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Structure and Bonding in Octaisopropoxydimolybdenum(IV).

by M. H. Chisholm,[1] F. A. Cotton,[2] M. W. Extine[2]
and W. W. Reichert[1]

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Departments of Chemistry

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Structure and Bonding in Octaisopropoxydimolybdenum(IV).

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ABSTRACT

The title compound was prepared by the action of isopropanol on tetra(dimethylamido)molybdenum(IV). X-ray crystallography has shown that it is a dinuclear $(Pr^iO)_3Mo(\mu\text{-}Pr^iO)_2Mo(OPr^i)_3$ molecule which has a rigorous crystallographic center of inversion and approximate C_{2h} symmetry. The bridges are unsymmetrical with Mo-O distances of 1.958(3)Å and 2.111(3)Å. The configuration of oxygen atoms about each metal atom is a slightly distorted trigonal bipyramid. The Mo-Mo distance, 2.523(1)Å, is consistent with the existence of a double bond between the metal atoms. The crystallographic parameters are: Space group, $P2_1/n$; a = 9.902(2)Å; b = 17.867(3)Å; c = 9.725(2)Å, β = 102.89(1)°; V = 1677.2(9)Å^3; Z = 2. The structure was refined employing anisotropic thermal parameters for all atoms except hydrogen atoms, which were omitted altogether, to R_1 = 0.040 and R_2 = 0.068. The question of whether the Mo-Mo bond is in fact a double bond is discussed, and it is shown that from the distance alone it is not possible to decide conclusively between a double bond or a single bond accompanied by coupling of one pair of electrons through the bridge system. The possibility of there being an unusual type of double bond consisting of a π and a δ component is outlined.

INTRODUCTION

It is well established[2,3] that dialkylamidometal compounds react readily with alcohols according to the general equation

$$M_x(NR_2)_y + yR'OH \rightarrow M_x(OR)_y + yHNR_2$$

It is also well known[2,3] that metal alkoxides tend to be oligomers as a result of OR groups serving as bridges. On the basis of this background, it was therefore to be expected that the following reaction, employing the well-characterized $Mo(NMe_2)_4$[4] as starting material, would proceed.

$$nMo(NMe_2)_4 + 4n\ ROH \rightarrow [Mo(OR)_4]_n + 4nHNMe_2$$

It has recently been shown[5] that it does, and in the case of R = $CHMe_2$ the value of n was indicated to be 2. Since the compound $Mo_2(OPr^i)_8$ is also diamagnetic, it was clearly of interest to investigate its structure to determine if the diamagnetism can be attributed to the existence of a metal-metal bond. We report here such an investigation.

EXPERIMENTAL

The compound was prepared as described elsewhere.[5] All manipulations of the compound were performed in an inert atmosphere.

A crystal measuring approximately 0.4 x 0.4 x 0.6 m was wedged in a thin-walled glass capillary under N_2 and mounted with its longest dimension nearly coincident with the phi axis. ω-scans of several intense low angle reflections had peak widths at half-height of ca. 0.2°. Cell constants and axial photographs showed that the crystal belonged to the monoclinic system with a = 9.902(2)Å, b = 17.867(3)Å, c = 9.725(2)Å, β = 102.89(1)°, and

$V = 1677.2(9)\text{Å}^3$. The volume is consistent with that expected for $Z = 2$.

Data were collected[6] at 23°C using a Syntex $P\bar{1}$ autodiffractometer and MoKα ($\lambda = 0.710730\text{Å}$) radiation monochromatized with a graphite crystal in the incident beam. Symmetrical θ-2θ scans ranging from 1.0° above $K\alpha_1$ to 1.0° below $K\alpha_2$, variable scan speeds ranging from 4.0 to 24.0°/min, and a background to scan-time ratio of 0.5 were employed. The intensities of three standard reflections were monitored frequently throughout data collection and showed an average overall decrease of 11%. A total of 2269 data having $0° < 2\theta(\text{MoK}\alpha) < 45°$ were collected. The data were reduced to a set of relative $|F_o|^2$ values and corrected for crystal decay. An absorption correction was not deemed necessary ($\mu = 8\ \text{cm}^{-1}$). The 1826 unique data having $|F_o|^2 > 3\sigma|F_o|^2$ were used to solve and refine the structure.

