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Mononuclear copper(II) complex [CuL₂] and two mononuclear mixed ligand palladium(II) complexes of the type [Pd(X)(L)] where HL = 2-(methylthio)phenylimino)methyl)phenol and X = benzoate or salicylate have been synthesized and characterized. Structural data shows both the Pd(II) complexes have distorted square planer geometry whereas there are two molecules in a unit cell in the Cu(II) complex-one molecule has square planer geometry and another molecule has distorted square pyramidal geometry. Tridentate NSO donor ligand acts as bidentate as well as tridentate ligand in the complexes.



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Copper(II) and Palladium(II) complexes with tridentate NSO donor Schiff base ligand: Synthesis, Characterization and Structures

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Abstract

Mononuclear copper(II) complex [CuL₂] and palladium(II) complexes [Pd(X)L] where X= benzoate(bz) or salicylate(sal) and HL = 2-(methylthio)phenylimino)methyl)phenol, a Schiff base ligand with NSO coordination sites have been synthesized and characterized by microanalyses, IR, UV-Visible spectra, conductivity measurement and magnetic studies. Crystal structures of all the complexes have been solved by single crystal X-ray diffraction studies and showed that there are two molecules in a unit cellin the [CuL₂] complex - one molecule has square planar geometry whereas second molecule has distorted square pyramidal geometry and palladium(II) complexes have distorted square planar geometry.

Key words. Mononuclear; copper(II);palladium (II); NSO donor ligand; spectra; structures.

1. Introduction

Schiff bases have been used as versatile chelating ligand in the synthesis of transition metal complexes because of their ease of preparation and structural variation. Schiff base containing transition metal complexes have been investigated extensively because they have wide application in the fields of biology and catalysis [1-10]. Metal complexes with Schiff base containing N and O donor atoms derived from aromatic amine and salicyldehyde have been studied extensively and

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there are many compounds reported in the literature. Transition metal complexes with Schiff base chelating ligand containing hetero N, S and O donor atoms are very interesting because compounds with NSO donor atoms have biologicaland catalytic activity and there are many reports on the catalytic andbioactivity of metal complexes with NSO coordinate ligand [11-17]. There has been considerable interest in palladium complexes with Schiff base ligand because of their potential catalytic and biological activity[18-23]. As properties of the metal complexes depend on the coordination environment around the metal centre, we are interested to see the coordination behaviour of NSO donor ligand in the copper(II) and palladium(II) complexes.

In this paper, we report on the synthesis, characterization and structures of mononuclearcopper(II) complex [CuL₂] and palladium(II) complexes of the type [Pd(X)L] where HL = 2-(methylthio)phenylimino)methyl)phenol, a tridentate N, S and O donor atoms containing ligand and X = benzoate or salicylate.

2. Experimental

2.1.Materials

All chemicals and solvents used were analytical grade reagents. 2-Methylthioaniline,Pd(CH₃COO)₂(Aldrich) and salicyldehyde (Loba) were reagent grade and used as received. 2-(methylthio)phenylimino)methyl)phenol (HL) was synthesized by refluxing the mixture of 2-methylthioaniline and salicyaldehyde in ethanol. Cu(ClO₄)₂.6H₂O was prepared by reaction of cupric carbonate with dilute HClO₄ acid, followed by slow evaporation of the solution.

2.2.. Instruments

The micro analysis (C, H and N) were carried out using a Perkin-Elmer IA 2400 series elemental analyzer. The IR spectra were recorded on a Perkin-Elmer FT-IR spectrometer RX1 spectrum using KBr pellets. UV-Vis spectra (1200- 190 nm) were recorded on a Shimadzu 3600 in CH₃CN solution. Solution conductivity were measured in CH₃CN solution using Equip-Tronics conductivity meter (model no. EQ-660A). Room temperature magnetic susceptibility of powder sample was measured by Guoy balance.

