Supporting Information

Synthesis, Electrochemistry, and Reactivity of New Iridium(III) and Rhodium(III) Hydrides

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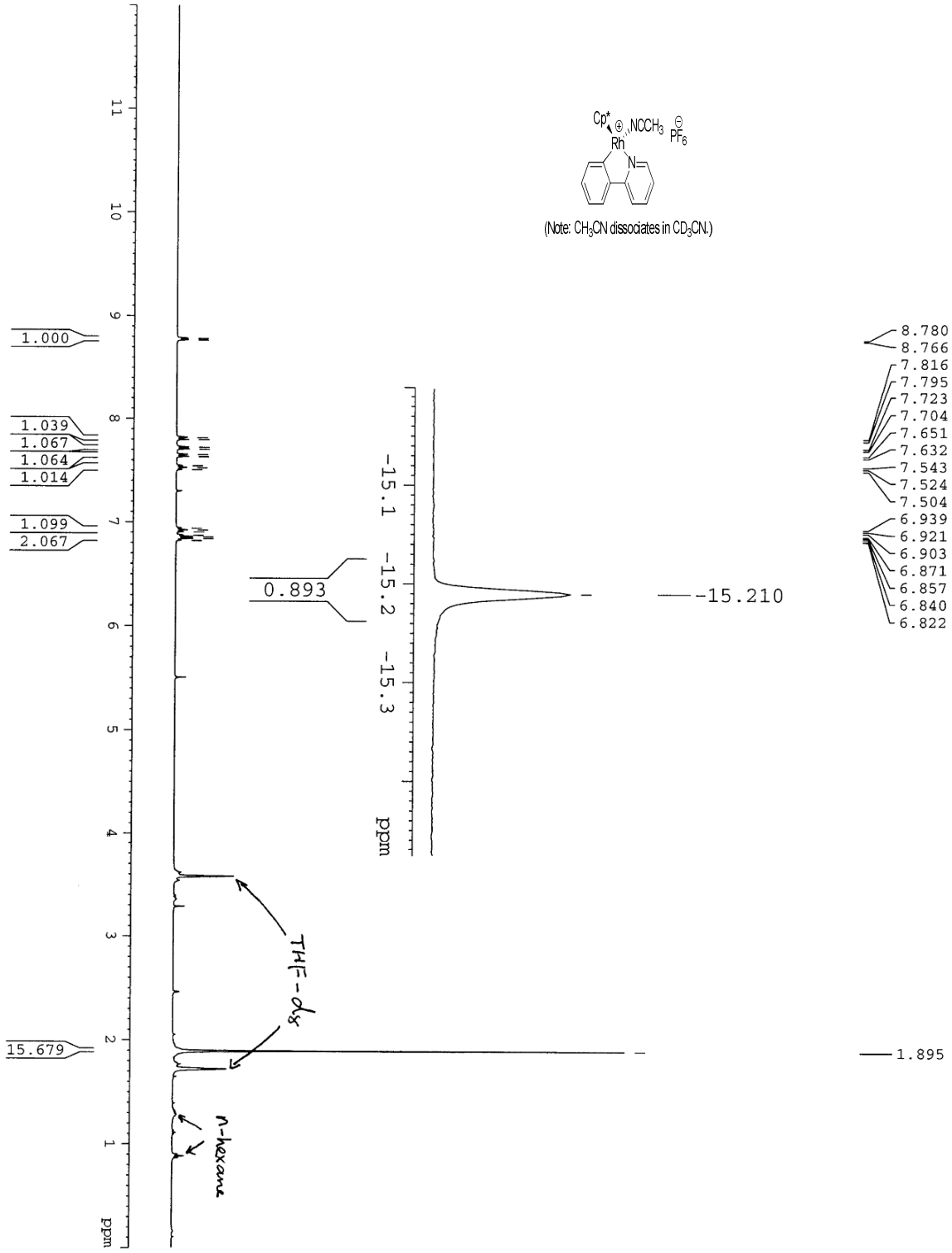