Systematic absences observed during data collection uniquely determined the space group to be $P2_1/n$, a non-standard setting of $P2_1/c$ (No. 14). The structure was solved[6] using standard heavy-atom techniques and refined to convergence using anisotropic thermal parameters for the 17 non-hydrogen atoms. Final residuals were

$$R_1 = \Sigma||F_o|-|F_c||/\Sigma|F_o| = 0.040$$
$$R_2 = [\Sigma w(|F_o|-|F_c|)^2/\Sigma w|F_o|^2]^{1/2} = 0.068.$$

The esd of an observation of unit weight was 1.66. A value of 0.07 was used for p in the calculation of the weights.[6] A final difference Fourier map revealed no chemically significant peaks. A table of observed and calculated structure factors (8 pages) is available as supplementary material. See any current masthead page for ordering information.

RESULTS AND DISCUSSION

Description of the Structure. The compound crystallizes in the monoclinic space group $P2_1/n$, with two molecules in the unit cell. The molecules therefore reside on inversion centers. Table I lists the atomic positional and thermal parameters.

The Mo_2O_8 portion of the molecule has essentially C_{2h} symmetry although the orientations of the $CHMe_2$ groups destroy the plane of symmetry, as can be seen in Fig. 1. The bond distances and angles are listed in Table II.

The Mo_2O_8 central portion of the structure can be viewed as two MoO_5 trigonal bipyramids joined along a common axial-equatorial edge. This is clearly seen in Fig. 2A. The three equatorial bonds make almost perfect (120°) trigonal angles, the actual values being 120.9(1)°, 120.2(2)° and 118.9(2)° and the MoO_3 unit is planar within experimental error. The axial O-Mo-O unit is slightly bent, 173.1(1)°, and is also slightly (ca. 5°) off of perpendicularity to the equatorial plane.

The bridging system is distinctly unsymmetrical, the two Mo-O distances differing by 0.15Å. However, at least part of this may be due to the fact that one bridge bond is to an equatorial position and the other to an axial position of a trigonal bipyramid, and even the terminal bonds to these two types of position differ by about 0.10Å.

The Metal-Metal Bond. The very short Mo-Mo distance of 2.523(1)Å together with the acute angles, 76.5(1)°, at the bridging oxygen atoms and the obtuse angles, 103.5(1)°, at the Mo atoms argue irrefutably for a direct bond between the metal atoms. The structural evidence in favor of the Mo-Mo bond is cogently presented in Fig. 2 where the $Mo_2(OPr)_8$ structure is contrasted directly with that of $Mo_2(OPr)_6(NO)_2$[7] in which there is no Mo-Mo bond and hence a net repul-

sive interaction between the metal atoms.

It is well known that the lengths of Mo-Mo single bonds vary greatly[8] depending upon formal oxidation number and the character of the ligands present, and also that when bridging groups are present it is not possible unequivocally to distinguish between direct coupling of electron spins (M-M bonding) and indirect coupling through the bridging ligands. Nevertheless, it seems reasonable to suggest that in this compound we are dealing with a double bond between the molybdenum atoms. Given that there is an Mo-Mo bond of some type (which the structural characteristics demand) and assuming, for simplicity, that only integral bond orders need be considered, the only possibilities are 1 and 2 since we are dealing with molybdenum atoms in the formal oxidation state +4. If we assume a bond order of 1 we have to postulate coupling of the remaining electron spins through the bridge system, whereas a bond order of 2 directly accounts for the lack of unpaired electrons.

The Mo-Mo distance cannot be used as evidence in determining the bond order unless careful attention is given to the details of the system of bridging ligands in this and any compound with which it is compared. Even then, such an argument is far from conclusive with the evidence currently available. It is true that most Mo-Mo single bonds previously reported[8] are longer (>2.6Å) than the Mo-Mo distance in the present case. It is also true that at least one compound, namely $Mo_2(OBu^t)_6(CO)$, r(Mo-Mo) = 2.498(1)Å[9] (and perhaps a second compound, MoO_2 with r(Mo-Mo) = 2.511Å[10]) that probably has a double bond, has a Mo-Mo bond length similar to that in the present compound. These facts are consistent with the assignment of a bond order of 2 in the present case, but do not require it. One weakness in the argument is that the presence of Mo=Mo bonds in $Mo_2(OBu^t)_6(CO)$ and particularly in MoO_2 is not absolutely certain.

