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A Monoclinic Crystal Form of Tris(triphenylphosphine)platinum(0)

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Abstract. $[\mathrm{Pt}\{\mathrm{P(C_6H_5)_3}\}_3]$, $M_r = 982{\cdot}0$, monoclinic, $P2_1/c$, $a = 21{\cdot}194$ (2), $b = 12{\cdot}432$ (2), $c = 18{\cdot}556$ (3) Å, $\beta = 112{\cdot}85$ (1)°, $V = 4505{\cdot}4$ Å^3, $Z = 4$, $D_x = 1{\cdot}45$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 32{\cdot}8$ cm^{-1}, $F(000) = 1968$, $T = 295$ K, $R = 0{\cdot}033$ and $wR = 0{\cdot}042$ for 5389 reflections with $|F^2| > \sigma(F^2)$. The geometry at Pt is approximately trigonal planar with Pt—P distances ranging from 2·262 (2) to 2·271 (2) Å and P—Pt—P angles varying between 117·20 (6) and 121·83 (6)°. The structure is quite distinct from that previously determined and is not related to it by any simple transformation.

Introduction. A large crystal of $[\mathrm{Pt(PPh_3)_3}]$ was isolated from an attempted preparation of $[\mathrm{Pt(PPh_3)_2(dppb)}]$ [dppb = 1,4-bis(diphenylphosphino)butane]. The structure of $[\mathrm{Pt(PPh_3)_3}]$ was initially determined more than 20 years ago (Albano, Bellon & Scatturin, 1966), but was apparently never fully refined. There are few literature reports of accurate structure determinations on tris(phosphine)platinum(0) complexes (Immirzi, Musco & Mann, 1977; van der Knaap *et al.*, 1984) so that an accurate determination of the structure of this important complex was of considerable interest.

Experimental. A large crystal of $[\mathrm{Pt(PPh_3)_3}]$ was isolated from a THF solution containing $[\mathrm{Pt(PPh_3)_2(C_2H_4)}]$, and a fragment *ca* 0·4 × 0·4 × 0·3 mm was cut from it. Data, Enraf–Nonius CAD-4 diffractometer, cell parameters on least-squares treatment of 25 reflections with $7 < \theta < 10°$, graphite-monochromated Mo $K\alpha$ radiation, θ–2θ scan mode, $\Delta\theta = (0{\cdot}8 + 0{\cdot}35\tan\theta)°$, maximum scan time 1 min. 8296 unique reflections measured for $2 < \theta < 25°$ and $\pm h + k + l$, and 5389 reflections with $|F^2| > \sigma(F^2)$ used in the refinement, $\sigma(F^2) = [\sigma^2(I) + (0{\cdot}04I)^2]^{1/2}/\mathrm{Lp}$. There was no crystal decay. Absorption correction using *DIFABS* (Walker & Stuart, 1983) after isotropic refinement, maximum and minimum corrections of 1·34 and 0·69. The structure was solved by routine heavy-atom methods and refined by full-matrix least squares with the non-H atoms anisotropic (523 parameters). H atoms were fixed at calculated positions with $B_{\mathrm{iso}} = 1{\cdot}3\ B_{\mathrm{eq}}$ for the C atoms to which they are bonded. The weighting scheme was $w = 1/\sigma^2(F)$ and the final residuals were $R = 0{\cdot}033$ and $wR = 0{\cdot}042$. $S = 1{\cdot}3$, $(\Delta/\sigma)_{\max} = 0{\cdot}01$, $\Delta\rho_{\max} = 0{\cdot}9$, $\Delta\rho_{\min} - 1{\cdot}1$ e Å^{-3}. Programs from the Enraf–Nonius *SDP-Plus* package (Frenz, 1984) were run on a MicroVAX computer. Atomic scattering

