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Key indicators

Single-crystal X-ray study
 T = 294 K
 Mean $\sigma(C-C)$ = 0.009 Å
 R factor = 0.065
 wR factor = 0.202
 Data-to-parameter ratio = 17.0

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

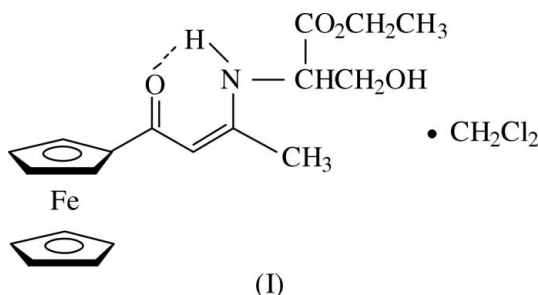
**Ethyl 2-[1-(ferrocenylcarbonyl)prop-1-en-2-yl-
 amino]-3-hydroxypropionate dichloromethane
 solvate**

The title compound, $[Fe(C_5H_5)(C_{14}H_{18}NO_4)] \cdot CH_2Cl_2$, has
 been synthesized by the reaction of ferrocenylacetone with
 DL-serine ethyl ester in ethanol. Electron delocalization is
 observed within the enaminone moiety.

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Comment

As a part of an investigation on the chemistry of enaminones
 (Shi *et al.*, 2004; Shi, 2005), the title compound, (I), has been
 synthesized in the laboratory and its crystal structure is
 reported here.



The molecular structure of (I) is shown in Fig. 1. Within the
 $O=C-C=C-N$ enaminone group, bond lengths and angles
 are comparable to those found in the related compound [3-
 (hydroxyethyl)amino-1-ferrocenylbut-2-en-1-one], (II) (Shi *et al.*,
 2005). The smaller difference of 0.04 (1) Å between the
 $C7-C8$ and $C8-C9$ bond distances (Table 1) clearly indicates
 electron delocalization within the enaminone group (Gilli *et al.*,
 2000). The $C9-C10$ bond distance, close to the typical
 Csp^2-Csp^2 single-bond distance of 1.48 Å, suggests that the
 $C10$ ring is not involved in the electron delocalization system
 of the enaminone group. The dihedral angle of 11.8 (3)°
 between the enaminone plane and the $C10$ cyclopentadienyl
 ring is slightly smaller than that [15.6 (2)°] in (II).

Carbonyl atom O4 is intramolecularly and intermolecularly
 hydrogen bonded with the enamine and hydroxy groups
 (Table 2).

Experimental

Ferrocenylacetone was prepared according to a literature method
 (Shi *et al.*, 2004). An ethanol solution of ferrocenylacetone and DL-
 serine ethyl ester in a 1:1 ratio was refluxed for 24 h. After the
 removal of solvent, the resulting residue was chromatographed on a
 silica gel column using CH_2Cl_2 /diethyl ether (2:1, v/v) as eluant. The
 red band was collected and further recrystallized from CH_2Cl_2 /
 petroleum ether to afford the enaminone (32% yield, m.p. 397–

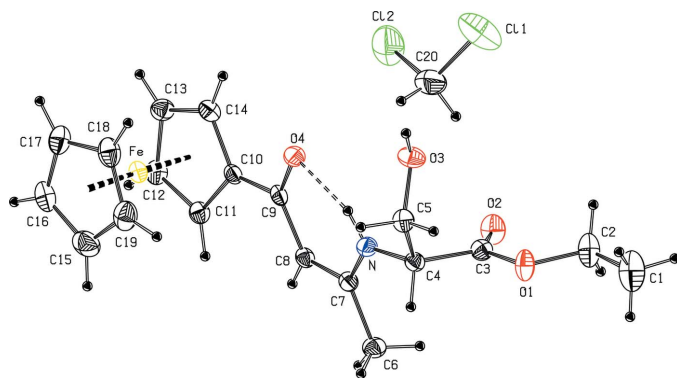


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). The thin broken line indicates an intramolecular hydrogen bond.

