are close to the statistically predicted value of 4.

Since these halide ligand scrambling reactions all occur in nonpolar, noncoordinating solvents and equilibria are rapidly established, we presume that the halogen interchange is occurring by the formation of halogen bridges between individual palladium complexes. Related observations of halide ligand scrambling of Pt(IV) phosphine complexes appears to involve catalysis by Pt(II).[17] For the 16-electron complexes described here, there is not electronic need to involve a redox step in the ligand exchange.

Acknowledgment. We thank the National Science Foundation (Grant CHE 7924515) for support and Matthey Bishop for a loan of palladium chloride.

Registry No. Pd(dpm)ClBr, 80594-57-6; Pd(dpm)BrI, 80594-58-7; Pd(dpm)ClI, 80594-59-8; Pd(dpm)Cl_2, 38425-01-3; Pd(dpm)Br_2, 77462-41-0; Pd(dpm)I_2, 77462-40-9; $Pd_2(dpm)_2Cl_2$, 64345-29-5; $Pd_2(dpm)_2Br_2$, 60482-68-0; $Pd_2(dpm)_2I_2$, 67477-87-6; $Pd_2(dpm)_2$ClBr, 80594-60-1; $Pd_2(dpm)_2$BrI, 80594-61-2; $Pd_2(dpm)_2$ClI, 80594-62-3.

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Axially Coordinated Tetrakis[*N*-(2,6-dimethylphenyl)acetamido]dimolybdenum(II) Compounds. Structures of $Mo_2[(xylyl)NC(CH_3)O]_4{\cdot}2THF$ and $Mo_2[(xylyl)NC(CH_3)O]_4{\cdot}L$ (L = NC_5H_5, $NC_5H_4CH_3$)

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The structures of three axially coordinated tetrakis(xylylacetamido)dimolybdenum(II) compounds are reported (xylyl = 2,6-dimethylphenyl): $Mo_2[(xylyl)NC(CH_3)O]_4{\cdot}2THF$ (**1**) and $Mo_2[(xylyl)NC(CH_3)O]_4{\cdot}L$ (L = NC_5H_5 (**2**), $NC_5H_4CH_3$ (**3**)). Compound **1** was solved in the orthorhombic space group *Pnna* with unit cell parameters a = 20.147 (5) Å, b = 48.02 (1) Å, c = 16.485 (4) Å, V = 15949 (11) Å^3, and Z = 12. Compound **2** crystallizes in the orthorhombic space group *Pccn* with unit cell parameters a = 30.414 (6) Å, b = 17.371 (3) Å, c = 18.489 (2) Å, V = 9768 (4) Å^3, and Z = 8. Compound **3** was solved in the orthorhombic space group *Pbcn* with cell parameters a = 15.635 (1) Å, b = 18.272 (1) Å, c = 15.509 (2) Å, V = 4431 (1) Å^3, and Z = 4. The crystals of **1** contain molecules on general positions and also on special positions where a twofold axis is collinear with the Mo–Mo bond. The two sorts of molecules are essentially identical, having a transoid (D_{2d}) arrangement of the bridging ligands, Mo–Mo bond lengths of 2.097 (3) and 2.093 (2) Å, and two axially coordinated THF molecules with average Mo–O distances of 2.58 (1) and 2.62 (1) Å. Molecules of **2** and **3** also have the transoid arrangement of bridging ligands with Mo–Mo distances of 2.101 (1) and 2.102 (1) Å and one axially coordinated pyridine (Mo–N = 2.563 (8) Å or 4-picoline (Mo–N = 2.592 (8) Å), respectively.

Introduction

Recent investigations in this laboratory of $Cr_2[RNC(R')O]_4{\cdot}L_2$ compounds[1-3] have shown that ligands of the type RNC(R′)O favor short metal–metal bonds and a marked sensitivity toward axial coordination. For comparison we have prepared and structurally characterized some analogous axially coordinated dimolybdenum compounds. Previous studies have suggested that dimolybdenum compounds, unlike their chromium analogues, do not in general accept axial ligands, although there are exceptions such as $Mo_2(S_2CR)_4{\cdot}2THF$.[4,5] However, the recent preparation of $Mo_2[(xylyl)NC(H)O]_4{\cdot}THF$[6] (xylyl = 2,6-dimethylphenyl) has lead us to believe that, as in the chromium analogues, ligands of the type RNC(R′)O enhance the sensitivity of the Mo_2^{4+} unit toward axial ligands. In order to examine this possibility in more detail, we have undertaken the preparation of additional examples of axially coordinated dimolybdenum compounds. We present a report of the successful preparation and structural characterization of three such derivatives, $Mo_2[(xylyl)NC(CH_3)O]_4{\cdot}2THF$ (**1**) and $Mo_2[(xylyl)NC(CH_3)O]_4{\cdot}L$, L = NC_5H_5 (**2**) and $NC_5H_4(p\text{-}CH_3)$ (**3**).

Experimental Section

2,6-Dimethylacetanilide was prepared from acetic anhydride and 2,6-xylidine. After recrystallization from ethanol, the compound had the literature melting point of 176 °C. All manipulations were carried out under an atmosphere of dry argon.

Preparation of $Mo_2[(xylyl)NC(CH_3)O]_4{\cdot}2THF$ (1). A solution of $Li[(xylyl)NC(CH_3)O]$ was prepared by dissolving 0.65 g (4 mmol) of 2,6-dimethylacetanilide in 25 mL of THF and adding the corresponding amount of *n*-butyllithium in hexane. The presence of any excess *n*-BuLi is indicated by the appearance of a yellow tint in the reaction mixture. Anhydrous dimolybdenum tetraacetate (0.42 g, 1 mmol) was added to this solution, and the solution was stirred for ~16 h at room temperature. Subsequent filtration gave a yellow solution. Slow evaporation of this solution with a continuous flow of argon produced a good crop of air-sensitive crystals with dimensions up to 1 mm.

Preparation of $Mo_2[(xylyl)NC(CH_3)O]_4{\cdot}L$, L = Pyridine (2) or 4-Picoline (3). The reaction of 0.04 mol of $Li[(xylyl)NC(CH_3)O]$ with 0.01 mol of $Mo_2(O_2CCH_3)_4$ was carried out as described above. After 10 h the reaction mixture was filtered and the solvent stripped from the yellow filtrate to leave a yellow solid residue. This residue was heated (130 °C) in vacuum (0.05 torr) for 8 h and then dissolved in benzene (30 mL). After filtration of this solution, pyridine or 4-picoline (0.5 mL, 0.06 mol) was added and it was allowed to stand quietly for 10 days. In each case 5–10 mg of well-formed crystals suitable for X-ray diffraction were obtained.

X-ray Data Collection. Suitable crystals of the compounds were coated with epoxy cement, sealed in thin glass capillaries, and mounted on an Enraf-Nonius CAD-4F automatic diffractometer. Intensity data were collected by using Mo Kα radiation ($\lambda_{\bar{\alpha}}$ = 0.71073 Å) monochromatized by a graphite crystal in the incident beam. The

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Table I. Crystallographic Parameters

parameter	1	2	3
space group	*Pnna*	*Pccn*	*Pbcn*
a, Å	20.147 (5)	30.414 (6)	15.635 (1)
b, Å	48.02 (1)	17.371 (3)	18.272 (1)
c, Å	16.485 (4)	18.489 (2)	15.509 (2)
α, deg	90.0	90.0	90.0
β, deg	90.0	90.0	90.0
γ, deg	90.0	90.0	90.0
V, Å^3	15949 (11)	9768 (4)	4431 (1)
d_{calcd}, g/cm^3	1.231	1.357	1.400
Z	12	8	4
formula wt	984.95	997.95	933.86
crystal size, mm	0.30 × 0.35 × 0.35	0.25 × 0.30 × 0.35	0.30 × 0.30 × 0.35
μ(Mo Kα), cm^{-1}	5.113	5.554	6.063
range 2θ, deg	0.01-45	0.01-45	0.01-45
no. of unique data	11352	7005	3263
no. of data, $F_o^2 > 3\sigma(F_o^2)$	3386	2493	1585
no. of variables	453	299	260
R_1	0.084	0.059	0.044
R_2	0.100	0.079	0.061
goodness of fit	2.382	2.134	1.783
largest shift[a]	0.05	0.30	0.20

[a] Largest shift in any atomic parameter in the last cycle of refinement, as a fraction of the esd.

