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N,*N*′-Di-2-pyridylmethylenediamine

Hui Wu,[a]* Jun Zhou,[a] Hui-Zhen Yu,[a] Lei-Lei Lu,[a] Zhou Xu,[a] Kai-Bei Yu[b] and Da-Qing Shi[a]

[a]Department of Chemistry, Xuzhou Normal University, Xuzhou, Jiangsu 221116, People's Republic of China, and [b]Chengdu Institute of Organic Chemistry, Chinese Academy of Science, Chengdu, Sichuan 610041, People's Republic of China

Correspondence e-mail: wuhui72@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.088
Data-to-parameter ratio = 12.9

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

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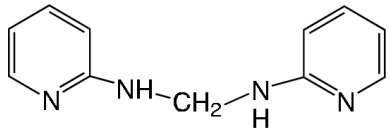
The title compound, $C_{11}H_{12}N_4$, was synthesized by the reaction of *N*,*N*-1,3-bis(hydroxymethyl)-5-fluorouracil with 2-aminopyridine in ethanol in the absence of a catalyst. The pyridine rings are approximately perpendicular to one another and are linked in the crystal structue *via* intermolecular N—H···N hydrogen bonds.

Comment

N,*N*-1,3-Bis(hydroxymethyl)-5-fluorouracil was synthesized by reacting methanal with 5-fluorouracil (5-FU), which possesses antitumour activity (Heidelderger, 1957). Some aminopyridines show anaesthetic properties and have been used as drugs for certain brain diseases (Okamato *et al.*, 1997). The title compound, (I), was synthesized by the reaction of *N*,*N*-1,3-bis(hydroxymethyl)-5-fluorouracil with 2-aminopyridine; this reaction may be reversible. We infer that the possible mechanism is that *N*,*N*-1,3-bis(hydroxymethyl)-5-fluorouracil releases methanal gradually in the presence of 2-aminopyridine, and then the methanal reacts with the 2-aminopyridine. In fact, if 2-aminopyridine were to react with methanal directly, the Schiff base (Mellor *et al.*, 1996) and not (I) could have been obtained. We report here the X-ray crystal structure of (I).



(I)

Bond lengths and angles in (I) show normal values (Table 1). The whole molecule is V-shaped (Fig. 1). The two pyridine rings are approximately perpendicular, the dihedral angle being 87.23 (5)°. Atom C6 deviates from the N1/C1/C2/C3/C4/C5 plane by 0.450 (2) Å and from the N4/C7/C8/C9/C10/C11 plane by 0.050 (2) Å.

The molecules of (I) are linked by an intermolecular N3—H3N···N1 hydrogen bond (Table 2), forming a one-dimensional chain along the *b* axis (Fig. 2).

Experimental

The title compound, (I), was prepared by reacting *N*,*N*-1,3-bis(hydroxymethyl)-5-fluorouracil with 2-aminopyridine (1:1) in ethanol (pH 4). Single crystals of (I) suitable for an X-ray study were obtained by slow evaporation of an aqueous ethanol solution (40% *v*/*v*) at 293 K over a period of 20 d.

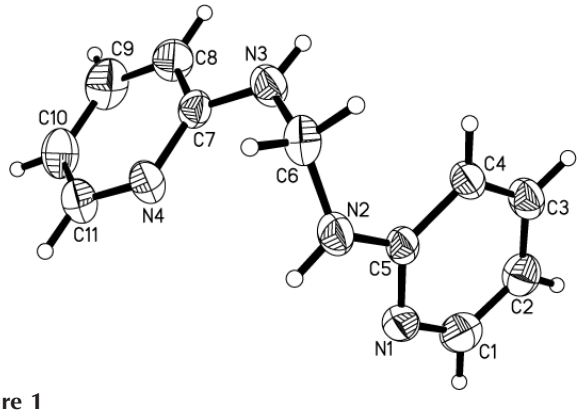


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Crystal data

$C_{11}H_{12}N_4$
M_r = 200.25
Monoclinic, $C2/c$
a = 17.960 (4) Å
b = 5.723 (1) Å
c = 20.419 (4) Å
β = 99.72 (2)°
V = 2068.8 (7) Å^3
Z = 8
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 38 reflections
θ = 3.8–13.7°
μ = 0.08 mm^{-1}
T = 296 (2) K
Rhomb, white
0.62 × 0.18 × 0.12 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
2229 measured reflections
1873 independent reflections
1057 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.3°
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 6$
$l = -24 \rightarrow 24$
3 standard reflections
every 97 reflections
intensity decay: 3.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.088
S = 0.81
1873 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.0065 (6)

Table 1
Selected geometric parameters (Å, °).

N2—C5	1.372 (2)	N3—C7	1.368 (2)
N2—C6	1.441 (2)	N3—C6	1.434 (2)
C5—N2—C6	123.16 (17)	N3—C6—N2	115.88 (17)
C7—N3—C6	122.55 (17)	N4—C7—N3	117.48 (17)
N1—C5—N2	116.13 (18)	N3—C7—C8	120.26 (19)
N2—C5—C4	121.91 (19)		

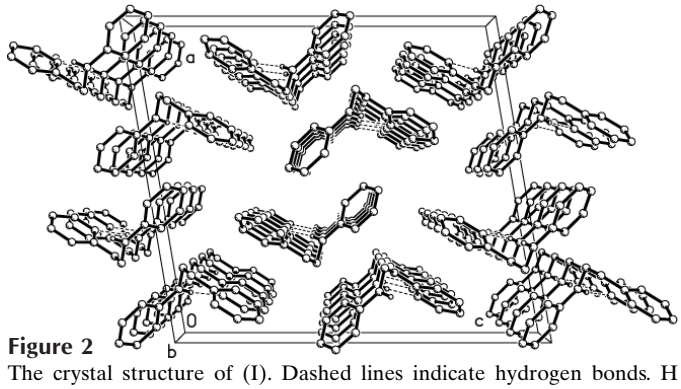


Figure 2
The crystal structure of (I). Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3N···N1^{i}	0.872 (9)	2.217 (14)	3.082 (2)	171.7 (12)

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

The H atoms on atoms N2 and N3 were located in difference Fourier syntheses and refined isotropically. All other H atoms were placed in theoretically calculated positions, with C—H distances of 0.93 Å in the pyridine rings and 0.97 Å for those on atom C6, and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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