Supporting Information

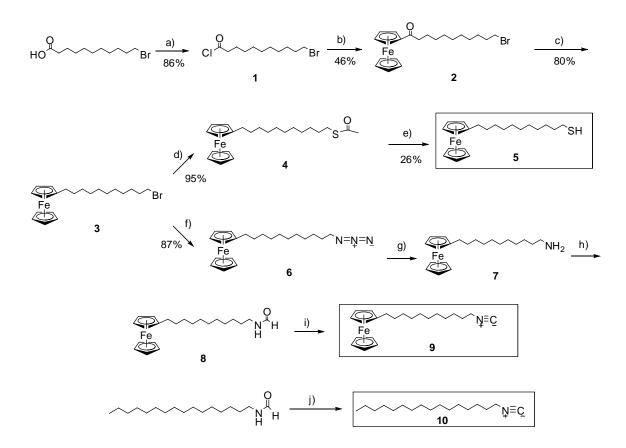
A Comprehensive Investigation of Self-Assembled Monolayer Formation on Ferromagnetic Thin Film Surfaces

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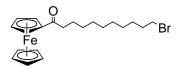
Synthesis



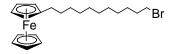
Scheme 1: Syntheses of compound 5, 9, and 10. a) SOCl₃, r.t.; b) ferrocene, AlCl₃, CH₂Cl₂, r.t.; c) AlCl₃, NaBH₄, THF, 0°C; d) potassium thiolacetate, THF, 75°C 1 hour, r.t. overnight; e) NaOH, EtOH, 85°C; f) NaN₃, DMF, r.t.; g) LiAlH₄, ether, r.t.; h) ethyl formate, r.t.; i) POCl₃, DIEA, THF, r.t.; j) POCl₃, TEA, THF, 0°C.

General Methods. Unless otherwise noted, all starting materials were obtained from commercial sources and used without further purification. All reactions were conducted under nitrogen atmosphere in flame dried glassware. Tetrahydrofuran (THF) was distilled over sodium/benzophenone ketyl under nitrogen, and methylene chloride (CH_2Cl_2) was dried using Innovative Technology, Inc.'s PureSolv purification column. Triethyalamine (TEA) and diisopropylethylamine (DIEA) were distilled over calcium hydride under nitrogen prior to use. Anhydrous toluene and anhydrous diethyl ether were obtained from commercial sources and used without further drying. NMR spectra were recorded at 300

or 400 MHz on Bruker AMX-300 or ADVANCE-400 spectrometers, respectively. Mass spectra were performed by the University of North Carolina at Chapel Hill mass spectrometry facility.

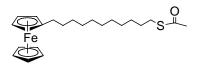


(**11-Bromoundecanoyl)ferrocene (2).** To a flask equipped with a vacuum distillation apparatus was added 11-bromoundecanoic acid (25 g, 94 mmol) and thionyl chloride (13.7 mL, 188.5 mmol). This solution was stirred overnight at room temperature. The following day, 11-Bromoundecanoyl chloride (1) was distilled (129°C at 0.285 torr) to give 23.0 g (86% yield) of a colorless oil. Separately, ferrocene (15.07 g, 81 mmol) and AlCl₃ (10.69 g, 81 mmol) were dissolved into 100 mL anhydrous CH₂Cl₂ and cooled to 0°C. A solution of compound **1** (23.0 g, 81 mmol) in 40 mL dry CH₂Cl₂ was then added drop wise over 1 hour. The mixture was warmed to room temperature and stirred for 12 hours. The resulting mixture was poured into water and extracted with ethyl acetate. The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated, and purified over a silica plug (hexane: CH₂Cl₂; 20:1). The reaction produced a yellow solid in 46% yield. ¹H NMR (300 MHz, CDCl₃): δ (ppm): 1.30 (m, 12H, CH₂), 1.67 (m, 2H, CH₂), 1.84 (p, 2H, *J* = 7.2 Hz, CH₂), 2.69 (t, 2H, *J* = 7.5 Hz, CH₂), 3.41 (t, 2H, *J* = 6.84 Hz, CH₂), 4.19 (s, 5H, H(Cp)), 4.49 (t, 2H, *J_{ab}=J_{ac}*= 1.75 Hz, H(Cp)), and 4.78 (t, 2H, H(Cp)).

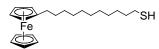


(11-Bromoundecyl)ferrocene (3). A mixture of 2 (16.15 g, 37.3 mmol), AlCl₃ (8.36 g, 63.4 mmol), and dry THF (200 mL) was cooled to 0°C. A solution of NaBH₄ (6.35 g, 167.9 mmol) in 40 mL dry THF was then added drop wise over 30 minutes. After stirring for 2 hours, the solution was slowly quenched with 50 mL of H₂O. The resulting mixture was poured into water and extracted with ethyl acetate. The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated, and

purified over a silica plug (hexane: CH₂Cl₂; 20:1). The reaction yielded red crystals in 80% yield. ¹H NMR (300 MHz, CDCl₃): δ (ppm): 1.28 (m, 12H, CH₂), 1.45 (m, 4H, CH₂), 1.85 (p, 2H, *J* = 7.48 Hz, CH₂), 2.31 (t, 2H, *J* = 7.94 Hz, CH₂), 3.41 (t, 2H, *J* = 6.86 Hz, CH₂), 4.03 (d, 2H, *J* = 1.57 Hz, H(Cp)), 4.05 (d, 2H, *J* = 1.48 Hz, H(Cp)), and 4.09 (s, 5H, H(Cp)).



