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# Structure of a Novel One-Dimensional Chlorpromazine–Copper(II) Complex Salt,* $[C_{17}H_{20}ClN_2S]_2[CuCl_4]_2$

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**Abstract.** $M_r = 1050{\cdot}4$, orthorhombic, *Pnma*, $a = 23{\cdot}745$ (4), $b = 13{\cdot}897$ (3), $c = 12{\cdot}580$ (2) Å, $V = 4151{\cdot}2$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}684$, $D_m$(flotation in $CCl_4$/EtOH) = 1·622 Mg m$^{-3}$, $\mu$(Mo $K\alpha$: $\lambda = 0{\cdot}71069$ Å) = 1·70 mm$^{-1}$, $F(000) = 2128$, room temperature. The structure was solved by the heavy-atom method and refined to a final $R$ value of 0·057 for 2161 observed reflections. In the crystal, to equilibrate the charge of the $CuCl_4^{2-}$, each chlorpromazine moiety needs a charge of 2+. Protonation of the N atom in the side chain accounts for 1+, and the other positive charge is spread over the phenothiazine ring resulting in the formation of a dimeric pair of chlorpromazines. In the pair of phenothiazine rings, the S—S′ distance of 2·944 (3) Å is extremely short suggesting some bonding interaction. The dimeric pairs stack one-dimensionally along **b**.

* 2-Chloro-10-(3-dimethylammoniopropyl)phenothiazinium tetrachlorocuprate(II).

**Introduction.** The structure of complexes formed through interactions between neuroleptics and copper ions is an interesting subject in connection with various important functions of copper enzymes in the human brain. However, reports on detailed X-ray structural analyses of the resultant complexes seem to be limited to those of diazepam as the anti-anxiety drug (Mosset, Tuchagues, Bonnet, Haran & Sharrock, 1980; Miyamae, Obata & Kawazura, 1982).

Chlorpromazine hydrochloride [2-chloro-10-(3-dimethylaminopropyl)phenothiazine hydrochloride, CPZ.HCl], one of the major tranquilizers well known on account of its outstanding effect, yields a black crystalline complex of composition $[C_{17}H_{20}ClN_2S]$-$[CuCl_4]$ when treated with $CuCl_2$ in ethanol containing HCl.

X-ray diffraction analysis of this crystal has revealed that in the complex, a pair of CPZ.H moieties piles up one-dimensionally as though necessitated by the $CuCl_4^{2-}$ anions.

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**Experimental.** Crystal *ca* 0·62 × 0·16 × 0·16 mm, Rigaku AFC-5, graphite monochromator; crystal data from least-squares fit on the basis of 22 $2\theta$ values, Mo $K\alpha$ radiation ($19 < 2\theta < 23°$, $\lambda = 0{\cdot}7107$ Å); intensity measurement performed to $2\theta = 55°$ ($h \geq 0$, $k \geq 0$, $l \geq 0$), $\theta$–$2\theta$ scan technique, scan speed 3° min$^{-1}$ ($\theta$); 5326 reflections measured, 2161 independent reflections having $|F|/\sigma(|F|) \geq 3{\cdot}0$ considered 'observed' and used for structure determination; Lp corrections applied; heavy-atom and Fourier methods, block-diagonal least-squares refinement with anisotropic thermal factors for all non-H atoms and isotropic ones for H atoms (H from $\Delta F$ maps); $\sum w||F_o|-|F_c||^2$ minimized, $w = 1/[\sigma^2 + (0{\cdot}02F_o)^2]$; final $R = 0{\cdot}057$ and $R_w = 0{\cdot}052$, $(\Delta/\sigma)_{max} = 0{\cdot}24$, final $\Delta\rho$ excursions $\leq |0{\cdot}6|$ e Å$^{-3}$; calculations performed on a FACOM M-160F computer of this university with *UNICS* III (Sakurai & Kobayashi, 1978). Complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974).

**Discussion.** Table 1 gives the positional parameters, Fig. 1 the numbering scheme.*

Fig. 2 shows the **c** projection of the unit-cell members. Bond distances and angles are given in Table 2. The differences between CPZ.H and the free CPZ molecule are as follows: In the hetero ring, the S—C bond lengths are shorter than in the free molecule [1·75 (1) Å (McDowell, 1969)] and the bond angle around the S atom is greater than that in the analogue [97·3 (3)° in the free molecule], indicating C—S double-bond character (*International Tables for X-ray Crystallography,* 1968). Furthermore, the bond angle C(11)—N(10)—C(14) is much greater than that in free CPZ [118·4 (5)°]. The dihedral angle between the planes of the lateral benzene rings of 175·0 (3)° is much greater than that of 139·4° in free CPZ. Thus the atoms of the phenothiazine ring are almost on a plane. These facts indicate that S(5) and N(10) participate in the $\pi$ conjugated system.

