

## Bis(2,4,6-trimethylpyridinium) hexachloridoplatinate(IV)

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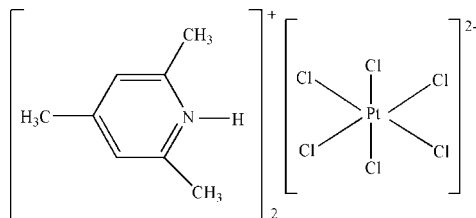
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å;  $R$  factor = 0.037;  $wR$  factor = 0.088; data-to-parameter ratio = 25.5.

The asymmetric unit of the title compound,  $(\text{C}_8\text{H}_{12}\text{N})_2[\text{PtCl}_6]$ , contains one independent protonated 2,4,6-trimethylpyridinium cation and one half of a centrosymmetric  $[\text{PtCl}_6]^{2-}$  anion. The Pt ion has an almost ideal octahedral coordination. In the crystal structure, intramolecular  $\text{N}-\text{H}\cdots\text{Cl}$  and intermolecular  $\text{C}-\text{H}\cdots\text{Cl}$  hydrogen bonds result in the formation of a supramolecular structure.

### Related literature

For general background, see: Rafizadeh *et al.* (2006); Yousefi, Amani & Khavasi (2007); Abedi *et al.* (2008); Hojjat Kashani *et al.* (2008). For related literature, see: Biradha & Zaworotko (1998); Hallfeldt & Urland (2002); Foces-Foces *et al.* (1999); Zordan & Brammer (2004); Hasan *et al.* (2001); Juan *et al.* (1998); Li & Liu (2003); Hu *et al.* (2003); Terzis & Mentzafos (1983); Zordan *et al.* (2005); Yousefi, Ahmadi *et al.* (2007); Yousefi *et al.* (2007a,b); Amani *et al.* (2008).



### Experimental

#### Crystal data

$(\text{C}_8\text{H}_{12}\text{N})_2[\text{PtCl}_6]$   
 $M_r = 652.15$   
 Triclinic,  $P\bar{1}$   
 $a = 7.6302$  (8) Å

$b = 9.1328$  (9) Å  
 $c = 9.4599$  (10) Å  
 $\alpha = 99.201$  (8)°  
 $\beta = 109.683$  (8)°

$\gamma = 108.471$  (8)°  
 $V = 561.87$  (12) Å<sup>3</sup>  
 $Z = 1$   
 Mo  $K\alpha$  radiation

$\mu = 6.96$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 $0.32 \times 0.30 \times 0.25$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1998)  
 $T_{\min} = 0.121$ ,  $T_{\max} = 0.176$   
 6510 measured reflections  
 2962 independent reflections  
 2952 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.099$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$   
 $wR(F^2) = 0.088$   
 $S = 1.18$   
 2962 reflections  
 116 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 1.20$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -1.44$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Pt1—Cl1	2.3225 (11)	Pt1—Cl3	2.3197 (13)
Pt1—Cl2	2.3199 (12)		
Cl2—Pt1—Cl1 <sup>i</sup>	89.35 (5)	Cl3—Pt1—Cl1 <sup>i</sup>	89.90 (5)
Cl2—Pt1—Cl1	90.65 (5)	Cl3—Pt1—Cl2	90.45 (6)
Cl3—Pt1—Cl1	90.10 (5)	Cl3—Pt1—Cl2 <sup>l</sup>	89.55 (6)

Symmetry code: (i)  $-x, -y + 1, -z$ .

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1D}\cdots\text{Cl2}$	0.86	2.45	3.301 (5)	173
$\text{C1}-\text{H1C}\cdots\text{Cl1}^{\text{ii}}$	0.96	2.81	3.743 (6)	165
$\text{C8}-\text{H8A}\cdots\text{Cl3}^{\text{iii}}$	0.96	2.80	3.731 (10)	163

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2526).

