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OCTA(ISOPROPOXY)DIMOLYBDENUM AND BIS(NITROSYL)HEXA(ISOPROPOXY)D--ETC(U)

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(6) Octa(isopropoxy)dimolybdenum and Bis(nitrosyl)hexa (isopropoxy)dimolybdenum. Structure and Bonding in Compounds Containing Molybdenum Atoms with Fourteen Valence Shell Electronic Configurations.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The compounds $Mo_2(OPr^i)_6(NO)_2$, I, and $Mo_2(OPr^i)_8$, II have been structurally characterized. Each compound has rigorous inversion symmetry and virtual C_{2v} symmetry and in each there is essentially trigonal bipyramidal coordination about each molybdenum atom. There is a central planar $Mo_2(\mu\text{-}O)_2$ moiety involving bridging isopropoxy ligands which form alternately long (axial) and short (equatorial) Mo-O bonds. The most striking differences

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered) between the two structures are (i) the Mo-to-Mo distances which are 3.335(2) and 2.525(1)Å, for I and II, respectively, and (ii) the angles of the $Mo_2(\mu\text{-}O)_2$ moiety. These differences are rationalized in terms of simple ligand field considerations. In compound I, which contains molybdenum in a formal oxidation state of +2, there are four electrons in d_{xz}, d_{yz} atomic orbitals which are extensively involved in back bonding to the NO^+ ligand. In compound II, which contains molybdenum in a formal oxidation state of +4, there are only two electrons in the d_{xz}, d_{yz} atomic orbitals. These electrons then form a Mo-Mo double bond; this accounts for the short Mo-to-Mo bond distance and the diamagnetic nature of compound II.

Octa(isopropoxy)dimolybdenum and Bis(nitrosyl)hexa(isopropoxy)dimolybdenum. Structure and Bonding in Compounds Containing Molybdenum Atoms with Fourteen Valence Shell Electronic Configurations.

Sir:

The occurrence of compounds containing metal-to-metal bonds of order 4,3,2 and 1 is now well recognized.[1] As yet, however, the systematic manner in which C-C bond order may be changed in organic chemistry has no parallel in transition metal chemistry. The products of addition/elimination reactions involving dinuclear compounds appear to be unpredictable, although a subsequent rationale may usually be advanced with judicious hindsight. We report here the structural characterization of two closely related and yet contrasting compounds, namely, $Mo_2(OPr^i)_6(NO)_2$, I, and $Mo_2(OPr^i)_8$, II. Each one may be considered formally as an addition product of $Mo_2(OPr^i)_6$, a compound containing a metal-to-metal triple bond.[2] Compound I is indeed directly formed by the addition of NO (2 equiv) to $Mo_2(OPr^i)_6$.[3] $Mo_2(OPr^i)_8$ is best prepared from the reaction between $Mo(NMe_2)_4$ and Pr^iOH.[4] The addition of 2 NO causes cleavage of the M-M triple bond, while addition of $2Pr^iO\cdot$ (a hypothetical reaction) transforms an M-M triple bond to an M-M double bond.[5]

Figure 1 shows the essential features of the coordination geometry in each molecule. Pertinent bond distances and angles are given in Tables I and II. Each compound has rigorous inversion symmetry and virtual C_{2v} symmetry and in each there is essentially trigonal bipyramidal coordination about each molybdenum atom. The asymmetry of the central planar $Mo_2(u\text{-}o)_2$ moiety is most pronounced in I due to the high trans-influence[6] of the nitrosyl ligands which occupy the other axial positions. The most striking differences between the two structures are (i) the Mo-to-Mo distances which are 3.335(2) and 2.525(1)Å, for I and II, respectively, and (ii) the angles of the $Mo_2(\mu\text{-}O)_2$ moiety.

With a Mo-Mo distance of 3.335(2)Å it may be safely assumed that no significant metal-to-metal bond exists in I, while in II, a Mo-Mo distance of 2.525Å is suggestive of a metal-to-metal double bond. This conclusion is supported by the following considerations of bonding. 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 I, 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 $\bar{Mo}\text{-}(NO^+)$. 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}. In compound II, 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 $d_{xz}\text{-}d_{xz}$ and $d_{yz}\text{-}d_{yz}$ 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 I and II the molybdenum atoms have 14-electron valence shell configurations.

