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Registry No. [Co(NH₃)₂(L-ala-gly-gly)]·2H₂O, 83351-01-3; Co(NH₃)₂(gly-gly-L-ala), 74808-51-8; Co(NH₃)₂(gly-gly-L-leu), 74808-52-9; Co(NH₃)₂(gly-gly-L-phe), 74808-53-0; Co(NH₃)₂(L-

leu-gly-gly), 83351-02-4; Co(NH₃)₂(L-phe-gly-gly), 74808-55-2; Co(NH₃)₂(L-tyr-gly-gly), 74808-56-3.

Supplementary Material Available: Listings of observed and calculated structure factors, atomic coordinates, thermal parameters, and deviations of the atoms from the metal and ligand atom planes for [Co(NH₃)₂(L-ala-gly-gly)]·2H₂O (15 pages). Ordering information is given on any current masthead page.

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The Molybdenum-Molybdenum Triple Bond. 12.¹ Preparation and Characterization of Bis(*tert*-butyl mercaptido)tetrakis(dimethylamido)dimolybdenum(M≡M)

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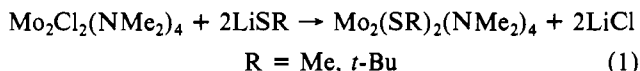
From the reaction between Mo₂Cl₂(NMe₂)₄ and LiSR (≥2 equiv) in hexane, yellow-orange crystalline compounds of Mo₂(SR)₂(NMe₂)₄ have been isolated, where R = Me and *t*-Bu. Crystal data for Mo₂(S-*t*-Bu)₂(NMe₂)₄ are *a* = 14.322 (4) Å, *b* = 17.514 (5) Å, *c* = 9.865 (2) Å, β = 101.39 (1)°, *V* = 2425.7 (1) Å³, *Z* = 4, *d*_{calcd} = 1.497 g cm⁻³, and space group *P*2₁/*n*. The solid-state molecular structure reveals an unbridged Mo≡Mo bond of distance 2.217 (1) Å and an antirotameric conformation for the Mo₂S₂N₄ skeleton, which has virtual C_{2h} symmetry. The important averaged bond distances and bond angles are Mo-S = 2.363 (3) Å, Mo-N = 1.95 (1) Å, Mo-Mo-S = 95 (1)°, and Mo-Mo-N = 104 (1)°. These observations are compared with related findings for compounds having a central (Mo≡Mo)⁶⁺ moiety. Mo₂(S-*t*-Bu)₂(NMe₂)₄ reacts with excess *t*-BuSH to give Mo₂(μ-S)₂(S-*t*-Bu)₄(HNMe₂)₂ as the only characterizable molybdenum-containing compound. Mo₂(S-*t*-Bu)₂(NMe₂)₄ reacts with excess ROH (R = *t*-Bu, *i*-Pr) to give Mo₂(OR)₆ compounds with the elimination of HNMe₂ and HS-*t*-Bu.

Introduction

Molybdenum and tungsten are now well-known to form an extensive series of dinuclear compounds with a central (M≡M)⁶⁺ unit.^{2,3} The simplest members of this series are the homoleptic compounds X₃M≡MX₃, where X = CH₂SiMe₃, NMe₂, OR (R = *t*-Bu, *i*-Pr), and the closely related compounds of formula M₂X₂Y₄, where X = Cl, Br and Y = NMe₂, CH₂SiMe₃. As part of a general program aimed at elucidating the coordination chemistry of the (Mo≡Mo)⁶⁺ unit, we investigated reactions between Mo₂(NMe₂)₆ and bulky thiols RSH (R = *t*-Bu, *i*-Pr, CH₂Ph). The products of these reactions were not, however, Mo₂(SR)₆ compounds that could have been formed by simple exchange reactions but rather were Mo₂(μ-S)₂(SR)₄(HNMe₂)₂ compounds.⁴ We now describe further reactions aimed at attaching mercaptido groups to the (Mo≡Mo)⁶⁺ unit.

Results and Discussion

Synthesis. Hydrocarbon solutions of Mo₂Cl₂(NMe₂)₄⁵ and LiSR (2 equiv) react according to the metathetic reaction (1).



