low-speed data lines to a CYBER 172-CDC 6600 multimain system where all computations were performed.

The cell dimensions obtained from 46 reflections at −170 °C with Mo Kα (λ = 0.71069 Å) were a = 21.608 (5) Å, b = 9.440 (2) Å, c = 24.076 (6) Å, β = 135.49 (1)°, V = 3442.5 (1) Å^3, Z = 4, d_{calcd} = 1.464 g cm^{-3}, and space group $C2/c$.

A total number of 6150 reflections were collected with use of standard moving-crystal moving-detector techniques with the following values: scan speed = 4.0° min^{-1}, scan width = 2.0 + dispersion, single background time at extremes of scan = 5 s, aperture size = 3.0 × 4.0 mm. Of the 6150 reflections collected, the number with unique amplitudes was 3050 and the number with $F > 2.33\sigma(F)$ was 2588. The limits of data collection were $5° < 2\theta < 50°$.

The structure was solved by a combination of direct and Fourier methods to give final residuals $R(F) = 0.0289$ and $R_w(F) = 0.0309$. The goodness of fit for the last cycle was 1.357, and the maximum Δ/σ for the last cycle was 0.65.

All of the hydrogen atoms were located and refined with use of isotropic thermal parameters. All nonhydrogen atoms were refined anisotropically.

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Registry No. $Mo_2Me_2(NMe_2)_2(C_7H_7N_3C_7H_7)_2$, 77080-92-3; $Mo_2Me_2(NMe_2)_4$, 67030-82-4.

Supplementary Material Available: A listing of observed and calculated structure factors (19 pages). Ordering information is given on any current masthead page.

Contribution from the Department of Chemistry and Molecular Structure Center,
Indiana University, Bloomington, Indiana 47405

The Molybdenum–Molybdenum Triple Bond. 10.[1] Bis(1,3-diphenyltriazenido)- and Bis(2-oxy-6-methylpyridino)tetraisopropoxydimolybdenum

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Hydrocarbon solutions of $Mo_2(O\text{-}i\text{-}Pr)_6$ (M≡M) react with each of 1,3-diphenyltriazine and 2-hydroxy-6-methylpyridine to give $Mo_2(O\text{-}i\text{-}Pr)_4(PhN_3Ph)_2$ and $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$, respectively. In both reactions, two isopropoxy groups are replaced by two bidentate groups which form cis bridges across the Mo≡Mo bond. The new compounds have been characterized by elemental analyses and IR and ^1H NMR spectroscopy; the 2-oxy-6-methylpyridino compound has been structurally characterized by a single-crystal X-ray study. Pertinent distances are Mo–Mo = 2.206 (1) Å, Mo–O (O-*i*-Pr) = 1.91 Å (average), Mo–N = 2.27 Å (average), and Mo–O (mhp) = 2.05 Å (average). Each molybdenum atom is coordinated to three oxygen atoms and a nitrogen atom, which lie roughly in a square plane. The molecule has virtual C_2 symmetry, and the two ends of the molecule avoid an eclipsed conformation by a slight twisting of the bridging groups: the average N–Mo–Mo–O torsional angle associated with the 2-oxy-6-methylpyridino ligand is 12°. These results are compared to other recent findings in the chemistry of Mo_2^{6+}-containing compounds. Crystal data for $Mo_2(O\text{-}i\text{-}Pr)_6(mhp)_2$ are a = 18.224 (8) Å, b = 15.991 (7) Å, c = 19.265 (10) Å, V = 5614 (1) Å^3, Z = 8, d_{calcd} = 1.402 g cm^{-3}, and space group $Pcab$.

Introduction

In contrast to the rather well-defined preference for six-coordinate octahedral geometries shown by chromium in its 3+ oxidation state, molybdenum(3+) and tungsten(3+) show a wide variety of coordination numbers and geometries and often form dinuclear compounds.[2] There are structurally characterized examples in which the metal atoms are coordinated to three, four, five, or six ligand atoms and to each other by metal-to-metal triple bonds.[3-5] Bioctahedra are also known and form an alternative to the structures involving unbridged M≡M bonds. These may be edge shared as in $W_2Cl_6(py)_4$[6] or face shared as in $W_2Cl_9^{3-}$.[7,8] As part of our continuing program in dinuclear transition-metal chemistry, we now report the syntheses and characterizations of two new Mo_2^{6+}-containing compounds.

