

SHORT-FORMAT PAPERS

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Structure of Chloro(η^5 -pentamethylcyclopentadienyl)bis(trimethylphosphine)iridium(III) Hexafluorophosphate

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Abstract. $[\text{IrCl}(\text{C}_{10}\text{H}_{15})\{\text{P}(\text{CH}_3)_3\}_2]\text{PF}_6$, $M_r = 660.01$, monoclinic, $P2_1/n$, $a = 11.082$ (1), $b = 16.227$ (2), $c = 13.589$ (1) Å, $\beta = 97.11$ (1)°, $V = 2424.9$ (4) Å³, $Z = 4$, $D_x = 1.80$, $D_m = 1.81$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 58.36$ cm⁻¹, $F(000) = 1288$, $T = 298$ K, $R = 0.021$ for 2566 observed reflections. The Ir atom is in an octahedral environment coordinated to a tridentate pentamethylcyclopentadienyl group, a Cl atom and two P atoms of trimethylphosphine groups. The Ir-C₅Me₅ centroid, Ir-Cl, Ir-P(1) and Ir-P(2) bond distances are 1.875 (1), 2.410 (1), 2.289 (1) and 2.300 (1) Å, respectively. The P(1)-Ir-Cl, P(2)-Ir-Cl and P(1)-Ir-P(2) bond angles of 85.83 (5), 86.69 (5) and 96.40 (5)° comply well with octahedral coordination about the Ir atom.

Experimental. Salt prepared by reacting dichloride with potassium hexafluorophosphate in 2-propanol (Gilbert & Bergman, 1985); side product in synthesis of dihydride (Janowicz & Bergman, 1983). Thin, transparent, light-yellow crystals grown from methylene chloride/toluene solution by evaporation. Data crystal 0.35 × 0.26 × 0.11 mm, mounted on a glass fiber, Enraf-Nonius CAD-4 diffractometer (controlled by a DEC PDP8/a with an RK05 disk), graphite-monochromatized Mo $K\alpha$ radiation, lattice parameters from 24 reflections, $31 \leq 2\theta \leq 40^\circ$. Total of 3153 reflections measured using θ - 2θ scans, $3 \leq 2\theta \leq 45^\circ$ (h : -11→11, k : 0→17, l : 0→14); scan range (0.55 + 0.35 tan θ)°, scan speeds: min. 0.9, max. 3.3° min⁻¹, intensities of three standard reflections (004, 020, 400) measured every 2 h showed no decay during data collection, orientation standards checked every 250 reflections with no reorientation necessary; data reduction through Frenz and Associates, Inc. (1981) *SDP*; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); correction for Lorentz-polarization and absorption effects (transmission max. 64%, min.

31%, av. 48%); phasing via *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982); 2566 unique reflections with 587 reflections [$I \leq 3\sigma(I)$] considered unobserved; systematic absences of $h0l$, $h + l = 2n + 1$ and $0k0$, $k = 2n + 1$ uniquely indicate $P2_1/n$.

Position of Ir atom obtained from Patterson synthesis; Fourier difference map located all other non-H atoms; H atoms placed at idealized positions 0.95 Å from appropriate C atoms. Final full-matrix least-squares refinement included positional and anisotropic

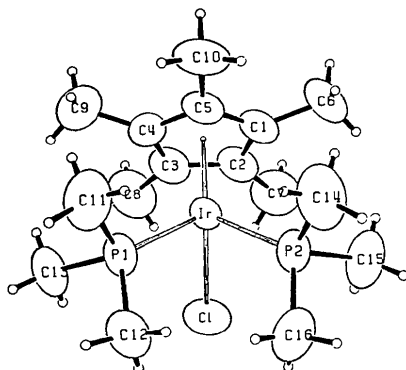
Table 1. Atomic coordinates and equivalent isotropic thermal parameters (Å²)

