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Synthesis, electrochemical and spectroscopic properties of dimeric oxomolybdenum(IV) and (VI) complexes containing terminal and bridged bidentate α-benzoinoxime ligands

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Abstract

Reactions between $Mo(CO)_6$ and α-benzoinoxime carried out under various atmospheric conditions, of either vacuum or argon, have resulted in a dramatically different products. When the reaction has been carried out under vacuum, the binuclear oxomolybdenum(VI) complex $[Mo_2O_5(HBNO)_2]$ (**1**) was obtained. The complex was found to be diamagnetic at room temperature (r.t.). However, changing the reaction condition to be under an inert atmosphere of argon, but not under scrupulously oxygen-free conditions, produced an oxomolybdenum dimer $[Mo_2O_2(HBNO)_4]$ (**2**). The observed magnetic moment for the complex is consistent with the formulation of a dimer containing two Mo(IV) metal ions. In addition, the absence of a strong coupling between the two molybdenum metal ions excludes the presence of Mo–Mo or Mo–O–Mo bonding. The IR spectral analysis was informative in assigning the vibrations due to $Mo_2O_5^{2+}$ and $Mo_2O_2^{4+}$ moieties for complexes **1** and **2**, respectively. The electrochemical behavior of the two complexes was completely different. While complex (**1**) displayed two reversible one-electron reductions, complex (**2**) showed one irreversible two-electron oxidation followed by a one-electron oxidation. Moreover, the magnitude of the comproportionation constant, $K_c = 1.03 \times 10^9$, calculated for complex (**1**) indicates an extensive charge delocalization between the two molybdenum centers.

Keywords: Molybdenum complexes; Benzoinoxime; Dimer; Magnetic moment; Electrochemical properties

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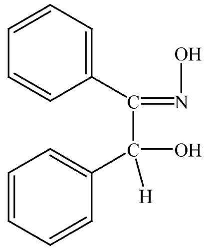
1. Introduction

The role of molybdenum in enzymes that catalyze a variety of net oxygen atom transfer reactions has stimulated interest in oxomolybdenum chemistry [1,2]. Much of the coordination chemistry of higher oxidation state molybdenum is found in classes of complexes containing terminal (Mo=O) and bridging (Mo–O–Mo) oxo ligands [3,4]. Complexes containing a single terminal oxo ligand are commonly found in the chemistry of Mo(IV) and Mo(V) but rarely found in the chemistry of Mo(VI) [5,6].

We have reported earlier that reaction between $Mo(CO)_6$ and ChryQ (chrysenequinone) gave five dimeric oxomolybdenum complexes [7]. Also, we reported the reaction between $Mo(CO)_6$ and dioxime ligand which results in a novel structure arrangement of molybdenum dimethylglyoxime complexes [8]. These have prompted us to investigate the reactions of



α-benzoinoxime ligand (H_2BNO)

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molybdenum hexacarbonyl with a monooxime ligand such as α-bezoinoxime.

In the present work, we describe the preparation and characterization of two new oxomolybdenum(VI) and (IV) dimeric complexes: $[Mo_2O_5(HBNO)_2]$ (**1**) and $[Mo_2O_4(HBNMO)_4]$ (**2**). The chemical formulas for both complexes were suggested based on their mass spectra and elemental analyses. Whereas, the coordination modes of the ligands to the molybdenum metal centers have been assigned by analyzing their IR and ^1H NMR spectra. Their magnetic properties were used to assign the oxidation states of the molybdenum metal ions. Also, the electrochemical behavior of the two complexes will be discussed in this paper.

2. Experimental

Molybdenum hexacarbonyl, ($Mo(CO)_6$, α-benzoinoxime (H_2BNO), tetrabutylammonium perchlorate were used as purchased from Aldrich chemical Co. Inc. Solvents were dried according to standard procedures. Solvents used for cyclic voltammetry were further purified by vacuum distillation and stored over molecular sieves: dimethylformamide (DMF) and dimethylsulfoxide (DMSO) from calcium hydride; acetonitirle from calcium chloride.

