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Syntheses, characterization, spectral properties, and catalytic activity of azo amido complexes of copper(II)

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Research Article

Keywords: 1N-(N,N-diethylaminoethyl)-2-arylazoaniline, Copper(II) complex, Crystal structure, Emission, Oxidation reaction

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Syntheses, characterization, spectral properties, and catalytic activity of azo amido complexes of copper(II)

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Abstract

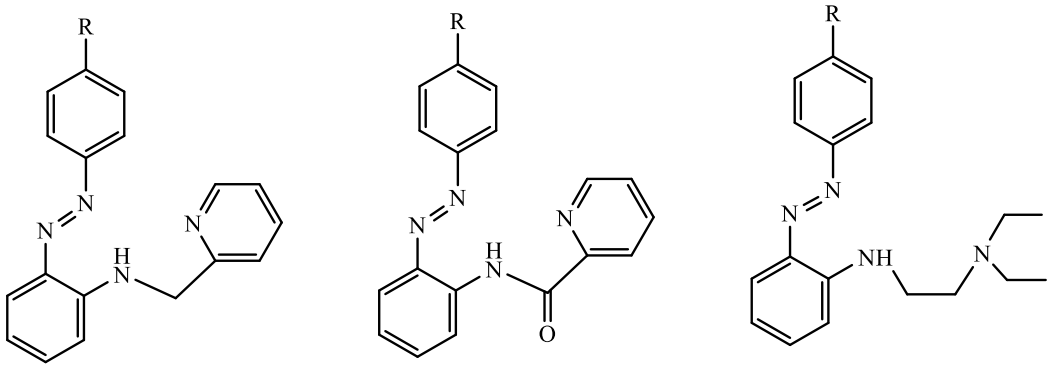
A potentially terdentate ligands 1N-(N,N-diethylaminoethyl)-2-arylazoaniline HL, [where HL = ArN=NC$_6$H$_4$N(H){C$_2$H$_4$N(C$_2$H$_5$)$_2$}; Ar = C$_6$H$_5$ (for HL^1, **3a**) or *p*-MeC$_6$H$_4$ (for HL^2, **3b**) or *p*-ClC$_6$H$_4$ (for HL^3, **3c**)] have been used, in its deprotonated form, to prepare a copper(II) complexes which has been structurally characterized. The ligands (L$^-$) afford a mononuclear complex Cu(L)Cl (**4**). In complex (**4**), (L$^-$) binds to the copper(II) center in a terdentate (N,N,N) fashion. The Cu^{II}N(azo)N$^{/}$(amido)N$^{//}$(amine) and Cl coordination environment is severely distorted from ideal square planar geometry. The Cu-N(amido) bond length is shortest and Cu-N(amine) bond length is larger. The distorted phenyl ring adjacent to the six-membered azo-amido chelate ring consist four longer and two shorter bonds indicates the delocalization. The delocalization of negative charge of the anionic ligands that formed *in situ* from the precursor (L$^-$) due to dissociation of an amino proton. The emission property of copper(II) complexes was examined in dichloromethane solvent. Cyclic voltammetry of **4c** complex shows a Cu(II)–Cu(I) oxidation at 0.68 V vs. SCE, followed by an oxidation of the coordinated ligand 3c at 1.01 V vs. SCE. Two reductions of the coordinated 1N-(N,N-diethylaminoethyl)-2-arylazoaniline ligand **3c** are observed – 1.14 V and – 0.65 V vs. SCE. The catalytic activity of complex **4a** has been investigated in the oxidation of benzyl alcohol by using H_2O_2 as oxidant. The effects of two influential parameters in catalytic reactions, amount of catalyst and temperature have been studied to obtain the optimum condition.

Keywords: 1N-(N,N-diethylaminoethyl)-2-arylazoaniline; Copper(II) complex; Crystal structure; Emission; Oxidation reaction.

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Introduction

This work originates from the binding mode of the extended tridentate N,N,N-donor ligands of 2-(arylazo)aniline, **1-3**. These ligands were synthesized by the reaction of 2-(arylazo)aniline with appropriate alkyl halides. The ligands **1-3**, upon deprotonation, acts as potential ligands for the transition metal ions [1-6]. In our previous communication we described about palladium(II) complexes of ligand **3** and its catalytic activity towards C-C bond formation [4]. This result encouraged to further investigate the possibility of present complex is exists or not.



