(Benzene-1,2-diolato-κ^2O,O')(2,2′-bipyridine-κ^2N,N')-palladium(II)

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.009 Å
R factor = 0.043
wR factor = 0.136
Data-to-parameter ratio = 16.2

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

In the title complex, [Pd(C₆H₄O₂)(C₁₀H₈N₂)], the central Pd atom has a distorted *cis* planar four-coordination geometry defined by two O atoms of the benzene-1,2-diolate dianion and the two N atoms of a 2,2′-bipyridine ligand. The molecule is essentially planar.

Comment

The palladium complex [Pd(bpy)(cbdca)] (where bpy is 2,2′-bipyridine and cbdca is 1,1-cyclobutanedicarboxylate), with an aromatic ligand and a *cis*-square planar coordination geometry, has been shown to have better cytotoxic activity than cisplatin, *cis*-diamminedichloroplatinum(II), against P$_{388}$ lymphocytic leukemia cells (Mansuri-Torshizi *et al.*, 2001).

(I)
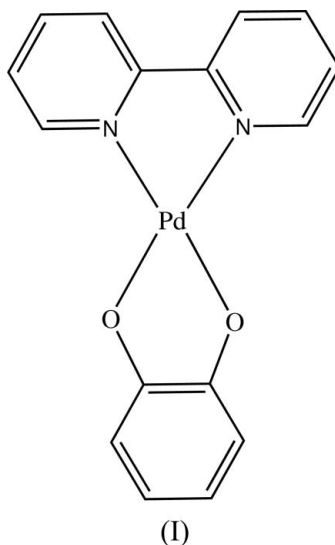
The planar aromatic ligands may interact easily with DNA, which is the principal target in the chemotherapy of tumors (Shehata, 2001; Cusumano & Giannetto,1997; Neidle *et al.*, 1987).

We have synthesized and determined the crystal structures of several *cis*-coordinated palladium complexes with aromatic ligands, *viz.* [Pd(bpy)(cbdca)], [Pb(phen)(cbdca)] (where phen is 1,10-phenanthroline; Muranishi & Okabe, 2004), [Pd(bpy)(nad)], [Pd(biq)(nad)] (where nad is 2,3-naphthalenediol and biq is biquinoline; Okabe *et al.*, 2004) and [Pd(phen)(ca)] (where ca is catechol, *i.e.* 1,2-benzenediol; Okabe *et al.*, 2003). In this study, [Pd(bpy)(ca)], (I), has been synthesized and its structure determined.

In complex (I), the central Pd atom has a distorted *cis*-square planar coordination geometry with the two N atoms of the heterocycle and the two O atoms of the dianion of the ca

ligand (Fig. 1). The overall structure resembles the structures of [Pd(byp)(nad)], (II), and [Pd(phen)(ca)], (III), which have similar bypyridyl and catecholate groups to (I).

The coordinate bond lengths and angles of (I) (Table 1) may be compared with those of (II) and (III), as well as those of other palladium complexes with non-aromatic ligands, such as [Pd(en)mm], (IV), and [Pd(en)cd], (V) (where en is ethyl-enediamine, mm is methyl malonate and cd is 1,1-cyclo-butanedicarboxylate; Tercero *et al.*, 2003). The Pd—O and Pd—N bond lengths of (I) resemble those of (II) and (III), in which the Pd—O bond lengths lie in the range 1.981 (2)–1.989 (2) Å and Pd—N in the range 2.001 (3)–2.019 (2) Å, but are slightly shorter than those of (IV) and (V), in which the Pd—O bond lengths lie in the range 1.995 (10)–2.027 (10) Å and Pd—N in the range 2.009 (11)–2.030 (2) Å. The O—Pd—O and N—Pd—N bond angles also resemble those of (II) and (III), for which O—Pd—O lie in the range 84.84 (9)–85.09 (7)° and N—Pd—N in the range 80.78 (9)–81.69 (8)°, but are a little smaller than those of (IV) and (V), for which O—Pd—O lie in the range 89.51 (17)–92.69 (7)° and N—Pd—N in the range 84.09 (18)–84.15 (8)°. These differences may be explained by the difference in intramolecular mobility of the O and N atoms between the complexes with aromatic ligands and those with non-aromatic ligands, as explained by Okabe *et al.* (2003).

