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2-[2-(4-Methoxyphenyl)-2,3-dihydro-1*H*-1,5-benzodiazepin-4-yl]phenol

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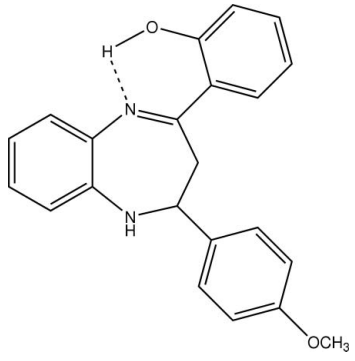
Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.003 Å; R factor = 0.055; wR factor = 0.065; data-to-parameter ratio = 10.7.

In the structure of title compound, $C_{22}H_{20}O_2N_2$, the 11-membered benzodiazepine ring system adopts a distorted boat conformation. The benzene ring of this system forms dihedral angles of 89.69 (12) and 48.82 (12)° with those of the phenol and methoxyphenyl substituents, respectively. The dihedral angle between the benzene rings is 49.61 (11)°. An intramolecular O—H···N hydrogen bond generates an S(6) ring.

Related literature

For the biological activity of heterocyclic scaffolds containing nitrogen atoms, see: MacDonald (2002); Gringauz (1999); Albright *et al.* (1998); Rahbaek *et al.* (1999). For related structures, see: Ravichandran *et al.* (2009*a*,*b*,*c*,*d*). For puckering parameters, see: Cremer & Pople (1975). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the weighting scheme, see: Prince (1982); Watkin (1994).



Experimental

Crystal data

$C_{22}H_{20}N_2O_2$
M_r = 344.41
Monoclinic, $C2/c$
a = 27.5064 (5) Å
b = 7.3811 (2) Å
c = 19.5038 (4) Å
β = 117.699 (2)°
V = 3506.02 (15) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 223 K
0.30 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
19187 measured reflections
2507 independent reflections
2836 reflections with $I > 3\sigma(I)$
R_{int} = 0.06

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.065
S = 1.04
2507 reflections
235 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···N1	0.87	1.74	2.523 (3)	148

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2181).

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supplementary materials

Acta Cryst. (2010). E**66**, o87-o88 [**doi:10.1107/S1600536809052258**]

2-[2-(4-Methoxyphenyl)-2,3-dihydro-1*H*-1,5-benzodiazepin-4-yl]phenol

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Comment

Heterocyclic scaffolds containing nitrogen atoms have received great attention in organic and medicinal chemistry because of their broad range of beneficial biological properties. These heterocyclic compounds such as benzodiazepines exhibit bioactive profile including anticonvulsant (MacDonald, 2002), hypnotic (Gringauz, 1999) and vasopressin antagonists (Albright *et al.*, 1998) activities. They are also used for treatment of gastrointestinal and central nervous system (*CNS*) disorder (Rahbaek *et al.*, 1999). As part of continuing work on heterocyclic compounds biologically active, we have synthesized new benzodiazepine derivative in order to explore the effects of substituents on activity and scaffold conformation of this compound class. In this paper, we present molecular structure of the title compound. The molecular structure of title compound is shown in Fig. 1. The benzodiazepine ring system adopts a distorted boat conformation as shown in the recent studies related to benzodiazepine derivatives (Ravichandran *et al.*, 2009*a*,*b*,*c*,*d*). The puckering parameters (Cremer & Pople, 1975) for this eleven-membered benzodiazepine ring system are: Q_2 = 1.087 (3) Å,Q_3 = 0.654 (3) Å,φ_2 = 320.74 (4)° and φ_3 = 26.7 (2)°. The benzene ring of this system forms dihedral angles of 89.69 (12)° and 48.82 (12)° with the phenyl rings of phenol and methoxy-phenyl fragments respectively which make them dihedral angle of 49.61 (11)°. Furthermore, there is in this structure the presence of O—H···N intra-molecular hydrogen bond, which generates an S (6) graph set motif (Bernstein *et al.*, 1995).

