Crystal structure of (acetonitrile-κ*N*)triiodido[2-(2-pyridyl)phenyl-κ^2*N*,*C*]platinum(IV), $C_{13}H_{11}I_3N_2Pt$

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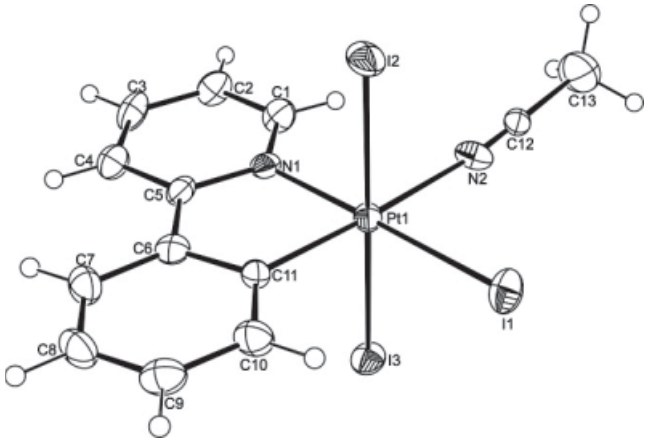

Abstract

$C_{13}H_{11}I_3N_2Pt$, monoclinic, $P2_1/n$ (no. 14), a = 7.2253(9) Å, b = 16.455(2) Å, c = 14.494(2) Å, β = 102.301(3)°, V = 1683.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.0307, $wR_{ref}(F^2)$ = 0.0714, T = 200 K.

Table 1. Data collection and handling.

Crystal:	brown sticks, size 0.09×0.12×0.14 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	138.23 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω
$2\theta_{max}$:	52.12°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10273, 3277
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2520
$N(param)_{refined}$:	173
Programs:	SHELX [2], ORTEP-3[3], PLATON [4]

Source of material

To a solution of K_2PtI_6 (0.209 g, 0.202 mmol) in H_2O (30 ml)/ EtOH (10 ml) was added 2-phenylpyridine (*ppy*; 0.095 g, 0.615 mmol) and stirred for 72 h at room temperature. The formed precipitate was separated by filtration, washed with H_2O, and dried at 50 °C, to give a dark brown powder (0.127 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}(CH_3). The highest peak (1.70 e·Å^{-3}) and the deepest hole (–0.88 e·Å^{-3}) in the difference Fourier map are located 0.90 Å and 1.27 Å from the atoms I2 and C8, respectively.

Discussion

Single crystals of the title complex $[PtI_3(C_{11}H_8N)(CH_3CN)]$ were obtained during crystallization from a CH_3CN solution of the reaction product prepared by the reaction of K_2PtI_6 and *ppy*. The structure of the neutral complex is much different from that of the product of the analogous reaction of ppy with K_2PtCl_6, in which an anionic Pt(IV) complex, $[PtCl_4(C_{11}H_8N)]^-$ with a 2-phenylpyridinium counter cation was synthesized [1]. In the title structure, the central Pt(IV) ion is six-coordinated by one N atom and one C atom of one chelating anionic 2-(2-pyridyl)-phenyl ligand, one N atom of CH_3CN molecule and three iodido ligands in a distorted octahedral manner. The main contribution to the distortion of the octahedron is made by the tight chelate angle ∠N1–Pt1–C11 = 81.1(3)°, which results in non-linear *trans* axes (∠N1–Pt1–I1 = 177.87(17)° and ∠C11–Pt1–N2 = 175.7(3)°). The apical I2–Pt1–I3 bond is almost linear with the bond angle of 178.45(2)°. Whereas the Pt–I bond lengths are almost equivalent (2.6401(6) - 2.6603(7) Å), the Pt–N bond lengths are slightly different, because of the different *trans* effects of the C and I atoms. The Pt1–N1 bond trans to the I atom (2.078(6) Å) is somewhat shorter than the Pt1–N2 bond *trans* to the C atom (2.207(7) Å). In the crystal, the pyridyl and phenyl rings of the anionic ligand are nearly parallel with the dihedral angle of 0.2(4)° between the ring planes. The complexes are stacked perpendicular to the I2–Pt1–I3 axis. π-π interactions between adjacent six-membered rings are present. For Cg1 (the centroid of ring N1-C5) and Cg1^{i} (symmetry code **i**: 1-x, 1-y, -z), the centroid-centroid distance is 3.926(5) Å, the planes are parallel and shifted by 2.066 Å.

