

## {5,5'-Dimethoxy-2,2'-[2,2-dimethylpropane-1,3-diylbis(nitrilomethanylylidene)]diphenolato}palladium(II)

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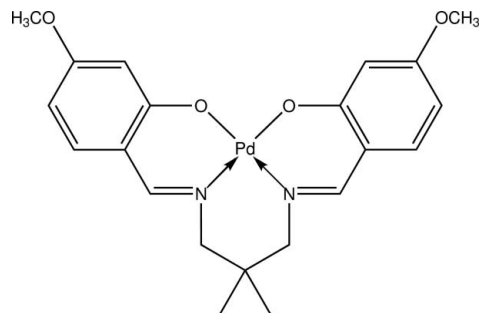
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.001$  Å;  $R$  factor = 0.016;  $wR$  factor = 0.043; data-to-parameter ratio = 32.1.

In the title compound,  $[\text{Pd}(\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_4)]$ , the complete molecule is generated by crystallographic mirror symmetry with the Pd and three C atoms lying on the mirror plane. The Pd—O and Pd—N distances are 1.9932 (6) and 2.0029 (7) Å, respectively. The dihedral angle between two benzene rings of the ligand is 79.21 (4)°. In the crystal, C—H...O hydrogen bonds link the molecules into layers parallel to the  $ab$  plane. These planes are further connected by C—H...O interactions, forming a three-dimensional network.

### Related literature

For related structures, see: Wan Nazihah Wan Ibrahim *et al.* (2008); Montazerzohori *et al.* (2009). For background to applications of palladium(II) complexes, see: Gupta *et al.* (2009); Lu *et al.* (2010); He & Cai (2011); Garoufis *et al.* (2008); Kumar *et al.* (2009); Islam *et al.* (2011). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



### Experimental

#### Crystal data

$[\text{Pd}(\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_4)]$

$M_r = 474.82$

Orthorhombic,  $Pnma$

$a = 11.5470$  (4) Å

$b = 20.9656$  (7) Å

$c = 7.8730$  (3) Å

$V = 1905.97$  (12) Å<sup>3</sup>

$Z = 4$

Mo  $K\alpha$  radiation

$\mu = 1.00$  mm<sup>-1</sup>

$T = 100$  K

$0.27 \times 0.24 \times 0.18$  mm

#### Data collection

Bruker APEX DUO CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2009)

$T_{\min} = 0.773$ ,  $T_{\max} = 0.839$

30756 measured reflections

4296 independent reflections

4134 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.016$

$wR(F^2) = 0.043$

$S = 1.13$

4296 reflections

134 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.50$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.62$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C8}-\text{H8A}\cdots\text{O1}^i$	0.99	2.37	3.3355 (10)	166
$\text{C12}-\text{H12A}\cdots\text{O2}^{ii}$	0.98	2.58	3.3719 (12)	138

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2196).

‡ Thomson Reuters ResearcherID: A-3561-2009.

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## supplementary materials

*Acta Cryst.* (2012). E68, m514–m515 [doi:10.1107/S1600536812013128]

**{5,5'-Dimethoxy-2,2'-[2,2-dimethylpropane-1,3-diylbis(nitrilomethanylylidene)]diphenolato}palladium(II)****Siti Kamilah Che Soh, Mustaffa Shamsuddin, Mohd Mustaqim Rosli and Hoong-Kun Fun****Comment**

Palladium(II)-Schiff base complexes have found extensive application in catalysis (Gupta *et al.*, 2009; Lu *et al.*, 2010; He and Cai 2011) and biological activities (Garoufis *et al.*, 2008). In particular, they are efficient and powerful phosphine-free catalysts for the formation of new C-C bonds in organic syntheses (Kumar *et al.*, 2009; Islam *et al.*, 2011). The title square-planar complex is analogous to the previously reported complex {2,2'-[(2,2 dimethylpropane-1,3-diyl)-bis(nitrilomethylidyne)]diphenolato}-palladium(II) ethanol hemisolvate (Wan Nazihah Wan Ibrahim *et al.*, 2008) in terms of geometry around the central palladium atom.

