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Ethyl 2-(2-oxo-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-1-yl)acetate

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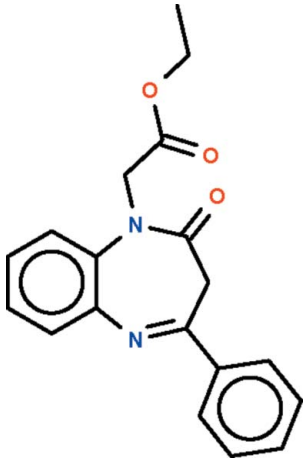
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.114; data-to-parameter ratio = 14.0.

The seven-membered ring in the title compound, $C_{19}H_{18}N_2O_3$, adopts a boat conformation with the two phenylene C atoms representing the stern and the methylene C atom the prow. The dihedral angle between the best plane through the seven-membered ring (r.m.s deviation = 0.343 Å) and the phenyl substituent is 31.9 (1)°. The dihedral angle between this best plane and the best plane through the ethoxycarbonylmethyl substituent (r.m.s. deviation = 0.058 Å) is 72.2 (1)°.

Related literature

For the background to 2,3-dihydro-1*H*-1,5-benzodiazepin-2-ones, see: Ahabchane *et al.* (1999). For a related structure, see: Ballo *et al.* (2010).



Experimental

Crystal data

$C_{19}H_{18}N_2O_3$
M_r = 322.35
Monoclinic, $P2_1/c$
a = 12.5198 (4) Å
b = 11.7911 (3) Å
c = 11.2058 (3) Å
β = 97.843 (2)°
V = 1638.75 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.30 × 0.15 × 0.10 mm

Data collection

Bruker X8 APEXII diffractometer
13943 measured reflections
3029 independent reflections
2195 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.114
S = 1.00
3029 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2051).

References

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supplementary materials

Acta Cryst. (2010). E**66**, o2070 [**doi:10.1107/S1600536810028278**]

Ethyl 2-(2-oxo-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-1-yl)acetate

D. Ballo, N. H. Ahabchane, H. Zouihri, E. M. Essassi and S. W. Ng

Comment

The background to the class of 2,3-dihydro-1*H*-1,5-benzodiazepin-2-ones is given in an earlier report (Ahabchane *et al.*, 1999). A recent study presents the crystal structure of 1-allyl-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-2-one (Ballo *et al.*, 2010). The present study has an ethoxycarbonylmethyl group in place of the allyl group (Scheme I, Fig. 1). The principal feature is the seven-membered ring that is fused to a phenylene ring. This ring adopts a boat-shaped conformation, two phenylene carbons representing the stern and the methylene carbon atom the prow [r.m.s deviation 0.343 Å]. The methyl carbon deviates by 0.604 Å from the best plane.

Experimental

To a solution of 4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-2-one (1 g, 4.2 mmol) in DMF (20 ml) was added ethyl chloro-acetate (0.5 g, 4.2 mmol), potassium carbonate (1 g, 7.4 mmol) and a catalytic quantity of tetra-*n*-butylammonium bromide. The mixture was stirred at room temperature for 24 h. The solution was filtered and the solvent removed under reduced pressure. The residue was recrystallized from ethanol to afford the title compound as yellow crystals.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2–1.5U_{eq}(C).

Figures

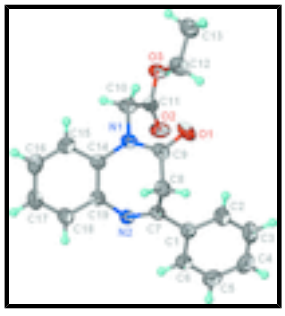


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of the molecule of $C_{19}H_{18}N_2O_3$ at the 50% probability level.

Ethyl 2-(2-oxo-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-1-yl)acetate

Crystal data

$C_{19}H_{18}N_2O_3$	$F(000) = 680$
$M_r = 322.35$	$D_x = 1.307$ Mg m^{-3}

Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3291 reflections
$a = 12.5198$ (4) Å	$\theta = 2.4–23.1°$
$b = 11.7911$ (3) Å	$\mu = 0.09$ mm^{-1}
$c = 11.2058$ (3) Å	$T = 293$ K
$\beta = 97.843$ (2)°	Prism, yellow
$V = 1638.75$ (8) Å^3	0.30 × 0.15 × 0.10 mm
$Z = 4$	

