Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

5-Acetyl-3-hydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one

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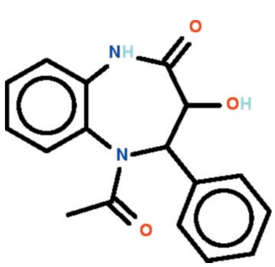
Received 7 November 2011; accepted 10 November 2011

Key indicators: single-crystal X-ray study; *T* = 293 K; mean σ(C–C) = 0.002 Å; *R* factor = 0.047; *wR* factor = 0.149; data-to-parameter ratio = 20.2.

In the title compound, $C_{17}H_{16}N_2O_3$, the seven-membered diazepine ring adopts a boat conformation with the hydroxy-substituted C atom at the prow and fused benzene ring C atoms at the stern. The phenyl substituent occupies an equatorial position. The amino group of the ring system is a hydrogen-bond donor to the oxo O atom of an inversion-related molecule, and the hydroxy group is a hydrogen-bond donor to the acetyl O atom of another inversion-related molecule. The two hydrogen bonds generate a ribbon motif parallel to [10$\overline{1}$] in the crystal structure.

Related literature

For a related 1,5-benzodiazepin-2(3*H*)-one structure, see: Rida *et al.* (2011).



Experimental

Crystal data

$C_{17}H_{16}N_2O_3$
M_r = 296.32
Triclinic, $P\overline{1}$
a = 8.9710 (1) Å
b = 9.3142 (1) Å
c = 9.4129 (1) Å
α = 81.563 (1)°
β = 68.921 (1)°
γ = 80.146 (1)°
V = 719.95 (1) Å^3
Z = 2
Mo *K*α radiation
μ = 0.10 mm^{-1}
T = 293 K
0.29 × 0.23 × 0.18 mm

Data collection

Bruker APEX DUO diffractometer
19110 measured reflections
4203 independent reflections
3584 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.149
S = 1.02
4203 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.89 (2)	2.04 (2)	2.924 (1)	175 (2)
O2—H2···O3^{ii}	0.83 (2)	2.09 (2)	2.905 (1)	168 (2)

Symmetry codes: (i) −*x*, −*y* + 1, −*z* + 1; (ii) −*x* + 1, −*y* + 1, −*z*.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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, 2010).

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: LH5376).

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supplementary materials

Acta Cryst. (2011). E**67**, o3337 [**doi:10.1107/S1600536811047878**]

5-Acetyl-3-hydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one

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Comment

The report on 3-hydroxy-4-phenyl-1-[(3-phenyl-4,5-dihydro-1,2-oxazol-5-yl)methyl]-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one provides the preparation and biological activity of this class of benzodiazepin-2-ones (Rida *et al.*, 2011). In the present study, the reactant, 3-ydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one, has two amino –NH– units in the ring system; however, only one site is acetylated when the compound is treated with acetic anhydride. In the title compound, the seven-membered diazepine ring adopts a boat conformation with the hydroxy-substituted C atom at the prow and fused-ring C atoms at the stern; the phenyl substituent occupies an equatorial position (Fig. 1). The amino group of the ring system is a hydrogen-bond donor to the oxo O atom of an inversion-related molecule, and the hydroxy group is hydrogen-bond donor to the acetyl O atom of another inversion-related molecule (Table 1). The two hydrogen bonds generate a ribbon motif parallel to [1 0 - 1] (Fig. 2).

Experimental

3-Hydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one (1 g. 3.9 mmol) was heated in acetic anhydride (20 ml) for 12 h. The precipitate was collected and recrystallized from ethanol to afford colorless crystals.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*(C). The amino and hydroxy H-atoms were located in a difference Fourier map and were freely refined.

Figures

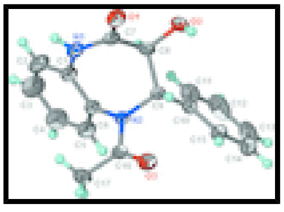

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{17}H_{16}N_2O_3$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

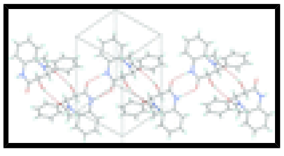

Fig. 2. Ribbon motif.

