

Scientific paper

Synthesis, Crystal Structure and Catalytic Activity of Dioxidomolybdenum(VI) Complex with Tridentate ONO Aroylhydrazone Ligand

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Abstract

A novel dioxidomolybdenum complex, [MoO₂L], derived from the tridentate aroylhydrazone 2-amino-*N'*-(2-hydroxy-3-methylbenzylidene)benzohydrazide (H₂L), has been prepared and characterized by elemental analysis, FT-IR, ¹H NMR, and single crystal structural X-ray diffraction. The complex has distorted square pyramidal structure in which the aroylhydrazone ligand behaves as a binegative donor, leaving the amino-N group uncoordinated towards the metal center. Single crystal structure analysis reveals that the Mo(VI) center is coordinated by the donor atoms of the binegative aroylhydrazone ligand and two oxido groups. Crystal structure of the complex is stabilized by hydrogen bonds. The complex functions as an effective olefin epoxidation catalyst with hydrogen peroxide as terminal oxidant and sodium hydrogen carbonate as a co-catalyst.

Keywords: Aroylhydrazone; Dioxidomolybdenum complex; Catalytic activity; Crystal structure

1. Introduction

In the last few years, considerable attention has focused on the coordination chemistry of molybdenum because of its biochemical significance¹ as well as involvement of Mo(VI) compounds in some industrial catalytic processes.² Molybdenum complexes are widely used as efficient catalysts in different processes such as the oxidation of olefins and sulfides.³ Aroylhydrazones are potential tridentate ligands.⁴ During search of literature, we found that most molybdenum complexes with aroylhydrazones have octahedral coordination.⁵ The only example of square pyramidal coordination of molybdenum complexes with aroylhydrazone ligand was reported by Ghosh and coworkers.⁶ In the present work, a novel molybde-

num(VI) complex with square pyramidal coordination, [MoO₂L], derived from the tridentate aroylhydrazone 2-amino-*N'*-(2-hydroxy-3-methylbenzylidene)benzohydrazide (H₂L; Scheme 1), was studied.

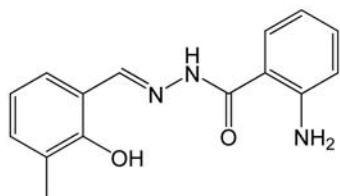
2. Experimental

2. 1. Materials and Methods

All chemicals and solvents used were of analytical reagent grade and used as received. MoO₂(acac)₂ was prepared as described in the literature.⁷ Micro analyses for C, H, N were carried out using a Perkin Elmer 2400 CHNS/O elemental analyzer. FT-IR spectra were recorded on a FT-IR 8400-Shimadzu as KBr discs in the range of 400–4000 cm⁻¹. ¹H NMR spectra were recorded at 25 °C on the Bruker AVANCE 300 MHz spectrometer. X-ray diffraction data were collected using a Bruker Smart Apex II diffractometer.

2. 2. Synthesis of H₂L

A methanolic solution (10 mL) containing 1.00 mmol of 2-hydroxy-3-methylbenzaldehyde (0.136 g) was

Scheme 1. H₂L

added dropwise to a methanolic solution of 2-aminobenzohydrazide (1.00 mmol, 0.151 g) with constant stirring. The mixture was refluxed for 30 min, and the resulting precipitate was filtered off, washed with cold methanol and dried in desiccator over silica gel. Yield: 0.23 g, 85%. For $C_{15}H_{15}N_3O_2$: anal. calcd., %: C, 66.90; H, 5.61; N, 15.60. Found, %: C, 66.73; H, 5.72; N, 15.75. FT-IR (KBr), cm^{-1} : $\nu(NH)$ 3232, $\nu(OH)$ 3431, $\nu(CH_{ar})$ 2830–3085, $\nu(C=O)$ 1650, $\nu(C=N)$ 1615, $\nu(N-N)$ 1149, $\nu(C-O)$ 1287. 1H NMR (300 MHz, DMSO- d^6 , ppm): δ = 12.10 (s, 1H; NH), 11.05 (s, 2H, NH_2), 10.76 (s, 1H; OH), 8.70 (s, 1H; $CH=N$), 6.80–7.15 (m, 5H, ArH), 7.43 (d, 1H, ArH), 7.69 (d, 1H, ArH), 2.13 (s, 3H, CH_3).