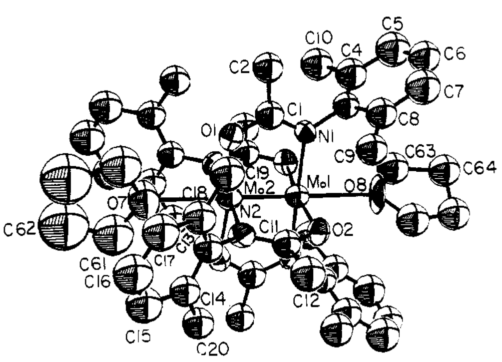

Figure 1. ORTEP drawing of $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2THF$ (**1**), molecule 1, showing atomic labeling scheme. Ellipsoids of thermal motion, scaled to enclose 30% of the electron density, represent each atom.

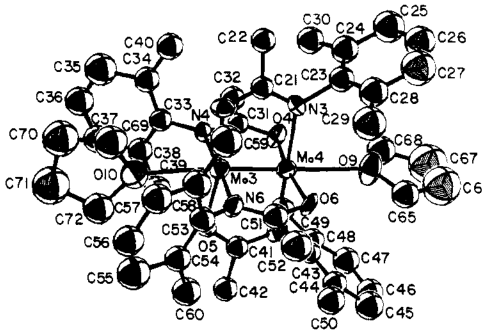

Figure 2. ORTEP drawing of $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2THF$ (**1**), molecule 2, showing the atomic labeling scheme. Ellipsoids of thermal motion, scaled to enclose 30% of the electron density, represent each atom.

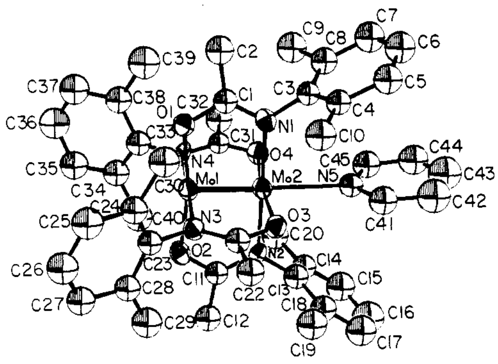

Figure 3. ORTEP drawing of $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot py \cdot C_6H_6$ (**2**) showing the atomic labeling scheme. Ellipsoids of thermal motion, scaled to enclose 30% of the electron density, represent each atom.

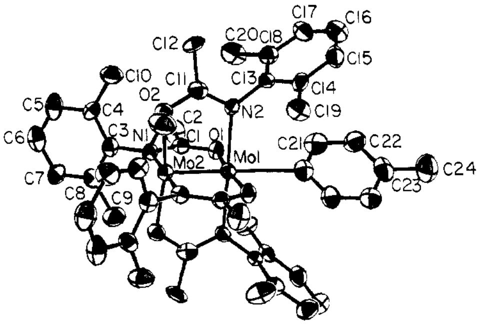

Figure 4. ORTEP drawing of $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot NC_5H_4CH_3$ (**3**) showing the atomic labeling scheme. Ellipsoids of thermal motion, scaled to enclose 30% of the electron density, represent each atom.

takeoff angle of the X-ray tube was 2.80°, and the temperature during data collection was 26 ± 2 °C. Twenty-five reflections in the range $12 < \theta < 16°$ were located by using the standard CAD-4 random automatic searching routine. The reflections were subsequently centered and used as input for the standard CAD-4 indexing routine.

Intensity data were measured with an ω–2θ motion. The scan width for each reflection was determined as 0.8 + 0.347 (tan θ), and each reflection was measured with a preliminary scan rate of 20.12°/min. The scan rate for the final scan was calculated from the preliminary scan such that the ratio $I/\sigma(I)$ would be at least 20 and the maximum scan time would not exceed 30 s. If in the preliminary scan $I/\sigma(I) \geq 20$, this measurement was used as the datum. The scan rates used varied from 20.12 to 2.51°/min. The width of the adjustable vertical aperature at the detector was given by (1.5 + tan θ) mm. The horizontal slit was 4 mm. Of the 96 steps in the profile scan, the first and last 16 steps were considered to be background. Intensities and standard deviations in the intensities were assigned as

$$I = [P - 2(B_1 + B_2)]S \qquad \sigma(I) = [P + 4(B_1 + B_2)]^{1/2}S$$

where P is the gross peak intensity, B_1 and B_2 are the background intensities, and S is the scan rate. During data collection three orientation standards as well as three intensity standards were measured every 100 reflections. Lorentz and polarization corrections were applied. A list of the pertinent crystallographic data collection parameters for the three compounds is given in Table I.

Solution and Refinement. For compound **1** the positions of the four crystallographically independent molybdenum atoms were located by application of direct methods from the program MULTAN.[7] The positions of the molybdenum atoms for compounds **2** and **3** were found by solution of the three-dimensional Patterson function. The remaining nonhydrogen atoms for all three compounds were found from successive Fourier and difference syntheses. Refinement on these positions gave the final discrepancy factors given in Table I. The final discrepancy indices are defined by the expressions

$$R_1 = (\sum||F_o| - |F_c||)/\sum|F_o|$$

$$R_2 = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$$

The function $\sum w(|F_o| - |F_c|)^2$ was minimized with the weighting factor, w, equal to $4F_o^2/\sigma(F_o^2)^2$. Atomic scattering factors were those of Cromer and Waber.[8] Anomalous dispersion effects were also included for the molybdenum atoms.[9]

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(7) Calculations were performed with the Molecular Structure Corp. PDP-11/45 computer and the Enraf-Nonius structure determination package with programs written chiefly by Frenz and Okaya.

(8) Cromer, D. T.; Waber, J. T. "International Tables for X-Ray Crystallography"; Kynoch Press: Birmingham, England, 1974; Vol. IV, Table 2.3.1.

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Table II. Atomic Positional and Isotropic Thermal Parameters for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2THF$ (**1**)

atom	x	y	z	atom	x	y	z
Mo(1)	0.48057 (14)	0.25000 (0)	0.7500 (0)	O(7)	0.7143 (11)	0.2500 (0)	0.7500 (0)
Mo(2)	0.58468 (14)	0.25000 (0)	0.7500 (0)	O(8)	0.3537 (9)	0.2500 (0)	0.7500 (0)
Mo(3)	0.39608 (10)	0.08259 (4)	0.2216 (1)	O(9)	0.6207 (7)	0.0885 (4)	0.2983 (9)
Mo(4)	0.49652 (9)	0.08415 (4)	0.2538 (1)	O(10)	0.2726 (9)	0.0756 (3)	0.1830 (12)
O(1)	0.5918 (7)	0.2134 (3)	0.6821 (8)	N(1)	0.4794 (8)	0.2121 (3)	0.6772 (9)
O(2)	0.4734 (7)	0.2728 (3)	0.6430 (8)	N(2)	0.5885 (8)	0.2734 (4)	0.6394 (9)
O(3)	0.3903 (7)	0.1238 (3)	0.1843 (8)	N(3)	0.5004 (8)	0.1271 (3)	0.2144 (10)
O(4)	0.5318 (7)	0.0731 (3)	0.1416 (8)	N(4)	0.4222 (7)	0.0706 (3)	0.0969 (8)
O(5)	0.3851 (6)	0.0408 (3)	0.2519 (8)	N(5)	0.4942 (8)	0.0404 (3)	0.2908 (10)
O(6)	0.4773 (7)	0.0964 (3)	0.3727 (7)	N(6)	0.3660 (8)	0.0946 (4)	0.3410 (10)

