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1-Benzyl-1,4-diazepan-5-one

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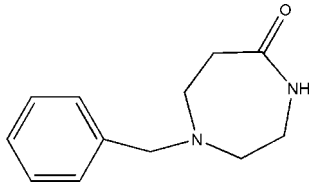
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.088; wR factor = 0.190; data-to-parameter ratio = 17.0.

The title compound, $C_{12}H_{16}N_2O$, is a diazepane intermediate that can be used as an inhibitor of human nitric oxide synthesis. In the molecule, the seven-membered ring has a chair-like conformation and the two rings are approximately perpendicular to one another, with a C—N—C—C torsion angle of 77.8 (4)°. Intermolecular N—H···O hydrogen bonds link the molecules into dimers around a centre of symmetry, with C—H···O interactions linking the dimers into infinite sheets.

Related literature

For related literature, see: Gopalakrishnan *et al.* (2007); Wlodarczyk *et al.* (2006).



Experimental

Crystal data

$C_{12}H_{16}N_2O$
M_r = 204.27
Monoclinic, $P2_1/c$
a = 12.602 (3) Å
b = 7.4920 (15) Å
c = 12.824 (3) Å
β = 111.00 (3)°
V = 1130.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.985, T_{max} = 0.992
2308 measured reflections
2205 independent reflections
1162 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.189
S = 0.99
2205 reflections
130 parameters
46 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O^{i}	0.86	2.00	2.821 (5)	160
C4—H4B···O^{ii}	0.97	2.51	3.377 (5)	149

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2179).

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supporting information

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1-Benzyl-1,4-diazepan-5-one

Fei Sha, Hao Xu, Shan Liu, Peng Wang and Jin-tang Wang

S1. Comment

The title compound is a 1-substituted 1,4-diazepan-5-one; an important class of heterocyclic compounds that have widespread applications from pharmaceuticals (Wlodarczyk *et al.*, 2006) to biology (Gopalakrishnan *et al.*, 2007). As part of our studies in this area, we report herein the synthesis and crystal structure of the title compound, (I).

In the molecule (Fig. 1) the 7-membered ring has a chair-like conformation with C1,C2,C3 and C4 forming the planar seat of the chair and N2 out of the plane on one side and N1—C5 out of the plane on the other side. The two rings are approximately perpendicular to one another with a C3—N2—C6—C7 torsion angle of 77.8 (4)°. Intermolecular N—H···O hydrogen bonds link the molecules into dimers around a center of symmetry with C—H···O interactions linking the dimers into infinite sheets (Fig. 2).

S2. Experimental

1-Benzyl-piperidin-4-one (18.9 g,0.1 mol) was added into a stirred mixture of sulfuric acid (40 ml) and dichloromethane (80 ml) at 273 K. Then, at 273 K, sodium azide (32.5 g,0.5 mol) was cautiously added over a period of 3 h and the resulting mixture was stirred for 1 h with the temperature kept at approximately 278 K. Then ice (1 kg) was quickly added and the solution was alkalized with ammonium hydroxide (15%,200 mL) to pH=11. The organic layer was separated with the water fraction extracted with dichloromethane (3x100mL). The organic extracts were combined,dried over $NaSO_4$, and concentrated *in vacuo*. The residue was recrystallized from EtOAc to give the title compound, (I) (yield: 13.0 g, 65%). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

S3. Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and 0.93 Å fro aromatic carbons and 0.97 Å for all others, and constrained to ride on their parent atoms, with U_{iso}(H) = xUeq(C,*N*), where *x* = 1.5 for methyl H, and *x* = 1.2 for all other H atoms.

