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

Abdelhanine Essaghouani,[a]* Younos Bouzian,[a] El Mokhtar Essassi,[a] Mohamed Saadi[b] and Lahcen El Ammari[b]

[a]Laboratoire de Chimie Organique Hétérocyclique URAC 21, Pôle de Compétences Pharmacochimie, Mohammed V University in Rabat, BP 1014 Avenue Ibn Battouta, Rabat, Morocco, and [b]Laboratoire de Chimie du Solide Appliquée, Faculty of Sciences, Mohammed V University in Rabat, Avenue Ibn Battouta, BP 1014, Rabat, Morocco. *Correspondence e-mail: aessaghouani@gmail.com

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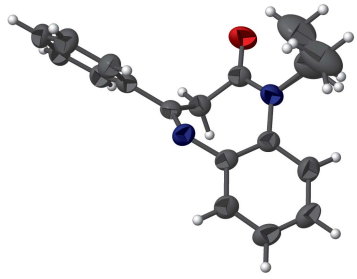
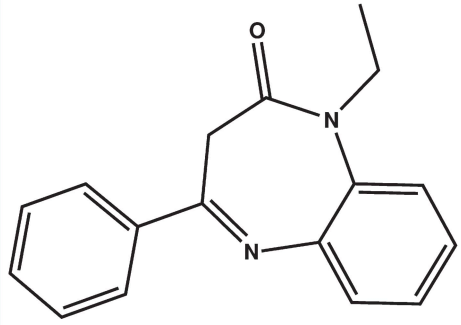
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{17}H_{16}N_2O$, consists of a benzodiazepin-2-one moiety substituted with a phenyl ring and an ethyl group. The seven-membered diazepine ring has a boat conformation and the fused benzene ring is nearly perpendicular to the phenyl ring, as indicated by the dihedral angle of 74.90 (8)°. The atoms of the ethyl group are disordered over two sets of sites, with a refined occupancy ratio of 0.603 (15):0.397 (15). In the crystal, molecules are linked by pairs of C—H···O hydrogen bonds, forming inversion dimers. The dimers are linked *via* a further C—H···O hydrogen bond, forming layers parallel to (001), which are in turn linked by C—H···π interactions, forming a three-dimensional structure.

3D view



Chemical scheme



Structure description

Benzodiazepines are very important compounds widely used as psychotropic agents (Zellou *et al.*, 1998*a*; Kanyonga *et al.*, 2009) and hypnotic agents (Zellou *et al.*, 1998*b*). Background to the class of 2,3-dihydro-1*H*-1,5-benzodiazepin-2-ones is given by Ahabchane *et al.* (2001). Continuing our interest in the synthesis of new 1,5-benzodiazepin-2-one derivatives, we report herein on the synthesis and crystal structure of the title compound, obtained under phase-transfer catalysis conditions.

The title compound, Fig. 1, contains a benzodiazepin-2-one moiety, which is linked to a phenyl ring (C10–C15) and to an ethyl group (C6–C7). The seven-membered diazepine ring displays a boat conformation, as indicated by the total puckering amplitude Q_T = 0.918 (2) Å, and spherical polar angle θ = 75.5 (1)°, with φ_2 = 129.4 (2)° and $\varphi 3$ = −77.0 (4)°. The dihedral angle between the two aromatic ring (C1–C6 and C10–C15) is 74.90 (8)°.

