A homoleptic (aryl isocyanide)iron(0) dimer. X-ray structure determination of nonakis(phenyl isocyanide)diiron

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were carried out under a nitrogen atmosphere. Reaction conditions and results are summarized in Table I. Hydroboration of 2-methyl-1-buten-3-yne (**1a**) with catecholborane in the presence of palladium catalyst generated in situ by mixing $Pd_2(dba)_3{\cdot}CHCl_3$ and $PPh_2(C_6F_5)$ is illustrative of the general methods for all catalytic reactions described in this study. A mixture of 8.3 mg (0.008 mmol) of $Pd_2(dba)_3{\cdot}CHCl_3$ and 11 mg (0.03 mmol) of $PPh_2(C_6F_5)$ in 1 mL of chloroform was stirred at room temperature until the solution changed from red-purple due to $Pd_2(dba)_3{\cdot}CHCl_3$ to yellow. To the catalyst solution was added successively at 25 °C 53 mg (0.80 mmol) of 2-methyl-1-buten-3-yne (**1a**) and 126 mg (1.0 mmol) of catecholborane, and the mixture was stirred at the same temperature for 30 min. Solvent was evaporated and the residue was distilled (bulb-to-bulb, bath temperature 100 °C/0.1 mmHg) to give 109 mg (73% yield) of the hydroboration product, which consisted of (3-methyl-1,2-butadienyl)-1,3,2-benzodioxaborole (**2a**) and {(*Z*)-3-methyl-1,3-butadienyl}-1,3,2-benzodioxaborole (**3a**) in a ratio of 83:17. The ratio was determined by the ^{1}H NMR spectrum. ^{1}H NMR ($CDCl_3$/TMS) data for the hydroboration products are as follows. (3-Methyl-1,2-butadienyl)-1,3,2-benzodioxaborole (**2a**): δ 1.79 (d, *J* = 3.4 Hz, 6 H), 5.18 (heptet, *J* = 3.4 Hz, 1 H), 7.01–7.13 (m, 2 H), 7.16–7.26 (m, 2 H). {(*Z*)-3-Methyl-1,3-butadienyl}-1,3,2-benzodioxaborole (**3a**): δ 1.97 (s, 3 H), 5.17 (s, 1 H), 5.20 (s, 1 H), 5.71 (d, *J* = 14.7 Hz, 1 H), 7.00 (d, *J* = 14.7 Hz, 1 H), 7.01–7.13 (m, 2 H), 7.16–7.26 (m, 2 H). {(*E*)-3-Methyl-1,3-butadienyl}-1,3,2-benzodioxaborole (**4a**): δ 1.94 (s, 3 H), 5.30 (s, 2 H), 5.87 (d, *J* = 18.1 Hz, 1 H), 7.01–7.13 (m, 2 H), 7.16–7.26 (m, 2 H), 7.46 (d, *J* = 18.1 Hz, 1 H). (3-Pentyl-1,2-butadienyl)-1,3,2-benzodioxaborole (**2b**): δ 0.88 (t, *J* = 7.2 Hz, 3 H), 1.20–1.60 (m, 6 H), 1.77 (d, *J* = 3.5 Hz, 3 H), 2.00–2.10 (m, 2 H), 5.23 (sextet, *J* = 3.5 Hz, 1 H), 7.00–7.12 (m, 2 H), 7.15–7.28 (m, 2 H). {(*Z*)-3-Pentyl-1,3-butadienyl}-1,3,2-benzodioxaborole (**3b**): δ 0.88 (t, *J* = 7.2 Hz, 3 H), 1.20–1.60 (m, 6 H), 2.28 (t, *J* = 5.7 Hz, 2 H), 5.08 (s, 1 H), 5.20 (s, 1 H), 5.71 (d, *J* = 14.7 Hz, 1 H), 6.96 (d, *J* = 14.7 Hz, 1 H), 7.00–7.12 (m, 2 H), 7.15–7.28 (m, 2 H). {(*E*)-3-Pentyl-1,3-butadienyl}-1,3,2-benzodioxaborole (**4b**): δ 0.88 (t, *J* = 7.2 Hz, 3 H), 1.20–1.60 (m, 6 H), 2.31 (t, *J* = 6.2 Hz, 2 H), 5.28 (s, 1 H), 5.32 (s, 1 H), 5.93 (d, *J* = 18.5 Hz, 1 H), 7.00–7.12 (m, 2 H), 7.15–7.28 (m, 2 H), 7.41 (d, *J* = 18.5 Hz, 1 H). (3-*tert*-Butyl-1,2-butadienyl)-1,3,2-benzodioxaborole (**2c**): δ 1.13 (s, 9 H), 1.78 (d, *J* = 3.3 Hz, 3 H), 5.22 (q, *J* = 3.3 Hz, 1 H), 7.01–7.13 (m, 2 H), 7.15–7.27 (m, 2 H). {(*Z*)-3-*tert*-Butyl-1,3-butadienyl}-1,3,2-benzodioxaborole (**3c**): δ 1.16 (s, 9 H), 4.93 (s, 1 H), 5.00 (s, 1 H), 5.74 (d, *J* = 14.1 Hz, 1 H), 7.00 (d, *J* = 14.1 Hz, 1 H), 7.01–7.13 (m, 2 H), 7.15–7.27 (m, 2 H). {(*E*)-3-*tert*-Butyl-1,3-butadienyl}-1,3,2-benzodioxaborole (**4c**): δ 1.16 (s, 9 H), 5.03 (s, 1 H), 5.35 (s, 1 H), 6.18 (d, *J* = 18.1 Hz, 1 H), 7.01–7.13 (m, 2 H), 7.15–7.27 (m, 2 H), 7.52 (d, *J* = 18.1 Hz, 1 H). {3-(Trimethylsilyl)-1,2-butadienyl}-1,3,2-benzodioxaborole (**2d**): δ 0.16 (s, 9 H), 1.79 (d, *J* = 3.3 Hz, 3 H), 4.81 (q, *J* = 3.3 Hz, 1 H), 7.01–7.12 (m, 2 H), 7.15–7.29 (m, 2 H). Analytical data for the products are as follows. Anal. Calcd for $C_{11}H_{11}BO_2$ [**2a** (a mixture of isomers, **2a**, **3a**, and **4a**)]: C, 71.03; H, 5.96. Found: C, 70.78; H, 5.90. Anal. Calcd for $C_{15}H_{19}BO_2$ [**2b** (a mixture of isomers, **2b**, **3b**, and **4b**)]: C, 74.41; H, 7.91. Found: C, 74.11; H, 7.95. For allenylboranes **2c** and **2d**, correct analyses could not be obtained due to the difficulty in purification.

