Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Benzyl-3-(ferrocenylmethyl)imidazolium iodide

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Received 26 April 2004
Accepted 5 May 2004
Online 15 May 2004

The title ferrocenyl imidazolium salt, $[Fe(C_5H_5)(C_{16}H_{16}N_2)]I$, results from the reaction of 1-benzylimidazole and (ferrocenylmethyl)trimethylammonium iodide in acetonitrile. It crystallizes in a monoclinic space group, showing disorder of a cyclopentadienyl ring.

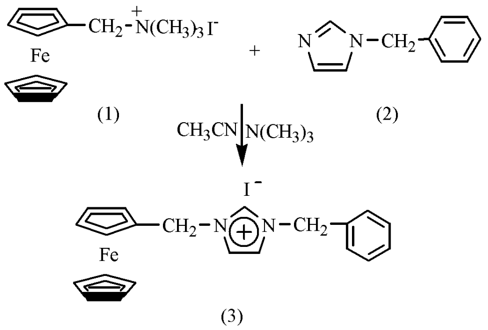
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.029
wR factor = 0.068
Data-to-parameter ratio = 13.5

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

Comment

The ferrocenyl moiety represents a quite bulky group with unique spatial requirements due to its sandwich shape, and electronically, the powerful donor capacity of ferrocene (Lukasser *et al.*, 1995). Thus, some ferrocene compounds are excellent non-linear optical materials, because they can act as strong electron donors and contain electron-flow bridges (Long, 1995), while carbenes containing ferrocenyl substituents are interesting compounds due to their stereoelectronic influence of the metallocenyl groups in terms of steric protection, electronic donation, and reversible redox chemistry (Bildstein *et al.*, 1999; Bildstein, 2001). The title compound, (3), was prepared *via* the reaction between ferrocenemethyltrimethylammonium iodide, (1), and 1-benzylimidazole, (2), as shown in the scheme below.



The structure of (3) shows that it is a quaternary ammonium salt formed from substituted imidazolium and an iodide anion (Fig. 1). The N1—C8 and N2—C8 distances are 1.317 (4) and 1.321 (4) Å, respectively. They are different from normal Csp^3—N (1.47 Å), Csp^2═N (1.28 Å) and Csp^2—N (1.36 Å) (March, 1977) bond lengths, intermediate between the Csp^2—N and Csp^2═N bonds. There is electron delocalization over atoms N1, C8 and N2. The N2—C11 and N1—C7 distances are 1.480 (4) and 1.469 (4) Å, respectively, close to normal Csp^3—N distances. Thus, the N atom at the 3-position of the imidazole ring forms the quaternary ammonium cation. In addition, the C11—C12 and C6—C7 distances are also a little different from normal values. The crystal packing is stabilized by a C—H···I hydrogen bond (Table 2 and Fig. 2).

DOI: 10.1107/S1600536804011006

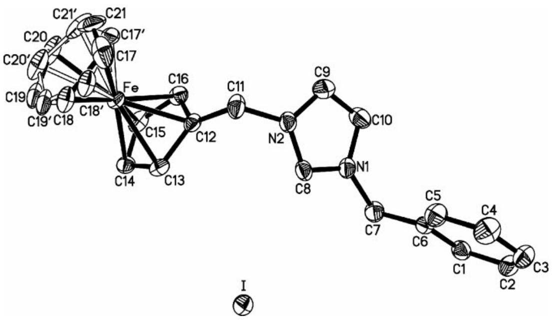


Figure 1
View of (3), showing the disordered cyclopentadienyl ring and 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

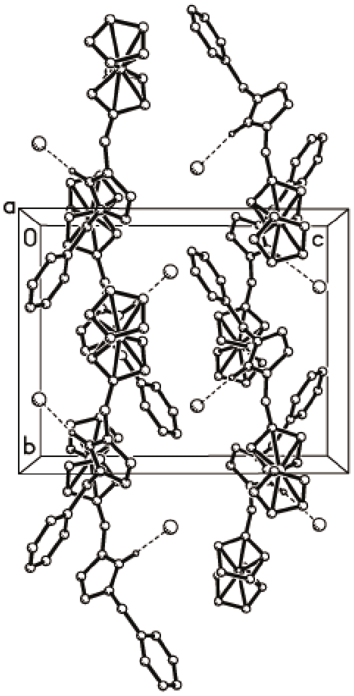


Figure 2
The crystal packing, viewed down the *a* axis. The H8···I hydrogen bonds are denoted by dashed lines. H atoms have been omitted for clarity.