atom	x	y	z	B, Å^2	atom	x	y	z	B, Å^2
C(1)	0.537 (1)	0.2017 (5)	0.659 (1)	6.0 (6)	C(37)	0.308 (1)	0.0297 (6)	−0.037 (2)	8.7 (8)
C(2)	0.547 (1)	0.1741 (5)	0.609 (1)	6.4 (7)	C(38)	0.358 (1)	0.0359 (5)	0.027 (1)	6.9 (7)
C(3)	0.422 (1)	0.1974 (4)	0.649 (1)	5.0 (6)	C(39)	0.383 (1)	0.0133 (5)	0.076 (2)	8.1 (8)
C(4)	0.399 (1)	0.2045 (5)	0.569 (1)	7.4 (7)	C(40)	0.377 (1)	0.1169 (5)	−0.005 (2)	7.3 (7)
C(5)	0.337 (1)	0.1928 (6)	0.537 (2)	9.0 (8)	C(41)	0.436 (1)	0.0278 (4)	0.283 (1)	5.3 (6)
C(6)	0.308 (1)	0.1727 (5)	0.591 (1)	7.4 (7)	C(42)	0.426 (1)	−0.0033 (5)	0.307 (1)	6.5 (6)
C(7)	0.326 (1)	0.1665 (6)	0.665 (2)	8.6 (8)	C(43)	0.550 (1)	0.0253 (4)	0.323 (1)	5.1 (6)
C(8)	0.386 (1)	0.1776 (5)	0.699 (1)	6.7 (7)	C(44)	0.562 (1)	0.0239 (5)	0.403 (1)	6.6 (7)
C(9)	0.416 (1)	0.1718 (5)	0.780 (1)	6.6 (6)	C(45)	0.620 (1)	0.0094 (6)	0.440 (2)	9.0 (8)
C(10)	0.434 (1)	0.2249 (5)	0.511 (2)	7.8 (7)	C(46)	0.657 (1)	−0.0044 (6)	0.381 (2)	8.4 (8)
C(11)	0.529 (1)	0.2806 (5)	0.609 (1)	5.5 (6)	C(47)	0.650 (1)	−0.0039 (6)	0.296 (2)	8.4 (8)
C(12)	0.517 (1)	0.2971 (5)	0.528 (2)	8.1 (8)	C(48)	0.590 (1)	0.0108 (5)	0.263 (1)	6.7 (6)
C(13)	0.647 (1)	0.2845 (5)	0.602 (1)	5.7 (6)	C(49)	0.573 (1)	0.0095 (5)	0.172 (1)	7.2 (7)
C(14)	0.679 (1)	0.2670 (5)	0.545 (1)	6.3 (6)	C(50)	0.520 (1)	0.0366 (5)	0.465 (1)	7.9 (7)
C(15)	0.743 (1)	0.2759 (5)	0.510 (2)	8.4 (8)	C(51)	0.411 (1)	0.0997 (5)	0.392 (1)	6.0 (6)
C(16)	0.766 (1)	0.3015 (6)	0.533 (2)	9.7 (9)	C(52)	0.402 (1)	0.1081 (6)	0.484 (2)	8.8 (8)
C(17)	0.730 (1)	0.3192 (6)	0.583 (2)	8.4 (8)	C(53)	0.300 (1)	0.1002 (5)	0.372 (1)	5.9 (6)
C(18)	0.669 (1)	0.3111 (5)	0.622 (1)	7.1 (7)	C(54)	0.266 (1)	0.0773 (5)	0.395 (1)	6.3 (6)
C(19)	0.633 (1)	0.3299 (5)	0.674 (2)	7.6 (7)	C(55)	0.187 (1)	0.0819 (6)	0.417 (2)	9.9 (8)
C(20)	0.650 (1)	0.2392 (5)	0.519 (1)	6.9 (7)	C(56)	0.168 (1)	0.1095 (5)	0.409 (2)	8.2 (8)
C(21)	0.444 (1)	0.1387 (4)	0.190 (1)	5.1 (6)	C(57)	0.206 (1)	0.1330 (5)	0.387 (2)	7.9 (7)
C(22)	0.437 (1)	0.1694 (5)	0.161 (1)	5.6 (6)	C(58)	0.278 (1)	0.1293 (5)	0.364 (1)	7.3 (7)
C(23)	0.563 (1)	0.1450 (5)	0.215 (1)	6.5 (6)	C(59)	0.320 (1)	0.1525 (6)	0.336 (2)	9.1 (8)
C(24)	0.599 (1)	0.1455 (5)	0.144 (1)	7.8 (7)	C(60)	0.293 (1)	0.0474 (5)	0.405 (2)	8.1 (8)
C(25)	0.664 (2)	0.1613 (6)	0.147 (2)	10.3 (9)	C(61)	0.754 (2)	0.2255 (7)	0.719 (2)	11.9 (10)
C(26)	0.674 (1)	0.1722 (6)	0.225 (2)	9.6 (9)	C(62)	0.830 (2)	0.2636 (8)	0.776 (3)	17.9 (16)
C(27)	0.637 (2)	0.1745 (7)	0.295 (2)	11.8 (10)	C(63)	0.313 (1)	0.2592 (5)	0.681 (2)	8.0 (8)
C(28)	0.575 (1)	0.1579 (6)	0.292 (2)	8.3 (8)	C(64)	0.238 (1)	0.2507 (6)	0.703 (1)	7.9 (7)
C(29)	0.536 (2)	0.1583 (6)	0.364 (2)	10.5 (9)	C(65)	0.644 (1)	0.0925 (5)	0.380 (2)	8.0 (8)
C(30)	0.580 (1)	0.1318 (5)	0.070 (2)	7.8 (7)	C(66)	0.716 (2)	0.0898 (8)	0.370 (2)	16.0 (14)
C(31)	0.488 (1)	0.0693 (4)	0.086 (1)	4.8 (5)	C(67)	0.739 (2)	0.0791 (8)	0.287 (3)	17.2 (15)
C(32)	0.517 (1)	0.0615 (5)	−0.001 (2)	7.0 (7)	C(68)	0.672 (1)	0.0815 (6)	0.241 (2)	9.2 (8)
C(33)	0.376 (1)	0.0645 (5)	0.034 (1)	5.4 (6)	C(69)	0.230 (2)	0.1005 (6)	0.161 (2)	10.4 (9)
C(34)	0.355 (1)	0.0880 (5)	−0.010 (1)	7.0 (7)	C(70)	0.171 (2)	0.0916 (7)	0.111 (2)	12.4 (11)
C(35)	0.303 (2)	0.0808 (6)	−0.076 (2)	11.1 (9)	C(71)	0.164 (2)	0.0575 (7)	0.131 (2)	14.0 (12)
C(36)	0.282 (1)	0.0518 (6)	−0.081 (2)	9.1 (8)	C(72)	0.226 (1)	0.0509 (6)	0.186 (2)	9.6 (9)

Results and Discussion

The structures and atom labeling schemes for the two crystallographically independent molecules of $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2THF$ (**1**) are shown in Figures 1 and 2. Figures 3 and 4 show the structures and atom labeling schemes for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot NC_5H_5 \cdot C_6H_6$ (**2**) and $Mo_2[(xylyl)NC(CH_3)O_4] \cdot NC_5H_4CH_3$ (**3**), respectively. Tables II–IV give the atomic positional parameters for compounds **1**, **2**, and **3**, respectively. Tables V–X give the bond distances and angles for the three compounds. The atomic thermal vibration parameters are available as supplementary material.

Compound **1** crystallized in the orthorhombic space group *Pnna* with 12 molecules per unit cell. Eight of the molecules occupy general positions within the cell while the remaining four reside on crystallographic twofold axes. Associated with each molecule are two axially coordinated THF molecules. The two crystallographically independent sets of molecules are essentially identical except that there is a conformational difference in the associated THF ligands. All of the coordinated THF molecules appear to adopt the half-chair conformation; those coordinated to the molecules on the twofold axes are disordered. There may well be additional molecules of THF, probably eight, in the unit cell. In the final stages of refinement residual electron density appeared in the difference map that could be attributed to a THF molecule. However, repeated attempts to refine these positions both at full and partial occupancies failed. It is presumably this neglected electron density that accounts for the relatively high R factors.

Compound **2** crystallizes in the orthorhombic space group *Pccn* with eight molecules per unit cell. Each molecule occupies a general position within the unit cell. Associated with each molecule is one axially coordinated pyridine ligand and one benzene molecule that occupies the voids between the molecules in the cell.

Compound **3** crystallizes in the orthorhombic space group *Pbcn* with four molecules per unit cell. Each molecule resides on a crystallographic twofold axis, which is coincident with the Mo–Mo bond. There is also one axially coordinated 4-picoline ligand associated with each molecule which also sits on the twofold axis.

