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Structure of *trans*(Cl,Cl),*trans*(P,P)-Dichlorobis{[2-(dimethylphosphino)ethyl]amine-*N*,*P*}iridium(III) Hexafluorophosphate, $[IrCl_2(edmp)_2].PF_6$

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Abstract. $[Ir(C_4H_{12}NP)_2Cl_2]PF_6$, $M_r = 618.3$, orthorhombic, *Fdd*2, $a = 23.659$ (8), $b = 32.794$ (10), $c = 10.042$ (5) Å, $V = 7791$ (5) Å^3, $Z = 16$, $D_m = 2.11$, $D_x = 2.11$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 7.44$ mm^{-1}, $F(000) = 4736$, $T = 297$ (1) K, $R = 0.036$ for 2230 unique observed reflections. The coordination geometry around the Ir atom is approximately octahedral with mean bond distances of 2.097 (10), 2.325 (4) and 2.358 (3) Å for Ir—N, Ir—P and Ir—Cl, respectively.

Experimental. The yellow prismatic crystals of the title compound were grown from the ethanol solution. D_m was measured by flotation in benzene/tetrabromoethane. X-ray intensities were measured on a Rigaku AFC-5 diffractometer with graphite-monochromated Mo $K\alpha$ radiation, θ–2θ scan, crystal size *ca* 0.3 × 0.5 × 0.5 mm. Mean ratio of five standard reflections, $\Sigma(|F_o|/|F_o|_{initial})/5$, was in the range 1.00–1.06. Unit-cell parameters were refined by least squares of 20 2θ values ($20 < 2\theta < 30°$). 2982 reflections were measured ($2\theta \leq 60°$, h 0–33, k 0–46, l 0–13), of which 2230 reflections were observed with $|F_o| > 3\sigma(|F_o|)$. Absorption correction was made by the Gaussian numerical integration method [transmission factors $0.090 < A < 0.195$ (Busing & Levy, 1957)]. Space group was uniquely determined to be *Fdd*2 from the systematic absences of the face-centered cell together with $0kl$, $k + l \neq 4n$ and $h0l$, $h + l \neq 4n$. Structure was solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); coordinates of all the non-H atoms refined by block-diagonal least squares with anisotropic thermal parameters using the *UNICS*III system (Sakurai & Kobayashi, 1979); nine among 24 H atoms were located on difference syntheses and others were calculated theoretically and included in the refinement with isotropic thermal parameters. Function $\Sigma w(|F_o| - |F_c|)^2$ was minimized with $w^{-1} = \sigma^2(|F_o|) + (0.015|F_o|)^2$. Final $R = 0.036$, $wR = 0.044$, $S = 1.85$ for 2230 reflections.§ $\Delta/\sigma < 0.14$, number of reflections/parameters = 7.56, $-3.3 < \Delta\rho < 1.6$ e Å^{-3}. The enantiomeric structure was rejected because of larger R factors, $R = 0.041$, $wR = 0.051$, $S = 2.13$. Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were performed on a FACOM M-380R of Keio University. Table 1 lists atomic coordinates and equivalent isotropic temperature factors for the non-H atoms and Table 2 gives selected bond lengths and angles. Fig. 1 is an *ORTEP* (Johnson, 1965) plot of the molecule.

Related literature. Preparation and characterization of rhodium(III) complexes containing (2-aminoethyl)dimethylphosphine (edmp) and crystal structures of *fac*-$[Rh(edmp)_3]Br_3.3H_2O$ and *trans*(Cl,Cl),-*cis*(P,P)-$[RhCl_2(edmp)_2]PF_6$ have been reported previously (Simonsen, Suzuki, Hamada, Kojima, Ohba, Sailo & Fujita, 1989). The Ir—Cl (*trans* to Cl) and Ir—P (*trans* to P) bond lengths are comparable with those in *mer*-$[IrCl_3(PMe_2Ph)_3]$, 2.359 (1)–2.368 (1) and 2.363 (1)–2.384 (1) Å, respectively (Robertson & Tucker, 1981). The Ir—N (*trans* to N) bond distances are similar to those in Δ, Λ-$[(H_2O)(en)_2$-

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§ Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54833 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0553]

