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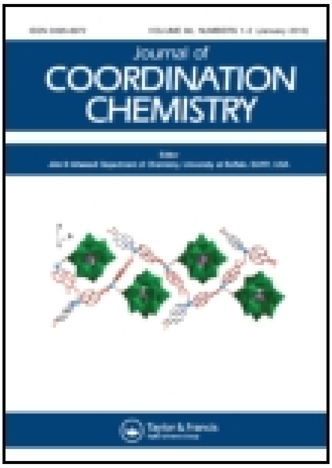

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gcoo20

Syntheses and structures of N′-(5-bromo-2-hydroxybenzylidene)-4-methoxybenzohydrazide and its dioxomolybdenum(VI) complex with catalytic epoxidation property

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Accepted author version posted online: 16 May 2013.Published online: 11 Jun 2013.

To cite this article: Yong-Ming Cui , Yan Wang , Ying-Jie Cai , Xue-Jun Long & Wu Chen (2013) Syntheses and structures of N′-(5-bromo-2-hydroxybenzylidene)-4-methoxybenzohydrazide and its dioxomolybdenum(VI) complex with catalytic epoxidation property, Journal of Coordination Chemistry, 66:13, 2325-2334, DOI: 10.1080/00958972.2013.805213

To link to this article: http://dx.doi.org/10.1080/00958972.2013.805213

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Journal of Coordination Chemistry, 2013
Vol. 66, No. 13, 2325–2334, http://dx.doi.org/10.1080/00958972.2013.805213

Syntheses and structures of *N′*-(5-bromo-2-hydroxybenzylidene)-4-methoxybenzohydrazide and its dioxomolybdenum(VI) complex with catalytic epoxidation property

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(*Received 12 January 2013; in final form 2 April 2013*)

A new Schiff base *N′*-(5-bromo-2-hydroxybenzylidene)-4-methoxybenzohydrazide (H_2BMH) and its dioxomolybdenum(VI) complex [MoO_2(BMH)(EtOH)] have been synthesized and characterized by elemental analysis, IR spectra, and single crystal X-ray determination. The Schiff base crystallized in the orthorhombic space group $P2_12_12_1$ and the complex crystallized in the triclinic space group $P\bar{1}$. The Schiff base coordinates to Mo through the phenolate O, imine N, and enolic O. The asymmetric unit of the complex contains two mononuclear dioxomolybdenum molecules. Each Mo is six-coordinate octahedral. The only significant difference between the complex and the ligand is the geometry involving donors. The coordination of the ligand to Mo is also reflected in the IR spectra. The complex shows high catalytic property and selectivity in epoxidation of cyclohexene with *t*-butylhydroperoxide as oxidant.

Keywords: Schiff base; Dioxomolybdenum(VI) complex; Crystal structure; Coordination compound; Epoxidation

1. Introduction

Molybdenum complexes have been investigated as oxidation catalysts for a variety of organic substrates [1–4], particularly for sulfoxidation and epoxidation of olefins [5–8]. Schiff bases, especially those of hydrazone type, have attracted interest in coordination chemistry [9, 10]. Dioxomolybdenum complexes derived from such hydrazone type Schiff bases have been reported to possess effective catalytic properties [11–13]. However, the number of such complexes is small. Recently, we reported some complexes derived from hydrazone type Schiff bases [14, 15]. As a further study of the coordination of such ligands to molybdenum, in the present work, a new Schiff base *N′*-(5-bromo-2-hydroxybenzylidene)-4-methoxybenzohydrazide (H_2BMH) and its dioxomolybdenum(VI) complex [MoO_2(BMH)(EtOH)] (scheme 1) have been synthesized and characterized. The particular interest is to compare the various properties related to the uncoordinated and coordinated molecule.

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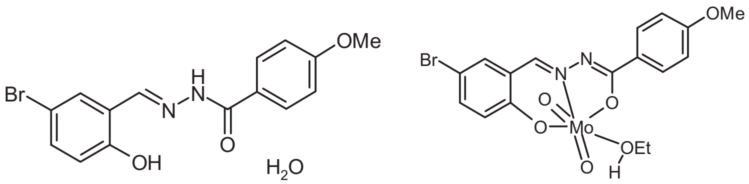


Scheme 1. H_2BMH and $[MoO_2(BMH)(EtOH)]$.

