

p-Ferrocenylaniline

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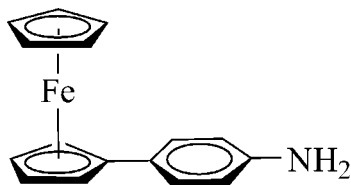
Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.044; wR factor = 0.095; data-to-parameter ratio = 16.5.

In *p*-ferrocenylaniline, $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{11}\text{H}_{10}\text{N})]$, an easily synthesized alternative for ferrocenylamine, the orientation of the amine H atoms allows for conjugation of the nitrogen lone pair with the π electrons of the benzene and cyclopentadienyl rings. No hydrogen-bonding or π - π stacking interactions are present in the solid state.

Related literature

For the synthesis of *p*-ferrocenylaniline, see: Hu *et al.* (2001*a,b*). For related structures, see: Perrine *et al.* (2005); Shafir *et al.* (2000); Rogers *et al.* (1988); Sunkel *et al.* (2000); Takaki *et al.* (1959); Roberts *et al.* (1988); Gallagher *et al.* (1997); Zeller *et al.* (2004). For applications of ferrocene-containing dimers in electrocatalysis, see: Togni & Hayashi (1995); Sawamura & Ito (1992); Nicolosi *et al.* (1994); in ferromagnetism, see: Miller *et al.* (1988); in chemical and biochemical sensing, see: Navarro *et al.* (2005); Brown *et al.* (2005); Okochi *et al.* (2005); Hickman *et al.* (1991); and in self-assembled monolayer chemistry, see: Chidsey *et al.* (1990); Creager & Rowe (1997).

For related literature, see: Herbstein (2000); Rogers *et al.* (1988).



Experimental

Crystal data

$[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{11}\text{H}_{10}\text{N})]$

$M_r = 277.14$

Orthorhombic, *Pbca*

$a = 9.6762$ (7) Å

$b = 8.0284$ (6) Å

$c = 31.515$ (2) Å

$V = 2448.2$ (3) Å³

$Z = 8$

Mo $K\alpha$ radiation

$\mu = 1.21$ mm⁻¹

$T = 100$ (2) K

$0.41 \times 0.19 \times 0.03$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Absorption correction: multi-scan

(*SADABS* in *SAINT-Plus*;

Bruker, 2003)

$T_{\min} = 0.712$, $T_{\max} = 0.960$

22095 measured reflections

2821 independent reflections

2679 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.095$

$S = 1.31$

2821 reflections

171 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.57$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.34$ e Å⁻³

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2417).

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supplementary materials

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p-Ferrocenylaniline

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Comment

Ferrocene-containing dimers have received considerable interest over the years due to their potential applications in electrocatalysis (Togni & Hayashi, 1995; Sawamura & Ito, 1992; Nicolosi *et al.*, 1994), ferromagnetism (Miller *et al.*, 1996), chemical and biochemical sensing (Navarro *et al.*, 2005; Brown *et al.*, 2005; Okochi *et al.*, 2005; Hickman *et al.*, 1991) and self-assembled monolayer chemistry (Chidsey *et al.*, 1990; Creager & Rowe, 1997). They are also of growing interest due to their unique structural, electrochemical and electronic structural properties. The production of dimeric ferrocenylamides has been hampered by the tedious synthesis of aminoferrocene, as it is difficult to produce in high enough yields to be useful in multi-step syntheses. In addition, the production of 1,1'-diaminoferrocene as the major product of most reaction pathways is a serious drawback. Although this compound can be purchased commercially, it is extremely expensive. Its phenyl derivative, the title compound *p*-ferrocenylaniline (I), on the other hand, can be easily produced in a two step synthesis from ferrocene by reaction with diazotated *p*-nitroaniline (Hu *et al.*, 2001*a*), followed by the reduction of the resulting *p*-nitrophenylferrocene with Sn/HCl to produce the title compound *p*-ferrocenylaniline (Hu *et al.*, 2001*b*). This paper presents the single-crystal structure of the title compound.

p-Ferrocenylaniline crystallizes in the orthorhombic space group *Pbca* with 8 molecules per unit cell (Figure 1). The conformation of the two Cp rings towards each other is close to eclipsed, and the substituted Cp and the phenyl ring are basically coplanar with a dihedral angle between the planes defined by C6 to C10 and C11 to C16 of 8.7 (2)°, a value not too different to that found for *p*-nitrophenylferrocene (13.5 (2)°, Zeller *et al.*, 2004; see also: Roberts *et al.*, 1988, Gallagher *et al.*, 1997).

