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THE MOLYBDENUM-MOLYBDENUM TRIPLE BOND. 9. BIS(1,3-DI-P-TOLYLTRI--ET

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THE MOLYBDENUM-MOLYBDENUM TRIPLE BOND. 9.

BIS(1,3-di-p-TOLYLTRIAZENIDO)BIS(DIMETHYLAMIDO)DIMETHYLDIMOLYBDENUM.

by

M.H. Chisholm, D.A. Haitko, J.C. Huffman and K. Folting

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) 1,2-Mo ₂ Me ₂ (NMe ₂) ₄ reacts in hydrocarbon solvents with 1,3-di-p-tolyl-triazine, C ₇ H ₈ NNNHC ₇ H ₈ , to give the title compound as an red crystalline solid. An X-ray study shows that in the solid state, the molecule has a crystallographically imposed C ₂ axis of symmetry. The Mo≡Mo bond, 2.174(1) Å, is bridged by a cis-pair of triazenido ligands which afford sufficient		

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THE MOLYBDENUM-MOLYBDENUM TRIPLE BOND. 9.¹

BIS(1,3-di-p-TOLYLTRIAZENIDO)BIS(DIMETHYLAMIDO)DIMETHYLDIMOLYBDENUM.

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Abstract

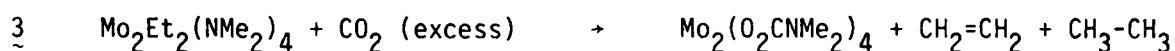
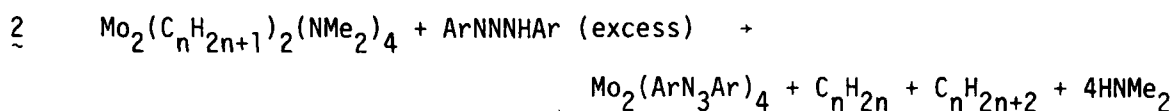
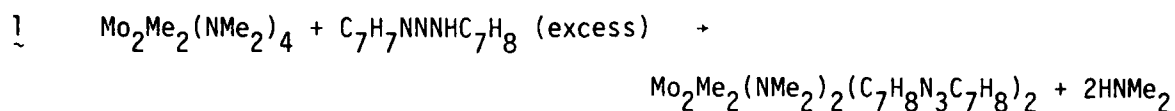
1,2-Mo₂Me₂(NMe₂)₄ reacts in hydrocarbon solvents with 1,3-di-p-tolyltriazine, C₇H₈NNHC₇H₈, to give the title compound as a red crystalline solid. An X-ray study shows that in the solid state, the molecule has a crystallographically imposed C₂ axis of symmetry. The Mo≡Mo bond, 2.174(1) Å, is bridged by a cis-pair of triazenido ligands which afford sufficient flexibility to allow a non-eclipsed geometry. Each molybdenum atom is coordinated to three nitrogen atoms and one carbon atom which roughly lie in a plane. Pertinent bond distances are Mo-C (methyl) = 2.193(4) Å, Mo-N (dimethylamido) = 1.948(3) Å, Mo-N (triazinido) = 2.157(3) and 2.283(3) Å, with the longer distance associated with the Mo-N bond which is trans to the Mo-CH₃ bond. These observations are compared with other findings in dimolybdenum and ditungsten chemistry (M≡M). Crystal data for Mo₂Me₂(NMe₂)₂-(C₇H₈N₃C₇H₈)₂ are a = 21.608(5) Å, b = 9.440(2) Å, c = 24.076(6) Å, β = 135.49(1)°, V = 3442.5(1) Å³, Z = 4, d_{calcd} = 1.464 g cm⁻³ and space group C2/c.

