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Structure of 7-Chloro-2,3-dihydro-1-methyl-5-phenyl-1*H*-1,4-benzodiazepine Hydrochloride (Medazepam Hydrochloride)

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Abstract

Medazepam hydrochloride, $C_{16}H_{16}ClN_2^+.Cl^-.H_2O$, crystallizes as the monohydrate from aqueous solution in the monoclinic space group $P2_1/c$ with a = 10·835 (8), b = 18·57 (1), c = 8·19 (1) Å, β = 96·15 (5)°, Z = 4. The structure was refined to R = 5·1% for 1622 observed counter amplitudes. Protonation occurs at N(4). Comparison with the crystal structure of medazepam base shows that protonation leads to an increase of some 7° in the angle at N(4) and a decrease of 4° in the endocyclic angle at the adjacent C(5). The angle between the 5-phenyl ring and the C(6)–(11) phenyl moiety of the 1,4-benzodiazepine system is 67°, slightly greater than in the free base.

Introduction

Medazepam (Sternbach, Reeder & Archer, 1963) exhibits only to a moderate degree the pharmacological characteristics typical of many 5-phenyl-1,4-benzodiazepines such as anxiolytic and muscle-relaxant activity (Randall, Schallek, Sternbach & Ning, 1974). Chemically, it differs from the more active diazepam (and other more active analogues) in the absence of a carbonyl group in the 2-position. The crystal structure of medazepam base has been determined (Gilli, Bertolasi, Sacerdoti & Borea, 1978) and we now report the structure of the hydrochloride salt as part of a study of possible correlations between molecular geometry and biological activity for 1,4-benzodiazepine derivatives. Earlier studies of structure–activity relationships for this class of compounds have been described by Camerman & Camerman (1974).

Experimental

Medazepam base obtained from Hoffmann–La Roche was treated with a slight excess of hydrochloric acid and bright orange-red crystals were obtained from the aqueous solution. A crystal *ca* 0·5 × 0·3 × 0·2 mm was mounted along the direction of elongation which coincided with c. After initial examination by photographic methods, final cell dimensions and intensities were measured on a Stoe two-circle computer-controlled diffractometer with graphite-monochromated Mo $K\alpha$ radiation and a scintillation counter. The ω-scan technique was employed with a stepping interval of 0·01° and a step time of 1 s. For layers $hk0$–$hk3$, the scan range was 1·4° and for the higher layers (equi-inclination angle $\mu > 7·5°$) a variable scan range was used, $\Delta\omega$ being calculated from $[A + (B \sin\mu/\tan\theta')]°$ where $2\theta'$ is the azimuth angle and A and B were assigned values of 1·0 and 0·5, respectively. Backgrounds for all reflexions were measured for 30 s at each end of the scan. After each layer of data collection, four standard reflexions on the zero layer were remeasured. There was no significant variation of intensity.

Of 3093 reflexions scanned within the range $0·1 < \sin\theta/\lambda < 0·6$ Å^{-1}, 1622 for which $I > 2·5\sigma(I)$ were considered to be observed and were used in the structure analysis. Absorption corrections were not applied.

Crystal data

$C_{16}H_{16}ClN_2^+.Cl^-.H_2O$, M_r = 325·2. Monoclinic, a = 10·835 (8), b = 18·57 (1), c = 8·19 (1) Å, β = 96·15 (5)°, U = 1638 Å^3, D_c = 1·318 Mg m^{-3}, Z = 4. $F(000)$ = 680. Systematic absences: $h0l$ when l is odd, $0k0$ when k is odd, space group $P2_1/c$. Mo $K\alpha$ radiation, λ = 0·71069 Å. μ(Mo $K\alpha$) = 0·34 mm^{-1}.