2. Experimental

2.1. *Materials and methods*

5-Bromosalicylaldehyde and 4-methoxybenzohydrazide were purchased from Alfa Aesar. $[MoO_2(acac)_2]$ was prepared according to the literature method [16]. All chemicals and solvents were of analytical grade and used as obtained. Microanalyses of the Schiff bases and dioxomolybdenum(VI) complex were performed with a Vario EL III CHNOS elemental analyzer. Infrared spectra were recorded as KBr pellets with an FTS-40 spectrophotometer.

2.2. *Synthesis of N'-(5-bromo-2-hydroxybenzylidene)-4-methoxybenzohydrazide (H_2BMH)*

A mixture of 5-bromosalicylaldehyde (2.01 g, 10 mM) and 4-methoxybenzohydrazide (1.66 g, 10 mM) in 50 mL ethanol was refluxed for 2 h and cooled to room temperature. The colorless solution was left in air for a week until most of the solvent was evaporated, yielding block-shaped single crystals of H_2BMH, isolated by filtration, and dried in air. Yield 2.82 g (77%). Anal. Calcd for $C_{15}H_{15}BrN_2O_4$ (%): C, 49.1; H, 4.1; N, 7.6. Found: C, 48.9; H, 4.1; N, 7.7. IR (KBr, ν_{max}/cm^{-1}): 3417 (OH), 3228 (NH), 1651 (C=O), 1615 (C=N).

2.3. *Synthesis of $[MoO_2(BMH)(EtOH)]$*

A stirred solution of $H_2BMH{\cdot}H_2O$ (0.367 g, 1 mM) in ethanol (20 mL) was mixed with $[MoO_2(acac)_2]$ (0.326 g, 1 mM) and the resulting reaction mixture was refluxed on a water bath for 1 h, and then cooled to room temperature, yielding a yellow solution. The solution was left at ambient temperature to slowly evaporate solvent for a week, yielding orange block-shaped single crystals of the complex. Yield 0.313 g (60%). Anal. Calcd for $C_{17}H_{17}BrMoN_2O_6$ (%): C, 39.2; H, 3.3; N, 5.4. Found: C, 39.0; H, 3.4; N, 5.2. IR (KBr, ν_{max}/cm^{-1}): 3410 (OH), 1609 (C=N), 1359 (C–O, enolic), 1259 (C–O, phenolate), 1031 (N–N), 915 (Mo=O), 642, 567, 466 (Mo–O, Mo–N).

2.4. *X-ray structure determination*

Data were collected on a Bruker SMART 1000 CCD area diffractometer using graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at 298(2) K and corrected with SADABS [17]. The structures were solved by direct methods [18] and refined on F^2 by

Table 1. Crystallographical and experimental data for H_2BMH and $[MoO_2(BMH)(EtOH)]$.

Compound	H_2BMH	$[MoO_2(BMH)(EtOH)]$
Formula	$C_{15}H_{15}BrN_2O_4$	$C_{17}H_{17}BrMoN_2O_6$
Mr	367.2	521.2
T (K)	298(2)	298(2)
Crystal shape/color	Block/colorless	Block/orange
Crystal size/mm^3	0.18 × 0.17 × 0.15	0.23 × 0.20 × 0.20
Crystal system	Orthorhombic	Triclinic
Space group	$P2_12_12_1$	$P\bar{1}$
a/Å	4.485(1)	7.825(1)
b/Å	6.682(1)	11.090(1)
c/Å	51.149(2)	22.471(2)
α/°	90	88.312(2)
β/°	90	87.747(2)
γ/°	90	75.371(2)
V/Å^3	1533.0(1)	1885.0(2)
Z	4	4
D_c/(g/cm^{-3})	1.591	1.836
β (Mo$K\alpha$) (cm^{-1})	3.841	2.852
$F(000)$	744	1032
R_{int}	0.0534	0.0359
Unique reflections	2841	7152
Observed reflections $I \geqslant 2\sigma(I)$	2627	5188
Min. and max. transmission	0.5448/0.5965	0.5600/0.5993
Parameters	210	491
Restraints	3	0
Goodness-of-fit on F^2	1.064	1.217
R_1, wR_2 $[I \geqslant 2\sigma(I)]^a$	0.0333, 0.0800	0.0651, 0.1459
R_1, wR_2 (all data)^a	0.0375, 0.0824	0.0949, 0.1552
Large diff. peak and hole/(e.Å^{-3})	0.376/−0.571	1.207/−0.860