2.3. Syntheses of complexes

2.3.1.Synthesis of complex $[CuL_2]$ (1)

A methanol solution (10 mL) of ligand HL (0.244g, 1.0 mmol) was added drop wise toa stirring methanol solution (10 mL) of Cu(ClO₄)₂.6H₂O (0.185g, 0.5mmol) and stirring was continued for 3 hrs. The solution was filtered and the filtrate was kept for slow evaporation. Dark green colored crystals were obtained from the filtrate after 5 days.

Yield. (0.160g, 50%). Found. C = 61.68, H = 4.40, N = 5.12%. Anal calc for C₂₈H₂₄N₂CuO₂S₂:C = 61.36, H = 4.38, N = 5.11%. IR (KBr pellet) cm⁻¹;v (C = N) 1604 s. UV-Vis spectra: λ_{max} /nm (ε_{max} /mol⁻¹cm⁻¹). 679(127). 398(5969), 231(25563), $\Lambda_{\rm M}$ (Ω^{-1} cm² mol⁻¹) = 14. μ_{eff} = 1.80 BM.

2.3.2.Synthesis of complex [Pd(bz)L] (2)

A solution of ligand HL (0.061g, 0.25mmol) in acetonitrile (10ml) was added drop by drop to a acetonitrile solution (10 mL) of Pd(CH₃COO)₂(0.056 g, 0.25 mmol) with stirring. To this, a solution of benzoic acid (0.25mmol) in acetonitrile (10 ml) was added and stirring was continued for3 hrs. Filtered the solution and the solution was kept for slow evaporation. Orange coloured crystals were obtained from the solution after 5 days.Yield.0.075g (63%). Found C = 53.70, H = 3.60, N = 2.90 %. Anal calc for C₂₁H₁₇NO₃PdS; C = 53.68, H = 3.62, N = 2.98 %. IR (KBr

pellet) cm⁻¹; v(C=N),1606s; v((Pd-N), 479.UV-Vis spectra: $\lambda_{max}/mol^{-1}cm^{-1}$). 447(7061), 317(14110), 242(35420).

2.3.3.Synthesis of complex [Pd(sal)L] (3)

The complex was synthesized by following the similar procedure to that of **2**, but salicylic acid was used in place of benzoic acid. Yield.0.082 g (67 %). Found C = 51.93, H = 3.55, N = 2.78 %. Anal calc for C₂₁H₁₈NO₄PdS; C = 51.87, H = 3.70, N = 2.88 %. IR (KBr pellet) cm⁻¹; (C=N), 1607s; v(Pd-N), 479.UV-Vis spectra $\lambda_{max}/mo(\epsilon_{max}/mol^{-1}cm^{-1})$. 446 (6077), 318 (11812), 227 (35590).

2.4.X-ray crystallography

Crystals for X-ray diffraction of the compounds **1**, **2** and**3**were obtained through slow evaporation of the solution. Single crystal X-ray diffraction intensity measurements were performed on Oxford X-CALIBUR-S diffractometer equipped with CCD detector using Mo-K_a radiation ($\lambda = 0.71073$ Å) for complex **1** and Cu-K_a radiation ($\lambda = 1.54184$ Å) for complexes **2** and **3**. The reflection were measured in the range of 3.71 to 28.09⁰ for complex **1**,4.68 to 88.79⁰ for complex **2**and4.065 to 73.03⁰ for complex **3**. Data reduction was carried out using the program CrysAlisPro, Agilent Technologies, Version 1.171.35.19 [24]. An absorption correction based on multi-scan method was applied [25].The structure was solved by direct methods and refinement was performed by the full-matrix least-square technique on F^2 using the programs SHELXS-97 and SHELXL-97 respectively [26]. All calculations were carried out using WinGX system Ver-1.64 [27]. All hydrogen atoms were refined with anisotropic displacement coefficients.

