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Bis{2-[1-(benzylimino)ethyl]phenolato}-palladium(II)

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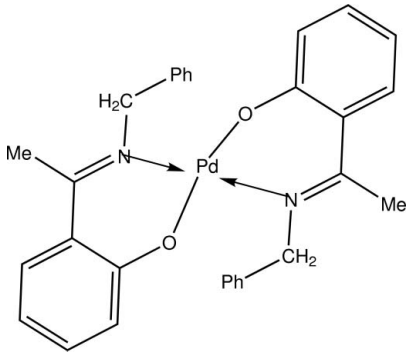
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.036; wR factor = 0.109; data-to-parameter ratio = 13.5.

In the title compound, $[Pd(C_{15}H_{14}NO)_2]$, the Pd atom lies on an inversion center and is coordinated by two ligand molecules through the O and N atoms in a bidentate manner, forming a slightly distorted square-planar geometry. The dihedral angle between the two benzene rings in the ligand is 76.53 (19)°. The molecular packing is stablized by C—H···O and C—H···π interactions.

Related literature

For the catalytic activity of palladium(II)–Schiff base complexes, see: Gupta & Sutar (2008); Lai *et al.* (2005) and for their antitumor activity, see: Garoufis *et al.* (2008). For related structures, see: Adrian *et al.* (2008); Wan Nazihah Wan Ibrahim *et al.* (2008); Chen & Xia (2009). For bond-length data, see: Allen *et al.* (2002).



Experimental

Crystal data

$[Pd(C_{15}H_{14}NO)_2]$
M_r = 554.94
Monoclinic, $P2_1/n$
a = 11.188 (2) Å
b = 9.4460 (17) Å
c = 11.984 (2) Å
β = 110.558 (4)°
V = 1185.8 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.81 mm^{-1}
T = 298 K
0.28 × 0.20 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.804, T_{max} = 0.908
6420 measured reflections
2177 independent reflections
1939 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.109
S = 1.18
2177 reflections
161 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.98 e Å^{-3}

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

*Cg*3 and *Cg*4 are the centroids of the C1–C6 and C10–C16 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9A···O1^{i}	0.97	2.18	2.862 (4)	127
C8—H8B···$Cg4^{ii}$	0.96	2.57	3.523 (5)	172
C13—H13A···$Cg3^{iii}$	0.93	2.87	3.626 (5)	139

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008), *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2311).

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supplementary materials

Acta Cryst. (2010). E**66**, m1100 [**doi:10.1107/S1600536810031375**]

Bis{2-[1-(benzylimino)ethyl]phenolato}palladium(II)

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Comment

The palladium- Schiff bases complexes have found various applications expecially as catalyst (Gupta & Sutar, 2008; Lai *et al.*, 2005) and antitumors activity (Garoufis *et al.*, 2008). The title compound 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 the geometry around the central palladium atom.

In the title molecule (Fig. 1), the palladium atom lies on an inversion center and is coordinated to two ligand molecules through the oxygen and nitrogen atoms in a bidentate manner to form a perfect square planar geometry. The bond distances and bond angles in the title complex are normal (Allen *et al.*, 2002). The Pd1—O and Pd1—N bond lengths of 1.981 (2) and 2.039 (2)Å, respectively, in a square planar geometry are typical of square planar Pd(II) of Schiff bases (Adrian *et al.*, 2008). The dihedral angle between the benzene rings is 76.5 (2)°. The molecule is stabilized by C—H..π (C8—H8B···*Cg*3, C8—H8C···*Cg*4 and C13H-13 A···*Cg*3) interactions (Table 1).

Experimental

The ligand, 2-hydroxyacetophenonebenzylimine, (1.1271 g, 5 mmol) was dissolved in hot ethanol (20 ml) in a round-bottomed flask. Palladium(II) acetate (0.5618 g, 2.5 mmol) was dissolved separately in hot ethanol (40 ml) and added into the flask containing the ligand solution. The mixture was stirred and refluxed for 5 h upon which green precipitate was formed. It was isolated by gravity filtration, washed with cold ethanol and air dried at room temperature. The solid product was recrystallized from chloroform yielding yellow crystals. Yield 87.80%; m.p. 529–530 K.

