Crystal structure of (acetonitrile-κ*N*)chlorido[2-(2-pyridyl)phenyl-κ^2*N*,*C*] platinum(II), $C_{13}H_{11}ClN_2Pt$

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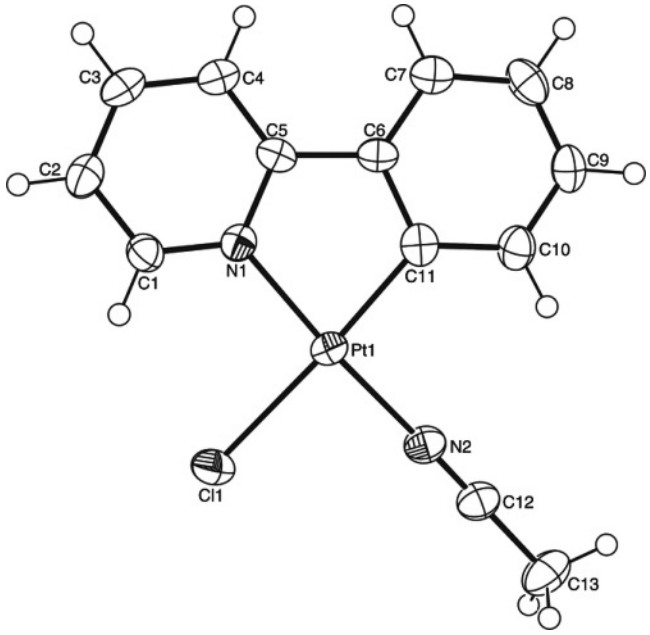

Abstract

$C_{13}H_{11}ClN_2Pt$, monoclinic, $P2_1/n$ (no. 14), a = 11.424(1) Å, b = 5.4949(7) Å, c = 20.060(3) Å, β = 104.987(2)°, V = 1216.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.0225, $wR_{ref}(F^2)$ = 0.0525, T = 200 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.06×0.08×0.12 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	117.31 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6980, 2346
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2013
$N(param)_{refined}$:	155
Programs:	SHELX [9], ORTEP-3 [10], PLATON [11]

Source of material

To a solution of K_2PtCl_4 (0.208 g, 0.50 mmol) and dipicolinic acid (0.085 g, 0.51 mmol) in H_2O (20 ml) and MeOH (10 ml) was added 2-phenylpyridine (*ppy*; 0.166 g, 1.07 mmol) and stirred for 48 h at room temperature. The formed brown precipitate was removed by filtration. The solvent of the filtrate was evaporated, the residue recrystallized from *N*,*N*-dimethylformamide/ether and filtered, to give a yellow powder (0.297 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 (aromatic) or 0.98 Å (CH_3) and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The highest peak (1.36 e·Å^{-3}) and the deepest hole (–0.82 e·Å^{-3}) in the difference Fourier map are located 1.06 Å and 1.83 Å from the atoms C6 and C5, respectively.

Discussion

This contribution is part of my continuing interest in the structural chemistry of Pt-*ppy* complexes [1-6]. Single crystals of the title complex [PtCl($C_{11}H_8N$)(CH_3CN)] were unexpectedly obtained during crystallization from a CH_3CN solution of the reaction product prepared by the reaction of K_2PtCl_4, dipicolinic acid and *ppy*. The complex is isomorphous with the previously reported iodido-Pt(II) complex [PtI($C_{11}H_8N$)(CH_3CN)] [3]. The crystal structure of the related chlorido-Pt(II) complex [PtCl($C_{11}H_8N$)(*ppy*)] has been investigated previously [7]. In the title crystal structure, the central Pt(II) ion is four-coordinated by one N atom and one C atom from the anionic 2-(2-pyridyl)phenyl ligand, one N atom of a CH_3CN molecule and one chlorido ligand in a distorted square-planar coordination geometry with the N atoms *trans* to each other. The main contribution to the distortion is made by the tight chelate angle of <N1–Pt1–C11 = 81.44(18)°, which results in non-linear *trans* axes (<N1–Pt1–N2 = 175.70(16)° and <C11–Pt1–Cl1 = 175.10(14)°). The two Pt–N bond lengths are similar with d(Pt1–N1/N2) = 2.012(4) Å and 1.980(4) Å. Because of the strong *trans* influence of the coordinated C11 atom, the Pt1–Cl1 bond length (2.4113(13) Å) is considerably longer than that reported for [$PtCl_2$(2,2'-bipyridine)] (2.306(2) Å) [8]. In the crystal, the pyridyl and phenyl rings of the chelating ligand are slightly inclined to the least-squares plane of the $PtClN_2C$ coordination unit (maximum deviation = 0.053(2) Å) at 10.1(2)° and 8.6(2)°, respectively. The dihedral angle between the two ring planes is 6.5(3)°.

