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*.

The authors thank the Natural Sciences and Engineering Research Council, Canada, for providing the diffractometer through an equipment grant to the University of Calgary, and the University of Calgary for financial support.

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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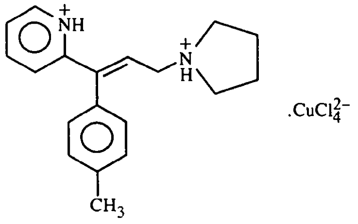
Abstract

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.



(1)

An *ORTEP*II (Johnson, 1976) drawing of compound (1) is shown in Fig. 1. The molecular dimensions in the cation are normal. The pyridyl and phenyl rings are individually planar and are inclined at an angle of 83.7 (2)° with respect to one another; the corresponding angle in the crystal structure of triprolidine hydrochloride hydrate was observed to be 73.4° (James & Williams, 1974).

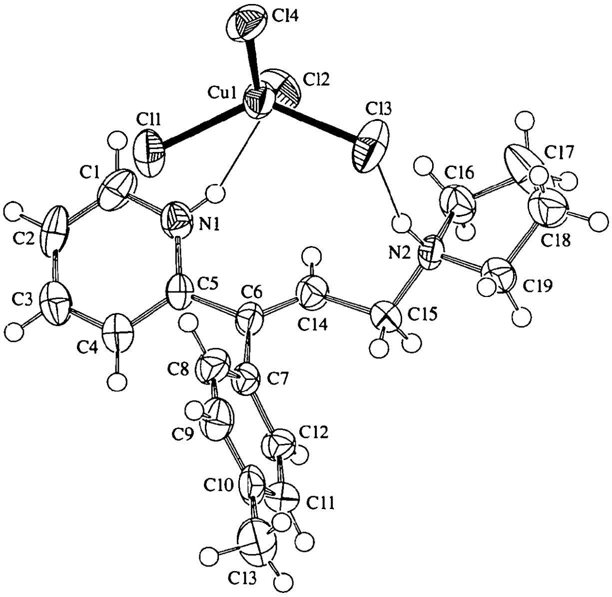


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (1) with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms have been assigned arbitrary radii.

The pyrrolidinyl ring in (1) has an N2-envelope conformation, with the N2 atom 0.459 (10) Å out of the plane of the remaining four C atoms of the ring, which is in agreement with the conformation of the corresponding ring in triprolidine hydrochloride hydrate. The orientation of the H atoms involved in hydrogen bonding with the anion, however, determine the overall conformation of the dication in (1), which is drastically different from that of the monocation in triprolidine hydrochloride hydrate, as evident from the C14—C15—N2—H2 torsion-angle values of 51 and −58°, respectively.

The $CuCl_4^{2-}$ anion is a flattened tetrahedron, with Cu—Cl distances in the range 2.230 (2)–2.290 (2) Å and four Cl—Cu—Cl angles in the range 95.80 (8)–101.40 (8)°, the remaining two Cl—Cu—Cl angles being 129.83 (9) and 136.83 (9)°. 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*), chloropyramine 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).

It is interesting to note that the anion is hydrogen bonded through two Cl atoms to the same cation forming independent anion–cation pairs with strong N· · ·Cl interactions of 3.094 (6) and 3.110 (6) Å.

Experimental

Triprolidine hydrochloride (0.315 g, 1 mmol; Sigma Inc.) in 15 ml 6 *M* 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 prisms of (1) were obtained and washed with acetone.