The new mercaptido compounds are yellow-orange crystalline solids that are exceedingly air and moisture sensitive. When heated under vacuum, they sublime at 90–100 °C (10⁻⁴ torr), though this is accompanied by some decomposition. They are

soluble in hydrocarbon solvents at room temperature, but only sparingly so at low temperatures. Elemental analyses and other characterization data are given in the Experimental Section.

Solid-State Structure of Mo₂(S-*t*-Bu)₂(NMe₂)₄. Final atomic positional parameters are given in Table I. Bond distances and angles are given in Tables II and III, respectively. An ORTEP view of the molecule giving the atomic numbering scheme used in the tables is given in Figure 1, and a view of the molecule down the Mo-Mo bond is shown in Figure 2.

The Mo-Mo distance of 2.217 (1) Å is typical of the distances found in Mo₂X₂(NMe₂)₄-containing compounds; cf. Mo-Mo = 2.201 (2) Å, which is found for the compounds where X = Cl⁵ and Me,⁶ both of which also crystallize from hydrocarbon solvent in the antirotamer. The Mo-N distances, 1.95 (1) Å (averaged), and the planar arrangement of the NMe₂ groups with respect to the Mo≡Mo bond are also typical of M₂X₂(NMe₂)₄ compounds. The Mo-N distances, 1.95 (1) Å (averaged), are somewhat shorter, however, than those found in Mo₂(NMe₂)₆, which span a range from 1.97 (1) to 2.00 (1) Å and average 1.98 Å.⁷ This is consistent with the view that substitution of a less π-donating group, SR for NMe₂, would enhance N-to-Mo π bonding in the remaining Mo-NMe₂ groups. The Mo-N distance, 1.95 (1) Å (averaged), is, however, greater than that in Mo₂Cl₂(NMe₂)₄, 1.93 (1) Å (averaged). Again, this is consistent with the further replacement of a more electronegative group and a weaker π-donating ligand, Cl for SR.

One might wonder whether the Mo-S distances 2.362 (2) and 2.363 (2) Å provide any indication of sulfur-to-molybdenum π bonding. Indeed, what Mo-S distance might one

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Table I. Fractional Coordinates^a and Isotropic Thermal Parameters for the Mo₂(S-*t*-Bu)₂(NMe₂)₄ Molecule

atom	x	y	z	10B _{iso} , Å ²
Mo(1)	7740.0 (3)	1501.9 (3)	6097.0 (5)	11
Mo(2)	7594.9 (3)	1292.6 (3)	3848.4 (5)	11
S(1)	8107 (1)	2814 (1)	5999 (2)	16
S(2)	7213 (1)	-15 (1)	3975 (1)	16
N(1)	6492 (3)	1263 (3)	6474 (5)	16
N(2)	8812 (3)	843 (3)	6933 (5)	16
N(3)	8847 (3)	1560 (3)	3483 (5)	15
N(4)	6543 (3)	1945 (3)	2971 (5)	17
C(1)	7921 (4)	3339 (3)	7568 (6)	20
C(2)	8244 (5)	2889 (4)	8873 (7)	23
C(3)	8485 (6)	4073 (5)	7599 (9)	28
C(4)	6858 (5)	3516 (5)	7384 (9)	31
C(5)	6401 (5)	1227 (4)	7928 (6)	18
C(6)	5585 (5)	1019 (4)	5615 (7)	22
C(7)	9414 (5)	332 (4)	6374 (7)	18
C(8)	9091 (5)	863 (4)	8447 (7)	23
C(9)	6976 (4)	-485 (3)	2249 (6)	15
C(10)	6011 (5)	-245 (5)	1441 (8)	24
C(11)	7752 (5)	-306 (5)	1451 (7)	25
C(12)	6951 (6)	-1331 (4)	2543 (8)	30
C(13)	6324 (5)	1905 (4)	1465 (7)	21
C(14)	5959 (5)	2526 (4)	3459 (7)	20
C(15)	8971 (5)	1584 (4)	2042 (7)	21
C(16)	9707 (4)	1851 (4)	4366 (7)	19
H(1)	797 (4)	242 (4)	896 (6)	33 (15)
H(2)	895 (4)	274 (3)	902 (5)	11 (11)
H(3)	820 (5)	320 (4)	968 (7)	37 (16)
H(4)	824 (4)	430 (3)	678 (6)	16 (12)
H(5)	841 (5)	430 (4)	818 (8)	42 (19)
H(6)	916 (4)	398 (3)	768 (5)	15 (11)
H(7)	647 (5)	302 (4)	734 (7)	47 (18)
H(8)	675 (5)	380 (4)	813 (8)	42 (17)
H(9)	668 (4)	381 (3)	655 (6)	14 (11)
H(10)	599 (4)	157 (3)	811 (5)	5 (10)
H(11)	621 (4)	67 (3)	822 (6)	25 (13)
H(12)	699 (4)	134 (3)	847 (6)	21 (13)
H(13)	511 (4)	135 (3)	565 (5)	9 (11)
H(14)	530 (5)	49 (4)	577 (7)	40 (16)
H(15)	564 (4)	104 (3)	468 (7)	28 (13)
H(16)	953 (5)	-21 (4)	686 (8)	50 (18)
H(17)	924 (4)	28 (4)	546 (7)	27 (14)
H(18)	1004 (4)	50 (3)	649 (6)	18 (12)
H(19)	867 (5)	117 (4)	879 (7)	31 (15)
H(20)	978 (4)	95 (3)	879 (5)	15 (11)
H(21)	894 (4)	40 (3)	883 (6)	19 (13)
H(22)	597 (5)	24 (4)	115 (7)	40 (18)
H(23)	589 (4)	-46 (4)	58 (7)	31 (15)

