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# (5*S*,6*R*)-3,4,5,6-Tetrahydro-5-methyl-6-phenyl-4-propyl-2*H*-1,3,4-oxadiazin-2-one

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**Key indicators**

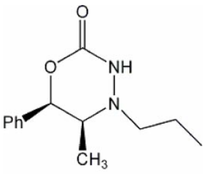
Single-crystal X-ray study
*T* = 193 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.031
*wR* factor = 0.088
Data-to-parameter ratio = 9.0

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

The title compound, $C_{13}H_{18}O_2N_2$, was isolated during the preparation of new chiral auxiliaries. Notably, this compound is unlike previously reported structures in that it does not contain an imide moiety. The crystal structure indicates the presence of intermolecular N—H···O hydrogen bonds.

## Comment

3,4,5,6-Tetrahydro-2*H*-1,3,4-oxadiazin-2-ones (Trepanier *et al.*, 1968; Hitchcock *et al.*, 2001) have been known for more than thirty years but have only recently been examined in terms of their synthesis (Hitchcock *et al.*, 2001), conformational analysis (Casper, Blackburn *et al.*, 2002), and asymmetric application (Casper & Hitchcock, 2003; Casper, Burgeson *et al.*, 2002). Specifically, we have been engaged in the development of novel oxadiazinones for application as chiral auxiliaries in the aldol addition reaction. We recently synthesized an N4-propyloxadiazinone (Hitchcock *et al.*, 2002) and have become interested in learning about the conformation adopted by this heterocycle. We report here the single-crystal X-ray structure analysis of the N4-propyloxadiazinone (I) (Fig. 1).



(I)

The structure of (I) is unique in that it is the first reported structure of an oxadiazinone that is not substituted at the N3 position. The oxadiazinone structures previously reported by Ferrence, Hitchcock and co-workers all contain a carbonyl group at the N3 position, which is believed to have an impact on the ring conformation (Burgeson *et al.*, 2004; Casper, Blackburn *et al.*, 2002; Casper *et al.*, 2002; Ferrence *et al.*, 2003; Hitchcock *et al.*, 2001, 2004). Although the title compound is not substituted at the N3 position, it still exists in a twist-boat conformation. The C14—N4—C5—C13 torsion angle is 163.16 (15)° and the C34—N24—C25—C33 torsion angle is 161.79 (15)°. The previously reported oxadiazinone structures having the twist-boat conformation have torsion angles in the range 169.07–176.66° (Hitchcock *et al.*, 2001; Burgeson *et al.*, 2004).

It should be noted that there are significant differences in the C2—N3 and the C22—N23 bond distances when the structure of (I) is compared with the previously reported oxadiazinone structures. The C2—N3 and C22—N23 distances

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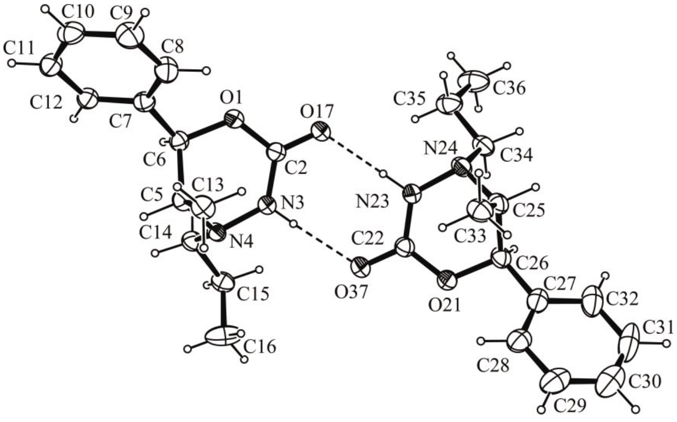

**Figure 1**
A view of the asymmetric unit of (I), showing the atom-numbering scheme and hydrogen bonding (dashed lines). Displacement ellipsoids are drawn at the 35% probability level and H atoms are drawn arbitrarily small for clarity.

