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Molybdenum(VI)- and Tungsten(VI)- Dioxo Complexes with Schiff Base Ligands

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Oxo-transfer chemistry of molybdenum and tungsten is of topical interest. Molybdenum has a special place in this type of chemistry owing to the biological relevance of the model reaction (1) involving oxo-transfer molybdoenzymes.[1]

$$[MoO_2(L)_n] + X \rightleftarrows [MoO(L)_n] + XO \qquad (1)$$

The oxo-transfer ability of MoO_2^{2+} core depends on the functionalities present in the ligands. It is well-known that the active sites of oxo-transfer molybdoenzymes involve a sulfur, oxygen, and nitrogen atoms.[2] In relation to these studies, we have taken up N-salicylidene-2-amino-4-*t*-butylphenol and N-salicylidene-2-amino-2-ethyl-1,3-propanediol as a trifunctional (ONO) dianionic Schiff base ligands.

sabpH₂   sapdH₂
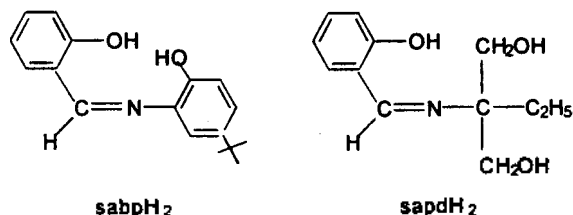
These ligands are important because their complexes with the MO_2^{2+} or MO^{2+} core could be of the form MO_2L or MOL (M=Mo, W), possessing one or two "open" coordination sites that can be utilized for substrate binding. It has been also reported that the phenyl substituents play an important role whether the isolated molybdenum(VI)-dioxo complexes are discrete monomers, $[MoO_2L(D)](D=MeOH$ or EtOH) or polymeric species, $[MoO_2L]_n$ containing $\cdots(O)$ $Mo=O \rightarrow Mo(O)=O \cdots$ chains in $[MoO_2(tridentate)]$ species.[3]

The electron-releasing substituent yield a polymer, whilst electron-withdrawing substituents favour the formation of monomers. In this relation study, the control effect of the ligands on the structures and chemical and spectroscopic properties of the molybdenum-oxo complexes studied here may help to identify the presence of different fragments or substituents attached to the active sites of relevant molybdoenzymes. In this work, we report some new monomeric or polymeric molybdenum(VI)-dioxo and tungsten(VI)-dioxo complexes of schiff base ligands with ONO donor system. Tungsten(VI)-dioxo complexes are very limited, because it is the non-availability of a suitable starting material. Spectroscopic properties and electrochemical behavior of the complexes have been discussed.

Experimental

Materials. Molybdenyl acetylacetonate, $[MoO_2(acac)_2]$ was obtained from Tokyo Kasei Chem. Ind. Co. Ltd. Tungstenyl acetylacetonate, $[WO_2(acac)_2]$ was prepared by literature method.[4] Salicylaldehyde, 2-amino-4-*t*-butylphenol, and 2-amino-2-ethyl-1,3-propanediol were obtained from Aldrich Chem. Co. Ltd. The schiff bases were prepared under Ar gas by the literature methods,[5] but was not isolated from methanol solution. All other chemical used for this work were of reagent grade and were employed without further purification. Reagent grade solvents were dried and distilled before use.

Physical measurements. C. H. N. for the complexes were analyzed by using a Carlo-Erba EA-1106R. The m.p.

measurements were performed by using a Haake melting point apparatus. The molar conductances of the complexes were measured by YSI-31 conductivity bridge. The IR spectra of solid samples in KBr were recorded on a Mattson Polaris FT-IR. The ^{1}H NMR spectra in DMSO-d_6 were recorded on a Bruker AM 200 spectrometer and referenced to TMS (internal). Electronic spectra were obtained on a Shimadzu UV-1601 UV/Vis spectrophotometer. Cyclic voltammograms were recorded on a Electrochemical Research System 270/6/0 (EG&G) consisting of PAR 263 Potentiostat/Galvanostat and Electrochemical analysis software 270. The electrochemical studies were conducted in oxygen-free DMSO solution containing 0.1 M tetrabutylammonium perchlorate(TBAP) as supporting electrolyte. We employed a three-electrode cell configuration consisting of a gold working, platinium counter, and a saturated calomel reference electrode.

