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1,4-Dimethylpiperazine-1,4-diium dibromide dihydrate

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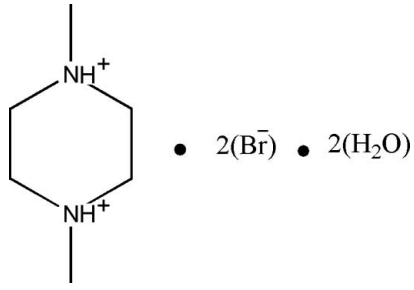
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.059; wR factor = 0.169; data-to-parameter ratio = 21.4.

In the title hydrated molecular salt, $C_6H_{16}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$, the complete 1,4-dimethylpiperazine-1,4-diium dication is generated by crystallographic inversion symmetry and both exocyclic C—N bonds are in equatorial orientations. In the crystal, the components are linked by N—H···O and O—H···Br hydrogen bonds, generating chains propagating in [110].

Related literature

For background to molecular ferroelectrics, see: Fu *et al.* (2009).



Experimental

Crystal data

$C_6H_{16}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$ M_r = 312.06
Triclinic, $P\overline{1}$
a = 6.2975 (13) Å
b = 7.0180 (14) Å
c = 7.2143 (14) Å
α = 71.54 (3)°
β = 86.62 (3)°
γ = 85.54 (3)°
V = 301.32 (10) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 6.70 mm^{-1}
T = 293 K
0.30 × 0.30 × 0.20 mm

Data collection

Rigaku SCXmini CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.154, T_{max} = 0.262
3084 measured reflections
1370 independent reflections
1131 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.169
S = 1.02
1370 reflections
64 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −1.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.91	1.83	2.737 (9)	174
O1—H1WA···Br1^{ii}	0.85 (6)	2.51 (9)	3.275 (7)	151 (11)
O1—H1WB···Br1	0.85 (3)	2.41 (4)	3.244 (7)	166 (7)

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y, -z+1$.

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* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

The author is grateful to the starter fund of Southeast University for financial support to buy the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6759).

References

Fu, D.-W., Ge, J.-Z., Dai, J., Ye, H.-Y. & Qu, Z.-R. (2009). *Inorg. Chem. Commun.* **12**, 994–997.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan .
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2012). E**68**, o1703 [doi:10.1107/S1600536812020272]

1,4-Dimethylpiperazine-1,4-diium dibromide dihydrate

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Comment

Dielectric constant measurements of compounds as a function of temperature is the basic methods to find the materials which possess potential ferroelectric phase changes (Fu *et al.*, 2009). The dielectric constant of the title compound has been measured,but showed no dielectric disuniformity in the range 113–353 K(mp.403–410 K).

The asymmetric unit of the title compound is shown in Fig. 1. crystallized in the monoclinic P-1 space group, The crystal packing Fig. 2 features weak intermolecular O—H···Br and N—H···O hydrogen bonds (Table 1).

Experimental

1,4-Dimethyl-piperazine (0.57 g) and an excess of hydrobromic acid (0.95 g) were dissolved in methanol without any precipitation under stirring at room temperature. Colourless blocks of the title compound were obtained by slow evaporation of a methanol solution at room temperature over two days.

Refinement

H atoms were placed in calculated positions (N—H = 0.89 Å; C—H = 0.96 Å and 0.97 Å for Csp^3 atoms), assigned fixed U_{iso} values [1.5Ueq(Csp^3,N)] and allowed to ride.The H1WA and H1WB on the O1 were restrained with O—H =0.85 Å yielding O1—H1 = 0.8448 Å and O1 —H2 = 0.8440 Å, with U_{iso}(H) = 1.2 U_{iso}(O).

