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Dichloro-*trans*-bis[tris(2-cyanoethyl)phosphine]-palladium(II)

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The title compound, dichloro-*trans*-bis[tris(2-cyanoethyl)-phosphine]palladium(II), $[PdCl_2(C_9H_{12}N_3P)_2]$, was obtained by the addition of tris(2-cyanoethyl)phosphine to dichloro-*trans*-bis[acetonitrile]palladium(II). The Pd atom lies on a centre of symmetry and the compound is isomorphous with the platinum analogue.

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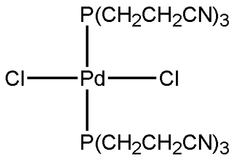
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.027
wR factor = 0.076
Data-to-parameter ratio = 17.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The use of tris(2-cyanoethyl)phosphine (CEP) as a ligand has attracted considerable interest since it has structural similarity to *n*-alkylphosphines and yet possesses different electronic properties (Cotton *et al.*, 1981). CEP has been utilized in the preparation of many metal complexes, including $[HgCl_2(CEP)]_n$ (Bell *et al.*, 1984) and $Ni_4(CO)_6(CEP)_4$ (Bennett *et al.*, 1967). The simple dichloro-*trans*-bis(CEP)platinum(II) complex is known (Khan *et al.*, 1993), and as part of our continuing studies on transition metal CEP complexes, we decided to examine the palladium analogue dichloro-*trans*-bis[tris(2-cyanoethyl)phosphine]palladium(II), (I).



$$Cl-\underset{P(CH_2CH_2CN)_3}{\overset{P(CH_2CH_2CN)_3}{Pd}}-Cl$$

(I)

The title compound lies with the palladium on a centre of symmetry (Fig. 1) and is isomorphous with the platinum analogue. All molecular geometry parameters lie within the normal ranges (Allen *et al.*, 1987; Orpen *et al.*, 1989).

Experimental

Compound (I) was synthesized by the reaction of $P(CH_2CH_2CN)_3$ with $[PdCl_2(CH_3CN)_2]$ generated *in situ* by heating $PdCl_2$ with CH_3CN under reflux for 1.5 h. Two equivalents of $P(CH_2CH_2CN)_3$ were added to the hot CH_3CN solution of $[PdCl_2(CH_3CN)_2]$, causing an immediate colour change from orange to yellow. The reaction was heated under reflux for a further hour and filtered. The solvent was removed *in vacuo* from the filtrate and the sample crystallized from CH_3CN to give an 84% yield of (I) as a yellow crystalline solid. Crystals suitable for X-ray diffraction were obtained by slow evaporation from a CH_3CN solution.

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Crystal data

$[PdCl_2(C_9H_{12}N_3P)_2]$
$M_r = 563.67$
Orthorhombic, $Pbca$
$a = 9.1308\ (11)$ Å
$b = 14.0077\ (19)$ Å
$c = 19.012\ (3)$ Å
$V = 2431.7\ (6)$ Å^3
$Z = 4$
$D_x = 1.540$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 reflections
$\theta = 15.3$–$22.4°$
$\mu = 1.13$ mm^{-1}
$T = 293\ (2)$ K
Block, yellow
$0.52 \times 0.43 \times 0.40$ mm

Data collection

Nonius MACH-3 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.966$, $T_{max} = 1.000$
2665 measured reflections
2256 independent reflections
1853 reflections with $I > 2\sigma(I)$
$R_{int} = 0.012$
$\theta_{max} = 25.5°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 16$
$l = -22 \rightarrow 22$
3 standard reflections frequency: 60 min intensity decay: 0.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.076$
$S = 1.19$
2256 reflections
133 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 3.6835P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

All H atoms were clearly defined in difference maps and were then treated as riding atoms with a C—H distance 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. Examination of the structure with *PLATON* (Spek, 2000) showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: *CAD*-4 *Data Collection Software* (Nonius, 1998); cell refinement: *CAD*-4 *Data Collection Software*; data reduction: *maXus* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELXL*97 and *PLATON*.

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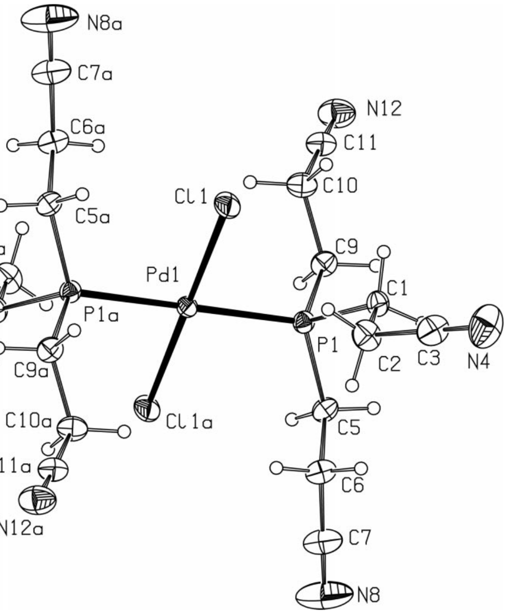

Figure 1
A view of the structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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