

Crystal structure of dichlorido(1,2-phenylenediamine- κ^2N,N')platinum(II)

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Received 31 May 2017

Accepted 7 June 2017

Edited by M. Weil, Vienna University of
Technology, Austria

Keywords: crystal structure; columnar structure;
infinite metal chain; platinum(II) complex;
hydrogen bonding.

CCDC reference: 1055177

Supporting information: this article has
supporting information at journals.iucr.org/e

The Pt^{II} atom in the title compound, [PtCl₂{(C₆H₄)(NH₂)₂}], lies on a twofold rotation axis and has a slightly distorted square-planar coordination environment defined by two N atoms of an 1,2-phenylenediamine ligand and two Cl⁻ ions. In the crystal, the planar complex molecules are stacked parallel to the *c* axis, resulting in a columnar structure. In a column, an infinite almost straight Pt···Pt chain is formed, suggesting weak metal–metal interactions [Pt···Pt = 3.3475 (8) Å]. The crystal packing is stabilized by a three-dimensional N—H···Cl hydrogen-bonding network between the amino groups and the Cl ligands of adjacent molecules.

1. Chemical context

The title compound, dichlorido(1,2-phenylenediamine- κ^2N,N')platinum(II) [PtCl₂{(C₆H₄)(NH₂)₂}], (I), which was originally prepared by Connors *et al.* (1972), is a member of the family of derivatives of *cis*-diamminedichlorido-platinum(II), *cis*-[PtCl₂(NH₃)₂] (*cis*-platin). Since the discovery of the antitumor activity of *cis*-platin (Rosenberg *et al.*, 1965), numerous derivatives and analogues of *cis*-platin have been prepared and investigated. However, reports on the corresponding crystal structures are rather scarce, probably because of the difficulty in obtaining crystals suitable for X-ray analysis, in part owing to poor solubility. Although the antitumor activity (Connors *et al.*, 1972; Meischen *et al.*, 1976) and the chemical stabilities (Köckerbauer & Bednarski, 1996) of the title compound have been reported, its crystal structure has not been determined so far. In the course of our study of the deprotonation and redox properties of a platinum complex with 1,2-phenylenediamine as a ligand (Konno & Matsushita, 2006*a,b*), we have successfully obtained single crystals of the title compound and report here its crystal structure.

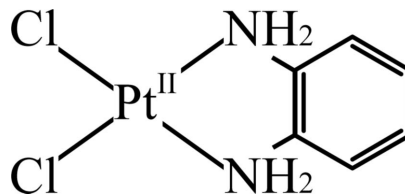
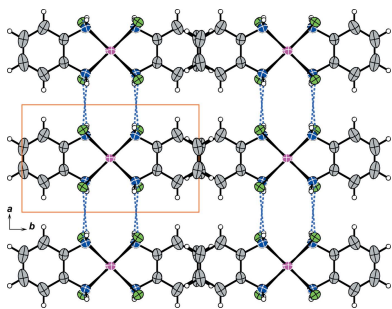


Table 1
Selected geometric parameters (Å, °).

Pt1–N1	2.040 (4)	C1–C2	1.372 (6)
Pt1–Cl1	2.3213 (13)	C1–C1 ⁱⁱ	1.386 (9)
Pt1–Pt1 ⁱ	3.3475 (8)	C2–C3	1.377 (11)
N1–C1	1.445 (6)	C3–C3 ⁱⁱ	1.38 (3)
N1–Pt1–N1 ⁱⁱ	83.6 (3)	Pt1 ⁱ –Pt1–Pt1 ⁱⁱⁱ	176.513 (11)
N1–Pt1–Cl1	91.39 (15)	C1–N1–Pt1	110.8 (3)
Cl1 ⁱⁱ –Pt1–Cl1	93.69 (7)	C2–C1–C1 ⁱⁱ	119.8 (3)
N1–Pt1–Pt1 ⁱ	92.07 (14)	C2–C1–N1	122.7 (5)
Cl1–Pt1–Pt1 ⁱ	93.80 (4)	C1 ⁱⁱ –C1–N1	117.4 (2)
N1–Pt1–Pt1 ⁱⁱⁱ	85.32 (14)	C1–C2–C3	120.3 (8)
Cl1–Pt1–Pt1 ⁱⁱⁱ	88.59 (4)	C2–C3–C3 ⁱⁱ	119.8 (6)

