Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(2*E*)-*N*,*N*′-Bis(pyridin-4-ylmethyl)but-2-ene-diamide dihydrate

Gareth O. Lloyd

Department of Chemistry, University of Stellenbosch, Private Bag X1, Matieland, South Africa

Correspondence e-mail: gol@sun.ac.za

Received 15 March 2005
Accepted 30 March 2005
Online 9 April 2005

Key indicators

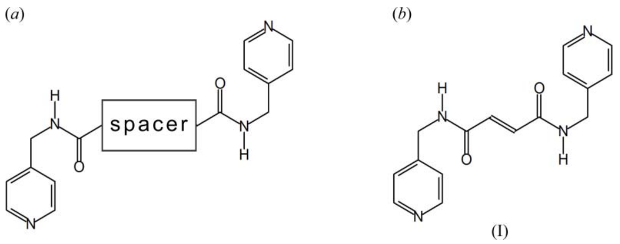
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.111
Data-to-parameter ratio = 17.4

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

In the centrosymmetric title compound, $C_{16}H_{16}N_4O_2\cdot2H_2O$, the water molecules donate two hydrogen bonds and accept one each, with O···O and O···N distances of 2.7498 (13) and 2.8152 (13) Å, respectively, and with an N···O distance of 2.7368 (13) Å. In the crystal structure, molecular channels are formed which are are occupied by water.

Comment

Pursuant to our studies of crystalline hydrates as well as supramolecular systems that contain molecular cavities or channels in addition to biologically relevant functional groups (*e.g.* amide groups and aromatic rings), the title compound, (I), was crystallized from a water-doped solvent in the hope of including water in the crystal structure (Atwood *et al.*, 2001; Barbour *et al.*, 1998; Kannan *et al.*, 2003; Lloyd *et al.*, 2005; Orr *et al.*, 1998).



Compound (I) is a member of a series of compounds that we have synthesized which conform to the general molecular structure (*a*) shown in the scheme. The spacer unit used for compound (I) is a *trans* C═C double bond which constrains the molecule into an elongated S shape (Fig. 1 and part *b* in scheme). Compound (I) can be considered to possess three planes, two formed by the pyridin-4-ylmethyl groups and the

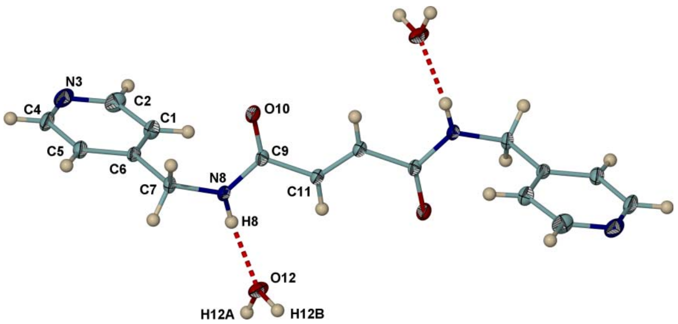


Figure 1
The molecular structure of (I), showing atom labels and 50% probability ellipsoids for non-H atoms. The dashed lines indicate hydrogen bonds and unlabelled atoms are related by the symmetry operator $(2 - x, -y, 1 - z)$.

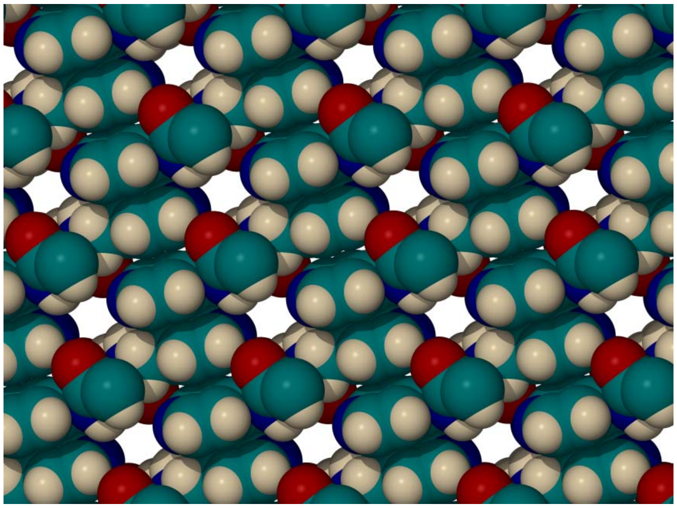


Figure 2
The molecular channels present in the crystal structure. Atoms are shown with van der Waals radii and water molecules have been removed to show the channels.

