

[1-Benzyl-3-(2-pyridyl)-1H-pyrazole- κ^2N^2,N^3]dichloridopalladium(II)

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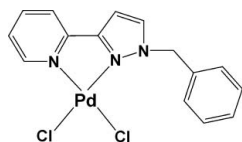
Received 28 October 2007; accepted 29 October 2007

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(C-C) = 0.006$ Å; R factor = 0.029; wR factor = 0.091; data-to-parameter ratio = 16.6.

In the title compound, $[PdCl_2(C_{15}H_{13}N_3)]$, the Pd^{II} center is four-coordinated by two N-atom donors from one 1-benzyl-3-(2-pyridyl)-1H-pyrazole ligand and by two Cl atoms in a distorted square-planar coordination environment. In the crystal structure, adjacent Pd^{II} mononuclear units are linked to form dimers through $C-H \cdots \pi$ interactions. The dimers are further interlinked by weak intermolecular $C-H \cdots Cl$ interactions.

Related literature

For related literature, see: Bell *et al.* (2003); Desiraju & Steiner (1999); Liu *et al.* (2007); Paul *et al.* (2004); Shi *et al.* (2005); Sony & Ponnuswamy (2006); Steel (2005); Ward *et al.* (2001); Singh *et al.* (2003); Zou *et al.* (2004).



Experimental

Crystal data

$[PdCl_2(C_{15}H_{13}N_3)]$
 $M_r = 412.58$
 Monoclinic, $C2/c$
 $a = 19.965$ (6) Å
 $b = 8.958$ (3) Å
 $c = 17.482$ (6) Å
 $\beta = 98.356$ (5)°

$V = 3093.4$ (17) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 1.54$ mm⁻¹
 $T = 293$ (2) K
 $0.19 \times 0.15 \times 0.11$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 1998)
 $T_{min} = 0.759$, $T_{max} = 0.849$
 8670 measured reflections
 3163 independent reflections
 2508 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.091$
 $S = 1.02$
 3163 reflections

190 parameters
 H-atom parameters constrained
 $\Delta\rho_{max} = 0.44$ e Å⁻³
 $\Delta\rho_{min} = -0.49$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Pd1—N3	2.048 (3)	Pd1—Cl2	2.2709 (11)
Pd1—N2	2.060 (3)	Pd1—Cl1	2.2889 (11)
N3—Pd1—N2	79.81 (12)	N3—Pd1—Cl1	175.60 (10)
N3—Pd1—Cl2	92.28 (10)	N2—Pd1—Cl1	99.78 (9)
N2—Pd1—Cl2	172.08 (9)	Cl2—Pd1—Cl1	88.14 (4)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C13—H13A \cdots Cg1 ⁱ	0.93	2.60	3.4635	155
C9—H9A \cdots Cl2 ⁱⁱ	0.93	2.83	3.721 (5)	162
C12—H12A \cdots Cl2 ⁱⁱ	0.93	2.78	3.712 (5)	176

Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $x, y - 1, z$. Cg1 is the centroid of the C1–C6 ring.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL and PLATON (Spek, 2003).

The authors thank Zhengzhou University of Light Industry and Henan Provincial Key Laboratory of Surface and Interface Science as well as Nankai University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2570).

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supplementary materials

Acta Cryst. (2007). E63, m2892 [doi:10.1107/S1600536807054049]

[1-Benzyl-3-(2-pyridyl)-1*H*-pyrazole- κ^2N^2,N^3]dichloridopalladium(II)

C.-S. Liu and X.-S. Shi

Comment

Nowadays, much attention has been focused on the synthetic approach and the structural control of metal-organic coordination architectures with ligands based on pyrazolyl-pyridine chelating units (Steel, 2005; Ward *et al.*, 2001;). In this field many novel functional complexes through the use of 3-(2-pyridyl)pyrazole and/or 3-(2-pyridyl)pyrazole-based ligands (Bell *et al.*, 2003; Paul *et al.*, 2004; Singh *et al.*, 2003; Ward *et al.*, 2001; Zou *et al.*, 2004) have been reported. Recently, we have reported the preparation of a non-planar ligand, 1-[3-(2-pyridyl)pyrazol-1-ylmethyl]benzene (denoted *L*) (Liu *et al.*, 2007; Shi *et al.*, 2005). Now we report here the crystal structure of a palladium complex of this ligand, [Pd(*L*)Cl₂].