In the title compound, the complete molecule (Fig. 1) is generated by crystallographic mirror symmetry with the Pd1, C9, C10 and C11 atoms lying on the mirror plane. All parameters are within normal ranges and comparable with the related structures (Wan Nazihah Wan Ibrahim *et al.*, 2008). The Pd—O and Pd—N distances are 1.9932 (6) Å and 2.0029 (7) Å respectively. The dihedral angle between two benzene ring (C1-C6 & C1A-C6A) is 79.21 (4)°.

In the crystal packing, the molecules are linked by C8—H8A<sup>i</sup>⋯O1<sup>i</sup> intermolecular into layers parallel to the *ab*-plane. These planes are further connected by C12—H12A<sup>ii</sup>⋯O2<sup>ii</sup> to form a three-dimensional network (Table 1 & Fig. 2).

**Experimental**

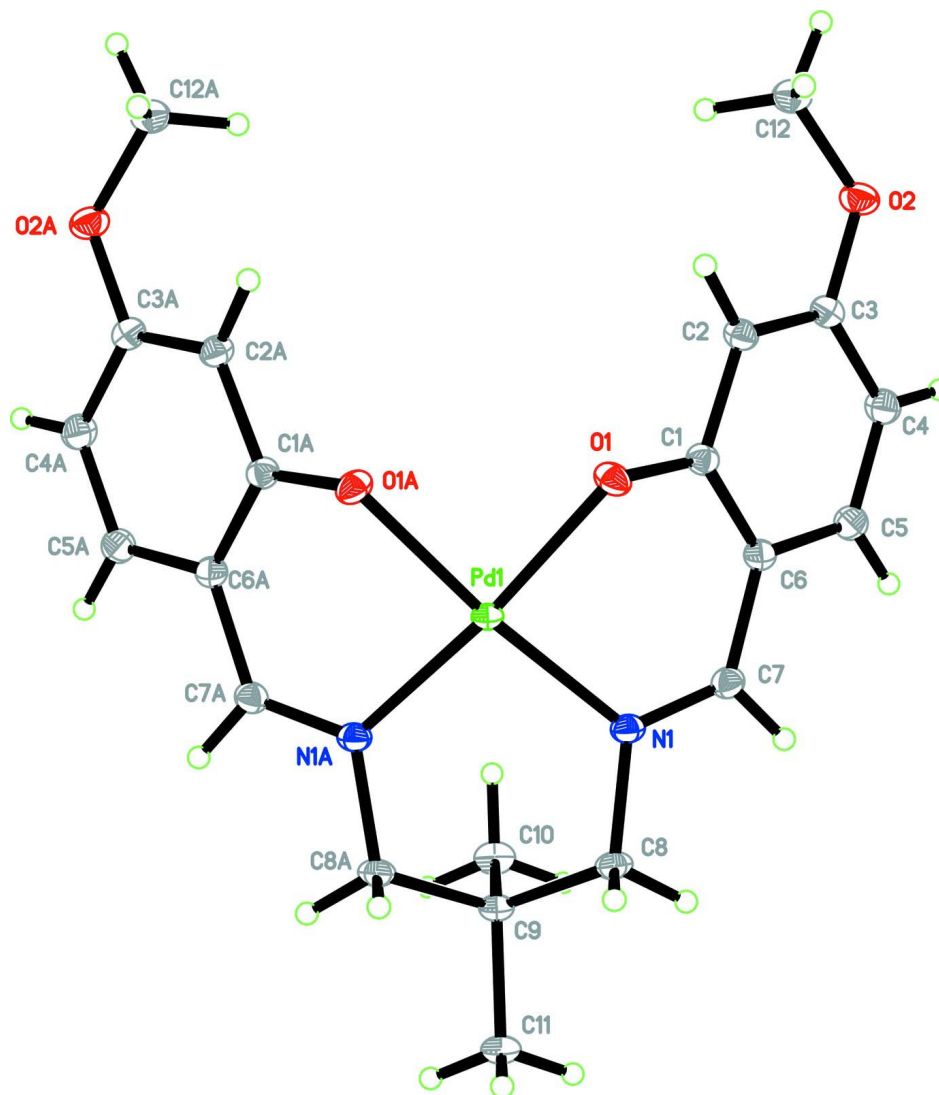
The complex obtained was synthesized by dissolving the *N,N'*-bis(4-methoxy-salicylidene)-2,2-dimethylpropane-1,3-diamine ligand (0.2000 g, 0.54 mmol) in dry acetonitrile (10 ml) in a three necked round bottom flask. Palladium(II) acetate (0.1212 g, 0.54 mmol) which was dissolved separately in dry acetonitrile (10 ml) was then added into the flask containing the ligand solution. The mixture was stirred and refluxed under N<sub>2</sub> gas atmosphere for 3 h. The yellow solid formed was separated by vacuum filtration, washed with cold acetonitrile and allowed to dry *in vacuo*. The solid product was then recrystallized from a mixture of dichloromethane/methanol (1:1 *v/v*). Slow evaporation of the solvent at room temperature over several days gave yellow crystals (yield: 78%). Melting point: 596 K-598 K.

