

John F. Gallagher,\* Keith  
Hanlon, Joshua Howarth† and  
Jean-Luc ThomasSchool of Chemical Sciences, Dublin City  
University, Dublin 9, Ireland

† Joint contribution.

Correspondence e-mail: john.gallagher@dcu.ie

## Key indicators

Single-crystal X-ray study  
 $T = 297$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.041  
 $wR$  factor = 0.105  
Data-to-parameter ratio = 18.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## 2-(3-Chlorophenyl)-1-ferrocenylmethyl-1H-1,3-benzimidazole: an electroactive agent for anion sensor and malarial parasite studies

The title compound,  $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{19}\text{H}_{14}\text{ClN}_2)]$ , a model electroactive agent for anion sensor and malarial parasite studies, has Fe—C bond lengths in the range 2.020 (3)–2.0543 (18) Å. The Fe···Cg distances (Cg indicates a ring centroid) are essentially similar, with values of 1.6467 (10) and 1.6487 (11) Å for the substituted and unsubstituted cyclopentadienyl rings, respectively, with a linear Cg···Fe···Cg angle of 179.12 (7)°. The Fe—C<sub>Cp</sub>—Csp<sup>3</sup> angle is 128.43 (13)° and the Fe1—C<sub>Cp</sub>—Csp<sup>3</sup>—N<sub>Bz</sub> torsion angle 110.27 (17)° (Cp is cyclopentadienyl and Bz is benzimidazole). Weak C—H···Cl contacts form the only intermolecular interactions of significance.

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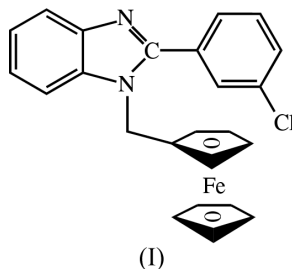
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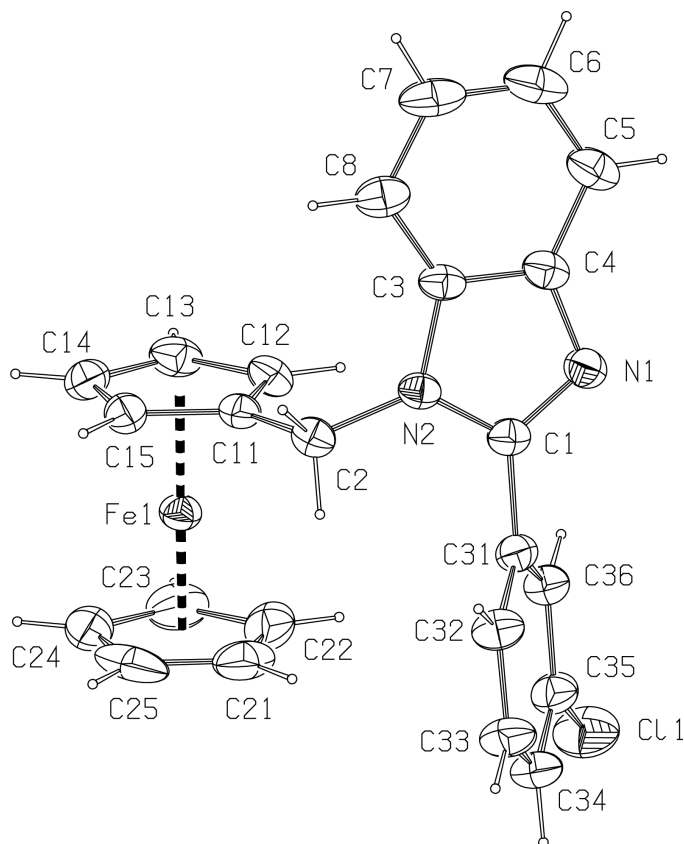
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## Comment

The synthesis of anion receptors is currently an area of intense research activity due primarily to the ubiquitous roles which anions play in both chemical and biological processes, for example, as substrates or co-factors for enzymes and as nucleophiles, redox agents and phase transfer catalysts. In the past decade, combinations of an organometallic moiety with an amide N—H group have been demonstrated to be essential components in many anion-recognition receptors (Beer, 1998; Kingston *et al.*, 1999). 1,3-Disubstituted imidazolium cations have been utilized as such recently (Sato *et al.*, 1999; Thomas *et al.*, 2000) and their synthetic intermediates (imidazolin-2-ylidenes) are also of interest as carbenes and the subject of several synthetic and structural investigations (Benito *et al.*, 1995; Bildstein *et al.*, 1998, 1999).

