Supplementary Material (ESI) for Green Chemistry

Supporting Information

Supporting Information for Green Chemistry

Pier Giorgio Cozzi* and Luca Zoli.

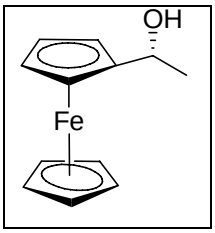
General: ^{1}H NMR spectra were recorded on Varian 200 MHz or Varian 300 MHz spectrometers. chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (deuterochloroform: δ 7.27 ppm). Data are reported as follows: chemical shifts, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants (Hz). ^{13}C NMR spectra were recorded on a Varian 50 MHz or Varian 75 MHz spectrometers with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard (deuterochloroform: δ 77.0 ppm). Mass spectra were performed at an ionizing voltage of 70 eV. Chromatographic purification was done with 240-400 mesh silica gel. Analytical gas chromatography (GC) was performed on a Hewlett-Packard HP 6890 gas chromatograph with a flame ionization detector and split mode capillary injection system, using a Crosslinked 5% PH ME Siloxane (30 m) column or a Megadex5 chiral (25 m) column. Elemental analyses were carried out by using a EACE 1110 CHNOS analyzer. Analytical high performance liquid chromatograph (HPLC) was performed on a HP 1090 liquid chromatograph equipped with a variable wavelength UV detector (deuterium lamp 190-600 nm), using Daicel Chiralcel^{TM} OD column (0.46 cm I.D. x 25 cm) (Daicel Inc.), Daicel Chiralcel^{TM} AD column (0.46 cm I.D. x 25 cm) (Daicel Inc.), Daicel Chiralcel^{TM} OF column (0.46 cm I.D. x 25 cm) (Daicel Inc.), Daicel Chiralcel^{TM} OJ column (0.46 cm I.D. x 25 cm) (Daicel Inc.); HPLC grade isopropanol and *n*hexane were used as the eluting solvents. All the reactions were carried out in deionised water under air, apart from the reactions in which pyrrole was used as nucleophile. Enantioenriched (95 % ee) ferrocenyl alcohol **1** was obtained from Johnson Matthey, and was prepared by the reduction of acetyl ferrocene with PPHOS ligand.[1] Enantioenriched ferrocenyl alcohol **2** was prepared according to the procedures described by Knochel[2] using the CBS (Corey-Bakshi-Shibata) protocol.[3] The absolute configuration of the products was established by

[1] W.-S. Lam, S. H. L. Kok, T. T.-L. Au-Yeung, J. Wu, H.-Y. Cheung, F.-L. Lam, C.-H. Yeung, A. S. C. Chan, *Adv. Synt. Catal.* **2006**, *348*, 370.
[2] K. Tappe, P. Knochel, *Tetrahedron:Asymmetry* **2004**, *15*, 91, and ref. therein.
[3] E. J. Corey, R. K. Bakshi, S. Shibata, *J. Am. Chem. Soc.* **1987**, *109*, 7925.

comparison with the HPLC elution order and/or $[\alpha]_D$ values of products reported in literature, or assumed by analogy.[4]

[4] P. Vicennati, P. G. Cozzi, *Eur. J. Org. Chem.* **2007**, 2248.

(*R*)-(1-hydroxyethyl)ferrocene (1).



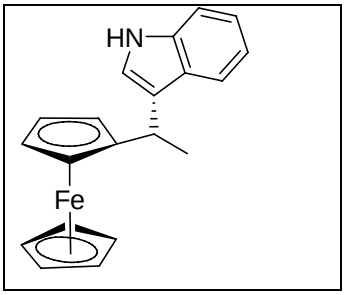
$C_{12}H_{14}FeO$ Fw = 230.04

$[\alpha]_D$ = -28.3° (c 0.16, $CHCl_3$)

Analytical data, see: W.-S. Lam, S. H. L. Kok, T. T.-L. Au-Yeung, J. Wu, H.-Y. Cheung, F.-L. Lam, C.-H. Yeung, A. S. C. Chan, *Adv. Synt. Catal.* **2006**, *348*, 370-374.

HPLC analysis OJ: isocratic, flux 0.5mL/m (hexane: *i*-PrOH) 85:1. TM: 12.61 min; tm: 13.86 min.

