

Supporting Information

A Carbanion Induced Ring Switching Synthesis of Spiranes: An Unprecedented Approach

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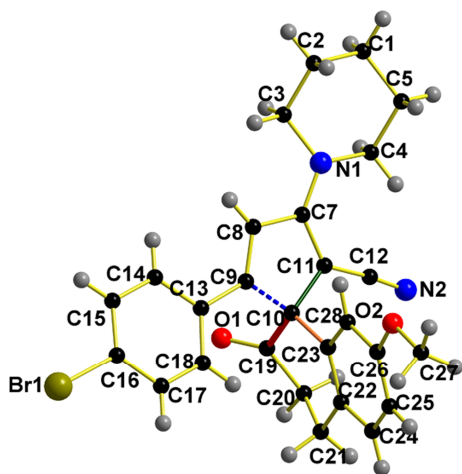
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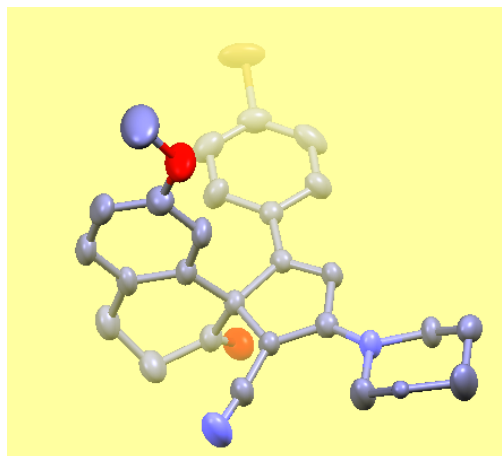
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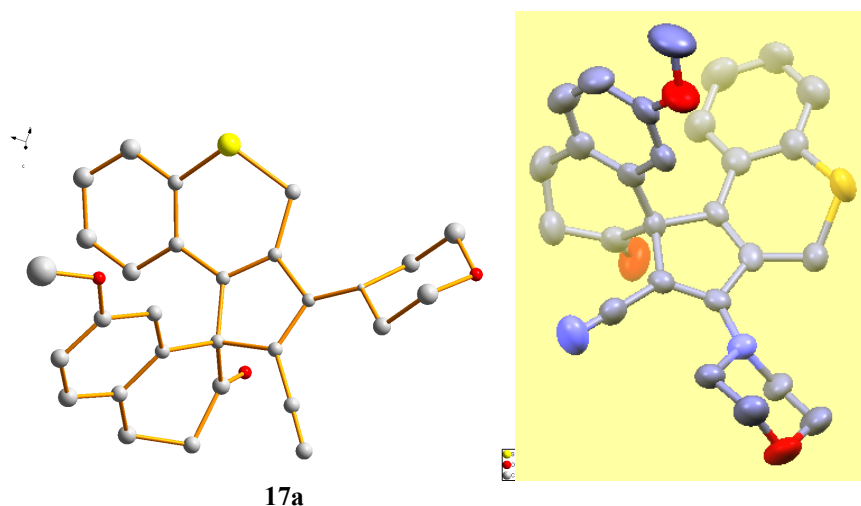
Perspective view of **11d** Showing absolute configuration:



11d



Perspective view of **17a** Showing absolute configuration:



XYZ Coordinates for dimer of **11d**

Counterpoise: corrected energy = -7750.365223271178

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	8	0	-4.380078	0.322314	2.654824
2	7	0	-8.680660	1.449877	0.414840
3	6	0	-5.534611	-0.490406	0.710929
4	6	0	-7.443283	0.930161	0.496376
5	6	0	-5.290949	-1.686359	-0.222109
6	6	0	-5.173863	0.834737	0.060790
7	6	0	-4.809479	-0.638486	2.057514
8	6	0	-5.483547	-2.622867	-2.441345
9	6	0	-4.603097	-2.832613	0.197093
10	6	0	-6.262343	1.599793	-0.037632
11	1	0	-6.268007	2.455064	-0.402075
12	6	0	-7.019701	-0.294633	1.000789
13	6	0	-3.821801	1.191166	-0.383453
14	6	0	-5.749482	-1.613088	-1.530890
15	1	0	-6.241003	-0.871591	-1.801396
16	6	0	-11.089578	1.036401	0.155851
17	1	0	-11.846189	0.590563	0.566545
18	1	0	-10.993370	0.695781	-0.747437
19	6	0	-4.729349	-2.043357	2.590435
20	1	0	-4.221300	-2.045872	3.417139
21	1	0	-5.622975	-2.365789	2.785347
22	6	0	-10.145910	3.207213	-0.504458
23	1	0	-10.050446	2.915827	-1.424612
24	1	0	-10.288567	4.166707	-0.507639
25	6	0	-4.075013	-2.958800	1.601420
26	1	0	-4.195106	-3.874247	1.898049
27	1	0	-3.121684	-2.775995	1.592162
28	7	0	-8.256427	-2.131932	2.244133
29	6	0	-3.529050	2.509394	-0.693821
30	1	0	-4.188229	3.158727	-0.595674
31	6	0	-2.812594	0.258444	-0.519264
32	1	0	-2.979312	-0.629368	-0.295240
33	6	0	-4.790351	-3.742053	-2.031748
34	1	0	-4.604279	-4.424409	-2.636241
35	6	0	-4.378745	-3.843613	-0.731040
36	1	0	-3.933826	-4.613882	-0.458565
37	6	0	-8.889276	2.896967	0.245522
38	1	0	-8.132620	3.273330	-0.232020

39	1	0	-8.927635	3.317076	1.118900
40	6	0	-9.844154	0.737335	0.939522
41	1	0	-9.982276	0.990615	1.865827
42	1	0	-9.672338	-0.216165	0.913758
43	6	0	-7.746142	-1.281752	1.680184
44	6	0	-11.342583	2.529939	0.110639
45	1	0	-12.135568	2.714351	-0.418068
46	1	0	-11.489805	2.867896	1.007654
47	6	0	-1.573710	0.611367	-0.976866
48	1	0	-0.909686	-0.032428	-1.068920
49	6	0	-2.274148	2.877332	-1.146048
50	1	0	-2.082436	3.765036	-1.344931
51	6	0	-1.316847	1.904635	-1.296427
52	35	0	0.396711	2.420766	-1.905714
53	8	0	-5.947805	-2.419985	-3.709696
54	6	0	-5.648175	-3.390866	-4.696046
55	1	0	-6.032731	-3.124701	-5.534387
56	1	0	-6.012970	-4.238623	-4.432549
57	1	0	-4.695301	-3.467590	-4.790166
58	35	0	-0.396711	-2.420766	1.905714
59	1	0	3.121684	2.775994	-1.592161
60	8	0	4.380079	-0.322313	-2.654824
61	7	0	8.680659	-1.449876	-0.414841
62	6	0	5.534612	0.490406	-0.710930
63	6	0	7.443283	-0.930162	-0.496375
64	6	0	5.290949	1.686358	0.222109
65	6	0	5.173863	-0.834738	-0.060790
66	6	0	4.809480	0.638486	-2.057515
67	6	0	5.483548	2.622867	2.441346
68	6	0	4.603097	2.832613	-0.197093
69	6	0	6.262342	-1.599793	0.037631
70	1	0	6.268007	-2.455065	0.402075
71	6	0	7.019701	0.294633	-1.000789
72	6	0	3.821802	-1.191166	0.383453
73	6	0	5.749482	1.613089	1.530890
74	1	0	6.241004	0.871591	1.801396
75	6	0	11.089578	-1.036401	-0.155850
76	1	0	11.846188	-0.590563	-0.566545
77	1	0	10.993371	-0.695781	0.747437
78	6	0	4.729349	2.043357	-2.590435
79	1	0	4.221301	2.045872	-3.417140
80	1	0	5.622975	2.365789	-2.785348
81	6	0	10.145909	-3.207213	0.504457
82	1	0	10.050446	-2.915826	1.424612
83	1	0	10.288568	-4.166707	0.507639
84	6	0	4.075013	2.958801	-1.601420
85	1	0	4.195106	3.874247	-1.898049
86	7	0	8.256427	2.131932	-2.244133
87	6	0	3.529051	-2.509394	0.693821
88	1	0	4.188229	-3.158727	0.595674
89	6	0	2.812594	-0.258444	0.519265
90	1	0	2.979311	0.629368	0.295240
91	6	0	4.790351	3.742054	2.031748
92	1	0	4.604279	4.424409	2.636241
93	6	0	4.378746	3.843613	0.731041
94	1	0	3.933825	4.613883	0.458564
95	6	0	8.889275	-2.896968	-0.245522
96	1	0	8.132620	-3.273330	0.232020
97	1	0	8.927635	-3.317075	-1.118899
98	6	0	9.844154	-0.737335	-0.939522
99	1	0	9.982277	-0.990615	-1.865827
100	1	0	9.672338	0.216165	-0.913757

101	6	0	7.746142	1.281752	-1.680185
102	6	0	11.342583	-2.529939	-0.110639
103	1	0	12.135569	-2.714351	0.418068
104	1	0	11.489805	-2.867896	-1.007653
105	6	0	1.573710	-0.611366	0.976866
106	1	0	0.909686	0.032429	1.068920
107	6	0	2.274148	-2.877331	1.146048
108	1	0	2.082436	-3.765036	1.344932
109	6	0	1.316847	-1.904634	1.296427
110	8	0	5.947805	2.419985	3.709697
111	6	0	5.648175	3.390866	4.696046
112	1	0	6.032731	3.124702	5.534387
113	1	0	6.012970	4.238623	4.432548
114	1	0	4.695301	3.467590	4.790166

Optimized XYZ coordinates for monomer of **11d**

Energy = -3875.18397073

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	8	0	0.024009	-0.468193	-3.109164
2	7	0	-3.503178	-1.673681	0.190788
3	6	0	-0.730267	0.465416	-1.027907
4	6	0	-2.379088	-1.069438	-0.232494
5	6	0	-0.404608	1.775305	-0.294168
6	6	0	-0.093087	-0.744148	-0.364747
7	6	0	-0.354265	0.516894	-2.516869
8	6	0	-0.177738	2.939639	1.811065
9	6	0	0.044602	2.917727	-0.969335
10	6	0	-1.043599	-1.577538	0.061603
11	1	0	-0.875801	-2.383534	0.493521
12	6	0	-2.213388	0.119966	-0.933804
13	6	0	1.354571	-0.938618	-0.227149
14	6	0	-0.539196	1.814907	1.087442
15	1	0	-0.874984	1.072094	1.534722
16	6	0	-5.817363	-1.427188	0.981177
17	1	0	-6.690537	-1.092799	0.724950
18	1	0	-5.551857	-0.979497	1.799887
19	6	0	-0.546233	1.853954	-3.179823
20	1	0	-0.245332	1.803042	-4.100923
21	1	0	-1.489386	2.079721	-3.183328
22	6	0	-4.525516	-3.426501	1.591769
23	1	0	-4.251702	-3.025708	2.431467
24	1	0	-4.562563	-4.387721	1.716956
25	6	0	0.218950	2.925220	-2.464821
26	1	0	-0.061188	3.788473	-2.806539
27	1	0	1.162526	2.821389	-2.667856
28	7	0	-3.888187	1.699310	-2.007016
29	6	0	1.846999	-2.185392	0.123316
30	1	0	1.254480	-2.892238	0.246090
31	6	0	2.264809	0.081109	-0.423461
32	1	0	1.959168	0.921524	-0.681292
33	6	0	0.281681	4.056775	1.146377
34	1	0	0.529621	4.816097	1.623452
35	6	0	0.369771	4.042356	-0.218763
36	1	0	0.657012	4.809716	-0.659400
37	6	0	-3.514841	-3.104150	0.537028
38	1	0	-2.633488	-3.362134	0.851487
39	1	0	-3.709186	-3.622538	-0.259374
40	6	0	-4.824124	-1.120634	-0.102412

41	1	0	-5.144607	-1.487798	-0.941311
42	1	0	-4.751574	-0.159730	-0.206072
43	6	0	-3.175799	0.962248	-1.506538
44	6	0	-5.895699	-2.921309	1.222377
45	1	0	-6.521894	-3.107167	1.940500
46	1	0	-6.209394	-3.369725	0.421725
47	6	0	3.605677	-0.117411	-0.246119
48	1	0	4.202059	0.583644	-0.375437
49	6	0	3.203695	-2.398113	0.292995
50	1	0	3.527717	-3.239284	0.520522
51	6	0	4.062893	-1.341289	0.119555
52	35	0	5.914989	-1.646274	0.344558
53	8	0	-0.313635	2.845834	3.166846
54	6	0	0.101964	3.943365	3.959297
55	1	0	-0.049233	3.744372	4.886165
56	1	0	-0.400222	4.723805	3.714510
57	1	0	1.037528	4.106157	3.814615

Optimized XYZ coordinates for **17a** (Ground State)

Energy = -1776.99180275

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	16	0	0.420761	3.229684	1.535300
2	8	0	-2.373641	-1.750108	3.228423
3	8	0	5.954064	-0.631614	0.655474
4	8	0	-0.079032	0.406382	-3.239462
5	7	0	1.397101	-3.556485	-1.275348
6	7	0	3.181190	-0.267354	0.166328
7	6	0	-0.377115	-0.442374	-0.985104
8	6	0	0.993903	-1.086549	-0.709265
9	6	0	1.862602	-0.134242	-0.230108
10	6	0	1.132929	1.134306	-0.093797
11	6	0	1.674816	2.408245	0.479764
12	6	0	-0.996197	3.112633	0.462350
13	6	0	-1.987231	4.086809	0.583328
14	6	0	-3.152254	4.005569	-0.172608
15	6	0	-3.326270	2.953278	-1.068443
16	6	0	-2.343874	1.979132	-1.188834
17	6	0	-1.165828	2.022780	-0.426515
18	6	0	-0.149596	0.979979	-0.501382
19	6	0	-0.524333	-0.474545	-2.546705
20	6	0	-1.246108	-1.680000	-3.101168
21	6	0	-2.632418	-1.812255	-2.453543
22	6	0	-2.559237	-1.809479	-0.942695
23	6	0	-3.547267	-2.451625	-0.193923
24	6	0	-3.537904	-2.460316	1.196337
25	6	0	-2.501378	-1.809420	1.872278
26	6	0	-1.503580	-1.167860	1.139607
27	6	0	-1.521699	-1.159736	-0.253702
28	6	0	3.725257	-1.601123	0.453863
29	6	0	4.983852	-1.445350	1.303168
30	6	0	5.439881	0.664760	0.392430
31	6	0	4.188085	0.597784	-0.476225
32	6	0	1.245003	-2.437932	-1.006570
33	6	0	-3.358248	-2.387437	4.029446
34	1	0	2.515556	2.211975	1.145256
35	1	0	1.998471	3.103927	-0.300998
36	1	0	-1.838238	4.915554	1.265871
37	1	0	-3.913280	4.771362	-0.071126
38	1	0	-4.222887	2.892201	-1.673813
39	1	0	-2.485877	1.175626	-1.898260
40	1	0	-0.656118	-2.574974	-2.876354
41	1	0	-1.307765	-1.565972	-4.184361
42	1	0	-3.110300	-2.732646	-2.799796

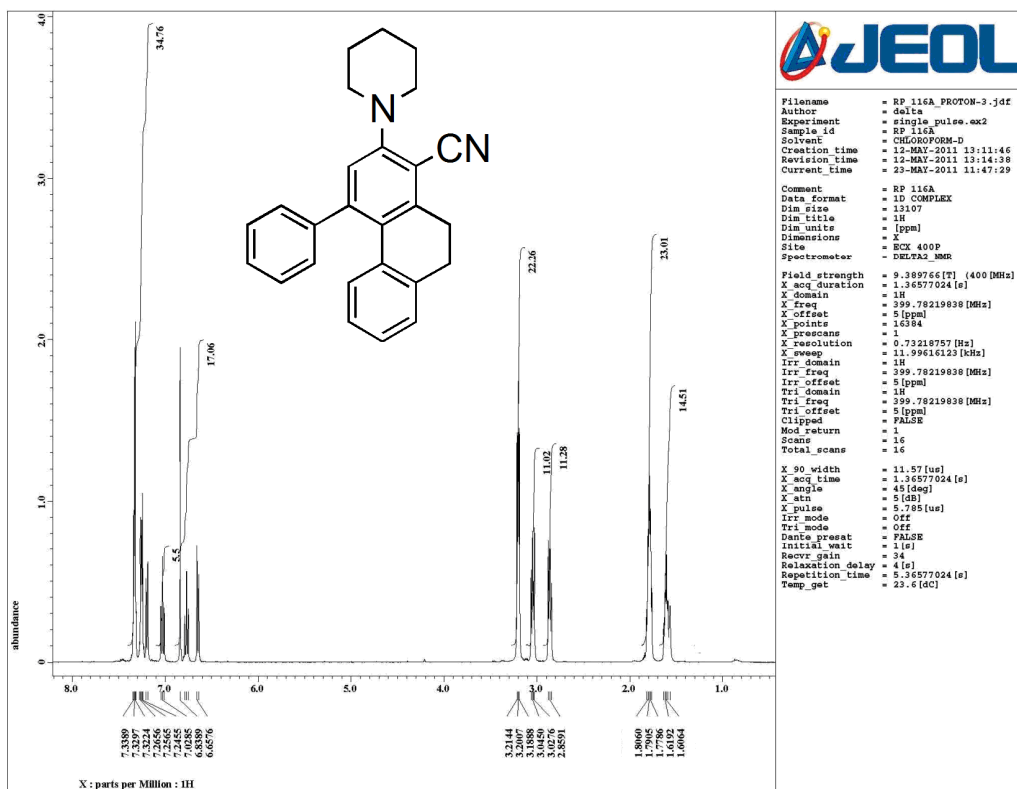
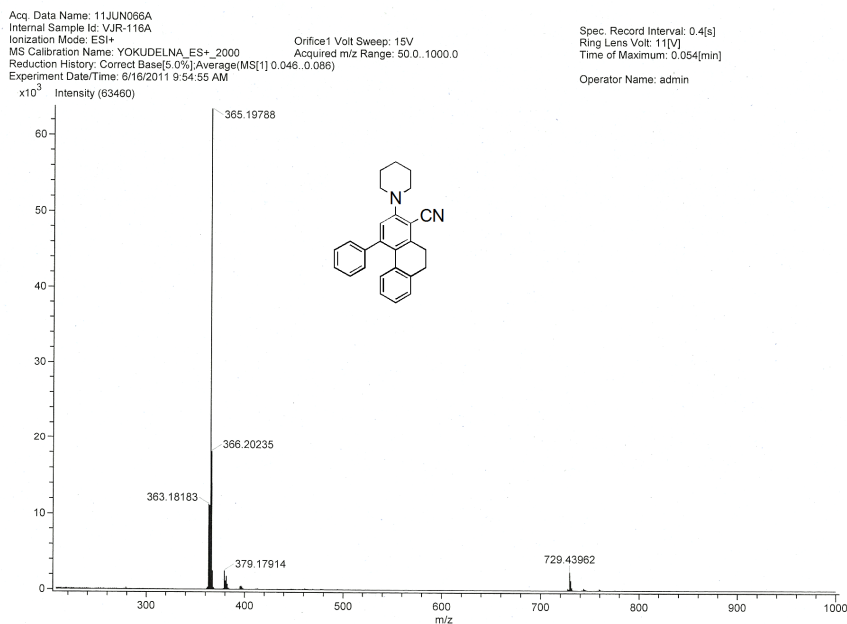
43	1	0	-3.272026	-0.990142	-2.801272
44	1	0	-4.349363	-2.965064	-0.714729
45	1	0	-4.323792	-2.974855	1.732251
46	1	0	-0.709685	-0.672493	1.685688
47	1	0	2.980384	-2.179656	1.001608
48	1	0	3.970428	-2.142983	-0.468967
49	1	0	4.719991	-1.011232	2.279099
50	1	0	5.452168	-2.417368	1.464323
51	1	0	5.208924	1.178958	1.338136
52	1	0	6.229385	1.214809	-0.122616
53	1	0	4.449685	0.186761	-1.462585
54	1	0	3.783514	1.596869	-0.634140
55	1	0	-4.352646	-1.957835	3.863318
56	1	0	-3.392493	-3.466725	3.842728
57	1	0	-3.060130	-2.211627	5.061701

