Crystal structure of bis(3-phenylpyridine-κ*N*)bis(thiocyanato-κ*S*) palladium(II), $C_{24}H_{18}N_4PdS_2$

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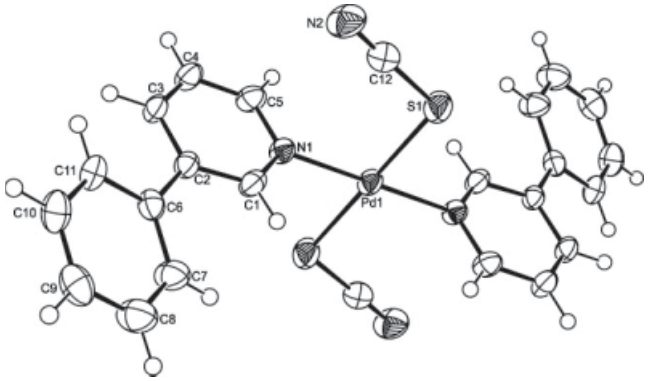

Abstract

$C_{24}H_{18}N_4PdS_2$, monoclinic, $P2_1/n$ (no. 14), a = 9.954(2) Å, b = 5.257(1) Å, c = 21.566(4) Å, β = 102.691(4)°, V = 1101.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.0343, $wR_{ref}(F^2)$ = 0.0892, T = 200 K.

Table 1. Data collection and handling.

Crystal:	yellow sticks, size 0.06×0.10×0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.52 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω
$2\theta_{max}$:	52.22°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6509, 2164
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1532
$N(param)_{refined}$:	142
Programs:	SHELX [2], ORTEP-3[3], PLATON [4]

Source of material

To a solution of Na_2PdCl_4 (0.1420 g, 0.483 mmol) in MeOH (30 ml) / H_2O (20 ml) were added KSCN (0.5094 g, 5.242 mmol) and 3-phenylpyridine (0.1778 g, 1.146 mmol) and stirred for 24 h at room temperature. The formed precipitate was separated by filtration, washed with H_2O and MeOH, and dried at 50 °C, to give a yellow powder (0.2188 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a CH_3CN solution.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C–H) = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C). The highest peak (1.16 e·Å^{-3}) and the deepest hole (-0.77 e·Å^{-3}) in the difference Fourier map are located 1.99 Å and 0.92 Å from the atoms H1 and Pd1, respectively.

Discussion

Crystal structure of the related chlorido-Pd(II) complex $[PdCl_2(C_{11}H_9N)_2]$ has been investigated previously [1]. The central Pd(II) ion in the title complex $[Pd(SCN)_2(C_{11}H_9N)_2]$ has a *trans*-S_2N_2 square-planar coordination geometry defined by two N atoms from two 3-phenylpyridine ligands and two S atoms of two SCN^- ligands. The Pd atom is located on an inversion center, and thus the asymmetric unit contains one half of the complex and the PdS_2N_2 moiety is exactly planar. The dihedral angle between the PdS_2N_2 moiety and the nearly planar pyridine ring is 85.17(9)°. In the crystal, the 3-phenylpyridine ligand is not planar, the dihedral angle between the pyridine and phenyl rings being 20.3(2)°. The thiocyanato ligand is almost linear displaying a S–C–N bond angle of 178.5(4)°. The S atoms are coordinated to the Pd atom with the nearly tetrahedral Pd–S–C bond angle of 105.4(2)°. The complex molecules are stacked in columns along [010]. In the columns, several intermolecular π-π interactions between the aromatic rings may be present. The shortest distance between Cg1 (the centroid of ring N1-C5) and Cg2^i (the centroid of ring C6-C11, symmetry code i: x,y-1,z) is 3.778(2) Å, and the dihedral angle between the ring planes is 20.3(2)°.

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.2430	0.3246	0.0500	0.036
H(3)	4*e*	0.4359	0.0577	0.2215	0.036
H(4)	4*e*	0.2637	–0.2446	0.2141	0.043
H(5)	4*e*	0.0870	–0.2599	0.1243	0.04
H(7)	4*e*	0.4339	0.4972	0.0442	0.049
H(8)	4*e*	0.5994	0.8158	0.0509	0.059
H(9)	4*e*	0.7373	0.9302	0.1491	0.052
H(10)	4*e*	0.7013	0.7358	0.2407	0.05
H(11)	4*e*	0.5360	0.4242	0.2355	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)	2*a*	0	0	0	0.0285(3)	0.0319(3)	0.0226(2)	–0.0045(2)	0.0007(2)	–0.0001(2)
S(1)	4*e*	–0.1565(1)	0.2435(2)	0.04058(5)	0.0390(6)	0.0530(7)	0.0301(6)	0.0078(6)	0.0017(4)	–0.0046(5)
N(1)	4*e*	0.1522(3)	0.0265(6)	0.0790(1)	0.027(2)	0.029(2)	0.023(2)	–0.006(2)	0.002(1)	–0.001(2)
N(2)	4*e*	–0.0240(4)	0.3632(8)	0.1667(2)	0.057(3)	0.061(3)	0.032(2)	–0.010(2)	0.008(2)	–0.012(2)
C(1)	4*e*	0.2495(4)	0.2063(8)	0.0838(2)	0.036(2)	0.033(2)	0.021(2)	0.006(2)	0.005(2)	0.002(2)
C(2)	4*e*	0.3599(4)	0.2279(7)	0.1361(2)	0.027(2)	0.029(2)	0.022(2)	0.004(2)	0.003(2)	–0.002(2)
C(3)	4*e*	0.3628(4)	0.0525(7)	0.1848(2)	0.030(2)	0.038(3)	0.020(2)	0.003(2)	–0.001(2)	0.000(2)
C(4)	4*e*	0.2616(4)	–0.1270(8)	0.1805(2)	0.041(3)	0.040(2)	0.027(2)	0.007(2)	0.005(2)	0.010(2)
C(5)	4*e*	0.1573(4)	–0.1356(8)	0.1271(2)	0.039(3)	0.031(2)	0.030(2)	–0.004(2)	0.009(2)	0.006(2)
C(6)	4*e*	0.4664(4)	0.4268(7)	0.1389(2)	0.025(2)	0.032(2)	0.031(2)	0.003(2)	0.004(2)	–0.004(2)
C(7)	4*e*	0.4877(4)	0.5453(8)	0.0845(2)	0.041(3)	0.053(3)	0.029(2)	–0.012(2)	0.009(2)	–0.006(2)
C(8)	4*e*	0.5871(5)	0.7340(9)	0.0885(2)	0.049(3)	0.056(3)	0.044(3)	–0.014(3)	0.016(2)	–0.003(2)
C(9)	4*e*	0.6681(4)	0.8038(9)	0.1466(2)	0.031(3)	0.042(3)	0.055(3)	–0.009(2)	0.006(2)	–0.013(2)
C(10)	4*e*	0.6469(4)	0.6874(8)	0.2005(2)	0.034(3)	0.041(3)	0.043(3)	–0.006(2)	–0.007(2)	–0.006(2)
C(11)	4*e*	0.5486(4)	0.5023(8)	0.1975(2)	0.032(2)	0.040(2)	0.028(2)	0.001(2)	–0.004(2)	0.001(2)
C(12)	4*e*	–0.0763(4)	0.3126(7)	0.1155(2)	0.039(3)	0.027(2)	0.038(3)	–0.003(2)	0.012(2)	–0.001(2)

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