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1-Acetyl-*r*-2,*c*-6-di-2-furyl-3,5-dimethylpiperidin-4-one

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Key indicators

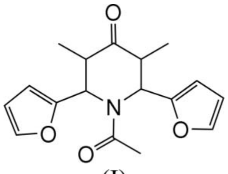
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.164
Data-to-parameter ratio = 23.1

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

In the title molecule, $C_{17}H_{19}NO_4$, the piperidine ring adopts a twist-boat conformation. The acetyl group at position 1 has a bisectional orientation. The planar furyl rings at positions 2 and 6 have axial and bisectional orientations, respectively. The methyl groups at positions 3 and 5 have bisectional and equatorial orientations, respectively. In the crystal structure, the molecules are stabilized by inter- and intramolecular C—H···O hydrogen bonds.

Comment

It has been reported that the calculated torsion angles in *N*-acetyl derivatives are significantly higher than those in the normal chair conformations and thus confirm the non-chair (twist-boat) conformation of the heterocyclic ring with a diaxial orientation of the phenyl and alkyl groups in *N*-acetyl derivatives (Bhavani *et al.*, 2000). Non-chair conformations have been proposed for *N*-benzoylpiperidones since the $A^{1,3}$ strain between the carbonyl group of the benzoyl group and the α-phenyl groups will be a minimum for this conformation, which is confirmed by *X*-ray diffraction studies of *N*-benzoyl-3-ethyl-2,6-diphenylpiperidin-4-one (Krishna Pillay *et al.*, 2000). Furthermore, compounds preferring a boat conformation in the crystalline state or in solution are of special interest since six-membered-ring molecules with an inherent preference for non-chair forms are rather rare (Gdaniec *et al.*, 1995).



(I)

The title compound, (I), has been analysed as part of our crystallographic studies on substituted piperidines (Balamurugan, *et al.*, 2006). The present *X*-ray diffraction study was undertaken to determine how the conformation of the system is affected by the substitution of an acetyl group at N, furyl rings at positions 2 and 6 and methyl groups at positions 3 and 5 of the piperidin-4-one. An *ORTEP-3* (Farrugia, 1997) drawing of the molecular structure of (I), with atomic numbering scheme, is shown in Fig. 1.

The sum of the angles C2—N1—C11 [118.8 (1)°], C2—N1—C6 [116.8 (1)°] and C6—N1—C11 [124.2 (1)°] indicates a coplanar orientation of the acetyl group at N1. The torsion angles C6—N1—C2—C21, N1—C2—C3—C31, H2—C2—C3—H3, N1—C6—C5—C51, H5—C5—C6—H6 and C2—N1—C6—C61 show that the furyl ring at position 2, the

