

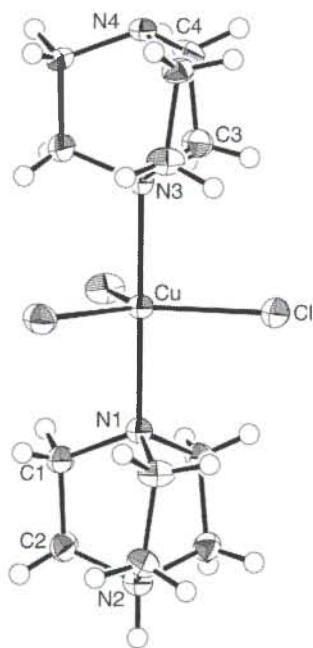
Crystal structure of $[\text{Cu}(\text{Hdabco})(\text{dabco})\text{Cl}_3]$ (dabco = 1,4-diazabicyclo[2.2.2]octane), $\text{C}_{12}\text{H}_{25}\text{Cl}_3\text{CuN}_4$

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Abstract

$\text{C}_{12}\text{H}_{25}\text{Cl}_3\text{CuN}_4$, trigonal, $R3$ (No. 146), $a = 10.681(2)$ Å, $c = 12.056(3)$ Å, $V = 1191.2$ Å³, $Z = 3$, $R_g(F) = 0.030$, $R_w(F) = 0.026$, $T = 293$ K.

Source of material

A DMF solution (20 ml) of 1,4-diazabicyclo[2.2.2]octane (dabco, 1.12 g, 10 mmol) was added to a DMF solution (20 ml) of $\text{CuCl}_2 \cdot 6\text{H}_2\text{O}$ (1.19 g, 5 mmol). The resulting solution was allowed to stand at room temperature and after a few days yellow crystals deposited which were washed with DMF [1].

Discussion

The Cu atom exists in a distorted trigonal bipyramidal geometry defined by three Cl atoms, that define the trigonal plane, and two N atoms derived from both a neutral and protonated dabco molecule. The molecule has crystallographic 3-fold symmetry. Molecules align in the lattice, parallel to the c -axis, connected via H-bonding interactions such that $d(\text{N}2\cdots\text{H}2\text{n}\cdots\text{N}4')$ is 1.71 Å, $d(\text{N}2\cdots\text{N}4')$ is 2.659(7) Å and the angle (from symmetry) is 180°.

The structure reported here is similar to that reported recently for $[\text{Ni}(\text{Hdabco})(\text{dabco})\text{Cl}_3]$, except that the latter was refined in $R32$ indicating additional 2-fold symmetry in the molecule and thus, disorder in the dabco-bound H atoms [2]. An attempt was made to refine the structure in $R32$, i.e. with disordered N–H. This resulted in a higher R_w and significant residual electron density. It is noted that the results reported herein indicate that the molecule has approximate 2-fold symmetry but that the best model was achieved assuming ordered N–H contacts.

Table 1. Data collection and handling.

Crystal:	yellow needle, size 0.05 x 0.08 x 0.39 mm
Wavelength:	Mo $K\alpha$ radiation (0.7107 Å)
μ :	15.80 cm ⁻¹
Diffractometer, scan mode:	Rigaku AFC6R, $\omega/2\theta$
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	690, 614
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 3 \sigma(I_{\text{obs}})$, 566
$N(\text{param})_{\text{refined}}$:	60
Programs:	TEXSAN [3], DIRDIF [4], DIFABS [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1a)	9b	-0.2128	-0.0824	-0.2046	0.0334
H(1b)	9b	-0.1322	0.0836	-0.1866	0.0334
H(2a)	9b	-0.0879	0.1264	-0.3634	0.0315
H(2b)	9b	-0.2036	-0.0347	-0.3813	0.0315
H(2n)	3a	0	0	-0.4678	0.0307
H(3a)	9b	0.2124	0.1304	0.2037	0.0346
H(3b)	9b	0.1310	0.2151	0.1858	0.0346
H(4a)	9b	0.0871	0.2135	0.3633	0.0295
H(4b)	9b	0.2030	0.1685	0.3810	0.0295

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Cu	3 <i>a</i>	0	0	0	0.0225(3)	0.0225	0.0166(4)	0.0112	0	0
Cl	9 <i>b</i>	0.2227(1)	0.0000(2)	−0.0001(1)	0.0288(6)	0.0483(8)	0.0274(7)	0.0243(6)	−0.0003(4)	−0.0019(5)
N(1)	3 <i>a</i>	0	0	−0.1748(5)	0.022(2)	0.0216	0.019(4)	0.0108	0	0
N(2)	3 <i>a</i>	0	0	−0.3889(6)	0.028(2)	0.0277	0.022(4)	0.0138	0	0
N(3)	3 <i>a</i>	0	0	0.1764(5)	0.019(2)	0.019	0.021(4)	0.0095	0	0
N(4)	3 <i>a</i>	0	0	0.3906(5)	0.024(2)	0.0244	0.015(3)	0.0122	0	0
C(1)	9 <i>b</i>	−0.1272(5)	0.0060(6)	−0.2210(4)	0.029(2)	0.042(3)	0.022(2)	0.025(2)	0.000(2)	−0.001(2)
C(2)	9 <i>b</i>	−0.1138(5)	0.0294(6)	−0.3469(4)	0.027(3)	0.031(3)	0.025(2)	0.018(2)	0.002(2)	0.004(2)
C(3)	9 <i>b</i>	0.1264(5)	0.1325(5)	0.2201(4)	0.025(2)	0.027(2)	0.023(2)	0.004(2)	0.001(2)	0.000(2)
C(4)	9 <i>b</i>	0.1128(5)	0.1421(5)	0.3468(4)	0.029(3)	0.023(2)	0.017(2)	0.009(2)	−0.007(2)	−0.003(2)

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