organic papers

3-[(2-Hydroxyphenyl)amino]-1-phenylbut-3-en-1-one ethanol solvate

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The title compound, $C_{16}H_{15}NO_2 \cdot C_2H_5OH$, has been synthesized by the reaction of benzoylacetone with 2-aminophenol in ethanol. The enaminone structure is stabilized by a strong intramolecular hydrogen bond (N—H$\cdots$O=C), while the one-dimensional infinite chain in the [010] direction is formed by the intermolecular O—H$\cdots$O hydrogen bond between the enaminone and the ethanol solvent molecule.

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.189
Data-to-parameter ratio = 17.2

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

Comment

Enaminones have been studied extensively, not only because of their very diverse reaction chemistry, but also owing to their applications in coordination chemistry (Kim *et al.*, 2001; Doherty *et al.*, 1999). As part of a systematic investigation of the chemistry of enaminones, the enaminone component of the title compound, (I), was obtained (Shi, Yang, Shen *et al.*, 2004; Shi, Yang, Song *et al.*, 2004) and its crystal structure is reported here.

(I)
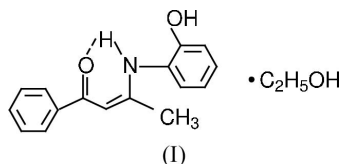
The parent unsolvated enaminone has already been studied (Glowiak & Sobczak, 1992). It crystallizes as two polymorphs, the orange triclinic form with two molecules, (I*a*) and (I*b*), in the asymmetric unit, and the colorless monoclinic form, (I*c*). Although the corresponding bond distances and angles in (I) are very similar to those in (I*a*), (I*b*) and (I*c*), the most striking
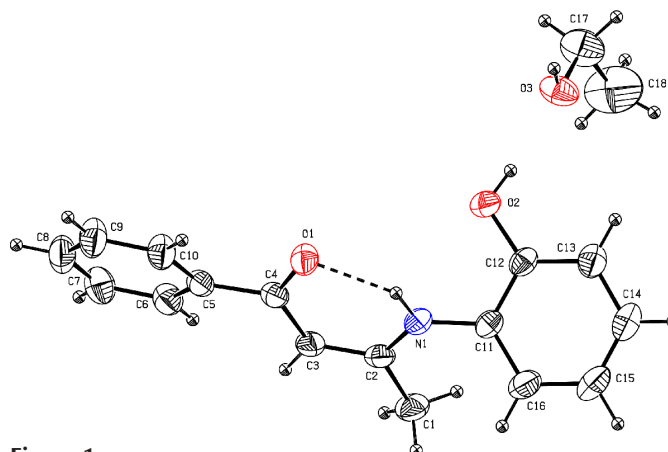

Figure 1
The asymmetric unit of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is shown as a dashed line.
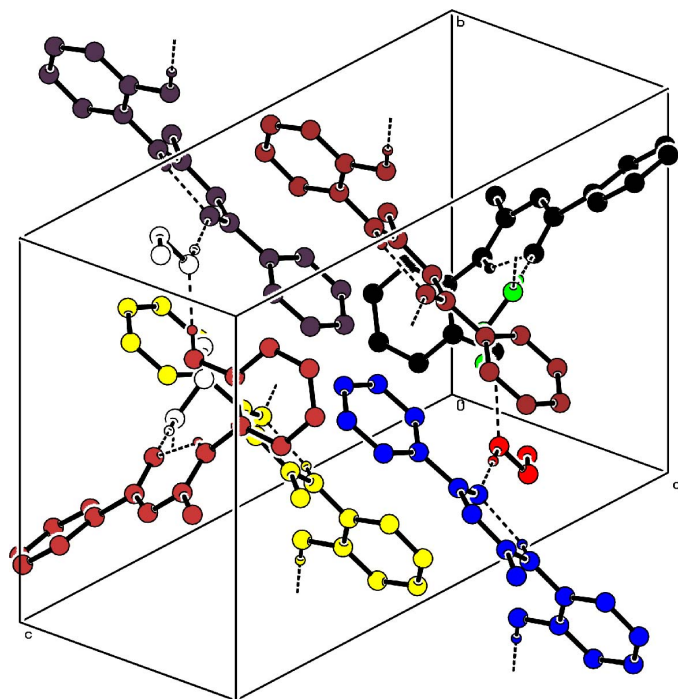
Figure 2
Packing diagram of the title compound. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