Reaction of Allenylboranes with Benzaldehyde. The reaction mixture, which results from the catalytic hydroboration of **1a** (0.8 mmol) in chloroform described above and includes allenylborane **2a**, was used for the reaction with benzaldehyde without isolation of **2a**. The chloroform solution was cooled with a dry ice/acetone bath, and 119 mg (1.1 mmol) of benzaldehyde was added. The mixture was allowed to warm to room temperature under stirring. Water was added, and the mixture was extracted with ether. The organic layer was dried over $MgSO_4$ and was concentrated in vacuo. The residue was chromatographed on silica gel (hexane/ether = 10/1) to give 88 mg (0.51 mmol) of homopropargyl alcohol **7a** in 63% yield: ^{1}H NMR ($CDCl_3$/TMS) δ 1.10 (s, 3 H), 1.27 (s, 3 H), 2.24 (s, 1 H), 2.42 (bs, 1 H), 4.49 (s, 1 H), 7.2–7.5 (m, 5 H). Reaction of the chloroform solution containing allenylborane **2b**, which was obtained from 79 mg (0.64 mmol) of **1b**, with benzaldehyde in a similar manner gave 56 mg (0.24 mmol) of **7b** as a 1:1 mixture of syn and anti isomers in 38% yield: ^{1}H NMR ($CDCl_3$/TMS) for diastereomer A δ 0.87 (t, *J* = 7.1 Hz, 3 H), 1.1–1.7 (m, 8 H), 1.23 (s, 3 H), 2.25 (s, 1 H), 2.34 (d, *J* = 4.0 Hz, 1 H), 4.56 (d, *J* = 4.0 Hz, 1 H), 7.2–7.5 (m, 5 H); ^{1}H NMR ($CDCl_3$/TMS) for diastereomer B δ 0.90 (t, *J* = 7.1 Hz, 3 H), 1.04 (s, 3 H), 1.1–1.7 (m, 8 H), 2.29 (s, 1 H), 2.46 (d, *J* = 3.7 Hz, 1 H), 4.55 (d, *J* = 3.7 Hz, 1 H), 7.2–7.5 (m, 5 H). Anal. Calcd for $C_{16}H_{22}O$: C, 83.43; H, 9.63. Found: C, 83.21; H, 9.66.

