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Chelate Rings in a Quadruply Bonded Dimetal Compound: Preparation and Structure of Diacetatodi(4-phenylimino-2-pentanonato)dimolybdenum

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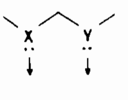
Lithium (4-phenylimino)-2-pentanonide reacts with $Mo_2(O_2CCH_3)_4$ to replace two acetate ligands and afford red, crystalline $Mo_2(O_2CCH_3)_2[PhNC(CH_3)CHC(CH_3)O]_2$. The crystal and molecular structures have been determined by X-ray crystallography. The crystals belong to space group $P\bar{1}$ with $a = 11.723(3)$ Å, $b = 10.402(2)$ Å, $c = 12.624(2)$ Å; $\alpha = 112.88(1)°$, $\beta = 95.04(1)°$, $\gamma = 107.01(1)°$, *and* $Z = 2$. *The structure has a cisoid arrangement of the bridging acetate ligands and one chelate ring on each metal atom. The unsymmetrical chelate ligands are so arranged that the virtual symmetry is C_2. The Mo–Mo distance is 2.131(1) Å.*

Introduction

The ligands most frequently found in compounds with a quadruple bond between a pair of metal atoms [1] are the bidentate bridging ones, 1, that prefer to, or at least can, direct their two available electron pairs along approximately parallel lines towards acceptor sites roughly 2.2 Å apart. The majority of quadruply bonded M_2 containing species contain ligands of this time either exclusively (*e.g.*, $M_2(O_2CR)_4$, $[M_2(SO_4)_4]^{n-}$), or mixed with some unidentate ligands (*e.g.*, $M_2(O_2CR)_2X_4$, $M_2(Ph_2PCH_2PPh_2)_2X_4$), although important species are known containing only unidentate ligands (*e.g.*, $[M_2X_8]^{n-}$, $M_2L_4X_4$).

The type of ligand that has been virtually unknown in complexes of quadruply-bonded M_2 species is the bidentate *chelating* ligand, *i.e.*, a ligand directing both of its donor pairs towards the same metal atom, 2, and thus incorporating it in a 5- or 6-membered chelate ring. There appears to be only one known example of a complex in which only ligands of this type occur and this is of a rather unique type, viz., $[Cr_2(C_4H_8)_4]^{4-}$, where C_4H_8 is the 1,4-tetramethylene group [2]. The only other examples of compounds containing *any* such ligands are a mixed acetato–pyrazolylborate complex of molybdenum [3] and the mixed acetatoacetylacetonato complex, recently reported by Garner *et al.* [4], both having structures of type 3.



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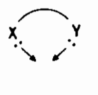

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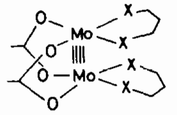

3

We report here the results of a study in which a β-ketoimido ligand, $[PhNC(CH_3)CHC(CH_3)CO]^-$ has been used to replace acetato ligands in $Mo_2(O_2CCH_3)_4$. Again, complete replacement was not achieved and the product is another molecule with a structure of type 3.

Experimental

Preparation

The ligand 4-phenylimino-2-pentanone, $C_6H_5NC(CH_3)C(H)C(CH_3)OH$, was prepared by condensation of 2,4-pentanedione with aniline under reflux. It was purified by distillation (bp. 155 °C/ 1 mm; m.p. 52 °C) and characterized by ^1H nmr: δ 1.97 (s, 3H), 2.10 (s, 3H), 5.27 (s, 1H), 7.3 (m, 5H), 12.9 (broad, 1H).

$Mo_2(CH_3COO)_2[C_6H_5NC(CH_3)C(H)C(CH_3)O]_2$

4-Phenylimine-2-pentanone (0.35 g, 2 mmol) was dissolved in 20 mL of THF and 1.3 ml (2 mmol) of a 1.6 m solution of n-butyllithium in hexane was added to obtain the colorless anion.

