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trans-Dichloro(tricyclohexylphosphino)(triethylphosphino)platinum(II)

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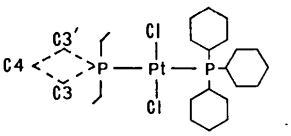
Abstract. $[PtCl_2P(C_2H_5)_3P(C_6H_{11})_3]$, $M_r = 664{\cdot}59$, orthorhombic, *Pcab*, $a = 12{\cdot}284\ (5)$, $b = 19{\cdot}409\ (6)$, $c = 23{\cdot}918\ (6)$ Å, $V = 5702{\cdot}5$ Å^3, $Z = 8$, $D_x = 1{\cdot}548$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 5{\cdot}28$ mm^{-1}, $F(000) = 2672$, $T = 291$ K, final $R = 0{\cdot}029$ for 1988 unique observed reflections with $I > 3\sigma(I)$. The Pt atom has square-planar coordination, with the P—Pt—C angle involving the $P(Et)_3$ ligand distorted due to the bulk of the tricyclohexylphosphine ligand. The phosphine ligands are oriented *trans* across the square plane of coordination. One of the methylene carbon atoms of the triethylphosphine is disordered, with each site having 50% population. Selected metrical details are ⟨Pt—Cl⟩ $= 2{\cdot}304\ (1)$, ⟨Pt—P⟩ $= 2{\cdot}333\ (42)$, and ⟨C—C⟩ $= 1{\cdot}523\ (12)$ Å.

Experimental. Crystals of title compound (I) obtained from Dr H. C. Clark of the University of Guelph; colorless rectangular-shaped crystal, $0{\cdot}19 \times 0{\cdot}28 \times 0{\cdot}47$ mm, glass fiber mount, Enraf–Nonius CAD-4F four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; unit-cell dimensions and systematic absences $0kl$, $l = 2n + 1$, $h0l$, $h = 2n + 1$, $hk0$, $k = 2n + 1$; cell constants from setting angles of 25 reflections ($4{\cdot}0 \le \theta \le 9{\cdot}8°$); correction for Lorentz, polarization effects, empirical absorption correction (North, Phillips & Mathews, 1968), t(min)–t(max) $= 0{\cdot}506$–$0{\cdot}997$; 291 K; intensity data collected by ω–2θ scan technique, variable scan rate of 0·38–3·35°min^{-1}, intensities of two check reflections (464 and 373) measured every 2 h revealed only random deviations (< 1%) from mean intensities; 3539 unique reflections collected ($3 < 2\theta < 45°$, h: 0→13, k: 0→20, l: 0→25), 1988 reflections with $I > 3\sigma(I)$ used in solution and refinement of the structure using *SDP/VAX* (Enraf–Nonius, 1983); Pt atom located by *MULTAN*11/82 (Main, 1982), remainder of non-hydrogen atoms by difference Fourier technique; H atoms calculated and included at fixed positions with $B = 5{\cdot}0$ Å^2; full-matrix least-squares refinement on F yielded $R = 0{\cdot}0289$, $wR = 0{\cdot}0278$, $w = \sigma^{-2}(F_o)$ with $\sigma(F_o) = \{I_p + I_b + [0{\cdot}03(I_p - I_b)]^2\}^{1/2}$, where I_p and I_b are the peak and background counts respectively, $S = 0{\cdot}928$, maximum shift-to-e.s.d. ratio of a parameter in final least-squares cycle was 0·006; maximum positive and minimum negative densities on final difference Fourier map 1·4 (1) and 0·6 (1) e Å^{-3} in vicinity of Pt atom; scattering factors and anomalous-dispersion terms taken from *International Tables for X-ray Crystallography* (1974).



(I)

The final atomic parameters are given in Table 1,† with interatomic distances and angles presented in

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† Lists of anisotropic thermal parameters, H-atom coordinates, dihedral angles, bond lengths and angles, structure factors, and illustrations of the ligand profile about the phosphine P atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43499 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. Fig. 1 presents a stereodrawing of the title compound; a view of the disordered triethylphosphine is shown in Fig. 2.

Related literature. The shape of the tricyclohexylphosphine ligand is very similar to those found in previous structures (Mullica, Oliver, Sappenfield & Grossie, 1986).

Table 1. *Atomic positional parameters* ($\times 10^4$, Pt $\times 10^5$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$, Pt $\times 10^4$)

