Crystal structure of 3-phenylpyridinium pentachloro-(3-phenylpyridine-κN)platinate(IV) hydrate, $[C_{11}H_{10}N][PtCl_5(C_{11}H_9N)] \cdot H_2O$

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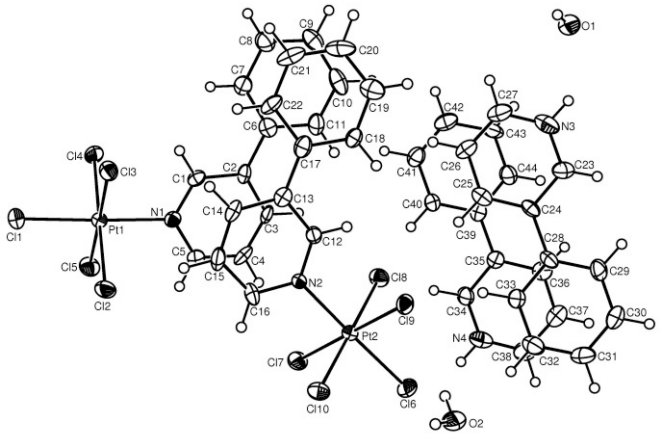

Abstract

$C_{22}H_{21}Cl_5N_2OPt$, monoclinic, $P12_1/c1$ (no. 14), a = 7.2901(5) Å, b = 21.941(2) Å, c = 29.264(2) Å, β = 92.829(2)°, V = 4675.2 Å^3, Z = 8, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.098, T = 200 K.

Source of material

To a suspension of K_2PtCl_6 (0.2431 g, 0.500 mmol) in H_2O (30 ml)/EtOH (10 ml) was added 3-phenylpyridine (0.1782 g, 1.148 mmol), and the mixture was refluxed for 3 h. The formed precipitate was separated by filtration, washed with H_2O and acetone, and dried at 50 °C, to give a yellow powder (0.1663 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a CH_3NO_2 solution.

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 molecule were located from the difference Fourier map and then allowed to ride on their parent O atom 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 (1.89 e Å^{-3}) and the deepest hole (–0.81 e Å^{-3}) in the difference Fourier map are located 1.18 Å and 0.91 Å from the Pt2 atom, respectively.

Discussion

The crystal structure of the title compound consists of a protonated 3-phenylpyridinium cation, an anionic Pt(IV) complex $[PtCl_5(C_{11}H_9N)]^-$ and a solvent water molecule. The asymmetric unit contains two crystallographically independent molecules with identical configuration within experimental errors. Each Pt(IV) ion has a slightly distorted octahedral environment defined by one N atom from the 3-phenylpyridine ligand and five Cl^- anions. The Pt—Cl bond lengths are slightly different, because of the different *trans* effects of the N and Cl atoms. The Pt1—Cl1 and Pt2—Cl6 bonds *trans* to the N atoms (2.305(2) Å and 2.307(2) Å) are somewhat shorter than the Pt—Cl bonds *trans* to the Cl atoms (2.313(2) - 2.322(2) Å). The pyridyl and phenyl rings of the two cations are nearly parallel. The dihedral angles between the ring planes are 1.8(3)° and 8.3(3)°, respectively. In the case of the coordinated 3-phenylpyridine ligands, however, the phenyl rings are inclined considerably to their carrier pyridine rings with dihedral angles of 29.1(2)° and 27.8(2)°, respectively. The anions are stacked in columns along [100] and display numerous intermolecular π–π interactions between adjacent six-membered rings. For Cg1 (the centroid of ring C6-C11) and Cg2^i (ring C17-C22, symmetry code i: x–1,y,z), the centroid–centroid distance is 3.619(5) Å and the dihedral angle between the ring planes is 2.1(4)°. Moreover, the complex ions, cations and water molecules are linked by intermolecular O–H···Cl and N–H···O hydrogen bonds with d(O···Cl) = 3.458(6) Å, 3.584(7) Å and d(N···O) = 2.899(9) - 3.04(1) Å. There are also intra- and intermolecular C–H···Cl hydrogen bonds with d(C···Cl) = 3.176(7) - 3.59(1)Å.

