Bis(benzimidazol-2-ylmethyl)amine–ethanol–water (2/1/2)

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.059
wR factor = 0.167
Data-to-parameter ratio = 14.1

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

In the asymmetric unit of the structure of the solvate of bis(benzimidazol-2-yl-methyl)amine (IDB), $2C_{16}H_{15}N_5 \cdot C_2H_6O \cdot 2H_2O$, there are two molecules of IDB, two uncoordinated water molecules and one ethanol solvent molecule. In the crystal structure, IDB, water and ethanol molecules are connected into a three-dimensional network *via* intermolecular N—H···O, O—H···N, O—H···O and C—H···π(arene) interactions.

Comment

In a continuation of our studies (Meng *et al.*, 2005) of the influence of solvents on the molecular and supramolecular structures of bis(benzimidazol-2-yl-methyl)amine (IDB), we report here the crystal structure of the title compound, (I).

(I)
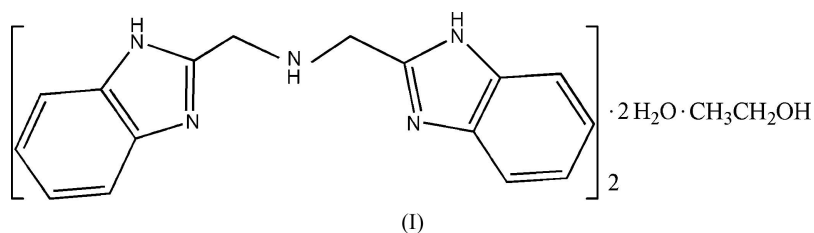
The asymmtric unit of (I) (Fig. 1) contains two molecules of IDB, two uncoordinated water molecules and one ethanol molecule. Both IDB molecules and one water molecule are disordered (see *Experimental*). The molecular geometric parameters of IDB are not significantly different from those of an earlier reported structure (Meng *et al.*, 2005), with the exception of the torsion angles of the atomic segment between the two benzimidazole units (Table 1).

The supramolecular aggregation in (I) is dominated by N—H···O, O—H···O and O—H···N hydrogen bonds, accompanied by C(or N)—H···π interactions, forming a three-dimensional structure (Table 2 and Fig. 2). Molecules are linked into a two-dimensional sheet in the *ab* plane by a combination of intermolecular hydrogen bonds and C(or N)—H···π interactions; O3—H3···N9 and N2—H2···O3 hydrogen bonds link adjacent two-dimensional layers into a three-dimensional network (Fig. 2).

Experimental

Bis(benzimidazol-2-yl-methyl)amine (IDB) was prepared according to the method described by Adams *et al.* (1990). IDB (0.27 g, 1 mmol) was dissolved in 10 ml 95% ethanol at 323 K. After slow cooling to room temperature, colorless crystals suitable for X-ray diffraction had collected at the bottom of the vessel.

Crystal data

$2C_{16}H_{15}N_5 \cdot C_2H_6O \cdot 2H_2O$
$M_r = 636.76$
Triclinic, $P\bar{1}$
$a = 8.6345 (8)$ Å
$b = 14.0418 (13)$ Å
$c = 14.2810 (13)$ Å
$\alpha = 92.724 (2)°$
$\beta = 104.845 (2)°$
$\gamma = 90.376 (2)°$

$V = 1671.5 (3)$ Å^3
$Z = 2$
$D_x = 1.265$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 292 (2)$ K
Block, colorless
$0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
14144 measured reflections

7160 independent reflections
4258 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 27.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.167$
$S = 1.03$
7160 reflections
509 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0783P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1
Selected torsion angles (°).

