Support Information

Cobalt-catalyzed Asymmetric Cyclopropanation with Diazosulfones: Rigidification of Ligand Chiral Enviroment via Hydrogen Bonding and Cyclization

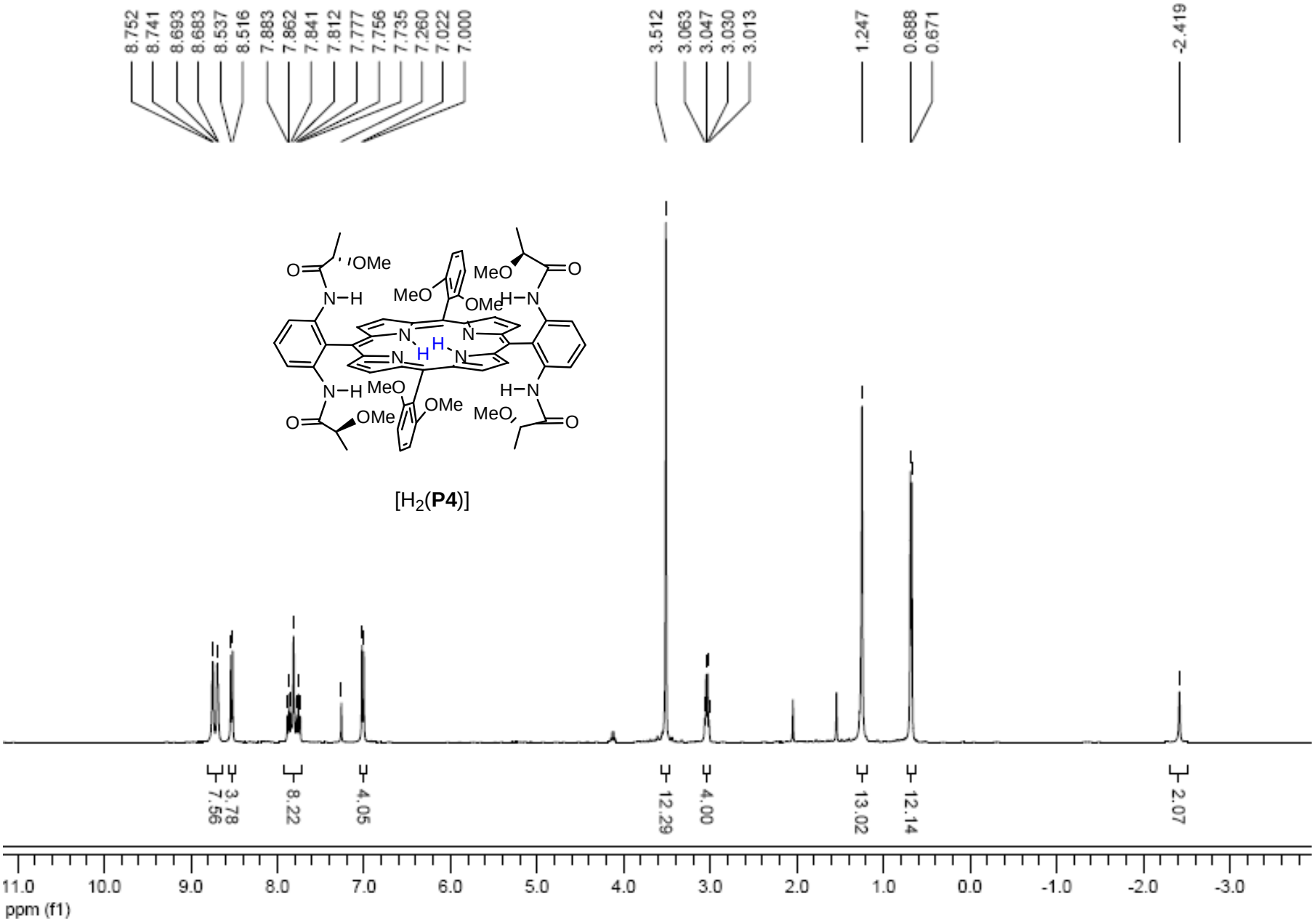
Shifa Zhu, Joshua V. Ruppel,

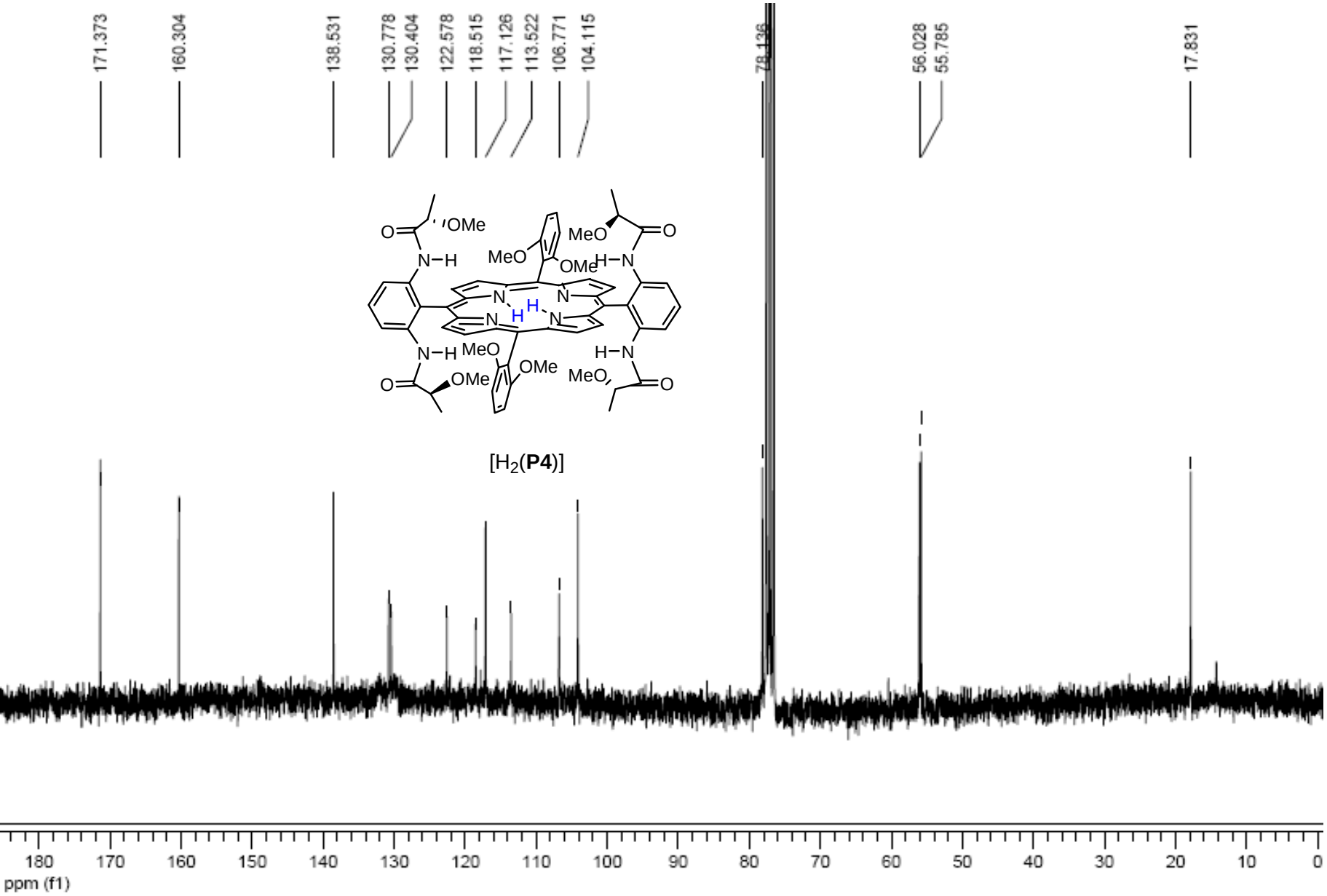
Hongjian Lu, Lukasz Wojtas, and X. Peter Zhang

