C(7)	0.1989 (2)	0.2934 (2)	0.3346 (1)	2.85 (4)
C(8)	0.1202 (2)	0.3808 (1)	0.2951 (1)	2.77 (4)
C(9)	0.1734 (3)	0.4713 (2)	0.3198 (1)	3.72 (4)
C(10)	0.0916 (3)	0.5486 (2)	0.2837 (2)	4.40 (5)
C(11)	−0.0404 (3)	0.5353 (2)	0.2229 (2)	4.73 (6)
C(12)	−0.0878 (3)	0.4435 (2)	0.1999 (2)	4.04 (5)
C(13)	−0.3447 (2)	0.2754 (2)	0.0835 (1)	3.31 (4)
C(14)	−0.4655 (2)	0.2492 (2)	0.0178 (1)	3.71 (5)
C(15)	−0.4786 (2)	0.1566 (2)	−0.0093 (1)	3.71 (5)
C(16)	−0.3714 (2)	0.0914 (2)	0.0312 (1)	3.37 (4)
C(17)	−0.2562 (2)	0.1212 (1)	0.0983 (1)	2.58 (3)
C(18)	−0.1468 (2)	0.0527 (1)	0.1508 (1)	2.79 (4)
C(19)	−0.1900 (2)	0.2376 (1)	0.2999 (1)	2.41 (3)
C(20)	0.0648 (2)	0.2222 (1)	0.1289 (1)	2.56 (4)

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

Co—N(1)	1.892 (1)	N(1)—C(7)	1.328 (2)
Co—N(2)	1.975 (2)	N(2)—C(8)	1.357 (2)
Co—N(3)	1.976 (1)	N(2)—C(12)	1.340 (3)
Co—N(4)	1.891 (2)	N(3)—C(13)	1.343 (2)
Co—C(19)	1.917 (2)	N(3)—C(17)	1.355 (2)
Co—C(20)	1.934 (2)	N(4)—C(1)	1.407 (3)
O(1)—C(18)	1.243 (2)	N(4)—C(18)	1.328 (2)
O(2)—C(7)	1.245 (2)	N(5)—C(19)	1.137 (3)
N(1)—C(6)	1.401 (3)	N(6)—C(20)	1.142 (3)
N(1)—Co—N(2)	82.71 (6)	N(3)—Co—N(4)	82.71 (6)
N(1)—Co—N(3)	167.12 (7)	N(3)—Co—C(19)	88.29 (7)
N(1)—Co—N(4)	84.42 (6)	N(3)—Co—C(20)	88.26 (7)
N(1)—Co—C(19)	92.23 (7)	N(4)—Co—C(19)	91.48 (7)
N(1)—Co—C(20)	91.13 (7)	N(4)—Co—C(20)	88.06 (7)
N(2)—Co—N(3)	110.17 (7)	C(19)—Co—C(20)	176.56 (7)
N(2)—Co—N(4)	167.09 (6)	Co—C(19)—N(5)	177.4 (1)
N(2)—Co—C(19)	89.99 (7)	Co—C(20)—N(6)	174.1 (2)
N(2)—Co—C(20)	91.22 (7)		

Table 3. *Coordination environment* (Å, °) *of the sodium cation*

Na···O(1^{i})	2.263 (2)	Na···N(5^{iii})	2.355 (2)
Na···O(2^{ii})	2.234 (2)	Na···N(6)	2.379 (2)
O(1^{i})···Na···O(2^{ii})	115.8 (1)	O(2^{ii})···Na···N(5^{iii})	115.5 (1)
O(1^{i})···Na···N(5^{iii})	91.1 (1)	O(2^{ii})···Na···N(6)	102.4 (2)
O(1^{i})···Na···N(6)	122.4 (1)	N(5^{iii})···Na···N(6)	110.1 (1)

Symmetry codes: (i) $-x, -y, -z$; (ii) $x+\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2}$; (iii) $x-\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2}$.

The structure was solved by the Patterson method and refined by full-matrix least squares. All calculations were performed using the *SDP* system (Enraf–Nonius, 1985) on a MicroVAX II computer.

We thank Dr S.-T. Mak for providing the crystals and the Hong Kong Research Grant Council and the University of Hong Kong for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: MU1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Carbonylchlorobis(tricyclohexylphosphine)iridium(I)

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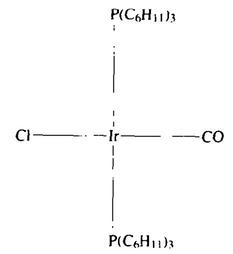
Abstract

The structure of *trans*-carbonylchlorobis(tricyclohexylphosphine)iridium(I), *trans*-$[IrCl(CO)\{P(C_6H_{11})_3\}_2]$, has been determined by single-crystal X-ray diffraction. The iridium(I) atom, situated on an inversion center, has a square-planar environment in which Ir(1)—P(1) = 2.345 (2), Ir(1)—Cl(1) = 2.398 (7), Ir(1)—CO = 1.78 (2) and C(1)—O(1) = 1.10 (2) Å.

