Crystal structure of dibromidobis(3-phenylpyridine-κ*N*)palladium(II), $C_{22}H_{18}Br_2N_2Pd$

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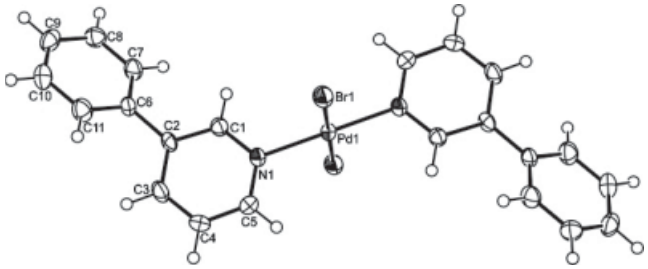

Abstract

$C_{22}H_{18}Br_2N_2Pd$, triclinic, $P\bar{1}$ (no. 2), a = 6.620(1) Å, b = 7.223(2) Å, c = 11.181(2) Å, α = 71.273(4)°, β = 89.765(4)°, γ = 76.928(4)°, V = 491.8 Å^3, Z = 1, $R_{gt}(F)$ = 0.0306, $wR_{ref}(F^2)$ = 0.0868, T = 200 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.11×0.14×0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	50.15 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω
$2\theta_{max}$:	52.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2977, 1858
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1433
$N(param)_{refined}$:	124
Programs:	SHELX [2], ORTEP-3[3], PLATON [4]

Source of material

To a solution of 3-phenylpyridine (0.1704 g, 1.098 mmol) in MeOH (30 ml) was added K_2PdBr_4 (0.2530 g, 0.502 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 (0.2293 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a dimethyl sulfoxide 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). The highest peak (1.55 e·Å^{-3}) and the deepest hole (-0.70 e·Å^{-3}) in the difference Fourier map are located 1.55 Å and 0.89 Å from the atoms H7 and Pd1, respectively.

Discussion

The title complex is isomorphous with the previously reported analogous Pd(II) complex $[PdCl_2(C_{11}H_9N)_2]$ [1]. In the complex, the central Pd(II) has a *trans*-Br_2N_2 square-planar coordination geometry defined by two N atoms from two distinct 3-phenylpyridine ligands and two bromido ligands. The Pd atom is located on an inversion center, and thus the asymmetric unit contains one half of the complex and the $PdBr_2N_2$ moiety is exactly planar. The dihedral angle between the $PdBr_2N_2$ moiety and the nearly planar pyridine ring is 83.4(1)°. In the crystal structure, the pyridine and the phenyl ring of each organic ligand enclose a dihedral angle of 33.7(2)°. The complexes are stacked in columns along [010] with d(Pd···Pd) = 7.2229(15) Å (= length of b axis). In the columns, several intermolecular π-π interactions between adjacent six-membered rings may be 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 3.947(4) Å. The planes are parallel and shifted by 1.642 Å.

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.0684	0.6572	0.2394	0.029
H(3)	2*i*	0.6671	0.3462	0.2780	0.036
H(4)	2*i*	0.7172	0.5663	0.0845	0.039
H(5)	2*i*	0.4426	0.8322	–0.0278	0.037
H(7)	2*i*	0.0233	0.3487	0.3710	0.038
H(8)	2*i*	–0.0266	0.1439	0.5715	0.040
H(9)	2*i*	0.2440	0.0213	0.7322	0.042
H(10)	2*i*	0.5659	0.0942	0.6885	0.044
H(11)	2*i*	0.6156	0.2996	0.4878	0.042

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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*a*	0	1	0	0.0231(3)	0.0239(3)	0.0201(3)	0.0007(2)	0.0010(2)	–0.0020(2)
Br(1)	2*i*	0.08238(9)	1.18985(9)	0.12988(5)	0.0317(3)	0.0319(3)	0.0295(4)	–0.0016(3)	–0.0009(3)	–0.0098(3)
N(1)	2*i*	0.2331(7)	0.7650(7)	0.0972(4)	0.024(2)	0.028(3)	0.021(2)	0.002(2)	–0.002(2)	–0.004(2)
C(1)	2*i*	0.2029(8)	0.6380(8)	0.2087(5)	0.023(3)	0.024(3)	0.024(3)	–0.004(2)	0.000(2)	–0.007(2)
C(2)	2*i*	0.3612(8)	0.4774(8)	0.2822(5)	0.017(3)	0.026(3)	0.023(3)	0.000(2)	–0.008(2)	–0.009(2)
C(3)	2*i*	0.5550(9)	0.4536(9)	0.2322(6)	0.020(3)	0.029(3)	0.035(3)	0.002(2)	–0.005(2)	–0.006(3)
C(4)	2*i*	0.5848(9)	0.5835(9)	0.1181(6)	0.023(3)	0.038(3)	0.032(3)	–0.002(3)	0.009(2)	–0.010(3)
C(5)	2*i*	0.4212(9)	0.7409(9)	0.0512(5)	0.032(3)	0.033(3)	0.021(3)	–0.002(3)	0.004(2)	–0.005(2)
C(6)	2*i*	0.3242(8)	0.3454(8)	0.4083(5)	0.025(3)	0.025(3)	0.022(3)	0.003(2)	–0.003(2)	–0.004(2)
C(7)	2*i*	0.1342(9)	0.2976(9)	0.4353(6)	0.026(3)	0.036(3)	0.029(3)	–0.002(3)	–0.006(2)	–0.009(3)
C(8)	2*i*	0.1041(9)	0.1766(9)	0.5544(6)	0.027(3)	0.035(3)	0.037(4)	–0.009(3)	0.005(3)	–0.010(3)
C(9)	2*i*	0.266(1)	0.1024(9)	0.6498(6)	0.051(4)	0.023(3)	0.028(3)	–0.009(3)	0.005(3)	–0.004(2)
C(10)	2*i*	0.455(1)	0.1467(9)	0.6243(6)	0.041(4)	0.026(3)	0.035(4)	–0.002(3)	–0.006(3)	–0.002(3)
C(11)	2*i*	0.484(1)	0.2682(9)	0.5046(6)	0.033(3)	0.027(3)	0.041(4)	–0.007(3)	0.001(3)	–0.005(3)

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