

two-dimensionally correlated NMR spectra; in the  $^1\text{H}$  spectrum, singlets at 4.89 p.p.m. ( $2H$ ) and 6.18 p.p.m. ( $1H$ ) are assigned respectively to H(C5) and H(C10), whereas the doublets ( $J = 5.7$  Hz) at 6.28 p.p.m. ( $1H$ ) and 7.12 p.p.m. ( $1H$ ) are the H(C1) and H(C2) signals; the aromatic protons resonate between 7.15 and 7.25 p.p.m. The only  $^{13}\text{C}$  aliphatic signal (47.5 p.p.m.) is assigned to C(5);  $^1\text{H}$ - $^{13}\text{C}$  correlated spectra allow the assignment of the C(10) (110.4 p.p.m.), C(2) (124.3 p.p.m.) and C(1) (133.2 p.p.m.) signals. The carbonyl moiety of the amide group resonates at 169.2 p.p.m.

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## Structure of (4*RS*)-6,7-Dimethoxy-4-phenyl-1,2,3,4-tetrahydroisoquinoline Hydrochloride

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**Abstract.**  $\text{C}_{17}\text{H}_{20}\text{NO}_2^+ \cdot \text{Cl}^-$ ,  $M_r = 305.80$ , triclinic,  $PI$ ,  $a = 5.959$  (1),  $b = 14.823$  (2),  $c = 19.307$  (2) Å,  $\alpha = 107.53$  (1),  $\beta = 95.96$  (2),  $\gamma = 81.94$  (1)°,  $V = 1607$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.25$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 20.26$  cm<sup>-1</sup>,  $F(000) = 324$ , room temperature,  $R = 0.0611$ ,  $wR = 0.0596$  for 2564 observed reflections. The two independent molecules *A* and *B* in the asymmetric unit show conformational differences. The methoxy groups are coplanar with the benzene ring and the flexible aromatic phenyl rings effectively rotated out of conjugation with the isoquinoline system, thus leading to favourable packing arrangements. Small conformational differences are observed in the piperidine rings: molecule *A* is best described as sofa or half boat; molecule *B* as half chair.

**Experimental.** The synthesis of the title compound has been reported previously (Mondeshka, Ivanov, Angelova, Pavlova, Uzunov, Panova, 1989). Colourless transparent crystals were obtained by

slow evaporation from methanol at room temperature. A selected crystal with dimensions  $0.58 \times 0.20 \times 0.20$  mm was mounted on a Siemens AED2 automated diffractometer equipped with a monochromated Cu  $K\alpha$  radiation source. Accurate unit-cell parameters were determined by a least-squares fit to setting angles of 35 strong and well centred reflections in the  $2\theta$  range  $25$ – $35^\circ$ . 4902 unique reflections were measured by the  $\omega$ - $2\theta$  scan technique up to a maximum of  $140^\circ$  in  $2\theta$ , covering the  $hkl$  range  $0 \leq h \leq 17$ ,  $-17 \leq k \leq 17$  and  $-23 \leq l \leq 23$ . Three standard reflections monitored periodically every 120 min showed no significant intensity variation. Data reduction included correction for background, Lorentz, polarization and absorption effects. The applied min. and max. transmission factors were 0.943 and 0.967, respectively. 2564 reflections were considered observed with  $I \geq 3\sigma(I)$ . The structure was solved by direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and subjected to blocked full-matrix least-

