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# The Synthesis and Characterization of an Oxymolybdenum(V) Complex of 1,3-Bis(methylseleno)propane

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Recent interest in the chemical properties [1, 2], biological functions [3–5] and spectroscopic aspects [6–8] of selenium has motivated us to report here the preparation and characterization of a six-coordinate molybdenum(V) complex obtained from the reaction of $MoOCl_3$ or $MoOCl_3(THF)_2$ (THF = tetrahydrofuran) with 1,3-bis(methylseleno)propane, L. Although a few studies of nickel(II) and palladium(II) [9–11] complexes of selenoethers have been reported, there are to our knowledge no molybdenum complexes with Se donors known.

## Experimental

All solvents were distilled under dinitrogen prior to use and all manipulations were performed under an inert atmosphere. The ligand, $MeS(CH_2)_3SMe$, was synthesized as previously described [11]. Vacuum line and Schlenk techniques were used in complex preparations; $MoOCl_3(THF)_2$ was prepared as previously described [12].

The complex was isolable from reaction of either $MoOCl_3$ or $MoOCl_3(THF)_2$; here we give details of the latter preparative route: the ligand $MeSe(CH_2)_3SeMe$ (0.46 g, 2.0 mmol) and $MoOCl_3(THF)_2$ (0.75 g, 2.1 mmol) were stirred in a 3:1 v/v mixture of toluene/dichloromethane (75 $cm^3$) in a rotoflow reactor for 24 h. After filtration to remove traces of unreacted $MoOCl_3(THF)_2$ the solution was concentrated to 20 $cm^3$ by application of a vacuum. On cooling in an ice bath for 3 h a fine green precipitate was deposited. This was filtered, washed with n-hexane (3 × 10 $cm^3$) and dried *in vacuo* for 3 h. Yield: 48%. *Anal.* Found: Mo, 21.1; Cl, 23.7; C, 13.6; H, 3.1%. $MoOCl_3C_5H_{12}Se_2$ requires: Mo, 21.4; Cl, 23.7; C, 13.4; H, 3.0%. Physical methods are as previously described [13].

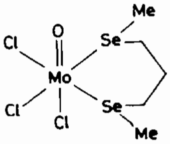
## Results and Discussion

The $MoOLCl_3$ (L = $CH_3Se(CH_2)_3SeCH_3$) complex is preparable from the reaction of either $MoOCl_3$ or $MoOCl_3(THF)_2$ with the ligand in a mixed toluene/dichloromethane solvent. We were somewhat surprised to discover that both these salts reacted with the ligand at approximately the same rate and that yields of $MoOLCl_3$ were almost identical for the two reactions. We have previously observed that $MoOCl_3(THF)_2$ is preferable to the insoluble polymeric $[MoOCl_3]_n$ salt [14].

The green $MoOLCl_3$ is quite stable in all but very moist air; its room temperature magnetic moment, 1.72 $\mu_B$, is consistent with values previously reported for molybdenum(V) complexes [15]. The mull infrared spectrum of the complex exhibits a sharp $\nu$(Mo=O) band at 950 $cm^{-1}$, indicating the presence of a terminal Mo=O linkage [15]. Moreover, the energy of this band is consistent with the complex being hexacoordinate; it is known that in pentacoordinate complexes this band appears *ca.* 1000 $cm^{-1}$ [16]. The presence of only one $\nu$(M=O) band suggests that only one isomer is present [13]. A prominent band at 330 $cm^{-1}$ is assignable to $\nu$(Mo–Cl), the width of its suggesting that it may be due to *cis* chlorines. An ill-defined shoulder on this main absorption may be due to the chlorine *trans* to the Mo=O group, this Mo–Cl bond being lengthened by *trans* influence of the molybdenyl group [15]. An absorption at 595 $cm^{-1}$ in the free ligand appears at 590 $cm^{-1}$ in the complex and is identified as $\nu$(C–Se) and $\rho(CH_3)$ is present *ca.* 970 $cm^{-1}$ as a weak absorption in the complex, shifted from 985 $cm^{-1}$ in the free ligand.

The room temperature e.p.r. spectrum of $MoOLCl_3$ in dichloromethane has one band at g = 1.953, A = 52G, consistent with the presence of molybdenum(V). Selenium coupling is not observed, suggesting that $^{77}Se$ < 30G. Some poorly resolved fine structure is observed when the spectra are recorded in the double derivative mode; this is a septet of relative line intensities 1:2:3:4:2:1, with a splitting of 2.5 G, consistent with two chlorines being in a similar environment.

On the basis of this evidence the complex $[MoOLCl_3]$ is assigned the hexacoordinate *fac* structure shown.



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