Crystal structure of *trans*-dibromidobis(4-phenylpyridine-κ*N*) platinum(II), $PtBr_2(C_{11}H_9N)_2$, $C_{22}H_{18}Br_2N_2Pt$

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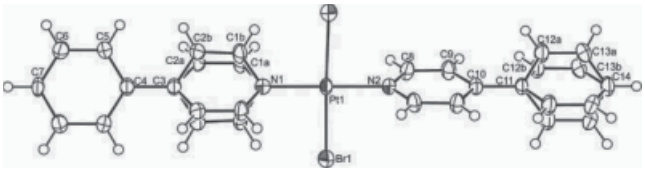

Abstract

$C_{22}H_{18}Br_2N_2Pt$, monoclinic, *C*2/*c* (no. 15), $a = 9.296(1)$ Å, $b = 24.252(3)$ Å, $c = 8.940(1)$ Å, $\beta = 98.893(2)°$, $V = 1991.4$ Å^3, $Z = 4$, $R_{gt(F)} = 0.0200$, $wR_{ref(F^2)} = 0.0518$, $T = 200$ K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.16×0.18×0.19 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	110.68 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω
$2\theta_{max}$:	52.08°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5979, 1894
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1610
$N(param)_{refined}$:	141
Programs:	SHELX [2], ORTEP-3 [3], PLATON [4]

Source of material

To a solution of K_2PtBr_4 (0.296 g, 0.50 mmol) in H_2O (20 ml) and EtOH (20 ml) was added 4-phenylpyridine (*ppy*; 0.159 g, 1.03 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 (0.301 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from an *N*,*N*-dimethylformamide (DMF) solution at 60 °C.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their respective parent atoms with d(C–H) = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. One pyridine ring (N1-C3) and one benzene ring (C11-C14) displayed relatively large anisotropic displacement parameters so that the rings appear to be partially disordered. The C1, C2, C12 and C13 atoms were refined anisotropically as disordered over two sites using EADP option of the SHELX program [2]. The site occupancy factors are 0.547(18) for the C1A and C2A atoms, and 0.528(11) for the C12A and C13A atoms, respectively. The highest peak (0.49 e·Å^{-3}) and the deepest hole (-0.85 e·Å^{-3}) in the difference Fourier map are located 0.56 Å and 0.78 Å from the atoms N2 and Pt1, respectively.

Discussion

The title complex $[PtBr_2(ppy)_2]$ is isomorphous with the previously reported chlorido-Pd(II) complex $[PdCl_2(ppy)_2]$ [1]. The asymmetric unit of the title crystal structure contains one half of a neutral Pt(II) complex. The complex is disposed about a twofold rotation axis running in the [010] direction passing through the Pt1, N1, C3, C4, C7, N2, C10, C11 and C14 atoms. The central Pt(II) ion has a *trans*-Br_2N_2 square-planar coordination geometry defined by two N atoms from two 4-phenylpyridine ligands and two bromido ligands. The two Pt–N bond lengths are nearly equal with d(Pt–N) = 2.017(4) and 2.024(4) Å, and the N–Pt–Br bonds are almost perpendicular. In the refinement, the pyridine ring N1-C3 and the benzene ring C11-C14 were found to be disordered over two sites with the site-occupancy factors of 0.547(18) and 0.528(11), respectively, for the major components. The dihedral angles between the major and minor rings are 27(1)° for the ring N1-C3 and 34.7(9)° for the ring C11-C14, respectively.

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	8*f*	0.547	0.1352	0.0311	0.9451	0.037
H(2A)	8*f*	0.547	0.1350	–0.0641	0.9485	0.037
H(1B)	8*f*	0.453	0.1959	0.0302	0.8679	0.037
H(2B)	8*f*	0.453	0.2020	–0.0637	0.8786	0.037
H(5)	8*f*		0.2117	–0.1473	0.8491	0.037
H(6)	8*f*		0.2108	–0.2432	0.8476	0.040
H(7)	4*e*		0	–0.2914	¾	0.035
H(8)	8*f*		0.0877	0.2158	0.9650	0.036
H(9)	8*f*		0.0923	0.3102	0.9699	0.036
H(12A)	8*f*	0.528	0.1932	0.3954	0.8920	0.043
H(13A)	8*f*	0.528	0.1961	0.4910	0.8827	0.046
H(12B)	8*f*	0.472	0.1011	0.3927	0.9676	0.043
H(13B)	8*f*	0.472	0.1011	0.4883	0.9668	0.046
H(14)	4*e*		0	0.5382	¾	0.043