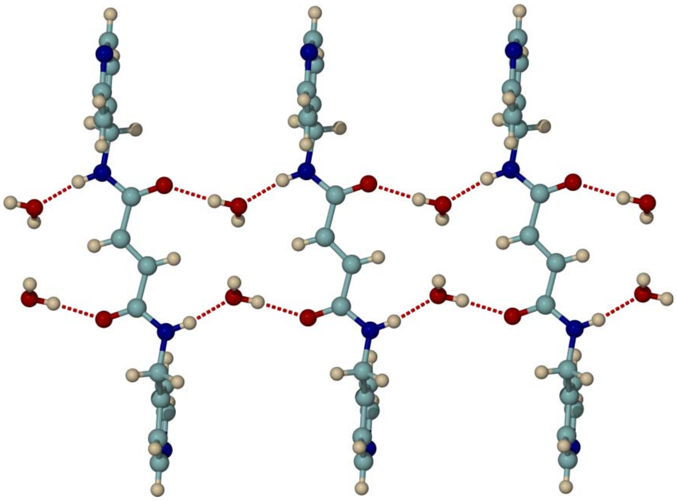


Figure 3
The 'β sheets' formed by molecules in (I). Hydrogen bonds are shown as dashed lines.

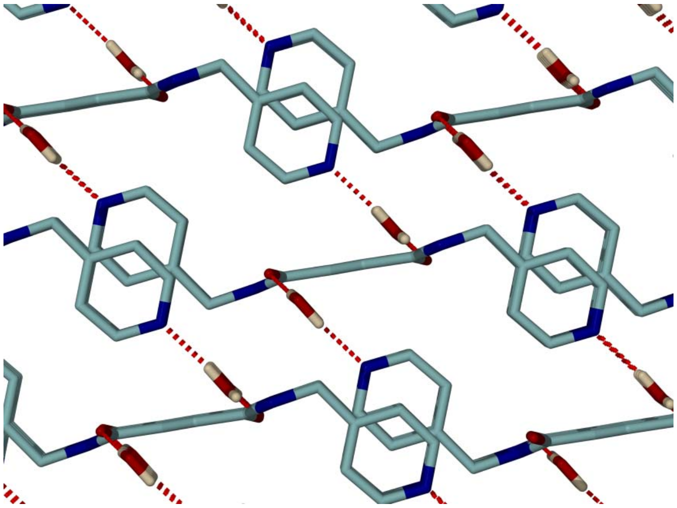


Figure 4
The hydrogen bonding (shown as dashed lines) and offset π–π interactions between 'β sheets'. Non-water H atoms have been omitted for clarity.

other by the amide groups and double bond. The r.m.s. deviation for the atoms in the pyridin-4-ylmethyl plane (N3/C4/C5/C6/C7/C1/C2) is 0.0034 Å and for those of the amides/double-bond plane (N8/C9/O10/C11/C11^{j}/O10^{j}/C9^{j}/N8^{j}) it is 0.0362 Å [symmetry code: (j) $2 - x, -y, 1 - z$]. The dihedral angle between the pyridin-4-ylmethyl and amides/double-bond planes is 81.10 (4)°. The coplanarity of the central atoms is further evidenced by the four torsion angles of the amides/double-bond plane: N8—C9—O10—C11 [178.34 (2)°], N8—C9—C11—C11^{j} [−171.1 (1)°], C9—C11—C11^{j}—C9^{j} (180.0°) and O10—C9—C11—C11^{j} [7.4 (2)°]. Water has been incorporated into (I) and forms two donating hydrogen bonds, O12—H12*B*···N3^{iii} and O12—H12*A*···O10^{ii} (see Table 1 for symmetry codes), and one accepting hydrogen bond, N8—H8···O12 (Table 1). In the crystal structure of (I), water molecules are located in channels (Fig. 2). Amide groups are well known to form β sheets in proteins (Sasaki & Liederman, 1996). A similar motif is observed in the present structure. The water molecules interrupt hydrogen bonding between the amide groups by donating a hydrogen bond and accepting another. Fig. 3 illustrates the 'β-sheet' formation. The 'β sheets' are stacked by means of the remaining water hydrogen bonds to the pyridyl groups, and by offset π–π interactions between pyridyl groups (centroid-to-centroid distance of 3.920 Å) (Fig. 4).

