

## Redetermination of (2,2'-bipyridine- $\kappa^2N,N'$ )dichloridopalladium(II) dichloromethane solvate

Nam-Ho Kim,<sup>a</sup> In-Chul Hwang<sup>b</sup> and Kwang Ha<sup>a\*</sup>

<sup>a</sup>School of Applied Chemical Engineering, The Research Institute of Catalysis, Chonnam National University, Gwangju 500-757, Republic of Korea, and <sup>b</sup>Institute of Basic Sciences, Pohang University of Science and Technology, Pohang 790-784, Republic of Korea

Correspondence e-mail: hakwang@chonnam.ac.kr

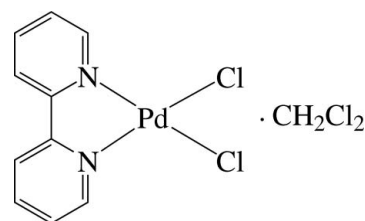
Received 23 April 2009; accepted 30 April 2009

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.010$  Å;  $R$  factor = 0.044;  $wR$  factor = 0.097; data-to-parameter ratio = 14.1.

In the title compound,  $[\text{PdCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)] \cdot \text{CH}_2\text{Cl}_2$ , the  $\text{Pd}^{2+}$  ion is four-coordinated in a slightly distorted square-planar environment by two N atoms of the 2,2'-bipyridine (bipy) ligand and two chloride ions. The compound displays intramolecular  $\text{C}-\text{H} \cdots \text{Cl}$  hydrogen bonds and pairs of complex molecules are connected by intermolecular  $\text{C}-\text{H} \cdots \text{Cl}$  hydrogen bonds. Intermolecular  $\pi-\pi$  interactions are present between the pyridine rings of the ligand, the shortest centroid-centroid distance being 4.096 (3) Å. As a result of the electronic nature of the chelate ring, it is possible to create  $\pi-\pi$  interactions to its symmetry-related counterpart [3.720 (2) Å] and also with a pyridine ring [3.570 (3) Å] of the bipy unit. The present structure is a redetermination of a previous structure [Vicente *et al.* (1997). Private communication (refcode PYCXMN02). CCDC, Cambridge, England]. In the new structure refinement all H atoms were located in a difference Fourier synthesis. Their coordinates were refined freely, together with isotropic displacement parameters.

### Related literature

For crystal structures of  $[\text{PdX}_2(\text{bipy})]$  ( $X = \text{Cl}$  or  $\text{Br}$ ), see: Maekawa *et al.* (1991); Vicente *et al.* (1997); Smeets *et al.* (1997). For related Pt(II, IV)-bipyridine complexes, see: Osborn & Rogers (1974); Hambley (1986); Sartori *et al.* (2005); Momeni *et al.* (2007); Kim *et al.* (2009).



### Experimental

#### Crystal data

|   |                                   |
|---|-----------------------------------|
| $[\text{PdCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)] \cdot \text{CH}_2\text{Cl}_2$ | $\gamma = 81.429$ (2)°            |
| $M_r = 418.41$  | $V = 715.58$ (15) Å <sup>3</sup>  |
| Triclinic, $P\bar{1}$   | $Z = 2$                           |
| $a = 8.7913$ (10) Å   | Mo $K\alpha$ radiation            |
| $b = 9.1115$ (11) Å   | $\mu = 2.03$ mm <sup>-1</sup>     |
| $c = 10.1846$ (12) Å  | $T = 293$ K                       |
| $\alpha = 72.481$ (2)°  | $0.20 \times 0.08 \times 0.08$ mm |
| $\beta = 66.983$ (2)°   |                                   |

#### Data collection

|  |  |
|--|--|
| Bruker SMART 1000 CCD diffractometer                     | 4221 measured reflections              |
| Absorption correction: multi-scan (SADABS; Bruker, 2000) | 2862 independent reflections           |
| $T_{\min} = 0.623$ , $T_{\max} = 0.850$                  | 2298 reflections with $I > 2\sigma(I)$ |
|  | $R_{\text{int}} = 0.023$               |

