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Structure of *trans*-Diiodobis[tris(pentafluorophenyl)phosphine]platinum(II)–Dichloromethane (1/2)

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Abstract. $[\mathrm{PtI_2(C_{18}F_{15}P)_2}].2\mathrm{CH_2Cl_2}$, $M_r = 1683{\cdot}2$, monoclinic, $P2_1/a$, $a = 19{\cdot}424\,(7)$, $b = 13{\cdot}719\,(4)$, $c = 9{\cdot}426\,(6)$ Å, $\beta = 111{\cdot}94\,(5)°$, $V = 2330\,(2)$ Å^3, $Z = 2$, $D_x = 2{\cdot}399$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 45{\cdot}3$ cm^{-1}, $F(000) = 1568$, $T = 293$ K, $R = 0{\cdot}061$ for 1776 unique reflections with $I \geq 3\sigma(I)$. The Pt atoms lie on sites of $\bar{1}$ symmetry and display slightly distorted square-planar coordination with P—Pt—I $= 85{\cdot}1\,(2)°$. The Pt—I and Pt—P distances of $2{\cdot}626\,(2)$ and $2{\cdot}292\,(6)$ Å are not lengthened appreciably by overcrowding of the bulky iodo and phosphine ligands.

Introduction. According to Tolman (1977) $(\mathrm{C_6F_5})_3\mathrm{P}$ is one of the most bulky of phosphine ligands, with a cone angle of 184°. Consistent with this, metal complexes of $(\mathrm{C_6F_5})_3\mathrm{P}$ display rotational isomerism (Kemmitt, Nichols & Peacock, 1967). In particular, spectroscopic studies suggest that the title complex may exist as eclipsed and staggered rotamers in solution (Docherty, Rycroft, Sharp & Webb, 1979). This analysis was carried out to establish the rotameric form of the solid compound and the extent of any distortion of the metal coordination arising from the presence of the bulky phosphine and iodo ligands.

Experimental. Red, transparent needle grown from dichloromethane by solvent extraction at 277 K; forms exhibited and face–centroid distances (mm): {110} 0·037, {001} 0·117; Enraf–Nonius CAD-4F diffractometer, Mo X-rays, graphite monochromator; cell dimensions by least-squares fit to the setting angles of 25 automatically centred reflections with $10 < \theta < 16°$; intensities of 3634 reflections, with $2 \leq \theta(\mathrm{Mo}\,K\alpha) \leq 25°$ and $h\,0\rightarrow23$, $k\,0\rightarrow16$, $l\,-11\rightarrow11$, measured from continuous $\theta/2\theta$ scans of 0·80° in θ increased by 25% at each end to assess background effects; corrections for Lp, crystal decomposition (up to 28% of I based on intensities of 200 and 020 standard reflections) and absorption effects (Gaussian quadrature, 192 grid points, transmission factors on F^2 0·66–0·77), no correction required for extinction; R_{int} 0·066 for 150 independent reflections measured at least twice; 3278 independent structure amplitudes; of these 1776 with $I \geq 3\sigma(I)$ used in the structure analysis. Patterson and difference syntheses; full-matrix least-squares refinement on F; $w^{-1} = \sigma^2(F) + 4 \times 10^{-4}F^2$; H atoms riding on bonded C atoms with C—H = 1·08 Å; final adjustment of 349 parameters (anisotropic U_{ij} for all non-H atoms) gave $R = 0{\cdot}061$, $wR = 0{\cdot}060$, S

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= 2·4; max. Δ/σ = 0·20; final $|\Delta\rho|$ values <0·90 e Å^{-3} except for regions <4·1 e Å^{-3} near Pt and I; neutral-atom scattering factors and complex anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); *SHELX76* crystallographic program package (Sheldrick, 1976). Final atomic coordinates are presented in Table 1.*

