In this structure, both C—H distances [1·06 (1)–1·11 (1) Å] and O—H distances of water [0·91 (2)–0·97 (1) Å] were determined. The average C—H and O—H distances are 1·078 (2) and 0·943 (5) Å, respectively.

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Structure of Histaminium Tetraaquadisulfatomanganate(II) Monohydrate*

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Abstract. $C_5H_{11}N_3^{2+}.[Mn(SO_4)_2(H_2O)_4]^{2-}.H_2O$, M_r = 450·28, monoclinic, $P2_1/n$, a = 6·819 (1), b = 18·129 (2), c = 13·109 (2) Å, β = 93·74 (1)°, V = 1617·2 (5) Å^3, Z = 4, D_m = 1·86 (1), D_x = 1·849 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·418 mm^{-1}, $F(000)$ = 932, room temperature, R = 0·032 for 2275 observed reflections. The structure contains a histaminium dication in *syn–anti* conformation. Two monodentate sulfate ligands and four water molecules form a slightly distorted octahedral environment around the Mn^{2+} ion. The structure contains a complicated network of O—H···O, N—H···O and C—H···O hydrogen bonds.

Introduction. In the vicinity of some biomembranes the predominant form of histamine is a dication (Durrant, Ganellin & Parsons, 1975). Therefore some structures of histaminium salts have already been studied (Veidis, Palenik, Schaffrin & Trotter, 1969; Bonnet & Jeannin, 1972; Yamane, Ashida & Kakudo, 1973; Bonnet,

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* 4-(2-Ammonioethyl)imidazolium tetraaquadisulfatomanganate(II) monohydrate.

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Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	U_{eq}
Mn	0·57990 (8)	0·31270 (3)	0·47062 (4)	0·0223 (1)
O(1)	0·3176 (4)	0·3122 (1)	0·5540 (2)	0·041 (1)
S(1)	0·2179 (1)	0·25736 (5)	0·61421 (6)	0·0214 (2)
O(2)	0·3625 (4)	0·2201 (1)	0·6848 (2)	0·0354 (9)
O(3)	0·0708 (4)	0·2960 (1)	0·6711 (2)	0·0345 (9)
O(4)	0·1177 (4)	0·2029 (2)	0·5474 (2)	0·049 (1)
O(5)	0·4663 (3)	0·4134 (1)	0·4023 (2)	0·0291 (8)
S(2)	0·2838 (1)	0·43435 (5)	0·33999 (6)	0·0203 (2)
O(6)	0·2498 (4)	0·3840 (1)	0·2531 (2)	0·0344 (9)
O(7)	0·1175 (3)	0·4324 (1)	0·4062 (2)	0·0301 (8)
O(8)	0·3124 (4)	0·5102 (1)	0·3031 (2)	0·0283 (8)
N(1)	0·2409 (4)	0·4598 (2)	0·0648 (2)	0·029 (1)
C(2)	0·2782 (5)	0·5314 (2)	0·0748 (3)	0·028 (1)
N(3)	0·2771 (4)	0·5608 (2)	−0·0177 (2)	0·0249 (9)
C(4)	0·2397 (5)	0·5063 (2)	−0·0904 (3)	0·022 (1)
C(5)	0·2163 (5)	0·4431 (2)	−0·0379 (3)	0·027 (1)
C(6)	0·2330 (5)	0·5250 (2)	−0·2019 (3)	0·026 (1)
C(7)	0·2252 (6)	0·4561 (2)	−0·2672 (3)	0·028 (1)
N(8)	0·2227 (4)	0·4757 (2)	−0·3784 (2)	0·029 (1)
O*W*(1)	0·4735 (4)	0·2290 (2)	0·3640 (2)	0·0375 (9)
O*W*(2)	0·6963 (4)	0·2167 (1)	0·5662 (2)	0·0304 (8)
O*W*(3)	0·8419 (4)	0·3182 (1)	0·3832 (2)	0·0331 (9)
O*W*(4)	0·7672 (4)	0·3779 (1)	0·5765 (2)	0·0417 (9)
O*W*(5)	0·4156 (4)	0·6825 (2)	0·1693 (2)	0·045 (1)