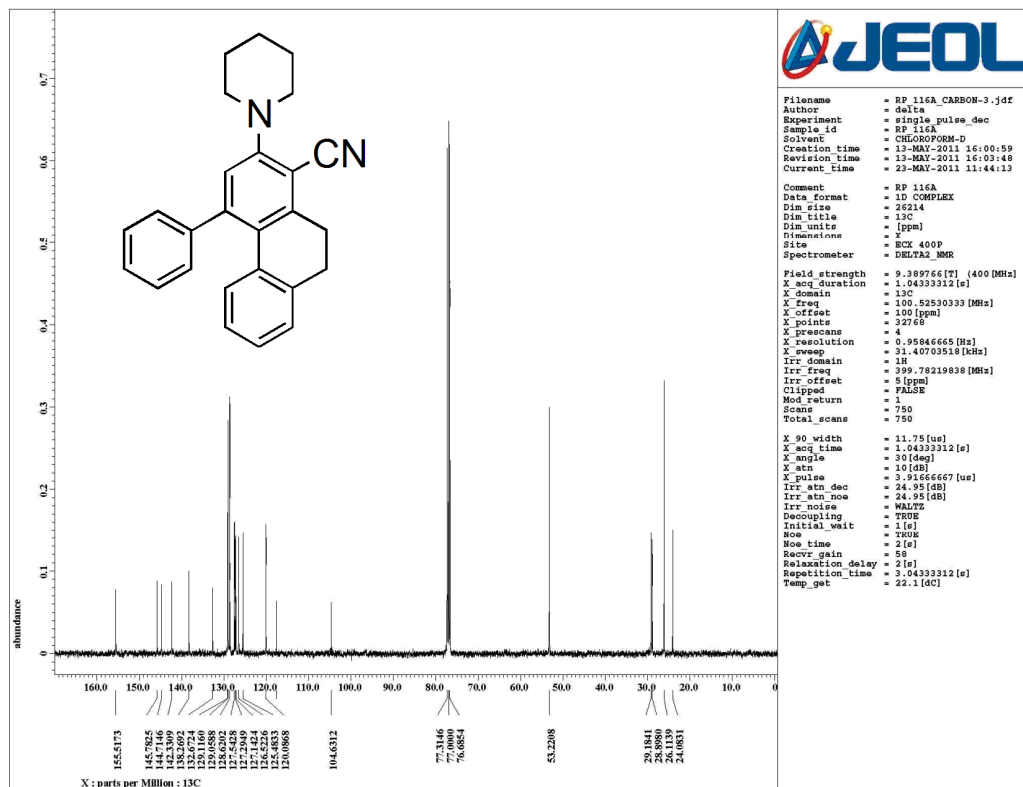
Optimized XYZ coordinates for 17a (Transition State)
Energy = -1776.98734748

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	16	0	0.358903	3.437059	1.076892
2	8	0	-3.132007	-0.744661	3.055672
3	8	0	5.949058	-0.431255	0.990702
4	8	0	-0.156944	0.047707	-3.229237
5	7	0	1.523984	-3.675433	-0.679331
6	7	0	3.193525	-0.182688	0.351420
7	6	0	-0.313473	-0.585970	-0.898078
8	6	0	1.057995	-1.159683	-0.490266
9	6	0	1.891221	-0.132158	-0.114279
10	6	0	1.140530	1.128364	-0.190383
11	6	0	1.652356	2.481875	0.197737
12	6	0	-1.023146	3.136001	-0.006076
13	6	0	-2.028399	4.101974	-0.056985
14	6	0	-3.174659	3.887089	-0.814886
15	6	0	-3.316583	2.704621	-1.537454
16	6	0	-2.319863	1.739427	-1.488032
17	6	0	-1.156283	1.920772	-0.722256
18	6	0	-0.125655	0.893869	-0.612289
19	6	0	-0.424986	-0.824322	-2.438351
20	6	0	-0.820323	-2.211577	-2.899018
21	6	0	-1.744824	-3.027891	-1.971388
22	6	0	-2.128115	-2.366796	-0.662130
23	6	0	-3.161565	-2.931065	0.090096
24	6	0	-3.540794	-2.432536	1.330825
25	6	0	-2.864590	-1.325399	1.850893
26	6	0	-1.839737	-0.739242	1.109216
27	6	0	-1.469880	-1.243283	-0.137957
28	6	0	3.736400	-1.448766	0.860760
29	6	0	4.961029	-1.148324	1.720747
30	6	0	5.435863	0.803043	0.514380
31	6	0	4.217725	0.588516	-0.377813
32	6	0	1.342518	-2.533684	-0.576900
33	6	0	-4.168268	-1.294602	3.855530
34	1	0	2.474221	2.400654	0.909165
35	1	0	1.993447	3.054111	-0.670780
36	1	0	-1.905406	5.027315	0.493909
37	1	0	-3.946857	4.647113	-0.848347
38	1	0	-4.200483	2.534497	-2.140654
39	1	0	-2.441557	0.830653	-2.059754
40	1	0	0.124212	-2.747621	-3.043636
41	1	0	-1.251856	-2.085972	-3.893305
42	1	0	-1.266264	-3.984612	-1.743218
43	1	0	-2.661743	-3.271171	-2.515597

44	1	0	-3.684850	-3.795644	-0.306515
45	1	0	-4.348487	-2.905453	1.872627
46	1	0	-1.341498	0.123626	1.532006
47	1	0	2.975393	-1.944765	1.464646
48	1	0	4.020378	-2.123913	0.042956
49	1	0	4.657701	-0.573409	2.608345
50	1	0	5.429845	-2.078117	2.045817
51	1	0	5.167620	1.453154	1.361444
52	1	0	6.239796	1.277520	-0.051025
53	1	0	4.520013	0.036319	-1.280210
54	1	0	3.811593	1.546734	-0.699859
55	1	0	-5.138885	-1.242182	3.349177
56	1	0	-3.957383	-2.334679	4.129201
57	1	0	-4.201777	-0.686103	4.757748

**Copy of HRMS and HNMRs:
 4-phenyl-2-(piperidin-1-yl)-9,10-dihydrophenanthrene-1-carbonitrile (10 a):**



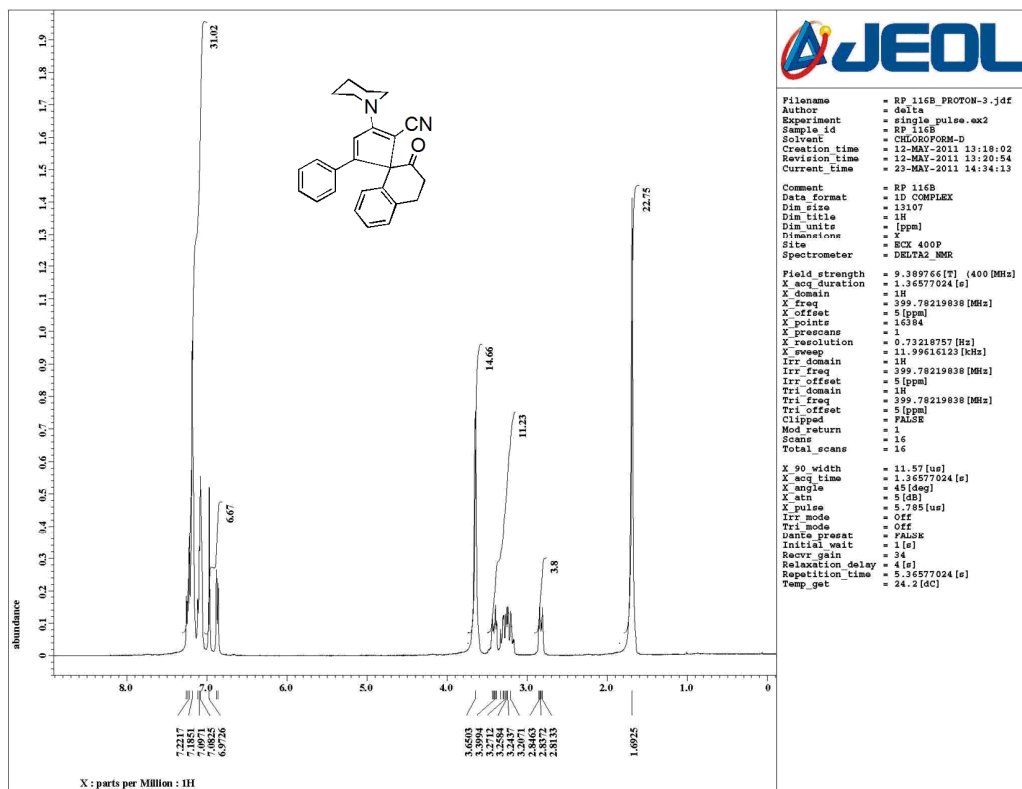
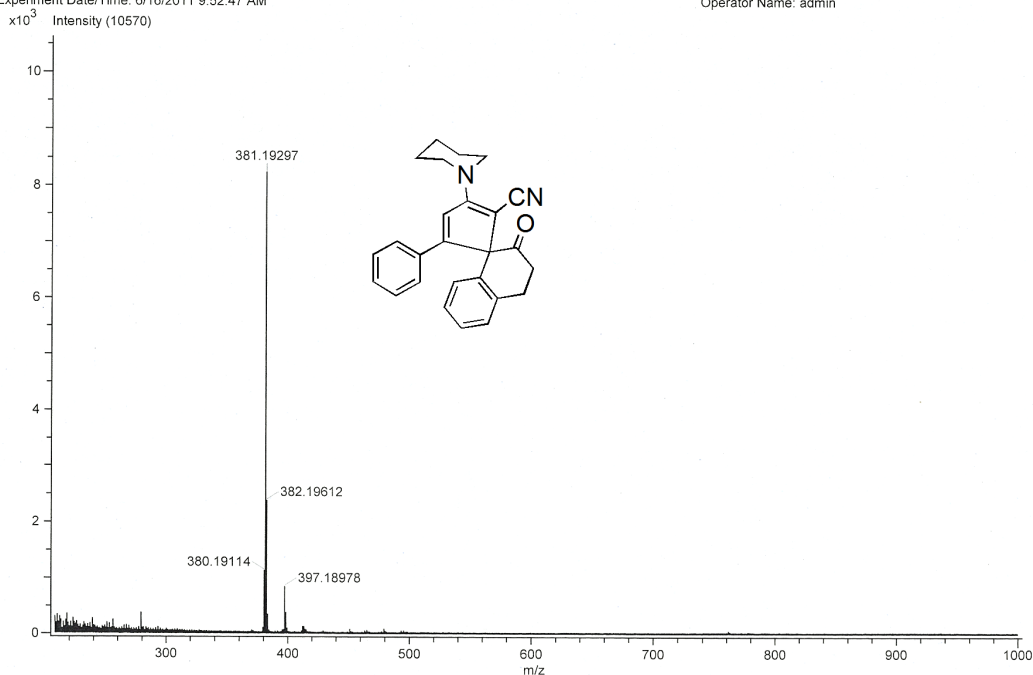


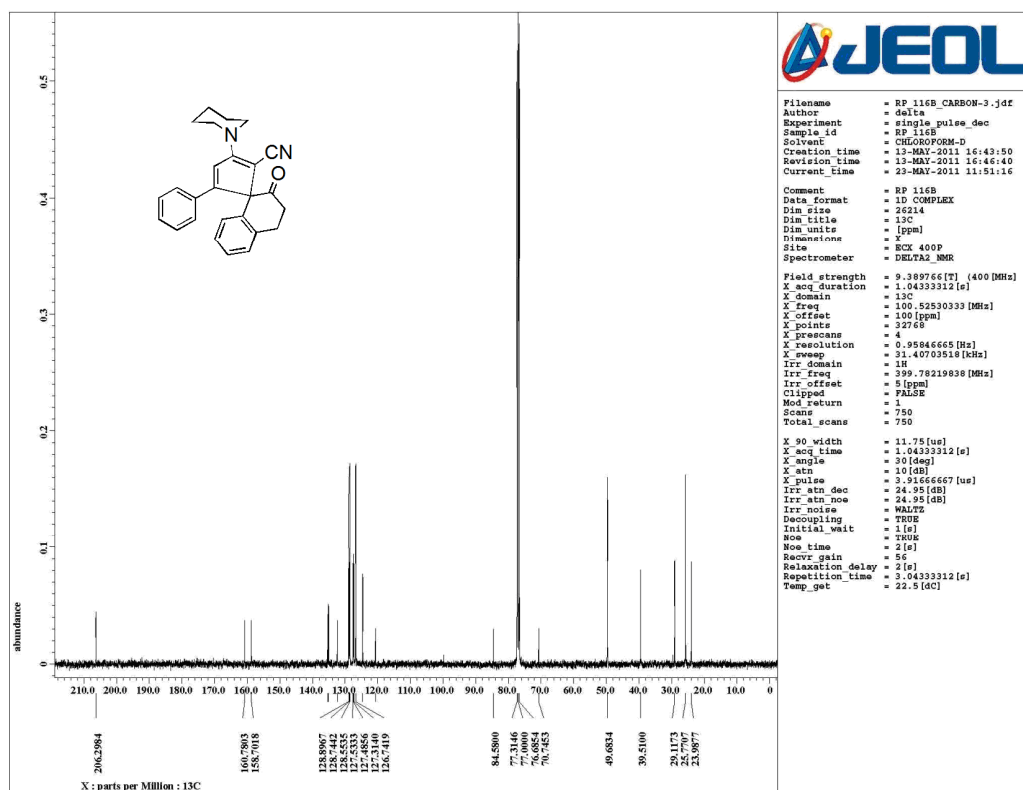
2'-oxo-5-phenyl-3-(piperidin-1-yl)-3',4'-dihydro-2'H-spiro[cyclopenta[2,4]diene-1,1'-naphthalene]-2-carbonitrile (11a):

Acq. Data Name: 11JUN064A
 Internal Sample Id: VJR-116B
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Correct Base[5.0%].Average[MS[1] 0.066..0.103]
 Experiment Date/Time: 6/16/2011 9:52:47 AM

Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.074[min]

Operator Name: admin





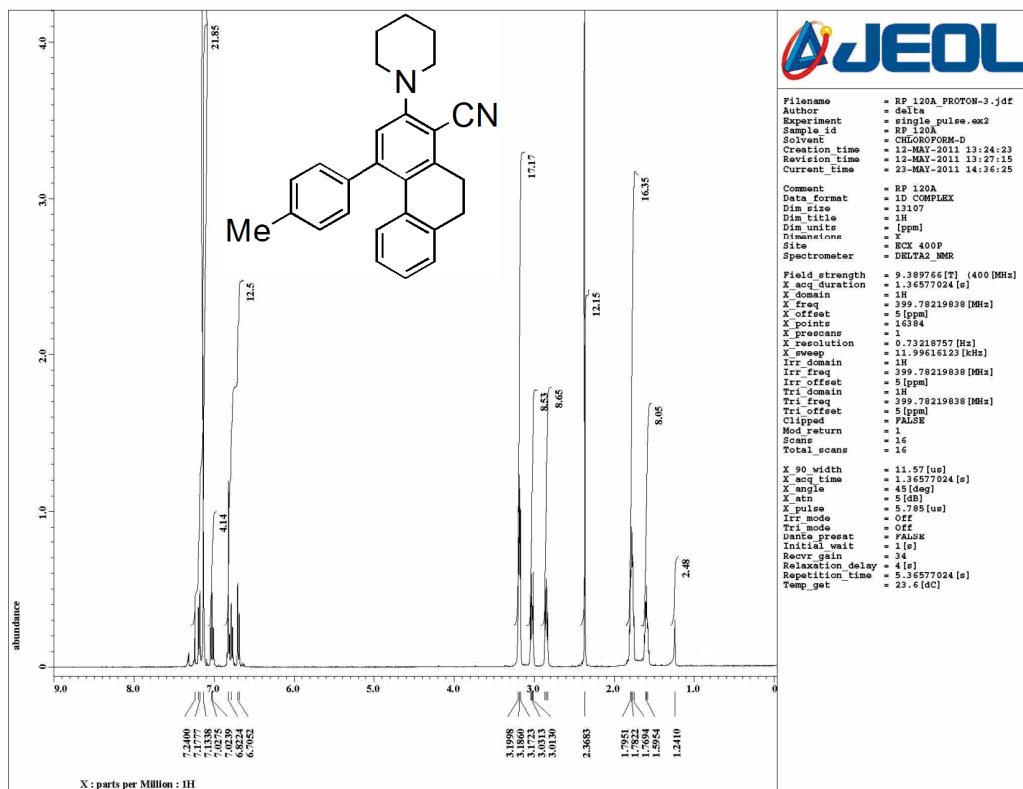
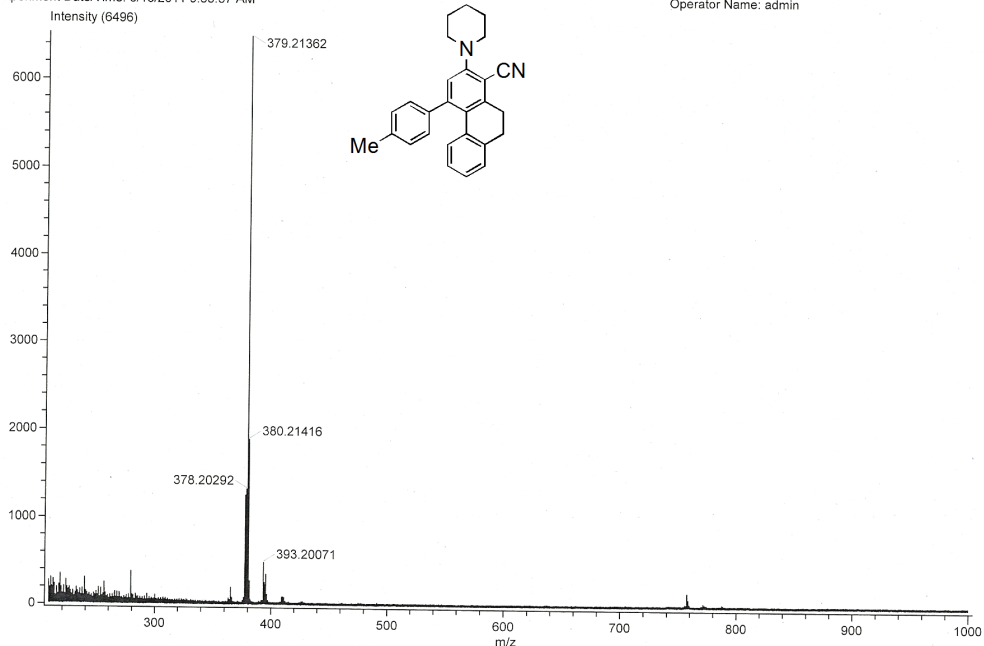
2-(piperidin-1-yl)-4-p-tolyl-9,10-dihydrophenanthrene-1-carbonitrile (10b):

