Crystal structure of *trans*-dichloridobis(isoquinoline-κ*N*)palladium(II), $C_{18}H_{14}Cl_2N_2Pd$

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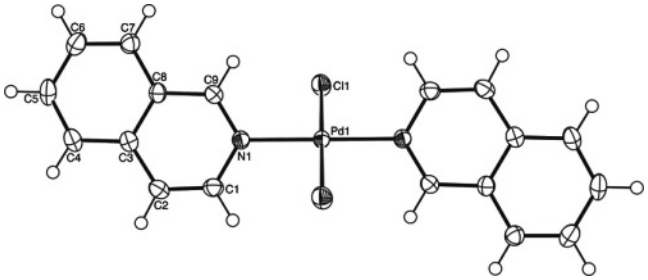

Abstract

$C_{18}H_{14}Cl_2N_2Pd$, monoclinic, $P2_1/n$ (no. 14), a = 4.7559(4) Å, b = 11.3651(9) Å, c = 15.080(1) Å, β = 95.852(2)°, V = 810.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.0206, $wR_{ref}(F^2)$ = 0.0537, T = 200 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.09×0.12×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.73 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω
$2\theta_{max}$:	51.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4765, 1545
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1410
$N(param)_{refined}$:	106
Programs:	SHELX [4], ORTEP-3 [5], PLATON [6]

Source of material

To a solution of isoquinoline (*iqu*; 0.138 g, 1.07 mmol) in MeOH (30 ml) was added Na_2PdCl_4 (0.148 g, 0.50 mmol) and stirred for 3 h at room temperature. The formed precipitate was separated by filtration, washed with H_2O and MeOH, and dried at 50 °C, to give a yellow powder (0.191 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a dimethyl sulfoxide (DMSO) solution at 90 °C.

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) = 1.2U_{eq}(C).

Discussion

This contribution is part of my general interest in the structures of dichlorido palladium(II) complexes [1, 2]. In the title complex $[PdCl_2(iqu)_2]$, the central Pd(II) ion has a *trans*-Cl_2N_2 square-planar coordination geometry defined by two N atoms from two *iqu* ligands and two chlorido ligands. Crystal structure of the related quinoline-Pd(II) complex $[PdCl_2(quinoline)_2]$ has been investigated previously [3]. The Pd atom is located on an inversion center, and thus the asymmetric unit contains one half of the complex and the $PdCl_2N_2$ coordination environment is exactly planar. The nearly planar *iqu* ligands, with a maximum deviation of 0.029(2) Å from the least-squares plane, are therefore parallel. The dihedral angle between the $PdCl_2N_2$ plane and *iqu* ligand is 50.69(4)°. In the crystal, the complex molecules are stacked into columns along [100] with d(Pd···Pd) = 4.7559(4) Å (= length of a axis). In the columns, several intermolecular π-π interactions between the six-membered rings are present, the shortest ring centroid-centroid distance being 3.782(1) Å between pyridine and benzene rings. Moreover, the complexes are connected by weak intermolecular C–H···Cl hydrogen bonds with d(C···Cl) = 3.611(2) Å, thereby forming helical chains also running along [100].

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.5119	0.6379	0.3306	0.030
H(2)	4*e*	0.2937	0.6415	0.1884	0.031
H(4)	4*e*	–0.0771	0.5533	0.0713	0.034
H(5)	4*e*	–0.4104	0.4090	0.0354	0.038
H(6)	4*e*	–0.5406	0.2770	0.1431	0.037
H(7)	4*e*	–0.3240	0.2840	0.2876	0.032
H(9)	4*e*	0.0389	0.3719	0.3966	0.028

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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)	2*a*	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	0.0215(1)	0.0193(2)	0.0194(1)	–0.00134(8)	–0.00101(9)	0.00091(7)
Cl(1)	4*e*	0.4242(1)	0.69908(5)	0.51367(3)	0.0392(3)	0.0211(3)	0.0276(3)	0.0014(2)	–0.0015(2)	0.0007(2)
N(1)	4*e*	0.2936(4)	0.5028(1)	0.3762(1)	0.0219(9)	0.020(1)	0.022(1)	0.0010(6)	0.0003(8)	0.0010(6)
C(1)	4*e*	0.3664(4)	0.5826(2)	0.3139(1)	0.027(1)	0.021(1)	0.027(1)	–0.0028(9)	0.0033(9)	0.0015(8)
C(2)	4*e*	0.2378(4)	0.5850(2)	0.2296(1)	0.031(1)	0.022(1)	0.025(1)	0.0010(9)	0.0062(9)	0.0034(8)
C(3)	4*e*	0.0201(5)	0.5036(2)	0.2025(2)	0.024(1)	0.022(1)	0.022(1)	0.0057(8)	0.0029(9)	–0.0013(7)
C(4)	4*e*	–0.1213(6)	0.4983(2)	0.1153(2)	0.036(1)	0.027(1)	0.022(1)	0.0058(9)	0.001(1)	0.0023(8)
C(5)	4*e*	–0.3213(5)	0.4138(2)	0.0946(2)	0.036(1)	0.034(1)	0.023(1)	0.006(1)	–0.0060(9)	–0.0029(9)
C(6)	4*e*	–0.3983(5)	0.3340(2)	0.1589(2)	0.030(1)	0.027(1)	0.034(1)	–0.0007(9)	–0.0028(9)	–0.0048(9)
C(7)	4*e*	–0.2701(4)	0.3377(2)	0.2441(1)	0.025(1)	0.025(1)	0.029(1)	0.0003(9)	0.0007(8)	0.0009(8)
C(8)	4*e*	–0.0557(4)	0.4225(2)	0.2673(1)	0.021(1)	0.023(1)	0.022(1)	0.0044(8)	0.0021(8)	–0.0011(8)
C(9)	4*e*	0.0896(4)	0.4265(2)	0.3532(1)	0.023(1)	0.022(1)	0.024(1)	–0.0005(8)	0.0032(8)	0.0017(8)

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