Table 1. Data collection and handling.

Crystal:	yellow stick, size 0.14 × 0.14 × 0.27 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	65.93 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	52.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	29003, 9121
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6179
$N(param)_{refined}$:	559
Programs:	SHELXS-97, SHELXL-97 [1], ORTEP-3 [2], PLATON [3]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.0964	0.1849	0.3283	0.032
H(3)	4*e*	0.0089	0.1677	0.1917	0.035
H(4)	4*e*	0.0018	0.0633	0.2012	0.043
H(5)	4*e*	0.0332	0.0212	0.2742	0.037
H(7)	4*e*	–0.0198	0.2756	0.3192	0.042
H(8)	4*e*	0.0071	0.3815	0.3143	0.052
H(9)	4*e*	0.1220	0.4249	0.2485	0.054
H(10)	4*e*	0.1995	0.3629	0.1879	0.057
H(11)	4*e*	0.1731	0.2592	0.1929	0.045
H(12)	4*e*	0.5154	0.1808	0.1768	0.031
H(14)	4*e*	0.5859	0.1635	0.3137	0.041
H(15)	4*e*	0.5544	0.0597	0.3050	0.047
H(16)	4*e*	0.5155	0.0170	0.2322	0.042
H(18)	4*e*	0.6759	0.2646	0.1848	0.039
H(19)	4*e*	0.7077	0.3700	0.1898	0.055
H(20)	4*e*	0.6228	0.4214	0.2548	0.065
H(21)	4*e*	0.5093	0.3683	0.3150	0.060
H(22)	4*e*	0.4763	0.2627	0.3119	0.044
H(3N)	4*e*	0.4991	0.4337	0.0250	0.059
H(23)	4*e*	0.5687	0.3597	–0.0240	0.052
H(25)	4*e*	0.4734	0.2395	0.0759	0.042
H(26)	4*e*	0.4026	0.3197	0.1237	0.053
H(27)	4*e*	0.4090	0.4195	0.0964	0.057
H(29)	4*e*	0.6273	0.2932	–0.0667	0.045
H(30)	4*e*	0.6927	0.2144	–0.1163	0.050
H(31)	4*e*	0.6703	0.1136	–0.0932	0.055
H(32)	4*e*	0.5923	0.0920	–0.0182	0.051
H(33)	4*e*	0.5350	0.1694	0.0323	0.045
H(4N)	4*e*	0.0647	0.0670	–0.0168	0.056
H(34)	4*e*	–0.0017	0.1407	0.0326	0.046
H(36)	4*e*	0.1550	0.2618	–0.0615	0.044
H(37)	4*e*	0.2148	0.1813	–0.1100	0.059
H(38)	4*e*	0.1748	0.0814	–0.0863	0.059
H(40)	4*e*	–0.0190	0.2073	0.0786	0.040
H(41)	4*e*	–0.0842	0.2868	0.1294	0.046
H(42)	4*e*	–0.0798	0.3866	0.1051	0.053
H(43)	4*e*	–0.0089	0.4089	0.0296	0.053
H(44)	4*e*	0.0570	0.3311	–0.0202	0.048
H(1A)	4*e*	0.4840	0.5271	0.0662	0.071
H(1B)	4*e*	0.2947	0.5310	0.0516	0.071
H(2A)	4*e*	0.8929	0.0420	0.0592	0.074
H(2B)	4*e*	1.0574	0.0123	0.0722	0.074