Acq. Data Name: 11JUN065A
 Internal Sample Id: VJR-120A
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+ 2000
 Reduction History: Correct Base[5.0%];Average(MS[1]) 0.068.0.110
 Experiment Date/Time: 6/16/2011 9:53:57 AM

Orifice1 Volt Sweep: 15V
 Acquired m/z Range: 50.0..1000.0

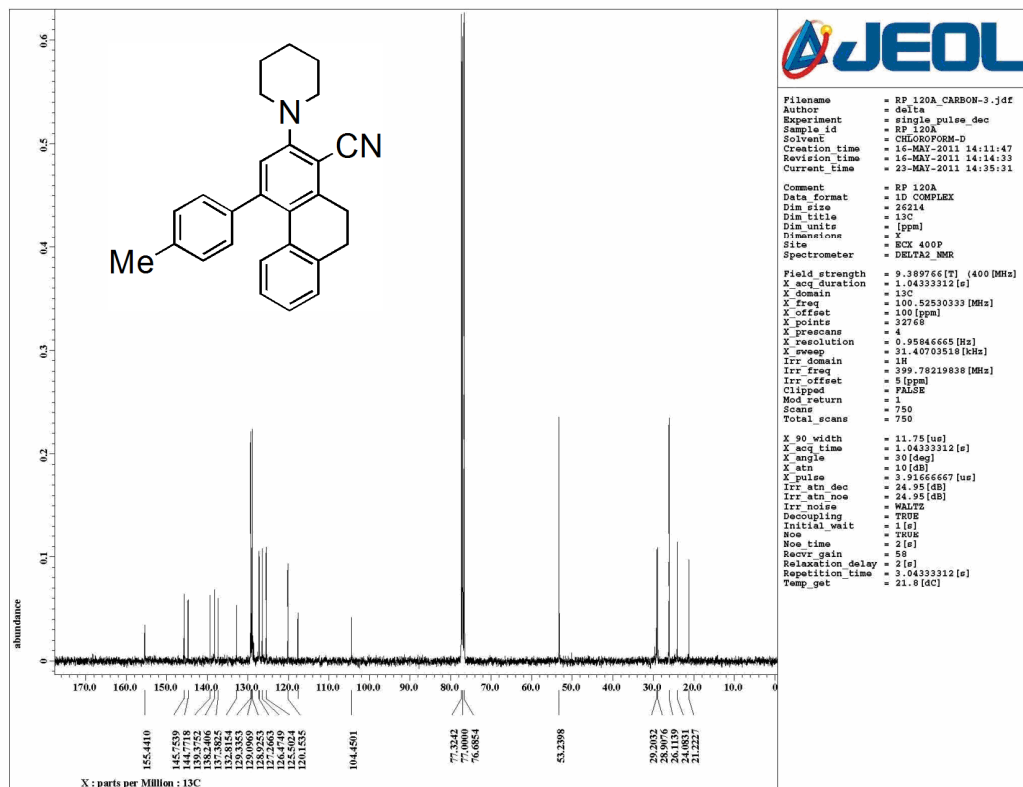
Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.076[min]

Operator Name: admin



Filename = RP_120A_PROTON-3_jdf
 Author = delta
 Experiment = single pulse.ex2
 Sample_id = RP_120A
 Solvent = CHLOROFORM-D
 Creation time = 12-MAY-2011 13:24:23
 Revision time = 12-MAY-2011 13:27:15
 Current time = 23-MAY-2011 14:26:25
 Comment = RP_120A
 Data format = 1D COMPLEX
 Dim size = 13107
 Dim title = 1H
 Dim units = [ppm]
 Dimension = X
 Size = KX 400P
 Spectrometer = DELTA2 MMR
 Field strength = 9.389766 [T] (400 [MHz])
 X_acq_duration = 1.36577024 [s]
 X_domain = 1H
 X_freq = 399.78219838 [MHz]
 X_offset = 5 [ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.73218757 [Hz]
 X_sweep = 11.99616123 [kHz]
 Irr_domain = 1H
 Irr_freq = 399.78219838 [MHz]
 Irr_offset = 5 [ppm]
 Tri_domain = 1H
 Tri_freq = 399.78219838 [MHz]
 Tri_offset = 5 [ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 16
 Total_scans = 16
 X_90_width = 11.57 [us]
 X_acq_time = 1.36577024 [s]
 X_angle = 45 [deg]
 X_atn = 5 [dB]
 X_pulse = 5.785 [us]
 Irr_mode = Off
 Tri_mode = Off
 Device preset = FALSW
 Initial wait = 1 [s]
 Recv_gain = 34
 Relaxation delay = 4 [s]
 Repetition_time = 5.36577024 [s]
 Temp_get = 23.6 [dC]

X : parts per Million : 1H



2'-oxo-3-(piperidin-1-yl)-5-p-tolyl-3',4'-dihydro-2'H-spiro[cyclopenta[2,4]diene-1,1'-naphthalene] (11b):

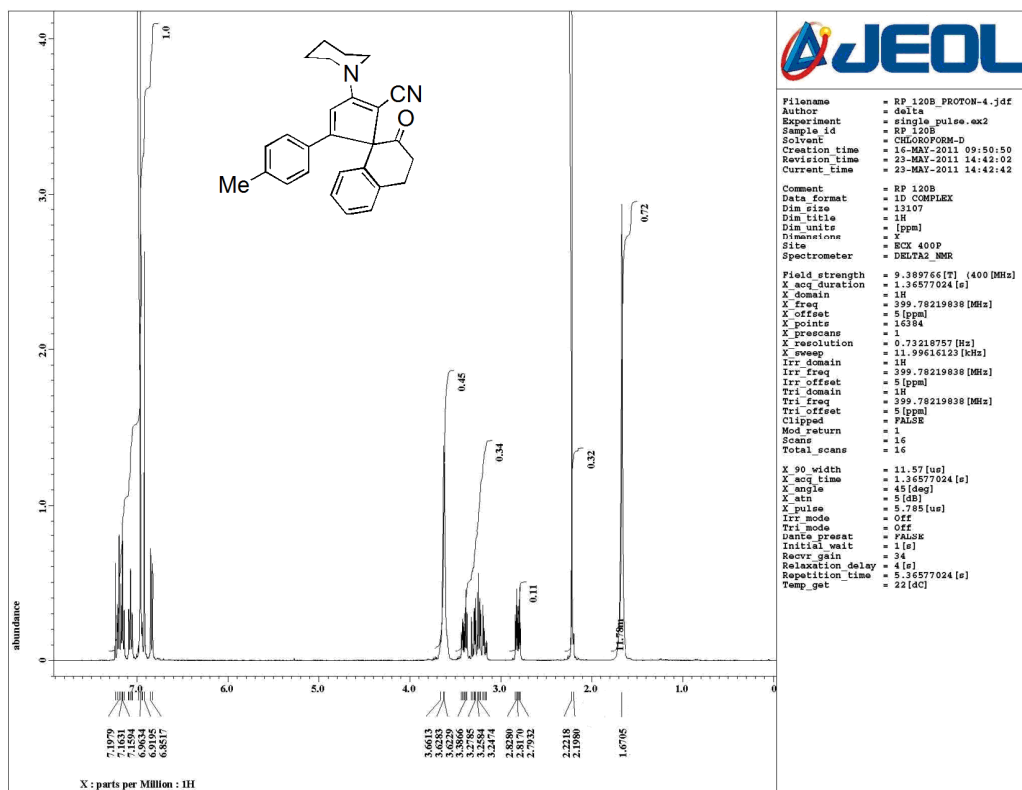
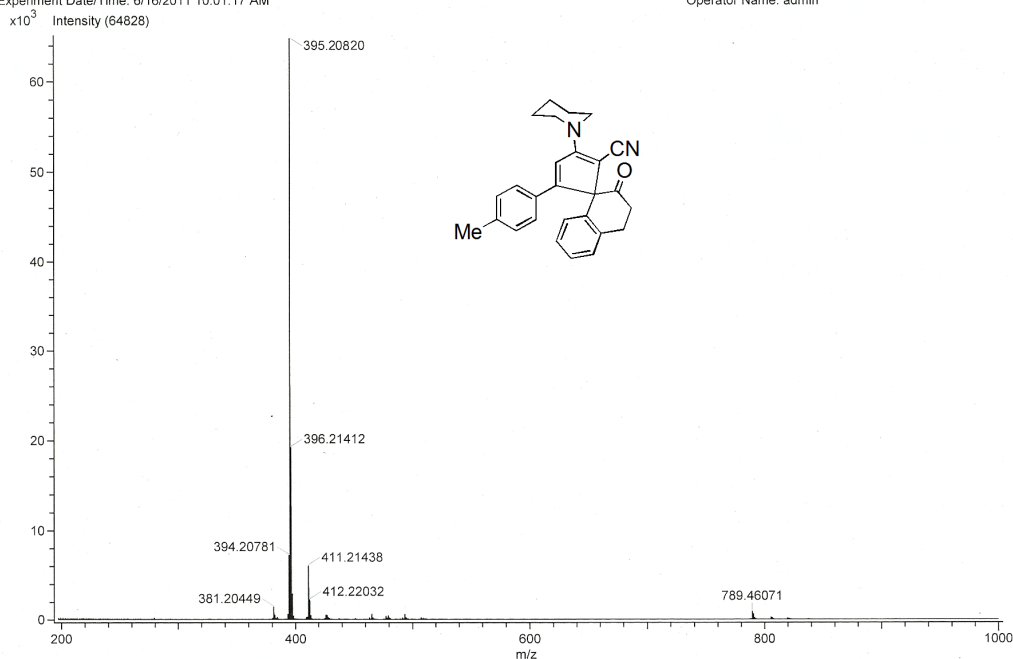
-2-carbonitrile

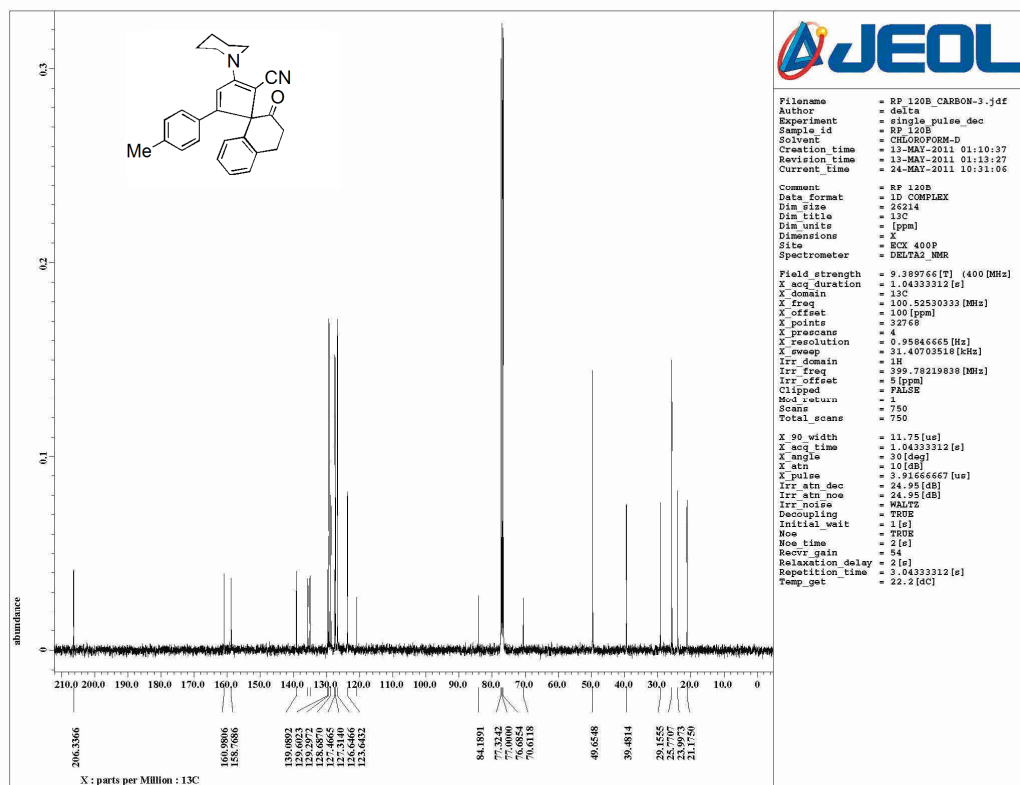
Acq. Data Name: 11JUN070A
 Internal Sample Id: VJR-120B
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Correct Base[5.0%];Average(MS[1] 0.054..0.076)
 Experiment Date/Time: 6/16/2011 10:01:17 AM

Orifice1 Volt Sweep: 15V
 Acquired m/z Range: 50.0..1000.0

Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.054[min]

Operator Name: admin





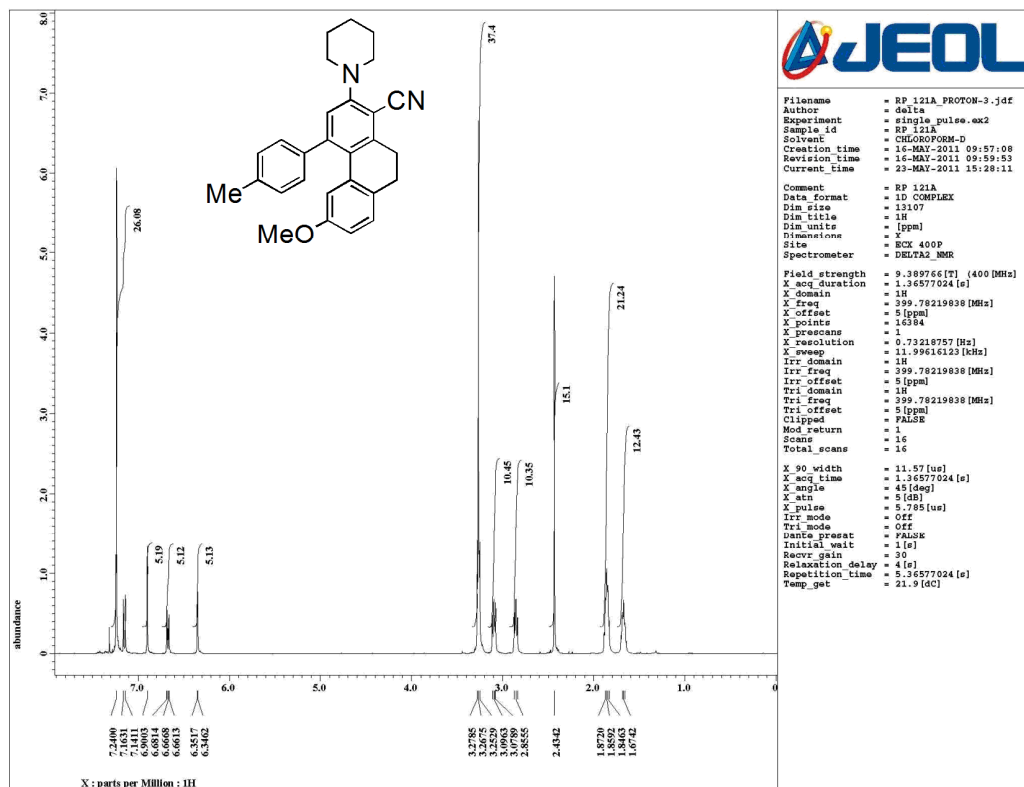
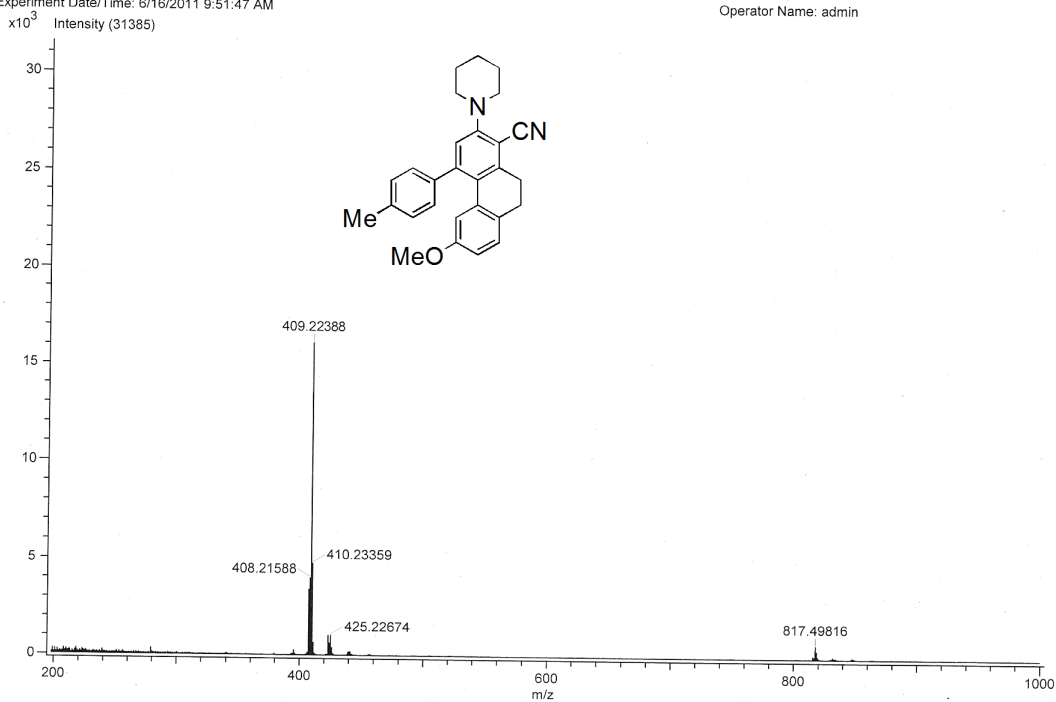
6-methoxy-2-(piperidin-1-yl)-4-p-tolyl-9,10-dihydrophenanthrene-1-carbonitrile (10c):

Acq. Data Name: 11JUN063A
 Internal Sample Id: VJR-121A
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Correct Base[5.0%];Average[MS[1] 0.066;.0.088]
 Experiment Date/Time: 6/16/2011 9:51:47 AM

