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Heptacarbonylbis(μ_3-η^2-diphenylacetylene)-(η^5-pentamethylcyclopentadienyl)triiridiumtungsten

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Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.012 Å
R factor = 0.022
wR factor = 0.025
Data-to-parameter ratio = 10.2

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

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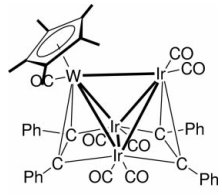
The title compound, [$WIr_3(\eta$-$C_{10}H_{15})(\mu_3$-η^2-$C_{14}H_{10})_2(CO)_7$], is a mixed-metal cluster with a tetrahedral metal core geometry. One diphenylacetylene ligand is μ_3-η^2-coordinated to the triiridium face, while the other is μ_3-η^2-coordinated to a diiridiumtungsten face. The W atom is ligated by a pentamethylcyclopentadienyl ligand and a terminal carbonyl, and the Ir atoms are each ligated by two terminal carbonyls.

Comment

The chemistry of the cyclopentadienyl-ligated tetrahedral mixed-metal cluster $WIr_3(CO)_{11}(\eta$-$C_5H_5)$ with diphenylacetylene has been examined in order to enhance understanding of how alumina-supported bimetallic W—Ir particles behave (Shapley *et al.*, 1993). We have now examined the reaction of the pentamethylcyclopentadienyl-ligated cluster $WIr_3(CO)_{11}(\eta$-$C_5Me_5)$ (Usher *et al.*, 2004) with diphenylacetylene, and have structurally characterized the product, (I).



(I)

In (I), the tetrahedral core geometry of the precursor is preserved, one alkyne bridges the triiridium face, and the other alkyne bridges a diiridiumtungsten face. Comparison of core bond distances with those of the cyclopentadienyl-containing analogue $WIr_3(\mu_3$-η^2-$C_2Ph_2)_2(CO)_7(\eta$-$C_5Me_5)$ (Shapley *et al.*, 1993) reveals a *ca* 0.03 Å lengthening of core distances involving tungsten; all other core distances are essentially identical.

Experimental

Diphenylacetylene (19.5 mg, 0.109 mmol) was added to an orange solution of $WIr_3(CO)_{11}(\eta$-$C_5Me_5)$ (20.5 mg, 0.0170 mmol) in toluene (25 ml) and the mixture was heated at reflux for 35 min. The resulting red solution was taken to dryness on a rotary evaporator, and the residue dissolved in a minimum of CH_2Cl_2 (*ca* 3 ml) and applied to preparative thin-layer chromatography plates. Elution with CH_2Cl_2/petrol (3/7) gave five bands: band 1 was yellow (R_F = 0.66); band 2 was purple (R_F = 0.51); band 3 was yellow (R_F = 0.46); band 4 was purple (R_F = 0.42); band 5 was purple (R_F = 0.23). Bands 3, 4 and 5 were in trace amounts and could not be identified. The contents of band 1 were identified as $Ir_2\{\mu$-η^4-C(Ph)C(Ph)C(Ph)C(Ph)$\}(CO)_5$ (2.5 mg, 0.0290 mmol, 17%) by comparison with literature IR and MS data (Notaras *et al.*, 2001). The contents of band 2 were identified as

