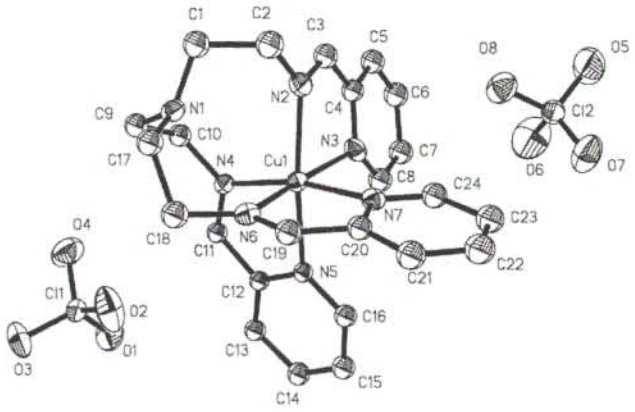
Crystal structure of [tris{2-(2′-pyridinecarboxaldimino)ethyl}amine]copper(II) perchlorate, $[N(CH_2CH_2NCHC_5H_4N)_3Cu](ClO_4)_2$

O. Diouf[I], D. Guinchle Sall[I], A. Salam Sall[I], U. Casellato[II] and R. Graziani*,[III]

[I] Universite Cheikh Anta Diop, Faculté des Sciences et Techniques, Departement de Chimie, Dakar, Senegal
[II] C.N.R. - ICTIMA, Corso Stati Uniti, 4, 1-35100 Padova, Italy
[III] Universitá di Padova, Dipartimento di Chimica Inorganica, Via Loredan, 4, 1-35131 Padova, Italy

Received March 17, 1999, CCDC-No. 1267/145



Abstract

$C_{24}H_{27}Cl_2CuN_7O_8$, monoclinic, $C12/c1$ (No. 15), $a = 28.411(6)$ Å, $b = 10.654(2)$ Å, $c = 19.250(4)$ Å, $\beta = 102.04(3)°$, $V = 5698.7$ Å^3, $Z = 8$, $R_{gt}(F) = 0.048$, $wR(F) = 0.048$, $T = 293$ K.

Source of material

2-Pyridinecarboxaldehyde (9 mmol) and 2,2′,2″-triaminoethylamine (3 mmol) were dissolved in degased absolute ethanol and stirred (30 min). The resulting yellow solution was mixed with an ethanol solution of $Cu(NO_3)_2 \cdot 3H_2O$ (3 mmol) and, after 1 h, with $LiClO_4$ in absolute ethanol. The copper complex was obtained as a dark green precipitate and was then recrystallized from CH_3CN.

Discussion

Hydrogen atoms were introduced in calculated positions. The hexadentate ligand is coordinated to the copper ion in a quasi regular octahedral arrangement. Cu–N bonds range from 2.051 Å to 2.342 Å. The N(2)–C(3), N(4)–C(11) and N(6)–C(19) bonds are clearly double (mean 1.26 Å). The two perchlorate ions are well resolved and structurally ordered. Despite of the different anion the title complex maintains essentially the structure found for the corresponding tetrafluoroborate compound [1].

Table 1. Data collection and handling.

Crystal:	prism, dark-green, max. dim. 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.1 cm^{-1}
Diffractometer, scan mode:	Philips PW1100 (FEBO SYSTEM), 2θ
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4282, 3821
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 4\,\sigma(F_{obs})$, 3740
$N(param)_{refined}$:	259
Program:	SHELX-76 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	8*f*	0.0859(2)	0.1410(5)	0.0274(3)	0.088
H(1B)	8*f*	0.0505(2)	0.1404(5)	0.0801(3)	0.088
H(2A)	8*f*	0.1177(2)	0.0201(6)	0.1297(3)	0.092
H(2B)	8*f*	0.1522(2)	0.1321(6)	0.1220(3)	0.092
H(3A)	8*f*	0.0869(2)	0.0101(5)	0.2259(3)	0.081
H(5A)	8*f*	0.0519(2)	−0.0119(5)	0.3355(3)	0.090
H(6A)	8*f*	0.0466(2)	0.0643(7)	0.4464(4)	0.099
H(7A)	8*f*	0.0829(2)	0.2510(7)	0.4846(3)	0.098
H(8A)	8*f*	0.1228(2)	0.3624(6)	0.4117(3)	0.088
H(9A)	8*f*	0.0129(2)	0.3302(5)	0.0455(3)	0.074
H(9B)	8*f*	0.0417(2)	0.4558(5)	0.0643(3)	0.074
H(10A)	8*f*	−0.0004(2)	0.4154(5)	0.1536(3)	0.074
H(10B)	8*f*	0.0221(2)	0.2805(5)	0.1655(3)	0.074
H(11A)	8*f*	0.0352(2)	0.5671(4)	0.2192(2)	0.060
H(13A)	8*f*	0.0703(2)	0.7347(4)	0.3079(2)	0.068
H(14A)	8*f*	0.1352(2)	0.8078(5)	0.3927(3)	0.077
H(15A)	8*f*	0.2062(2)	0.6931(5)	0.4138(3)	0.078
H(16A)	8*f*	0.2105(2)	0.5112(5)	0.3527(2)	0.070
H(17A)	8*f*	0.0969(2)	0.3989(6)	−0.0051(3)	0.082
H(17B)	8*f*	0.1376(2)	0.3026(6)	0.0259(3)	0.082
H(18A)	8*f*	0.1677(2)	0.5015(5)	0.0561(3)	0.083
H(18B)	8*f*	0.1230(2)	0.5356(5)	0.0888(3)	0.083
H(19A)	8*f*	0.2324(2)	0.4329(5)	0.1325(3)	0.077
H(21A)	8*f*	0.3069(2)	0.3784(6)	0.2261(4)	0.093
H(22A)	8*f*	0.3398(2)	0.2853(6)	0.3349(4)	0.105
H(23A)	8*f*	0.2884(2)	0.2027(5)	0.4029(4)	0.095
H(24A)	8*f*	0.2065(2)	0.2136(5)	0.3594(3)	0.082

