The Crystal Structure of Dioxobis-8-hydroxyquinolinatomolybdenum(VI)

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Summary *X*-Ray crystal studies of $MoO_2(C_9H_6NO)_2$ reveal that the O–Mo–O group has the *cis*-configuration.

LARSON and MOORE, considering dioxo-compounds of Mo^{VI}, concluded that the O–Mo–O group in $MoO_2(C_9H_6NO)_2$ must be linear.[1] This is not consistent with the results of numerous i.r. and *X*-ray investigations on dioxo-compounds of Mo^{VI}.[2] We have therefore carried out an *X*-ray study of $MoO_2(C_9H_6NO)_2$ crystals and obtained the following crystal data: $a = 13{\cdot}51$, $b = 9{\cdot}52$, $c = 13{\cdot}62$ Å, $\beta = 110°$; space group C_2/c, $Z = 4$, $D_0 = 1{\cdot}637$, $D_c = 1{\cdot}639$ g./cm.^{3}, $U = 1646$ Å^3.

The experiment was performed with the equi-inclination Weissenberg goniometer, unfiltered Cu-K_α radiation being used. Intensities of 760 independent non-zero reflections were estimated visually, disregarding absorption corrections ($\mu = 70{\cdot}7$ cm.$^{-1}$).

The structure was solved by the standard heavy-atom method and refined by the isotropic full-matrix least-squares method to $R = 0{\cdot}113$, with the mean overall temperature factor $B = 1{\cdot}7$ Å^2. The maximum standard deviation for interatomic distances is 0·03 Å and for angles 1·9°.

The molecular configuration and some significant bond lengths and angles are shown in the Figure.

As is the case for all molybdenum compounds containing terminal oxygen atoms, the bonds *trans* to the oxygen atoms are essentially longer. The Mo–N and Mo–O bond lengths are in good agreement with those in MoO_3-dien (2·32 Å and 1·71 Å, respectively).[3]

The crystals of $MoO_2(C_9H_6NO)_2$ are isostructural with $TiCl_2(C_9H_6NO)_2$.[4] In the latter compound, the Ti–N bond also is much longer (2·20 Å). On the other hand, in complexes with quinoline groups located in one plane: $Zn(C_9H_6NO)_2,2H_2O$,[5] α- and β-$Cu(C_9H_6NO)_2$,[6,7] and $Pd(C_9H_6NO)_2$,[8] the M–N bond lengths (2·10—1·97 Å) are only slightly different from M–O bond lengths (2·07—1·92 Å).

Structural and (in part) spectral studies of recent years reveal a distinct *trans*-effect in compounds of V, Nb, Ta, Mo, W, and Re in the presence of metal–ligand multiple bonds, oxygen and nitrogen atoms being regarded as *trans*-active ligands. It follows from the above data that p_π–d_π donor interaction favours labilization of a *trans*-partner.[2]

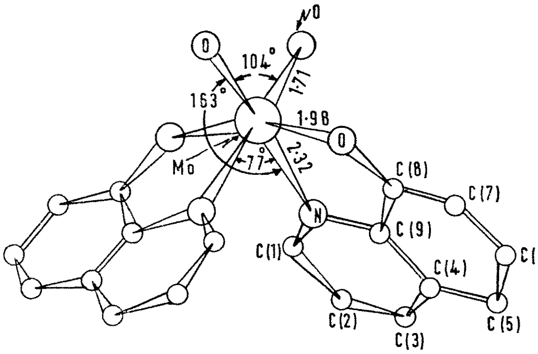

FIGURE. *Configuration of* $MoO_2(C_9H_6NO)_2$.

The Ti–Cl bond length, 2·283 ± 0·016 Å, in $TiCl_2(C_9H_6NO)_2$ is shorter than that in K_2TiCl_2[9] (2·35 ± 0·03 Å), which is an indication of a partial multiple bond. Comparison of the Ti–N and Mo–N bond lengths in two isostructural compounds suggests that labilization of these bonds has one and the same cause.

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