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**supplementary materials**

*Acta Cryst.* (2008). E64, m1267-m1268 [ doi:10.1107/S160053680802881X ]

## Bis(2,4,6-trimethylpyridinium) hexachloridoplatinate(IV)

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### Comment

In recent years, there has been considerable interest in proton transfer systems and their structures (Rafizadeh *et al.*, 2006; Yousefi, Amani & Khavasi, 2007; Abedi *et al.*, 2008; Hojjat Kashani *et al.*, 2008). Several proton transfer systems using 2,4,6-trimethylpyridine, with proton donor molecules, such as [2,4,6-tmpy.H]<sub>2</sub>[H<sub>2</sub>BTEC], (II), (Biradha & Zaworotko, 1998), {[2,4,6-tmpy.H]<sub>10</sub>[Er(H<sub>2</sub>O)Cl<sub>5</sub>]<sub>2</sub>[ErCl<sub>6</sub>]3Cl}, (III), (Hallfeldt & Urland, 2002), [2,4,6-tmpy.H][2-NBA], (IV) and [2,4,6-tmpy.H][3,5-NBA], (V), (Foces-Foces *et al.*, 1999) [where 2,4,6-tmpy.H is 2,4,6-trimethylpyridinium, H<sub>2</sub>BTEC is dihydrogen-1,2,4,5-benzenetetracarboxylate, 2-NBA is 2-nitrobenzoate and 3,5-NBA is 3,5-nitrobenzoate] have been synthesized and characterized by single-crystal X-ray diffraction methods.

There are also several proton transfer systems using H<sub>2</sub>[PtCl<sub>6</sub>] with proton acceptor molecules, such as [HpyBr-3]<sub>2</sub>[PtCl<sub>6</sub>].2H<sub>2</sub>O, (VI), and [HpyI-3]<sub>2</sub>[PtCl<sub>6</sub>].2H<sub>2</sub>O, (VII), (Zordan & Brammer, 2004), [BMIM]<sub>2</sub>[PtCl<sub>6</sub>], (VIII), and [EMIM]<sub>2</sub>[PtCl<sub>6</sub>], (IX), (Hasan *et al.*, 2001), {(DABCO)H<sub>2</sub>[PtCl<sub>6</sub>]}, (X), (Juan *et al.*, 1998), {*p*-C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>ImMe)<sub>2</sub>[PtCl<sub>6</sub>]}, (XI), (Li & Liu, 2003), [het][PtCl<sub>6</sub>].2H<sub>2</sub>O, (XII), (Hu *et al.*, 2003), [9-MeGuaH]<sub>2</sub>[PtCl<sub>6</sub>].2H<sub>2</sub>O, (XIII), (Terzis & Mentzafos, 1983), [HpyCl-3]<sub>3</sub>[PtCl<sub>6</sub>]Cl, (XIV), (Zordan *et al.*, 2005), [2,9-dmphen.H]<sub>2</sub>[PtCl<sub>6</sub>], (XV), (Yousefi, Ahmadi *et al.*, 2007), [H<sub>2</sub>DA18C6][PtCl<sub>6</sub>].2H<sub>2</sub>O, (XVI), (Yousefi *et al.*, 2007a), [2,6-dmpy.H]<sub>2</sub>[PtCl<sub>6</sub>], (XVII), (Amani *et al.*, 2008) and [TBA]<sub>3</sub>[PtCl<sub>6</sub>]Cl, (XVIII), (Yousefi *et al.*, 2007b) [where hpy is halo-pyridinium, BMIM<sup>+</sup> is 1-*n*-butyl-3-methylimidazolium, EMIM<sup>+</sup> is 1-ethyl-3-methylimidazolium, DABCO is 1,4-diazabicyclooctane, Im is imidazolium, het is 2-( $\alpha$ -hydroxyethyl)thiamine, 9-MeGuaH is 9-methylguaninium, 2,9-dmphen.H is 2,9-dimethyl-1,10-phenanthroline, H<sub>2</sub>DA18C6 is 1,10-Diazonia-18-crown-6, 2,6-dmpy.H is 2,6-dimethylpyridinium and TBA is tribenzylammonium] have been synthesized and characterized by single-crystal X-ray diffraction methods. We report herein the synthesis and crystal structure of the title compound, (I).

The asymmetric unit of (I), (Fig. 1) contains one independent protonated 2,4,6-trimethylpyridinium cation and one half of a centrosymmetric [PtCl<sub>6</sub>]<sup>2-</sup> anion. The Pt ion has an octahedral coordination. In cation, the bond lengths and angles are in good agreement with the corresponding values in (II) and (IV). In [PtCl<sub>6</sub>]<sup>2-</sup> anion, the Pt—Cl bond lengths and Cl—Pt—Cl bond angles (Table 1) are also within normal ranges, as in (XVI), (XVII) and (XVIII).

In the crystal structure (Fig. 2), intramolecular N—H $\cdots$ Cl and intermolecular C—H $\cdots$ Cl hydrogen bonds (Table 2) result in the formation of a supramolecular structure, in which they may be effective in the stabilization of the structure.

