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cis-{1-Butyl-3-[2-(phenylsulfanyl)ethyl]-4-imidazolin-2-yl-κ^2C^2,S'}dichloridoplatinum(II)

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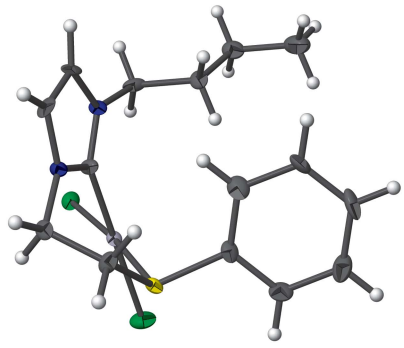
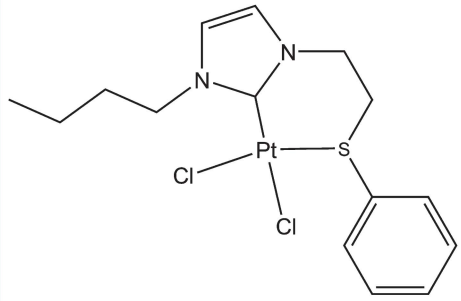
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $[PtCl_2(C_{15}H_{20}N_2S)]$, comprises one Pt^{II} ion, one *N*-heterocyclic carbene(NHC)-thioether ligand and two chloride ions. The Pt^{II} ion is four-coordinated by one C atom and one S atom of the NHC-thioether ligand, and by two chloride ions, forming an approximately square-planar geometry. In the crystal, the molecules are linked *via* C—H···Cl and C—H···π interactions, forming a layer parallel to the *ab* plane.

3D view



Chemical scheme



Structure description

Nitrogen heterocyclic carbene (NHC) exhibits attractive advantages such as simple operation and mild conditions in organic catalytic synthesis (Enders *et al.*, 2007). In addition, as a neutral two-electron donor, NHC is currently regarded as the most effective ligand for the synthesis of new organometallic complexes owing to its unique features (Hahn & Jahnke, 2008; Nelson & Nolan, 2013). The first distinctive characteristic is the strong donor property of NHC ligands, which makes the interaction with metal center closer (Perrin *et al.*, 2001; Chianese *et al.*, 2003). The second one is that NHC can be flexibly modified by introducing functional groups onto the nitrogen atoms of the *N*-heterocycle ring. Over the past two decades, numerous attempts have been made to construct diverse donor-functionalized NHCs and their organometallic complexes, and N-, O- and P-functionalized NHCs have been developed and applied in organic synthesis, drug discovery and materials science (Kühl, 2007). However, there are still rare investigations of NHC with S-donor complexes (Liu *et al.*, 2017). As soft and electron-rich ligands, thioethers usually have versatile coordination chemistry, and can form strong *M*—S bonds with the metal center (Bierenstiel & Cross, 2011; Yuan & Huynh, 2012). The development of new organometallic complexes bearing NHC-thioether ligands (Rosen *et al.*, 2013) is thus highly desirable. In recent years, NHC complexes with group 10 metals have received increasing attention because of their catalytic activities. In contrast to

