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Synthesis, Characterization and Crystal Structures of Oxidovanadium(V) and Dioxidomolybdenum(VI) Complexes Derived from 2-Bromo-*N'*-(2-Hydroxy-3-Methoxybenzylidene)benzohydrazide with Catalytic Property

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Abstract—A hydrazone compound 2-bromo-*N'*-(2-hydroxy-3-methoxybenzylidene)benzohydrazide (H_2L) was prepared and characterized by IR, UV-Vis and NMR spectra. Based on the hydrazone compound, a new oxidovanadium(V) and a new dioxidomolybdenum(VI) complexes, [VO(L)(OEt)(EtOH)] (**I**) and $[MoO_2(L)(OH_2)]$ (**II**), were prepared and characterized by IR, UV-Vis, NMR spectra, and single crystal X-ray diffraction (CIF files nos. 1866755 (**I**) and 1866756 (**II**)). Complex **I** crystallizes as the monoclinic space group $P2_1/c$ with unit cell dimensions a = 12.8733(12), b = 13.5088(13), c = 11.9262(11) Å, β = 91.765(2)°, V = 2073.0(3) Å^3, Z = 4, R_1 = 0.0524, wR_2 = 0.1329, *GOOF* = 1.245. Complex **II** crystallizes as the monoclinic space group $P2_1/c$ with unit cell dimensions a = 8.1140(9), b = 16.2975(19), c = 13.4318(15) Å, β = 106.456(2)°, V = 1703.4(3) Å^3, Z = 4, R_1 = 0.0296, wR_2 = 0.0728, GOOF = 1.046. X-ray analysis indicates that the complexes are mononuclear species with the metal atoms in octahedral coordination. The complexes were studied for catalytic oxidation property on some olefins with *tert*-butyl hydrogen peroxide as oxidant.

Keywords: hydrazone, vanadium complex, molybdenum complex, crystal structure, catalytic property

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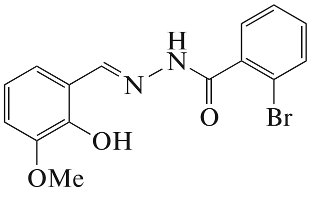
INTRODUCTION

In recent years, study on the oxidation of olefins has received particular attention in material chemistry. High-valent vanadium and molybdenum complexes have attracted continuing interest due to their important practical applications as catalysts in the ammoxidation of propene, the sulfoxidation and epoxidation of olefins, etc. [1–7]. The ability of vanadium and molybdenum to form stable complexes with oxygen- and nitrogen-containing ligands led to the development of vanadium and molybdenum Schiff base complexes which are efficient catalysts both in homogeneous and heterogeneous reactions [8–13]. Recently, we have reported some metal complexes with Schiff bases, and their biological aplications [14–16], as well as catalytic properties [17, 18]. In this paper, as a continuation of the work on the exploration of new catalytic materials based on vanadium and molybdenum complexes, a new oxidovanadium(V) and dioxidomolybdenum(VI) complexes, [VO(L)(OEt)(EtOH)] (**I**) and $[MoO_2(L)(OH_2)]$ (**II**), derived from hydrazone compound 2-bromo-*N'*-(2-hydroxy-3-methoxybenzylidene)benzohydrazide (H_2L) were prepared.



(H_2L)

EXPERIMENTAL

Materials and methods. $VO(Acac)_2$ and $MoO_2(Acac)_2$ and organic materials were purchased from Sigma-Aldrich and used as received. All other reagents were of analytical reagent grade. Elemental analyses of C, H and N were carried out in a Perkin-Elmer automated model 2400 Series II CHNS/O analyzer. FT-IR spectra were obtained on a Perkin-Elmer 377 FT-IR spectrometer with samples prepared as KBr pellets. UV-Vis spectra were obtained on a Lambda 900 spectrometer. X-ray diffraction was carried out on a Bruker APEX II CCD diffractometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker FT-NMR 300 MHz spectrometer using $(CD_3)_2SO$ as solvent. Gas chromatography (GC) analyses were performed on a Shimadzu GC-2010 gas chromatograph.