^a Fractional coordinates are $\times 10^4$ for non-hydrogen atoms and $\times 10^3$ for hydrogen atoms.

Table II. Bond Distances (Å) for the Mo₂(S-*t*-Bu)₂(NMe₂)₄ Molecule

A	B	dist	A	B	dist
Mo(1)	Mo(2)	2.217 (1)	N(2)	C(8)	1.468 (8)
Mo(1)	S(1)	2.363 (2)	N(3)	C(15)	1.468 (8)
Mo(1)	N(1)	1.942 (5)	N(3)	C(16)	1.454 (8)
Mo(1)	N(2)	1.967 (5)	N(4)	C(13)	1.458 (8)
Mo(2)	S(2)	2.364 (2)	N(4)	C(14)	1.456 (8)
Mo(2)	N(3)	1.954 (5)	C(1)	C(2)	1.502 (9)
Mo(2)	N(4)	1.953 (5)	C(1)	C(3)	1.517 (9)
S(1)	C(1)	1.864 (6)	C(1)	C(4)	1.528 (10)
S(2)	C(9)	1.860 (6)	C(9)	C(10)	1.513 (9)
N(1)	C(5)	1.466 (8)	C(9)	C(11)	1.516 (9)
N(1)	C(6)	1.468 (8)	C(9)	C(12)	1.511 (9)
N(2)	C(7)	1.427 (8)			

expect when an RS group coordinates to a (Mo≡Mo)⁶⁺ unit? If we take the covalent radius of C(sp³) to be 0.77 Å and assume that the Mo-C distance, 2.17 Å, in Mo₂(CH₃)₂(NMe₂)₄ represents a pure Mo-C σ bond, then we may take the covalent radius of molybdenum to be 1.40 Å. The C-S distances in the *t*-BuS ligands are 1.86 Å, which gives a covalent radius for sulfur of 1.09 Å. One might therefore es-

