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3-Pyridylmethyl urea dihydrate

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The geometrical parameters of the title compound, $C_{13}H_{14}N_4O\cdot2H_2O$, are normal. The carbonyl group occupies a twofold symmetry axis to generate the complete 3-pyridylmethyl urea molecule. The crystal packing is defined by hydrogen bonding involving the water molecule, as well as $\pi\cdots\pi$ stacking and weak C—H$\cdots\pi$ interactions.

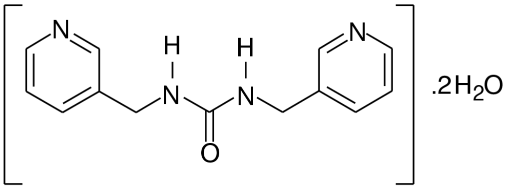
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.090
Data-to-parameter ratio = 12.0

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

Comment

The geometrical parameters of the title compound, (I) (Fig. 1), are normal and comparable to those reported for the silver complex of the same ligand by Schauer *et al.* (1997). In (I), the carbonyl group occupies a twofold symmetry axis to generate the complete molecule. The same situation occurs in several of the related ureylenedicarboxylic acid phases (Zhao *et al.*, 1993) which are of interest for their supramolecular packing motifs.



(I)

The crystal packing for (I) (Fig. 2) is defined by hydrogen bonding involving the water molecule as well as $\pi\cdots\pi$ stacking involving pairs of aromatic moieties and weak C—H$\cdots\pi$ interactions. The hydrogen-bonding motif (Fig. 3) involves stacks of 3-pyridylmethyl urea and water molecules propagating in the [010] direction. The water molecule serves to bridge adjacent 3-pyridylmethyl urea molecules by acting as both an N1—H1$\cdots$O2 acceptor and an O2—H3$\cdots$O1 donor (Table 2). Crystal symmetry dictates that adjacent 3-pyridylmethyl urea molecules are linked by a pair of water molecules (Fig. 3) related by the twofold symmetry axis. Additionally, an O2—H2$\cdots$N2 hydrogen bond to a pyridyl-N atom acceptor serves as a crosslink to an adjacent 3-pyridylmethyl urea/water stack.

Crystal symmetry generates a distinctive motif (Fig. 4) of C5—H51$\cdots\pi\cdots\pi\cdots$H51—C5 interactions. Thus each pyridyl moiety interacts with a similar species by $\pi\cdots\pi$ stacking on one ring face, and accepts a weak C—H$\cdots\pi$ interaction on the other. The separation of adjacent pyridyl ring centroids is 3.698 Å, with the ring centroid given by the coordinates 0.2581 0.4220 0.3877 (Spek, 1990). These interactions are orientated in the (011) plane.

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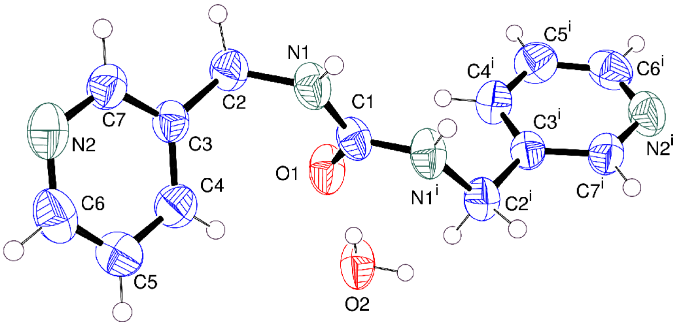

Figure 1
Molecular structure of (I); 50% displacement ellipsoids, circles of arbitrary radius for H atoms. Symmetry code: (i) $-x, y, -z + \frac{1}{2}$.

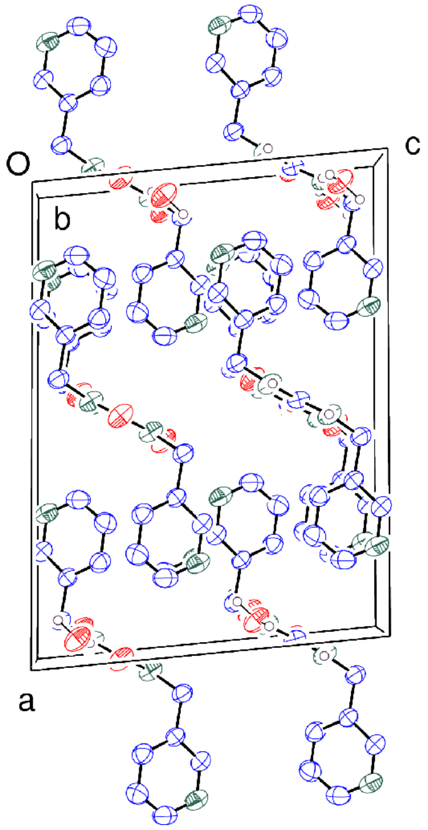

Figure 2
Packing diagram for (I) with C—H H atoms omitted for clarity. Atom colours as in Fig. 1; 50% displacement ellipsoids.

Experimental

The title compound was recrystallized from hot water.