Synthesis of the hydrazone H_2L. The ethanolic solution (20 mL) of 3-methoxysalicylaldehyde (0.01 mol, 1.52 g) was added to the ethanolic solution (30 mL) of 2-bromobenzohydrazide (0.01 mol, 2.12 g) with stirring. The mixture was stirred for 30 min at room temperature and left to slowly evaporate to give colorless crystalline product, which were recrystallized from ethanol and dried in vacuum containing anhydrous $CaCl_2$.

For $C_{15}H_{13}N_2O_3Br$ (H_2L)

Anal. calcd., %	C, 51.60	H, 3.75	N, 8.02
Found, %	C, 51.45	H, 3.83	N, 8.13

The yield was 87%. IR (KBr; ν_{max}, cm^{-1}): 3443 w ν(OH), 3335 w ν(NH), 1651 s ν(C=O), 1602 w ν(C=N), 1521 m ν(Ar–O), 1288 m ν(C–O). UV-Vis data (EtOH; λ_{max}, nm): 221, 292, 333. ^{1}H NMR (300 MHz, d^6-DMSO; δ, ppm): 12.47 (s., 1H, O*H*), 11.18 (s., 1H, N*H*), 8.85 (s., 1H, C*H*=N), 8.73 (d., 1H, Ar*H*), 8.15 (d., 1H, Ar*H*), 8.07 (d., 1H, Ar*H*), 7.67 (m., 1H, Ar*H*), 7.08–7.03 (m., 2H, Ar*H*), 6.87 (t., 1H, Ar*H*), 3.82 (m., 3H, OC*H*$_3$).

Synthesis of complex I. The hydrazone compound H_2L (0.1 mmol, 34.8 mg) and $VO(Acac)_2$ (0.1 mmol, 26.5 mg) were mixed in ethanol (20 mL). The mixture was refluxed for 1 h and then cooled to room temperature. Single crystals of the complex, suitable for X-ray diffraction, were grown from the solution upon slowly evaporation within a few days. The crystals were isolated by filtration, washed with ethanol and dried in vacuum containing anhydrous $CaCl_2$.

For $C_{19}H_{22}N_2O_6BrV$

Anal. calcd., %	C, 45.17;	H, 4.39;	N, 5.54.
Found, %:	C, 44.96;	H, 4.51;	N, 5.63.

The yield was 45%. IR (KBr; ν_{max}, cm^{-1}): 3423 w ν(OH), 1607 s ν(C=N), 1446 m ν(C=C), 1349 m ν(Ar–O), 1095 m ν(C–O), 980 s ν(V=O). UV-Vis data (EtOH; λ_{max}, nm): 220, 306, 453. ^{1}H NMR (300 MHz, d^6-DMSO; δ, ppm): 8.86 (s., 1H, C*H*=N), 7.78–7.74 (m., 2H, Ar*H*), 7.49 (t, 1H, Ar*H*), 7.42 (t, 1H, Ar*H*), 7.30 (d, 1H, Ar*H*), 7.18 (d, 1H, Ar*H*), 6.92 (t, 1H, Ar*H*), 5.68–5.56 (q., 2H, C*H*$_2$), 3.85 (s., 3H, OC*H*$_3$), 3.47–3.43 (q., 2H, C*H*$_2$), 1.51 (t., 3H, C*H*$_3$), 1.06 (t., 3H, C*H*$_3$).

Synthesis of complex II. The hydrazone compound H_2L (0.1 mmol, 34.8 mg) and $MoO_2(Acac)_2$ (0.1 mmol, 32.6 mg) were mixed in ethanol (20 mL). The mixture was refluxed for 1 h and then cooled to room temperature. Single crystals of the complex, suitable for X-ray diffraction, were grown from the solution upon slowly evaporation within a few days. The crystals were isolated by filtration, washed with ethanol and dried in vacuum containing anhydrous $CaCl_2$.

For $C_{15}H_{13}N_2O_6BrMo$

Anal. calcd., %	C, 36.54	H, 2.66	N, 5.68
Found, %	C, 36.72	H, 2.77	N, 5.56

The yield was 63%. IR (KBr; ν_{max}, cm^{-1}): 3361, 3283 w ν(OH), 1598 s ν(C=N), 1446 m ν(C=C), 1355 s ν(Ar–O), 1158 s ν(C–O), 948 s, 862 s ν(Mo=O). UV-Vis data (EtOH; λ_{max}, nm): 220, 290. ^{1}H NMR (300 MHz, d^6-DMSO; δ, ppm): 8.91 (s., 1H, C*H*=N), 7.76–7.71 (m., 2H, Ar*H*), 7.51–7.43 (m., 2H, Ar*H*), 7.32–7.25 (q., 2H, Ar*H*), 7.03 (t., 1H, Ar*H*), 3.82 (s., 3H, OC*H*$_3$).