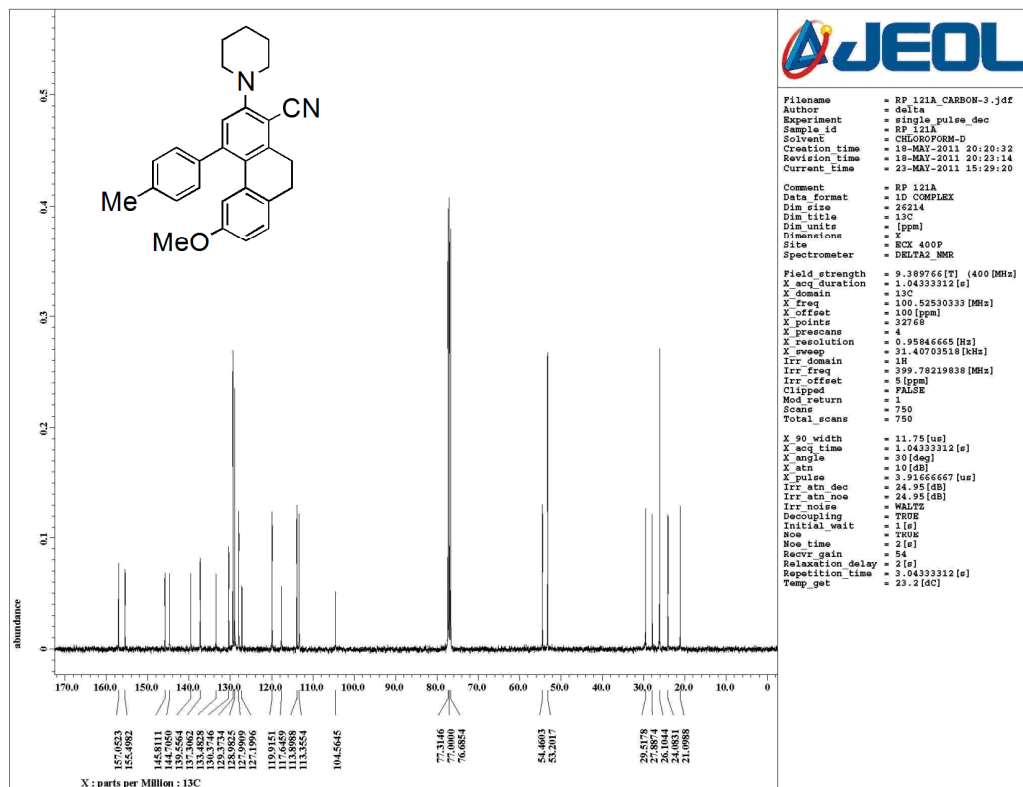
Orifice1 Volt Sweep: 15V
 Acquired m/z Range: 50.0..1000.0

Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.074[min]

Operator Name: admin



Filename = RP_121A_PROTON-3_jdr
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = RP_121A
 Solvent = CHLOROFORM-D
 Creation_time = 16-MAY-2011 09:57:08
 Revision_time = 16-MAY-2011 09:59:53
 Current_time = 23-MAY-2011 15:28:11
 Comment = RP_121A
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dir_title = 1H
 Dim_units = [ppm]
 Dimensions = 2
 Size = EXX_400P
 Spectrometer = DELTA2_MMR
 Field_strength = 9.389766[T] (400 [MHz])
 X_acq_duration = 1.36577024[s]
 X_domain = 1H
 X_freq = 399.78219838 [MHz]
 X_offset = 5 [ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.73218757 [Hz]
 X_r sweep = 11.99616123 [kHz]
 Irr_domain = 1H
 Irr_freq = 399.78219838 [MHz]
 Irr_offset = 5 [ppm]
 Tri_domain = 1H
 Tri_freq = 399.78219838 [MHz]
 Tri_offset = 5 [ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 16
 Total_scans = 16
 X_90_width = 11.57 [us]
 X_acq_time = 1.36577024[s]
 X_angle = 45 [deg]
 X_atn = 5 [dB]
 X_pulse = 5.785 [us]
 Irr_mode = Off
 Irr_presat = Off
 Data_presat = FALSE
 Initial_wait = 1 [s]
 Recov_gain = 30
 Relaxation_delay = 4 [s]
 Repetition_time = 5.36577024[s]
 Temp_set = 21.9 [degC]

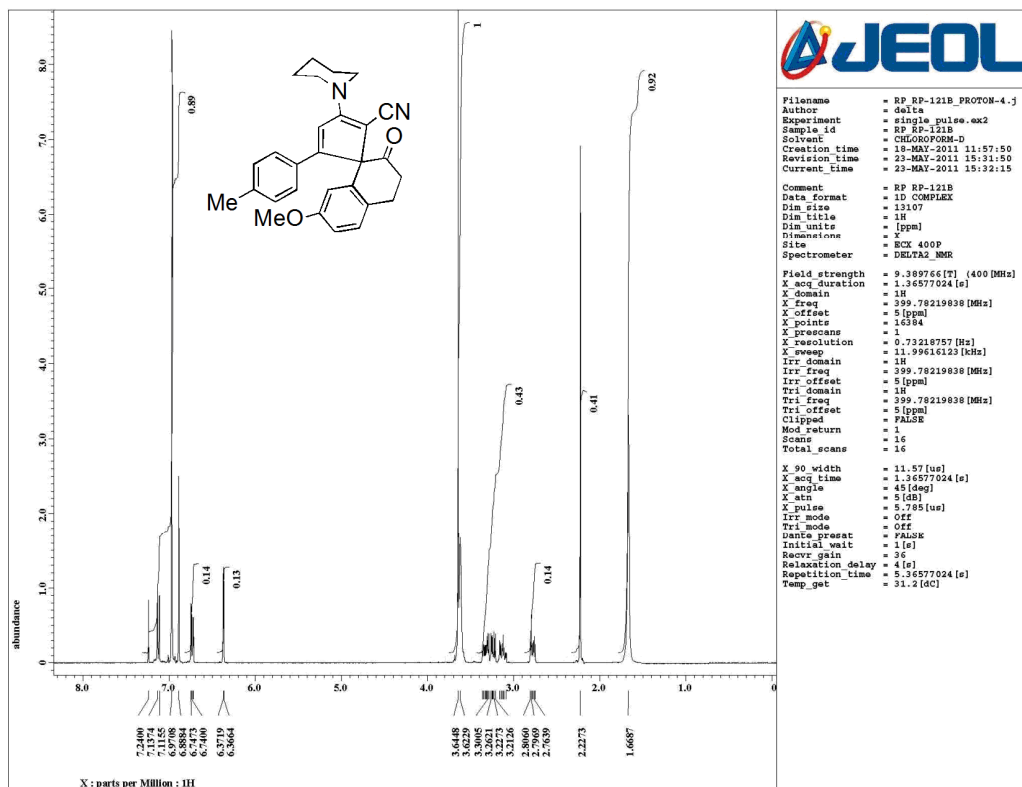
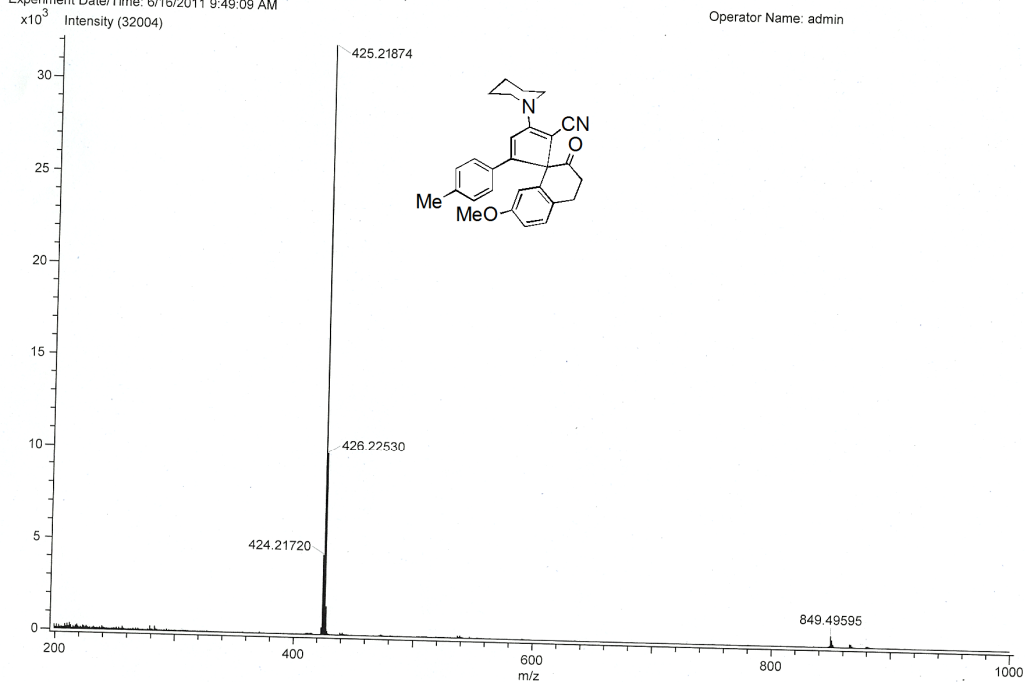


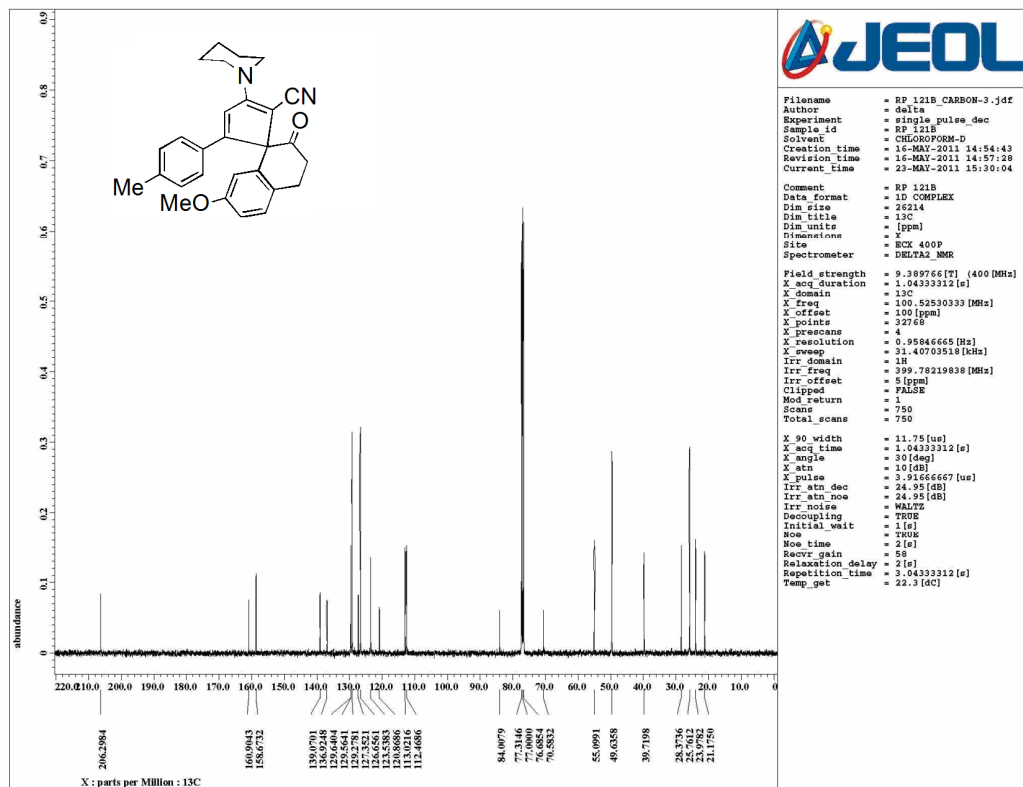
7'-methoxy-2'-oxo-3-(piperidin-1-yl)-5-p-tolyl-3',4'-dihydro-2'H-spiro[cyclopenta[2,4]diene-1,1'-naphthalene]-2-carbonitrile (11c):

Acq. Data Name: 11JUN062A
 Internal Sample Id: VJR-121B
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Correct Base[S 0%],Average(MS[1] 0.043..0.107)
 Experiment Date/Time: 6/16/2011 9:49:09 AM

Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.082[min]

Operator Name: admin





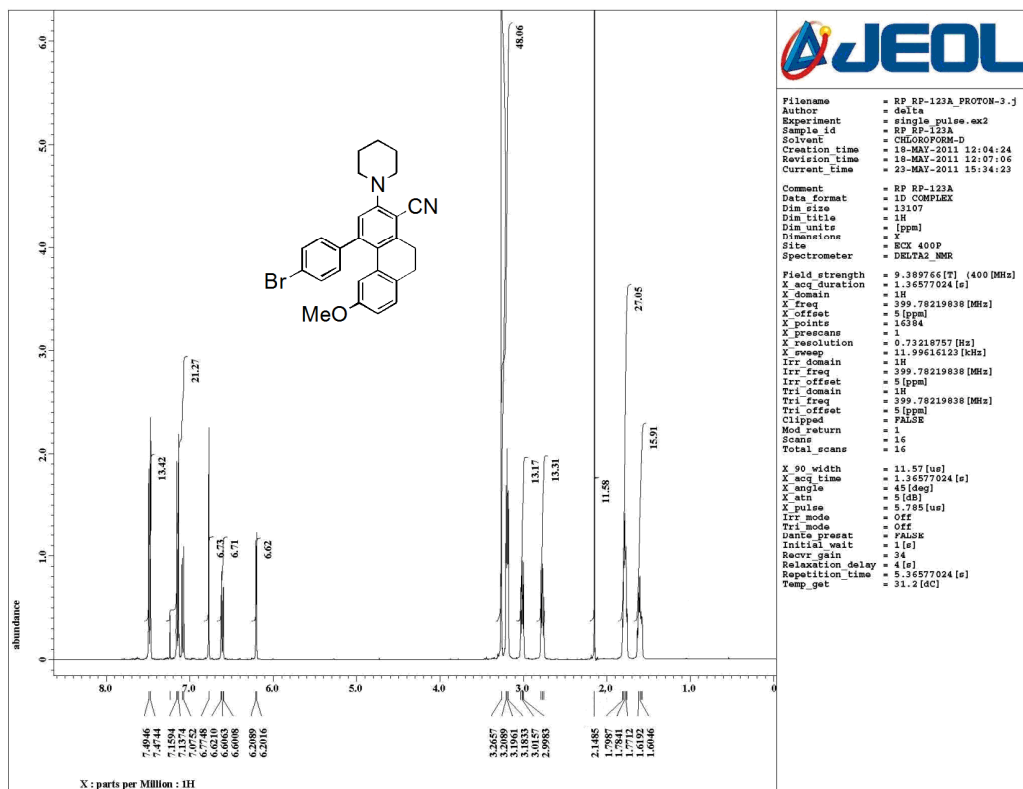
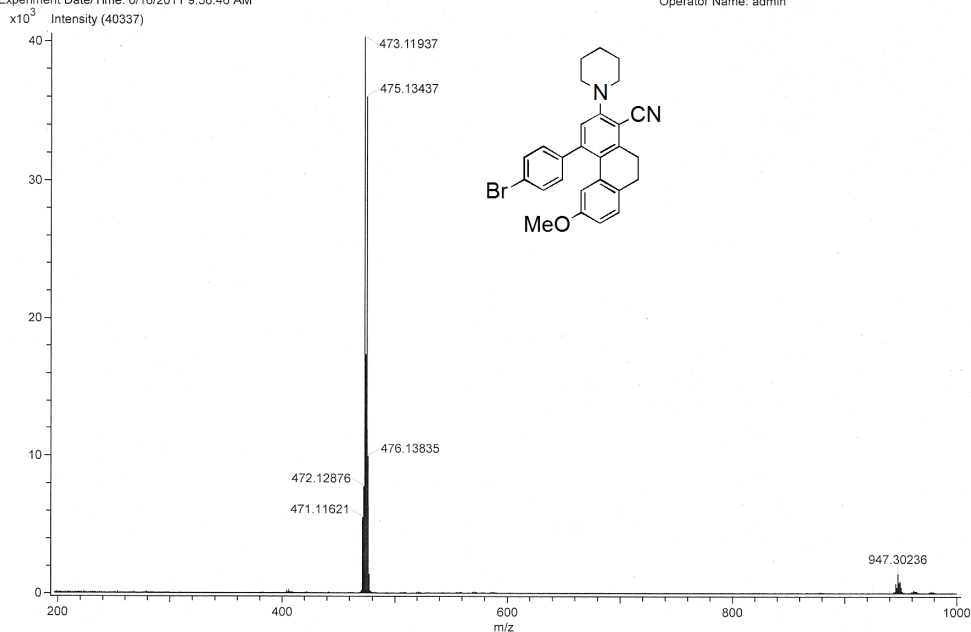
4-(4-bromophenyl)-6-methoxy-2-(piperidin-1-yl)-9,10-dihydrophenanthrene-1-carbonitrile (10d)

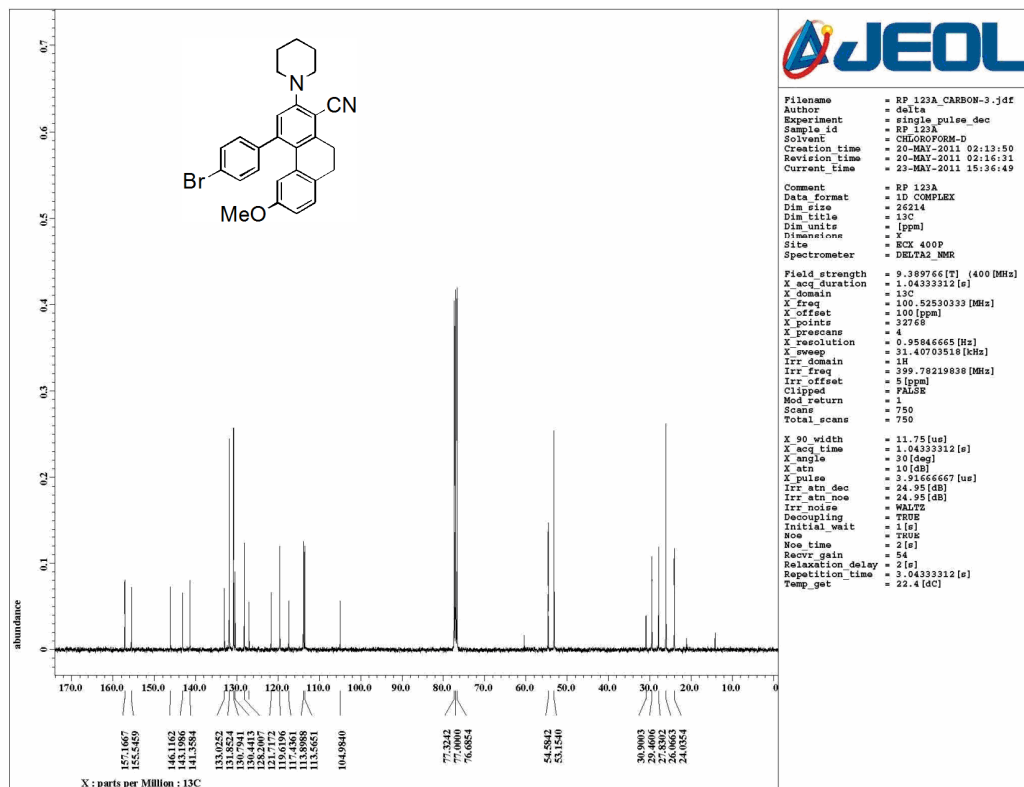
Acq. Data Name: 11JUN068A
 Internal Sample Id: VJR-123A
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Average(MS[1] 0.064, 0.071)
 Experiment Date/Time: 6/16/2011 9:56:46 AM

