Crystal structure of *cis*-tetrabromidobis(4-phenylpyridine-κ*N*) platinum(IV), $C_{22}H_{18}Br_4N_2Pt$

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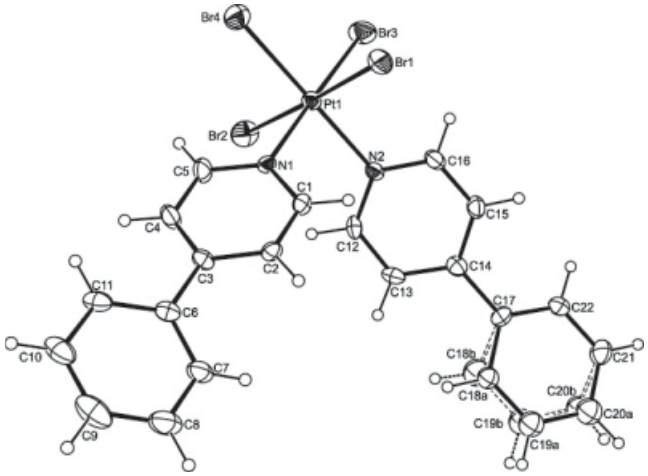

Abstract

$C_{22}H_{18}Br_4N_2Pt$, monoclinic, $P2_1/c$ (no. 14), a = 9.039(1) Å, b = 18.009(3) Å, c = 14.807(2) Å, β = 104.659(3)°, V = 2331.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.0432, $wR_{ref}(F^2)$ = 0.1124, T = 200 K.

Table 1. Data collection and handling.

Crystal:	yellow sticks, size 0.12×0.14×0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	128.83 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω
$2\theta_{max}$:	52.14°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14257, 4522
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3059
$N(param)_{refined}$:	260
Programs:	SHELX [2], ORTEP-3[3], PLATON [4]

Source of material

To a solution of K_2PtCl_6 (0.243 g, 0.500 mmol) in H_2O (50 ml) were added KBr (0.549 g, 4.613 mmol) and 4-phenylpyridine (*ppy*; 0.161 g, 1.035 mmol) in EtOH (10 ml) and stirred for 24 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 (0.039 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) = 1.2U_{eq}(C). The highest peak (1.49 e·Å^{-3}) and the deepest hole (–1.32 e·Å^{-3}) in the difference Fourier map are located 0.59 Å and 0.64 Å from the atoms Pt1 and Br2, respectively. In the refinement, one of the phenyl rings was found to be partially disordered. Atoms C18, C19 and C20 were isotropically as disordered over two sites with a site occupancy factor of 0.59(3) for the major components.

Discussion

In the title complex $[PtBr_4(ppy)_2]$, the central Pt(IV) ion is six-coordinated by two N atoms from two different 4-phenylpyridine ligands and four bromido ligands in a slightly distorted octahedral manner. The *ppy* ligands are *cis* coordinated with respect to each other. By contrast, in the related $[PtCl_2(ppy)_2]\cdot H_2O$, the *ppy* ligands are arranged in *trans* geometry [1]. The distances d(Pt–N) are: 2.089(7) Å and 2.101(7) Å, whereas the d(Pt–Br) range from 2.4236(12) Å - 2.4582(12) Å. In the crystal structure, the phenyl ring C17-C22 is disordered over two sites. The dihedral angle between the mostly and less occupied rings is 22(1)°. The *ppy* ligands are not planar. The phenyl ring C6-C11 is inclined by 26.3(5)° relative to its carrier pyridine ring. The dihedral angles between the pyridine and disordered phenyl ring are 31.6(7)° for the mostly occupied ring and 9.7(9)° for the less occupied ring. The complexes are stacked in columns along [100] and display numerous intermolecular π-π interactions between the adjacent six-membered rings. For Cg1 (the centroid of ring N1-C5) and Cg2^i (the centroid of ring C6-C11, symmetry code i: -x, -y, 1-z), the centroid-centroid distance is 3.944(6) Å, the dihedral angle between the planes is 26.3(5)°.

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	4*e*		0.1574	0.2259	0.3806	0.033
H(2)	4*e*		0.0223	0.1854	0.4831	0.034
H(4)	4*e*		0.3322	0.0261	0.5714	0.048
H(5)	4*e*		0.4660	0.0673	0.4672	0.048
H(7)	4*e*		–0.0283	0.1702	0.6222	0.051
H(8)	4*e*		–0.1837	0.1218	0.7144	0.071
H(9)	4*e*		–0.1601	–0.0044	0.7572	0.076
H(10)	4*e*		0.0045	–0.0823	0.7046	0.066
H(11)	4*e*		0.1495	–0.0352	0.6083	0.048
H(12)	4*e*		0.4740	0.2735	0.5059	0.038
H(13)	4*e*		0.3907	0.3833	0.5574	0.041
H(15)	4*e*		0.2813	0.4558	0.2913	0.043
H(16)	4*e*		0.3626	0.3435	0.2454	0.044
C(18A)	4*e*	0.59	0.189(3)	0.505(1)	0.533(1)	0.038(5)
H(18A)	4*e*	0.59	0.1936	0.462	0.5701	0.045
C(19A)	4*e*	0.59	0.112(3)	0.566(1)	0.552(1)	0.052(6)
H(19A)	4*e*	0.59	0.0575	0.5638	0.5994	0.062
C(20A)	4*e*	0.59	0.114(3)	0.632(1)	0.500(2)	0.056(7)
H(20A)	4*e*	0.59	0.0668	0.6758	0.5151	0.068

