Ruthenium Catalyzed Asymmetric Reduction of Isoxazolium Salts: Access to Optically Active Δ^4-Isoxazolines

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Supporting Information

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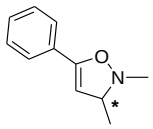
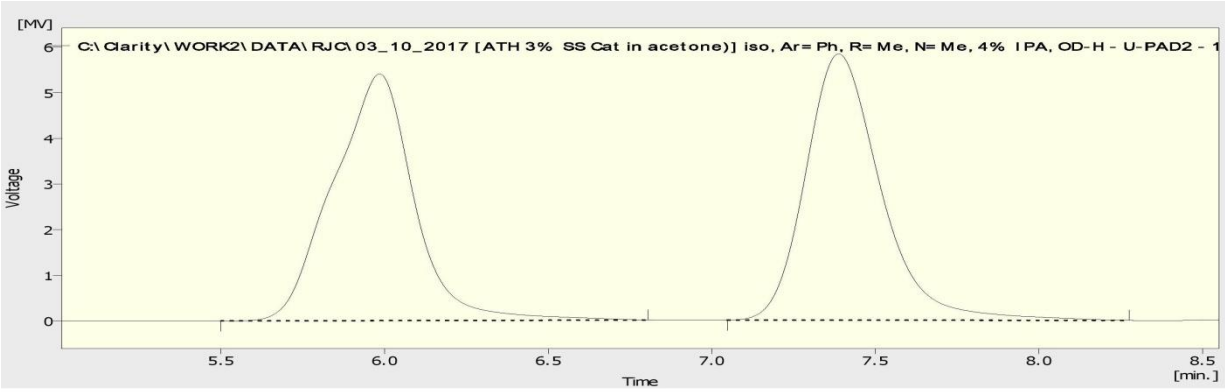
I. HPLC spectra of (+)-Δ^4-isoxazolines (3)



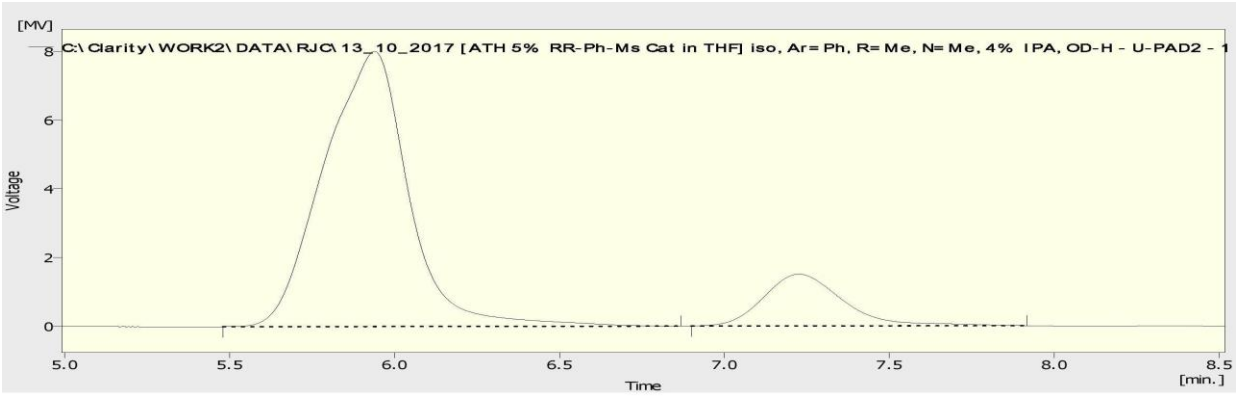
3a

HPLC (Diacel OD-H column, Hexane:IPA = 96:4, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 5.94 min, t_2 = 7.22 min; $[\alpha]_D^{25}$ = +58.80° (c = 0.99, $CHCl_3$).



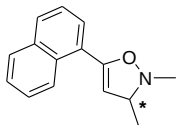
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\03_10_2017 [ATH 3% SS Cat in acetone)] iso, Ar=Ph, R=Me, N=Me, 4% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	5.984	929.590	54.045	49.9	48.1	0.27	
2	7.388	932.440	58.359	50.1	51.9	0.24	
	Total	1862.030	112.404	100.0	100.0		



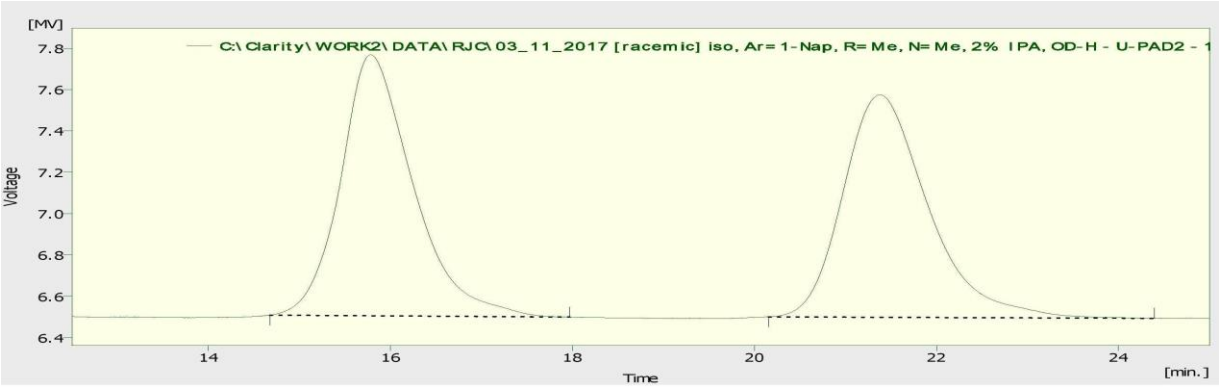
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\13_10_2017 [ATH 5% RR-Ph-Ms Cat in THF] iso, Ar=Ph, R=Me, N=Me, 4% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	5.940	1430.493	80.103	85.1	84.2	0.28	
2	7.224	250.243	15.036	14.9	15.8	0.25	
	Total	1680.737	95.139	100.0	100.0		



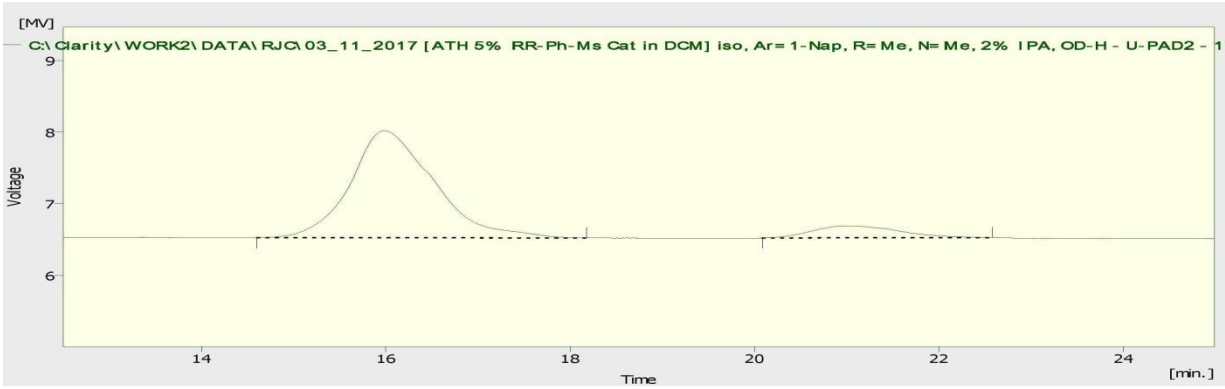
3b

HPLC (Diacel OD-H column, Hexane:IPA = 98:2, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 15.98 min, t_2 = 21.04 min; $[\alpha]_D^{26}$ = +19.55° (c = 0.99, $CHCl_3$).



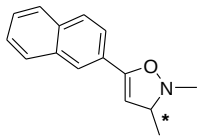
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\03_11_2017 [racemic] iso, Ar=1-Nap, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	15.780	698.620	12.628	50.0	54.0	0.81	
2	21.384	698.806	10.763	50.0	46.0	0.98	
	Total	1397.426	23.391	100.0	100.0		



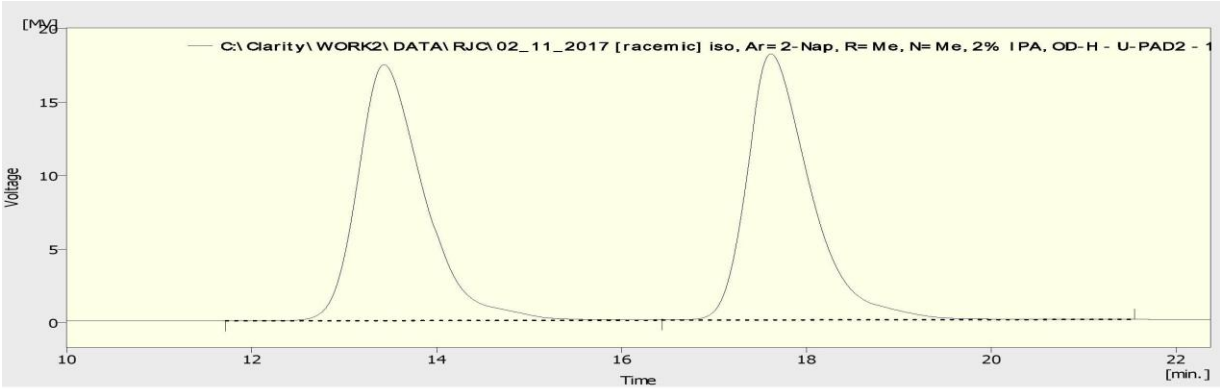
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\03_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] iso, Ar=1-Nap, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	15.980	940.369	14.947	89.6	89.9	0.94	
2	21.040	108.907	1.686	10.4	10.1	1.04	
	Total	1049.276	16.632	100.0	100.0		



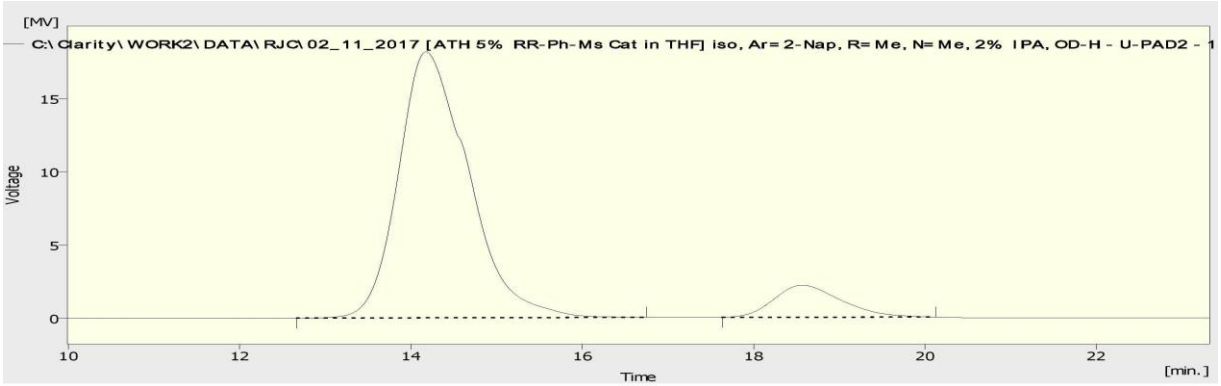
3c

HPLC (Diacel OD-H column, Hexane:IPA = 98:2, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 14.72 min, t_2 = 18.56 min; $[\alpha]_D^{25}$ = +58.77° (c = 1.02, $CHCl_3$).



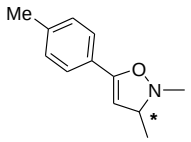
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\02_11_2017 [racemic] iso, Ar=2-Nap, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	13.432	8674.652	173.930	50.1	49.0	0.74	
2	17.616	8630.061	180.851	49.9	51.0	0.70	
	Total	17304.714	354.781	100.0	100.0		



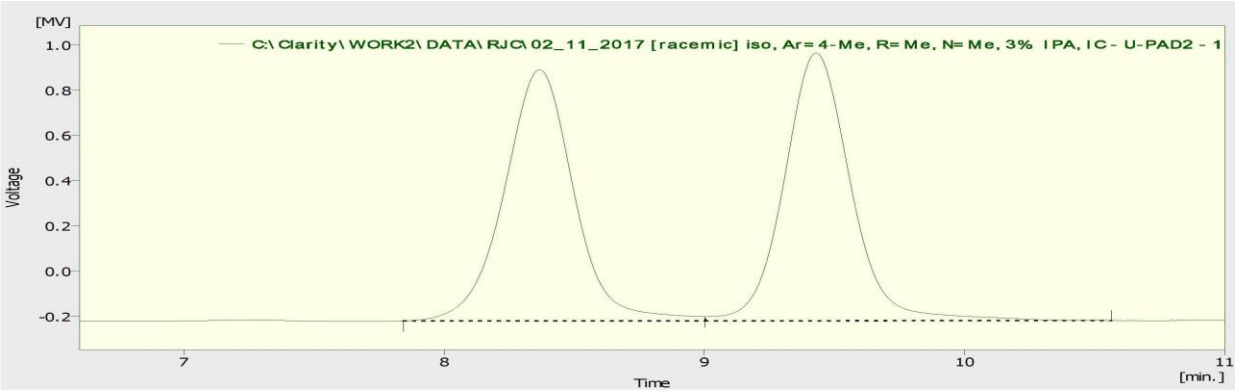
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\02_11_2017 [ATH 5% RR-Ph-Ms Cat in THF] iso, Ar=2-Nap, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	14.176	10292.073	181.481	89.6	89.2	0.89	
2	18.564	1190.437	21.903	10.4	10.8	0.84	
	Total	11482.510	203.384	100.0	100.0		



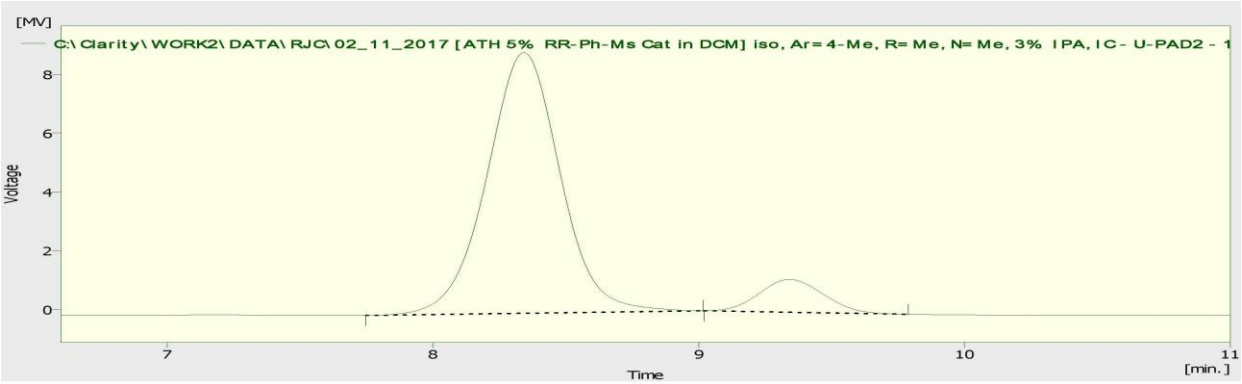
3d

HPLC (Diacel IC column, Hexane:IPA = 97:3, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 8.34 min, t_2 = 9.34 min; $[\alpha]_D^{25}$ = +53.77° (c = 1.01, $CHCl_3$).



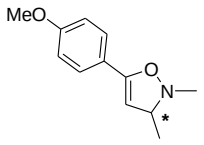
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\02_11_2017 [racemic] iso, Ar=4-Me, R=Me, N=Me, 3% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	8.368	206.532	11.112	49.6	48.4	0.28	
2	9.428	210.000	11.841	50.4	51.6	0.27	
	Total	416.533	22.953	100.0	100.0		



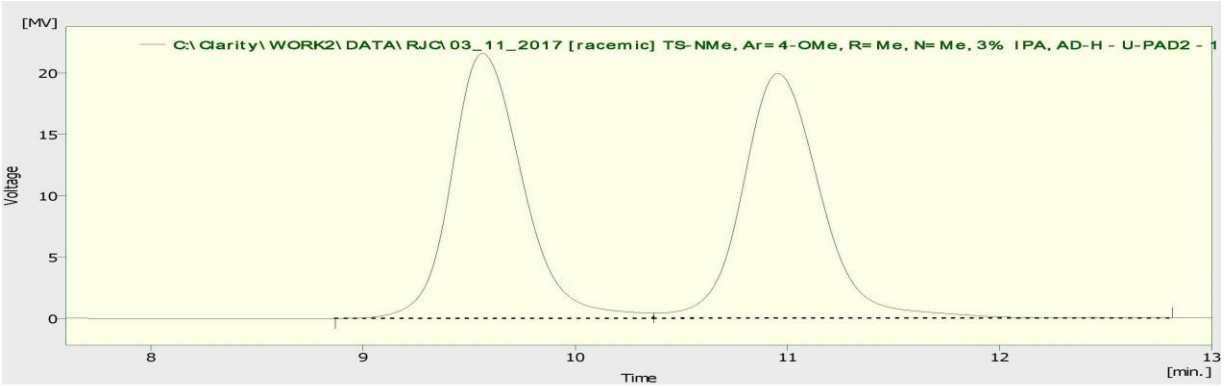
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\02_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] iso, Ar=4-Me, R=Me, N=Me, 3% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	8.344	1700.613	88.650	90.1	88.8	0.29	
2	9.344	187.336	11.167	9.9	11.2	0.27	
	Total	1887.949	99.817	100.0	100.0		



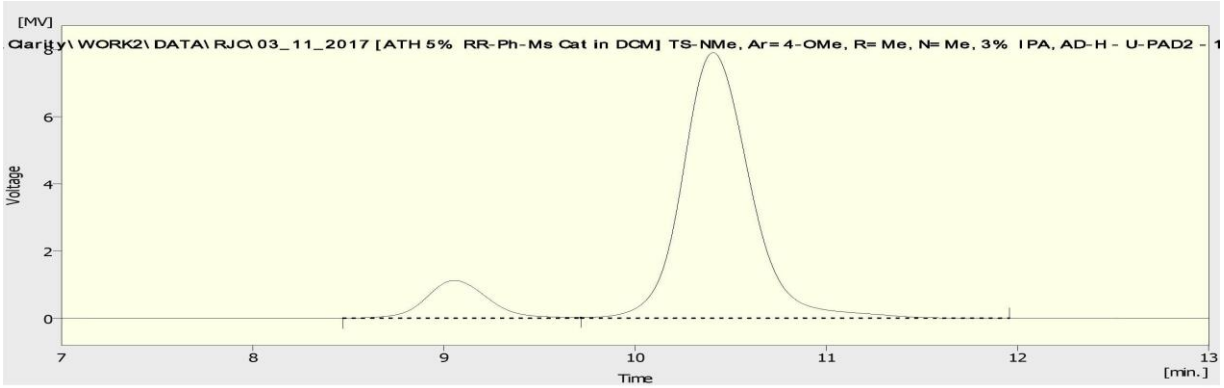
3e

HPLC (Diacel AD-H column, Hexane:IPA = 97:3, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 9.05 min, t_2 = 10.41 min; $[\alpha]_D^{22}$ = +19.95° (c = 1.01, $CHCl_3$).



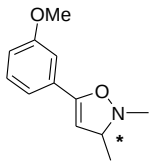
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\03_11_2017 [racemic] TS-NMe, Ar=4-OMe, R=Me, N=Me, 3% IPA, AD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	9.564	5048.353	215.720	49.9	52.0	0.35	
2	10.956	5077.787	199.275	50.1	48.0	0.38	
	Total	10126.139	414.995	100.0	100.0		



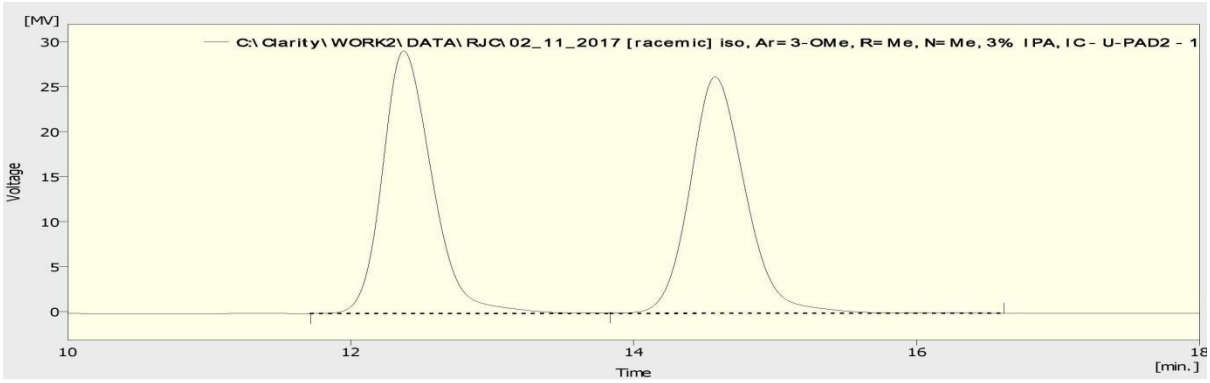
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\03_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] TS-NMe, Ar=4-OMe, R=Me, N=Me, 3% IPA, AD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	9.052	252.630	11.193	11.5	12.4	0.34	
2	10.408	1940.912	78.968	88.5	87.6	0.37	
	Total	2193.542	90.162	100.0	100.0		



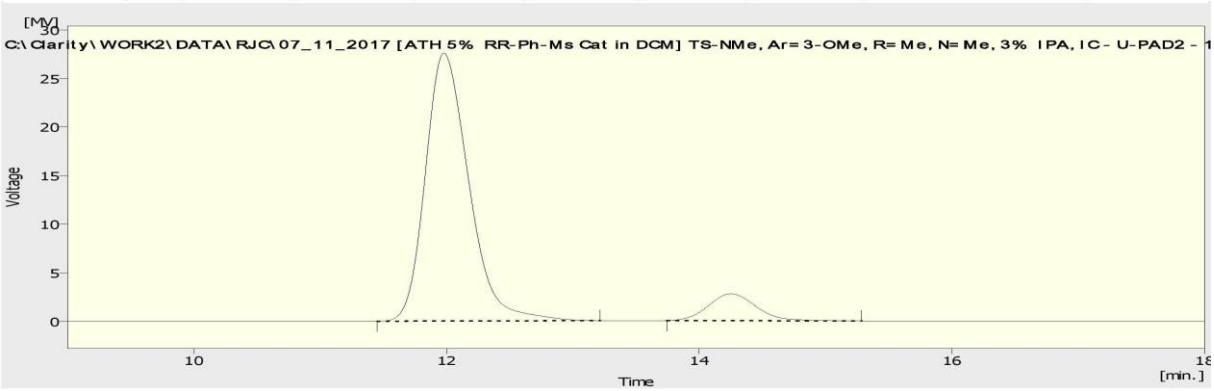
3f

HPLC (Diacel IC column, Hexane:IPA = 97:3, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 11.98 min, t_2 = 14.25 min; $[\alpha]_D^{23}$ = +39.93° (c = 0.99, $CHCl_3$).