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt(1)	4*e*		0	0.123743(8)	$\frac{3}{4}$	0.0290(1)	0.0155(1)	0.0278(1)	0	0.00239(9)	0
Br(1)	8*f*		–0.25533(4)	0.12552(2)	0.77916(5)	0.0316(2)	0.0272(2)	0.0550(3)	–0.0002(2)	0.0075(2)	–0.0028(2)
N(1)	4*e*		0	0.0403(2)	$\frac{3}{4}$	0.025(3)	0.024(3)	0.030(3)	0	0.003(2)	0
N(2)	4*e*		0	0.2069(2)	$\frac{3}{4}$	0.026(3)	0.019(2)	0.031(3)	0	0.003(2)	0
C(1A)	8*f*	0.547	0.079(2)	0.0116(7)	0.864(2)	0.041(9)	0.020(2)	0.030(9)	–0.001(4)	–0.001(5)	–0.005(5)
C(2A)	8*f*	0.547	0.079(2)	–0.0453(7)	0.866(2)	0.027(9)	0.025(2)	0.039(9)	–0.001(5)	0.000(4)	0.006(5)
C(1B)	8*f*	0.453	0.113(3)	0.0105(9)	0.819(2)	0.041(9)	0.020(2)	0.030(9)	–0.001(4)	–0.001(5)	–0.005(5)
C(2B)	8*f*	0.453	0.119(2)	–0.0454(9)	0.825(2)	0.027(9)	0.025(2)	0.039(9)	–0.001(5)	0.000(4)	0.006(5)
C(3)	4*e*		0	–0.0757(2)	$\frac{3}{4}$	0.028(3)	0.019(3)	0.033(3)	0	0.013(3)	0
C(4)	4*e*		0	–0.1369(2)	$\frac{3}{4}$	0.030(3)	0.020(3)	0.032(3)	0	0.008(2)	0
C(5)	8*f*		0.1254(5)	–0.1665(2)	0.8086(5)	0.029(2)	0.022(2)	0.042(3)	–0.002(2)	0.006(2)	0.002(2)
C(6)	8*f*		0.1248(5)	–0.2236(2)	0.8080(5)	0.033(2)	0.026(2)	0.041(3)	0.002(2)	0.008(2)	0.003(2)
C(7)	4*e*		0	–0.2523(2)	$\frac{3}{4}$	0.038(4)	0.014(3)	0.039(4)	0	0.015(3)	0
C(8)	8*f*		0.0514(5)	0.2355(2)	0.8752(5)	0.039(3)	0.021(2)	0.027(2)	0.004(2)	–0.001(2)	0.001(2)
C(9)	8*f*		0.0537(5)	0.2917(2)	0.8787(5)	0.040(3)	0.022(2)	0.027(2)	–0.001(2)	0.001(2)	–0.004(2)
C(10)	4*e*		0	0.3225(2)	$\frac{3}{4}$	0.029(3)	0.019(3)	0.027(3)	0	0.009(2)	0
C(11)	4*e*		0	0.3836(2)	$\frac{3}{4}$	0.028(3)	0.016(3)	0.031(3)	0	0.006(2)	0
C(12A)	8*f*	0.528	0.115(2)	0.4143(5)	0.833(2)	0.042(8)	0.021(3)	0.042(7)	–0.001(5)	–0.004(4)	0.001(4)
C(13A)	8*f*	0.528	0.116(1)	0.4712(5)	0.830(1)	0.039(7)	0.023(3)	0.052(7)	–0.008(4)	0.002(4)	–0.005(4)
C(12B)	8*f*	0.472	0.060(2)	0.4122(6)	0.879(2)	0.042(8)	0.021(3)	0.042(7)	–0.001(5)	–0.004(4)	0.001(4)
C(13B)	8*f*	0.472	0.060(2)	0.4690(5)	0.878(2)	0.039(7)	0.023(3)	0.052(7)	–0.008(4)	0.002(4)	–0.005(4)
C(14)	4*e*		0	0.4990(2)	$\frac{3}{4}$	0.040(4)	0.017(3)	0.053(4)	0	0.013(3)	0

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