Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dibromido(η^5-pentamethylcyclopentadienyl)(5-phenyldibenzophosphole-κP)-iridium(III) dichloromethane solvate

Mai Kotera and Takayoshi Suzuki*

Department of Chemistry, Graduate School of Science, Osaka University, Toyonaka 560-0043, Japan
Correspondence e-mail: suzuki@chem.sci.osaka-u.ac.jp

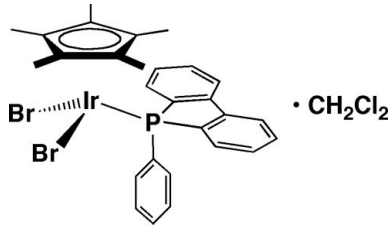
Received 24 May 2007; accepted 31 May 2007

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.012 Å; R factor = 0.040; wR factor = 0.126; data-to-parameter ratio = 21.2.

The title compound, $[IrBr_2(\eta^5\text{-}C_{10}H_{15})(C_{18}H_{13}P)]\cdot CH_2Cl_2$, is the first example of an iridium(III) phosphole complex to be characterized by X-ray crystallographic analysis. The Ir—P bond length is 2.2828 (15) Å, which is shorter by 0.04 Å than that of the analogous PPh_3 complex. This is indicative of the steric compactness of 5-phenyldibenzophosphole compared with PPh_3. The dibenzophosphole portion of the ligand is nearly planar, and the planes of the dibenzophosphole units of neighbouring molecules are stacked on top of each other to form a dimer in the crystal structure. The closest C···C distances between the planes are ∼3.9 Å.

Related literature

For related literature, see: Affandi *et al.* (1988); Attar *et al.* (1990); Farrugia (1997); Harder *et al.* (1991); Hayashi *et al.* (1983); Kessler *et al.* (1993); Le Bras *et al.* (1997); Matsuura *et al.* (1992); Meehan *et al.* (1997).



Experimental

Crystal data

$[IrBr_2(C_{10}H_{15})(C_{18}H_{13}P)]\cdot CH_2Cl_2$
M_r = 832.42
Monoclinic, $P2_1/a$
a = 11.0793 (5) Å
b = 23.3836 (10) Å
c = 12.8133 (5) Å
β = 117.746 (1)°
V = 2937.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.52 mm^{-1}
T = 200 (2) K
0.22 × 0.16 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: numerical (*ABSCOR*; Higashi, 1999)
T_{min} = 0.294, T_{max} = 0.406
28567 measured reflections
6708 independent reflections
6185 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.126
S = 1.08
6708 reflections
317 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.76 e Å^{-3}
$\Delta\rho_{min}$ = −2.74 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ir1—C1	2.155 (6)	Ir1—Br1	2.5022 (8)
Ir1—C3	2.160 (6)	Ir1—Br2	2.5118 (8)
Ir1—C2	2.169 (6)	P1—C17	1.807 (6)
Ir1—C4	2.231 (6)	P1—C28	1.809 (6)
Ir1—C5	2.233 (6)	P1—C11	1.823 (6)
Ir1—P1	2.2829 (15)		
P1—Ir1—Br1	89.56 (4)	C28—P1—C11	104.2 (3)
P1—Ir1—Br2	87.13 (4)	C17—P1—Ir1	117.0 (2)
Br1—Ir1—Br2	91.05 (3)	C28—P1—Ir1	116.6 (2)
C17—P1—C28	91.6 (3)	C11—P1—Ir1	119.48 (19)
C17—P1—C11	103.6 (3)		

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

This work was supported by a Grant-in-Aid for Scientific Research (No. 16550055) from the Ministry of Education, Culture, Sports, Science and Technology of Japan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2170).

References

Affandi, S., Nelson, J. H., Alcock, N. W., Howarth, O. W., Alyea, E. C. & Sheldrick, G. M. (1988). *Organometallics*, **7**, 1724–1734.
Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Attar, S., Bearden, W. H., Alcock, N. W., Alyea, E. C. & Nelson, J. H. (1990). *Inorg. Chem.* **29**, 425–433.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Harder, S., Brandsma, L., Kanters, J. A., Duisenberg, A. & van Lenthe, J. H. (1991). *J. Organomet. Chem.* **420**, 143–154.
Hayashi, T., Tanaka, M., Ogata, I., Kodama, T., Takahashi, T., Uchida, Y. & Uchida, T. (1983). *Bull. Chem. Soc. Jpn*, **56**, 1780–1785.
Higashi, T. (1999). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Kessler, J. M., Nelson, J. H., Frye, J. S., DeCian, A. & Fischer, J. (1993). *Inorg. Chem.* **32**, 1048–1052.
Le Bras, J., Amouri, H. & Vaissermann, J. (1997). *J. Organomet. Chem.* **548**, 305–307.
Matsuura, Y., Okude, K., Ichida, H., Miyamoto, T. K. & Sasaki, Y. (1992). *Acta Cryst.* C**48**, 357–358.
Meehan, P. R., Alyea, E. C. & Ferguson, G. (1997). *Acta Cryst.* C**53**, IUC9700001.
Rigaku (1998). *PROCESS-AUTO*. Version 1.06. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

  doi:10.1107/S1600536807026621

supplementary materials

Acta Cryst. (2007). E**63**, m1818 [**doi:10.1107/S1600536807026621**]

Dibromido(η^5-pentamethylcyclopentadienyl)(5-phenyldibenzophosphole-κP)iridium(III) dichloromethane solvate

