Notes

Synthesis of 1′-Substituted Derivatives of 1,2,3,4,5-Pentaphenylferrocene

David C. D. Butler and Christopher J. Richards*

Department of Chemistry, Queen Mary, University of London, Mile End Road, London, E1 4NS, U.K.

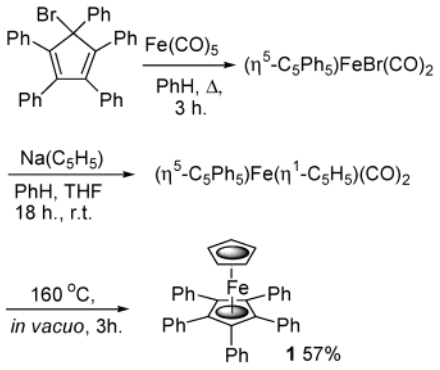
Received June 24, 2002

Summary: Pentaphenylferrocene was synthesized in 57% overall yield from 1-bromopentaphenylcyclopentadiene. Functionalization of the unsubstituted cyclopentadienyl ring using the Friedel−Crafts reaction gave 1′-formyl- and 1′-(2-chlorobenzoyl)-substituted derivatives. Hydrolysis of the latter provided the corresponding 1′-carboxylic acid. This was readily transformed via a modified Curtius rearrangement into 1′-amino-1,2,3,4,5-pentaphenylferrocene. This methodology also provided (η^5-aminocyclopentadienyl)(η^4-tetraphenylcyclobutadiene)-cobalt and gave an improved synthesis of aminoferrocene.

Introduction

Since its discovery, the chemistry of ferrocene has been prevalent within the literature, and it continues to be a subject of intense study.[1] Substituted derivatives such as pentamethylferrocene, which offer contrasting steric and redox properties, have also been extensively investigated.[2] Recently, phosphine-substituted derivatives of pentaphenylferrocene were reported as effective ligands in palladium-catalyzed transformations,[3] and enantiopure planar chiral pentaphenylferrocene derivatives have been utilized as nucleophilic catalysts in asymmetric synthesis.[4] These 1,2,3,4,5-pentaphenylferrocenes containing 1′- and 1′,2′-substituents, respectively, are prepared either by introduction of five phenyl groups onto a ferrocene precursor[3a,5] or by use of a prefunctionalized cyclopentadiene to construct the metallocene.[4d,g] Pentaphenylferrocene **1** itself has received hardly any attention, although some aspects of its physical chemistry have been explored.[6] In addition, there is a single report on the direct functionalization of this metallocene, although no experimental methods or spectroscopic results were presented.[7] We report herein an improved method for the preparation of pentaphenylferrocene **1** and describe the synthesis of 1′-substituted derivatives.[8] We additionally report on the facile synthesis of aminometallocenes, including an improved synthesis of aminoferrocene itself.

Scheme 1



Results and Discussion

Pentaphenylferrocene was prepared by a modification of the original synthesis by Pauson and McVey[9] employing a procedure with no requirement for the isolation and purification of air-sensitive intermediates (Scheme 1). This method provides a rapid and reliable synthesis of multigram quantities of **1** in an overall yield of 57%. The commercially available starting material 1-bromopentaphenylcyclopentadiene may also be prepared by an efficient literature method from inexpensive starting materials.[10]

* Corresponding author. E-mail: c.j.richards@qmul.ac.ul. Fax: +44 (0)207 882 7794.

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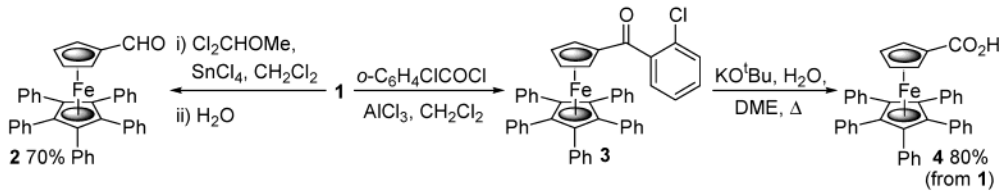
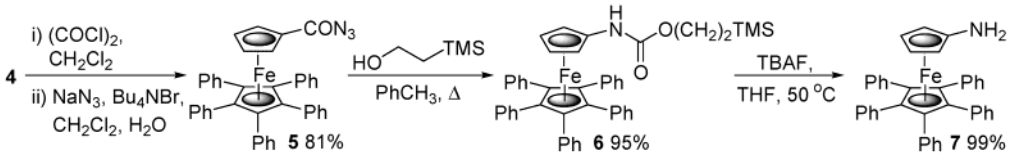
10.1021/om0204989 CCC: $22.00
Publication on Web 11/01/2002