5-Acetyl-3-hydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin- 2(3*H*)-one

Crystal data

$C_{17}H_{16}N_2O_3$	$Z = 2$
$M_r = 296.32$	$F(000) = 312$
Triclinic, $P\bar{1}$	$D_x = 1.367$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.9710$ (1) Å	Cell parameters from 9950 reflections
$b = 9.3142$ (1) Å	$\theta = 2.2$–34.5°
$c = 9.4129$ (1) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 81.563$ (1)°	$T = 293$ K
$\beta = 68.921$ (1)°	Prism, colorless
$\gamma = 80.146$ (1)°	0.29 × 0.23 × 0.18 mm
$V = 719.95$ (1) Å^3	

Data collection

Bruker APEX DUO diffractometer	3584 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.024$
graphite	$\theta_{max} = 30.0°$, $\theta_{min} = 2.2°$
ω scans	$h = -12 \rightarrow 12$
19110 measured reflections	$k = -13 \rightarrow 13$
4203 independent reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.047$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.149$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0876P)^2 + 0.1414P]$ where $P = (F_o^2 + 2F_c^2)/3$
4203 reflections	$(\Delta/\sigma)_{max} = 0.001$
208 parameters	$\Delta\rho_{max} = 0.30$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.27$ e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.18813 (12)	0.41278 (9)	0.39332 (11)	0.0488 (2)
O2	0.50968 (12)	0.39185 (9)	0.34285 (10)	0.0430 (2)

O3	0.41433 (13)	0.74792 (11)	−0.06449 (10)	0.0513 (2)
N1	0.14350 (13)	0.64817 (10)	0.44597 (12)	0.0426 (2)
N2	0.37829 (11)	0.75893 (9)	0.18338 (9)	0.03178 (19)
C1	0.19921 (15)	0.78134 (12)	0.44620 (13)	0.0406 (3)
C2	0.1358 (2)	0.85962 (16)	0.57413 (16)	0.0656 (5)
H2A	0.0547	0.8253	0.6599	0.079*
C3	0.1927 (3)	0.98823 (18)	0.5745 (2)	0.0756 (6)
H3	0.1499	1.0396	0.6609	0.091*
C4	0.3123 (2)	1.04144 (16)	0.4483 (2)	0.0639 (4)
H4	0.3501	1.1280	0.4496	0.077*
C5	0.37548 (17)	0.96508 (13)	0.31996 (15)	0.0468 (3)
H5	0.4570	0.9999	0.2349	0.056*
C6	0.31790 (13)	0.83653 (11)	0.31723 (12)	0.0346 (2)
C7	0.24114 (14)	0.52550 (11)	0.39399 (12)	0.0363 (2)
C8	0.42230 (13)	0.53221 (11)	0.34285 (11)	0.0329 (2)
H8	0.4407	0.5843	0.4171	0.039*
C9	0.48415 (12)	0.61900 (10)	0.18549 (10)	0.0293 (2)
H9	0.4758	0.5617	0.1099	0.035*
C10	0.65889 (13)	0.64317 (11)	0.13784 (12)	0.0345 (2)
C11	0.72509 (17)	0.67606 (14)	0.24003 (18)	0.0493 (3)
H11	0.6625	0.6825	0.3425	0.059*
C12	0.8864 (2)	0.69927 (17)	0.1874 (3)	0.0685 (5)
H12	0.9311	0.7212	0.2555	0.082*
C13	0.98046 (18)	0.69010 (16)	0.0359 (3)	0.0722 (6)
H13	1.0876	0.7066	0.0020	0.087*
C14	0.91519 (18)	0.65653 (16)	−0.0649 (2)	0.0624 (4)
H14	0.9786	0.6493	−0.1671	0.075*
C15	0.75549 (15)	0.63361 (13)	−0.01454 (15)	0.0450 (3)
H15	0.7120	0.6115	−0.0835	0.054*
C16	0.35154 (14)	0.81314 (12)	0.05077 (12)	0.0371 (2)
C17	0.24042 (18)	0.95367 (15)	0.05114 (17)	0.0529 (3)
H17A	0.1983	0.9597	−0.0303	0.079*
H17B	0.2988	1.0350	0.0367	0.079*
H17C	0.1532	0.9560	0.1472	0.079*
H1	0.042 (2)	0.635 (2)	0.498 (2)	0.057 (5)*
H2	0.517 (2)	0.353 (2)	0.266 (2)	0.065 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0575 (5)	0.0342 (4)	0.0491 (5)	−0.0188 (4)	−0.0046 (4)	−0.0074 (3)
O2	0.0622 (5)	0.0290 (4)	0.0370 (4)	0.0006 (3)	−0.0196 (4)	−0.0017 (3)
O3	0.0650 (6)	0.0559 (6)	0.0302 (4)	0.0024 (4)	−0.0175 (4)	−0.0038 (4)
N1	0.0456 (5)	0.0311 (5)	0.0406 (5)	−0.0124 (4)	0.0015 (4)	−0.0043 (4)
N2	0.0399 (4)	0.0259 (4)	0.0254 (4)	−0.0040 (3)	−0.0067 (3)	−0.0015 (3)
C1	0.0511 (6)	0.0282 (5)	0.0330 (5)	−0.0082 (4)	−0.0009 (4)	−0.0050 (4)
C2	0.0924 (12)	0.0422 (7)	0.0385 (6)	−0.0147 (7)	0.0118 (7)	−0.0120 (5)
C3	0.1156 (15)	0.0465 (8)	0.0507 (8)	−0.0142 (9)	−0.0023 (9)	−0.0248 (6)