Table 1. Atomic coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses

$$B_{\text{eq}} = (4/3) \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
<b>Molecule A*</b>				
Cl(A)	0.8651 (3)	0.5492 (1)	1.1148 (1)	3.75 (5)
C(1)	0.5847 (12)	0.6004 (6)	0.9243 (3)	3.4 (2)
N(2)	0.8040 (12)	0.6185 (5)	0.9697 (3)	3.4 (2)
C(3)	0.8726 (13)	0.7121 (5)	0.9760 (3)	3.2 (2)
C(4)	0.9317 (11)	0.7177 (4)	0.9011 (3)	2.4 (2)
C(4A)	0.7652 (10)	0.6648 (4)	0.8401 (3)	2.1 (2)
C(5)	0.7919 (10)	0.6698 (4)	0.7691 (3)	2.5 (2)
C(6)	0.6565 (10)	0.6196 (4)	0.7112 (3)	2.6 (2)
O(6)	0.6736 (8)	0.6190 (3)	0.6404 (2)	3.7 (2)
C(61)	0.8426 (15)	0.6670 (6)	0.6258 (4)	4.3 (3)
C(7)	0.4973 (10)	0.5680 (4)	0.7222 (3)	2.4 (2)
O(7)	0.3727 (7)	0.5213 (3)	0.6612 (2)	3.6 (1)
C(71)	0.2112 (14)	0.4644 (6)	0.6696 (4)	4.2 (3)
C(8)	0.4714 (11)	0.5624 (4)	0.7918 (3)	2.6 (2)
C(8A)	0.6110 (9)	0.6120 (4)	0.8502 (3)	2.2 (2)
C(41)	0.9265 (11)	0.8196 (4)	0.9029 (3)	2.5 (2)
C(42)	0.7391 (12)	0.8867 (5)	0.9245 (4)	3.3 (2)
C(43)	0.7348 (15)	0.9795 (5)	0.9251 (4)	4.4 (3)
C(44)	0.9219 (18)	1.0069 (6)	0.9034 (4)	4.8 (3)
C(45)	1.1003 (17)	0.9445 (7)	0.8805 (4)	5.1 (3)
C(46)	1.1100 (13)	0.8491 (5)	0.8804 (4)	3.8 (2)
<b>Molecule B</b>				
Cl(B)	0.4448 (3)	-0.2390 (1)	-0.9072 (1)	4.39 (6)
C(1)'	0.2530 (13)	-0.2972 (6)	-0.7508 (3)	3.6 (2)
N(2)'	0.1163 (11)	-0.2739 (4)	-0.8142 (3)	3.6 (2)
C(3)'	0.0064 (14)	-0.1767 (6)	-0.7958 (4)	3.8 (2)
C(4)'	-0.1725 (11)	-0.1557 (4)	-0.7391 (3)	2.7 (2)
C(4A)'	-0.0782 (10)	-0.2002 (4)	-0.6782 (3)	2.5 (2)
C(5)'	-0.2002 (11)	-0.1747 (5)	-0.6145 (3)	2.8 (2)
C(6)'	-0.1217 (11)	-0.2155 (5)	-0.5594 (3)	3.0 (2)
O(6)'	-0.2359 (8)	-0.1960 (3)	-0.4964 (2)	4.3 (2)
C(61)'	-0.4172 (18)	-0.1208 (7)	-0.4835 (5)	6.2 (4)
C(7)'	0.0674 (11)	-0.2789 (4)	-0.5652 (3)	2.8 (2)
O(7)'	0.1257 (8)	-0.3161 (3)	-0.5078 (2)	4.0 (2)
C(71)'	0.3304 (16)	-0.3795 (7)	-0.5101 (4)	5.1 (3)
C(8)'	0.1884 (12)	-0.3049 (5)	-0.6272 (3)	3.1 (2)
C(8A)'	0.1090 (11)	-0.2636 (4)	-0.6842 (3)	2.7 (2)
C(41)'	-0.2381 (11)	-0.0509 (5)	-0.7154 (3)	3.0 (2)
C(42)'	-0.4334 (14)	-0.0107 (6)	-0.7471 (4)	4.3 (3)
C(43)'	-0.4863 (16)	0.0870 (7)	-0.7298 (5)	5.0 (3)
C(44)'	-0.3487 (17)	0.1447 (7)	-0.6830 (5)	4.8 (3)
C(45)'	-0.1595 (16)	0.1086 (6)	-0.6506 (4)	4.5 (3)
C(46)'	-0.1037 (14)	0.0117 (6)	-0.6667 (4)	4.0 (2)

