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## Structure Reports

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# N-Benzyl-N-ethylmorpholinium chloride

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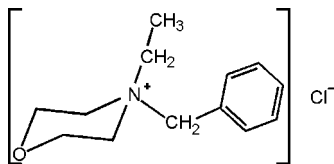
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 Key indicators: single-crystal X-ray study;  $T = 113$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.032;  $wR$  factor = 0.090; data-to-parameter ratio = 15.5.

In the crystal structure of the title compound,  $\text{C}_{13}\text{H}_{20}\text{NO}^+\cdot\text{Cl}^-$ , the morpholine ring is in a chair conformation and the molecules are linked by weak intermolecular  $\text{C}-\text{H}\cdots\text{Cl}$  hydrogen bonding.

## Related literature

For details of the importance of quaternary morpholine halides see: Kim *et al.* (2005, 2006).



## Experimental

### Crystal data

$\text{C}_{13}\text{H}_{20}\text{NO}^+\cdot\text{Cl}^-$   
 $M_r = 241.75$   
 Monoclinic,  $P2_1/c$   
 $a = 13.179$  (3) Å

$b = 8.4176$  (17) Å  
 $c = 12.255$  (3) Å  
 $\beta = 108.48$  (3)°  
 $V = 1289.5$  (4) Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.28$  mm<sup>-1</sup>

$T = 113$  (2) K  
 $0.16 \times 0.16 \times 0.06$  mm

### Data collection

Rigaku Saturn CCD area-detector diffractometer  
 Absorption correction: multi-scan (*CrystalClear*; Rigaku/MS, 2005)  
 $T_{\min} = 0.957$ ,  $T_{\max} = 0.984$

7151 measured reflections  
 2266 independent reflections  
 2017 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.031$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.090$   
 $S = 1.07$   
 2266 reflections

146 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.19$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C1}-\text{H1B}\cdots\text{Cl1}^{\text{i}}$	0.99	2.74	3.4724 (16)	131
$\text{C5}-\text{H5B}\cdots\text{Cl1}^{\text{ii}}$	0.99	2.61	3.5500 (16)	158
$\text{C11}-\text{H11}\cdots\text{Cl1}$	0.95	2.66	3.5501 (16)	156
$\text{C12}-\text{H12B}\cdots\text{Cl1}^{\text{iii}}$	0.99	2.61	3.5062 (16)	151

Symmetry codes: (i)  $-x + 1, -y + 2, -z + 1$ ; (ii)  $x, -y + \frac{3}{2}, z + \frac{1}{2}$ ; (iii)  $x, -y + \frac{5}{2}, z + \frac{1}{2}$ .

Data collection: *CrystalClear* (Rigaku/MS, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXS97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2129).

## References

- Kim, K. S., Choi, S., Cha, J. H., Yeon, S. H. & Lee, H. (2006). *J. Mater. Chem.* **16**, 1315–1317.  
 Kim, K. S., Park, S. Y., Yeon, S. H. & Lee, H. (2005). *Electrochim. Acta*, **50**, 5673–5678.  
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**supplementary materials**

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## ***N*-Benzyl-*N*-ethylmorpholinium chloride**

**Y.-J. Bian**

### **Comment**

Quaternary morpholine halides are valuable precursors for the preparation of ionic liquids by ion metathesis (Kim *et al.*,2005). Ionic liquids based on the morpholinium cation are favored because of their low cost, easy synthesis, and electrochemical stability (Kim *et al.*,2006). Here we report a new structure of this class of compounds.

In the crystal structure the morpholine ring adopts a chair conformation (Fig. 1). The cations and anions are connected via weak C—H···Cl hydrogen bonding into a three-dimensional network (Tab 1).

### **Experimental**

Benzyl chloride(0.12 mol) was added to a solution of 4-ethylmorpholine(0.1 mol) in 20 ml of acetonitrile under stirring. The mixture was stirred at 60 °C for 5 h. The solvent was removed under reduced pressure. The remaining brownish, viscous liquid crystallized slowly at room temperature in a mixture of ethanol and acetone [1/20(v/v)]. Single-crystals were obtained by slow evaporation of the solvent from a solution in a mixture of ethanol and acetone [1/20(v/v)].

### **Refinement**

The H atoms were positioned with idealized geometry and were refined isotropically using a riding model with C—H = 0.96–0.97 Å and  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$  for aromatic and methylene H atoms as well as  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$  for methyl H atoms.