Crystal data

$C_{13}N_4OH_{14}\cdot 2H_2O$
$M_r = 278.31$
Monoclinic, $C2/c$
$a = 16.5784\ (11)$ Å
$b = 7.1077\ (5)$ Å
$c = 12.1751\ (8)$ Å
$\beta = 95.475\ (2)^\circ$
$V = 1428.10\ (17)$ Å^3
$Z = 4$
$D_x = 1.294$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2260 reflections
$\theta = 2.5$–25.0°
$\mu = 0.09$ mm^{-1}
$T = 298\ (2)$ K
Lump, colourless
0.48 × 0.24 × 0.24 mm

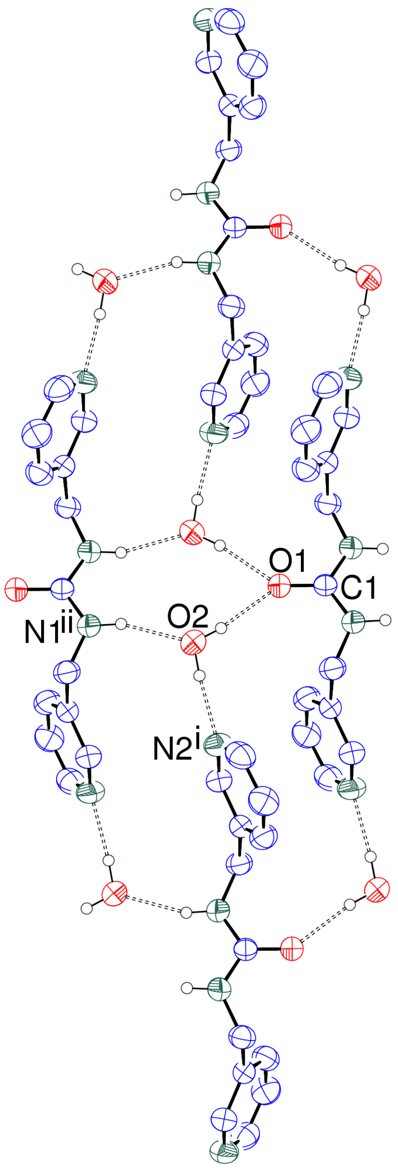

Figure 3
Detail showing the hydrogen-bonding scheme in (I) with H bonds indicated by dashed lines. Atom colours as in Fig. 1; 50% displacement ellipsoids. Symmetry codes: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $-x, y - 1, \frac{1}{2} - z$.

Data collection

Bruker SMART1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999*a*)
$T_{min} = 0.803$, $T_{max} = 0.928$
4018 measured reflections
1260 independent reflections
1047 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.0^\circ$
$h = -14 \rightarrow 19$
$k = -8 \rightarrow 8$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.090$
$S = 1.06$
1260 reflections
105 parameters
H-atom treatment, see text
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.2493P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.11$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0129 (16)

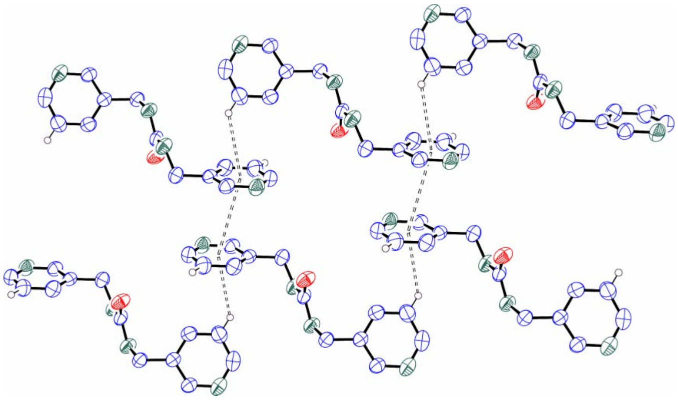

Figure 4
Detail showing C—H···π and π–π interactions in (I). Atom colours as in Fig. 1; 50% displacement ellipsoids.

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.244 (2)	C2—C3	1.5105 (17)
N1—C1	1.3495 (13)	C3—C7	1.3772 (16)
N1—C2	1.4459 (17)	C3—C4	1.3848 (17)
N2—C6	1.3294 (19)	C4—C5	1.3697 (19)
N2—C7	1.3391 (16)	C5—C6	1.375 (2)
C1—N1^{i}	1.3495 (13)		
C1—N1—C2	123.12 (11)	C7—C3—C2	121.17 (11)
C6—N2—C7	116.98 (11)	C4—C3—C2	121.98 (11)
O1—C1—N1	122.38 (7)	C5—C4—C3	119.83 (12)
N1—C1—N1^{i}	115.25 (15)	C4—C5—C6	118.81 (13)
N1—C2—C3	113.15 (10)	N2—C6—C5	123.11 (13)
C7—C3—C4	116.84 (11)	N2—C7—C3	124.43 (12)
C2—N1—C1—N1^{i}	−172.41 (10)	C2—N1—C1—O1	7.59 (12)
C1—N1—C2—C3	91.90 (12)		

Symmetry code: (i) $-x, y, \frac{1}{2}-z$.

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

π is the centroid of the N2-pyridyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N2^{i}	0.92 (2)	1.95 (2)	2.8633 (15)	175.3 (17)
O2—H3···O1	0.87 (2)	2.01 (2)	2.8699 (15)	171.5 (17)
N1—H1···O2^{ii}	0.866 (16)	2.023 (16)	2.8495 (14)	159.3 (13)
C5—H51···π^{iii}	0.93	3.08	3.74	129

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

The positions and $U_{\rm iso}$ for the N—H and O—H H atoms were freely refined; C—H H atoms refined by riding, with $U_{\rm iso}$ constrained to be 1.2 × that of $U_{\rm eq}$ for the attached C atom.

Data collection: *SMART* (Bruker, 1999*b*); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999*b*); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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