Crystal structure of diaqua-dinitrato-κ^1*O*-bis(2-(pyridin-4-ylthio)-pyrazine-κ^1*N*)cadmium(II), $C_{18}H_{18}CdN_8O_8S_2$

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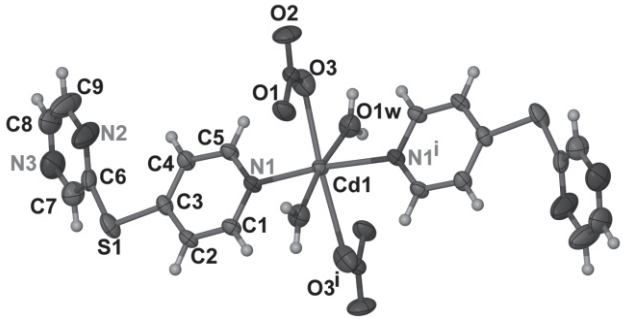

Abstract

$C_{18}H_{18}CdN_8O_8S_2$, monoclinic, $P2_1/c$ (no. 14), a = 12.9345(5) Å, b = 12.9120(5) Å, c = 7.2991(3) Å, β = 91.085(2)°, V = 1218.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.0401, $wR_{ref}(F^2)$ = 0.0939, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.4×0.4×0.55 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.29 cm^{-1}
Diffractometer, scan mode:	Bruker Smart ApexII CCD, φ and ω
$2\theta_{max}$:	55.89°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10893, 2876
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2040
$N(param)_{refined}$:	169
Programs:	SHELX [1], DIAMOND [2]

Source of material

All organic reagents and solvents purchased from commercial sources were used without further purification. Sodium pyridine-4-thiolate (0.66 g, 5 mmol) dissolved in methanol was added to 10 ml acetonictrile containing 2-chloro-pyrazine (0.58 g, 5 mmol) with stirring at room temperature. After 5 hours, the solvent were removed under vacuum. The products were then separated and purified by column chromatography on silica gel (eluent: dichloromethane/methanol = 95:5, *v*/*v*), and the di-[2-(pyridine-4-ylthio)pyrazine] was obtained in 33.1% yield. Di-[2-(pyridine-4-ylthio)pyrazine] (38 mg, 0.2 mmol) and $Cd(NO_3)_2{\cdot}6H_2O$ (345 mg, 0.1 mmol) were dissolved and mixed in acetonitrile, the filtrate of which was then left to stand in air to let the solvent evaporate slowly. The colourless block-shaped crystals of the title compound were deposited, yield 20.1 mg (31% based on di-[2-(pyridine-4-ylthio)pyrazine]).

Experimental details

All hydrogen atoms were added using riding models. The U_{iso} values of the hydrogen atoms were set to 1.2U_{eq}(C,N).

Discussion

Pyridyl-based ligands have been widely used to construct different transition complexes with fascinating structures and attractive properties [3–5]. Dipyridyl ligands with the bent C–S–C fragment exhibit diverse coordination geometries in construction various transition metal complexes [6–8], and the coordination chemistry of these N-positional isomers has been extensively studied. In the present context, we report a new complex derived from 2-(pyridine-4-ylthio)pyrazine and cadmium(II)nitrate. In the asymmetric unit of the title complex, there is one 2-(pyridine-4-ylthio)pyrazine, one water molecule, one nitrate anion and half of a Cd(II) ion. The Cd(II) lies in an inversion center and adopts an $N_2O_2(H_2O)_2$ octahedral geometry with two 4-pyridyl N locating at the axial positions (Fig.). The Cd1–N1 bond length is 2.249(3) Å, and the Cd1–O1w equals 2.402(3) Å. The C3–S1–C6 angle is 106.85(18)°, comparable to that 104.27(7)° of C–S–C in di-pyrazinyl sulfide [8].