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*	0.08248(4)	0.06368(1)	0.37313(1)	0.0320(2)	0.0228(2)	0.0250(2)	0.0003(2)	0.0037(1)	0.0002(1)
Cl(1)	4*e*	0.0990(3)	0.0269(1)	0.44697(7)	0.062(2)	0.037(1)	0.031(1)	–0.001(1)	0.005(1)	0.0083(9)
Cl(2)	4*e*	0.2937(3)	–0.00858(9)	0.35181(8)	0.046(1)	0.029(1)	0.048(1)	0.011(1)	0.011(1)	0.002(1)
Cl(3)	4*e*	0.3205(3)	0.13052(9)	0.39277(7)	0.039(1)	0.034(1)	0.031(1)	–0.007(1)	–0.007(1)	0.0025(9)
Cl(4)	4*e*	–0.1320(3)	0.13524(9)	0.39368(7)	0.041(1)	0.036(1)	0.034(1)	0.010(1)	0.009(1)	–0.0031(9)
Cl(5)	4*e*	–0.1532(3)	–0.0039(1)	0.35456(7)	0.039(1)	0.034(1)	0.047(1)	–0.011(1)	0.002(1)	0.000(1)
N(1)	4*e*	0.0675(8)	0.0996(3)	0.3075(2)	0.025(4)	0.027(4)	0.025(4)	–0.004(3)	–0.001(3)	–0.004(3)
C(1)	4*e*	0.077(1)	0.1601(4)	0.3019(3)	0.023(4)	0.032(5)	0.024(5)	0.003(4)	–0.003(4)	–0.007(3)
C(2)	4*e*	0.058(1)	0.1885(4)	0.2595(2)	0.027(5)	0.039(5)	0.018(4)	0.004(4)	0.000(3)	0.005(4)
C(3)	4*e*	0.026(1)	0.1506(4)	0.2214(3)	0.036(5)	0.040(5)	0.012(4)	0.004(4)	0.003(4)	0.001(3)
C(4)	4*e*	0.020(1)	0.0891(4)	0.2271(3)	0.036(5)	0.052(6)	0.021(5)	–0.001(4)	0.007(4)	–0.016(4)
C(5)	4*e*	0.040(1)	0.0642(4)	0.2705(3)	0.035(5)	0.027(5)	0.030(5)	–0.002(4)	0.001(4)	–0.004(4)
C(6)	4*e*	0.072(1)	0.2558(4)	0.2563(3)	0.023(5)	0.036(5)	0.036(5)	0.005(4)	–0.002(4)	0.005(4)
C(7)	4*e*	0.025(1)	0.2934(4)	0.2922(3)	0.033(5)	0.039(6)	0.033(5)	0.011(4)	0.001(4)	0.003(4)
C(8)	4*e*	0.041(1)	0.3564(4)	0.2897(3)	0.052(6)	0.036(6)	0.042(6)	0.001(5)	–0.004(5)	0.005(4)
C(9)	4*e*	0.108(1)	0.3819(4)	0.2506(3)	0.042(6)	0.035(6)	0.057(7)	0.003(5)	–0.006(5)	0.016(5)
C(10)	4*e*	0.155(1)	0.3451(5)	0.2148(3)	0.038(6)	0.051(7)	0.053(7)	–0.002(5)	–0.008(5)	0.028(5)
C(11)	4*e*	0.138(1)	0.2837(4)	0.2177(3)	0.038(6)	0.045(6)	0.029(5)	0.006(4)	0.003(4)	0.009(4)
Pt(2)	4*e*	0.49852(4)	0.05952(1)	0.13338(1)	0.0327(2)	0.0216(2)	0.0258(2)	–0.0010(1)	0.0028(1)	–0.0005(1)
Cl(6)	4*e*	0.4826(3)	0.0212(1)	0.05987(7)	0.064(2)	0.038(1)	0.031(1)	–0.009(1)	0.006(1)	–0.0107(9)
Cl(7)	4*e*	0.2628(3)	–0.00492(9)	0.15274(7)	0.043(1)	0.030(1)	0.046(1)	–0.011(1)	0.010(1)	–0.0018(9)
Cl(8)	4*e*	0.2843(3)	0.13348(9)	0.11173(7)	0.041(1)	0.032(1)	0.035(1)	0.004(1)	–0.008(1)	0.0021(9)
