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# Structure of a Fischer Carbene Complex (CO)₅W=C(OEt)[1-(8-Iodonaphthyl)]

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Abstract. Pentacarbonyl[ethoxy(8-iodonaphthyl)-methylene]tungsten, $[W(CO)_5\{C(OC_2H_5)(C_{10}H_6I)\}]$, $M_r = 634.0$, monoclinic, $P2_1/c$, $a = 15.664(5)$, $b = 9.373(2)$, $c = 13.964(4)$ Å, $\beta = 110.79(2)°$, $V = 1917(2)$ Å^3, $Z = 4$, $D_x = 2.20$ g cm^{-3}, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 77.8$ cm^{-1}, $F(000) = 1176$, $T = 293$ K, $R = 0.031$ for 2581 reflections with $F_o^2 > 3\sigma(F_o^2)$. The W(CO)₅ group is bonded to the organic carbene with a W=C bond length of 2.180(6) Å and the W—C bonds to the carbonyl groups average 2.04(3) Å. In the W—carbene bonding the W=C—O bond angle is 132.1(5) and the W=C—C bond angle is 121.0(4)°.

Experimental. Title compound (I) obtained by the reaction of 1-lithio-8-iodonaphthalene with tungsten hexacarbonyl followed by ethylation with triethyloxonium tetrafluoroborate. Orange crystals obtained

(I)
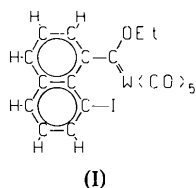
by slow evaporation of a pentane/dichloromethane solution. Data crystal $0.14 \times 0.20 \times 0.46$ mm mounted with epoxy on a glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ

scans of 4–16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $14 < 2\theta < 18$°. Analytical absorption correction based on crystal-face measurements varied from 0·51 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·59 Å^{-1}, $-18 < h < 18$, $0 < k < 11$, $0 < l < 16$. Three standard reflections (500, $23\bar{1}$, $60\bar{2}$) indicated crystal decomposition of less than 0·9% over 32·9 h of data collection. 3506 reflections measured, 3351 unique ($R_{int} = 0.02$), 770 reflections with $I < 3\sigma(I)$ where $\sigma^2(I) = \sigma_{cs}^2(I) + (0.05I)^2$; $\sigma_{cs}(I)$ is standard deviation of
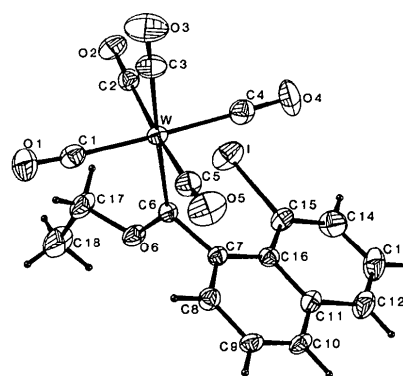

Fig. 1. *ORTEP* (Johnson, 1976) drawing and numbering scheme. The ellipsoids are drawn at the 30% probability surface and H atoms have been given arbitrary radii for figure clarity.

Table 1. *Fractional coordinates and isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
W	0.16593 (2)	0.17558 (3)	0.93437 (2)	2.626 (5)
I	0.23337 (4)	0.56383 (8)	1.10367 (5)	6.03 (2)
O(1)	0.0518 (5)	−0.0989 (9)	0.9403 (6)	8.4 (2)
O(2)	0.0707 (5)	0.1507 (8)	0.6930 (5)	6.5 (2)
O(3)	0.3159 (5)	−0.0507 (8)	0.9337 (6)	7.4 (2)
O(4)	0.2552 (5)	0.1693 (8)	1.1785 (5)	6.9 (2)
O(5)	−0.0009 (4)	0.3617 (7)	0.9454 (5)	4.9 (1)
O(6)	0.2377 (3)	0.4736 (6)	0.8767 (4)	3.7 (1)
C(1)	0.0929 (6)	0.001 (1)	0.9398 (7)	4.8 (2)
C(2)	0.1069 (5)	0.1645 (9)	0.7791 (6)	4.0 (2)
C(3)	0.2638 (5)	0.033 (1)	0.9319 (6)	4.3 (2)
C(4)	0.2230 (6)	0.1759 (9)	1.0905 (7)	4.5 (2)
C(5)	0.0603 (5)	0.2994 (8)	0.9414 (5)	3.3 (2)
C(6)	0.2537 (4)	0.3544 (7)	0.9307 (5)	2.6 (1)
C(7)	0.3553 (4)	0.3466 (7)	0.9848 (5)	2.6 (1)
C(8)	0.4012 (5)	0.2740 (9)	0.9321 (6)	3.4 (2)
C(9)	0.4962 (5)	0.2578 (9)	0.9713 (6)	3.5 (2)
C(10)	0.5440 (5)	0.3153 (8)	1.0622 (6)	3.5 (2)
C(11)	0.5017 (5)	0.3890 (8)	1.1198 (6)	3.1 (2)
C(12)	0.5569 (6)	0.444 (1)	1.2162 (7)	4.9 (2)
C(13)	0.5165 (7)	0.522 (1)	1.2755 (7)	5.7 (2)
C(14)	0.4247 (6)	0.548 (1)	1.2366 (7)	5.3 (2)
C(15)	0.3699 (5)	0.4974 (8)	1.1437 (6)	3.5 (2)
C(16)	0.4051 (4)	0.4112 (7)	1.0818 (5)	2.7 (1)
C(17)	0.1459 (5)	0.518 (1)	0.8148 (8)	5.2 (2)
C(18)	0.1536 (7)	0.651 (1)	0.7678 (9)	7.8 (3)