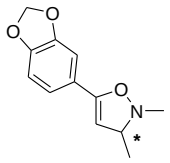
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\02_11_2017 [racemic] iso, Ar=3-OMe, R=Me, N=Me, 3% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	12.376	7110.623	291.758	49.9	52.6	0.37	
2	14.576	7143.885	262.830	50.1	47.4	0.42	
	Total	14254.509	554.588	100.0	100.0		



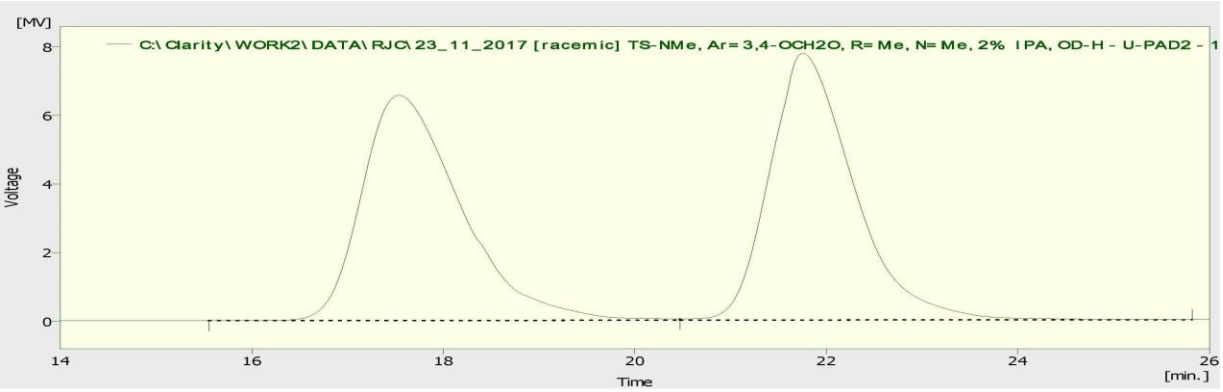
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\07_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] TS-NMe, Ar=3-OMe, R=Me, N=Me, 3% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	11.980	6595.135	275.840	90.0	90.9	0.36	
2	14.252	728.795	27.683	10.0	9.1	0.40	
	Total	7323.931	303.523	100.0	100.0		



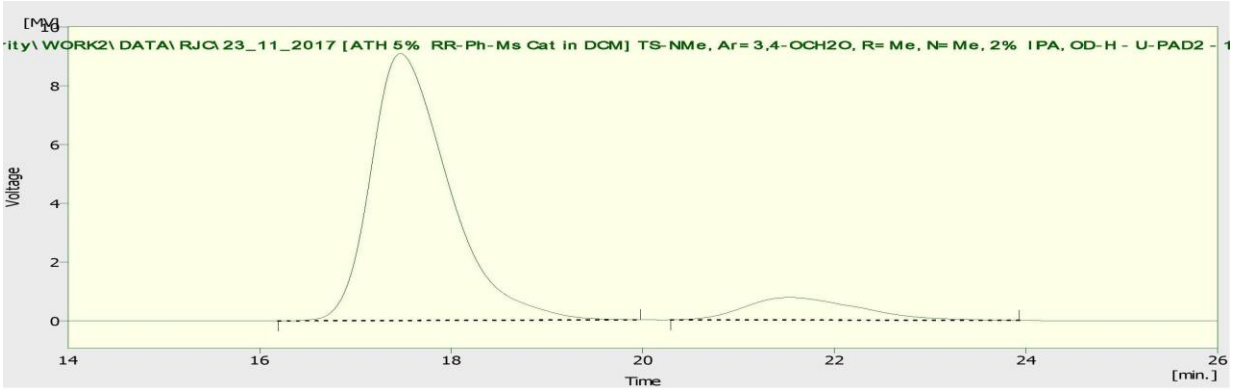
3g

HPLC (Diacel OD-H column, Hexane:IPA = 98:2, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 17.47 min, t_2 = 21.52 min; $[\alpha]_D^{21}$ = +59.24° (c = 1.01, $CHCl_3$).



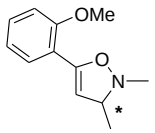
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\23_11_2017 [racemic] TS-NMe, Ar=3,4-OCH2O, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	17.536	4702.539	65.632	49.9	45.8	1.08	
2	21.756	4728.515	77.721	50.1	54.2	0.92	
	Total	9431.054	143.353	100.0	100.0		



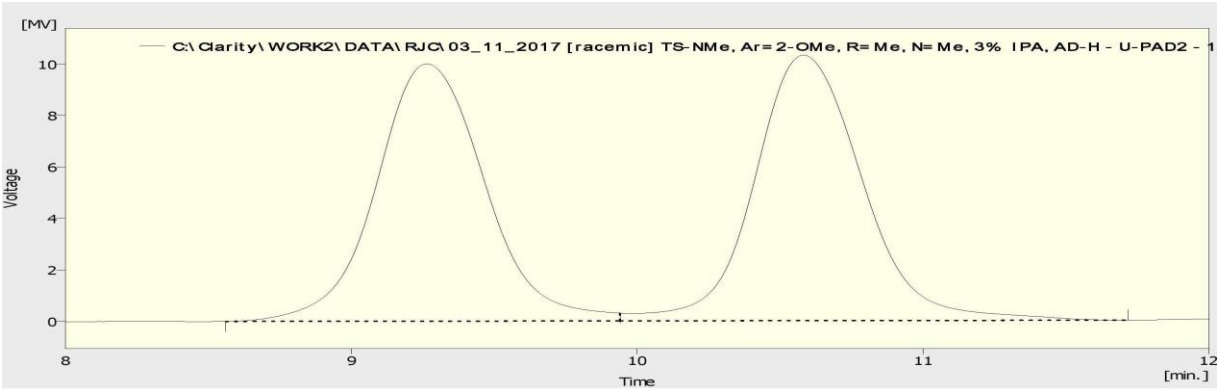
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\23_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] TS-NMe, Ar=3,4-OCH2O, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	17.468	5126.146	90.823	89.0	92.2	0.85	
2	21.524	635.896	7.696	11.0	7.8	1.32	
	Total	5762.042	98.518	100.0	100.0		



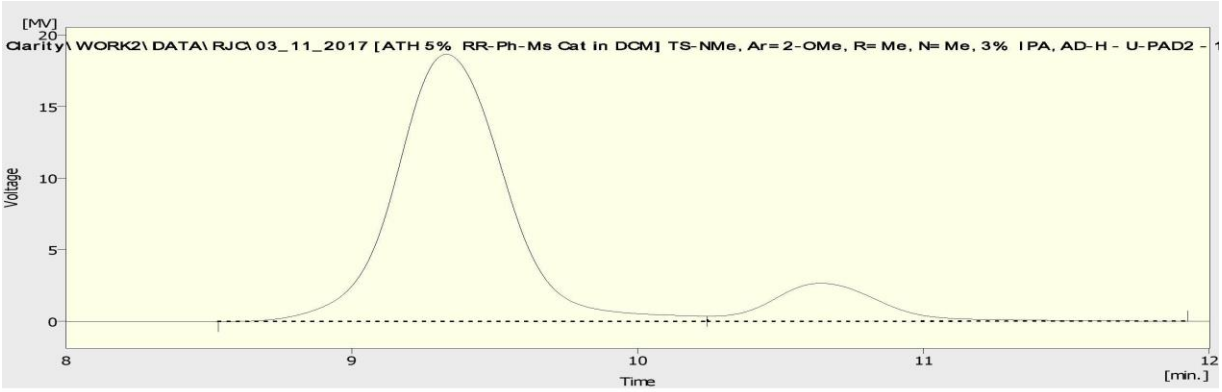
3h

HPLC (Diacel AD-H column, Hexane:IPA = 97:3, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 9.33 min, t_2 = 10.64 min; $[\alpha]_D^{23}$ = +59.75° (c = 1.00, $CHCl_3$).



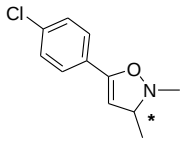
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\03_11_2017 [racemic] TS-NMe, Ar=2-OMe, R=Me, N=Me, 3% IPA, AD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	9.264	2641.499	99.936	49.3	49.2	0.40	
2	10.580	2717.113	103.110	50.7	50.8	0.40	
	Total	5358.612	203.046	100.0	100.0		



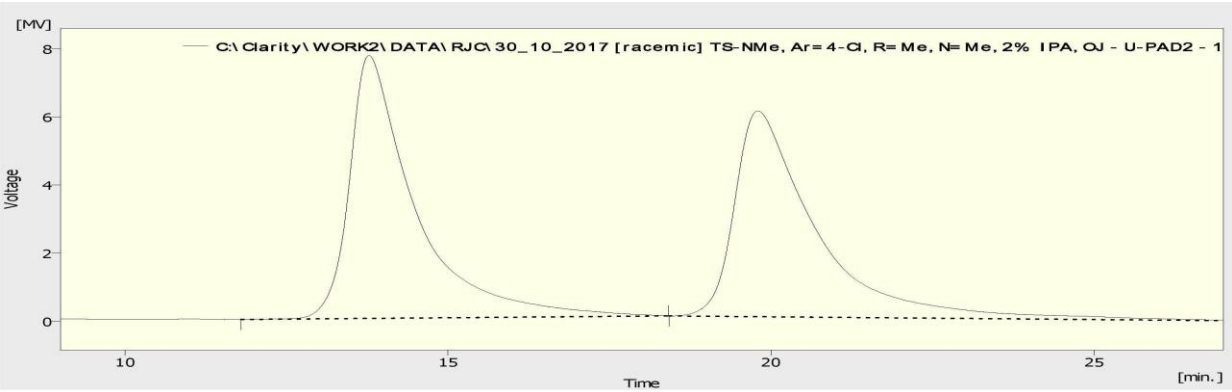
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\03_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] TS-NMe, Ar=2-OMe, R=Me, N=Me, 3% IPA, AD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	9.332	5004.127	186.714	87.6	87.6	0.40	
2	10.640	708.426	26.541	12.4	12.4	0.40	
	Total	5712.553	213.254	100.0	100.0		



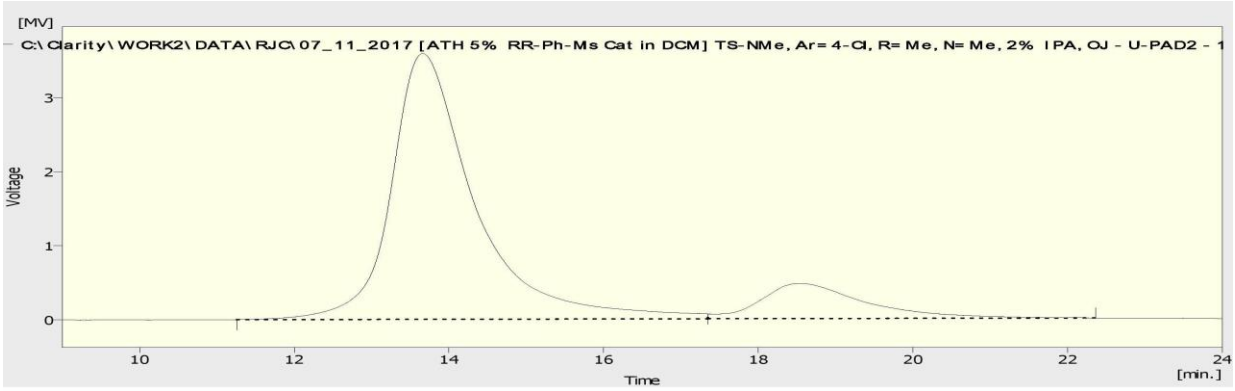
3i

HPLC (Diacel OJ column, Hexane:IPA = 98:2, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 13.66 min, t_2 = 18.55 min; $[\alpha]_D^{22}$ = +39.28° (c = 1.01, $CHCl_3$).



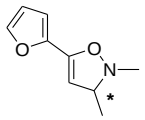
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\30_10_2017 [racemic] TS-NMe, Ar=4-Cl, R=Me, N=Me, 2% IPA, OJ - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	13.776	5467.257	77.258	50.8	56.1	0.92	
2	19.796	5298.029	60.454	49.2	43.9	1.16	
	Total	10765.286	137.712	100.0	100.0		



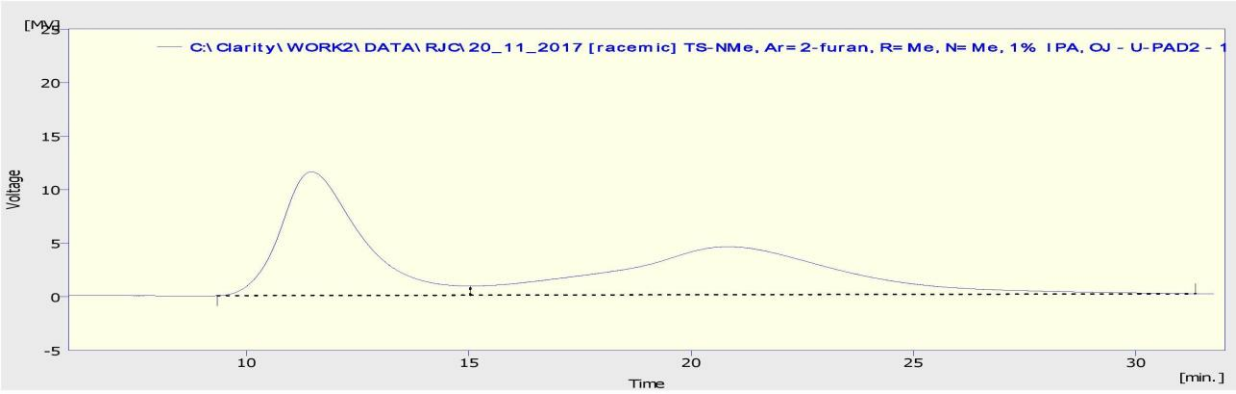
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\07_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] TS-NMe, Ar=4-Cl, R=Me, N=Me, 2% IPA, OJ - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	13.664	2705.487	35.904	85.8	88.3	1.00	
2	18.552	446.944	4.757	14.2	11.7	1.32	
	Total	3152.430	40.661	100.0	100.0		



3j

HPLC (Diacel OJ column, Hexane:IPA = 99:1, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 10.60 min, t_2 = 18.00 min; $[\alpha]_D^{23}$ = +45.74° (c = 1.02, $CHCl_3$).

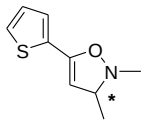


Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\20_11_2017 [racemic] TS-NMe, Ar=2-furan, R=Me, N=Me, 1% IPA, OJ - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	11.464	14588.555	115.534	47.6	72.0	1.81	
2	20.816	16047.640	44.830	52.4	28.0	5.04	
	Total	30636.196	160.364	100.0	100.0		

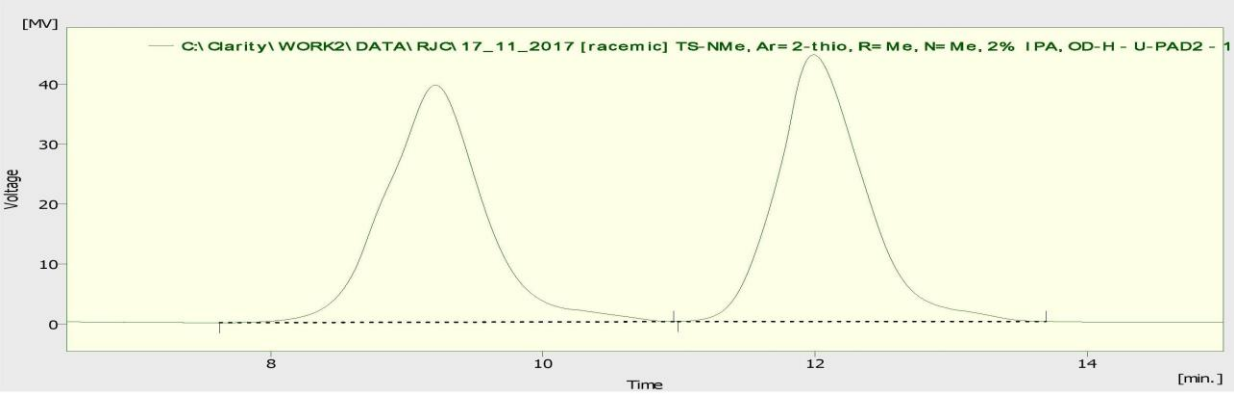
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\20_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] TS-NMe, Ar=2-furan, R=Me, N=Me, 1% IPA, OJ - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	10.600	8829.771	83.758	81.0	92.2	1.48	
2	17.996	2077.296	7.121	19.0	7.8	5.38	
	Total	10907.067	90.879	100.0	100.0		



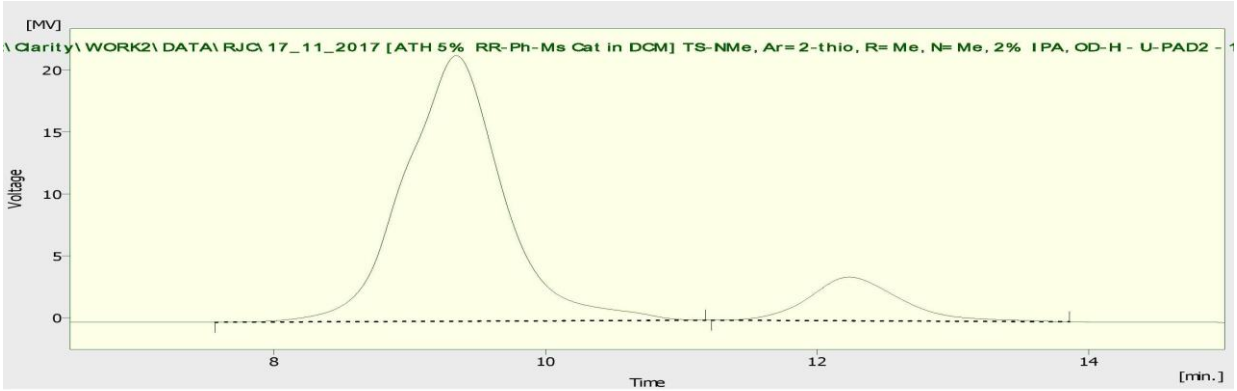
3k

HPLC (Diacel OD-H column, Hexane:IPA = 98:2, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 9.34 min, t_2 = 12.24 min; $[\alpha]_D^{20}$ = +67.69° (c = 1.00, $CHCl_3$).