Comment

The chemistry of complexes with the formula *trans*-$[IrCl(CO)(PR_3)_2]$ has been well studied (Dickson, 1983). Here we report the structure of the title complex, (I), which contains tricyclohexylphosphine ligands.



(I)

The unit cell of the title complex contains one *trans*-$[IrCl(CO)\{P(C_6H_{11})_3\}_2]$ molecule, as the Ir atom is situated on the inversion center at (0, 0, 0). The chloride and carbonyl ligands are necessarily disordered such that these ligands occupy mutually *trans* sites with 50% probability. This Cl/CO disorder is also present in the related crystal structures of *trans*-$[IrCl(CO)(PPh_3)_2]$ (Churchill, Fettinger, Buttrey, Barkan & Thompson, 1988) and *trans*-$[IrCl(CO)\{P(o\text{-tolyl})_3\}_2]$ (Brady *et al.*, 1975). It should also be mentioned that *trans*-$[IrCl(CO)\{P(C_6H_{11})_3\}_2]$ is isomorphous and nearly isostructural with $[OsClH(CO)\{P(C_6H_{11})_3\}_2]$ (Moers, Noordik & Beurskens, 1981).

The structure of (I) contains no abnormal distances. The Ir—P distance is 2.345 (2) Å, slightly longer than those found in the complexes *trans*-$[IrCl(CO)(PR_3)_2]$ in which the aryl groups *R* = phenyl [2.330 (1) Å] and *R* = *o*-tolyl [2.338 (5) Å] are present.

A slightly elongated Ir—P distance of 2.366 (4) Å is also found in the crystal structure of (η^4-1,5-cyclooctadiene)(pyridine)(tricyclohexylphosphine)iridium(I) hexafluorophosphate (Abbassioun, Hitchcock & Chaloner, 1989). In both tricyclohexylphosphine complexes one of the C—P—C angles is larger than the other two. In (I) the C—P—C angles are 103.8 (3), 103.0 (3) and 109.6 (3)°. These angles are 101.1 (6), 103.1 (7) and 110.8 (7)° in [Ir(η^4-1,5-C$_8$H$_{12}$)(C$_5$H$_5$N){P(C$_6$H$_{11}$)$_3$}][PF$_6$]. The observation of one C—P—C angle larger than the others is also observed, although to a lesser extent, in free P(C$_6$H$_{11}$)$_3$ [103.1 (3), 103.2 (3) and 105.1 (3)°] (Davies, Dutremez & Pinkerton, 1991).

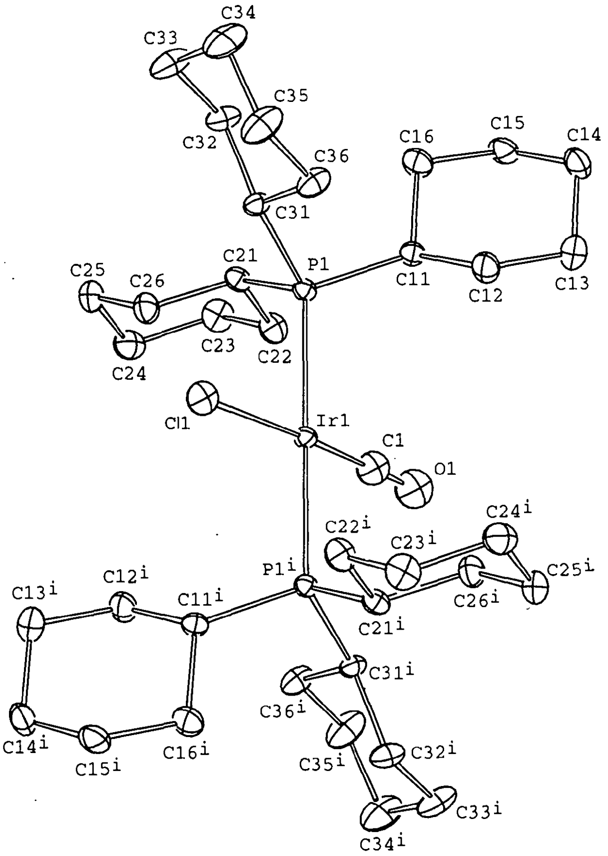


Fig. 1 . *ORTEP* (Johnson, 1965) view of (I) with 25% probability displacement ellipsoids.

