organic ligand, triphenylphosphine oxide, has been extensively investigated in its many crystal modifications, most recently by Spek (1987) and Baures & Silverton (1990).

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(η-1,5-Cyclooctadiene)iodomethyl(2,4-pentanedionato-*O*,*O'*)iridium(III)

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Abstract. $[Ir(C_5H_7O_2)(I)(CH_3)(C_8H_{12})]$, $M_r = 541{\cdot}45$, monoclinic, $P2_1/c$, $a = 7{\cdot}879\,(7)$, $b = 16{\cdot}639\,(2)$, $c = 11{\cdot}856\,(2)$ Å, $\beta = 99{\cdot}36\,(4)°$, $V = 1533{\cdot}5\,(3)$ Å^3, $Z = 4$, $D_m = 2{\cdot}34\,(1)$, $D_x = 2{\cdot}344$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 112{\cdot}4$ cm^{-1}, $F(000) = 1012$, final $R = 0{\cdot}075$ for 2377 absorption-corrected reflections. This structure determination confirms the *trans* oxidative addition of CH_3I to [Ir(acac)(cod)]. The Ir—C(olefinic) distances range from 2·14 (2) to 2·19 (2) Å with an average of 2·17 (2) Å. Ir—C(methyl) = 2·17 (3), Ir—I = 2·833 (3) and both Ir—O distances are 2·05 (2) Å. Both the methyl C and I atoms are displaced towards the acac ring-atom plane [C—Ir—I = 156·6 (7)°] probably owing to steric restrictions imposed by H atoms on the olefinic C atoms.

Experimental. The complex was prepared from [Ir(acac)(cod)] and a tenfold molar excess CH_3I in acetone, vacuum distillation of the solvent and subsequent recrystallization by slow evaporation of a petroleum ether (b.p. 353–373 K) solution. Density of the yellow needle-shaped crystals was determined by the flotation method in thallous formate solution. Crystal size 0·11 × 0·12 × 0·2 mm, Enraf–Nonius CAD-4F diffractometer, graphite monochromator, Mo $K\alpha$ radiation, $\omega/2\theta$-scan technique, variable scan width where $\Delta\omega = (0{\cdot}58 + 0{\cdot}34\tan\theta)°$, scan rate 5·49° min^{-1} in ω, unit-cell parameters from least-squares refinement of 25 reflections with $6 < \theta < 20°$, measuring range $3 < \theta < 25°$, empirical absorption corrections (North, Phillips & Mathews, 1968), with minimum correction factor 0·65 and maximum 0·99, three standard reflections measured every 3600 s of X-ray exposure time varied −1·9%, all possible reflections with $\sin\theta/\lambda < 0{\cdot}60$ Å^{-1} in the index ranges $0 < h < 9$, $0 < k < 19$, $-14 < l < 14$. 2780 unique reflections of which 2377 observed reflections with $I > 2{\cdot}0\sigma(I)$ were used for all calculations (*XRAY*72, Stewart, Kruger, Ammon, Dickinson & Hall, 1972). The structure was solved by the heavy-atom method and subjected to anisotropic full-matrix least-squares refinement on F (163 variables). H atoms were not placed. Neutral-atom scattering factors (Cromer &

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($\text{Å}^2 \times 10^3$)

	x	y	z	U_{eq}*
Ir	8588 (1)	6253 (1)	2875 (1)	30 (1)
I	5584 (2)	6967 (1)	1641 (2)	46 (1)
O(1)	6862 (21)	5492 (9)	3443 (16)	42 (10)
O(2)	8589 (21)	5684 (10)	1343 (14)	41 (9)
C(1)	10377 (32)	5292 (15)	3454 (26)	49 (16)
C(11)	6153 (31)	4890 (15)	2810 (29)	52 (17)
C(12)	6495 (35)	4670 (14)	1724 (29)	53 (17)
C(13)	7535 (32)	5037 (15)	1070 (26)	48 (15)
C(14)	4883 (41)	4428 (16)	3382 (36)	77 (22)
C(15)	7683 (40)	4707 (19)	− 102 (27)	61 (19)
C(22)	12223 (33)	6912 (19)	3785 (25)	54 (17)
C(23)	11327 (38)	6920 (23)	4867 (29)	71 (21)
C(24)	9433 (31)	6592 (14)	4620 (21)	38 (13)
C(25)	8037 (27)	7094 (14)	4190 (20)	34 (12)
C(26)	8287 (43)	7992 (15)	3817 (24)	56 (17)
C(27)	9309 (44)	8118 (15)	2805 (33)	72 (21)
C(28)	9746 (33)	7299 (15)	2199 (23)	47 (15)
C(29)	11024 (28)	6786 (12)	2683 (22)	35 (13)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Mann, 1968) and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1962). Final $R = 0{\cdot}075$ and $wR = 0{\cdot}088$ with unit weights taken, the relatively high R value should be attributed to the small size of the crystal resulting in poor-quality diffraction data and possibly to the less-stringent criterion used above for recognizing unobserved reflections. $(\Delta\rho)_{max} = 5{\cdot}3$, $(\Delta\rho)_{min} = -4{\cdot}9$ e Å^{-3} within 1 Å from iridium, $(\Delta/\sigma)_{max} = 0{\cdot}026$. Final atomic coordinates are given in Table 1* and main geometrical parameters in Table 2, according to the numbering scheme given in Fig. 1.