M. Kotera and T. Suzuki

Comment

A large number of iridium(III) phosphine complexes has been characterized by X-ray analysis, while the structural study of the analogous phosphole complexes is limited. In fact, there has been no reports on the crystal structures of iridum(III) phosphole complexes, and only one paper describing the phosphole complex of iridium(I) (Hayashi *et al.*, 1983). Here, we report the crystal structure of iridium(III) complex containing 5-phenyldibenzophosphole, $[Cp^*IrBr_2(PhPC_{12}H_8)]\cdot CH_2Cl_2$ ($Cp^* = \eta^5$-C_5Me_5), (I). A perspective drawing of (I) is shown in Fig. 1. The structural parameters of the 5-phenyldibenzophosphole moiety are comparable to those of free ligand (Meehan *et al.*, 1997) and of the other metal complexes (Affandi *et al.*, 1988; Attar *et al.*, 1990; Matsuura *et al.*, 1992; Kessler *et al.*, 1993). The Ir—P bond is 2.2828 (15) Å, which is shorter by 0.04 Å than that of $[Cp^*IrCl_2(PPh_3)]$ (2.324 (3) Å; Le Bras *et al.*, 1997). This would be due to the steric compactness of 5-phenyldibenzophosphole than PPh_3. The dibenzophosphole moiety is nearly planar; the P atom is deviated by only 0.081 (7) Å from the least-square plane defined by biphenyl group. In the crystal, the dibenzophosphole planes in the neighboring molecules are stacked with each other (Fig. 2); the closest C···C distances between the planes are ~3.9 Å.

Experimental

The title complex, (I), was obtained as a by-product from the reaction mixture of $[Cp^*IrCl_2]_2$, $Ph_2PC_6H_4Br$ (Harder *et al.*, 1991) and ^{n}BuLi in THF (which was aimed to prepare $[Cp^*IrCl(Ph_2PC_6H_4)]$), together with the corresponding (bromo)(chloro) and dicholoro complexes, $[Cp^*IrBrCl(PhPC_{12}H_8)]\cdot CH_2Cl_2$ (II) and $[Cp^*IrCl_2(PhPC_{12}H_8)]\cdot CH_2Cl_2$ (III), respectively. The resulting red powdery product was recrystallized from a mixture of dichloromethane and methanol, affording red prismatic crystals. Although the crystal structure analyzed was consistent with the dibromo complex (I), the elemental analyses and the NMR spectra of the bulk sample fit well to the 4:4:1 mixture of the dibromo (I), (bromo)(chloro) (II) and dichloro (III) complexes. Anal. Found: C 43.59, H 3.79%. Calcd for $C_{28}H_{28}(Br/Cl)_2IrP\cdot CH_2Cl_2$: C 43.44, H 3.77%. ^{1}H NMR (399.7 MHz, 303 K, CD_2Cl_2): δ 1.34 (d, J = 2.4 Hz, Cp*; I), 1.30 (d, J = 2.4 Hz, Cp*; II), 1.26 (d, J = 2.4 Hz, Cp*; III), 7.22–8.20 (m, Ph—$PC_{12}H_8$). ^{31}P{^{1}H} NMR (202.4 MHz, 303 K, CD_2Cl_2): δ −17.78 (s; I), −14.64 (s; II), −11.34 (s; III).

Refinement

The H atoms were located geometrically and constrained to ride on their parent atoms with C—H = 0.95–0.99 Å with U_{iso}(H) = 1.2U_{eq}(C). The highest peak and deepest hole in the difference Fourier map are located 0.42 and 0.38 Å, respectively, from atoms Ir1 and Br1.

Figures

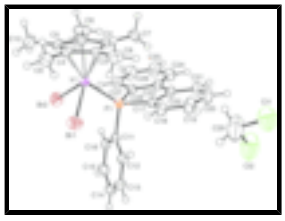


Fig. 1. An *ORTEP-3* (Farrugia, 1997) view of the components of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

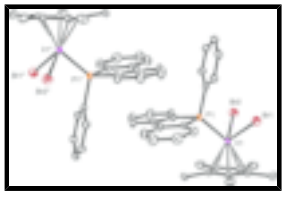


Fig. 2. A plot of the dimeric pair of (I). H atoms and the dichloromethane molecule have been omitted for clarity. The asterisk (*) corresponds to symmetry code ($1-x$, $1-y$, $1-z$).

Dibromido(η^5-pentamethylcyclopentadienyl)(5-\ phenyldibenzophosphole-κ*P*)iridium(III) dichloromethane solvate

Crystal data

$[IrBr_2(C_{10}H_{15})(C_{18}H_{13}P)]\cdot CH_2Cl_2$	F_{000} = 1600
M_r = 832.42	D_x = 1.882 Mg m^{-3}
Monoclinic, $P2_1/a$	Mo $K\alpha$ radiation λ = 0.71075 Å
Hall symbol: -P 2yab	Cell parameters from 17738 reflections
a = 11.0793 (5) Å	θ = 3.2–27.4º
b = 23.3836 (10) Å	μ = 7.52 mm^{-1}
c = 12.8133 (5) Å	T = 200 (2) K
β = 117.746 (1)º	Block, red
V = 2937.9 (2) Å^3	0.22 × 0.16 × 0.12 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	6708 independent reflections
Radiation source: fine-focus sealed tube	6185 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.045
Detector resolution: 10.00 pixels mm^{-1}	θ_{max} = 27.5º
T = 200(2) K	θ_{min} = 3.2º
ω scans	h = −14→14
Absorption correction: numerical (ABSCOR; Higashi, 1999)	k = −30→30
T_{min} = 0.294, T_{max} = 0.406	l = −16→16
28567 measured reflections	