Finally, we note two other reactions of $Mo_2(OR)_6$ compounds which result in the formation of fourteen valence shell electronic configurations for molybdenum:[2]

(i) $Mo_2(OR)_6 + 2L \rightleftharpoons Mo_2(OR)_6L_2$ where L = an amine

(ii) $Mo_2(OR)_6 + 2CO_2 \rightleftharpoons Mo_2(OR)_4(O_2COR)_2$.

Here the M-M triple bond is retained (Mo-Mo = 2.242(2)Å) between molybdenum atoms which are four coordinated.[7]

Acknowledgements

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4. M. H. Chisholm, W. W. Reichert and P. Thornton, J. Amer. Chem. Soc., submitted for publication.

5. Crystals of I and II were grown from hexane solutions.

Data for both compounds were collected at 23°C on a Syntex P$\bar{1}$ autodiffractometer using monochromatized MoKα radiation (λ = 0.710730Å). Unique data having 0.0°<2θMoKα<45.0° were collected and data having I>3σ(I) were considered observed and used in structure solution and refinement. Structures were solved and refined using the Enraf-Nonius Structure Determination Package and a PDP 11/45 compouter owned by Molecular Structure Corporation, College Station, Texas.

Crystal data for $Mo_2(OPr^i)_3$: a = 9.902(2), b = 17.867(3), c = 9.725(2)Å, β = 102.89(1), V = 1677.2(9)$Å^3$, Z = 2, space group = $P2_1/n$. The molecule has Ci symmetry. Refinement of non-hydrogen atoms (1826 obs. and 154 variables) employing anisotropic thermal parameters yielded R = 0.040 and R_w = 0.068.

Crystal data for $Mo_2(OPr^i)_6(NO)_2$: a = 10.823(1), b = 15.848(2), c = 9.885(2)Å, α = 90.21(2), β = 115.93(2), γ = 82.42(1)°, V = 1509.4(4)$Å^3$, Z = 4, space group P$\bar{1}$. There are two unique molecules per asymmetric unit, each possessing C_i symmetry. Refinement of non-hydrogen atoms (2052 obs. and 181 variables) employing anisotropic thermal parameters for the Mo, O, and N atoms and isotropic thermal parameters for the carbon atoms yielded R = 0.061 and R_w = 0.093.

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8. Alfred P. Sloan Fellow 1976-78.

Table I. Selected Bond Distances (Å) and Angles (Deg) in $Mo_2(OPr^i)_8$.[a]

Atoms	Distance
Mo-Mo'	2.523(1)
-O(1)	1.958(3)
-O(1)'	2.111(3)
-O(2)	1.872(3)
-O(3)	1.976(3)
-O(4)	1.884(3)

Atoms	Angle
Mo'-Mo-O(1)	54.45(9)
-O(1)'	49.00(9)
-O(2)	108.9(1)
-O(3)	137.9(1)

Atoms	Angle
Mo-Mo'-O(4)	105.1(1)
O(1)-Mo-O(1)'	103.5(1)
-O(2)	120.9(1)
-O(3)	83.5(1)
-((4)	120.2(2)
O(1)'-Mo-O(2)	84.9(1)
-O(3)	173.1(1)
-O(4)	81.0(1)
O(2)-Mo-O(3)	91.2(1)
-O(4)	118.9(2)
O(3)-Mo-O(4)	95.8(2)

[a]Atoms are labelled as in Figure 1A. Esd's are in parentheses.

Table II. Selected Bond Distances (Å) and Angles (Deg) in $Mo_2(OPr^i)_6(NO)_2$.[a]

