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INTEGRATED
RESEARCH ADVANCES

Chemistry of Oxomolybdenum compounds having Schiff bases

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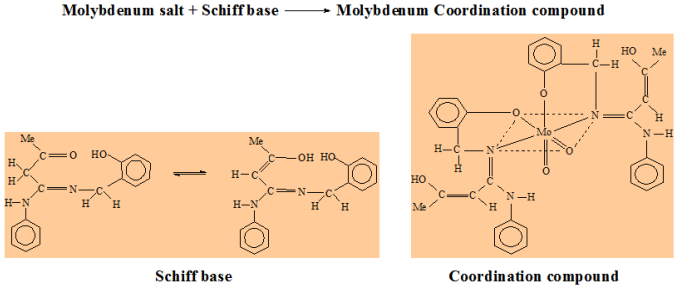
ABSTRACT



Schiff base compounds are excellent ligands due to their rich coordination chemistry with transition metal ions. This paper consists the study of the coordination compounds of dioxomolybdenum(VI) ions with different Schiff bases. These coordination compounds have wide applications in agricultural, industrial and pharmaceutical chemistry. These coordination compounds have been characterized on the basis of various physio-chemical techniques like IR, mass, reflectance, ^{1}H NMR, X-ray and magnetic susceptibility measurements. Thermal techniques such as TGA and DTA are also used for the study of these compounds.

Keywords: Coordination compounds, Schiff base, Dioxomolybdenum ion, spectral studies.

INTRODUCTION

Molybdenum is relatively harmless as compared to other transition metals. Although molybdenum exhibits a variety of oxidation states but (+6) state is the most stable and thoroughly investigated state, where it forms a large number of coordination compounds. The coordination compounds containing the oxomolybdenum group dominates the higher oxidation states. Molybdenum is an essential trace element for animals and plants. Owing to the importance in numerous biochemical redox reactions associated with enzymes, a considerable interest in the coordination compounds of molybdenum has drawn the attention of Chemists. It is the only element among the heavier transition elements which appears to have a major role as a trace element in enzymes. It is also one of the most biologically active elements and is an essential micronutrient for microorganisms, animals and plants.[1] Nature has incorporated molybdenum into a number of redox enzymes. About 20 different enzymes containing molybdenum are known in animals. Enzymes such as nitrogenase, nitrate reductase, xanthine dehydrogenase, xanthine oxidase, sulphite oxidase and aldehyde oxidase contain molybdenum atoms bonded through O, N and/or S atoms. These Mo sites are supposed to be active centers for the catalytic activity of the enzymes.[2]

It is actively involved in N_2 fixation. Mo concentration also affects protein synthesis, metabolism and growth. The O atom transfer properties of the MoO_2(VI) compounds play a significant role in investigating the mechanism of Mo oxotransferase.[3] The ability of MoO_2(VI) ions to form the coordination compounds with a wide range of ligands has been reported. These coordination compounds have found versatile uses in our life as antiinflammatory,[4] analgesic,[5] antibiotic,[6] antioxidative,[7,8] antifungal,[9-11] antibacterial,[12-14] antiviral,[15] antifertility,[16] enzymatic,[17] antitumor[18,19] compounds. We present here a brief survey of the chemistry of coordination compounds of MoO_2(VI) ions.

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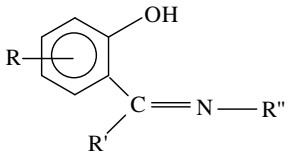
Coordination Compounds of Dioxomolybdenum(VI) ions

The ligands are coordinated to MoO_2(VI) ions in a mono-, bi-, tri-, tetra- and polydentate fashions.

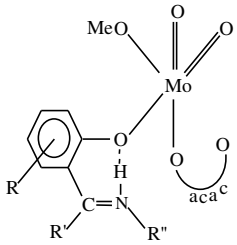
A. With monodentate ligands:

Cindric *et al.*[20] have claimed to become the first to synthesize the monomeric, air- and moisture-stable, yellow crystalline, six-coordinate compound, [MoO_2(LH)(OMe)(acac)] (where LH = **1**; R = H, 5,6-benzo, R' = H, R" = C_6H_5, $CH_2C_6H_5$). Interestingly, instead of the deprotonation of the ligand, the reaction takes place by the deprotonation of MeOH. It is worth to mention that potentially the bidentate ON donor ligand is bonded to the metal