Experimental

To a solution of 1-(2-hydroxyphenyl)-3-(*p*-tolyl) propenone (1.3 g, 5.4 mmol) and 1, 2-diaminobenzene in anhydrous ethanol (20 ml), was added triethylamine (6 ml, 32.4 mmol). The reaction mixture was stirred under shelter from the light for 24 h. The resulting mixture was cooled at room temperature then kept in the freezer all night long. The precipitate was then filtered and purified by chromatography silica gel. Elution solvent: hexane/ethyl acetate (90/10). We obtained yellow single crystals of title compound with a yield of 56% (m.p.: 413–415 K; Rf: 1/2, hexane/ethyl acetate: 80/20).

Refinement

The H atoms were all located in a difference of Fourier map. They were all initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.95–0.97 Å, O—H = 0.87 Å, N—H = 0.88 Å and U_{iso}(H)in the range 1.2–1.7 times U_{eq} of the parent atom), after which their positions were refined with riding constraints.

Figures

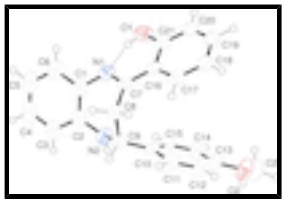


Fig. 1. The molecular structure of the title compound and the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

2-[2-(4-Methoxyphenyl)-2,3-dihydro-1*H*-1,5-benzodiazepin-4-yl]phenol

Crystal data

$C_{22}H_{20}N_2O_2$	$F(000) = 1456$
$M_r = 344.41$	$D_x = 1.305$ Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point = 413–415 K
Hall symbol: -C 2yc	Mo *K*α radiation, λ = 0.71073 Å
a = 27.5064 (5) Å	Cell parameters from 19187 reflections
b = 7.3811 (2) Å	θ = 0–0°
c = 19.5038 (4) Å	μ = 0.08 mm^{-1}
β = 117.699 (2)°	*T* = 223 K
V = 3506.02 (15) Å^3	Block, yellow
Z = 8	0.30 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.06$
graphite	$\theta_{max} = 29.1°$, $\theta_{min} = 1.7°$
φ and ω scans	*h* = −37→32
19187 measured reflections	*k* = −10→10
2507 independent reflections	*l* = −25→25
2836 reflections with $I > 3\sigma(I)$	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.055$	H-atom parameters constrained
$wR(F^2) = 0.065$	Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 76.3 80.0 28.8 -10.0 -11.5
S = 1.04	$(\Delta/\sigma)_{max} = 0.0004$

2507 reflections

235 parameters

0 restraints

$\Delta\rho_{max} = 0.25$ e Å^{-3}

$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.93812 (7)	0.1834 (2)	0.28604 (9)	0.0613
O2	0.74005 (6)	0.9744 (2)	0.19252 (11)	0.0715
N1	0.92187 (7)	0.2773 (2)	0.15267 (10)	0.0441
N2	0.82826 (7)	0.4219 (3)	0.02002 (10)	0.0518
C1	0.90766 (9)	0.2197 (3)	0.07668 (13)	0.0465
C2	0.86133 (9)	0.2877 (3)	0.01157 (13)	0.0481
C3	0.84587 (10)	0.2063 (4)	−0.06017 (14)	0.0600
C4	0.87638 (12)	0.0637 (4)	−0.06710 (17)	0.0694
C5	0.92265 (12)	0.0009 (4)	−0.00312 (18)	0.0690
C6	0.93752 (10)	0.0773 (3)	0.06788 (16)	0.0572
C7	0.92184 (7)	0.4477 (3)	0.16870 (12)	0.0385
C8	0.91134 (8)	0.5878 (3)	0.10725 (12)	0.0404
C9	0.85020 (8)	0.6032 (3)	0.04950 (12)	0.0435
C10	0.81802 (8)	0.6940 (3)	0.08532 (11)	0.0405
C11	0.82500 (9)	0.8789 (3)	0.10081 (14)	0.0547
C12	0.79940 (9)	0.9683 (3)	0.13693 (15)	0.0584
C13	0.76448 (8)	0.8738 (3)	0.15756 (14)	0.0526
C14	0.75569 (9)	0.6925 (3)	0.14123 (13)	0.0509
C15	0.78302 (8)	0.6032 (3)	0.10597 (12)	0.0475
C16	0.93285 (8)	0.4968 (3)	0.24729 (12)	0.0384
C17	0.93531 (8)	0.6780 (3)	0.27066 (12)	0.0442
C18	0.94419 (8)	0.7245 (3)	0.34386 (13)	0.0506
C19	0.95159 (9)	0.5901 (3)	0.39714 (13)	0.0532
C20	0.95014 (9)	0.4115 (3)	0.37696 (13)	0.0532
C21	0.94043 (8)	0.3622 (3)	0.30257 (13)	0.0455
C22	0.69528 (10)	0.8907 (4)	0.19955 (17)	0.0766
H82	0.9309	0.5495	0.0775	0.0489*
H81	0.9244	0.7079	0.1309	0.0491*
H191	0.9568	0.6222	0.4495	0.0668*
H91	0.8471	0.6809	0.0055	0.0540*
H111	0.8489	0.9442	0.0849	0.0673*
H141	0.7308	0.6258	0.1547	0.0624*
H151	0.7763	0.4730	0.0942	0.0604*
H201	0.9565	0.3167	0.4133	0.0656*
H51	0.9445	−0.0943	−0.0083	0.0953*
H171	0.9305	0.7735	0.2334	0.0558*
H181	0.9460	0.8507	0.3583	0.0644*
H21	0.7927	0.4133	−0.0098	0.0665*
H41	0.8660	0.0108	−0.1167	0.0911*
H121	0.8062	1.0951	0.1497	0.0723*
H31	0.8135	0.2496	−0.1043	0.0789*

