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Stereochemistry of Anticholinergic Agents. V.* Crystal and Molecular Structure of Thiphenamil Hydrochloride

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(*Received* 15 *May* 1974; *accepted* 18 *May* 1974)

Thiphenamil hydrochloride, *S*-(2-diethylaminoethyl) diphenylthioacetate hydrochloride, crystallizes as the monohydrate from butanone in the monoclinic space group $P2_1/c$ with $a=19{\cdot}41$ (1), $b=7{\cdot}52$ (1), $c=14{\cdot}78$ (1) Å, $\beta=103{\cdot}90$ (5)° and $Z=4$. The structure was determined by Patterson and Fourier methods from three-dimensional X-ray counter data and refined by least-squares calculations to R 5·5% for 2233 observed amplitudes. Estimated standard deviations for bond lengths, bond angles and torsion angles average 0·006 Å, 0·3°, and 0·5°. In the thiphenamil cation, the acetylthiocholine-like moiety has the C–C(=O)–S–C grouping antiplanar, C(=O)–S–C–C, synclinal, and S–C–C–N^+, antiplanar. The C(=O)–S and S–C bond lengths are 1·780 (4) and 1·814 (4) Å. The molecular geometry is compared with those of related anticholinergic and cholinergic molecules.

Introduction

Certain derivatives of acetylcholine incorporating ring substituents in the acyl group and larger groups on the nitrogen atom act as atropine-like antagonists of acetylcholine at the parasympathetic post-ganglionic (muscarinic) receptor (Triggle, 1965). The crystal structures of a number of these compounds have been determined and their conformations compared (Guy & Hamor, 1974*a*). We now present the results of a crystal structure analysis of *S*-(2-diethylaminoethyl) diphenylthioacetate hydrochloride (thiphenamil hydrochloride), the thiolester analogue of the anticholinergic adiphenine hydrochloride, the structure of which has already been described (Guy & Hamor, 1973). Thiphenamil has an anticholinergic activity *ca* 20% of

* Part IV: Guy & Hamor (1974*a*).

that of atropine as measured by its inhibitory effect of spasms induced in isolated guinea-pig ileum by acetylcholine stimulation (Parkes, 1955). It is also extremely active in antagonizing spasms induced by barium ions which act directly on the muscle cells.

Experimental

Thiphenamil hydrochloride was obtained from Wm. P. Poythress and Co. and was recrystallized from butanone yielding colourless plate-like crystals. The density was measured by flotation in a carbon tetrachloride/petroleum spirit (80–100) mixture. Approximate cell dimensions were determined by oscillation, Weissenberg and precession photographs, final cell dimensions being measured on a Stoe two-circle computer-controlled diffractometer with graphite-monochromated Mo $K\alpha$ radiation and a scintillation counter. Intensities were collected from a crystal of dimensions $0{\cdot}75\times0{\cdot}3\times0{\cdot}15$ mm mounted about the unique (b) axis. The ω-scan technique was employed with a stepping interval of 0·01° and a step time of 1s. For layers $h0l$–$h3l$, $\Delta\omega$ was taken as 1·4° and for the fourth and higher layers (equi-inclination angle, $\mu>10°$) a variable scan range was employed, $\Delta\omega$ being calculated from $(A+B\sin\mu/\tan\theta')°$ where $2\theta'$ is the azimuth angle (Buerger, 1942) and A and B were assigned values of 1·0 and 0·5 respectively. Background for all reflexions was measured for 30s at each end of the scan. After each layer, four standard reflexions on the zero layer were re-measured to monitor the stability of the system. There was no systematic variation of intensity with time in the course of the data collection. Based on these measurements, interlayer scale factors ranging from 1·00 to 1·08 were derived and applied to the appropriate layers of reflexions.

Of 3698 reflexions scanned within the range $0{\cdot}08<\sin\theta/\lambda<0{\cdot}60$, 2233 for which $I>2{\cdot}5\sigma(I)$ were considered to be observed and were used in the structure analysis. In converting intensities to structure amplitudes the polarization factor appropriate to monochromated radiation was used. Absorption corrections were not applied.