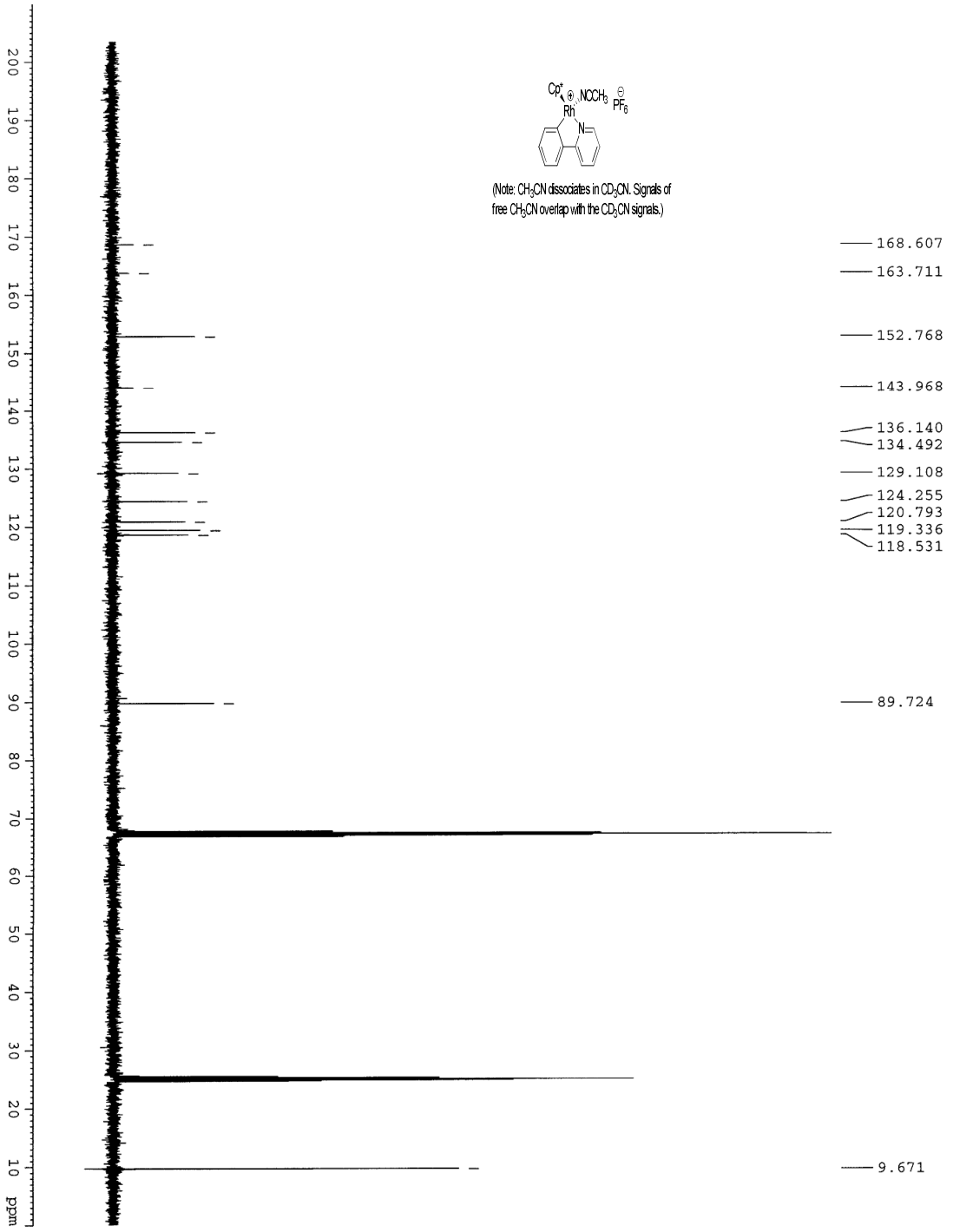
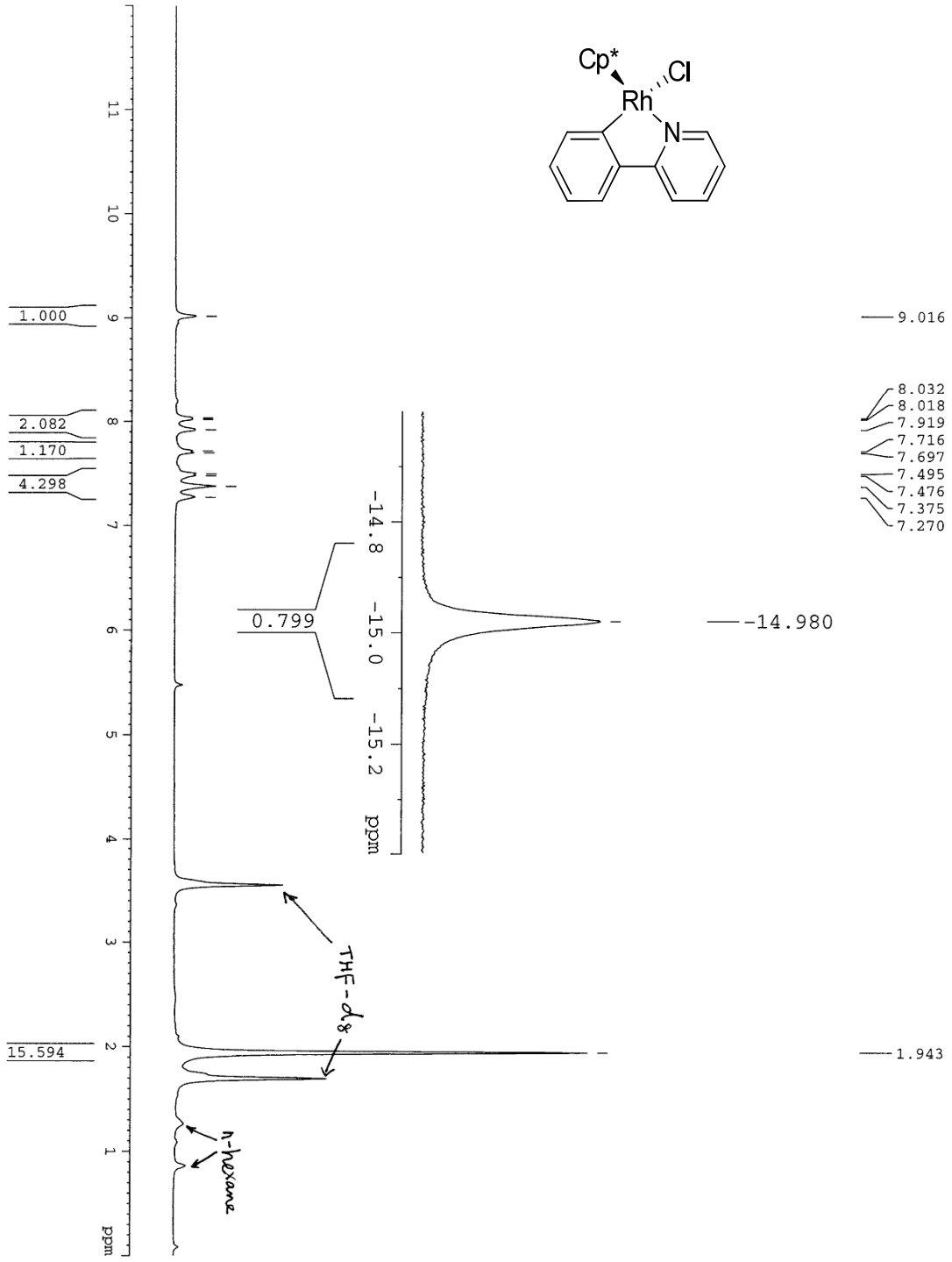
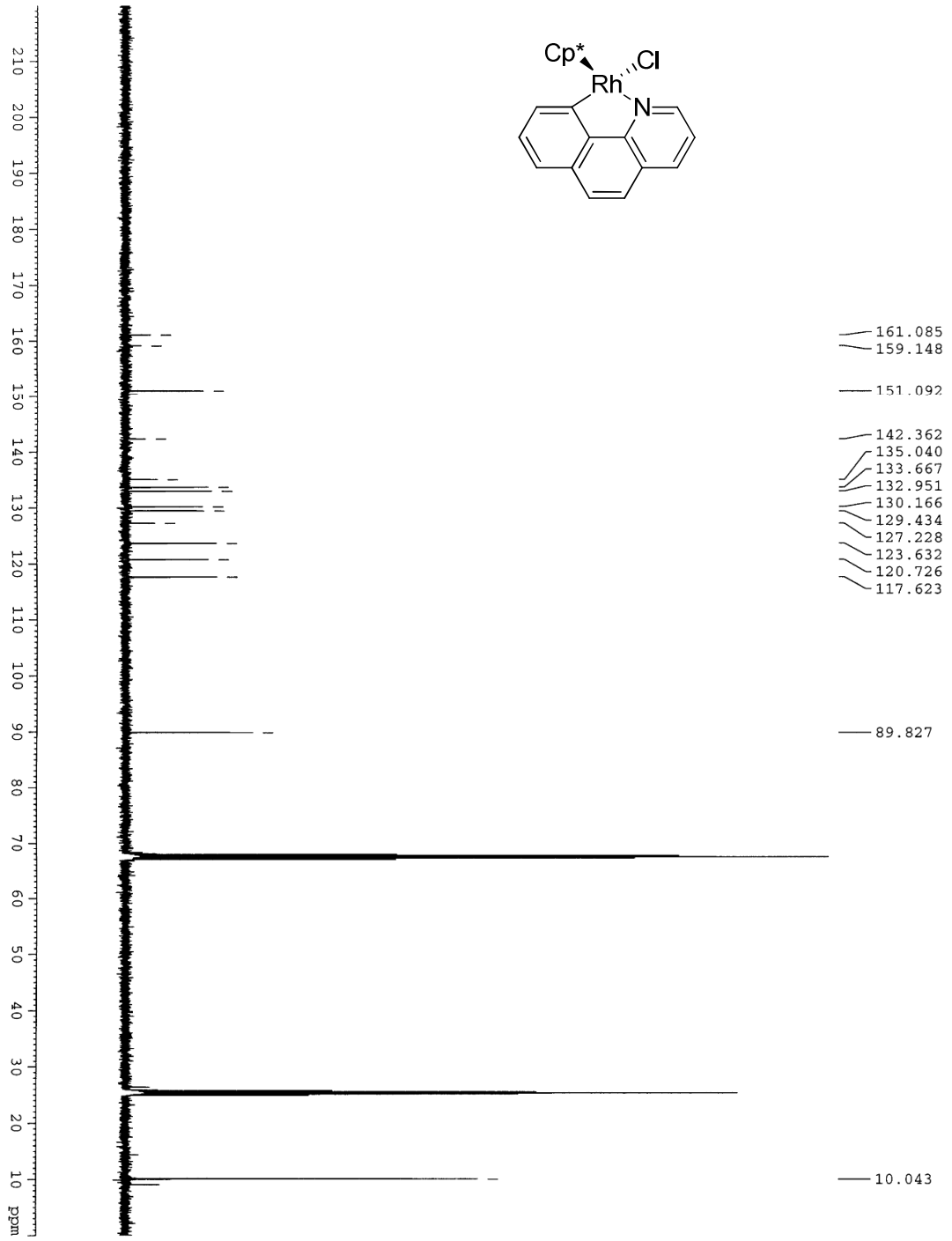
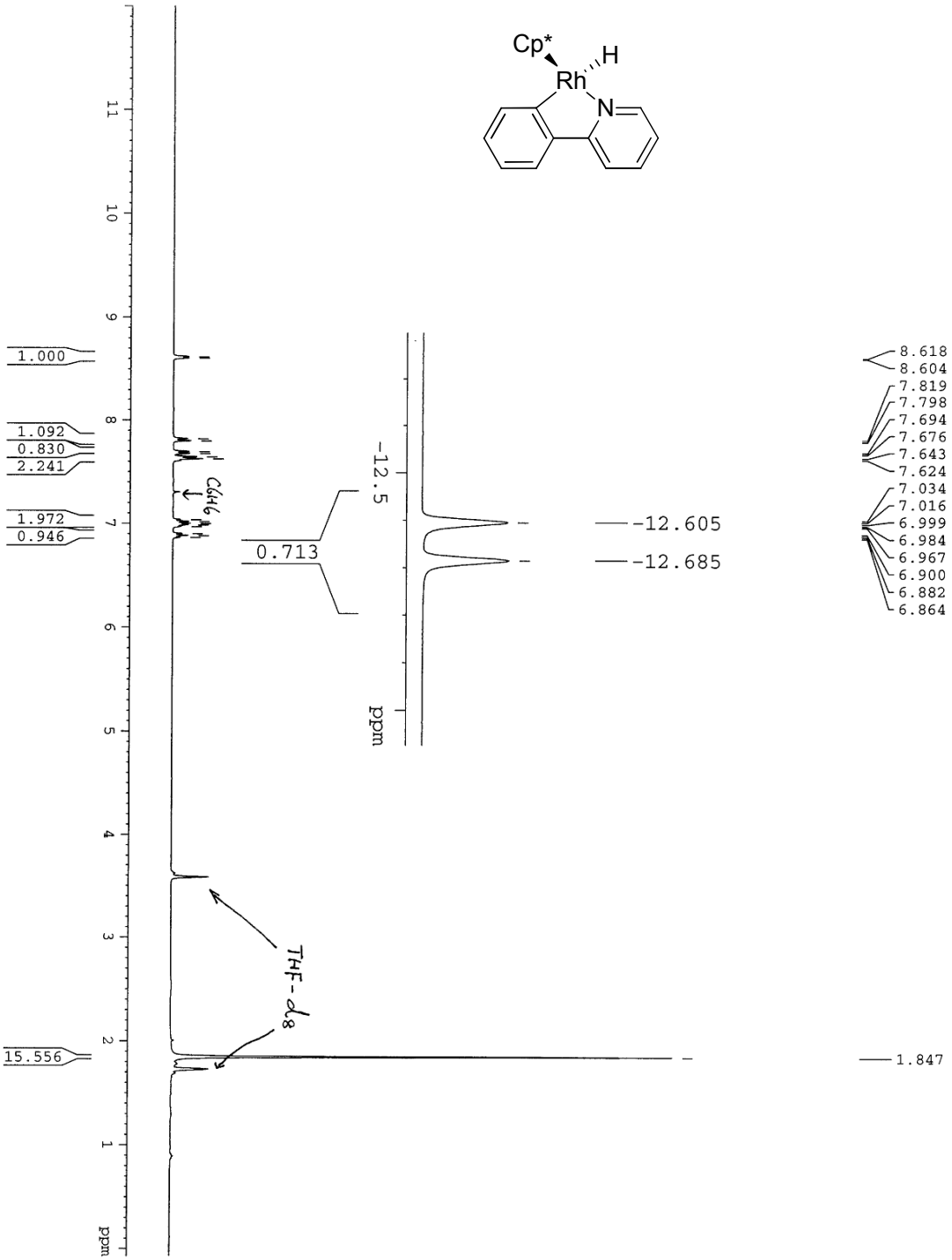
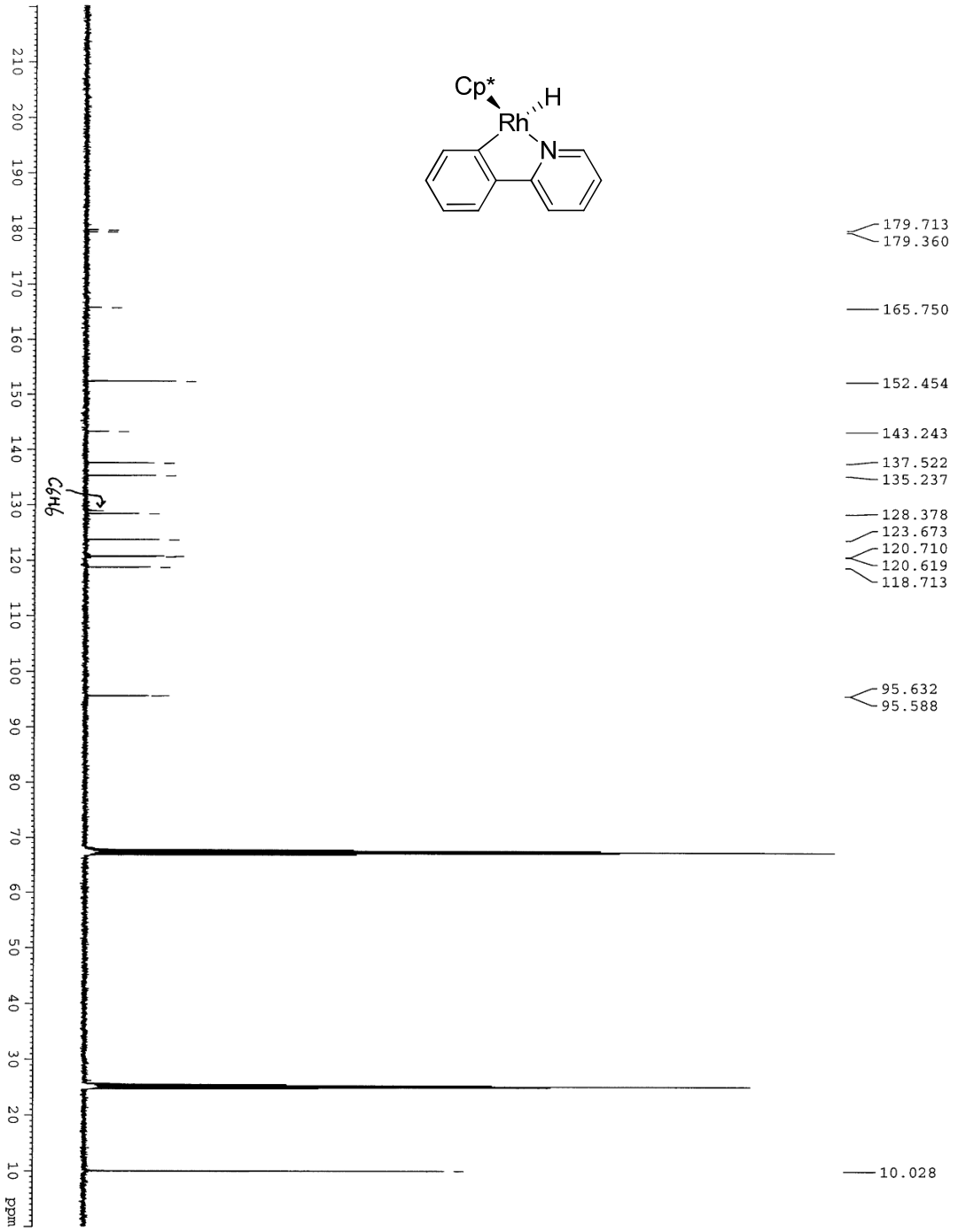

