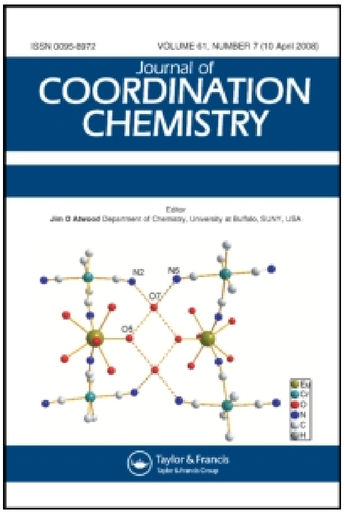

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Synthesis, chemical, and electrochemical studies of oxomolybdenum(IV) complexes with two tridentate ONS donor ligands

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This manuscript reports the synthesis, characterization, chemical, and electrochemical studies of several oxomolybdenum (IV) complexes of tridentate ONS donor ligands obtained by the condensation of 2-hydroxyacetophenone with S-benzyl and S-methyl dithiocarbazates. The complexes are of the forms $[\mathrm{Mo^{IV}OL}]$ and $[\mathrm{Mo^{IV}OL(N–N)}]$, where N–N = 2,2′-bipyridine and 1,10-phenanthroline, derived from the corresponding $[\mathrm{MoO_2L}]$ complexes through oxo abstraction by triphenylphosphine in the absence/presence of N–N bidentate donors. Electrochemical behavior of these complexes have been investigated for insight into the redox behavior of the Mo(IV) centers of these complexes.

Keywords: Oxomolybdenum(IV) complexes; Dithiocarbazate; Schiff bases; $\mathrm{PPh_3}$

1. Introduction

As part of our studies on dioxomolybdenum(VI) and oxomolybdenum(IV) complexes, we report here the synthesis, characterization, and reactivity of some oxomolybdenum(IV) complexes with binegative tridentate dithiocarbazate-type Schiff-base ligands containing O, N, and S donors [1, 2] (scheme 1).

Oxomolybdenum(IV) complexes with similar ONS ligands but different N–N donor co-ligands were also obtained by one-pot synthesis from the corresponding *cis*-dioxomolybdenum(VI) precursors. The complexes are of the forms $[\mathrm{Mo^{IV}OL}]$ and $[\mathrm{Mo^{IV}OL(N–N)}]$, where N–N = bipy and phen. Electrochemical behavior of these complexes give insight into the redox behavior of the Mo(IV) centers.

Binegative tridentate ligands are of intrinsic interest because their complexes with $[\mathrm{MoO_2}]^{2+}$ or $[\mathrm{MoO}]^{2+}$ core result in $[\mathrm{MoO_2L}]$ and $[\mathrm{MoOL}]$, leaving one or two "open" coordination sites which can be utilized for substrate binding. Vacant sites of $[\mathrm{Mo^{IV}OL}]$

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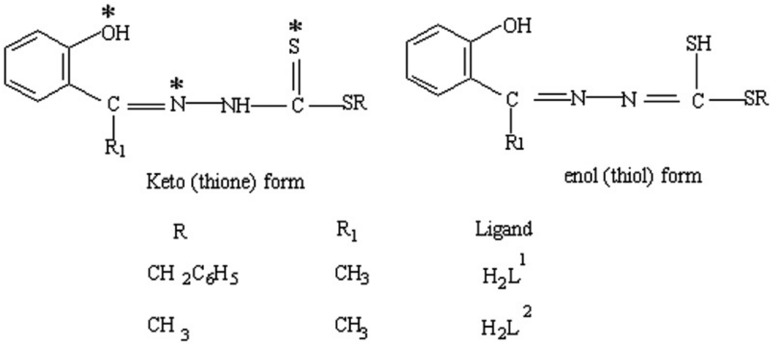

Scheme 1. Tautomeric thione and thiol forms of the Schiff bases.

can be utilized for binding a neutral chelating bidentate ligand which can play a very important role in the stabilization of Mo(IV) and Mo(V) oxidation states [3].

