

Yao-Cheng Shi* and
Su-Hua ZhangSchool of Chemistry, Yangzhou University, 130
XiMenWai Street, Yangzhou 225002, People's
Republic of China

Correspondence e-mail: yzssyc@yzcn.net

Key indicators

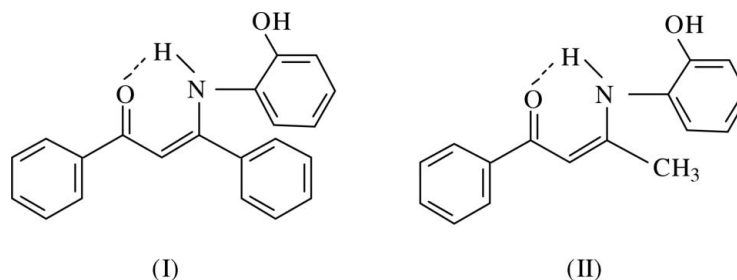
Single-crystal X-ray study
 $T = 295\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
 R factor = 0.051
 wR factor = 0.154
Data-to-parameter ratio = 13.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3-[(2-Hydroxyphenyl)amino]-1,3-diphenyl-
prop-2-en-1-one

The title compound, $\text{C}_{21}\text{H}_{17}\text{NO}_2$, has been synthesized by the condensation of dibenzoylmethane and 2-aminophenol in the presence of *p*-TsOH. In the crystalline state, molecules are linked by intermolecular $\text{O}-\text{H}\cdots\text{O}=\text{C}$ hydrogen bonds which, in combination with intramolecular $\text{N}-\text{H}\cdots\text{O}$ bonds, form centrosymmetric $R_2^2(14)$ dimers.

Received 24 October 2005
Accepted 24 November 2005
Online 30 November 2005

Comment

Enaminones have been studied extensively not only because of their plentiful reaction chemistry (Christoffers *et al.*, 2003; Tietze *et al.*, 1989), but also owing to their applications in coordination chemistry (Kim *et al.*, 2001; Doherty *et al.*, 1999) and as chiral auxiliaries in organic synthesis (Christoffers, 2003). In a continuation of an investigation of the chemistry of enaminones, we have prepared the title compound, (I), by the condensation of dibenzoylmethane and 2-aminophenol (Shi *et al.*, 2004, 2005).



The title compound has been shown by ^1H NMR spectroscopy to exist in solution in the enaminone and not the enol-imine form (Shi *et al.*, 2004, 2005). IR spectroscopy shows only the enaminone form in the solid state; this has been further confirmed in this study of its crystal structure (Fig. 1).

As also observed in the related compound 3-[(2-hydroxyphenyl)amino]-1-phenylbut-2-en-1-one, (II) (Shi, 2005), the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$ linkage of the molecule of (I) is planar; the bond lengths indicate electron delocalization (Gilli *et al.*, 2000). Moreover, none of the three benzene rings ($\text{C}11-\text{C}16$, $\text{C}21-\text{C}26$ and $\text{C}31-\text{C}36$) is coplanar with the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$ plane; they make dihedral angles of 29.04 (19), 58.6 (2) and 35.53 (17) $^\circ$, respectively, with the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$ plane. Furthermore, the $\text{C}1-\text{C}11$ and $\text{C}3-\text{C}21$ bond lengths, which are both typical of a single bond ($\text{C}sp^2-\text{C}sp^2$), suggest that the $\text{C}11-\text{C}16$ and $\text{C}21-\text{C}26$ rings are not involved in the conjugation of the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$ chain (Table 1).

As in (II), a strong intramolecular hydrogen bond between the enamine N atom and carbonyl O atom in (I) stabilizes the

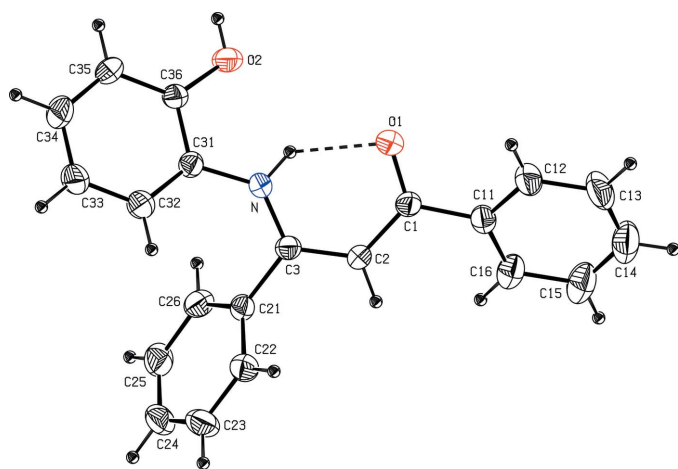


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is shown as a dashed line.