Cp*
NCCH3
Rh
N
PF6
(Note: CH3CN dissociates in CD3CN.)
1.000
1.039
1.067
1.064
1.014
1.099
2.067
15.679
0.893
-15.1
-15.2
-15.3
ppm
-15.210
8.780
8.766
7.816
7.795
7.723
7.704
7.651
7.632
7.543
7.524
7.504
6.939
6.921
6.903
6.871
6.857
6.840
6.822
1.895
THF-ds
n-hexane
ppm

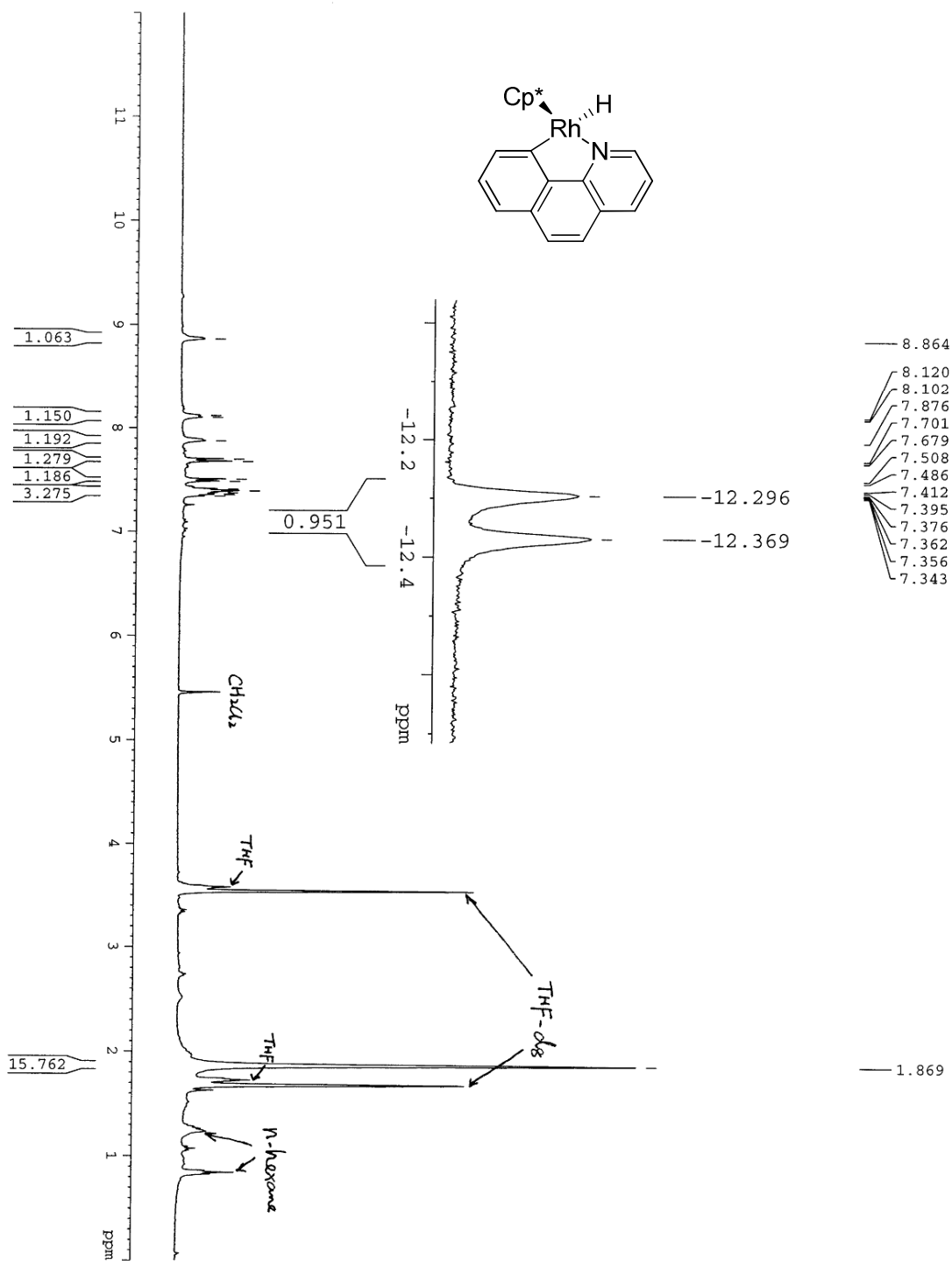

Cp*
Rh
NCCH3
PF6
N
(Note: CH3CN dissociates in CD3CN. Signals of free CH3CN overlap with the CD3CN signals.)
168.607
163.711
152.768
143.968
136.140
134.492
129.108
124.255
120.793
119.336
118.531
89.724
9.671
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

