Crystal structure of μ-dichlorobis(*N*-benzyl-bis(2-pyridylmethyl)amino)dicopper(II) diperchlorate ethanol solvate (1:⅔), $[Cu_2Cl_2\{N(C_6H_6N)_2(C_7H_7)\}_2][ClO_4]_2 \cdot \frac{2}{3}\, C_2H_5OH$

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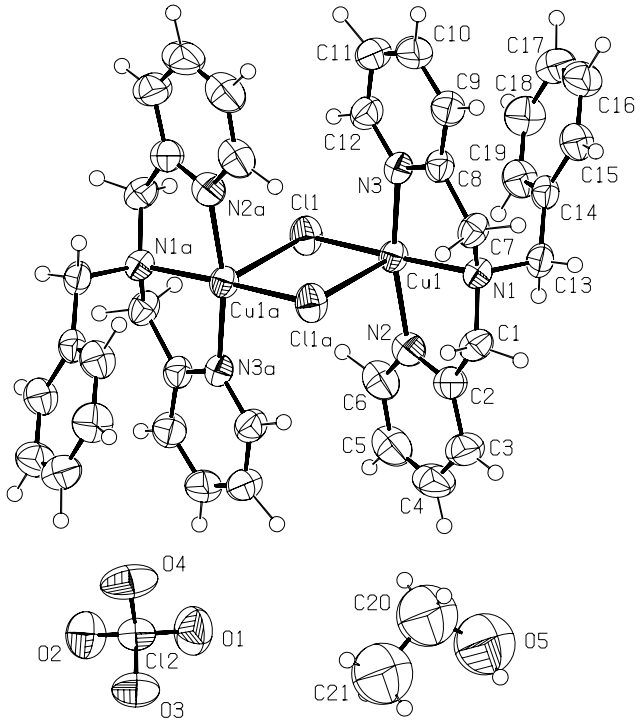

Abstract

$C_{39.33}H_{42}Cl_4Cu_2N_6O_{8.67}$, trigonal, $R\bar{3}$ (no. 148), a = 27.819(4) Å, c = 14.447(6) Å, V = 9682.8 Å^3, Z = 9, $R_{gt}(F)$ = 0.040, $wR_{ref}(F^2)$ = 0.109, T = 293 K.

Source of material

The title compound was prepared by stirring equimolar amounts of copper(II) chloride, *N*-benzyl-bis(2-pyridylmethyl)amine, and sodium perchlorate (1:1:1) in ethanol at 293 K in air for 6 h. The mixture was filtered and the recrystallization was performed from ethanol by diethyl ether diffusion.

Discussion

The title compound forms a dinuclear cation in which the two copper atoms are bridged by two chloride ions and the *N*-benzyl-bis(2-pyridylmethyl)amine (bnbpa) groups serve as outer ligands. The perchlorate ions are not coordinated to copper. Disordered ethanol molecule with statistically filled position completes the crystal structure. The Cu(II) centers have a distorted square pyramidal environment with $\tau = 0.15$ [1]. The square plane is occupied by three nitrogen atoms (two pyridine and one aliphatic amine) and one chloride ion. The three Cu—N bond lengths (2.029(2) Å, 1.988(3) Å and 1.976(2) Å) and the Cu—Cl length (2.2472(9) Å) fall within the expected range for similar bis(2-pyridylmethyl)amine complexes [2,3]. The further chloride ion is situated in axial position (2.890(1) Å).

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Table 1. Data collection and handling.

Crystal:	black prism, size 0.2 × 0.2 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.63 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ
$2\theta_{max}$:	52.62°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11789, 4148
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3548
$N(param)_{refined}$:	262
Programs:	SIR92 [4], SHELXL-97 [5]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	18*f*		0.1852	0.1171	0.2151	0.048
H(1B)	18*f*		0.1399	0.0561	0.2413	0.046
H(3)	18*f*		0.1123	0.0299	0.0727	0.060
H(4)	18*f*		0.1370	0.0085	–0.0684	0.089
H(5)	18*f*		0.2250	0.0219	–0.0888	0.069
H(6)	18*f*		0.2871	0.0568	0.0311	0.074
H(7A)	18*f*		0.2008	0.1013	0.4184	0.050
H(7B)	18*f*		0.2262	0.1502	0.3465	0.030
H(9)	18*f*		0.2758	0.1615	0.5353	0.045
H(10)	18*f*		0.3655	0.1912	0.5839	0.059
H(11)	18*f*		0.4266	0.1830	0.4851	0.068
H(12)	18*f*		0.3971	0.1474	0.3396	0.050
H(13A)	18*f*		0.1562	0.0104	0.3615	0.050
H(13B)	18*f*		0.1796	–0.0056	0.2758	0.060
H(15)	18*f*		0.1995	0.0231	0.5113	0.064
H(16)	18*f*		0.2547	0.0054	0.6074	0.100
H(17)	18*f*		0.3147	–0.0207	0.5466	0.076
H(18)	18*f*		0.3177	–0.0310	0.3891	0.085
H(19)	18*f*		0.2629	–0.0130	0.2922	0.067
O(5)	18*f*	0.33	0.0055	0.0180	0.2979	0.152
H(5O)	18*f*	0.33	0.0319	0.0495	0.2926	0.228
C(20)	18*f*	0.33	–0.0024	–0.0153	0.2185	0.152
H(20A)	18*f*	0.33	–0.0360	–0.0513	0.2207	0.183
H(20B)	18*f*	0.33	0.0292	–0.0211	0.2160	0.183
C(21)	18*f*	0.33	0.0043	0.0194	0.1339	0.152
H(21A)	18*f*	0.33	0.0052	0.0028	0.0792	0.228
H(21B)	18*f*	0.33	0.0380	0.0546	0.1316	0.228
H(21C)	18*f*	0.33	–0.0269	0.0253	0.1373	0.228

