Crystal structure of dichlorobis(4-phenylpyridine-κN)-platinum(II) — water (1:1), $PtCl_2(C_{11}H_9N)_2 \cdot H_2O$

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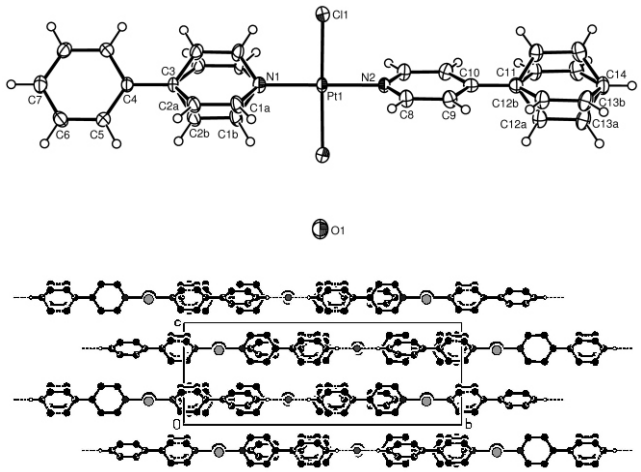

Abstract

$C_{22}H_{20}Cl_2N_2OPt$, monoclinic, *C*12/*c*1 (no. 15), a = 9.4199(5) Å, b = 23.687(1) Å, c = 8.8435(5) Å, β = 101.257(1)°, V = 1935.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.026, $wR_{ref}(F^2)$ = 0.072, 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 (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 an *N*,*N*-dimethylformamide (DMF) solution at 70 °C.

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). One pyridyl ring (N1-C3) and one phenyl ring (C11-C14) displayed relatively large displacement factors so that the rings appear to be partially disordered. The C1, C2, C12 and C13 atoms were refined anisotropically as disordered over two sites using EADP instruction for the atoms [1]. The site occupancy factors are 0.55(1) for the C1A and C2A atoms, and 0.545(7) for the C12A and C13A atoms, respectively. The H atoms of the solvent H_2O molecule could neither be located from Fourier difference maps nor added geometrically. The highest peak (0.99 e Å^{-3}) and the deepest hole (–1.45 e Å^{-3}) in the difference Fourier map are located 0.88 Å and 0.45 Å from the atoms Pt1 and O1, respectively.

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Discussion

The asymmetric unit of the title crystal structure contains one half of a neutral Pt(II) complex and one half of a water solvent molecule (figure, top). The compound is disposed about a twofold rotation axis running in the [010] direction passing through the Pt1, N1, C3, C4, C7, N2, C10, C11, C14 and O1 atoms. In the complex, the central Pt(II) ion has a *trans*-Cl_2N_2 square-planar coordination defined by two N atoms from two distinct 4-phenylpyridine ligands and two Cl^- anions. The two d(Pt—N) are almost equal with 2.021(5) Å and 2.032(5) Å, respectively. In the crystal structure, the pyridyl ring N1-C3 and the phenyl ring C11-C14 are disordered over two sites. The dihedral angles between the major and minor rings are 31.2(9)° for the ring N1-C3 and 40.5(6)° for the ring C11-C14, respectively. The molecules are stacked in columns along [100] with d(Pt···Pt) = 9.4199(5) Å (= length of a axis) and display numerous intermolecular π–π interactions between the adjacent six-membered rings. For Cg1 (the centroid of ring N1-C1A-C2A-C3) and Cg1^i (symmetry code i: –x,–y,1–z), the centroid-centroid distance is 4.505(6) Å, the planes of Cg1 and Cg1^i are parallel and their centres are shifted by 3.580 Å. In addition, the complex and the H_2O molecule are linked by intermolecular C–H···O hydrogen bonds with d(C7–H7···O1) = 2.759(8) Å and d(C14–H14···O1) = 2.679(8) Å forming a one-dimensional chain structure along [010] (figure, bottom).

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.17 × 0.22 × 0.29 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	75.43 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	56.54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6690, 2286
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1867
$N(param)_{refined}$:	146
Programs:	SHELXS-97, SHELXL-97 [1], ORTEP-3 [2], PLATON [3]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	8*f*	0.55	0.1347	0.0320	0.9508	0.039
H(2A)	8*f*	0.55	0.1332	–0.0659	0.9603	0.040
H(1B)	8*f*	0.45	0.2043	0.0291	0.8630	0.039
H(2B)	8*f*	0.45	0.2043	–0.0672	0.8819	0.040
H(5)	8*f*		0.2120	–0.1520	0.8565	0.039
H(6)	8*f*		0.2113	–0.2498	0.8498	0.040
H(7)	4*e*		0	–0.3002	¼	0.041
H(8)	8*f*		0.0994	0.2191	0.9673	0.037

Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(9)	8*f*		0.103	0.317	0.9730	0.037
H(12A)	8*f*	0.545	0.2029	0.4015	0.8782	0.038
H(13A)	8*f*	0.545	0.2088	0.5007	0.8693	0.044
H(12B)	8*f*	0.455	0.1021	0.4022	0.9733	0.038
H(13B)	8*f*	0.455	0.11	0.5003	0.9734	0.044
H(14)	4*e*		0	0.5505	¾	0.042

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt(1)	4*e*		0	0.125593(9)	¾	0.0305(2)	0.0151(1)	0.0285(1)	0	0.00262(9)	0
Cl(1)	8*f*		–0.2408(1)	0.12652(5)	0.7675(2)	0.0321(6)	0.0248(6)	0.0563(8)	0.0019(4)	0.0091(5)	0.0005(5)
N(1)	4*e*		0	0.0398(2)	¾	0.033(3)	0.021(3)	0.026(3)	0	0.002(2)	0
N(2)	4*e*		0	0.2109(2)	¾	0.029(3)	0.018(2)	0.028(3)	0	0.004(2)	0
C(1A)	8*f*	0.55(1)	0.078(1)	0.0115(5)	0.868(2)	0.038(7)	0.020(3)	0.036(7)	0.005(4)	–0.004(4)	–0.009(4)
C(2A)	8*f*	0.55	0.079(1)	–0.0469(6)	0.873(2)	0.032(7)	0.024(3)	0.039(7)	0.005(4)	–0.003(3)	0.002(4)
C(1B)	8*f*	0.45	0.119(2)	0.0091(6)	0.818(2)	0.038(7)	0.020(3)	0.036(7)	0.005(4)	–0.004(4)	–0.009(4)
C(2B)	8*f*	0.45	0.122(2)	–0.0480(7)	0.826(2)	0.032(7)	0.024(3)	0.039(7)	0.005(4)	–0.003(3)	0.002(4)
C(3)	4*e*		0	–0.0783(2)	¾	0.028(3)	0.013(3)	0.036(3)	0	0.006(3)	0
C(4)	4*e*		0	–0.1411(3)	¾	0.030(3)	0.018(3)	0.029(3)	0	0.008(3)	0
C(5)	8*f*		0.1258(5)	–0.1715(2)	0.8123(6)	0.027(3)	0.026(2)	0.043(3)	–0.002(2)	0.003(2)	0.001(2)
C(6)	8*f*		0.1246(5)	–0.2298(2)	0.8097(6)	0.032(3)	0.028(2)	0.038(3)	0.007(2)	0.003(2)	0.001(2)
C(7)	4*e*		0	–0.2601(3)	¾	0.042(4)	0.023(3)	0.039(4)	0	0.010(3)	0
C(8)	8*f*		0.0581(5)	0.2395(2)	0.8768(5)	0.038(3)	0.024(2)	0.028(2)	0.001(2)	0.002(2)	–0.001(2)
C(9)	8*f*		0.0602(5)	0.2981(2)	0.8806(5)	0.039(3)	0.028(2)	0.024(2)	0.003(2)	0.001(2)	–0.003(2)
C(10)	4*e*		0	0.3296(3)	¾	0.025(3)	0.030(3)	0.030(3)	0	0.007(3)	0
C(11)	4*e*		0	0.3915(3)	¾	0.032(4)	0.025(3)	0.034(4)	0	0.009(3)	0
C(12A)	8*f*	0.545(7)	0.121(1)	0.4215(4)	0.825(1)	0.042(5)	0.026(3)	0.025(4)	0.002(3)	0.000(3)	0.001(3)
C(13A)	8*f*	0.545	0.124(1)	0.4809(4)	0.822(1)	0.054(6)	0.029(3)	0.025(5)	–0.009(4)	0.004(3)	–0.006(3)
C(12B)	8*f*	0.455	0.061(1)	0.4222(5)	0.882(1)	0.042(5)	0.026(3)	0.025(4)	0.002(3)	0.000(3)	0.001(3)
C(13B)	8*f*	0.455	0.065(1)	0.4806(5)	0.883(1)	0.054(6)	0.029(3)	0.025(5)	–0.009(4)	0.004(3)	–0.006(3)
C(14)	4*e*		0	0.5104(3)	¾	0.044(4)	0.020(3)	0.043(4)	0	0.012(3)	0
O(1)	4*e*		½	0.1235(2)	¾	0.053(4)	0.034(3)	0.053(4)	0	0.003(3)	0

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