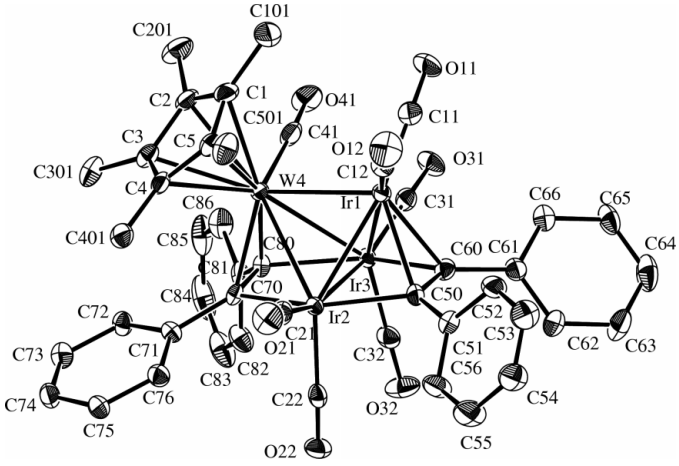


Figure 1
A view of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

$WIr_3(\mu_3\text{-}\eta^2\text{-}C_2Ph_2)_2(CO)_7(\eta\text{-}C_5Me_5)$ (16 mg, 0.011 mmol, 65%). Analysis calculated for $C_{45}H_{35}Ir_3O_7W$: C 37.32, H 2.44%; found: C 37.17, H 2.40%. MS (secondary ion, Cs^+): $1448 - 28n$ ($[M - n\mathrm{CO}]^+$, n = 0–7. IR (c–C_6H_{12}): ν(CO) 2049 (*s*), 2022 (*vs*), 2012 (*vs*), 1995 (*w*), 1973 (*s*), 1954 (*w*), 1928 (*w*), 1770 (*vw*). ^{1}H NMR ($CDCl_3$): δ 7.20–6.80 (*m*, 20H, Ph), 2.01 (*s*, 15H, C_5Me_5).

Crystal data

$[WIr_3(C_{10}H_{15})(C_{14}H_{10})_2(CO)_7]$	D_x = 2.354 Mg m^{-3}
M_r = 1448.28	Mo Kα radiation
Monoclinic, $P2_1/c$	Cell parameters from 62525 reflections
a = 15.7919 (2) Å	θ = 3–27°
b = 14.3952 (2) Å	μ = 12.59 mm^{-1}
c = 17.9788 (2) Å	T = 200 K
β = 90.7217 (6)°	Needle, black
V = 4086.75 (9) Å^3	0.22 × 0.10 × 0.08 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	9375 independent reflections
φ and ω scans	5135 reflections with $I > 3\sigma(I)$
Absorption correction: by integration (Coppens, 1970; Mackay *et al.*, 2000)	R_{int} = 0.07
	θ_{max} = 27.5°
T_{min} = 0.179, T_{max} = 0.410	$h = -20 \rightarrow 20$
85630 measured reflections	$k = -18 \rightarrow 18$
	$l = -23 \rightarrow 21$

Refinement

Refinement on F	Weighting scheme: Chebychev polynomial with 5 parameters (Carruthers & Watkin, 1979): 0.912, 0.196, 1.01, 0.0217, 0.309
R = 0.022	$(\Delta/\sigma)_{max}$ = 0.006
wR = 0.025	$\Delta\rho_{max}$ = 1.10 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −1.59 e Å^{-3}
5135 reflections	
505 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å).

Ir1—Ir2	2.6928 (4)	Ir2—C70	2.175 (6)
Ir1—Ir3	2.6657 (4)	Ir3—W4	2.8309 (4)
Ir1—W4	2.7328 (4)	Ir3—C60	2.150 (7)
Ir1—C50	2.217 (7)	Ir3—C80	2.202 (6)
Ir1—C60	2.210 (6)	W4—C70	2.211 (7)
Ir2—Ir3	2.6006 (3)	W4—C80	2.188 (7)
Ir2—W4	2.7825 (4)	C50—C60	1.403 (9)
Ir2—C50	2.114 (7)	C70—C80	1.411 (9)

H atoms were made to ride on their parent C atoms, with C—H = 1.00 Å and U_{iso}(H) = 1.2U_{eq}(C). $\Delta\rho_{max}$ and $\Delta\rho_{min}$ are located in the vicinity of the metal atoms.

Data collection: *COLLECT* (Nonius, 1997–2001); cell refinement: *DENZO* and *SCALEPACK*; data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 2001); molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1992–1997); software used to prepare material for publication: *CRYSTALS*.

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