In the title compound, the Pd^{II} center is four-coordinated by two N-atoms from one *L* ligand and two Cl atoms (Table 1). The coordination geometry around the Pd^{II} center can be described as distorted square planar (Fig. 1).

In the crystal structure, the Pd^{II} mononuclear units at (*x*, *y*, *z*) and (1 - *x*, 2 - *y*, -*z*) are interconnected to form a dimer through intermolecular C—H... π interactions involving C1—C6 (centroid Cg1) benzene rings of the *L* ligands (Sony and Ponnuswamy, 2006) (Table 2). The dimers are further interlinked to form a chain along the *b* axis by weak intermolecular C—H...Cl (Table 2) interactions (Desiraju & Steiner, 1999).

Experimental

The ligand 1-[3-(2-pyridyl)pyrazol-1-ylmethyl]benzene (*L*) was synthesized according to the method reported in the literature (Liu *et al.*, 2007; Shi *et al.*, 2005). A solution of PdCl₂ (0.1 mmol) in the mixture solution of methanol (15 ml) and acetonitrile (5 ml) was added to *L* (0.1 mmol). A yellow solid formed was filtered off and the resulting solution was kept at room temperature. Yellow single crystals suitable for X-ray analysis were obtained by slow evaporation of the solvent after several days (yield: ~30%). Analysis calculated for (C₁₅H₁₃PdCl₂N₂): C 45.20, H 3.29, N 7.03%; found: C 45.11, H 3.41, N 7.21%.

Refinement

H atoms were included in calculated positions and treated in the subsequent refinement as riding atoms, with C—H = 0.93 (aromatic) or 0.97 Å (methylene) and O—H = 0.82 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{O})$.

Figures

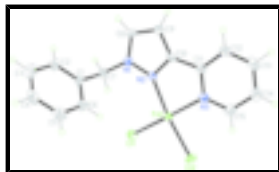


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

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Crystal data

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$M_r = 412.58$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 19.965\ (6)\ \text{\AA}$

$b = 8.958\ (3)\ \text{\AA}$

$c = 17.482\ (6)\ \text{\AA}$

$\beta = 98.356\ (5)^\circ$

$V = 3093.4\ (17)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 1632$

$D_x = 1.772\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 831 reflections

$\theta = 2.7\text{--}26.4^\circ$

$\mu = 1.54\ \text{mm}^{-1}$

$T = 293\ (2)\ \text{K}$

Block, yellow

$0.19 \times 0.15 \times 0.11\ \text{mm}$

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293\ (2)\ \text{K}$

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 1998)

