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2-Methyl-4-(pyridin-2-yl)-3*H*-1,5-benzodiazepine

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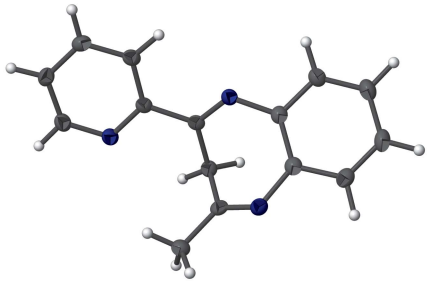
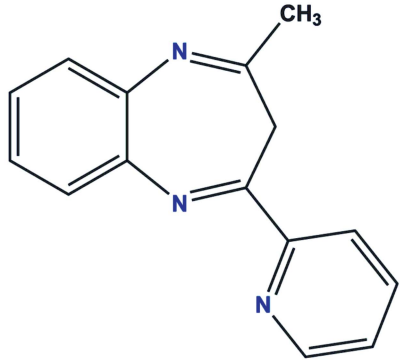
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{13}N_3$, the seven-membered ring adopts a boat conformation. In the crystal, inversion-related C—H···N hydrogen bonds form dimers, which pack in an alternating fashion.

3D view



Chemical scheme



Structure description

The renewed interest in bicyclic heterocycles derived from 1,5-benzodiazepine is based mainly on their biological properties and therapeutic functions (Wang *et al.*, 2015). As a continuation of our research into 1,5-benzodiazepine derivatives (Tjiou *et al.*, 2005), we prepared the title compound and characterized it by X-ray diffraction.

In the title compound (Fig. 1), the seven-membered ring adopts a boat conformation with Cremer–Pople puckering parameters $Q(2)$ = 0.8492 (11) Å, $Q(3)$ = 0.2494 (12) Å, $\varphi(2)$ = 205.89 (8)° and $\varphi(3)$ = 307.6 (3)°. The total puckering amplitude is 0.8850 (12) Å. The C1–C6 phenyl ring makes a dihedral angle of 30.05 (6)° with the N3/C11–C15 pyridine ring. In the crystal, inversion-related C15—H15···N3 hydrogen bonds form weak dimers, which pack in an alternating fashion (Table 1 and Fig. 2).

Synthesis and crystallization

A mixture of *o*-phenylenediamine (3 mmol) and 1-(pyridin-2-yl)butane-1,3-dione (3 mmol) in 30 ml of xylene was heated under reflux for 3 h. The reaction mixture was cooled at room temperature, the precipitated solid was collected by filtration and recrystallized from dry ethanol to give yellow crystals, m.p. 99–100°C.

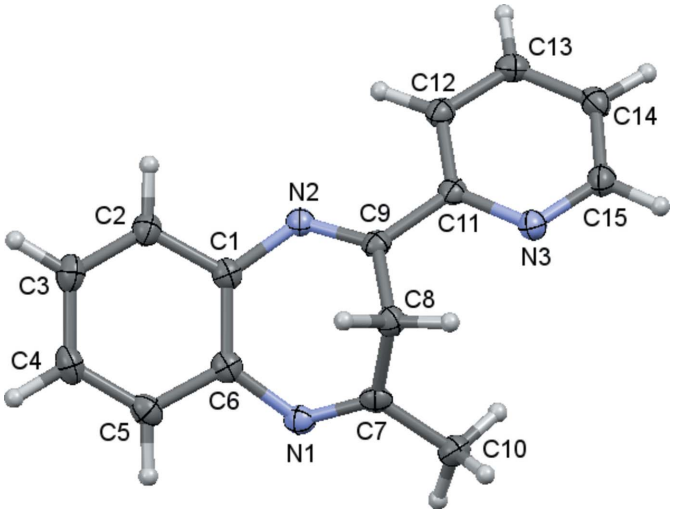


Figure 1
The molecular structure of the title molecule with the labelling scheme and 50% probability ellipsoids.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

JTM thanks Tulane University for support of the Tulane Crystallography Laboratory.

