Supporting Information.

Fine-Tuning of Ferrocene Redox Potentials Towards Multiplex DNA Detection.

Barrie J. Marsh, Lauren Hampton, Sean Goggins and Christopher G. Frost*.

Department of Chemistry, University of Bath, Bath, BA2 7AY, UK

Atlas, Derby Court, Epsom Square, White Horse Business Park, Trowbridge, BA14 0XG, UK.

Table Of Contents:

General Considerations 2
Etherification of ferrocene methanol with various diols 3
Acylation of ferrocene derived alcohols 7
NMR data for ferrocene alcohols 11
NMR data for ferrocene esters 28
1. General.

All reactions were carried out using oven dried glassware under and inert atmosphere of nitrogen unless otherwise stated. Solvents were dried prior to use using an Innovative Technology Inc. PS-400-7 solvent purification system. Reagents were used as supplied without further purification either from Sigma Aldrich Company Ltd or Alfa Aesar Ltd. Analytical thin layer chromatography was performed using commercial available aluminium backed plates coated with ALUGRAM® Xtra SIL G/UV254 from Macherey-Nagel Ltd. Plates were visualised under ultraviolet light (254 nm) or by chemical staining with potassium permanganate stain followed by gentle heating. Column chromatography was performed using silica gel 60Å pore size, 200-440 mesh particle size from Sigma Aldrich Company Ltd. The $^1$H and $^{13}$C NMR spectra were recorded on a Brüker Avance 300 Mhz instruments. Chemical shifts are recorded in ppm and reference internally to C$_6$H$_6$ or CHCl$_3$ (as stated) at 7.16 and 7.26 ppm respectively for $^1$H NMR and 128.1 or 77.0 ppm for $^{13}$C NMR respectively. Mass spectrum analysis were recorded on a µTOF electrospray time of flight mass spectrometer (ESI-TOF). IR spectra were recorded on a Perkin Elmer 1600 FT IR spectrometer with selected absorbances quoted as $\delta$ cm$^{-1}$. Melting points were recorded using a Buchi 535 melting point apparatus. Ferrocene methanol was synthesised via literature procedure.$^1$
2. General Procedure A for etherification of ferrocene methanol with various diols.

Ferrocene methanol (1 eq) was dissolved in appropriate diol (5 ml/mmol or 10 eql) and then treated with ytterbium triflate (5 mol%). The reaction was stirred at room temperature until TLC analysis showed full conversion. The reaction mixture was then diluted with ethyl acetate (20 mL) and the organics then washed with water (20 mL) and brine (sat.) (20 mL). The organic layer was then dried over MgSO\(_4\), then filtered and concentrated \textit{in vacuo}. Purification was then carried out by silica-gel chromatography eluting with hexane 1:1 ethyl acetate to give the desired product.

2-(Ferrocenyloxy)ethanol (1).

Using general procedure A with ferrocene methanol (545 mg, 2.5 mmol), ytterbium triflate (77 mg, 5 mol%) and ethylene glycol (10 mL). This gave the desired product as a yellow oil (389 mg, 59%). IR; \(\nu_{\text{max}}\) (thin film) (cm\(^{-1}\)): 3431, 3100, 2903, 2856, 1464, 1396, 1234; \(^{1}\)H NMR (300 MHz, C\(_6\)D\(_6\)); \(\delta_{\text{H}}\): 4.19–4.16 (m, 4H), 4.04 (s, 7H), 3.61 (br s, 2H), 3.38 (\(t\), \(J\)=4.8 Hz, 2H), 1.99 (br s, 1H); \(^{13}\)C NMR (75 MHz, C\(_6\)D\(_6\)); \(\delta_{\text{C}}\) 84.3, 71.8, 70.0, 69.9, 69.2, 69.1, 62.4. HRMS (ESI) calculated for C\(_{13}\)H\(_{16}\)FeO\(_2\)Na m/z 283.0397 found 283.0372 (m/z + Na\(^{+}\)); Oxidation potential: 181 mV.

3-(Ferrocenyloxy)propan-1-ol (2).