It is even more important, however, that there are several cases in which compounds that cannot have Mo-Mo bond orders greater than 1 have Mo-Mo distances comparable to the present one. Thus, we have the Mo_3O_{13} unit in $Zn_2Mo_3O_8$, for which the Mo-Mo distance is 2.524Å[11] and the structurally similar $[Mo_3O_4(C_2O_4)_3(H_2O)_3]^{2-}$ ion[12] where the distance is 2.486Å. In these Mo^{IV} compounds there are equilateral triangles of molybdenum atoms and it is reasonable to believe that each molybdenum atom forms to single bonds to its neighbors, but in any case there are not enough electrons available to form bonds of any greater order than 1. It can certainly be argued that the different arrangement of bridging oxygen atoms in the trinuclear species, particularly the presence of one oxygen atom that is symmetrically bound to all three metal atoms, causes a close approach of the metal atoms to one another. However, a consideration of these compounds drives home the point that no conclusion can be drawn about the bond order simply from the distance.

We are, however, inclined to believe that there is actually a direct double bond. The concept of only an Mo-Mo single bond with the remaining two electrons coupled through the bridging system is disfavored by the fact that the configurations at the bridging oxygen atoms are distinctly pyramidal, whereas good spin coupling would presumably be possible only with a planar configuration. The nature of such a double bond is dependent upon the structural properties of this molecule. It is instructive to analyze this aspect of the problem by contrasting the $Mo_2(OPr^i)_8$ molecule with the $Mo_2(OPr^i)_6(NO)_2$ molecule, since there is a trigonal bipyramidal arrangement of ligands about the metal atoms in both compounds.

A trigonal bipyramidal field splits the metal d orbitals into three sets $e'(D_{x^2-y^2}, d_{xy})$, $e''(d_{xz}, d_{yz})$ and $a'(d_{z^2})$ with the d_{xz},d_{yz} degenerate pair

lying lowest in energy. In the nitrosyl, each Mo atom may be assumed, formally, to have four 4d electrons after the formation of σ bonds to each of the five ligands, provided we also use the conventional though purely formal description of the linear Mo-N-O moiety as $\bar{M}o$-(NO^+). These four electrons should then fill up the $e''(d_{xz},d_{yz})$ orbitals, where they can participate very effectively in backbonding to the NO, thus explaining the very low (1632 cm^{-1}) value of ν_{NO} and the absence of an Mo-Mo bond. In $Mo_2(OPr^i)_8$, where the formal oxidation number of Mo is +2, each Mo atom has two 4d electrons. It is possible to envision the formation of a double bond as the result of d_{xz}-d_{xz} and d_{yz}-d_{yz} overlaps. This could be construed as a combination of one π bond and one δ bond, but whether the lower symmetry that actually exists will materially alter such a formal description is problematic. In any event, in both compounds the molybdenum atoms have 14-electron valence shell configurations. If the double bond in $Mo_2(OPr^i)_8$ does consist of this rather unusual combination of a π and a δ combination instead of the conventional σ + π pair, this might explain why it is relatively long since, in general, δ components of multiple bonds are always much less effective than σ ones.

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Table I. POSITIONAL AND THERMAL PARAMETERS AND THEIR ESTIMATED STANDARD DEVIATIONS.