Crystal data

$C_{20}H_{25}NOS.HCl.H_2O$, $M=382{\cdot}0$. Monoclinic, $a=19{\cdot}41$ (1), $b=7{\cdot}52$ (1), $c=14{\cdot}78$ (1), $\beta=103{\cdot}90$ (5)°, $U=2095{\cdot}6$ Å^3, $D_m=1{\cdot}216$, $Z=4$, $D_x=1{\cdot}209$, $F(000)=816$. Systematic absences: $h0l$ when l is odd, $0k0$ when k is odd, space group $P2_1/c$ (C_{2h}^5). Mo $K\alpha$ radiation, $\lambda=0{\cdot}71069$ Å; μ(Mo $K\alpha$) = 2·9 cm^{-1}.

Determination of the structure

The coordinates of both the chloride ion and the sulphur atom were located from a Patterson synthesis. Calculated phase angles (R 51 %) were used with the observed structure amplitudes to evaluate a Fourier synthesis from which the positions of all non-hydrogen atoms, except the terminal methyl carbon atoms and three carbon atoms in each of the phenyl rings, were obtained. A further Fourier synthesis located all non-hydrogen atoms and five cycles of least-squares refinement with isotropic temperature factors reduced R to 21 %. This rather high R value suggested the omission of one or more atoms, and a difference synthesis was computed and examined in conjunction with the previous Fourier maps. The coordinates of a peak of *ca* 5 e Å^{-3}, previously thought to have been spurious, were now included as an oxygen atom of a possible molecule of water of crystallization. A further cycle of isotropic refinement reduced R to 15 %, when the atoms were allowed to vibrate anisotropically. Hydrogen atom positions were located from a difference synthesis and included in their calculated positions [assuming C(sp^3)–H 1·10, C(sp^2)–H 1·08 and N–H 1·04 Å] but their parameters were not refined. The hydrogen atoms of the water molecule were located as two definite maxima in the difference synthesis at distances of 0·93 and 0·96 Å from the oxygen atom with an H–O–H angle 90°. Refinement was terminated when the calculated shifts were all $<0{\cdot}1\sigma$, and R

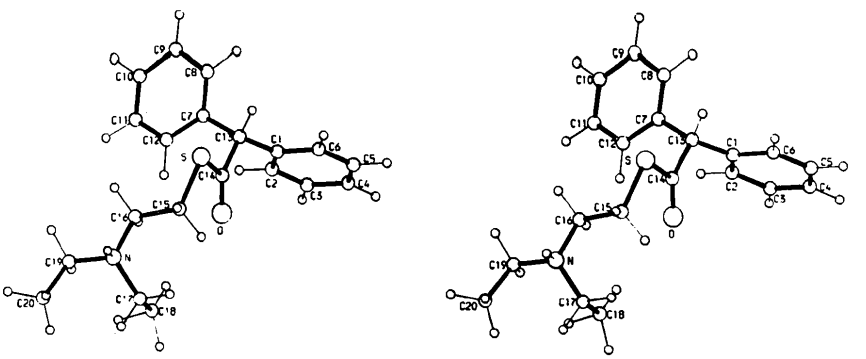

Fig. 1. Stereoscopic view of the thiphenamil cation along the y axis.

5·5% for the 2233 observed structure amplitudes.* Final atomic coordinates are listed in Table 1 and the thermal parameters in Table 2.