$T_{\min} = 0.759$, $T_{\max} = 0.849$

8670 measured reflections

3163 independent reflections

2508 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 26.4^\circ$

$\theta_{\min} = 2.1^\circ$

$h = -23 \rightarrow 24$

$k = -11 \rightarrow 11$

$l = -15 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.091$

$S = 1.02$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

3163 reflections $\Delta\rho_{\max} = 0.44 \text{ e } \text{\AA}^{-3}$
 190 parameters $\Delta\rho_{\min} = -0.49 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	0.420580 (14)	0.88810 (3)	0.548219 (17)	0.02885 (12)
C1	0.29531 (19)	0.7276 (4)	0.3303 (2)	0.0326 (9)
C2	0.2583 (2)	0.6509 (5)	0.2711 (3)	0.0488 (12)
H2A	0.2230	0.5893	0.2808	0.059*
C3	0.2736 (3)	0.6651 (7)	0.1953 (3)	0.0655 (15)
H3A	0.2483	0.6133	0.1548	0.079*
C4	0.3257 (3)	0.7553 (5)	0.1814 (3)	0.0588 (14)
H4A	0.3357	0.7655	0.1314	0.071*
C5	0.3630 (3)	0.8305 (5)	0.2408 (3)	0.0540 (12)
H5A	0.3986	0.8911	0.2312	0.065*
C6	0.3481 (2)	0.8170 (5)	0.3150 (2)	0.0413 (10)
H6A	0.3738	0.8686	0.3552	0.050*
C7	0.27897 (19)	0.7142 (5)	0.4114 (2)	0.0360 (9)
H7A	0.2769	0.8133	0.4333	0.043*
H7B	0.2347	0.6686	0.4098	0.043*
C8	0.3326 (2)	0.4749 (4)	0.4638 (2)	0.0403 (10)
H8A	0.3025	0.4099	0.4350	0.048*
C9	0.3871 (2)	0.4338 (4)	0.5144 (2)	0.0411 (10)
H9A	0.4016	0.3373	0.5274	0.049*
C10	0.41689 (19)	0.5672 (4)	0.5427 (2)	0.0321 (9)
C11	0.4763 (2)	0.6006 (4)	0.5991 (2)	0.0328 (9)
C12	0.5161 (2)	0.4936 (5)	0.6407 (3)	0.0456 (11)
H12A	0.5059	0.3929	0.6332	0.055*
C13	0.5704 (2)	0.5360 (5)	0.6926 (3)	0.0519 (12)
H13A	0.5969	0.4649	0.7215	0.062*
C14	0.5853 (2)	0.6851 (6)	0.7015 (3)	0.0602 (14)
H14A	0.6227	0.7163	0.7357	0.072*
C15	0.5440 (2)	0.7882 (5)	0.6588 (3)	0.0554 (13)

supplementary materials

H15A	0.5541	0.8891	0.6653	0.066*
N1	0.32876 (16)	0.6254 (3)	0.46161 (18)	0.0330 (7)
N2	0.38106 (15)	0.6844 (3)	0.51007 (17)	0.0284 (7)
N3	0.49013 (16)	0.7480 (4)	0.60870 (19)	0.0352 (8)
Cl1	0.33790 (5)	1.04250 (11)	0.48807 (6)	0.0442 (3)
Cl2	0.47619 (5)	1.09442 (11)	0.59927 (7)	0.0451 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.03081 (18)	0.02245 (16)	0.03280 (18)	-0.00040 (12)	0.00294 (12)	0.00010 (12)
C1	0.032 (2)	0.031 (2)	0.033 (2)	0.0043 (16)	-0.0004 (17)	-0.0011 (16)
C2	0.043 (3)	0.056 (3)	0.044 (3)	-0.008 (2)	-0.006 (2)	0.000 (2)
C3	0.070 (4)	0.080 (4)	0.039 (3)	0.011 (3)	-0.016 (3)	-0.013 (3)
C4	0.084 (4)	0.062 (3)	0.032 (2)	0.028 (3)	0.011 (3)	0.008 (2)
C5	0.070 (3)	0.044 (3)	0.053 (3)	0.003 (2)	0.025 (3)	0.006 (2)
C6	0.046 (2)	0.040 (2)	0.039 (2)	-0.004 (2)	0.010 (2)	-0.0039 (19)
C7	0.029 (2)	0.042 (2)	0.037 (2)	-0.0048 (17)	0.0017 (17)	-0.0012 (18)
C8	0.051 (3)	0.031 (2)	0.037 (2)	-0.0148 (19)	0.002 (2)	-0.0025 (18)
C9	0.058 (3)	0.026 (2)	0.040 (2)	-0.0046 (19)	0.006 (2)	0.0003 (18)
C10	0.039 (2)	0.0260 (18)	0.031 (2)	-0.0008 (17)	0.0058 (18)	0.0038 (16)
C11	0.038 (2)	0.028 (2)	0.033 (2)	0.0037 (17)	0.0070 (18)	0.0014 (17)
C12	0.054 (3)	0.034 (2)	0.048 (3)	0.006 (2)	0.005 (2)	0.004 (2)
C13	0.046 (3)	0.051 (3)	0.056 (3)	0.018 (2)	-0.003 (2)	0.012 (2)
C14	0.042 (3)	0.058 (3)	0.073 (3)	0.006 (2)	-0.017 (3)	0.004 (3)
C15	0.045 (3)	0.042 (3)	0.074 (3)	-0.006 (2)	-0.010 (2)	-0.003 (2)
N1	0.0356 (18)	0.0317 (18)	0.0306 (17)	-0.0065 (14)	0.0011 (14)	0.0006 (14)
N2	0.0304 (16)	0.0243 (16)	0.0299 (16)	-0.0011 (14)	0.0025 (14)	0.0011 (13)
N3	0.0330 (18)	0.0292 (17)	0.0414 (19)	0.0007 (14)	-0.0017 (15)	0.0014 (14)
Cl1	0.0472 (6)	0.0307 (5)	0.0503 (6)	0.0081 (5)	-0.0079 (5)	-0.0003 (5)
Cl2	0.0428 (6)	0.0265 (5)	0.0632 (7)	-0.0029 (4)	-0.0012 (5)	-0.0093 (5)