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

*Cg*1 is the centroid of the phenyl ring (C10–C15).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1^{i}	0.93	2.55	3.464 (2)	168
C13—H13···O1^{ii}	0.93	2.52	3.412 (2)	162
C4—H4···*Cg*1^{iii}	0.93	2.80	3.568 (1)	140

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

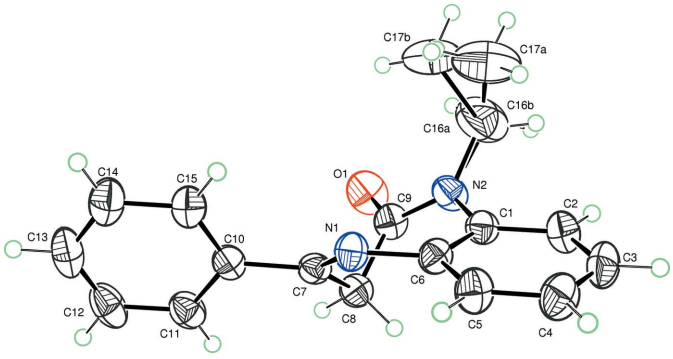


Figure 1
Molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

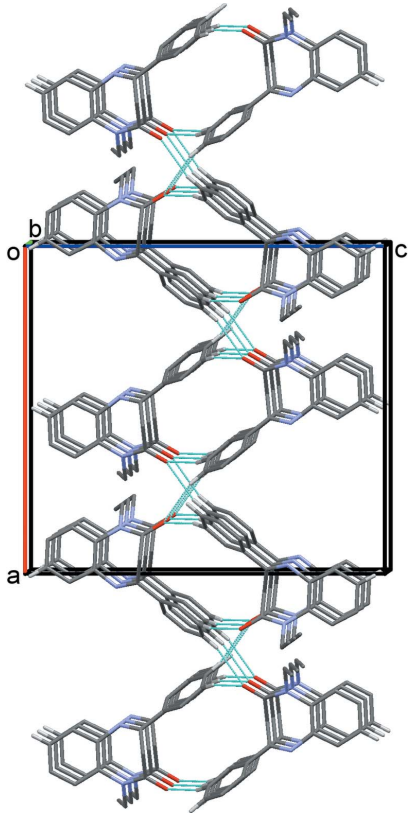


Figure 2
A view along the *b* axis of the crystal packing of the title compound. C—H···O hydrogen bonds are shown as dashed lines (see Table 1); H atoms not involved in these interactions have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{16}N_2O$
M_r	264.32
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	296
a, *b*, *c* (Å)	16.5042 (4), 9.6896 (3), 18.1221 (5)
V (Å^3)	2898.07 (14)
Z	8
Radiation type	Mo *Kα*
μ (mm^{-1})	0.08
Crystal size (mm)	0.35 × 0.31 × 0.22
Data collection	
Diffractometer	Bruker X8 *APEX*
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.626, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	31360, 3199, 2418
R_{int}	0.036
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.641
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.048, 0.146, 1.05
No. of reflections	3199
No. of parameters	201
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.38, −0.18

Computer programs: *APEX2* and *SAINT-Plus* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015)*ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3* for Windoes (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

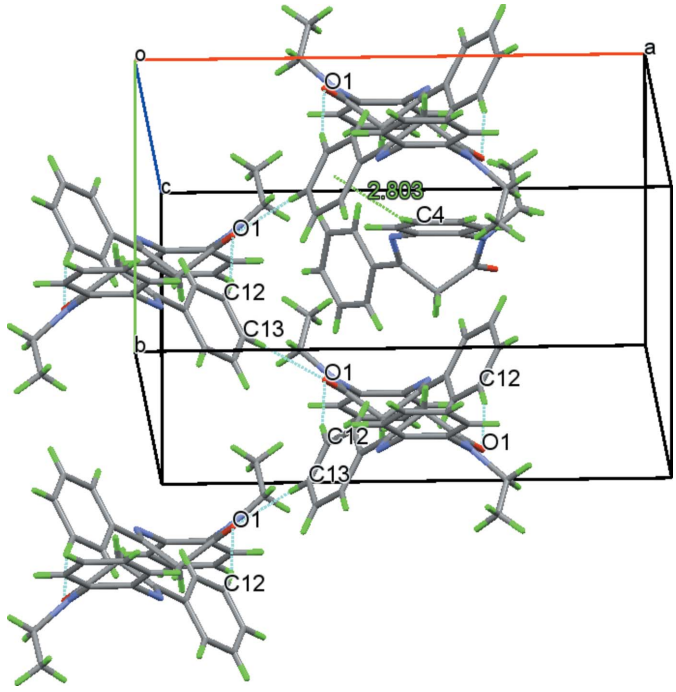


Figure 3
A partial view of the crystal packing of the title compound, with the C—H···O hydrogen bonds and C—H···π interactions shown as dashed lines (see Table 1).