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

2. Structural commentary

The molecular structure of (I) is displayed in Fig. 1. The platinum compound (I) is isostructural with the palladium compound $[\text{PdCl}_2\{(\text{C}_6\text{H}_4)(\text{NH}_2)_2\}]$ reported previously (Konno & Matsushita, 2017). The Pt^{II} atom lies on a twofold rotation axis. Hence the asymmetric unit comprises half of a $[\text{PtCl}_2\{(\text{C}_6\text{H}_4)(\text{NH}_2)_2\}]$ molecule, the other half being completed by application of twofold rotation symmetry. The Pt^{II} atom is coordinated by two N atoms of an 1,2-phenylenediamine ligand and by two Cl[−] ions in a slightly distorted square-planar configuration (Table 1). The r.m.s. deviation of the least-squares plane formed by atoms Pt1, N1, C1, C2 and C3 is 0.0121 Å. The structural parameters of the coordination sphere around Pt^{II} in the crystal of (I) (Table 1) are consistent with those found in *cis*- $[\text{PtCl}_2(\text{NH}_3)_2]$ (Milburn & Truter, 1966), $[\text{PtCl}_2(\text{en})]$ (en is ethylenediamine; Iball *et al.*, 1975), *cis*- $[\text{PtCl}_2(L)_2]$ (*L* is cyclohexylamine; Lock *et al.*, 1980), $[\text{PtCl}_2(\text{cis-dac})] \cdot 0.33\text{-hydrate}$ (dac is 1,2-diaminocyclohexane; Lock & Pilon, 1981), *cis*- $[\text{PtCl}_2(L')(\text{NH}_3)]$ (*L'* is cyclobutylamine; Rochon & Melanson, 1986), $[\text{PtCl}_2(\text{Me}_2\text{en})]$ (*Me*₂en is *N,N*-dimethylethylenediamine; Melanson *et al.*, 1987), $[\text{PtCl}_2(\text{tn})]$ (tn is 1,3-diaminopropane; Odoko & Okabe, 2006), $[\text{PtCl}_2(L'')]$ (*L''* is 2-morpholinoethylamine; Shi *et al.*, 2006), $[\text{PtCl}_2(\text{Me}_4\text{en})]$ (*Me*₄en is *N,N,N',N'*-tetramethylethylenediamine; Asiri *et al.*, 2012). Bond lengths and angles of the 1,2-phenylenediamine moiety (Table 1) are not significantly

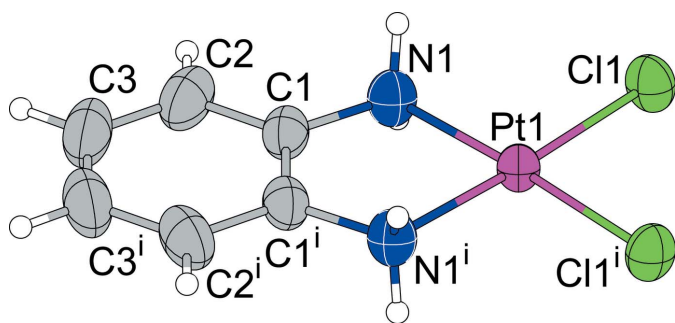


Figure 1
A view of the molecular structure of compound (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms. [Symmetry code: (i) $-x + 1, y, -z + \frac{1}{2}$]

different from those found in the bis(1,2-phenylenediamine)-platinum(II) complex, $[\text{Pt}(\text{C}_6\text{H}_8\text{N}_2)_2]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$ [$\text{N}-\text{C} = 1.450 (2) \text{ \AA}$, $\text{C}-\text{C} = 1.365 (6)–1.389 (4) \text{ \AA}$; Konno & Matsushita, 2006a] or in isostructural dichlorido(1,2-phenylenediamine)palladium(II) [$\text{N}-\text{C} = 1.458 (2) \text{ \AA}$, $\text{C}-\text{C} = 1.371 (3)–1.416 (8) \text{ \AA}$; Konno & Matsushita, 2017].

