



A MOLYBDENUM MEDIATED TRANSFORMATION OF A CARBOHYDRAZONE LIGAND LEADING TO THE FORMATION OF A HYDRAZONE WITH AN ENHANCEMENT OF FLUORESCENCE EMISSION

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ABSTRACT

A combined effect of acid catalysis and Mo mediated C=N bond cleavage on the Schiff base ligand *N*'-[(5-bromo-2-hydroxyphenyl)methylidene]-5-methyl-1*H*-pyrazole-3-carbohydrazide(L) which is prepared by the condensation of 5-methyl 3-pyrazole carbohydrazide with 5-bromo salicylaldehyde, leads to its transformation to the compound 2,2'-[(hydrazine-1,2-diylidenedimethylidene)bis(4-bromophenol) (I) with the formation of a Mo(IV) complex in the reaction media. The resulting bis salicylhydrazone was characterized by UV-Vis, IR, NMR and also by X-ray crystallography. The compound I was found to have a more pronounced emission behavior as compared to the used ligand L. But the Mo(IV) complex which was produced in the reaction medium as a result of the oxidative cleavage of the ligand, was found to suffer fluorescence quenching with respect to the ligand.

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INTRODUCTION

In one of our previous publications [1] we have unveiled a distinctive property of dioxomolybdenum(VI) ion to mediate a reaction leading to the cleavage of a hydrazone type of ligand used for its complexation. Since we have spent considerable amount of time studying and reviewing [2] the complexation behavior of Mo(VI) ions towards hydrazone and carbohydrazone type ligands derived from heterocycles, we were intrigued by the unusual nature of the aforementioned Mo mediated reaction. Since then we have engaged ourselves in the investigation of suitable conditions and precursors which might trigger such a reaction in other related ligands. We have reported the complexation of Mo(VI) and Mo(IV) ions by a series of pyrazole derived carbohydrazone ligands a few years back[3]. The Schiff base ligands obtained by the condensation of 5-methyl-3-pyrazole carbohydrazide with salicylaldehyde, substituted salicylaldehyde and ortho hydroxy acetophenone were found to be good tridentate donor for Mo(IV) and Mo(VI) species and produced complexes which had fairly good thermal and redox stability. While preparing the complexes of these ligands, with the common Mo(VI)

precursor MoO₂(acac)₂, no evidence of any ligand transformation could be traced in neutral medium as well as in acid medium. However we have found here that with (NH₄)₂MoO₄ as the metal precursor and using acidic medium, a transformation of one of these ligands, take place which is very likely Mo mediated and closely related to the one observed earlier. We have also found that the emission behavior of the used ligand and the transformed product are completely different. The Mo(IV) complex which is produced in the reaction medium show fluorescence quenching with respect to the used ligand. The formation of a green residue, later characterized as a Mo(IV) complex of the ligand gives evidence of the oxidative cleavage of the ligand mediated by Mo(VI) which in turn is reduced to Mo(IV) species.

Experimental Section

MATERIALS

MoO₂(acac)₂ was prepared as described in the literature[4]. Reagent grade solvents are used as received. 5 (3)-Methyl pyrazole -3 (5)-carbohydrazide was prepared as described in literature [5]. 5-bromo salicylaldehyde, (NH₄)₂MoO₄ were used as received from commercial source. Ligand L was prepared by using the literature method [3]. Tetraethyl ammonium perchlorate (TEAP), used for electrochemical work, was prepared as reported in the literature [6].

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Preparation of 2, 2'-[(hydrazine-1,2-diylidenedimethylidene]bis(4-bromophenol) (**1**) and [C₁₀N₄O₅H₁₀Mo](Complex **2**):

The compound **1** was prepared by refluxing an acidified (HCl, pH 2.5) ethanolic solution (25 ml) of (NH₄)₂MoO₄ (0.5 mmol, 0.098 g) with ethanolic solution of **L** (0.5 mmol, 0.161 g). On mixing the two components, immediately an orange yellow colour was observed. The mixture was refluxed for 1h. The colour of the solution turned green. The compound **1** separated as a yellow microcrystalline compound during reflux. It was filtered, washed with ethanol and dried over fused CaCl₂. After filtration of compound **1** the green filtrate was kept for evaporation for a week. During that time a green precipitate of complex **2** was obtained. It was again filtered, washed with ethanol and dried over fused CaCl₂.

