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4-Benzylmorpholin-4-ium chloride

Dana Copolovici,* Cristian Silvestru and Richard A. Varga

Faculty of Chemistry and Chemical Engineering, "Babes-Bolyai" University, Arany Janos Str. No. 11, RO-400028, Cluj-Napoca, Romania
Correspondence e-mail: mban@chem.ubbcluj.ro

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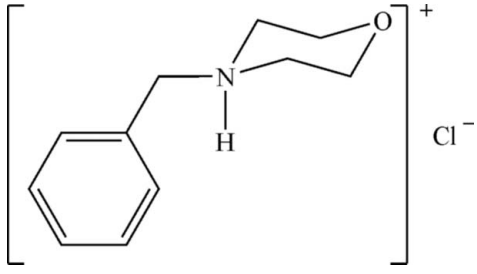
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.004 Å; R factor = 0.048; wR factor = 0.109; data-to-parameter ratio = 17.7.

Ions of the title compound, $C_{11}H_{16}NO^{+}\cdot Cl^{-}$, are linked by an N—H$\cdots$Cl^{-} and four C—H$\cdots$Cl^{-} interactions, generating a two-dimensional layer with no interactions between different morpholinium groups; the morpholinium group adopts an approximately ideal chair conformation.

Related literature

Bernstein *et al.* (1995); Bruno *et al.* (2006); Calas *et al.* (1997); Cao & Hu (2006); Dega-Szafran, Szafran & Katrusiak (2004); Stone *et al.* (1958).



Experimental

Crystal data

$C_{11}H_{16}NO^{+}\cdot Cl^{-}$
M_r = 213.70
Monoclinic, $P2_1/n$
a = 7.0181 (6) Å
b = 9.3458 (9) Å
c = 17.3003 (16) Å
β = 90.958 (2)°
V = 1134.56 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 297 (2) K
0.49 × 0.34 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.865, T_{max} = 0.947
11765 measured reflections
2321 independent reflections
2180 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.109
S = 1.17
2321 reflections
131 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$Cl1	0.88 (2)	2.19 (2)	3.062 (2)	172 (2)
C7—H7$B\cdots$Cl1^{i}	0.97	2.86	3.725 (2)	149
C8—H8$A\cdots$Cl1^{i}	0.97	2.80	3.690 (2)	153
C8—H8$B\cdots$Cl1^{ii}	0.97	2.87	3.576 (2)	130
C11—H11$A\cdots$Cl1^{iii}	0.97	2.82	3.731 (2)	157

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL* (Bruker, 2001); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* 3 (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2007).

Financial support from the National University Research Council (CNCSIS, Romania; Research Project No. A-709/2006 and TD-93/2006) is greatly appreciated. We also thank the National Center for X-Ray Diffraction from Cluj-Napoca for support in the solid-state structure determination and Ph.D. Student Vilma Bojan for the NMR investigations.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG3089).

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supplementary materials

Acta Cryst. (2007). E**63**, o2465 [**doi:10.1107/S1600536807017060**]

4-Benzylmorpholin-4-ium chloride

D. Copolovici, C. Silvestru and R. A. Varga

Comment

Many morpholine derivatives and their salts show bacteriostatic activity and have potential as drugs (Dega-Szafran *et al.*, 2004; Calas *et al.*, 1997). The atomic numbering scheme and the conformation of 4-benzylmorpholin-4-ium chloride (I) are depicted in Fig. 1. The morpholium N—H forms an interaction with the Cl^- ion (Table 1) with a H1···Cl1 distance similar to the N—H···Cl interaction in $C_8H_8NO_4^+.Cl^-.H_2O$ (2.3 Å) (Bruno *et al.*, 2006) and $C_{12}H_{15}Cl_2FNO_2^+.Cl^-$ (2.21 Å) (Cao & Hu, 2006). The Cl^- ion is also involved in four other intermolecular contacts with three molecules (details in Table 1). These C—H···Cl interactions lead to the formation of a 2-D network which contains units with graph-set motifs $R^2_4(10)$ and $R^1_2(6)$ (Bernstein *et al.*, 1995). There are no hydrogen-bonding interactions between different morpholinium groups.