Department of Chemistry, University of South Florida, Tampa, Florida 33620-5250

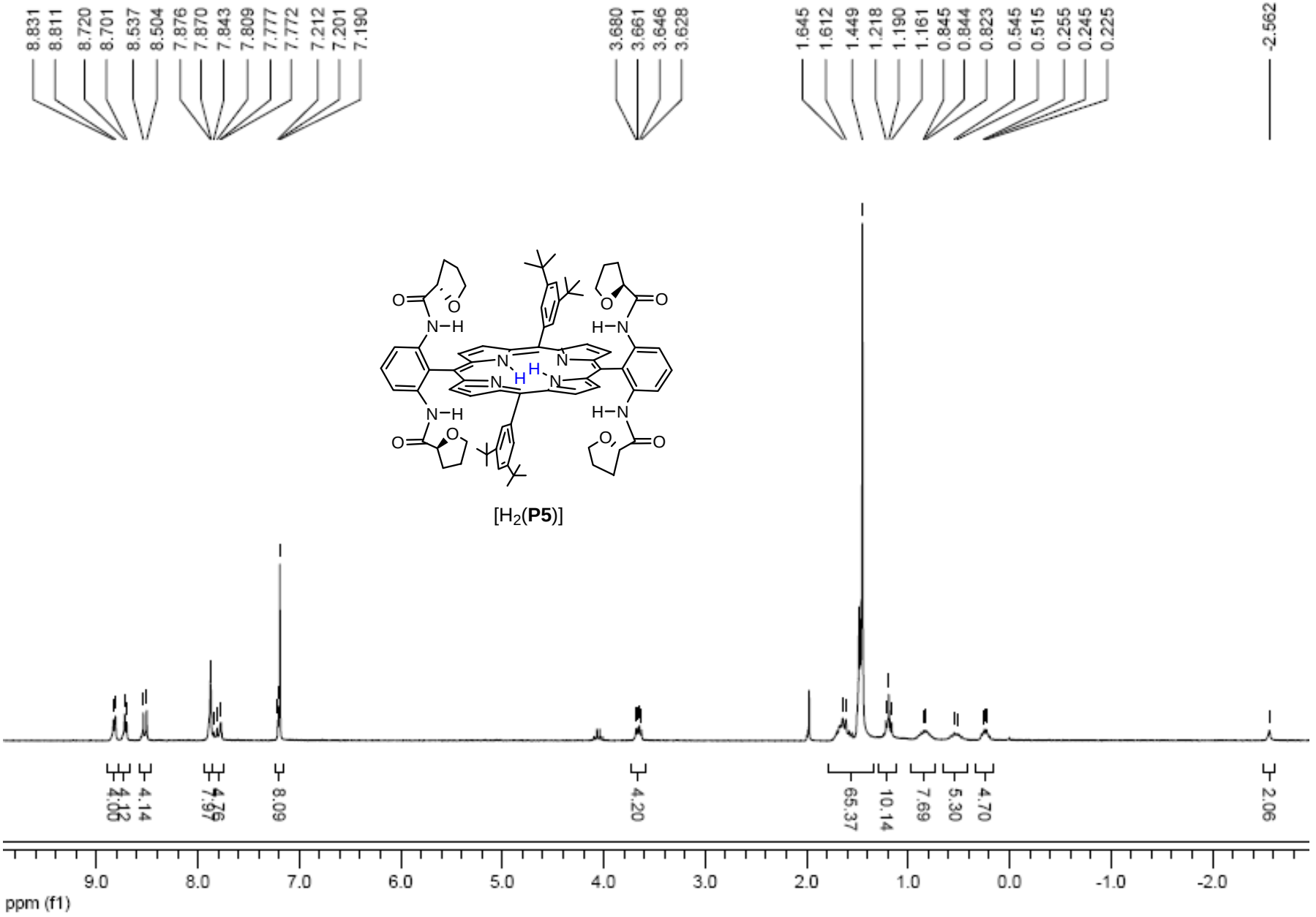
Table of Contents

NMR spectral of [H2(P4)]	SS-3,4
NMR spectral of [H2(P5)]	SS-5,6
NMR spectral of [H2(P6)]	SS-7,8
X-Ray Struture and Top View of [H2(P6)]	SS-9-12
NMR spectral of TsCHN2	SS-13,14
NMR spectral of MsCHN2	SS-15
NMR spectral of NsCHN2	SS-16
NMR spectral and HPLC data of 1-Methyl-4-(2-phenylcyclopropylsulfonyl)benzene	SS-17-21
X-Ray sturcture of 1-Methyl-4-(2-phenylcyclopropylsulfonyl)benzene	SS-22
NMR spectral and HPLC data of 1-Methoxy-4-(2-phenylcyclopropylsulfonyl)benzene	SS-23-26
NMR spectral and HPLC data of 1-Nitro-4-(2-phenylcyclopropylsulfonyl)benzene	SS-27-30
NMR spectral and HPLC data of 1-Methoxy-4-(2-tosylcyclopropyl)benzene	SS-31-34
NMR spectral and HPLC data of 1-(2-Tosylcyclopropyl)-4-(trifluoromethyl)benzene	SS-35-38
NMR spectral and HPLC data of 1-tert-Butyl-4-(2-tosylcyclopropyl)benzene	SS-39-42
NMR spectral and HPLC data of 1-Nitro-3-(2-tosylcyclopropyl)benzene	SS-43-46
NMR spectral and HPLC data of 2-(2-Tosylcyclopropyl)naphthalene	SS-47-50
NMR spectral and HPLC data of Methyl 2-tosylcyclopropanecarboxylate	SS-51-56
NMR spectral and HPLC data of Ethyl 2-tosylcyclopropanecarboxylate	SS-57-60
NMR spectral and HPLC data of 2-Tosylcyclopropanecarbonitrile	SS-61-64
NMR spectral and HPLC data of 1-(2-Tosylcyclopropyl)ethanone	SS-65-68

