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Dicarbonyl(η^5-cyclopentadienyl)(trimethylsilyl isocyanide)iron(II) trifluoromethanesulfonate

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.008 Å
R factor = 0.068
wR factor = 0.171
Data-to-parameter ratio = 19.0

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

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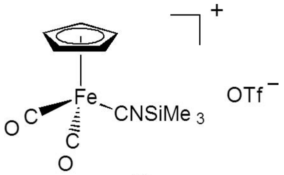
In the title compound, $[Fe(C_5H_5)(C_4H_9NSi)(CO)_2](CF_3SO_3)$, the Fe atom is coordinated by two carbonyl groups, a trimethylsilylisocyanide ligand and a cyclopentadienyl ligand in a typical piano-stool structure. The Fe—CN, C≡N and CN—Si bond distances are 1.862 (4), 1.157 (6) and 1.794 (4) Å, respectively, and the Fe—C—N—Si arrangement is almost linear.

Comment

The crystal structure of $[CpFe(CO)_2(CNSiMe_3)]OTf$ (Cp = cyclopentadienyl and OTf = trifluoromethanesulfonate), (I), shows that the Fe^{II} atom has two carbonyl groups, a trimethylsilylisocyanide substituent and a cyclopentadienyl (Cp) ring coordinated in an η^5-fashion, resulting in a typical piano-stool structure (Fig. 1).



(I)

Only four crystal structures have been reported to date for silylisocyanide complexes: $(NEt_4)_2[Si\{(NC)(CO)_5Cr\}_6]$ (Fritz *et al.*, 1992), $[Re(dppe)_2Cl(CNSiMe_3)]$ (dppe = diphenylphosphinoethane; da Silva *et al.*, 2000), $[Cp^*(PMe_3)(^iPr)Rh(CNSiPh_3)][BAr^f_4]$ [$Ar^f = C_6H_3(CF_3)_2$-3,5; Taw *et al.*, 2002] and $[Cp(CO)MeFe(CNSiPh_3)]$ (Nakazawa *et al.*, 2004). The Fe—CN distance [1.862 (4) Å] in (I) is comparable with those previously reported in $[CpFe(CO)_2(CN)]$ [1.933 (4) Å; Darensbourg *et al.*, 2001] and $[CpFe(Me)(PPh_3)(CNSiPh_3)]$ [1.760 (5) Å; Nakazawa *et al.*, 2004]. The CN—Si bond distance [1.794 (4) Å] in (I) is longer than that reported previously [1.760 (5) Å; Nakazawa *et al.*, 2004]. The angles around the Fe^{II} atom [C6—Fe1—C7 = 94.6 (3)°, C6—Fe1—C8 93.2 (2)° and C7—Fe1—C8 = 93.1 (2)°] are similar to those in $[CpFe(CO)_2(CN)]$ (Darensbourg *et al.*, 2001). The C≡N distance [1.157 (6) Å] is unremarkable and the Fe—C—N—Si arrangement in (I) is almost linear [Fe—C—N = 178.2 (4)° and C—N—Si = 174.7 (4)°], whereas the corresponding angle for $[CpFe(Me)(PPh_3)(CNSiPh_3)]$ is bent significantly [C—N—Si = 156.7 (5)°; Nakazawa *et al.*, 2004] because of steric repulsion between the bulky PPh_3 and $SiPh_3$ groups.

Experimental

TMSOTf (trimethylsilyl trifluoromethanesulfonate; 0.2 ml, 1.11 mmol) was added to a CH_2Cl_2 solution (3 ml) of $CpFe(CO)_2$-

(CN) (100 mg, 0.49 mmol) prepared according to literature methods (Darensbourg, 1975; Piper *et al.*, 1955). After stirring mixture for 1 h at 195 K, the solvent was removed under reduced pressure to give a brown oil, which was washed repeatedly with diethyl ether and dried *in vacuo* to give [CpFe(CO)$_2$(CNSiMe$_3$)]OTf, (I), as a pale-brown powder (150 mg, 0.35 mmol, 71%). Single crystals were obtained by solvent diffusion at 253 K over a few days from a CH$_2$Cl$_2$ layer containing (I) and an overlayer of hexane. IR (CH$_2$Cl$_2$, cm^{-1}): ν_{CO} 2062, 2018; ν_{CN} 2121; ^{1}H NMR (400 MHz, CD$_2$Cl$_2$): δ −0.05 (*s*, SiMe$_3$, 9 H), 5.20 (*s*, Cp, 5 H); ^{13}C NMR (100.3 MHz, CD$_2$Cl$_2$): δ −0.40 (*s*, SiMe$_3$), 87.5 (*s*, Cp), 122.2 (*q*, J = 318 Hz, CF$_3$), 207.9 (*s*, CO).

