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2-(4-Pyridinio)benzimidazolium tetrachloridopalladium(II)

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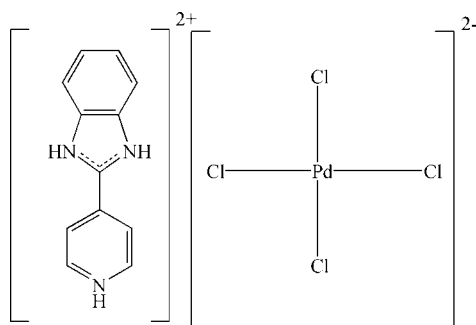
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.030; wR factor = 0.074; data-to-parameter ratio = 18.5.

The asymmetric unit of the title compound, $(\text{C}_{12}\text{H}_{11}\text{N}_3)^+$ - $[\text{PdCl}_4]^{2-}$, consists of a 2-(4-pyridinio)benzimidazolium cation and two half $[\text{PdCl}_4]^{2-}$ anions, which are located on inversion centres. The cations lie in sheets parallel to $(\bar{2}1\bar{2})$. The cations and anions are connected by $\text{N}-\text{H}\cdots\text{Cl}$ and $\text{C}-\text{H}\cdots\text{Cl}$ contacts.

Related literature

For related structures, see: Alcade *et al.* (1992); Chen *et al.* (2006); Huang *et al.* (2004); Wang *et al.* (1999).



Experimental

Crystal data

$(\text{C}_{12}\text{H}_{11}\text{N}_3)[\text{PdCl}_4]$
 $M_r = 445.44$
 Triclinic, $P\bar{1}$

$a = 8.2221$ (1) Å
 $b = 8.3964$ (2) Å
 $c = 12.3768$ (5) Å

$\alpha = 94.09$ (3)°
 $\beta = 97.42$ (2)°
 $\gamma = 116.102$ (10)°
 $V = 752.95$ (11) Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 1.93$ mm⁻¹
 $T = 293$ (2) K
 $0.30 \times 0.15 \times 0.04$ mm

Data collection

Rigaku Mercury CCD diffractometer
 Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2002)
 $T_{\min} = 0.713$, $T_{\max} = 0.916$

5851 measured reflections
 3406 independent reflections
 2816 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.074$
 $S = 1.06$
 3406 reflections

184 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.81$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.61$ e Å⁻³

Table 1
 Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{Cl4}$	0.86	2.55	3.207 (3)	134
$\text{N2}-\text{H2A}\cdots\text{Cl3}^{\dagger}$	0.86	2.32	3.165 (2)	167
$\text{N3}-\text{H3A}\cdots\text{Cl1}$	0.86	2.28	3.138 (3)	172
$\text{C5}-\text{H5A}\cdots\text{Cl1}^{\text{ii}}$	0.93	2.64	3.556 (3)	167

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

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2006) and *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2834).

References

- Alcade, E., Dinares, I., Perez-Garia, L. & Roca, T. (1992). *Synthesis*, pp. 295–398.
 Brandenburg, K. & Putz, H. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
 Chen, L. J., He, X., Xia, C. K., Zhang, Q. Z., Chen, J. T., Yang, W. B. & Lu, C. Z. (2006). *Cryst. Growth Des.* **9**, 2076–2085.
 Huang, X.-C., Zeng, M.-H. & Ng, S. W. (2004). *Acta Cryst.* **E60**, o939–o940.
 Rigaku (2002). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Wang, Z. Y., Wilson, S. R., Foxman, B. M. & Lin, W. B. (1999). *Cryst. Eng.* **2**, 91–100.

supplementary materials

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2-(4-Pyridinio)benzimidazolium tetrachloridopalladium(II)

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Comment

The 2-(4-pyridyl)benzimidazole ligand is often used to act as terminal or bridging ligand in complexes, the noncoordinating N—H and N groups act as hydrogen bond donor or acceptor for the formation of hydrogen bonds, contributing to the crystal packing. Herein we report the synthesis and structure of title complex.

The asymmetry unit of the crystal structure of the title compound comprises one protonated 2-(4-pyridinio)benzimidazolium cation and two independent half $[\text{PdCl}_4]^{2-}$ anion. Each Pd^{II} atom has a slightly distorted square planar coordination geometry (Fig. 1). The N—H \cdots Cl interactions generate a two-dimensional sheet structure, as show in Fig. 2. The sheets are further connected into a three-dimensional network *via* C—H \cdots Cl contacts (Fig. 3).