$$B_{\text{eq}} = \frac{1}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Ir	0.23997 (2)	0.18028 (1)	0.15697 (1)	3.313 (4)
Cl	0.3127 (2)	0.2795 (1)	0.2824 (1)	6.24 (4)
P(1)	0.2121 (1)	0.09480 (9)	0.2863 (1)	4.67 (3)
P(2)	0.0550 (1)	0.24578 (9)	0.1491 (1)	4.85 (3)
P(3)	0.2318 (1)	0.0184 (1)	0.6711 (1)	5.27 (4)
F(1)	0.1334 (5)	-0.0005 (3)	0.5825 (3)	11.3 (1)
F(2)	0.1938 (4)	-0.0599 (3)	0.7258 (3)	11.3 (1)
F(3)	0.2678 (4)	0.0999 (3)	0.6181 (3)	10.3 (1)
F(4)	0.1342 (4)	0.0698 (3)	0.7201 (4)	11.6 (1)
F(5)	0.3281 (4)	0.0390 (3)	0.7623 (3)	11.9 (1)
F(6)	0.3299 (5)	-0.0285 (4)	0.6254 (5)	16.2 (2)
C(1)	0.2509 (5)	0.1914 (3)	-0.0073 (4)	4.9 (1)
C(2)	0.3553 (5)	0.2277 (4)	0.0458 (4)	4.8 (1)
C(3)	0.4213 (5)	0.1626 (4)	0.1039 (4)	4.9 (1)
C(4)	0.3570 (5)	0.0889 (3)	0.0872 (4)	4.6 (1)
C(5)	0.2468 (5)	0.1070 (3)	0.0226 (3)	4.7 (1)
C(6)	0.1712 (7)	0.2298 (5)	-0.0920 (4)	7.7 (2)
C(7)	0.3975 (7)	0.3144 (4)	0.0400 (5)	7.7 (2)
C(8)	0.5442 (6)	0.1742 (4)	0.1638 (6)	7.7 (2)
C(9)	0.4009 (7)	0.0044 (4)	0.1156 (5)	7.7 (2)
C(10)	0.1600 (6)	0.0437 (4)	-0.0243 (5)	7.3 (2)
C(11)	0.1363 (7)	-0.0012 (4)	0.2540 (6)	8.0 (2)
C(12)	0.1276 (7)	0.1277 (5)	0.3828 (4)	7.7 (2)
C(13)	0.3555 (6)	0.0678 (4)	0.3569 (5)	7.1 (2)
C(14)	-0.0711 (6)	0.1953 (5)	0.0786 (7)	8.7 (2)
C(15)	0.0643 (7)	0.3463 (4)	0.0932 (6)	8.4 (2)
C(16)	-0.0092 (7)	0.2745 (5)	0.2597 (6)	9.4 (2)

Table 2. Selected bond distances (Å) and angles (°)