Recently, benzimidazole systems have attracted our considerable attention in synthetic and applied biological research (Howarth *et al.*, 2000; Thomas *et al.*, 2000; Howarth & Hanlon, 2001). Compound (I), depicted in Fig. 1, is obtained from 2-(3-chlorophenyl)benzimidazole and (trimethyl)ammoniumferrocenylmethyl iodide (Pauson *et al.*, 1966; Ferguson *et al.*, 1994a) and is an important electroactive model compound for application in anion sensor studies (Thomas *et al.*, 2000), as well as in malarial parasite research (Howarth & Hanlon, 2001).





**Figure 1**  
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

In (I), selected bond lengths and angles are listed in Table 1. The Fe—C bond lengths are in the range 2.020 (3)–2.0543 (18) Å; the Fe1...Cg(1,2) distances (Cg1 and Cg2 are centroids of the cyclopentadienyl rings) are comparable, 1.6467 (10) and 1.6487 (11) Å for the substituted and unsubstituted rings, respectively, with a linear Cg1...Fe1...Cg2 angle of 179.12 (7)°. The cyclopentadienyl rings are essentially eclipsed, with a C21—Fe1—C11—C2 torsion angle of  $-2.8$  (2)° and there is no evidence for disorder in the unsubstituted C<sub>5</sub>H<sub>5</sub> ring. The Fe—C<sub>Cp</sub>—Csp<sup>3</sup> angle is 128.43 (13)° and similar to 128.3 (3)° in *N*-ferrocenylmethyl-2-ferrocenylbenzimidazole, (II) (Benito *et al.*, 1995), or 127.86 (12) and 126.80 (12)° in the two independent molecules of racemic ferrocenyl(phenyl)methanol (Ferguson *et al.*, 1994b). The benzimidazole is twisted with respect to the ferrocenyl moiety, with Fe1—C<sub>Cp</sub>—Csp<sup>3</sup>—N2 = 110.27 (17)° and C<sub>Cp</sub>—Csp<sup>3</sup>—N2—C3 = 71.7 (2)° [the N1—C1—C31—C32 angle is 126.9 (2)°], and due to the bulky ferrocenyl and benzimidazole groups avoiding possible sterically hindered H...H contacts. In the crystal structure, the shortest H...Cl1 contacts and involving C25—H25 and C36—H36 [C...Cl 3.802 (5) and 3.670 (2) Å] are listed in Table 2.

A search of the October 2000 version of the Cambridge Structural Database using ConQuest Version 1.1 (Allen & Kennard, 1993) for molecules similar to (I) shows that such

compounds are relatively rare and include *N*-ferrocenylmethyl-2-ferrocenylbenzimidazole (ZIJPIR; Benito *et al.*, 1995) and *N*-ferrocenylmethyl-2-ferrocenylbenzimidazolium tetrafluoroborate (GIFLUC; Li *et al.*, 1998), which differ from (I) primarily through replacement of the chlorophenyl group by a ferrocenyl group.

## Experimental

To a mixture of 2-(chlorophenyl)benzimidazole (2.5 g, 10 mmol) and K<sub>2</sub>CO<sub>3</sub> (2.07 g, 15 mmol) in CH<sub>3</sub>CN (100 ml) was added (trimethyl)ammoniumferrocenylmethyl iodide (4.05 g, 10.5 mmol) (Pauson *et al.*, 1966; Ferguson *et al.*, 1994a), and the mixture was refluxed for 12 h. The reaction was cooled to room temperature, water added and the suspension extracted into CHCl<sub>3</sub>. The organic layer was washed with water, dried (MgSO<sub>4</sub>) and evaporated under vacuum to leave a brown solid. The crude product was purified by column chromatography on silica gel using CH<sub>2</sub>Cl<sub>2</sub>—CH<sub>3</sub>OH (97:3) as eluent. The title compound was obtained as a light-brown powder. Yield 2.8 g (64%), m.p. 435–438 K (uncorrected). Analysis for C<sub>24</sub>H<sub>19</sub>ClFeN<sub>2</sub>, calculated: C 67.57, H 4.45, N 6.56%; found: C 67.47, H 4.51, N 6.52%. IR (KBr, ν cm<sup>-1</sup>), 3051, 2971, 2308, 1708, 1639, 1456, 1422, 1364, 1330, 1261, 1153, 1101, 1027, 1008, 895, 741, 706, 695. <sup>1</sup>H NMR [400 MHz, δH (p.p.m.), CDCl<sub>3</sub>], 7.82 (*m*, 2H, aryl-H), 7.69 (*m*, 1H, C<sub>6</sub>H<sub>4</sub>), 7.56–7.48 (*m*, 3H, aryl-H and C<sub>6</sub>H<sub>4</sub>), 7.34 (*m*, 2H, C<sub>6</sub>H<sub>4</sub>), 5.22 (*s*, 2H, Fc-CH<sub>2</sub>), 4.13–4.07 (*m*, 9H, Fc-H). <sup>13</sup>C NMR (δC, CDCl<sub>3</sub>), 152.27, 143.27, 136.04, 135.08, 132.79, 130.4, 130.35, 130.08, 128.21, 123.49, 123.05, 120.48, 110.86, 83.36, 69.25, 69.14, 68.71, 44.95.

