

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Chlorido[4-(pyridin-2-yl- κ N)pyrimidine-2-sulfonato- κ^2 N³,O]palladium(II)Hai-Bin Zhu^{a*} and Xian-Shan Hou^b

^aSchool of Chemistry and Chemical Engineering, Southeast University, Nanjing 211189, People's Republic of China, and ^bJiangsu Hengrui Pharmaceutical Company, Lianyungang 222002, People's Republic of China
Correspondence e-mail: zhuhabin@seu.edu.cn

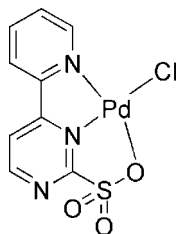
Received 20 November 2010; accepted 24 November 2010

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.021; wR factor = 0.053; data-to-parameter ratio = 14.2.

In the title compound, $[\text{Pd}(\text{C}_9\text{H}_6\text{N}_3\text{O}_3\text{S})\text{Cl}]$, the Pd^{II} ion is coordinated by one O and two N atoms from a 4-(pyridin-2-yl)pyrimidine-2-sulfonate ligand and one chloride anion in a distorted square-planar geometry. In the crystal, all molecules are situated on mirror planes and interact through weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For antitumor drugs with platinum, see: Wong (1999). For recent advances in developing of antitumor palladium-based coordination compounds, see: Caires (2007).



Experimental

Crystal data

 $[\text{Pd}(\text{C}_9\text{H}_6\text{N}_3\text{O}_3\text{S})\text{Cl}]$ $M_r = 378.09$ Orthorhombic, $Pnma$ $a = 15.4598$ (16) Å $b = 6.5974$ (7) Å $c = 11.0844$ (12) Å $V = 1130.5$ (2) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 2.06$ mm⁻¹ $T = 298$ K $0.19 \times 0.15 \times 0.12$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

 $T_{\text{min}} = 0.697$, $T_{\text{max}} = 0.781$

9511 measured reflections

1522 independent reflections

1433 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.020$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.021$ $wR(F^2) = 0.053$ $S = 1.06$

1522 reflections

107 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.60$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.61$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C8}-\text{H8A}\cdots\text{O1}^{\text{i}}$	0.93	2.48	3.379 (4)	164
$\text{C7}-\text{H7A}\cdots\text{O2}^{\text{ii}}$	0.93	2.60	3.238 (3)	127
$\text{C7}-\text{H7A}\cdots\text{O2}^{\text{iii}}$	0.93	2.60	3.238 (3)	127

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + \frac{1}{2}, -y, z + \frac{1}{2}$; (iii) $-x + \frac{1}{2}, y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT-Plus (Bruker, 2007); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

The authors acknowledge financial support from the China Postdoctoral Research Fund (20070411010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5004).

References

- Bruker (2001). SADABS Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). APEX2 and SAINT-Plus. Bruker AXS Inc., Madison, Wisconsin, USA.
Caires, A. C. F. (2007). *Anti-Cancer Agents Med. Chem.* **7**, 484–498.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Wong, E. & Giandomenico, C. M. (1999). *Chem. Rev.* **99**, 2451–2466.

supplementary materials

Acta Cryst. (2010). E66, m1685 [doi:10.1107/S1600536810049184]

Chlorido[4-(pyridin-2-yl- κ N)pyrimidine-2-sulfonato- κ^2 N³,O]palladium(II)

H.-B. Zhu and X.-S. Hou

Comment

In order to overcome the drawbacks of antitumor platinum drugs in clinical treatment (Wong *et al.*, 1999), design and screening of anticancer palladium-based coordination compounds have been actively pursued in recent years (Caires, 2007). In this paper, we report a new palladium (II) coordination compound based on **2-ppsa** ligand (**2-ppsa** = 4-(pyridin-2-yl)pyrimidine-2-sulfonate).

In the title compound (Fig. 1), each palladium(II) atom in a distorted square-planar environment is coordinated by one O and two N atoms, and one chloro anion (Pd1—N1 = 2.002 (2) Å; Pd1—N2 = 1.947 (2) Å; Pd1—O1 = 2.081 (2) Å; Pd1—Cl1 = 2.2918 (7) Å). **2-ppsa** ligand offers two N atoms and one sulfonato O atom in NNO-chelation manner (N1—Pd1—N2 80.8 (1)°; N2—Pd1—O1 83.60 (9)°). In sulfonato group, the S1—O1 bond distance (1.494 (2) Å) is slightly longer than that for S1—O2(O2ⁱ) bond (1.428 (2) Å) due to the O1—Pd1 coordination. Weak C—H···O hydrogen bonds (Table 1) are involved into intermolecular interactions.

