Table 6. *Hydrogen bonds*

X–H···*Y*	*X*···*Y*	*X*–H	H···*Y*	*X*–H···*Y*	Symmetry operation on *Y*
O(*W*)–H(*W*)1···O(3′)	2·860 (8) Å	1·11 Å	1·77 Å	163°	*x*,*y*,*z*
O(*W*)–H(*W*)2···N(9)	3·046 (7)	1·15	1·93	163	$\frac{1}{2}-x+1, 1-y, \frac{1}{2}+z-1$
O(3′)–H(O3′)···O(2)	2·730 (7)	0·93	1·86	155	$-x+2, \frac{1}{2}+y, \frac{1}{2}-z+1$
N(1′)–H(N1′)···O(*W*)	2·951 (6)	1·03	1·96	160	$\frac{1}{2}+x-1, \frac{1}{2}-y+1, -z+1$

this angle is described by the sequence O(0′)–C(5′)–C(6′)–O(3′) and its value of −56·2(7)° is in the range ±60–±30° common for pyranoside derivatives.

Nucleoside conformation and molecular packing

The orientation of the base relative to the sugar ring, described in terms of rotation about the N(7)–C(4′) glycosyl bond for the sequence C(8)–N(7)–C(4′)–C(5′), is *anti* [75·5 (7)°] (Sundaralingam, 1975).

The packing is dominated by hydrogen bonds (Table 6). The water molecule acts as a donor to the carbohydrate O(3′) and to the base N(9) as well as an acceptor to the acetamido N(1′)–H. Thus the water molecule is involved in hydrogen bonds with the sugar moiety by O(*W*)–H(*W*)1···O(3′), 2·860 (8), and N(1′)–H(N1′)···O(*W*), 2·951 (6) Å, and with the base residue by O(*W*)–H(*W*)2···N(9), 3·046 (7) Å. Sugar–base interaction is realized through the O(3′)–H(O3′)···O(2), 2·730 (7) Å, hydrogen bond. Base stacking does not occur.

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The Structure of Ethoxycarbonylcholine Iodide*

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Abstract

$C_8H_{18}NO_3^+.I^-$ crystallizes in space group $P\bar{1}$ with a = 11·632 (6), b = 14·658 (8), c = 15·24 (1) Å, α = 97·75 (6), β = 89·44 (3), γ = 101·07 (5)°, Z = 8, U = 2527 Å^3, D_m = 1·609, D_c = 1·59 Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 2·56 mm^{-1}, $F(000)$ = 1200. The final R = 0·062 for 5525 reflections. The ethoxycarbonylcholine ions adopt a variety of conformations. The potential originating in the ester moiety seems to make some packing patterns especially favourable.

* IUPAC name: (ethoxycarbonyloxyethyl)trimethylammonium iodide.

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Introduction

Methoxy- and ethoxycarbonylcholine iodides were synthesized as part of an investigation of the influence of various groups *Y*– on the physicochemical and biological properties of choline esters with the common formula Y–$COOCH_2CH_2\overset{+}{N}(CH_3)_3.X^-$. The crystal structure of the methoxy compound (Jensen, 1979) showed the ester in its fully extended conformation. In this paper the crystal structure of a triclinic form of ethoxycarbonylcholine iodide is described. Ethoxycarbonylcholine iodide also crystallizes in a less stable tetragonal form with an order/disorder structure which, judged from the preliminary films, must be very similar to the crystal structure of acetylcholine iodide (Jagner & Jensen, 1977). Further investigations of the tetragonal form are not planned.

Experimental

Ethoxycarbonylcholine iodide was synthesized by reacting *N*,*N*-dimethylaminoethanol with ethyl chloroformate (Vieler & Galsomias, 1968) followed by methylation with methyl iodide. Single crystals were grown by diffusion of dimethoxymethane into an aqueous ethanolic solution of the compound.

Cell parameters were refined by least-squares techniques from the diffractometer-measured θ angles for 47 reflections. The density was measured by flotation. The melting point was determined on a Leitz hot-stage microscope.

Intensities were measured at room temperature from a crystal 0·3 × 0·3 × 0·4 mm mounted on a Nonius CAD-3 diffractometer. Graphite-monochromated Mo $K\alpha$ radiation and the ω-scan technique were used.

