Bis{[1-(1H-inden-3-yl)cyclopentyl]-η^5-cyclopentadienyl}hexacarbonyldimolydenum(I)(*Mo—Mo*)

Jin Lin, Hai-Bin Song and Shan-Sheng Xu*

State Key Laboratory of Elemento-Organic Chemistry, College of Chemistry, Nankai University, Tianjin 300071, People's Republic of China
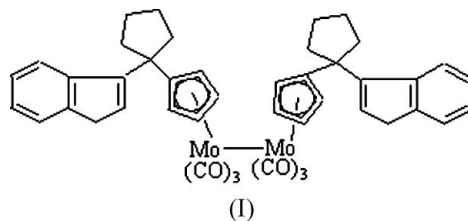
Correspondence e-mail: shanshengxu@126.com

The title centrosymmetric complex, $[Mo_2(C_{19}H_{19})(CO)_6]$ or $[\{(\eta^5\text{-}C_5H_4)C(CH_2)_4(C_9H_7)\}Mo(CO)_3]_2$, is a binuclear metal carbonyl complex containing an Mo—Mo single bond [3.2405 (9) Å]. The cyclopentane ring adopts an envelope conformation. The dihedral angle between the indene and cyclopentadiene rings is 57.0 (2)°.

Comment

Reaction of the lithium salt $[C_5H_4\text{–}C(CH_3)_2\text{-}C_9H_7]^-\cdot Li^+$ with $(CH_3CN)_3M(CO)_3$ (M = Mo, W) affords $[\{C_5H_4\text{-}C(CH_3)_2\text{-}C_9H_7\}M(CO)_3]^-\cdot Li^+$, and subsequent reaction with MeI leads to the formation of a neutral complex $[\eta^5\text{-}C_5H_4\text{–}C(CH_3)_2\text{-}C_9H_7]M(CO)_3Me$ (Alt *et al.*, 1993). Reaction of the dilithium salt $[C_5H_4\text{-}C(CH_3)_2\text{-}C_9H_6]^{2-}\cdot Li^{2+}$ with $Mo(CO)_3(CH_3CN)_3$ and oxidation with acetic acid and Fe^{III} produced a dimer, $\{M(CO)_3[\eta^5\text{-}C_5H_4\text{-}C(CH_3)_2\text{-}C_9H_7]\}_2$, in which the indenyl ring was unmetallated (Fierro *et al.*, 1996). The title compound, $\{(\eta^5\text{-}C_5H_4)C(CH_2)_4(C_9H_7)Mo(CO)_3\}_2$, (I), was obtained by the reaction of $[C_5H_5\text{-}C(CH_2)_4\text{-}C_9H_7]$ with $Mo(CO)_3(CH_3CN)_3$ in xylene.

(I)

A view of (I), with the atomic numbering scheme, is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. Compound (I) is a centrosymmetric binuclear metal–carbonyl complex containing an Mo—Mo single bond. As shown in Fig. 1, only the cyclopentadienyl ring of the organic ligand coordinates with the Mo atom, and the indenyl group acts as a substituent. The Mo1$\cdots$$Cg$1 distance is 2.0113 (19) Å, where Cg1 is the centroid of the cyclopentadiene ring. The Cg1—Mo1—C1, Cg1—Mo1—C2 and Cg1—Mo1—C3 angles are 116.09 (13), 125.76 (13) and 127.68 (13)°, respectively. The Mo—Mo bond distance is 3.2405 (9) Å and agrees with that observed in the analogous structures $(\eta^5\text{-}C_5H_5)_2Mo_2(CO)_5(CNCH_3)$ [3.230 (1) Å; Adams *et al.*, 1973] and $(\eta^5\text{-}C_5H_5)_2Mo_2(CO)_6$ [3.235 (1) Å; Adams *et al.*, 1974].

The two cyclopentadienyl rings are parallel by virtue of the centre of symmetry. The cyclopentane ring in the ligand adopts an envelope conformation, with atom C9 as the flap. The dihedral angle between the indene and cyclopentadiene rings is 57.0 (2)°. The crystal packing is stabilized by weak C—H$\cdots$O interactions (Table 2).

metal-organic papers

Experimental

A solution of [C_5H_5–$C(CH_2)_4$-C_9H_7] (497 mg, 2 mmol) (Yan *et al.*, 1998) and $Mo(CO)_3(CH_3CN)_3$ [obtained by the reaction of $Mo(CO)_6$ (1000 mg, 3.79 mmol) with CH_3CN (15 ml) under refluxing for 4 h] (Tate *et al.*, 1962) in xylene (40 ml) was refluxed for 10 h. The solvent was removed under vacuum and the residue was chromatographed on an Al_2O_3 column using petroleum ether/CH_2Cl_2 (1:1) as eluent. The red band was collected and after several days red crystals were obtained (yield 0.06 g, 4%). Analysis calculated for $C_{44}H_{38}Mo_2O_6$: C 61.84, H 4.48%; found: C 61.78, H 4.65%.