Crystal data

$(C_{19}H_{24}N_2)[CuCl_4]$
$M_r = 485.74$
Monoclinic
$P2_1/n$
$a = 10.106$ (4) Å
$b = 11.800$ (5) Å
$c = 18.301$ (4) Å
$\beta = 94.42$ (2)°
$V = 2175.9$ (13) Å^3
$Z = 4$
$D_x = 1.483$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 1.50$ mm^{-1}
$T = 200$ (2) K
Prism
0.30 × 0.20 × 0.16 mm
Yellow

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.721$, $T_{\max} = 0.787$
4047 measured reflections
3835 independent reflections

1605 reflections with $F > 4\sigma(F)$
$R_{\rm int} = 0.0910$
$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 14$
$l = -21 \rightarrow 21$
3 standard reflections every 200 reflections intensity decay: 0.55%

Refinement

Refinement on F^2
$R(F) = 0.041$
$wR(F^2) = 0.092$
$S = 1.087$
3818 reflections
236 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.0669P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.42$ e Å^{-3}

H atoms riding, with C—H and N—H 0.95 Å

Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Cu1—Cl1	2.230 (2)	C5—C6	1.479 (8)
Cu1—Cl4	2.235 (2)	C6—C14	1.319 (8)
Cu1—Cl2	2.257 (2)	C6—C7	1.501 (8)
Cu1—Cl3	2.290 (2)	C10—C13	1.506 (9)
N1—C1	1.343 (8)	C14—C15	1.509 (8)
N1—C5	1.352 (7)	C16—C17	1.509 (9)
N2—C16	1.487 (7)	C17—C18	1.467 (10)
N2—C15	1.489 (7)	C18—C19	1.526 (9)
N2—C19	1.499 (7)		
Cl1—Cu1—Cl4	101.40 (8)	C14—C6—C5	120.3 (6)
Cl1—Cu1—Cl2	99.04 (7)	C14—C6—C7	121.4 (6)
Cl4—Cu1—Cl2	136.83 (8)	C5—C6—C7	118.3 (5)
Cl1—Cu1—Cl3	129.83 (9)	C6—C14—C15	124.1 (6)
Cl4—Cu1—Cl3	95.80 (8)	N2—C15—C14	111.4 (5)
Cl2—Cu1—Cl3	99.59 (8)	N2—C16—C17	105.2 (6)
C1—N1—C5	123.3 (6)	C18—C17—C16	108.7 (6)
C16—N2—C15	115.7 (5)	C17—C18—C19	105.2 (6)
C16—N2—C19	105.9 (5)	N2—C19—C18	103.4 (5)
C15—N2—C19	112.2 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl2	0.95	2.31	3.110 (6)	154
N2—H2···Cl3	0.95	2.18	3.080 (6)	161

The space group $P2_1/n$ was determined uniquely from the systematic absences of $h0l$, $h + l = 2n + 1$, 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: FG1262). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-[2,3-Bis(methylthio)hexane-*S*,*S*′]dichlorozinc(II), *trans*-[1,2-Bis(methylthio)cyclohexane-*S*,*S*′]dichlorozinc(II) and *cis*-[5,6-Bis(methylthio)-1,3-cycloheptadiene-*S*,*S*′]dichlorozinc(II)

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Abstract

The crystal structures of *trans*-[2,3-bis(methylthio)hexane-*S*,*S*′]dichlorozinc(II), $[ZnCl_2(C_8H_{18}S_2)]$, *trans*-[1,2-bis(methylthio)cyclohexane-*S*,*S*′]dichlorozinc(II), $[ZnCl_2(C_8H_{16}S_2)]$, and *cis*-[5,6-bis(methylthio)-1,3-cycloheptadiene-*S*,*S*′]dichlorozinc(II), $[ZnCl_2(C_9H_{14}S_2)]$, contain distorted tetrahedral Zn atoms coordinated to two Cl and two S atoms, with Zn—Cl and Zn—S distances in the ranges 2.190 (2)–2.222 (2) and 2.369 (2)–2.475 (1) Å, respectively. The Cl—Zn—Cl and S—Zn—S angles in the three compounds are in the ranges 117.35 (4)–122.63 (4) and 88.11 (5)–91.12 (6)°, respectively.

Comment

The addition of dimethyl disulfide to an alkene using a Lewis acid catalyst such as BF_3 is a well known process (Caserio, Fisher & Kim, 1985). The mechanism is thought to proceed *via* a thiiranium ion intermediate which undergoes a ring opening by the incoming thiomethyl anion to give *anti* addition across the double bond. Our interest in montmorillonite clays prompted us

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