Table 2. *Some bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

W—C(1)	2.011 (9)	C(1)—W—C(2)	87.1 (4)
W—C(2)	2.035 (8)	C(1)—W—C(3)	84.9 (4)
W—C(3)	2.041 (8)	C(1)—W—C(4)	90.0 (4)
W—C(4)	2.042 (9)	C(1)—W—C(5)	88.8 (3)
W—C(5)	2.052 (8)	C(1)—W—C(6)	175.8 (3)
W—C(6)	2.480 (6)	C(2)—W—C(3)	90.5 (3)
I—C(15)	2.104 (7)	C(2)—W—C(4)	177.0 (3)
O(1)—C(1)	1.140 (10)	C(2)—W—C(5)	90.6 (3)
O(2)—C(2)	1.140 (10)	C(2)—W—C(6)	93.8 (3)
O(3)—C(3)	1.128 (9)	C(3)—W—C(4)	88.4 (3)
O(4)—C(4)	1.153 (10)	C(3)—W—C(5)	173.5 (3)
O(5)—C(5)	1.140 (9)	C(3)—W—C(6)	91.0 (3)
O(6)—C(6)	1.321 (8)	C(4)—W—C(5)	90.2 (3)
O(6)—C(17)	1.450 (8)	C(4)—W—C(6)	89.1 (3)
C(6)—C(7)	1.502 (9)	C(5)—W—C(6)	95.3 (3)
C(7)—C(8)	1.378 (10)	C(6)—O(6)—C(17)	121.9 (6)
C(7)—C(16)	1.435 (9)	W—C(1)—O(1)	178.1 (9)
C(8)—C(9)	1.400 (10)	W—C(2)—O(2)	175.5 (8)
C(9)—C(10)	1.337 (11)	W—C(3)—O(3)	175.9 (8)
C(10)—C(11)	1.393 (11)	W—C(4)—O(4)	176.9 (8)
C(11)—C(12)	1.414 (10)	W—C(5)—O(5)	176.3 (7)
C(11)—C(16)	1.430 (9)	W—C(6)—O(6)	132.1 (5)
C(12)—C(13)	1.411 (13)	W—C(6)—C(7)	121.0 (4)
C(13)—C(14)	1.366 (13)	O(6)—C(6)—C(7)	106.3 (5)
C(14)—C(15)	1.361 (11)	C(6)—C(7)—C(8)	114.6 (6)
C(15)—C(16)	1.429 (10)	C(6)—C(7)—C(16)	125.3 (6)
C(17)—C(18)	1.439 (13)	C(8)—C(7)—C(16)	120.0 (6)
W—I	4.264 (1)		

I based on counting statistics. No extinction correction was made. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declerq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\Sigma w(F_o - F_c)^2$. All H atoms were located on difference maps; for the refinement they were constrained to idealized positions (C—H = 0·95Å) with fixed isotropic *B* values of 1·2 times the *B* value of the attached C atoms. All non-H atoms were refined anisotropically for a total of 235 parameters. $R = 0.031$, $wR = 0.040$, GOF = 1·2, where non-Poisson $w^{-1} = [\sigma^2(I) + (0.05I)^2]/4F^2$. Final $(\Delta/\sigma)_{max} < 0.02$, $\rho_{max} = 0.8$ (1) and $\rho_{min} = -8$ (1) e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*. Table 1 gives the atomic coordinates and Table 2 selected bond distances and angles.* Fig. 1 shows the molecule with the numbering scheme.

Related literature. The structural chemistry of transition-metal carbene complexes has been reviewed by Schubert (1983). Comparable structures with (CO)$_5$W—carbene linkages include (CO)$_5$-WC(OEt)C$_5$H$_8$CH=CPh$_2$ (Daran & Jeannin, 1980) and (CO)$_5$WCPh$_2$ (Casey, Burkhardt, Bunnell & Calabrese, 1977). This structure can be considered a precursor for an organic halide chelate of the type described by Burk, Crabtree & Holt (1984).

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*Tables of angles in the organic moiety, anisotropic temperature factors and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51916 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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