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1-Acetyl-4-phenyl-5a,6,7,8,9,9a-hexahydro-5H-1,5-benzodiazepin-2(1H)-one

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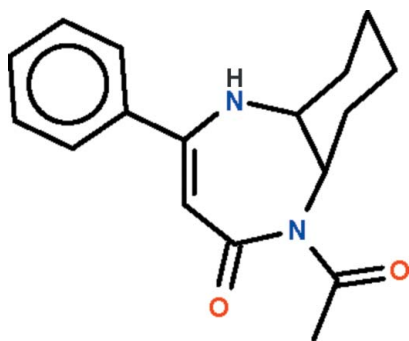
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Key indicators: single-crystal X-ray study; $T = 193$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.046; wR factor = 0.139; data-to-parameter ratio = 23.5.

The seven-membered ring of the title compound, $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_2$, adopts an approximate boat conformation while the cyclohexyl ring adopts a chair conformation. In the crystal, adjacent molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into a zigzag chain running along the c axis of the monoclinic unit cell.

Related literature

For the crystal structures of anhydrous and hydrated 7-phenyl-1,2,3,4-tetrahydro-1,4-diazepin-5-ones, see: Clark *et al.* (1999); Chammache *et al.* (2001).


Experimental
Crystal data

$\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_2$
 $M_r = 284.35$
Monoclinic, $P2_1/c$
 $a = 9.6794$ (2) Å
 $b = 14.0095$ (3) Å
 $c = 11.2832$ (2) Å
 $\beta = 98.053$ (1)°

$V = 1514.95$ (5) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 193$ K
 $0.6 \times 0.6 \times 0.6$ mm

Data collection

Bruker APEXII diffractometer
Absorption correction: none
27399 measured reflections

4591 independent reflections
3878 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.139$
 $S = 1.05$
4591 reflections
195 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.38$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O1}^{\text{i}}$	0.88 (1)	2.001 (9)	2.854 (1)	164 (1)

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2009).

We thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2546).

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supporting information

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1-Acetyl-4-phenyl-5a,6,7,8,9,9a-hexahydro-5H-1,5-benzodiazepin-2(1H)-one

Hanane Benzeid, Nathalie Saffon, Bernard Garrigues, El Mokhtar Essassi and Seik Weng Ng

S1. Experimental

4-Phenyl-5a,6,7,8,9,9a-hexahydro-1H-benzo[*b*][1,4]diazepin-2(3H)-one (1 g) was refluxed in acetic acid (20 ml) for 12 h. The precipate was collected and recrystallized from ethanol to afford colorless crystals in 90% yield.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with $U_{\text{iso}}(\text{H})$ set to 1.2–1.5 $U_{\text{eq}}(\text{C})$. The amino H-atom was located in a difference Fourier map and was refined with an N—H 0.88±0.01 Å restraint.