We have followed the general strategy of the synthesis of Mo(IV) complexes with a $[MoO]^{2+}$ core from the corresponding $[MoO_2]^{2+}$ complexes through oxo abstraction by triphenylphosphine in the absence/presence of N–N bidentate donors in a purified dry N_2 atmosphere [4]. This oxo transfer reaction may be visualized as a simple bimolecular reaction that involves the interaction between one $[Mo^{VI}O_2L]$ and one PPh_3 in the activated complex leading to the transfer of oxygen by the donation of the lone pair of electrons of phosphorus into the antibonding $Mo{=}O\pi^*$ orbital, leading to the formation of the P–O bond and an Mo(IV)–oxo complex with a $4d_{xy}^2$ configuration. Hence, the reaction may be considered as a two-electron redox/oxygen atom transfer process; $[Mo^{VI}O_2]^{2+}$, an established class 1 electrophile, is known to oxidize electron-rich olefins [5].

2. Results and discussion

2.1. *Synthesis and characterization of the ligands*

The Schiff-base ligands (H_2L^1 and H_2L^2) were prepared by condensing S-benzyl and S-methyl dithiocarbazates with 2-hydroxyacetophenone in ethanol [2]. The ligands were satisfactorily characterized by elemental analyses, IR, and ^1H NMR data.

2.2. *Synthesis and characterization of oxomolybdenum(IV) complexes*

The oxomolybdenum(IV) complexes (**1** and **2**), $[Mo^{IV}OL]$, were prepared by [4] refluxing the solution of the appropriate $[Mo^{VI}O_2L]$ complex with PPh_3 in 1 : 1.5 molar proportions in dry degassed CH_3CN under dry nitrogen. The orange solution turned dark brown and a brown compound separated out within 10 min, which was filtered and washed several times with dry degassed CH_3CN and dried *in vacuo* over anhydrous $CaCl_2$.

$$Mo^{VI}O_2L + PPh_3 \xrightarrow{CH_3CN} Mo^{IV}OL + OPPh_3$$

The oxomolybdenum(IV) complexes (**3**–**6**), [$Mo^{IV}OL(N–N)$], were prepared by [4] refluxing the appropriate [$Mo^{VI}O_2L$] complex with PPh_3 in the presence of bipy or phen in 1 : 1.5 : 10 molar proportions in dry degassed CH_2Cl_2 for 1–2 h under dry nitrogen. The green solutions so formed were poured into *n*-hexane whereupon green solids separated were filtered and washed with dry CH_2Cl_2 several times and dried *in vacuo* over anhydrous $CaCl_2$.

$$Mo^{VI}O_2L + N{-}N + PPh_3 \xrightarrow{CH_2Cl_2} Mo^{IV}OL(N{-}N) + OPPh_3$$

The [$Mo^{IV}OL$] and [$Mo^{IV}OL(N–N)$] complexes are amorphous solids, stable at room temperature, and diamagnetic, consistent with a d^2 Mo(IV) center [4, 6–8]. The complexes are readily soluble in alcohol, CH_2Cl_2, CH_3CN, DMF, and DMSO. The brown color of DMSO solutions of **1** and **2** gradually change to the characteristic orange color of the parent [$Mo^{VI}O_2L$] complexes with the simultaneous emission of Me_2S. This has been verified by comparing the electronic spectrum with that of the parent [$Mo^{VI}O_2L$] complex. When brown solutions of Mo(IV) complexes in DMF are reacted with pyridine N-oxide, the color changes to orange indicating oxo transfer from pyridine N-oxide to $[MoO]^{2+}$. These observations indicate that the Mo(IV) center abstracts the oxo group from DMSO and pyridine N-oxide to regenerate the parent dioxo Mo(VI) complex [9–11]. Molar conductivity data in 10^{-3} M CH_2Cl_2 solution indicate that they are non-electrolytes. All the oxomolybdenum(IV) complexes are satisfactorily characterized by elemental analyses, IR, electronic spectra, and cyclic voltammetric data.

2.3. *IR spectra*

Characteristic IR bands of the two ligands and the corresponding dioxomolybdenum(VI) complexes were reported [1, 2]. The IR bands of oxomolybdenum(IV) complexes are given in section 3.4 and a representative IR spectrum is shown in Supplementary material. A single strong band at ~970 cm^{-1} for [$Mo^{IV}OL$] complexes and a similar band at ~940 cm^{-1} for the [$Mo^{IV}OL(N–N)$] complexes are attributed to $\nu(Mo{=}O)_t$ [4, 7, 8], as opposed to the twin symmetric and antisymmetric stretching bands for the $[MoO_2]^{2+}$ moiety in the [$Mo^{VI}O_2L$] precursors [6]. Coordination of bipy or phen to $[Mo^{IV}O]^{2+}$ lowers the $\nu(Mo{=}O)_t$ stretching frequency considerably [10, 12, 13]. Comparing the IR spectra of [$Mo^{VI}O_2L$], [$Mo^{IV}OL$], and [$Mo^{IV}OL(N–N)$], the main ligand framework remains unaltered. It is likely that the [$Mo^{IV}OL$] complexes are polymeric [4, 7, 8], probably through Mo=O···Mo linkage. This is further supported by the presence of a medium intensity band around 815 cm^{-1} characteristic of Mo=O···Mo species [9], which is absent in the spectra of [$Mo^{IV}OL(N–N)$] complexes. A strong $\nu(C{=}N)$ band at 1590–1604 cm^{-1} indicates the coordination of azomethine nitrogen to the oxo Mo(IV) center [14, 15]. A new band in the 619–634 cm^{-1} region in the complexes is assigned to $\nu(Mo–N)$ [12, 13]. Thioenolate sulfur coordination is corroborated by the $\nu(Mo–S)$ band around 300–400 cm^{-1} [16].