2. 3. Synthesis of $[MoO_2L]$

A methanolic solution (10 mL) of $MoO_2(acac)_2$ (0.10 mmol, 0.033 g) was added to a solution of H_2L (0.10 mmol, 0.027 g) in methanol (10 mL) and the resulting orange mixture was refluxed for 30 min. After cooling, the solution was filtered and left to stand overnight. Orange single crystals suitable for crystallography separated after a week and were dried in a vacuum desiccator over silica gel. Yield: 0.021 g, 63%. For $C_{15}H_{13}MoN_3O_4$: anal. calcd., %: C, 45.58; H, 3.32; N, 10.63. Found, %: C, 45.40; H, 3.41; N, 10.54. IR (KBr) cm^{-1} : $\nu(OH)$ 3466, $\nu(NH_2)$ 3340, $\nu(NH)$ 3233, $\nu(CH)$ 2810–2990, $\nu(C=N)$ 1611, $\nu(C=C_{ring})$ 1434, $\nu(N-N)$ 1157, $\nu(C-O)$ 1257, $\nu_{sym}(cis-MoO_2)$ 949, $\nu_{asym}(cis-MoO_2)$ 861. 1H NMR (400 MHz, DMSO- d^6 , ppm): δ = 11.13 (s, 2H, NH_2), 8.77 (s, 1H; $CH=N$), 6.70–7.80 (m, 7H; ArH), 2.15 (s, 3H, CH_3).

2. 4. X-Ray Structure Determination

The crystal of the complex was measured on a Bruker SMART Apex II CCD diffractometer using Mo $K\alpha$ ra-

diation ($\lambda = 0.71073 \text{ \AA}$) and a graphite monochromator at 25 °C. Unit cell and reflection data were obtained by standard methods⁸ and are summarized in Table 1. The structure was solved, refined, and prepared for publication using the SHELXTL package (structure solution refinements and molecular graphics).⁹ The structure was solved using direct methods and refined on F^2 using full matrix least squares techniques with anisotropic displacement factors for all non-hydrogen atoms. The amino and methanol

Table 1. Crystal data and structure refinement for the complex

Parameter	Value
Molecular formula	$C_{15}H_{13}MoN_3O_4$
Formula weight	395.22
Temperature (K)	298(2)
Crystal system	triclinic
Space group	$P\bar{1}$
a (Å)	9.7363(14)
b (Å)	11.7491(17)
c (Å)	13.908(2)
α (°)	102.355(2)
β (°)	94.650(2)
γ (°)	104.522(2)
V (Å ³)	1489.1(4)
Z	4
D_{calc} (g/cm ³)	1.763
Absorption coefficient (mm ⁻¹)	0.905
$F(000)$	792
Reflections collected	12376
Independent reflection (R_{int})	5427 (0.0357)
Reflections observed ($I > 2\sigma(I)$)	4342
Data/restraints/parameters	5427/6/429
Goodness-of-fit on F^2	1.091
Final R indices ($I > 2\sigma(I)$)	0.0469, 0.1137
R indices (all data)	0.0612, 0.1232
Highest peak and deepest hole (e Å ⁻³)	1.072, -0.969