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.126$

$S = 1.08$

6708 reflections

317 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 23.6384P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 1.76$ e Å^{-3}

$\Delta\rho_{min} = -2.74$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00141 (19)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ir1	0.59098 (2)	0.358232 (9)	0.903861 (19)	0.02182 (11)
Br1	0.67400 (9)	0.43878 (4)	1.04905 (7)	0.0467 (2)
Br2	0.83164 (8)	0.32885 (4)	0.96032 (8)	0.0465 (2)
Cl1	−0.1229 (3)	0.68037 (15)	0.6740 (3)	0.0792 (8)
Cl2	0.1036 (4)	0.67924 (19)	0.6196 (4)	0.1039 (13)
P1	0.60296 (15)	0.41650 (6)	0.76603 (13)	0.0222 (3)
C1	0.4956 (7)	0.2768 (3)	0.8362 (7)	0.0324 (14)
C2	0.3938 (6)	0.3206 (3)	0.7905 (6)	0.0275 (12)
C3	0.3896 (7)	0.3487 (3)	0.8888 (6)	0.0272 (12)
C4	0.4822 (7)	0.3181 (3)	0.9943 (6)	0.0318 (14)
C5	0.5475 (7)	0.2743 (3)	0.9627 (7)	0.0360 (15)
C6	0.5320 (9)	0.2348 (3)	0.7667 (9)	0.051 (2)
H6A	0.4914	0.1975	0.7665	0.061*
H6B	0.6314	0.2309	0.8028	0.061*
H6C	0.4970	0.2484	0.6855	0.061*
C7	0.2987 (7)	0.3322 (3)	0.6636 (6)	0.0383 (16)
H7A	0.3451	0.3242	0.6161	0.046*
H7B	0.2706	0.3725	0.6540	0.046*
H7C	0.2181	0.3077	0.6374	0.046*
C8	0.2938 (8)	0.3953 (3)	0.8842 (7)	0.0401 (16)
H8A	0.2522	0.4130	0.8059	0.048*
H8B	0.3445	0.4242	0.9440	0.048*
H8C	0.2223	0.3790	0.8997	0.048*
C9	0.5046 (9)	0.3308 (4)	1.1176 (7)	0.0470 (19)
H9A	0.5943	0.3165	1.1751	0.056*
H9B	0.4336	0.3120	1.1305	0.056*
H9C	0.5005	0.3722	1.1274	0.056*
C10	0.6511 (9)	0.2319 (4)	1.0427 (9)	0.054 (2)

H10A	0.6936	0.2463	1.1238	0.065*
H10B	0.7212	0.2263	1.0174	0.065*
H10C	0.6060	0.1953	1.0391	0.065*
C11	0.7462 (6)	0.4656 (3)	0.8101 (5)	0.0259 (12)
C12	0.7382 (8)	0.5204 (3)	0.8489 (6)	0.0338 (14)
H12	0.6588	0.5320	0.8537	0.041*
C13	0.8465 (8)	0.5581 (3)	0.8804 (6)	0.0392 (16)
H13	0.8408	0.5955	0.9070	0.047*
C14	0.9620 (8)	0.5419 (3)	0.8734 (6)	0.0449 (19)
H14	1.0355	0.5680	0.8951	0.054*
C15	0.9707 (7)	0.4882 (4)	0.8351 (7)	0.0423 (17)
H15	1.0507	0.4773	0.8304	0.051*
C16	0.8643 (7)	0.4492 (3)	0.8029 (6)	0.0333 (14)
H16	0.8713	0.4121	0.7764	0.040*
C17	0.4560 (7)	0.4607 (3)	0.6799 (6)	0.0280 (12)
C18	0.3897 (8)	0.4989 (3)	0.7191 (7)	0.0367 (15)
H18	0.4214	0.5050	0.8010	0.044*
C19	0.2749 (9)	0.5281 (4)	0.6349 (8)	0.051 (2)
H19	0.2288	0.5547	0.6599	0.061*
C20	0.2282 (9)	0.5183 (4)	0.5150 (8)	0.054 (2)
H20	0.1508	0.5386	0.4589	0.065*
C21	0.2930 (8)	0.4794 (3)	0.4762 (7)	0.0433 (17)
H21	0.2591	0.4727	0.3942	0.052*
C22	0.4073 (7)	0.4502 (3)	0.5573 (6)	0.0310 (13)
C23	0.4847 (7)	0.4055 (3)	0.5328 (6)	0.0320 (14)
C24	0.4606 (9)	0.3845 (4)	0.4231 (6)	0.0458 (19)
H24	0.3896	0.4001	0.3528	0.055*
C25	0.5406 (11)	0.3410 (4)	0.4178 (7)	0.053 (2)
H25	0.5241	0.3272	0.3426	0.064*
C26	0.6434 (10)	0.3167 (4)	0.5166 (7)	0.049 (2)
H26	0.6960	0.2863	0.5093	0.059*
C27	0.6702 (8)	0.3370 (3)	0.6280 (6)	0.0357 (15)
H27	0.7406	0.3205	0.6976	0.043*
C28	0.5904 (7)	0.3823 (3)	0.6347 (5)	0.0275 (12)
C29	0.0387 (14)	0.6521 (6)	0.7106 (13)	0.090 (4)
H29A	0.1023	0.6615	0.7937	0.109*
H29B	0.0323	0.6100	0.7031	0.109*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ir1	0.02293 (15)	0.02034 (14)	0.02187 (15)	0.00001 (8)	0.01015 (10)	0.00051 (8)
Br1	0.0534 (5)	0.0449 (4)	0.0441 (4)	−0.0071 (3)	0.0247 (4)	−0.0091 (3)
Br2	0.0401 (4)	0.0448 (4)	0.0530 (5)	0.0051 (3)	0.0204 (4)	0.0048 (3)
Cl1	0.0736 (18)	0.100 (2)	0.0667 (16)	0.0096 (16)	0.0346 (14)	0.0189 (15)
Cl2	0.091 (2)	0.131 (3)	0.107 (3)	0.032 (2)	0.060 (2)	0.052 (2)
P1	0.0242 (7)	0.0215 (7)	0.0199 (7)	−0.0004 (5)	0.0093 (6)	0.0001 (5)
C1	0.032 (3)	0.020 (3)	0.047 (4)	−0.005 (2)	0.020 (3)	−0.004 (3)

