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(Ferrocenylmethyl)trimethylammonium triiodide

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.005 Å
R factor = 0.026
wR factor = 0.062
Data-to-parameter ratio = 25.4

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

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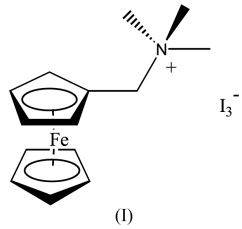
The salt (ferrocenylmethyl)trimethylammonium iodide, $[Fe(C_5H_5)(C_9H_{15}N)]I$, is used in an established procedure to attach ferrocenylmethyl frameworks to secondary amines. The title salt, $[Fe(C_5H_5)(C_9H_{15}N)]I_3$, obtained by diffusion of Et_2O vapour into a CH_2Cl_2 solution containing equimolar $[Fe(C_5H_5)(C_9H_{15}N)]I$ and I_2, shows the expected cation balanced by an asymmetric but isolated triiodide counteranion [I—I = 3.0006 (4) and 2.8728 (4) Å].

Comment

A well established procedure for the introduction of ferrocenylmethyl frameworks into secondary amines involves the use of the salt (ferrocenylmethyl)trimethylammonium iodide, $[Fe(C_5H_5)(C_9H_{15}N)]I$ (Beer *et al.*, 1991). A number of crystal structures containing the (ferrocenylmethyl)trimethylammonium cation, $[Fe(C_5H_5)(C_9H_{15}N)]^+$, have been reported, including $[Fe(C_5H_5)(C_9H_{15}N)]I$ (Ferguson *et al.*, 1994). The reaction of $[Fe(C_5H_5)(C_9H_{15}N)]I$ (Lednicer & Hauser, 1960) with one equivalent of I_2 in CH_2Cl_2 gave the title compound, $[Fe(C_5H_5)(C_9H_{15}N)]I_3$, (I), in good yield by diffusion of Et_2O vapour into the reaction mixture.



(I)

An X-ray diffraction analysis of (I) shows the expected cation balanced by an asymmetric triiodide counter-anion [I1—I2 = 3.0006 (4) and I2—I3 2.8728 (4) Å]. Although the triiodide does not participate in any significant I···I intermolecular interactions, and the intramolecular geometry of the (ferrocenylmethyl)trimethylammonium cation shows no unusual features, there are a number of C—H···I contacts in the range 3.07–3.33 Å. The shortest I···C contact here is one of 3.791 (3) Å between atoms I3 and C15($1-x$, $1-y$, $-z$), compared with one of 3.954 (2) Å in $[Fe(C_5H_5)(C_9H_{15}N)]I$ (Ferguson *et al.*, 1994).

Experimental

Et_2O vapour was allowed to diffuse into a CH_2Cl_2 (10 ml) solution of (ferrocenylmethyl)trimethylammonium iodide (0.05 g, 0.13 mmol) and I_2 (0.033 g, 0.13 mmol). Well shaped blocky crystals of the title

compound were formed in 80% yield. Elemental analysis, found (calculated for $C_{14}H_{20}FeI_3N$): C 26.42 (26.32), H 3.24 (3.15), N 2.29 (2.20)%. FT Raman (500–10 cm^{-1}): 131.4, 101.1 cm^{-1}.

Crystal data

$[Fe(C_5H_5)(C_9H_{15}N)]I_3$	D_x = 2.228 Mg m^{-3}
M_r = 638.86	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 7671 reflections
a = 11.6959 (9) Å	θ = 2.3–27.6°
b = 10.6136 (9) Å	μ = 5.64 mm^{-1}
c = 15.3474 (12) Å	T = 150 (2) K
β = 91.638 (2)°	Block, red
V = 1904.4 (3) $Å^3$	0.50 × 0.36 × 0.30 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4393 independent reflections
	3925 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.019
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)	θ_{max} = 27.6°
	$h = -15 \rightarrow 14$
T_{min} = 0.064, T_{max} = 0.180	$k = -13 \rightarrow 12$
13 480 measured reflections	$l = -20 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 2.356P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.026	
$wR(F^2)$ = 0.062	
S = 1.09	$(\Delta/\sigma)_{max}$ = 0.001
4393 reflections	$\Delta\rho_{max}$ = 1.08 e $Å^{-3}$
173 parameters	$\Delta\rho_{min}$ = −1.18 e $Å^{-3}$
H-atom parameters constrained	Extinction correction: *SHELXL*97 (Sheldrick, 1997)
	Extinction coefficient: 0.00106 (8)

Table 1
Selected geometric parameters (Å, °).

I1—I2	3.0006 (4)	I2—I3	2.8728 (4)
I1—I2—I3	178.600 (10)		

All H atoms were placed in geometrically calculated positions, with C—H distances of 0.98, 0.99 and 1.00 Å for methyl, methylene and cyclopentadienyl groups, respectively, and with $U_{iso}(H)$ = $1.2U_{eq}(C)$. The largest residual electron-density peaks and holes all lie close to I atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* and *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *enCIFer* (CCDC, 2003) and *PLATON* (Spek, 2003).

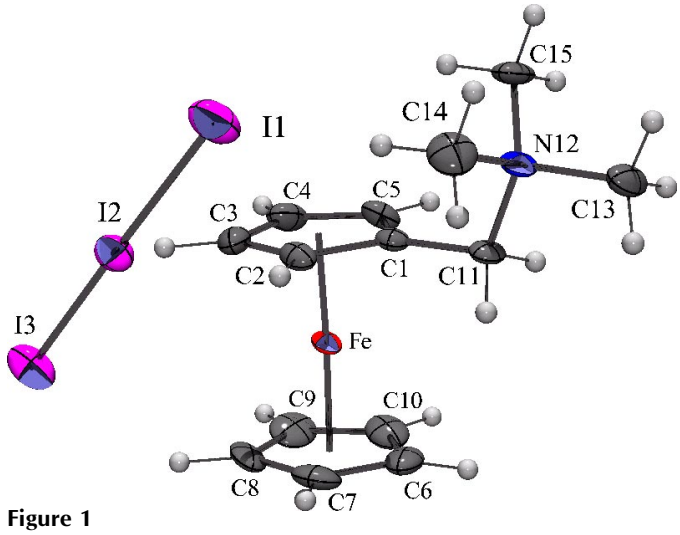


Figure 1
A view of the title salt, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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