

Shu-Ping Huang,* Hai-Tang Li,
He-Ping Shi and Gai-Feng TanThe Institute of Modern Chemistry, Shanxi
University Taiyuan, People's Republic of China

Correspondence e-mail: sphuang@sxu.edu.cn

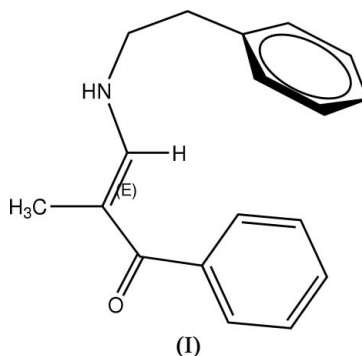
Key indicators

Single-crystal X-ray study
 $T = 183$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.068
 wR factor = 0.189
Data-to-parameter ratio = 14.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(E)-2-Methyl-3-(phenethylamino)-1-phenyl-
prop-2-en-1-one**The title compound, a secondary enamine $\text{PhCOC}(\text{CH}_3)\text{-CHNH}(\text{CH}_2)_2\text{Ph}$ or $\text{C}_{18}\text{H}_{19}\text{NO}$, is U-shaped. The dihedral angle between the two phenyl rings is $109.7(4)^\circ$. $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules to form a chain.

Received 20 December 2005

Accepted 17 March 2006

Comment

The enamine functional group can undergo nucleophilic attack at both its N and C atoms (Alvarez *et al.*, 2001). Enamines are an important class of compound used for selective alkylation and acylation of carbonyl compounds (Cook, 1988) and as valuable intermediates for the synthesis of biologically active natural products (Carruther, 1978). Enamines are known to exist in equilibrium with their imine isomers. The main reason that the title compound, (I), exists as an enamine and not an imine, as expected, is that there is an electron-withdrawing carbonyl group at the β -C atom of the enamine (Capon & Wu, 1990).

Selected geometric parameters of (I) are listed in Table 1 and the molecular structure is illustrated in Fig. 1. The bond lengths are as expected for an enamine group. Furthermore, the presence of H1 bonded to N1 confirms the formation of an enamine group.

It is clear from Fig. 1 that the alkene is in the *E* configuration. In the sequence of atoms C2–C1–C7–C8–C10–N1, all the C atoms are sp^2 hybridized. Despite the *E* configuration, these five C atoms are not in the same plane but form a helical motif. The torsion angles in Table 1 show that, in order to reduce the steric hindrance, some degree of planarity about the π -bond is lost. The dihedral angle between the two phenyl groups is $109.7(4)^\circ$, and their closest atom–atom contact is $3.587(7)$ Å between C3 and C16. $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds [$\text{N1}-\text{H1} = 0.86(3)$ Å, $\text{H1}\cdots\text{O1}^i = 2.11(3)$ Å, $\text{N1}\cdots\text{O1}^i = 2.943(3)$ Å and $\text{N1}-\text{H1}\cdots\text{O1}^i = 162(3)^\circ$; symmetry code: (i) $\frac{3}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$] link molecules into a chain along the *b* axis.

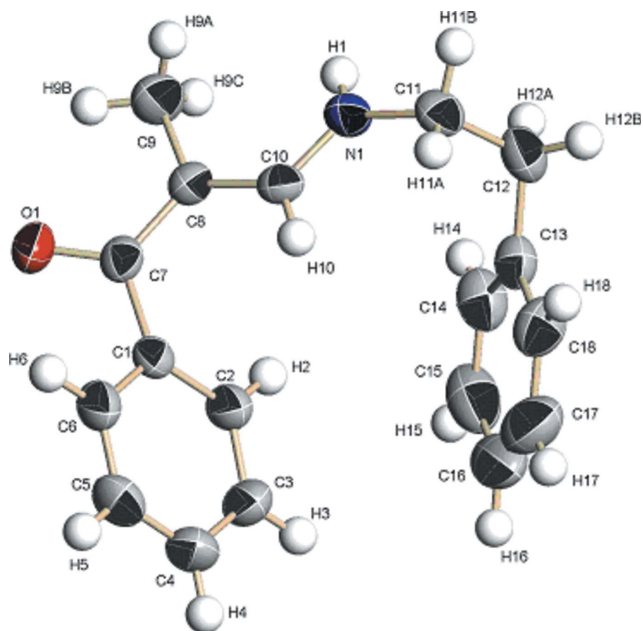


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by small spheres of arbitrary radii.