Orifice1 Volt Sweep: 15V
 Acquired m/z Range: 50.0..1000.0

Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.069[min]

Operator Name: admin





5-(4-bromophenyl)-7'-methoxy-2'-oxo-3-(piperidin-1-yl)-3',4'-dihydro-2'H-spiro[cyclopenta[2,4]naphthalene]-2-carbonitrile (11d):

diene-1,1'-

Acq. Data Name: 11JUN067A

Internal Sample Id: VJR-123B

Ionization Mode: ESI+

MS Calibration Name: YOKUDELNA_ES+_2000

Reduction History: Correct Base[5.0%]; Average[MS[1] 0.081..0.117]

Experiment Date/Time: 6/16/2011 9:55:51 AM

Orifice1 Volt Sweep: 15V

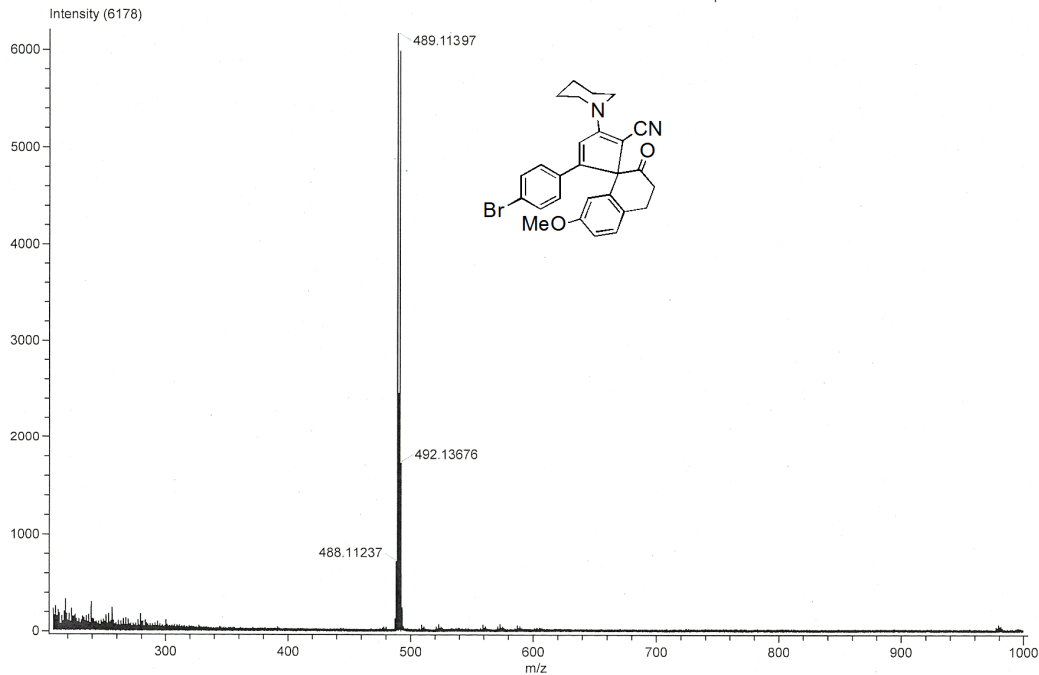
Acquired m/z Range: 50.0..1000.0

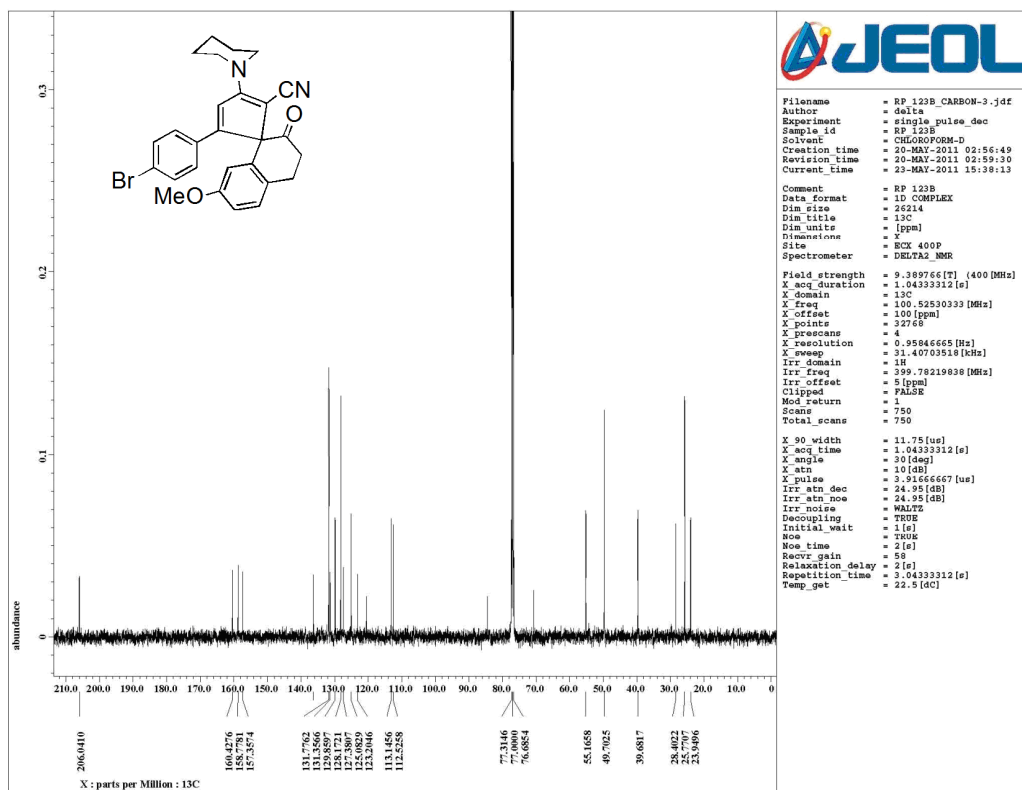
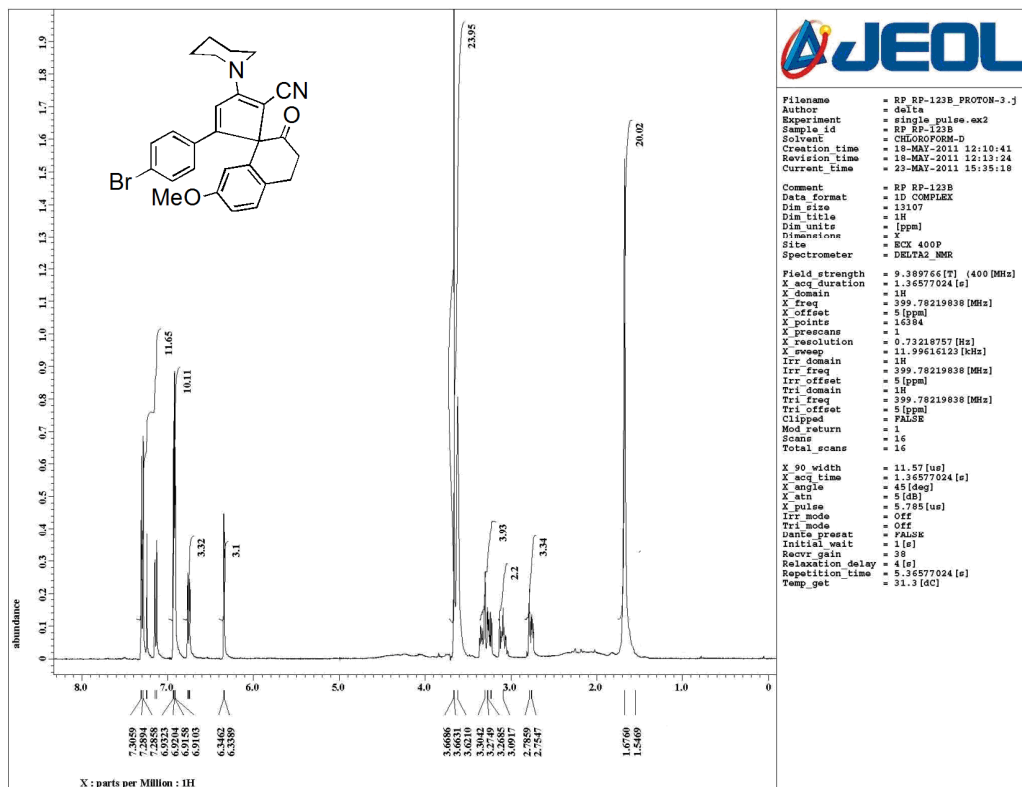
Spec. Record Interval: 0.4[s]

Ring Lens Volt: 11[V]

Time of Maximum: 0.088[min]

Operator Name: admin



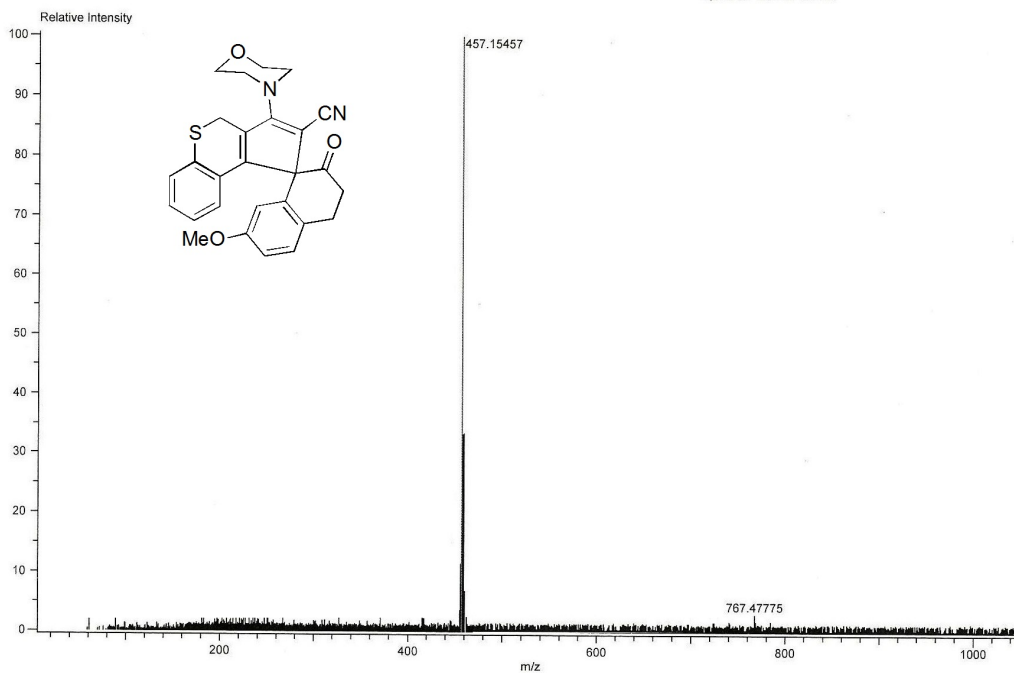


7'-methoxy-3-morpholino-2'-oxo-3',4'-dihydro-2'H,4H-spiro[cyclopenta[c]thiochromene-1,1'-naphthalene]-2-carbonitrile (17a):

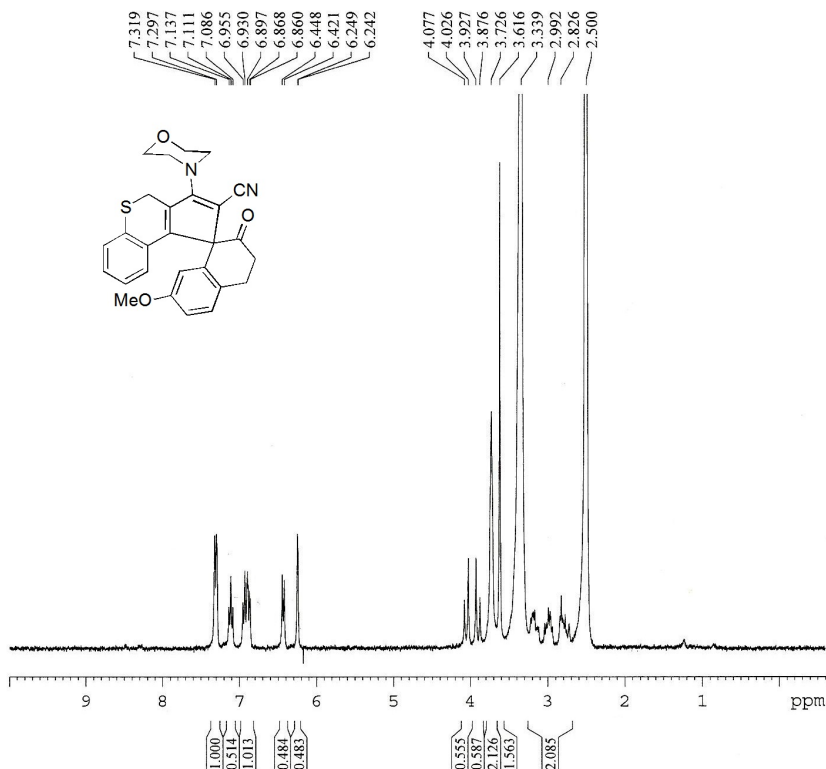
Acq. Data Name: 10FEB119A
 Internal Sample Id: VJR-77
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Determine m/z/Peak Detect[Centroid,50,Area];Correct Base[5.0%];Smooth[3];Add[Correct Base[5.0%];Average(MS[1] 0.302..0.312);,1.0];Correct Base[5.0%];...
 Experiment Date/Time: 2/11/2010 3:44:31 PM

Acq. Data Name(FullPath): E:\JAN10\FEB10\10FEB119A
 Ion Guide RF Volt: 550V
 Orifice1 Volt Sweep: 28V
 Acquired m/z Range: 10.0..1050.0

Sample Comments: DART
 Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 13[V]
 Time of Maximum: 0.109[min]
 Average(MS[1] 0.302..0.312);,1.0];Correct Base[5.0%];...
 Operator Name: admin



VJR-77



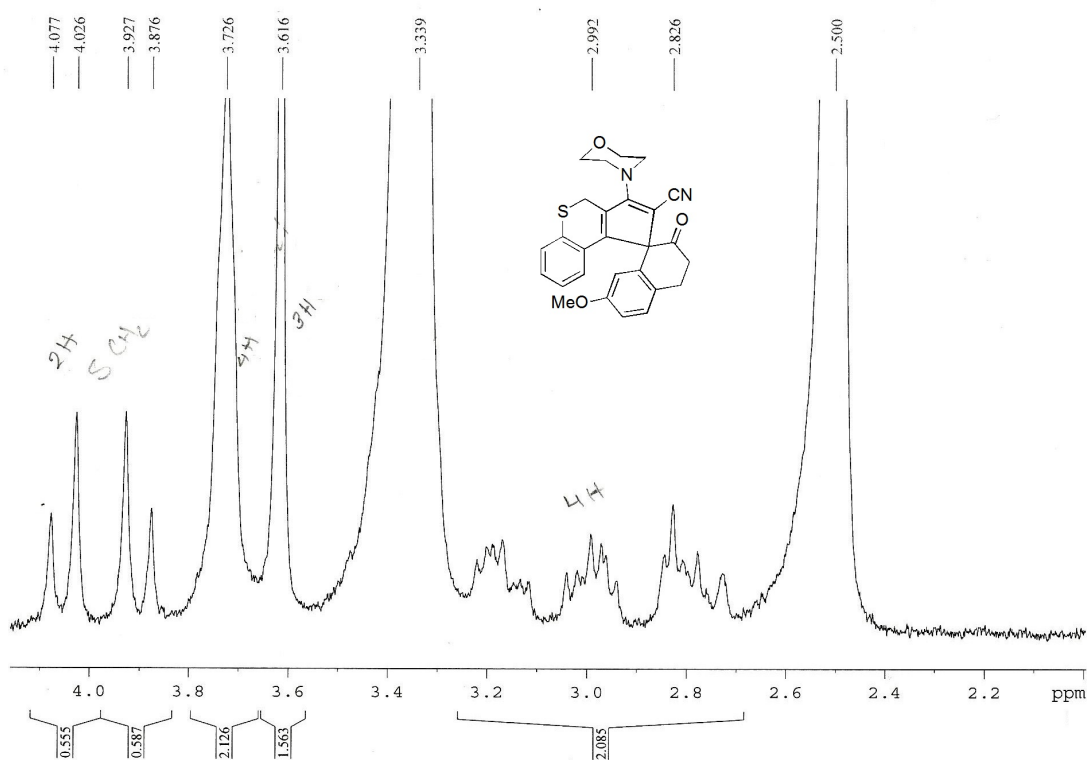
Current Data Parameters
 NAME vjr77.temp
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100212
 Time_ 16.16
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 32
 DS 2
 SWH 9578.544 Hz
 FIDRES 0.146157 Hz
 AQ 3.4220291 sec
 RG 1149.4
 DW 52.200 usec
 DE 6.00 usec
 TE 294.2 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

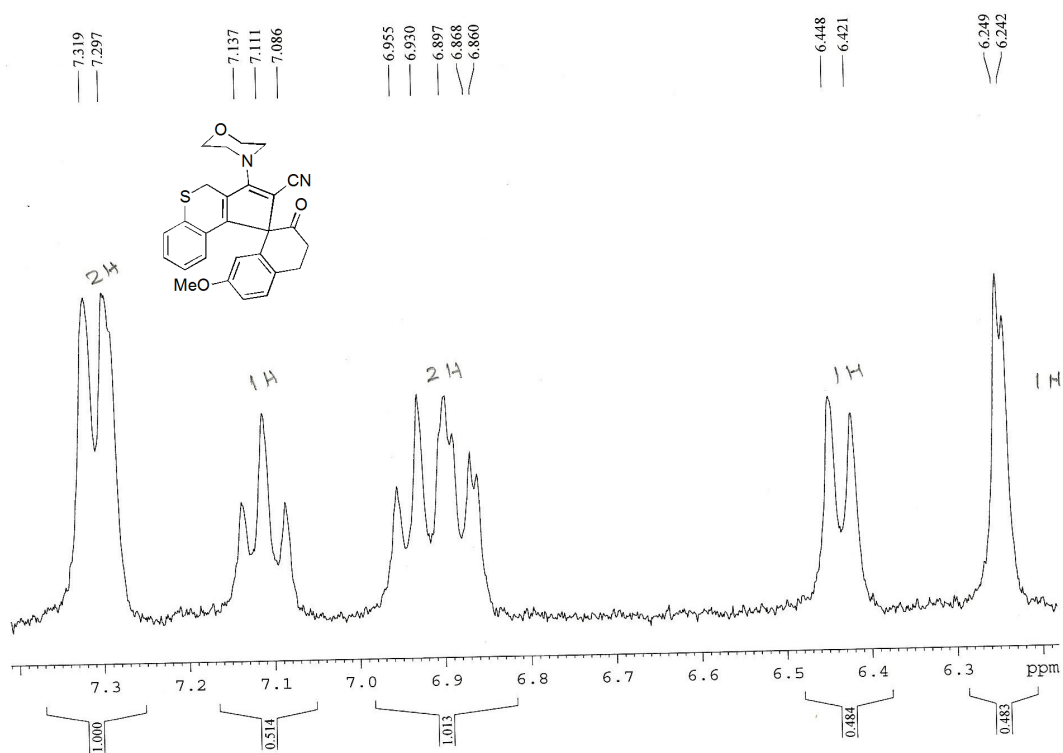
===== CHANNEL f1 =====
 NUC1 1H
 P1 6.88 usec
 PL1 -3.00 dB
 SFO1 300.1318534 MHz