Using general procedure A with ferrocene methanol (545 mg, 2.5 mmol), ytterbium triflate (77 mg, 5 mol%) and 1,3-propanediol (10 mL). This gave the desired product as an orange solid (514 mg, 75%). Mpt: 40–42 °C; IR; \(\nu_{\text{max}}\) (thin film) (cm\(^{-1}\)): 3408, 3082, 2940, 2856, 1453, 1356, 1270, 1058; \(^{1}\)H NMR (300 MHz, C\(_6\)D\(_6\)); \(\delta_{\text{H}}\): 4.17–4.13 (m, 4H), 4.07–4.03 (m,
7H), 3.72 (app q, J=5.3, 2H), 3.48 (t, J=5.8, 2H), 2.19 (t, J=5.3, 1H), 1.87–1.52 (m, 2H); "C NMR (75 MHz, C6D6); δC: 84.7, 69.8, 69.6, 69.5, 68.9, 62.1, 33.1; HRMS (ESI) calculated for C14H18FeO2Na m/z 297.0553 found 297.0560 (m/z + Na+); Oxidation potential: 178 mV.

4-(Ferrocenyloxy)butan-1-ol (3).

Using general procedure A with ferrocene methanol (500 mg, 2.35 mmol), ytterbium triflate (71 mg, 5 mol%) and 1,4-butanediol (10 mL). This gave the desired product as an orange oil (636 mg, 95%). IR; v_max (thin film) (cm⁻¹): 3407, 3094, 2938, 2848, 1639, 1446, 1410, 1375, 1232, 1054; "H NMR (300 MHz, C6D6); δH: 4.21–4.2 (m, 4H), 4.07–4.05 (m, 7H), 3.55 (br s, 2H), 3.38 (t, J=5.5, 2H), 2.08 (br s, 1H), 1.66–1.56 (m, 4H); "C NMR (75 MHz, C6D6); δC: 84.6, 70.5, 69.9, 69.7, 69.2, 68.9, 62.9, 31.1, 27.6; HRMS (ESI) calculated for C15H20FeO2Na m/z 311.0710 found 311.0710 (m/z + Na+); Oxidation potential: 184 mV.

5-(Ferrocenyloxy)pentan-1-ol (4).

Using general procedure A with ferrocene methanol (545 mg, 2.5 mmol), ytterbium triflate (77 mg, 5 mol%) and 1,5-pentanediol (10 mL). This gave the desired product as a viscous orange oil (676 mg, 89%). IR; v_max (thin film) (cm⁻¹): 3383, 3085, 2926, 1854, 1433, 1343, 1232; "H NMR (300 MHz, C6D6); δH: 4.07–4.06 (m, 4H), 3.90–3.87 (m, 7H), 3.28–3.22 (m, 4H), 1.47–1.42 (m, 2H), 1.30–1.21 (m, 4H); "C NMR (75 MHz, C6D6); δC: 85.0, 70.5, 69.9, 69.6, 69.2, 68.9, 62.8, 33.3, 30.3, 23.3; HRMS (ESI) calculated for C16H22FeO2Na m/z 325.0866 found 325.0880(m/z + Na+); Oxidation potential: 186 mV.
6-(Ferrocenyloxy)hexan-1-ol (5).\(^3\)

Using general procedure A with ferrocene methanol (420 mg, 1.9 mmol), ytterbium triflate (58.9 mg, 5 mol%) and 1,6-hexanediol (2.25 g, 19 mmol) with dry acetonitrile (5 cm\(^3\)) as solvent. This gave the desired product as a dark orange solid (538 mg, 89%). Mpt: 38–40 °C; IR; \(\nu_{\text{max}}\) (thin film) (cm\(^{-1}\)): 3369, 3102, 2928, 2852, 1479, 1453, 1344, 1232, 1090 \(^1\)H NMR (300 MHz, C\(_6\)D\(_6\)); \(\delta_H\): 4.28–4.25 (m, 4H), 4.08–4.00 (m, 7H), 3.47–3.43 (m, 4H), 1.69–1.60 (m, 2H), 1.49–1.28 (m, 6H); \(^13\)C NMR (75 MHz, C\(_6\)D\(_6\)); \(\delta_C\): 85.2, 70.5, 69.9, 69.7, 69.2, 68.9, 62.9, 33.5, 30.7, 26.9, 26.3; HRMS (ESI) calculated for C\(_{17}\)H\(_{24}\)FeO\(_2\)Na m/z 339.1023 found 339.1024 (m/z + Na\(^+\)); Oxidation potential: 195 mV.

7-(Ferrocenyloxy)heptan-1-ol (6).