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Table 1. *Positional parameters* ($\times 10^4$, $\times 10^5$ *for* Ir) *and equivalent isotropic temperature factors* ($Å^2 \times 10$) (*Hamilton*, 1959)

	x	y	z	B_{eq}
Ir	7259 (2)	8520 (1)	0*	22
Cl(1)	−185 (1)	565 (1)	−190 (4)	32
Cl(2)	1630 (1)	1147 (1)	202 (5)	41
P(1)	949 (2)	622 (1)	−2126 (3)	30
P(2)	480 (1)	1080 (1)	2115 (4)	30
N(1)	1028 (5)	278 (3)	558 (12)	31
N(2)	402 (5)	1418 (3)	−610 (12)	31
C(1)	1168 (7)	102 (4)	−1816 (16)	43
C(2)	1537 (9)	862 (5)	−2871 (19)	54
C(3)	396 (8)	574 (7)	−3368 (17)	55
C(4)	104 (7)	1563 (4)	1665 (19)	43
C(5)	−7 (7)	774 (6)	3115 (18)	52
C(6)	1048 (7)	1207 (5)	3264 (16)	46
C(7)	1422 (6)	91 (4)	−408 (17)	42
C(8)	391 (7)	1725 (4)	502 (19)	45
P(3)	1659 (2)	−248 (1)	3895 (5)	43
F(1)	2141 (8)	−162 (9)	2995 (24)	193
F(2)	1306 (6)	−405 (4)	2704 (18)	106
F(3)	1187 (9)	−305 (9)	4824 (23)	207
F(4)	2012 (8)	−110 (6)	5118 (21)	144
F(5)	1462 (11)	177 (5)	3716 (27)	182
F(6)	1858 (12)	−681 (6)	4122 (23)	177

* Parameter kept fixed for origin definition.

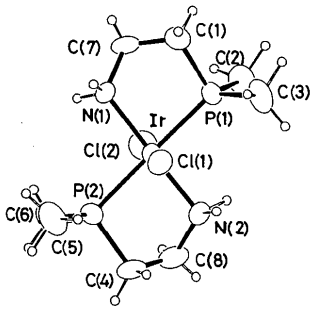

Fig. 1. An *ORTEP* (Johnson, 1965) drawing of the molecule with 50% probability ellipsoids. H atoms are represented by circles of radius 0.08 Å.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

Ir—Cl(1)	2.359 (3)	Ir—P(2)	2.326 (4)
Ir—Cl(2)	2.356 (3)	Ir—N(1)	2.090 (10)
Ir—P(1)	2.325 (3)	Ir—N(2)	2.099 (10)
Cl(1)—Ir—Cl(2)	179.2 (1)	P(2)—Ir—N(1)	97.5 (3)
Cl(1)—Ir—P(1)	90.2 (1)	P(2)—Ir—N(2)	83.7 (3)
Cl(1)—Ir—P(2)	88.5 (1)	N(1)—Ir—N(2)	177.9 (4)
Cl(1)—Ir—N(1)	88.6 (3)	Ir—P(1)—C(1)	102.3 (4)
Cl(1)—Ir—N(2)	89.8 (3)	Ir—P(1)—C(2)	117.6 (7)
Cl(2)—Ir—P(1)	90.4 (1)	Ir—P(1)—C(3)	119.7 (6)
Cl(2)—Ir—P(2)	90.9 (1)	Ir—P(2)—C(4)	99.8 (5)
Cl(2)—Ir—N(1)	92.1 (3)	Ir—P(2)—C(5)	118.9 (6)
Cl(2)—Ir—N(2)	89.6 (3)	Ir—P(2)—C(6)	117.9 (5)
P(1)—Ir—P(2)	178.6 (1)	Ir—N(1)—C(7)	114.4 (9)
P(1)—Ir—N(1)	82.9 (3)	Ir—N(2)—C(8)	112.4 (9)
P(1)—Ir—N(2)	95.8 (3)		

$Ir(OH)Ir(en)_2(OH)](S_2O_6)_{3/2}ClO_4.2.75H_2O$, 2.054 (8)–2.087 (8) Å [en = ethylenediamine (Galsbøl, Larsen, Rasmussen & Springborg, 1986)].

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Trichloromethoxytin(IV) Dimethanolate, $SnCl_3(OCH_3).2CH_3OH$

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Abstract. $M_r = 320.19$, triclinic, $P\bar{1}$, $a = 7.552$ (2), $b = 8.114$ (2), $c = 9.714$ (4) Å, $\alpha = 93.88$ (2), $\beta = 102.04$ (3), $\gamma = 114.80$ (2)°, $V = 520.3$ (4) Å^3, $Z = 2$, $D_x = 2.045$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 29.57$ cm^{-1}, $F(000) = 308$, $T = 210$ (2) K, $R = 0.056$ for 2651 reflections with $F_o \geq 4\sigma(F_o)$. The structure

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