C2—C1—N1—C9	166.1 (7)	C18—C17—N6—C25	−166.3 (7)
C10—C9—N1—C1	179.2 (7)	C26—C25—N6—C17	130.9 (7)
C2—C1—N1′—C9′	98.3 (7)	C18—C17—N6′—C25	162.9 (7)
C10—C9′—N1′—C1	−169.3 (7)	C26—C25—N6′—C17	166.4 (7)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1′—H1′$\cdots$N3	0.861 (10)	2.44 (4)	3.097 (10)	134 (4)
N2—H2$\cdots$O3	0.860 (9)	1.909 (11)	2.756 (2)	168 (2)
N6′—H6′$\cdots$O1	0.99 (8)	2.597 (16)	3.352 (19)	133 (8)
N8—H8$\cdots$O1	0.874 (9)	2.040 (11)	2.902 (2)	169 (2)
N10—H10$\cdots$O1	0.864 (9)	2.159 (12)	2.984 (2)	159 (2)
O2—H2A$\cdots$N7	0.830 (10)	2.041 (13)	2.862 (3)	170 (3)
O2′—H2D$\cdots$N7	0.823 (10)	1.881 (16)	2.704 (11)	178 (16)
O3—H3$\cdots$N9^i	0.831 (10)	1.911 (13)	2.732 (2)	169 (4)
N1′—H1′$\cdots$O2′^{ii}	0.861 (10)	2.35 (10)	3.045 (15)	138 (12)
N5—H5A$\cdots$O2′^{ii}	0.847 (10)	1.958 (18)	2.758 (12)	157 (2)
N5—H5A$\cdots$O2^{ii}	0.847 (10)	2.060 (11)	2.907 (3)	178 (2)
O1—H1E$\cdots$N4^{iii}	0.833 (10)	2.007 (11)	2.836 (3)	173 (3)
O1—H1F$\cdots$N1′^{iv}	0.834 (10)	2.22 (2)	2.835 (10)	131 (2)
O1—H1F$\cdots$O2′^{v}	0.834 (10)	2.231 (19)	2.941 (11)	143 (2)
O1—H1F$\cdots$N1^{iv}	0.834 (10)	2.616 (18)	3.347 (5)	147 (2)
O2—H2B$\cdots$N3^{ii}	0.836 (10)	2.046 (11)	2.881 (3)	176 (4)
O2′—H2C$\cdots$N5^{ii}	0.822 (10)	2.31 (13)	2.758 (12)	115 (12)
N1—H1$\cdots$Cg1^{vi}	0.862 (10)	2.91 (2)	3.632 (5)	143 (3)
C9′—H9D$\cdots$Cg1^{vi}	0.97	2.56	3.485 (11)	159
C30—H30$\cdots$Cg1^{iv}	0.93	2.91	3.661 (3)	138
C6—H6$\cdots$Cg2^{vii}	0.93	2.91	3.661 (3)	139
C7—H7$\cdots$Cg3^{vii}	0.93	2.93	3.758 (3)	149
C15—H15$\cdots$Cg4^{vii}	0.93	3.00	3.713 (3)	135
C23—H23$\cdots$Cg5^{iv}	0.93	2.88	3.600 (3)	136

Symmetry codes: (i) $x + 1, y, z$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 1, -y, -z + 1$; (iv) $x, y, z + 1$; (v) $-x + 1, -y + 1, -z + 2$; (vi) $-x + 2, -y, -z$; (vii) $x + 1, y, z - 1$.
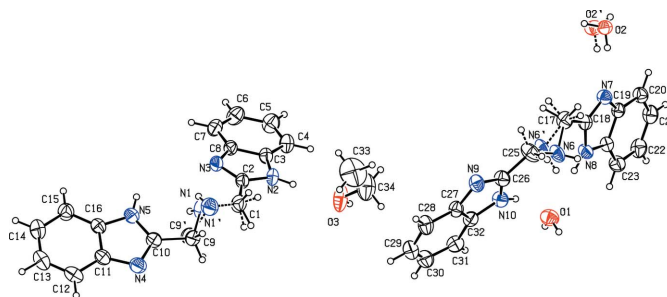

Figure 1
The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids. Dashed lines indicate the minor disorder components.
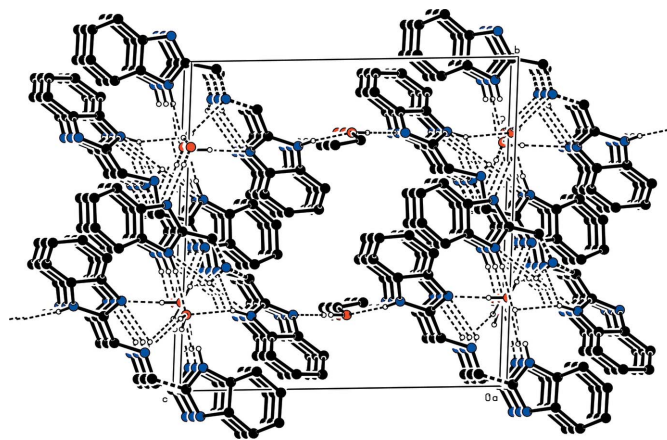

Figure 2
Part of the crystal structure of (I), showing the formation of a three-dimensional network. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Hydrogen bonds are shown as dashed lines.

Both IDB molecules are disordered. The disorder corresponds to two orientations of the –CH$_2$–NH–CH$_2$ segments between benzimidazole units in each molecule. In addition, one of the uncoordinated water molecules is disordered over two sites. Bond distances in the disordered groups were constrained using *DFIX* and *SADI* commands (*SHELXL97*; Sheldrick, 1997). The site-occupancy factors for disordered components were intially refined but were eventually fixed at 0.72/0.28 for N1/N1′ and C9/C9′, 0.74/0.26 for N6/N6′ and 0.79/0.21 for O2/O2′.

H atoms bonded to C atoms were assigned C—H distances of 0.93 (aromatic), 0.97 (CH$_2$) or 0.96 Å (CH$_3$), with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). H atoms bonded to N atoms were located in difference maps and were refined with U_{iso}(H) = 1.2U_{eq}(N). The H atoms of the water molecules and hydroxyl group were also located in a difference Fourier map and were refined with the following restraints; O—H = 0.82 (1) Å, and H$\cdots$H = 1.39 (1) Å, with U_{iso}(H) = 1.2U_{eq}(O).

There is a closer than normal contact between two H atoms, *viz.* H5A$\cdots$H2C of 1.47 Å. H2C is bonded to the minor component of a disordered uncoordinated water O atom and constitutes *ca* 0.21 of an H atom. There may be some discrepancy in the position of this H atom

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to

solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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