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1*H*-Pyrido[3,4-*b*]azepine-2,5(3*H*,4*H*)-dione: a hydrogen-bonded dimeric structure

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.124
Data-to-parameter ratio = 13.4

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

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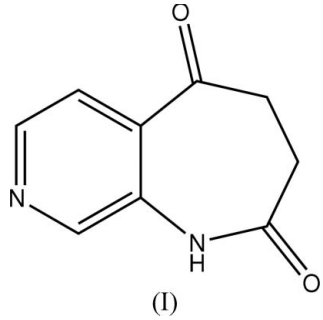
In the asymmetric unit of the title compound, $C_9H_8N_2O_2$, there are two crystallographically independent molecules, each of which forms a dimer, *via* N—H···O hydrogen bonds, with an inversion-related molecule.

Comment

The title compound, (I), is an isomer of 6,7,8,9-tetrahydro-5*H*-pyrid[2,3-*b*]azepine-5,8-dione (Gu *et al.*, 2006). It 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), the seven-menbered rings in the two crystallographically independent molecules have a half-chair-like conformation. Each independent molecule forms a dimer, *via* N—H···O hydrogen bonds, with a molecule related by an inversion center (Fig. 1 and Table 1). These hydrogen bonds generate $R_2^2(8)$ rings (Bernstein *et al.*, 1995). The dimers are connected by weak C—H···N and C—H···O hydrogen bonds (Fig. 2 and Table 1).

Experimental

The title compound was prepared from 3-aminopyridine (purchased from Shanhai Minghe Chemical Co.) according to a literature method (Estel *et al.*, 1989; Bhaskar & Rajesh, 1996; 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
Monoclinic, $P2_1/n$
a = 11.346 (3) Å
b = 10.708 (2) Å
c = 13.161 (3) Å
β = 93.219 (4)°
V = 1596.5 (6) Å^3
Z = 8
D_x = 1.466 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
Needle, colorless
0.36 × 0.16 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.962, T_{max} = 0.988
8803 measured reflections
3139 independent reflections
2116 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 26.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.124
S = 1.01
3139 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.0991P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.04	2.885 (2)	166
N4—H4···O4^{ii}	0.86	2.10	2.893 (2)	153
C7—H7A···N1^{iii}	0.97	2.61	3.499 (3)	152
C15—H15B···O1^{iv}	0.97	2.50	3.419 (3)	158

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x, -y, -z+1$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iv) $x-1, y, z$.

H atoms were positioned geometrically (C—H = 0.93–0.97 and N—H = 0.86 Å) and refined as riding 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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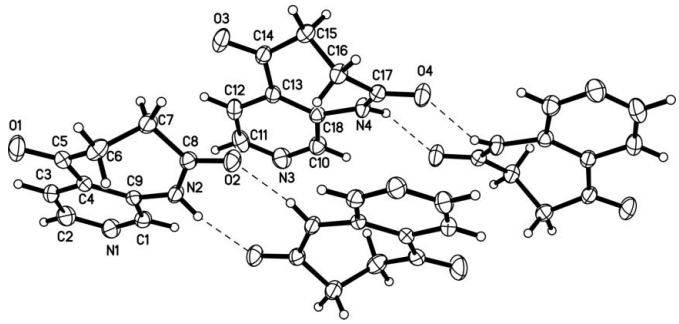

Figure 1
The structures of the two independent hydrogen-bonded dimers of (I), with the atom-labeling scheme and 30% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

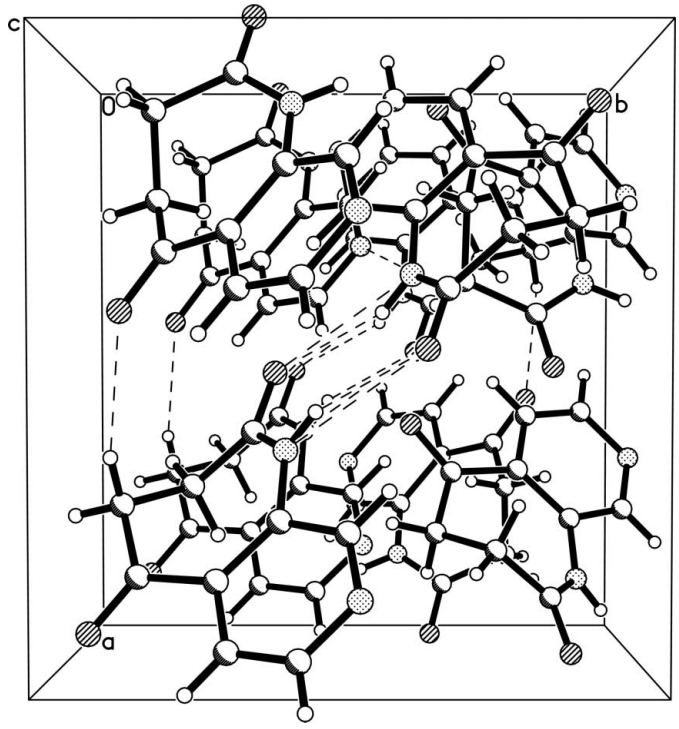

Figure 2
A view of the packing of (I) along the *c* axis. Hydrogen bonds are shown as dashed lines.

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