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Chloropyramine Tetrachlorocuprate(II)

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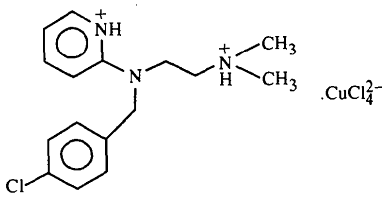
Abstract

The crystal structure of *N*-(4-chlorobenzyl)-*N*′,*N*′-dimethyl-*N*-(2-pyridinio)-1,2-ethanediammonium tetrachlorocopper(II), $(C_{16}H_{22}ClN_3)[CuCl_4]$, contains the dication of chloropyramine, a potent anti-allergic agent effective on H_1-type receptors. The $CuCl_4^{2-}$ anion exhibits a flattened-tetrahedral geometry. The ammonium H atom is weakly bonded to two Cl atoms of the anion and the pyridyl N atom is involved in a short hydrogen bond to a Cl atom of the anion.

Comment

As a continuation of our studies (Parvez & Sabir, 1997*a*,*b*) on the conformations of cations of antihistamines effective on H_1-receptors, we have prepared the tetrachlorocuprate(II) salt of chloropyramine, (1), the crystal structure of which is described in this paper.



(1)

An *ORTEP*II (Johnson, 1976) drawing of (1) is shown in Fig. 1. The molecular dimensions of the cation are normal with mean bond distances N—C_{sp^3} 1.474 (9), N—C_{sp^2} 1.360 (3) and C—C_{aromatic} 1.37 (2) Å, and bond distances C_{sp^3}—C_{sp^3} 1.533 (12), C_{sp^3}—C_{sp^2} 1.506 (13) and C—Cl 1.749 (12) Å.

The pyridyl and phenyl rings are individually planar, with maximum deviations of 0.016 (6) and 0.012 (8) Å, respectively, and are inclined at an angle of 79.3 (3)° with respect to one another. The ethylenediammonium side chain has a somewhat extended conformation.

The $CuCl_4^{2-}$ anion is a flattened tetrahedron, with Cu—Cl distances in the range 2.229 (3)–2.276 (3) Å and four Cl—Cu—Cl angles in the range 96.08 (11)–101.29 (11)°, the remaining two angles being 127.87 (12) and 138.06 (12)°. Similar geometry of the $CuCl_4^{2-}$ anion has been reported in a number of structures, *e.g.* clemizole tetrachlorocuprate(II) (Parvez & Sabir, 1997*a*), triprolidine tetrachlorocuprate(II) (Parvez & Sabir, 1997*b*), dicytosinium tetrachlorocuprate(II) (Ogawa, Nishitani, Fujiwara, Shirotake & Tomita, 1979), chlorpromazine tetrachlorocuprate(II) (Obata, Kawazura & Miyamae, 1984), promazine tetrachlorocuprate(II) (Von Dreele & Harris, 1983) and fenethazine tetrachlorocuprate(II) (Obata, Yoshimori, Yamada & Kawazura, 1985).

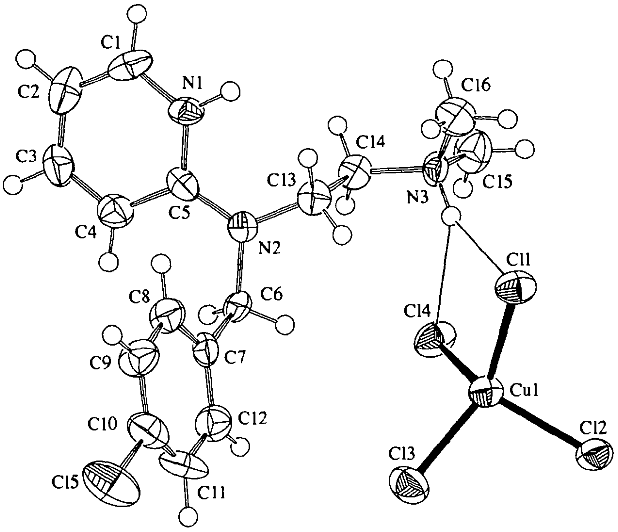


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (1) with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms have been assigned arbitrary radii.

