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(Cyclobutane-1,1-dicarboxylato-κ^2O,O')-(di-2-pyridylamine-κ^2N,N')palladium(II)

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.020
wR factor = 0.074
Data-to-parameter ratio = 15.1

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

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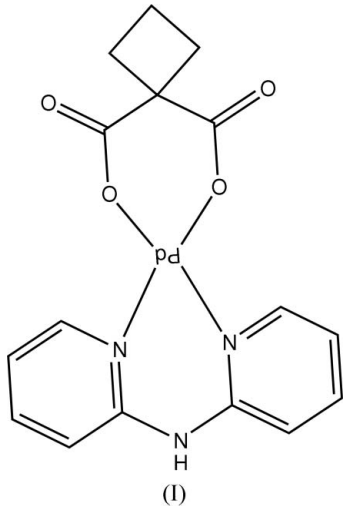
In the title complex, [Pd($C_6H_6O_2$)($C_{10}H_9N_3$)], the Pd atom has a distorted *cis*-square-planar geometry defined by bidentate di-2-pyridylamine and cyclobutane-1,1-dicarboxylate ligands. The complexes interact with each other *via* N—H···O hydrogen bonds, forming a head-to-tail polymeric chain along the *b* axis.

Comment

Novel Pd^{II} complexes are important for the design of new therapeutic drugs (*e.g.* Mansuri-Torshizi *et al.*, 2001; Lee *et al.*, 1994; Ali *et al.*, 2002; Giovagnini *et al.*, 2005). In a previous study, we reported the structures of Pd^{II} complexes with 2,2′-bypyridine (bpy) or 1,10-phenanthroline (phen), and cyclobutane-1,1-dicarboxylate (cbdca) (Muranishi & Okabe, 2004), *i.e.* [Pd(cbdca)(bpy)], (II), and [Pd(cbdca)(phen)], (III), respectively. Here, the 2,2′-bipyridylamine (bpa) ligand, with a central amine group, has been used as the *N*,*N*′-bidentate ligand. As a result of the high rotational flexibility around the bridging amine group in bpa, the two pyridine rings adopt either coplanar or tilted conformations in their coordination complexes (Shepherd *et al.*, 2000). In the present study, we report the structure of the title complex [Pd(cbdca)(bpa)], (I).



(I)

The Pd atom in (I) (Fig. 1) has a distorted *cis*-square-planar coordination geometry involving two N atoms of the bpa ligand and two O atoms of the cbdca ligand. The overall structure closely resembles those found in complexes (II) and (III) (Muranishi & Okabe, 2004). The six-membered chelate rings Pd1/N1/C5/N2/C6/N3 and Pd1/O1/C11/C12/C16/O4 formed between the Pd and the bpa and cbdca ligands, respectively, are non-planar.

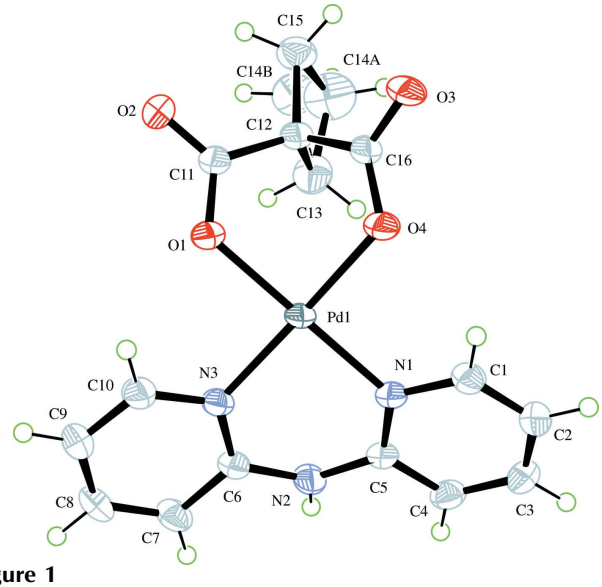


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level. Atoms C14*A* and C14*B* are disordered.