Compound **1**: Yield 0.024g(25%) Elemental analysis: Anal.Calc. for C₇H₅BrNO : C:42.21 H :2.51 N:7.03; Found C:42.03 H :2.48 N:6.94. The crystals of **1** were obtained by slow evaporation of a mixed solution of DMF and diethyl ether. UV-VIS(λ_{max} /nm) (ϵ /dm³mol⁻¹cm⁻¹) : 295 (15480), 367 (18456), IR(ν /cm⁻¹): ν (N-H) 3278, ν (O-H) 3193 ν (C-N) 1549, ν (C=C) 1528.

Complex **2**: [C₁₀N₄O₅H₁₀Mo] Yield:0.059 g(50%) Elemental analysis: Anal.Calc. for C₅N₂O₃H₅Mo : C:33.24 H :2.77 N:15.51; Found C:33.17 H :2.69 N:15.31 IR(ν /cm⁻¹)(Mo=O): 915, ν (N-H) 3318, ν (C=O) 1652, ν (C=N) 1622, ν (C=C) 1555.2. UV-VIS(λ_{max} /nm) (ϵ /dm³mol⁻¹cm⁻¹) : 548(310) CV (E/Volts): E_{pa} = 1.33 [Mo(VI)/Mo(IV)], E_{pc} = -1.25, E_{pa} = -0.67 [Mo(IV)/Mo(III)] ΔE_p =0.58, E_{1/2}=0.925.

Physical Measurements

Elemental analyses were performed at IACS, Kolkata on a Perkin–Elmer 2400 CHN analyzer. The 1H NMR spectra of compound **1** and complex **2** were recorded on a Bruker AM300L (300 MHz) superconducting FT-NMR. IR spectra were recorded as a KBr pellet on a Perkin–Elmer FTIR spectrophotometer. UV–Vis spectra and DRS were measured on a Hitachi U-3501 spectrophotometer. Cyclic Voltammetry was carried out using a Sycopel model AEW21820F/L instrument. The measurements were carried out at 293 K in DMF solutions containing 0.2 M TEAP as supporting electrolyte and 10⁻³-10⁻⁴ M solution of the respective complex deoxygenated by bubbling with nitrogen. The working, counter and reference electrodes used were a Pt wire, platinum coil and an SCE, respectively. The fluorescence spectra were recorded on a Perkin Elmer LS50B fluorimeter.

Crystallographic Measurements

The crystals were measured on an Oxford Diffraction Gemini diffractometer using Mo K α radiation (λ =0.71073 Å) and a graphite monochromator at 73°C. The unit cell dimensions were obtained from a least squares fit to the setting angles of 25, accurately determined reflections. The data were collected using a ω -2 θ scan technique with a 2 θ range of 4-55° and were corrected by an empirical method for absorption and Lorentz and polarization effect. Unit cell data were obtained by standard methods [7, 8] Calculations were carried out using a CrysAlis CCD, Oxford Diffraction Ltd., Version 1.171.32.5 (data reduction), SHELXTL (absorption corrections, structure solution refinements and molecular graphics) [9]. Crystal data and details of data collection are listed in Table I.