The Mo–Mo bond lengths for the three compounds are very similar, being 2.097 (3) and 2.093 (2) Å for **1** and 2.101 (1) and 2.102 (1) Å in **2** and **3**, respectively. The distances are, as expected, longer than those found in $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2CH_2Cl_2$,[2] 2.083 (2) Å, $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2CH_2Br_2$,[3] 2.085 (2) Å, and the related compound $Mo_2[PhNC(CH_3)O]_4 \cdot 2THF$,[10] and substantially longer than the

Table III. Atomic Positional and Isotropic Thermal Parameters for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot NC_5H_5 \cdot C_6H_6$ (2)

atom	*x*	*y*	*z*	atom	*x*	*y*	*z*
Mo(1)	0.37346 (3)	0.49920 (9)	0.63805 (5)	O(4)	0.3492 (3)	0.6164 (5)	0.7559 (4)
Mo(2)	0.37283 (4)	0.50147 (8)	0.75164 (5)	N(1)	0.3053 (3)	0.4578 (6)	0.7507 (5)
O(1)	0.3095 (3)	0.4576 (5)	0.6276 (5)	N(2)	0.4403 (3)	0.5452 (7)	0.7515 (5)
O(2)	0.4380 (3)	0.5380 (5)	0.6295 (5)	N(3)	0.3978 (3)	0.3816 (6)	0.6391 (6)
O(3)	0.3972 (3)	0.3901 (5)	0.7613 (4)	N(4)	0.3496 (3)	0.6172 (5)	0.6354 (6)

atom	*x*	*y*	*z*	*B*, Å²	atom	*x*	*y*	*z*	*B*, Å²
N(5)	0.3712 (3)	0.5090 (6)	0.8901 (5)	3.7 (2)	C(26)	0.4176 (5)	0.2593 (10)	0.4455 (8)	5.6 (4)
C(1)	0.2875 (4)	0.4447 (7)	0.6889 (7)	3.6 (3)	C(27)	0.4523 (5)	0.3039 (8)	0.4718 (8)	4.6 (3)
C(2)	0.2403 (4)	0.4138 (8)	0.6772 (8)	4.9 (4)	C(28)	0.4465 (4)	0.3425 (8)	0.5400 (8)	4.1 (3)
C(3)	0.2803 (4)	0.4428 (7)	0.8122 (7)	3.3 (3)	C(29)	0.4841 (5)	0.3865 (9)	0.5710 (9)	5.6 (4)
C(4)	0.2834 (4)	0.3715 (7)	0.8480 (7)	3.6 (3)	C(30)	0.3276 (5)	0.2800 (9)	0.5893 (8)	5.3 (4)
C(5)	0.2623 (5)	0.3588 (8)	0.9176 (8)	4.9 (4)	C(31)	0.3421 (4)	0.6488 (7)	0.6966 (7)	3.2 (3)
C(6)	0.2380 (5)	0.4203 (9)	0.9451 (8)	5.8 (4)	C(32)	0.3254 (5)	0.7347 (8)	0.7035 (8)	5.1 (4)
C(7)	0.2328 (4)	0.4977 (10)	0.9116 (8)	5.8 (4)	C(33)	0.3427 (4)	0.6602 (8)	0.5683 (8)	4.3 (3)
C(8)	0.2543 (4)	0.4964 (9)	0.8426 (7)	4.4 (3)	C(34)	0.3760 (4)	0.7012 (9)	0.5342 (9)	4.9 (4)
C(9)	0.2468 (5)	0.5804 (9)	0.8031 (8)	6.0 (4)	C(35)	0.3701 (5)	0.7298 (9)	0.4663 (9)	5.9 (4)
C(10)	0.3070 (5)	0.3046 (9)	0.8136 (8)	5.4 (4)	C(36)	0.3274 (5)	0.7225 (9)	0.4343 (9)	6.6 (5)
C(11)	0.4595 (4)	0.5517 (8)	0.6875 (8)	3.7 (3)	C(37)	0.2922 (5)	0.6905 (9)	0.4659 (10)	6.3 (4)
C(12)	0.5074 (4)	0.5783 (8)	0.6765 (8)	4.7 (4)	C(38)	0.3002 (5)	0.6538 (8)	0.5361 (9)	4.8 (3)
C(13)	0.4653 (4)	0.5662 (8)	0.8145 (8)	4.2 (3)	C(39)	0.2626 (5)	0.6156 (10)	0.5752 (9)	6.8 (5)
C(14)	0.4650 (4)	0.6426 (8)	0.8370 (8)	4.1 (3)	C(40)	0.4211 (5)	0.7084 (10)	0.5707 (9)	5.8 (4)
C(15)	0.4883 (5)	0.6660 (9)	0.9006 (10)	6.2 (4)	C(41)	0.3793 (5)	0.4438 (9)	0.9296 (9)	5.1 (4)
C(16)	0.5125 (6)	0.6093 (11)	0.9382 (10)	7.4 (5)	C(42)	0.3755 (5)	0.4459 (10)	1.0075 (10)	7.2 (5)
C(17)	0.5141 (5)	0.5296 (9)	0.9149 (9)	6.4 (5)	C(43)	0.3607 (5)	0.5145 (10)	1.0370 (10)	7.5 (5)
C(18)	0.4895 (4)	0.5083 (8)	0.8511 (8)	4.7 (3)	C(44)	0.3529 (5)	0.5755 (9)	0.9984 (9)	6.2 (4)
C(19)	0.4933 (5)	0.4284 (9)	0.8234 (9)	6.0 (4)	C(45)	0.3571 (5)	0.5737 (9)	0.9236 (9)	5.1 (4)
C(20)	0.4408 (5)	0.7038 (9)	0.7960 (8)	5.2 (4)	C(200)	0.3902 (7)	0.5344 (14)	0.2551 (12)	11.3 (7)
C(21)	0.4049 (4)	0.3489 (7)	0.7015 (7)	3.6 (3)	C(201)	0.3939 (6)	0.5716 (11)	0.3260 (11)	8.3 (5)
C(22)	0.4234 (4)	0.2674 (8)	0.7127 (8)	4.4 (3)	C(202)	0.3901 (7)	0.5323 (12)	0.3996 (12)	9.8 (6)
C(23)	0.4052 (4)	0.3391 (7)	0.5727 (7)	3.5 (3)	C(203)	0.3786 (6)	0.4536 (12)	0.3826 (12)	8.8 (6)
C(24)	0.3715 (4)	0.2941 (8)	0.5474 (8)	4.2 (3)	C(204)	0.3690 (6)	0.4197 (12)	0.3220 (12)	9.3 (6)
C(25)	0.3777 (5)	0.2491 (9)	0.4803 (9)	5.6 (4)	C(205)	0.3758 (6)	0.4526 (12)	0.2600 (12)	8.9 (6)

Table IV. Atomic Positional Parameters for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot NC_5H_4CH_3$ (3)

atom	*x*	*y*	*z*	atom	*x*	*y*	*z*
Mo(1)	0.5000 (0)	0.37429 (5)	0.2500 (0)	C(10)	0.2938 (6)	0.1886 (6)	0.1114 (7)
Mo(2)	0.5000 (0)	0.25923 (5)	0.2500 (0)	C(11)	0.3305 (5)	0.3095 (5)	0.3037 (6)
O(1)	0.4584 (4)	0.3816 (3)	0.1227 (4)	C(12)	0.2370 (5)	0.2960 (5)	0.3348 (7)
O(2)	0.3738 (3)	0.2496 (3)	0.2902 (4)	C(13)	0.3112 (5)	0.4357 (4)	0.3005 (6)
N(1)	0.4595 (4)	0.2583 (3)	0.1144 (4)	C(14)	0.3154 (5)	0.4778 (4)	0.3778 (6)
N(2)	0.3652 (4)	0.3735 (3)	0.2917 (5)	C(15)	0.2634 (6)	0.5404 (5)	0.3855 (6)
N(3)	0.5000 (0)	0.5162 (5)	0.2500 (0)	C(16)	0.2079 (6)	0.5631 (5)	0.3177 (7)
C(1)	0.4478 (5)	0.3232 (4)	0.0791 (5)	C(17)	0.2052 (6)	0.5198 (5)	0.2411 (7)
C(2)	0.4188 (7)	0.3320 (5)	−0.0146 (6)	C(18)	0.2563 (5)	0.4561 (5)	0.2347 (5)
C(3)	0.4487 (6)	0.1930 (4)	0.0633 (5)	C(19)	0.3723 (6)	0.4549 (5)	0.4509 (6)
C(4)	0.3693 (6)	0.1581 (5)	0.0632 (6)	C(20)	0.2506 (7)	0.4100 (6)	0.1519 (6)
C(5)	0.3594 (7)	0.0953 (5)	0.0120 (6)	C(21)	0.5553 (6)	0.5542 (5)	0.3006 (7)
C(6)	0.4293 (7)	0.0690 (5)	−0.0367 (6)	C(22)	0.5593 (6)	0.6297 (5)	0.3018 (7)
C(7)	0.5078 (6)	0.1058 (5)	−0.0378 (6)	C(23)	0.5000 (0)	0.6700 (7)	0.2500 (0)
C(8)	0.5163 (5)	0.1668 (4)	0.0122 (6)	C(24)	0.5000 (0)	0.7547 (8)	0.2500 (0)
C(9)	0.6020 (6)	0.2077 (6)	0.0090 (7)				

