almost in the plane formed by C(8)—C(15)—C(16)—C(13). There are two intermolecular hydrogen bonds between the neighbouring molecules. Bond distances O(1^i)···O(2^{ii}), O(2^i)···O(4^{iii}) and bond angles O(1^i)—H(33^i)···O(2^{ii}), O(2^i)—H(34^i)···O(4^{iii}) are found as 2.804 (6), and 2.823 (7) Å, and 178 (7) and 168 (6)°, respectively. The symmetry codes are: (i) x, y, z; (ii) $-x + 1, y + \frac{1}{2}, -z + 1$; (iii) $-x, y - \frac{1}{2}, -z + 1$.

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Structure of Oxmetidine Dihydrochloride (SKF 92994)

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Abstract. 5-(1,3-Benzodioxol-5-ylmethyl)-2-[2-(5-methyl-1H-imidazol-4-ylmethylthio)ethylamino]-4(1H)-pyrimidone dihydrochloride, $C_{19}H_{23}N_5O_3S^{2+}.2Cl^-$, $M_r = 472.4$, triclinic, $P\bar{1}$, $a = 8.291$ (2), $b = 10.759$ (3), $c = 13.169$ (3) Å, $\alpha = 108.42$ (2), $\beta = 93.28$ (2), $\gamma = 100.3$ (3)°, $V = 1088.6$ Å^3, $Z = 2$, $D_x = 1.44$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.42$ mm^{-1}, $F(000) = 492$, $T = 294$ K, final $R = 0.038$ for 3184 unique observed reflexions. All bonded distances and interbond angles lie within the range of values for compounds of a similar nature. The planar imidazole and isocytosine groups are connected by a four-atom folded methylthioethyl chain with *gauche* linkages at the S atom. Intermolecular bonding includes N—H···Cl hydrogen bonds and van der Waals forces.

Introduction. Oxmetidine (1) was identified as a highly effective histamine H2 antagonist inhibiting gastric acidity in man and effective in the treatment of duodenal ulcers. The compound was withdrawn in the later stages of clinical development owing to a

low incidence of side effects (Brown, Blakemore, Durant, Emmett, Ganellin, Parsons, Rawlings & Walker, 1988).
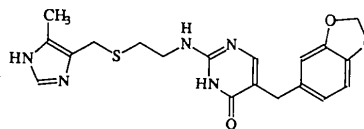
(1)

Experimental. The compound was prepared at Smith Kline and French Research Ltd, and was characterized by elemental analysis, proton NMR, and infrared and mass spectrometry (Brown *et al.*, 1988). The crystals were prepared in Oxford by several recrystallizations from a mixture of *n*-propanol and water. After preliminary X-ray precession and Weissenberg photography the X-ray data were collected with an Enraf–Nonius CAD-4 diffractometer from a crystal of size 0.5 × 0.5 × 0.3 mm, following the manufacturer's recommended procedures to optimize the data collection parameters. Lattice parameters were obtained by the least-squares best fit of 25

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Table 1. *Positional parameters and equivalent isotropic temperature factors* (Å^2)