The magnetic susceptibility measurements were obtained with an Oxford Faraday magnetometer. Diamagnetic corrections were estimated from Pascal's constants to obtain the corrected molar paramagnetic susceptibilities of the compounds, χ_M^{corr} [9,10]. The magnetic moments were calculated by the equation $\mu_{eff} = 2.828 \times (\chi_M^{corr} \cdot T)^{1/2}$. Infrared spectra (4000–400 cm^{-1}) were recorded as KBr pellets on a Unicam Masttson 1000 FTIR spectrometer. The electronic absorption spectra were recorded by using a model 1601 Shimadzu spectrometer. Samples of $5–8 \times 10^{-4}$ M concentrations in DMSO were measured against the solvent in the reference cell. Elemental analyses were performed using a Perkin–Elmer 2400 CHN elemental analyzer. Mass Spectra were obtained on a Finnigan Mat SSQ 700 mass spectrometer. ^1H NMR spectra were carried out on a Spectrospin-Brucker AC 200 MHz NMR spectrometer. Cyclic voltammetery measurements were carried out using a model 303 HMDE in conjunction with a PAR model 173 potentiostat and PAR 175 universal programmer. A platinum disc was used as working electrode and a platinum wire as auxiliary electrode. The reference electrode was a saturated calomel electrode (SCE). Tetrabutylammonium perchlorate was used as the supporting electrolyte. Solution concentrations were approximately1.5×10^{-3} M in complex and 0.1 M in supporting electrolyte.

2.1. *Synthesis of μ-oxo-bis(μ-benzoinoximatodioxomolybdenum(VI)), $[Mo_2O_5(HBNO)_2]$ (1)*

$Mo(CO)_6$ (0.20 g, 0.76 mmol) and α-benzoinoxime (0.17 g, 0.75 mmol) dissolved in 50 ml of a 1:1 THF–hexane solution were mixed at room temperature (r.t.). The mixture was heated to reflux under reduced pressure for 2 h. The resultant dark brown solution was then rotary evaporated until precipitation was observed. The mixture was then cooled in an ice bath for 1 h. Brown microcrystals were collected by vacuum filtration and washed with cold THF. The complex was left to dry in vacuo for ~3 h to give a yield of 0.11 g (40%).

Anal. Calc. for $C_{28}H_{24}N_2O_9Mo_2$ (MW = 724.386): C, 46.43; H, 3.34; N, 3.87. Found: C, 46.32; H, 3.26; N, 3.70%. Selected mass spectra fragments, *m/z*: 725, 707, 690.6, 498.2, 271, 240.1.

2.2. *Synthesis of μ-bis(benzoinoximato)-bis(benzoinoximatooxomolybdenum(IV)), $[Mo_2O_2(HBNO)_4]$ (2)*

$Mo(CO)_6$ (0.22 g, 0.83 mmol) and α-benzoinoxime (0.36 g, 1.58 mmol) were dissolved in 50 ml of a 2:1 THF–hexane solution at r.t. under an inert atmosphere of argon. The mixture was heated at 65 °C for 4 days. A crude purple-blue solid was separated from the reaction mixture. A concentrated solution of the crude solid in dichloromethane was subjected to chromatography on Al_2O_3 column with CH_2Cl_2–hexane (80:20% v/v). The purple band was eluted and collected. Purple microcrystals were obtained from the eluent by slow evaporation of the solvent with a flow of argon. The product was obtained in 0.31 g (66%) yield.

Anal. Calc. for $C_{56}H_{48}N_4O_{10}Mo_2$ (MW = 1128.896): C, 59.58; H, 4.29; N, 4.96. Found: C, 59.5; H, 3.8; N, 5.2. Selected Mass spectra Fragments, *m/z*: 1130, 939.4, 831.3, 564, 303.

3. Results and discussion

Stoichiometric reactions between molybdenum hexacarbonyl, $Mo(CO)_6$ and α-benzoinoxime, H_2BNO under different atmospheric conditions in THF–hexane solvent have yielded two different binuclear oxomolybdenum complexes. The brown complex, $Mo_2O_5(HBNO)_2$ (**1**), was obtained by reacting $Mo(CO)_6$ with the oxime ligand in refluxing THF–hexane solvent in a sealed tube under vacuum. The second complex, $Mo_2O_2(HBNO)_4$ (**2**), was obtained as a purple solid from the reaction of $Mo(CO)_6$ and H_2BNO under an inert atmosphere of argon. However, efforts was not made to assure an oxygen-free system, and this could explain the formation of the compound **2** as an oxomolybdenum complex which my result by the reaction with trace oxygen.

