Crystal structure of bis(benzo[*h*]quinolinium) hexachloroplatinate(IV) dihydrate, $[C_{13}H_{10}N]_2[PtCl_6] \cdot 2H_2O$

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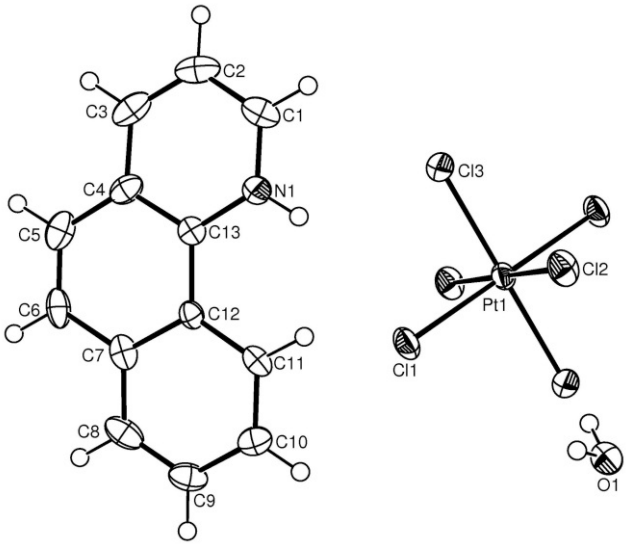

Abstract

$C_{26}H_{24}Cl_6N_2O_2Pt$, triclinic, $P\bar{1}$ (no. 2), a = 7.222(3) Å, b = 9.429(4) Å, c = 10.814(4) Å, α = 77.275(8)°, β = 74.195(8)°, γ = 80.075(7)°, V = 686.2 Å^3, Z = 1, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 0.075, T = 200 K.

Source of material

To a solution of K_2PtCl_6 (0.2429 g, 0.500 mmol) in H_2O (30 ml) was added benzo[*h*]quinoline (0.1807 g, 1.008 mmol), and the mixture was refluxed for 3 h. After addition of EtOH (20 ml) to the reaction mixture, the formed precipitate was then separated by filtration, washed with EtOH and dried at 50 °C, to give a yellow powder (0.1722 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a CH_3NO_2 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 molecules were located from the difference Fourier map and then allowed to ride on their parent O atoms 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 (0.96 e Å^{-3}) and the deepest hole (–1.06 e Å^{-3}) in the difference Fourier map are located 1.21 Å and 1.02 Å from the Pt1 atom, respectively.

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Discussion

The asymmetric unit of the title crystal structure contains a protonated benzo[*h*]quinolinium cation, half of an anionic Pt(IV) complex $[PtCl_6]^{2-}$ and a solvent water molecule. The octahedral $[PtCl_6]^{2-}$ complex is located on an inversion center. The distances d(Pt—Cl) are nearly equivalent (2.320(2) Å, 2.323(2) Å and 2.330(2) Å). These values are comparable to those observed in the analogous acridinium compound $(C_{13}H_{10}N)_2[PtCl_6] \cdot 2H_2O$ [1]. In the crystal structure, the complex ions and the water molecules are linked by intermolecular O–H···Cl hydrogen bonds with d(O···Cl) = 3.352(5) Å, 3.389(4) Å and 3.515(5) Å, thereby forming a chain-like structure along [100]. Each water molecule, as an H-bond acceptor, is linked to the benzo[*h*]quinolinium N–H group with d(N1–H1N···O1) = 2.753(6) Å. In addition, the nearly planar cations are stacked in columns also along [100]. When viewed down [100], the successive cations are stacked in the opposite direction. In the columns, numerous intermolecular π–π interactions between adjacent six-membered rings are present. For Cg1 (the centroid of ring N1-C13) and Cg2^i (ring C4-C13, symmetry code i: 1–x,2–y,–z), the centroid-centroid distance is 3.598(4) Å and the dihedral angle between the ring planes is 1.9(3)°.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.06 × 0.08 × 0.27 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	57.26 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	52.16°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4234, 2599
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2506
$N(param)_{refined}$:	169
Programs:	SHELXS-97, SHELXL-97 [2], ORTEP-3 [3], PLATON [4]

