Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

3-Acetoxy-4-(4-hydroxyphenethylamino)-but-3-en-1-one

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Received 21 March 2005
Accepted 6 April 2005
Online 16 April 2005

Key indicators

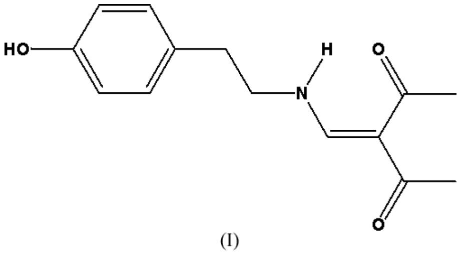
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.049
wR factor = 0.112
Data-to-parameter ratio = 7.1

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

In the crystal structure of the title compound, $C_{14}H_{17}NO_3$, intermolecular O—H···O hydrogen bonds [O···O = 2.692 (4) Å] link the molecules into extended one-dimensional chains. These chains are, in turn, linked into a three-dimensional network by weak intermolecular C—H···O interactions [C···O = 3.368 (5) Å].

Comment

Recently, secondary enamines have attracted a great deal of attention (Duthaler, 2003; Stanovnik & Svete, 2004; Elassar & El-Khair, 2003). Metalloenamine carbanions, derived from enamines or enolizable imines, are useful substrates for regio- and stereoselective C—C bond-formation reactions with electrophilic reagents. Our interest is in the synthesis of secondary enamine derivatives based on tetrahydrofolate coenzyme models (Li *et al.*, 2004), which can transfer the —C═C— or ═CH— unit *via* mechanisms analogous to those operative in biochemical processes (Bieraugel *et al.*, 1983; Pandit & Bieraugel, 1979). As part of our studies, we have prepared the title compound, (I), and determined its crystal structure.



(I)

Selected geometric parameters for (I) are listed in Table 1 and the molecular structure is illustrated in Fig. 1. The N1—C9 bond length of 1.301 (5) Å is shorter than the normal N—C

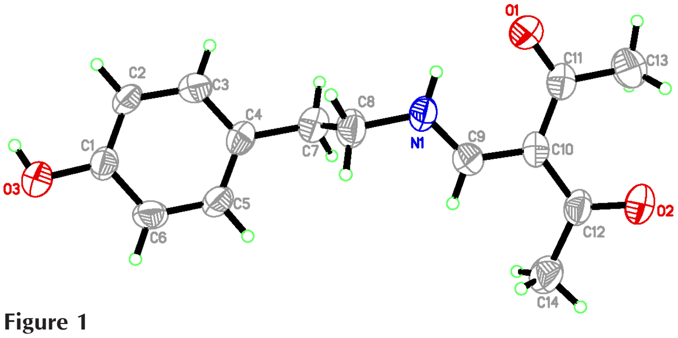


Figure 1
A view of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

doi:10.1107/S1600536805010706

bond (1.47 Å based on covalent radii; Orpen *et al.*,, 1992). This, in conjunction with the N1—C9—C10 angle of 127.8 (4)°, indicates the presence of delocalized electron density about N1/C9/C10.

In the crystal structure of (I), intermolecular O—H···O hydrogen bonds link the molecules into extended one-dimensional chains along the vector [310] (see Table 2 for hydrogen-bond geometries). These stronger interactions are formed by a hydroxyl O atom acting as a donor and a carbonyl O atom acting as an acceptor (Fig. 2). Furthermore, weaker C—H···O interactions connect the one-dimensional chains into a three-dimensional network. An intramolecular N—H···O hydrogen bond is also present, with graph set *S*(6) (Bernstein *et al.*, 1995).