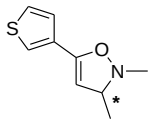
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\17_11_2017 [racemic] TS-NMe, Ar=2-thio, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	9.208	19781.827	395.401	49.9	47.1	0.76	
2	11.988	19834.879	444.743	50.1	52.9	0.67	
	Total	39616.706	840.144	100.0	100.0		



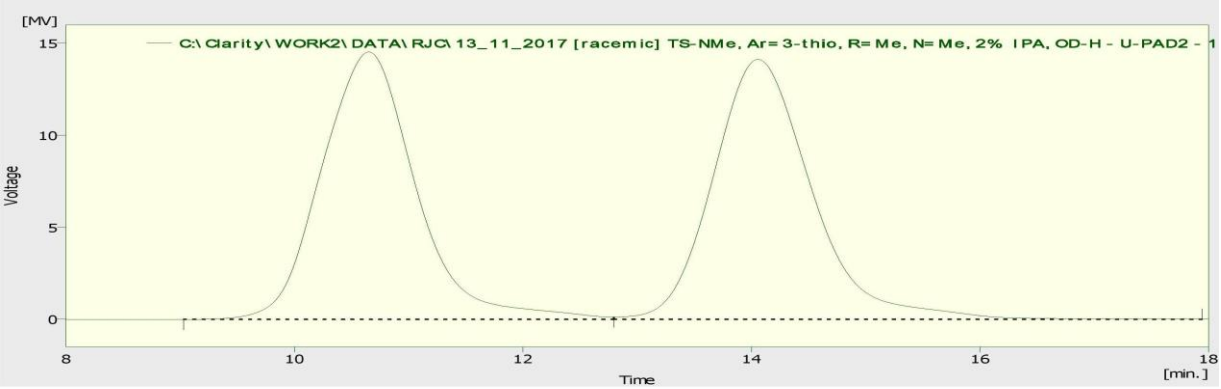
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\17_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] TS-NMe, Ar=2-thio, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	9.344	10782.671	214.679	86.7	85.9	0.76	
2	12.236	1657.208	35.340	13.3	14.1	0.70	
	Total	12439.878	250.019	100.0	100.0		



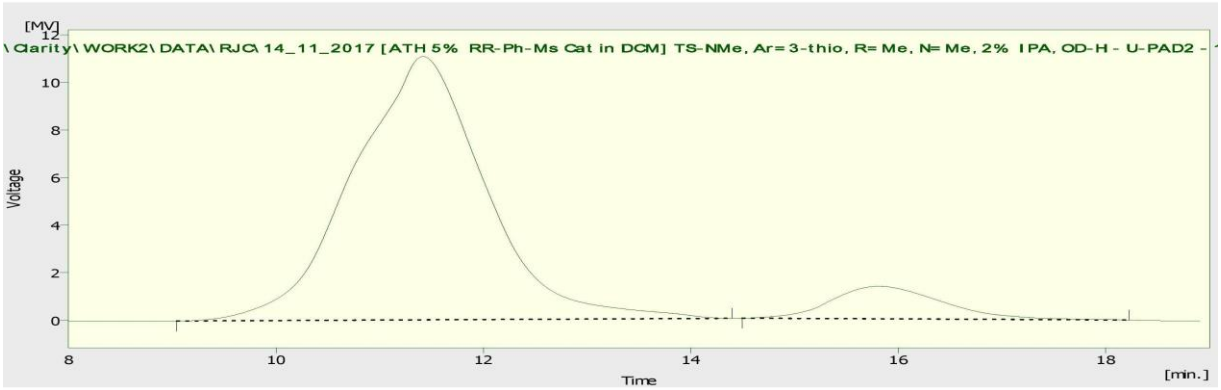
3l

HPLC (Diacel OD-H column, Hexane:IPA = 98:2, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 11.42min, t_2 = 15.82 min; $[\alpha]_D^{23}$ = +51.90° (c = 1.01, $CHCl_3$).



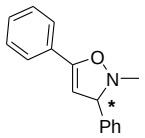
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\13_11_2017 [racemic] TS-NMe, Ar=3-thio, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	10.652	8328.023	145.242	49.7	50.7	0.87	
2	14.056	8439.137	141.141	50.3	49.3	0.90	
	Total	16767.160	286.384	100.0	100.0		



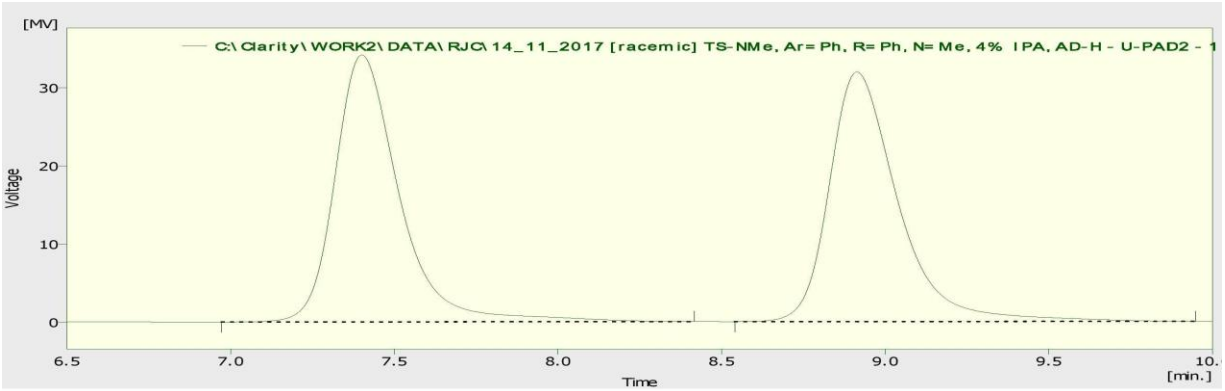
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\14_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] TS-NMe, Ar=3-thio, R=Me, N=Me, 2% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	11.416	9801.053	110.629	90.6	89.0	1.38	
2	15.816	1019.167	13.676	9.4	11.0	1.15	
	Total	10820.220	124.305	100.0	100.0		



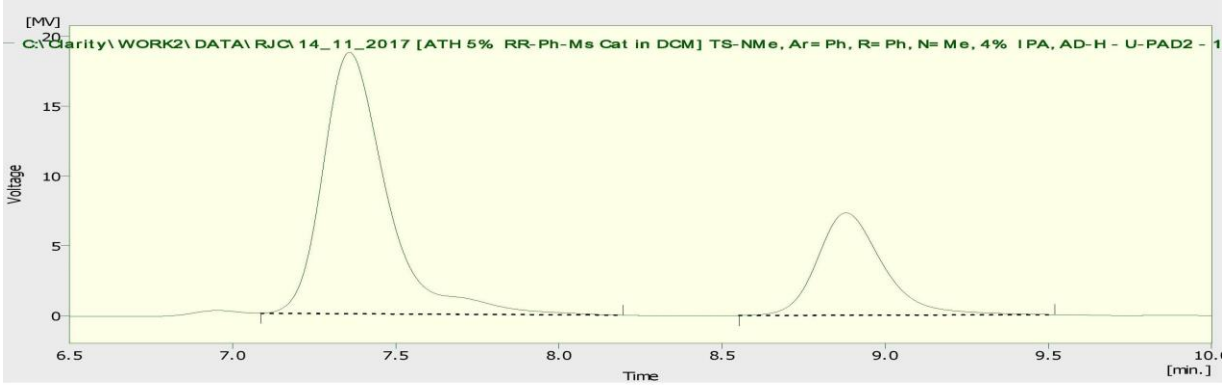
3m

HPLC (Diacel AD-H column, Hexane:IPA = 96:4, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 7.36min, t_2 = 8.88 min; $[\alpha]_D^{25}$ = +56.52° (c = 1.01, $CHCl_3$).



Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\14_11_2017 [racemic] TS-NMe, Ar=Ph, R=Ph, N=Me, 4% IPA, AD-H - U-PAD2 - 1)

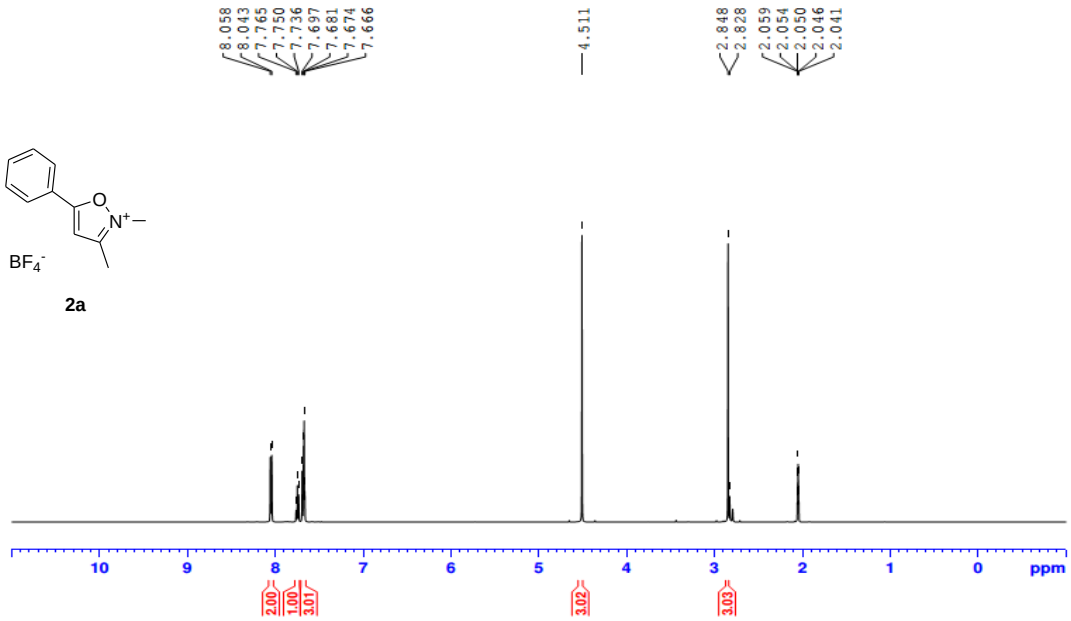
	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	7.400	4677.941	341.716	50.1	51.7	0.20	
2	8.912	4661.037	319.496	49.9	48.3	0.22	
	Total	9338.978	661.212	100.0	100.0		

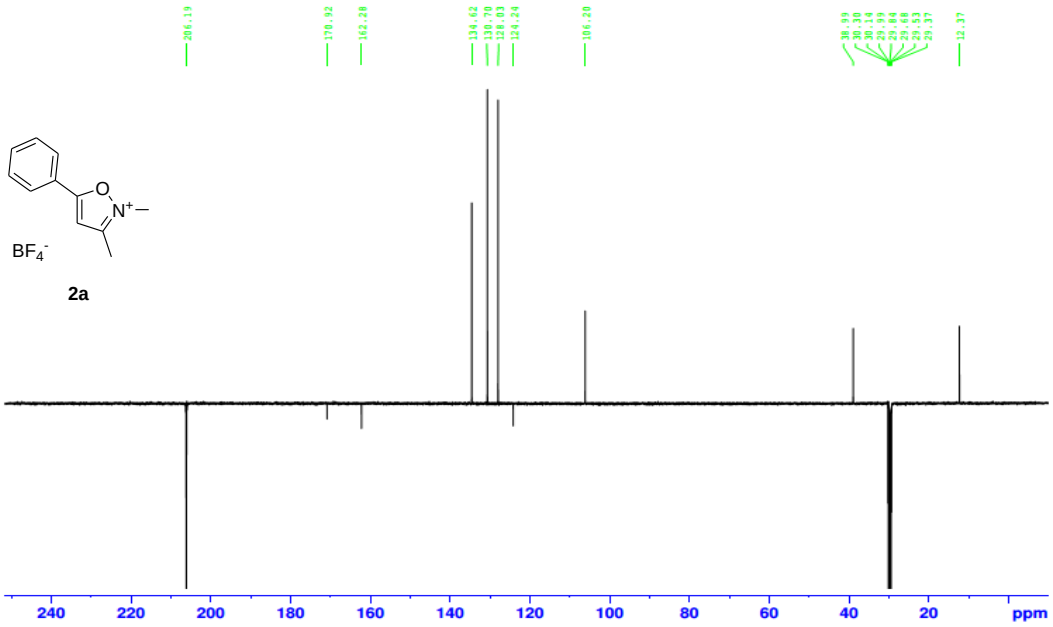


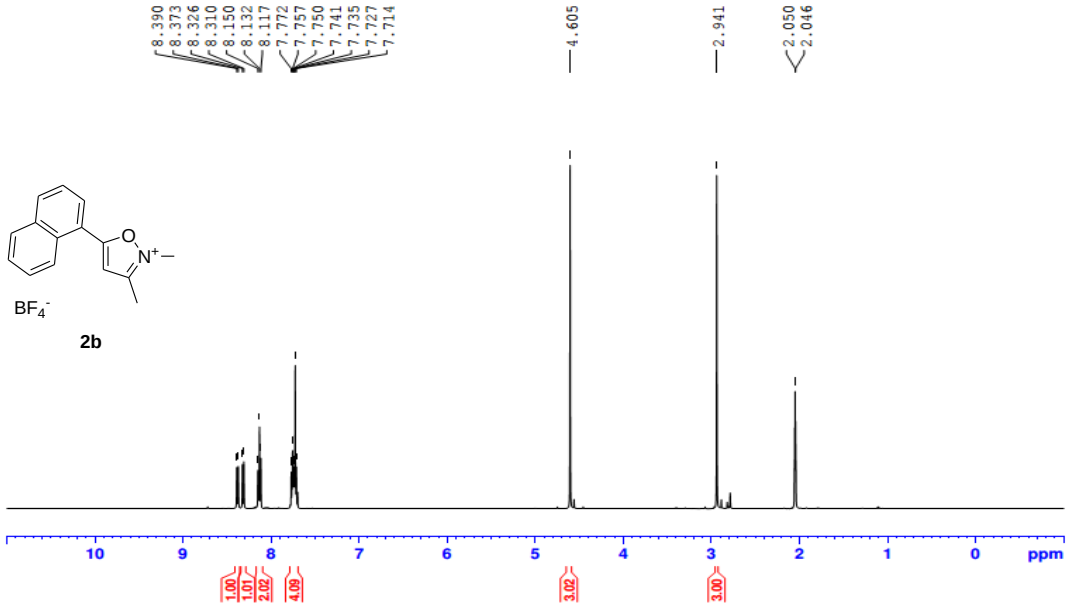
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\14_11_2017 [ATH 5% RR-Ph-Ms Cat in DCM] TS-NMe, Ar=Ph, R=Ph, N=Me, 4% IPA, AD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	7.360	2555.548	187.298	71.4	71.8	0.20	
2	8.880	1025.129	73.415	28.6	28.2	0.21	
	Total	3580.677	260.713	100.0	100.0		

III. NMR Spectra of 2 and 3



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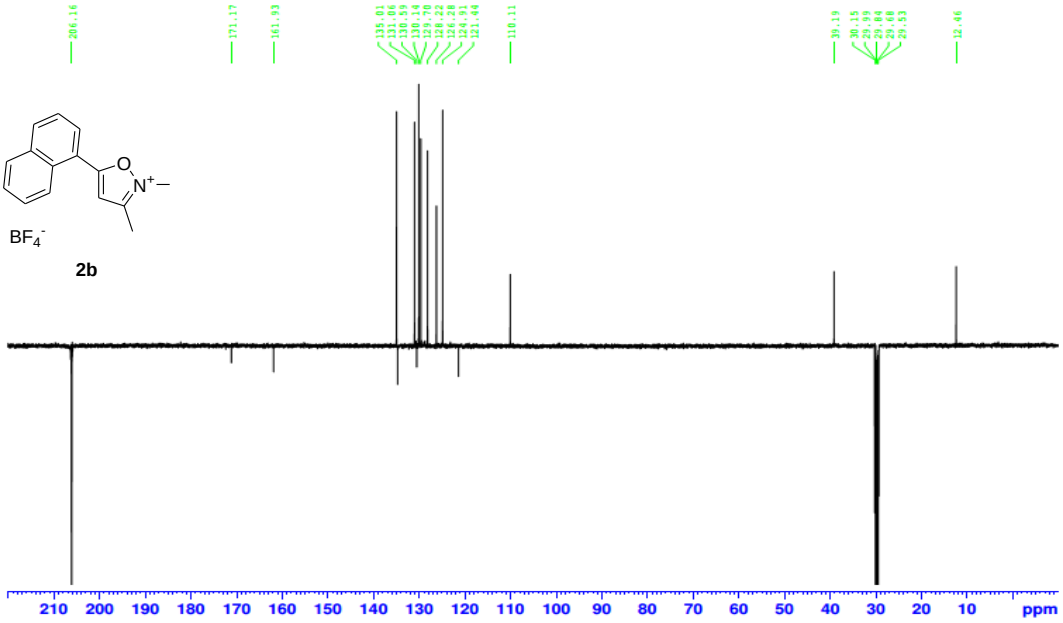
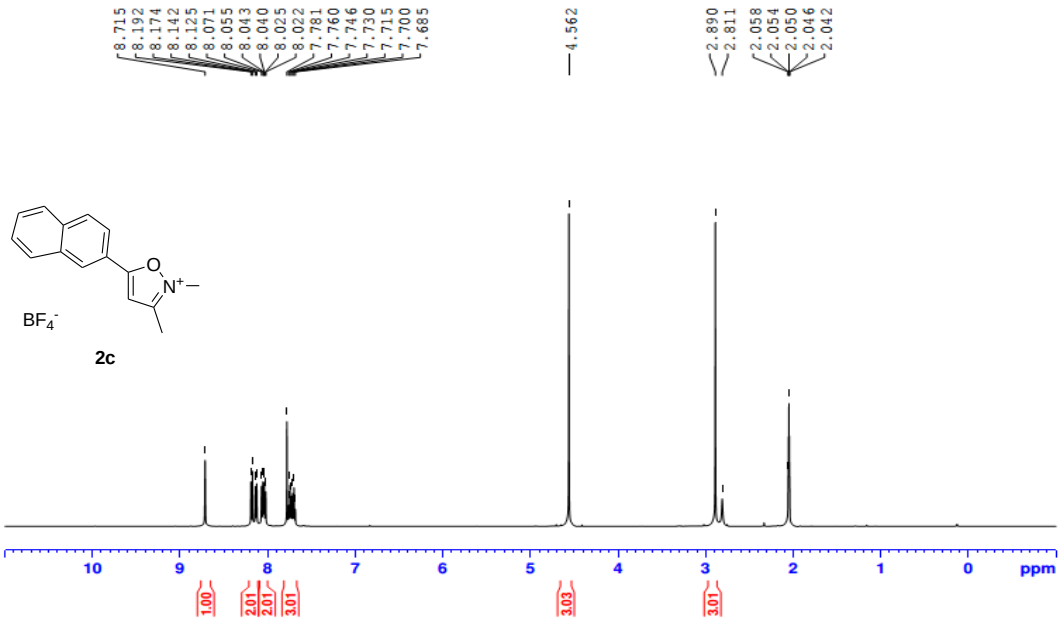
8.326
8.310
8.150
8.132
8.117
7.772
7.757
7.750
7.741
7.735
7.727
7.714
4.605
2.941
2.050
2.046
BF4-
2b
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.01
2.02
4.09
3.02
3.00