Table 2. *Selected bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Mn—O(1)	2·156 (3)	Mn—O*W*(2)	2·259 (2)
Mn—O(5)	2·156 (2)	Mn—O*W*(3)	2·187 (2)
Mn—O*W*(1)	2·156 (3)	Mn—O*W*(4)	2·174 (3)
S(1)—O(1)	1·464 (3)	N(1)—C(2)	1·327 (5)
S(1)—O(2)	1·471 (2)	C(2)—N(3)	1·325 (4)
S(1)—O(3)	1·466 (3)	N(3)—C(4)	1·385 (4)
S(1)—O(4)	1·459 (3)	C(4)—C(5)	1·351 (5)
S(2)—O(5)	1·492 (2)	C(5)—N(1)	1·380 (4)
S(2)—O(6)	1·467 (3)	C(4)—C(6)	1·498 (5)
S(2)—O(7)	1·473 (2)	C(6)—C(7)	1·512 (5)
S(2)—O(8)	1·475 (2)	C(7)—N(8)	1·499 (4)

O(1)—Mn—O(5)	85·8 (1)	O*W*(2)—Mn—O*W*(3)	93·2 (1)
O(1)—Mn—O*W*(1)	94·0 (1)	O*W*(2)—Mn—O*W*(4)	83·9 (1)
O(1)—Mn—O*W*(2)	89·3 (1)	O*W*(3)—Mn—O*W*(4)	81·0 (1)
O(1)—Mn—O*W*(3)	177·3 (1)	C(5)—N(1)—C(2)	108·7 (3)
O(1)—Mn—O*W*(4)	98·7 (1)	N(1)—C(2)—N(3)	108·3 (3)
O(5)—Mn—O*W*(1)	103·3 (1)	C(2)—N(3)—C(4)	109·4 (3)
O(5)—Mn—O*W*(2)	170·8 (1)	N(3)—C(4)—C(5)	106·1 (3)
O(5)—Mn—O*W*(3)	91·6 (1)	C(4)—C(5)—N(1)	107·5 (3)
O(5)—Mn—O*W*(4)	89·1 (1)	N(3)—C(4)—C(6)	120·2 (3)
O*W*(1)—Mn—O*W*(2)	84·8 (1)	C(5)—C(4)—C(6)	133·7 (3)
O*W*(1)—Mn—O*W*(3)	86·8 (1)	C(4)—C(6)—C(7)	111·3 (3)
O*W*(1)—Mn—O*W*(4)	162·9 (1)	C(6)—C(7)—N(8)	110·7 (3)

Jeannin & Laaouini, 1975; Wojtczak, Jaskólski & Kosturkiewicz, 1988). The present investigation has been undertaken as part of our studies on histamine compounds.

Experimental. Crystals from water, density by flotation, 15 reflections with $20 < 2\theta < 28°$ used to obtain lattice parameters, 2744 unique reflections $2\theta \leq 50°$ (h: $-8 \rightarrow 8$, k: $0 \rightarrow 21$, l: $0 \rightarrow 15$) measured on a Syntex $P2_1$ diffractometer, crystal 0·35 × 0·45 × 0·35 mm, graphite-monochromated Mo $K\alpha$ radiation, $\lambda =$ 0·71069 Å, profile analysis according to Lehmann & Larsen (1974), no significant intensity variation for $2\bar{9}\bar{4}$ and $3\bar{4}\bar{2}$ check reflections monitored every 100 reflections, 2275 observed reflections $I \geq 2\sigma(I)$, no absorption correction. Structure solved by Patterson method, $\sum w(\Delta F)^2$ minimized in full-matrix least-squares refinement using *SHELX*76 (Sheldrick, 1976), $w = \sigma^{-2}(F)$ from counting statistics, H-atom positions from $\Delta\rho$ map and not refined, extinction $x = 0{\cdot}00262\,(14)$, $F_c' = F_c(1 - 0{\cdot}0001xF_c^2/\sin\theta)$, final $R = 0{\cdot}032$, $wR = 0{\cdot}040$, $S = 1{\cdot}427$, $|\Delta/\sigma|_{max}$ in the final refinement 0·011, $\Delta\rho_{max} = 0{\cdot}32$, $\Delta\rho_{min} = -0{\cdot}32$ e Å^{-3}, scattering factors from *International Tables for X-ray Crystallography* (1974). Other programs: *ORTEP*II (Johnson, 1976), *PLUTO* (Motherwell & Clegg, 1978) and local programs (Jaskólski, 1982). RIAD-32 computer.