Data collection

Bruker X8 APEXII diffractometer	2195 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.033$
graphite	$\theta_{max} = 25.5°$, $\theta_{min} = 2.5°$
φ and ω scans	$h = -15\rightarrow15$
13943 measured reflections	$k = -14\rightarrow14$
3029 independent reflections	$l = -13\rightarrow13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.037$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.114$	H-atom parameters constrained
$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
3029 reflections	$(\Delta/\sigma)_{max} = 0.001$
217 parameters	$\Delta\rho_{max} = 0.12$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.15$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.45383 (9)	0.67927 (10)	0.34251 (11)	0.0662 (4)
O2	0.43414 (9)	0.64804 (9)	0.06491 (11)	0.0604 (3)
O3	0.60915 (8)	0.69533 (8)	0.08966 (10)	0.0488 (3)
N1	0.39095 (10)	0.82128 (10)	0.21765 (12)	0.0460 (3)
N2	0.16060 (9)	0.76218 (10)	0.14862 (11)	0.0438 (3)
C1	0.15443 (12)	0.57820 (13)	0.23365 (13)	0.0452 (4)
C2	0.21662 (14)	0.49224 (14)	0.29222 (16)	0.0599 (5)
H2	0.2822	0.5097	0.3381	0.072*
C3	0.18218 (17)	0.38119 (15)	0.28306 (18)	0.0690 (5)
H3	0.2253	0.3242	0.3214	0.083*
C4	0.08518 (16)	0.35429 (14)	0.21804 (18)	0.0648 (5)
H4	0.0618	0.2793	0.2132	0.078*

C5	0.02215 (14)	0.43823 (15)	0.15974 (18)	0.0631 (5)
H5	−0.0440	0.4202	0.1156	0.076*
C6	0.05711 (13)	0.54944 (13)	0.16671 (15)	0.0525 (4)
H6	0.0147	0.6057	0.1259	0.063*
C7	0.19264 (11)	0.69709 (12)	0.23782 (13)	0.0423 (4)
C8	0.27085 (12)	0.73756 (13)	0.34441 (13)	0.0483 (4)
H8A	0.2497	0.8118	0.3702	0.058*
H8B	0.2715	0.6853	0.4113	0.058*
C9	0.38018 (12)	0.74323 (13)	0.30496 (14)	0.0478 (4)
C10	0.49294 (13)	0.81974 (13)	0.16772 (16)	0.0536 (4)
H10A	0.5523	0.8288	0.2323	0.064*
H10B	0.4948	0.8828	0.1124	0.064*
C11	0.50622 (12)	0.71043 (12)	0.10251 (13)	0.0431 (4)
C12	0.63354 (14)	0.59312 (14)	0.02606 (16)	0.0592 (5)
H12A	0.6050	0.5992	−0.0586	0.071*
H12B	0.6010	0.5277	0.0593	0.071*
C13	0.75293 (14)	0.58019 (15)	0.04048 (16)	0.0669 (5)
H13A	0.7712	0.5132	−0.0011	0.100*
H13B	0.7803	0.5736	0.1245	0.100*
H13C	0.7843	0.6453	0.0074	0.100*
C14	0.31063 (11)	0.90246 (12)	0.17520 (12)	0.0414 (4)
C15	0.34293 (13)	1.01399 (12)	0.15912 (14)	0.0511 (4)
H15	0.4153	1.0333	0.1776	0.061*
C16	0.26962 (15)	1.09539 (14)	0.11653 (16)	0.0595 (5)
H16	0.2924	1.1694	0.1066	0.071*
C17	0.16234 (15)	1.06815 (14)	0.08833 (15)	0.0596 (5)
H17	0.1125	1.1238	0.0604	0.072*
C18	0.12908 (13)	0.95835 (13)	0.10160 (14)	0.0528 (4)
H18	0.0566	0.9402	0.0810	0.063*
C19	0.20182 (12)	0.87328 (12)	0.14543 (12)	0.0418 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0551 (8)	0.0761 (8)	0.0638 (8)	0.0232 (6)	−0.0050 (6)	0.0014 (6)
O2	0.0439 (7)	0.0632 (7)	0.0729 (8)	−0.0078 (6)	0.0036 (5)	−0.0194 (6)
O3	0.0384 (6)	0.0511 (6)	0.0573 (7)	0.0031 (4)	0.0083 (5)	−0.0115 (5)
N1	0.0373 (7)	0.0501 (7)	0.0512 (8)	0.0040 (5)	0.0081 (6)	−0.0036 (6)
N2	0.0392 (7)	0.0486 (7)	0.0432 (7)	0.0003 (5)	0.0037 (5)	0.0044 (6)
C1	0.0456 (9)	0.0518 (9)	0.0405 (9)	0.0050 (7)	0.0138 (7)	0.0039 (7)
C2	0.0617 (11)	0.0583 (11)	0.0589 (11)	0.0074 (8)	0.0049 (8)	0.0115 (8)
C3	0.0826 (14)	0.0581 (11)	0.0687 (12)	0.0148 (10)	0.0184 (10)	0.0180 (9)
C4	0.0781 (13)	0.0476 (10)	0.0750 (13)	−0.0020 (9)	0.0333 (10)	0.0026 (9)
C5	0.0538 (11)	0.0612 (11)	0.0772 (13)	−0.0070 (8)	0.0189 (9)	−0.0040 (9)
C6	0.0464 (9)	0.0532 (9)	0.0594 (10)	0.0026 (7)	0.0123 (7)	0.0050 (8)
C7	0.0364 (8)	0.0518 (9)	0.0398 (8)	0.0053 (6)	0.0088 (6)	0.0034 (7)
C8	0.0523 (10)	0.0549 (9)	0.0372 (8)	0.0053 (7)	0.0047 (7)	0.0028 (7)
C9	0.0441 (9)	0.0546 (9)	0.0421 (9)	0.0077 (7)	−0.0031 (7)	−0.0086 (7)