Experimental

Bpy (7 mg, 0.0448 mmol) dissolved in dimethylformamide (DMF, 1.0 ml) was reacted with palladium acetate, [Pd(CH₃COOH)₂] (10 mg), dissolved in DMF (2 ml), and then bpy (5 mg) dissolved in DMF (1 ml) was added. The mixture was allowed to stand at room temperature for a number of days to give red plate crystals of the complex.

Crystal data

[Pd(C$_6$H$_4$O$_2$)(C$_{10}$H$_8$N$_2$)]	$D_x = 1.840$ Mg m^{-3}
$M_r = 370.70$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 25
$a = 7.017$ (2) Å	reflections
$b = 23.873$ (2) Å	$\theta = 14.8$–15.0°
$c = 8.118$ (3) Å	$\mu = 1.39$ mm^{-1}
$\beta = 100.30$ (3)°	$T = 296.2$ K
$V = 1338.0$ (6) Å^3	Thick plate, red
$Z = 4$	$0.30 \times 0.30 \times 0.15$ mm

Data collection

Rigaku AFC-5R diffractometer	$R_{int} = 0.013$
ω–2θ scans	$\theta_{max} = 27.5°$
Absorption correction: ψ scan	$h = 0 \rightarrow 9$
(North *et al.*, 1968)	$k = 0 \rightarrow 31$
$T_{min} = 0.632$, $T_{max} = 0.812$	$l = -10 \rightarrow 10$
3414 measured reflections	3 standard reflections
3079 independent reflections	every 150 reflections
2709 reflections with $I > 2\sigma(I)$	intensity decay: 0.1%

Refinement

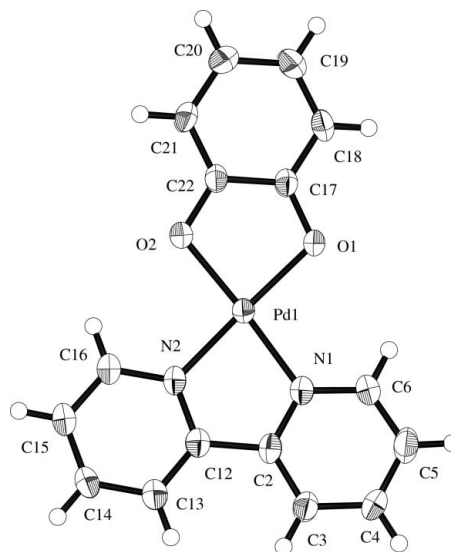|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.043$	$+ 6.095P]$
$wR(F^2) = 0.136$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.21$	$(\Delta/\sigma)_{max} < 0.001$
3079 reflections	$\Delta\rho_{max} = 1.94$ e Å^{-3}
190 parameters	$\Delta\rho_{min} = -1.62$ e Å^{-3}
H-atom parameters constrained	

Figure 1
Molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms correspond to 50% probability.

Table 1
Selected geometric parameters (Å, °).

Pd1—O1	1.992 (4)	Pd1—N2	1.993 (4)
Pd1—O2	1.980 (4)	O1—C17	1.345 (7)
Pd1—N1	2.003 (4)	O2—C22	1.354 (6)
O1—Pd1—O2	85.6 (1)	O2—Pd1—N1	175.7 (2)
O1—Pd1—N1	97.9 (2)	O2—Pd1—N2	96.2 (2)
O1—Pd1—N2	178.1 (2)	N1—Pd1—N2	80.3 (2)

All H atoms were located in difference Fourier maps, and then were placed at ideal positions and treated as riding, with a C—H distance of 0.93 Å and U_{iso}(H) = 1.2U_{eq}(carrier atom). The two highest ghost peaks lie between the complexes.

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*.

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