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3-(3-Hydroxyanilino)-1,3-diphenylprop-2-en-1-one

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Key indicators

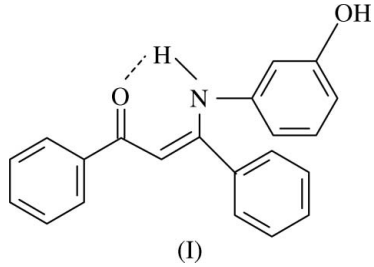
Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.004 Å
R factor = 0.086
wR factor = 0.163
Data-to-parameter ratio = 13.8

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

The asymmetric unit of the title compound, $C_{21}H_{17}NO_2$, contains two discrete molecules with similar geometric parameters. Intramolecular N—H···O=C hydrogen bonding stabilizes each of the two molecules. Intermolecular O—H···O=C hydrogen bonds form two [100] chains having $R_2^2(16)$ rings. Intermolecular C—H···O—H and C—H···O=C hydrogen bonds lead to the formation of $R_2^2(7)$ and $R_2^1(6)$ rings.

Comment

Enaminones have been studied not only as ligands in coordination chemistry (Shi *et al.*, 2004, 2005), but also as materials for nitrogen-containing heterocycles and chiral auxiliaries in organic synthesis (Christoffers *et al.*, 2003). As part of a continuing investigation of the chemistry of enaminones, the title compound, (I), has been synthesized by the condensation of dibenzoylmethane and 3-aminophenol (Shi & Zhang, 2005*a*,*b*; Shi *et al.*, 2006).



(I)

The asymmetric unit of the title compound contains two independent molecules (*A* and *B*). In these molecules, the bond distances are almost identical. As also observed for the related structure of 3-(2-hydroxyphenyl)amino-1,3-diphenylprop-2-en-1-one, (II) (Shi & Zhang, 2005*b*), the O=C—C=C—N fragment is planar. The bond distances indicate electron delocalization (Shi *et al.*, 2006). The O=C—C=C—N fragment forms dihedral angles with the three benzene rings of 28.48 (16)° (C1–C6 ring), 47.86 (16)° (C10–C15 ring) and 39.57 (15)° (C16–C21 ring) for molecule *A* and 26.88 (16)° (C22–C27 ring), 45.55 (15)° (C31–C36 ring) and 52.33 (15)° (C37–C42 ring) for molecule *B*. Interestingly, the above dihedral angles suggest that the three benzene rings in each of molecules *A* and *B* are not involved in the conjugation of the O=C—C=C—N unit. This conclusion is further supported by the C6—C7, C9—C10 and N1—C16 bond distances for molecule *A* and the C27—C28, C30—C31 and N2—C37 distances for molecule *B* (Table 1), which are typical of Csp^2—Csp^2 and Nsp^2—Csp^2 single bonds.

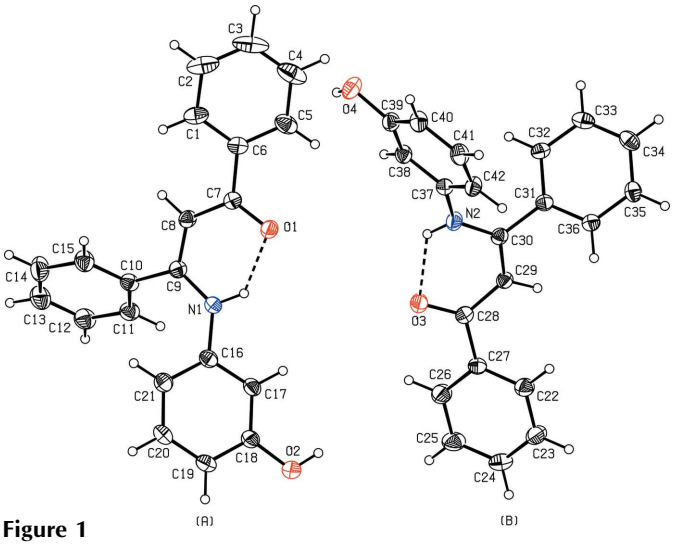


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are drawn as dashed lines.

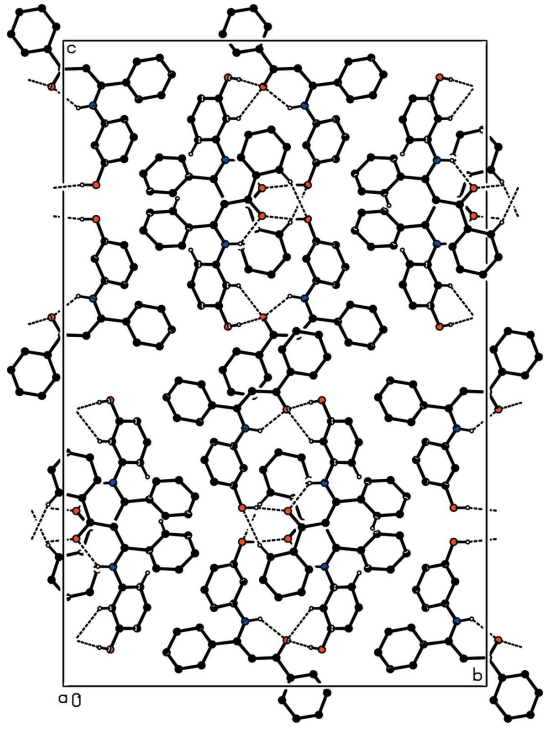


Figure 2
The crystal structure of (I). Hydrogen bonds are drawn as dashed lines. H atoms not involved in these interactions have been omitted.

