Crystal structure of dichlorobis(3-phenylpyridine-κN)-palladium(II), $PdCl_2(C_{11}H_9N)_2$

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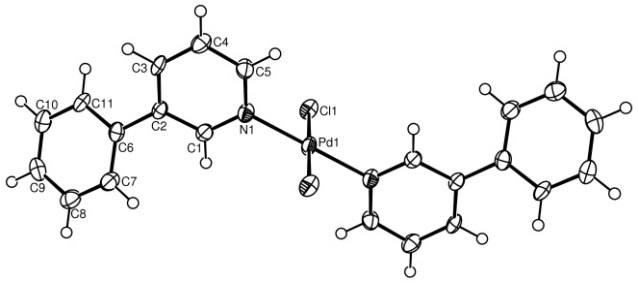

Abstract

$C_{22}H_{18}Cl_2N_2Pd$, triclinic, $P\bar{1}$ (no. 2), a = 6.5863(5) Å, b = 7.3144(6) Å, c = 10.7240(9) Å, α = 72.185(2)°, β = 87.846(2)°, γ = 74.661(2)°, V = 473.8 Å^3, Z = 1, $R_{gt}(F)$ = 0.049, $wR_{ref}(F^2)$ = 0.101, T = 200 K.

Source of material

To a solution of Na_2PdCl_4 (0.2938 g, 0.999 mmol) in H_2O (20 ml) was added 3-phenylpyridine (0.3120 g, 2.010 mmol) in EtOH (10 ml) and stirred for 3 h at room temperature. The formed precipitate was separated by filtration, washed with H_2O and EtOH, and dried in air to give a pale yellow powder (0.4387 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a CH_3CN solution.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.95 Å and U_{iso}(H) = 1.2 U_{eq}(C). The highest peak (0.66 e Å^{-3}) and the deepest hole (–0.56 e Å^{-3}) in the difference Fourier map are located 1.33 Å and 0.86 Å from the Pd1 atom, respectively.

Discussion

In the title crystal structure, the Pd(II) ion has a *trans*-Cl_2N_2 square-planar coordination defined by two N atoms from two distinct 3-phenylpyridine ligands and two Cl^- anions. The Pd atom is located at the inversion center. Therefore, the asymmetric unit contains one half of the complex and the $PdCl_2N_2$ moiety is exactly planar. The dihedral angle between the $PdCl_2N_2$ moiety and the nearly planar pyridine ring is 87.2(1)°. In the crystal structure, the pyridine and phenyl rings are not parallel: the dihedral angle between the ring planes is 34.1(2)°. The molecules are stacked in columns along [010] with d(Pd···Pd) = 7.3144(6) Å (= length of b axis). In the columns, several intermolecular π–π interactions between adjacent six-membered rings are present. For Cg1 (the centroid of ring C6-C11) and Cg1^{i} (symmetry code i: 1–x,–y,1–z), the centroid-centroid distance is 3.784(3) Å, the ring planes are parallel and shifted by 1.402 Å.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.09 × 0.11 × 0.16 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.70 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2984, 1814
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1481
$N(param)_{refined}$:	124
Programs:	SHELXS-97, SHELXL-97 [1], ORTEP-3 [2], PLATON [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.0720	0.6500	0.2376	0.030
H(3)	2*i*	0.6830	0.3454	0.2709	0.031
H(4)	2*i*	0.7406	0.5759	0.0770	0.035
H(5)	2*i*	0.4562	0.8395	–0.0321	0.033
H(7)	2*i*	0.0323	0.3424	0.3668	0.036
H(8)	2*i*	–0.0192	0.1385	0.5715	0.040
H(9)	2*i*	0.2443	0.0153	0.7366	0.039
H(10)	2*i*	0.5661	0.0937	0.6964	0.040
H(11)	2*i*	0.6229	0.2950	0.4909	0.035

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd(1)	1*a*	0	1	0	0.0225(3)	0.0256(4)	0.0215(3)	–0.0004(2)	–0.0016(2)	0.0000(3)
Cl(1)	2*i*	0.0769(2)	1.1765(2)	0.1282(1)	0.0279(7)	0.0315(8)	0.0299(7)	–0.0014(6)	–0.0031(6)	–0.0083(6)
N(1)	2*i*	0.2400(6)	0.7664(6)	0.0932(4)	0.025(2)	0.026(3)	0.021(2)	–0.003(2)	–0.002(2)	–0.004(2)
C(1)	2*i*	0.2101(8)	0.6347(8)	0.2057(5)	0.021(3)	0.027(3)	0.025(3)	–0.005(2)	–0.001(2)	–0.005(2)
C(2)	2*i*	0.3726(7)	0.4759(7)	0.2783(4)	0.022(3)	0.017(3)	0.023(2)	–0.001(2)	–0.005(2)	–0.009(2)
C(3)	2*i*	0.5695(8)	0.4547(7)	0.2264(5)	0.020(3)	0.018(3)	0.030(3)	0.006(2)	–0.006(2)	–0.005(2)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(4)	2*i*	0.6043(8)	0.5893(8)	0.1114(5)	0.022(3)	0.033(3)	0.030(3)	0.000(2)	0.001(2)	–0.012(3)
C(5)	2*i*	0.4342(8)	0.7451(8)	0.0471(5)	0.026(3)	0.026(3)	0.025(3)	–0.002(2)	0.006(2)	–0.004(2)
C(6)	2*i*	0.3328(8)	0.3431(7)	0.4066(5)	0.028(3)	0.018(3)	0.026(3)	–0.001(2)	–0.001(2)	–0.007(2)
C(7)	2*i*	0.1419(8)	0.2927(8)	0.4332(5)	0.024(3)	0.034(3)	0.029(3)	–0.004(2)	–0.002(2)	–0.008(3)
C(8)	2*i*	0.1114(8)	0.1716(8)	0.5549(5)	0.024(3)	0.036(3)	0.037(3)	–0.003(3)	0.002(2)	–0.010(3)
C(9)	2*i*	0.2676(9)	0.0979(8)	0.6528(5)	0.044(4)	0.021(3)	0.028(3)	–0.007(3)	0.005(3)	–0.004(3)
C(10)	2*i*	0.4578(9)	0.1443(8)	0.6290(5)	0.038(3)	0.028(3)	0.027(3)	–0.002(3)	–0.008(2)	–0.004(3)
C(11)	2*i*	0.4908(8)	0.2648(8)	0.5070(5)	0.020(3)	0.028(3)	0.032(3)	0.002(2)	–0.008(2)	–0.004(3)

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