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(Acetato)tris(*N*,*N*′-diphenylacetamidinato)dimolybdenum and Bis(acetato)bis[*N*,*N*′-bis(2,6-xylyl)acetamidinato]dimolybdenum Tetrakis(tetrahydrofuranate). Two New Mixed-Ligand Quadruply Bonded Dimolybdenum Compounds

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The structures of the two mixed-ligand dimolybdenum compounds $Mo_2[[(2,6\text{-xylyl})N]_2CCH_3]_2(CH_3CO_2)_2\cdot 4THF$ (**1**) and $Mo_2[(PhN)_2CCH_3]_3(CH_3CO_2)$ (**2**) are reported. Compound **1** crystallizes in the triclinic space group $P\bar{1}$ with unit cell parameters a = 11.637 (2) Å, b = 11.665 (1) Å, c = 11.824 (2) Å, α = 90.82 (1)°, β = 108.75 (1)°, γ = 70.02 (1)°, V = 1420.2 (6) Å^3, and Z = 1. Each molecule has 2 axially coordinated THF molecules and resides on a center of inversion. The Mo–Mo bond length is 2.107 (1) Å, and the average Mo–O(acetate) and Mo–N bond lengths are 2.107 (4) and 2.162 (5) Å, respectively. The Mo–O(THF) distance is 2.709 (6) Å. The structure of compound **2** was also solved in the triclinic space group $P\bar{1}$ with unit cell parameters a = 12.516 (3) Å, b = 17.698 (4) Å, c = 10.214 (2) Å, α = 93.88 (2)°, β = 113.17 (2)°, γ = 71.95 (2)°, V = 2004 (1) Å^3, and Z = 2. Each molecule occupies a general position within the unit cell. The Mo–Mo distance is 2.082 (1) Å, and the Mo–O distances average ~2.162 (5) Å and indicate the operation of a structural trans effect in the molecule.

Introduction

Although a wide variety of quadruply bonded dimolybdenum compounds have now been prepared,[1] only five mixed-ligand complexes have been fully characterized. These are $[Mo_2(CH_3CO_2)_2Cl_4][Ph_4As]_2\cdot 2MeOH$,[2] $Mo_2[CH_3CO_2]_2(pd)_2$ (pd = 2,4-pentanedionate),[2] $Mo_2[(C_6H_5)NC(CH_3)CHC(CH_3)O]_2[CH_3CO_2]_2$,[3] the two pyrazolylborate compounds $Mo_2[(pz)_2BH]_2[CH_3CO_2]_2$[4] and $Mo_2[(pz)_2BEt_2]_2[CH_3CO_2]_2\cdot CS_2$,[4] and the recently prepared $Mo_2(ambt)_3(CH_3CO_2)\cdot 2THF$[5] (ambt = 2-amino-4-methylbenzothiazolate). Of these, all except $[Mo_2(O_2CCH_3)_2Cl_4](Ph_4As)_2\cdot 2MeOH$[2] and $Mo_2(ambt)_3(CH_3CO_2)\cdot 2THF$[5] contain at least two chelating ligands. We felt it of interest, therefore, to examine in more detail the structural features of compounds having only nonchelating ligands. In addition, recent results obtained in the structural investigation of the compound $Mo_2(ambt)_3(CH_3CO_2)\cdot 2THF$[5] have suggested that a structural trans effect might be causing an increase in the M–O bond lengths. The further study of this point provided an additional incentive for this investigation. We report here the structures of two new mixed-ligand compounds, $Mo_2[[(2,6\text{-xylyl})N]_2CCH_3]_2[CH_3CO_2]_2\cdot 4THF$ (**1**) and $Mo_2[(PhN)_2CCH_3]_3(CH_3CO_2)$ (**2**).

Experimental Section

The preparation of *N*,*N*′-diphenylacetamidine has been reported earlier.[6] All manipulations were carried out under an atmosphere of dry argon.

Preparation of *N*,*N*′-Bis(2,6-xylyl)acetamidine. *N*,*N*′-Bis(2,6-xylyl)acetamidine was prepared by heating 2′,6′-dimethylacetanilide and phosphorus trichloride to 125 °C for 3 h. Basification with Na_2CO_3/H_2O, extraction with ether, and recrystallization from ethanol afforded the slightly colored compound. Its identify was confirmed by a high-resolution mass spectrum and proton NMR ($CDCl_3/Me_4Si$): δ 1.77 (3 H), 2.25 (12 h), 4.67 (1 H), 7.07 (6 H).

Table I. Crystallographic Parameters

parameter	1	2
space group	$P\bar{1}$	$P\bar{1}$
a, Å	11.637 (2)	12.516 (3)
b, Å	11.665 (1)	17.968 (4)
c, Å	11.824 (2)	10.214 (2)
α, deg	90.82 (1)	93.88 (2)
β, deg	108.75 (1)	113.17 (2)
γ, deg	70.02 (1)	71.95 (2)
V, Å^3	1420.2 (6)	2004 (1)
Z	1	2
fw	1025.06	916.92
cryst size, mm	0.20 × 0.20 × 0.25	0.10 × 0.30 × 0.25
μ, cm^{-1}	4.799	6.640
2θ range, deg	0–45	0–45
no. of data	3716	4004
no. of data with $F_o^2 > 3\sigma(F_o^2)$	2675	3713
no. of variables	271	487
R_1	0.065	0.054
R_2	0.087	0.071
esd	2.297	1.598
$d_{x(\text{calcd})}$, g/cm^3	1.198	1.519
diffractometer	CAD-4F	Syntex $P\bar{1}$

Preparation of $Mo_2[[(2,6\text{-xylyl})N]_2CCH_3]_2(CH_3CO_2)_2\cdot 4THF$ (1). A solution of 0.27 g (1 mmol) of *N*,*N*′-bis(2,6-xylyl)acetamidine in 25 mL of THF was reacted with 1 equiv of *n*-butyllithium in 0.65 mL of 1.6 M hexane solution. To this solution was added 0.11 g of 0.25 mM tetrakis(acetato)dimolybdenum, and after 16 h of stirring the mixture was filtered. The remaining solution was evaporated slowly under a slight vacuum, whereupon yellow cyrstals were obtained which were found to lose solvent easily.

