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Crystal structure of diaqua-bis[*N*-(2-pyridyl)carbonylaniline]copper(II) dinitrate, $Cu(C_{12}H_{16}N_2O)_2(H_2O)_2(NO_3)_2$

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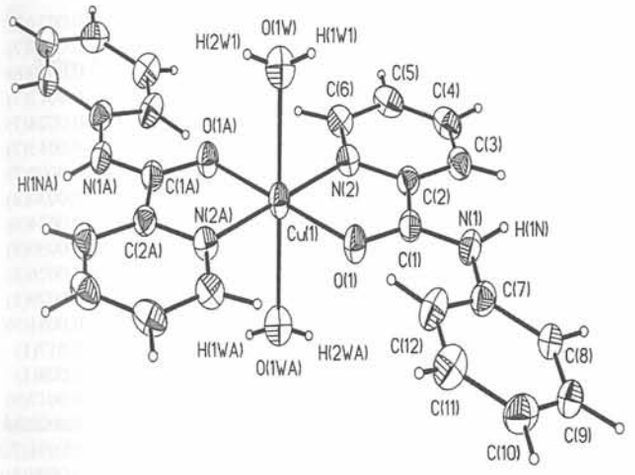

Abstract

$C_{24}H_{24}CuN_6O_{10}$, monoclinic, $P12_1/c1$ (No. 14), $a = 7.441(2)$ Å, $b = 11.458(2)$ Å, $c = 14.850(3)$ Å, $\beta = 95.941(5)°$, $V = 1259.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.049$, $wR_{ref}(F^2) = 0.126$, $T = 120$ K.

Source of material

N-(2-pyridyl)carbonylaniline was synthesized in accordance with published procedure [1]. The green crystals of the title compound were obtained from the reaction of a solution of *N*-(2-pyridyl)carbonylaniline (0.396 g, 2 mmol) in hot acetonitrile (5 ml) and a hot aqueous solution of copper(II)nitrate (0.188 g, 1 mmol) with *N*-(2-pyridyl)-carbonylaniline (0.200 g, 2 mmol) in 15 ml acetonitrile. The final solution was brought to the boil and allowed to cool slowly overnight, depositing green crystals (0.372 g, yield 60%; mp 563 K). The green single crystals suitable for X-rax analysis were obtained by slow diffusion (2 days) of diethyl ether in methanolic solution of title compound. Elemental analyses were consistent with the composition $C_{24}H_{24}CuN_6O_{10}$ (found: C, 45.38%; H, 3.75%; N, 13.41%; calc.: C, 46.45.%; H, 3.87.%; N, 13.54%).

Discussion

Numerous copper(II) complexes with nitrogen or oxygen donor ligands have been synthesized and studied. Some of these complexes serve as structural models for the active site in enzymes [2]. In the recent years the interest in the determination of X-ray crystal structures of biologically active compounds increased [3]. In this work, we describe the crystal structure of diaqua-bis[*N*-(2-pyridyl)-carbonylaniline]copper(II) dinitrate, $Cu(C_{12}H_{16}N_2O)_2(H_2O)_2(NO_3)_2$. The cationic complex is mononuclear and occupies special positions in the inversion centers. In the cation, the metal atom is coordinated to two *N*-(2-pyridyl)carbonylaniline (L), via one pyridine nitrogen and one carbonyl oxygen, and to two H_2O molecules. The *N*-(2-pyridyl)carbonylaniline (L) ligand behaves as a bidentate ligands, forming a five-membered metallcycle. The coordination of Cu is a distorted octahedral. The molecules have an inversion center with approximate C_{2h} symmetry and are formed by two bidentate *N*-(2-pyridyl)carbonylaniline ligands through their O and N atoms and two water molecules. Two oxygen atoms from amide and two nitrogen atoms from pyridyl are in *trans* positions [the angles ∠O(amide)–Cu–O(amide) and ∠N(pyridyl)–Cu–O(pyridyl) are 180°], and also the two coordinated water molecules are *trans* [∠O(water)–Cu–O(water) is 180°]. The bond distances Cu—N (1.977 Å) Cu—O(amide) (1.965 Å) and Cu—O(water) (2.405 Å) are consistent with previously described values [2]. The bite angles of ∠O1–Cu1–N2 and ∠O1#1–Cu1–N2#1 are 82.06° and 82.06°, respectively, being similar to previously reported [2]. The nitrate anions in this compound show rotational disorder over two positions with unequal occupancies. Cations are linked by hydrogen bonding. The coordinated *N*-(2-pyridyl)carbonylaniline (L) molecules and the two coordinated water molecules are involved in hydrogen bonding acting as hydrogen-bond donors with coordinated O and N atoms as potential hydrogen-bond acceptors. The hydrogen bonding yields infinite chains parallel to the crystallgraphic vectors *a* and *b*. Each cation is bonded to four neighbors and assembles the molecules into a one-dimensional chain.

Table 1. Data collection and handling.