Table 1. Crystal data for the complexes1, 2 and 3

	$[CuL_2]$	[PdL(bz)]	[PdL(sal)]	
Empirical formula	$C_{42}H_{36}Cu_{1.5}N_3O_3S_3\\$	C ₂₁ H ₁₇ N O ₃ PdS	C ₂₁ H ₁₇ NO ₄ PdS	
Formula weight	822.23	469.82	485.848	
Temperature (K)	293(2)	293(2)	293(2)	
Wavelength (Å)	0.71073	1.54184	1.54184	
Crystal system	Monoclinic	Monoclinic	Monoclinic	
Space group	P21/n	P21/c	$P2_{1}/c$	
a (Á)	11.1682(4)	9.5640(8)	9.1817(3)	
b (Å)	21.9010(8)	10.5093(10)	10.71128(5)	
c (Á)	15.3682(6)	19.0108(16)	19.5035(8)	
α (°)	90.00	90.00	90.00	
β (°)	100.893(4)	96.383(9)	98.257(4)	
γ (°)	90.00	90.00	90.00	
Volume (Å ³)	3691.3(2)	1859.2(3)	1898.51(13)	
Z	4	4	4	
Density (Mg/m ³)	1.480	1.678	1.700	
Absorption coefficient	1.087	9.284	9.156	
(mm ⁻¹)				
F(000)	1698	944	976	
Theta range for data	3.7140 to 28.0900	4.68 to 88.79	4.065 to 73.03	
collection (°)				
Index ranges	-13≤h≤14,	-11≤h≤11,	-11≤h≤11,	
	-29≤k≤29,	-5≤k≤13,	-13≤k≤12,	

	-19≤1≤20	-23≤l≤2	-23≤l≤24
Reflections collected	26416	6158	10010
Independent reflections	8683	3730	3793
	[R(int) = 0.0299]	[R(int) = 0.0623]	
Absorption correction	Semi-empirical form	Semi-empirical form	Semi-empirical form
	equivalents	equivalents	equivalents
Max. and min.	0.78729 and 1.0000	1.00 and 0.625	1.00 and 0.3225
transmission		Ś	
Refinement method	Full-matrix least-squares	Full-matrix least-squares	Full-matrix least-
	on F^2	on F^2	squares on F^2
Data/ restraints/	8683/ 0 / 487	3730/0/248	3793/0/257
parameters			
Goodness-of-fit on F^2	1.052	1.152	1.047
Final R indices	RI = 0.0412,	R1 = 0.0532,	R1 = 0.0751,
[I>2sigma(I)]	wR2 = 0.0919	wR2 = 0.1431	wR2 = 0.1990
R indices (all data)	R1 = 0.0638,	R1 = 0.0532,	R1 = 0.0826,
	wR2 = 0.1019	wR2 = 0.1431	wR2 = 0.2132

3. Results and Discussion

3.1. Synthesis

Mononuclear copper(II) complex [CuL₂] was readily obtained with good yield (~50%) by the reaction of Cu(ClO₄)₂.6H₂O with the ligand HL in 1:2 mole ratio in methanol at room temperature [scheme 1]. The molecular composition of the complex was supported by micro

analyses, IR and solution conductivity measurement. Molar conductivity measurement of the complex in CH₃CN solution (~10⁻³M) shows that the complex has very low molar conductivity $(\Lambda_M \sim 10 \ \Omega^{-1} \text{cm}^2 \text{ mol}^{-1})$, indicating the complex isneutral and no counter anion is present. Infrared spectrumof the complex also indicate the absence of ClO₄⁻ ion in the complex.Single crystal X-ray diffraction study of the copper(II) complex shows there are two molecules in the unit cell-one molecule has square planar geometry where the ligand acts as bidentate and another molecule has distorted square pyramidal geometry where one ligand acts as bidentate ligand with N,O coordination mode but another ligand acts as tridentate with N, S and O coordination mode.The structure and bioactivies of the ternary five coordinated copper(II) complexes with this NSO donor ligand and nitrogen containing heterocycle ligand is reported in the literature[11, 28-29]. The complex is moderately soluble in organic solventssuch as acetonitrile, methanol, ethanol, dichloromethane, acetone etc.