Experimental

The title compound was synthesized by a carbon–nitrogen transfer reaction. Under the protection of nitrogen, propiophenone (0.01 mol) was added dropwise to a reaction flask containing an equimolar solution of lithium diisopropylamide (0.01 mol) in tetrahydrofuran (20 ml) at about 195 K. The mixture was stirred for 2 h at room temperature, after which 3,4-dimethyl-1-tosylimidazolium iodide (0.01 mol) was added. After 3 h, the reaction mixture was poured into cold water (50 ml) and extracted with dichloromethane (3 × 30 ml). The organic extract was dried with MgSO₄ and concentrated under reduced pressure. The residue and phenylethylamine (0.01 mol) were added to acetonitrile (20 ml). The solution was refluxed until thin-layer chromatography indicated that the reaction was complete (3–4 h) and then concentrated under reduced pressure. The residue was purified by column chromatography over silica gel with ethyl acetate as eluent. Colourless single crystals of (I) were grown from a methanol solution.

Crystal data

C₁₈H₁₉NO
M_r = 265.34
 Monoclinic, *P*2₁/*n*
a = 10.046 (3) Å
b = 9.390 (3) Å
c = 16.060 (5) Å
 β = 101.916 (4)°
V = 1482.3 (8) Å³
Z = 4

D_x = 1.189 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 1696 reflections
 θ = 2.2–22.4°
 μ = 0.07 mm⁻¹
T = 183 (2) K
 Block, colourless
 0.30 × 0.30 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
*T*_{min} = 0.978, *T*_{max} = 0.978
 5910 measured reflections

2609 independent reflections
 1968 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.028
 θ _{max} = 25.0°
h = -11 → 10
k = -11 → 11
l = -19 → 15

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.068
wR(*F*²) = 0.189
S = 1.01
 2609 reflections
 186 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1103P)^2 + 0.14P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.18 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1–C7	1.236 (3)	C7–C8	1.431 (3)
N1–C10	1.335 (3)	C8–C10	1.359 (3)
N1–C11	1.448 (3)		
O1–C7–C8	122.0 (2)	C10–C8–C9	120.7 (2)
O1–C7–C1	116.5 (2)	N1–C10–C8	125.7 (2)
C10–C8–C7	121.1 (2)	N1–C10–H10	117.2
C2–C1–C7–O1	131.3 (2)	C7–C8–C10–N1	-176.8 (2)
C6–C1–C7–C8	141.4 (2)	C12–C13–C14–C15	-178.7 (2)
C1–C7–C8–C10	-22.2 (4)	C12–C13–C18–C17	178.9 (3)
C1–C7–C8–C9	162.9 (2)		

The H atom bonded to N1 was located in a difference map and refined isotropically. Other H atoms were positioned geometrically [C–H = 0.93–0.97 Å] and refined as riding, with *U*_{iso}(H) = 1.2 or 1.5 times *U*_{eq}(C).

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

References

- Alvarez, G., de Palomar, J. & Paz, J. L. G. (2001). *J. Mol. Struct. (Theochem)*, **541**, 111–117.
 Bruker (1998). SMART, SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.
 Capon, B. & Wu, Z.-P. J. (1990). *Org. Chem.* **55**, 2317–2324.
 Carruther, W. (1978). *Some Modern Methods of Organic Reactions*, p. 29. Cambridge University Press.
 Cook, A. G. (1988). *Enamines: Synthesis, Structure and Reactions*, 2nd ed., p. 103. New York: Marcel Dekker.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.