The bond lengths about the Pd atom (Table 1) are comparable with those in (II) [Pd—O = 2.002 (2) and 2.004 (2) Å; Pd—N = 1.999 (2) and 1.998 (2) Å] and in the monohydrate (III*a*) [Pd—O = 2.003 (4) and 2.005 (4) Å; Pd—N = 1.991 (5) and 1.994 (5) Å] and dihydrate (III*b*) [Pd—O = 1.982 (3) and 2.001 (3) Å; Pd—N = 2.002 (4) and 2.010 (4) Å]. The O—Pd—O angle in (I) is also comparable with that in (II), (III*a*) and (III*b*), while the N—Pd—N angle is wider than those formed by the five-membered chelate rings [80.80 (8)° in (II), 82.2 (2)° in (III*a*) and 82.0 (2)° in (III*b*)]. The dihedral angle between the pyridine rings in the bpa ligand is 19.1 (1)°, indicating that the bpa ligand adopts a nearly planar conformation.

As shown in Fig. 2, the amine group of the bpa ligand forms an N—H···O hydrogen bond to the cbdca ligand, forming a head-to-tail polymeric chain along the *b* axis [H5···O2^{i} = 2.17 Å, N2···O2^{i} = 2.865 (4) Å, N2—H5···O2^{i} = 138 Å; symmetry code: (i) *x*, *y* − 1, *z*].

Experimental

Complex (I) was prepared by reacting di-2-pyridylamine with [Pd(CH$_3$COOH)$_2$] for 15 min at room temperature (molar ratio of 1:1) in a dimethylformamide solution. This was followed by the addition of an equimolar amount of cyclobutane-1,1-dicarboxylic acid. The mixture was left to stand at room temperature and yellow platelets of (I) appeared after a few days.

Crystal data

[Pd(C$_6$H$_6$O$_2$)(C$_{10}$H$_9$N$_3$)]
M_r = 419.73
Triclinic, $P\overline{1}$
a = 9.115 (6) Å
b = 9.698 (8) Å
c = 10.227 (6) Å
α = 67.92 (3)°
β = 67.30 (2)°
γ = 71.21 (3)°
V = 756.1 (9) Å^3
Z = 2
D_x = 1.844 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.25 mm^{-1}
T = 296 K
Platelet, yellow
0.30 × 0.30 × 0.10 mm

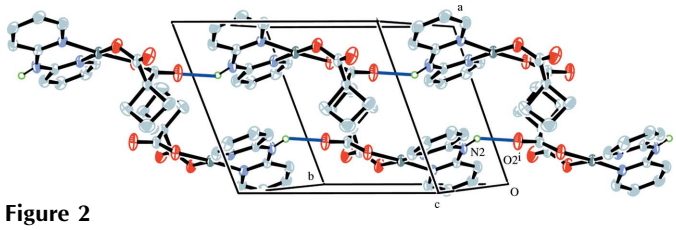


Figure 2
A view of the hydrogen bonding (blue lines) between the complex molecules. H atoms not involved in hydrogen bonding have been omitted. [Symmetry code: (i) *x*, *y* − 1, *z*.]

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.702, T_{max} = 0.882
7482 measured reflections
3425 independent reflections
3244 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.013
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.074
S = 1.27
3425 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.0721P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—O1	2.0169 (19)	Pd1—N1	2.007 (2)
Pd1—O4	2.001 (2)	Pd1—N3	2.012 (2)
O1—Pd1—O4	89.60 (9)	O4—Pd1—N1	88.71 (10)
O1—Pd1—N1	176.60 (7)	O4—Pd1—N3	177.47 (7)
O1—Pd1—N3	90.90 (9)	N1—Pd1—N3	90.92 (10)

H atoms were included in the riding-model approximation, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C). One C atom, C14, in the cyclobutane ring of the cbdca ligand was disordered over two positions, and was refined as C14*A* and C14*B* with site-occupancy factors of 0.5.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2005) and *CRYSTALS* (Betteridge *et al.*, 2003); 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: *CrystalStructure*.

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