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Bis[2-oxopyridine *N*-oxide(1−)-κ^2O,O']palladium(II)

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In the title palladium complex, [Pd($C_5H_4NO_2$)$_2$] or [Pd(opo)$_2$] [opo = 2-oxopyridine N-oxide(1−)], the Pd atom is in a square-planar environment of two opo ligands in a *trans* configuration.

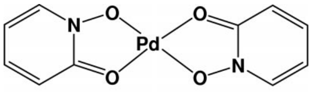
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
R factor = 0.038
wR factor = 0.080
Data-to-parameter ratio = 13.4

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

Comment

Pyridine *N*-oxide and its derivatives are a group of interesting ligands since a large number of metal complexes have been reported in the past decades (Landers & Phillips, 1982; Casellato *et al.*, 1983; Scarrow *et al.*, 1985). However, the chelating derivatives of pyridine *N*-oxide with donor atoms in the 2-position, such as *o*-mercaptopyridine *N*-oxide (Chen *et al.*, 1991; Shi *et al.*, 1997; Lobaba & Verma, 1999) and 2-hydroxypyridine *N*-oxide (Hopo) (Newbold & Spring, 1948; Hubbard *et al.*, 1979; Casellato *et al.*, 1983; Scarrow *et al.*, 1985; Farkas *et al.*, 2000), have been scarcely studied. Although some reports have appeared on the synthesis and chacterization of metal complexes with 2-hydroxypyridine *N*-oxide (Newbold & Spring, 1948), little work has been carried out on X-ray crystal structure determination (Casellato *et al.*, 1983; Riley *et al.*, 1983; Scarrow *et al.*, 1985). We report here the X-ray crystal structure of Pd(opo)$_2$, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The Pd(opo)$_2$ molecule has a regular square-planar geometry around the central Pd atom coordinated by two opo ligands. Two *N*-oxide O atoms are in a *trans* configuration. The whole molecule is nearly planar, with a r.m.s. deviation of 0.02 (1) Å for atoms O1, O2, O3, O4, Pd1. The average Pd—O(*N*-oxide) bond distance in (I) of 1.978 (3) Å is shorter than that reported for Pd(mpo)$_2$ (2.043 Å; Hmpo = *o*-mercapto pyridine *N*-oxide; Shi *et al.*, 1997). The average Pd—O(C═O) bond distance of 1.992 (3) Å is shorter than those in the tetra-

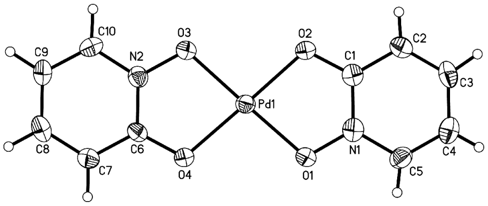


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

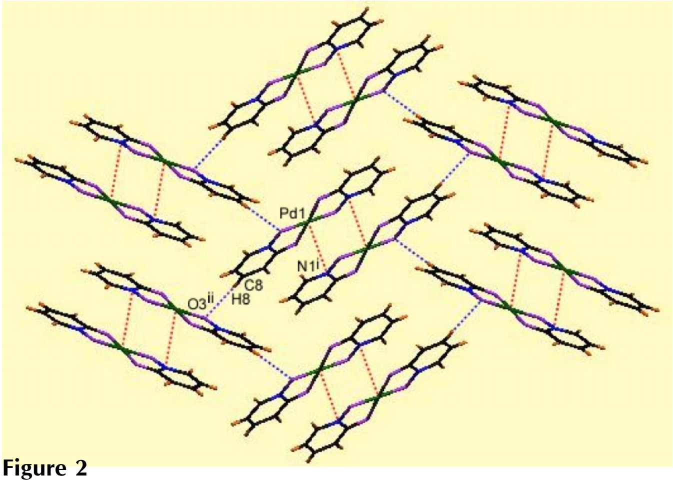


Figure 2
The extended layer structure of (I) [symmetry codes: (i) $2 - x, 1 - y, -z$; (ii) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$].

chlorocatechol complex $[Pd(C_6Cl_4O_2)(PPh_3)_2]$ [average = 2.033 (3) Å; Pierpont & Downs, 1975] and close to those reported for $[Pd(PhC(O)CC(O)Me)_2]$ (1.98 Å; Butler *et al.*, 1998). The weak interactions Pd1$\cdots$N1($2 - x$, $1 - y$, $-z$) [3.572 (4) Å] and C8—H8$\cdots$O3($1 - x$, $y - \frac{1}{2}$, $\frac{1}{2} - z$) connect neighboring molecules into a two-dimensional network (Fig. 2).

Experimental

To a solution of Hopo (0.22 g, 2.0 mmol) and $NaOCH_3$ (0.11 g, 2.0 mmol) in 60 ml of CH_3OH/CH_3CN (2:1) mixed solvent was added $PdCl_2$ (0.17 g, 1.0 mmol) and the mixture was refluxed overnight. After removing the insoluble solids by filtration, the filtrate was left to stand at room temperature for several days to give brown block-shaped crystals of $Pd(opo)_2$ in a yield of 40%. Analysis calculated for $C_{10}H_8N_2O_4Pd$: C 36.77, H 2.47, N 8.58%; found: C 37.10, H 2.75, N 8.50%. IR (KBr, cm^{-1}): 3090 (*w*), 1618 (*m*, C—O), 1551 (*s*), 1495 (*s*), 1438 (*w*), 1348 (*s*), 1246 (*m*), 1166 (*s*, N—O), 1143 (*w*), 1108 (*w*), 875 (*w*), 782 (*s*), 749 (*s*), 698 (*w*), 643 (*s*), 557 (*w*), 536 (*w*).

Crystal data

$[Pd(C_5H_4NO_2)_2]$
$M_r = 326.58$
Monoclinic, $P2_1/c$
$a = 6.937$ (2) Å
$b = 8.674$ (2) Å
$c = 17.491$ (4) Å
$\beta = 94.94$ (1)°
$V = 1048.6$ (5) Å^3
$Z = 4$

$D_x = 2.069$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 789 reflections
$\theta = 3.4$–23.6°
$\mu = 1.77$ mm^{-1}
$T = 298$ (2) K
Block, brown
0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.664$, $T_{max} = 0.765$
4617 measured reflections

2065 independent reflections
1403 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 26.0°$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -21 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.080$
$S = 1.05$
2065 reflections
154 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.87$ e Å^{-3}
$\Delta\rho_{min} = -0.77$ e Å^{-3}

Table 1
Selected geometric parameters (Å).

Pd1—O3	1.974 (3)	Pd1—O1	1.982 (4)
Pd1—O2	1.981 (4)	Pd1—O4	1.997 (4)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8—H8$\cdots$O3^{ii}	0.92	2.53	3.391 (7)	155

Symmetry code: (ii) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

All H atoms were positioned geometrically (C—H = 0.86–0.96 Å), with $U_{iso} = 1.2U_{eq}$ of the parent atom

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *MERCURY* (Version 1.2.1; Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXTL*.

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