Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Methyl-2-(2-pyridyl)hexahydropyrimidine

Saud Al-Resayes

Department of Chemistry, King Saud University, PO Box 2455, Riyadh 11451, Saudi Arabia
Correspondence e-mail: sresayes@ksu.edu.sa

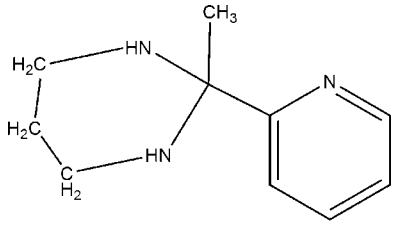
Received 29 June 2009; accepted 4 July 2009

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.115; data-to-parameter ratio = 10.3.

In the aminal-type title compound, $C_{10}H_{15}N_3$, the six-membered hexahydropyrimidine ring adopts a chair conformation and the N atoms are pyramidally coordinated. One of the two amido –NH units engages in intermolecular hydrogen bonding with the pyridyl N atom, generating a helical chain running along the b axis of the orthorhombic unit cell.

Related literature

The title compound is used in Fe(II) spin-crossover materials; see: Bréfuel *et al.* (2007).



Experimental

Crystal data

$C_{10}H_{15}N_3$
M_r = 177.25
Orthorhombic, $P2_12_12_1$
a = 8.4070 (17) Å
b = 10.371 (2) Å
c = 11.363 (2) Å
V = 990.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 294 K
0.35 × 0.15 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2007)
T_{min} = 0.97, T_{max} = 0.99
8017 measured reflections
1324 independent reflections
1206 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.115
S = 1.01
1324 reflections
128 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N1^{i}	0.89 (3)	2.31 (3)	3.188 (2)	168 (2)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2007); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006) and *PLUTO* (Motherwell *et al.*, 1999); software used to prepare material for publication: *publCIF* (Westrip, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2608).

References

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bréfuel, N., Shova, S. & Tuchagues, J.-P. (2007). *Eur. J. Inorg. Chem.* pp. 4326–4334.
Motherwell, W. D. S., Shields, G. P. & Allen, F. H. (1999). *Acta Cryst.* B**55**, 1044–1056.
Rigaku/MSC (2007). *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2009). *publCIF*. In preparation.

supporting information

Acta Cryst. (2009). E**65**, o1874 [doi:10.1107/S1600536809025963]

2-Methyl-2-(2-pyridyl)hexahydropyrimidine

Saud Al-Resayes

S1. Comment

The title compound was known as precursor for syntheses of umsymmetrical tetradentate Schiff base ligands that form bistable Fe(II) spin crossover materials (Bréfuel *et al.* 2007).

The molecular packing of the title compound is supported by N— H···N intermolecular hydrogen bondings at H···A distance of 2.31 (3) and D— H···A angle of 168 (2)° and calculated with Pluto (Motherwell *et al.*, 1999), see Figure 2.

S2. Experimental

1,3-Propane diamine (1 g, 0.013 mmol) was mixed with 1-(2-pyridinyl)-1-ethanone (1.635 g, 0.013 mmol) in 30 ml e thanol. The mixture was stirred under reflux for 5 h. The solution was concentrated under reduced pressure and the product was precipitated by addition of 30 ml of cool distilled water. Product was filtered off and washed three times with 15 ml of distilled water then dried under vacuum. Crude product was recrystallized from ethanol and allowed to stand at room temperature. Crystals were collected after 2 weeks.

S3. Refinement

Hydrogen atoms were refined isotropically and were constrained to the ideal geometry using an appropriate riding model with U_{iso}(H) fixed at 1.2 times U_{eq} of the pivot atom. The —NH hydrogen atoms was located from difference Fourier map and refined isotropically without constraints. 949 Friedel-pair reflections were merged for a weak anomalous scatterer structure.

