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(*S*)-3-[(1-Ethoxycarbonyl-2-phenylethyl)amino]-1-ferrocenylbut-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.012 Å
R factor = 0.044
wR factor = 0.121
Data-to-parameter ratio = 8.8

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

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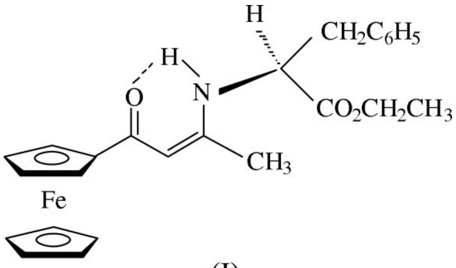
The title compound {systematic name: (*S*)-ethyl 2-[1-(1-ferrocenylcarbonyl)prop-1-en-2-ylamino]-3-phenylpropionate}, [Fe(C_5H_5)($C_{20}H_{22}NO_3$)], has been synthesized by the reaction of ferrocenoylacetone with ethyl L-phenylalanine in ethanol. The two nearly parallel cyclopentadienyl rings in the ferrocenyl moiety adopt an eclipsed conformation.

Comment

Enaminones are important ligands in coordination chemistry (Li *et al.*, 2004; Kim *et al.*, 2001; Doherty *et al.*, 1999) and important chiral auxiliaries in organic synthesis (Christoffers, 2003). In continuation of our investigation of the chemistry of ferrocenyl enaminones (Shi, Sui & Cheng, 2005; Shi, Sui *et al.*, 2005), the title compound, (I), has been prepared by the reaction of ferrocenoylacetone and L-phenylalanine ethyl ester in an ethanol solution.



(I)

The molecular structure of (I) is shown in Fig. 1. The bond distances (Table 1) within the O═C—C═C—N system show electron delocalization (Gilli *et al.*, 2000). The dihedral angle

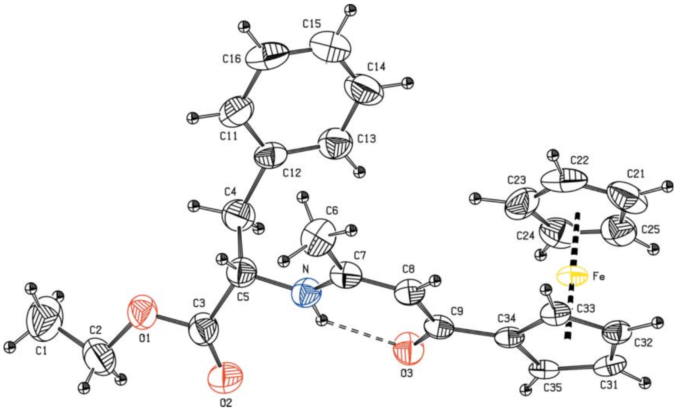


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). The double dashed line indicates the intramolecular hydrogen bond.

between the O═C—C═C—N plane and the substituted cyclopentadienyl ring is 7.7 (4)°. The C9—C34 bond distance suggests a single bond. The two nearly parallel cyclopentadienyl rings in the ferrocenyl moiety adopt an eclipsed conformation.

Classical N—H···O intramolecular hydrogen bonds and weak intermolecular C—H···O hydrogen bonds are observed in the crystal structure of (I) (Table 2).

Experimental

The title compound was synthesized by refluxing a solution of ferrocenoylacetone and L-phenylalanine ethyl ester in a 1:1 molar ratio for 4 h in an ethanol solution. After removal of the solvent, the resulting residue was chromatographed on a silica-gel column using CH_2Cl_2–ethanol (20:1, *v*/*v*) as eluent. The red band was collected and further recrystallized from CH_2Cl_2–petroleum ether (1:2 *v*/*v*) to afford the title compound (32% yield, m.p. 357–358 K). Analysis, calculated for $C_{25}H_{27}FeNO_3$: C 67.43, H 6.11, N 3.15%; found: C 67.45, H 6.35, N 3.06%. Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3117 (*br*, *w*, NH), 1739 (*s*, O═COEt), 1601 (*vs*, O═C), 1567 (*m*, C═C); UV (λ_{max}, nm, in DMF): 336.50 (K-band, 1.35×10^4), 455.00 (CT-band, 0.071×10^4); ^1H NMR (600 MHz, $CDCl_3$, δ, p.p.m.): 11.23 (1H, *s*, NH), 7.23–7.31 (5H, *m*, C_6H_5), 5.21 (1H, *s*, CH), 4.76, 4.71, 4.37 (4H, *s*, C_5H_4), 4.31 (1H, *m*, CHN), 4.19–4.23 (2H, *q*, ^3J = 5.4 Hz, OCH_2), 4.16 (5H, *s*, C_5H_5), 3.21–3.24, 3.00–3.04 (2H, 2*q*, CH_2Ph), 1.68 (3H, *s*, CH_3), 1.24–1.27 (3H, *t*, ^3J = 5.4 Hz, CH_3).

Crystal data

$[Fe(C_5H_5)(C_{20}H_{22}NO_3)]$
$M_r = 445.33$
Monoclinic, $P2_1$
$a = 10.029$ (2) Å
$b = 7.4860$ (15) Å
$c = 15.608$ (3) Å
$\beta = 100.26$ (3)°
$V = 1153.1$ (4) Å^3
$Z = 2$

$D_x = 1.283$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
$\mu = 0.68$ mm^{-1}
$T = 293$ 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)
$T_{min} = 0.827$, $T_{max} = 0.923$
2543 measured reflections
2407 independent reflections
1770 reflections with $I > 2\sigma(I)$

$R_{int} = 0.026$
$\theta_{max} = 26.0°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 8$
$l = -18 \rightarrow 18$
3 standard reflections every 200 reflections intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.121$
$S = 1.04$
2407 reflections
273 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.3415P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å).

O1—C2	1.438 (9)	N—C7	1.340 (9)
O1—C3	1.320 (9)	C7—C8	1.371 (10)
O2—C3	1.180 (10)	C8—C9	1.433 (9)
O3—C9	1.245 (8)	C9—C34	1.484 (9)
N—C5	1.435 (9)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H1N···O3	0.86	1.97	2.655 (8)	135
C2—H2*B*···O3^i	0.97	2.58	3.472 (14)	152

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

The absolute configuration of (I) was assigned by reference to an unchanging chiral centre for L-phenylalanine ethyl ester in the synthetic procedure. Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å and torsion angles refined from electron density, with $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were placed in geometrically idealized positions, with C—H = 0.93–0.98 Å and N—H = 0.86 Å, and refined in riding mode, with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

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: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003) and *OSCAIL-X* (McArdle, 1995).

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References

Christoffers, J. (2003). *Chem. Eur. J.* **9**, 4862–4867.
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.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
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.
McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
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. & Cheng, H.-J. (2005). *Acta Cryst.* E**61**, m1563–m1565.
Shi, Y.-C., Sui, C.-X., Song, H.-B. & Jian, P.-M. (2005). *J. Coord. Chem.* **58**, 363–371.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Acta Cryst. (2005). E**61**, m1878–m1879 Shi *et al.* • $[Fe(C_5H_5)(C_{20}H_{22}NO_3)]$ **m1879**