The complexes were initially characterized on the basis of their mass spectra and elemental analyses. The parent ion peak (m/z) observed in the mass spectra at 725 and 1130 for complexes **1** and **2**, respectively, indicated dimeric structures. The mass spectrum of complex **1** has revealed peaks which could assigned to fragments containing two molybdenum atoms, and which clearly represented fragments derived from the dimer $Mo_2O_5(HBNO)_2$. Thus, fragments formed by loss of one or two OH groups from the dimer were revealed by peaks at $m/z = 707$ and 690.6, respectively. Significantly, peaks corresponding to MO_2O_5 and Mo_2O_3 fragments were observed at $m/z = 271$ and 240.1. On the other hand, the mass spectrum of the complex **2** differed significantly from that of complex **1** in having peaks, which could be assigned to fragments containing one and two molybdenum atoms. Thus, the mass spectrum of complex **2** could be divided into two regions. Peaks corresponding to fragments containing two molybdenum atoms occurred in the range $m/z >$ 564 and others containing one molybdenum atom occurred in the range $m/z \leq 564$. A peak corresponding to the monomer $[MoO(HBNO)]^+$ was observed at $m/z = 564$, indicating the possibility of dimer dissociation inot monomers.

Analyzing the IR spectra of complexes **1** and **2** was useful in assigning the ligands coordination modes to molybdenum centers. The IR data are given in Table 1. Both complexes displayed two bands corresponding to the OH and C=N stretching vibrations of the oxime group. These bands were observed, respectively, at 3435 and 1535 cm^{-1} for complex **1** and at 3428 and 1571 cm^{-1} for complex **2**. The OH stretch has shifted ~100 cm^{-1} (with respect to the free ligand) to higher frequency in both complexes. In contrast, the band due to C=N stretch displayed a shift to lower frequency of 135 and 99 cm^{-1} for complexes **1** and **2**, respectively. Such behavior evidently indicates that the α-benzoinoxime ligands coordinate to molybdenum centers through the nitrogen atom of the C=N–OH group [11,12]. The IR spectrum of complex **1** showed two strong bands at 973 and 901 cm^{-1} corresponding, respectively, to asymmetric and symmetric Mo=O stretching vibrations and one band at 730 cm^{-1} due to the asymmetric stretching vibration of Mo–O–Mo moiety. These vibrations compare well with those reported for $[NH_4]_2[Mo_2O_5(cat)_2]\cdot 2H_2O$ [13], and $[Mo_2O_5(chrySQ)_2]$ [7] complexes. This suggests that complex **1** has a similar structure that consists of two cis-dioxomolybdenum moieties (*cis*-MoO_2) bonded to each other through an oxo-bridging ligand. The IR spectrum of complex **2** revealed one strong band at 915 cm^{-1} corresponding to the Mo=O asymmetric stretching vibration. The absence

Table 1
Characteristic IR absorption bands of molybdenum benzoinoxime complexes

Complex	IR data (cm^{-1})[a]									
	ν_{OH}	$\nu_{C=N}$	$\nu_{C=C}$	ν_{C-O}	ν_{N-O}	$\nu_{Mo=O\ (asym)}$	$\nu_{Mo=O\ (sym)}$	$\nu_{Mo-O-Mo}$	ν_{Mo-O}	ν_{Mo-N}
H_2BNO	3326 (vs, b)	1670 (m)	1603 (m), 1495 (s)	1244 (s)	1015 (s)					
$Mo_2O_5(HBNO)_2$	3435 (vs, b)	1535 (sh)	1580 (m), 1491 (s)	1248 (m)	1054 (m), 1012 (s)	973 (s)	901 (s)	740 (sh)	634 (w)	570 (w)
$Mo_2O_2(HBNO)_4$	3428 (s)	1571 (sh)	1599 (sh), 1490 (s)	1330 (s), 1250 (m)	1055 (m), 1012 (s)		847 (m)		640 (sh)	577 (s)

[a] w, weak; m, medium; s, strong; vs, very strong; b, broad.