It is interesting to note that the ammonium H3 atom is weakly bonded to two Cl atoms of the anion, with H···Cl interactions of 2.49 and 2.50 Å; the N···Cl separations are 3.200 (7) and 3.324 (7) Å. The pyridyl N1 atom is also hydrogen bonded to a Cl atom of a symmetry-related anion, with a H···Cl interaction of 2.34 Å and an N1···Cl2 separation of 3.175 (7) Å.

Experimental

Chloropyramine hydrochloride (0.326 g, 1 mmol; Sigma Inc.) in 20 ml concentrated HCl was mixed with $CuCl_2.2H_2O$ (0.171 g, 1 mmol) and boiled for 10 min. On allowing the solution to stand overnight, yellow needles of (1) were obtained which were washed with acetone.

Crystal data

$(C_{16}H_{22}ClN_3)[CuCl_4]$
$M_r = 497.16$
Monoclinic
$P2_1/a$
$a = 14.355$ (2) Å
$b = 7.836$ (2) Å
$c = 20.353$ (2) Å
$\beta = 106.74$ (1)°
$V = 2192.4$ (7) Å^3
$Z = 4$
$D_x = 1.506$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 12$–20°
$\mu = 1.61$ mm^{-1}
$T = 296$ (2) K
Needle
0.60 × 0.10 × 0.05 mm
Yellow

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.829$, $T_{\max} = 0.923$
4211 measured reflections
3894 independent reflections
1257 reflections with $F > 4\sigma(F)$
$R_{\rm int} = 0.0996$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 9$
$l = -24 \rightarrow 23$
3 standard reflections every 200 reflections intensity decay: 0.44%

Refinement

Refinement on F^2
$R(F) = 0.041$
$wR(F^2) = 0.099$
$S = 0.907$
3884 reflections
227 parameters
H atoms riding, with C—H and N—H 0.95 Å
$w = 1/[\sigma^2(F_o^2) + (0.0684P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.52$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Cu1—Cl3	2.229 (3)	N2—C13	1.463 (11)
Cu1—Cl1	2.237 (3)	N2—C6	1.468 (11)
Cu1—Cl4	2.246 (3)	N3—C15	1.473 (12)
Cu1—Cl2	2.276 (3)	N3—C14	1.481 (11)
Cl5—C10	1.749 (12)	N3—C16	1.488 (11)
N1—C1	1.356 (12)	C6—C7	1.506 (13)
N1—C5	1.362 (11)	C13—C14	1.533 (12)
N2—C5	1.361 (12)		
Cl3—Cu1—Cl1	138.06 (12)	C5—N2—C6	118.8 (8)
Cl3—Cu1—Cl4	100.11 (12)	C13—N2—C6	117.2 (8)
Cl1—Cu1—Cl4	96.08 (11)	C15—N3—C14	110.0 (8)
Cl3—Cu1—Cl2	98.68 (11)	C15—N3—C16	111.4 (8)
Cl1—Cu1—Cl2	101.29 (11)	C14—N3—C16	112.6 (7)
Cl4—Cu1—Cl2	127.87 (12)	N2—C6—C7	115.2 (8)
C1—N1—C5	123.3 (9)	N2—C13—C14	109.5 (8)
C5—N2—C13	123.8 (8)	N3—C14—C13	111.9 (8)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl2$^{\rm i}$	0.95	2.34	3.175 (7)	147
N3—H3···Cl1	0.95	2.50	3.324 (7)	145
N3—H3···Cl4	0.95	2.49	3.200 (7)	131

Symmetry code: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z$.

The space group $P2_1/a$ was determined uniquely from the systematic absences of $h0l$, $h = 2n + 1$, and $0k0$, $k = 2n + 1$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Software used to prepare material for publication: *TEXSAN*.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: FG1261). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Triprolidine Tetrachlorocuprate(II)

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Abstract

The crystal structure of (*E*)-2-[3-(1-pyrrolidinio)-1-(*p*-tolyl)-1-propenyl]pyridinium tetrachlorocopper(II), $(C_{19}H_{24}N_2)[CuCl_4]$, contains the dication of triprolidine, a potent anti-allergic agent. The $CuCl_4^{2-}$ anion exhibits a flattened-tetrahedral geometry and is hydrogen bonded through two Cl atoms to the same cation, forming independent anion–cation pairs.

Comment

As a continuation of our studies (Parvez & Sabir, 1997*a*,*b*) on the conformations of cations of antihistamines effective on H_1-receptors, we have prepared the tetrachlorocuprate(II) salt of triprolidine, (1), the crystal structure of which is presented in this paper.

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