2.4. *Electronic spectra*

Electronic spectra of **1**–**6** were recorded in dry dichloromethane and the spectral data of complexes are presented in section 3.4 (a representative electronic spectrum is provided

Table 1. Cyclic voltammetric results[a] (V *vs.* SCE) for oxomolybdenum(IV) complexes at 298 K.

Complexes	Mo(IV)/Mo(VI) Epa (V)	Mo(VI)/Mo(V) Epc (V)	Mo(V)/Mo(IV) Epc (V)
MoOL^1 (**1**)	+0.54	−0.44	−1.04
MoOL^2 (**2**)	+0.51	−0.45	−1.02
MoOL^1(bipy) (**3**)	+0.52	−0.48	−0.88
MoOL^2(bipy) (**4**)	+0.50	−0.44	−1.01
MoOL^1(*o*-phen) (**5**)	+0.51	−0.49	−0.95
MoOL^2(*o*-phen) (**6**)	+ 0.48	−0.45	−1.01

[a] Solvent: DMF (dry, degassed); supporting electrolyte: 0.1 M TEAP; solution strength: 10^{-3} M; working electrode: platinum; reference electrode: SCE; 50.

in the Supplementary material). Spectra of [Mo^{IV}OL] and [Mo^{IV}OL(N–N)] complexes exhibit several bands in the 658–240 nm range. Absorption maxima in the 470–400 nm range may be assigned to the thiolato S(pπ)–Mo(dπ) LMCT transition [4, 6] from the promotion of an electron from the filled HOMO of primarily sulfur pπ character to the empty LUMO of molybdenum. Bands at 380–300 nm are assigned to nitrogen to molybdenum and oxygen to molybdenum charge transfer transitions [6, 17]. Bands below 300 nm are due to intra-ligand transitions. The complexes exhibit one absorption maximum around 657 nm characteristic of $[MoO]^{2+}$ [4, 7, 8].

2.5. *Electrochemical properties*

Electron transfer behavior of **1**–**6** has been examined in dry degassed DMF solution using cyclic voltammetry at a platinum electrode with 0.1 M TEAP as the supporting electrolyte. Cyclic voltammetric data of all the Mo(IV) complexes are included in table 1 and a representative voltammogram is shown in figure 1. All the complexes exhibit a reduction and oxidation wave. For [Mo^{IV}OL] complexes an initial positive scan at a rate of 50 mV s^{-1} gives only a single irreversible oxidative response at +0.51 to +0.54 V, which is assigned to Mo(IV)/Mo(VI) [4, 17, 18]. This electrogenerated Mo(VI) species undergoes post-electrochemical reaction with the solvent. On scan reversal this Mo(VI) species exhibits two one-electron reductive responses around –0.44 to –0.49 V and –0.88 to –1.04 V corresponding to Mo(VI)/Mo(V) and Mo(V)/Mo(IV) processes [4, 6–8], respectively. The [Mo^{IV}OL(N–N)] complexes show similar electrochemical behavior to [Mo^{IV}OL] complexes.