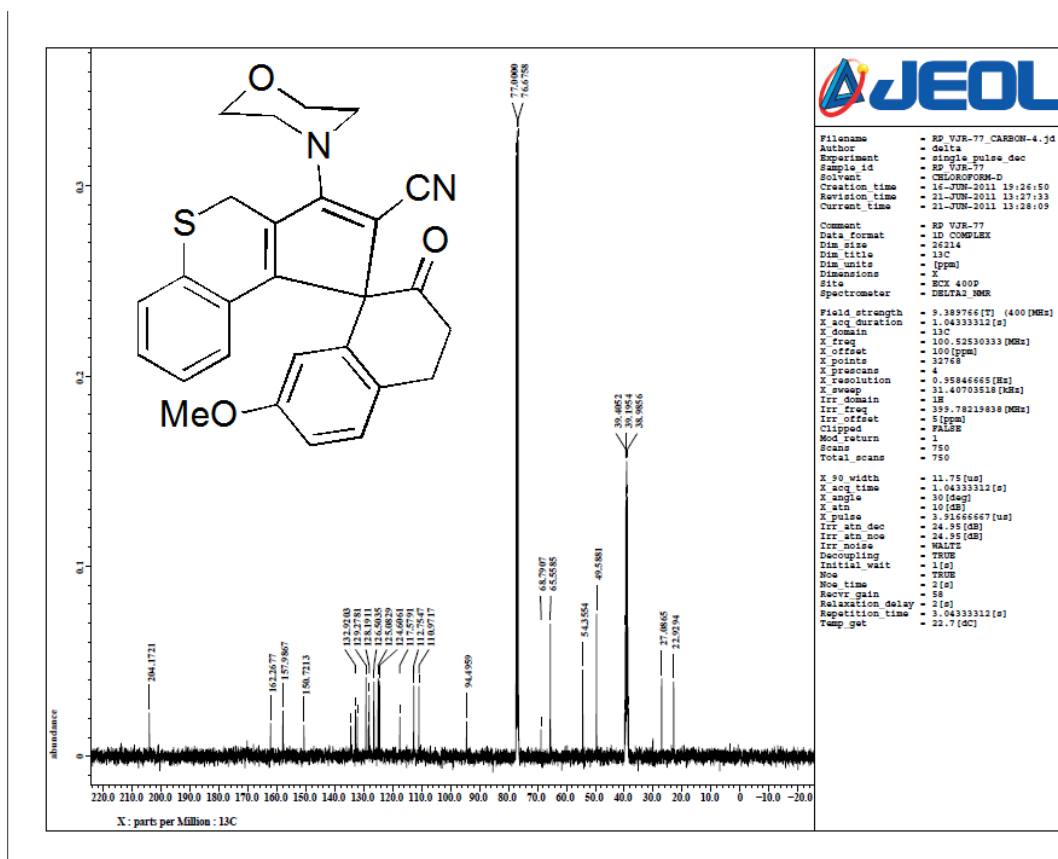
F2 - Processing parameters
 SI 32768
 SF 300.1300005 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

VJR-77



VJR-77

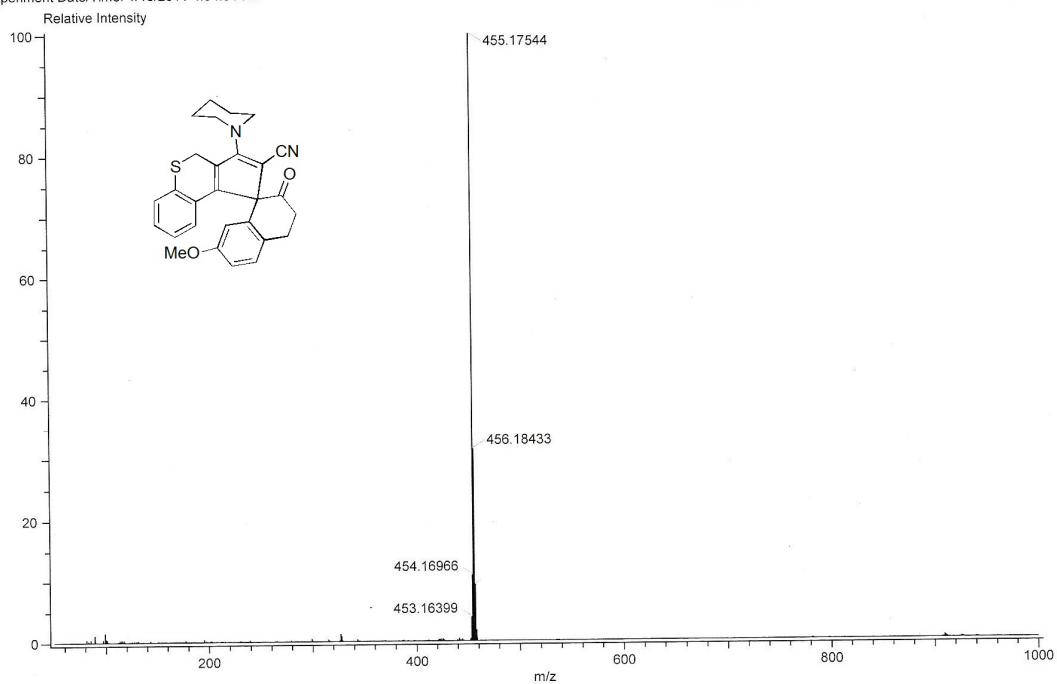




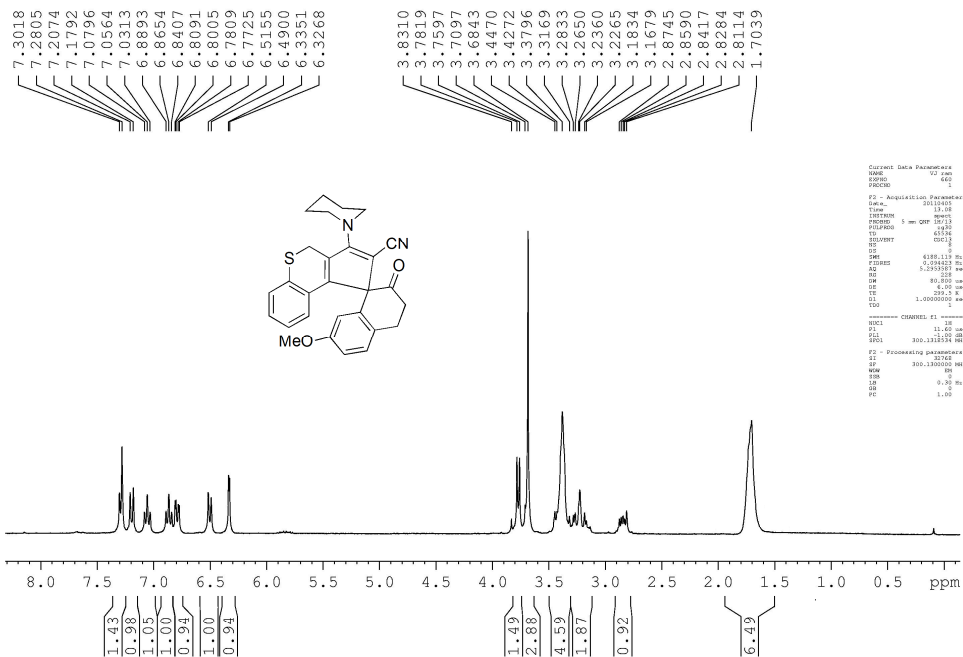
7'-methoxy-2'-oxo-3-(piperidin-1-yl)-3',4'-dihydro-2'H,4H-spiro[cyclopenta[c]thiochromene-1,1'-naphthalene]-2-carbonitrile (17b):

Acq. Data Name: 10JAN076A
 Internal Sample Id: VJR-106
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Correct Base[5.0%];Average(MS[1] 0.081, 0.086)
 Experiment Date/Time: 1/19/2011 4:54:51 PM

Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.094[min]
 Operator Name: admin

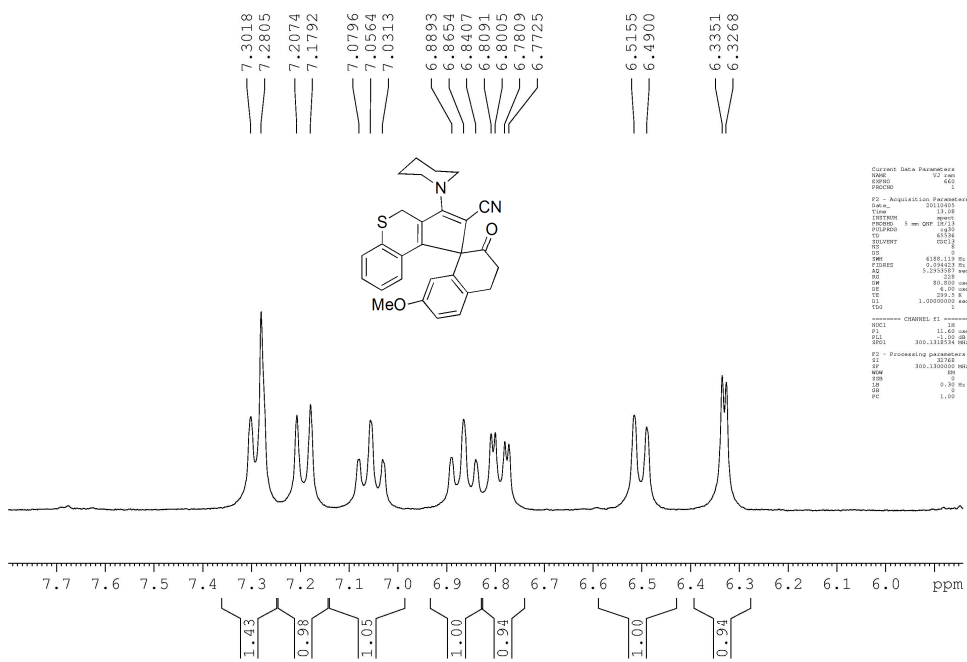


AM-106



Current Data Parameters
 NAME: VJR-106
 EXPNO: 602
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_ : 10/19/2011
 Time: 13.08
 INSTRUM: spect
 PULPROG: zgpg30
 POLPRG2: zgpg30
 VS: 400
 NS: 600
 DS: 4
 SWH: 8000.000 Hz
 FIDRES: 0.094327 Hz
 AQ: 0.25000000 sec
 RG: 80.000 used
 GB: 0.00000000 sec
 SE: 30.00 Hz
 SI: 1.00000000 sec
 SFO: 400.1313534 MHz
 ===== CHANNEL f1 =====
 NU1: 1
 PC1: 11.00 used
 PS: 11.00000000 sec
 SFO1: 300.1313534 MHz
 F2 - Processing parameters
 F1: 400.1313534 MHz
 F2: 300.1313534 MHz
 WOH: 80
 LB: 4.00 Hz
 GB: 0.00000000 sec
 SE: 1.00

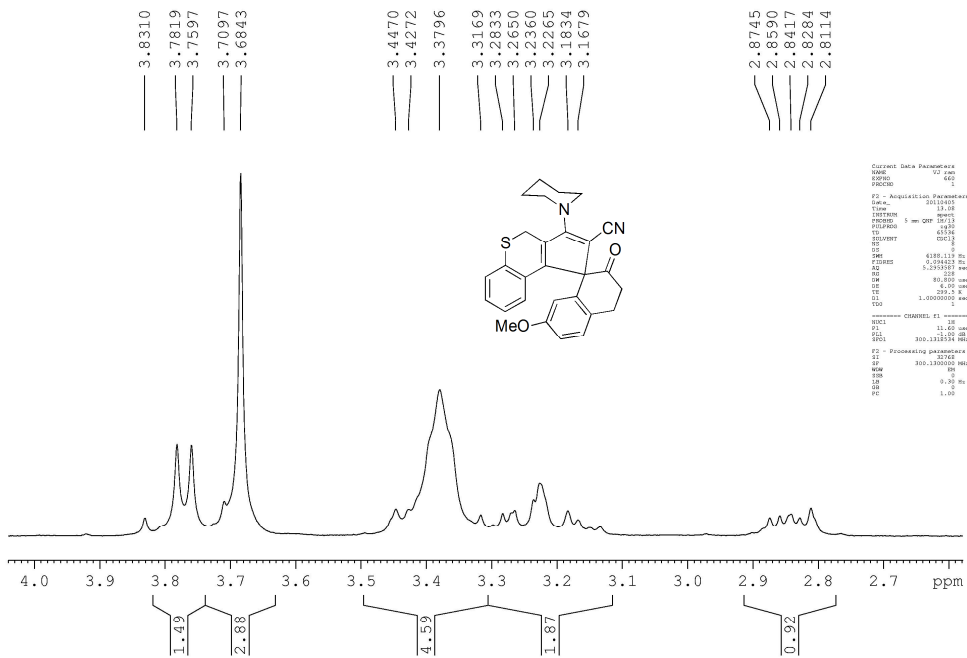
AM-106



```

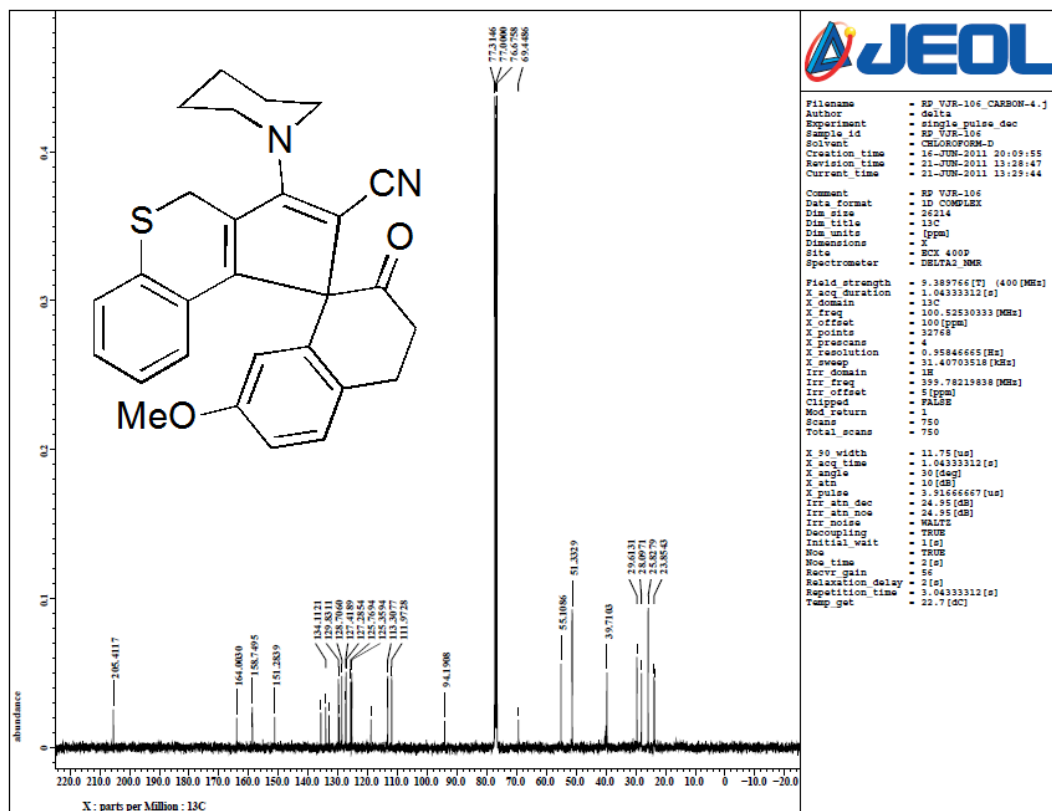
Current Data Parameters
Date_   12 Jan
Time_   16:02
PROCNO  1
F2 - Acquisition Parameters
Date_   20110805
Time_   11:05
INSTRUM spect
PROBHD  5 mm QNP 1H/1
PULPROG zgpg30
TD       65536
SOLVENT CDCl3
NS       8
DS       4
SWH      6188.115 Hz
FIDRES  0.29842 Hz
AQ       5.255087 sec
RG       327.5
SQ       89.810 usec
DE       4.10 usec
TE       300.2 K
D1       1.0000000 sec
===== CHANNEL f1 =====
NUC1     13C
P1       11.00 usec
PL1      0.00 dB
SFO1     100.6283500 MHz
F2 - Processing parameters
SI       300.1300000 MHz
SF       300.1300000 MHz
WDW      EM
SSB      0.00 Hz
GB       0.00 Hz
PC       1.00
    
```