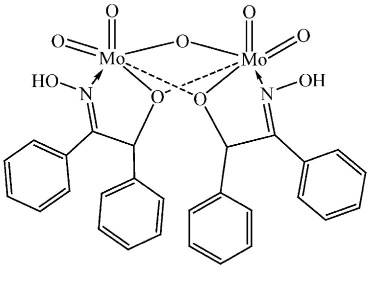
of the Mo=O symmetric stretching vibration may suggest a symmetrical molecule in which the two Mo=O moieties are located in a transposition to one another. These results are in agreement with those observed for $[Mo^{VI}O(O_2C_6H_2(t\text{-Bu})_2)_2]_2$ complex [4]. It is noteworthy that the $[Mo^{VI}O(O_2C_6H_2(t\text{-Bu})_2)_2]_2$ complex showed the Mo=O asymmetric stretching vibration at higher frequency (990 cm^{-1}) compared with that observed for complex **2.** This may attributed to the lower oxidation state of the Mo center in complex **2** (vide infra) which could result in a shift of the Mo=O stretching vibration to lower frequency. On the bases of the above IR spectral studies, supported by elemental analyses and mass spectroscopic data, the structures given in Fig. 1 are suggested for complexes **1** and **2**.

The electronic absorption spectra recorded for the two complexes and the free ligand as well in DMSO solution over a range of 190–900 nm are summarized in Table 2. The free ligand displayed two absorptions at 272 and 405 nm corresponding, respectively, to π–π* and n–π* transitions. These transitions were observed at 288 and 460 nm for complex **1** and at 275 and 484 nm for complex **2**, respectively. In addition, the intense absorptions observed at 318 nm ($\log \epsilon = 4.0$) for complex **1** and at 291 nm ($\log \epsilon = 3.7$) for complex **2** were assigned to the oxygen-to-molybdenum charge transfer transition. This transition occurs at lower energy compared with that reported for $MoOCl_4$ complex (270 nm) [14]. This is reasonable since the $MoOCl_4$ complex contains no donor ligands trans to the oxo ligands.

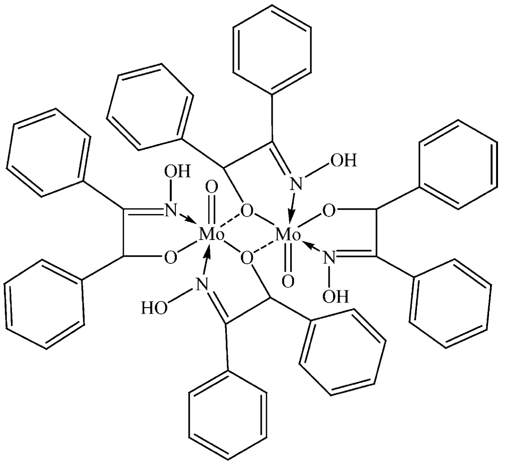
Table 2
The electronic absorption data for α-benzoinoxime and its molybdenum complexes recorded in DMSO

Compound	λ (nm; log ε)
H_2BNO	272 (2.6), 405 [a] (1.4)
$Mo_2O_5(HBNO)_2$	288 (3.9), 318 (4.0), 460 [b] (2.2)
$Mo_2O_2(HBNO)_4$	272 (4.2), 291 (3.7), 484 [b] (2.4)

[a] Shoulder.
[b] Broad.



(1)



(2)

Fig. 1. Suggested structures for $Mo_2O_5(HBNO)_2$ **1** and $Mo_2O_2(HBNO)_4$ **2** complexes.