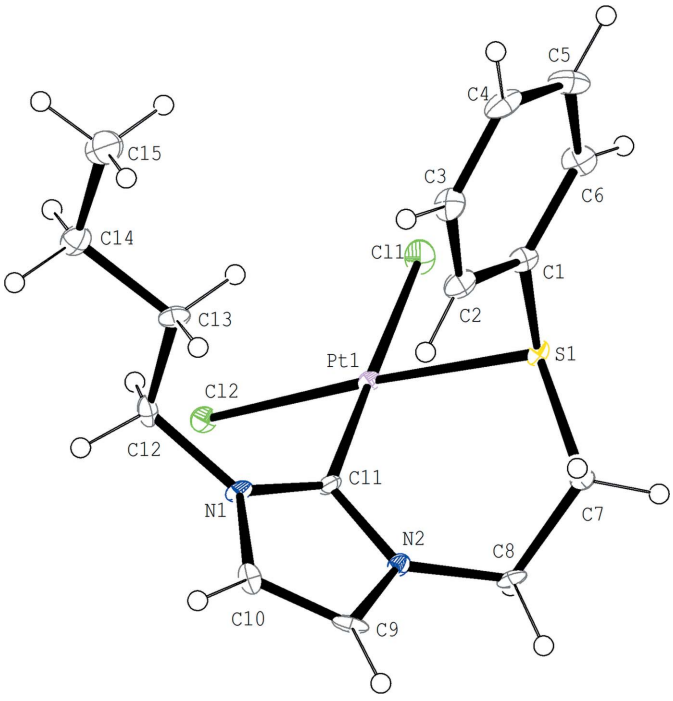


Figure 1
The structure of the title complex, with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

complexes of lighter homologues, Pt^{II}-NHC complexes have been less well studied. The novel title metal Pt^{II} complex combined with an NHC-thioether ligand was designed and synthesized.

The asymmetric unit of the title complex is composed of one Pt^{II} ion, one NHC-thioether ligand, and two chloride ions. As shown in Fig. 1, the Pt^{II} ion is four-coordinated by one C atom and one S atom of the NHC-thioether ligand, and by two chloride ions in a nearly square-planar environment. The thioether side chain coordinates to the Pt^{II} atom in a chelating fashion, forming a six-membered ring with a distorted boat conformation. The Pt—C and Pt—S bond lengths are 1.968 (12) and 2.266 (3) Å, respectively, while the C—Pt—S bond angle is 87.93 (11)°. The two Pt—Cl bond lengths are different from each other [Pt1—Cl1 = 2.360 (3) Å and Pt1—Cl2 = 2.329 (3) Å]. In the crystal, molecules are linked *via* C—H···Cl and C—H···π interactions (Table 1), forming a layer parallel to the *ab* plane (Figs. 2 and 3). A weak intramolecular C—H···π interaction is also observed.

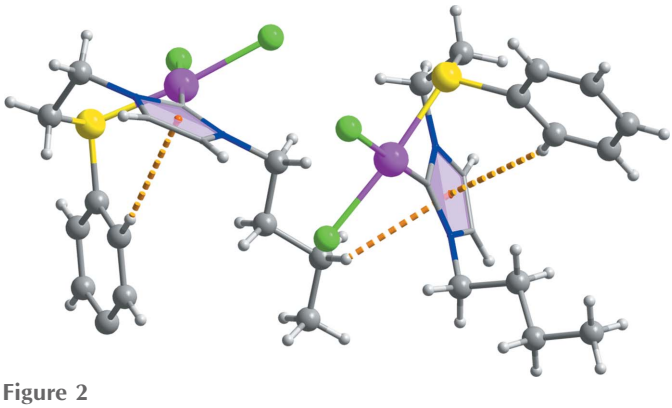


Figure 2
A packing diagram of the title compound, showing intra- and intermolecular C—H···π interactions (dashed lines).

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

*Cg*1 is the centroid of the N1/C10/C9/N2/C11 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···Cl2^{i}	0.93	2.58	3.485 (12)	163
C2—H2···*Cg*1	0.93	2.98	3.828 (13)	151
C14—H14*A*···*Cg*1^{ii}	0.97	2.82	3.480 (13)	126

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$[PtCl_2(C_{15}H_{20}N_2S)]$
M_r	526.38
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, *b*, *c* (Å)	8.4254 (3), 10.1535 (4), 20.2262 (10)
V (Å^3)	1730.30 (13)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	8.53
Crystal size (mm)	0.12 × 0.11 × 0.09
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, AtlasS2
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.310, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11246, 3045, 2911
R_{int}	0.050
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.073, 1.10
No. of reflections	3045
No. of parameters	190
No. of restraints	6
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.27, −0.90
Absolute structure	Flack *x* determined using 1166 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.020 (7)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

