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Structure of Org 32763: 3-Methoxyimino-quinuclidinium Chloride

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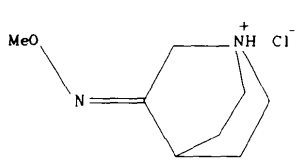
Abstract

The quinuclidine fragment of $C_8H_{15}N_2O^+.Cl^-$ has mirror symmetry with the greater part of the atoms lying in the mirror plane. The three six-membered rings of the cage all approach ideal boat conformations. The extended conformation of the side chain is such that the N—O bond is in the *anti* position with respect to the C1—C5 bond, where C5 is a bridgehead atom. The protonated N atom of the cage donates a hydrogen bond to the Cl^- anion; the three C atoms bonded to the protonated N atom are involved in short intermolecular C—H···Cl^- contacts.

Comment

Org 32763 (I) is a muscarinic agonist that might be of practical value in cholinergic replacement therapy for Alzheimer's disease. The compound was characterized by NMR and IR methods, but required a definite structure determination to show most structural details of interest. The title compound (I) has mirror symmetry. The mirror plane at $z = \frac{3}{4}$ which generates the molecule, contains the greater part of the atoms: all non-H atoms except C3 and C4, and three H atoms. The quinuclidine cage consists of three six-membered rings which all approach ideal boat conformations, despite the fact that two of the rings share an sp^2-hybridized C atom. The conformation about the C=N bond is such that the N—O bond is in the *anti* position with respect to the C1—C5 bond where C5 is the bridgehead C atom.



(I)

The protonated N atom of the cage donates a hydrogen bond to the Cl^- anion at $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} + z$, with N···$Cl^-$ 2.991 (2) Å and N—H···Cl^- 169 (2)°. There are two short intermolecular C—H···Cl^- contacts, C2—H···Cl^- ($-\frac{1}{2} + x, \frac{1}{2} - y, \frac{3}{2} - z$) and C3—H···$Cl^-$ ($-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} - z$), with H···$Cl^-$ distances of 2.82 (2) and 2.74 (2) Å, respectively. Thus, as a result of the mirror symmetry, the quinuclidine residue has a total of four C—H···Cl^- contacts. It is noteworthy that these contacts all involve C atoms bonded to the protonated quinuclidine N atom.

The bond distances and angles are in the normal range. The two C_{sp^2}—C_{sp^3} bonds are short [1.497 (3) Å] and the angles around C_{sp^2} are 113.5 (2)° (endocyclic) and 120.4 (2) and 126.2 (2)° (exocyclic), the larger angle being related to the synperiplanar conformation. The remaining angles of the cage are close to tetrahedral.

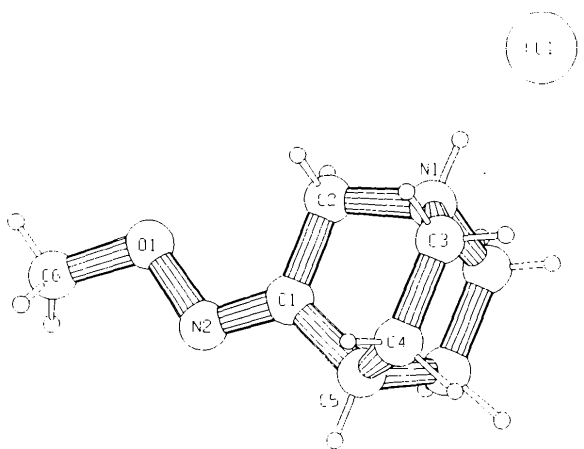


Fig. 1. Perspective view and atomic numbering of the title compound.

Experimental

Crystal data

$C_8H_{15}N_2O^+.Cl^-$
M_r = 190.67
Orthorhombic
Pnam
a = 12.8909 (7) Å
b = 11.1618 (6) Å
c = 6.8110 (3) Å
V = 980.01 (9) Å^3
Z = 4
D_x = 1.292 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å

Cell parameters from 25 reflections
θ = 14.09–18.03°
μ = 0.346 mm^{-1}
T = 295 K
Block-shaped
0.85 × 0.50 × 0.50 mm
Colourless
Crystal source: Organon International BV, Oss, The Netherlands

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none

R_{int} = 0.036
θ_{max} = 30.26°
h = −18 → 18
k = −15 → 15
l = 0 → 9

6603 measured reflections
1579 independent reflections
1310 observed reflections
$[I > 2.5\sigma(I)]$
3 standard reflections
frequency: 60 min
intensity variation: 2.1%