The two flattened phenothiazine rings related by a mirror plane make up a pair. These pairs are stacked in a parallel fashion to form a column along **b**, with the amine side chain alternately oriented in opposite directions. An eclipsed overlapping of the flattened phenothiazine rings in the dimeric pair results in an interplanar distance of 3·38 (1) Å, the average of distances C(11$^{i}$)···C(11$^{ii}$), C(12$^{i}$)···C(12$^{ii}$), C(13$^{i}$)···C(13$^{ii}$) and C(14$^{i}$)···C(14$^{ii}$) [symmetry operations: (i) $x, y, z$; (ii) $x, \frac{1}{2}-y, z$]. S(5$^{i}$)···S(5$^{ii}$), 2·944 (3) Å, is extremely short with respect to twice the van der Waals radius (3·70 Å; Pauling, 1967) and suggests some bonding interaction. N(10$^{i}$)···N(10$^{ii}$) is 3·34 (1) Å.

The overlapping feature of the dimers is illustrated in Fig. 2, in which the face-to-face counterparts in the two adjacent dimers are drawn projected on the least-squares plane of the lower phenothiazine ring. The observed overlapping mode can be understood on the basis of the mutual avoidance of the $\pi$-electron clouds in the rings, giving an interplanar distance of 3·57 (1) Å* and the shortest interatomic distance of 3·59 (1) Å for C(7$^{i}$)···C(12$^{ii}$).

The amine side chain bonded through N(10) is *trans*, as can be seen from Fig. 2, whereas that in the free molecule is *gauche*. This feature may be related to the dimer formation.

The $CuCl_4^{2-}$ anions exhibit two different geometries: one is nearly tetrahedral [$CuCl_4(A)$] and the other is extremely flattened [$CuCl_4(B)$]. The structure of the $CuCl_4^{2-}$ ion is sensitive to its surroundings and takes a flattened tetrahedral shape with Cl—Cu—Cl bond angles in the ranges 124~130° (two) and 103~100° (four) (Wells, 1975). The complex anion $CuCl_4^{2-}(A)$ does not deviate appreciably from regular tetrahedral geometry, whereas the deviation is quite large in $CuCl_4^{2-}(B)$. This deformation of the latter may be due to the different environment: $CuCl_4^{2-}(B)$ is located between the two side chains of the pair of CPZ.H's, Cl(3)$B$···N(18) being as short as 3·315 (6) Å. This explains the flattened configuration of $CuCl_4^{2-}$ ($B$) and proves the presence of an electrostatic interaction between $CuCl_4^{2-}(B)$ and the protonated terminal N atoms.† $CuCl_4^{2-}(B)$ is also close to the phenothiazine ring of the adjacent column, giving the shortest interatomic distance of 3·396 (3) Å for Cl(1)$B$···S(5). On the other hand, $CuCl_4^{2-}(A)$ is located in the hollow formed by the CPZ.H columns.

The crystal consists of a pair of CPZ.H and a pair of $CuCl_4^{2-}$ ($A$ and $B$). To counterbalance the eight $CuCl_4^{2-}$, there are eight CPZ units in the cell which presumably have a 2+ charge. Protonation of N(18) is clearly established, accounting for 1+. Thus the other positive charge must be spread over the aromatic ring. The planar deformation of the phenothiazine ring in its cation radical has been well documented (Shinghabhandhu, Robinson, Fang & Geiger, 1975; Clarke, Gilbert, Hanson & Kirk, 1978; Hester & Williams, 1981); therefore, we may assume that the parallel stacking of the phenothiazine rings is due to a formation of $CPZ.H^{2+}$ radical cations.

* Lists of structure factors, anisotropic thermal parameters, atomic parameters of H atoms, and intermolecular distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38831 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* Since the phenothiazine planes are almost perpendicular to **b** (see Fig. 2), the distance was calculated by the equation [$|b|/2-3{\cdot}38$ (the interplanar distance of the dimer)] Å.

