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(1,2-Benzenediolato-κ^2O,O')(2,2′-biquinoline-κ^2N,N')-palladium(II)

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.032
wR factor = 0.089
Data-to-parameter ratio = 16.3

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

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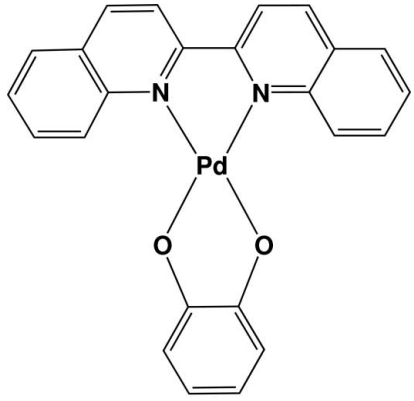
In the title complex, [Pd($C_6H_4O_2$)($C_{18}H_{12}N_2$)], the central Pd atom has a distorted *cis*-planar four-coordination geometry defined by two O atoms of a 1,2-benzenediolate dianion and two N atoms of a 2,2′-biquinoline ligand. The overall structure of the complex is not planar, with a dihedral angle of 136.23 (8)° between the quinoline and benzenediolate mean planes.

Comment

A large number of investigations have focused on the design of palladium(II) or platinum(II) complexes because of their biological significance in areas such as antitumor, antimicrobial and antiviral activity (Corbi *et al.*, 2005; Giovagnini *et al.*, 2005).



(I)

Palladium complexes with aromatic ligands and *cis*-square planar coordination geometry have been intensively studied because of their cytotoxic activity (Mansuri-Torshizi *et al.*, 2001; Afrasiabi *et al.*, 2004; Rebolledo *et al.*, 2005; Padhye *et al.*, 2005) or DNA binding properties (Cusumano & Giannetto, 1997; Tercero *et al.*, 2003); such binding is the principal target in the chemotherapy of tumors (Shehata, 2001; Cusumano & Giannetto, 1997; Neidle *et al.*, 1987).

As an extension of these studies, we have synthesized and determined the crystal structures of a number of *cis*-coordinated palladium complexes with aromatic ligands including Pd(bpy)(cbdca) and Pb(phen)(cbdca) (Muranishi & Okabe, 2004), Pd(phen)(ca) (Okabe *et al.*, 2003), Pd(bpy)(nad) and Pd(biq)(nad) (Okabe *et al.*, 2004) and Pd(bpy)(ca) (Okabe *et al.*, 2005), where bpy is 2,2′-bipyridine, cbdca is 1,1-cyclobutanedicarboxylate, phen is 1,10-phenanthroline, nad is 2,3-naphthalenediolate, biq is biquinoline and ca is catecholate or 1,2-benzenediolate.

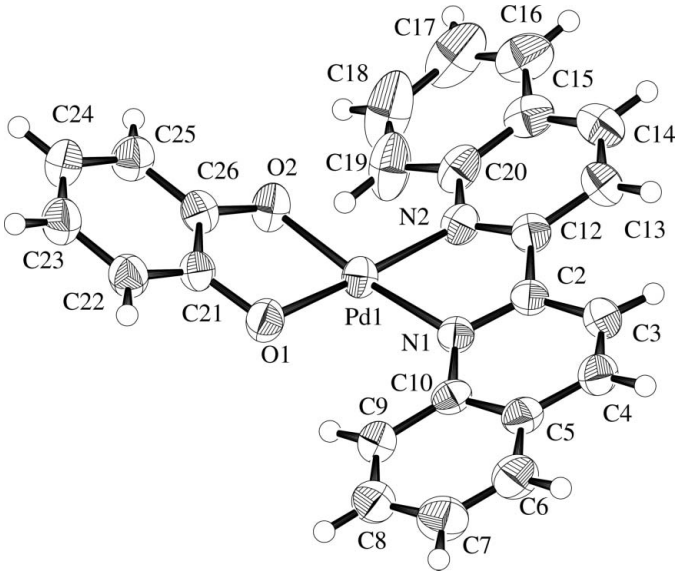


Figure 1
The molecular structure of (I), showing 50% displacement ellipsoids for the non-H atoms.

In this study, the title compound, Pd(biq)(ca), (I), has been synthesized and its structure determined. In complex (I), the central Pd atom has a distorted *cis*-square-planar coordination geometry arising from the two N atoms of biq and the two O atoms of ca (Fig. 1).

The overall structure is not planar and resembles that of Pd(biq)(nad), (II) (Okabe *et al.*, 2004), which has the same biq ligand as (I) and two analogous anionic O atoms of the 2,3-naphthalenediolate ligand. The dihedral angle between the biq and ca mean planes in (I) is 136.23 (8)°, with a corresponding value for (II) of 148.73 (7)° (where a dihedral angle of 180° indicates a flat structure). This shows that the distortion of (I) from the planar conformation is larger than in (II).

The Pd—*X* (*X* = N, O) bond lengths and angles in (I) are similar to those of (II) (Table 1). This resemblance suggests similar coordination bond strength in both complexes.

Experimental

Firstly, biq (5.0 mg dissolved in 2 ml dimethylformamide, DMF) was reacted with palladium acetate, $Pd(CH_3COOH)_2$ (4.4 mg in 2 ml DMF) for 15 min at room temperature (molar ratio of 1:1). An equimolar amount of caH_2 dissolved in DMF was then added with stirring. This mixture was left to stand at room temperature and yielded blue block-shaped crystals of (I) over a period of days.

Crystal data

$[Pd(C_6H_4O_2)(C_{18}H_{12}N_2)]$
M_r = 470.81
Monoclinic, $P2_1/c$
a = 11.042 (2) Å
b = 9.487 (2) Å
c = 17.796 (2) Å
β = 95.635 (10)°
V = 1855.2 (6) Å^3
Z = 4
D_x = 1.686 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 14.4–15.0°
μ = 1.02 mm^{-1}
T = 296.2 K
Plate, blue
0.20 × 0.15 × 0.10 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.832, T_{max} = 0.903
4749 measured reflections
4267 independent reflections
2996 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 27.5°
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 12$
$l = -23 \rightarrow 23$
3 standard reflections
every 150 reflections
intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.089
S = 1.07
4267 reflections
262 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Comparative selected geometric parameters (Å, °).

	(I)	(II)[i]
Pd1—O1	1.987 (2)	1.992 (4)
Pd1—O2	1.990 (2)	1.982 (4)
Pd1—N1	2.055 (3)	2.039 (4)
Pd1—N2	2.047 (2)	2.037 (4)
O1—Pd1—O2	83.19 (9)	83.6 (2)
O1—Pd1—N1	98.19 (10)	98.4 (2)
O1—Pd1—N2	173.8 (1)	171.1 (2)
O2—Pd1—N1	172.1 (1)	174.4 (2)
O2—Pd1—N2	97.49 (10)	97.0 (2)
N1—Pd1—N2	80.3 (1)	80.3 (2)

Note: (i) Okabe *et al.* (2004).

All H atoms were located in difference Fourier maps and then repositioned in idealized locations and treated as riding, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(carrier).

Data collection: MSC/AFC Diffractometer Control Software (Molecular Structure Corporation, 1992); cell refinement: MSC/AFC Diffractometer Control Software; data reduction: *TEXSAN* (Molecular Structure Corporation, 2000); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN* (Molecular Structure Corporation, 2000).

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