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Pentane-1,5-diammonium tetrachloridopalladate(II)

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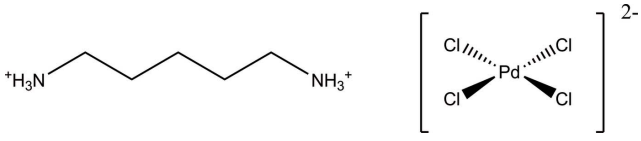
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.089; data-to-parameter ratio = 24.8.

In the title compound, $[NH_3(CH_2)_5NH_3][PdCl_4]$, the square-planar $[PdCl_4]^{2-}$ anions are centrosymmetric while the diammonium cation lies in a general position. In addition to electrostatic interactions, the two species are linked through N—H···Cl hydrogen bonds to form a three-dimensional network.

Related literature

The title compound is isostructural with its tetrachlorido- and tetrabromidocuprate(II) analogues (Garland *et al.*, 1990). For similar tetrachloridopalladate(II) compounds, see: Willett & Willett (1977); Berg & Søtofte (1976); Maris *et al.* (1996).



Experimental

Crystal data

$(C_5H_{16}N_2)[PdCl_4]$
M_r = 352.40
Monoclinic, $P2_1/c$
a = 8.091 (2) Å
b = 7.276 (2) Å
c = 20.843 (5) Å
β = 98.279 (2)°
V = 1214.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.37 mm^{-1}
T = 298 K
0.19 × 0.15 × 0.08 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: integration (Blessing; 1989)
T_{min} = 0.662, T_{max} = 0.833
2816 measured reflections
2783 independent reflections
2771 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
5 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.089
S = 0.99
2783 reflections
112 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 e Å^{-3}

Table 1
Selected bond lengths (Å).

Pd1—Cl2	2.3129 (4)	Pd2—Cl3	2.3160 (4)
Pd1—Cl1	2.3183 (6)	Pd2—Cl4	2.3207 (6)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···Cl2^{i}	0.89	2.88	3.4171 (11)	120
N1—H1C···Cl4^{ii}	0.89	2.51	3.3539 (17)	158
N1—H1A···Cl2^{iii}	0.89	2.53	3.3107 (13)	147
N1—H1B···Cl1	0.89	2.60	3.4680 (12)	165
N7—H7A···Cl1^{iv}	0.89	2.53	3.2512 (15)	138
N7—H7B···Cl4^{v}	0.89	2.51	3.3702 (12)	163
N7—H7C···Cl3^{iv}	0.89	2.44	3.2821 (13)	158
N7—H7A···Cl2^{vi}	0.89	2.70	3.4614 (13)	145
N7—H7B···Cl3^{v}	0.89	2.86	3.3907 (11)	120

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x+1, -y+1, -z$; (iii) $x, y+1, z$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (vi) $x+1, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992); cell refinement: *CAD-4-PC Software*; data reduction: modified version of *NRC-2/NRC2A* (Ahmed *et al.*, 1973); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ATOMS* (Dowty, 2003) and *Materials Studio* (Accelrys, 2002); software used to prepare material for publication: *UdMX* (Maris, 2004) and *publCIF* (Westrip, 2007).

Dr Jean Michel Leger is acknowledged for assistance during a preliminary investigation.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2439).

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Pentane-1,5-diammonium tetrachloridopalladate(II)

Thierry Maris

S1. Comment

Extensive studies have been carried out on the crystal structures, phase transitions and physical properties of two-dimensional perovskite-like compounds of the families $(C_nH_{2n+1}NH_3)_2MX_4$ and $[NH_3\text{-}(CH_2)_n\text{-}NH_3]MX_4$, where *X* represents a halogen atom and *M* is a divalent metal. A few tetrachloropalladate compounds of these families have been structurally characterized: $(C_3H_7NH_3)_2$ [$PdC1_4$] (Willett & Willett, 1977), $[NH_3\text{-}(CH_2)_2\text{-}NH_3]$[$PdC1_4$] (Berg & Søtofte, 1976) and $[NH_3\text{-}(CH_2)_4\text{-}NH_3]$[$PdC1_4$] (Maris *et al.*, 1996). We report here the crystal structure, determined at room temperature, of the title palladium-chloride compound $[NH_3\text{-}(CH_2)_5\text{-}NH_3][PdCl_4]$ (I).

