

Supplementary Information

The Voltammetry of Decamethylferrocene and Cobaltacene in Supercritical Difluoromethane (R32)

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Table 1. Cyclic voltammetry data obtained for experiments with various concentrations of DmFc with 20 mM [NBuⁿ₄] [BF₄] in scR32 at 87 °C, \approx 2550 p.s.i and 10 mV s⁻¹ for varying sizes of microelectrode.

$a / \mu\text{m}$	c / mM	i_L / nA	$\Delta E_{(1/4-3/4)} / \text{mV}$	$E_{1/2} / \text{V}$	$\Delta E_{1/4} / \text{mV}$	$\Delta E_{3/4} / \text{mV}$	$d\ln[(i_L/i)-1]/dE / \text{V}^{-1}$	n
9.945	0.2	6	66.2	0.119	34.2	32	29.55	0.92
	0.53	12.18	80	0.153	41	39	22.38	0.69
	1.06	33.53	70	0.164	42	28	28.05	0.87
	2.13	64.88	57	0.187	38	29	31.85	0.99
13	0.2	8.29	60	0.108	33	27	40.94	1.27
	0.53	18.1	59.5	0.126	32.5	27	33.16	1.03
	1.06	48.06	73	0.154	40	33	26.87	0.83
	2.13	79.44	73	0.190	38	35	26.75	0.83
	5.14	208.85	70	0.201	37	33	32.65	1.01
	11.2	450.42	73	0.227	38	35	29.49	0.91
24.65	0.2	17.12	54	0.102	33	21	31.54	0.98
	0.53	38.22	64	0.120	31	33	34.76	1.08
	1.06	94.87	59	0.136	30	29	37.59	1.17
	2.13	178.56	60	0.169	31	29	38.01	1.18
	5.14	408.36	70	0.192	38	32	31.59	0.98
	11.2	943.11	73	0.220	40	33	29.69	0.92

Table 2. Data obtained from cyclic voltammograms in 0.39 mM DmFc and 20 mM [NBuⁿ₄][BF₄] in scR32 at 360 K and \approx 17.6 MPa for various scan rates at a baffled 1 mm Ø Au disc.

$v / \text{V s}^{-1}$	E_{pa} / mV	E_{pc} / mV	$\Delta E_p / \text{mV}$	$E_{pa/2} / \text{mV}$	$E_{pc/2} / \text{mV}$	$E_{pa} - E_{pa/2} / \text{mV}$	$E_{pc} - E_{pc/2} / \text{mV}$	$i_{pa} / \mu\text{A}$	$i_{pc} / \mu\text{A}$	i_{pa}/i_{pc}
0.1	170	16	154	70	108	100	92	1.67	1.47	1.14
0.2	170	16	154	70	114	100	98	2.17	2.17	1.00
0.3	178	12	166	73	112	105	100	2.61	2.55	1.02
0.4	182	7	175	75	110	107	103	2.97	2.9	1.02
0.5	184	4.4	180	74	110	110	105.6	3.28	3.2	1.03
0.6	186	0.52	185	78	107	108	106.5	3.56	3.5	1.02
0.7	194	-3	197	78	106	116	109	3.81	3.62	1.05

0.8	194	-8	202	78.6	107	115	115	4.07	3.92	1.04
0.9	195	-7	202	78.6	105	116	112	4.27	4.14	1.03
1.0	199	-9	208	80	105	119	114	4.48	4.28	1.04