In the crystal, molecules are linked by C12—H12···O1 and C13—H13···O1 hydrogen bonds involving the same acceptor atom, forming layers parallel to the *ab* plane (Fig. 2 and Table 1). The layers are connected by C4—-H4···π interactions, building a three-dimensional structure (Fig. 3 and Table 1).

Synthesis and crystallization

To a solution of 4-phenyl-1, 5-benzodiazepin-2-one (2.36 g, 10 mmol) in DMF (40 ml) was added ethyl bromide (2.16 g, 20 mmol), potassium carbonate (2.77 g, 20 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 colourless crystals (yield 70%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The atoms of the ethyl group (C6 and C7) are disordered over two sets of sites (C6*A*/C6*B* and C7*A*/C7*B*), with a refined occupancy ratio of 0.603 (15): 0.397 (15).

Acknowledgements

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full crystallographic data

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Crystal data

$C_{17}H_{16}N_2O$
M_r = 264.32
Orthorhombic, *Pbca*
a = 16.5042 (4) Å
b = 9.6896 (3) Å
c = 18.1221 (5) Å
V = 2898.07 (14) Å^3
Z = 8
F(000) = 1120
D_x = 1.212 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3199 reflections
θ = 2.6–27.1°
μ = 0.08 mm^{-1}
T = 296 K
Block, colourless
0.35 × 0.31 × 0.22 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.626, T_{max} = 0.746
31360 measured reflections
3199 independent reflections
2418 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.1°, θ_{min} = 2.6°
h = −21→21
k = −12→12
l = −23→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.146
S = 1.05
3199 reflections
201 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 0.8641P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0023 (7)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.39886 (10)	0.78881 (16)	0.79928 (8)	0.0422 (4)	
C2	0.35482 (11)	0.7994 (2)	0.86484 (9)	0.0539 (4)	
H2	0.2985	0.7990	0.8631	0.065*	
C3	0.39311 (12)	0.8103 (2)	0.93194 (9)	0.0577 (5)	
H3	0.3627	0.8172	0.9750	0.069*	
C4	0.47671 (12)	0.8111 (2)	0.93555 (9)	0.0578 (5)	
H4	0.5028	0.8200	0.9808	0.069*	
C5	0.52080 (11)	0.79865 (19)	0.87187 (9)	0.0511 (4)	
H5	0.5771	0.7972	0.8747	0.061*	
C6	0.48392 (10)	0.78807 (15)	0.80279 (8)	0.0402 (3)	
C7	0.52244 (9)	0.83690 (15)	0.68217 (8)	0.0382 (3)	
C8	0.45039 (9)	0.93206 (16)	0.67409 (9)	0.0434 (4)	
H8A	0.4461	0.9925	0.7166	0.052*	
H8B	0.4559	0.9884	0.6301	0.052*	
C9	0.37702 (10)	0.84114 (18)	0.66843 (9)	0.0473 (4)	
C10	0.57784 (9)	0.81512 (17)	0.61856 (8)	0.0417 (4)	
C11	0.59420 (10)	0.9202 (2)	0.56865 (9)	0.0524 (4)	
H11	0.5685	1.0051	0.5736	0.063*	
C12	0.64874 (11)	0.8993 (2)	0.51135 (10)	0.0628 (5)	
H12	0.6602	0.9707	0.4787	0.075*	
C13	0.68578 (11)	0.7736 (3)	0.50286 (10)	0.0653 (6)	
H13	0.7219	0.7596	0.4642	0.078*	
C14	0.66943 (11)	0.6682 (2)	0.55163 (11)	0.0629 (5)	
H14	0.6943	0.5828	0.5455	0.075*	
C15	0.61633 (10)	0.68835 (19)	0.60955 (9)	0.0507 (4)	
H15	0.6063	0.6170	0.6426	0.061*	
C16A	0.2855 (6)	0.6691 (11)	0.7269 (5)	0.117 (4)	0.603 (15)
H16A	0.2414	0.7031	0.7572	0.140*	0.603 (15)
H16B	0.2663	0.6665	0.6763	0.140*	0.603 (15)
C17A	0.3022 (5)	0.5430 (7)	0.7474 (8)	0.115 (3)	0.603 (15)
H17A	0.2546	0.4867	0.7426	0.172*	0.603 (15)
H17B	0.3446	0.5065	0.7169	0.172*	0.603 (15)
H17C	0.3196	0.5432	0.7980	0.172*	0.603 (15)
C16B	0.2849 (3)	0.6870 (8)	0.7291 (4)	0.0355 (18)	0.397 (15)
H16C	0.2429	0.7284	0.6989	0.043*	0.397 (15)
H16D	0.2637	0.6713	0.7783	0.043*	0.397 (15)
C17B	0.3180 (5)	0.5401 (8)	0.6917 (10)	0.097 (4)	0.397 (15)
H17D	0.2740	0.4758	0.6881	0.146*	0.397 (15)
H17E	0.3393	0.5583	0.6434	0.146*	0.397 (15)