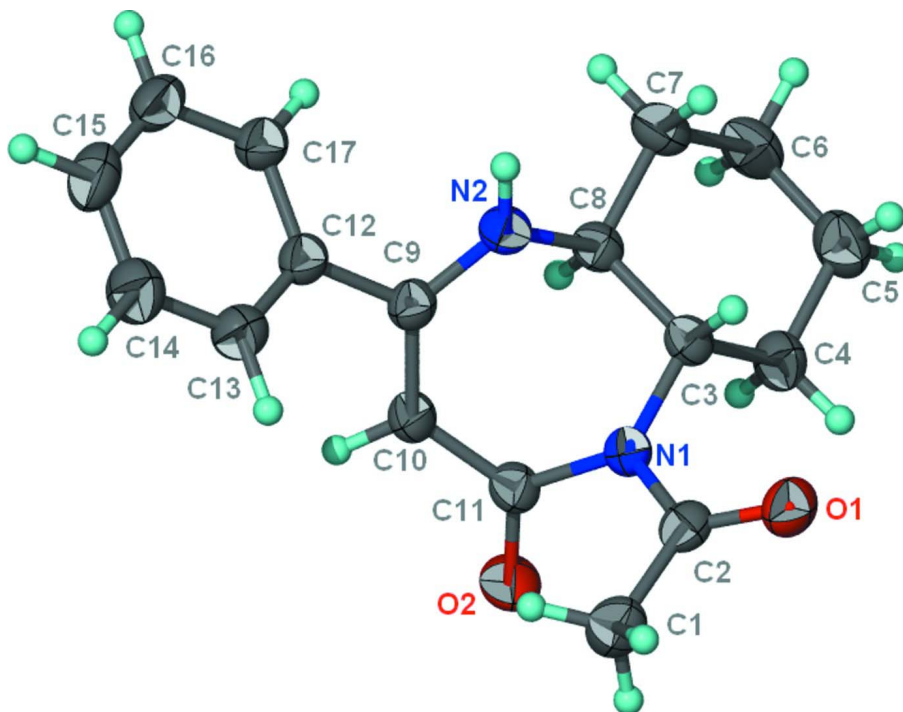


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

1-Acetyl-4-phenyl-5a,6,7,8,9,9a-hexahydro-5H-1,5-benzodiazepin-2(1H)-one

Crystal data

$C_{17}H_{20}N_2O_2$

$M_r = 284.35$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.6794$ (2) Å

$b = 14.0095$ (3) Å

$c = 11.2832$ (2) Å

$\beta = 98.053$ (1)°

$V = 1514.95$ (5) Å³

$Z = 4$

$F(000) = 608$

$D_x = 1.247$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9928 reflections

$\theta = 5.2$ – 30.6 °

$\mu = 0.08$ mm⁻¹

$T = 193$ K

Block, colorless

$0.6 \times 0.6 \times 0.6$ mm

Data collection

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

27399 measured reflections

4591 independent reflections

3878 reflections with $I > 2\sigma(I)$

$R_{int} = 0.024$

$\theta_{max} = 30.5$ °, $\theta_{min} = 5.2$ °

$h = -13$ → 13

$k = 0$ → 20

$l = 0$ → 16

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.139$

$S = 1.05$

4591 reflections

195 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0824P)^2 + 0.2971P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.38$ e Å⁻³

$\Delta\rho_{min} = -0.30$ e Å⁻³

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

	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
O1	0.38581 (9)	0.66240 (6)	0.46797 (7)	0.03445 (19)
O2	0.32929 (9)	0.42092 (6)	0.27399 (8)	0.0390 (2)
N1	0.40005 (8)	0.57744 (6)	0.29988 (7)	0.02455 (17)
N2	0.44012 (9)	0.65352 (6)	0.07259 (8)	0.02644 (18)
H2	0.4411 (15)	0.7098 (7)	0.0384 (12)	0.038 (4)*
C1	0.18397 (12)	0.57212 (10)	0.39317 (10)	0.0398 (3)
H1A	0.1299	0.6227	0.4252	0.060*
H1B	0.1843	0.5152	0.4436	0.060*
H1C	0.1417	0.5566	0.3114	0.060*
C2	0.33153 (10)	0.60571 (7)	0.39210 (8)	0.0266 (2)
C3	0.53214 (10)	0.62624 (7)	0.28648 (9)	0.02480 (19)
H3	0.5220	0.6951	0.3071	0.030*
C4	0.65550 (11)	0.58553 (8)	0.37093 (10)	0.0321 (2)