Table I Crystal data and structure refinement for Compound **1**

Identification code	sksg46	
Empirical formula	C7 H5 Br N O	
Formula weight	199.03	
Temperature	346(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P 1	
Unit cell dimensions	a = 6.4470(5) Å b = 7.2662(12) Å c = 7.8548(8) Å	α = 79.081(10)° β = 79.474(8)° γ = 72.467(10)°
Volume	341.40(7) Å ³	
Z	2	
Density (calculated)	1.936 Mg/m ³	
Absorption coefficient	5.938 mm ⁻¹	
F(000)	194	
Crystal size	0.41 x 0.35 x 0.26 mm ³	
Theta range for data collection	5.05 to 32.57°	
Index ranges	-9<=h<=9, -10<=k<=10, -11<=l<=11	
Reflections collected	4416	
Independent reflections	2136 [R(int) = 0.0367]	
Completeness to theta = 25.00°	98.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.26279	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2136 / 0 / 92	
Goodness-of-fit on F ²	1.037	
Final R indices [I>2 σ (I)]	R1 = 0.0496, wR2 = 0.1164	
R indices (all data)	R1 = 0.1016, wR2 = 0.1544	
Largest diff. peak and hole	0.803 and -0.964 e.Å ⁻³	

Table II Bond lengths [Å] for compound **1**

Br-C(4)	1.901(5)
O(1)-C(1)	1.341(6)
O(1)-H(1)	0.8200
N-C(7)	1.288(6)
N-N#1	1.398(7)
C(1)-C(6)	1.396(6)
C(1)-C(2)	1.419(6)
C(2)-C(3)	1.383(6)
C(2)-C(7)	1.459(6)
C(3)-C(4)	1.372(6)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.386(7)
C(5)-C(6)	1.374(7)
C(5)-H(5A)	0.9300
C(6)-H(6A)	0.9300
C(7)-H(7A)	0.9300

Table III Angles [°] for compound **1**

C(1)-O(1)-H(1)	109.5
C(7)-N-N#1	113.3(4)
O(1)-C(1)-C(6)	119.0(4)
O(1)-C(1)-C(2)	123.0(4)
C(6)-C(1)-C(2)	118.0(4)
C(3)-C(2)-C(1)	119.5(4)
C(3)-C(2)-C(7)	118.7(4)
C(1)-C(2)-C(7)	121.7(4)
C(4)-C(3)-C(2)	120.9(4)
C(4)-C(3)-H(3A)	119.5
C(2)-C(3)-H(3A)	119.5
C(3)-C(4)-C(5)	120.5(4)
C(3)-C(4)-Br	120.7(3)
C(5)-C(4)-Br	118.8(3)
C(6)-C(5)-C(4)	119.4(4)
C(6)-C(5)-H(5A)	120.3
C(4)-C(5)-H(5A)	120.3

C(5)-C(6)-C(1)	121.7(4)
C(5)-C(6)-H(6A)	119.2
C(1)-C(6)-H(6A)	119.2
N-C(7)-C(2)	121.1(4)
N-C(7)-H(7A)	119.4
C(2)-C(7)-H(7A)	119.4

Symmetry transformations used to generate equivalent atoms: #1 -x+1,-y+1,-z-1

Table IV Hydrogen bonds for Compound 1 [\AA and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
O(1)-H(1)...N	0.82	1.95	2.648(5)	142.3
O(1)-H(1)...Br#2	0.82	3.12	3.525(3)	113.4