The asymmetric unit of (I) contains one cation in general position and two distinct half $[PdCl_4]^{2-}$ units (Fig. 1). The Pd atoms lie on inversion centers and display a square-planar coordination environment with four Cl^- ligands. The Pd—Cl distances range from 2.3129 (4) to 2.3207 (6) Å (Table 1). The $[PdCl_4]$ moieties pack *via* longer Pd—Cl contacts (3.0244 (9) and 3.1788 (9) Å) to form puckered two-dimensional layers in the (*a*,*b*) plane. The cations are located between these layers and the whole crystallographic organization can be described as a succession of organic and inorganic layers. The diammonium chain adopts a left-handed conformation at one end with a terminal C—C—C—N torsion angles of 67.86 (12)°. The whole chain makes an angle of 83.55 (3)° with the palladium layer.

The link between the two moieties and the crystal packing is achieved by several hydrogen bonds involving the H atoms of the ammonium groups and the Cl atoms. The three shortest hydrogen bonds (Fig. 2) show a pattern similar to the hydrogen bond scheme found in the tetrachloro and tetrabromocuprate(II) analogues. (Garland *et al.* 1990). Additional contacts (Table 2) with longer hydrogen chlorine distances and more acute N—H···Cl angles are also present.

S2. Experimental

Crystals for X-ray structural analysis were grown by slow evaporation at room temperature of a saturated aqueous solution obtained by dissolving $PdCl_2$ (0.2 g, 1.12 mmol) and $NH_2(CH_2)_5NH_2$ (0.12 g, 1.12 mmol) in an excess of concentrated HCl.

S3. Refinement

H atoms of the carbon chain skeleton were positioned geometrically and refined using a riding model with U_{iso}(H) values of $1.2U_{eq}$(C). H atoms of the ammonium groups were located from difference Fourier map and refined as riding atoms with U_{iso}(H) values of $1.5U_{eq}$(N).

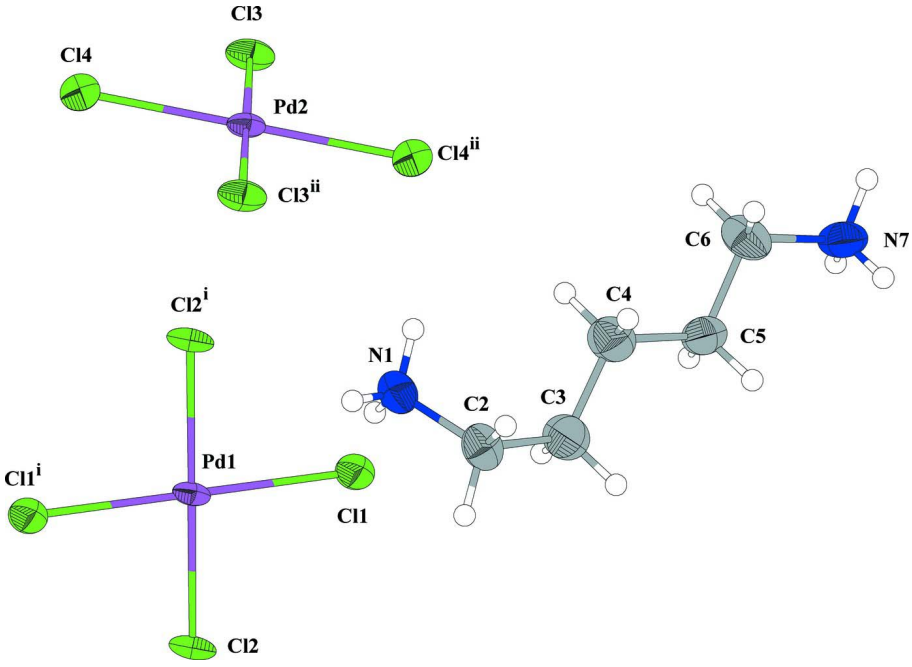

Figure 1

The structure of (I) with thermal ellipsoids shown at the 50% probability level. Symmetry codes: (i) -*x*, -*y*, -*z*; (ii) 1 - *x*, 1 - *y*, -*z*.