Table III. Bond Angles (Deg) for the Mo₂(S-*t*-Bu)₂(NMe₂)₄ Molecule

A	B	C	angle
Mo(2)	Mo(1)	S(1)	95.6 (0)
Mo(2)	Mo(1)	N(1)	104.4 (1)
Mo(2)	Mo(1)	N(2)	103.5 (1)
S(1)	Mo(1)	N(1)	116.0 (1)
S(1)	Mo(1)	N(2)	115.2 (1)
N(1)	Mo(1)	N(2)	117.7 (2)
Mo(1)	Mo(2)	S(2)	94.8 (0)
Mo(1)	Mo(2)	N(3)	103.7 (1)
Mo(1)	Mo(2)	N(4)	105.0 (1)
S(2)	Mo(2)	N(3)	118.2 (1)
S(2)	Mo(2)	N(4)	115.1 (1)
N(3)	Mo(2)	N(4)	115.7 (2)
Mo(1)	S(1)	C(1)	112.1 (2)
Mo(2)	S(2)	C(9)	112.5 (2)
Mo(1)	N(1)	C(5)	117.4 (4)
Mo(1)	N(1)	C(6)	134.0 (4)
C(5)	N(1)	C(6)	108.2 (5)
Mo(1)	N(2)	C(7)	133.4 (4)
Mo(1)	N(2)	C(8)	116.4 (4)
C(7)	N(2)	C(8)	110.1 (5)
Mo(2)	N(3)	C(15)	118.5 (4)
Mo(2)	N(3)	C(16)	132.3 (4)
C(15)	N(3)	C(16)	108.6 (5)
Mo(2)	N(4)	C(13)	114.5 (4)
Mo(2)	N(4)	C(14)	134.8 (4)
C(13)	N(4)	C(14)	110.4 (5)
S(1)	C(1)	C(2)	112.5 (4)
S(1)	C(1)	C(3)	105.8 (5)
S(1)	C(1)	C(4)	107.7 (4)
C(2)	C(1)	C(3)	110.7 (6)
C(2)	C(1)	C(4)	109.8 (6)
C(3)	C(1)	C(4)	110.2 (6)
S(2)	C(9)	C(10)	110.0 (4)
S(2)	C(9)	C(11)	111.3 (4)
S(2)	C(9)	C(12)	105.3 (4)
C(10)	C(9)	C(11)	110.9 (5)
C(10)	C(9)	C(12)	108.6 (6)
C(11)	C(9)	C(12)	110.5 (6)

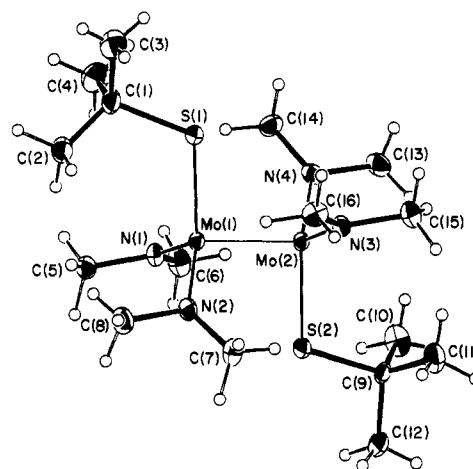


Figure 1. ORTEP view of the Mo₂(S-*t*-Bu)₂(NMe₂)₄ molecule showing the atom numbering scheme used in the tables. Non-hydrogen atoms are represented by ellipsoids drawn at the 50% probability level; hydrogen atoms have been given an artificial thermal parameter.

timate a covalent Mo-S bond distance of 2.49 Å. If one were to use the S-S distance in S₆ to estimate the covalent radius of divalent sulfur, 1.03 Å, then the Mo-S distance would be 2.43 Å. In any event, the observed distance, 2.36 Å, would appear to be smaller, by perhaps 0.1 Å, than what might be viewed as a reasonable Mo-S estimate based on covalent radii considerations. This shortening might be a result of some S-to-Mo π bonding, though the case is not a very strong one as some shortening would be expected from a consideration of electronegativity differences between the elements.

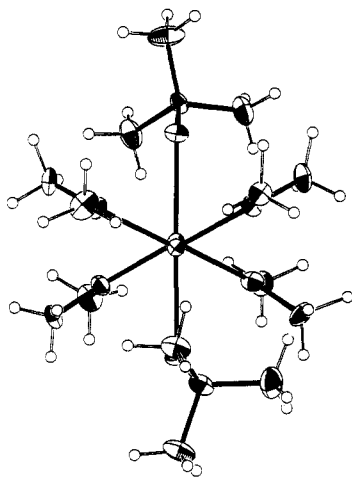


Figure 2. ORTEP diagram of the $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$ molecule as viewed almost down the Mo-Mo bond.

Reactions. $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$ reacts in hydrocarbon solvents with $t\text{-BuSH}$ (≥ 4 equiv) to give the previously characterized compound $\text{Mo}_2(\mu\text{-S})_2(\text{S-}t\text{-Bu})_4(\text{HNMe}_2)_2$. With the alcohols $t\text{-BuOH}$ and $i\text{-PrOH}$, $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$ reacts to give $\text{Mo}_2(\text{OR})_6$ compounds with retention of the $\text{Mo}\equiv\text{Mo}$ bond and elimination of amine and *tert*-butyl mercaptan.

