Crystal structure of η^2-1-allyl-3-methylimidazolium trichloroplatinate(II), $[C_7H_{11}N_2][PtCl_3]$

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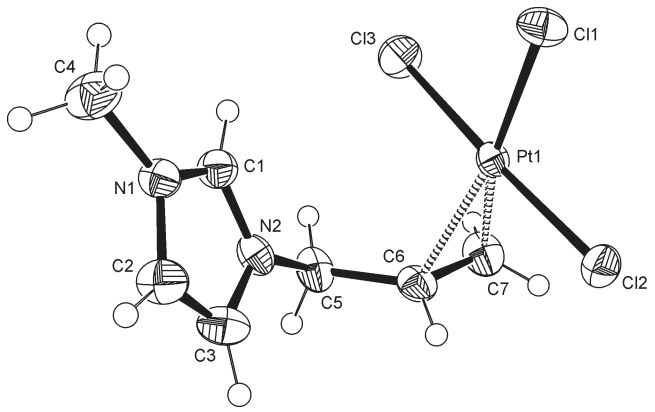

Abstract

$C_7H_{11}Cl_3N_2Pt$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 7.6873(2)$ Å, $b = 11.9159(3)$ Å, $c = 12.1398(3)$ Å, $V = 1112.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.018$, $wR_{ref}(F^2) = 0.045$, $T = 233$ K.

Source of material

Potassium η^2-ethylene trichloroplatinate(II) hydrate (Zeise's salt) [CARN: 123334-22-5] was stirred with an equimolar amount of 1-allyl-3-methylimidazolium BF_4 [CARN: 851606-63-8] [1] for three days at room temperature. The precipitate was collected by filtration, washed with water, and redissolved in DMF. Yellow crystals of the zwitterionic title compound, suitable for X-ray diffraction, were obtained from this solution by diffusion with diethyl ether at −20 °C.

Experimental details

Considering the slight distortion due to the metal coordination, hydrogen atoms at C6 and C7 were not added geometrically, but were found and refined with bond restraints ($d = 0.93$ Å).

Discussion

The platinum(II) atom is four-coordinated with a square-planar configuration, the center of the C=C bond being one of the vertices. The double bond slightly deviates (7°) from the direction perpendicular to the coordination plane. Bond lengths of the trichloroplatinate(II) moiety compare well with those in known Pt-alkene complexes [2-6]. Thus, a distance of 2.025 Å between the metal atom and the center of the C6—C7 bond is found. The C5—C6 bond is rotated out of the imidazolium ring plane by 75.0°, and the N2–C5–C6–C7 torsion angle is −148.3°. Several weak C–H···Cl contacts are observed (H···acceptor distance, donor···acceptor distance, donor–H···acceptor angle): C1–H···Cl2^{i} 2.81 Å, 3.620 Å, 146°; C4–H···Cl2^{i} 2.78 Å, 3.697 Å, 158°; C2–H···Cl1^{ii} 2.86 Å, 3.538 Å, 130°; C4–H···Cl2^{ii} 2.80 Å, 3.571 Å, 137°; C3–H···Cl1^{iii} 2.71 Å, 3.577 Å, 155°; C5–H···Cl2^{iv} 2.90 Å, 3.600 Å, 129° (symmetry code (i): 7/2−x,1−y,1/2+z; (ii): −1/2+x, 1/2−y,2−z; (iii): 7/2−x, 1−y,-1/2+z; (iv): −1+x,y,z). Of course, the 1-allyl-3-methylimidazolium cation can be converted to a heterocyclic carbene. Thus, the respective silver(I) carbene complex [7] and an iridium(I) compound in which the metal is coordinated to both the carbene and the allyl group [8] have been described. Crystal structures of other 1-allyl-3-methylimidazolium salts such as the tetraphenylborate and dibromodichloropalladate(II) [1], the iodide [9], as well as the related 1-allyl-2,3-dimethylimidazolium bromide [10] have been reported previously.

Table 1. Data collection and handling.

Crystal:	yellow plate, size 0.08 × 0.2 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	132.95 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	51.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7074, 2180
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2169
$N(param)_{refined}$:	131
Programs:	SHELXS-97 [11], SHELXL-97 [12]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	1.4865	0.4649	1.0905	0.037
H(2)	4*a*	1.4023	0.2182	0.8771	0.046
H(3)	4*a*	1.4383	0.3927	0.7739	0.047
H(4A)	4*a*	1.4170	0.2798	1.1785	0.070
H(4B)	4*a*	1.5085	0.1836	1.1095	0.070
H(4C)	4*a*	1.3045	0.2005	1.1017	0.070
H(5A)	4*a*	1.4825	0.6329	0.9672	0.040
H(5B)	4*a*	1.4128	0.6132	0.8459	0.040
H(6)	4*a*	1.712(6)	0.576(4)	0.791(3)	0.03(1)
H(7B)	4*a*	1.727(8)	0.774(4)	0.920(5)	0.06(2)
H(7A)	4*a*	1.851(6)	0.722(6)	0.823(4)	0.06(2)

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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*a*	1.88810(2)	0.58707(1)	0.96917(1)	0.02131(9)	0.02055(9)	0.0262(1)	–0.00066(7)	–0.00125(7)	–0.00039(7)
Cl(1)	4*a*	2.0769(2)	0.4899(1)	1.0834(1)	0.0386(6)	0.0355(6)	0.0321(6)	0.0092(5)	–0.0086(5)	–0.0001(5)
Cl(2)	4*a*	2.0560(2)	0.5379(1)	0.8200(1)	0.0293(5)	0.0407(7)	0.0321(6)	0.0011(5)	0.0047(5)	0.0008(5)
Cl(3)	4*a*	1.7329(2)	0.6409(1)	1.1224(1)	0.0378(6)	0.0478(8)	0.0352(7)	0.0044(6)	0.0058(5)	–0.0107(6)
N(1)	4*a*	1.4398(5)	0.3171(3)	1.0172(3)	0.034(2)	0.024(2)	0.029(2)	0.000(2)	–0.003(2)	0.001(2)
N(2)	4*a*	1.4738(5)	0.4695(3)	0.9244(4)	0.023(2)	0.026(2)	0.037(2)	0.002(2)	–0.006(2)	–0.002(2)
C(1)	4*a*	1.4700(6)	0.4251(4)	1.0245(4)	0.030(2)	0.034(3)	0.029(3)	0.001(2)	–0.000(2)	–0.001(2)
C(2)	4*a*	1.4235(8)	0.2898(4)	0.9067(5)	0.052(3)	0.027(2)	0.036(3)	0.000(2)	–0.004(3)	–0.001(2)
C(3)	4*a*	1.4438(7)	0.3849(5)	0.8509(4)	0.047(3)	0.040(3)	0.030(3)	0.006(2)	–0.006(2)	–0.007(2)
C(4)	4*a*	1.4154(8)	0.2386(4)	1.1096(5)	0.069(4)	0.033(3)	0.038(3)	–0.004(3)	0.000(3)	0.006(3)
C(5)	4*a*	1.5000(6)	0.5891(4)	0.8998(5)	0.025(2)	0.026(2)	0.048(3)	0.005(2)	–0.007(2)	0.010(3)
C(6)	4*a*	1.6772(6)	0.6127(4)	0.8552(4)	0.031(2)	0.028(3)	0.031(3)	0.002(2)	–0.008(2)	0.007(2)
C(7)	4*a*	1.7627(7)	0.7152(4)	0.8753(5)	0.035(3)	0.027(3)	0.048(3)	0.004(2)	–0.007(2)	0.010(2)

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