Dimolybdenum tetraacetate (0.22 g, 0.5 mmol) was added under stirring and the solution developed a dark red color. After 12 hours the mixture was filtered into a tubular flask, and a layer of hexane (25 mL) was placed carefully over the solution. After a few days red crystals which had grown by slow diffusion were collected for X-ray diffraction. Evaporation of the remaining solution produced more of the air-sensitive red material, which, according to mass spectroscopic measurements (230 °C, 10^{-6} mm), consisted of but one species with the formula $C_{26}H_{30}N_2O_6Mo_2$: Exp. 662.013291; Calc. 662.02135.

X-ray Data Collection

The crystals of $Mo_2(O_2CCH_3)[C_6H_5NC(CH_3)CHC(CH_3)O]_2$ were transferred in a nitrogen-filled dry box to a petri dish containing degassed mineral oil. The crystals were then removed from the dry box and examined under a microscope. A crystal that appeared suitable for X-ray analysis was then transferred to a slide on which there was a drop of soft epoxy. The crystal was coated with epoxy, drawn up into a capillary and mounted on a Syntex P$\bar{1}$ automatic diffractometer.

Preliminary rotation and oscillation photographs indicated that the crystal was triclinic and examination of selected reflections indicated that it was a single crystal of good quality. Several intense reflections had ω scans with widths at half-height of less than 0.3°. Careful centering on 15 reflections in the range $23° < 2\theta < 29°$ selected to give varied crystal orientations, gave the unit cell parameters a = 11.723(3) Å, b = 10.402(2) Å, c = 12.624(2) Å, α = 112.88(1)°, β = 95.04(1)°, γ = 107.01(1)°. The volume, 1320.8(1.2) Å^3, is consistent with $Z = 2$. The procedures preliminary to data collection have been described previously [5].

The intensity data were collected using Mo Kα (λ = 0.71073) radiation monochromatized in the incident beam with a graphite crystal. A total of 3456 independent reflections with $0 < 2\theta < 45°$ were collected at 22 ± 4 °C using the θ–2θ scan technique. Variable scan rates from 4–24° min^{-1} were used with a scan range of Kα_1 − 1.0° to Kα_2 + 1.0° and a scan to background time ratio of 2. The intensities of three standard reflections, measured every 97 reflec-

TABLE I. Positional and Thermal Parameters and their Estimated Standard Deviations.