* A list of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30503 (15 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with estimated standard deviations in parentheses*

	x	*y*	*z*
C(1)	9319 (2)	5575(5)	1482 (2)
C(2)	9205 (2)	7264 (6)	1116 (3)
C(3)	9727 (3)	8149 (7)	791 (3)
C(4)	10375 (3)	7355 (7)	839 (3)
C(5)	10497 (2)	5716 (8)	1217 (4)
C(6)	9982 (2)	4817 (6)	1540 (3)
C(7)	8267 (2)	5702 (5)	2227 (2)
C(8)	8510 (2)	6266 (6)	3146 (3)
C(9)	8100 (3)	7350 (6)	3560 (3)
C(10)	7441 (3)	7923 (6)	3053 (4)
C(11)	7200 (2)	7381 (6)	2137 (4)
C(12)	7602 (2)	6280 (5)	1725 (3)
C(13)	8750 (2)	4540 (5)	1798 (2)
C(14)	8304 (2)	3453 (5)	983 (3)
C(15)	7433 (2)	656 (5)	273 (3)
C(16)	6710 (2)	1556 (5)	100 (2)
C(17)	6469 (2)	905 (6)	−1626 (3)
C(18)	6656 (3)	2756 (8)	−1876 (3)
C(19)	5488 (2)	1695 (6)	−856 (3)
C(20)	4916 (2)	874 (8)	−1623 (3)
N	6199 (2)	824 (4)	−754 (2)
S	7998 (1)	1421 (1)	1365 (1)
O	8167 (2)	3910 (4)	181 (2)
O(*w*)	5581 (2)	−5727 (5)	1206 (2)
Cl	6056 (1)	−3161 (2)	−333 (1)
H(C2)	8689	7929	1090
H(C3)	9623	9474	482
H(C4)	10782	8083	565
H(C5)	11027	5095	1261
H(C6)	10085	3472	1836
H(C8)	9041	5817	3536
H(C9)	8298	7756	4290
H(C10)	7111	8797	3376
H(C11)	6668	7803	1753
H(C12)	7425	5875	984
H(C13)	9034	3600	2346
H^1(C15)	7691	981	−313
H^2(C15)	7373	−805	284
H^1(C16)	6774	3004	−3
H^2(C16)	6492	1364	715
H^1(C17)	6954	58	−1527
H^{2}C(17)	6061	326	−2212
H^1(C18)	6105	3603	−2075
H^2(C18)	6651	2860	−2640
H^3(C18)	6994	3528	−1357
H^1(C19)	5531	3130	−1014
H^2(C19)	5320	1598	−188
H^1(C20)	4949	−571	−1571
H^2(C20)	4926	1509	−2299
H^3(C20)	4383	1167	−1487
H(N)	6104	−520	−618
H^1(O*w*)	5150	−6000	750
H^2(O*w*)	5750	−5125	750

Table 2. *Anisotropic thermal parameters* ($\times 10^4$) *for the heavier atoms in the form*
$T = exp\,[-2\pi^2(U_{11}h^2a^{*2} + \ldots + 2U_{12}hka^*b^* + \ldots)]$

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	383	485	423	−1	19	−96
C(2)	489	597	747	48	199	136
C(3)	692	633	854	−51	265	106
C(4)	630	763	752	−148	240	−159
C(5)	453	842	1100	36	272	−160
C(6)	507	579	911	74	152	−14
C(7)	471	366	452	−57	105	16
C(8)	664	560	487	−62	156	−22
C(9)	1008	551	720	−186	461	−167
C(10)	944	431	1101	−27	665	−25
C(11)	587	605	977	99	352	144
C(12)	510	499	595	19	148	41
C(13)	473	421	369	−11	54	24
C(14)	429	460	439	34	108	0
C(15)	544	421	526	−10	31	−102
C(16)	481	481	454	−51	116	−111
C(17)	561	620	430	19	146	−73
C(18)	1042	887	653	−266	198	172
C(19)	452	694	671	70	149	−109
C(20)	526	986	779	17	23	−117
N	488	422	431	−25	116	−35
S	592	422	514	−55	−20	49
O	787	768	405	−263	68	23
O(*w*)	834	1119	509	−96	14	122
Cl	658	505	761	−109	160	92

The weighting scheme used in the final cycles of refinement was: $w^{1/2} = |F_o|/15{\cdot}0$ if $|F_o| < 15{\cdot}0$, and $w^{1/2} = 15{\cdot}0/|F_o|$ if $|F_o| > 15{\cdot}0$, chosen to give approximately constant values for the average of $\sum w(|F_o| - |F_c|)^2$ in groups of increasing $|F_o|$ and $\sin\theta/\lambda$. The scattering factors for all atoms except hydrogen were those of Hanson, Herman, Lea & Skillman (1964). For the hydrogen atoms, the scattering factors of Stewart, Davidson & Simpson (1965) were used.

