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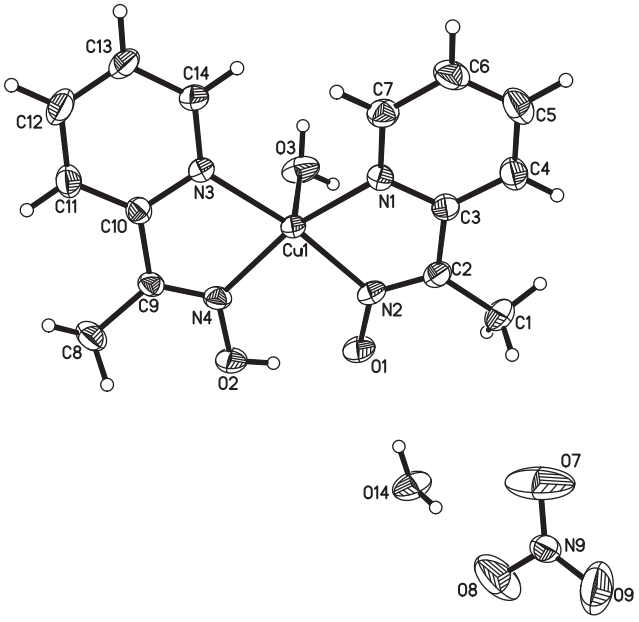
Crystal structure of aqua(1-(2-pyridyl)ethanone oxime-κ²*N*,*N*′)(1-(2-pyridyl)ethanone oximato-κ²*N*, *N*′) nitrate monohydrate, $C_{14}H_{19}N_5O_7Cu$



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Abstract

$C_{14}H_{19}N_5O_7Cu$, triclinic, $P\bar{1}$ (no. 2), $a = 10.8164(15)$ Å, $b = 11.4831(17)$ Å, $c = 15.423(2)$ Å, $\alpha = 101.035(3)°$, $\beta = 100.538(2)°$, $\gamma = 101.708(2)°$, $V = 1791.5(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0416$, $wR_{ref}(F^2) = 0.1131$, $T = 298(2)$ K.

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One half of the asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.49 × 0.43 × 0.37 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.27 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	25°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9414, 6232, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4364
$N(param)_{refined}$:	487
Programs:	Bruker programs [1], SHELX [2, 3]

Source of materials

Hydroxylamine hydrochloride (0.695 g, 10 mmol) was added to sodium hydroxide (0.401 g, 10 mmol) in deionized water (10 mL). The solution was added dropwise to 2-acetylpyridine (1.211 g, 10 mmol) in methanol (10 mL). The reaction was stirred for 3 h. A solution of copper nitrate trihydrate (2.416 g, 10 mmol) in MeOH (10 mL) was added to the above solution of (py)C(Me)NOH. The resulting green solution was stirred for about 6 h and was then allowed to slowly concentrate by solvent evaporation at room temperature. Green block crystals were obtained within a week. The elemental analysis calculated for $C_{14}H_{19}N_5O_7Cu$: C 38.84, H 4.42, N 16.18%; found: C 38.67, H 4.29, N 16.01%.

Experimental details

Hydrogen atoms were assigned with common isotropic displacement factors $U_{iso}(H) = 1.2$ times U_{eq} (C, pyridyl), and $U_{iso}(H) = 1.5$ times U_{eq} (O, water), (C, methyl).