Experimental

Diffraction-quality crystals were obtained from the overnight storage at room temperature of a concentrated 2:1 reaction mixture of P(C$_6$H$_{11}$)$_3$CS$_2$ and Vaska's complex, *trans*-[IrCl(CO)(PPh$_3$)$_2$], in carbon disulfide under an argon atmosphere. The Nujol-mull IR spectrum of these yellow crystalline plates exhibited a carbonyl stretching frequency at 1934 cm^{-1}, which is comparable to the literature value (Brady *et al.*, 1975). A suitable crystal was dipped in Paratone-N and sealed in a glass capillary for the crystallographic study.

Crystal data

[IrCl(CO)(C$_{18}$H$_{33}$P)$_2$]
M_r = 816.49
Triclinic
$P\overline{1}$
a = 10.251 (3) Å
b = 10.789 (4) Å
c = 9.943 (4) Å
α = 109.25 (3)°
β = 90.92 (3)°
γ = 113.76 (3)°
V = 936.0 (6) Å^3
Z = 1
D_x = 1.449 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 15 reflections
θ = 3.69–8.77°
μ = 3.750 mm^{-1}
T = 293 (2) K
Plate
0.20 × 0.14 × 0.08 mm
Lemon yellow

Data collection

Syntex $P2_1$ diffractometer
ω scans
Absorption correction: none
3373 measured reflections
3165 independent reflections
2980 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int}$ = 0.0367

$\theta_{\rm max}$ = 25.00°
$h = 0 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -10 \rightarrow 10$
3 standard reflections monitored every 50 reflections
intensity variation: none

Refinement

Refinement on F^2
$R(F)$ = 0.0447
$wR(F^2)$ = 0.0838
S = 0.986
3165 reflections
190 parameters
Calculated weights $w = 1/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\rm max}$ = 0.222

$\Delta\rho_{\rm max}$ = 1.424 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.893 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

$U_{\rm iso}$ for Cl(1), C(1) and O(1); $U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for all others.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Ir(1)	0	0	0	0.02456 (14)
P(1)	0.0291 (2)	0.2039 (2)	0.2020 (2)	0.0254 (4)
Cl(1)	0.0705 (6)	−0.1039 (6)	0.1507 (7)	0.0474 (12)
O(1)	−0.1054 (18)	0.1109 (17)	−0.1828 (16)	0.074 (6)
C(1)	−0.065 (2)	0.070 (2)	−0.111 (2)	0.053 (6)
C(11)	0.1101 (7)	0.3751 (6)	0.1675 (7)	0.0285 (15)
C(12)	0.2472 (8)	0.3993 (7)	0.1022 (8)	0.043 (2)
C(13)	0.2898 (9)	0.5271 (8)	0.0509 (8)	0.052 (2)
C(14)	0.3041 (9)	0.6650 (8)	0.1713 (8)	0.052 (2)
C(15)	0.1694 (9)	0.6406 (7)	0.2380 (8)	0.047 (2)
C(16)	0.1297 (9)	0.5143 (7)	0.2915 (8)	0.044 (2)
C(21)	−0.1462 (7)	0.1955 (7)	0.2548 (7)	0.030 (2)
C(22)	−0.2416 (8)	0.2072 (8)	0.1456 (8)	0.043 (2)
C(23)	−0.3822 (8)	0.2013 (8)	0.1968 (8)	0.046 (2)
C(24)	−0.4648 (8)	0.0622 (8)	0.2247 (8)	0.047 (2)
C(25)	−0.3711 (8)	0.0504 (8)	0.3360 (8)	0.047 (2)
C(26)	−0.2288 (7)	0.0571 (8)	0.2860 (8)	0.040 (2)
C(31)	0.1312 (7)	0.2249 (7)	0.3698 (6)	0.031 (2)
C(32)	0.1110 (8)	0.3165 (8)	0.5165 (7)	0.043 (2)
C(33)	0.1798 (9)	0.2982 (10)	0.6398 (8)	0.057 (2)
C(34)	0.3396 (9)	0.3383 (11)	0.6374 (8)	0.068 (3)
C(35)	0.3626 (9)	0.2540 (11)	0.4942 (8)	0.061 (2)
C(36)	0.2912 (8)	0.2683 (9)	0.3675 (7)	0.044 (2)

Table 2. *Selected geometric parameters* (Å, °)

