

3,3'-Dimethyl-1,1'-methylene-dimidazolium dibromide

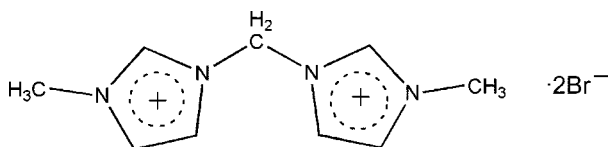
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.028; wR factor = 0.066; data-to-parameter ratio = 16.1.

In the crystal structure of the title compound, $\text{C}_9\text{H}_{14}\text{N}_4^{2+} \cdot 2\text{Br}^-$, the cation and anions have crystallographic mirror symmetry, with the mirror plane running through the central CH_2 group for the cation. The latter are stacked along the a axis, forming channels hosting the bromide anions. The crystal packing is stabilized by $\text{C}-\text{H} \cdots \text{Br}$ hydrogen-bonding interactions, generating a two-dimensional network.

Related literature

For related structures, see: Jin *et al.* (2007); Eicher *et al.* (2003).

Experimental

Crystal data

 $\text{C}_9\text{H}_{14}\text{N}_4^{2+} \cdot 2\text{Br}^-$
 $M_r = 338.06$
Monoclinic, $P2_1/m$
 $a = 4.7310$ (5) Å
 $b = 11.3861$ (12) Å $c = 11.8419$ (15) Å
 $\beta = 93.672$ (1)°
 $V = 636.59$ (12) Å³
 $Z = 2$
Mo $K\alpha$ radiation $\mu = 6.34$ mm⁻¹
 $T = 298$ K $0.32 \times 0.10 \times 0.07$ mm

Data collection

Bruker SMART diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.227$, $T_{\max} = 0.638$ 3349 measured reflections
1188 independent reflections
928 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.066$
 $S = 1.07$
1188 reflections74 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.63$ e Å⁻³
 $\Delta\rho_{\min} = -0.27$ e Å⁻³**Table 1**
Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{C4}-\text{H4} \cdots \text{Br1}^{\text{i}}$	0.93	2.84	3.724 (3)	158
$\text{C3}-\text{H3} \cdots \text{Br1}^{\text{ii}}$	0.93	2.81	3.699 (3)	160
$\text{C1}-\text{H1A} \cdots \text{Br1}^{\text{iii}}$	0.97	2.76	3.723 (4)	172
$\text{C2}-\text{H2} \cdots \text{Br2}^{\text{iv}}$	0.93	2.82	3.627 (3)	146
$\text{C1}-\text{H1B} \cdots \text{Br2}^{\text{iv}}$	0.97	2.81	3.652 (5)	146

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINTE* (Siemens, 1996); data reduction: *SAINTE*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2350).

References

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The title compound was synthesized as the precursor of a chelating N-heterocyclic carbene ligand, which can be generated by deprotonating the ring between the two N atoms of the two imidazolium cations. (Jin *Zhi*, 2007).

The structure consists of dimethylethylenediimidazolium cations and bromide anions (Fig. 1). The cation has crystallographically imposed mirror symmetry, with atom C1 located on a mirror plane. Both independent bromide anions also lie on a mirror plane. The C1—N1 bond length is 1.455 (4) Å, and the N1—C1—N1 bond angle is 111.0 (4)°. The C2—N1—C4 bond angle of 108.4 (3)° is similar to those observed in free imidazole (Eicher *Zhi*, 2003). The relative orientation of the imidazolium ring with respect to the other imidazolium ring can be described by the value of -95.4 (4)° of the C2—N1—C1—N1 torsion angle. In the crystal, the cations are stacked along the *a* axis forming channels that are occupied by the bromide anions (Fig. 2). Adjacent molecules are connected into a two-dimensional network through C—H...Br hydrogen interactions (Table 1).

A mixture of 1-methylimidazole (0.1 mol) and dichloromethane (0.05 mol) was reacted under nitrogen atmosphere with stirring at 350 K for 48 h. The resulting clear solution was evaporated under vacuum. Colourless crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate solution over a period of two weeks. (yield 83%) Anal. Calcd (%) for C₉H₁₄Br₂N₄ (Mr = 338.06): C, 32.03; H, 4.09; N, 16.62. Found (%): C, 31.95; H, 4.14; N, 16.57.

All H atoms were placed geometrically and treated as riding on their parent atoms, with C—H = 0.93–0.97 Å, and with $U_{iso}(H) = 1.2 U_{eq}(C)$ or $1.5 U_{eq}(C)$ for methyl H atoms.

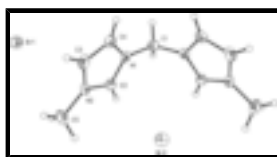


Fig. 1. The molecular structure of the compound, with atom labels and 50% probability displacement ellipsoids. Unlabelled atoms are related to labelled atoms by (x, 0.5-y, z)

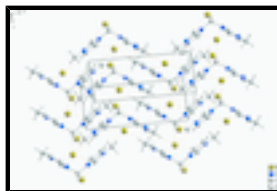


Fig. 2. Crystal packing of the compound, showing the two-dimensional network structure formed by C—H...Br hydrogen bonds (dashed lines).