Table 2. Selected bond lengths (Å) and angles (°) for the complex

Bond lengths (Å)			
Mo(1)–O(1)	1.912(3)	Mo(1)–O(2)	1.992(3)
Mo(1)–O(3)	1.693(4)	Mo(1)–O(4)	1.706(4)
Mo(1)–N(1)	2.231(4)	Mo(2)–O(5)	1.905(3)
Mo(2)–O(6)	1.993(3)	Mo(2)–O(7)	1.691(4)
Mo(2)–N(4)	2.242(4)	Mo(2)–O(8)	1.700(3)
Bond angles (°)			
O(3)–Mo(1)–O(4)	106.7(2)	O(3)–Mo(1)–O(1)	99.78(18)
O(4)–Mo(1)–O(1)	101.66(17)	O(3)–Mo(1)–O(2)	97.34(17)
O(4)–Mo(1)–O(2)	96.57(16)	O(1)–Mo(1)–O(2)	150.13(15)
O(3)–Mo(1)–N(1)	98.35(18)	O(4)–Mo(1)–N(1)	153.65(17)
O(1)–Mo(1)–N(1)	81.61(15)	O(2)–Mo(1)–N(1)	71.79(14)
O(7)–Mo(2)–O(8)	106.79(18)	O(7)–Mo(2)–O(5)	100.15(17)
O(8)–Mo(2)–O(5)	101.52(16)	O(7)–Mo(2)–O(6)	96.83(16)
O(8)–Mo(2)–O(6)	97.92(16)	O(5)–Mo(2)–O(6)	149.17(14)
O(7)–Mo(2)–N(4)	96.62(16)	O(8)–Mo(2)–N(4)	155.41(16)
O(5)–Mo(2)–N(4)	81.21(14)	O(6)–Mo(2)–N(4)	71.37(13)

H atoms were located from a difference Fourier map and refined isotropically, with N–H, O–H, and H...H distances restrained to 0.90(1), 0.85(1), and 1.43(2) Å, respectively. Positions of the remaining hydrogen atoms were calculated from the geometry of the molecular skeleton and their thermal displacement parameters were refined isotropically on a groupwise basis. Selected bond lengths and angles are reported in Table 2. H-bonding distances and angles are shown in Table 3.

Table 3. Hydrogen bonding interactions (Å, °)

<i>D</i> – <i>H</i> ... <i>A</i>	<i>d</i> (<i>D</i> – <i>H</i>)	<i>d</i> (<i>H</i> ... <i>A</i>)	<i>d</i> (<i>D</i> ... <i>A</i>)	Angle (<i>D</i> – <i>H</i> ... <i>A</i>)
N(3)–H(3A)...N(2)	0.90(1)	2.12(4)	2.795(5)	131(5)
N(6)–H(6B)...O(4) ⁱ	0.90(1)	2.50(3)	3.311(5)	151(5)
N(6)–H(6A)...N(5)	0.90(1)	2.13(4)	2.810(5)	132(5)
N(3)–H(3B)...O(8)	0.90(1)	2.51(3)	3.308(5)	149(5)

Symmetry code: i) *x*, 1 + *y*, –1 + *z*.

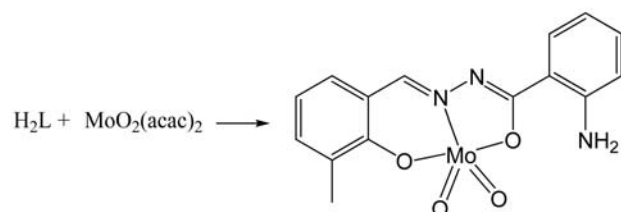
2. 5. Catalytic Epoxidation of Olefins

To a solution of olefins (0.28 mmol), NaHCO₃ (0.11 mmol) and catalyst (9.4 × 10^{–4} mmol) in MeCN (0.5 mL) was added H₂O₂ (1.1 mmol, 30% H₂O₂ in water) as oxidant. After the reaction was over, for the products analysis, the solution was subjected to multiple ether extraction, and the extract was also concentrated down to 0.5 mL by distillation in a rotary evaporator at room temperature and then a sample (2 μL) was taken from the solution and analyzed by GC. The retention times of the peaks were compared with those of commercial standards, and chlorobenzene was used as an internal standard for GC yield calculation.