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses for non-*H *atoms*

$$B_{\rm eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
Pt	0·24765 (1)	0·15326 (2)	0·23711 (1)	3·205 (4)
P(1)	0·26363 (7)	0·3321 (1)	0·22821 (8)	3·48 (4)
P(2)	0·32950 (7)	0·0318 (1)	0·24486 (8)	3·47 (3)
P(3)	0·14505 (7)	0·0998 (1)	0·23671 (8)	3·41 (3)
C(1)	0·3103 (3)	0·4055 (5)	0·3198 (3)	4·0 (1)
C(2)	0·3035 (3)	0·5156 (5)	0·3269 (3)	5·5 (2)
C(3)	0·3420 (4)	0·5687 (6)	0·3955 (4)	7·0 (2)
C(4)	0·3881 (4)	0·5117 (7)	0·4570 (4)	8·1 (3)
C(5)	0·3965 (4)	0·4022 (7)	0·4513 (4)	6·8 (2)
C(6)	0·3569 (3)	0·3484 (5)	0·3828 (3)	5·1 (2)
C(7)	0·3115 (3)	0·3660 (5)	0·1667 (3)	3·7 (1)
C(8)	0·3516 (3)	0·4562 (6)	0·1770 (3)	5·3 (2)
C(9)	0·3848 (4)	0·4759 (7)	0·1265 (4)	6·8 (2)
C(10)	0·3765 (3)	0·4078 (7)	0·0659 (4)	6·4 (2)
C(11)	0·3378 (4)	0·3170 (6)	0·0567 (4)	6·4 (2)
C(12)	0·3062 (3)	0·2954 (6)	0·1079 (3)	5·4 (2)
C(13)	0·1871 (3)	0·4178 (5)	0·1838 (3)	4·3 (1)
C(14)	0·1443 (3)	0·4299 (6)	0·2244 (4)	5·5 (2)
C(15)	0·0827 (3)	0·4845 (6)	0·1915 (4)	6·2 (2)
C(16)	0·0627 (4)	0·5272 (6)	0·1179 (5)	7·0 (2)
C(17)	0·1041 (4)	0·5167 (6)	0·0768 (4)	6·9 (2)
C(18)	0·1675 (3)	0·4617 (5)	0·1098 (3)	5·1 (2)
C(19)	0·4067 (3)	0·0745 (5)	0·2298 (3)	4·0 (1)
C(20)	0·4365 (3)	0·1724 (6)	0·2610 (4)	5·7 (2)
C(21)	0·4936 (4)	0·2110 (7)	0·2497 (5)	7·2 (2)
C(22)	0·5202 (3)	0·1519 (6)	0·2051 (4)	6·4 (2)
C(23)	0·4927 (3)	0·0556 (7)	0·1737 (4)	6·1 (2)
C(24)	0·4356 (3)	0·0158 (6)	0·1863 (3)	5·2 (2)
C(25)	0·3648 (3)	−0·0434 (5)	0·3373 (3)	3·7 (1)
C(26)	0·3221 (3)	−0·0672 (6)	0·3749 (3)	4·8 (2)
C(27)	0·3456 (4)	−0·1269 (6)	0·4435 (4)	6·0 (2)
C(28)	0·4108 (4)	−0·1625 (6)	0·4758 (4)	6·1 (2)
C(29)	0·4543 (4)	−0·1395 (6)	0·4390 (5)	7·2 (2)
C(30)	0·4315 (3)	−0·0800 (6)	0·3698 (4)	6·0 (2)
C(31)	0·2980 (3)	−0·0755 (5)	0·1716 (3)	4·1 (1)
C(32)	0·3024 (4)	−0·1829 (6)	0·1892 (4)	6·0 (2)
C(33)	0·2734 (4)	−0·2593 (6)	0·1294 (5)	7·7 (2)
C(34)	0·2436 (4)	−0·2276 (7)	0·0537 (4)	7·7 (2)
C(35)	0·2402 (4)	−0·1205 (7)	0·0348 (4)	7·5 (2)
C(36)	0·2662 (4)	−0·0439 (6)	0·0931 (4)	6·2 (2)
C(37)	0·1350 (3)	−0·0402 (5)	0·2638 (3)	3·9 (1)
C(38)	0·1524 (3)	−0·1220 (5)	0·2233 (4)	4·7 (2)
C(39)	0·1505 (4)	−0·2291 (6)	0·2440 (4)	6·1 (2)
C(40)	0·1314 (4)	−0·2541 (6)	0·3053 (5)	6·7 (2)
C(41)	0·1140 (4)	−0·1754 (6)	0·3446 (4)	6·6 (2)
C(42)	0·1157 (3)	−0·0685 (6)	0·3250 (4)	5·7 (2)
C(43)	0·1226 (3)	0·1769 (5)	0·3078 (3)	4·0 (1)
C(44)	0·0572 (3)	0·2077 (6)	0·2961 (4)	5·3 (2)
C(45)	0·0447 (4)	0·2702 (7)	0·3523 (4)	7·2 (2)
C(46)	0·0987 (4)	0·3004 (7)	0·4186 (4)	7·4 (2)
C(47)	0·1639 (4)	0·2716 (8)	0·4307 (4)	8·1 (2)
C(48)	0·1759 (4)	0·2096 (7)	0·3753 (4)	6·6 (2)
C(49)	0·0687 (3)	0·1183 (5)	0·1473 (3)	3·7 (1)
C(50)	0·0660 (3)	0·2059 (6)	0·1020 (4)	5·3 (2)
C(51)	0·0075 (4)	0·2300 (6)	0·0366 (4)	6·0 (2)
C(52)	−0·0476 (4)	0·1667 (6)	0·0148 (4)	6·8 (2)
C(53)	−0·0463 (4)	0·0776 (7)	0·0595 (6)	9·2 (3)
C(54)	0·0125 (4)	0·0542 (7)	0·1260 (5)	7·2 (2)

