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Structure of N^1-(3-Phenoxypropyl)-4,7-diaza-1-azoniatricyclo[5.2.1.$0^{4,10}$]decane Bromide Hydrate

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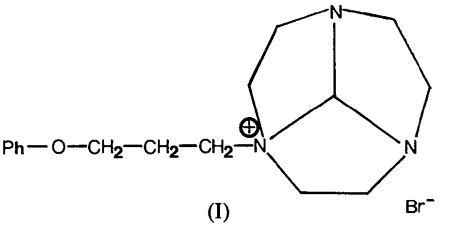
Abstract

The structure of the title compound shows a distortion towards the amidinium form with the bond between the capping C atom and the quaternary N atom [1.668 (9) Å] being significantly longer than the distances between this C atom and the other two N atoms [1.419 (9) and 1.398 (9) Å].

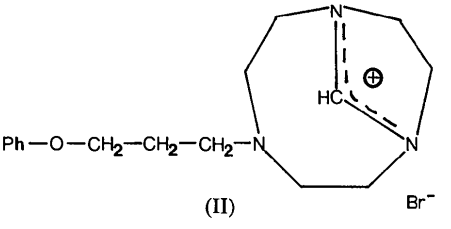
Comment

The structure of the title compound was determined as it is an intermediate in the general preparative route to mono-*N*-substituted cyclic triazanonanes. It was prepared in near quantitative yield by allowing equimolar amounts of 1-bromo-3-phenoxypropane and 1,4,7-triazatricyclo[5.2.1.$0^{4,10}$]decane to react in tetrahydrofuran at room temperature in the dark (Atkins, 1980). The preparation of a related *N*-CH_2Ph derivative has been reported (Weisman, Vachon, Johnson & Gronbeck, 1987). The most interesting feature of the structure concerns the bonding of the 'capping' C atom, C(1). Formally, the N(1) atom is a quaternary nitrogen as in (I), bearing



(I)

a positive charge, and has C—N—C angles in the range 102.3 (6)–116.1 (6)°. However, the structure shows considerable distortion towards the amidinium form, (II); the C(1)—N(1) distance



(II)

[1.668 (9) Å] is long for a C—N single bond and is significantly longer than C(1)—N(4) [1.419 (9) Å] and C(1)—N(7) [1.398 (9) Å]. These in turn are significantly shorter than the other C—N bond distances. The structure also contains water of solvation [O(2)] at less than full occupancy; least-squares refinement afforded a site occupancy factor of 0.72 (2).

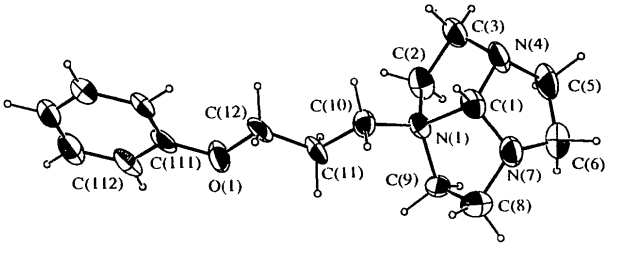


Fig. 1. Molecular structure and atomic labelling scheme for the cation. Thermal ellipsoids are shown at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

Experimental

Crystal data

$C_{16}H_{24}N_3O^+.Br^-.0.72H_2O$
M_r = 367.25
Monoclinic
$P2_1/a$
a = 8.431 (1) Å
b = 20.452 (3) Å
c = 10.615 (3) Å
β = 111.42 (1)°
D_x = 1.38 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 17–20°
μ = 2.389 mm^{-1}
T = 298 K
Cleaved from large prism

V = 1704.5 (6) Å^3
Z = 4
0.5 × 0.3 × 0.2 mm
Colourless

Data collection

Enraf–Nonius Turbo CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (Walker & Stuart, 1983)
T_{min} = 0.72, T_{max} = 1.41
2842 measured reflections
2553 independent reflections
1227 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.031
θ_{max} = 25°
$h = -9 \rightarrow 0$
$k = 0 \rightarrow 23$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 120 min
intensity variation: 2.8%

Refinement

Refinement on F
R = 0.048
wR = 0.041
S = 1.33
1227 reflections
190 parameters
Only H-atom U's refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984) and the *GX Crystallographic Program System* (Mallinson & Muir, 1985).