Atoms	Molecule I	Molecule II
	Distance	
Mo(1)-Mo(1)'	3.334(2)	3.337(2)
O(1)	1.951(6)	1.946(6)
O(1)'	2.195(6)	2.194(6)
O(2)	1.850(7)	1.849(8)
O(3)	1.861(6)	1.857(7)
N(1)	1.747(9)	1.761(10)
N(1)-O(4)	1.205(11)	1.184(11)
	Angles	
Mo(1)'-Mo(1)-O(1)	39.1(2)	38.9(2)
O(1)'	34.1(2)	33.8(2)
O(2)	102.8(2)	102.8(3)
O(3)	101.4(2)	102.6(2)
N(1)	137.9(3)	138.0(3)
O(1)-Mo(1)-O(1)'	73.1(3)	72.7(3)
O(2)	119.3(3)	119.1(3)
O(3)	117.3(3)	118.6(3)
N(1)	98.8(4)	99.1(4)
O(1)'-Mo(1)-O(2)	88.4(3)	84.6(3)
O(3)	83.8(3)	84.6(3)
N(1)	171.9(4)	171.9(4)
O(2)-Mo(1)-O(3)	115.1(3)	114.2(3)
N(1)	100.8(4)	100.0(4)
O(3)-Mo(1)-N(1)	99.5(4)	99.4(4)
Mo(1)-N(1)-O(4)	178(1)	177(1)

[a]Atoms are labelled as in Figure 1b. Esd's are in parentheses.

Table III. POSITIONAL AND THERMAL PARAMETERS AND THEIR ESTIMATED STANDARD DEVIATIONS. for $Mo_2(OPr^i)_8$.

ATOM Atom	X	Y	Z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo	0.01279(5)	0.06291(3)	0.06040(5)	0.00689(5)	0.00182(2)	0.00756(6)	-0.00019(5)	0.00315(9)	-0.00019(5)
O(1)	0.0269(4)	0.0441(2)	-0.1343(4)	0.0078(4)	0.0022(1)	0.0081(4)	-0.0008(4)	0.0043(7)	0.0006(4)
O(2)	0.1705(4)	0.0614(2)	0.2083(4)	0.0093(5)	0.0026(1)	0.0098(5)	-0.0014(4)	-0.0006(8)	-0.0012(4)
O(3)	0.0501(4)	0.1675(2)	0.0146(4)	0.0124(5)	0.0020(1)	0.0109(5)	-0.0016(4)	0.0059(8)	-0.0007(4)
O(4)	-0.1610(4)	0.0821(2)	0.1019(5)	0.0098(5)	0.0027(1)	0.0151(6)	0.0018(5)	0.0100(8)	0.0005(5)
C(1)	0.1153(7)	0.0677(3)	-0.2279(7)	0.0121(7)	0.0029(2)	0.0105(7)	-0.0001(7)	0.011(1)	0.0026(6)
C(2)	0.0510(8)	0.1349(4)	-0.3089(8)	0.0174(10)	0.0037(3)	0.0134(9)	0.0014(9)	0.009(1)	0.0060(8)
C(3)	0.2653(7)	0.0834(4)	-0.1423(8)	0.0091(7)	0.0046(3)	0.0176(10)	-0.0026(8)	0.010(1)	0.0007(9)
C(4)	0.2491(7)	0.0941(4)	0.2916(6)	0.0097(7)	0.0034(2)	0.0099(7)	-0.0003(7)	-0.003(1)	0.0016(8)
C(5)	0.2331(9)	0.0134(5)	0.4438(7)	0.0185(12)	0.0066(4)	0.0108(9)	0.0005(12)	0.003(2)	0.0029(11)
C(6)	0.4001(8)	0.0152(5)	0.2799(9)	0.0097(8)	0.0045(3)	0.0210(11)	0.0004(9)	0.002(2)	0.0015(11)
C(7)	0.0972(8)	0.2232(4)	0.1190(8)	0.0186(11)	0.0021(2)	0.0161(10)	-0.0038(8)	0.006(2)	-0.0023(8)
C(8)	0.0268(10)	0.2970(5)	0.0607(10)	0.0278(17)	0.0024(3)	0.0264(17)	0.0003(11)	0.011(3)	-0.0018(10)
C(9)	0.2545(9)	0.2317(5)	0.1447(11)	0.0165(11)	0.0047(3)	0.0260(15)	-0.0087(10)	-0.001(2)	-0.0024(12)
C(10)	-0.2292(7)	0.1526(4)	0.1115(8)	0.0123(7)	0.0029(2)	0.0166(9)	0.0055(7)	0.009(1)	-0.0001(8)
C(11)	-0.3765(10)	0.1467(6)	0.0167(13)	0.0193(13)	0.0062(4)	0.0329(20)	0.0107(12)	-0.013(3)	-0.0038(16)
C(12)	-0.2447(12)	0.1630(6)	0.2615(10)	0.0460(19)	0.0076(4)	0.0205(13)	0.0232(13)	0.027(2)	0.0019(13)

The form of the anisotropic thermal parameter is: $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}\ell^2+\beta_{12}hk+\beta_{13}h\ell+\beta_{23}k\ell)]$.

