at room temperature can be regarded as optimal condition for the acylation as shown in Table 7, (entry 4).



Investigations of the scope of the substrates are conducted under the optimized condition as mentioned above and the results are given in Table 8. Various benzyl alcohols with electrondonating and electron-withdrawing substituents undergo acylation in quite high yield (entries 2-7). Halogenated benzyl alcohols such as p-flouro and p-bromobenzyl alcohol produce acylated products (entries 5 and 6). This indicates that electronic effect of halogen atom may have little influence on the acylation. Cinnamyl alcohol is able to produce 80 % yields of the acetates in 40 min reaction time, (entries 7). This procedure is also applicable to secondary aromatic alcohol with "relatively" longer reaction time of 50 min (entry 8).

Though the mechanism of this reaction not clears at this stage, the possible mechanism for this catalytic study is lewis acidic nature of copper complex which activates the subatrate. When compared with other catalytic systems ^[40-42], it is clear that present catalytic method needs relatively less amount of catalyst (0.80 mmol) without any additive at room temperature under solvent-free condition in comparatively shorter reaction time.

Keter Table /							
Entry	Substrate	Time (min.)	Yield (%) ^a				
1	Benzyl alcohol	30	90				
2	Methoxy	35	88				
3	Methyl	35	85				
4	Nitro	30	85				

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5	Flouro	40	90
6	Bromo	40	88
7	ОН	40	80
8	OH	50	81

^a Isolated yield

ACYLATION OF ALCOHOLS

Complex 1 (0.8mmol) is added to the mixture of alcohol (1.0 mmol) and acetic anhydride (2.0 mmol). The reaction mixture is stirred at room temperature for the appropriate time **Table 7**. The reaction is monitored by TLC. Water is added after the completion of the reaction that has extracted with ethylacetate (3×20 mL). The organic phase is dried over Na₂SO₄ and concentrated for purification by column chromatography with silica gel. Representative datas are given and the corresponding NMR spectra also given. They are shown in **Fig. 6a,b**.

Benzyl Acetate (**Table 8**, entry 1): ¹H NMR (CDCl₃, 200 MHz): δ 2.00 (s, 3H), 5.01 (s, 2H), 7.32-7.37 (m, 5H), ¹³C NMR (CDCl3, 100 MHz): δ 20.94, 66.19, 128.04, 128.06, 128.14, 128.36, 135.76, 170.58.







	Table 6 Acylati	on of benzyl alcohol	with	acetic an	hydride	e by complex 1
7	Catalyst	Acetic anhydride		(•)	T	X7° 11 (0/)9

Entry	(mol %)	Acetic annydride (equiv)	Time (min)	Temp	Yield (%) ^a
1	0.20	2	120	RT	40
2	0.30	2	80	RT	57
3	0.40	2	80	RT	70
4	0.80	2	30	RT	94
5	0.80	1	40	RT	20
6	0.80	2	30	$0^{\circ}C$	90

^a Isolated yield

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In this paper, the synthesis of new Schiff base (L) and their metal complexes 1-2 are reported. The structural characterizations of the synthesized compounds are made by using the elemental and spectroscopic analysis. The X-ray crystallography study confirms the structure of newly synthesised Schiff base (L). The ligand (L) is crystallized into a monoclinic lattice with a space group of P2₁. From the data obtained, tetrahedrally distorted square planar geometry has been assigned for all the complexes. The spectral data show that the Schiff bases act as monobasic bidentate NO chelating agents coordinating the metal ion via the azomethine nitrogen atom and the phenolic oxygen atom. Complex 1 exhibits catalytic activity for acylation of benzyl alcohol with acetic anhydride. It has been concluded that by using 0.80 equiv of complex 1 as catalylst with 2.0 equiv of acetic anhydride at room temperature, can be regarded as optimal condition for the acylation.

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