Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# $W_2Ir_2(\mu_4\text{-}\eta^2\text{-}C_2Ph_2)(\mu\text{-}CO)_4(CO)_4(\eta\text{-}C_5H_4Me)_2$

**Nigel T. Lucas, Eleni G. A. Notaras and Mark G. Humphrey***

Department of Chemistry, Australian National University, Canberra ACT 0200, Australia

Correspondence e-mail: mark.humphrey@anu.edu.au

**Key indicators**

Single-crystal X-ray study
$T$ = 200 K
Mean $\sigma$(C–C) = 0.008 Å
$R$ factor = 0.026
$wR$ factor = 0.030
Data-to-parameter ratio = 17.9

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

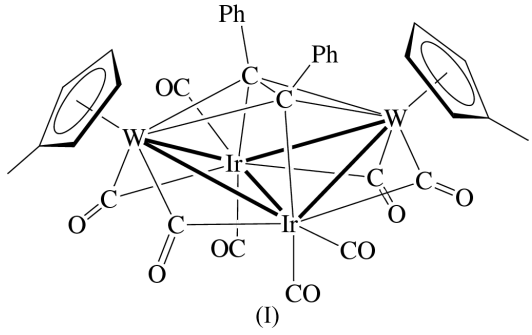


Received 21 February 2001
Accepted 6 March 2001
Online 9 March 2001

The title compound, octacarbonyl-$\mu$-diphenylacetylene-bis($\eta^5$-methylcyclopentadienyl)diiridiumditungsten, $W_2Ir_2(\mu_4\text{-}\eta^2\text{-}C_2Ph_2)(\mu\text{-}CO)_4(CO)_4(\eta\text{-}C_5H_4Me)_2$ or $[W_2Ir_2(C_6H_7)_2(C_{14}H_{10})(CO)_8]$, is a mixed-metal cluster with a butterfly metal core geometry, W atoms at the wing-tip sites and Ir atoms at the hinge positions. A diphenylacetylene ligand is $\mu_4$-$\eta^2$-coordinated with the C—C bond parallel to the Ir—Ir vector, completing a pseudo-octahedral $W_2Ir_2C_2$ unit. The W atoms are each ligated by a methylcyclopentadienyl ligand, and the Ir atoms are each ligated by two terminal carbonyls, with the coordination completed by four bridging carbonyls, each of which lies across a W—Ir linkage.

## Comment

The chemistry of the cyclopentadienyl-ligated tetrahedral mixed-metal cluster $W_2Ir_2(CO)_{10}(\eta^5\text{-}C_5H_5)_2$ with alkynes has been examined in order to enhance understanding of how alumina-supported bimetallic W—Ir particles behave; in particular, diphenylacetylene reacts with this ditungstendiiridium cluster to afford $W_2Ir_2(\mu_4\text{-}\eta^2\text{-}C_2Ph_2)(CO)_8(\eta\text{-}C_5H_5)_2$ (Shapley *et al.*, 1984). We have now investigated the reaction of the methylcyclopentadienyl-ligated cluster analogue $W_2Ir_2(CO)_{10}(\eta^5\text{-}C_5H_4Me)_2$ (Lucas *et al.*, 2001) with diphenylacetylene, and structurally characterized the product, (I). The alkyne formally inserts into the W—W bond of the precursor tetrahedral $W_2Ir_2$ cluster to afford a *closo*-octahedral $W_2Ir_2C_2$ cluster. The $W_2Ir_2C_2$ core bond distances in the title complex are essentially identical to those of the phenylacetylene-containing analogue $W_2Ir_2(\mu_4\text{-}\eta^2\text{-}PhC_2H)(\mu\text{-}CO)_4(CO)_4(\eta\text{-}C_5H_5)_2$ (Waterman *et al.*, 1998).




(I)

## Experimental

Diphenylacetylene (19.5 mg, 0.110 mmol) was added to a red-brown solution of $W_2Ir_2(CO)_{10}(\eta^5\text{-}C_5H_4Me)_2$ (26.6 mg, 0.023 mmol) in $CH_2Cl_2$ (20 ml) and the resultant mixture stirred at reflux for 5.5 h. The resulting dark green solution was taken to dryness on a rotary

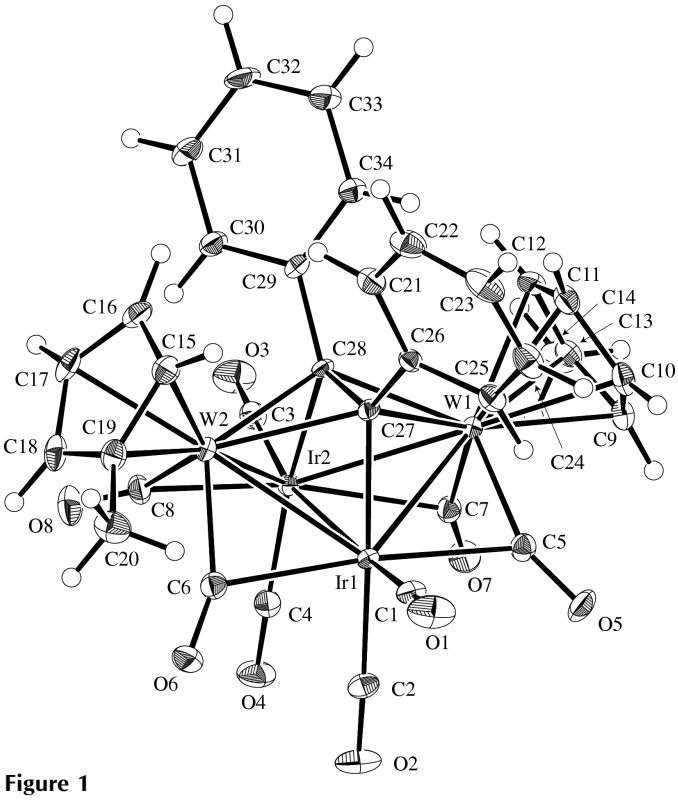



**Figure 1**
A view of the title compound with the atom-numbering scheme. Displacement ellipsoids are shown at the 30% probability level.