X-ray crystallography. X-ray diffraction was carried out at a Bruker APEX II CCD area diffractometer equipped with MoK_α radiation (λ = 0.71073 Å). The collected data were reduced with SAINT [19], and multi-scan absorption correction was performed using SADABS [20]. The structures of the complexes were solved by direct method, and refined against F^2 by full-matrix least-squares method using SHELXTL [21]. All of the non-hydrogen atoms were refined anisotropically. The ethanol hydrogen atom in **I** and the water hydrogen in **II** were located from electronic density maps and refined isotropically. The remaining hydrogen atoms were placed in calculated positions and constrained to ride on their parent atoms. The crystallographic data and refinement parameters for the compounds are listed in Table 1. Selected bond lengths and angles are listed in Table 2.

The supplementary crystallographic data for **I** and **II** have been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1866755 and 1866756, respectively; deposit@ccdc.cam.ac.uk or http://www.ccdc.ac.uk/conts/retrieving.html).

Catalytic oxidation experiment. Catalytic experiment was carried out in a 50 mL glass round-bottom flask fitted with a reflux condenser and placed in an oil bath at 353(2) K under continuous stirring. The catalytic oxidation experiment was carried out as follows: molybdenum(VI) complex (0.032 mmol) was dissolved in 10 mL 1,2-dichloroethane. Then 10 mmol alkene was added to the reaction mixture and 30 mmol TBHP (*tert*-butyl hydroperoxide) was added. The reaction mixture was refluxed for 1 h. The reaction products were monitored at periodic time intervals using gas chromatography. The oxidation products were identified by comparison with authentic samples (retention times in GC).

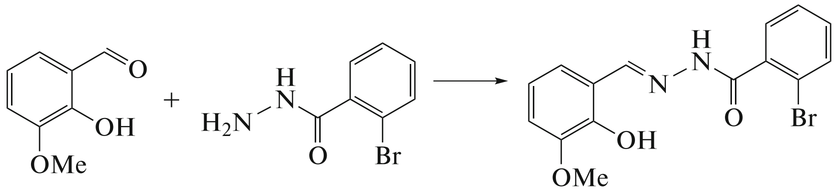
Table 1. Crystallographic data and structure refinement for **I** and **II**

Parameter	Value	
	I	**II**
Formula weight	505.24	493.12
Crystal shape/color	Block/yellow	Block/brown
T, K	298(2)	298(2)
Crystal dimensions, mm	0.20 × 0.18 × 0.18	0.31 × 0.27 × 0.23
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a, Å	12.8733(12)	8.1140(9)
b, Å	13.5088(13)	16.2975(19)
c, Å	11.9262(11)	13.4318(15)
β, deg	91.765(2)	106.456(2)
V, Å^3	2073.0(3)	1703.4(3)
Z	4	4
ρ_{calcd}, g cm^{-3}	1.619	1.923
$\mu(\mathrm{Mo}K_\alpha)$, mm^{-1}	2.444	3.150
$F(000)$	1024	968
Measured reflections	12100	9891
Unique reflections	3865	3167
Observed reflections ($I \geq 2\sigma(I)$)	2772	2603
Min and max transmission	0.6406 and 0.6674	0.4322 and 0.4834
Parameters	262	233
Restraints	3	3
Goodness of fit on F^2	1.245	1.046
R_1, wR_2 ($I \geq 2\sigma(I)$)*	0.0524, 0.1329	0.0296, 0.0728
R_1, wR_2 (all data)*	0.0763, 0.1450	0.0425, 0.0781

* $R_1 = F_o - F_c/F_o$, $wR_2 = [\Sigma\, w(F_o^2 - F_c^2)/\Sigma\, w(F_o^2)^2]^{1/2}$.