Preparation of $Mo_2[(PhN)_2CCH_3]_3(CH_3CO_2)$ (2). A total of 1 mmol of $Li[C_6H_5NC(CH_3)NHC_6H_5]$ was prepared by adding *n*-BuLi in hexane to a solution of 0.21 g of *N*,*N*′-diphenylacetamidine in 20 mL of TMF until a yellow color persisted. Dimolybdenum tetraacetate (0.11 g, 0.25 mmol was added, and a slow reaction commenced, producing a yellow precipitate after some hours.

After 4 days of stirring at room temperature more THF (40 mL) was added in order to dissolve the precipitate. This solution was then filtered into a tubular flask, and a layer of hexane (40 mL) was placed over the THF solution. Slow diffusion caused yellow crystals to grow on the glass wall. The mass spectrum obtained from these crystals as well as from the residue of the evaporated mother liquid showed, however, that *only three* of the four acetate groups had been replaced. The mass spectrum (300 °C, 10^{-6} torr) gave a peak at m/e 881.144 32 while the value calculated for the corresponding isotopic species $C_{44}H_{42}N_6O_2{}^{97}Mo^{98}Mo$ is 881.148 85.

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Table II. Positional and Thermal Parameters and Their Estimated Standard Deviations for $Mo_2[[(2,6\text{-xylyl})N]_2CCH_3]_2(CH_3CO_2)_2\cdot 4THF$ (1)[a]

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mo	0.05547 (7)	0.05530 (7)	0.00152 (7)	3.86 (2)	3.61 (2)	3.31 (2)	−1.92 (2)	1.19 (2)	−0.36 (2)
O(1)	0.2142 (5)	−0.0968 (5)	−0.0050 (5)	4.0 (2)	4.1 (2)	3.9 (2)	−1.6 (2)	1.1 (2)	−0.1 (2)
O(2)	0.0980 (5)	−0.2151 (5)	−0.0073 (5)	4.4 (2)	4.0 (2)	4.5 (2)	−1.7 (2)	1.9 (2)	−0.5 (2)
O(3)	0.2002 (8)	0.1956 (8)	0.0063 (8)	12.0 (4)	12.8 (4)	7.3 (4)	−8.3 (3)	2.5 (3)	−0.5 (4)
N(1)	0.1343 (6)	0.0317 (6)	0.1951 (6)	4.1 (2)	4.1 (3)	3.8 (3)	−2.0 (2)	1.0 (2)	−0.6 (2)
N(2)	0.0144 (6)	−0.0880 (6)	0.1927 (5)	4.2 (2)	4.0 (3)	3.3 (3)	−1.9 (2)	1.2 (2)	−0.3 (2)
C(1)	0.2034 (8)	−0.2031 (8)	−0.0064 (8)	5.8 (4)	5.2 (4)	4.0 (4)	−2.3 (3)	1.6 (3)	−1.0 (3)
C(2)	0.3168 (10)	−0.3163 (10)	−0.0094 (11)	5.7 (4)	4.3 (5)	10.9 (7)	−0.3 (4)	3.8 (4)	−0.8 (5)
C(3)	0.0982 (7)	−0.0371 (7)	0.2562 (7)	3.8 (3)	3.9 (3)	3.8 (3)	−1.3 (2)	1.0 (3)	0.0 (3)
C(4)	0.1496 (9)	−0.0570 (8)	0.3929 (8)	7.0 (4)	6.6 (4)	3.0 (3)	−3.7 (3)	0.8 (3)	−0.1 (3)
C(5)	−0.0260 (8)	−0.1624 (7)	0.2588 (7)	5.6 (3)	4.7 (3)	3.1 (3)	−2.9 (2)	1.1 (3)	−0.0 (3)
C(6)	−0.1335 (8)	−0.1047 (9)	0.2966 (7)	5.6 (3)	7.1 (4)	3.7 (3)	−3.1 (3)	2.2 (3)	−1.2 (3)
C(7)	−0.1718 (9)	−0.1796 (10)	0.3589 (9)	6.2 (4)	8.8 (5)	5.3 (4)	−3.7 (3)	2.2 (3)	−0.1 (4)
C(8)	−0.1062 (10)	−0.3052 (9)	0.3801 (9)	9.8 (5)	7.3 (5)	5.9 (5)	−5.2 (3)	3.0 (4)	−0.6 (4)
C(9)	−0.0009 (10)	−0.3610 (9)	0.3425 (9)	8.8 (5)	5.8 (5)	6.0 (5)	−3.1 (4)	2.5 (4)	0.1 (4)
C(10)	0.0430 (9)	−0.2881 (8)	0.2826 (8)	7.3 (4)	4.7 (4)	4.6 (4)	−3.1 (3)	2.1 (3)	0.1 (3)
C(11)	0.1622 (11)	−0.3481 (10)	0.2482 (11)	6.9 (5)	4.7 (5)	8.9 (6)	0.5 (4)	3.2 (4)	1.8 (5)
C(12)	−0.2013 (9)	0.0314 (9)	0.2760 (9)	6.7 (4)	4.2 (4)	6.7 (5)	−0.5 (4)	3.6 (3)	−0.3 (4)
C(13)	0.2243 (7)	0.0870 (7)	0.2646 (7)	4.4 (3)	4.9 (3)	3.1 (3)	−2.7 (2)	1.1 (2)	−0.8 (3)
C(14)	0.1747 (9)	0.2046 (8)	0.3016 (8)	6.4 (4)	4.7 (4)	3.9 (4)	−3.0 (3)	1.1 (3)	−0.8 (3)
C(15)	0.2638 (10)	0.2580 (9)	0.3653 (9)	7.8 (5)	6.8 (4)	5.7 (5)	−4.0 (3)	1.2 (4)	−0.7 (4)
C(16)	0.3959 (9)	0.2003 (9)	0.3944 (10)	7.3 (4)	7.2 (5)	6.0 (5)	−4.3 (3)	1.8 (4)	−1.0 (4)
C(17)	0.4432 (9)	0.0843 (11)	0.3602 (9)	5.7 (4)	10.5 (6)	5.2 (5)	−4.5 (3)	1.3 (4)	−1.1 (5)
C(18)	0.3565 (8)	0.0255 (9)	0.2946 (8)	4.2 (3)	6.5 (4)	4.2 (4)	−2.5 (3)	0.7 (3)	−0.2 (4)
C(19)	0.4089 (11)	−0.1022 (11)	0.2634 (11)	5.3 (5)	6.5 (6)	8.0 (6)	0.1 (5)	0.4 (5)	−1.9 (5)
C(20)	0.0328 (9)	0.2657 (9)	0.2821 (9)	4.8 (4)	5.5 (5)	6.7 (5)	−1.3 (4)	1.4 (4)	−1.9 (4)