Experimental

To a solution of 4-benzylmorpholine (83 mg, 0.46 mmol) in methanol (10 ml) was added HCl (0.06 ml, 12 M) and the reaction mixture was stirred at room temperature for 1 h. Colourless crystals suitable for X-ray diffraction were grown by slow evaporation of the solution. Yield: 84 mg (90%). M.p.: 244-246°C [243-244 °C (Stone *et al.*, 1958)]. Compound (I) was also obtained during the work up of $[2\text{-}\{O(CH_2CH_2)_2NCH_2\}C_6H_4]_2SbCl$. Spectroscopic analysis: ^{1}H NMR (D_2O, 300 MHz): δ 3.16 (m, 2H, CH_2), 3.36 (m, 2H, CH_2), 3.69 (m, 2H, CH_2), 4.01 (m, 2H, CH_2), 4.28 (s, 2H, CH_2, $C_6H_5CH_2$), 7.45 (m, 5H, C_6H_5). ^{13}C NMR (D_2O, 75.46 MHz): δ 51.13 (s, N—CH_2), 60.72 (s, $C_6H_5CH_2$), 63.56 (s, O—CH_2), 127.77 (s, C_6H_5, C_p), 129.24 (s, C_6H_5, C_m), 130.28 (s, C_6H_5, C_o), 131.22 (s, C_6H_5, C_i).

Refinement

All C-bound H atoms were placed in calculated positions (C—H = 0.93-0.97 Å) and treated using a riding model with U_{iso}= 1.2U_{eq}(C) for aryl H atoms. The hydrogen H1 atom bonded to N1 atom was calculated and fixed at a standard N—H distance of 0.88 (2) Å.

Figures

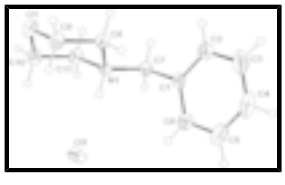

Fig. 1. : A view of compound (I) showing the atom-numbering scheme at 30% probability displacement ellipsoids.

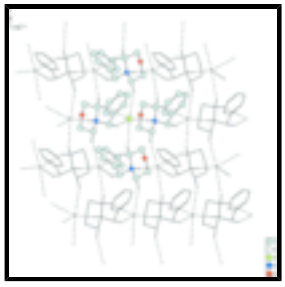

Fig. 2. : A view of the two-dimensional supramolecular motif showing the hydrogen bonds (indicated as dotted lines).

4-Benzylmorpholin-4-ium chloride

Crystal data

$C_{11}H_{16}NO^{+}\cdot Cl^{-}$	F_{000} = 456
M_r = 213.70	D_x = 1.251 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 518 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.0181 (6) Å	Cell parameters from 4130 reflections
b = 9.3458 (9) Å	θ = 2.4–27.9º
c = 17.3003 (16) Å	μ = 0.31 mm^{-1}
β = 90.958 (2)º	T = 297 (2) K
V = 1134.56 (18) Å^3	Block, colourless
Z = 4	0.49 × 0.34 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2321 independent reflections
Radiation source: fine-focus sealed tube	2180 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.036
T = 297(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	h = −8→8
T_{min} = 0.865, T_{max} = 0.947	k = −11→11
11765 measured reflections	l = −21→21

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 0.5699P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.048	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.109	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
S = 1.17	$\Delta\rho_{min}$ = −0.21 e Å^{-3}

2321 reflections
131 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4842 (3)	0.5884 (2)	0.61068 (11)	0.0375 (4)
C2	0.3416 (4)	0.6760 (3)	0.58221 (13)	0.0550 (6)
H2	0.2207	0.6723	0.6033	0.066*
C3	0.3779 (5)	0.7696 (3)	0.52235 (15)	0.0745 (8)
H3	0.2813	0.8286	0.5034	0.089*
C4	0.5550 (5)	0.7758 (3)	0.49090 (14)	0.0714 (8)
H4	0.5789	0.8393	0.4508	0.086*
C5	0.6962 (4)	0.6889 (3)	0.51822 (14)	0.0624 (7)
H5	0.8164	0.6928	0.4965	0.075*
C6	0.6620 (3)	0.5954 (3)	0.57785 (12)	0.0481 (5)
H6	0.7595	0.5364	0.5962	0.058*
C7	0.4460 (3)	0.4873 (2)	0.67572 (11)	0.0380 (4)
H7A	0.4997	0.3946	0.6635	0.046*
H7B	0.3094	0.4755	0.6805	0.046*
C8	0.4605 (3)	0.68275 (19)	0.77400 (11)	0.0344 (4)
H8A	0.3224	0.6837	0.7756	0.041*
H8B	0.5003	0.7520	0.7358	0.041*
C9	0.5421 (3)	0.7227 (2)	0.85218 (11)	0.0427 (5)
H9A	0.6798	0.7282	0.8493	0.051*
H9B	0.4952	0.8164	0.8667	0.051*
C10	0.5645 (3)	0.4847 (2)	0.89033 (12)	0.0462 (5)
H10A	0.5334	0.4170	0.9307	0.055*
H10B	0.7022	0.4896	0.8871	0.055*
C11	0.4822 (3)	0.4336 (2)	0.81445 (11)	0.0404 (5)
H11A	0.5345	0.3405	0.8021	0.049*
H11B	0.3450	0.4239	0.8183	0.049*