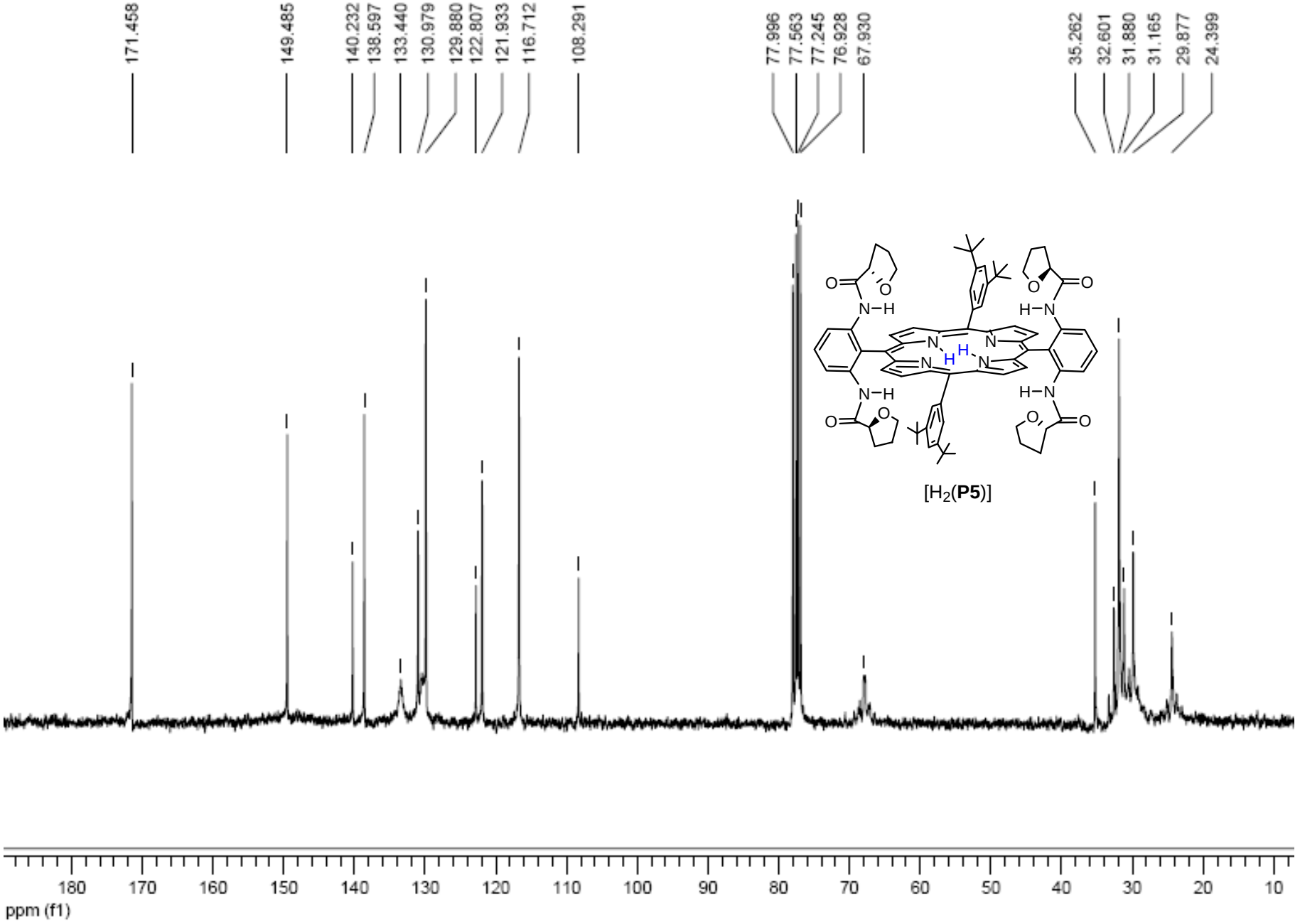

8.752
8.741
8.693
8.683
8.537
8.516
7.883
7.862
7.841
7.812
7.777
7.756
7.735
7.260
7.022
7.000
3.512
3.063
3.047
3.030
3.013
1.247
0.688
0.671
-2.419
[H2(P4)]
7.66
3.78
8.22
4.05
12.29
4.00
13.02
12.14
2.07
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
ppm (f1)