H4A	0.6663	0.5168	0.3538	0.039*
H4B	0.6375	0.5918	0.4548	0.039*
C5	0.78980 (12)	0.63873 (10)	0.35493 (12)	0.0412 (3)
H5A	0.8697	0.6087	0.4059	0.049*
H5B	0.7828	0.7058	0.3812	0.049*
C6	0.81573 (12)	0.63687 (9)	0.22446 (12)	0.0382 (3)
H6A	0.8330	0.5703	0.2009	0.046*
H6B	0.8999	0.6750	0.2160	0.046*
C7	0.69074 (11)	0.67727 (8)	0.14134 (10)	0.0322 (2)
H7A	0.7086	0.6732	0.0572	0.039*
H7B	0.6774	0.7453	0.1607	0.039*
C8	0.55891 (10)	0.62073 (7)	0.15649 (9)	0.02496 (19)
H8	0.5762	0.5523	0.1379	0.030*
C9	0.33011 (10)	0.59655 (7)	0.03819 (8)	0.02568 (19)
C10	0.30297 (11)	0.51532 (7)	0.10041 (9)	0.0280 (2)
H10	0.2498	0.4668	0.0563	0.034*
C11	0.34703 (10)	0.49695 (7)	0.22489 (9)	0.02630 (19)
C12	0.24115 (11)	0.62065 (7)	-0.07600 (9)	0.0269 (2)
C13	0.09583 (12)	0.61485 (8)	-0.08454 (10)	0.0333 (2)
H13	0.0541	0.5972	-0.0164	0.040*
C14	0.01201 (13)	0.63481 (9)	-0.19226 (11)	0.0390 (3)
H14	-0.0867	0.6311	-0.1976	0.047*
C15	0.07343 (14)	0.66027 (9)	-0.29194 (10)	0.0400 (3)
H15	0.0164	0.6734	-0.3657	0.048*
C16	0.21775 (14)	0.66671 (8)	-0.28440 (10)	0.0365 (2)
H16	0.2590	0.6844	-0.3527	0.044*
C17	0.30146 (12)	0.64719 (7)	-0.17679 (9)	0.0308 (2)
H17	0.4000	0.6519	-0.1716	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0456 (5)	0.0305 (4)	0.0279 (4)	-0.0012 (3)	0.0075 (3)	-0.0051 (3)
O2	0.0502 (5)	0.0266 (4)	0.0384 (4)	-0.0081 (3)	-0.0005 (4)	0.0075 (3)
N1	0.0264 (4)	0.0238 (4)	0.0234 (4)	-0.0018 (3)	0.0033 (3)	-0.0015 (3)
N2	0.0308 (4)	0.0209 (4)	0.0275 (4)	-0.0017 (3)	0.0038 (3)	0.0017 (3)
C1	0.0286 (5)	0.0589 (7)	0.0326 (5)	-0.0009 (5)	0.0063 (4)	0.0009 (5)
C2	0.0301 (5)	0.0263 (4)	0.0236 (4)	0.0030 (3)	0.0038 (3)	0.0026 (3)
C3	0.0256 (4)	0.0214 (4)	0.0273 (4)	-0.0012 (3)	0.0034 (3)	-0.0019 (3)
C4	0.0281 (5)	0.0344 (5)	0.0324 (5)	0.0006 (4)	-0.0010 (4)	-0.0002 (4)
C5	0.0286 (5)	0.0494 (7)	0.0440 (6)	-0.0053 (5)	-0.0006 (4)	-0.0051 (5)
C6	0.0272 (5)	0.0390 (6)	0.0492 (7)	-0.0038 (4)	0.0080 (4)	-0.0051 (5)
C7	0.0314 (5)	0.0278 (5)	0.0387 (5)	-0.0050 (4)	0.0096 (4)	-0.0028 (4)
C8	0.0258 (4)	0.0208 (4)	0.0286 (4)	-0.0003 (3)	0.0049 (3)	-0.0016 (3)
C9	0.0310 (5)	0.0233 (4)	0.0229 (4)	-0.0006 (3)	0.0047 (3)	-0.0026 (3)
C10	0.0357 (5)	0.0228 (4)	0.0251 (4)	-0.0053 (3)	0.0030 (4)	-0.0028 (3)
C11	0.0290 (4)	0.0219 (4)	0.0277 (4)	-0.0014 (3)	0.0029 (3)	-0.0005 (3)
C12	0.0326 (5)	0.0246 (4)	0.0235 (4)	-0.0022 (3)	0.0036 (3)	-0.0012 (3)

