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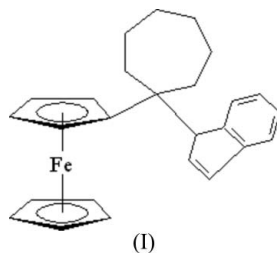
Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.036
 wR factor = 0.080
Data-to-parameter ratio = 15.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1-Ferrocenyl-1-(1*H*-indenyl)cycloheptane

In the title compound, $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{21}\text{H}_{23})]$, the cyclopentadienyl (Cp) rings deviate slightly from an eclipsed geometry. The cycloheptane ring adopts a chair conformation. The unsubstituted and substituted Cp rings form dihedral angles of 55.62 (12) and 54.13 (10) $^\circ$, respectively, with the indene ring system.

Comment

Because of their catalytic properties, a number of ferrocene-substituted indenyl derivatives, *e.g.* 2-(3-indenyl)-2-(ferrocenyl)propane (Gaede, 2000), 1-(ferrocenyl)indene and 2-(ferrocenyl)indene (Plenio, 1992), have been structurally characterized. We present here the synthesis and crystal structure of the title compound, (I).



A view of (I), with the atomic numbering scheme, is shown in Fig. 1; selected bond lengths and angles are given in Table 1. The $\text{Fe1}\cdots\text{Cg1}$ and $\text{Fe1}\cdots\text{Cg2}$ distances are 1.666 (2) and 1.661 (2) Å, respectively, and the $\text{Cg1}-\text{Fe1}-\text{Cg2}$ angle is 176.86 (8) $^\circ$, where Cg1 and Cg2 are the centroids of the unsubstituted and substituted Cp rings, respectively. The Cp rings deviate slightly from an eclipsed geometry. The indene ring system is planar, with an r.m.s. deviation of 0.025 Å. The unsubstituted and substituted Cp rings form dihedral angles of 55.62 (12) and 54.13 (10) $^\circ$, respectively, with the indene plane. The cycloheptane ring adopts a chair conformation. The crystal packing is stabilized by weak $\text{C}-\text{H}\cdots\pi$ interactions (Table 2).

Experimental

A solution of indene (1.39 ml, 12 mmol) in tetrahydrofuran (THF, 100 ml) was reacted with *n*-butyllithium (12 mmol) at 273 K for 2 h and then stirred at room temperature for 4 h. A solution of 6,6-hexamethylenefulvene (1.92 g, 12 mmol) in THF (20 ml) was added dropwise to the above reaction system at 273 K with stirring. When the addition was completed, the solution was warmed to room temperature and stirring was continued overnight. Cyclopentadienyllithium (12 mmol) in THF (20 ml) was added to this reaction mixture. $\text{FeCl}_2\cdot 1.44\text{THF}$ (2.78 g, 12 mmol) was then added to the above mixture and the resulting mixture stirred overnight. The

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solvent was removed in a vacuum. The residue was run through a short column of Al_2O_3 with CH_2Cl_2 , yielding compound (I), which was collected and purified by chromatography on alumina, to give crystals of (I) (1.23 g, yield 25.86%). Analysis calculated for $\text{C}_{26}\text{H}_{28}\text{Fe}$: C 78.79, H 7.12%; found: C 78.50, H 6.99%.

Crystal data

$[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{21}\text{H}_{23})]$
 $M_r = 396.33$
 Monoclinic, C_c
 $a = 6.2777$ (16) Å
 $b = 34.562$ (9) Å
 $c = 9.321$ (2) Å
 $\beta = 90.443$ (4)°
 $V = 2022.3$ (9) Å³
 $Z = 4$

$D_x = 1.302$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2325 reflections
 $\theta = 2.5\text{--}24.8^\circ$
 $\mu = 0.75$ mm⁻¹
 $T = 294$ (2) K
 Block, red
 $0.26 \times 0.24 \times 0.22$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.736$, $T_{\max} = 0.847$
 5671 measured reflections

3703 independent reflections
 3102 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\text{max}} = 26.4^\circ$
 $h = -7 \rightarrow 7$
 $k = -42 \rightarrow 20$
 $l = -10 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.080$
 $S = 1.00$
 3703 reflections
 244 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.20$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³
 Absolute structure: Flack (1983), with 1633 Friedel pairs
 Flack parameter: 0.01 (2)

Table 1

Selected bond lengths (Å).

Fe1—C1	2.042 (3)	Fe1—C6	2.048 (3)
Fe1—C2	2.029 (3)	Fe1—C7	2.033 (3)
Fe1—C3	2.048 (4)	Fe1—C8	2.034 (3)
Fe1—C4	2.063 (3)	Fe1—C9	2.053 (3)
Fe1—C5	2.051 (3)	Fe1—C10	2.095 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
$\text{C8—H8}\cdots\text{Cg1}^i$	0.93	2.76	3.689 (4)	173
$\text{C17—H17B}\cdots\text{Cg3}^{ii}$	0.97	2.67	2.987 (3)	100
$\text{C20—H20}\cdots\text{Cg4}^{iii}$	0.93	2.87	3.618 (5)	138
$\text{C25—H25}\cdots\text{Cg2}^{ii}$	0.93	2.80	3.540 (4)	137

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) x, y, z ; (iii) $x, -y, z - \frac{1}{2}$. Cg1, Cg2, Cg3 and Cg4 denote the centroids of rings C1–C5, C6–C10, C18–C21/C26 and C21–C26, respectively.

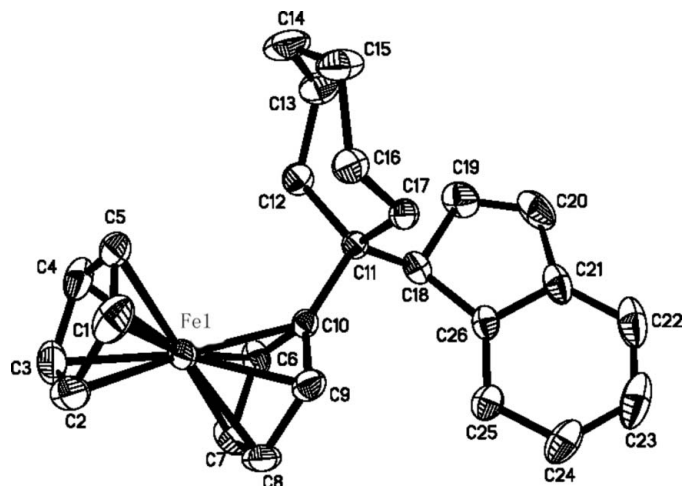


Figure 1

The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

H atoms were placed in calculated positions, with C–H = 0.93 or 0.97 Å, and included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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