

cis-Dichlorido[4,4,5,5-tetramethyl-2-(2-pyridyl)-2-imidazoline-1-oxyl]-palladium(II) tetrahydrofuran hemisolvate

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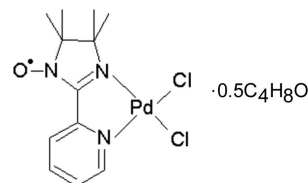
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}–\text{C}) = 0.013$ Å; R factor = 0.057; wR factor = 0.153; data-to-parameter ratio = 15.4.

The asymmetric unit of the title complex, $[\text{PdCl}_2(\text{C}_{12}\text{H}_{16}\text{N}_3\text{O})] \cdot 0.5\text{C}_4\text{H}_8\text{O}$, consists of one palladium complex in a general position and one half tetrahydrofuran (THF) solvent molecule, with the O atom lying on a twofold rotation axis. The Pd^{II} atom is bound to one chelating imino nitroxide radical through two N atoms, one from the pyridyl ring and the other from the imidazoline ring. The coordination of the metal centre is completed by two Cl atoms in a *cis* configuration, leading to a quasi-square-planar coordination of the metal centre. The four atoms that define the Pd^{II} coordination environment and the eight atoms that belong to the pyridylimine fragment are coplanar, with no deviation larger than 0.087 (5) Å. In the crystal structure, intermolecular interactions shorter than the corresponding van der Waals radii sum are observed only between Pd^{II} complexes, and no short contact is observed around the THF molecule. Weak $\text{C}–\text{H} \cdots \text{O}$ and $\text{C}–\text{H} \cdots \text{Cl}$ interactions yield a two-dimensional network of complexes in the (101) plane.

Related literature

For related literature, see: Caneschi *et al.* (1991); Davis *et al.* (1972); Evans *et al.* (1968); Fettouhi *et al.* (2003); Li *et al.* (2004); Ma *et al.* (2006, 2007); Oshio *et al.* (1996); Ueda *et al.* (2003, 2005); Ullman & Holm (1970); Xu *et al.* (2007).



Experimental

Crystal data

$[\text{PdCl}_2(\text{C}_{12}\text{H}_{16}\text{N}_3\text{O})] \cdot 0.5\text{C}_4\text{H}_8\text{O}$	$V = 3359.6$ (4) Å ³
$M_r = 431.65$	$Z = 8$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 19.1398$ (10) Å	$\mu = 1.43$ mm ⁻¹
$b = 15.2061$ (12) Å	$T = 293$ (2) K
$c = 13.8291$ (10) Å	$0.7 \times 0.3 \times 0.3$ mm
$\beta = 123.415$ (3)°	

Data collection

Nonius KappaCCD diffractometer	3065 independent reflections
Absorption correction: none	2049 reflections with $I > 2\sigma(I)$
5707 measured reflections	$R_{\text{int}} = 0.059$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$	199 parameters
$wR(F^2) = 0.152$	H-atom parameters constrained
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.61$ e Å ⁻³
3065 reflections	$\Delta\rho_{\text{min}} = -0.95$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D–H \cdots A$	$D–H$	$H \cdots A$	$D \cdots A$	$D–H \cdots A$
$\text{C4}–\text{H4} \cdots \text{O1}^{\text{i}}$	0.93	2.61	3.307 (8)	132
$\text{C10}–\text{H10B} \cdots \text{Cl1}^{\text{ii}}$	0.96	2.77	3.691 (7)	160

Symmetry codes: (i) $-x + 1, -y, -z + 1$; (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); 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: DN2318).

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***cis*-Dichlorido[4,4,5,5-tetramethyl-2-(2-pyridyl)-2-imidazoline-1-oxyl]palladium(II) tetrahydrofuran hemisolvate**

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Comment

Organic nitronyl nitroxide radicals have attracted much attention for their magnetic properties in general and ferromagnetism in particular. Then several transition metal complexes with stable nitronyl nitroxide radical ligands have been prepared and extensively investigated (Ma *et al.*, 2007; Xu *et al.*, 2007; Ma *et al.*, 2006; Ueda *et al.*, 2005; Li *et al.*, 2004; Ueda *et al.*, 2003; Oshio *et al.*, 1996; Caneschi *et al.*, 1991). Our contribution in this field is the synthesis (see Scheme 1) and structure characterization of the title compound.