The magnetic susceptibility measurements of the $Mo_2O_2(HBNO)_4$ complex were carried out on a solid sample at 298 K. The effective magnetic moment per molecule (μ_{eff}/molecule) was found to be 4.32 B.M. For a dimolybdenum(IV) complex, an intramolecular magnetic exchange will produce $S = 0$, $S = 1$ and $S = 2$ magnetic states. When the exchange is antiferromagnetic, the $S = 0$ state will be the ground state. Raymond and co-workers [15], have reported that dimolybenum(IV) complexes containing Mo–Mo bond are diamagnetic as a result of molybdenum–molybdenum bonding interaction. In addition, Mono-oxo-bridged metal ion dimers usually exhibit a greater degree of exchange interactions [16]. These make the observed magnetic moment for the $Mo_2O_2(HBNO)_4$ complex fits well with the suggested dimeric structure containing two Mo(IV) centers bonded through the benzoinoxime ligand and excludes the presence of Mo–Mo or Mo–O–Mo bonding. However, the decrease in the magnetic moment than the spin-only value expected for four unpaired electrons (4.9 B.M) may be attributed to the weak intramolecular interaction between the two Mo(IV) centers through the benzoinoxime bridging ligands. The magnetic susceptibility determination carried out on $Mo_2O_5(HBNO)_2$ indicated a diamagnetic complex.

The ^1H NMR spectrum of α-benzoinoxime ligand in DMSO-d_6 solvent revealed complex signals centered at 7.4 ppm due to the resonance of the ten-phenyl protons. It also, showed two doublets at 5.65 and 4.42 ppm corresponding, respectively, to H–C–OH* and H*–C–OH protons. In addition, a signal due to the oximic OH appeared at 11 ppm. The diamagnetic $Mo_2O_5(HBNO)_2$ complex showed a sharp ^1H NMR spectrum in DMSO-d_6 solvent. The spectrum has featured the disappearance of the signal corresponding to the alcoholic OH (H–C–OH*). In addition, the signal due to the oximic OH proton displayed an up field shift to 11.4 ppm. This, in turn, suggests that the $Mo_2O_5(HBNO)_2$ complex has a structure in which the α-benzoinoxime ligands coordinate to the molybdenum(VI) centers through the nitrogen of the oxime group and the deprotonated alcoholic oxygen.

The electrochemical properties of complexes **1** and **2** were investigated by cyclic voltammetry. The voltammograms shown in Figs. 2 and 3 indicated a different electrochemical behavior for the molybdenum metal centers in the two complexes.

The cyclic voltammetric reduction of the neutral $Mo_2O_5(HBNO)_2$ complex in MeCN solvent at a scan rate of 200 mV s^{-1} (Fig. 2) occurred in two reversible one-electron steps at potentials of -0.147 ($\Delta E_p = 94$ mV) and -0.680 V ($\Delta E_p = 93$ mV) versus SCE. The diffusion-control one-electron reduction character of the two reduction processes is established by the fact that their voltammetric peak current vary linearly with the square root of the scan rate between 20 and 500 mV s^{-1}. In addition, the constant $i_{pc}/v^{1/2}$ ratio and $i_{pa}/i_{pc} \approx 1.0$ (Table 3) at different scan rate is a typical diffusion control one-electron process [17]. Consequently, the first reduction at -0.147 V could be attributed to Mo^{VI}–O–Mo^{VI}/Mo^{VI}–O–Mo^{V} couple and the second at -0.680 V to the formation of the $Mo_2{}^{V}O_5$ species.

$$[Mo_2{}^{VI}O_5(HBNO)_2] \overset{e^-}{\rightleftharpoons} [Mo_2{}^{V,VI}O_5(HBNO)_2] \overset{e^-}{\rightleftharpoons} [Mo_2{}^{V}O_5(HBNO)_2]^{2-}$$

Our interpretation has gained a further support by carrying out ^{1}H NMR measurement on a 0.15 M tetra-*n*-butlylammonium perchlorate solution of the $Mo_2O_5(HBNMO)_2$ complex in MeCN solvent reduced at -0.726 mV (vs. SCE). The spectrum showed sharp signals due to the aromatic protons of the benzoinoxime ligands, indicating a diamagnetic complex. This is a characteristic of an oxobridged molybdenum(V) dimer [18].