206.16
171.17
161.93
135.01
131.06
130.59
130.14
129.79
128.22
126.28
124.91
121.44
110.11
39.19
30.15
29.99
29.84
29.68
29.53
12.46
BF4-
2b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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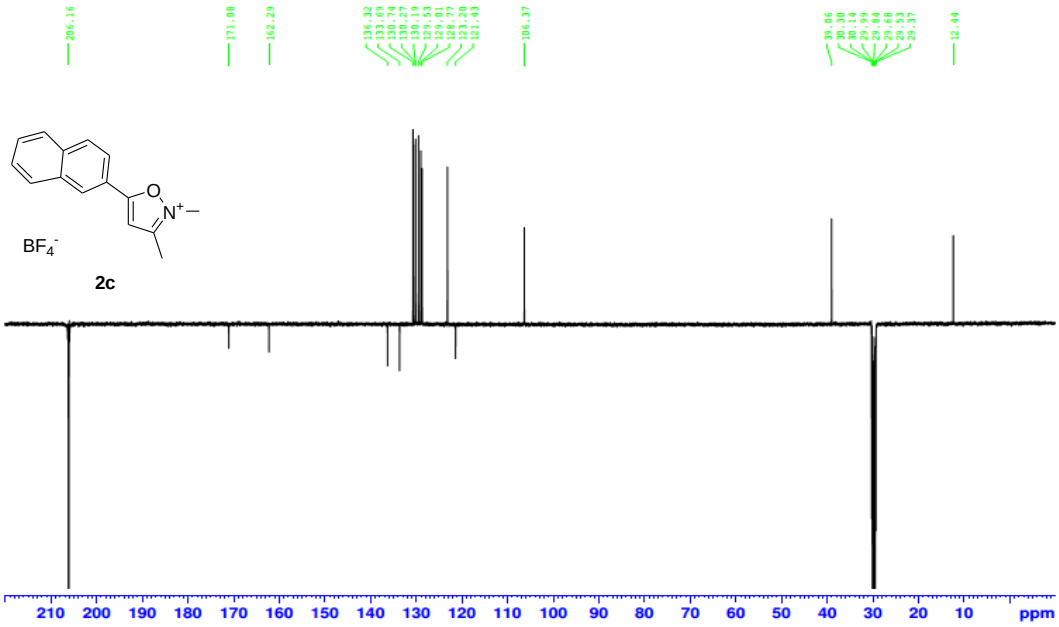
8.192
8.174
8.142
8.125
8.071
8.055
8.043
8.040
8.025
8.022
7.781
7.760
7.746
7.730
7.715
7.700
7.685
4.562
2.890
2.811
2.058
2.054
2.050
2.046
2.042
BF4-
2c
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.01
2.01
3.01
3.03
3.01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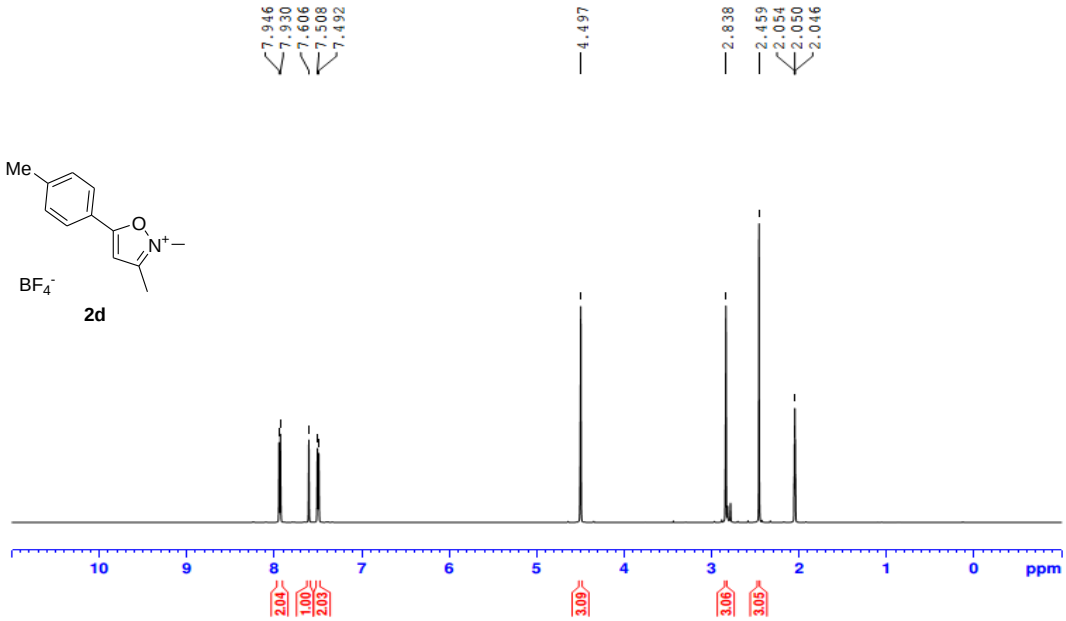
171.08
162.29
136.32
133.69
130.74
130.27
130.13
129.53
129.01
128.77
123.20
121.43
106.37
39.06
30.30
30.14
29.99
29.84
29.68
29.53
29.37
12.44
BF4-
2c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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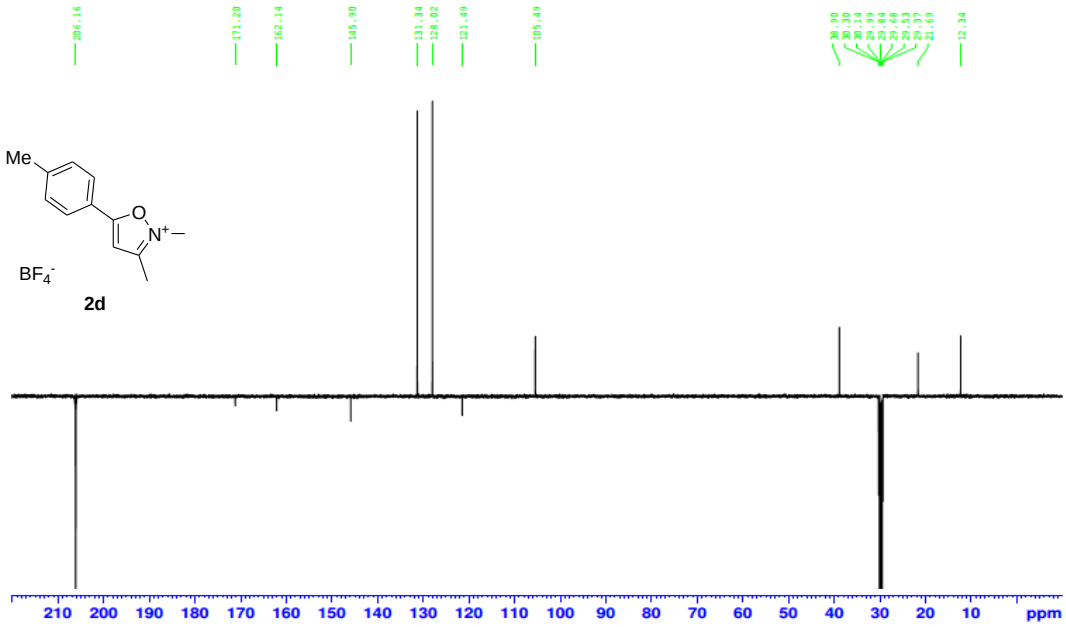
7.930
7.606
7.508
7.492
4.497
2.838
2.459
2.054
2.050
2.046
Me
O
N+
BF4-
2d
10
9
8
7
6
5
4
3
2
1
0
ppm
2.04
1.00
2.03
3.09
3.06
3.05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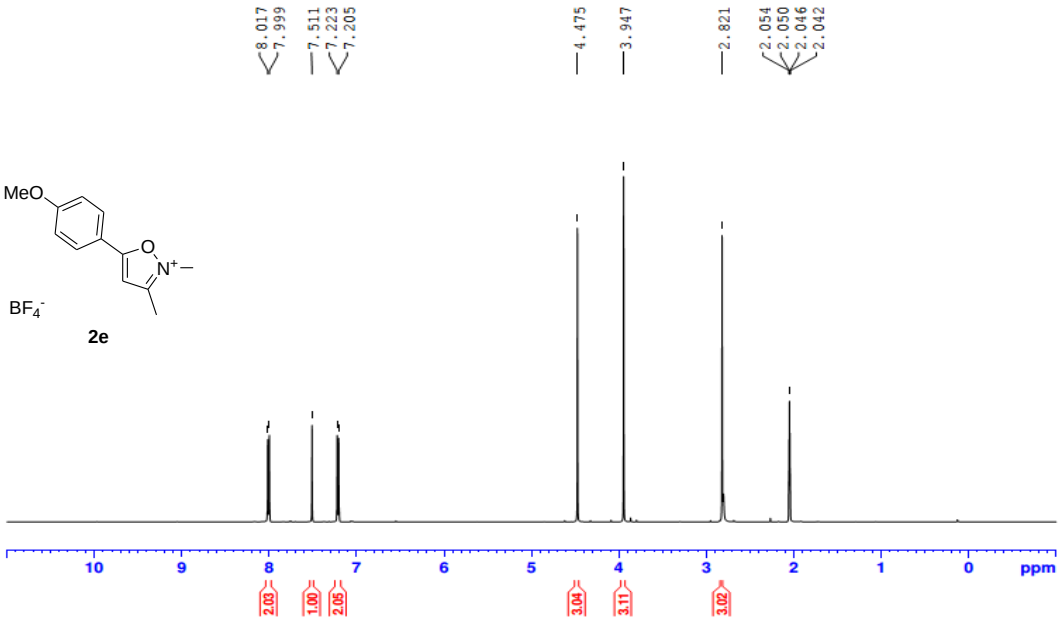
145.90
131.34
128.02
121.49
105.49
38.90
30.30
30.14
29.99
29.84
29.68
29.53
29.37
21.69
12.34
Me
O
N+
BF4-
2d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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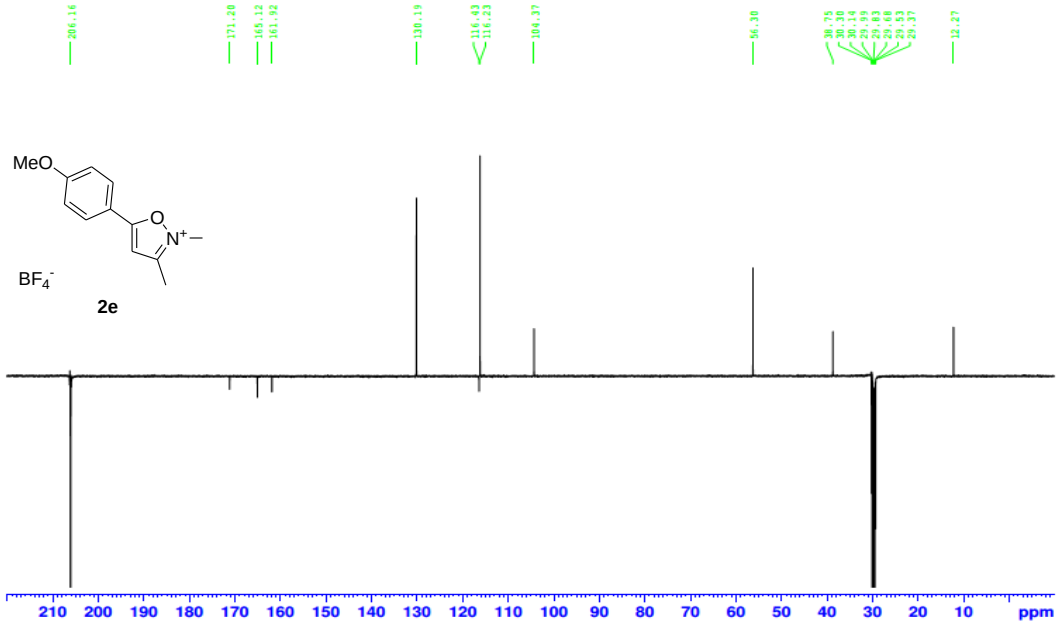
7.511
7.223
7.205
4.475
3.947
2.821
2.054
2.050
2.046
2.042
MeO
O
N+
BF4-
2e
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
1.00
2.05
3.04
3.11
3.02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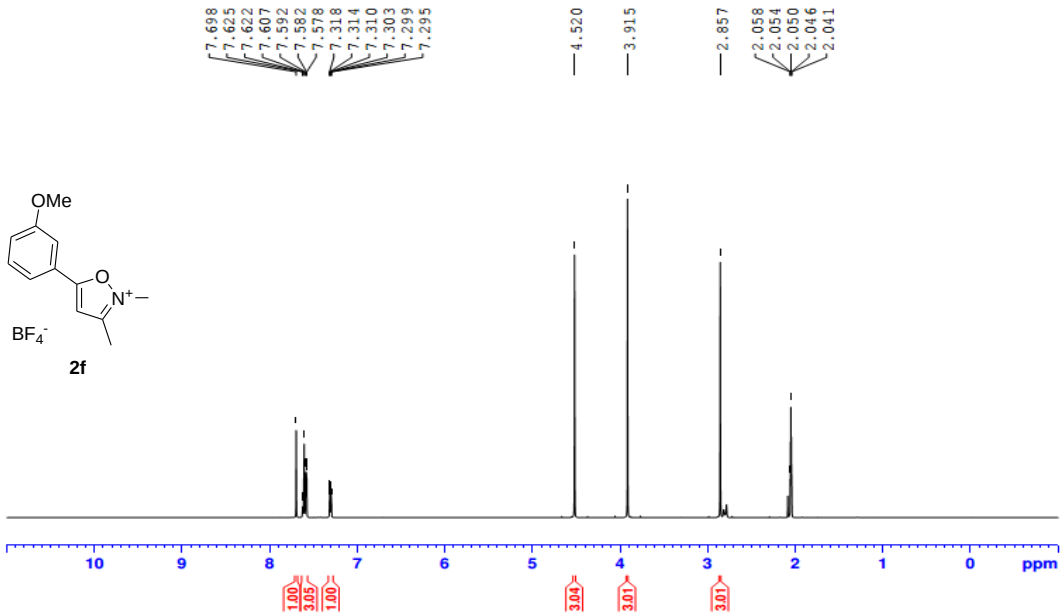
165.12
161.92
130.19
116.43
116.23
104.37
56.30
30.75
30.30
30.14
29.99
29.83
29.68
29.53
29.37
12.27
MeO
O
N+
BF4-
2e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.625
7.622
7.607
7.592
7.582
7.578
7.318
7.314
7.310
7.303
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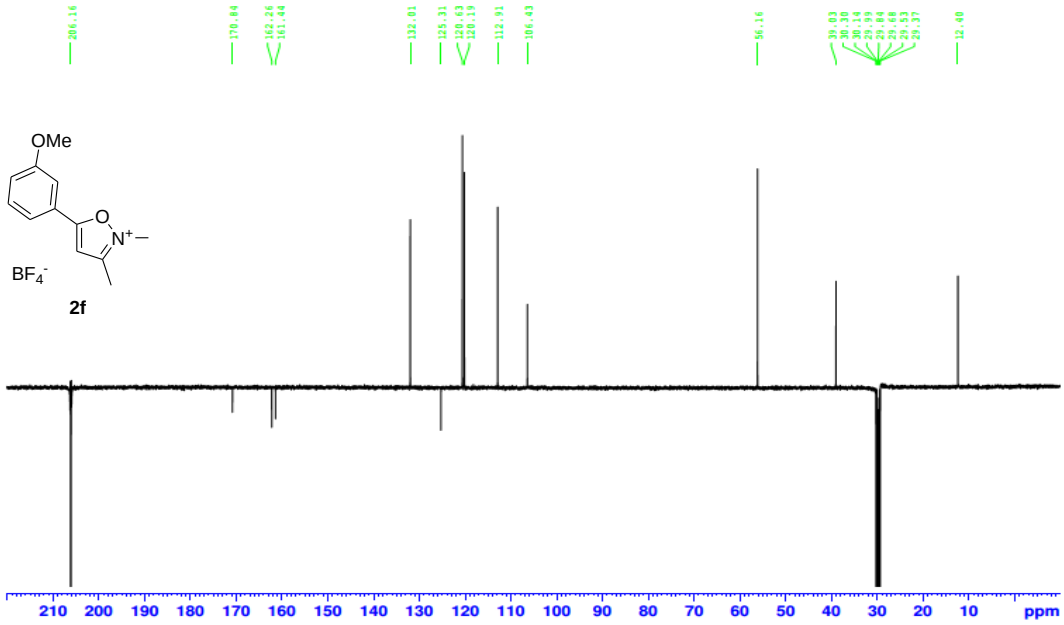
7.295
4.520
3.915
2.857
2.058
2.054
2.050
2.046
2.041
OMe
O
N+
BF4-
2f
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
3.05
1.00
3.04
3.01
3.01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161.44
132.01