(*S*)-1-(3-indolethyl)ferrocene (4a).



$C_{20}H_{19}FeN$ Fw = 329.09

R_f = 0.3 (Cyclohexane/Diethylether 8/2)

$[\alpha]_D$ = -53° (c 0.6, $CHCl_3$)

Yellow solid, mp = 133 °C.

IR: 3440, 3089, 1618 cm^{-1}

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 7.84 (bs, 1H), 7.69 (d, J = 7.2 Hz, 1H), 7.35 (dd, J = 1.4, 7.2 Hz, 1H), 7.16 (dq, J = 1.4, 7.2 Hz, 2H), 6.79 (s, 1H), 4.35-4.05 (m, 9+1 H), 1.73 (d, J = 7.0 Hz, 3H).

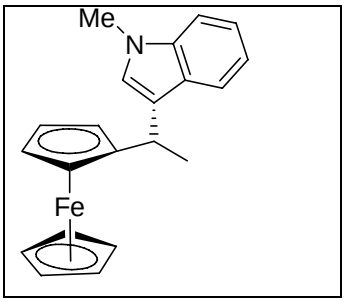
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 136.4, 126.6, 123.4, 121.9, 120.8, 119.4, 119.2, 111.3, 95.6, 69.3 (5C), 68.6, 67.8, 67.3, 67.0, 30.8, 21.8.

ESI MS: 330 (M+1), 329 (M), 213.

HPLC analysis AD: isocratic, flux 0.8mL/m (hexane: *i*-PrOH) 85:15. TM: 10.41 min; tm: 11.47 min.

Elemental Analysis calcd for $C_{20}H_{19}FeN$: C, 72.97. H, 5.82. N, 4.25. Found C, 73.03. H, 5.92. N, 4.31.

(*R*)-1-[3-(1-*N*-methylindole)ethyl]ferrocene (4b).



$C_{21}H_{21}FeN$ Fw = 343.1

R_f = 0.5 (Cyclohexane/Diethylether 95/5)

$[\alpha]_D$ = - 94° (c 0.38, $CHCl_3$)

Yellow solid, mp = 104-107 °C

IR: 3415, 3085, 1467 cm^{-1}

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.67 (d, J = 7.8 Hz, 1H), 7.30-7.10 (m, 3H), 6.60 (s, 1H), 4.45-4.05 (m, 9+1 H), 3.72 (s, 3H), 1.72 (d, J = 7.2 Hz, 3H).

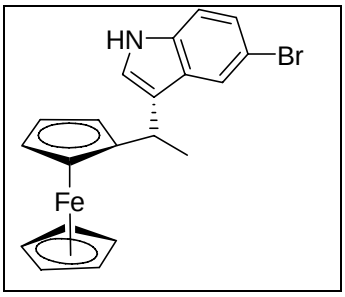
^{13}C-NMR ($CDCl_3$, 75 MHz) δ: 137.3, 127.1, 125.8, 122.1, 121.7, 119.7, 118.8, 109.5, 95.3, 69.0 (5C), 68.6, 67.7, 67.1, 66.7, 32.9, 30.9, 22.2.

ESI MS: 344 (M+1), 343 (M).

HPLC analysis AD: isocratic, flux 0.7mL/m (hexane: *i*-PrOH) 85:15. TM: 6.25 min; tm: 7.06 min.

Elemental Analysis calcd for CHFeN: C, 69.89. H, 5.52. Found C, 69.70. H, 5.42.

(*R*)-1-[3-(5-bromoindole)ethyl]ferrocene (4c).



$C_{20}H_{18}BrFeN$ Fw = 408.54

R_f = 0.31 (Cyclohexane/Diethylether 8/2)

$[\alpha]_D$ = -58° (c 0.45, $CHCl_3$)

Dark yellow solid, mp = 163 °C

IR: 3414, 3093, 1458 cm^{-1}

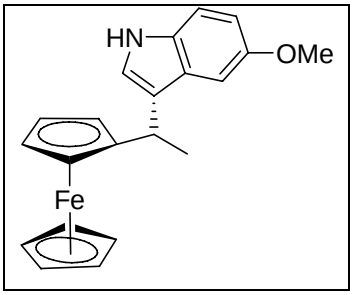
^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.87 (bs, 1H), 7.78 (s, 1H), 7.31-7.15 (m, 2H), 6.74 (s, 1H), 4.37 (m, 9H), 4.06 (bs, 1H), 1.69 (d, J = 5.7 Hz, 3H).