[1]

ions through its phenolic OH group and not through the azomethine N atom. The ν_s(O==Mo==O) and the ν_{as}(O==Mo==O) stretches occur at ~930 and 900 cm^{-1} favoring the presence of *cis*-MoO_2 structure. The compound (when R = H) crystallizes in a triclinic fashion with a = 8.0478 Å, b = 10.2829 Å, c = 13.7790 Å, α = 76.541°, β = 82.295° and γ = 77.4°. Within the same ligand, the phenolic H atom is attached to azomethine N atom and forms an intramolecular H-bond of 2.548 Å with the O atom coordinated to Mo. The octahedral coordination of Mo is completed by two oxo-O, two acetylacetonato O and one O atom each of LH and methoxo ligand. A distorted octahedral structure (**2**; R = H, 5,6-benzo, R' = H, R" = C_6H_5, $CH_2C_6H_5$) has been suggested to it.

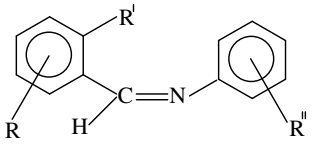


[2]

B. With bidentate ligands:

The syntheses of yellow to red, six-coordinate compounds, [MoO_2L_2] (where LH = monobasic bidentate ligand, **1**; R = H, 3-Me, 3-MeO, R' = H, R" = Me, Et, 1-Pr, 1-Bu, 1-C_7H_{15}, C_6H_5, 4-EtC_6H_4, 4-FC_6H_4, 3-ClC_6H_4, 4-ClC_6H_4, 4-BrC_6H_4, 4-IC_6H_4, 4-OHC_6H_4, 4-$NO_2C_6H_4$, 3-MeC_6H_4, 4-MeC_6H_4, 2-$OMeC_6H_4$, 4-$OMeC_6H_4$, 3,4-$Cl_2C_6H_3$ and $CH_2C_6H_5$) have been reported[21-23]. The compounds are sparingly soluble in MeOH, EtOH, CH_2Cl_2 and $CHCl_3$. The single crystal X-rays structure of [MoO_2L_2] (when R = H, R' = H, R" = 1-Pr) has cofirmed the presence of a *cis*-MoO_2 octahedral environment in it[24].

Zelentsov *et al.*[25] have synthesized the compounds, [MoO_2L_2] (where LH = **3**; R = R" = H, R' = OH). LH behaves as a monobasic bidentate ON donor ligand. The syntheses of [$MoO_2L'_2$] (where L'H = **3**; R = H, R' = OH, R" = H, 5-Cl, 5-Br, 5-NO_2, 5-OMe have also been reported[26-32]. The IR data are indicative of the monobasic bidentate ON donor nature of L'H.



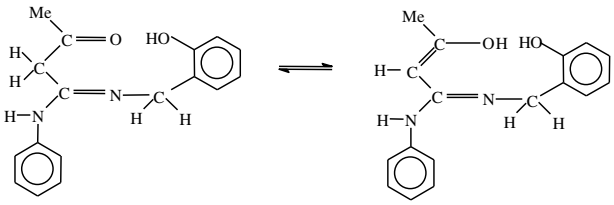
[3]

The syntheses of [$MoO_2(LH)_2$] and [$MoO_2(L'H_2)_2$] (where LH_2 = **1**; R = H, R' = Me, R" = OH and $L'H_3$ = **1**; R = 4-OH, R' = C_4H_9, R" = OH) have been reported[33]. Both LH_2 and $L'H_3$ behave as the monobasic bidentate ON donor ligands.

Although LH_2 (**3**; R = H, R' = OH, R" = 2-OH), $L'H_2$ (**3**; R = H, R' = OH, R" = 4-COOH) and $L"H_2$ (**3**; R = H, R' = OH, R" = 2-SH) are potentially the dibasic tridentate ONO/ONS donor ligands, but behave[34] as the monobasic bidentate ON donor ligands in [$MoO_2(LH)_2$], [$MoO_2(L'H)_2$] and [$MoO_2(L"H)_2$], respectively.

The synthesis of a six-coordinate heterochelate, [MoO_2(acac)(LH)] (here LH_2 = **3**; R = H, R' = OH, R" = 2-COOH) has been reported[35]. The monobasic bidentate ligand coordinates to the metal ions through its carboxylic O and azomethine N atoms.