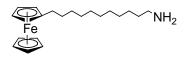
(11-Acetylthiolylundecyl)ferrocene (4). To a flask was added 3 (8.0 g, 19.1 mmol), potassium thioacetate (2.73 g, 23.9 mmol), and dry THF (100 mL). The solution was brought to reflux for 1 hour, and then allowed to cool to room temperature overnight. The resulting mixture was poured into water and extracted with ethyl acetate. The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated, and purified via column chromatography over silica gel (10:1; hexane:CH₂Cl₂). The reaction yielded red crystals (95 % yield). ¹H NMR (300 MHz, CDCl₃): δ (ppm): 1.27 (m, 14H, CH₂-), 1.52 (m, 4H, CH₂), 2.30 (t, 2H, CH₂), 2.32 (s, 3H, -S-CO-CH₃), 2.86 (t, 2H, CH₂), 4.03 (d, 2H, *J* = 1.60 Hz, H(Cp)), 4.05 (d, 2H, *J* = 1.49 Hz, H(Cp)), and 4.09 (s, 5H, H(Cp)).



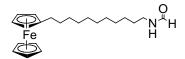
(11-Thiolundecyl)ferrocene (5). A mixture of 4 (7.48 g, 18.1 mmol), NaOH (32.7 mL of 1M solution), and ethanol (200 mL) was refluxed overnight. The mixture was then poured into water and extracted with ethyl acetate. The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated, and purified via column chromatography over silica gel (10:1; hexane:CH₂Cl₂). The reaction produced red crystals (26% yield) with the major side product being the disulfide analog. ¹H NMR (300 MHz, CDCl₃): δ (ppm): 1.27 (m, 14H, CH₂), 1.50 (m, 2H, CH₂), 1.60 (m, 2H, CH₂), 2.31 (t, 2H, *J* = 7.91 Hz, CH₂), 2.52 (q, 2H, *J* = 7.18 Hz, CH₂), 4.03 (d, 2H, *J* = 1.50 Hz, H(Cp)), 4.05 (d, 2H, *J* = 1.36, H(Cp)), and 4.09 (s, 5H, H(Cp)). ¹³C NMR (100 MHz, CDCl₃): δ (ppm): 24.64, 28.37, 29.07,

29.51, 29.58, 29.65, 31.11, 34.05, 66.96, 68.04, 68.43, and 89.59. MS: *m/z* calcd for [M]+ (C₂₁H₃₂FeS): 372.16; found: 372.2.

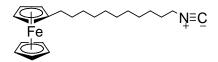
(11-Azidoundecyl)ferrocene (6). To a round bottomed flask was added 3 (2.30 g, 5.5 mmol) and DMF (60 mL). Once dissolved, NaN₃ (415 mg, 6.10 mmol) was added and the resulting mixture was allowed to stir overnight at room temperature. The mixture was then poured into water and extracted with ethyl acetate. The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated, and purified over a silica plug (hexanes) yielding a red oil. ¹H NMR (400 MHz, CDCl₃): δ (ppm): 1.28 (m, 14H, CH₂), 1.48 (m, 2H, CH₂), 1.58 (p, 2H, *J* = 7.6 Hz, CH₂), 2.29 (t, 2H, *J* = 7.6 Hz, CH₂), 3.25 (t, 2H, *J* = 7.2 Hz, CH₂), 4.05 (s, 2H, H(Cp)), 4.08 (s, 2H, H(Cp)), 4.11 (s, 5H, H(Cp)).



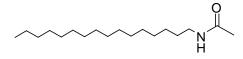
(11-Aminoundecyl)ferrocene (7). To a flask was added LiAlH₄ (400 mg, 10.5 mmol) and 40 mL anhydrous diethyl ether. Separately, a solution of **6** (1.8 g, 4.77 mmol) and diethyl ether (50 mL) was prepared. This solution was added dropwise to the reducing solution over 1.5 hours. The reaction was quenched with 20 mL of 5mM NaOH. The mixture was then poured into water and extracted with ethyl acetate. The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated, and purified over a silica plug (hexane). ¹H NMR (400 MHz, CDCl₃): δ (ppm): 1.25 (m, 14H, CH₂), 1.45 (m, 4H, CH₂), 2.29 (t, 2H, *J* = 8.0 Hz, CH₂), 3.28 (q, 2H, *J* = 6.8 Hz, CH₂), 4.02 (s, 2H, H(Cp)), 4.04 (s, 2H, H(Cp)), 4.07 (s, 5H, H(Cp)), 8.15 (s, 2H, NH₂).



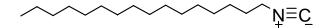
(11-Formamidylundecyl)ferrocene (8). Molecule 8 was prepared according to previously reported literature procedures.¹ ¹H NMR (400 MHz, CDCl₃): δ (ppm): 1.27 (m, 14H, CH₂), 1.43 (m, 2H, CH₂), 1.49 (m, 2H, CH₂), 2.30 (t, 2H, *J* = 7.8 Hz, CH₂), 2.67 (t, 2H, *J* = 7.0 Hz, CH₂), 4.03 (d, 2H, *J* = 2.0 Hz, H(Cp)), 4.04 (d, 2H, *J* = 1.6 Hz, H(Cp)), 4.08 (s, 5H, H(Cp)).



