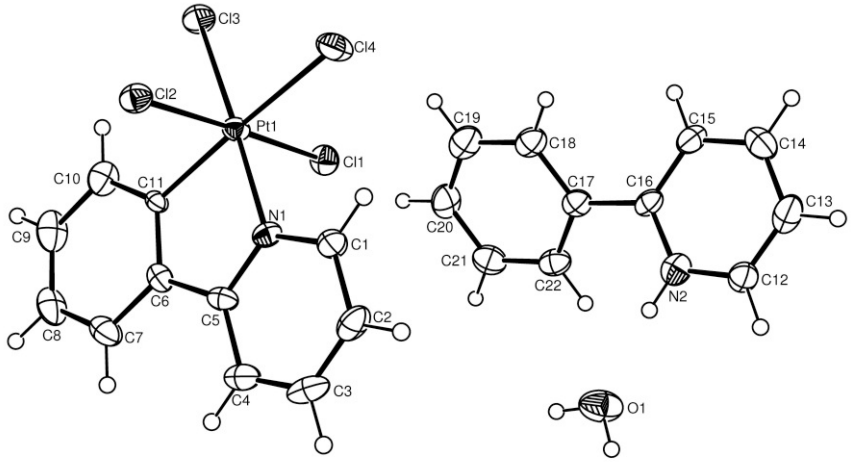
Crystal structure of 2-phenylpyridinium tetrachloro-[2-(2-pyridyl)phenyl-κ^2*N*,*C*]platinate(IV) monohydrate, $[C_{11}H_{10}N][PtCl_4(C_{11}H_8N)] \cdot H_2O$

Kwang Ha*

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

Received June 26, 2011, accepted and available on-line December 22, 2011; CCDC no. 1267/3606



Abstract

$C_{22}H_{20}Cl_4N_2OPt$, monoclinic, $P12_1/c1$ (no. 14), a = 14.157(1) Å, b = 9.1855(8) Å, c = 17.053(1) Å, β = 92.039(2)°, V = 2216.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.037, $wR_{ref}(F^2)$ = 0.086, T = 200 K.

Source of material

To a suspension of K_2PtCl_6 (0.2426 g, 0.499 mmol) in H_2O (30 ml)/EtOH (10 ml) was added 2-phenylpyridine (0.1707 g, 1.100 mmol), and the mixture was refluxed for 3 h. After evaporation of the solvent, the residue was washed with H_2O and dried at 50 °C, to give a yellow powder (0.2834 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a CH_3CN solution. The crystallization water originating from the synthesis is still present.

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) = 0.88 Å and U_{iso}(H) = 1.2 U_{eq}(C,N). The H atoms of the solvent water molecule were located from the difference Fourier map and then allowed to ride on their parent O atom in the final cycles of refinement with d(O—H) = 0.84 Å and U_{iso}(H) = 1.5 U_{eq}(O). The highest peak (3.06 e Å^{-3}) and the deepest hole (–1.28 e Å^{-3}) in the difference Fourier map are located 1.86 Å and 0.88 Å from the atoms Cl3 and Pt1, respectively.

Discussion

The asymmetric unit of the title crystal structure consists of a protonated 2-phenylpyridine cation, an anionic Pt(IV) complex $[PtCl_4(C_{11}H_8N)]^-$ and a solvent water molecule. In the complex, the central Pt(IV) ion is six-coordinated by one N atom and one C atom from the chelating anionic 2-(2-pyridyl)phenyl ligand and four Cl^- anions in a distorted octahedral manner. The main contribution to the distortion of the octahedron is made by the tight chelate angle ∠N1–Pt1–C11 = 81.1(2)°, which results in non-linear *trans* axes (∠N1–Pt1–Cl3 = 175.8(2)° and ∠C11–Pt1–Cl4 = 175.1(2)°). The apical bonds Cl1–Pt1–Cl2 are almost linear with the bond angle of 177.95(6)°. The bond lengths Pt—Cl are slightly different, because of the different *trans* effects of the C, N and Cl atoms. The bond Pt1—Cl4 *trans* to the C atom (2.415(2) Å) is somewhat longer than the bonds Pt1–Cl1/Cl2/Cl3 *trans* to the N or Cl atoms (2.312(2) - 2.349(2) Å). In the crystal structure, the pyridyl and phenyl rings of the anionic ligand are nearly parallel with the dihedral angle of 6.4(3)° between the ring planes. However, the phenyl ring of the cation inclined with the dihedral angle of 31.8(3)° with respect to the carrier pyridinium plane. The compound displays numerous intermolecular π–π interactions between adjacent six-membered rings. For Cg1 (the centroid of ring N1-C5) and Cg2^i (ring N2-C16, symmetry code i: 1–x,1–y,–z), the centroid-centroid distance is 3.740(4) Å and the dihedral angle between the ring planes is 1.2(3)°. Moreover, the complex ion and the water molecule are linked by intermolecular O–H···Cl hydrogen bonds with d(O···Cl) = 3.196(5) Å and 3.359(5) Å, thereby forming a chain-like structure along [001]. Each water molecule, as an H-bond acceptor, is linked to the pyridinium N–H group with d(N2–H2N···O1) = 2.775(8) Å.