C2	0.025 (3)	0.027 (3)	0.029 (3)	−0.008 (2)	0.011 (2)	−0.003 (2)
C3	0.026 (3)	0.024 (3)	0.032 (3)	−0.003 (2)	0.015 (3)	−0.002 (2)
C4	0.032 (3)	0.036 (3)	0.031 (3)	−0.009 (3)	0.017 (3)	0.003 (3)
C5	0.033 (3)	0.026 (3)	0.050 (4)	−0.003 (3)	0.020 (3)	0.011 (3)
C6	0.048 (5)	0.037 (4)	0.075 (6)	−0.009 (3)	0.035 (4)	−0.021 (4)
C7	0.033 (4)	0.047 (4)	0.026 (3)	−0.016 (3)	0.007 (3)	−0.002 (3)
C8	0.037 (4)	0.037 (4)	0.057 (5)	0.001 (3)	0.030 (4)	−0.004 (3)
C9	0.048 (4)	0.065 (5)	0.031 (4)	−0.015 (4)	0.021 (3)	0.001 (4)
C10	0.040 (4)	0.047 (5)	0.068 (6)	0.002 (4)	0.020 (4)	0.027 (4)
C11	0.028 (3)	0.027 (3)	0.020 (3)	−0.004 (2)	0.008 (2)	0.003 (2)
C12	0.040 (4)	0.029 (3)	0.029 (3)	−0.003 (3)	0.013 (3)	0.001 (3)
C13	0.050 (4)	0.029 (3)	0.031 (3)	−0.013 (3)	0.013 (3)	−0.003 (3)
C14	0.046 (4)	0.044 (4)	0.030 (3)	−0.020 (3)	0.006 (3)	0.008 (3)
C15	0.027 (3)	0.057 (5)	0.037 (4)	−0.004 (3)	0.010 (3)	0.010 (3)
C16	0.032 (3)	0.033 (3)	0.032 (3)	0.001 (3)	0.012 (3)	0.007 (3)
C17	0.029 (3)	0.025 (3)	0.031 (3)	0.003 (2)	0.014 (3)	0.002 (2)
C18	0.038 (4)	0.035 (3)	0.039 (4)	0.008 (3)	0.019 (3)	0.006 (3)
C19	0.046 (4)	0.049 (5)	0.060 (5)	0.024 (4)	0.026 (4)	0.016 (4)
C20	0.043 (4)	0.060 (5)	0.052 (5)	0.021 (4)	0.016 (4)	0.025 (4)
C21	0.040 (4)	0.047 (4)	0.032 (4)	0.006 (3)	0.008 (3)	0.011 (3)
C22	0.034 (3)	0.031 (3)	0.026 (3)	0.000 (3)	0.012 (3)	0.008 (3)
C23	0.037 (3)	0.033 (3)	0.025 (3)	−0.007 (3)	0.013 (3)	0.001 (3)
C24	0.062 (5)	0.050 (4)	0.023 (3)	−0.008 (4)	0.017 (3)	−0.001 (3)
C25	0.082 (7)	0.052 (5)	0.030 (4)	0.001 (5)	0.030 (4)	−0.009 (4)
C26	0.072 (6)	0.044 (4)	0.041 (4)	0.007 (4)	0.034 (4)	−0.007 (3)
C27	0.047 (4)	0.036 (3)	0.029 (3)	0.003 (3)	0.022 (3)	−0.003 (3)
C28	0.035 (3)	0.026 (3)	0.021 (3)	−0.003 (2)	0.013 (2)	−0.001 (2)
C29	0.081 (9)	0.100 (9)	0.087 (9)	0.032 (7)	0.037 (7)	0.046 (8)

Geometric parameters (Å, °)

Ir1—C1	2.155 (6)	C10—H10B	0.9800
Ir1—C3	2.160 (6)	C10—H10C	0.9800
Ir1—C2	2.169 (6)	C11—C12	1.393 (9)
Ir1—C4	2.231 (6)	C11—C16	1.406 (10)
Ir1—C5	2.233 (6)	C12—C13	1.389 (10)
Ir1—P1	2.2829 (15)	C12—H12	0.9500
Ir1—Br1	2.5022 (8)	C13—C14	1.376 (12)
Ir1—Br2	2.5118 (8)	C13—H13	0.9500
Cl1—C29	1.756 (13)	C14—C15	1.369 (12)
Cl2—C29	1.748 (14)	C14—H14	0.9500
P1—C17	1.807 (6)	C15—C16	1.391 (10)
P1—C28	1.809 (6)	C15—H15	0.9500
P1—C11	1.823 (6)	C16—H16	0.9500
C1—C2	1.431 (9)	C17—C18	1.389 (9)
C1—C5	1.447 (10)	C17—C22	1.426 (9)
C1—C6	1.502 (10)	C18—C19	1.405 (10)
C2—C3	1.440 (9)	C18—H18	0.9500
C2—C7	1.495 (9)	C19—C20	1.394 (13)