#### Refinement

|                                 |   |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.044$ | 203 parameters                                |
| $wR(F^2) = 0.097$               | All H-atom parameters refined                 |
| $S = 1.05$                      | $\Delta\rho_{\max} = 0.56$ e Å <sup>-3</sup>  |
| 2862 reflections                | $\Delta\rho_{\min} = -0.69$ e Å <sup>-3</sup> |

**Table 1**

Selected bond lengths (Å).

|        |           |         |             |
|--------|-----------|---------|-------------|
| Pd1—N2 | 2.025 (4) | Pd1—Cl2 | 2.2853 (14) |
| Pd1—N1 | 2.029 (4) | Pd1—Cl1 | 2.2964 (14) |

**Table 2**

Hydrogen-bond geometry (Å, °).

| $D-\text{H} \cdots A$           | $D-\text{H}$ | $\text{H} \cdots A$ | $D \cdots A$ | $D-\text{H} \cdots A$ |
|---------------------------------|--------------|---------------------|--------------|-----------------------|
| C1—H1 $\cdots$ Cl2              | 0.93 (5)     | 2.59 (5)            | 3.230 (6)    | 126 (4)               |
| C2—H2 $\cdots$ Cl2 <sup>i</sup> | 0.93 (5)     | 2.79 (5)            | 3.595 (7)    | 145 (4)               |
| C10—H10 $\cdots$ Cl1            | 0.86 (5)     | 2.68 (5)            | 3.248 (6)    | 125 (4)               |
| C11—H11B $\cdots$ Cl1           | 0.99 (6)     | 2.63 (6)            | 3.578 (8)    | 161 (5)               |

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: SHELXL97.

This work was supported by the Korea Research Foundation Grant funded by the Korean Government (MOEHRD) (KRF-2007-412-J02001).

---

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2220).

---

## References

- Bruker (2000). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Hambley, T. W. (1986). *Acta Cryst.* **C42**, 49–51.
- Kim, N.-H., Hwang, I.-C. & Ha, K. (2009). *Acta Cryst.* **E65**, m180.
- Maekawa, M., Munakata, M., Kitagawa, S. & Nakamura, M. (1991). *Anal. Sci.* **7**, 521–522.
- Momeni, B. Z., Hamzeh, S., Hosseini, S. S. & Rominger, F. (2007). *Inorg. Chim. Acta*, **360**, 2661–2668.
- Osborn, R. S. & Rogers, D. (1974). *J. Chem. Soc. Dalton Trans.* pp. 1002–1004.
- Sartori, D. A., Hurst, S. K., Wood, N., Larsen, R. D. & Abbott, E. H. (2005). *J. Chem. Crystallogr.* **35**, 995–998.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Smeets, W. J. J., Spek, A. L., Hoare, J. L., Canty, A. J., Hovestad, N. & van Koten, G. (1997). *Acta Cryst.* **C53**, 1045–1047.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Vicente, J., Abad, J. A., Rink, B. & Arellano, M. C. R. (1997). Private communication (refcode PYCXMN02). CCDC, Cambridge, England.

**supplementary materials**

*Acta Cryst.* (2009). E65, m615-m616 [ doi:10.1107/S1600536809016262 ]

## Redetermination of (2,2'-bipyridine- $\kappa^2N,N'$ )dichloridopalladium(II) dichloromethane solvate

N.-H. Kim, I.-C. Hwang and K. Ha

### Comment

The asymmetric unit of the title compound,  $[\text{PdCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)] \cdot \text{CH}_2\text{Cl}_2$ , contains a neutral  $\text{Pd}^{\text{II}}$  complex and a solvent molecule (Fig. 1). The compound crystallized in the triclinic space group  $P\bar{1}$ , whereas the previously reported complex  $[\text{PdCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)]$  crystallized in the orthorhombic space group  $C222_1$  (Maekawa *et al.*, 1991). The X-ray structure analysis of the title compound was previously carried out (Vicente *et al.*, 1997) and the structure is stored in CCDC code: RAGVUQ. The stored and our crystal structures are in agreement, however, the new structure determination provides data on hydrogen bonding based on H atoms positions located from a difference Fourier synthesis and refined isotropically.

