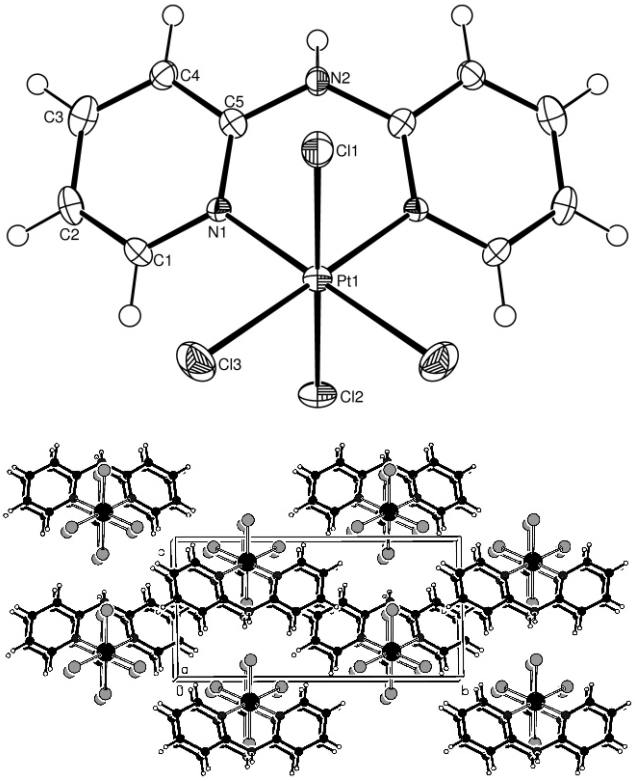
Crystal structure of tetrachloro(2,2′-dipyridylamine-κ^2N,N')platinum(IV), $PtCl_4(C_{10}H_9N_3)$

Kwang Ha*

Chonnam National University, School of Applied Chemical Engineering, Research Institute of Catalysis, Gwangju 500-757, Republic of Korea

Received July 8, 2011, accepted and available on-line November 5, CCDC no. 1267/3610



Abstract

$C_{10}H_9Cl_4N_3Pt$, monoclinic, $P2_1/m$ (no. 11), a = 6.7195(6) Å, b = 14.014(1) Å, c = 7.6712(7) Å, β = 114.863(2)°, V = 655.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.026, $wR_{ref}(F^2)$ = 0.052, T = 200 K.

Source of material

To a solution of K_2PtCl_6 (0.2431 g, 0.500 mmol) in H_2O (30 ml) was added 2,2′-dipyridylamine (0.1725 g, 1.008 mmol), and the mixture was refluxed for 2 h. The formed dark brown precipitate was removed by filtration, and the solvent of the filtrate was evaporated. The residue was washed with H_2O/acetone (1:5) and dried at 50 °C, to give a yellow powder (0.1495 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, d(N—H) = 0.92 Å and U_{iso}(H) = 1.2 U_{eq}(C,N). The highest peak (3.05 e Å^{-3}) and the deepest hole (–1.31 e Å^{-3}) in the difference Fourier map are located 1.60 Å and 0.85 Å from the atoms Cl1 and Pt1, respectively.

Discussion

In the title complex, the Pt(IV) ion is six-coordinated by two N atoms from the chelating 2,2′-dipyridylamine ligand and four Cl^- anions in a slightly distorted octahedral manner (figure, top). The complex is disposed about a crystallographic mirror plane parallel to the *ac* plane passing through the Pt1, Cl1, Cl2 and N2 atoms, which are located at the special positions (x,¼,z). The bond distances Pt—N and Pt—Cl are comparable to those observed in the related Pt(II) complex $[PtCl_2(C_{10}H_9N_3)]$ [1,2,3]. The dihedral angle between the least-squares planes of the pyridyl rings is 34.0(2)°.

In the crystal structure, the complexes are stacked in columns along [100]. When viewed down [010], the successive complexes are stacked in the opposite direction (figure, bottom). In the columns, intermolecular π–π interactions between adjacent pyridyl rings are present. The distance between Cg1 (the centroid of ring N1-C5) and Cg1^{i} (symmetry code i: –x,–y,1–z) is 3.578(3) Å, and the ring planes are parallel and shifted for 1.337 Å. The complexes are linked by intermolecular N–H···Cl hydrogen bonds with d(N2–H2N···Cl2) = 3.454(5) Å, to form chains running along [001].

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.04 × 0.10 × 0.19 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	115.02 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	56.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4630, 1629
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1379
$N(param)_{refined}$:	88
Programs:	SHELXS-97, SHELXL-97 [4], ORTEP-3 [5], PLATON [6]

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

Atom	Site	x	y	z	U_{iso}
H(2N)	2*e*	0.2146	¼	0.3866	0.022
H(1)	4*f*	0.2266	0.0563	0.9337	0.024
H(2)	4*f*	0.3349	–0.0700	0.7987	0.028
H(3)	4*f*	0.3172	–0.0538	0.4882	0.031
H(4)	4*f*	0.2339	0.0947	0.3372	0.023

* e-mail: hakwang@chonnam.ac.kr

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)	2*e*	0.02005(4)	¼	0.82038(4)	0.0204(2)	0.0171(1)	0.0145(2)	0	0.0086(1)	0
Cl(1)	2*e*	–0.2911(3)	¼	0.5323(2)	0.0196(9)	0.0178(8)	0.021(1)	0	0.0028(8)	0
Cl(2)	2*e*	0.3304(3)	¼	1.1089(2)	0.029(1)	0.033(1)	0.0128(9)	0	0.0046(8)	0
Cl(3)	4*f*	–0.1442(2)	0.13552(9)	0.9329(2)	0.0369(9)	0.0284(7)	0.0337(8)	–0.0028(6)	0.0234(7)	0.0073(6)
N(1)	4*f*	0.1641(6)	0.1483(2)	0.7165(5)	0.016(2)	0.013(2)	0.012(2)	0.001(2)	0.005(2)	–0.000(2)
N(2)	2*e*	0.1368(9)	¼	0.4607(8)	0.029(4)	0.012(3)	0.020(3)	0	0.017(3)	0
C(1)	4*f*	0.2265(8)	0.0635(3)	0.8105(7)	0.025(3)	0.017(2)	0.017(3)	0.001(2)	0.008(2)	0.005(2)
C(2)	4*f*	0.2890(8)	–0.0115(3)	0.7308(8)	0.021(3)	0.014(2)	0.033(3)	0.003(2)	0.011(3)	0.006(2)
C(3)	4*f*	0.2846(8)	–0.0011(3)	0.5497(8)	0.021(3)	0.021(3)	0.040(4)	–0.002(2)	0.019(3)	–0.008(2)
C(4)	4*f*	0.2327(8)	0.0860(3)	0.4596(7)	0.022(3)	0.019(2)	0.021(3)	–0.004(2)	0.012(2)	–0.003(2)
C(5)	4*f*	0.1780(7)	0.1618(3)	0.5498(7)	0.012(3)	0.017(2)	0.019(3)	–0.001(2)	0.006(2)	0.002(2)

Acknowledgment. This work was supported by Priority Research Centers Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (grant no. 2010-0029626).

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