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Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.019
 wR factor = 0.052
Data-to-parameter ratio = 20.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.*trans*-Chloromethyldipyridinepalladium(II)

The title compound, $[\text{Pd}(\text{CH}_3)\text{Cl}(\text{C}_5\text{H}_5\text{N})_2]$, has been synthesized by the reaction of $[\text{PdMeCl}(\text{COD})]$ (COD is 1,5-cyclooctadiene) with pyridine in dichloromethane; it is square-planar. The crystal structure features dipole–dipole and π stacking interactions.

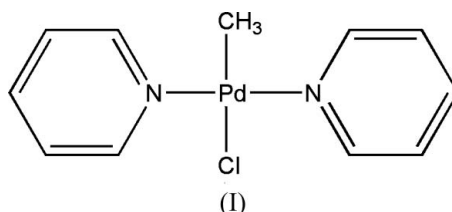
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Comment

trans- $[\text{Pd}(\text{pyridine})_2(\text{Me})\text{Cl}]$, (I), was prepared by addition of an excess of pyridine to $[\text{PdMeCl}(\text{COD})]$ (COD is 1,5-cyclooctadiene). Fig. 1 shows the molecular geometry in the crystal structure and the atom-labelling scheme. The crystal structure comprises ordered individual square-planar molecules of *trans*- $[\text{Pd}(\text{pyridine})_2(\text{Me})\text{Cl}]$ in a general position; the torsion angles $\text{C}2-\text{N}1-\text{Pd}1-\text{C}1$ and $\text{C}7-\text{N}2-\text{Pd}1-\text{C}1$ are $60.31(13)$ and $54.71(13)^\circ$, respectively. The angle between the planes of the pyridine rings is $67.33(5)^\circ$.



Selected geometric parameters are given in Table 1. The bond lengths are in the usual range for $\text{Pd}^{\text{II}}-\text{C}, \text{Cl}, \text{N}$ (Allen *et al.*, 1987). The molecules in the crystal structure are packed in pairs with a $\text{Pd} \cdots \text{Pd}$ distance of $3.7731(3)$ Å. In these pairs, the methyl ligand sits above the chloride ligand and *vice versa* in each case, which may reflect a dipole–dipole interaction between the two molecules (see Fig. 2). Some π stacking [interplanar distance of $3.403(3)$ Å] occurs between the

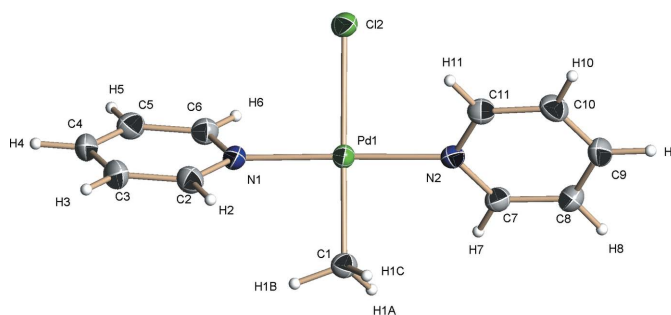


Figure 1

The molecular structure with the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level, with H atoms represented as spheres of arbitrary size.

pyridine rings in adjacent pairs (see Fig. 3), with the rings offset by about one ring width.

The crystal structure is not isostructural with either $[M(\text{pyridine})_2\text{Cl}_2]$ where $M = \text{Pd}$ (Viossat *et al.*, 1993) or Pt (Colamarino & Orioli, 1975).

Experimental

A round-bottomed flask was charged with $[\text{PdMeCl}(\text{COD})]$ (0.100 g, 0.378 mmol) and CH_2Cl_2 (20 ml). Pyridine (0.15 ml, 1.833 mmol) was added to the solution and the mixture was stirred for 1 h. Hexane (50 ml) was added to the mixture and the volume was reduced to ca 20 ml. The resulting white solid was isolated by filtration, washed with two portions of diethyl ether (2×20 ml) and dried under vacuum to give a white solid (1.030 g, 0.327 mmol, 87%). A pale-yellow crystal of irregular shape was selected. IR (cm^{-1} , powder film): 1603 (s, pyridine). ^1H NMR (CDCl_3): δ 8.80 (m, 4H, *o*-pyridine), 7.69 (m, 2H, *p*-pyridine), 7.27 (m, 4H, *m*-pyridine), 0.73 (s, 3H, Pd— CH_3).

