Bis(cyclopentadienyliron)arene Complexes: A New Route to the Synthesis and Functionalization of Polyaromatic Ethers[1]

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Received August 16, 1993⊗

A new development in the chemistry of arenes activated toward S_NAr reactions by the cyclopentadienyliron ($FeCp^+$) moiety is presented in this work. A class of diiron complexes of diphenoxybenzenes was prepared in a highly efficient and very mild fashion. Dihydroxy aromatic compounds served as dinucleophiles, allowing for the formation of the diiron complexes. This could be achieved in either a one or two step procedure. A wide variety of dinucleophiles were incorporated into this study, as well as a number of $FeCp^+$ activated arenes. It is shown that these reactions are not inhibited by bulky substituents on either the dinucleophiles or activated arenes. The diiron complexes themselves could also undergo S_NAr reactions, provided that the complexed arenes contained a chlorine substituent. This allowed for the functionalization of the complexes with species that could not be introduced directly in their syntheses. The carbon nucleophiles generated from ethyl cyanoacetate or (phenylsulfonyl)acetonitrile could be attached to the complexed ethers in this manner. The $FeCp^+$ moieties were removed easily by photolytic demetalation which allowed for the recovery of a wide range of functionalized diphenoxybenzenes. This methodology is advantageous over all those previously reported and should be a practical route to the synthesis of aromatic ethers.

Introduction

Aromatic ethers are undoubtedly one of the most important classes of organic chemistry, finding extensive application both in their pure form and as fragments of more complex molecules. A vast amount of research has focused on the synthesis of both low and high molecular weight polyaromatic ethers.[2] These types of materials are very important due to their thermal stability and resistance to radiation damage.[3] They are tough, hydrolytically stable, and creep resistant and exhibit high performance as synthetic lubricants.[4] Aromatic ether linkages also appear very frequently in naturally occurring macrocyclic peptides, most notably the vancomycin family of antibiotics.[5] It is very important to point out that there are few general routes for the synthesis of polyaromatic ethers. The Ullmann condensation reaction is a primary method, where the condensation occurs between an appropriate aryl halide and the alkali metal salt of a hydroxy aromatic compound.[6] The reaction is catalyzed by a copper salt or oxide and usually takes place at elevated temperatures (100–300 °C). Aside from these harsh conditions, this type of reaction suffers from the presence of side products and relatively poor yields. Conventional aromatic nucleophilic substitution (S_NAr) also provides a route to aromatic ethers.[7] When this methodology is applied, the starting halogenated aromatic compound should contain electron-withdrawing groups to facilitate the substitution reactions. The subsequent removal of these activating groups could result in additional and sometimes unwanted steps. Furthermore, Yamamura and his co-workers have also used thallium(III) nitrate in coupling reactions to produce phenyl ether linkages leading to the synthesis of antifungal antibiotics.[8]

Nucleophilic aromatic substitution reactions may also be achieved using metal activated halo-aromatic complexes. Various metal moieties such as $Cr(CO)_3$, $Mn(CO)_3^+$, $FeCp^+$, and $RuCp^+$ have been used in activating arene compounds toward nucleophilic substitution reactions.[9] The use of the chromium tricarbonyl moiety in activating the halobenzene ring toward S_NAr with some phenoxides and alkoxides provided a route to the synthesis of some aryl ethers.[10] Recently, Percec and Okita reported the S_NAr reaction of (1,4-dichlorobenzene)$Cr(CO)_3$ with mono- and diphenoxides.[11] They have observed that the

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⊗ Abstract published in *Advance ACS Abstracts,* December 1, 1993.

[1] Presented in part at the 33rd National Organic Chemistry Symposium, Bozeman, MT, June 13–17, 1993; Abstract No. A-6.

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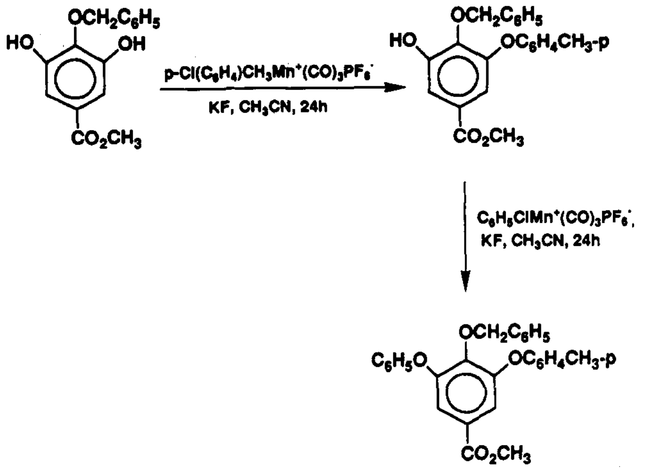
Scheme 1



substitution of the first chlorine was extremely fast while the second chlorine substitution was quite slow due to the incorporation of the phenoxy group. Moreover, a mixture of products was obtained with some of these nucleophiles. Other approaches to the synthesis of aryl ethers using $Mn(CO)_3^+$, $FeCp^+$, and $RuCp^+$ have also been developed.[12] Di- and triaryl ethers were synthesized on the basis of the structure of the starting arene complexes and the oxygen containing nucleophiles. The sequential arylation of dihydroxybenzenes using the $Mn(CO)_3^+$ moiety has been investigated by Pearson and his group,[12a–c] according to Scheme 1. The $FeCp^+$ system has allowed for the synthesis of mono- and dietheric linkages.[12e,f] The activation of the aromatic system using $RuCp^+$ toward the synthesis of polyaromatic ether has been briefly described in a communication by Segal in 1985.[13]

This paper is a continuation of our studies into the reactivity of (arene)$CpFe^+$ complexes. Here, we describe the synthesis, characterization, and functionalization of a variety of aromatic ether compounds using diiron complexes.

Results and Discussion

Recently, we have been interested in the synthesis and reactivity of bis(cyclopentadienyliron)arene complexes due to their wide range of applications. In a previous communication we have reported our initial results in the syntheses of some complexes of polyaromatic ethers.[14] It has been our intent to demonstrate the synthetic utility of this new methodology in the synthesis of aromatic ethers, by preparing various classes of compounds difficult to synthesize by existing techniques. We proceed to develop these reactions with a wide range of dihydroxy aromatic nucleophiles.

Scheme 2

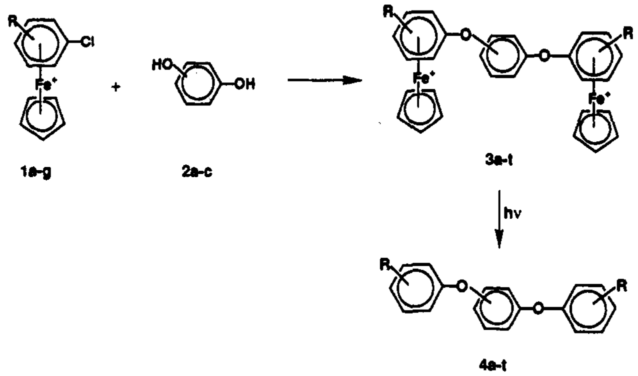


Table 1. Reaction of (substituted chlorobenzene)$FeCp^+$ (1a–g) with Isomeric Dihydroxybenzene (2a–c) Followed by Photolytic Demetalation

		diaryl ether, yield (%)		
R	mono-iron	1,2-dihydroxy-benzene	1,3-dihydroxy-benzene	1,4-dihydroxy-benzene
H	**1a**	**3a** (79), **4a** (95)	**3b** (95), **4b** (94)	**3c** (95), **4c** (82)
2-CH_3	**1b**	**3d** (82), **4d** (90)	**3e** (88), **4e** (72)	**3f** (89), **4f** (85)
3-CH_3	**1c**	**3g** (86), **4g** (85)	**3h** (90), **4h** (91)	**3i** (88), **4i** (80)
4-CH_3	**1d**	**3j** (84), **4j** (96)	**3k** (88), **4k** (88)	**3l** (85), **4l** (78)
2-Cl	**1e**		**3m** (94), **4m** (82)	**3n** (90), **4n** (84)
3-Cl	**1f**	**3o** (83), **4o** (87)	**3p** (85), **4p** (87)	**3q** (90), **4q** (93)
4-Cl	**1g**	**3r** (83), **4r** (98)	**3s** (92), **4s** (76)	**3t** (88), **4t** (76)

Isomeric bis(η^6-aryloxy-η^5-cyclopentadienyliron)benzene hexafluorophosphates **3a–t** were prepared under very mild conditions. The chloroarene complexes **1a–g**, isomeric dihydroxybenzenes **2a–c**, and potassium carbonate in *N,N*-dimethylformamide were stirred for 12–24 h at room temperature under a nitrogen atmosphere. All reactions proceeded smoothly to give complexes **3a–t** in yields ranging between 79 and 95% (Scheme 2, Table 1). Although these results indicate the generality of this route for the synthesis of aryl ether complexes, in the case of 1,2-dihydroxybenzene we have observed a lower reactivity, which was evident in the longer reaction time required (24 h). It is also important to notice that in the reaction of 1,2-dichlorobenzene complex **1e** with 1,2-dihydroxybenzene **2a**, the product 1,2-bis(η^6-2-chlorophenoxy-η^5-cyclopentadienyliron)benzene hexafluorophosphate was not synthesized. When the synthesis of this complex was attempted under conditions equivalent to the others, a mixture of products resulted, including η^6-dibenzodioxin-η^5-cyclopentadienyliron hexafluorophosphate, η^6-2-(2-hydroxyphenoxy)chlorobenzene-η^5-cyclopentadienyliron hexafluorophosphate, and starting material **1e**. The dibenzodioxin complex has been previously synthesized from these reagents in a 1:1 molar ratio of the starting complex **1e** and the nucleophile **2a**.[15] Even when an excess of the monoiron starting complex was used, as an attempt to prepare the monosubstituted complex, the product obtained was the dibenzodioxin complex. ^1H and ^{13}C NMR and elemental analysis were used to characterize the prepared aromatic ether complexes **3a–t**. In the ^1H NMR spectra of these complexes, a very distinctive singlet arising from the cyclopentadienyl (Cp) protons appeared in the 5.13–5.38 ppm range. This distinctiveness is also evident

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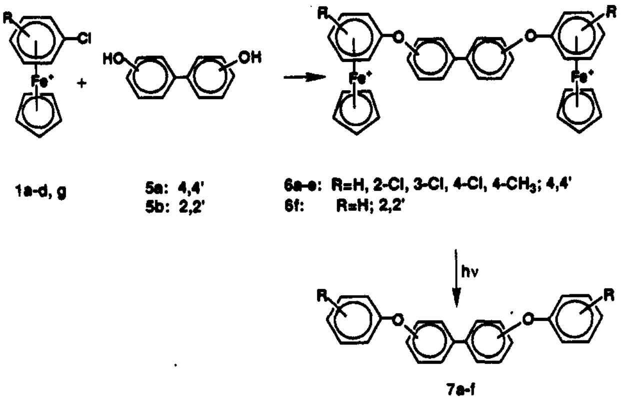
in the ^{13}C NMR, in which the Cp carbons give rise to a singlet in the 78.1–80.6 ppm range. In these diiron complexes, both Cp's are equivalent, resulting in a single resonance. Nevertheless, in some cases, the spectra of the 2- and 3-substituted complexes exhibit the presence of two Cp resonances due to the presence of two diastereoisomers of approximately equal ratio. In addition, complication of the ^1H and ^{13}C NMR spectra of the complexed arenes was present. Complex **3d** clearly shows the presence of the two diastereoisomers, as described in the Experimental Section.

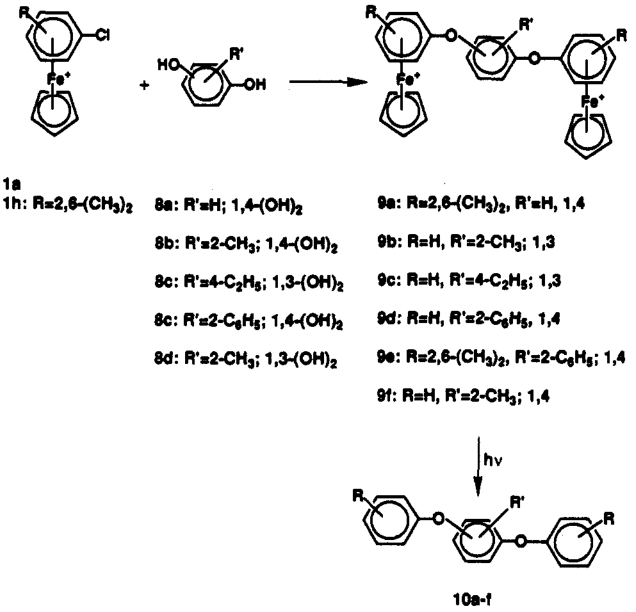
In this synthetic strategy, photolysis was used as a means to liberate the aryl ethers from their cyclopentadienyliron moieties. Solutions of these complexes were subjected to intense visible light radiation for a period of 4 h. Typically, a dichloromethane/acetonitrile solvent system was used, which proved to be very successful. A similar photolytic procedure has been applied by Nesmeyanov, in which some (arene)CpFe$^+$ complexes were demetalated to give the arene ligands, ferrocene, and an Fe(II) salt.[16] This photolytic degradation was proven to be greatly dependent on the nature of the solvent. Gill and Mann[17] have also given a detailed description of the photoinduced replacement of the arene by one six-electron or three two-electron ligands. This technique was successfully applied to obtain compounds **4a–t** in very high yield (72–98%). The identities of these compounds were confirmed by NMR, elemental analysis, mps, and MS. An obvious feature of the NMR spectra of these compounds is the disappearance of the Cp peaks as well as a downfield shift in the resonances of the decomplexed arene protons and carbons.