^{13}C-NMR ($CDCl_3$, 75 MHz) δ: 135.1, 128.5, 124.9, 123.3, 122.2, 112.9, 112.7, 102.6, 95.7, 69.7 (5C), 68.9, 68.4, 67.7, 67.2, 30.8, 21.9.

ESI MS: 409 (M+1), 408 (M).

HPLC analysis AD: isocratic, flux 0.7mL/m (hexane: *i*-PrOH) 85:15. TM: 10.97 min; tm: 11.71 min. Elemental Analysis calcd for $C_{20}H_{18}BrFeN$: C, 58.86. H, 4.45. N, 3.43. Found C, 58.90. H, 4.49. N, 3.40.

(*R*)-1-[3-(5-Methoxyindole)ethyl]ferrocene (4d).



$C_{21}H_{21}FeNO$ Fw = 359.24

R_f = 0.31 (Cyclohexane/Diethylether 8/2)

$[\alpha]_D$ = -57° (c 0.35, $CHCl_3$)

Yellow oil.

IR:3414, 3091, 1622, 1581, 1210, 1172 cm^{-1}

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.74 (bs, 1H), 7.22 (d, J = 8.9 Hz, 1H), 7.07 (d, J = 1.5 Hz, 1H), 6.83 (dd, J = 8.9, 1.5 Hz, 1H), 6.64 (s, 1H), 4.23 (m, 9H), 4.09 (q, 1H), 3.87 (s, 3H), 1.69 (d, J = 6.7 Hz, 3H).

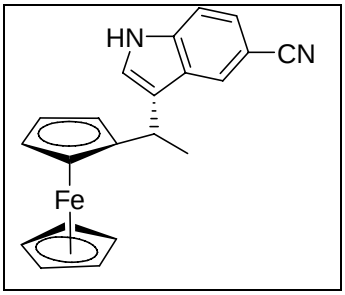
^{13}C-NMR ($CDCl_3$, 75 MHz) δ: 153.7, 131.5, 126.8, 123.0, 121.5, 111.9, 111.7, 101.4, 95.0, 68.8 (5C), 68.2, 67.5, 66.9, 66.6, 56.0, 30.6, 21.6.

HPLC analysis AD: isocratic, flux 0.8mL/m (hexane: *i*-PrOH) 85:15. TM: 15.47 min; tm: 18.52 min.

ESI MS: 360 (M+1), 359 (M).

Elemental Analysis calcd for $C_{21}H_{21}FeNO$: C, 70.21. H, 5.89. N, 3.90. Found C, 70.26. H, 5.91. N, 3.92.

(*R*)-1-[3-(5-Cyanoindole)ethyl]ferrocene (4e).



$C_{21}H_{18}FeN_2$ Fw = 354.2

R_f = 0.31 (Cyclohexane/Diethylether 1/1)

$[\alpha]_D$ = -27° (c 0.25, $CHCl_3$)

Yellow solid, mp = 76-78 °C

IR: 3387, 2913, 1094 cm^{-1}

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 8.13 (bs, 1H), 7.90 (s, 1H), 7.28 (s, 2H), 6.80 (s, 1H), 4.10-3.90 (m, 9H + 1H), 1.60 (d, J = 6.8 Hz, 3H).

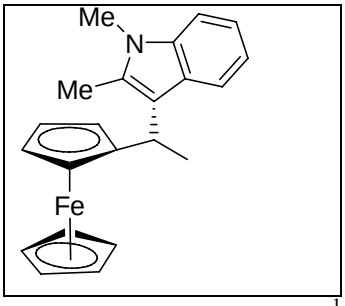
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 137.9, 126.1, 125.0, 124.6, 123.9, 122.8, 121.0, 112.0, 101.9, 94.5, 69.1 (5C), 68.3, 67.9, 67.4, 66.6, 30.5, 21.5.

ESI MS: 355 (M+1), 354 (M). 344 (M+1), 343.

HPLC analysis AD: isocratic, flux 0.9mL/m (hexane: *i*-PrOH) 85:15. TM: 12.83 min; tm: 12.86 min.