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Supplementary Material Available: Figures of ^{1}H NMR spectra of hydroboration products **2a–d**, **3a–c**, and **4a–c** (5 pages). Ordering information is given on any current masthead page.

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A Homoleptic (Aryl Isocyanide)iron(0) Dimer. X-ray Structure Determination of Nonakis(phenyl isocyanide)diiron

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Summary: Nonakis(phenyl isocyanide)diiron was prepared by sodium amalgam reduction of either *cis*- or *trans*-$[FeI_2(CNPh)_4]$. The new complex was characterized spectroscopically and by single-crystal X-ray analysis. Crystal data: monoclinic, space group $P2_1/n$, a = 12.692 (5) Å, b = 27.086 (8) Å, c = 15.735 (3) Å, β = 92.90 (2)°, V = 5402 (3) Å^3, Z = 4, R = 0.032.

Homoleptic metal isocyanide complexes form an important group of species that allow a comparison with their carbonyl analogs. Moreover, they offer the possibility of controlling the electronic and steric requirements of the complex by changing the substituents on the nitrogen atom of the isocyanide ligands. A few examples of homoleptic isocyanide metal(0) dimers are known,[1] but only for the alkyl isocyanide derivatives $[Fe_2(CNEt)_9]$[2] and $[Co_2(CNBu^t)_8]$[3] has an X-ray structural determination been

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Table I. Selected Fractional Positional and Thermal Parameters (Esd's in Parentheses)

atom	x	y	z	$100U_{eq}$,[a] Å^2
Fe1	0.46241 (3)	0.16855 (2)	0.84316 (3)	4.35 (1)
Fe2	0.40018 (3)	0.13104 (2)	0.70748 (3)	3.92 (1)
C1	0.3360 (2)	0.1861 (1)	0.7723 (2)	4.3 (1)
N1	0.2657 (2)	0.2178 (1)	0.7700 (2)	5.5 (1)
C11	0.1805 (3)	0.2248 (1)	0.7099 (2)	5.2 (1)
C2	0.4341 (2)	0.0973 (1)	0.8162 (2)	4.6 (1)
N2	0.4443 (2)	0.0585 (1)	0.8566 (2)	5.6 (1)
C21	0.4289 (3)	0.0084 (1)	0.8323 (2)	5.7 (1)
C3	0.5287 (2)	0.1727 (1)	0.7334 (2)	4.3 (1)
N3	0.6030 (2)	0.1935 (1)	0.7022 (2)	5.8 (1)
C31	0.6404 (2)	0.1960 (1)	0.6190 (2)	5.5 (1)
C4	0.5901 (3)	0.1481 (1)	0.8932 (2)	5.4 (1)
N4	0.6714 (2)	0.1350 (1)	0.9235 (2)	7.0 (1)
C41	0.7656 (3)	0.1176 (2)	0.9599 (3)	7.8 (2)
C5	0.3819 (3)	0.1569 (1)	0.9354 (2)	5.0 (1)
N5	0.3312 (2)	0.1464 (1)	0.9926 (2)	6.4 (1)
C51	0.2802 (3)	0.1226 (1)	1.0578 (2)	5.9 (1)
C6	0.4793 (2)	0.2349 (1)	0.8580 (2)	4.8 (1)
N6	0.4874 (2)	0.2779 (1)	0.8659 (2)	6.1 (1)
C61	0.4748 (3)	0.3278 (1)	0.8487 (2)	5.6 (1)
C7	0.2704 (3)	0.1017 (1)	0.7007 (2)	4.7 (1)
N7	0.1862 (2)	0.0835 (1)	0.6931 (2)	6.4 (1)
C71	0.0810 (3)	0.0705 (1)	0.6998 (2)	5.6 (1)
C8	0.4845 (2)	0.0844 (1)	0.6586 (2)	4.6 (1)
N8	0.5376 (2)	0.0551 (1)	0.6276 (2)	6.2 (1)
C81	0.5980 (2)	0.0180 (1)	0.5919 (2)	5.5 (1)
C9	0.3770 (2)	0.1640 (1)	0.6052 (2)	4.5 (1)
N9	0.3625 (2)	0.1804 (1)	0.5372 (2)	5.9 (1)
C91	0.3542 (3)	0.1982 (1)	0.4534 (2)	5.0 (1)