3. Results and Discussion

3. 1. Synthesis and Characterization

The reaction of MoO₂(acac)₂ with the tridentate aroylhydrazine ligand H₂L in methanol led to the formation of *cis*-MoO₂ complex in a good yield (Scheme 2). The complex is stable at room temperature and soluble in DMSO, DMF, ethanol, acetonitrile and less soluble in ot-



Scheme 2. Synthesis procedure of the complex

her common solvents like dichloromethane, chloroform, and insoluble in benzene, *n*-hexane and CCl₄.

3. 2. ¹H NMR Spectra

¹H NMR data of the aroylhydrazine ligand when compared with the complex reveals that the ligand serves as tridentate binategative ONO donor with amino group remaining free. The amino residue of the ligand shows signal for N–H at 11.05 ppm, which is found unperturbed in the complex. The disappearance of the signal at 12.10 ppm in the free ligand upon complexation suggests that during complexation the [–CO–NH–] group undergoes tautomerization to [–C(OH)=N–] followed by deprotonation of the –OH group. The azomethine C–H signal in the complex is shifted up-field from its original position in the free ligand upon coordination of the –CH=N– group, on account of reduction of electron density at the azomethine C–H. The aromatic protons also show some deviation in the complex as compared to the free ligand since in the complex they are in direct conjugation to the coordinated O and N of the hydrazine ligands.

3. 3. IR Spectra

IR spectrum of the free aroylhydrazine ligand shows multiple bands at 3232 cm^{–1} for ν(N–H) of amino group, 1650 cm^{–1} for ν(C=O) and 3431 cm^{–1} for ν(O–H).¹⁰ The ν(C=O) band is absent in the spectrum of the complex as the ligand binds in binategative mode losing protons from the carbohydrazide group. The strong peak at 1611 cm^{–1} can be assigned to ν(C=N).¹¹ The complex exhibits two characteristic bands at 949 cm^{–1} and 861 cm^{–1} for symmetric and antisymmetric stretching of *cis* MoO₂ core.¹²

3. 4. Structure Description

The perspective view of the complex together with the atom numbering scheme are shown in Fig. 1. The asymmetric unit of the complex contains two complex molecules which are linked by hydrogen bonds. The coordination geometry around each molybdenum atom in the complex reveals a distorted square pyramidal environment with an NO₄ chromophore. The ligand molecule behaves as binategative tridentate one binding through the phenolate oxygen, the enolate oxygen and the imine nitrogen and occupies three positions in the basal plane. The fourth donor of the basal plane is furnished by one oxo group. The other oxo group is located at the apical position. The Mo atoms are found to be deviated from the corresponding mean basal planes by 0.365(1) Å for Mo(1) molecule and 0.354(1) Å for Mo(2) molecule. The C(8)–O(2) and C(23)–O(6) bond lengths are closer to single bond length rather than C–O double bond length. However, the shorter length compared to C–O single bond

may be attributed to extended electron delocalization in the ligand. Similarly shortening of C(8)–N(2) and C(23)–N(5) lengths together with the elongation of N(1)–N(2) and N(4)–N(5) lengths also supports the electron cloud delocalization in the ligand system. The ligand molecules form five-membered and six-membered chelate rings with the Mo centers.

The question arises as to whether the coordination polyhedra around the five-coordinated Mo atoms can be described as distorted square pyramid or distorted trigonal bipyramid. Further information can be obtained by determining the structural index τ which represents the relative amount of trigonality (square pyramid, $\tau = 0$; trigonal bipyramid, $\tau = 1$); $\tau = (\beta - \alpha)/60^\circ$, α and β being the two largest angles around the central atom.¹³ The values of τ are 0.059 for Mo(1) and 0.104 for Mo(2). The coordination geometries of the Mo atoms in the complex can therefore be described as distorted square pyramids. Molecules are linked through intermolecular hydrogen bonds to form 2D sheets (Fig. 2).