H17F	0.3599	0.5017	0.7222	0.146*	0.397 (15)
N1	0.53607 (8)	0.77094 (14)	0.74201 (7)	0.0431 (3)	
N2	0.35601 (8)	0.77348 (15)	0.73142 (7)	0.0493 (4)	
O1	0.34016 (9)	0.82593 (16)	0.61053 (7)	0.0717 (4)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0481 (8)	0.0429 (8)	0.0355 (8)	−0.0032 (7)	−0.0018 (6)	0.0032 (6)
C2	0.0507 (9)	0.0656 (11)	0.0455 (9)	−0.0018 (8)	0.0060 (7)	0.0068 (8)
C3	0.0683 (11)	0.0695 (12)	0.0354 (8)	0.0053 (9)	0.0100 (8)	0.0034 (8)
C4	0.0705 (12)	0.0707 (12)	0.0321 (8)	0.0021 (9)	−0.0064 (8)	0.0023 (8)
C5	0.0493 (9)	0.0662 (11)	0.0377 (9)	0.0035 (8)	−0.0054 (7)	0.0056 (8)
C6	0.0481 (8)	0.0397 (8)	0.0328 (7)	0.0034 (6)	−0.0018 (6)	0.0029 (6)
C7	0.0423 (8)	0.0382 (8)	0.0342 (7)	−0.0048 (6)	−0.0035 (6)	−0.0004 (6)
C8	0.0506 (9)	0.0404 (8)	0.0392 (8)	0.0016 (7)	−0.0013 (6)	0.0057 (6)
C9	0.0462 (9)	0.0561 (10)	0.0398 (8)	0.0007 (7)	−0.0067 (7)	0.0027 (7)
C10	0.0402 (8)	0.0521 (9)	0.0327 (7)	−0.0070 (7)	−0.0043 (6)	0.0007 (6)
C11	0.0536 (9)	0.0589 (10)	0.0445 (9)	−0.0093 (8)	−0.0014 (7)	0.0072 (8)
C12	0.0521 (10)	0.0957 (16)	0.0405 (9)	−0.0206 (10)	−0.0021 (8)	0.0151 (9)
C13	0.0397 (9)	0.1147 (18)	0.0415 (9)	−0.0090 (11)	0.0041 (7)	−0.0050 (10)
C14	0.0498 (10)	0.0829 (14)	0.0559 (11)	0.0075 (9)	0.0058 (8)	−0.0085 (10)
C15	0.0473 (9)	0.0604 (10)	0.0445 (9)	0.0027 (8)	0.0036 (7)	0.0011 (8)
C16A	0.164 (8)	0.112 (7)	0.074 (5)	−0.057 (5)	−0.007 (4)	0.013 (5)
C17A	0.114 (4)	0.078 (3)	0.153 (9)	−0.016 (3)	−0.053 (6)	0.006 (4)
C16B	0.013 (2)	0.043 (3)	0.050 (4)	−0.0084 (17)	−0.0139 (19)	0.006 (3)
C17B	0.105 (5)	0.056 (4)	0.131 (9)	−0.012 (3)	−0.027 (6)	−0.012 (5)
N1	0.0454 (7)	0.0496 (7)	0.0342 (7)	0.0037 (6)	−0.0002 (5)	0.0029 (6)
N2	0.0489 (7)	0.0574 (9)	0.0414 (7)	−0.0125 (6)	−0.0063 (6)	0.0044 (6)
O1	0.0691 (9)	0.1001 (11)	0.0459 (7)	−0.0143 (7)	−0.0212 (6)	0.0101 (7)