The synthesis of six-coordinate [$MoO_2(LH_2)_2$] (here LH_3 = **4**) has been reported[36]. LH_3 acts as a monobasic bidentate ON donor ligand coordinating through ON donor ligand coordinating through its phenolic O and azomethine N atoms. The ν_s(O==M==O) and ν_{as}(O==M==O) stretches occur at 910 and 890 cm^{-1}, respectively. The absence of a band at ~770 cm^{-1} in the present MoO_2(VI) coordination compound indicates the absence of an oligomeric structure with ···Mo==O···Mo==O··· interaction. An octahedral structure has been suggested for the compound.

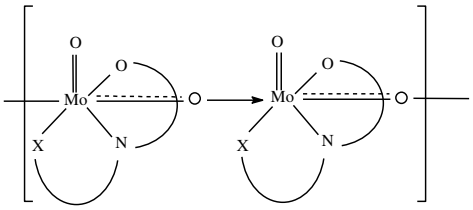


[4]

C. With tridentate ligands:

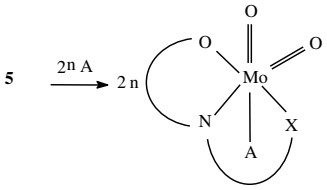
The syntheses of the compounds of LH_2, [**1**; R = H, 5-Cl, 5-Br, 5-NO_2, 5-Me, 5-OMe, 5,6-benzo, R' = H, R" = $(CH_2)_2OH$] have been reported by a number of researcher teams[29,37]. The compounds are of the types, [MoO_2L] or [MoO_2LS] (here S =

H_2O, EtOH). On heating at 100 °C, the monomeric, yellow coordination compound, $[MoO_2L(H_2O)]$ and $[MoO_2L(EtOH)]$ form the brown, five-coordinate, oligomeric compound,



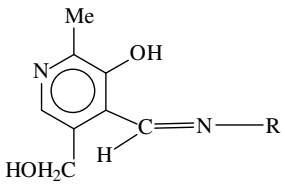
[5]

$[MoO_2L]$ (**5**; X = O). The latter upon salvation with DMF or DMSO forms the six-coordinate solvated adduct, $[MoO_2LA]$ (**6**; X = O, A = DMF, DMSO) which exhibits two bands between 890-925 and 910-950 cm^{-1} due to the ν_{as}(O==Mo==O) and the ν_s(O==Mo==O) stretches, respectively. However, $[MoO_2L]$ exhibits only one band at 930 cm^{-1} due to the ν(Mo==O) stretch and a strong band at ~800 cm^{-1} due to the ···Mo==O···Mo==O··· stretch suggesting an oligomeric structure (**5**, X = O).



[6]

The syntheses of the oligomeric coordination compounds, $[MoO_2(LH)]$ [where LH_3 = **7;** R = $CH_2C_6H_4$(2-OH), $C_{10}H_6COOH$], $[MoO_2(L'H)]$, [here $L'H_3$ = **7**, R = C_6H_4 (2-OH)], $[MoO_2L'']$ [here $L''H_2$ = **1**; R = H, 5,6-benzo, R' = H, R'' = $CH_2C_6H_4$(2-OH)], their monomeric, six-coordinate adducts, $[MoO_2(LH)A]$, $[MoO_2(L'H)A]$, $[MoO_2L''A]$ (here A = H_2O, MeOH, py, DMSO) and monomeric, seven-coordinate heterochelates, $[MoO_2(LH)B]$ and $[MoO_2L''B]$ (B = bipy) have been reported[38,39]. The compounds/ adducts/heterochelates are soluble in MeOH, EtOH, py, DMF and DMSO but insoluble in



[7]

H_2O. They are non-electrolytes in DMF. They exhibit a band at ~930 cm^{-1} due to the ν(Mo==O) stretch and a strong band at ~770 cm^{-1} due to the ···Mo==O···Mo==O··· interaction suggesting their oligomeric structure (**5**; X = O). The ligands behave as the dibasic tridentate ONO donors in the compounds/adducts/heterochelates coordinating through their phenolic and/or carboxylic O and azomethine N atoms. B acts as a neutral bidentate NN donor ligand in the heterochelates. The alcoholic O atom of pyridoxal moiety does not take part in the coordination. An octahedral structure (**6**; X = O, A = H_2O, MeOH, py, DMSO) for the adducts and a pentagonal-bipyramidal structure (**6**; X = O, A = bipy) for the heterochelates have been suggested.