C3—C4	1.450 (9)	C19—H19	0.9500
C3—C8	1.502 (9)	C20—C21	1.387 (12)
C4—C5	1.416 (10)	C20—H20	0.9500
C4—C9	1.510 (10)	C21—C22	1.386 (10)
C5—C10	1.504 (10)	C21—H21	0.9500
C6—H6A	0.9800	C22—C23	1.476 (10)
C6—H6B	0.9800	C23—C24	1.393 (10)
C6—H6C	0.9800	C23—C28	1.396 (9)
C7—H7A	0.9800	C24—C25	1.370 (13)
C7—H7B	0.9800	C24—H24	0.9500
C7—H7C	0.9800	C25—C26	1.371 (13)
C8—H8A	0.9800	C25—H25	0.9500
C8—H8B	0.9800	C26—C27	1.399 (10)
C8—H8C	0.9800	C26—H26	0.9500
C9—H9A	0.9800	C27—C28	1.408 (10)
C9—H9B	0.9800	C27—H27	0.9500
C9—H9C	0.9800	C29—H29A	0.9900
C10—H10A	0.9800	C29—H29B	0.9900
C1—Ir1—C3	65.2 (2)	C3—C8—H8B	109.5
C1—Ir1—C2	38.7 (2)	H8A—C8—H8B	109.5
C3—Ir1—C2	38.9 (2)	C3—C8—H8C	109.5
C1—Ir1—C4	63.6 (3)	H8A—C8—H8C	109.5
C3—Ir1—C4	38.5 (2)	H8B—C8—H8C	109.5
C2—Ir1—C4	63.7 (2)	C4—C9—H9A	109.5
C1—Ir1—C5	38.5 (3)	C4—C9—H9B	109.5
C3—Ir1—C5	64.1 (2)	H9A—C9—H9B	109.5
C2—Ir1—C5	63.9 (3)	C4—C9—H9C	109.5
C4—Ir1—C5	37.0 (3)	H9A—C9—H9C	109.5
C1—Ir1—P1	114.2 (2)	H9B—C9—H9C	109.5
C3—Ir1—P1	114.97 (18)	C5—C10—H10A	109.5
C2—Ir1—P1	97.34 (17)	C5—C10—H10B	109.5
C4—Ir1—P1	153.27 (19)	H10A—C10—H10B	109.5
C5—Ir1—P1	152.2 (2)	C5—C10—H10C	109.5
C1—Ir1—Br1	154.99 (19)	H10A—C10—H10C	109.5
C3—Ir1—Br1	98.68 (17)	H10B—C10—H10C	109.5
C2—Ir1—Br1	135.27 (18)	C12—C11—C16	119.3 (6)
C4—Ir1—Br1	91.78 (18)	C12—C11—P1	120.0 (5)
C5—Ir1—Br1	118.2 (2)	C16—C11—P1	120.7 (5)
P1—Ir1—Br1	89.56 (4)	C13—C12—C11	119.9 (7)
C1—Ir1—Br2	97.47 (18)	C13—C12—H12	120.1
C3—Ir1—Br2	155.72 (17)	C11—C12—H12	120.1
C2—Ir1—Br2	133.26 (18)	C14—C13—C12	120.6 (7)
C4—Ir1—Br2	119.53 (18)	C14—C13—H13	119.7
C5—Ir1—Br2	91.69 (18)	C12—C13—H13	119.7
P1—Ir1—Br2	87.13 (4)	C15—C14—C13	119.9 (7)
Br1—Ir1—Br2	91.05 (3)	C15—C14—H14	120.0
C17—P1—C28	91.6 (3)	C13—C14—H14	120.0
C17—P1—C11	103.6 (3)	C14—C15—C16	121.0 (8)
C28—P1—C11	104.2 (3)	C14—C15—H15	119.5

