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Synthesis and Characterization of Molybdenum(V)-Oxo Complexes with ONO-Donors

Jung-Sook Kim, Hee-Jung Kim, and Bon-Kweon Koo*

Department of Chemistry Education, Hyosung Women's University, Kyungsan 713-702, Korea

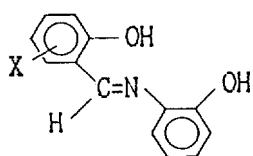
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Six-coordinate molybdenum(V)-oxo complexes $(\text{PyH})[\text{MoOCl}_2\text{L}]$ and $(\text{R}_4\text{N})[\text{MoOCl}_2\text{L}]$ ($\text{R}=\text{CH}_3$ and C_2H_5) with N-salicylidene-2-aminophenol(L^1) and its derivatives($\text{L}^2=5\text{-CH}_3$, $\text{L}^3=3\text{-CH}_3\text{O}$, $\text{L}^4=5,6\text{-C}_4\text{H}_4$ and $\text{L}^5=5\text{-NO}_2$) as ONO-donor ligands have been synthesized and the spectral and electrochemical properties of the complexes by elemental analysis, molar conductivity, UV-vis, IR, ^1H NMR and CV have been studied.

Introduction

The chemistry of molybdenum-oxo complexes with Schiff base ligands has received the intense attention because of its relevance to the active sites of molybdoenzymes, and many papers have been published in this area.¹

It is well-known² that the biological redox process of the molybdoenzymes involves oxidation states Mo(VI), Mo(V) and Mo(IV). In addition, extended X-ray absorption fine structure (EXAFS) spectroscopic studies have implicated the presence of oxygen and nitrogen atoms at the active sites of oxo-transfer molybdoenzymes.^{1a,3} In this context we have taken up N-salicylidene-2-aminophenol and its derivatives with ONO-donors as Schiff base ligands:



X	
H	H_2L^1
5- CH_3	H_2L^2
3- CH_3O	H_2L^3
5,6- C_4H_4	H_2L^4
5- NO_2	H_2L^5

These ligands are of particular interest because chelation may occur either from ON or ONO sites, in principle. It was also reported⁴ that functionalities present in the ligands play a vital role in controlling whether the molybdenum-oxo complexes are monomers or polymeric species, as well as the oxo-transfer ability. Thus, with the aim of simulating many characteristic properties for the active sites of molybdoenzymes, we have synthesized a series of dichlorooxomolybdenum(V) complexes $(\text{PyH})[\text{MoOCl}_2\text{L}]$ and $(\text{R}_4\text{N})[\text{MoOCl}_2\text{L}]$ ($\text{R}=\text{CH}_3$ and C_2H_5). In this paper, we report the synthesis, spectral properties and the electrochemical behaviors of the complexes.

Materials. Most chemicals used in synthesis were of reagent grade and used without further purification. Pyridinium pentachloromolybdate(V) $(\text{PyH})_2[\text{MoOCl}_5]$ ⁵ and 5-methylsalicylaldehyde⁶ were prepared by literature methods. All the Schiff base ($\text{H}_2\text{L}^1\text{-H}_2\text{L}^5$) were prepared by the same method as the previous paper and identified by IR and ^1H NMR.⁷ All solvents were dried by standard procedures and distilled before use.

Physical Measurements. Elemental analyses for C, H,

N were carried out by Kolon R and D center, and molybdenum was analyzed by using a Labtam ICP spectrometer (Model 8440). The M. P. measurements were performed by using a Hakke melting point apparatus. The molar conductivities of the complexes were measured by a YSI Conductivity bridge (Model 31). The IR spectra of solid samples in KBr were recorded on a Mattson Polaris FT-IR. The ^1H MMR spectra in $\text{DMSO}-d_6$ were recorded on a Varian FT NMR 300 spectrometer and referenced to TMS (internal). Electronic spectra were obtained on a Shimadzu UV-160A UV-visible spectrophotometer. Cyclic voltammograms were recorded on an Electrochemical Research System 270/6/0 (EG & G) consisting of PAR 263 Potentiostat/Galvanostat and an Electrochemical Analysis Software 270. The electrochemical studies were conducted in oxygen-free DMSO solution containing 0.05 M TBAP (tetrabutylammoniumperchlorate) as supporting electrolyte. We employed a three-electrode cell configuration consisting of a gold working, a platinum counter and a saturated calomel reference electrode.

