

4-Methyl-1*H*-1,5-benzodiazepin-2(3*H*)-one

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Received 30 November 2018

Accepted 3 December 2018

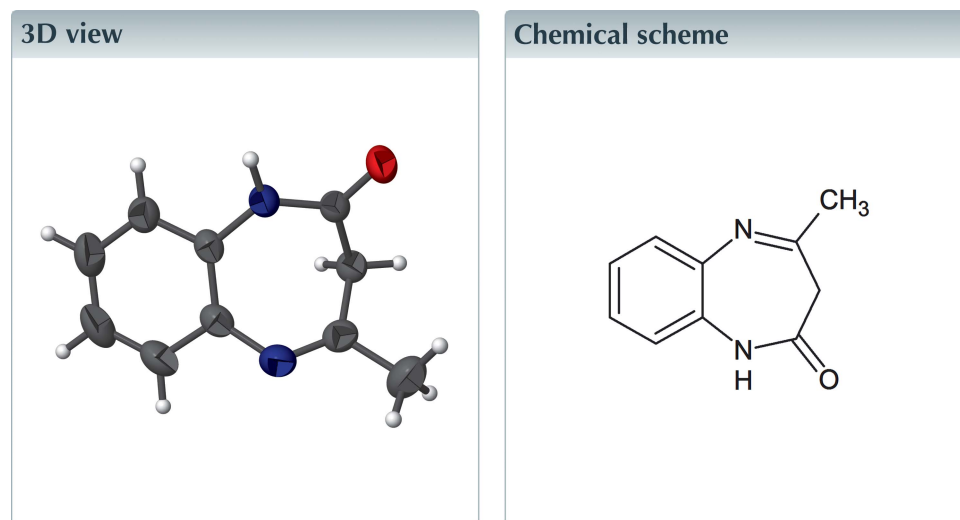
Edited by W. T. A. Harrison, University of Aberdeen, Scotland

Keywords: crystal structure; benzodiazepines; hydrogen bonds.

CCDC reference: 1882884

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

In the title compound, C₁₀H₁₀N₂O, the seven-membered heterocycle displays a half-chair conformation. In the crystal, N—H···O and C—H···O hydrogen bonds link the molecules into chains propagating along the *a*-axis direction.



Structure description

Benzodiazepines are a class of drugs that act on the GABA-A receptor as an allosteric modulator and increase the frequency of opening of its chloride channel (Twyman *et al.* 1989). The biological effects of these drugs include hypnotic, anticonvulsant and muscle relaxant properties (De Sarro *et al.* 1995). As part of our studies in this area, we now describe the synthesis and structure of the title compound.

The molecule of the title compound (Fig. 1) is built up from fused six- and seven-membered rings. The seven-membered ring displays a half-chair conformation as indicated by the puckering amplitude $Q_T = 0.8734$ (16) Å and spherical polar angle $\varphi_2 = 205.48$ (11)° and $\varphi_3 = 310.2$ (4)°. In the crystal, the molecules are linked by N—H···O and C—H···O hydrogen bonds (Table 1), generating [100] chains as shown in Fig. 2.

Synthesis and crystallization

Ethyl acetoacetate (0.011 mmol) was added to a stirred solution of benzene-1,2-diamine (0.01 mmol) in 100 ml of xylene. The mixture was stirred at reflux for 1 h. The resulting precipitate was filtered, washed with ethanol, then dried giving a white powder. Colourless prisms of the title compound were obtained by recrystallization from diethyl ether solution.