Crystal data

[$Mo_2(C_{19}H_{19})(CO)_6$]	$Z = 2$
$M_r = 854.62$	$D_x = 1.556$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 19.038$ (4) Å	$\mu = 0.74$ mm^{-1}
$b = 7.7015$ (16) Å	$T = 293$ (2) K
$c = 13.091$ (3) Å	Plate, red
$\beta = 108.134$ (3)°	$0.30 \times 0.20 \times 0.08$ mm
$V = 1824.1$ (7) Å^3	

Data collection

Bruker SMART CCD 1000 area-detector diffractometer	9906 measured reflections
φ and ω scans	3693 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2385 reflections with $I > 2\sigma(I)$
$T_{min} = 0.691$, $T_{max} = 1.000$	$R_{int} = 0.056$
(expected range = 0.652–0.943)	$\theta_{max} = 26.3°$

Refinement

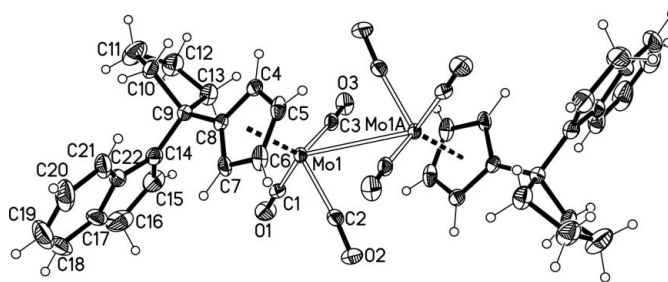|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0217P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.037$	$+ 1.246P]$
$wR(F^2) = 0.079$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.001$
3693 reflections	$\Delta\rho_{max} = 0.52$ e Å^{-3}
235 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}
H-atom parameters constrained	

Figure 1
The structure of (I), showing 30% probability displacement ellipsoids. Atoms labelled with the suffix A and other unlabelled atoms are generated by the symmetry operation ($-x$, $1 - y$, $-z$).

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C5—H5$\cdots$O3^i	0.98	2.40	3.334 (5)	160
C6—H6$\cdots$O1^{ii}	0.98	2.53	3.499 (6)	171

Symmetry codes: (i) $x, -y + \frac{3}{2}, z - \frac{1}{2}$; (ii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$.

H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

Table 1
Selected geometric parameters (Å, °).

Mo1—C1	1.958 (4)	Mo1—C5	2.367 (4)
Mo1—C2	1.977 (4)	Mo1—C6	2.329 (4)
Mo1—C3	1.985 (4)	Mo1—C7	2.306 (4)
Mo1—C4	2.353 (4)	Mo1—C8	2.359 (4)
C1—Mo1—C2	77.65 (16)	C6—Mo1—C4	58.00 (15)
C1—Mo1—C3	78.33 (16)	C1—Mo1—C8	89.62 (15)
C2—Mo1—C3	106.13 (17)	C2—Mo1—C8	139.49 (15)
C1—Mo1—C7	90.54 (16)	C3—Mo1—C8	108.65 (15)
C2—Mo1—C7	105.28 (16)	C7—Mo1—C8	35.72 (13)
C3—Mo1—C7	143.38 (15)	C6—Mo1—C8	58.99 (14)
C1—Mo1—C6	122.14 (16)	C4—Mo1—C8	35.14 (13)
C2—Mo1—C6	95.71 (16)	C1—Mo1—C5	146.51 (15)
C3—Mo1—C6	153.31 (17)	C2—Mo1—C5	119.02 (17)
C7—Mo1—C6	35.24 (14)	C3—Mo1—C5	118.95 (17)
C1—Mo1—C4	120.81 (15)	C7—Mo1—C5	58.06 (15)
C2—Mo1—C4	152.75 (17)	C6—Mo1—C5	34.59 (15)
C3—Mo1—C4	97.73 (16)	C4—Mo1—C5	34.72 (14)
C7—Mo1—C4	58.36 (14)	C8—Mo1—C5	58.44 (14)

References

Adams, R. D., Brice, M. & Cotton, F. A. (1973). *J. Am. Chem. Soc.* **95**, 6594–6602.

Adams, R. D., Collins, D. M. & Cotton, F. A. (1974). *Inorg. Chem.* **13**, 1086–1090.

Alt, H. G., Han, J. S. & Rogers, R. D. (1993). *J. Organomet. Chem.* **445**, 115–124.

Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (1999). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Fierro, R., Bitterwolf, T. E., Rheingold, A. L., Yap, G. P. A. & Liable-Sands, L. M. (1996). *J. Organomet. Chem.* **524**, 19–30.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997a). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Tate, D. P., Knipple, W. R. & Augl, J. M. (1962). *Inorg. Chem.* **1**, 433–434.

Yan, X., Chernega, A., Green, M. L. H., Sanders, J., Souter, J. & Ushioda, T. (1998). *J. Mol. Catal. A*, **128**, 119–141.