C4	0.0897 (11)	0.0368 (6)	0.0631 (9)	−0.0180 (7)	−0.0139 (8)	−0.0185 (6)
C5	0.0571 (7)	0.0316 (5)	0.0465 (6)	−0.0148 (5)	−0.0066 (5)	−0.0058 (4)
C6	0.0432 (5)	0.0258 (4)	0.0298 (5)	−0.0061 (4)	−0.0052 (4)	−0.0040 (3)
C7	0.0491 (6)	0.0288 (5)	0.0270 (4)	−0.0114 (4)	−0.0062 (4)	−0.0004 (3)
C8	0.0469 (5)	0.0254 (4)	0.0259 (4)	−0.0063 (4)	−0.0116 (4)	−0.0009 (3)
C9	0.0385 (5)	0.0249 (4)	0.0240 (4)	−0.0057 (3)	−0.0091 (3)	−0.0028 (3)
C10	0.0389 (5)	0.0254 (4)	0.0381 (5)	−0.0054 (4)	−0.0111 (4)	−0.0034 (4)
C11	0.0556 (7)	0.0410 (6)	0.0608 (8)	−0.0076 (5)	−0.0281 (6)	−0.0112 (5)
C12	0.0610 (9)	0.0441 (7)	0.1198 (16)	−0.0066 (6)	−0.0511 (10)	−0.0159 (8)
C13	0.0406 (7)	0.0385 (7)	0.1285 (17)	−0.0077 (5)	−0.0178 (9)	−0.0063 (8)
C14	0.0456 (7)	0.0411 (7)	0.0778 (10)	−0.0069 (5)	0.0034 (7)	0.0027 (6)
C15	0.0454 (6)	0.0376 (6)	0.0426 (6)	−0.0069 (5)	−0.0042 (5)	−0.0013 (4)
C16	0.0414 (5)	0.0350 (5)	0.0313 (5)	−0.0071 (4)	−0.0097 (4)	0.0036 (4)
C17	0.0566 (7)	0.0426 (6)	0.0510 (7)	0.0028 (5)	−0.0171 (6)	0.0076 (5)

Geometric parameters (Å, °)