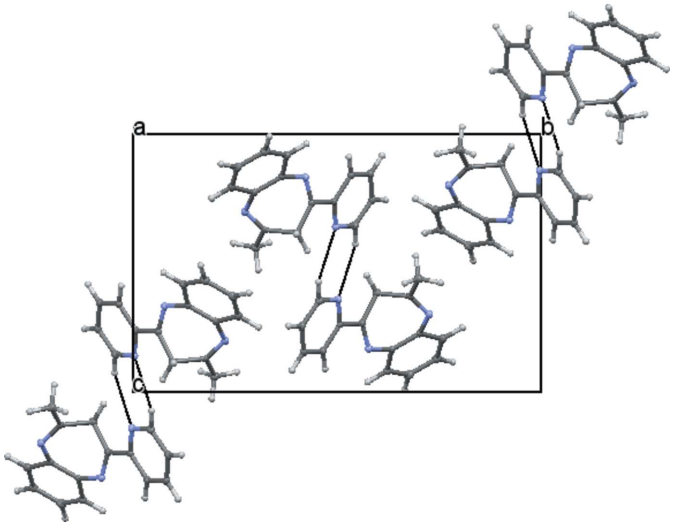


Figure 2
Packing viewed along the *a*-axis direction with C—H···O hydrogen bonds shown as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15···N3^{i}	0.989 (13)	2.590 (13)	3.4900 (16)	151.3 (10)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{13}N_3$
M_r	235.28
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	5.9642 (6), 17.7004 (17), 11.3963 (11)
β (°)	100.695 (1)
V (Å^3)	1182.2 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.27 × 0.20 × 0.07
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.84, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11157, 3013, 2144
R_{int}	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.686
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.108, 0.97
No. of reflections	3013
No. of parameters	215
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.20

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2008) and *SHELXTL* (Sheldrick, 2008).

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full crystallographic data

IUCrData (2018). **3**, x180953 [https://doi.org/10.1107/S2414314618009537]