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}
Br	0.18821 (11)	0.10429 (4)	−0.08481 (9)	0.054
O(1)	0.1833 (6)	0.5310 (2)	0.6270 (5)	0.049
O(2)	0.4307 (12)	0.4178 (7)	0.5896 (9)	0.151
N(1)	0.2467 (7)	0.3892 (3)	1.0110 (6)	0.033
N(4)	0.2554 (8)	0.3791 (3)	1.2441 (7)	0.046
N(7)	0.1304 (8)	0.2956 (3)	1.0953 (7)	0.043
C(1)	0.1455 (10)	0.3634 (4)	1.1106 (9)	0.040
C(2)	0.4011 (9)	0.4229 (4)	1.1063 (8)	0.044
C(3)	0.3514 (10)	0.4360 (4)	1.2310 (8)	0.051
C(5)	0.3595 (11)	0.3207 (5)	1.3017 (9)	0.062
C(6)	0.2673 (12)	0.2657 (4)	1.2098 (9)	0.064
C(8)	0.1363 (12)	0.2822 (4)	0.9617 (9)	0.063
C(9)	0.2779 (11)	0.3264 (4)	0.9502 (8)	0.048
C(10)	0.1338 (8)	0.4360 (3)	0.9072 (8)	0.038
C(11)	0.2014 (9)	0.4551 (4)	0.7985 (7)	0.039
C(12)	0.1129 (10)	0.5161 (4)	0.7278 (8)	0.043
C(111)	0.1313 (8)	0.5878 (3)	0.5546 (8)	0.040
C(112)	0.2168 (10)	0.6043 (4)	0.4687 (10)	0.048
C(113)	0.1748 (7)	0.6616 (3)	0.3925 (6)	0.056
C(114)	0.0473 (7)	0.7025 (3)	0.4023 (7)	0.058
C(115)	−0.0381 (9)	0.6859 (3)	0.4883 (9)	0.054
C(116)	0.0039 (7)	0.6286 (2)	0.5644 (5)	0.044

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

O(1)—C(12)	1.433 (8)	O(1)—C(111)	1.374 (5)
N(1)—C(1)	1.668 (9)	N(1)—C(2)	1.495 (9)
N(1)—C(9)	1.503 (9)	N(1)—C(10)	1.507 (9)
N(4)—C(1)	1.419 (9)	N(4)—C(3)	1.452 (9)
N(4)—C(5)	1.477 (10)	N(7)—C(1)	1.398 (9)
N(7)—C(6)	1.469 (10)	N(7)—C(8)	1.463 (10)
C(2)—C(3)	1.550 (11)	C(5)—C(6)	1.505 (11)
C(8)—C(9)	1.537 (11)	C(10)—C(11)	1.513 (10)
C(11)—C(12)	1.508 (10)		
C(12)—O(1)—C(111)	117.5 (5)	C(1)—N(1)—C(2)	103.8 (6)
C(1)—N(1)—C(9)	102.3 (6)	C(1)—N(1)—C(10)	109.3 (5)
C(2)—N(1)—C(9)	116.1 (6)	C(2)—N(1)—C(10)	111.3 (6)
C(9)—N(1)—C(10)	113.1 (6)	C(1)—N(4)—C(3)	105.6 (7)
C(1)—N(4)—C(5)	107.7 (7)	C(3)—N(4)—C(5)	114.8 (7)
C(1)—N(7)—C(6)	107.8 (7)	C(1)—N(7)—C(8)	105.3 (7)
C(6)—N(7)—C(8)	115.5 (7)	N(1)—C(1)—N(4)	105.2 (6)
N(1)—C(1)—N(7)	106.6 (6)	N(4)—C(1)—N(7)	109.8 (7)
N(1)—C(2)—C(3)	102.5 (6)	N(4)—C(3)—C(2)	106.2 (7)
N(4)—C(5)—C(6)	104.3 (7)	N(7)—C(6)—C(5)	106.8 (7)
N(7)—C(8)—C(9)	105.6 (7)	N(1)—C(9)—C(8)	101.9 (6)
N(1)—C(10)—C(11)	114.1 (6)	C(10)—C(11)—C(12)	109.7 (7)
O(1)—C(12)—C(11)	106.4 (7)	O(1)—C(111)—C(112)	115.8 (3)
O(1)—C(111)—C(116)	124.2 (3)		

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 71368 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1050]

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2-(4-Chlorophenylazo)-2-methyl-1,3-indandione

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Abstract

In the title compound, $C_{16}H_{11}ClN_2O_2$, the 2-methyl-1,3-dioxoindan-2-yl and 4-chlorophenyl groups are *trans* to each other. The five-membered ring of the