Results and Discussions

Syntheses. Addition of 1,3-diphenyltriazine and 2-hydroxy-6-methylpyridine (2 equiv) to hydrocarbon solutions of $Mo_2(O\text{-}i\text{-}Pr)_6$ (M≡M)[9] leads to the formation of $Mo_2(O\text{-}i\text{-}Pr)_4(PhN_3Ph)_2$ and $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$, respectively, with the elimination of 2 equiv of isopropyl alcohol (mhp is used, hereafter, as an abbreviation for the anion derived from deprotonation of 2-hydroxy-6-methylpyridine). The reaction appears quantitative and irreversible.

Properties. Both compounds are red, air-sensitive, crystalline solids. They are appreciably soluble in aromatic hydrocarbon solvents, but noticeably less soluble in alkanes. Infrared data, ^1H NMR data, and elemental analyses are given in the Experimental Section.

Solid-State Structure of $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$. Final atomic positional parameters are given in Table I, anisotropic thermal parameters are given in Table II, and bond distances and angles are given in Tables III and IV, respectively. An ORTEP view of the molecule, giving the atom numbering scheme used in the tables, is shown in Figure 1; a view of the molecule down the Mo–Mo axis is shown in Figure 2. Torsional angles for

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0020-1669/81/1320-2215$01.25/0

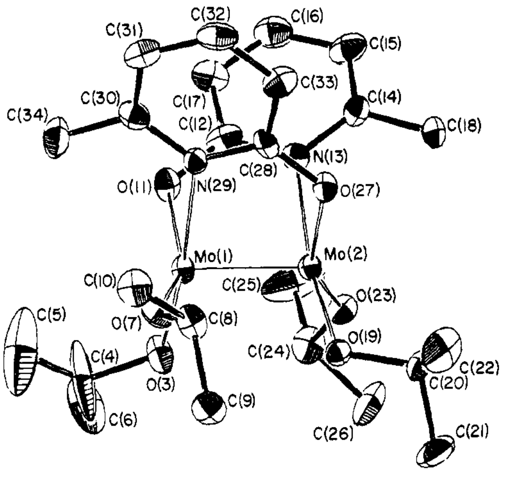

Figure 1. ORTEP view of the $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ molecule showing the atom numbering scheme used in the tables. Atoms are represented by their thermal ellipsoids drawn at the 50% probability level.

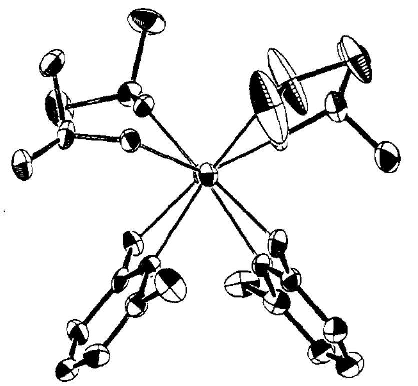

Figure 2. ORTEP view of the $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ molecule viewed down the Mo≡Mo bond.

Table I. Fractional Coordinates for the $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ Molecule[a-c]