1 **2** **3**

R = H, CH_3, Cl

The selective oxidation of organic molecules catalyzed by copper complexes is one of the most important processes for the oxo functionalization [7-13]. The oxidation of alcohols to aldehydes or ketones are important reactions in synthetic organic chemistry and they are utilized particularly in the manufacture of medicines, flavours, fragrances, and fine chemicals [14-15]. Several copper complexes for the hydrogen peroxide oxidation of alcohols have been developed [10-13, 16-20], and the interest is growing continuously in the search for new efficient catalysts. However, most of the

known conventional methods still use toxic solvents along with hazardous conditions. The development of efficient and environmentally benign oxidation processes using catalyst, and can proceed in aqueous or solvent-free medium under mild conditions for the oxidation of alcohols to the corresponding carbonyl compounds is an important area of research [19-22].

Herein I describe the synthesis of new azo amido complexes of copper(II) by the reaction of 1N-(N,N-diethylaminoethyl)-2-arylazoaniline, HL (**3**) with $CuCl_2.2H_2O$. Formation of the azo amido chelate of composition Cu(L)Cl, **4**, have been authenticated on the basis of spectral analysis and confirmed by X-ray study. The fluorescence property of the ligand and complexes were studied. The redox property of the complex **4c** has been examined electrochemically. In this article, homogeneous oxidation of benzyl alcohol to benzaldehyde has been examined in the presence of catalytic amounts of **4a** and using H_2O_2 as oxidant in mild conditions.

Experimental

The solvents used in the reactions were of reagent grade (E. Marck, Kolkata, India) and were purified and dried by reported procedure [23]. Orthophenylenediamine, nitrobenzene, 4-chloronitrobenzene, 4-methylnitrobenzene and sodium hydroxide were purchased from Loba, India. Copper chloride dihydrate and benzyl alcohol were purchased from E. mark, India. 2-Bromo-N,N-diethylethanamine hydrobromide was purchased from Aldrich. 2-(Arylazo)anilines and 1N-(N,N-diethylaminoethyl)-2-arylazoanilines were prepared according to the reported procedure [24-25]. Microanalysis (C, H, N) was performed using a Perkin-Elmer 2400 C, H, N, S / O series II elemental analyzer. IR spectra were recorded on a Nicolet 6700 FTIR spectrometer (Thermo Scientific). Electronic spectra were recorded on a Shimadzu UV- 1800 PC spectrophotometer. The fluorescence emission spectra were recorded by using JASCO FP-8500 fluorescence spectrometer. Electrochemical measurement was performed

under a dinitrogen atmosphere using CHI6138E electrochemistry system. The supporting electrolyte was tetrabutylammonium perchlorate (0.1 M) and potentials are referenced to Ag/AgCl electrode.

Preparation of Complexes

All the complexes, $Cu(L^1)Cl$, $Cu(L^2)Cl$ and $Cu(L^3)Cl$, were prepared following similar procedures. A representative procedure for $Cu(L^1)Cl$ is given below.

$Cu(L^1)Cl$: A solution of HL^1 (0.146 g, 0.52 mmol) in 5 cm^3 dichloromethanes in a long test tube. (0.09 g, 0.52 mmol) $CuCl_2.2H_2O$ dissolve in 5 cm^3 methanol and taken in a syringe and slowly added to the ligand solution carefully. Then the test tube cover with a paper tightly and kept for several days. Pure dark pink-violet crystal was formed which was wash with petroleum ether and water several times to remove unreacted ligands and copper salt. Yield: 50%. $C_{18}H_{23}ClN_4Cu$ (394.41): calcd. C, 54.82, H, 5.88, N, 14.21; found: C, 54.45, H, 5.53, N, 14.56. UV/Vis spectrum (CH_2Cl_2) λ_{max} (ε, $M^{-1}cm^{-1}$) = 520 (8100), 335 (10050), 240 (17770). IR (KBr pellets, cm^{-1}): ν = 1493 (N=N).

$Cu(L^2)Cl$: Complex $Cu(L^2)Cl$ was prepared using HL^2 in place of HL^1. Yield: $Cu(L^2)Cl$, 45%. $C_{19}H_{25}ClN_4Cu$ (408.43): calcd. C, 55.87, H, 6.17, N, 13.72; found: C, 55.45, H, 5.83; N, 13.40. UV/Vis spectrum (CH_2Cl_2) λ_{max} (ε, $M^{-1}cm^{-1}$) = 525 (10100), 335 (14490), 235 (22930). IR (KBr pellets, cm^{-1}): ν = 1493 (N=N).