### Experimental

For the preparation of the title compound, (I), a solution of 2,4,6-trimethylpyridine (0.18 g, 1.48 mmol) in methanol (15 ml) was added to a solution of H<sub>2</sub>PtCl<sub>6</sub>.6H<sub>2</sub>O, (0.38 g, 0.74 mmol) in acetonitrile (25 ml) and the resulting yellow solution was

## supplementary materials

stirred for 10 min at 313 K. Then, it was left to evaporate slowly at room temperature. After one week, orange prismatic crystals of (I) were isolated (yield; 0.35 g; 72.5%).

### Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ .

### Figures

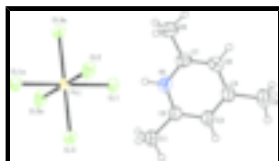


Fig. 1. The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level [symmetry code: (a)  $x, y + 1, z$ ].

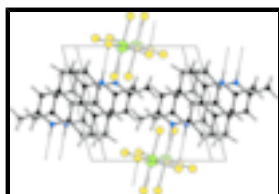


Fig. 2. A packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

### Bis(2,4,6-trimethylpyridinium)hexachloridoplatinate(IV)

#### Crystal data

$(\text{C}_8\text{H}_{12}\text{N})_2[\text{PtCl}_6]$

$M_r = 652.15$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 7.6302\ (8)\ \text{\AA}$

$b = 9.1328\ (9)\ \text{\AA}$

$c = 9.4599\ (10)\ \text{\AA}$

$\alpha = 99.201\ (8)^\circ$

$\beta = 109.683\ (8)^\circ$

$\gamma = 108.471\ (8)^\circ$

$V = 561.87\ (12)\ \text{\AA}^3$

$Z = 1$

$F_{000} = 314$

$D_x = 1.927\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1715 reflections

$\theta = 3.0\text{--}29.1^\circ$

$\mu = 6.96\ \text{mm}^{-1}$

$T = 298\ (2)\ \text{K}$

Prism, orange

$0.32 \times 0.30 \times 0.25\ \text{mm}$

#### Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298\ (2)\ \text{K}$

$\varphi$  and  $\omega$  scans

2962 independent reflections

2952 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.099$

$\theta_{\text{max}} = 29.1^\circ$

$\theta_{\text{min}} = 3.0^\circ$

Absorption correction: multi-scan  
(SADABS; Sheldrick, 1998)  $h = -10 \rightarrow 10$   
 $T_{\min} = 0.121$ ,  $T_{\max} = 0.176$   $k = -12 \rightarrow 12$   
 6510 measured reflections  $l = -12 \rightarrow 12$

### Refinement

Refinement on  $F^2$  Hydrogen site location: inferred from neighbouring sites  
 Least-squares matrix: full H-atom parameters constrained  
 $R[F^2 > 2\sigma(F^2)] = 0.037$   $w = 1/[\sigma^2(F_o^2) + (0.0557P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $wR(F^2) = 0.088$   $(\Delta/\sigma)_{\max} = 0.010$   
 $S = 1.18$   $\Delta\rho_{\max} = 1.20 \text{ e } \text{\AA}^{-3}$   
 2962 reflections  $\Delta\rho_{\min} = -1.44 \text{ e } \text{\AA}^{-3}$   
 116 parameters Extinction correction: SHELXTL (Sheldrick, 1998),  
 $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$   
 Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.115 (6)  
 Secondary atom site location: difference Fourier map

### 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.0000	0.5000	0.0000	0.03163 (12)
Cl1	0.1751 (2)	0.65180 (16)	0.26337 (11)	0.0496 (3)
Cl2	-0.2671 (2)	0.5772 (2)	-0.01978 (15)	0.0522 (3)
Cl3	-0.1501 (2)	0.27660 (16)	0.07023 (13)	0.0486 (3)
N1	-0.2844 (7)	0.7163 (6)	0.3174 (5)	0.0473 (9)
H1D	-0.2918	0.6752	0.2261	0.057*
C1	-0.2767 (12)	0.4641 (8)	0.3642 (6)	0.0567 (14)
H1A	-0.1645	0.4737	0.3364	0.068*
H1B	-0.4017	0.3993	0.2738	0.068*
H1C	-0.2670	0.4137	0.4465	0.068*
C2	-0.2721 (8)	0.6278 (7)	0.4197 (5)	0.0448 (10)
C3	-0.2540 (10)	0.6948 (7)	0.5686 (6)	0.0496 (11)