atom	x	y	z	B, Å²
C(21)	0.154 (2)	0.328 (2)	0.016 (2)	12.0 (5)
C(22)	0.267 (2)	0.360 (2)	−0.004 (2)	17.3 (8)
C(23)	0.371 (2)	0.247 (2)	0.002 (2)	19.2 (9)
C(24)	0.334 (2)	0.136 (2)	−0.004 (2)	12.5 (5)
C(25)	0.347 (2)	0.646 (2)	0.639 (2)	19.5 (9)
C(26)	0.468 (3)	0.556 (3)	0.655 (3)	24.2 (12)
C(27)	0.429 (3)	0.504 (3)	0.770 (3)	25.5 (13)
C(28)	0.393 (3)	0.439 (3)	0.641 (2)	22.3 (11)
C(29)	0.280 (2)	0.533 (2)	0.650 (3)	21.4 (11)

[a] The form of the anisotropic thermal parameter is $\exp[-1/4(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

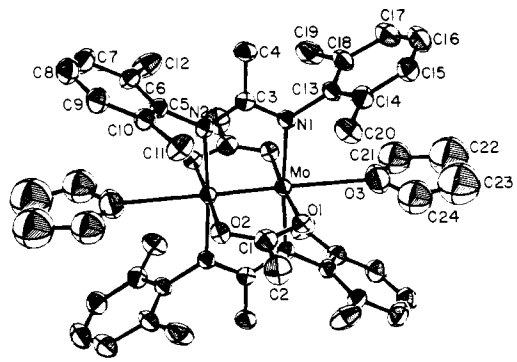

Figure 1. Structure of compound **1** showing the atomic labeling scheme. Each unlabeled atom is related to a labeled one by an inversion center at the midpoint of the Mo–Mo bond. Atoms are represented by their ellipsoids of thermal vibration scaled to enclose 30% of the electron density.

X-ray Crystallography. Suitable crystals were secured to the end of a thin glass fiber with epoxy cement and mounted on either a Syntex $P\bar{1}$ or Enraf-Nonius CAD-4F automatic diffractometer. Data for compound **1** were collected on an Enraf-Nonius CAD-4F while data on compound **2** were collected on a Syntex $P\bar{1}$. Procedures for both data collection and refinement of the structures have been described previously for both the CAD-4F[7] and the Syntex $P\bar{1}$.[6] Absorption corrections were not deemed necessary for compound **1** but for **2** ψ scans indicated that a correction would be advisable, and it was made semiempirically with use of ψ scans at $\chi = 90°$ in the manner previously described. Transmission factors ranged from 91.3% to 99.9%.

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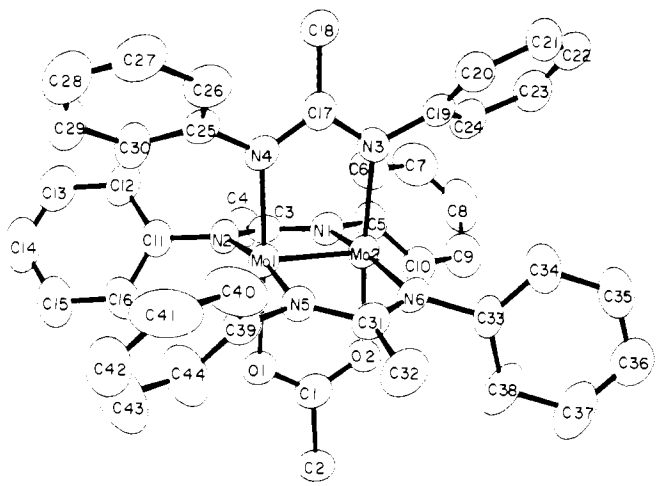

Figure 2. Molecular structure of compound **2**, showing the atom labeling scheme.

Basic crystallographic information on both compounds is given in Table I.

Results and Discussion

Atomic positional and thermal parameters for $Mo_2[[(2,6\text{-xylyl})N]_2CCH_3]_2(CH_3CO_2)\cdot 4THF$ (**1**) and $Mo_2[(PhN)_2CCH_3]_3(CH_3CO_2)$ (**2**) are listed in Tables II and III, respectively. Figures 1 and 2 show the molecular structures and atomic labeling schemes for the two compounds. Bond distances and angles are presented in Tables IV and V for **1** and in Tables VI and VII for **2**.

Table III. Positional and Thermal Parameters and Their Estimated Standard Deviations for $Mo_2[(PhN)_2CCH_3]_3(CH_3CO_2)$ (2)[a]