ATOM	X	Y	Z	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)
Mo	0.01279(5)	0.06291(3)	0.06040(5)	2.57(2)	2.32(2)	2.72(2)	-0.07(2)	0.58(2)	-0.06(2)
O(1)	0.0269(4)	0.0441(2)	-0.1343(4)	2.9(1)	2.8(2)	2.9(1)	-0.3(1)	0.8(1)	0.2(1)
O(2)	0.1705(4)	0.0614(2)	0.2083(4)	3.5(2)	3.3(2)	3.5(2)	-0.5(1)	-0.1(2)	-0.4(1)
O(3)	0.0501(4)	0.1675(2)	0.0146(4)	4.6(2)	2.5(2)	3.9(2)	-0.6(2)	1.1(1)	-0.2(2)
O(4)	-0.1610(4)	0.0821(2)	0.1019(5)	3.6(2)	3.5(2)	5.4(2)	0.6(2)	1.8(1)	0.2(2)
C(1)	0.1153(7)	0.0677(3)	-0.2279(7)	4.5(3)	3.7(3)	3.8(3)	-0.0(2)	2.0(2)	0.9(2)
C(2)	0.0510(8)	0.1349(4)	-0.3089(8)	6.5(4)	4.7(3)	4.8(3)	0.5(3)	1.6(3)	2.6(3)
C(3)	0.2653(7)	0.0834(4)	-0.1423(8)	3.4(3)	5.9(4)	6.3(3)	-0.9(3)	1.8(2)	0.2(3)
C(4)	0.2491(7)	0.0041(4)	0.2916(6)	3.6(3)	4.3(3)	3.6(3)	-0.1(3)	-0.6(2)	0.5(3)
C(5)	0.2331(9)	0.0134(5)	0.4438(7)	6.9(4)	8.5(5)	3.9(3)	0.2(4)	0.6(3)	1.0(4)
C(6)	0.4001(8)	0.0152(5)	0.2799(9)	3.6(3)	5.8(4)	7.5(4)	0.1(3)	0.3(3)	0.5(4)
C(7)	0.0972(8)	0.2232(4)	0.1190(8)	6.9(4)	2.7(3)	5.9(4)	-1.3(3)	1.1(3)	-0.8(3)
C(8)	0.0268(10)	0.2970(5)	0.0607(10)	10.3(6)	3.1(4)	9.5(6)	0.1(4)	2.0(5)	-0.6(3)
C(9)	0.2545(9)	0.2317(5)	0.1447(11)	6.1(4)	6.0(4)	9.3(5)	-3.0(3)	-0.2(4)	-0.8(4)
C(10)	-0.2292(7)	0.1526(4)	0.1115(8)	4.6(3)	3.7(3)	6.0(3)	1.9(2)	1.7(2)	-0.0(3)
C(11)	-0.3765(10)	0.1467(6)	0.0167(13)	7.2(5)	7.9(5)	11.8(7)	3.7(4)	-2.3(5)	-1.3(5)
C(12)	-0.2447(12)	0.1630(6)	0.2615(10)	17.1(7)	9.8(5)	7.4(5)	8.0(4)	5.0(4)	0.7(4)

The form of the anisotropic thermal parameter is:

$$\exp[-1/4(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$$

Table II. Bond Distances (Å) and Angles (Deg).[a]

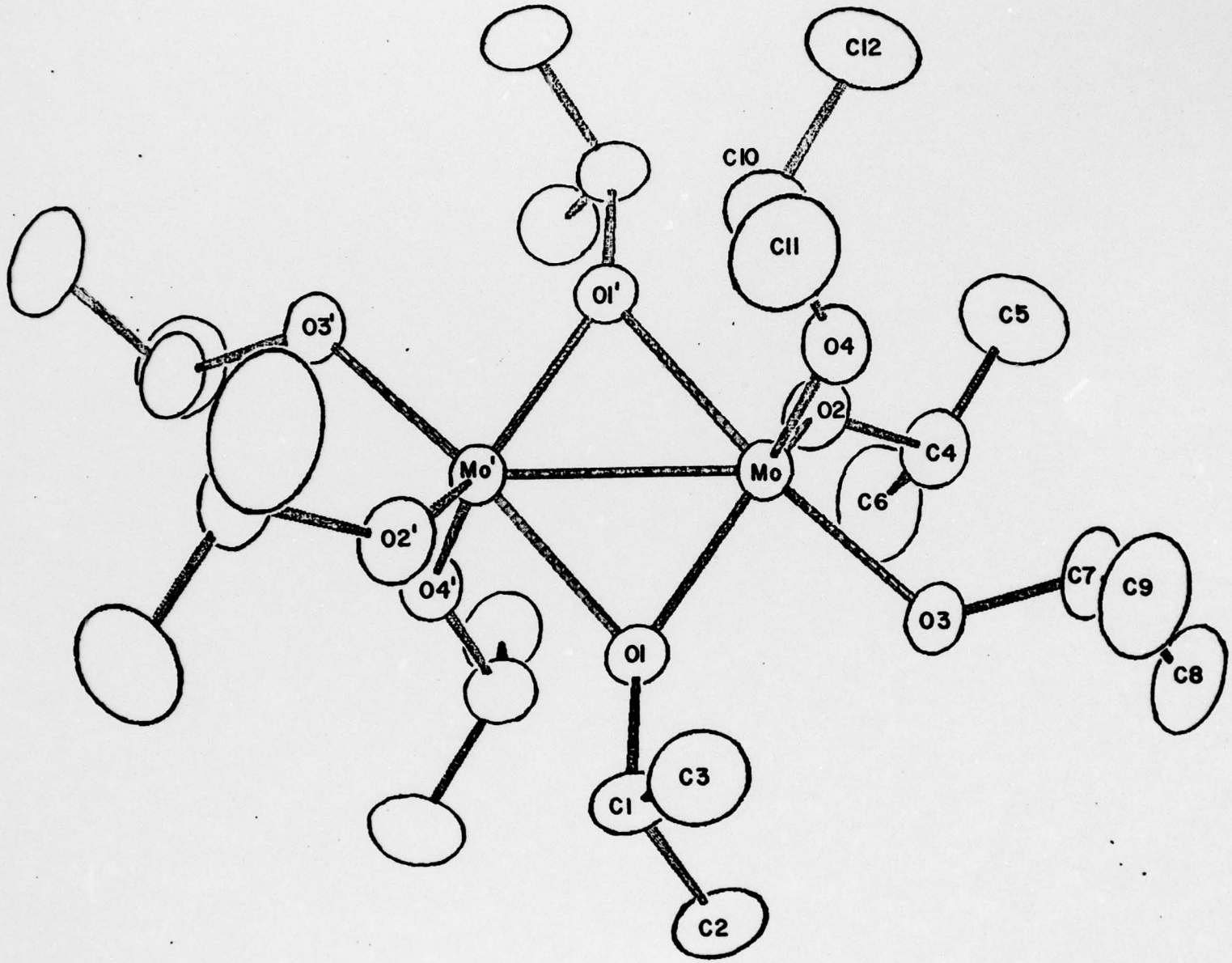
Atoms	Atoms	Distance
Mo	Mo	2.523(1)
Mo	O(1)	1.958(3)
Mo	O(1)'	2.111(3)
Mo	O(2)	1.872(3)
Mo	O(3)	1.976(3)
Mo	O(4)	1.884(3)
O(1)	C(1)	1.460(6)
O(2)	C(4)	1.424(6)
O(3)	C(7)	1.424(6)
O(4)	C(10)	1.443(6)
C(1)	C(2)	1.498(8)
C(1)	C(3)	1.558(8)
C(4)	C(5)	1.533(8)
C(4)	C(6)	1.538(8)
C(7)	C(8)	1.538(9)
C(7)	C(9)	1.529(9)
C(10)	C(11)	1.545(9)
C(10)	C(12)	1.512(10)

