

2,2'-Dimethyl-3,3'-(*p*-phenylenedimethylene)-
diimidazol-1-ium dibromide

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In the centrosymmetric title compound, $C_{16}H_{20}N_4^{2+} \cdot Br_2^{-}$, the Br^- counter-ions form hydrogen bonds with protonated imidazole groups. Neighbouring cations are held together *via* weak $C-H \cdots Br$ hydrogen bonds and offset $\pi-\pi$ interactions, resulting in the formation of a three-dimensional structure.

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Key indicators

Single-crystal X-ray study

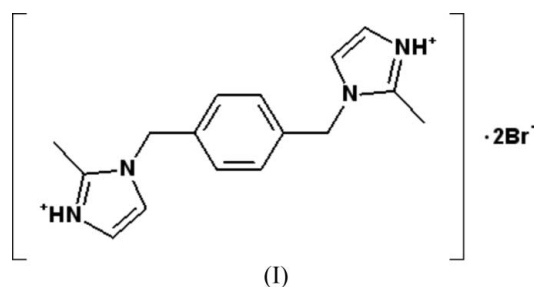
 $T = 100$ KMean $\sigma(C-C) = 0.003$ Å R factor = 0.025 wR factor = 0.064

Data-to-parameter ratio = 19.5

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Comment

The synthesis of metal complexes with flexible ditopic ligands, especially ligands with imidazole-type rings separated by an aromatic spacer, has attracted a great deal of interest because of the possibility of obtaining topologically interesting structures (Carlucci *et al.*, 2005; Liu, Hu *et al.*, 2001). Conformational flexibility is not only a feature of the ligand's reactions with metal salts, but also the generation of different supramolecular assemblies of salts and neutral ligand structures in the solid state. For example, bitmb [bitmb = 1,3-bis(imidazol-1-ylmethyl)-2,4,6-trimethylbenzene] adopts the *cis* conformation in its hexafluorophosphate salt (Liu, Su *et al.*, 2001), whereas neutral bix dihydrate [bix = 1,4-bis(imidazol-1-ylmethyl)benzene] (Abrahams *et al.*, 1998) and the perchlorate salt of bimp [bimp = 4,4'-bis(imidazol-1-ylmethyl)biphenyl] (Fei *et al.*, 2000) exhibit *trans* conformations.



In our studies of complexes generated from 1,4-bis(2-methylimidazol-1-ylmethyl)benzene (Dobrzańska, Lloyd *et al.*, 2005; Dobrzańska, Raubenheimer & Barbour, 2005), we have unexpectedly obtained single crystals of the dibromide salt, (I), of the ligand. The asymmetric unit comprises one half-cation and a Br^- counter-ion (Fig. 1). The cation is disposed about an inversion centre and from symmetry adopts a *trans* conformation with respect to the plane of the aromatic spacer. The cation is hydrogen-bonded in opposite directions *via* protonated imidazole rings to Br^- anions (Table 1). The two relatively weak $C5-H5A \cdots Br1^i$ and $C5-H5B \cdots Br1^{ii}$ hydrogen bonds (symmetry codes as listed in Table 1) generate a three-dimensional supramolecular assembly (Fig. 2) stabilized by offset $\pi-\pi$ interactions, with a centroid-

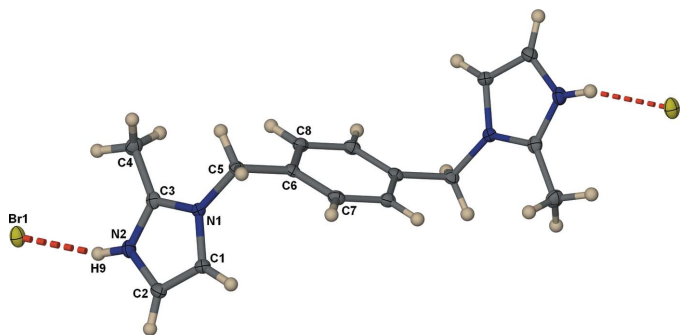


Figure 1
The structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms. N–H···Br hydrogen bonds are shown as dashed red lines. The unlabelled half of the asymmetric unit is related by the symmetry operation $(2 - x, 2 - y, 1 - z)$.

centroid distance between the two nearest C₆-aromatic ring planes of 3.65 Å (Fig. 3).

Experimental

α,α' -Dibromo-*p*-xylene was added to a methanolic solution of 1,4-bis(2-methylimidazol-1-ylmethyl)benzene in a 1:1 molar ratio and refluxed for 17 h at 343 K. Colourless crystals suitable for diffraction were obtained by slow evaporation of a methanolic solution of (I).

