Crystal structure of *trans*-bis(nitrato-κ*O*)bis(4-phenylpyridine-κ*N*)palladium(II), $Pd(NO_3)_2(C_{11}H_9N)_2$, $C_{22}H_{18}N_4O_6Pd$

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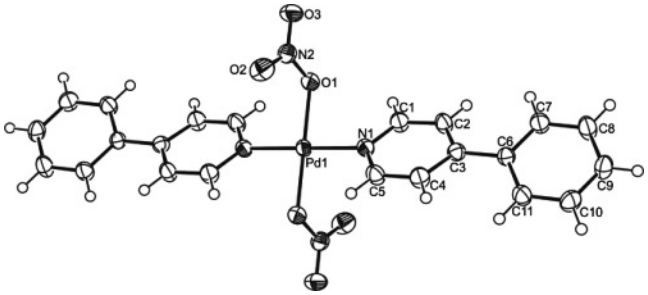

Abstract

$C_{22}H_{18}N_4O_6Pd$, monoclinic, *C*2/*c* (no. 15), $a = 20.336(3)$ Å, $b = 8.424(1)$ Å, $c = 15.024(2)$ Å, $\beta = 127.673(2)°$, $V = 2037.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0188$, $wR_{ref}(F^2) = 0.0526$, $T = 200$ K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.24×0.26×0.39 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.62 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω
$2\theta_{max}$:	52.1°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5884, 1901
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1695
$N(param)_{refined}$:	151
Programs:	SHELX [4], ORTEP-3 [5], PLATON [6]

Source of material

To a solution of $Pd(NO_3)_2{\cdot}2H_2O$ (0.130 g, 0.49 mmol) in acetone (30 ml) was added 4-phenylpyridine (*ppy*; 0.161 g, 1.04 mmol) and stirred for 7 h at room temperature. The formed precipitate was separated by filtration, washed with ether, and dried under vacuum, to give a yellow powder (0.19 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a CH_3NO_2 solution.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their respective parent atoms with d(C–H) = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. A completeness of only 94% has been achieved.

Discussion

The crystal structures of related dihalogenido platinum(II) complexes has been investigated previously [1, 2]. In the title complex $[Pd(NO_3)_2(ppy)_2]$, the central Pd(II) ion is four-coordinated by two N atoms from two *ppy* ligands and two O atoms of two nitrato ligands in a slightly distorted square-planar manner. The Pd atom is located on an inversion center, and thus the asymmetric unit contains one half of the complex and the PdN_2O_2 moiety is exactly planar. The dihedral angle between the PdN_2O_2 unit plane and the nearly planar pyridine ring is 36.9(1)°. In the crystal, the *ppy* ligand is not planar, the dihedral angle between the pyridine and phenyl ring is 17.9(1)°. The Pd–O and Pd–N bond lengths are nearly equal with d(Pd–O) = 2.0116(13) Å and d(Pd–N) = 2.0267(15) Å. The complex molecules are stacked in columns along [001]. In the columns, several intermolecular π-π interactions between the aromatic rings are present. The shortest distance between Cg1 (the centroid of ring C6-C11) and Cg1^{i} (symmetry code i: $-x,-y,-z$) is 3.772(2) Å.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	8*f*	0.0935	0.7410	0.2651	0.040
H(2)	8*f*	0.0023	0.5609	0.1296	0.039
H(4)	8*f*	0.1628	0.2118	0.3266	0.040
H(5)	8*f*	0.2478	0.3993	0.4600	0.041
H(7)	8*f*	–0.0598	0.3958	–0.0035	0.042
H(8)	8*f*	–0.1553	0.2096	–0.1327	0.048
H(9)	8*f*	–0.1441	–0.0562	–0.0808	0.044
H(10)	8*f*	–0.0355	–0.1332	0.1018	0.046
H(11)	8*f*	0.0623	0.0497	0.2304	0.042

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd(1)	4*c*	$\frac{1}{4}$	$\frac{3}{4}$	$\frac{1}{2}$	0.0248(1)	0.0224(1)	0.0226(1)	–0.00146(7)	0.0107(1)	0.00009(7)
N(1)	8*f*	0.17806(9)	0.5878(2)	0.3774(1)	0.0272(8)	0.0270(8)	0.0245(8)	–0.0006(6)	0.0131(7)	0.0005(6)
C(1)	8*f*	0.1068(1)	0.6312(2)	0.2789(2)	0.034(1)	0.0244(9)	0.032(1)	0.0016(8)	0.0149(9)	0.0007(8)
C(2)	8*f*	0.0526(1)	0.5243(2)	0.1972(2)	0.029(1)	0.029(1)	0.026(1)	0.0015(8)	0.0098(8)	0.0004(8)
C(3)	8*f*	0.0705(1)	0.3625(2)	0.2124(2)	0.0283(9)	0.0263(9)	0.0265(9)	–0.0002(7)	0.0171(8)	–0.0003(7)
C(4)	8*f*	0.1461(1)	0.3199(2)	0.3127(2)	0.035(1)	0.0230(9)	0.034(1)	0.0033(8)	0.0161(9)	0.0008(8)
C(5)	8*f*	0.1969(1)	0.4326(2)	0.3918(2)	0.031(1)	0.030(1)	0.028(1)	0.0035(8)	0.0115(8)	0.0030(8)
C(6)	8*f*	0.0114(1)	0.2434(2)	0.1286(2)	0.025(1)	0.026(1)	0.026(1)	0.0004(6)	0.0149(9)	–0.0007(7)
C(7)	8*f*	–0.0543(1)	0.2880(2)	0.0186(2)	0.036(1)	0.0278(9)	0.031(1)	0.0000(9)	0.0148(9)	0.0011(8)
C(8)	8*f*	–0.1112(1)	0.1772(2)	–0.0583(2)	0.034(1)	0.034(1)	0.030(1)	–0.0007(9)	0.0092(9)	–0.0028(9)
C(9)	8*f*	–0.1047(1)	0.0197(2)	–0.0280(2)	0.034(1)	0.030(1)	0.038(1)	–0.0063(8)	0.0177(9)	–0.0079(8)
C(10)	8*f*	–0.0403(1)	–0.0254(2)	0.0799(2)	0.042(1)	0.0244(9)	0.042(1)	–0.0018(8)	0.022(1)	0.0007(8)
C(11)	8*f*	0.0176(1)	0.0839(2)	0.1569(2)	0.034(1)	0.031(1)	0.029(1)	–0.0004(8)	0.0133(9)	0.0016(8)
N(2)	8*f*	0.2812(1)	0.9306(2)	0.3693(1)	0.0394(9)	0.0253(8)	0.0313(8)	–0.0060(7)	0.0204(8)	–0.0045(7)
O(1)	8*f*	0.22282(8)	0.9093(2)	0.3821(1)	0.0312(7)	0.0316(7)	0.0286(7)	–0.0009(6)	0.0144(6)	0.0043(6)
O(2)	8*f*	0.34690(9)	0.8576(2)	0.4309(1)	0.0428(8)	0.0391(8)	0.058(1)	0.0082(7)	0.0329(8)	0.0096(7)
O(3)	8*f*	0.2650(1)	1.0252(2)	0.2972(1)	0.0570(9)	0.0390(8)	0.0366(8)	–0.0057(7)	0.0273(7)	0.0072(7)

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