atom	10^4x	10^4y	10^4z	$10B_{iso}$, Å^2
Mo(1)	2062.9 (4)	3167.7 (5)	3018.5 (4)	16
Mo(2)	2046.1 (4)	3879.4 (5)	3996.3 (4)	15
O(3)	1558 (4)	3964 (4)	2440 (4)	28
O(7)	3011 (3)	3164 (4)	2607 (3)	20
O(11)	1132 (3)	2534 (4)	3291 (3)	21
N(13)	1282 (4)	2988 (5)	4407 (4)	18
O(19)	2954 (3)	4617 (4)	3811 (3)	17
O(23)	1373 (3)	4673 (4)	4039 (3)	19
O(27)	2806 (3)	3029 (4)	4489 (3)	18
N(29)	2546 (4)	2087 (4)	3626 (4)	17
C(4)	1335 (9)	3820 (11)	1733 (7)	77
C(5)	1484 (10)	3139 (12)	1399 (9)	90
C(6)	608 (7)	4265 (9)	1640 (7)	50
C(8)	3751 (5)	3243 (7)	2838 (5)	22
C(9)	4060 (5)	4071 (7)	2564 (6)	27
C(10)	4178 (5)	2477 (7)	2569 (6)	27
C(12)	919 (5)	2503 (6)	3947 (6)	23
C(14)	1059 (5)	2983 (6)	5084 (5)	22
C(15)	451 (6)	2513 (7)	5307 (6)	28
C(16)	87 (6)	2020 (7)	4811 (6)	31
C(17)	318 (5)	2000 (7)	4129 (6)	27
C(18)	1496 (6)	3523 (6)	5580 (5)	28
C(20)	3149 (5)	5165 (6)	4366 (5)	21
C(21)	3106 (7)	6087 (6)	4089 (7)	39
C(22)	3924 (6)	4941 (8)	4615 (7)	42
C(24)	771 (6)	4973 (6)	3612 (6)	27
C(25)	93 (7)	4490 (8)	3787 (9)	55
C(26)	684 (7)	5911 (7)	3796 (7)	40
C(28)	2859 (5)	2262 (6)	4246 (4)	16
C(30)	2605 (5)	1288 (6)	3374 (5)	21
C(31)	2962 (6)	666 (6)	3723 (6)	27
C(32)	3282 (6)	852 (7)	4373 (6)	28
C(33)	3248 (5)	1658 (6)	4641 (6)	24
C(34)	2246 (6)	1137 (7)	2673 (6)	32

[a] The isotropic thermal parameters listed for those atoms refined anisotropically are the isotropic equivalents. [b] Numbers in parentheses in this and all following tables refer to the errors in the least significant digits. [c] Estimated standard deviations greater than 29 are not statistically significant but are left "unrounded", since the tables are all produced automatically by the X-TEL interactive programs.

the $Mo_2O_6N_2$ skeleton are given in Table V.

In the solid state, the compound is composed of discrete molecules which have virtual but not crystallographically imposed C_2 symmetry. The Mo-to-Mo distance is 2.206 (1) Å, which is slightly shorter than that found in the parent-type compounds, $Mo_2(OCH_2CMe_3)_6$ (2.222 (2) Å),[9] but is well within the range observed for $(Mo{\equiv}Mo)^{6+}$-containing compounds.[10] Each molybdenum atom is coordinated to four ligand atoms that lie roughly in a plane; these are the two oxygen atoms of a pair of cis isopropoxy groups and an oxygen atom and a nitrogen atom of a pair of cis-bridging mhp ligands. The Mo–O distances associated with alkoxy groups fall within the range 1.90–1.92 Å, which suggests the presence of some oxygen p-to-molybdenum d π bonding. The Mo–O distances associated with the mhp ligands, 2.05 (1) Å (averaged), are similar to those found for bridging O_2CX ligands coordinated to the $(Mo{\equiv}Mo)^{6+}$ unit.[11] The Mo—N distance, 2.266 (8) Å, though considerably longer than that found[12] in the quadruply bridged compound $Mo_2(mhp)_4$ (M≡M), 2.167 (16) Å, is most definitely shorter than that found in $Mo_2(NMe_2)_4(mhp)_2$ (M≡M), 2.325 (10) Å.[13] Indeed, the Mo—N distance is comparable to that found[14] for $W_2(O\text{-}i\text{-}Pr)_6(py)_2$ (M≡M), and since metal–ligand distances are virtually identical in related M_2^{6+}-containing compounds[15] (M = Mo, W), this implies a comparable nitrogen-to-molybdenum dative bond. As is seen later, there is good evidence to support the view that the N-to-Mo bond is maintained when $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ is dissolved in hydrocarbons and, furthermore, that this bond is stronger than the analogous N-to-Mo bond in $Mo_2(NMe_2)_4(mhp)_2$.