Cl1	0.96354 (7)	0.55740 (6)	0.75391 (3)	0.04869 (18)
N1	0.5280 (2)	0.53743 (15)	0.75195 (8)	0.0298 (3)
O1	0.4920 (2)	0.62161 (16)	0.90967 (8)	0.0474 (4)
H1	0.653 (3)	0.539 (2)	0.7483 (11)	0.035 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0425 (10)	0.0391 (10)	0.0307 (9)	−0.0011 (8)	−0.0046 (8)	−0.0078 (8)
C2	0.0561 (14)	0.0662 (15)	0.0426 (12)	0.0132 (12)	−0.0063 (10)	−0.0028 (11)
C3	0.104 (2)	0.0709 (18)	0.0483 (14)	0.0241 (17)	−0.0155 (15)	0.0058 (13)
C4	0.122 (3)	0.0585 (16)	0.0336 (12)	−0.0110 (16)	−0.0023 (14)	0.0046 (11)
C5	0.0741 (17)	0.0709 (17)	0.0426 (12)	−0.0183 (14)	0.0092 (12)	−0.0018 (12)
C6	0.0469 (12)	0.0565 (13)	0.0411 (11)	−0.0007 (10)	0.0011 (9)	−0.0041 (10)
C7	0.0377 (10)	0.0376 (10)	0.0387 (10)	−0.0044 (8)	−0.0013 (8)	−0.0083 (8)
C8	0.0358 (10)	0.0286 (9)	0.0388 (10)	−0.0001 (7)	−0.0007 (8)	−0.0012 (7)
C9	0.0502 (12)	0.0367 (10)	0.0412 (11)	−0.0036 (9)	−0.0002 (9)	−0.0056 (9)
C10	0.0529 (12)	0.0467 (12)	0.0390 (11)	0.0001 (10)	0.0021 (9)	0.0094 (9)
C11	0.0473 (11)	0.0307 (10)	0.0434 (11)	−0.0041 (8)	0.0053 (9)	0.0050 (8)
Cl1	0.0277 (3)	0.0459 (3)	0.0725 (4)	−0.0008 (2)	0.0016 (2)	0.0025 (3)
N1	0.0263 (8)	0.0293 (8)	0.0339 (8)	−0.0013 (6)	0.0019 (6)	0.0002 (6)
O1	0.0565 (9)	0.0516 (9)	0.0343 (7)	−0.0019 (7)	0.0076 (6)	−0.0022 (6)

Geometric parameters (Å, °)

C1—C2	1.378 (3)	C8—N1	1.490 (2)
C1—C6	1.381 (3)	C8—C9	1.507 (3)
C1—C7	1.497 (3)	C8—H8A	0.9700
C2—C3	1.382 (4)	C8—H8B	0.9700
C2—H2	0.9300	C9—O1	1.420 (2)
C3—C4	1.366 (4)	C9—H9A	0.9700
C3—H3	0.9300	C9—H9B	0.9700
C4—C5	1.359 (4)	C10—O1	1.419 (3)
C4—H4	0.9300	C10—C11	1.504 (3)
C5—C6	1.376 (3)	C10—H10A	0.9700
C5—H5	0.9300	C10—H10B	0.9700
C6—H6	0.9300	C11—N1	1.492 (2)
C7—N1	1.505 (2)	C11—H11A	0.9700
C7—H7A	0.9700	C11—H11B	0.9700
C7—H7B	0.9700	N1—H1	0.88 (2)
C2—C1—C6	118.7 (2)	C9—C8—H8B	109.7
C2—C1—C7	120.37 (19)	H8A—C8—H8B	108.2
C6—C1—C7	120.88 (18)	O1—C9—C8	111.65 (16)
C1—C2—C3	120.1 (2)	O1—C9—H9A	109.3
C1—C2—H2	119.9	C8—C9—H9A	109.3
C3—C2—H2	119.9	O1—C9—H9B	109.3
C4—C3—C2	120.4 (3)	C8—C9—H9B	109.3
C4—C3—H3	119.8	H9A—C9—H9B	108.0