Computations were carried out on the Birmingham University 1906A computer with local versions of *FORDAP*, the Zalkin Fourier program and *ORFLS* and *ORFFE*, the Busing, Martin & Levy full-matrix least-squares and function and error programs.

Discussion

Table 3 contains bond lengths, bond angles and torsion angles, and the results of mean plane calculations are in Table 4. The stereochemistry of the thiphenamil cation is illustrated in Fig. 1, which also indicates the atomic numbering.

The structure of the acetylthiocholine moiety of thiphenamil may be compared with that of acetylthiocholine in crystals of the bromide (Shefter & Mautner, 1969; Shefter, 1974) and with the conformation calculated for acetylthiocholine (henceforth AcSCh) by the PCILO method (Pullman & Courrière, 1972). The thiolester group, C(13), C(14), C(15), S and O, is planar to within 0·06 Å and adopts the antiplanar conformation typical of esters [torsion angle C(13)–C(14)–S–C(15), ±174°]. The corresponding group of atoms of AcSCh bromide deviates from

planarity by as much as 0·26 Å with the C–C(=O)–S–C torsion angle ±150°. A greater difference occurs in the arrangement about the S–C(15) bond, the C(=O)–S–C–C torsion angle being ∓82° in thiphenamil and ±129° in AcSCh. In both structures the S–C–C–N^+ grouping is antiplanar and the methyl groups are oriented close to the ideally staggered arrangement with respect to the substituents on C(16). In the theoretical calculations the C–C(=O)–S–C and one of the Me–N^+–C–C torsion angles are assumed to be 180° and the values calculated for C(=O)–S–C–C and S–C–C–N^+ are 60–80 and 180°, respectively, in better agreement with the situation in thiphenamil than in AcSCh bromide. The antiplanar conformation of the S–C–C–N^+ grouping of AcSCh is maintained also in solution (Cushley & Mautner, 1970; Culvenor & Ham, 1970). The conformation of propionylthiocholine in crystals of the iodide (Shefter & Mautner, 1969) is intermediate between that of AcSCh and the comparable moiety of thiphenamil. AcSeCh (Shefter & Kennard, 1966) has a conformation very similar to that of AcSCh but the AcSeCh moiety of 2-dimethylaminoethyl selenolbenzoate hydrochloride (Dexter, 1972) closely resembles the situation in thiphenamil.

The acetylcholine moiety of adiphenine hydrochloride, the oxy-analogue of thiphenamil, adopts a different conformation with C(=O)–O–C–C antiplanar and O–C–C–N^+ synclinal, similar to that of acetylcholine in crystals of the chloride (Herdklotz & Sass, 1970) and in solution (Culvenor & Ham, 1966). Other anticholinergics based on acetylcholine, which are esters of secondary alcohols where the nitrogen atom forms part of a ring system, however, have the C(=O)–O–C–CN^+ grouping synclinal (Guy & Hamor, 1974*a*, *b*, and references therein).

Table 3. *Molecular dimensions*

(*a*) Bond distances (Å) with standard deviations in parentheses

C(1)—C(2)	1·378 (6)	C(1)—C(13)	1·515 (5)
C(2)—C(3)	1·391 (6)	C(7)—C(13)	1·526 (5)
C(3)—C(4)	1·379 (7)	C(13)–C(14)	1·538 (5)
C(4)—C(5)	1·351 (8)	C(14)–O	1·202 (4)
C(5)—C(6)	1·384 (7)	C(14)–S	1·780 (4)
C(6)—C(1)	1·390 (6)	S———C(15)	1·814 (4)
C(7)—C(8)	1·393 (5)	C(15)–C(16)	1·523 (5)
C(8)—C(9)	1·380 (6)	C(16)–N	1·510 (5)
C(9)—C(10)	1·387 (7)	N———C(17)	1·505 (5)
C(10)–C(11)	1·385 (8)	C(17)–C(18)	1·507 (7)
C(11)–C(12)	1·376 (6)	N———C(19)	1·502 (5)
C(12)–C(7)	1·396 (5)	C(19)–C(20)	1·516 (6)

