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Key indicators

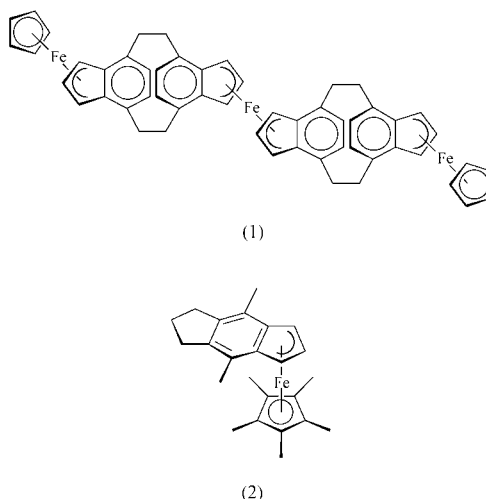
Single-crystal X-ray study
T = 178 K
Mean $\sigma(\text{C}-\text{C})$ = 0.004 Å
R factor = 0.038
wR factor = 0.103
Data-to-parameter ratio = 14.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>. $(\eta^5\text{-Pentamethylcyclopentadienyl})(\eta^5\text{-5,6,7-trihydro-4,8-dimethyl-}s\text{-indacenyl})\text{iron(II)}$ In the title compound, $[\text{Fe}(\text{C}_{10}\text{H}_{15})(\text{C}_{14}\text{H}_{15})]$, the coordinating
rings make an angle of $3.4(2)^\circ$ with each other and are rotated
away from the ideal eclipsed conformation by *ca* 13° .

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Comment

In our continuing studies of the preparation and uses of novel
multiple-metal metallocenes containing the [2.2]paracyclo-
phane system as the bridging unit, such as the tris-iron
complex (1) (Hopf & Dannheim, 1988; *cf.* Hopf *et al.*, 1986),
we needed the title complex, (2), incorporating an *s*-indacene
unit as a 'half cyclophane', as a reference compound. The
isostructural ruthenium analogue is presented in the following
paper (Jones *et al.*, 2002).

The molecule of the title compound is shown in Fig. 1. The distances from the Fe atom to the centroids, *Cg*, of the coordinating ring planes, which are essentially parallel [interplanar angle $3.4(2)^\circ$], are 1.674 Å to the C1–C8A ring and 1.643 Å to the C11–C15 ring, with an angle at Fe of 179.5° . The coordination of the indacenyl ring shows a very slight distortion towards η^3 , with Fe–C3A and Fe–C8A being the longest distances. The rings are rotated appreciably away from the ideal eclipsed conformation, with torsion angles such as C12–Cg1–Cg2–C3 = -13° .

Experimental

The title compound was prepared in 80% yield by first metallating 1,5,6,7-tetrahydro-4,8-dimethyl-*s*-indacene with methyl lithium in anhydrous tetrahydrofuran, and then quenching the anions thus formed with equivalent amounts of the complex $[(\text{C}_5\text{Me}_5)\text{Fe}(\text{CH}_3\text{CN})_3]\text{PF}_6$. It was characterized by spectroscopic and analytical data (Hartig,

1991), and by the crystal structure described here. Single crystals were obtained by slow cooling of a warm saturated solution in hexane.

Crystal data

[Fe(C ₁₀ H ₁₅)(C ₁₄ H ₁₅)]	Z = 2
<i>M_r</i> = 374.33	<i>D_x</i> = 1.307 Mg m ⁻³
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> α radiation
<i>a</i> = 8.6838 (15) Å	Cell parameters from 50 reflections
<i>b</i> = 9.713 (2) Å	θ = 10–11.5°
<i>c</i> = 11.566 (3) Å	μ = 0.80 mm ⁻¹
α = 80.62 (2)°	<i>T</i> = 178 (2) K
β = 81.09 (2)°	Tablet, red
γ = 88.82 (2)°	0.40 × 0.40 × 0.15 mm
<i>V</i> = 950.9 (4) Å ³	