Table IV. POSITIONAL AND THERMAL PARAMETERS AND THEIR ESTIMATED STANDARD DEVIATIONS. for $Mo_2(OP^i_r)_6(NO)_2$.

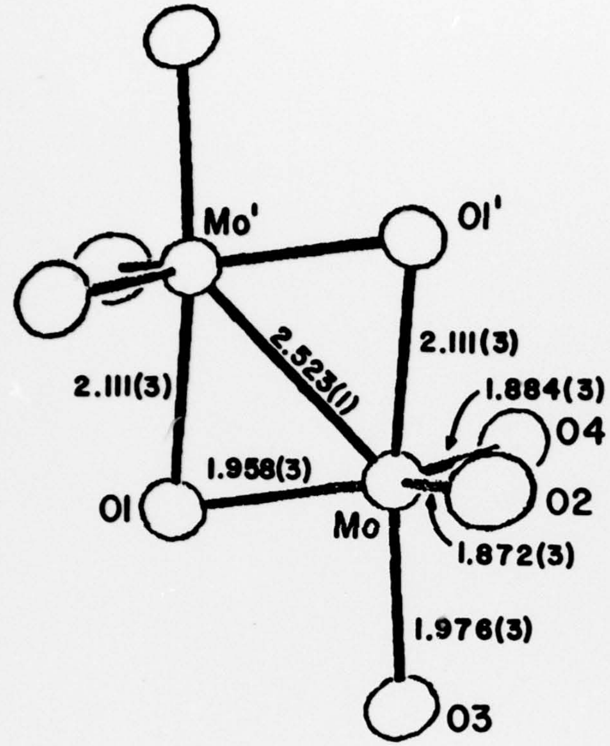
Atom	X	Y	Z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo(1)	-0.1085(1)	0.05358(8)	0.0592(1)	0.0133(1)	0.00683(6)	0.0181(1)	-0.0010(1)	0.0164(2)	0.0008(2)
Mo(2)	0.3931(1)	0.55137(8)	0.5638(1)	0.0140(1)	0.00647(6)	0.0197(2)	-0.0028(2)	0.0138(2)	-0.0004(2)
O(1)	0.0053(7)	-0.0572(5)	0.0883(6)	0.0133(8)	0.0058(4)	0.018(1)	-0.001(1)	0.017(1)	0.004(1)
O(2)	-0.0235(9)	0.1478(6)	0.1392(9)	0.0199(11)	0.0075(5)	0.020(1)	-0.003(1)	0.019(2)	-0.004(1)
O(3)	-0.2702(8)	0.0760(6)	-0.1203(9)	0.0124(9)	0.0088(5)	0.022(1)	-0.004(1)	0.012(2)	0.002(1)
O(4)	-0.2279(10)	0.0250(8)	0.2695(11)	0.0302(13)	0.0154(9)	0.036(1)	-0.001(2)	0.049(2)	0.007(2)
O(5)	0.4039(7)	0.5251(5)	0.3764(8)	0.0094(9)	0.0085(5)	0.015(1)	-0.001(1)	0.003(2)	0.001(1)
O(6)	0.4957(10)	0.6310(6)	0.6820(11)	0.0242(14)	0.0083(6)	0.027(2)	-0.006(1)	0.020(2)	-0.006(2)
O(7)	0.3676(8)	0.4652(6)	0.6717(9)	0.0164(10)	0.0080(5)	0.025(1)	-0.005(1)	0.020(2)	0.000(1)
O(8)	0.1159(11)	0.6538(7)	0.4427(13)	0.0214(14)	0.0106(7)	0.040(2)	0.009(2)	0.024(3)	0.003(2)
N(1)	-0.181(1)	0.0355(8)	0.182(1)	0.021(1)	0.0113(8)	0.024(1)	0.004(2)	0.030(2)	0.004(2)
N(2)	0.226(1)	0.6108(7)	0.489(1)	0.017(1)	0.0085(7)	0.026(2)	0.000(2)	0.021(2)	0.002(2)
C(1)	0.034(1)	-0.1349(9)	0.182(2)	7.7(4)					
C(2)	0.100(2)	-0.1130(11)	0.357(2)	9.6(5)					
C(3)	-0.103(2)	-0.1690(11)	0.139(2)	9.4(5)					
C(4)	-0.011(2)	0.1915(11)	0.274(2)	9.3(5)					
C(5)	0.121(2)	0.1494(14)	0.416(2)	13.4(7)					
C(6)	-0.093(2)	0.2814(14)	0.250(2)	13.9(7)					
C(7)	-0.413(2)	0.0696(11)	0.140(2)	10.3(5)					
C(8)	-0.49[illegible](2)	0.1504(16)	-0.212(3)	16.2(8)					
C(9)	-0.470(3)	0.0096(17)	-0.230(3)	17.7(10)					
C(10)	0.309(2)	0.5507(10)	0.215(2)	8.8(4)					
C(11)	0.175(2)	0.5100(13)	0.173(2)	12.4(6)					
C(12)	0.289(2)	0.6457(13)	0.196(2)	12.0(6)					
C(13)	0.454(2)	0.7252(14)	0.698(2)	13.6(7)					
C(14)	0.492(3)	0.7677(10)	0.596(3)	18.9(10)					
C(15)	0.404(4)	0.7357(22)	0.859(4)	23.8(13)					