In the complex, the  $\text{Pd}^{2+}$  ion is four-coordinated in a distorted square-planar environment by two N atoms of the 2,2'-bipyridine (bipy) ligand and two Cl ions. The main contribution to the distortion is the tight  $\text{N1-Pd1-N2}$  chelate angle ( $81.04(16)^\circ$ ), which results in non-linear *trans* arrangement ( $\angle \text{N1-Pd1-Cl1} = 175.78(12)^\circ$  and  $\angle \text{N2-Pd1-Cl2} = 174.97(12)^\circ$ ). The  $\text{Pd1-N}$  and  $\text{Pd1-Cl}$  bond lengths are almost equal ( $\text{Pd1-N}$ : 2.029(4) and 2.025(4) Å;  $\text{Pd1-Cl}$  2.2964(14) and 2.2853(14) Å), respectively (Table 1), and close to those reported for  $[\text{PdCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)]$  (Maekawa *et al.*, 1991). The best least-squares plane of the atoms of the planar complex, except Cl2, reveals mean deviation of 0.026(5) Å; deviation of Cl2 from this plane is 0.184(2) Å. The compound displays the inter- and intramolecular  $\text{C-H}\cdots\text{Cl}$  hydrogen bonds (Table 2) and molecules are connected by intermolecular hydrogen bonds and stacked in layers along the *c* axis (Fig. 2). Intermolecular  $\pi$ - $\pi$  interactions (defined by separation distance between the ring centroids (the symmetry operation for second plane 1-x, 1-y, -z) involve the planes:  $\text{Pd1, N1} \rightarrow \text{N2}$  and its symmetry related counterpart, 3.720(2) Å, planes are parallel and shifted for 1.506 Å;  $\text{Pd1, N1} \rightarrow \text{N2}$  and  $\text{N1} \rightarrow \text{C5}$ , 3.570(3) Å, the dihedral angle between planes is  $1.54^\circ$ , no shift;  $\text{N1} \rightarrow \text{C5}$  and  $\text{N2} \rightarrow \text{C10}$ , 4.096(3) Å the dihedral angle between planes is  $2.72^\circ$ , no shift.

### Experimental

To a solution of  $\text{Na}_2\text{PdCl}_4$  (0.300 g, 1.020 mmol) in EtOH (30 ml) was added 2,2'-bipyridine (0.159 g, 1.018 mmol) and stirred for 5 h at room temperature. The precipitate obtained was separated by filtration and washed with EtOH and water and dried under vacuum, to give a yellow powder (0.302 g). Crystals suitable for X-ray analysis were obtained by slow evaporation from a  $\text{CH}_2\text{Cl}_2$  solution.

### Refinement

All H atoms were located from Fourier difference maps and refined isotropically.

## Figures

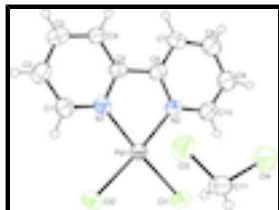


Fig. 1. The structure of the title compound, with displacement ellipsoids drawn at the 40% probability level for non-H atoms.

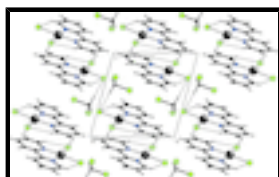


Fig. 2. Crystal packing of I. Hydrogen-bond interactions are drawn with dashed lines.

## (2,2'-Bipyridine- $\kappa^2N,N'$ )dichloridopalladium(II) dichloromethane solvate

### Crystal data

[PdCl<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)]·CH<sub>2</sub>Cl<sub>2</sub>

$M_r = 418.41$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 8.7913$  (10) Å

$b = 9.1115$  (11) Å

$c = 10.1846$  (12) Å

$\alpha = 72.481$  (2)°

$\beta = 66.983$  (2)°

$\gamma = 81.429$  (2)°

$V = 715.58$  (15) Å<sup>3</sup>

$Z = 2$

$F_{000} = 408$

$D_x = 1.942$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 983 reflections

$\theta = 2.3$ – $21.7$ °

$\mu = 2.03$  mm<sup>-1</sup>

$T = 293$  K

Stick, yellow

$0.20 \times 0.08 \times 0.08$  mm

### Data collection

Bruker SMART 1000 CCD  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$  K

$\varphi$  and  $\omega$  scans

Absorption correction: Multi-scan  
(SADABS; Bruker, 2000)

