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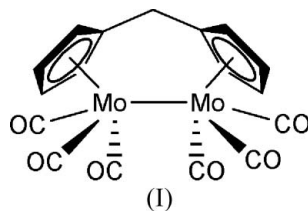
Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.032
 wR factor = 0.084
Data-to-parameter ratio = 13.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>. μ -(η^5, η^5 -Methylenedicyclopentadienyl)-
bis[tricarbonylmolybdenum(II)]The binuclear title complex, $\text{CH}_2[\text{C}_5\text{H}_4\text{Mo}^{\text{II}}(\text{CO})_3]_2$ or $[\text{Mo}_2(\text{C}_{11}\text{H}_{10})(\text{CO})_6]$, has an Mo—Mo single bond length of 3.1547 (6) Å. Both cyclopentadienyl rings coordinate the two Mo atoms and make a dihedral angle of 60.19 (16)°. The Mo—CO bond lengths range from 1.959 (4) to 1.996 (4) Å.

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Comment

Binuclear metal complexes have been intensively studied, largely because of their potential applications in catalysis (Marks, 1992). Several such complexes with the formula $\text{CH}_2[\text{C}_5\text{H}_4\text{M}(\text{CO})_3]_2$ ($M = \text{Cr}, \text{Mo}, \text{W}$), in which two cyclopentadienyl metal units are coupled by a methylene linkage, have been synthesized. Among these organometallic compounds, the chromium and tungsten derivatives have also been characterized by X-ray structural analysis (Bitterwolf & Rheingold, 1991; Brunner *et al.*, 1992). Here we describe the structure of the molybdenum analogue $\text{CH}_2[\text{C}_5\text{H}_4\text{Mo}(\text{CO})_3]_2$, (I).The X-ray structure analysis of (I) shows that each cyclopentadienyl (Cp) ring of the methylene-bridged bis-cyclopentadienyl ligand is η^5 -coordinated to one Mo atom (Fig. 1). Selected bond lengths and angles are given in Table 1. Both Cp rings are coordinated to the Mo atoms, forming a dihedral angle of 60.19 (16)°. In the crystal structure, there are intermolecular C—H...O hydrogen bonds (Fig. 2 and Table 2).

Experimental

All reactions were carried out under an inert atmosphere. THF was distilled from Na/benzophenone. Other solvents were of reagent grade and used without further purification. The starting material $\text{CH}_2(\text{C}_5\text{H}_4\text{Na})_2$ was prepared according to the literature (Bitterwolf & Rheingold, 1991). To a solution of $\text{Mo}(\text{CO})_3(\text{MeCN})_3$ (10.0 mmol) in THF (10 ml) was added dropwise a solution of $\text{CH}_2(\text{C}_5\text{H}_4\text{Na})_2$ (5.0 mmol) in THF (15 ml). The resulting mixture was refluxed for 3 h, and then cooled to 298 K. The solvent was removed under reduced pressure and the residue dissolved in CH_2Cl_2 (15 ml) to give a solution to which $\text{Fe}_2(\text{SO}_4)_3$ (3.5 mmol) and acetic acid (1.0 ml) in water (15 ml) were added slowly. The organic layer was separated and dried over anhydrous MgSO_4 . After removal of the solvent, the crude

compound was purified by column chromatography (Al_2O_3 , CHCl_3) to afford a red solid (26% yield). Crystals suitable for X-ray diffraction were obtained from a mixture of CH_2Cl_2 and *n*-hexane (1:1) at 298 K.

