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4-Methylmorpholinium bromide

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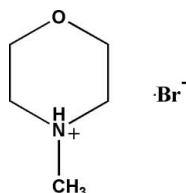
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.037; wR factor = 0.098; data-to-parameter ratio = 19.9.

The six-membered ring in the title salt, $\text{C}_5\text{H}_{12}\text{NO}^+\cdot\text{Br}^-$, has a chair conformation. In the crystal, the cations are linked to the anions by $\text{N}-\text{H}\cdots\text{Br}$ hydrogen bonds.

Related literature

For background to phase transition materials, see: Hang *et al.* (2009); Zhang *et al.* (2009).



Experimental

Crystal data

$\text{C}_5\text{H}_{12}\text{NO}^+\cdot\text{Br}^-$
 $M_r = 182.07$
Monoclinic, $P2_1/m$
 $a = 7.3282$ (15) Å
 $b = 7.4170$ (15) Å

$c = 7.3928$ (15) Å
 $\beta = 92.72$ (3)°
 $V = 401.37$ (14) Å³
 $Z = 2$
Mo $K\alpha$ radiation

$\mu = 5.04$ mm⁻¹
 $T = 293$ K

0.40 × 0.30 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan
(*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.178$, $T_{\max} = 0.365$

4192 measured reflections
995 independent reflections
866 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.098$
 $S = 0.97$
995 reflections

50 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.33$ e Å⁻³
 $\Delta\rho_{\min} = -0.72$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1B}\cdots\text{Br1}$	0.90	2.30	3.202 (4)	179

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *PRPKAPPA* (Ferguson, 1999).

The author is grateful to the starter fund of Southeast University for supporting the purchase of a diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2770).

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supplementary materials

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Comment

As a study of phase transition materials, including organic ligands (Hang *et al.* 2009), metal-organic coordination compounds (Zhang *et al.*, 2009), organic-inorganic hybrids, we studied the dielectric properties of the title compound, but there was no distinct anomaly observed from 90 K to 420 K, (m.p. 450 K) unfortunately. In this article, the crystal structure of (I) is showed.

The structure is composed of the *N*-Methylmorpholinium cations, hydrobromide anions (Fig. 1). in space group P21/m.

Packing structure of the title compound along b-axis are shown in Figure 2. *N*-Methylmorpholinium molecules are linked *via* hydrogen bonds of the type N—H \cdots Br hydrogen bonds forming a two-dimensional planar sheets with hydrobromide anions. The hydrogen bonds are given in Table 1. The H atom of the protonated ring N atom (H1b) is donated to the Br⁻ anions, being involved in a strong N—H \cdots Br hydrogen bond. Br⁻ anions take part in electrostatics equilibrium with the *N*-Methylmorpholinium cations. The associated distances and angles are: Br \cdots H—N 3.202 (4) Å, and 179.3°.

Experimental

The title compound was prepared by reaction of stoichiometric amounts of *N*-Methylmorpholinium and concentrated hydrobromic acid in methanol. The obtained solution was filtered, and left at room temperature for 5 days. colorless crystals were obtained by slow evaporation.

Refinement

Positional parameters of all the H atoms were calculated geometrically and were allowed to ride on the C, N atoms to which they are bonded, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$,

$$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N}).$$

Figures

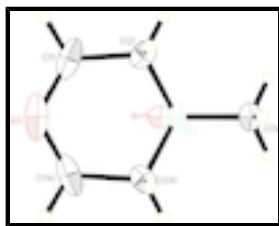


Fig. 1. The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level, and all H atoms have been omitted for clarity.

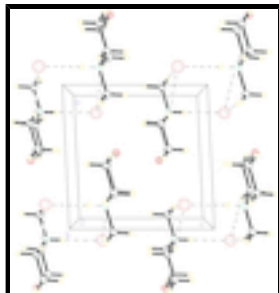


Fig. 2. A view of the packing of the title compound, stacking along the *b* axis. Dashed lines indicate hydrogen bonds.

4-Methylmorpholinium bromide

Crystal data

$C_5H_{12}NO^+ \cdot Br^-$	$F(000) = 184$
$M_r = 182.07$	$D_x = 1.506 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: $-P 2yb$	Cell parameters from 0 reflections
$a = 7.3282 (15) \text{ \AA}$	$\theta = 3.8\text{--}27.5^\circ$
$b = 7.4170 (15) \text{ \AA}$	$\mu = 5.04 \text{ mm}^{-1}$
$c = 7.3928 (15) \text{ \AA}$	$T = 293 \text{ K}$
$\beta = 92.72 (3)^\circ$	Prism, colourless
$V = 401.37 (14) \text{ \AA}^3$	$0.40 \times 0.30 \times 0.20 \text{ mm}$
$Z = 2$	

Data collection

Rigaku Mercury2 diffractometer	995 independent reflections
Radiation source: fine-focus sealed tube graphite	866 reflections with $I > 2\sigma(I)$
Detector resolution: $13.6612 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.046$
CCD_Profile_fitting scans	$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.8^\circ$
Absorption correction: multi-scan (<i>CrystalClear</i> ; Rigaku, 2005)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.178$, $T_{\text{max}} = 0.365$	$k = -9 \rightarrow 9$
4192 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.037$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.098$	H-atom parameters constrained
$S = 0.97$	$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.1157P]$
	where $P = (F_o^2 + 2F_c^2)/3$