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Table 2. continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
C(18B)	4*e*	0.41	0.281(5)	0.514(2)	0.552(2)	0.06(1)
H(18B)	4*e*	0.41	0.3348	0.4802	0.5975	0.073
C(19B)	4*e*	0.41	0.206(4)	0.581(2)	0.575(2)	0.06(1)
H(19B)	4*e*	0.41	0.1803	0.5852	0.6334	0.067
C(20B)	4*e*	0.41	0.177(4)	0.635(2)	0.515(2)	0.041(8)
H(20B)	4*e*	0.41	0.1468	0.6808	0.5360	0.049
H(21)	4*e*		0.1900	0.6754	0.3929	0.060
H(22)	4*e*		0.2861	0.5661	0.3485	0.048

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}
Pt(1)	4*e*	0.45272(4)	0.19077(2)	0.32636(2)	0.0280(2)	0.0331(2)	0.0270(2)	0.0018(2)	0.0089(2)	0.0006(2)
Br(1)	4*e*	0.2179(1)	0.19753(6)	0.19918(7)	0.0495(7)	0.0541(6)	0.0346(6)	0.0042(6)	0.0031(5)	0.0011(5)
Br(2)	4*e*	0.6848(1)	0.17731(7)	0.45218(9)	0.0382(7)	0.0702(8)	0.0579(7)	0.0061(6)	0.0054(6)	–0.0062(6)
Br(3)	4*e*	0.6014(2)	0.24089(6)	0.22459(8)	0.0661(9)	0.0553(7)	0.0545(7)	–0.0087(6)	0.0355(6)	–0.0031(6)
Br(4)	4*e*	0.4893(1)	0.06632(6)	0.27359(8)	0.0566(8)	0.0492(7)	0.0554(7)	0.0047(6)	0.0206(6)	–0.0011(5)
N(1)	4*e*	0.3247(9)	0.1501(4)	0.4149(5)	0.024(5)	0.027(4)	0.025(4)	0.005(3)	0.008(3)	–0.001(3)
N(2)	4*e*	0.4164(9)	0.2982(4)	0.3718(5)	0.026(5)	0.034(4)	0.023(4)	0.002(3)	0.008(3)	0.003(3)
C(1)	4*e*	0.194(1)	0.1846(5)	0.4198(6)	0.020(5)	0.030(5)	0.029(5)	0.003(4)	0.000(4)	0.001(4)
C(2)	4*e*	0.114(1)	0.1602(5)	0.4807(6)	0.023(5)	0.032(5)	0.032(5)	0.005(4)	0.010(4)	–0.001(4)
C(3)	4*e*	0.160(1)	0.1000(5)	0.5392(6)	0.024(6)	0.039(5)	0.021(5)	–0.002(4)	0.001(4)	0.001(4)
C(4)	4*e*	0.294(1)	0.0672(6)	0.5323(7)	0.044(7)	0.045(6)	0.033(5)	0.004(5)	0.014(5)	0.016(5)
C(5)	4*e*	0.375(1)	0.0918(5)	0.4704(7)	0.036(7)	0.043(6)	0.041(6)	0.015(5)	0.011(5)	0.015(5)
C(6)	4*e*	0.070(1)	0.0721(5)	0.6025(6)	0.025(6)	0.041(5)	0.024(5)	–0.008(4)	0.004(4)	–0.006(4)
C(7)	4*e*	–0.025(1)	0.1188(6)	0.6369(7)	0.038(7)	0.059(7)	0.033(5)	0.010(6)	0.015(5)	0.005(5)
C(8)	4*e*	–0.115(1)	0.0906(7)	0.6933(8)	0.051(8)	0.088(9)	0.042(7)	0.015(7)	0.021(6)	0.001(6)
C(9)	4*e*	–0.101(2)	0.0150(8)	0.7180(8)	0.056(9)	0.09(1)	0.048(7)	–0.037(8)	0.015(7)	0.006(7)
C(10)	4*e*	–0.004(2)	–0.0315(7)	0.6869(7)	0.08(1)	0.059(7)	0.030(6)	–0.024(7)	0.012(6)	–0.004(5)
C(11)	4*e*	0.082(1)	–0.0034(6)	0.6297(7)	0.047(7)	0.046(6)	0.032(5)	–0.013(5)	0.016(5)	–0.012(5)
C(12)	4*e*	0.430(1)	0.3108(5)	0.4618(7)	0.029(6)	0.028(5)	0.032(5)	0.002(4)	–0.002(4)	0.009(4)
C(13)	4*e*	0.383(1)	0.3764(5)	0.4928(6)	0.041(7)	0.041(6)	0.023(5)	–0.008(5)	0.012(5)	–0.006(4)
C(14)	4*e*	0.324(1)	0.4333(5)	0.4287(6)	0.036(6)	0.031(5)	0.028(5)	–0.008(4)	0.005(4)	–0.003(4)
C(15)	4*e*	0.319(1)	0.4187(5)	0.3370(7)	0.044(7)	0.027(5)	0.034(5)	–0.001(5)	0.005(5)	0.005(4)
C(16)	4*e*	0.366(1)	0.3516(5)	0.3093(6)	0.050(7)	0.038(6)	0.024(5)	–0.002(5)	0.010(5)	0.002(4)
C(17)	4*e*	0.265(1)	0.5044(5)	0.4567(7)	0.060(8)	0.027(5)	0.038(6)	0.007(5)	0.013(6)	–0.006(4)
C(21)	4*e*	0.186(1)	0.6311(6)	0.4270(8)	0.062(9)	0.031(5)	0.059(7)	0.002(5)	0.021(6)	0.005(5)
C(22)	4*e*	0.250(1)	0.5667(5)	0.4036(7)	0.051(7)	0.041(6)	0.033(5)	0.004(5)	0.021(5)	0.005(4)

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