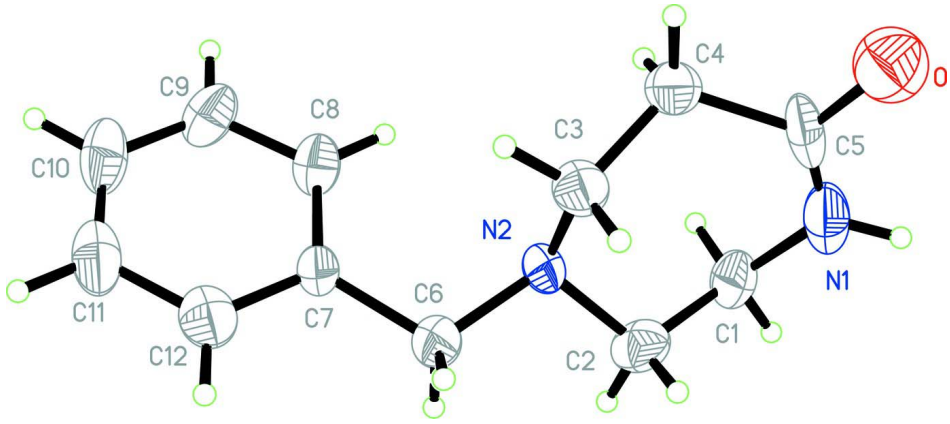

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

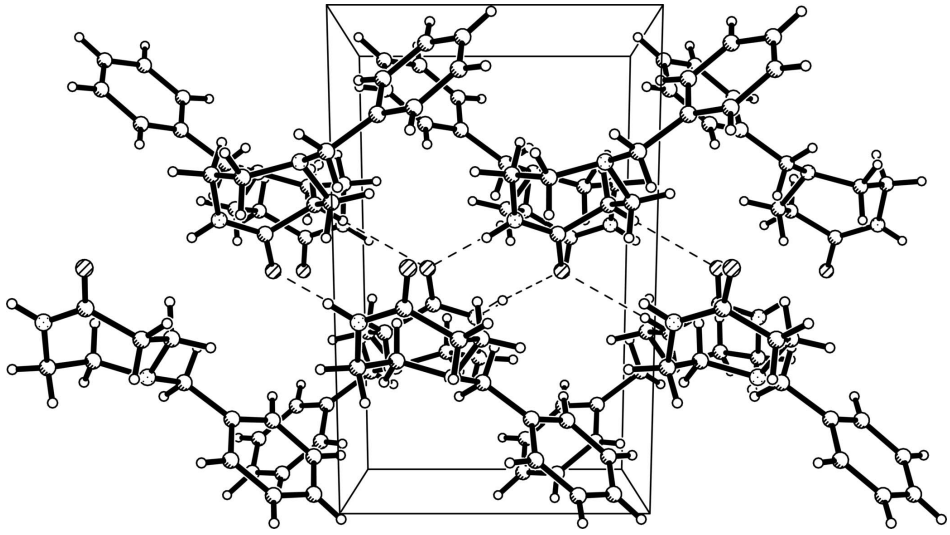

Figure 2

A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_{12}H_{16}N_2O$
$M_r = 204.27$
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Hall symbol: -P 2ybc
$a = 12.602\ (3)$ Å
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$c = 12.824\ (3)$ Å
$\beta = 111.00\ (3)°$
$V = 1130.3\ (4)$ Å^3
$Z = 4$

$F(000) = 440$
$D_x = 1.200$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–12°$
$\mu = 0.08$ mm^{-1}
$T = 298$ K
BLOCK, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.985, T_{max} = 0.992
2308 measured reflections
2205 independent reflections
1162 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 26.0°, θ_{min} = 1.7°
$h = -15\rightarrow14$
$k = 0\rightarrow9$
$l = 0\rightarrow15$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.189
S = 1.00
2205 reflections
130 parameters
46 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 1.65P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.3935 (3)	−0.4319 (6)	0.5667 (3)	0.0870 (12)
H1A	0.4312	−0.5275	0.5667	0.104*
O	0.5027 (3)	−0.2681 (5)	0.4894 (3)	0.108
N2	0.2757 (3)	−0.1384 (4)	0.6724 (2)	0.0542 (8)
C1	0.2993 (4)	−0.4434 (5)	0.6094 (4)	0.0705 (12)
H1B	0.2940	−0.5657	0.6319	0.085*
H1C	0.2289	−0.4158	0.5487	0.085*
C2	0.3090 (4)	−0.3232 (5)	0.7059 (4)	0.0697 (11)
H2A	0.2613	−0.3696	0.7446	0.084*
H2B	0.3869	−0.3245	0.7579	0.084*
C3	0.3581 (3)	−0.0442 (5)	0.6349 (3)	0.0632 (10)
H3A	0.4334	−0.0571	0.6912	0.076*
H3B	0.3396	0.0820	0.6278	0.076*
C4	0.3596 (3)	−0.1132 (6)	0.5236 (3)	0.0683 (11)
H4A	0.2817	−0.1299	0.4735	0.082*