$Cu(L^3)Cl$: Complex $Cu(L^3)Cl$ was prepared using HL^3 in place of HL^1. Yield: $Cu(L^3)Cl$, 55%. $C_{18}H_{22}Cl_2N_4Cu$ (428.85): calcd. C, 50.41, H, 5.17, N, 13.06; found: C, 50.67, H, 4.85, N, 13.35. UV/Vis spectrum (CH_2Cl_2) λ_{max} (ε, $M^{-1}cm^{-1}$) = 515 (8800), 330 (10840), 240 (19200). IR (KBr pellets, cm^{-1}): ν = 1492 (N=N). $E_{1/2}$ [V]: 0.68, 1.01, – 1.14 and – 0.65.

General Procedure for catalytic oxidation of benzyl alcohol

To a solution of benzyl alcohol (5 mmol), catalyst **4a** (0.012 mmol), and 30 % H_2O_2 5 mL was added and the mixture was vigorously stirred at 70^0C for 2 h. The progress of the reaction was monitored by TLC. After completion of the reaction the mixture was poured into water and the product was extracted with dichloromethane (3×20 ml) and dried over Na_2SO_4. The solvent was removed and the product was passed through silica column (60-120 mesh). The complex remains trapped into silica column, upon removal of the solvent, pure product was obtained. The yield of the product obtained from the reaction was determined after isolation, and characterized by spectral data.

X-ray structure analysis

Single crystal of Cu(L^3)Cl was grown by slow diffusion of methanolic solution of $CuCl_2.4H_2O$ into a dichloromethane solution of ligand HL^3 at room temperature. Single-crystal of suitable dimension was used for data collection. Data were collected by ω- scan technique on a Bruker SMART APEX CCD diffractometer, with graphite-monochromated Mo-K_α (k = 0.71073 Å) radiation at 295(2) K. Structure solution was done by direct method with SHELXS–97 program [26,27]. Full matrix least square refinements on F^2 were performed using SHELXL–97 program [26,27]. All non-hydrogen atoms were refined anisotropically using reflections I > 2σ (I). Hydrogen atoms were included in the refinement process as per the riding model. The data collection parameters and relevant crystal data are collected in Table 1.

Figs S1–S9 shows the UV/Vis, IR and Emission spectrum of the complexes 4a-c. CCDC 2585668 contain the supplementary crystallographic data for Cu(L^3)Cl. This data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table 1 Crystallographic data for Cu(L^3)Cl

Formula	$C_{18}H_{22}Cl_2N_4Cu$
Mol. wt.	428.84

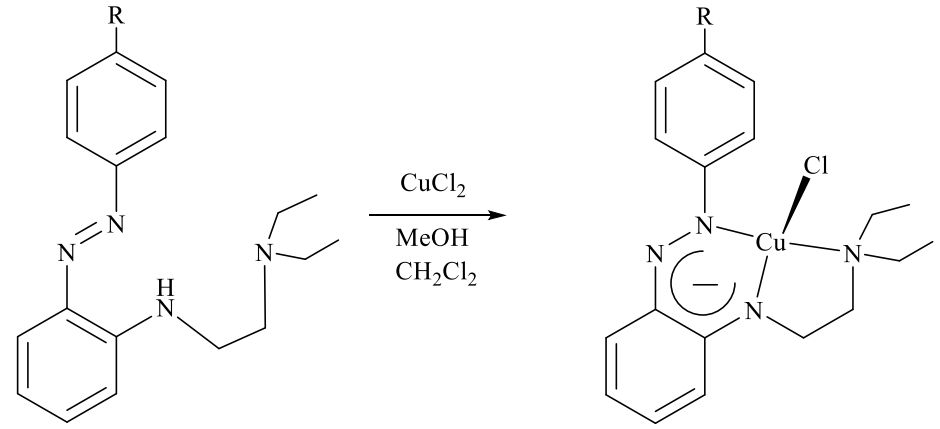
Crystal system	Monoclinic
Space group	P1 21/c1
*A (*Å)	9.75660(10)
*B (*Å)	25.4945(3)
*C (*Å)	7.96290(10)
α (^{0})	90
β (^{0})	104.8600(10)
γ (^{0})	90
Wavelength (Å)	0.71073
Volume (Å)	1914.44(4)
F(000)	884
Crystal dimensions (mm)	0.50 x 0.30 x 0.02
Z	4
Temperature (K)	295(2)
Density/mg/m^{-3}	1.488
Abs. coeff (mm^{-1})	1.428
Theta Min-Max (^{0})	2.16 to 28.30
Reflection measured	18392
Unique reflections (R_{int})	4728, 0.0405
Final R_1 (wR_2) (all)	0.0656, 0.0907
Final R_1 (wR_2) [I>2σ(I)]	0.0399, 0.0822
Goodness of fit	1.009