Concluding Remarks

Though the target molecule $\text{Mo}_2(\text{S-}t\text{-Bu})_6(\text{M}\equiv\text{M})$ is not obtained from the reaction between $\text{Mo}_2(\text{NMe}_2)_6$ and $t\text{-BuSH}$, we have now shown that RS ligands can coordinate to the central $(\text{Mo}\equiv\text{Mo})^{6+}$ unit, in the presence of other supporting ligands, in the $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$ molecule.

Experimental Section

General procedures and the preparation of $\text{Mo}_2\text{Cl}_2(\text{NMe}_2)_4$ have been described previously.⁵ Elemental analyses were performed by Canadian Microanalytical Services Ltd., Vancouver, Canada. ^1H NMR spectra were obtained with Varian EM 390 and HR 220 spectrometers. Infrared spectra were obtained from Nujol mulls between CsI plates with a Perkin-Elmer 283 spectrometer.

Lithium mercaptides were prepared from the reactions between $n\text{-BuLi}$ with the appropriate thiols. The latter were distilled and stored over molecular sieves under a nitrogen atmosphere prior to use.

Dry and oxygen-free solvents and atmospheres were used throughout.

Preparation of $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$. $\text{Mo}_2\text{Cl}_2(\text{NMe}_2)_4$ (0.17 g, 0.39 mmol) was dissolved in hexane (30 mL) at room temperature. $\text{LiS-}t\text{-Bu}$ (0.077 g, 0.80 mmol) was added while the solution was stirred, and the reaction mixture was left stirring for 24 h at ca. 35 °C. The solution was filtered to remove finely divided LiCl , and the yellow-orange filtrate was collected and placed in the cold section (ca. -15 °C) of a refrigerator. After several days, yellow-orange crystals of the compound had formed and these were collected by filtration and dried in vacuo (0.154 g, 0.28 mmol, 73% yield based on Mo). An alternative method of purification involved sublimation from the dried filtrate at 95 °C (10^{-4} torr). However, yields were typically lower, ca. 40% based on Mo, because some thermal decomposition accompanies sublimation.

Anal. Calcd for $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$: C, 35.16; H, 7.69; N, 10.26; S, 11.72. Found: C, 34.97; H, 7.60; N, 10.21; S, 10.89.

^1H NMR data (toluene- d_6 , 34 °C, 220 MHz): $\delta(\text{S-}t\text{-Bu})$ 1.40, $\delta(\text{NMe}_2)$ 3.40.

IR data (cm^{-1}): 1260 m, 950 ms, 800 m, 680 wm, 56 ms, 350–360 ms.

[The sparingly soluble nature of the $\text{Mo}_2(\text{SR})_2(\text{NMe}_2)_4$ compounds ($\text{R} = t\text{-Bu, Me}$), especially at low temperatures, has prevented measurement of the barriers to rotation about Mo-N bonds.]

Preparation of $\text{Mo}_2(\text{SMe})_2(\text{NMe}_2)_4$. $\text{Mo}_2\text{Cl}_2(\text{NMe}_2)_4$ (0.312 g, 0.711 mmol) was dissolved in hexane (30 mL) at room temperature. LiSMe (0.08 g, 1.48 mmol) was added, and the reaction mixture was stirred for 24 h at ca. 40 °C. The solution was filtered to remove LiCl , and the yellow-brown filtrate was collected and stripped to

dryness. Purification by sublimation at 95 °C (10^{-4} torr) yielded the yellow-orange solid $\text{Mo}_2(\text{SMe})_2(\text{NMe}_2)_4$, 0.125 g (ca. 40% based on Mo).

Anal. Calcd for $\text{Mo}_2(\text{SMe})_2(\text{NMe}_2)_4$: C, 25.97; H, 6.49; N, 12.12; S, 13.85; Cl, 0.00. Found: C, 25.64; H, 6.24; N, 12.05; S, 13.03; Cl, 0.17.

^1H NMR data (toluene- d_6 , 34 °C): $\delta(\text{SMe})$ 2.4, $\delta(\text{NMe}_2)$ 3.30.

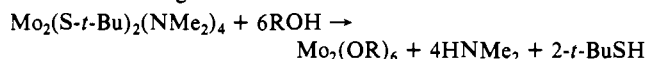
IR data (cm^{-1}): 1260 m, 940–950 s, 810 wm, 670–680 wm, 550 wm, 330–360 wm (broad).