The synthesis of six-coordinate $[MoO_2(LH)(MeOH)]$ here (LH_3 = **4**) has been reported[36]. LH_3 acts as a monobasic tridentate ON donor ligand coordinating through its phenolic O, azomethine N and enolic oxygen atoms. The ν_s(O==M==O) and ν_{as}(O==M==O) stretches occur at 935 and 905 cm^{-1}, respectively. An octahedral structure has been suggested for the compound.

The syntheses of the oligomeric coordination compounds, $[MoO_2L]$ [LH_2 = **1**; R = H, 5,6-benzo, R' = H, R'' = $CH_2C_6H_4$(2-OH); R = 5,6-benzo, R' = H, R'' = C_6H_4(2-CH_2OH)] and $[MoO_2L'(MeOH)]_2{\cdot}MeOH$ [$L'H_2$ = **1**; R = H, R' = H, R'' = C_6H_4(2-CH_2OH)] have been reported[40]. $[MoO_2L]$ [R = H, R' = H, R'' = $CH_2C_6H_4$(2-OH)] is greenish-yellow and decomposes at 300 °C, while $[MoO_2L]$ [R = 5,6-benzo, R' = H, R'' = $CH_2C_6H_4$(2-OH)] is brown and decomposes at 325 °C. The compounds are soluble in py, DMF and DMSO. They are non-electrolytes in DMF (Λ_M = 3-7 mho cm^2 mol^{-1}). $[MoO_2L'(MeOH)]_2{\cdot}MeOH$ loses the coordinated and non-coordinated MeOH molecules at 170 °C and forms, $[MoO_2L']$. The latter starts decomposing at 280 °C and continues upto 580 °C. $[MoO_2L]$ exhibits only one ν(O==M==O) stretch between 918-955 cm^{-1} and a sharp broad band between 695-770 cm^{-1} due to the ν(M==O) stretch as a result of ···Mo==O···Mo==O··· interaction suggesting their oligomeric structure (**5**; X = O). The ν_{as}(O==M==O) and the ν_s(O==M==O) stretches occur at 911 and 933 cm^{-1} in $[MoO_2L'(MeOH)]_2{\cdot}MeOH$, respectively. LH_2 and $L'H_2$ behave as the dibasic tridentate ligands coordinating through their phenolic O, azomethine N and alcoholic O atoms. A distorted-octahedral geometry has been suggested for $[MoO_2L'(MeOH)]_2{\cdot}MeOH$.

Dutta *et al.*[32] have synthesized the monomeric, six-coordinate compounds, $[MoO_2(LH)(H_2O)]$ and $[MoO_2(LH)(EtOH)]$ [here LH_3 = **4**; R = (2-OH)C_6H_4CH, (2-OH)$C_{10}H_6$CH, R' = C_6H_4(2-OH), X = O]. The former compound is obtained by refluxing a MeOH solution of $[MoO_2(acac)_2]$ and LH_2 in 1:1 molar ratio, while the latter by refluxing an EtOH solution of Na_2MoO_4 and LH_2 in 1:1 molar ratio.

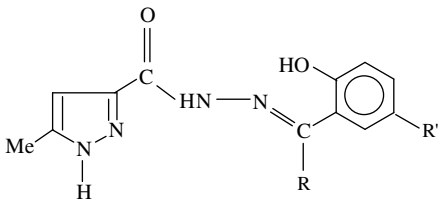
An orange, non-electrolyte, $[MoO_2(LH)(H_2O)]$ [LH_3 = **4**; R = (2-OH)C_6H_4CH, R' = C_6H_4(2-OH), X = O] is obtained[41] by the reaction of $[MoO_2(acac)_2]$ and LH_3 in 1:1 molar ratio. The compound is soluble in DMF, DMSO, MeCN, MeOH and CH_2Cl_2. The compound on crystallization from MeOH results in formation of $[MoO_2(LH)(MeOH)]$. It shows O atom transfer reaction to PPh_3.