O1—C7	1.2261 (13)	C7—C8	1.5297 (16)
O2—C8	1.4046 (13)	C8—C9	1.5382 (13)
O2—H2	0.83 (2)	C8—H8	0.9800
O3—C16	1.2247 (14)	C9—C10	1.5148 (14)
N1—C7	1.3510 (15)	C9—H9	0.9800
N1—C1	1.4149 (14)	C10—C11	1.3897 (17)
N1—H1	0.89 (2)	C10—C15	1.3905 (16)
N2—C16	1.3637 (14)	C11—C12	1.395 (2)
N2—C6	1.4302 (13)	C11—H11	0.9300
N2—C9	1.4776 (12)	C12—C13	1.378 (3)
C1—C2	1.3886 (17)	C12—H12	0.9300
C1—C6	1.3950 (15)	C13—C14	1.375 (3)
C2—C3	1.381 (2)	C13—H13	0.9300
C2—H2A	0.9300	C14—C15	1.3818 (19)
C3—C4	1.378 (3)	C14—H14	0.9300
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.3814 (19)	C16—C17	1.5031 (17)
C4—H4	0.9300	C17—H17A	0.9600
C5—C6	1.3897 (15)	C17—H17B	0.9600
C5—H5	0.9300	C17—H17C	0.9600
C8—O2—H2	109.8 (14)	N2—C9—C10	111.43 (8)
C7—N1—C1	124.00 (10)	N2—C9—C8	109.66 (8)
C7—N1—H1	114.4 (12)	C10—C9—C8	113.46 (9)
C1—N1—H1	120.4 (12)	N2—C9—H9	107.3
C16—N2—C6	122.88 (9)	C10—C9—H9	107.3
C16—N2—C9	118.55 (8)	C8—C9—H9	107.3
C6—N2—C9	118.41 (8)	C11—C10—C15	119.09 (12)
C2—C1—C6	119.01 (11)	C11—C10—C9	122.52 (10)
C2—C1—N1	120.58 (11)	C15—C10—C9	118.38 (10)
C6—C1—N1	120.40 (10)	C10—C11—C12	119.33 (15)
C3—C2—C1	120.25 (13)	C10—C11—H11	120.3
C3—C2—H2A	119.9	C12—C11—H11	120.3

C1—C2—H2A	119.9	C13—C12—C11	120.92 (16)
C4—C3—C2	120.84 (13)	C13—C12—H12	119.5
C4—C3—H3	119.6	C11—C12—H12	119.5
C2—C3—H3	119.6	C14—C13—C12	119.71 (14)
C3—C4—C5	119.44 (13)	C14—C13—H13	120.1
C3—C4—H4	120.3	C12—C13—H13	120.1
C5—C4—H4	120.3	C13—C14—C15	120.03 (16)
C4—C5—C6	120.37 (12)	C13—C14—H14	120.0
C4—C5—H5	119.8	C15—C14—H14	120.0
C6—C5—H5	119.8	C14—C15—C10	120.91 (14)
C5—C6—C1	120.05 (10)	C14—C15—H15	119.5
C5—C6—N2	121.01 (10)	C10—C15—H15	119.5
C1—C6—N2	118.94 (9)	O3—C16—N2	120.90 (10)
O1—C7—N1	122.08 (11)	O3—C16—C17	121.05 (11)
O1—C7—C8	121.49 (10)	N2—C16—C17	118.04 (10)
N1—C7—C8	116.36 (9)	C16—C17—H17A	109.5
O2—C8—C7	111.68 (8)	C16—C17—H17B	109.5
O2—C8—C9	110.91 (8)	H17A—C17—H17B	109.5
C7—C8—C9	111.53 (9)	C16—C17—H17C	109.5
O2—C8—H8	107.5	H17A—C17—H17C	109.5
C7—C8—H8	107.5	H17B—C17—H17C	109.5
C9—C8—H8	107.5		