Preparation of the Complexes. $(\text{PyH})[\text{MoOCl}_2\text{L}]$ ($\text{L}=\text{L}^1\text{-L}^5$). The same procedure was followed for the preparations of all the complexes. Pyridinium pentachloromolybdate(V) (10 mmol) in methanol (40 mL) was added to a suspension of an appropriate Schiff base (10 mmol) in methanol (50 mL) with stirring at 50°C . After complete dissolution of the Schiff base into the solution, pyridine (20 mmol) in 10 mL of methanol was slowly added to the solution to immediately give a precipitate of the desired compound. After the mixture was allowed to cool to 25°C , lustrous black crystals were collected by filtration, washed with a small amount of methanol and diethyl ether, and dried in a vacuum oven.

Analytical data for the complexes prepared are as follows; **$(\text{PyH})[\text{MoOCl}_2\text{L}^1]$ (1)** Yield: 67%. mp 220°C . Anal. Calcd for $\text{C}_{18}\text{H}_{15}\text{N}_2\text{O}_3\text{Cl}_2\text{Mo}$: C, 45.59; H, 3.19; N, 5.90; Mo, 20.23; Found: C, 45.60; H, 3.21; N, 5.69; Mo, 19.98. **$(\text{PyH})[\text{MoOCl}_2\text{L}^2]$ (2)** Yield: 70%. mp $229\text{-}231^\circ\text{C}$. Anal. Calcd for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_3\text{Cl}_2\text{Mo}$: C, 46.74; H, 3.51; N, 5.74; Mo, 19.65; Found: C, 46.44; H, 3.53; N, 5.58; Mo, 19.41. **$(\text{PyH})[\text{MoOCl}_2\text{L}^3]$ (3)** Yield: 75%. mp $192\text{-}193^\circ\text{C}$. Anal. Calcd for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_4\text{Cl}_2\text{Mo}$: C, 45.26; H, 3.40; N, 5.56; Mo, 19.02; Found: C, 45.31; H, 3.49; N, 5.60; Mo, 18.91. **$(\text{PyH})[\text{MoOCl}_2\text{L}^4]$ (4)** Yield: 71%. mp It does not melt up to 350°C . Anal. Calcd for $\text{C}_{22}\text{H}_{17}\text{N}_2\text{O}_3\text{Cl}_2\text{Mo}$: C, 50.40; H, 3.27; N, 5.34; Mo, 18.30; Found: C, 48.93; H, 3.26; N, 5.40; Mo, 18.57. **$(\text{PyH})[\text{MoOCl}_2\text{L}^5]$ (5)** Yield: 56%. mp $256\text{-}258^\circ\text{C}$. Anal. Calcd for $\text{C}_{18}\text{H}_{14}\text{N}_3\text{O}_5\text{Cl}_2\text{Mo}$: C, 41.64; H, 2.72; N, 8.09; Mo, 18.48; Found: C, 41.56; H, 2.75; N, 8.05; Mo, 18.62.

$(\text{Et}_4\text{N})[\text{MoOCl}_2\text{L}^3]$ ($\text{R}=\text{CH}_3$, C_2H_5). $(\text{PyH})[\text{MoOCl}_2\text{L}^3]$ (5 mmol) synthesized by following the procedure as mentioned above was dissolved in hot methanol (150 mL). To this solution was added an appropriate tetraalkylammonium salts (150 mmol) to immediately give a precipitate of the lustrous black crystals. The crystals were collected by filtration, washed well with ethanol, and dried in a vacuum oven.

Analytical data for the complexes prepared are as follows;

$(\text{Me}_4\text{N})[\text{MoOCl}_2\text{L}^3]$ (6) Yield: 62%. mp $219\text{-}220^\circ\text{C}$. Anal. Calcd for $\text{C}_{19}\text{H}_{23}\text{N}_2\text{O}_4\text{Cl}_2\text{Mo}$: C, 43.49; H, 4.65; N, 5.62; Found: C, 43.32; H, 4.63; N, 5.61; **$(\text{Et}_4\text{N})[\text{MoOCl}_2\text{L}^3]$ (7)** Yield: 65%. mp 212°C . Anal. Calcd for $\text{C}_{22}\text{H}_{31}\text{N}_2\text{O}_4\text{Cl}_2\text{Mo}$: C, 47.67;

