Crystal structure of chlorotris(4-phenylpyridine-κN)platinum(II) trichloro(4-phenylpyridine-κN)platinate(II), $[PtCl(C_{11}H_9N)_3][PtCl_3(C_{11}H_9N)]$

Kwang Ha*

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

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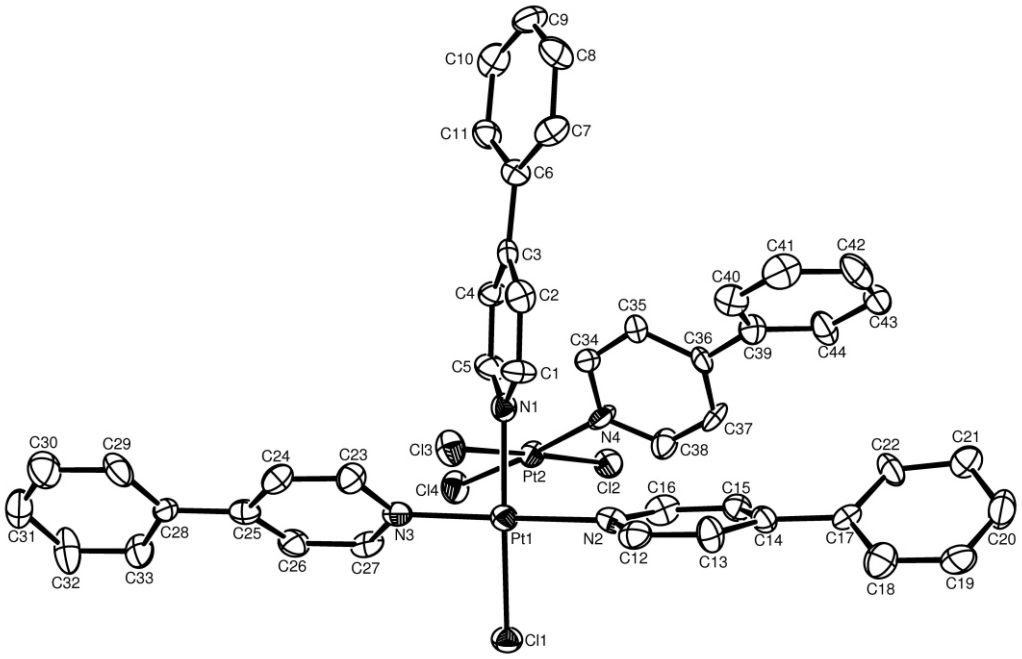

Abstract

$C_{44}H_{36}Cl_4N_4Pt_2$, orthorhombic, $P2_12_12_1$ (no. 19), a = 13.4463(6) Å, b = 16.3575(7) Å, c = 17.9570(8) Å, V = 3949.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.053, $wR_{ref}(F^2)$ = 0.103, T = 200 K.

Source of material

To a solution of K_2PtCl_4 (0.2070 g, 0.499 mmol) in H_2O (20 ml) and EtOH (10 ml) was added 4-phenylpyridine (phpy; 0.1558 g, 1.004 mmol) and refluxed for 3 h. The formed precipitate was separated by filtration, washed with H_2O and EtOH, and dried at 50 °C, to give a pale yellow powder (0.2595 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) = 1.2 U_{eq}(C). The highest peak (1.52 e Å^{-3}) and the deepest hole (–0.69 e Å^{-3}) in the difference Fourier map are located 0.03 Å and 1.67 Å from the atoms Pt2 and H23, respectively. The Flack parameter is –0.02(1) in the final cycles of refinement.

Discussion

The crystal structure of the title compound consists of a cationic Pt(II) complex $[PtCl(C_{11}H_9N)_3]^+$ and an anionic Pt(II) complex $[PtCl_3(C_{11}H_9N)]^-$. Each Pt(II) ion is four-coordinated in a slightly distorted square-planar environment. In the cationic complex, the Pt(II) ion is coordinated by one Cl^- anion and three N atoms from three phpy ligands, whereas in the anionic complex, the Pt(II) ion is coordinated by three Cl^- anions and one N atom from a phpy ligand. The Pt—N and Pt—Cl bond lengths are roughly equal, respectively (d(Pt—N) = 2.01(1) - 2.04(1) Å and d(Pt—Cl) = 2.283(4) - 2.301(3) Å). In the crystal structure, the phenyl rings are not parallel to their respective carrier pyridine rings, making dihedral angles of 16.9(8)°, 33.2(4)°, 8.5(3)° and 37.5(3)°. The compound displays numerous intra- and intermolecular π–π interactions between the adjacent six-membered rings. The centroid–centroid distance between Cg1 (the centroid of ring N4–C38) and Cg2^i (ring N3–C27, symmetry code i: 1–x,–½+y,½–z) is 3.615(7) Å, and the dihedral angle between the ring planes is 14.2(7)°. There are also intra- and intermolecular C–H···Cl hydrogen bonds with d(C···Cl) = 3.21(2) - 3.64(2) Å.