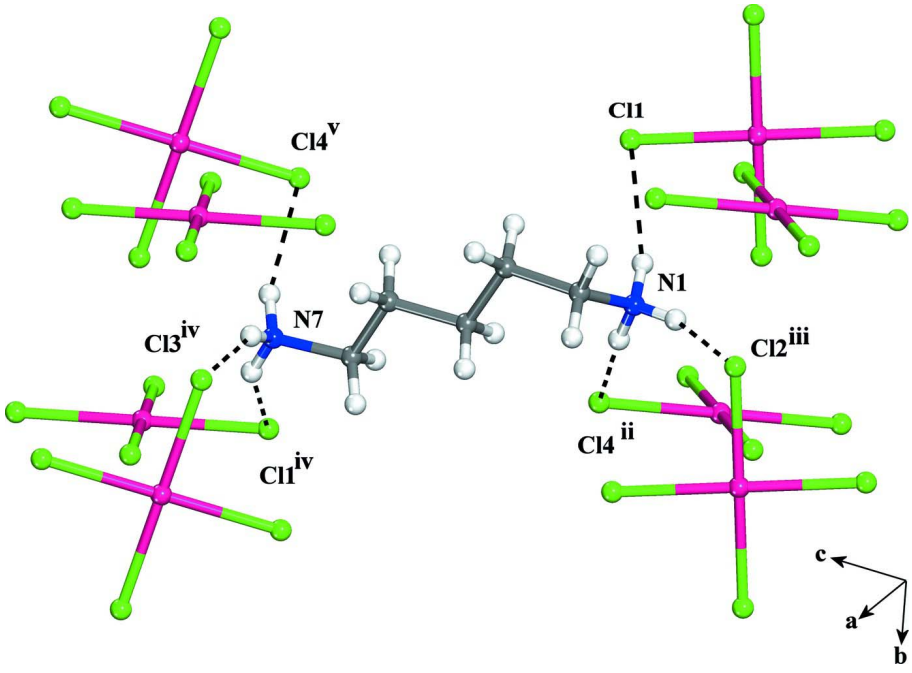

Figure 2

Packing diagram showing the shortest N—H···Cl hydrogen bond interactions as dashed lines. Symmetry codes: (ii) 1 - *x*, 1 - *y*, -*z*; (iii) *x*, *y* + 1, *z*; (iv) 1 - *x*, *y* + 1/2, 1/2 - *z*; (v) *x*, 1/2 - *y*, 1/2 + *z*.

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Crystal data

$(C_5H_{16}N_2)[PdCl_4]$
$M_r = 352.40$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.091$ (2) Å
$b = 7.276$ (2) Å
$c = 20.843$ (5) Å
$\beta = 98.279$ (2)°
$V = 1214.2$ (5) Å^3
$Z = 4$
$F(000) = 696$
$D_x = 1.928$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5$–16.8°
$\mu = 2.37$ mm^{-1}
$T = 298$ K
Plate, dark red
0.19 × 0.15 × 0.08 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: integration (Blessing; 1989)
$T_{min} = 0.662$, $T_{max} = 0.833$
2816 measured reflections
2783 independent reflections
2771 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 27.4°$, $\theta_{min} = 2.0°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 26$
5 standard reflections every 60 min
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.089$
$S = 0.99$
2783 reflections
112 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0883P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.74$ e Å^{-3}
$\Delta\rho_{min} = -0.69$ e Å^{-3}

Special details

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Pd1	0.0000	0.0000	0.0000	0.01948 (10)
Cl1	0.09323 (3)	0.01856 (2)	0.110313 (8)	0.03247 (10)
Cl2	−0.195673 (16)	−0.21942 (2)	0.017283 (7)	0.02727 (10)
Pd2	0.5000	0.5000	0.0000	0.01998 (10)
Cl3	0.711649 (16)	0.285055 (19)	0.022152 (8)	0.02908 (10)
Cl4	0.51179 (2)	0.46485 (3)	−0.109913 (8)	0.03058 (10)
N1	0.07248 (18)	0.49394 (9)	0.10015 (5)	0.0386 (2)