Table 1. Infrared Spectral Data and Molar Conductivities for the complexes

Complex	IR*		Λ_M (mho $\text{cm}^2 \text{mol}^{-1}$)
	$\nu(\text{Mo}=\text{O})$	$\nu(\text{C}=\text{N})$	
$(\text{PyH})[\text{MoOCl}_2\text{L}^1]$ (1)	947	1600(1631)	53
$(\text{PyH})[\text{MoOCl}_2\text{L}^2]$ (2)	958	1615(1629)	54
$(\text{PyH})[\text{MoOCl}_2\text{L}^3]$ (3)	941	1598(1632)	50
$(\text{PyH})[\text{MoOCl}_2\text{L}^4]$ (4)	965	1596(1629)	53
$(\text{PyH})[\text{MoOCl}_2\text{L}^5]$ (5)	946	1596(1634)	58
$(\text{Me}_4\text{N})[\text{MoOCl}_2\text{L}^3]$ (6)	938	1599	53
$(\text{Et}_4\text{N})[\text{MoOCl}_2\text{L}^3]$ (7)	933	1606	52

*free ligand vibration in parentheses.

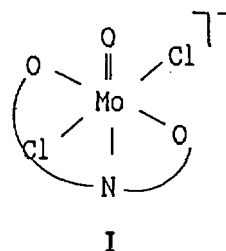
H, 5.64; N, 5.05; Found: C, 48.24; H, 5.62; N, 5.05

Results and Discussion

The molybdenum(V)-oxo complexes $(\text{PyH})[\text{MoOCl}_2\text{L}]$ (1-5) are synthesized by the reaction of $(\text{PyH})_2[\text{MoOCl}_5]$ with the appropriate ligand and the tetraalkylammonium salts of $[\text{MoOCl}_2\text{L}]^+$ (6, 7) are obtained from the methanol solutions of $(\text{PyH})[\text{MoOCl}_2\text{L}]$ in the presence of three-fold molar excess of the corresponding tetraalkylammonium chloride/bromide. All complexes are air stable in the solid state and have poor solubilities in the common organic solvents except DMF and DMSO.

Molar conductivities for the complexes determined at a concentration of $ca. 1 \times 10^{-3} \text{ mol dm}^{-3}$ DMSO fall in the range of 50 to 58 $\text{mho cm}^2 \text{mol}^{-1}$ (Table 1). This results clearly indicate that the complexes behave as 1 : 1 electrolytes in DMSO,⁸ consistent with the formula described below.

The complexes are formulated on the basis of the elemental analyses and a variety of physical measurements. A reasonable structural formulation is a six-coordinate octahedron with an axial $\text{Mo}=\text{O}$ group and the tridentate Schiff base ligand bound across axial and two equatorial sites (see below):



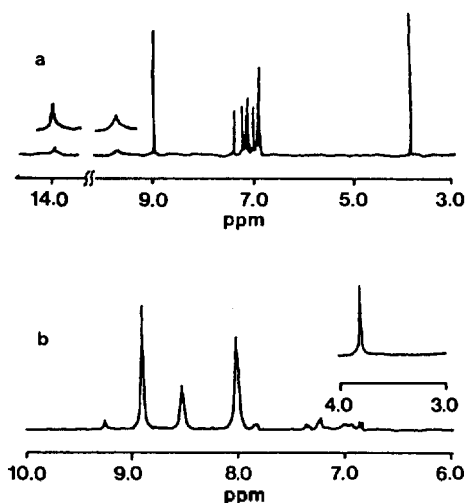
This geometry is found in the related six-coordinate complex $(\text{PyH})[\text{MoO}(\text{NCS})_2\text{L}]$, where L is the S-methyl 3-(2-Hydroxyphenyl)methylenedithiocarbamate ligand.⁹

The Schiff base ligand can act either as a monoanionic bidentate ON^{10} or as a dianionic tridentate ONO donor,⁷ depending on the conditions (eg. oxidation state of the metal ion). Infrared spectra of I exhibited no band at $ca. 2700 \text{ cm}^{-1}$ that would result from the νOH of the free ligands.¹⁰ The intense bands at $ca. 1630 \text{ cm}^{-1}$ associated with the $\text{C}=\text{N}$ stretching vibration of free ligands are shifted to $ca. 1600 \text{ cm}^{-1}$ in the complexes, indicating the coordination of the

