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# (Benzimidazol-3-ium-2-ylmethyl)(benzimidazol-2-ylmethyl)aminium sulfate

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**Key indicators**

Single-crystal X-ray study
*T* = 292 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.060
*wR* factor = 0.170
Data-to-parameter ratio = 12.6

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

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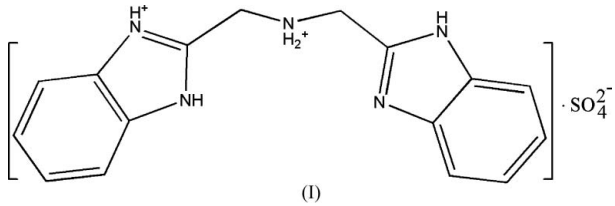
In the title crystal structure, $C_{16}H_{17}N_5^{2+}\cdot SO_4^{2-}$, cations and anions are linked *via* N—H···O hydrogen bonds, while ion pairs form extended one-dimensional chains through intermolecular N—H···N hydrogen bonds.

## Comment

The structures of several compounds containing more than one benzimidazole moiety have been reported recently, *e.g.* Qin *et al.* (2005), Yan *et al.* (2004), Qin *et al.* (2004) and Tarazon Navarro & McKee (2003). The reaction of bis(benzimidazol-2-ylmethyl)amine (IDB) and $(NH_4)_2Fe(SO_4)_2\cdot 7H_2O$ in ethanol unexpectedly produced the title compound, (I).




(I)

The title compound is shown in Fig.1 and selected bond lengths are given in Table 1. Both the amine group and one of the benzimdazole N atoms are protonated. Unlike in the earlier determination of bis(benzimidazol-2-yl-methyl)amine (Tarazon Navarro & McKee, 2003), no disorder is observed for the amine N or methylene C atoms in the title compound. This may be the result of the hydrogen bonding which is present in the title structure (Fig.2 and Table 2). In the crystal structure, π–π stacking interactions are also observed, where

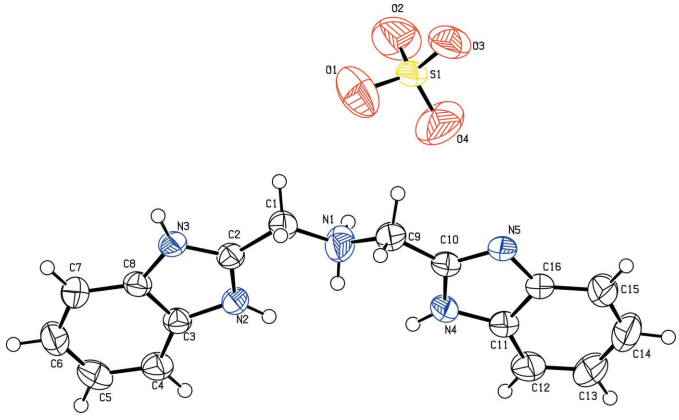



**Figure 1**
View of the molecular structure of the title compound, showing 50% probablity displacement ellipsoids.

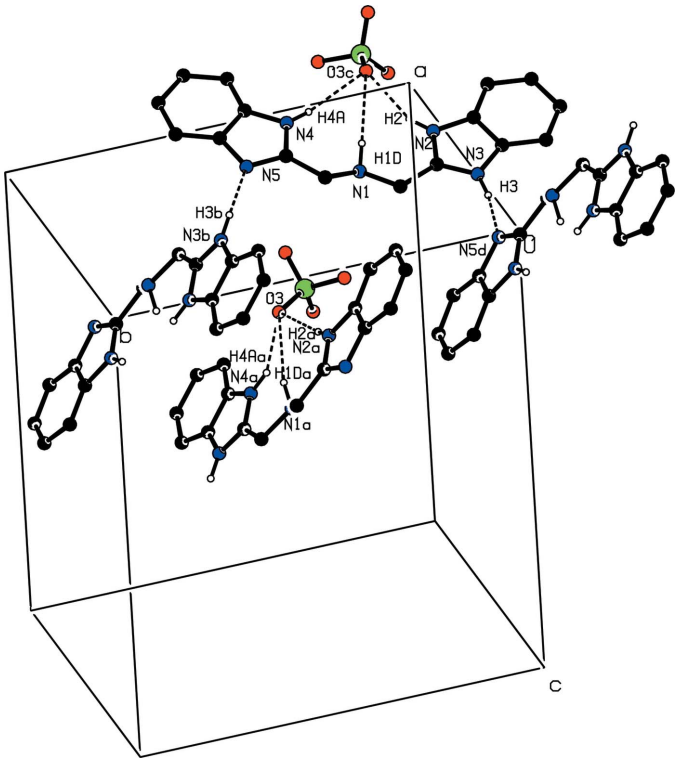



**Figure 2**
Plot of the crystal packing, with N—H···O and N—H···N hydrogen bonds shown as dashed lines. H atoms not involved in the hydrogen bonds have been omitted for clarity. Atoms labelled with the lower case suffixes are related by the symmetry codes: (*a*) $x, \frac{1}{2}-y, \frac{1}{2}-z$; (*b*) $2-x, \frac{1}{2}-y, \frac{1}{2}-z$; (*c*) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (*d*) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$.