Scheme 2



Scheme 3



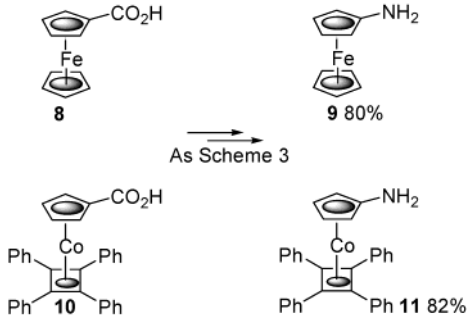
The initial step of the sequence involves refluxing a mixture of 1-bromopentaphenylcyclopentadiene and $Fe(CO)_5$ in benzene (3 h) to obtain a solution of crude (η^5-C_5Ph_5)$FeBr(CO)_2$, which is converted to insoluble (η^5-C_5Ph_5)(η^1-C_5H_5)$Fe(CO)_2$ by reaction with Na(η^5-C_5H_5) at ambient temperature. Subsequent pyrolysis of the crude product at 160 °C in vacuo gives a mixture containing **1** and 10−15% pentaphenylcyclopentadiene, even when moisture is rigorously excluded. Pure **1** can be obtained from this mixture by a single recrystallization from toluene. We have found that a larger amount of pentaphenylcyclopentadiene is produced when 1-bromopentaphenylcyclopentadiene is used in a concentration greater than 1 g per 20 cm^3 of solvent.

Functionalization of the unsubstituted cyclopentadienyl ring was achieved by employing the Friedel−Crafts reaction (Scheme 2). Addition of α,α-dichloromethyl-methyl ether to a cooled solution of **1** and $SnCl_4$ gave 1,2,3,4,5-pentaphenyl-1′-formylferrocene **2** in 70% yield. No formylation of the phenyl groups was noted using these conditions, with only **2** and starting material being isolated from the product mixture. Other Lewis acids ($AlCl_3$, $TiCl_4$) and formylating agents (e.g., phenylmethylformamide with $POCl_3$) gave much poorer results. A further substitution reaction was performed with 2-chlorobenzoyl chloride in the presence of $AlCl_3$. The resulting aryl ketone **3** was hydrolyzed as previously described for the synthesis of ferrocene carboxylic acid,[11] providing an excellent overall yield of pentaphenylferrocene carboxylic acid **4**.

It is noteworthy that all attempts to effect direct lithiation of **1** were unsuccessful, including the use of ^{t}BuLi at elevated temperatures or a superbasic mixture of BuLi and KO^tBu.[12] In all cases only starting material was recovered. In this context it has been reported that attempts to lithiate the related 18-electron complex (η^5-cyclopentadienyl)(η^4-tetraphenylcyclobutadiene)cobalt also failed, although this metallocene was successfully mercurated.[13] Repetition of this mercuration procedure with **1** resulted only in oxidation of pentaphenylferrocene by Hg^{2+}, as revealed by a change in the reaction mixture from yellow to dark orange-red with an accompanying deposition of elemental mercury. The process is reversible, and pentaphenylferrocene was reclaimed by reduction with sodium thiosulfate solution.

