

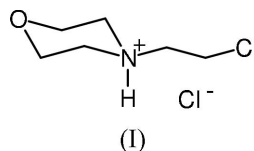
## 4-(2-Chloroethyl)morpholinium chloride

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## Key indicators

Single-crystal X-ray study  
 $T = 120$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.028  
 $wR$  factor = 0.078  
Data-to-parameter ratio = 18.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The structure of the title compound,  $\text{C}_6\text{H}_{13}\text{ClNO}^+\cdot\text{Cl}^-$ , comprises a cation with the morpholine ring in the chair conformation, and a single hydrogen-bonding association between the morpholinium NH group and the  $\text{Cl}^-$  anion.Received 22 February 2005  
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## 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  $\text{Cl}^-$  anion ( $\text{N}\cdots\text{Cl} = 3.036$  Å).

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

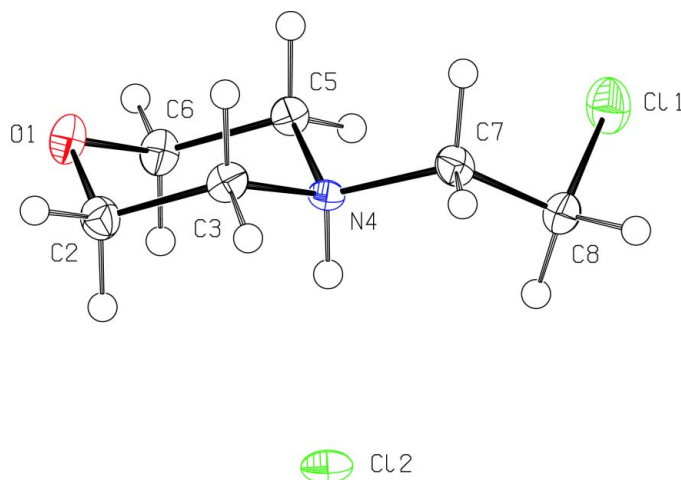


Figure 1

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<sup>-</sup> 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<sub>2</sub>CO<sub>3</sub> (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 <sub>6</sub> H <sub>13</sub> ClNO <sup>+</sup> ·Cl <sup>-</sup>	Z = 2
M <sub>r</sub> = 186.07	D <sub>x</sub> = 1.408 Mg m <sup>-3</sup>
Triclinic, P1̄	Mo Kα radiation
a = 6.9876 (3) Å	Cell parameters from 1914 reflections
b = 8.1549 (4) Å	θ = 2.9–27.5°
c = 8.6495 (3) Å	μ = 0.68 mm <sup>-1</sup>
α = 63.530 (2)°	T = 120 (2) K
β = 85.004 (3)°	Plate, colourless
γ = 85.179 (2)°	0.28 × 0.24 × 0.06 mm
V = 438.97 (3) Å <sup>3</sup>	

#### Data collection

Nonius KappaCCD diffractometer	1494 reflections with I > 2σ(I)
φ and ω scans	R <sub>int</sub> = 0.032
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	θ <sub>max</sub> = 26.0°
T <sub>min</sub> = 0.833, T <sub>max</sub> = 0.961	h = –8 → 8
7581 measured reflections	k = –10 → 9
1716 independent reflections	l = –10 → 10

#### Refinement

Refinement on F <sup>2</sup>	w = 1/[σ <sup>2</sup> (F <sub>o</sub> <sup>2</sup> ) + (0.0469P) <sup>2</sup> + 0.2143P]
R[F <sup>2</sup> > 2σ(F <sup>2</sup> )] = 0.028	where P = (F <sub>o</sub> <sup>2</sup> + 2F <sub>c</sub> <sup>2</sup> )/3
wR(F <sup>2</sup> ) = 0.078	(Δ/σ) <sub>max</sub> < 0.001
S = 0.94	Δρ <sub>max</sub> = 0.20 e Å <sup>-3</sup>
1716 reflections	Δρ <sub>min</sub> = –0.28 e Å <sup>-3</sup>
94 parameters	
H atoms treated by a mixture of independent and constrained refinement	