RESULTS AND DISCUSSION

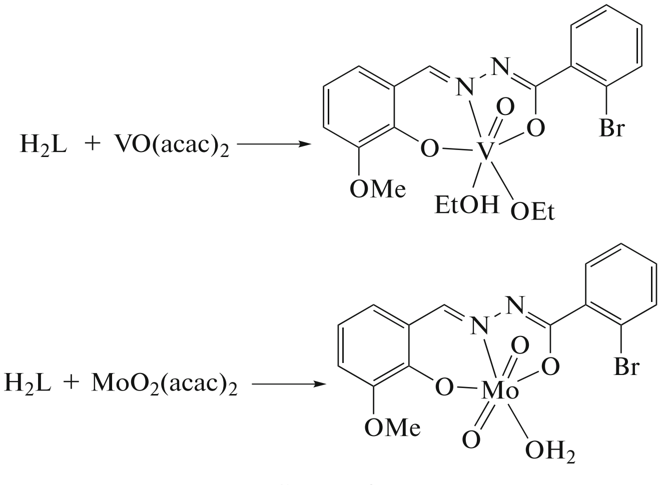
The hydrazone compound H_2L was readily prepared by the condensation reaction of a 1 : 1 molar ratio of 2-bromobenzohydrazide with 3-methoxysalicylaldehyde in ethanol (Scheme 1). Complexes **I** and **II** were prepared by the reaction of the hydrazone compound with $VO(Acac)_2$ and $MoO_2(Acac)_2$ in ethanol, respectively (Scheme 2), followed by recrystallization. Elemental analyses of the compounds are in accordance with the molecular structures proposed by the X-ray analysis.



Scheme 1.

Table 2. Selected bond distances (Å) and angles (deg) for complexes **I** and **II**

Bond	*d*, Å	Bond	*d*, Å
		I	
V(1)–O(1)	1.850(3)	V(1)–O(2)	1.949(3)
V(1)–O(3)	1.583(3)	V(1)–O(5)	1.772(3)
V(1)–O(4)	2.374(3)	V(1)–N(1)	2.128(3)
		II	
Mo(1)–O(1)	1.908(2)	Mo(1)–O(2)	2.017(2)
Mo(1)–O(4)	1.687(2)	Mo(1)–O(3)	1.711(2)
Mo(1)–O(5)	2.301(2)	Mo(1)–N(1)	2.236(3)
Angle	ω, deg	Angle	ω, deg
		I	
O(3)V(1)O(5)	102.21(17)	O(3)V(1)O(1)	99.75(17)
O(5)V(1)O(1)	103.56(14)	O(3)V(1)O(2)	102.19(16)
O(5)V(1)O(2)	93.86(14)	O(1)V(1)O(2)	148.30(14)
O(3)V(1)N(1)	91.93(16)	O(5)V(1)N(1)	163.08(16)
O(1)V(1)N(1)	82.83(13)	O(2)V(1)N(1)	73.88(13)
O(3)V(1)O(4)	175.51(16)	O(5)V(1)O(4)	81.62(14)
O(1)V(1)O(4)	76.92(12)	O(2)V(1)O(4)	79.74(12)
N(1)V(1)O(4)	84.68(13)		
		II	
O(4)Mo(1)O(3)	106.44(12)	O(4)Mo(1)O(1)	98.24(12)
O(3)Mo(1)O(1)	105.17(11)	O(4)Mo(1)O(2)	97.55(12)
O(3)Mo(1)O(2)	95.99(11)	O(1)Mo(1)O(2)	148.68(11)
O(4)Mo(1)N(1)	92.91(11)	O(3)Mo(1)N(1)	158.31(11)
O(1)Mo(1)N(1)	81.02(10)	O(2)Mo(1)N(1)	71.31(10)
O(5)Mo(1)O(4)	168.98(11)	O(3)Mo(1)O(5)	84.51(10)
O(1)Mo(1)O(5)	79.63(11)	O(2)Mo(1)O(5)	79.72(10)
N(1)Mo(1)O(5)	76.08(9)		



Scheme 2.