* Correspondence author (e-mail: graziani@chim02.chin.unipd.it)

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)	8*f*	0.13625(2)	0.33949(5)	0.23586(3)	0.0429(3)	0.0496(3)	0.0489(3)	–0.0027(2)	0.0109(2)	–0.0028(2)
Cl(1)	8*f*	0.02742(4)	0.7870(1)	0.11593(6)	0.0582(7)	0.0647(7)	0.0497(6)	0.0045(6)	0.0064(5)	0.0005(5)
O(1)	8*f*	0.0220(2)	0.8292(5)	0.1833(2)	0.175(5)	0.126(4)	0.065(3)	0.027(4)	0.014(3)	–0.022(3)
O(4)	8*f*	–0.0067(2)	0.6898(6)	0.0984(3)	0.114(4)	0.140(5)	0.122(4)	–0.042(4)	0.000(3)	0.004(4)
O(2)	8*f*	0.0721(2)	0.7329(7)	0.1184(3)	0.084(3)	0.188(6)	0.161(5)	0.056(4)	0.052(3)	0.080(5)
O(3)	8*f*	0.0177(2)	0.8820(5)	0.0643(3)	0.145(5)	0.105(3)	0.093(3)	0.033(3)	0.027(3)	0.040(3)
Cl(2)	8*f*	0.20945(5)	0.0027(1)	0.48770(8)	0.0731(9)	0.0719(9)	0.0709(8)	0.0058(7)	0.0107(7)	–0.0056(6)
O(5)	8*f*	0.2085(3)	–0.1164(7)	0.5164(4)	0.187(7)	0.140(5)	0.177(6)	–0.042(5)	–0.027(5)	0.062(5)
O(6)	8*f*	0.1786(3)	0.076(1)	0.5174(4)	0.164(7)	0.28(1)	0.180(7)	0.083(7)	0.014(5)	–0.103(7)
O(7)	8*f*	0.2558(2)	0.0521(7)	0.5040(3)	0.099(4)	0.194(6)	0.116(4)	–0.034(4)	0.003(3)	0.033(4)
O(8)	8*f*	0.1910(2)	–0.0000(5)	0.4129(2)	0.120(4)	0.125(4)	0.081(3)	–0.022(3)	0.010(3)	0.002(3)
N(1)	8*f*	0.0840(2)	0.3033(4)	0.0774(2)	0.066(3)	0.065(2)	0.050(2)	–0.010(2)	0.008(2)	–0.009(2)
C(1)	8*f*	0.0818(2)	0.1686(5)	0.0738(3)	0.091(4)	0.062(3)	0.065(3)	–0.010(3)	0.015(3)	–0.018(3)
C(2)	8*f*	0.1208(2)	0.1108(6)	0.1309(3)	0.089(4)	0.064(3)	0.084(4)	0.003(3)	0.030(3)	–0.008(3)
N(2)	8*f*	0.1165(2)	0.1580(4)	0.2012(2)	0.061(2)	0.058(2)	0.065(2)	0.003(2)	0.014(2)	0.005(2)
C(3)	8*f*	0.0974(2)	0.0901(5)	0.2411(3)	0.068(3)	0.050(3)	0.085(4)	–0.002(2)	0.018(3)	0.008(3)
C(4)	8*f*	0.0912(2)	0.1332(5)	0.3108(3)	0.050(3)	0.056(3)	0.077(3)	0.002(2)	0.013(2)	0.021(2)
C(5)	8*f*	0.0663(2)	0.0638(5)	0.3519(3)	0.060(3)	0.066(3)	0.102(4)	0.002(3)	0.026(3)	0.028(3)
C(6)	8*f*	0.0631(2)	0.1092(7)	0.4177(4)	0.072(4)	0.087(4)	0.094(4)	0.017(3)	0.028(3)	0.049(4)
C(7)	8*f*	0.0844(2)	0.2197(7)	0.4400(3)	0.071(4)	0.108(5)	0.070(3)	0.026(4)	0.023(3)	0.031(4)
C(8)	8*f*	0.1084(2)	0.2863(6)	0.3960(3)	0.070(4)	0.073(3)	0.076(4)	0.007(3)	0.011(3)	0.008(3)
N(3)	8*f*	0.1113(1)	0.2442(4)	0.3317(2)	0.054(2)	0.059(2)	0.074(3)	0.000(2)	0.017(2)	0.016(2)