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

Atom	Site	x	y	z	U_{iso}
H(1N)	2*i*	0.1076	0.9049	0.2773	0.032
H(1)	2*i*	0.1241	1.0801	0.3799	0.041
H(2)	2*i*	0.2545	1.2941	0.2573	0.047
H(3)	2*i*	0.3458	1.3214	0.0310	0.044
H(5)	2*i*	0.3844	1.2227	–0.1734	0.039
H(6)	2*i*	0.3600	1.0355	–0.2698	0.037
H(8)	2*i*	0.2987	0.7805	–0.2504	0.041
H(9)	2*i*	0.2048	0.5599	–0.1260	0.040
H(10)	2*i*	0.0979	0.5355	0.1010	0.036
H(11)	2*i*	0.0875	0.7289	0.2036	0.029
H(1A)	2*i*	–0.0511	0.2794	0.5335	0.052
H(1B)	2*i*	0.1189	0.2595	0.5557	0.052

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)	1*h*	½	½	½	0.0274(2)	0.0290(2)	0.0184(2)	–0.0091(1)	–0.0040(1)	–0.0026(1)
Cl(1)	2*i*	0.3930(2)	0.5382(2)	0.3103(1)	0.0369(8)	0.0409(9)	0.0205(7)	–0.0074(7)	–0.0123(7)	–0.0027(7)
Cl(2)	2*i*	0.1805(2)	0.5058(2)	0.6218(2)	0.0244(8)	0.0469(9)	0.0264(8)	–0.0093(7)	0.0009(6)	–0.0061(7)
Cl(3)	2*i*	0.4814(2)	0.7523(2)	0.4839(2)	0.054(1)	0.0274(8)	0.0263(8)	–0.0113(7)	–0.0036(7)	–0.0033(7)
N(1)	2*i*	0.1534(7)	0.9852(5)	0.2287(5)	0.030(3)	0.025(3)	0.023(3)	–0.004(2)	–0.004(2)	–0.006(2)
C(1)	2*i*	0.1664(9)	1.0915(7)	0.2875(7)	0.031(3)	0.044(4)	0.032(4)	–0.003(3)	–0.006(3)	–0.019(3)
C(2)	2*i*	0.2414(9)	1.2192(7)	0.2152(7)	0.031(4)	0.033(4)	0.061(5)	–0.002(3)	–0.015(3)	–0.024(4)
C(3)	2*i*	0.2960(9)	1.2341(7)	0.0817(7)	0.026(3)	0.021(3)	0.060(5)	–0.001(3)	–0.010(3)	–0.005(3)
C(4)	2*i*	0.2805(8)	1.1244(6)	0.0182(6)	0.022(3)	0.021(3)	0.041(4)	–0.001(2)	–0.010(3)	–0.005(3)
C(5)	2*i*	0.3369(8)	1.1358(7)	–0.1202(6)	0.022(3)	0.031(3)	0.037(4)	–0.003(3)	–0.002(3)	0.005(3)
C(6)	2*i*	0.3240(8)	1.0245(7)	–0.1772(6)	0.025(3)	0.042(4)	0.017(3)	–0.002(3)	0.002(3)	0.005(3)
C(7)	2*i*	0.2569(8)	0.8910(6)	–0.1000(6)	0.015(3)	0.036(3)	0.022(3)	0.004(2)	–0.004(2)	–0.003(3)
C(8)	2*i*	0.2578(8)	0.7707(7)	–0.1579(6)	0.023(3)	0.054(4)	0.030(4)	0.002(3)	–0.005(3)	–0.022(3)
C(9)	2*i*	0.2011(8)	0.6398(7)	–0.0847(6)	0.028(3)	0.038(4)	0.035(4)	–0.003(3)	–0.002(3)	–0.020(3)
C(10)	2*i*	0.1383(8)	0.6256(6)	0.0501(6)	0.030(3)	0.025(3)	0.037(4)	–0.002(3)	–0.008(3)	–0.009(3)
C(11)	2*i*	0.1337(8)	0.7401(6)	0.1114(6)	0.021(3)	0.034(3)	0.020(3)	0.001(2)	–0.006(2)	–0.008(3)
C(12)	2*i*	0.1969(8)	0.8742(6)	0.0381(5)	0.020(3)	0.026(3)	0.017(3)	–0.004(2)	–0.003(2)	–0.003(2)
C(13)	2*i*	0.2077(8)	0.9951(6)	0.0967(6)	0.018(3)	0.025(3)	0.023(3)	–0.001(2)	–0.005(2)	–0.005(3)
O(1)	2*i*	0.0260(6)	0.2125(4)	0.5642(4)	0.041(3)	0.037(2)	0.028(2)	–0.010(2)	–0.009(2)	–0.006(2)

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