H4B	0.3932	−0.0222	0.4913	0.082*
C5	0.4230 (3)	−0.2848 (7)	0.5289 (4)	0.0765 (13)
C6	0.2595 (3)	−0.0404 (6)	0.7642 (3)	0.0685 (12)
H6A	0.3332	−0.0018	0.8157	0.082*
H6B	0.2272	−0.1204	0.8043	0.082*
C7	0.1827 (3)	0.1221 (5)	0.7265 (3)	0.0531 (9)
C8	0.1029 (3)	0.1333 (5)	0.6142 (4)	0.0643 (11)
H8A	0.1017	0.0489	0.5606	0.077*
C9	0.0285 (4)	0.2739 (6)	0.5892 (4)	0.0779 (13)
H9A	−0.0223	0.2845	0.5161	0.094*
C10	0.0241 (4)	0.3960 (7)	0.6626 (5)	0.0865 (15)
H10A	−0.0288	0.4879	0.6406	0.104*
C11	0.0969 (5)	0.3858 (6)	0.7691 (5)	0.0869 (15)
H11A	0.0954	0.4710	0.8213	0.104*
C12	0.1748 (4)	0.2447 (6)	0.7999 (4)	0.0713 (12)
H12A	0.2230	0.2354	0.8741	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.065 (2)	0.109 (3)	0.076 (3)	0.031 (2)	0.0121 (19)	−0.027 (2)
O	0.108	0.108	0.108	0.000	0.039	0.000
N2	0.0666 (19)	0.0520 (18)	0.0461 (17)	0.0091 (16)	0.0229 (15)	−0.0087 (15)
C1	0.084 (3)	0.048 (2)	0.083 (3)	0.011 (2)	0.034 (2)	−0.003 (2)
C2	0.085 (3)	0.050 (2)	0.073 (3)	−0.004 (2)	0.027 (2)	0.011 (2)
C3	0.072 (3)	0.055 (2)	0.070 (3)	−0.005 (2)	0.035 (2)	0.002 (2)
C4	0.054 (2)	0.089 (3)	0.065 (2)	0.012 (2)	0.0255 (19)	0.016 (2)
C5	0.051 (2)	0.100 (3)	0.084 (3)	0.017 (2)	0.030 (2)	−0.033 (3)
C6	0.069 (3)	0.079 (3)	0.055 (2)	0.025 (2)	0.020 (2)	0.003 (2)
C7	0.060 (2)	0.048 (2)	0.058 (2)	0.0113 (18)	0.0293 (19)	0.0002 (18)
C8	0.052 (2)	0.058 (2)	0.080 (3)	0.003 (2)	0.020 (2)	−0.009 (2)
C9	0.064 (3)	0.064 (3)	0.092 (3)	0.012 (2)	0.011 (2)	0.007 (3)
C10	0.066 (3)	0.074 (3)	0.131 (5)	0.018 (3)	0.050 (3)	−0.009 (3)
C11	0.098 (4)	0.061 (3)	0.119 (4)	0.001 (3)	0.061 (4)	−0.018 (3)
C12	0.078 (3)	0.064 (3)	0.084 (3)	−0.011 (2)	0.043 (3)	−0.003 (2)

Geometric parameters (Å, º)

N1—C5	1.311 (6)	C4—H4A	0.9700
N1—C1	1.477 (5)	C4—H4B	0.9700
N1—H1A	0.8600	C6—C7	1.523 (5)
O—C5	1.284 (5)	C6—H6A	0.9700
N2—C6	1.462 (4)	C6—H6B	0.9700
N2—C2	1.465 (5)	C7—C12	1.345 (5)
N2—C3	1.471 (4)	C7—C8	1.433 (5)
C1—C2	1.500 (5)	C8—C9	1.370 (5)
C1—H1B	0.9700	C8—H8A	0.9300
C1—H1C	0.9700	C9—C10	1.328 (6)