Geometric parameters (\AA , $^\circ$)

Pd1—N3	2.048 (3)	C8—C9	1.349 (6)
Pd1—N2	2.060 (3)	C8—N1	1.350 (5)
Pd1—Cl2	2.2709 (11)	C8—H8A	0.9300
Pd1—Cl1	2.2889 (11)	C9—C10	1.393 (5)
C1—C2	1.367 (5)	C9—H9A	0.9300
C1—C6	1.380 (6)	C10—N2	1.349 (5)
C1—C7	1.506 (5)	C10—C11	1.460 (5)
C2—C3	1.408 (7)	C11—N3	1.354 (5)
C2—H2A	0.9300	C11—C12	1.382 (5)
C3—C4	1.366 (7)	C12—C13	1.362 (6)
C3—H3A	0.9300	C12—H12A	0.9300
C4—C5	1.364 (7)	C13—C14	1.372 (7)
C4—H4A	0.9300	C13—H13A	0.9300
C5—C6	1.377 (6)	C14—C15	1.382 (6)
C5—H5A	0.9300	C14—H14A	0.9300

C6—H6A	0.9300	C15—N3	1.334 (5)
C7—N1	1.462 (5)	C15—H15A	0.9300
C7—H7A	0.9700	N1—N2	1.353 (4)
C7—H7B	0.9700		
N3—Pd1—N2	79.81 (12)	N1—C8—H8A	125.4
N3—Pd1—Cl2	92.28 (10)	C8—C9—C10	105.2 (4)
N2—Pd1—Cl2	172.08 (9)	C8—C9—H9A	127.4
N3—Pd1—C11	175.60 (10)	C10—C9—H9A	127.4
N2—Pd1—C11	99.78 (9)	N2—C10—C9	110.1 (3)
Cl2—Pd1—C11	88.14 (4)	N2—C10—C11	117.1 (3)
C2—C1—C6	119.3 (4)	C9—C10—C11	132.8 (4)
C2—C1—C7	120.4 (4)	N3—C11—C12	121.3 (4)
C6—C1—C7	120.3 (3)	N3—C11—C10	114.5 (3)
C1—C2—C3	120.0 (4)	C12—C11—C10	124.2 (4)
C1—C2—H2A	120.0	C13—C12—C11	119.9 (4)
C3—C2—H2A	120.0	C13—C12—H12A	120.1
C4—C3—C2	119.7 (5)	C11—C12—H12A	120.1
C4—C3—H3A	120.2	C12—C13—C14	119.0 (4)
C2—C3—H3A	120.2	C12—C13—H13A	120.5
C5—C4—C3	120.2 (5)	C14—C13—H13A	120.5
C5—C4—H4A	119.9	C13—C14—C15	119.1 (4)
C3—C4—H4A	119.9	C13—C14—H14A	120.4
C4—C5—C6	120.3 (5)	C15—C14—H14A	120.4
C4—C5—H5A	119.8	N3—C15—C14	122.3 (4)
C6—C5—H5A	119.8	N3—C15—H15A	118.8
C5—C6—C1	120.5 (4)	C14—C15—H15A	118.8
C5—C6—H6A	119.7	C8—N1—N2	109.7 (3)
C1—C6—H6A	119.7	C8—N1—C7	126.2 (3)
N1—C7—C1	112.8 (3)	N2—N1—C7	124.0 (3)
N1—C7—H7A	109.0	C10—N2—N1	105.9 (3)