Elemental Analysis calcd for $C_{21}H_{18}FeN_2$: C, 71.20. H, 5.12. N, 7.91 Found C, 71.15. H, 5.10. N, 7.94.

(*R*)-1-[3-(1-*N*-methy-2-methylindole)ethyl]ferrocene (4g).



$C_{22}H_{23}FeN$ Fw = 357.26
R_f = 0.5 (Cyclohexane/Diethylether 95/5)
$[\alpha]_D$ = -171° (c 0.98, $CHCl_3$)
orange solid, mp = 105 °C

IR: 3415, 3085, 1467 cm^{-1}

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 7.35 (d, J = 6.8 Hz, 1H), 7.13 (d, J = 8.1 Hz, 1H), 7.00 (dd, J = 6.8 Hz, 8.1 Hz, 1H), 6.88 (dd, J = 8.1 Hz, 6.8 Hz, 1H), 4.35 (m, 1H), 4.20 (q, J = 7.2 Hz, 1H), 4.06 (m, 5H), 4.02 (m, 1H), 3.94 (m , 2H), 3.54 (s, 3H), 2.26 (s, 3H), 1.59 (d, J = 7.20 Hz, 3H).

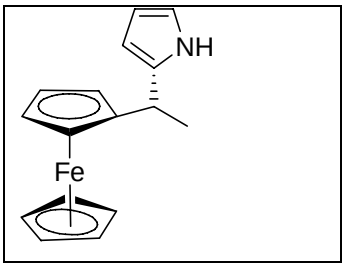
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 136.5, 131.3, 126.2, 120.1, 119.2, 118.3, 116.3, 108.4, 94.7, 68.6 (5C), 67.5 (2C), 66.9, 66.4, 31.1, 29.3, 20.9, 10.6.

ESI MS: 357 (M+1), 356 (M).

HPLC analysis OD: ramp, flux 0.6mL/m (hexane: *i*-PrOH) from 99:1 to 90:10 in 20min. TM: 14.79 min; tm: 19.94 min.

Elemental Analysis calcd for $C_{22}H_{23}FeN$: C, 73.96. H, 6.49. N, 3.92. Found C, 74.00. H, 6.54. N, 3.89.

(*R*)-1-(2-Pyrrolethyl)ferrocene (4h).



$C_{16}H_{17}FeN$ Fw = 279.15

R_f = 0.5 (Cyclohexane/Diethylether 8/2)

$[\alpha]_D$ = -46° (c 0.8, $CHCl_3$)

orange solid, mp = 94 °C.

IR: 3440, 3089, 1618 cm^{-1}

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 7.93 (bs, 1H), 6.58 (s, 1H) 6.08 (d, J = 2.8 Hz , 1H), 5.90 (s, 1H), 4.10-4.01 (m, 9H), 3.84 (q, J = 7.0 Hz, 1 H), 1.58 (d, J = 7.0 Hz, 3H).

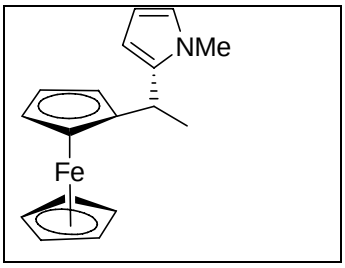
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 136.7, 115.8, 107.9, 103.8, 93.1, 68.5 (5C), 67.6, 67.4, 67.3, 66.0, 32.1, 21.2.

ESI MS: 280 (M+1), 279 (M).

HPLC analysis AD: ramp, flux 0.6mL/m (hexane: *i*-PrOH) from 99.5:0.5 to 80:20 in 20 min. TM: 15.07 min; tm: 19.55 min.

Elemental Analysis calcd for $C_{16}H_{17}FeN$: C, 68.84. H, 6.14.N, 5.02. Found C, 68.80. H, 6.18. N, 5.00.

(*R*)-1-[3-(1-*N*-methylpyrrole)ethyl]ferrocene (4i).