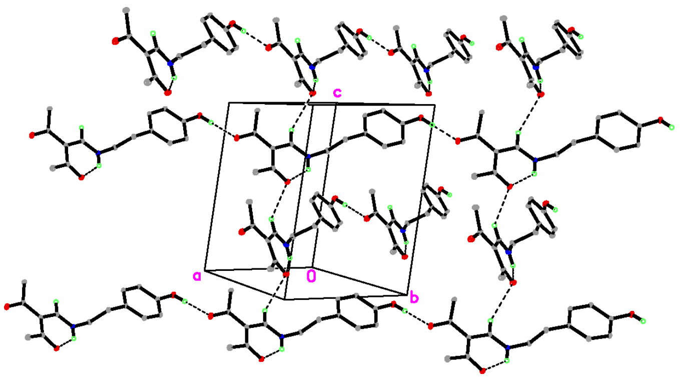


Figure 2
The packing of (I). Dashed lines indicate hydrogen bonds and H atoms not involved in hydrogen bonding have been omitted.

Experimental

Sodium hydride (1.8 mmol) was added to a solution of acetylacetone (1.5 mmol) in dry tetrahydrofuran (10 ml), which was cooled with an ice–water bath. The reaction mixture was stirred for 30 min, 1-(3-nitrophenylsulfonyl)-3,4-dimethylimidazolinium iodide (1 mmol) was added and the mixture was allowed to warm to room temperature, continuously stirred for 3 h, and then quenched with water. The solution was extracted with dichloromethane, dried over anhydrous sodium sulfate, and concentrated. The residue was purified by column chromatography to afford *N*,*N*,*N*′-trisubstituted 2-methylethylenediamines as a yellow oil. A solution of *N*,*N*,*N*′-trisubstituted 2-methylethylenediamines (1 mmol) and 4-hydroxyphenylethylamine (1 mmol) in anhydrous acetonitrile (10 ml) was refluxed, concentrated, and the residue purified by column chromatography to afford the title compound as white crystals. Compound (I) (100 mg) was dissolved in ethanol (10 ml). The solution was allowed to evaporate slowly over several days and colourless crystals suitable for X-ray analysis were collected.

Crystal data

$C_{14}H_{17}NO_3$
$M_r = 247.29$
Monoclinic, *Cc*
$a = 7.400\ (2)$ Å
$b = 16.462\ (5)$ Å
$c = 10.999\ (3)$ Å
$\beta = 98.558\ (4)^\circ$
$V = 1324.9\ (7)$ Å^3
$Z = 4$

$D_x = 1.240$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3626 reflections
$\theta = 2.5$–26.8°
$\mu = 0.09$ mm^{-1}
$T = 298\ (2)$ K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.974$, $T_{\max} = 0.983$
3186 measured reflections

1173 independent reflections
1134 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\max} = 25.0^\circ$
$h = -8 \rightarrow 8$
$k = -19 \rightarrow 16$
$l = -10 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.112$
$S = 1.20$
1173 reflections
166 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 0.4543P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C9—N1	1.301 (5)	N1—C8	1.452 (5)
C9—C10	1.388 (5)		
N1—C9—C10	127.8 (4)	N1—C8—C7	112.7 (3)
C9—N1—C8	124.4 (4)		
C9—N1—C8—C7	−90.7 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1	0.86	2.00	2.629 (4)	129
O3—H3*A*···O2$^{\rm i}$	0.82	1.87	2.692 (4)	179
C9—H9*A*···O1$^{\rm ii}$	0.93	2.58	3.368 (5)	143

Symmetry codes: (i) $x - \frac{3}{2}, y + \frac{1}{2}, z$; (ii) $x, -y, z + \frac{1}{2}$.

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, O—H = 0.82 Å and N—H = 0.86 Å. They were included in a riding-model approximation, with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C,N) or $1.5U_{\rm eq}$(methyl-C,O). In the absence of significant anomalous dispersion effects, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SHELXTL* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge financial support of this work by the Natural Science Foundation of China (grant No. 20272034 to CZX). The authors are also grateful for the support of the Shanxi Provincial Natural Science Foundation (grant Nos. 20041007 and 20041006).

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