Crystal structure of dichlorobis(2-phenylpyridine-κN)palladium(II), $PdCl_2(C_{11}H_9N)_2$

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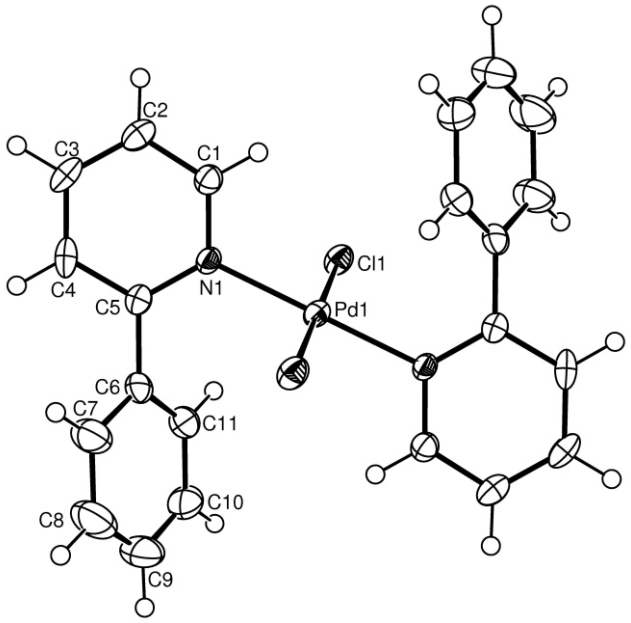
Received April 4, 2011, accepted and available on-line August 24, 2011; CCDC no. 1267/3498



Abstract

$C_{22}H_{18}Cl_2N_2Pd$, triclinic, $P\bar{1}$ (no. 2), a = 7.1075(5) Å, b = 8.0015(6) Å, c = 9.6728(7) Å, α = 77.772(2)°, β = 89.032(2)°, γ = 68.724(1)°, V = 499.9 Å^3, Z = 1, $R_{gt}(F)$ = 0.039, $wR_{ref}(F^2)$ = 0.085, T = 200 K.

Source of material

To a solution of Na_2PdCl_4 (0.2942 g, 1.000 mmol) in H_2O (20 ml) was added 2-phenylpyridine (0.3166 g, 2.040 mmol) and stirred for 3 h at room temperature. The formed precipitate was separated by filtration, washed with H_2O and EtOH, and dried at 50 °C to give a yellow powder (0.3365 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 (0.65 e Å^{-3}) and the deepest hole (–0.57 e Å^{-3}) in the difference Fourier map are located 1.05 Å and 0.92 Å from the atoms Cl1 and Pd1, respectively.

Discussion

In the title complex, the central Pd(II) ion has a *trans*-Cl_2N_2 square-planar coordination defined by two N atoms from two different 2-phenylpyridine ligands and two Cl^- anions. The Pd atom is located at an inversion center, and therefore the asymmetric unit contains one half of the complex and the $PdCl_2N_2$ moiety is exactly plane. The dihedral angle between the $PdCl_2N_2$ moiety and the nearly planar pyridine ring is 66.61(9)°. In the crystal structure, the pyridine and phenyl rings are not parallel: the dihedral angle between the ring planes is 50.3(1)°, and the molecules are stacked in columns along [100] with d(Pd···Pd) = 7.1075(5) Å (= length of a axis). In the columns, several intermolecular π–π interactions between adjacent six-membered rings are present. For Cg1 (the centroid of ring N1–C5) and Cg1^i (symmetry code i: 2–x,–y,–z), the centroid–centroid distance is 3.597(3) Å, the planes are parallel and shifted by 0.942 Å.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.07 × 0.14 × 0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.04 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3114, 1910
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1690
$N(param)_{refined}$:	124
Programs:	SHELXS-97, SHELXL-97 [1], ORTEP-3 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.5791	0.2356	–0.1746	0.030
H(2)	2*i*	0.7220	–0.0768	–0.1652	0.035
H(3)	2*i*	0.8826	–0.2775	0.0478	0.037
H(4)	2*i*	0.9065	–0.1566	0.2434	0.037
H(7)	2*i*	0.6662	0.0161	0.4122	0.056
H(8)	2*i*	0.6699	0.1463	0.6051	0.069
H(9)	2*i*	0.7670	0.3977	0.5819	0.064
H(10)	2*i*	0.8774	0.5113	0.3668	0.049
H(11)	2*i*	0.8661	0.3867	0.1711	0.040

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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*e*	½	½	0	0.0252(3)	0.0183(3)	0.0239(3)	–0.0061(2)	0.0028(2)	–0.0069(2)
Cl(1)	2*i*	0.7314(2)	0.5510(1)	–0.1576(1)	0.0275(6)	0.0243(6)	0.0314(6)	–0.0087(5)	0.0081(5)	–0.0082(5)
N(1)	2*i*	0.6515(5)	0.2261(4)	0.0256(4)	0.019(2)	0.019(2)	0.026(2)	–0.003(2)	–0.000(2)	–0.008(2)
C(1)	2*i*	0.6449(6)	0.1545(6)	–0.0881(5)	0.025(2)	0.020(2)	0.029(2)	–0.007(2)	0.004(2)	–0.005(2)
C(2)	2*i*	0.7295(6)	–0.0308(6)	–0.0835(5)	0.026(2)	0.026(2)	0.038(3)	–0.009(2)	0.008(2)	–0.015(2)
C(3)	2*i*	0.8254(6)	–0.1486(6)	0.0415(5)	0.030(2)	0.015(2)	0.048(3)	–0.007(2)	0.008(2)	–0.011(2)
C(4)	2*i*	0.8374(6)	–0.0771(6)	0.1573(5)	0.021(2)	0.022(2)	0.044(3)	–0.005(2)	–0.002(2)	0.001(2)
C(5)	2*i*	0.7475(6)	0.1125(6)	0.1480(4)	0.020(2)	0.020(2)	0.029(2)	–0.005(2)	0.003(2)	–0.006(2)
C(6)	2*i*	0.7593(6)	0.1921(6)	0.2714(4)	0.024(2)	0.027(2)	0.023(2)	–0.004(2)	–0.004(2)	–0.003(2)
C(7)	2*i*	0.7057(8)	0.1194(8)	0.4014(5)	0.063(4)	0.050(3)	0.034(3)	–0.030(3)	0.005(3)	–0.011(3)
C(8)	2*i*	0.7091(9)	0.1961(9)	0.5162(6)	0.074(4)	0.069(4)	0.030(3)	–0.029(4)	0.006(3)	–0.008(3)
C(9)	2*i*	0.7686(9)	0.3436(8)	0.5032(6)	0.060(4)	0.061(4)	0.038(3)	–0.014(3)	–0.007(3)	–0.021(3)
C(10)	2*i*	0.8307(8)	0.4126(7)	0.3755(5)	0.045(3)	0.036(3)	0.046(3)	–0.015(3)	–0.007(3)	–0.016(2)
C(11)	2*i*	0.8249(7)	0.3378(6)	0.2594(5)	0.030(2)	0.034(3)	0.032(3)	–0.007(2)	–0.006(2)	–0.007(2)

Acknowledgment. This work was supported by Priority Research Centers Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (grant no. 2010-0029626).

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