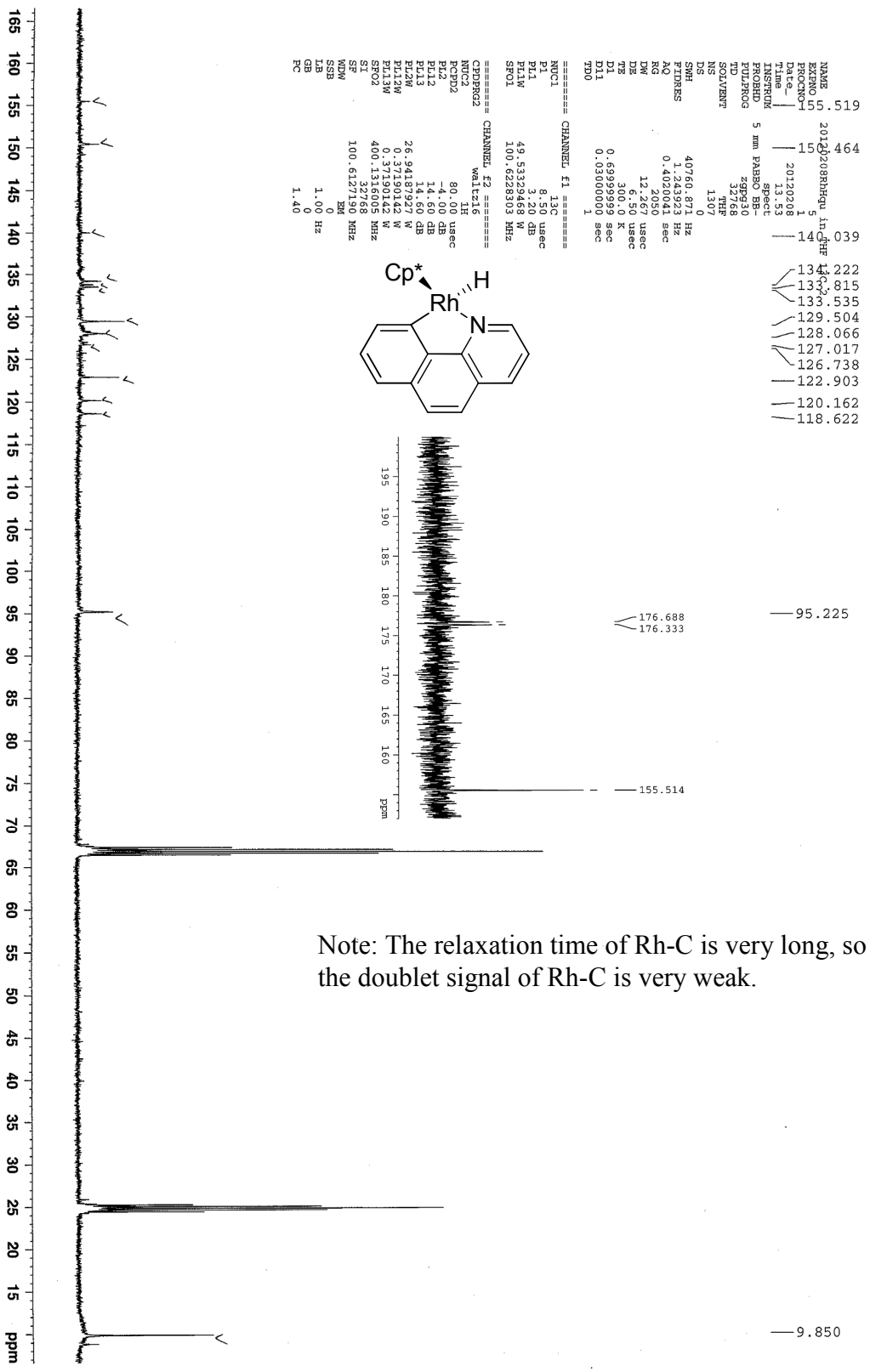

Cp*
Cl
Rh
N
1.000
2.082
1.170
4.298
15.594
9.016
8.032
8.018
7.919
7.716
7.697
7.495
7.476
7.375
7.270
-14.980
0.799
-14.8
-15.0
-15.2
ppm
THF-d8
n-hexane
1.943
11
10
9
8
7
6
5
4
3
2
1
ppm


Cp*
Rh
Cl
N
161.085
159.148
151.092
142.362
135.040
133.667
132.951
130.166
129.434
127.228
123.632
120.726
117.623
89.827
10.043
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

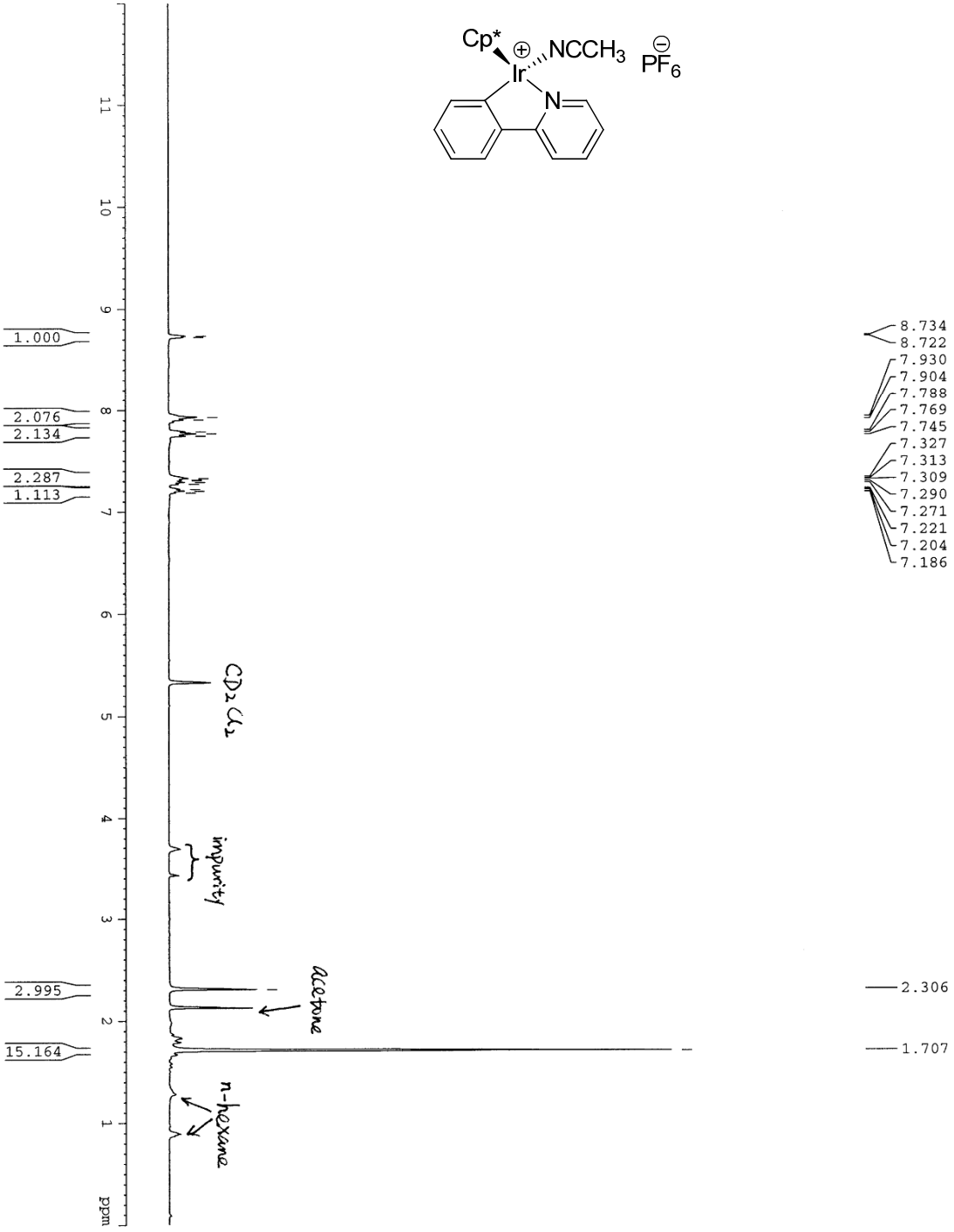

Cp*
Rh
H
N
1.000
1.092
0.830
2.241
1.972
0.946
15.556
C6H6
THF-d8
-12.5
0.713
ppm
-12.605
-12.685
8.618
8.604
7.819
7.798
7.694
7.676
7.643
7.624
7.034
7.016
6.999
6.984
6.967
6.900
6.882
6.864
1.847

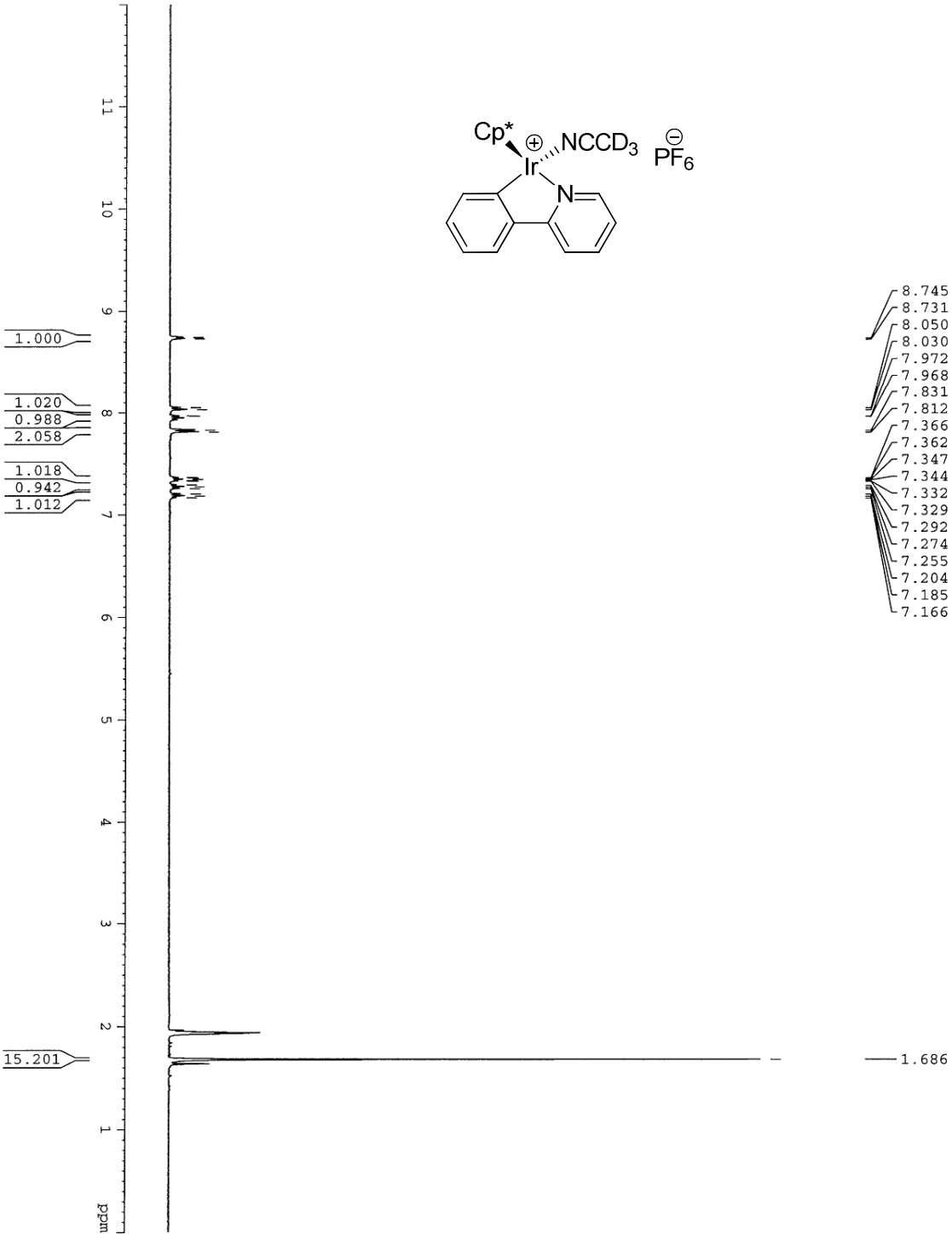

Cp*
Rh
H
N
C6H6
179.713
179.360
165.750
152.454
143.243
137.522
135.237
128.378
123.673
120.710
120.619
118.713
95.632
95.588
10.028
ppm

