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Structure of [1,2-Bis(diphenylphosphino)ethane](η^5-cyclopentadienyl)(iodo)iron

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Abstract. $[\mathrm{FeI}(C_5H_5)(C_{26}H_{24}P_2)]$, M_r = 646·28, triclinic, $P\overline{1}$, a = 12·607 (2), b = 13·218 (1), c = 9·253 (2) Å, α = 105·80 (2), β = 101·48 (1), γ = 67·55 (1)°, V = 1363·4 Å^3, Z = 2, D_x = 1·574 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 18·06 cm^{-1}, $F(000)$ = 648, T = 293 (1) K, R = 0·024 for 3837 observed data with $I > 3\sigma(I)$. (The standard reduced cell obtained by the transformation matrix $[00\overline{1}/100/010]$ is a = 9·253, b = 12·607, c = 13·218 Å, α = 67·55, β = 74·20, γ = 78·52°.) The Fe atom is coordinated to an iodine [Fe—I 2·643 (1) Å], two P atoms of the ligand $(Ph_2PCH_2)_2$ [Fe—P 2·188 (1) and 2·189 (1) Å], and a cyclopentadiene ring [Fe—C in the range 2·054 (3)–2·106 (3) Å] with important angles: I—Fe—P 93·12 (2) and 89·61 (2)° and P—Fe—P 86·22 (3)°.

Experimental. Crystals were obtained by slowly evaporating solvent from a solution of [Fe(Cp)(dppe)I] ($Cp = C_5H_5$, $dppe = Ph_2PCH_2CH_2PPh_2$) (Treichel & Molzahn, 1979; Green & Whitely, 1971) in benzene. A dark-red crystal of approximate size 0·35 × 0·31 × 0·27 mm was mounted on a glass fiber for data collection. Unit-cell dimensions were determined from a least-squares fit of the setting angles of 25 reflections with $10 < \theta < 15°$ on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. Intensity data were collected by a conventional $\omega/2\theta$ scan method using variable scan speed (1·2–3·3° min^{-1}) in the range $2 < \theta < 25°$ with h −14→14, k 4→14, and l 0→10. The intensities of 4443 unique reflections were measured of which 3837 had $I > 3\sigma(I)$ and were used for structure solution and refinement. The intensities of three reflections chosen as standard and measured at 2 h exposure-time intervals did not show significant variation. Data were corrected for Lorentz and polarization factors and for empirical absorption (North, Phillips & Mathews, 1968); maximum and minimum correction factors were 0·9994 and 0·9476, respectively.

The structure was solved by the heavy-atom method and refined by full-matrix least-squares calculations on F's. Anisotropic temperature factors were allowed for non-H atoms. A difference map revealed all the H atoms which were included in the subsequent refinements and allowed to refine with an overall isotropic temperature factor, B = 4·0 Å^2. The refinement converged with R = 0·024 and wR = 0·033, where $w = [\sigma^2(F_o) + (0{\cdot}050\,F_o)^2]^{-1}$. At the conclusion of the refinement, maximum shift/e.s.d was < 0·02 for the non-H atoms, the final difference map showed peaks between −0·30 and 0·23 e Å^{-3}, and S = 1·040. Atomic scattering factors for non-H atoms were taken from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965); allowance was made for anomalous dispersion (Cromer & Liberman, 1970). The computer programs used in this study were from the Enraf–Nonius *Structure Determination Package* (B.A. Frenz & Associates Inc., 1985) and *ORTEP* (Johnson, 1976).