Computing details

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

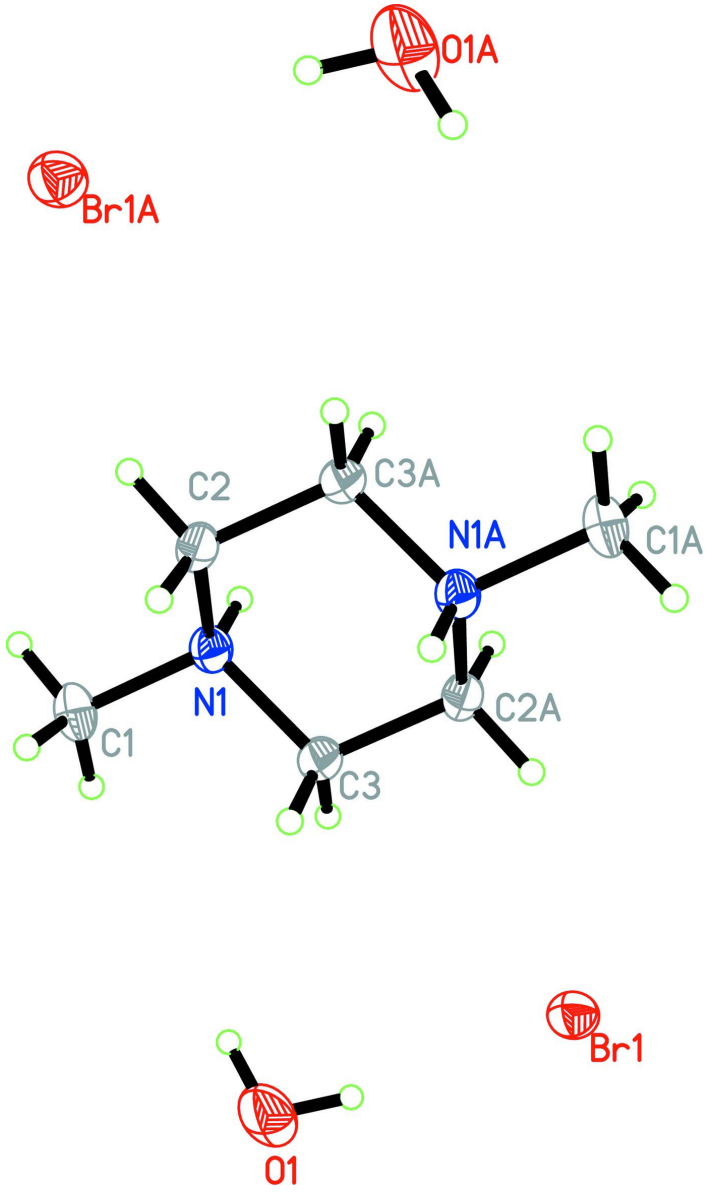

Figure 1

The molecular structure of the title compound with 30% probability displacement ellipsoids.

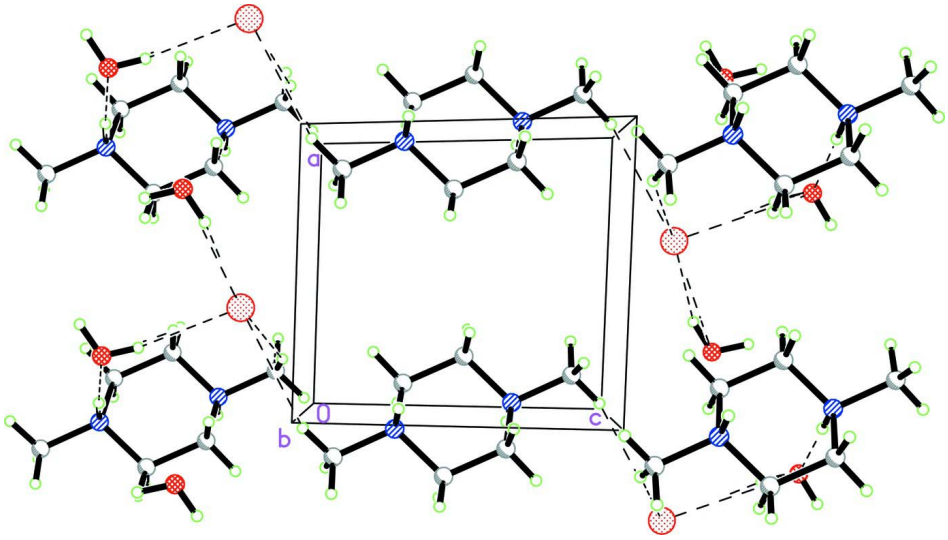

Figure 2

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

1,4-Dimethylpiperazine-1,4-diium dibromide dihydrate

Crystal data

$C_6H_{16}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$
$M_r = 312.06$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.2975\ (13)$ Å
$b = 7.0180\ (14)$ Å
$c = 7.2143\ (14)$ Å
$\alpha = 71.54\ (3)°$
$\beta = 86.62\ (3)°$
$\gamma = 85.54\ (3)°$
$V = 301.32\ (10)$ Å^3
$Z = 1$
$F(000) = 156$
$D_x = 1.720$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\theta = 3.1–27.5°$
$\mu = 6.70$ mm^{-1}
$T = 293$ K
Block, colorless
0.30 × 0.30 × 0.20 mm

Data collection

Rigaku SCXmini CCD 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)
$T_{min} = 0.154$, $T_{max} = 0.262$
3084 measured reflections
1370 independent reflections
1131 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -8\rightarrow 7$
$k = -9\rightarrow 9$
$l = -9\rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.169$
$S = 1.02$
1370 reflections
64 parameters
3 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