Experimental

Compound (I) was synthesized by the reaction of 4-aminomethylpyridine with fumaryl dichloride in a 2:1 molar ratio. Crystals suitable for single-crystal X-ray diffraction analysis were grown from a concentrated solution of (I) in a dimethylformamide–water (5:1) mixture. Compound (I) is soluble in most polar solvents, including water, although the best single crystals were grown from a dimethylformamide/water mixture.

Crystal data

$C_{16}H_{16}N_4O_2 \cdot 2H_2O$
$M_r = 332.36$
Triclinic, $P\overline{1}$
$a = 6.7867$ (9) Å
$b = 7.0884$ (9) Å
$c = 9.5620$ (13) Å
$\alpha = 107.597$ (2)°
$\beta = 101.854$ (2)°
$\gamma = 97.474$ (2)°
$V = 419.92$ (10) Å^3

$Z = 1$
$D_x = 1.314$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4737 reflections
$\theta = 3.1$–28.3°
$\mu = 0.10$ mm^{-1}
$T = 100$ (2) K
Block, colourless
0.25 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997; Blessing, 1995)
$T_{\min} = 0.976$, $T_{\max} = 0.986$

4737 measured reflections
1927 independent reflections
1776 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 28.3$°
$h = -9 \rightarrow 8$
$k = -9 \rightarrow 9$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.1309P]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.111$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.03$	$\Delta\rho_{\max} = 0.32$ e Å^{-3}
1927 reflections	$\Delta\rho_{\min} = -0.29$ e Å^{-3}
111 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N8—H8···O12	0.88	1.89	2.7368 (13)	161
O12—H12A···O10$^{\rm i}$	0.85	1.91	2.7498 (13)	170
O12—H12B···N3$^{\rm ii}$	0.89	1.93	2.8152 (13)	173

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

All non-water H atoms were positioned geometrically (C—H = 0.95 and 0.99 Å and N—H = 0.88 Å) and constrained to ride on their parent atoms; $U_{\rm iso}$(H) values were set at 1.2 times $U_{\rm eq}$(C,N). Water H atoms were constrained geometrically to 0.90 (5) Å and the $U_{\rm iso}$(H) values were refined independently.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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*.

The author thanks the National Research Foundation of South Africa for financial support.

References

Atwood, J. L. & Barbour, L. J. (2003). *Cryst. Growth Des.* **3**, 3–8.

Atwood, J. L., Barbour, L. J., Ness, T. J., Raston, P. L. & Raston, C. L. (2001). *J. Am. Chem. Soc.* **123**, 7192–7193.

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.

Barbour, L. J., Orr, G. W. & Atwood, J. L. (1998). *Chem. Commun.* **393**, 671–672.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Bruker (2001). *SMART*. Version 5.625. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2002). *SAINT*. Version 6.36a. Bruker AXS Inc., Madison, Wisconsin, USA.

Kannan, R., Katti, K. K., Barbour, L. J., Barnes, C. L. & Katti, K. V. (2003). *J. Am. Chem. Soc.* **125**, 6955–6961.

Lloyd, G. O., Atwood, J. L. & Barbour, L. J. (2005). *Chem. Commun.* pp. 1845–1847.

Orr, G. W., Barbour, L. J. & Atwood, J. L. (1998). Nature, **10**, 859–860.

Sasaki, T. & Liederman, M. (1996). *Comprehensive Supramolecular Chemistry*, edited by A. L. Atwood, J. E. D. Davis, D. D. MacNicol and F. Vögtle, Vol. 4, pp. 193–242. Oxford: Elsevier.

Sheldrick, G. M. (1997). *SADABS* (Version 2.05), *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.