

4,4-Dimethyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-oneNaghmana Rashid,^{a*} Mashooda Hasan,^a Nurdiyana M. Yusof^b and Bohari M. Yamin^b^aDepartment of Chemistry, Allama Iqbal Open University, H-8, Islamabad, Pakistan, and^bSchool of Chemical Sciences and Food Technology, Univeriti Kebangsaan Malaysia, UKM 43500 Bangi Selangor, Malaysia

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Key indicators

Single-crystal X-ray study

 $T = 298\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$ R factor = 0.038 wR factor = 0.100

Data-to-parameter ratio = 14.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

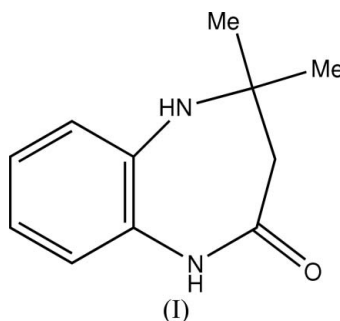
In the title compound, $\text{C}_{11}\text{H}_{14}\text{N}_2\text{O}$, the diazepine ring adopts a skewed boat conformation. The molecule is stabilized by $\text{N}-\text{H}\cdots\text{O}$ intermolecular hydrogen bonds, forming a zigzag chain parallel to the b axis.

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Comment

Benzodiazepines are among the most important scaffolds in medicinal chemistry, representing the prototypical 'privileged structure' (Evans *et al.* 1988). Compounds based on the diazepine skeleton are psychotherapeutic agents widely used for the treatment of anxiety and neurosis (Sternbach, 1978). The derivatives of 1,5-benzodiazepin-2-one belong to a pharmacologically important subclass of benzodiazepines represented by the commercially available drug Lofendazam [7-chloro-5-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one]. The idea that the biological activity of this type of compounds depends not only on the pharmacophoric grouping but also on the conformation of the seven-membered ring (Aversa *et al.*, 1981) prompted us to investigate its crystal structure.



The diazepine ring (Fig. 1) adopts a somewhat skewed boat conformation compared to the flat boat in 6,7-dimethyl-5-phenyl-1*H*-thieno[2,3-*e*][1,4]diazepin-2(3*H*)-one (Scammells *et al.*, 2001). The bond lengths and angles in (I) are in normal ranges (Allen *et al.*, 1987).

In the crystal structure, the molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ (Table 1) forming a zigzag chain of dimers (Fig. 2) parallel to the b axis.

Experimental

Equimolar quantities of 1,2-phenylenediamine (1.08 g, 0.01 mol) and 3-methylbut-2-enoic acid (1.00 g, 0.01 mol) were refluxed in 4 *N* HCl (30 ml) for 8 h to synthesize the title compound, (I), following a previously reported procedure (Hasan *et al.*, 1990). After neutral-