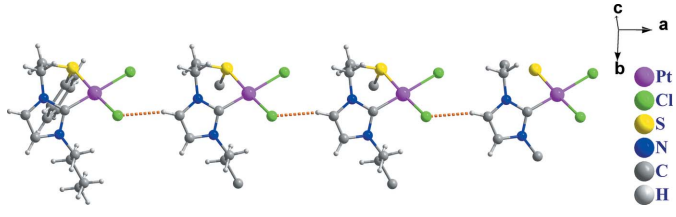


Figure 3
A view of the crystal packing of the title complex. Dashed lines denote the intermolecular C—H···Cl hydrogen bonds.

Synthesis and crystallization

N-Heterocyclic carbene (NHC)-thioether ligand was synthesized by a slight modification of a reported procedure (Liu *et al.*, 2017). Butyl-imidazole and 2-chloroethylbenzene sulfide (molar ratio 1: 1) were dissolved in acetonitrile at 393 K for 2 days to obtain a dark-brown liquid, and then the solvent was removed by evaporation. The residue was washed repeatedly with diethyl ether, and a brownish-yellow solid was obtained.

The title complex was synthesized from the reaction of the NHC-thioether ligand with potassium tetrachloroplatinate. A reaction tube was charged with the NHC-thioether ligand (0.1710 g, 0.576 m*M*) and 6 ml of acetonitrile. The tube was evacuated and back-filled with nitrogen. Then a solution of potassium tetrachloroplatinate (0.200 g, 0.480 m*M*) in 2 ml of water was added in the dark. Keeping it in the dark, the reaction mixture was allowed to stir at 353 K for 24 h. The mixture was concentrated *in vacuo* and purified by silica gel column chromatography. Pale-yellow rectangular crystals were obtained from the solution at room temperature.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The anisotropy of displacement ellipsoid of atom C9 was restrained with *ISOR*.

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full crystallographic data

IUCrData (2020). **5**, x201433 [https://doi.org/10.1107/S2414314620014339]