C1—C7—H7A	109.0	C10—N2—Pd1	113.4 (2)
N1—C7—H7B	109.0	N1—N2—Pd1	140.6 (2)
C1—C7—H7B	109.0	C15—N3—C11	118.3 (4)
H7A—C7—H7B	107.8	C15—N3—Pd1	126.5 (3)
C9—C8—N1	109.1 (3)	C11—N3—Pd1	115.0 (3)
C9—C8—H8A	125.4		
C6—C1—C2—C3	-0.8 (7)	C1—C7—N1—C8	-79.0 (5)
C7—C1—C2—C3	179.4 (4)	C1—C7—N1—N2	96.7 (4)
C1—C2—C3—C4	0.3 (8)	C9—C10—N2—N1	0.3 (4)
C2—C3—C4—C5	0.4 (8)	C11—C10—N2—N1	-179.1 (3)
C3—C4—C5—C6	-0.5 (7)	C9—C10—N2—Pd1	179.8 (3)
C4—C5—C6—C1	-0.1 (7)	C11—C10—N2—Pd1	0.5 (4)
C2—C1—C6—C5	0.7 (6)	C8—N1—N2—C10	-0.6 (4)
C7—C1—C6—C5	-179.5 (4)	C7—N1—N2—C10	-176.9 (3)
C2—C1—C7—N1	106.9 (4)	C8—N1—N2—Pd1	-179.9 (3)
C6—C1—C7—N1	-72.8 (5)	C7—N1—N2—Pd1	3.7 (6)
N1—C8—C9—C10	-0.5 (5)	N3—Pd1—N2—C10	1.4 (3)
C8—C9—C10—N2	0.1 (5)	C11—Pd1—N2—C10	-174.1 (2)

supplementary materials

C8—C9—C10—C11	179.3 (4)	N3—Pd1—N2—N1	-179.2 (4)
N2—C10—C11—N3	-3.3 (5)	C11—Pd1—N2—N1	5.2 (4)
C9—C10—C11—N3	177.6 (4)	C14—C15—N3—C11	-0.7 (7)
N2—C10—C11—C12	176.4 (4)	C14—C15—N3—Pd1	174.9 (4)
C9—C10—C11—C12	-2.8 (7)	C12—C11—N3—C15	0.9 (6)
N3—C11—C12—C13	0.2 (6)	C10—C11—N3—C15	-179.5 (4)
C10—C11—C12—C13	-179.5 (4)	C12—C11—N3—Pd1	-175.2 (3)
C11—C12—C13—C14	-1.3 (7)	C10—C11—N3—Pd1	4.5 (4)
C12—C13—C14—C15	1.4 (8)	N2—Pd1—N3—C15	-179.0 (4)
C13—C14—C15—N3	-0.4 (8)	C12—Pd1—N3—C15	1.6 (4)
C9—C8—N1—N2	0.7 (5)	N2—Pd1—N3—C11	-3.3 (3)
C9—C8—N1—C7	176.9 (3)	C12—Pd1—N3—C11	177.3 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C13—H13A \cdots Cg1 ⁱ	0.93	2.60	3.4635	155
C9—H9A \cdots Cl2 ⁱⁱ	0.93	2.83	3.721 (5)	162
C12—H12A \cdots Cl2 ⁱⁱ	0.93	2.78	3.712 (5)	176

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x, y-1, z$.

Fig. 1