Reaction of $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$ + $t\text{-BuSH}$. $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$ (ca. 0.1 g, ca. 0.183 mmol) was dissolved in hexane and cooled to -78 °C by a CO_2 /acetone bath. Excess $t\text{-BuSH}$ (ca. 0.3 mL, ca. 2.6 mmol) was added via syringe, and the reaction mixture was allowed to warm to room temperature, with stirring. During this time, the color of the solution was observed to change from yellow-orange to red-orange to progressively darker green. After ca. 8 h, dark green crystals had precipitated. They were filtered and characterized by ^1H NMR and IR spectroscopy which indicated that the product is the recently reported compound $\text{Mo}_2(\mu\text{-S})_2(\text{S-}t\text{-Bu})_4(\text{HNMe}_2)_2$.⁴

NMR tube-scale reactions using a deficiency of $t\text{-BuSH}$ showed the volatiles to include HNMe_2 , $t\text{-BuNMe}_2$, and isobutylene.

Reaction of $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$ + $t\text{-BuOH}$ (or $i\text{-PrOH}$). $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$ (0.11 g, ca. 0.201 mmol) was dissolved in 20 mL of hexane and cooled to 0 °C by an ice bath. Excess $t\text{-BuOH}$ (5 mL of a $t\text{-BuOH}$ /benzene azeotrope) was added by syringe. As the reaction was allowed to warm, the color turned red-orange. The reaction was allowed to stir overnight. Solvent and volatiles were removed in vacuo, leaving a red-orange solid. By ^1H NMR and IR spectroscopy, the product appears to be the well-known hexaalkoxide $\text{Mo}_2(\text{O-}t\text{-Bu})_6$. Similar results are seen with $i\text{-PrOH}$, which gave $\text{Mo}_2(\text{O-}i\text{-Pr})_6$.

NMR tube-scale reactions show the volatiles to be dimethylamine and $t\text{-BuSH}$ along with unreacted ROH:



X-ray Structural Determination of $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$. General operating procedures and computational techniques have been described.⁹

A crystal of dimensions 0.28 × 0.28 × 0.24 mm was mounted in a nitrogen-filled glovebag and transferred to the liquid-nitrogen boil-off cold stream of the diffractometer. The cell dimensions, determined from 42 reflections at -160 °C, were $a = 14.322$ (4) Å, $b = 17.514$ (5) Å, $c = 9.865$ (2) Å, $\beta = 101.39$ (1)°, and $V = 2425.7$ (1) Å³; $Z = 4$, $d_{\text{calcd}} = 1.497$ g cm⁻³, and space group $P2_1/n$.

Of the 4448 reflections collected, the number with unique intensities was 3187, while the number with $F > 2.33\sigma(F)$ was 2656. Data were collected by using 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, 4 s; aperture size, 3.0 × 4.0 mm. The limits of data collection were 5° < 2θ < 50°.

The structure was solved by a combination of direct-methods and Fourier techniques. All hydrogen atoms were located and refined. Final full-matrix refinement included isotropic thermal parameters for non-hydrogen atoms, as well as positional parameters and an overall scale factor. A final difference Fourier synthesis was essentially featureless, the largest peak being 0.6 e Å⁻³.

The final residuals are $R(F) = 0.033$ and $R_w(F) = 0.034$. The goodness of fit for the last cycle was 0.843, and the maximum Δ/σ was 0.05.

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Registry No. $\text{Mo}_2(\text{S-}t\text{-Bu})_2(\text{NMe}_2)_4$, 83312-38-3; $\text{Mo}_2(\text{SMe})_2(\text{NMe}_2)_4$, 83312-39-4; $\text{Mo}_2\text{Cl}_2(\text{NMe}_2)_4$, 63301-82-6; $\text{LiS-}t\text{-Bu}$, 16203-42-2; LiSMe , 35638-70-1; $t\text{-BuSH}$, 75-66-1; $t\text{-BuOH}$, 75-65-0; $i\text{-PrOH}$, 67-63-0; Mo, 7439-98-7.

Supplementary Material Available: A listing of structure factor amplitudes (20 pages). Ordering information is given on any current masthead page.

(9) Caulton, K. G.; Huffman, J. C.; Lewis, L. N. *Inorg. Chem.* **1980**, *19*, 2755.