Crystal data

[Fe(C$_5$H$_5$)(C$_4$H$_9$NSi)(CO)$_2$]-(CF$_3$SO$_3$)	D_x = 1.546 Mg m^{-3}
M_r = 425.23	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 7143 reflections
a = 17.370 (5) Å	θ = 3.1–27.5°
b = 13.131 (4) Å	μ = 1.05 mm^{-1}
c = 8.011 (2) Å	T = 153.2 K
β = 91.803 (7)°	Block, yellow
V = 1826.4 (9) Å^3	0.30 × 0.13 × 0.10 mm
Z = 4	

Data collection

Rigaku/MSC Mercury CCD diffractometer	4113 independent reflections
ω scans	3344 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (Jacobson, 1998)	$R_{\rm int}$ = 0.042
$T_{\rm min}$ = 0.758, $T_{\rm max}$ = 0.900	$\theta_{\rm max}$ = 27.5°
17930 measured reflections	$h = -22 \rightarrow 22$
	$k = -14 \rightarrow 17$
	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 7.0396P]$
$R[F^2 > 2\sigma(F^2)]$ = 0.068	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.171	$(\Delta/\sigma)_{\rm max}$ = 0.001
S = 1.16	$\Delta\rho_{\rm max}$ = 1.65 e Å^{-3}
4113 reflections	$\Delta\rho_{\rm min}$ = −0.60 e Å^{-3}
217 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Fe1—C1	2.079 (6)	Fe1—C8	1.862 (4)
Fe1—C2	2.088 (6)	Si1—N1	1.794 (4)
Fe1—C3	2.099 (5)	Si1—C9	1.842 (6)
Fe1—C4	2.090 (5)	Si1—C10	1.840 (7)
Fe1—C5	2.088 (5)	Si1—C11	1.842 (7)
Fe1—C6	1.786 (6)	N1—C8	1.157 (6)
Fe1—C7	1.791 (6)		
C6—Fe1—C7	94.6 (3)	N1—Si1—C10	106.3 (2)
C6—Fe1—C8	93.2 (2)	N1—Si1—C11	104.8 (2)
C7—Fe1—C8	93.1 (2)	Si1—N1—C8	174.7 (4)
N1—Si1—C9	103.8 (2)	Fe1—C8—N1	178.2 (4)

H atoms were refined using a riding model, with C—H = 0.95 Å, and fixed individual displacement parameters [$U_{\rm iso}$(H) = $U_{\rm eq}$(C)].

Data collection: *CrystalClear* (Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2000); program(s) used to solve structure: *DIRDIF94* (Beurskens *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

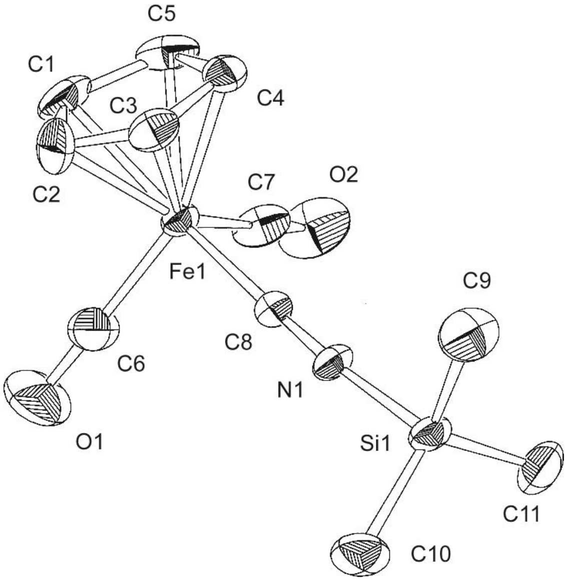


Figure 1
ORTEPII (Johnson, 1976) drawing of the cation of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

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