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$R_2^2(14)$ dimers in 3-(hydroxyethyl)amino-1-phenylbut-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.071
wR factor = 0.220
Data-to-parameter ratio = 16.2

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

The title compound, $C_6H_5COCH{=}C(NHCH_2CH_2OH)CH_3$ or $C_{12}H_{15}NO_2$, has been synthesized by the reaction of benzoylacetone and ethanolamine in ethanol. In the crystalline state, molecules are linked by intermolecular hydrogen bonds (O—H···O═C) to form centrosymmetric $R_2^2(14)$ dimers.

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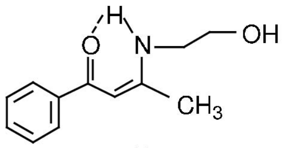
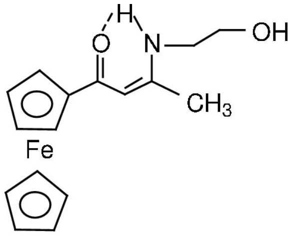
Comment

The title compound, (I), has been synthesized as part of an ongoing investigation of the chemistry of enaminones (Shi, 2005*a*,*b*,c) and its crystal structure has been determined (Fig. 1). An organometallic analog of (I), hereafter (II), has already been studied (Shi *et al.*, 2005). The bond lengths and angles of the O═C—C═C—N system in (I) are similar to the corresponding values in (II) (Table 1). For the molecules of (I) and (II), the bond lengths in the O═C—C═C—N system indicate electron delocalization (Shi, Yang, Shen *et al.*, 2004; Shi, Yang, Song & Liu, 2004; Gilli *et al.*, 2000; Gilchrist, 1997). The dihedral angle between the O═C—C═C—N system and the aromatic ring in (I) is 8.3 (2)°; the value in (II) is 14.8 (2)°. Therefore, the above dihedral angles suggest that the benzene ring in (I) and the substituted cyclopentadienyl ring in (II) are not involved in the conjugation of the O═C—C═C—N system; this is in agreement with the fact that the bonds linking the O═C—C═C—N system and the aromatic ring in (I) and (II) are typical single bonds.



(I)



(II)

As in (II), the strong intramolecular hydrogen bond involving the enamine N and carbonyl O atom in (I) stabilizes the enaminone, whereas intermolecular O—H···O═C hydrogen bonds form centrosymmetric $R_2^2(14)$ dimers (Bernstein *et al.*, 1995; Table 2 and Fig. 2). Interestingly, non-classical C—H···O═C hydrogen bonds are present in the crystalline state of (I), whereas the corresponding intermolecular hydrogen bonds in (II) are not observed.

Experimental

The title compound was synthesized by refluxing a solution of benzoylacetone and ethanolamine (1:1) in ethanol for 4 h. It was recrystallized from CH_2Cl_2–petroleum ether as colorless crystals (m.p. 359–361 K). IR (KBr): 3332 (*br*, *s*, NH, OH), 1602 (*vs*, C=O), 1532 (*vs*, C=C) cm^{-1}.H NMR (600 MHz, $CDCl_3$): δ 11.498 (1H, *s*, NH), 7.838, 7.395 (2H, *m*, 3 H, *m*, C_6H_5), 5.663 (1H, *s*, CH), 3.814 (2H, *t*,^3J = 5.4 Hz, OCH_2), 3.492 (2H, *t*, ^3J = 5.4 Hz, NCH_2), 2.881 (*br*, 1H, OH), 2.032 (3H, *s*, CH_3).

Crystal data

$C_{12}H_{15}NO_2$
M_r = 205.25
Monoclinic, $P2_1/n$
a = 5.6330 (11) Å
b = 25.961 (5) Å
c = 7.9770 (16) Å
β = 102.26 (3)°
V = 1139.9 (4) Å^3
Z = 4
D_x = 1.196 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.08 mm^{-1}
T = 293 K
Block, Colorless
0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min}$ = 0.955, $T_{\max}$ = 0.976
2449 measured reflections
2224 independent reflections
1044 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.050
$\theta_{\max}$ = 26.0°
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 31$
$l = -9 \rightarrow 9$
3 standard reflections every 200 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.220
S = 0.97
2224 reflections
137 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.8P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.32 e Å^{-3}
$\Delta\rho_{\min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.065 (8)

Table 1
Selected geometric parameters (Å, °).

