Crystal structure of diaqua-2,6-diacetylpyridine-bis(acetylhydrazone)copper(II) complex dinitrate hydrate, $[Cu(C_{13}H_{17}N_5O_2)(H_2O)_2](NO_3)_2 \cdot H_2O$

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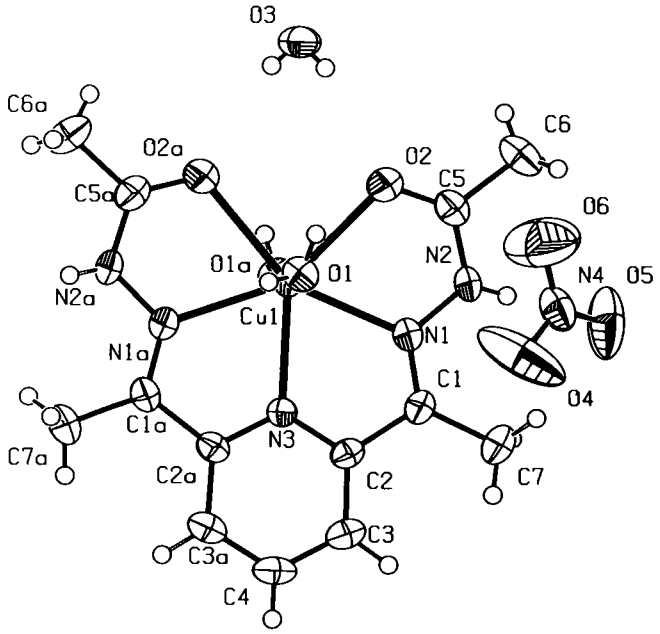

Abstract

$C_{13}H_{23}CuN_7O_{11}$, orthorhombic, *Pbcn* (No. 60), $a = 21.006(4)$ Å, $b = 14.558(3)$ Å, $c = 6.9119(4)$ Å, $V = 2113.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.039$, $wR_{ref}(F^2) = 0.117$, $T = 293$ K.

Source of material

2,6-Diacetylpyridine (10 mmol) and acetylhydrazide (20 mmol) were dissolved in water-ethanol (30/70) with gentle warming. The white product which separated was filtered, washed with 70% ethanol and ice cold ethanol and dried in vacuo to obtain 2,6-diacetylpyridine-bis-(acetylhydrazone) (H_2L). An ethanol solution of $Cu(NO_3)_2 \cdot 3H_2O$ was then added to a 70% ethanol of suspension of the ligand. After heating and stirring for some hours the copper (II) complex was obtained as a green precipitate. Crystals of $[Cu(H_2L)(H_2O)_2](NO_3)_2 \cdot H_2O$ were grown by slow evaporation from an acetonitrile solution.

Discussion

The copper(II) atom has a pentagonal bipyramidal coordination geometry with the hydrazone molecule occupying the equatorial girdle and two water molecules the axial positions. Valency angles show distorsions from an ideal pentagonal bipyramidal arrangement. In particular, ∠(N3–Cu–N1) = 68.99(5)° differs from the ideal value of 72° [1].

Relevant bond distances are: *d*(N1—Cu) = 2.256(2) Å, *d*(N3—Cu) = 2.260(3) Å, *d*(O2—Cu) = 2.367(2) Å, *d*(O1—Cu) = 1.935(2) Å. The O1—Cu length is indicative of a strong link between the Cu and the water molecule. The N1—C1 (1.278(3) Å) and C5—O2 (1.237(3) Å) bonds are essentially double in character.

Table 1. Data collection and handling.