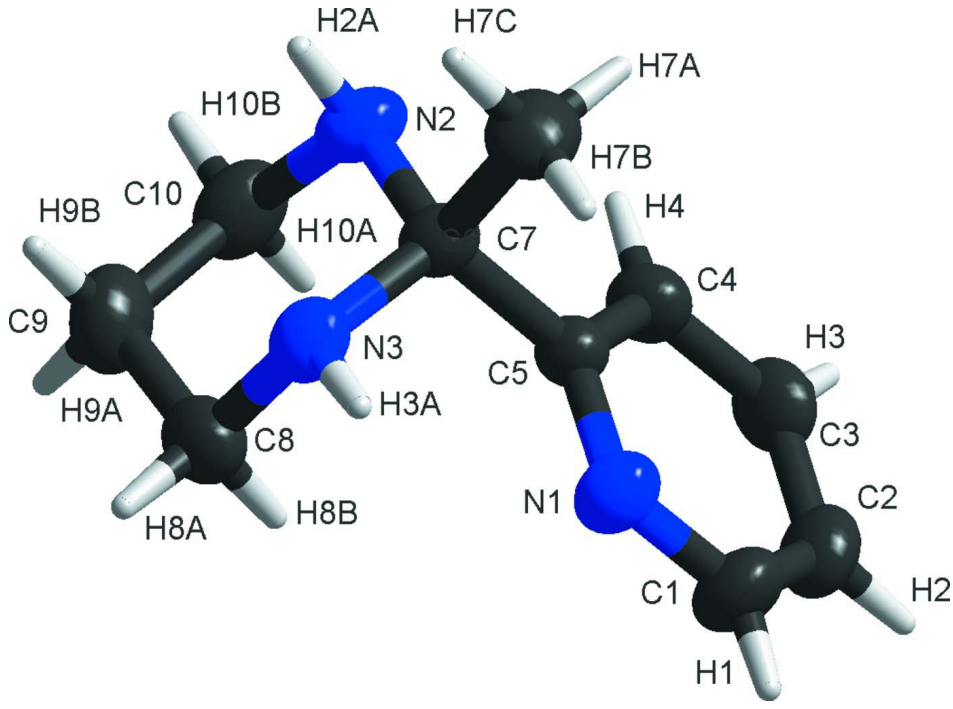


Figure 1

Perspective drawings of the title compound showing the atom-numbering scheme. The atomic displacement ellipsoids are shown at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

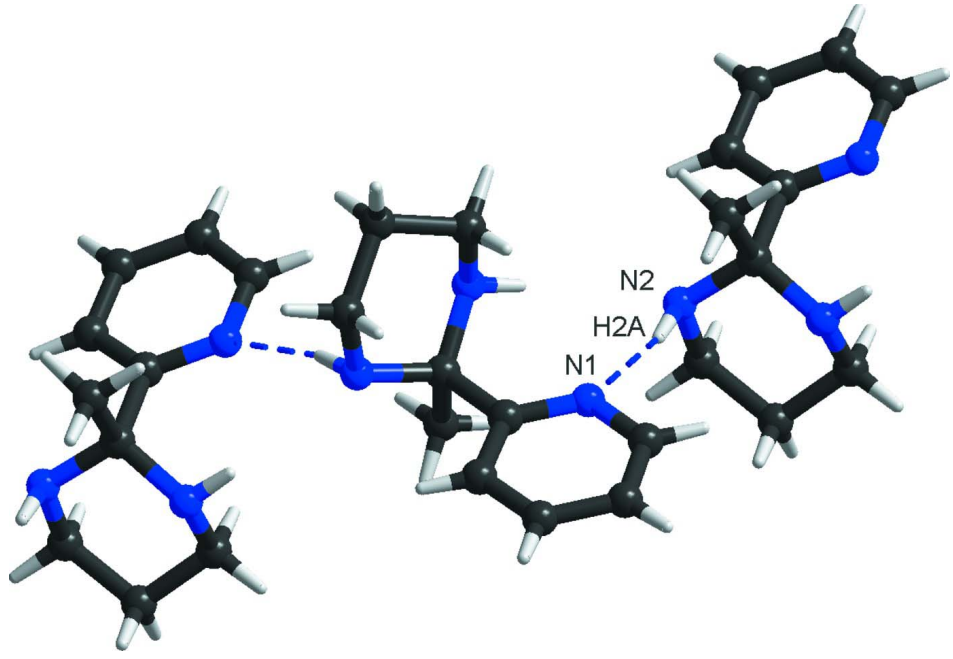


Figure 2

N— H···N intermolecular hydrogen bonding pattern of the title compound with hydrogen bonding shown as broken lines. Symmetry code: -*x*, *y* + 1/2, -*z* + 1/2.