† H(18) is located at distances of 0·79 (5) Å from N(18) and 2·61 (5) Å from Cl(3)$B$.

Table 1. *Atomic coordinates* ($\times 10^4$) *and* $B_{eq}$ *values* ($\text{Å}^2$)

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$$

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| Cu*A* | 3287 (1) | 2500 | −670 (1) | 3·10 (4) |
| Cu*B* | 2839 (1) | 2500 | 3921 (1) | 3·20 (4) |
| Cl(1)*A* | 3273 (1) | 2500 | 1099 (3) | 5·88 (12) |
| Cl(2)*A* | 2329 (1) | 2500 | −927 (2) | 4·37 (10) |
| Cl(3)*A* | 3763 (1) | 1259 (2) | −1369 (2) | 6·45 (9) |
| Cl(1)*B* | 3294 (1) | 2500 | 5533 (2) | 3·14 (8) |
| Cl(2)*B* | 1909 (1) | 2500 | 3585 (3) | 4·98 (11) |
| Cl(3)*B* | 3028 (1) | 956 (1) | 3578 (2) | 3·52 (6) |
| Cl* | 547 (1) | 1104 (3) | 5039 (2) | 6·20 (11) |
| Cl′* | 1036 (6) | 1429 (14) | −2938 (11) | 6·88 (55) |
| C(1) | 608 (3) | 1159 (6) | 2925 (6) | 3·31 (23) |
| C(2) | 239 (3) | 1164 (6) | 3792 (6) | 4·31 (27) |
| C(3) | −329 (3) | 1237 (6) | 3734 (6) | 3·88 (25) |
| C(4) | −559 (3) | 1301 (5) | 2766 (6) | 3·41 (23) |
| S(5) | −592 (1) | 1441 (1) | 691 (2) | 3·01 (6) |
| C(6) | −268 (3) | 1305 (6) | −1316 (6) | 3·74 (25) |
| C(7) | 101 (4) | 1250 (6) | −2123 (6) | 4·56 (28) |
| C(8) | 669 (4) | 1175 (6) | −1927 (6) | 4·26 (27) |
| C(9) | 864 (3) | 1194 (6) | −899 (6) | 3·57 (24) |
| N(10) | 711 (2) | 1300 (4) | 1022 (4) | 2·37 (16) |
| C(11) | 367 (3) | 1247 (5) | 1911 (5) | 2·36 (20) |
| C(12) | −227 (3) | 1312 (5) | 1841 (6) | 2·50 (20) |
| C(13) | −83 (3) | 1309 (5) | −249 (5) | 2·59 (21) |
| C(14) | 502 (3) | 1266 (5) | −19 (5) | 2·47 (20) |
| C(15) | 1329 (3) | 1307 (5) | 1161 (5) | 2·40 (19) |
| C(16) | 1587 (3) | 316 (5) | 1169 (6) | 2·94 (21) |
| C(17) | 2223 (3) | 432 (5) | 1052 (6) | 3·14 (22) |
| N(18) | 2518 (2) | −500 (4) | 950 (5) | 3·17 (18) |
| C(19) | 3080 (4) | −367 (7) | 476 (8) | 6·17 (34) |
| C(20) | 2560 (4) | −1033 (6) | 1966 (7) | 4·96 (29) |

* The populations of Cl[H(8)] and Cl′[H(8)′] are 0·83 and 0·17, respectively.

Table 2. *Bond distances* (Å) *and angles* (°)

| | $CuCl_4^{2-}$(*A*) | $CuCl_4^{2-}$(*B*) |
|---|---|---|
| Cu–Cl(1) | 2·226 (4) | 2·297 (3) |
| Cu–Cl(2) | 2·296 (3) | 2·250 (3) |
| Cu–Cl(3) | 2·242 (3) | 2·234 (2) |
| Cl(1)–Cu–Cl(2) | 97·3 (1) | 128·8 (1) |
| Cl(1)–Cu–Cl(3) | 113·6 (1) | 94·4 (1) |
| Cl(2)–Cu–Cl(3) | 116·3 (1) | 99·3 (1) |
| Cl(3)–Cu–Cl(3′) | 100·6 (1) | 147·7 (1) |

