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# *trans*-Chloromethyldipyridinepalladium(II)

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**Key indicators**

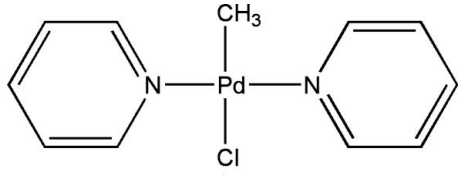
Single-crystal X-ray study
$T$ = 173 K
Mean $\sigma$(C–C) = 0.002 Å
$R$ factor = 0.019
$wR$ factor = 0.052
Data-to-parameter ratio = 20.3

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

The title compound, $[Pd(CH_3)Cl(C_5H_5N)_2]$, has been synthesized by the reaction of [PdMeCl(COD)] (COD is 1,5-cyclooctadiene) with pyridine in dichloromethane; it is square-planar. The crystal structure features dipole–dipole and $\pi$ stacking interactions.

## Comment

*trans*-[Pd(pyridine)$_2$(Me)Cl], (I), was prepared by addition of an excess of pyridine to [PdMeCl(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*-[Pd(pyridine)$_2$(Me)Cl] in a general position; the torsion angles C2—N1—Pd1—C1 and C7—N2—Pd1—C1 are 60.31 (13) and 54.71 (13)°, respectively. The angle between the planes of the pyridine rings is 67.33 (5)°.




(I)

Selected geometric parameters are given in Table 1. The bond lengths are in the usual range for $Pd^{II}$—C,Cl,N (Allen *et al.*, 1987). The molecules in the crystal structure are packed in pairs with a Pd···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 $\pi$ stacking [interplanar distance of 3.403 (3) Å] occurs between the

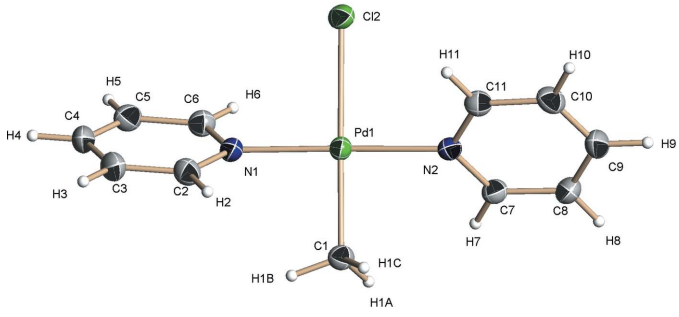



**Figure 1**
The molecular structure with the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level, with H atoms represented as spheres of arbitrary size.

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 $[M(\text{pyridine})_2Cl_2]$ where $M$ = Pd (Viossat *et al.*, 1993) or Pt (Colamarino & Orioli, 1975).

## Experimental

A round-bottomed flask was charged with [PdMeCl(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). $^1$H NMR ($CDCl_3$): $\delta$ 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*

| | |
|---|---|
| $[Pd(CH_3)Cl(C_5H_5N)_2]$ | $D_x$ = 1.727 Mg m$^{-3}$ |
| $M_r$ = 315.08 | Mo $K\alpha$ radiation |
| Monoclinic, $C2/c$ | Cell parameters from 157 reflections |
| $a$ = 13.2867 (9) Å | $\theta$ = 2.4–27.5° |
| $b$ = 11.9185 (8) Å | $\mu$ = 1.72 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 × 0.30 × 0.20 mm |
| $Z$ = 8 | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 2785 independent reflections |
| $\varphi$ and $\omega$ scans | 2555 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003) | $R_{int}$ = 0.030 |
| $T_{min}$ = 0.583, $T_{max}$ = 0.710 | $\theta_{max}$ = 27.5° |
| 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 | $w = 1/[\sigma^2(F_o^2) + (0.0245P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.052 | $(\Delta/\sigma)_{max}$ = 0.001 |
| $S$ = 1.29 | $\Delta\rho_{max}$ = 0.32 e Å$^{-3}$ |
| 2785 reflections | $\Delta\rho_{min}$ = −0.74 e Å$^{-3}$ |
| 137 parameters | |

**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 0.98Å and with $U_{iso}$(H) values of 1.2 and 1.5 times $U_{eq}$(C) for aromatic and methyl H atoms, respectively.