Discussion. The atomic parameters, selected bond lengths and angles are given in Tables 1 and 2.* The atom-numbering scheme is shown in Fig. 1.

The geometry of the imidazolium ring is similar to that found in other histaminium dications. However, the N—C bonds in the N—C—N fragment [1·327 (4), 1·325 (4) Å] are relatively long and are among the longest ring N—C bonds observed in histaminium

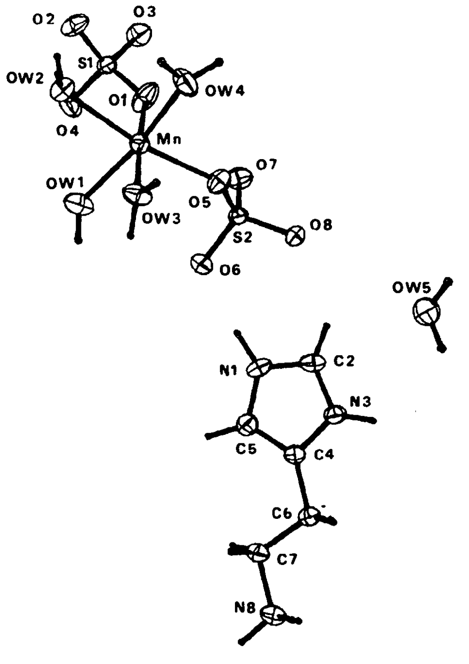

Fig. 1. A perspective view of the ions with atomic numbering scheme.

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, H-atom parameters, details of H bonds and best planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51276 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

dications (Veidis *et al.*, 1969; Bonnet *et al.*, 1972, 1975; Yamane *et al.*, 1973; Wojtczak *et al.*, 1988). On the other hand these bonds are similar to those found in substituted derivatives of histaminium dications (Nardelli, Pelizzi, Vitali, Bordi, Plazzi & Vitali, 1987) as well as in imidazolium cations (Blessing & McGandy, 1972; Freeman, Huq, Rosalky & Taylor, 1975). The imidazolium ring is planar [$\sum(\Delta/\sigma)^2$ 2·568].

The bond lengths and angles in the side chain are similar to those found in other histamines.

Most of the histaminium dications studied so far exist in the *syn–anti* conformation. However, in histaminium dinitrate the *syn–syn* conformation was found (Wojtczak, Jaskólski & Kosturkiewicz, 1988). In the present structure, the histaminium dication exists in the usual *syn–anti* conformation, the C(4)—C(6)—C(7)—N(8) and C(5)—C(4)—C(6)—C(7) torsion angles being −178·8 (3) and −9·6 (4)°, respectively.

The two sulfate ions act as monodentate ligands and form Mn—O(1) and Mn—O(5) bonds [both distances 2·156 (3) Å]. The S(1) sulfate ion has a slightly distorted tetrahedral geometry, the S—O distances varying from 1·459 (3) to 1·471 (2) Å. The distortion of the S(2) sulfate ion is more pronounced, the S(2)—O(5) bond being longer [1·492 (2) Å] than the other S(2)—O bonds [1·467 (3) to 1·475 (2) Å]. The O—S—O angles in both sulfate ions are close to the tetrahedral value and vary from 107·1 (2) to 110·6 (2)°.