Without a metalation protocol, our attempts to generate further derivatives of **1** instead focused on acid **4**. Ferrocenecarboxylic acid **8** has previously been converted into aminoferrocene through formation of an intermediate acyl azide, followed by Curtius rearrangement in the presence of acetic anhydride and subsequent hydrolysis of the resulting *N*-acetyl aminoferrocene (30−37% yield).[14] Thus, the required acyl azide **5** was obtained cleanly in 81% yield from the acid chloride (prepared from the carboxylic acid with oxalyl chloride) by reaction with NaN_3 under phase transfer conditions (Scheme 3). The Curtius rearrangement was best achieved by heating **5** in the presence of 2-trimethylsilylethanol.[15] Carbamate **6** was formed cleanly and was quantitatively deprotected on treatment with excess TBAF to give amine **7** in 76% overall yield.

Scheme 4



The success of this reaction sequence prompted its repetition on acids **8** and **10**[16] to give the known and new amines **9** and **11**, again in excellent overall yield. Aminoferrocene **10** has been the subject of many synthetic investigations.[14,17] In addition to the Curtius protocol mentioned above it may be obtained in moderate (~40%)[17a] to low (12%)[17d] yield by direct lithiation of ferrocene followed by addition of a nitrogen electro-

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phile. Given both the ready availability of **8**[11] and its commercial availability, we believe the Curtius methodology for the synthesis of aminoferrocene represents a significant improvement over these existing methods. We found that the acyl azides synthesized were stable to normal manipulations in the solid state and in solution, although no direct investigation of their stability was performed.

All attempts to introduce a halogen onto the unsubstituted cyclopentadieyl ring of pentaphenylferrocene have so far proved unsuccessful. Due to its instability toward mild oxidizing agents, direct halogenation or mercuration of **1** or diazotization of **7** followed by the Sandmeyer reaction (even under nonaqueous conditions employing alkyl nitrites in place of strong acids) are not viable methods to obtain halopentaphenylferrocenes. However, the improved synthesis of **1** reported in this paper together with the generation of carbon- and nitrogen-substituted derivatives offers many possibilities for the incorporation of this bulky metallocene into new structures.

Experimental Section

All reactions were performed under an atmosphere of dinitrogen employing standard Schlenk techniques[18] in oven-dried (150 °C) glassware. In the following section, petroleum ether refers to the fraction boiling between 40 and 60 °C. CH_2Cl_2 and DME were distilled under nitrogen from CaH_2. Toluene was distilled under nitrogen from molten sodium. Benzene was distilled from sodium benzophenone ketyl. Column chromatography was performed on Matrix silica 60 (35−70 μm). 1-Bromopentaphenylcyclopentadiene was prepared as previously described.[10] All other reagents were obtained from commercial sources and used as received.

1,2,3,4,5-Pentaphenylferrocene, 1. 1-Bromopentaphenylcyclopentadiene (20.0 g, 38 mmol) was dissolved in benzene (400 mL). $Fe(CO)_5$ (8.9 g, 6.0 mL, 45 mmol) was added in a single portion, and the resulting purple solution was heated at reflux for a period of 3 h. On cooling, NaCp (40 mL of a 2.0 M solution in THF, 80 mmol) was added in a single portion and the mixture was stirred at room temperature for 18 h. The resulting orange precipitate was filtered and washed with toluene, dried, and heated at 160 °C in vacuo for 2 h. The tan solid thus obtained was dissolved in hot toluene (400 mL) and passed through a plug of silica to give a dark orange solution. Removal of the solvent by rotary evaporation and recrystallization from boiling toluene gave the title compound (12.3 g, 57% yield). Mp > 300 °C (lit.[9] 356−358 °C). Anal. Found: C, 84.87; H, 5.46. Calcd for $C_{40}H_{30}Fe$: C, 84.80; H, 5.34. ^{1}H NMR (δ; 400 MHz, $CDCl_3$): 4.05 (5 H, s, C_5H_5), 6.86−6.92 (20 H, m, Ar), 6.92−6.99 (5 H, m, Ar). $^{13}C\{^1H\}$ NMR (δ; 100 MHz, $CDCl_3$): 75.1 (C_5H_5), 87.9 (C_5Ph_5), 126.0 (Ar-para), 127.0 (Ar), 132.2 (Ar), 136.0 (Ar-ipso). MS (*m*/*z*; APCI): 567 ($[M + 1]^+$, 100%).