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z-1 #2 x+1,y,z-1

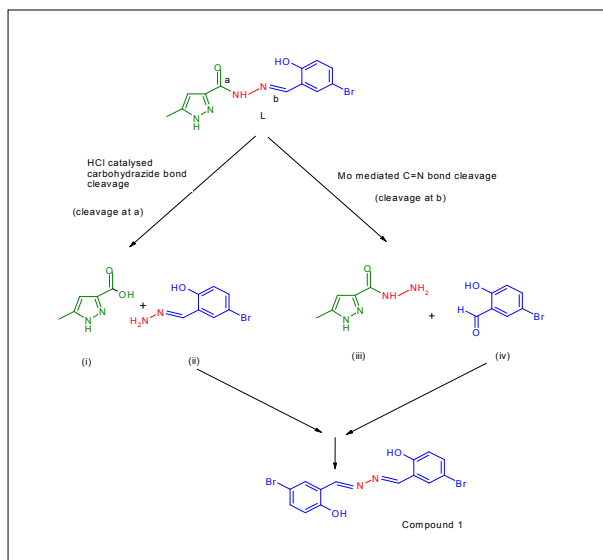


Figure 1 Reaction scheme showing the reactions leading to compound 1

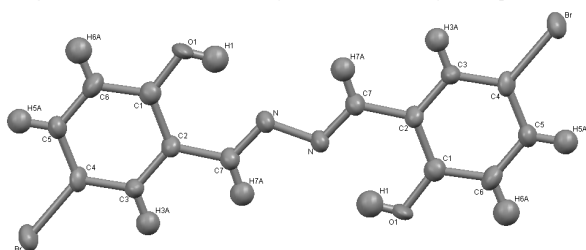


Figure 2 Representative diagram of the crystal structure of compound 1

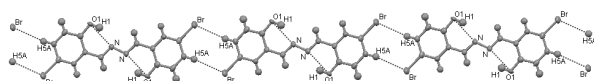


Figure 3 Intramolecular and intermolecular H-bonding in compound 1

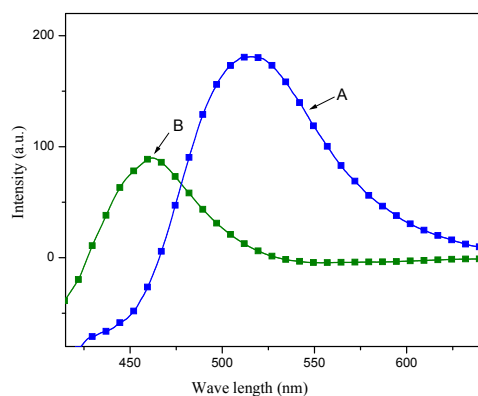


Figure 4 Fluorescence spectra of compound 1(A) ($\lambda_{\text{ex}}=293\text{nm}$, 367nm) and L (B) ($\lambda_{\text{ex}}=395\text{nm}$). The spectra of of L has been magnified thrice to fit in the same scale.

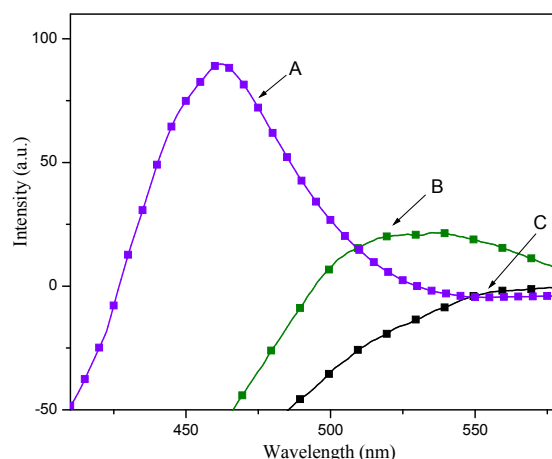


Figure 5 (A) Fluorescence spectra of L ($\lambda_{\text{ex}}=395\text{nm}$), (B) After addition of $(\text{NH}_4)_2\text{MoO}_4$ and HCl to a solution of L ($\lambda_{\text{ex}}=410\text{nm}$) (C) After completion of ligand cleavage reaction ($\lambda_{\text{ex}}=370\text{nm}$). All the spectra were recorded in DMF solutions which were $2\times$

The structures were solved using direct methods and refined on F^2 using full matrix least squares techniques with anisotropic displacement factors for all non-hydrogen atoms. Positions of the Hydrogen atom were calculated from the geometry of the molecular skeleton and their thermal displacement parameters were refined isotropically on a group wise basis. Selected bond lengths and angles are reported in Table II and Table III.