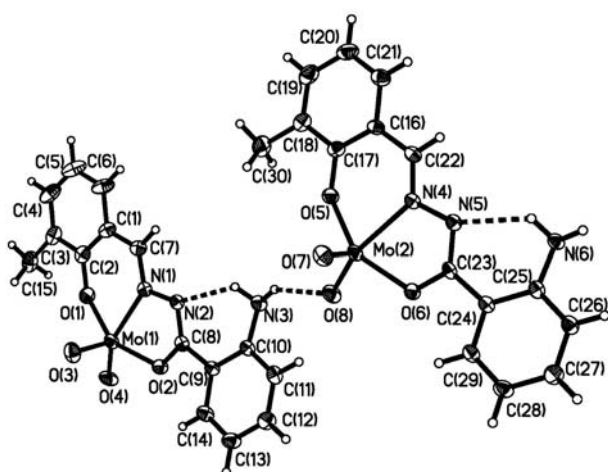


Figure 1. An ORTEP diagram of the complex with atom labeling scheme and 30% probability thermal ellipsoids for all non-hydrogen atoms. Hydrogen bonds are shown as dashed lines.

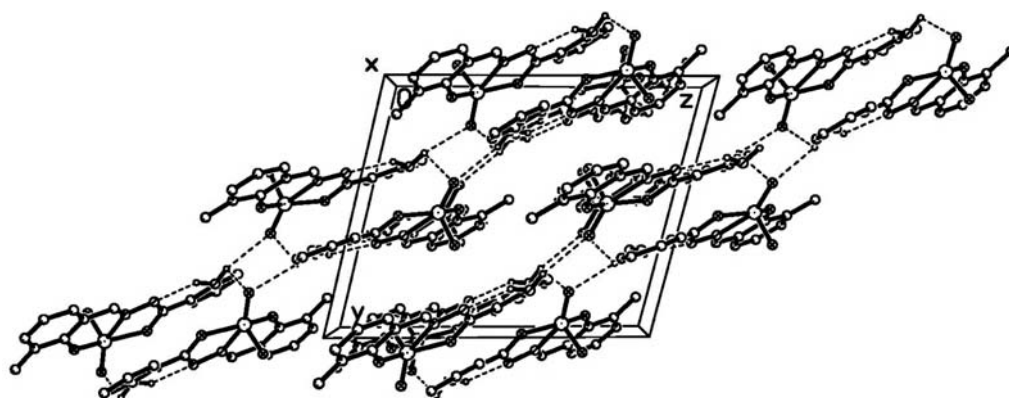
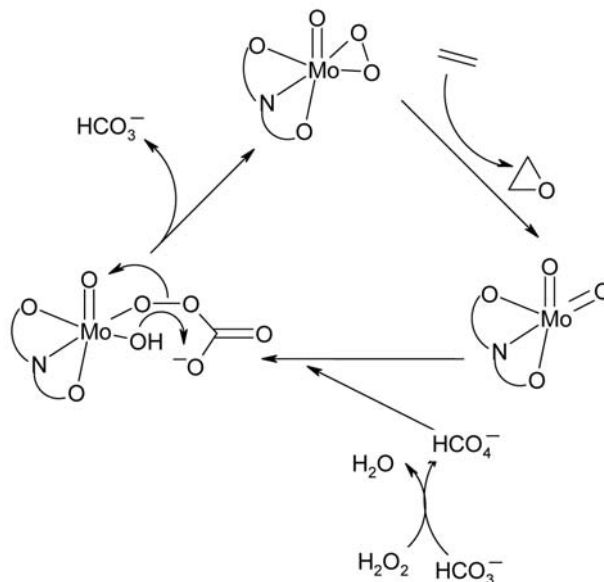


Figure 2. Packing diagram of the complex, viewed along the *x* axis. Hydrogen bonds are shown as dashed lines.