The molecular structure of the complex [Pd(IM₂py)Cl₂] 0.5THF(I) is shown in Fig. 1 while selected geometric parameters are given in Table 1. Focusing the coordination square of Pd^{II}, one can notice that Cl2 atom deviates significantly from the plane defined with atoms N3/N1/Pd1/Cl1 by 0.0843 (23) Å. The mean bond distance of Pd—Cl 2.277 Å is in agreement with the values observed in a similar complex while the average bond length of Pd—N 2.045 Å is slightly longer than seen previously (Fettouhi *et al.*, 2003). Due to a chelation of the iminonitroxide radical with Pd^{II} ion, the four atoms which define the Pd^{II} coordination plane and the eight atoms which belong to both pyridyl ring and imino fragment are coplanar, the larger deviation to the plane is equal to 0.087 (5) and -0.064 (5) Å for O1 and N3 respectively. Only *sp*³ carbon C7 and C8 from imino and methyl carbon C9, C10, C11 and C12 deviate significantly from the mean plane.

In the packing, one can notice that intermolecular interactions shorter than the corresponding van-der-Waals radii are only observed between Pd^{II} complexes, no short contact are observed around THF molecule. Centrosymmetric contacts take place between imino and pyridyl ring (O1—H4) of one neighbouring complex as well as contacts between chlorine Cl1 and H10B atom of methylene group (Table 1). These contacts yield a two-dimensional network of interacting complexes along the (101) plane (Fig. 2). Another short contact is observed between two adjacent 2-D networks thanks to a van-der-Waals interaction between two C1 atoms of pyridyl rings of two neighbouring molecules with C1—C1 3.393 (16) Å. The shortest distance between two palladium take place between ions from two adjacent planes (Pd1—Pd1 = 3.648 (2) Å), onto a plane, the shorter Pd1—Pd1 distance is 8.7834 (7) Å. THF solvent molecules are lying between two planes. A contact O1s—H1 of 2.7445 Å is observed with pyridyl ring.

Experimental

Dichlorobis benzonitrile palladium(II) PdCl₂(PhCN)₂ and 2-(*ortho*-pyridyl)-4,4,5,5-tetramethyl imidazoline-1-oxyl-3-oxyl (NIT2Py) were synthesized according to literature method (Evans *et al.*, 1968; Ullman & Holm, 1970; Davis *et al.*, 1972). The complex Pd(IM₂py)Cl₂ was synthesized as follows: The reaction was performed under a dry nitrogen atmosphere using standard schlenk technique. All solvents used were distilled under nitrogen. To a solution of PdCl₂(PhCN)₂ (0.1 g;0.26 mmol) in 30 ml of toluene was added with stirring a solution of the radical NIT2Py (0.12 g;0.52 mmol) in 20 ml of toluene. After 2 h of stirring at room temperature, the mixture was filtered and the solvent removed under reduced

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pressure. Parallelepipedic brown crystals of complex (I) suitable for *x*-ray crystallographic analysis were obtained by slow diffusion of hexane in THF solution of complex (I).

Refinement

All H atoms were placed in calculated positions and treated as riding model with C—H ranging from 0.93 Å [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$] for pyridyl ring to 0.96—0.97 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ — $1.2U_{\text{eq}}(\text{C})$ for methyl and methylene respectively.

Figures

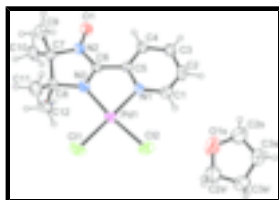


Fig. 1. An *ORTEP* drawing of title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme. The labels of the H atoms have been omitted for clarity. [Symmetry codes: (i) $-x + 1, y, -z + 1.5$]

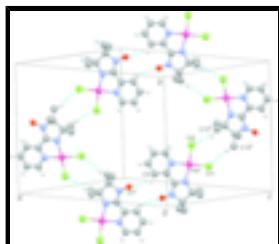


Fig. 2. Partial packing view showing the formation of a two-dimensional network through C—H...O and C—H...Cl intermolecular interactions. H bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $-x -+1, -y, -z + 1$; (ii) $1/2 - x, y + 1/2, 3/2 - z$]



Fig. 3. The formation of the title compound.

cis-Dichlorido[4,4,5,5-tetramethyl-2-(2-pyridyl)-2-imidazoline-1-oxyl]palladium(II) tetrahydrofuran hemisolvate

Crystal data

$[\text{PdCl}_2(\text{C}_{12}\text{H}_{16}\text{N}_3\text{O})] \cdot 0.5\text{C}_4\text{H}_8\text{O}$

$M_r = 431.65$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 19.1398 (10) \text{ \AA}$