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1N···O1 ⁱ	0.92 (2)	1.94 (2)	2.8481 (17)	169 (2)
C8–H8A···O1 ⁱⁱ	0.97	2.57	3.480 (2)	156

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

Yield = 90% (white solid); m.p. = 176–178°C. ¹H NMR (300.13 MHz; CDCl₃): 2.40 (3H, –CH₃, s); 3.15 (2H, –CH₂–, s); 7.08–7.37 (4H_{arom}, m); 9.32 (1H, –NH, s). ¹³C NMR (75.47 MHz; CDCl₃): 28.01 (–CH₃); 43.57 (–CH₂–); 121.88–

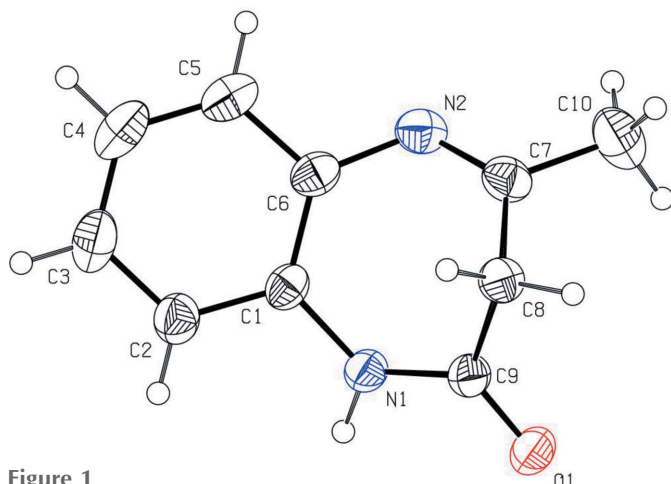


Figure 1
Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

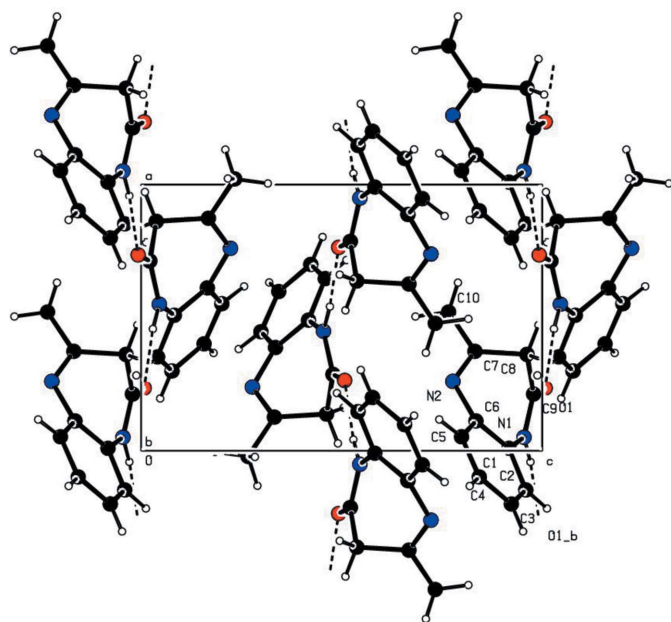


Figure 2
Structure of the title compound, showing molecules linked through N–H···O and C–H···O hydrogen bonds (dashed lines).

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₀ H ₁₀ N ₂ O
<i>M</i> _r	174.20
Crystal system, space group	Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.5676 (2), 10.7802 (2), 11.4092 (3)
<i>V</i> (Å ³)	930.77 (4)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ^{−1})	0.08
Crystal size (mm)	0.32 × 0.28 × 0.20
Data collection	
Diffractometer	Bruker X8 APEXII CCD area-detector
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	25369, 1825, 1747
<i>R</i> _{int}	0.018
(sin θ / λ) _{max} (Å ^{−1})	0.616
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.028, 0.086, 1.03
No. of reflections	1825
No. of parameters	123
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ^{−3})	0.14, −0.15
Absolute structure	Flack <i>x</i> determined using 705 quotients [(<i>I</i> ⁺) − (<i>I</i> [−])] / [(<i>I</i> ⁺) + (<i>I</i> [−])] (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	−0.3 (2)

Computer programs: APEX2 and SAINT (Bruker, 2009), SHELXT (Sheldrick, 2015a), SHELXL2014 (Sheldrick, 2015b), PLATON (Spek, 2009) and publCIF (Westrip, 2010).

139.62 (6C_{arom}); 162.89 (CN); 167.36 (CO). MS–EI: [*M* + 1]⁺ = 175.