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)	18*f*	0.28905(1)	0.10394(1)	0.22687(2)	0.0360(2)	0.0384(2)	0.0405(2)	0.0189(2)	0.0039(1)	–0.0011(1)
Cl(1)	18*f*	0.36878(3)	0.12452(3)	0.15518(5)	0.0474(4)	0.0538(4)	0.0567(5)	0.0307(4)	0.0147(3)	0.0049(3)
N(1)	18*f*	0.21589(9)	0.07800(9)	0.2934(2)	0.033(1)	0.037(1)	0.043(1)	0.016(1)	0.0007(9)	–0.0013(9)
N(2)	18*f*	0.2392(1)	0.0672(1)	0.1196(2)	0.054(2)	0.039(1)	0.044(1)	0.023(1)	–0.001(1)	–0.003(1)
N(3)	18*f*	0.32106(9)	0.13408(9)	0.3498(2)	0.034(1)	0.036(1)	0.045(1)	0.016(1)	0.0028(9)	0.001(1)
C(1)	18*f*	0.1782(1)	0.0795(1)	0.2222(2)	0.038(2)	0.049(2)	0.056(2)	0.022(1)	–0.005(1)	–0.003(1)
C(2)	18*f*	0.1879(1)	0.0591(1)	0.1321(2)	0.051(2)	0.035(2)	0.050(2)	0.019(1)	–0.007(1)	0.000(1)
C(3)	18*f*	0.1481(2)	0.0362(1)	0.0630(3)	0.059(2)	0.051(2)	0.066(2)	0.020(2)	–0.018(2)	–0.002(2)
C(4)	18*f*	0.1627(2)	0.0229(2)	–0.0205(3)	0.093(3)	0.050(2)	0.054(2)	0.022(2)	–0.026(2)	–0.008(2)
C(5)	18*f*	0.2148(2)	0.0309(1)	–0.0329(3)	0.097(3)	0.046(2)	0.049(2)	0.029(2)	–0.005(2)	–0.007(2)
C(6)	18*f*	0.2519(2)	0.0524(1)	0.0387(2)	0.078(3)	0.047(2)	0.049(2)	0.032(2)	0.004(2)	–0.004(1)
C(7)	18*f*	0.2282(1)	0.1185(1)	0.3697(2)	0.036(1)	0.046(2)	0.048(2)	0.020(1)	0.005(1)	–0.006(1)
C(8)	18*f*	0.2852(1)	0.1375(1)	0.4080(2)	0.040(2)	0.035(1)	0.042(2)	0.015(1)	0.002(1)	–0.001(1)
C(9)	18*f*	0.3009(1)	0.1590(1)	0.4961(2)	0.053(2)	0.051(2)	0.045(2)	0.022(2)	0.002(1)	–0.004(1)
C(10)	18*f*	0.3542(2)	0.1764(2)	0.5249(2)	0.062(2)	0.059(2)	0.045(2)	0.021(2)	–0.010(2)	–0.003(2)
C(11)	18*f*	0.3906(1)	0.1719(1)	0.4663(2)	0.048(2)	0.055(2)	0.062(2)	0.021(2)	–0.014(2)	0.002(2)
C(12)	18*f*	0.3726(1)	0.1505(1)	0.3795(2)	0.039(2)	0.050(2)	0.057(2)	0.022(1)	–0.001(1)	0.002(1)
C(13)	18*f*	0.1900(1)	0.0192(1)	0.3286(2)	0.037(2)	0.037(2)	0.049(2)	0.009(1)	0.004(1)	0.003(1)
C(14)	18*f*	0.2258(1)	0.0077(1)	0.3910(2)	0.040(2)	0.032(1)	0.044(2)	0.012(1)	0.005(1)	0.001(1)
C(15)	18*f*	0.2236(1)	0.0125(1)	0.4860(2)	0.057(2)	0.056(2)	0.046(2)	0.030(2)	0.009(1)	0.001(1)
C(16)	18*f*	0.2566(2)	0.0019(2)	0.5437(2)	0.071(2)	0.068(2)	0.045(2)	0.033(2)	–0.004(2)	–0.005(2)
C(17)	18*f*	0.2921(2)	–0.0139(2)	0.5076(3)	0.057(2)	0.062(2)	0.065(2)	0.031(2)	–0.011(2)	–0.005(2)
C(18)	18*f*	0.2941(2)	–0.0198(2)	0.4138(3)	0.064(2)	0.071(2)	0.067(2)	0.044(2)	0.003(2)	–0.005(2)
C(19)	18*f*	0.2611(1)	–0.0090(1)	0.3558(2)	0.062(2)	0.057(2)	0.046(2)	0.032(2)	0.008(1)	–0.004(1)
Cl(2)	18*f*	0.11395(3)	0.16646(4)	0.29978(6)	0.0526(4)	0.0596(5)	0.0572(5)	0.0349(4)	–0.0076(3)	–0.0087(3)
O(1)	18*f*	0.1106(1)	0.1244(1)	0.3590(2)	0.097(2)	0.087(2)	0.094(2)	0.041(2)	–0.003(2)	0.026(2)
O(2)	18*f*	0.1689(1)	0.1952(1)	0.2620(2)	0.061(2)	0.090(2)	0.100(2)	0.041(2)	0.014(2)	0.011(2)
O(3)	18*f*	0.0742(1)	0.1423(1)	0.2272(2)	0.079(2)	0.092(2)	0.071(2)	0.041(2)	–0.026(1)	–0.024(2)
O(4)	18*f*	0.1045(1)	0.2049(1)	0.3484(2)	0.077(2)	0.104(2)	0.117(3)	0.059(2)	–0.020(2)	–0.057(2)

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