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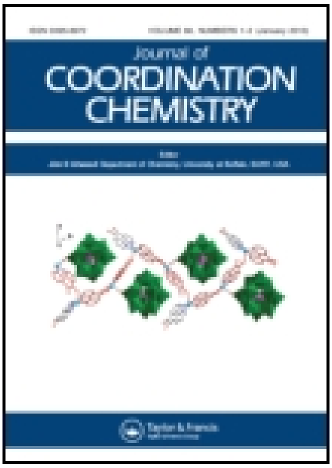

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# Syntheses, crystal structures, and catalysis by polymeric dioxomolybdenum(VI) complexes with similar (iso)nicotinohydrazones

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# Syntheses, crystal structures, and catalysis by polymeric dioxomolybdenum(VI) complexes with similar (iso)nicotinohydrazones

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Polymeric dioxomolybdenum(VI) complexes, $[MoO_2L]_n$ ($L = L^1 = N'$-(2-hydroxybenzylidene) nicotinohydrazide for **1**, $L = L^2 = N'$-(2-hydroxy-5-methoxybenzylidene)isonicotinohydrazide for **2**), were prepared and characterized by physico-chemical, spectroscopic methods, and single-crystal X-ray determination. Complex **1** crystallizes in the orthorhombic space group $P2_12_12_1$ with $a = 7.6568(4)$, $b = 11.6315(7)$, $c = 15.2211(9)$ Å, $V = 1355.59(13)$ Å$^3$, $Z = 4$, $R_1 = 0.0181$, $wR_2 = 0.0464$, and $S = 1.097$. Complex **2** crystallizes in the monoclinic space group $P2_1/n$ with $a = 11.2516(6)$, $b = 11.8134(7)$, $c = 12.4371(6)$ Å, $\beta = 116.2210(10)°$, $V = 1483.02(14)$ Å$^3$, $Z = 4$, $R_1 = 0.0399$, $wR_2 = 0.0874$, and $S = 1.096$. X-ray analysis indicates that Mo in each complex is octahedral with two oxo groups and $N_2O_2$ donor set of the nicotinohydrazone or isonicotinohydrazone. The complexes are efficient catalysts for oxidation of olefins.

*Keywords*: Hydrazone; Schiff base; Molybdenum complex; Crystal structure; Catalytic property

## 1. Introduction

The mechanism of molybdenum oxotransferase has been investigated. Molybdenum complexes are used in organic chemistry, in particular, for oxidations of various compounds [1–5]. Dioxomolybdenum complexes have been investigated as oxidation catalysts, particularly for sulfoxidation and epoxidation of olefins [6–9]. The synthesis, characterization, and reactivity studies of a number of dioxomolybdenum complexes with Schiff bases have been reported [10–13]. Some of the complexes possess oxygen transfer properties, oxidizing thiols, hydrazine, polyketones, and tertiary phosphines [14, 15]. The catalytic ability of dioxomolybdenum(VI) complexes with benzohydrazones toward the oxidation of sulfides has received satisfactory results [16, 17]. However, the number of documented dioxomolybdenum(VI) complexes catalyzing the peroxidic oxidation of sulfides is still very limited. There are no reports on the catalytic properties of dioxomolybdenum complexes with nicotinohydrazone or isonicotinohydrazone ligands. The rare reports on such complexes focus on preparation and structural characterization [18–21]. In the present paper, two dioxomolybdenum(VI) complexes, $[MoO_2L^1]_n$ (**1**) and

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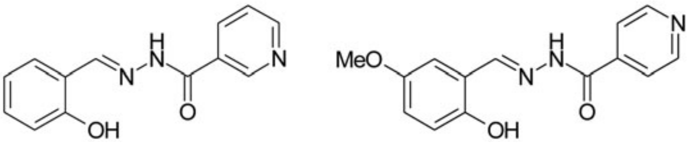


Scheme 1. Ligands $L^1$ (left) and $L^2$ (right).