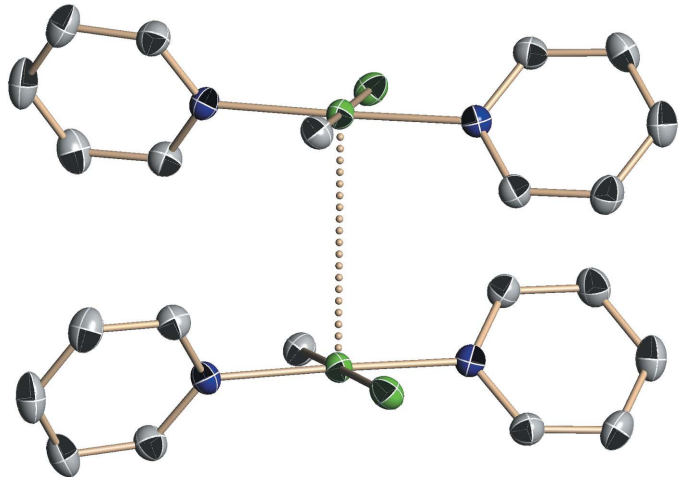


**Figure 2**
Pair of molecules within the crystal structure. H atoms have been omitted for clarity.

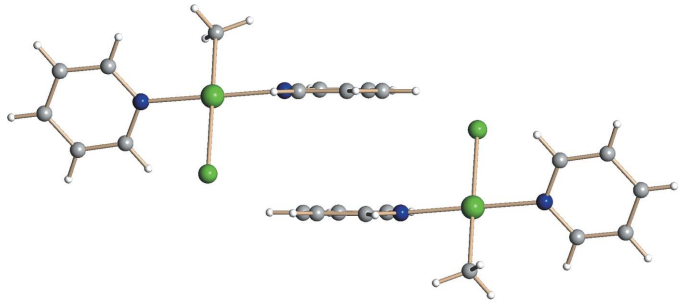


**Figure 3**
The $\pi$ stacking in the crystal structure.

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*.

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# supporting information

*Acta Cryst.* (2005). E**61**, m2651–m2652 [https://doi.org/10.1107/S1600536805037268]

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*Crystal data*

$[Pd(CH_3)Cl(C_5H_5N)_2]$
$M_r = 315.08$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 13.2867\ (9)$ Å
$b = 11.9185\ (8)$ Å
$c = 16.2352\ (10)$ Å
$\beta = 109.503\ (1)°$
$V = 2423.5\ (3)$ Å$^3$
$Z = 8$
$F(000) = 1248$
$D_x = 1.727$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 157 reflections
$\theta = 2.4–27.5°$
$\mu = 1.72$ mm$^{-1}$
$T = 173$ K
Irregular block, pale yellow
0.40 × 0.30 × 0.20 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.2 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.583$, $T_{max} = 0.710$
12619 measured reflections
2785 independent reflections
2555 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -17\rightarrow16$
$k = -15\rightarrow15$
$l = -19\rightarrow21$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.052$
$S = 1.29$
2785 reflections
137 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0245P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.74$ e Å$^{-3}$