Experimental

An aqueous solution of PdCl_2 10 ml (0.108 g, 0.61 mmol), 2-(4-pyridyl)benzimidazole (Alcade *et al.*, 1992) (0.12 g, 0.61 mmol) and concentrated HCl (5 ml) was stirred continuously for about 30 min. the solution was allowed to stand at room temperature for several days and produced red crystals of the title compound (yield 85%).

Refinement

After checking their presence in the different map, all H atoms were fixed geometrically and allowed to ride on their parent atoms, with C—H = 0.93 Å, N—H = 0.86 Å, $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C,N})$.

Figures

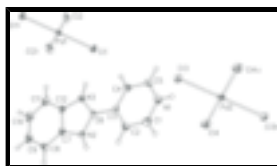


Fig. 1. The structure of the title compound with the atomic labels and 30% probability displacement ellipsoids for non-H atoms. Symmetry codes: (i) $2 - x, 2 - y, -z$; (ii) $-x, -y, 1 - z$.

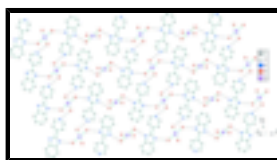


Fig. 2. Hydrogen bond pattern of (I). The H atoms have been omitted for clarity; hydrogen bonds are shown as dashed lines.



Fig. 3. The crystal packing of (I), the N—H \cdots Cl interactions are shown as dashed lines.

2-(4-Pyridinio)benzimidazolium tetrachloridopalladium(II)

Crystal data

(C ₁₂ H ₁₁ N ₃)[PdCl ₄]	$Z = 2$
$M_r = 445.44$	$F_{000} = 436$
Triclinic, $P\bar{1}$	$D_x = 1.964 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 8.2221 (1) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.3964 (2) \text{ \AA}$	Cell parameters from 1904 reflections
$c = 12.3768 (5) \text{ \AA}$	$\theta = 3.3\text{--}27.5^\circ$
$\alpha = 94.09 (3)^\circ$	$\mu = 1.93 \text{ mm}^{-1}$
$\beta = 97.42 (2)^\circ$	$T = 293 (2) \text{ K}$
$\gamma = 116.102 (10)^\circ$	Prism, red
$V = 752.95 (11) \text{ \AA}^3$	$0.30 \times 0.15 \times 0.04 \text{ mm}$

Data collection

Rigaku Mercury CCD diffractometer	3406 independent reflections
Radiation source: fine-focus sealed tube	2816 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.017$
$T = 293(2) \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
ω scans	$\theta_{\text{min}} = 3.3^\circ$
Absorption correction: Multi-scan (CrystalClear; Rigaku, 2002)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.713$, $T_{\text{max}} = 0.916$	$k = -10 \rightarrow 10$
5851 measured reflections	$l = -16 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H-atom parameters constrained
$wR(F^2) = 0.074$	$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2 + 0.584P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
3406 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
184 parameters	$\Delta\rho_{\text{max}} = 0.81 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.61 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	1.0000	1.0000	0.0000	0.02528 (9)
Pd2	0.0000	0.0000	0.5000	0.02595 (9)
C1	0.2054 (5)	0.5925 (5)	0.3660 (3)	0.0415 (8)
H1B	0.1425	0.5664	0.4248	0.050*
C2	0.2983 (5)	0.7664 (4)	0.3483 (3)	0.0367 (7)
H2B	0.2983	0.8586	0.3946	0.044*
C3	0.3925 (4)	0.8040 (4)	0.2610 (2)	0.0281 (6)
C4	0.3862 (5)	0.6625 (4)	0.1924 (3)	0.0389 (8)
H4A	0.4468	0.6845	0.1325	0.047*
C5	0.2904 (5)	0.4902 (4)	0.2129 (3)	0.0390 (8)
H5A	0.2849	0.3947	0.1669	0.047*
C6	0.4975 (4)	0.9893 (4)	0.2438 (2)	0.0269 (6)
C7	0.6195 (4)	1.2852 (4)	0.2627 (3)	0.0318 (7)
C8	0.6740 (5)	1.4663 (4)	0.2955 (3)	0.0428 (8)
H8A	0.6320	1.5050	0.3532	0.051*
C9	0.7928 (5)	1.5835 (5)	0.2379 (4)	0.0519 (10)
H9A	0.8324	1.7056	0.2571	0.062*
C10	0.8567 (5)	1.5272 (5)	0.1519 (4)	0.0523 (10)
H10A	0.9340	1.6124	0.1137	0.063*
C11	0.8097 (5)	1.3500 (5)	0.1212 (3)	0.0428 (8)
H11A	0.8555	1.3130	0.0649	0.051*
C12	0.6896 (4)	1.2291 (4)	0.1793 (3)	0.0326 (7)
N1	0.2050 (4)	0.4609 (4)	0.2996 (2)	0.0376 (6)
H1A	0.1475	0.3527	0.3128	0.045*
N2	0.5007 (3)	1.1324 (3)	0.2995 (2)	0.0299 (6)
H2A	0.4383	1.1300	0.3505	0.036*
N3	0.6107 (4)	1.0441 (3)	0.1709 (2)	0.0305 (6)
H3A	0.6313	0.9751	0.1258	0.037*
Cl1	0.69047 (11)	0.81997 (12)	-0.00991 (7)	0.0429 (2)
Cl2	0.95830 (12)	0.96403 (11)	-0.18905 (6)	0.0393 (2)
Cl3	0.30176 (11)	0.19239 (11)	0.48955 (7)	0.03794 (19)
Cl4	-0.09172 (12)	0.20039 (11)	0.43218 (7)	0.0396 (2)