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.5075	0.7389	–0.0230	0.065
H(2A)	4*e*	0.6119	0.8803	0.0022	0.069
H(4A)	4*e*	0.8512	0.6905	0.1034	0.071
H(5A)	4*e*	0.7393	0.5542	0.0767	0.065
H(7A)	4*e*	0.9435	0.9922	0.3349	0.109
H(8A)	4*e*	1.2005	0.8541	0.2928	0.185
H(9A)	4*e*	1.1351	0.7561	0.0658	0.240
H(1WA)	4*e*	0.5874	0.4143	0.3665	0.113
H(1WB)	4*e*	0.6320	0.3573	0.2364	0.113

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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}
Cd(1)	2*d*	½	½	0	0.0517(2)	0.0391(2)	0.0799(3)	–0.0020(2)	–0.0211(2)	–0.0026(2)
S(1)	4*e*	0.8134(1)	0.91853(8)	0.0701(2)	0.0967(8)	0.0513(6)	0.1086(9)	–0.0238(6)	–0.0289(7)	0.0072(6)
N(1)	4*e*	0.6125(2)	0.6323(2)	0.0232(4)	0.052(2)	0.037(1)	0.054(2)	0.001(1)	–0.013(1)	–0.001(1)
N(2)	4*e*	0.9945(4)	0.8166(5)	0.0574(9)	0.086(3)	0.142(5)	0.224(6)	–0.037(3)	0.046(4)	–0.096(5)
N(3)	4*e*	1.0817(4)	0.9346(5)	0.3351(8)	0.076(3)	0.127(4)	0.156(5)	–0.022(3)	–0.021(3)	–0.003(4)
C(1)	4*e*	0.5776(3)	0.7291(3)	0.0026(5)	0.056(2)	0.044(2)	0.061(2)	0.006(2)	–0.012(2)	–0.001(2)
C(2)	4*e*	0.6397(3)	0.8144(3)	0.0174(5)	0.072(2)	0.037(2)	0.063(2)	0.003(2)	–0.009(2)	0.001(2)
C(3)	4*e*	0.7443(3)	0.8020(3)	0.0551(5)	0.066(2)	0.044(2)	0.048(2)	–0.012(2)	–0.010(2)	0.001(1)
C(4)	4*e*	0.7815(3)	0.7022(3)	0.0775(5)	0.052(2)	0.055(2)	0.069(2)	–0.005(2)	–0.015(2)	0.005(2)
C(5)	4*e*	0.7137(3)	0.6210(3)	0.0609(5)	0.053(2)	0.041(2)	0.068(2)	0.004(2)	–0.013(2)	0.003(2)
C(6)	4*e*	0.9411(3)	0.8865(3)	0.1401(6)	0.070(3)	0.061(2)	0.088(3)	–0.025(2)	0.017(2)	–0.005(2)
C(7)	4*e*	0.9847(4)	0.9435(4)	0.2774(7)	0.078(3)	0.094(4)	0.099(4)	–0.012(3)	–0.009(3)	–0.009(3)
C(8)	4*e*	1.1323(5)	0.8651(6)	0.255(1)	0.063(4)	0.105(5)	0.29(1)	–0.026(4)	0.020(5)	–0.014(6)
C(9)	4*e*	1.0931(5)	0.8058(7)	0.119(2)	0.075(4)	0.155(7)	0.37(1)	–0.027(4)	0.058(6)	–0.122(9)
N(4)	4*e*	0.3672(2)	0.6192(2)	0.2815(4)	0.055(2)	0.042(2)	0.059(2)	0.000(1)	0.003(1)	–0.004(1)
O(1)	4*e*	0.4302(2)	0.5594(2)	0.3551(5)	0.068(2)	0.065(2)	0.135(3)	0.009(2)	–0.024(2)	0.014(2)
O(2)	4*e*	0.3213(3)	0.6821(3)	0.3708(5)	0.091(2)	0.102(2)	0.113(3)	0.027(2)	0.015(2)	–0.050(2)
O(3)	4*e*	0.3563(3)	0.6125(3)	0.1148(4)	0.143(3)	0.092(2)	0.052(2)	–0.009(2)	–0.001(2)	0.005(2)
O(1W)	4*e*	0.5898(2)	0.4115(2)	0.2449(4)	0.075(2)	0.090(2)	0.060(2)	–0.000(2)	–0.010(1)	0.011(1)

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