	x	*y*	*z*	U_{eq} *
Pt	38550 (3)	6651 (2)	12704 (2)	433 (1)
Cl(1)	2517 (2)	1380 (2)	924 (2)	67 (1)
Cl(2)	5126 (3)	−72 (2)	1648 (2)	75 (1)
P1	2528 (3)	10 (2)	1697 (2)	57 (1)
P2	5232 (2)	1406 (2)	935 (1)	43 (1)
C1	1212 (11)	18 (6)	1376 (6)	98 (5)
C2	324 (11)	−412 (6)	1634 (7)	103 (6)
C3†	2630 (17)	57 (10)	2467 (10)	43 (7)
C3*B*†	1992 (20)	581 (13)	2282 (12)	88 (10)
C4	2690 (14)	747 (9)	2686 (6)	131 (7)
C5	2817 (11)	−893 (7)	1769 (7)	150 (6)
C6	2826 (14)	−1246 (8)	1252 (13)	286 (14)
Ring *A*				
C7	6561 (8)	958 (5)	833 (4)	40 (3)
C8	6533 (9)	376 (6)	409 (5)	68 (4)
C9	7569 (10)	−58 (6)	458 (6)	73 (5)
C10	8581 (9)	364 (6)	392 (5)	67 (4)
C11	8585 (8)	981 (6)	788 (5)	55 (4)
C12	7558 (8)	1417 (6)	746 (5)	53 (4)
Ring *B*				
C13	5488 (9)	2082 (5)	1468 (4)	44 (3)
C14	5999 (9)	1782 (5)	2004 (4)	56 (4)
C15	6228 (12)	2373 (6)	2424 (5)	72 (4)
C16	5175 (11)	2748 (6)	2573 (5)	68 (4)
C17	4668 (9)	3028 (6)	2040 (5)	59 (4)
C18	4455 (8)	2466 (5)	1618 (5)	48 (3)
Ring *C*				
C19	4822 (8)	1910 (5)	310 (4)	43 (3)
C20	4486 (10)	1440 (6)	−171 (5)	72 (5)
C21	3970 (12)	1871 (7)	−637 (5)	89 (5)
C22	4718 (12)	2437 (7)	−833 (5)	88 (5)
C23	5090 (11)	2889 (6)	−355 (5)	73 (5)
C24	5615 (9)	2466 (6)	112 (5)	56 (4)

* Equivalent isotropic thermal parameter (U_{eq}) defined as $\frac{1}{3}$ the trace of the orthogonalized U_{ij} tensor.

† C3 and C3*B* at 50% population.

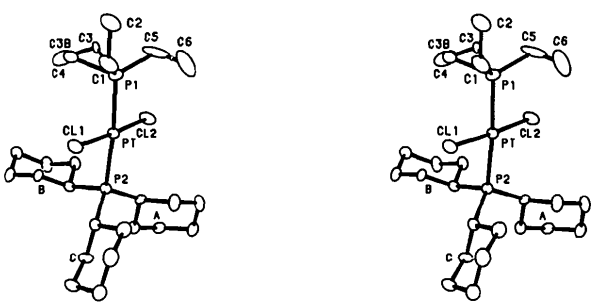

Fig. 1. Stereodrawing of the title compound displaying ellipsoids of 20% thermal probability. Hydrogen atoms are omitted for clarity. The cyclic organic substituents are labelled *A* (C7–C12), *B* (C13–C18) and *C* (C19–C24).

Table 2. *Bond lengths* (Å) *and angles* (°)

Pt–Cl(1)	2·305 (3)	C1–P1–C3	119·2 (8)
Pt–Cl(2)	2·303 (3)	C1–P1–C3*B*	89·9 (9)
Pt–P1	2·305 (3)	C1–P1–C5	103·2 (6)
Pt–P2	2·362 (3)	C3–P1–C3*B*	42·4 (10)
P1–C1	1·789 (14)	C3–P1–C5	86·5 (9)
P1–C3	1·848 (24)	C3*B*–P1–C5	124·4 (10)
P1–C3*B*	1·904 (27)	Pt–P2–C7	112·8 (3)
P1–C5	1·797 (14)	Pt–P2–C13	108·5 (3)
P2–C7	1·865 (10)	Pt–P2–C19	113·6 (3)
P2–C13	1·856 (10)	C7–P2–C13	105·7 (5)
P2–C19	1·856 (11)	C7–P2–C19	112·2 (5)
C1–C2	1·505 (19)	C13–P2–C19	103·2 (5)
C3–C3*B*	1·358 (32)	P1–C1–C2	118·3 (10)
C3–C4	1·441 (27)	P1–C3–C3*B*	71·0 (16)
C3*B*–C4	1·330 (31)	P1–C3–C4	114·3 (14)
C5–C6	1·412 (32)	C3*B*–C3–C4	56·7 (15)
		P1–C3*B*–C3	66·6 (15)
		P1–C3*B*–C4	116·9 (17)
Cl(1)–Pt–Cl(2)	177·0 (1)	C3–C3*B*–C4	64·8 (17)
Cl(1)–Pt–P1	89·2 (1)	C3–C4–C3*B*	58·5 (15)
Cl(1)–Pt–P2	91·3 (1)	P1–C5–C6	113·0 (13)
Cl(2)–Pt–P1	87·9 (1)	P2–C7–C8	114·5 (7)
Cl(2)–Pt–P2	91·5 (1)	P2–C7–C12	116·6 (7)
P1–Pt–P2	173·3 (1)	P2–C13–C14	112·0 (7)
Pt–P1–C1	116·4 (5)	P2–C13–C18	111·7 (7)
Pt–P1–C3	111·4 (7)	P2–C19–C20	111·5 (7)
Pt–P1–C3*B*	104·4 (8)	P2–C19–C24	116·7 (7)
Pt–P1–C5	116·1 (5)		

		Distances (Å)		Angles (°)	
Ring		Mean	Range	Mean	Range
A	C7–C12	1·521 (11)	1·497–1·530	111·2 (13)	109·4–113·1
B	C13–C18	1·527 (14)	1·508–1·550	110·3 (12)	108·7–112·0
C	C19–C24	1·522 (9)	1·506–1·529	111·0 (10)	109·6–111·9

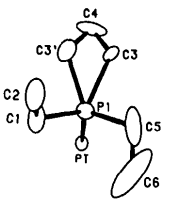

Fig. 2. The disordered triethylphosphine ligand viewed approximately down the P–Pt bond. Hydrogen atoms and the tricyclohexylphosphine ligand are omitted for clarity.

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