Solution Properties: **^{1}H NMR Studies.** The ^{1}H NMR spectra for $Mo_2(O\text{-}i\text{-}Pr)_4(PhN_3Ph)_2$ and $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ have been recorded in the temperature range 16–96 °C in toluene-d_8 at 220 MHz. The spectra for both compounds are invariant within this temperature range and are consistent with cis-bridged structures. For $Mo_2(O\text{-}i\text{-}Pr)_4(PhN_3Ph)_2$, there is a septet for the methyne proton resonance and a pair of doublets for the methyl protons of the O-*i*-Pr ligands. For $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$, there are two methyne signals in the integral ratio 1:1 and two pairs of doublets for the methyl groups of the O-*i*-Pr ligands. For both compounds, the presence of cis-bridging groups generates diastereotropic isopropyl methyl groups since the molecules lack planes of symmetry containing the Mo-O-*i*-Pr ligands. For $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$, there are two types of O-*i*-Pr ligands, namely, those trans to oxygen atoms and those trans to nitrogen atoms.

Certain points are worthy of noting:

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(10) For a listing, see ref 14.

(11) Compare 2.10 Å (average) found in $Mo_2(O\text{-}t\text{-}Bu)_4(O_2CO\text{-}t\text{-}Bu)_2$: Chisholm, M. H.; Cotton, F. A.; Extine, M. W.; Reichert, W. W. *J. Am. Chem. Soc.* **1978**, *100*, 1727.

(12) Cotton, F. A.; Fanwick, P. E.; Niswander, R. H.; Sekutowski, J. C. *J. Am. Chem. Soc.* **1978**, *100*, 4725.

(13) Chisholm, M. H.; Folting, K.; Huffman, J. C.; Rothwell, I. P. *Inorg. Chem.* **1981**, *20*, 1496.

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(15) See discussion in ref 11.

Table II. Anisotropic Thermal Parameters for the $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ Molecule[a]

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo(1)	18.5 (4)	23.8 (4)	17.5 (4)	1.4 (4)	−1.9 (4)	−2.5 (4)
Mo(2)	18.2 (4)	20.6 (4)	17.6 (4)	0.9 (4)	−1.0 (4)	−1.0 (4)
O(3)	43 (4)	40 (4)	22 (4)	18 (4)	−5 (3)	−5 (3)
O(7)	20 (3)	34 (4)	20 (3)	4 (3)	4 (3)	6 (3)
O(11)	18 (3)	36 (4)	25 (4)	−2 (3)	−2 (3)	−5 (3)
N(13)	20 (4)	24 (4)	24 (5)	3 (4)	7 (4)	0 (4)
O(19)	19 (3)	22 (3)	22 (3)	−3 (3)	0 (3)	−0 (3)
O(23)	21 (3)	21 (3)	32 (4)	5 (3)	−8 (3)	1 (3)
O(27)	22 (4)	23 (3)	22 (3)	6 (3)	−2 (3)	−2 (3)
N(29)	21 (4)	23 (4)	19 (4)	−3 (4)	0 (4)	−0 (4)
C(4)	115 (13)	138 (15)	38 (8)	100 (12)	−53 (9)	−52 (9)
C(5)	100 (14)	134 (16)	109 (15)	73 (13)	−68 (12)	−85 (13)
C(6)	64 (9)	92 (11)	36 (7)	48 (8)	−22 (7)	−7 (7)
C(8)	14 (5)	43 (6)	27 (5)	−0 (5)	1 (4)	−0 (5)
C(9)	26 (6)	43 (7)	34 (6)	−10 (5)	2 (5)	4 (5)
C(10)	22 (5)	39 (6)	41 (6)	11 (5)	2 (5)	−2 (6)
C(12)	20 (5)	20 (5)	47 (6)	3 (4)	7 (5)	3 (5)
C(14)	35 (6)	23 (5)	27 (6)	1 (5)	0 (5)	−4 (4)
C(15)	34 (6)	24 (5)	49 (7)	3 (5)	13 (6)	0 (6)
C(16)	35 (6)	36 (7)	45 (7)	−0 (5)	7 (5)	6 (6)
C(17)	27 (6)	39 (7)	37 (7)	0 (5)	−0 (5)	9 (5)
C(18)	56 (8)	29 (6)	20 (6)	−5 (5)	8 (5)	−6 (5)
C(20)	29 (6)	22 (5)	29 (6)	−4 (4)	−3 (5)	−5 (5)
C(21)	68 (8)	18 (5)	61 (9)	−2 (6)	−9 (7)	1 (6)
C(22)	41 (7)	53 (8)	65 (9)	−3 (6)	−36 (7)	−2 (7)
C(24)	26 (6)	36 (6)	40 (7)	7 (5)	−6 (5)	−2 (5)
C(25)	30 (7)	44 (8)	135 (14)	−10 (6)	−29 (8)	31 (9)
C(26)	60 (8)	26 (6)	65 (9)	9 (6)	−35 (7)	−15 (6)
C(28)	13 (4)	33 (5)	16 (5)	−1 (4)	6 (4)	−0 (4)
C(30)	24 (5)	32 (6)	25 (6)	3 (4)	8 (4)	3 (5)
C(31)	32 (6)	24 (6)	46 (7)	−0 (5)	2 (6)	4 (5)
C(32)	25 (6)	35 (6)	46 (7)	0 (5)	9 (5)	14 (6)
C(33)	25 (5)	25 (6)	42 (7)	6 (4)	−2 (5)	2 (5)
C(34)	53 (7)	35 (6)	33 (6)	11 (6)	−9 (6)	−12 (5)