Reflections were measured in the range $3{\cdot}5° \le \theta \le 25{\cdot}0°$, but due to instrumental difficulties the data set is incomplete. The intensity of one (and only one) of the three standard reflections had dropped to about $\frac{2}{3}$ of its initial value by the end of the data collection (high values of I), but no attempt to correct this was made. Of the 7038 reflections measured, 5525 were considered observed at the $1{\cdot}5\sigma(I)$ significance level, where σ is the standard deviation from counting statistics. No absorption corrections were made.

Structure determination and refinement

The trial structure was obtained by the heavy-atom method and refined to a final R of 0·062 for the 5525 observed reflections with XRAY 76 (Stewart, 1976). The final cycles included one scale factor and positional and anisotropic thermal parameters for all non-hydrogen atoms. Most of the H atoms were located in a difference map and all H atoms were included in their calculated positions (C–H = 1·0 Å) as a fixed contribution. The isotropic temperature factors chosen for the H atoms (B = 5·0–12·0 Å^2) reflect the thermal parameters of the atoms to which they are bonded.

The quantity minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = 1$ for $F_o \le 50$ and $(50/F_o)^2$ for $F_o \ge 50$. The scattering factors used for H were those of Stewart, Davidson & Simpson (1965) and for all other atoms those listed in *International Tables for X-ray Crystallography* (1974). The real part of the dispersion correction was included for I. All atoms but I^- were treated as uncharged.*

Results and discussion

The atomic coordinates are listed in Table 1. Table 2 gives the bond distances, Table 3 the bond angles, and Table 4 the torsion angles. Fig. 1 shows an *ORTEP*

* Lists of structure factors, anisotropic thermal parameters for non-hydrogen atoms and positional and thermal parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35913 (73 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

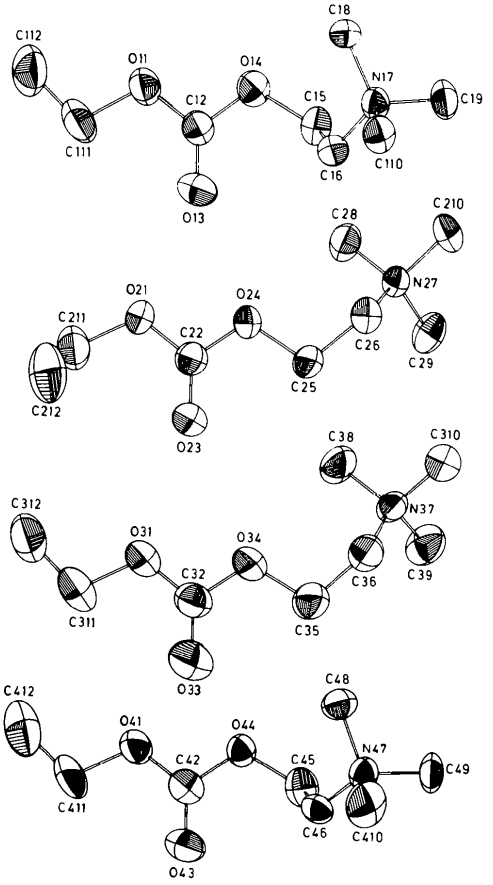

Fig. 1. A view of the four ethoxycarbonylcholine ions showing the atom numbering. Atoms are represented by thermal ellipsoids drawn at the 50% probability level.

Table 1. *Fractional atomic coordinates* ($\times 10^4$, *for* I $\times 10^5$) *and equivalent isotropic temperature factors*