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2), *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}[a^2a^{*2}B_{11} + b^2b^{*2}B_{22} + c^2c^{*2}B_{33} + ab(\cos\gamma)a^*b^*B_{12} + ac(\cos\beta)a^*c^*B_{13} + bc(\cos\alpha)b^*c^*B_{23}].$$

	x	y	z	B_{eq}
I	0·75669 (2)	0·12565 (2)	0·51181 (2)	3·950 (4)
Fe	0·76118 (3)	0·13433 (3)	0·23121 (4)	2·728 (8)
P1	0·57825 (5)	0·23764 (5)	0·20719 (7)	2·91 (1)
P2	0·79394 (5)	0·29247 (5)	0·31546 (7)	2·79 (1)
C1	0·7899 (3)	−0·0359 (2)	0·1373 (4)	4·09 (7)
C2	0·8965 (2)	−0·0218 (2)	0·2014 (3)	3·87 (7)
C3	0·9142 (2)	0·0483 (3)	0·1305 (4)	4·35 (7)
C4	0·8201 (3)	0·0811 (3)	0·0222 (3)	4·86 (8)
C5	0·7448 (3)	0·0272 (3)	0·0261 (4)	4·94 (9)
C6	0·5552 (2)	0·3865 (2)	0·3067 (3)	3·66 (7)
C7	0·6653 (2)	0·3986 (2)	0·4010 (3)	3·36 (6)
C11	0·5190 (2)	0·2501 (2)	0·0117 (3)	3·15 (6)
C12	0·5629 (2)	0·3011 (2)	−0·0616 (3)	3·99 (7)
C13	0·5283 (3)	0·3044 (3)	−0·2119 (3)	4·44 (8)
C14	0·4508 (3)	0·2547 (3)	−0·2927 (3)	4·55 (8)
C15	0·4075 (3)	0·2028 (3)	−0·2225 (4)	4·48 (8)
C16	0·4408 (2)	0·1990 (2)	−0·0716 (3)	3·73 (7)
C21	0·4644 (2)	0·2075 (2)	0·2661 (3)	3·34 (6)
C22	0·4864 (2)	0·1040 (2)	0·2933 (3)	3·83 (7)
C23	0·3992 (2)	0·0772 (3)	0·3280 (4)	4·51 (7)
C24	0·2907 (3)	0·1527 (3)	0·3332 (4)	5·17 (8)
C25	0·2662 (3)	0·2577 (3)	0·3066 (4)	5·29 (9)
C26	0·3527 (2)	0·2853 (3)	0·2729 (3)	4·28 (7)
C31	0·8211 (2)	0·3559 (2)	0·1787 (3)	3·30 (6)
C32	0·7449 (3)	0·4531 (2)	0·1381 (4)	4·11 (7)
C33	0·7659 (3)	0·4939 (3)	0·0295 (4)	5·59 (9)
C34	0·8640 (4)	0·4386 (3)	−0·0411 (4)	6·7 (1)
C35	0·9429 (3)	0·3440 (3)	0·0002 (4)	6·7 (1)
C36	0·9222 (3)	0·3025 (3)	0·1090 (4)	5·10 (8)
C41	0·9115 (2)	0·3067 (2)	0·4617 (3)	3·18 (6)
C42	0·9167 (2)	0·4118 (2)	0·5339 (3)	4·07 (7)
C43	1·0088 (3)	0·4223 (3)	0·6392 (4)	4·79 (8)
C44	1·0963 (2)	0·3291 (3)	0·6707 (4)	4·73 (8)
C45	1·0930 (2)	0·2256 (3)	0·5993 (4)	4·43 (7)
C46	1·0012 (2)	0·2131 (2)	0·4957 (3)	3·56 (6)

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Table 2. *Molecular dimensions with e.s.d.'s in parentheses*

(*a*) Bond distances (Å)