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Crystal data

$[PtCl_2(C_{15}H_{20}N_2S)]$
M_r = 526.38
Orthorhombic, $P2_12_12_1$
a = 8.4254 (3) Å
b = 10.1535 (4) Å
c = 20.2262 (10) Å
V = 1730.30 (13) Å^3
Z = 4
$F(000)$ = 1008
D_x = 2.021 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6072 reflections
θ = 2.3–29.3°
μ = 8.53 mm^{-1}
T = 100 K
Block, colourless
0.12 × 0.11 × 0.09 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, AtlasS2 diffractometer
Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 5.2684 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2015)
T_{min} = 0.310, T_{max} = 1.000
11246 measured reflections
3045 independent reflections
2911 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −9→10
k = −10→12
l = −24→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.073
S = 1.10
3045 reflections
190 parameters
6 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0237P)^2 + 8.5737P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.90 e Å^{-3}
Absolute structure: Flack x determined using 1166 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.020 (7)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pt1	0.63221 (5)	0.45155 (4)	0.36816 (2)	0.01172 (13)
Cl1	0.8671 (4)	0.3701 (3)	0.41687 (15)	0.0251 (7)
Cl2	0.7685 (3)	0.4970 (3)	0.27062 (15)	0.0166 (7)
S1	0.5012 (4)	0.3876 (3)	0.46058 (16)	0.0132 (6)
N1	0.3999 (10)	0.6337 (9)	0.2997 (5)	0.013 (2)
N2	0.2959 (10)	0.4495 (10)	0.3301 (5)	0.013 (2)
C1	0.4671 (13)	0.5293 (12)	0.5109 (6)	0.017 (3)
C2	0.3753 (16)	0.6352 (11)	0.4903 (6)	0.022 (3)
H2	0.324679	0.633418	0.449406	0.027*
C3	0.3602 (18)	0.7440 (11)	0.5317 (6)	0.023 (3)
H3	0.301393	0.816485	0.517916	0.027*
C4	0.4319 (15)	0.7454 (13)	0.5932 (7)	0.026 (3)
H4	0.421705	0.818696	0.620501	0.031*
C5	0.5187 (15)	0.6376 (14)	0.6141 (7)	0.028 (3)
H5	0.565556	0.637828	0.655695	0.034*
C6	0.5362 (14)	0.5289 (13)	0.5730 (6)	0.023 (3)
H6	0.594069	0.456209	0.587208	0.028*
C7	0.2978 (14)	0.3408 (13)	0.4385 (6)	0.017 (3)
H7A	0.270431	0.259251	0.460755	0.021*
H7B	0.225027	0.408416	0.453583	0.021*
C8	0.2785 (13)	0.3224 (11)	0.3637 (7)	0.016 (3)
H8A	0.358118	0.261421	0.347493	0.019*
H8B	0.174644	0.285757	0.354193	0.019*
C9	0.1750 (14)	0.5213 (13)	0.3000 (6)	0.020 (3)
H9	0.069889	0.495503	0.294379	0.024*
C10	0.2414 (14)	0.6358 (12)	0.2805 (6)	0.015 (3)
H10	0.190345	0.703945	0.258351	0.018*
C11	0.4343 (14)	0.5184 (11)	0.3288 (6)	0.016 (3)
C12	0.5054 (15)	0.7492 (12)	0.2959 (6)	0.018 (3)
H12A	0.477671	0.801717	0.257510	0.021*
H12B	0.614370	0.719995	0.290687	0.021*
C13	0.4914 (14)	0.8325 (11)	0.3576 (6)	0.018 (3)
H13A	0.533278	0.783493	0.394901	0.022*
H13B	0.380163	0.850067	0.366229	0.022*
C14	0.5803 (14)	0.9636 (13)	0.3520 (6)	0.023 (3)
H14A	0.691832	0.946477	0.343681	0.028*
H14B	0.538795	1.012962	0.314704	0.028*
C15	0.5640 (17)	1.0449 (15)	0.4139 (7)	0.037 (4)
H15A	0.620925	1.126163	0.408726	0.055*

H15B	0.453847	1.063416	0.421815	0.055*
H15C	0.606756	0.996986	0.450767	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.0068 (2)	0.0125 (2)	0.0158 (2)	−0.00009 (17)	0.0003 (2)	−0.0010 (2)
Cl1	0.0119 (14)	0.0317 (17)	0.0317 (18)	0.0032 (16)	−0.0018 (16)	0.0097 (14)
Cl2	0.0094 (15)	0.0230 (15)	0.0174 (16)	−0.0007 (11)	0.0022 (12)	−0.0003 (12)
S1	0.0116 (15)	0.0097 (15)	0.0183 (17)	−0.0005 (11)	0.0018 (13)	0.0005 (13)
N1	0.004 (5)	0.014 (5)	0.022 (6)	0.001 (4)	−0.004 (4)	0.000 (4)
N2	0.010 (3)	0.012 (3)	0.016 (3)	0.000 (2)	−0.001 (2)	0.001 (2)
C1	0.011 (6)	0.020 (7)	0.020 (7)	0.000 (5)	0.004 (5)	−0.006 (6)
C2	0.013 (6)	0.024 (7)	0.030 (7)	−0.002 (6)	0.002 (7)	−0.003 (6)
C3	0.029 (7)	0.010 (6)	0.029 (8)	0.005 (6)	0.002 (7)	−0.003 (5)
C4	0.021 (7)	0.023 (7)	0.033 (9)	−0.006 (5)	0.011 (6)	−0.018 (7)
C5	0.017 (7)	0.042 (9)	0.026 (9)	0.001 (6)	−0.005 (6)	−0.012 (7)
C6	0.013 (7)	0.025 (8)	0.032 (8)	0.007 (5)	−0.004 (5)	0.000 (6)
C7	0.015 (6)	0.022 (7)	0.016 (7)	−0.007 (5)	−0.002 (5)	0.001 (6)
C8	0.008 (6)	0.016 (6)	0.023 (7)	−0.003 (4)	−0.005 (6)	−0.004 (6)
C9	0.012 (7)	0.031 (8)	0.018 (7)	0.005 (5)	−0.012 (5)	−0.005 (6)
C10	0.015 (6)	0.013 (6)	0.017 (7)	0.010 (5)	0.003 (5)	0.004 (6)
C11	0.014 (6)	0.012 (7)	0.020 (7)	−0.005 (5)	−0.007 (5)	0.003 (5)
C12	0.013 (7)	0.022 (7)	0.018 (7)	0.003 (5)	0.002 (5)	0.010 (6)
C13	0.010 (6)	0.021 (6)	0.023 (8)	0.000 (5)	−0.005 (5)	−0.003 (6)
C14	0.016 (6)	0.022 (7)	0.031 (8)	0.000 (5)	−0.003 (5)	0.003 (6)
C15	0.039 (8)	0.024 (7)	0.047 (9)	−0.001 (7)	−0.017 (7)	0.007 (8)

