Crystal structure of bis(quinolinium) hexachloroplatinate(IV) dihydrate, $[C_9H_8N]_2[PtCl_6] \cdot 2H_2O$

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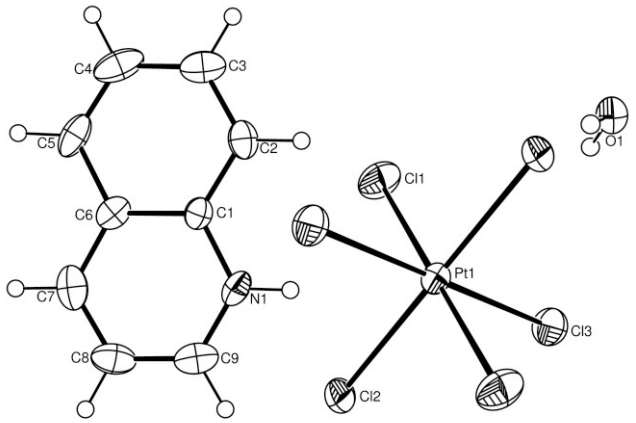

Abstract

$C_{18}H_{20}Cl_6N_2O_2Pt$, triclinic, $P\bar{1}$ (no. 2), a = 7.2602(6) Å, b = 8.0823(7) Å, c = 10.6370(9) Å, α = 78.774(2)°, β = 70.177(2)°, γ = 79.724(2)°, V = 571.7 Å^3, Z = 1, $R_{gt}(F)$ = 0.042, $wR_{ref}(F^2)$ = 0.106, T = 200 K.

Source of material

To a solution of K_2PtCl_6 (0.2432 g, 0.500 mmol) in H_2O (30 ml) was added quinoline (0.1569 g, 1.215 mmol), and the mixture was refluxed for 3 h. The formed brown precipitate was removed by filtration, and the solvent of the filtrate was evaporated. The residue was washed with H_2O/acetone (1:5) and dried at 50 °C, to give a yellow powder (0.2072 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 molecules were located from the difference Fourier map 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 (2.72 e Å^{-3}) and the deepest hole (–1.14 e Å^{-3}) in the difference Fourier map are located 1.70 Å and 1.01 Å from the atoms H1A and Pt1, respectively.

Discussion

The asymmetric unit of the title crystal strucutre contains a protonated quinolinium cation, half of an anionic Pt(IV) complex $[PtCl_6]^{2-}$ and a solvent water molecule. The octahedral $[PtCl_6]^{2-}$ dianion is located on an inversion center. The distances d(Pt—Cl) are nearly equal (2.313(2) Å, 2.318(2) Å and 2.320(2) Å). These values are similar to those observed in the analogous compound $(C_{13}H_{10}N)_2[PtCl_6] \cdot 2H_2O$ [1]. In the crystal structure, the nearly planar cations are stacked in columns along [100]. When viewed down [100], the successive cations are stacked in the opposite direction. In the columns, several intermolecular π–π interactions between adjacent six-membered rings are present. The distance between Cg1 (the centroid of ring C1-C6) and Cg1^{i} (symmetry code i: –x,2–y,–z) is 3.521(6) Å, and the ring planes are parallel and shifted by 0.970 Å. Moreover, the complex ions and the water molecules are linked by intermolecular O–H···Cl hydrogen bonds with d(O···Cl) = 3.325(7) Å and 3.423(7) Å, to form chains also running along [100], and each water molecule, as an H-bond acceptor, is linked to the quinolinium N–H group with d(N1–H1···O1) = 2.715(9) Å.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.04 × 0.08 × 0.31 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	68.56 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3512, 2153
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2097
$N(param)_{refined}$:	133
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(1)	2*i*	0.0889	0.9234	0.2913	0.045
H(2)	2*i*	0.1037	0.7034	0.1615	0.045
H(3)	2*i*	0.1932	0.6231	–0.0501	0.057
H(4)	2*i*	0.3229	0.8126	–0.2441	0.064
H(5)	2*i*	0.3792	1.0780	–0.2291	0.053
H(7)	2*i*	0.3588	1.2968	–0.0790	0.053
H(8)	2*i*	0.2713	1.3667	0.1348	0.058
H(9)	2*i*	0.1316	1.1757	0.3209	0.060
H(1A)	2*i*	–0.0294	0.2927	0.5492	0.065
H(1B)	2*i*	0.1644	0.2089	0.5362	0.065

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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	0.0312(3)	0.0290(3)	0.0206(2)	–0.0075(2)	–0.0064(2)	–0.0025(2)
Cl(1)	2*i*	0.3525(4)	0.5280(3)	0.3341(2)	0.053(1)	0.056(2)	0.033(1)	–0.002(1)	–0.024(1)	–0.009(1)
Cl(2)	2*i*	0.4699(4)	0.7931(3)	0.4841(2)	0.064(2)	0.023(1)	0.031(1)	–0.011(1)	–0.005(1)	–0.0016(9)
Cl(3)	2*i*	0.1944(3)	0.5009(3)	0.6659(2)	0.033(1)	0.047(1)	0.035(1)	–0.013(1)	0.0052(9)	–0.010(1)
N(1)	2*i*	0.142(1)	0.994(1)	0.2184(7)	0.040(4)	0.049(5)	0.021(3)	–0.006(4)	–0.007(3)	–0.001(3)
C(1)	2*i*	0.193(1)	0.941(1)	0.0952(8)	0.027(4)	0.036(5)	0.029(4)	–0.004(4)	–0.006(3)	–0.003(4)
C(2)	2*i*	0.161(1)	0.780(1)	0.0832(9)	0.037(5)	0.032(5)	0.040(5)	–0.001(4)	–0.016(4)	0.008(4)
C(3)	2*i*	0.212(1)	0.733(1)	–0.042(1)	0.045(6)	0.051(6)	0.055(6)	0.010(5)	–0.027(5)	–0.024(5)
C(4)	2*i*	0.291(2)	0.847(2)	–0.158(1)	0.051(6)	0.072(8)	0.044(6)	0.023(6)	–0.029(5)	–0.029(6)
C(5)	2*i*	0.323(1)	1.004(1)	–0.1491(9)	0.035(5)	0.065(7)	0.023(4)	0.006(5)	–0.009(4)	0.004(4)
C(6)	2*i*	0.271(1)	1.059(1)	–0.0208(8)	0.027(4)	0.043(5)	0.030(4)	0.005(4)	–0.015(4)	–0.006(4)
C(7)	2*i*	0.302(1)	1.218(1)	–0.003(1)	0.027(5)	0.046(6)	0.053(6)	–0.004(4)	–0.012(4)	0.010(5)
C(8)	2*i*	0.251(2)	1.259(1)	0.123(1)	0.054(6)	0.045(6)	0.060(7)	0.001(5)	–0.034(5)	–0.018(5)
C(9)	2*i*	0.169(2)	1.145(1)	0.233(1)	0.055(6)	0.054(7)	0.050(6)	0.002(5)	–0.024(5)	–0.022(5)
O(1)	2*i*	0.045(1)	0.2005(8)	0.5497(6)	0.053(4)	0.037(4)	0.034(3)	–0.012(3)	–0.006(3)	–0.001(3)

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