| | | | |
|---|---|---|---|
| C(1)–C(2) | 1·40 (1) | C(8)–C(9) | 1·37 (1) |
| C(2)–C(3) | 1·35 (1) | C(7)–C(8) | 1·38 (1) |
| C(3)–C(4) | 1·34 (1) | C(6)–C(7) | 1·34 (1) |
| C(4)–C(12) | 1·41 (1) | C(6)–C(13) | 1·41 (1) |
| C(12)–C(11) | 1·42 (1) | C(14)–C(13) | 1·42 (1) |
| C(1)–C(11) | 1·40 (1) | C(9)–C(14) | 1·40 (1) |
| C(15)–C(16) | 1·51 (1) | C(16)–C(17) | 1·53 (1) |
| N(10)–C(11) | 1·39 (1) | N(18)–C(17) | 1·48 (1) |
| N(10)–C(14) | 1·40 (1) | N(18)–C(19) | 1·47 (1) |
| N(10)–C(15) | 1·48 (1) | N(18)–C(20) | 1·48 (1) |
| S(5)–C(12) | 1·70 (1) | Cl–C(2) | 1·73 (1) |
| S(5)–C(13) | 1·70 (1) | | |
| C(11)–C(1)–C(2) | 116·9 (7) | C(14)–C(9)–C(8) | 122·5 (7) |
| C(1)–C(2)–C(3) | 125·6 (7) | C(9)–C(8)–C(7) | 119·8 (7) |
| C(2)–C(3)–C(4) | 117·4 (7) | C(8)–C(7)–C(6) | 120·6 (7) |
| C(3)–C(4)–C(12) | 121·7 (7) | C(7)–C(6)–C(13) | 121·0 (7) |
| C(4)–C(12)–C(11) | 120·5 (6) | C(6)–C(13)–C(14) | 119·9 (6) |
| C(12)–C(11)–C(1) | 118·0 (6) | C(13)–C(14)–C(9) | 116·2 (6) |
| C(4)–C(12)–S(5) | 114·9 (5) | C(6)–C(13)–S(5) | 116·1 (5) |
| C(11)–C(12)–S(5) | 124·6 (5) | C(14)–C(13)–S(5) | 124·0 (5) |
| C(1)–C(11)–N(10) | 119·8 (6) | C(12)–S(5)–C(13) | 102·6 (3) |
| C(12)–C(11)–N(10) | 122·2 (6) | C(11)–N(10)–C(14) | 123·0 (5) |
| C(9)–C(14)–N(10) | 121·5 (6) | C(11)–N(10)–C(15) | 119·3 (5) |
| C(13)–C(14)–N(10) | 122·3 (6) | C(14)–N(10)–C(15) | 117·5 (5) |
| C(16)–C(15)–N(10) | 113·5 (6) | C(17)–N(18)–C(19) | 110·8 (6) |
| C(15)–C(16)–C(17) | 107·8 (6) | C(17)–N(18)–C(20) | 113·3 (6) |
| C(16)–C(17)–N(18) | 112·7 (6) | C(19)–N(18)–C(20) | 110·5 (6) |
| C(1)–C(2)–Cl | 116·2 (6) | C(3)–C(2)–Cl | 118·2 (6) |

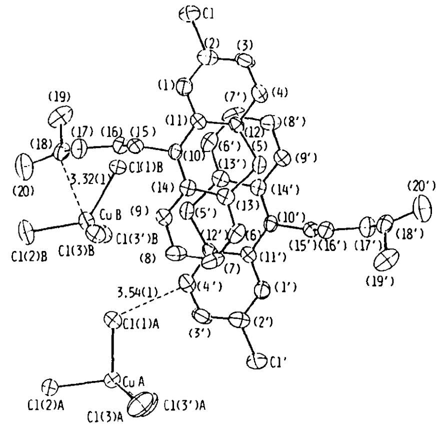


Fig. 1. The overlapping mode between the counterparts in the adjacent dimers, with atom numbering.

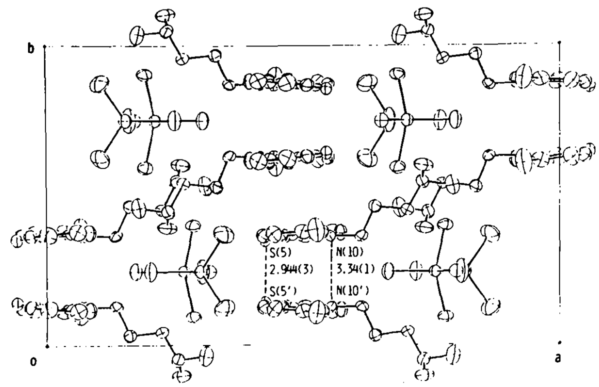


Fig. 2. *ORTEP* (Johnson, 1965) drawing of the **c** projection of the crystal. Hydrogens are omitted for clarity. (Distances in Å.)