$[MoO_2L^2]_n$ (**2**) (scheme 1; $L^1 = N'$-(2-hydroxybenzylidene)nicotinohydrazide for **1**, $L^2 = N'$-(2-hydroxy-5-methoxybenzylidene)isonicotinohydrazide for **2**), were prepared and studied for structures and catalytic properties.

## 2. Experimental

### 2.1. *Materials and methods*

All chemicals and solvents were of analytical reagent grade, purchased from Beijing Chemical Reagent Company. $L^1$ and $L^2$ 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 the JASCO FT-IR model 420 spectrophotometer with KBr disks from 4000 to 200 $cm^{-1}$. GC analyses were performed on a Shimadzu GC-2014 gas chromatograph.

### 2.2. *Syntheses of the complexes*

**2.2.1. $[MoO_2L^1]_n$ (1).** A hot ethanol solution (15 mL) of $MoO_2(acac)_2$ (0.33 g, 1 mM) was added to a hot ethanol solution (15 mL) of $L^1$ (0.24 g, 1 mM). The mixture was refluxed for 30 min and then cooled to room temperature to give an orange solution. Single crystals suitable for X-ray diffraction were formed by slow evaporation of the solution containing the complex in air for a few days. Yield: 55%. Anal. Calcd for $C_{13}H_9MoN_3O_4$: C, 42.5; H, 2.5; N, 11.4. Found: C, 42.3; H, 2.6; N, 11.6%.

**2.2.2. $[MoO_2L^2]_n$ (2).** A hot ethanol solution (15 mL) of $MoO_2(acac)_2$ (0.33 g, 1 mM) was added to a hot ethanol solution (15 mL) of $L^2$ (0.27 g, 1 mM). The mixture was refluxed for 30 min and then cooled to room temperature to give an orange solution. Single crystals suitable for X-ray diffraction were formed by slow evaporation of the solution containing the complex in air for a few days. Yield: 62%. Anal. Calcd for $C_{14}H_{11}MoN_3O_5$: C, 42.3; H, 2.8; N, 10.6. Found: C, 42.4; H, 2.8; N, 10.5%.

### 2.3. *X-ray structure determination*

X-ray measurements were performed using a Bruker Smart 1000 CCD diffractometer with graphite-monochromated MoK$\alpha$ radiation ($\lambda = 0.71073$ Å) using the $\omega$-scan technique. Determinations of the Laue class, orientation matrix, and cell dimensions were performed according to the established procedures where Lorentz polarization and absorption

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

| | **1** | **2** |
|---|---|---|
| Chemical formula | $C_{13}H_9MoN_3O_4$ | $C_{14}H_{11}MoN_3O_5$ |
| Fw | 367.2 | 397.2 |
| Crystal shape/color | Block/orange | Block/orange |
| Crystal size (mm) | 0.21 × 0.18 × 0.18 | 0.23 × 0.20 × 0.20 |
| $T$ (K) | 298(2) | 298(2) |
| $\lambda$ (MoK$\alpha$) (Å) | 0.71073 | 0.71073 |
| Crystal system | Orthorhombic | Monoclinic |
| Space group | $P2_12_12_1$ | $P2_1/n$ |
| $a$ (Å) | 7.657(1) | 11.252(1) |
| $b$ (Å) | 11.632(1) | 11.813(1) |
| $c$ (Å) | 15.221(1) | 12.437(1) |
| $\beta$ (°) | 90 | 116.221(1) |
| $V$ (Å$^3$) | 1355.6(1) | 1483.0(1) |
| $Z$ | 4 | 4 |
| $\mu$ (MoK$\alpha$) (cm$^{-1}$) | 0.987 | 0.914 |
| $T$ (min) | 0.8196 | 0.8173 |
| $T$ (max) | 0.8424 | 0.8383 |
| $D_c$ (g cm$^{-3}$) | 1.799 | 1.779 |
| Reflections/parameters | 2513/191 | 2087/209 |
| Unique reflections | 2443 | 2717 |
| Goodness of fit on $F^2$ | 1.097 | 1.096 |
| $R_{int}$ | 0.0191 | 0.0485 |
| $R_1$ [$I \geqslant 2\sigma(I)$] | 0.0181 | 0.0399 |
| $wR_2$ [$I \geqslant 2\sigma(I)$] | 0.0464 | 0.0874 |
| $R_1$ (all data) | 0.0191 | 0.0625 |
| $wR_2$ (all data) | 0.0468 | 0.1006 |