$b = 15.2061 (12) \text{ \AA}$

$c = 13.8291 (10) \text{ \AA}$

$\beta = 123.415 (3)^\circ$

$V = 3359.6 (4) \text{ \AA}^3$

$Z = 8$

$F_{000} = 1736$

$D_x = 1.707 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

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

Cell parameters from 2981 reflections

$\theta = 2.6$ – 25.4°

$\mu = 1.43 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Thick plate, brown

$0.7 \times 0.3 \times 0.3 \text{ mm}$

Data collection

Nonius KappaCCD

2049 reflections with $I > 2\sigma(I)$

diffractometer
 Radiation source: fine-focus sealed tube $R_{\text{int}} = 0.059$
 Monochromator: graphite $\theta_{\text{max}} = 25.4^\circ$
 $T = 293(2)$ K $\theta_{\text{min}} = 3.0^\circ$
 φ and ω scans $h = -22 \rightarrow 22$
 Absorption correction: none $k = -17 \rightarrow 18$
 5707 measured reflections $l = -16 \rightarrow 16$
 3065 independent reflections

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.057$ H-atom parameters constrained
 $wR(F^2) = 0.152$ $w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 4.5052P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.03$ $(\Delta/\sigma)_{\text{max}} = 0.001$
 3065 reflections $\Delta\rho_{\text{max}} = 0.61 \text{ e } \text{\AA}^{-3}$
 199 parameters $\Delta\rho_{\text{min}} = -0.95 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

Special details

Experimental. Multiscan absorption correction methods did not yield a better refinement agreement, then no correction was applied.

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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4332 (5)	0.2973 (4)	0.6038 (6)	0.0604 (18)
H1	0.4196	0.3511	0.6214	0.073*
C2	0.4905 (5)	0.2955 (5)	0.5741 (8)	0.072 (2)
H2	0.5154	0.3475	0.5723	0.087*
C3	0.5113 (5)	0.2173 (5)	0.5470 (6)	0.0603 (18)
H3	0.5497	0.2151	0.5258	0.072*
C4	0.4733 (5)	0.1419 (4)	0.5520 (6)	0.0558 (18)
H4	0.4851	0.0877	0.5330	0.067*
C5	0.4172 (4)	0.1475 (4)	0.5858 (5)	0.0443 (14)

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C6	0.3741 (4)	0.0747 (4)	0.5988 (5)	0.0445 (14)
C7	0.3264 (5)	-0.0669 (4)	0.6000 (6)	0.0560 (17)
C8	0.2934 (4)	0.0028 (4)	0.6501 (6)	0.0498 (16)
C9	0.2604 (6)	-0.0980 (6)	0.4783 (7)	0.081 (2)
H9A	0.2328	-0.0479	0.4294	0.122*
H9B	0.2201	-0.1339	0.4806	0.122*
H9C	0.2867	-0.1317	0.4480	0.122*
C10	0.3725 (5)	-0.1451 (5)	0.6789 (8)	0.078 (2)
H10A	0.3899	-0.1843	0.6415	0.117*
H10B	0.3359	-0.1757	0.6946	0.117*
H10C	0.4208	-0.1246	0.7503	0.117*
C11	0.1983 (5)	0.0032 (5)	0.5897 (7)	0.076 (2)
H11A	0.1824	0.0501	0.6202	0.114*
H11B	0.1805	-0.0520	0.6030	0.114*
H11C	0.1723	0.0116	0.5080	0.114*
C12	0.3364 (5)	-0.0032 (5)	0.7811 (6)	0.067 (2)
H12A	0.3951	-0.0143	0.8167	0.101*
H12B	0.3121	-0.0502	0.7994	0.101*
H12C	0.3292	0.0513	0.8098	0.101*
N1	0.3961 (3)	0.2250 (3)	0.6084 (5)	0.0470 (12)
N2	0.3857 (4)	-0.0129 (3)	0.5888 (5)	0.0512 (13)
N3	0.3211 (3)	0.0884 (3)	0.6270 (5)	0.0509 (13)
O1	0.4363 (3)	-0.0447 (3)	0.5657 (5)	0.0687 (14)
Cl1	0.21890 (16)	0.20499 (14)	0.7093 (2)	0.0865 (7)
Cl2	0.30744 (14)	0.36626 (13)	0.67356 (19)	0.0761 (6)
Pd1	0.30983 (3)	0.21827 (3)	0.65241 (5)	0.0534 (2)
O1S	0.5000	0.4980 (7)	0.7500	0.177 (7)
C2S	0.5251 (8)	0.5487 (7)	0.6926 (10)	0.108 (3)
H2S1	0.4949	0.5319	0.6116	0.130*
H2S2	0.5845	0.5408	0.7263	0.130*
C3S	0.5078 (9)	0.6386 (6)	0.7032 (11)	0.118 (4)
H3S1	0.4590	0.6595	0.6312	0.142*
H3S2	0.5552	0.6758	0.7235	0.142*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.069 (5)	0.050 (4)	0.066 (5)	0.003 (3)	0.040 (4)	-0.004 (3)
C2	0.077 (6)	0.059 (5)	0.091 (6)	-0.023 (4)	0.052 (5)	-0.002 (4)
C3	0.060 (4)	0.068 (5)	0.065 (5)	-0.006 (4)	0.041 (4)	0.000 (4)
C4	0.071 (5)	0.053 (4)	0.064 (5)	0.003 (3)	0.050 (4)	0.003 (3)
C5	0.050 (4)	0.045 (3)	0.039 (3)	-0.002 (3)	0.026 (3)	-0.002 (3)
C6	0.050 (4)	0.046 (3)	0.048 (4)	0.004 (3)	0.034 (3)	0.003 (3)
C7	0.068 (5)	0.046 (3)	0.073 (5)	-0.006 (3)	0.050 (4)	0.001 (3)
C8	0.050 (4)	0.053 (4)	0.054 (4)	0.000 (3)	0.034 (4)	0.008 (3)
C9	0.095 (7)	0.079 (5)	0.083 (6)	-0.018 (5)	0.057 (6)	-0.017 (5)
C10	0.100 (7)	0.060 (5)	0.105 (7)	0.009 (4)	0.075 (6)	0.022 (4)
C11	0.061 (5)	0.085 (6)	0.091 (6)	-0.010 (4)	0.048 (5)	0.000 (5)