2.070 (1) Å Mo–Mo distance found in $Mo_2[PhNC[C(CH_3)_3]O]_4$.[6] The increased bond lengths in **1**, **2**, and **3** are most assuredly due predominantly to the presence of the axial ligands. The average $Mo–O_b$ and $Mo–N_b$ distances in the three compounds are typical of these systems, as seen in Table XI, which presents a summary of selected structural parameters for these and related systems. The average $Mo–L_{ax}$ distances are 2.58 (1) and 2.61 (1) Å in **1** and 2.563 (8) and 2.592 (8) Å in **2** and **3**, respectively. These distances differ very little from each other and the 2.590 (7) and 2.564 (5) Å $Mo–L_{ax}$ distances observed in $Mo_2[(xylyl)NC(H)O]_4 \cdot 2THF$. These distances are, however, substantially shorter than the ~2.7 Å $Mo–THF_{ax}$ distance observed in the dithio derivatives $Mo_2[S_2CR]_4 \cdot 2THF$.[4,5]

Unlike their chromium counterparts, dimolybdenum compounds have not, in general, shown a marked tendency toward the acceptance of axial ligands. Until recently, there have been few examples of axially coordinated dimolybdenum compounds. Of the carboxylato compounds, only $Mo_2(O_2CCF_3)_4$ has been shown to form stable solid compounds with axial ligands. In addition, the dithiol compounds of the type $Mo_2(S_2CR)_4$,[4,5] where R = CH_3, C_6H_5, and OC_2H_5, appear to routinely accept axial ligands, and we have recently prepared two compounds, $Mo_2[(xylyl)NCHO]_4 \cdot 2THF$[6] and $Mo_2[(xylyl)NC(CH_3)N(xylyl)]_2(O_2CCH_3)_2 \cdot 4THF$,[11] each having two axially coordinated THF molecules.

The compounds reported here add significantly to the available data on both the general structure properties and, more specifically, on the occurrence of axial ligation of Mo_2^{4+} compounds in which the four bridging ligands are amidato anions, $RNC(R')O^-$. The structural data available for all such compounds are assembled in Table XI. Clearly, these structures vary in several important respects from one compound to another, although some features, e.g., Mo–O and Mo–N bond lengths, are essentially invariant.

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Table V. Bond Distances for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2THF$ (1)

atoms	dist, Å	atom	dist, Å	atoms	dist, Å	atoms	dist, Å
Mo(1)-Mo(2)	2.097 (3)	N(2)-C(11)	1.34 (2)	C(54)-C(55)	1.64 (2)	C(14)-C(15)	1.49 (2)
-O(2)	2.082 (9)	-C(13)	1.43 (2)	-C(60)	1.55 (2)	-C(20)	1.52 (2)
-N(1)	2.18 (1)	N(3)-C(21)	1.33 (2)	C(55)-C(56)	1.39 (2)	C(15)-C(16)	1.37 (2)
Mo(2)-O(1)	2.09 (1)	-C(23)	1.53 (2)	C(56)-C(57)	1.41 (2)	C(16)-C(17)	1.39 (2)
-N(2)	2.14 (1)	C(28)-C(29)	1.42 (2)	C(57)-C(58)	1.51 (2)	C(17)-C(18)	1.44 (2)
Mo(3)-Mo(4)	2.093 (2)	C(31)-C(32)	1.59 (2)	C(61)-C(62)	1.63 (3)	C(18)-C(19)	1.45 (2)
-O(3)	2.078 (9)	C(33)-C(34)	1.41 (2)	C(62)-C(62)′	1.55 (5)	C(21)-C(22)	1.56 (2)
-O(5)	2.082 (9)	-C(38)	1.42 (2)	C(63)-C(64)	1.60 (2)	C(23)-C(24)	1.37 (2)
-N(4)	2.20 (1)	C(34)-C(35)	1.55 (2)	C(64)-C(64)′	1.55 (3)	-C(28)	1.43 (2)
-N(6)	2.14 (1)	-C(40)	1.46 (2)	N(4)-C(31)	1.35 (2)	C(24)-C(25)	1.51 (2)
Mo(4)-O(4)	2.052 (0)	C(35)-C(36)	1.46 (3)	-C(33)	1.43 (2)	-C(30)	1.44 (2)
-O(6)	2.08 (1)	C(36)-C(37)	1.39 (2)	N(5)-C(41)	1.32 (2)	C(25)-C(26)	1.40 (2)
-N(3)	2.16 (1)	C(37)-C(38)	1.48 (2)	-C(43)	1.44 (2)	C(26)-C(27)	1.38 (3)
-N(5)	2.19 (1)	C(38)-C(39)	1.44 (2)	N(6)-C(51)	1.26 (2)	C(27)-C(28)	1.48 (3)
O(1)-C(1)	1.29 (2)	C(41)-C(42)	1.56 (2)	-C(53)	1.44 (2)	C(65)-C(66)	1.47 (3)
O(2)-C(11)	1.31 (2)	C(43)-C(44)	1.34 (2)	C(1)-C(2)	1.58 (2)	C(66)-C(67)	1.53 (3)
O(3)-C(21)	1.30 (2)	-C(48)	1.45 (2)	C(3)-C(4)	1.43 (2)	C(67)-C(68)	1.56 (3)
O(4)-C(31)	1.28 (2)	C(44)-C(45)	1.49 (2)	-C(8)	1.44 (2)	C(69)-C(70)	1.51 (3)
O(5)-C(41)	1.31 (2)	-C(50)	1.46 (2)	C(4)-C(5)	1.47 (2)	C(70)-C(71)	1.67 (3)
O(6)-C(51)	1.39 (2)	C(45)-C(46)	1.39 (2)	-C(10)	1.54 (2)	C(71)-C(72)	1.58 (3)
O(7)-C(61)	1.51 (2)	C(46)-C(47)	1.42 (2)	C(5)-C(6)	1.43 (2)	Mo(1)-O(8)	2.56 (1)
O(8)-C(63)	1.47 (2)	C(47)-C(48)	1.49 (2)	C(6)-C(7)	1.30 (2)	Mo(2)-O(7)	2.61 (2)
O(9)-C(65)	1.44 (2)	C(48)-C(49)	1.54 (2)	C(7)-C(8)	1.44 (2)	Mo(3)-O(10)	2.59 (1)
-C(68)	1.43 (2)	C(51)-C(52)	1.58 (2)	C(8)-C(9)	1.50 (2)	Mo(4)-O(9)	2.62 (1)
O(10)-C(69)	1.52 (2)	C(53)-C(54)	1.36 (2)	C(11)-C(12)	1.58 (2)		
-C(72)	1.51 (2)	-C(58)	1.47 (2)	C(13)-C(14)	1.42 (2)		
N(1)-C(1)	1.30 (2)			-C(18)	1.39 (2)		
-C(3)	1.44 (2)						