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mo(1)	0.94645 (5)	0.74967 (3)	0.49406 (6)	2.93 (2)	2.88 (2)	3.06 (2)	−0.69 (2)	0.95 (2)	−0.01 (2)
Mo(2)	0.76689 (5)	0.78053 (3)	0.34444 (6)	3.03 (2)	2.99 (2)	3.08 (2)	−0.72 (2)	0.91 (2)	0.09 (2)
O(1)	1.0027 (4)	0.8036 (3)	0.3595 (4)	3.8 (2)	3.7 (2)	3.7 (2)	−1.0 (1)	1.6 (1)	0.2 (2)
O(2)	0.8106 (4)	0.8359 (3)	0.1991 (5)	3.6 (2)	4.1 (2)	3.7 (2)	−1.4 (1)	1.1 (1)	0.3 (2)
N(1)	0.8034 (5)	0.6752 (3)	0.2329 (5)	4.0 (2)	3.4 (2)	3.0 (2)	−1.3 (2)	1.2 (2)	−0.5 (2)
N(2)	0.9985 (5)	0.6412 (3)	0.3969 (6)	3.1 (2)	3.1 (2)	3.6 (2)	−0.7 (2)	1.0 (2)	−0.0 (2)
N(3)	0.7070 (5)	0.7338 (3)	0.4810 (6)	3.0 (2)	3.3 (2)	3.9 (2)	−1.1 (2)	1.2 (2)	−0.2 (2)
N(4)	0.9035 (5)	0.7009 (3)	0.6446 (5)	3.7 (2)	2.8 (2)	3.3 (2)	−0.7 (2)	1.1 (2)	0.2 (2)
N(5)	0.9178 (4)	0.8600 (3)	0.5838 (5)	3.2 (2)	2.6 (2)	3.4 (2)	−0.8 (2)	1.3 (2)	−0.3 (2)
N(6)	0.7237 (5)	0.8961 (3)	0.4206 (5)	3.4 (2)	2.6 (2)	3.5 (2)	−0.3 (2)	1.3 (2)	0.4 (2)
C(1)	0.9183 (6)	0.8386 (4)	0.2439 (7)	4.8 (3)	3.1 (3)	3.8 (3)	−1.0 (2)	2.1 (2)	−0.3 (2)
C(2)	0.9485 (7)	0.8876 (5)	0.1551 (8)	4.6 (3)	5.9 (4)	5.8 (3)	−1.8 (2)	1.6 (2)	2.4 (3)
C(3)	0.9177 (6)	0.6255 (4)	0.2765 (7)	3.9 (3)	2.8 (3)	3.7 (3)	−1.1 (2)	1.8 (2)	0.0 (2)
C(4)	0.9530 (7)	0.5595 (5)	0.1858 (8)	5.1 (3)	4.8 (4)	4.8 (3)	−1.2 (3)	2.3 (2)	−1.4 (3)
C(5)	0.7073 (6)	0.6650 (4)	0.1066 (7)	4.0 (3)	4.9 (3)	3.1 (3)	−1.2 (3)	1.5 (2)	−1.1 (3)
C(6)	0.6730 (7)	0.5959 (5)	0.0863 (9)	5.5 (3)	5.7 (4)	5.5 (4)	−3.2 (3)	1.6 (3)	−1.6 (3)
C(7)	0.5688 (8)	0.5940 (5)	−0.0343 (10)	7.2 (4)	7.3 (4)	6.7 (5)	−4.3 (3)	1.8 (4)	−2.0 (4)
C(8)	0.5006 (8)	0.6588 (6)	−0.1296 (10)	5.0 (4)	9.8 (6)	5.6 (4)	−1.8 (4)	0.7 (4)	−2.0 (4)
C(9)	0.5341 (8)	0.7257 (6)	−0.1128 (9)	5.3 (4)	8.0 (5)	4.8 (4)	−1.6 (4)	1.4 (3)	0.1 (4)
C(10)	0.6379 (7)	0.7295 (5)	0.0072 (8)	4.1 (3)	5.9 (4)	3.6 (3)	−1.4 (3)	1.1 (2)	0.6 (3)
C(11)	1.1232 (6)	0.5929 (4)	0.4519 (7)	3.6 (3)	3.6 (3)	3.4 (3)	−0.3 (2)	1.7 (2)	−0.5 (2)
C(12)	1.1575 (7)	0.5154 (4)	0.5044 (8)	5.0 (3)	3.6 (3)	4.4 (3)	0.4 (3)	1.9 (2)	−0.2 (3)
C(13)	1.2822 (8)	0.4731 (5)	0.5667 (9)	6.2 (4)	4.3 (4)	5.8 (4)	0.2 (3)	2.5 (3)	−0.2 (3)
C(14)	1.3722 (8)	0.5061 (6)	0.5771 (9)	4.7 (4)	7.1 (5)	5.4 (4)	0.1 (4)	1.3 (3)	−1.3 (4)
C(15)	1.3372 (7)	0.5846 (6)	0.5207 (9)	4.2 (3)	6.7 (5)	6.1 (4)	−0.3 (3)	1.8 (3)	−0.8 (4)
C(16)	1.2129 (6)	0.6273 (5)	0.4608 (8)	3.7 (3)	5.5 (4)	4.7 (3)	−1.3 (3)	1.6 (2)	−0.8 (3)
C(17)	0.7889 (6)	0.7009 (4)	0.6123 (7)	3.6 (3)	3.0 (3)	3.5 (3)	−0.8 (2)	1.3 (2)	−0.5 (2)
C(18)	0.7544 (6)	0.6575 (4)	0.7036 (8)	4.0 (3)	5.0 (3)	4.1 (3)	−1.4 (2)	1.2 (2)	1.0 (3)
C(19)	0.5804 (6)	0.7472 (4)	0.4424 (7)	3.5 (3)	3.3 (3)	3.9 (3)	−1.0 (2)	0.9 (2)	0.9 (2)
C(20)	0.5220 (7)	0.7807 (5)	0.5317 (8)	5.1 (3)	4.4 (4)	5.5 (3)	−0.3 (3)	3.0 (2)	0.6 (3)
C(21)	0.3941 (7)	0.7948 (5)	0.4840 (9)	3.9 (3)	5.5 (4)	6.1 (4)	−0.2 (3)	2.2 (3)	1.4 (3)
C(22)	0.3252 (7)	0.7801 (5)	0.3476 (9)	4.2 (3)	5.2 (4)	7.6 (4)	−0.9 (3)	1.7 (3)	2.6 (3)
C(23)	0.3839 (7)	0.7485 (5)	0.2545 (10)	4.0 (3)	5.9 (4)	6.9 (4)	−2.0 (3)	0.2 (3)	1.6 (3)
C(24)	0.5120 (7)	0.7301 (4)	0.3010 (8)	4.3 (3)	3.7 (3)	5.1 (4)	−1.5 (2)	0.7 (3)	0.2 (3)
C(25)	0.9934 (6)	0.6787 (4)	0.7850 (7)	4.1 (3)	2.7 (3)	3.5 (3)	−1.4 (2)	1.1 (2)	0.1 (2)
C(26)	0.9694 (7)	0.7051 (4)	0.9053 (7)	6.1 (3)	4.9 (3)	3.2 (3)	−2.2 (3)	2.0 (2)	−0.6 (3)
C(27)	1.0623 (8)	0.6835 (5)	1.0436 (8)	7.4 (4)	5.2 (3)	4.0 (3)	−3.1 (3)	1.5 (3)	−0.2 (3)
C(28)	1.1790 (8)	0.6390 (5)	1.0602 (9)	6.9 (4)	5.4 (4)	4.8 (4)	−2.6 (3)	0.4 (3)	0.3 (3)
C(29)	1.2063 (8)	0.6165 (5)	0.9396 (9)	4.8 (4)	5.2 (4)	5.2 (4)	−1.6 (3)	−0.0 (3)	0.0 (3)
C(30)	1.1129 (7)	0.6354 (4)	0.8020 (8)	4.0 (3)	4.2 (3)	4.4 (3)	−1.2 (3)	0.1 (3)	0.1 (3)
C(31)	0.8125 (6)	0.9172 (4)	0.5211 (7)	4.0 (3)	2.7 (3)	3.3 (3)	−1.0 (2)	1.4 (2)	−0.4 (2)
C(32)	0.7947 (7)	1.0032 (4)	0.5613 (8)	6.0 (4)	3.0 (3)	5.0 (3)	−1.0 (3)	2.0 (3)	−0.8 (3)
C(33)	0.6114 (6)	0.9537 (4)	0.3334 (7)	2.9 (3)	3.2 (3)	3.9 (3)	0.0 (2)	1.0 (2)	−0.1 (2)
C(34)	0.5056 (8)	0.9575 (6)	0.3376 (11)	3.9 (4)	10.1 (6)	10.9 (5)	0.8 (4)	2.8 (3)	5.4 (4)
C(35)	0.3982 (9)	0.0138 (7)	0.2473 (14)	4.0 (4)	12.7 (7)	19.3 (8)	2.1 (4)	4.5 (4)	10.2 (5)
C(36)	0.3969 (8)	1.0627 (5)	0.1520 (11)	4.1 (4)	6.1 (4)	8.3 (5)	−1.7 (3)	−0.6 (4)	2.3 (4)
C(37)	0.5049 (9)	1.0567 (6)	0.1415 (10)	7.3 (5)	6.4 (5)	6.6 (4)	1.2 (4)	2.2 (4)	2.4 (4)
C(38)	0.6159 (9)	1.0015 (5)	0.2322 (9)	7.7 (4)	6.1 (5)	7.7 (4)	2.1 (4)	4.2 (3)	3.8 (3)
C(39)	1.0177 (6)	0.8745 (4)	0.6993 (7)	3.7 (3)	3.0 (3)	3.7 (3)	−0.8 (2)	1.3 (2)	0.1 (2)
C(40)	1.0149 (8)	0.8912 (5)	0.8323 (8)	7.8 (4)	10.0 (5)	3.4 (3)	−5.7 (3)	2.0 (3)	−1.3 (3)
C(41)	1.1228 (9)	0.8992 (6)	0.9470 (9)	10.8 (5)	10.9 (5)	3.9 (4)	−7.3 (3)	1.8 (3)	−0.0 (3)
C(42)	1.2267 (8)	0.8884 (5)	0.9254 (10)	5.1 (4)	6.9 (4)	5.9 (5)	−2.6 (3)	−0.4 (4)	0.0 (4)
C(43)	1.2290 (9)	0.8712 (7)	0.7944 (12)	5.2 (4)	12.0 (7)	9.6 (6)	−3.8 (4)	2.3 (4)	−3.6 (6)
C(44)	1.1253 (8)	0.8641 (6)	0.6814 (9)	4.8 (4)	8.8 (5)	6.1 (4)	−2.4 (3)	2.0 (3)	−2.3 (4)