Results and discussion

Synthesis

Reaction of 1N-(N,N-diethylaminoethyl)-2-arylazoaniline, HL with $CuCl_2.2H_2O$ in 1:1 molar ratio in dichloromethane and methanol biphasic solution upon slow diffusion (Scheme 1), after few days standing a dark violet crystalline compound which upon purification by washing with petroleum ether and water gave pure Cu(L)Cl with 50% yield. The deprotonated HL ligand coordinated with Cu(II)

in a terdentate (N,N,N) fashion through azo nitrogen, deprotonated amido nitrogen, and amino nitrogen atom forming two fused rings- one six membered and other five membered.



3
HL
R = H (3a), CH_3(3b), Cl(3c)

4
Cu(L)Cl
R = H (4a), CH_3(4b), Cl(4c)

Scheme 1. Synthesis of Cu(L)Cl complexes

Characterization

The complexes **4a-c** are soluble in common organic solvents such as CH_2Cl_2, $CHCl_3$, and MeOH etc. to give pink-violet solutions. The colour arises from strong ligand-to-metal charge transfer (LMCT) band at 440 nm and 520 nm [5,28,29]. Higher energy transition at 240 nm is due to intra-ligand charge-transfer. A representative absorption spectrum of complex Cu(L^3)Cl is displayed in fig. 1. UV-Vis spectra of all the compounds are deposited as ESI (Figs S1-S3) and data are collected in Section 3.