Using general procedure A with ferrocene methanol (109 mg, 0.5 mmol), ytterbium triflate (15.5 mg, 5 mol%) and 1,7-heptanediol (2 ml). This gave the desired product as an orange oil (116 mg, 70%). IR; \(\nu_{\text{max}}\) (thin film) (cm\(^{-1}\)) 3371, 3092, 2927, 2852, 2121, 1634, 1466, 1343, 1233; \(^1\)H NMR (300 MHz, C\(_6\)D\(_6\)); \(\delta_H\): 4.29–4.26 (m, 4H), 4.09–4.06 (m, 7H), 3.49–3.41 (m, 4H), 1.71–1.62 (m, 2H), 1.54–1.28 (m, 8H); \(^13\)C NMR (75 MHz, C\(_6\)D\(_6\)); \(\delta_C\): 85.2, 70.6, 69.9, 69.7, 69.2, 68.8, 63.1, 33.5, 30.6, 30.0, 27.1, 26.5; HRMS (ESI) calculated for C\(_{18}\)H\(_{26}\)FeO\(_2\)Na m/z 353.1179 found 353.1191 (m/z + Na\(^+\)); Oxidation potential: 212 mV.

8-(Ferrocenyloxy)octan-1-ol (7).\(^3\)

Using general procedure A with ferrocene methanol (109 mg, 0.5 mmol), ytterbium triflate (15.5 mg, 5 mol%) and 1,8-octanediol (731 mg, 5 mmol) in a minimal amount of dry 1,4-dioxane (5 ml). This gave the desired product as an orange oil (65 mg, 38%). IR; \(\nu_{\text{max}}\) (thin film) (cm\(^{-1}\)) 3407, 3094, 2924, 2852, 1726, 1455, 1343, 1233, 1094; \(^1\)H NMR (300 MHz,
$\text{C}_6\text{D}_6$; $\delta_H$: 4.30–4.26 (m, 4H), 4.09–4.06 (m, 7H), 3.51–3.42 (m, 4H), 1.73–1.64 (m, 2H), 1.45–1.30 (m, 10H); $^{13}\text{C}$ NMR (75 MHz, $\text{C}_6\text{D}_6$); $\delta_C$: 85.2, 70.6, 69.9, 69.6, 69.2, 68.9, 63.1, 33.6, 30.7, 30.2, 30.2, 27.0, 26.5; HRMS (ESI) calculated for $\text{C}_{19}\text{H}_{28}\text{FeO}_2\text{Na} \uparrow m/z$ 367.1336 found 367.1353 ($m/z + \text{Na}^+$). Oxidation potential: 233 mV.

8-(Ferrocenyloxy)nonan-1-ol (8).³

Using general procedure A with ferrocene methanol (109 mg, 0.5 mmol), ytterbium triflate (15.5 mg, 5 mol%) and 1,9-nonanediol (801 mg, 5 mmol) in a minimal amount of dry 1,4 dioxane (5 ml). This gave the desired product as an orange oil (66 mg, 37%). IR; $\nu_{\text{max}}$(thin film) (cm⁻¹): 3407, 3093, 2925, 2852, 1725, 1434; $^1\text{H}$ NMR (300 MHz, $\text{C}_6\text{D}_6$); $\delta_H$: $\delta_H = 4.19$ (s, 2H), 4.16–4.15 (m, 2H), 4.06 (s, 7H), 3.55 (t, $J=6.7$, 2H), 3.33 (t, $J=6.7$, 2H), 1.52–1.45 (m, 4H), 1.28–1.21 (m, 10H); $^{13}\text{C}$ NMR (75 MHz, CDCl₃); $\delta_C$: 83.7, 70.1, 69.5, 69.1, 68.4, 63.0, 32.8, 29.7, 29.6, 29.4, 29.4, 26.2, 25.7; HRMS (ESI) calculated for $\text{C}_{20}\text{H}_{30}\text{FeO}_2\text{Na} \uparrow m/z$ 381.1493 found 381.1561 ($m/z + \text{Na}^+$). Oxidation potential: 260 mV.

8-(Ferrocenyloxy)decan-1-ol (9).

