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Key indicators

Single-crystal X-ray study
 T = 295 K
 Mean $\sigma(C-C)$ = 0.008 Å
 R factor = 0.060
 wR factor = 0.184
 Data-to-parameter ratio = 15.8

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

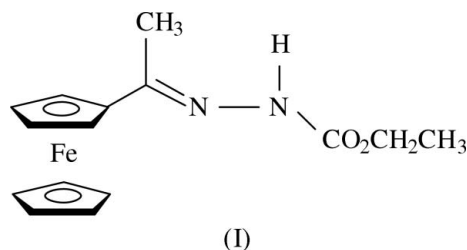
Ethyl 3-(1-ferrocenylethylidene)carbazate

The title compound, $[Fe(C_5H_5)(C_{10}H_{13}N_2O_2)]$, has been synthesized by the reaction of acetylferrocene and hydrazine ethyl carbazate. Molecules of the title compound form centrosymmetric $R_2^2(8)$ dimers *via* intermolecular N—H···O=C hydrogen bonds.

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Comment

The title compound, (I), has been synthesized as part of our ongoing investigation of the chemistry of ferrocenyl derivatives (Shi *et al.*, 2004; Shi, 2004). Although dithiocarbazate derivatives have been widely studied, the corresponding ferrocenyl carbazates have barely been explored. In order to exploit the chemistry of ferrocenyl carbazates, the crystal structure of (I) has been determined (Fig. 1).



Compound (I) crystallizes in the keto form and exists as the conformer in which the N1—N2 bond adopts a *trans* geometry with respect to C=O, while the ethyl group adopts a *cis* geometry. The ferrocenyl and ethyl groups are *cis* to each other across the N1—N2 and C=O bonds. Moreover, both cyclopentadienyl rings in the ferrocenyl group are in the eclipsed conformation. The bond lengths and angles of the C=N—N(H)—C group are comparable with those of two

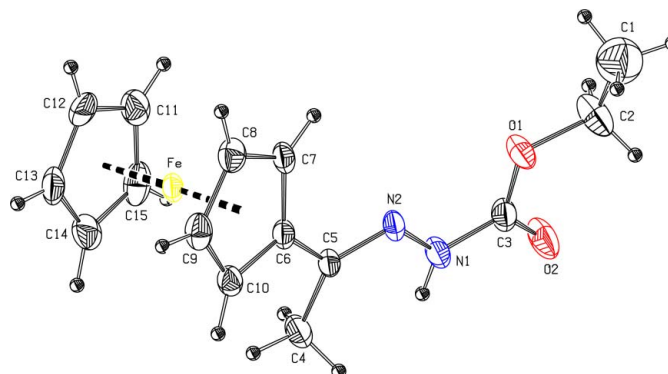


Figure 1
 The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

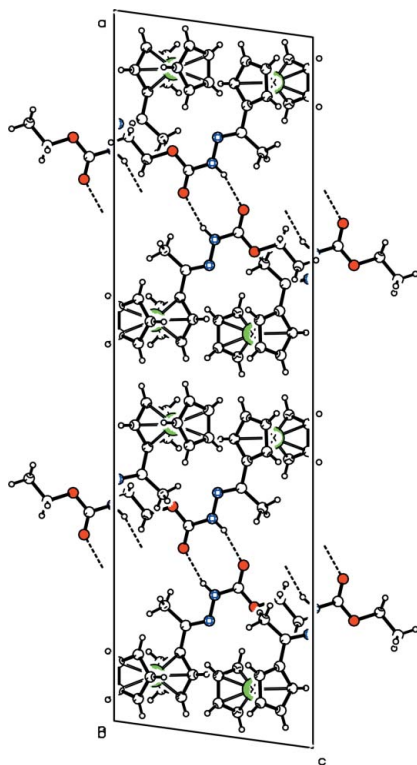


Figure 2

A projection, along the *b* axis, of part of the packing diagram for (I). Dashed lines indicate hydrogen bonds.

dithiocarbamate compounds, Ar–C(CH₃)=N–N(H)–C(SCH₂Ph)=S [Ar = 2-furyl, (II) (Khoo *et al.*, 2005); Ar = 2-thiophenyl, (III) (Chan *et al.*, 2003)] (Table 1). The dihedral angles between the C=N–N(H) plane and the aromatic ring and the ester group are 26.2 (3) and 5.1 (7)°, respectively, whereas the corresponding values are 2.90 (16) and 1.8 (2)° for (II), and 5.39 (13) and 11.37 (16)° for (III).

As in (II) and (III), paired intermolecular N–H···O=C hydrogen bonds lead to the formation of centrosymmetric R₂²(8) dimers (Bernstein *et al.*, 1995; Table 2; Fig. 2).

Experimental

The title compound was synthesized by refluxing a solution of acetylferrocene and hydrazine ethyl carbazate (1:1) in ethanol for 4 h (yield, 82%). Orange single crystals of (I), suitable for X-ray analysis, were obtained by slow evaporation of a CH₂Cl₂–petroleum ether (1:2 *v/v*) solution (m.p. 452.6–454.1 K). IR (KBr, *ν*, cm^{−1}): 3448.35 (*br*, *m*, NH), 1668 (*s*, C=O), 1599 (*vs*, C=N).

Crystal data

[Fe(C₅H₅)(C₁₀H₁₃N₂O₂)]
M_r = 314.16
 Monoclinic, C2/c
a = 36.770 (7) Å
b = 7.6040 (15) Å
c = 10.366 (2) Å
 β = 97.82 (3)°
V = 2871.4 (10) Å³
Z = 8

D_x = 1.454 Mg m^{−3}
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 10–13°
 μ = 1.05 mm^{−1}
T = 295 K
 Block, orange
 0.40 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 T_{\min} = 0.747, T_{\max} = 0.803
 2848 measured reflections
 2804 independent reflections
 2178 reflections with $I > 2\sigma(I)$

R_{int} = 0.039
 θ_{max} = 26.0°
 $h = 0 \rightarrow 45$
 $k = 0 \rightarrow 9$
 $l = -12 \rightarrow 12$
 3 standard reflections
 every 200 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.060
 $wR(F^2)$ = 0.184
 S = 1.07
 2804 reflections
 177 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.098P)^2 + 11.3526P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 1.46 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.76 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1–C2	1.453 (6)	N2–C5	1.290 (6)
O1–C3	1.313 (6)	C1–C2	1.509 (8)
O2–C3	1.216 (6)	C4–C5	1.503 (7)
N1–C3	1.353 (6)	C5–C6	1.461 (6)
O1–C2–C1	107.9 (6)	N2–C5–C4	125.4 (4)
O1–C3–O2	124.2 (5)	N2–C5–C6	115.4 (4)
O1–C3–N1	114.3 (4)	C4–C5–C6	119.2 (4)
O2–C3–N1	121.6 (5)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1N···O2 ⁱ	0.86	2.09	2.927 (6)	164

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

All H atoms were placed in geometrically idealized positions and subsequently treated as riding atoms, with C–H = 0.93–0.98 Å and N–H = 0.86 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ and $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The highest peak is located 1.12 Å from atom H2B.

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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