Recently, more involved studies have been developed to prepare a related class of molecules in which the aromatic linkage is a biphenyl unit. These include the Scholl reaction and Ni-catalyzed coupling reactions where some aromatic ethers are coupled to form the carbon–carbon bond.[18] This approach allows for the inclusion of a biphenyl unit in aromatic ether systems. However, this synthesis relies on the availability of preexisting chlorinated aromatic ethers. To achieve this goal using our unique methodology, we have carried out S$_N$Ar reactions using 2,2′- and 4,4′-dihydroxybiphenyls as nucleophiles. The reaction proceeded very smoothly under conditions applied in the case of the dihydroxybenzenes to yield complexes **6a–f**, as shown in Scheme 3. The photolysis of these products resulted in the liberation of the bis(aryloxy)biphenyl compounds (**7a–f**) as white solids in yields ranging from 75 to 96%. Our ability to synthesize these biphenyl ethers is beneficial in that it can draw from a larger pool of starting materials. In addition, it creates a more extensive number of compounds that can be coupled by Scholl and nickel-catalyzed coupling reactions.

Scheme 3



Scheme 4



To further study the flexibility of our systems, we carried out reactions with sterically crowded nucleophiles as well as monoiron complexes. Scheme 4 displays the range of such diiron complexes prepared. In such reactions we were able to prepare symmetric (**9a–c**) and unsymmetric (**9d–f**) bimetallic complexes. In these reactions, no steric inhibition was observed even with the bulky phenyl group and/or with two methyl groups in the 2,6-position to the site of substitution. Thus, we were able to prepare a variety of substituted aromatic polyether complexes with various substituents on the complexed or uncomplexed arenes in very good yield. In complexes **9d–f**, the ^1H and ^{13}C NMR showed the presence of two nonequivalent Cp resonances, as expected due to the unsymmetric nature of the complex. The ratio of these Cp's was integrated to approximately a 1:1 ratio. The photochemical degradation of these complexes produced the free aromatic ethers, **10a–f**, as summarized in the Experimental Section.

Although we were able to prepare these types of complexes in one step, a two step synthesis was also employed (Scheme 5) to prepare unsymmetrical diiron complexes. The first step in this synthesis involved the reaction of the monoiron complexes **1a,d** with an excess of 1,4-dihydroxybenzene (**2c**). The reaction produced monosubstituted complexes **11a,b**, which were then reacted with an equimolar amount of complex **1g** to produce the unsymmetric bimetallic complexes **12a,b**. Of course, this two-step synthetic procedure results in a slightly lower yield, but it allows for the synthesis of a very important class of unsymmetrical aromatic ethers. Photolysis was implemented in the usual fashion to give **13a,b**.

To further explore the versatility of this synthetic methodology, we have examined the S$_N$Ar reactions with

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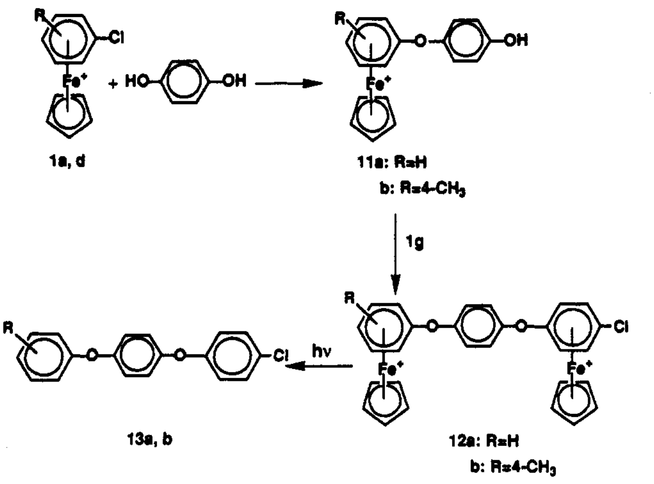
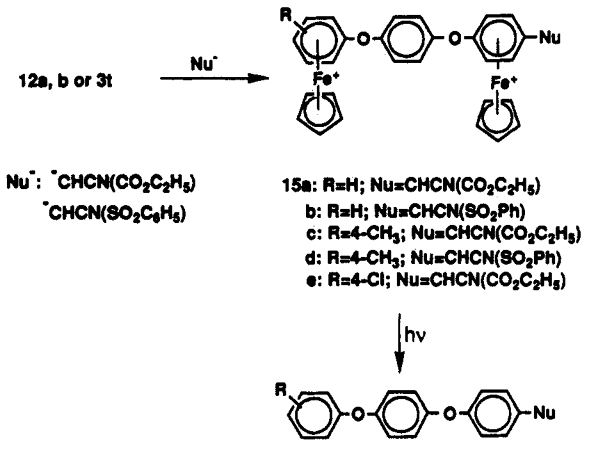
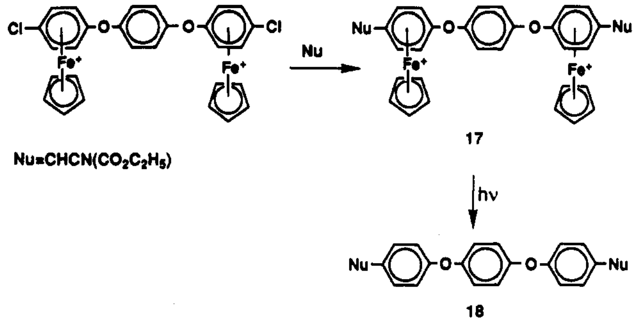
Scheme 5



Scheme 6



Scheme 7



carbon nucleophiles, due to our interest in the carbon-carbon bond formation using the CpFe$^+$ moiety.[19] Recently, we have developed a route to the synthesis of arylated (phenylsulfonyl)acetonitrile and ethyl cyanoacetates, using monoiron analogues.[20] These types of materials are valuable intermediates in the synthesis of alkanoic acid esters and some heterocyclic compounds, such as azetinones and pyrimidines.[21] It has also been established that phenoxy–alkanoic acids exhibit antiinflammatory properties.[22] In this paper we have used (phenylsulfonyl)acetonitrile and ethyl cyanoacetate as nucleophiles to functionalize the bimetallic ether complexes. As shown in Scheme 6, the reaction of complexes **12a** or **12b** with these two nucleophiles led to the formation of the desired products **15a–e** in very high yield. The reaction conditions used in this study were identical to those employed in our recent investigation of the monoiron complexes.[19a]

Multiple functionalization of the diiron complexes could also be achieved through double nucleophilic substitution, where the starting complex contains a chlorine atom on each complexed aromatic ring. For example, complex **3t** was reacted with 2 equiv of ethyl cyanoacetate to yield the desired disubstituted product **17** in 93% yield (Scheme 7). This complex could also be achieved using a stepwise reaction in which complex **3t** and the nucleophile were mixed in an equimolar ratio in the presence of potassium carbonate to give the monosubstituted product **15e**. This product could then undergo S$_N$Ar reaction with another equimolar amount of the nucleophile to produce the disubstituted complex **17**. The liberation of the functionalized aromatic ethers was carried out in the usual fashion. These types of reactions demonstrate the ability to incorporate aromatic ethers into more extensive complex molecules offering a wider range of substituted phenoxy–alkanoic acid esters and heterocyclic precursors.

Conclusion

Most techniques in the synthesis of aromatic ethers suffer from either low yields or restrictions in the range of ethers they can produce. The Ullmann condensation reaction, aside from the harsh conditions it employs, often results in low yields, even with simple substituents such as Cl, CH$_3$, OH, and NO$_2$. Conventional activation of aromatic halides with nitro group substituents toward S$_N$Ar reactions does not provide a practical route to the synthesis of these compounds, due to the complications arising from the removal of the nitro groups.[16a] Even though displacement of a nitro group activated by electron withdrawing groups in the ortho or para positions provides a route to the synthesis of some functionalized aromatic ethers, this method is unable to provide any meta isomers.

In this work, double nucleophilic substitution reactions with various dihydroxy aromatic compounds and a variety of cyclopentadienyliron complexes of substituted chlorobenzenes provided a new and flexible route to the synthesis of bis(cyclopentadienyliron) complexes of isomeric polyaromatic ethers. Further functionalization of this class of complexes can be promoted by the presence of the two cyclopentadienyliron moieties, as demonstrated by the incorporation of carbon nucleophiles in the present work. Photolysis provided a very efficient route for the liberation of the ethers and functionalized ethers. The ease in which functionalization of polyaromatic ethers occurs in our system has a distinct advantage over any of the conventional techniques. Other methods involving metal complexes suffer from the high cost of RuCp$^+$ and

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$Mn(CO)_3^+$ and the inability to prepare some dichloroarene–manganese tricarbonyl complexes efficiently.[9c,12b] The iron system provides the most general, least expensive, and least toxic route to the synthesis of this class of compounds.

Experimental Section

^1H and ^{13}C NMR spectra were recorded at 200 and 50 MHz, respectively, on a Gemini 200 NMR spectrometer, with chemical shifts calculated from the solvent signals. Coupling constants were calculated in Hz. MS spectra were obtained on a Hewlett-Packard 5970 Series mass selective detector as well as a VG 7070E-HF tandem mass spectrometer. Signal positions are given in *m/z* units. IR spectra were recorded on Perkin-Elmer Model 781 spectrophotometer. Melting points were measured in a capillary using a Mel-Temp II and are uncorrected. Elemental analyses were performed at the University of Saskatchewan.

Starting Materials. Starting complexes **1a–h** were prepared by way of previously reported ligand exchange reactions.[23] Anhydrous aluminum chloride, aluminum powder, ferrocene, ethyl cyanoacetate, (phenylsulfonyl)acetonitrile, ammonium hexafluorophosphate, chloroarenes, and the oxygen containing nucleophiles are commercially available and were used without further purification. All solvents (reagent grade) were used without purification, with the exception of freshly distilled tetrahydrofuran. Silica gel 60–100 mesh, was used in the column chromatographic purification of the liberated arenes.

Nucleophilic Substitution Reactions. 1. Description of Experimental Procedure for Complexes 3a–t, 6a–f, and 9a–f. Typically, 1 mmol of the appropriate (η^6-arene-η^5-cyclopentadienyliron) hexafluorophosphate complex was combined with 0.5 mmol of a dihydroxy aromatic nucleophile and added to a 50-mL round bottom flask containing excess K_2CO_3 and 10 mL of DMF. The resulting green-gold solution was stirred under an N_2 atmosphere for a variable amount of time, changing to a yellow-brown color. A standard workup procedure was followed, in which the reaction mixture was filtered through a sintered glass crucible into a 10% (v/v) HCl solution, causing a granular precipitate. Acetone washings were added to the filtrate, causing dissolution of the product. This solution was then concentrated by evaporation of the acetone under reduced pressure, and the desired diiron complex precipitated as a yellow granular solid by the inclusion of an aqueous NH_4PF_6 solution. At this point the product was recovered by filtration and washed with several portions of cold water. After drying for several hours under vacuum, the product was washed with small amounts of diethyl ether, and further dried. The resulting products (fine yellow powders) did not require additional purification. Reaction times, yields, and analytical data for these complexes have been summarized below.

1,2-Bis([η^6-phenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3a). A reaction time of 24 h gave 0.314 g (79%). Anal. Calcd for $C_{28}H_{24}F_{12}Fe_2O_2P_2$: C, 42.35; H, 3.05. Found: C, 42.43; H, 3.13. $\delta_H(CD_3COCD_3)$: 5.22 (s, 10H, Cp), 6.32 (t, 2H, *J* = 5.6 Hz, complexed ArH), 6.40–6.48 (m, 8H, complexed ArH), 7.58–7.74 (m, 4H, ArH). $\delta_C(CD_3COCD_3)$: 78.00 (4C, complexed ArC), 78.35 (10C, Cp), 86.40 (2C, complexed ArC), 87.94 (4C, complexed ArC), 124.21 (2C, ArC), 129.39 (2C, ArC), 133.32 (2C, quat complexed ArC), 145.30 (2C, quat ArC).

1,3-Bis([η^6-phenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3b). A reaction time of 16 h gave 0.377 g (95%). Anal. Calcd for $C_{28}H_{24}F_{12}Fe_2O_2P_2$: C, 42.35; H, 3.05. Found: C, 42.47; H, 3.15. $\delta_H(CD_3COCD_3)$: 5.24 (s, 10H, Cp), 6.29 (t, 2H, *J* = 5.6 Hz, complexed ArH), 6.42–6.49 (m, 8H, complexed ArH), 7.35–7.45 (m, 3H, ArH), 7.74 (t, 1H, *J* = 8.0 Hz, ArH). $\delta_C(CD_3COCD_3)$: 78.09 (10C, Cp), 78.22 (4C, complexed ArC), 85.95 (2C, complexed ArC), 87.71 (4C, complexed ArC), 114.26, 118.28, 133.51 (4C, ArC), 133.79 (2C, quat complexed ArC), 155.69 (2C, quat ArC).

1,4-Bis([η^6-phenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3c). A reaction time of 12 h gave 0.377 g (95%). Anal. Calcd for $C_{28}H_{24}F_{12}Fe_2O_2P_2$: C, 42.35; H, 3.05. Found: C, 42.43; H, 3.13. $\delta_H(CD_3COCD_3)$: 5.26 (s, 10H, Cp), 6.31 (br s, 2H, complexed ArH), 6.45 (br s, 8H, complexed ArH), 7.59 (s, 4H, ArH). $\delta_C(CD_3COCD_3)$: 77.79 (4C, complexed ArC), 78.21 (10C, 2Cp), 85.93 (2C, complexed ArC), 87.93 (4C, complexed ArC), 124.47 (4C, ArC), 134.73 (2C, quat complexed ArC), and 151.91 (2C, quat ArC).

1,2-Bis([η^6-2-methylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3d). A reaction time of 24 h gave 0.337 g (82%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 43.90; H, 3.48. $\delta_H(CD_3COCD_3)$: 2.21, 2.52 (2s, 6H, CH_3), 5.13 (s, 10H, Cp), 6.20–6.35 (m, 6H, complexed ArH), 6.47 (d, 1H, *J* = 6.4 Hz, complexed ArH), 6.52 (d, 1H, *J* = 6.4 Hz, complexed ArH), 7.50–7.75 (m, 4H, ArH). $\delta_C(CD_3COCD_3)$: 15.85, 16.35 (2C, CH_3), 77.00, 77.45 (2C, complexed ArC), 78.54 (10C, Cp), 85.79, 85.84 (2C, complexed ArC), 86.16, 86.24 (2C, complexed ArC), 89.86, 89.91 (2C, complexed ArC), 93.24, 93.89 (2C, quat complexed ArC), 123.36, 124.08 (2C, ArC), 128.87, 129.40 (2C, ArC), 131.07, 131.41 (2C, quat complexed ArC), 144.89, 145.44 (2C, quat ArC).