The coordination sphere of the Mn^{2+} ion (distorted octahedron) is formed by two monodentate sulfate ligands bonded in *cis* positions and four water molecules. The Mn—O distances vary from 2·156 (3) to 2·259 (2) Å and the O—Mn—O angles from 81·0 (1) to 103·3 (1)° and from 162·9 (1) to 177·3 (1)°. Such a deformation seems to be due to intra- and intermolecular hydrogen bonds involving all ligands (Fig. 2).

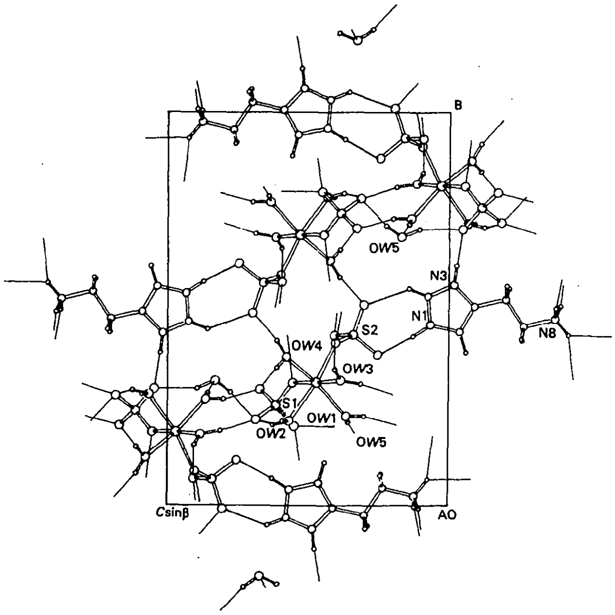

Fig. 2. Projection of the structure down the *a* axis showing hydrogen bonds.

Within the coordination sphere an intramolecular O(2)···H—O*W*(2) hydrogen bond is observed with an O···O distance of 2·839 (4) Å. The histaminium N(1)—H, C(2)—H and N(3)—H ring groups are H-bond donors, the N(1)···O(6), C(2)···O(8) and N(3)···O(4) (0·5−*x*, 0·5+*y*, 0·5−*z*) distances being 2·822 (4), 3·011 (4) and 2·709 (4) Å, respectively. The ammonium group of the histaminium side chain acts as a donor in three H bonds. One of them, formed by the N(8)—H(82) group, is bifurcated, the N(8)···O(1) (*x*, *y*, −1+*z*) and N(8)···O(7) (*x*, *y*, −1+*z*) distances being 3·173 (4) and 2·974 (4) Å, respectively. The remaining bonds are N(8)···O(7) (−*x*, 1−*y*, −*z*) [2·860 (4) Å] and N(8)···O(5) (1−*x*, 1−*y*, −*z*) [2·953 (4) Å]. The O*W*(2)—H*W*(22) group is a donor in a bifurcated hydrogen bond, the O*W*(2)···O(3) (1+*x*, *y*, *z*) and O*W*(2)···O(4) (1+*x*, *y*, *z*) distances being 3·168 (4) and 2·911 (4) Å, respectively. The remaining O*W*—H*W* groups are single H-bond donors. Of the water oxygen atoms, only O*W*(5) is a hydrogen-bond acceptor, the O*W*(5)···O*W*(1) (0·5−*x*, 0·5+*y*, 0·5−*z*) distance being 2·791 (4) Å. All the above *D*—H···*A* contacts fulfil the H···*A* criterion (Hamilton, 1968) and the *D*—H···*A* angular criterion (Jaskólski, 1984) for H bonds.