The hybridization of the amino group shows a tendency towards *sp*³ hybridization with angles between 114 and 117° (Table 1). The nitrogen atom is located slightly above the plane of the phenyl ring by 0.074 (3) Å. Together with the orientation of the amino hydrogen atoms, which are located below the phenyl plane by 0.11 (4) and 0.23 (4) Å, this allows for a conjugation of the nitrogen lone pair with the π electrons of the aromatic ring. This is in agreement with the ¹H NMR spectral values, which are typical for ferrocenyl compounds with electron donating substituents: The cyclopentadienyl and phenyl protons have been shifted upfield from TMS, thus further conforming extensive π -overlap and electronic communication along the unsaturated backbone.

In contrast to aminoferrocene (Perrine *et al.*, 2005) and also 1,1'-diaminoferrocene (Shafir *et al.*, 2000), but similar to other structurally analyzed ferrocenyl amines (Rogers *et al.* 1998, Sunkel *et al.*, 2000, Takaki *et al.*, 1959) the title compound does not form hydrogen bonds in the solid state. Also no π - π stacking interactions are found in the structure: neighboring molecules are roughly perpendicular to each other with the C—H groups pointing towards the π -electrons of neighboring Cp and aromatic units (but at distances larger than the sum of the van-der-Waals radii). The packing and the lattice energy thus seems to be solely based on weak dispersion interactions.

Experimental

p-Nitrophenylferrocene was prepared by the method of Hu *et al.* (2001a). The crude product was purified using activated silica as the stationary phase and 95:5 hexanes:ethyl acetate as the eluent (yield 48%). ¹H NMR (400 MHz, CDCl₃): 4.067 (s, 5H, C₅H₅), 4.487 (pt, 2H, C₅H₄), 4.751 (pt, 2H, C₅H₄), 7.710 (d, 2H, C₆H₄), 8.150 (d, 2H, C₆H₄). MS: calculated for (*M*⁺) 307 m/z, found 307 m/z. *p*-Nitrophenylferrocene was also analyzed by single-crystal diffraction (Zeller *et al.*, 2004) confirming the previously reported structure (Roberts *et al.*, 1988, Gallagher *et al.*, 1997). *p*-Ferrocenylaniline was prepared *via* the method of Hu *et al.* (2001b) from *p*-nitrophenylferrocene. The product was purified *via* column chromatography using activated silica as the stationary phase and 70:30 hexanes:ethyl acetate as the eluent (yield 88%). ¹H NMR: 3.620 (s, 2H, NH₂), 4.045 (s, 5H, C₅H₅), 4.249 (pt, 2H, C₅H₄), 4.552 (pt, 2H, C₅H₄), 6.651 (d, 2H, C₆H₄), 7.303 (d, 2H, C₆H₄). MS: calculated for (*M*⁺) 277 m/z, found 277 m/z.

X-ray quality single crystals of *p*-ferrocenylaniline were prepared *via* kinetically controlled solvent evaporation from 70:30 hexanes:ethyl acetate solutions to give yellow platelets.

Refinement

Cp and aromatic H atoms were placed in calculated positions [C—H = 0.95 Å] and were refined with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ of the adjacent carbon atom. Amine hydrogen atom positions and isotropic displacement parameters were freely refined.

The s.u. values of the cell parameters are taken from the software recognizing that the values are unreasonably small (Herbstein, 2000).