corrections were applied. Absorption corrections were applied by fitting a pseudoellipsoid to the $\psi$-scan data of selected strong reflections over a wide range of $2\theta$ angles. The positions of nonhydrogen atoms were located with direct methods. Subsequently, Fourier syntheses were used to locate the remaining nonhydrogen atoms. All nonhydrogen atoms were refined anisotropically. 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 complex are summarized in table 1. Selected bond lengths and angles are given in table 2.

## 3. Results and discussion

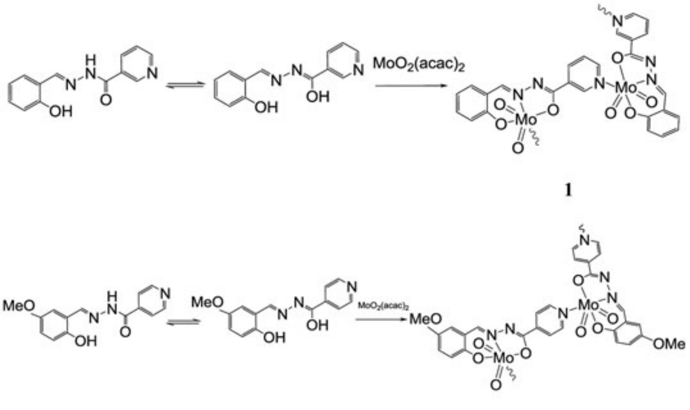
### 3.1. *Synthesis*

The hydrazones were readily prepared by condensation of salicylaldehyde with nicotinohydrazide, and 5-methoxysalicylaldehyde with isonicotinohydrazide, respectively, in ethanol. The dioxomolybdenum(VI) complexes were synthesized by refluxing hydrazones and $MoO_2(acac)_2$ in ethanol in a 1 : 1 M proportion. The reaction progress (scheme 2) is accompanied by a change of the solution from colorless to orange. We have attempted to prepare and grow diffraction quality crystals from various solvents; however, good quality crystals were finally obtained from ethanol. The chemical formulae of the complexes have been confirmed by elemental analyses, IR spectra, and X-ray single crystal structure determination.