Preparation of the complexes. [MoO$_2$(sabp) (MeOH)] (1a). One mmol (0.122 g) of salicylaldehyde was added to a methanol solution (10 mL) of 1 mmol (0.169 g) of 2-amino-4-t-butylphenol and refluxed for 1.5h, resulting in a yellow solution. After the solution was cooled to room temperature, 1 mmol (0.326 g) of MoO$_2$(acac)$_2$ in 20 mL of methanol was added with stirring. This reaction mixture was refluxed for 4h, after which the solution was allowed to stand for 24h in a refrigerator. The brown compound that separated out was filtered, washed throughly with cold methanol, diethylether, and dried in vacuum oven: Yield 0.214 g (50%). mp 171-173 °C: Anal. Calcd for C$_{18}$H$_{21}$NO$_5$Mo: C, 50.59; H, 4.95; N, 3.27. Found: C, 50.29; H, 4.88; N, 3.19. Λ$_M$(Mho cm^2 mol^{-1}): 1.1. UV/Vis. (nm, logε): 428 (3.63), 406 (3.61), 311 (4.24). Significant infrared bands (cm^{-1}): 909 (νMo=O, asym), 933 (νMo=O, sym), 1612 (νC=N). ^{1}H NMR (200 MHz. DMSO-d_6): δ 1.33 (s, 9H, -C(CH$_3$)$_3$), 3.17 (d, 3H, CH$_3$OH), 4.09 (q, 1H, CH$_3$OH), 6.73-7.82 (m, 7H, ArH), 9.32 (s, 1H, N=CH).

[MoO$_2$(sapd)(MeOH)] (1b). This was prepared similarly, by following the method described above for the prepartion of **1a**, with use of 2-amino-2-ethyl-1,3-propanediol instead of 2-amino-4-t-butylphenol. Yield 0.084 g (22%). mp 225 °C: Anal. Calcd for C$_{13}$H$_{19}$NO$_6$Mo: C, 40.96; H, 5.02; N, 3.67. Found: C, 40.49; H, 4.82; N, 3.69. Λ$_M$(Mho cm^2 mol^{-1}): 1.9. UV/Vis. (nm, logε): 347 (3.56), 313 (3.43). Significant infrared bands (cm^{-1}): 899 (νMo=O, asym), 924 (νMo=O, sym), 1632 (νC=N), ^{1}H NMR (200 MHz, DMSO-d_6): δ 0.84 (t, 3H, -CH$_2$CH$_3$), 1.87 (q, 2H, -CH$_2$CH$_3$), 3.17 (d, 3H, CH$_3$OH), 3.50, 3.58 (dd, 2H, -CH$_2$OH), 4.08 (q, 1H, CH$_3$OH), 4.29, 4.48 (dd, 2H, -CH$_2$O), 5.04 (t, 1H, -CH$_2$OH), 6.87-7.68 (m, 4H, ArH), 8.46 (s, 1H, N=CH).

[MoO$_2$(sabp)(Py)] (1c). The saturated solution of 0.5 mmol (0.214 g) of **1a** in pyridine was refluxed for 6h. The reaction solution was kept for 24h in refrigerator. The orange solid that seperated out was filtered, washed with diethylethyl, and dried in vacuum oven. Yield 0.166 g (70%). mp 290-293 °C: Anal. Calcd for C$_{22}$H$_{22}$N$_2$O$_4$Mo: C, 55.70; H, 4.67; N, 5.91. Found: C, 55.46; H, 4.80; N, 5.86. Λ$_M$(Mho cm^2 mol^{-1}): 1.1. UV/Vis. (nm, logε): 428 (3.68), 408 (3.65), 307 (4.29). Significant infrared bands (cm^{-1}): 903 (νMo=O, asym), 928 (νMo=O, sym), 1602 (νC=N). ^{1}H NMR (200 MHz. DMSO-d_6): δ 1.33 (s, 9H, -C(CH$_3$)$_3$), 6.73-8.58 (m, 12H, ArH), 9.31 (s, 1H, N=CH).