Geometric parameters (Å, º)

C1—C2	1.397 (2)	C9—O1	1.2217 (19)
C1—C6	1.405 (2)	C9—N2	1.361 (2)
C1—N2	1.426 (2)	C10—C11	1.389 (2)
C2—C3	1.375 (2)	C10—C15	1.393 (2)
C3—C4	1.381 (3)	C11—C12	1.389 (3)
C4—C5	1.370 (2)	C12—C13	1.371 (3)
C5—C6	1.396 (2)	C13—C14	1.378 (3)
C6—N1	1.408 (2)	C14—C15	1.381 (2)
C7—N1	1.2787 (19)	C16A—C17A	1.307 (11)
C7—C10	1.486 (2)	C16A—N2	1.544 (10)
C7—C8	1.512 (2)	C16B—N2	1.442 (6)
C8—C9	1.501 (2)	C16B—C17B	1.669 (13)
C2—C1—C6	118.80 (14)	C11—C10—C15	118.81 (15)
C2—C1—N2	118.88 (15)	C11—C10—C7	121.38 (15)

C6—C1—N2	122.27 (13)	C15—C10—C7	119.78 (14)
C3—C2—C1	121.26 (16)	C10—C11—C12	120.37 (18)
C2—C3—C4	120.11 (16)	C13—C12—C11	120.18 (18)
C5—C4—C3	119.35 (16)	C12—C13—C14	119.95 (17)
C4—C5—C6	122.04 (16)	C13—C14—C15	120.46 (19)
C5—C6—C1	118.42 (14)	C14—C15—C10	120.22 (17)
C5—C6—N1	116.34 (14)	C17A—C16A—N2	116.0 (7)
C1—C6—N1	125.17 (13)	N2—C16B—C17B	103.9 (5)
N1—C7—C10	118.59 (13)	C7—N1—C6	119.81 (13)
N1—C7—C8	121.69 (14)	C9—N2—C1	123.13 (13)
C10—C7—C8	119.69 (13)	C9—N2—C16B	117.6 (3)
C9—C8—C7	106.45 (13)	C1—N2—C16B	119.3 (3)
O1—C9—N2	122.36 (15)	C9—N2—C16A	117.6 (4)
O1—C9—C8	122.08 (15)	C1—N2—C16A	119.2 (4)
N2—C9—C8	115.52 (13)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the phenyl ring (C10–C15).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1^{i}	0.93	2.55	3.464 (2)	168
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Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1; (ii) *x*+1/2, −*y*+3/2, −*z*+1; (iii) *x*, −*y*+3/2, *z*+1/2.