H61	0.9693	0.0325	0.1134	0.0794*
H222	0.6805	0.9858	0.2210	0.1264*
H223	0.6683	0.8514	0.1473	0.1269*
H221	0.7094	0.7849	0.2350	0.1273*
H11	0.9322	0.1725	0.2385	0.0949*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0752 (11)	0.0399 (10)	0.0615 (10)	0.0001 (8)	0.0257 (9)	0.0135 (8)
O2	0.0518 (10)	0.0654 (12)	0.1058 (14)	−0.0043 (9)	0.0437 (10)	−0.0231 (10)
N1	0.0415 (10)	0.0383 (10)	0.0543 (11)	−0.0021 (8)	0.0238 (9)	0.0006 (8)
N2	0.0363 (9)	0.0547 (12)	0.0585 (12)	−0.0069 (9)	0.0171 (9)	−0.0094 (10)
C1	0.0465 (12)	0.0374 (12)	0.0636 (15)	−0.0115 (10)	0.0324 (12)	−0.0060 (11)
C2	0.0481 (13)	0.0476 (13)	0.0560 (14)	−0.0169 (11)	0.0305 (11)	−0.0092 (11)
C3	0.0607 (15)	0.0623 (16)	0.0641 (16)	−0.0240 (13)	0.0348 (13)	−0.0153 (13)
C4	0.0840 (19)	0.0658 (18)	0.0814 (19)	−0.0386 (16)	0.0579 (17)	−0.0348 (16)
C5	0.0725 (18)	0.0542 (16)	0.100 (2)	−0.0212 (14)	0.0570 (18)	−0.0257 (16)
C6	0.0564 (13)	0.0436 (13)	0.0811 (17)	−0.0112 (12)	0.0401 (13)	−0.0116 (13)
C7	0.0296 (10)	0.0354 (11)	0.0507 (12)	−0.0019 (8)	0.0187 (9)	0.0036 (9)
C8	0.0369 (10)	0.0366 (11)	0.0494 (12)	−0.0043 (9)	0.0216 (9)	0.0028 (10)
C9	0.0388 (11)	0.0449 (13)	0.0442 (12)	−0.0031 (10)	0.0171 (9)	0.0070 (10)
C10	0.0321 (10)	0.0391 (12)	0.0441 (12)	−0.0001 (9)	0.0123 (9)	0.0076 (9)
C11	0.0426 (12)	0.0419 (14)	0.0816 (17)	−0.0022 (10)	0.0307 (12)	0.0095 (12)
C12	0.0417 (12)	0.0384 (13)	0.0939 (19)	−0.0027 (10)	0.0305 (13)	−0.0044 (13)
C13	0.0370 (12)	0.0511 (14)	0.0664 (15)	0.0017 (11)	0.0211 (11)	−0.0061 (12)
C14	0.0436 (12)	0.0480 (14)	0.0668 (15)	−0.0064 (11)	0.0304 (11)	−0.0008 (12)
C15	0.0457 (12)	0.0391 (12)	0.0596 (14)	−0.0059 (10)	0.0262 (11)	0.0006 (11)
C16	0.0296 (10)	0.0379 (11)	0.0459 (12)	0.0015 (8)	0.0161 (9)	0.0061 (9)
C17	0.0392 (11)	0.0400 (13)	0.0526 (13)	0.0036 (9)	0.0206 (10)	0.0063 (10)
C18	0.0472 (13)	0.0501 (13)	0.0550 (14)	0.0061 (11)	0.0242 (11)	−0.0012 (11)
C19	0.0447 (12)	0.0661 (16)	0.0488 (13)	0.0071 (12)	0.0217 (11)	0.0046 (12)
C20	0.0476 (13)	0.0612 (15)	0.0501 (13)	0.0048 (12)	0.0221 (11)	0.0162 (12)
C21	0.0369 (11)	0.0418 (12)	0.0544 (13)	0.0019 (10)	0.0182 (10)	0.0107 (11)
C22	0.0595 (15)	0.087 (2)	0.097 (2)	−0.0058 (16)	0.0485 (16)	−0.0176 (18)