Determination of the structure

The structure was solved by direct methods with *SHELX* (Sheldrick, 1976). An E map based on 590

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	*y*	*z*
Cl(7)	−4377 (1)	−228 (1)	7809 (1)
Cl^-	−3 (1)	3251 (1)	3140 (1)
C(2)	−233 (4)	817 (2)	2499 (5)
C(3)	−85 (4)	1297 (2)	4001 (5)
C(5)	−2192 (4)	1632 (2)	4388 (4)
C(6)	−3172 (4)	688 (2)	5939 (5)
C(7)	−3488 (4)	−22 (2)	6203 (5)
C(8)	−3131 (4)	−559 (2)	5184 (6)
C(9)	−2451 (4)	−386 (2)	3912 (5)
C(10)	−2098 (4)	320 (2)	3607 (4)
C(11)	−2475 (3)	866 (2)	4661 (4)
C(12)	−1571 (4)	13 (3)	818 (5)
C(1′)	−3164 (4)	2189 (2)	4475 (4)
C(2′)	−4398 (4)	2028 (2)	3938 (5)
C(3′)	−5293 (4)	2561 (3)	3893 (6)
C(4′)	−4977 (5)	3251 (3)	4426 (6)
C(5′)	−3762 (5)	3413 (2)	4969 (5)
C(6′)	−2843 (4)	2886 (2)	4995 (4)
N(1)	−1479 (3)	505 (2)	2243 (4)
N(4)	−1115 (3)	1810 (2)	4000 (4)
O(*w*)	1952 (4)	2269 (4)	1342 (6)
H1(2)	25	1183	1611
H2(2)	514	472	2904
H1(3)	19	1033	4998
H2(3)	620	1587	4081
H(4)	−916	2307	3685
H(6)	−3475	1020	6596
H(8)	−3406	−1053	5497
H(9)	−2139	−718	3170
H1(12)	−2433	−196	766
H2(12)	−1025	−419	1151
H3(12)	−1226	244	−444
H(2′)	−4594	1528	3502
H(3′)	−6104	2409	3593
H(4′)	−5761	3620	4264
H(5′)	−3281	3875	5456
H(6′)	−1948	2980	5265
H(O*w*)	1449	2860	5045

reflexions ($E > 1{\cdot}2$) revealed the positions of the Cl^- ion and all non-hydrogen atoms of the medazepam cation. Refinement was carried out by least-squares calculations, first with isotropic then anisotropic thermal parameters. H atoms were located from a difference synthesis and were included with isotropic temperature factors, but their coordinates were not refined. When the refinement had appeared to converge, with all calculated shifts $<0{\cdot}1\sigma$, R was still unexpectedly high (12·2%). This suggested that one or more atoms had been omitted from the model. A peak of *ca* 3e $Å^{-3}$ present in the various difference maps, computed during the refinement process, but hitherto ignored, was now included in the calculations as an O atom of a possible molecule of water of crystallization. Only one of the H atoms of the water molecule could, however, be located. Initially the O atom was included with an isotropic temperature factor and its site occupation factor was also allowed to vary. This refined to a value very close to 1·0 with an isotropic temperature factor $U = 0{\cdot}16$ $Å^2$. A difference synthesis, however, indicated that the O atom exhibited strong anisotropic thermal motion, and in subsequent calculations its site occupation factor was fixed at unity and it was allowed to vibrate anisotropically. Refinement was terminated when all calculated shifts were $<0{\cdot}1\sigma$, and R was 5·1% for the 1622 observed amplitudes. A final difference synthesis showed no significant features. Final atomic coordinates are in Table 1.* The weighting scheme was $w = 1/[\sigma^2(F)]$, where $\sigma(F)$ is the e.s.d. in the observed amplitudes based on counting statistics.

Computations were carried out on the University of Birmingham ICL 1906A computer.

Results and discussion

Fig. 1 shows a stereoscopic view of the cation and the atomic numbering. Protonation has occurred at N(4). The proton has been located from a difference

* Lists of observed and calculated structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34993 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Intermolecular contacts* (Å)

Distances involving only C, N and O atoms are listed up to 3·5 Å, and those involving the chlorine atom or the chloride ion up to 3·7 Å. H atom contacts are listed only for hydrogen bonds. E.s.d.'s are *ca* 0·006 Å for distances involving only the heavier atoms. H atom contacts have e.s.d.'s of *ca* 0·05 Å.