**Refinement**

All H atoms attached to C atoms were fixed geometrically and refined as riding with C—H = 0.95–0.99 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{C-methyl})$ . A rotating group model was applied to the methyl group.

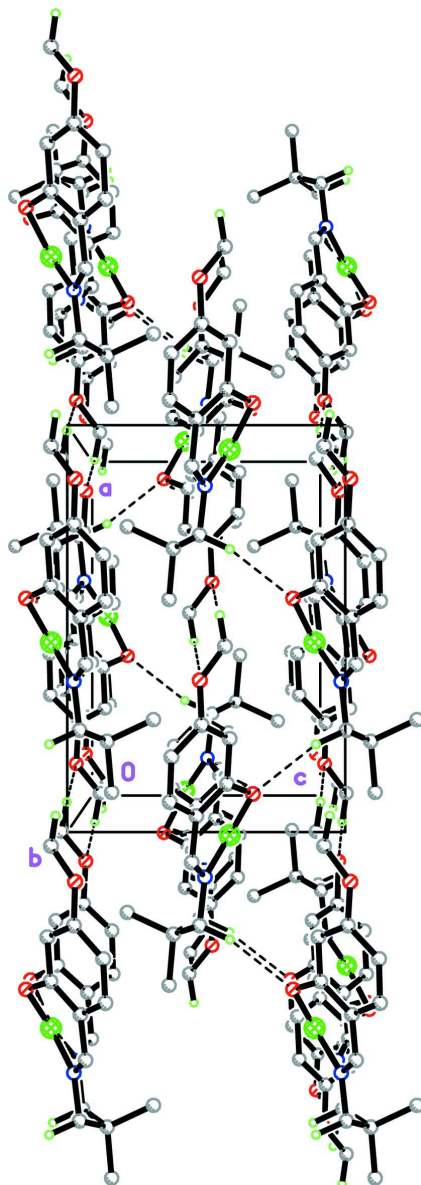
**Computing details**

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).



**Figure 1**

The molecular structure, showing 50% probability displacement ellipsoids. Hydrogen atoms are shown as spheres of arbitrary radius.

**Figure 2**

The crystal packing of (I). Dashed lines indicate hydrogen bonds. H atoms not involved in the hydrogen bond interactions have been omitted for clarity.