Experimental

The CH₃CN solution of PdCl₂ (0.1 mmol) was layered above the aqueous solution of **2-ppsa** sodium salt (0.1 mmol). Orange crystals suitable for X-ray diffraction analysis were obtained after one week.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

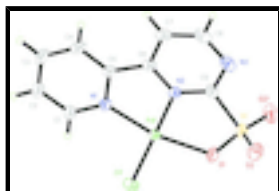


Fig. 1. The molecular structure of the title compound showing the atomic numbering and 30% probability displacement ellipsoids [symmetry code: (i) $x, -y + 1/2, z$].

Chlorido[4-(pyridin-2-yl- κ N)pyrimidine-2-sulfonato- κ^2 N³,O]palladium(II)

Crystal data

[Pd(C₉H₆N₃O₃S)Cl]

$F(000) = 736$

supplementary materials

$$M_r = 378.09$$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$$a = 15.4598 (16) \text{ \AA}$$

$$b = 6.5974 (7) \text{ \AA}$$

$$c = 11.0844 (12) \text{ \AA}$$

$$V = 1130.5 (2) \text{ \AA}^3$$

$$Z = 4$$

$$D_x = 2.221 \text{ Mg m}^{-3}$$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1522 reflections

$$\theta = 2.3\text{--}25.5^\circ$$

$$\mu = 2.06 \text{ mm}^{-1}$$

$$T = 298 \text{ K}$$

Block, orange

$$0.19 \times 0.15 \times 0.12 \text{ mm}$$

Data collection

Bruker APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube
graphite

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2001)

$$T_{\min} = 0.697, T_{\max} = 0.781$$

9511 measured reflections

1522 independent reflections

1433 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.020$$

$$\theta_{\max} = 28.3^\circ, \theta_{\min} = 2.3^\circ$$

$$h = -20 \rightarrow 20$$

$$k = -8 \rightarrow 8$$

$$l = -12 \rightarrow 14$$

Refinement

Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.021$$

$$wR(F^2) = 0.053$$

$$S = 1.06$$

1522 reflections

107 parameters

0 restraints

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0313P)^2 + 0.4917P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.002$$

$$\Delta\rho_{\max} = 0.60 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\min} = -0.61 \text{ e \AA}^{-3}$$

Extinction correction: *SHELXL97* (Sheldrick, 2008),

$$F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.0057 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	0.026756 (12)	0.2500	0.969638 (16)	0.02762 (9)
Cl1	-0.11601 (4)	0.2500	1.02532 (7)	0.04094 (18)
S1	0.08397 (5)	0.2500	0.70360 (6)	0.03980 (17)
N1	0.08013 (14)	0.2500	1.1342 (2)	0.0307 (4)
N2	0.14834 (13)	0.2500	0.9239 (2)	0.0296 (4)
C6	0.20748 (17)	0.2500	1.0130 (3)	0.0326 (5)
C5	0.16952 (17)	0.2500	1.1331 (3)	0.0333 (6)
C9	0.17219 (18)	0.2500	0.8096 (3)	0.0359 (6)
N3	0.25317 (17)	0.2500	0.7706 (3)	0.0472 (6)
C7	0.29440 (19)	0.2500	0.9801 (3)	0.0446 (7)
H7A	0.3382	0.2500	1.0376	0.053*
C4	0.2151 (2)	0.2500	1.2392 (3)	0.0443 (7)
H4A	0.2753	0.2500	1.2380	0.053*
C1	0.0393 (2)	0.2500	1.2392 (3)	0.0410 (7)
H1A	-0.0209	0.2500	1.2396	0.049*
C8	0.3128 (2)	0.2500	0.8575 (4)	0.0517 (8)
H8A	0.3706	0.2500	0.8344	0.062*
C3	0.1715 (2)	0.2500	1.3479 (3)	0.0509 (8)
H3B	0.2019	0.2500	1.4203	0.061*
C2	0.0826 (2)	0.2500	1.3478 (3)	0.0507 (8)
H2A	0.0521	0.2500	1.4201	0.061*
O1	0.00639 (14)	0.2500	0.78404 (18)	0.0474 (6)
O2	0.09105 (11)	0.0660 (3)	0.63642 (15)	0.0575 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.02287 (12)	0.03579 (13)	0.02421 (12)	0.000	-0.00022 (6)	0.000
Cl1	0.0254 (3)	0.0465 (4)	0.0508 (5)	0.000	0.0048 (3)	0.000
S1	0.0425 (4)	0.0520 (4)	0.0249 (3)	0.000	0.0033 (3)	0.000
N1	0.0315 (11)	0.0333 (11)	0.0273 (10)	0.000	-0.0022 (9)	0.000
N2	0.0256 (10)	0.0309 (11)	0.0322 (11)	0.000	-0.0004 (9)	0.000
C6	0.0267 (12)	0.0322 (13)	0.0389 (14)	0.000	-0.0033 (11)	0.000
C5	0.0319 (12)	0.0324 (13)	0.0356 (14)	0.000	-0.0052 (11)	0.000
C9	0.0347 (13)	0.0384 (14)	0.0347 (14)	0.000	0.0072 (11)	0.000
N3	0.0373 (13)	0.0543 (16)	0.0499 (16)	0.000	0.0166 (12)	0.000
C7	0.0256 (14)	0.0476 (17)	0.060 (2)	0.000	-0.0022 (13)	0.000
C4	0.0425 (16)	0.0455 (16)	0.0449 (17)	0.000	-0.0164 (14)	0.000
C1	0.0433 (16)	0.0487 (17)	0.0309 (14)	0.000	0.0010 (12)	0.000
C8	0.0294 (14)	0.058 (2)	0.068 (2)	0.000	0.0110 (15)	0.000
C3	0.063 (2)	0.0545 (19)	0.0348 (16)	0.000	-0.0199 (16)	0.000
C2	0.063 (2)	0.060 (2)	0.0291 (15)	0.000	-0.0005 (15)	0.000
O1	0.0333 (10)	0.0831 (17)	0.0257 (10)	0.000	-0.0025 (9)	0.000
O2	0.0666 (10)	0.0630 (11)	0.0430 (8)	-0.0049 (8)	0.0037 (8)	-0.0151 (8)

