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(2*E*)-2-(1-Methylpiperidin-2-ylidene)-1-phenylethanone

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In the title compound, $C_{14}H_{17}NO$, the piperidine ring is in a half-chair conformation. The molecules are linked into *C*(7) chains by an intermolecular C—H···O hydrogen bond.

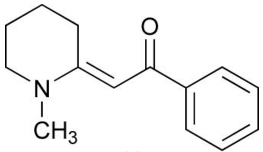
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.051
wR factor = 0.199
Data-to-parameter ratio = 13.3

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

Comment

Enaminones (β-acylated enamines) feature prominently in our research programme as intermediates for the synthesis of alkaloids and other nitrogen-containing heterocycles (Michael *et al.*, 1999). We required the title compound, (I), a simple enaminone, as a model for probing the reactivity of exocyclic enaminones towards reducing agents. Compound (I) has previously been prepared as a key intermediate in the synthesis of piperidine alkaloids isolated from the genus *Sedum* (Ghiaci & Adibi, 1996).



(I)

A view of the molecular structure of (I) is given in Fig. 1. The piperidine ring adopts a half-chair conformation [puckering amplitude Q_T = 0.551 (4) Å, θ = 124.0(3° and φ = 22.8 (4)° (Cremer & Pople, 1975)]. The bond lengths for the enaminone functionality from N1 to O1 are comparable with values reported in the literature (Allen *et al.*, 1987), but the delocalization does not extend to the phenyl ring [O1—C9—C10—C11 = −15.8 (3)°].

The crystal structure of (I) is built up by an intramolecular C—H···O hydrogen bond and weak intermolecular C—H···O hydrogen bonds that link the molecules into chains. Atom C7 in the molecule at (x, y, z) acts as a hydrogen-bond donor *via* atom H7*A* to atom O1 in the molecule at (x, $\frac{1}{2} - y$, $z - \frac{1}{2}$), thereby generating by translation a *C*(7) chain (Etter *et al.*, 1990; Bernstein *et al.*, 1995) running parallel to the [001] direction (Fig. 2 and Table 1).

Experimental

(2*E*)-2-(1-Methylpiperidin-2-ylidene)-1-phenylethanone, (I), was prepared in 77% yield from 1-methylpiperidine-2-thione and phenacyl bromide by the method of Ghiaci & Adibi (1996) (m.p. 340–343 K; literature m.p. 341–343 K). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.83–7.86 (2H, *m*, 11-H and 15-H), 7.35–7.39 (3H, *m*, 12-H, 13-H, 14-H), 5.65 (1H, *s*, 8-H), 3.31–3.45 (4H, *m*, 3-H and 6-H), 2.98 (3H, *s*, NCH_3), 1.78–1.86 and 1.65–1.71 (4H, 2 × *m*, 4-H and 5-H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 187.5 (C═O), 164.8 (C2), 143.1 (C10), 129.9

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(C13), 127.9 and 127.1 (C11, C12, C14, C15), 90.7 (C8), 52.0 (C6), 40.2 (NCH_3), 28.3 (C3), 23.1 and 19.4 (C4, C5). Crystals suitable for X-ray crystallography were obtained as pale-brown blocks by slow growth from a solution in EtOAc/hexane (approximately 1:1).

Crystal data

$C_{14}H_{17}NO$
$M_r = 215.29$
Monoclinic, $P2_1/c$
$a = 8.006$ (2) Å
$b = 9.441$ (3) Å
$c = 15.535$ (4) Å
$\beta = 95.197$ (6)°
$V = 1169.5$ (6) Å^3
$Z = 4$
$D_x = 1.223$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 173$ (2) K
Block, pale brown
0.38 × 0.28 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.962$, $T_{\max} = 0.982$
5068 measured reflections
2178 independent reflections
1563 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$
$\theta_{\max} = 25.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.199$
$S = 1.06$
2178 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1371P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*D*···O1	0.99	2.21	2.804 (3)	117
C7—H7*A*···O1$^{\rm i}$	0.98	2.54	3.469 (3)	159

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

Atoms C4 and C5 of the piperidine ring (see Fig. 1) are disordered. They were resolved by finding alternative positions from the difference Fourier map, and subsequently refined anisotropically over two positions with an occupancy of 0.607 (6) for C4*A* and C5*A*, and 0.393 (6) for the alternative positions C4*B* and C5*B*. H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H bond lengths of 0.95 (aromatic CH), 0.98 (CH_3), 0.99 (CH_2) or 0.95 Å (CH), and isotropic displacement parameters equal to 1.2 (CH and CH_2) or 1.5 (CH_3) times $U_{\rm eq}$ of the parent atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker (1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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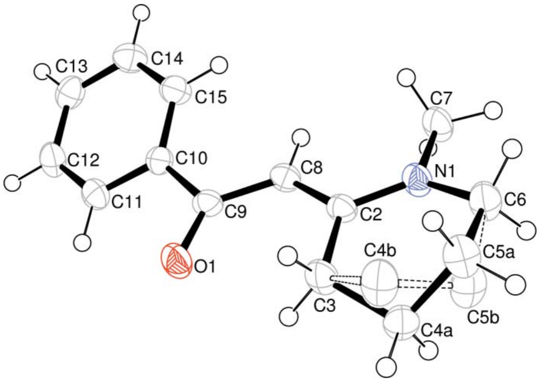

Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Atoms C4 and C5 in the piperidine ring are disordered; the minor occupancy disordered component is shown with dashed bonds.

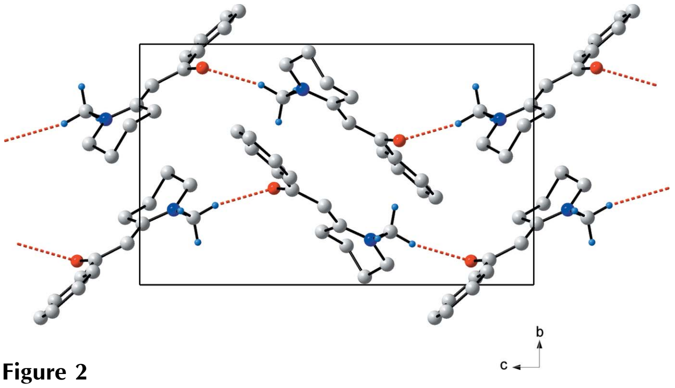

Figure 2
Packing diagram of (I), viewed along the *a* axis. The intermolecular C—H···O hydrogen bridges are shown as dashed red lines. All other H atoms and the minor occupancy disordered component have been omitted for clarity.

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