C2—H2A	0.9700	C9—H9A	0.9300
C2—H2B	0.9700	C10—C11	1.347 (7)
C3—C4	1.525 (5)	C10—H10A	0.9300
C3—H3A	0.9700	C11—C12	1.400 (6)
C3—H3B	0.9700	C11—H11A	0.9300
C4—C5	1.502 (6)	C12—H12A	0.9300

C5—N1—C1	124.0 (4)	H4A—C4—H4B	107.4
C5—N1—H1A	118.0	O—C5—N1	126.5 (4)
C1—N1—H1A	118.0	O—C5—C4	112.2 (5)
C6—N2—C2	110.3 (3)	N1—C5—C4	121.3 (3)
C6—N2—C3	109.9 (3)	N2—C6—C7	113.7 (3)
C2—N2—C3	112.8 (3)	N2—C6—H6A	108.8
N1—C1—C2	115.6 (4)	C7—C6—H6A	108.8
N1—C1—H1B	108.4	N2—C6—H6B	108.8
C2—C1—H1B	108.4	C7—C6—H6B	108.8
N1—C1—H1C	108.4	H6A—C6—H6B	107.7
C2—C1—H1C	108.4	C12—C7—C8	117.6 (4)
H1B—C1—H1C	107.4	C12—C7—C6	121.5 (4)
N2—C2—C1	113.3 (3)	C8—C7—C6	120.2 (3)
N2—C2—H2A	108.9	C9—C8—C7	117.1 (4)
C1—C2—H2A	108.9	C9—C8—H8A	121.5
N2—C2—H2B	108.9	C7—C8—H8A	121.5
C1—C2—H2B	108.9	C10—C9—C8	124.2 (5)
H2A—C2—H2B	107.7	C10—C9—H9A	117.9
N2—C3—C4	113.0 (3)	C8—C9—H9A	117.9
N2—C3—H3A	109.0	C9—C10—C11	119.6 (5)
C4—C3—H3A	109.0	C9—C10—H10A	120.2
N2—C3—H3B	109.0	C11—C10—H10A	120.2
C4—C3—H3B	109.0	C10—C11—C12	118.9 (5)
H3A—C3—H3B	107.8	C10—C11—H11A	120.6
C5—C4—C3	115.7 (3)	C12—C11—H11A	120.6
C5—C4—H4A	108.4	C7—C12—C11	122.6 (4)
C3—C4—H4A	108.4	C7—C12—H12A	118.7
C5—C4—H4B	108.4	C11—C12—H12A	118.7
C3—C4—H4B	108.4		

C5—N1—C1—C2	−58.5 (6)	C3—N2—C6—C7	77.8 (4)
C6—N2—C2—C1	165.8 (3)	N2—C6—C7—C12	−167.9 (4)
C3—N2—C2—C1	−70.9 (4)	N2—C6—C7—C8	22.3 (5)
N1—C1—C2—N2	79.5 (5)	C12—C7—C8—C9	3.2 (6)
C6—N2—C3—C4	−166.9 (3)	C6—C7—C8—C9	173.4 (4)
C2—N2—C3—C4	69.6 (4)	C7—C8—C9—C10	−1.8 (7)
N2—C3—C4—C5	−78.3 (4)	C8—C9—C10—C11	0.5 (8)
C1—N1—C5—O	−178.8 (4)	C9—C10—C11—C12	−0.7 (7)
C1—N1—C5—C4	−1.4 (7)	C8—C7—C12—C11	−3.6 (6)
C3—C4—C5—O	−122.4 (4)	C6—C7—C12—C11	−173.7 (4)
C3—C4—C5—N1	59.8 (6)	C10—C11—C12—C7	2.4 (7)

C2—N2—C6—C7	−157.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O^{i}	0.86	2.00	2.821 (5)	160
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