## supplementary materials

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H3	-0.2448	0.6366	0.6410	0.059*
C4	-0.2496 (10)	0.8492 (8)	0.6091 (6)	0.0533 (12)
C5	-0.2300 (17)	0.9231 (11)	0.7709 (8)	0.077 (2)
H5A	-0.3464	0.9469	0.7620	0.092*
H5B	-0.1094	1.0211	0.8227	0.092*
H5C	-0.2215	0.8483	0.8310	0.092*
C6	-0.2651 (10)	0.9340 (7)	0.4965 (7)	0.0540 (12)
H6	-0.2611	1.0380	0.5221	0.065*
C7	-0.2858 (9)	0.8639 (7)	0.3499 (6)	0.0493 (11)
C8	-0.3106 (14)	0.9436 (10)	0.2211 (9)	0.0687 (18)
H8A	-0.4370	0.8775	0.1325	0.082*
H8B	-0.2007	0.9562	0.1898	0.082*
H8C	-0.3100	1.0478	0.2589	0.082*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pt1	0.03766 (15)	0.03245 (15)	0.02614 (14)	0.01452 (9)	0.01484 (8)	0.00745 (7)
C11	0.0642 (7)	0.0451 (6)	0.0288 (4)	0.0166 (5)	0.0150 (4)	0.0042 (3)
C12	0.0546 (6)	0.0726 (8)	0.0457 (5)	0.0397 (6)	0.0252 (5)	0.0200 (5)
C13	0.0621 (7)	0.0406 (6)	0.0440 (5)	0.0148 (5)	0.0274 (5)	0.0148 (4)
N1	0.049 (2)	0.052 (2)	0.0380 (16)	0.0178 (18)	0.0172 (15)	0.0113 (15)
C1	0.076 (4)	0.061 (3)	0.045 (2)	0.039 (3)	0.028 (2)	0.016 (2)
C2	0.052 (2)	0.047 (2)	0.0385 (18)	0.024 (2)	0.0190 (17)	0.0110 (16)
C3	0.062 (3)	0.052 (3)	0.041 (2)	0.028 (2)	0.0239 (19)	0.0121 (18)
C4	0.062 (3)	0.054 (3)	0.044 (2)	0.026 (2)	0.023 (2)	0.0060 (19)
C5	0.116 (7)	0.067 (5)	0.052 (3)	0.044 (5)	0.039 (4)	0.005 (3)
C6	0.059 (3)	0.042 (3)	0.057 (3)	0.018 (2)	0.023 (2)	0.012 (2)
C7	0.046 (2)	0.047 (3)	0.049 (2)	0.0134 (19)	0.0164 (18)	0.0172 (19)
C8	0.074 (4)	0.063 (4)	0.067 (3)	0.025 (3)	0.024 (3)	0.033 (3)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Pt1—C11	2.3225 (11)	C3—H3	0.9300
Pt1—C11 <sup>i</sup>	2.3225 (11)	C4—C6	1.409 (9)
Pt1—C12	2.3199 (12)	C4—C5	1.507 (8)
Pt1—C12 <sup>i</sup>	2.3199 (12)	C5—H5A	0.9600
Pt1—C13 <sup>i</sup>	2.3197 (13)	C5—H5B	0.9600
Pt1—C13	2.3197 (13)	C5—H5C	0.9600
N1—H1D	0.8600	C6—C7	1.365 (9)
C1—C2	1.488 (8)	C6—H6	0.9300
C1—H1A	0.9600	C7—N1	1.338 (8)
C1—H1B	0.9600	C7—C8	1.505 (8)
C1—H1C	0.9600	C8—H8A	0.9600
C2—N1	1.355 (7)	C8—H8B	0.9600
C2—C3	1.385 (6)	C8—H8C	0.9600
C3—C4	1.388 (8)		
C11—Pt1—C11 <sup>i</sup>	180.0	C2—C3—C4	119.7 (5)