Figures

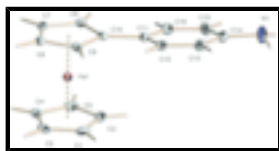


Fig. 1. The molecular structure with the atomic numbering scheme. Thermal displacement parameters are at the 50% probability level, dotted lines represent the Fe- π_{Cp} bonds.



Fig. 2. Scheme of the synthesis of I starting with ferrocene (Hu *et al.*, 2001a,b).

p-Ferrocenylaniline

Crystal data

[Fe(C₅H₅)(C₁₁H₁₀N)]

$M_r = 277.14$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 9.6762$ (7) Å

$b = 8.0284$ (6) Å

$c = 31.515$ (2) Å

$F_{000} = 1152$

$D_x = 1.504$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 8682 reflections

$\theta = 2.5$ – 30.5°

$\mu = 1.21$ mm⁻¹

$T = 100$ (2) K

$V = 2448.2 (3) \text{ \AA}^3$
 $Z = 8$

Plate, yellow
 $0.41 \times 0.19 \times 0.03 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2821 independent reflections
Radiation source: fine-focus sealed tube	2679 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.030$
$T = 100(2) \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.3^\circ$
Absorption correction: multi-scan (SADABS in SAINT-Plus; Bruker, 2003)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.712$, $T_{\text{max}} = 0.960$	$k = -10 \rightarrow 10$
22095 measured reflections	$l = -40 \rightarrow 40$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.095$	$w = 1/[\sigma^2(F_o^2) + (0.0245P)^2 + 5.1787P]$
$S = 1.31$	where $P = (F_o^2 + 2F_c^2)/3$
2821 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
171 parameters	$\Delta\rho_{\text{max}} = 0.57 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.5396 (3)	-0.0175 (3)	0.61108 (8)	0.0200 (5)
H1	0.4875	-0.0652	0.6336	0.024*
C2	0.6406 (3)	0.1104 (3)	0.61509 (8)	0.0193 (5)