[a] $U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_ia_ja_i{}^*a_j{}^*$.

carried out, so our study on [$Fe_2(CNPh)_9$] (1) represents the first X-ray structural analysis of an homoleptic aryl isocyanide metal(0) dimer.

Whereas [$Fe_2(CNEt)_9$] had been synthetized by UV irradiation of [$Fe(CNEt)_5$][2] (following a procedure comparable to that used for the synthesis of [$Fe_2(CO)_9$][4]), we have instead prepared 1 by direct Na–Hg reduction of [$FeI_2(CNPh)_4$].

Results and Discussion

When a tetrahydrofuran solution of either *cis*- or *trans*-[$FeI_2(CNPh)_4$] was treated with an excess of sodium amalgam at room temperature, a very air-sensitive red solution of complex 1 was readily obtained. The IR spectrum of this solution contains bands that can be assigned both to terminal (2092 (s), 2058 (s), 2002 (m) cm^{-1}) and bridging (1674 (s) cm^{-1}) isocyanide ligands, in agreement with the solid-state structure determined by the X-ray study.

The synthesis of 1 from [$FeI_2(CNPh)_4$] obviously requires some decomposition of the reaction mixture in order to provide the extra PhNC ligand needed. Surprisingly, however, when the reaction was carried out in the presence of 0.5 equiv of free isocyanide the yield in complex 1 did not improve. Instead, the formation of perceptible amounts of the mononuclear compound [$Fe(CNPh)_5$][5] was observed.

Crystals of 1 suitable for X-ray diffraction were obtained by recrystallization from diethyl ether and sealed in glass capillaries under nitrogen. Crystallographic data are summarized in Tables I and II. The molecular structure illustrated in Figure 1 shows that 1 has six terminal and three bridging isocyanide ligands with a nearly octahedral coordination of isocyanides around each iron atom. The Fe–Fe distance of 2.458 (1) Å is nearly the same than that found in [$Fe_2(CNEt)_9$][2] (2.461 (2) Å) and shorter than that corresponding to [$Fe_2(CO)_9$][6] (2.531 (1) Å).

Table II. Selected Bond Lengths (Å) and Angles (deg) for $Fe_2(CNPh)_9$

		Bond Lengths			
Fe1–Fe2	2.458 (1)	Fe1–C1	1.966 (3)	Fe1–C2	2.005 (3)
Fe1–C3	1.962 (3)	Fe1–C4	1.851 (3)	Fe1–C5	1.844 (3)
Fe1–C6	1.822 (4)	Fe2–C1	2.003 (3)	Fe2–C2	1.967 (3)
Fe2–C3	2.008 (3)	Fe2–C7	1.828 (3)	Fe2–C8	1.849 (3)
Fe2–C9	1.851 (3)	C1–N1	1.237 (3)	C2–N2	1.231 (4)
C3–N3	1.223 (4)	C4–N4	1.170 (4)	C5–N5	1.167 (4)
C6–N6	1.175 (4)	C7–N7	1.176 (4)	C8–N8	1.164 (4)
C9–N9	1.165 (4)				