2.6. *Study of reactivity*

Oxo-transfer from DMSO: The oxygen atom transfer from DMSO to [Mo^{IV}OL] and [Mo^{IV}OL(N–N)] complexes has been studied spectrophotometrically in DMF solution. As soon as [Mo^{IV}OL]/[Mo^{IV}OL(N–N)] is dissolved in DMSO a change of color commences, ultimately becoming yellow-orange with all the characteristic features of [Mo^{VI}O$_2$L]. This change is almost instantaneous for the [Mo^{IV}OL] complexes, while solutions of [Mo^{IV}OL(N–N)] take around 10 min to undergo such a change. The bands at 657 nm gradually disappear within this time. These changes are accompanied by a distinct smell of Me$_2$S. Similar changes were also observed with the addition of pyridine N-oxide to DMF solution of the samples. These observations clearly indicate [16, 17]

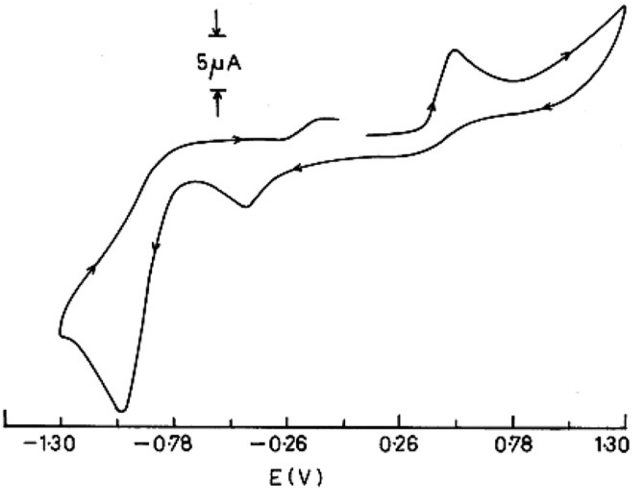

Figure 1. Cyclic voltammogram of $MoOL^2$ in DMF at 298 K.

the transfer of oxygen from DMSO/pyridine N-oxide to $[MoO]^{2+}$, leading to the formation of $[MoO_2]^{2+}$. The reaction may be represented as

$$MoOL + DMSO \xrightarrow{DMF} MoO_2L + Me_2S$$

The orange complexes $[Mo^{VI}O_2L]$ are isolated from the concentrated DMSO solution of the corresponding $[Mo^{IV}OL]$-type complexes by adding excess ether and characterized by elemental analysis and spectroscopic techniques.

This oxo-accepting behavior of these complexes with a consequent oxidation at the Mo center from IV to VI may be related to the oxo-transfer behavior of oxido-reductase enzymes, which bring about a two-electron reduction of the substrate by removing an oxo ligand.

3. Experimental

3.1. *Materials*

$[MoO_2(acac)_2]$ was prepared as described in the literature [19]. Reagent grade solvents were dried and distilled prior to use. All other chemicals used for preparative work were of reagent grade, available commercially and used without purification.

3.2. *Physical measurements*

Elemental analyses were performed on a Perkin-Elmer 240 C, H, and N analyzer. NMR spectra were recorded on a Bruker 300 L NMR spectrometer operating at 300 MHz with TMS as internal standard. IR spectra were recorded as KBr pellets on a Perkin-Elmer model 883 infrared spectrophotometer. Electronic spectra were recorded on a HITACHI U-3501 UV-Vis recording spectrophotometer. Magnetic susceptibility was measured with a PAR model 155 vibrating sample magnetometer with $Hg[Co(SCN)_4]$ as calibrant. Electrochemical data were collected on a Sycopel model AEW2 1820 F/S instrument at 298 K using a Pt working electrode, Pt auxiliary electrode, and SCE reference electrode. Cyclic voltammograms were recorded in DMF containing 0.1 M TEAP as a supporting electrolyte.

3.3. *Synthesis of Schiff-base ligands*

H_2L^1 and H_2L^2 were prepared by a method reported previously [2].

3.4. *Synthesis of MoOL complexes*

3.4.1. MoOL^1 (1). To a refluxing solution of 0.442 g (1.0 mmol) of MoO_2L^1 in 25 mL dry degassed methanol, 0.393 g (1.5 mmol) of PPh_3 in 15 mL of degassed methanol under purified dinitrogen was added. The orange-red solution turned dark brown and a brown compound separated within 10 min, was filtered, washed well with methanol, and dried *in vacuo* over anhydrous $CaCl_2$. Yield: ~80%. Anal. Calcd for $C_{16}H_{14}N_2S_2O_2Mo$ (%): C, 45.07; H, 3.28; N, 6.57; Mo, 22.53. Found (%): C, 44.62; H, 3.22; N, 6.45; Mo, 21.87. IR (KBr pellet), cm^{-1} : $\nu_{(C=N)}$ 1595 (s), $\nu_{(Mo=O)}$ 977 (vs), $\nu_{(Mo-S)}$ 330 (m), $\nu_{(Mo-N)}$ 634 (s), UV-Vis (CH_2Cl_2) [λ_{max} (nm) (ϵ ($dm^3\,mol^{-1}cm^{-1}$))]: 300 (3567), 339 (sh) (2402); 388 (2424), 470 (2454).