Introduction

An extensive coordination chemistry surrounds the $(M \equiv M)^{6+}$ unit for molybdenum and tungsten. The metal atoms may be coordinated to 3, 4, 5 or 6 ligand atoms and for a given coordination number, there appears to be a preference for a specific geometry.² The data set is, however, still very limited and caution must be applied against any wish to state rules for these systems. We are, however, trying to establish trends in coordination preferences and reactivity patterns. We report here the preparation and structure of $Mo_2Me_2(NMe_2)_2(C_7H_8N_3C_7H_8)_2$. The structure of the closely related compound $Mo_2(NMe_2)_4(C_7H_8N_3C_7H_8)_2$ was previously reported.⁴

Results and Discussion

Synthesis. In hydrocarbon solvents $Mo_2Me_2(NMe_2)_4$ ⁵ and 1,3-di-p-tolyl-triazine react upon mixing at room temperature to give red solutions from which $Mo_2Me_2(NMe_2)_2(C_7H_8N_3C_7H_8)_2$ can be obtained as red crystals. Even in the presence of excess triazine, no further displacement of dimethylamido ligands occurs. The reaction shown below in equation 1 appears stoichiometric and irreversible. This contrasts with the reactions between 1,3-diaryl-triazines (phenyl and p-tolyl) and $Mo_2R_2(NMe_2)_4$ compounds, where $R = Et$ and Pr^i , which react according to equation 2.⁶ Here by reductive elimination of alkene and alkane, and further replacement of dimethylamido groups by triazenido ligands, the Mo-to-Mo triple bond is transformed to a quadruple bond.⁷ In the related reaction between $Mo_2Et_2(NMe_2)_4$ and CO_2 , which proceeds according to equation 3,⁸ an intermediate, $Mo_2Et_2(NMe_2)_2(O_2CNMe_2)_2$, has been detected by 1H nmr spectroscopic studies.⁶ The structure of the $Mo_2Me_2(NMe_2)_2(C_7H_8N_3C_7H_8)_2$ molecule was thus of particular interest to us.



Solid State Structure. In the crystalline state, the compound is composed of discrete molecules of $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$. An ORTEP view of the molecule looking down the C_2 axis of symmetry and giving the atom numbering scheme used in the tables is shown in Figure 1. A view of the molecule looking down the Mo-Mo bond is shown in Figure 2. Final atomic coordinates and thermal parameters are given in Table 1 and 2, respectively. Complete listings of bond distances and angles are given in Tables 3 and 4, respectively. Pertinent torsional angles are given in Table 5. Tables listing a number of least squares planes calculated for this molecule and the deviations of atoms from these planes, together with dihedral angles between planes, are available as supplementary material.

The Mo-to-Mo distance is $2.174(1) \text{ \AA}$, which is crystallographically significantly shorter than that in either the parent compound⁵ $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_4$, $2.201(1) \text{ \AA}$, or the closely related compound $\text{Mo}_2(\text{NMe}_2)_4(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$, $2.212(1) \text{ \AA}$.⁴ Little chemical significance can be attributed to such a small variation in Mo-to-Mo distance, however, and the observed distance falls within the previously observed distances associated with compounds containing a central $(\text{Mo}\equiv\text{Mo})^{6+}$ unit.⁹ The Mo-C (methyl) and Mo-N (dimethylamido) distances are essentially identical to those found for $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_4$.

A comparison with the $\text{Mo}_2(\text{NMe}_2)_4(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ structure is interesting. In the latter, the triazenido ligands are bidentate, but not bridging. The four nitrogen atoms bonded to each molybdenum atom also lie roughly within a plane and a staggered conformation with respect to each end of the molecule is achieved by the interlocking of the Mo-NC_2 blades which are preferentially aligned along the Mo-Mo axis in order to maximize $\text{N p-to-Mo d } \pi$ -bonding. In the $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ structure, the replacement of one NMe_2 ligand by the less sterically demanding Me ligand allows the triazenido ligands to act as bridging bidentate ligands without introducing an eclipsed configuration. The observed skew geometry, which is revealed in the view of the molecule shown in Figure 2 and in the torsional angles listed in Table 5, is quite reconcilable with steric considerations. The only metal-ligand distances, which may reveal the influence of electronic factors, are the two Mo-N (triazenido) distances. The Mo-N distance trans to the Mo-NMe_2 bond is longer by more than $0.1 \text{ \AA}$ than that trans to the Mo-CH_3 bond. This could be attributable to a trans influence phenomenon which is typically found in square planar platinum(II) compounds¹⁰ and could similarly operate in dimetallic systems where metal-ligand bonding utilizes essentially sp^2d hybrid atomic orbitals, e.g. as in $(\text{Mo}\equiv\text{Mo})^{4+}$ containing compounds.¹¹ However, in $(\text{Mo}\equiv\text{Mo})^{6+}$ containing compounds, the molybdenum atoms have five atomic orbitals available for forming bonds in the xy plane (s , p_x , p_y , d_{xy} and $d_{x^2-y^2}$) and can indeed form five bonds in a pentagonal plane as is seen in the structure of the $\text{W}_2\text{Me}_2(\text{O}_2\text{CNEt}_2)_4$ molecule.¹² An inspection of the angles subtended from molybdenum to the ligating atoms in the $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ molecule shows the trans-angles N-Mo-N and C-Mo-N are $164.1(1)^\circ$ and $146.7(1)^\circ$, respectively. While the former may reasonably be called a

trans-angle, the latter much smaller angle of $146.7(1)^\circ$ cannot. Thus, it seems that with the availability of five molybdenum atomic orbitals, the methyl group and triazenido nitrogen donor are not forced into mutual competition and the high trans-influence which is associated with the methyl ligand¹⁰ can be obviated.

¹H nmr data for $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ are given in the Experimental Section and are consistent with the maintenance in solution of a structure akin to that found in the crystal.

Concluding Remarks

The present work provides an additional observation that the $(\text{Mo}\equiv\text{Mo})^{6+}$ unit will readily coordinate four ligand atoms to each molybdenum atom in a planar manner. The geometry with respect to each end of the molecule is reconcilable with steric considerations and consistent with the view that the $\text{Mo}\equiv\text{Mo}$ bond, $\sigma^2\pi^4$, is cylindrical. For compounds in which the molybdenum atoms are coordinated to four ligand atoms, the overall geometry with respect to each end of the molecule may be eclipsed, as is seen for $\text{Mo}_2(\text{OBu}^t)_2-(\text{O}_2\text{COBu}^t)_2$ ¹³ and $\text{Mo}_2(\text{O}_2\text{P}(\text{O})\text{OH})_4^{2-}$,¹⁴ partially staggered as in $\text{Mo}_2(\text{OSiMe}_3)_6-(\text{HNMe}_2)_2$ ¹⁵ and $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ or fully staggered as in $\text{Mo}_2(\text{NMe}_2)_4-(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$.⁴ In all of these compounds, the four ligand atoms coordinated to each molybdenum lie roughly in a square plane: coordination along the M-M axis is not found. The latter may not be favored because it would weaken the M-M σ -bond. Cotton and his coworkers¹⁶ have shown this to be the case for the $(\text{Mo}\equiv\text{Mo})^{4+}$ moiety.

It is likely that $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ and $\text{Mo}_2\text{Et}_2(\text{NMe}_2)_2-(\text{O}_2\text{CNEt}_2)_2$ compounds, which are intermediates in reactions 2 and 3, have similar ground state geometries to that of $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$.

Further studies are being directed toward characterizing the reactivity patterns of $\text{Mo}_2\text{R}_2(\text{NMe}_2)_2(\text{chelate})_2$ compounds.

Experimental Section

General procedures and the preparation of $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_4$ have been described.⁵ Note the use of dry and oxygen-free solvents and atmospheres.

Preparation of $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$

All manipulations were carried out in a Vacuum Atmospheres Dri Box facility equipped with a self-contained cold storage box maintained at -30°C .

In a typical preparation, $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_4$ (0.38 g, 0.95 mmol) was added to a 5 mL round-bottomed flask containing toluene (30 mL). The solution changed immediately in color from golden, characteristic of $1,2\text{-Mo}_2\text{R}_2(\text{NMe}_2)_4$ compounds, to an intense red as p-tolyltriazene (0.49 g, 2.3 mmol) was added with stirring. Hexane (ca. 10 ml) was then added and the flask was placed in the refrigeration unit of the dry-box. After 4 days, the solution yielded ruby-red crystals, ca. 0.4 g, which were collected and used for the X-ray crystallographic analysis. Satisfactory elemental analyses were not obtained. This situation was also found for $\text{Mo}_2(\text{NMe}_2)_4(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ and we believe results from solvent inclusion into some, but not all crystals. [In this X-ray study, no solvent molecule was present in the unit cell].

^1H nmr data obtained in benzene- d_6 at 220 MHz at 20°C : δ (Mo- CH_3) = 1.50, δ (p-tolyl- CH_3) = 2.03, 2.19 in the integral ratio 1:1; δ (NMe-distal) = 2.53; δ (NMe-proximal) = 4.13. δ in the ppm relative to Me_4Si .

X-Ray Structural Determination. A crystal of dimensions 0.18 x 0.20 x 0.21 mm was mounted in a nitrogen-filled glove-bag and transferred to the liquid-nitrogen boil-off system of the diffractometer.¹⁷ The diffractometer utilized for data collection was designed and constructed locally. A Picker four-circle goniostat equipped with a Furnas Monochromator (HOG crystal) and Picker X-ray generator is interfaced to a TI 980 minicomputer with Slo-syn stepping motors to drive the angles. Centering is accomplished using top/-bottom--left/right slit assemblies. The minicomputer is interfaced by low-speed data lines to a CYBER 172-CDC 6600 multimain system where all computations are performed.

The cell dimensions obtained from 46 reflections at -170°C with $\text{MoK}\alpha$ ($\lambda = 0.71069 \text{ \AA}$) were $a = 21.608(5) \text{ \AA}$, $b = 9.440(2) \text{ \AA}$, $c = 24.076(6) \text{ \AA}$, $\beta = 135.49(1)^{\circ}$, $V = 3442.5(1) \text{ \AA}^3$, $Z = 4$, $d_{\text{calcd}} = 1.464 \text{ g cm}^{-3}$ and space group = $C2/c$.

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

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 delta/sigma for the last cycle was 0.65.

All of the hydrogen atoms were located and refined using isotropic thermal parameters. All non-hydrogen atoms were refined anisotropically.

Acknowledgement. We thank the Office of Naval Research, the Marshall H. Wrubel Computing Center and the taxpayers of the State of Indiana for financial support of this work.

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Table 1. Fractional Coordinates for $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$.

Note 1: The isotropic thermal parameters listed for those atoms refined anisotropically are the isotropic equivalent.

Note 2: Number in parentheses in this and all following tables refer to the error in the last significant digits.

Note 3: 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.

Mo(1)	4959.8(2)	3483.4(3)	2023.4(1)	13
N(2)	5432(2)	1695(3)	2053(2)	19
C(3)	5315(3)	1419(4)	1387(3)	27
C(4)	5802(3)	449(4)	2558(3)	26
C(5)	3584(2)	2926(4)	963(2)	20
N(6)	4199(2)	5558(3)	1628(1)	13
N(7)	3834(2)	5927(3)	1856(1)	14
N(8)	4015(2)	5061(3)	2384(1)	14
C(9)	3998(2)	6484(3)	1050(2)	16
C(10)	4229(2)	6053(4)	665(2)	17
C(11)	4076(2)	6925(4)	115(2)	20
C(12)	3680(2)	8239(4)	-80(2)	20
C(13)	3449(2)	8659(4)	305(2)	20
C(14)	3607(2)	7805(4)	864(2)	19
C(15)	3520(3)	9189(5)	-676(2)	26
C(16)	3688(2)	5538(3)	2699(2)	14
C(17)	3431(2)	4533(3)	2920(2)	15
C(18)	3188(2)	4940(4)	3298(2)	17
C(19)	3208(2)	6351(3)	3471(2)	17
C(20)	3447(2)	7349(4)	3226(2)	19
C(21)	3680(2)	6960(4)	2846(2)	18
C(22)	2992(3)	6801(4)	3917(2)	23
H(23)	5010(21)	2201(38)	1027(19)	14(7)
H(24)	5886(25)	1188(37)	1566(22)	23(8)
H(25)	5010(27)	594(48)	1139(24)	34(9)
H(26)	5420(24)	-359(41)	2265(20)	22(7)
H(27)	6314(25)	232(38)	2713(21)	23(8)
H(28)	5880(25)	650(44)	3000(23)	31(9)
H(29)	3158(24)	3292(37)	919(21)	21(8)
H(30)	3457(26)	3325(43)	552(24)	31(9)
H(31)	3523(24)	1991(48)	898(22)	27(8)
H(32)	4466(20)	5176(34)	776(18)	8(6)
H(33)	4234(24)	6625(38)	-105(22)	25(8)
H(34)	3182(21)	9523(39)	185(19)	14(7)
H(35)	3461(20)	8080(35)	1114(19)	12(7)
H(36)	4061(27)	9622(42)	-442(22)	28(8)
H(37)	3340(31)	8757(50)	-1066(29)	47(12)
H(38)	3147(32)	9822(55)	-846(28)	48(13)
H(39)	3444(21)	3589(34)	2812(19)	14(6)
H(40)	3005(20)	4291(35)	3419(18)	11(6)
H(41)	3456(22)	8235(40)	3326(20)	17(7)
H(42)	3846(22)	7540(38)	2724(20)	20(8)
H(43)	3504(30)	7269(50)	4399(28)	46(11)
H(44)	2469(24)	7337(37)	3566(21)	20(7)
H(45)	2880(27)	6031(46)	4040(24)	29(9)

Table 2. Anisotropic Thermal Parameters for $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$.^a

Atom	U11	U22	U33	U12	U13	U23
Mo(1)	197(2)	127(2)	198(2)	-17(1)	158(1)	-20(1)
N(2)	281(15)	183(14)	375(16)	-33(12)	273(14)	-56(13)
C(3)	444(23)	266(21)	479(23)	-96(20)	382(21)	-149(20)
C(4)	314(20)	173(18)	555(26)	11(16)	326(20)	-42(18)
C(5)	251(18)	263(20)	253(18)	-84(15)	184(16)	-67(15)
N(6)	181(13)	157(13)	184(12)	-9(11)	141(11)	4(11)
N(7)	198(13)	166(13)	195(13)	-5(11)	145(12)	2(11)
N(8)	205(13)	161(13)	210(13)	26(10)	162(12)	38(11)
C(9)	195(15)	191(15)	179(14)	-36(14)	124(14)	-20(13)
C(10)	232(17)	209(17)	202(16)	21(14)	155(15)	10(13)
C(11)	275(18)	322(19)	202(16)	-56(15)	167(16)	-32(14)
C(12)	249(17)	278(19)	172(15)	-73(14)	132(14)	1(14)
C(13)	282(18)	175(17)	263(17)	-0(15)	180(16)	26(14)
C(14)	290(18)	227(17)	242(17)	-2(14)	209(16)	3(14)
C(15)	397(23)	320(22)	267(20)	-43(20)	242(19)	37(18)
C(16)	176(15)	202(15)	163(15)	25(14)	118(14)	10(14)
C(17)	203(15)	144(15)	193(15)	-1(13)	135(14)	-0(13)
C(18)	233(17)	218(17)	230(16)	-2(13)	176(15)	41(13)
C(19)	209(16)	272(18)	200(15)	28(14)	154(14)	9(14)
C(20)	286(18)	180(18)	282(18)	6(14)	208(16)	-29(14)
C(21)	282(18)	149(16)	306(18)	-10(14)	230(16)	22(14)
C(22)	342(21)	316(21)	317(20)	16(17)	268(19)	-19(17)

All U's are $\times 10^{-4}$

^aThe form of the exponent is: $\exp[-2\pi^2(U_{11}h^2a^{*2} + \dots + 2U_{12}hka^*b^* + \dots)]$.

Table 3. Bond Distances, in Angstroms,
for the $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ Molecule.^a

A	B	Distance
Mo(1)	Mo(1)'	2.174(1)
Mo(1)	N(2)	1.948(3)
Mo(1)	N(6)	2.283(3)
Mo(1)'	N(8)'	2.157(3)
Mo(1)	C(5)	2.193(4)
N(2)	C(3)	1.457(5)
N(2)	C(4)	1.454(5)
N(6)	N(7)	1.290(3)
N(6)	C(9)	1.425(4)
N(7)	N(8)	1.314(3)
N(8)	C(16)	1.428(4)
C(9)	C(10)	1.391(4)
C(9)	C(14)	1.389(5)
C(10)	C(11)	1.383(5)
C(11)	C(12)	1.386(5)
C(12)	C(13)	1.389(5)
C(12)	C(15)	1.507(5)
C(13)	C(14)	1.385(5)
C(16)	C(17)	1.383(4)
C(16)	C(21)	1.392(5)
C(17)	C(18)	1.388(4)
C(18)	C(19)	1.387(5)
C(19)	C(20)	1.391(5)
C(19)	C(22)	1.505(5)
C(20)	C(21)	1.373(5)

^aBond distances to hydrogen atoms are given in the supplementary data.

Table 4. Bond Angles, in Degrees,
for the $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ Molecule.^a

A	B	C	Angle
Mo(1)'	Mo(1)	N(2)	108.4(1)
Mo(1)'	Mo(1)	N(6)	86.1(1)
Mo(1)'	Mo(1)	N(8)'	91.7(1)
Mo(1)'	Mo(1)	C(5)	104.5(1)
N(2)	Mo(1)	N(6)	164.1(1)
N(2)	Mo(1)	N(8)'	108.0(1)
N(2)	Mo(1)	C(5)	94.3(1)
N(6)	Mo(1)	N(8)'	77.2(1)
N(6)	Mo(1)	C(5)	75.2(1)
N(8)'	Mo(1)	C(5)	146.7(1)
Mo(1)	N(2)	C(3)	116.1(2)
Mo(1)	N(2)	C(4)	133.8(2)
C(3)	N(2)	C(4)	109.6(3)
Mo(1)	N(6)	N(7)	122.1(2)
Mo(1)	N(6)	C(9)	124.1(2)
N(7)	N(6)	C(9)	113.4(2)
N(6)	N(7)	N(8)	113.1(2)
Mo(1)'	N(8)	N(7)	121.8(2)
Mo(1)'	N(8)	C(16)	122.0(2)
N(7)	N(8)	C(16)	113.4(2)
N(6)	C(9)	C(10)	117.9(3)
N(6)	C(9)	C(14)	123.8(3)
C(10)	C(9)	C(14)	118.3(3)
C(9)	C(10)	C(11)	120.6(3)
C(10)	C(11)	C(12)	121.8(3)
C(11)	C(12)	C(13)	117.2(3)
C(11)	C(12)	C(15)	121.5(3)
C(13)	C(12)	C(15)	121.2(3)
C(12)	C(13)	C(14)	121.7(3)
C(9)	C(14)	C(13)	120.5(3)
N(8)	C(16)	C(17)	118.3(3)
N(8)	C(16)	C(21)	122.7(3)
C(17)	C(16)	C(21)	118.8(3)
C(16)	C(17)	C(18)	120.3(3)
C(17)	C(18)	C(19)	121.2(3)
C(18)	C(19)	C(20)	117.6(3)
C(18)	C(19)	C(22)	121.8(3)
C(20)	C(19)	C(22)	120.5(3)
C(19)	C(20)	C(21)	121.6(3)
C(16)	C(21)	C(20)	120.3(3)

^aBond angles involving hydrogen atoms are given in the supplementary data.

Table 5. Torsional Angles, in Degrees,
for the $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ Molecule.

(a) Along the Mo(1) - Mo(1)' Bond

N(2)	Mo(1)	Mo(1)'	C(5)'	51.66
N(2)	Mo(1)	Mo(1)'	N(6)'	125.31
N(2)	Mo(1)	Mo(1)'	N(8)	-157.67
C(5)	Mo(1)	Mo(1)'	N(6)'	-135.05
C(5)	Mo(1)	Mo(1)'	N(8)	-58.03
N(6)	Mo(1)	Mo(1)'	N(8)	15.62
N(8)'	Mo(1)	Mo(1)'	N(8)	92.64

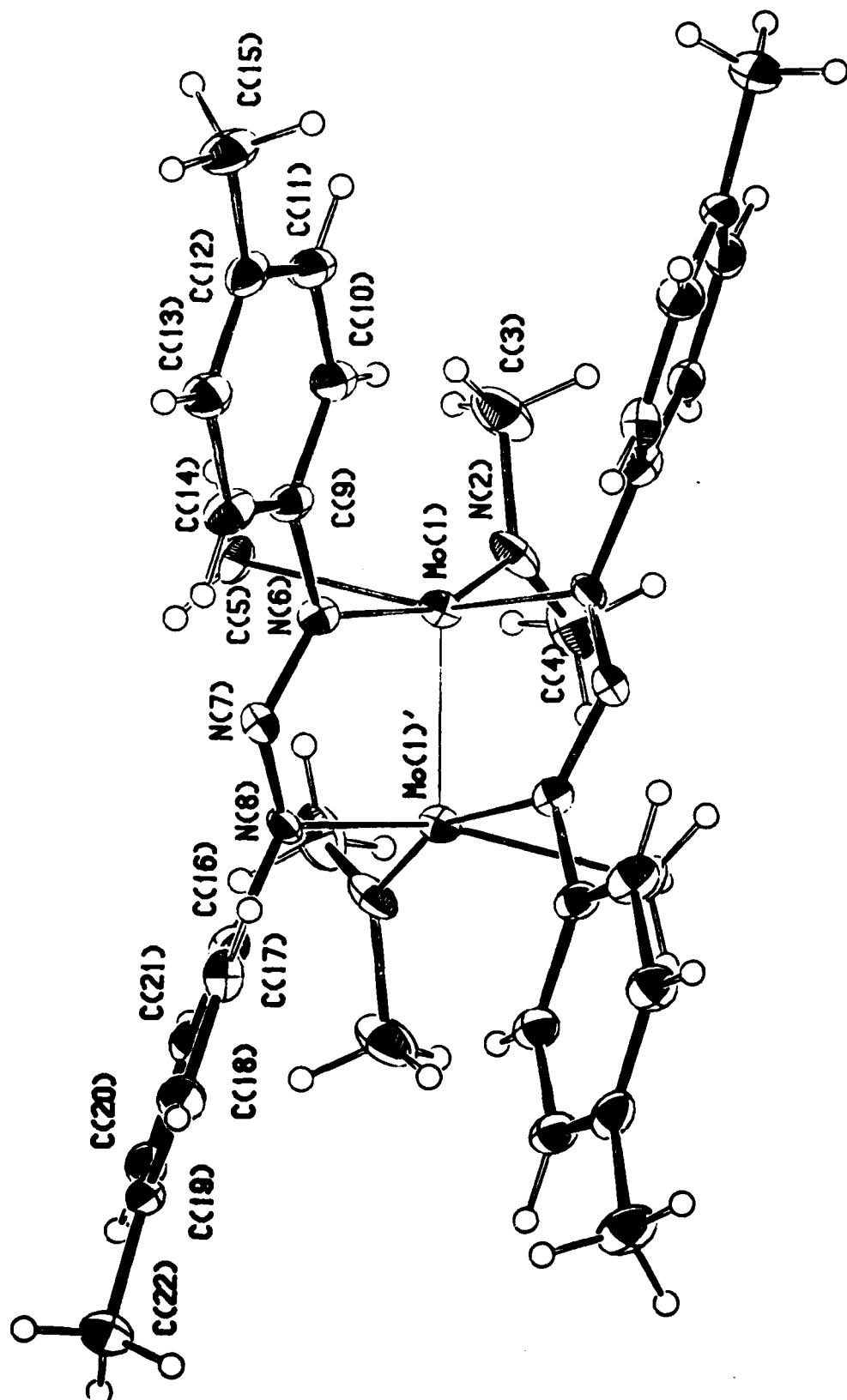
(b) In the Mo-Mo-Triazenido Ring

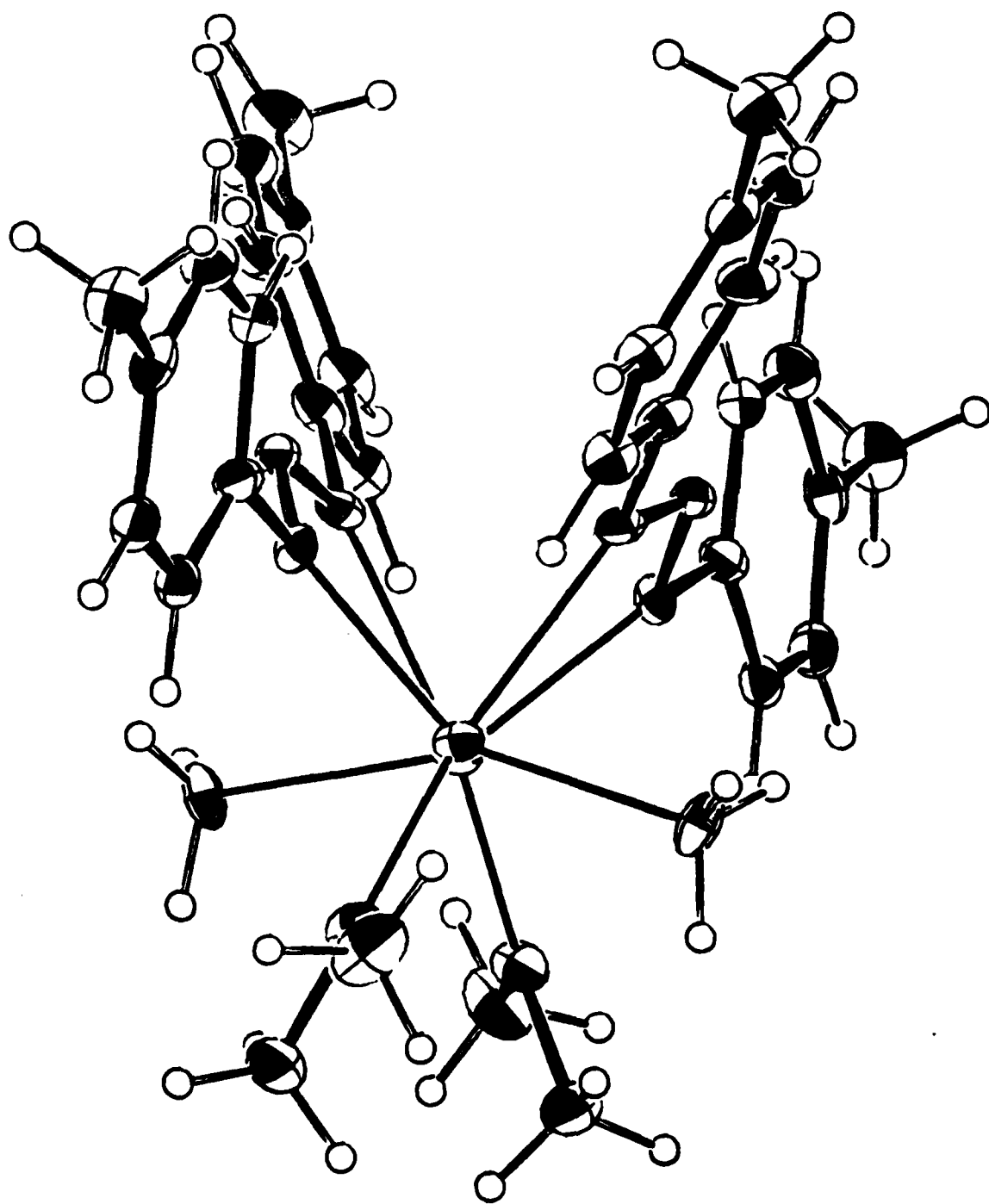
Mo(1)'	Mo(1)	N(6)	N(7)	-18.70
Mo(1)	N(6)	N(7)	N(8)	5.76
N(6)	N(7)	N(8)	Mo(1)'	12.63
N(7)	N(8)	Mo(1)'	Mo(1)	-23.25
N(8)	Mo(1)'	Mo(1)	N(6)	15.62
Mo(1)'	Mo(1)	N(6)	C(9)	168.40
N(8)	N(7)	N(6)	C(9)	179.36
Mo(1)	Mo(1)'	N(8)	C(16)	176.94
N(6)	N(7)	N(8)	C(16)	174.03

Captions to Figures

Figure 1. An ORTEP view of the $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ molecule viewed down the C_2 axis of symmetry and showing the atom numbering scheme used in the tables. Atoms are represented by their thermal ellipsoids drawn at the 50% probability level.

Figure 2. An ORTEP view of the $\text{Mo}_2\text{Me}_2(\text{NMe}_2)_2(\text{C}_7\text{H}_8\text{N}_3\text{C}_7\text{H}_8)_2$ molecule viewed down the Mo=Mo bond.





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