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3,3'-{Ethane-1,2-diylbis[carbonyl-bis(azanediyl)]}dipyridinium tetrachloridoplatinate(II)

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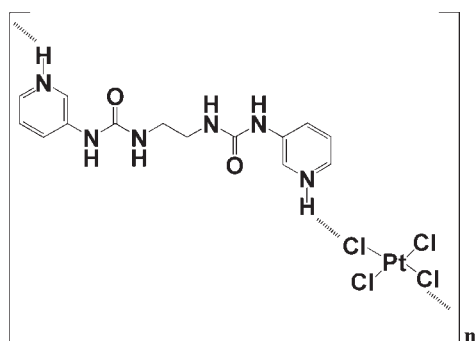
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Key indicators: single-crystal X-ray study; *T* = 100 K; mean  $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$ ; disorder in main residue; *R* factor = 0.025; *wR* factor = 0.064; data-to-parameter ratio = 14.2.

In the crystal structure of the title compound,  $(\text{C}_{14}\text{H}_{18}\text{N}_6\text{O}_2)\text{-}[\text{PtCl}_4]$ , the cation and square-planar anion are located on special positions (on a twofold axis and an inversion centre, respectively). In the crystal structure,  $\text{N}-\text{H}\cdots\text{Cl}$  hydrogen bonds lead to a staircase-like motif. The central ethane backbone of the cation is disordered over two positions of equal occupancy.

Related literature

For organic-inorganic hybrid compounds displaying  $\text{N}-\text{H}\cdots\text{Cl}$ -metal hydrogen bonds, see: Adams *et al.* (2007); Deifela & Cahill (2009). Orpen *et al.* (2004); Krishna Kumar *et al.* (2005, 2006).



Experimental

Crystal data

$(\text{C}_{14}\text{H}_{18}\text{N}_6\text{O}_2)[\text{PtCl}_4]$

*M<sub>r</sub>* = 639.23

Monoclinic, *C*2/*c*  
*a* = 17.8126 (13) Å  
*b* = 7.0799 (5) Å  
*c* = 15.5765 (12) Å  
 $\beta$  = 103.332 (1)°  
*V* = 1911.4 (2) Å<sup>3</sup>

*Z* = 4  
 Mo *K*α radiation  
 $\mu$  = 7.92 mm<sup>-1</sup>  
*T* = 100 K  
 0.22 × 0.14 × 0.05 mm

Data collection

Bruker APEXII CCD area-detector diffractometer  
 Absorption correction: multi-scan (*SADABS*; Bruker, 2006)  
*T<sub>min</sub>* = 0.275, *T<sub>max</sub>* = 0.693

7045 measured reflections  
 1877 independent reflections  
 1693 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$   
 $wR(F^2) = 0.064$   
*S* = 0.97  
 1877 reflections  
 132 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 1.51 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.89 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> - <i>H</i> ⋯ <i>A</i>	<i>D</i> - <i>H</i>	<i>H</i> ⋯ <i>A</i>	<i>D</i> ⋯ <i>A</i>	<i>D</i> - <i>H</i> ⋯ <i>A</i>
<i>N</i> 1- <i>H</i> 1⋯ <i>Cl</i> 1 <sup>i</sup>	0.76 (5)	2.55 (5)	3.259 (4)	156 (5)

Symmetry code: (i)  $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$ .

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2* and *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*, *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5171).

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## supporting information

*Acta Cryst.* (2010). E66, m270 [doi:10.1107/S1600536810004253]

### 3,3'-{Ethane-1,2-diylbis[carbonylbis(azanediy)]}dipyridinium tetrachloridoplatinate(II)

N. N. Adarsh, D. Krishna Kumar and Parthasarathi Dastidar

#### S1. Comment

Suitable single crystals of the title compound were obtained by the resultant reaction of **BPEBU** and  $[K_2(PtCl_4)]$  in acidic medium (see experimental). The asymmetric unit consists of half of  $(PtCl_4)$  and one half molecule of bis-protonated **BPEBU**. The ORTEP diagram with 50% probability is given in Fig. 1. The metal center Pt(II) displayed a slight distorted square planar geometry [ $Cl-Pt-Cl = 89.91(4)-90.09(4)^\circ$ ]. In the crystal structure, the bis-protonated ligand **BPEBU** displays *syn-anti-syn* conformation (scheme 1).  $N-H\cdots Cl$  hydrogen bonds lead to a staircase like structure (Fig. 2).

