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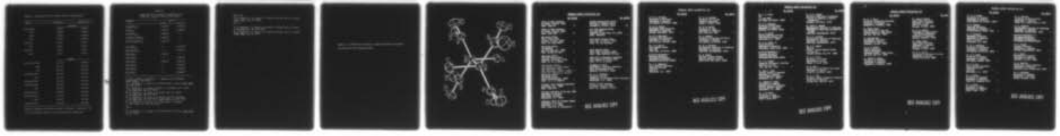

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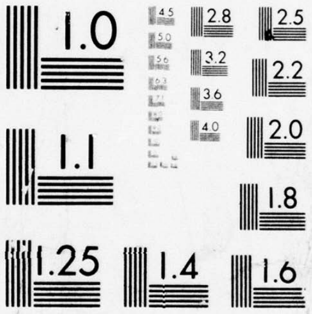

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The Molybdenum-to-Molybdenum Triple Bond. 5. Preparation and Structure of Dimethyltetrakis(dimethylamido)dimolybdenum.

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The reaction of $Mo_2Cl_2(NMe_2)_4$ with methyllithium affords $Mo_2Me_2(MNe_2)_4$, an air-sensitive, yellow crystalline compound. This new compound has been structurally characterized by x-ray crystallography. The crystals are monoclinic, space group $P2_1/c$, with unit cell dimensions of a = 15.342(2)Å, b = 13.578(2)Å, c = 8.264(1)Å, $\beta = 97.26(1)^0$, V = 1707.7(4)$Å^3$ and Z = 4. The asymetric unit consists of two independent dinuclear molecules each lying at a crystallographic center of symmetry. The two molecules are virtually identical and each one has

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virtual C_{2h} symmetry, consistent with a anti rotameric conformation. The following mean distances, averaged over all crystallographically independent ones assuming C_{2h} symmetry in each molecule are: Mo-Mo, 2.201(1)Å; Mo-C, 2.175(6)Å; Mo-N, 1.954(5)Å.

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The Molybdenum-to-Molybdenum Triple Bond. 5. Preparation and Structure of Dimethyltetrakis(dimethylamido)dimolybdenum.[1]

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ABSTRACT

The reaction of $Mo_2Cl_2(NMe_2)_4$ with methyllithium affords $Mo_2Me_2(MNe_2)_4$, an air-sensitive, yellow crystalline compound. This new compound has been structurally characterized by x-ray crystallography. The crystals are monoclinic, space group $P2_1/c$, with unit cell dimensions of $a = 15.342(2)$ Å $b = 13.578(2)$ Å, $c = 8.264(1)$ Å, $\beta = 97.26(1)°$, $V = 1707.7(4)$ Å^3 and $Z = 4$. The asymmetric unit consists of two independent dinuclear molecules each lying at a crystallographic center of symmetry. The two molecules are virtually identical and each one has virtual C_{2h} symmetry, consistent with a anti rotameric conformation. The following mean distances, averaged over all crystallographically independent ones assuming C_{2h} symmetry in each molecule are: Mo-Mo, 2.201(1) Å; Mo-C, 2.175(6) Å; Mo-N, 1.954(5) Å.

INTRODUCTION

The existence of an extensive chemistry centering around the triply-bonded Mo-Mo and W-W units is now well established and has recently been reviewed.[3,4] A firm structural base for interpreting this chemistry has been provided by more than a score of x-ray crystallographic structure determinations. Among these, however, there have been only a few pairs of molybdenum and tungsten compounds with the same or very similar sets of ligands. The comparisons afforded by such pairs are valuable in developing the comparative chemistry of the M-M multiple bonds in the 2nd and 3rd transition series.

To further this structural basis we report here the preparation and structural characterization of dimethyltetrakis(dimethylamido)dimolybdenum, $Mo_2Me_2(NMe_2)_4$. The structure may be compared to that of the close ditungsten analog, $W_2Me_2(NEt_2)_4$, the structure of which has already been reported.[5] The preparation of this new compound was accomplished by taking advantage of our recently published route[6] to substitution products of the readily available $Mo_2(NMe_2)_6$, viz., reaction (1), and then replacing Cl by methyl by means of the methyllithium reagent, reaction (2). At a later time we

$$Mo_2(NMe_2)_6 + 2\ Me_3SiCl \rightarrow Mo_2Cl_2(NMe_2)_4 + 2\ Me_3SiNMe_2 \qquad (1)$$

$$Mo_2Cl_2(NMe_2)_4 + 2\ MeLi \rightarrow Mo_2Me_2(NMe_2)_4 + 2\ LiCl \qquad (2)$$

shall report further studies of reactions of type 2 which have also allowed the isolation of alkyls, such as $M_2Et_2(NMe_2)_4$, for which β-H elimination is not impossible.