	Bond Angles		
C2–Fe1–C1	88.8 (1)	C3–Fe1–C1	82.0 (1)
C3–Fe1–C2	87.1 (1)	C4–Fe1–C2	86.9 (1)
C4–Fe1–C3	89.2 (1)	C5–Fe1–C1	91.1 (1)
C5–Fe1–C2	84.3 (1)	C5–Fe1–C4	97.0 (1)
C6–Fe1–C1	85.5 (1)	C6–Fe1–C3	90.2 (1)
C6–Fe1–C4	98.4 (1)	C6–Fe1–C5	97.7 (1)
C2–Fe2–C1	88.9 (1)	C3–Fe2–C1	80.0 (1)
C3–Fe2–C2	86.9 (1)	C7–Fe2–C1	88.0 (1)
C7–Fe2–C2	90.3 (1)	C8–Fe2–C2	86.5 (1)
C8–Fe2–C3	89.3 (1)	C8–Fe2–C7	102.5 (1)
C9–Fe2–C1	91.9 (1)	C9–Fe2–C3	89.8 (1)
C9–Fe2–C7	93.2 (1)	C9–Fe2–C8	92.2 (1)
Fe2–C1–Fe1	76.6 (1)	N1–C1–Fe1	138.7 (2)
N1–C1–Fe2	144.4 (2)	C11–N1–C1	129.7 (3)
Fe2–C2–Fe1	76.4 (1)	N2–C2–Fe1	134.3 (2)
N2–C2–Fe2	149.1 (3)	C21–N2–C2	131.7 (3)
Fe2–C3–Fe1	76.5 (1)	N3–C3–Fe1	138.9 (2)
N3–C3–Fe2	144.3 (2)	C31–N3–C3	134.0 (3)
N4–C4–Fe1	178.9 (3)	C41–N4–C4	177.6 (4)
N5–C5–Fe1	175.9 (3)	C51–N5–C5	166.2 (4)
N6–C6–Fe1	177.9 (3)	C61–N6–C6	159.6 (3)
N7–C7–Fe2	177.4 (3)	C71–N7–C7	165.7 (3)
N8–C8–Fe2	179.8 (1)	C81–N8–C8	177.0 (3)
N9–C9–Fe2	173.6 (3)	C91–N9–C9	174.6 (3)

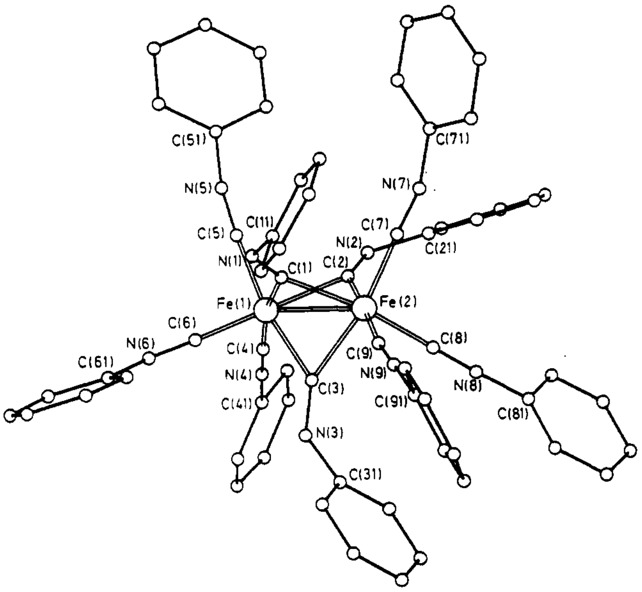

Figure 1. Pluto drawing of the molecular structure of the complex [$Fe_2(CNPh)_9$] (1) together with the atomic numbering scheme.

The bridging isocyanide ligands contain essentially sp^2-type nitrogen atoms with a CNC mean angle of 131.8 (3)°. For the terminal ligands, the angles CNC range from 159.5 (4)° in C6N6C61 to 177.7 (4)° in C4N4C41, thus

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showing the flexibility of isocyanides to accomplish the electronic and steric requirements of the molecule.

An interesting feature in the structure of 1 is the fact that the three bridging isocyanide ligands have the aryl groups directed toward the same iron atom. This is a remarkable difference with the structure of $[Fe_2(CNEt)_9]$ in which one bridging isocyanide ligand has the ethyl substituent directed toward a different iron atom than the ethyl substituents in the two remaining bridging isocyanides.[2] There is some asymmetry in the coordination of the bridging isocyanide ligands to the two iron atoms. Indeed, the distances from the bridge carbon atoms to the iron centers differ by 0.040 (3) Å (mean), with the longer bond lying syn to the phenyl group for C1 and C3 and anti to the phenyl group for C2. Other bond distances and angles are in the range expected for iron isocyanide derivatives.