#### S2. Experimental

**Synthesis:** The title compound was synthesized by layering a methanolic solution of **BPEBU** (30 mg, 0.1 mmol) containing HCl (MeOH : HCl = 8 ml : 2 ml) over an aqueous solution of  $[K_2(PtCl_4)]$  (41.5 mg, 0.1 mmol) containing HCl ( $H_2O$  : HCl = 8 ml : 2 ml). After a period of two weeks, X-ray quality single crystals were obtained (yield = 30 mg, 47%). FT-IR (KBr,  $cm^{-1}$ ): 3325 (b, O—H, N—H stretch), 3082 (b, aromatic C—H stretch), 2965 (b, aliphatic C—H), 1686 (s, urea C=O stretch), 1612 (s, urea N—H bend), 1580, 1520, 1480, 1422, 1331, 1264, 1238, 1191, 1111, 1024, 989, 864, 800, 694.

#### S3. Refinement

H-atoms bonded to C were refined using a riding model with C—H ranging from 0.95 Å to 0.99 Å and  $U(H) = 1.2U_{eq}(C)$ . The coordinates of the H atoms bonded to N were refined with  $U(H) = 1.2U_{eq}(N)$ . The central C—C bond in the cation (located on the 2 fold axis) is disordered over two equally occupied positions.

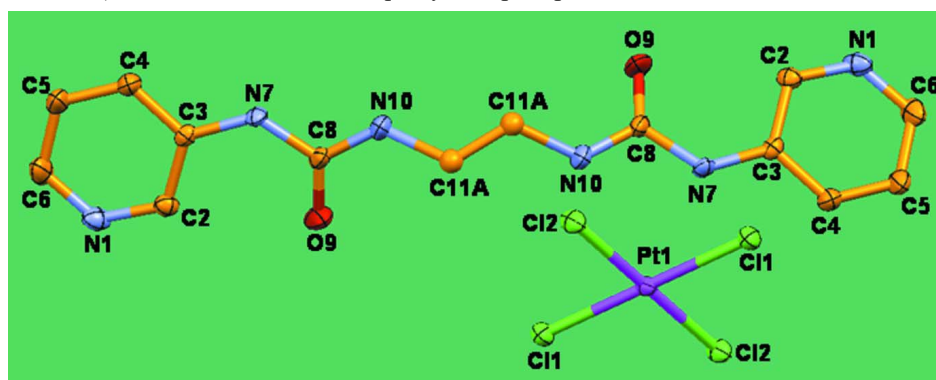


Figure 1

Ortep diagram of title compound with anisotropic displacement ellipsoids drawn at the 50% probability level. H atoms omitted for clarity.