$C_{17}H_{19}FeN$ Fw = 293.18

R_f = 0.34 (Cyclohexane/Dichloromethane 3/1)

$[\alpha]_D$ = +31° (c 0.8, $CHCl_3$)

orange solid, mp = 51 °C

IR: 3096, 2925, 1488, cm^{-1}

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.53 (d, J = 1.8 Hz, 1H), 6.08 (dd, J = 2.9 Hz, 1H), 5.85 (d, J = 1.8 Hz, 1H), 4.20-4.14 (m, 5H), 4.13-4.07 (s, 3H), 4.08-4.00 (s, 1H), 3.86 (q, J = 7.1 Hz, 1H), 3.61 (s, 3H), 1.68 (d = 7.1 Hz, 3H).

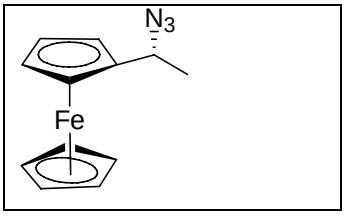
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 138.1, 120.9, 106.4, 105.0, 94.2, 68.4 (5C), 67.5, 67.2, 66.7 (2C), 33.8, 30.6, 21.3.

ESI MS: 294 (M+1), 293 (M), 213.

HPLC analysis OJ: ramp, flux 0.8mL/m (hexane: *i*-PrOH) from 99.5:0.5 to 80:20. TM: 12.41 min; tm: 16.05 min.

Elemental Analysis calcd for $C_{17}H_{19}FeN$: C, 69.64. H, 6.53. N, 4.78. Found C, 69.60. H, 6.58. N, 4.80.

(*R*)-(1-azidoethyl)ferrocene (4j).



$C_{12}H_{13}FeN_3$ Fw = 255.1
R_f =0.6 (Cyclohexane/Diethylether 95/5)
$[\alpha]_D$ = -48° (c 3.2, $CHCl_3$)
Orange oil

IR: 3092, 2097 cm^{-1}

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 4.38 (q, J = 6.6 Hz, 1H), 4.24 (m, 9H), 1.59 (d, J = 6.6 Hz, 3H).

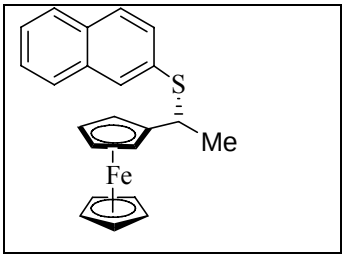
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 89.1, 69.2 (5C), 68.5 (2C), 67.7 (2C), 57.2, 20.1.

ESI MS: 255.9 (M+1), 254.9 (M), 230.

HPLC analysis OJ: ramp, flux 0.7mL/m (hexane: *i*-PrOH) from 99:1 to 97:3 in 20min. TM: 9.65 min; tm: 10.32 min.

Elemental Analysis calcd for $C_{12}H_{13}FeN_3$: C, 56.50. H, 5.14. N, 16.47. Found C, 56.30. H, 5.04. N, 16.30.

(*R*)-[1-(2-thionaphtyl)ethyl]ferrocene (4l).



$C_{22}H_{20}FeS$ Fw = 372.30
R_f = 0.65 (hexane)
$[\alpha]_D$ = -50° (c 2.3 , $CHCl_3$)
orange solid, mp = 64-69°C

IR: 3056, 1454 cm^{-1}

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 7.806-7.75 (m, 4H), 7.52-7.44 (m, 3H), 4.29 (q, J = 6.9 Hz, 1H) 4.23-4.10 (m, 9H), 1.71 (d, J = 6.9 Hz, 3H).

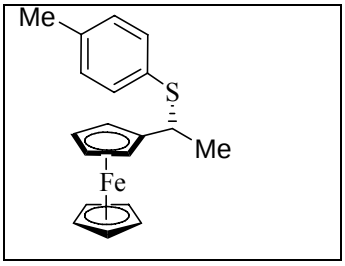
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 133.7, 132.9, 132.5, 131.8, 130.6, 128.3, 127.8, 127.6, 126.5, 126.2, 91.0, 68.9 (5C), 68.1, 67.9, 67.8, 66.3, 43.7, 21.4.

ESI MS: not ionisable.

HPLC analysis OJ: ramp, flux 0.6mL/m (hexane: *i*-PrOH) from 99:1 to 80:20 in 20min. TM: 27.2 min; t: 33.7 min.

Elemental Analysis calcd for $C_{22}H_{20}FeS$: C, 70.97 H, 5.41. Found C, 70.90. H, 5.38.