Refinement

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

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full crystallographic data

IUCrData (2018). 3, x181718 [https://doi.org/10.1107/S2414314618017182]

4-Methyl-1*H*-1,5-benzodiazepin-2(3*H*)-one

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4-Methyl-1*H*-1,5-benzodiazepin-2(3*H*)-one*Crystal data*

C₁₀H₁₀N₂O

M_r = 174.20

Orthorhombic, *P*2₁2₁2₁

a = 7.5676 (2) Å

b = 10.7802 (2) Å

c = 11.4092 (3) Å

V = 930.77 (4) Å³

Z = 4

F(000) = 368

D_x = 1.243 Mg m⁻³

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 245 reflections

θ = 0.5–32°

μ = 0.08 mm⁻¹

T = 296 K

Prism, colourless

0.32 × 0.28 × 0.20 mm

Data collection

Bruker X8 APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω and φ scans

25369 measured reflections

1825 independent reflections

1747 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.018

θ_{max} = 26.0°, θ_{min} = 3.6°

h = -9→9

k = -13→13

l = -13→14

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.028

wR(*F*²) = 0.086

S = 1.03

1825 reflections

123 parameters

1 restraint

Primary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

w = 1/[σ²(*F_o*²) + (0.0631*P*)² + 0.0577*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.14 e Å⁻³

Δρ_{min} = -0.15 e Å⁻³

Absolute structure: Flack *x* determined using

705 quotients [(*F*⁻)-(*F*)]/[(*F*⁺)+(*F*)] (Parsons *et al.*,
2013)

Absolute structure parameter: -0.3 (2)

Special details

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. The H atoms were located in a difference map and treated as riding with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and with *U*_{iso}(H) = 1.2–1.5*U*_{eq}(C, N).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0092 (2)	0.51751 (13)	0.91727 (13)	0.0373 (3)
C9	0.2131 (2)	0.35141 (15)	0.97843 (13)	0.0379 (3)
C6	0.1120 (2)	0.58213 (14)	0.83489 (13)	0.0410 (4)
C8	0.3621 (2)	0.44253 (15)	0.96221 (14)	0.0402 (3)
H8A	0.4723	0.4076	0.9906	0.048*
H8B	0.3383	0.5185	1.0049	0.048*
C2	-0.1472 (2)	0.56990 (16)	0.95911 (15)	0.0466 (4)
H2	-0.2167	0.5258	1.0119	0.056*
C5	0.0527 (3)	0.69915 (16)	0.79881 (16)	0.0550 (5)
H5	0.1177	0.7428	0.7433	0.066*
C7	0.3736 (2)	0.46818 (16)	0.83226 (14)	0.0439 (4)
C3	-0.2001 (3)	0.68650 (18)	0.92309 (17)	0.0601 (5)
H3	-0.3034	0.7213	0.9524	0.072*
C4	-0.0983 (3)	0.75106 (17)	0.84313 (19)	0.0641 (6)
H4	-0.1325	0.8300	0.8193	0.077*
C10	0.5213 (3)	0.4081 (3)	0.76664 (19)	0.0667 (6)
H10A	0.5044	0.4201	0.6840	0.100*
H10B	0.5230	0.3209	0.7837	0.100*
H10C	0.6316	0.4445	0.7900	0.100*
N1	0.05078 (16)	0.39599 (12)	0.95461 (12)	0.0394 (3)
N2	0.26065 (19)	0.53402 (13)	0.77731 (12)	0.0459 (3)
O1	0.23581 (16)	0.24292 (11)	1.00743 (13)	0.0519 (3)
H1N	-0.042 (3)	0.3435 (19)	0.9707 (19)	0.057 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0376 (7)	0.0352 (7)	0.0392 (7)	0.0012 (6)	-0.0087 (6)	0.0009 (6)
C9	0.0332 (7)	0.0387 (8)	0.0419 (7)	0.0000 (6)	-0.0015 (6)	0.0047 (6)
C6	0.0467 (8)	0.0381 (7)	0.0382 (7)	-0.0027 (7)	-0.0076 (6)	0.0028 (6)
C8	0.0325 (7)	0.0438 (8)	0.0443 (8)	-0.0043 (6)	-0.0057 (6)	0.0036 (6)
C2	0.0424 (8)	0.0501 (9)	0.0473 (8)	0.0056 (7)	-0.0035 (7)	0.0015 (7)
C5	0.0670 (12)	0.0448 (9)	0.0531 (10)	-0.0003 (9)	-0.0084 (9)	0.0125 (8)
C7	0.0378 (8)	0.0483 (8)	0.0454 (8)	-0.0078 (7)	0.0004 (6)	0.0011 (7)
C3	0.0616 (11)	0.0585 (11)	0.0601 (10)	0.0239 (9)	-0.0067 (9)	-0.0046 (9)
C4	0.0828 (14)	0.0448 (9)	0.0648 (12)	0.0172 (10)	-0.0152 (11)	0.0076 (9)
C10	0.0471 (10)	0.0954 (16)	0.0576 (11)	0.0069 (11)	0.0063 (8)	-0.0033 (11)
N1	0.0317 (6)	0.0351 (6)	0.0514 (7)	-0.0029 (5)	-0.0022 (5)	0.0078 (5)
N2	0.0466 (7)	0.0498 (8)	0.0413 (6)	-0.0051 (7)	-0.0003 (6)	0.0054 (6)
O1	0.0383 (6)	0.0411 (6)	0.0764 (8)	0.0023 (5)	-0.0022 (6)	0.0167 (5)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.395 (2)	C2—H2	0.9300
C1—C6	1.405 (2)	C5—C4	1.370 (3)