\* The xyz coordinates of the two chiral molecules A and B are given for the 4(R)-configurational state.

squares refinement with the *SHELX76* system (Sheldrick, 1976). The function minimized was  $\sum w(|F_o| - |F_c|)^2$  where  $w^{-1} = \sigma^2(F_o) + 0.0002(F_o)^2$  and the e.s.d.'s,  $\sigma(F_o)$ , were based on counting statistics. All non-H atoms were refined anisotropically, the H-atom positions were located in a difference Fourier map and included with different isotropic group thermal factors for methyl, phenyl and other H atoms. Eleven low-angle reflections:  $\bar{1}10$ ,  $010$ ,  $\bar{1}20$ ,  $020$ ,  $\bar{1}30$ ,  $140$ ,  $240$ ,  $0\bar{3}1$ ,  $0\bar{4}1$ ,  $\bar{1}31$  and  $\bar{1}31$ , with  $||F_o| - |F_c|| > 3\sigma$ , were excluded from the refinement in the last cycles. The refinement was terminated when all shift/e.s.d. ratios were less than 0.006 for non-H atoms and 0.02 for H atoms. The final discrepancy indices were  $R = 0.0611$  and  $wR = 0.0596$ ,  $S = 0.82$ . Max. and min. residual heights in electron density are 0.37 and  $-0.78 \text{ e \AA}^{-3}$ . Atomic scattering factors taken from *SHELX76*.

Table 2. Bond lengths ( $\text{\AA}$ ), angles ( $^\circ$ ), selected torsion angles ( $^\circ$ ) and H-bonding scheme for the two independent molecules A and B with e.s.d.'s in parentheses