C13	0.0335 (5)	0.0366 (5)	0.0300 (5)	-0.0015 (4)	0.0056 (4)	0.0012 (4)
C14	0.0347 (5)	0.0422 (6)	0.0383 (6)	0.0006 (4)	-0.0006 (4)	0.0002 (5)
C15	0.0506 (7)	0.0390 (6)	0.0277 (5)	0.0019 (5)	-0.0041 (5)	-0.0006 (4)
C16	0.0519 (7)	0.0348 (5)	0.0233 (4)	-0.0036 (5)	0.0067 (4)	-0.0006 (4)
C17	0.0383 (5)	0.0283 (5)	0.0263 (4)	-0.0042 (4)	0.0065 (4)	-0.0023 (4)

Geometric parameters (Å, °)

O1—C2	1.2303 (13)	C6—H6A	0.9900
O2—C11	1.2238 (12)	C6—H6B	0.9900
N1—C2	1.3686 (12)	C7—C8	1.5317 (14)
N1—C11	1.4591 (12)	C7—H7A	0.9900
N1—C3	1.4761 (12)	C7—H7B	0.9900
N2—C9	1.3438 (13)	C8—H8	1.0000
N2—C8	1.4574 (13)	C9—C10	1.3815 (13)
N2—H2	0.878 (9)	C9—C12	1.4849 (14)
C1—C2	1.5055 (15)	C10—C11	1.4328 (14)
C1—H1A	0.9800	C10—H10	0.9500
C1—H1B	0.9800	C12—C13	1.3987 (15)
C1—H1C	0.9800	C12—C17	1.3995 (14)
C3—C4	1.5298 (14)	C13—C14	1.3919 (16)
C3—C8	1.5268 (13)	C13—H13	0.9500
C3—H3	1.0000	C14—C15	1.3904 (17)
C4—C5	1.5309 (16)	C14—H14	0.9500
C4—H4A	0.9900	C15—C16	1.3904 (19)
C4—H4B	0.9900	C15—H15	0.9500
C5—C6	1.5279 (18)	C16—C17	1.3892 (16)
C5—H5A	0.9900	C16—H16	0.9500
C5—H5B	0.9900	C17—H17	0.9500
C6—C7	1.5315 (17)		
C2—N1—C11	119.73 (8)	C6—C7—C8	109.85 (9)
C2—N1—C3	117.57 (8)	C6—C7—H7A	109.7
C11—N1—C3	122.54 (8)	C8—C7—H7A	109.7
C9—N2—C8	121.44 (8)	C6—C7—H7B	109.7
C9—N2—H2	117.6 (10)	C8—C7—H7B	109.7
C8—N2—H2	120.8 (10)	H7A—C7—H7B	108.2
C2—C1—H1A	109.5	N2—C8—C3	112.54 (8)
C2—C1—H1B	109.5	N2—C8—C7	111.00 (8)
H1A—C1—H1B	109.5	C3—C8—C7	109.71 (8)
C2—C1—H1C	109.5	N2—C8—H8	107.8
H1A—C1—H1C	109.5	C3—C8—H8	107.8
H1B—C1—H1C	109.5	C7—C8—H8	107.8
O1—C2—N1	120.71 (9)	N2—C9—C10	122.81 (9)
O1—C2—C1	120.62 (9)	N2—C9—C12	117.08 (8)
N1—C2—C1	118.49 (9)	C10—C9—C12	119.99 (9)
N1—C3—C4	112.03 (8)	C9—C10—C11	126.47 (9)
N1—C3—C8	109.94 (7)	C9—C10—H10	116.8