Geometric parameters (Å, °)

O1—C21	1.353 (2)	C9—H91	1.002
O1—H11	0.867	C10—C11	1.392 (3)
O2—C13	1.376 (3)	C10—C15	1.378 (3)
O2—C22	1.440 (3)	C11—C12	1.374 (3)
N1—C1	1.411 (3)	C11—H111	0.975
N1—C7	1.297 (3)	C12—C13	1.389 (3)
N2—C2	1.405 (3)	C12—H121	0.964
N2—C9	1.470 (3)	C13—C14	1.371 (3)
N2—H21	0.877	C14—C15	1.397 (3)
C1—C2	1.409 (3)	C14—H141	0.972
C1—C6	1.393 (3)	C15—H151	0.986

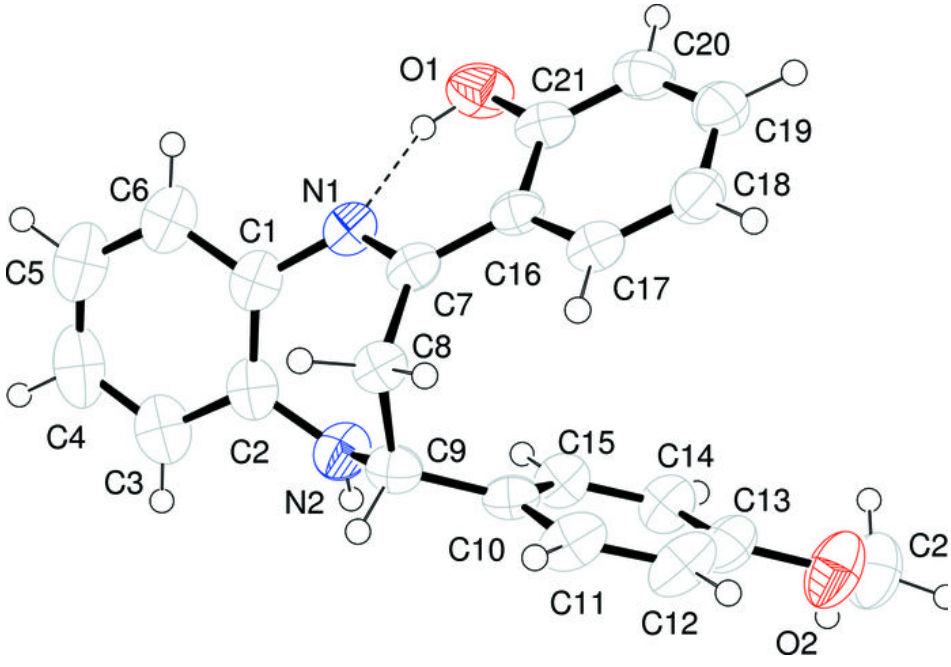
C2—C3	1.395 (3)	C16—C17	1.405 (3)
C3—C4	1.391 (3)	C16—C21	1.409 (3)
C3—H31	0.960	C17—C18	1.375 (3)
C4—C5	1.384 (4)	C17—H171	0.976
C4—H41	0.956	C18—C19	1.381 (3)
C5—C6	1.370 (3)	C18—H181	0.968
C5—H51	0.960	C19—C20	1.371 (3)
C6—H61	0.970	C19—H191	0.993
C7—C8	1.505 (3)	C20—C21	1.396 (3)
C7—C16	1.463 (3)	C20—H201	0.953
C8—C9	1.533 (3)	C22—H222	0.997
C8—H82	0.999	C22—H223	0.985
C8—H81	0.987	C22—H221	0.995
C9—C10	1.515 (3)		
C21—O1—H11	108.1	C11—C10—C15	117.3 (2)
C13—O2—C22	116.84 (19)	C10—C11—C12	122.0 (2)
C1—N1—C7	120.97 (18)	C10—C11—H111	117.5
C2—N2—C9	121.12 (16)	C12—C11—H111	120.5
C2—N2—H21	117.0	C11—C12—C13	119.7 (2)
C9—N2—H21	117.0	C11—C12—H121	120.9
N1—C1—C2	122.16 (19)	C13—C12—H121	119.3
N1—C1—C6	117.7 (2)	C12—C13—O2	115.8 (2)
C2—C1—C6	119.7 (2)	C12—C13—C14	119.6 (2)
C1—C2—N2	120.6 (2)	O2—C13—C14	124.6 (2)
C1—C2—C3	118.5 (2)	C13—C14—C15	119.8 (2)