Related literature. This structure determination supplements the mechanistic details of a kinetic study concerning the oxidative addition of CH_3I to [Ir(β-diketone)(cod)] complexes. Bond distances and angles can be compared with relevant data in *fac*-$Ir(CH_3)_3PMe_2Ph)_3$ (Lundquist, Folting, Huffman & Caulton, 1988), a partly metallacyclic norbornadiene complex $[Ir(C_7H_8)_3(acac)]$ (Bezman, Bird, Fraser & Osborn, 1980), a tetrafluorobenzobarrelene complex $[Ir(I)_2(TFB)(phen)]ClO_4.(CH_3)_2CO$ (Oro, Carmona & Esteruelas, 1986) and $[Ir_3(H)(\mu_3\text{-}S)_2(cod)_3(PMe_3)]$ (Bright, Jones, Koschmieder & Nunn, 1988).

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Table 2. *Bond lengths* (Å) *and angles* (°)

Ir—C(1)	2·17 (3)	O(2)—C(13)	1·37 (3)
Ir—O(1)	2·05 (2)	C(11)—C(14)	1·51 (5)
Ir—O(2)	2·05 (2)	C(13)—C(15)	1·52 (4)
Ir—I	2·833 (3)	C(22)—C(29)	1·50 (3)
Ir—C(24)	2·14 (2)	C(22)—C(23)	1·56 (5)
Ir—C(25)	2·19 (2)	C(23)—C(24)	1·57 (4)
Ir—C(28)	2·18 (3)	C(24)—C(25)	1·41 (3)
Ir—C(29)	2·16 (2)	C(25)—C(26)	1·58 (3)
O(1)—C(11)	1·32 (3)	C(26)—C(27)	1·56 (5)
C(11)—C(12)	1·41 (5)	C(27)—C(28)	1·60 (4)
C(12)—C(13)	1·36 (4)	C(28)—C(29)	1·37 (3)
C(1)—Ir—I	156·6 (7)	C(12)—C(11)—C(14)	121 (2)
O(1)—Ir—I	83·4 (4)	C(11)—C(12)—C(13)	129 (2)
O(2)—Ir—I	81·7 (4)	C(12)—C(13)—C(15)	120 (2)
C(1)—Ir—O(1)	82·7 (8)	O(2)—C(13)—C(15)	112 (2)
C(1)—Ir—O(2)	81·1 (9)	Ir—O(2)—C(13)	119 (2)
C(25)—Ir—I	81·6 (5)	C(22)—C(23)—C(24)	113 (2)
C(28)—Ir—I	80·8 (7)	C(23)—C(24)—C(25)	122 (2)
C(1)—Ir—C(24)	78·7 (10)	C(24)—C(25)—C(26)	122 (2)
C(1)—Ir—C(29)	78·1 (9)	C(25)—C(26)—C(27)	116 (2)
C(24)—Ir—C(25)	37·9 (8)	C(26)—C(27)—C(28)	114 (2)
C(28)—Ir—C(29)	36·9 (9)	C(27)—C(28)—C(29)	123 (2)
O(1)—Ir—O(2)	95·8 (7)	C(28)—C(29)—C(22)	126 (2)
Ir—O(1)—C(11)	122 (2)	C(29)—C(22)—C(23)	114 (2)
O(1)—C(11)—C(12)	126 (3)		

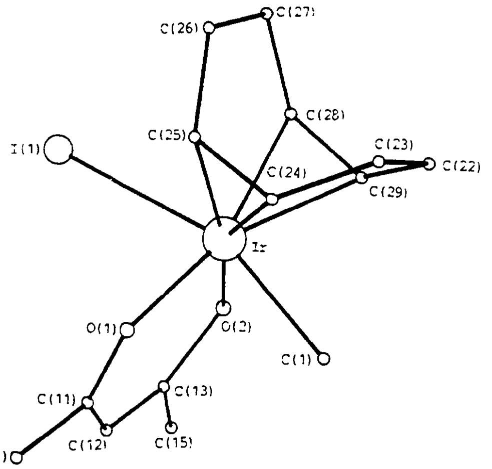

Fig. 1. View of the title molecule with atom numbering.

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* Lists of important non-bonding distances, anisotropic thermal parameters, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52103 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.