factors from *International Tables for X-ray Crystallography* (1974). Atomic parameters are given in Table 1,* selected distances and angles in Table 2. Fig. 1 shows the numbering scheme, Fig. 2 the molecular packing.

Table 2. *Selected intramolecular distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Pt—P(1)	2·265 (2)	Pt—P(2)	2·262 (2)
Pt—P(3)	2·271 (2)	P(1)—C(1)	1·842 (5)
P(1)—C(7)	1·846 (7)	P(1)—C(13)	1·846 (6)
P(2)—C(19)	1·842 (6)	P(2)—C(25)	1·838 (5)
P(2)—C(31)	1·835 (6)	P(3)—C(37)	1·847 (6)
P(3)—C(43)	1·835 (7)	P(3)—C(49)	1·827 (5)

P(1)—Pt—P(2)	121·83 (6)	P(1)—Pt—P(3)	117·20 (6)
P(2)—Pt—P(3)	120·97 (6)	Pt—P(1)—C(1)	117·5 (2)
Pt—P(1)—C(7)	113·8 (2)	Pt—P(1)—C(13)	118·0 (2)
C(1)—P(1)—C(7)	103·3 (3)	C(1)—P(1)—C(13)	100·9 (3)
C(7)—P(1)—C(13)	101·0 (3)	Pt—P(2)—C(19)	120·2 (2)
Pt—P(2)—C(25)	115·0 (2)	Pt—P(2)—C(31)	112·9 (2)
C(19)—P(2)—C(25)	103·0 (2)	C(19)—P(2)—C(31)	100·7 (3)
C(25)—P(2)—C(31)	102·8 (3)	Pt—P(3)—C(37)	119·1 (2)
Pt—P(3)—C(43)	111·1 (2)	Pt—P(3)—C(49)	118·4 (2)
C(37)—P(3)—C(43)	102·0 (3)	C(37)—P(3)—C(49)	102·2 (2)
C(43)—P(3)—C(49)	101·6 (3)		

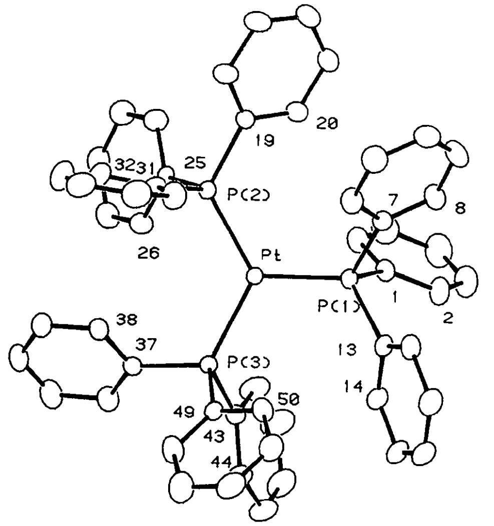

Fig. 1. Molecular structure and numbering scheme for [Pt(PPh$_3$)$_3$]. Atoms not otherwise indicated are C atoms.