[a] All U values are $\times 10^3$. The form of the exponent is $\exp[-2\pi^2(U_{11}h^2a^{*2} + 2U_{12}hka^*b^* + \ldots)]$.

Table III. Bond Distances (Å) for the $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ Molecule

A	B	distance	A	B	distance
Mo(1)	Mo(2)	2.206 (1)	C(4)	C(5)	1.295 (19)
Mo(1)	O(3)	1.926 (7)	C(4)	C(6)	1.515 (16)
Mo(1)	O(7)	1.901 (6)	C(8)	C(9)	1.532 (14)
Mo(1)	O(11)	2.044 (6)	C(8)	C(10)	1.541 (14)
Mo(1)	N(29)	2.266 (7)	C(12)	C(17)	1.404 (13)
Mo(2)	O(19)	1.920 (6)	C(14)	C(15)	1.407 (14)
Mo(2)	O(23)	1.897 (6)	C(14)	C(18)	1.514 (14)
Mo(2)	O(27)	2.048 (6)	C(15)	C(16)	1.406 (15)
Mo(2)	N(13)	2.266 (8)	C(16)	C(17)	1.380 (15)
O(3)	C(4)	1.438 (13)	C(20)	C(21)	1.569 (13)
O(7)	C(8)	1.425 (10)	C(20)	C(22)	1.534 (14)
O(11)	C(12)	1.324 (12)	C(24)	C(25)	1.496 (15)
O(19)	C(20)	1.428 (11)	C(24)	C(26)	1.549 (14)
O(23)	C(24)	1.453 (11)	C(28)	C(33)	1.419 (13)
O(27)	C(28)	1.316 (11)	C(30)	C(31)	1.366 (14)
N(13)	C(12)	1.351 (12)	C(30)	C(34)	1.521 (14)
N(13)	C(14)	1.365 (12)	C(31)	C(32)	1.412 (15)
N(29)	C(28)	1.353 (11)	C(32)	C(33)	1.390 (14)
N(29)	C(30)	1.371 (12)			

1. No detectable concentrations of trans-bridged isomers are present. If trans-bridged species are in equilibrium with cis-bridged species, cis $\rightleftharpoons$ trans bridge isomerization is slow on the NMR time scale below 100 °C.