Atom	*X*	*Y*	*Z*	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)
Mo(1)	0.97065(6)	0.02879(6)	0.84368(5)	2.51(3)	2.39(2)	2.42(2)	0.85(2)	0.42(2)	0.91(2)
Mo(2)	0.99340(6)	0.04558(7)	0.68334(5)	2.37(3)	2.64(2)	2.31(2)	0.82(2)	0.27(2)	0.88(2)
O(1)	0.8149(5)	0.0863(5)	0.8389(4)	3.4(2)	3.9(2)	3.0(2)	2.2(2)	1.0(2)	1.5(1)
O(2)	1.1119(5)	−0.0272(5)	0.8991(5)	3.2(2)	2.7(2)	4.0(2)	1.1(2)	0.0(2)	1.2(1)
O(3)	1.0762(5)	0.2570(5)	0.9445(4)	3.8(2)	2.9(2)	2.7(2)	0.7(2)	0.3(2)	0.7(1)
O(4)	0.8302(5)	0.0913(5)	0.6668(4)	3.0(2)	3.9(2)	3.1(2)	1.5(2)	0.9(2)	1.8(1)
O(5)	0.9014(5)	−0.1644(5)	0.5471(4)	2.4(2)	3.2(2)	3.1(2)	0.8(2)	0.1(2)	0.5(2)
O(6)	1.0906(5)	0.2768(5)	0.7770(4)	3.4(2)	3.0(2)	3.1(2)	1.0(2)	0.7(2)	1.5(1)
N(1)	0.8599(6)	−0.2030(6)	0.7847(5)	2.2(2)	2.8(2)	3.0(2)	0.6(2)	0.4(2)	1.3(2)
N(2)	1.1590(6)	−0.0019(6)	0.6528(5)	2.4(2)	2.2(2)	2.3(2)	0.5(2)	0.6(2)	0.6(2)
C(1)	1.2506(7)	−0.1560(8)	0.8914(7)	2.7(3)	4.6(3)	4.7(3)	2.0(2)	0.9(3)	2.5(2)
C(2)	1.1213(7)	−0.1594(8)	0.8609(6)	2.9(3)	4.4(3)	2.4(3)	1.5(2)	0.6(3)	1.6(2)
C(3)	1.0278(7)	−0.2911(8)	0.8000(7)	2.8(3)	4.0(3)	4.1(3)	1.6(2)	1.3(3)	2.3(2)
C(4)	0.9024(7)	−0.3118(8)	0.7732(6)	3.3(3)	2.8(2)	2.8(3)	0.5(2)	0.7(3)	1.6(2)
C(5)	0.8113(9)	−0.4706(9)	0.7299(7)	5.0(5)	2.8(3)	4.4(4)	0.4(3)	1.1(4)	1.4(3)
C(6)	0.7291(7)	−0.2368(8)	0.7635(6)	2.7(3)	2.9(3)	3.0(3)	0.8(2)	0.5(3)	1.1(2)
C(7)	0.6639(8)	−0.2719(9)	0.6527(7)	3.6(4)	4.4(3)	3.2(3)	1.1(3)	0.3(3)	1.8(2)
C(8)	0.5383(8)	−0.2992(10)	0.6358(8)	3.0(4)	6.2(4)	3.5(4)	1.5(3)	0.1(3)	1.2(3)
C(9)	0.4787(8)	−0.2854(9)	0.7298(8)	3.7(4)	4.7(3)	5.1(4)	1.4(3)	1.7(3)	2.5(3)
C(10)	0.5481(8)	−0.2475(9)	0.8394(7)	3.4(4)	4.1(3)	3.8(3)	1.2(3)	1.0(3)	1.8(2)
C(11)	0.6721(8)	−0.2232(8)	0.8577(7)	2.8(3)	3.5(3)	3.8(3)	0.5(3)	1.4(3)	1.6(2)
C(12)	0.8411(8)	−0.4220(8)	0.4187(7)	2.9(4)	2.3(3)	3.8(4)	0.0(3)	0.0(3)	0.1(3)
C(13)	0.9405(8)	−0.2740(8)	0.5032(6)	3.6(3)	3.1(3)	1.8(3)	1.1(2)	0.7(3)	1.0(2)
C(14)	1.0600(7)	−0.2649(8)	0.5227(7)	3.0(3)	3.5(3)	3.0(3)	1.1(2)	0.7(3)	1.3(2)
C(15)	1.1639(7)	−0.1330(8)	0.5865(6)	3.4(3)	4.3(3)	2.7(3)	1.8(2)	1.0(3)	1.8(2)
C(16)	1.2873(7)	−0.1526(9)	0.5734(8)	2.8(3)	4.8(3)	4.5(4)	2.2(2)	0.5(3)	1.4(3)
C(17)	1.2722(7)	0.1245(8)	0.7035(6)	1.8(3)	3.4(3)	2.4(3)	0.8(2)	0.3(2)	1.1(2)
C(18)	1.3244(7)	0.1886(8)	0.6347(7)	2.7(3)	4.4(3)	3.8(3)	0.9(3)	1.0(3)	2.5(2)
C(19)	1.4331(8)	0.3178(9)	0.6882(7)	3.8(4)	6.0(3)	4.9(3)	1.9(3)	1.3(3)	3.4(2)
C(20)	1.4851(8)	0.3798(8)	0.8083(7)	3.2(3)	3.9(3)	4.3(3)	1.4(3)	0.7(3)	1.9(2)
C(21)	1.4300(8)	0.3104(9)	0.8750(7)	3.4(4)	4.0(3)	3.5(3)	1.0(3)	0.5(3)	1.5(2)
C(22)	1.3223(8)	0.1823(9)	0.8223(7)	3.1(4)	3.7(3)	2.6(3)	0.8(3)	−0.3(3)	0.8(2)
C(23)	0.7740(8)	0.1002(8)	0.7497(7)	3.0(3)	3.0(3)	4.2(4)	1.0(2)	0.2(3)	1.1(2)
C(24)	0.6527(8)	0.1277(10)	0.7405(8)	3.9(3)	6.8(4)	5.3(4)	3.6(3)	1.6(3)	3.0(3)
C(25)	1.1122(8)	0.3337(8)	0.8887(7)	3.7(4)	2.4(3)	4.0(3)	0.6(3)	0.2(3)	1.3(2)
C(26)	1.1831(12)	0.4994(10)	0.9565(8)	9.0(7)	3.5(4)	3.9(4)	0.5(4)	−0.5(5)	0.9(3)