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.02766 (18)	0.02197 (16)	0.02590 (17)	0.01006 (13)	0.00961 (13)	0.00102 (12)
Pd2	0.02897 (18)	0.02667 (17)	0.02514 (17)	0.01380 (14)	0.01011 (13)	0.00409 (13)
C1	0.046 (2)	0.0370 (18)	0.0417 (19)	0.0152 (16)	0.0212 (16)	0.0093 (15)
C2	0.044 (2)	0.0273 (16)	0.0375 (18)	0.0135 (15)	0.0144 (15)	0.0000 (13)
C3	0.0252 (15)	0.0271 (15)	0.0322 (16)	0.0113 (12)	0.0079 (12)	0.0042 (12)
C4	0.041 (2)	0.0327 (17)	0.0446 (19)	0.0152 (15)	0.0205 (16)	0.0054 (15)
C5	0.044 (2)	0.0293 (16)	0.045 (2)	0.0169 (15)	0.0129 (16)	-0.0019 (14)
C6	0.0248 (14)	0.0272 (14)	0.0292 (15)	0.0119 (12)	0.0059 (12)	0.0036 (12)
C7	0.0278 (16)	0.0287 (15)	0.0382 (17)	0.0123 (13)	0.0045 (13)	0.0077 (13)
C8	0.042 (2)	0.0283 (17)	0.059 (2)	0.0178 (15)	0.0087 (17)	0.0022 (16)
C9	0.041 (2)	0.0237 (16)	0.084 (3)	0.0088 (15)	0.008 (2)	0.0118 (18)
C10	0.043 (2)	0.041 (2)	0.071 (3)	0.0129 (18)	0.016 (2)	0.027 (2)
C11	0.0372 (19)	0.045 (2)	0.047 (2)	0.0156 (16)	0.0148 (16)	0.0177 (17)
C12	0.0295 (17)	0.0289 (15)	0.0392 (17)	0.0125 (13)	0.0065 (14)	0.0077 (13)
N1	0.0400 (16)	0.0259 (13)	0.0485 (17)	0.0138 (12)	0.0150 (13)	0.0101 (12)
N2	0.0311 (14)	0.0261 (13)	0.0356 (14)	0.0144 (11)	0.0118 (11)	0.0031 (11)
N3	0.0338 (14)	0.0278 (13)	0.0322 (14)	0.0140 (11)	0.0141 (11)	0.0045 (11)
Cl1	0.0280 (4)	0.0474 (5)	0.0406 (5)	0.0061 (4)	0.0134 (3)	-0.0099 (4)
Cl2	0.0485 (5)	0.0319 (4)	0.0271 (4)	0.0082 (4)	0.0105 (3)	0.0025 (3)
Cl3	0.0306 (4)	0.0347 (4)	0.0446 (5)	0.0098 (3)	0.0157 (4)	-0.0004 (3)
Cl4	0.0478 (5)	0.0428 (4)	0.0433 (5)	0.0293 (4)	0.0193 (4)	0.0180 (4)

Geometric parameters (\AA , $^\circ$)