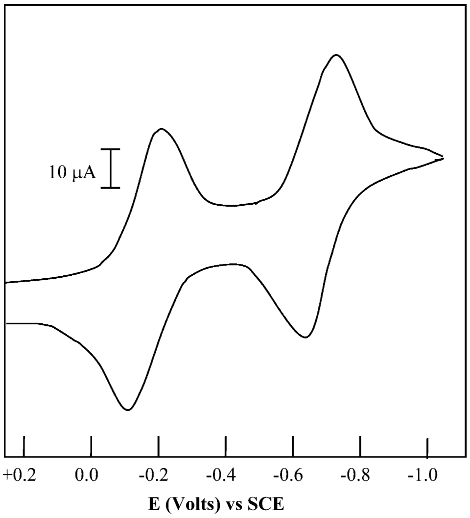


Fig. 2. The cyclic voltammogram for complex **1** in MeCN at scan rate 200 mV s^{-1}.

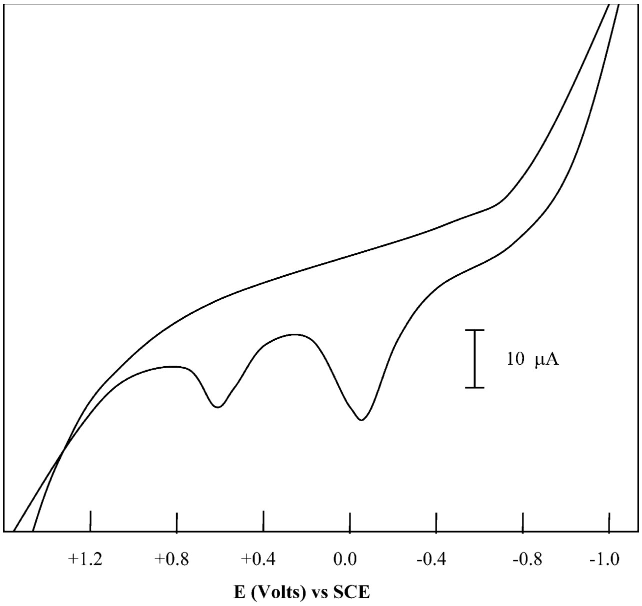


Fig. 3. The cyclic voltammogram for complex **2** in DMF at scan rate 100 mV s^{-1}.

Pierpont and co-workers [19,20] have reported two-electron reduction for dimeric molybdenum(VI) complexes of the catecholate ligand. In these complexes the two Mo(VI) metal centers bridge through the catecholate ligand. In the present work, the two one-electron reduction observed for the $Mo_2O_5(HBNO)_2$ complex could be attributed to the extensive delocalization of the charge between the molybdenum centers through the oxo-bridging ligand. The degree of charge delocalization can be determined by calculating the comproportionation constant, $K_c = \exp(\Delta EF/RT)$ [21,22]. For the $Mo_2O_5(HBNMO)_2$ complex; $\Delta E = 533$ mV, and this corresponds to $K_c = 1.03 \times 10^9$.

On the contrary, the $Mo_2O_2(HBNMO)_4$ complex does not undergo reduction but exhibits two irreversible oxidations at -0.084 and $+0.610$ V versus SCE in DMF solvent (Fig. 3). We were not able to determine directly the number of electrons transferred in each stage of the cyclic voltammogram due to the irreversibility of the processes. However, we can assume according to the value of the current at -0.084 V ($i_p = 16.5$ μA) is twice that for the second oxidation process at $+0.610$ V ($i_p = 8.4$ μA) that a dimeric $Mo_2{}^{V}O_2$ complex $[Mo_2{}^{V}O_2(HBNO)_4]^{2+}$ is the product of the first oxidation and a monomeric Mo(VI) species is the product of the second [23]. Attempts have been made to isolate the Mo(V) dimeric complex $[Mo_2{}^{V}O_5(HBNO)_4]^{2+}$ but were unsuccessful since the solution color of the neutral complex turns to greenish-yellow at -0.084 V (vs. SCE) and rapidly decomposes to unidentified product upon separation.

