

# Tricarbonyl(methyl)( $\eta^5$ -1,2,4-tricyclohexylcyclopentadienyl)molybdenum

Helmut Sitzmann,  
Marc D. Walter and  
Gotthelf Wolmershäuser\*

Fachbereich Chemie, Universität Kaiserslautern,  
Erwin-Schrödinger-Straße, 67663 Kaiserslautern,  
Germany

Correspondence e-mail:  
wolmersh@chemie.uni-kl.de

## Key indicators

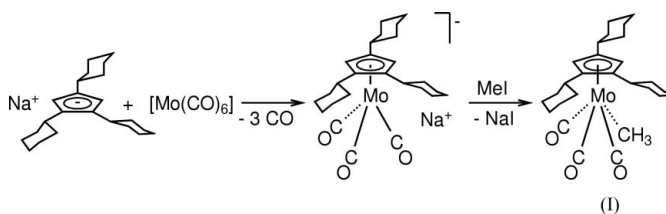
Single-crystal X-ray study  
 $T = 293\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$   
 $R$  factor = 0.034  
 $wR$  factor = 0.062  
Data-to-parameter ratio = 16.0

For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The title compound,  $[\text{Mo}(\text{CH}_3)(\text{C}_{23}\text{H}_{35})(\text{CO})_3]$ , has been synthesized and characterized by NMR spectroscopy, elemental analysis, and by an X-ray crystal structure investigation.

## Comment

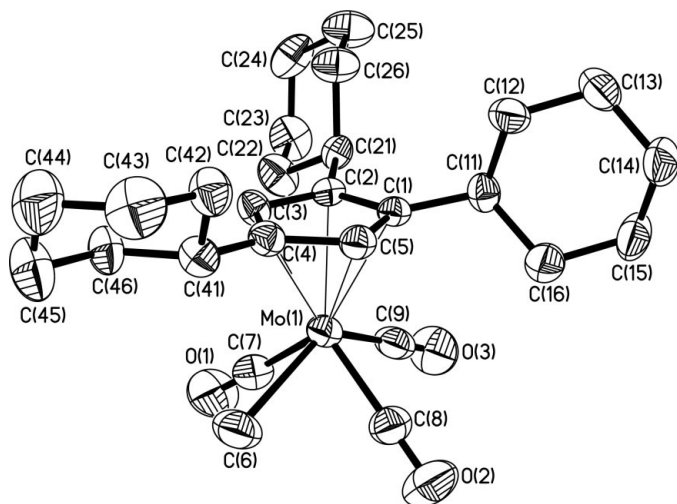
The title compound, (I), is the first known half-sandwich complex of the 1,2,4-tricyclohexylcyclopentadienyl ligand. The perpendicular distance of Mo from the five-membered ring plane [2.0206 (15) Å] and all Mo—C distances are comparable to the values found for other tricarbonyl(cyclopentadienyl)-methylmolybdenum derivatives (El Mouatassim *et al.*, 1995; Lai *et al.*, 1993; Bell *et al.*, 1988). All cyclohexyl (Cy) substituents exhibit a chair conformation. The angles between the cyclopentadienyl ring plane and one plane for each substituent, defined by the cyclopentadienyl ring atom connected to the *ipso*-C atom and the two nearest methylene C atoms, are 83.17 (13)° for 1-Cy and 68.27 (14)° for 2-Cy. With these two substituents rotated away from each other, a maximum of space is provided for carbonyl C9—O3. The Cy substituent in the 4-position adopts a dihedral angle of 41.91 (18)° with the Cp ring plane, avoiding any close contact with the methyl group bonded to Mo. The absence of steric strain has also been noted for hexa(cyclohexyl)ferrocene, the only other known complex of the 1,2,4-tricyclohexylcyclopentadienyl ligand (Burman *et al.*, 1994).



## Experimental

A mixture of lithium tricyclohexylcyclopentadienide (637 mg, 2.0 mmol), molybdenum hexacarbonyl (539 mg, 2.0 mmol), and tetrahydrofuran (50 ml) was stirred at reflux temperature for 5 h and allowed to cool to ambient temperature. Methyl iodide (281 mg, 2.0 mmol) was added and the mixture was heated to reflux temperature for 2 h. The solvent was evaporated and the residue extracted with toluene (30 ml). Evaporation of the filtered toluene solution and recrystallization from hexane–tetrahydrofuran (5:1) at ambient temperature yielded yellow crystalline (I) (712 mg, 1.41 mmol, 70%).  $\text{C}_{27}\text{H}_{38}\text{MoO}_3$  ( $M_r = 506.54$ ), calculated: C 64.02, H 7.56%; found: C 63.31, H 7.64%.  $^1\text{H NMR}$  ( $\text{C}_6\text{D}_6$ , 400.13 MHz): 4.75

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**Figure 1**  
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

(*s*, 2H, ring H), 2.11–0.93 (*m*, cyclohexyl protons), 0.45 p.p.m. (*s*, 3H, Mo–methyl).  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 100.62 MHz): 243.2 (1C, carbonyl), 229.1 (2C, carbonyl), 120.9 (*s*, 1C, ring C), 117.3 (*s*, 2C, ring C), 86.6 (*dd*, 2 C,  $J = 163 \text{ Hz}/5 \text{ Hz}$ , ring CH), 37.7 (2C, methylene), 36.4 (1C, *ipso*-CH), 35.7 (2C, *ipso*-CH), 35.2 (2C, cyclohexyl), 34.4 (2C, cyclohexyl), 27.1 (2C, cyclohexyl), 27.0 (2C, cyclohexyl), 26.5 (2C, cyclohexyl), 26.4 (1C, cyclohexyl), 26.2 (2C, cyclohexyl), –14.3 p.p.m. (*q*, 1C,  $J = 136 \text{ Hz}$ , Mo–methyl). C,H coupling constants of cyclohexyl carbon signals could not be determined, because of signal superimposition.

#### Crystal data

$[\text{Mo}(\text{CH}_3)(\text{C}_{23}\text{H}_{35})(\text{CO})_3]$   
 $M_r = 506.51$   
 Monoclinic,  $P2_1/n$   
 $a = 10.8664$  (8) Å  
 $b = 9.8320$  (5) Å  
 $c = 23.3279$  (17) Å  
 $\beta = 95.838$  (9)°  
 $V = 2479.4$  (3) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.357 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 1480 reflections  
 $\theta = 2\text{--}25^\circ$   
 $\mu = 0.55 \text{ mm}^{-1}$   
 $T = 293$  (2) K  
 Transparent block, colourless  
 $0.25 \times 0.13 \times 0.08 \text{ mm}$

#### Data collection

Stoe IPDS diffractometer  
 $\varphi$  scans  
 Absorption correction: analytical  
 (*ABST* in *PLATON*; Spek, 2000)  
 $T_{\min} = 0.922$ ,  $T_{\max} = 0.979$   
 18023 measured reflections  
 4495 independent reflections

2569 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.097$   
 $\theta_{\max} = 25.4^\circ$   
 $h = -13 \rightarrow 13$   
 $k = -11 \rightarrow 11$   
 $l = -28 \rightarrow 28$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.062$   
 $S = 0.80$   
 4495 reflections  
 281 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.001P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.47 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.28 \text{ e \AA}^{-3}$

H atoms were placed geometrically and refined with a riding model.

Data collection: *EXPOSE* in *IPDS* (Stoe & Cie, 1997); cell refinement: *CELL* in *IPDS*; data reduction: *INTEGRATE* in *IPDS*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1998); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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