[a] The form of the anisotropic thermal parameter is $\exp[-1/4(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*b^* + 2B_{23}klb^*c^*)]$.

The structure of **1** was solved in the triclinic space group $P\bar{1}$ with each molecule residing on a center of inversion. Each molecule has two acetate and two $[(2,6\text{-xylyl})N]_2CCH_3$ ligands, with like ligands trans to each other. Associated with each molecule are two THF molecules that occupy the axial positions. In addition there are two THF molecules that occupy the voids between the molecules within the cell. As is almost always the case with lattice THF molecules, the latter are extremely disordered and could not be accurately defined. Since these THF molecules are of little importance to us, we simply used the top five peaks, found in a difference Fourier map in this region of space, in the least-squares analysis to account for the electron density present. The bond distances and angles associated with these peaks therefore make no chemical sense and are not reported in the tables. The axially bonded THF molecules are observed to adopt the twist conformation as opposed to the half-chair conformation. The 2,6-xylyl groups of the amidato ligands are essentially perpendicular to the N(1)–Mo(1)–Mo(2)–N(2) plane as evidenced by an average dihedral angle of 91.4°. The presence of axially coordinated THF molecules in this compound is significant in that quadruply bonded dimolybdenum compounds do not frequently show a tendency to have axial ligands. Of the carboxylato compounds, only $Mo_2[O_2CCF_3]_4$ has been shown to accept axial ligands.[11] On the other hand, dithio ligands of the type $Mo_2[S_2CR]_4$, where R = CH_3, C_6H_5, or OC_2H_5, appear to routinely accept axial ligands.[8,9] The

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(9) Ricard, L.; Karagiannidis, P.; Weiss, R. *Inorg. Chem.* **1973**, *12*, 2179.

Table IV. Bond Distances for $Mo_2[[(2,6\text{-xylyl})N]_2CCH_3]_2(CH_3CO_2)_2 \cdot 4THF$ (1)

atoms	dist, Å	atoms	dist, Å	atoms	dist, Å	atoms	dist, Å
Mo–Mo′	2.107 (1)	N(1)–C(3)	1.337 (7)	C(6)–C(7)	1.41 (1)	C(15)–C(16)	1.38 (1)
–O(1)	2.101 (4)	–C(13)	1.443 (7)	–C(12)	1.50 (1)	C(16)–C(17)	1.39 (1)
–O(2)′	2.114 (4)	N(2)–C(3)	1.333 (7)	C(7)–C(8)	1.38 (1)	C(17)–C(18)	1.43 (1)
–O(3)	2.709 (6)	–C(5)	1.456 (7)	C(8)–C(9)	1.39 (1)	C(18)–C(19)	1.50 (1)
–N(1)	2.156 (5)	C(1)–C(2)	1.53 (1)	C(9)–C(10)	1.42 (1)	C(21)–C(22)	1.56 (2)
–N(2)′	2.167 (5)	C(3)–C(4)	1.523 (8)	C(10)–C(11)	1.51 (1)	C(22)–C(23)	1.44 (2)
O(1)–C(1)	1.287 (8)	C(5)–C(6)	1.411 (5)	C(13)–C(14)	1.417 (9)	C(23)–C(24)	1.50 (2)
O(2)–C(1)	1.277 (8)	–C(10)	1.391 (9)	–C(18)	1.381 (9)		
O(3)–C(21)	1.47 (1)			C(14)–C(15)	1.399 (9)		
–C(24)	1.51 (2)			–C(20)	1.50 (1)		

