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Carbonyl(η^5-cyclopentadienyl)(isocyanotriphenylborato-κC)(triphenylphosphine-κP)iron(II)

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In the title compound, $[Fe(C_5H_5)(C_{19}H_{15}BN)(C_{18}H_{15}P)(CO)]$, the Fe^{II} atom is coordinated by a carbonyl group, a triphenylphosphine ligand, an isocyanotriphenylborate ligand and a cyclopentadienyl ligand in a piano-stool structure. Two crystallographically independent molecules exist in the asymmetric unit; the Fe—CN, C≡N and CN—B bond distances are 1.866 (3), 1.157 (3) and 1.584 (4) Å, respectively, for one molecule, and 1.879 (3), 1.157 (3) and 1.587 (4) Å, respectively, for the other.

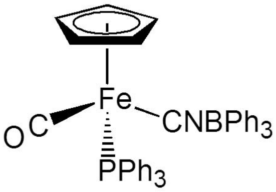
Key indicators

Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.005 Å
R factor = 0.070
wR factor = 0.116
Data-to-parameter ratio = 18.2

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

Comment

In the title compound, $[FeCp(CNBPh_3)(CO)(PPh_3)]$ (Cp is cyclopentadienyl), (I), the Fe^{II} atom is coordinated by a CO group, a triphenylphosphine ligand, an isocyanotriphenylborate ligand and a cyclopentadienyl (Cp) ring in an η^5 fashion, resulting in a typical piano-stool structure (Fig. 1). Only three crystal structures have been reported to date for iron–isocyanotriphenylborate complexes, namely $[Fe(Me)(CO)_2(PMe_3)_2(CNBPh_3)]$ (Ginderow, 1980), $[Fe(TIM)(CNBPh_3)_2]$ (TIM is 2,3,9,10-tetramethyl-1,4,8,11-tetraazacyclotetradeca-1,3,8,10-tetraene; Amrhein *et al.*, 1996) and *trans*-$[FeH(CNBPh_3)(dppe)_2]$ (dppe is diphenylphosphinoethane; Almeida *et al.*, 1999).



(I)

The Fe—CN distance [1.866 (3) Å] of the Fe1 molecule in (I) is the shortest among the reported structures; this distance is 1.910 (3) Å in $[Fe(Me)(CO)_2(PMe_3)_2(CNBPh_3)]$ (Ginderow, 1980), 1.904 (3) Å in $[Fe(TIM)(CNBPh_3)_2]$ (Amrhein *et al.*, 1996) and 1.914 (3) Å in *trans*-$[FeH(CNBPh_3)(dppe)_2]$ (Almeida *et al.*, 1999). The Fe—C—N—B geometric parameters of the Fe1 and Fe2 molecules in (I) [N—B = 1.584 (4) and 1.587 (4) Å, Fe—C—N = 173.9 (3) and 174.6 (3)°, and C—N—B = 173.8 (3) and 177.0 (3)°] are similar to those in the previously reported complexes.

Experimental

$[FeCp(CN)(CO)(PPh_3)]$ was prepared according to the literature method of Faller & Johnson (1975). To a solution of [FeCp(CN)-

(CO)(PPh$_3$)] (100 mg, 0.23 mmol) in CH$_2$Cl$_2$ (5 ml) was added trifluoromethanesulfonic acid (0.1 ml, 1.61 mmol), and the mixture was stirred for 1 h at 195 K. The solvent was then removed under reduced pressure to give a yellow oil. Diethyl ether (3 ml) and NaBPh$_4$ (700 mg, 2.05 mmol) were added to the oil. Stirring the resulting yellow solution for 10 min at room temperature caused the formation of a yellow solid. Volatile materials were removed under reduced pressure and the solid was washed repeatedly with EtOH and dried *in vacuo* to give [FeCp(CNBPh$_3$)(CO)(PPh$_3$)], (I), as a yellow powder (140 mg, 0.21 mmol, 92%). Single crystals of (I) were obtained by solvent diffusion at 253 K over a few days from an acetone layer containing (I) and an overlayer of EtOH. Spectroscopic analysis: IR (CH$_2$Cl$_2$): $\nu_{\rm CO}$ 1985 cm^{-1}, $\nu_{\rm CN}$ 2166 cm^{-1}; ^{1}H NMR (400 MHz, CDCl$_3$, δ, p.p.m.): 4.61 (*s*, Cp, 5H), 7.01–7.42 (*m*, Ph, 30H); ^{31}P NMR (161.7 MHz, CDCl$_3$, δ, p.p.m.): 69.0 (*s*).