N(4)···Cl^-	3·05	C(2′)···C(5′^{i})	3·49
O(*w*)···Cl^-	3·26	C(6′)···Cl^-	3·64
O(*w*)···Cl^{-i}	3·33	C(5)···Cl^{-ii}	3·68
N(4)···Cl^{-ii}	3·48	C(3)···Cl^-	3·70
C(3)···Cl^{-ii}	3·48		
H(4)···Cl^-	2·09	H(O*w*)···Cl^-	2·22

Superscripts refer to the following equivalent positions:

(i) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$.

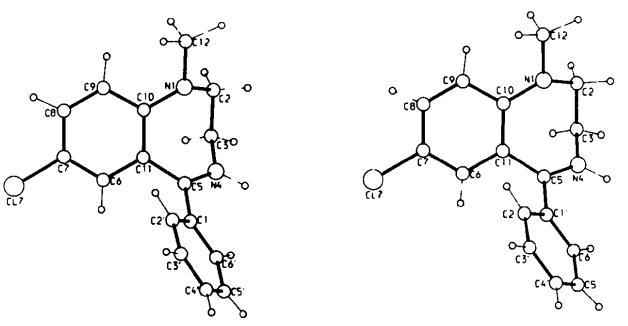

Fig. 1. Stereoscopic view of the medazepam cation in a direction perpendicular to the mean plane through atoms N(1), C(5)–(11).

Table 3. *Molecular dimensions for medazepam hydrochloride and comparison with the corresponding values in medazepam base (Gilli, Bertolasi, Sacerdoti & Borea, 1978)*

	Medazepam.HCl	Medazepam base
(*a*) Bond lengths (Å)		
C(12)–N(1)	1·477 (5)	1·468*
N(1)–C(2)	1·463 (5)	1·458
C(2)–C(3)	1·512 (6)	1·495
C(3)–N(4)	1·467 (5)	1·472
N(4)–C(5)	1·285 (4)	1·274
C(5)–C(11)	1·479 (5)	1·485
C(11)–C(6)	1·394 (5)	1·385
C(6)–C(7)	1·385 (5)	1·383
C(7)–Cl(7)	1·754 (4)	1·741
C(7)–C(8)	1·382 (6)	1·350
C(8)–C(9)	1·377 (6)	1·375
C(9)–C(10)	1·397 (5)	1·396
C(10)–C(11)	1·419 (5)	1·419
C(10)–N(1)	1·406 (5)	1·393
C(5)–C(1′)	1·483 (5)	1·490
C(1′)–C(2′)	1·394 (5)	1·392
C(2′)–C(3′)	1·382 (6)	1·395
C(3′)–C(4′)	1·385 (6)	1·382
C(4′)–C(5′)	1·377 (6)	1·371
C(5′)–C(6′)	1·395 (6)	1·384
C(6′)–C(1′)	1·395 (5)	1·385
(*b*) Bond angles (°) (e.s.d.'s are 0·3–0·4°)		
C(12)–N(1)–C(2)	110·0	110·0*
C(12)–N(1)–C(10)	118·6	117·2
C(10)–N(1)–C(2)	119·6	119·7
N(1)–C(2)–C(3)	111·5	113·2
N(4)–C(3)–C(2)	111·5	111·4
C(5)–N(4)–C(3)	123·0	116·2
N(4)–C(5)–C(11)	119·5	123·5
N(4)–C(5)–C(1′)	120·1	117·6
C(11)–C(5)–C(1′)	120·3	118·8
C(11)–C(6)–C(7)	120·4	120·2
C(6)–C(7)–Cl(7)	119·3	118·9
C(6)–C(7)–C(8)	120·2	120·9
C(8)–C(7)–Cl(7)	120·5	120·0
C(7)–C(8)–C(9)	119·7	120·4
C(8)–C(9)–C(10)	122·2	120·9
C(9)–C(10)–C(11)	117·3	118·3
C(9)–C(10)–N(1)	122·3	121·9
C(11)–C(10)–N(1)	120·2	119·7
C(5)–C(11)–C(6)	118·7	119·9
C(5)–C(11)–C(10)	121·1	120·8
C(10)–C(11)–C(6)	120·1	119·2
C(6′)–C(1′)–C(2′)	119·6	118·4
C(6′)–C(1′)–C(5)	120·5	120·3
C(2′)–C(1′)–C(5)	119·8	121·2
C(1′)–C(2′)–C(3′)	120·1	120·3
C(2′)–C(3′)–C(4′)	120·3	119·8
C(3′)–C(4′)–C(5′)	120·0	120·3
C(4′)–C(5′)–C(6′)	120·5	119·7
C(5′)–C(6′)–C(1′)	119·5	121·3