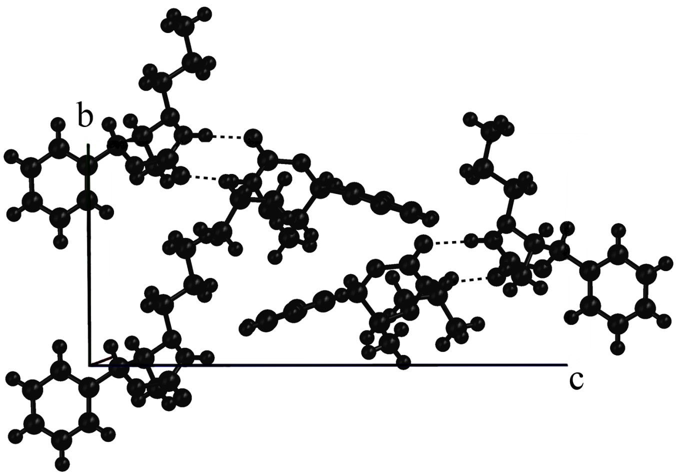

**Figure 2**
A packing diagram of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

in (I) are 1.337 (2) and 1.342 (2) Å, respectively. Distances previously reported in the literature for substituted oxadiazinone structures are in the range 1.389–1.418 Å (Casper *et al.*, 2002; Ferrence *et al.*, 2003). The shortening of this bond in (I) indicates greater double-bond character. Interestingly, the N—N bond length averages 1.425 Å, which is within the range of N—N bond lengths observed for the N3 carbonyl-substituted oxadiazinone structures (Casper, Blackburn *et al.*, 2002; Hitchcock *et al.*, 2001; Ferrence *et al.*, 2003). Table 1 contains selected bond lengths and angles for (I).

Hydrogen-bonding interactions clearly play a role in the crystal packing of the title compound. The key intermolecular interaction occurs between the NH group of the oxadiazinone ring and the carbonyl group on a neighboring molecule. Similar N—H, H···O and N···O distances and N—H···O angles are reported in the literature for analogous systems (Eckert *et al.*, 2001; Aakeröy *et al.*, 1996; Wash *et al.*, 1997).

## Experimental

The title compound was prepared as previously reported (Hitchcock, Casper *et al.*, 2002). Single crystals suitable for X-ray analysis were obtained from methylene chloride and hexanes using the solvent diffusion method.

### *Crystal data*

$C_{13}H_{18}N_2O_2$
$M_r = 234.29$
Monoclinic, $P2_1$
$a = 7.7432$ (4) Å
$b = 8.8457$ (4) Å
$c = 19.2021$ (9) Å
$\beta = 91.690$ (1)°
$V = 1314.66$ (11) Å$^3$
$Z = 4$

$D_x = 1.184$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 7993 reflections
$\theta = 2.5$–26.4°
$\mu = 0.08$ mm$^{-1}$
$T = 193$ (2) K
Prism, colorless
0.71 × 0.40 × 0.39 mm

### *Data collection*

Bruker PLATFORM /SMART 1000 CCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.945$, $T_{\max} = 0.969$
9368 measured reflections

2857 independent reflections
2780 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.014$
$\theta_{\max} = 26.4$°
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 10$
$l = -23 \rightarrow 23$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.088$
$S = 1.09$
2857 reflections
316 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 0.105P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.14$ e Å$^{-3}$
Extinction correction: *SHELXL*
Extinction coefficient: 0.043 (4)

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—C2 | 1.351 (2) | O21—C22 | 1.347 (2) |
| C2—O17 | 1.223 (2) | C22—O37 | 1.224 (2) |
| C2—N3 | 1.337 (2) | C22—N23 | 1.342 (2) |
| N3—N4 | 1.4215 (19) | N23—N24 | 1.428 (2) |
| | | | |
| O17—C2—N3 | 123.58 (15) | O37—C22—N23 | 122.60 (16) |
| O17—C2—O1 | 117.46 (15) | O37—C22—O21 | 118.03 (16) |
| N3—C2—O1 | 118.94 (15) | N23—C22—O21 | 119.35 (15) |
| C2—N3—N4 | 126.09 (14) | C22—N23—N24 | 125.80 (15) |
| | | | |
| C2—N3—N4—C5 | −25.2 (2) | C22—N23—N24—C25 | −21.3 (2) |
| C14—N4—C5—C13 | 163.16 (15) | C34—N24—C25—C33 | 161.79 (15) |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3···O37 | 0.82 (2) | 2.05 (2) | 2.870 (2) | 177 (2) |
| N23—H23···O17 | 0.88 (2) | 1.90 (2) | 2.778 (2) | 178 (2) |

The H atoms were initially located in a difference Fourier map; all the H atoms (except for the carbamate H atoms, N—H) were treated as riding (C—H = 0.95–1.00 Å), with isotropic displacement parameters fixed at $1.2U_{\rm eq}$ of the parent atom. A meaningful Flack parameter could not be determined due to the fact that Mo radiation was used in conjunction with a light-atom sample (Flack & Bernardinelli, 2000); accordingly, Friedel pairs were merged. The absolute configuration of the title compound was determined exclusively from the commercially available chemical precursor, (1*R*,2*S*)-norephedrine.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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