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1,1′-(Propane-1,3-diyl)bis[2-(methylsulfanyl)-1*H*-benzimidazole] dihydrate

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Key indicators

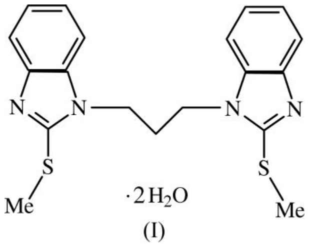
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.119
Data-to-parameter ratio = 17.4

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

In the title compound, $C_{19}H_{20}N_4S_2{\cdot}2H_2O$, the two 1*H*-benzimidazole groups are bridged by a propane chain; the dihedral angle between the benzimidazole ring systems is 74.61 (3)°. The organic molecules and solvent water molecules form O—H···N hydrogen bonds. There are also O—H···O, C—H···S, C—H···π and π–π interactions in the crystal structure.

Comment

The benzimidazole ring system has an interesting chemistry, and it is an effective pharmacophore in medicinal chemistry. Bis-benzimidazoles have potent activity against a number of microorganisms, including those that lead to AIDS-related infections (Bell *et al.*, 1993). These compounds bind to DNA in AT-rich sequences. Recently, drugs derived from benzimidazole have received much attention owing to the fact that the benzimidazole residue is a constituent of vitamin B_{12} (Skalitzky *et al.*, 2003), which supports their potential use as therapeutics (Hauel *et al.*, 2002; Valdez *et al.*, 2002). Benzimidazole, sometimes called 1,3-dideazapurine, and its derivatives can serve as model compounds for purine due to the structural similarity (Seela & Wenzel, 1995; Moreno *et al.*, 2004). In this paper, we report the crystal structure of the title compound, (I).



(I)

The asymmetric unit of (I) is shown Fig. 1. The organic molecule has two 1*H*-benzimidazole groups which are linked by a propane chain. The dihedral angles between benzimidazole plane *A* (C2–C7/N1/C1/N2), propane plane *B* (C8–C10) and the other benzimidazole plane *C* (C12–C17/N3/C11/N4) are *A*/*B* = 85.66 (12), *A*/*C* = 74.61 (3) and *B*/*C* = 67.80 (17)°. The geometric parameters for (I) agree well with those reported for other structures of bis(benzimidazole)s with rigid two-carbon bridges (Stibrany *et al.*, 2005). In particular, in the N—C—N fragments, the C1—N1 and C11—N4 distances are *ca* 0.05 Å shorter than the C1—N2 and C11—N3 distances (Table 1), consistent with the partial double-bond character of the former bonds.

In (I), the organic molecules are linked by solvent water molecules, forming O—H···N hydrogen bonds (Table 2) in