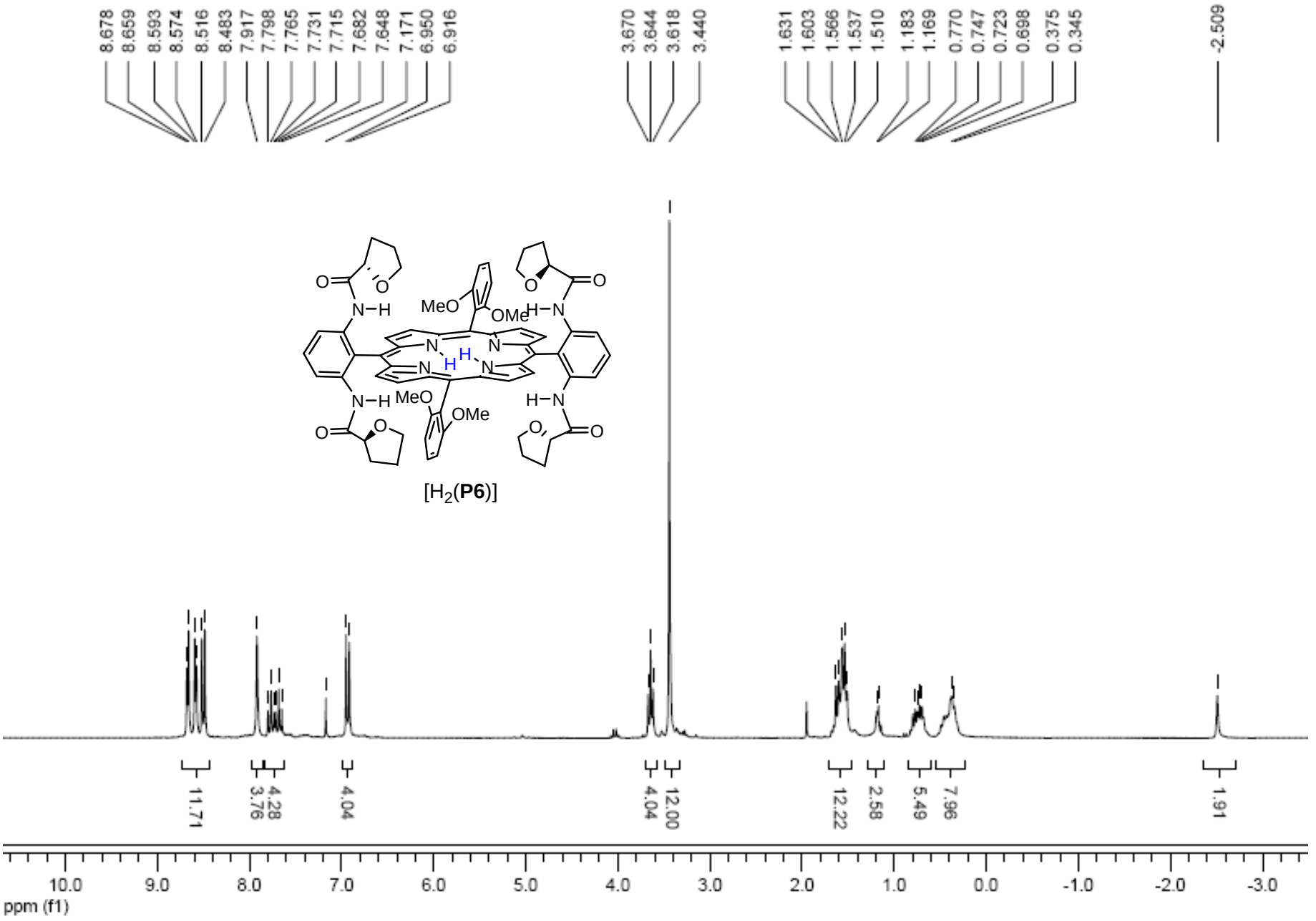

171.373
160.304
138.531
130.778
130.404
122.578
118.515
117.126
113.522
106.771
104.115
78.136
56.028
55.785
17.831
OMe
MeO
MeO
OMe
H–N
N–H
N
N
H
H
N
N
N–H
MeO
H–N
OMe
OMe
MeO
O
O
O
O
O
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm (f1)

[H_2(**P4**)]

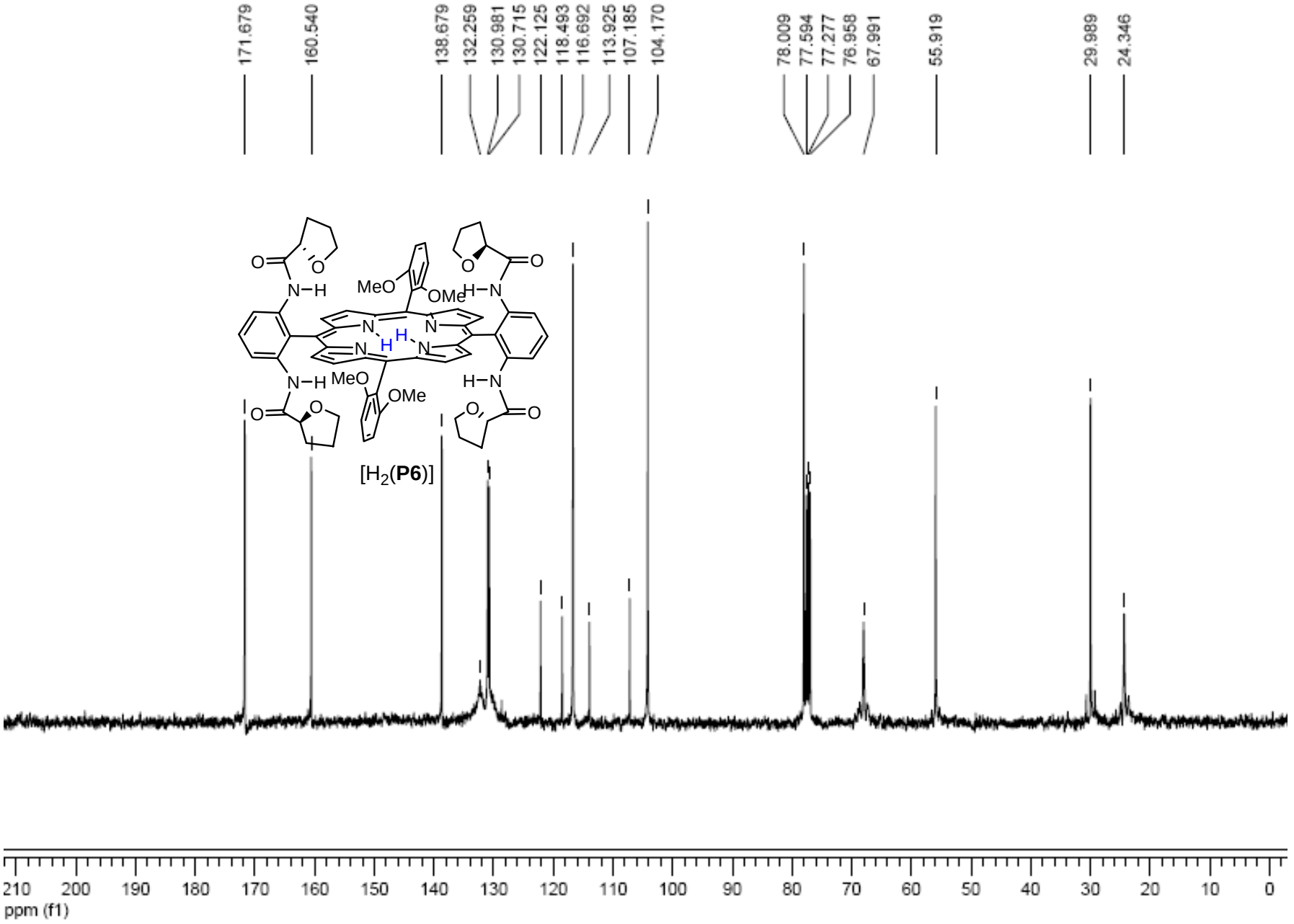

8.831
8.811
8.720
8.701
8.537
8.504
7.876
7.870
7.843
7.809
7.777
7.772
7.212
7.201
7.190
3.680
3.661
3.646
3.628
1.645
1.612
1.449
1.218
1.190
1.161
0.845
0.844
0.823
0.545
0.515
0.255
0.245
0.225
-2.562
4.00
4.12
4.14
7.67
7.76
8.09
4.20
65.37
10.14
7.69
5.30
4.70
2.06
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
ppm (f1)