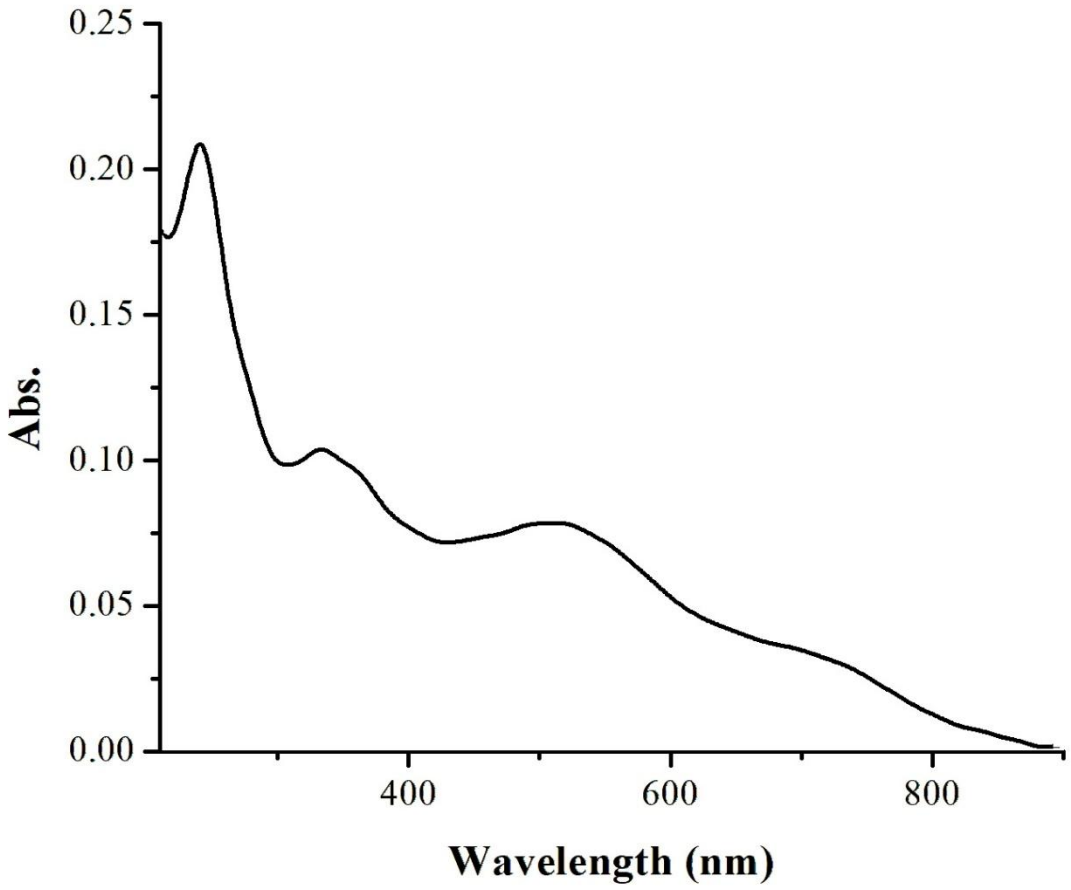


Fig. 1. UV-Vis spectrum of $Cu(L^3)Cl$ in dichloromethane

The fluorescence emission experiment was performed using a JASCO FP-8500 fluorescence spectrometer over the range 365–700 nm with a rectangular quartz cuvette of path length 1 cm at room temperature. Experiments were done in dichloromethane solution of dilute copper(II) complex with a scan speed 500 nm/min. The copper(II) complexes exhibited an emission band at 630 nm upon excitation at 340 nm, whereas the ligands **HL** not to show any absorption upon excitation. A representative florescence spectrum of complex $Cu(L^3)Cl$ is displayed in fig. 2. The emission properties of copper(II) complexes arise due to metal ligand chelation effect and the overall process is referred as "CHEF" [30]. Emission spectra of all the compounds are deposited as ESI (Figures S4-S6)

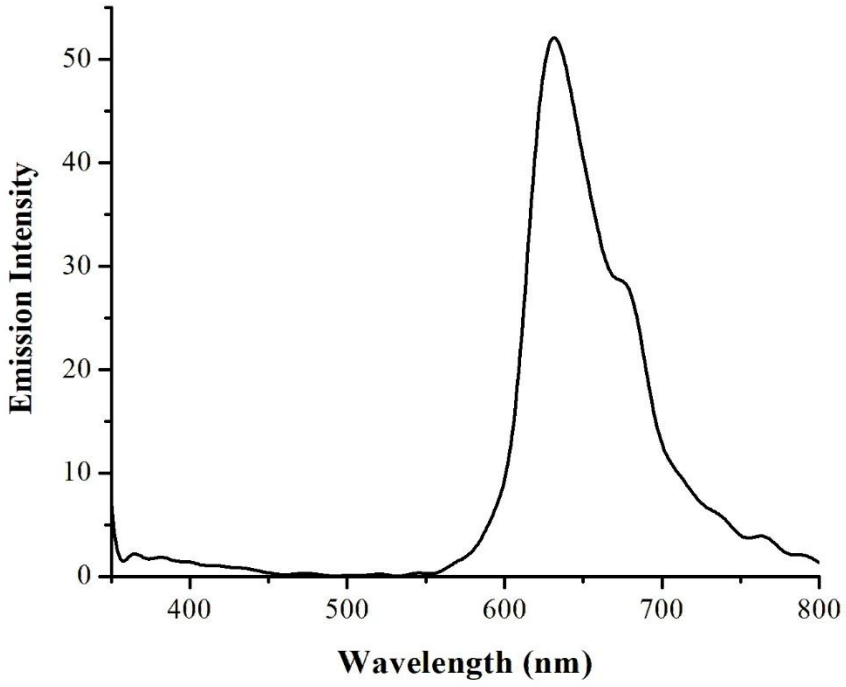


Fig. 2. Emission spectrum of Cu(L^3)Cl in dichloromethane.

The ν_{N-H} stretching frequency 3233 cm^{-1} of the free ligand is absent in the spectrum of the copper(II) complex, confirming that the ligand is coordinated in the deprotonated form [4]. The $\nu_{N=N}$ stretching frequency of Cu(L)Cl complexes appeared at ~1506 cm^{-1} shifted to lower frequency ~1493 cm^{-1} after formation of the chelates, which was consistent with coordination of the azo nitrogen [2-4,6].