Like the other enaminones previously reported, the title compound exhibits strong N—H···O═C intramolecular hydrogen bonds which stabilize molecules *A* and *B* (Table 2). Furthermore, three types of O—H···O═C, C—H···O—H and C—H···O═C intermolecular hydrogen bonds are found (Fig. 2). The O2—H2O···O3^{i} hydrogen bond (symmetry codes as in Table 2) generates one [100] chain having R_2^2 (16) rings (Bernstein *et al.*, 1995); the O4—H4O···O1^{ii} hydrogen bond forms another chain in the [100] direction, whereas the O—H···O═C hydrogen bonds in (II) result in centrosymmetric R_2^2 (14) dimers. The C26—H26···O2^{ii} and C38—H38···O1^{ii} hydrogen bonds lead to the formation of R_2^2 (7) and R_2^1(6) rings. In addition, the title compound displays a C—H···π hydrogen bond involving atom C36 in molecule *B* at (*x*, *y*, *z*) as a hydrogen-bond donor and phenyl group C31–C36 (centroid *Cg*5) in the type *B* molecule at ($x-\frac{1}{2}$, $-y-\frac{1}{2}$, $-z$) as an acceptor.

Experimental

A solution of dibenzoylmethane (0.449 g, 2 mmol) and 3-aminophenol (0.218 g, 2 mmol) in 20 ml of absolute ethanol in the presence of *p*-TsOH (10 mg) was refluxed for 4 d. After removal of the solvent *in vacuo*, the residue was separated with the eluant (diethyl ether and petroleum ether, *v*/*v* 1:2) by thin layer chromatography to give the title compound as a yellow solid. Recrystallization from dichloromethane and petroleum ether (2:3 *v*/*v*) afforded crystals suitable for X-ray analysis (m.p. 442.45–442.95 K).

Crystal data

$C_{21}H_{17}NO_2$
M_r = 315.36
Orthorhombic, *Pbca*
a = 7.1855 (5) Å
b = 24.4898 (19) Å
c = 37.352 (3) Å
V = 6572.9 (9) Å^3
Z = 16
D_x = 1.275 Mg m^{-3}
Mo *K*α radiation
μ = 0.08 mm^{-1}
T = 193 K
Block, yellow
0.50 × 0.30 × 0.24 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.963, T_{max} = 0.980
57180 measured reflections
6016 independent reflections
5034 reflections with $I > 2\sigma(I)$
R_{int} = 0.081
θ_{max} = 25.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.087
$wR(F^2)$ = 0.163
S = 1.30
6016 reflections
436 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 4.4272P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C6—C7	1.484 (4)	C27—C28	1.490 (4)
C7—O1	1.268 (3)	C28—O3	1.258 (3)
C7—C8	1.414 (4)	C28—C29	1.429 (4)
C8—C9	1.378 (4)	C29—C30	1.371 (4)
C9—N1	1.345 (4)	C30—N2	1.353 (4)
C9—C10	1.486 (4)	C30—C31	1.492 (4)
C16—N1	1.425 (4)	C37—N2	1.426 (4)
C18—O2	1.374 (4)	C39—O4	1.369 (3)
O1—C7—C8	122.4 (3)	O3—C28—C27	120.0 (3)
O1—C7—C6	117.8 (3)	C29—C28—C27	118.7 (3)
C8—C7—C6	119.8 (3)	C30—C29—C28	124.6 (3)
C9—C8—C7	124.9 (3)	N2—C30—C29	122.1 (3)
N1—C9—C8	120.6 (3)	N2—C30—C31	118.4 (2)
N1—C9—C10	121.0 (3)	C29—C30—C31	119.4 (3)
C8—C9—C10	118.2 (3)	C9—N1—C16	130.2 (2)
O3—C28—C29	121.3 (3)	C30—N2—C37	128.3 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O3^{i}	0.84	1.87	2.699 (3)	171
O4—H4O···O1^{ii}	0.84	1.87	2.706 (3)	175
N1—H1N···O1	0.88	1.98	2.653 (3)	132
N2—H2N···O3	0.88	2.04	2.681 (3)	129
C38—H38···O1^{ii}	0.95	2.48	3.189 (4)	132
C26—H26···O2^{ii}	0.95	2.59	3.391 (4)	143
C36—H36···*Cg*5^{iii}	0.95	2.68	3.488 (3)	144

Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$; (iii) $x-\frac{1}{2}, -y-\frac{1}{2}, -z$. Cg5 is the centroid of the ring C31–C36.

All H atoms were placed at geometrically idealized positions and subsequently treated as riding atoms, with C—H = 0.95 Å, N—H = 0.88 Å and O—H = 0.84 Å, and U_{iso}(H) values of $1.2U_{eq}$(C,N) and $1.5U_{eq}$(O).

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); 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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