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6,7,8,9-Tetrahydro-5*H*-pyrido[2,3-*b*]azepine-5,8-dione: a hydrogen-bonded dimer structure

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Key indicators

Single-crystal X-ray study
T = 187 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.149
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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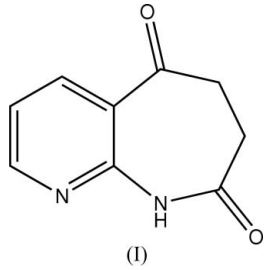
In the title compound, $C_9H_8N_2O_2$, two crystallographically independent molecules form a dimer structure, in which two N—H···N hydrogen bonds generate an intermolecular $R_2^2(8)$ ring.

Comment

The title compound, (I), is an intermediate in the synthesis of 1-azakepaullone (Kunick *et al.*, 2004), which is an inhibitor of glycogen synthase kinase-3 (Doble & Woodgett, 2003) and a potential antidiabetic drug (Wagmann & Nuss, 2001).



(I)

In the crystal structure of (I), two crystallographically independent molecules form a dimer through N—H···N hydrogen bonds (Fig. 1 and Table 1). These hydrogen bonds generate an $R_2^2(8)$ ring (Bernstein *et al.*, 1995). The dihedral angle between the two pyridine rings in the dimer is 7.6 (2)°. Atom C7 of the seven-membered ring has *endo* and *exo* conformations in the two independent molecules (Fig. 1).

The crystal structure consists of packed hydrogen-bonded dimers (Fig. 2). A π–π interaction is observed between the pyridine C10–C13/N2/C14 rings of neighbouring dimers; the centroid-to-centroid distance, $Cg\cdots Cg(-x, 2-y, -z)$, is 3.616 (2) Å and the interplanar spacing is 3.232 (2) Å.

Experimental

Compound (I) was prepared from 2-aminopyridine-3-carboxylic acid (purchased from Aldrich) according to the literature method of Kunick *et al.* (2003). Single crystals suitable for X-ray diffraction were obtained from an ethanol solution.

Crystal data

$C_9H_8N_2O_2$
M_r = 176.17
Triclinic, $P\overline{1}$
a = 8.2902 (11) Å
b = 8.5284 (11) Å
c = 11.1261 (15) Å
α = 98.155 (2)°
β = 98.996 (2)°
γ = 91.754 (2)°
V = 767.98 (18) Å^3
Z = 4
D_x = 1.524 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 187 (2) K
Block, colourless
0.20 × 0.13 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.978, T_{max} = 0.989
4341 measured reflections
2959 independent reflections
2109 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.149
S = 1.02
2959 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0819P)^2 + 0.0598P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N2^{i}	0.88	2.21	3.051 (2)	161
N4—H4A···N1^{i}	0.88	2.25	3.042 (2)	150

Symmetry code: (i) $-x, -y+2, -z+1$.

H atoms were found in a difference Fourier map and refined as riding, with C—H = 0.95–0.99 Å and N—H = 0.88 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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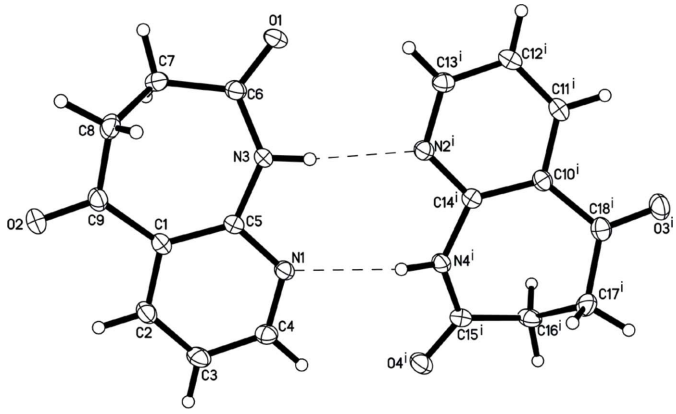

Figure 1
A view of the hydrogen-bonded dimer of (I), with the atom-labelling scheme and 30% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines. [Symmetry code (i) is as in Table 1].

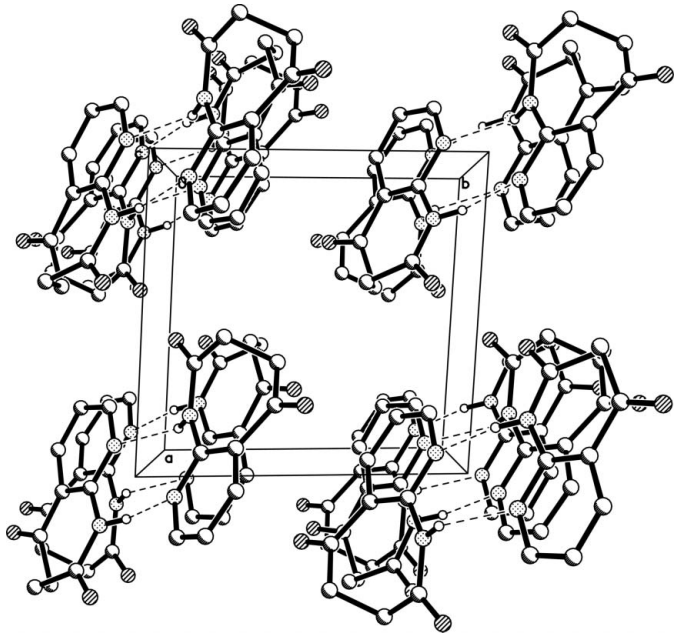

Figure 2
A packing view of (I), along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

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