Suitable crystals of Cu(L^3)Cl, were grown by slow diffusion of dichloromethane solution of ligand HL^3 into methanol solution of copper chloride. A perspective view of the metal coordination environment of Cu(L^3)Cl has been shown in fig. 3 and selected bond distances and angles are collected in Table 2. The copper(II) ion is coordinated to potentially tridentate HL^3 ligands. Thus HL^3, in its deprotonated form, utilizes all its donor sites for metal coordination and a chloride ligand are satisfying its tetra coordination. The geometry about copper is distorted square planar justifying the oxidation state of Cu(II) and the ter-coordinated uninegative ligand with a chloride, since the complex is a non-electrolyte. The copper(II) center is coordinated by alkyl amino nitrogen N(4),

deprotonated amido nitrogen N(3), and azo nitrogen N(1). The chelate bite angles of ligand (HL^3) are considerably less than the ideal square planar value (90^0) and angles between trans atoms are N(1)-Cu(1)-N(4) = 162.48^0 N(3)-Cu(1)-Cl(2) = 153.63^0. The coordination sphere Cu(N,N,N)Cl is non-planar (deviation from mean-plane 0.442 Å). As a consequence, the Cu-N(1)(azo), Cu-N(3)(amido), Cu-N(4)(amine) and Cu-Cl(2) coordination environment is severely distorted from ideal square planar geometry. The Cu(1)–N(amido) bond length is the shorter (0.178 Å) than Cu–N(amine) bond length, similarly N(3)-C(12) is shorter (0.039 Å) than N(20-C(7). The Cu(1)–N(azo), Cu(1)–N(amido), Cu(1)-N(alkyl) and Cu(1)–Cl(2) lengths (1.978(2) Å, 1.910(2) Å, 2.078(2) Å and 2.2352(7) Å, respectively) are within the normal range [6,31,32]. The bond lengths of phenyl ring adjacent to the six membered chelate ring were suitably altered due to delocalization of the negative charge [2-4,6]. Effect of the charge delocalization which is reflected in the adjacent phenyl ring with two short (~1.35 Å) and four long (~1.418Å) carbon-carbon bond distances [2-4,6].

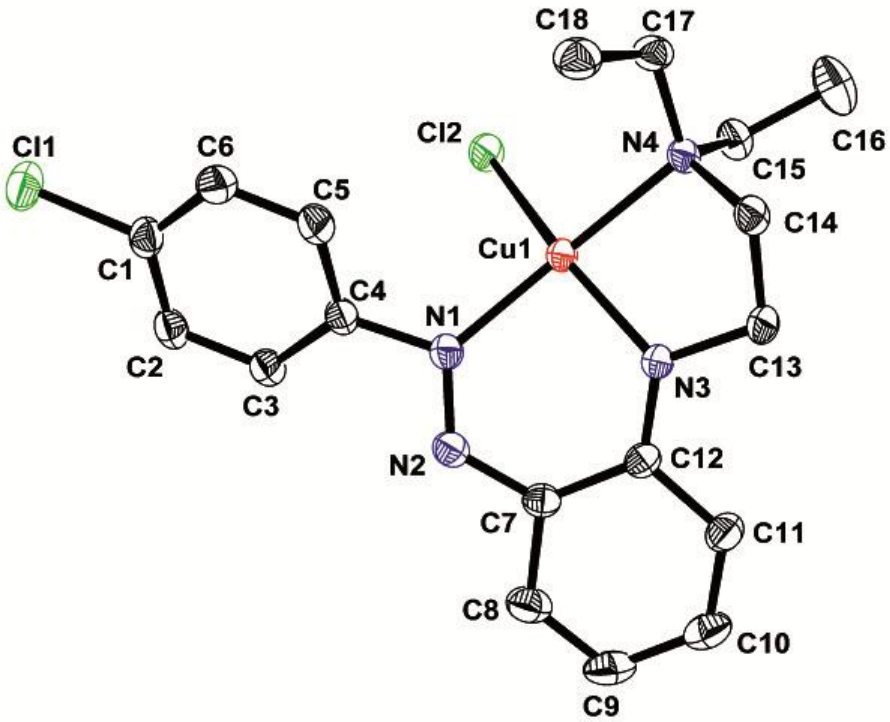


Fig. 3 Molecular structure of Cu(L^3)Cl with atom numbering scheme. Hydrogen atoms are omitted for clarity.