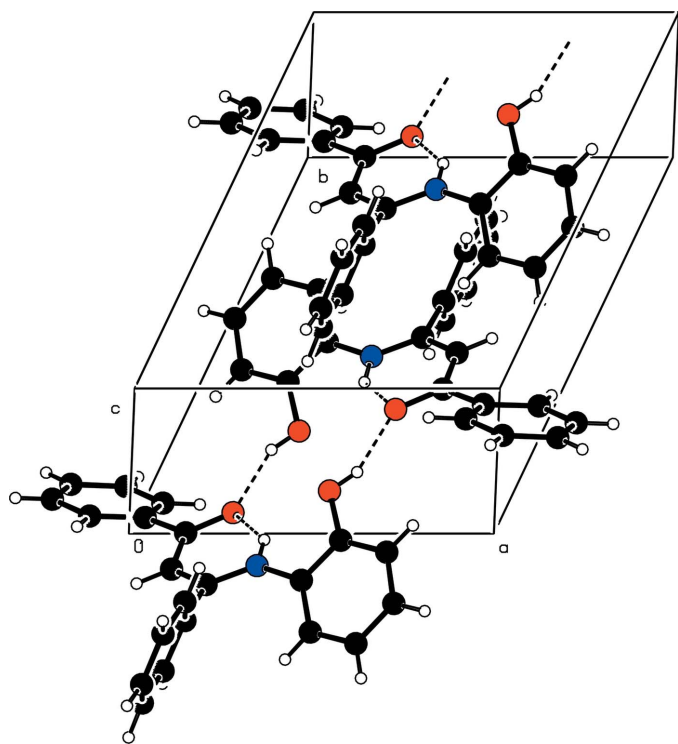


Figure 2
Packing diagram of (I). Dashed lines indicate hydrogen bonds.

enaminone, while intermolecular $O-H \cdots O=C$ hydrogen bonds form centrosymmetric $R_2^2(14)$ dimers of (I) (Table 2 and Fig. 2). In addition, $C-H \cdots O-H$ hydrogen bonds are present in the crystal structure of (I) (Table 2).

Experimental

An ethanol solution of dibenzoylmethane and 2-aminophenol (1:1) in the presence of a catalytic trace amount of *p*-TsOH was refluxed for 24 h. After removal of the solvent, the residue was chromatographed

on silica-gel plates with CH_2Cl_2 and ethyl acetate (50:1 *v/v*). The yellow band was collected and further recrystallized from an ethanol solution at 278 K to afford the title compound (*m.p.* 481.6–482.7 K). IR (KBr): 1583 (*vs*, $C=C$), 1634 (*vs*, $C=O$), 3160 cm^{-1} (*br, m, NH*); 1H NMR (600 MHz, $CDCl_3$): δ 6.570 (*s*, 1H, OH), 6.635 (*s*, 1H, CH), 6.623–6.647, 6.937–6.949, 6.984–7.012, 7.353–7.423, 7.495–7.520, 7.544–7.566, 8.032–8.048 (*t*, 1H, *d*, 1H, *t*, 1H, *m*, 5H, *t*, 2H, *t*, 2H, *d*, 2H, 14H of three benzene rings), 12.677 (*s*, 1H, NH).

Crystal data

| | |
|-----------------------------|--|
| $C_{21}H_{17}NO_2$ | $Z = 2$ |
| $M_r = 315.36$ | $D_x = 1.217\text{ Mg m}^{-3}$ |
| Triclinic, $P\bar{1}$ | Mo $K\alpha$ radiation |
| $a = 9.0490(18)\text{ \AA}$ | Cell parameters from 25 reflections |
| $b = 10.242(2)\text{ \AA}$ | $\theta = 9\text{--}12^\circ$ |
| $c = 11.013(2)\text{ \AA}$ | $\mu = 0.08\text{ mm}^{-1}$ |
| $\alpha = 69.26(3)^\circ$ | $T = 295\text{ K}$ |
| $\beta = 81.68(3)^\circ$ | Prism, yellow |
| $\gamma = 64.33(3)^\circ$ | $0.30 \times 0.20 \times 0.20\text{ mm}$ |
| $V = 860.3(4)\text{ \AA}^3$ | |