Cl2—Pt1—Cl1 <sup>i</sup>	89.35 (5)	C2—C3—H3	120.2
Cl2 <sup>i</sup> —Pt1—Cl1 <sup>i</sup>	90.65 (5)	C4—C3—H3	120.2
Cl2—Pt1—Cl1	90.65 (5)	C3—C4—C6	118.9 (5)
Cl2—Pt1—Cl2 <sup>i</sup>	180.0	C3—C4—C5	120.0 (6)
Cl3—Pt1—Cl1	90.10 (5)	C6—C4—C5	121.1 (6)
Cl3 <sup>i</sup> —Pt1—Cl1 <sup>i</sup>	90.10 (5)	C4—C5—H5A	109.5
Cl3—Pt1—Cl1 <sup>i</sup>	89.90 (5)	C4—C5—H5B	109.5
Cl3—Pt1—Cl2	90.45 (6)	H5A—C5—H5B	109.5
Cl3 <sup>i</sup> —Pt1—Cl2 <sup>i</sup>	90.45 (6)	C4—C5—H5C	109.5
Cl3—Pt1—Cl2 <sup>i</sup>	89.55 (6)	H5A—C5—H5C	109.5
Cl3 <sup>i</sup> —Pt1—Cl3	180.0	H5B—C5—H5C	109.5
C2—N1—H1D	118.0	C7—C6—C4	120.1 (5)
C7—N1—C2	123.9 (5)	C7—C6—H6	119.9
C7—N1—H1D	118.0	C4—C6—H6	119.9
C2—C1—H1A	109.5	N1—C7—C6	118.9 (5)
C2—C1—H1B	109.5	N1—C7—C8	117.6 (6)
H1A—C1—H1B	109.5	C6—C7—C8	123.5 (6)
C2—C1—H1C	109.5	C7—C8—H8A	109.5
H1A—C1—H1C	109.5	C7—C8—H8B	109.5
H1B—C1—H1C	109.5	H8A—C8—H8B	109.5
N1—C2—C3	118.5 (5)	C7—C8—H8C	109.5
N1—C2—C1	117.3 (4)	H8A—C8—H8C	109.5
C3—C2—C1	124.2 (5)	H8B—C8—H8C	109.5
C1—C2—N1—C7	178.8 (6)	C3—C4—C6—C7	0.6 (10)
C3—C2—N1—C7	-1.9 (9)	C5—C4—C6—C7	-179.0 (7)
N1—C2—C3—C4	0.2 (9)	C4—C6—C7—N1	-2.2 (10)
C1—C2—C3—C4	179.5 (6)	C4—C6—C7—C8	177.8 (7)
C2—C3—C4—C6	0.4 (10)	C6—C7—N1—C2	2.9 (9)
C2—C3—C4—C5	180.0 (7)	C8—C7—N1—C2	-177.0 (6)

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

*Hydrogen-bond geometry (Å, °)*

<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—H1D $\cdots$ Cl2	0.86	2.45	3.301 (5)	173
C1—H1C $\cdots$ Cl1 <sup>ii</sup>	0.96	2.81	3.743 (6)	165
C8—H8A $\cdots$ Cl3 <sup>iii</sup>	0.96	2.80	3.731 (10)	163

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



Fig. 1

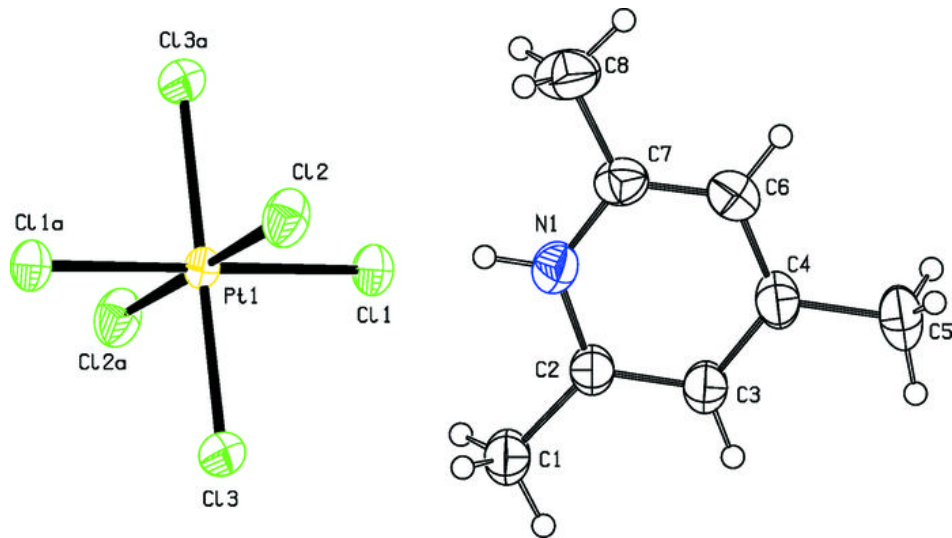


Fig. 2