**{5,5'-Dimethoxy-2,2'-[2,2-dimethylpropane-1,3-diylbis(nitrilomethanylylidene)]diphenolato}palladium(II)**

*Crystal data*

[Pd(C<sub>21</sub>H<sub>24</sub>N<sub>2</sub>O<sub>4</sub>)]

*M<sub>r</sub>* = 474.82

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

*a* = 11.5470 (4) Å

*b* = 20.9656 (7) Å

*c* = 7.8730 (3) Å

*V* = 1905.97 (12) Å<sup>3</sup>

*Z* = 4

*F*(000) = 968

*D<sub>x</sub>* = 1.655 Mg m<sup>-3</sup>

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 9824 reflections

θ = 3.3–35.1°

μ = 1.00 mm<sup>-1</sup>

*T* = 100 K

Block, yellow

0.27 × 0.24 × 0.18 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer	30756 measured reflections
Radiation source: fine-focus sealed tube	4296 independent reflections
Graphite monochromator	4134 reflections with $I > 2\sigma(I)$
$\varphi$ and $\omega$ scans	$R_{\text{int}} = 0.020$
Absorption correction: multi-scan (SADABS; Bruker, 2009)	$\theta_{\text{max}} = 35.1^\circ$ , $\theta_{\text{min}} = 2.8^\circ$
$T_{\text{min}} = 0.773$ , $T_{\text{max}} = 0.839$	$h = -18 \rightarrow 18$
	$k = -33 \rightarrow 33$
	$l = -8 \rightarrow 12$

Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.016$	H-atom parameters constrained
$wR(F^2) = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0183P)^2 + 0.777P]$
$S = 1.13$	where $P = (F_o^2 + 2F_c^2)/3$
4296 reflections	$(\Delta/\sigma)_{\text{max}} = 0.003$
134 parameters	$\Delta\rho_{\text{max}} = 0.50 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.62 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

**Experimental.** The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	0.535617 (6)	0.2500	0.088830 (10)	0.00982 (2)
O1	0.42276 (5)	0.31425 (3)	0.17501 (8)	0.01411 (10)
O2	0.12833 (5)	0.45403 (3)	-0.02329 (9)	0.01723 (11)
N1	0.63494 (6)	0.31998 (3)	-0.00483 (9)	0.01240 (11)
C1	0.39331 (7)	0.36205 (4)	0.07712 (9)	0.01203 (11)
C2	0.27757 (7)	0.38427 (4)	0.08646 (10)	0.01310 (12)
H2A	0.2251	0.3651	0.1642	0.016*
C3	0.24037 (7)	0.43360 (4)	-0.01673 (10)	0.01370 (12)
C4	0.31792 (7)	0.46562 (4)	-0.12613 (11)	0.01645 (13)
H4A	0.2924	0.5004	-0.1939	0.020*
C5	0.43136 (7)	0.44543 (4)	-0.13264 (11)	0.01566 (13)
H5A	0.4844	0.4675	-0.2038	0.019*
C6	0.47127 (6)	0.39285 (4)	-0.03651 (10)	0.01282 (12)
C7	0.59049 (7)	0.37365 (4)	-0.05364 (10)	0.01349 (12)
H7A	0.6415	0.4033	-0.1059	0.016*