C2—C3—H3	119.8	O1—C10—C11	111.00 (17)
C5—C4—C3	119.9 (2)	O1—C10—H10A	109.4
C5—C4—H4	120.0	C11—C10—H10A	109.4
C3—C4—H4	120.0	O1—C10—H10B	109.4
C4—C5—C6	120.3 (3)	C11—C10—H10B	109.4
C4—C5—H5	119.8	H10A—C10—H10B	108.0
C6—C5—H5	119.8	N1—C11—C10	109.97 (16)
C5—C6—C1	120.5 (2)	N1—C11—H11A	109.7
C5—C6—H6	119.7	C10—C11—H11A	109.7
C1—C6—H6	119.7	N1—C11—H11B	109.7
C1—C7—N1	113.07 (15)	C10—C11—H11B	109.7
C1—C7—H7A	109.0	H11A—C11—H11B	108.2
N1—C7—H7A	109.0	C8—N1—C11	109.52 (14)
C1—C7—H7B	109.0	C8—N1—C7	112.93 (14)
N1—C7—H7B	109.0	C11—N1—C7	110.42 (14)
H7A—C7—H7B	107.8	C8—N1—H1	109.0 (13)
N1—C8—C9	109.75 (15)	C11—N1—H1	106.8 (13)
N1—C8—H8A	109.7	C7—N1—H1	107.9 (13)
C9—C8—H8A	109.7	C10—O1—C9	109.93 (15)
N1—C8—H8B	109.7		
C6—C1—C2—C3	−0.5 (3)	N1—C8—C9—O1	−57.6 (2)
C7—C1—C2—C3	179.7 (2)	O1—C10—C11—N1	58.7 (2)
C1—C2—C3—C4	0.1 (4)	C9—C8—N1—C11	54.2 (2)
C2—C3—C4—C5	0.4 (4)	C9—C8—N1—C7	177.69 (15)
C3—C4—C5—C6	−0.5 (4)	C10—C11—N1—C8	−55.0 (2)
C4—C5—C6—C1	0.0 (4)	C10—C11—N1—C7	−179.95 (16)
C2—C1—C6—C5	0.5 (3)	C1—C7—N1—C8	56.0 (2)
C7—C1—C6—C5	−179.80 (19)	C1—C7—N1—C11	179.01 (16)
C2—C1—C7—N1	−104.2 (2)	C11—C10—O1—C9	−61.0 (2)
C6—C1—C7—N1	76.1 (2)	C8—C9—O1—C10	60.6 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1	0.88 (2)	2.19 (2)	3.062 (2)	172 (2)
C7—H7B···Cl1^{i}	0.97	2.86	3.725 (2)	149
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C8—H8B···Cl1^{ii}	0.97	2.87	3.576 (2)	130
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Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*+3/2, *y*+1/2, −*z*+3/2; (iii) −*x*+3/2, *y*−1/2, −*z*+3/2.

Fig. 1

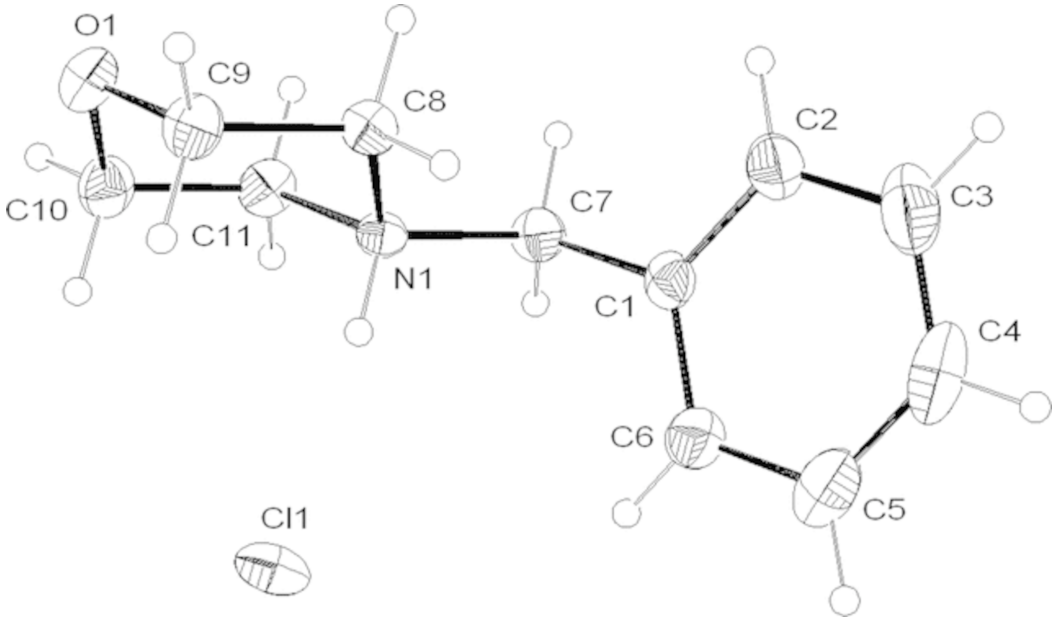

Fig. 2