Table V. Bond Angles for $Mo_2[[(2,6\text{-xylyl})N]_2CCH_3]_2(CH_3CO_2)_2 \cdot 4THF$ (1)

atoms	angle, deg	atoms	angle, deg	atoms	angle, deg	atoms	angle, deg
Mo′–Mo–O(1)	91.8 (1)	Mo–N(1)–C(3)	119.1 (4)	N(1)–C(3)–N(2)	117.4 (5)	C(5)–C(10)–C(9)	118.3 (7)
–O(2)′	91.6 (1)	–C(13)	124.0 (4)	–C(4)	121.8 (6)	–C(11)	122.0 (6)
–N(1)	92.4 (1)	C(3)–N(1)–C(13)	116.9 (5)	N(2)–C(3)–C(4)	120.8 (5)	C(9)–C(10)–C(11)	119.6 (7)
–N(2)′	92.2 (1)	Mo–N(2)–C(3)	118.9 (4)	N(2)–C(5)–C(6)	119.0 (6)	N(1)–C(13)–C(14)	118.9 (6)
O(1)–Mo–O(2)′	176.6 (2)	–C(5)	123.4 (4)	–C(10)	118.7 (6)	–C(18)	120.2 (6)
–N(1)	90.0 (2)	C(3)–N(2)–C(5)	117.7 (5)	C(6)–C(5)–C(10)	122.3 (6)	C(14)–C(13)–C(18)	120.9 (6)
–N(2)′	89.8 (2)	O(1)–C(1)–O(2)	121.0 (6)	C(5)–C(6)–C(7)	117.8 (7)	C(13)–C(14)–C(15)	117.8 (7)
O(2)–Mo–N(1)	90.1 (2)	–C(2)	119.8 (7)	–C(12)	121.2 (6)	–C(20)	121.4 (6)
–N(2)′	89.8 (2)	O(2)–C(1)–C(2)	119.2 (6)	C(7)–C(6)–C(12)	121.0 (7)	C(15)–C(14)–C(20)	120.6 (7)
N(1)–Mo–N(2)′	175.4 (2)	C(15)–C(16)–C(17)	119.0 (7)	C(6)–C(7)–C(8)	120.4 (8)	C(14)–C(15)–C(16)	122.7 (8)
Mo–O(1)–C(1)	117.9 (4)	C(16)–C(17)–C(18)	120.6 (8)	C(7)–C(8)–C(9)	121.3 (7)	O(3)–C(21)–C(22)	100 (1)
Mo–O(2)–C(1)	117.6 (4)	C(13)–C(18)–C(17)	119.0 (7)	C(8)–C(9)–C(10)	119.8 (8)	C(21)–C(22)–C(23)	108 (2)
C(21)–O(3)–C(24)	118.1 (9)	–C(19)	120.9 (6)			C(22)–C(23)–C(24)	114 (2)
		C(17)–C(18)–C(19)	120.1 (7)			O(3)–C(24)–C(23)	98 (1)

Table VI. Bond Distances for $Mo_2[(PhN)_2CCH_3]_3(CH_3CO_2)$ (2)

atoms	dist, Å	atoms	dist, Å	atoms	dist, Å	atoms	dist, Å
Mo(1)–Mo(2)	2.082 (1)	N(4)–C(17)	1.340 (7)	C(6)–C(7)	1.41 (1)	C(22)–C(23)	1.41 (1)
–O(1)	2.165 (4)	–C(25)	1.418 (7)	C(7)–C(8)	1.38 (1)	C(23)–C(24)	1.42 (1)
–N(2)	2.160 (5)	N(5)–C(31)	1.338 (7)	C(8)–C(9)	1.37 (1)	C(25)–C(26)	1.396 (8)
–N(4)	2.132 (5)	–(39)	1.421 (8)	C(9)–C(10)	1.41 (1)	–C(30)	1.402 (9)
–N(5)	2.115 (4)	N(6)–C(31)	1.321 (7)	C(11)–C(12)	1.400 (9)	C(26)–C(27)	1.41 (1)
Mo(2)–O(2)	2.160 (4)	–C(33)	1.441 (7)	–C(16)	1.412 (9)	C(27)–C(28)	1.38 (1)
–N(1)	2.161 (5)	C(1)–C(2)	1.524 (9)	C(12)–C(13)	1.40 (1)	C(34)–C(35)	1.41 (1)
–N(3)	2.140 (5)	C(28)–C(29)	1.41 (1)	C(13)–C(14)	1.39 (1)	C(35)–C(36)	1.35 (1)
–N(6)	2.151 (5)	C(29)–C(30)	1.408 (9)	C(14)–C(15)	1.43 (1)	C(36)–C(37)	1.37 (1)
O(1)–C(1)	1.270 (7)	C(31)–C(32)	1.541 (8)	C(15)–C(16)	1.40 (1)	C(37)–C(38)	1.42 (1)
O(2)–C(1)	1.258 (7)	C(33)–C(34)	1.32 (1)	C(17)–C(18)	1.512 (8)	C(39)–C(40)	1.383 (9)
N(1)–C(3)	1.342 (7)	–C(38)	1.42 (1)	C(19)–C(20)	1.381 (9)	–C(44)	1.38 (1)
–C(5)	1.425 (7)	C(3)–C(4)	1.520 (9)	–C(24)	1.429 (9)	C(40)–C(41)	1.44 (1)
N(2)–C(3)	1.329 (7)	C(5)–C(6)	1.412 (9)	C(20)–C(21)	1.42 (1)	C(41)–C(42)	1.36 (1)
–C(11)	1.428 (7)	–C(10)	1.404 (9)	C(21)–C(22)	1.38 (1)	C(42)–C(43)	1.36 (1)
N(3)–C(17)	1.360 (7)					C(43)–C(44)	1.39 (1)
–C(19)	1.418 (7)						

