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Key indicators

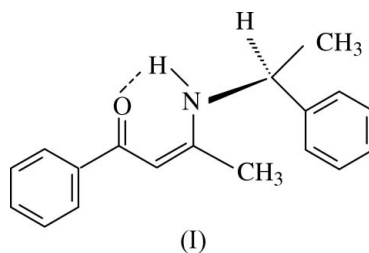
Single-crystal X-ray study
 $T = 297$ K
Mean $\sigma(\text{C}-\text{C}) = 0.010$ Å
 R factor = 0.057
 wR factor = 0.212
Data-to-parameter ratio = 9.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

1-Phenyl-3-[(1-phenylethyl)amino]but-2-en-1-one

The title compound, $\text{C}_{18}\text{H}_{19}\text{NO}$, has been synthesized by the condensation reaction of benzoylacetone and L-(−)- α -phenylethylamine. The enaminone structure is stabilized by a strong $\text{N}-\text{H}\cdots\text{O}=\text{C}$ intramolecular hydrogen bond.

Comment

Enaminones are not only important ligands in coordination chemistry (Doherty *et al.*, 1999; Kim *et al.*, 2001), but are also important chiral auxiliaries in organic synthesis (Christoffers, 2003). As part of an ongoing investigation of the chemistry of enaminones, we have synthesized the title compound, (I), by the condensation reaction of benzoylacetone and L-(−)- α -phenylethylamine (Shi *et al.*, 2004, 2005).



The title compound, (I), has been shown by ^1H NMR spectroscopy to exist in solution in the enaminone and not the enol-imine form. IR spectroscopy shows only the enaminone form in the solid state; this has been further confirmed by the determination of its crystal structure (Fig. 1).

As also noted in the related compound 1-phenyl-3-[(hydroxyethyl)amino]but-2-en-1-one (II) (Shi, 2005), the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$ fragment of the molecule of (I) is planar; the bond lengths indicate electron delocalization (Gilchrist, 1997). The $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$ plane is twisted by $18.6(3)^\circ$ with respect to the benzene ring (C1–C6). The C6–C7 bond, which is typical of a $\text{Csp}^2-\text{Csp}^2$ single bond, suggests that the benzene ring (C1–C6) is not involved in the conjugation of the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$ group (Table 1).

As in (II), an intramolecular $\text{N}-\text{H}\cdots\text{O}=\text{C}$ hydrogen bond is also present in the crystal structure of (I) (Table 2).

Experimental

The title compound was synthesized by refluxing an ethanol solution of benzoylacetone and L-(−)- α -phenylethylamine (1:1) for 24 h. After removal of the solvent, the oil was solidified by adding petroleum ether and further recrystallized from diethyl ether/petroleum ether (1:2 v/v; yield 79.6%). Colorless crystals suitable for X-ray analysis were obtained by slow evaporation of a diethyl ether solution at 278 K (m.p. 344.1–345.8 K). Analysis calculated for $\text{C}_{18}\text{H}_{19}\text{NO}$: C

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81.48, H 7.22, N 5.28%; found: C 81.23, H 7.62, N 5.00%. IR (KBr, cm^{-1}): 3058 (w, NH), 1578 (vs, O=C), 1543 (s, C=C). UV (λ_{max} , in DMF, nm): 344 (K-band, 1.92×10^4). $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 11.88 (1H, s, NH), 7.90–7.92, 7.41–7.45 (d, 2H, q, 3H, COC_6H_5), 7.35–7.38, 7.31–7.33, 7.26–7.29 (t, 2H, d, 2H, t, 1H, C_6H_5), 5.72 (s, 1H, CH), 4.75–4.80 (m, 1H, C*H), 1.96 (s, 3H, CH_3), 1.63–1.64 (d, 3H, C* CH_3).

Crystal data

$\text{C}_{18}\text{H}_{19}\text{NO}$

$M_r = 265.34$

Orthorhombic, $P2_12_12_1$

$a = 10.257$ (2) Å

$b = 11.856$ (2) Å

$c = 12.892$ (3) Å

$V = 1567.8$ (5) Å³

$Z = 4$

$D_x = 1.124$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 25 reflections

$\theta = 10$ – 13°

$\mu = 0.07$ mm⁻¹

$T = 297$ K

Prism, colorless

$0.20 \times 0.20 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction: ψ scan (North *et al.*, 1968)

$T_{\text{min}} = 0.974$, $T_{\text{max}} = 0.987$

1767 measured reflections

1767 independent reflections

810 reflections with $I > 2\sigma(I)$

$\theta_{\text{max}} = 26.0^\circ$

$h = 0 \rightarrow 12$

$k = 0 \rightarrow 14$

$l = 0 \rightarrow 15$

3 standard reflections every 200 reflections intensity decay: none

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.057$

$wR(F^2) = 0.212$

$S = 1.04$

1767 reflections

184 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0927P)^2 + 0.2856P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.13$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.13$ e Å⁻³

Extinction correction: *SHELXL97*

Extinction coefficient: 0.120 (13)

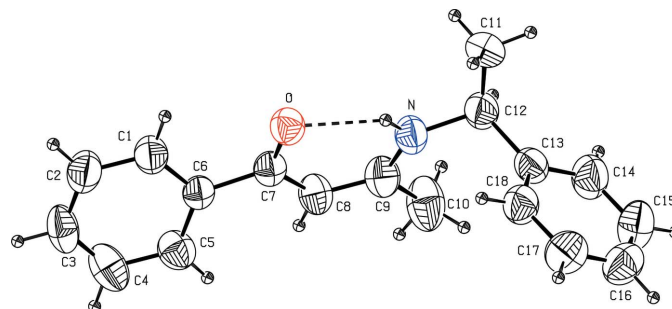


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates the intramolecular hydrogen bond.

The absolute configuration of (I) was assigned by reference to an unchanging chiral center for L-(−)- α -phenylethylamine in the synthetic procedure. In the absence of significant anomalous scattering effects, Friedel pairs were merged. All H atoms were placed at geometrically idealized positions, with C–H = 0.93–0.98 Å and N–H = 0.86 Å, and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ and $1.5U_{\text{eq}}(\text{methyl C})$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Table 1

Selected geometric parameters (Å, °).

O–C7	1.256 (6)	C8–C9	1.370 (8)
N–C9	1.321 (7)	C9–C10	1.514 (9)
N–C12	1.473 (7)	C11–C12	1.516 (10)
C6–C7	1.487 (7)	C12–C13	1.509 (8)
C7–C8	1.405 (8)		
O–C7–C6	118.6 (5)	N–C9–C10	117.8 (6)
O–C7–C8	122.3 (5)	C8–C9–C10	120.3 (7)
C6–C7–C8	119.1 (5)	N–C12–C11	107.9 (6)
C9–N–C12	127.7 (6)	N–C12–C13	112.7 (5)
C7–C8–C9	124.9 (6)	C11–C12–C13	113.0 (6)
N–C9–C8	121.8 (6)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N–H1N \cdots O	0.86	1.99	2.663 (6)	135

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