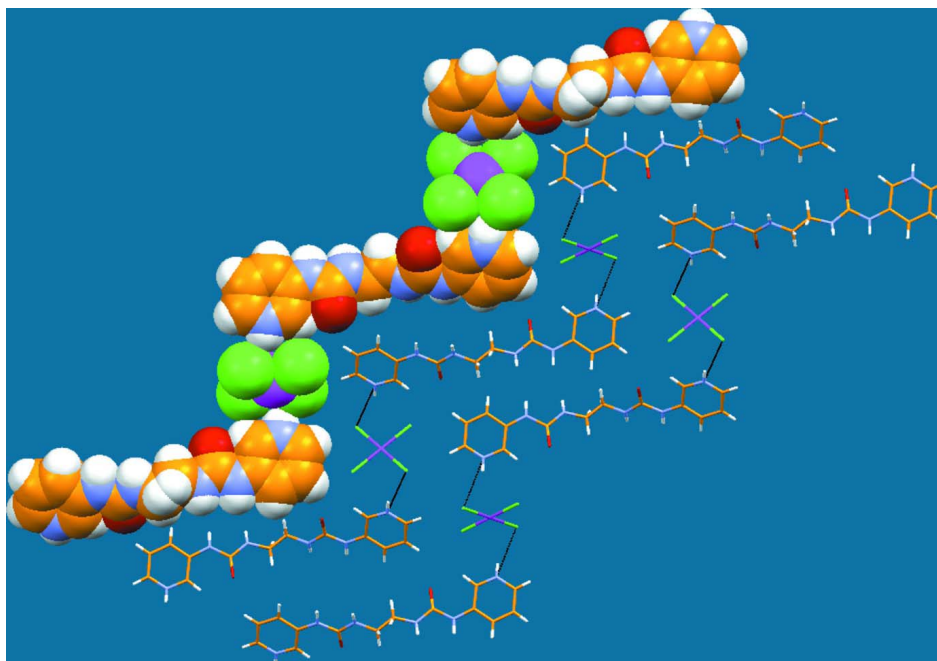


Figure 2

Staircase like hydrogen bonded network via N—H...Cl interactions

### 3,3'-{Ethane-1,2-diylbis[carbonylbis(azanediy)]}dipyridinium tetrachloridoplatinate(II)

#### Crystal data

(C<sub>14</sub>H<sub>18</sub>N<sub>6</sub>O<sub>2</sub>)[PtCl<sub>4</sub>]

*M<sub>r</sub>* = 639.23

Monoclinic, *C2/c*

*a* = 17.8126 (13) Å

*b* = 7.0799 (5) Å

*c* = 15.5765 (12) Å

$\beta$  = 103.332 (1)°

*V* = 1911.4 (2) Å<sup>3</sup>

*Z* = 4

*F*(000) = 1224

*D<sub>x</sub>* = 2.221 Mg m<sup>-3</sup>

Mo *K*α radiation,  $\lambda$  = 0.71073 Å

Cell parameters from 566 reflections

$\theta$  = 2.4–27.8°

$\mu$  = 7.92 mm<sup>-1</sup>

*T* = 100 K

Plate, yellow

0.22 × 0.14 × 0.05 mm

#### Data collection

Bruker APEXII CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$ - $\omega$  scan

Absorption correction: multi-scan

(*SADABS*; Bruker, 2006)

*T<sub>min</sub>* = 0.275, *T<sub>max</sub>* = 0.693

7045 measured reflections

1877 independent reflections

1693 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.044

$\theta_{\max}$  = 26.0°,  $\theta_{\min}$  = 2.4°

*h* = -21→21

*k* = -8→8

*l* = -19→19

#### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.025

*wR*(*F*<sup>2</sup>) = 0.064

*S* = 0.97

1877 reflections

132 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 atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 6.3133P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 1.51 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.89 \text{ e } \text{\AA}^{-3}$