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Pd1 | 0.062581 (9) | 0.008712 (9) | 0.372793 (8) | 0.01997 (6) |
| Cl2 | 0.23773 (3) | −0.04033 (3) | 0.36210 (3) | 0.02563 (10) |
| N1 | 0.10037 (11) | 0.17528 (11) | 0.37603 (8) | 0.0223 (3) |
| N2 | 0.02377 (11) | −0.15749 (10) | 0.37380 (8) | 0.0217 (3) |
| C1 | −0.08206 (13) | 0.04720 (14) | 0.38455 (10) | 0.0246 (3) |
| H1A | −0.1394 | 0.0220 | 0.3322 | 0.037* |
| H1B | −0.0872 | 0.1286 | 0.3910 | 0.037* |
| H1C | −0.0888 | 0.0095 | 0.4361 | 0.037* |
| C2 | 0.10611 (14) | 0.24128 (14) | 0.44480 (11) | 0.0278 (4) |
| H2 | 0.0854 | 0.2113 | 0.4909 | 0.033* |
| C3 | 0.14110 (14) | 0.35124 (14) | 0.45058 (13) | 0.0339 (4) |
| H3 | 0.1460 | 0.3953 | 0.5005 | 0.041* |
| C4 | 0.16875 (15) | 0.39606 (14) | 0.38265 (14) | 0.0365 (4) |
| H4 | 0.1926 | 0.4715 | 0.3852 | 0.044* |
| C5 | 0.16141 (14) | 0.33025 (14) | 0.31124 (13) | 0.0330 (4) |
| H5 | 0.1791 | 0.3598 | 0.2635 | 0.040* |
| C6 | 0.12756 (13) | 0.21961 (13) | 0.31028 (11) | 0.0271 (4) |
| H6 | 0.1235 | 0.1738 | 0.2614 | 0.032* |
| C7 | −0.06616 (13) | −0.20216 (13) | 0.31811 (11) | 0.0249 (3) |
| H7 | −0.1147 | −0.1547 | 0.2764 | 0.030* |
| C8 | −0.09049 (14) | −0.31500 (13) | 0.31948 (12) | 0.0301 (4) |
| H8 | −0.1546 | −0.3443 | 0.2792 | 0.036* |
| C9 | −0.01998 (15) | −0.38495 (14) | 0.38043 (12) | 0.0332 (4) |
| H9 | −0.0351 | −0.4626 | 0.3826 | 0.040* |
| C10 | 0.07253 (14) | −0.33933 (14) | 0.43770 (12) | 0.0311 (4) |
| H10 | 0.1219 | −0.3852 | 0.4802 | 0.037* |
| C11 | 0.09239 (14) | −0.22639 (14) | 0.43250 (11) | 0.0260 (4) |
| H11 | 0.1566 | −0.1958 | 0.4716 | 0.031* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Pd1 | 0.02057 (9) | 0.01871 (8) | 0.02094 (9) | −0.00136 (4) | 0.00736 (6) | −0.00087 (4) |
| Cl2 | 0.0216 (2) | 0.0274 (2) | 0.0272 (2) | −0.00200 (15) | 0.00724 (16) | −0.00500 (15) |
| N1 | 0.0211 (7) | 0.0210 (6) | 0.0253 (7) | −0.0023 (5) | 0.0087 (6) | −0.0027 (5) |
| N2 | 0.0200 (7) | 0.0218 (6) | 0.0243 (7) | 0.0001 (5) | 0.0086 (5) | 0.0008 (5) |
| C1 | 0.0240 (8) | 0.0251 (8) | 0.0260 (8) | 0.0010 (6) | 0.0100 (7) | 0.0030 (6) |
| C2 | 0.0301 (9) | 0.0269 (8) | 0.0281 (9) | 0.0009 (7) | 0.0120 (7) | −0.0032 (7) |
| C3 | 0.0327 (10) | 0.0262 (8) | 0.0441 (11) | −0.0033 (7) | 0.0143 (8) | −0.0117 (7) |
| C4 | 0.0312 (10) | 0.0202 (8) | 0.0626 (13) | −0.0029 (7) | 0.0215 (9) | −0.0034 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C5 | 0.0313 (10) | 0.0287 (8) | 0.0452 (11) | 0.0007 (7) | 0.0213 (8) | 0.0063 (7) |
| C6 | 0.0271 (9) | 0.0278 (8) | 0.0280 (9) | −0.0005 (7) | 0.0114 (7) | −0.0007 (7) |
| C7 | 0.0215 (8) | 0.0251 (8) | 0.0274 (8) | −0.0002 (6) | 0.0075 (7) | 0.0020 (6) |
| C8 | 0.0240 (9) | 0.0272 (8) | 0.0382 (10) | −0.0047 (7) | 0.0093 (8) | −0.0035 (7) |
| C9 | 0.0336 (10) | 0.0221 (8) | 0.0481 (11) | −0.0015 (7) | 0.0192 (8) | 0.0015 (7) |
| C10 | 0.0310 (10) | 0.0265 (8) | 0.0368 (10) | 0.0074 (7) | 0.0125 (8) | 0.0079 (7) |
| C11 | 0.0245 (9) | 0.0280 (8) | 0.0251 (8) | 0.0011 (6) | 0.0077 (7) | 0.0006 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Pd1—N1 | 2.0441 (13) | C4—C5 | 1.376 (3) |
| Pd1—C1 | 2.0457 (17) | C4—H4 | 0.9500 |
| Pd1—N2 | 2.0484 (13) | C5—C6 | 1.392 (2) |
| Pd1—Cl2 | 2.4612 (4) | C5—H5 | 0.9500 |
| N1—C6 | 1.344 (2) | C6—H6 | 0.9500 |
| N1—C2 | 1.347 (2) | C7—C8 | 1.385 (2) |
| N2—C7 | 1.344 (2) | C7—H7 | 0.9500 |
| N2—C11 | 1.354 (2) | C8—C9 | 1.390 (2) |
| C1—H1A | 0.9800 | C8—H8 | 0.9500 |
| C1—H1B | 0.9800 | C9—C10 | 1.381 (2) |
| C1—H1C | 0.9800 | C9—H9 | 0.9500 |
| C2—C3 | 1.383 (2) | C10—C11 | 1.380 (2) |
| C2—H2 | 0.9500 | C10—H10 | 0.9500 |
| C3—C4 | 1.381 (3) | C11—H11 | 0.9500 |
| C3—H3 | 0.9500 | | |
| | | | |
| N1—Pd1—C1 | 90.58 (6) | C5—C4—C3 | 119.31 (16) |
| N1—Pd1—N2 | 177.92 (5) | C5—C4—H4 | 120.3 |
| C1—Pd1—N2 | 88.24 (6) | C3—C4—H4 | 120.3 |
| N1—Pd1—Cl2 | 90.15 (4) | C4—C5—C6 | 118.77 (17) |
| C1—Pd1—Cl2 | 178.55 (5) | C4—C5—H5 | 120.6 |
| N2—Pd1—Cl2 | 90.99 (4) | C6—C5—H5 | 120.6 |
| C6—N1—C2 | 118.22 (14) | N1—C6—C5 | 122.36 (16) |
| C6—N1—Pd1 | 119.36 (10) | N1—C6—H6 | 118.8 |
| C2—N1—Pd1 | 122.27 (11) | C5—C6—H6 | 118.8 |
| C7—N2—C11 | 118.14 (14) | N2—C7—C8 | 122.24 (15) |
| C7—N2—Pd1 | 123.18 (10) | N2—C7—H7 | 118.9 |
| C11—N2—Pd1 | 118.68 (11) | C8—C7—H7 | 118.9 |
| Pd1—C1—H1A | 109.5 | C7—C8—C9 | 119.23 (16) |
| Pd1—C1—H1B | 109.5 | C7—C8—H8 | 120.4 |
| H1A—C1—H1B | 109.5 | C9—C8—H8 | 120.4 |
| Pd1—C1—H1C | 109.5 | C10—C9—C8 | 118.67 (15) |
| H1A—C1—H1C | 109.5 | C10—C9—H9 | 120.7 |
| H1B—C1—H1C | 109.5 | C8—C9—H9 | 120.7 |
| N1—C2—C3 | 122.29 (17) | C11—C10—C9 | 119.22 (16) |
| N1—C2—H2 | 118.9 | C11—C10—H10 | 120.4 |
| C3—C2—H2 | 118.9 | C9—C10—H10 | 120.4 |
| C4—C3—C2 | 119.03 (17) | N2—C11—C10 | 122.49 (16) |