C12	0.074 (5)	0.078 (5)	0.060 (5)	-0.003 (4)	0.044 (4)	0.005 (4)
N1	0.051 (3)	0.043 (3)	0.049 (3)	-0.002 (2)	0.029 (3)	0.002 (2)
N2	0.059 (3)	0.043 (3)	0.067 (4)	0.006 (3)	0.044 (3)	0.006 (2)
N3	0.056 (3)	0.052 (3)	0.057 (3)	0.006 (3)	0.039 (3)	0.005 (2)
O1	0.083 (4)	0.052 (3)	0.103 (4)	0.014 (3)	0.072 (4)	0.004 (3)
C11	0.0970 (17)	0.0960 (15)	0.1066 (18)	0.0216 (13)	0.0814 (16)	0.0034 (12)
C12	0.0873 (15)	0.0578 (10)	0.0865 (15)	0.0200 (10)	0.0499 (13)	-0.0037 (9)
Pd1	0.0586 (4)	0.0552 (3)	0.0549 (4)	0.0116 (3)	0.0366 (3)	-0.0002 (2)
O1S	0.249 (17)	0.068 (6)	0.35 (2)	0.000	0.246 (18)	0.000
C2S	0.116 (9)	0.094 (7)	0.134 (10)	-0.007 (6)	0.081 (8)	-0.014 (7)
C3S	0.178 (12)	0.073 (6)	0.142 (10)	-0.025 (7)	0.113 (10)	-0.009 (6)

Geometric parameters (Å, °)