$^aR_1 = \sum||Fo|\text{-}|Fc||/\sum|Fo|$; $wR_2 = [\sum w\,(Fo^2 - Fc^2)^2/\sum w(Fo^2)^2]^{1/2}$.

full-matrix least-squares using SHELXL [19]. All non-hydrogen atoms were refined anisotropically. H2 attached to N2 and the water hydrogens in H_2BMH were located from difference Fourier maps and refined isotropically, with N–H, O–H, and H···H distances restrained to 0.90(1), 0.85(1), and 1.37(2) Å, respectively. The other hydrogens were placed in calculated positions and included in the last cycle of refinement. Crystal data and details of the data collection and refinement are listed in table 1. Selected bond lengths and angles are listed in table 2. Hydrogen bonding parameters are given in table 3.

2.5. *Catalytic epoxidation test*

Epoxidation of cyclohexene by dioxomolybdenum(VI) complex with *tert*-butyl hydroperoxide (TBHP) was carried out according to the following procedure. A mixture of catalyst (0.003 mM), cyclohexene (6.56 g, 0.08 M), and 1,2-dichloroethane (5 mL) was placed in a three-necked round-bottomed flask equipped with a condenser and a magnetic stirrer. The mixture was stirred for 5 min at 353(2) K and then 2 mL (0.02 M) anhydrous TBHP was added. The reaction was monitored at certain time intervals to determine the concentrations of TBHP and 1,2-epoxycyclohexene by GC and was left to proceed until near complete conversion of TBHP.

Table 2. Selected bond lengths/Å and angles/° for H_2BMH and [MoO_2(BMH)(EtOH)].

H_2BMH			
N1–C7	1.284(4)	N1–N2	1.377(3)
N2–C8	1.354(4)	C8–O2	1.228(4)
C2–O1	1.350(4)		
[MoO_2(BMH)(EtOH)]			
Mo1–O1	1.929(6)	Mo1–O2	2.016(6)
Mo1–N1	2.243(7)	Mo1–O4	2.321(6)
Mo1–O5	1.688(6)	Mo1–O6	1.690(7)
Mo2–O7	1.928(6)	Mo2–O8	2.003(5)
Mo2–N3	2.252(7)	Mo2–O10	2.368(7)
Mo2–O11	1.697(6)	Mo2–O12	1.709(7)
N1–C7	1.29(1)	N1–N2	1.397(9)
N2–C8	1.32(1)	C8–O2	1.31(1)
C2–O1	1.34(1)	N3–C24	1.29(1)
N3–N4	1.398(9)	N4–C25	1.30(1)
C25–O8	1.32(1)	C19–O7	1.35(1)
O5–Mo1–O6	103.8(3)	O5–Mo1–O1	103.3(3)
O6–Mo1–O1	99.6(3)	O5–Mo1–O2	97.5(3)
O6–Mo1–O2	96.2(3)	O1–Mo1–O2	149.8(3)
O5–Mo1–N1	158.6(3)	O6–Mo1–N1	95.8(3)
O1–Mo1–N1	81.3(2)	O2–Mo1–N1	71.7(2)
O5–Mo1–O4	83.9(3)	O6–Mo1–O4	171.6(3)
O1–Mo1–O4	81.5(3)	O2–Mo1–O4	79.3(2)
N1–Mo1–O4	76.1(2)	O11–Mo2–O12	105.4(3)
O11–Mo2–O7	102.0(3)	O12–Mo2–O7	98.7(3)
O11–Mo2–O8	98.4(3)	O12–Mo2–O8	95.8(3)
O7–Mo2–O8	150.9(3)	O11–Mo2–N3	155.7(3)
O12–Mo2–N3	97.8(3)	O7–Mo2–N3	81.5(2)
O8–Mo2–N3	71.5(2)	O11–Mo2–O10	81.7(3)
O12–Mo2–O10	171.8(3)	O7–Mo2–O10	83.5(3)
O8–Mo2–O10	79.0(2)	N3–Mo2–O10	74.7(2)

Table 3. Hydrogen bonding for the ligand and the complex.