(11-Isocyanoundecyl)ferrocene (9). A mixture of 8 (1.5 g, 4.02 mmol), DIEA (5.0 mL, 28.14 mmol), and dry THF (20 mL) were stirred at room temperature. To this was added POCl₃ (0.42 mL, 4.42 mmol) drop wise at 0°C over 30 minutes. The mixture was allowed to stir for 2 hours then was poured into water and extracted with ethyl acetate. The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated, and purified over a silica plug resulting in orange crystals. ¹H NMR (300 MHz, CDCl₃): δ (ppm):1.28 (m, 12H, CH₂), 1.46 (m, 4H, CH₂), 1.68 (m, 2H, CH₂), 2.31 (t, 2H, *J* = 8.0 Hz, CH₂), 3.38 (m, 2H, CH₂), 4.03 (d, 2H, *J* = 1.42 Hz, H(Cp)), 4.05 (d, 2H, *J* = 1.28 Hz, H(Cp)), and 4.09 (s, 5H, H(Cp)). ¹³C NMR (100 MHz, CDCl₃): δ (ppm): 26.33, 28.70, 29.13, 29.36, 29.49, 29.54, 29.59, 29.63, 31.11, 41.56, 66.96, 68.04, 68.43, and 89.56. MS: *m*/z calcd for [M]+ (C₂₂H₃₁FeN): 365.18; found: 365.2.



1-Formamidylhexadecane (**10**). Molecule **10** was prepared according to previously reported literature procedures by heating a solution of hexadecylamine (4 g) and ethyl formate (40 mL).¹ The reaction solution was concentrated and the product was purified via column chromatography over silica gel (1:1 ethyl acetate:hexanes). Following chromatography, the collected fractions were rotovapped yielding a white solid (3.56 g, 83.5%).



1-Isocyanohexadecane (11). To a dry flask was added N-hexadecylformamide (2.0 g, 7.42 mmol), THF (33mL), and triethylamine (5.21 mL, 37.1 mmol). The solution was cooled to 0°C and POCl₃ (0.77 mL, 8.16 mmol) was added drop wise over 80 minutes. The mixture was allowed to stir for 2 hours then was poured into water and extracted with ethyl acetate. The combined organic extracts were washed with brine, dried (Na₂SO₄), concentrated, and purified over a silica plug (hexane) resulting in light yellow oil. ¹H NMR (300 MHz, CDCl₃): δ (ppm): 0.88 (t, 3H, *J* = 6.95, CH₃), 1.25 (m, 24H, CH₂), 1.43 (m, 2H, CH₂), 1.67 (m, 2H, CH₂), 3.38 (m, 2H, CH₂). ¹³C NMR (100 MHz, CDCl₃): δ (ppm): 14.07, 22.67, 26.33, 28.70, 29.14, 29.34, 29.49, 29.58, 29.64, 29.67, 31.91, and 41.54.

Characterization of SAMs on ferromagnetic surfaces

Attempts were made to probe the presence of pinholes in SAM layers on magnetic surfaces by performing cyclic voltammetry in the presence of a solution-phase redox couple. The first attempts were made using the familiar $K_3[Fe(CN)_6]$ (aq) system. However, the cyclic voltammograms were consistent with surface-based redox phenomena and showed no signs of the reversible $Fe^{III/II}$ redox wave.² The second set of experiments was performed using 5 mM methyl viologen (PF₆ salt) in 0.1 M tetrabutylammonium hexafluorophosphate in acetonitrile (after 15 minute argon purge).³ Comparison of the cyclic voltammograms for HDT/Ni and HDT/Co with that for bare surfaces showed similar current densities and similar peak-to-peak separations for the oxidative and reductive waves. The area of the electrode exposed to the solution was more hydrophilic after the CV experiment as evidenced by a dramatic lowering of the contact angle of a water droplet (~100-110° to ~70°). By submerging the entire electrode in the methyl viologen solution for 10-15 minutes, it became obvious that significant etching of the metal electrodes occurs in the presence of methyl viologen. Presumably, electron transfer to a good electron acceptor like methyl viologen oxidizes the metal leading to SAM loss, enlargement of

existing pinholes, and/or formation of new pinholes. In the final series of experiments, the redox couple was changed to ferrocene in 0.1 M tetrabutylammonium hexafluorophosphate/THF in order to avoid immediate exposure to an electron acceptor. CVs performed for bare surfaces in the absence of SAMs showed large oxidative currents for ferrocene that are presumably due to *in situ* oxidation of the metal surface or the native oxide by $Fe^{III}(Cp)_2$ (Figure S1a). CVs performed for glovebox HDT SAMs on Co, for example, in the presence of ferrocene showed relatively lower currents (Figure S1b). This result is consistent with incomplete blocking behavior of the HDT SAM and that the presence of the SAM only minimizes oxidation of the metal or native oxide by $Fe^{III}(Cp)_2$. The fact that the presence of SAM allows for passage of non-zero currents to ferrocene suggests the presence of pinholes in the SAM layer. Moreover, for HDT/Ni (glovebox) the contact angle of the surface exposed to cyclic voltammetry in the presence of ferrocene decreases from 110° to 88°. This suggests that the SAM coverage decreases during electrochemical evaluation, creating pinholes *in situ*, and compromising meaningful interpretation and reliability of the data.

Spectroscopic ellipsometry measurements were attempted to determine the thickness of hexadecanethiol on Ni, Co, and Fe. Spectra were obtained from 300-1000 nm at 65, 70, and 75° incident angles using a V-VASE variable angle spectroscopic ellipsometer (J.A. Woolam). Two sets of samples varying in complexity were attempted: M/Ti/SiO_x/Si and M/glass. Difficulties arose for both sample types when trying to fit the spectral curves using experimentally-determined or literature refractive indeces for the film components along with film thicknesses experimentally determined by QCM analysis. Reliable and reasonable SAM thicknesses were extremely difficult to attain unlike with SAMs/Au, similar to previous ellipsometric studies for SAMs on oxidizable surfaces like Cu and Ag.⁴

One concern with the electroreduction procedure is the introduction of water into the SAM solution which may affect SAM formation on oxidizable surfaces. Control experiments performed with Ni in the glovebox showed that argon-purged HDT solutions (1 mM and 10 mM) containing 0.1 - 0.6 mM water gave identical contact angles as those prepared in the absence of water.