1,2,3,4,5-Pentaphenyl-1′-formylferrocene, 2. 1,2,3,4,5-Pentaphenylferrocene **1** (1.00 g, 1.8 mmol) was dissolved in dichloromethane (30 mL) and cooled in an ice bath. $SnCl_4$ (5.3 mL of a 1.0 M solution in dichloromethane) was added dropwise over 5 min, and the solution was stirred for a further 15 min. α,α-Dichloromethylmethyl ether (0.61 g, 0.48 cm^3, 5.3 mmol) was added over 1 min, whereupon the ice bath was removed and the dark mixture was stirred for 2 h followed by the additon of water (30 mL). The red organic fraction was separated from the aqueous fraction which was extracted with dichloromethane (15 mL). The combined organic fractions were dried ($MgSO_4$) and filtered, and the solvent was removed in vacuo. Column chromatography (CH_2Cl_2) gave starting material as an initial orange band (0.25 g) and a second red band, which yielded, after removal of the solvent, the title compound as a cherry-red solid (0.73 g, 70%). An analytical sample was obtained by recrystallization from CH_2Cl_2. Mp: 290 °C (dec). Anal. Found: C, 82.15; H, 5.15. Calcd for $C_{41}H_{30}FeO\cdot 1/4H_2O$: C, 82.21; H, 5.13. IR (ν_{max}; CH_2Cl_2): 1682 (C═O) cm^{-1}. ^{1}H NMR (δ; 400 MHz, $CDCl_3$): 4.55 (2 H, s, Fc-β), 4.84 (2 H, s, Fc-α), 6.98 (10 H, d, J = 7, Ar-ortho), 7.06 (10 H, t, J = 7, Ar-meta), 7.14 (5 H, t, J = 7, Ar-para), 9.87 (1 H, s, C*H*O). $^{13}C\{^1H\}$ NMR (δ; 100 MHz, $CDCl_3$): 74.8 (Cp), 79.7 (Cp), 81.7 (Cp-ipso), 88.6 (C_5Ph_5), 126.6 (Ar-para), 127.2 (Ar), 132.2 (Ar), 134.4 (Ar-ipso), 197.0 (*C*═O). MS (*m*/*z*; FAB): 594 (M^+, 100%), 566 (7), 501 (7). High-resolution MS (*m*/*z*, FAB): found for M^+ 594.1663; calcd for $C_{41}H_{30}FeO$, 594.1646.

1,2,3,4,5-Pentaphenyl-1′-(2-chlorobenzoyl)ferrocene, 3. 1,2,3,4,5-Pentaphenylferrocene **1** (6.05 g, 10.7 mmol) was dissolved in CH_2Cl_2 (200 mL) and the reaction vessel cooled in an ice bath. Granular $AlCl_3$ (1.50 g, 11.3 mmol) was added to the orange solution and stirred for 0.5 h at 0 °C, whereupon 2-chlorobenzoyl chloride (2.07 g, 1.50 mL, 11.8 mmol) was added dropwise via syringe over 1 min. The dark mixture was stirred at room temperature for 18 h, then poured onto ice. The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (2 × 20 mL). The combined organic fractions were dried ($MgSO_4$) and filtered, and the solvent was removed in vacuo to give a crimson solid. Column chromatography (1:1 CH_2Cl_2/petroleum ether) gave starting material as an orange band followed by a purple band, which yielded, after removal of solvent, the title compound as a red solid (6.84 g, 91%). An analytical sample was obtained by recrystallization from a mixture of CH_2Cl_2 and petroleum ether. Mp: 195−198 °C. Anal. Found: C, 76.75; H, 4.71. Calcd for $C_{47}H_{33}ClFeO\cdot 1/2CH_2Cl_2$: C, 76.32; H, 4.58. IR (ν_{max}; CH_2Cl_2): 1643 (C═O) cm^{-1}. ^{1}H NMR (δ; 400 MHz, $CDCl_3$): 4.55 (2 H, t, J = 3, Cp-β), 4.86 (2 H, t, J = 3, Cp-α), 6.14 (1 H, d, J = 11, Ar), 6.92 (10 H, d, J = 7, Ar-ortho), 7.00 (10 H, t, J = 7, Ar-meta), 7.05−7.08 (1 H, m, Ar), 7.11 (5 H, t, J = 7, Ar-para), 7.36 (2 H, d, J = 6, Ar). $^{13}C\{^1H\}$ NMR (δ; 100 MHz, $CDCl_3$): 79.9 (Cp), 83.0 (Cp), 88.5 (C_5Ph_5), 126.6 (Ph_5-para), 126.8 (Ar), 127.3 (Ph_5), 130.0 (Ar), 130.1 (Ar), 131.0 (Ar), 131.1 (Ar) 132.4 (Ph_5), 134.6 (Ph_5-ipso), 140.1 (Ar), 196.3 (*C*═O). MS (*m*/*z*; FAB): 706 (M^+, 37%), 704 (M^+, 100%), 566 (28%). High-resolution MS (*m*/*z*, FAB): found for M^+ 704.1580; calcd for $C_{47}H_{33}ClFeO$, 704.1569.