C8	0.76027 (6)	0.31049 (4)	-0.02515 (11)	0.01447 (13)
H8A	0.7964	0.3083	0.0887	0.017*
H8B	0.7932	0.3479	-0.0847	0.017*
C9	0.79183 (9)	0.2500	-0.12444 (14)	0.01195 (16)
C10	0.73574 (10)	0.2500	-0.29972 (15)	0.01630 (19)
H10A	0.7594	0.2874	-0.3609	0.024*
H10B	0.6530	0.2500	-0.2876	0.024*
C11	0.92430 (10)	0.2500	-0.14109 (16)	0.01623 (19)
H11A	0.9487	0.2874	-0.2017	0.024*
H11B	0.9584	0.2500	-0.0299	0.024*
C12	0.04407 (7)	0.41728 (5)	0.06862 (13)	0.01862 (15)
H12A	-0.0332	0.4353	0.0496	0.028*
H12B	0.0622	0.4185	0.1902	0.028*
H12C	0.0457	0.3730	0.0288	0.028*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pd1	0.00822 (4)	0.01192 (4)	0.00933 (4)	0.000	0.00093 (2)	0.000
O1	0.0140 (2)	0.0151 (2)	0.0133 (2)	0.00348 (18)	0.00348 (19)	0.00199 (19)
O2	0.0125 (2)	0.0189 (3)	0.0203 (3)	0.0032 (2)	0.0008 (2)	0.0040 (2)
N1	0.0097 (2)	0.0144 (3)	0.0131 (3)	-0.0006 (2)	0.0015 (2)	-0.0015 (2)
C1	0.0122 (3)	0.0127 (3)	0.0112 (3)	0.0005 (2)	0.0008 (2)	-0.0011 (2)
C2	0.0122 (3)	0.0141 (3)	0.0130 (3)	0.0013 (2)	0.0015 (2)	0.0002 (2)
C3	0.0129 (3)	0.0144 (3)	0.0137 (3)	0.0016 (2)	-0.0001 (2)	-0.0007 (2)
C4	0.0165 (3)	0.0157 (3)	0.0172 (3)	0.0016 (2)	0.0013 (3)	0.0036 (3)
C5	0.0163 (3)	0.0145 (3)	0.0162 (3)	0.0001 (2)	0.0027 (3)	0.0021 (2)
C6	0.0121 (3)	0.0128 (3)	0.0135 (3)	0.0000 (2)	0.0017 (2)	0.0001 (2)
C7	0.0126 (3)	0.0138 (3)	0.0140 (3)	-0.0013 (2)	0.0020 (2)	-0.0007 (2)
C8	0.0089 (3)	0.0173 (3)	0.0171 (3)	-0.0012 (2)	0.0013 (2)	-0.0035 (3)
C9	0.0088 (4)	0.0152 (4)	0.0119 (4)	0.000	0.0012 (3)	0.000
C10	0.0142 (4)	0.0232 (5)	0.0115 (4)	0.000	0.0000 (3)	0.000
C11	0.0097 (4)	0.0200 (5)	0.0189 (5)	0.000	0.0025 (4)	0.000
C12	0.0132 (3)	0.0195 (4)	0.0232 (4)	0.0009 (3)	0.0012 (3)	0.0018 (3)

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

Pd1—O1 <sup>i</sup>	1.9932 (6)	C5—H5A	0.9500
Pd1—O1	1.9932 (6)	C6—C7	1.4406 (11)
Pd1—N1 <sup>i</sup>	2.0029 (7)	C7—H7A	0.9500
Pd1—N1	2.0029 (7)	C8—C9	1.5337 (10)
O1—C1	1.3092 (9)	C8—H8A	0.9900
O2—C3	1.3637 (10)	C8—H8B	0.9900
O2—C12	1.4366 (11)	C9—C10	1.5244 (16)
N1—C7	1.2951 (10)	C9—C8 <sup>i</sup>	1.5336 (10)
N1—C8	1.4695 (10)	C9—C11	1.5353 (15)
C1—C2	1.4172 (11)	C10—H10A	0.9600
C1—C6	1.4239 (11)	C10—H10B	0.9600
C2—C3	1.3835 (11)	C11—H11A	0.9599
C2—H2A	0.9500	C11—H11B	0.9600