It must be recalled that the two-electron oxidation observed for the $Mo_2O_2(HBNMO)_4$ complex indicates a

Table 3
Cyclic voltammetric data for $Mo_2O_5(HBNO)_2$ complex in MeCN

v (mV s^{-1})	First wave				Second wave			
	ΔE_p (mV)	i_{pc} (μA)	$i_{pc}/v^{1/2}$	i_{pa}/i_{pc}	ΔE_p (mV)	i_{pc} (μA)	$i_{pc}/v^{1/2}$	i_{pa}/i_{pc}
20	87	10.8	2.47	0.96	90	11.3	2.58	0.92
50	93	17.4	2.46	0.96	92	17.2	2.43	0.93
100	90	23.8	2.38	0.97	92	22.5	2.25	0.94
200	94	32.6	2.31	0.97	93	33.3	2.36	0.93
500	96	51.5	2.30	0.98	94	52.4	2.34	0.95

lower delocalization between the two Mo(IV) centers and exclude the presence of oxo-bridging ligand. This agrees with the IR and the magnetic susceptibility data and confirms the suggested structure of the complex.

References

[1] R.W. Hay, Bioinorganic Chemistry, Ellis Horwood, London, 1991, p. 21.
[2] M.J. Kendrick, M.T. May, M.J. Plishka, K.D. Robinson, Metals in Biological Systems, Ellis Horwood, London, 1992, p. 113.
[3] R.M. Buchanan, C.G. Pierpont, Inorg. Chem. 18 (1979) 1616.
[4] C.G. Pierpont, R.M. Buchanan, J. Am. Chem. Soc. 97 (1975) 6450.
[5] M.J. Morris, Coord. Chem. Rev. 172 (1998) 181.
[6] W.E. Newton, J.W. McDonald, K. Yamanouchi, J.H. Enemark, Inorg. Chem. 18 (1979) 162.
[7] R.M. Ramadan, A.S. Attia, M.F. El-Shahat, Polyhedron 15 (1996) 301.
[8] R.M. Ramadan, M.S.A. Hamza, A.S. Attia, Polyhedron 16 (1997) 229.
[9] L.N. Mulay, Magnetic Susceptibility, Wiley, New York, 1963, p. 1779.
[10] I. Lewis, R.G. Wilkins, Modern Coordination Chemistry, Interscience, New York, 1960, p. 403.
[11] A.S. Attia, S.F. El-Mashtouly, M.F. El-Shahat, Synth. React. Inorg. Met.-Org. Chem. 32 (2002) 509.
[12] N.S. Biradar, V.B. Mahale, V.H. Kulkarni, J. Inorg. Nucl. Chem. 35 (1973) 2565.
[13] W.P. Griffith, C.A. Pumphrey, T. Rainey, J. Chem. Soc., Dalton Trans. (1986) 1125.
[14] M.L. Larson, F.W. Moore, Inorg. Chem. 5 (1966) 801.
[15] D.L. Hughes, J.D. Lane, R.L. Richards, C. Shortman, J. Chem. Soc. Dalton Trans. (1994) 621.
[16] L. Borer, L. Thalken, C. Ceccarelli, M. Glik, J.H. Zhang, W.M. Reiff, Inorg. Chem. 22 (1983) 1719–1724.
[17] K.R. Dunbar, R.A. Waiton, Inorg. Chim. Acta 87 (1984) 185.
[18] B. Korpar-Colig, M. Cindric, D. Matkovic-Calogovic, V. Vrdoljak, B. Kamenar, Polyhedron 21 (2002) 147.
[19] L.A. deLearie, C.G. Pierpont, Inorg. Chem. 27 (1988) 3842.
[20] R.M. Buchanan, C.G. Pierpont, Inorg. Chem. 18 (1979) 1616.
[21] A.W. Bott, Curr. Sep. 16 (1997) 61.
[22] T. Ito, T. Hamaguchi, H. Nagino, T. Yamaguchi, H. Kido, I.S. Zavarine, T. Richmond, J. Washington, C.P. Kubiak, J. Am. Chem. Soc. 121 (1999) 4625.
[23] V. Ung, S.M. Couchman, H.C. Jeffery, J.A. McCleverty, M.D. Ward, F. Totti, D. Catteschi, Inorg. Chem. 38 (1999) 365.