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## Key indicators

Single-crystal X-ray study  
 $T = 295$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å  
 $R$  factor = 0.039  
 $wR$  factor = 0.130  
Data-to-parameter ratio = 12.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(Z)-1-Ferrocenyl-3-hydroxy-3-(2-pyridyl)-prop-2-en-1-one**

The title compound,  $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{13}\text{H}_{10}\text{NO}_2)]$ , has been synthesized by the Claisen condensation reaction of acetylferrocene and ethyl picolinate in the presence of sodium ethoxide. The enone structure is stabilized by a strong  $[\text{OH} \cdots \text{O}=\text{C}]$  intramolecular hydrogen bond.

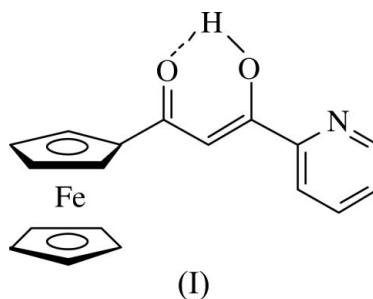
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## Comment

Enaminones are important ligands in coordination chemistry (Doherty *et al.*, 1999; Kim *et al.*, 2001; Li *et al.*, 2004). In continuation of our investigation of the chemistry of ferrocenyl enaminones, we have prepared the title compound, (I), as an intermediate, *via* the Claisen condensation of acetylferrocene and ethyl picolinate (Shi *et al.*, 2004, 2005).



Compound (I) has been shown by  $^1\text{H}$  NMR spectroscopy to exist in solution in the enol and not the keto form. IR spectroscopy shows only the enol form in the solid state; this has been further confirmed in the present study of its crystal structure (Fig. 1).

As also noted in the related compound 1-ferrocenyl-3-hydroxybut-2-en-1-one, (II) (Bell *et al.*, 1992), the  $\text{O}=\text{C}-\text{C}=\text{C}-\text{O}$  fragment of the molecule of (I) is planar. The bond lengths indicate electron delocalization (Gilli *et al.*, 2000). The  $\text{O}=\text{C}-\text{C}=\text{C}-\text{O}$  plane is twisted with respect to the pyridyl

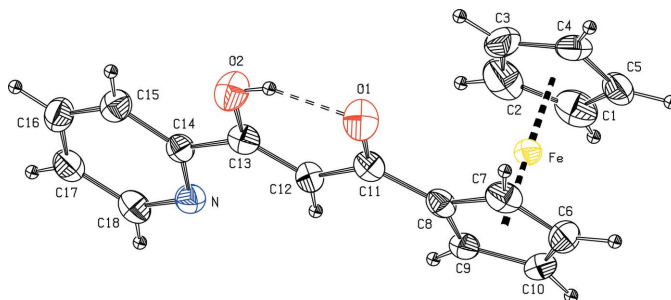
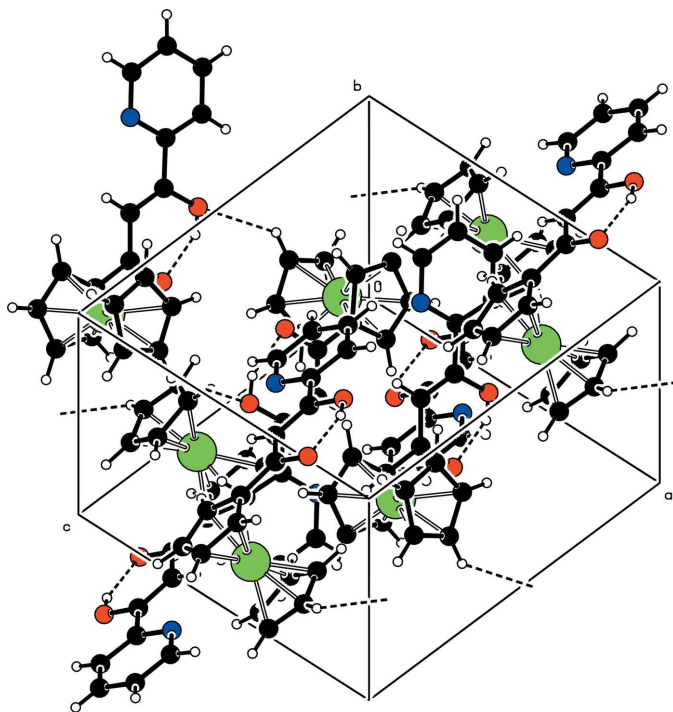


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.