RESULTS AND DISCUSSION

Synthesis. - The compounds $M_2Cl_2(NMe_2)_4$, where M = Mo and W, react with ether and tetrahydrofuran: reaction 2 was carried out in toluene. $Mo_2Me_2(NMe_2)_4$ was isolated from 2 as a yellow, crystalline, diamagnetic, air-sensitive compound in greater than 70% yield. It is thermally stable and may be sublimed at 100°C, 10^{-2} torr. In the mass spectrometer a molecular ion, $Mo_2Me_2(NMe_2)_4^+$ together with many other Mo_2-containing ions were observed. Ir, nmr and analytical data are recorded in the Experimental Section.

Structural Results. - $Mo_2(NMe_2)_4Me_2$ is isostructural to $M_2(NMe_2)_4Cl_2$. The asymmetric unit consists of one half of each of two independent dinuclear molecules, each molecule having crystallographically imposed C_i symmetry deviates only slightly from C_{2h}. Atomic thermal and positional parameters are given in Table 1. An ORTEP of molecule 1 is shown in Figure 1. Bond distances and angles are given in Table 2. The atom labelling scheme used for molecule II parallels that used for molecule I with Mo(2), N(3)-N(4), and C(7)-C(12) replacing Mo(1), N(1)-N(2), and C(1)-C(6), respectively.

Discussion of Structure. - The crystal structure of $Mo_2(NMe_2)_4Me_2$ is closely similar to those[6] of $Mo_2(NMe_2)_4Cl_2$ and $W_2(NMe_2)_4Cl_2$. In all of these, the presence of four molecules in a unit cell belonging to the space group $P2_1/c$ does not correspond, as is usually the case, to the molecule being the asymmetric unit, with the four molecules then being related by the two types of crystallographic inversion center at 0,0,0 and 1/2,0,1/2. In these structures the asymmetric unit is composed of halves of two different molecules. This means that there are two crystallographically independent molecules, each type located on a crystallographic center of inversion. However, as Table II shows, the differences between the two molecules of $Mo_2(NMe_2)_4Me_2$ are completely insignificant, not only chemically but terms of the statistical validity of the differences.

The structure takes the form of the anti rotamer, as has been the case in every $M_2(NR_2)_4X_2$ structure so far examined. The Mo-Mo distance is entirely typical for $\mathrm{Mo}\overset{3^{\circ}}{\text{—}}\mathrm{Mo}$ bonds in compounds of this class, as Table III makes clear. The mean Mo-N distance, 1.954(5)Å, is not significantly different from those found in other structures of the $Mo_2(NR_2)_{6-n}X_n$ type, which have always been in the range 1.94-1.97Å. It is interesting that once again the Mo-X and W-X distances in molecules where n = 2 are practically identical. Thus, for example, in the two $M_2(NMe_2)_4Cl_2$ molecules[6] the Mo-Cl and W-Cl distances are 2.348(5)Å and 2.329(5)Å. The Mo-C distance found here, 2.175(6)Å, is indistinguishable from that in $W_2Me_2(NEt_2)_4$, namely 2.171(11)Å.

EXPERIMENTAL SECTION

General chemical procedures and the preparation of $Mo_2Cl_2(NMe_2)_4$ have been described previously.[6]

Preparation. - Methyllithium (8.34 mmol) in ether (4.53 mL) was placed in a dried, round-bottom flask (100 mL) under a nitrogen atmosphere. The ether was removed *in vacuo*. The resulting white solids (MeLi) and $MoCl_2(NMe_2)_4$ (4.17 mmol) were then dissolved in toluene (40 mL). The solution so formed was initially cooled to *ca*. 0°C (ice-bath) for 1/2 h and then warmed to room temperature for 1 1/2 h with stirring. The solvent was stirred and the solids dried at 25°C, 10^{-2} torr for 1 h. Hexane (40 mL) was added and the solution was filtered under a nitrogen atmosphere using standard Schlenk techniques. The yellow filtrate was collected, reduced in volume to *ca* 8 mL, and coded to *ca*. -10°C, yielding a yellow crystalline product $Mo_2Me_2(NMe_2)_4$ (1.20 g; 73% yield based on eq. 2), which was collected by filtration and dried in vacuum (25°C, 10^{-2} torr). Anal. Calcd for $C_{10}H_{30}N_4Mo_2$: C, 30.16; H, 7.59; N, 14.07. Found: C, 29.9; M, 7.46; N, 13.8. ^1H nmr data, 60 MHz, 40°C, toluene-d_8 solvent: $\delta(Me) = 1.17$, $\delta(NMe_2) = 3.29$ (δ in ppm rel TMS). Ir data, nujol mull, CsI plates (1500-200 cm^{-1} region): (1422(w), 1310(w), 1242(m), 1172(w), 1150(m), 1122(w), 1042(m), 952(s), 940(vs), 590(m), 500(s), 335(m,br).