supplementary materials

Geometric parameters (\AA , $^\circ$)

Pd1—N2	1.947 (2)	C5—C4	1.372 (4)
Pd1—N1	2.002 (2)	C9—N3	1.324 (4)
Pd1—O1	2.081 (2)	N3—C8	1.333 (5)
Pd1—Cl1	2.2918 (7)	C7—C8	1.388 (5)
S1—O2	1.4282 (17)	C7—H7A	0.9300
S1—O2 ⁱ	1.4282 (17)	C4—C3	1.381 (5)
S1—O1	1.494 (2)	C4—H4A	0.9300
S1—C9	1.800 (3)	C1—C2	1.378 (5)
N1—C1	1.324 (4)	C1—H1A	0.9300
N1—C5	1.382 (3)	C8—H8A	0.9300
N2—C9	1.319 (4)	C3—C2	1.374 (5)
N2—C6	1.346 (4)	C3—H3B	0.9300
C6—C7	1.392 (4)	C2—H2A	0.9300
C6—C5	1.455 (4)		
N2—Pd1—N1	80.76 (10)	N2—C9—N3	125.3 (3)
N2—Pd1—O1	83.60 (9)	N2—C9—S1	114.5 (2)
N1—Pd1—O1	164.36 (9)	N3—C9—S1	120.2 (2)
N2—Pd1—Cl1	179.48 (7)	C9—N3—C8	114.7 (3)
N1—Pd1—Cl1	98.72 (7)	C8—C7—C6	117.0 (3)
O1—Pd1—Cl1	96.92 (6)	C8—C7—H7A	121.5
O2—S1—O2 ⁱ	116.39 (15)	C6—C7—H7A	121.5
O2—S1—O1	111.87 (8)	C5—C4—C3	119.8 (3)
O2 ⁱ —S1—O1	111.87 (8)	C5—C4—H4A	120.1
O2—S1—C9	106.40 (9)	C3—C4—H4A	120.1
O2 ⁱ —S1—C9	106.40 (9)	N1—C1—C2	122.4 (3)
O1—S1—C9	102.63 (12)	N1—C1—H1A	118.8
C1—N1—C5	119.0 (2)	C2—C1—H1A	118.8
C1—N1—Pd1	127.17 (19)	N3—C8—C7	124.5 (3)
C5—N1—Pd1	113.83 (18)	N3—C8—H8A	117.8
C9—N2—C6	121.0 (2)	C7—C8—H8A	117.8
C9—N2—Pd1	121.33 (19)	C2—C3—C4	119.2 (3)
C6—N2—Pd1	117.69 (19)	C2—C3—H3B	120.4
N2—C6—C7	117.6 (3)	C4—C3—H3B	120.4
N2—C6—C5	113.4 (2)	C3—C2—C1	119.1 (3)
C7—C6—C5	129.0 (3)	C3—C2—H2A	120.4
C4—C5—N1	120.4 (3)	C1—C2—H2A	120.4
C4—C5—C6	125.3 (3)	S1—O1—Pd1	117.92 (12)
N1—C5—C6	114.3 (2)		

Symmetry codes: (i) $x, -y+1/2, z$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8—H8A \cdots O1 ⁱⁱ	0.93	2.48	3.379 (4)	164
C7—H7A \cdots O2 ⁱⁱⁱ	0.93	2.60	3.238 (3)	127

C7—H7A \cdots O2^{iv}

0.93

2.60

3.238 (3)

127

Symmetry codes: (ii) $x+1/2, -y+1/2, -z+3/2$; (iii) $-x+1/2, -y, z+1/2$; (iv) $-x+1/2, y+1/2, z+1/2$.

Fig. 1