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

tions, showed no significant variation during the period of data collection. Lorentz and polarization corrections were applied [6]. The Mo Kα linear absorption coefficient for the crystal was 9.77 cm^{-1}; absorption corrections were, therefore, considered unnecessary.

Solution and Refinement [*6*]

The structure was solved in the triclinic space group P$\bar{1}$. The positions of the two independent molybdenum atoms were found by solution of the three-dimensional Patterson function. Independent application of direct methods confirmed the positions of the Mo atoms. The remaining non-hydrogen atoms were found from successive Fourier and difference syntheses. Full-matrix anisotropic least squares refinement of all heavy atom coordinates and temperature factors gave final discrepancy factors of

$$R_1 = (\Sigma||F_o| - |F_c||)/\Sigma|F_o| = 0.041$$

$$R_2 = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2} = 0.055$$

and a goodness of fit parameter of 1.15. The function $\Sigma w(|F_o| - |F_c|)^{1/2}$ was minimized with the weighting factor, w, equal to $4F_o^2/\sigma(F_o^2)^2$. All structure factor calculations and least-squares refinements were executed using only those 2480 reflections for which $F_o^2 > 3\sigma(F_o)^2$. Atomic scattering factors were those of Cromer and Waber [7]. Anomalous dispersion effects were included in the scattering factors of molybdenum [8].

Results

The structure refined very smoothly to convergence using anisotropic thermal vibration parameters for all atoms. The calculated and observed structure factors are available as supplementary material. The atomic positional and thermal parameters are listed in Table I. The structure of the molecule is shown in Fig. 1 where the atomic numbering scheme is defined. There are two molecules in the unit cell, related to each other by a crystallographic inversion center. The molecules, which have no imposed crystallographic symmetry, are packed with entirely normal van der Waals contacts.

The bond lengths and bond angles are listed in Tables II and III, respectively. The molecule has virtual C_2 symmetry, with the C_2 axis bisecting the Mo–Mo bond so as to allow rotational interchange of the two acetato and two β-ketoiminato ligands. Fig. 2 presents a view directly down this axis.

Discussion

In the preparation of $Mo_2(O_2CCH_3)_2[PhNC(CH_3)CHC(CH_3)O]_2$ a sufficient quantity of the β-ketoiminato ion, as the lithium salt, was used to displace all four acetate ligands. Moreover, the reaction commenced immediately and appeared to reach completion in a matter of only a few minutes. Nevertheless, the product has only two of the acetate groups replaced. As can be clearly seen in Fig. 2, the phenyl groups would not interfere, sterically, with

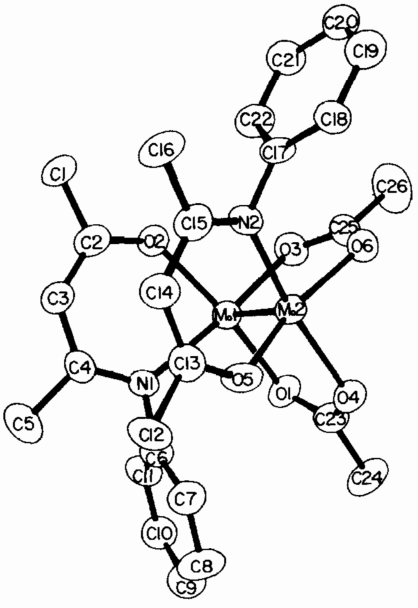

Fig. 1. The $Mo_2(O_2CCH_3)_2[PhNC(CH_3)CHC(CH_3)O]_2$ molecule, with the atomic labeling scheme defined.

TABLE II. Bond Distances (Å).