C1—N1	1.4131 (19)	C5—H5	0.9300
C9—O1	1.2275 (19)	C7—N2	1.276 (2)
C9—N1	1.347 (2)	C7—C10	1.493 (3)
C9—C8	1.507 (2)	C3—C4	1.382 (3)
C6—C5	1.401 (2)	C3—H3	0.9300
C6—N2	1.402 (2)	C4—H4	0.9300
C8—C7	1.511 (2)	C10—H10A	0.9600
C8—H8A	0.9700	C10—H10B	0.9600
C8—H8B	0.9700	C10—H10C	0.9600
C2—C3	1.382 (2)	N1—H1N	0.918 (18)
C2—C1—C6	119.86 (14)	C6—C5—H5	119.1
C2—C1—N1	117.45 (14)	N2—C7—C10	119.78 (16)
C6—C1—N1	122.54 (14)	N2—C7—C8	123.06 (15)
O1—C9—N1	121.47 (14)	C10—C7—C8	117.11 (16)
O1—C9—C8	123.32 (14)	C4—C3—C2	119.55 (18)
N1—C9—C8	115.16 (13)	C4—C3—H3	120.2
C5—C6—N2	116.89 (15)	C2—C3—H3	120.2
C5—C6—C1	117.76 (16)	C5—C4—C3	120.20 (17)
N2—C6—C1	125.06 (14)	C5—C4—H4	119.9
C9—C8—C7	106.43 (13)	C3—C4—H4	119.9
C9—C8—H8A	110.4	C7—C10—H10A	109.5
C7—C8—H8A	110.4	C7—C10—H10B	109.5
C9—C8—H8B	110.4	H10A—C10—H10B	109.5
C7—C8—H8B	110.4	C7—C10—H10C	109.5
H8A—C8—H8B	108.6	H10A—C10—H10C	109.5
C3—C2—C1	120.80 (17)	H10B—C10—H10C	109.5
C3—C2—H2	119.6	C9—N1—C1	126.51 (13)
C1—C2—H2	119.6	C9—N1—H1N	115.8 (14)
C4—C5—C6	121.79 (18)	C1—N1—H1N	117.5 (14)
C4—C5—H5	119.1	C7—N2—C6	120.87 (14)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...O1 ⁱ	0.92 (2)	1.94 (2)	2.8481 (17)	169 (2)
C8—H8A...O1 ⁱⁱ	0.97	2.57	3.480 (2)	156

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