Refinement

The H atoms were positioned geometrically with C—H = 0.97, 96 and 0.93 Å for methyl, methylene and aromatic groups, respectively, and constrained to ride on their parent atoms with U_{iso}(H) = 1.5(methyl) or 1.2(methylene and aromatic) × U_{eq}(C). The highest peak and deepest hole are located at 0.90 Å from Pd1 atom.

Figures

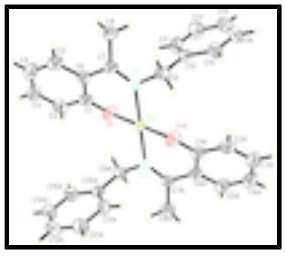


Fig. 1. The molecular structure of the title complex with displacement ellipsods drawn at the 50% probability level.

Bis{2-[1-(benzylimino)ethyl]phenolato}palladium(II)

Crystal data

$[Pd(C_{15}H_{14}NO)_2]$	$F(000) = 568$
$M_r = 554.94$	$D_x = 1.554$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 4170 reflections
$a = 11.188$ (2) Å	$\theta = 2.1$–$25.5°$
$b = 9.4460$ (17) Å	$\mu = 0.81$ mm^{-1}
$c = 11.984$ (2) Å	$T = 298$ K
$\beta = 110.558$ (4)°	Block, yellow
$V = 1185.8$ (4) Å^3	0.28 × 0.20 × 0.12 mm
$Z = 2$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2177 independent reflections
Radiation source: fine-focus sealed tube	1939 reflections with $I > 2/s(I)$
graphite	$R_{int} = 0.038$
Detector resolution: 83.66 pixels mm^{-1}	$\theta_{max} = 25.5°$, $\theta_{min} = 2.1°$
ω scan	$h = -13 \rightarrow 13$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$k = -11 \rightarrow 11$
$T_{min} = 0.804$, $T_{max} = 0.908$	$l = -14 \rightarrow 11$
6420 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.036$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.109$	H-atom parameters constrained
$S = 1.18$	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.7043P]$ where $P = (F_o^2 + 2F_c^2)/3$
2177 reflections	$(\Delta/\sigma)_{max} < 0.001$
161 parameters	$\Delta\rho_{max} = 0.88$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.98$ e Å^{-3}

