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(*Z*)-4-Anilinopent-3-en-2-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.139
Data-to-parameter ratio = 11.6

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

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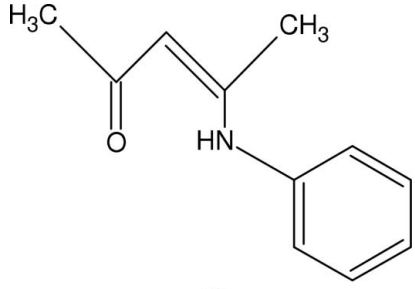
The title compound, $C_{11}H_{13}NO$, crystallizes as the *Z* isomer of the β-enamino–ketone. An intramolecular hydrogen-bonding interaction exists between the N—H and C═O groups.

Comment

The chelating tendencies of β-diketones have been extensively studied because of their applications in pharmaceutical compounds, biochemistry, biomedical research and immunochemistry (Sandler & Karo, 1986; Chen & Rhodes, 1996) and as precursors for the preparation of a large number of heterocyclic compounds (Khosropour *et al.*, 2004). Previous studies of some cyclizations involved the preparation of 4-methylaminopent-3-en-2-ones (Kascheres *et al.*, 2001), 5-(1-methylethylaminomethylene)-1,3-dioxane-4,6-diones and 5-(dimethylethylaminomethylene)-2,2-dimethyl-1,3-dioxane-4,6-diones (Zhuo, 1997). The anticonvulsant activities of various acyclic and cyclic enaminoketones have been studied in connection with their lipophilicity, steric, electronic and hydrogen-bonding effects (Edafiogho *et al.*, 1994; Hinko *et al.*, 1993). The condensation products of acetylacetone with various amines exist in three forms, *viz*. the Schiff base, the ketamine and the enimine. The interchange between the ketamine and enimine tautomers involves a small displacement in the equilibrium position of the acidic proton.



(I)

In the title compound, (I), the C2═O1 and C2—C3 bond distances [1.224 (3) and 1.424 (4) Å, respectively] confirm the existence of the enamino–ketone. The bond distances in the C3═C4—N1 chain indicate greater electron delocalization [C3═C4 = 1.365 (3) Å and N1—C4 = 1.365 (3) Å]. The enamino–ketone fragment (N1—C4═C3—C2═O1) is essentially planar, the maximum deviation being 0.011 (3) Å for atom C2. The dihedral angle between this fragment and the phenyl ring is 32.06 (9)°. This is a result of the steric hindrance between the C5-methyl group and the phenyl ring, which hinders conjugation between the two groups. The

molecule is the *Z* isomer and an intramolecular hydrogen bond is present between the keto and imino groups [N1—H1 = 0.89 (3) Å, H1···O1 = 1.92 (3) Å, N1···O1 = 2.675 (3) Å and N1—H1···O1 = 140 (2)°].

Experimental

The title compound was prepared by modification of a published method (Fustero *et al.*, 1999). Aniline (15.26 ml, 0.1 mol) was added to acetylacetone (20 ml, 0.1 mol) and two drops of H_2SO_4 in benzene (50 ml). The reaction mixture was refluxed for 4 h. On cooling, the product was filtered and recrystallized from a chloroform/*n*-hexane (9:1 *v*/*v*) mixture.

Crystal data

$C_{11}H_{13}NO$
M_r = 175.22
Monoclinic, $P2_1/c$
a = 9.157 (3) Å
b = 11.040 (7) Å
c = 10.130 (5) Å
β = 106.10 (3)°
V = 983.9 (9) Å^3
Z = 4
D_x = 1.183 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
θ = 6.0–17.8°
μ = 0.08 mm^{-1}
T = 293 (2) K
Prism, colorless
0.60 × 0.40 × 0.40 mm

Data collection

Philips PW 1100 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF [local program based on Walker & Stuart (1983)]
T_{min} = 0.893, T_{max} = 0.970
1812 measured reflections
1717 independent reflections
1120 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 12$
1 standard reflections
every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.139
S = 1.04
1717 reflections
148 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.3446P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Methyl H atoms were located in difference Fourier syntheses and refined as rigid rotating groups, riding on their parent atoms, with C—H = 0.96 Å and U_{iso}(H) = U_{eq}(C). All other H atoms were found in a difference map and refined freely.

Data collection: local program; cell refinement: local program; data reduction: local program; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PARST* (Nardelli, 1995).

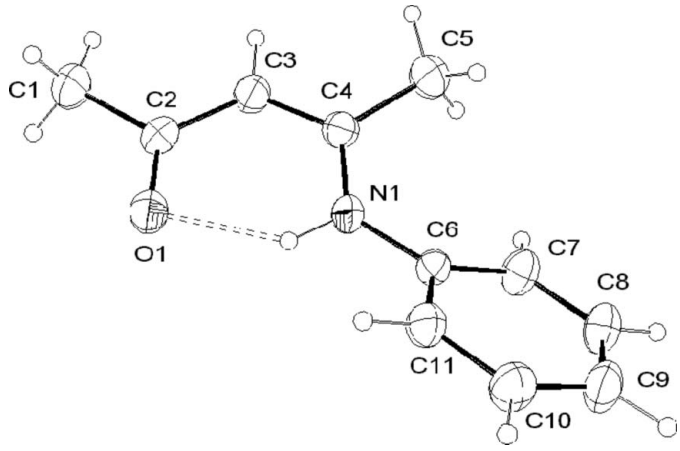


Figure 1
View of the structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The dashed lines indicate the intramolecular hydrogen bond.

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