Crystal data

$[\text{Mo}_2(\text{C}_{11}\text{H}_{10})(\text{CO})_6]$	$V = 842.9 (2) \text{ \AA}^3$
$M_r = 502.13$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.978 \text{ Mg m}^{-3}$
$a = 7.6725 (13) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 7.7312 (13) \text{ \AA}$	$\mu = 1.52 \text{ mm}^{-1}$
$c = 15.520 (3) \text{ \AA}$	$T = 298 (2) \text{ K}$
$\alpha = 85.996 (2)^\circ$	Block, red
$\beta = 81.517 (2)^\circ$	$0.35 \times 0.17 \times 0.12 \text{ mm}$
$\gamma = 67.804 (2)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	4449 measured reflections
φ and ω scans	3081 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 1999)	2730 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.619$, $T_{\max} = 0.839$	$R_{\text{int}} = 0.023$
	$\theta_{\max} = 25.5^\circ$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.048P)^2]$
$wR(F^2) = 0.084$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{\max} = 0.001$
3081 reflections	$\Delta\rho_{\max} = 0.40 \text{ e \AA}^{-3}$
226 parameters	$\Delta\rho_{\min} = -0.74 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Mo1—C12	1.962 (4)	Mo2—C17	1.959 (4)
Mo1—C13	1.976 (3)	Mo2—C15	1.985 (4)
Mo1—C14	1.980 (4)	Mo2—C16	1.996 (4)
Mo1—C9	2.318 (4)	Mo2—C1	2.314 (4)
Mo1—C10	2.328 (4)	Mo2—C5	2.337 (4)
Mo1—C11	2.341 (4)	Mo2—C4	2.348 (3)
Mo1—C8	2.375 (3)	Mo2—C2	2.364 (4)
Mo1—C7	2.398 (3)	Mo2—C3	2.367 (4)
Mo1—Mo2	3.1547 (6)		
C12—Mo1—C13	79.05 (14)	C10—Mo1—C8	57.78 (13)
C12—Mo1—C14	76.58 (15)	C12—Mo1—C7	143.24 (14)
C12—Mo1—C9	86.58 (15)	C14—Mo1—C7	109.00 (13)
C13—Mo1—C9	130.79 (15)	C10—Mo1—C7	58.33 (13)
C14—Mo1—C10	151.17 (15)	C8—Mo1—C7	34.30 (12)
C9—Mo1—C10	35.10 (15)	C4—Mo2—C2	58.20 (14)
C14—Mo1—C11	143.58 (14)	C17—Mo2—C3	139.35 (16)
C9—Mo1—C11	58.10 (14)	C15—Mo2—C3	132.17 (15)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1 \cdots O2 ⁱ	0.98	2.60	3.330 (5)	132
C10—H10 \cdots O6 ⁱⁱ	0.98	2.52	3.369 (5)	144

Symmetry codes: (i) $-x, -y + 2, -z + 1$; (ii) $-x, -y + 2, -z$.

All H atoms were included in calculated positions and refined as riding [C—H = 0.98 (aromatic CH) and 0.97 \AA (methylene CH_2) and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$].

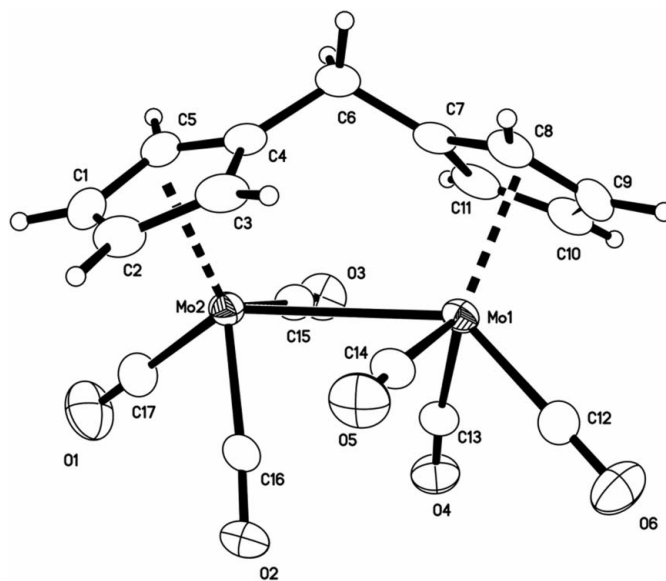


Figure 1

The molecular structure of (I), showing the atom labelling. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

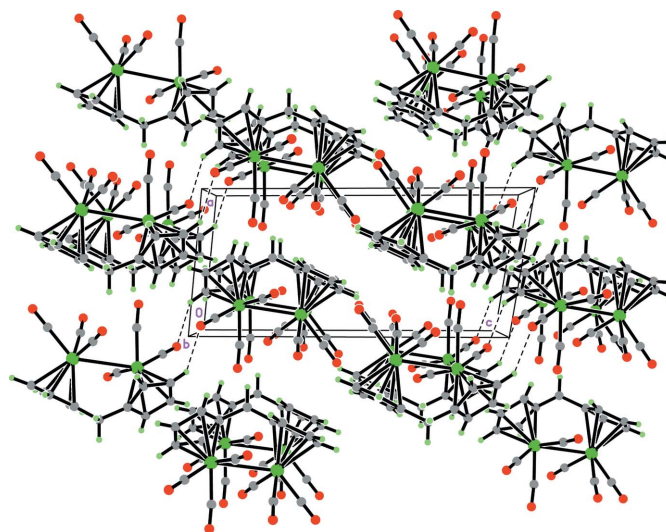


Figure 2

View of the packing of (I), showing the C—H \cdots O contacts (dashed lines).

Data collection: SMART (Bruker, 1999); cell refinement: SMART; data reduction: SAINT (Bruker, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2001); software used to prepare material for publication: SHELXTL.

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