Crystal structure of di(tetramethylammonium)tetrachloropalladate(II), $[\text{N}(\text{CH}_3)_4]_2[\text{PdCl}_4]$

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Abstract

$\text{C}_8\text{H}_{24}\text{Cl}_4\text{N}_2\text{Pd}$, tetragonal, $P4_2/mnm$ (no. 136), $a = 8.8287(2) \text{ \AA}$, $c = 11.4264(3) \text{ \AA}$, $V = 890.6 \text{ \AA}^3$, $Z = 2$, $R_{\text{gt}}(F) = 0.024$, $wR_{\text{ref}}(F^2) = 0.072$, $T = 293 \text{ K}$.

Source of material

$[\text{N}(\text{CH}_3)_4]_2[\text{PdCl}_4]$ was prepared by mixing stoichiometric amounts of PdCl_2 and $[\text{tma}]\text{Cl}$ in 0.01 M hydrochloric acid [1]. Dark orange-yellow coloured crystals were grown from solution at room temperature. The crystals were sensitive to moisture and were kept in mother liquor.

Experimental details

All hydrogen atoms were located from fourier maps and refined isotropically.

Discussion

In the course of our work on redox-active tetrahalogenopalladates(II) [2] we have prepared $[\text{N}(\text{CH}_3)_4]_2[\text{PdCl}_4]$, which is built up by a layered structure of isolated complex cations and anions. $[\text{PdCl}_4]^{2-}$ anions exhibit bond lengths of $d(\text{Pd}—\text{Cl}1) = 231.0(1) \text{ pm}$ and $d(\text{Pd}—\text{Cl}2) = 229.6(1) \text{ pm}$. Within $[\text{N}(\text{CH}_3)_4]^+$ cations distances of $d(\text{C}—\text{N}) = 148.3(3) \text{ pm}$, $d(\text{C}—\text{H}1) = 88(3) \text{ pm}$, $d(\text{C}—\text{H}2) = 83(3) \text{ pm}$ and $d(\text{C}—\text{H}3) = 95(3) \text{ pm}$ are found. The closest intermolecular contact exists between chlorine and hydrogen: $d(\text{H}1—\text{Cl}2) = 294.3(26) \text{ pm}$. The structure of $[\text{N}(\text{CH}_3)_4]_2[\text{PdCl}_4]$ resembles the motif of $[\text{NH}_4]_2[\text{CuCl}_2] \cdot 2\text{H}_2\text{O}$ [3].

Table 1. Data collection and handling.

Crystal:	orange-yellow, irregular, size $0.1 \times 0.2 \times 0.3 \text{ mm}$
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	16.21 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, ω
$2\theta_{\text{max}}$:	61.64°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	15404, 545
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 349
$N(\text{param})_{\text{refined}}$:	36
Programs:	SHELXS-97 [4], SHELXL-97 [5], DIAMOND [6]

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	x	y	z	U_{iso}
H(1)	16k	0.199(4)	0.592(3)	0.222(2)	0.09(1)
H(2)	16k	0.087(4)	0.629(3)	0.140(2)	0.08(1)
H(3)	16k	0.173(5)	0.476(5)	0.149(3)	0.12(2)

Table 3. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd	2a	0	0	0	0.0383(3)	U_{11}	0.0715(4)	0.0017(2)	0	0
Cl(1)	4f	0.18506(9)	x	0	0.0575(5)	U_{11}	0.0895(8)	−0.0133(5)	0	0
Cl(2)	4e	0	0	0.2010(1)	0.0714(7)	U_{11}	0.0733(8)	−0.0158(5)	0	0
N	4d	0	$\frac{1}{2}$	$\frac{1}{4}$	0.048(2)	U_{11}	0.065(2)	0	0	0
C	16k	0.1219(4)	0.5632(5)	0.1754(4)	0.061(2)	0.087(3)	0.099(2)	−0.002(2)	0.007(2)	0.021(2)

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