C1—N1	1.329 (8)	C10—H10A	0.9600
C1—C2	1.367 (11)	C10—H10B	0.9600
C1—H1	0.9300	C10—H10C	0.9600
C2—C3	1.370 (10)	C11—H11A	0.9600
C2—H2	0.9300	C11—H11B	0.9600
C3—C4	1.381 (9)	C11—H11C	0.9600
C3—H3	0.9300	C12—H12A	0.9600
C4—C5	1.388 (8)	C12—H12B	0.9600
C4—H4	0.9300	C12—H12C	0.9600
C5—N1	1.338 (7)	N1—Pd1	2.052 (5)
C5—C6	1.451 (8)	N2—O1	1.269 (6)
C6—N3	1.289 (7)	N3—Pd1	2.037 (5)
C6—N2	1.370 (8)	C11—Pd1	2.280 (2)
C7—N2	1.476 (8)	C12—Pd1	2.273 (2)
C7—C9	1.520 (11)	O1S—C2S ⁱ	1.370 (10)
C7—C10	1.524 (10)	O1S—C2S	1.370 (10)
C7—C8	1.576 (9)	C2S—C3S	1.433 (13)
C8—N3	1.505 (8)	C2S—H2S1	0.9700
C8—C12	1.526 (9)	C2S—H2S2	0.9700
C8—C11	1.529 (10)	C3S—C3S ⁱ	1.479 (18)
C9—H9A	0.9600	C3S—H3S1	0.9700
C9—H9B	0.9600	C3S—H3S2	0.9700
C9—H9C	0.9600		
N1—C1—C2	122.5 (6)	H10B—C10—H10C	109.5
N1—C1—H1	118.8	C8—C11—H11A	109.5
C2—C1—H1	118.8	C8—C11—H11B	109.5
C1—C2—C3	120.1 (7)	H11A—C11—H11B	109.5
C1—C2—H2	120.0	C8—C11—H11C	109.5
C3—C2—H2	120.0	H11A—C11—H11C	109.5
C2—C3—C4	117.9 (6)	H11B—C11—H11C	109.5
C2—C3—H3	121.1	C8—C12—H12A	109.5
C4—C3—H3	121.1	C8—C12—H12B	109.5
C3—C4—C5	119.4 (6)	H12A—C12—H12B	109.5
C3—C4—H4	120.3	C8—C12—H12C	109.5

supplementary materials

C5—C4—H4	120.3	H12A—C12—H12C	109.5
N1—C5—C4	121.5 (6)	H12B—C12—H12C	109.5
N1—C5—C6	112.1 (5)	C1—N1—C5	118.6 (6)
C4—C5—C6	126.3 (6)	C1—N1—Pd1	126.5 (4)
N3—C6—N2	112.8 (5)	C5—N1—Pd1	114.9 (4)
N3—C6—C5	120.7 (5)	O1—N2—C6	125.7 (5)
N2—C6—C5	126.4 (5)	O1—N2—C7	123.5 (5)
N2—C7—C9	106.0 (6)	C6—N2—C7	110.6 (5)
N2—C7—C10	109.5 (6)	C6—N3—C8	110.5 (5)
C9—C7—C10	110.5 (7)	C6—N3—Pd1	112.6 (4)
N2—C7—C8	100.9 (5)	C8—N3—Pd1	136.0 (4)
C9—C7—C8	113.9 (6)	N3—Pd1—N1	79.58 (19)
C10—C7—C8	115.1 (6)	N3—Pd1—Cl2	173.28 (15)
N3—C8—C12	106.3 (5)	N1—Pd1—Cl2	93.72 (14)
N3—C8—C11	109.4 (5)	N3—Pd1—Cl1	98.44 (15)
C12—C8—C11	110.5 (5)	N1—Pd1—Cl1	176.70 (15)
N3—C8—C7	102.5 (4)	Cl2—Pd1—Cl1	88.28 (8)
C12—C8—C7	113.4 (6)	C2S ⁱ —O1S—C2S	111.5 (11)
C11—C8—C7	114.1 (6)	O1S—C2S—C3S	107.6 (9)
C7—C9—H9A	109.5	O1S—C2S—H2S1	110.2
C7—C9—H9B	109.5	C3S—C2S—H2S1	110.2
H9A—C9—H9B	109.5	O1S—C2S—H2S2	110.2
C7—C9—H9C	109.5	C3S—C2S—H2S2	110.2
H9A—C9—H9C	109.5	H2S1—C2S—H2S2	108.5
H9B—C9—H9C	109.5	C2S—C3S—C3S ⁱ	105.0 (6)
C7—C10—H10A	109.5	C2S—C3S—H3S1	110.7
C7—C10—H10B	109.5	C3S ⁱ —C3S—H3S1	110.7
H10A—C10—H10B	109.5	C2S—C3S—H3S2	110.7
C7—C10—H10C	109.5	C3S ⁱ —C3S—H3S2	110.7
H10A—C10—H10C	109.5	H3S1—C3S—H3S2	108.8

Symmetry codes: (i) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4 \cdots O1 ⁱⁱ	0.93	2.61	3.307 (8)	132
C10—H10B \cdots Cl1 ⁱⁱⁱ	0.96	2.77	3.691 (7)	160

Symmetry codes: (ii) $-x+1, -y, -z+1$; (iii) $-x+1/2, y-1/2, -z+3/2$.