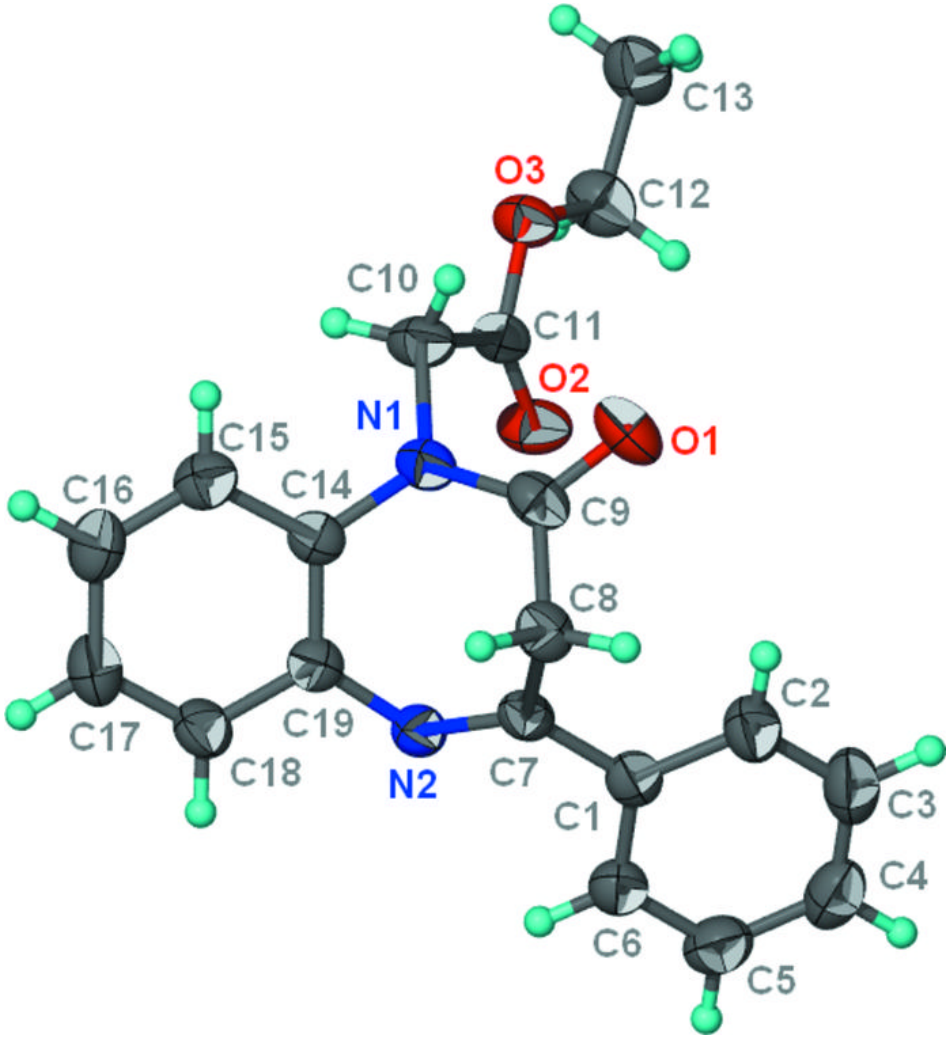
C10	0.0390 (9)	0.0538 (9)	0.0696 (11)	−0.0012 (7)	0.0129 (8)	−0.0137 (8)
C11	0.0364 (8)	0.0487 (8)	0.0433 (9)	0.0009 (7)	0.0025 (6)	−0.0019 (7)
C12	0.0610 (11)	0.0597 (10)	0.0570 (11)	0.0105 (8)	0.0082 (8)	−0.0162 (8)
C13	0.0651 (12)	0.0721 (12)	0.0681 (12)	0.0205 (9)	0.0253 (9)	0.0010 (9)
C14	0.0413 (8)	0.0456 (8)	0.0383 (8)	0.0031 (6)	0.0085 (6)	−0.0049 (6)
C15	0.0521 (10)	0.0487 (9)	0.0536 (10)	−0.0049 (7)	0.0108 (7)	−0.0090 (7)
C16	0.0766 (13)	0.0426 (9)	0.0599 (11)	−0.0012 (8)	0.0115 (9)	−0.0004 (8)
C17	0.0711 (12)	0.0516 (10)	0.0552 (10)	0.0119 (9)	0.0053 (9)	0.0088 (8)
C18	0.0480 (9)	0.0592 (10)	0.0498 (10)	0.0056 (7)	0.0019 (7)	0.0078 (8)
C19	0.0430 (8)	0.0473 (8)	0.0349 (8)	0.0009 (7)	0.0050 (6)	−0.0007 (6)