2-Methyl-2-(2-pyridyl)hexahydropyrimidine

Crystal data

$C_{10}H_{15}N_3$
$M_r = 177.25$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 8.4070$ (17) Å
$b = 10.371$ (2) Å
$c = 11.363$ (2) Å
$V = 990.7$ (3) Å^3
$Z = 4$
$F(000) = 384$
$D_x = 1.188$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7168 reflections
$\theta = 3.0$–$27.5°$
$\mu = 0.07$ mm^{-1}
$T = 294$ K
Plate, yellow
0.35 × 0.15 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2007)
$T_{min} = 0.97$, $T_{max} = 0.99$
8017 measured reflections
1324 independent reflections
1206 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 12$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.115$
$S = 1.01$
1324 reflections
128 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0907P)^2 + 0.0352P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.20810 (17)	0.34068 (14)	0.21846 (15)	0.0473 (4)
C1	0.3442 (2)	0.27459 (18)	0.2066 (2)	0.0562 (5)
H1	0.3416	0.1859	0.2183	0.067*
N2	0.05820 (18)	0.66511 (13)	0.16234 (14)	0.0475 (3)

H2A	−0.026 (3)	0.711 (2)	0.187 (2)	0.063 (6)*
C2	0.4872 (2)	0.3304 (2)	0.17791 (18)	0.0553 (5)
H2	0.5789	0.2809	0.1707	0.066*
N3	−0.08570 (15)	0.46514 (15)	0.18351 (14)	0.0436 (3)
H3A	−0.087 (3)	0.391 (2)	0.221 (2)	0.063 (6)*
C3	0.49114 (19)	0.4615 (2)	0.16021 (17)	0.0518 (4)
H3	0.5861	0.5025	0.1410	0.062*
C4	0.35197 (19)	0.53176 (17)	0.17129 (16)	0.0445 (4)
H4	0.3523	0.6205	0.1599	0.053*
C5	0.21182 (17)	0.46810 (14)	0.19969 (13)	0.0364 (3)
C6	0.05402 (18)	0.53948 (14)	0.22108 (14)	0.0385 (3)
C7	0.0394 (3)	0.5618 (2)	0.35388 (16)	0.0574 (5)
H7A	0.1299	0.6097	0.3812	0.086*
H7B	0.0352	0.4802	0.3936	0.086*
H7C	−0.0560	0.6095	0.3701	0.086*
C8	−0.0901 (2)	0.44394 (19)	0.05613 (18)	0.0536 (4)
H8A	−0.1817	0.3918	0.0356	0.064*
H8B	0.0051	0.3987	0.0311	0.064*
C9	−0.1001 (3)	0.5740 (2)	−0.00463 (18)	0.0630 (5)
H9A	−0.0978	0.5624	−0.0893	0.076*
H9B	−0.1990	0.6164	0.0161	0.076*
C10	0.0403 (2)	0.6562 (2)	0.03398 (18)	0.0574 (5)
H10A	0.1370	0.6202	0.0009	0.069*
H10B	0.0274	0.7424	0.0022	0.069*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0406 (7)	0.0382 (6)	0.0631 (9)	0.0007 (6)	0.0038 (7)	0.0057 (6)
C1	0.0532 (10)	0.0419 (8)	0.0737 (12)	0.0105 (8)	0.0012 (9)	0.0040 (9)
N2	0.0480 (7)	0.0342 (6)	0.0602 (8)	0.0046 (6)	0.0084 (7)	0.0023 (6)
C2	0.0406 (8)	0.0658 (11)	0.0594 (10)	0.0145 (8)	0.0010 (7)	−0.0043 (9)
N3	0.0321 (6)	0.0439 (7)	0.0549 (8)	−0.0012 (5)	0.0053 (5)	0.0062 (7)
C3	0.0340 (7)	0.0649 (10)	0.0564 (9)	−0.0048 (7)	0.0033 (7)	−0.0035 (9)
C4	0.0388 (7)	0.0425 (7)	0.0522 (9)	−0.0059 (6)	0.0040 (6)	−0.0022 (7)
C5	0.0340 (7)	0.0353 (7)	0.0400 (7)	−0.0006 (6)	0.0011 (6)	−0.0017 (6)
C6	0.0371 (7)	0.0359 (7)	0.0427 (7)	0.0006 (6)	0.0067 (6)	0.0007 (6)
C7	0.0614 (11)	0.0643 (11)	0.0466 (9)	0.0089 (9)	0.0088 (8)	−0.0057 (8)
C8	0.0415 (8)	0.0590 (10)	0.0603 (10)	−0.0022 (8)	−0.0019 (7)	−0.0084 (8)
C9	0.0568 (11)	0.0812 (13)	0.0509 (10)	0.0073 (10)	−0.0036 (8)	0.0088 (10)
C10	0.0591 (10)	0.0551 (9)	0.0579 (10)	0.0062 (9)	0.0097 (9)	0.0181 (9)