C17—P1—Ir1	117.0 (2)	C16—C15—H15	119.5
C28—P1—Ir1	116.6 (2)	C15—C16—C11	119.2 (7)
C11—P1—Ir1	119.48 (19)	C15—C16—H16	120.4
C2—C1—C5	108.2 (6)	C11—C16—H16	120.4
C2—C1—C6	127.2 (7)	C18—C17—C22	121.1 (6)
C5—C1—C6	124.2 (7)	C18—C17—P1	128.6 (5)
C2—C1—Ir1	71.2 (3)	C22—C17—P1	110.3 (5)
C5—C1—Ir1	73.7 (4)	C17—C18—C19	118.5 (7)
C6—C1—Ir1	126.9 (5)	C17—C18—H18	120.7
C1—C2—C3	108.0 (6)	C19—C18—H18	120.7
C1—C2—C7	126.7 (6)	C20—C19—C18	120.4 (8)
C3—C2—C7	125.1 (6)	C20—C19—H19	119.8
C1—C2—Ir1	70.1 (4)	C18—C19—H19	119.8
C3—C2—Ir1	70.2 (3)	C21—C20—C19	120.9 (7)
C7—C2—Ir1	129.4 (4)	C21—C20—H20	119.5
C2—C3—C4	107.0 (6)	C19—C20—H20	119.5
C2—C3—C8	127.3 (6)	C22—C21—C20	120.0 (7)
C4—C3—C8	125.1 (6)	C22—C21—H21	120.0
C2—C3—Ir1	70.9 (4)	C20—C21—H21	120.0
C4—C3—Ir1	73.4 (4)	C21—C22—C17	119.1 (7)
C8—C3—Ir1	127.6 (5)	C21—C22—C23	127.5 (7)
C5—C4—C3	108.9 (6)	C17—C22—C23	113.3 (6)
C5—C4—C9	125.8 (7)	C24—C23—C28	119.4 (7)
C3—C4—C9	125.3 (7)	C24—C23—C22	127.3 (7)
C5—C4—Ir1	71.6 (4)	C28—C23—C22	113.3 (6)
C3—C4—Ir1	68.1 (3)	C25—C24—C23	119.1 (7)
C9—C4—Ir1	126.5 (5)	C25—C24—H24	120.4
C4—C5—C1	107.6 (6)	C23—C24—H24	120.4
C4—C5—C10	127.7 (8)	C24—C25—C26	122.8 (7)
C1—C5—C10	124.7 (7)	C24—C25—H25	118.6
C4—C5—Ir1	71.4 (4)	C26—C25—H25	118.6
C1—C5—Ir1	67.8 (3)	C25—C26—C27	119.4 (8)
C10—C5—Ir1	126.5 (5)	C25—C26—H26	120.3
C1—C6—H6A	109.5	C27—C26—H26	120.3
C1—C6—H6B	109.5	C26—C27—C28	118.5 (7)
H6A—C6—H6B	109.5	C26—C27—H27	120.8
C1—C6—H6C	109.5	C28—C27—H27	120.8
H6A—C6—H6C	109.5	C23—C28—C27	120.9 (6)
H6B—C6—H6C	109.5	C23—C28—P1	111.4 (5)
C2—C7—H7A	109.5	C27—C28—P1	127.7 (5)
C2—C7—H7B	109.5	Cl2—C29—Cl1	111.7 (6)
H7A—C7—H7B	109.5	Cl2—C29—H29A	109.3
C2—C7—H7C	109.5	Cl1—C29—H29A	109.3
H7A—C7—H7C	109.5	Cl2—C29—H29B	109.3
H7B—C7—H7C	109.5	Cl1—C29—H29B	109.3
C3—C8—H8A	109.5	H29A—C29—H29B	107.9
C1—Ir1—P1—C17	85.8 (3)	C2—Ir1—C4—C5	80.8 (4)
C3—Ir1—P1—C17	13.1 (3)	P1—Ir1—C4—C5	129.3 (4)
C2—Ir1—P1—C17	49.4 (3)	Br1—Ir1—C4—C5	−138.1 (4)