**Figure 2**  
A packing diagram for (I). Dashed lines indicate hydrogen bonds.

and cyclopentadienyl rings by 3.3 (2) and 9.4 (2)°, respectively. The two cyclopentadienyl rings in the ferrocenyl group are parallel [dihedral angle 1.5 (3)°] and adopt a staggered conformation. The C13–C14 bond, which is typical of a single bond ( $Csp^2-Csp^2$ ), suggests that the pyridyl ring is not involved in the conjugation of the  $O=C-C=C-O$  group.

Unlike (II), as well as the intramolecular hydrogen bond [ $O-H \cdots O=C$ ], intermolecular hydrogen bonds [ $C-H \cdots O=C$ ] are also present in the crystal structure of (I) (Fig. 2, Table 2).

## Experimental

The title compound was synthesized by refluxing a tetrahydrofuran solution of acetylferrocene and ethyl picolinate in the presence of sodium ethoxide (1:1:1) for 4 h, as described previously (Shi *et al.*, 2004). Red crystals of (I) were obtained by slow evaporation of a  $CH_2Cl_2$ –petroleum ether (1:2 *v/v*) solution (m.p. 417.6–418.4 K). Spectroscopic analysis: IR (KBr,  $\nu$ ,  $cm^{-1}$ ): 3438.84 (*m*, OH), 3101.12 (*s*, CH), 1593.7 (*vs*, O=C), 1546.10 (*vs*, C=C);  $^1H$  NMR (600 MHz,  $CDCl_3$ ,  $\delta$ , p.p.m.): 16.254 (1H, *s*, OH), 8.731, 8.149, 7.881, 7.440 (4H, 4*m*, pyridyl), 7.098 (1H, *s*, CH), 4.993, 4.599 (2H, 2H, 2*m*,  $C_5H_4$ ), 4.246 (5H, *s*,  $C_5H_5$ ).

### Crystal data

$[Fe(C_5H_5)(C_{13}H_{10}NO_2)]$   
 $M_r = 333.16$   
 Monoclinic,  $P2_1/c$   
 $a = 10.547$  (2) Å  
 $b = 12.804$  (3) Å  
 $c = 11.195$  (2) Å  
 $\beta = 104.08$  (3)°  
 $V = 1466.4$  (5) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.509$  Mg  $m^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 25 reflections  
 $\theta = 10$ –13°  
 $\mu = 1.03$   $mm^{-1}$   
 $T = 295$  K  
 Block, red  
 0.4 × 0.2 × 0.1 mm

### Data collection

Enraf–Nonius CAD4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  $\psi$  scan (North *et al.*, 1968)  
 $T_{min} = 0.773$ ,  $T_{max} = 0.901$   
 2721 measured reflections  
 2577 independent reflections  
 1949 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.045$   
 $\theta_{max} = 25.0^\circ$   
 $h = 0 \rightarrow 12$   
 $k = 0 \rightarrow 15$   
 $l = -13 \rightarrow 12$   
 3 standard reflections every 200 reflections  
 intensity decay: 0.1%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.130$   
 $S = 1.10$   
 2577 reflections  
 200 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 1.5624P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.44$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.32$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97* (Sheldrick, 1997)  
 Extinction coefficient: 0.0118 (16)

**Table 1**  
Selected geometric parameters (Å, °).

O1–C11	1.293 (5)	C11–C12	1.415 (5)
O2–C13	1.303 (5)	C12–C13	1.361 (6)
C8–C11	1.450 (6)	C13–C14	1.484 (6)
O1–C11–C8	118.3 (4)	O2–C13–C12	122.4 (4)
O1–C11–C12	119.5 (4)	O2–C13–C14	114.5 (4)
C8–C11–C12	122.2 (4)	C12–C13–C14	123.2 (4)
C11–C12–C13	121.4 (4)		

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O2–H2O $\cdots$ O1	0.82	1.76	2.495 (5)	148
C4–H4 $\cdots$ O2 <sup>i</sup>	0.98	2.57	3.351 (6)	137

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

All H atoms were placed in geometrically idealized positions and were treated as riding atoms, with C–H distances in the range 0.93–0.98 Å and O–H = 0.82 Å.  $U_{iso}(H)$  values were set at  $1.2U_{eq}(C)$  and  $1.5U_{eq}(C_{methyl}, O)$ .

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*.

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## References

- Bell, W., Crayston, J. A., Glidewell, C., Mazid, M. A. & Hursthouse, M. B. (1992). *J. Organomet. Chem.* **434**, 115–121.  
 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*. Version 5.0. 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.
- Li, X.-F., Dai, K., Ye, W.-P., Pan, L. & Li, Y.-S. (2004). *Organometallics*, **23**, 1223–1230.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst. A* **24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- 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.