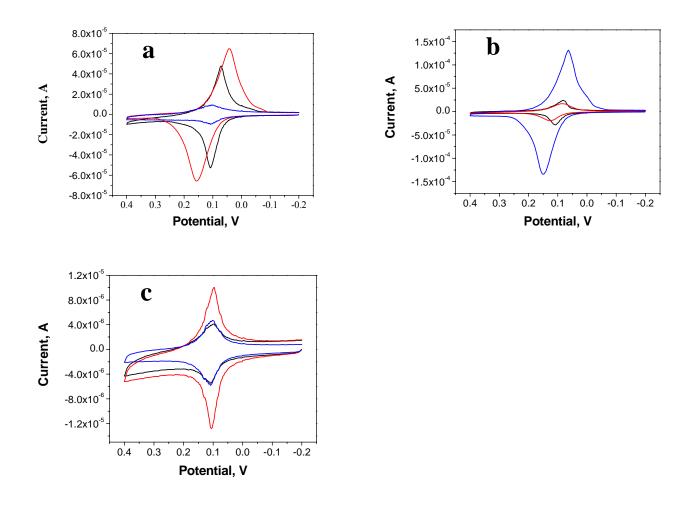


Figure S1. Cyclic voltammograms for (a) Ni, (b) Co, and (c) Fe surfaces derivatized with $Fc-(CH_2)_{11}$ -SH (1 mM ethanolic solution) prepared in the glovebox (black lines), after electroreduction (red lines), or under atmospheric conditions (blue lines). The data for Ni and Co were recorded at 500 mV/s while that for Fe were recorded at 200 mV/s.

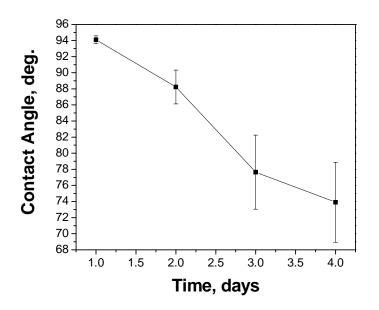


Figure S2. Contact angle versus time for a Co surface treated with 10 mM hexadecanethiol in ethanol under atmospheric conditions.

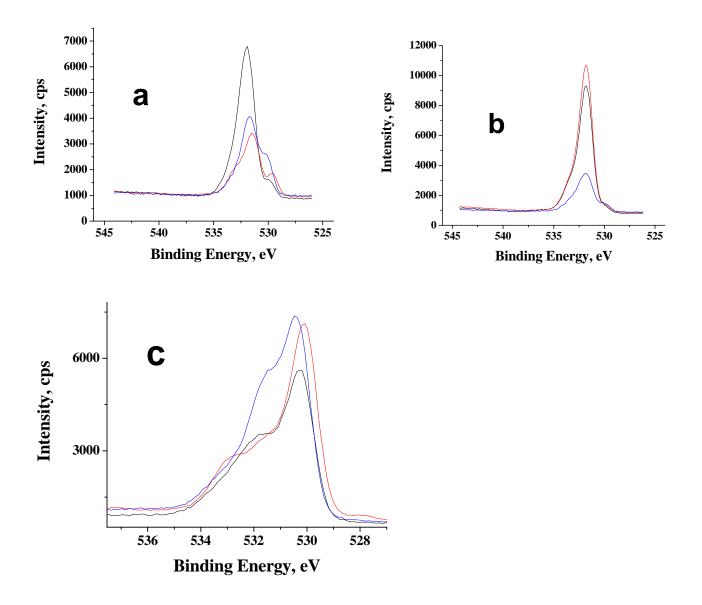


Figure S3. XPS O 1s regional scans for hexadecanethiol on electroreduced (a) Ni, (b) Co, and (c) Fe after minimal exposure to atmospheric conditions (< 1 h) as a function of electroreduction time: 1 min (black line), 5 min (red line), and 10 min (blue line).

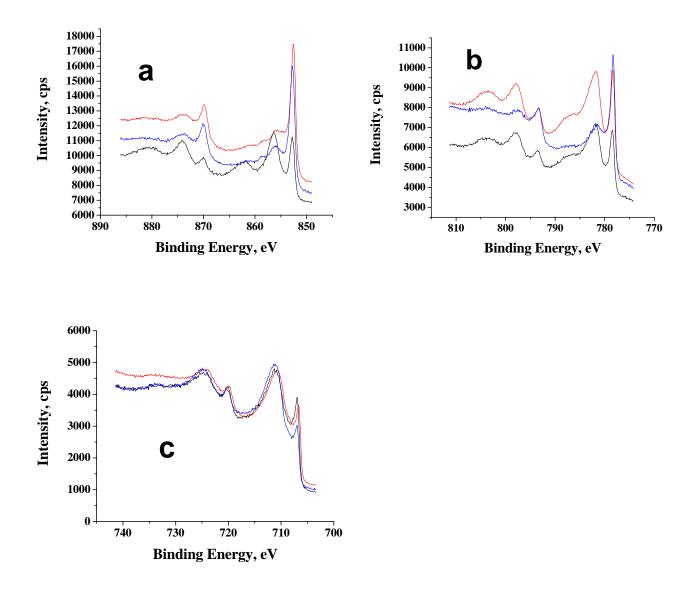


Figure S4. XPS Metal 2p regional scans for hexadecanethiol on electroreduced (a) Ni, (b) Co, and (c) Fe after minimal exposure to atmospheric conditions (< 1 h) as a function of electroreduction time: 1 min (black line), 5 min (red line), and 10 min (blue line).