Scheme 1. Synthesis of the complexes

Mononuclear palladium(II) complexes of the type [Pd(X)L] were readily obtained in good yield (65%) by the reaction of $Pd(CH_3COO)_2$, ligand HL and HX (where HX= salicyclic acid / benzoic acid) in 1 : 1 : 1 mole ratio in acetonitrile at room temperature. Orange coloured crystals

of [Pd(X)L] were obtained by slow evaporation of acetonitrile solution of the complexes. Both the complexes have slightly distorted square planar geometry. The Schiff base ligand L acts as tridentate monoprotic NSO coordinate ligand in both the complexes. The microanalysis data of the two complexes are found to be consistent with their molecular compositions. There is no change in composition even after adding excess benzoic / salicylic acid. Molar conductivity measurements in CH₃CN solution show the complexes are neutral. The complexes are soluble in common organic solvents like dichloromethane, methanol, acetonitrile etc.

3.2.IR data

The IR spectra of the complexes were assigned by comparing the IR spectra of the ligand HL. The IR spectrum of all the complexes exhibit one strong band at 1604 cm⁻¹ correspond to v(C=N) of imine andthe absence of ligand's phenolic –OH band in the region of 3320 cm⁻¹ in the compounds confirming the coordination of phenolic-OH with metal centre in the complexes.Infrared spectra of both complexes **2**and **3** are nearly identical.For both the compounds, one weak band exhibited at479cm⁻¹ due to v(Pd-N)[30] and one strong absorption band exhibited at 1376 and 1335 cm⁻¹ for the complexes **2** and **3** respectively, due to asymmetric stretching vibration of monodentate –COO⁻group confirming the coordinate of the palladium(II) ions.For [Pd(sal)L], the phenolic-OH is not coordinate only through oxygen atom of carboxylate ion. This was confirmed by single crystal X-ray diffraction study of the complex **3**. This type of non-coordination of phenolic-OH group in the complexes was reported in the literature [31].Finally, coordination of ligand with metal atoms were confirmed by single crystal X-ray diffraction studies.

3.3. UV-Vis spectra and Magnetic data

The UV-Vis spectra of both the complexes were recorded in the range of 200-1100 nm in CH₃CN. A broad absorption band observed at 679 nm with $\varepsilon = \sim 131 \text{ mol}^{-1} \text{ cm}^{-1}$ for complex **1** which is attributed to d-d transition. For palladium(II) complexes, both the complexes show a band at ~446 nm with high molar extinction coefficient (>6000 mol⁻¹cm⁻¹) and it may be due to charge transfer transitions. The higher energy bandsat 398 nm for copper(II) complex and at 318 nm for palladium(II) complexes with higher molar extinction coefficient values are due to charge transfer (LMCT) transition. The other high energy intensity transitions at 227 and 247 nm are due to the intra ligand π - π * and n- π * transition.