Cl(9)	4*e*	0.7373(3)	0.12316(9)	0.11420(7)	0.040(1)	0.037(1)	0.031(1)	–0.012(1)	0.009(1)	–0.0009(9)
Cl(10)	4*e*	0.7107(3)	–0.0146(1)	0.15532(8)	0.047(1)	0.031(1)	0.048(2)	0.013(1)	0.005(1)	0.001(1)
N(2)	4*e*	0.5163(8)	0.0962(3)	0.1987(2)	0.026(4)	0.020(4)	0.023(4)	0.003(3)	0.004(3)	0.002(3)
C(12)	4*e*	0.528(1)	0.1562(3)	0.2035(3)	0.028(5)	0.026(5)	0.025(5)	0.000(4)	0.003(4)	0.003(3)
C(13)	4*e*	0.557(1)	0.1852(4)	0.2458(3)	0.025(5)	0.037(5)	0.028(5)	0.001(4)	0.000(4)	–0.001(4)
C(14)	4*e*	0.567(1)	0.1466(4)	0.2839(3)	0.034(5)	0.051(6)	0.017(5)	–0.002(4)	0.002(4)	–0.004(4)
C(15)	4*e*	0.550(1)	0.0853(4)	0.2788(3)	0.046(6)	0.050(6)	0.023(5)	0.000(5)	0.011(4)	0.009(4)
C(16)	4*e*	0.526(1)	0.0600(4)	0.2356(3)	0.040(5)	0.036(5)	0.029(5)	–0.002(4)	–0.003(4)	0.016(4)
C(17)	4*e*	0.577(1)	0.2513(4)	0.2481(3)	0.029(5)	0.047(6)	0.026(5)	0.003(4)	–0.002(4)	0.002(4)
C(18)	4*e*	0.643(1)	0.2851(4)	0.2118(3)	0.041(6)	0.037(5)	0.018(5)	–0.001(4)	–0.005(4)	–0.002(3)
C(19)	4*e*	0.661(1)	0.3479(4)	0.2146(3)	0.046(6)	0.036(6)	0.055(7)	–0.001(5)	–0.008(5)	–0.001(5)
C(20)	4*e*	0.611(1)	0.3783(4)	0.2531(4)	0.058(7)	0.031(6)	0.072(8)	0.016(5)	–0.008(6)	–0.018(5)
C(21)	4*e*	0.544(1)	0.3469(5)	0.2885(3)	0.048(6)	0.054(7)	0.048(7)	0.011(5)	–0.004(5)	–0.030(5)
C(22)	4*e*	0.525(1)	0.2839(4)	0.2869(3)	0.039(6)	0.049(6)	0.023(5)	0.007(4)	–0.005(4)	–0.014(4)
N(3)	4*e*	0.492(1)	0.3959(3)	0.0348(3)	0.060(6)	0.026(5)	0.060(6)	–0.002(4)	–0.014(5)	0.004(4)
C(23)	4*e*	0.534(1)	0.3504(4)	0.0061(3)	0.055(7)	0.031(6)	0.043(6)	–0.003(5)	0.001(5)	–0.009(4)
C(24)	4*e*	0.528(1)	0.2902(3)	0.0203(3)	0.026(5)	0.021(4)	0.036(5)	–0.006(4)	–0.008(4)	0.005(3)
C(25)	4*e*	0.478(1)	0.2801(4)	0.0647(3)	0.035(5)	0.026(5)	0.045(6)	–0.001(4)	–0.003(4)	0.005(4)
C(26)	4*e*	0.436(1)	0.3277(4)	0.0933(3)	0.052(6)	0.046(6)	0.034(6)	0.004(5)	0.008(5)	–0.013(4)
C(27)	4*e*	0.441(1)	0.3864(4)	0.0775(3)	0.055(7)	0.042(6)	0.044(6)	0.004(5)	–0.003(5)	–0.017(5)
C(28)	4*e*	0.569(1)	0.2399(4)	–0.0122(3)	0.037(5)	0.021(5)	0.034(5)	–0.001(4)	–0.005(4)	0.007(4)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(29)	4*e*	0.619(1)	0.2522(4)	–0.0566(3)	0.033(5)	0.040(6)	0.042(6)	–0.005(4)	0.011(4)	0.009(4)