Table IV. (continued)

C(16)	0.239(2)	0.4529(11)	0.686(2)	10.4(5)
C(17)	0.237(3)	0.3565(17)	0.682(3)	16.8(9)
C(18)	0.245(2)	0.4928(16)	0.825(3)	15.6(8)

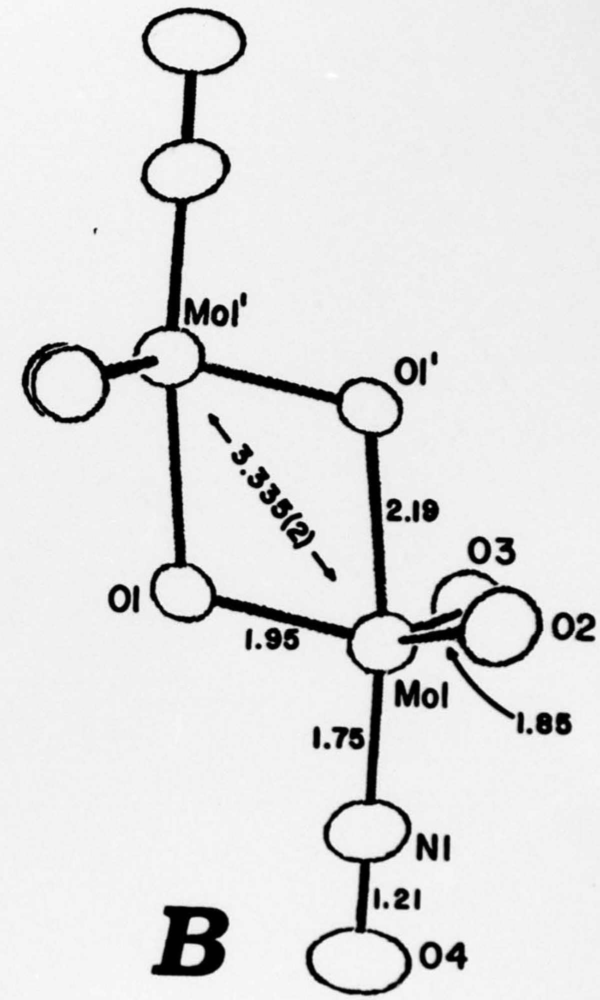
The form of the anisotropic thermal parameter is: $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}\ell^2+\beta_{12}hk+\beta_{13}h\ell+\beta_{23}k\ell)]$.

Figure 1. Coordination geometries of (A) $Mo_2(OPr^i)_8$ and (B) $Mo_2(OPr^i)_6(NO)_2$ 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_{2v} 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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