Crystal:	deep green prism, size 0.3 × 0.4 × 0.6 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.39 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	59.16°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14185, 3521
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2611
$N(param)_{refined}$:	214
Programs:	SHELXTL-plus [4], SHELXTL-97 [5], SADABS [6]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	4*e*	0.3158	0.3985	0.0413	0.037
H(4A)	4*e*	0.0204	0.3837	–0.0267	0.041
H(5A)	4*e*	–0.0886	0.2042	–0.0796	0.041
H(6A)	4*e*	0.1042	0.0451	–0.0701	0.036
H(8A)	4*e*	0.8289	0.4931	0.1488	0.040
H(9A)	4*e*	1.1239	0.5163	0.2099	0.047
H(10A)	4*e*	1.2991	0.3555	0.2537	0.045
H(11A)	4*e*	1.1777	0.1692	0.2339	0.044
H(12A)	4*e*	0.8809	0.1457	0.1716	0.039
H(1W1)	4*e*	0.4105	–0.0219	0.1870	0.046
H(2W1)	4*e*	0.4031	–0.1236	0.1498	0.057
H(1N)	4*e*	0.5822	0.3645	0.1219	0.047

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu(1)	2*b*		1/2	0	0	0.0237(2)	0.0213(2)	0.0333(2)	0.0025(1)	–0.0055(1)	–0.0074(1)
O(1W)	4*e*		0.3877(2)	–0.0604(1)	0.1389(1)	0.0416(9)	0.0353(9)	0.0334(8)	0.0030(7)	–0.0034(7)	–0.0012(7)
O(1)	4*e*		0.6485(2)	0.1236(1)	0.0619(1)	0.0266(7)	0.0228(7)	0.0347(8)	0.0015(6)	–0.0044(6)	–0.0069(6)
N(1)	4*e*		0.6446(2)	0.3096(1)	0.1122(1)	0.0340(9)	0.0184(8)	0.0311(9)	–0.0025(7)	0.0029(7)	–0.0017(7)
N(2)	4*e*		0.3156(2)	0.1248(2)	–0.0087(1)	0.0245(8)	0.0254(8)	0.0267(8)	0.0016(7)	–0.0010(6)	–0.0024(7)
C(1)	4*e*		0.5671(3)	0.2188(2)	0.0682(1)	0.030(1)	0.0210(9)	0.0238(9)	–0.0003(8)	0.0032(8)	–0.0013(7)
C(2)	4*e*		0.3771(3)	0.2283(2)	0.0254(1)	0.029(1)	0.023(1)	0.0228(9)	0.0001(8)	0.0036(7)	–0.0002(7)
C(3)	4*e*		0.2705(3)	0.3273(2)	0.0190(1)	0.038(1)	0.025(1)	0.029(1)	0.0048(9)	0.0056(9)	0.0030(8)
C(4)	4*e*		0.0946(3)	0.3183(2)	–0.0214(1)	0.036(1)	0.036(1)	0.031(1)	0.014(1)	0.0065(9)	0.0074(9)
C(5)	4*e*		0.0301(3)	0.2120(2)	–0.0538(2)	0.025(1)	0.046(1)	0.030(1)	0.0090(9)	0.0003(8)	0.0030(9)
C(6)	4*e*		0.1461(3)	0.1168(2)	–0.0471(1)	0.027(1)	0.033(1)	0.029(1)	0.0007(9)	0.0001(8)	–0.0026(8)
C(7)	4*e*		0.8261(3)	0.3168(2)	0.1525(1)	0.034(1)	0.025(1)	0.0247(9)	–0.0054(8)	0.0029(8)	–0.0029(8)
C(8)	4*e*		0.8992(3)	0.4279(2)	0.1653(2)	0.038(1)	0.025(1)	0.040(1)	–0.0063(9)	0.011(1)	–0.0081(9)
C(9)	4*e*		1.0753(3)	0.4417(2)	0.2024(2)	0.041(1)	0.035(1)	0.044(1)	–0.015(1)	0.013(1)	–0.017(1)
C(10)	4*e*		1.1804(3)	0.3457(2)	0.2284(2)	0.036(1)	0.047(1)	0.029(1)	–0.013(1)	0.0007(9)	–0.008(1)
C(11)	4*e*		1.1076(3)	0.2342(2)	0.2166(1)	0.042(1)	0.038(1)	0.027(1)	–0.006(1)	–0.0052(9)	0.0017(9)
C(12)	4*e*		0.9300(3)	0.2201(2)	0.1790(1)	0.042(1)	0.026(1)	0.028(1)	–0.0080(9)	–0.0053(9)	0.0022(8)
N(3)	4*e*		0.4339(2)	0.6171(1)	0.1534(1)	0.0264(8)	0.0204(8)	0.0325(9)	0.0002(7)	0.0034(7)	0.0001(7)
O(2)	4*e*	0.80	0.5109(3)	0.5224(2)	0.1802(1)	0.036(1)	0.025(1)	0.037(1)	0.0061(8)	–0.0047(9)	0.0004(8)
O(3)	4*e*	0.80	0.3458(3)	0.6197(2)	0.0775(1)	0.039(1)	0.035(1)	0.032(1)	0.0035(9)	–0.0039(8)	0.0071(8)
O(4)	4*e*	0.80	0.4401(4)	0.7011(2)	0.2049(2)	0.072(2)	0.027(1)	0.047(1)	0.008(1)	–0.001(1)	–0.0109(9)
O(2A)	4*e*	0.20	0.376(1)	0.7142(6)	0.1240(5)	0.039(4)	0.022(4)	0.038(4)	0.012(3)	–0.004(3)	0.007(3)
O(3A)	4*e*	0.20	0.461(1)	0.5410(7)	0.0958(6)	0.040(5)	0.027(4)	0.043(4)	0.000(4)	0.006(4)	–0.012(3)
O(4A)	4*e*	0.20	0.489(1)	0.6056(7)	0.2339(5)	0.046(5)	0.039(5)	0.028(4)	0.001(4)	–0.012(3)	0.011(3)

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