1,2,3,4,5-Pentaphenylferrocene-1′-carboxylic Acid, 4. 1,2,3,4,5-Pentaphenyl-1′-(2-chlorobenzoyl)ferrocene **3** (6.84 g, 9.7 mmol) was taken up in DME (100 mL). KO^{t}Bu (5.4 g 48 mmol) and H_2O (0.26 mL, 14 mmol) were added, and the resulting solution was heated at reflux for 1 h, cooled to room temperature, and acidified with 1 M HCl (50 cm^3). The tan solid obtained was separated by filtration, washed with water, and partially dried, and the solid was suspended in toluene (100 mL). Residual water was removed by azeotropic distillation until a volume of ca. 50 mL of toluene remained. The resulting suspension was cooled, and the orange solid was isolated by filtration. Drying in air then in vacuo afforded the title compound as a light orange powder (5.23 g, 88%). Mp: 267−270 °C (dec). Anal. Found: C, 80.71; H, 5.25. Calcd for $C_{41}H_{30}FeO_2$: C, 80.66; H, 4.95. IR (ν_{max}; CH_2Cl_2): 1724 (C═O, monomer) and 1678 (C═O, dimer) cm^{-1}. ^{1}H NMR (δ; 400 MHz, $CDCl_3$/DMSO-d_6): 4.32 (2 H, t, J = 2, Cp-β), 4.74 (2 H, t, J = 2, Cp-α), 6.90−7.25 (25 H, m, Ar). $^{13}C\{^1H\}$ NMR (δ; 100 MHz, $CDCl_3$): 75.7 (Cp), 77.2 (Cp-ipso), 78.1 (Cp), 88.0 (C_5Ph_5), 126.2 (Ar-para), 127.0 (Ar), 132.2 (Ar), 134.9 (Ar-ipso), 170.7 (*C*═O). MS (*m*/*z*; FAB): 610 (M^+, 100%).

1,2,3,4,5-Pentaphenyl-1′-acylazidoferrocene, 5. 1,2,3,4,5-Pentaphenylferrocene-1′-carboxylic acid **4** (2.00 g, 3.3 mmol) was suspended in CH_2Cl_2 (20 mL). Oxalyl chloride (0.84 g, 0.58 mL, 6.6 mmol) was added, followed by a drop of dimethylformamide. The mixture was stirred at room temperature for 3

(18) Shriver, D. F. *The Manipulation of Air-Sensitive Compounds*; McGraw-Hill: New York, 1969.