[MoO$_2$(sabp)(γ-Pic)] (1d) and [MoO$_2$(sabp) (DMSO)] (1e). The complexes were prepared by similar method. The solid was obtained by slow evaporation in air. Analytical data are as followings.

(1d): Yield 0.117 g (48%). mp 210-211 °C. Anal. Calcd for C$_{23}$H$_{24}$N$_2$O$_4$Mo: C, 56.56; H, 4.95; N, 5.74. Found: C, 56.21; H, 4.92; N, 6.02. Λ$_M$(Mho cm^2 mol^{-1}): 0.9. UV/Vis. (nm, logε): 428 (3.66), 408 (3.64), 311 (4.27). Significant infrared bands (cm^{-1}): 905 (νMo=O, asym), 929 (νMo=O, sym), 1601 (νC=N), ^{1}H NMR (200 MHz, DMSO-d_6): δ 1.32 (s, 9H, -C(CH$_3$)$_3$), 2.31 (s, 3H, CH$_3$-C$_5$H$_4$N), 6.73-8.43 (m, 11H, ArH), 9.32 (s, 1H, N=CH).

(1e): Yield 0.05 g (21%). mp 198-201 °C. Anal. Calcd for C$_{19}$H$_{23}$NO$_5$SMo: C, 48.21; H, 4.89; N, 2.96. Found: C, 48.21; H, 4.92; N, 3.02. Λ$_M$(Mho cm^2 mol^{-1}): 0.9. UV/Vis. (nm, logε): 425 (3.68), 411 (3.68), 311 (3.87). Significant infrared bands (cm^{-1}): 913 (νMo=O, asym), 932 (νMo=O, sym), 1613 (νC=N). ^{1}H NMR (200 MHz. DMSO-d_6): δ 1.33 (s, 9H, -C(CH$_3$)$_3$), 2.54 (s, 6H, (CH$_3$)$_2$SO), 6.74-7.82 (m, 7H, ArH), 9.33 (s, 1H, N=CH).

[WO$_2$(sabp)] (2a). 1 mmol (0.122 g) of salicylaldehyde was added to a methanol solution (10 mL) of 1 mmol (0.165 g) of 2-amino-4-t-butylphenol and refluxed for 1.5h, resulting in a yellow solution. After the solution was cool to room temperature, 1 mmol (0.416 g) of WO$_2$(acac)$_2$ in 10 mL of methanol was added with stirring. This reaction mixture was refluxed for 26h. The hot solution was filtered, the filterate was concentrated by rotary evaporator to 1/3 of initial volume. And then the solution was kept at refrigerator for overnight. The yellow solid was washed with diethylether, dried *in vacuum* oven. Yield 0.184 g (38%). mp 300 °C above. Anal. Calcd for C$_{17}$H$_{17}$NO$_4$W: C, 42.26; H, 3.55; N, 2.89. Found: C, 42.05; H, 3.67; N, 2.91. Λ$_M$ (Mho cm^2 mol^{-1}): 4.2. UV/Vis. (nm, logε): 413 (3.71), 345 (3.54), 284 (4.00). Significant infrared bands (cm^{-1}): 817, 893 (νW=O, asym), 952 (νW=O, sym), 1612 (νC=N). ^{1}H NMR (200 MHz. DMSO-d_6): δ 1.33 (s, 9H, -C(CH$_3$)$_3$), 6.79-7.85 (m, 7H, ArH), 9.33 (s, 1H, N=CH).