$T_{\min} = 0.623$ ,  $T_{\max} = 0.850$

4221 measured reflections

2862 independent reflections

2298 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 26.4$ °

$\theta_{\min} = 2.3$ °

$h = -10 \rightarrow 10$

$k = -7 \rightarrow 11$

$l = -12 \rightarrow 12$

### Refinement

Refinement on  $F^2$

Secondary atom site location: difference Fourier map

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.044$$

$$wR(F^2) = 0.097$$

$$S = 1.05$$

2862 reflections

203 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.0352P)^2 + 0.5604P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.56 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.69 \text{ e } \text{\AA}^{-3}$$

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

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Pd1 | 0.32112 (5)  | 0.28572 (4)  | 0.21130 (4)  | 0.04479 (15)                     |
| Cl1 | 0.36850 (18) | 0.02764 (15) | 0.30403 (16) | 0.0612 (4)                       |
| Cl2 | 0.1458 (2)   | 0.22805 (17) | 0.11857 (19) | 0.0720 (5)                       |
| N1  | 0.2916 (5)   | 0.5169 (5)   | 0.1348 (4)   | 0.0454 (10)                      |
| N2  | 0.4786 (5)   | 0.3551 (5)   | 0.2803 (4)   | 0.0446 (10)                      |
| C1  | 0.1884 (7)   | 0.5895 (7)   | 0.0665 (6)   | 0.0545 (14)                      |
| H1  | 0.126 (6)    | 0.526 (6)    | 0.052 (5)    | 0.058 (16)*                      |
| C2  | 0.1803 (8)   | 0.7475 (7)   | 0.0175 (7)   | 0.0610 (15)                      |
| H2  | 0.101 (7)    | 0.800 (6)    | -0.020 (6)   | 0.063 (17)*                      |
| C3  | 0.2768 (8)   | 0.8325 (7)   | 0.0413 (7)   | 0.0620 (16)                      |
| H3  | 0.273 (6)    | 0.930 (6)    | 0.018 (5)    | 0.043 (14)*                      |
| C4  | 0.3831 (7)   | 0.7585 (6)   | 0.1120 (6)   | 0.0515 (13)                      |
| H4  | 0.451 (6)    | 0.809 (6)    | 0.128 (5)    | 0.046 (14)*                      |
| C5  | 0.3887 (6)   | 0.6010 (6)   | 0.1590 (5)   | 0.0417 (11)                      |
| C6  | 0.4959 (6)   | 0.5099 (6)   | 0.2384 (5)   | 0.0466 (12)                      |
| C7  | 0.6069 (8)   | 0.5739 (7)   | 0.2675 (7)   | 0.0644 (16)                      |
| H7  | 0.623 (7)    | 0.683 (7)    | 0.236 (6)    | 0.081 (19)*                      |
| C8  | 0.7003 (9)   | 0.4784 (8)   | 0.3427 (8)   | 0.077 (2)                        |
| H8  | 0.775 (8)    | 0.512 (8)    | 0.355 (7)    | 0.09 (2)*                        |
| C9  | 0.6833 (8)   | 0.3223 (8)   | 0.3858 (8)   | 0.0717 (18)                      |
| H9  | 0.734 (6)    | 0.255 (6)    | 0.442 (5)    | 0.049 (15)*                      |
| C10 | 0.5692 (7)   | 0.2645 (7)   | 0.3546 (7)   | 0.0579 (15)                      |