Geometric parameters (Å, °)

O1—C9	1.2207 (17)	C8—C9	1.496 (2)
O2—C11	1.1955 (17)	C8—H8A	0.9700
O3—C11	1.3281 (17)	C8—H8B	0.9700
O3—C12	1.4537 (17)	C10—C11	1.502 (2)
N1—C9	1.363 (2)	C10—H10A	0.9700
N1—C14	1.4224 (17)	C10—H10B	0.9700
N1—C10	1.4625 (19)	C12—C13	1.489 (2)
N2—C7	1.2805 (18)	C12—H12A	0.9700
N2—C19	1.4102 (18)	C12—H12B	0.9700
C1—C6	1.383 (2)	C13—H13A	0.9600
C1—C2	1.387 (2)	C13—H13B	0.9600
C1—C7	1.480 (2)	C13—H13C	0.9600
C2—C3	1.378 (2)	C14—C15	1.395 (2)
C2—H2	0.9300	C14—C19	1.4005 (19)
C3—C4	1.366 (3)	C15—C16	1.368 (2)
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.374 (2)	C16—C17	1.375 (2)
C4—H4	0.9300	C16—H16	0.9300
C5—C6	1.381 (2)	C17—C18	1.374 (2)
C5—H5	0.9300	C17—H17	0.9300
C6—H6	0.9300	C18—C19	1.398 (2)
C7—C8	1.515 (2)	C18—H18	0.9300
C11—O3—C12	115.86 (12)	C11—C10—H10A	109.5
C9—N1—C14	124.03 (13)	N1—C10—H10B	109.5
C9—N1—C10	116.31 (12)	C11—C10—H10B	109.5
C14—N1—C10	119.65 (12)	H10A—C10—H10B	108.0
C7—N2—C19	119.99 (13)	O2—C11—O3	125.19 (14)
C6—C1—C2	118.26 (15)	O2—C11—C10	124.83 (14)
C6—C1—C7	120.43 (14)	O3—C11—C10	109.95 (12)
C2—C1—C7	121.26 (14)	O3—C12—C13	107.85 (13)
C3—C2—C1	120.61 (17)	O3—C12—H12A	110.1
C3—C2—H2	119.7	C13—C12—H12A	110.1
C1—C2—H2	119.7	O3—C12—H12B	110.1
C4—C3—C2	120.46 (17)	C13—C12—H12B	110.1
C4—C3—H3	119.8	H12A—C12—H12B	108.4
C2—C3—H3	119.8	C12—C13—H13A	109.5