The monomeric, brown-red/orange-red, six-coordinate compounds, $[MoO_2(LH)(H_2O)]$ [LH_3 = **4**; R = (2-OH)Y-C_6H_3CH, R' = C_6H_4(2-OH), Y = H, 5-Cl, 5-Br, 5-NO_2, 3-OMe, 3-OEt, 3,5-Cl_2, 5,6-benzo, X = O], $[MoO_2(LH)(THF)]$ [LH_3 = **4**; R = (2-OH)C_6H_4CH, R' = C_6H_4(2-OH), X = O], $[MoO_2(L'H)S]$ [$L'H_3$ (**4**; R = (2-OH)C_6H_4CH, (2-OH)$C_{10}H_6$CH, (2-OH)C_6H_4CMe, (2-

OH)$C_{10}H_6$CMe, R' = C_6H_3(2-OH)Me, S = H_2O, py, X = O] and [MoO_2(L'H)(THF)] [LH_3 = **4**; R = (2-OH)C_6H_4CH, R' = C_6H_3(2-OH)Me, X = O] have been synthesized[42]. The compounds/adducts are soluble in MeOH, EtOH, DMSO, THF and dioxane but insoluble in H_2O. They are non-electrolyte in THF (Λ_M = 0.19-8.7 mho cm^2 mol^{-1}). [MoO_2(L'H)S] (S = H_2O, py, THF) shows the weight loss between 95-150 °C due to the loss of H_2O, THF or py molecules. [MoO_2(L'H)(H_2O)] [LH_2 = **4**; R = (2-OH)C_6H_4CH, R' = C_6H_3(2-OH)Me, X = O] loses H_2O molecule between 95-100 °C. The mass loss occurs at 200-480 °C due to the decomposition of organic skeleton resulting in the formation of MoO_3 between 460-480 °C. On subsequent heating, continuous mass loss is attributed to the volatile nature of MoO_3. LH_3 behaves as a dibasic tridentate ONO donor ligand in the compounds/adducts coordinating through its phenolic O, azomethine N and enolic O atoms. The compounds exhibit the ν_s(O==M==O) and ν_{as}(O==M==O) stretches between 915-950 and 890-925 cm^{-1}, respectively. The former band gets splitted in some compounds by ~10 cm^{-1} due to the crystal packing effect. The replacement of H_2O by THF or py in [MoO_2(L'H)S] (S = H_2O, py, THF) affects the energy difference ($\Delta\nu$) between ν_s(O==M==O) and ν_{as}(O==M==O) stretches. $\Delta\nu$ is in the order: H_2O > THF > py. A monomeric structure (**6**; X = O, A = H_2O, py, THF) with planar tridentate ligands occupying meridional position is suggested for them.

Prabhakaran *et al.*[43] have observed that the energy separation ($\Delta\nu$) between ν_s(O==M==O) and ν_{as}(O==M==O) stretches in [MoO_2(LH)A] [where LH_3 = **4**; R = (2-OH)C_6H_4CMe, R' = C_6H_4(2-OH), X = O, A = H_2O, DMF, DMSO, THF or py-N-oxide] is in the order: H_2O > DMF > py-N-oxide > THF > DMSO and the thermal stability of the compounds is in the order: H_2O > py-N-oxide > DMSO > DMF > THF.

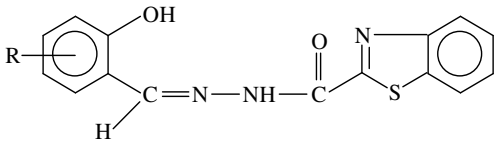
The orange-yellow compound, [MoO_2L(EtOH)] (where LH_2 = **8**; R = H, Me; R' = H, Br, NO_2) is obtained by refluxing an EtOH



[8]

solution of [MoO_2(acac)$_2$] and LH_2 in 1:1 molar ratio[44]. The compound (when R = H, R' = NO_2) is the most easily reducible by electrochemical studies. It undergoes oxo abstraction reaction at room temperature even at small concentration (~10^{-4} mol/L) resulting in the reduction of Mo(VI) to Mo(IV) during the course of oxo transfer reaction towards PPh_3.

The monomeric, non-electrolyte (Λ_M = 1.5-9.1 mho cm^2 mol^{-1} in DMF), six-coordinate compounds, [MoO_2L(MeOH)] (here LH_2 = **9**; R = H, 5,6 benzo) are obtained by refluxing a MeOH solution of [MoO_2(acac)$_2$] and LH_2 in 1:1 molar ratio for 2 *h*[45]. The coordinated MeOH molecule is lost at ~110 °C.