(*b*) Selected non-bonded distances (Å)

N···S	4·12	N···H[C(13)]	6·60
N···C(14)	4·71	N···Centre of ring C(1)–(6)	8·25
N···O	4·40	N···Centre of ring C(7)–(12)	7·02
N···C(13)	6·13		

(*c*) Bond angles (°); mean standard deviation 0·3°

C(6)—C(1)—C(2)	117·5	C(12)–C(7)—C(13)	122·6
C(1)—C(2)—C(3)	121·0	C(1)—C(13)–C(7)	113·6
C(2)—C(3)—C(4)	120·4	C(1)—C(13)–C(14)	110·3
C(3)—C(4)—C(5)	118·9	C(7)—C(13)–C(14)	110·1
C(4)—C(5)—C(6)	121·3	C(13)–C(14)–O	124·8
C(5)—C(6)—C(1)	120·8	C(13)–C(14)–S	112·1
C(12)–C(7)—C(8)	118·5	O———C(14)–S	123·1
C(7)—C(8)—C(9)	121·1	C(14)–S———C(15)	99·6
C(8)—C(9)—C(10)	120·0	S———C(15)–C(16)	110·2
C(9)—C(10)–C(11)	119·2	C(15)–C(16)–N	111·8
C(10)–C(11)–C(12)	121·0	C(16)–N———C(17)	114·0
C(11)–C(12)–C(7)	120·2	C(16)–N———C(19)	109·2
C(2)—C(1)—C(13)	122·3	N———C(17)–C(18)	113·6
C(6)—C(1)—C(13)	120·2	N———C(19)–C(20)	112·9
C(8)—C(7)—C(13)	118·8	C(17)–N———C(19)	113·6

(*d*) Torsion angles (°); mean standard deviation for angles not involving hydrogen atoms 0·5°. (Also present in the crystal are the centrosymmetrically related rotamers with torsion angles of opposite sign)

C(2)———C(1)—C(13)–C(14)	89·3
C(6)———C(1)—C(13)–C(14)	−89·6
C(8)———C(7)—C(13)–C(14)	156·5
C(12)———C(7)—C(13)–C(14)	−26·0
C(2)———C(1)—C(13)–C(7)	−34·9
C(6)———C(1)—C(13)–C(7)	146·3
C(1)———C(13)–C(7)—C(8)	−79·3
C(1)———C(13)–C(7)—C(12)	98·3
C(1)———C(13)–C(14)–O	−33·4
C(1)———C(13)–C(14)–S	147·4
C(7)———C(13)–C(14)–O	92·7
C(7)———C(13)–C(14)–S	−86·5
C(13)—C(14)–S———C(15)	174·0
O———C(14)–S———C(15)	−5·2
C(14)—S———C(15)–C(16)	−82·0
S———C(15)–C(16)–N	−174·4
C(15)—C(16)–N———C(17)	−55·1
C(15)—C(16)–N———C(19)	177·1
C(16)—N———C(17)–C(18)	−57·9
C(16)—N———C(19)–C(20)	−172·6
C(18)—C(17)–N———C(19)	67·8
C(17)—N———C(19)–C(20)	59·1
H(C13)–C(13)–C(14)–S	31·5
H(N)—N———C(16)–C(15)	65

Table 4. *Mean plane calculations*

(*a*) Deviations (Å) of atoms from least-squares planes. In the equations of the planes, x, y and z are fractional coordinates relative to the cell axes.