## Crystal data

[Fe(C<sub>5</sub>H<sub>5</sub>)(C<sub>19</sub>H<sub>14</sub>ClN<sub>2</sub>)]  
M<sub>r</sub> = 426.71  
Monoclinic, P2<sub>1</sub>/n  
a = 11.6838 (9) Å  
b = 9.9220 (6) Å  
c = 16.7493 (14) Å  
β = 90.542 (6)°  
V = 1941.6 (2) Å<sup>3</sup>  
Z = 4

D<sub>x</sub> = 1.46 Mg m<sup>-3</sup>  
Mo Kα radiation  
Cell parameters from 27 reflections  
θ = 4.5–16.1°  
μ = 0.93 mm<sup>-1</sup>  
T = 297 (1) K  
Block, orange  
0.37 × 0.24 × 0.21 mm

## Data collection

Bruker P4 diffractometer  
ω–2θ scans  
Absorption correction: ψ scan  
(North *et al.*, 1968)  
T<sub>min</sub> = 0.682, T<sub>max</sub> = 0.746  
5483 measured reflections  
4669 independent reflections  
3646 reflections with I > 2σ(I)

R<sub>int</sub> = 0.033  
θ<sub>max</sub> = 28.0°  
h = –15 → 1  
k = –13 → 1  
l = –22 → 22  
3 standard reflections every 197 reflections  
intensity decay: 2%

## Refinement

Refinement on F<sup>2</sup>  
R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.041  
wR(F<sup>2</sup>) = 0.105  
S = 1.03  
4669 reflections  
253 parameters  
H-atom parameters constrained

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0495P)<sup>2</sup> + 0.6062P]  
where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> < 0.001  
Δρ<sub>max</sub> = 0.70 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = –0.55 e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Fe1—C11	2.0543 (18)	Fe1—C25	2.020 (3)
Fe1—C12	2.0419 (19)	C11—C35	1.742 (2)
Fe1—C13	2.038 (2)	N1—C1	1.310 (3)
Fe1—C14	2.034 (2)	N1—C4	1.389 (3)
Fe1—C15	2.040 (2)	N2—C1	1.378 (3)
Fe1—C21	2.031 (3)	N2—C2	1.456 (2)
Fe1—C22	2.034 (3)	N2—C3	1.386 (2)
Fe1—C23	2.032 (2)	C1—C31	1.480 (3)
Fe1—C24	2.026 (2)	C2—C11	1.504 (3)
C1—N1—C4	104.77 (17)	N1—C1—C31	123.68 (19)
C1—N2—C2	128.44 (16)	N2—C1—C31	122.80 (18)
C1—N2—C3	105.85 (16)	N2—C2—C11	111.86 (15)
C2—N2—C3	124.26 (17)	C2—C11—Fe1	128.43 (13)
N1—C1—N2	113.52 (17)		
C3—N2—C2—C11	71.7 (2)	C21—Fe1—C11—C2	−2.8 (2)
N2—C2—C11—C12	18.6 (3)	N2—C1—C31—C36	128.8 (2)
N2—C2—C11—Fe1	110.27 (17)		

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C25—H25 $\cdots$ Cl1 <sup>i</sup>	0.93	3.05	3.802 (5)	139
C36—H36 $\cdots$ Cl1 <sup>ii</sup>	0.93	3.03	3.670 (2)	127

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

All H atoms bound to C atoms were treated as riding, with *SHELXL97* (Sheldrick, 1997) defaults for C—H distances and with  $U_{iso}(H) = 1.5U_{eq}(C)$  for methyl H atoms and  $1.2U_{eq}(C)$  for the remainder. Examination of the structure with *PLATON* (Spek, 1998) showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

*ORTEPIII* (Burnett & Johnson, 1996) and *PLATON* (Spek, 1998); software used to prepare material for publication: *SHELXL97* and *PREP8* (Ferguson, 1998).

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