Table 3. Geometric parameters of hydrogen bonds for complexes **I** and **II***

D–H···A	Distance, Å			Angle D–H···A, deg
	D–H	H···A	D···A	
		I		
O(4)–H(4)···N(2)$^{\text{i}}$	0.85(1)	2.22(3)	2.958(4)	146(5)
		II		
O(5)–H(5B)···O(3)$^{\text{ii}}$	0.85(1)	1.92(2)	2.741(3)	165(4)
O(5)–H(5A)···N(2)$^{\text{iii}}$	0.85(1)	1.94(2)	2.774(4)	167(4)

* Symmetry codes: $^{\text{i}}$ x, $3/2 - y$, $-1/2 + z$; $^{\text{ii}}$ $1 - x$, $1 - y$, $1 - z$; $^{\text{iii}}$ $-x$, $1 - y$, $1 - z$.

In the spectra of the hydrazone compound and the complexes, the weak and broad bands in the region 3450–3280 cm^{-1} are assigned to the vibration of O–H bonds. The weak and sharp band located at 3335 cm^{-1} of the hydrazone compound is assigned to the vibration of N–H bond. The position of the bands demonstrates that the N–H hydrazone protons are engaged in hydrogen bonding. The intense bands at 1651 cm^{-1} of the hydrazone compound is generated by ν(C=O) vibration, whereas the band at 1602 cm^{-1} by the ν(C=N) one. The non-observation of the ν(C=O) bands, that present in the spectra of the hydrazone compound, indicates the enolization of the amide functionality upon coordination to the V- and Mo-centers. Instead strong bands at 1607 cm^{-1} for **I** and 1598 cm^{-1} for **II** are observed, which can be attributed to the asymmetric stretching vibration of the conjugated CH=N–N=C–O groups, characteristic for the coordination of the enolate form of the compounds. The bands at 980 cm^{-1} for **I** is assigned to ν(V=O) [22], and the bands at 948 and 862 cm^{-1} for **II** is assigned to ν(Mo=O) [23].

In the electronic spectra of the complexes, the lowest energy transition band is observed at 453 nm for **I**, while no such band for **II**, which is attributed to LMCT transition as charge transfer from *p*-orbital on the lone-pair of ligands' oxygen atoms to the empty *d*-orbital of the vanadium atom. The other strong bands in the range of 280–320 nm and at about 220 nm in the spectra of both complexes are similar to the absorption band in the spectrum of the hydrazone compound, so they are attributed to the intra-ligand $\pi \rightarrow \pi^*$ absorption peak of the ligands.

Molecular structures of complexes **I** and **II** are shown in Fig. 1. In both complexes, the coordination geometry around the metal atoms can be described as distorted octahedral with the tridentate hydrazone ligands coordinated in a meridional fashion, forming five- and six-membered chelate rings with bite angles of 73.88(13)° and 82.83(13)° (**I**), and 71.31(10)° and 81.02(10)° (**II**), respectively, typical for this type of ligand systems [24]. In complex **I**, one ethanolato ligand lies *trans* to the hydrazone imino N atom. One ethanol O atom *trans* to the oxo group completes the distorted octahedral coordination sphere at a rather elongated distance of 2.374(3) Å due to the *trans* influence of the oxo group. In complex **II**, one oxo group (O(3)) lies *trans* to the hydrazone imino N atom. One water O atom *trans* to the other oxo group (O(4)) completes the distorted octahedral coordination sphere at a rather elongated distance of 2.301(2) Å due to the *trans* influence of the oxo group. As expected, the hydrazone compounds in both complexes coordinate in their doubly deprotonated enolate form which is consistent with the observed O(2)–C(9) and N(2)–C(9) bond lengths of 1.298(5) and 1.296(5) Å in **I**, and 1.300(4) and 1.310(4) Å in **II**, respectively. This agrees with reported vanadium or molybdenum complexes containing the enolate form of this ligand type [25–28]. The dihedral angles between the two benzene rings of the hydrazone ligands are 73.6(4)° for **I** and 7.6(5)° for **II**.

In the crystal packing structure of complex **I**, the complex molecules are linked by ethanol molecules through intermolecular hydrogen bonds of O–H···N (Table 3), leading to the formation of one-dimensional chains along the *z*-axis direction (Fig. 2a). In the crystal packing structure of complex **II**, the complex molecules are linked by water molecules through intermolecular hydrogen bonds of O–H···O and O–H···N (Table 3), leading to the formation of dimers (Fig. 2b).