Table 2. ^1H NMR Data^a for the Complexes in $\text{DMSO-}d_6$

Complex	ArH	N=CH ^b	Py	Others
1	6.83-7.84 (m, 8H)	9.28 (s, 1H) (8.95)	7.98 (t, 2H, <i>m</i> -) 8.49 (t, 1H, <i>p</i> -) 8.89 (d, 2H, <i>o</i> -)	
2	6.81-7.80 (m, 7H)	9.20 (s, 1H) (8.88)	7.70 (s, 2H, <i>m</i> -) 8.16 (d, 1H, <i>p</i> -) 8.74 (s, 2H, <i>o</i> -)	3.17 (s, 3H, 5-CH ₃)
3	6.83-7.83 (m, 7H)	9.26 (s, 1H) (8.94)	8.01 (s, 2H, <i>m</i> -) 8.53 (s, 1H, <i>p</i> -) 8.90 (s, 2H, <i>o</i> -)	3.81 (s, 3H, 3-CH ₃ O)
4	6.84-8.15 (m, 10H)	9.92 (s, 1H) (9.49)	7.93 (d, 2H, <i>m</i> -) 8.29 (s, 1H, <i>p</i> -) 8.71 (d, 2H, <i>o</i> -)	
5	6.83-7.70 (m, 7H)	9.43 (s, 1H)	7.96 (s, 2H, <i>m</i> -) 8.47 (s, 1H, <i>p</i> -) 8.88 (s, 2H, <i>o</i> -)	
6	6.82-8.27 (m, 7H)	9.24 (s, 1H)		3.12 (s, 12H, (CH ₃) ₄ N ⁺) 3.82 (s, 3H, 3-CH ₃ O)
7	6.83-8.29 (m, 7H)	9.25 (s, 1H)		1.18 (t, 12H (CH ₂ CH ₂) ₄ N ⁺) 3.22 (q, 8H, (CH ₂ CH ₂) ₄ N ⁺) 3.82 (s, 3H, 3-CH ₃ O)

^aChemical shifts are given in ppm vs Me₄Si. ^bfree ligand resonances in the lower parentheses.

**Figure 1.** ^1H NMR spectra of (a) H_2L^3 and (b) $(\text{PyH})[\text{MoOCl}_2\text{L}^3]$.

azomethine nitrogen to the metal ion.¹¹ These facts are also supported by the ^1H NMR data for the corresponding compounds. The OH proton resonance peaks of the two phenolic groups of free ligands in the range of 9.60-10.20 ppm and 13.33-15.65 ppm have disappeared upon complex formation, and the azomethine proton peaks of the ligands, which appeared as a sharp in the range of 8.88-9.49 ppm, are shifted to downfield (9.20-9.92 ppm) upon complexation.¹²

These observations confirmed that the ligands coordinate to the molybdenum atom through the charged two phenolic oxygen atoms and the nitrogen atom of the azomethine groups. The dianionic tridentate ligand can be bound by two modes, i.e., -facial and meridional-but the facial mode is sterically precluded by the ligand planarity.¹³

Infrared spectra for the complexes showed a single intense absorption peak at ca. 940 cm^{-1} (Table 1). This absorptions are assigned to the molybdenum-terminal oxygen vibration $\nu_{\text{Mo=O}}$, by comparison with the previously reported data.^{12,7,9} These are also accordance with the data ($\sim 940 \text{ cm}^{-1}$) for the complexes $\text{Mo}^{\text{V}}\text{OX}_2\text{L}_3$ (X=Cl, Br, I, L=tertiary phosphine) in which two X ions are *cis* to Mo=O group.

The ^1H NMR spectra of H_2L^3 and the complex (3) are shown in Figure 1. The relevant assignments of the spectra are summarized in Table 2, together with the data for the

Table 3. UV-Visible Bands for some Mo(V) Complexes in DMSO

Complex	$\lambda_{\text{max/nm}}$ (log ϵ , $\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$)		
	$\lambda_{\text{max/nm}}$	$\log \epsilon$	$\lambda_{\text{max/nm}}$
1	792 (1.67)	447 (sh)	306 (4.07)
2	707 (2.00)	425 (3.69)	306 (4.58)
3	756 (1.53)	440 (sh)	311 (4.02)
4	699 (2.12)	462 (3.90)	346 (4.24)
5	732 (1.99)	375 (sh)	314 (4.21)
6	824 (1.60)	440 (sh)	317 (4.18)
7	823 (1.69)	447 (sh)	317 (4.16)