evaporator, and the residue dissolved in a minimum of $CH_2Cl_2$ (*ca* 3 ml) and applied to preparative TLC plates. Elution with $CH_2Cl_2$/petroleum spirit (3/2) gave two bands. The first band ($R_F$ = 0.76) was identified as unreacted starting cluster. The contents of the second and major band ($R_F$ = 0.42) were crystallized from $CH_2Cl_2$/methanol at 276 K to afford dark green crystals identified as $W_2Ir_2(\mu_4\text{-}\eta^2\text{-}C_2Ph_2)(\mu\text{-}CO)_4(CO)_4(\eta\text{-}C_5H_4Me)_2$ (20.1 mg, 70%). Analysis calculated for $C_{34}H_{24}Ir_2O_8W_2$: C 31.11 H 1.84%. Found: C 30.98 H 1.85%. MS (secondary ion, $Cs^+$): $[M - nCO]^+$, $n$ = 0–8. IR ($CH_2Cl_2$): $\nu$(CO) 2063 (*s*), 2035 (*s*), 2009 (*w*), 1990 (*w*), 1812 (*m*), 1755 (*m*) $cm^{-1}$. $^1H$ NMR: $\delta$ 7.22–7.02 (*m*, 10H, Ph), 4.58 (*s*, 4H, $C_5H_4Me$), 4.44 (*s*, 4H, $C_5H_4Me$), 2.13 (*s*, 6H, Me) p.p.m..

*Crystal data*

$[W_2Ir_2(C_6H_7)_2(C_{14}H_{10})(CO)_8]$
$M_r$ = 1312.70
Monoclinic, $P2_1/c$
$a$ = 16.8073 (1) Å
$b$ = 9.5849 (1) Å
$c$ = 19.0626 (2) Å
$\beta$ = 90.8932 (6)°
$V$ = 3070.54 (4) Å$^3$
$Z$ = 4
$D_x$ = 2.839 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 78199 reflections
$\theta$ = 3.4–30.0°
$\mu$ = 16.20 mm$^{-1}$
$T$ = 200 K
Block, dark green
0.30 × 0.25 × 0.10 mm

*Data collection*

Nonius KappaCCD diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: by integration [Gaussian (Coppens, 1970) implemented in *maXus* (Mackay *et al.*, 1999)]
$T_{min}$ = 0.050, $T_{max}$ = 0.327
90 662 measured reflections
8980 independent reflections
7429 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.077
$\theta_{max}$ = 30.0°
$h = -23 \rightarrow 23$
$k = -13 \rightarrow 13$
$l = -26 \rightarrow 26$

*Refinement*

Refinement on $F$
$R$ = 0.026
$wR$ = 0.030
$S$ = 0.82
7429 reflections
415 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.0004|F_o|^2]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.36 e Å$^{-3}$
$\Delta\rho_{min}$ = −2.33 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °) for (I).

| | | | |
|---|---|---|---|
| Ir1—Ir2 | 2.6951 (2) | Ir2—C28 | 2.138 (4) |
| Ir1—W1 | 2.8145 (2) | W1—C27 | 2.331 (4) |
| Ir1—W2 | 2.7804 (2) | W1—C28 | 2.353 (4) |
| Ir1—C27 | 2.125 (4) | W2—C(27 | 2.369 (4) |
| Ir2—W1 | 2.8018 (2) | W2—C28 | 2.345 (4) |
| Ir2—W2 | 2.8109 (3) | C27—C28 | 1.487 (6) |
| | | | |
| Ir2—Ir1—C27 | 73.9 (1) | Ir1—C27—C28 | 106.3 (3) |
| Ir1—Ir2—C28 | 73.2 (1) | Ir2—C28—C27 | 106.6 (3) |

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: *PATTY* in *DIRDIF*92 (Beurskens *et al.*, 1992); program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1997); software used to prepare material for publication: *TEXSAN*.

We thank the Australian Research Council (ARC) for financial support in the form of a Large Grant and the funds to purchase the diffractometer, and Johnson–Matthey Technology Centre for the generous loan of iridium salts. NTL was an Australian Postgraduate Awardee and MGH holds an ARC Australian Senior Research Fellowship.

## References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.
Coppens, P. (1970). *The Evaluation of Absorption and Extinction in Single-Crystal Structure Analysis*. *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 255–270. Copenhagen: Munksgaard.
Lucas, N. T., Notaras, E. G. A. & Humphrey, M. G. (2001). *Acta Cryst.* E**57**, m132–133.
Mackay, S., Gilmore, C. J., Edwards, C., Stewart, N. & Shankland, K. (1999). *maXus*. *Computer Program for the Solution and Refinement of Crystal Structures*. Nonius, The Netherlands, MacScience, Japan, and The University of Glasgow, Scotland.
Molecular Structure Corporation (1997). *TEXSAN*. Version 1.7. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Nonius (1999). *KappaCCD Software*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
Shapley, J. R., McAteer, C. H., Churchill, M. R. & Biondi, L. V. (1984). *Organometallics*, **3**, 1595–1596.
Waterman, S. M., Humphrey, M. G., Tolhurst, V.-A., Bruce, M. I., Low, P. J. & Hockless, D. C. R. (1998). *Organometallics*, **17**, 5789–5795.