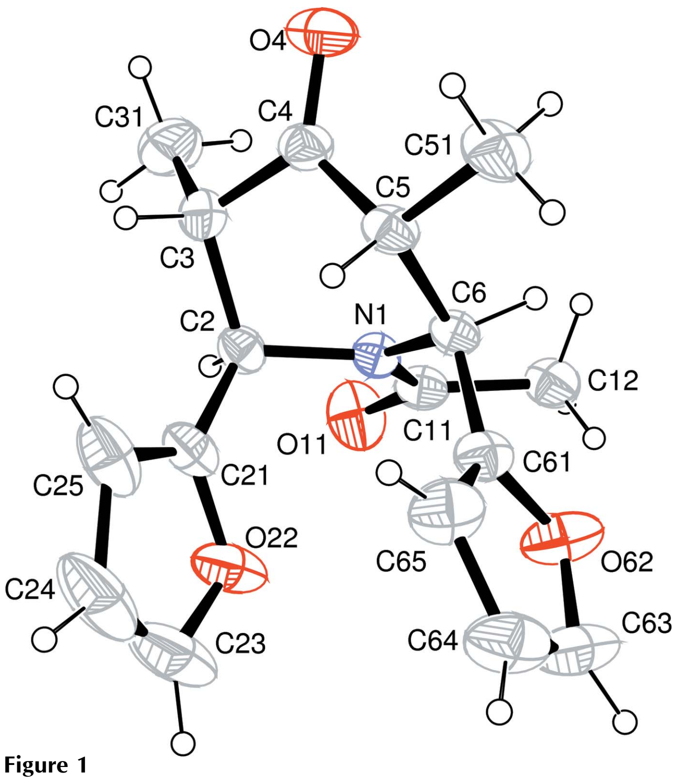


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

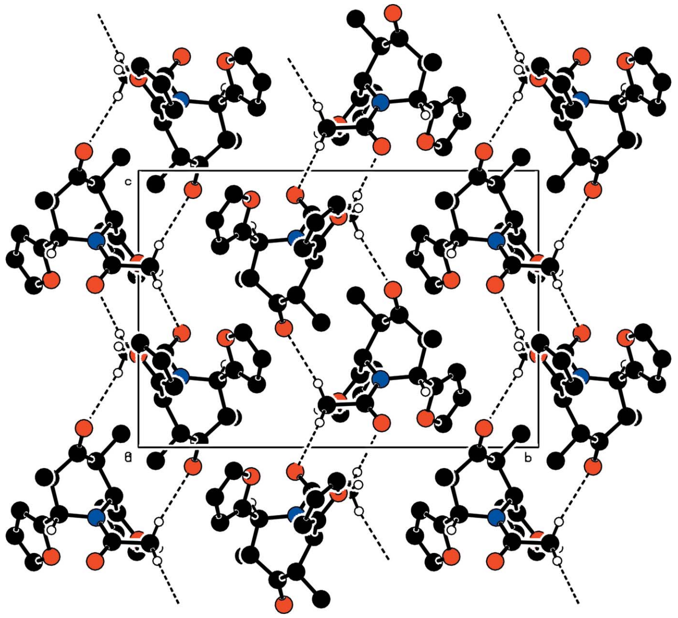


Figure 2
The molecular packing of (I), viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

methyl group at position 3, the methyl group at position 5 and the furyl ring at position 6 are in axial, bisectional, equatorial and bisectional orientations, respectively.

The displacements of N1, C2, C3, C4, C5 and C6 from the least-squares plane defined by atoms C2/C3/C5/C6 are −0.540 (2), 0.182 (1), −0.178 (1), −0.520 (2), 0.177 (1) and −0.181 (1) Å, respectively. This indicates that the piperidine ring adopts a twist-boat conformation (see Table 1 for torsion angles). The mean plane of atoms C2/C3/C5/C6 and the furyl rings attached to C2 and C6 make dihedral angles of 89.3 (1) and 75.5 (1)°, respectively. The dihedral angle between the two furyl rings is 19.8 (1)°. In the solid state, molecules are stabilized by intra- and intermolecular C—H···O interactions (see Table 2).

Experimental

A mixture of 3,5-dimethyl-*r*-2,*c*-6-di-2-furylpiperidin-4-one (3.01 g, 0.01 ml), acetic anhydride (3 ml, 0.03 mol) and triethylamine (3 ml, 0.03 mol) was refluxed for 6–8 h. The reaction mixture was cooled to room temperature and poured into ice-cold water. The solid mass which separated was filtered off, dried and recrystallized from petroleum ether (333–353 K). The yield of the isolated product was 1.5 g (50%).

Crystal data

$C_{17}H_{19}NO_4$
$M_r = 301.33$
Monoclinic, $P2_1/c$
$a = 9.1342$ (7) Å
$b = 15.7260$ (10) Å
$c = 11.1749$ (8) Å
$\beta = 103.055$ (3)°
$V = 1563.72$ (19) Å^3
$Z = 4$
$D_x = 1.280$ Mg m^{-3}
Mo *Kα* radiation
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.20 × 0.15 × 0.15 mm

Data collection

Bruker SMART diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.982$, $T_{max} = 0.986$
20527 measured reflections
4676 independent reflections
3038 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 30.4°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.164$
$S = 1.04$
4676 reflections
202 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.2676P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1
Selected torsion angles (°).

N1—C2—C3—C4	−28.97 (16)	C2—N1—C6—C61	89.34 (14)
C2—C3—C4—C5	−30.22 (18)	C6—N1—C2—C21	−63.12 (15)
C3—C4—C5—C6	60.09 (17)	N1—C2—C3—C31	95.83 (15)
C4—C5—C6—N1	−25.83 (16)	H2—C2—C3—H3	99
C5—C6—N1—C2	−34.76 (15)	C51—C5—C6—N1	−152.39 (13)
C6—N1—C2—C3	65.28 (15)	H5—C5—C6—H6	−152

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O11	0.98	2.22	2.704 (2)	109
C12—H12*A*···O11^{i}	0.96	2.48	3.432 (2)	170
C12—H12*B*···O62	0.96	2.44	3.206 (2)	137
C12—H12*C*···O4^{ii}	0.96	2.58	3.466 (2)	153

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

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H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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References

Balamurugan, S., Thiruvalluvar, A., Manimekalai, A., Selvaraju, K. & Maruthavanan, T. (2006). *Acta Cryst.* E**62**, o2005–o2006.

Bhavani, N., Natarajan, D. & Manimekalai, A. (2000). *Indian J. Chem. Sect. B*, **39**, 16–20.

Bruker (2004). *SMART* (Version 5.054), *SAINT* (Version 7.23A) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Gdaniec, M., Milewska, J. M. & Polonski, T. (1995). *J. Org. Chem.* **60**, 7411–7418.

Krishna Pillay, M., Krishna Kumar, R., Nagarajan, A. & Jeyaraman, G. (2000). *Indian J. Chem. Sect. B*, **39**, 419–425.

Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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