125.31
120.63
120.19
112.91
106.43
56.16
39.83
30.30
30.14
29.99
29.64
29.48
29.53
29.37
12.40
OMe
O
N+
BF4-
2f
210
200
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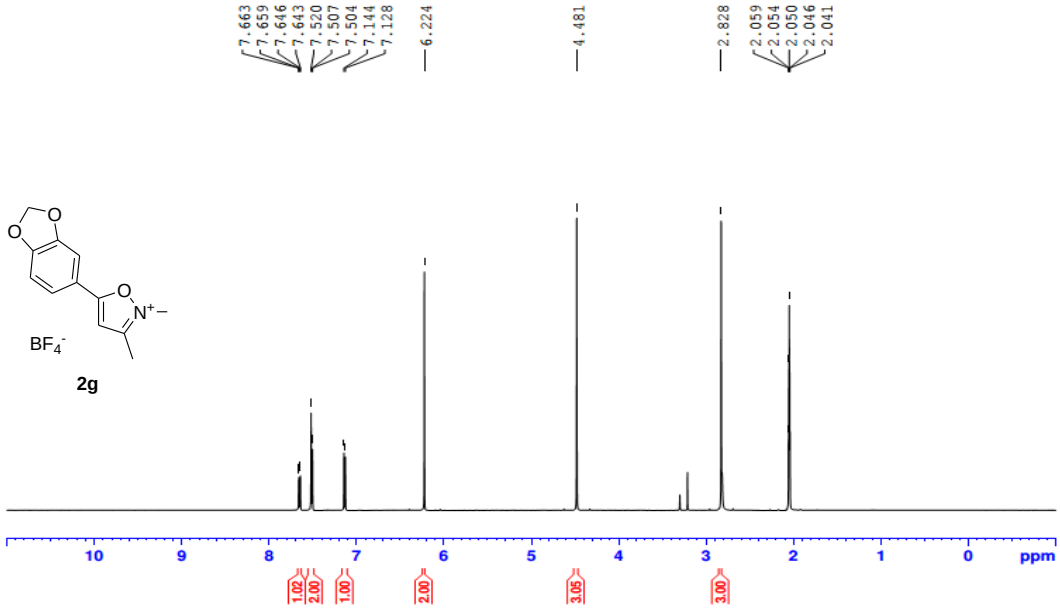
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.643
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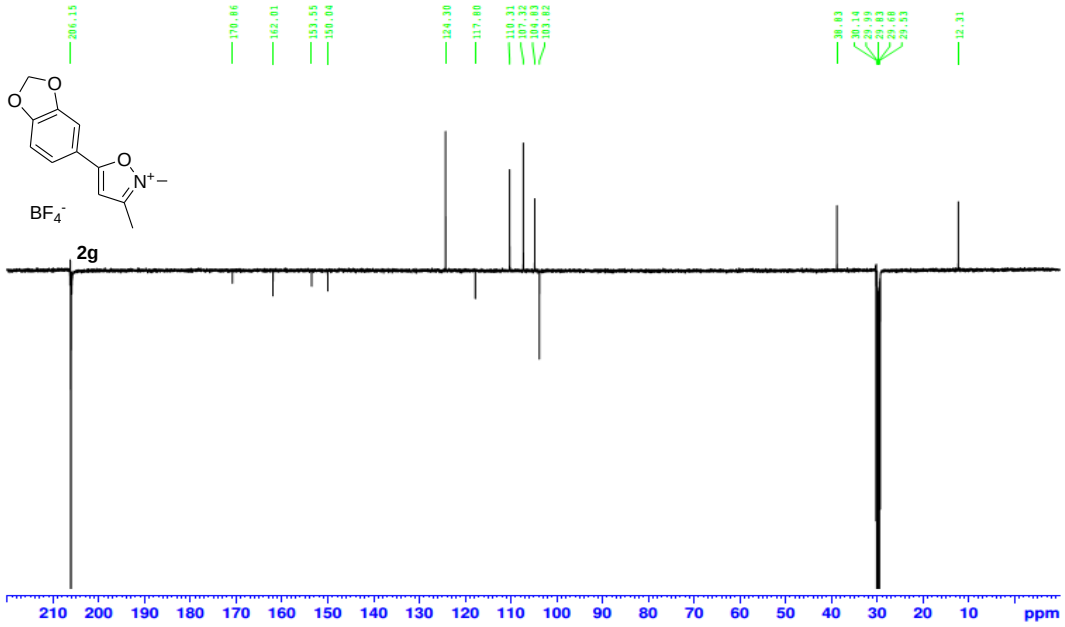
7.507
7.504
7.144
7.128
6.224
4.481
2.828
2.059
2.054
2.050
2.046
2.041
BF4-
2g
1.02
2.00
1.00
2.00
3.05
3.00
ppm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110.31
107.32
104.83
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30.03
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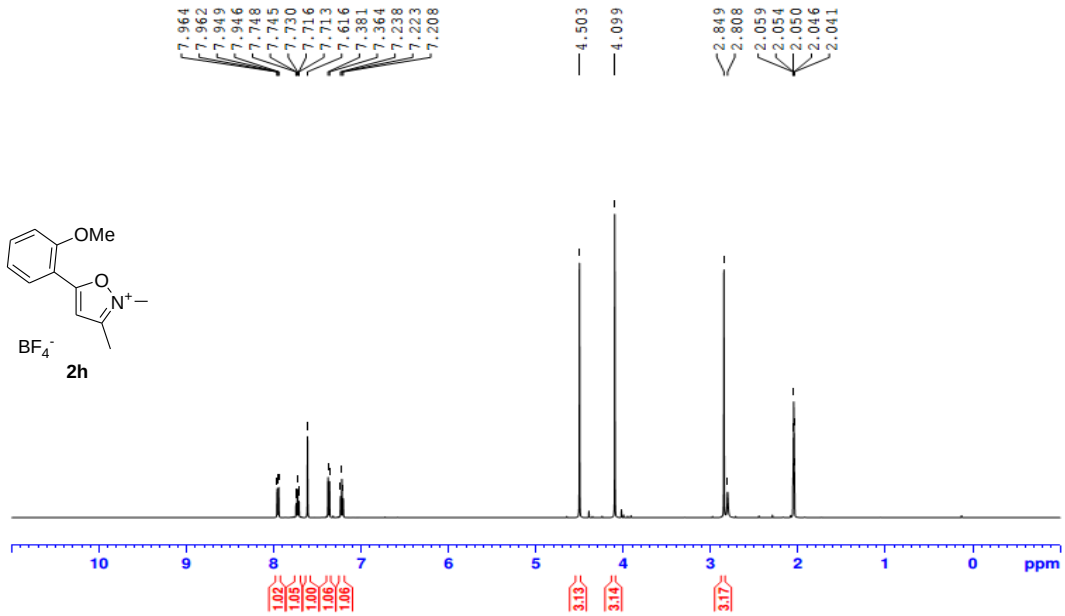
29.68
29.53
12.31
BF4-
2g
ppm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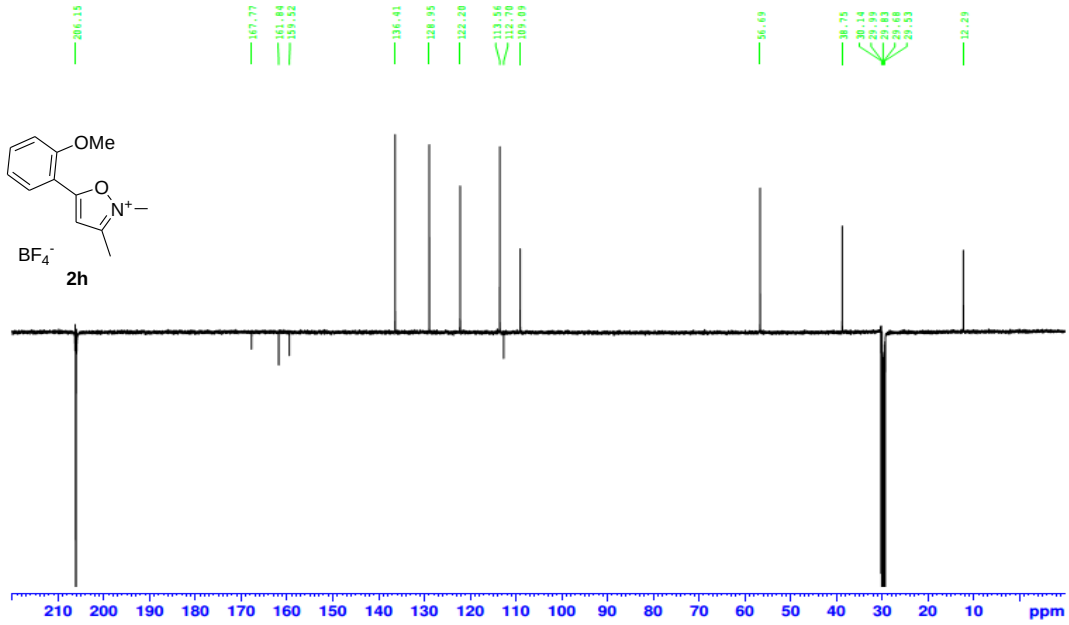
7.946
7.748
7.745
7.730
7.716
7.713
7.616
7.381
7.364
7.238
7.223
7.208
4.503
4.099
2.849
2.808
2.059
2.054
2.050
2.046
2.041
OMe
O
N+
BF4-
2h
10
9
8
7
6
5
4
3
2
1
0
ppm
1.02
1.05
1.00
1.06
1.06
3.13
3.14
3.17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159.52
136.41
128.95
122.20
113.56
112.70
109.09
56.69
38.75
30.14
29.99
29.93
29.83
29.68
29.53
12.29
OMe
O
N+
BF4-
2h
210
200
190
180
170
160
150
140
130
120
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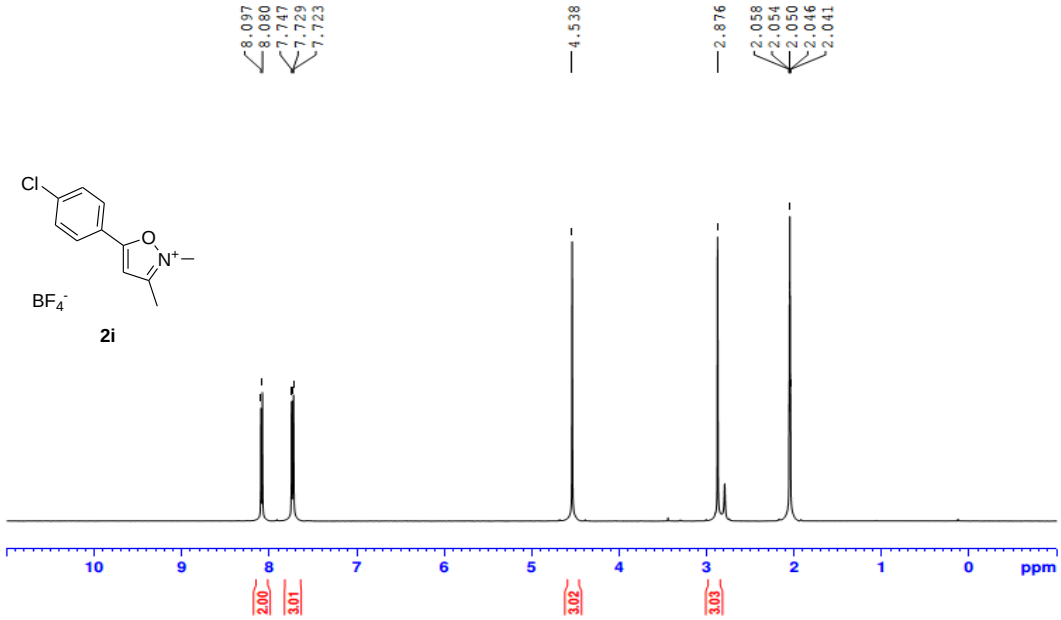
90
80
70
60
50
40
30
20
10
ppm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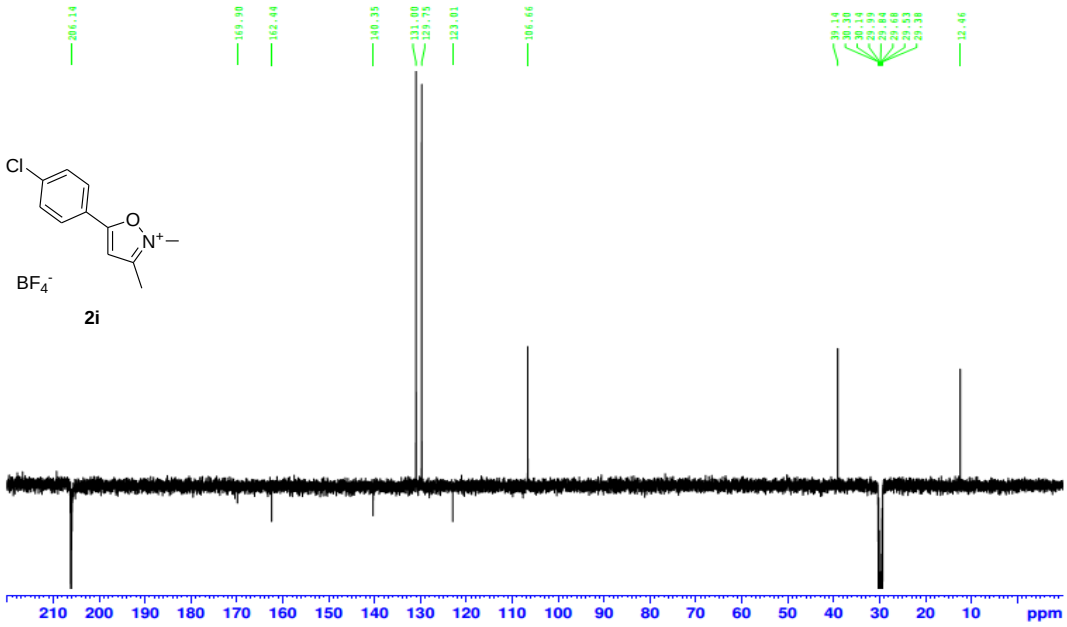
7.729
7.723
4.538
2.876
2.058
2.054
2.050
2.046
2.041
Cl
O
N+
BF4-
2i
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
3.01
3.02
3.03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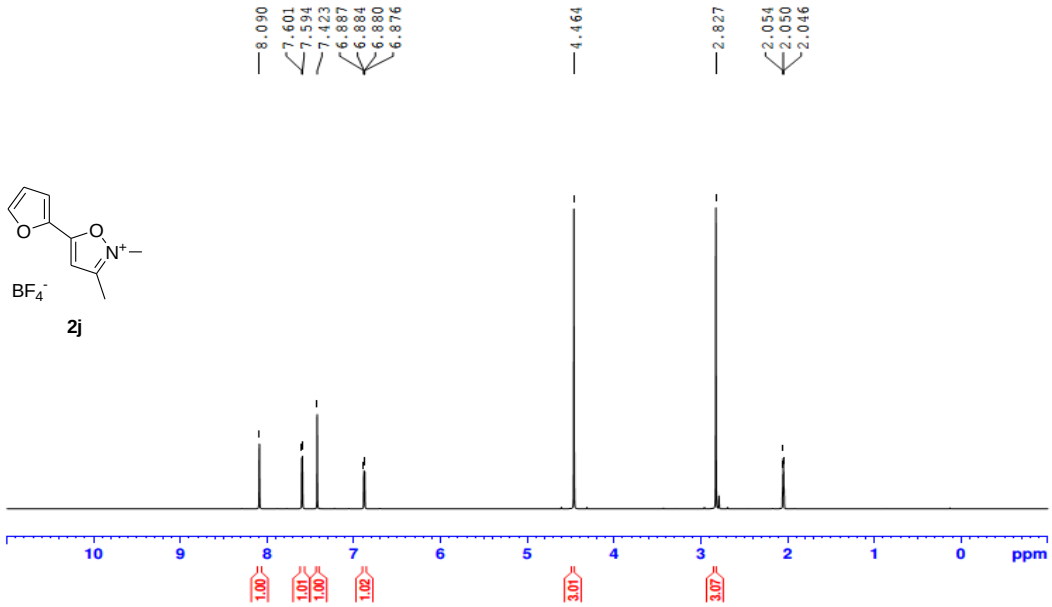
140.35
131.00
129.75
123.01
106.66
39.14
30.30
30.14
29.99
29.84
29.69
29.54
29.39
12.46
Cl
O
N+
BF4-
2i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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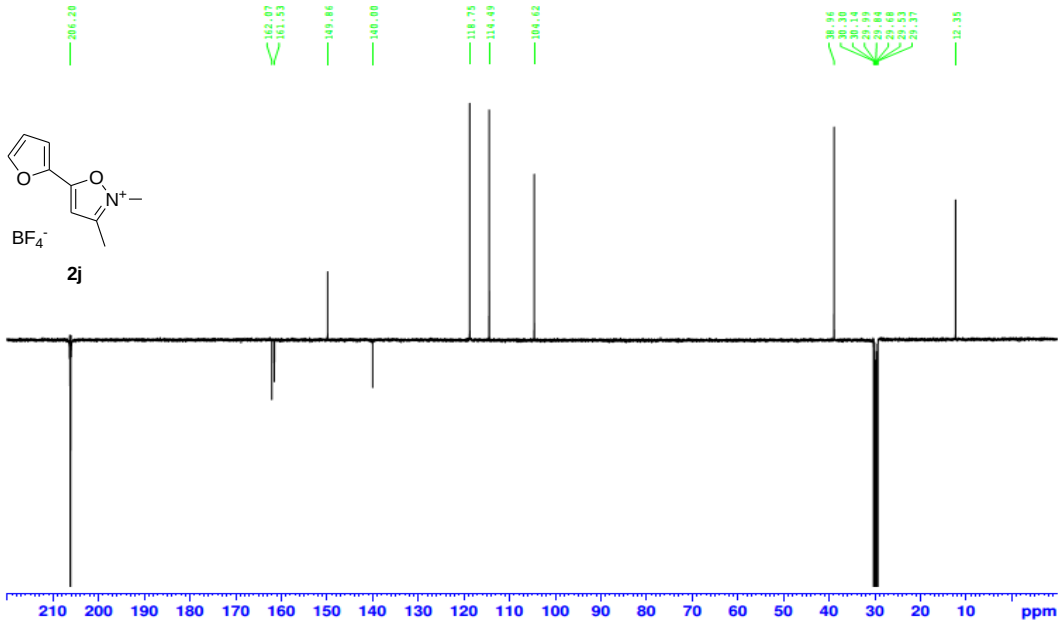
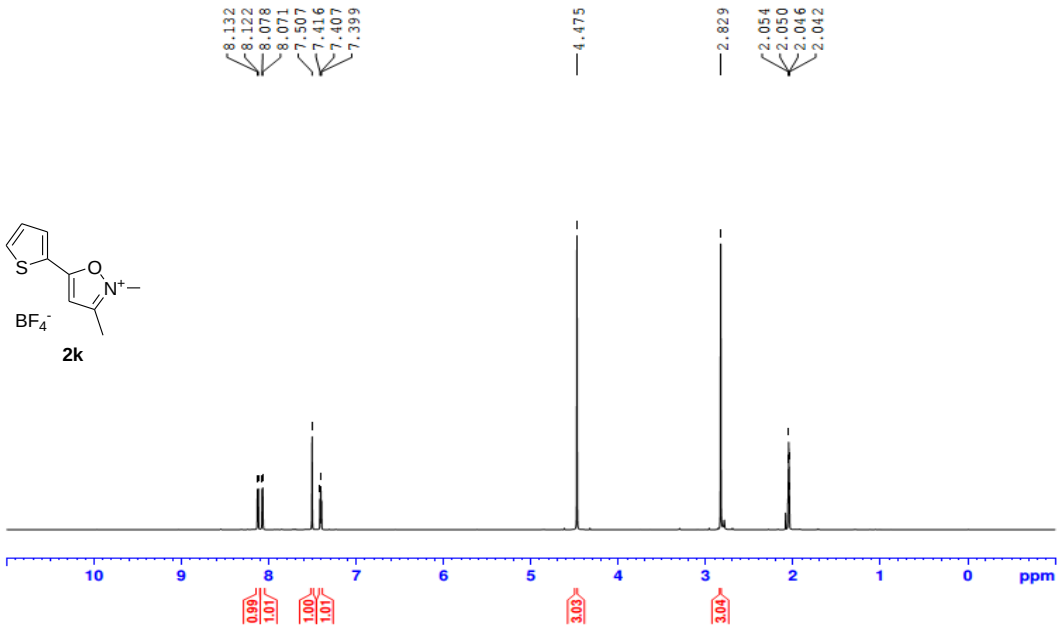
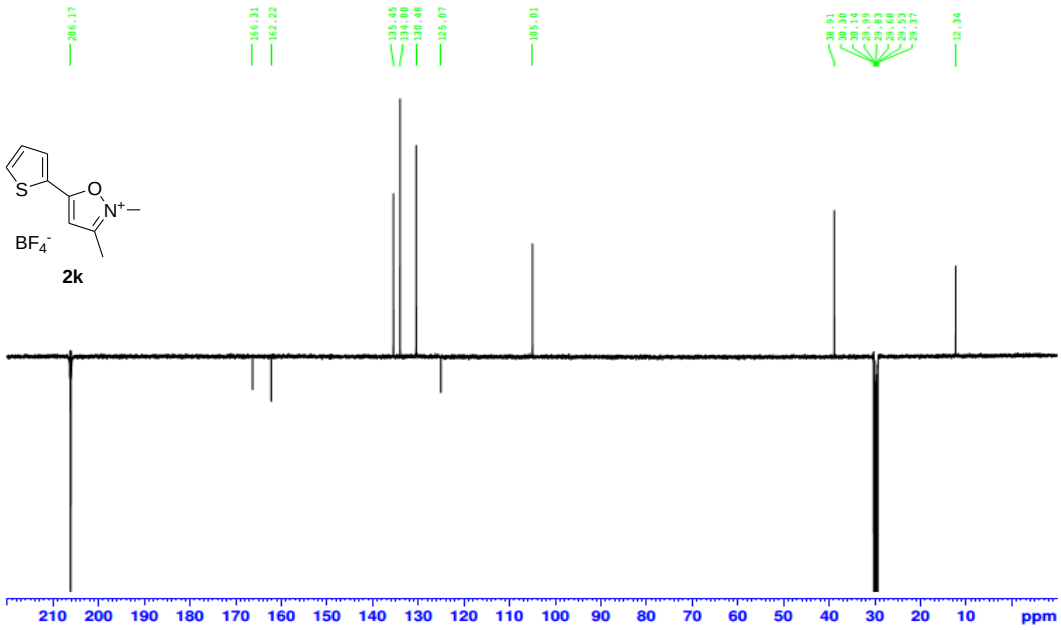
7.601
7.594
7.423
6.887
6.884
6.880
6.876
4.464
2.827
2.054
2.050
2.046
BF4-
2j
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.01
1.00
1.02
3.01
3.07