Refinement

Refinement on F
$R = 0.0386$
$wR = 0.0395$
$S = 0.43$
1310 reflections
92 parameters
All H-atom parameters refined
Calculated weights
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.086$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	U_{eq}
O1	0.0128 (1)	0.4348 (1)	$\frac{3}{4}$	0.0418 (4)
N1	0.1375 (1)	0.0982 (2)	$\frac{3}{4}$	0.0401 (5)
N2	0.1218 (1)	0.4234 (2)	$\frac{3}{4}$	0.0424 (5)
C1	0.1466 (2)	0.3132 (2)	$\frac{3}{4}$	0.0369 (6)
C2	0.0731 (2)	0.2094 (2)	$\frac{3}{4}$	0.0360 (5)
C3	0.2037 (1)	0.0937 (2)	0.5703 (3)	0.0539 (6)
C4	0.2775 (2)	0.1999 (2)	0.5697 (3)	0.0663 (6)
C5	0.2584 (2)	0.2767 (2)	$\frac{1}{4}$	0.0527 (8)
C6	−0.0133 (2)	0.5597 (2)	$\frac{3}{4}$	0.0463 (6)
Cl1	0.47766 (5)	0.36551 (5)	$\frac{1}{4}$	0.0509 (2)

Table 2. *Geometric parameters* (Å, °)

O1—N2	1.411 (2)	C1—C2	1.497 (3)
O1—C6	1.434 (3)	C1—C5	1.498 (4)
N1—C2	1.493 (3)	C3—C4	1.520 (3)
N1—C3	1.493 (2)	C4—C5	1.518 (3)
N2—C1	1.271 (3)		
N2—O1—C6	108.7 (2)	C2—C1—C5	113.5 (2)
C2—N1—C3	110.2 (1)	N1—C2—C1	106.9 (2)
C2—N1—C3^i	110.2 (1)	N1—C3—C4	109.5 (2)
C3—N1—C3^i	110.1 (1)	C3—C4—C5	109.7 (2)
O1—N2—C1	109.7 (2)	C1—C5—C4	108.0 (2)
N2—C1—C2	126.2 (2)	C1—C5—C4^i	108.0 (2)
N2—C1—C5	120.4 (2)	C4—C5—C4^i	108.0 (2)

Symmetry code: (i) $x, y, \frac{3}{2} - z$.

The systematic extinctions ($h0l$, h odd; $0kl$, $k + l$ odd) are consistent with space groups $Pna2_1$ and *Pnam* (alternative setting of *Pnma*, No. 62). The structure was solved in space group $Pna2_1$, which resulted in a model having mirror symmetry. Therefore, the analysis was continued in space group *Pnam* [general positions: $\pm(x, y, z; \frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} + z; x, y, \frac{1}{2} - z; \frac{1}{2} - x, \frac{1}{2} + y, -z)$], which, in contrast to $Pna2_1$, yielded normal non-deviating geometries. The positions of the H atoms were located from $\Delta\rho$ syntheses and included in the refinement with an overall isotropic thermal parameter, which refined to 0.074 (2) Å^2. Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *SET4* (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1990*b*). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *EUCLID* (Spek, 1982). Software used to prepare material for publication: *PLATON* (Spek, 1990*a*).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71500 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1095]

References

Boer, J. L. de & Duisenberg, A. J. M. (1984). *Acta Cryst.* A**40**, C-410.
Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467-473.
Spek, A. L. (1982). *The EUCLID Package. Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.
Spek, A. L. (1990*a*). *Acta Cryst.* A**46**, C-34.
Spek, A. L. (1990*b*). *HELENA. Program for Data Reduction*. Laboratorium voor Kristal- en Structuurchemie, Univ. of Utrecht, The Netherlands.

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2,7-Dimethyl-3,5-octadiyne-2,7-diol Dichloromethane Solvate: a Clathrate Comprising Hydrogen-Bonded Supramolecular Tunnels Containing Dichloromethane Guest Molecules

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Abstract

The title molecule, $C_{10}H_{14}O_2.0.06CH_2Cl_2$, forms an unusual tubular structure in which double-walled channels of approximately 5 Å internal diameter extend throughout the crystal. Groups of three molecules are positioned around a threefold axis to form a section of the tube. These molecules are configured so that methyl and acetylene groups line the tube and hydroxyl groups form the outer wall. As each hydroxyl group is near to a threefold screw axis, the channel walls are reinforced by infinite hydrogen-bonded columns parallel to c [O···O 2.68 (1), 2.771 (9) Å]. Although dichloromethane fits well in the channels, the final guest-to-host ratio of 0.06:1 indicates