AM-106



```

Current Data Parameters
Date_   12 Jan
Time_   16:02
PROCNO  1
F2 - Acquisition Parameters
Date_   20110805
Time_   11:05
INSTRUM spect
PROBHD  5 mm QNP 1H/1
PULPROG zgpg30
TD       65536
SOLVENT CDCl3
NS       8
DS       4
SWH      6188.115 Hz
FIDRES  0.29842 Hz
AQ       5.255087 sec
RG       327.5
SQ       89.810 usec
DE       4.10 usec
TE       300.2 K
D1       1.0000000 sec
===== CHANNEL f1 =====
NUC1     13C
P1       11.00 usec
PL1      0.00 dB
SFO1     100.6283500 MHz
F2 - Processing parameters
SI       300.1300000 MHz
SF       300.1300000 MHz
WDW      EM
SSB      0.00 Hz
GB       0.00 Hz
PC       1.00
    
```



```

Filename      - EP VJR-106 CARBON-4.j
Author       - delta
Experiment   - single pulse_dac
Sample Id    - EP VJR-106
Solvent      - CDCl3
Creation time - 16-JUN-2011 20:09:55
Revision time - 21-JUN-2011 13:28:47
Current time  - 21-JUN-2011 13:29:44

Comment      - EP VJR-106
Data format  - 1D COMPLEX
Dir size     - 26214
Dir title    - 13C
Dir units    - [ppm]
Dimensions   - X
Site         - EXI 400P
Spectrometer - DELTA2 NMR

Field strength - 9.389766 [T] (400 [MHz])
X_acq duration - 1.04333312 [s]
X_domain       - 13C
X_freq         - 100.62530333 [MHz]
X_offset       - 100 [ppm]
X_points       - 32768
X_procscans   - 4
X_resolution   - 0.38848688 [Hz]
X_sweep        - 31.40703518 [kHz]
Irr_domain     - 1B
Irr_freq       - 399.78219838 [MHz]
Irr_offset     - 5 [ppm]
Clipped        - FALSE
Mod return     - 1
Scans          - 750
Total_scans    - 750

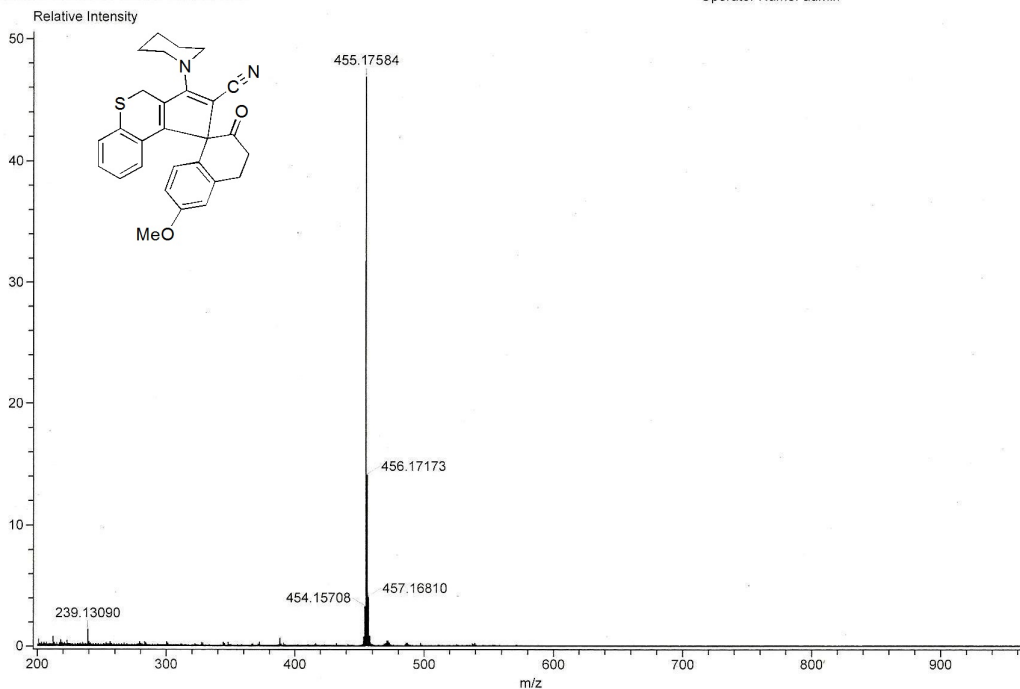
X_90 width    - 11.75 [us]
X_acq time    - 1.04333312 [s]
X_angle       - 30 [deg]
X_atn         - 10 [dB]
X_pulse       - 3.9466667 [us]
Irr_atn dac   - 24.95 [dB]
Irr_atn mod   - 24.95 [dB]
Irr_noise     - Waifs
Decoupling    - TRUE
Initial wait   - 2 [s]
Mod           - TRUE
Mod time      - 2 [s]
Recvr gain    - 56
Relaxation delay - 2 [s]
Repetition time - 3.04333312 [s]
Temp_set      - 22.7 [degC]
    
```

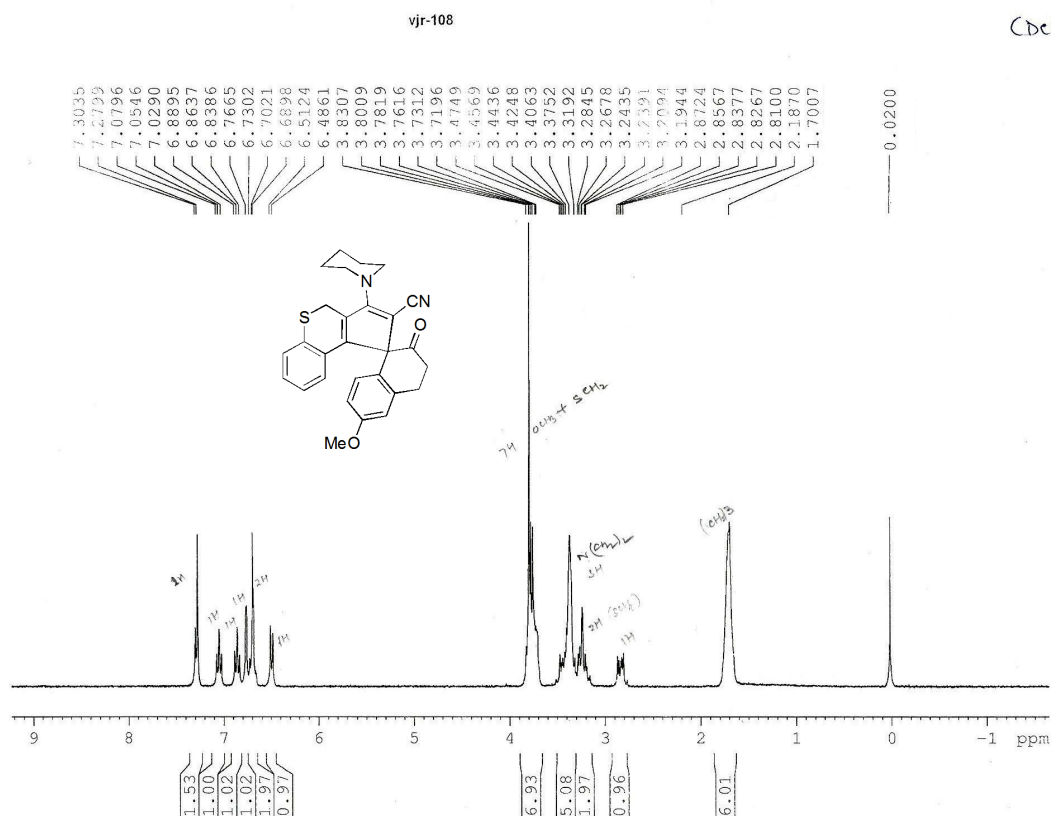
6'-methoxy-2'-oxo-3-(piperidin-1-yl)-3',4'-dihydro-2'H,4H-spiro[cyclopenta[c]thiochromene-1,1'-naphthalene]-2-carbonitrile (17c):

Acq. Data Name: 11FEB034A
 Internal Sample Id: VJR-106
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Correct Base[5.0%]; Average(MS[1] 0.409..0.425)
 Experiment Date/Time: 2/7/2011 2:08:04 PM

Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.417[min]

Operator Name: admin





8-chloro-6'-methoxy-2'-oxo-3-(piperidin-1-yl)-3',4'-dihydro-2'H,4H-spiro[cyclopenta[c]thiochromene-1,1'-

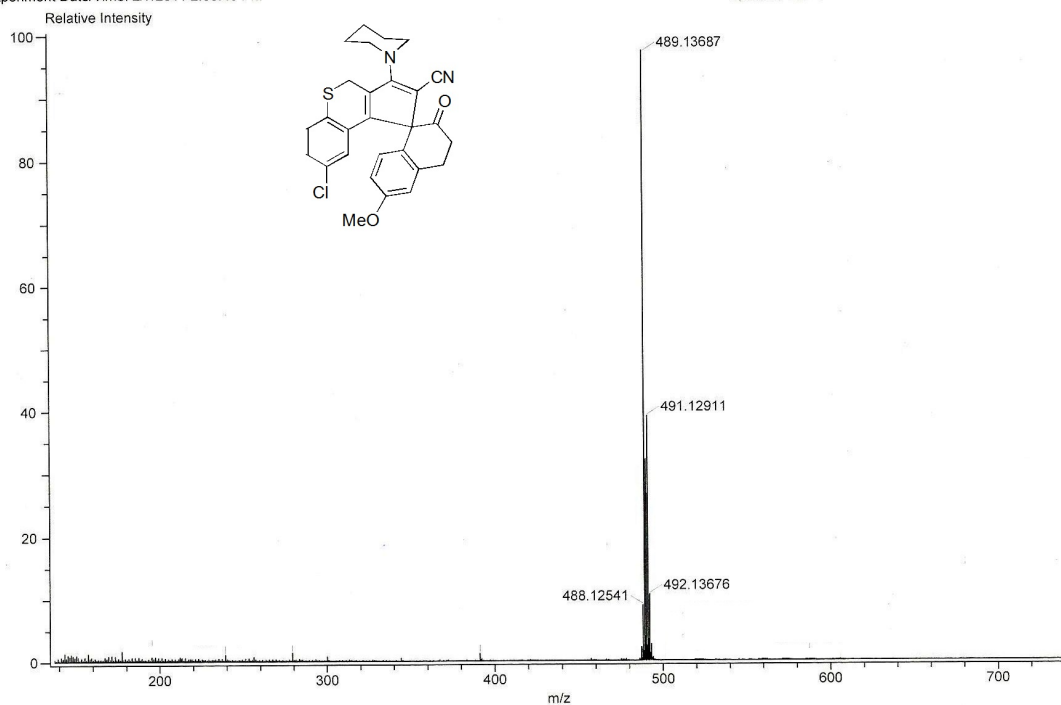
naphthalene]-2-carbonitrile (17d):

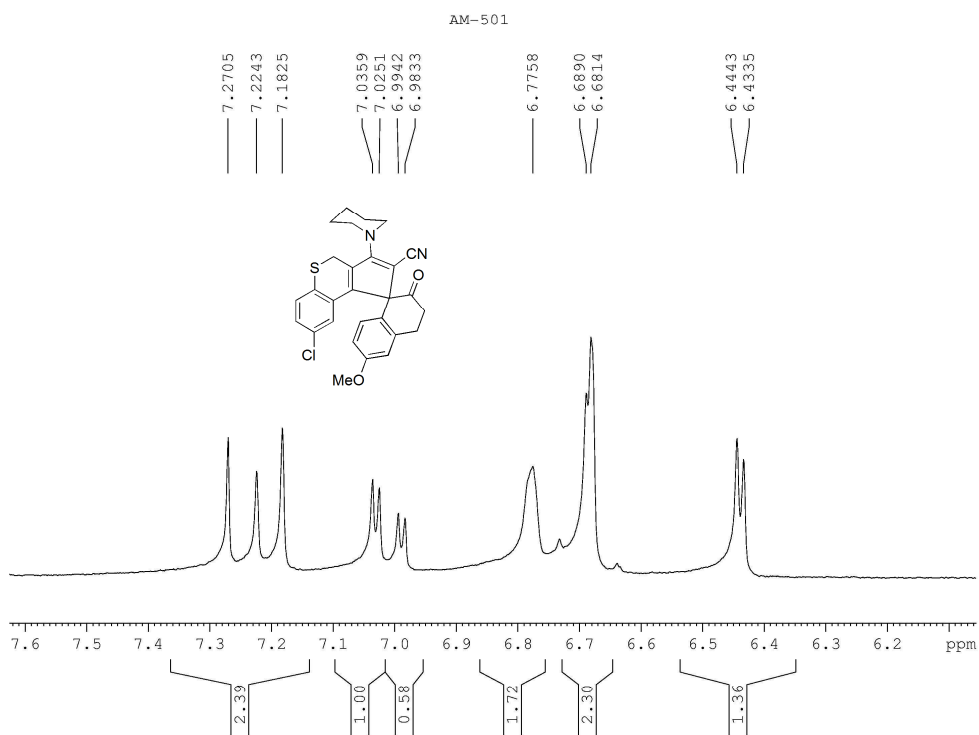
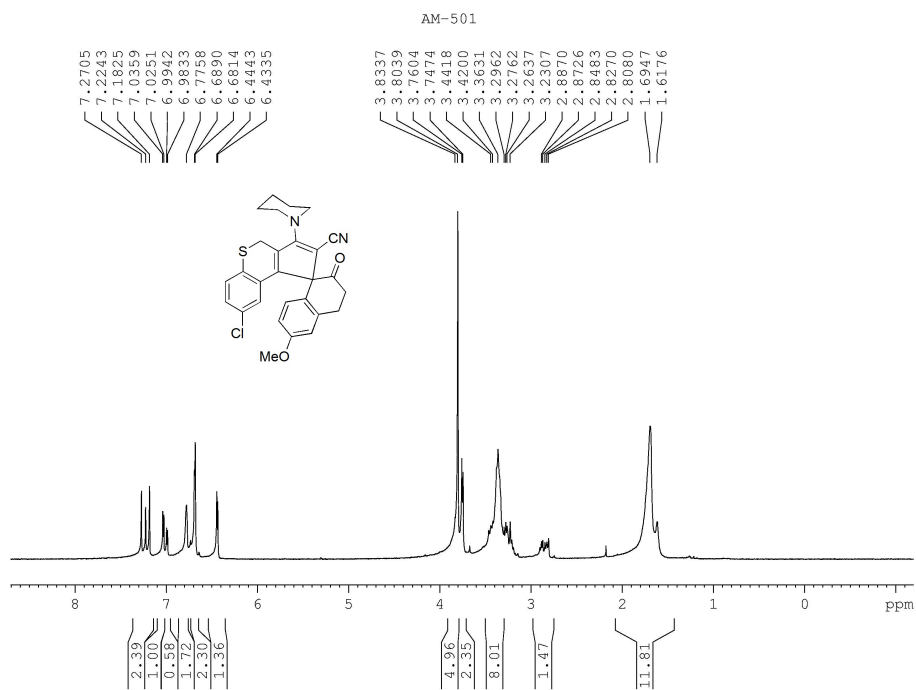
Acq. Data Name: 11FEB032A
 Internal Sample Id: VJR-109
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Correct Base[5.0%];Average[MS[1]0.100.0.112]
 Experiment Date/Time: 2/7/2011 2:03:46 PM

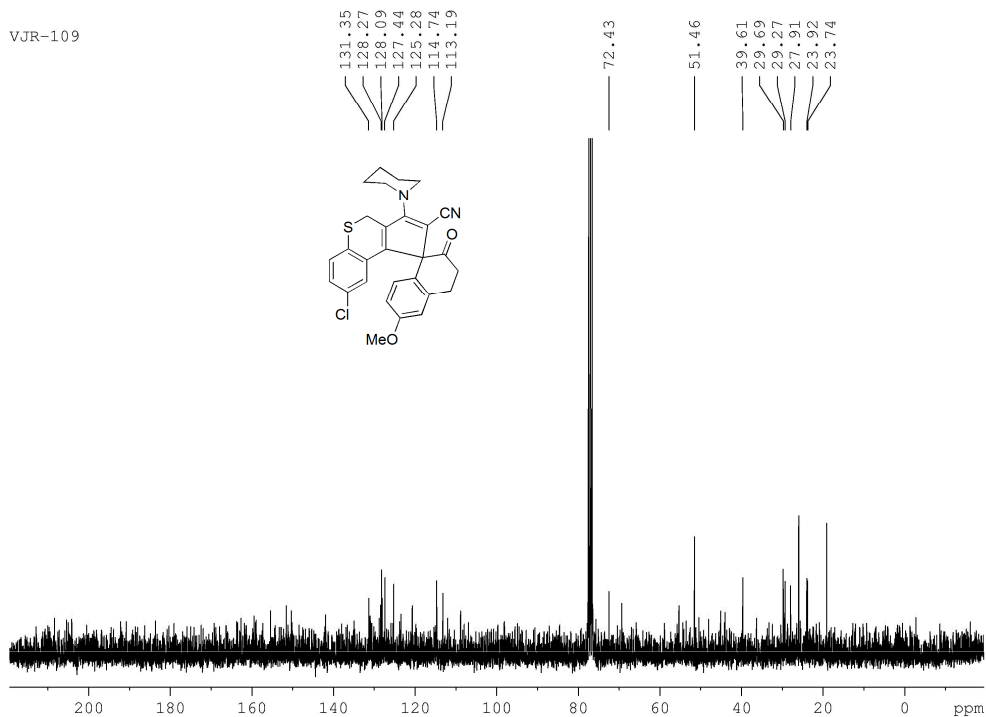
Orifice1 Volt Sweep: 15V
 Acquired m/z Range: 50.0..1000.0

Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.102[min]

Operator Name: admin







6'-methoxy-2'-oxo-3-(piperidin-1-yl)-3',4,4',5-tetrahydro-2'H-spiro[cyclopenta[a]naphthalene-1,1'-naphthalene]-2-carbonitrile (17e):

Acq. Data Name: 11FEB033A

Internal Sample Id: VJR-110

Ionization Mode: ESI+

MS Calibration Name: YOKUDELNA_ES+_2000

Reduction History: Correct Base[5.0%];Average(MS[1] 0.045..0.195)