N2—C2—C3	120.6 (2)	C13—C14—H141	120.1
C2—C3—C4	120.4 (3)	C15—C14—H141	120.1
C2—C3—H31	118.6	C14—C15—C10	121.6 (2)
C4—C3—H31	120.9	C14—C15—H151	119.3
C3—C4—C5	120.6 (2)	C10—C15—H151	119.2
C3—C4—H41	119.9	C7—C16—C17	122.07 (19)
C5—C4—H41	119.5	C7—C16—C21	120.80 (19)
C4—C5—C6	119.4 (3)	C17—C16—C21	117.1 (2)
C4—C5—H51	120.6	C16—C17—C18	122.2 (2)
C6—C5—H51	120.0	C16—C17—H171	118.5
C1—C6—C5	121.3 (3)	C18—C17—H171	119.4
C1—C6—H61	118.5	C17—C18—C19	119.7 (2)
C5—C6—H61	120.2	C17—C18—H181	120.2
N1—C7—C8	119.71 (19)	C19—C18—H181	120.2
N1—C7—C16	118.03 (19)	C18—C19—C20	120.0 (2)
C8—C7—C16	122.26 (18)	C18—C19—H191	120.2
C7—C8—C9	112.03 (16)	C20—C19—H191	119.7
C7—C8—H82	108.3	C19—C20—C21	121.0 (2)
C9—C8—H82	107.3	C19—C20—H201	121.4
C7—C8—H81	110.6	C21—C20—H201	117.5
C9—C8—H81	108.3	C16—C21—C20	120.0 (2)
H82—C8—H81	110.3	C16—C21—O1	122.1 (2)
C8—C9—N2	109.30 (17)	C20—C21—O1	117.9 (2)
C8—C9—C10	111.76 (17)	O2—C22—H222	105.8

N2—C9—C10	111.18 (16)	O2—C22—H223	107.2
C8—C9—H91	107.4	H222—C22—H223	112.5
N2—C9—H91	109.2	O2—C22—H221	109.1
C10—C9—H91	107.8	H222—C22—H221	111.4
C9—C10—C11	118.86 (19)	H223—C22—H221	110.6
C9—C10—C15	123.85 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H11···N1	0.87	1.74	2.523 (3)	148

Fig. 1


O1
C21
C20
C19
C18
C17
C16
C7
C8
N1
C1
C6
C5
C4
C3
C2
N2
C9
C15
C14
C13
C10
C11
C12
O2
C2