## supplementary materials

---

|      |             |              |              |             |
|------|-------------|--------------|--------------|-------------|
| H10  | 0.562 (6)   | 0.166 (6)    | 0.376 (5)    | 0.043 (14)* |
| C11  | 0.0507 (9)  | 0.1175 (7)   | 0.6220 (8)   | 0.0614 (16) |
| H11A | -0.043 (7)  | 0.071 (6)    | 0.685 (6)    | 0.062 (17)* |
| H11B | 0.115 (7)   | 0.087 (7)    | 0.529 (7)    | 0.09 (2)*   |
| Cl3  | -0.0049 (2) | 0.31218 (19) | 0.55636 (18) | 0.0740 (5)  |
| Cl4  | 0.1895 (2)  | 0.1016 (2)   | 0.7099 (2)   | 0.0887 (6)  |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$      | $U^{13}$      | $U^{23}$      |
|-----|-------------|-------------|-------------|---------------|---------------|---------------|
| Pd1 | 0.0465 (2)  | 0.0387 (2)  | 0.0512 (3)  | -0.00670 (17) | -0.01825 (18) | -0.01196 (18) |
| Cl1 | 0.0744 (9)  | 0.0386 (7)  | 0.0735 (9)  | -0.0059 (7)   | -0.0309 (8)   | -0.0121 (7)   |
| Cl2 | 0.0836 (11) | 0.0561 (9)  | 0.0965 (12) | -0.0203 (8)   | -0.0543 (9)   | -0.0117 (8)   |
| N1  | 0.045 (2)   | 0.044 (2)   | 0.048 (2)   | -0.005 (2)    | -0.017 (2)    | -0.0118 (19)  |
| N2  | 0.049 (2)   | 0.039 (2)   | 0.050 (2)   | -0.0036 (19)  | -0.021 (2)    | -0.0121 (19)  |
| C1  | 0.052 (3)   | 0.054 (3)   | 0.062 (3)   | -0.002 (3)    | -0.028 (3)    | -0.012 (3)    |
| C2  | 0.063 (4)   | 0.050 (4)   | 0.068 (4)   | 0.008 (3)     | -0.029 (3)    | -0.009 (3)    |
| C3  | 0.072 (4)   | 0.039 (3)   | 0.074 (4)   | -0.002 (3)    | -0.025 (3)    | -0.016 (3)    |
| C4  | 0.057 (3)   | 0.039 (3)   | 0.063 (3)   | -0.005 (3)    | -0.025 (3)    | -0.016 (3)    |
| C5  | 0.041 (3)   | 0.039 (3)   | 0.044 (3)   | -0.004 (2)    | -0.010 (2)    | -0.016 (2)    |
| C6  | 0.046 (3)   | 0.044 (3)   | 0.045 (3)   | -0.004 (2)    | -0.012 (2)    | -0.011 (2)    |
| C7  | 0.080 (4)   | 0.052 (4)   | 0.078 (4)   | -0.015 (3)    | -0.045 (4)    | -0.013 (3)    |
| C8  | 0.084 (5)   | 0.070 (5)   | 0.097 (5)   | -0.016 (4)    | -0.058 (4)    | -0.009 (4)    |
| C9  | 0.074 (4)   | 0.063 (4)   | 0.092 (5)   | -0.004 (3)    | -0.054 (4)    | -0.009 (4)    |
| C10 | 0.065 (4)   | 0.045 (3)   | 0.067 (4)   | -0.002 (3)    | -0.032 (3)    | -0.008 (3)    |
| C11 | 0.065 (4)   | 0.052 (4)   | 0.072 (4)   | -0.009 (3)    | -0.026 (3)    | -0.019 (3)    |
| Cl3 | 0.0761 (10) | 0.0652 (10) | 0.0759 (10) | 0.0022 (8)    | -0.0354 (9)   | -0.0044 (8)   |
| Cl4 | 0.0906 (12) | 0.0701 (11) | 0.1211 (15) | 0.0040 (9)    | -0.0663 (12)  | -0.0139 (10)  |