Special details

Experimental. Analytical calculation for $C_{30}H_{28}N_2O_2Pd$: C,64.93; H,5.09; N,5.05. Found: C,64.58; H,4.97; N,4.94. IR (cm-1): ν(C=N) 1586, ν(C—O) 1323, ν(C—H) 2977, ν(C—N) 1355, ν(Pd—O) 695, ν(Pd—N) 476.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.5000	0.5000	0.0000	0.02879 (16)
O1	0.4462 (2)	0.5912 (3)	0.1236 (2)	0.0409 (6)
N1	0.6541 (2)	0.6317 (3)	0.0459 (2)	0.0323 (6)
C1	0.5319 (3)	0.6226 (3)	0.2285 (3)	0.0335 (7)
C2	0.4944 (3)	0.6076 (4)	0.3284 (3)	0.0422 (8)
H2A	0.4128	0.5748	0.3178	0.051*
C3	0.5765 (4)	0.6406 (4)	0.4419 (3)	0.0468 (9)
H3A	0.5503	0.6291	0.5069	0.056*
C4	0.6971 (4)	0.6906 (4)	0.4583 (4)	0.0498 (10)
H4A	0.7529	0.7116	0.5346	0.060*
C5	0.7346 (3)	0.7095 (4)	0.3632 (4)	0.0428 (9)
H5A	0.8156	0.7458	0.3760	0.051*
C6	0.6551 (3)	0.6758 (3)	0.2454 (3)	0.0326 (7)
C7	0.7007 (3)	0.6991 (3)	0.1464 (3)	0.0335 (7)
C8	0.8047 (4)	0.8088 (4)	0.1658 (4)	0.0437 (9)
H8A	0.7961	0.8529	0.0912	0.066*
H8B	0.8867	0.7638	0.1977	0.066*
H8C	0.7974	0.8793	0.2208	0.066*
C9	0.7070 (3)	0.6577 (3)	−0.0483 (3)	0.0347 (7)
H9A	0.6993	0.5717	−0.0945	0.042*
H9B	0.7972	0.6790	−0.0114	0.042*
C10	0.6432 (3)	0.7774 (3)	−0.1320 (3)	0.0321 (7)
C11	0.5685 (3)	0.8786 (4)	−0.1050 (3)	0.0423 (8)
H11A	0.5541	0.8737	−0.0333	0.051*
C12	0.5149 (5)	0.9877 (4)	−0.1846 (5)	0.0554 (12)
H12A	0.4646	1.0553	−0.1657	0.066*
C13	0.5354 (6)	0.9965 (4)	−0.2906 (5)	0.0569 (13)
H13A	0.4998	1.0701	−0.3432	0.068*
C14	0.6090 (4)	0.8957 (4)	−0.3186 (4)	0.0531 (10)
H14A	0.6231	0.9007	−0.3905	0.064*
C15	0.6619 (3)	0.7873 (4)	−0.2398 (3)	0.0438 (9)
H15A	0.7112	0.7193	−0.2595	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0270 (2)	0.0261 (2)	0.0302 (3)	−0.00145 (11)	0.00621 (17)	0.00023 (12)
O1	0.0325 (12)	0.0481 (14)	0.0402 (14)	−0.0030 (10)	0.0104 (11)	−0.0108 (11)
N1	0.0297 (13)	0.0273 (13)	0.0396 (16)	0.0003 (11)	0.0116 (12)	0.0024 (12)
C1	0.0351 (16)	0.0236 (15)	0.039 (2)	0.0035 (13)	0.0098 (15)	−0.0020 (14)
C2	0.0437 (19)	0.0346 (18)	0.053 (2)	−0.0016 (15)	0.0227 (18)	−0.0025 (16)
C3	0.057 (2)	0.049 (2)	0.036 (2)	0.0019 (18)	0.0177 (18)	−0.0037 (17)
C4	0.053 (2)	0.054 (2)	0.034 (2)	0.0033 (18)	0.0051 (18)	−0.0084 (18)
C5	0.0366 (18)	0.041 (2)	0.046 (2)	0.0004 (15)	0.0083 (17)	−0.0055 (17)
C6	0.0357 (16)	0.0268 (16)	0.0317 (18)	0.0023 (13)	0.0074 (14)	−0.0005 (13)
C7	0.0301 (16)	0.0257 (15)	0.038 (2)	0.0009 (13)	0.0041 (14)	0.0009 (14)
C8	0.0424 (19)	0.0353 (19)	0.050 (2)	−0.0090 (15)	0.0115 (18)	−0.0001 (17)
C9	0.0300 (16)	0.0345 (17)	0.040 (2)	−0.0021 (13)	0.0133 (15)	−0.0002 (14)
C10	0.0320 (16)	0.0330 (16)	0.0305 (18)	−0.0065 (13)	0.0099 (14)	−0.0012 (14)
C11	0.053 (2)	0.043 (2)	0.0287 (19)	0.0076 (16)	0.0111 (16)	0.0007 (16)
C12	0.074 (3)	0.040 (2)	0.052 (3)	0.0167 (18)	0.021 (3)	0.0026 (17)
C13	0.076 (4)	0.038 (2)	0.051 (3)	0.0001 (17)	0.016 (3)	0.0121 (16)
C14	0.072 (3)	0.051 (2)	0.043 (2)	−0.010 (2)	0.029 (2)	0.0042 (18)
C15	0.050 (2)	0.043 (2)	0.046 (2)	−0.0010 (16)	0.0268 (18)	−0.0005 (17)

Geometric parameters (Å, °)

Pd1—O1	1.981 (2)	C7—C8	1.514 (4)
Pd1—O1^{i}	1.981 (2)	C8—H8A	0.9600
Pd1—N1^{i}	2.039 (3)	C8—H8B	0.9600
Pd1—N1	2.039 (3)	C8—H8C	0.9600
O1—C1	1.319 (4)	C9—C10	1.514 (5)
N1—C7	1.299 (4)	C9—H9A	0.9700
N1—C9	1.468 (4)	C9—H9B	0.9700
C1—C2	1.407 (5)	C10—C11	1.380 (5)
C1—C6	1.414 (4)	C10—C15	1.382 (5)
C2—C3	1.384 (5)	C11—C12	1.389 (6)
C2—H2A	0.9300	C11—H11A	0.9300
C3—C4	1.377 (5)	C12—C13	1.371 (8)
C3—H3A	0.9300	C12—H12A	0.9300
C4—C5	1.357 (6)	C13—C14	1.375 (6)
C4—H4A	0.9300	C13—H13A	0.9300
C5—C6	1.415 (5)	C14—C15	1.377 (6)
C5—H5A	0.9300	C14—H14A	0.9300
C6—C7	1.463 (5)	C15—H15A	0.9300
O1—Pd1—O1^{i}	180.00 (8)	C7—C8—H8A	109.5
O1—Pd1—N1^{i}	90.97 (10)	C7—C8—H8B	109.5
O1^{i}—Pd1—N1^{i}	89.03 (10)	H8A—C8—H8B	109.5
O1—Pd1—N1	89.03 (10)	C7—C8—H8C	109.5

