

# Tricarbonylido( $\eta^5$ -methylcyclopentadienyl)-tungsten

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## Key indicators

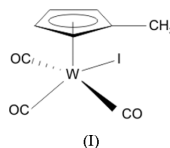
Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å  
 $R$  factor = 0.020  
 $wR$  factor = 0.047  
Data-to-parameter ratio = 19.8

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

The title complex,  $[\text{W}(\eta^5\text{-C}_5\text{H}_5\text{CH}_3)\text{I}(\text{CO})_3]$ , lies on a crystallographic mirror plane, with only one half-molecule per asymmetric unit. This four-legged piano-stool tungsten complex is strained at the W atom, with the *trans*-C–W–I angle  $[130.93(18)^\circ]$  larger than the *cis*-C–W–C angle  $[111.1(3)^\circ]$ . A possible *trans* influence due to a larger  $\pi$  back-bonding is marginal, with an insignificant difference between the W–C bond length *trans* to I  $[1.984(6)$  Å] and the other W–C<sub>CO</sub> bond lengths  $[1.996(5)$  Å].

## Comment

The title compound, (I), was prepared as part of our ongoing interest in the coordination and solid-state reactivity of distorted square-pyramidal complexes with a *cyclopentadienyl* group sitting at the apex position (Smith *et al.*, 1996). In an earlier study on a related complex, *viz.*  $(\eta^5\text{-C}_5\text{H}_5)\text{W}(\text{CO})_3\text{Cl}$  (Buono & Churchill, 1981), no clear evidence for a *trans* influence by Cl on the W–C<sub>CO</sub> bond length was observed. In this study, a marginal *trans* effect due to an increased  $\pi$  back-bonding of the carbonyl ligand *trans* to I was detected, with the W–C<sub>CO</sub> bond length *trans* to I being  $1.984(6)$  Å and the W–C<sub>CO</sub> bond length *trans* to CO being  $1.996(5)$  Å. Interatomic angles and distances agree with those reported for similar compounds (Töfke & Behrens, 1987). A mirror plane runs through the molecule, with the iodide ligand eclipsed by the methyl ring substituent, giving an I–W–C4–C9 torsion angle of  $0^\circ$ .



## Experimental

The title complex was prepared by a modification of a general procedure (Piper & Wilkinson, 1956). A dimethoxyethane suspension of  $\text{W}(\text{CO})_6$  and  $\text{C}_5\text{H}_5\text{Me}$  was refluxed for 24 h, cooled to room temperature and iodine was added to the solution with continued stirring. After 3 h, the solvent was removed *in vacuo*. Extraction with hexane afforded the title compound as orange needles. X-ray quality crystals were obtained at room temperature by crystallization from pentane. IR ( $\text{CH}_2\text{Cl}_2$ ,  $\text{cm}^{-1}$ )  $\nu_{\text{CO}}$ : 2031 (s), 1951 (s).

## Crystal data

$[\text{W}(\text{C}_6\text{H}_8)\text{I}(\text{CO})_3]$   
 $M_r = 473.9$   
Monoclinic,  $C2/m$   
 $a = 13.5020(16)$  Å  
 $b = 8.4577(10)$  Å  
 $c = 10.1886(12)$  Å  
 $\beta = 104.949(2)^\circ$   
 $V = 1124.1(2)$  Å<sup>3</sup>  
 $Z = 4$

$D_x = 2.8$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 939  
reflections  
 $\theta = 2.9\text{--}28.3^\circ$   
 $\mu = 13.00$  mm<sup>-1</sup>  
 $T = 293(2)$  K  
Needle, orange  
 $0.35 \times 0.12 \times 0.10$  mm

Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 1999)  
 $T_{\min} = 0.165$ ,  $T_{\max} = 0.273$   
 3934 measured reflections

1485 independent reflections  
 1363 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$   
 $\theta_{\max} = 28.3^\circ$   
 $h = -12 \rightarrow 17$   
 $k = -11 \rightarrow 11$   
 $l = -13 \rightarrow 11$

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.020$   
 $wR(F^2) = 0.047$   
 $S = 1.06$   
 1485 reflections  
 75 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.021P)^2 + 2.5564P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.80 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.57 \text{ e } \text{\AA}^{-3}$   
 Extinction correction: SHELXL97  
 Extinction coefficient: 0.00169 (9)

Table 1

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

W1–C1	1.984 (6)	W1–I2	2.8460 (5)
W1–C2	1.996 (5)	C4–C9	1.504 (9)
C2 <sup>i</sup> –W1–C2	111.1 (3)	C1–W1–I2	130.93 (18)

Symmetry code: (i)  $x, 1 - y, z$ .

H atoms attached to aromatic C atoms were positioned geometrically and allowed to ride on parents during refinement, with C–H = 0.93  $\text{\AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$ . The disordered methyl H atoms were accounted for using a two-site model, in which the H atoms were placed geometrically and allowed to ride, with C–H = 0.96  $\text{\AA}$  and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{parent atom})$ .

Data collection: SMART-NT (Bruker, 1998); cell refinement: SAINT-Plus (Bruker, 1999); data reduction: SAINT-Plus and XPREP (Bruker, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure:

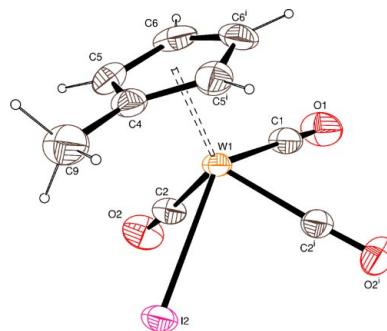


Figure 1

View of (I) (50% probability displacement ellipsoids); the symmetry code is as in Table 1.

SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON (Spek, 2003).

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