### **Figures**

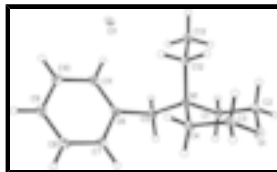


Fig. 1. Crystal structure of the title compound with the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level.

## ***N*-Benzyl-*N*-ethylmorpholinium chloride**

### *Crystal data*

$\text{C}_{13}\text{H}_{20}\text{NO}^+\cdot\text{Cl}^-$

$M_r = 241.75$

Monoclinic,  $P2_1/c$

$a = 13.179(3) \text{ \AA}$

$F_{000} = 520$

$D_x = 1.245 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3736 reflections

# supplementary materials

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$b = 8.4176 (17) \text{ \AA}$	$\theta = 1.6\text{--}27.9^\circ$
$c = 12.255 (3) \text{ \AA}$	$\mu = 0.28 \text{ mm}^{-1}$
$\beta = 108.48 (3)^\circ$	$T = 113 (2) \text{ K}$
$V = 1289.5 (4) \text{ \AA}^3$	Prism, colorless
$Z = 4$	$0.16 \times 0.16 \times 0.06 \text{ mm}$

## Data collection

Rigaku Saturn CCD area-detector diffractometer	2266 independent reflections
Radiation source: rotating anode	2017 reflections with $I > 2\sigma(I)$
Monochromator: confocal	$R_{\text{int}} = 0.031$
$T = 113(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
$\omega$ and $\varphi$ scans	$\theta_{\text{min}} = 2.9^\circ$
Absorption correction: multi-scan (CrystalClear; Rigaku/MSO, 2005)	$h = -15 \rightarrow 14$
$T_{\text{min}} = 0.957$ , $T_{\text{max}} = 0.984$	$k = -9 \rightarrow 10$
7151 measured reflections	$l = -7 \rightarrow 14$

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.032$	H-atom parameters constrained
$wR(F^2) = 0.090$	$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.1492P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
2266 reflections	$(\Delta/\sigma)_{\text{max}} = 0.002$
146 parameters	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
	Extinction correction: none

## Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
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Cl1	0.31149 (3)	1.03231 (4)	0.23458 (3)	0.02272 (14)
O1	0.37595 (8)	0.96775 (12)	0.89939 (8)	0.0230 (3)
N1	0.30801 (9)	0.93219 (13)	0.64941 (10)	0.0165 (3)
C1	0.41986 (11)	0.89933 (18)	0.72853 (12)	0.0199 (3)
H1A	0.4258	0.7859	0.7509	0.024*
H1B	0.4718	0.9204	0.6870	0.024*
C2	0.44732 (12)	1.00099 (18)	0.83543 (12)	0.0220 (3)
H2A	0.5218	0.9794	0.8838	0.026*
H2B	0.4420	1.1146	0.8135	0.026*
C3	0.27034 (12)	1.00809 (18)	0.83116 (13)	0.0227 (3)
H3A	0.2674	1.1225	0.8116	0.027*
H3B	0.2208	0.9892	0.8760	0.027*
C4	0.23423 (11)	0.91112 (18)	0.72105 (12)	0.0201 (3)
H4A	0.2319	0.7974	0.7407	0.024*
H4B	0.1610	0.9438	0.6752	0.024*
C5	0.28285 (11)	0.80630 (17)	0.55463 (12)	0.0198 (3)
H5A	0.3283	0.8261	0.5054	0.024*
H5B	0.3025	0.7008	0.5908	0.024*
C6	0.16760 (11)	0.80131 (16)	0.47930 (12)	0.0182 (3)
C7	0.09501 (12)	0.70572 (17)	0.51050 (13)	0.0224 (3)
H7	0.1174	0.6504	0.5817	0.027*
C8	-0.00926 (12)	0.69048 (18)	0.43888 (13)	0.0260 (4)
H8	-0.0586	0.6273	0.4620	0.031*
C9	-0.04183 (12)	0.76705 (18)	0.33380 (13)	0.0263 (4)
H9	-0.1128	0.7536	0.2834	0.032*
C10	0.02947 (12)	0.86372 (19)	0.30209 (13)	0.0268 (4)
H10	0.0068	0.9180	0.2304	0.032*
C11	0.13372 (12)	0.88136 (18)	0.37469 (12)	0.0224 (3)
H11	0.1820	0.9483	0.3529	0.027*
C12	0.29561 (12)	1.09797 (17)	0.59839 (12)	0.0227 (3)
H12A	0.2232	1.1076	0.5417	0.027*
H12B	0.3008	1.1757	0.6606	0.027*
C13	0.37708 (14)	1.1417 (2)	0.53976 (13)	0.0328 (4)
H13A	0.4493	1.1329	0.5950	0.049*
H13B	0.3646	1.2512	0.5113	0.049*
H13C	0.3700	1.0695	0.4751	0.049*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl1	0.0235 (2)	0.0170 (2)	0.0296 (2)	-0.00200 (13)	0.01123 (17)	-0.00037 (14)
O1	0.0241 (6)	0.0242 (6)	0.0192 (5)	-0.0001 (4)	0.0046 (5)	0.0020 (4)
N1	0.0168 (6)	0.0128 (6)	0.0188 (6)	-0.0008 (5)	0.0043 (5)	-0.0006 (5)
C1	0.0151 (7)	0.0203 (8)	0.0219 (7)	0.0008 (6)	0.0023 (6)	0.0000 (6)
C2	0.0194 (7)	0.0230 (8)	0.0213 (7)	-0.0024 (6)	0.0035 (6)	-0.0002 (6)
C3	0.0225 (8)	0.0234 (8)	0.0227 (8)	-0.0002 (6)	0.0080 (6)	-0.0017 (6)
C4	0.0178 (7)	0.0189 (8)	0.0250 (8)	-0.0014 (6)	0.0086 (6)	-0.0012 (6)
C5	0.0205 (7)	0.0140 (7)	0.0243 (7)	-0.0012 (6)	0.0065 (6)	-0.0044 (6)