difference between them lies in the dihedral angles between the O=C—C=C—N plane and the benzene and substituted benzene rings. The dihedral angles are 18.8 and 41.0° for (Ia), 14.3 and 56.8° for (Ib) and 28.1 and 85.5° for (Ic); in (I), the values are 21.96 (13) and 46.68 (13)°. For each of (I), (Ia), (Ib) and (Ic), the bond lengths in the O=C—C=C—N system indicate electron delocalization (Shi, Yang, Shen *et al.*, 2004; Shi, Yang, Song *et al.*, 2004; Gilli *et al.*, 2000; Arici *et al.*, 1999). Interestingly, the above dihedral angles suggest that neither the benzene nor the substituted benzene rings are involved in the conjugation of the O=C—C=C—N system.

As in (Ia), (Ib) and (Ic), the enamine N atom and carbonyl O atom in (I) form a strong intramolecular hydrogen bond which stabilizes the enaminone (Table 2). Strong inter-molecular O—H···O hydrogen bonds are also present in the crystal structure. These lead to infinite one-dimensional chains in the [010] direction.

Experimental

The title compound was synthesized by refluxing a solution of benzoylacetone and 2-aminophenol (1:1) in ethanol for 6 h. It was recrystallized from ethanol as yellow crystals which are unstable and easily lose ethanol to form the yellow enaminone [90% yield; m.p. 443–444 K; literature m.p. 437–438 K (colorless crystals), 437–439 K (orange crystals) (Glowiak & Sobczak, 1992)]. IR (KBr, cm^{-1}): 3159.61 (*br, s*, NH, OH), 1604.55 (*vs*, C=O), 1574.13 (*s*, C=C). UV (λ_{max}, in DMF): 256.00 (B band, 1.36×10^4), 359.5 (K band, 3.52×10^4) nm. ^{1}H NMR (300 MHz, CDCl$_3$): δ 12.35 (1H, *s*, NH), 8.61 (1H, *s, br*, OH), 7.84–7.87, 7.41–7.43 (2H, *m*, 3H, *m*, C$_6$H$_5$), 6.82–7.03 (4H, 3*m*, C$_6$H$_4$), 5.66 (1H, *s*, CH), 1.61 (3H, *s*, CH$_3$).

Crystal data

C$_{16}$H$_{15}$NO$_2$·C$_2$H$_6$O	$D_x = 1.172$ Mg m^{-3}
$M_r = 299.36$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 874
$a = 7.663$ (4) Å	reflections
$b = 12.119$ (7) Å	$\theta = 2.8$–24.6°
$c = 18.303$ (9) Å	$\mu = 0.08$ mm^{-1}
$\beta = 93.217$ (14)°	$T = 293$ (2) K
$V = 1697.1$ (16) Å^3	Block, yellow
$Z = 4$	$0.24 \times 0.22 \times 0.16$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3484 independent reflections
	1948 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.033$
Absorption correction: multi-scan	$\theta_{max} = 26.5°$
(*SADABS*; Sheldrick, 1996;	$h = -9 \rightarrow 6$
Blessing, 1995)	$k = -11 \rightarrow 15$
$T_{min} = 0.967$, $T_{max} = 0.987$	$l = -21 \rightarrow 23$
9539 measured reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.098P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.058$	$+ 0.1387P]$
$wR(F^2) = 0.189$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
3484 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
203 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.264 (3)	C2—C3	1.385 (4)
O2—C12	1.370 (3)	C3—C4	1.420 (3)
N1—C2	1.339 (3)	C4—C5	1.496 (3)
N1—C11	1.414 (3)		
C2—N1—C11	130.3 (2)	C2—C3—C4	124.1 (2)
N1—C2—C3	120.6 (2)	O1—C4—C3	121.9 (2)
N1—C2—C1	120.1 (2)	O1—C4—C5	118.5 (2)
C3—C2—C1	119.4 (2)	C3—C4—C5	119.6 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	1.92	2.628 (3)	139
O2—H2···O3	0.82	1.84	2.657 (3)	171
O3—H3···O1^i	0.82	1.86	2.685 (3)	178

Symmetry code: (i) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

All H atoms were located in difference maps and were subse-quently treated as riding atoms, with C—H = 0.93 (olefinic and aromatic), 0.96 (methyl) or 0.97 Å (methylene), N—H = 0.86 Å and O—H = 0.82 Å. U_{iso}(H) values were set at $1.2U_{eq}$(C,N), $1.5U_{eq}$(C$_{methyl}$) or $1.5U_{eq}$(O).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997a); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2005); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997b).

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