Table VII. Bond Angles for $Mo_2[(PhN)_2CCH_3]_3(CH_3CO_2)$ (2)

atoms	angle, deg	atoms	angle, deg	atoms	angle, deg	atoms	angle, deg
Mo(2)–Mo(1)–O(1)	91.8 (1)	C(5)–C(6)–C(7)	118.5 (7)	C(42)–C(43)–C(44)	120.6 (9)	C(19)–C(24)–C(23)	118.8 (7)
–N(2)	92.7 (1)	C(6)–C(7)–C(8)	120.6 (8)	C(39)–C(44)–C(43)	120.9 (8)	N(4)–C(25)–C(26)	122.1 (6)
–N(4)	92.9 (1)	C(7)–C(8)–C(9)	121.6 (8)	C(3)–N(1)–C(5)	122.1 (5)	–C(30)	118.4 (6)
–N(5)	92.2 (1)	C(8)–C(9)–C(10)	119.1 (8)	Mo(1)–N(2)–C(3)	119.3 (4)	C(26)–C(25)–C(30)	119.2 (6)
O(1)–Mo(1)–N(2)	87.4 (2)	C(5)–C(10)–C(9)	120.3 (7)	–C(11)	119.4 (4)	C(25)–C(26)–C(27)	120.5 (7)
–N(4)	174.2 (2)	N(2)–C(11)–C(12)	121.8 (6)	C(3)–N(2)–C(11)	120.8 (5)	C(26)–C(27)–C(28)	120.0 (7)
–N(5)	84.8 (2)	–C(16)	117.8 (6)	Mo(2)–N(3)–C(17)	119.6 (4)	C(27)–C(28)–C(29)	120.0 (7)
N(2)–Mo(1)–N(4)	96.0 (2)	C(12)–C(11)–C(16)	120.3 (6)	–C(19)	119.6 (4)	C(28)–C(29)–C(30)	120.0 (8)
–N(5)	170.9 (2)	C(11)–C(12)–C(13)	118.8 (7)	C(17)–N(3)–C(19)	120.2 (5)	C(25)–C(30)–C(29)	120.0 (7)
N(4)–Mo(1)–N(5)	91.4 (2)	C(12)–C(13)–C(14)	121.8 (7)	Mo(1)–N(4)–C(17)	120.1 (4)	N(5)–C(31)–N(6)	116.1 (5)
Mo(1)–Mo(2)–O(2)	92.1 (1)	C(13)–C(14)–C(15)	119.5 (7)	–C(25)	118.9 (4)	–C(32)	122.6 (6)
–N(1)	92.2 (1)	C(14)–C(15)–C(16)	118.7 (8)	C(17)–N(4)–C(25)	120.4 (5)	N(6)–C(31)–C(32)	121.3 (6)
–N(3)	92.4 (1)	C(11)–C(16)–C(15)	120.9 (7)	Mo(1)–N(5)–C(31)	119.8 (4)	N(6)–C(33)–C(34)	121.7 (7)
–N(6)	92.4 (1)	N(3)–C(17)–N(4)	115.0 (6)	–C(35)	118.4 (4)	–C(38)	117.6 (6)
O(2)–Mo(2)–N(1)	86.6 (2)	–C(18)	121.1 (6)	C(31)–N(5)–C(35)	121.4 (5)	C(34)–C(33)–C(38)	120.4 (7)
–N(3)	174.6 (2)	N(4)–C(17)–C(18)	123.3 (5)	Mo(2)–N(6)–C(31)	118.4 (4)	C(33)–C(34)–C(35)	119.2 (8)
–N(6)	84.2 (2)	N(3)–C(19)–C(20)	122.4 (6)	–C(33)	117.9 (4)	C(34)–C(35)–C(36)	123.1 (9)
N(1)–Mo(2)–N(3)	96.2 (2)	–C(24)	117.0 (6)	C(31)–N(6)–C(33)	121.1 (5)	C(35)–C(36)–C(37)	118.3 (8)
–N(6)	169.8 (2)	C(20)–C(19)–C(24)	120.4 (6)	O(1)–C(1)–O(2)	123.7 (6)	C(36)–C(37)–C(38)	120.4 (8)
N(3)–Mo(2)–N(6)	92.7 (2)	C(19)–C(20)–C(21)	119.4 (7)	–C(2)	118.4 (6)	C(33)–C(38)–C(37)	118.4 (8)
Mo(1)–O(1)–C(1)	115.9 (4)	C(20)–C(21)–C(22)	121.7 (7)	O(2)–C(1)–C(2)	118.0 (6)	N(5)–C(39)–C(40)	122.3 (6)
Mo(2)–O(2)–C(1)	116.0 (4)	C(21)–C(22)–C(23)	119.0 (7)	N(1)–C(3)–N(2)	116.1 (5)	–C(44)	118.5 (6)
Mo(2)–N(1)–C(3)	119.3 (4)	C(22)–C(23)–C(24)	120.7 (7)	–C(4)	121.1 (6)	C(40)–C(39)–C(44)	119.0 (7)
–C(5)	118.3 (4)	C(40)–C(41)–C(42)	120.2 (8)	N(2)–C(3)–C(4)	122.6 (6)	C(39)–C(40)–C(41)	119.1 (8)
C(6)–C(5)–C(10)	119 (6)	C(41)–C(42)–C(43)	120.3 (8)	N(1)–C(5)–C(6)	122.2 (6)		
				–C(10)	117.6 (6)		

Table VIII. Structural Parameters for Dimolybdenum Compounds with Axial THF Ligands

compd	Mo–Mo, Å	Mo–O_{THF}, Å	ref
$Mo_2(S_2CCH_3)_4 \cdot 2THF$	2.141 (1), 2.133 (1)	2.667 (7), 2.712 (6)	8
$Mo_2(S_2CC_6H_5)_4 \cdot 2THF$	2.139 (2)	2.727 (7)	8
$Mo_2(S_2COC_2H_5)_4 \cdot 2THF$	2.125 (1)	2.795 (1)	9
$Mo_2[(2,6\text{-xylyl})NC(H)O]_4 \cdot 2THF$	2.113 (1)	2.582 (7)	10
$Mo_2[[(2,6\text{-xylyl})N]_2CCH_3]_2(CH_3CO_2)_2 \cdot 4THF$	2.107 (1)	2.709 (6)	this work