995 reflections	$(\Delta/\sigma)_{\max} < 0.001$
50 parameters	$\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.72 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.5268 (5)	0.7500	0.3399 (6)	0.0810 (14)
N1	0.1476 (4)	0.7500	0.2465 (5)	0.0352 (7)
H1B	0.1507	0.7500	0.1250	0.062 (16)*
C1	0.4418 (5)	0.5939 (7)	0.2671 (6)	0.0749 (13)
H1A	0.4478	0.5968	0.1377	0.096 (16)*
H1C	0.5054	0.4884	0.3106	0.110 (18)*
C2	0.2434 (4)	0.5854 (5)	0.3149 (5)	0.0488 (8)
H2A	0.1864	0.4810	0.2604	0.054 (10)*
H2B	0.2359	0.5765	0.4438	0.059 (11)*
C5	-0.0464 (6)	0.7500	0.2948 (7)	0.0484 (11)
H5A	-0.1058	0.6449	0.2455	0.061 (11)*
H5B	-0.0534	0.7500	0.4234	0.059 (16)*
Br1	0.15472 (6)	0.7500	-0.18624 (5)	0.0464 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0332 (17)	0.143 (4)	0.066 (3)	0.000	-0.0037 (18)	0.000
N1	0.0288 (16)	0.047 (2)	0.0300 (17)	0.000	0.0003 (13)	0.000
C1	0.047 (2)	0.116 (4)	0.062 (3)	0.034 (2)	0.0023 (18)	-0.007 (3)
C2	0.0464 (17)	0.0512 (19)	0.0482 (19)	0.0130 (15)	-0.0023 (13)	-0.0017 (15)
C5	0.032 (2)	0.056 (3)	0.057 (3)	0.000	0.004 (2)	0.000
Br1	0.0608 (3)	0.0470 (3)	0.0314 (3)	0.000	0.0005 (2)	0.000

Geometric parameters (\AA , $^\circ$)

O1—C1	1.409 (5)	C1—H1A	0.9600
O1—C1 ⁱ	1.409 (5)	C1—H1C	0.9578
N1—C5	1.482 (5)	C2—H2A	0.9597

supplementary materials

N1—C2 ⁱ	1.485 (4)	C2—H2B	0.9596
N1—C2	1.485 (4)	C5—H5A	0.9562
N1—H1B	0.8997	C5—H5B	0.9550
C1—C2	1.514 (5)		
C1—O1—C1 ⁱ	110.5 (4)	C2—C1—H1C	110.2
C5—N1—C2 ⁱ	111.2 (2)	H1A—C1—H1C	108.0
C5—N1—C2	111.2 (2)	N1—C2—C1	109.3 (3)
C2 ⁱ —N1—C2	110.6 (3)	N1—C2—H2A	109.3
C5—N1—H1B	108.1	C1—C2—H2A	109.9
C2 ⁱ —N1—H1B	107.8	N1—C2—H2B	110.3
C2—N1—H1B	107.8	C1—C2—H2B	109.6
O1—C1—C2	110.9 (3)	H2A—C2—H2B	108.4
O1—C1—H1A	108.8	N1—C5—H5A	109.4
C2—C1—H1A	108.9	N1—C5—H5B	109.7
O1—C1—H1C	110.1	H5A—C5—H5B	109.5
C1 ⁱ —O1—C1—C2	-61.8 (5)	C2 ⁱ —N1—C2—C1	-54.4 (4)
C5—N1—C2—C1	-178.4 (3)	O1—C1—C2—N1	57.8 (4)

Symmetry codes: (i) $x, -y+3/2, z$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1B \cdots Br1	0.90	2.30	3.202 (4)	179.

Fig. 1

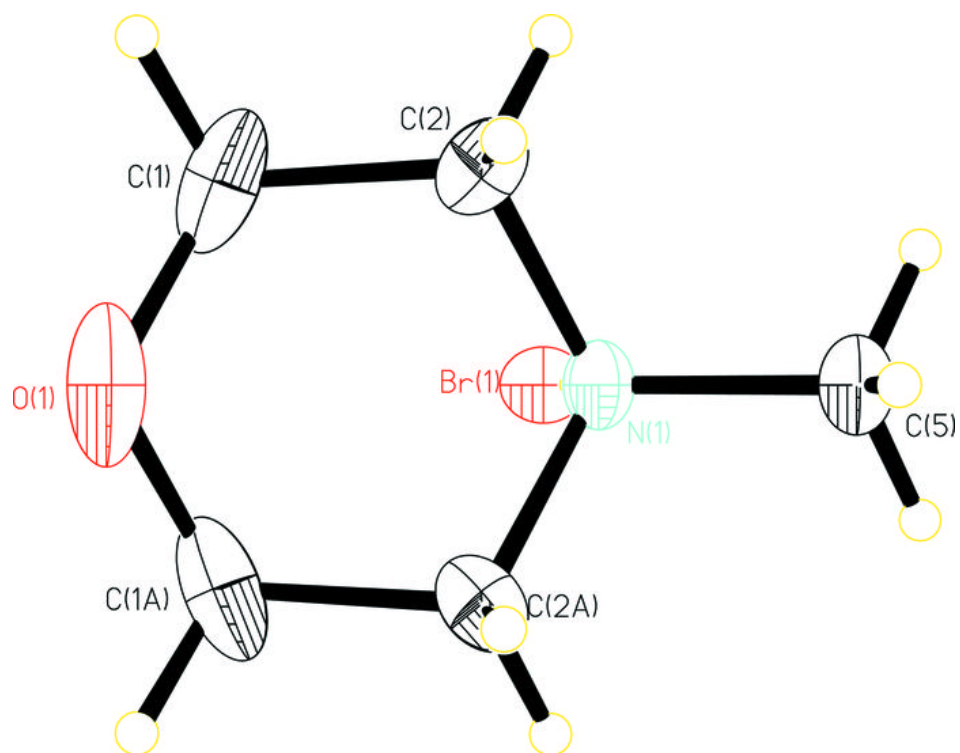


Fig. 2