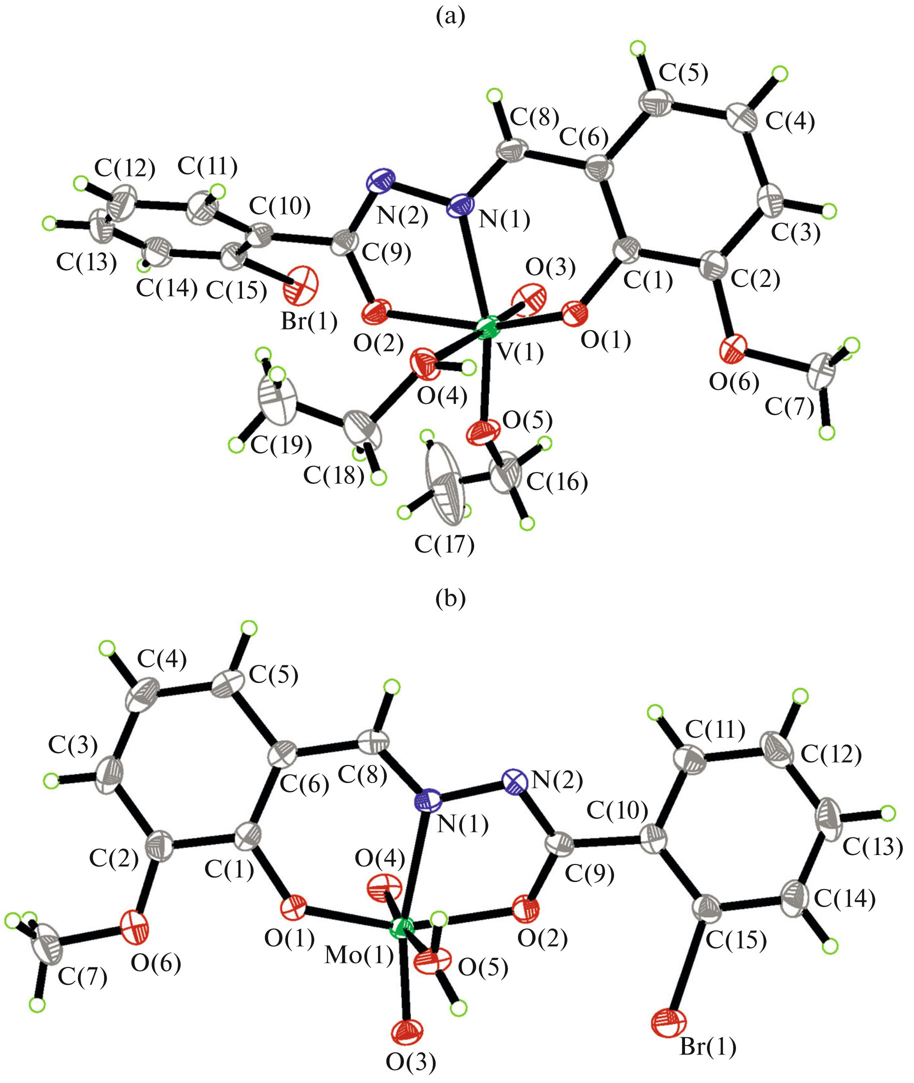
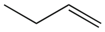
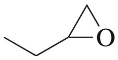
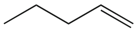
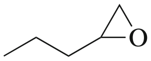
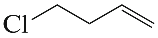
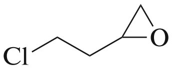
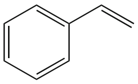
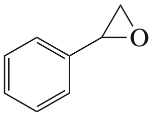
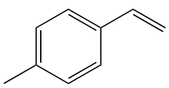
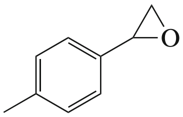
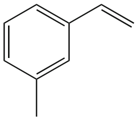
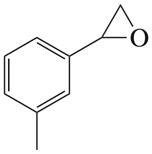
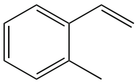
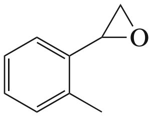
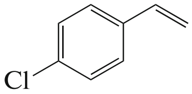
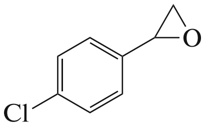
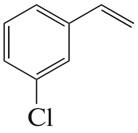
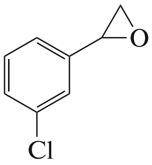
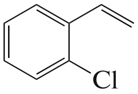
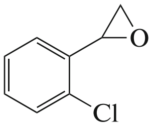


Fig. 1. Perspective views of complexes **I** (a) and **II** (b) with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.