Table IX. Weighted Least-Squares Planes[a] and Dihedral Angles for $Mo_2[(PhN)_2CCH_3]_3(CH_3CO_2)$ (2)

plane	atoms	*A*	*B*	*C*	*D*
P_1	N(5), Mo(1), Mo(2), N(6)	0.7026	0.3071	−0.6419	10.9084
P_2	C(33), C(34), C(35), C(36), C(37), C(38)	−0.0896	−0.7274	−0.6804	−15.1447
P_3	C(39), C(40), C(41), C(42), C(43), C(44)	0.0803	0.9581	−0.2749	14.0427
P_4	N(1), Mo(2), Mo(1), N(2)	0.6846	0.4391	−0.5818	12.6641
P_5	C(5), C(6), C(7), C(8), C(9), C(10)	0.7813	−0.3815	−0.4914	4.5652
P_6	C(11), C(12), C(13), C(14), C(15), C(16)	0.2160	−0.3712	−0.9031	−4.3621
P_7	N(3), Mo(2), Mo(1), N(4)	0.0978	−0.9156	−0.3901	−12.4237
P_8	C(19), C(20), C(21), C(22), C(23), C(24)	0.2674	0.9023	−0.3383	12.9617
P_9	C(25), C(26), C(27), C(28), C(29), C(30)	0.5456	0.8375	−0.0309	17.0285

planes	angle, deg	planes	angle, deg	planes	angle, deg
P_1/P_2	81.3	P_4/P_5	49.1	P_7/P_8	131.9
P_1/P_3	58.2	P_4/P_6	59.3	P_7/P_9	131.9

[a] The equation of the plane is of the Form $AX + BY + CZ - D = 0$, where *A*, *B*, *C*, and *D* are constants and *X*, *Y*, and *Z* are orthogonalized coordinates.

Table X. Comparison of the M–O Distances in Mixed-Ligand Dimolybdenum Compounds

compd	Mo–Mo, Å	Mo–O, Å	ref
$Mo_2(CH_3CO_2)_2(pd)_2$	2.129 (1)	2.10 (2)	2
$[Mo_2(CH_3CO_2)Cl_4](Ph_4As)_2 \cdot 2MeOH$	2.086 (2)	2.12 (1)	2
$Mo_2((pz)_2BEt_2)_2(CH_3CO_2)_2 \cdot CS_2$	2.129 (1)	2.110 (5)	4
$Mo_2((pz)_2BH_2)_2(CH_3CO_2)_2$	2.147 (3)	2.11 (2)	4
$Mo_2(PhNC(CH_3)CHC(CH_3)O)_2 \cdot (CH_3CO_2)_2$	2.131 (1)	2.088 (5)	3
$Mo_2(ambt)_3(CH_3CO_2) \cdot 2THF$	2.093 (1)	2.16 (1)	5

compound $Mo_2[(2,6\text{-xylyl})NC(H)O]_2 \cdot 2THF$[10] also has axially coordinated THF ligands. The Mo–O(THF) distance of 2.709 (6) Å in **1** is comparable to those in the dithio ligand derivatives (see Table VIII). The Mo–Mo distance of 2.107 (1) Å is slightly longer than that observed in $Mo_2(CH_3CO_2)_4$[12] (2.0934 (8) Å) and the related compound $Mo_2[(PhN)_2CPh]_4$[13] (2.090 (1) Å). This increase in the Mo–Mo bond length is most likely due to the presence of the coordinated THF molecules, since, at least to a first approximation, in the absence of coordinated THF we might expect the bond length to be an average of that of the bond lengths in $Mo_2(CH_3CO_2)_4$[12] and $Mo_2[(PhN)_2CPh]_4$,[13] i.e., ~2.092 Å. The average Mo–N distance of 2.162 (5) Å is approximately the same as that found in $Mo_2[(PhN)_2CPh]_4$[13] (2.15 (1) Å). The average Mo–O distance, 2.108 (4) Å, is essentially identical with that in $Mo_2(CH_3CO_2)_4$.[12] The remaining distances show no unusual features.

The structure of compound **2** was solved in the triclinic space group $P\bar{1}$ with each molecule occupying a general position within the unit cell. Each molecule has three $(PhN)_2CCH_3$ ligands and one acetato ligand. All of the phenyl rings of the $(PhN)_2CCH_3$ ligands, except one, are rotated by approximately 56° relative to the NMo_2N planes of each ligand (see Table IX). It is of interest to note that in order to minimize steric repulsive interactions between the phenyl rings the two trans $(PhN)_2CCH_3$ ligands are bent toward the acetato ligand by an average of ca. 4.8°. The Mo–Mo bond length, 2.082 (1) Å, is somewhat shorter than that in the related compound $Mo_2[(PhN)_2CPh]_4$[13] (2.090 (1) Å) but essentially the same as that found in $Mo_2[(PhN)_2N]_4$[14] (2.083 (2) Å). Similar distances are also found in $Mo_2[(CH_2)_2P(CH_3)_2]_4$[15] (2.082 (1) Å), $Mo_2[PhNC(CH_3)O]_4 \cdot 2THF$[16] (2.086 (1) Å), and $Mo_2(dmmp)_4 \cdot CH_2Cl_2$[17] (2.083 (2) Å) (dmmp = the anion of 4,6-dimethyl-2-mercaptopyrimidine). The average Mo–O distance, 2.162 (5) Å, is considerably longer than the average of about 2.11 Å found in the carboxylato compounds and the other known mixed-ligand compounds (see Table X). On the other hand, it is essentially identical with that found in $Mo_2(ambt)_3(CH_3CO_2) \cdot 2THF$[5] (2.16 (1) Å). There are two distinctly different Mo–N bond lengths in the compound. The average Mo–N bond length to the $[(PhN)_2CCH_3]$ ligand that is trans to the acetato ligand is 2.136 (5) Å whereas the remaining Mo–N distances average 2.147 (5) or 2.157 (5) Å omitting the abnormally short Mo–N(5) distance.

In a previous report of the structure of $Mo_2(ambt)_3(CH_3CO_2) \cdot 2THF$,[5] it was suggested that the increase in the Mo–O(OAc) bond length was a manifestation of a trans effect in the molecule. The unusually long Mo–O(OAc) bond length in **2** together with the shortening of the Mo–N bond lengths of the ligand trans to the acetato ligand adds further support to the suggestion that a trans effect is operative in these species.

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Registry No. **1**, 76037-17-7; **2**, 76037-15-5; *N,N'*-bis(2,6-xylyl)-acetamidine, 54708-07-5; $Mo_2[CH_3CO_2]_4$, 14221-06-8; 2′,6′-dimethylacetanilide, 2198-53-0.

Supplementary Material Available: Tables of observed and calculated structure factors (28 pages). Ordering information is given on any current masthead page.

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