Table 2 Selected Bond Distances (Å) and Angles (deg) for Compound Cu(L^3)Cl

Distances			
Cu(1)-N(3)	1.910(2)	Cu(1)-N(1)	1.978(2)
Cu(1)-N(4)	2.078(2)	Cu(1)-Cl(2)	2.2352(7)
N(1)-N(2)	1.287(3)	N(1)-C(4)	1.439(3)
N(2)-C(7)	1.364(3)	N(3)-C(12)	1.325(3)
N(4)-C(14)	1.479(3)	N(3)-C(13)	1.471(3)
N(4)-C(17)	1.488(3)	N(4)-C(15)	1.502(3)
C(13)-C(14)	1.502(4)	C(7)-C(8)	1.418(4)
C(7)-C(12)	1.437(4)	C(8)-C(9)	1.357(4)
C(9)-C(10)	1.392(5)	C(11)-C(12)	1.426(4)
C(10)-C(11)	1.350(4)		
Angles			
N(3)-Cu(1)-N(1)	90.16(9)	N(3)-Cu(1)-N(4)	85.02(9)
N(1)-Cu(1)-N(4)	162.48(9)	N(3)-Cu(1)-Cl(1)	153.63(7)
N(1)-Cu(1)-Cl(2)	97.22(6)	N(4)-Cu(1)-Cl(2)	94.50(6)
N(2)-N(1)-C(4)	111.3(2)	N(2)-N(1)-Cu(1)	126.43(18)
N(1)-N(2)-C(7)	122.4(2)	C(4)-N(1)-Cu(1)	121.06(17)
C(12)-N(3)-C(13)	119.3(2)	C(12)-N(3)-Cu(1)	125.55(17)
C(13)-N(3)-Cu(1)	114.09(17)	C(14)-N(4)-C(17)	111.2(2)
C(17)-N(4)-C(15)	111.5(2)	C(14)-N(4)-Cu(1)	102.26(15)
C(15)-N(4)-Cu(1)	108.38(15)	C(17)-N(4)-Cu(1)	113.69(16)

Electrochemical property of the complex **4c** has been studied by cyclic voltammetry in 9:1 dichloromethane - acetonitrile mixed solvent (0.1 M TBAP) at scan rate of 0.1 Vs^{-1} (Fig. 4). The complex **4c** shows two oxidative responses on the positive side of SCE and two reductive responses on the negative side. The first quasi reversible oxidative response at 0.68 V is assigned to Cu(II)/Cu(I) oxidation [33,34] and the irreversible response at 1.01 V is assigned to ligand oxidation. Both the reductive responses –1.14 V and –0.65 V are tentatively assigned to reduction of the coordinated ligand **3c**.

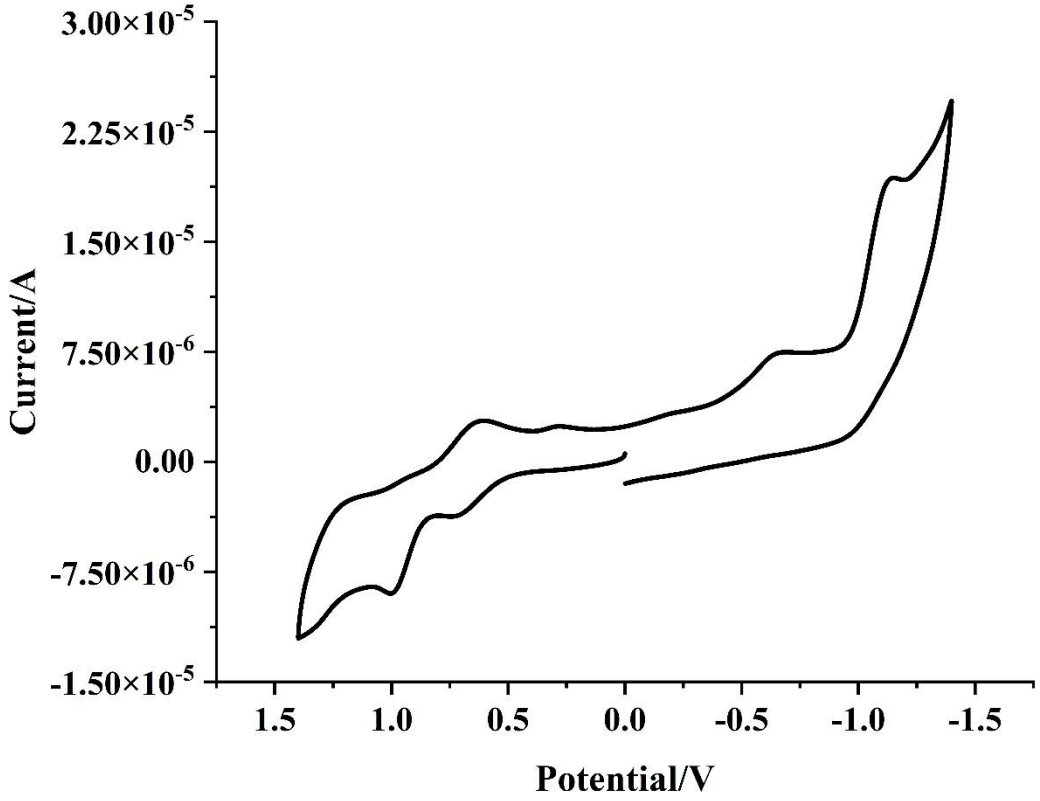
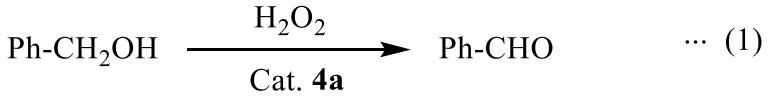