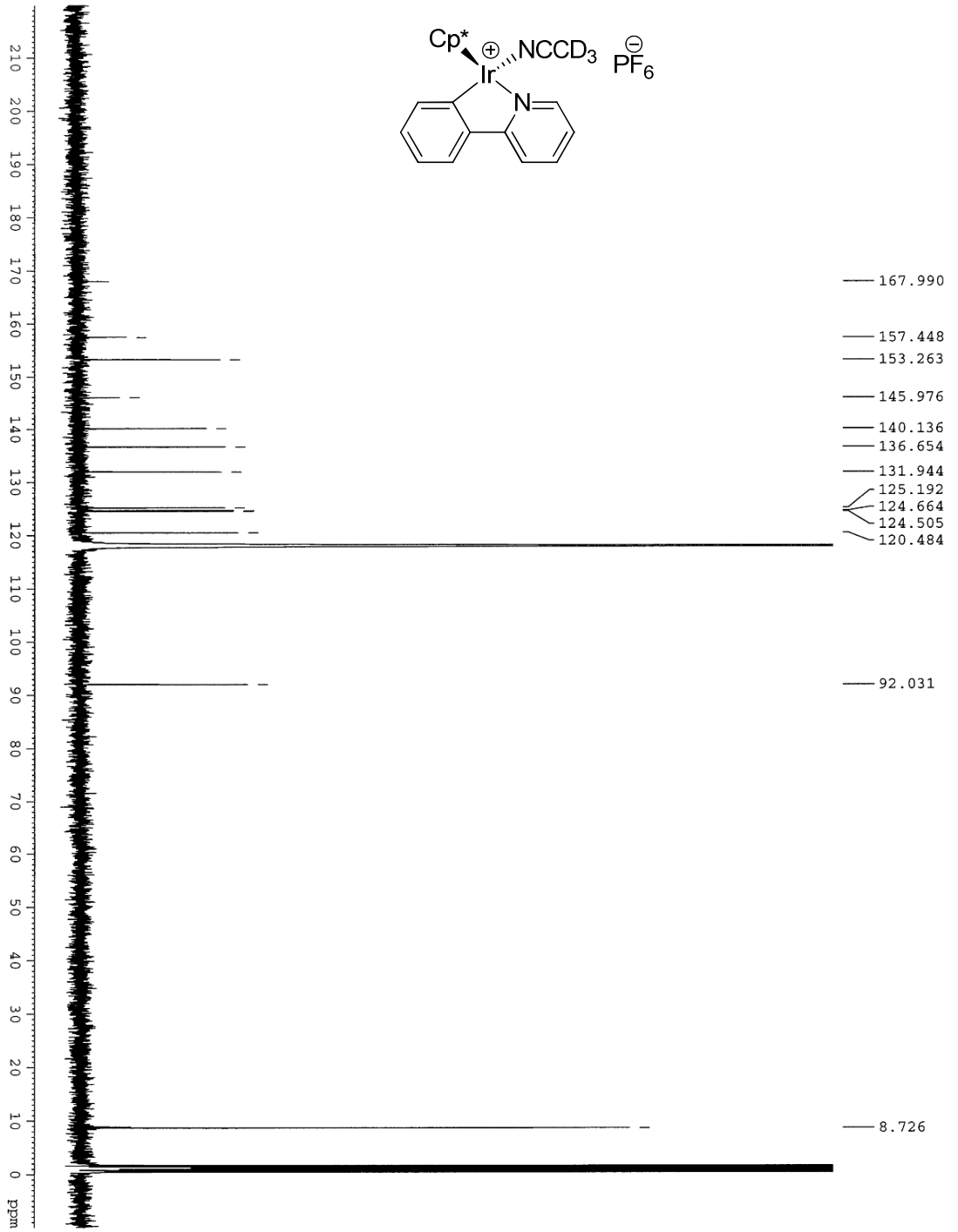

Cp*
Rh
H
N
1.063
1.150
1.192
1.279
1.186
3.275
15.762
0.951
-12.2
-12.4
ppm
-12.296
-12.369
8.864
8.120
8.102
7.876
7.701
7.679
7.508
7.486
7.412
7.395
7.376
7.362
7.356
7.343
1.869
CH2Cl2
THF
THF
THF-d8
n-hexane
ppm

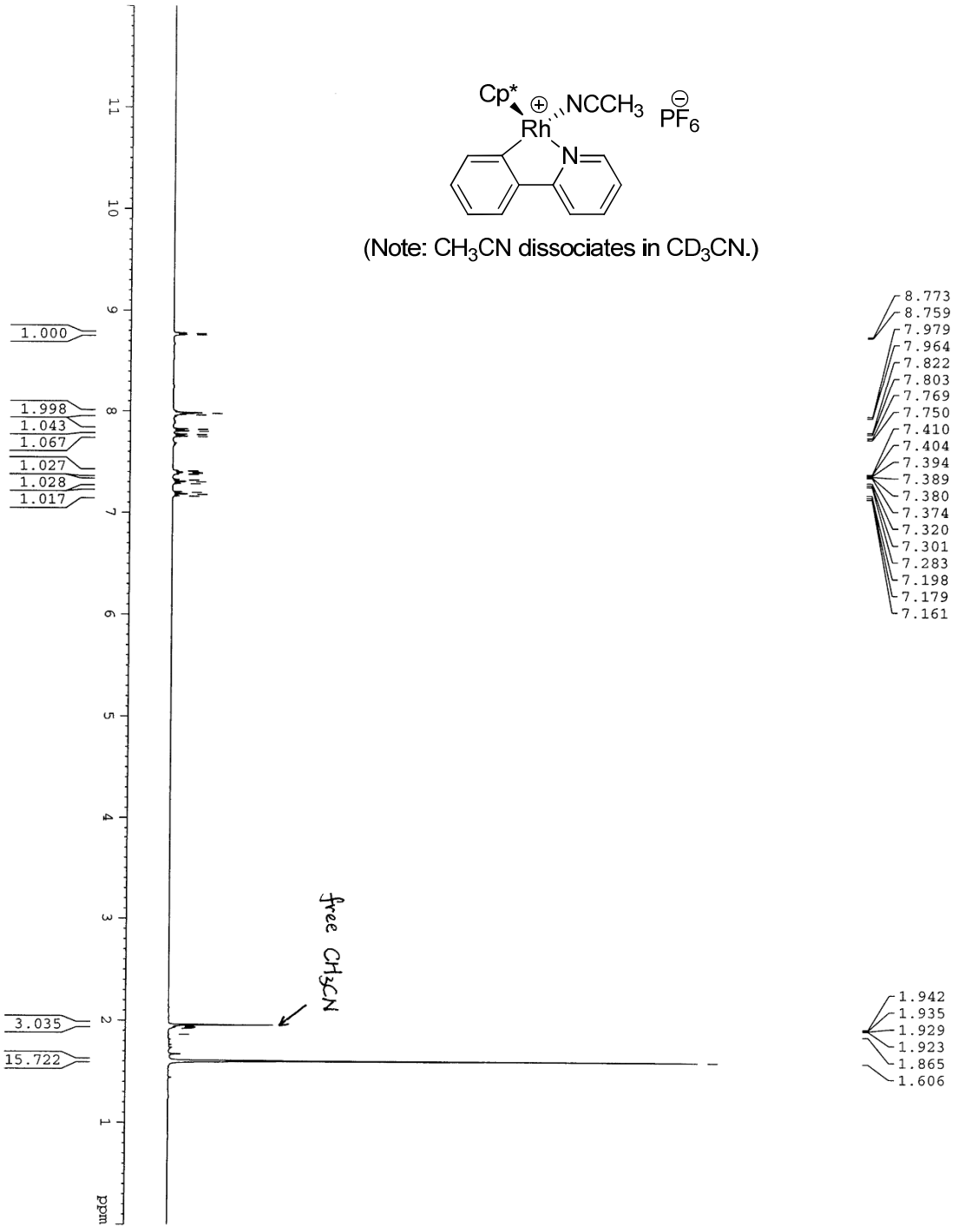


Note: The relaxation time of Rh-C is very long, so the doublet signal of Rh-C is very weak.


Cp*
NCCH3
PF6
Ir
N
1.000
2.076
2.134
2.287
1.113
2.995
15.164
8.734
8.722
7.930
7.904
7.788
7.769
7.745
7.327
7.313
7.309
7.290
7.271
7.221
7.204
7.186
2.306
1.707
CD2Cl2
impurity
acetone
n-hexane
ppm


Cp*
Ir
NCCD3
PF6
N
8.745
8.731
8.050
8.030
7.972
7.968
7.831
7.812
7.366
7.362
7.347
7.344
7.332
7.329
7.292
7.274
7.255
7.204
7.185
7.166
1.000
1.020
0.988
2.058
1.018
0.942
1.012
15.201
1.686
ppm


Cp*
NCCD3
PF6
Ir
N
167.990
157.448
153.263
145.976
140.136
136.654
131.944
125.192
124.664
124.505
120.484
92.031
8.726
ppm


Cp*
NCCH3
PF6
Rh
N
(Note: CH3CN dissociates in CD3CN.)
8.773
8.759
7.979
7.964
7.822
7.803
7.769
7.750
7.410
7.404
7.394
7.389
7.380
7.374
7.320
7.301
7.283
7.198
7.179
7.161
1.000
1.998
1.043
1.067
1.027
1.028
1.017
free CH3CN
3.035
15.722
1.942
1.935
1.929
1.923
1.865
1.606
ppm

