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Dichloro[2,2′-hydroxy(ethoxy)methylenedipyridine-κ^2N,N']palladium(II)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.032
wR factor = 0.078
Data-to-parameter ratio = 14.4

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

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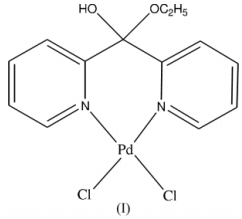
In the title complex, $[PdCl_2(C_{13}H_{14}N_2O_2)]$, the central Pd^{II} atom is bonded to two pyridine N atoms and two terminal Cl atoms. The coordination geometry of the Pd atom is square planar with a slight tetrahedral distortion. The two Pd—N distances are 2.029 (3) and 2.057 (3) Å, and the N—Pd—N angle is 86.56 (13)°. The Pd—Cl distances are 2.2929 (11) and 2.2959 (11) Å, and the Cl—Pd—Cl angle is 90.84 (5)°.

Comment

The condensation products formed when metal complexes of di-2-pyridyl ketone (dpk) are reacted with some nucleophiles, including water, have been reported previously (Annibale *et al.*, 1981; Sommerer *et al.*, 1997). Herein we report the structure of the title compound, (I), which is formed *via* the reaction of dpk with $PdCl_2$ in CH_3CN/CH_3CH_2OH. In (I), the N1—Pd—N2 angle is narrower than the ideal value (for square-planar coordination) of 90° [86.56 (13)°]. This angle can be compared with corresponding angles in [Pd{dpk-$(OH)_2$}]Cl_2 and [Pd(2,2′-dipyridyl)]Cl_2, where the N—Pd—N angles are 87.1 (2) (Annibale *et al.*, 1981) and 80.5 (4)° (Beer *et al.*, 1997), respectively. The N2—Pd—N1 [86.56 (13)°], N2—Pd—Cl2 [90.71 (9)°], N1—Pd—Cl1 [91.91 (10)°] and Cl1—Pd—Cl2 [90.84 (5)°] angles approach the ideal value of 90°; this indicates that the coordination geometry of the Pd atom is square planar with a slight tetrahedral distortion.



(I)

Experimental

For the preparation of compound (I), *p*-phenylenediamine was combined with dpk in a 1:2 stoichiometric ratio in CH_3CN/CH_3CH_2OH, followed by addition of $PdCl_2$. The resulting solution was filtered and slow evaporation of the clear filtrate gave clear yellow crystals, suitable for X-ray diffraction studies (yield: 0.07 g, 75%). Analysis calculated for $C_{13}H_{14}Cl_2N_2O_2Pd$: C 38.43, H 3.48, N 6.90%; found: C 38.23, H 3.09, N 6.68%.

Crystal data

$[PdCl_2(C_{13}H_{14}N_2O_2)]$
M_r = 407.56
Monoclinic, $P2_1/n$
a = 9.8277 (1) Å
b = 12.0717 (3) Å
c = 12.6885 (1) Å
β = 93.162 (2)°
V = 1503.03 (2) Å^3
Z = 4
D_x = 1.801 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3244 reflections
θ = 2.3–25.0°
μ = 1.59 mm^{-1}
T = 293 (2) K
Column, yellow
0.36 × 0.22 × 0.20 mm

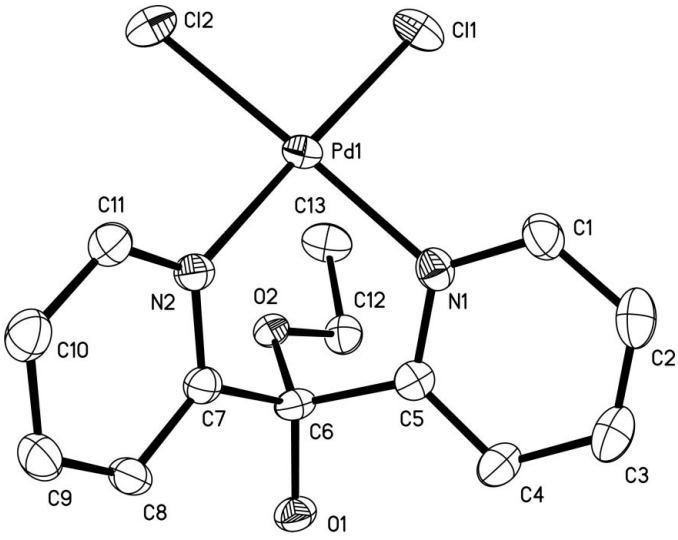


Figure 1
A view of the structure of the title compound, showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

Data collection

Siemens SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.558, T_{max} = 0.727
4510 measured reflections
2600 independent reflections
2270 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.0°
$h = -9 \rightarrow 11$
$k = -9 \rightarrow 14$
$l = -15 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.078
S = 1.15
2600 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0225P)^2 + 2.9262P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—N2	2.029 (3)	Pd1—Cl1	2.2929 (11)
Pd1—N1	2.057 (3)	Pd1—Cl2	2.2959 (11)
N2—Pd1—N1	86.56 (13)	N1—Pd1—Cl2	176.93 (10)
N2—Pd1—Cl1	178.32 (10)	Cl1—Pd1—Cl2	90.84 (5)
N1—Pd1—Cl1	91.91 (10)	C11—N2—Pd1	122.0 (3)
N2—Pd1—Cl2	90.71 (9)	C7—N2—Pd1	119.1 (3)

H atoms were included in calculated positions and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SHELXTL* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Annibale, G., Canovese, L., Cattalini, L., Natile, G., Biagini-Cingi, M., Manotti-lanfredi, A. & Tiripicchio, A. (1981). *J. Chem. Soc. Dalton Trans.* pp. 2280–2287.
Beer, P. D., Fletcher, N. C., Drew, M. G. B. & Wear, T. J. (1997). *Polyhedron*, **16**, 815–823.
Bruker (1999). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXL*97. University of Göttingen, Germany.
Siemens (1994). *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sommerer, S. O., Jircitano, A. J., Westcott, B. L., Abboud, K. A. & Krause Bauer, J. A. (1997). *Acta Cryst.* C**53**, 707–710.