Geometric parameters (Å, º)

Pt1—Cl1	2.360 (3)	C6—H6	0.9300
Pt1—Cl2	2.329 (3)	C7—H7A	0.9700
Pt1—S1	2.266 (3)	C7—H7B	0.9700
Pt1—C11	1.968 (12)	C7—C8	1.533 (18)
S1—C1	1.786 (12)	C8—H8A	0.9700
S1—C7	1.834 (12)	C8—H8B	0.9700
N1—C10	1.390 (15)	C9—H9	0.9300
N1—C11	1.343 (15)	C9—C10	1.349 (17)
N1—C12	1.474 (15)	C10—H10	0.9300
N2—C8	1.465 (15)	C12—H12A	0.9700
N2—C9	1.393 (14)	C12—H12B	0.9700
N2—C11	1.360 (14)	C12—C13	1.513 (17)
C1—C2	1.388 (17)	C13—H13A	0.9700
C1—C6	1.385 (17)	C13—H13B	0.9700
C2—H2	0.9300	C13—C14	1.532 (16)
C2—C3	1.394 (16)	C14—H14A	0.9700
C3—H3	0.9300	C14—H14B	0.9700
C3—C4	1.383 (19)	C14—C15	1.507 (18)

C4—H4	0.9300	C15—H15A	0.9600
C4—C5	1.383 (19)	C15—H15B	0.9600
C5—H5	0.9300	C15—H15C	0.9600
C5—C6	1.388 (19)		