Crystal:	colourless cubic, size 0.33 × 0.33 × 0.33 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.05 cm^{-1}
Diffractometer, scan mode:	CAD4, ω/2θ
$2\theta_{max}$:	53.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2303, 2303
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1824
$N(param)_{refined}$:	186
Programs:	SHELXS-86 [2], SHELXL-97 [3], ORTEPII [4], WinGX [5], PLATON [6]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*d*	–0.012(2)	0.330(3)	0.565(6)	0.05(1)
H(2)	8*d*	–0.055(1)	0.272(3)	0.560(6)	0.07(1)
H(3)	4*c*	0	–0.127(1)	1/4	0.06(2)
H(4)	8*d*	0.092(2)	–0.057(3)	0.312(5)	0.06(1)
H(5)	8*d*	0.184(1)	0.271(3)	0.329(7)	0.07(1)
H(6)	8*d*	0.1663	0.4991	0.3739	0.10(2)
H(7)	8*d*	0.2082	0.4392	0.2348	0.18(3)
H(8)	8*d*	0.2088	0.4201	0.458	0.11(2)
H(9)	8*d*	0.169(2)	0.032(3)	0.383(6)	0.07(1)
H(10)	8*d*	0.197(3)	0.099(4)	0.252(7)	0.10(2)
H(11)	8*d*	0.195(3)	0.126(4)	0.471(8)	0.13(2)
H(12)	8*d*	–0.021(3)	0.470(4)	0.73(1)	0.13(3)

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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}
Cu(1)	4*c*	0	0.27552(3)	1/4	0.0255(3)	0.0272(3)	0.0288(3)	0	–0.0003(2)	0
O(1)	8*d*	–0.0187(1)	0.2844(2)	0.5235(3)	0.039(1)	0.037(1)	0.033(1)	–0.0034(8)	0.0041(9)	–0.0036(8)
O(2)	8*d*	0.07298(9)	0.3966(1)	0.3054(3)	0.038(1)	0.037(1)	0.065(1)	0.0027(8)	–0.002(1)	0.0000(9)
O(3)	4*c*	0	0.4391(2)	3/4	0.057(2)	0.030(1)	0.057(2)	0	–0.000(2)	0
O(4)	8*d*	0.1374(3)	0.1977(3)	0.8696(7)	0.210(5)	0.102(3)	0.119(3)	–0.101(3)	0.075(3)	–0.035(2)
O(5)	8*d*	0.2221(2)	0.2430(3)	0.7335(5)	0.036(1)	0.166(4)	0.094(3)	0.008(2)	0.000(1)	–0.038(2)
O(6)	8*d*	0.1532(2)	0.3347(3)	0.8365(8)	0.152(4)	0.079(3)	0.173(4)	0.028(3)	0.040(3)	–0.010(3)
N(1)	8*d*	0.0983(1)	0.2200(1)	0.3104(4)	0.028(1)	0.034(1)	0.036(1)	–0.0023(9)	–0.0019(9)	–0.0002(9)
N(2)	8*d*	0.1452(1)	0.2834(2)	0.3340(4)	0.024(1)	0.038(1)	0.055(2)	–0.0053(9)	–0.007(1)	0.001(1)
N(3)	4*c*	0	0.1203(2)	1/4	0.028(1)	0.027(1)	0.029(1)	0	–0.001(1)	0
N(4)	8*d*	0.1717(1)	0.2566(2)	0.8148(5)	0.037(1)	0.060(2)	0.056(2)	–0.013(1)	0.004(1)	–0.011(1)
C(1)	8*d*	0.1105(1)	0.1340(2)	0.3184(4)	0.028(1)	0.038(1)	0.034(1)	0.005(1)	–0.002(1)	–0.000(1)
C(2)	8*d*	0.0541(1)	0.0746(2)	0.2838(3)	0.035(1)	0.028(1)	0.030(1)	0.006(1)	0.000(1)	–0.0007(9)
C(3)	8*d*	0.0557(2)	–0.0212(2)	0.2855(4)	0.048(2)	0.029(1)	0.038(2)	0.008(1)	0.000(1)	0.001(1)
C(4)	4*c*	0	–0.0684(3)	1/4	0.062(3)	0.023(2)	0.040(2)	0	0.000(2)	0
C(5)	8*d*	0.1291(1)	0.3727(2)	0.3270(4)	0.038(1)	0.039(1)	0.043(2)	–0.008(1)	–0.000(1)	0.000(1)
C(6)	8*d*	0.1829(2)	0.4387(3)	0.3505(7)	0.059(2)	0.048(2)	0.091(3)	–0.024(2)	–0.010(2)	0.000(2)
C(7)	8*d*	0.1747(2)	0.0934(3)	0.3594(7)	0.034(2)	0.050(2)	0.087(3)	0.011(1)	–0.008(2)	0.001(2)

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