[1,3-Bis(dicyclohexylphosphino)propane](η²-ethylene)platinum(0)

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Abstract. $[Pt(C_2H_4)(C_{27}H_{50}P_2)]$, $M_r = 659.8$, monoclinic, $P2_1/n$, $a = 9.587$ (4), $b = 9.265$ (2), $c = 16.208$ (5) Å, $\beta = 90.67$ (3)°, $V = 1440$ (1) Å^3, $Z = 2$, $D_x = 1.52$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 50.5$ cm^{-1}, $F(000) = 672$, $T = 294$ K, $R = 0.099$ for 1152 observed reflections. The molecule lies about a crystallographic inversion centre and is disordered. Refinement was by a combination of full-matrix and restrained least-squares processes. The Pt atom has essentially square-planar coordination, with the six-membered PtP_2C_3 ring in an envelope conformation with a C atom [C(2)] at the flap.

Experimental. Small colourless needle crystals grown in *n*-hexane by reacting 1,3-bis(dicyclohexylphosphino)-propaneplatinum(0) with ethylene gas under a pressure of 7 MPa. Data crystal $0.05 \times 0.15 \times 0.50$ mm, data collected using a CAD-4 diffractometer with graphite-monochromatized radiation, $\omega/2\theta$ scans, $4 < 2\theta < 40°$, cell constants and orientation matrix from setting angles of 25 reflections with $10 < \theta < 15°$. Data corrected for Lorentz, polarization and absorption (Gaussian integration with corrections ranging from 0.483 to 0.777); h 0 to 9, k 0 to 8, l −15 to +15. Three reflections measured as standards every 2 h of exposure time, no evidence of crystal decay. 1990 reflections measured (the 0*kl* and 1*kl* nets were measured twice as a check on crystal quality and diffractometer counting chain stability), 1326 unique (*R* factor on averaging 0.014), 1152 observed with $I > 3\sigma(I)$. Space group uniquely from systematic absences (*h0l*, $h + l = 2n +$ 1; 0*k*0, $k = 2n + 1$). With $Z = 2$, the molecule is disordered about an inversion centre, but the analysis revealed that the PCy_2 groups appear to be largely unaffected by the disorder; the Pt, C_2H_4 and $(CH_2)_3$ moieties are distributed over two sites. The Pt and P coordinates were determined from a Patterson synthesis; a subsequent difference synthesis phased with Pt and P contributions clearly revealed the C atoms of the two unique cyclohexyl rings, with chair conformations and with the C atoms bonded to P [C(11) and C(21)] essentially equidistant from the PtP_2 plane. A study of models shows that this conformation (in which neither cyclohexyl is clearly axial nor equatorial) leads to a C(2) envelope conformation for the six-membered PtP_2C_3 ring. Initial refinement by full-matrix least-squares calculations with isotropic thermal parameters was followed by another difference synthesis which revealed a diffuse electron-density cloud with five maxima, to which could be fitted a plausible model for an ethylene ligand coordinated to Pt, and the $-(CH_2)_3-$ group linking the two P atoms in the complex in a six-membered ring with an envelope conformation as anticipated. Because of the serious disorder problem, final refinement (on *F*) was done using a restrained least-squares process with appropriate geometrical constraints and the Pt and P atoms allowed anisotropic motion; no allowance for H atoms. Final $R = 0.099$, $wR = 0.16$, $S = 3.8$; the *R* factor was not unreasonable in view of the disorder. Ratio of maximum shift to e.s.d. in the final cycle was less than 0.1. Weighting scheme of the form $w = 1/[\sigma^2F_o + 0.04F_o^2]$. Density in final difference map ± 1.3 e Å^{-3} near the Pt atom, no chemically significant features. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations done on a PDP11/73 system using the Enraf–Nonius (1982) *SDP-Plus* programs.

Table 1. *Atomic positional and thermal parameters with their e.s.d.'s in parentheses*

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $B_{eq} = \frac{4}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + ab\cos\gamma B_{12} + ac\cos\beta B_{13} + bc\cos\alpha B_{23})$.

	x	y	z	B_{eq}/B_{iso}(Å)
Pt	0.4356 (2)	0.1031 (2)	0.0105 (1)	3.14 (4)
P(1)	0.3459 (9)	−0.0925 (10)	−0.0218 (5)	6.2 (2)
Ce(1)	0.266 (17)	0.248 (15)	0.030 (9)	18 (6)*
Ce(2)	0.392 (14)	0.314 (14)	0.052 (8)	14 (4)*
C(1)	0.479 (9)	−0.234 (8)	−0.039 (5)	7 (2)*
C(2)	0.627 (12)	−0.252 (12)	0.000 (6)	11 (4)*
C(3)	0.705 (6)	−0.107 (7)	−0.008 (4)	4 (2)*
C(11)	0.225 (4)	−0.183 (4)	0.046 (2)	6 (1)*
C(12)	0.286 (4)	−0.186 (4)	0.135 (2)	6 (1)*
C(13)	0.192 (4)	−0.262 (4)	0.198 (2)	6 (1)*
C(14)	0.170 (4)	−0.421 (4)	0.172 (2)	6 (1)*
C(15)	0.112 (4)	−0.430 (4)	0.083 (2)	6 (1)*
C(16)	0.207 (4)	−0.345 (4)	0.024 (2)	7 (1)*
C(21)	0.260 (3)	−0.119 (4)	−0.122 (2)	5 (1)*
C(22)	0.106 (4)	−0.069 (4)	−0.125 (2)	6 (1)*
C(23)	0.037 (4)	−0.103 (4)	−0.209 (2)	8 (1)*
C(24)	0.119 (4)	−0.024 (4)	−0.277 (2)	7 (1)*
C(25)	0.274 (4)	−0.071 (4)	−0.276 (2)	7 (1)*
C(26)	0.340 (4)	−0.035 (4)	−0.190 (2)	7 (1)*