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

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.10 × 0.13 × 0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	68.31 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	56.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15781, 5399
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3668
$N(param)_{refined}$:	271
Programs:	SHELXS-97, SHELXL-97 [1], ORTEP-3 [2], PLATON [3]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.6061	0.6259	–0.0506	0.039
H(2)	4*e*	0.5247	0.4202	–0.0976	0.047

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3)	4*e*	0.5725	0.1905	–0.0587	0.050
H(4)	4*e*	0.7043	0.1613	0.0253	0.044
H(7)	4*e*	0.8400	0.1612	0.1011	0.047
H(8)	4*e*	0.9717	0.1793	0.1865	0.054
H(9)	4*e*	1.0196	0.4076	0.2326	0.056
H(10)	4*e*	0.9419	0.6182	0.1887	0.046
H(2N)	4*e*	0.2213	0.5708	0.1770	0.044
H(12)	4*e*	0.0977	0.5984	0.0936	0.044
H(13)	4*e*	0.0511	0.8308	0.0592	0.056
H(14)	4*e*	0.1354	1.0296	0.1104	0.051
H(15)	4*e*	0.2638	0.9910	0.1975	0.048
H(18)	4*e*	0.4167	0.9109	0.2188	0.047
H(19)	4*e*	0.5500	0.8544	0.2964	0.057
H(20)	4*e*	0.5520	0.6463	0.3734	0.055
H(21)	4*e*	0.4218	0.4931	0.3733	0.053
H(22)	4*e*	0.2868	0.5474	0.2954	0.045
H(1A)	4*e*	0.2959	0.3309	0.2014	0.087
H(1B)	4*e*	0.2220	0.2946	0.1575	0.087