Experimental

The title compound was prepared according to a known procedure (Thomas *et al.*, 2000). A mixture of (ferrocenylmethyl)trimethylammonium iodide (Rausch & Slegel, 1969) (0.77 g, 2.00 mmol) and *N*-benzylimidazole (0.32 g, 2.02 mmol) in MeCN (15 ml) was refluxed for 40 h. The solvent was removed *in vacuo*. The residue was a brown viscous liquid. This was dissolved in dichloromethane (2 ml) and ethyl acetate (500 ml) was added. After stirring, it was kept overnight at room temperature and orange crystals were obtained. Spectroscopic analysis, IR (KBr, cm^{-1}): 3453 (*m*), 2859 (*m*), 1557 (*m*), 1501 (*m*), 1451 (*m*), 1233 (*m*), 1145 (*s*), 1105 (*m*), 1078 (*m*), 1031 (*m*), 1001 (*m*), 821 (*m*), 716 (*s*), 626 (*m*); ^{1}H NMR ($CDCl_3$): 10.015 (*s*, 1H, 2-imiH), 7.443 (*s*, 2 × 1H, 4- and 5-imiH), 7.726 (*m*, 5H, Ar), 5.477 (*s*, 2H, CH_2-Ar), 5.309 (*s*, 2H, CH_2-Fc), 4.457 (*s*, 2H, CpH), 4.180 (*s*, 7H, CpH); ^{13}C NMR ($CDCl_3$): 134.971 (2-imi), 132.483 (C1 of Ar), 129.384, 129.262, 128.972 (Ar), 127.583, 121.600 (imidazole), 78.328 (C1 of Cp), 69.612, 69.536, 68.987 (Cp), 53.052 (CH_2Ar), 49.800 (CH_2Fc); analysis calculated for $C_{21}H_{21}FeIN_2$: C 52.06, H 4.33, N 5.78%; found: C 52.26, H 4.89, N 5.54%.

Crystal data

$[Fe(C_5H_5)(C_{16}H_{16}N_2)]I$
$M_r = 484.15$
Monoclinic, $P2_1/n$
$a = 13.086\ (2)$ Å
$b = 11.043\ (2)$ Å
$c = 13.729\ (2)$ Å
$\beta = 92.11\ (2)^\circ$
$V = 1982.5\ (5)$ Å^3
$Z = 4$

$D_x = 1.622$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 34 reflections
$\theta = 2.8$–14.8°
$\mu = 2.32$ mm^{-1}
$T = 292\ (2)$ K
Block, orange
0.30 × 0.22 × 0.22 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: multi-scan (*SHELXTL*; Siemens, 1994)
$T_{\min} = 0.727$, $T_{\max} = 0.845$
4120 measured reflections
3593 independent reflections
2818 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$
$\theta_{\max} = 25.3^\circ$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 13$
$l = -16 \rightarrow 16$
3 standard reflections every 97 reflections intensity decay: 3.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.068$
$S = 0.93$
3593 reflections
266 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0392P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.47$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0032 (2)

Table 1
Selected geometric parameters (Å, °).

Fe—C12	2.012 (3)	Fe—C15	2.048 (3)
Fe—C17	2.017 (12)	N1—C8	1.317 (4)
Fe—C18	2.024 (6)	N1—C10	1.369 (4)
Fe—C21	2.026 (17)	N1—C7	1.469 (4)
Fe—C13	2.027 (3)	N2—C8	1.321 (4)
Fe—C20	2.033 (11)	N2—C9	1.370 (5)
Fe—C14	2.036 (3)	N2—C11	1.480 (4)
Fe—C16	2.039 (3)	C6—C7	1.513 (5)
Fe—C19	2.045 (7)	C11—C12	1.487 (4)
C8—N1—C7	124.9 (3)	C9—N2—C11	126.2 (3)
C10—N1—C7	126.6 (3)	N1—C7—C6	113.6 (3)
C8—N2—C9	107.9 (3)	N1—C8—N2	109.0 (3)
C8—N2—C11	125.7 (3)	N2—C11—C12	110.8 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···I	0.96 (3)	3.00 (3)	3.876 (4)	152 (2)

The unsubstituted cyclopentadienyl ring shows disorder between two positions, which were refined with occupancies of 67 and 33%. The H atoms attached to C8, C9 and C10 were found in a difference Fourier map and refined freely. All remaining H atoms were positioned geometrically and refined using a riding model [C—H = 0.93–0.93 Å; $U_{\text{iso}} = 1.2U_{\text{eq}}$(C)].

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank the NSF (grant Nos. 20021001 and 20172024) and the 'Hundred Scientist Program' of the Chinese Academy of Sciences for financial support of this work.

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