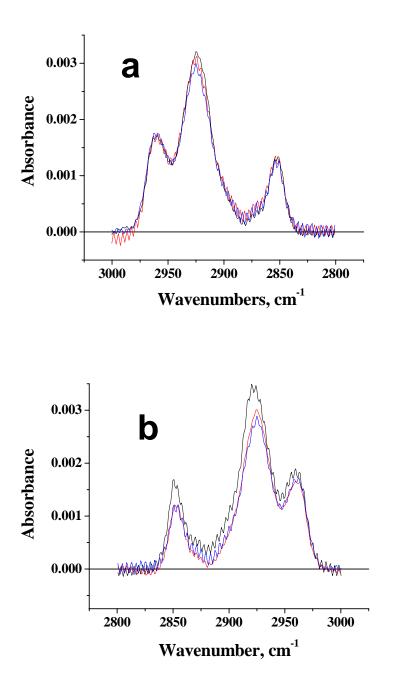


Figure S5. Reflectance-absorbance infrared spectra for hexadecanethiol prepared (a) under atmosphere conditions and (b) under glovebox conditions on nickel (black line), cobalt (red line), and iron (blue line).

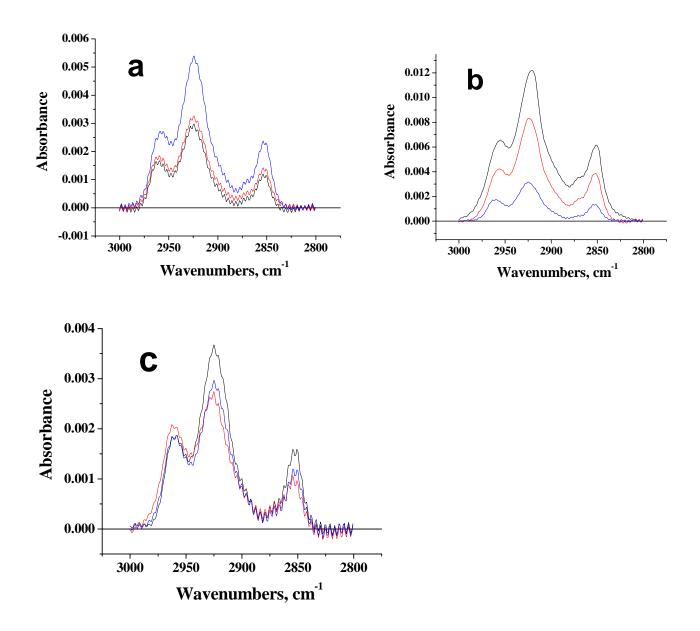


Figure S6. Reflectance-absorbance infrared spectra for hexadecanethiol (from 10 mM solution) prepared by electroreduction conditions at $V_{app} = -1.2$ V vs Ag/AgCl as a function of electroreduction time for (a) Ni, (b) Co, and (c) Fe: 1 min (black line), 5 min, (red line), and 10 min (blue line).

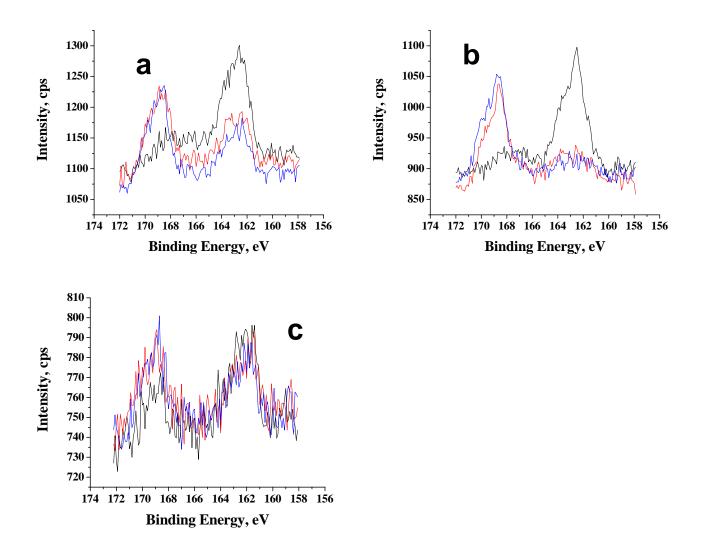


Figure S7. XPS S 2p regional scans for hexadecanethiol SAMs on (a) Ni, (b) Co, and (c) Fe prepared in the glovebox after minimal exposure to atmospheric conditions (black line), after eight days constant exposure to atmospheric conditions (red line), and after fifteen days constant exposure to atmospheric conditions (blue line).

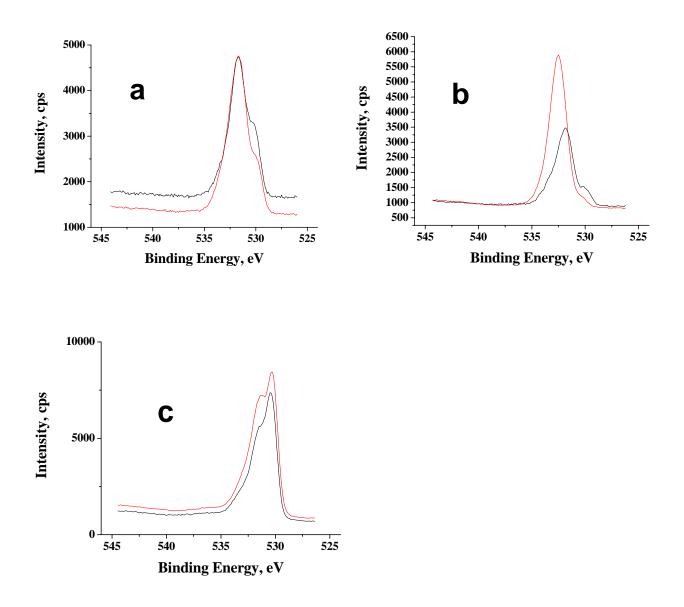


Figure S8. XPS O 1s regional scans for hexadecanethiol on electroreduced (a) Ni, (b) Co, and (c) Fe after minimal exposure to atmospheric conditions (black line) and after 5 days constant exposure to atmospheric conditions (red line).