h, after which the solvent and residual oxalyl chloride were removed in vacuo. The red solid thus obtained was taken up in CH_2Cl_2 (20 mL). Tetrabutylammonium bromide (0.004 g, 0.01 mmol) was added, followed by a solution of NaN_3 (0.32 g, 4.9 mmol) in H_2O (4 mL), and the mixture was stirred rapidly at room temperature for 18 h. Additional water (50 mL) was added to the mixture, and the organic layer was separated. The aqueous layer was extracted with CH_2Cl_2 (2 × 20 mL), the combined organic layers were dried ($MgSO_4$) and filtered, and the solvent was removed in vacuo. Column chromatography (1:1 CH_2Cl_2/petroleum ether) gave the title compound as a red solid (1.69 g, 81%). Mp: 250 °C (dec). IR (ν_{max}; CH_2Cl_2): 2137 (N_3) 1682 (C═O) cm^{-1}. ^1H NMR (δ; 400 MHz, $CDCl_3$): 4.04 (2 H, t, $J = 2$, Cp-β), 4.76 (2 H, t, $J = 2$, Cp-α), 6.95−7.01 (20 H, m, Ar), 7.05−7.10 (5 H, m, Ar). $^{13}C\{^1H\}$ NMR (δ; 100 MHz, $CDCl_3$): 76.3 (Cp), 78.0 (Cp-ipso), 80.1 (Cp), 89.0 (C_5Ph_5), 127.7 (Ar-para), 127.7 (Ar), 132.7 (Ar), 134.9 (Ar-ipso), 176.1 (*C*═O). MS (*m*/*z*; FAB): 607 ($[M^+ - N_2]^+$, 100%). High-resolution MS (*m*/*z*, FAB): found for $[M - N_2]^+$ 607.1608; calcd for $C_{41}H_{29}FeNO$, 607.1598.

1,2,3,4,5-Pentaphenylferrocene-1′-(2-trimethylsilyl)-ethyl Carbamate, 6. 1,2,3,4,5-Pentaphenyl-1′-acylazideferrocene **5** (1.48 g, 2.3 mmol) was dissolved in toluene (15 mL), the soluton was heated to 105 °C, and 2-(trimethylsilyl)ethanol (0.67 mL, 4.7 mmol) was added in a single portion. The red solution obtained was stirred at this temperature for 3 h, by which time the color had changed to dark orange. The mixture was cooled to room temperature, NaOH (1 M, 50 mL) added, and the solution stirred for 5 min. The organic layer was separated and the aqueous layer extracted with CH_2Cl_2 (2 × 20 mL). The combined organic layers were dried ($MgSO_4$) and filtered, and the solvent was removed in vacuo to give an orange solid. Chromatography (1:1 CH_2Cl_2/petroleum ether) gave the title compund as an orange solid (1.61 g, 95%). An analytical sample (fine orange needles) was obtained by recrystallization from a mixture of CH_2Cl_2 and petroleum ether. Mp: 132−134 °C. Anal. Found: C, 75.65; H, 5.94. N, 1.97. Calcd for $C_{46}H_{43}FeNO_2Si \cdot 1/4H_2O$: C, 75.66; H, 6.00; N, 1.92. IR (ν_{max}; CH_2Cl_2): 1726 (C═O) cm^{-1}. ^1H NMR (δ; 400 MHz, $CDCl_3$): 0.0 (9 H, s, $Si(CH_3)_3$), 0.9 (2 H, m, CH_2TMS), 4.04−4.15 (4 H, m, Cp + OCH_2), 4.40 (2 H, brs, Cp), 5.70 (1 H, brs, NH), 7.00−7.20 (25 H, m, Ar). $^{13}C\{^1H\}$ NMR (δ; 100 MHz, $CDCl_3$): −1.4 ($Si(CH_3)_3$), 17.6 (CH_2TMS), 63.5 (Cp), 67.9 (Cp), 72.4 (OCH_2), 87.8 (C_5Ph_5), 96.0 (Cp-ipso), 126.2 (Ar-para), 127.2 (Ar), 132.2 (Ar), 135.5 (Ar-ipso), 154.7 (*C*═O). MS (*m*/*z*; FAB): 725 (M^+, 100%), 698 (31), 625 (45), 581 (27), 501 (25). High-resolution MS (*m*/*z*, FAB): found for M^+ 725.2440; calcd for $C_{46}H_{43}FeNO_2Si$, 725.2412.