DISCUSSION

We have used the Schiff base ligand prepared by the condensation of 5-bromo salicylaldehyde and 5-methyl-3-pyrazole carbohydrazone as a representative of the class of carbohydrazone ligands for our study of Mo mediated ligand transformation. When equimolar proportion of the ligand and $(\text{NH}_4)_2\text{MoO}_4$ (taken in HCl medium) was refluxed in an ethanolic solution, a bright yellow crystalline compound appeared. The solution turned green and after evaporation of the mother liquor a green residue was obtained. Single crystals of the bright yellow compound were grown and it was found to be an organic compound, 2,2'-(hydrazone-1,2-diylidenedimethylidene)bis(4-bromophenol(1)). Its formation may be only described by the breaking of the original ligand at both the amide bond ($\text{C}=\text{O}$) as well as at the imine bond ($\text{C}=\text{N}$). The 5-bromo salicyl hydrazone produced by the cleavage of the used ligand at the amide ($\text{C}=\text{ONH}$) bond undergoes condensation with the 5-bromo salicyl aldehyde which is made free by the cleavage at the Imine bond ($\text{C}=\text{N}$). (figure 1) Though hydrolysis of the amide functionality may be attributed to acid catalysis, the cleavage of $\text{C}=\text{N}$ bond only could have taken place by a Mo mediated oxidative cleavage. We have subjected the ligand to various level of HCl concentration in refluxing ethanolic solution. Only two products were obtained in each case. 5-methyl-3-pyrazole carboxylic acid and 5-bromo salicyl hydrazone. This is evidence of only hydrolytic cleavage of amide bond under acid catalyzed condition. Further when an ethanolic solution of the ligand was treated with a suspension of $(\text{NH}_4)_2\text{MoO}_4$ in neutral medium no evidence of any ligand transformation was found. Here only dioxomolybdenum(VI) complex of the ligand was

obtained, which has already a known compound reported by us. The green compound that is obtained as a side product from the mother liquor was characterized as a Mo(IV) complex of the 5-methyl-3-pyrazole carboxylate anion produced in the reaction medium. A composition of $[\text{Mo}(\text{IV})\text{O}(\text{5-methyl-3-pyrazolecarboxylato})_2]$ i.e. $[\text{C}_{10}\text{N}_4\text{O}_5\text{H}_{10}\text{Mo}]$ has been assigned to it from UV Vis, IR and electrochemical studies. This Mo(IV) complex is an outcome of the oxidative cleavage that dioxomolybdenum(VI) performs on the ligand and itself gets reduced. The compound **1** was first reported by M. P. Jain, *et al* [10] and its complexation with Tellurium was reported by T. N. Srivastava [11]. We are reporting the crystal structure of this compound for the first time and a new method of its preparation.

Structure description

The compound **1** crystallizes with P -1 space group with a single molecule per unit cell. The two C=N bonds are anti to each other. This leaves two phenyl rings, the two phenolic OH groups, and the two bromo groups anti to one another (figure 2). There is almost complete planarity in the molecule. The two hydrogens of the phenolic OH group are above and below the plane of the molecule. The N-N bond distance is found to be 1.398 Å which shows that this is single bond in nature whereas the C=N bond shows double bond character at 1.288 Å. Because of the large size of the Br atom the two C-Br bonds are found to be nearly 2 Å in length. All the other bonds (i.e C-C and C-O) are in the normal range. All the bond angles except one C7-N-N#1 shows values applicable to sp^2 hybridized centres. C7-N-N#1 has a much lower value of 113° . There is an E conformation about the two nitrogen atoms.

There are two types of H-bonding in the molecule (Table IV). The first one is an intramolecular one between the O1, H1 and N. Two such H-bonds are present on either side of the N-N bond. The second one is intermolecular type between C5, H5A and Br (figure 3). H bonds involving Br are not common. This intermolecular type bond helps to create an infinite linear chain of molecules in the crystal. In the crystals these infinite chains are found to be stacked one above the other. Profound π - π stacking between the phenyl rings which are at cg-cg distance of 3.73 Å helps in this arrangement.