Crystal data

C ₁₆ H ₂₀ N ₄ ²⁺ ·2Br ⁻	$D_x = 1.674 \text{ Mg m}^{-3}$
$M_r = 428.18$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 3298 reflections
$a = 7.7378 (8) \text{ \AA}$	$\theta = 2.8\text{--}28.3^\circ$
$b = 16.0080 (16) \text{ \AA}$	$\mu = 4.77 \text{ mm}^{-1}$
$c = 7.2599 (7) \text{ \AA}$	$T = 100 (2) \text{ K}$
$\beta = 109.115 (2)^\circ$	Plate, colourless
$V = 849.68 (15) \text{ \AA}^3$	$0.46 \times 0.21 \times 0.03 \text{ mm}$
$Z = 2$	

Data collection

Bruker APEX CCD area-detector diffractometer	1966 independent reflections
ω scans	1803 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$R_{\text{int}} = 0.016$
$T_{\text{min}} = 0.218, T_{\text{max}} = 0.870$	$\theta_{\text{max}} = 28.3^\circ$
5257 measured reflections	$h = -9 \rightarrow 8$
	$k = -20 \rightarrow 20$
	$l = -9 \rightarrow 7$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 0.3603P]$
$R[F^2 > 2\sigma(F^2)] = 0.025$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.064$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.65 \text{ e \AA}^{-3}$
1966 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
101 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bond geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
N2–H9···Br1	0.88	2.37	3.214 (2)	162
C5–H5A···Br1 ⁱ	0.99	2.80	3.702 (2)	152
C5–H5B···Br1 ⁱⁱ	0.99	2.89	3.736 (2)	144

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

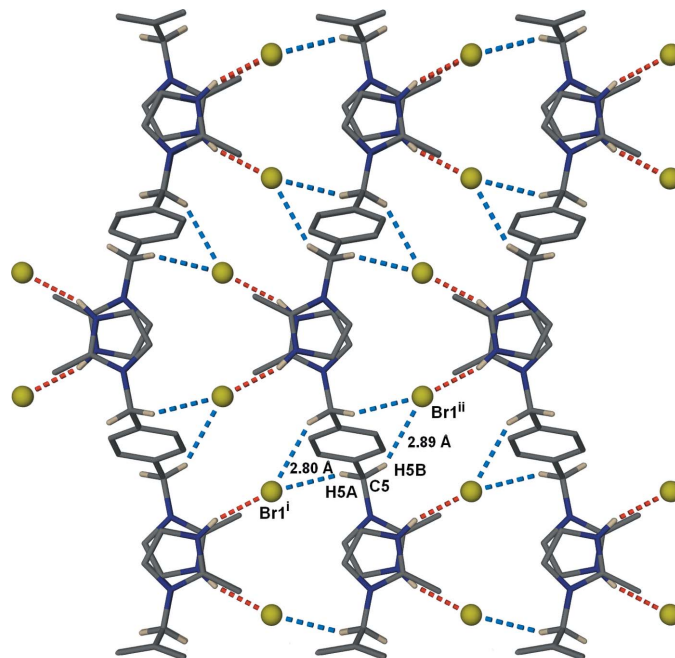


Figure 2

Packing diagram of (I), viewed along [001]. Hydrogen bonds are shown as dashed blue (C–H···Br) and red (N–H···Br) lines. H atoms not participating in hydrogen bonding have been omitted for clarity. Symmetry codes are as listed in Table 1.

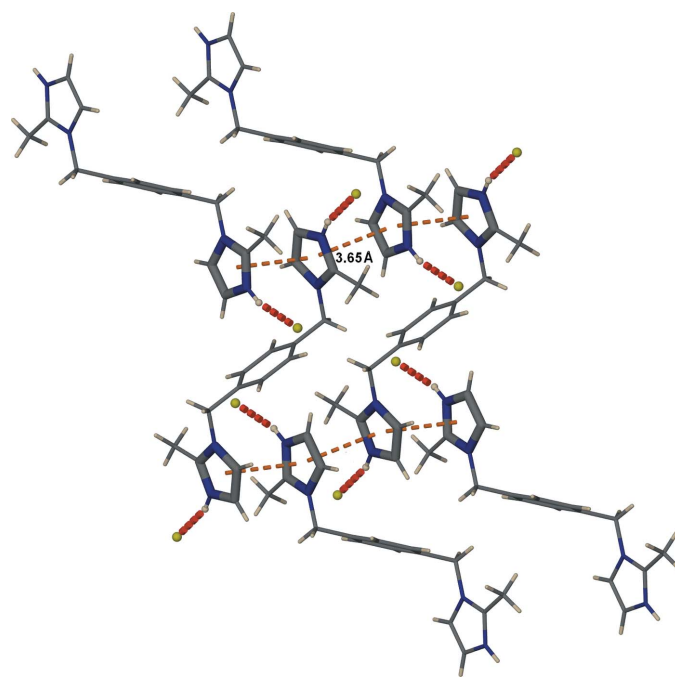


Figure 3

Capped-stick representation showing the π – π stacking geometry of (I). N–H···Br hydrogen bonds are shown as dashed red lines.

H atoms were positioned geometrically, with C–H = 0.95 (aromatic), 0.98 (methyl) and 0.99 Å (methylene), and N–H = 0.88 Å, and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ and $1.5U_{\text{eq}}(\text{methyl C})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Atwood & Barbour, 2003; Barbour, 2001); software used to prepare material for publication: *X-SEED*.

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