3.4. Description of crystal structures

3.4.1. Crystal structure of [CuL₂]

The ORTEP diagram of complex $[CuL_2]$ with atom-lebeling scheme is shown in Fig. 1. The Xray crystal structure confirms that complex 1 crystallise in monoclinic crystal system with centro symmetric space group *P21/n*. In the asymmetric unit, there are two independent Cu(II) centers [Cu(1) and Cu(2)] are present and the bonding of the Cu(1) center is slightly different from Cu(2). In Cu(1), ligand behaves as a bidentate ligand with N and O coordination site and bonded through nitrogen atom of the immino nitrogen atom and oxygen atom of the phenol and Cu(1) atom adopts square planar geometry. The equatorial bond distances of Cu(1)–N(1) [1.985(2) Å], Cu(1)–N(1)i [1.985(2) Å], Cu(1)–O(1) [1.922(18) Å]and Cu(1)–O(1)i [1.922(18) Å] are nearly equal and two sulfur atoms of the ligand remain uncoordinated. The bond angles O(1)-Cu(1)-O(1)i and N(1)-Cu(1)-N(1)i are 180.0° and O(1)-Cu(1)-N(1) and O(1)-Cu(1)-N(1)i are 89.26(8) and 90.74(8)°, respectively. In Cu(2), copper atom has CuN₂O₂S coordination environment and adopts distorted square pyramidal geometry [$\tau = 0.10$] and two L ligands show two different coordination modes- one behave as bidentate ligand with nitrogen and oxygen coordination sites similar to Cu(1) whereas another ligand behave as tridendate with nitrogen, oxygen and sulfur coordination sites. In the case of Cu(2) center, the equatorial bond distances of Cu(2)–N(2), Cu(2)–N(3), Cu(2)–O(2) and Cu(2)–O(3) are 2.008(2), 1.999(19), 1.911(16) and 1.917(17) Å respectively, are nearly equalbut has long axial Cu(2)–S(3) [2.859(2) Å]bond. Similar long Cu-S bond distance in Cu(II) complex with NSO donor ligand is reported in the literature.[11].The bond angles O(2)-Cu(2)-N(3), O(3)-Cu(2)-N(2), O(2)-Cu(2)-O(3), O(3)-Cu(2)-N(3), O(2)-Cu(2)-N(2), N(2) and N(3)-Cu(2)-N(2) are 158.64(8), 153.12(8), 89.38(7), 91.81(8), 91.17 and 97.24(8)° respectively.



Fig.1. ORTEP diagram depicting the cationic part of the complex **1** with atom numbering scheme (30% probability factor for the thermal ellipsoids).

Table 2. Selected bond lengths (Å) and angles $(^{0})$ of complexes

Bond lengths (Å)

[Cu	L_2]	[Pd(I	bz)L]	[Pd(s	al)L]
Cu(1)–O(1)	1.922(18)	Pd(1)-O(3)	2.013(9)	Pd(1) - O(1)	1.987(5)
Cu(1)–O(1)i	1.922(18)	Pd(1)-N(1)	2.019(7)	Pd(1) - N(1)	1.991(5)
Cu(1)–N(1)	1.985(2)	Pd(1)-O(1)	2.048(7)	Pd(1) - O(2)	2.043(4)
Cu(1)–N(1)i	1.985(2)	Pd(1)-S(1)	2.245(3)	Pd(1) - S(1)	2.248(18)
Cu(2)–O(2)	1.911(16)				
Cu(2)–O(3)	1.912(17)			5	
Cu(2)–N(3)	1.999(19)				
Cu(2)–N(2)	2.008(2)				
Cu(2)-S(2)	2.859(5)				
Bond angels (⁰))		Y		

Bond angels (⁰)

CuL_2		[PdL(ba)]		[PdL(sal)]	
O(1)-Cu(1)-O(1)i	180.0	O(3)-Pd(1)-N(1)	95.8(4)	O(1) - Pd(1) - N(1)	94.2(2)
O(1)-Cu(1)-N(1)	90.74(8)	O(3)-Pd(1)-O(1)	86.6(4)	O(1) - Pd(1) - O(2)	86.6(2)
N(1)-Cu(1)-N(1)i	180.0	N(1)-Pd(1)-O(1)	177.6(4)	N(1) - Pd(1) - O(2)	178.9(2)
O(3)-Cu(2)-N(2)	153.12(18)	O(3)-Pd(1)-S(1)	178.0(2)	O(1) - Pd(1) - S(1)	178.28(13)
N(3)-Cu(2)-N(2)	97.24(8)	N(1)-Pd(1)-S(1)	86.1(3)	N(1) - Pd(1) - S(1)	87.34(16)
O(2)-Cu(2)-N(2)	91.17(8)				
O(3)-Cu(2)-N(3)	91.81(8)				
O(2)-Cu(2)-O(3)	89.38(7)				
O(1)-Cu(1)-N(1)	89.26(8)				