C(9)	8*f*	0.0391(2)	0.3693(5)	0.0789(3)	0.057(3)	0.071(3)	0.052(3)	–0.004(2)	–0.002(2)	–0.008(2)
C(10)	8*f*	0.0282(2)	0.3663(5)	0.1530(3)	0.052(3)	0.070(3)	0.061(3)	–0.006(2)	0.005(2)	–0.007(2)
N(4)	8*f*	0.0691(1)	0.4175(3)	0.2051(2)	0.048(2)	0.052(2)	0.043(2)	–0.004(2)	0.009(2)	–0.003(2)
C(11)	8*f*	0.0642(2)	0.5240(4)	0.2322(2)	0.046(2)	0.059(3)	0.045(2)	0.002(2)	0.012(2)	0.005(2)
C(12)	8*f*	0.1039(2)	0.5792(4)	0.2836(2)	0.053(3)	0.048(2)	0.039(2)	0.002(2)	0.013(2)	0.002(2)
C(13)	8*f*	0.0990(2)	0.6901(4)	0.3181(2)	0.066(3)	0.056(3)	0.050(3)	0.011(2)	0.015(2)	–0.004(2)
C(14)	8*f*	0.1376(2)	0.7338(5)	0.3680(3)	0.083(4)	0.060(3)	0.049(3)	0.000(3)	0.013(2)	–0.010(2)
C(15)	8*f*	0.1796(2)	0.6656(5)	0.3806(3)	0.075(3)	0.067(3)	0.048(3)	–0.011(3)	–0.002(2)	–0.004(2)
C(16)	8*f*	0.1819(2)	0.5566(5)	0.3436(2)	0.055(3)	0.062(3)	0.055(3)	0.006(2)	0.003(2)	0.006(2)
N(5)	8*f*	0.1450(1)	0.5127(3)	0.2951(2)	0.051(2)	0.049(2)	0.042(2)	0.001(2)	0.009(2)	–0.000(2)
C(17)	8*f*	0.1157(2)	0.3642(6)	0.0385(3)	0.075(3)	0.084(4)	0.047(3)	–0.003(3)	0.016(2)	–0.002(2)
C(18)	8*f*	0.1443(2)	0.4680(5)	0.0814(3)	0.065(3)	0.073(3)	0.071(3)	–0.007(3)	0.016(3)	0.004(3)
N(6)	8*f*	0.1690(1)	0.4166(4)	0.1501(2)	0.054(2)	0.059(2)	0.068(2)	–0.007(2)	0.018(2)	–0.011(2)
C(19)	8*f*	0.2142(2)	0.4050(5)	0.1644(3)	0.056(3)	0.068(3)	0.073(3)	–0.001(3)	0.026(2)	–0.005(3)
C(20)	8*f*	0.2378(2)	0.3474(5)	0.2316(3)	0.049(3)	0.057(3)	0.080(3)	–0.001(2)	0.021(2)	–0.015(3)
C(21)	8*f*	0.2871(2)	0.3442(6)	0.2540(4)	0.050(3)	0.083(4)	0.102(5)	0.000(3)	0.021(3)	–0.012(3)
C(22)	8*f*	0.3066(2)	0.2890(6)	0.3188(4)	0.051(3)	0.074(4)	0.129(6)	0.008(3)	0.001(4)	–0.024(4)
C(23)	8*f*	0.2763(2)	0.2398(5)	0.3591(4)	0.078(4)	0.061(3)	0.090(4)	0.014(3)	–0.001(3)	–0.008(3)
C(24)	8*f*	0.2271(2)	0.2471(5)	0.3325(3)	0.066(3)	0.060(3)	0.077(4)	0.007(3)	0.013(3)	–0.005(3)
N(7)	8*f*	0.2081(1)	0.2990(4)	0.2709(2)	0.049(2)	0.050(2)	0.066(2)	0.003(2)	0.014(2)	–0.004(2)

References

1. Lingafelter, E. C.; Bailey, M. F.; Howe, N. L.; Kirchner, R. M.; Mealli, C.; Torre, L. P.: (Tris(2-(2'-pyridinecarboxaldimino)ethyl)amine)copper(II) bis(tetrafluoroborate). Am. Cryst. Assoc., Abstr. Papers (Summer) (1970) 81.
2. Sheldrick, G. M.: SHELXL-76, Programs for Crystal Structures Determination. Cambridge 1976.