D–H···*A*	*d*(*D*–H) (Å)	*d*(H···*A*) (Å)	*d*(*D*···*A*) (Å)	∠(*D*–H···*A*) (°)
H_2BMH				
O1–H1···N1	0.82	1.97	2.678(3)	144
O1–H1···O4^{i}	0.82	2.58	3.062(3)	119
N2–H2···O4^{ii}	0.90(1)	2.00(2)	2.886(3)	169(4)
O4–H4B···O1	0.85(1)	2.05(2)	2.880(4)	167(5)
[MoO_2(BMH)(EtOH)]				
O4–H4A···N2^{iii}	0.93	1.93	2.814(9)	159(3)
O10–H10A···N4^{iv}	0.93	1.94	2.819(9)	157(3)

Symmetry codes: (i) $-1+x, y, z$; (ii) $-1+x, -1+y, z$; (iii) $1-x, -y, -z$; (iv) $-x, -y, 1-z$.

3. Results and discussion

Reaction between equimolar quantities of [$MoO_2(acac)_2$] with H_2BMH in ethanol afforded [MoO_2(BMH)(EtOH)]. BMH adopts an enolic tautomeric form on complexation. The Schiff base crystallizes as colorless crystals and the dioxomolybdenum(VI) complex crystallizes as orange crystals. Both the ligand and the complex are stable in air at room temperature and soluble in common polar organic solvents, such as DMSO, DMF, MeOH, EtOH, and MeCN, but insoluble in water.

3.1. *IR spectra*

IR spectra of the Schiff base and the dioxomolybdenum(VI) complex provide information about metal-ligand bonding. Assignments are based on typical group frequencies. The medium and broad band at 3380–3450 cm^{-1} is assigned to ν(O–H). The ν(C=O) at 1651 cm^{-1} and the sharp ν(NH) of 3228 cm^{-1} for H_2BMH are absent in the complex, indicating enolization of the amide functionality and subsequent proton replacement by molybdenum, in accord with the results obtained by X-ray determination. The ν(C–O) (enolic) of the complex is at 1359 cm^{-1}. The strong band at 1615 cm^{-1} of the Schiff base is assigned to azomethine and observed at 1609 cm^{-1} in the complex, indicating coordination of the azomethine N to Mo [20]. The medium band observed at 915 cm^{-1} for the complex is assigned to Mo=O stretch [21]. Bands indicative of Mo–O and Mo–N vibrations are at 450–650 cm^{-1} [11, 22].

3.2. *Structures of the Schiff base ligand and the dioxomolybdenum(VI) complex*

Figures 1 and 2 give perspective views of the mono-hydrated Schiff base and the dioxomolybdenum(VI) complex together with atomic labeling systems. There is an intramolecular O1–H1···N1 hydrogen bond in the Schiff base and the dihedral angle between the two benzene rings is 21.1(3)°. All the bond lengths in the Schiff base are comparable to those observed in similar Schiff base compounds [23, 24].

The complex, derived from the Schiff base ligand, is a mononuclear dioxomolybdenum (VI) compound. There are two independent molecules, A and B (figure 2), in the asymmetric unit of the complex. BMH forms one five-membered and one six-membered chelate rings with bite angles of 71.7(2) and 81.3(2)° for molecule A, and of 71.5(2) and 81.5(2)° for molecule B. The Mo ions are in distorted octahedral O_5N coordination spheres. The phenolate O, imine N, and enolic O of BMH and one methoxy O constitute the equatorial planes, with mean deviations (the maximum deviation of the atoms constituting the mean plane) of 0.020(3) Å for molecule A and 0.064(3) Å for molecule B. The two axial positions are occupied by one oxo and one ethanol. Displacements of Mo from the equatorial planes toward the axial oxo are 0.312(2) Å for molecule A and 0.327(2) Å for molecule B. The *cis* bond angles are 71.5(2)–105.4(3)° and the *trans* bond angles are

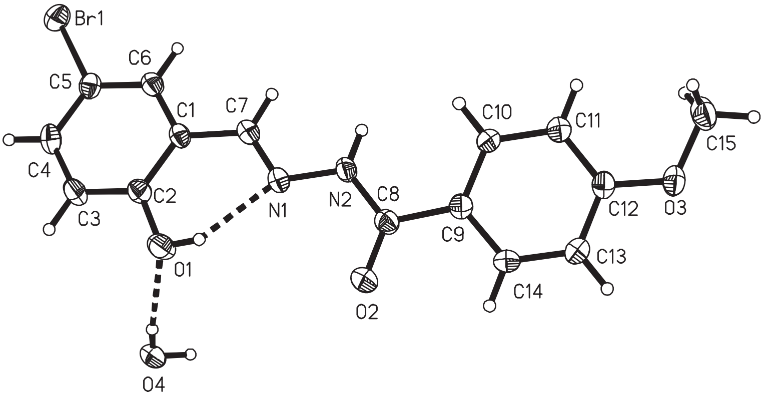


Figure 1. ORTEP plots (30% probability level) and atom labeling scheme for H_2BMH. Hydrogen bonds are shown as dashed lines.