O1^{i}—Pd1—N1	90.97 (10)	H8A—C8—H8C	109.5
N1^{i}—Pd1—N1	180.00 (11)	H8B—C8—H8C	109.5
C1—O1—Pd1	120.0 (2)	N1—C9—C10	114.3 (3)
C7—N1—C9	119.5 (3)	N1—C9—H9A	108.7
C7—N1—Pd1	125.1 (2)	C10—C9—H9A	108.7
C9—N1—Pd1	115.2 (2)	N1—C9—H9B	108.7
O1—C1—C2	117.0 (3)	C10—C9—H9B	108.7
O1—C1—C6	124.2 (3)	H9A—C9—H9B	107.6
C2—C1—C6	118.7 (3)	C11—C10—C15	118.3 (3)
C3—C2—C1	121.4 (3)	C11—C10—C9	123.0 (3)
C3—C2—H2A	119.3	C15—C10—C9	118.6 (3)
C1—C2—H2A	119.3	C10—C11—C12	120.3 (4)
C4—C3—C2	119.7 (3)	C10—C11—H11A	119.9
C4—C3—H3A	120.1	C12—C11—H11A	119.9
C2—C3—H3A	120.1	C13—C12—C11	120.5 (4)
C5—C4—C3	120.1 (4)	C13—C12—H12A	119.7
C5—C4—H4A	119.9	C11—C12—H12A	119.7
C3—C4—H4A	119.9	C12—C13—C14	119.6 (4)
C4—C5—C6	122.4 (4)	C12—C13—H13A	120.2
C4—C5—H5A	118.8	C14—C13—H13A	120.2
C6—C5—H5A	118.8	C13—C14—C15	119.8 (4)
C1—C6—C5	117.6 (3)	C13—C14—H14A	120.1
C1—C6—C7	122.5 (3)	C15—C14—H14A	120.1
C5—C6—C7	119.9 (3)	C14—C15—C10	121.4 (3)
N1—C7—C6	122.5 (3)	C14—C15—H15A	119.3
N1—C7—C8	120.9 (3)	C10—C15—H15A	119.3
C6—C7—C8	116.5 (3)		
O1^{i}—Pd1—O1—C1	−151 (100)	C4—C5—C6—C7	179.6 (3)
N1^{i}—Pd1—O1—C1	138.5 (2)	C9—N1—C7—C6	−178.3 (3)
N1—Pd1—O1—C1	−41.5 (2)	Pd1—N1—C7—C6	6.9 (4)
O1—Pd1—N1—C7	20.0 (3)	C9—N1—C7—C8	2.7 (4)
O1^{i}—Pd1—N1—C7	−160.0 (3)	Pd1—N1—C7—C8	−172.1 (2)
N1^{i}—Pd1—N1—C7	156 (100)	C1—C6—C7—N1	−24.1 (5)
O1—Pd1—N1—C9	−155.0 (2)	C5—C6—C7—N1	157.2 (3)
O1^{i}—Pd1—N1—C9	25.0 (2)	C1—C6—C7—C8	155.0 (3)
N1^{i}—Pd1—N1—C9	−19 (100)	C5—C6—C7—C8	−23.7 (5)
Pd1—O1—C1—C2	−144.1 (2)	C7—N1—C9—C10	−89.1 (3)
Pd1—O1—C1—C6	38.9 (4)	Pd1—N1—C9—C10	86.1 (3)
O1—C1—C2—C3	−178.7 (3)	N1—C9—C10—C11	17.5 (4)
C6—C1—C2—C3	−1.5 (5)	N1—C9—C10—C15	−163.5 (3)
C1—C2—C3—C4	0.7 (6)	C15—C10—C11—C12	−0.5 (6)
C2—C3—C4—C5	1.0 (6)	C9—C10—C11—C12	178.6 (4)
C3—C4—C5—C6	−1.8 (6)	C10—C11—C12—C13	−0.1 (7)
O1—C1—C6—C5	177.7 (3)	C11—C12—C13—C14	0.5 (8)
C2—C1—C6—C5	0.7 (4)	C12—C13—C14—C15	−0.3 (7)
O1—C1—C6—C7	−1.0 (5)	C13—C14—C15—C10	−0.3 (6)
C2—C1—C6—C7	−178.0 (3)	C11—C10—C15—C14	0.7 (5)