	Molecule A	Molecule B	Molecule A	Molecule B
C(1)—N(2)	1.505 (9)	1.500 (9)	O(6)—C(61)	1.407 (11)
C(1)—C(8A)	1.518 (9)	1.538 (9)	C(7)—O(7)	1.374 (7)
N(2)—C(3)	1.468 (11)	1.451 (10)	C(7)—C(8)	1.396 (9)
C(3)—C(4)	1.552 (9)	1.541 (10)	O(7)—C(71)	1.416 (11)
C(4)—C(4A)	1.551 (8)	1.537 (9)	C(8)—C(8A)	1.408 (8)
C(4)—C(41)	1.497 (9)	1.488 (9)	C(41)—C(42)	1.395 (9)
C(4A)—C(5)	1.421 (8)	1.418 (9)	C(41)—C(46)	1.387 (11)
C(4A)—C(8A)	1.352 (9)	1.345 (8)	C(42)—C(43)	1.368 (11)
C(5)—C(6)	1.390 (8)	1.386 (10)	C(43)—C(44)	1.383 (14)
C(6)—O(6)	1.380 (7)	1.390 (8)	C(44)—C(45)	1.326 (13)
C(6)—C(7)	1.369 (10)	1.354 (9)	C(45)—C(46)	1.406 (13)
			<b>Molecule A</b>	<b>Molecule B</b>
N(2)—C(1)—C(8A)			108.3 (7)	109.0 (7)
C(1)—N(2)—C(3)			112.7 (7)	112.8 (5)
N(2)—C(3)—C(4)			111.0 (6)	113.6 (7)
C(3)—C(4)—C(41)			109.8 (5)	107.6 (7)
C(3)—C(4)—C(4A)			110.0 (6)	109.3 (7)
C(4A)—C(4)—C(41)			111.7 (5)	115.5 (5)
C(4)—C(4A)—C(8A)			124.3 (5)	122.5 (6)
C(4)—C(4A)—C(5)			115.6 (7)	117.7 (7)
C(5)—C(4A)—C(8A)			120.0 (6)	119.7 (6)
C(4A)—C(5)—C(6)			118.5 (7)	118.8 (8)
C(5)—C(6)—C(7)			121.0 (5)	121.6 (6)
C(5)—C(6)—O(6)			122.5 (7)	122.1 (8)
O(6)—C(6)—C(7)			116.5 (6)	116.3 (6)
C(6)—O(6)—C(61)			118.6 (6)	118.2 (6)
C(6)—C(7)—C(8)			120.6 (7)	120.4 (7)
C(6)—C(7)—O(7)			116.1 (6)	116.3 (7)
O(7)—C(7)—C(8)			123.2 (7)	123.3 (7)
C(7)—O(7)—C(71)			118.2 (5)	118.0 (6)
C(7)—C(8)—C(8A)			118.4 (7)	118.6 (8)
C(4A)—C(8A)—C(8)			121.4 (6)	120.9 (7)
C(1)—C(8A)—C(8)			116.6 (7)	114.8 (7)
C(1)—C(8A)—C(4A)			121.9 (6)	124.2 (6)
C(4)—C(41)—C(46)			119.7 (8)	122.8 (8)
C(4)—C(41)—C(42)			122.3 (8)	120.7 (7)
C(42)—C(41)—C(46)			118.0 (8)	116.7 (8)
C(41)—C(42)—C(43)			121.8 (9)	120.8 (8)
C(42)—C(43)—C(44)			118.8 (9)	120.2 (10)
C(43)—C(44)—C(45)			121.0 (10)	120.8 (10)
C(44)—C(45)—C(46)			121.2 (11)	120.1 (9)
C(41)—C(46)—C(45)			119.1 (9)	121.4 (8)
N(2)—C(1)—C(8A)—C(4A)*			-23.4 (11)	-17.7 (12)
C(8A)—C(1)—N(2)—C(3)			56.1 (9)	47.8 (10)
C(1)—N(2)—C(3)—C(4)			-66.0 (9)	-64.2 (10)
N(2)—C(3)—C(4)—C(4A)			38.3 (9)	43.4 (10)
N(2)—C(3)—C(4)—C(41)			161.6 (7)	169.5 (7)
C(3)—C(4)—C(4A)—C(8A)			-7.6 (11)	-13.0 (11)
C(4)—C(4A)—C(8A)—C(1)			0.7 (12)	1.5 (13)
N2(A)—H2b(A)···Cl(A)†			3.233 (7) $\text{\AA}$	(x, y, z)
N2(A)—H2a(A)···Cl(A)			3.096 (6)	(2-x, 1-y, 2-z)
N2(B)—H2b(B)···Cl(A)			3.083 (6)	(1+x, 1+y, 2+z)
N2(B)—H2a(B)···Cl(B)			2.982 (7)	(x, y, z)

\* The torsion angles of the two chiral molecules A and B are given for the 4(R) configurational state.

† H-bond distances ( $\text{\AA}$ ) between the protonated N atoms and the Cl anions of the two independent A and B molecules. The symmetry translation code (in parentheses) refers to the N atom.

Fractional coordinates of non-H atoms are given in Table 1. ‡ Table 2 reports bond distances, bond angles, some selected torsion angles and the H-bond geometry. Fig. 1 shows a perspective view of the

‡ Lists of structure factors, anisotropic thermal parameters, H-atom coordinates with isotropic thermal parameters and some selected topographical data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53568 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

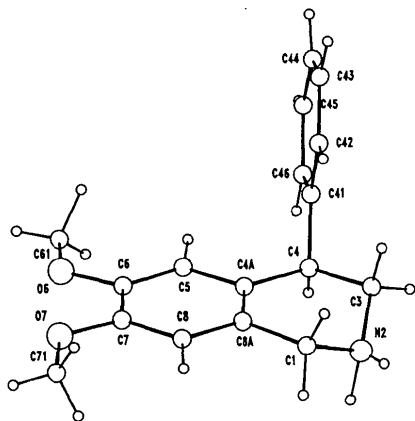


Fig. 1. A plot of molecule *A* showing the atomic numbering scheme. The numbering of molecule *B* follows the same pattern but is primed in the lists for clarity. The chirally oriented phenyl ring adopts the 4(*R*) configurational state.