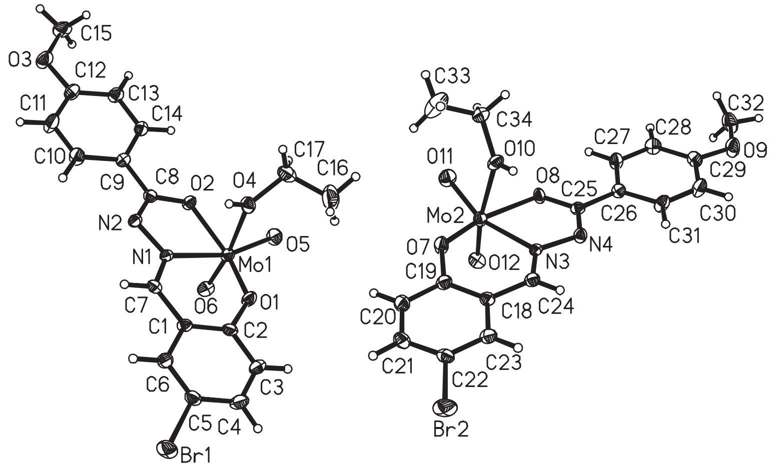


Figure 2. ORTEP plots (30% probability level) and atom labeling scheme for [MoO_2(BMH)(EtOH)].

149.8(3)–171.8(3)°. The Mo–O and Mo–N bond lengths are comparable with those reported for dioxomolybdenum(VI) complexes with similar ligands [25, 26]. As commonly observed in analogous species, the elongated Mo1–O4 and Mo2–O10 bonds *trans* to the oxo groups in the complex indicate weak coordination of ethanol at the axial positions. The dihedral angles between the two benzene rings in the complex are 5.7(2)° for molecule A and 5.5(2)° for molecule B. The relatively large deviations from linearity in the O1–Mo1–O2 and O7–Mo2–O8 angles (150°) are caused by the strain created by the five- and six-membered chelate rings. From the above analysis, it can be seen that molecules A and B are very similar.

The Schiff base coordinates to Mo through the phenolate O, imine N, and enolic O. The bond lengths of C7=N1 and C24=N3 [1.29(1) Å], and N1–N2 and N3–N4 [1.40(1) Å] in

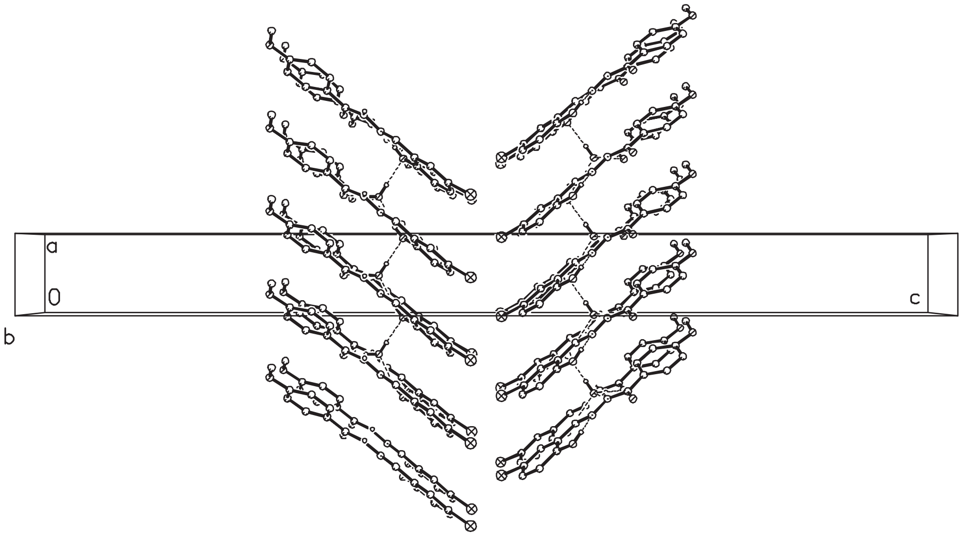


Figure 3. Molecular packing diagram for H_2BMH, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