H1A	0.0198	0.5472	0.0645	0.058*
H1B	0.0736	0.3727	0.0945	0.058*
H1C	0.1769	0.5355	0.1080	0.058*
C2	−0.01184 (11)	0.53613 (14)	0.15356 (5)	0.03569 (19)
H2A	−0.1260	0.4924	0.1441	0.043*
H2B	−0.0157	0.6686	0.1583	0.043*
C3	0.06862 (13)	0.45279 (14)	0.21793 (4)	0.0467 (2)
H3A	−0.0062	0.4706	0.2498	0.056*
H3B	0.0809	0.3215	0.2121	0.056*
C4	0.23397 (14)	0.5307 (2)	0.24383 (5)	0.0461 (3)
H4A	0.2259	0.6637	0.2437	0.055*
H4B	0.3137	0.4964	0.2154	0.055*
C5	0.29934 (12)	0.46619 (13)	0.31257 (4)	0.04003 (19)
H5A	0.2217	0.5039	0.3415	0.048*
H5B	0.3047	0.3330	0.3132	0.048*
C6	0.46483 (13)	0.54043 (14)	0.33643 (4)	0.0408 (2)
H6A	0.4639	0.6727	0.3306	0.049*
H6B	0.5461	0.4890	0.3115	0.049*
N7	0.51365 (16)	0.49611 (10)	0.40599 (6)	0.0420 (2)
H7A	0.6148	0.5415	0.4195	0.063*
H7B	0.5151	0.3747	0.4112	0.063*
H7C	0.4403	0.5456	0.4289	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.01015 (15)	0.01755 (17)	0.03049 (14)	−0.00113 (1)	0.00208 (12)	−0.00208 (2)
Cl1	0.02387 (16)	0.04333 (16)	0.02952 (15)	−0.00249 (6)	0.00153 (11)	0.00146 (5)
Cl2	0.01297 (15)	0.02395 (15)	0.04395 (15)	−0.00589 (4)	0.00088 (10)	0.00250 (5)
Pd2	0.01077 (15)	0.01766 (17)	0.03179 (14)	0.00019 (1)	0.00401 (12)	−0.00195 (2)
Cl3	0.01415 (15)	0.02303 (15)	0.04964 (15)	0.00526 (5)	0.00317 (10)	−0.00050 (5)
Cl4	0.02803 (16)	0.03239 (15)	0.03202 (15)	−0.00018 (7)	0.00674 (10)	0.00025 (5)
N1	0.0377 (5)	0.0413 (5)	0.0359 (4)	−0.0059 (2)	0.0023 (4)	−0.0095 (2)
C2	0.0383 (5)	0.0295 (3)	0.0379 (4)	0.0061 (3)	0.0008 (3)	−0.0068 (3)
C3	0.0466 (5)	0.0525 (5)	0.0396 (4)	−0.0105 (4)	0.0016 (3)	0.0077 (4)
C4	0.0370 (5)	0.0562 (5)	0.0433 (5)	−0.0005 (4)	−0.0005 (4)	0.0053 (4)
C5	0.0324 (5)	0.0496 (4)	0.0390 (4)	−0.0051 (3)	0.0082 (3)	0.0042 (3)
C6	0.0456 (5)	0.0268 (3)	0.0465 (4)	−0.0104 (4)	−0.0055 (4)	0.0088 (3)
N7	0.0300 (5)	0.0518 (5)	0.0443 (5)	−0.0080 (2)	0.0057 (5)	0.0125 (3)

Geometric parameters (Å, º)

Pd1—Cl2	2.3129 (4)	C3—C4	1.4814 (13)
Pd1—Cl2^{i}	2.3129 (4)	C3—H3A	0.9700
Pd1—Cl1	2.3183 (6)	C3—H3B	0.9700
Pd1—Cl1^{i}	2.3183 (6)	C4—C5	1.5279 (16)
Pd2—Cl3^{ii}	2.3160 (4)	C4—H4A	0.9700
Pd2—Cl3	2.3160 (4)	C4—H4B	0.9700