## supplementary materials

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C6	0.0195 (7)	0.0127 (7)	0.0216 (7)	0.0000 (6)	0.0054 (6)	-0.0052 (6)
C7	0.0266 (8)	0.0139 (7)	0.0247 (8)	-0.0027 (6)	0.0053 (7)	0.0001 (6)
C8	0.0223 (8)	0.0187 (8)	0.0362 (9)	-0.0044 (6)	0.0082 (7)	-0.0017 (7)
C9	0.0203 (7)	0.0208 (8)	0.0325 (8)	0.0022 (6)	0.0007 (7)	-0.0042 (7)
C10	0.0298 (8)	0.0251 (9)	0.0221 (8)	0.0047 (7)	0.0034 (7)	0.0010 (6)
C11	0.0262 (8)	0.0200 (8)	0.0226 (8)	-0.0018 (6)	0.0100 (7)	-0.0017 (6)
C12	0.0305 (8)	0.0122 (7)	0.0210 (7)	-0.0014 (6)	0.0020 (6)	0.0012 (6)
C13	0.0501 (10)	0.0252 (9)	0.0227 (8)	-0.0146 (8)	0.0108 (8)	0.0002 (7)

### *Geometric parameters (Å, °)*

O1—C3	1.4191 (18)	C5—H5B	0.9900
O1—C2	1.4300 (17)	C6—C11	1.391 (2)
N1—C1	1.5111 (18)	C6—C7	1.393 (2)
N1—C4	1.5129 (18)	C7—C8	1.382 (2)
N1—C12	1.5167 (18)	C7—H7	0.9500
N1—C5	1.5290 (18)	C8—C9	1.381 (2)
C1—C2	1.510 (2)	C8—H8	0.9500
C1—H1A	0.9900	C9—C10	1.388 (2)
C1—H1B	0.9900	C9—H9	0.9500
C2—H2A	0.9900	C10—C11	1.388 (2)
C2—H2B	0.9900	C10—H10	0.9500
C3—C4	1.519 (2)	C11—H11	0.9500
C3—H3A	0.9900	C12—C13	1.515 (2)
C3—H3B	0.9900	C12—H12A	0.9900
C4—H4A	0.9900	C12—H12B	0.9900
C4—H4B	0.9900	C13—H13A	0.9800
C5—C6	1.507 (2)	C13—H13B	0.9800
C5—H5A	0.9900	C13—H13C	0.9800
C3—O1—C2	108.83 (11)	C6—C5—H5B	108.6
C1—N1—C4	106.37 (10)	N1—C5—H5B	108.6
C1—N1—C12	112.87 (11)	H5A—C5—H5B	107.6
C4—N1—C12	110.00 (10)	C11—C6—C7	119.02 (14)
C1—N1—C5	107.05 (11)	C11—C6—C5	121.18 (13)
C4—N1—C5	109.58 (10)	C7—C6—C5	119.63 (13)
C12—N1—C5	110.82 (11)	C8—C7—C6	120.69 (14)
C2—C1—N1	111.70 (12)	C8—C7—H7	119.7
C2—C1—H1A	109.3	C6—C7—H7	119.7
N1—C1—H1A	109.3	C9—C8—C7	120.09 (14)
C2—C1—H1B	109.3	C9—C8—H8	120.0
N1—C1—H1B	109.3	C7—C8—H8	120.0
H1A—C1—H1B	107.9	C8—C9—C10	119.78 (14)