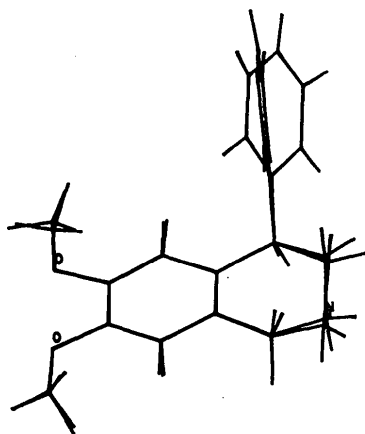


Fig. 2. Geometrical fit of the two independent molecules *A* and *B* [each molecule in the 4(*R*) chiral form] showing the conformational displacement of the phenyl rings. In molecule *A* the phenyl ring is perpendicular to the plane of the paper.

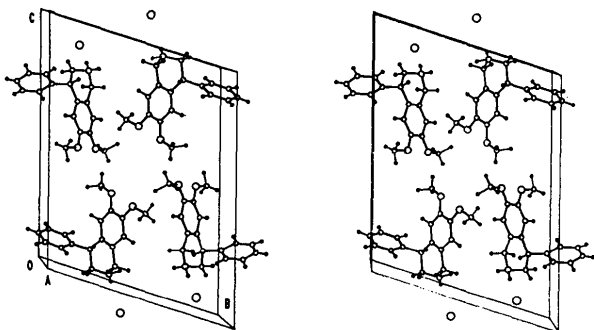


Fig. 3. A crystal packing diagram showing the unit-cell contents viewed down the *a* axis. The large open circles represent the Cl atoms.

4(*R*)-chiral state of molecule *A* with the atom-numbering scheme; the numbering of molecule *B* follows the same pattern (primed in the lists for clarity). A comparative overlay of the two independent molecules *A* and *B* is presented in Fig. 2, clearly visualizing the displacement of the phenyl rings. All H atoms attached to the N atoms in molecules *A* and *B* are involved in H bonding to the chloride anions (*cf.* Table 2). In Fig. 3 the packing diagram is illustrated.

**Related literature.** A variety of 4-phenyl-1,2,3,4-tetrahydroisoquinoline derivatives have been extensively examined in order to elucidate their antidepressant-like activity. Structural data of several compounds have been published by Toome, Blount, Grethe & Uskokovic (1970), Kaiser, Dandridge, Garvey, Hahn, Sarau, Setler, Bass & Clardy (1982), Kobayashi, Tokumoto & Taira (1984), Dandridge, Kaiser, Brenner, Gaitanopoulos, Davis, Webb, Foley & Sarau (1984), Kunstmann, Gerhards, Kruse, Leven, Paulus, Schacht, Schmitt & Witte (1987) and Maryanoff, McComsey, Gardocki, Shank, Costanzo, Nortey, Schneider & Setler (1987).

The absolute stereochemistry of the two optical antipodes of the title compound (HCl forms) has also been investigated by X-ray diffraction methods, thus permitting assignments of the antipodes as the (+)-4(*R*)- and (-)-4(*S*)-enantiomers. Their crystal structures are isostructurally related, crystallizing in the monoclinic space group  $P2_1$  ( $Z = 2$ ), and within experimental errors adopting the same unit-cell parameters. A complete three-dimensional X-ray investigation of the (+)-4(*R*)-enantiomer has been performed. The resolution and absolute stereochemistry of 6,7-dimethoxy-4-phenyl-1,2,3,4-tetrahydroisoquinoline will be published elsewhere (Mondeshka, Angelova, Stensland, Werner & Ivanov, 1990).