Crystal data

[Fe(C$_5$H$_5$)(C$_{19}$H$_{15}$BN)(C$_{18}$H$_{15}$P)-(CO)]
M_r = 679.39
Monoclinic, $P2_1/c$
a = 15.1206 (6) Å
b = 18.5827 (8) Å
c = 24.890 (1) Å
β = 90.033 (2)°
V = 6993.7 (5) Å^3
Z = 8

D_x = 1.290 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 13 416 reflections
θ = 4.0–27.5°
μ = 0.51 mm^{-1}
T = 203.2 K
Prism, yellow
0.23 × 0.08 × 0.05 mm

Data collection

Rigaku/MSC Mercury CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\rm min}$ = 0.755, $T_{\rm max}$ = 0.975
54 667 measured reflections

15 701 independent reflections
11415 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.051
$\theta_{\rm max}$ = 27.5°
$h = -19 \rightarrow 14$
$k = -16 \rightarrow 24$
$l = -29 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.117
S = 1.16
15 701 reflections
865 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0137P)^2 + 4.3115P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.31 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—C1	2.086 (4)	Fe2—C47	2.097 (3)
Fe1—C2	2.099 (4)	Fe2—C48	2.099 (3)
Fe1—C3	2.108 (3)	Fe2—C49	1.752 (3)
Fe1—C4	2.103 (3)	Fe2—C50	1.879 (3)
Fe1—C5	2.087 (3)	Fe2—P2	2.2264 (9)
Fe1—C6	1.750 (4)	O1—C6	1.148 (4)
Fe1—C7	1.866 (3)	O2—C49	1.148 (4)
Fe1—P1	2.2248 (9)	N1—C7	1.157 (3)
Fe2—C44	2.108 (3)	N1—B1	1.584 (4)
Fe2—C45	2.109 (3)	N2—C50	1.157 (3)
Fe2—C46	2.087 (3)	N2—B2	1.587 (4)
C6—Fe1—C7	92.49 (14)	C50—Fe2—P2	94.70 (9)
C6—Fe1—P1	93.02 (12)	C7—N1—B1	173.8 (3)
C7—Fe1—P1	94.06 (9)	C50—N2—B2	177.0 (3)
C49—Fe2—C50	91.57 (13)	Fe1—C7—N1	173.9 (3)
C49—Fe2—P2	90.08 (10)	Fe2—C50—N2	174.6 (3)

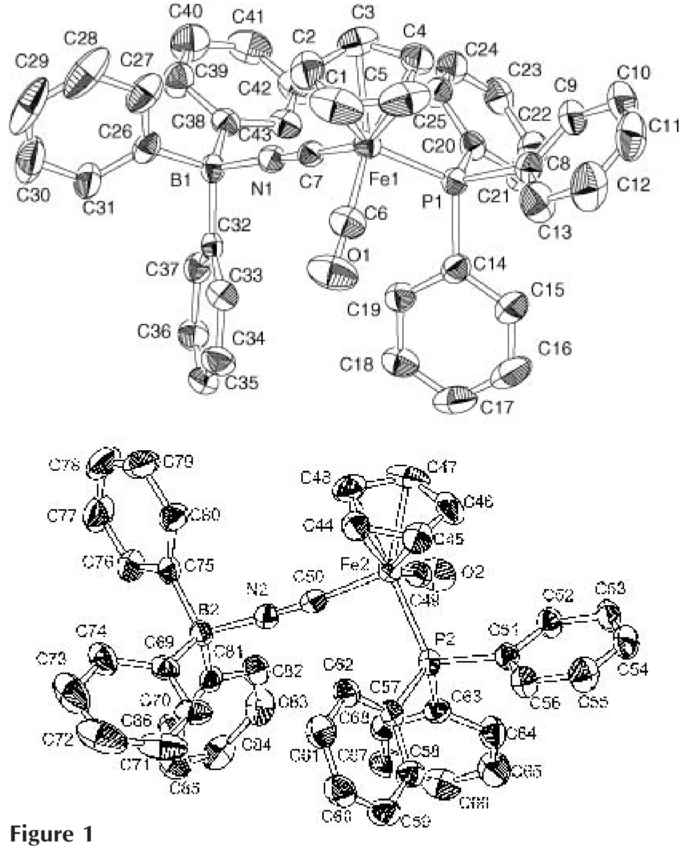


Figure 1
A drawing of both Fe molecule of [FeCp(CNBPh$_3$)(CO)(PPh$_3$)], (I), showing the atom-labelling schemes. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

H atoms were refined using a riding model, with C—H = 0.95 Å and with 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*.

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