Comment

Pyridyl oximes have the general formula (py)C(*R*)NOH, where py is a pyridyl group attached to the oxime carbon atom and *R* can be a donor or a non-donor group [4, 5]. There are also ligands containing more pyridyl and/or oxime groups. The anions of pyridyl oximes are versatile ligands for a variety of objectives, including preparation of polynuclear complexes, coordination polymers, mixed-metal chemistry and interesting magnetic characteristics [6, 7]. Due to these reasons, we used (py)C(Me)NOH as ligand, and obtained the title copper complex. In this paper, we report the synthesis of a copper complex with (py)C(Me)NOH ligand. The Cu atom is coordinated by a H_2O ligand, an *N*,*N*′-chelating

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.42050(4)	0.88339(4)	0.19732(3)	0.03716(14)
Cu2	0.07060(4)	0.61512(4)	0.25334(3)	0.04202(15)
N1	0.3371(3)	0.8942(3)	0.3059(2)	0.0407(7)
N2	0.5092(3)	0.7818(3)	0.2644(2)	0.0447(8)
N3	0.3244(3)	0.9663(3)	0.1120(2)	0.0387(7)
N4	0.4614(3)	0.8114(3)	0.0810(2)	0.0422(8)
N5	0.1357(3)	0.5808(3)	0.3756(2)	0.0474(8)
N6	−0.0173(3)	0.7144(3)	0.3309(3)	0.0522(9)
N7	0.1897(3)	0.5621(3)	0.1715(2)	0.0414(8)
N8	0.0199(3)	0.6877(3)	0.1492(2)	0.0496(9)
N9	0.4703(4)	0.3057(4)	0.3311(3)	0.0541(9)
N10	0.0143(4)	0.2031(4)	0.2467(3)	0.0613(10)
O1	0.5930(3)	0.7239(3)	0.2320(2)	0.0618(8)
O2	0.5377(3)	0.7323(3)	0.0726(2)	0.0576(8)
H2	0.5686	0.7235	0.1229	0.086*
O3	0.5800(3)	1.0449(2)	0.2508(2)	0.0664(9)
H3A	0.6410	1.0340	0.2894	0.080*
H3B	0.5561	1.1082	0.2726	0.080*
O4	−0.0984(3)	0.7783(3)	0.2979(2)	0.0749(10)
O5	−0.0687(3)	0.7552(3)	0.1442(2)	0.0753(10)
H5	−0.0949	0.7635	0.1911	0.113*
O6	−0.0934(2)	0.4521(2)	0.20382(18)	0.0485(7)
H6A	−0.1613	0.4684	0.2181	0.058*
H6B	−0.0764	0.3941	0.2270	0.058*
O7	0.4527(4)	0.4035(4)	0.3530(4)	0.152(2)
O8	0.5288(5)	0.2895(4)	0.2730(3)	0.139(2)
O9	0.4402(6)	0.2233(5)	0.3633(3)	0.148(2)
O10	0.0918(4)	0.2673(4)	0.2162(3)	0.0986(13)
O11	0.0289(4)	0.1035(4)	0.2590(3)	0.1080(15)
O12	−0.0813(4)	0.2366(3)	0.2646(3)	0.0868(11)
O13	0.8085(3)	0.9987(3)	0.3230(2)	0.0699(9)
H13A	0.8228	0.9280	0.3193	0.084*
H13B	0.8680	1.0422	0.3054	0.084*
O14	0.6821(3)	0.5051(3)	0.2368(2)	0.0731(10)
H14A	0.6473	0.5652	0.2371	0.088*
H14B	0.6301	0.4470	0.2483	0.088*
C1	0.5657(5)	0.7200(4)	0.4067(3)	0.0677(14)
H1A	0.5404	0.7319	0.4636	0.102*
H1B	0.5453	0.6340	0.3790	0.102*
H1C	0.6574	0.7537	0.4169	0.102*
C2	0.4946(4)	0.7824(3)	0.3456(3)	0.0454(10)
C3	0.4009(4)	0.8502(3)	0.3723(3)	0.0423(9)
C4	0.3750(4)	0.8679(4)	0.4576(3)	0.0539(11)
H4	0.4179	0.8360	0.5019	0.065*