Plane (*a*) C(1)–C(6)

$$2{\cdot}600x + 3{\cdot}009y + 12{\cdot}536z = 3{\cdot}370$$

C(1) 0·011, C(2) −0·008, C(3) −0·002, C(4) 0·008, C(5) −0·005, C(6) −0·005, C(13) 0·075

Plane (*b*) C(7)–C(12)

$$9{\cdot}952x + 6{\cdot}030y - 6{\cdot}233z = 0{\cdot}328$$

C(7) 0·002, C(8) −0·006, C(9) 0·005, C(10) −0·000, C(11) −0·004, C(12) 0·003, C(13) −0·044

Plane (*c*) C(13)–C(15), O, S

$$-16{\cdot}729x + 3{\cdot}667y + 5{\cdot}070z = 4{\cdot}620$$

C(13) −0·049, C(14) 0·018, C(15) −0·054, O 0·027, S 0·058

Plane (*d*) C(13), C(14), O, S

$$-17{\cdot}098x + 3{\cdot}450y + 4{\cdot}814z = 4{\cdot}569$$

C(13) −0·001, C(14) 0·004, O −0·002, S −0·001, C(15) −0·179

(*b*) Dihedral angles (°)

(*a*)–(*b*)	83·7	(*b*)–(*c*)	95·4
(*a*)–(*c*)	78·1	(*b*)–(*d*)	96·9
(*a*)–(*d*)	80·1		

Cushley & Mautner, 1970; Culvenor & Ham, 1970; Dexter, 1972; Guy & Hamor, 1974*a*, *b*; Herdklotz & Sass, 1970; Shefter & Kennard, 1966; Shefter & Mautner, 1969

Anticholinergic activity does not seem to depend on which of these two conformations the acetylcholine moiety adopts. Probably, of greater importance is the spatial relationship between the cationic head of the molecule and the ring substituents in the acyl group.

In thiphenamil, the orientation of the phenyl rings relative to the ester group differs from the situation in adiphenine. Compared with adiphenine, the bonds linking the phenyl rings to C(13) are rotated *ca* 37° about C(13)–C(14). The orientations of the rings about C(1)–C(13) and C(7)–C(13) are also different (Guy & Hamor, 1973). The conformation of this portion of the molecule is similar to that found in the crystal structure of the related anticholinergic agent piperidolate hydrochloride (Guy & Hamor, 1974*b*). However, because of the different arrangements about S–C(15) and C(15)–C(16), the orientation of the phenyl rings relative to the cationic head is quite different in thiphenamil.

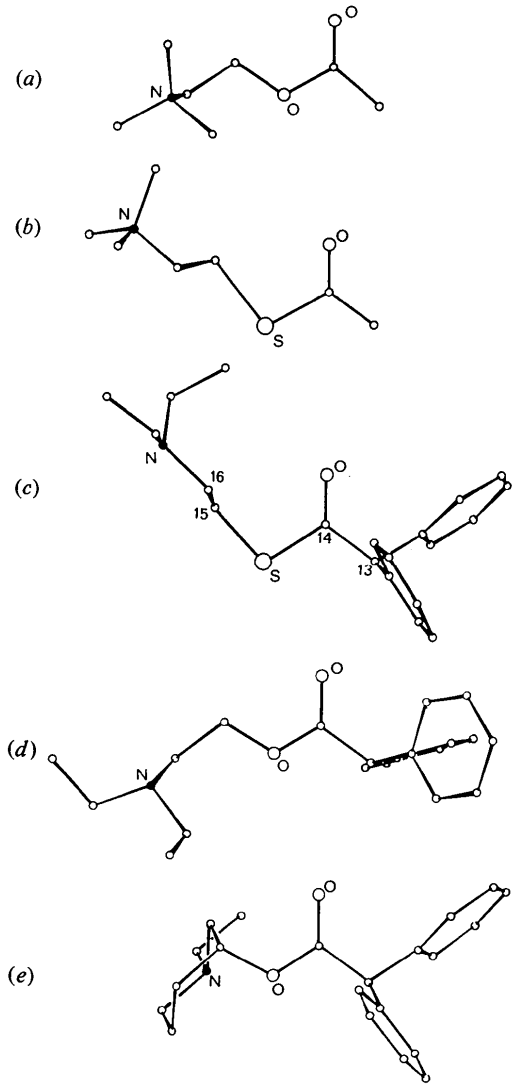

Fig. 2. Drawing of (*a*) acetylcholine, (*b*) acetylthiocholine, (*c*) thiphenamil, (*d*) adiphenine, and (*e*) piperidolate as viewed in a direction perpendicular to the mean plane of the ester group.