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

Pt—P(1)	2·07 (1)	C(11)—C(16)	1·55 (5)	
Pt—P(1^i)	2·10 (1)	C(12)—C(13)	1·53 (5)	
Pt—Ce(1)	2·14 (1)	C(13)—C(14)	1·54 (5)	
Pt—Ce(2)	2·10 (1)	C(14)—C(15)	1·54 (5)	
P(1)—C(1)	1·86 (8)	C(15)—C(16)	1·54 (5)	
P(1)—C(11)	1·81 (4)	C(21)—C(22)	1·55 (5)	
P(1)—C(21)	1·83 (3)	C(21)—C(26)	1·56 (5)	
P(1^i)—C(3)	1·98 (7)	C(22)—C(23)	1·53 (5)	
Ce(1)—Ce(2)	1·39 (20)	C(23)—C(24)	1·54 (6)	
C(1)—C(2)	1·55 (13)	C(24)—C(25)	1·55 (6)	
C(2)—C(3)	1·55 (12)	C(25)—C(26)	1·55 (5)	
C(11)—C(12)	1·56 (5)			

P(1)—Pt—P(1^i)	113·0 (4)	P(1^i)—C(3)—C(2)	132 (5)
P(1)—Pt—Ce(1)	106 (4)	P(1)—C(11)—C(12)	110 (2)
P(1)—Pt—Ce(2)	144 (4)	P(1)—C(11)—C(16)	112 (3)
P(1^i)—Pt—Ce(1)	141 (4)	C(12)—C(11)—C(16)	104 (3)
P(1^i)—Pt—Ce(2)	102 (4)	C(11)—C(12)—C(13)	114 (3)
Ce(1)—Pt—Ce(2)	38 (5)	C(12)—C(13)—C(14)	110 (3)
Pt—P(1)—C(1)	112 (3)	C(13)—C(14)—C(15)	110 (3)
Pt—P(1)—C(11)	121 (1)	C(14)—C(15)—C(16)	110 (3)
Pt—P(1)—C(21)	121 (1)	C(11)—C(16)—C(15)	115 (3)
C(1)—P(1)—C(11)	102 (3)	P(1)—C(21)—C(22)	114 (2)
C(1)—P(1)—C(21)	95 (3)	P(1)—C(21)—C(26)	110 (2)
C(11)—P(1)—C(21)	101 (2)	C(22)—C(21)—C(26)	108 (3)
Pt—P(1^i)—C(3)	106 (2)	C(21)—C(22)—C(23)	112 (3)
Pt—Ce(1)—Ce(2)	70 (8)	C(22)—C(23)—C(24)	109 (3)
Pt—Ce(2)—Ce(1)	72 (8)	C(23)—C(24)—C(25)	110 (3)
P(1)—C(1)—C(2)	130 (6)	C(24)—C(25)—C(26)	110 (3)
C(1)—C(2)—C(3)	108 (7)	C(21)—C(26)—C(25)	109 (3)

Symmetry code: (i) $1-x, -y, -z$.

The structure is defined by the coordinates and dimensions given in Tables 1 and 2, and Fig. 1.*

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* Lists of structure factors, anisotropic thermal parameters and mean-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43566 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 1. View of the disordered $[Pt(C_2H_4)(C_{27}H_{50}P_2)]$ complex. Ellipsoids are at the 20% level. The asterisk is the inversion centre at ($\frac{1}{4}$00). The atoms shown by small spheres and connected by line bonds are the sites of the inversion related Pt, C_2H_4 and $-(CH_2)_3-$ moieties.

Related literature. The reactivity of the complex described here is discussed by Clark & Hampden-Smith (1986). For details of the related dichloro[1,3-bis-(diphenylphosphino)propane]palladium(II) see Steffen & Palenik (1976).

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Redetermination of the Structure of Tetrakis(acetylacetonato)zirconium(IV)

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Abstract. $[Zr(C_5H_7O_2)_4]$, $M_r = 487·7$, monoclinic, $C2/c$, $a = 21·662$ (2), $b = 8·360$ (1), $c = 14·107$ (1) Å, $\beta = 116·708$ (6)°, $V = 2282·1$ Å^3, $Z = 4$, $D_x = 1·419$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0·71073$ Å, $\mu = 0·51$ mm^{-1}, $F(000) = 1008$, $T = 293$ K, $R = 0·020$ for 1940 unique reflections with $F > 4\sigma(F)$. Zr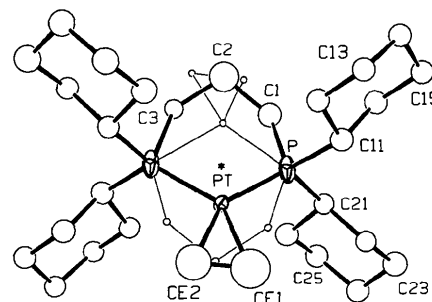, on a crystallographic twofold axis, is coordinated by four chelating ligands. The coordination polyhedron is approximately a square antiprism, with a small distortion towards dodecahedral geometry. The results confirm those of the earlier determination [Silverton & Hoard (1963). *Inorg. Chem.* **2,** 243–249]. Estimated standard deviations of final atomic coordinates and of molecular-geometry parameters are reduced in the