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

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.13 × 0.17 × 0.18 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	73.85 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	52.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	25024, 7738
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5119
$N(param)_{refined}$:	487
Programs:	SHELXS-97, SHELXL-97 [1], ORTEP-3 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.3217	0.5430	0.3792	0.054
H(2)	4*a*	0.2668	0.4594	0.4705	0.058
H(4)	4*a*	0.4417	0.2816	0.3899	0.049
H(5)	4*a*	0.4962	0.3718	0.3019	0.062
H(7)	4*a*	0.1790	0.3620	0.5241	0.060
H(8)	4*a*	0.1232	0.2707	0.6139	0.079
H(9)	4*a*	0.1992	0.1492	0.6320	0.068
H(10)	4*a*	0.3501	0.1216	0.5714	0.071
H(11)	4*a*	0.4142	0.2166	0.4889	0.056
H(12)	4*a*	0.2897	0.6477	0.2556	0.060
H(13)	4*a*	0.1279	0.6550	0.2160	0.059
H(15)	4*a*	0.1647	0.4449	0.1040	0.052
H(16)	4*a*	0.3306	0.4430	0.1421	0.061
H(18)	4*a*	0.0080	0.6749	0.1291	0.072
H(19)	4*a*	–0.1544	0.6816	0.0892	0.076
H(20)	4*a*	–0.2414	0.5585	0.0694	0.083
H(21)	4*a*	–0.1652	0.4320	0.0975	0.062
H(22)	4*a*	0.0010	0.4326	0.1283	0.062
H(23)	4*a*	0.5523	0.5619	0.3919	0.058
H(24)	4*a*	0.7038	0.5739	0.4461	0.061
H(26)	4*a*	0.8365	0.5055	0.2585	0.066
H(27)	4*a*	0.6824	0.4992	0.2017	0.053
H(29)	4*a*	0.8381	0.5639	0.5001	0.074
H(30)	4*a*	0.9894	0.5794	0.5576	0.091
H(31)	4*a*	1.1320	0.5693	0.4921	0.080
H(32)	4*a*	1.1257	0.5477	0.3616	0.093
H(33)	4*a*	0.9718	0.5338	0.3027	0.069
H(34)	4*a*	0.4641	0.2240	0.2460	0.065
H(35)	4*a*	0.2985	0.2319	0.2834	0.059
H(37)	4*a*	0.2219	0.2843	0.0718	0.055
H(38)	4*a*	0.3860	0.2687	0.0380	0.068
H(40)	4*a*	0.1786	0.3333	0.3061	0.067
H(41)	4*a*	0.0186	0.3648	0.3459	0.083
H(42)	4*a*	–0.1182	0.3250	0.2730	0.077
H(43)	4*a*	–0.0948	0.2599	0.1606	0.073
H(44)	4*a*	0.0682	0.2327	0.1204	0.070