3. Supramolecular features

As shown in Fig. 2, the neutral planar molecules of (I) stack parallel to the *c* axis, resulting in a columnar structure. The planar $[\text{PtCl}_2\{(\text{C}_6\text{H}_4)(\text{NH}_2)_2\}]$ units are arranged in parallel and the 1,2-phenylenediamine moieties alternate with each other as a result of the *c*-glide operation. In the column, an infinite, almost straight $[\text{Pt} \cdots \text{Pt} \cdots \text{Pt} = 176.513 (11)^\circ]$ platinum chain is formed with a short interatomic distance [$\text{Pt} \cdots \text{Pt} = 3.3475 (8) \text{ \AA}$], suggesting weak metal–metal interactions. The infinite palladium chain of the isostructural Pd complex is straighter [$\text{Pd} \cdots \text{Pd} \cdots \text{Pd} = 179.232 (7)^\circ$] than the platinum chain. The $\text{Pt} \cdots \text{Pt}$ distance in (I) is slightly shorter than those of *cis*- $[\text{PtCl}_2(\text{NH}_3)_2]$ [3.372 (2) and 3.409 (2) Å; Milburn & Truter, 1966] or $[\text{PtCl}_2(\text{en})]$ [3.381 Å; Iball *et al.*, 1975], and is considerably shorter than that of $[\text{PtCl}_2(\text{tn})]$ [3.646 Å; Odoko & Okabe, 2006], all of which have similar columnar structures.

The intermolecular $\text{Pt} \cdots \text{Pt}$ distance of (I) suggests that the columnar structure is stabilized by weak metal–metal interactions. The columnar structure of (I) is further stabilized by

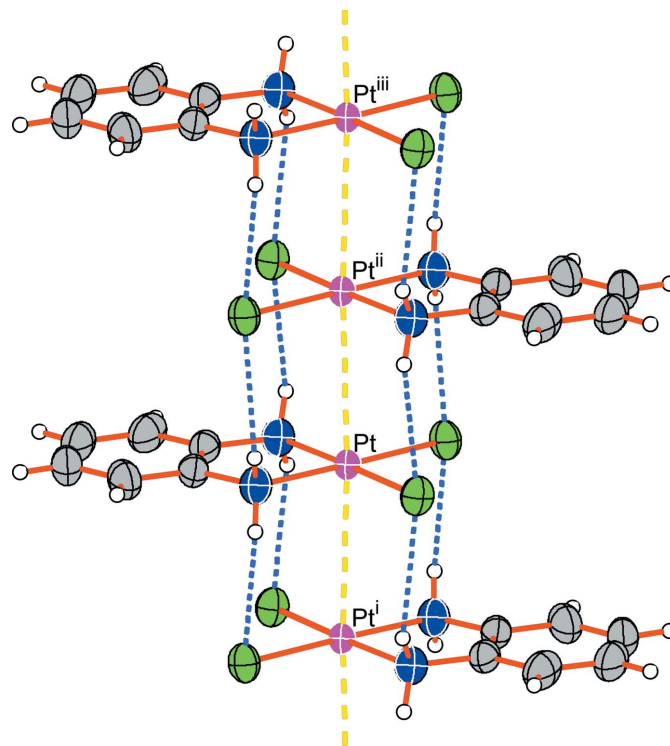


Figure 2
A view of the columnar structure of compound (I). Light-blue dashed lines represent hydrogen bonds between adjacent molecules in the column. Yellow dashed lines indicate the short contact between Pt atoms in the column. [Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $x, y, z + 1$]

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots Cl1^{iv}$	0.90	2.57	3.353 (4)	146
$N1-H1B\cdots Cl1^v$	0.90	2.71	3.381 (4)	133
$N1-H1B\cdots Cl1^{vi}$	0.90	2.73	3.320 (5)	124

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

intermolecular $N-H\cdots Cl$ hydrogen bonds between adjacent molecules in the column (Fig. 2 and Table 2). Intercolumnar hydrogen bonds also help to stabilize the crystal packing of the columns (Fig. 3, and Table 2).