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.2965	0.2973	0.0277	0.041
H(2)	4*e*	0.1090	0.4121	0.0017	0.044
H(3)	4*e*	0.2342	0.5363	0.0629	0.047
H(4)	4*e*	0.5457	0.5445	0.1450	0.039
H(7)	4*e*	0.8539	0.5394	0.2166	0.044
H(8)	4*e*	1.1643	0.5235	0.2945	0.048
H(9)	4*e*	1.3053	0.3971	0.3045	0.050
H(10)	4*e*	1.1393	0.2838	0.2360	0.044
H(13A)	4*e*	0.2747	0.0155	–0.0167	0.092
H(13B)	4*e*	0.2683	0.0720	–0.1071	0.092
H(13C)	4*e*	0.1298	0.0901	–0.0369	0.092

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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)	4*e*	0.71181(4)	0.25407(2)	0.12464(2)	0.0253(2)	0.0223(2)	0.0232(2)	–0.0006(1)	0.0078(1)	–0.0003(1)
I(1)	4*e*	0.95657(8)	0.13304(3)	0.15996(4)	0.0411(3)	0.0291(3)	0.0519(4)	0.0089(2)	0.0140(3)	0.0012(3)
I(2)	4*e*	0.80245(9)	0.27656(3)	–0.04159(4)	0.0519(4)	0.0409(3)	0.0271(3)	–0.0090(3)	0.0177(3)	–0.0044(2)
I(3)	4*e*	0.61178(8)	0.23326(3)	0.28939(4)	0.0411(3)	0.0361(3)	0.0293(3)	0.0017(2)	0.0166(3)	0.0050(2)
N(1)	4*e*	0.5275(9)	0.3524(4)	0.0977(4)	0.031(4)	0.028(4)	0.020(4)	–0.004(3)	0.004(3)	0.004(3)
N(2)	4*e*	0.4998(9)	0.1642(4)	0.0560(5)	0.027(4)	0.045(5)	0.022(4)	–0.002(3)	0.006(3)	–0.004(3)
C(1)	4*e*	0.348(1)	0.3483(5)	0.0511(6)	0.028(5)	0.034(5)	0.040(5)	–0.005(4)	0.003(4)	0.004(4)
C(2)	4*e*	0.236(1)	0.4156(5)	0.0362(6)	0.025(4)	0.039(5)	0.044(6)	0.003(4)	0.003(4)	0.008(4)
C(3)	4*e*	0.311(1)	0.4889(5)	0.0719(6)	0.042(5)	0.027(5)	0.053(6)	0.010(4)	0.021(5)	0.010(4)
C(4)	4*e*	0.495(1)	0.4939(5)	0.1202(6)	0.032(5)	0.030(5)	0.038(5)	0.003(3)	0.011(4)	0.004(4)
C(5)	4*e*	0.607(1)	0.4245(4)	0.1327(5)	0.033(5)	0.023(4)	0.026(5)	0.002(3)	0.008(4)	0.007(3)
C(6)	4*e*	0.805(1)	0.4194(5)	0.1802(5)	0.027(4)	0.033(5)	0.023(4)	–0.002(3)	0.012(3)	0.003(3)
C(7)	4*e*	0.911(1)	0.4871(5)	0.2210(6)	0.044(6)	0.032(5)	0.036(5)	–0.010(4)	0.014(4)	–0.005(4)
C(8)	4*e*	1.094(1)	0.4778(5)	0.2665(6)	0.041(5)	0.043(5)	0.036(5)	–0.018(4)	0.005(4)	–0.008(4)
C(9)	4*e*	1.177(1)	0.4028(5)	0.2721(6)	0.033(5)	0.053(6)	0.036(5)	–0.002(4)	0.001(4)	0.007(4)
C(10)	4*e*	1.079(1)	0.3353(5)	0.2319(6)	0.031(5)	0.041(5)	0.034(5)	0.000(4)	–0.001(4)	–0.002(4)
C(11)	4*e*	0.892(1)	0.3433(4)	0.1854(5)	0.034(5)	0.022(4)	0.020(4)	–0.003(3)	0.006(3)	0.000(3)
C(12)	4*e*	0.406(1)	0.1269(5)	0.0176(6)	0.048(6)	0.022(4)	0.020(5)	0.003(4)	0.017(4)	0.000(3)
C(13)	4*e*	0.256(1)	0.0711(6)	–0.0411(7)	0.072(7)	0.055(7)	0.053(7)	–0.017(5)	0.000(6)	–0.006(5)

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