Pd1—C12	2.2987 (8)	C7—N2	1.380 (4)
Pd1—C11	2.2989 (8)	C7—C12	1.394 (4)
Pd2—Cl4	2.2937 (8)	C7—C8	1.395 (4)
Pd2—Cl3	2.3170 (8)	C8—C9	1.366 (5)
C1—N1	1.328 (4)	C8—H8A	0.9300
C1—C2	1.368 (4)	C9—C10	1.387 (6)
C1—H1B	0.9300	C9—H9A	0.9300
C2—C3	1.385 (4)	C10—C11	1.373 (5)
C2—H2B	0.9300	C10—H10A	0.9300
C3—C4	1.388 (4)	C11—C12	1.388 (4)
C3—C6	1.458 (4)	C11—H11A	0.9300
C4—C5	1.371 (5)	C12—N3	1.386 (4)
C4—H4A	0.9300	N1—Cl4	3.207 (3)
C5—N1	1.336 (4)	N1—H1A	0.8600
C5—Cl1 ⁱ	3.556 (3)	N2—Cl3 ⁱⁱ	3.165 (2)
C5—H5A	0.9300	N2—H2A	0.8600
C6—N2	1.330 (3)	N3—Cl1	3.138 (3)
C6—N3	1.338 (3)	N3—H3A	0.8600
Cl2—Pd1—Cl2 ⁱⁱⁱ	180.00 (4)	N2—C6—N3	108.6 (3)
Cl2—Pd1—Cl1	89.78 (4)	N2—C6—C3	125.9 (2)

Cl2 ⁱⁱⁱ —Pd1—C11	90.22 (4)	N3—C6—C3	125.5 (3)
Cl2—Pd1—C11	89.78 (4)	N2—C7—C12	106.6 (3)
Cl2 ⁱⁱⁱ —Pd1—C11	90.22 (4)	N2—C7—C8	131.9 (3)
C11—Pd1—C11	0.00 (4)	C12—C7—C8	121.5 (3)
Cl2—Pd1—C11 ⁱⁱⁱ	90.22 (4)	C9—C8—C7	116.0 (3)
Cl2 ⁱⁱⁱ —Pd1—C11 ⁱⁱⁱ	89.78 (4)	C9—C8—H8A	122.0
C11—Pd1—C11 ⁱⁱⁱ	180.0	C7—C8—H8A	122.0
C11—Pd1—C11 ⁱⁱⁱ	180.0	C8—C9—C10	122.4 (3)
Cl4—Pd2—C14	0.00 (7)	C8—C9—H9A	118.8
Cl4—Pd2—C14 ^{iv}	180.0	C10—C9—H9A	118.8
Cl4—Pd2—C14 ^{iv}	180.0	C11—C10—C9	122.2 (3)
Cl4—Pd2—C13 ^{iv}	90.27 (3)	C11—C10—H10A	118.9
Cl4—Pd2—C13 ^{iv}	90.27 (3)	C9—C10—H10A	118.9
Cl4 ^{iv} —Pd2—C13 ^{iv}	89.73 (3)	C10—C11—C12	116.1 (3)
Cl4—Pd2—C13	89.73 (3)	C10—C11—H11A	122.0
Cl4—Pd2—C13	89.73 (3)	C12—C11—H11A	122.0
Cl4 ^{iv} —Pd2—C13	90.27 (3)	N3—C12—C11	132.4 (3)
Cl3 ^{iv} —Pd2—C13	180.0	N3—C12—C7	105.9 (3)
N1—C1—C2	120.2 (3)	C11—C12—C7	121.7 (3)
N1—C1—H1B	119.9	C1—N1—C5	122.5 (3)
C2—C1—H1B	119.9	C1—N1—C14	85.01 (19)
C1—C2—C3	119.5 (3)	C5—N1—C14	151.2 (2)
C1—C2—H2B	120.3	C1—N1—H1A	118.7
C3—C2—H2B	120.3	C5—N1—H1A	118.7
C2—C3—C4	118.6 (3)	C6—N2—C7	109.5 (2)
C2—C3—C6	119.9 (3)	C6—N2—C13 ⁱⁱ	134.59 (18)
C4—C3—C6	121.5 (3)	C7—N2—C13 ⁱⁱ	115.88 (18)
C5—C4—C3	119.9 (3)	C6—N2—H2A	125.2
C5—C4—H4A	120.0	C7—N2—H2A	125.2
C3—C4—H4A	120.0	C6—N3—C12	109.4 (2)
N1—C5—C4	119.3 (3)	C6—N3—C11	130.00 (19)
N1—C5—C11 ⁱ	129.9 (2)	C12—N3—C11	120.44 (18)
C4—C5—C11 ⁱ	110.8 (2)	C6—N3—H3A	125.3
N1—C5—H5A	120.4	C12—N3—H3A	125.3
C4—C5—H5A	120.4		