4. Synthesis and crystallization

Compound (I) was prepared using a method modified from that described by Connors *et al.* (1972) as follows. To an aqueous HCl solution (1.0 M, 15 ml) of $K_2[PtCl_4]$ (0.241 mmol, 100 mg) was slowly added an aqueous HCl solution (1.0 M, 15 ml) of 1,2-phenylenediamine (0.241 mmol, 26 mg), and then the solution was sealed in a screw-cap vial and was kept at room temperature for one week in the dark. Pale-brown needle-like crystals suitable for X-ray analysis were obtained (yield 52%). Elemental analysis found: C 19.26, H 2.23, N 7.30%; calculated for $C_6H_8Cl_2N_2Pt$: C 19.26, H 2.16, N 7.49%. Elemental analysis was carried out by the Laboratory of Organic Elemental Analysis, Department of Chemistry, Graduate School of Science, The University of Tokyo.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. One reflection (010) was omitted

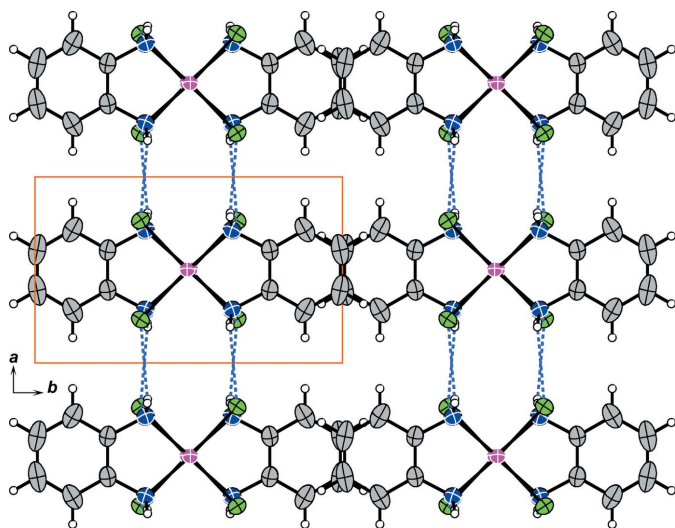


Figure 3
The crystal packing of compound (I), viewed along the c axis. Light-blue dashed lines represent intercolumnar hydrogen bonds. Solid orange lines indicate the unit cell.

Table 3
Experimental details.

Crystal data	
Chemical formula	$[PtCl_2(C_6H_8N_2)]$
M_r	374.13
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	7.087 (2), 10.446 (3), 6.6920 (16)
β (°)	116.61 (2)
V (Å ³)	442.9 (2)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	16.38
Crystal size (mm)	0.26 × 0.13 × 0.07
Data collection	
Diffractometer	Rigaku R-AXIS RAPID imaging-plate
Absorption correction	Multi-scan (<i>ABSCOR</i> ; Higashi, 1995)
T_{min}, T_{max}	0.116, 0.304
No. of measured, independent and observed [$F^2 > 2\sigma(F^2)$] reflections	10875, 1587, 1480
R_{int}	0.030
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.757
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.034, 0.099, 1.18
No. of reflections	1587
No. of parameters	52
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	4.62, -1.74

Computer programs: *RAPID-AUTO* (Rigaku, 1998), *SIR92* (Altomare *et al.*, 1994), *DIAMOND* (Brandenburg, 2017), *SHELXL97* (Sheldrick, 2008) and *pubCIF* (Westrip, 2010).

in the final refinement because it was obstructed by the beam-stop. H atoms were placed in geometrically calculated positions and refined as riding, with $C(\text{aromatic})-H = 0.93$ and $N-H = 0.90$ Å, and with $U_{iso}(H) = 1.2U_{eq}(C, N)$. The maximum and minimum electron density peaks are located 0.80 and 0.74 Å, respectively, from atom Pt1.