Fe-I	2·643 (1)	C(12)—C(13)	1·379 (5)
Fe—P(1)	2·188 (1)	C(13)—C(14)	1·369 (5)
Fe—P(2)	2·189 (1)	C(14)—C(15)	1·364 (5)
Fe—C(1)	2·094 (3)	C(15)—C(16)	1·385 (5)
Fe—C(2)	2·106 (3)	C(21)—C(22)	1·371 (4)
Fe—C(3)	2·085 (3)	C(21)—C(26)	1·392 (4)
Fe—C(4)	2·054 (3)	C(22)—C(23)	1·392 (4)
Fe—C(5)	2·062 (3)	C(23)—C(24)	1·351 (5)
Fe—C(cp*)	1·707 (3)	C(24)—C(25)	1·383 (6)
P(1)—C(6)	1·868 (3)	C(25)—C(26)	1·385 (5)
P(1)—C(11)	1·840 (3)	C(31)—C(32)	1·375 (4)
P(1)—C(21)	1·840 (3)	C(31)—C(36)	1·390 (4)
P(2)—C(7)	1·842 (3)	C(32)—C(33)	1·372 (5)
P(2)—C(31)	1·842 (3)	C(33)—C(34)	1·364 (6)
P(2)—C(41)	1·828 (3)	C(34)—C(35)	1·363 (6)
C(1)—C(2)	1·416 (5)	C(35)—C(36)	1·376 (5)
C(1)—C(5)	1·396 (5)	C(41)—C(42)	1·387 (4)
C(2)—C(3)	1·373 (5)	C(41)—C(46)	1·382 (4)
C(3)—C(4)	1·403 (5)	C(42)—C(43)	1·384 (5)
C(4)—C(5)	1·398 (6)	C(43)—C(44)	1·360 (5)
C(6)—C(7)	1·524 (4)	C(44)—C(45)	1·360 (5)
C(11)—C(12)	1·380 (4)	C(45)—C(46)	1·379 (4)
C(11)—C(16)	1·395 (4)		

(*b*) Bond angles (°)

I—Fe—P(1)	93·12 (2)	C(12)—C(13)—C(14)	120·4 (4)
I—Fe—P(2)	89·61 (2)	C(13)—C(14)—C(15)	119·2 (3)
P(1)—Fe—P(2)	86·22 (3)	C(14)—C(15)—C(16)	121·3 (3)
I—Fe—C(cp*)	122·2 (1)	C(11)—C(16)—C(15)	119·9 (3)
P(1)—Fe—C(cp)	126·9 (1)	P(1)—C(21)—C(22)	120·1 (2)
P(2)—Fe—C(cp)	127·5 (1)	P(1)—C(21)—C(26)	121·1 (3)
Fe—P(1)—C(6)	110·84 (9)	C(22)—C(21)—C(26)	118·7 (3)
Fe—P(1)—C(11)	113·92 (8)	C(21)—C(22)—C(23)	120·8 (2)
Fe—P(1)—C(21)	123·26 (9)	C(22)—C(23)—C(24)	120·3 (3)
C(6)—P(1)—C(11)	102·5 (1)	C(23)—C(24)—C(25)	120·1 (4)
C(6)—P(1)—C(21)	103·4 (1)	C(24)—C(25)—C(26)	120·0 (3)
C(11)—P(1)—C(21)	100·4 (1)	C(21)—C(26)—C(25)	120·1 (3)
Fe—P(2)—C(7)	108·01 (10)	P(2)—C(31)—C(32)	123·4 (2)
Fe—P(2)—C(31)	118·22 (8)	P(2)—C(31)—C(36)	119·1 (2)
Fe—P(2)—C(41)	122·04 (9)	C(32)—C(31)—C(36)	117·4 (3)
C(7)—P(2)—C(31)	105·0 (1)	C(31)—C(32)—C(33)	121·4 (3)
C(7)—P(2)—C(41)	102·8 (1)	C(32)—C(33)—C(34)	120·4 (3)
C(31)—P(2)—C(41)	98·7 (1)	C(33)—C(34)—C(35)	119·5 (4)
C(2)—C(1)—C(5)	106·8 (3)	C(34)—C(35)—C(36)	120·3 (4)
C(1)—C(2)—C(3)	108·2 (3)	C(31)—C(36)—C(35)	120·9 (3)
C(2)—C(3)—C(4)	109·1 (3)	P(2)—C(41)—C(42)	120·6 (2)
C(3)—C(4)—C(5)	106·8 (3)	P(2)—C(41)—C(46)	120·8 (2)
C(1)—C(5)—C(4)	109·0 (3)	C(42)—C(41)—C(46)	118·5 (2)
P(1)—C(6)—C(7)	111·2 (2)	C(41)—C(42)—C(43)	120·6 (2)
P(2)—C(7)—C(6)	110·8 (2)	C(42)—C(43)—C(44)	120·0 (3)
P(1)—C(11)—C(12)	118·9 (2)	C(43)—C(44)—C(45)	120·0 (3)
P(1)—C(11)—C(16)	122·6 (3)	C(44)—C(45)—C(46)	121·1 (3)
C(12)—C(11)—C(16)	118·1 (3)	C(41)—C(46)—C(45)	119·9 (3)
C(11)—C(12)—C(13)	121·1 (3)		