X-Ray Crystallography. - A crystal of $Mo_2(NMe_2)_4Me_2$ measuring *ca*. 0.2 x 0.25 x 0.4 mm was mounted in a thin-walled glass capillary embedded in epoxy resin with the major crystal axis nearly coincident with the ϕ axis of the goniometer. Crystal quality was checked with ω-scans of several intense low-angle reflections which had peak widths at half-height of *ca*. 0.2°. Cell constants and axial photographs indicated that the crystal belonged to the monoclinic system with $a = 15.342(2)$Å, $b = 13.578(2)$Å, $c = 8.264(1)$Å, $\beta = 97.26(1)°$, $V = 1707.7(4)$Å^3. The observed volume is consistent with that expected for Z = 4.

Data were collected at 22±2°C on a Syntex P$\bar{1}$ autodiffractometer equipped with a graphite crystal monochromator in the incident beam and using MoKα (λ = 0.710730Å) radiation. The θ-2θ scan technique was used with scans ranging from 1.1° above and 1.1° below the calculated $K\alpha_1$, $K\alpha_2$ doublet, variable scan speeds of from 4.0 to 24.0°/min and with a scan to background time ratio of 2.0. The intensities of three standard reflections were monitored frequently throughout data collection and showed no decrease in intensity. The integrated intensities of 2240 unique, non-systematically absent reflections having $0° < 2\theta < 45°$ were recorded. The data were reduced to a set of relative $|F_o|^2$ values and were not corrected for absorption (μ = 14.3 cm^{-1}). The 1716 reflections having $|F_o|^2 > 3\sigma(|F_o|^2)$ were used in subsequent structure solution and refinement. Systematic absences on 0k0 (k=2n+1) and h0ℓ (ℓ = 2n+1) uniquely determined the space group to be $P2_1/c$ (No. 14).

Since the cell constants of $Mo_2(NMe_2)_4Me_2$ were very similar to those of $Mo_2(NMe_2)_4Cl_2$ and both compounds crystallized in the same space group, the starting atomic positions for $Mo_2(NMe_2)_4Me_2$ were taken from the $Mo_2(NMe_2)_4Cl_2$ structure,[6] assuming Mo-Me = Mo-Cl. The positions were sufficiently close to allow refinement to proceed smoothly to convergence. In the final stages of refinement anisotropic thermal parameters were used for all atoms. The final unweighted and weighted residuals were R_1 = 0.037 and R_2 = 0.060, respectively. A value of 0.07 was used for p in the calculation of the weights. The esd of an observation of unit weight was 1.412. The largest peaks in a final difference Fourier map were about where some methyl-group hydrogen atoms might be expected, but no attempt was made to introduce or refine these.

A table of observed and calculated structure factor tables (3 pages) is available as supplementary material. Ordering instructions will be found on any current masthead page.

Acknowledgements

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Table I. POSITIONAL AND THERMAL PARAMETERS AND THEIR ESTIMATED STANDARD DEVIATIONS.[a]