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*	0.77380(2)	0.68191(3)	0.06797(1)	0.0287(1)	0.0203(1)	0.0246(1)	–0.0007(1)	–0.00400(9)	–0.0013(1)
Cl(1)	4*e*	0.6647(1)	0.6754(2)	0.16557(9)	0.0341(8)	0.036(1)	0.0292(8)	0.0014(8)	0.0038(7)	–0.0031(8)
Cl(2)	4*e*	0.8868(1)	0.6821(2)	–0.02789(9)	0.0358(9)	0.0317(9)	0.0293(8)	–0.0041(8)	0.0045(7)	–0.0001(7)
Cl(3)	4*e*	0.8588(1)	0.8640(2)	0.1367(1)	0.039(1)	0.033(1)	0.050(1)	–0.0050(8)	–0.0055(8)	–0.0111(8)
Cl(4)	4*e*	0.6862(1)	0.8687(2)	–0.0027(1)	0.051(1)	0.024(1)	0.047(1)	0.0016(8)	–0.0092(9)	0.0019(8)
N(1)	4*e*	0.7021(3)	0.5136(6)	0.0151(3)	0.028(3)	0.026(3)	0.029(3)	–0.006(2)	–0.002(2)	–0.003(2)
C(1)	4*e*	0.6260(4)	0.5312(8)	–0.0349(4)	0.031(4)	0.029(4)	0.036(4)	–0.001(3)	–0.006(3)	–0.004(3)
C(2)	4*e*	0.5779(5)	0.4091(9)	–0.0626(4)	0.031(4)	0.050(5)	0.036(4)	–0.011(4)	–0.003(3)	–0.014(4)
C(3)	4*e*	0.6064(5)	0.2734(8)	–0.0399(4)	0.053(5)	0.037(5)	0.036(4)	–0.022(4)	0.008(4)	–0.008(3)
C(4)	4*e*	0.6839(5)	0.2559(8)	0.0100(4)	0.046(4)	0.027(4)	0.037(4)	–0.008(3)	0.014(3)	–0.005(3)
C(5)	4*e*	0.7322(5)	0.3787(7)	0.0378(4)	0.037(4)	0.019(4)	0.026(3)	–0.001(3)	0.005(3)	–0.003(3)
C(6)	4*e*	0.8139(4)	0.3797(7)	0.0925(3)	0.030(4)	0.027(4)	0.025(3)	0.006(3)	0.004(3)	0.003(3)
C(7)	4*e*	0.8613(5)	0.2541(8)	0.1187(4)	0.048(5)	0.028(4)	0.043(4)	0.008(4)	0.007(4)	0.006(3)
C(8)	4*e*	0.9385(5)	0.2644(9)	0.1697(4)	0.044(5)	0.047(5)	0.045(5)	0.017(4)	0.010(4)	0.027(4)
C(9)	4*e*	0.9673(5)	0.400(1)	0.1963(4)	0.041(4)	0.067(6)	0.031(4)	0.013(4)	–0.004(3)	0.017(4)
C(10)	4*e*	0.9212(5)	0.5252(9)	0.1708(4)	0.036(4)	0.049(5)	0.029(4)	0.000(4)	–0.005(3)	0.007(3)
C(11)	4*e*	0.8454(4)	0.5128(6)	0.1195(3)	0.023(3)	0.016(3)	0.022(3)	0.003(3)	0.003(2)	0.003(2)
N(2)	4*e*	0.2051(4)	0.6605(7)	0.1645(3)	0.035(3)	0.045(4)	0.031(3)	–0.003(3)	0.005(3)	0.003(3)
C(12)	4*e*	0.1310(5)	0.6801(9)	0.1146(4)	0.033(4)	0.037(5)	0.040(4)	0.000(4)	0.002(3)	–0.010(4)
C(13)	4*e*	0.1039(5)	0.816(1)	0.0942(4)	0.030(4)	0.061(6)	0.049(5)	–0.001(4)	0.000(3)	–0.017(4)
C(14)	4*e*	0.1535(5)	0.9333(9)	0.1247(4)	0.045(4)	0.036(5)	0.046(4)	0.010(4)	0.008(4)	0.004(4)
C(15)	4*e*	0.2297(5)	0.9102(8)	0.1762(4)	0.031(4)	0.038(5)	0.050(4)	–0.009(3)	0.000(3)	–0.004(4)
C(16)	4*e*	0.2565(5)	0.7714(8)	0.1967(4)	0.027(4)	0.034(4)	0.032(4)	–0.007(3)	0.007(3)	–0.004(3)
C(17)	4*e*	0.3386(5)	0.7345(8)	0.2487(4)	0.036(4)	0.033(4)	0.031(4)	–0.002(3)	0.004(3)	–0.009(3)
C(18)	4*e*	0.4174(5)	0.8253(8)	0.2500(4)	0.039(4)	0.041(5)	0.038(4)	–0.006(4)	0.004(3)	–0.009(4)
C(19)	4*e*	0.4964(5)	0.7921(9)	0.2962(4)	0.043(4)	0.053(6)	0.047(5)	–0.007(4)	0.006(4)	–0.020(4)
C(20)	4*e*	0.4975(5)	0.6690(9)	0.3416(4)	0.041(4)	0.054(6)	0.041(4)	0.003(4)	–0.013(3)	–0.012(4)
C(21)	4*e*	0.4204(5)	0.5781(9)	0.3416(4)	0.056(5)	0.038(5)	0.038(4)	0.002(4)	0.003(4)	–0.001(4)
C(22)	4*e*	0.3402(5)	0.6101(8)	0.2953(4)	0.042(4)	0.036(5)	0.035(4)	–0.007(3)	0.008(3)	–0.003(3)
O(1)	4*e*	0.2463(4)	0.3666(6)	0.1810(3)	0.086(4)	0.042(4)	0.045(3)	–0.006(3)	–0.013(3)	0.001(3)

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).

References

1. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
2. Farrugia, L. J.: ORTEP-3 for Windows - a version of ORTEP-III with a Graphical User Interface (GUI) J. Appl. Crystallogr. **30** (1997) 565.
3. Spek, A. L.: Single-crystal structure validation with the program PLATON. J. Appl. Crystallogr. **36** (2003) 7-13.