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Pt1	0.5000	0.5000	0.0000	0.01584 (10)	
Cl1	0.39171 (5)	0.65021 (15)	-0.07921 (6)	0.0228 (2)	
Cl2	0.50224 (6)	0.69911 (14)	0.11747 (7)	0.0226 (2)	
N1	0.1742 (2)	-0.0639 (6)	-0.1087 (3)	0.0250 (8)	
H1	0.139 (3)	-0.108 (7)	-0.096 (3)	0.030*	
C2	0.2331 (3)	-0.0133 (5)	-0.0434 (3)	0.0193 (9)	
H2	0.2297	-0.0254	0.0163	0.023*	
C3	0.3001 (2)	0.0577 (6)	-0.0639 (2)	0.0160 (8)	
C4	0.3007 (2)	0.0717 (7)	-0.1528 (3)	0.0223 (8)	
H4	0.3455	0.1183	-0.1690	0.027*	
C5	0.2379 (3)	0.0195 (6)	-0.2176 (3)	0.0305 (12)	
H5	0.2391	0.0310	-0.2781	0.037*	
C6	0.1730 (3)	-0.0497 (7)	-0.1941 (3)	0.0304 (10)	
H6	0.1288	-0.0861	-0.2378	0.036*	
N7	0.36384 (19)	0.1145 (5)	-0.0011 (2)	0.0176 (7)	
C8	0.3757 (2)	0.0918 (6)	0.0896 (3)	0.0214 (9)	
O9	0.3290 (2)	0.0132 (4)	0.1240 (3)	0.0367 (10)	
N10	0.4417 (2)	0.1661 (6)	0.1358 (2)	0.0275 (9)	
C11A	0.4643 (5)	0.2153 (13)	0.2283 (5)	0.0202 (16)*	0.50
H11A	0.4227	0.1819	0.2582	0.024*	0.50
H11B	0.4740	0.3528	0.2351	0.024*	0.50
C11B	0.4631 (5)	0.1063 (12)	0.2315 (5)	0.0185 (16)*	0.50
H11C	0.4728	-0.0314	0.2365	0.022*	0.50
H11D	0.4213	0.1381	0.2614	0.022*	0.50
H7	0.400 (3)	0.164 (6)	-0.020 (3)	0.022*	
H10	0.467 (3)	0.219 (7)	0.116 (3)	0.032*	

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pt1	0.01344 (14)	0.01733 (15)	0.01748 (14)	-0.00161 (7)	0.00507 (9)	-0.00225 (7)
Cl1	0.0172 (5)	0.0302 (5)	0.0211 (5)	0.0044 (4)	0.0046 (4)	0.0037 (4)
Cl2	0.0192 (5)	0.0254 (5)	0.0239 (5)	-0.0025 (4)	0.0062 (4)	-0.0096 (4)
N1	0.0220 (19)	0.0246 (19)	0.030 (2)	-0.0071 (16)	0.0105 (16)	-0.0072 (17)
C2	0.019 (2)	0.019 (2)	0.021 (2)	-0.0005 (14)	0.0066 (19)	-0.0024 (15)
C3	0.020 (2)	0.0134 (17)	0.0147 (19)	0.0009 (16)	0.0045 (16)	-0.0006 (16)
C4	0.020 (2)	0.030 (2)	0.018 (2)	-0.0010 (18)	0.0067 (17)	-0.0012 (18)
C5	0.023 (3)	0.050 (3)	0.018 (2)	0.0020 (19)	0.004 (2)	-0.0089 (18)
C6	0.023 (2)	0.040 (2)	0.026 (3)	-0.002 (2)	0.003 (2)	-0.017 (2)
N7	0.0184 (17)	0.0221 (18)	0.0135 (16)	-0.0055 (13)	0.0064 (14)	0.0016 (13)
C8	0.019 (2)	0.033 (2)	0.0128 (19)	0.0068 (17)	0.0043 (16)	0.0044 (17)
O9	0.028 (2)	0.063 (3)	0.0219 (18)	-0.0011 (14)	0.0108 (16)	0.0151 (14)
N10	0.022 (2)	0.046 (2)	0.0143 (18)	-0.0013 (16)	0.0037 (15)	-0.0045 (16)