Data collection

| | |
|---|------------------------------------|
| Enraf–Nonius CAD-4 diffractometer | $R_{\text{int}} = 0.030$ |
| $\omega/2\theta$ scans | $\theta_{\text{max}} = 25.0^\circ$ |
| Absorption correction: ψ scan (North <i>et al.</i> , 1968) | $h = 0 \rightarrow 10$ |
| $T_{\text{min}} = 0.972$, $T_{\text{max}} = 0.981$ | $k = -10 \rightarrow 12$ |
| 3224 measured reflections | $l = -12 \rightarrow 13$ |
| 3018 independent reflections | 3 standard reflections |
| 1453 reflections with $I > 2\sigma(I)$ | every 200 reflections |
| | intensity decay: none |

Refinement

| | |
|---------------------------------|---|
| Refinement on F^2 | $w = 1/[\sigma^2(F_o^2) + (0.0521P)^2]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.051$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.154$ | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| $S = 1.02$ | $\Delta\rho_{\text{max}} = 0.17\text{ e \AA}^{-3}$ |
| 3018 reflections | $\Delta\rho_{\text{min}} = -0.17\text{ e \AA}^{-3}$ |
| 219 parameters | Extinction correction: <i>SHELXL97</i> |
| H-atom parameters constrained | Extinction coefficient: 0.035 (5) |

Table 1

Selected geometric parameters (\AA , $^\circ$).

| | | | |
|-----------|-----------|-----------|-----------|
| O1–C1 | 1.266 (4) | C1–C2 | 1.411 (4) |
| O2–C36 | 1.355 (4) | C2–C3 | 1.382 (4) |
| N–C3 | 1.339 (4) | C1–C11 | 1.478 (5) |
| N–C31 | 1.414 (4) | C3–C21 | 1.490 (4) |
| C3–N–C31 | 131.4 (3) | C1–C2–C3 | 124.8 (3) |
| O1–C1–C2 | 121.4 (3) | N–C3–C2 | 121.0 (3) |
| O1–C1–C11 | 118.0 (3) | N–C3–C21 | 119.6 (3) |
| C2–C1–C11 | 120.5 (3) | C2–C3–C21 | 119.4 (3) |

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|--------------------------|-------|--------------|--------------|----------------|
| $N-H1N \cdots O1$ | 0.86 | 1.95 | 2.640 (4) | 136 |
| $O2-H2O \cdots O1^i$ | 0.82 | 1.83 | 2.596 (4) | 154 |
| $C15-H15 \cdots O2^{ii}$ | 0.93 | 2.59 | 3.352 (6) | 140 |

Symmetry codes: (i) $-x + 1, -y + 2, -z + 1$; (ii) $-x, -y + 2, -z + 1$.

All H atoms were placed at geometrically idealized positions and were treated as riding atoms, with $C-H = 0.93\text{ \AA}$, $N-H = 0.86\text{ \AA}$ and

O—H = 0.82 Å. $U_{\text{iso}}(\text{H})$ values were set at $1.2U_{\text{eq}}(\text{C,N})$ and $1.5U_{\text{eq}}(\text{O})$.

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*.

The authors thank the Natural Science Foundation of Jiangsu Province (No. 05KJB150151) and Yangzhou University for financial support.

References

- Christoffers, J. (2003). *Chem. Eur. J.* **9**, 4862–4867.
- Christoffers, J., Kreidler, B., Unger, S. & Frey, W. (2003). *Eur. J. Org. Chem.* pp. 2845–2853.
- Doherty, S., Errington, R. J., Housley, N., Ridland, J., Clegg, W. & Elsegood, M. R. J. (1999). *Organometallics*, **18**, 1018–1029.
- Enraf–Nonius. (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
- Gilli, P., Bertolasi, V., Ferretti, V. & Gilli, G. (2000). *J. Am. Chem. Soc.* **122**, 10405–10412.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- Kim, J., Hwang, J. W., Kim, Y., Lee, M. H., Han, Y. & Do, Y. (2001). *J. Organomet. Chem.* **620**, 1–7.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Shi, Y.-C. (2005). *Acta Cryst.* **E61**, o1130–o1132.
- Shi, Y.-C., Sui, C.-X., Song, H.-B. & Jian, P.-M. (2005). *J. Coord. Chem.* **58**, 363–371.
- Shi, Y.-C., Yang, H.-M., Shen, W.-B., Yan, C.-G. & Hu, X.-Y. (2004). *Polyhedron*, **23**, 15–21.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Tietze, L. F., Bergmann, A., Brill, G., Brüggemann, K., Hartief, U. & Voss, E. (1989). *Chem. Ber.* **122**, 83–94.