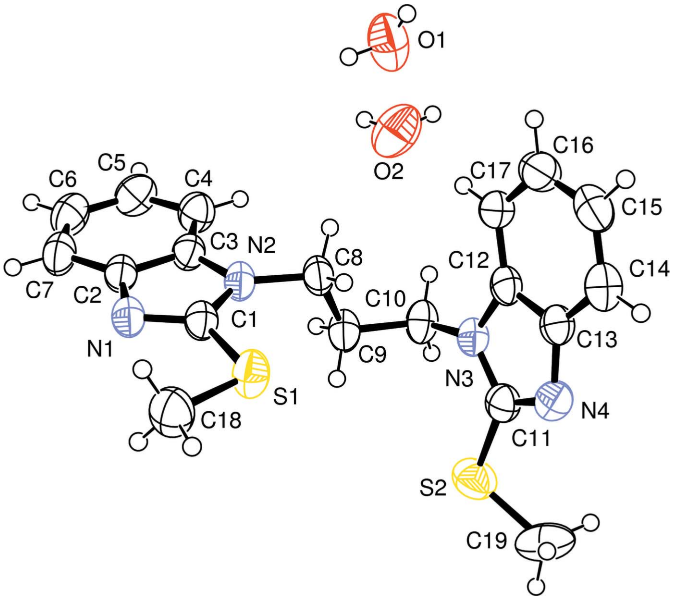


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

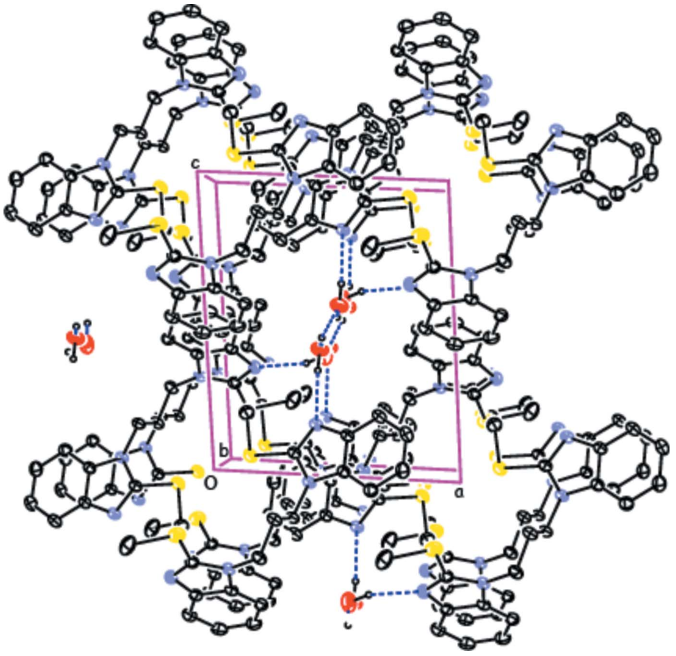


Figure 2
The crystal structure of (I). H atoms have been omitted unless they are involved in hydrogen bonds (dashed lines).

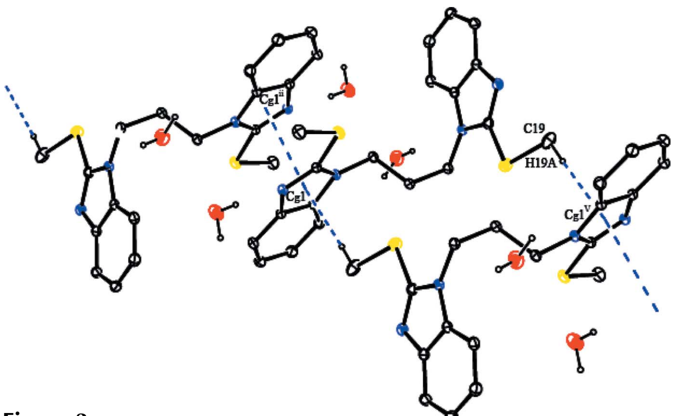


Figure 3
Intermolecular π–π and C—H$\cdots\pi$ interactions (dashed lines). Other H atoms have been omitted. [Symmetry codes: (ii) $1 - x, -y, -z$; (v) $2 - x, 1 - y, -z$.]

the crystal structure. As a result of these interactions, the structure exhibits channels of cavities passing through the centre of the unit cell and parallel to the *b* axis, with a mean diameter of 7.1807 (6) Å (Fig. 2). In addition, the other columns of cavity lie at the edges of the unit cell parallel to the *b* axis. There are also O—H$\cdots$O hydrogen bonds between water molecules, intramolecular C—H$\cdots$S, intermolecular C—H$\cdots\pi$ and π–π interactions in the crystal structure (Fig. 3). The imidazole rings are oriented in such a way that the perpendicular distance between the the *Cg*1 (N1/C1/N2/C3/C2) and $Cg1^{ii}$ rings is 3.478 Å, with the ring centroids separated by 3.5346 (11) Å [symmetry code: (ii) $1 - x, -y, -z$]. A C—H$\cdots\pi$ interaction is observed between *Cg*1 and the C19 methyl group: H19*A* $\cdots$ $Cg1^{v}$ = 2.5833 Å, C19—H19*A*$\cdots Cg1^{v}$ = 175.1° [symmetry code: (v) $2 - x, 1 - y, -z$].

Experimental

To a mixture of 2-methylthio-1*H*-benzimidazole (0.91 g, 3.14 mmol) and finely powdered NaOH (0.5 g, 12.5 mmol) in DMSO (7 ml) was added dropwise 1,3-dibromopropane (0.35 ml, 3.45 mmol). The resulting solution was stirred at 308–313 K for 1 h. Water was then added to the reaction mixture, and the solid that precipitated was collected and recrystallized from ethanol (yield 85%, m.p. 391–393 K).