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Structure of Tetraphenylphosphonium Trichlorocuprate

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Abstract. $[P(C_6H_5)_4][CuCl_3]$, $M_r = 509{\cdot}3$, monoclinic, $P2_1/n$, $a = 13{\cdot}601\,(2)$, $b = 19{\cdot}272\,(3)$, $c = 9{\cdot}216\,(2)$ Å, $\beta = 107{\cdot}9\,(1)°$, $V = 2297{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}47$ g cm^{-3}, $\lambda(\mathrm{Ag}\,K\alpha) = 0{\cdot}56087$ Å, $\mu = 7{\cdot}1$ cm^{-1}, $F(000) = 1036$, $T = 298$ K, $R = 0{\cdot}049$, $wR = 0{\cdot}062$ for 1432 reflexions $>2\sigma(F)$. The structure consists of segregated stacks of the non planar $[Cu_2Cl_6]^{2-}$ anions and of the respective cations, $[Ph_4P]^+$. The isolated copper dimers, $[Cu_2Cl_6]^{2-}$, are bibridged over the inversion center. The average Cu—Cl bond lengths are 2·191 (terminal) and 2·310 Å (bridging) with bridging Cu—Cl—Cu angle averaging 93·1°. The P atom in the tetraphenylphosphonium cation, $[Ph_4P]^+$, is tetrahedrally coordinated by four phenyl C atoms with $\langle P—C\rangle = 1{\cdot}796$ Å; the phenyl rings of the tetraphenylphosphonium are planar, with $\langle C—C\rangle$ in the four phenyl rings ranging from 1·384 to 1·389 Å.

Introduction. Halide transition-metal complexes aroused particular interest owing to their magnetic properties which are good examples for magnetostructural correlation in exchange coupled systems. Thus, $[N(CH_3)_4][MnCl_3]$ constitutes a typical example where the antiferromagnetic behavior of manganese ions in the $MnCl_3$ chain is entirely predictable by the Heisenberg interaction model and to some lesser extent the observed ferromagnetic exchange between Cu ions in $[N(CH_3)_4][CuCl_3]$ can also be understood by the same theoretical basis (Landée & Willett, 1979; Peersy, Morigin & Smara, 1973).

More recently another copper complex, piperazinium hexachlorodicuprate, has been synthesized and its crystal structure determined; in this compound the trichlorocuprate groups are condensed into infinite $[Cu_2Cl_6]^{2-}$ chains zigzagging along the *a* axis. Magnetic susceptibility as a function of temperature, maximum at $T = 15$ K and then tending to zero, has been successfully predicted, $S = \frac{1}{2}$, as an alternating antiferromagnetic exchange with a relatively small value of J_1/K, $-12{\cdot}5$ K, and with an alternation parameter, (J_2/J_1) close to 0·6, in good agreement with the structural data (Daoud, Ben Salah, Chappert, Renard, Cheikhrouhou, Tran Qui & Verdaguer, 1986). In a continuation of magnetostructural correlation studies of transition-metal complexes a new tetraphenylphosphonium chlorocuprate, $[P(Ph)_4][CuCl_3]$, is synthetized and its crystal structure is reported in this paper.

Experimental. Red-yellow needle-shaped crystals of the title compound were grown by slow evaporation at ambient temperature in an aqueous saturated equimolar solution of $(Ph)_4PCl$ and $CuCl_2$. Several recrystallizations in saturated HCl solution were necessary to obtain single crytals suitable for X-ray analysis: CAD-4 diffractometer, crystal size $0{\cdot}08 \times 0{\cdot}07 \times 0{\cdot}14$ mm, $\lambda(\mathrm{Ag}\,K\alpha)$, random orientation, no absorption correction, ω scan, 2° min^{-1}, scan range $= 1{\cdot}1°$, $2\theta_{\max} = 20°$, $-15 \le h \le 15$, $-23 \le k \le 23$, $0 \le l \le 11$. Unit-cell parameters from 24 reflexions with $10 \le 2\theta \le 18°$, 3672 reflexions measured, averaged to 1432 unique reflexions $\ge 2\sigma(F)$, internal agreement factor was 3·7%. Three standard reflexions, no intensity variation.

The structure was solved using the Patterson heavy-atom method which revealed the positions of Cu, Cl and P atoms. The remaining atoms were located by succeeding difference Fourier syntheses. H positions, unsuccessfully determined by the Fourier method, were assigned according to the phenyl geometry. Three final cycles including H positions showed no improvement in

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