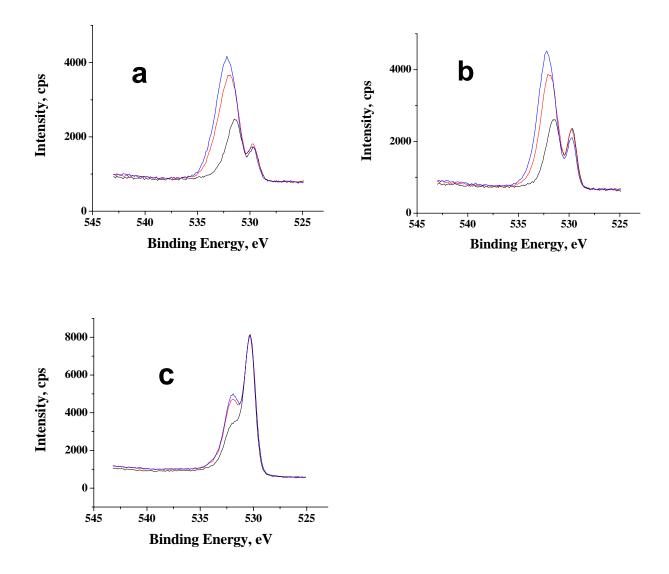


Figure S9. XPS O 1s regional scans for hexadecanethiol SAMs on (a) Ni, (b) Co, and (c) Fe prepared in the glovebox after minimal exposure to atmospheric conditions (black line), after eight days constant exposure to atmospheric conditions (red line), and after fifteen days constant exposure to atmospheric conditions (blue line).

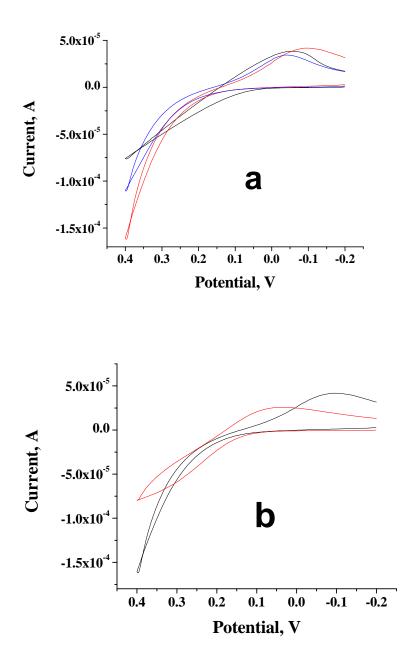


Figure S10. (a) Cyclic voltammograms of bare Ni (black line), bare Co (red line), and bare Fe (blue line) surfaces performed in the presence of 1 mM ferrocene in 0.1 M tetrabutylammonium hexafluorophosphate in dry, distilled tetrahydrofuran. (b) Cyclic voltammograms of bare Co (black line) and HDT/Co (glovebox) (red line) performed in the presence of 1 mM ferrocene in 0.1 M tetrabutylammonium hexafluorophosphate in dry, distilled tetrahydrofuran.

Table S1. XPS O 1s data for all glovebox and electroreduced samples analyzed including the binding energies (BE) of peaks determined from Lorentzian-Gaussian fits as well as integrated areas for the peaks.

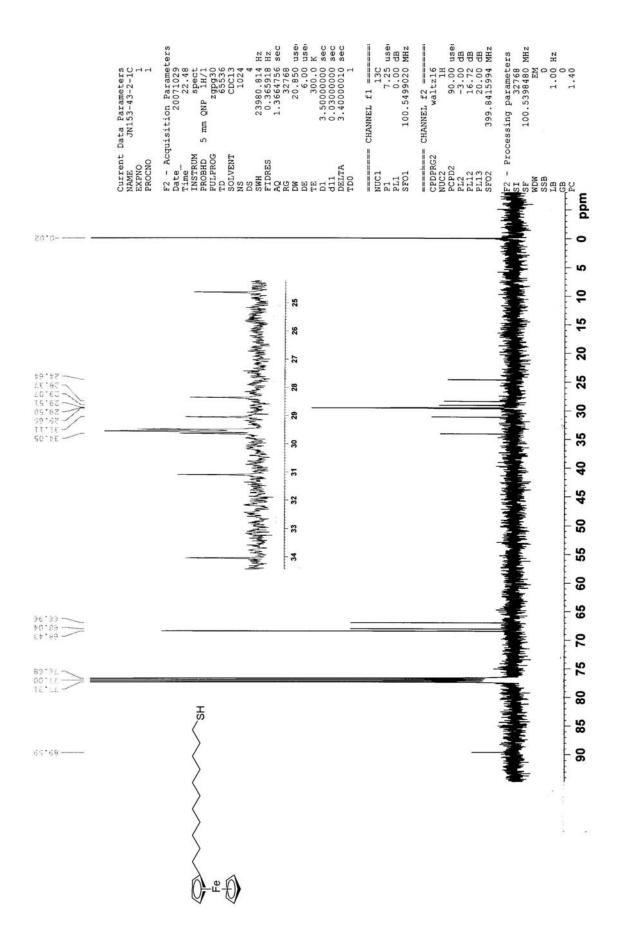
Sample	BE, eV	Area %	BE, eV	Area %	BE, eV	Area %
		(Area)		(Area)		(Area)
Comput			531.5	66.5	529.8	33.5
Co, sput			551.5	(10633)	529.0	(5359)
Co bare	532.8	5.5	531.8	64.4	529.6	30.1
		(8182)		(96319)		(45030)
Co, ER, 1 min	533.3	23.1	531.8	73.9	529.9	2.9
		(12670)		(40488)		(1611)
Co, ER, 5 min	533.4	18.3	531.79	79.8	529.7	2
11111		(5595)		(24439)		(604)
Co, ER, 10 min, 1 h	533.3	21.4	531.8	67.1	529.9	11.4
111111, 1 11		(4288)		(13417)		(2289)
Co, ER, 10 min, 5 d	534.2	7	532.5	90.2	530.1	2.8
IIIII, 5 u		(1205)		(15646)		(486)
Co, GB, 1 d			531.5	68.7	529.6	31.3
				(10921)		(4986)
Co, GB, 8 d	533.6	4.8	531.9	75.8	529.7	19.4
		(1264)		(19883)		(5100)
C CD	524.2	2.2	522.2	02.0	520.7	14
Co, GB,	534.3	2.2	532.2	83.8	529.7	14
15 d		(689)		(26752)		(4480)
Ni sput			531.2	96.7	529.5	3.3
				(20931)		(712)