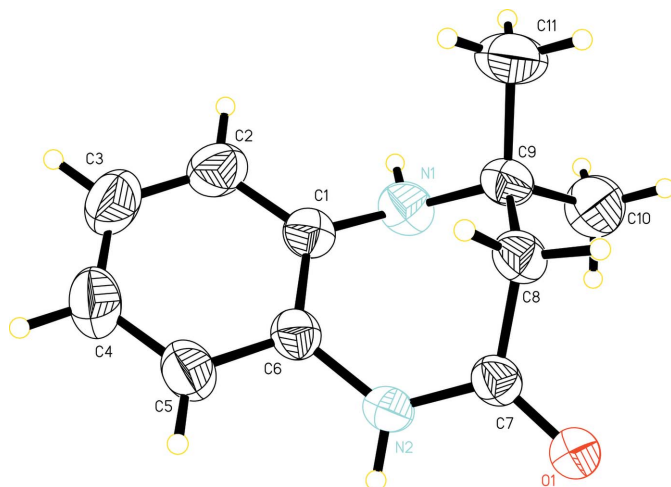


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

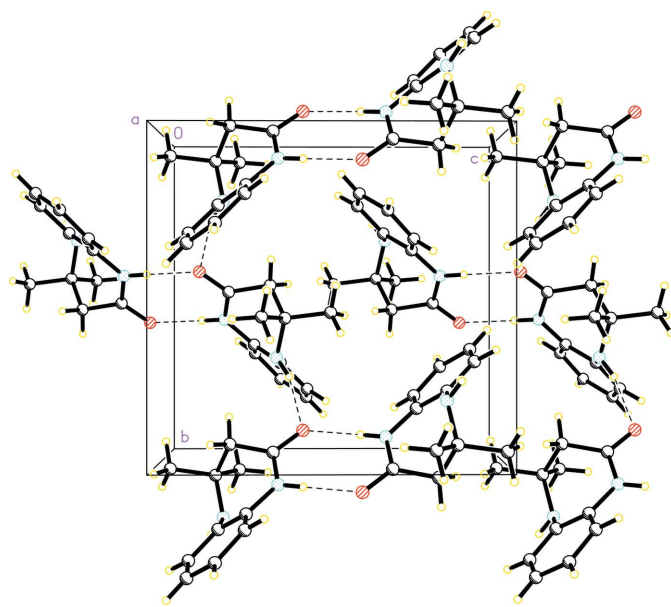


Figure 2
The packing of (I), viewed down the *a* axis. The intermolecular hydrogen bonds are denoted by dashed lines.

ization with sodium carbonate, the solid obtained was washed with water and purified by column chromatography using silica gel in petroleum ether–acetone (1:4) ($R_F = 0.15$) and recrystallized from ethanol (yield: 10%, 190 mg; m.p. 526 K).

Crystal data

$C_{11}H_{14}N_2O$ $Z = 4$
 $M_r = 190.24$ $D_x = 1.213 \text{ Mg m}^{-3}$
 Monoclinic, $P2_1/n$ Mo $K\alpha$ radiation
 $a = 9.355 (3) \text{ \AA}$ $\mu = 0.08 \text{ mm}^{-1}$
 $b = 10.335 (3) \text{ \AA}$ $T = 298 (2) \text{ K}$
 $c = 10.781 (3) \text{ \AA}$ Block, colourless
 $\beta = 91.106 (6)^\circ$ $0.50 \times 0.48 \times 0.22 \text{ mm}$
 $V = 1042.1 (5) \text{ \AA}^3$

Data collection

Bruker SMART APEX CCD area-detector diffractometer 5658 measured reflections
 2048 independent reflections
 ω scans 1656 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan (*SADABS*; Bruker, 2000) $R_{int} = 0.022$
 $T_{min} = 0.961, T_{max} = 0.982$ $\theta_{max} = 26.0^\circ$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0418P)^2 + 0.1994P]$
 $R[F^2 > 2\sigma(F^2)] = 0.038$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.100$ $(\Delta/\sigma)_{max} < 0.001$
 $S = 1.04$ $\Delta\rho_{max} = 0.16 \text{ e \AA}^{-3}$
 2048 reflections $\Delta\rho_{min} = -0.12 \text{ e \AA}^{-3}$
 138 parameters Extinction correction: *SHELXL97*
 H atoms treated by a mixture of independent and constrained refinement Extinction coefficient: 0.050 (4)

Table 1
Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1A···O1 ⁱ	0.863 (14)	2.175 (14)	3.0105 (18)	162.9 (13)
N2–H2A···O1 ⁱⁱ	0.869 (11)	2.014 (11)	2.8824 (18)	179.4 (13)

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

H atoms on C were positioned geometrically ($C-H = 0.93-0.97 \text{ \AA}$) and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$ where $x = 1.5$ for methyl and $x = 1.2$ for other H atoms. H atoms attached to N were refined isotropically.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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