Data collection

Nicolet R3 diffractometer	θ_{\max} = 25.0°
ω scans	<i>h</i> = -10 → 10
Absorption correction: none	<i>k</i> = 0 → 11
3561 measured reflections	<i>l</i> = -13 → 13
3346 independent reflections	3 standard reflections every 147 reflections
2889 reflections with <i>I</i> > 2 σ (<i>I</i>)	intensity decay: none
<i>R</i> _{int} = 0.017	

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 0.467P]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.103$	$(\Delta/\sigma)_{\max} = 0.004$
<i>S</i> = 1.08	$\Delta\rho_{\max} = 0.60 \text{ e \AA}^{-3}$
3346 reflections	$\Delta\rho_{\min} = -0.63 \text{ e \AA}^{-3}$
233 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å).

Fe—C12	2.036 (2)	Fe—C14	2.051 (2)
Fe—C13	2.037 (2)	Fe—C15	2.053 (2)
Fe—C3	2.045 (2)	Fe—C1	2.054 (2)
Fe—C2	2.049 (2)	Fe—C3A	2.096 (2)
Fe—C11	2.049 (2)	Fe—C8A	2.102 (2)

The starting coordinates for refinement were taken from the isostructural Ru analogue (following paper; Jones *et al.*, 2002). Methyl H atoms were identified in difference syntheses, idealized and then refined, using rigid methyl groups allowed to rotate but not tip. Other H atoms were included, using a riding model, with fixed C—H bond lengths (aromatic 0.95, methyl 0.98 and methylene 0.99 Å); *U*_{iso}(H) values were fixed at 1.2*U*_{eq} of the parent atom. The methyl group at C20 converged slowly.

Data collection: *P3* (Nicolet, 1987); cell refinement: *P3*; data reduction: *XDISK* (Nicolet, 1987); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

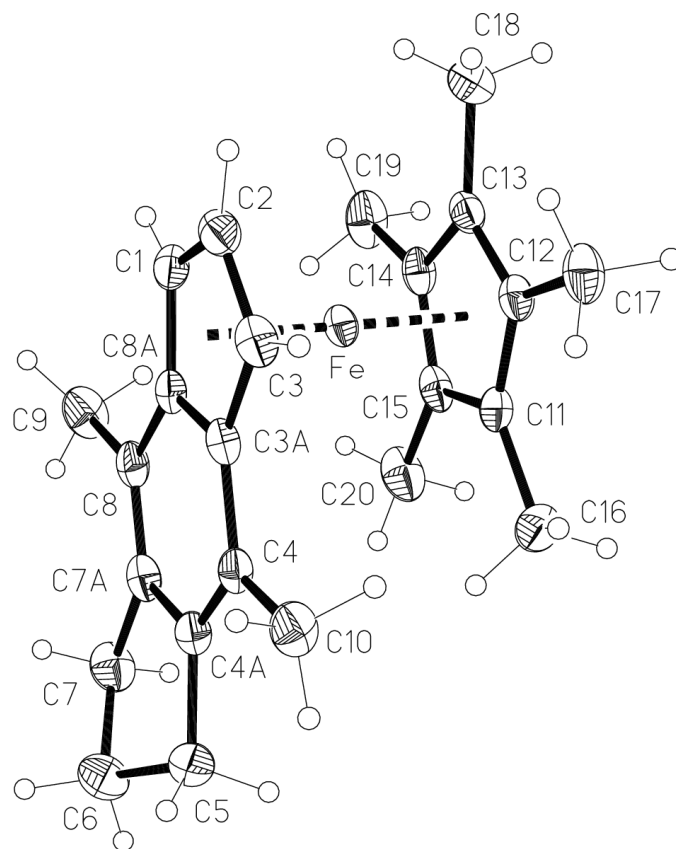


Figure 1

The molecule of compound (2) in the crystal. Ellipsoids represent 50% probability levels. H-atom radii are arbitrary.

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