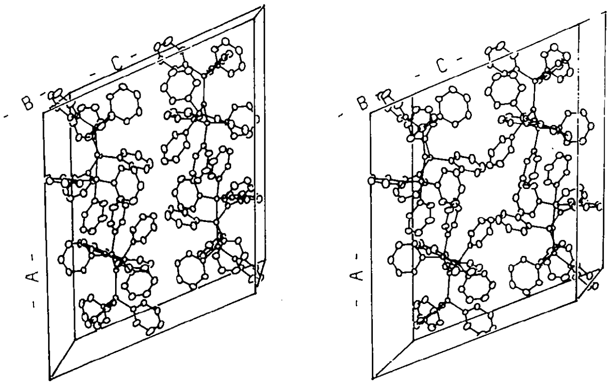

Fig. 2. Stereo packing diagram for [Pt(PPh$_3$)$_3$].

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* Lists of structure factors, anisotropic thermal parameters, least-squares planes, bond distances and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51910 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. The space group found in our structural determination is quite different from that previously established (triclinic, $P\bar{1}$, $a = 12{\cdot}65$, $b = 12{\cdot}25$, $c = 17{\cdot}05$ Å, $\alpha = 76{\cdot}25$, $\beta = 105{\cdot}75$, $\gamma = 119{\cdot}6°$, $Z = 2$, $R = 0{\cdot}102$), and is not related to it by any simple transformation. It is not clear if this observation is related to the unusual circumstances in which our crystal was grown. The solvent from which the crystal was originally grown is not given.

The coordination at Pt is approximately trigonal, with P—Pt—P angles of 117·20 (6), 120·97 (6) and 121·83 (6)°, and the four atoms very close to coplanar. Pt—P distances ranged from 2·262 (2) to 2·271 (2) Å, similar to those previously noted in the other space group. The distances are somewhat shorter than those for bonds to PPh_3 in $[Pt(PPh_3)_2(Ph_2C{=}PAr)]$ [2·288 (3) and 2·294 (3) Å] (van der Knaap *et al.*, 1984), presumably due to steric crowding in that species. Unlike the original structural determination there is no indication of any π–π interaction between pairs of benzene rings, and their orientations seem to be determined entirely with the object of minimizing steric interactions.

The structure may be compared with that of the related complex $[Pt(PCy_3)_3]$ (Immirzi *et al.*, 1977), which was of considerably higher symmetry, with one of the independent molecules having C_3 crystallographic symmetry and the other two being close to C_3 symmetry. The bonds, as might be expected for this more hindered species, were slightly longer, 2·303 (3) Å. In both cases the molecules were close to perfectly planar and ligand repulsions were alleviated by meshing.

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Structure of [*N,N'*-Propylenebis(2-pyrrolylmethyleneaminato)]nickel(II)

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Abstract. $[Ni(C_{13}H_{14}N_4)]$, $M_r = 284{\cdot}98$, monoclinic, $P2_1/c$, $a = 10{\cdot}096\ (2)$, $b = 9{\cdot}360\ (2)$, $c = 30{\cdot}565\ (4)$ Å, $\beta = 119{\cdot}45\ (2)°$, $V = 2515{\cdot}1\ (9)$ Å^3, $Z = 8$, $D_x = 1{\cdot}505$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 20{\cdot}22$ cm^{-1}, $F(000) = 1168$, $T = 298\ (2)$ K, 437 parameters refined, final $R = 0{\cdot}038$ for all 2587 reflections. The complex takes a square-planar geometry around the Ni atom. Although the coordination geometries of the two crystallographically independent complexes are quite similar to each other, significant differences in planarity and Ni—N distances are observed.

Introduction. Synthetic macrocyclic ligands containing pyrrole fragments are of interest as models for the oxygenase, oxidase, and oxygen transport and storage proteins (Spiro, 1980; Sigel, 1981). A binucleating tetrapyrrolic macrocycle has recently been synthesized at this Institution (Acholla & Mertes, 1984), and the structure of the dicopper complex has been reported (Acholla, Takusagawa & Mertes, 1985). In order to compare the chemistry of the binuclear complex to its mononuclear analogue, [*N,N'*-propylenebis(2-pyrrolylmethyleneaminato)]-metal complexes were synthesized and the structure of the nickel(II) complex was determined.

Experimental. The title compound was synthesized by the method described by Weber (1967). A reddish brown crystal, ~0·1 × 0·1 × 0·05 mm, obtained by recrystallization from a 1:1 CH_2Cl_2–C_6H_{12} mixed solution, was mounted on a glass fiber oriented approximately along [10$\bar{2}$]. Cell constants were

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