Cl2—Pt1—Cl1	90.54 (11)	N2—C8—C7	109.9 (10)
S1—Pt1—Cl1	87.93 (11)	N2—C8—H8A	109.7
S1—Pt1—Cl2	174.77 (11)	N2—C8—H8B	109.7
C11—Pt1—Cl1	179.0 (4)	C7—C8—H8A	109.7
C11—Pt1—Cl2	90.4 (4)	C7—C8—H8B	109.7
C11—Pt1—S1	91.1 (4)	H8A—C8—H8B	108.2
C1—S1—Pt1	108.5 (4)	N2—C9—H9	127.0
C1—S1—C7	101.4 (6)	C10—C9—N2	106.0 (10)
C7—S1—Pt1	109.2 (4)	C10—C9—H9	127.0
C10—N1—C12	123.6 (10)	N1—C10—H10	126.1
C11—N1—C10	110.1 (10)	C9—C10—N1	107.7 (10)
C11—N1—C12	125.9 (9)	C9—C10—H10	126.1
C9—N2—C8	126.2 (9)	N1—C11—Pt1	131.4 (8)
C11—N2—C8	123.2 (9)	N1—C11—N2	105.7 (9)
C11—N2—C9	110.5 (10)	N2—C11—Pt1	122.8 (8)
C2—C1—S1	122.8 (9)	N1—C12—H12A	109.5
C6—C1—S1	116.6 (10)	N1—C12—H12B	109.5
C6—C1—C2	120.7 (12)	N1—C12—C13	110.8 (10)
C1—C2—H2	120.5	H12A—C12—H12B	108.1
C1—C2—C3	118.9 (12)	C13—C12—H12A	109.5
C3—C2—H2	120.5	C13—C12—H12B	109.5
C2—C3—H3	119.7	C12—C13—H13A	109.0
C4—C3—C2	120.7 (12)	C12—C13—H13B	109.0
C4—C3—H3	119.7	C12—C13—C14	112.7 (10)
C3—C4—H4	120.1	H13A—C13—H13B	107.8
C3—C4—C5	119.8 (12)	C14—C13—H13A	109.0
C5—C4—H4	120.1	C14—C13—H13B	109.0
C4—C5—H5	119.9	C13—C14—H14A	109.3
C4—C5—C6	120.2 (13)	C13—C14—H14B	109.3
C6—C5—H5	119.9	H14A—C14—H14B	108.0
C1—C6—C5	119.7 (12)	C15—C14—C13	111.7 (11)
C1—C6—H6	120.2	C15—C14—H14A	109.3
C5—C6—H6	120.2	C15—C14—H14B	109.3
S1—C7—H7A	109.3	C14—C15—H15A	109.5
S1—C7—H7B	109.3	C14—C15—H15B	109.5
H7A—C7—H7B	107.9	C14—C15—H15C	109.5
C8—C7—S1	111.8 (8)	H15A—C15—H15B	109.5
C8—C7—H7A	109.3	H15A—C15—H15C	109.5
C8—C7—H7B	109.3	H15B—C15—H15C	109.5

Pt1—S1—C1—C2	62.3 (11)	C8—N2—C9—C10	175.0 (11)
Pt1—S1—C1—C6	−118.3 (9)	C8—N2—C11—Pt1	3.2 (16)
Pt1—S1—C7—C8	14.3 (10)	C8—N2—C11—N1	−174.1 (10)

S1—C1—C2—C3	−177.4 (10)	C9—N2—C8—C7	−108.8 (12)
S1—C1—C6—C5	178.0 (10)	C9—N2—C11—Pt1	178.8 (8)
S1—C7—C8—N2	−67.4 (11)	C9—N2—C11—N1	1.5 (14)
N1—C12—C13—C14	−171.7 (10)	C10—N1—C11—Pt1	−179.0 (10)
N2—C9—C10—N1	−0.9 (13)	C10—N1—C11—N2	−2.0 (14)
C1—S1—C7—C8	128.8 (9)	C10—N1—C12—C13	86.3 (13)
C1—C2—C3—C4	−2 (2)	C11—N1—C10—C9	1.8 (14)
C2—C1—C6—C5	−2.5 (19)	C11—N1—C12—C13	−85.0 (14)
C2—C3—C4—C5	0 (2)	C11—N2—C8—C7	66.0 (14)
C3—C4—C5—C6	1 (2)	C11—N2—C9—C10	−0.4 (14)
C4—C5—C6—C1	0 (2)	C12—N1—C10—C9	−170.7 (11)
C6—C1—C2—C3	3.2 (19)	C12—N1—C11—Pt1	−6.7 (19)
C7—S1—C1—C2	−52.6 (11)	C12—N1—C11—N2	170.3 (10)
C7—S1—C1—C6	126.8 (10)	C12—C13—C14—C15	179.8 (10)

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the N1/C10/C9/N2/C11 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···Cl2^{i}	0.93	2.58	3.485 (12)	163
C2—H2···*Cg*1	0.93	2.98	3.828 (13)	151
C14—H14*A*···*Cg*1^{ii}	0.97	2.82	3.480 (13)	126

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