*cp refers to the center of the cyclopentadiene ring.

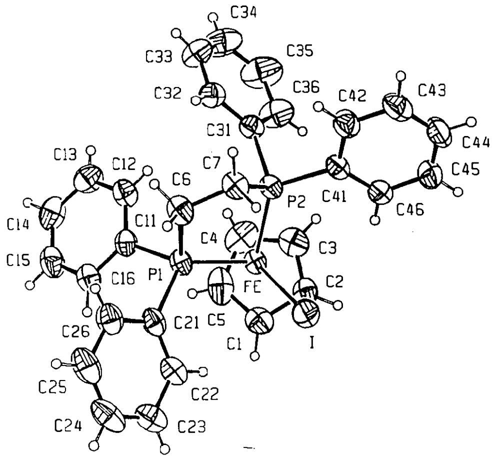

Fig. 1. Perspective drawing of the molecular structure with crystallographic numbering scheme. The shapes of the ellipsoids correspond to 50% probability contours of atomic displacement; H atoms have been plotted with arbitrary radii.

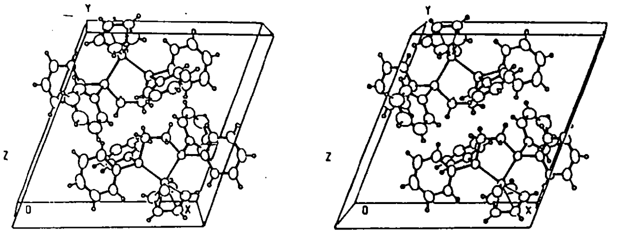

Fig. 2. Stereoscopic view of the unit cell showing molecular packing.

Table 1 gives the atomic coordinates and bond lengths and angles are listed in Table 2.* The molecular structure and the atom-numbering scheme are shown in Fig. 1, Fig. 2 shows the packing in the unit cell.

Related literature. The related complexes [Fe(Cp)(dppe)Br] (King, Houk & Pannell, 1969) and [Fe(Cp)(dppe)Cl] (King *et al.*, 1969; Mays & Sears, 1973) have been synthesized. The synthesis of [Fe(Cp)(dppe)MgBr] (Felkin, Knowles, Meunier, Mitschler, Richard & Weiss, 1974; Felkin, Knowles & Meunier, 1978) has been reported. The synthesis and characterization of [Fe(Cp)(dppe)H] has also been reported (Felkin *et al.*, 1978; Mays & Sears, 1973).