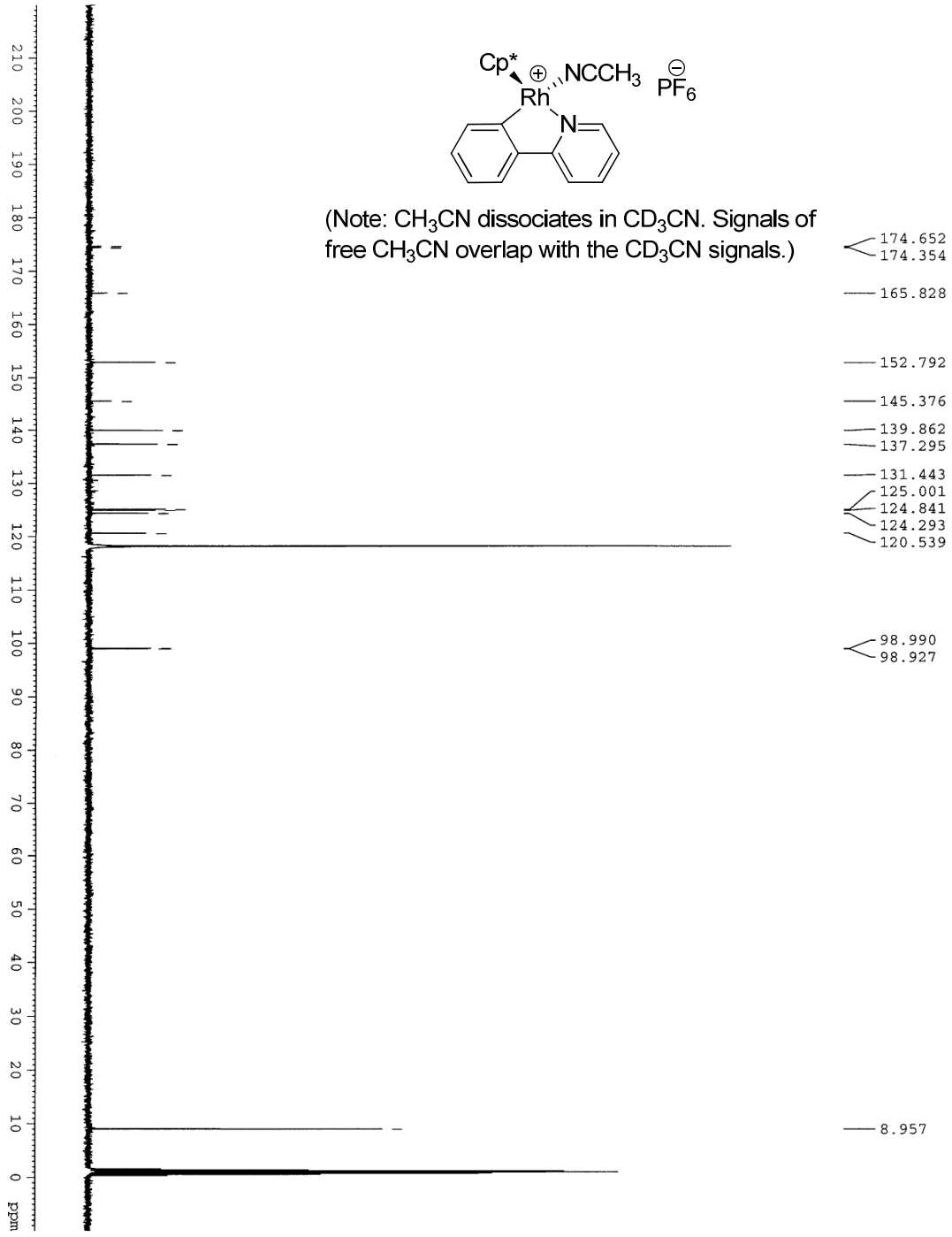
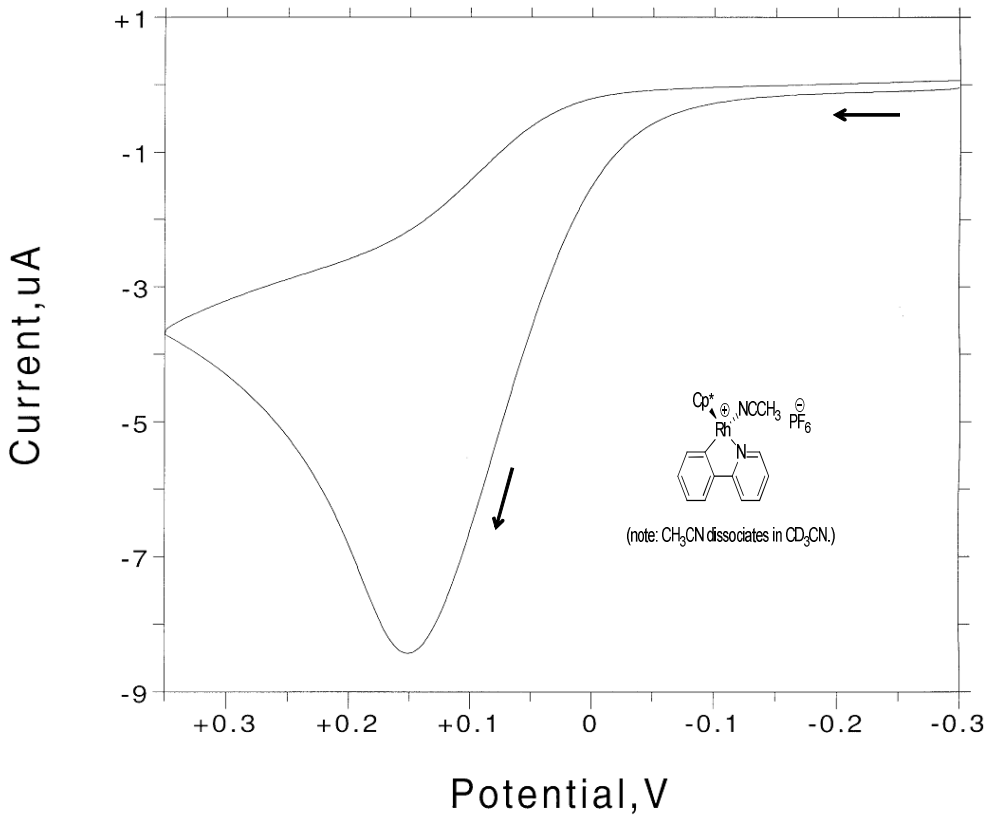
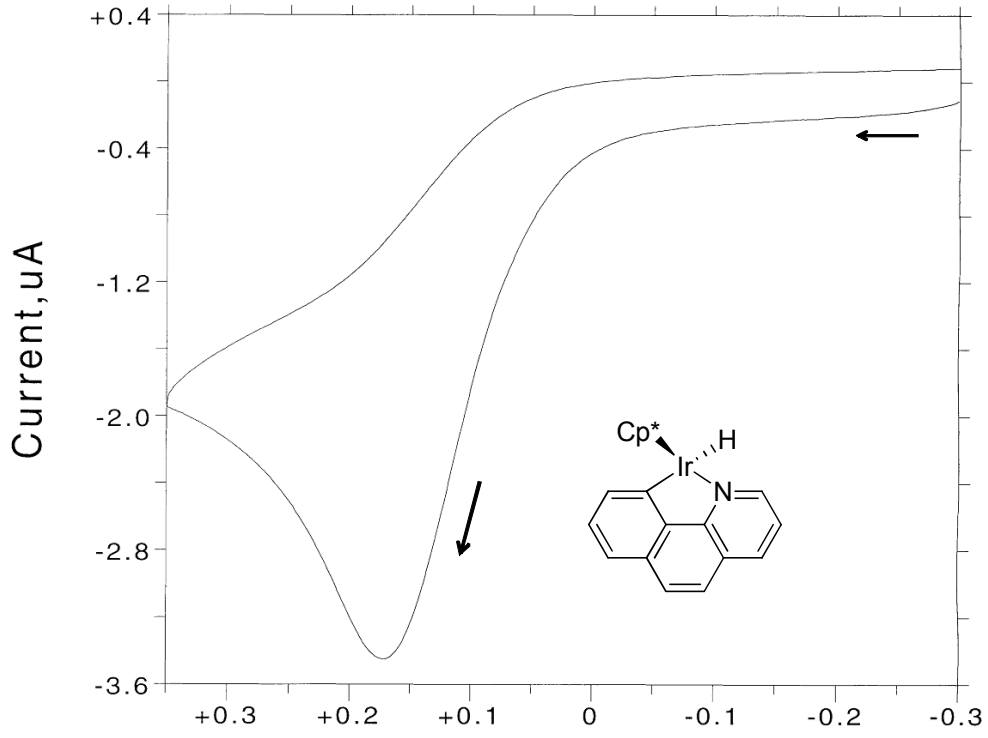
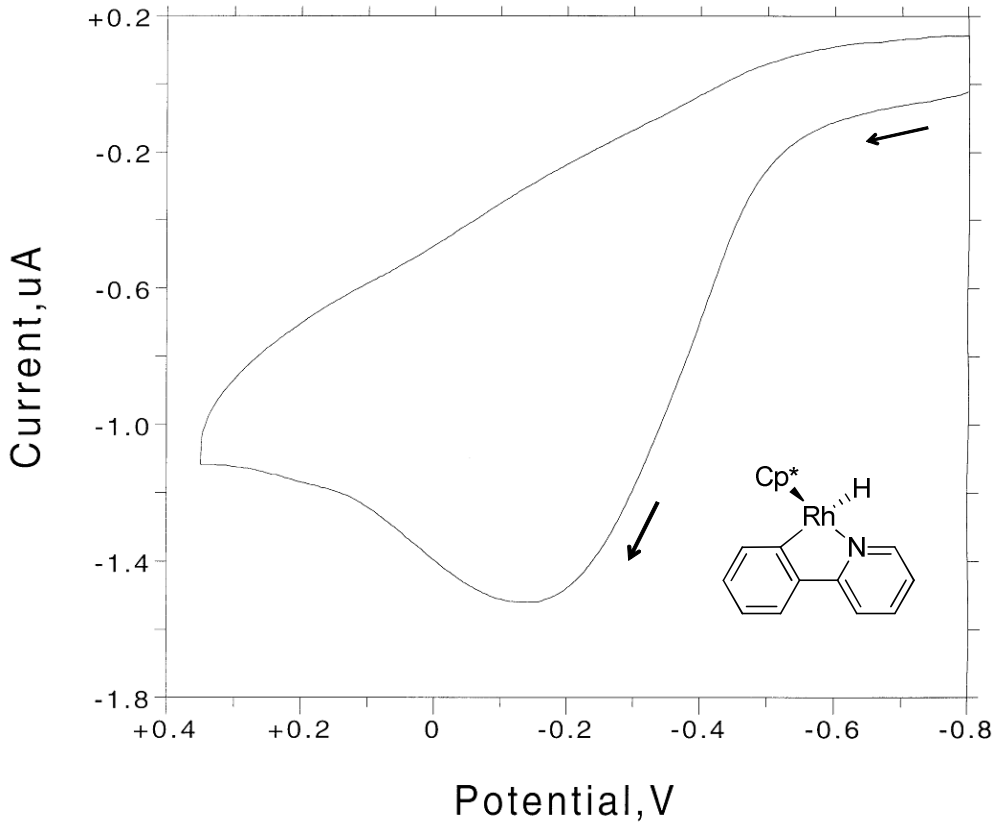