**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<sub>eq</sub> 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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## supporting information

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## 4-(2-Chloroethyl)morpholinium chloride

*Crystal data*

$C_6H_{13}ClNO^+Cl^-$

$M_r = 186.07$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 6.9876$  (3) Å

$b = 8.1549$  (4) Å

$c = 8.6495$  (3) Å

$\alpha = 63.530$  (2)°

$\beta = 85.004$  (3)°

$\gamma = 85.179$  (2)°

$V = 438.97$  (3) Å<sup>3</sup>

$Z = 2$

$F(000) = 196$

$D_x = 1.408$  Mg m<sup>-3</sup>

Melting point: 458 K

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1914 reflections

$\theta = 2.9$ – $27.5$ °

$\mu = 0.68$  mm<sup>-1</sup>

$T = 120$  K

Plate, colourless

$0.28 \times 0.24 \times 0.06$  mm

*Data collection*

Nonius KappaCCD  
diffractometer

Radiation source: Bruker Nonius FR591  
rotating anode

10 cm confocal mirrors monochromator

Detector resolution: 9.091 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  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  $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 26.0$ °,  $\theta_{\min} = 3.8$ °

$h = -8 \rightarrow 8$

$k = -10 \rightarrow 9$

$l = -10 \rightarrow 10$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.078$

$S = 0.94$

1716 reflections

94 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

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<sup>-3</sup>

$\Delta\rho_{\min} = -0.28$  e Å<sup>-3</sup>

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.31450 (17)	-0.10081 (15)	0.17258 (14)	0.0211 (3)
C2	0.2513 (2)	0.0634 (2)	0.0296 (2)	0.0206 (4)
H21	0.3005	0.0597	-0.0797	0.026*
H22	0.1090	0.0712	0.0319	0.026*
C3	0.3210 (2)	0.2316 (2)	0.0350 (2)	0.0171 (3)
H31	0.2755	0.3437	-0.0652	0.021*
H32	0.4634	0.2269	0.0287	0.021*
N4	0.24469 (19)	0.23719 (18)	0.20020 (17)	0.0133 (3)
H4	0.118 (3)	0.243 (2)	0.203 (2)	0.017*
C5	0.3049 (2)	0.0621 (2)	0.3499 (2)	0.0159 (3)
H51	0.4467	0.0529	0.3546	0.020*
H52	0.2479	0.0611	0.4592	0.020*
C6	0.2391 (2)	-0.0999 (2)	0.3312 (2)	0.0193 (4)
H61	0.0968	-0.0946	0.3346	0.024*
H62	0.2820	-0.2151	0.4295	0.024*
C7	0.3033 (2)	0.4064 (2)	0.2071 (2)	0.0176 (3)
H71	0.4406	0.3899	0.2337	0.022*
H72	0.2906	0.5116	0.0917	0.022*
C8	0.1861 (3)	0.4513 (2)	0.3400 (2)	0.0222 (4)
H81	0.1832	0.5855	0.3010	0.028*
H82	0.0522	0.4168	0.3460	0.028*
C11	0.27566 (7)	0.33778 (6)	0.55327 (6)	0.02923 (15)
C12	-0.19260 (5)	0.24738 (5)	0.21272 (5)	0.02018 (14)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0271 (7)	0.0174 (6)	0.0196 (6)	-0.0017 (5)	0.0038 (5)	-0.0097 (5)
C2	0.0225 (9)	0.0226 (9)	0.0188 (8)	-0.0015 (7)	-0.0008 (7)	-0.0110 (7)
C3	0.0183 (8)	0.0189 (8)	0.0115 (7)	-0.0003 (6)	0.0018 (6)	-0.0048 (6)
N4	0.0105 (6)	0.0147 (7)	0.0139 (6)	-0.0015 (5)	-0.0008 (5)	-0.0054 (5)
C5	0.0188 (8)	0.0135 (8)	0.0130 (8)	-0.0014 (6)	-0.0011 (6)	-0.0036 (6)
C6	0.0227 (9)	0.0157 (8)	0.0179 (8)	-0.0043 (7)	0.0039 (7)	-0.0063 (7)
C7	0.0195 (8)	0.0124 (8)	0.0185 (8)	-0.0042 (6)	-0.0010 (6)	-0.0042 (7)
C8	0.0217 (9)	0.0188 (9)	0.0291 (9)	0.0020 (7)	-0.0040 (7)	-0.0133 (8)
C11	0.0381 (3)	0.0306 (3)	0.0224 (2)	-0.0013 (2)	-0.00025 (19)	-0.0151 (2)
C12	0.0122 (2)	0.0228 (2)	0.0189 (2)	-0.00099 (16)	-0.00073 (15)	-0.00332 (18)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N4—C5	1.496 (2)	C6—C5	1.513 (2)
N4—C7	1.4995 (19)	C6—H61	0.99
N4—C3	1.4994 (19)	C6—H62	0.99
N4—H4	0.883 (19)	C5—H51	0.99
C2—C3	1.515 (2)	C5—H52	0.99