C4—Ir1—P1—C17	6.8 (5)	Br2—Ir1—C4—C5	−45.9 (4)
C5—Ir1—P1—C17	94.5 (5)	C1—Ir1—C4—C3	−82.6 (4)
Br1—Ir1—P1—C17	−86.2 (2)	C2—Ir1—C4—C3	−39.3 (4)
Br2—Ir1—P1—C17	−177.3 (2)	C5—Ir1—C4—C3	−120.1 (6)
C1—Ir1—P1—C28	−21.1 (3)	P1—Ir1—C4—C3	9.2 (6)
C3—Ir1—P1—C28	−93.8 (3)	Br1—Ir1—C4—C3	101.7 (4)
C2—Ir1—P1—C28	−57.5 (3)	Br2—Ir1—C4—C3	−166.0 (3)
C4—Ir1—P1—C28	−100.1 (5)	C1—Ir1—C4—C9	158.8 (8)
C5—Ir1—P1—C28	−12.4 (5)	C3—Ir1—C4—C9	−118.5 (8)
Br1—Ir1—P1—C28	166.9 (2)	C2—Ir1—C4—C9	−157.8 (8)
Br2—Ir1—P1—C28	75.8 (2)	C5—Ir1—C4—C9	121.4 (9)
C1—Ir1—P1—C11	−147.9 (3)	P1—Ir1—C4—C9	−109.3 (7)
C3—Ir1—P1—C11	139.4 (3)	Br1—Ir1—C4—C9	−16.8 (7)
C2—Ir1—P1—C11	175.7 (3)	Br2—Ir1—C4—C9	75.4 (7)
C4—Ir1—P1—C11	133.1 (5)	C3—C4—C5—C1	−0.4 (7)
C5—Ir1—P1—C11	−139.2 (5)	C9—C4—C5—C1	179.4 (6)
Br1—Ir1—P1—C11	40.0 (2)	Ir1—C4—C5—C1	−58.4 (4)
Br2—Ir1—P1—C11	−51.0 (2)	C3—C4—C5—C10	−179.8 (7)
C3—Ir1—C1—C2	37.3 (4)	C9—C4—C5—C10	0.0 (11)
C4—Ir1—C1—C2	80.2 (4)	Ir1—C4—C5—C10	122.2 (7)
C5—Ir1—C1—C2	116.3 (6)	C3—C4—C5—Ir1	58.0 (4)
P1—Ir1—C1—C2	−70.2 (4)	C9—C4—C5—Ir1	−122.2 (7)
Br1—Ir1—C1—C2	90.6 (6)	C2—C1—C5—C4	−2.6 (7)
Br2—Ir1—C1—C2	−160.4 (4)	C6—C1—C5—C4	−175.5 (6)
C3—Ir1—C1—C5	−79.0 (4)	Ir1—C1—C5—C4	60.6 (5)
C2—Ir1—C1—C5	−116.3 (6)	C2—C1—C5—C10	176.8 (6)
C4—Ir1—C1—C5	−36.1 (4)	C6—C1—C5—C10	4.0 (11)
P1—Ir1—C1—C5	173.5 (3)	Ir1—C1—C5—C10	−119.9 (7)
Br1—Ir1—C1—C5	−25.7 (7)	C2—C1—C5—Ir1	−63.3 (4)
Br2—Ir1—C1—C5	83.3 (4)	C6—C1—C5—Ir1	123.9 (7)
C3—Ir1—C1—C6	160.2 (8)	C1—Ir1—C5—C4	−118.8 (6)
C2—Ir1—C1—C6	122.9 (9)	C3—Ir1—C5—C4	−36.8 (4)
C4—Ir1—C1—C6	−156.9 (8)	C2—Ir1—C5—C4	−80.2 (4)
C5—Ir1—C1—C6	−120.8 (9)	P1—Ir1—C5—C4	−131.7 (4)
P1—Ir1—C1—C6	52.6 (7)	Br1—Ir1—C5—C4	49.2 (4)
Br1—Ir1—C1—C6	−146.5 (6)	Br2—Ir1—C5—C4	141.3 (4)
Br2—Ir1—C1—C6	−37.6 (7)	C3—Ir1—C5—C1	82.0 (4)
C5—C1—C2—C3	4.7 (7)	C2—Ir1—C5—C1	38.6 (4)
C6—C1—C2—C3	177.2 (7)	C4—Ir1—C5—C1	118.8 (6)
Ir1—C1—C2—C3	−60.3 (4)	P1—Ir1—C5—C1	−12.9 (7)
C5—C1—C2—C7	−170.2 (6)	Br1—Ir1—C5—C1	168.0 (3)
C6—C1—C2—C7	2.3 (11)	Br2—Ir1—C5—C1	−99.9 (4)
Ir1—C1—C2—C7	124.9 (6)	C1—Ir1—C5—C10	117.6 (9)
C5—C1—C2—Ir1	64.9 (4)	C3—Ir1—C5—C10	−160.4 (8)
C6—C1—C2—Ir1	−122.5 (7)	C2—Ir1—C5—C10	156.2 (8)
C3—Ir1—C2—C1	−118.7 (5)	C4—Ir1—C5—C10	−123.6 (9)
C4—Ir1—C2—C1	−79.8 (4)	P1—Ir1—C5—C10	104.7 (8)
C5—Ir1—C2—C1	−38.4 (4)	Br1—Ir1—C5—C10	−74.4 (8)
P1—Ir1—C2—C1	120.1 (4)	Br2—Ir1—C5—C10	17.7 (8)

Br1—Ir1—C2—C1	−143.1 (3)	C17—P1—C11—C12	43.0 (6)
Br2—Ir1—C2—C1	27.1 (5)	C28—P1—C11—C12	138.2 (5)
C1—Ir1—C2—C3	118.7 (5)	Ir1—P1—C11—C12	−89.4 (5)
C4—Ir1—C2—C3	38.9 (4)	C17—P1—C11—C16	−135.5 (5)
C5—Ir1—C2—C3	80.3 (4)	C28—P1—C11—C16	−40.3 (6)
P1—Ir1—C2—C3	−121.2 (3)	Ir1—P1—C11—C16	92.1 (5)
Br1—Ir1—C2—C3	−24.4 (5)	C16—C11—C12—C13	−0.3 (9)
Br2—Ir1—C2—C3	145.8 (3)	P1—C11—C12—C13	−178.8 (5)
C1—Ir1—C2—C7	−121.6 (8)	C11—C12—C13—C14	0.2 (10)
C3—Ir1—C2—C7	119.7 (8)	C12—C13—C14—C15	0.0 (11)
C4—Ir1—C2—C7	158.6 (7)	C13—C14—C15—C16	−0.1 (11)
C5—Ir1—C2—C7	−160.0 (7)	C14—C15—C16—C11	0.0 (10)
P1—Ir1—C2—C7	−1.5 (7)	C12—C11—C16—C15	0.2 (9)
Br1—Ir1—C2—C7	95.3 (6)	P1—C11—C16—C15	178.7 (5)
Br2—Ir1—C2—C7	−94.5 (6)	C28—P1—C17—C18	175.0 (7)
C1—C2—C3—C4	−4.8 (7)	C11—P1—C17—C18	−79.9 (7)
C7—C2—C3—C4	170.1 (6)	Ir1—P1—C17—C18	53.9 (7)
Ir1—C2—C3—C4	−65.0 (4)	C28—P1—C17—C22	−2.6 (5)
C1—C2—C3—C8	−176.6 (6)	C11—P1—C17—C22	102.4 (5)
C7—C2—C3—C8	−1.6 (10)	Ir1—P1—C17—C22	−123.8 (4)
Ir1—C2—C3—C8	123.2 (7)	C22—C17—C18—C19	−1.5 (11)
C1—C2—C3—Ir1	60.2 (4)	P1—C17—C18—C19	−178.9 (6)
C7—C2—C3—Ir1	−124.8 (6)	C17—C18—C19—C20	0.8 (13)
C1—Ir1—C3—C2	−37.2 (4)	C18—C19—C20—C21	0.5 (14)
C4—Ir1—C3—C2	−115.2 (5)	C19—C20—C21—C22	−1.0 (14)
C5—Ir1—C3—C2	−79.9 (4)	C20—C21—C22—C17	0.3 (11)
P1—Ir1—C3—C2	69.3 (4)	C20—C21—C22—C23	176.6 (8)
Br1—Ir1—C3—C2	162.9 (3)	C18—C17—C22—C21	1.0 (10)
Br2—Ir1—C3—C2	−84.6 (5)	P1—C17—C22—C21	178.8 (6)
C1—Ir1—C3—C4	78.1 (4)	C18—C17—C22—C23	−175.8 (6)
C2—Ir1—C3—C4	115.2 (5)	P1—C17—C22—C23	2.0 (7)
C5—Ir1—C3—C4	35.4 (4)	C21—C22—C23—C24	1.8 (12)
P1—Ir1—C3—C4	−175.5 (3)	C17—C22—C23—C24	178.3 (7)
Br1—Ir1—C3—C4	−81.9 (4)	C21—C22—C23—C28	−176.5 (7)
Br2—Ir1—C3—C4	30.7 (6)	C17—C22—C23—C28	0.0 (8)
C1—Ir1—C3—C8	−160.1 (7)	C28—C23—C24—C25	0.6 (12)
C2—Ir1—C3—C8	−122.9 (8)	C22—C23—C24—C25	−177.6 (8)
C4—Ir1—C3—C8	121.8 (8)	C23—C24—C25—C26	0.6 (15)
C5—Ir1—C3—C8	157.2 (7)	C24—C25—C26—C27	−0.8 (15)
P1—Ir1—C3—C8	−53.6 (7)	C25—C26—C27—C28	−0.4 (13)
Br1—Ir1—C3—C8	40.0 (6)	C24—C23—C28—C27	−1.8 (10)
Br2—Ir1—C3—C8	152.5 (5)	C22—C23—C28—C27	176.7 (6)
C2—C3—C4—C5	3.2 (7)	C24—C23—C28—P1	179.6 (6)
C8—C3—C4—C5	175.2 (6)	C22—C23—C28—P1	−2.0 (7)
Ir1—C3—C4—C5	−60.1 (5)	C26—C27—C28—C23	1.7 (11)
C2—C3—C4—C9	−176.6 (6)	C26—C27—C28—P1	−179.9 (6)
C8—C3—C4—C9	−4.6 (10)	C17—P1—C28—C23	2.6 (5)
Ir1—C3—C4—C9	120.1 (7)	C11—P1—C28—C23	−101.9 (5)
C2—C3—C4—Ir1	63.4 (4)	Ir1—P1—C28—C23	124.1 (4)

