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Carbonyl(η^5-cyclopentadienyl)(3,5-dimethylpyridine-κN)(triethylsilyl)iron(II)

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Key indicators

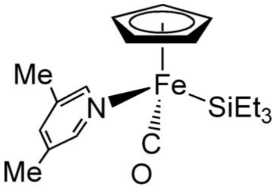
Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.054
wR factor = 0.147
Data-to-parameter ratio = 16.8

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

In the title compound, [Fe(C_5H_5)($C_6H_{15}Si$)(C_7H_9N)(CO)], the Fe atom is coordinated by carbonyl, 3,5-dimethylpyridine, triethylsilyl and cyclopentadienyl ligands in a typical piano-stool structure. The Fe—Si and Fe—N distances are 2.3341 (9) and 1.982 (2) Å, respectively.

Comment

It is well known that pyridine is a weak ligand toward transition metal complexes. A large number of molecular structures of transition metal complexes with pyridine have been determined by X-ray crystallographic analysis. However, only two crystal structures have been reported to date for group 8 transition metal triads having 3,5-dimethylpyridine, *viz.* [$RuCl_2(Me_2SO)_2$(3,5-dimethylpyridine)(1,2-dimethylimidazole)] (Alessio *et al.*, 1998) and [Ru(terpy*)(phen)(3,5-dimethylpyridine)][PF_6]$_2$ [terpy* = 4-(3,5-di-*tert*-butyl)phenyl-2,2′;6′,2″-terpyridine] (Bonnet *et al.*, 2003). This paper is the first report of an X-ray crystal structure of an iron complex with 3,5-dimethylpyridine.



(I)

The title complex, [(C_5H_5)(CO)Fe($SiEt_3$)($C_5NH_3Me_2$-3,5)], (I), was synthesized by the reaction of (C_5H_5)$(CO)_2$Fe($SiEt_3$) with 3,5-dimethylpyridine under photolysis. Complex (I) (Fig. 1) has a typical three-legged piano-stool configuration; the iron has a terminal CO ligand, a triethylsilyl ligand, a 3,5-dimethylpyridine and a cyclopentadienyl ligand (Cp) bonded in an η^5-fashion. Structural disorder was observed in the Cp group [major component s.o.f. = 0.64 (2) and minor = 0.36 (2)]. The Fe1—Si1, Fe1—N1 and Fe1—C12 distances [2.3341 (9), 1.982 (2) and 1.722 (3) Å, respectively] in (I) are similar to those previously reported {[Cp*(CO)Fe($SiMe_2NPh_2$)(py)]; Cp* = C_5Me_5, 2.3330 (4), 1.991 (1) and 1.716 (2) Å; Iwata *et al.*, 2003}. The N1—Fe1—Si1 angle [88.91 (7)°] seems to show no steric repulsion between a 3,5-dimethylpyridine and an $SiEt_3$ ligand.

Experimental

A benzene solution (20 ml) containing Cp$(CO)_2$Fe($SiEt_3$) (0.96 mmol, 280 mg) and 3,5-dimethylpyridine (0.96 mmol, 0.11 ml) was subjected to irradiation with a 400 W medium pressure mercury arc lamp for 16 h, the CO generated being removed every few hours. The removal of volatile materials under reduced pressure led to the formation of a dark-red oil, which was dissolved in hexane (2 ml). After the hexane solution had been cooled at 233 K, the resulting dark-red powder was filtered off and dried *in vacuo* to give (I) (yield 0.75 mmol, 278 mg, 78%). Red crystals of (I) suitable for the X-ray diffraction study were obtained over a few days from a hexane solution at 253 K.

Crystal data

$[Fe(C_5H_5)(C_6H_{15}Si)(C_7H_9N)(CO)]$
$M_r = 371.37$
Monoclinic, $P2_1/c$
$a = 7.9025\ (5)$ Å
$b = 11.8868\ (8)$ Å
$c = 20.5798\ (16)$ Å
$\beta = 97.079\ (4)^\circ$
$V = 1918.4\ (2)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.85$ mm^{-1}
$T = 203\ (2)$ K
0.25 × 0.08 × 0.05 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min} = 0.815$, $T_{\max} = 0.959$
14867 measured reflections
4356 independent reflections
4022 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.147$
$S = 1.00$
4356 reflections
260 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{\max} = 0.68$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—C1	2.120 (16)	Fe1—C4*B*	2.155 (17)
Fe1—C1*B*	2.12 (3)	Fe1—C5	2.107 (7)
Fe1—C2	2.10 (2)	Fe1—C5*B*	2.18 (2)
Fe1—C2*B*	2.04 (4)	Fe1—C12	1.722 (3)
Fe1—C3	2.102 (9)	Fe1—N1	1.982 (2)
Fe1—C3*B*	2.068 (15)	Fe1—Si1	2.3341 (9)
Fe1—C4	2.111 (9)	O1—C12	1.164 (4)
C12—Fe1—N1	96.84 (12)	N1—Fe1—Si1	88.91 (7)
C12—Fe1—Si1	84.43 (11)		

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.97 (CH_3), 0.98 (CH_2) or 0.94 Å (CH) and $U_{\rm iso}(H) = 1.2U_{\rm eq}$(parent atom) (for CH and CH_2) or $1.5U_{\rm eq}$(parent atom) (for CH_3).

Data collection: *CrystalClear* (Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

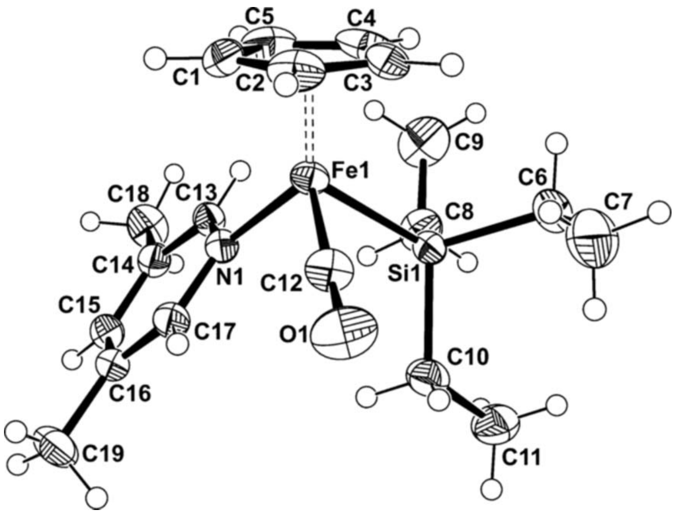


Figure 1
The molecular structure of the title compound, (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids. Only the major disorder component of the Cp atoms is shown.

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Acta Cryst. (2007). E**63**, m1312–m1313 Itazaki *et al.* • $[Fe(C_5H_5)(C_6H_{15}Si)(C_7H_9N)(CO)]$ **m1313**