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Hydrogen-bonded $R_2^2(16)$ dimers in 1-ferrocenyl-3-(2-hydroxymethylphenyl)aminobut-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.056
wR factor = 0.177
Data-to-parameter ratio = 14.5

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

Molecules of the title compound, $[Fe(C_5H_5)(C_{16}H_{16}NO_2)]$, are stabilized by intramolecular hydrogen bonds (N—H···O=C) and are linked by intermolecular hydrogen bonds (O—H···O=C) to form centrosymmetric $R_2^2(16)$ dimers.

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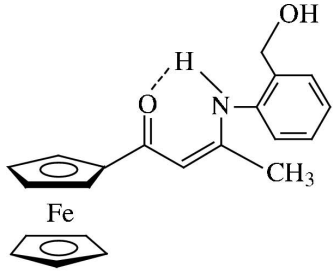
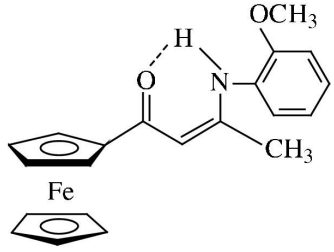
Comment

The title compound, (I), has been synthesized as part of a systematic investigation of the chemistry of enaminones (Shi *et al.*, 2005). Its crystal structure is reported here (Fig. 1). A related compound having the same elemental composition, (II), has already been studied (Shi, Yang, Song & Liu, 2004). It crystallizes with two molecules, (II*a*) and (II*b*), in the asymmetric unit. Although the corresponding bond lengths and angles of the O=C—C=C—N system in (I) are similar to those in (II*a*) and (II*b*), the most striking difference between them lies in the dihedral angles between the O=C—C=C—N plane and the substituted cyclopentadienyl and benzene rings (Table 1; Shi, Yang, Shen *et al.*, 2004; Gilli *et al.*, 2000). Interestingly, the above dihedral angles suggest that the benzene rings are not involved in the conjugation of the O=C—C=C—N system.



(I)



(II)

As in molecules (II*a*) and (II*b*), the enamine N atom and carbonyl O atom in (I) form a strong intramolecular hydrogen bond that stabilizes the enaminone (Table 2). Strong intermolecular O—H···O=C hydrogen bonds are also present in the crystal structure of (I). The paired intermolecular hydrogen bonds lead to centrosymmetric $R_2^2(16)$ dimers (Bernstein *et al.*, 1995) (Fig. 2).

Experimental

The title compound was prepared by refluxing an ethanol solution of ferrocenoylacetone (1.351 g, 5 mmol) and 2-(hydroxymethyl)aniline (0.616 g, 5 mmol) for 12 h (1.313 g, 3.5 mmol, 70% yield; m.p. 416–417 K). Recrystallization from CH_2Cl_2/petroleum ether yielded single crystals suitable for X-ray crystallographic analysis.

Crystal data

$[Fe(C_5H_5)(C_{16}H_{16}NO_2)]$
M_r = 375.24
Monoclinic, $P2_1/n$
a = 15.590 (3) Å
b = 7.5280 (15) Å
c = 15.755 (3) Å
β = 107.85 (3)°
V = 1760.0 (7) Å^3
Z = 4
D_x = 1.416 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9–12°
μ = 0.87 mm^{-1}
T = 293 (2) K
Block, red
0.3 × 0.2 × 0.1 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.801, T_{max} = 0.911
3535 measured reflections
3407 independent reflections
2158 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 26.0°
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 9$
$l = -19 \rightarrow 19$
3 standard reflections
every 200 reflections
frequency: 120 min
intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.177
S = 0.96
3407 reflections
235 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0756P)^2 + 3.6852P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C3	1.444 (6)	C8—C9	1.507 (7)
N1—C8	1.314 (6)	C8—C10	1.372 (7)
O1—C11	1.265 (6)	C10—C11	1.406 (7)
O2—C7	1.399 (7)	C11—C12	1.472 (7)
C3—N1—C8	127.8 (4)	C8—C10—C11	125.2 (5)
O2—C7—C4	114.1 (4)	O1—C11—C10	122.4 (4)
N1—C8—C10	121.9 (5)	O1—C11—C12	118.0 (4)
N1—C8—C9	118.1 (5)	C10—C11—C12	119.6 (4)
C10—C8—C9	120.0 (5)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*n*···O1	0.85 (4)	2.06 (4)	2.684 (6)	130 (4)
O2—H2*o*···O1^{i}	0.85 (4)	1.97 (4)	2.814 (7)	172 (7)

Symmetry code: (i) $1-x, 1-y, 1-z$.

All H atoms bonded to C atoms were placed at geometrically idealized positions and were treated as riding atoms, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2 U_{eq}(C). H atoms bonded to N and O were refined with a distance restraint of N—H = O—H = 0.85 (4) Å and U_{iso}(H) = 1.2 U_{eq}(N,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: *WinGX* (Farrugia, 2005); software used to prepare material for publication: *WinGX*.

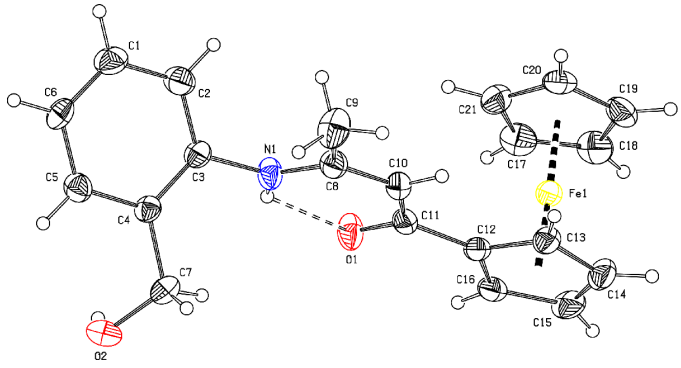


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is indicated by a dashed line.

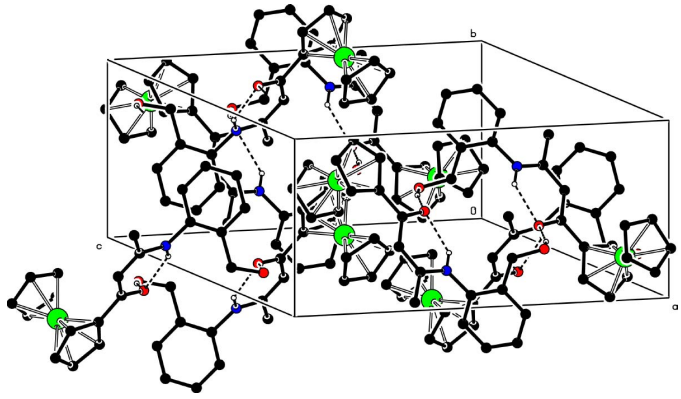


Figure 2
The packing of (I). Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

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