C3—C4	1.4122 (12)	C12—H12A	0.9800
C4—C5	1.3776 (12)	C12—H12B	0.9800
C4—H4A	0.9500	C12—H12C	0.9800
C5—C6	1.4144 (11)		
O1 <sup>i</sup> —Pd1—O1	85.03 (3)	C1—C6—C7	122.43 (7)
O1 <sup>i</sup> —Pd1—N1 <sup>i</sup>	90.27 (3)	N1—C7—C6	126.41 (7)
O1—Pd1—N1 <sup>i</sup>	174.10 (3)	N1—C7—H7A	116.8
O1 <sup>i</sup> —Pd1—N1	174.10 (3)	C6—C7—H7A	116.8
O1—Pd1—N1	90.27 (3)	N1—C8—C9	113.67 (7)
N1 <sup>i</sup> —Pd1—N1	94.20 (4)	N1—C8—H8A	108.8
C1—O1—Pd1	119.13 (5)	C9—C8—H8A	108.8
C3—O2—C12	117.06 (7)	N1—C8—H8B	108.8
C7—N1—C8	118.40 (7)	C9—C8—H8B	108.8
C7—N1—Pd1	121.25 (5)	H8A—C8—H8B	107.7
C8—N1—Pd1	120.32 (5)	C10—C9—C8 <sup>i</sup>	111.13 (6)
O1—C1—C2	117.78 (7)	C10—C9—C8	111.13 (6)
O1—C1—C6	123.55 (7)	C8 <sup>i</sup> —C9—C8	111.56 (9)
C2—C1—C6	118.67 (7)	C10—C9—C11	110.24 (9)
C3—C2—C1	120.53 (7)	C8 <sup>i</sup> —C9—C11	106.28 (6)
C3—C2—H2A	119.7	C8—C9—C11	106.28 (6)
C1—C2—H2A	119.7	C9—C10—H10A	109.5
O2—C3—C2	123.47 (7)	C9—C10—H10B	109.5
O2—C3—C4	115.41 (7)	H10A—C10—H10B	109.5
C2—C3—C4	121.11 (7)	C9—C11—H11A	109.6
C5—C4—C3	118.66 (7)	C9—C11—H11B	109.3
C5—C4—H4A	120.7	H11A—C11—H11B	109.5
C3—C4—H4A	120.7	O2—C12—H12A	109.5
C4—C5—C6	121.96 (7)	O2—C12—H12B	109.5
C4—C5—H5A	119.0	H12A—C12—H12B	109.5
C6—C5—H5A	119.0	O2—C12—H12C	109.5
C5—C6—C1	118.93 (7)	H12A—C12—H12C	109.5
C5—C6—C7	118.62 (7)	H12B—C12—H12C	109.5
O1 <sup>i</sup> —Pd1—O1—C1	-132.69 (5)	C3—C4—C5—C6	1.59 (13)
N1—Pd1—O1—C1	43.75 (6)	C4—C5—C6—C1	-3.42 (13)
O1—Pd1—N1—C7	-29.00 (6)	C4—C5—C6—C7	178.41 (8)
N1 <sup>i</sup> —Pd1—N1—C7	147.14 (5)	O1—C1—C6—C5	-177.99 (7)
O1—Pd1—N1—C8	153.13 (6)	C2—C1—C6—C5	1.59 (11)
N1 <sup>i</sup> —Pd1—N1—C8	-30.72 (7)	O1—C1—C6—C7	0.11 (12)
Pd1—O1—C1—C2	144.53 (6)	C2—C1—C6—C7	179.69 (7)
Pd1—O1—C1—C6	-35.88 (10)	C8—N1—C7—C6	-176.76 (8)
O1—C1—C2—C3	-178.41 (7)	Pd1—N1—C7—C6	5.33 (11)
C6—C1—C2—C3	1.98 (11)	C5—C6—C7—N1	-164.51 (8)
C12—O2—C3—C2	-6.96 (12)	C1—C6—C7—N1	17.38 (13)
C12—O2—C3—C4	171.91 (8)	C7—N1—C8—C9	-125.73 (8)
C1—C2—C3—O2	174.91 (7)	Pd1—N1—C8—C9	52.19 (9)
C1—C2—C3—C4	-3.90 (12)	N1—C8—C9—C10	57.06 (10)



O2—C3—C4—C5	-176.79 (8)	N1—C8—C9—C8 <sup>i</sup>	-67.56 (11)
C2—C3—C4—C5	2.11 (13)	N1—C8—C9—C11	177.01 (7)

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

*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>
C8—H8 <i>A</i> $\cdots$ O1 <sup>ii</sup>	0.99	2.37	3.3355 (10)	166
C12—H12 <i>A</i> $\cdots$ O2 <sup>iii</sup>	0.98	2.58	3.3719 (12)	138

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