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x, y+1, z$; (iii) $-x+2, -y+2, -z$; (iv) $-x, -y, -z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots C14	0.86	2.55	3.207 (3)	134
N2—H2A \cdots C13 ⁱⁱ	0.86	2.32	3.165 (2)	167
N3—H3A \cdots C11	0.86	2.28	3.138 (3)	172
C5—H5A \cdots C11 ⁱ	0.93	2.64	3.556 (3)	167

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

Fig. 1

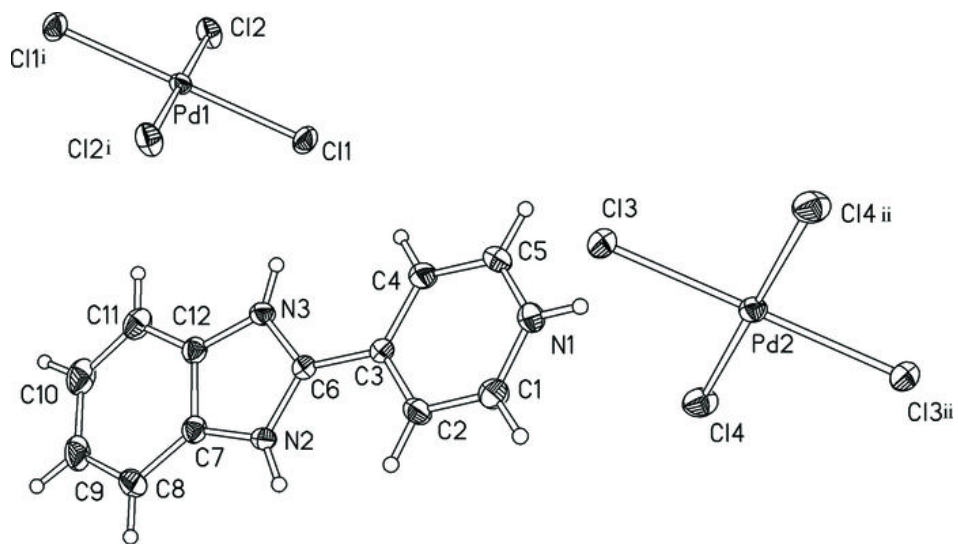


Fig. 2

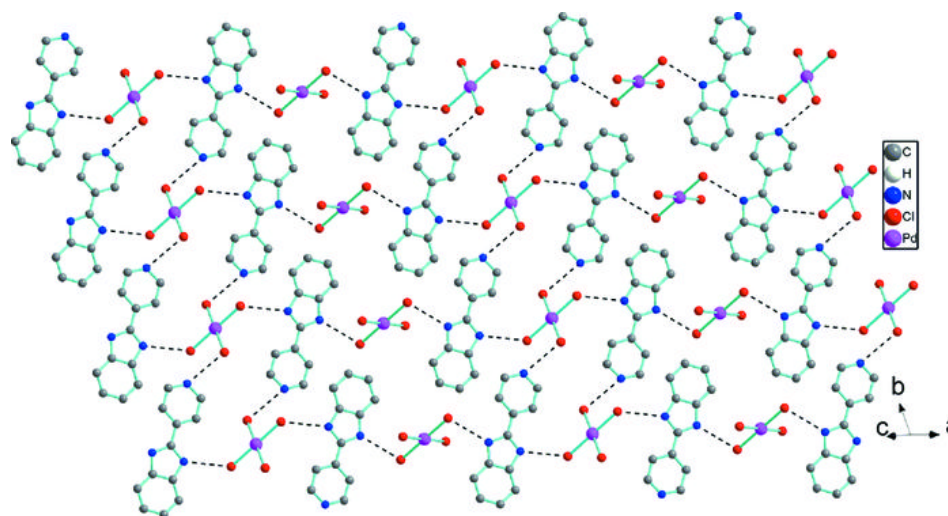


Fig. 3