2. The appearance of two types of O-*i*-Pr ligands up to 100 °C in the spectra of $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ indicates that O-*i*-Pr group exchange is slow on the NMR time scale. A number of possible reactions could have led to this site exchange. These include (i) rapid equilibration between bridged and nonbridged forms of the molecule (this was observed[13] for $Mo_2(NMe_2)_4(mhp)_2$), (ii) pairwise exchange of O-*i*-Pr ligands across the Mo–Mo bond, and (iii) equatorial to axial (with respect to the Mo–Mo axis) site exchange. Evidently none of the above are particularly facile processes.

Concluding Remarks. The present work provides two further examples of $(Mo{\equiv}Mo)^{6+}$-containing compounds in which the molybdenum atoms are coordinated to four ligand atoms that lie roughly in a plane. The occurrence of cis-bridged structures in $Mo_2(O\text{-}t\text{-}Bu)_4(O_2CO\text{-}t\text{-}Bu)_2$,[11] $Mo_2Me_2(NMe_2)_2(PhN_3Ph)_2$,[12] and $Mo_2(NMe_2)_4(mhp)_2$[13] and now in $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ is quite striking. Even in solution, we find no evidence for trans-bridged isomers. Evidently, the availability of five molybdenum atomic orbitals (s, p_x, p_y, d_{xy}, and $d_{x^2-y^2}$), which may be used to form bonds in the xy plane (the z axis is defined along the Mo–Mo axis), allows for a considerable degree of flexibility in metal–ligand bond angles within this plane. The angle between the bonds formed to the bridging ligands is small, typically being between 75 and 80°, while the angle between the bonds to the cis Or or NMe_2 groups is large, falling in the range 100–110°. Furthermore, the two ends of the molecule avoid an eclipsed geometry by a twisting about the Mo–Mo bond. This generates two rather different trans angles at each molybdenum atom: one is ca. 170° and the other ca. 148°. A marked trans influence is not observed, or at least not reflected, in metal–ligand distances, though the shortest bond distances are to ligands which have the small, ca. 148°, trans angle.

Finally, it should be noted that the shorter Mo-to-pyridine nitrogen bond distances in $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ relative to that in $Mo_2(NMe_2)_4(mhp)_2$ and the solution behavior of the two compounds show that substitution of O-*i*-Pr for NMe_2 increases the Lewis base affinity of the Mo_2^{6+} center toward the pyridine nitrogen donor.

Experimental Section

General procedures, including the preparation of $Mo_2(O\text{-}i\text{-}Pr)_6$, have been described previously.[9] Note the use of dry and oxygen-free atmospheres and solvents.

Table IV. Bond Angles (Deg) for the $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ Molecule