Atoms			Angle
Mo'	Mo	O(1)	54.45(9)
Mo'	Mo	O(1)'	49.00(9)
Mo'	Mo	O(2)	108.9(1)
Mo'	Mo	O(3)	137.9(1)
Mo'	Mo	O(4)	105.1(1)
O(1)	Mo	O(1)'	103.5(1)
O(1)	Mo	O(2)	120.9(1)

Atoms			Angle
O(1)	Mo	O(3)	83.5(1)
O(1)	Mo	O(4)	120.2(2)
O(1)'	Mo	O(4)	84.9(1)
O(1)'	Mo	O(3)	173.1(1)
O(1)	Mo	O(4)	81.0(1)
O(2)	Mo	O(3)	91.2(1)
O(2)	Mo	O(4)	118.9(2)
O(3)	Mo	O(4)	95.8(2)
Mo	O(1)	Mo'	76.5(1)
Mo	O(1)	C(1)	137.4(3)
Mo'	O(1)	C(1)	131.1(3)
Mo	O(2)	C(4)	134.7(3)
Mo	O(3)	C(7)	123.3(3)
Mo	O(4)	C(10)	129.5(3)
O(1)	C(1)	C(2)	108.4(5)
O(1)	C(1)	C(3)	110.6(5)
C(2)	C(1)	C(3)	112.4(5)
O(2)	C(4)	C(5)	108.1(5)
O(2)	C(4)	C(6)	106.3(4)
C(5)	C(4)	C(6)	111.7(5)
O(3)	C(7)	C(8)	106.7(5)
O(3)	C(7)	C(9)	110.2(5)
C(8)	C(7)	C(9)	109.7(6)
O(4)	C(10)	C(11)	107.2(5)
O(4)	C(10)	C(12)	108.6(5)
C(11)	C(10)	C(12)	107.4(7)

[a]Figures in parentheses are estimated standard deviations in the least significant digits.

Figure 1. A view of the $Mo_2(OCHMe_2)_8$ molecule using 40% probability ellipsoids to represent the atoms and showing the atom labelling scheme. The molecule has rigorous C_i symmetry.