Geometric parameters (Å, º)

N1—C1	1.340 (2)	C4—H4	0.9300
N1—C5	1.339 (2)	C5—C6	1.539 (2)
C1—C2	1.374 (3)	C6—C7	1.532 (2)
C1—H1	0.9300	C7—H7A	0.9600

N2—C10	1.469 (3)	C7—H7B	0.9600
N2—C6	1.464 (2)	C7—H7C	0.9600
N2—H2A	0.89 (3)	C8—C9	1.517 (3)
C2—C3	1.375 (3)	C8—H8A	0.9700
C2—H2	0.9300	C8—H8B	0.9700
N3—C6	1.468 (2)	C9—C10	1.521 (3)
N3—C8	1.464 (3)	C9—H9A	0.9700
N3—H3A	0.88 (3)	C9—H9B	0.9700
C3—C4	1.384 (2)	C10—H10A	0.9700
C3—H3	0.9300	C10—H10B	0.9700
C4—C5	1.389 (2)		

C1—N1—C5	117.95 (14)	N2—C6—C5	109.60 (12)
N1—C1—C2	123.75 (16)	C7—C6—C5	107.30 (14)
N1—C1—H1	118.1	C6—C7—H7A	109.5
C2—C1—H1	118.1	C6—C7—H7B	109.5
C10—N2—C6	113.21 (14)	H7A—C7—H7B	109.5
C10—N2—H2A	105.3 (17)	C6—C7—H7C	109.5
C6—N2—H2A	108.1 (16)	H7A—C7—H7C	109.5
C3—C2—C1	118.18 (16)	H7B—C7—H7C	109.5
C3—C2—H2	120.9	N3—C8—C9	108.52 (16)
C1—C2—H2	120.9	N3—C8—H8A	110.0
C6—N3—C8	112.72 (13)	C9—C8—H8A	110.0
C6—N3—H3A	109.1 (16)	N3—C8—H8B	110.0
C8—N3—H3A	110.1 (16)	C9—C8—H8B	110.0
C2—C3—C4	119.15 (16)	H8A—C8—H8B	108.4
C2—C3—H3	120.4	C10—C9—C8	108.91 (16)
C4—C3—H3	120.4	C10—C9—H9A	109.9
C5—C4—C3	119.22 (16)	C8—C9—H9A	109.9
C5—C4—H4	120.4	C10—C9—H9B	109.9
C3—C4—H4	120.4	C8—C9—H9B	109.9
N1—C5—C4	121.74 (15)	H9A—C9—H9B	108.3
N1—C5—C6	115.44 (13)	N2—C10—C9	113.65 (16)
C4—C5—C6	122.65 (13)	N2—C10—H10A	108.8
N3—C6—N2	110.72 (13)	C9—C10—H10A	108.8
N3—C6—C7	107.56 (13)	N2—C10—H10B	108.8
N2—C6—C7	108.46 (14)	C9—C10—H10B	108.8
N3—C6—C5	113.03 (12)	H10A—C10—H10B	107.7

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···N1^{i}	0.89 (3)	2.31 (3)	3.188 (2)	168 (2)

Symmetry code: (i) −*x*, *y*+1/2, −*z*+1/2.