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# Structure of Oxonium Tris(triethylammonium) Octamolybdate(4–) Dihydrate, $(C_6H_{16}N)_3(H_3O)[Mo_8O_{26}].2H_2O$*

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**Abstract.** $M_r = 1544{\cdot}8$, monoclinic, $P2_1/a$, $a = 21{\cdot}271$ (9), $b = 11{\cdot}837$ (1), $c = 20{\cdot}189$ (9) Å, $\beta = 117{\cdot}92$ (5)°, $V = 4491$ (3) Å$^3$, $Z = 4$, $D_x = 2{\cdot}29$ g cm$^{-3}$, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 21{\cdot}99$ cm$^{-1}$, $F(000) = 3008$, $T = 293$ K, $R = 0{\cdot}043$ for 9441 observed data. Two crystallographically independent octamolybdates are situated at the different inversion centers and have approximately the same structure as that of $\beta$-$[Mo_8O_{26}]^{4-}$. The $H_3O$ cation connects the two independent anions with complicated hydrogen bonds.

**Introduction.** Various alkylammonium polymolybdates reveal photochromic properties in the solid state (Yamase & Ikawa, 1977). From the crystal structure and ESR spectra of the three monoalkylammonium salts hexakis(isopropylammonium) dihydrogenoctamolybdate dihydrate (IPAM2), $(C_3H_{10}N)_6[H_2Mo_8O_{28}].2H_2O$ (Isobe, Marumo, Yamase & Ikawa, 1978; Yamase, 1978), hexakis(propylammonium) heptamolybdate trihydrate (PAM), $(C_3H_{10}N)_6[Mo_7O_{24}].3H_2O$, and hexakis(isopropylammonium) heptamolybdate trihydrate (IPAM), $(C_3H_{10}N)_6[Mo_7O_{24}].3H_2O$ (Ohashi, Yanagi, Sasada & Yamase, 1982; Yamase, 1982), it has been elucidated that the Mo atom is photoreduced from VI to V in an $MoO_6$ octahedral site, accompanying transfer of a hydrogen-bonding proton from the cation to the anion. In order to ascertain the mechanism for the trialkylammonium salts, the crystal structure of the title compound (TEAM) has been analyzed.

**Experimental.** Colorless prismatic crystals obtained by a similar method to that reported previously (Yamase & Ikawa, 1977); composition: C 13·94, H 3·76, N 3·57%; calculated for $(C_6H_{16}N)_3(H_3O)[Mo_8O_{26}].2H_2O$: C 13·99, H 3·11, N 2·72%; systematic absences: $h0l$ for $h = 2n + 1$, $0k0$ for $k = 2n + 1$; approximate dimensions of crystal 0·2 × 0·2 × 0·3 mm; Rigaku AFC-4 diffractometer, graphite monochromator, cell parameters refined by least squares on basis of 24 independent $2\theta$ values, $20 < 2\theta < 30°$; intensity measurement up to $2\theta = 55°$ ($\pm h+k+l$ set; $h$ 0–25, $k$ 0–15, $l$ 0–26), $\theta$–$2\theta$ scan, speed 2° min$^{-1}$($\theta$); 3 standard reflections showed intensity variation <5%; 10 834 reflections measured, 9441 intensities with $|F_o| > 3\sigma(|F_o|)$ considered observed and used for the structure determination; correction for Lorentz and polarization, absorption ignored; direct methods (*MULTAN*78, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and subsequent difference-Fourier calculation, block-diagonal least squares (*HBLS*, Ohashi, 1975), anisotropic thermal parameters for all non-H atoms; H atoms bonded to N atoms located on difference map and other H-atom positions obtained geometrically, $\sum w(|F_o|-|F_c|)^2$ minimized, $w = [\sigma^2(|F_o|) + (C|F_o|)^2]^{-1}$, $C$ adjusted so that constant values of $\langle w(|F_o|-|F_c|)^2\rangle$ obtained in different $|F_o|$ and $\sin\theta$ intervals, $C = 0{\cdot}015$; $R = 0{\cdot}043$, $R_w = 0{\cdot}056$ for 9441 observed reflections; atomic scattering factors including the anomalous

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