| | | | |
|---|---|---|---|
| C4—C3—H3 | 120.5 | N2—C11—H11 | 118.8 |
| C2—C3—H3 | 120.5 | C10—C11—H11 | 118.8 |
| | | | |
| C1—Pd1—N1—C6 | −124.28 (13) | C3—C4—C5—C6 | −0.9 (3) |
| Cl2—Pd1—N1—C6 | 56.97 (12) | C2—N1—C6—C5 | 0.2 (2) |
| C1—Pd1—N1—C2 | 60.31 (13) | Pd1—N1—C6—C5 | −175.38 (13) |
| Cl2—Pd1—N1—C2 | −118.44 (13) | C4—C5—C6—N1 | 1.0 (3) |
| C1—Pd1—N2—C7 | 54.71 (13) | C11—N2—C7—C8 | 0.4 (2) |
| Cl2—Pd1—N2—C7 | −126.52 (12) | Pd1—N2—C7—C8 | 179.50 (13) |
| C1—Pd1—N2—C11 | −126.19 (13) | N2—C7—C8—C9 | 0.1 (3) |
| Cl2—Pd1—N2—C11 | 52.58 (12) | C7—C8—C9—C10 | −0.2 (3) |
| C6—N1—C2—C3 | −1.5 (2) | C8—C9—C10—C11 | −0.3 (3) |
| Pd1—N1—C2—C3 | 173.95 (13) | C7—N2—C11—C10 | −0.9 (2) |
| N1—C2—C3—C4 | 1.6 (3) | Pd1—N2—C11—C10 | 179.97 (14) |
| C2—C3—C4—C5 | −0.3 (3) | C9—C10—C11—N2 | 0.8 (3) |