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## [2-(1-Aza-4,7,10-trioxacyclododecyl)ethyl]dimethylammonium Iodide. An Intramolecular Trifurcated Hydrogen Bond

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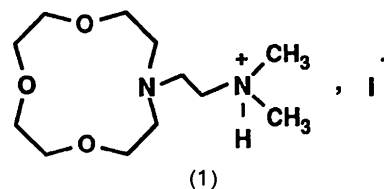
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**Abstract.**  $C_{12}H_{27}N_2O_3^+ \cdot I^-$ ,  $M_r = 374.3$ , monoclinic,  $P2_1/c$ ,  $a = 23.925$  (3),  $b = 7.7933$  (8),  $c = 19.182$  (3) Å,  $\beta = 113.113$  (10)°,  $V = 3289.5$  (8) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.511$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 19.3$  cm<sup>-1</sup>,  $F(000) = 1520$ ,  $T = 296$  K,  $R = 0.045$  for 3684 observations having  $I > 1\sigma(I)$  (of 5785 unique data). There are two independent formula units in the asymmetric unit. The two macrocyclic cations have nearly identical conformations, with the 12-membered ring in the crown conformation, having all four donor atoms on the same side of the ring. The side arm, which contains the quaternary ammonium, is folded over the ring, with the H atom pointing towards the ring. The two independent cations exhibit a mean difference in 15 torsion angles describing their conformations of only 1.5°, with a maximum individual difference of 4.7 (11)°. The N—H hydrogen atom is involved in a trifurcated intramolecular hydrogen bond with the ring N and two O atoms of the ring. The N...acceptor distance range is 2.828 (8)–3.158 (8) Å, and the H...acceptor distance range is 2.25 (10)–2.40 (10) Å.

**Experimental.** A light-yellow irregular crystal fragment of (1), m.p. 475–476 K, grown by slow evaporation from ethanol, having approximate dimensions 0.38 × 0.35 × 0.32 mm, mounted in a glass capillary in random orientation, was used for data collection on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite-crystal incident-beam monochromator and Mo  $K\alpha$  radiation. Cell dimensions were obtained from setting angles of 25 reflections

having  $25 < \theta < 30^\circ$  using Cu  $K\alpha$  radiation ( $\lambda = 1.54184$  Å). Space group determined to be  $P2_1/c$  from systematic absences  $h0l$  with  $l$  odd,  $0k0$  with  $k$  odd.



One quadrant of data was collected ( $0 \leq h \leq 28$ ,  $0 \leq k \leq 9$ ,  $-22 \leq l \leq 20$ ) using  $\omega-2\theta$  scans, within  $1 < \theta < 25^\circ$ . Scan rates varied  $1.0-4.0^\circ \text{ min}^{-1}$ . Three standard reflections (800, 040, 008), measured every 10 000 s, decreased in intensity by 9.7%; thus a linear decay correction was applied. Lorentz and polarization corrections were applied. An empirical absorption correction, based on a series of  $\psi$  scans, yielded relative transmission coefficients ranging from 0.798 to 0.997.

The transformation (001,  $\bar{2}0\bar{1}$ ,  $0\bar{1}0$ ) yields a  $C$ -centered cell with near-orthorhombic metric, cell dimensions  $a = 19.182$  (3),  $b = 44.010$  (6),  $c = 7.7933$  (8) Å,  $\alpha = \beta = 90$ ,  $\gamma = 90.52$  (1)°. Diffraction patterns resemble symmetry  $mmm$ . In order to ascertain that the symmetry is  $2/m$ , a full sphere of low-angle data having  $1 < \theta < 15^\circ$ ,  $-17 < h < 17$ ,  $-5 < k < 5$ ,  $-13 < l < 13$  was collected in the same fashion as the original quadrant. The value of  $R_{\text{int}}$  for the low-angle data averaged under  $mmm$  symmetry was 0.113, while  $R_{\text{int}}$  for all 10 739 data averaged

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