Cp*
NCCH3
PF6
Rh
N
(Note: CH3CN dissociates in CD3CN. Signals of free CH3CN overlap with the CD3CN signals.)
174.652
174.354
165.828
152.792
145.376
139.862
137.295
131.443
125.001
124.841
124.293
120.539
98.990
98.927
8.957
ppm

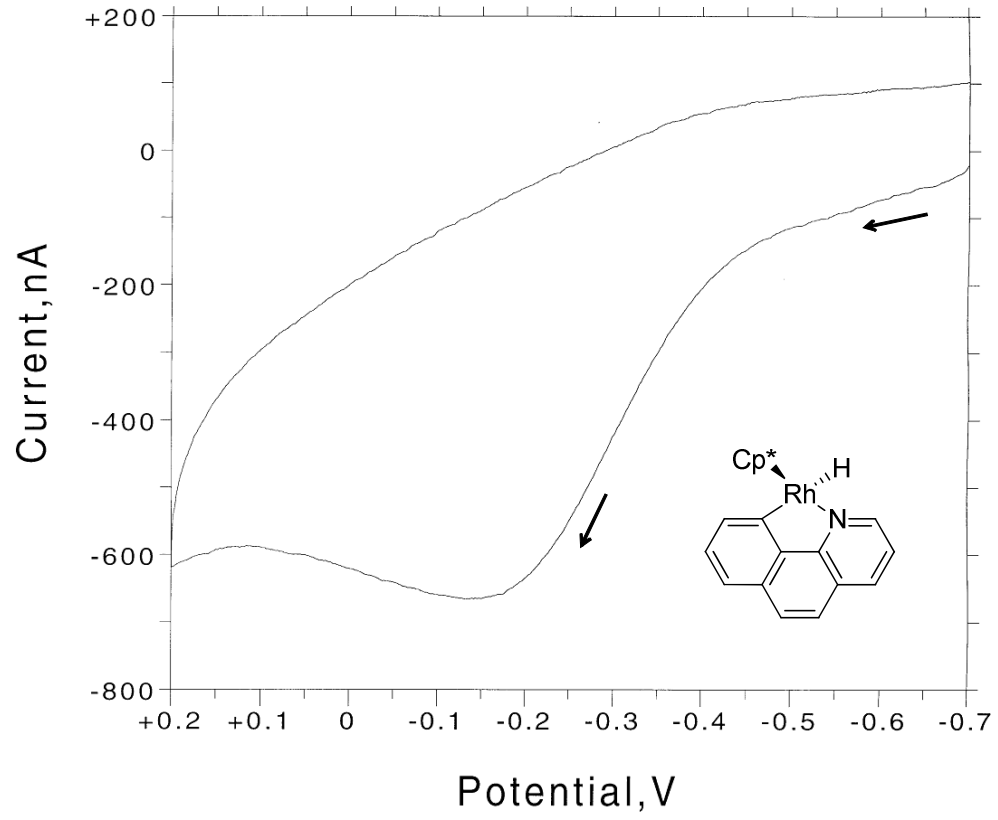
Table 1: Crystal, intensity collection and refinement data for 9 and 9'.

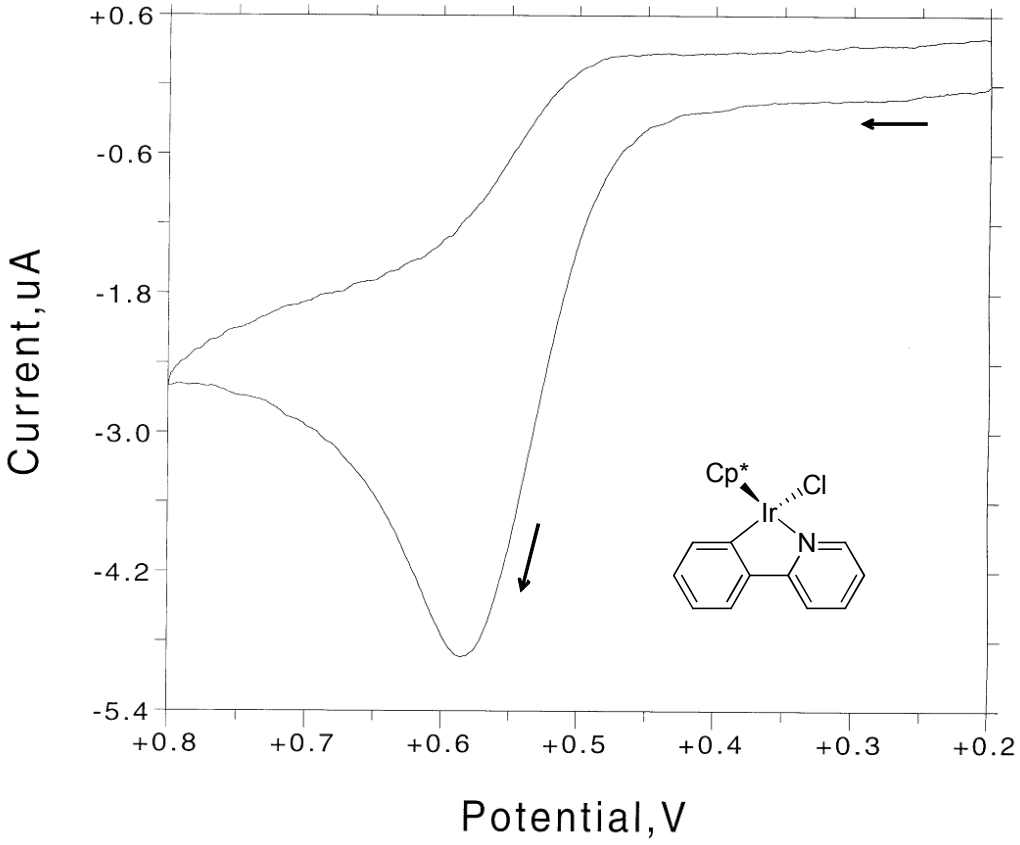
	9	**9'**
lattice	Monoclinic	Monoclinic
formula	$C_{23}H_{26}F_6IrN_2P$	$C_{23}H_{26}F_6N_2PRh$
formula weight	667.63	578.34
space group	$P2_1/c$	$P2_1/c$
a/Å	23.294(2)	23.252(2)
b/Å	13.2622(12)	13.3085(12)
c/Å	15.6968(15)	15.6748(14)
α/°	90	90
β/°	101.8780(10)	101.9910(10)
γ/°	90	90
V/Å^3	4745.4(8)	4744.8(7)
Z	4	4
temperature (K)	150(2)	150(2)
radiation (λ, Å)	0.71073	0.71073
ρ (calcd.) g cm^{-3}	1.869	1.619
μ (Mo Kα), mm^{-1}	5.755	0.848
θ max, deg.	32.71	31.82
no. of data collected	80448	79872
no. of data	16687	16127
no. of parameters	646	682
R_1 [$I > 2\sigma(I)$]	0.0331	0.0364
wR_2 [$I > 2\sigma(I)$]	0.0643	0.0781
R_1 [all data]	0.0564	0.0557
wR_2 [all data]	0.0721	0.0861
GOF	1.009	1.023

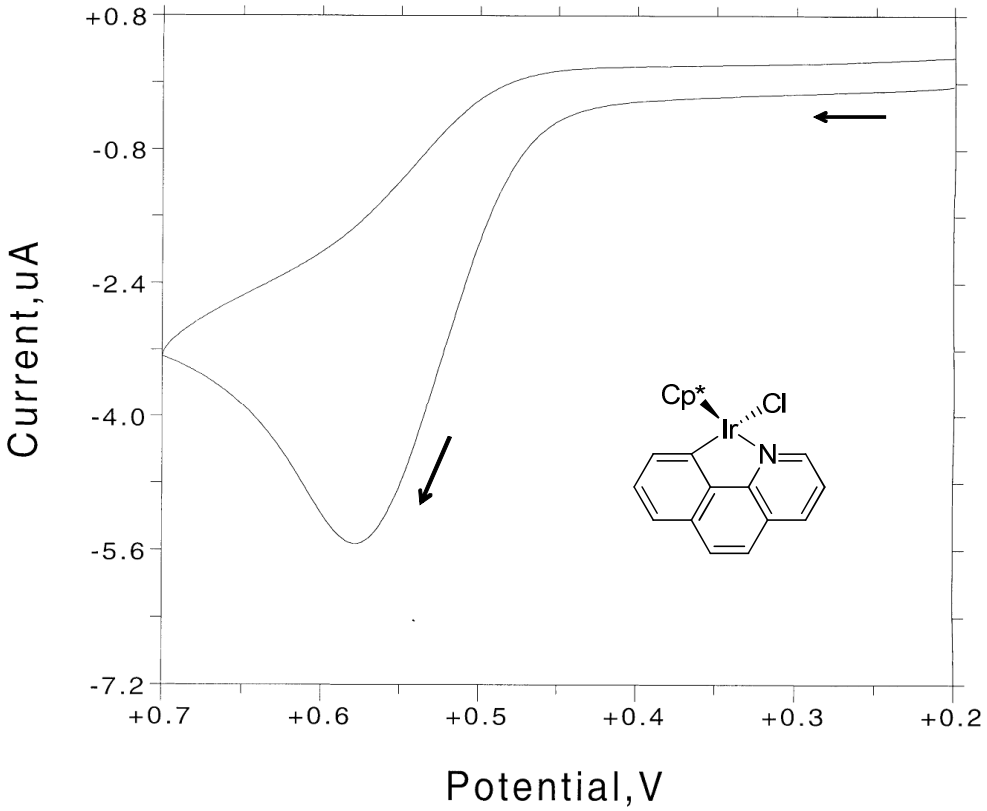

+1
-1
-3
-5
-7
-9
Current,uA
+0.3
+0.2
+0.1
0
-0.1
-0.2
-0.3
Potential,V
Cp*
NCCH3
PF6
Rh
N
(note: CH3CN dissociates in CD3CN.)

