Crystal structure of (2,2'-bipyrimidine-κ^2*N*,*N*')tetrachloridoplatinum(IV) acetone hemisolvate, $C_{9.50}H_9Cl_4N_4O_{0.50}Pt$

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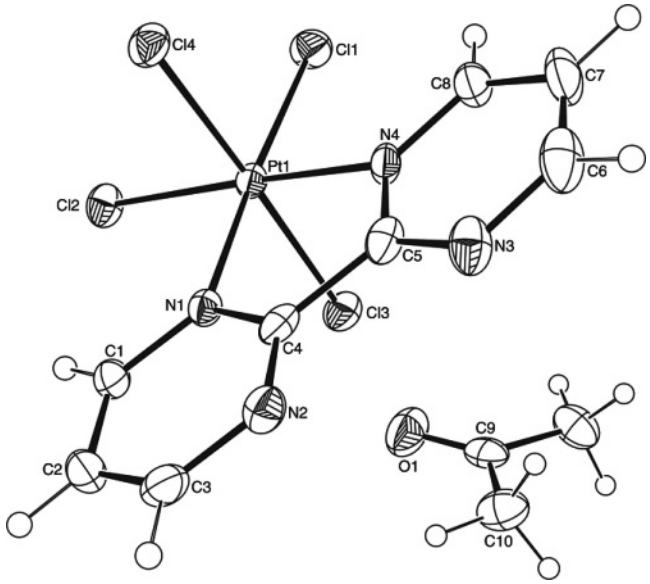

Abstract

$C_{9.50}H_9Cl_4N_4O_{0.50}Pt$, orthorhombic, *Pbcn* (no. 60), a = 17.708(2) Å, b = 12.974(1) Å, c = 12.363(1) Å, V = 2840.4 Å^3, Z = 8, $R_{gt}(F)$ = 0.0249, $wR_{ref}(F^2)$ = 0.0591, T = 200 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.12×0.14×0.14 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	106.24 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω
$2\theta_{max}$:	52.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16427, 2754
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2167
$N(param)_{refined}$:	174
Programs:	SHELX [4], ORTEP-3 [5], PLATON [6]

Source of material

To a solution of K_2PtCl_6 (0.488 g, 1.01 mmol) in H_2O (70 ml) was added 2,2'-bipyrimidine (*bpym*; 0.164 g, 1.04 mmol) and refluxed for 5 h. The formed precipitate was separated by filtration, washed with H_2O, and dried at 50 °C, to give a yellow powder (0.201 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from an acetone solution.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C–H) = 0.95 Å (CH) or 0.98 Å (CH_3) and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The highest peak (1.92 e·Å^{-3}) and the deepest hole (–0.68 e·Å^{-3}) in the difference Fourier map are located 1.90 Å and 1.49 Å from the atoms Cl2 and Cl1, respectively.

Discussion

This contribution is part of my continuing interest in the structural chemistry of Pt-bipyrimidine complexes [1-3]. The asymmetric unit of the title compound $[PtCl_4(bpym)]\cdot 0.5(C_3H_6O)$ contains a neutral Pt(IV) complex and one half of an acetone solvent molecule. The acetone is disposed about a twofold rotation axis running in the [010] direction passing through the C=O group. In the complex, the central Pt(IV) ion is six-coordinated by two pyrimidine N atoms from a chelating *bpym* ligand and four chlorido ligands in a distorted octahedral manner. The main contribution to the distortion of the octahedron is made by the tight chelate angle <N1–Pt1–N4 = 80.20(16)°, which results in non-linear *trans* axes (<N1–Pt1–Cl1 = 175.43(11)° and <N4–Pt1–Cl2 = 174.52(12)°). The apical Cl3–Pt1–Cl4 bond is slightly bent with the bond angle of 176.32(5)°. The two Pt–N and four Pt–Cl bond lengths are almost equivalent, respectively (d(Pt–N) = 2.039(4) Å and 2.042(4) Å; d(Pt–Cl) = 2.2908(12), 2.2988(13), 2.3069(13) and 2.318(1) Å. In the crystal, the two pyrimidine rings of the *bpym* ligand are nearly parallel with the dihedral angle of 2.1(3)° between the ring planes. The complex displays intramolecular C–H···Cl hydrogen bonds with d(C···Cl) = 3.255(5) and 3.300(6) Å.