C4—C3—C8	110.90 (8)	C11—C10—H10	116.8
N1—C3—H3	107.9	O2—C11—C10	123.97 (9)
C4—C3—H3	107.9	O2—C11—N1	118.06 (9)
C8—C3—H3	107.9	C10—C11—N1	117.51 (8)
C3—C4—C5	110.28 (9)	C13—C12—C17	119.30 (10)
C3—C4—H4A	109.6	C13—C12—C9	120.13 (9)
C5—C4—H4A	109.6	C17—C12—C9	120.55 (9)
C3—C4—H4B	109.6	C14—C13—C12	120.40 (10)
C5—C4—H4B	109.6	C14—C13—H13	119.8
H4A—C4—H4B	108.1	C12—C13—H13	119.8
C4—C5—C6	111.23 (10)	C15—C14—C13	119.66 (11)
C4—C5—H5A	109.4	C15—C14—H14	120.2
C6—C5—H5A	109.4	C13—C14—H14	120.2
C4—C5—H5B	109.4	C14—C15—C16	120.46 (11)
C6—C5—H5B	109.4	C14—C15—H15	119.8
H5A—C5—H5B	108.0	C16—C15—H15	119.8
C5—C6—C7	111.19 (9)	C17—C16—C15	119.92 (10)
C5—C6—H6A	109.4	C17—C16—H16	120.0
C7—C6—H6A	109.4	C15—C16—H16	120.0
C5—C6—H6B	109.4	C16—C17—C12	120.26 (11)
C7—C6—H6B	109.4	C16—C17—H17	119.9
H6A—C6—H6B	108.0	C12—C17—H17	119.9
C11—N1—C2—O1	168.01 (9)	C8—N2—C9—C12	-158.47 (9)
C3—N1—C2—O1	-7.51 (14)	N2—C9—C10—C11	24.81 (16)
C11—N1—C2—C1	-16.81 (14)	C12—C9—C10—C11	-159.20 (10)
C3—N1—C2—C1	167.66 (9)	C9—C10—C11—O2	-174.30 (11)
C2—N1—C3—C4	81.59 (10)	C9—C10—C11—N1	13.72 (15)
C11—N1—C3—C4	-93.80 (10)	C2—N1—C11—O2	-55.02 (13)
C2—N1—C3—C8	-154.61 (8)	C3—N1—C11—O2	120.27 (11)
C11—N1—C3—C8	30.00 (11)	C2—N1—C11—C10	117.44 (10)
N1—C3—C4—C5	-179.46 (9)	C3—N1—C11—C10	-67.27 (12)
C8—C3—C4—C5	57.29 (11)	N2—C9—C12—C13	-138.24 (10)
C3—C4—C5—C6	-55.11 (13)	C10—C9—C12—C13	45.54 (14)
C4—C5—C6—C7	55.70 (14)	N2—C9—C12—C17	43.26 (13)
C5—C6—C7—C8	-57.34 (12)	C10—C9—C12—C17	-132.96 (10)
C9—N2—C8—C3	-80.75 (11)	C17—C12—C13—C14	0.29 (16)
C9—N2—C8—C7	155.86 (9)	C9—C12—C13—C14	-178.23 (10)
N1—C3—C8—N2	52.05 (10)	C12—C13—C14—C15	0.27 (18)
C4—C3—C8—N2	176.50 (8)	C13—C14—C15—C16	-0.56 (19)
N1—C3—C8—C7	176.16 (8)	C14—C15—C16—C17	0.29 (18)
C4—C3—C8—C7	-59.39 (10)	C15—C16—C17—C12	0.27 (17)
C6—C7—C8—N2	-176.16 (8)	C13—C12—C17—C16	-0.56 (16)
C6—C7—C8—C3	58.84 (11)	C9—C12—C17—C16	177.95 (9)
C8—N2—C9—C10	17.64 (14)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2 \cdots O1 ⁱ	0.88 (1)	2.00 (1)	2.854 (1)	164 (1)

Symmetry code: (i) $x, -y+3/2, z-1/2$.