398 K). Analysis calculated for $C_{19}H_{23}FeNO_4$: C 59.24, H 6.02, N 3.64%; found: C 60.12, H 6.31, N 3.27%. IR (KBr, cm^{-1}): 3355 (*br*, *w*, OH), 3098 (*w*, NH), 1741 (*m*, O=COEt), 1600 (*vs*, O=C), 1525 (*s*, C=C). UV (λ_{max} , in DMF, nm): 257.50 (B band, 0.91×10^4), 335.50 (K band, 1.63×10^4), 445.50 (CT band, 0.079×10^4). 1H NMR (600 MHz, $CDCl_3$, p.p.m.): δ 11.14 (1H, *s*, NH), 5.37 (1H, *s*, CH), 4.73, 4.38 (4H, *2s*, C_5H_4), 4.29–4.31 (1H, *m*, CHN), 4.23–4.27 (2H, *q*, OCH_2Me), 4.16 (5H, *s*, C_5H_5), 3.98–3.99 (2H, *d*, CH_2), 2.03 (3H, *s*, CH_3), 1.29–1.31 (3H, *t*, CH_3).

Crystal data

$[Fe(C_5H_5)(C_{14}H_{18}NO_4)] \cdot CH_2Cl_2$
 $M_r = 470.16$
Monoclinic, $P2_1/c$
 $a = 10.836$ (3) Å
 $b = 22.2556$ (15) Å
 $c = 10.023$ (2) Å
 $\beta = 114.11$ (3)°
 $V = 2206.3$ (9) Å³
 $Z = 4$

$D_x = 1.416$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
 $\theta = 10$ – 13°
 $\mu = 0.95$ mm⁻¹
 $T = 293$ K
Block, red
 $0.40 \times 0.30 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
Absorption correction: empirical (using intensity measurements)
 ψ scan (North *et al.*, 1968)
 $T_{min} = 0.702$, $T_{max} = 0.821$
4580 measured reflections
4325 independent reflections

2542 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.041$
 $\theta_{max} = 26.0^\circ$
 $h = -13 \rightarrow 12$
 $k = -27 \rightarrow 0$
 $l = 0 \rightarrow 12$
3 standard reflections every 200 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.065$
 $wR(F^2) = 0.202$
 $S = 1.06$
4325 reflections
255 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0984P)^2 + 1.6775P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.60$ e Å⁻³
 $\Delta\rho_{min} = -0.75$ e Å⁻³

Table 1
Selected geometric parameters (Å, °).

N—C4	1.434 (6)	O4—C9	1.266 (6)
N—C7	1.337 (6)	C1—C2	1.479 (8)
O1—C2	1.425 (6)	C6—C7	1.497 (7)
O1—C3	1.316 (7)	C7—C8	1.381 (7)
O2—C3	1.191 (7)	C8—C9	1.421 (7)
O3—C5	1.404 (7)	C9—C10	1.469 (7)
O1—C3—O2	124.8 (5)	C6—C7—C8	120.1 (5)
O1—C3—C4	110.7 (5)	C7—C8—C9	123.3 (5)
O2—C3—C4	124.6 (5)	O4—C9—C8	122.8 (5)
N—C7—C6	119.2 (5)	O4—C9—C10	118.1 (5)
N—C7—C8	120.6 (5)	C8—C9—C10	119.1 (4)

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O3—H3 \cdots O4 ⁱ	0.82	1.92	2.681 (6)	154
N—H1N \cdots O4	0.86	1.92	2.617 (6)	137
C20—H20A \cdots O4	0.97	2.55	3.270 (10)	131

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

Methyl H atoms were placed at calculated positions ($C-H = 0.96$ Å) to fit the electron density, with $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were placed at calculated positions ($C-H = 0.93$ – 0.98 Å, $N-H = 0.86$ Å and $O-H = 0.82$ Å) and were treated as riding atoms during the refinement, with $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$.

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) and *OSCAILX* (McArdle, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997) and *OSCAILX*; molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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