1,3-Bis([η^6-2-methylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3e). A reaction time of 16 h gave 0.361 g (88%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 43.95; H, 3.38. $\delta_H(CD_3COCD_3)$: 2.62 (s, 6H, CH_3), 5.17, 5.19 (s, 10H, Cp), 6.21 (t, 2H, *J* = 5.2 Hz, complexed ArH), 6.27 (d 2H, *J* = 5.4 Hz, complexed ArH), 6.34 (t, 2H, *J* = 5.4 Hz, complexed ArH), 6.54 (d, 2H, *J* = 5.6 Hz Hz, complexed ArH), 7.28–7.37 (m, 3H, ArH), 7.65–7.76 (m, 1H, ArH). $\delta_C(CD_3COCD_3)$: 16.25 (2C, CH_3), 77.88, 78.14 (2C, complexed ArC), 78.48 (10C, Cp), 85.58 (2C, complexed ArC), 86.28 (2C, complexed ArC), 89.71 (2C, complexed ArC), 94.46 (2C, quat complexed ArC), 113.46 (ArC), 118.49, 118.69 (2C, ArC), 131.87 (2C, quat complexed ArC), 133.50 (ArC), 156.51 (2C, quat ArC).

1,4-Bis([η^6-2-methylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3f). A reaction time of 12 h gave 0.366 g (89%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 43.70; H, 3.35. $\delta_H(CD_3COCD_3)$: 2.67 (s, 6H, CH_3), 5.19, 5.21 (2s, 10H, Cp), 6.22–6.25 (m, 6H, complexed ArH), 6.54–6.57 (m, 2H, complexed ArH), 7.58 (s 4H, ArH). $\delta_C(CD_3COCD_3)$: 16.31 (2C, CH_3), 76.80 (2C, complexed ArC), 78.34 (10C, Cp), 85.23 (2C, complexed ArC), 86.17 (2C, complexed ArC), 89.73 (2C, complexed ArC), 93.75 (2C, quat complexed ArC), 124.13 (4C, ArC), 132.88 (2C, quat complexed ArC), 152.16 (2C, quat ArC).

1,2-Bis([η^6-3-methylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3g). A reaction time of 24 h gave 0.354 g (86%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 43.98; H, 3.41. $\delta_H(CD_3COCD_3)$: 2.54 (s, 6H, CH_3), 5.15 (s, 10H, Cp), 6.22 (d, 2H, *J* = 5.8 Hz, complexed ArH), 6.31–6.41 (m, 6H, complexed ArH), 7.53–7.70 (m, 4H, ArH). $\delta_C(CD_3COCD_3)$: 20.35 (2C, CH_3), 76.07, 76.10 (2C, complexed ArC), 78.63 (10C, Cp), 78.78, 78.88 (2C, complexed ArC), 86.73 (2C, complexed ArC), 86.97 (2C, complexed ArC), 104.04 (2C, quat complexed ArC), 124.26, 124.31 (2C, ArC), 129.31 (2C, ArC), 133.12, 133.15 (2C, quat complexed ArC), 145.37 (2C, quat ArC).

1,3-Bis([η^6-3-methylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3h). A reaction time of 16 h gave 0.370 g (90%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 44.02; H, 3.32. $\delta_H(CD_3COCD_3)$: 2.57 (s, 6H, CH_3), 5.19 (s, 10H, Cp), 6.22 (d, 2H, *J* = 5.2 Hz, complexed ArH), 6.33–6.42 (m, 4H, complexed ArH), 6.52 (s, 2H, complexed ArH), 7.30–7.38 (m, 3H, ArH), 7.70 (t, 1H, *J* = 8.3 Hz, ArH). $\delta_C(CD_3COCD_3)$: 20.34 (2C, CH_3), 76.69, 76.74 (2C, complexed ArC), 78.52 (10C, Cp), 79.45 (2C, complexed ArC), 86.49 (2C, complexed ArC), 86.96 (2C, complexed ArC), 103.99 (2C, quat complexed ArC), 114.00, 114.03 (ArC), 119.07 (2C, ArC), 133.42 (2C, quat complexed ArC), 133.48 (ArC), 156.00 (2C, quat ArC).

1,4-Bis([η^6-3-methylphenoxy-η^5-cyclopentadienyl]iron)-

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[23] (a) Khand, I. U.; Pauson, P. L.; Watts, W. F. *J. Chem. Soc. C* 1968, 2261. (b) Abd-El-Aziz, A. S.; Lee, C. C.; Piorko, A.; Sutherland, R. G. *Synth. Commun.* 1988, *18*, 291.

benzene Hexafluorophosphate (3i). A reaction time of 12 h gave 0.362 g (88%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 43.89; H, 3.50. δ_H(CD_3COCD_3): 2.39 (s, 6H, CH_3), 5.20 (s, 10H, Cp), 6.20–6.23 (m, 2H, complexed ArH), 6.30–6.36 (m, 4H, complexed ArH), 6.48 (s, 2H, complexed ArH), 7.53 (s, 4H, ArH). δ_C(CD_3COCD_3): 20.33 (2C, CH_3), 76.01 (2C, complexed ArC), 78.42 (10C, Cp), 79.02, 79.06 (2C, complexed ArC), 86.24 (2C, complexed ArC), 86.83 (2C, complexed ArC), 103.97 (2C, quat complexed ArC), 123.98 (4C, ArC), 132.98 (2C, quat complexed ArC), 151.96 (2C, quat ArC).

1,2-Bis([η^6-4-methylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3j). A reaction time of 24 h gave 0.345 g (84%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 43.83; H, 3.39. δ_H(CD_3COCD_3): 2.46 (s, 6H, CH_3), 5.14 (s, 10H, Cp), 6.27–6.37 (m, 8H, complexed ArH), 7.52–7.67 (m, 4H, ArH); δ_C(CD_3COCD_3) 19.81 (2C, CH_3), 77.12 (4C, complexed ArC), 78.68 (10C, Cp), 87.86 (4C, complexed ArC), 102.14 (2C, quat complexed ArC), 124.10 (2C, ArC), 129.24 (2C, ArC), 132.25 (2C, quat complexed ArC), 145.45 (2C, quat ArC).

1,3-Bis([η^6-4-methylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3k). A reaction time of 16 h gave 0.362 g (88%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 43.97; H, 3.49. δ_H(CD_3COCD_3): 2.49 (s, 6H, CH_3), 5.21 (s, 10H, Cp), 6.35–6.47 (m, 8H, complexed ArH), 7.33–7.39 (m, 3H, ArH), 7.72 (t, 1H, J = 8.3 Hz, ArH). δ_C(CD_3COCD_3): 19.89 (2C, CH_3), 77.68 (4C, complexed ArC), 78.64 (10C, Cp), 87.91 (4C, complexed ArC), 101.89 (2C, quat complexed ArC), 114.16 (ArC), 119.19 (2C, ArC), 132.87 (2C, quat complexed ArC), 133.54 (ArC), 156.03 (2C, quat ArC).

1,4-Bis([η^6-4-methylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3l). A reaction time of 12 h gave 0.349 g (85%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 44.01; H, 3.35. δ_H(CD_3COCD_3): 2.51 (s, 6H, CH_3), 5.22 (s, 10H, Cp), 6.32–6.44 (m, 8H, complexed ArH), 7.55 (s, 4H, ArH). δ_C(CD_3COCD_3): 19.94 (2C, CH_3), 77.09 (4C, complexed ArC), 78.60 (10C, Cp), 87.92 (4C, complexed ArC), 101.63 (2C, quat complexed ArC), 124.21 (4C, ArC), 133.61 (2C, quat complexed ArC), 152.01 (2C, quat ArC).

1,3-Bis([η^6-2-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3m). A reaction time of 16 h gave 0.406 g (94%). Anal. Calcd for $C_{28}H_{22}Cl_2F_{12}Fe_2O_2P_2$: C, 38.97; H, 2.57. Found: C, 39.10; H, 2.67. δ_H(CD_3COCD_3): 5.31, 5.33 (2s, 10H, Cp), 6.38 (t, 2H, J = 6.4 Hz, complexed ArH), 6.43 (t, 2H, J = 6.2 Hz, complexed ArH), 6.62 (d, 2H, J = 5.6 Hz, complexed ArH), 6.98 (d, 2H, J = 5.6 Hz, complexed ArH), 7.46 (dd, 1H, J = 8.3, J = 2.3 Hz, ArH), 7.59 (br s, 1H, ArH), 7.78 (m, 1H, ArH). δ_C(CD_3COCD_3): 78.39, 78.66 (2C, complexed ArC), 80.38, 80.41 (10C, Cp), 86.20 (2C, complexed ArC), 86.92, 86.96 (2C, complexed ArC), 89.16 (2C, complexed ArC), 98.57 (2C, quat complexed ArC), 113.51, 113.57 (2C, ArC), 119.06, 119.29 (2C, ArC), 130.54, 130.66 (2C, quat complexed ArC), 133.82, 133.90 (2C, ArC), 155.98, 156.09 (2C, quat ArC).

1,4-Bis([η^6-2-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3n). A reaction time of 12 h gave 0.388 g (90%). Anal. Calcd for $C_{28}H_{22}Cl_2F_{12}Fe_2O_2P_2$: C, 38.97; H, 2.57. Found: C, 39.04; H, 2.60. δ_H(CD_3COCD_3): 5.33, 5.35 (2s, 10H, Cp), 6.33 (t, 2H, J = 6.1 Hz, complexed ArH), 6.45 (d, 2H, J = 5.8 Hz, complexed ArH), 6.50 (t, 2H, J = 6.0 Hz, complexed ArH), 7.00 (d, 2H, J = 6.0 Hz, complexed ArH), 7.72, 7.73 (2s, 4H, ArH); δ_C(CD_3COCD) 77.32, 77.39 (2C, complexed ArC), 80.27, 80.32 (10C, 2Cp), 85.89 (2C, complexed ArC), 86.89 (2C, complexed ArC), 89.15 (2C, complexed ArC), 97.90 (2C, quaternary complexed ArC), 124.39 (4C, ArC), 131.69 (2C, quaternary complexed ArC), 152.16, 152.23 (2C, quaternary ArC).

1,2-Bis([η^6-3-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3o). A reaction time of 24 h gave 0.358 g (83%). Anal. Calcd for $C_{28}H_{22}Cl_2F_{12}Fe_2O_2P_2$: C, 38.97; H, 2.57. Found: C, 39.07; H, 2.64. δ_H(CD_3COCD_3): 5.33 (s, 10H, Cp), 6.44 (d 2H, J = 6.4 Hz, complexed ArH), 6.59 (t, 2H, J = 6.4 Hz, complexed ArH), 6.69 (d, 2H, J = 6.2 Hz, complexed ArH), 6.94 (s, 2H, complexed ArH), 7.62–7.74 (m, 4H, ArH). δ_C(CD_3COCD_3): 76.60, 76.68 (2C, complexed ArC), 78.82 (2C, complexed ArC) 80.55 (10C, Cp), 86.79 (2C, complexed ArC), 86.97 (2C, complexed ArC), 107.04 (2C, quat complexed ArC), 124.18 (2C, ArC), 129.68 (2C, ArC), 133.02 (2C, quat complexed ArC), 144.88 (2C, quat ArC).

1,3-Bis([η^6-3-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3p). A reaction time of 16 h gave 0.367 g (85%). Anal. Calcd for $C_{28}H_{22}Cl_2F_{12}Fe_2O_2P_2$: C, 38.97; H, 2.57. Found: C, 38.85; H, 2.69. δ_H(CD_3COCD_3): 5.37, 5.38 (2s, 10H, Cp), 6.50–6.74 (m, 8H, complexed ArH), 7.38–7.73 (m, 4H, ArH). δ_C(CD_3COCD_3): 77.04 (2C, complexed ArC), 79.15 (2C, complexed ArC), 80.21 (10C, Cp), 86.30 (2C, complexed ArC), 86.65 (2C, complexed ArC), 106.88 (2C, quat complexed ArC), 113.73 (ArC), 119.13 (2C, ArC), 133.47 (ArC), 133.17 (2C, quat complexed ArC), 155.40 (2C, quat ArC).

1,4-Bis([η^6-3-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3q). A reaction time of 12 h gave 0.388 g (90%). Anal. Calcd for $C_{28}H_{22}Cl_2F_{12}Fe_2O_2P_2$: C, 38.97; H, 2.57. Found: C, 38.88; H, 2.47. δ_H(CD_3COCD_3): 5.35, 5.36 (2s, 10H, Cp), 6.42 (d, 2H, J = 6.2 Hz, complexed ArH), 6.55 (t, 2H, J = 6.4 Hz, complexed ArH), 6.70 (d, 2H, J = 6.2 Hz, complexed ArH), 7.62 (s, 4H, ArH). δ_C(CD_3COCD_3): 76.61 (2C, complexed ArC), 79.10 (2C, complexed ArC), 80.47 (10C, Cp), 86.45 (2C, complexed ArC), 86.94 (2C, complexed ArC), 107.21 (2C, quaternary complexed ArC), 124.25 (4C, ArC), 134.22 (2C, quaternary complexed ArC), 151.98 (2C, quaternary ArC).

1,2-Bis([η^6-4-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3r). A reaction time of 24 h gave 0.358 g (83%). Anal. Calcd for $C_{28}H_{22}Cl_2F_{12}Fe_2O_2P_2$: C, 38.97; H, 2.57. Found: C, 39.02; H, 2.53. δ_H(CD_3COCD_3): 5.33 (s, 10H, Cp), 6.52 (d, 4H, J = 6.7 Hz, complexed ArH), 6.79 (d, 4H, J = 6.7 Hz, complexed ArH), 7.55–7.70 (m, 4H, ArH). δ_C(CD_3COCD_3): 77.32 (4C, complexed ArC), 80.70 (10C, Cp), 87.90 (4C, complexed ArC), 105.39 (2C, quat complexed ArC), 124.25 (2C, ArC), 129.74 (2C, ArC), 132.57 (2C, quat complexed ArC), 145.00 (2C, quat ArC).