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H2	0.6677	0.1632	0.6408	0.023*
C3	0.6937 (2)	0.1451 (3)	0.57415 (8)	0.0191 (5)
H3	0.7629	0.2250	0.5676	0.023*
C4	0.6253 (2)	0.0393 (3)	0.54461 (8)	0.0180 (5)
H4	0.6404	0.0364	0.5148	0.022*
C5	0.5305 (2)	-0.0611 (3)	0.56757 (8)	0.0185 (5)
H5	0.4711	-0.1432	0.5558	0.022*
C6	0.4561 (2)	0.4356 (3)	0.57041 (7)	0.0155 (5)
H6	0.5264	0.5181	0.5709	0.019*
C7	0.4063 (2)	0.3527 (3)	0.53379 (8)	0.0164 (5)
H7	0.4371	0.3705	0.5055	0.020*
C8	0.3022 (2)	0.2383 (3)	0.54663 (7)	0.0159 (5)
H8	0.2514	0.1662	0.5285	0.019*
C9	0.2876 (2)	0.2508 (3)	0.59135 (8)	0.0150 (4)
H9	0.2253	0.1880	0.6083	0.018*
C10	0.3826 (2)	0.3742 (3)	0.60670 (8)	0.0140 (4)
C11	0.3971 (2)	0.4271 (3)	0.65136 (7)	0.0148 (5)
C12	0.3038 (3)	0.3733 (3)	0.68236 (8)	0.0176 (5)
H12	0.2324	0.2979	0.6747	0.021*
C13	0.3132 (3)	0.4273 (3)	0.72402 (8)	0.0207 (5)
H13	0.2485	0.3888	0.7444	0.025*
C14	0.4174 (3)	0.5385 (3)	0.73622 (8)	0.0221 (5)
C15	0.5119 (3)	0.5911 (3)	0.70560 (8)	0.0223 (5)
H15	0.5842	0.6652	0.7134	0.027*
C16	0.5018 (3)	0.5370 (3)	0.66416 (8)	0.0194 (5)
H16	0.5672	0.5749	0.6439	0.023*
Fe1	0.48564 (3)	0.18548 (4)	0.575918 (10)	0.01213 (11)
N1	0.4219 (3)	0.5988 (4)	0.77774 (8)	0.0317 (6)
H1A	0.495 (4)	0.645 (5)	0.7857 (12)	0.034 (10)*
H1B	0.384 (4)	0.535 (5)	0.7961 (12)	0.041 (11)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0177 (11)	0.0187 (12)	0.0237 (13)	0.0051 (10)	0.0035 (10)	0.0087 (10)
C2	0.0168 (11)	0.0213 (12)	0.0197 (12)	0.0043 (10)	-0.0037 (9)	-0.0002 (10)
C3	0.0095 (10)	0.0178 (11)	0.0301 (14)	0.0018 (9)	0.0012 (9)	0.0032 (10)
C4	0.0154 (11)	0.0195 (12)	0.0190 (12)	0.0069 (9)	0.0033 (9)	-0.0001 (10)
C5	0.0152 (11)	0.0114 (10)	0.0288 (13)	0.0033 (9)	-0.0011 (9)	-0.0019 (10)
C6	0.0150 (11)	0.0119 (10)	0.0198 (12)	0.0004 (9)	-0.0023 (9)	0.0018 (9)
C7	0.0183 (11)	0.0164 (11)	0.0144 (11)	0.0056 (9)	-0.0002 (9)	0.0021 (9)
C8	0.0142 (10)	0.0165 (11)	0.0168 (11)	0.0052 (9)	-0.0032 (9)	-0.0019 (9)
C9	0.0109 (10)	0.0144 (10)	0.0196 (11)	0.0019 (9)	-0.0001 (9)	-0.0011 (9)
C10	0.0118 (10)	0.0127 (10)	0.0175 (11)	0.0018 (9)	0.0011 (9)	0.0008 (9)
C11	0.0144 (11)	0.0140 (11)	0.0161 (11)	0.0038 (9)	-0.0010 (9)	-0.0002 (9)
C12	0.0160 (11)	0.0173 (11)	0.0196 (12)	0.0005 (9)	-0.0013 (9)	0.0011 (10)
C13	0.0213 (12)	0.0232 (12)	0.0176 (12)	0.0021 (10)	0.0028 (10)	0.0029 (10)
C14	0.0261 (13)	0.0228 (13)	0.0175 (12)	0.0046 (11)	-0.0040 (10)	-0.0010 (10)

C15	0.0222 (12)	0.0220 (13)	0.0229 (13)	-0.0025 (11)	-0.0037 (10)	-0.0046 (10)
C16	0.0175 (11)	0.0191 (12)	0.0216 (12)	-0.0019 (10)	0.0008 (9)	0.0003 (10)
Fe1	0.00978 (17)	0.01245 (17)	0.01414 (17)	0.00071 (12)	0.00034 (12)	0.00026 (13)
N1	0.0414 (15)	0.0375 (15)	0.0163 (11)	-0.0077 (12)	-0.0016 (11)	-0.0064 (11)

Geometric parameters (Å, °)