Table VI. Bond Angles for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2THF$ (1)

atoms	angle, deg	atoms	angle, deg	atoms	angle, deg	atoms	angle, deg
Mo(2)-Mo(1)-O(2)	94.0 (3)	C(69)-O(10)-C(72)	106 (2)	C(13)-C(14)-C(15)	119 (2)	N(5)-C(41)-C(42)	122 (2)
-N(1)	90.6 (4)	Mo(1)-N(1)-C(1)	116 (1)	-C(20)	122 (2)	N(5)-C(43)-C(44)	121 (2)
O(2)-Mo(1)-O(2)′	172.0 (6)	-C(3)	127 (1)	C(15)-C(14)-C(20)	118 (2)	-C(48)	115 (1)
-N(1)	88.4 (4)	C(1)-N(1)-C(3)	117 (1)	C(14)-C(15)-C(16)	116 (2)	C(44)-C(43)-C(48)	124 (2)
-N(1)′	91.5 (4)	Mo(2)-N(2)-C(11)	115 (1)	C(15)-C(16)-C(17)	123 (2)	C(43)-C(44)-C(45)	124 (2)
N(1)-Mo(1)-N(1)′	178.7 (7)	-C(13)	126 (1)	C(16)-C(17)-C(18)	123 (2)	-C(50)	125 (2)
Mo(1)-Mo(2)-O(1)	93.9 (3)	C(11)-N(2)-C(13)	118 (1)	C(13)-C(18)-C(17)	115 (2)	C(45)-C(44)-C(50)	112 (2)
-N(2)	92.1 (4)	Mo(4)-N(3)-C(21)	117 (1)	-C(19)	123 (2)	C(44)-C(45)-C(46)	111 (2)
O(1)-Mo(2)-O(1)′	172.2 (6)	-C(23)	124 (1)	C(17)-C(18)-C(19)	122 (2)	C(45)-C(46)-C(47)	129 (2)
-N(2)	89.0 (4)	C(21)-N(3)-C(23)	118 (1)	O(3)-C(21)-N(3)	120 (2)	C(46)-C(47)-C(48)	117 (2)
-N(2)′	90.8 (4)	Mo(3)-N(4)-C(31)	112 (1)	-C(22)	115 (2)	C(43)-C(48)-C(47)	115 (2)
N(2)-Mo(2)-N(2)′	175.9 (8)	-C(33)	125 (1)	N(3)-C(21)-C(22)	124 (2)	-C(49)	124 (2)
Mo(4)-Mo(3)-O(3)	95.5 (3)	C(31)-N(4)-C(33)	123 (1)	N(3)-C(23)-C(24)	116 (2)	C(47)-C(48)-C(49)	120 (2)
-O(5)	94.4 (3)	Mo(4)-N(5)-C(41)	116 (1)	-C(28)	113 (2)	O(6)-C(51)-N(6)	121 (2)
-N(4)	90.9 (3)	-C(43)	124 (1)	C(24)-C(23)-C(28)	132 (2)	-C(52)	111 (2)
-N(6)	91.8 (4)	C(41)-N(5)-C(43)	120 (2)	C(23)-C(24)-C(25)	116 (2)	N(6)-C(51)-C(52)	128 (2)
O(3)-Mo(3)-O(5)	170.2 (4)	Mo(3)-N(6)-C(51)	118 (1)	-C(30)	125 (2)	N(6)-C(53)-C(54)	115 (2)
-N(4)	89.2 (4)	-C(53)	129 (1)	C(25)-C(24)-C(30)	119 (2)	-C(58)	115 (2)
-N(6)	90.0 (4)	C(51)-N(6)-C(53)	113 (2)	C(24)-C(25)-C(26)	110 (2)	C(53)-C(54)-C(58)	129 (2)
O(5)-Mo(3)-N(4)	89.8 (4)	O(1)-C(1)-N(1)	122 (2)	C(25)-C(26)-C(27)	136 (2)	C(53)-C(54)-C(55)	116 (2)
-N(6)	90.5 (4)	-C(2)	114 (2)	C(26)-C(27)-C(28)	113 (2)	-C(60)	127 (2)
N(4)-Mo(3)-N(6)	177.3 (4)	N(1)-C(1)-C(2)	124 (2)	C(23)-C(28)-C(27)	113 (2)	C(55)-C(54)-C(60)	117 (2)
Mo(3)-Mo(4)-O(4)	95.6 (3)	N(1)-C(3)-C(4)	116 (2)	-C(29)	131 (2)	C(54)-C(55)-C(56)	112 (2)
-O(6)	94.0 (3)	-C(8)	122 (2)	C(27)-C(28)-C(29)	116 (2)	C(55)-C(56)-C(57)	129 (2)
-N(3)	89.6 (3)	C(4)-C(3)-C(8)	122 (2)	O(4)-C(31)-N(4)	125 (1)	C(56)-C(57)-C(58)	120 (2)
-N(5)	90.9 (3)	C(3)-C(4)-C(5)	121 (2)	-C(32)	116 (1)	C(53)-C(58)-C(57)	112 (2)
O(4)-Mo(4)-O(6)	170.4 (4)	-C(10)	125 (2)	N(4)-C(31)-C(32)	119 (1)	-C(59)	125 (2)
-N(3)	88.0 (4)	C(5)-C(4)-C(10)	114 (2)	N(4)-C(33)-C(34)	114 (2)	C(57)-C(58)-C(59)	123 (2)
-N(5)	90.6 (4)	C(4)-C(5)-C(6)	112 (2)	-C(38)	115 (2)	O(7)-C(61)-C(62)	103 (2)
O(6)-Mo(4)-N(3)	91.1 (4)	C(5)-C(6)-C(7)	129 (2)	C(34)-C(33)-C(38)	131 (2)	C(61)-C(62)-C(62)′	108 (1)
-N(5)	90.3 (4)	C(6)-C(7)-C(8)	120 (2)	C(33)-C(34)-C(35)	113 (2)	O(8)-C(63)-C(64)	106 (1)
N(3)-Mo(4)-N(5)	178.5 (5)	C(3)-C(8)-C(7)	117 (2)	-C(40)	130 (2)	C(63)-C(64)-C(64)′	104 (1)
Mo(2)-O(1)-C(1)	118 (1)	-C(9)	116 (2)	C(35)-C(34)-C(40)	117 (2)	O(9)-C(65)-C(66)	102 (2)
Mo(1)-O(2)-C(11)	117 (1)	C(7)-C(8)-C(9)	127 (2)	C(34)-C(35)-C(36)	116 (2)	C(65)-C(66)-C(67)	115 (3)
Mo(3)-O(3)-C(21)	117 (1)	O(2)-C(11)-N(2)	122 (2)	C(35)-C(36)-C(37)	127 (2)	C(66)-C(67)-C(68)	99 (2)
Mo(4)-O(4)-C(31)	117 (1)	-C(12)	112 (2)	C(36)-C(37)-C(38)	118 (2)	O(9)-C(68)-C(67)	109 (2)
Mo(3)-O(5)-C(41)	118 (1)	N(2)-C(11)-C(12)	126 (2)	C(33)-C(38)-C(37)	115 (2)	O(10)-C(69)-C(70)	111 (2)
Mo(4)-O(6)-C(51)	115 (1)	N(2)-C(13)-C(14)	116 (2)	-C(39)	126 (2)	C(69)-C(70)-C(71)	104 (2)
C(61)-O(7)-C(61)′	116 (2)	-C(18)	121 (2)	C(37)-C(38)-C(39)	118 (2)	C(70)-C(71)-C(72)	104 (2)
C(63)-O(8)-C(63)′	112 (2)	C(14)-C(13)-C(18)	124 (2)	O(5)-C(41)-N(5)	121 (2)	O(10)-C(72)-C(71)	108 (2)
C(65)-O(9)-C(68)	114 (1)			-C(42)	117 (2)		

First, there are two ways of arranging the four amidato ligands consistent with having two ligands oriented in each direction: (1) cisoid, giving C_{2h} symmetry, and (2) transoid, giving D_{2d} symmetry. Both arrangements are found among