Ir—Cl	2.410 (1)	P(2)—C(15)	1.808 (6)
Ir—P(1)	2.289 (1)	P(2)—C(16)	1.801 (7)
Ir—P(2)	2.300 (1)	C(1)—C(2)	1.414 (7)
Ir—C ₅ , Me ₃ centroid	1.875 (1)	C(2)—C(3)	1.459 (7)
Ir—C(1)	2.258 (4)	C(3)—C(4)	1.397 (7)
Ir—C(2)	2.233 (4)	C(4)—C(5)	1.444 (7)
Ir—C(3)	2.236 (5)	C(1)—C(5)	1.431 (6)
Ir—C(4)	2.255 (5)	C(1)—C(6)	1.497 (7)
Ir—C(5)	2.188 (4)	C(2)—C(7)	1.488 (7)
P(1)—C(11)	1.798 (6)	C(3)—C(8)	1.510 (8)
P(1)—C(12)	1.785 (6)	C(4)—C(9)	1.489 (8)
P(1)—C(13)	1.805 (6)	C(5)—C(10)	1.495 (7)
P(2)—C(14)	1.791 (7)		
P(1)—Ir—Cl	85.83 (5)	C(15)—P(2)—C(16)	99.5 (4)
P(1)—Ir—P(2)	96.40 (5)	C(16)—P(2)—C(14)	101.7 (4)
P(1)—Ir—C ₅ , Me ₃ centroid	126.37 (3)	C(1)—C(2)—C(3)	107.4 (4)
Cl—Ir—P(2)	86.69 (5)	C(2)—C(3)—C(4)	108.7 (4)
C(1)—Ir—C ₅ , Me ₃ centroid	122.10 (4)	C(3)—C(4)—C(5)	107.5 (4)
P(2)—Ir—C ₅ , Me ₃ centroid	126.88 (3)	C(4)—C(5)—C(1)	108.2 (4)
C(11)—P(1)—Ir	116.18 (23)	C(5)—C(1)—C(2)	107.9 (4)
C(12)—P(1)—Ir	121.03 (22)	C(6)—C(1)—C(5)	125.5 (5)
C(13)—P(1)—Ir	111.18 (21)	C(6)—C(1)—C(2)	125.9 (5)
C(11)—P(1)—C(12)	99.6 (3)	C(7)—C(2)—C(1)	127.5 (5)
C(12)—P(1)—C(13)	100.9 (3)	C(7)—C(2)—C(3)	125.0 (5)
C(13)—P(1)—C(11)	105.9 (3)	C(8)—C(3)—C(2)	124.1 (5)
C(14)—P(2)—Ir	116.42 (25)	C(8)—C(3)—C(4)	127.0 (5)
C(15)—P(2)—Ir	109.85 (23)	C(9)—C(4)—C(3)	127.0 (5)
C(16)—P(2)—Ir	121.51 (23)	C(9)—C(4)—C(5)	124.8 (5)
C(14)—P(2)—C(15)	105.7 (3)	C(10)—C(5)—C(4)	124.8 (5)

Fig. 1. View of the $[\text{IrCl}(\text{C}_{10}\text{H}_{15})\{\text{P}(\text{CH}_3)_3\}_2]^+$ cation along the pseudo mirror plane with atomic labeling (ORTEP, Johnson, 1965).

thermal parameters for all non-H atoms; function minimized $\sum w(F_o - F_c)^2$ where $w = 1/\sigma^2(F_o^2)$ and $\sigma(F_o^2) = [(\sigma_F)^2 + (0.035F_o^2)^2]^{1/2}$, based on counting statistics; final $R = 0.021$, $wR = 0.027$, $S = 1.36$; $(\Delta/\sigma)_{\text{max}} = 0.07$ on final cycle; difference Fourier height max. = $0.49 \text{ e } \text{Å}^{-3}$.

The atomic coordinates are listed in Table 1 and bond distances and angles are presented in Table 2.* A view of the molecule along the pseudo mirror plane and the numbering scheme are shown in Fig. 1.

The crystal-structure analysis was performed by the authors at the U. C. Berkeley X-ray Diffraction Facility (CHEXRAY) under the supervision of Dr F. J. Hollander, Professor D. H. Templeton and Mr B. Borgias as part of a requirement for Chemistry 208.

* Lists of structure factors, anisotropic thermal parameters, positional and isotropic thermal parameters for calculated H atoms and selected least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42642 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Five-Coordinate Mononuclear Copper Complex*

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Abstract. Dibromo(1-methyl-2-imidazolecarbaldehyde azine)copper(II), $\text{C}_{10}\text{H}_{12}\text{Br}_2\text{CuN}_6$, $M_r = 439.6$, mono-

clinic, Cc , $a = 7.540 (1)$, $b = 13.613 (2)$, $c = 13.537 (3) \text{ Å}$, $\beta = 95.69 (2)^\circ$, $V = 1382.6 \text{ Å}^3$, $Z = 4$, $D_x = 2.112 \text{ g cm}^{-3}$, $\text{Cu K}\alpha_1$, $\lambda = 1.54056 \text{ Å}$, $\mu = 90.4 \text{ cm}^{-1}$, $F(000) = 852$, $T = 296 \text{ K}$, final $R = 0.038$

* NRCC No. 25323.