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

Pt1—Cl1 <sup>i</sup>	2.2971 (9)	C6—H6	0.9500
Pt1—Cl1	2.2971 (9)	N7—C8	1.389 (5)
Pt1—Cl2 <sup>i</sup>	2.3028 (9)	N7—H7	0.83 (5)
Pt1—Cl2	2.3028 (9)	C8—O9	1.223 (5)
N1—C6	1.330 (6)	C8—N10	1.337 (6)
N1—C2	1.331 (6)	N10—C11A	1.447 (8)
N1—H1	0.76 (5)	N10—C11B	1.511 (9)
C2—C3	1.397 (6)	N10—H10	0.71 (5)
C2—H2	0.9500	C11A—C11B <sup>ii</sup>	1.513 (10)
C3—N7	1.377 (5)	C11A—H11A	0.9900
C3—C4	1.392 (5)	C11A—H11B	0.9900
C4—C5	1.374 (7)	C11B—C11A <sup>ii</sup>	1.513 (10)
C4—H4	0.9500	C11B—H11C	0.9900
C5—C6	1.380 (7)	C11B—H11D	0.9900
C5—H5	0.9500		
Cl1 <sup>i</sup> —Pt1—Cl1	180.0	C3—N7—C8	126.6 (3)
Cl1 <sup>i</sup> —Pt1—Cl2 <sup>i</sup>	90.11 (4)	C3—N7—H7	116 (3)
Cl1—Pt1—Cl2 <sup>i</sup>	89.89 (4)	C8—N7—H7	117 (3)
Cl1 <sup>i</sup> —Pt1—Cl2	89.89 (4)	O9—C8—N10	123.1 (4)
Cl1—Pt1—Cl2	90.11 (4)	O9—C8—N7	122.6 (4)
Cl2 <sup>i</sup> —Pt1—Cl2	180.0	N10—C8—N7	114.3 (4)
C6—N1—C2	124.9 (4)	C8—N10—C11A	129.8 (5)
C6—N1—H1	118 (4)	C8—N10—C11B	114.3 (4)
C2—N1—H1	117 (4)	C11A—N10—C11B	30.3 (4)
N1—C2—C3	119.0 (4)	C8—N10—H10	122 (4)
N1—C2—H2	120.5	C11A—N10—H10	105 (4)
C3—C2—H2	120.5	C11B—N10—H10	123 (4)
N7—C3—C4	119.4 (4)	N10—C11A—C11B <sup>ii</sup>	107.6 (6)

N7—C3—C2	123.4 (4)	N10—C11A—H11A	110.2
C4—C3—C2	117.2 (4)	C11B <sup>ii</sup> —C11A—H11A	110.2
C5—C4—C3	121.3 (4)	N10—C11A—H11B	110.2
C5—C4—H4	119.3	C11B <sup>ii</sup> —C11A—H11B	110.2
C3—C4—H4	119.3	H11A—C11A—H11B	108.5
C4—C5—C6	119.4 (5)	N10—C11B—C11A <sup>ii</sup>	105.2 (5)
C4—C5—H5	120.3	N10—C11B—H11C	110.7
C6—C5—H5	120.3	C11A <sup>ii</sup> —C11B—H11C	110.7
N1—C6—C5	118.1 (4)	N10—C11B—H11D	110.7
N1—C6—H6	121.0	C11A <sup>ii</sup> —C11B—H11D	110.7
C5—C6—H6	121.0	H11C—C11B—H11D	108.8
C6—N1—C2—C3	-1.1 (7)	C3—N7—C8—O9	1.0 (7)
N1—C2—C3—N7	179.7 (4)	C3—N7—C8—N10	-177.7 (4)
N1—C2—C3—C4	0.2 (6)	O9—C8—N10—C11A	-17.3 (8)
N7—C3—C4—C5	-178.9 (4)	N7—C8—N10—C11A	161.5 (6)
C2—C3—C4—C5	0.6 (6)	O9—C8—N10—C11B	13.7 (7)
C3—C4—C5—C6	-0.5 (7)	N7—C8—N10—C11B	-167.5 (5)
C2—N1—C6—C5	1.2 (7)	C8—N10—C11A—C11B <sup>ii</sup>	122.2 (6)
C4—C5—C6—N1	-0.3 (7)	C11B—N10—C11A—C11B <sup>ii</sup>	53.4 (10)
C4—C3—N7—C8	-172.8 (4)	C8—N10—C11B—C11A <sup>ii</sup>	-176.5 (5)
C2—C3—N7—C8	7.7 (6)	C11A—N10—C11B—C11A <sup>ii</sup>	-48.3 (10)

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

*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—H1 $\cdots$ Cl2 <sup>iii</sup>	0.76 (5)	2.55 (5)	3.259 (4)	156 (5)

Symmetry code: (iii)  $-x+1/2, -y+1/2, -z$ .