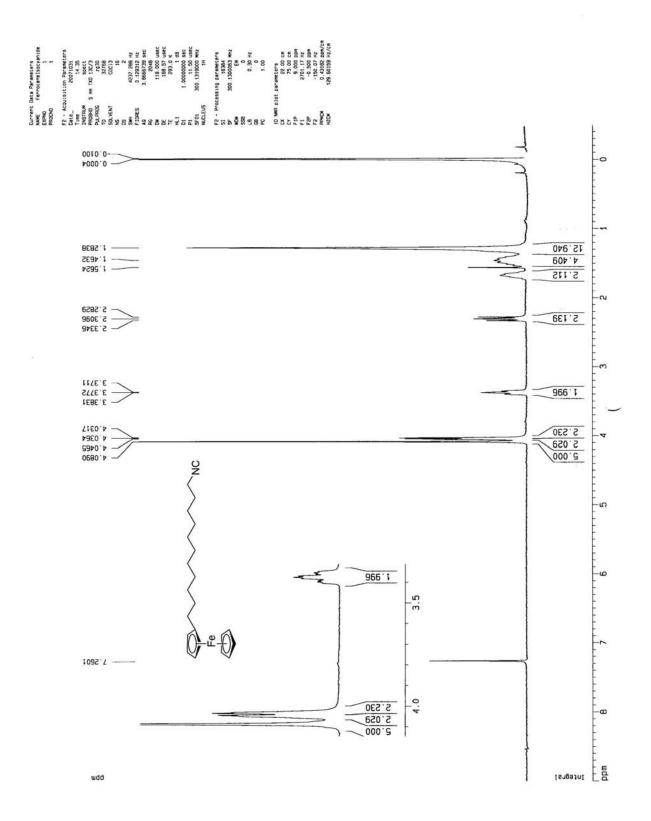
Ni bare	532.8	5.5	531.8	64.4	529.6	30.1
		(6152)		(72420)		(33857)
Ni, ER, 1	533.5	14	532	81.2	529.7	4.9
min		(5740)		(33391)		(2004)
Ni, ER, 5 min	533.2	17.8	531.5	69.8	529.5	12.4
		(1888)		(7410)		(1320)
Ni, ER, 10	532.5	33.4	531.6	40.5	530.2	26.1
min, 1 h		(8687)		(10524)		(6796)
Ni, ER, 10	533.3	19.6	531.6	71.6	529.9	8.8
min, 5 d		(2987)		(10900)		(1345)
Ni, GB, 1d	533.2	9.1	531.4	69.5	529.6	21.4
		(1158)		(8866)		(2726)
Ni, GB, 8 d	533.5	4.9	531.95	84.5	529.6	10.6
		(1144)		(19623)		(2464)
Ni, GB,	534.2	3	532.2	88.6	529.6	8.4
15 d		(899)		(26409)		(2509)
Fe, sput			531.7	40.2	530	59.8
				(2187)		(3248)
Fe, bare			531.7	34.2	530.2	65.8
				(16720)		(32238)
Fe, ER	533.6	4.2	531.9	50.8	530.3	45
1min		(1664)		(19967)		(17695)
Fe, ER,	533.1	9.3	531.5	43.4	530.1	47.3
5min		(2070)		(9690)		(10545)
Fe, ER, 10	533.1	13	531.5	41.5	530.3	45.6
min, 1 h		(6647)		(21249)		(23344)
Fe, ER, 10	533	13	531.3	63	530.2	24
min, 5d		(4288)		(20764)		(7916)