[WO$_2$(sapd)] (2b). This was prepared by the similar method to **2a** with use of 1 mmol (0.125 g) of 2-amino-2-ethyl-1,3-propandiol instead of 2-amino-4-t-butylphenol. Yield 0.175 g (40%). mp 297 °C Anal. Calcd for C$_{12}$H$_{15}$NO$_5$W: C, 32.97; H, 3.46; N, 3.20. Found: C, 32.84; H, 3.47; N, 3.39. Λ$_M$(Mho cm^2 mol^{-1}): 1.2. UV/Vis. (nm, logε): 347 (3.53), 300 (2.42). Significant infrared bands (cm^{-1}): 828, 903 (νW=O, asym), 948 (νW=O, sym). ^{1}H NMR (200 MHz. DMSO-d_6): δ 0.83 (t, 3H, -CH$_2$CH$_3$), 1.86 (q, 2H, -CH$_2$CH$_3$), 3.52, 3.57 (dd, 2H, -CH$_2$OH), 4.37, 4.73 (dd, 2H, -CH$_2$O), 5.08 (t, 1H, -CH$_2$OH), 6.93-7.71 (m, 4H, ArH), 8.44 (s, 1H, N=CH).

Results and Discussion

The molybdenum(VI) and tungsten(VI) complexes are synthesized by the reaction of MoO$_2$(acac)$_2$/WO$_2$(acac)$_2$ with each of the Schiff base ligands derived from salicylaldehyde and appropriate amines in methanol solution. The complexes of the type [MoO$_2$(sabp)(D)] could be synthesized by the substitution reaction of the type MoO$_2$(sabp)(MeOH) + D → MoO$_2$(sabp)(D), where D is different donor molecules such as pyridine, γ-picoline, and dimethylsulfoxide. All com-

Notes

Bull. Korean Chem. Soc. **1998**, Vol. 19, No. 5   589

plexes are air stable in the solid state and have poor solubilities in the common organic solvents, but they are highly soluble in DMF and DMSO.

Molar conductivities for the complexes determined at a concentration of $\sim 1 \times 10^{-3}$ mol dm^{-3} DMSO fall in the range of 0.9 to 4.2 mho cm^2 mol^{-1}.[6] These results clearly indicate that the complexes behave as non-electrolyte in DMSO, consistent with the formula described below. The complexes are formulated on the basis of the elemental analyses and a variety of physical measurements.

These geometries are found in the related six-coordinate complex [MoO$_2$L(γ-Pic)], where L is the salicylaldehyde thiosemicarbazone.[7]

I   II
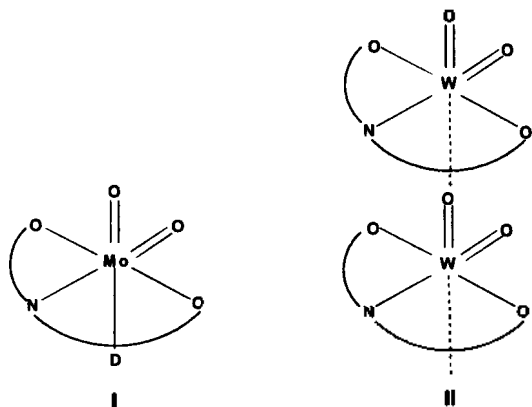
Infrared spectra for the molybdenum(VI) complexes except the type of [WO$_2$L] showed two intense absorption peaks at ~ 900 cm^{-1} and ~ 930 cm^{-1}. These bands are assigned to the molybdenum-oxygen terminal stretching vibration, $\nu(cis\text{-MoO}_2)$ by comparison with the spectra previously reported.[8] The $\nu_{sym}(\text{Mo}=\text{O}_t)$ for D binding in the complexes, **1a**, **1c**, **1d**, and **1e** increased in the order: py<γ-pic<DMSO<MeOH. This indicates that the order of D binding ability to metal is as following: MeOH<DMSO<γ-pic <py, because strong donor binding to molybdenum reduces the bond strength of *trans*-Mo=O$_t$ to D binding position.[1,8] The complexes of the type WO$_2$L exhibited a broad intense band at ~ 820 cm^{-1} with the strong bands at ~ 900 cm^{-1} and ~ 950 cm^{-1} due to $\nu(cis\text{-WO}_2)$. The appearance of this band indicates that the complexes are polymeric in nature, polymerization most probably taking place via W=O$\cdots$>W linkages(II) in the solid state.[7,9] When they are dissolved in coordinating solvents such as DMSO, MeOH, the oligomeric chain is probably broken and the solvent molecule is coordinated in the sixth coordination position, as is evident from the type of complex WO$_2$(sal-pho)(solvent)[10] (sal-pho=N-2-hydroxyphenylsalicylaldiminato). The molybdenum(VI)-dioxo complexes exhibited characteristic bands corresponding to the bond monodentate donor. The complexes **1a**, **1b** exhibited a broad bands at ~ 3400 cm^{-1} due to the presence of coordinated methanol molecule. The appearance of a medium intensity $\nu(\text{S}=\text{O})$ band at 1008 cm^{-1} of **1e** represents that DMSO is coordinated to MoO$_2$ group.[3] The existence of D binding to MoO$_2$ group is also supported from the results of ^{1}H NMR measurement. The ^{1}H NMR spectrum of **1c** showed proton resonances due to ortho-protons of pyridine at 8.58 ppm as doublet. The resonances for the protons of ortho-position and methyl group

