

## Structure of Trimethyl[(*cis*-3-methylcyclopentyl)methyl]ammonium Iodide, C<sub>10</sub>H<sub>22</sub>N<sup>+</sup>·I<sup>-</sup>

BY R. BARDI AND A. M. PIAZZESI

*Biopolymer Research Centre, University of Padua, Via Marzolo 1, 35100 Padova, Italy*

AND A. DEL PRA AND L. VILLA

*Istituto Chimico Farmaceutico, University of Milan, Viale Abruzzi 42, 20131 Milano, Italy*

(Received 30 November 1982; accepted 9 February 1983)

**Abstract.**  $M_r = 283.2$ , orthorhombic,  $Pna2_1$ ,  $a = 13.359$  (5),  $b = 12.315$  (5),  $c = 8.007$  (4) Å,  $Z = 4$ ,  $U = 1317.3$  Å<sup>3</sup>,  $D_m = 1.42$ ,  $D_x = 1.427$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 2.37$  mm<sup>-1</sup>,  $F(000) = 568$ ,  $T = 298$  K. Final  $R = 0.066$  for 666 observed reflections. The overall conformation of the cation, described by the torsion angle N(1)–C(4)–C(5)–C(6),  $-167^\circ$ , markedly differs from that found in many muscarinic agents.

**Introduction.** The crystal structure of the title compound (TMC2) has been determined as part of an investigation of the conformational properties of some muscarinic agents, which we are examining in an attempt to correlate molecular conformation and biological activity.

**Experimental.** Single crystals obtained by slow evaporation of a solution in absolute ethanol, under reduced constant pressure in a dry atmosphere,  $D_m$  by flotation, crystal  $\sim 0.12 \times 0.12 \times 0.15$  mm; Philips PW 1100, graphite monochromator; unit-cell parameters obtained from least-squares refinement of angular values of 20 carefully centered high-angle reflections;  $\theta$ – $2\theta$  scan, scan speed  $0.025^\circ\text{s}^{-1}$ , scan width  $1.00^\circ$ ,  $2\theta$  range  $4$ – $50^\circ$ ; two standard reflections every 180 min; 1236 data measured, 666 with  $I \geq 3\sigma(I)$ ; trial structure obtained by heavy-atom method, refined by full-matrix least squares,  $\sum w(|F_o| - |F_c|)^2$  minimized,  $w = 2.725[\sigma^2(F_o) + 0.00024F_o^2]^{-1}$ ; scattering factors from *International Tables for X-ray Crystallography* (1974), both real and imaginary components of anomalous dispersion included for I only (*International Tables for X-ray Crystallography*, 1974); refinement with anisotropic thermal factor for I<sup>-</sup>, isotropic for the others; all H atoms included in their calculated idealized positions (C–H = 1.0 Å) as a fixed contribution; final conventional  $R = 0.066$ ,  $R_w = 0.055$  for 666 observed reflections; calculations carried out on the CYBER 76 with *SHELX76* (Sheldrick, 1976).

**Discussion.** The final positional parameters and isotropic temperature factors are given in Table 1,\* and interatomic distances and interbond angles with e.s.d.'s calculated from the full-variance–covariance matrix are in Table 2.

\* The anisotropic temperature factors for I and lists of structure factors, torsion angles and Figs. 2 and 3 have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38414 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and isotropic temperature factors (Å<sup>2</sup>  $\times 10^3$ ) with e.s.d.'s in parentheses

	x	y	z	$U_{iso}$
C(1)	8505 (19)	–1144 (23)	6462 (37)	72 (7)
C(2)	8971 (12)	456 (12)	4828 (44)	62 (5)
C(3)	8796 (22)	–1289 (25)	3518 (40)	84 (7)
C(4)	7284 (14)	–410 (17)	4376 (24)	62 (5)
C(5)	6717 (16)	367 (16)	5411 (33)	87 (6)
C(6)	5591 (14)	159 (15)	4957 (64)	88 (5)
C(7)	5073 (18)	1196 (19)	5743 (30)	101 (6)
C(8)	3983 (15)	1294 (16)	5217 (50)	112 (6)
C(9)	5729 (14)	2011 (16)	4963 (78)	105 (6)
C(10)	6791 (13)	1593 (12)	5063 (75)	79 (5)
N(1)	8371 (11)	–569 (10)	4826 (41)	62 (4)
I	3321 (1)	3696 (1)	0	80 (1)*

$$* U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

Table 2. Final interatomic distances (Å) and bond angles ( $^\circ$ ) with e.s.d.'s in parentheses

N(1)–C(1)	1.50 (4)	C(5)–C(10)	1.54 (3)
N(1)–C(2)	1.49 (2)	C(6)–C(7)	1.58 (3)
N(1)–C(3)	1.48 (4)	C(7)–C(8)	1.52 (3)
N(1)–C(4)	1.51 (2)	C(7)–C(9)	1.47 (4)
C(4)–C(5)	1.47 (3)	C(9)–C(10)	1.51 (2)
C(5)–C(6)	1.57 (3)		
C(1)–N(1)–C(2)	109 (2)	C(4)–C(5)–C(6)	105 (2)
C(1)–N(1)–C(3)	107 (2)	C(5)–C(6)–C(7)	101 (2)
C(1)–N(1)–C(4)	113 (2)	C(6)–C(7)–C(8)	112 (2)
C(2)–N(1)–C(3)	107 (2)	C(6)–C(7)–C(9)	97 (2)
C(2)–N(1)–C(4)	114 (1)	C(8)–C(7)–C(9)	113 (2)
C(3)–N(1)–C(4)	106 (2)	C(7)–C(9)–C(10)	108 (2)
N(1)–C(4)–C(5)	116 (2)	C(9)–C(10)–C(5)	106 (1)
C(4)–C(5)–C(10)	120 (2)	C(10)–C(5)–C(6)	100 (1)