Fig. 4. Cyclic Voltammogram of $Cu(L^3)Cl$ in dichloromethane acetonitrile solution (0.1 M TBAP) at scan rate of 0.1 Vs^{-1}.

Oxidation of benzyl alcohol

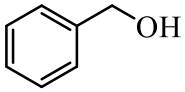
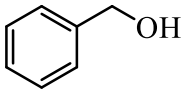
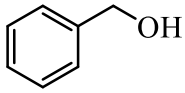
The preliminary investigation of the catalytic property of complex **4a** was taken for the oxidation of benzyl alcohol to benzaldehyde using H_2O_2 (eq. 1) as oxidizing agent, under atmospheric pressure and in absence of any additive other than the substrate. The yield has been optimized by varying the



··· (1)

catalyst amount and also varying the reaction temperature using fixed volume (5 mL) of hydrogen peroxide (30%) for 2h time. The product was isolated after separation and the conversions was calculated on the basis of isolated yield. The results of the oxidation studies were shown in Table 3. The impact of temperature was studied at 25^0C and 75^0C for the oxidation reaction. The oxidation of benzyl alcohol by H_2O_2 at 25^0C was very slow (yield ~25%) for 2h. There is no significant change of yields of benzaldehyde were obtained with catalyst amounts varying from 0.006 - 0.012 mmol.

Table 3 Oxidation of benzyl alcohol catalyzed by **4a**

Entry	Equiv H_2O_2 (30%)	Time (h)	Amount of **4a** (mmol)	Temp. ^{0}C	Yield (%)
	5	2	0.006	25	20
	5	2	0.006	75	83
	5	2	0.012	75	85

Conclusion

In this work reports the synthesis, characterization, and properties of a new four-coordinate copper(II) complex coordinated by a deprotonated 1N-(N,N-diethylaminoethyl)-2-arylazoaniline ligand providing an amido-N, an amino-N, and an azo-N coordination. The Ni–N (amido) bond lengths [1.910(2) Å] is the shortest and Ni–N(azo), Ni-N(amine) bond lengths [1.978(2) and 2.078(2) Å] are the longest. The effective interaction with the copper(II) center through strong σ-donating amido nitrogen and the amino, azo nitrogen remains weakly coordinated to the copper(II) center. The

present study affords notable information on the coordination ability of an azo group. The structural analysis reveals that the distorted phenyl ring adjacent to the six-membered azo-amido chelate ring consist four longer and two shorter bonds indicates the delocalization of the negative charge within the ligand backbone. The delocalization of negative charge of the anionic ligands that formed *in situ* from the precursor (L^-) due to dissociation of an amido proton. The redox response of the copper complex 4c showed both metal and ligand centric oxidation and rection. The emission behavior of copper(II) complexes was examined. The activity of complex **4a** as catalyst towards oxidation of benzyl alcohol to benzaldehyde was tested in presence of H_2O_2 as an oxidant in mild conditions.

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Declaration of Interest Statement

The article is original and unpublished and is not being considered for publication elsewhere. The author declares that there has no conflict of interests that could influence this work.

Credit authorship contribution statement

Jahar Lal Pratihar: Supervision.

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