Table VII. Bond Distances for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot NC_5H_5 \cdot C_6H_6$ (**2**)

atoms	dist, Å	atoms	dist, Å	atoms	dist, Å	atoms	dist, Å
Mo(1)-Mo(2)	2.101 (1)	N(3)-C(21)	1.30 (1)	C(14)-C(15)	1.43 (2)	C(34)-C(35)	1.36 (2)
-O(1)	2.085 (6)	-C(23)	1.45 (1)	-C(20)	1.50 (1)	-C(40)	1.54 (1)
-O(2)	2.080 (6)	N(4)-C(31)	1.28 (1)	C(15)-C(16)	1.41 (2)	C(35)-C(36)	1.43 (1)
-N(3)	2.173 (7)	-C(33)	1.46 (1)	C(16)-C(17)	1.45 (2)	C(36)-C(37)	1.34 (2)
-N(4)	2.174 (7)	N(5)-C(41)	1.37 (1)	C(17)-C(18)	1.44 (2)	C(37)-C(38)	1.47 (2)
Mo(2)-O(3)	2.080 (7)	-C(45)	1.35 (1)	C(18)-C(19)	1.48 (2)	C(38)-C(39)	1.51 (2)
-O(4)	2.124 (7)	C(1)-C(2)	1.55 (1)	C(21)-C(22)	1.54 (1)	C(41)-C(42)	1.44 (2)
-N(1)	2.189 (8)	C(3)-C(4)	1.41 (1)	C(23)-C(24)	1.37 (1)	C(42)-C(43)	1.38 (2)
-N(2)	2.189 (8)	-C(8)	1.35 (1)	-C(28)	1.40 (1)	C(43)-C(44)	1.30 (2)
-N(5)	2.563 (8)	C(4)-C(5)	1.46 (1)	C(24)-C(25)	1.48 (2)	C(44)-C(45)	1.39 (2)
O(1)-C(1)	1.33 (1)	-C(10)	1.51 (1)	-C(30)	1.56 (1)	C(200)-C(201)	1.46 (2)
O(2)-C(11)	1.28 (1)	C(5)-C(6)	1.40 (1)	C(25)-C(26)	1.39 (1)	-C(205)	1.49 (2)
O(3)-C(21)	1.34 (1)	C(6)-C(7)	1.49 (2)	C(26)-C(27)	1.40 (1)	C(201)-C(202)	1.53 (2)
O(4)-C(31)	1.25 (1)	C(7)-C(8)	1.43 (1)	C(27)-C(28)	1.44 (1)	C(202)-C(203)	1.44 (2)
N(1)-C(1)	1.28 (1)	C(8)-C(9)	1.65 (2)	C(28)-C(29)	1.49 (1)	C(203)-C(204)	1.30 (2)
-C(3)	1.39 (1)	C(11)-C(12)	1.54 (1)	C(31)-C(32)	1.58 (1)	C(204)-C(205)	1.30 (2)
N(2)-C(11)	1.32 (1)	C(13)-C(14)	1.39 (1)	C(33)-C(34)	1.39 (1)		
-C(13)	1.44 (1)	-C(18)	1.42 (1)	-C(38)	1.43 (1)		

Table VIII. Bond Angles for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot NC_5H_5 \cdot C_6H_6$ (**2**)

atoms	angle, deg	atoms	angle, deg	atoms	angle, deg	atoms	angle, deg
Mo(2)-Mo(1)-O(1)	95.2 (2)	C(1)-N(1)-C(3)	117.6 (9)	N(2)-C(11)-C(12)	124 (1)	C(27)-C(28)-C(29)	119 (1)
-O(2)	94.5 (2)	Mo(2)-N(2)-C(11)	116.3 (7)	N(2)-C(13)-C(14)	118 (1)	O(4)-C(31)-N(4)	123.5 (9)
-N(3)	90.7 (2)	-C(13)	125.6 (6)	-C(18)	118 (1)	-C(32)	114 (1)
-N(4)	90.1 (2)	C(11)-N(2)-C(13)	118.1 (9)	C(14)-C(13)-C(18)	122 (1)	N(4)-C(31)-C(32)	122 (1)
O(1)-Mo(1)-O(2)	170.2 (2)	Mo(1)-N(3)-C(21)	118.2 (7)	C(13)-C(14)-C(15)	121 (1)	N(4)-C(33)-C(34)	123 (1)
-N(3)	89.6 (3)	-C(23)	121.6 (6)	-C(20)	122 (1)	-C(38)	116 (1)
-N(4)	90.8 (3)	C(21)-N(3)-C(23)	120.2 (8)	C(15)-C(14)-C(20)	117 (1)	C(34)-C(33)-C(38)	121 (1)
O(2)-Mo(1)-N(3)	89.0 (3)	Mo(1)-N(4)-C(31)	116.4 (7)	C(14)-C(15)-C(16)	118 (1)	C(33)-C(34)-C(35)	121 (1)
-N(4)	90.4 (3)	-C(33)	123.2 (7)	C(15)-C(16)-C(17)	122 (1)	-C(40)	120 (1)
N(3)-Mo(1)-N(4)	179.1 (3)	C(31)-N(4)-C(33)	120.4 (8)	C(16)-C(17)-C(18)	118 (1)	C(35)-C(34)-C(40)	119 (1)
Mo(1)-Mo(2)-O(3)	93.8 (2)	C(41)-N(5)-C(45)	119.9 (9)	C(13)-C(18)-C(17)	118 (1)	C(34)-C(35)-C(36)	118 (1)
-O(4)	93.3 (2)	O(1)-C(1)-N(1)	121.0 (8)	-C(19)	123 (1)	C(35)-C(36)-C(37)	125 (1)
-N(1)	89.7 (2)	-C(2)	114 (1)	C(17)-C(18)-C(19)	119 (1)	C(36)-C(37)-C(38)	116 (1)
-N(2)	89.8 (2)	N(1)-C(1)-C(2)	125 (1)	O(3)-C(21)-N(3)	118.1 (9)	C(33)-C(38)-C(37)	119 (1)
-N(5)	178.1 (2)	N(1)-C(3)-C(4)	120.8 (9)	-C(22)	116 (1)	-C(39)	121 (1)
O(3)-Mo(2)-O(4)	172.8 (2)	-C(8)	122.3 (9)	N(3)-C(21)-C(22)	126 (1)	C(37)-C(38)-C(39)	119 (1)
-N(1)	90.8 (3)	C(4)-C(3)-C(8)	117 (1)	N(3)-C(23)-C(24)	117.7 (9)	N(5)-C(41)-C(42)	120 (1)
-N(2)	89.3 (3)	C(3)-C(4)-C(5)	121.4 (9)	-C(28)	119.0 (9)	C(41)-C(42)-C(43)	116 (1)
O(4)-Mo(2)-N(1)	90.4 (3)	-C(10)	120.8 (9)	C(24)-C(23)-C(28)	123 (1)	C(42)-C(43)-C(44)	123 (1)
-N(2)	89.6 (3)	C(5)-C(4)-C(10)	117.8 (9)	C(23)-C(24)-C(25)	120 (1)	C(43)-C(44)-C(45)	121 (1)
N(1)-Mo(2)-N(2)	179.5 (2)	C(4)-C(5)-C(6)	116 (1)	-C(30)	124 (1)	N(5)-C(45)-C(44)	120 (1)
Mo(1)-O(1)-C(1)	116.5 (6)	C(5)-C(6)-C(7)	127 (1)	C(25)-C(24)-C(30)	116 (1)	C(201)-C(200)-C(205)	113 (2)
Mo(1)-O(2)-C(11)	118.6 (6)	C(6)-C(7)-C(8)	108 (1)	C(24)-C(25)-C(26)	116 (1)	C(200)-C(201)-C(202)	126 (1)
Mo(2)-O(3)-C(21)	119.2 (6)	C(3)-C(8)-C(7)	131 (1)	C(25)-C(26)-C(27)	125 (1)	C(201)-C(202)-C(203)	104 (2)
Mo(2)-O(4)-C(31)	116.7 (6)	-C(9)	120.4 (9)	C(26)-C(27)-C(28)	118 (1)	C(202)-C(203)-C(204)	132 (2)
Mo(2)-N(1)-C(1)	117.6 (7)	C(7)-C(8)-C(9)	108 (1)	C(23)-C(28)-C(27)	118 (1)	C(203)-C(204)-C(205)	122 (2)
-C(3)	124.8 (6)	O(2)-C(11)-N(2)	120.6 (9)	-C(29)	123 (1)	C(200)-C(205)-C(205)	121 (2)
		-C(12)	115 (1)				