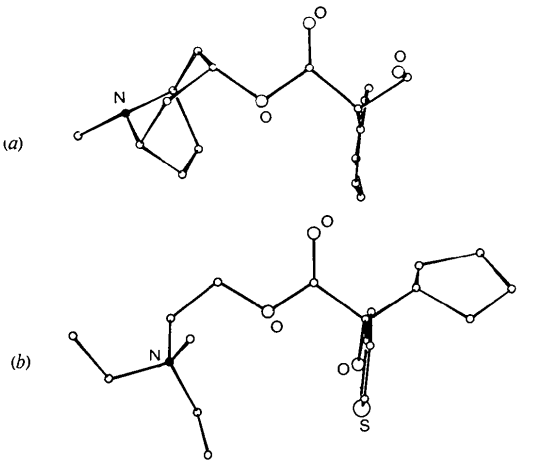

Fig. 3. (*a*) Atropine, and (*b*) penthienate, viewed as in Fig. 2.

This is illustrated in Fig. 2, which shows the acetylcholine, AcSCh, thiphenamil, adiphenine and piperidolate cations viewed in a direction perpendicular to the mean plane of their ester groups. In Fig. 3 is a similar view of the more potent anticholinergics (−)-hyoscyamine (atropine) hydrobromide (Kussäther & Haase, 1972) and penthienate bromide (Guy & Hamor, 1974*a*). The resemblance between penthienate and atropine is obvious. A more detailed comparison of structural features amongst anticholinergic molecules related to acetylcholine and possible relationships between conformation and pharmacological activity has been given elsewhere (Guy & Hamor, 1974*a*). Intramolecular non-bonded distances in thiphenamil [Table 3(*b*)] generally differ considerably from the corresponding separations in acetylcholine derivatives.

Bond lengths (mean σ 0·006 Å) agree well with expected values, although the C(4)–C(5) bond length of 1·351 Å is significantly shorter than the standard aromatic length. Within the thiolester group, the C(14)–O length of 1·202 Å, is in good agreement with the length of this bond in esters (generally in the range 1·19–1·21 Å). C(14)–S is shorter by 0·034 Å than S–C(15). The latter (1·814 Å) corresponds to a single bond between sulphur and sp^3-hybridized carbon. The shortening of the former can be attributed to the different hybridization state of C(14), and its length (1·780 Å) is what might be expected for a C(sp^2)–S single bond. In esters the analogous C(sp^2)–O and O–C(sp^3) bonds differ by considerably more (*ca* 0·1 Å) owing, presumably, to electron delocalization between the carbonyl and ester oxygen atoms, so that the C(sp^2)–O bond possesses some double-bond character. In 2-dimethylaminoethyl selenolbenzoate

hydrochloride (Dexter, 1972), the two C–Se bond lengths are identical while the C–O(carbonyl) length is 1·201 Å. Thus in the thiol- and selenolesters, the C–O(carbonyl) bond is similar to that in esters, without, however, there being apparently any shortening of the C(sp^2)–S,Se lengths below the single-bond value. These results are not entirely in agreement with those reported for AcSCh, propionylthiocholine and AcSeCh, but owing to the large standard deviations quoted for these structures, detailed comparison of bond lengths would not be very meaningful.