C5	0.2861(5)	0.9325(4)	0.4767(3)	0.0631(13)
H5A	0.2712	0.9484	0.5349	0.076*
C6	0.2191(5)	0.9736(4)	0.4092(3)	0.0604(12)
H6	0.1560	1.0151	0.4204	0.072*
C7	0.2466(4)	0.9525(4)	0.3249(3)	0.0501(10)
H7	0.2004	0.9798	0.2792	0.060*
C8	0.4192(5)	0.7788(4)	−0.0840(3)	0.0614(12)
H8A	0.3700	0.8098	−0.1287	0.092*
H8B	0.5089	0.7983	−0.0863	0.092*
H8C	0.3873	0.6916	−0.0962	0.092*
C9	0.4058(4)	0.8360(3)	0.0082(3)	0.0395(9)
C10	0.3298(4)	0.9272(3)	0.0240(3)	0.0402(9)
C11	0.2726(4)	0.9743(4)	−0.0437(3)	0.0532(11)
H11	0.2777	0.9462	−0.1033	0.064*
C12	0.2076(5)	1.0636(4)	−0.0227(3)	0.0662(13)
H12	0.1666	1.0949	−0.0681	0.079*
C13	0.2041(5)	1.1055(4)	0.0658(3)	0.0651(13)
H13	0.1616	1.1664	0.0815	0.078*
C14	0.2647(4)	1.0559(4)	0.1316(3)	0.0491(10)
H14	0.2639	1.0861	0.1919	0.059*
C15	−0.0510(5)	0.7922(5)	0.4813(4)	0.0777(15)
H15A	−0.0205	0.7821	0.5412	0.117*
H15B	−0.0252	0.8771	0.4808	0.117*
H15C	−0.1439	0.7648	0.4640	0.117*
C16	0.0060(4)	0.7186(4)	0.4157(3)	0.0530(11)
C17	0.0937(4)	0.6438(4)	0.4437(3)	0.0483(10)
C18	0.1316(5)	0.6361(5)	0.5321(3)	0.0693(14)
H18	0.1025	0.6806	0.5778	0.083*
C19	0.2126(5)	0.5623(6)	0.5527(4)	0.0810(17)
H19	0.2390	0.5565	0.6123	0.097*
C20	0.2534(5)	0.4979(6)	0.4847(4)	0.0828(17)
H20	0.3091	0.4482	0.4973	0.099*
C21	0.2114(4)	0.5070(5)	0.3968(3)	0.0671(14)
H21	0.2369	0.4599	0.3504	0.081*
C22	0.0332(4)	0.7140(4)	−0.0020(3)	0.0582(12)
H22A	0.0806	0.6881	−0.0459	0.087*
H22B	−0.0582	0.6829	−0.0279	0.087*
H22C	0.0537	0.8018	0.0150	0.087*
C23	0.0697(4)	0.6661(3)	0.0800(3)	0.0445(10)
C24	0.1637(3)	0.5915(3)	0.0893(3)	0.0392(9)
C25	0.2206(4)	0.5514(4)	0.0206(3)	0.0509(10)
H25	0.2002	0.5718	−0.0349	0.061*
C26	0.3082(4)	0.4807(4)	0.0340(3)	0.0637(13)
H26	0.3470	0.4525	−0.0122	0.076*
C27	0.3366(4)	0.4533(4)	0.1162(3)	0.0624(13)
H27	0.3961	0.4067	0.1273	0.075*
C28	0.2763(4)	0.4952(4)	0.1830(3)	0.0543(11)
H28	0.2970	0.4759	0.2389	0.065*

(py)C(Me)NOH molecule and a (py)C(Me)NO molecule. The coordination geometry around the Cu centre is a distorted square-pyramidal CuN_4O configuration. The O3 atom of H_2O ligand occupy the axial sites. The N1, N2, N3 and N4 of (py)C(Me)NO/(py)C(Me)NOH are in the equatorial plane. The Cu—N bond lengths range from 1.976(3) Å to 2.060(3) Å. The bond angles surrounding the Cu atom are in the range of 78.82(13)° to 171.38(13)°. The same is true for the second crystallographically independent complex which is part of the asymmetric unit. Bond lengths and angles are in the expected ranges [8–10].

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