206.20
162.07
161.53
149.86
140.00
118.75
114.49
104.62
38.96
30.30
30.14
29.99
29.86
29.68
29.53
29.37
12.35
BF4-
2j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


8.132
8.122
8.078
8.071
7.507
7.416
7.407
7.399
4.475
2.829
2.054
2.050
2.046
2.042
S
O
N+
BF4-
2k
10
9
8
7
6
5
4
3
2
1
0
ppm
0.99
1.01
1.00
1.01
3.03
3.04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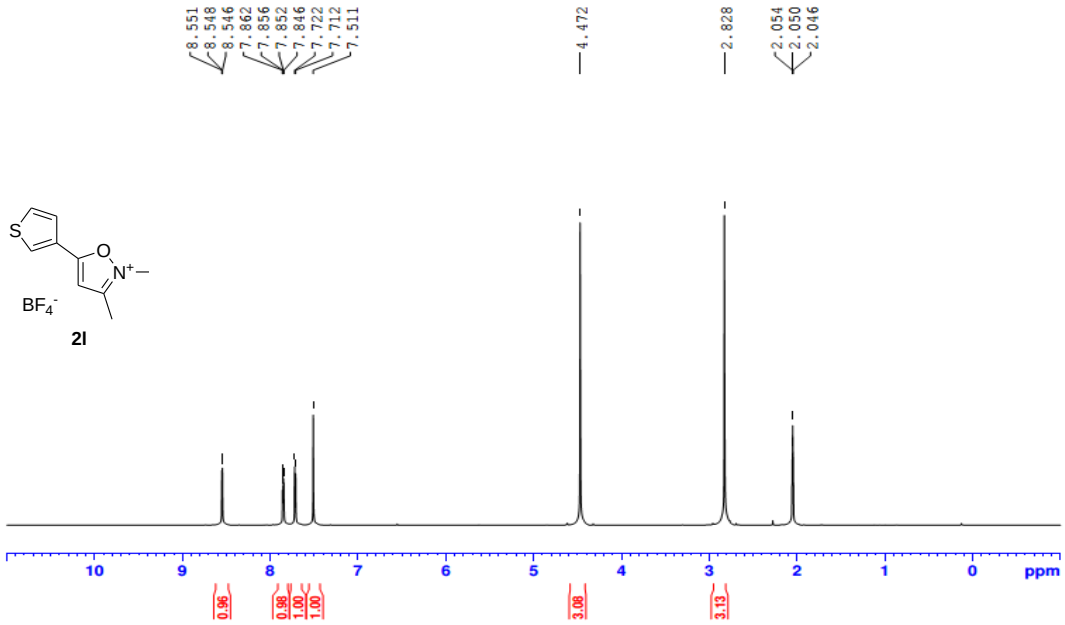
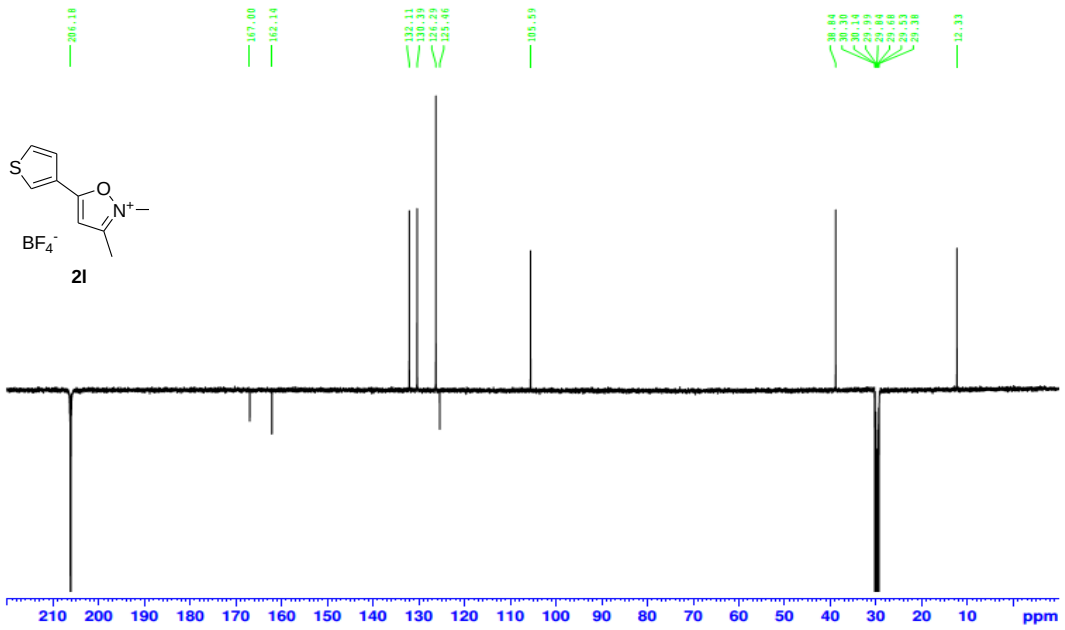
166.31
162.22
135.45
134.00
130.48
125.07
105.01
38.91
30.30
30.14
29.99
29.83
29.68
29.53
29.37
12.34
S
O
N+
BF4-
2k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.551
8.548
8.546
7.862
7.856
7.852
7.846
7.722
7.712
7.511
4.472
2.828
2.054
2.050
2.046
S
O
N+
BF4-
2l
10
9
8
7
6
5
4
3
2
1
0
ppm
0.96
0.98
1.00
1.00
3.08
3.13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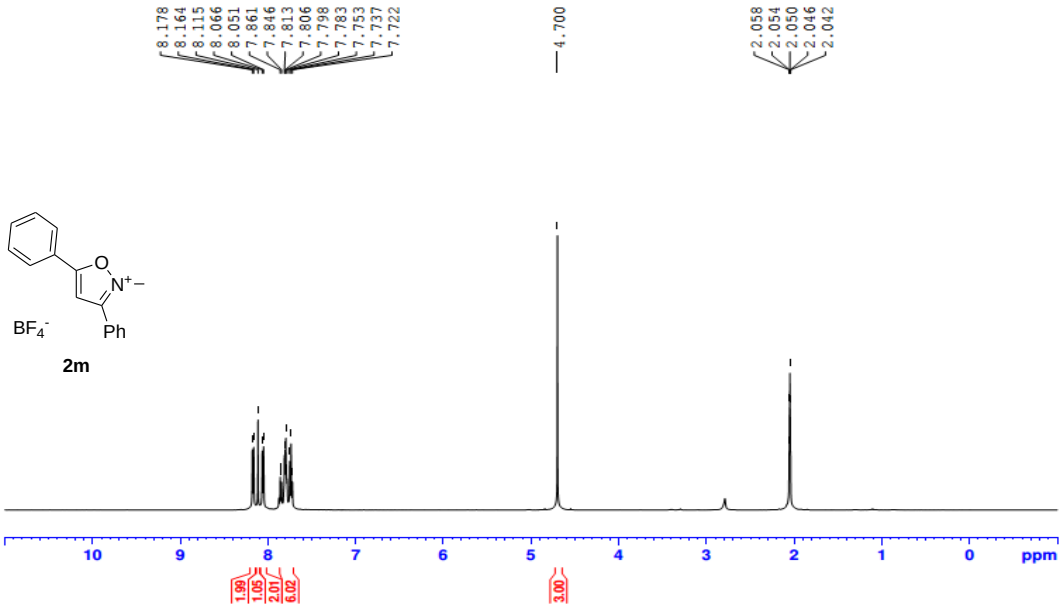
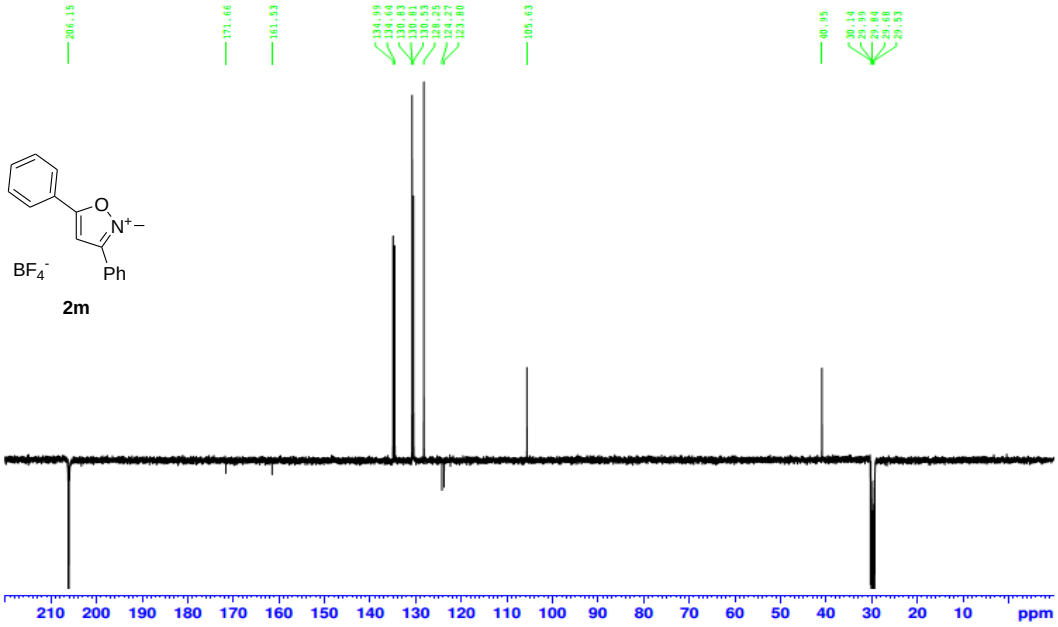
167.00
162.14
132.11
130.39
126.29
125.46
105.59
38.84
30.30
30.14
29.99
29.84
29.66
29.53
29.38
12.33
S
O
N+
BF4-
2l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.178
8.164
8.115
8.066
8.051
7.861
7.846
7.813
7.806
7.798
7.783
7.753
7.737
7.722
4.700
2.058
2.054
2.050
2.046
2.042
BF4-
Ph
2m
10
9
8
7
6
5
4
3
2
1
0
ppm
1.99
1.05
2.01
6.02
3.00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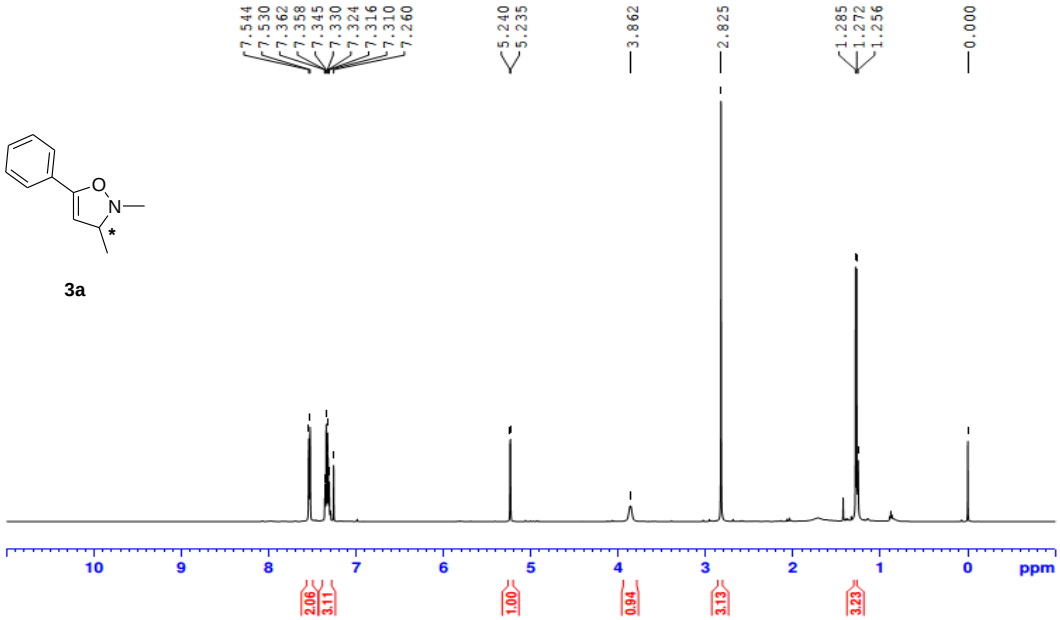
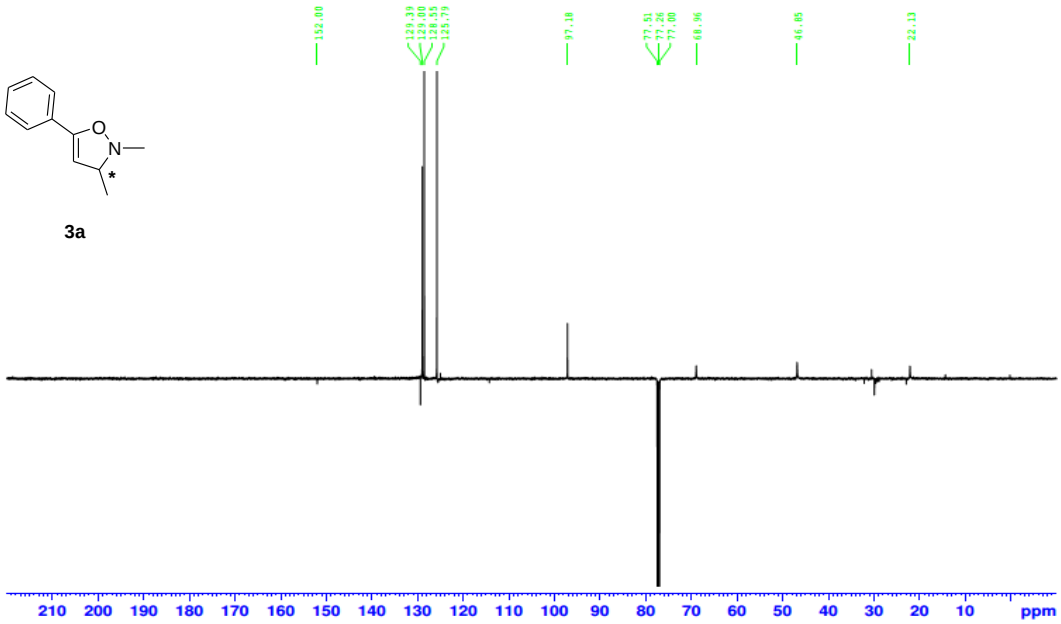
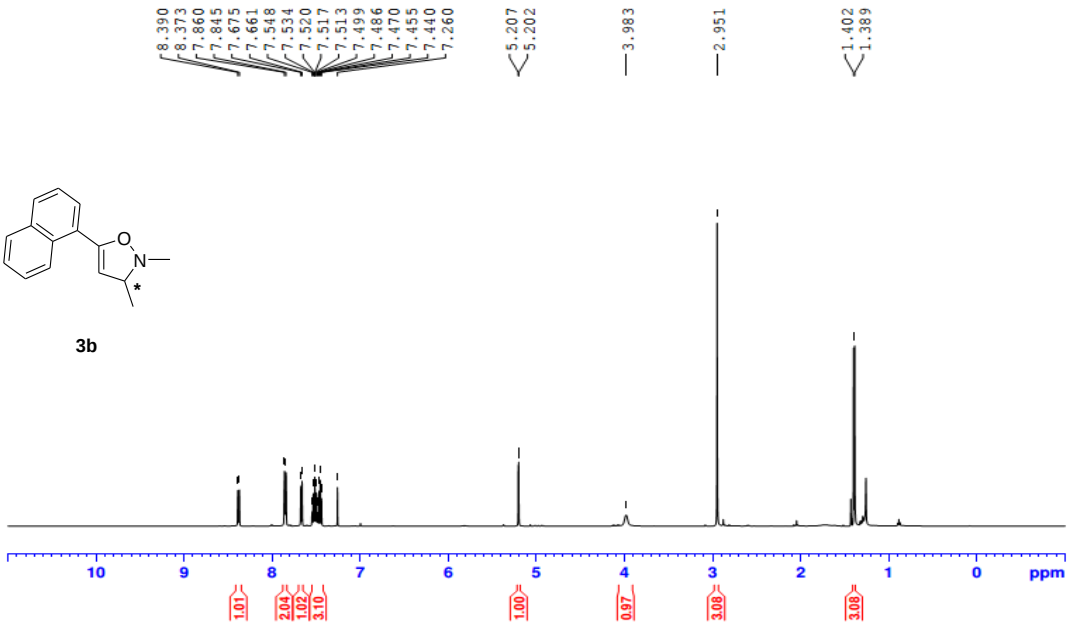
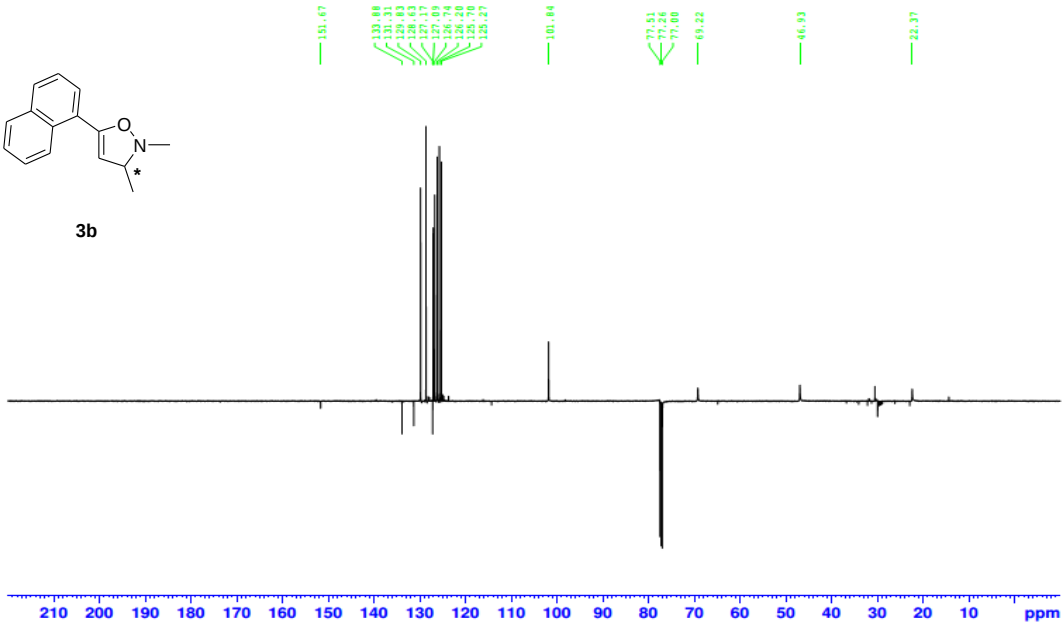
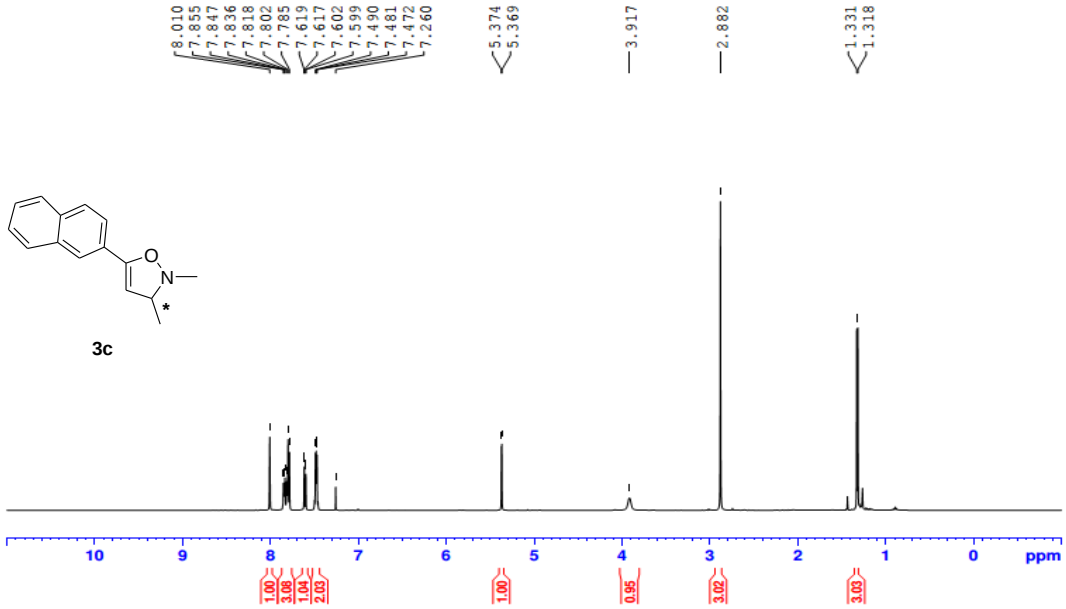
171.66
161.53
134.99
134.64
130.83
130.81
130.53
128.25
124.27
123.80
105.63
40.95
30.14
29.99
29.84
29.68
29.53
BF4-
Ph
2m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

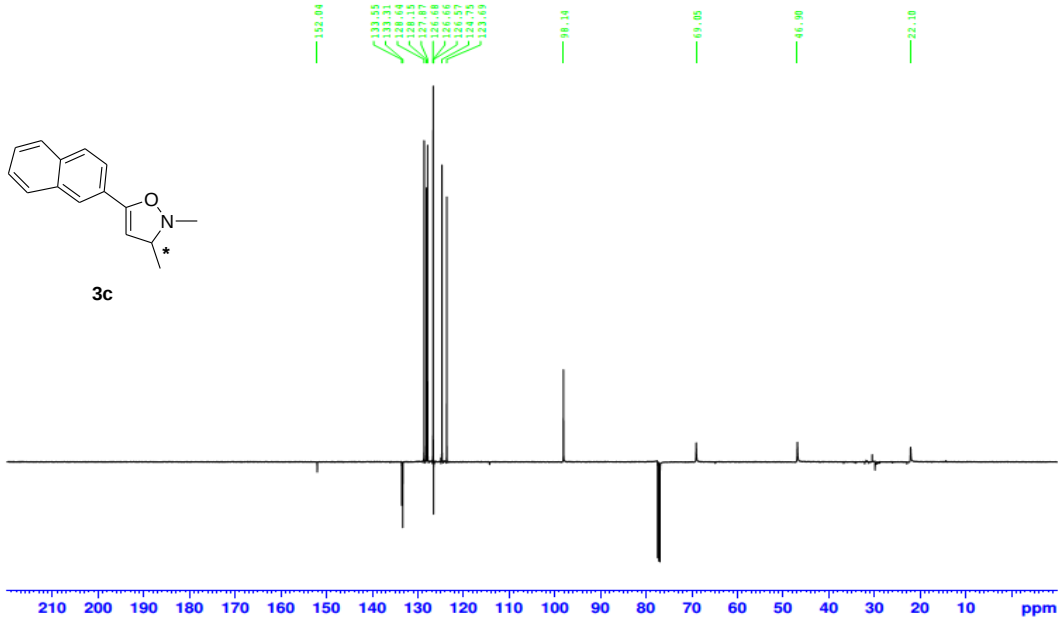

7.544
7.530
7.362
7.358
7.345
7.330
7.324
7.316
7.310
7.260
5.240
5.235
3.862
2.825
1.285
1.272
1.256
0.000
O
N
*
3a
10
9
8
7
6
5
4
3
2
1
0
ppm
2.06
3.11
1.00
0.94
3.13
3.23

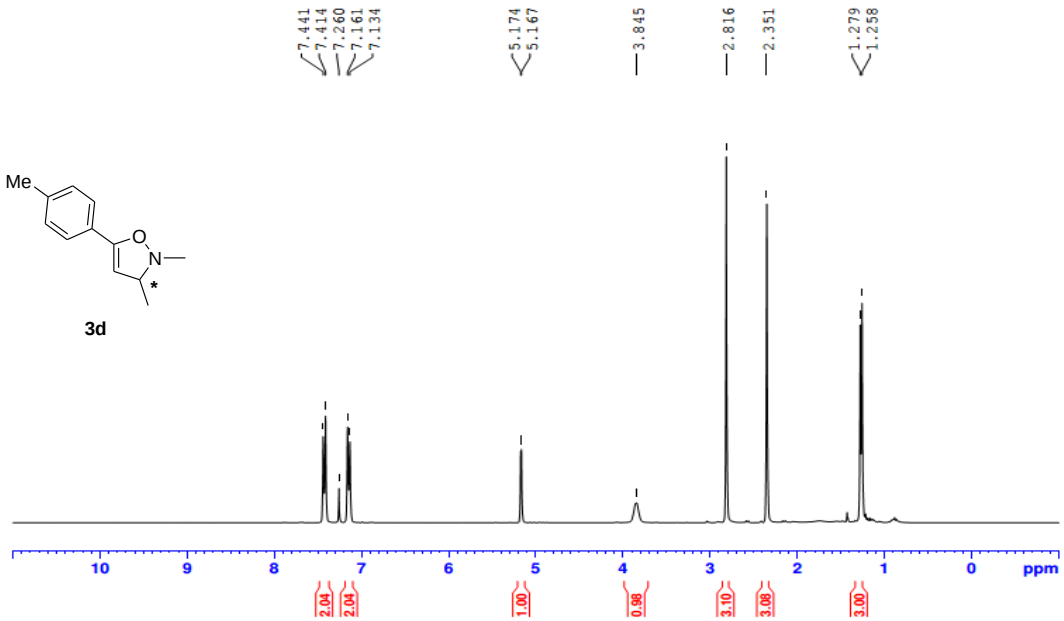

152.00
129.39
129.00
128.55
125.79
97.18
77.51
77.26
77.00
68.36
46.85
22.13
O
N
*
3a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

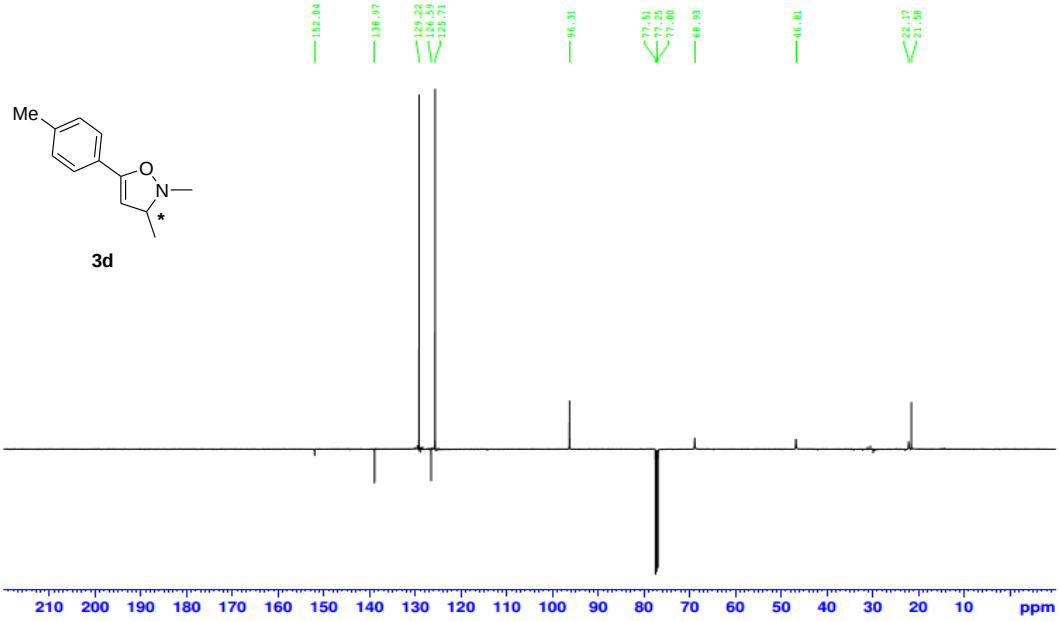

8.390
8.373
7.860
7.845
7.675
7.661
7.548
7.534
7.520
7.517
7.513
7.499
7.486
7.470
7.455
7.440
7.260
5.207
5.202
3.983
2.951
1.402
1.389
O
N
*
3b
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.04
1.02
3.10
1.00
0.97
3.08
3.08