N—C2	1.449 (4)	C3—C4	1.512 (4)
N—C4	1.318 (4)	C4—C5	1.386 (5)
O1—C1	1.426 (4)	C5—C6	1.414 (4)
O2—C6	1.254 (4)	C6—C7	1.496 (5)
C1—C2	1.490 (5)		
C2—N—C4	127.0 (3)	C3—C4—C5	120.0 (3)
O1—C1—C2	109.5 (3)	C4—C5—C6	124.2 (3)
N—C2—C1	109.3 (3)	O2—C6—C5	122.2 (3)
N—C4—C3	118.2 (3)	O2—C6—C7	118.2 (3)
N—C4—C5	121.7 (3)	C5—C6—C7	119.6 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H*n*···O2	0.86	1.98	2.658 (4)	135
O1—H*o*···O2$^{\rm i}$	0.82	1.93	2.747 (4)	179
C3—H3*b*···O2$^{\rm ii}$	0.96	2.54	3.443 (5)	158

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

All H atoms were located in difference maps and subsequently 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. $U_{\rm iso}$(H) values were set at $1.2U_{\rm eq}$(C,N), $1.5U_{\rm eq}$($C_{\rm methyl}$) or $1.5U_{\rm eq}$(O).

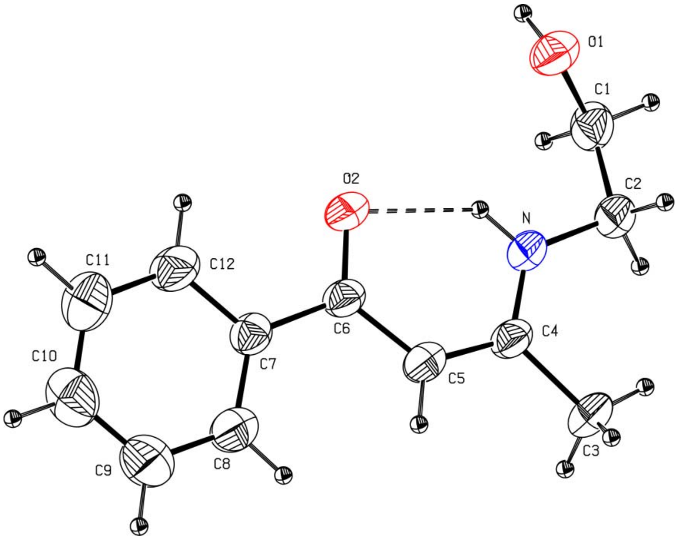


Figure 1
The molecular structure of (I). Displacement ellipsoids for non-H atoms are drawn at the 30% probability level. The intramolecular hydrogen bond is shown as a dashed line.

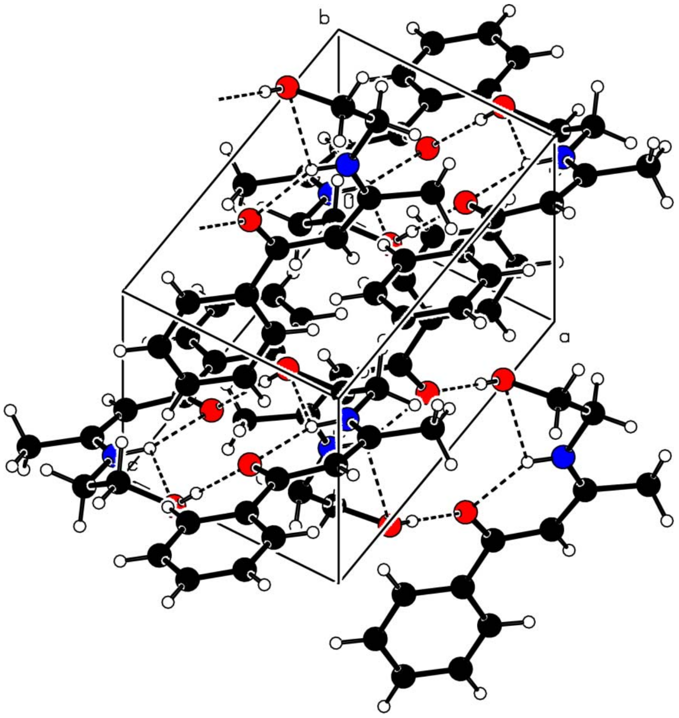


Figure 2
Packing diagram of (I). Dashed lines indicate hydrogen bonds.

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: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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