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*a*	0.46465(4)	0.53439(3)	0.24509(3)	0.0370(3)	0.0438(3)	0.0439(3)	–0.0008(3)	0.0026(3)	0.0041(3)
Cl(1)	4*a*	0.5233(3)	0.6047(2)	0.1431(2)	0.053(2)	0.052(2)	0.041(2)	–0.005(2)	0.007(2)	0.005(2)
N(1)	4*a*	0.4131(8)	0.4658(7)	0.3316(5)	0.033(6)	0.041(7)	0.048(7)	0.004(7)	–0.003(5)	0.001(6)
N(2)	4*a*	0.3236(8)	0.5414(7)	0.2055(6)	0.050(8)	0.034(6)	0.037(6)	–0.005(6)	0.015(5)	–0.008(6)
N(3)	4*a*	0.6023(8)	0.5310(6)	0.2890(6)	0.051(8)	0.039(6)	0.039(6)	–0.004(6)	0.003(5)	0.005(6)
C(1)	4*a*	0.347(1)	0.4888(8)	0.3815(7)	0.045(9)	0.038(8)	0.051(9)	0.009(6)	0.019(7)	0.014(7)
C(2)	4*a*	0.314(1)	0.4390(8)	0.4355(8)	0.04(1)	0.05(1)	0.06(1)	0.007(7)	0.004(7)	–0.006(8)
C(3)	4*a*	0.3459(9)	0.3575(8)	0.4415(7)	0.024(8)	0.049(9)	0.031(7)	–0.001(6)	–0.002(6)	–0.009(7)
C(4)	4*a*	0.415(1)	0.3355(7)	0.3893(7)	0.042(8)	0.036(7)	0.046(8)	0.006(7)	0.001(8)	0.009(7)
C(5)	4*a*	0.447(1)	0.3894(9)	0.3366(7)	0.05(1)	0.06(1)	0.042(8)	–0.010(8)	0.015(7)	0.001(8)
C(6)	4*a*	0.304(1)	0.2995(8)	0.4961(7)	0.043(9)	0.038(8)	0.028(7)	0.000(7)	0.012(6)	–0.012(6)
C(7)	4*a*	0.216(1)	0.3137(8)	0.5339(8)	0.07(1)	0.038(9)	0.042(9)	0.009(8)	–0.003(8)	–0.001(7)
C(8)	4*a*	0.181(1)	0.258(1)	0.5862(8)	0.04(1)	0.08(1)	0.07(1)	–0.01(1)	0.017(8)	0.00(1)
C(9)	4*a*	0.227(1)	0.188(1)	0.5985(8)	0.06(1)	0.06(1)	0.05(1)	0.013(9)	0.005(8)	0.021(8)
C(10)	4*a*	0.316(1)	0.1712(9)	0.5622(8)	0.08(1)	0.038(9)	0.06(1)	0.004(9)	–0.015(9)	–0.002(8)
C(11)	4*a*	0.353(1)	0.2276(9)	0.5126(7)	0.037(9)	0.051(9)	0.054(9)	0.002(8)	0.006(7)	0.002(9)
C(12)	4*a*	0.264(1)	0.6055(8)	0.2249(7)	0.07(1)	0.046(9)	0.035(8)	–0.009(8)	–0.003(7)	–0.002(7)
C(13)	4*a*	0.168(1)	0.6100(8)	0.2012(8)	0.034(9)	0.043(9)	0.07(1)	0.007(7)	–0.004(8)	–0.012(8)
C(14)	4*a*	0.127(1)	0.5498(8)	0.1557(7)	0.038(9)	0.041(9)	0.040(8)	–0.008(7)	–0.001(6)	–0.001(7)
C(15)	4*a*	0.189(1)	0.4874(8)	0.1351(7)	0.05(1)	0.039(9)	0.046(9)	–0.001(7)	0.000(7)	0.001(7)
C(16)	4*a*	0.288(1)	0.4853(9)	0.1592(8)	0.06(1)	0.05(1)	0.043(8)	0.018(8)	0.014(7)	–0.006(8)
C(17)	4*a*	0.021(1)	0.5530(8)	0.1303(6)	0.05(1)	0.040(8)	0.029(7)	0.007(8)	–0.003(6)	0.007(6)
C(18)	4*a*	–0.027(1)	0.6256(9)	0.1200(8)	0.06(1)	0.07(1)	0.053(9)	0.007(9)	–0.003(9)	–0.004(8)
C(19)	4*a*	–0.123(1)	0.6303(9)	0.0974(9)	0.06(1)	0.05(1)	0.08(1)	0.007(9)	–0.001(9)	0.025(9)
C(20)	4*a*	–0.175(1)	0.557(1)	0.0867(8)	0.04(1)	0.11(2)	0.05(1)	0.01(1)	–0.010(8)	0.01(1)
C(21)	4*a*	–0.129(1)	0.4819(9)	0.1012(8)	0.06(1)	0.04(1)	0.051(9)	0.004(8)	–0.008(8)	–0.002(8)
C(22)	4*a*	–0.032(1)	0.4833(9)	0.1208(7)	0.039(9)	0.07(1)	0.049(8)	–0.022(8)	0.002(8)	–0.011(8)
C(23)	4*a*	0.610(1)	0.5519(8)	0.3633(7)	0.04(1)	0.06(1)	0.047(9)	0.009(7)	0.002(7)	0.001(7)
C(24)	4*a*	0.701(1)	0.5581(8)	0.3952(8)	0.06(1)	0.06(1)	0.033(8)	0.007(8)	–0.002(8)	0.001(8)
C(25)	4*a*	0.791(1)	0.5432(8)	0.3599(7)	0.06(1)	0.029(8)	0.043(8)	0.000(7)	0.007(7)	0.000(7)
C(26)	4*a*	0.779(1)	0.5193(9)	0.2867(8)	0.04(1)	0.07(1)	0.06(1)	0.000(8)	0.014(7)	0.011(8)
C(27)	4*a*	0.686(1)	0.5146(7)	0.2526(7)	0.08(1)	0.034(8)	0.018(6)	0.001(7)	0.005(8)	–0.012(7)
C(28)	4*a*	0.889(1)	0.5494(7)	0.3977(7)	0.05(1)	0.035(8)	0.035(7)	–0.008(6)	–0.009(7)	0.006(7)
C(29)	4*a*	0.897(1)	0.5612(9)	0.4709(8)	0.05(1)	0.08(1)	0.06(1)	–0.029(9)	0.014(8)	–0.015(9)
C(30)	4*a*	0.987(1)	0.570(1)	0.506(1)	0.05(1)	0.11(1)	0.07(1)	–0.01(1)	0.01(1)	–0.00(1)
C(31)	4*a*	1.070(1)	0.5643(8)	0.468(1)	0.07(1)	0.05(1)	0.09(1)	0.003(9)	–0.03(1)	–0.008(9)
C(32)	4*a*	1.066(1)	0.551(1)	0.390(1)	0.04(1)	0.11(1)	0.08(1)	–0.00(1)	–0.00(1)	–0.04(1)
C(33)	4*a*	0.976(1)	0.543(1)	0.3548(7)	0.06(1)	0.08(1)	0.034(8)	–0.01(1)	0.005(8)	–0.016(8)

Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt(2)	4*a*	0.58484(4)	0.23049(3)	0.10975(3)	0.0420(3)	0.0540(3)	0.0398(3)	0.0056(3)	0.0007(3)	0.0003(3)
Cl(2)	4*a*	0.5398(3)	0.1508(2)	0.0108(2)	0.061(3)	0.071(2)	0.042(2)	–0.007(2)	0.007(2)	–0.010(2)
Cl(3)	4*a*	0.6333(3)	0.3134(3)	0.2064(2)	0.061(3)	0.077(3)	0.060(2)	–0.005(2)	0.001(2)	–0.023(2)
Cl(4)	4*a*	0.7481(3)	0.2059(3)	0.0829(2)	0.044(3)	0.104(3)	0.056(2)	0.002(2)	0.007(2)	–0.005(2)
N(4)	4*a*	0.4406(8)	0.2464(7)	0.1385(5)	0.052(9)	0.069(8)	0.033(6)	0.012(6)	–0.005(5)	0.020(6)
C(34)	4*a*	0.414(1)	0.2362(8)	0.2102(7)	0.06(1)	0.057(9)	0.044(8)	–0.023(9)	–0.015(8)	0.017(7)
C(35)	4*a*	0.316(1)	0.2430(8)	0.2331(7)	0.037(9)	0.06(1)	0.050(9)	–0.012(7)	–0.008(7)	0.005(8)
C(36)	4*a*	0.242(1)	0.2664(8)	0.1819(7)	0.031(8)	0.040(8)	0.052(9)	–0.014(7)	–0.006(6)	–0.001(7)
C(37)	4*a*	0.270(1)	0.2729(8)	0.1090(7)	0.052(9)	0.054(8)	0.031(7)	0.020(7)	–0.016(7)	0.007(8)
C(38)	4*a*	0.369(1)	0.2633(9)	0.0890(7)	0.05(1)	0.09(1)	0.037(8)	0.005(9)	–0.001(7)	–0.001(8)
C(39)	4*a*	0.141(1)	0.2819(9)	0.2087(8)	0.05(1)	0.05(1)	0.051(9)	–0.004(8)	–0.005(7)	–0.002(8)
C(40)	4*a*	0.124(1)	0.3192(8)	0.2755(8)	0.06(1)	0.044(9)	0.06(1)	0.000(8)	0.004(8)	–0.005(8)
C(41)	4*a*	0.029(2)	0.3371(9)	0.3000(9)	0.08(1)	0.07(1)	0.06(1)	0.02(1)	0.01(1)	–0.001(9)
C(42)	4*a*	–0.053(1)	0.3138(8)	0.256(1)	0.03(1)	0.06(1)	0.10(1)	0.000(7)	0.02(1)	–0.00(1)
C(43)	4*a*	–0.039(1)	0.2756(9)	0.1902(9)	0.04(1)	0.06(1)	0.08(1)	–0.009(9)	–0.006(9)	0.019(9)
C(44)	4*a*	0.058(1)	0.2596(8)	0.1666(9)	0.04(1)	0.047(9)	0.09(1)	–0.008(7)	–0.005(8)	–0.002(8)

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