(*R*)-[1-(4-methylthiophenol)ethyl]ferrocene (4m).



$C_{19}H_{20}FeS$ Fw = 336.21
R_f = 0.16 (hexane)
$[\alpha]_D$ = -68° (c 1.04, $CHCl_3$)
Yellow oil

IR: 2916, 1499 cm^{-1}

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 7.28 (d, J = 7.8 Hz, 2H), 7.10 (d, J = 7.8 Hz, 2H), 4.25-3.99 (m, 10H), 2.36 (s, 3H), 1.63 (d, J = 6.8 Hz, 3H).

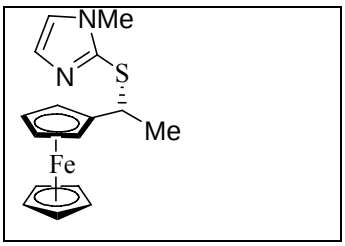
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 137.4, 133.8 (2C), 131.4, 129.4 (2C), 91.0, 68.6 (5C), 67.9, 67.6, 67.5, 66.0 44.0, 21.1 (2C).

ESI MS: 337 (M+1), 336 (M).

HPLC analysis OJ: isocratic, flux 0.8mL/m (hexane: *i*-PrOH) 98:2. TM: 14.7 min; t: 15.9 min.

Elemental Analysis calcd for $C_{22}H_{20}FeS$: C, 70.97 H, 5.41. Found C, 70.90 . H, 5.37.

(*R*)-[1-(*N*-methyl-(3-thioimidazole)ethyl]ferrocene (4n).



$C_{16}H_{18}FeN_2S$ Fw = 326.23.

R_f = 0.65 (hexane)

$[\alpha]_D$ = -66° (c 1.14, $CHCl_3$)

Yellow solid, mp = 139-142 °C

IR: 3117, 2929, 1560, 1523, cm^{-1}

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 6.51 (d, J = 2.4 Hz, 1H), 6.40 (d, J = 2.4 Hz, 1H), 5.93 (q, J = 6.8 Hz, 1H), 4.30-4.15 (m, 9H) 3.59 (s, 3H), 1.69 (d, J = 6.8 Hz, 3H).

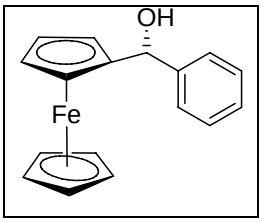
^{13}C-NMR ($CDCl_3$, 75 MHz) δ: 160.0, 117.5, 113.9, 87.6, 69.0 (5C), 68.9, 68.7, 67.8, 65.9, 52.6, 34.8, 19.3.

ESI MS: 327 (M+1), 326 (M), 213.

HPLC analysis OJ: isocratic, flux 0.6mL/m (hexane: *i*-PrOH) 80:20. TM: 24.8 min; t: 31.1 min.

Elemental Analysis calcd for: $C_{16}H_{18}FeN_2S$ C, 58.91 H, 5.56. N, 8.59. Found C, 58.87. H, 5.69. N, 8.61.

(*R*)-(3-Hydroxyphenylmethyl)ferrocene (2).



$C_{17}H_{16}FeO$ Fw = 292.06

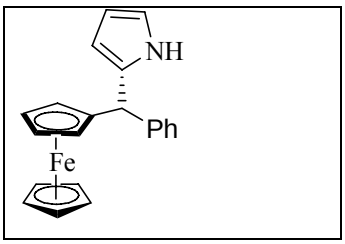
$[\alpha]_D$ = -92.5° (c 1.2, $CHCl_3$)

Analytical data for (*S*)-(3-Hydroxyphenylmethyl)ferrocene; see: K. Tappe, P. Knochel, *Tetrahedron: Asymmetry* **2004**, *15*, 91.

(HPLC analysis OD: ramp, flux 0.6mL/m (hexane: *i*-PrOH) from 99:1 to 90:10 in 20 min. TM: 25.24 min; tm: 31.48 min. 94%ee.

Elemental Analysis calcd for $C_{17}H_{16}FeO$: C, 69.89. H, 5.52. Found C, 69.70. H, 5.42.

(*R*)-(2-pyrrolephenylmethyl)ferrocene (5h).