Using general procedure A with ferrocene methanol (430 mg, 2.5 mmol), ytterbium triflate (61 mg, 5 mol%) and 1,10-decanediol (3.4 g, 19.7 mmol) in a minimal amount of dry 1,4 dioxane (15 ml). This gave the desired product as an orange oil (346 mg, 47%). IR; $\nu_{\text{max}}$(thin film) (cm⁻¹): 3437, 3076, 2916, 2849, 1464, 1349; $^1\text{H}$ NMR (300 MHz, $\text{C}_6\text{D}_6$); $\delta_H$: 4.30 (s, 2H), 4.26 (s, 2H), 4.09–4.07 (s, 7H), 3.52–3.44 (m, 4H), 1.75–1.66 (m, 2H), 1.48–1.32 (m, 14H); $^{13}\text{C}$ NMR (75 MHz, $\text{C}_6\text{D}_6$); $\delta_C$: 85.3, 70.6, 69.8, 69.7, 69.2, 68.8, 63.1, 33.6, 30.8, 30.4, 30.4, 30.3, 30.2, 27.1, 26.6; HRMS (ESI) calculated for $\text{C}_{21}\text{H}_{32}\text{FeO}_2\text{Na} \uparrow m/z$ 395.1649 found 395.1637 ($m/z + \text{Na}^+$). Oxidation potential: 285 mV.
General Procedure B for the Acylation of ferrocene derived alcohols.

To a solution of the ferrocene alcohol (0.5 mmol) and DMAP (6 mg, 10 mol%) in dry THF (5 ml) was added acetic anhydride (90 ml, 1 mmol) dropwise over a 2 minute period. Once addition of acetic anhydride was complete the solution was allowed to stir at room temperature for 5 minutes (TLC analysis at this time shows full conversion of the starting material). The reaction was then diluted with EtOAc (20 ml) and the organics washed with water (20 ml), NaHCO$_3$ (sat) (20 ml) and brine (sat) (20 ml). The organics were then dried over MgSO$_4$, filtered and concentrated in vacuo, to give the desired product without need for further purification.

2-(Ferrocenyloxy)ethyl acetate (10).

Using general procedure B with 2-(ferrocenyloxy)ethanol (1) (131 mg, 0.5 mmol, 1eq). This gave the desired compound as an orange solid (132 mg, 87%). Mpt: 41–43 °C; IR; $\nu_{\text{max}}$ (thin film) (cm$^{-1}$): 3082, 2981, 2964, 2863, 1731, 1460; $^1$H NMR (300 MHz, C$_6$D$_6$); $\delta_{\text{H}}$: 4.22–4.17 (m, 6H), 4.06–4.04 (m, 7H), 3.48–3.45 (m, 2H), 1.74 (s, 3H); $^{13}$C NMR (75 MHz, C$_6$D$_6$); $\delta_{\text{C}}$: 170.6, 84.4, 69.9, 69.7, 69.2, 68.9, 68.2, 63.9, 20.9; HRMS (ESI) calculated for C$_{15}$H$_{18}$FeO$_3$Na m/z 325.0503 found 325.0475 (m/z + Na$^+$); Oxidation potential: 193 mV.

3-(Ferrocenyloxy)propyl acetate (11).

Using general procedure B with 2-(ferrocenyloxy)propan-1-ol (2) (138 mg, 0.5 mmol, 1eq). This gave the desired compound as an orange oil (151 mg, 95%); IR; $\nu_{\text{max}}$ (thin film) (cm$^{-1}$): 3094, 2980, 2856, 1734, 1365; $^1$H NMR (300 MHz, C$_6$D$_6$); $\delta_{\text{H}}$: 4.24 (t, $J$=6.5, 2H), 4.20 (s,
4H), 4.06–4.04 (m, 7H), 3.41 (t, J=6.2, 2H), 1.86–1.78 (m, 2H), 1.74 (s, 3H); $^{13}$C NMR (75 MHz, C$_6$D$_6$): δC: 170.5, 84.9, 70.2, 69.9, 69.6, 69.2, 68.9, 64.6, 27.0, 26.4, 20.9; HRMS (ESI) calculated for C$_{18}$H$_{24}$FeO$_3$Na m/z 367.0973 found 367.0985 (m/z + Na$^+$); Oxidation potential: 232 mV.

4-(Ferrocenyloxy)butyl acetate (12).

Using general procedure B with 2-(ferrocenyloxy)butan-1-ol (3) (145 mg, 0.5 mmol, 1eq). This gave the desired compound as an orange oil (144 mg, 86%); IR; $\nu$$_{max}$ (thin film) (cm$^{-1}$): 3094, 2980, 2941, 2861, 1735, 1460; $^1$H NMR (300 MHz, CDCl$_3$); δH: 4.25–4.23 (m, 4H), 4.08–4.02 (m, 9H), 3.39 (t, J=6.2, 2H); 13C NMR (75 MHz, CDCl$_3$); δ: 170.5, 85.1, 70.2, 69.9, 69.6, 69.2, 68.9, 64.7, 30.2, 29.2, 23.5, 20.9; HRMS (ESI) calculated for C$_{18}$H$_{24}$FeO$_3$Na m/z 367.0973 found 367.0985 (m/z + Na$^+$); Oxidation potential: 232 mV.