	x	y	z	U_{eq}
C(1)	0.8263 (4)	0.0424 (3)	0.1490 (2)	0.0419
C(2)	0.7135 (3)	0.0100 (2)	0.2252 (2)	0.0315
N(3)	0.7714 (3)	-0.0097 (2)	0.3185 (2)	0.0371
C(4)	0.6470 (4)	-0.0337 (3)	0.3731 (2)	0.0423
N(5)	0.5109 (3)	-0.0305 (2)	0.3185 (2)	0.0363
C(6)	0.5478 (3)	-0.0026 (2)	0.2255 (2)	0.0296
C(7)	0.4156 (3)	0.0068 (3)	0.1488 (2)	0.0353
S(8)	0.4518 (1)	0.1636 (1)	0.1205 (1)	0.0380
C(9)	0.4594 (4)	0.2851 (3)	0.2527 (2)	0.0362
C(10)	0.2895 (4)	0.2955 (3)	0.2847 (2)	0.0365
N(11)	0.2983 (3)	0.4099 (2)	0.3828 (2)	0.0337
C(12)	0.2239 (3)	0.4080 (2)	0.4683 (2)	0.0280
N(13)	0.2140 (3)	0.5247 (2)	0.5428 (2)	0.0307
C(14)	0.1335 (3)	0.5386 (3)	0.6348 (2)	0.0308
O(15)	0.1242 (3)	0.6498 (2)	0.6922 (2)	0.0429
C(16)	0.0642 (3)	0.4135 (3)	0.6509 (2)	0.0300
C(17)	0.0753 (3)	0.2994 (3)	0.5752 (2)	0.0329
N(18)	0.1530 (3)	0.2953 (2)	0.4846 (2)	0.0320
C(19)	-0.0252 (3)	0.4156 (3)	0.7481 (2)	0.0370
C(20)	0.0869 (3)	0.4318 (3)	0.8490 (2)	0.0317
C(21)	0.1803 (4)	0.5573 (3)	0.9115 (2)	0.0379
C(22)	0.2720 (4)	0.5656 (3)	1.0039 (2)	0.0377
O(23)	0.3685 (3)	0.6781 (2)	1.0797 (2)	0.0558
C(24)	0.4408 (4)	0.6333 (3)	1.1588 (2)	0.0472
O(25)	0.3741 (3)	0.4941 (2)	1.1325 (2)	0.0488
C(26)	0.2757 (4)	0.4565 (3)	1.0354 (2)	0.0373
C(27)	0.1869 (4)	0.3321 (3)	0.9760 (3)	0.0449
C(28)	0.0913 (4)	0.3221 (3)	0.8812 (2)	0.0399
Cl(29)	0.1445 (1)	-0.0071 (1)	0.3688 (1)	0.0315
Cl(30)	0.5760 (1)	0.2762 (1)	0.5469 (1)	0.0439

reflexions, $12 < 2\theta < 28°$; and intensity data were collected using an ω–2θ scan, for maximum $\sin\theta/\lambda = 0.66\ \text{Å}^{-1}$, and $h\ -10 \rightarrow 10$, $k\ -13 \rightarrow 13$, $l\ -1 \rightarrow 17$. Three standard reflexions, measured after every 100 reflexions, showed no significant variations during the data collection. 6425 reflexions were measured of which 5234 were unique ($R_{int} = 0.020$). Of these, 3184 with $I > 3\sigma(I)$ were used in the refinement. Absorption was ignored. The structure was solved by direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined by the least-squares method minimizing $\Sigma w|\Delta F|^2$ first for full matrix with the non-H atoms with isotropic temperature factors, then with a five-block approximation to the normal matrix [(i) C(1, x, y, z) to C(30), (ii) H(1, x, y, z) to H(23), (iii) C(1, U_{ij}) to C(15), (iv) C(16, U_{ij}) to Cl(30), (v) dummy U_{iso} and scale] and a four-term Chebyshev polynomial weighting scheme (Carruthers & Watkin, 1979) with coefficients -1.9, -8.6, -9.9 and -4.6. After the final refinement cycle $R = 0.0376$, $wR = 0.0449$ and $(\Delta/\sigma)_{max} = 2.68$, and from the final difference electron density map $\Delta\rho_{max} = 0.39\ \text{e}\ \text{Å}^{-3}$. Extinction effects appeared to be negligible.* Atomic scattering factors were taken from *International Tables for*

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* Lists of anisotropic displacement parameters, H-atom coordinates, bond distances involving H atoms, torsion angles, least-squares-planes data, intra- and intermolecular contacts, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55327 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0106]

Table 2. *Bond distances* (Å), *bond angles* (°), *selected torsion angles* (°), *intermolecular contacts* (Å) *and intramolecular distances* (Å)

C(1)—C(2)	1.488 (4)	C(16)—C(17)	1.337 (4)
C(2)—N(3)	1.385 (3)	C(17)—N(18)	1.380 (3)
N(3)—C(4)	1.324 (4)	N(18)—C(12)	1.337 (3)
C(4)—N(5)	1.315 (4)	C(16)—C(19)	1.512 (4)
N(5)—C(6)	1.387 (3)	C(19)—C(20)	1.521 (4)
C(6)—C(2)	1.357 (3)	C(20)—C(21)	1.397 (4)
C(6)—C(7)	1.486 (4)	C(21)—C(22)	1.368 (4)
C(7)—S(8)	1.818 (3)	C(22)—O(23)	1.378 (3)
S(8)—C(9)	1.806 (3)	O(23)—C(24)	1.424 (4)
C(9)—C(10)	1.507 (4)	C(24)—O(25)	1.422 (4)
C(10)—N(11)	1.464 (3)	O(25)—C(26)	1.379 (3)
N(11)—C(12)	1.318 (3)	C(26)—C(22)	1.367 (4)
C(12)—N(13)	1.347 (3)	C(26)—C(27)	1.366 (4)
N(13)—C(14)	1.396 (3)	C(27)—C(28)	1.403 (4)
C(14)—O(15)	1.215 (3)	C(28)—C(20)	1.379 (4)
C(14)—C(16)	1.451 (4)		