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

| | | | |
|---|---|---|---|
| **1** | | | |
| Bond lengths | | | |
| Mo1–O1 | 1.927(2) | Mo1–O2 | 2.012(2) |
| Mo1–O3 | 1.691(2) | Mo1–O4 | 1.700(2) |
| Mo1–N1 | 2.234(2) | Mo1–N3A | 2.476(2) |
| C8–O2 | 1.319(3) | C8–N2 | 1.295(3) |
| N1–N2 | 1.392(3) | | |
| Bond angles | | | |
| O3–Mo1–O4 | 106.1(1) | O3–Mo1–O1 | 99.5(1) |
| O4–Mo1–O1 | 103.0(1) | O3–Mo1–O2 | 96.4(1) |
| O4–Mo1–O2 | 97.5(1) | O1–Mo1–O2 | 149.3(1) |
| O3–Mo1–N1 | 94.8(1) | O4–Mo1–N1 | 157.6(1) |
| O1–Mo1–N1 | 80.9(1) | O2–Mo1–N1 | 71.7(1) |
| O3–Mo1–N3A | 170.0(1) | O4–Mo1–N3A | 82.9(1) |
| O1–Mo1–N3A | 82.5(1) | O2–Mo1–N3A | 77.6(1) |
| N1–Mo1–N3A | 75.7(1) | | |
| **2** | | | |
| Bond lengths | | | |
| Mo1–O1 | 1.922(3) | Mo1–O2 | 2.021(3) |
| Mo1–O4 | 1.689(3) | Mo1–O5 | 1.687(3) |
| Mo1–N1 | 2.235(4) | Mo1–N3B | 2.455(4) |
| C9–O2 | 1.313(5) | C9–N2 | 1.297(5) |
| N1–N2 | 1.394(5) | | |
| Bond angles | | | |
| O5–Mo1–O4 | 105.7(2) | O5–Mo1–O1 | 100.9(2) |
| O4–Mo1–O1 | 103.9(2) | O5–Mo1–O2 | 96.7(2) |
| O4–Mo1–O2 | 96.0(2) | O1–Mo1–O2 | 148.5(1) |
| O5–Mo1–N1 | 93.0(2) | O4–Mo1–N1 | 159.1(2) |
| O1–Mo1–N1 | 81.1(1) | O2–Mo1–N1 | 72.1(1) |
| O5–Mo1–N3B | 170.7(2) | O4–Mo1–N3B | 82.2(2) |
| O1–Mo1–N3B | 81.6(1) | O2–Mo1–N3B | 77.2(1) |
| N1–Mo1–N3B | 78.4(1) | | |

Symmetry codes: A: $1/2+x$, $3/2-y$, $1-z$; B: $3/2-x$, $1/2+y$, $1/2-z$.



**1**

**2**

Scheme 2. The preparation of the complexes.

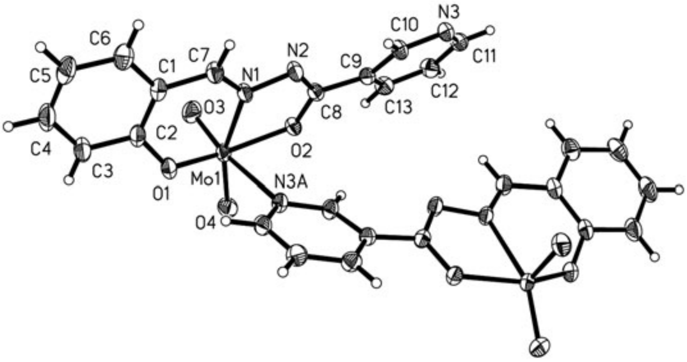


Figure 1. Molecular structure of **1** with 30% probability thermal ellipsoids. Atoms labeled with the suffix A and unlabeled are at the symmetry position $1/2+x$, $3/2-y$, $1-z$.

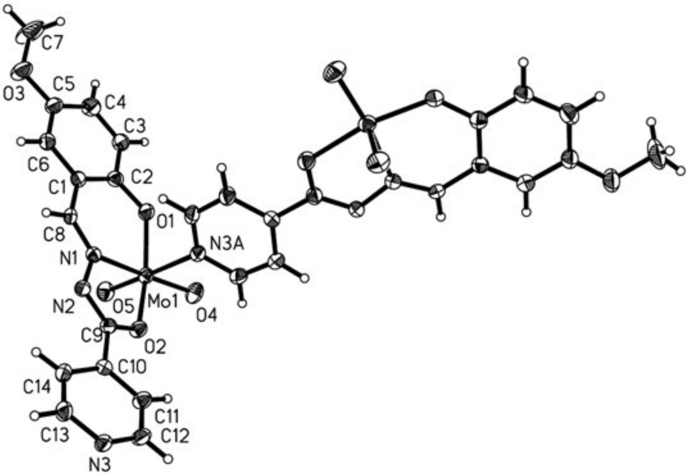


Figure 2. Molecular structure of **2** with 30% probability thermal ellipsoids. Atoms labeled with the suffix B and unlabeled are at the symmetry position $3/2-x$, $1/2+y$, $1/2-z$.