Experiment Date/Time: 2/7/2011 2:05:11 PM

Orifice1 Volt Sweep: 15V

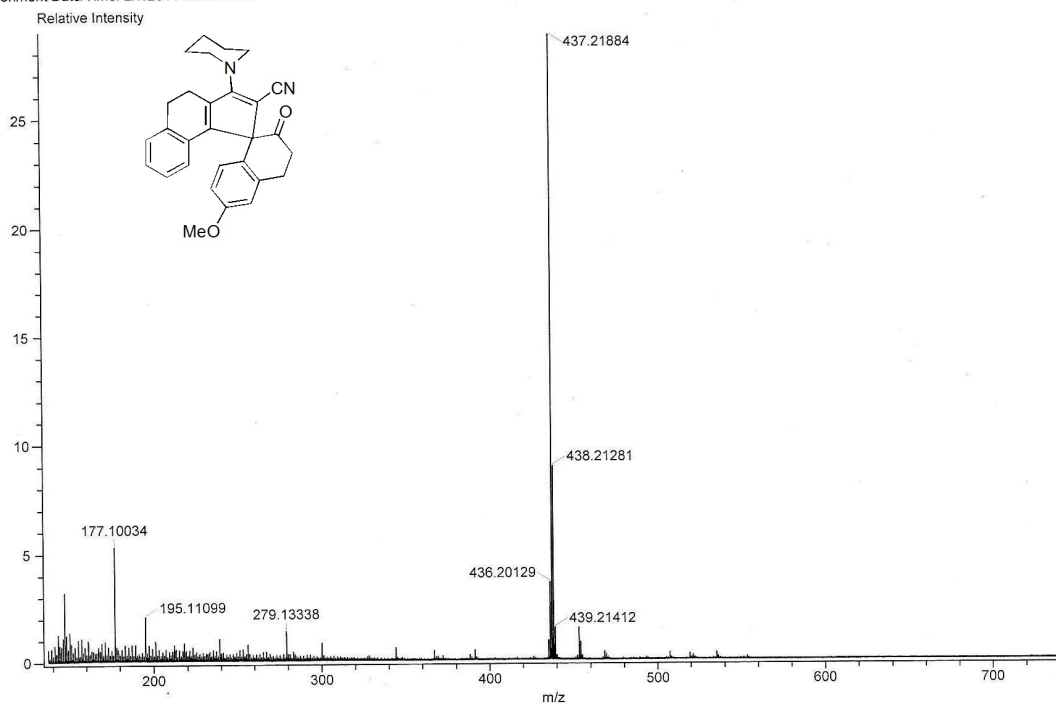
Acquired m/z Range: 50.0..1000.0

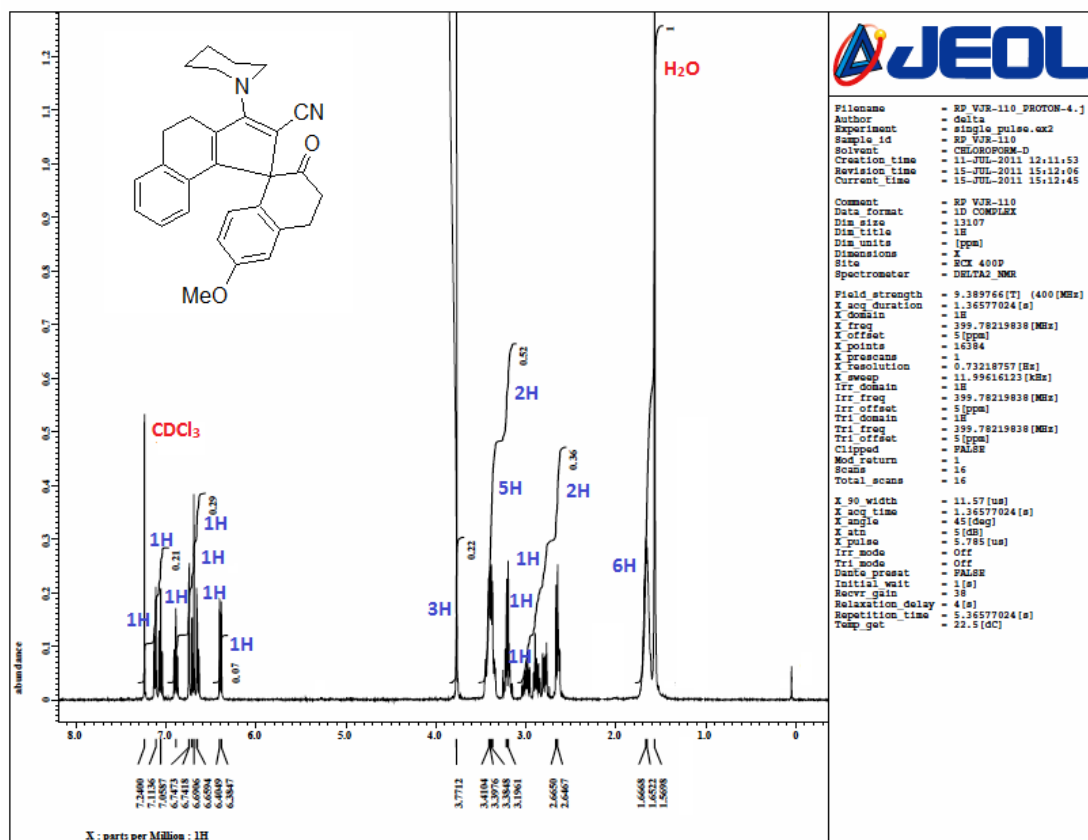
Spec. Record Interval: 0.4[s]

Ring Lens Volt: 11[V]

Time of Maximum: 0.103[min]

Operator Name: admin



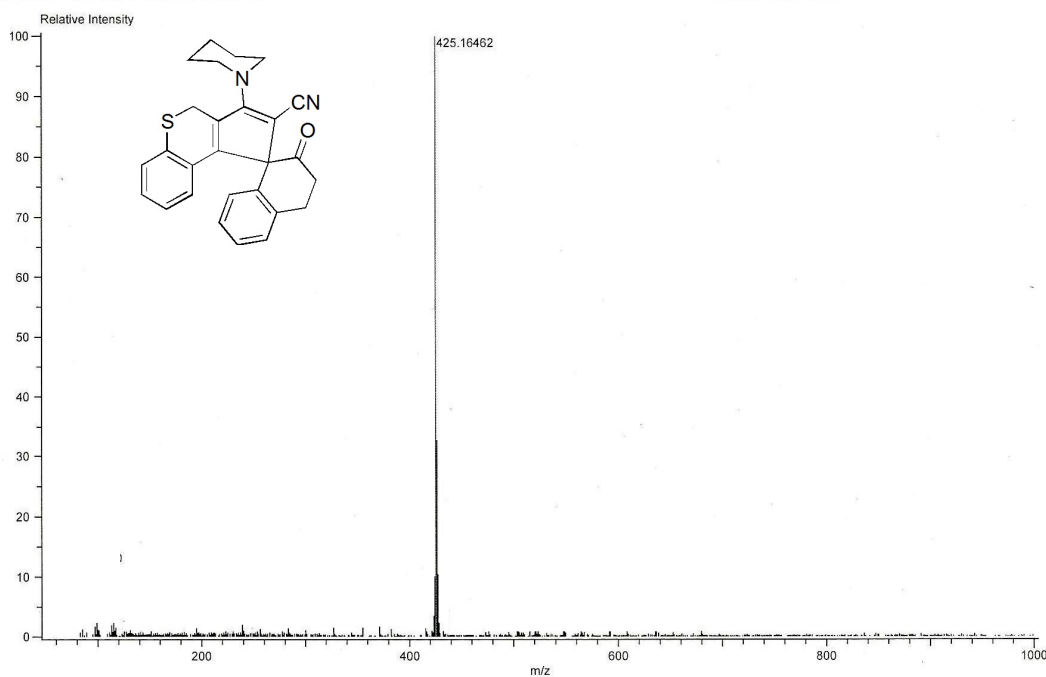


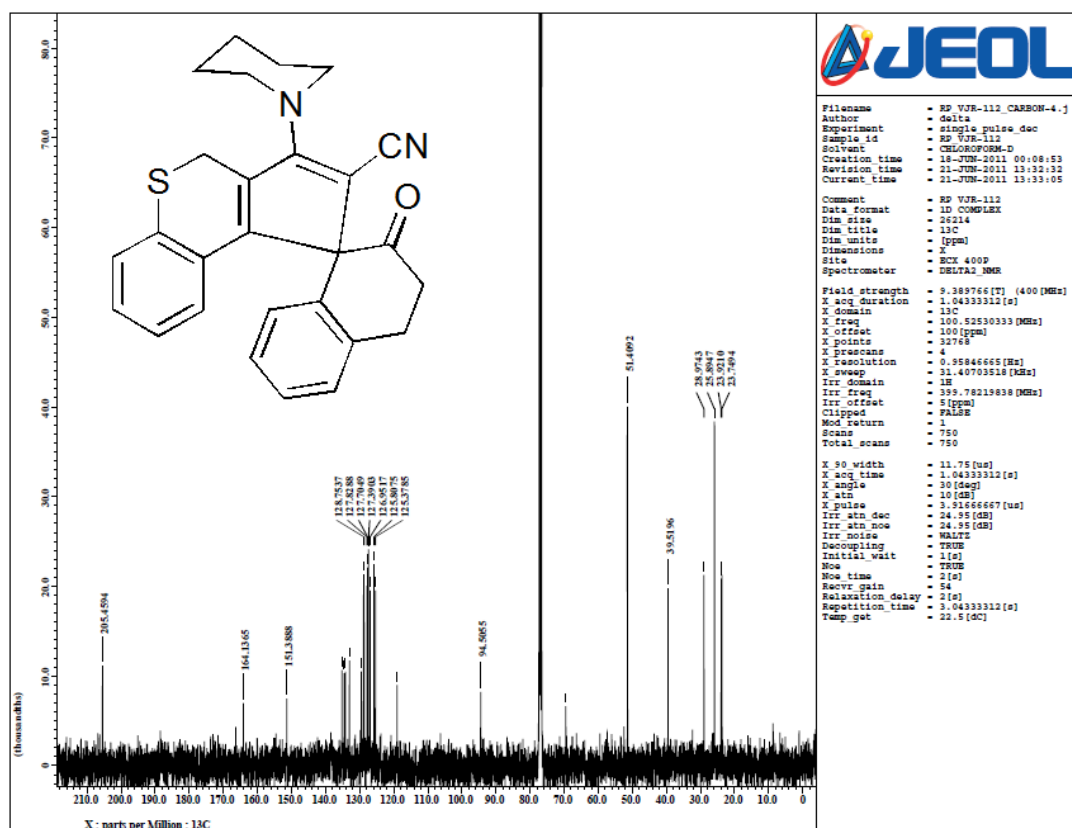
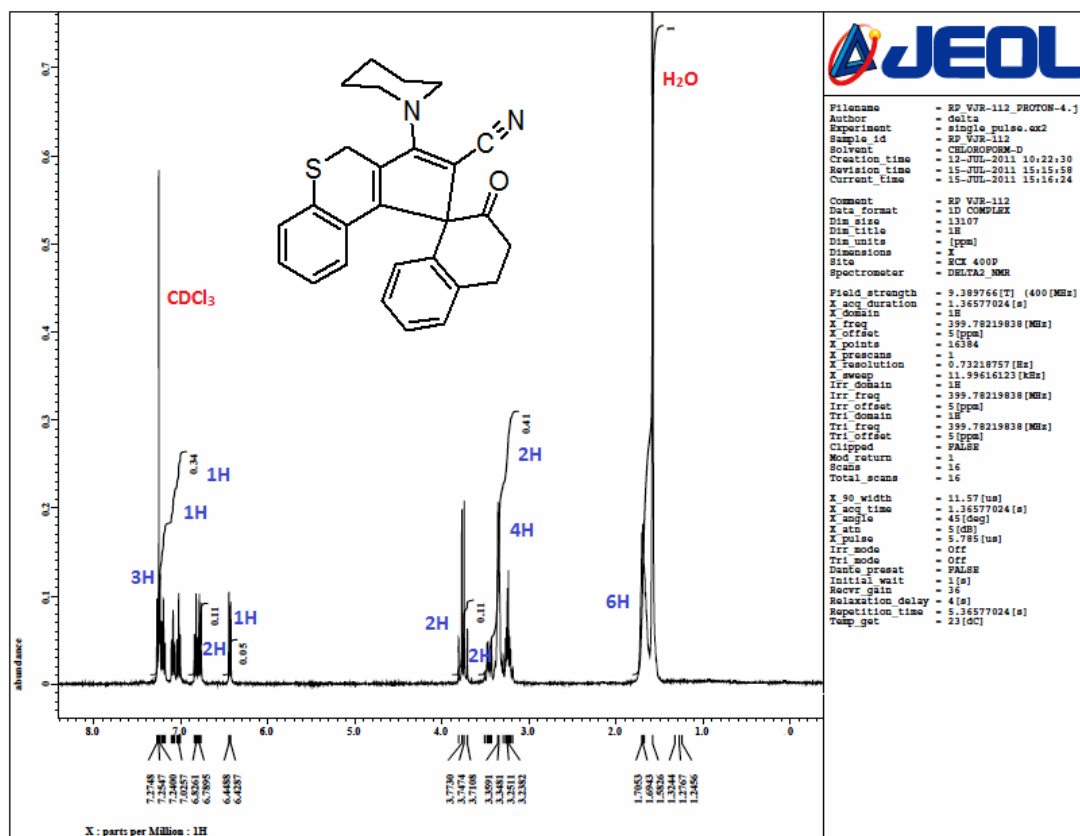
2'-oxo-3-(piperidin-1-yl)-3',4'-dihydro-2'H,4H-spiro[cyclopenta[c]thiochromene-1,1'-naphthalene]-2-carbonitrile (17f):

Acq. Data Name: 11FEB145A
 Internal Sample Id: VJR-112
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+ 2000
 Reduction History: Determine m/z[Peak Detect[Centroid,50,Area];Correct Base[5.0%];Smooth[3]];Add[Correct Base[5.0%];Average[MS[1] 0.162..0.163];1.0];Correct Base[5.0%];...

Orifice1 Volt Sweep: 15V
 Acquired m/z Range: 50.0..1000.0

Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.083[min]
 Operator Name: admin



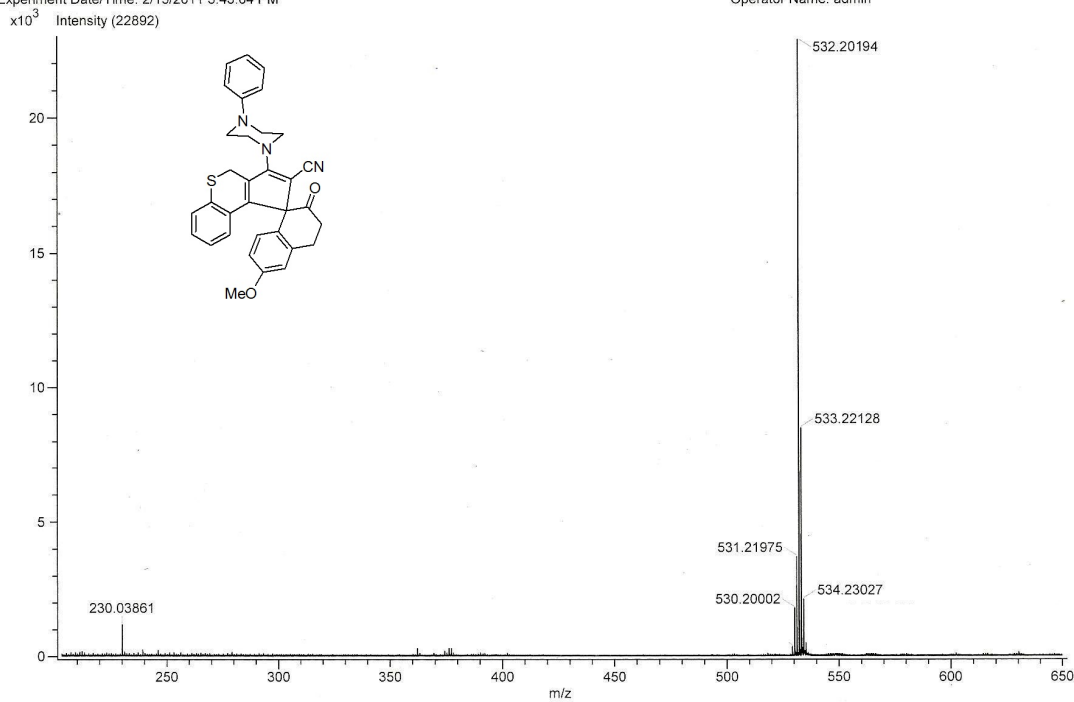


6'-methoxy-2'-oxo-3-(4-phenylpiperazin-1-yl)-3',4'-dihydro-2'H,4H-spiro[cyclopenta[c]thiochromene-1,1'-naphthalene]-2-carbonitrile (12g):

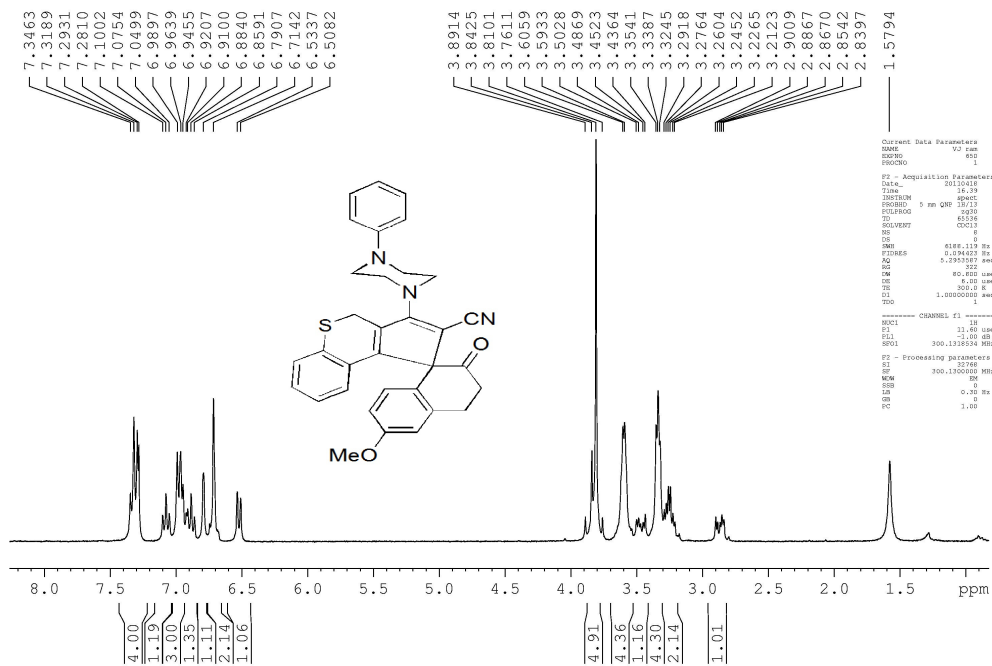
thiochromene-1,1'-

Acq. Data Name: 11FEB144A
 Internal Sample Id: VJR-113
 Ionization Mode: ESI+
 MS Calibration Name: YOKUDELNA_ES+_2000
 Reduction History: Correct Base[5.0%]; Average(MS[1]) 0.106..0.110
 Experiment Date/Time: 2/15/2011 3:43:04 PM

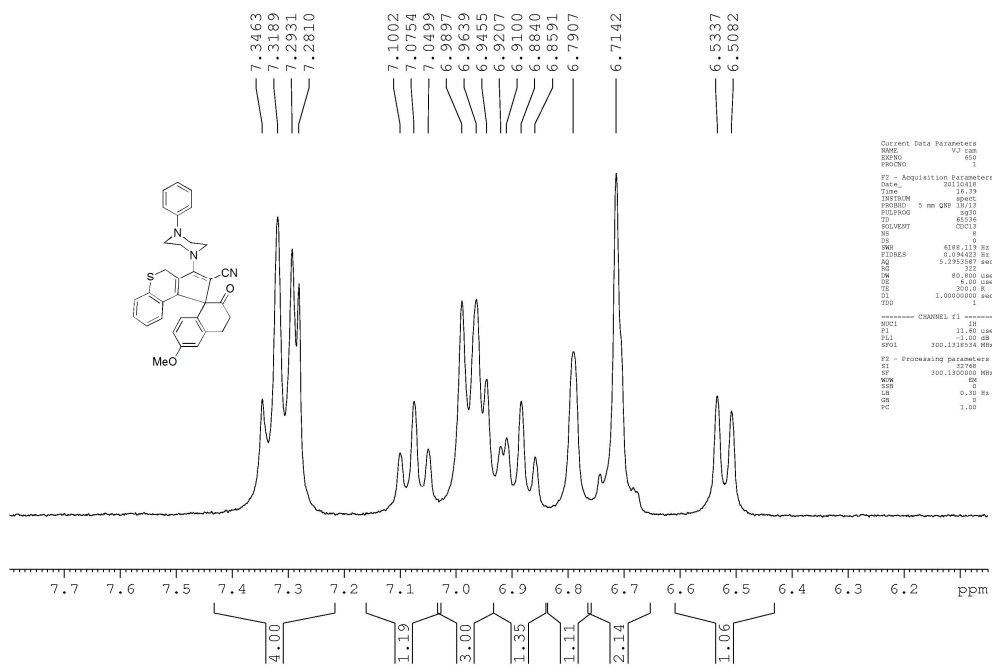
Spec. Record Interval: 0.4[s]
 Ring Lens Volt: 11[V]
 Time of Maximum: 0.109[min]
 Operator Name: admin



AM-512



AM-512



AM-512