	x	y	z	B_{eq} (Å²)†
I(1)	23215 (6)	55371 (6)	50174 (5)	4·8
I(2)	22285 (6)	23613 (5)	21424 (6)	4·8
I(3)	28666 (5)	55265 (4)	114 (5)	3·7
I(4)	12883 (7)	18050 (7)	68834 (7)	6·1
C(112)	−203 (13)	6860 (12)	9720 (10)	7·0
C(111)	863 (12)	7404 (10)	9422 (10)	6·3
O(11)	1198 (6)	6897 (5)	8581 (6)	4·7
C(12)	2301 (8)	7168 (7)	8350 (7)	3·6
O(13)	2998 (7)	7807 (5)	8713 (6)	4·9
O(14)	2489 (5)	6592 (5)	7619 (5)	4·2
C(15)	3668 (8)	6776 (7)	7289 (8)	4·0
C(16)	4502 (8)	6379 (7)	7808 (7)	3·6
N(17)	4671 (6)	5401 (5)	7466 (6)	3·2
C(18)	3524 (8)	4721 (7)	7363 (8)	4·5
C(19)	5282 (9)	5395 (8)	6585 (8)	3·8
C(110)	5416 (8)	5120 (7)	8135 (8)	4·1
C(212)	506 (11)	9571 (12)	1027 (13)	8·1
C(211)	1370 (10)	9554 (8)	1713 (10)	5·4
O(21)	2166 (6)	8909 (5)	1432 (6)	4·7
C(22)	3052 (8)	9217 (7)	939 (8)	3·9
O(23)	3247 (6)	9960 (5)	652 (7)	5·6
O(24)	3733 (5)	8578 (4)	820 (5)	3·9
C(25)	4731 (7)	8822 (6)	257 (8)	3·7
C(26)	5287 (8)	7972 (6)	15 (7)	3·2
N(27)	6140 (6)	7810 (5)	677 (6)	3·1
C(28)	5613 (9)	7756 (8)	1580 (8)	4·4
C(29)	7233 (8)	8580 (8)	746 (9)	4·4
C(210)	6498 (9)	6883 (7)	343 (9)	5·1
C(312)	6723 (12)	9748 (11)	6155 (14)	8·1
C(311)	5880 (12)	9803 (11)	6865 (12)	7·2
O(31)	4749 (7)	9264 (6)	6528 (7)	6·1
C(32)	3845 (10)	9399 (8)	7021 (10)	4·8
O(33)	3840 (8)	9898 (7)	7703 (8)	7·3
O(34)	2889 (6)	8843 (6)	6621 (6)	5·4
C(35)	1795 (10)	8910 (9)	7056 (9)	5·2
C(36)	905 (9)	8052 (8)	6654 (8)	4·7
N(37)	360 (6)	8105 (6)	5774 (6)	3·8
C(38)	1264 (10)	8272 (10)	5090 (9)	5·8
C(39)	−370 (10)	8847 (9)	5874 (9)	5·8
C(310)	−438 (10)	7167 (9)	5489 (9)	5·3
C(412)	6150 (15)	7057 (12)	4844 (12)	7·8
C(411)	5331 (13)	7572 (11)	4539 (12)	7·8
O(41)	4768 (6)	7081 (5)	3695 (6)	4·7
C(42)	3799 (9)	7331 (7)	3471 (8)	3·9
O(43)	3375 (7)	7964 (5)	3830 (6)	5·5
O(44)	3368 (5)	6761 (4)	2729 (5)	3·5
C(45)	2292 (9)	6949 (7)	2386 (8)	4·3
C(46)	1252 (8)	6603 (6)	2915 (7)	3·5
N(47)	559 (6)	5641 (6)	2555 (6)	3·6
C(48)	1339 (8)	4915 (7)	2424 (8)	5·1
C(49)	−55 (9)	5645 (9)	1698 (9)	4·1
C(410)	−349 (9)	5365 (10)	3231 (9)	5·2

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

(Johnson, 1971) drawing of the four crystallographically independent ethoxycarbonylcholine ions. It can be seen that none of the four ethoxycarbonylcholine ions has a conformation similar to methoxycarbonylcholine iodide (Jensen, 1979). Two of the four ions (1 and 4) have conformations which in

Table 2. *Bond lengths* (Å)