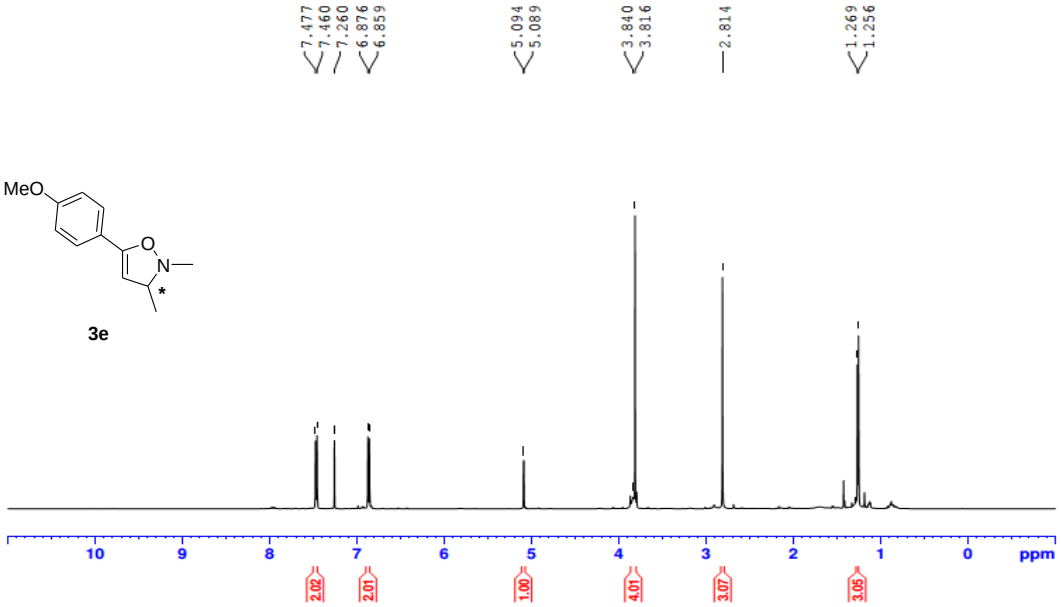

151.67
133.88
131.31
129.83
128.63
127.17
127.09
126.74
126.20
125.70
125.27
101.84
77.51
77.26
77.00
69.22
46.93
22.37
O
N
*
3b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

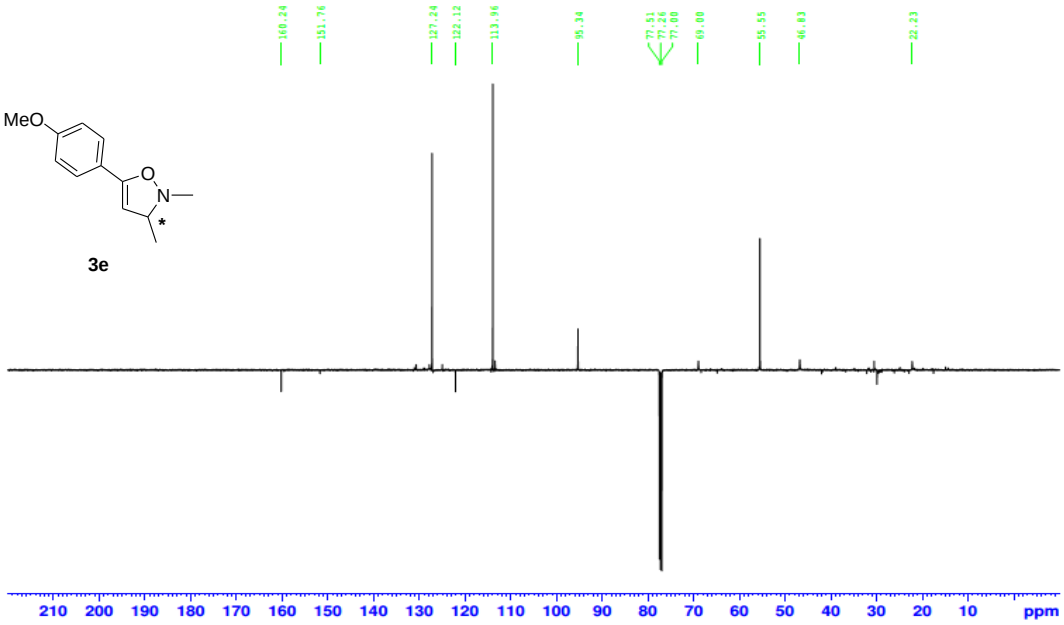

8.010
7.855
7.847
7.836
7.818
7.802
7.785
7.619
7.617
7.602
7.599
7.490
7.481
7.472
7.260
5.374
5.369
3.917
2.882
1.331
1.318
O
N
*
3c
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
3.08
1.04
2.03
1.00
0.95
3.02
3.03

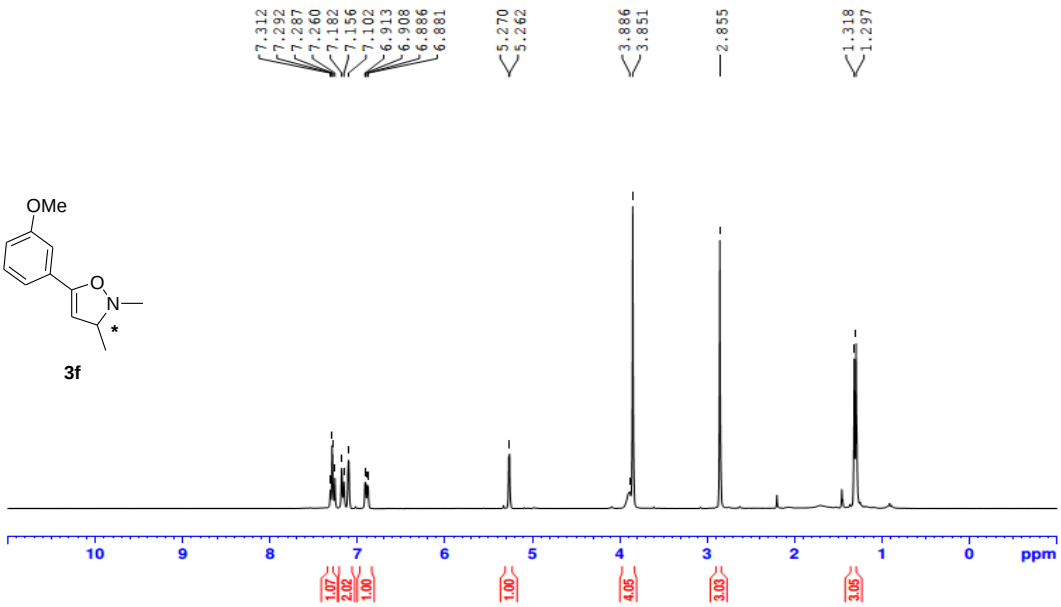

152.04
133.55
133.31
128.64
128.15
127.87
126.68
126.66
126.57
124.75
123.69
98.14
69.85
46.90
22.10
O
N
*
3c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

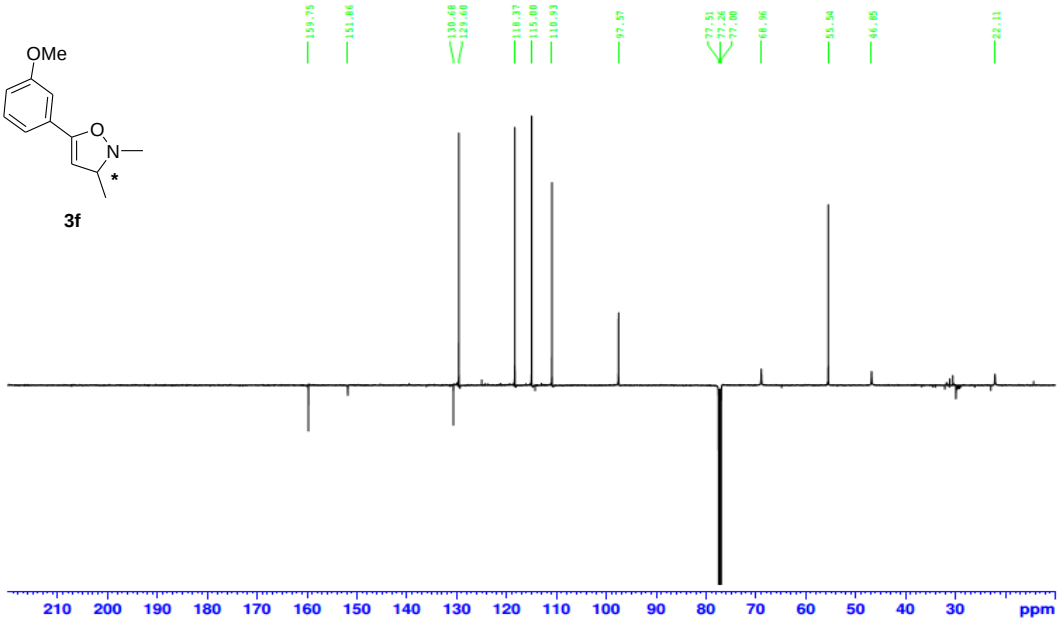

7.441
7.414
7.260
7.161
7.134
5.174
5.167
3.845
2.816
2.351
1.279
1.258
Me
O
N
*
3d
10
9
8
7
6
5
4
3
2
1
0
ppm
2.04
2.04
1.00
0.98
3.10
3.08
3.00

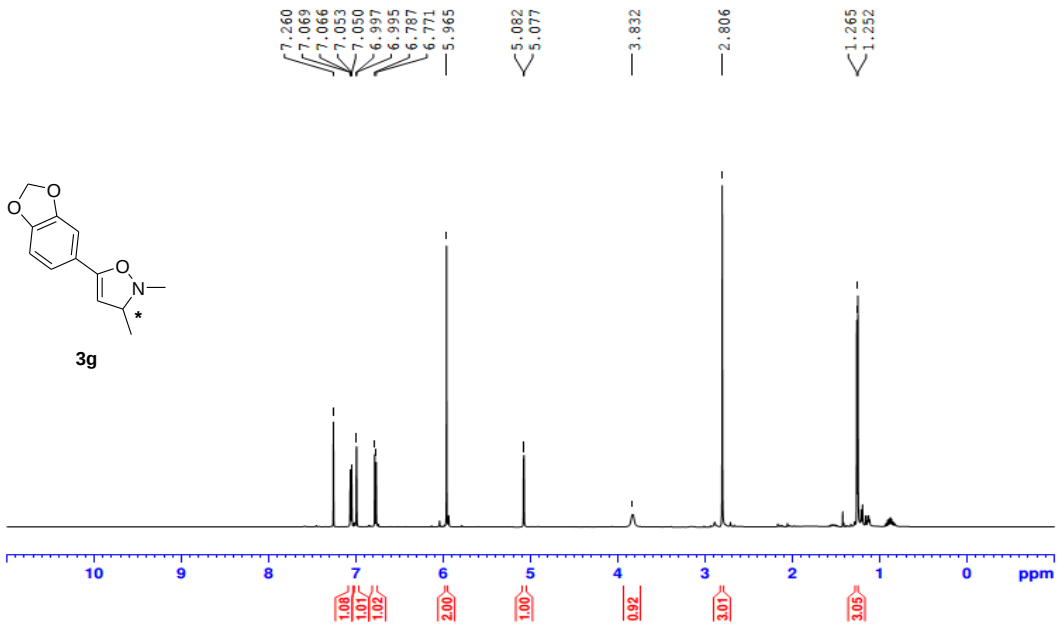

152.04
138.97
129.22
126.59
125.71
96.31
77.51
77.26
77.00
68.93
46.81
22.17
21.58
Me
O
N
*
3d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

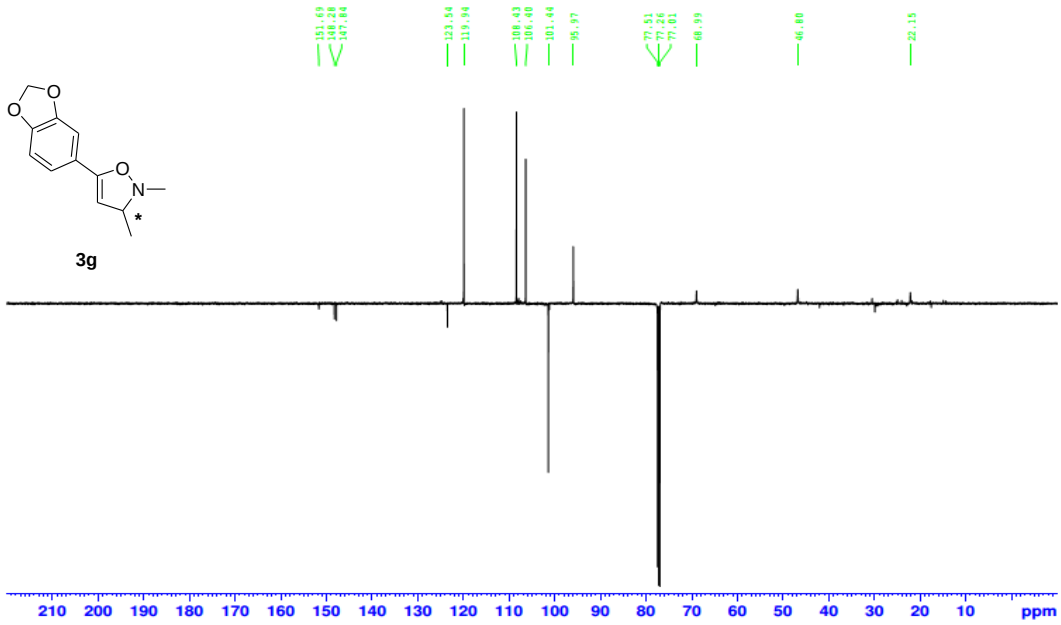

7.477
7.460
7.260
6.876
6.859
5.094
5.089
3.840
3.816
2.814
1.269
1.256
MeO
O
N
*
3e
10
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.01
1.00
4.01
3.07
3.05

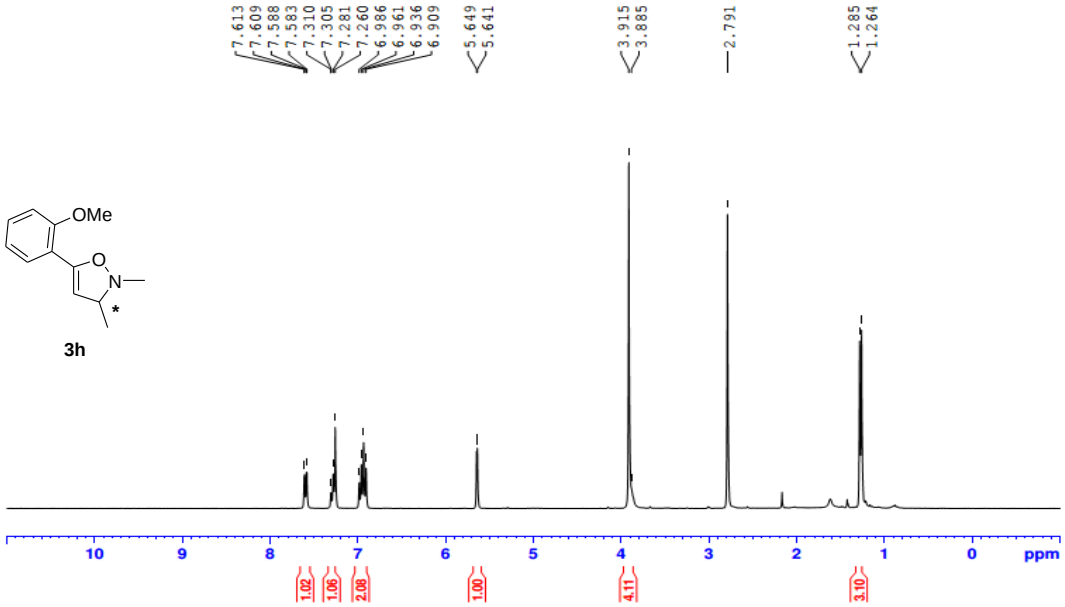

160.24
151.76
127.24
122.12
113.96
95.34
77.51
77.26
77.00
69.00
55.55
46.03
22.23
MeO
O
N
*
3e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

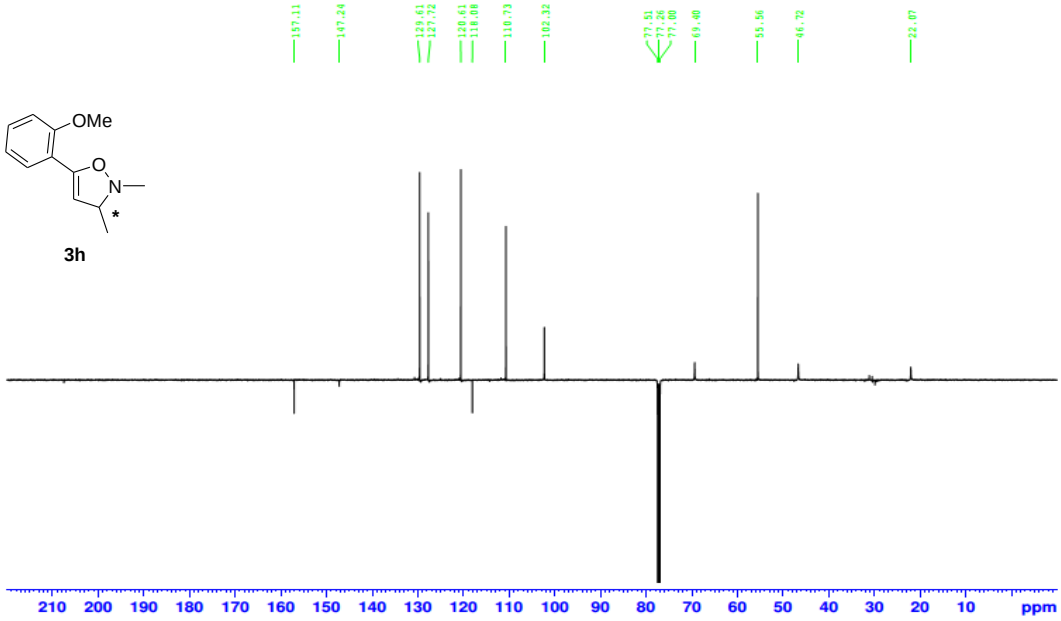

7.312
7.292
7.287
7.260
7.182
7.156
7.102
6.913
6.908
6.886
6.881
5.270
5.262
3.886
3.851
2.855
1.318
1.297
OMe
O
N
*
3f
10
9
8
7
6
5
4
3
2
1
0
ppm
1.07
2.02
1.00
1.00
4.05
3.03
3.05

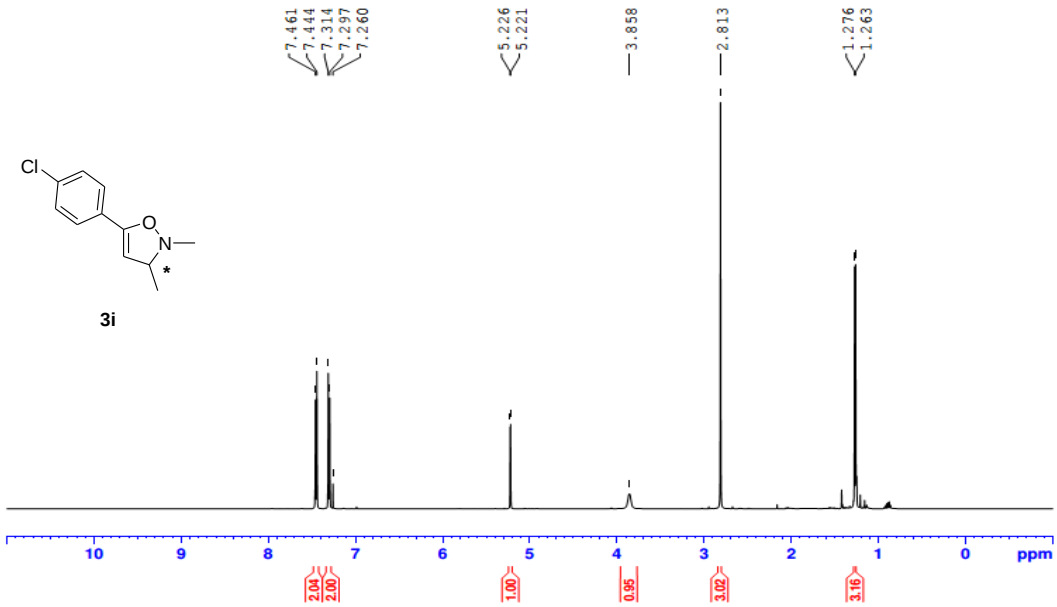

159.75
151.86
130.68
129.60
118.37
115.00
110.93
97.57
77.51
77.26
77.00
68.96
55.54
46.85
22.11
OMe
O
N
*
3f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