Table IX. Bond Distances for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot NC_5H_4CH_3$ (**3**)

atoms	dist, Å	atoms	dist, Å
Mo(1)-Mo(2)	2.102 (1)	C(5)-C(6)	1.41 (1)
-O(1)	2.084 (5)	C(6)-C(7)	1.40 (1)
-N(2)	2.204 (5)	C(7)-C(8)	1.36 (1)
-N(3)	2.592 (8)	C(8)-C(9)	1.54 (1)
Mo(2)-O(2)	2.077 (4)	C(11)-C(12)	1.559 (9)
-N(1)	2.196 (6)	C(13)-C(14)	1.425 (9)
O(1)-C(1)	1.274 (7)	-C(18)	1.386 (9)
O(2)-C(11)	1.304 (8)	C(14)-C(15)	1.409 (9)
N(1)-C(1)	1.320 (8)	-C(19)	1.50 (1)
-C(3)	1.442 (8)	C(15)-C(16)	1.42 (1)
N(2)-C(11)	1.303 (8)	C(16)-C(17)	1.43 (1)
-C(13)	1.422 (8)	C(17)-C(18)	1.42 (1)
N(3)-C(21)	1.359 (9)	C(18)-C(20)	1.54 (1)
C(1)-C(2)	1.530 (9)	C(21)-C(22)	1.38 (1)
C(3)-C(4)	1.395 (9)	C(22)-C(23)	1.43 (1)
-C(8)	1.405 (9)	C(23)-C(24)	1.55 (1)
C(4)-C(5)	1.40 (1)		
-C(10)	1.50 (1)		

these compounds although D_{2d} symmetry is the more prevalent. In all cases where there are xylyl rather than phenyl groups on the nitrogen atoms, the arrangement is transoid. This is very likely to be the result of steric effects, since the methyl substituents on the xylyl groups constrain the planes of these groups to be essentially perpendicular to the amidato ligand planes. Two such xylyl groups would interact repulsively in a cisoid arrangement. On the other hand, the unsubstituted phenyl groups can rotate to angles of 45° or perhaps even less with respect to the amidato ligand plane, thereby allowing the cisoid arrangement. Whether the cisoid arrangement is electronically preferred is not clear, but the available results are perhaps slightly suggestive of this, since it is found in both of the two cases where it is sterically possible.

The other major differences among the compounds have to do with the extent of axial coordination. Some have two axial ligands (**1**, **4**), some have one axial ligand (**2**, **3**), one has no axial ligands (**5**), and three have such remote axial ligands (**6**, **7**, **8**) that it is doubtful if there is any significant axial bond formation. Broadly speaking, the Mo–Mo distances are inversely related to the extent of axial ligation. Thus in **5**, with no axial bonding at all, we find the shortest bond, 2.070 (1) Å, and in the three compounds **6**–**8**, where there is perhaps marginal axial interaction, the bonds are longer, ranging from

Table X. Bond Angles for $Mo_2[(xylyl)NC(CH_3)O]_4 \cdot NC_5H_4CH_3$ (3)

atoms	angle, deg	atoms	angle, deg	atoms	angle, deg	atoms	angle, deg
Mo(2)-Mo(1)-O(1)	93.7 (1)	Mo(2)-O(2)-C(11)	118.0 (4)	C(3)-C(4)-C(5)	118.2 (7)	C(14)-C(13)-C(18)	120.2 (6)
-N(2)	89.6 (1)	Mo(2)-N(1)-C(1)	115.5 (4)	-C(10)	121.9 (6)	C(13)-C(14)-C(15)	118.8 (7)
-N(3)	180.0 (0)	-C(3)	124.6 (4)	C(5)-C(4)-C(10)	119.8 (7)	-C(19)	120.8 (6)
O(1)-Mo(1)-O(1)′	172.7 (2)	C(1)-N(1)-C(3)	119.9 (6)	C(4)-C(5)-C(6)	119.7 (8)	C(15)-C(14)-C(19)	120.3 (7)
-N(2)	88.8 (2)	Mo(1)-N(2)-C(11)	116.5 (4)	C(5)-C(6)-C(7)	121.4 (7)	C(14)-C(15)-C(16)	121.7 (7)
-N(2)′	91.2 (2)	-C(13)	126.2 (4)	C(6)-C(7)-C(8)	118.1 (8)	C(15)-C(16)-C(17)	118.0 (7)
N(2)-Mo(1)-N(2)′	179.3 (3)	C(11)-N(2)-C(13)	117.1 (6)	C(3)-C(8)-C(7)	121.7 (7)	C(16)-C(17)-C(18)	119.8 (7)
Mo(1)-Mo(2)-O(2)	94.9 (1)	C(21)-N(3)-C(21)	118.5 (9)	-C(9)	120.6 (7)	C(13)-C(18)-C(17)	121.3 (7)
-N(1)	90.5 (1)	O(1)-C(1)-N(1)	121.0 (6)	C(7)-C(8)-C(9)	117.6 (7)	-C(20)	120.2 (7)
O(2)-Mo(2)-O(2)′	170.3 (2)	-C(2)	117.0 (6)	O(2)-C(11)-N(2)	121.0 (6)	C(17)-C(18)-C(20)	118.5 (7)
-N(1)	90.7 (2)	N(1)-C(1)-C(2)	122.0 (7)	-C(12)	113.8 (6)	N(3)-C(21)-C(22)	123.2 (8)
-N(1)′	89.2 (2)	N(1)-C(3)-C(4)	118.9 (6)	N(2)-C(11)-C(12)	125.2 (6)	C(21)-C(22)-C(23)	118.5 (8)
N(1)-Mo(2)-N(1)′	179.1 (3)	-C(8)	120.2 (6)	N(2)-C(13)-C(14)	119.0 (6)	C(22)-C(23)-C(22)	118 (1)
Mo(1)-O(1)-C(1)	119.4 (4)	C(4)-C(3)-C(8)	120.9 (6)	-C(18)	120.8 (6)	-C(24)	121.0 (5)

Table XI. Comparison of Selected Structural Parameters in Tetrakis(amidato)dimolybdenum(II) Compounds

	dist, Å					
compd	Mo-Mo	Mo-L_{ax}	Mo-O_b	Mo-N_b	ref	sym[c] of $Mo_2[RNC(R')O]_4$
$Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2THF$ (1)	2.097 (3)[a]	2.58 $(1)_{av}$	2.09 $(1)_{av}$	2.16 $(1)_{av}$	this work	D_{2d}
	2.093 (2)[b]	2.62 $(1)_{av}$	2.07 $(1)_{av}$	2.17 $(1)_{av}$		
$Mo_2[(xylyl)NC(CH_3)O]_4 \cdot py \cdot C_6H_6$ (2)	2.101 (1)	2.563 (8)	2.092 $(7)_{av}$	2.181 $(1)_{av}$	this work	D_{2d}
$Mo_2[(xylyl)NC(CH_3)O]_4 \cdot$picoline (3)	2.102 (1)	2.592 (8)	2.080 $(5)_{av}$	2.200 (6)	this work	D_{2d}
$Mo_2[(xylyl)NC(H)O]_4 \cdot 2THF$ (4)	2.113 (1)	2.590 (7)	2.095 $(3)_{av}$	2.186 $(4)_{av}$	6	D_{2d}
		2.564 (5)				
$Mo_2[PhNC[C(CH_3)_3]O]_4$ (5)	2.070 (1)		2.084 $(4)_{av}$	2.154 $(5)_{av}$	6	C_{2h}
$Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2CH_2Cl_2$ (6)	2.083 (2)	3.417 (6)	3.08 $(1)_{av}$	2.17 $(1)_{av}$	2	D_{2d}
		3.59 (1)				
$Mo_2[(xylyl)NC(CH_3)O]_4 \cdot 2CH_2Br_2$ (7)	2.085 (2)	3.544 (4)	2.08 $(1)_{av}$	2.20 $(1)_{av}$	3	D_{2d}
		3.390 (3)				
$Mo_2[PhNC(CH_3)O]_4 \cdot 2THF$ (8)	2.086 (2)	2.990 (6)	2.09 $(2)_{av}$	2.16 $(2)_{av}$	10	C_{2h}

[a] Molecule 1. [b] Molecule 2. [c] D_{2d} means that N is trans to N at each end. C_{2h} means that N is cis to N at each end.

2.083 (2) to 2.086 (2) Å. The four molecules in which there is firm bonding of one or two axial ligands have Mo-Mo bond lengths in the range 2.093 (2)-2.113 (1) Å.

It is interesting that among the four molecules with axial ligands the two with only one axial pyridine or substituted pyridine ligand have Mo-Mo bond lengths intermediate between those in the two molecules with two axial THF ligands. Apparently, one strongly basic ligand such as pyridine has about the same effect on the Mo-Mo bond as two of a less basic ligand like THF.

It is clear from the information presented in Table XI that for the dimolybdenum unit, the sensitivity of the M-M bond length to axial ligation or changes in the R and R′ groups of the $RNC(R')O^-$ ligands is rather slight and in comparison to that of the Cr-Cr bond it is virtually negligible.

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Supplementary Material Available: Tables of observed and final calculated structures factors and atomic thermal vibration parameters (36 pages). Ordering information is given on any current masthead page.