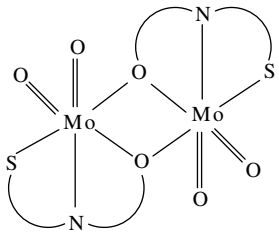


[9]

The ligand acts as a dibasic tridentate ONO donor coordinating through its phenolic O, azomethine N and enolic O atoms. The ν_s(O==M==O) and ν_{as}(O==M==O) stretches occur at 935-940 and 910 cm^{-1}, respectively. The S atom of benzothiazole moiety does not take part in coordination.

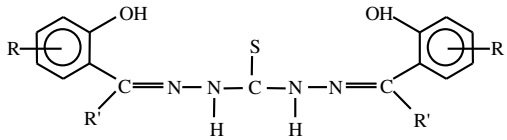
The syntheses of the coordination compounds with dibasic tridentate ONS donor ligand, LH_2 [**1**; R = H, 5-Cl, 5-Br, 5-NO_2, 5-Me, 3-C_4H_9, 5-C_4H_9, 5-OMe, 5,6-benzo, R' = H, R'' = $(CH_2)_2$SH] have been reported[26-31,37]. Their stoichiometries are [MoO_2L] and [MoO_2LA] (A = H_2O, EtOH).

The synthesis of a dimeric, six-coordinate compound, [MoO_2L]$_2$ (where LH_2 = **3**; R = H, R' = OH, R'' = 2-SH) has been reported[37]. An octahedral structure (**10**) involving phenoxo bridges has been suggested.



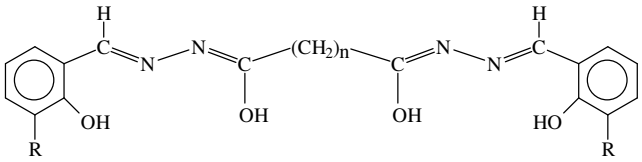
[10]

Although LH_4 (**11**; R = H, Br, NO_2, R' = H, Me) is potentially a dibasic pentadentate ONSNO donor ligand, but behaves[46] as a dibasic tridentate ONS donor ligand in orange-red, six-coordinate, non-electrolyte (in DMF), [MoO_2(LH_2)(MeOH)]. The ν(Mo—O) and ν(Mo—N) stretches appear at ~595 and between 510-560 cm^{-1}, respectively.

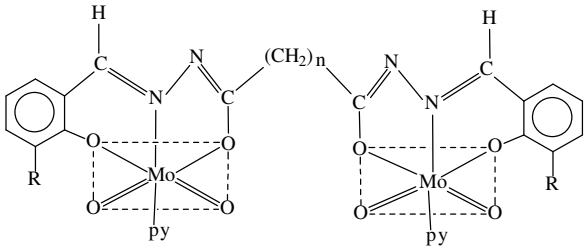


[11]

The dimeric, six-coordinate compounds, [(MoO_2py)$_2$L] (where LH_4 = **12**; R = H, OMe, n = 0, 1, 2, 4, 8) have been synthesized[47]. The py molecules are lost between 150-200 °C. An octahedral structure (**13**; R = H, OMe, n = 0, 1, 2, 4, 8) has been suggested to these compounds.

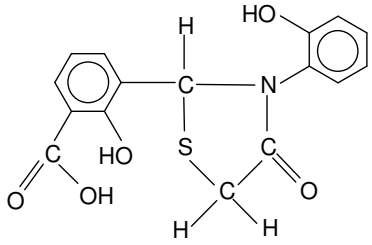


[12]



[13]

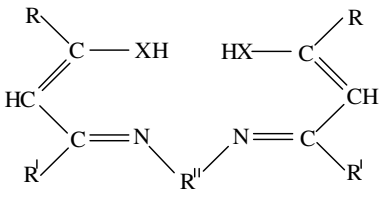
The synthesis of a six coordinate, non-electrolyte (Λ_M = 6.2 mho cm^2 mol^{-1} in DMF), $[MoO_2(LH)(MeOH)]$ (here LH_3 = **14**) has been reported[48]. The compound is soluble in water, DMSO, DMF, partially soluble in MeOH and EtOH. The dibasic tridentate ligand coordinates through its phenolic O, carboxylic O and S atoms. The ν_s(O==Mo==O) and the ν_{as}(O==Mo==O) stretches occur at 946 and other 910 cm^{-1}, respectively.