Discussion. The crystal structure is composed of discrete *trans*-$[PtI_2\{(C_6F_5)_3P\}_2]$ and CH_2Cl_2 molecules, the latter being held rather loosely. The platinum atoms lie on crystallographic centres of symmetry: this implies both that the metal coordination is exactly planar and that the solid contains a rotamer with a staggered arrangement of the P—C bonds of the *trans*-phosphine ligands when these bonds are viewed along the P—Pt—P axis. The orientation of the phosphine ligands (Fig. 1) causes the P—C(21) bonds almost to coincide with the metal coordination plane [I—Pt—P—C(21) = 178 (1)°]. Although near coincidence of a P—C bond with the metal coordination plane is common in Pt^{II}—PR_3 complexes and favours release of overcrowding, this feature is not thought to be present in tris(pentafluorophenyl)phosphineplatinum(II) complexes in solution (Docherty *et al.*, 1979).

In the title complex there are several short contacts between the atoms of different ligands. The most significant of these involves an I′···C(22) separation of 3·49 (2) Å which is relieved by opening of the I′—Pt—P angle to 94·9 (2)°.† The Pt—P bond length of 2·292 (6) Å agrees well with the mean value of 2·30 Å for Pt—P(*trans*-P) distances in Pt^{II} complexes containing less bulky phosphines (Manojlović-Muir, 1978). It contrasts with values of 2·371 (2) and 2·348 (2) Å found in *trans*-PtI_2L_2 complexes with L = tricyclohexylphosphine (Alcock & Leviston, 1974) and L = tri-*o*-tolylphosphine (Alyea, Dias, Ferguson & Roberts, 1979) where ligand overcrowding is thought to lengthen the Pt—P bonds. The Pt—I bond length found here [2·626 (2) Å] is also comparable with the corresponding values of 2·612 (1) and 2·622 (1) Å found in the latter two complexes. These values are only slightly greater than that of 2·599 (1) Å for the Pt—I distance in *trans*-$[PtI_2(PMe_3)_2]$ where overcrowding is less severe (Hitchcock, Jacobson & Pidcock, 1977).

Other distances and angles in the title complex (Table 2) are normal (see Solomun, 1979).

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Table 1. *Atomic fractional coordinates and isotropic displacement parameters* (Å^2)

For non-H atoms U is the mean latent root of the anisotropic displacement tensor. For H atoms it is the assumed isotropic displacement parameter.