* Mean values taken over two independent molecules.

Table 3 (*cont.*)

(*c*) Selected torsion angles (°) (e.s.d.'s are 0·5–0·6°)

		Medazepam base Molecule	
	Medazepam.HCl	*A*	*B*
C(10)–N(1)–C(2)–C(3)	−34·7	−26·5	29·3
N(1)–C(2)–C(3)–N(4)	−49·1	−59·4	56·4
C(2)–C(3)–N(4)–C(5)	77·4	76·4	−77·1
C(3)–N(4)–C(5)–C(11)	−7·5	−1·2	2·9
N(4)–C(5)–C(11)–C(10)	−42·3	−47·4	47·7
C(5)–C(11)–C(10)–N(1)	−2·1	−0·3	−0·6
C(11)–C(10)–N(1)–C(2)	66·8	59·7	−60·8
N(4)–C(5)–C(1′)–C(2′)	142·3	153·4	−164·3
C(11)–C(5)–C(1′)–C(2′)	−34·5	−26·2	13·7
C(9)–C(10)–N(1)–C(2)	−118·4	−123·2	123·3
C(6)–C(11)–C(5)–N(4)	140·5	134·0	−133·8
C(12)–N(1)–C(2)–C(3)	−177·2	−167·6	169·0
C(12)–N(1)–C(10)–C(9)	21·0	14·7	−14·1

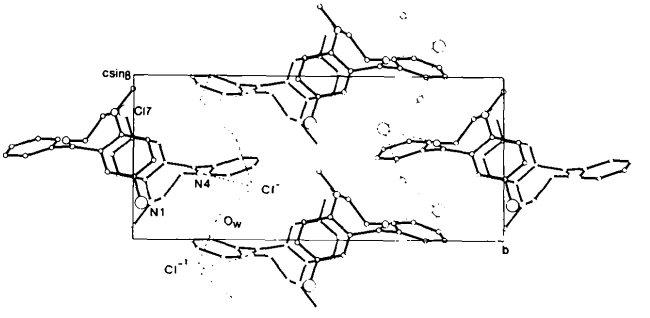

Fig. 2. The crystal structure projected along *x*. Hydrogen bonds are shown by broken lines.

synthesis; it takes part in hydrogen bonding with the Cl^- ion. The distances N(4)···Cl^- and H(4)···Cl^- are 3·05 and 2·09 Å, the proton lying close to the N(4)···Cl^- line. The molecule of water of crystallization appears to form two rather weak hydrogen bonds to symmetrically related Cl^- ions. This is illustrated in Fig. 2 which shows the packing viewed along *x*. The shorter intermolecular contacts are listed in Table 2. Apart from the hydrogen-bond interactions noted above, none of these is substantially shorter than the sum of the van der Waals radii of the atoms concerned.

Bond lengths, bond angles and selected torsion angles are listed in Table 3 and the results of mean-plane calculations are in Table 4. It is of interest to compare molecular dimensions and conformation with those of the free base. Medazepam base crystallizes with two independent molecules in the asymmetric unit which are indistinguishable as far as bond lengths and angles are concerned, but differ in the orientation of the 5-phenyl ring (Gilli *et al.*, 1978). To facilitate the comparison we have listed in Table 3, beside our values, the mean of the bond lengths and angles of the two independent molecules of medazepam base.