Absorption and Emission behavior

The absorption spectra of Ligand **L**, taken in a 10^{-3} (M) DMF solution, shows a broad low intensity peak at 405 nm and a sharp peak at 334 nm. This may be due to the n - π^* and π - π^* transitions. There are other peaks in the higher energy regions too. The compound **1** shows two distinctive peaks at 367 nm and 293 nm in the same solvent and concentration. Both these peaks are moderately intense. Upon addition of the acidified solution of $(\text{NH}_4)_2\text{MoO}_4$ to the ligand solution in DMF, a change in its absorption behavior is observed. The peak at 405 nm disappears and a new peaks at 410 nm and 333 nm appears. These are similar to our earlier reported dioxomolybdenum (VI) complex of the same ligand and shows that complexation of the Mo(VI) species takes place in the solution first. These peaks are assigned as charge transfer transitions of the type N ($p\pi$)-Mo($d\pi$) LMCT and O($p\pi$)-Mo($d\pi$) LMCT respectively [12, 13, 14], as the ligand based orbitals are either N or O donor types. After the solution was refluxed for an hour, it turned green. This solution had a broad absorption peak at 325 nm and another low intensity peak at 548 nm both of which are

of charge transfer type. The isolated complex **2** showed the same maxima in the UV-Vis solution spectra in DMF.

Organic molecules containing a fluorophoric unit combined with site/s for guest binding purpose [15-30] have found application in building up fluorescent signaling system for biomedical research [31] and chemical logics [32-35]. Metal ions can act effectively as guests for these molecules because of their ability to enhance, shift or quench [36-38] luminescent emissions of these organic ligands by coordination. The changes brought about by metal binding are mechanistically of four types, photoinduced electron transfer (PET) [39, 40] photoinduced charge transfer (PCT)[41-43], formation of monomer/excimer [44-46], energy transfer[47-49] and proton transfer[50-52]. Transition metal ions with partially filled d-orbitals are known to induce fluorescence quenching or enhancement by oxidative or reductive PET and energy transfer processes [53-55]. Luminescent properties of pyrazole derived ligands and their complexes have been reported a number of times in the recent past[56-58]. Though our ligand (**L**) has been reported earlier, its emission behavior has not been explored till date.

The fluorescence spectra of the ligand **L**, compound **1** and complex **2** was recorded in 2×10^{-5} (M) solution in DMF solution at room temperature (figure 4). When excited at a wavelength of either of its absorption maxima, strong fluorescence was observed with maxima at 515 nm for compound **1**. Again when the ligand **L** was excited at both wavelengths of its absorption maxima i.e. at 395 nm and 334 nm, a fluorescence emission was observed only in the former case. The intensities of emission were much higher in case of compound **1** than in **L** when recorded in the same solvent maintaining same molar concentration. In recent years emission behavior of a number of pyrazole based ligands have been studied and we identify the pyrazole unit responsible for the fluorescence of the ligand **L**. But the strong emission from the compound **1** was interesting as no particular fluorophore could be identified here.

The change in emission behavior of **L** on addition of the acidified $(\text{NH}_4)_2\text{MoO}_4$ solution was also studied. We have mentioned that on addition of $(\text{NH}_4)_2\text{MoO}_4$ solution to a solution of **L**, a yellow colour developed. On being excited at 410 nm and 333 nm, weak emission at 534 nm was observed which shows evidence of fluorescence quenching due to complexation of the Mo(VI) ion with **L**. The emission behavior of the same solution changes after refluxing and separation of compound **1**. The green solution and the green residue from it which was later characterized as complex **2** were both found to be non fluorescent when excited at 370 nm (figure 5).

CONCLUSION

The production of compound **1** in the reaction medium containing $(\text{NH}_4)_2\text{MoO}_4$, HCl and **L** emphasizes on the capability of Mo(VI) species to cleave a C=N bond. Though here, in formation of compound **1**, an associative role of the acid medium is very probable. Moreover, the fact that a compound with a higher fluorescence emission compared to the ligand has resulted in the process makes the reaction even more interesting.

Appendix A. Supplementary data

CCDC 1831986 contains the supplementary crystallographic data for compound 1. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, 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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