3.4.2. Crystal structures of complexes2 and3.

The ORTEP view with atom numbering scheme of complexes 2 and 3 are shown in the Fig. 2 and 3. Complexes 2 and 3 are iso-structural, consisting of monomeric palladium centre with slightly distorted square planer geometry. Both the complexeswere crystallized in the monoclinic crystal system with $P2_{1/c}$ space group. The ligand HL act as a tridentate NSO donor ligand and coordinated to metal centre via one nitrogen atom N(1) of imine group, one oxygen atom O(3) of phenolic OH group and one sulphur atom S(1) of $-SCH_3$ group. For both the complexes, the coordination sphere around Pd(II) centre can be described as square planer, with PdNO₂S coordination environment, comprises of NSO coordination sites from ligand L and one oxygen atom from -COOH of benzoic or salicyclic acid. Ligand L forms two chelate rings with the palladium metal centre - one six member ring comprising Pd-O(3)-C(8)-C(13)-C(14)-N(1)-Pd and one five member ring with Pd-S(1)-C(20)-C(15)-N(1)-Pd.The Pd-N distances are similar to the other reported Pd(II) compounds [32]. For the complex 2, the equatorial bond lengths of Pd(1)-O(1), Pd(1)-N(1)and Pd(1)-O(3) 2.048(7),2.019(5),are and



Fig.2. ORTEP diagram of the complex **2** (30% probability factor for the thermal ellipsoids, Hatom are omitted for clarity).

2.013(4) Årespectively are nearly equal and shorter thanPd(1)-S(1) [2.245(3)] bond length. The bond angles N(1)-Pd(1)-O(2) [178.9(2)⁰] and O(1)-Pd(1)-S(1) [178.28(13)^o] are close to 180° .



Fig.3. ORTEP diagram of the complex **3** (30% probability factor for the thermal ellipsoids, Hatom are omitted for clarity).

For the complex **3**, the equatorial bond length of Pd(1)-S(1) [2.244(3) Å]is longer than other three equatorial bonds i.e.Pd(1)-O(2) [2.043(4)Å], Pd(1)-N(1)[1.991(5)Å], Pd(1)-O(1) [1.987(5)Å]but the bond angles of N(1)-Pd(1)-O(1) and O(3)-Pd(1)-S(1) are 177.6(4) and 178.0(2)°, respectivelyare close to 180° . In both the palladium complexes, Pd-S bond lengths are nearly equal (~2.248Å)but much longer than other three bond lengths in the equatorial plane and produce distortion in the molecules. The Pd-S bond distance is much shorter than Cu-S bond distance but similar to other Pd-S bond distance reported in the literature [32].

4. Conclusion

We have synthesized mononuclear copper(II) complex $[CuL_2]$ and two palladium(II) complexes [PdL(bz / sal)] using tridentate NSO coorinate Schiff base ligand HL and

characterized.Structural datashowed palladium complexes have distorted square planar geometry and the ligand behave as tridentate NSO donor ligand whereas it behaves as tridentate NSO as well as bidentate NO donor ligand in the two copper(II) complexes with square pyramidal and square planar geometry. The Cu-S bond length is much longer than Pd-S bond.

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Appendix A. Supplementary Materials

CCDC 1445396, 1046793 and 1472158 contains the supplementary crystallographic data for for complexes **1**,complex **2** and complex **3** respectively. These data can be obtained free of charge via <u>http://www.ccdc.cam.ac.uk/conts/retrieving.html</u>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e. mail: deposit@ccdc.cam.ac.uk

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- Synthesis and characterization of mononuclear Cu(II) and Pd(II) complexes.
- NSO donor ligand shows two different coordination modes in the copper complexes.
- One copper complexes have square planar and another has square pyramidal geometry.
- In palladium complexes, both Pd atoms have distorted square planar geometry.
- Pd-S bond lengths are shorter than Cu-S bond length.