C8—C3—C4—Ir1	−124.7 (6)	C17—P1—C28—C27	−175.9 (7)
C1—Ir1—C4—C5	37.5 (4)	C11—P1—C28—C27	79.6 (7)
C3—Ir1—C4—C5	120.1 (6)	Ir1—P1—C28—C27	−54.4 (7)

Fig. 1

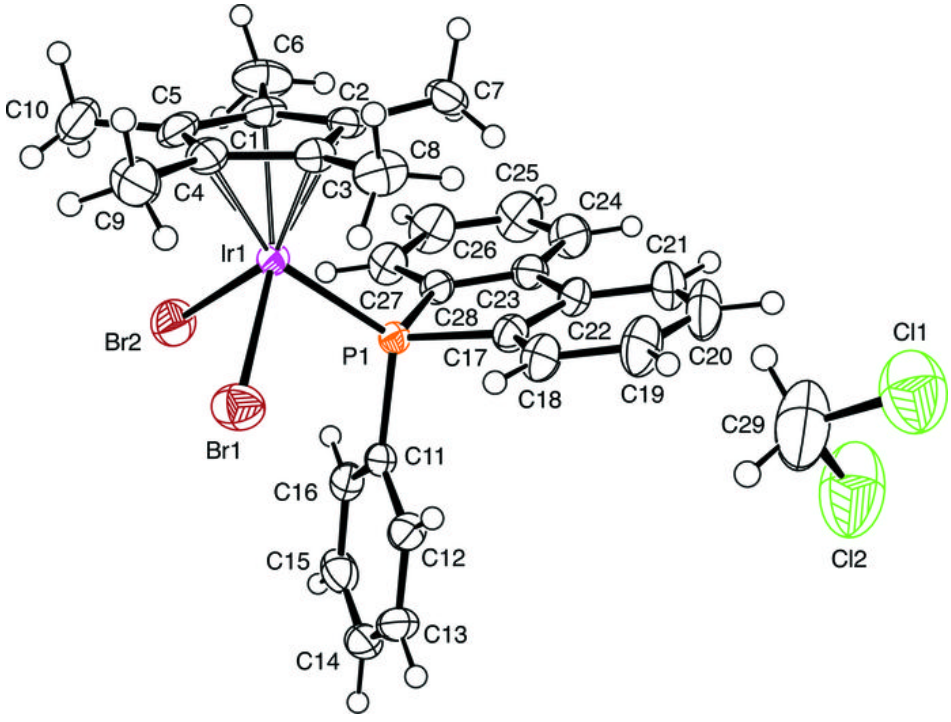

C6
C5
C2
C7
C10
C1
C8
C25
C24
C4
C3
C9
C26
C21
Ir1
C27
C23
C28
C22
Br2
P1
C17
C20
Cl1
C18
C19
C29
Br1
C11
C16
C12
C15
C13
C14
Cl2

Fig. 2