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Crystal data

$C_{15}H_{13}N_3$
M_r = 235.28
Monoclinic, $P2_1/n$
a = 5.9642 (6) Å
b = 17.7004 (17) Å
c = 11.3963 (11) Å
β = 100.695 (1)°
V = 1182.2 (2) Å^3
Z = 4
$F(000)$ = 496
D_x = 1.322 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3540 reflections
θ = 2.3–28.7°
μ = 0.08 mm^{-1}
T = 100 K
Plate, colorless
0.27 × 0.20 × 0.07 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.84, T_{max} = 0.99
11157 measured reflections
3013 independent reflections
2144 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 29.2°, θ_{min} = 2.2°
h = −8→8
k = −23→23
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.108
S = 0.97
3013 reflections
215 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0657P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Experimental. The diffraction data were collected in three sets of 363 frames (0.5° width in ω) at φ = 0, 120 and 240°. A scan time of 30 sec/frame was used.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.34913 (16)	0.27417 (5)	0.30084 (8)	0.0224 (2)
N2	0.27587 (16)	0.42244 (5)	0.16780 (8)	0.0204 (2)
N3	0.76518 (16)	0.49702 (5)	0.35890 (8)	0.0228 (2)
C1	0.11011 (19)	0.36589 (6)	0.16439 (10)	0.0207 (3)
C2	−0.0941 (2)	0.37687 (7)	0.08182 (11)	0.0244 (3)
H2	−0.111 (2)	0.4250 (7)	0.0379 (11)	0.025 (3)*
C3	−0.2649 (2)	0.32385 (8)	0.06639 (11)	0.0281 (3)
H3	−0.409 (2)	0.3340 (8)	0.0098 (12)	0.037 (4)*
C4	−0.2331 (2)	0.25575 (7)	0.12854 (11)	0.0282 (3)
H4	−0.353 (2)	0.2159 (8)	0.1160 (11)	0.033 (4)*
C5	−0.0300 (2)	0.24196 (7)	0.20519 (11)	0.0252 (3)
H5	0.001 (2)	0.1925 (8)	0.2492 (11)	0.029 (3)*
C6	0.1437 (2)	0.29660 (6)	0.22717 (10)	0.0215 (3)
C7	0.4687 (2)	0.32169 (7)	0.37092 (10)	0.0217 (3)
C8	0.3899 (2)	0.40186 (7)	0.38289 (10)	0.0217 (3)
H8A	0.224 (2)	0.4008 (7)	0.3882 (10)	0.023 (3)*
H8B	0.483 (2)	0.4274 (7)	0.4525 (11)	0.024 (3)*
C9	0.41139 (19)	0.43848 (6)	0.26626 (10)	0.0196 (3)
C10	0.6968 (2)	0.29969 (8)	0.43983 (12)	0.0267 (3)
H10A	0.815 (2)	0.3338 (8)	0.4217 (12)	0.039 (4)*
H10B	0.702 (2)	0.3068 (8)	0.5264 (13)	0.036 (4)*
H10C	0.736 (2)	0.2461 (9)	0.4232 (12)	0.036 (4)*
C11	0.59968 (19)	0.49276 (6)	0.26147 (10)	0.0193 (2)
C12	0.6041 (2)	0.53489 (6)	0.15832 (10)	0.0224 (3)
H12	0.477 (2)	0.5293 (7)	0.0896 (11)	0.023 (3)*
C13	0.7871 (2)	0.58193 (7)	0.15521 (11)	0.0239 (3)
H13	0.792 (2)	0.6104 (7)	0.0838 (12)	0.028 (3)*
C14	0.9602 (2)	0.58643 (7)	0.25468 (11)	0.0245 (3)
H14	1.090 (2)	0.6186 (7)	0.2554 (11)	0.026 (3)*
C15	0.9410 (2)	0.54339 (7)	0.35369 (11)	0.0255 (3)
H15	1.062 (2)	0.5457 (7)	0.4260 (12)	0.030 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0229 (5)	0.0219 (5)	0.0233 (5)	−0.0002 (4)	0.0060 (4)	0.0016 (4)
N2	0.0201 (5)	0.0188 (5)	0.0219 (5)	0.0006 (4)	0.0023 (4)	−0.0003 (4)
N3	0.0242 (5)	0.0210 (5)	0.0220 (5)	−0.0009 (4)	0.0007 (4)	−0.0015 (4)
C1	0.0213 (6)	0.0219 (6)	0.0198 (6)	−0.0004 (5)	0.0061 (5)	−0.0028 (4)
C2	0.0224 (6)	0.0255 (6)	0.0246 (6)	0.0011 (5)	0.0024 (5)	−0.0021 (5)
C3	0.0199 (6)	0.0343 (7)	0.0294 (7)	−0.0007 (5)	0.0023 (5)	−0.0037 (5)
C4	0.0237 (6)	0.0312 (7)	0.0304 (7)	−0.0079 (6)	0.0069 (5)	−0.0058 (5)
C5	0.0279 (7)	0.0239 (6)	0.0254 (6)	−0.0030 (5)	0.0091 (5)	−0.0012 (5)
C6	0.0224 (6)	0.0229 (6)	0.0206 (6)	0.0007 (5)	0.0074 (5)	−0.0028 (4)
C7	0.0252 (6)	0.0218 (6)	0.0195 (6)	−0.0004 (5)	0.0074 (5)	0.0046 (4)
C8	0.0226 (6)	0.0231 (6)	0.0194 (6)	−0.0010 (5)	0.0040 (5)	−0.0008 (5)
C9	0.0207 (6)	0.0174 (5)	0.0204 (6)	0.0040 (5)	0.0030 (5)	−0.0004 (4)
C10	0.0260 (7)	0.0269 (7)	0.0261 (7)	0.0017 (5)	0.0019 (5)	0.0036 (5)
C11	0.0213 (6)	0.0162 (5)	0.0200 (6)	0.0028 (4)	0.0028 (4)	−0.0022 (4)
C12	0.0249 (6)	0.0195 (6)	0.0215 (6)	0.0023 (5)	0.0010 (5)	−0.0011 (5)
C13	0.0288 (7)	0.0197 (6)	0.0238 (6)	0.0021 (5)	0.0065 (5)	0.0022 (5)
C14	0.0234 (6)	0.0196 (6)	0.0307 (7)	−0.0021 (5)	0.0055 (5)	−0.0017 (5)
C15	0.0240 (6)	0.0240 (6)	0.0265 (6)	−0.0007 (5)	−0.0006 (5)	−0.0026 (5)

Geometric parameters (Å, º)

N1—C7	1.2816 (14)	C7—C8	1.5089 (17)
N1—C6	1.4080 (15)	C8—C9	1.5055 (16)
N2—C9	1.2871 (14)	C8—H8A	1.001 (13)
N2—C1	1.4023 (14)	C8—H8B	0.990 (12)
N3—C15	1.3418 (15)	C9—C11	1.4865 (16)
N3—C11	1.3439 (14)	C10—H10A	0.981 (15)
C1—C2	1.4076 (16)	C10—H10B	0.989 (14)
C1—C6	1.4152 (16)	C10—H10C	1.003 (15)
C2—C3	1.3724 (17)	C11—C12	1.3965 (16)
C2—H2	0.984 (13)	C12—C13	1.3784 (17)
C3—C4	1.3931 (19)	C12—H12	0.987 (12)
C3—H3	0.990 (14)	C13—C14	1.3865 (17)
C4—C5	1.3775 (17)	C13—H13	0.963 (13)
C4—H4	0.997 (13)	C14—C15	1.3835 (17)
C5—C6	1.4050 (16)	C14—H14	0.961 (14)
C5—H5	1.008 (13)	C15—H15	0.989 (13)
C7—C10	1.4909 (17)		
C7—N1—C6	120.57 (10)	C9—C8—H8B	112.6 (7)
C9—N2—C1	120.46 (10)	C7—C8—H8B	111.4 (7)
C15—N3—C11	117.27 (10)	H8A—C8—H8B	111.9 (10)
N2—C1—C2	115.98 (10)	N2—C9—C11	117.84 (10)
N2—C1—C6	124.87 (10)	N2—C9—C8	122.15 (10)
C2—C1—C6	118.76 (10)	C11—C9—C8	119.97 (10)