the complex are similar to those [1.28(1) and 1.38(1) Å] in the Schiff base. Bond lengths of C2–O1 [1.34(1) Å] and C19–O7 [1.35(1) Å] in the complex are comparable to that [1.35(1) Å] in the Schiff base, which might be associated with the presence of the intramolecular O1–H1···N1 hydrogen bond. Distances between C8 and N2 [1.32(1) Å] and C25 and N4 [1.30(1) Å] in the complex are shorter than between C8 and N2 [1.35(1) Å] in the ligand, and the distances between C8 and O2 [1.31(1) Å] and C25 and O8 [1.32(1) Å] in the complex are longer than between C8 and O2 [1.23(1) Å] in the ligand, indicating enolization of N2–C8–O2 and N4–C25–O8 in the complex.

In the crystal structure of the Schiff base, molecules are linked via intermolecular N–H···O and O–H···O hydrogen bonds, forming a 3-D network, as shown in figure 3. In the crystal structure of the complex, adjacent two molecules are linked via intermolecular O–H···N hydrogen bonds, forming a dimer, as shown in figure 4.

3.3. *Catalytic epoxidation of cyclohexene*

According to Sobczak's idea [27], the probable mechanism of the catalytic epoxidation is shown in scheme 2. First, formation of the intermediate complex [MoO_2(BMH)(*t*-BuOOH)] and activation of TBHP. Second, interaction between cyclohexene and TBHP molecule activated in the coordination sphere of the molybdenum complex. Third, formation of epoxide and conversion of TBHP into *t*-BuOH. Finally, substitution of *t*-BuOH by TBHP and reproduction of the intermediate complex [MoO_2(BMH)(*t*-BuOOH)].

The catalytic activity of dioxomolybdenum(VI) complex was determined from the yield for epoxidation of cyclohexene using TBHP as oxidant. The yields in the reaction are

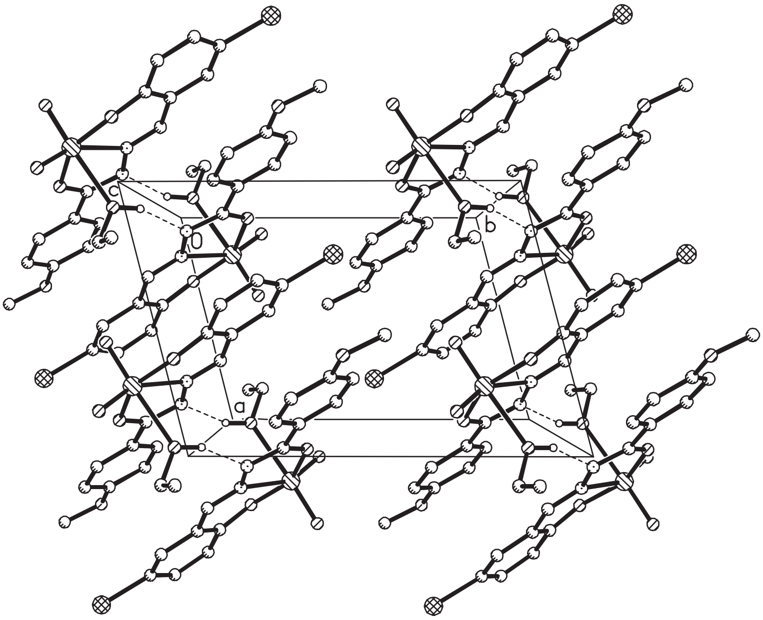
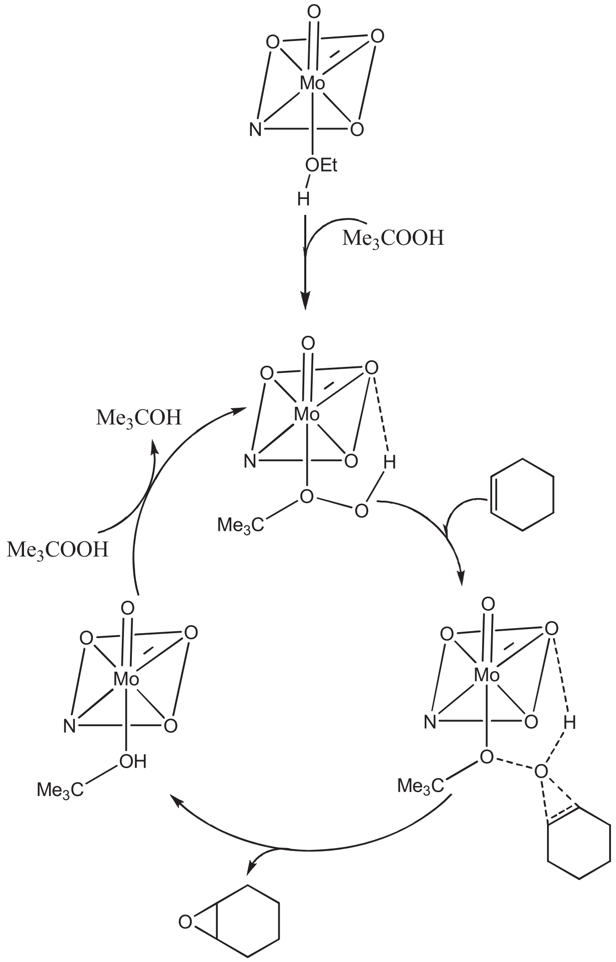