C(30)	4*e*	0.658(1)	0.2053(4)	–0.0862(3)	0.043(6)	0.058(7)	0.025(5)	–0.001(5)	0.003(4)	–0.002(4)
C(31)	4*e*	0.647(1)	0.1458(4)	–0.0726(3)	0.054(7)	0.034(6)	0.048(6)	0.004(5)	–0.004(5)	–0.014(4)
C(32)	4*e*	0.600(1)	0.1332(4)	–0.0280(3)	0.052(6)	0.030(5)	0.045(6)	0.001(5)	–0.008(5)	0.002(4)
C(33)	4*e*	0.564(1)	0.1790(4)	0.0018(3)	0.049(6)	0.035(5)	0.028(5)	0.002(4)	0.002(4)	0.002(4)
N(4)	4*e*	0.082(1)	0.1048(3)	–0.0255(3)	0.049(5)	0.028(5)	0.064(6)	–0.002(4)	0.001(4)	–0.006(4)
C(34)	4*e*	0.043(1)	0.1503(4)	0.0035(3)	0.054(6)	0.034(6)	0.026(5)	0.000(5)	–0.006(4)	0.003(4)
C(35)	4*e*	0.067(1)	0.2107(4)	–0.0084(3)	0.024(5)	0.034(5)	0.031(5)	0.008(4)	–0.001(4)	0.002(4)
C(36)	4*e*	0.135(1)	0.2212(4)	–0.0517(3)	0.033(5)	0.039(6)	0.037(6)	0.003(4)	–0.007(4)	–0.002(4)
C(37)	4*e*	0.172(1)	0.1733(5)	–0.0805(3)	0.049(6)	0.059(7)	0.041(6)	0.003(5)	0.008(5)	–0.006(5)
C(38)	4*e*	0.147(1)	0.1146(5)	–0.0669(3)	0.045(6)	0.051(7)	0.051(7)	0.002(5)	–0.004(5)	–0.012(5)
C(39)	4*e*	0.027(1)	0.2602(4)	0.0238(3)	0.023(5)	0.037(5)	0.025(5)	0.001(4)	–0.004(4)	0.001(4)
C(40)	4*e*	–0.016(1)	0.2483(4)	0.0683(3)	0.042(6)	0.031(5)	0.027(5)	0.003(4)	0.002(4)	0.005(4)
C(41)	4*e*	–0.056(1)	0.2960(4)	0.0989(3)	0.036(5)	0.048(6)	0.032(5)	0.009(4)	0.004(4)	0.000(4)
C(42)	4*e*	–0.053(1)	0.3546(4)	0.0846(3)	0.042(6)	0.048(6)	0.041(6)	0.003(5)	–0.007(5)	–0.018(5)
C(43)	4*e*	–0.011(1)	0.3678(4)	0.0398(3)	0.060(7)	0.020(5)	0.051(7)	0.004(4)	–0.001(5)	–0.002(4)
C(44)	4*e*	0.028(1)	0.3214(4)	0.0103(3)	0.049(6)	0.037(6)	0.034(5)	0.002(5)	–0.002(4)	0.007(4)
O(1)	4*e*	0.4035(8)	0.5243(3)	0.0448(2)	0.051(4)	0.043(4)	0.047(4)	0.000(3)	0.007(3)	–0.004(3)
O(2)	4*e*	0.9821(9)	0.0214(3)	0.0506(2)	0.071(5)	0.037(4)	0.040(4)	–0.001(3)	0.000(3)	–0.010(3)

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References

1. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
2. Farrugia, L. J.: ORTEP-3 for Windows - a version of ORTEP-III with a Graphical User Interface (GUI). J. Appl. Crystallogr. **30** (1997) 565.
3. Spek, A. L.: Single-crystal structure validation with the program PLATON. J. Appl. Crystallogr. **36** (2003) 7-13.