C(1)—C(2)—N(3)	122.0 (2)	C(14)—C(16)—C(17)	118.0 (2)
C(1)—C(2)—C(6)	132.0 (2)	C(14)—C(16)—C(19)	119.7 (2)
C(6)—C(2)—N(3)	105.9 (2)	C(17)—C(16)—C(19)	122.3 (2)
C(2)—N(3)—C(4)	110.0 (2)	C(16)—C(17)—N(18)	123.2 (2)
N(3)—C(4)—N(5)	107.7 (2)	C(17)—N(18)—C(12)	121.0 (2)
C(4)—N(5)—C(6)	109.8 (2)	C(16)—C(19)—C(20)	114.2 (2)
N(5)—C(6)—C(2)	106.5 (2)	C(19)—C(20)—C(21)	120.7 (2)
N(5)—C(6)—C(7)	121.0 (2)	C(19)—C(20)—C(28)	119.5 (2)
C(2)—C(6)—C(7)	132.5 (2)	C(21)—C(20)—C(28)	119.8 (2)
C(6)—C(7)—S(8)	113.7 (2)	C(20)—C(21)—C(22)	117.5 (2)
C(7)—S(8)—C(9)	102.0 (1)	C(21)—C(22)—O(23)	128.0 (3)
S(8)—C(9)—C(10)	112.1 (2)	C(21)—C(22)—C(26)	122.4 (3)
C(9)—C(10)—N(11)	110.7 (2)	O(23)—C(22)—C(26)	109.6 (2)
C(10)—N(11)—C(12)	125.9 (2)	C(22)—O(23)—C(24)	106.2 (2)
N(11)—C(12)—N(13)	119.2 (2)	O(23)—C(24)—O(25)	108.1 (2)
N(11)—C(12)—N(18)	123.5 (2)	C(24)—O(25)—C(26)	105.9 (2)
N(13)—C(12)—N(18)	117.3 (2)	O(25)—C(26)—C(27)	128.1 (3)
C(12)—N(13)—C(14)	125.8 (2)	O(25)—C(26)—C(22)	110.1 (2)
N(13)—C(14)—C(16)	114.7 (2)	C(22)—C(26)—C(27)	121.7 (3)
N(13)—C(14)—O(15)	119.4 (2)	C(26)—C(27)—C(28)	116.6 (3)
O(15)—C(14)—C(16)	125.9 (2)	C(27)—C(28)—C(20)	122.0 (3)

C(2)—C(6)—C(7)—S(8)	-55.7 (3)	N(5)—C(6)—C(7)—S(8)	125.2 (3)
C(6)—C(7)—S(8)—C(9)	-60.2 (3)	C(7)—S(8)—C(9)—C(10)	-79.9 (3)
S(8)—C(9)—C(10)—N(11)	-170.2 (3)	C(9)—C(10)—N(11)—C(12)	-132.0 (4)
C(10)—N(11)—C(12)—N(13)	-166.1 (4)	C(10)—N(11)—C(12)—N(18)	12.3 (4)
C(17)—C(16)—C(19)—C(20)	97.0 (4)	C(14)—C(16)—C(19)—C(20)	-86.5 (4)
C(16)—C(19)—C(20)—C(21)	78.4 (4)	C(16)—C(19)—C(20)—C(28)	-104.1 (4)

Cl(29)···N(3')	3.120 (3)	Cl(30)···N(13")	3.085 (3)
Cl(30)···N(11")	3.164 (3)		

N(3)···N(11)	6.384 (4)	N(5)···N(11)	5.185 (4)
N(3)···N(13)	7.965 (4)	N(5)···N(13)	6.738 (4)
N(3)···N(18)	6.677 (4)	N(5)···N(18)	5.048 (4)

Symmetry code: (i) $x - 1$, y, z; (ii) $-x + 1$, $-y + 1$, $-z + 1$.