	$n=1$	$n=2$	$n=3$	$n=4$
C(n12)–C(n11)	1·44 (2)	1·46 (2)	1·46 (2)	1·44 (3)
C(n11)–O(n1)	1·48 (2)	1·47 (1)	1·46 (2)	1·48 (2)
O(n1)–C(n2)	1·33 (1)	1·31 (1)	1·32 (2)	1·31 (1)
C(n2)–O(n3)	1·19 (1)	1·21 (1)	1·19 (2)	1·20 (1)
C(n2)–O(n4)	1·35 (1)	1·33 (1)	1·34 (1)	1·35 (1)
O(n4)–C(n5)	1·44 (1)	1·45 (1)	1·44 (1)	1·45 (1)
C(n5)–C(n6)	1·50 (2)	1·51 (1)	1·53 (2)	1·49 (1)
C(n6)–N(n7)	1·51 (1)	1·49 (1)	1·51 (2)	1·52 (1)
N(n7)–C(n8)	1·50 (1)	1·51 (1)	1·48 (2)	1·52 (1)
N(n7)–C(n9)	1·51 (1)	1·52 (1)	1·49 (2)	1·50 (2)
N(n7)–C(n10)	1·49 (1)	1·52 (1)	1·52 (1)	1·51 (1)

Table 3. *Bond angles* (°)

	$n=1$	$n=2$	$n=3$	$n=4$
C(n12)–C(n11)–O(n1)	108·1 (10)	112·8 (12)	108·0 (13)	110·4 (13)
C(n11)–O(n1)–C(n2)	114·8 (8)	117·0 (9)	115·3 (11)	116·0 (10)
O(n1)–C(n2)–O(n3)	126·7 (10)	126·7 (10)	128·2 (11)	127·4 (10)
O(n1)–C(n2)–O(n4)	107·4 (7)	108·5 (9)	107·3 (11)	107·4 (9)
O(n3)–C(n2)–O(n4)	126·0 (9)	124·8 (9)	124·5 (11)	125·1 (10)
C(n2)–O(n4)–C(n5)	114·6 (7)	114·0 (8)	115·5 (9)	114·6 (8)
O(n4)–C(n5)–C(n6)	112·1 (9)	109·1 (8)	106·4 (9)	112·5 (9)
C(n5)–C(n6)–N(n7)	116·5 (8)	116·1 (8)	116·8 (10)	115·0 (8)
C(n6)–N(n7)–C(n8)	111·4 (7)	112·1 (7)	111·3 (8)	111·4 (7)
C(n6)–N(n7)–C(n9)	110·1 (8)	110·7 (8)	110·0 (9)	112·2 (9)
C(n6)–N(n7)–C(n10)	106·8 (7)	107·1 (8)	107·4 (9)	107·1 (8)
C(n8)–N(n7)–C(n9)	109·4 (7)	109·4 (8)	111·3 (10)	109·0 (8)
C(n8)–N(n7)–C(n10)	109·1 (8)	109·0 (9)	108·5 (9)	108·6 (9)
C(n9)–N(n7)–C(n10)	109·9 (8)	108·2 (7)	108·3 (9)	108·5 (8)

discussions about the flexibility of acetylcholine are often referred to as *gauche–gauche*. The two remaining ethoxycarbonylcholine ions (2 and 3) have the so-called *trans–gauche* conformation. The ethoxy group of (2) is, presumably due to packing effects, found in the less stable *gauche* conformation. Corresponding bond lengths of the four independent ions do not show significant differences. This is also true for most of the bond angles, but the angle O(n4)–C(n5)–C(n6) is found to be more open in *gauche–gauche* than in *trans–gauche* conformers.

Packing

A stereodiagram of the packing is shown in Fig. 2. The packing seems to be dominated by weak polar interactions and no clearly hydrophobic areas are found. The I^- ions have contacts to a number of the methyl and methylene groups of the quaternary ammonium groups, but other types of contacts also seem to be important. Contacts from the ester groups of ions (1) and (4) to an I^- ion are listed in Table 5, and the positions of these I^- ions relative to the ester group seem to be very favourable. This is shown in Fig. 3, which depicts a projection of pertinent ions and the potential of dimethyl carbonate as calculated by Johansen, Rettrup & Jensen (1980). Very similar contacts between a halogenide ion and the ester group

Table 4. *Main torsion angles* (°)