A	B	C	angle
Mo(2)	Mo(1)	O(3)	100.7 (2)
Mo(2)	Mo(1)	O(7)	107.1 (2)
Mo(2)	Mo(1)	O(11)	95.4 (2)
Mo(2)	Mo(1)	N(29)	85.7 (2)
O(3)	Mo(1)	O(7)	101.2 (3)
O(3)	Mo(1)	O(11)	94.6 (3)
O(3)	Mo(1)	N(29)	171.5 (3)
O(7)	Mo(1)	O(11)	149.3 (3)
O(7)	Mo(1)	N(29)	81.9 (3)
O(11)	Mo(1)	N(29)	79.1 (3)
Mo(1)	Mo(2)	O(19)	102.2 (2)
Mo(1)	Mo(2)	O(23)	109.3 (2)
Mo(1)	Mo(2)	O(27)	95.4 (2)
Mo(1)	Mo(2)	N(13)	85.8 (2)
O(19)	Mo(2)	O(23)	99.6 (3)
O(19)	Mo(2)	O(27)	92.5 (3)
O(19)	Mo(2)	N(13)	169.7 (3)
O(23)	Mo(2)	O(27)	149.3 (3)
O(23)	Mo(2)	N(13)	83.7 (3)
O(27)	Mo(2)	N(13)	80.1 (3)
Mo(1)	O(3)	C(4)	125.3 (8)
Mo(1)	O(7)	C(8)	136.9 (6)
Mo(1)	O(11)	C(12)	120.5 (6)
Mo(2)	O(19)	C(20)	115.4 (5)
Mo(2)	O(23)	C(24)	139.2 (6)
Mo(2)	O(27)	C(28)	119.8 (6)
Mo(2)	N(13)	C(12)	118.2 (6)
Mo(2)	N(13)	C(14)	122.9 (6)
C(12)	N(13)	C(14)	118.5 (8)
Mo(1)	N(29)	C(28)	117.5 (6)
Mo(1)	N(29)	C(30)	123.9 (6)
C(28)	N(29)	C(30)	118.2 (8)
O(3)	C(4)	C(5)	123.1 (14)
O(3)	C(4)	C(6)	106.6 (10)
C(5)	C(4)	C(6)	121.3 (14)
O(7)	C(8)	C(9)	108.5 (8)
O(7)	C(8)	C(10)	107.6 (8)
C(9)	C(8)	C(10)	112.6 (8)
O(11)	C(12)	N(13)	117.4 (8)
O(11)	C(12)	C(17)	119.3 (10)
N(13)	C(12)	C(17)	123.2 (10)
N(13)	C(14)	C(15)	122.0 (9)
N(13)	C(14)	C(18)	116.3 (9)
C(15)	C(14)	C(18)	121.7 (9)
C(14)	C(15)	C(16)	117.7 (10)
C(15)	C(16)	C(17)	121.1 (10)
C(12)	C(17)	C(16)	117.5 (10)
O(19)	C(20)	C(21)	108.1 (8)
O(19)	C(20)	C(22)	108.6 (8)
C(21)	C(20)	C(22)	111.8 (9)
O(23)	C(24)	C(25)	109.0 (9)
O(23)	C(24)	C(26)	105.5 (8)
C(25)	C(24)	C(26)	111.3 (10)
O(27)	C(28)	N(29)	118.4 (8)
O(27)	C(28)	C(33)	118.7 (8)
N(29)	C(28)	C(33)	122.8 (9)
N(29)	C(30)	C(31)	122.8 (9)
N(29)	C(30)	C(34)	115.4 (9)
C(31)	C(30)	C(34)	121.8 (9)
C(30)	C(31)	C(32)	118.6 (10)
C(31)	C(32)	C(33)	120.4 (10)
C(28)	C(33)	C(32)	117.1 (9)

Table V. Torsional Angles (Deg) Along the Mo–Mo Bond in the $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$ Molecule

O(3)	Mo(1)	Mo(2)	N(13)	107.8 (3)
O(3)	Mo(1)	Mo(2)	O(19)	−78.8 (3)
O(3)	Mo(1)	Mo(2)	O(23)	26.1 (3)
O(3)	Mo(1)	Mo(2)	O(27)	−172.5 (3)
O(7)	Mo(1)	Mo(2)	N(13)	−146.8 (3)
O(7)	Mo(1)	Mo(2)	O(19)	26.6 (3)
O(7)	Mo(1)	Mo(2)	O(23)	131.5 (3)
O(7)	Mo(1)	Mo(2)	O(27)	−67.1 (3)
O(11)	Mo(1)	Mo(2)	N(13)	12.1 (3)
O(11)	Mo(1)	Mo(2)	O(19)	−174.5 (3)
O(11)	Mo(1)	Mo(2)	O(23)	−69.7 (3)
O(11)	Mo(1)	Mo(2)	O(27)	91.8 (3)
N(29)	Mo(1)	Mo(2)	N(13)	−66.6 (3)
N(29)	Mo(1)	Mo(2)	O(19)	106.9 (3)
N(29)	Mo(1)	Mo(2)	O(23)	−148.3 (3)
N(29)	Mo(1)	Mo(2)	O(27)	13.1 (3)