C12
C10
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O1'
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C4
Mo'
Mo
C6
O2'
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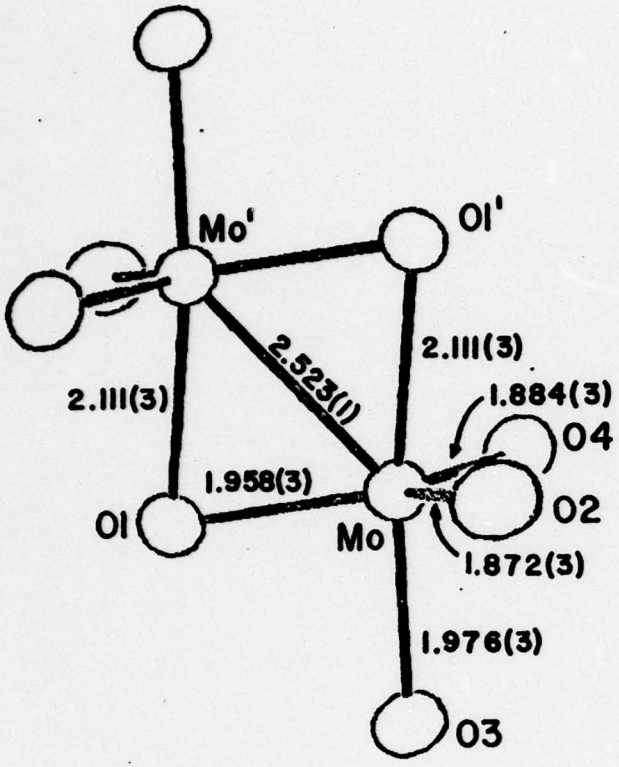
C7
C9
O3
O1
C8
C3
C1
C2

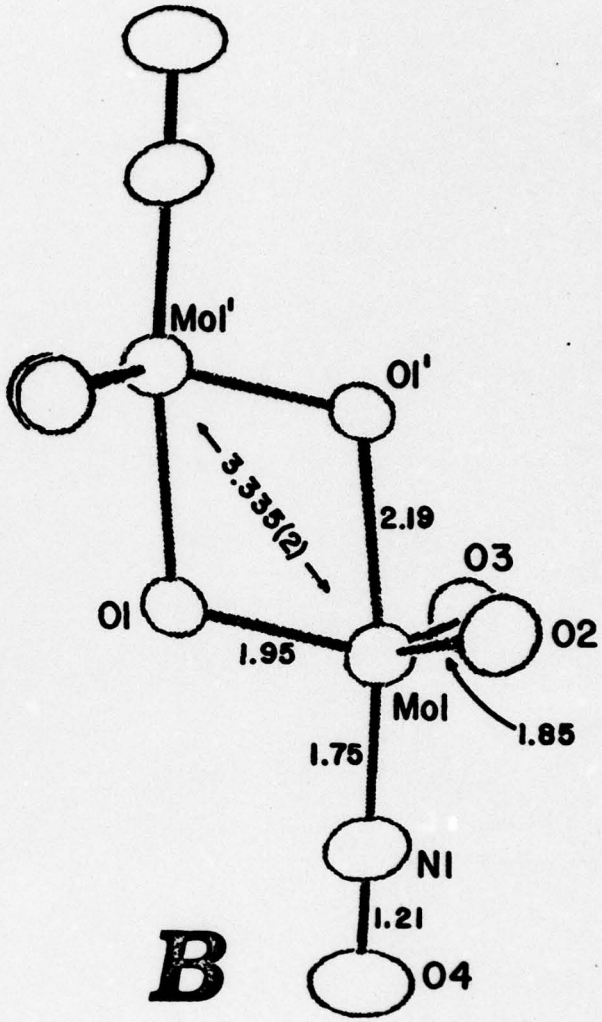
Figure 2. A comparison of the coordination geometries in $Mo_2(OPr^i)_8$ and $Mo_2(OPr^i)_6(NO)_2$ showing some pertinent bond distances. Distances shown for B are averaged over two independent molecules. In both A and B the molecules possess rigorous C_i and virtual C_{2h} symmetry.



A

$Mo_2(O\text{-}i\text{-}Pr)_8$

Skeleton



B

$Mo_2(O\text{-}i\text{-}Pr)_6(NO)_2$

Skeleton

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