Figure 4. Molecular packing diagram for [MoO_2(BMH)(EtOH)], viewed along the *c* axis. Hydrogen bonds are shown as dashed lines.



Scheme 2. Probable mechanism of cyclohexene epoxidation with the complex as catalyst and TBHP as oxidant.

expressed as $Y = (C_{\text{epox}}/C_{\text{TBHP}}) \times 100\%$, where C_{epox} and C_{TBHP} (M L^{-1}) are the concentration of epoxide formed and initial concentration of TBHP, respectively. The selectivity with respect to TBHP reacted was calculated by the ratio $S = (C_{\text{epox}}/(C_{\text{TBHP}} - C)) \times 100$, where C is the final TBHP concentration. The yields are shown in figure 5. After 20 min of reaction, the yield is nearly 100% with selectivity at 100%. This is comparable to the catalytic property of a multiwall carbon nanotube supported tungsten hexacarbonyl [28], which can convert the cyclohexene to its epoxide with 100% yield and conversion within 2 h. When compared to the multiwall carbon nanotube supported manganese(III) tetraphenylporphyrin (97% conversion and 97% epoxide yield within 150 min) [29], the complex in the present work has more efficient catalysis. Maurya and coworkers reported oxidation of cyclohexane with TBHP catalyzed by a Schiff base copper(II) complex [30]. Bagherzadeh and Zare reported oxidation of olefins with a Schiff base manganese(III) complex [31]. However, the conversion and selectivity of the copper(II) and manganese(III) complexes are low. In addition, there are many other species including vanadium [32, 33] having efficient

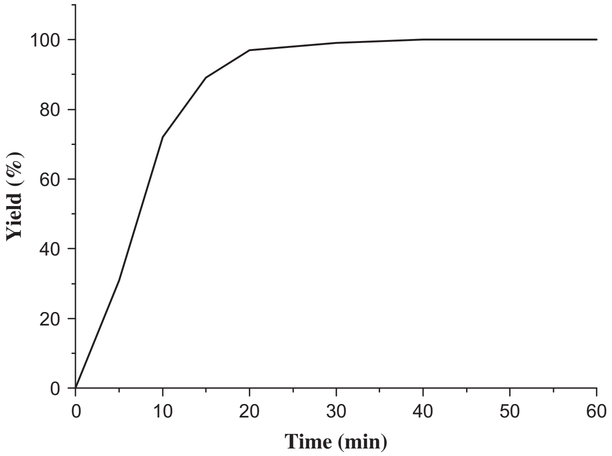


Figure 5. Yield and time curve of the catalytic epoxidation reaction.

catalytic epoxidation properties. In general, vanadium and molybdenum complexes are more efficient for catalytic epoxidation with higher selectivity than other metal complexes.

4. Conclusion

Using new tridentate Schiff base ligand prepared from 5-bromosalicylaldehyde with 4-methoxybenzohydrazide in ethanol, a new dioxomolybdenum(VI) complex has been synthesized. The structures of the Schiff base and the complex were characterized and compared. The dianionic Schiff base ligand coordinates to Mo through phenolate-O, imine-N, and enolic-O. The complex shows high catalytic activity and selectivity in epoxidation of cyclohexene with TBHP as oxidant. Further work needs to be done to explore its application in various organic syntheses.

Supplementary material

CCDC 919060 and 919061 contain the supplementary crystallographic data for this article. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgment

This work was financed by the Education Office of Hubei Province (Q20101610).

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