1-Butene, 1-pentene, 4-chloro-1-butene, phenylene 4-methylphenylene, 3-methylphenylene, 2-methylphenylene, 4-chlorophenylene, 3-chlorophenylene, and 2-chlorophenylene were used as the substrates. The catalytic oxidation results are given in Table 4. The conversion for both aliphatic and aromatic substrates are 100% for either the vanadium complex or the molybdenum complex. High epoxide yields (82–90%) are obtained for aliphatic substrates for the molybdenum complex, while somewhat lower yields (73–83%) for the vanadium complex. As for the aromatic substrates, the epoxide yields (90–100%) are higher than the aliphatic substrates for both complexes. The catalytic oxidation property of the molybdenum complex is comparable to those of the dioxidomolybdenum(VI) complexes with phloroglucinol and resorcinol derived ligands [29] and with hydrazone ligands [30, 31]. The catalytic oxidation property of the vanadium complex is also similar to those of the oxidovanadium(V) complexes with hydrazone ligands [32].

In summary, a new oxidovanadium(V) complex and a new dioxidomolybdenum(VI) complex were obtained from the hydrazone ligand 2-bromo-*N*′-(2-hydroxy-3-methoxybenzylidene)benzohydrazide. The V and Mo atoms in the complexes are in octahedral coordination. The complexes have effective catalytic properties on oxidation of various olefins with *tert*-butyl hydrogen peroxide as the oxidant.

Table 4. Details of the catalytic oxidation of olefins catalyzed by the complexes*

Substrate	Product	Yields, %**	
		I	**II**
		77.5	82.1
		73.2	84.3
		82.5	89.2
		95.8	92.6
		95.0	94.5
		93.1	95.3
		90.9	93.7
		100	100
		100	100
		100	100

* The molar ratio of catalyst : substrate : TBHP is 1 : 300 : 1000. The reaction was performed in mixture of $CH_3OH–CH_2Cl_2$ (v : v = 3 : 1).
** The yields (%) were measured relative to the starting substrate after 1 h.

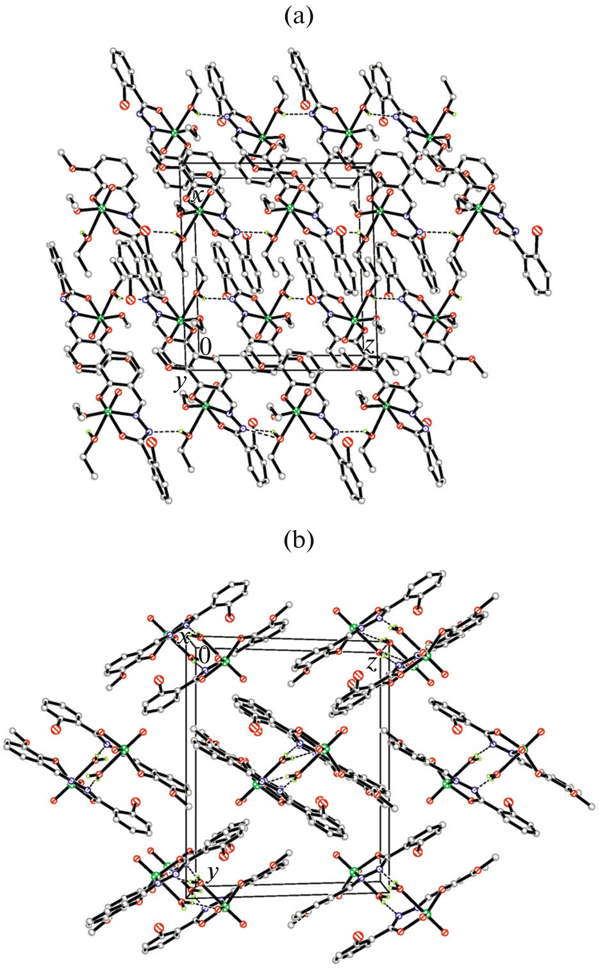


Fig. 2. Molecular packing structures of complex **I** (a) and **II** (b) with hydrogen bonds shown as dotted lines.

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