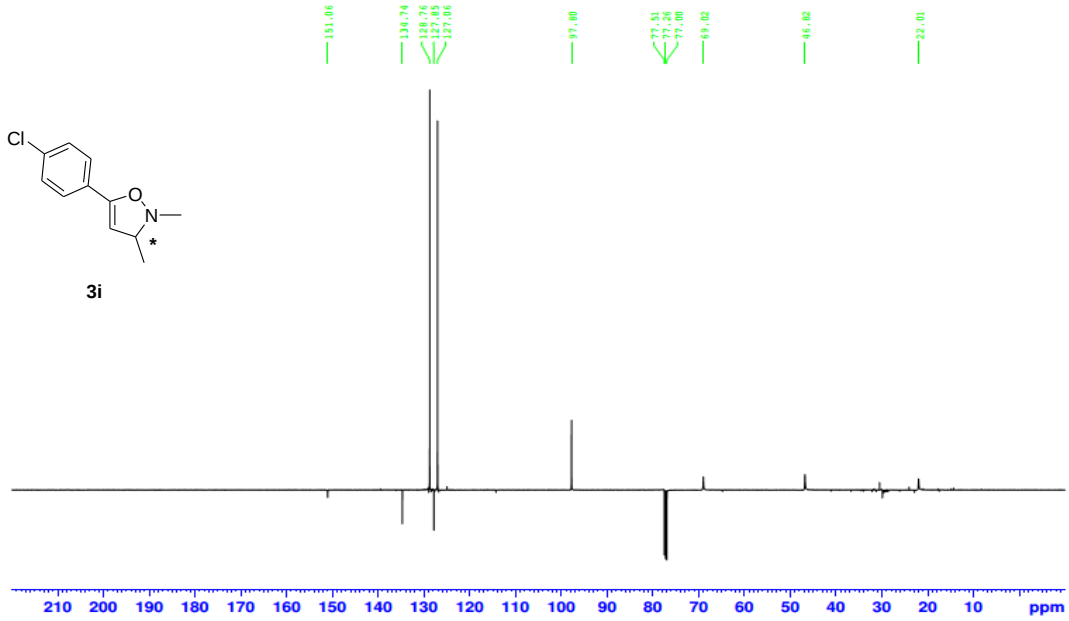

7.260
7.069
7.066
7.053
7.050
6.997
6.995
6.787
6.771
5.965
5.082
5.077
3.832
2.806
1.265
1.252
3g
10 9 8 7 6 5 4 3 2 1 0 ppm
1.08
1.01
1.02
2.00
1.00
0.92
3.01
3.05

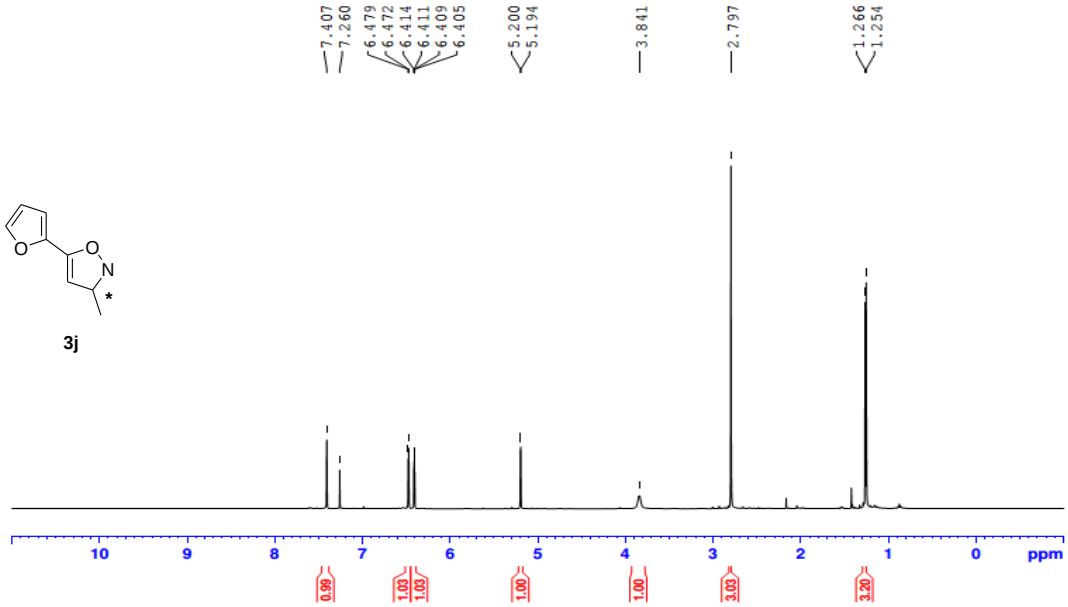

151.69
150.28
148.28
147.84
123.54
119.94
108.43
106.40
101.44
95.97
77.51
77.26
77.01
68.99
46.80
22.15
3g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

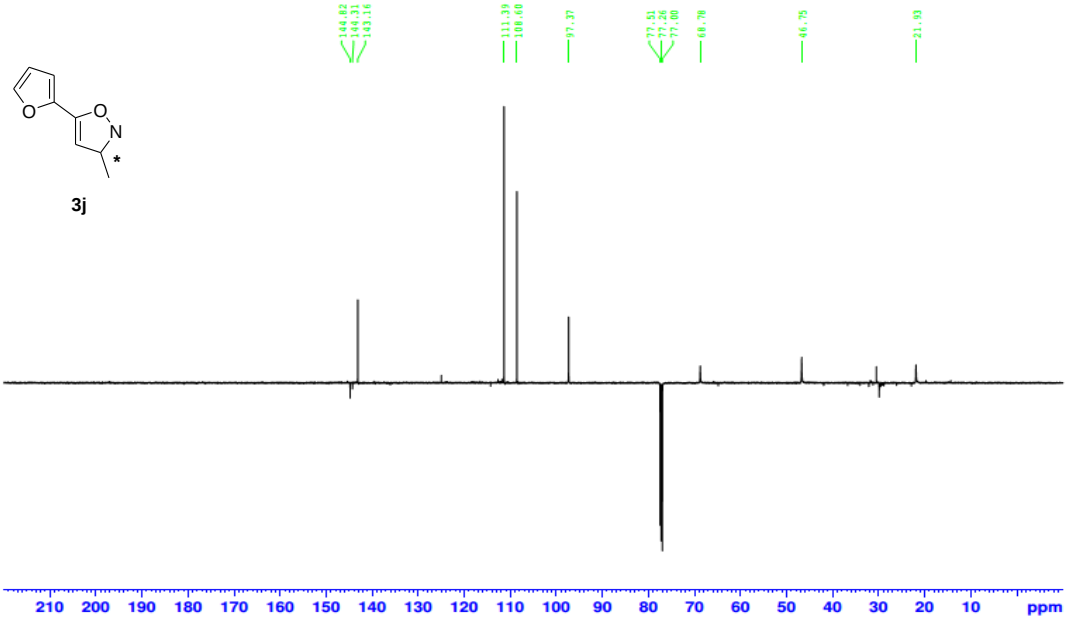

7.613
7.609
7.588
7.583
7.310
7.305
7.281
7.260
6.986
6.961
6.936
6.909
5.649
5.641
3.915
3.885
2.791
1.285
1.264
OMe
O
N
*
3h
10
9
8
7
6
5
4
3
2
1
0
ppm
1.02
1.06
2.08
1.00
4.11
3.10

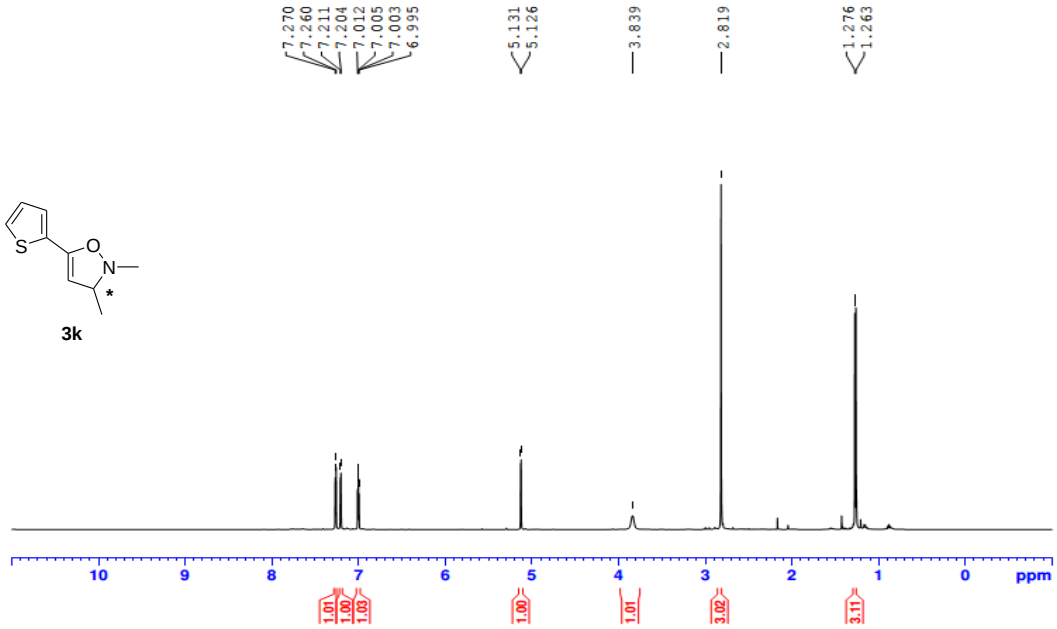

157.11
147.24
129.61
127.72
120.61
118.08
110.73
102.32
77.51
77.26
77.00
69.40
55.56
46.72
22.07
OMe
O
N
*
3h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

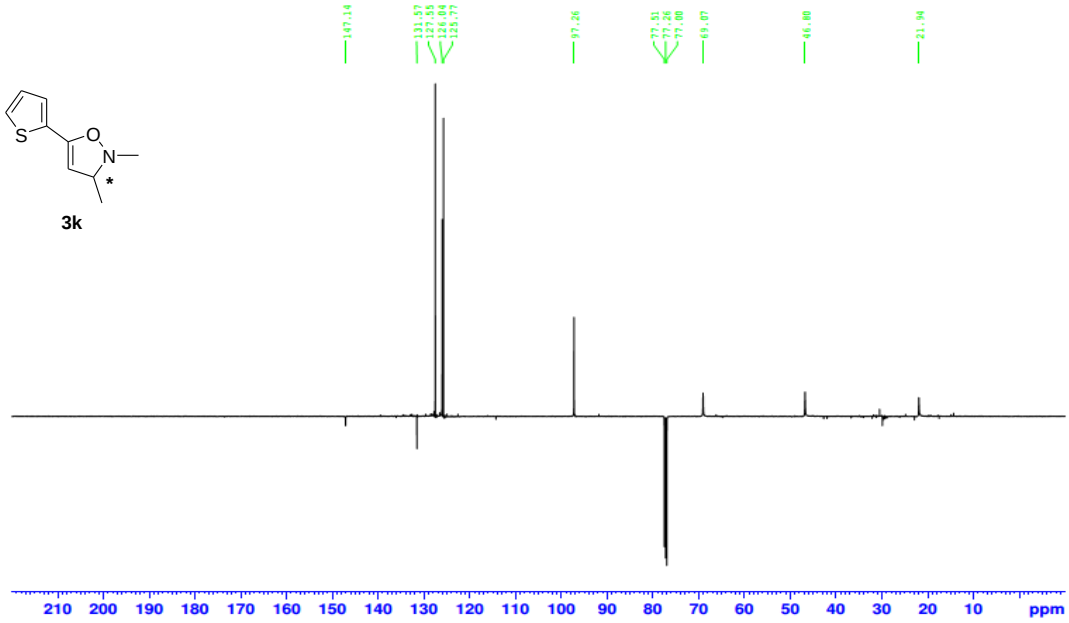

7.461
7.444
7.314
7.297
7.260
5.226
5.221
3.858
2.813
1.276
1.263
Cl
O
N
*
3i
10 9 8 7 6 5 4 3 2 1 0 ppm
2.04
2.00
1.00
0.95
3.02
3.16

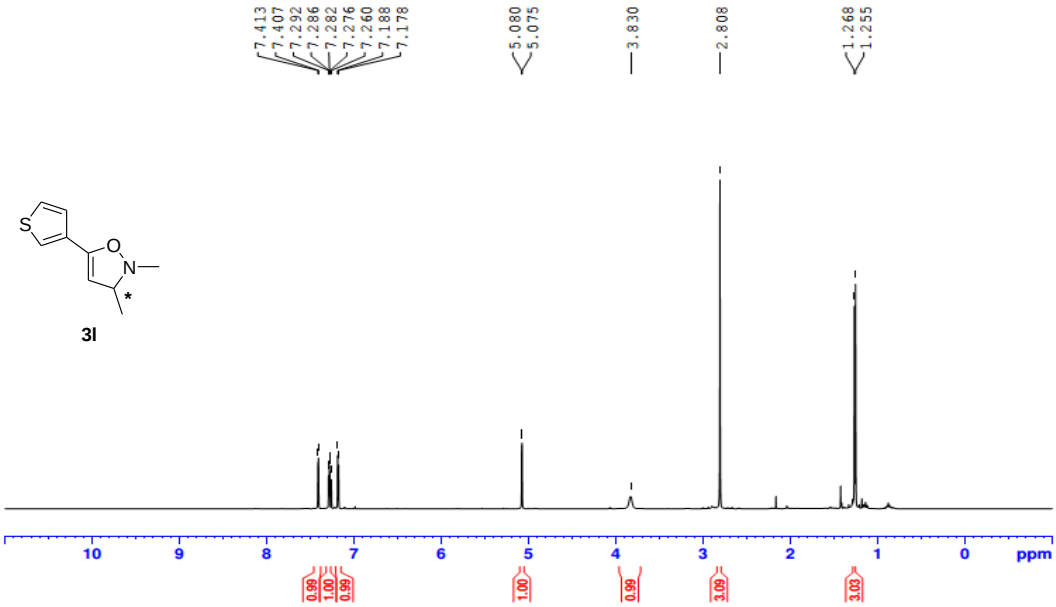

151.06
134.74
129.76
127.85
127.06
97.80
77.51
77.26
77.00
69.02
46.82
22.01
Cl
O
N
*
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

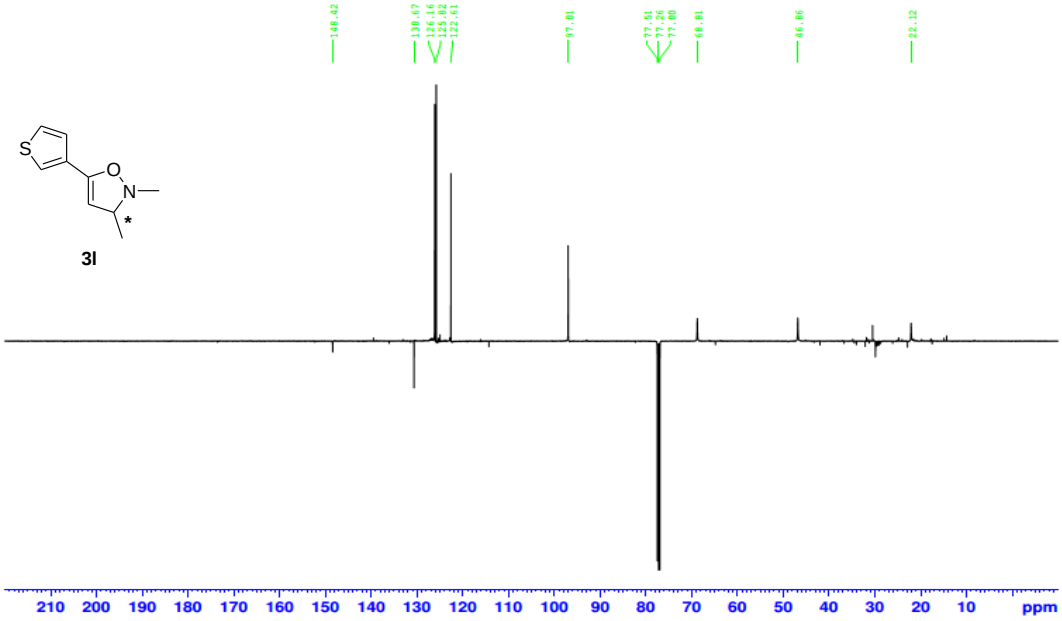

7.407
7.260
6.479
6.472
6.414
6.411
6.409
6.405
5.200
5.194
3.841
2.797
1.266
1.254
3j
10 9 8 7 6 5 4 3 2 1 0 ppm
0.99
1.03
1.03
1.00
1.00
3.03
3.20

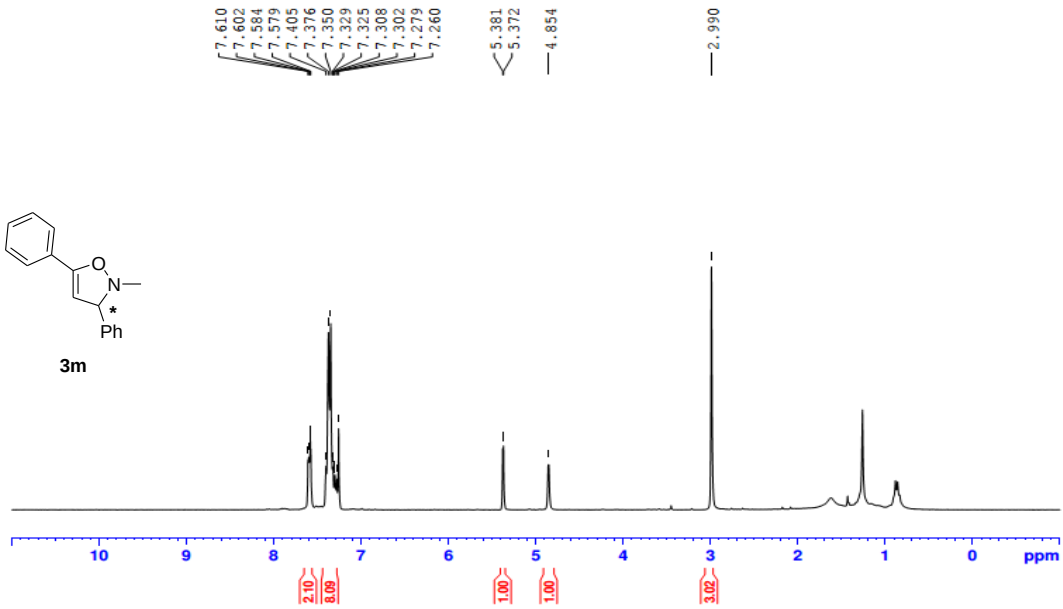

144.82
144.31
143.16
111.39
108.60
97.37
77.51
77.26
77.00
68.78
46.75
21.93
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

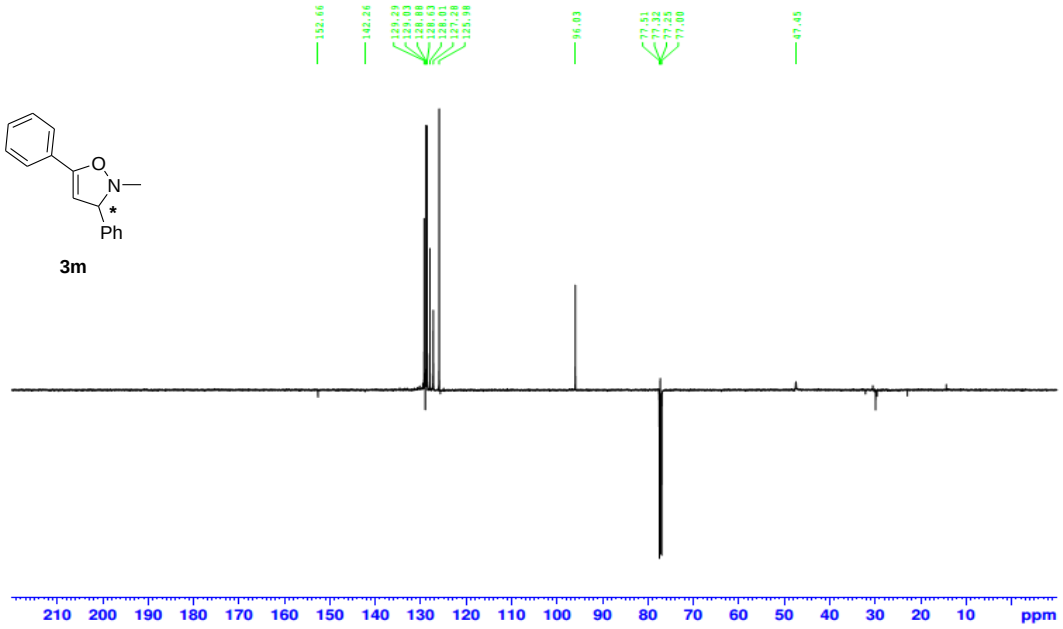

7.270
7.260
7.211
7.204
7.012
7.005
7.003
6.995
5.131
5.126
3.839
2.819
1.276
1.263
S
O
N
*
3k
10 9 8 7 6 5 4 3 2 1 0 ppm
1.01
1.00
1.03
1.00
1.01
3.02
3.11


147.14
131.57
127.55
126.04
125.77
97.26
77.51
77.26
77.00
69.07
46.80
21.94
S
O
N
*
3k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.413
7.407
7.292
7.286
7.282
7.276
7.260
7.188
7.178
5.080
5.075
3.830
2.808
1.268
1.255
S
O
N
*
3l
10
9
8
7
6
5
4
3
2
1
0
ppm
0.99
1.00
0.99
1.00
0.99
3.09
3.03


148.42
130.67
126.16
125.82
122.61
97.01
77.51
77.26
77.00
68.81
46.86
22.12
S
O
N
*
3l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.610
7.602
7.584
7.579
7.405
7.376
7.350
7.329
7.325
7.308
7.302
7.279
7.260
5.381
5.372
4.854
2.990
O
N
*
Ph
3m
10
9
8
7
6
5
4
3
2
1
0
ppm
2.10
8.09
1.00
1.00
3.02


152.66
142.26
129.29
129.03
128.88
128.63
128.01
127.28
125.98
96.03
77.51
77.32
77.25
77.00
47.45
O
N
*
Ph
3m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm