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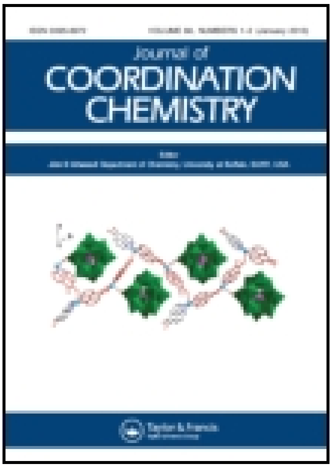

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Synthesis and structures of two molybdenum(VI) complexes derived from similar benzohydrazone ligands with catalytic properties

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Synthesis and structures of two molybdenum(VI) complexes derived from similar benzohydrazone ligands with catalytic properties

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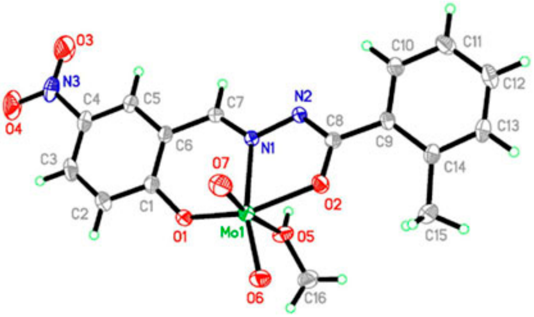

Two new structurally similar molybdenum(VI) complexes, $[MoO_2L^1(CH_3OH)]$ (**1**) and $[MoO_2L^2(CH_3OH)]$ (**2**), where L^1 is the dianionic form of *N′*-(2-hydroxy-5-nitrobenzylidene)-2-methylbenzohydrazide and L^2 is the dianionic form of *N′*-(2-hydroxy-4-methoxybenzylidene)-2-methylbenzohydrazide, were prepared and structurally characterized by elemental analysis, infrared spectra, and single-crystal X-ray diffraction. **1** crystallizes in the monoclinic space group $P2_1/c$, with unit cell dimensions $a = 7.941(1)$, $b = 14.337(2)$, $c = 15.141(2)$ Å, $\beta = 92.782(2)°$, $V = 1721.8(4)$ Å^3, $Z = 4$, $R_1 = 0.0286$, $wR_2 = 0.0650$, GOOF = 1.028. **2** crystallizes in the triclinic space group *P*-1, with unit cell dimensions $a = 8.003(1)$, $b = 10.608(1)$, $c = 10.880(1)$ Å, $\alpha = 95.745(2)°$, $\beta = 97.627(2)°$, $\gamma = 105.762(2)°$, $V = 872.0(2)$ Å^3, $Z = 2$, $R_1 = 0.0226$, $wR_2 = 0.0595$, GOOF = 1.116. X-ray analysis indicates that Mo in the complexes are coordinated by the phenolate oxygen, imino nitrogen, and enolate oxygen of the benzohydrazone, methanol, and two oxo groups, generating octahedral coordination. The oxidation of olefins with the complexes as catalysts was evaluated, indicating that the complexes showed excellent catalytic efficiency in oxidation of most aliphatic and aromatic substrates under mild conditions using *tert*-butyl hydrogen peroxide as oxidant.

Keywords: Benzohydrazone ligand; Molybdenum complex; Crystal structure; Hydrogen bonding; Catalytic oxidation

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1. Introduction

Development of processes for oxidation of olefins is a goal long pursued. The challenge is to make such processes environmentally friendly, for instance, by using non-toxic reagents and energy-efficient catalytic methods [1]. Molybdenum complexes with various ligands have extensive use in organic synthesis, in particular for various oxidations of organic compounds [2–5]. Dioxomolybdenum complexes have attracted attention as oxidation catalysts for organic substrates, especially for the sulfoxidation and epoxidation of olefins [6–9]. The synthesis and reactivity studies of a number of dioxomolybdenum complexes with Schiff bases have been reported [10–13]. Some of the complexes possess oxygen atom transfer properties as they were found to oxidize thiols, hydrazine, polyketones, and tertiary phosphines [14, 15]. The catalytic capability of molybdenum(VI) complexes with benzohydrazone type Schiff base ligands towards oxidation of sulfides have received satisfactory results [16, 17]. However, the number of documented molybdenum(VI) complexes catalyzing the peroxidic oxidation of sulfides is still limited; rare reports on such complexes focus on the preparation and structural characterization [18–21]. The structure–activity relationship is not clear. In this work, two structurally similar molybdenum(VI) complexes, $[MoO_2L^1(CH_3OH)]$ (**1**) and $[MoO_2L^2(CH_3OH)]$ (**2**), where L^1 is the dianionic form of *N′*-(2-hydroxy-5-nitrobenzylidene)-2-methylbenzohydrazide (H_2L^1), bearing an electron-withdrawing group, and L^2 is the dianionic form of *N′*-(2-hydroxy-4-methoxybenzylidene)-2-methylbenzohydrazide (H_2L^2), bearing an electron-donating group, were prepared and studied for their structures and catalytic oxidation property on olefins (scheme 1).

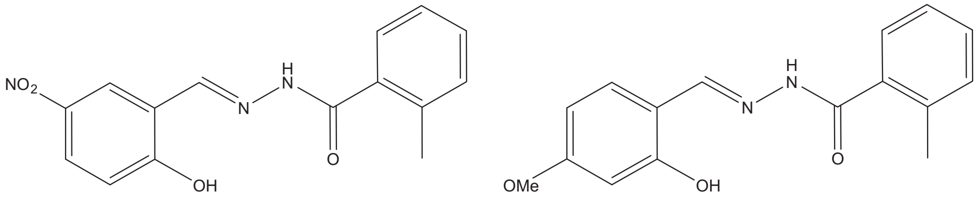
2. Experimental

2.1. *Materials and methods*

All chemicals and solvents were of analytical reagent grade and were purchased from Beijing Chemical Reagent Company. The benzohydrazones H_2L^1 and H_2L^2 were prepared according to the literature method [22]. Microanalyses (C, H, N) were performed using a Perkin-Elmer 2400 elemental analyzer. Infrared spectra were carried out using a JASCO FT-IR model 420 spectrophotometer with KBr disks from 4000 to 400 cm^{-1}. GC analyses were performed on a Shimadzu GC-2014 gas chromatograph.

2.2. *Syntheses of the complexes*

2.2.1. $[MoO_2L^1(CH_3OH)]$ (1). A methanolic solution (15 mL) of $MoO_2(acac)_2$ (0.33 g, 1.0 mM) was added to a methanolic solution (15 mL) of H_2L^1 (0.30 g, 1.0 mM). The



Scheme 1. The benzohydrazone ligands H_2L^1 (left) and H_2L^2 (right).

mixture was stirred at room temperature for 1 h to give a brown solution. Single crystals of the complex, suitable for X-ray diffraction, were formed at the bottom of the vessel on slow evaporation of the solution in air for a few days. Yield: 71%. Anal. Calcd for $C_{16}H_{15}MoN_3O_7$: C, 42.03; H, 3.31; N, 9.19. Found: C, 41.87; H, 3.40; N, 9.32%.

2.2.2. [$MoO_2L^2(CH_3OH)$] (2). This complex was prepared according to the same method as **1**, but with H_2L^1 replaced by H_2L^2 (0.28 g, 1.0 mM). Yield: 63%. Anal. Calcd for $C_{17}H_{18}MoN_2O_6$: C, 46.17; H, 4.10; N, 6.33. Found: C, 46.02; H, 4.18; N, 6.22%.

2.3. *X-ray structure determination*

X-ray measurements were performed using a Bruker Smart 1000 CCD diffractometer with graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) using the ω-scan technique. Determination of the Laue class, orientation matrix, and cell dimensions were performed according to established procedures where Lorentz polarization and absorption corrections were applied. Absorption corrections were applied by fitting a pseudo-ellipsoid to the

Table 1. Crystal data for **1** and **2**.

	1	**2**
Chemical formula	$C_{16}H_{15}MoN_3O_7$	$C_{17}H_{18}MoN_2O_6$
Fw	457.25	442.27
Crystal shape/color	Block/brown	Block/brown
Crystal size (mm)	0.27 × 0.26 × 0.23	0.32 × 0.30 × 0.27
T (K)	298(2)	298(2)
λ (MoKα) (Å)	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	*P2₁/c*	*P-1*
a (Å)	7.941(1)	8.003(1)
b (Å)	14.337(2)	10.608(1)
c (Å)	15.141(2)	10.880(1)
α (°)		95.745(2)
β (°)	92.782(2)	97.627(2)
γ (°)		105.762(2)
V (Å^3)	1721.8(4)	872.0(2)
Z	4	2
μ (MoKα) (cm^{-1})	0.808	0.789
T (min)	0.8113	0.7863
T (max)	0.8359	0.8152
D_c (g cm^{-3})	1.764	1.684
F (0 0 0)	920	448
Collected reflections	10,005	8468
Unique reflections	3725	3275
Observed reflections [$I \geq 2\sigma(I)$]	2641	3036
Parameters	249	241
Restraints	1	1
Goodness-of-fit on F^2	1.028	1.116
R_{int}	0.0244	0.0204
R_1 [$I \geq 2\sigma(I)$]	0.0286	0.0226
wR_2 [$I \geq 2\sigma(I)$]	0.0650	0.0595
R_1 (all data)	0.0491	0.0258
wR_2 (all data)	0.0741	0.0611

ψ-scan data of selected strong reflections over a wide range of 2θ angles. The positions of non-hydrogen atoms were located with direct methods. Subsequent Fourier syntheses were used to locate the remaining non-hydrogen atoms. All non-hydrogen atoms were refined anisotropically. The methanol hydrogens were located from difference Fourier maps and refined isotropically, with O–H distances restrained to 0.85(1) Å. The remaining hydrogens were placed in calculated positions and constrained to ride on their parent. The analysis was performed with SHELXS-97 and SHELXL-97 [23, 24]. The crystallographic data for the complexes are summarized in table 1. Selected bond lengths and angles are given in table 2.

2.4. *Oxidation*

Catalytic experiments were carried out in a 50-mL glass round-bottom flask fitted with a reflux condenser and placed in an oil bath at prearranged temperature under continuous stirring. The oxidation was carried out as follows: molybdenum(VI) complexes (0.032 mM) were dissolved in 10 mL 1,2-dichloroethane. Then, 10 mM alkene was added to the reaction mixture and 30 mM TBHP was added. The reaction mixture was refluxed for 1 h. The reaction products were monitored at time intervals using gas chromatography. The oxidation products were identified by comparison with authentic samples (retention times in GC).

Table 2. Selected bond lengths (Å) and angles (°) for **1** and **2**.

1			
Bond lengths			
Mo1–O1	1.934(2)	Mo1–O2	2.0123(19)
Mo1–O5	2.337(2)	Mo1–O6	1.695(2)
Mo1–O7	1.691(2)	Mo1–N1	2.246(2)
Bond angles			
O7–Mo1–O6	105.81(11)	O7–Mo1–O1	100.13(11)
O6–Mo1–O1	103.41(9)	O7–Mo1–O2	95.24(10)
O6–Mo1–O2	97.86(9)	O1–Mo1–O2	149.11(8)
O7–Mo1–N1	94.73(9)	O6–Mo1–N1	157.73(9)
O1–Mo1–N1	80.83(8)	O2–Mo1–N1	71.28(7)
O7–Mo1–O5	170.13(9)	O6–Mo1–O5	82.81(9)
O1–Mo1–O5	82.14(9)	O2–Mo1–O5	78.56(8)
N1–Mo1–O5	76.07(7)		
2			
Bond lengths			
Mo1–O1	1.9296(15)	Mo1–O2	2.0135(14)
Mo1–O4	2.3524(16)	Mo1–O5	1.6953(15)
Mo1–O6	1.6966(17)	Mo1–N1	2.2303(16)
Bond angles			
O5–Mo1–O6	106.01(8)	O5–Mo1–O1	102.84(7)
O6–Mo1–O1	98.70(8)	O5–Mo1–O2	97.87(7)
O6–Mo1–O2	96.75(7)	O1–Mo1–O2	149.52(6)
O5–Mo1–N1	156.00(7)	O6–Mo1–N1	96.58(7)
O1–Mo1–N1	81.20(6)	O2–Mo1–N1	71.03(6)
O5–Mo1–O4	81.75(7)	O6–Mo1–O4	171.49(7)
O1–Mo1–O4	82.64(6)	O2–Mo1–O4	78.45(6)
N1–Mo1–O4	75.27(6)		

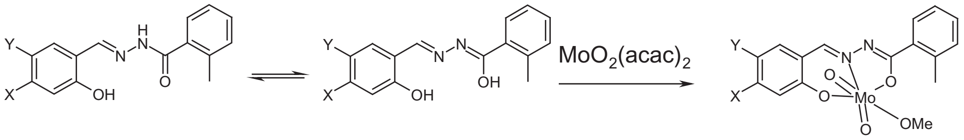
3. Results and discussion

3.1. *Synthesis*

The benzohydrazones were readily prepared by condensation of 2-methylbenzohydrazide with 5-nitrosalicylaldehyde and 4-methoxysalicylaldehyde, respectively, in methanol. The molybdenum(VI) complexes were synthesized by stirring equimolar quantities of benzohydrazones with $MoO_2(acac)_2$ in methanol at room temperature (scheme 2). The chemical formulas of the complexes have been confirmed by elemental analyses, IR spectra, and single-crystal X-ray diffraction.

3.2. *Structure description of 1 and 2*

The molecular structures of **1** and **2** are shown in figures 1 and 2, respectively. The coordination geometry around Mo can be described as distorted octahedral, with phenolate oxygen, imino nitrogen, and enolate oxygen of the benzohydrazone ligand, and one oxo defining the equatorial plane, and with the other oxo and one methanol occupying axial positions. The benzohydrazone ligands coordinate to Mo in a meridional fashion forming five- and six-membered chelate rings with bite angles of 71.28(7)° and 80.83(8)° for **1** and 71.03(6)° and 81.20(6)° for **2**. The dihedral angles between the benzene rings of the benzohydrazone ligands are 11.1(3)° for **1** and 6.1(3)° for **2**. The displacements of the Mo in **1** and **2** from the equatorial mean planes toward the apical oxo are 0.323(1) and 0.334(1) Å, respectively. The benzohydrazone ligands are coordinated in their dianionic form, which is evident from the N2–C8 and O2–C8 bond lengths. The abnormal bond values indicate the presence of the enolate form of the ligand amides. The Mo–O, Mo–N, and Mo=O bonds



Scheme 2. The preparation of the complexes, **1**: $X = H$, $Y = NO_2$; **2**: $X = OMe$, $Y = H$.

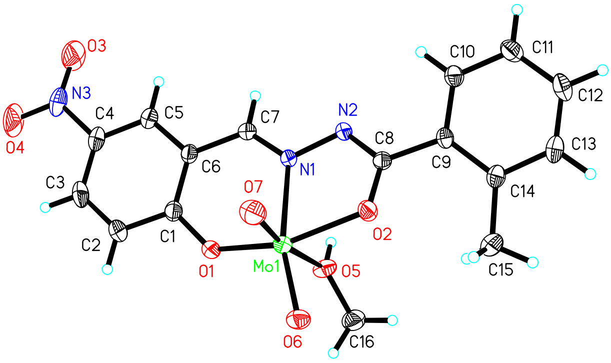


Figure 1. Molecular structure of **1** with 30% probability thermal ellipsoids.

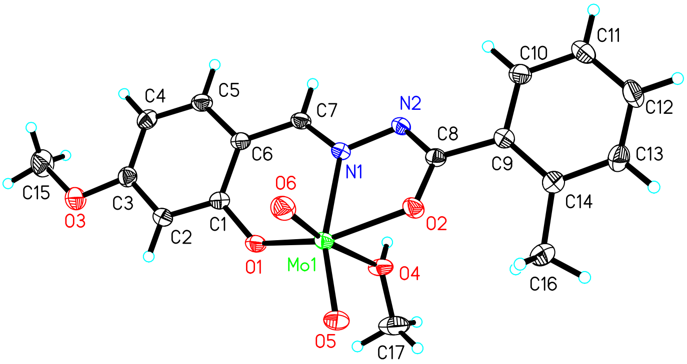


Figure 2. Molecular structure of **2** with 30% probability thermal ellipsoids. Atoms labeled with the suffix B and unlabeled are at the symmetry position 3/2 − *x*, 1/2 + *y*, 1/2 − *z*.

are within normal ranges and are similar to those observed in similar molybdenum(VI) complexes [18–21, 25–28]. The distances between Mo and methanol oxygen are much longer than the other bonds, which is not uncommon in similar structures. Complex **2** is very similar to that reported by Xu and Li [29] with the only difference in the position of the methyl groups of the ligands.

In the crystal structures of the complexes, adjacent [$MoO_2L(CH_3OH)$] molecules are linked through two intermolecular O–H···N hydrogen bonds [**1**: O5–H5 = 0.85(1) Å, H5···N2^{i} = 1.98(1) Å, O5···N2^{i} = 2.819(3) Å, O5–H5···N2^{i} = 171(4)°; **2**: O4–H4A = 0.85(1) Å, H4A···N2^{ii} = 1.92(1) Å, O4···N2^{ii} = 2.756(2) Å, O4–H4A···N2^{ii} = 171(3)°; symmetry codes: (i) $-x$, $2-y$, $-z$; (ii) $1-x$, $-y$, $1-z$], to form dimers (figure 3 for **1** and figure 4 for **2**).

3.3. *IR spectra*

The benzohydrazones show stretching bands attributed to C=O, C=N, C–OH and NH at 1650–1660, 1615–1618, 1180, and 3205–3225 cm^{-1}, respectively [30]. For the complexes,

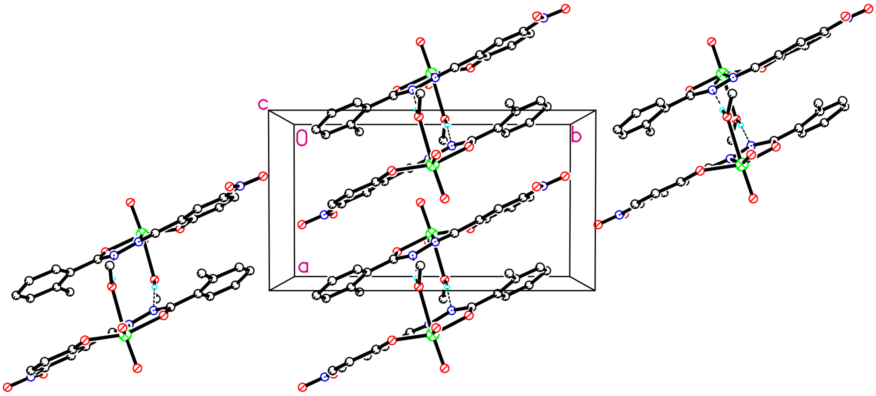


Figure 3. Molecular packing of **1** viewed along the *c* axis.