C3—C2—C1	121.40 (12)	C7—C10—H10A	110.5 (8)
C3—C2—H2	121.7 (7)	C7—C10—H10B	110.5 (8)
C1—C2—H2	116.9 (7)	H10A—C10—H10B	104.0 (11)
C2—C3—C4	119.91 (12)	C7—C10—H10C	111.7 (8)
C2—C3—H3	119.5 (8)	H10A—C10—H10C	110.0 (12)
C4—C3—H3	120.6 (8)	H10B—C10—H10C	110.0 (11)
C5—C4—C3	119.78 (12)	N3—C11—C12	122.66 (10)
C5—C4—H4	119.4 (7)	N3—C11—C9	116.74 (10)
C3—C4—H4	120.8 (7)	C12—C11—C9	120.56 (10)
C4—C5—C6	121.56 (12)	C13—C12—C11	118.88 (11)
C4—C5—H5	121.9 (8)	C13—C12—H12	122.3 (7)
C6—C5—H5	116.5 (8)	C11—C12—H12	118.9 (7)
C5—C6—N1	116.31 (10)	C12—C13—C14	119.11 (11)
C5—C6—C1	118.43 (11)	C12—C13—H13	119.1 (8)
N1—C6—C1	124.86 (10)	C14—C13—H13	121.8 (8)
N1—C7—C10	120.31 (11)	C15—C14—C13	118.27 (11)
N1—C7—C8	121.79 (10)	C15—C14—H14	120.5 (7)
C10—C7—C8	117.86 (11)	C13—C14—H14	121.2 (7)
C9—C8—C7	104.25 (9)	N3—C15—C14	123.80 (11)
C9—C8—H8A	107.9 (7)	N3—C15—H15	116.2 (8)
C7—C8—H8A	108.4 (7)	C14—C15—H15	120.0 (8)

C9—N2—C1—C2	147.82 (11)	C10—C7—C8—C9	106.51 (11)
C9—N2—C1—C6	−39.40 (16)	C1—N2—C9—C11	173.32 (9)
N2—C1—C2—C3	177.04 (11)	C1—N2—C9—C8	−4.15 (16)
C6—C1—C2—C3	3.79 (17)	C7—C8—C9—N2	71.43 (14)
C1—C2—C3—C4	−3.46 (18)	C7—C8—C9—C11	−105.99 (11)
C2—C3—C4—C5	−0.03 (18)	C15—N3—C11—C12	−0.86 (16)
C3—C4—C5—C6	3.16 (18)	C15—N3—C11—C9	177.18 (10)
C4—C5—C6—N1	−175.86 (10)	N2—C9—C11—N3	−166.77 (10)
C4—C5—C6—C1	−2.76 (17)	C8—C9—C11—N3	10.75 (15)
C7—N1—C6—C5	−146.53 (11)	N2—C9—C11—C12	11.31 (16)
C7—N1—C6—C1	40.87 (16)	C8—C9—C11—C12	−171.17 (10)
N2—C1—C6—C5	−173.28 (10)	N3—C11—C12—C13	1.07 (17)
C2—C1—C6—C5	−0.68 (16)	C9—C11—C12—C13	−176.89 (10)
N2—C1—C6—N1	−0.82 (17)	C11—C12—C13—C14	−0.47 (17)
C2—C1—C6—N1	171.78 (11)	C12—C13—C14—C15	−0.25 (18)
C6—N1—C7—C10	−174.01 (10)	C11—N3—C15—C14	0.08 (18)
C6—N1—C7—C8	3.42 (16)	C13—C14—C15—N3	0.47 (19)
N1—C7—C8—C9	−70.98 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15···N3^{i}	0.989 (13)	2.590 (13)	3.4900 (16)	151.3 (10)

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