The crystal structure is illustrated in Fig. 4, which shows a projection along *y*. Intermolecular contact distances are listed in Table 5. The $N^+\cdots Cl^-$ distance of 3·09 Å corresponds to a fairly strong N^+–H$\cdots Cl^-$ hydrogen bond and the hydrogen atom lies close to the $N^+\cdots Cl^-$ line (angle H–$N^+\cdots Cl^-$ 5°). The molecule of water of crystallization is hydrogen bonded to two centrosymmetrically related chloride ions, the H$\cdots Cl^-$ distances being each *ca* 2·36 Å, the O$\cdots Cl^-$ distances 3·28 and 3·24 Å, and the H–O$\cdots Cl^-$ angles 8 and 20°. The Cl$\cdots$O$\cdots Cl^{I}$ angle is 106·7°. Thus each chloride ion is hydrogen bonded to the nitrogen atom of a thiphenamil cation and to two centrosymmetrically related water molecules, this system of hydrogen bonds linking the cations in pairs about centres of symmetry at $(\frac{1}{2}, -\frac{1}{2}, 0)$ *etc.* The geometry of the hydrogen-bond interactions of the water molecule appears to be typical (Pedersen, 1974).

Table 5. *Intermolecular contacts* (< 3·8 Å), *excluding hydrogen atoms*

N$\cdots\cdots$Cl	3·09	C(7)$\cdots$O(w^{V})	3·64
O(w)$\cdots$Cl	3·28	O(w)$\cdots$C(4^{IV})	3·66
O(w)$\cdots Cl^{I}$	3·24	C(17)$\cdots$C(13^{VI})	3·67
O(w)$\cdots$C(5^{II})	3·26	S$\cdots\cdots$C(19^{IV})	3·71
Cl$\cdots\cdots$C(6^{II})	3·45	Cl$\cdots\cdots$C(8^{II})	3·74
C(18)$\cdots$C(5^{III})	3·54	Cl$\cdots\cdots$C(20^{IV})	3·75
C(19)$\cdots$C(5^{III})	3·57	C(12)$\cdots$C(16^{VI})	3·75
O(w)$\cdots$C(7^{IV})	3·58		

Superscripts refer to the following equivalent positions:

I	$1-x, -1-y, -z$	IV	$x, -1+y, z$
II	$x, -\frac{1}{2}-y, \frac{1}{2}+z$	V	$1-x, -y, z$
III	$x, \frac{1}{2}-y, \frac{1}{2}+z$	VI	$2-x, \frac{1}{2}+y, \frac{1}{2}-z$

We thank Professors M. Stacey and J. C. Robb for their interest, the University of Birmingham for the award of a Research Scholarship (to J.J.G.), the staff of the Birmingham University Computer Centre for their assistance, and Wm. P. Poythress and Company for a gift of thiphenamil hydrochloride. The drawing of acetylthiocholine is based on coordinates kindly provided by Professor E. Shefter (1974).

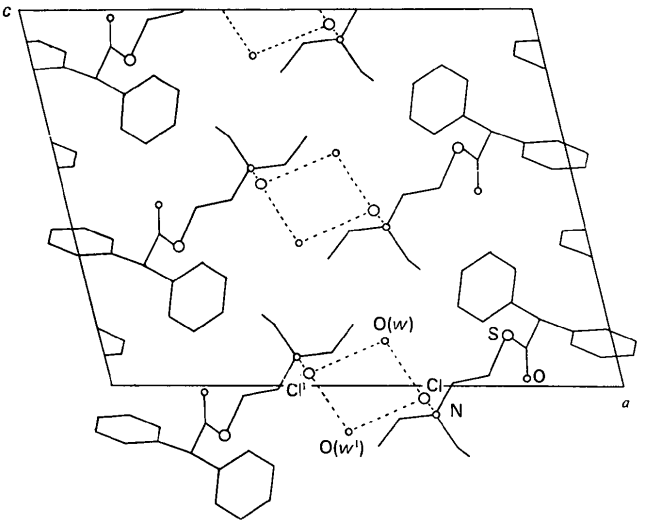

Fig. 4. The crystal structure projected along the *y* axis. Hydrogen bonds are shown by broken lines.

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