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

Atom	Site	x	y	z	U_{iso}
H(1)	8*d*	0.1685	0.4454	0.1098	0.034
H(2)	8*d*	0.1181	0.3880	–0.0559	0.039
H(3)	8*d*	0.0642	0.2239	–0.0606	0.041
H(6)	8*d*	0.0615	–0.0525	0.3708	0.055
H(7)	8*d*	0.1105	0.0149	0.5301	0.054
H(8)	8*d*	0.1689	0.1781	0.5236	0.040
H(10A)	8*d*	0.4115	0.1075	0.2206	0.071
H(10B)	8*d*	0.4807	0.0905	0.1393	0.071
H(10C)	8*d*	0.4263	0.1879	0.1248	0.071

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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}
Pt(1)	8*d*	0.18455(1)	0.34880(1)	0.34171(2)	0.0216(1)	0.0186(1)	0.0246(1)	0.00139(8)	0.00072(8)	0.00073(8)
Cl(1)	8*d*	0.22803(8)	0.3722(1)	0.5152(1)	0.0339(8)	0.0412(8)	0.0278(7)	0.0054(6)	–0.0042(6)	–0.0033(6)
Cl(2)	8*d*	0.21959(8)	0.51399(9)	0.2997(1)	0.0326(8)	0.0204(6)	0.0406(7)	–0.0043(6)	–0.0004(6)	0.0003(5)
Cl(3)	8*d*	0.30075(7)	0.2897(1)	0.2846(1)	0.0286(8)	0.0345(8)	0.0362(7)	0.0080(6)	0.0022(6)	–0.0013(6)
Cl(4)	8*d*	0.06452(7)	0.4039(1)	0.3890(1)	0.0250(8)	0.0319(7)	0.0404(7)	0.0048(6)	0.0045(6)	0.0019(6)
N(1)	8*d*	0.1443(2)	0.3161(3)	0.1910(3)	0.022(2)	0.019(2)	0.025(2)	0.000(2)	0.001(2)	0.001(2)
N(2)	8*d*	0.0825(3)	0.1860(3)	0.0914(4)	0.033(3)	0.024(2)	0.035(3)	0.001(2)	–0.002(2)	–0.003(2)
N(3)	8*d*	0.0843(3)	0.0651(3)	0.2767(4)	0.027(3)	0.027(3)	0.053(3)	–0.005(2)	–0.001(2)	0.008(2)
N(4)	8*d*	0.1464(3)	0.2022(3)	0.3668(3)	0.029(3)	0.018(2)	0.034(2)	0.005(2)	0.006(2)	0.007(2)
C(1)	8*d*	0.1466(3)	0.3788(4)	0.1041(4)	0.032(3)	0.023(3)	0.029(3)	0.002(2)	0.006(2)	0.005(2)
C(2)	8*d*	0.1169(3)	0.3458(4)	0.0069(4)	0.034(3)	0.035(3)	0.027(3)	0.005(3)	0.000(2)	0.006(2)
C(3)	8*d*	0.0852(3)	0.2480(4)	0.0054(4)	0.037(4)	0.035(3)	0.030(3)	0.008(3)	–0.002(3)	–0.010(3)
C(4)	8*d*	0.1133(3)	0.2218(4)	0.1803(4)	0.024(3)	0.022(3)	0.035(3)	0.004(2)	0.006(2)	–0.006(2)
C(5)	8*d*	0.1143(3)	0.1574(4)	0.2788(4)	0.023(3)	0.023(3)	0.041(3)	0.003(2)	0.001(3)	0.001(2)
C(6)	8*d*	0.0835(3)	0.0142(5)	0.3695(5)	0.030(4)	0.033(3)	0.074(5)	–0.003(3)	0.008(3)	0.017(3)
C(7)	8*d*	0.1130(4)	0.0530(4)	0.4645(5)	0.046(4)	0.032(3)	0.057(4)	0.002(3)	0.009(3)	0.023(3)
C(8)	8*d*	0.1464(3)	0.1495(4)	0.4607(5)	0.036(4)	0.029(3)	0.036(3)	0.002(3)	0.004(3)	0.008(2)
O(1)	4*c*	½	0.2959(5)	¼	0.061(5)	0.035(4)	0.077(5)	0	0.021(4)	0
C(9)	4*c*	½	0.2036(7)	¼	0.042(6)	0.049(6)	0.030(4)	0	0.016(4)	0
C(10)	8*d*	0.4504(4)	0.1421(5)	0.1775(5)	0.039(4)	0.061(4)	0.041(3)	0.005(3)	–0.003(3)	–0.011(3)

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