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

|            |             |          |           |
|------------|-------------|----------|-----------|
| Pd1—N2     | 2.025 (4)   | C4—H4    | 0.90 (5)  |
| Pd1—N1     | 2.029 (4)   | C5—C6    | 1.480 (7) |
| Pd1—Cl2    | 2.2853 (14) | C6—C7    | 1.371 (7) |
| Pd1—Cl1    | 2.2964 (14) | C7—C8    | 1.377 (9) |
| N1—C1      | 1.335 (6)   | C7—H7    | 0.96 (6)  |
| N1—C5      | 1.360 (6)   | C8—C9    | 1.369 (9) |
| N2—C10     | 1.338 (7)   | C8—H8    | 0.83 (6)  |
| N2—C6      | 1.359 (6)   | C9—C10   | 1.375 (8) |
| C1—C2      | 1.375 (8)   | C9—H9    | 0.90 (5)  |
| C1—H1      | 0.93 (5)    | C10—H10  | 0.86 (5)  |
| C2—C3      | 1.360 (8)   | C11—Cl4  | 1.743 (6) |
| C2—H2      | 0.93 (5)    | C11—Cl3  | 1.765 (6) |
| C3—C4      | 1.376 (8)   | C11—H11A | 0.90 (5)  |
| C3—H3      | 0.85 (5)    | C11—H11B | 0.99 (6)  |
| C4—C5      | 1.369 (7)   |          |           |
| N2—Pd1—N1  | 81.04 (16)  | N1—C5—C4 | 120.6 (5) |
| N2—Pd1—Cl2 | 174.97 (12) | N1—C5—C6 | 115.2 (4) |

|                |             |               |            |
|----------------|-------------|---------------|------------|
| N1—Pd1—C12     | 94.27 (12)  | C4—C5—C6      | 124.2 (5)  |
| N2—Pd1—C11     | 94.74 (12)  | N2—C6—C7      | 121.4 (5)  |
| N1—Pd1—C11     | 175.78 (12) | N2—C6—C5      | 115.0 (4)  |
| C12—Pd1—C11    | 89.94 (5)   | C7—C6—C5      | 123.6 (5)  |
| C1—N1—C5       | 119.3 (5)   | C6—C7—C8      | 118.8 (6)  |
| C1—N1—Pd1      | 126.5 (4)   | C6—C7—H7      | 122 (4)    |
| C5—N1—Pd1      | 114.2 (3)   | C8—C7—H7      | 119 (4)    |
| C10—N2—C6      | 118.9 (4)   | C9—C8—C7      | 120.2 (6)  |
| C10—N2—Pd1     | 126.6 (4)   | C9—C8—H8      | 118 (5)    |
| C6—N2—Pd1      | 114.4 (3)   | C7—C8—H8      | 121 (5)    |
| N1—C1—C2       | 121.4 (6)   | C8—C9—C10     | 118.5 (6)  |
| N1—C1—H1       | 116 (3)     | C8—C9—H9      | 124 (3)    |
| C2—C1—H1       | 123 (3)     | C10—C9—H9     | 117 (3)    |
| C3—C2—C1       | 119.7 (6)   | N2—C10—C9     | 122.2 (6)  |
| C3—C2—H2       | 118 (4)     | N2—C10—H10    | 118 (3)    |
| C1—C2—H2       | 122 (4)     | C9—C10—H10    | 119 (3)    |
| C2—C3—C4       | 119.2 (6)   | C14—C11—C13   | 111.0 (3)  |
| C2—C3—H3       | 124 (3)     | C14—C11—H11A  | 111 (4)    |
| C4—C3—H3       | 117 (3)     | C13—C11—H11A  | 106 (4)    |
| C5—C4—C3       | 119.8 (5)   | C14—C11—H11B  | 106 (3)    |
| C5—C4—H4       | 118 (3)     | C13—C11—H11B  | 103 (4)    |
| C3—C4—H4       | 123 (3)     | H11A—C11—H11B | 120 (5)    |
| N2—Pd1—N1—C1   | -177.3 (4)  | C3—C4—C5—N1   | 0.8 (8)    |
| C12—Pd1—N1—C1  | 4.6 (4)     | C3—C4—C5—C6   | -178.8 (5) |
| N2—Pd1—N1—C5   | 2.4 (3)     | C10—N2—C6—C7  | 1.7 (8)    |
| C12—Pd1—N1—C5  | -175.7 (3)  | Pd1—N2—C6—C7  | -175.3 (4) |
| N1—Pd1—N2—C10  | 179.8 (5)   | C10—N2—C6—C5  | -179.1 (4) |
| C11—Pd1—N2—C10 | -0.3 (5)    | Pd1—N2—C6—C5  | 4.0 (5)    |
| N1—Pd1—N2—C6   | -3.5 (3)    | N1—C5—C6—N2   | -2.0 (6)   |
| C11—Pd1—N2—C6  | 176.4 (3)   | C4—C5—C6—N2   | 177.6 (5)  |
| C5—N1—C1—C2    | 1.2 (8)     | N1—C5—C6—C7   | 177.3 (5)  |
| Pd1—N1—C1—C2   | -179.2 (4)  | C4—C5—C6—C7   | -3.1 (8)   |
| N1—C1—C2—C3    | -1.3 (9)    | N2—C6—C7—C8   | -1.0 (9)   |
| C1—C2—C3—C4    | 1.1 (9)     | C5—C6—C7—C8   | 179.8 (6)  |
| C2—C3—C4—C5    | -0.8 (9)    | C6—C7—C8—C9   | 0.7 (11)   |
| C1—N1—C5—C4    | -0.9 (7)    | C7—C8—C9—C10  | -1.2 (11)  |
| Pd1—N1—C5—C4   | 179.4 (4)   | C6—N2—C10—C9  | -2.2 (9)   |
| C1—N1—C5—C6    | 178.7 (4)   | Pd1—N2—C10—C9 | 174.4 (5)  |
| Pd1—N1—C5—C6   | -1.0 (5)    | C8—C9—C10—N2  | 1.9 (10)   |