5-(Ferrocenyloxy)pentyl acetate (13).

Using general procedure B with 2-(ferrocenyloxy)pentan-1-ol (4) (152 mg, 0.5 mmol, 1eq). This gave the desired compound as an orange oil (94 mg, 54%); IR; $\nu$$_{max}$ (thin film) (cm$^{-1}$): 3094, 2980, 2941, 2861, 1735, 1460; $^1$H NMR (300 MHz, CD$_2$D$_6$); δH: 4.25–4.23 (m, 4H), 4.08–4.02 (m, 9H), 3.39 (t, J=6.2, 2H), 1.76 (s, 3H), 1.60–1.36 (m, 6H); $^{13}$C NMR (75 MHz, CDCl$_3$); δC: 170.5, 85.1, 70.2, 69.9, 69.6, 69.2, 68.9, 64.7, 30.2, 29.2, 23.5, 20.9; HRMS (ESI) calculated for C$_{18}$H$_{24}$FeO$_3$Na m/z 367.0973 found 367.0985 (m/z + Na$^+$); Oxidation potential: 232 mV.
6-(Ferrocenyloxy)hexyl acetate (14).

Using general procedure B with 2-(ferrocenyloxy)hexan-1-ol (5) (159 mg, 0.5 mmol, 1eq). This gave the desired compound as an orange oil (154 mg, 86%); IR; \( \nu_{\text{max}} \) (thin film) (cm\(^{-1}\)): 3094, 2936, 2856, 1735, 1462; \(^1\)H NMR (300 MHz, C\(_6\)D\(_6\)); \( \delta \): 4.27 (s, 2H), 4.25 (br s, 2H), 4.09–4.01 (m, 9H), 3.42 (t, \( J=6.4 \), 1H), 1.77 (s, 1H), 1.64–1.44 (m, 4H), 1.38–1.21 (m, 4H); \(^{13}\)C NMR (75 MHz, C\(_6\)D\(_6\)); \( \delta \): 170.5, 85.2, 70.3, 69.9, 69.6, 69.2, 68.9, 64.7, 30.5, 29.4, 26.7, 26.5, 20.9; HRMS (ESI) calculated for C\(_{19}\)H\(_{26}\)FeO\(_3\)Na m/z 381.1129 found 381.1131 (m/z + Na\(^+\)); Oxidation potential: 257 mV.

7-(Ferrocenyloxy)heptyl acetate (15).

Using general procedure B with 2-(ferrocenyloxy)heptan-1-ol (6) (74 mg, 0.22 mmol, 1eq), acetic anhydride (40 \( \mu \)l, 0.44 mmol, 2 eq) and DMAP (2.7 mg, 0.02 mmol, 10 mol%). This gave the desired compound as an orange oil (80 mg, 97%); IR; \( \nu_{\text{max}} \) (thin film) (cm\(^{-1}\)): 3094, 2936, 2856, 1735, 1462; \(^1\)H NMR (300 MHz, C\(_6\)D\(_6\)); \( \delta \): 4.29 (s, 2H), 4.26–4.25 (m, 2H), 4.09–4.03 (m, 9H), 3.46 (t, \( J=6.4 \), 2H), 1.77 (s, 3H), 1.68–1.61 (m, 2H), 1.57–1.36 (m, 5H), 1.24–1.22 (m, 3H); \(^{13}\)C NMR (75 MHz, C\(_6\)D\(_6\)); \( \delta \): 170.5, 85.2, 70.5, 69.9, 69.7, 69.2, 68.9, 64.8, 30.6, 29.7, 29.3, 26.9, 26.6, 20.9; HRMS (ESI) calculated for C\(_{20}\)H\(_{28}\)FeO\(_3\)Na m/z 395.1286 found 395.1280 (m/z + Na\(^+\)); Oxidation potential: 280 mV.

8-(Ferrocenyloxy)octyl acetate (16).

