

3-[2-(Hydroxymethyl)anilino]-1-phenyl-
but-2-en-1-oneYao-Cheng Shi* and Hong-Jian
ChengSchool of Chemistry, Yangzhou University, 130
XiMenWai Street, Yangzhou 225002, People's
Republic of China

Correspondence e-mail: yzssyc@yzcn.net

Key indicators

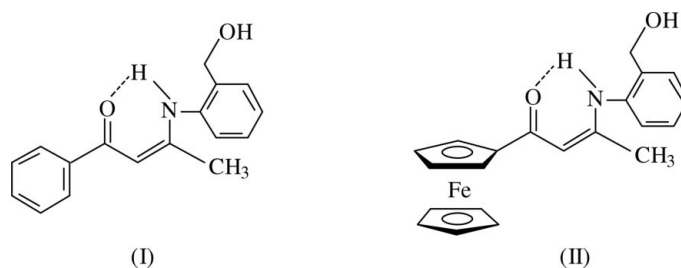
Single-crystal X-ray study
 $T = 295\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.053
 wR factor = 0.190
Data-to-parameter ratio = 15.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_{17}\text{H}_{17}\text{NO}_2$, has been synthesized by the
condensation reaction of benzoylacetone and 2-hydroxy-
methylaniline. The molecules are connected *via* $\text{O}-\text{H}\cdots\text{O}(\text{=C})$
hydrogen bonds, forming centrosymmetric $R_2^2(16)$
dimers.

Received 17 November 2005

Accepted 5 December 2005

Online 10 December 2005

Comment

Enaminones have been studied extensively, not only because
of plentiful reaction chemistry (Christoffers *et al.*, 2003; Tietze
et al., 1989), but also owing to their applications in coordina-
tion chemistry (Kim *et al.*, 2001; Doherty *et al.*, 1999) as well as
chiral auxiliaries in organic synthesis (Christoffers, 2003). As
part of an ongoing investigation of the chemistry of
enaminones, compound (I) has been synthesized by the
condensation of benzoylacetone and 2-hydroxymethylaniline
(Shi *et al.*, 2004, 2005) and its crystal structure has been
determined (Fig. 1). An organometallic analog of (I), here-
after (II), has already been studied (Shi, 2005).The bond lengths and angles of the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$
system in (I) are similar to the corresponding values in (II)
(Table 1). For (I) and (II), the bond lengths in the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$
system indicate electron delocalization (Gilli *et al.*,
2000). The dihedral angles between the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$
system and the planes of the $\text{C}1-\text{C}6$ and $\text{C}11-\text{C}16$ rings in
(I) are $18.94(14)$ and $41.06(12)^\circ$, respectively. The corre-
sponding angles in (II) are $5.4(3)$ and $67.8(3)^\circ$, respectively.
The bonds linking the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$ system and the
aromatic ring in (I) and (II) are typical single bonds ($\text{Csp}^2-\text{Csp}^2$),
suggesting that the $\text{C}1-\text{C}6$ benzene ring in (I) and the
substituted cyclopentadienyl ring in (II) are not involved in
the conjugation of the $\text{O}=\text{C}-\text{C}=\text{C}-\text{N}$ system.As in (II), the strong intramolecular hydrogen bond
between the enamine N atom and the carbonyl O atom in (I)
stabilizes the enaminone. Furthermore, intermolecular $\text{O}-\text{H}\cdots\text{O}=\text{C}$
hydrogen bonds form centrosymmetric $R_2^2(16)$
dimers. Unlike in (II), $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2)
are also observed in (I).

Experimental

An ethanol solution of benzoylacetone and 2-(hydroxymethyl)aniline (1:1) was refluxed for 28 h. After removal of the solvent, the residue was recrystallized from CH_2Cl_2 and diethyl ether (1:1, *v/v*) at 278 K to afford the yellow compound (I) (yield, 52%; m.p., 391.2–392.6 K).

Crystal data

$\text{C}_{17}\text{H}_{17}\text{NO}_2$	$D_x = 1.260 \text{ Mg m}^{-3}$
$M_r = 267.32$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
$a = 8.5030 (17) \text{ \AA}$	$\theta = 9\text{--}14^\circ$
$b = 11.543 (2) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 14.717 (3) \text{ \AA}$	$T = 295 \text{ K}$
$\beta = 102.74 (3)^\circ$	Block, yellow
$V = 1408.9 (5) \text{ \AA}^3$	$0.40 \times 0.30 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.028$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 26.0^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = 0 \rightarrow 10$
$T_{\text{min}} = 0.953$, $T_{\text{max}} = 0.984$	$k = 0 \rightarrow 14$
2957 measured reflections	$l = -18 \rightarrow 17$
2766 independent reflections	3 standard reflections every 200 reflections
1867 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1005P)^2 + 0.3124P]$
$R[F^2 > 2\sigma(F^2)] = 0.053$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.190$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
2766 reflections	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
184 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.023 (6)

Table 1

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

O1–C7	1.255 (3)	C6–C7	1.501 (3)
O2–C17	1.408 (3)	C7–C8	1.409 (3)
N–C9	1.331 (3)	C8–C9	1.383 (3)
N–C11	1.423 (3)	C9–C10	1.498 (3)
C9–N–C11	129.7 (2)	N–C9–C8	120.6 (2)
O1–C7–C6	118.7 (2)	N–C9–C10	120.2 (2)
O1–C7–C8	121.9 (2)	C8–C9–C10	119.2 (2)
C6–C7–C8	119.4 (2)		
C5–C6–C7–C8	17.7 (4)	C9–N–C11–C12	−48.7 (4)
C7–C8–C9–N	3.3 (4)	C15–C16–C17–O2	8.6 (3)

Table 2

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

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
N–H1N \cdots O1	0.86	1.95	2.640 (3)	136
O2–H2O \cdots O1 ⁱ	0.82	1.89	2.701 (2)	172
C12–H12 \cdots O2 ⁱⁱ	0.93	2.45	3.297 (3)	152

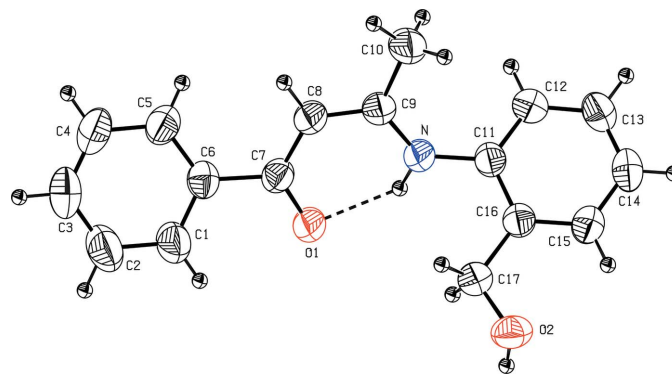
Symmetry codes: (i) $-x + 1, -y + 2, -z$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$.

Figure 1

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

All H atoms were placed at geometrically idealized positions, and treated as riding atoms, with C–H = 0.93 \AA , N–H = 0.86 \AA and O–H = 0.82 \AA , and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ or $1.5U_{\text{eq}}(\text{O})$.

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 (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**, m811–m812.
- 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.