	x	*y*	*z*	*U*
Pt	0·00000	0·00000	0·00000	0·033
I	−0·01410 (10)	0·01580 (10)	0·26550 (20)	0·050
P	−0·0428 (3)	0·1573 (4)	−0·0391 (7)	0·038
F(12)	0·1150 (7)	0·1502 (9)	0·2173 (18)	0·064
F(13)	0·1837 (7)	0·2894 (10)	0·4143 (16)	0·060
F(14)	0·1248 (8)	0·4694 (10)	0·3923 (19)	0·075
F(15)	−0·0089 (8)	0·5087 (8)	0·1635 (16)	0·065
F(16)	−0·0833 (7)	0·3682 (9)	−0·0266 (17)	0·054
F(22)	0·0806 (7)	0·2104 (9)	−0·1244 (16)	0·057
F(23)	0·0880 (9)	0·3058 (13)	−0·3628 (19)	0·086
F(24)	−0·0396 (10)	0·3812 (13)	−0·5794 (19)	0·093
F(25)	−0·1717 (9)	0·3442 (11)	−0·5582 (18)	0·073
F(26)	−0·1801 (7)	0·2444 (11)	−0·3242 (16)	0·062
F(32)	−0·1674 (7)	0·0456 (10)	−0·2458 (16)	0·059
F(33)	−0·3027 (7)	0·0125 (12)	−0·2445 (20)	0·086
F(34)	−0·3478 (7)	0·1050 (11)	−0·0399 (19)	0·076
F(35)	−0·2547 (8)	0·2312 (11)	0·1651 (17)	0·078
F(36)	−0·1182 (7)	0·2568 (10)	0·1774 (15)	0·056
C(11)	0·0102 (14)	0·2501 (15)	0·0960 (31)	0·051
C(12)	0·0805 (14)	0·2362 (16)	0·2041 (30)	0·050
C(13)	0·1195 (12)	0·3086 (18)	0·3028 (26)	0·037
C(14)	0·0886 (13)	0·4000 (17)	0·2892 (26)	0·045
C(15)	0·0200 (14)	0·4214 (16)	0·1768 (30)	0·048
C(16)	−0·0172 (11)	0·3469 (17)	0·0831 (23)	0·035
C(21)	−0·0509 (12)	0·2205 (16)	−0·2161 (24)	0·035
C(22)	0·0166 (11)	0·2383 (17)	−0·2385 (25)	0·041
C(23)	0·0209 (16)	0·2915 (20)	−0·3546 (27)	0·056
C(24)	−0·0423 (20)	0·3275 (21)	−0·4615 (30)	0·075
C(25)	−0·1095 (15)	0·3094 (18)	−0·4499 (28)	0·049
C(26)	−0·1123 (14)	0·2558 (19)	−0·3344 (28)	0·048
C(31)	−0·1375 (12)	0·1553 (16)	−0·0402 (28)	0·044
C(32)	−0·1864 (13)	0·0919 (16)	−0·1426 (29)	0·047
C(33)	−0·2590 (13)	0·0743 (19)	−0·1424 (31)	0·055
C(34)	−0·2803 (13)	0·1220 (18)	−0·0390 (30)	0·051
C(35)	−0·2335 (13)	0·1849 (19)	0·0618 (30)	0·055
C(36)	−0·1621 (12)	0·2011 (18)	0·0643 (27)	0·045
Cl(1)	0·1942 (7)	−0·0539 (9)	0·6065 (12)	0·133
Cl(2)	0·2297 (6)	0·1041 (8)	0·8210 (15)	0·144
C	0·1839 (17)	0·0651 (27)	0·6223 (36)	0·101
H(1)	0·20850	0·10740	0·55730	0·050
H(2)	0·12610	0·08560	0·58990	0·050

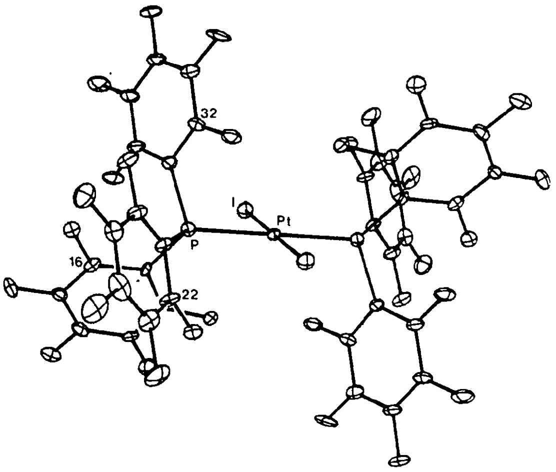

Fig. 1. A perspective view of the molecule showing 20% vibrational ellipsoids. Ring C atoms are numbered serially C(n1)···C(n6) with C(n1) attached to P. F atoms have the same serial number as the C atom to which they are attached.

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* Lists of structure amplitudes, anisotropic displacement parameters and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43228 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† Primed atoms are derived from those in Table 1 by the transformation $\bar{x}, \bar{y}, \bar{z}$.

Table 2. *Selected interatomic distances* (Å) *and angles* (°)

Pt—I	2·626 (2)	Pt—P	2·292 (6)
P—C(11)	1·823 (25)	P—C(21)	1·834 (23)
P—C(31)	1·836 (23)	Cl(1)—C	1·658 (39)
Cl(2)—C	1·828 (35)		

	Range	Mean
C—F	1·309 (26)–1·365 (29)	1·340 (7)
C—C	1·332 (36)–1·431 (34)	1·380 (8)