Ir(1)—C(1)	1.78 (2)	C(15)—C(16)	1.530 (9)
Ir(1)—P(1)	2.345 (2)	C(21)—C(22)	1.519 (9)
Ir(1)—P(1$^{\mathrm{i}}$)	2.345 (2)	C(21)—C(26)	1.529 (9)
Ir(1)—Cl(1)	2.398 (7)	C(22)—C(23)	1.521 (10)
P(1)—C(11)	1.847 (6)	C(23)—C(24)	1.518 (10)
P(1)—C(31)	1.853 (6)	C(24)—C(25)	1.523 (10)
P(1)—C(21)	1.856 (6)	C(25)—C(26)	1.530 (10)
O(1)—C(1)	1.10 (2)	C(31)—C(36)	1.516 (9)
C(11)—C(12)	1.520 (9)	C(31)—C(32)	1.540 (8)
C(11)—C(16)	1.531 (8)	C(32)—C(33)	1.512 (9)
C(12)—C(13)	1.529 (9)	C(33)—C(34)	1.519 (11)
C(13)—C(14)	1.520 (10)	C(34)—C(35)	1.497 (11)
C(14)—C(15)	1.506 (11)	C(35)—C(36)	1.529 (9)
C(1)—Ir(1)—P(1)	89.0 (6)	C(14)—C(15)—C(16)	111.2 (6)
C(1)—Ir(1)—P(1$^{\mathrm{i}}$)	91.0 (6)	C(15)—C(16)—C(11)	110.4 (6)
P(1)—Ir(1)—P(1$^{\mathrm{i}}$)	180.0	C(22)—C(21)—C(26)	110.2 (6)
C(1)—Ir(1)—Cl(1)	176.1 (7)	C(22)—C(21)—P(1)	114.2 (5)
P(1)—Ir(1)—Cl(1)	91.24 (13)	C(26)—C(21)—P(1)	110.5 (4)
P(1$^{\mathrm{i}}$)—Ir(1)—Cl(1)	88.76 (13)	C(21)—C(22)—C(23)	111.2 (6)
C(11)—P(1)—C(31)	109.6 (3)	C(24)—C(23)—C(22)	111.2 (6)
C(11)—P(1)—C(21)	103.8 (3)	C(23)—C(24)—C(25)	110.0 (6)
C(31)—P(1)—C(21)	103.0 (3)	C(24)—C(25)—C(26)	110.7 (6)
C(11)—P(1)—Ir(1)	112.6 (2)	C(21)—C(26)—C(25)	111.6 (6)
C(31)—P(1)—Ir(1)	114.5 (2)	C(36)—C(31)—C(32)	109.5 (6)
C(21)—P(1)—Ir(1)	112.4 (2)	C(36)—C(31)—P(1)	113.9 (5)
O(1)—C(1)—Ir(1)	178.3 (15)	C(32)—C(31)—P(1)	118.7 (5)
C(12)—C(11)—C(16)	110.3 (6)	C(33)—C(32)—C(31)	110.9 (6)
C(12)—C(11)—P(1)	114.9 (4)	C(32)—C(33)—C(34)	111.5 (7)
C(16)—C(11)—P(1)	116.7 (5)	C(35)—C(34)—C(33)	111.1 (7)
C(11)—C(12)—C(13)	111.0 (6)	C(34)—C(35)—C(36)	112.6 (7)
C(14)—C(13)—C(12)	111.7 (6)	C(31)—C(36)—C(35)	111.1 (6)
C(15)—C(14)—C(13)	111.7 (6)		

Symmetry code: (i) $-x$, $-y$, $-z$.

The results of a ψ scan indicated that no correction for absorption was required. The position of the Ir atom was input for a *SHELXS*86 (Sheldrick, 1990) tangential expansion (*TEXP*) using 135 *E* values greater than 1.5. The resulting difference map revealed the atomic positions for the chloride and tricyclohexylphosphine ligands. These atoms, including occupancies of 50% for the Ir and Cl atoms, were input into *SHELXL*93 (Sheldrick, 1994). The C- and O-atom positions of the carbonyl group were determined, and the carbonyl group was assigned an occupancy of 50%. The Cl and carbonyl-group atoms were refined isotropically. All other non-H atoms were refined anisotropically. Idealized methine (C—H = 0.98 Å) and methylene (C—H = 0.97 Å) H atoms of the cyclohexyl groups were geometrically located then refined with fixed isotropic displacement parameters (U = 0.05 Å^2) using a riding model. Geometrical calculations were performed using *SHELXL*93. Data collection: *P*2$_1$ *Assembler Language Data Collection Programs* (Syntex, 1975). Data reduction: local program (Tipton, 1977). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: BK1025). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[1,1′-Bis(diphenylphosphino)ferrocene]tetracarbonylmolybdenum

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Abstract

Two crystallographically independent but chemically similar molecules (*A* and *B*) form the asymmetric unit of tetracarbonyl-1κ^4C-bis[μ-1κP:2(η^5)-diphenylphosphinocyclopentadienyl]ironmolybdenum, [{($C_{17}H_{14}P)_2Fe$}Mo(CO)$_4$]. The [bis(diphenylphosphino)ferrocene] ligand coordinates *via* the two P atoms, completing the *cis*-octahedral coordination of molybdenum. The carbonyl ligands *trans* to these phosphorus donors show the expected carbon–metal bond shortening. The ferrocene ligands have slightly non-parallel cyclopentadienyl rings, with staggered arrangements, and the P atoms are significantly elevated from the planes of the cyclopentadienyl rings.

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