C3—H31	0.99	C7—C8	1.512 (2)
C3—H32	0.99	C7—H71	0.99
C2—O1	1.427 (2)	C7—H72	0.99
C2—H21	0.99	C8—C11	1.7980 (18)
C2—H22	0.99	C8—H81	0.99
O1—C6	1.4289 (19)	C8—H82	0.99
C5—N4—C7	114.05 (12)	O1—C6—H62	109.4
C5—N4—C3	109.14 (12)	C5—C6—H62	109.4
C7—N4—C3	110.77 (12)	H61—C6—H62	108.0
C5—N4—H4	107.2 (12)	N4—C5—C6	110.00 (13)
C7—N4—H4	106.9 (11)	N4—C5—H51	109.7
C3—N4—H4	108.6 (11)	C6—C5—H51	109.7
N4—C3—C2	109.09 (13)	N4—C5—H52	109.7
N4—C3—H31	109.9	C6—C5—H52	109.7
C2—C3—H31	109.9	H51—C5—H52	108.2
N4—C3—H32	109.9	N4—C7—C8	113.63 (13)
C2—C3—H32	109.9	N4—C7—H71	108.8
H31—C3—H32	108.3	C8—C7—H71	108.8
O1—C2—C3	111.28 (13)	N4—C7—H72	108.8
O1—C2—H21	109.4	C8—C7—H72	108.8
C3—C2—H21	109.4	H71—C7—H72	107.7
O1—C2—H22	109.4	C7—C8—C11	114.08 (12)
C3—C2—H22	109.4	C7—C8—H81	108.7
H21—C2—H22	108.0	C11—C8—H81	108.7
C2—O1—C6	109.92 (12)	C7—C8—H82	108.7
O1—C6—C5	111.36 (13)	C11—C8—H82	108.7
O1—C6—H61	109.4	H81—C8—H82	107.6
C5—C6—H61	109.4		
C5—N4—C3—C2	56.13 (16)	C3—N4—C5—C6	-55.60 (16)
C7—N4—C3—C2	-177.50 (12)	O1—C6—C5—N4	57.83 (17)
N4—C3—C2—O1	-59.50 (17)	C5—N4—C7—C8	-73.70 (17)
C3—C2—O1—C6	60.97 (17)	C3—N4—C7—C8	162.71 (13)
C2—O1—C6—C5	-59.89 (17)	N4—C7—C8—C11	86.66 (15)
C7—N4—C5—C6	179.93 (13)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N4—H4 $\cdots$ C12	0.883 (19)	2.16 (2)	3.0435 (14)	178 (2)