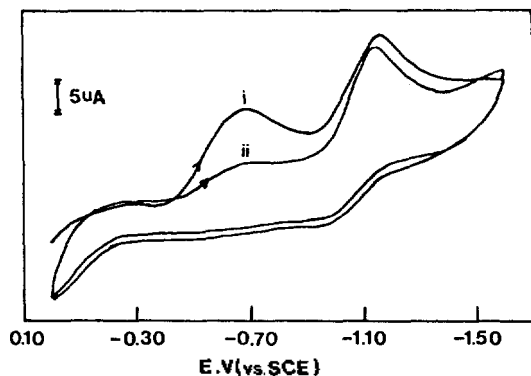
other complexes. The Schiff base ligand possesses two phenolic groups and their aromatic proton resonances occurred as multiplet in the range of 6.81-8.29 ppm.⁷ The aromatic protons of the pyridinium ion as a counter ion are easily distinguished from those of the Schiff base ligand by comparison of the spectra of the corresponding free ligands and those of the complexes containing Me₄N⁺ and Et₄N⁺ instead of PyH⁺ as counter ion. The *ortho*-, *meta*-, and *para*- position for the pyridinium ion of all the complexes except complex (4) appeared at ca. 8.82, 7.92, and 8.39 ppm with relative intensity ratio of 2 : 2 : 1.¹⁵ The methyl- and methylene protons for the tetraethylammonium ion of complex (7) appeared at 1.18 and 3.22 ppm with relative intensity of 3 : 2.¹⁶ The proton resonances for the substituents 5-CH₃ and 3-CH₃O on salicyl phenyl moiety of complexes (2) and (3) each occurred at 3.17 ppm and at 3.81 ppm as a singlet, respectively.⁷ However, the resonances for the protons of aromatic and 3-CH₃O group of ligand showed very weak peak intensities by comparison with PyH⁺ (Figure 1). This is ascribed to the paramagnetic complex due to Mo^V(d^1) state.

Electronic spectral data for the complexes are shown in Table 3, and showed three absorption bands in the region 300-900 nm. The absorption bands can be assigned on the basis of the energy levels reported by Sabat⁵ for tetragonal oxo-complexes of d^1 molybdenum ion (C_{4v}). One low energy band in the region 707-824 nm is assigned to the first crystal field transition $^2B_2 \rightarrow ^2E$ of the three spin-allowed $d-d$ transitions ($^2B_2 \rightarrow ^2E$, $^3B_2 \rightarrow ^2B_1$, and $^2B_2 \rightarrow ^2A_1$) as expected.⁵ The appearance of this low-energy band is the characteristic

Table 4. Cyclic Voltammetric Results^a for the Complexes in 0.05 M TBAP-DMSO at 25 °C

Complex	Epc ^b , V vs SCE	
1	-0.64	-1.10 (-1.00)
2	-0.60	-1.16 (-1.03)
3	-0.65	-1.15 (-1.01)
4	-0.70	-1.07 (-0.92)
5	-0.66	-0.98 (-0.79)
6	-0.61	-1.14 (-1.00)
7	-0.65	-1.16 (-1.00)

^a solute concentration $\sim 10^{-3}$ M; Working electrode gold; reference electrode SCE; Counter electrode platinum. ^b Values in parentheses are coupled oxidation peaks observed with complete CV cycle.

**Figure 2.** Cyclic voltammograms for -1×10^{-3} M (PyH)[MoOCl₂L³] in 0.05 M TBAP-DMSO (scan rate 50 mV s⁻¹): (i) first scan. (ii) second scan.

of Mo^v mononuclear oxo-complexes and essentially independent ligand.^{1a5} Other transitions are obscured by the intense charge transfer band.^{1a17} The weak absorption band found in the region 425-462 nm is probably due to L→M charge transfer transition.¹⁸ The very intense absorption band observed in the region 306-346 nm may be assigned to an intraligand transition, by comparison with the band of the free Schiff base ligand.

The electrochemical behavior of the monomeric molybdenum(V)-oxo complexes in DMSO/0.05 M TBAP has been studied by cyclic voltammetry at a gold working electrode, and the data in the potential range 0.0 to -1.5 V (vs SCE) are summarized in Table 4. The complexes displayed two successive cathodic responses near -0.64 V and -1.10 V. By comparison with those of the authentic one-electron reduction observed in the cases of other monomeric Mo(V)-oxo complexes,^{1a7} each of the reductions involves a metal-centered one-electron process and is due to the reduction of Mo(V)→Mo(IV) and Mo(IV)→Mo(III). The second reduction wave near -1.10 V is found to be coupled to a weak and broad anodic peak around -0.94 V. The cathodic peak near -0.64 V obtained from an initial negative scan is greatly reduced in intensity in the second cycle, which yields only the reductive response near -1.10 V (Figure 2). This indica-

tes that species reduced near -1.10 V is reoxidized near -0.94 V and this oxidized product is found to be reduced again near -1.10 V. The reduction potentials (Epc) are also sensitive to the nature of the substituents on the salicyl phenyl ring. As the substituents become more electron withdrawing (Me<H<NO₂), the Epc of the second reduction are shifted more anodic indicating easier reducibility of the respective molybdenum(IV) center.

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