C3—C4—C5	119.83 (17)	C12—C13—H13B	109.5
C3—C4—H4	120.1	H13A—C13—H13B	109.5
C5—C4—H4	120.1	C12—C13—H13C	109.5
C4—C5—C6	119.99 (17)	H13A—C13—H13C	109.5
C4—C5—H5	120.0	H13B—C13—H13C	109.5
C6—C5—H5	120.0	C15—C14—C19	119.38 (13)
C5—C6—C1	120.83 (16)	C15—C14—N1	118.26 (13)
C5—C6—H6	119.6	C19—C14—N1	122.32 (13)
C1—C6—H6	119.6	C16—C15—C14	120.93 (16)
N2—C7—C1	118.53 (13)	C16—C15—H15	119.5
N2—C7—C8	121.78 (13)	C14—C15—H15	119.5
C1—C7—C8	119.65 (13)	C15—C16—C17	120.26 (15)
C9—C8—C7	107.47 (12)	C15—C16—H16	119.9
C9—C8—H8A	110.2	C17—C16—H16	119.9
C7—C8—H8A	110.2	C18—C17—C16	119.74 (16)
C9—C8—H8B	110.2	C18—C17—H17	120.1
C7—C8—H8B	110.2	C16—C17—H17	120.1
H8A—C8—H8B	108.5	C17—C18—C19	121.43 (16)
O1—C9—N1	121.36 (15)	C17—C18—H18	119.3
O1—C9—C8	123.30 (15)	C19—C18—H18	119.3
N1—C9—C8	115.23 (13)	C18—C19—C14	118.24 (13)
N1—C10—C11	110.92 (12)	C18—C19—N2	116.89 (13)
N1—C10—H10A	109.5	C14—C19—N2	124.72 (13)
C6—C1—C2—C3	0.4 (2)	C14—N1—C10—C11	−116.19 (14)
C7—C1—C2—C3	−177.05 (15)	C12—O3—C11—O2	−1.0 (2)
C1—C2—C3—C4	−1.3 (3)	C12—O3—C11—C10	−179.08 (14)
C2—C3—C4—C5	1.0 (3)	N1—C10—C11—O2	20.5 (2)
C3—C4—C5—C6	0.2 (3)	N1—C10—C11—O3	−161.39 (13)
C4—C5—C6—C1	−1.1 (3)	C11—O3—C12—C13	−169.68 (13)
C2—C1—C6—C5	0.9 (2)	C9—N1—C14—C15	135.77 (15)
C7—C1—C6—C5	178.28 (15)	C10—N1—C14—C15	−43.45 (18)
C19—N2—C7—C1	−175.16 (12)	C9—N1—C14—C19	−46.5 (2)
C19—N2—C7—C8	2.8 (2)	C10—N1—C14—C19	134.31 (15)
C6—C1—C7—N2	−26.6 (2)	C19—C14—C15—C16	1.0 (2)
C2—C1—C7—N2	150.72 (15)	N1—C14—C15—C16	178.85 (13)
C6—C1—C7—C8	155.40 (14)	C14—C15—C16—C17	−0.2 (2)
C2—C1—C7—C8	−27.3 (2)	C15—C16—C17—C18	−0.8 (3)
N2—C7—C8—C9	−74.71 (17)	C16—C17—C18—C19	1.2 (3)
C1—C7—C8—C9	103.20 (15)	C17—C18—C19—C14	−0.4 (2)
C14—N1—C9—O1	−176.16 (13)	C17—C18—C19—N2	−176.23 (14)
C10—N1—C9—O1	3.1 (2)	C15—C14—C19—C18	−0.7 (2)
C14—N1—C9—C8	7.5 (2)	N1—C14—C19—C18	−178.44 (13)
C10—N1—C9—C8	−173.24 (12)	C15—C14—C19—N2	174.79 (14)
C7—C8—C9—O1	−111.25 (16)	N1—C14—C19—N2	−2.9 (2)
C7—C8—C9—N1	65.00 (16)	C7—N2—C19—C18	−140.71 (15)
C9—N1—C10—C11	64.53 (18)	C7—N2—C19—C14	43.7 (2)

Fig. 1


C13
O3
C12
C10
C11
O2
N1
O1
C15
C16
C14
C9
C8
C2
C17
C19
C18
N2
C7
C1
C3
C4
C6
C5