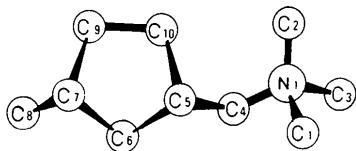


Fig. 1. Perspective view of the cation with the atom-numbering scheme. Selected intramolecular non-bonding distances are: C(1)···C(6) 4.38 (3), N(1)···C(6) 3.82 (2) and N(1)···C(8) 6.30 (2) Å.

A perspective view of the cation, with atom-numbering scheme and selected intramolecular non-bonding distances, is presented in Fig. 1.

The C(*sp*<sup>3</sup>)—C(*sp*<sup>3</sup>) bond lengths range from 1.47 (3) to 1.58 (3) Å and the four C—N<sup>+</sup> distances are in the range 1.48 (4)—1.51 (2) Å.

The most relevant aspect of the structure of the molecule is the orientation of the cyclopentane ring with respect to the ammonium tetrahedral N. Therefore the molecular structure of TMC2 is essentially a one-parameter problem: the N(1)—C(4)—C(5)—C(6) torsion angle. The other torsion angles have threefold symmetry,  $\tau = \pm 60$  or  $180^\circ$ .  $\tau[\text{N}(1)\text{—C}(4)\text{—C}(5)\text{—C}(6)]$  is  $-167^\circ$  in TMC2, indicating that the overall conformation adopted by this cation is very similar to that found in carbon aliphatic chains and is not that

which has been suggested to be necessary for muscarinic activity in acetylcholine and its analogues (Baker, Chothia, Pauling & Petcher, 1971).

The puckering amplitude  $Q$  is 0.52 (4) Å (Cremer & Pople, 1975). In addition there is a pseudo twofold axis through C(10) and C(6)—C(7) (see Fig. 2).<sup>\*</sup> The asymmetry parameter of Duax, Weeks & Rohrer (1976),  $\Delta C_2[\text{C}(10)] = 2 (2)^\circ$ , indicates that the conformation of the ring is 'half-chair'.

The environment of the I<sup>-</sup> ions (Fig. 3)<sup>\*</sup> is approximately tetrahedral with I<sup>-</sup>···N<sup>+</sup> distances ranging from 4.4 to 4.7 Å, with the other packing distances as expected from van der Waals radii.

<sup>\*</sup> See previous footnote.

### References

- BAKER, R. W., CHOTHIA, C. H., PAULING, P. J. & PETCHER, T. J. (1971). *Nature (London)*, **230**, 439–445.  
 CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1367.  
 DUAX, W. L., WEEKS, C. M. & ROHRER, D. C. (1976). *Top. Stereochem.* **9**, 284–286.  
*International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 71–103. Birmingham: Kynoch Press.  
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

*Acta Cryst.* (1983). **C39**, 790–792

## Structure of [2-(Ethylideneaminoxy)ethyl]trimethylammonium Iodide, C<sub>7</sub>H<sub>17</sub>N<sub>2</sub>O<sup>+</sup>·I<sup>-</sup>

BY R. BARDI\* AND A. M. PIAZZESI

*Biopolymer Research Centre, University of Padua, Via Marzolo 1, 35100 Padova, Italy*

AND A. DEL PRA

*Istituto Chimico Farmaceutico, University of Milan, Viale Abruzzi 42, 20131 Milano, Italy*

(Received 24 January 1983; accepted 14 February 1983)

**Abstract.**  $M_r = 272.1$ , monoclinic,  $P2_1/c$ ,  $a = 8.188 (4)$ ,  $b = 13.748 (5)$ ,  $c = 10.174 (5)$  Å,  $\beta = 98.1 (1)^\circ$ ,  $Z = 4$ ,  $U = 1133.8 (9)$  Å<sup>3</sup>,  $D_m = 1.59$ ,  $D_x = 1.594$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 2.85$  mm<sup>-1</sup>,  $F(000) = 536$ ,  $T = 298$  K. Final  $R = 0.047$  for 1420 observed reflections. The conformation of the molecule, described by the torsion angles about the non-rigid bonds, is very similar to that found in many muscarinic agents.

**Introduction.** As a part of our investigation of the crystal structures of molecules affecting cholinergic

transmission systems we have completed the single-crystal X-ray analysis of the structure of the title compound (TMON hereinafter). This compound possesses a very interesting pharmacological activity (Groppetti, Zappia, Pirola & Rossi, 1966) and has been considered in an extensive structure–activity analysis by Pratesi (Pratesi, Villa, Ferri, De Micheli, De Amici, Grana, Santagostino Barbone, Silipo & Vittoria, 1983).

**Experimental.** Single crystals obtained by slow evaporation of a solution in absolute ethanol, under reduced constant pressure in a dry atmosphere,  $D_m$  measured by flotation; approximate unit-cell parameters estimated from preliminary Weissenberg

\* To whom correspondence should be addressed.