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(2,2′-Bipyridine-κ^2N,N')(3-oxidonaphthalene-2-carboxylato-$\kappa^2O:O'$)palladium(II) hemihydrate

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Key indicators

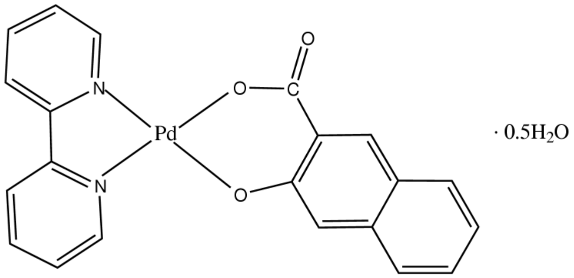
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.031
wR factor = 0.044
Data-to-parameter ratio = 15.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Pd(C_{11}H_6O_3)(C_{10}H_8N_2)]\cdot0.5H_2O$, contains two monoclear Pd^{II} complexes and one water molecule in the asymmetric unit. The Pd atom of each complex has a similar distorted *cis*-square-planar coordination geometry and is bonded to two N atoms from 2,2′-bipyridine ligand, and one carboxylate O and one naphthol O atom from the 3-oxidonaphthalene-2-carboxylate ligand. There are intermolecular π–π interactions, together with hydrogen bonds involving the solvent water molecule.

Comment

Palladium(II) complexes including planar aromatic ligands are considered to be potential DNA intercalators (Sarneski *et al.*, 1977; Coyer *et al.*, 1991). We have reported several palladium(II) complexes with heterocyclic aromatic ligands (Okabe *et al.*, 2003, 2004; Muranishi & Okabe., 2004). In the present study, we have prepared the title compound, (I), and determined its structure.



(I)

In (I), two independent palladium(II) complexes exist in the asymmetric unit (Fig. 1). In both complex molecules, each Pd^{II} atom has similar distorted *cis*-square-planar coordination geometry, in which the Pd^{II} atom is bonded to two N atoms from the 2,2′-bipyridine (bpy) ligand, and one carboxylate O atom and the oxide O atom from the 3-oxidonaphthalene-2-carboxylate (nc) ligand. The Pd—O(oxide) bond is slightly shorter and stronger than the Pd—O(carboxylate) bond (Table 1). The Pd—O and Pd—N distances in (I) may be compared with those in [Pd(bpy)(dhn)] [dhn is 2,3-dihydroxynaphthalene; Pd—O = 1.981 (3)–1.984 (2) Å and Pd—N = 2.001 (3)–2.003 (2) Å], [Pd(bq)(dhn)] [bq is biquinoline; Pd—O = 1.982 (4)–1.992 (4) Å and Pd—N = 2.037 (4)–2.039 (4) Å; Okabe *et al.*, 2004] or [Pd(phen)(ca)] [phen is 1,10-phenanthrorine and ca is catechol; Pd—O = 1.981–1.989 Å and Pd—N = 2.003 (2)–2.019 (2) Å; Okabe *et al.*, 2003], since all of these palladium(II) complexes have *O,O′*-bidentate or *N,N′*-aromatic ligands analogous to (I). Both

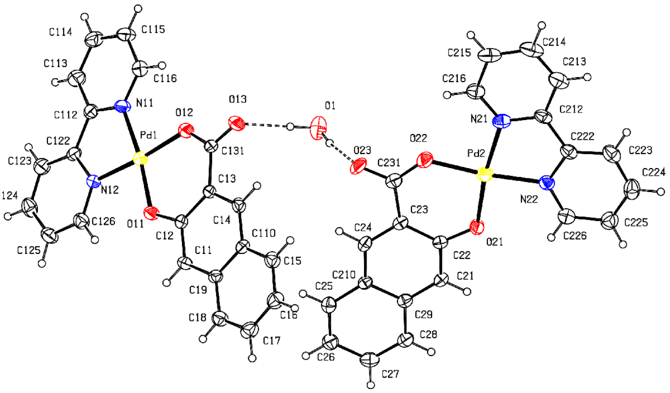


Figure 1
ORTEP-3 (Farrugia, 1997) drawing of the asymmetric unit of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

Pd—O bonds in (I) are somewhat stronger than the corresponding bonds in the above reported complexes, while the Pd—N bond distances of all these heterocyclic nitrogen ligands are similar to each other.