3.4.2. MoOL^2 (2). This compound was prepared by following the method described above for the preparation of $MoOL^1$. Yield : ~80%. Anal. Calcd for $C_{10}H_{10}N_2S_2O_2Mo$ (%) : C, 34.28; H, 2.86; N, 8.00; Mo, 27.43. Found (%) : C, 33.75; H, 2.80; N, 7.98; Mo, 26.90. IR (KBr pellet), cm^{-1} : $\nu_{(C=N)}$ 1595 (m), $\nu_{(Mo=O)}$ 965 (vs), $\nu_{(Mo-S)}$ 365 (m), $\nu_{(Mo-N)}$ 636 (m), UV-Vis (CH_2Cl_2) [λ_{max} (nm) (ϵ ($dm^3\,mol^{-1}cm^{-1}$))]: 235 (4091), 298 (2124); 387 (1475), 468 (1572).

3.5. *Synthesis of MoOL(N–N) complexes, where N–N is bipy or phen*

Compounds **3**–**6** were prepared under dry N_2 by refluxing the parent MoO_2L with a mole ratio 1 : 10 : 1.5 of MoO_2L, N–N and PPh_3 in dry degassed CH_2Cl_2 for ~1–2 h. The green solutions formed were poured into *n*-hexane whereupon green solids separated were filtered, washed with CH_2Cl_2, and dried *in vacuo* over anhydrous $CaCl_2$. Yield : ~70–75%.

3.5.1. MoOL^1(bipy) (3). Anal. Calcd for $C_{26}H_{22}N_4S_2O_2Mo$ (%) : C, 53.60; H, 3.78; N, 9.62; Mo, 16.49. Found (%) : C, 52.68; H, 3.52; N, 9.47; Mo, 16.31. IR (KBr pellet), cm^{-1}: $\nu_{(C=N)}$ 1600 (vs), $\nu_{(Mo=O)}$ 950 (s), $\nu_{(Mo-S)}$ 301 (w), $\nu_{(Mo-N)}$ 630 (m), UV-Vis (CH_2Cl_2) [λ_{max}(nm) (ϵ ($dm^3\,mol^{-1}cm^{-1}$))]: 302 (12935), 342 (7900); 459 (1107).

3.5.2. MoOL^2(bipy) (4). Anal. Calcd for $C_{20}H_{18}N_4S_2O_2Mo$ (%) : C, 47.64; H, 3.56; N, 11.07; Mo, 18.97. Found (%) : C, 46.98; H, 3.45; N, 11.00; Mo, 19.20. IR (KBr pellet), cm^{-1}: $\nu_{(C=N)}$ 1596 (s), $\nu_{(Mo=O)}$ 954 (s), $\nu_{(Mo–S)}$ 390 (m), $\nu_{(Mo–N)}$ 637 (s), UV-Vis (CH_2Cl_2) [λ_{max}(nm) (ϵ ($dm^3\,mol^{-1}cm^{-1}$))]: 272 (6132), 384 (2210); 469 (2189).

3.5.3. MoOL^1(o-phen) (5). Anal. Calcd for $C_{28}H_{22}N_4S_2O_2Mo$ (%) : C, 55.44; H, 3.63; N, 9.24; Mo, 15.84. Found (%) : C, 54.80; H, 3.41; N, 9.17; Mo, 16.00. IR (KBr pellet), cm^{-1} : $\nu_{(C=N)}$ 1590 (s), $\nu_{(Mo=O)}$ 918 (vs), $\nu_{(Mo–S)}$ 326 (m), $\nu_{(Mo–N)}$ 619 (m), UV-Vis (CH_2Cl_2) [λ_{max}(nm) (ϵ ($dm^3\,mol^{-1}cm^{-1}$))] : 264 (22430), 297 sh (12796); 337 (8502).

3.5.4. MoOL^2(o-phen) (6). Anal. Calcd for $C_{22}H_{18}N_4S_2O_2Mo$ (%) : C, 49.81; H, 3.39; N, 10.56; Mo, 18.11. Found (%) : C, 49.00; H, 3.29; N, 10.47; Mo, 18.92. IR (KBr pellet), cm^{-1}: $\nu_{(C=N)}$ 1595 (m), $\nu_{(Mo=O)}$ 952 (vs), $\nu_{(Mo–S)}$ 395 (m), $\nu_{(Mo–N)}$ 637 (m), UV-Vis (CH_2Cl_2) [λ_{max}(nm) (ϵ ($dm^3\,mol^{-1}cm^{-1}$))] : 273 (5283), 385 (2171); 469 (2123).