Atoms	*Distance*	*Atoms*	*Distance*
Mo(1)–Mo(2)	2.131(1)	C(6)–C(7)	1.39(1)
Mo(1)–O(1)	2.084(5)	C(6)–C(11)	1.39(1)
Mo(1)–N(1)	2.158(6)	C(7)–C(8)	1.39(1)
Mo(1)–O(2)	2.061(5)	C(8)–C(9)	1.41(1)
Mo(1)–O(3)	2.109(5)	C(9)–C(10)	1.39(1)
Mo(2)–O(4)	2.114(5)	C(10)–C(11)	1.38(1)
Mo(2)–O(5)	2.067(5)	C(13)–C(12)	1.52(1)
Mo(2)–N(2)	2.169(6)	C(13)–C(14)	1.37(1)
Mo(2)–O(6)	2.106(5)	C(14)–C(15)	1.42(1)
O(1)–C(23)	1.26(1)	C(15)–C(16)	1.53(1)
N(1)–C(4)	1.33(1)	C(17)–C(18)	1.37(1)
N(1)–C(6)	1.44(1)	C(17)–C(22)	1.38(1)
O(2)–C(2)	1.311(9)	C(18)–C(19)	1.42(1)
O(3)–C(25)	1.27(1)	C(19)–C(20)	1.39(1)
O(4)–C(23)	1.27(1)	C(20)–C(21)	1.39(1)
O(5)–C(13)	1.295(9)	C(21)–C(22)	1.41(1)
N(2)–C(15)	1.32(1)	C(23)–C(24)	1.53(1)
N(2)–C(17)	1.44(1)	C(25)–C(26)	1.51(1)
O(6)–C(25)	1.27(1)		
C(1)–C(2)	1.52(1)		
C(2)–C(3)	1.35(1)		
C(3)–C(4)	1.41(1)		
C(4)–C(5)	1.53(1)		

TABLE III. Bond Angles, Degrees.

Atoms	*Angle*	*Atoms*	*Angle*
Mo(2)–Mo(1)–O(1)	90.8(2)	N(1)–C(4)–C(3)	124.3(7)
Mo(2)–Mo(1)–N(1)	103.3(2)	N(1)–C(4)–C(5)	119.0(8)
Mo(2)–Mo(1)–O(2)	107.8(2)	C(3)–C(4)–C(5)	116.7(8)
Mo(2)–Mo(1)–O(3)	91.4(2)	N(1)–C(6)–C(7)	119.8(8)
O(1)–Mo(1)–N(1)	91.2(2)	N(1)–C(6)–C(11)	118.9(7)
O(1)–Mo(1)–O(2)	161.4(2)	C(7)–C(6)–C(11)	121.1(8)
O(1)–Mo(1)–O(3)	88.6(2)	C(6)–C(7)–C(8)	118.6(8)
N(1)–Mo(1)–O(2)	85.4(2)	C(7)–C(8)–C(9)	121.2(9)
N(1)–Mo(1)–O(3)	165.2(2)	C(8)–C(9)–C(10)	118.0(9)
O(2)–Mo(1)–O(3)	90.0(2)	C(9)–C(10)–C(11)	121.8(9)
Mo(1)–Mo(2)–O(4)	91.4(2)	C(6)–C(11)–C(10)	119.1(8)
Mo(1)–Mo(2)–O(5)	107.0(2)	O(5)–C(13)–C(12)	114.3(7)
Mo(1)–Mo(2)–N(2)	104.3(2)	O(5)–C(13)–C(14)	125.9(8)
Mo(1)–Mo(2)–O(6)	90.9(2)	C(12)–C(13)–C(14)	119.7(8)
O(4)–Mo(2)–O(5)	88.1(2)	C(13)–C(14)–C(15)	126.4(8)
O(4)–Mo(2)–N(2)	164.2(2)	N(2)–C(15)–C(14)	124.5(7)
O(4)–Mo(2)–O(6)	88.7(2)	N(2)–C(15)–C(16)	120.8(8)
O(5)–Mo(2)–N(2)	85.7(2)	C(14)–C(15)–C(16)	114.7(8)
O(5)–Mo(2)–O(6)	161.9(2)	N(2)–C(17)–C(18)	119.9(7)
N(2)–Mo(2)–O(6)	92.6(2)	N(2)–C(17)–C(22)	118.5(7)
C(4)–N(1)–C(6)	118.9(6)	C(18)–C(17)–C(22)	121.5(8)
C(15)–N(2)–C(17)	117.8(6)	C(17)–C(18)–C(19)	118.8(8)
O(2)–C(2)–C(1)	114.2(7)	C(18)–C(19)–C(20)	121.0(8)
O(2)–C(2)–C(3)	125.6(8)	C(19)–C(20)–C(21)	118.4(8)
C(1)–C(2)–C(3)	120.2(8)	C(20)–C(21)–C(22)	120.7(8)
C(2)–C(3)–C(4)	126.4(8)		
C(17)–C(22)–C(21)	119.5(8)		
O(1)–C(23)–O(4)	121.9(8)		
O(1)–C(23)–C(24)	119.0(9)		
O(4)–C(23)–C(24)	119.1(8)		
O(3)–C(25)–O(6)	122.4(7)		
O(3)–C(25)–C(26)	119.5(8)		
O(6)–C(25)–C(26)	118.1(8)		
Mo(1)–O(1)–C(23)	118.9(8)		
Mo(1)–N(1)–C(4)	125.5(8)		
Mo(1)–N(1)–C(6)	115.5(8)		
Mo(1)–O(2)–C(2)	127.9(8)		
Mo(1)–O(3)–C(25)	117.2(8)		
Mo(2)–O(5)–C(13)	128.5(8)		
Mo(2)–N(2)–C(15)	125.8(8)		
Mo(2)–N(2)–C(17)	116.2(8)		
Mo(2)–O(6)–C(25)	116.7(8)		
Mo(2)–O(4)–C(23)	117.9(8)		