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.9262	0.6952	0.4547	0.037
H(2)	4*e*	1.0624	0.9796	0.4331	0.042
H(3)	4*e*	0.9898	1.3007	0.3571	0.044
H(4)	4*e*	0.7851	1.3201	0.3005	0.038
H(7)	4*e*	0.5838	1.2865	0.2447	0.040

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(8)	4*e*	0.3789	1.2160	0.1921	0.046
H(9)	4*e*	0.2843	0.8730	0.2220	0.044
H(10)	4*e*	0.3868	0.6109	0.3066	0.042
H(13A)	4*e*	0.3756	–0.0813	0.4186	0.069
H(13B)	4*e*	0.2765	0.1305	0.3991	0.069
H(13C)	4*e*	0.3470	0.0844	0.4781	0.069

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*	0.65263(2)	0.60291(3)	0.40793(1)	0.0284(1)	0.0239(1)	0.0289(1)	–0.00156(8)	0.01030(8)	0.00072(8)
Cl(1)	4*e*	0.7836(1)	0.4114(2)	0.50788(7)	0.0403(7)	0.0409(7)	0.0431(8)	0.0020(6)	0.0099(6)	0.0161(6)
N(1)	4*e*	0.7786(3)	0.8329(7)	0.3907(2)	0.027(2)	0.032(2)	0.021(2)	–0.003(2)	0.009(2)	–0.003(2)
N(2)	4*e*	0.5201(4)	0.3863(7)	0.4186(2)	0.036(2)	0.035(2)	0.031(3)	–0.002(2)	0.014(2)	0.002(2)
C(1)	4*e*	0.8973(4)	0.8228(9)	0.4227(2)	0.029(3)	0.036(3)	0.027(3)	0.002(2)	0.007(2)	0.003(2)
C(2)	4*e*	0.9785(5)	0.992(1)	0.4106(3)	0.031(3)	0.041(3)	0.033(3)	–0.008(2)	0.009(2)	–0.005(2)
C(3)	4*e*	0.9357(5)	1.180(1)	0.3652(3)	0.040(3)	0.036(3)	0.039(3)	–0.010(2)	0.019(2)	–0.001(2)
C(4)	4*e*	0.8148(4)	1.1909(9)	0.3317(3)	0.037(3)	0.029(3)	0.033(3)	–0.004(2)	0.013(2)	0.001(2)
C(5)	4*e*	0.7359(4)	1.0137(8)	0.3435(2)	0.035(3)	0.022(2)	0.020(3)	0.004(2)	0.008(2)	–0.002(2)
C(6)	4*e*	0.6074(4)	0.9921(9)	0.3093(2)	0.035(3)	0.023(2)	0.027(3)	0.002(2)	0.012(2)	0.000(2)
C(7)	4*e*	0.5439(5)	1.1499(8)	0.2579(3)	0.041(3)	0.028(3)	0.032(3)	0.002(2)	0.012(2)	–0.001(2)
C(8)	4*e*	0.4230(5)	1.1071(9)	0.2262(3)	0.035(3)	0.045(3)	0.035(3)	0.017(2)	0.006(2)	0.001(2)
C(9)	4*e*	0.3668(5)	0.9047(9)	0.2447(3)	0.024(3)	0.043(3)	0.040(3)	0.003(2)	0.003(2)	–0.005(2)
C(10)	4*e*	0.4279(4)	0.7486(9)	0.2951(3)	0.027(3)	0.036(3)	0.041(3)	0.002(2)	0.009(2)	–0.005(2)
C(11)	4*e*	0.5491(4)	0.7869(9)	0.3300(2)	0.030(3)	0.032(3)	0.027(3)	0.003(2)	0.010(2)	–0.008(2)
C(12)	4*e*	0.4474(5)	0.2528(9)	0.4237(3)	0.039(3)	0.033(3)	0.033(3)	–0.002(3)	0.015(2)	–0.001(2)
C(13)	4*e*	0.3541(5)	0.083(1)	0.4304(3)	0.052(4)	0.047(4)	0.045(4)	–0.017(3)	0.024(3)	–0.004(3)

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