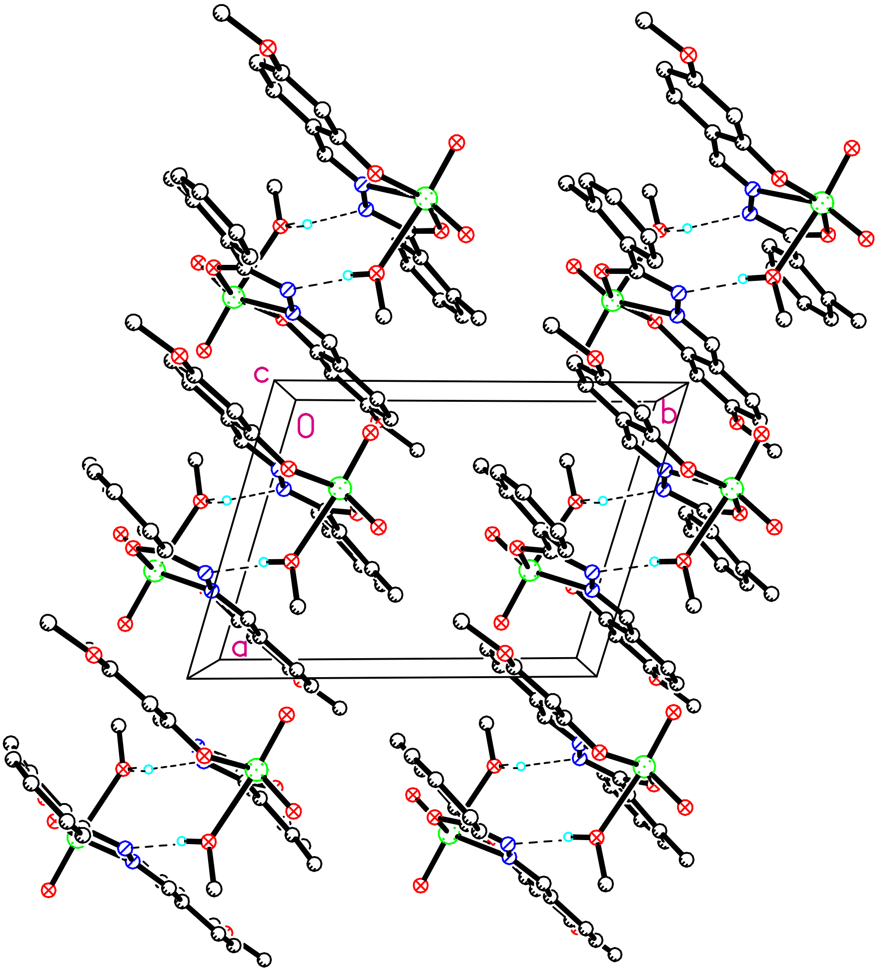


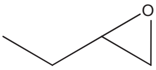
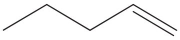
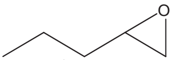
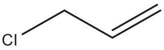
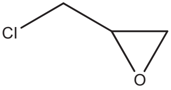
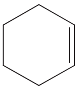
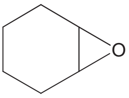
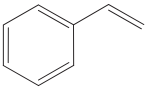
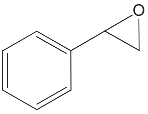
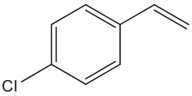
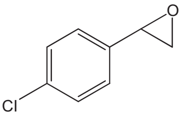
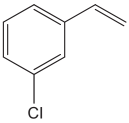
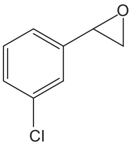
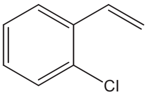
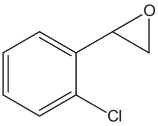
Figure 4. Molecular packing of **2** viewed along the *c* axis.

absence of the bands characteristic of N–H and C=O groups indicates enolization of the benzohydrazone ligands and coordination through the deprotonated enolic oxygen. The Mo=O stretch occurs as double bands at about 910 and 940 cm^{-1} for both complexes, assigned to the symmetric and asymmetric stretching modes of MoO_2 [30, 31]. The strong bands indicative of C=N groups of the complexes are located at 1607 cm^{-1} for **1** and 1605 cm^{-1} for **2** [32]. The weak peaks observed at low wavenumbers (400–600 cm^{-1}) may be attributed to Mo–O and Mo–N vibrations [31].

3.4. *Catalytic oxidation results*

The catalytic results are listed in table 3. Generally, excellent epoxide yields and selectivities were observed for all aliphatic and aromatic substrates. Oxidation of aromatic substrates gave the corresponding epoxides in 100% yields, while in the oxidation of aliphatic

Table 3. Details of the catalytic oxidation of olefins catalyzed by **1** and **2**.[a]

Substrate	Product		Conversion (%)[b]	Selectivity (%)
		**1**	93.3	100
		2	88.7	100
		**1**	91.2	100
		2	86.0	100
		**1**	94.8	100
		2	91.9	100
		**1**	100	100
		2	100	100
		**1**	100	100
		2	100	100
		**1**	100	100
		2	100	100
		**1**	100	100
		2	100	100
		**1**	100	100
		2	100	100

[a]The molar ratio of catalyst : substrate : TBHP is 1 : 300 : 1000. The reactions were performed in mixture of CH_3OH/CH_2Cl_2 (V : V = 6 : 4; 1.5 mL).

[b]The GC conversion (%) was measured relative to the starting substrate after 1 h.

substrates, the conversion is less than 100%. Based on this consideration, the isolated double bonds are less reactive than the conjugated ones. The phenomenon is in good agreement with those reported previously [9, 33–35]. From a detailed comparison between the catalytic properties of **1** and **2**, it can be seen that the former has a little higher conversion than the latter for aliphatic substrates. However, for aromatic substrates, there is no obvious difference. In general, the complexes in the present work have more effective oxidation properties than manganese(III) complexes containing N,O-type bidentate oxazine ligands [36] and Schiff base ligands [37, 38], and copper(II) complexes with 16- and 17-membered diaza dioxa macrocyclic Schiff bases [39], and similar results were found for tungsten(VI) complex and molybdenum(VI) complexes with hydrazone or Schiff base ligands [40–42].

4. Conclusion

Two new structurally similar molydenum(VI) complexes with benzohydrazone ligands, *N*′-(2-hydroxy-5-nitrobenzylidene)-2-methylbenzohydrazide and *N*′-(2-hydroxy-4-methoxybenzylidene)-2-methylbenzohydrazide, have been prepared and structurally characterized by single-crystal X-ray structure determination and infrared spectra. The benzohydrazone ligands coordinate to Mo through the phenolate oxygen, imino nitrogen, and enolate oxygen with molybdenum having octahedral coordination. The complexes are effective catalysts for oxidation of various olefins.

Supplementary material

CCDC 966411 (**1**) and 966412 (**2**) contain the supplementary crystallographic data for the complex. These data can be obtained free of charge via http://www.ccdc.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (+44)1223-336-033; or E-mail: deposit@ccdc.cam.ac.uk.

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