^{13}C NMR spectra of 1 revealed a dynamic behavior in solution. At room temperature the resonances of the iron-bonded carbon atoms of the isocyanide ligands were not observed, probably due to a broadening effect derived from exchange between terminal and bridging ligands. However, on cooling to −60 °C, a singlet for each the terminal (182.0 ppm) and bridging (251.3 ppm) isocyanides was present in the spectrum. The apparent equivalence of the six terminal isocyanides probably implies a rapid inversion at the nitrogen in the bridging isocyanides, as it has been postulated in other cases.[2,3] This inversion could not be slowed down for 1 even after cooling to −90 °C.

Experimental Section

General Procedures. All operations were performed under an atmosphere of dry nitrogen. Phenyl isocyanide was prepared according to the method of Ugi.[7] *cis*- and *trans*-$[FeI_2(CNPh)_4]$ were obtained by a literature method.[8] The NMR spectra were recorded on a Bruker AC-300 spectrometer. The IR spectra were recorded on a Perkin-Elmer FT 1720X spectrometer.

Synthesis of Nonakis(phenyl isocyanide)diiron. To a solution of *cis*-$[FeI_2(CNPh)_4]$ (0.5 g, 0.69 mmol) in 50 mL of tetrahydrofuran was added an excess of a 1% sodium amalgam (0.07 g of sodium, 3.04 mmol). After being stirred at room temperature for 1 h, the red solution obtained was filtered and the solvent evaporated to dryness. The residue was then extracted with toluene (50 mL) and filtered. Evaporation of the solvent left a red oil, which was recrystallized from diethyl ether to give 0.23 g (72%) of red crystals. IR (THF): 2092 (s), 2058 (s), 2002 (m), 1674 (s) cm^{-1}. ^{13}C NMR (toluene-d_8 at −60 °C): δ 182.0 (N*C*Fe terminal), 251.3 (N*C*Fe bridging). Anal. Calcd for $C_{63}H_{45}N_9Fe_2$: C, 72.77; H, 4.36; N, 12.12. Found: C, 72.51; H, 4.36; N, 11.93.

When *trans*-$[FeI_2(CNPh)_4]$ was used as starting material, 1 was obtained in a similar yield by following the same procedure as for the cis isomer.

X-ray Crystallographic Analysis. Red crystals of complex 1, suitable for X-ray diffraction study were obtained by recrystallization from Et_2O. Crystal data for 1: $C_{63}H_{45}Fe_2N_9$, M_r = 1039.80, monoclinic, space group $P2_1/n$, a = 12.692 (5) Å, b = 27.086 (8) Å, c = 15.735 (3) Å, β = 92.90 (2)°, V = 5402 (3) $Å^3$, Z = 4, ρ_{calcd} = 1.28 g/cm^3, T = 293 K; crystal dimensions 0.36 × 0.30 × 0.26 mm^3; Mo Kα radiation (λ = 0.71073 Å) graphite-monochromated; 10034 reflections measured on a Enraf-Nonius CAD 4 (ω–2θ scan technique), range $0 < \theta < 25°$ and $-15 \leq h \leq 15$, $0 \leq k \leq 32$, $0 \leq l \leq 18$; 9426 unique reflections (R_{int} = 0.019, averaging double measured) and 5154 observed with $I > 3\sigma(I)$. Semiempirical[9] and empirical[10] absorption corrections were applied; μ = 5.82 cm^{-1}. The structure was solved by direct methods (SHELX86)[11] and anisotropically refined (SHELX 76 local version)[12] to a final R = 0.032, R_w = 0.032. (668 parameters and $\omega = 1/(\sigma^2(F_o) + 0.008F_o^2)$; maximum shift/error 0.011, ρ_{max} = 0.18 $e/Å^3$ ρ_{min} = −0.016 $e/Å^3$.

All calculations were made on a MicroVax-3400 computer at the Scientific Computer Center of the University of Oviedo.

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Registry No. 1, 141928-23-6; *cis*-$[FeI_2(CNPh)_4]$, 137679-82-4; *trans*-$[FeI_2(CNPh)_4]$, 137766-34-8.

Supplementary Material Available: Complete listings of bond lengths and angles, anisotropic thermal parameters, positional parameters, hydrogen atom parameters, distances and angles involving H atoms, least-squares-planes data, and principal torsion angles (19 pages). Ordering information is given on any current masthead page.

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