Crystal data

$C_{19}H_{20}N_4S_2\cdot 2H_2O$
$M_r = 404.56$
Triclinic, $P\bar{1}$
$a = 9.2655$ (7) Å
$b = 9.9754$ (7) Å
$c = 11.2980$ (8) Å
$\alpha = 102.125$ (6)°
$\beta = 93.415$ (6)°
$\gamma = 96.443$ (6)°
$V = 1010.78$ (13) Å^3
$Z = 2$
$D_x = 1.329$ Mg m^{-3}
Mo *K*α radiation
$\mu = 0.28$ mm^{-1}
$T = 296$ K
Prism, colorless
0.57 × 0.36 × 0.18 mm

Data collection

Stoe IPDS-2 area-detector diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.880$, $T_{max} = 0.958$
19069 measured reflections
4506 independent reflections
3322 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 27.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.119$
$S = 1.04$
4506 reflections
259 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 0.0747P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}

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Table 1

Selected geometric parameters (Å, °).

S1—C1	1.7319 (19)	N2—C8	1.457 (2)
S2—C11	1.738 (2)	N3—C11	1.363 (2)
N1—C1	1.318 (2)	N3—C10	1.457 (2)
N2—C1	1.365 (2)	N4—C11	1.313 (3)
C18—S1—C1—N1	−6.5 (2)	C10—N3—C11—N4	176.71 (16)
N2—C8—C9—C10	174.45 (16)	C19—S2—C11—N4	−14.5 (2)
C8—C9—C10—N3	66.1 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O2—H4*W*⋯N4^{i}	0.83 (3)	2.05 (3)	2.851 (3)	162 (5)
O1—H1*W*⋯N1^{ii}	0.820 (16)	2.088 (18)	2.905 (2)	175 (3)
O1—H2*W*⋯O1^{iii}	0.82 (4)	2.10 (5)	2.905 (3)	167 (7)
O2—H3*W*⋯O2^{iv}	0.83 (2)	2.25 (4)	2.922 (4)	139 (4)
C8—H8*A*⋯S1	0.97	2.72	3.1284 (19)	106
C10—H10*A*⋯S2	0.97	2.79	3.138 (2)	102

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

Water H atoms were located in difference maps and refined isotropically with bond restraints O—H = 0.83 (2) Å. C-bound H atoms were positioned geometrically and treated as riding, with C—H = 0.93–0.97 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) or 1.5$U_{\rm eq}$(methyl C).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Bell, C. A., Dykstra, C. C., Naimen, N. A., Cory, M., Fairley, T. A. & Tidwell, R. R. (1993). *Antimicrob. Agents Chemother.* **37**, 2668–2673.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Hauel, N. H., Nar, H., Prirpke, H., Ries, U., Stassen, J. M. & Wienen, W. (2002). *J. Med. Chem.* **45**, 1757–1766.

Moreno, M. J. S., Botello, A. F., Coca, R. B. G., Griesser, R., Ochocki, J., Kotynski, A., Gutierrez, J. N., Moreno, V. & Sigel, H. (2004). *Inorg. Chem.* **43**, 1311–1322.

Seela, F. & Wenzel, T. (1995). *Helv. Chim. Acta*, **78**, 833–846.

Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

Skalitzky, D. J., Marakovits, J. T., Maegley, K. A., Ekker, A., Yu, X.-H., Hostomsky, Z., Webber, S. E., Eastman, B. W., Almassy, R., Li, J., Curtin, N. J., Newell, D. R., Calvert, A. H., Griffin, R. J. & Golding, B. T. (2003). *J. Med. Chem.* **46**, 210–213.

Stibrany, R. T., Schugar, H. J. & Potenza, J. A. (2005). *Acta Cryst.* C**61**, o354–o357.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

Valdez, J., Cedillo, R., Camos, A. H., Yepez, L., Luis, F. H., Vazquez, G. N., Tapia, A., Cortes, R., Hernandez, M. & Castillo, R. (2002). *Bioorg. Med. Chem. Lett.* **12**, 2221–2224.

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