Atom	X	Y	Z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo(1)	0.45070(4)	0.05707(5)	0.46198(8)	0.00292(3)	0.00341(3)	0.0109(1)	0.00098(5)	0.00134(9	0.0007(1)
Mo(2)	-0.02775(4)	0.07171(5)	0.02880(8)	0.00299(3)	0.00361(3)	0.0128(1)	-0.00063(5)	0.00145(10	-0.0014(1)
N(1)	0.3605(4)	0.0385(5)	0.6069(8)	0.0031(3)	0.0058(4)	0.014(1)	-0.0002(6)	0.001(1)	-0.001(1)
N(2)	0.5181(5)	0.1797(5)	0.4644(8)	0.0049(4)	0.0040(4)	0.015(1)	0.0001(6)	0.001(1)	0.001(1)
N(3)	0.0732(5)	0.1602(5)	0.0640(9)	0.0050(4)	0.0039(4)	0.018(1)	-0.0020(6)	0.001(1)	-0.003(1)
N(4)	-0.1224(4)	0.0931(5)	-0.1481(8)	0.0030(3)	0.0054(4)	0.014(1)	-0.0002(6)	-0.002(1)	-0.000(1)
C(1)	0.4037(5)	0.0110(7)	0.214(1)	0.0039(4)	0.0066(6)	0.012(1)	-0.0004(8)	-0.002(1)	-0.002(1)
C(2)	-0.0798(6)	0.0370(7)	0.255(1)	0.0050(5)	0.0076(6)	0.016(1)	0.0008(9)	0.007(1)	-0.001(2)
C(3)	0.3548(6)	-0.0209(8)	0.752(1)	0.0049(5)	0.0084(6)	0.018(2)	0.0003(10)	0.006(1)	0.008(2)
C(4)	0.2888(6)	0.1139(7)	0.590(1)	0.0032(4)	0.0077(6)	0.023(2)	0.0032(8)	0.001(1)	-0.005(2)
C(5)	0.6087(6)	0.2072(7)	0.523(1)	0.0049(5)	0.0065(6)	0.022(2)	-0.0034(9)	0.000(2)	-0.001(2)
C(6)	0.4637(7)	0.2666(7)	0.424(1)	0.0087(6)	0.0039(5)	0.029(2)	0.0039(10)	0.006(2)	0.005(2)
C(7)	0.1688(6)	0.1489(8)	0.050(1)	0.0032(4)	0.0080(7)	0.026(2)	-0.0034(8)	0.003(1)	-0.000(2)
C(8)	0.0523(7)	0.2645(7)	0.086(1)	0.0072(6)	0.0040(5)	0.030(2)	-0.0014(9)	-0.001(2)	-0.005(2)
C(9)	-0.1507(6)	0.0454(8)	-0.306(1)	0.0054(5)	0.0078(6)	0.017(2)	0.0013(10)	-0.002(2)	-0.004(2)
C(10)	-0.1733(6)	0.1866(7)	-0.134(1)	0.0055(5)	0.0065(6)	0.028(2)	0.0033(9)	0.001(2)	-0.000(2)

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[a] 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 II. Bond Distances (Å) and Angles (Deg) for $Mo_2(NMe_2)_4Me_2$.[a]

Atoms	Molecule I	Molecule II
	Distances	
Mo(1)-Mo(1)'	2.201(1)	2.201(1)
-N(1)	1.958(6)	1.952(6)
-N(2)	1.959(6)	1.948(6)
-C(1)	2.173(7)	2.176(8)
N(1)-C(3)	1.46(1)	1.49(1)
-C(4)	1.50(1)	1.47(1)
N(2)-C(5)	1.46(1)	1.47(1)
-C(6)	1.46(1)	1.50(1)
	Angles	
Mo(1)'-Mo(1)-N(1)	104.4(2)	104.8(2)
-N(2)	104.7(2)	104.0(2)
-C(1)	101.6(2)	100.9(2)
N(1)-Mo(1)-N(2)	121.1(2)	121.4(3)
-C(1)	111.3(3)	112.1(3)
N(2)-Mo(1)-C(1)	111.5(3)	111.0(3)
Mo(1)-N(1)-C(3)	134.0(5)	133.9(5)
-C(4)	115.1(5)	115.5(5)
C(3)-N(1)-C(4)	109.6(6)	109.9(6)
Mo(1)-N(2)-C(5)	134.4(5)	135.1(5)
-C(6)	113.5(5)	114.5(5)
C(5)-N(2)-C(6)	111.2(7)	110.0(6)

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[a] Atoms are labelled as shown for Molecule I in Figure 1. Molecules I and II lie on inversion centers at 1/2,0,1/2 and 0,0,0, respectively.

TABLE III

Lengths of M-M Triple Bonds in Compounds of the Types M_2L_6, $M_2L_4X_2$ and Some of their Adducts

Compound	M-M, Å M = Mo	 M = W
$M_2(CH_2SiMe_3)_6$	2.167(?)[a]	2.255(2)[b]
$M_2(NMe_2)_6$	2.214(3)[c]	2.294(2)[d]
$M_2(OCH_2CMe_3)_6$	2.222(2)[e]	---
$M_2(OSiMe_3)_6(NHMe_2)_2$	2.242(1)[f]	---
$M_2(OBu^t)_4(O_2COBu^t)_2$	2.241(1)[g]	---
$M_2(O_2CNMe_2)_6$	---	2.279(1)[h]
$M_2Me_2(O_2CNEt_2)_4$	---	2.272(1)[h]
$M_2Me_2(NMe_2)_4$	2.201(1)[i]	---
$M_2Me_2(NEt_2)_4$	---	2.291(1)[j]
$M_2Cl_2(NMe_2)_4$	2.201(2)[k]	2.285(2)[k]
$M_2Cl_2(NEt_2)_4$	---	2.301(1)[l]
$M_2Br_2(NEt_2)_4$	---	2.301(2)[m]
$M_2I_2(NEt_2)_4$	---	2.300(4)[m]

---

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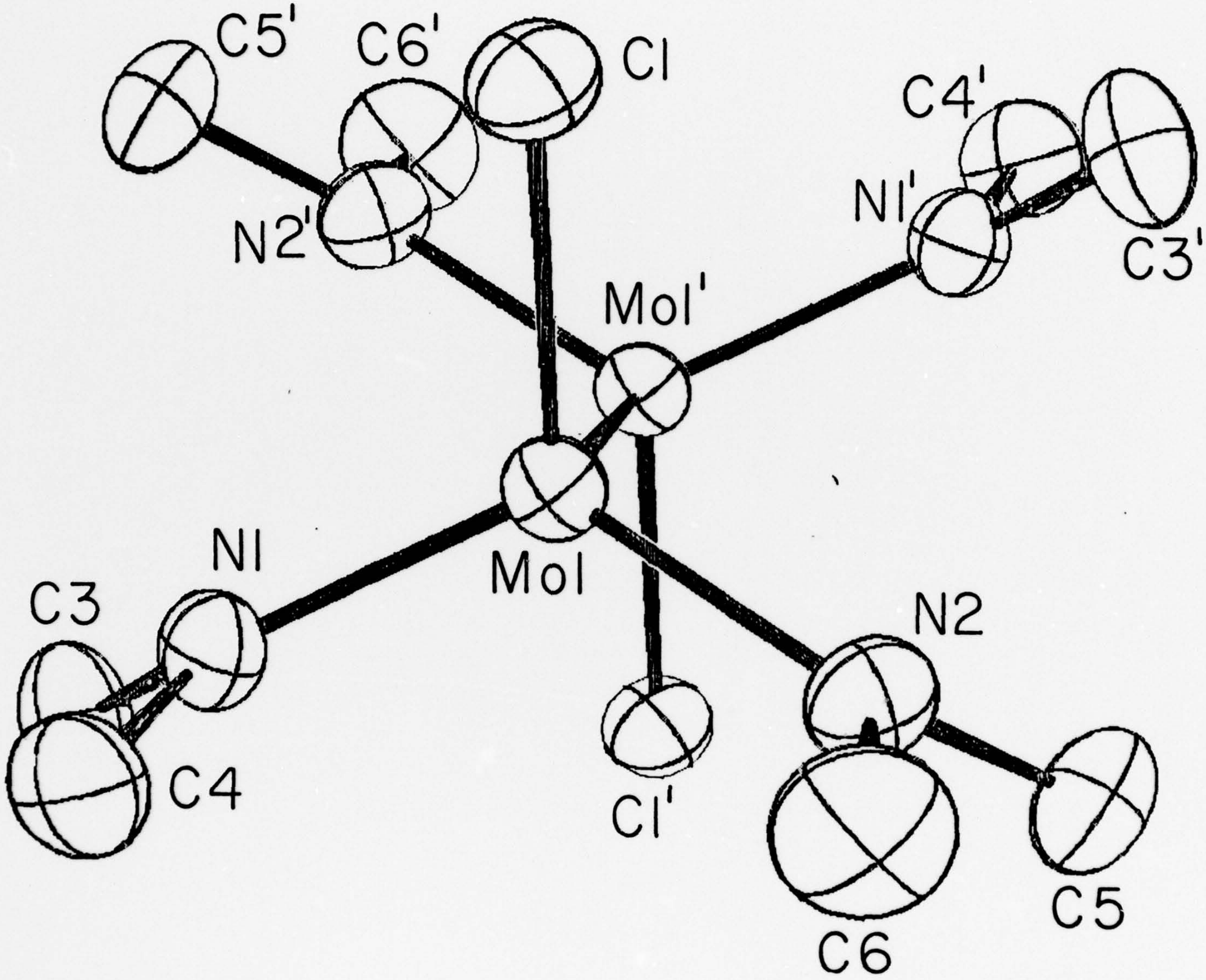
Figure 1. An ORTEP view of Molecule I using 50% probability ellipsoids and showing the atom labelling scheme.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