Funding information

Funding for this research was provided by: Ministry of Education, Culture, Sports, Science and Technology, MEXT-Supported Program for the Strategic Research Foundation at Private Universities (award No. S1311027).

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supporting information

Acta Cryst. (2017). E73, 1009-1012 [https://doi.org/10.1107/S2056989017008477]

Crystal structure of dichlorido(1,2-phenylenediamine- κ^2N,N')platinum(II)

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Computing details

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *RAPID-AUTO* (Rigaku, 1998); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2017); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

Dichlorido(1,2-phenylenediamine- κ^2N,N')platinum(II)

Crystal data

[PtCl₂(C₆H₈N₂)]

$M_r = 374.13$

Monoclinic, *P2/c*

Hall symbol: -P 2yc

$a = 7.087$ (2) Å

$b = 10.446$ (3) Å

$c = 6.6920$ (16) Å

$\beta = 116.61$ (2)°

$V = 442.9$ (2) Å³

$Z = 2$

$F(000) = 340$

$D_x = 2.805$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å

Cell parameters from 12924 reflections

$\theta = 2.0$ – 32.6 °

$\mu = 16.38$ mm⁻¹

$T = 296$ K

Needle, pale brown

$0.26 \times 0.13 \times 0.07$ mm

Data collection

Rigaku R-AXIS RAPID imaging-plate diffractometer

Radiation source: X-ray sealed tube

Graphite monochromator

Detector resolution: 10.00 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.116$, $T_{\max} = 0.304$

10875 measured reflections

1587 independent reflections

1480 reflections with $F^2 > 2\sigma(F^2)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 32.6$ °, $\theta_{\min} = 3.2$ °

$h = -10 \rightarrow 10$

$k = -15 \rightarrow 15$

$l = -10 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.099$

$S = 1.18$

1587 reflections

52 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

$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.3105P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 4.62$ e Å⁻³

$\Delta\rho_{\min} = -1.74$ e Å⁻³

Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c [1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0039 (12)

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.

Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)

- 2.7022 (0.0123) x - 0.0000 (0.0000) y + 6.6740 (0.0026) z = 0.3174 (0.0059)

* 0.0000 (0.0000) Pt1 * -0.0185 (0.0028) C11 * 0.0206 (0.0042) N1 * 0.0050 (0.0039) C1 * -0.0017 (0.0044) C2 * 0.0031 (0.0116) C3 * 0.0185 (0.0028) C11_\$6 * -0.0206 (0.0042) N1_\$6 * -0.0050 (0.0039) C1_\$6 * 0.0017 (0.0044) C2_\$6 * -0.0031 (0.0116) C3_\$6

Rms deviation of fitted atoms = 0.0121

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 (\AA^2)

	x	y	z	U_{iso}^*/U_{eq}
Pt1	0.5000	0.504875 (16)	0.2500	0.03029 (13)
C11	0.23327 (19)	0.65687 (13)	0.1392 (2)	0.0429 (3)
N1	0.2863 (7)	0.3592 (4)	0.1666 (8)	0.0407 (9)
H1A	0.2144	0.3653	0.2480	0.049*
H1B	0.1934	0.3657	0.0213	0.049*
C1	0.3910 (7)	0.2364 (4)	0.2066 (6)	0.0400 (8)
C2	0.2835 (10)	0.1225 (5)	0.1621 (9)	0.0563 (12)
H2	0.1370	0.1224	0.1016	0.068*
C3	0.391 (3)	0.0081 (5)	0.207 (2)	0.068 (3)
H3	0.3182	-0.0690	0.1780	0.082*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.02635 (17)	0.02835 (16)	0.03208 (18)	0.000	0.00944 (11)	0.000
C11	0.0335 (5)	0.0383 (5)	0.0513 (6)	0.0058 (4)	0.0140 (4)	0.0012 (4)
N1	0.0347 (18)	0.0371 (18)	0.043 (2)	-0.0017 (14)	0.0108 (16)	-0.0011 (15)
C1	0.050 (2)	0.0328 (18)	0.0365 (18)	-0.0028 (15)	0.0185 (17)	-0.0012 (14)
C2	0.067 (3)	0.046 (3)	0.054 (3)	-0.018 (2)	0.026 (2)	-0.005 (2)
C3	0.112 (10)	0.037 (3)	0.063 (6)	-0.017 (3)	0.047 (6)	-0.006 (2)