### 3.2. *Structure description of the complexes*

The asymmetric units of the structures of **1** and **2** are shown in figures 1 and 2, respectively. The coordination geometry around Mo can be described as slightly distorted octahedral, with phenolic O, imino N, and enolic O of the hydrazone, one oxo defining the equatorial plane, one N of a (iso)nicotinyl, and the other oxo occupying axial positions. The hydrazones coordinate to Mo meridional forming five- and six-membered chelate rings with bite angles of 71.71(6) and 80.90(7)° for **1,** and 72.07(12) and 81.09 (13)° for **2**. The dihedral angles between the benzene ring and the pyridine ring of the hydrazone are 32.9(3)° for **1** and 17.4(3)° for **2**. Displacements of Mo in both **1** and **2** from the equatorial mean planes toward the apical oxo are 0.33(1) Å. The hydrazones coordinate in their dianionic form, evident from N2–C8 and O2–C8 bond lengths in **1,** and N2–C9 and O2–C9 bond lengths in **2** (table 2). The abnormal bond values indicate the

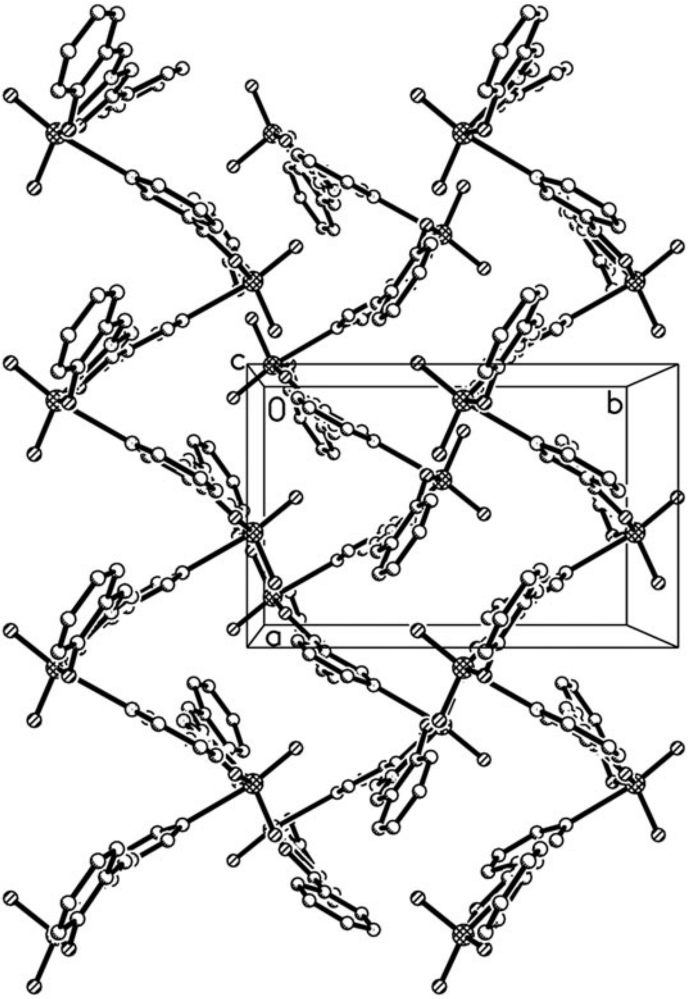


Figure 3. Molecular packing of **1** viewed along the *c* axis.