of γ-picoline in complex **1d** appeared at 8.43 ppm and 2.31 ppm, respectively. The resonances for the ortho-protons of neutral donor molecule are easily distinguished from comparison the ^{1}H NMR spectra of **1c** and **1d** with the spectra of other complexes prepared in this work.

The Schiff base ligands can act either as a monoanionic bidentate ON[11] or as a dianionic tridentate ONO[12] donor depending on the conditions such as oxidation state of the metal, but all physicochemical properties of the complexes support dianionic tridentate ONO chelation of the ligands. The intense bands at ~ 1610 cm^{-1} associated with the C=N stretching frequency in the complexes of sabp ligand are shifted to low frequency by comparing with the frequency 1630 cm^{-1} of the free ligand, N-salicylidene-2-aminophenol reported previously.[12] The sapd ligand complexes showed intense bands at ~ 1630 cm^{-1}. ^{1}H NMR data for the azomethine proton resonances are also shifted to down field (~ 9.30 ppm) compared to the resonances (~ 8.30 ppm) of the free ligand, N-salicylidene-2-aminophenol and its derivatives reported previously.[13] The sapd complexes showed the strong azomethine proton resonances at ~ 8.45 ppm of up field, comparing to the sabp complexes deshielded by inductive effect. This is supported from the IR data for $\nu(\text{C}=\text{N})$. Also, the OH proton resonance of free schiff base ligand is not found on complexation, except the complexes with sapd ligand. These results confirm that the Schiff base ligand is bonded to the metal through the two phenolic oxygens and azomethine nitrogen as dibasic tridentate ligand. The ^{1}H NMR spectra of the complex **1a** showed the proton resonances for each of the phenolic aromatic, 4-butyl, methyl and OH group of methanol as neutral monodentate at 6.73-7.82, 1.33, 3.17, and 4.09 ppm. The ratio of integrated intensities for the proton resonances of Schiff base and methanol also showed 17:4 consistent with the stoichiometric ratio 1:1, where the integral ratio for the ArH:N=CH:*t*-Bu of sabp ligand showed 7:1:9 corresponding to stoichiometric ratio. However, the complex of **2a** showed similar ^{1}H NMR spectra to the complex **1a**, except disappearing the proton resonances for the methanol as neutral donor molecule.