Bond lengths agree closely. The maximum deviation occurs in C(7)–C(8) where our length of 1·382 Å differs by 0·032 Å from the mean value in medazepam

Table 4. *Mean-plane calculations*

(*a*) Deviations of atoms from planes (Å) (e.s.d.'s are *ca* 0·004 Å)

Plane I: Phenyl ring C(6)–(11)		Plane II: Phenyl ring C(1′)–C(6′)	
C(6)	−0·005	C(1′)	−0·002
C(7)	0·004	C(2′)	0·009
C(8)	0·001	C(3′)	−0·009
C(9)	−0·004	C(4′)	0·003
C(10)	0·002	C(5′)	0·003
C(11)	0·002	C(6′)	−0·004
Cl(7)	−0·021	C(5)	−0·099
N(1)	−0·095	C(11)	0·545
C(12)	−0·664	N(4)	−0·794
C(2)	1·000		
C(3)	1·653		
N(4)	0·657		
C(5)	−0·053		
C(1′)	−0·972		
C(2′)	−2·212		
C(3′)	−3·117		
C(4′)	−2·783		
C(5′)	−1·557		
C(6′)	−0·642		

(*b*) Interplanar angle

Plane I–plane II	67·2 (5)°

(1·350 Å). This deviation may, however, be spurious, since the two measurements of this bond in medazepam, 1·336 and 1·364 Å, differ widely. The only other deviation >0·015 Å occurs in C(2)–C(3). Here our length of 1·512 Å is greater by 0·017 Å than the mean length in medazepam base. The two measurements for this bond in medazepam base agree closely (1·493 and 1·498 Å) so that this difference may well be real and related to the effect of protonation at the adjacent N atom. N(4)–C(5), 1·285 (4) Å, agrees to within the limits of experimental error with the accepted value of 1·274 Å (Lofthus, 1959) for a C–N double bond.

In general, bond angles for the base and cation also agree closely. Some large differences do, however, occur. In particular the angle at N(4) averages 116·2° in the base but is 123·0° when protonation takes place in the cation. A similar change in bond angle at N in unsaturated six-membered heterocyclic systems, when the lone pair of electrons on the trigonally hybridized N atom forms a bond with a proton, has been noted previously (Singh, 1965). The endocyclic angle at the adjacent C(5) is smaller by 4° in the cation. This difference can probably be regarded as a geometric consequence of the increase in angle at N(4). Similarly, the decrease in the endocyclic angle at C(5) leads to increases of 2·5 and 1·5° in the external angles at C(5), the larger increase being in N(4)–C(5)–C(1′). In both cation and free base the sum of angles at C(5) is close to 360°. Apart from deviations of 1·8° at C(6′) and 1·7° at C(2) no other bond angle differs by more than 1·5°.

The conformation of the cation is defined by the torsion angles listed in Table 3. Again the corresponding values for medazepam, calculated from the data of Gilli *et al.* (1978), are listed for comparison. Apart from the torsion angles relating to the orientation of the 5-phenyl ring, the two independent molecules of medazepam base are very similar, although of opposite conformational chirality. Torsion angles for the hydrochloride, however, differ from those of the base by considerable amounts. Within the 1,4-benzodiazepine system differences of 5–10° exist between the torsion angles of the hydrochloride and those of both molecules *A* and *B* of the base in most cases [Table 3(*c*)]. Presumably these differences are largely a consequence of the changes in endocyclic bond angle at N(4) and C(5) brought about by protonation.