1,2,3,4,5-Pentaphenyl-1′-aminoferrocene, 7. 1,2,3,4,5-Pentaphenylferrocene-1′-(2-trimethylsilyl)ethyl carbamate **6** (1.55 g, 2.1 mmol) was dissolved in a 1 M solution of tetrabutylammonium fluoride in THF (8.6 mL, 8.6 mmol) and the solution heated to 50 °C for 15 min. The solvent was removed in vacuo, then water (50 mL) and CH_2Cl_2 (50 mL) were added. The organic layer was separated, and the aqueous layer extracted with CH_2Cl_2 (2 × 20 mL). The combined organic layers were dried ($MgSO_4$) and filtered through silica, and the solvent was removed in vacuo to give the title compound as an orange solid (1.23 g, 99%). An analytical sample was obtained by recrystallization from a mixture of CH_2Cl_2 and petroleum ether. Mp: 270−275 °C (dec). IR (ν_{max}; CH_2Cl_2): 3690 (NH_2), 1601, 1502 cm^{-1}. ^1H NMR (δ; 400 MHz, $CDCl_3$): 2.50 (2 H, brs, NH_2), 3.80 (2 H, brs, Cp), 3.90 (2 H, brs, Cp), 6.90−7.20 (25 H, m, Ar). $^{13}C\{^1H\}$ NMR (δ; 100 MHz, $CDCl_3$): 64.1 (Cp), 71.3 (Cp), 87.5 (C_5Ph_5), 107.2 (Cp-ipso), 125.9 (Ar-para), 127.1 (Ar), 132.2 (Ar), 136.1 (Ar-ipso). MS (*m*/*z*; FAB): 581 (100%, M^+), 242 (87). High-resolution MS (*m*/*z*, FAB): found for M^+ 581.1833; calcd for $C_{40}H_{31}FeN$, 581.1806.

Aminoferrocene, 9. Using the same procedure, ferrocenecarboxylic acid **8** (2.29 g, 10.0 mmol) was converted via the corresponding acyl azide (IR (ν_{max}; CH_2Cl_2) 2135 (N_3), 1684 (C═O) cm^{-1}) into **9** obtained as a brown solid (1.61 g, 80%). Mp: 126−130 °C (dec) (lit.[17c] 148−150 °C). IR (ν_{max}; CH_2Cl_2): 3757 and 3692 (NH_2) cm^{-1}. ^1H NMR (δ; 400 MHz, $CDCl_3$): 2.60 (2 H, brs, NH_2); 3.85 (2 H, brs, Fc), 4.00 (2 H, brs, Cp), 4.09 (5 H, s, C_5H_5). $^{13}C\{^1H\}$ NMR (δ; 100 MHz, $CDCl_3$): 58.8 (Fc) 63.5 (Fc), 68.9 (C_5H_5), 105.6 (Fc-ipso).

(η^5-Aminocyclopentadienyl)(η^4-tetraphenylcyclobutadiene)cobalt, 11. Using the same procedure acid **10**[16] (3.18, 6.1 mmol) was converted via the corresponding acyl azide (IR (ν_{max}; CH_2Cl_2) 2137 (N_3), 1682 (C═O) cm^{-1}) into **11** obtained as a dark orange-brown crystalline solid (2.46 g, 82%). Mp: 212−215 °C. IR (ν_{max}; CH_2Cl_2): 3688 (NH_2), 1597, 1501 cm^{-1}. ^1H NMR (δ; 400 MHz, $CDCl_3$): 2.16 (2 H, brs, NH_2), 4.13 (2 H, t, $J = 2$, Cp), 4.39 (2 H, t, $J = 2$, Cp), 7.15−7.30 (12 H, m, Ar), 7.35−7.50 (8 H, m, Ar). $^{13}C\{^1H\}$ NMR (δ; 100 MHz, $CDCl_3$): 72.6, 74.6, 78.1 (2 × Cp and C_4H_4), 113.3 (Cp-ipso), 125.8 (Ar-para), 128.0 (Ar), 128.6 (Ar), 136.7 (Ar-ipso). MS (*m*/*z*; FAB): 495 (M^+, 84%), 415 (100). High-resolution MS (*m*/*z*, FAB): found for M^+ 495.1413; calcd for $C_{33}H_{26}CoN$, 495.1397.

Acknowledgment. We wish to thank the EPSRC for financial support (GR/M55893) and the EPSRC National Mass Spectrometry Service Centre, University of Wales, Swansea.

OM0204989