	$n=1$	$n=2$	$n=3$	$n=4$
C(n12)–C(n11)–O(n1)–C(n2)	∓162·7 (12)	±81·1 (12)	∓166·6 (13)	±162·4 (12)
C(n11)–O(n1)–C(n2)–O(n4)	±175·0 (10)	±174·2 (9)	∓179·2 (12)	∓175·2 (10)
O(n1)–C(n2)–O(n4)–C(n5)	∓178·5 (9)	±178·1 (8)	∓178·6 (11)	∓179·6 (8)
C(n2)–O(n4)–C(n5)–C(n6)	±79·2 (11)	∓168·3 (8)	∓164·5 (11)	∓75·6 (10)
O(n4)–C(n5)–C(n6)–N(n7)	±92·0 (10)	∓83·8 (9)	∓80·8 (12)	∓95·8 (10)
C(n5)–C(n6)–N(n7)–C(n8)	∓54·7 (12)	±55·2 (10)	±58·7 (13)	±54·3 (12)
C(n5)–C(n6)–N(n7)–C(n9)	±67·0 (10)	∓67·3 (11)	∓65·2 (12)	∓68·1 (11)
C(n5)–C(n6)–N(n7)–C(n10)	∓173·7 (8)	±174·8 (8)	±177·3 (10)	±172·9 (10)

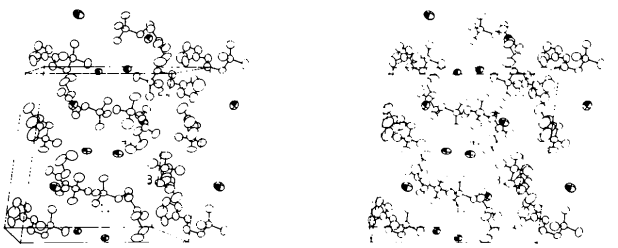

Fig. 2. Stereoview of the packing. y is →, x is ↓.

Table 5. *Distances* (Å) *from gauche–gauche ethoxycarbonylcholine ions to related* I^- *ions*

	$n=1$, $m_q=3_{(x,y,1+z)}$	$n=4$, $m_q=1_{(x,y,z)}$
C(n11)···I^-(m_q)	4·13 (2)	4·28 (2)
O(n1)···I^-(m_q)	3·93 (1)	4·00 (1)
C(n2)···I^-(m_q)	3·87 (2)	3·91 (1)
O(n3)···I^-(m_q)	4·09 (1)	4·18 (1)
O(n4)···I^-(m_q)	4·22 (1)	4·20 (1)
C(n6)···I^-(m_q)	4·07 (1)	4·07 (1)
C(n8)···I^-(m_q)	4·16 (2)	4·07 (2)
C(n10)···I^-(m_q)	4·17 (1)	4·12 (2)

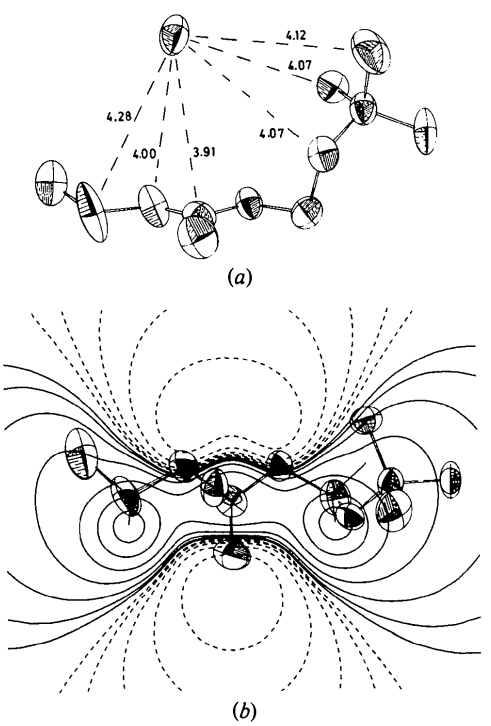

Fig. 3. (*a*) Ethoxycarbonylcholine ion 4 with an I^- ion. (*b*) The same ions with the electrostatic potential calculated for dimethyl carbonate. The ester moiety of the ethoxycarbonylcholine ion is merging with the corresponding atoms of dimethyl carbonate. The electrostatic potential is that found in a plane $3a_o$ (~1·6 Å) above the plane of the ester atoms, and it represents fairly well the potential which meets the I^- ion. Dashed lines indicate negative regions. First contours are ±4·2 kJ mol^{-1}; neighbouring contours differ by a factor of 2.