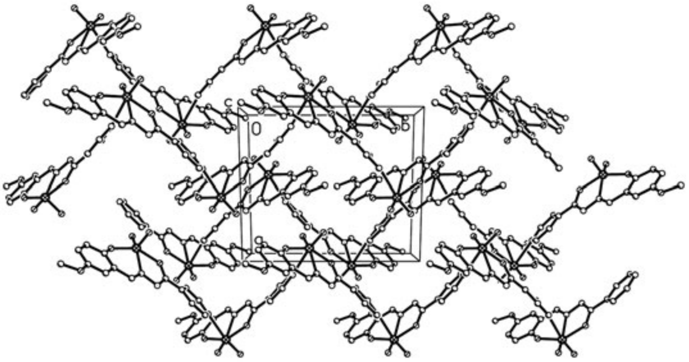
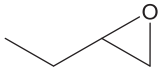
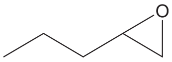
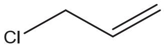
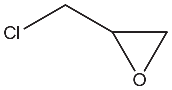
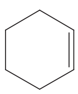
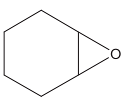
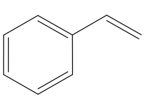
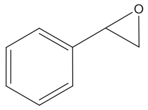
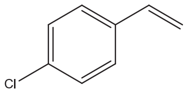
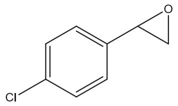
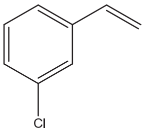
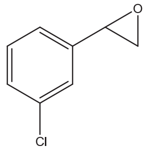
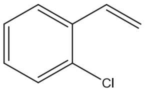
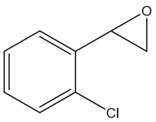


Figure 4. Molecular packing of **2** viewed along the *c* axis.

Table 3. Catalytic oxidation of olefins catalyzed by **1** and **2**.[a]

| Substrate | Product | | Conversion (%)[b] (TON)[c] | Selectivity (%) |
|---|---|---|---|---|
|  |  | **1** | 91 (263) | 100 |
| | | **2** | 90 (255) | 100 |
|  |  | **1** | 87 (271) | 100 |
| | | **2** | 85 (277) | 100 |
|  |  | **1** | 93 (268) | 100 |
| | | **2** | 93 (265) | 100 |
|  |  | **1** | 100 (289) | 100 |
| | | **2** | 100 (285) | 100 |
|  |  | **1** | 100 (281) | 100 |
| | | **2** | 100 (276) | 100 |
|  |  | **1** | 100 (292) | 100 |
| | | **2** | 100 (288) | 100 |
|  |  | **1** | 100 (280) | 100 |
| | | **2** | 100 (285) | 100 |
|  |  | **1** | 100 (272) | 100 |
| | | **2** | 100 (281) | 100 |

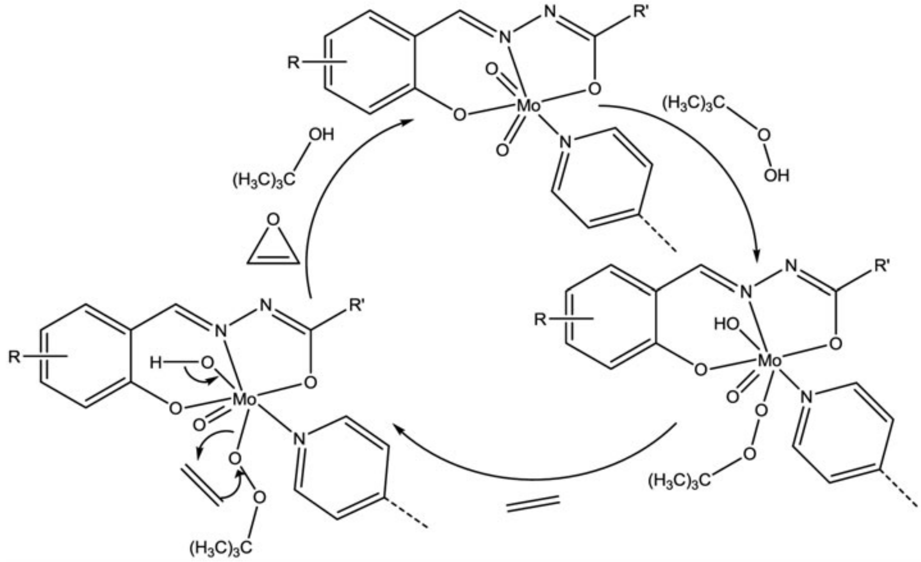
[a] The molar ratio of catalyst : substrate : TBHP is 1 : 300 : 1000. The reactions were performed in mixture of $CH_3OH/CH_2Cl_2$ ($V:V=6:4$; 1.5 mL).