[14]

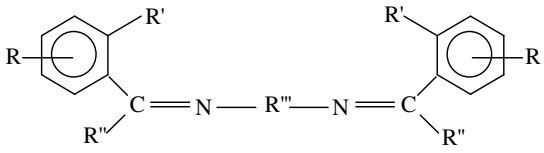
D. With tetradentate ligands:

Dey *et al.*[29] have synthesized the monomeric, six-coordinate compounds, $[MoO_2L]$ [where LH_2 = **15**; R = R' = Me, R'' = C_2H_4, 1,2-C_6H_4, X = O]. The ligands act as the dibasic tetradentate ONNO donors.



[15]

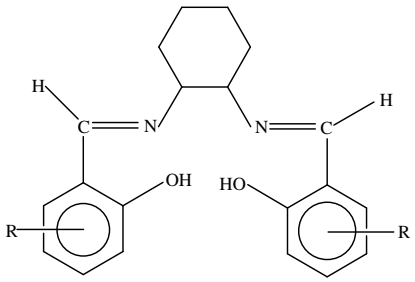
Yamanouchi *et al.*[21] have synthesized the six-coordinate compounds, $[MoO_2L]$ [here LH_2 = **16**; R = H, 3-OMe, R' = OH, R'' = H, Me, R''' = C_2H_4, $(CH_2)_3$, $(CH_2)_4$, $(CH_2)_6$, CH(Me)CH_2, $(CH_2)_2NH(CH_2)_2$, 1,2-C_6H_4].



[16]

Kudryavstev *et al.*[49] have synthesized the monomeric, six-coordinate compounds, $[MoO_2L]$ (here LH_2 = **16**; R = H, 4-Me, 5-Br, R' = OH, R'' = H, R''' = C_2H_4) by the reaction of an ethereal solution of MoO_2Cl_2 and the LH_2 in 1:1 molar ratio. The latter acts as a dibasic tetradentate ONNO donor ligand.

Ambroziak *et al.*[50] have reported the syntheses of $[MoO_2L]$ [where LH_2 = **17**; R = H, 3,5-Cl_2, 3,5-Br_2, 5-NO_2, 4,6-$(OMe)_2$]. The compounds are fairly soluble in $HCCl_2CCl_2H$ and



[17]

DMSO, while slightly soluble in organic solvents. They exhibit two bands one at 928 cm^{-1} and other at 872 cm^{-1} due to the ν_s(O==Mo==O) and the ν_{as}(O==Mo==O) stretches, respectively. The presence of electron donor substituents in the salicylidene ring strengthen the Mo−−N bond and increases the stability of the compound, while the electron withdrawing groups decrease their stability.

Kudryavstev *et al.*[49] have synthesized the monomeric, eight-coordinate compounds, $[MoO_2Cl_2L)]$ (here L = **16**; R = R'' = H, R' = NH_2, R''' = C_2H_4, 1,2-C_6H_4) by the reaction of an ethereal solution of MoO_2Cl_2 and L in 1:1 molar ratio. The latter acts as the neutral tetradentate N_4 donor ligand.

E. With polydentate ligands:

The syntheses of binuclear coordination compound, [(MoO2)2L] (where LH4 = flexibly-bridged tetrabasic hexadentate ligand obtained from the condensation of methylene or dithiobis(salicylaldehyde) with 2-aminoethanol, 2-amino-2-methylpropanol, 1-amino-2-propanol, 2-aminophenol, S-methyldithiocarbazate and S-benzyldithio- carbazate) have been reported51,52. Each MoO2(VI) ion achieves a pseudo-octahedral oligomeric structure via Mo==O···Mo bridging. [(MoO2)2L] reacts with py and forms the corresponding adduct, [MoO2)2L(py)2]. Here py binds at the sixth labile sites in both [MoO2]2+ units. The oligomeric coordination compounds show the ν(Mo==O) stretches between 910-938 cm-1 and the ν(Mo==O) band at ~820 cm−1 due to Mo==O□□□Mo

interaction. In a single endothermic step at 180°C, [(MoO2)2L(py)2] loses py and gets converted to [(MoO2)2L].

CONCLUSION

The ligands and their coordination compounds presented in this paper provide a brief account of the synthesis, structure, spectral measurement and study of biological activity of some oxomolybdenum(VI) compounds. A comparative antimicrobial study of the ligands and their compounds indicated that the chelation might be helpful in therapeutic potential of a drug and these compounds can work as the potential lead molecule for drug designing.

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