1,3-Bis([η^6-4-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3s). A reaction time of 16 h gave 0.397 g (92%). Anal. Calcd for $C_{28}H_{22}Cl_2F_{12}Fe_2O_2P_2$: 38.97; H, 2.57. Found: C, 38.95; H, 2.62. δ_H(CD_3COCD_3): 5.37 (s, 10H, Cp), 6.61 (d, 4H, J = 5.4 Hz, complexed ArH), 6.79 (d, 4H, J = 5.4 Hz, complexed ArH), 7.38–7.44 (m, 3H, ArH), 7.76 (t, 1H, J = 8.2 Hz, ArH). δ_C(CD_3COCD_3): 77.64 (4C, complexed ArC), 80.50 (10C, Cp), 87.74 (4C, complexed ArC), 105.07 (2C, quat complexed ArC), 114.17 (ArC), 119.50 (2C, ArC), 133.17 (2C, quat complexed ArC), 133.75 (ArC), 155.59 (2C, quat ArC).

1,4-Bis([η^6-4-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (3t). A reaction time of 12 h gave 0.380 g (88%). Anal. Calcd for $C_{28}H_{22}Cl_2F_{12}Fe_2O_2P_2$: C, 38.97; H, 2.57. Found: C, 38.91; H, 2.57. δ_H(CD_3COCD_3): 5.38 (s, 10H, Cp), 6.56 (d, 4H, J = 7.0 Hz, complexed ArH), 6.78 (d, 4H, J = 7.0 Hz, complexed ArH), 7.59 (s 4H, ArH). δ_C(CD_3COCD_3): 77.16 (4C, complexed ArC), 80.57 (10C, Cp), 87.86 (4C, complexed ArC), 105.06, (2C, quaternary complexed ArC), 124.43 (4C, ArC), 134.03 (2C, quaternary complexed ArC), 151.96 (2C, quaternary ArC).

Nucleophilic Substitution Reactions with Biphenols. 4,4′-Bis([η^6-phenoxy-η^5-cyclopentadienyl]iron)biphenyl Hexafluorophosphate (6a). A reaction time of 12 h gave 0.382 (88%). Anal. Calcd for $C_{34}H_{28}F_{12}Fe_2O_2P_2$: C, 46.93; H, 3.24. Found: C, 46.80; H, 3.12. δ_H(CD_3COCD_3): 5.28 (s, 10H, Cp), 6.32 (t, 2H, J = 6.2 Hz, complexed ArH), 6.46–6.55 (m, 6H, complexed ArH), 7.55 (d, 4H, J = 8.8, ArH), 7.91 (d, 4H, J = 8.8 Hz, ArH). δ_C(CD_3COCD_3): 78.11 (4C, complexed ArC), 78.17 (10C, Cp), 85.96 (2C, complexed ArC), 87.90 (4C, complexed ArC), 122.21 (4C, ArC), 130.09 (4C, ArC), 134.32 (2C, quat complexed ArC), 138.83 (2 C, quat ArC), 154.00 (2C, quat ArC).

4,4′-Bis([η^6-2-chlorophenoxy-η^5-cyclopentadienyl]iron)biphenyl Hexafluorophosphate (6b). A reaction time of 12 h gave 0.347 g (74%). Anal. Calcd for $C_{34}H_{26}Cl_2F_{12}Fe_2O_2P_2$: C, 43.49; H, 2.79. Found: C, 43.61; H, 2.83. δ_H(CD_3COCD_3): 5.36 (s, 10H, Cp), 6.39–6.52 (m, 6H, complexed ArH), 6.96–7.02 (m, 2H, complexed ArH), 7.56 (d, 4H, J = 8.8 Hz, arH), 7.93 (d, 4H,

J = 8.8 Hz, ArH). $\delta_C(CD_3COCD_3)$: 77.97 (2C, complexed ArC), 80.27 (10C, Cp), 85.90 (2C, complexed ArC), 86.81 (2C, complexed ArC), 86.16 (2C, complexed ArC), 98.48 (2C, quat complexed ArC), 121.72 (4C, ArC), 130.18 (4C, ArC), 130.18 (2C, quat complexed ArC), 138.88 (2C, quat ArC), 154.20 (2C, quat ArC).

4,4′Bis([η^6-3-chlorophenoxy-η^5-cyclopentadienyl]iron)biphenyl Hexafluorophosphate (6c). A reaction time of 12 h gave 0.404 g (86%). Anal. Calcd for $C_{34}H_{26}Cl_2F_{12}Fe_2O_2P_2$: C, 43.49; H, 2.79. Found: C, 43.39; H, 2.82. $\delta_H(CD_3COCD_3)$: 5.39 (s, 10H, Cp), 6.46 (d, 2H, J = 5.8 Hz, complexed ArH), 6.58–6.72 (m, 4H, complexed ArH), 6.91 (s, 2H, complexed ArH), 7.54 (d, 4H, J = 8.4 Hz, ArH), 7.92 (m, 4H, J = 8.4 Hz, ArH). $\delta_C(CD_3COCD_3)$: 77.04 (2C, complexed ArC), 79.23 (2C, complexed ArC), 80.52 (10C, Cp), 86.47 (2C, complexed ArC), 87.13 (2C, complexed ArC), 107.18 (2C, quat complexed ArC), 122.17 (4C, ArC), 130.19 (4C, ArC), 134.10 (2C, quat complexed ArC), 138.95 (2C, quat ArC), 153.87 (2C, quat ArC).

4,4′-Bis([η^6-4-chlorophenoxy-η^5-cyclopentadienyl]iron)biphenyl Hexafluorophosphate (6d). A reaction time of 12 h gave 0.427 g (91%). Anal. Calcd for $C_{34}H_{26}Cl_2F_{12}Fe_2O_2P_2$: C, 43.49; H, 2.79. Found: C, 43.33; H, 2.85. $\delta_H(CD_3COCD_3)$: 5.41 (s, 10H, Cp), 6.57 (d, 4H, J = 7.0 Hz, complexed ArH), 6.85 (d, 4H, J = 7.0 Hz, complexed ArH), 7.49 (d, 4H, J = 8.6 Hz, ArH), 7.90 (d, 4H, J = 8.6 Hz, ArH). $\delta_C(CD_3COCD_3)$: 76.45 (4C, complexed ArC), 79.43 (10C, Cp), 86.83 (4C, complexed ArC), 103.74 (2C, quat complexed ArC), 121.32 (4C, ArC), 129.16 (4C, ArC), 132.01 (2C, quat complexed ArC), 137.31 (2C, quat ArC), 152.74 (2C, quat ArC).

4,4′-Bis([η^6-4-methylphenoxy-η^5-cyclopentadienyl]iron)biphenyl Hexafluorophosphae (6e). A reaction time of 12 h gave 0.431 g (96%). Anal. Calcd for $C_{36}H_{32}F_{12}Fe_2O_2P_2$: C, 48.14; H, 3.59. Found: C, 48.28; H, 3.62. $\delta_H(CD_3COCD_3)$: 2.50 (s, 6H, CH$_3$), 5.23 (s, 10H, Cp), 6.38 (s, 8H, complexed ArH), 7.46 (d, 4H, J = 8.8 Hz, ArH), 7.88 (d, 4H, J = 8.8 Hz, ArH). $\delta_C(CD_3COCD_3)$: 19.81 (2C, CH$_3$), 77.26 (4C, complexed ArC), 78.50 (10C, Cp), 87.82 (4C, complexed ArC), 101.60 (2C, quat complexed ArC), 122.04 (4C, ArC), 129.98 (4C, ArC), 133.15 (2C, complexed ArC), 138.62 (2C, quat ArC), 154.05 (2C, quat ArC).

2,2′-Bis([η^6-phenoxy-η^5-cyclopentadienyl]iron)biphenyl Hexafluorophosphate (6f). A reaction time of 24 h gave 0.383 g (88%). Anal. Calcd for $C_{34}H_{28}F_{12}Fe_2O_2P_2$: C, 46.93; H, 3.24. Found: C, 46.99; H, 3.24. $\delta_H(CD_3COCD_3)$: 5.00 (s, 10H, Cp), 6.19–6.27 (m, 2H, complexed ArH), 6.30–6.42 (m, 8H, complexed ArH), 7.35–7.68 (m, 8H, ArH). $\delta_C(CD_3COCD_3)$: 78.19 (10C, Cp), 78.77 (4C, complexed ArC), 86.11 (2C, complexed ArC), 87.82 (4C, complexed ArC), 121.28 (2C, ArC), 127.48 (2C, ArC), 130.49 (2C, quat. ArC), 131.80 (2C, ArC), 133.34 (2C, ArC), 133.59 (2C, quat complexed ArC), 152.65 (2C, quat ArC).

Reactions with Sterically Crowded Nucleophiles. 1,4-Bis([η^6-2,6-dimethylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (9a). A reaction time of 16 h gave 0.361 g (85%). Anal. Calcd for $C_{32}H_{32}F_{12}Fe_2O_2P_2$: C, 45.21; H, 3.79. Found: C, 45.37; H, 3.82. $\delta_H(CD_3COCD_3)$: 2.46 (s, 12H, CH$_3$), 5.19 (s, 10H, Cp), 6.28 (t, 2H, J = 6.1 Hz, complexed ArH), 6.47 (d, 4H, J = 6.15 Hz, complexed ArH), 6.97 (s, 4H, ArH). $\delta_C(CD_3COCD_3)$: 16.63 (4C, CH$_3$), 79.39 (10C, Cp), 86.55 (2C, complexed ArC), 88.79 (4C, complexed ArC), 99.45 (4C, quat complexed ArC), 117.58 (4C, ArC), 126.79 (2C, quat complexed ArC), 153.55 (2C, quat ArC).

2-Methyl-1,3-bis([η^6-phenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (9b). A reaction time of 12 h gave 0.384 g (95%). Anal. Calcd for $C_{29}H_{26}F_{12}Fe_2O_2P_2$: C, 43.10; H, 3.24. Found: C, 43.26; H, 3.32. $\delta_H(CD_3COCD_3)$: 2.19 (s, 3H, CH$_3$), 5.28 (s, 10H, Cp), 6.30–6.40 (br s, 2H, complexed ArH), 6.40–6.52 (br s, 8H, complexed ArH), 7.34 (d, 2H, J = 8.4 Hz, ArH), 7.55 (t, 1H, J = 8.4 Hz, ArH). $\delta_C(CD_3COCD_3)$: 9.99 (CH$_3$), 78.01 (4C, complexed ArC), 78.15 (10C, Cp), 85.88 (2C, complexed ArC), 87.82 (4C, complexed ArC), 119.19 (2C, ArC), 129.92 (ArC), 123.79 (quat ArC), 134.05 (2C, quat complexed ArC), 154.08 (2C, quat ArC).

4-Ethyl-1,3-bis([η^6-phenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (9c). A reaction time of 12 h gave 0.349 g (85%). Anal. Calcd for $C_{30}H_{28}F_{12}Fe_2O_2P_2$: C, 43.83; H, 3.43. Found: C, 43.70; H, 3.34. $\delta_H(CD_3COCD_3)$: 1.26 (t, 3H, J = 7.5 Hz, CH$_3$), 2.69 (q, 2H, J = 7.6 Hz, CH$_2$), 5.23 (s, 5H, Cp), 5.25 (s, 5H, Cp), 6.53–6.26 (m, 10H, complexed ArH), 7.33–7.38 (m, 2H, ArH), 7.64 (d, 1H, J = 8.9 Hz, ArH). $\delta_C(CD_3COCD_3)$: 14.47 (CH$_3$), 23.12 (CH$_2$), 78.15, 78.22 (10C, Cp), 78.07, 78.45, 86.02, 86.22, 87.85, 87.98 (10C, complexed ArC), 113.63, 119.56, 133.29 (3C, ArC), 134.12, 134.31 (2C, quat complexed ArC), 134.69, 153.36, 153.60 (3C, quat ArC).

2-Phenyl-1,4-bis([η^6-phenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (9d). A reaction time of 12 h gave 0.392 g (90%). Anal. Calcd for $C_{34}H_{28}F_{12}Fe_2O_2P_2$: C, 46.93; H, 3.24. Found: C, 47.16; H, 3.12. $\delta_H(CD_3COCD_3)$: 4.81 (s, 5H, Cp), 5.29 (s, 5H, Cp), 6.12–6.59 (m, 10H, complexed ArH), 7.35–7.75 (m, 8H, ArH). $\delta_C(CD_3COCD_3)$: 77.58 (2C, complexed ArC), 77.87 (10C, Cp), 78.15 (2C, complexed ArC), 85.49, 85.85 (2C, complexed ArC), 87.42 (2C, complexed ArC), 87.84 (2C, complexed ArC), 123.14, 124.98, 125.17, 129.46, 129.62, 130.41 (8C, ArC), 134.35, 134.59 (2C, quat complexed ArC), 136.73, 138.01, 148.92, 152.08 (4C, quat ArC).

2-Phenyl-1,4-bis([η^6-2,6-dimethylphenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (9e). A reaction time of 24 h gave 0.398 g (86%). Anal. Calcd for $C_{38}H_{36}F_{12}Fe_2O_2P_2$: C, 47.82; H, 3.80. Found: C, 47.99; H, 3.92. $\delta_H(CD_3COCD_3)$: 2.39 (s, 6H, CH$_3$), 2.52 (s, 6H, CH$_3$), 4.97 (s, 5H, Cp), 5.20 (s, 5H, Cp), 6.19–6.38 (m, 2H, complexed ArH), 6.41 (d, 2H, J = 6.2 Hz, complexed ArH), 6.47 (d, 2H, J = 6.2 Hz, complexed ArH), 6.76 (d, 1H, J = 9.0 Hz, ArH), 6.90 (d, 1H, J = 9.0Hz, ArH), 7.18 (d, 1H, J = 3.0 Hz, ArH), 7.42–7.63 (m, 3H, ArH), 7.74–7.82 (m, 2H, ArH). $\delta_C(CD_3COCD_3)$: 16.75 (4C, CH$_3$), 79.19 (5C, Cp), 79.38 (5C, Cp), 86.38, 86.50, 88.79 (8C, complexed ArC), 99.00, 99.47 (4C, quat complexed ArC), 116.24, 116.44, 118.42 (3C, ArC), 126.65 (quat ArC), 126.85 (2C, quat complexed ArC), 129.09, 129.27, 130.50 (5C, ArC), 133.67, 137.06, 150.10 (3C, quat ArC), 153.78 (4C, quat ArC).