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

[c] TON = (mM of product)/(mM of catalyst).

presence of the enolate form of the amide groups. The Mo–O, Mo–N, and Mo=O bonds are within normal ranges and similar to those observed in similar dioxomolybdenum(VI) complexes [18–21, 25–28]. The distances between Mo and nitrogen of the (iso)nicotinyl moieties, 2.476(2) Å in **1** and 2.455(4) Å in **2**, represent the largest bond lengths within the distorted octahedral coordination.

In crystal structures of the complexes, $[MoO_2L(CH_3OH)]$ units are linked via hydrazone ligands to form infinite one-dimensional zigzag chains running parallel to the *a* axis for **1** (figure 3) and *b* axis for **2** (figure 4).



Scheme 3. Proposed catalytic oxidation mechanism.

### 3.3. *IR spectra*

The hydrazones show stretches attributed to C=O, C=N, C–OH, and NH at 1654, 1615, 1180, and 3200 $cm^{-1}$, respectively [29]. For the complexes, absence of the bands characteristic of N–H and C=O indicates enolization of the hydrazone and coordination through the deprotonated enolic-oxygen. The Mo=O stretches occur as single and strong bands at 912 $cm^{-1}$ for **1** and **2**, assigned to the asymmetric stretch of $MoO_2$ [29, 30]. Strong bands indicative of C=N groups of the complexes are located at 1612 $cm^{-1}$ for **1** and 1605 $cm^{-1}$ for **2** [31]. Weak peaks observed at 460–600 $cm^{-1}$ may be attributed to Mo–O and Mo–N vibrations [30].

### 3.4. *Catalytic oxidation*

Catalysis was carried out according to the literature by using *tert*-butyl hydrogen peroxide (TBHP) as oxidant [9]. The 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 oxidation of aliphatic substrates, the conversion is less than 100%. Based on this, isolated double bonds are less reactive than conjugated ones, in agreement with those reported previously [9]. There are no distinct differences for catalytic properties between the two complexes. The proposed mechanism of the catalytic reactions is shown in scheme 3. First, TBHP was activated by coordination to Mo with formation of seven-coordinate molybdenum. Then a molecule of an olefin as a nucleophile attacks the electrophilic oxygen of the coordinated TBHP, forming the corresponding epoxides. TBHP was reduced to *tert*-butyl alcohol. The catalytic properties of the complexes reported here are superior to those of manganese complexes derived from hydrazone or tetraphenylporphyrin ligands [32, 33], and also superior to that of the tungsten complexes supported by multi-wall carbon nanotube [34].

## 4. Conclusion

A pair of polymeric dioxomolydenum(VI) complexes with similar (iso)nicotinohydrazone ligands, *N′*-(2-hydroxybenzylidene)nicotinohydrazide and *N′*-(2-hydroxy-5methoxybenzylidene)isonicotinohydrazide, have been prepared and structurally characterized by a single crystal X-ray structure determination and infrared spectroscopy. The hydrazones coordinate to Mo through phenolic O, imino N, ethanolic O, and pyridine N. Both complexes have catalytic properties on oxidation of various olefins to their corresponding epoxides.

## Supplementary material

CCDC 922464 (**1**) and 922465 (**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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