Preparation of $Mo_2(O\text{-}i\text{-}Pr)_4(PhN_3Ph)_2$. To a solution of $Mo_2(O\text{-}i\text{-}Pr)_6$ (0.50 g) in toluene (20 mL) was added 1,3-diphenyltriazine (0.38 g, 1 mol/mol of Mo), and the solution was stirred for 0.5 h. The solvent was removed in vacuo, and the resulting dark solids were extracted with hexane. Cooling the dark-brown solution to −15 °C gave crystals of the product. Anal. Calcd for $Mo_2C_{36}H_{48}N_6O_4$: C, 52.68; H, 5.90; N, 10.24. Found: C, 52.55; H, 6.18; N, 10.16. ^1H NMR data obtained in toluene-d_8 at 220 MHz and +16 °C: $\delta(OCHMe_2)$ = 6.07 septet; $\delta(OCHMe_2)$ = 1.49 d, 1.53 d; δ(aromatic protons) = 6.5–7.2 (a series of unresolved multiplets). IR data obtained from Nujol mulls between CsI plates, ν in cm^{-1}: 1595 m, 1370 m, 1270 s, 1110 vs, 1100 m, 1070 w, 1030 m, 990 m, 960 w, 810 s, 760 m, 690 m, 590 m, 535 m, 355 m, 310 w.

Preparation of $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$. Following the same procedure as above, we reacted $Mo_2(O\text{-}i\text{-}Pr)_6$ (0.50 g) and 2-hydroxy-6-methylpyridine (0.20 g, 1 mol/mol of Mo) to give deep red crystals from hexane. Anal. Calcd for $Mo_2C_{24}H_{40}O_6N_2$: C, 44.72; H, 6.46; N, 4.32. Found: C, 45.08; H, 6.46; N, 4.32. ^1H NMR data obtained in toluene-d_8 at 220 MHz and +16 °C: $\delta(OCHMe_2)$ = 4.72, 7.14 (septets); $\delta(OCHMe_2)$ = 1.81 d, 1.34 d, 1.42 d, 1.19 d; δ(*Me*py) = 1.59 s; δ(aromatic protons) = 5.92 d, 6.45 d, 6.89 dd. IR data obtained from Nujol mulls between CsI plates, ν in cm^{-1}: 1610 s, 1562 m, 1365 w, 1355 m, 1345 m, 1270 m, 1252 w, 1170 m, 1121 s, 1042 w, 1021 w, 987 s, 955 ms, 857 w, 840 w, 800 s, 768 s, 748 w, 600 m, 550 m, 510 m, 355 m.

X-ray Structure Determination. A crystal of dimensions 0.25 × 0.25 × 0.35 mm was mounted in a nitrogen-filled glovebag and transferred to the liquid-nitrogen boil-off cooling system of the diffractometer.[16] The cell dimensions obtained from 36 reflections at −144 °C with Mo Kα (λ = 0.71069 Å) were a = 18.224 (8) Å, b = 15.991 (7) Å, c = 19.265 (10) Å, V = 5614 (1) $Å^3$, Z = 8, d_{calcd} = 1.402 g cm^{-3}, and space group *Pcab*.

A total number of 5613 reflections were collected with use of standard moving-crystal, moving-detector techniques with the following values: scan speed = 3° min^{-1}, scan width = 2.0° + dispersion, single background time at extremes of scan = 5 s, aperture size = 3.0 × 4.0 mm. The limits of data collection were $5° \le 2\theta \le 50°$. Of the 5613 reflections collected, the number with unique intensities was 4868. The number of reflections with $F > 2.33\sigma(F)$ was 3539.

The structure was solved by a combination of direct and Fourier methods to give residuals $R(F)$ = 0.0643 and $R_w(F)$ = 0.0634. The goodness of fit for the last cycle was 1.401, and the maximum δ/σ for the last cycle was 0.05.

All nonhydrogen atoms were located and refined by full-matrix least-squares methods using anisotropic thermal parameters. No hydrogen atoms were included in the calculations. Several peaks in the final difference map could have been interpreted as hydrogen atoms. However, the large thermal parameters associated with the isopropyl methyl carbon atoms make an attempted location and refinement not worthwhile.

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Registry No. $Mo_2(O\text{-}i\text{-}Pr)_4(PhN_3Ph)_2$, 77450-42-1; $Mo_2(O\text{-}i\text{-}Pr)_4(mhp)_2$, 77450-43-2; $Mo_2(O\text{-}i\text{-}Pr)_6$, 62521-20-4.

Supplementary Material Available: A listing of structure factor amplitudes (31 pages). Ordering information is given on any current masthead page.

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