$C_{21}H_{19}FeN$ Fw = 341.22

R_f = 0.44 (Cyclohexane/Diethylether 5/1)

$[\alpha]_D$ = +11° (c 1.15, $CHCl_3$)

Yellow solid, mp= 125 °C

IR: 3419, 3092, 2926, 1693, 1451 cm^{-1}

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 8.03 (bs, 1H), 7.38-7.21 (m, 5H), 6.68 (m, 1H) 6.15 (m, 1H), 5.89 (m, 1H), 5.15 (s, 1H), 4.28-4.00 (m, 9H).

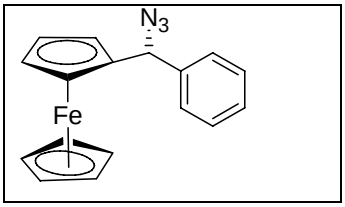
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 143.9, 134.7, 128.7 (2C), 128.4 (2C), 126.7, 116.5, 108.2, 106.7, 91.1, 68.9, (5C), 68.7, 68.1, 68.0, 67.5, 45.2.

ESI MS: 342 (M+1), 341 (M), 339.

HPLC analysis AD: ramp, flux 0.6mL/m (hexane: *i*-PrOH) from 99.5:0.5 to 80:20 in 20 min. TM: 15.72 min; tm: 19.45 min.

Elemental Analysis calcd for $C_{21}H_{19}FeN$: C, 73.92. H, 5.61. N, 4.10. Found C, 73.95. H, 5.66. N, 4.06.

(*R*)-(3-azidophenylmethyl)ferrocene (5j).



$C_{17}H_{15}FeN_3$ Fw = 317.16

R_f = 0.65 (hexane)

$[\alpha]_D$ = -44° (c 2.00 , $CHCl_3$)

Red oil

IR: 3089, 2929, 2082 1458 cm^{-1}

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.45-7.30 (m, 5H), 5.45 (s, 1H), 4.34-4.04 (m, 9H).

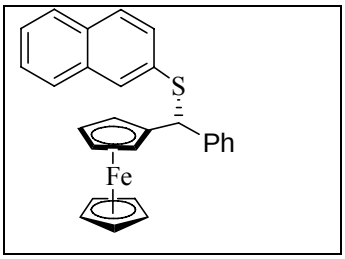
^{13}C-NMR ($CDCl_3$, 75 MHz) δ: 139.7, 128.5 (2C), 128.2, 127.4 (2C), 88.6, 69.0 (5C), 68.3, 68.1, 67.3, 67.2, 65.7.

ESI MS: 565.8 (D-N_2), 317 (M), 292, 276 (M-N_3)

HPLC analysis OD: ramp, flux 0.6mL/m (hexane: *i*-PrOH) from 99.5:0.5 to 98:2 in 20min. TM: 17.88 min; tm: 28.53 min.

Elemental Analysis calcd for $C_{17}H_{15}FeN_3$: C, 64.38. H, 4.77. N, 13.25. Found C, 64.42. H, 4.78. N, 13.20.

(*R*)-[(2-thionaphtyl)phenylmethyl]ferrocene (5l).



$C_{27}H_{22}FeS$ Fw = 434.26

R_f = 0.65 (hexane)

$[\alpha]_D$ = -19° (c 1.84, $CHCl_3$)

Yellow solid, mp = 123 °C

IR: 1490, 11450 cm^{-1}

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 7.77-7.63 (m, 4H), 7.45-7.41 (m, 3H), 7.34-7.21 (m, 5H), 5.26 (s, 1H), 4.20-4.13 (m, 9H).

^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 142.1, 133.6, 133.3, 132.3, 131.2, 130.0, 128.5 (2C), 128.3 (2C), 128.1, 127.7, 127.6, 127.3, 126.3, 126.1, 89.7, 69.2 (5C), 68.7, 68.3, 67.9, 67.8, 54.4.

ESI MS: 434.9 (M+1), 434.0 (M), 275.

HPLC analysis OJ: ramp, flux 0.6mL/m (hexane: *i*-PrOH) from 99:1 to 80:20 in 20 min. TM: 19.34 min; tm: 23.54 min.

Elemental Analysis calcd for $C_{27}H_{22}FeS$: C, 74.66. H, 5.10. Found C, 74.70. H, 5.14.