C1—C5	1.418 (4)	C8—C9	1.420 (3)
C1—C2	1.423 (4)	C8—Fe1	2.045 (2)
C1—Fe1	2.039 (2)	C8—H8	0.9500
C1—H1	0.9500	C9—C10	1.435 (3)
C2—C3	1.417 (4)	C9—Fe1	2.045 (2)
C2—Fe1	2.034 (2)	C9—H9	0.9500
C2—H2	0.9500	C10—C11	1.477 (3)
C3—C4	1.423 (4)	C10—Fe1	2.057 (2)
C3—Fe1	2.040 (2)	C11—C12	1.399 (3)
C3—H3	0.9500	C11—C16	1.402 (3)
C4—C5	1.420 (3)	C12—C13	1.386 (3)
C4—Fe1	2.044 (2)	C12—H12	0.9500
C4—H4	0.9500	C13—C14	1.400 (4)
C5—Fe1	2.043 (2)	C13—H13	0.9500
C5—H5	0.9500	C14—C15	1.395 (4)
C6—C7	1.417 (3)	C14—N1	1.396 (3)
C6—C10	1.434 (3)	C15—C16	1.380 (4)
C6—Fe1	2.036 (2)	C15—H15	0.9500
C6—H6	0.9500	C16—H16	0.9500
C7—C8	1.422 (3)	N1—H1A	0.84 (4)
C7—Fe1	2.038 (2)	N1—H1B	0.86 (4)
C7—H7	0.9500		
C5—C1—C2	107.8 (2)	C13—C12—C11	121.5 (2)
C5—C1—Fe1	69.84 (14)	C13—C12—H12	119.2
C2—C1—Fe1	69.36 (14)	C11—C12—H12	119.2
C5—C1—H1	126.1	C12—C13—C14	120.5 (2)
C2—C1—H1	126.1	C12—C13—H13	119.8
Fe1—C1—H1	126.3	C14—C13—H13	119.8
C3—C2—C1	108.1 (2)	C15—C14—N1	121.5 (3)
C3—C2—Fe1	69.88 (14)	C15—C14—C13	118.4 (2)
C1—C2—Fe1	69.73 (14)	N1—C14—C13	120.1 (3)
C3—C2—H2	126.0	C16—C15—C14	120.8 (2)
C1—C2—H2	126.0	C16—C15—H15	119.6
Fe1—C2—H2	126.0	C14—C15—H15	119.6
C2—C3—C4	108.0 (2)	C15—C16—C11	121.5 (2)
C2—C3—Fe1	69.42 (14)	C15—C16—H16	119.3
C4—C3—Fe1	69.74 (13)	C11—C16—H16	119.3
C2—C3—H3	126.0	C2—Fe1—C6	116.61 (10)
C4—C3—H3	126.0	C2—Fe1—C7	150.28 (11)
Fe1—C3—H3	126.4	C6—Fe1—C7	40.70 (9)
C5—C4—C3	107.8 (2)	C2—Fe1—C1	40.91 (10)
C5—C4—Fe1	69.66 (13)	C6—Fe1—C1	150.55 (10)