Larger differences (>20°) occur when the torsion angles about C(5)–C(1′), which relate to the orientation of the 5-phenyl ring, are compared with those of molecule *B*. The angle between the mean planes of phenyl rings C(6)–(11) and C(1′)–(6′), which we use as a measure of the orientation of the 5-phenyl ring, is 67·2°. The corresponding angles in the two molecules of medazepam base are smaller, 62·9° (molecule *A*) and 55·4° (molecule *B*). These values may be compared with the corresponding angles in the biologically much more active 5-phenyl-1,4-benzodiazepines, clonazepam, where they are 83·7 and 77·8° for the two independent molecules in the unit cell (Chananont, Hamor & Martin, 1979), lorazepam, where they are 81·0 and 73·4° for two independent molecules (Bandoli & Clemente, 1976), nitrazepam, 61·8° (Gilli, Bertolasi, Sacerdoti & Borea, 1977) and diazepam, 54·7° (Camerman & Camerman, 1972).

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The Structure of 8-Thioxoadenosine Monohydrate

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Abstract

The structure of 8-thioxoadenosine monohydrate, $C_{10}H_{13}N_5O_4S.H_2O$, has been determined. The crystals are monoclinic, space group $P2_1$, with $a = 8{\cdot}758\ (1)$, $b = 6{\cdot}717\ (1)$, $c = 11{\cdot}462\ (2)$ Å, $\beta = 95{\cdot}22\ (1)°$, $Z = 2$. The structure was refined to $R = 0{\cdot}048$. The conformation about C(4′)–C(5′) is *gauche–trans*, and the puckering of the ribose is C(2′)-*endo*–C(3′)-*exo*. The molecule exhibits a *syn* conformation about the glycosyl bond in spite of the absence of an intramolecular O(5′)–H···N(3) hydrogen bond. The bases are stacked into columns, overlapping mainly at the S substituents of adjacent molecules. All the donor atoms are involved in hydrogen bonds, while the S atom acts as a hydrogen-bond acceptor.

Introduction

It is generally accepted that in purine nucleosides bulky substituents at the 8 position of the purine bases lead to a *syn* conformation about the glycosyl bond for steric reasons. This has been found in a number of crystal structures of such analogs (Yasuniwa *et al.*, 1979; Birnbaum & Shugar, 1978).

The title compound could form the thioketo structure rather than the alternative tautomeric structure. Therefore, it is of considerable interest to study the influence of a geometrical change in the imidazole portion of the adenine base and the steric effect of the thioxo substituent on the overall conformation of the molecule. It is also of interest to estimate the effects of the thioxo substituent on the base stacking and hydrogen bonding.

Experimental

The title compound was synthesized (Ikehara & Yamada, 1971), and crystallized from aqueous solution as greenish-yellow prisms. The crystals belong to the space group $P2_1$. The cell constants were determined from a least-squares refinement of the setting angles of 20 medium-angle reflections measured on a Rigaku–Denki automated diffractometer. The density was measured by flotation. Crystallographic data are given in Table 1. Intensity data were collected on the diffractometer with Ni-filtered Cu $K\alpha$ radiation, employing the ω–2θ scan mode. There were 939 unique reflections up to $2\theta = 110°$, of which 931 were considered as observed. The intensities were corrected for Lorentz and polarization factors but not for absorption.

The structure was determined by the heavy-atom method and the parameters of the nonhydrogen atoms were refined by block-diagonal least squares with anisotropic temperature parameters. All H atoms were located on difference Fourier maps and refined with isotropic temperature parameters. The final R value was 0·048. Tables 2 and 3 list the final positional

Table 1. *Crystal data*

$C_{10}H_{13}N_5O_4S.H_2O$	$M_r = 317{\cdot}33$
Monoclinic	Space group $P2_1$
$a = 8{\cdot}758\ (1)$ Å	$Z = 2$
$b = 6{\cdot}717\ (1)$	$F(000) = 332$
$c = 11{\cdot}462\ (2)$	$V = 671{\cdot}5\ (1)$ Å^3
$\beta = 95{\cdot}22\ (1)°$	$D_m = 1{\cdot}566$ Mg m^{-3}
λ(Cu $K\alpha$) = 1·5418 Å	$D_x = 1{\cdot}569$
$\mu = 2{\cdot}40$ mm^{-1}	

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