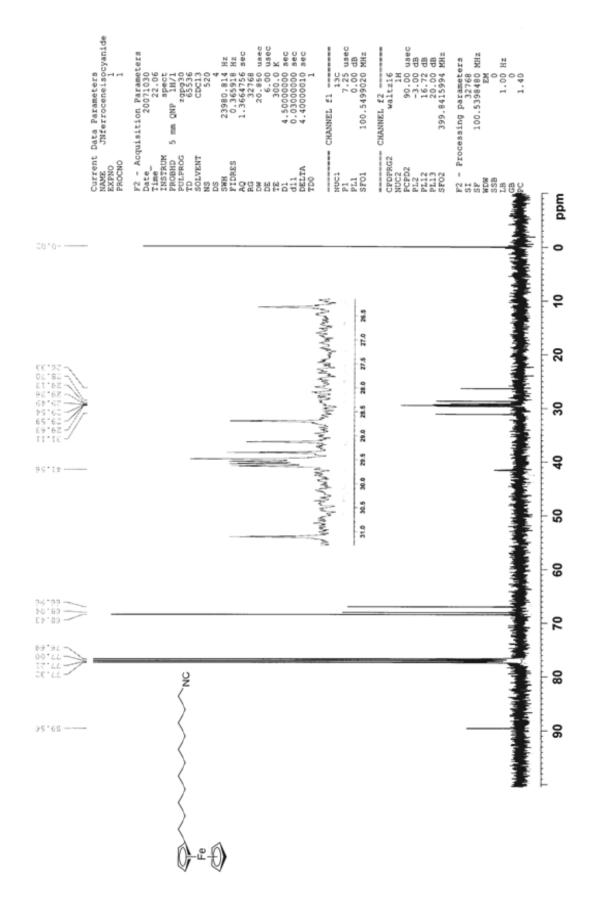
Fe,GB, 1 h	533.7	3.2	531.9	35.5	530.3	61.3
		(1377)		(15163)		(26222)
Fe,GB, 8 d	532.5	6.7	531.9	40.6	530.3	52.7
		(3257)		(19658)		(25502)
Fe,GB,15 d			531.9	50.1	530.3	49.9
				(26589)		(26478)

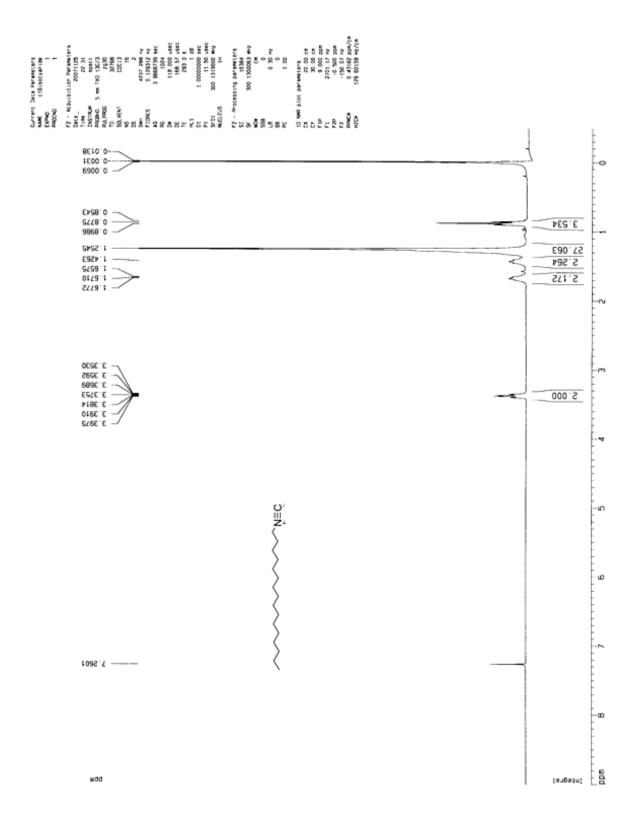
NMR Spectra

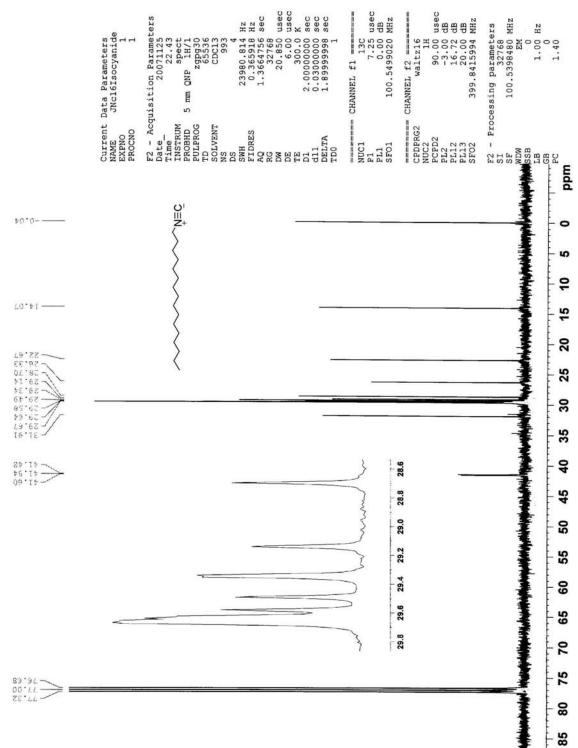
423-200 (00 20) (00 200 (00 200 (00 200 (00 200 (00 200 (00 20) (00 200 (00 200 (00 20) (00 200 (00 20) (00 200 (00 20) (00 200 (00 200 (00 200 (00 20) (00 200 (00 200 (00 20) (00 200 (00 20) (00 200 (00 20) (00 200 (00 20) (00 200 (00 20) (00 200 (00 200 (00 200 (00 200 (00 200 (00 200 (00 200 (00 200 (00 200 (00 200 (00 200 (00 200 (00 20 91 permeters 22.00 cs 30.00 cs 3.000 per 2.001 per 2.001 t3 Hc 0.43142 per/cr 1.30 60159 Hc/Cn 511/2 Derenters 15384 300 1300553 Hrz 64 0 Ŷ Current Data Paraweters NAME JA150/41/2/5 EXEMD 1 PRODAD 1 1 8 ° 8 5101 10.40 F2 - Acou Carte. Trine Eveloped Procesor Procesor SolvEvi Solv . เหตุฐายส 0 1.2732 -5166.1 ¥26'SI ÷ £.234 -n 5.5340 2.5308 875.5 S.053 9.0 5.33P FΜ 4.0302 5.335 4.0882 - 12 2'000 ŝ ŝ -00 -1~ - 1'5233 - 60 (engeant - B udd











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