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> |
|--------------------------|-------------|---------------|-----------------------|-------------------------|
| C1—H1...C12              | 0.93 (5)    | 2.59 (5)      | 3.230 (6)             | 126 (4)                 |
| C2—H2...C12 <sup>i</sup> | 0.93 (5)    | 2.79 (5)      | 3.595 (7)             | 145 (4)                 |
| C10—H10...C11            | 0.86 (5)    | 2.68 (5)      | 3.248 (6)             | 125 (4)                 |
| C11—H11B...C11           | 0.99 (6)    | 2.63 (6)      | 3.578 (8)             | 161 (5)                 |

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



Fig. 1

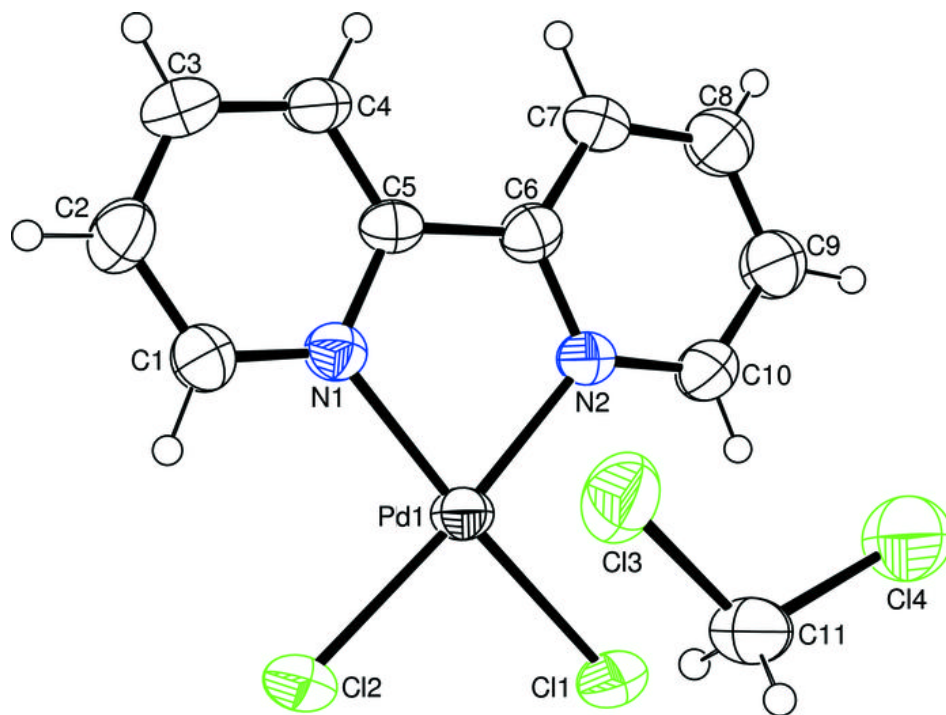


Fig. 2