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* Tables of anisotropic temperature factors, H-atom coordinates, bond lengths involving H, mean-planes data and lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52111 (48 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of *cis*-Dichlorobis(dimethyl sulfide)platinum(II)

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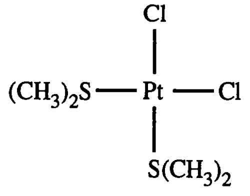
Abstract. $[\mathrm{PtCl_2\{S(CH_3)_2\}_2}]$, $M_r = 390{\cdot}26$, monoclinic, $P2_1/n$, $a = 8{\cdot}719\,(2)$, $b = 13{\cdot}186\,(4)$, $c = 9{\cdot}328\,(1)$ Å, $\beta = 106{\cdot}30\,(1)°$, $V = 1029{\cdot}3\,(7)$ Å^3, $Z = 4$, $D_x = 2{\cdot}518$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 146{\cdot}2$ cm^{-1}, $F(000) = 720$, $T = 296\,(1)$ K, 3559 unique reflections measured, final $R = 0{\cdot}034$ over 2495 reflections having $F_o > 3{\cdot}0\sigma(F_o)$. The geometry about the Pt atom is square planar with a maximum deviation of 0·007 (2) Å from the least-squares plane. Pt—S bond lengths: 2·269 (1) and 2·272 (1) Å; S—Pt—S angle: 94·75 (5)°. Pt—Cl bond lengths: 2·315 (1) and 2·319 (1) Å; Cl—Pt—Cl angle: 89·69 (5)°. S—Pt—Cl angles: 174·10 (5), 91·11 (5), 84·44 (5) and 179·18 (5)°. Average S—C bond length: 1·785 (3) Å. Centrosymmetrically related molecules stack in chains along the **c** direction with alternating Pt—Pt distances of 3·9971 (4) and 5·4147 (4) Å, and Pt—Pt—Pt angles of 164·52 (1)°. The closest intermolecular contact is between Cl2 and Cl at $-x - \frac{1}{2}$, $y - \frac{1}{2}$, $-z - \frac{1}{2}$ with a distance of 3·429 (7) Å between them.

Experimental. The title compound (I) was prepared from tetrachloroplatinate(II) and dimethyl sulfide by the method of Roulet & Barbey (1973). The sample crystal was mounted on a glass fiber in a random orientation. Details of data collection and structure refinement are given in Table 1. Space group determined from systematic absences: $h0l$ with $h + l$ odd; $0k0$ with k odd. The position of the Pt atom was determined from a Patterson map, and the remaining atoms were located in succeeding difference Fourier syntheses. Refinement by full-matrix least squares with Enraf–Nonius *SDP/VAX* (Frenz, 1978); non-H atoms anisotropic; H atoms located by ΔF synthesis and placed in calculated positions with B_{iso} of the calculated H atoms given values of 1·3 times B_{eq} of the C atom. H atoms included in the structure-factor calculations riding on



$$\begin{array}{c} \mathrm{Cl} \\ | \\ (\mathrm{CH_3})_2\mathrm{S}—\mathrm{Pt}—\mathrm{Cl} \\ | \\ \mathrm{S(CH_3)_2} \end{array}$$

(I)

Table 1. *Experimental details*

Crystal description	Yellow, fragment of a plate, 0·38 × 0·30 × 0·30 mm
Instrument	Enraf–Nonius CAD-4 diffractometer, graphite monochromator
Corrections	Lorentz–polarization Linear decay (0·952–1·130 on I) Empirical absorption (0·38–0·99 on I) Extinction [3·23 (7) × 10^{-7}]
Maximum 2θ (°)	64·0
hkl ranges	$h = 0$–13 $k = 0$–19 $l = -13$–13
No. of reflections	3894 total 3559 unique
R_{int} of averged reflections	0·031
No. unobserved reflections	1064
Reflections included	2495 with $F_o > 3\sigma(F_o)$
Solution	Heavy-atom method
Function minimized	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
Weights	$4F_o^2\mathrm{Lp}^2/[S^2(C + R^2B) + (0{\cdot}02F_o^2)^2]$ Lp = Lorentz–polarization S = scan rate C = total integrated peak count R = ratio of scan time to background counting time B = total background count
Parameters refined	83
Unweighted residual, R	0·034
Weighted residual, wR	0·038
Goodness of fit, S	1·62
Maximum Δ/σ	0·01

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