In the Pd1 molecule, the dihedral angle between the two pyridine rings of bpy is 0.6 (14)°. The dihedral angles between the naphthalene ring of the nc ligand and the N11 and N12-containing pyridine rings are 1.9 (11) and 2.5 (10)°, respectively. In the Pd2 molecule, the corresponding values are 2.9 (14) (two pyridine rings of bpy), 3.0 (11) (nc and N21 ring) and 1.1 (12)° (nc and N22 ring). These indicate that the overall structure of each complex molecule is essentially planar. In the crystal packing, shown in Fig. 2, one water molecule links the two independent complex molecules by hydrogen bonds (Table 2). Furthermore, the Pd1 molecule is stacked above an adjacent Pd2 molecule at (x, $-y-\frac{1}{2}$, $z-\frac{1}{2}$), with a Pd$\cdots$Pd distance of 3.8379 (3) Å.

Experimental

Brown needle-shaped crystals of (I) were obtained by slow evaporation of a dimethylformamide solution of a mixture of 3-hydroxynaphthalene-2-carboxylic acid, 2,2′-bipyridine and Pd(CH$_3$COO)$_2$ (molar ratio 1:1:1) at room temperature.

Crystal data

[Pd(C$_{11}$H$_6$O$_3$)(C$_{10}$H$_8$N$_2$)]·0.5H$_2$O
M_r = 457.77
Monoclinic, $P2_1/c$
a = 21.399 (8) Å
b = 7.504 (5) Å
c = 24.891 (16) Å
β = 120.15 (4)°
V = 3456 (4) Å^3
Z = 8
D_x = 1.759 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16 615 reflections
θ = 3.2–27.4°
μ = 1.10 mm^{-1}
T = 293 (2) K
Needle, brown
0.40 × 0.05 × 0.05 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min}$ = 0.798, $T_{\max}$ = 0.946
33 195 measured reflections
7876 independent reflections
3381 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.082
$\theta_{\max}$ = 27.5°
$h = -27 \rightarrow 25$
$k = -9 \rightarrow 8$
$l = -32 \rightarrow 24$

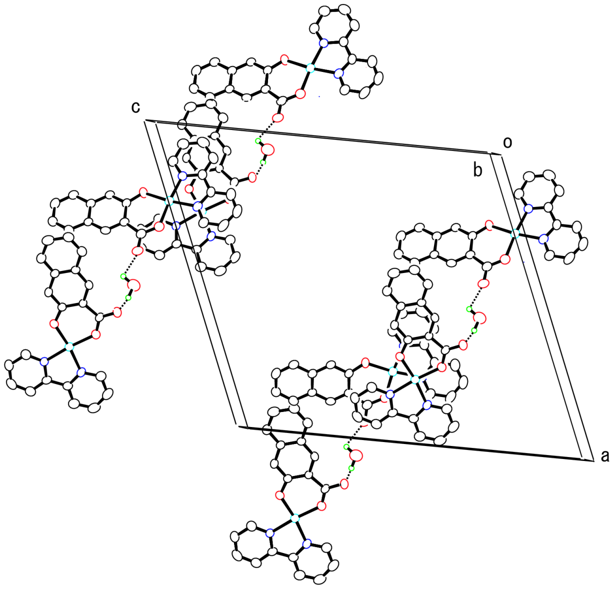


Figure 2
Part of the molecular packing of (I). Dashed lines indicate hydrogen bonds.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.044
S = 0.66
7876 reflections
496 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0133P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.41 e Å^{-3}
$\Delta\rho_{\min}$ = −0.39 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—O11	1.942 (2)	Pd2—O21	1.951 (2)
Pd1—O12	1.977 (2)	Pd2—O22	1.978 (2)
Pd1—N12	1.989 (3)	Pd2—N22	1.998 (3)
Pd1—N11	2.012 (3)	Pd2—N21	2.005 (3)
O11—Pd1—O12	94.56 (9)	O21—Pd2—O22	94.73 (10)
O11—Pd1—N12	91.42 (11)	O21—Pd2—N22	91.62 (12)
O12—Pd1—N12	173.99 (10)	O22—Pd2—N22	173.65 (11)
O11—Pd1—N11	171.81 (10)	O21—Pd2—N21	171.95 (10)
O12—Pd1—N11	93.62 (11)	O22—Pd2—N21	93.17 (11)
N12—Pd1—N11	80.39 (12)	N22—Pd2—N21	80.48 (13)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$O13	0.94	1.84	2.777 (3)	176
O1—H2$\cdots$O23	0.94	1.91	2.844 (3)	171

All H atoms, including those of the water molecule, were located in difference Fourier maps, and treated as riding, with C—H distances of 0.93 Å and O—H distances of 0.94 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom). The goodness-of-fit parameter was relatively low (S = 0.66), although the weighting scheme was optimized.

Data collection: *RAPID-AUTO* (Rigaku, 2003); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004) and *CRYSTALS* (Watkin *et al.*, 1996); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999) and *DIRDIF*99 (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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