2-Methyl-1,4-bis([η^6-phenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (9f). A reaction time of 12 h gave 0.337 g (83%). Anal. Calcd for $C_{29}H_{26}F_{12}Fe_2O_2P_2$: C, 43.10; H, 3.24. Found: C, 43.26; H, 3.32. $\delta_H(CD_3COCD_3)$: 2.28 (s, 3H, CH$_3$), 5.24, 5.26 (2s, 10H, Cp), 6.27–6.34 (m, 2H, complexed ArH), 6.43–6.49 (m, 8H, complexed ArH), 7.35–7.54 (m, 3H, ArH). $\delta_C(CD_3COCD_3)$: 16.87 (CH$_3$), 77.45 (2C, complexed ArC), 77.65 (2C, complexed ArC), 78.11 (10C, Cp), 85.77 (2C, complexed ArC), 87.79 (2C, complexed ArC), 87.87 (2C, complexed ArC), 121.50 (ArC), 123.90 (ArC), 125.47 (ArC), 134.16 (2C, quat ArC), 134.61, 134.70 (2C, quat complexed ArC), 150.09 (quat ArC), 151.66 (2C, quat ArC).

2. Description of Experimental Procedure for Complexes 11a,b. Monoiron complex **1a** or **1d** (4 mmol) was added to a 100-mL round bottom flask containing an 8-fold excess of hydroquinone (3.52 g, 32 mmol), K_2CO_3 (1.11 g, 8 mmol), and 20 mL of a THF/DMF solvent system (4:1 ratio). The flask was fitted with a condensor column and stirred magnetically under N_2 at reflux temperatures for 8 h. The contents of the flask were then added to a separatory funnel, to which was also added 150 mL of CH_2Cl_2 and 20 mL of a 10% HCl solution. The organic phase was extracted and washed repeatedly with water to remove the reaction solvents. The CH_2Cl_2 solution was then dried over $MgSO_4$ and filtered and the solvent removed by evaporation under reduced pressure until a tacky solid was obtained. This solid was then washed with diethyl ether to remove most of the excess hydroquinone, whereupon the crude product was twice precipitated from CH_2Cl_2/diethyl ether as a dark yellow powder. Yields and analytical data for these complexes have been recorded below.

[η^6-{(4-Hydroxyphenoxy)benzene}-η^5-cyclopentadienyl]iron Hexafluorophosphate (11a). A reaction time of 8 h gave 0.958 g (53%). Anal. Calcd for $C_{17}H_{15}F_6FeO_2P$: C, 45.16; H, 3.34. Found: C, 45.01; H, 3.24. $\delta_H(CD_3COCD_3)$: 5.23 (s, 5H, Cp), 6.27–6.48 (m, 5H, complexed ArH), 7.00 (d, 2H, J = 9.0 Hz, ArH), 7.22 (d, 2H, J = 8.8 Hz, ArH), 8.71 (s, 1H, OH). $\delta_C(CD_3COCD_3)$: 76.79 (2C, complexed ArC), 77.77 (5C, Cp), 85.35 (complexed ArC), 87.59 (2C, complexed ArC), 117.75 (2C, ArC), 123.09 (2C, ArC), 135.51 (quat complexed ArC), 145.99, 156.70 (2C, quat ArC). IR (cm^{-1}): 3535 (OH).

[η^6-{(4-Hydroxyphenoxy)toluene}-η^5-cyclopentadienyl]iron Hexafluorophosphate (11b). A reaction time of 8 h gave 1.380 g (74%). Anal. Calcd for $C_{18}H_{17}F_6FeO_2P$: C, 46.38; H, 3.68. Found: C, 46.26; H, 3.59. $\delta_H(CD_3COCD_3)$: 2.47 (s, 3H, CH_3), 5.18 (s, 5H, Cp), 6.21 (d, 2H, J = 6.8 Hz, complexed ArH), 6.33 (d, 2H, J = 6.8 Hz, complexed ArH), 6.98 (d, 2H, J = 8.8 Hz, ArH), 7.19 (d, 2H, J = 9.0 Hz, ArH), 8.67 (s, 1H, OH). $\delta_C(CD_3COCD_3)$: 19.77 (CH_3), 76.04 (2C, complexed ArC), 78.23 (5C, Cp), 87.65 (2C, complexed ArC), 101.06 (quat complexed ArC), 117.74 (2C, ArC), 123.06 (2C, ArC), 134.55 (quat complexed ArC), 146.19, 156.68 (2C, quat ArC). IR (cm^{-1}): 3550 (OH).

3. Description of the Experimental Procedure for Complexes 12a and 12b. These complexes were prepared from an equimolar ratio (1 mmol) of **11a** or **11b** with monoiron complex **1g**, a 2-fold excess of K_2CO_3, and DMF as the solvent. The resulting gold solution was stirred magnetically under an N_2 atmosphere for 12 h. The desired diiron complexes were isolated in the same manner as complexes **3a–t**, **6a–f**, and **9a–f**.

1-([η^6-Phenoxy-η^5-cyclopentadienyl]iron)-4-([η^6-4-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (12a). A reaction time of 12 h gave 0.762 g (92%). Anal. Calcd for $C_{28}H_{23}ClF_{12}Fe_2O_2P_2$: C, 40.59; H, 2.80. Found: C, 40.70; H, 2.66. $\delta_H(CD_3COCD_3)$: 5.24 (s, 5H, Cp), 5.36 (s, 5H, Cp), 6.23–6.35 (m, 1H, complexed ArH), 6.42–6.44 (m, 4H, complexed ArH), 6.53 (d, 2H, J = 7.0 Hz, complexed ArH), 6.74 (d, 2H, J = 6.8 Hz, complexed ArH), 7.58 (s, 4H, ArH). $\delta_C(CD_3COCD_3)$: 77.01 (2C, complexed ArC), 77.56 (2C, complexed ArC), 78.08 (5C, Cp), 80.49 (5C, Cp), 85.80 (complexed ArC), 87.76 (4C, complexed ArC), 104.94 (quat complexed ArC), 124.30 (2C, ArC), 124.50 (2C, ArC), 134.01, 134.57 (2C, quat complexed ArC), 151.66, 151.92 (2C, quat ArC).

1-([η^6-4-Methylphenoxy-η^5-cyclopentadienyl]iron)-4-([η^6-4-chlorophenoxy-η^5-cyclopentadienyl]iron)benzene Hexafluorophosphate (12b). A reaction time of 12 h gave 0.724 g (86%). Anal. Calcd for $C_{29}H_{25}ClF_{12}Fe_2O_2P_2$: C, 41.34; H, 2.99. Found: C, 41.49; H, 3.08. $\delta_H(CD_3COCD_3)$: 2.51 (s, 3H, CH_3), 5.21 (s, 5H, Cp), 5.38 (s, 5H, Cp), 6.31–6.41 (m, 4H, complexed ArH), 6.56 (d, 2H, J = 7.0 Hz, complexed ArH), 6.77 (d, 2H, J = 7.0 Hz, complexed ArH), 7.57 (s, 4H, ArH). $\delta_C(CD_3COCD_3)$: 19.86 (CH_3), 76.90 (2C, complexed ArC), 77.06 (2C, complexed ArC), 78.51 (5C, Cp), 80.50 (5C, Cp), 87.79 (4C, complexed ArC), 101.56, 104.99 (2C, quat complexed ArC), 124.25 (2C, ArC), 124.36 (2C, ArC), 133.51, 134.03 (2C, quat complexed ArC), 151.61, 152.17 (2C, quat ArC).

4. Description of the Experimental Procedure for Complexes 15a–e. Complex **12a**, **12b**, or **3t** was combined with either ethyl cyanoacetate or (phenylsulfonyl)acetonitrile in equimolar amounts (0.5 mmol), and the mixture was added to a 50-mL round bottom flask containing 0.138 g (1 mmol) of K_2CO_3 and 10 mL of DMF. The cherry-red solution was stirred magnetically at room temperature for 5 h, with the exception of the mixture leading to complex **15e**. This mixture was stirred for 1 h at 50 °C, and then for an additional 9 h at room temperature. Isolation of complexes **15a–e** proceeded as for complexes **3a–t**, **6a–f**, and **9a–f**.

1-[η^6-Phenoxy-η^5-cyclopentadienyliron]-4-[η^6-(ethyl {4-phenoxy}cyanoacetate)-η^5-cyclopentadienyliron]benzene Hexafluorophosphate (15a). A reaction time of 5 h gave 0.421 g (93%). Anal. Calcd for $C_{33}H_{29}F_{12}Fe_2NO_4P_2$: C, 43.79; H, 3.23, N, 1.55. Found: C, 43.61; H, 3.23; N, 1.49. $\delta_H(CD_3COCD_3)$: 1.28 (t, 3H, J = 7.0 Hz, CH_3), 4.30 (q, 2H, J = 7.0 Hz, CH_2), 5.26 (s, 5H, Cp), 5.38 (s, 5H, Cp), 5.83 (s, 1H, CH), 6.27–6.50 (m, 5H, complexed ArH), 6.59–6.63 (m, 4H, complexed ArH), 7.55–7.70 (m, 4H, ArH). $\delta_C(CD_3COCD_3)$: 14.06 (CH_3), 42.50 (CH), 64.72 (CH_2), 77.07, 77.19 (2C, complexed ArC), 77.59 (2C, complexed ArC), 78.10 (5C, Cp), 79.56 (5C, Cp), 85.81, 87.79, 88.87 (5C, complexed ArC), 93.90 (quat complexed ArC), 115.37 (CN), 124.57, 124.53 (4C, ArC), 134.62, 135.14 (2C, quat complexed ArC), 151.50, 151.98 (2C, quat ArC), 163.88 (CO). IR (cm^{-1}): 1750 (CO), 2260 (CN).

1-[η^6-Phenoxy-η^5-cyclopentadienyliron]-4-[η^6-{4-phenoxy}(phenylsulfonyl)acetonitrile-η^5-cyclopentadienyliron]benzene Hexafluorophosphate (15b). A reaction time of 5 h gave 0.472 g (97%). Anal. Calcd for $C_{36}H_{29}F_{12}Fe_2NO_4P_2S$: C, 44.43; H, 3.00; N, 1.44. Found: C, 44.53; H, 2.85; N, 1.53. $\delta_H(CD_3COCD_3)$: 5.25 (s, 5H, Cp), 5.36 (s, 5H, Cp), 6.25–6.70 (m, 10H, CH and complexed ArH), 7.58–7.70 (m, 4H, ArH), 7.72–8.10 (m, 5H, SO_2Ph). $\delta_C(CD_3COCD_3)$: 61.17 (CH), 77.46, 77.56 (4C, complexed ArC), 78.06 (5C, Cp), 79.82 (5C, Cp), 85.78, 86.30, 87.74, 89.00 (5C, complexed ArC), 113.11 (CN), 124.61, 124.68 (4C, ArH), 130.81, 130.90 (4C, SO_2Ph), 134.56, 135.00 (3C, quat complexed ArC), 135.71 (quat SO_2Ph), 137.28 (SO_2Ph), 151.34, 152.03 (2C, quat ArC). IR (cm^{-1}): 1160, 1345 (SO_2), 2260 (CN).

1-[η^6-4-Methylphenoxy-η^5-cyclopentadienyliron]-4-[η^6-(ethyl {4-phenoxy}cyanoacetate)-η^5-cyclopentadienyliron]benzene Hexafluorophosphate (15c). A reaction time of 5 h gave 0.359 g (78%). Anal. Calcd for $C_{34}H_{31}F_{12}Fe_2NO_4P_2$: C, 44.43; H, 3.40; N, 1.52. Found: C, 44.64; H, 3.47; N, 1.50. $\delta_H(CD_3COCD_3)$: 1.28 (t, 3H, J = 7.0 Hz, CH_2CH_3), 2.51 (s, 3H, $ArCH_3$), 4.30 (q, 2H, J = 7.0 Hz, CH_2), 5.21 (s, 5H, Cp), 5.37 (s, 5H, Cp), 5.82 (s, 1H, CH), 6.30–6.45 (m, 4H, complexed ArH), 6.55–6.70 (m, 4H, complexed ArH), 7.53–7.66 (m, 4H, ArH). $\delta_C(CD_3COCD_3)$: 14.03 (CH_2CH_3), 19.84 ($ArCH_3$), 42.47 (CH), 64.68 (CH_2), 76.85, 77.02, 77.17 (4C, complexed ArC), 78.48 (5C, Cp), 79.52 (5C, Cp), 85.72, 87.74, 88.83 (4C, complexed ArC), 93.90, 101.48 (2C, quat complexed ArC), 115.33 (CN), 124.36 (2C, ArC), 124.42 (2C, ArC), 133.51, 135.11 (2C, quat complexed ArC), 151.36 (quat ArC), 152.15 (quat ArC), 163.85 (CO). IR (cm^{-1}): 1753 (CO), 2260 (CN).