Pd2—Cl4	2.3207 (6)	C5—C6	1.4629 (13)
Pd2—Cl4^{ii}	2.3207 (6)	C5—H5A	0.9700
N1—C2	1.4205 (14)	C5—H5B	0.9700
N1—H1A	0.8900	C6—N7	1.4822 (14)
N1—H1B	0.8900	C6—H6A	0.9700
N1—H1C	0.8900	C6—H6B	0.9700
C2—C3	1.5298 (13)	N7—H7A	0.8900
C2—H2A	0.9700	N7—H7B	0.8900
C2—H2B	0.9700	N7—H7C	0.8900
Pd1···Cl3^{iii}	3.2044 (9)	Pd2···Cl2^{i}	3.1788 (9)
Pd1···Cl3^{iv}	3.2044 (9)	Pd2···Cl2^{v}	3.1789 (9)
Cl2—Pd1—Cl2^{i}	180.0	C4—C3—H3B	108.6
Cl2—Pd1—Cl1	91.031 (9)	C2—C3—H3B	108.6
Cl2^{i}—Pd1—Cl1	88.970 (10)	H3A—C3—H3B	107.6
Cl2—Pd1—Cl1^{i}	88.969 (10)	C3—C4—C5	113.37 (10)
Cl2^{i}—Pd1—Cl1^{i}	91.030 (10)	C3—C4—H4A	108.9
Cl1—Pd1—Cl1^{i}	180.000 (3)	C5—C4—H4A	108.9
Cl3^{ii}—Pd2—Cl3	180.0	C3—C4—H4B	108.9
Cl3^{ii}—Pd2—Cl4	90.681 (8)	C5—C4—H4B	108.9
Cl3—Pd2—Cl4	89.317 (8)	H4A—C4—H4B	107.7
Cl3^{ii}—Pd2—Cl4^{ii}	89.320 (9)	C6—C5—C4	112.44 (8)
Cl3—Pd2—Cl4^{ii}	90.682 (8)	C6—C5—H5A	109.1
Cl4—Pd2—Cl4^{ii}	180.0	C4—C5—H5A	109.1
C2—N1—H1A	109.5	C6—C5—H5B	109.1
C2—N1—H1B	109.5	C4—C5—H5B	109.1
H1A—N1—H1B	109.5	H5A—C5—H5B	107.8
C2—N1—H1C	109.5	C5—C6—N7	110.74 (8)
H1A—N1—H1C	109.5	C5—C6—H6A	109.5
H1B—N1—H1C	109.5	N7—C6—H6A	109.5
N1—C2—C3	114.20 (9)	C5—C6—H6B	109.5
N1—C2—H2A	108.7	N7—C6—H6B	109.5
C3—C2—H2A	108.7	H6A—C6—H6B	108.1
N1—C2—H2B	108.7	C6—N7—H7A	109.5
C3—C2—H2B	108.7	C6—N7—H7B	109.5
H2A—C2—H2B	107.6	H7A—N7—H7B	109.5
C4—C3—C2	114.57 (9)	C6—N7—H7C	109.5
C4—C3—H3A	108.6	H7A—N7—H7C	109.5
C2—C3—H3A	108.6	H7B—N7—H7C	109.5
N1—C2—C3—C4	67.86 (12)	C3—C4—C5—C6	178.30 (9)
C2—C3—C4—C5	171.37 (9)	C4—C5—C6—N7	172.18 (9)

Symmetry codes: (i) $-x$, $-y$, $-z$; (ii) $-x+1$, $-y+1$, $-z$; (iii) $-x+1$, $-y$, $-z$; (iv) $x-1$, y, z; (v) $x+1$, $y+1$, z.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···Cl2^{i}	0.89	2.88	3.4171 (11)	120
N1—H1*C*···Cl4^{ii}	0.89	2.51	3.3539 (17)	158
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N7—H7*B*···Cl3^{viii}	0.89	2.86	3.3907 (11)	120

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) −*x*+1, −*y*+1, −*z*; (vi) *x*, *y*+1, *z*; (vii) −*x*+1, *y*+1/2, −*z*+1/2; (viii) *x*, −*y*+1/2, *z*+1/2; (ix) *x*+1, −*y*+1/2, *z*+1/2.