Using general procedure B with 2-(ferrocenyloxy)octan-1-ol (7) (57 mg, 0.16 mmol, 1eq), acetic anhydride (30 \( \mu \)l, 0.32 mmol, 2 eq) and DMAP (2 mg, 0.016 mmol, 10 mol%). This
gave the desired compound as an orange oil (52 mg, 83%); IR; $\nu_{\text{max}}$ (thin film) (cm$^{-1}$): 3094, 2930, 2854, 1736, 1464; $^1$H NMR (300 MHz, C$_6$D$_6$); $\delta_{H}$: 4.30 (s, 2H), 4.26 (s, 2H), 4.09 – 4.04 (m, 9H), 3.48 (t, $J$=6.4, 2H), 1.78 (s, 3H), 1.71–1.62 (m, 2H), 1.50–1.38 (m, 5H), 1.29–1.23 (m, 5H); $^{13}$C NMR (75 MHz, C$_6$D$_6$); $\delta_{C}$: 170.5, 85.2, 70.5, 69.9, 69.7, 69.2, 68.9, 64.8, 30.7, 30.1, 29.9, 29.4, 26.9, 26.6, 20.9; HRMS (ESI) calculated for C$_{21}$H$_{30}$FeO$_3$Na m/z 409.1442 found 409.1476 (m/z + Na$^+$); Oxidation potential: 302 mV.

9-(Ferrocenyloxy)nonyl acetate (17).

Using general procedure B with 2-(ferrocenyloxy)nonan-1-ol (8) (51 mg, 0.14 mmol, 1eq), acetic anhydride (28 µl, 0.28 mmol, 2 eq) and DMAP (1.7 mg, 0.014 mmol, 10 mol%). This gave the desired compound as an orange oil (43 mg, 76%); IR; $\nu_{\text{max}}$ (thin film) (cm$^{-1}$): 3093, 2927, 2854, 1737, 1464; $^1$H NMR (300 MHz, C$_6$D$_6$); $\delta_{H}$: 4.31 (s, 2H), 4.27–4.26 (m, 2H), 4.09–4.05 (m, 9H), 3.50 (t, $J$=6.4, 2H), 1.79 (s, 3H), 1.74–1.65 (m, 2H), 1.51–1.40 (m, 5H), 1.27–1.23 (m, 7H); $^{13}$C NMR (75 MHz, C$_6$D$_6$); $\delta_{C}$: 170.5, 85.2, 70.6, 69.9, 69.7, 69.2, 68.9, 64.8, 30.8, 30.2, 30.2, 29.9, 29.4, 27.1, 26.6, 20.9; HRMS (ESI) calculated for C$_{22}$H$_{34}$FeO$_3$Na m/z 423.1598 found 423.1651 (m/z + Na$^+$); Oxidation potential: 327 mV.

10-(Ferrocenyloxy)decel acetate (18).

Using general procedure B with 2-(ferrocenyloxy)decan-1-ol (9) (187 mg, 0.5 mmol, 1eq). This gave the desired compound as an orange oil (194 mg, 93%); IR; $\nu_{\text{max}}$ (thin film) (cm$^{-1}$): 3098, 2926, 2853, 1737, 1464; $^1$H NMR (300 MHz, C$_6$D$_6$); $\delta_{H}$: 4.30 (d, $J$=2.2, 2H), 4.27–4.26 (m, 2H), 4.09–4.06 (m, 9H), 3.50 (td, $J$=6.4, 2.7, 2H), 1.79 (s, 3H), 1.75–1.66 (m, 2H), 1.53–1.48 (m, 5H), 1.34–1.23 (m, 9H); $^{13}$C NMR (75 MHz, C$_6$D$_6$); $\delta_{C}$: 170.6, 85.2, 70.6, 69.9, 69.7, 69.2, 68.9, 64.8, 30.7, 30.4, 30.3, 30.3, 30.1, 29.9, 29.4, 27.1, 26.6, 20.9; HRMS (ESI) calculated for C$_{23}$H$_{36}$FeO$_3$Na m/z 437.1755 found 437.1785 (m/z + Na$^+$); Oxidation potential: 335 mV.
(3)

```
Fe

O
```

**Spectrometer:** mm 300
**Author:**
**Solvent:** CHLOROFORM-D
**Temperature:** 294.5 °C
**Pulse Sequence:** zg3g30
**Number of Scans:** 256
**Receiver Gain:** 91295
**Relaxation Delay:** 2.00000
**Pulse Width:** 7.50000
**Acquisition Time:** 1.6122
**Acquisition Date:** 2013-10-25T13:50:00
**Modification Date:** 2013-10-25T13:50:46
**Spectrometer Frequency:** 75.49
**Spectral Width:** 20828.2
**Lowest Frequency:** -146.96
**Number Acquired:** 123
**Acquired Size:** 32368
**Spectral Size:** 65536
(5)
(14)
4. References.