1-[η^6-4-Methylphenoxy-η^5-cyclopentadienyliron]-4-[η^6-{4-phenoxy}(phenylsulfonyl)acetonitrile-η^5-cyclopentadienyliron]benzene Hexafluorophosphate (15d). A reaction time of 5 h gave 0.449 g (91%). Anal. Calcd for $C_{37}H_{31}F_{12}Fe_2NO_4P_2S$: C, 45.01; H, 3.17; N, 1.42. Found: C, 45.16; H, 3.10; N, 1.49. $\delta_H(CD_3COCD_3)$ 2.52 (s, 3H, CH_3), 5.21 (s, 5H, Cp), 5.36 (s, 5H, Cp), 6.30–6.70 (m, 9H, CH and complexed ArH), 7.58–7.72 (m, 4H, ArH), 7.73–8.05 (m, 5H, SO_2Ph). $\delta_C(CD_3COCD_3)$: 19.88 (CH_3), 61.19 (CH), 76.93, 77.53, 77.59 (4C, complexed ArC), 78.53 (5C, Cp), 79.84 (5C, Cp), 86.38, 87.80 (3C, complexed ArC), 88.84 (quat complexed ArC), 90.06 (complexed ArC), 101.54 (quat complexed ArC), 113.16 (CN), 124.45 (4C, ArC), 130.85 (2C, SO_2Ph), 130.92 (4C, SO_2Ph), 135.09, 135.76 (2C, quat complexed ArC), 137.29 (SO_2Ph), 135.76 (quat SO_2Ph), 151.30, 152.29 (2C, quat ArC). IR (cm^{-1}): 1165, 1350 (SO_2), 2270 (CN).

1-[η^6-4-Chlorophenoxy-η^5-cyclopentadienyliron]-4-[η^6-(ethyl {4-phenoxy}cyanoacetate)-η^5-cyclopentadienyliron]benzene Hexafluorophosphate (15e). A reaction time of 10 h gave 0.428 g (91%). Anal. Calcd for $C_{33}H_{28}F_{12}Fe_2NO_4P_2$: C, 42.18; H, 3.00; N, 1.49. Found: C, 42.26; H, 3.09; N, 1.56. $\delta_H(CD_3COCD_3)$: 1.28 (t, 3H, J = 7.0 Hz, CH_3), 4.30 (q, 2H, J = 7.0 Hz, CH_2), 5.37 (s, 10H, Cp), 5.83 (s, 1H, CH), 6.50–6.79 (m, 8H, complexed ArH), 7.55–7.70 (m, 4H, ArH). $\delta_H(CD_3COCD_3)$: 14.00 (CH_3), 42.43 (CH), 64.65 (CH_2), 76.93, 76.97 (4C, complexed ArC), 79.48 (5C, Cp), 80.44 (5C, Cp), 85.67, 87.69, 88.78 (4C, complexed ArC), 93.87, 104.88 (2C, quat complexed ArC), 115.30 (CN), 124.36, 124.39 (4C, ArC), 133.94, 135.05 (2C, quat complexed ArC), 151.63, 151.79 (2C, quat ArC), 163.82 (CO). IR (cm^{-1}): 1756 (CO), 2180 (CN).

5. Description of the Experimental Procedure for Complex 17. To 0.433 g (0.5 mmol) of complex **3t** were added 0.113 g (1.0 mmol) of ethyl cyanoacetate, 0.345 g (2.5 mmol) K_2CO_3, and 10 mL of DMF. The red solution was magnetically stirred under an N_2 atmosphere for 5 h, whereupon the diiron complex was isolated in the usual fashion.

1,4-Bis[η^6-(ethyl {4-phenoxy}cyanoacetate)-η^5-cyclopentadienyliron]benzene Hexafluorophosphate (17). A reaction time of 5 h gave 0.473 g (93%). Anal. Calcd for $C_{38}H_{34}F_{12}Fe_2N_2O_6P_2$: C, 44.91; H, 3.37; N, 2.76. Found: C, 44.84; H, 3.34; N, 2.40. $\delta_H(CD_3COCD_3)$: 1.27 (t, 6H, J = 7.0 Hz, CH_3), 4.30 (q, 4H, J = 7.0 Hz, CH_2), 5.36 (s, 10H, Cp), 5.82 (s, 2H, CH), 6.59–6.63 (m, 8H, complexed ArH), 7.66 (s, 4H, ArH). $\delta_C(CD_3COCD_3)$: 14.00 (2C, CH_3), 42.44 (2C, CH), 64.65 (2C, CH_2), 76.98, 77.10 (4C, complexed ArC), 79.49 (10C, Cp), 85.72, 88.80 (4C, complexed ArC), 93.90 (2C, quat complexed ArC), 115.35 (2C, CN), 124.60 (4C, ArC), 135.08 (2C, quat complexed ArC), 151.65 (2C, quat ArC), 163.85 (2 CO). IR (cm^{-1}): 1750 (CO), 2165 (CN).

Photolysis Procedure. A 0.5-mmol quantity of a diiron complex was added to a 50-mL Pyrex photolysis tube and dissolved in 40 mL of CH_2Cl_2/CH_3CN. The solution was then purged with N_2 for 30 min and then irradiated under intense visible light (Xenon lamp) for 4 h. The photolyzed solution was concentrated by evaporation under reduced pressure and added to a silica gel column prepared from a hexane slurry. Ferrocene was eluted as a yellow band with hexane, whereupon the desired product was eluted with hexane/$CHCl_3$. The solution containing the product was evaporated to dryness and the resulting material placed under vacuum.

1,2-Diphenoxybenzene (4a). Photolysis resulted in 0.126 g (95%) of a white solid. Anal. Calcd for $C_{18}H_{14}O_2$: C, 82.42; H, 5.38. Found: C, 82.34; H, 5.31. δ_H(CDCl$_3$): 6.90–7.33 (m, 14H, ArH). δ_C(CDCl$_3$): 117.60 (4C, ArC), 121.60 (2C, ArC), 122.79 (2C, ArC), 124.70 (2C, ArC), 129.46 (4C, ArC), 147.73 (2C, quat ArC), 157.45 (2C, quat ArC). Mp: 89–90 °C. MS, m/z: M$^+$, 262 (100).

1,3-Diphenoxybenzene (4b). Photolysis resulted in 0.123 g (94%) of a white solid. Anal. Calcd for $C_{18}H_{14}O_2$: C, 82.42; H, 5.38. Found: C, 82.49; H, 5.39. δ_H(CDCl$_3$): 6.72–6.77 (m, 3H, ArH), 7.03–7.39 (m, 11H, ArH). δ_C(CDCl$_3$): 109.30 (1C, ArC), 113.11 (2C, ArC), 119.12 (4C, ArC), 123.55 (2C, ArC), 129.74 (4C, ArC), 130.31 (ArC), 156.65 (2C, quat ArC), 158.63 (2C, quat ArC). Mp: 59–61 °C (lit.[4b] mp: 59.5–60.4 °C). MS, m/z: M$^+$, 262 (100).

1,4-Diphenoxybenzene (4c). Photolysis resulted in 0.108 g (82%) of a white solid. Anal. Calcd for $C_{18}H_{14}O_2$: C, 82.42; H, 5.38. Found: C, 82.51; H, 5.46. δ_H(CDCl$_3$): 6.98 (s, 4H, ArH), 6.99–7.35 (m, 10H, ArH). δ_C(CDCl$_3$): 118.30 (4C, ArC), 120.44 (4C, ArC), 122.99 (2C, ArC), 129.72 (4C, ArC), 152.72 (2C, quat ArC), 157.78 (2C, quat ArC). Mp: 87 °C. MS, m/z: M$^+$, 262 (100).

1,2-Bis(2-methylphenoxy)benzene (4d). Photolysis resulted in 0.131 g (90%) of a yellowish oil. Anal. Calcd for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.88; H, 6.31. δ_H(CDCl$_3$): 2.07 (s, 6H, CH$_3$), 6.75–6.79 (m, 2H, ArH), 6.92–7.17 (m, 10H, ArH). δ_C(CDCl$_3$): 15.80 (2C, CH$_3$), 117.12 (2C, ArC), 120.36 (2C, ArC), 123.02 (2C, ArC), 123.99 (2C, ArC), 126.75 (2C, ArC), 128.61 (2C, quat ArC), 131.07 (2C, ArC), 155.21 (2C, quat ArC). MS, m/z: M$^+$, 290 (100).

1,3-Bis(2-methylphenoxy)benzene (4e). Photolysis resulted in 0.105 g (72%) of a yellowish oil. Anal. Calcd for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.79; H, 6.29. δ_H(CDCl$_3$): 2.27 (s, 6H, CH$_3$), 6.56–6.59 (m, 3H, ArH), 6.96–7.28 (m, 9H, ArH). δ_C(CDCl$_3$): 16.12 (2C, CH$_3$), 106.61 (ArC), 110.81 (2C, ArC), 119.96 (2C, ArC), 124.22 (2C, ArC), 127.13 (2C, ArC), 130.03 (2C, quat ArC), 130.14 (ArC), 131.44 (2C, ArC), 154.04 (2C, quat ArC), 159.25 (2C, quat ArC). MS, m/z: M$^+$, 290 (100).

1,4-Bis(2-methylphenoxy)benzene (4f). Photolysis resulted in 0.123 g (85%) of a yellowish oil. Anal. Calcd for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.58; H, 6.25. δ_H(CDCl$_3$): 2.26 (s, 6H, CH$_3$), 6.87 (s, 4H, ArH), 6.94–7.24 (m, 8H, ArH). δ_C(CDCl$_3$): 16.16 (2C, CH$_3$), 118.67 (2C, ArC), 118.99 (4C, ArC), 123.51 (2C, ArC), 127.03 (2C, ArC), 129.41 (2C, quat ArC), 131.38 (2C, ArC), 152.85 (2C, quat ArC), 155.27 (2C, quat ArC). MS, m/z: M$^+$, 290 (100).

1,2-Bis(3-methylphenoxy)benzene (4g). Photolysis resulted in 0.122 g (85%) of a yellowish oil. Anal. Calcd for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.62; H, 6.28. δ_H(CDCl$_3$): 2.29 (s, 6H, CH$_3$), 6.71–6.87 (m, 6H, ArH), 7.06–7.16 (m, 6H, ArH). δ_C(CDCl$_3$): 21.46 (2C, CH$_3$), 114.63 (2C, ArC), 118.34 (2C, ArC), 121.47 (2C, ArC), 123.57 (2C, ArC), 124.47 (2C, ArC), 129.12 (2C, ArC), 139.54 (2C, quat ArC), 147.80 (2C, quat ArC), 157.42 (2C, quat ArC). MS, m/z: M$^+$, 290 (100).

1,3-Bis(3-methylphenoxy)benzene (4h). Photolysis resulted in 0.132 g (91%) of a yellowish oil. Anal. Calcd for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.71; H, 6.27. δ_H(CDCl$_3$): 2.34 (s, 6H, CH$_3$), 6.69–6.95 (m, 9H, ArH), 7.18–7.30 (m, 3H, ArH). δ_C(CDCl$_3$): 21.35 (2C, CH$_3$), 109.25 (ArC), 113.00 (2C, ArC), 116.12 (2C, ArC), 119.78 (2C, ArC), 124.34 (2C, ArC), 129.43 (2C, ArC), 130.21 (ArC), 139.92 (2C, quat ArC), 156.62 (2C, quat ArC), 158.68 (2C, quat ArC). MS, m/z: M$^+$, 290 (100).

1,4-Bis(3-methylphenoxy)benzene (4i). Photolysis resulted in 0.116 g (80%) of a yellowish oil. Anal. Calcd for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.89; H, 6.20. δ_H(CDCl$_3$): 2.32 (s, 6H, CH$_3$), 6.76–6.90 (m, 6H, ArH), 6.97 (s, 4H, ArH), 7.15–7.24 (m, 2H, ArH). δ_C(CDCl$_3$): 21.38 (2C, CH$_3$), 115.29 (2C, ArC), 119.00 (2C, ArC), 120.32 (4C, ArC), 123.79 (2C, ArC), 129.40 (2C, ArC), 139.90 (2C, quat ArC), 152.71 (2C, quat ArC), 157.73 (2C, quat ArC). MS, m/z: M$^+$, 290 (100).

1,2-Bis(4-methylphenoxy)benzene (4j). Photolysis resulted in 0.139 g (96%) of a white solid. Anal. Calcd for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.61; H, 6.33. δ_H(CDCl$_3$): 2.30 (s, 6H, CH$_3$), 6.78–6.86 (m, 4H, ArH), 6.96–7.10 (m, 8H, ArH). δ_C(CDCl$_3$): 20.63 (2C, CH$_3$), 117.83 (4C, ArC), 120.86 (2C, ArC), 124.17 (2C, ArC), 129.95 (4C, ArC), 132.32 (2C, quat ArC), 148.11 (2C, quat ArC), 155.14 (2C, quat ArC). Mp: 68 °C. MS, m/z: M$^+$, 290 (100).

1,3-Bis(4-methylphenoxy)benzene (4k). Photolysis resulted in 0.128 g (88%) of a yellowish oil. Anal. Calcd for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.88; H, 6.31. δ_H(CDCl$_3$): 2.32 (s, 6H, CH$_3$), 6.62–6.66 (m, 3H, ArH), 6.89–6.93 (m, 4H, ArH), 7.10–7.19 (m, 5H, ArH). δ_C(CDCl$_3$): 20.70 (2C, CH$_3$), 108.45 (ArC), 112.27 (2C, ArC), 119.29 (4C, ArC), 130.15 (ArC), 130.24 (4C, ArC), 133.19 (2C, quat ArC), 154.21 (2C, quat ArC), 159.13 (2C, quat ArC). MS, m/z: M$^+$, 290 (100).

1,4-Bis(4-methylphenoxy)benzene (4l). Photolysis resulted in 0.113 g (78%) of a white solid. Anal. Calcd for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.81; H, 6.16. δ_H(CDCl$_3$): 2.32 (s, 6H, CH$_3$), 6.88–6.92 (m, 4H, ArH), 6.95 (s, 4H, ArH), 7.10–7.15 (m, 4H, ArH). δ_C(CDCl$_3$): 20.63 (2C, CH$_3$), 118.43 (4C, ArC), 119.90 (4C, ArC), 130.18 (4C, ArC), 132.55 (2C, quat ArC), 152.98 (2C, quat ArC), 155.42 (2C, quat ArC). Mp: 82–83 °C (lit.[5d] mp 79–81 °C). MS, m/z: M$^+$, 290 (100).

