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4-(2-Chloroethyl)morpholinium chloride

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Key indicators

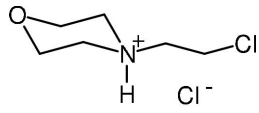
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.028
wR factor = 0.078
Data-to-parameter ratio = 18.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, $C_6H_{13}ClNO^+\cdot Cl^-$, comprises a cation with the morpholine ring in the chair conformation, and a single hydrogen-bonding association between the morpholinium NH group and the Cl^- anion.

Comment

The title compound, (I), is used as an intermediate for the synthesis of the antispasmodic drug pinaverium bromide, and is also used as an intermediate for the synthesis of biologically active heterocycles (Baronnet *et al.*, 1974). A search of the Cambridge Structural Database (Version 5.26; Allen, 2002) reveals that there are 90 known structures that contain the morpholinium cation. Of these there are 24 that have an *N*-ethyl chain, or longer, including the structure of 4-(2-fluoroethyl)morpholinium chloride (Briggs *et al.*, 2004). This compound crystallizes in monoclinic space group $P2_1/n$, with the morpholine ring in the chair conformation and a single hydrogen-bonding association between the morpholinium NH group and the Cl^- anion (N···Cl = 3.036 Å).



(I)

The structure of the title compound comprises a cation with the morpholine ring also in the chair conformation (Fig. 1), and a single hydrogen-bonding association similarly between

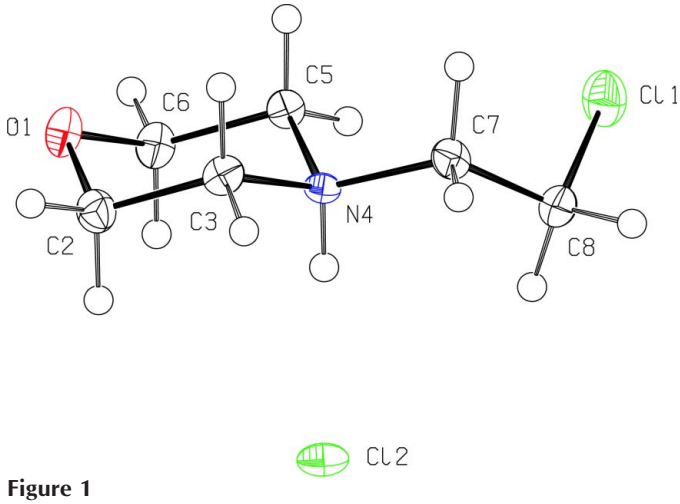


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius.

the morpholinium NH group and the Cl^- anion (Table 1). Three torsion angles that define the conformation of the chloroethyl chain are C2—C3—N4—C7 [−177.50 (12)°], C3—N4—C7—C8 [162.71 (13)°] and N4—C7—C8—Cl1 [86.66 (15)°]. The equivalent angles in the fluoro analogue are 178.46, −78.85 and −173.68°, respectively.

Experimental

An equimolar mixture of morpholine (0.87 g, 10 mmol), anhydrous K_2CO_3 (1.38 g, 10 mmol) and 1-bromo-2-chloroethane (1.43 g, 10 mmol) was stirred at room temperature in dimethylformamide (10 ml) for 6 h. The collected product was subsequently converted to the hydrochloride salt using isopropyl alcohol and HCl (80:20). Crystals of compound (I) were grown from methanol.

Crystal data

$C_6H_{13}ClNO^+\cdot Cl^-$
M_r = 186.07
Triclinic, $P\overline{1}$
a = 6.9876 (3) Å
b = 8.1549 (4) Å
c = 8.6495 (3) Å
α = 63.530 (2)°
β = 85.004 (3)°
γ = 85.179 (2)°
V = 438.97 (3) Å^3
Z = 2
D_x = 1.408 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1914 reflections
θ = 2.9–27.5°
μ = 0.68 mm^{-1}
T = 120 (2) K
Plate, colourless
0.28 × 0.24 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.833, T_{max} = 0.961
7581 measured reflections
1716 independent reflections
1494 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 26.0°
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 9$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.078
S = 0.94
1716 reflections
94 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.2143P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···Cl2	0.883 (19)	2.16 (2)	3.0435 (14)	178 (2)

The H atom attached to the N atom was located in a difference Fourier synthesis and its positional parameters were refined. Other H atoms were included in the refinement at calculated positions, in the riding-model approximation, with a C—H distance of 0.99 Å. The isotropic displacement parameters for all H atoms were set equal to 1.25U_{eq} of the carrier atom.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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