Crystal data

$[\text{Pd}(\text{CH}_3)\text{Cl}(\text{C}_5\text{H}_5\text{N})_2]$	$D_x = 1.727 \text{ Mg m}^{-3}$
$M_r = 315.08$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 157 reflections
$a = 13.2867$ (9) Å	$\theta = 2.4\text{--}27.5^\circ$
$b = 11.9185$ (8) Å	$\mu = 1.72 \text{ mm}^{-1}$
$c = 16.2352$ (10) Å	$T = 173$ (2) K
$\beta = 109.5030$ (10)°	Irregular block, pale yellow
$V = 2423.5$ (3) Å ³	$0.40 \times 0.30 \times 0.20 \text{ mm}$
$Z = 8$	

Data collection

Bruker SMART CCD area-detector diffractometer	2785 independent reflections
φ and ω scans	2555 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$R_{\text{int}} = 0.030$
$T_{\text{min}} = 0.583$, $T_{\text{max}} = 0.710$	$\theta_{\text{max}} = 27.5^\circ$
12619 measured reflections	$h = -17 \rightarrow 16$
	$k = -15 \rightarrow 15$
	$l = -19 \rightarrow 21$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.019$	$w = 1/[\sigma^2(F_o^2) + (0.0245P)^2]$
$wR(F^2) = 0.052$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.29$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2785 reflections	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
137 parameters	$\Delta\rho_{\text{min}} = -0.74 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Pd1—N1	2.0441 (13)	Pd1—N2	2.0484 (13)
Pd1—C1	2.0457 (17)	Pd1—Cl2	2.4612 (4)
N1—Pd1—C1	90.58 (6)	N1—Pd1—Cl2	90.15 (4)
N1—Pd1—N2	177.92 (5)	C1—Pd1—Cl2	178.55 (5)
C1—Pd1—N2	88.24 (6)	N2—Pd1—Cl2	90.99 (4)
Cl2—Pd1—N1—C6	56.97 (12)	C1—Pd1—N1—C2	60.31 (13)

H atoms were treated as riding, with C—H distances of 0.95 and 0.98 Å and with $U_{\text{iso}}(\text{H})$ values of 1.2 and 1.5 times $U_{\text{eq}}(\text{C})$ for aromatic and methyl H atoms, respectively.

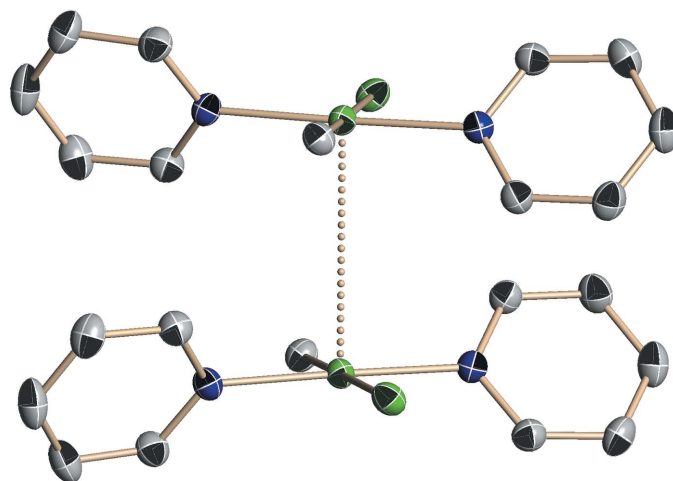


Figure 2

Pair of molecules within the crystal structure. H atoms have been omitted for clarity.

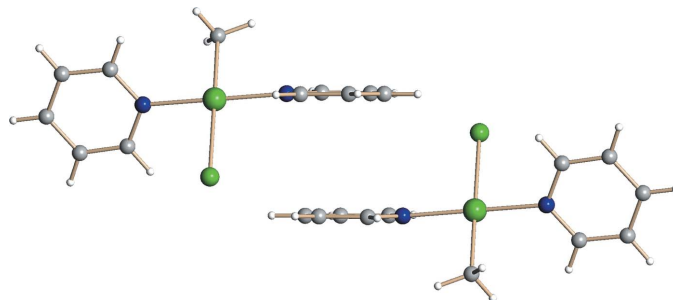


Figure 3

The π stacking in the crystal structure.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bruker (1998). *SMART* (Version 5.054) and *SAINT* (Version 6.0). Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
- Colamarino, P. & Orioli, P. L. (1975). *J. Chem. Soc. Dalton Trans.* pp. 1656–1659.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
- Viossat, B., Dung, N.-H. & Robert, F. (1993). *Acta Cryst. C49*, 84–85.