C4—C5—C6—C1	0.9 (5)	C9—C10—C15—C14	−178.4 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*.

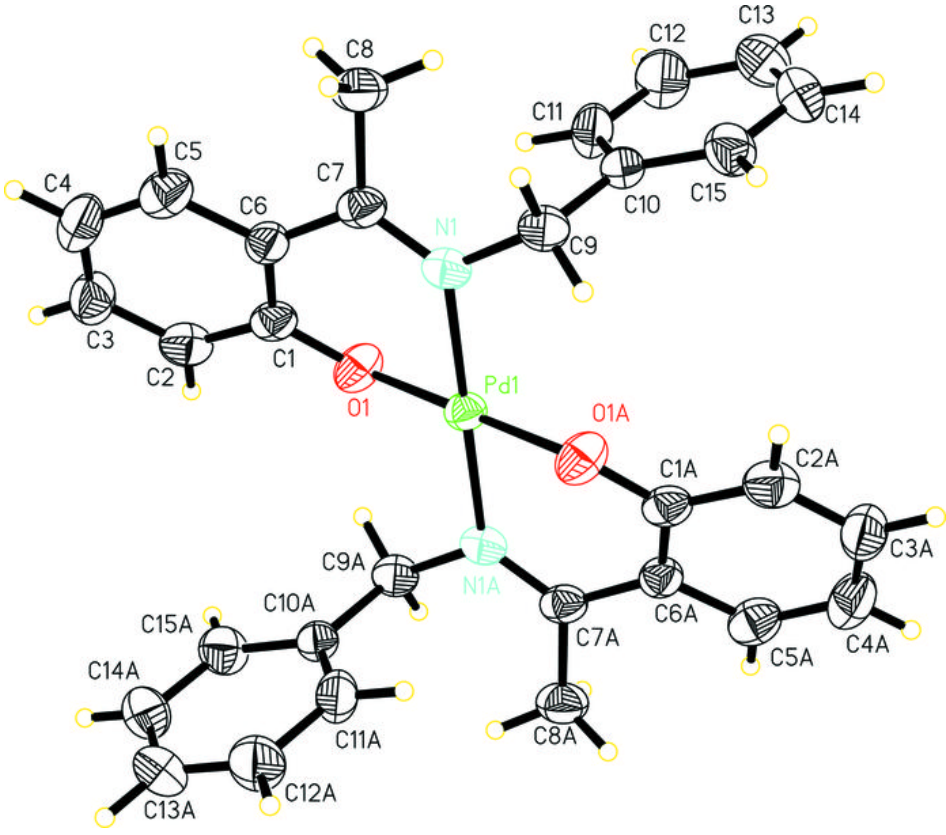
Hydrogen-bond geometry (Å, °)

Cg3 and Cg4 are the centroids of the ???? and ???? rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9A···O1^{i}	0.97	2.18	2.862 (4)	127
C11—H11A···N1	0.93	2.58	2.900 (5)	101
C8—H8B···Cg4^{ii}	0.96	2.57	3.523 (5)	172
C13—H13A···Cg3^{iii}	0.93	2.87	3.626 (5)	139

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

Fig. 1


C8
C12
C13
C11
C14
C5
C7
C4
C6
C10
C15
N1
C9
C3
C1
C2
O1
Pd1
O1A
C1A
C2A
C3A
C9A
N1A
C10A
C6A
C15A
C7A
C5A
C4A
C14A
C11A
C8A
C12A
C13A