1,3-Bis(2-chlorophenoxy)benzene (4m). Photolysis resulted in 0.136 g (82%) of a yellowish oil. Anal. Calcd for $C_{18}H_{12}Cl_2O_2$: C, 65.28; H, 3.65. Found: C, 65.40; H, 3.68. δ_H(CDCl$_3$): 6.58–6.99 (m, 3H, ArH), 7.02–7.44 (m, 9H, ArH). δ_C(CDCl$_3$): 107.61 (ArC), 112.25 (2C, ArC), 121.25 (2C, ArC), 125.08 (2C, ArC), 126.06 (2C, quat ArC), 127.98 (2C, ArC), 130.41 (ArC), 130.82 (2C, ArC), 151.89 (2C, quat ArC), 158.31 (2C, quat ArC). MS, m/z: M$^+$, 330 (23.6); M + 2, 332 (15.4); M + 4, 334 (1.4).

1,4-Bis(2-chlorophenoxy)benzene (4n). Photolysis resulted in 0.139 g (84%) of a white solid. Anal. Calcd for $C_{18}H_{12}Cl_2O_2$: C, 65.28; H, 3.65. Found: C, 65.21; H, 3.61. δ_H(CDCl$_3$): 6.95 (s, 6H, ArH), 7.05 (t, 2H, J = 8.0 Hz, ArH), 7.17 (t, 2H, J = 8.0 Hz, ArH), 7.44 (d, 2H, J = 8.0 Hz, ArH). δ_C(CDCl$_3$): 119.54 (4C, ArC), 120.05 (2C, ArC), 124.42 (2C, ArC), 125.40 (2C, quat ArC), 127.89 (2C, ArC), 130.78 (2C, ArC), 152.54 (2C, quat ArC), 152.93 (2C, quat ArC). Mp: 89–90 °C. MS, m/z: 330 (100); M + 2, 332 (64); M + 4, 334 (11).

1,2-Bis(3-chlorophenoxy)benzene (4o). Photolysis resulted in 0.144 g (87%) of a yellowish oil. Anal. Calcd for $C_{18}H_{12}Cl_2O_2$: C, 65.28; H, 3.65. Found: C, 65.35; H, 3.71. δ_H(CDCl$_3$): 6.71–7.21 (m, 12H, ArH). δ_C(CDCl$_3$): 115.42 (2C, ArC), 117.51 (2C, ArC), 122.33 (2C, ArC), 122.96 (2C, ArC), 125.65 (2C, ArC), 130.23 (2C, ArC), 134.85 (2C, quat ArC), 146.93 (2C, quat ArC), 158.12 (2C, quat ArC). MS, m/z: M$^+$, 330 (100); M + 2, 332 (65); M + 4, 334 (11).

1,3-Bis(3-chlorophenoxy)benzene (4p). Photolysis resulted in 0.145 g (87%) of a yellowish oil. Anal. Calcd for $C_{18}H_{12}Cl_2O_2$: C, 65.28; H, 3.65. Found: C, 65.25; H, 3.62. δ_H(CDCl$_3$): 6.70 (t, 1H, J = 2.2 Hz, ArH), 6.79 (dd, 2H, J = 8.2, 2.4 Hz, ArH), 6.92 (dd, 2H, J = 8.2, 2.2 Hz, ArH), 7.03 (t, 2H, J = 2.2 Hz, ArH), 7.10 (m, 2H, ArH), 7.25–7.36 (m, 3H, J = 8.2 Hz, ArH). δ_C(CDCl$_3$): 110.15 (ArC), 114.24 (2C, ArC), 117.07 (2C, ArC), 119.25 (2C, ArC), 123.74 (2C, ArC), 130.57 (ArC), 135.13 (2C, quat ArC), 157.59 (2C, quat ArC), 157.88 (2C, quat ArC). MS, m/z: M$^+$, 330 (100); M + 2, 332 (64); M + 4, 334 (10).

1,4-Bis(3-chlorophenoxy)benzene (4q). Photolysis resulted in 0.154 g (93%) of a yellowish oil. Anal. Calcd for $C_{18}H_{12}Cl_2O_2$: C, 65.28; H, 3.65. Found: C, 65.40; H, 3.66. δ_H(CDCl$_3$): 7.01 (s, 4H, ArH), 6.84–7.28 (m, 8H, ArH). δ_C(CDCl$_3$): 116.33 (2C, ArC), 118.45 (2C, ArC), 120.93 (4C, ArC), 123.19 (2C, ArC), 130.52 (2C, ArC), 135.09 (2C, quat ArC), 152.34 (2C, quat ArC), 158.52 (2C, quat ArC). MS, *m/z*: M$^+$, 330 (100); M + 2, 332 (64); M + 4, 334 (11).

1,2-Bis(4-chlorophenoxy)benzene (4r). Photolysis resulted in 0.163 g (98%) of a yellowish oil. Anal. Calcd for $C_{18}H_{12}Cl_2O_2$: C, 65.28; H, 3.65. Found: C, 65.25; H, 3.70. δ_H(CDCl$_3$): 6.76–6.80 (m, 4H, ArH), 7.08–7.23 (m, 8H, ArH). δ_C(CDCl$_3$): 118.53 (4C, ArC), 121.91 (2C, ArC), 125.34 (2C, ArC), 127.82 (2C, quat ArC), 129.48 (4C, ArC), 147.23 (2C, quat ArC), 155.98 (2C, quat ArC). MS, *m/z*: M$^+$, 330 (100); M + 2, 332 (68); M + 4, 334 (12).

1,3-Bis(4-chlorophenoxy)benzene (4s). Photolysis resulted in 0.126 g (76%) of a white solid. Anal. Calcd for $C_{18}H_{12}Cl_2O_2$: C, 65.28; H, 3.65. Found: C, 65.34; H, 3.60. δ_H(CDCl$_3$): 6.64 (t, 1H, J = 2.2 Hz, ArH), 6.70 (dd, 2H, J = 8.2, 2.2 Hz, ArH), 6.92–6.97 (m, 4H, ArH), 7.22–7.31 (m, 5H, ArH). δ_C(CDCl$_3$): 109.32 (ArC), 113.44 (2C, ArC), 120.38 (4C, ArC), 129.81 (4C, ArC), 128.76 (2C, quat ArC), 130.61 (ArC), 155.25 (2C, quat ArC), 158.39 (2C, quat ArC). Mp: 59 °C. MS, *m/z*: M$^+$, 330 (100); M + 2, 332 (64); M + 4, 334 (11).

1,4-Bis(4-chlorophenoxy)benzene (4t). Photolysis resulted in 0.127 g (76%) of a white solid. Anal. Calcd for $C_{18}H_{12}Cl_2O_2$: C, 65.28; H, 3.65. Found: C, 65.38; H, 3.69. δ_H(CDCl$_3$): 6.97 (s, 4H, ArH), 6.89–6.93 (m, 4H, ArH), 7.24–7.29 (m, 4H, ArH). δ_C(CDCl$_3$): 120.04 (4C, ArC), 121.00 (4C, ArC), 128.61 (2C, quat ArC), 130.22 (4C, ArC), 153.12 (2C, quat ArC), 156.78 (2C, quat ArC). Mp: 94–95 °C. MS, *m/z*: M$^+$, 330 (100); M + 2, 332 (66); M + 4, 334 (11).

4,4′-Diphenoxybiphenyl (7a). Photolysis resulted in 0.162 g (96%) of a white solid. Anal. Calcd for $C_{24}H_{18}O_2$: C, 85.18; H, 5.36. Found: C, 85.29; H, 5.40. δ_H(CDCl$_3$): 7.02–7.10 (m, 10H, ArH), 7.24–7.38 (m, 4H, ArH), 7.49–7.53 (m, 4H, ArH). δ_C(CDCl$_3$): 118.96 (4C, ArC), 119.07 (4C, ArC), 123.35 (2C, ArC), 128.15 (4C, ArC), 129.77 (4C, ArC), 135.63 (2C, quat ArC), 156.62 (2C, quat ArC), 157.14 (2C, quat ArC). Mp: 149–150 °C. MS, *m/z*: M$^+$, 338 (100).

4,4′-Bis(2-chlorophenoxy)biphenyl (7b). Photolysis resulted in 0.173 g (85%) of a white solid. Anal. Calcd for $C_{24}H_{16}Cl_2O_2$: C, 70.78; H, 3.96. Found: C, 70.89; H, 3.91. δ_H(CDCl$_3$): 6.99–7.29 (m, 10H, ArH), 7.46–7.56 (m, 6H, ArH). δ_C(CDCl$_3$): 118.05 (4C, ArC), 120.95 (2C, ArC), 124.80 (2C, ArC), 125.91 (2C, quat ArC), 127.94 (2C, ArC), 128.15 (4C, ArC), 130.78 (2C, ArC), 135.60 (2C, quat ArC), 152.30 (2C, quat ArC), 156.32 (2C, quat ArC). Mp: 114–115 °C. MS, *m/z*: 406 (69); M + 2, 408 (44); M + 4, 410 (9).

4,4′-Bis(3-chlorophenoxy)biphenyl (7c). Photolysis resulted in 0.190 g (94%) of a white solid. Anal. Calcd for $C_{24}H_{16}Cl_2O_2$: C, 70.78; H, 3.96. Found: C, 70.91; H, 4.01. δ_H(CDCl$_3$): 6.88–7.56 (m, 16H, ArH). δ_C(CDCl$_3$): 116.78 (2C, ArC), 118.87 (2C, ArC), 119.64 (4C, ArC), 123.33 (2C, ArC), 128.39 (4C, ArC), 130.52 (2C, ArC), 135.08 (2C, quat ArC), 136.23 (2C, quat ArC), 155.78 (2C, quat ArC), 158.20 (2C, quat ArC). Mp: 67–68 °C. MS, *m/z*: M$^+$, 406 (100); M + 2, 408 (68); M + 4, 410 (13).

4,4′-Bis(4-chlorophenoxy)biphenyl (7d). Photolysis resulted in 0.152 g (75%) of a white solid. Anal. Calcd for $C_{24}H_{16}Cl_2O_2$: C, 70.78; H, 3.96. Found: C, 70.68; H, 3.94. δ_H(CDCl$_3$): 6.94–7.05 (m, 8H, ArH), 7.23–7.31 (m, 4H, ArH), 7.48–7.55 (m, 4H, ArH). δ_C (CDCl$_3$): 119.14 (4C, ArC), 120.12 (4C, ArC), 128.29 (4C, ArC), 128.30 (2C, quat ArC), 129.76 (4C, ArC), 135.89 (2C, quat ArC), 155.81 (2C, quat ArC), 156.31 (2C, quat ArC). Mp: 179 °C. MS, *m/z*: M$^+$, 406 (100); M + 2, 408 (68); M + 4, 410 (12).

4,4′-Bis(4-methylphenoxy)biphenyl (7e). Photolysis resulted in 0.189 g (93%) of a white solid. Anal. Calcd for $C_{26}H_{22}O_2$: C, 85.22; H, 6.05. Found: C, 85.37; H, 5.98. δ_H(CDCl$_3$): 2.33 (s, 6H, CH$_3$), 6.92–7.17 (m, 12H, ArH), 7.46–7.51 (m, 4H, ArH). δ_C(CDCl$_3$): 20.72 (2C, CH$_3$), 118.53 (4C, ArC), 119.18 (4C, ArC), 128.05 (4C, ArC), 130.27 (4C, ArC), 133.10 (2C, quat ArC), 135.30 (2C, quat ArC), 154.64 (2C, quat ArC), 157.14 (2C, quat ArC). Mp: 172–173 °C. MS, *m/z*: 366 (100).

2,2′-Diphenoxybiphenyl (7f). Photolysis resulted in 0.154 g (91%) of a white solid. Anal. Calcd for $C_{24}H_{18}O_2$: C, 85.18; H, 5.36. Found: C, 85.10; H, 5.38. δ_H(CDCl$_3$): 6.81–7.43 (m, 18H, ArH). δ_C(CDCl$_3$): 118.69 (2C, ArC), 118.78 (4C, ArC), 122.66 (2C, ArC), 123.04 (2C, ArC), 129.22 (2C, ArC), 129.37 (4C, ArC), 129.70 (2C, quat ArC), 131.96 (2C, ArC), 154.66 (2C, quat ArC), 157.42 (2C, quat ArC). Mp: 90–91 °C. MS, *m/z*: M$^+$, 338 (100).

1,4-Bis(2,6-dimethylphenoxy)benzene (10a). Photolysis resulted in 0.124 g (79%) of a white solid. Anal. Calcd for $C_{22}H_{22}O_2$: C, 82.99; H, 6.96. Found: C, 82.73; H, 6.91. δ_H(CDCl$_3$): 2.10 (s, 12H, CH$_3$), 6.60 (s, 4H, ArH), 6.99–7.05 (m, 6H, ArH). δ_C(CDCl$_3$) 16.39 (4C, CH$_3$), 115.43 (4C, ArC), 124.86 (2C, ArC), 128.94 (4C, ArC), 131.50 (4C, quat ArC), 151.62 (2C, quat ArC), 152.33 (2C, quat ArC). Mp: 177–78 °C. MS, *m/z*: M$^+$, 318 (100).

2-Methyl-1,4-diphenoxybenzene (10b). Photolysis resulted in 0.106 g (77%) of a white solid. Anal. Calcd for $C_{19}H_{16}O_2$: C, 82.58; H, 5.84. Found: C, 82.68; H, 5.89. δ_H(CDCl$_3$): 2.19 (s, 3H, CH$_3$), 6.84–7.34 (m, 13H, ArH). δ_C(CDCl$_3$): 16.33 (CH$_3$), 116.71 (2C, ArC), 117.65 (2C, ArC), 118.40 (2C, ArC), 121.35 (2C, ArC), 121.86 (ArC), 122.15 (ArC), 122.96 (ArC), 129.64 (ArC), 129.70 (ArC), 131.88 (quat ArC), 149.85 (quat ArC), 153.12 (quat ArC), 157.71 (quat ArC), 158.25 (quat ArC). Mp: 52 °C. MS, *m/z*: M$^+$, 276 (100).