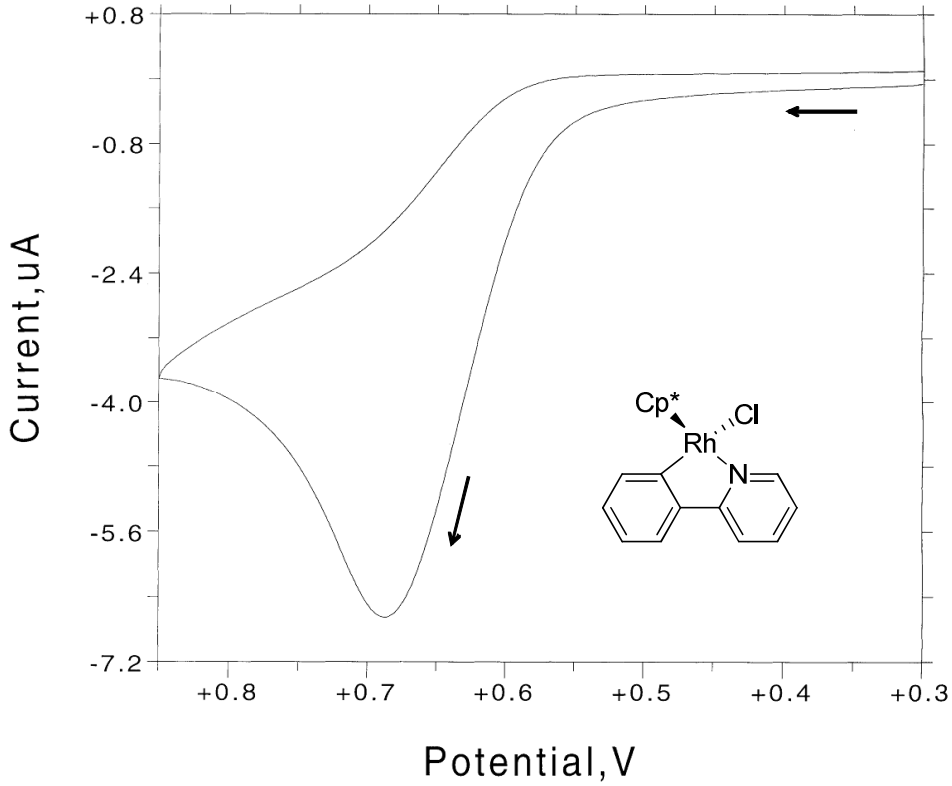

+0.4
-0.4
-1.2
-2.0
-2.8
-3.6
Current,uA
+0.3
+0.2
+0.1
0
-0.1
-0.2
-0.3
Cp*
H
Ir
N

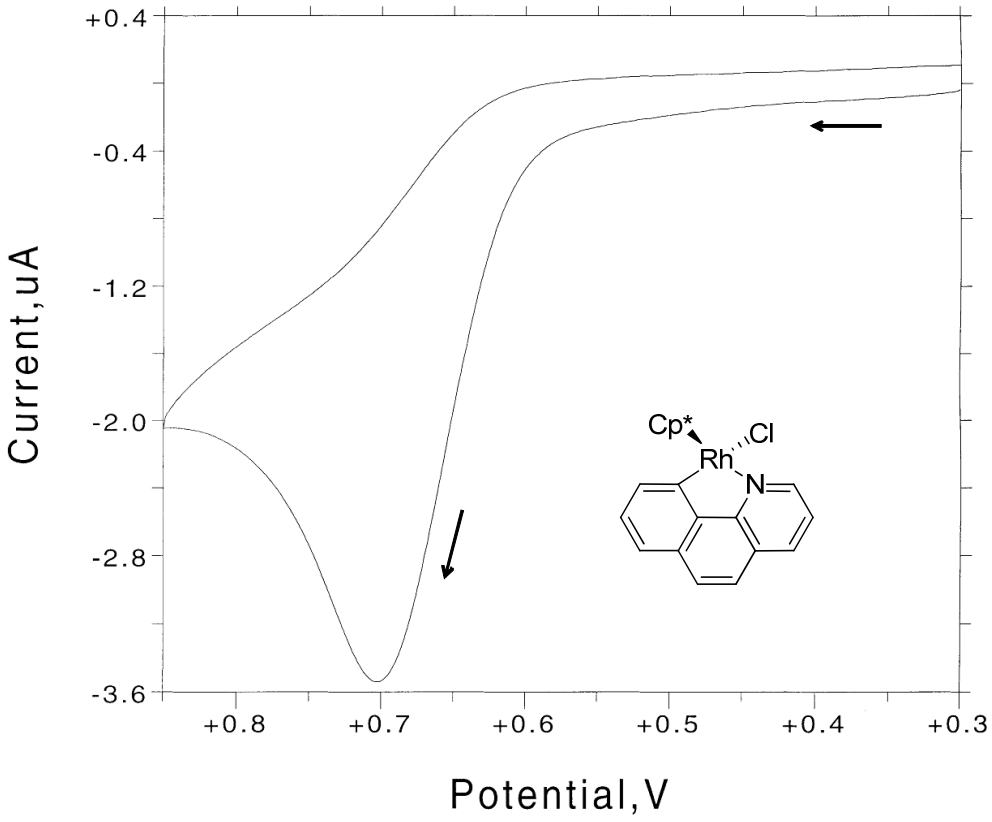

+0.2
-0.2
-0.6
-1.0
-1.4
-1.8
Current,uA
+0.4
+0.2
0
-0.2
-0.4
-0.6
-0.8
Potential,V
Cp*
Rh
H
N

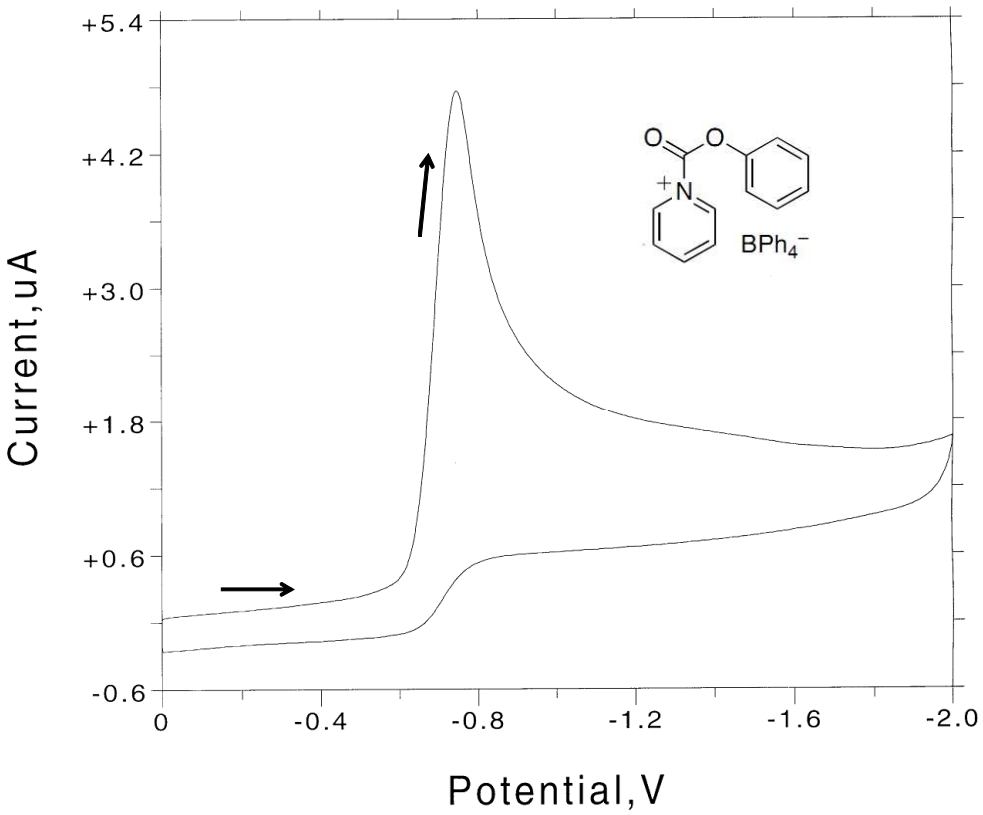

+200
0
-200
-400
-600
-800
Current,nA
+0.2
+0.1
0
-0.1
-0.2
-0.3
-0.4
-0.5
-0.6
-0.7
Potential,V
Cp*
Rh
H
N


+0.6
-0.6
-1.8
-3.0
-4.2
-5.4
Current,uA
+0.8
+0.7
+0.6
+0.5
+0.4
+0.3
+0.2
Potential,V
Cp*
Ir
Cl
N


+0.8
-0.8
-2.4
-4.0
-5.6
-7.2
Current,uA
+0.7
+0.6
+0.5
+0.4
+0.3
+0.2
Potential,V
Cp*
Ir
Cl
N


+0.8
-0.8
-2.4
-4.0
-5.6
-7.2
Current,uA
+0.8
+0.7
+0.6
+0.5
+0.4
+0.3
Potential,V
Cp*
Rh
Cl
N


+0.4
-0.4
-1.2
-2.0
-2.8
-3.6
Current,uA
+0.8
+0.7
+0.6
+0.5
+0.4
+0.3
Potential,V
Cp*
Rh
Cl
N


+5.4
+4.2
+3.0
+1.8
+0.6
-0.6
Current,uA
0
-0.4
-0.8
-1.2
-1.6
-2.0
Potential,V
O
O
+N
BPh4−