

$\text{W}_2\text{Ir}_2(\text{CO})_{10}(\eta^5\text{-C}_5\text{H}_4\text{Me})_2$

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Key indicators

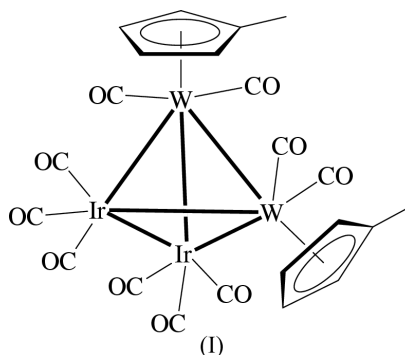
Single-crystal X-ray study
 $T = 200\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.013\text{ \AA}$
 R factor = 0.042
 wR factor = 0.054
Data-to-parameter ratio = 19.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, decacarbonyl- $1\kappa^2\text{C},2\kappa^2\text{C},3\kappa^3\text{C},4\kappa^3\text{C}$ -bis-[1,2(η^5)-methylcyclopentadienyl]-tetrahedro-ditungstendiiridium, $[\text{W}_2\text{Ir}_2(\text{C}_6\text{H}_7)_2(\text{CO})_{10}]$, is a mixed-metal cluster with tetrahedral metal-core geometry. The W atoms are each ligated by a η^5 -methylcyclopentadienyl ligand and two carbonyl ligands, and the Ir atoms are each ligated by three carbonyl ligands.

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Comment

Mixed-metal clusters containing very different metals have attracted significant recent interest, with tungsten–iridium clusters the focus of a considerable number of studies (Waterman *et al.*, 2000). A precursor to much of the existing tungsten–iridium cluster chemistry is the cyclopentadienyl-containing cluster $\text{W}_2\text{Ir}_2(\text{CO})_{10}(\eta^5\text{-C}_5\text{H}_5)_2$ (Shapley *et al.*, 1981). The methylcyclopentadienyl-containing clusters can be prepared analogously, as detailed below. This structural study, which reveals two independent molecules in the asymmetric unit, shows that the metal–metal core bond distances in the title complex, (I), are essentially identical to those of the cyclopentadienyl-containing analogue (Churchill *et al.*, 1982).



Experimental

$\text{Na}[\text{W}(\text{CO})_3(\eta^5\text{-C}_5\text{H}_4\text{Me})]$ was prepared from Na (46.8 mg, 2.03 mmol), methylcyclopentadiene (410 mg, 5.11 mmol) and $\text{W}(\text{CO})_6$ (543 mg, 5.10 mmol). $\text{IrCl}(\text{CO})_2(p\text{-toluidine})$ (598 mg, 1.53 mmol) was added to the crude solid $\text{Na}[\text{W}(\text{CO})_3(\eta^5\text{-C}_5\text{H}_4\text{Me})]$ in CH_2Cl_2 (20 ml), and the mixture stirred at room temperature for 2 h. The solvent was removed *in vacuo*, and the resulting red–brown residue dissolved in CH_2Cl_2 (*ca* 3 ml) then applied to preparative thin-layer chromatography (TLC) plates. Elution with CH_2Cl_2 /petroleum spirit (2/3) gave two bands. Crystallization of the contents of the second and major band ($R_F = 0.52$) from CH_2Cl_2 /methanol by liquid diffusion at 276 K over 24 h afforded a red–brown crystalline product identified as $\text{W}_2\text{Ir}_2(\text{CO})_{10}(\eta^5\text{-C}_5\text{H}_4\text{Me})_2$ (733 mg, 81%). Analysis calculated for $\text{C}_{22}\text{H}_{14}\text{Ir}_2\text{O}_{10}\text{W}_2$: C 22.20, H 1.19%; found: C