4-Ethyl-1,3-diphenoxybenzene (10c). Photolysis resulted in 0.115 g (79%) of a yellowish oil. Anal. Calcd for $C_{19}H_{16}O_2$: C, 82.73; H, 6.25. Found: C, 82.91; H, 6.28. δ_H(CDCl$_3$): 1.24 (t, 3H, J = 7.5 Hz, CH$_3$), 2.66 (q, 2H, J = 7.6 Hz, CH$_2$), 6.63–7.37 (m, 13H, ArH). δ_C(CDCl$_3$): 14.53 (CH$_3$), 22.77 (CH$_2$), 110.65 (ArC), 114.39 (ArC), 117.73 (ArC), 118.53 (ArC), 122.75 (ArC), 123.11 (ArC), 129.68 (ArC), 129.71 (ArC), 130.33 (ArC), 130.66 (quat ArC), 155.13 (quat ArC), 156.00 (quat ArC), 157.32 (quat ArC), 157.66 (quat ArC). MS, *m/z*: M$^+$, 290 (93).

2-Phenyl-1,4-diphenoxybenzene (10d). Photolysis resulted in 0.154 g (91%) of a white solid. Anal. Calcd for $C_{24}H_{18}O_2$: C, 85.18; H, 5.36. Found: C, 85.09; H, 5.29. δ_H(CDCl$_3$): 6.53–6.90 (m, 18H, ArH). δ_C(CDCl$_3$): 117.54 (2C, ArC), 118.48 (ArC), 119.14 (ArC), 121.42 (ArC), 121.89 (2C, ArC), 122.38 (ArC), 123.15 (ArC), 127.46 (ArC), 128.14 (2C, ArC), 129.03 (2C, ArC), 129.56 (2C, ArC), 129.77 (2C, ArC), 135.21 (quat ArC), 137.08 (quat ArC), 148.91 (quat ArC), 153.22 (quat ArC), 157.51 (quat ArC), 158.18 (quat ArC). Mp: 91 °C. MS, *m/z*: M$^+$, 338 (100).

2-Phenyl-1,4-bis(2,6-dimethylphenoxy)benzene (10e). Photolysis resulted in 0.178 g (90%) of a white solid. Anal. Calcd for $C_{28}H_{26}O_2$: C, 85.25; H, 6.64. Found: C, 85.17; H, 6.65. δ_H(CDCl$_3$): 2.10 (s, 6H, CH$_3$), 2.17 (s, 6H, CH$_3$), 6.28 (d, 1H, J = 9.0 Hz, ArH), 6.43 (dd, 1H, J = 3.2, 9.0 Hz, ArH), 6.85 (d, 1H, J = 3.0 Hz, ArH), 6.96–7.08 (m, 6H, ArH), 7.31–7.47 (m, 3H, ArH), 7.61–7.70 (m, 2H, ArH). δ_C(CDCl$_3$): 16.48 (4C, CH$_3$), 113.69 (ArC), 113.94 (ArC), 117.03 (ArC), 124.72 (ArC), 124.89 (ArC), 127.10 (ArC), 127.96 (2C, ArC), 128.92 (2C, ArC), 128.95 (2C, ArC), 129.51 (2C, ArC), 130.87 (quat ArC), 131.31 (2C, quat ArC), 131.45 (2C, quat ArC), 138.03 (quat ArC), 148.95 (quat ArC), 151.47 (quat ArC), 151.70 (quat ArC), 152.20 (quat ArC). Mp: 119–120 °C. MS, *m/z*: M$^+$, 394 (100).

2-Methyl-1,3-diphenoxybenzene (10f). Photolysis resulted in 0.124 g (90%) of a yellowish oil. Anal. Calcd for $C_{19}H_{16}O_2$: C, 82.58; H, 5.84. Found: C, 82.56; H, 5.89. δ_H(CDCl$_3$): 2.14 (s, 3H, CH$_3$), 6.69–7.35 (m, 13H, ArH). δ_C(CDCl$_3$): 9.37 (CH$_3$), 115.09 (2C, ArC), 117.55 (4C, ArC), 122.41 (quat ArC), 122.63 (2C, ArC), 126.79 (1C, ArC), 129.69 (4C, ArC), 155.86 (2C, quat ArC), 157.70 (2C, quat ArC). MS, *m/z*: M$^+$, 276 (100).

1-(4-Chlorophenoxy)-4-phenoxybenzene (13a). Photolysis resulted in 0.123 g (83%) of a white solid. Anal. Calcd for $C_{18}H_{13}ClO_2$: C, 72.85; H, 4.42. Found: C, 72.99; H, 4.48. δ_H(CDCl$_3$): 6.99 (s, 4H, ArH), 6.90–7.13 (m, 5H, ArH), 7.25–7.37 (m, 4H, ArH). δ_C(CDCl$_3$): 118.41 (2C, ArC), 119.42 (2C, ArC), 120.43 (2C, ArC), 120.51 (2C, ArC), 123.15 (2C, ArC), 127.95 (quat ArC), 129.69 (2C, ArC), 129.76 (ArC), 152.22 (quat ArC), 153.09

(quat ArC), 156.47 (quat ArC), 157.59 (quat ArC). Mp: 63–64 °C. MS, *m/z*: M^+, 296 (100); M + 2, 298 (33).

1-(4-Chlorophenoxy)-4-(4-methylphenoxy)benzene (13b). Photolysis resulted in 0.127 g (82%) of a white solid. Anal. Calcd for $C_{19}H_{15}ClO_2$: C, 73.43; H, 4.86. Found: C, 73.58; H, 4.79. $\delta_H(CDCl_3)$: 2.35 (s, 3H, CH_3), 6.99 (s, 4H, ArH), 6.91–7.01 (m, 4H, ArH), 7.14–7.18 (m, 2H, ArH), 7.26–7.34 (m, 2H, ArH). $\delta_C(CDCl_3)$: 20.68 (CH_3), 118.66 (2C, ArC), 119.29 (2C, ArC), 119.84 (2C, ArC), 120.52 (2C, ArC), 127.83 (quat ArC), 129.65 (2C, ArC), 130.26 (2C, ArC), 132.84 (quat ArC), 151.81 (quat ArC), 153.74 (quat ArC), 155.06 (quat ArC), 156.58 (quat ArC). Mp: 122 °C. MS, *m/z*: M^+, 310 (100); M + 2, 312 (35).

1-(Ethyl {4-phenoxy}cyanoacetate)-4-phenoxybenzene (16a). Photolysis resulted in 0.133 g (71%) of a yellowish oil. Anal. Calcd for $C_{23}H_{19}NO_4$: C, 73.98; H, 5.13; N, 3.75. Found: C, 73.85; H, 5.10; N, 3.77. $\delta_H(CDCl_3)$: 1.28 (t, 3H, J = 7.0 Hz, CH_3), 4.24 (q, 2H, J = 7.0 Hz, CH_2), 6.97 (s, 1H, CH), 7.00 (s, 4H, ArH), 6.97–7.12 (m, 5H, ArH), 7.29–7.41 (m, 4H, ArH). $\delta_C(CDCl_3)$: 13.86 (CH_3), 42.92 (CH), 63.26 (CH_2), 115.65 (CN), 118.31 (2C, ArC), 118.44 (2C, ArC), 120.35 (2C, ArC), 121.05 (2C, ArC), 123.17 (2C, ArC), 123.96 (quat ArC), 129.36 (2C, ArC), 129.73 (ArC), 151.44 (quat ArC), 153.39 (quat ArC), 157.40 (quat ArC), 158.82 (quat ArC), 164.98 (CO). MS, *m/z*: M^+, 373 (24.2). IR (cm^{-1}): 1750 (CO), 2245 (CN).

1-({4-Phenoxy}(phenylsulfonyl)acetonitrile)-4-phenoxybenzene (16b). Photolysis resulted in 0.148 g (67%) of an off-white solid. Anal. Calcd for $C_{26}H_{19}NO_4S$: C, 70.73; H, 4.34; N, 3.17. Found: C, 70.67; H, 4.35; N, 3.21. $\delta_H(CDCl_3)$: 5.08 (s, 1H, CH), 6.94–7.80 (m, 18H, ArH). $\delta_C(CDCl_3)$: 62.37 (CH), 113.41 (CN), 117.84 (2C, ArC), 118.57 (2C, ArC), 118.87 (quat ArC), 120.36 (2C, ArC), 121.31 (2C, ArC), 123.32 (ArC), 129.24 (2C, ArC), 129.79 (2C, ArC), 130.06 (2C, SO_2Ph), 131.36 (2C, SO_2Ph), 135.27 (SO_2Ph), 134.42 (quat SO_2Ph), 150.95 (quat ArC), 153.76 (quat ArC), 57.22 (quat ArC), 160.14 (quat ArC). Mp: 170–171 °C. MS, *m/z*: M^+, 441 (4.0). IR (cm^{-1}): 1155, 1335 (SO_2).

1-(Ethyl {4-phenoxy}cyanoacetate)-4-(4-methylphenoxy)benzene (16c). Photolysis resulted in 0.143 g (74%) of a yellowish oil. Anal. Calcd for $C_{24}H_{21}NO_4$: C, 74.40; H, 5.46; N, 3.62. Found: C, 74.52; H, 5.44; N, 3.67. $\delta_H(CDCl_3)$: 1.28 (t, 3H, J = 7.0 Hz, $CH_2\mathit{CH_3}$), 2.32 (s, 3H, $ArCH_3$), 4.24 (q, 2H, J = 7.0 Hz, CH_2), 4.66 (s, 1H, CH), 6.88–7.40 (m, 12H, ArH). $\delta_C(CDCl_3)$: 13.88 ($CH_2\mathit{CH_3}$), 20.66 ($ArCH_3$), 42.96 (CH), 63.29 (CH_2), 115.67 (CN), 118.24 (2C, ArC), 118.72 (2C, ArC), 119.78 (2C, ArC), 121.07 (2C, ArC), 123.87 (1C, quat ArC), 129.35 (2C, ArC), 130.25 (2C, ArC), 132.90 (1C, quat ArC), 151.08 (1C, quat ArC), 154.08 (1C, quat ArC), 154.91 (1C, quat ArC), 158.96 (1C, quat ArC), 165.02 (CO). MS, *m/z*: M^+, 387 (6.6). IR (cm^{-1}): 1750 (CO), 2250 (CN).

1-({4-Phenoxy}(phenylsulfonyl)acetonitrile)-4-(4-methylphenoxy)benzene (16d). Photolysis resulted in 0.200 g (88%) of an off-white solid. Anal. Calcd for $C_{27}H_{21}NO_4S$: C, 71.19; H, 4.65; N, 3.07. Found: C, 71.30; H, 4.61; N, 3.11. $\delta_H(CDCl_3)$: 2.33 (s, 3H, CH_3), 5.08 (s, 1H, CH), 6.89–6.95 (m, 4H, ArH), 6.98 (s, 4H, ArH), 7.12–7.24 (m, 4H, ArH), 7.54–7.79 (m, 5H, SO_2Ph). $\delta_C(CDCl_3)$: 20.66 (CH_3), 62.37 (CH), 113.43 (CN), 117.75 (2C, ArC), 118.81 (quat ArC), 118.82 (2C, ArC), 119.76 (2C, ArC), 121.27 (2C, ArC), 129.23 (2C, ArC), 130.28 (2C, ArC), 130.05 (2C, SO_2Ph), 131.33 (2C, SO_2Ph), 133.03 (quat ArC), 134.24 (quat SO_2Ph), 135.25 (SO_2Ph), 150.57 (quat ArC), 154.39 (quat ArC), 154.79 (quat ArC), 160.22 (quat ArC). Mp: 153–154 °C. MS, *m/z*: M^+, 455 (5.5). IR (cm^{-1}): 1155, 1340 (SO_2).

1-(Ethyl {4-phenoxy}cyanoacetate)-4-(4-chlorophenoxy)benzene (16e). Photolysis resulted in 0.149 g (73%) of a yellowish oil. Anal. Calcd for $C_{23}H_{18}NO_4Cl$: C, 67.73; H, 4.45; N, 3.43. Found: C, 67.81; H, 4.47; N, 3.48. $\delta_H(CDCl_3)$ 1.28 (t, 3H, J = 7.0 Hz, CH_3), 4.24 (q, 2H, J = 7.0 Hz, CH_2), 4.67 (s, 1H, CH), 6.90–7.41 (m, 12H, ArH). $\delta_C(CDCl_3)$: 13.90 (CH_3), 42.97 (CH), 63.34 (CH_2), 115.46 (CN), 118.46 (2C, ArC), 119.63 (2C, ArC), 120.09 (2C, ArC), 120.46 (2C, ArC), 124.15 (quat ArC), 129.44 (2C, ArC), 129.73 (2C, ArC), 151.88 (quat ArC), 152.26 (quat ArC), 152.97 (quat ArC), 156.14 (quat ArC), 158.68 (quat ArC), 164.99 (CO). MS, *m/z*: M^+, 407 (100); M + 2, 409 (35). IR (cm^{-1}): 1750 (CO), 2250 (CN).

1,4-Bis(ethyl {4-phenoxy}cyanoacetate)benzene (18). Photolysis resulted in 0.182 g (75%) of a yellowish oil. Anal. Calcd for $C_{28}H_{24}N_2O_6$: C, 69.41; H, 4.99; N, 5.78. Found: C, 69.44; H, 5.02; N, 5.74. $\delta_H(CDCl_3)$: 1.27 (t, 3H, J = 7.0 Hz, CH_3), 4.23 (q, 2H, J = 7.0 Hz, CH_2), 4.68 (s, 2H, CH), 6.98–7.00 (m, 4H, ArH), 7.02 (s, 4H, ArH), 7.38–7.42 (m, 4H, ArH). $\delta_C(CDCl_3)$: 13.85 (CH_3), 42.92 (CH), 63.29 (CH_2), 115.63 (CN), 118.52 (4C, ArC), 121.05 (4C, ArC), 124.21 (2C, quat ArC), 129.42 (4C, ArC), 153.23 (2C, quat ArC), 158.54 (2C, quat ArC), 164.95 (CO). MS, *m/z*: M^+, 484 (9.4). IR (cm^{-1}): 1750 (CO), 2250 (CN).

Acknowledgment. Financial support for this work provided by the Natural Sciences and Engineering Research Council of Canada (NSERC) and the University of Winnipeg is gratefully acknowledged. D.C.S. (graduate student, University of Manitoba) thanks NSERC for funding. We would also like to thank Dr. W. Danchura for technical assistance.