supplementary materials

C3—C4—Fe1	69.46 (14)	C7—Fe1—C1	167.68 (11)
C5—C4—H4	126.1	C2—Fe1—C3	40.70 (10)
C3—C4—H4	126.1	C6—Fe1—C3	107.04 (10)
Fe1—C4—H4	126.4	C7—Fe1—C3	117.30 (10)
C1—C5—C4	108.2 (2)	C1—Fe1—C3	68.61 (10)
C1—C5—Fe1	69.51 (14)	C2—Fe1—C5	68.56 (10)
C4—C5—Fe1	69.69 (14)	C6—Fe1—C5	167.00 (10)
C1—C5—H5	125.9	C7—Fe1—C5	129.35 (10)
C4—C5—H5	125.9	C1—Fe1—C5	40.65 (10)
Fe1—C5—H5	126.5	C3—Fe1—C5	68.48 (10)
C7—C6—C10	108.6 (2)	C2—Fe1—C4	68.61 (10)
C7—C6—Fe1	69.75 (14)	C6—Fe1—C4	128.19 (10)
C10—C6—Fe1	70.29 (13)	C7—Fe1—C4	108.23 (10)
C7—C6—H6	125.7	C1—Fe1—C4	68.54 (10)
C10—C6—H6	125.7	C3—Fe1—C4	40.80 (10)
Fe1—C6—H6	125.9	C5—Fe1—C4	40.65 (10)
C6—C7—C8	108.2 (2)	C2—Fe1—C8	167.25 (10)
C6—C7—Fe1	69.55 (13)	C6—Fe1—C8	68.60 (10)
C8—C7—Fe1	69.89 (13)	C7—Fe1—C8	40.76 (10)
C6—C7—H7	125.9	C1—Fe1—C8	129.28 (10)
C8—C7—H7	125.9	C3—Fe1—C8	151.31 (10)
Fe1—C7—H7	126.3	C5—Fe1—C8	109.07 (10)
C9—C8—C7	107.9 (2)	C4—Fe1—C8	118.36 (10)
C9—C8—Fe1	69.70 (13)	C2—Fe1—C9	128.50 (10)
C7—C8—Fe1	69.35 (13)	C6—Fe1—C9	68.64 (10)
C9—C8—H8	126.1	C7—Fe1—C9	68.47 (10)
C7—C8—H8	126.1	C1—Fe1—C9	108.39 (10)
Fe1—C8—H8	126.5	C3—Fe1—C9	166.38 (10)
C8—C9—C10	108.7 (2)	C5—Fe1—C9	118.54 (10)
C8—C9—Fe1	69.68 (13)	C4—Fe1—C9	151.82 (10)
C10—C9—Fe1	69.95 (13)	C8—Fe1—C9	40.62 (9)
C8—C9—H9	125.7	C2—Fe1—C10	106.83 (10)
C10—C9—H9	125.7	C6—Fe1—C10	41.02 (9)
Fe1—C9—H9	126.3	C7—Fe1—C10	68.87 (10)
C6—C10—C9	106.6 (2)	C1—Fe1—C10	117.16 (10)
C6—C10—C11	127.9 (2)	C3—Fe1—C10	127.48 (10)
C9—C10—C11	125.5 (2)	C5—Fe1—C10	151.29 (10)
C6—C10—Fe1	68.69 (13)	C4—Fe1—C10	166.22 (10)
C9—C10—Fe1	69.09 (13)	C8—Fe1—C10	68.86 (9)
C11—C10—Fe1	127.96 (17)	C9—Fe1—C10	40.96 (9)
C12—C11—C16	117.4 (2)	C14—N1—H1A	117 (3)
C12—C11—C10	121.0 (2)	C14—N1—H1B	114 (3)
C16—C11—C10	121.6 (2)	H1A—N1—H1B	115 (4)

Fig. 1

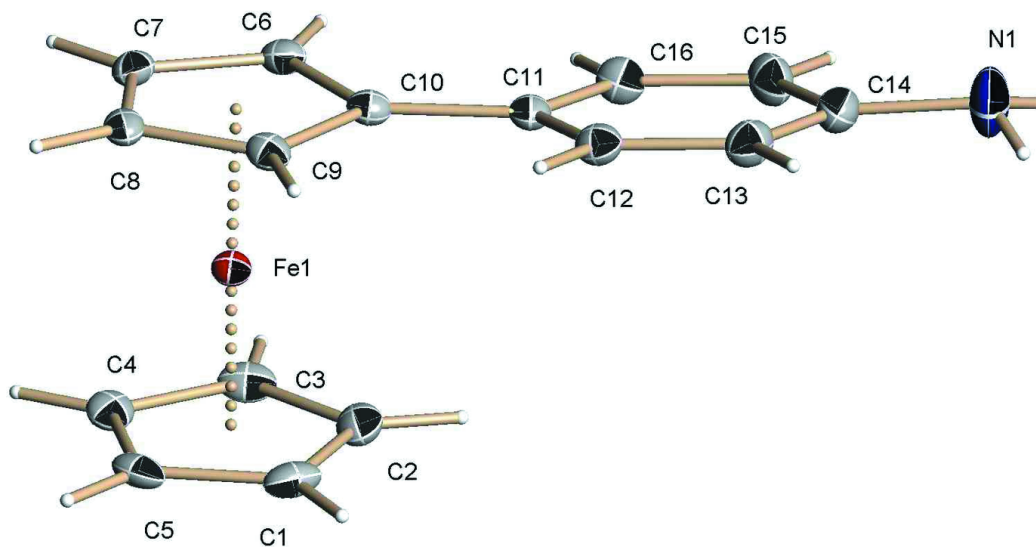


Fig. 2