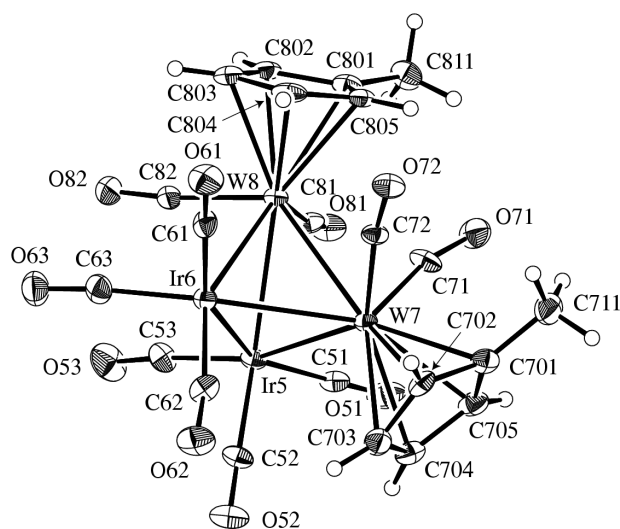
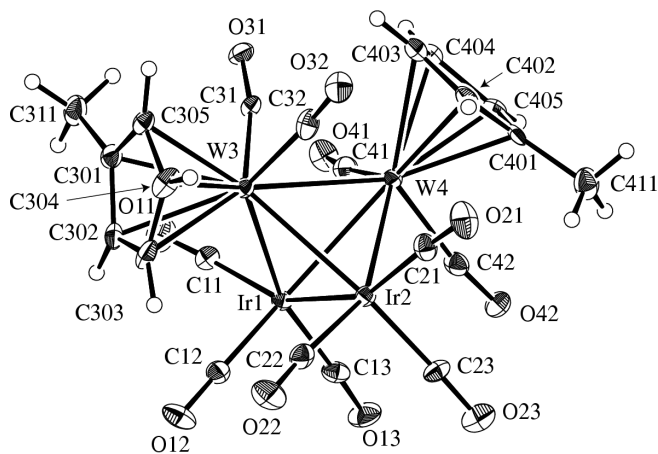


Figure 1

A view of both molecules in the asymmetric unit of the title compound with the atom-numbering scheme. Displacement ellipsoids are shown at the 30% probability level.

22.56 H 1.27%. MS (secondary ion, Cs⁺): [M - nCO]⁺, n = 2–10. IR (cyclohexane): $\nu(\text{CO})$ 2063 (*m*), 2028 (*s*), 2006 (*m*), 1995 (*m*), 1982 (*m*), 1965 (*w*), 1948 (*w*), 1921 (*w*), 1890 (*w*), 1827 (*w*) cm⁻¹. ¹H NMR: δ 5.25 (*t*, *J*_{HH} = 2 Hz, 4H, C₅H₄Me), 4.94 (*t*, *J*_{HH} = 2 Hz, 4H, C₅H₄Me), 2.18 (*s*, 6H, Me) p.p.m.

Crystal data

[W₂Ir₂(C₆H₇)₂(CO)₁₀]
M_r = 1190.49
 Monoclinic, *P*₂₁/*n*
a = 18.3035 (1) Å
b = 8.5629 (1) Å
c = 31.2876 (2) Å
 β = 94.7702 (4)°
V = 4886.75 (6) Å³
Z = 8

D_x = 3.236 Mg m⁻³
 Mo *K* α radiation
 Cell parameters from 267775 reflections
 θ = 2.9–30.0°
 μ = 20.34 mm⁻¹
T = 200 K
 Needle, red–brown
 0.40 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: by integration Gaussian (Coppens, 1970) implemented in *maXus* (Mackay *et al.*, 1999)
*T*_{min} = 0.029, *T*_{max} = 0.193
 129 999 measured reflections

14 253 independent reflections
 12 656 reflections with *I* > 2 σ (*I*)
*R*_{int} = 0.077
 θ _{max} = 30.1°
h = –25 → 25
k = –12 → 10
l = –44 → 44

Refinement

Refinement on *F*²
R = 0.042
wR = 0.054
S = 1.81
 12656 reflections
 649 parameters

H-atom parameters not refined
 $w = 1/[\sigma^2(F_o) + 0.0004|F_o|^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.032$
 $\Delta\rho_{\text{max}} = 3.35 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -2.86 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å).

Ir1–Ir2	2.7242 (4)	Ir5–W7	2.8021 (5)
Ir1–W3	2.8032 (4)	Ir5–W8	2.8596 (4)
Ir1–W4	2.8577 (4)	Ir6–W7	2.8363 (5)
Ir2–W3	2.8330 (4)	Ir6–W8	2.8482 (4)
Ir2–W4	2.8476 (4)	W3–W4	2.9891 (4)
Ir5–Ir6	2.7271 (4)	W7–W8	3.0108 (4)

Data collection: *KappaCCD Software* (Nonius, 1999); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *PATY* in *DIRDIF92* (Beurskens *et al.*, 1992); program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1997); software used to prepare material for publication: *TEXSAN*.

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