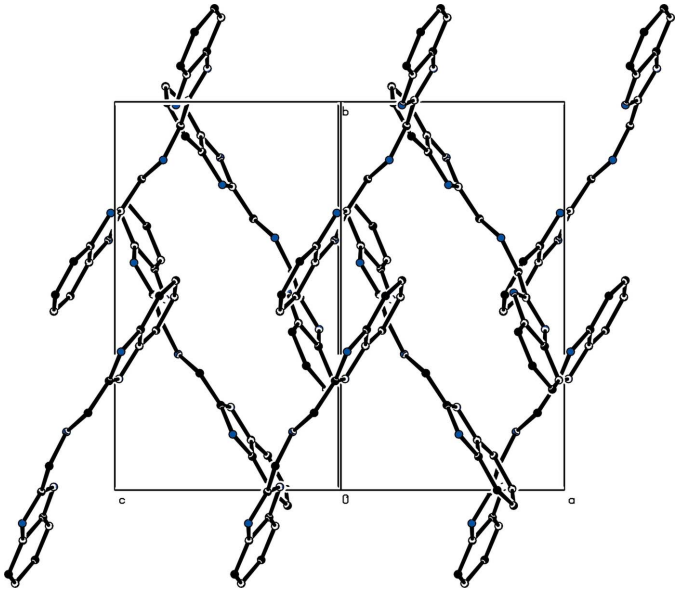



**Figure 3**
Packing diagram showing the $\pi$–$\pi$ stacking between symmetry-related benzimidizole rings. H atoms have been omitted for clarity.

parallel pairs of benzimidazole rings, related by inversion centers, are separated by a perpendicular distance of 3.42 Å (Fig.3).

## Experimental

Bis(benzimidazol-2-yl-methyl)amine (IDB) was prepared according to the method described by Adams *et al.* (1990). Compound (I) was synthesized by reaction of IDB (0.27 g, 1 mmol) and $(NH_4)_2Fe(SO_4)_2{\cdot}7H_2O$ (0.41 g, 1 mmol) in ethanol (20 ml) at 333 K for 2 h. The resulting soultion was filtered and single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of the filtrate at room temperature over a period of one week.

*Crystal data*

$C_{16}H_{17}N_5^{2+}{\cdot}SO_4^{2-}$
$M_r = 375.41$
Monoclinic, $P2_1/c$
$a = 8.4723\ (11)$ Å
$b = 13.7762\ (18)$ Å
$c = 14.8752\ (19)$ Å
$\beta = 103.150\ (2)^\circ$
$V = 1690.6\ (4)$ Å$^3$
$Z = 4$
$D_x = 1.475$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 3387 reflections
$\theta = 2.6$–$23.2^\circ$
$\mu = 0.23$ mm$^{-1}$
$T = 292\ (2)$ K
Block, yellow
0.30 × 0.20 × 0.18 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
$\omega$ scans
11984 measured reflections
2953 independent reflections
2305 reflections with $I > 2\sigma(I)$
$R_{int} = 0.089$
$\theta_{max} = 25.0^\circ$
$h = -10 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -17 \rightarrow 17$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.171$
$S = 1.06$
2953 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.099P)^2 + 0.3832P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.58$ e Å$^{-3}$
$\Delta\rho_{min} = -0.42$ e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| N3—C2 | 1.309 (3) | N5—C16 | 1.355 (3) |
| N3—C8 | 1.419 (3) | N2—C2 | 1.313 (3) |
| N4—C10 | 1.339 (3) | N2—C3 | 1.417 (3) |
| N4—C11 | 1.373 (4) | C1—N1 | 1.414 (4) |
| N5—C10 | 1.354 (3) | N1—C9 | 1.482 (4) |

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*D*···O3$^{i}$ | 0.90 | 2.57 | 3.443 (4) | 164 |
| N2—H2···O3$^{i}$ | 0.86 | 2.02 | 2.825 (3) | 156 |
| N4—H4*A*···O3$^{i}$ | 0.86 | 2.32 | 3.165 (4) | 168 |
| N3—H3···N5$^{ii}$ | 0.86 | 1.84 | 2.681 (3) | 165 |

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

All H atoms were placed in idealized positions (amine N—H = 0.96 Å, imine N—H = 0.86 Å, methylene C—H = 0.97 Å and aromatic C—H = 0.93 Å) and included in the refinement in riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}$(carrier atom).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics and publication material: *PLATON* (Spek, 2003).

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