Fig. 1

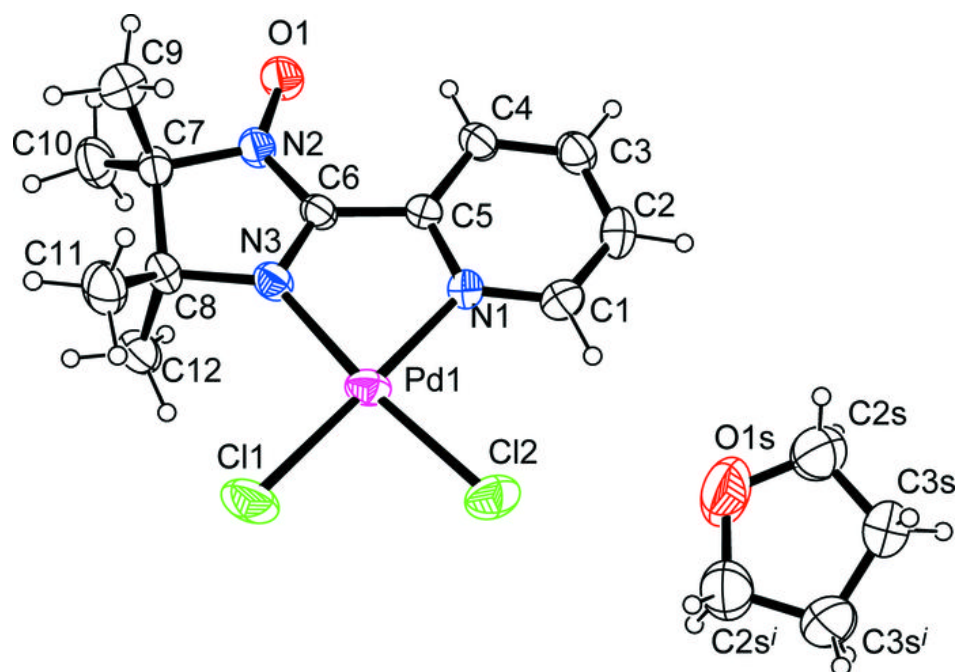


Fig. 2

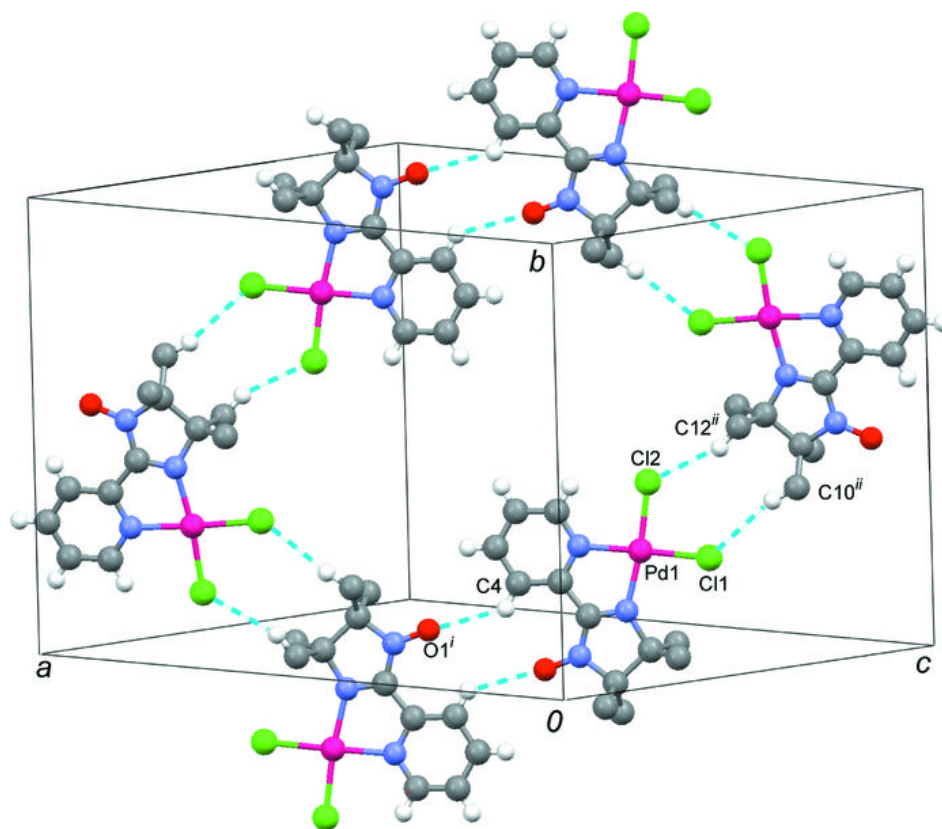


Fig. 3