C7—N1—C1—C2	−134.65 (15)	C6—N2—C9—C10	−87.10 (10)
C7—N1—C1—C6	45.87 (19)	C16—N2—C9—C8	−145.05 (10)
C6—C1—C2—C3	−1.6 (3)	C6—N2—C9—C8	39.37 (12)
N1—C1—C2—C3	178.87 (17)	O2—C8—C9—N2	173.40 (8)
C1—C2—C3—C4	0.3 (3)	C7—C8—C9—N2	48.24 (11)
C2—C3—C4—C5	0.1 (3)	O2—C8—C9—C10	−61.29 (11)
C3—C4—C5—C6	0.7 (3)	C7—C8—C9—C10	173.56 (8)
C4—C5—C6—C1	−2.0 (2)	N2—C9—C10—C11	84.57 (12)
C4—C5—C6—N2	177.86 (14)	C8—C9—C10—C11	−39.79 (14)
C2—C1—C6—C5	2.5 (2)	N2—C9—C10—C15	−94.64 (11)
N1—C1—C6—C5	−178.04 (12)	C8—C9—C10—C15	140.99 (10)
C2—C1—C6—N2	−177.42 (13)	C15—C10—C11—C12	0.26 (18)
N1—C1—C6—N2	2.06 (18)	C9—C10—C11—C12	−178.95 (11)
C16—N2—C6—C5	−67.48 (16)	C10—C11—C12—C13	0.0 (2)
C9—N2—C6—C5	107.89 (12)	C11—C12—C13—C14	−0.5 (2)
C16—N2—C6—C1	112.42 (13)	C12—C13—C14—C15	0.7 (2)
C9—N2—C6—C1	−72.21 (14)	C13—C14—C15—C10	−0.4 (2)
C1—N1—C7—O1	−178.75 (11)	C11—C10—C15—C14	−0.10 (18)
C1—N1—C7—C8	4.08 (17)	C9—C10—C15—C14	179.14 (11)
O1—C7—C8—O2	−18.12 (14)	C6—N2—C16—O3	175.01 (10)
N1—C7—C8—O2	159.07 (10)	C9—N2—C16—O3	−0.35 (16)
O1—C7—C8—C9	106.61 (11)	C6—N2—C16—C17	−6.10 (16)
N1—C7—C8—C9	−76.20 (12)	C9—N2—C16—C17	178.53 (10)
C16—N2—C9—C10	88.48 (11)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.89 (2)	2.04 (2)	2.924 (1)	175 (2)
O2—H2···O3^{ii}	0.83 (2)	2.09 (2)	2.905 (1)	168 (2)

Symmetry codes: (i) $-x$, $-y+1$, $-z+1$; (ii) $-x+1$, $-y+1$, $-z$.

Fig. 1

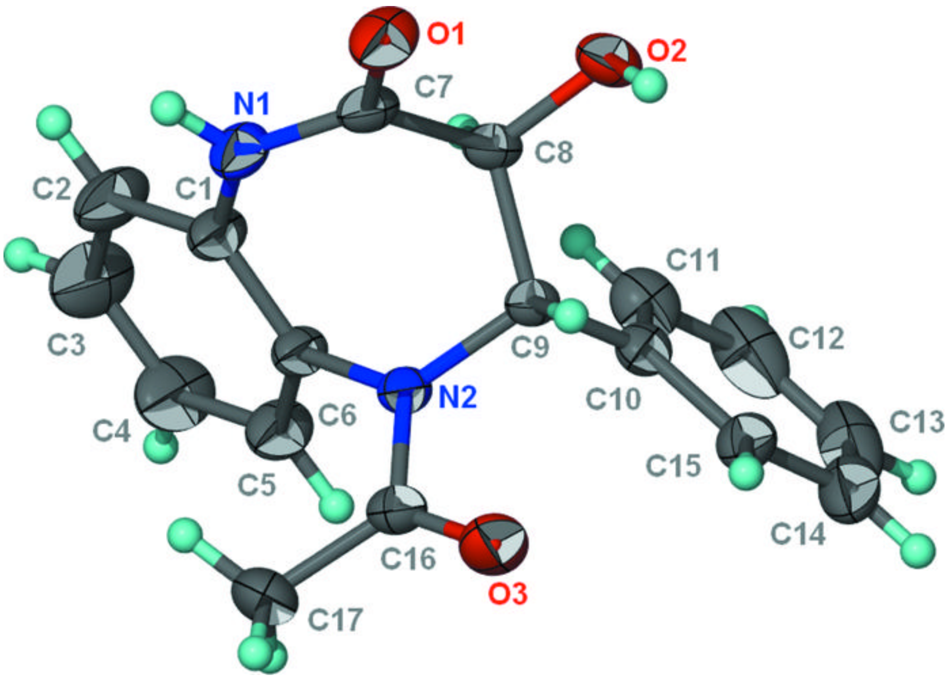
O1
O2
N1
C7
C8
C2
C1
C11
C3
C9
C12
C10
N2
C6
C13
C4
C15
C5
C14
C16
O3
C17

Fig. 2