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

Pt1—N1	2.040 (4)	N1—H1B	0.9000
Pt1—N1 ⁱ	2.040 (4)	C1—C2	1.372 (6)
Pt1—C11 ⁱ	2.3213 (13)	C1—C1 ⁱ	1.386 (9)
Pt1—C11	2.3213 (13)	C2—C3	1.377 (11)
Pt1—Pt1 ⁱⁱ	3.3475 (8)	C2—H2	0.9300

Pt1—Pt1 ⁱⁱⁱ	3.3475 (8)	C3—C3 ⁱ	1.38 (3)
N1—C1	1.445 (6)	C3—H3	0.9300
N1—H1A	0.9000		
N1—Pt1—N1 ⁱ	83.6 (3)	C1—N1—Pt1	110.8 (3)
N1—Pt1—Cl1 ⁱ	174.82 (12)	C1—N1—H1A	109.5
N1 ⁱ —Pt1—Cl1 ⁱ	91.39 (15)	Pt1—N1—H1A	109.5
N1—Pt1—Cl1	91.39 (15)	C1—N1—H1B	109.5
N1 ⁱ —Pt1—Cl1	174.82 (12)	Pt1—N1—H1B	109.5
Cl1 ⁱ —Pt1—Cl1	93.69 (7)	H1A—N1—H1B	108.1
N1—Pt1—Pt1 ⁱⁱ	92.07 (14)	C2—C1—C1 ⁱ	119.8 (3)
N1 ⁱ —Pt1—Pt1 ⁱⁱ	85.32 (14)	C2—C1—N1	122.7 (5)
Cl1 ⁱ —Pt1—Pt1 ⁱⁱ	88.59 (4)	C1 ⁱ —C1—N1	117.4 (2)
Cl1—Pt1—Pt1 ⁱⁱ	93.80 (4)	C1—C2—C3	120.3 (8)
N1—Pt1—Pt1 ⁱⁱⁱ	85.32 (14)	C1—C2—H2	119.8
N1 ⁱ —Pt1—Pt1 ⁱⁱⁱ	92.07 (14)	C3—C2—H2	119.8
Cl1 ⁱ —Pt1—Pt1 ⁱⁱⁱ	93.80 (4)	C2—C3—C3 ⁱ	119.8 (6)
Cl1—Pt1—Pt1 ⁱⁱⁱ	88.59 (4)	C2—C3—H3	120.1
Pt1 ⁱⁱ —Pt1—Pt1 ⁱⁱⁱ	176.513 (11)	C3 ⁱ —C3—H3	120.1
Pt1 ⁱⁱ —Pt1—N1—C1	84.8 (3)	Pt1 ⁱⁱⁱ —Pt1—N1—C1	-92.9 (3)

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1A...Cl1 ^{iv}	0.90	2.57	3.353 (4)	146
N1—H1B...Cl1 ^v	0.90	2.71	3.381 (4)	133
N1—H1B...Cl1 ^{vi}	0.90	2.73	3.320 (5)	124

Symmetry codes: (iv) $x, -y+1, z+1/2$; (v) $x, -y+1, z-1/2$; (vi) $-x, -y+1, -z$.