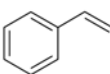
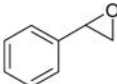
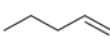



3. 5. Catalytic Property

The complex showed effective catalytic property in the oxidation of various olefins to their corresponding epoxides. The details of catalytic properties with respect to epoxidation of olefins with the complex as catalyst are given in Table 4. Excellent epoxide yields and selectivity were observed for all aliphatic and aromatic substrates. The results of catalytic studies using dioxomolybdenum catalysts reveal that the efficiency of catalyst toward all the substrates is similar with maximum conversion, TON, and selectivity. The mechanism for the epoxidation of various olefins to epoxides using the two complexes as catalysts has been proposed by comparison with the literature report (Scheme 3).¹⁴ When H₂O₂ was used as a sole oxidant the catalytic efficiency is not high, but when NaHCO₃ was added as a co-catalyst the efficiency of the system increases many times. The key aspect of such a reaction is that H₂O₂ and hydrogen carbonate react in an equilibrium process to



Scheme 3. Proposed mechanism of catalytic oxidation process

Table 4. The catalytic oxidation results^a

Substrate	Product	Conversion (%) ^b (TON) ^c	Selectivity (%) ^d
		100 (321)	100
		100 (305)	100
		91 (287)	100
		93 (292)	100

^a The molar ratios for catalyst: substrate: NaHCO₃:H₂O₂ are 1:298:117:1170. The reactions were performed in (70:30) mixture of CH₃OH/CH₂Cl₂ (1.2 mL) under air at room temperature. ^b The GC conversion (%) are measured relative to the starting olefin after 74.5 min. ^c TON = (mmol of product)/mmol of catalyst. ^d Selectivity to epoxide = (epoxide%/(epoxide% + aldehyde%)) × 100.

produce peroxydicarbonate, HCO₄, which is a more reactive nucleophile than H₂O₂ and speeds up the epoxidation reaction.¹⁵ The basic principle of the catalytic reaction is the conversion of oxoperoxo-complex to dioxo-complex transferring oxo species to the olefins and the conversion of dioxo complex to the oxoperoxo complex reacting with HCO₄⁻ to regain the catalytic activity.

4. Conclusion

A novel dioxidomolybdenum(VI) complex with aroylhydrazone ligand 2-amino-*N'*-(2-hydroxy-3-methylbenzylidene)benzohydrazide has been prepared and structurally characterized using X-ray structure analysis, FT-IR and ¹H NMR spectra. The complex has square pyramidal geometry with positions around the central atom being occupied with donor atoms of the aroylhydrazone ligand, and two oxo groups. The complex showed effective catalytic property in the oxidation of various olefins to their corresponding epoxides.

5. Supplementary Material

CCDC reference number 1435811 contains the supplementary crystallographic data for this article. These data can be obtained free of charge at <http://www.ccdc.cam.ac.uk>, or from Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033; Email: deposit@ccdc.cam.ac.uk.

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Povzetek

Sintetizirali smo nov dioksidomolibdenov kompleks, [MoO₂L], z vezavo troveznega aroilhidrazona 2-amino-*N'*-(2-hidroksi-3-metilbenziliden)benzohidrazida (H₂L) in ga okarakterizirali z elementno analizo, FT-IR, ¹H NMR in monokristalno rentgensko difrakcijo. Kompleks ima popačeno kvadratno piramidalno strukturo, kjer je aroilhidrazonski ligand dinegativni donor, medtem ko amino-*N* skupina ni koordinirana na kovinski center. Monokristalna strukturna analiza razkrije, da je Mo(VI) center koordiniran z donorskimi atomi dinegativnega aroilhidrazonskega liganda in dveh oksido skupin. Kristalna struktura je stabilizirana z vodikovimi vezmi. Kompleks je učinkovit katalizator za epoksidacijo olefinov z vodikovim peroksidom kot oksidantom in z natrijevim hidrogenkarbonatom kot kokatalizatorjem.