are also found in the crystals of acetylcholine bromide (Svinning & Sørum, 1975) and of acetylcholine iodide (Jagner & Jensen, 1977), in which one of the I^- ions in the asymmetric unit has contacts of the type described above to four surrounding ester groups. Such contacts may contribute to the stabilization of the *gauche–gauche* conformation. However, *gauche–gauche* conformers have also been found in crystal structures in which no such contacts to single ions exist, *e.g.* in the two crystal forms of acetylcholine hydrogen (+)-tartrate (Jensen, 1977).

The activity of methoxy- and ethoxycarbonylcholine iodides on nicotinic as well as muscarinic acetylcholine receptors has been investigated by Lambrecht (1979). While methoxycarbonylcholine iodide was found to have substantial agonistic effects on both types of receptors, the effects of ethoxycarbonylcholine iodide were very weak. These differences in the biological activity can hardly be related to the conformational differences observed in the crystal structures. In solution all choline esters have been found to prefer the *gauche* O–C–C–$\overset{+}{N}$ conformation, but the energy barriers and the energy differences between different conformers are low (*e.g.* Pullman & Port, 1973). Calculations (Johansen, Rettrup & Jensen, 1980) have shown that the electronic properties of the ester moiety of the methoxy and the ethoxy compound are essentially identical, and therefore only packing forces can be responsible for the variation observed in the solid-state conformations of the alkyloxycarbonylcholine esters. In cholinergic agonists the groups which can be regarded as corresponding to the acetoxy group in acetylcholine

are generally very small, while antagonists have voluminous groups in this area. The most reasonable explanation for the lack of biological activity of ethoxycarbonylcholine iodide seems to be that the ethoxy group is too big to allow agonist activity and too small to give the compound antagonist character.

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Structure de la (Dichloro-2,6 phényl)-2 Méthyl-1 Guanidine Base et Chlorohydrate; un Analogue Ouvert de la Clonidine

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Abstract

$C_8H_9Cl_2N_3$ is a ring-opened analogue of clonidine which exhibits hypotensive activity. Crystals of the free base (M_r = 218·1) and hydrochloride.$\frac{1}{2}H_2O$ (M_r = 263·5) have been isolated and studied by X-ray single-crystal diffraction methods. Crystals of the free base are monoclinic, space group $P2_1/c$, with a = 7·911 (2), b = 15·569 (1), c = 9·130 (3) Å, β = 67·76 (2)°, Z = 4, D_x = 1·39 Mg m^{-3}, $F(000)$ = 448, whereas crystals of the hydrated hydrochloride, $C_8H_{10}Cl_2N_3^+.Cl^-.\frac{1}{2}H_2O$, are monoclinic, space group $C2/c$, with a = 17·887 (2), b = 12·775 (1), c = 13·293 (1) Å, β = 52·60 (1)°, Z = 8, D_x = 1·45 Mg m^{-3}, $F(000)$ = 1120. Both structures were solved by direct methods and the atomic parameters were refined by a least-squares procedure, giving final R factors of 0·046 and 0·056 for 1365 and 1902 structure amplitudes respectively. The free base occurs as a dimer. The Cl^- and H_2O are involved in hydrogen bonds with the three partly positively charged N atoms in the salt.

Introduction

Les structures cristallines du phosphate de clonidine (Carpy, Hickel & Leger, 1979*a*) et de quelques composés apparentés à ce médicament tels le phosphate de xylazine (Carpy, Gadret & Leger, 1979), le nitrate de tolonidine (Carpy, Hickel & Leger, 1979*b*), la tiaménidine sous forme base (Leger, Hickel & Carpy, 1979), *etc.* ont été récemment déterminées dans le but d'essayer de dégager des relations conformation–activité dans ce groupe particulièrement intéressant d'antihypertenseurs.

Parmi une série d'aryl et d'arylalkylguanidines synthétisées et testées pour leurs propriétés hypotensives (Rouot, Leclerc, Wermuth, Miesch & Schwartz, 1978), la (dichloro-2,6 phényl)-2 méthyl-1

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