X-ray Crystallography (1974, Vol. IV, p. 202). The *CRYSTALS* program was used for data reduction and refinement (Watkin, Carruthers & Betteridge, 1985).

Discussion. Atomic coordinates and molecular geometries are reported in Tables 1 and 2, Fig. 1 shows the atomic numbering scheme and molecular conformation, and Fig. 2 the crystal packing.

The crystal structure consists of oxmetidine cations and Cl anions in a ratio of 1:2. There are three crystallographically distinct N—H···Cl hydrogen bonds. Cl(29) hydrogen bonds to N(3), and Cl(30) forms hydrogen bonds to N(11) and N(13) so that each cation hydrogen bonds two Cl anions to form a three-ion unit with an overall zero charge. There are no hydrogen-bonding interactions between the cations.
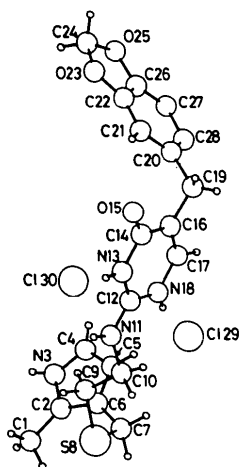
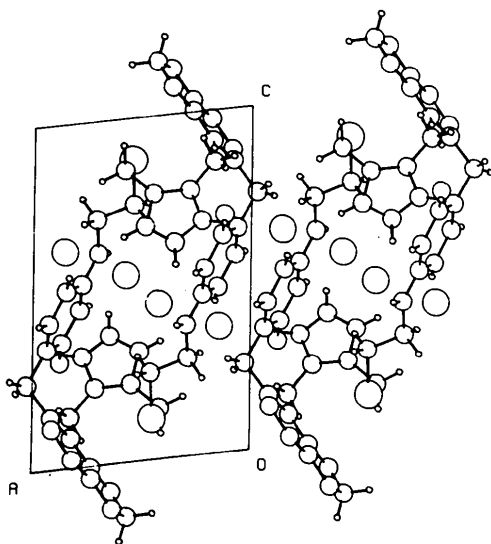Fig. 1. The molecular conformation of oxmetidine dihydrochloride and atomic numbering.

Fig. 2. Crystal structure in projection down **b**.

The aromatic group, 4-methylimidazolyl, is planar, and the methylthioethyl chain has the expected folded conformation with *gauche* linkages (both in the same sense) at C(7)—S(8) and S(8)—C(9). Similar *gauche* linkages at S atoms have been observed for the methylthioethyl chain in all H2 antagonists in the solid state except for cimetidine monohydrate (Kojić-Prodić, Ružić-Toroš, Bresciani-Pahor & Randaccio, 1980) and for famotidine (Yanagisawa, Hirata & Ishii, 1987). The isocytosine residue is planar. In the dication in (1), the isocytosine is protonated on both ring N atoms and the problem of a tautomeric form does not arise. Examination of the Cambridge Structural Database indicates that in six-membered rings, protonated N atoms have endocyclic angles > 120° and unprotonated N atoms endocyclic angles < 120°. Further, N atoms in peptide-like linkages form C—N—C angles of about 124°, significantly > 120°. Thus the expectation is that the dication should have both endocyclic angles at the N atoms > 120°. This is observed in (1) (Table 2).

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Structure of 2,3-Naphthalenediamine Dihydrochloride Dihydrate

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Abstract. $C_{10}H_{12}N_2^{2+}.2Cl^-.2H_2O$, $M_r = 267.15$, monoclinic, $P2_1/c$, $a = 7.057$ (2), $b = 13.017$ (3), $c = 13.982$ (3) Å, $\beta = 92.57$ (4)°, $V = 1283.1$ (5) Å^3, $Z = 4$, $D_x = 1.382$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 4.95$ cm^{-1}, $F(000) = 560$, $T = 298$ K, final $R = 0.036$, $wR = 0.026$ for 1814 uniquely observed $[I \geq 2\sigma(I)]$