the replacement of the other two acetate groups. It does not seem therefore that the replacement of only two acetate groups by the chelating ligands is the result either of failure to allow a sluggish reaction to reach completion, or of steric hindrance. Thus, there may be an electronic factor favoring the formation of the product observed both here and in the case of the acetylacetonate [4]. In the preparation of the latter compound, however, the situation was somewhat clouded by the fact that severe conditions were required even to obtain the disubstituted compound.

It is important to note, however, that the reaction of $Mo_2(O_2CCH_3)_4$ with $Na(Et_2Bpz_2)$ in slight excess

TABLE IV. Comparison of $Mo_2(O_2CCH_3)_2(LL)_2$ Structures[a].

Ligand LL	*Mo–Mo*, Å	*Mo–O(Ac)*, Å	*Mo–O(LL)*, Å	*Mo–N(LL)*, Å	*Ref.*
$Et_2B(C_3N_2H_2)_2$	2.129(1)	2.110(8)		2.162(5)	3
$C_5H_7O_2$	2.129(1)	2.10(2)	2.09(1)	–	4
$PhNC(CH_3)CHC(CH_3)O$	2.131(1)	2.10(2)	2.064(3)	2.163(5)	b

[a] Arithmetic average values of Mo–O and Mo–N distances are given with mean deviations from the average in parentheses. [b] This work.

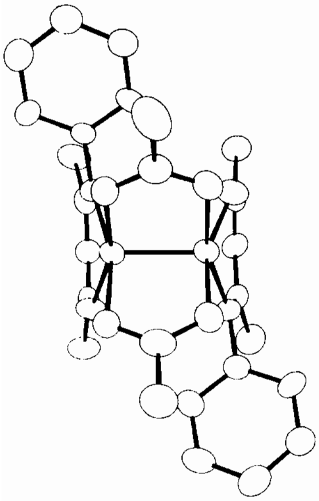

Fig. 2. A view of the molecule directly down the virtual C_2 axis.

did appear to give $Mo_2(pz_2BEt_2)_4$ as well as the disubstituted compound, although the completely substituted product has not yet been fully (*i.e.*, crystallographically) characterized. We intend to make further efforts to characterize this, and possibly other, tetrachelate complexes of Mo_2^{4+}.