4. Concluding remarks

Mononuclear Schiff-base complexes of transition metal ions including the MoO_2^{2+} core are widely studied. However, mononuclear oxomolybdenum(IV) complexes of ONS chelator Schiff bases are not adequately studied, mainly because of the difficulty involved in stabilizing the mononuclear MoO^{2+} core. This study provides a general way of synthesizing mononuclear $Mo^{IV}OL$ and $Mo^{IV}OL(N–N)$ complexes (where L^{2-} is the dianion of the ONS donor Schiff base) which can undergo reversible and facile MoO^{2+} transformations because of the combined presence of hard (O and N) and soft (S) donors in strategic positions of the Schiff base skeleton leading to stable five- or six-membered metallocycles. Binegative Schiff bases satisfy the 2+ charge of the metal leading to the formation of uncharged complexes which are stable and easy to isolate from the reaction medium. The presence of bipy and phen augment the stability of MoO^{2+} complexes and prevent polymerization.

References

[1] N.R. Pramanik, S. Ghosh, T.K. Raychaudhuri, S. Ray, R.J. Butcher, S.S. Mandal. *Polyhedron*, **23**, 1595 (2004).

[2] N.R. Pramanik, S. Ghosh, T.K. Raychaudhuri, S. Chaudhuri, M.G.B. Drew, S.S. Mandal. *J. Coord. Chem.*, **60**, 2177 (2007).

[3] J.M. Berg, K.O. Hodgson, A.E. Bruce, J.L. Corbin, N. Pariyadath, E.I. Stiefel. *Inorg. Chim. Acta*, **90**, 25 (1984).

[4] S. Purohit, A.P. Koley, L.S. Prasad, P.T. Manoharan, S. Ghosh. *Inorg. Chem.*, **28**, 3735 (1989).

[5] M.A.F. Sisemore, M. Selke, J.N. Burnstyn, J.S. Valentine. *Inorg. Chem.*, **36**, 979 (1997).

[6] A. Rana, R. Dinda, P. Sengupta, S. Ghosh, L.R. Falvello. *Polyhedron*, **21**, 1023 (2002), and references therein.

[7] R. Dinda, P. Sengupta, S. Ghosh, H.M. Figge, W.S. Sheldrick. *J. Chem. Soc., Dalton Trans.*, 4434 (2002) and references therein.

[8] R. Dinda, P. Sengupta, S. Ghosh, W.S. Sheldrick. *Eur. J. Inorg. Chem.*, 363 (2003), and references therein.

[9] I.W. Boyed, J.T. Spence. *Inorg. Chem.*, **21**, 1602 (1982).

[10] K. Yamanouchi, J.H. Enemark. *Inorg. Chem.*, **18**, 1626 (1979).

[11] (a) R.H. Holm, J.M. Berg. *Pure Appl. Chem.*, **56**, 1645 (1984); (b) J.A. Craig, E.W. Harlan, B.S. Snyder, M.A. Whitener, R.H. Holm. *Inorg. Chem.*, **28**, 2082 (1989).
[12] S.K. Dutta, D.B. McConville, W.J. Youngs, M. Chaudhury. *Inorg. Chem.*, **36**, 2517 (1997).
[13] Y.-L. Zhai, X.-X. Xu, X. Wang. *Polyhedron*, **11**, 415 (1992).
[14] A. Syamal, K.S. Kale. *Inorg. Chem.*, **4**, 867 (1965).
[15] M. Goodgame, P.J. Hayward. *J. Chem. Soc. A*, 632 (1966).
[16] M. Chaudhury. *J. Chem. Soc., Dalton Trans.*, 115 (1984).
[17] C. Bustos, O. Burckhardt, R. Schrebler, D. Carrillo, A.M. Arif, A.H. Cowley, C.M. Nunn. *Inorg. Chem.*, **29**, 3996 (1990).
[18] A.P. Koley, S. Purohit, S. Ghosh, L.S. Prasad, P.T. Manoharan. *J. Chem. Soc., Dalton Trans.*, 2607 (1998).
[19] W.C. Fernelius, K. Terada, B.E. Bryant. *Inorg. Synth.*, **6**, 147 (1960).