O1—C2—C1	110.26 (12)	C8—C9—H9	120.1
O1—C2—H2A	109.6	C10—C9—H9	120.1
C1—C2—H2A	109.6	C11—C10—C9	120.23 (14)
O1—C2—H2B	109.6	C11—C10—H10	119.9
C1—C2—H2B	109.6	C9—C10—H10	119.9
H2A—C2—H2B	108.1	C10—C11—C6	120.17 (14)
O1—C3—C4	111.57 (12)	C10—C11—H11	119.9

O1—C3—H3A	109.3	C6—C11—H11	119.9
C4—C3—H3A	109.3	C13—C12—N1	114.80 (13)
O1—C3—H3B	109.3	C13—C12—H12A	108.6
C4—C3—H3B	109.3	N1—C12—H12A	108.6
H3A—C3—H3B	108.0	C13—C12—H12B	108.6
N1—C4—C3	112.00 (11)	N1—C12—H12B	108.6
N1—C4—H4A	109.2	H12A—C12—H12B	107.5
C3—C4—H4A	109.2	C12—C13—H13A	109.5
N1—C4—H4B	109.2	C12—C13—H13B	109.5
C3—C4—H4B	109.2	H13A—C13—H13B	109.5
H4A—C4—H4B	107.9	C12—C13—H13C	109.5
C6—C5—N1	114.73 (11)	H13A—C13—H13C	109.5
C6—C5—H5A	108.6	H13B—C13—H13C	109.5
N1—C5—H5A	108.6		
C4—N1—C1—C2	-54.32 (14)	N1—C5—C6—C11	95.68 (15)
C12—N1—C1—C2	66.40 (15)	N1—C5—C6—C7	-89.09 (16)
C5—N1—C1—C2	-171.41 (11)	C11—C6—C7—C8	-0.1 (2)
C3—O1—C2—C1	-62.64 (15)	C5—C6—C7—C8	-175.43 (13)
N1—C1—C2—O1	61.35 (15)	C6—C7—C8—C9	1.7 (2)
C2—O1—C3—C4	61.11 (15)	C7—C8—C9—C10	-2.2 (2)
C1—N1—C4—C3	52.26 (15)	C8—C9—C10—C11	1.1 (2)
C12—N1—C4—C3	-70.29 (15)	C9—C10—C11—C6	0.5 (2)
C5—N1—C4—C3	167.65 (12)	C7—C6—C11—C10	-1.0 (2)
O1—C3—C4—N1	-57.89 (16)	C5—C6—C11—C10	174.27 (13)
C1—N1—C5—C6	169.51 (11)	C1—N1—C12—C13	52.59 (16)
C4—N1—C5—C6	54.55 (15)	C4—N1—C12—C13	171.21 (12)
C12—N1—C5—C6	-67.02 (15)	C5—N1—C12—C13	-67.46 (15)

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1B $\cdots$ C11 <sup>i</sup>	0.99	2.74	3.4724 (16)	131
C5—H5B $\cdots$ C11 <sup>ii</sup>	0.99	2.61	3.5500 (16)	158
C11—H11 $\cdots$ C11	0.95	2.66	3.5501 (16)	156
C12—H12B $\cdots$ C11 <sup>iii</sup>	0.99	2.61	3.5062 (16)	151

Symmetry codes: (i)  $-x+1, -y+2, -z+1$ ; (ii)  $x, -y+3/2, z+1/2$ ; (iii)  $x, -y+5/2, z+1/2$ .

Fig. 1