The structure of $Mo_2(O_2CCH_3)_2[PhNC(CH_3)CHC(CH_3)O]_2$ is very similar to those of the pyrazolylborate and acetylacetonate complexes, and Table IV compares corresponding bond lengths. The present substance provides a test of the previous suggestion [4] that the Mo–Mo distance in the $Mo_2(O_2CMe)_2(acac)_2$ molecule was greater than that in $Mo_2(O_2CMe)_4$ because of a weak intermolecular axial bond (2.709 Å) of the Mo•••O(Ac) type. This is unlikely to be correct since the present bond length is essentially the same but the closest intermolecular contact in the axial direction is 3.47 Å.

The Mo–Mo–O(OAc) angles average 91.1(2)° which is the typical bonding angle for acetate groups The Mo–Mo–X (chelate) (X = N, O) angles average 105.6(2)°. The remaining angles about the molybdenum atoms range from 85.4(2) to 92.6(2). The geometry about the molybdenum atoms can therefore be described as a distorted square pyramid.

All distances and angles within the ligands have, within experimental error, the expected values. The C–O distances for the chelate and the acetate ligands average 1.308(9) Å and 1.27(1) Å, respectively, and reflect the retention of partial double bond character in these bonds upon coordination. The N–C (non-phenyl) distances in the chelating ligand average 1.32(1) Å and also reflect some double bond character in these bonds. The N–C (phenyl) distances average 1.44(1) Å.

Partial delocalization in the chelate is also reflected in the C–C distances within the six-member chelate ring which average 1.37(1) Å. This is in contrast to the $C–CH_3$ distances for the chelate which average 1.52(1) Å.

It is interesting to note that the C–C distances nearest the oxygen atom in the chelates are slightly shorter 1.36(1) Å, than the C–C distance nearest the nitrogen atom 1.41(1) Å, although the difference is not statistically definite. The C–C distances in the acetate ions have an average value of 1.52(1) Å.

The C–C distances in the phenyl groups range from 1.37(1) Å to 1.42(1) Å with an average value of 1.39(1) Å and the C–C–C angles within the rings range from 118.0(9)° to 121.8(9)° with the average being 120.0(9)°.

The phenyl rings are oriented perpendicular to the chelate rings (dihedral angles 89.5° and 78.4°). This orientation appears to be a result of steric interactions between the phenyl ring and the adjacent methyl group on the chelate ligand.

As can be seen in Fig. 2, the six-membered metal chelate rings adopt half chair conformations, the dihedral angles between the N–C–C–C–O and N–Mo–O planes (Table V) being 13.3° and 16.4°. This type of bending is characteristic of six-membered β-ketonato- and β-ketoiminatometal rings in general [9].

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TABLE V. Some Planes and Dihedral Angles in the Chelate Rings.

	Displacementss	*Err.*
Plane = P_1 N(1)–Mo(1)–O(2)		
A = 0.4105	N(1) = 0.0	0.006
B = 0.1915	Mo(1) = 0.0	0.001
C = −0.8915	O(2) = 0.0	0.005
D = −5.0962		
Plane = P_2 O(2)–C(2)–C(3)–C(4)–N(1)		
A = 0.2570	O(2) = −0.017	0.005
B = 0.4304	C(2) = 0.024	0.007
C = −0.8653	C(3) = 0.039	0.008
D = −7.9110	C(4) = −0.079	0.007
	N(1) = 0.003	0.006
Plane = P_3 N(2)–Mo(2)–O(5)		
A = 0.0013	N(2) = 0.0	0.006
B = 0.7539	Mo(2) = 0.0	0.001
C = −0.6569	O(5) = 0.0	0.005
D = −7.5429		
Plane = P_4 O(5)–C(13)–C(14)–C(15)–N(2)	O(5) = 0.011	0.005
A = 0.1176	C(13) = −0.012	0.007
B = 0.6034	C(14) = −0.036	0.008
C = −0.7887	C(15) = 0.063	0.008
D = −6.4577	N(2) = −0.023	0.006
Angles between Planes:		
P_1/P_2 16.4°		
P_3/P_4 13.3°		

Supplementary Material Available

A table of observed and calculated structure factors (11 pages) is available on request from F.A.C.

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