I—Pt—P	85·1 (2)	Pt—P—C(11)	118·0 (8)
Pt—P—C(21)	119·6 (8)	Pt—P—C(31)	106·8 (8)
C(11)—P—C(21)	98·0 (11)	C(11)—P—C(31)	108·5 (12)
C(21)—P—C(31)	104·9 (11)	P—C(11)—C(12)	124·4 (17)
P—C(11)—C(16)	119·9 (19)	C(12)—C(11)—C(16)	115·4 (21)
P—C(21)—C(22)	116·4 (16)	P—C(21)—C(26)	130·9 (18)
C(22)—C(21)—C(26)	112·7 (20)	P—C(31)—C(32)	116·1 (18)
P—C(31)—C(36)	126·6 (18)	C(32)—C(31)—C(36)	116·7 (21)
Cl(1)—C—Cl(2)	110·6 (19)		

C—C—F angles are in the range 116·1 (21)–123·0 (22)°. C—C—C angles not given above are in the range 117·4 (21)–124·6 (24)°.

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Structure of Disodium D-Fructose 6-Phosphate Heptahydrate

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Abstract. $2Na^+.C_6H_{11}O_9P^{2-}.7H_2O$, $M_r = 430{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}856\,(6)$, $b = 11{\cdot}007\,(6)$, $c = 16{\cdot}361\,(8)$ Å, $V = 1775\,(3)$ Å^3, $Z = 4$, $D_m = 1{\cdot}62$, $D_x = 1{\cdot}61\,(1)$ Mg m^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}29$ mm^{-1}, $F(000) = 904$, $T = 294\,(1)$ K, final $R = 0{\cdot}040$ for 2500 non-zero reflexions. The length of the phosphate ester bond is 1·609 (2) Å. The furanose ring is in the ${}^{2}_{3}T$ conformation. The torsion angles O(6)—C(6)—C(5)—O(5) and O(6)—C(6)—C(5)—C(4) are 65·6 (5) and −174·5 (5)° and the conformation about the C(5)—C(6) bond is therefore *gauche–trans*. The crystal structure is stabilized by a three-dimensional hydrogen-bonding network.

Introduction. Different D-fructose phosphates are known to play important roles in living organisms. However, of four known fructose phosphates: fructose 1,6-diphosphate (F1,6-dp), fructose 6-phosphate (F6P), fructose 1-phosphate (F1P) and fructose 2,6-diphosphate (F2,6-dp) only the crystal structure of trisodium D-fructose 1,6-diphosphate octahydrate has been published (Narendra, Seshadri & Viswamitra, 1985). This paper presents the structure of Na_2(F6P).$7H_2O$. This investigation follows the author's earlier studies on sodium salts of D-glucose 6-phosphate and D-2-phosphoglycerate (Lis, 1985*a,b*).

Experimental. Crystals of disodium D-fructose 6-phosphate heptahydrate were obtained by slow concentration in a refrigerator (~273 K) of an aqueous solution of the commercially available compound (Boehringer). The compound crystallized as colorless multi-faceted, often rounded, crystals. D_m by flotation in 1,2-dibromoethane/CCl_4; preliminary examination by rotation and Weissenberg photographs. An irregular specimen, edge length 0·4 to 0·5 mm, was cut from a large crystal. Syntex $P2_1$ diffractometer, Mo $K\alpha$ radiation for lattice parameters (15 reflexions in the range $18 < 2\theta < 28°$) and intensity measurements; ω–2θ scan technique. 2919 reflexions measured below $\theta = 30°$; h 0→13, k 0→15, l 0→23; after every 50 reflexions two standards measured, variation 5%; most calculations performed on a Nova 1200 computer with locally modified *XTL/XTLE* programs (Syntex, 1976); neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); real and imaginary components of anomalous dispersion included for all non-H atoms. Structure solved by direct methods; H atoms from difference synthesis; refinement based on F values; Na, P, O, C anisotropic, H isotropic; 2500 reflexions with $I > 2\sigma(I)$, $w = 1/\sigma^2(F_o)$ gave $R = 0{\cdot}040$ and $wR = 0{\cdot}038$; max. Δ/σ 0·14. No absorption and extinction corrections. Residual electron

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