The complexes showed similar electronic spectra, suggesting that they have essentially similar structure. Since molybdenum(VI) and tungsten(VI) ion have no d electron, absorption bands of pure d-d origins are not expected to appear. The bands at *ca.* 428 nm (logε=3.63-3.68) for molybdenum and at 413 nm (logε=3.71) for the tungsten com-

Table 1. Cyclic Voltammetric Results[a] for the Complexes at 20 °C

Complex	E_{pc}, V (*vs* SCE)
MoO$_2$(sabp)(MeOH) (**1a**)	-1.12, -1.68
MoO$_2$(sapd)(MeOH) (**1b**)	-1.00, -1.46
MoO$_2$(sabp)(Py) (**1c**)	-1.12, -1.68
MoO$_2$(sabp)(γ-Pic) (**1d**)	-1.13, -1.68
MoO$_2$(sabp)(DMSO) (**1e**)	-1.14, $\cdot 1.72$
WO$_2$(sabp) (**2a**)	-1.25, -1.64
WO$_2$(sapd) (**2b**)	-1.02, -1.58

[a] Solvent, DMSO; solute concentration, $\sim 1 \times 10^{-3}$ M; working electrode, gold; reference electrode, SCE; counter electrode, Pt; scan rate, 50 mVs^{-1}.
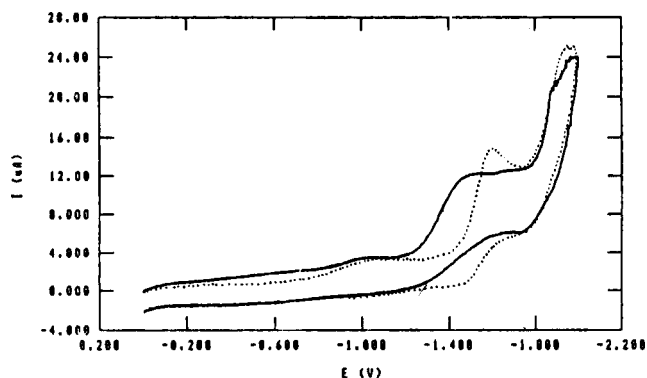
Figure 1. Cyclic voltammograms for ~1 × 10⁻³ M [MoO₂(Sapd) (MeOH)] (**1b**): — and [WO₂(Sapd)] (**2b**): ⋯ in 0.1 M TBAP-DMSO (scan rate 50 mVs⁻¹).

plexes may possibly be assigned as charge transfer transition from oxygen orbital to a metal d-orbital.[14,15]

The cyclic voltammetry of the molybdenum(VI)- and tungsten(VI)-dioxo complexes have been studied using a gold working electrode and NBu_4ClO_4 as supporting electrolyte in DMSO solvent. The complexes exhibit two successive reductive responses in the potential range 0.00 to $-2.00V$ (Table 1). The cyclic voltammograms for the representative complexes (**1b** and **2b**) are showen in Figure 1. On comparing the cyclic voltammograms of other similar monomeric Mo(VI)-and W(VI)-dioxo complexes,[15,16,17] it appears that the initial reduction wave at *ca.* $-1.12V$ is due to the reduction of Mo(VI) or W(VI) to Mo(V) or W(V) species and a Mo(V) or W(V) species generated electrochemically undergo a further reduction to generate a Mo (IV) or W(IV) species at *ca.* $-1.68V$. The E_{pc} of the W(VI)/W(V) couple is shifted in the cathodic direction compared to Mo(VI)/Mo(V) couple, which means the reduction of W (VI) complexes is more difficult than Mo(VI) species. This fact agrees with larger values of the stretching frequencies of WO_2 *vs.* MoO_2 group. It is known that the reduction potentials are sensitive to the nature of the substituents on the salicyl phenyl ring.[7] But, the reduction of the complexes with sabp ligand occurred at more negative potentials in comparison with that of sapd ligand complexes, which violate the anticipation from the electron donor ability of ligand. In addition, the cyclic voltammograms showed that the tendency of reduction to a variety of D binding molecules is inconsistent with that of stretching frequencies, v_{sym} (MoO_2).

Supporting Information Available. Supplementary materials for the IR and NMR data are available from B. K. Koo.

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