$w = 1/[\sigma^2(F_o^2) + (0.0897P)^2 + 0.3362P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.024$

$\Delta\rho_{max} = 0.80$ e Å^{-3}
$\Delta\rho_{min} = -1.12$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.0403 (8)	0.3473 (7)	0.6882 (7)	0.0319 (11)
H1A	−0.0362	0.2477	0.6754	0.038*
C2	−0.1103 (10)	0.5311 (10)	0.6709 (9)	0.0355 (13)
H2A	−0.2225	0.4973	0.7717	0.043*
H2B	−0.0331	0.6371	0.6904	0.043*
C3	0.2067 (9)	0.3955 (10)	0.5259 (9)	0.0346 (13)
H3A	0.2991	0.2759	0.5350	0.042*
H3B	0.2934	0.4979	0.5406	0.042*
C1	0.1381 (11)	0.2748 (10)	0.8819 (10)	0.0428 (15)
H1B	0.2564	0.1808	0.8788	0.064*
H1C	0.0342	0.2098	0.9796	0.064*
H1D	0.1872	0.3869	0.9129	0.064*
Br1	0.61718 (10)	0.21220 (10)	0.19573 (10)	0.0445 (3)
O1	0.7864 (11)	0.0695 (11)	0.6378 (10)	0.0737 (19)
H1WA	0.675 (9)	0.027 (18)	0.707 (10)	0.11 (5)*
H1WB	0.757 (12)	0.090 (12)	0.519 (4)	0.05 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.035 (3)	0.031 (2)	0.028 (3)	−0.003 (2)	−0.002 (2)	−0.007 (2)
C2	0.037 (3)	0.043 (3)	0.027 (3)	0.001 (3)	0.002 (2)	−0.012 (3)
C3	0.031 (3)	0.041 (3)	0.031 (3)	0.003 (2)	−0.001 (2)	−0.013 (3)
C1	0.056 (4)	0.043 (4)	0.028 (3)	0.000 (3)	−0.010 (3)	−0.008 (3)
Br1	0.0393 (4)	0.0481 (5)	0.0451 (5)	−0.0090 (3)	−0.0021 (3)	−0.0116 (3)
O1	0.080 (4)	0.084 (4)	0.055 (4)	−0.048 (4)	−0.012 (3)	−0.007 (3)

Geometric parameters (Å, º)

N1—C1	1.482 (8)	C3—H3A	0.9700
N1—C3	1.498 (8)	C3—H3B	0.9700
N1—C2	1.516 (8)	C1—H1B	0.9600
N1—H1A	0.9100	C1—H1C	0.9600
C2—C3^{i}	1.497 (9)	C1—H1D	0.9600

C2—H2A	0.9700	O1—H1WA	0.853 (10)
C2—H2B	0.9700	O1—H1WB	0.854 (10)
C3—C2^{i}	1.497 (9)		
C1—N1—C3	111.3 (5)	N1—C3—H3A	109.2
C1—N1—C2	111.1 (5)	C2^{i}—C3—H3A	109.2
C3—N1—C2	109.7 (5)	N1—C3—H3B	109.2
C1—N1—H1A	108.2	C2^{i}—C3—H3B	109.2
C3—N1—H1A	108.2	H3A—C3—H3B	107.9
C2—N1—H1A	108.2	N1—C1—H1B	109.5
C3^{i}—C2—N1	110.7 (5)	N1—C1—H1C	109.5
C3^{i}—C2—H2A	109.5	H1B—C1—H1C	109.5
N1—C2—H2A	109.5	N1—C1—H1D	109.5
C3^{i}—C2—H2B	109.5	H1B—C1—H1D	109.5
N1—C2—H2B	109.5	H1C—C1—H1D	109.5
H2A—C2—H2B	108.1	H1WA—O1—H1WB	107 (3)
N1—C3—C2^{i}	111.9 (5)		
C1—N1—C2—C3^{i}	179.4 (5)	C1—N1—C3—C2^{i}	180.0 (5)
C3—N1—C2—C3^{i}	56.0 (7)	C2—N1—C3—C2^{i}	−56.7 (7)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{ii}	0.91	1.83	2.737 (9)	174
O1—H1*WA*···Br1^{iii}	0.85 (6)	2.51 (9)	3.275 (7)	151 (11)
O1—H1*WB*···Br1	0.85 (3)	2.41 (4)	3.244 (7)	166 (7)

Symmetry codes: (ii) *x*−1, *y*, *z*; (iii) −*x*+1, −*y*, −*z*+1.