Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 4-Methylmorpholinium bromide

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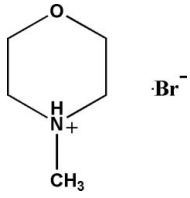
Received 8 May 2010; accepted 12 May 2010

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–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, $C_5H_{12}NO^+\cdot Br^-$, has a chair conformation. In the crystal, the cations are linked to the anions by N—H···Br hydrogen bonds.

## Related literature

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



## Experimental

### Crystal data

$C_5H_{12}NO^+\cdot Br^-$
$M_r$ = 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) Å$^3$
$Z$ = 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 $I > 2\sigma(I)$
$R_{int}$ = 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···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$B$···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).

## References

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# supporting information

*Acta Cryst.* (2010). E**66**, o1375 [https://doi.org/10.1107/S1600536810017447]

## 4-Methylmorpholinium bromide

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### S1. 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···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 Brl$^-$ anions, being involved in a strong N—H···Br hydrogen bond. Br$^-$ anions take part in electrostatics equilibrium with the *N*-Methylmorpholinium cations. The associated distances and angles are: Br···H—N 3.202 (4) Å, and 179.3°.

### S2. 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.

### S3. 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 $U_{iso}$(H) = 1.2U$_{eq}$(C),

$U_{iso}$(H) = 1.2U$_{eq}$(N).

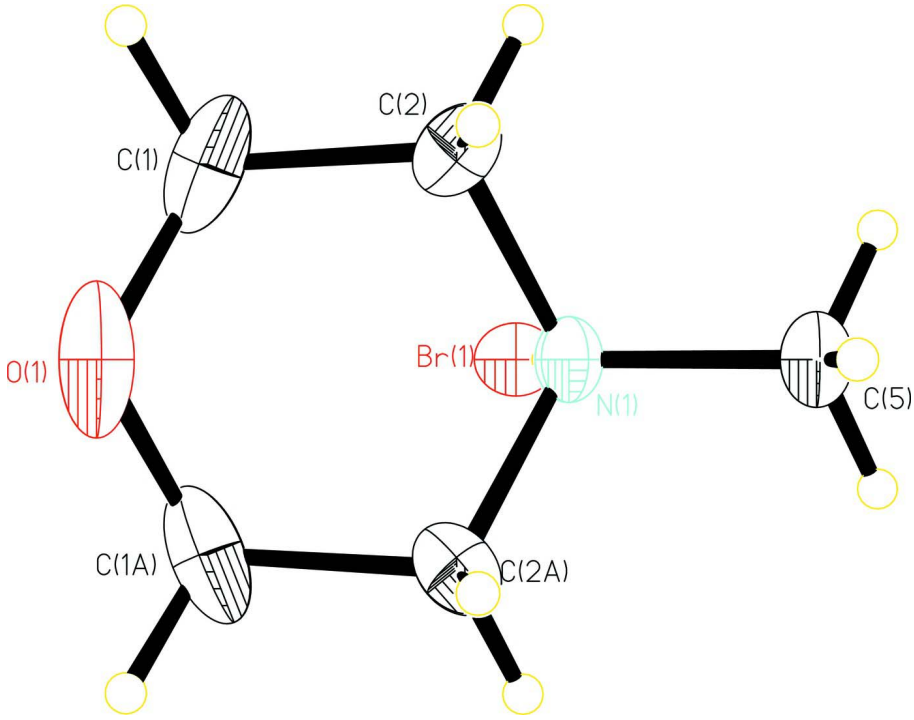


**Figure 1**

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.

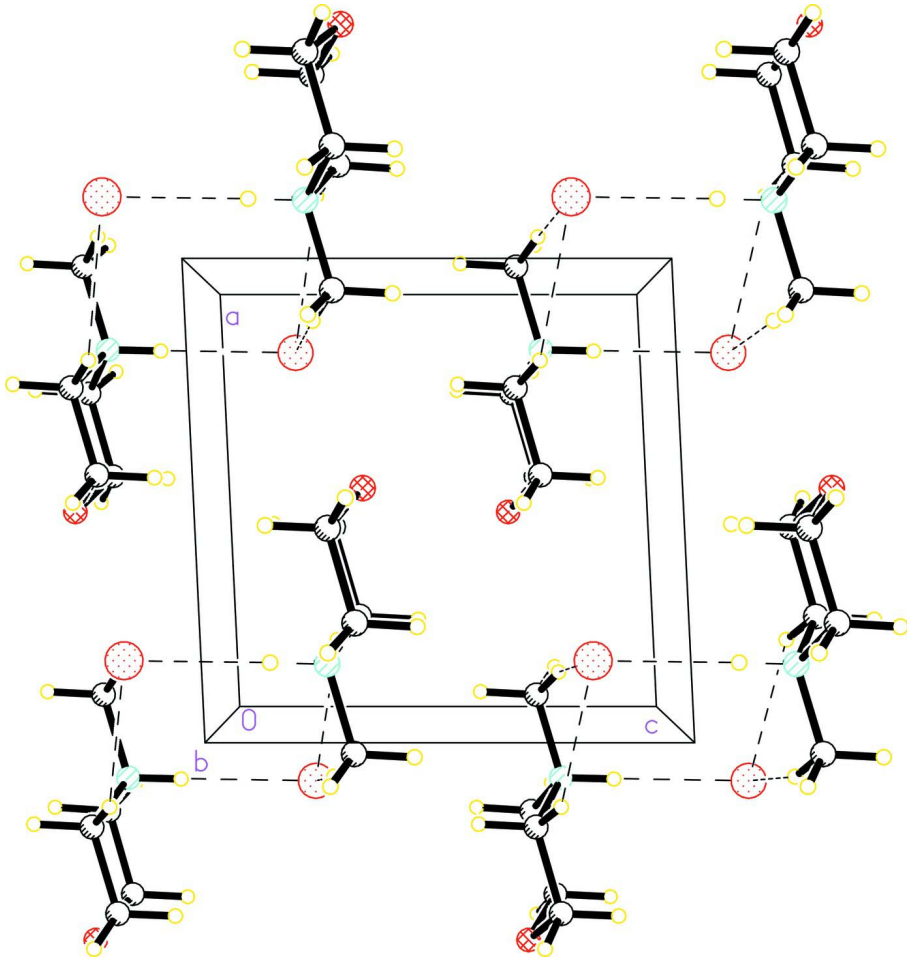


**Figure 2**

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

### 4-Methylmorpholinium bromide

*Crystal data*

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

*Data collection*

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm$^{-1}$
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min}$ = 0.178, $T_{max}$ = 0.365
4192 measured reflections

995 independent reflections
866 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.8°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -9 \rightarrow 9$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.098$
$S = 0.97$
995 reflections
50 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.1157P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å$^{-3}$
$\Delta\rho_{min} = -0.72$ e Å$^{-3}$

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 (Å, º)*

| | | | |
|---|---|---|---|
| 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 |
| 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 code: (i) $x$, $-y+3/2$, $z$.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*B*···Br1 | 0.90 | 2.30 | 3.202 (4) | 179 |