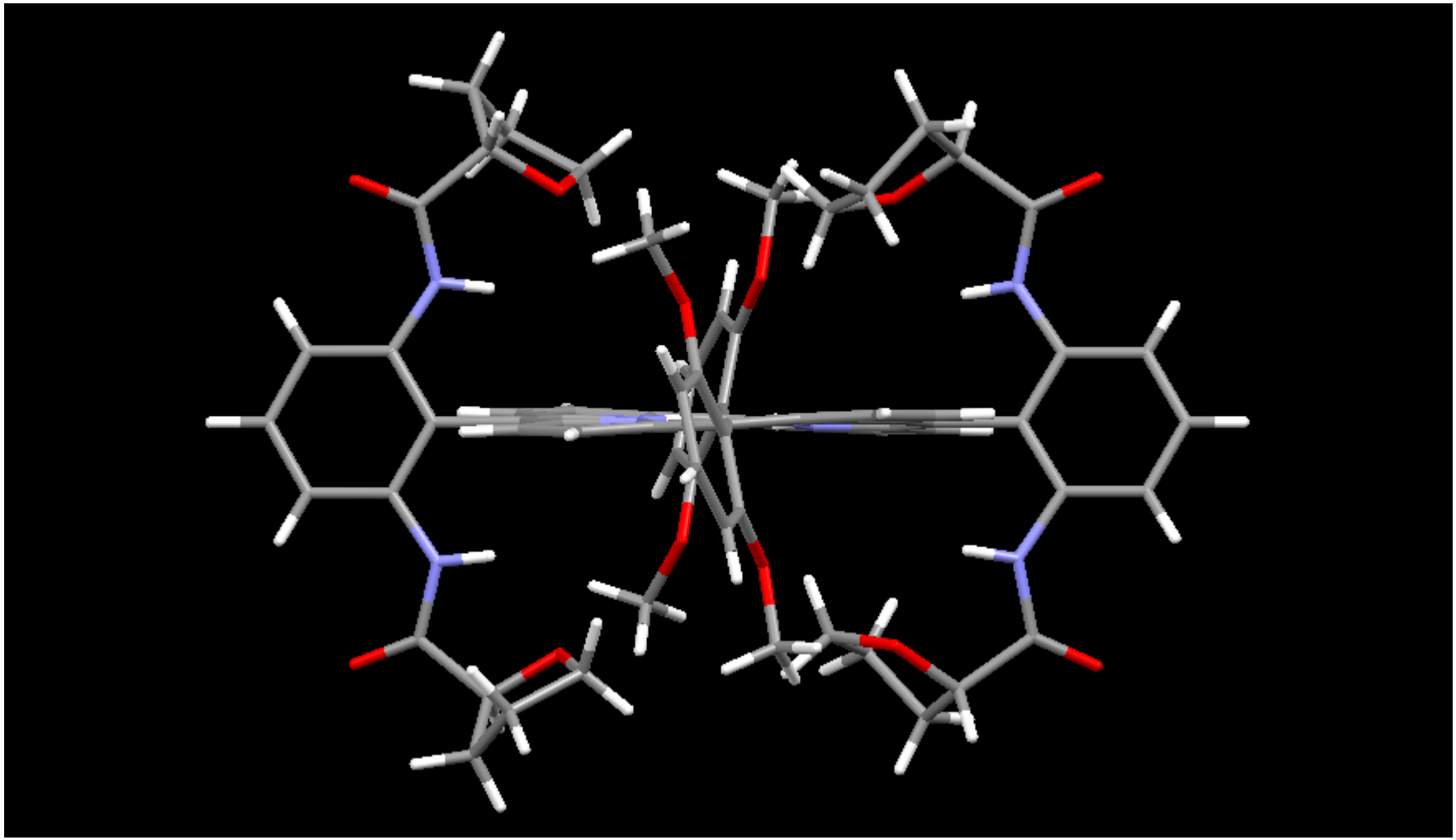
[H_2(**P5**)]


171.458
149.485
140.232
138.597
133.440
130.979
129.880
122.807
121.933
116.712
108.291
77.996
77.563
77.245
76.928
67.930
35.262
32.601
31.880
31.165
29.877
24.399
[H2(P5)]
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm (f1)


8.678
8.659
8.593
8.574
8.516
8.483
7.917
7.798
7.765
7.731
7.715
7.682
7.648
7.171
6.950
6.916
3.670
3.644
3.618
3.440
1.631
1.603
1.566
1.537
1.510
1.183
1.169
0.770
0.747
0.723
0.698
0.375
0.345
-2.509
MeO
OMe
MeO
OMe
11.71
3.76
4.28
4.04
4.04
12.00
12.22
2.58
5.49
7.96
1.91
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
ppm (f1)

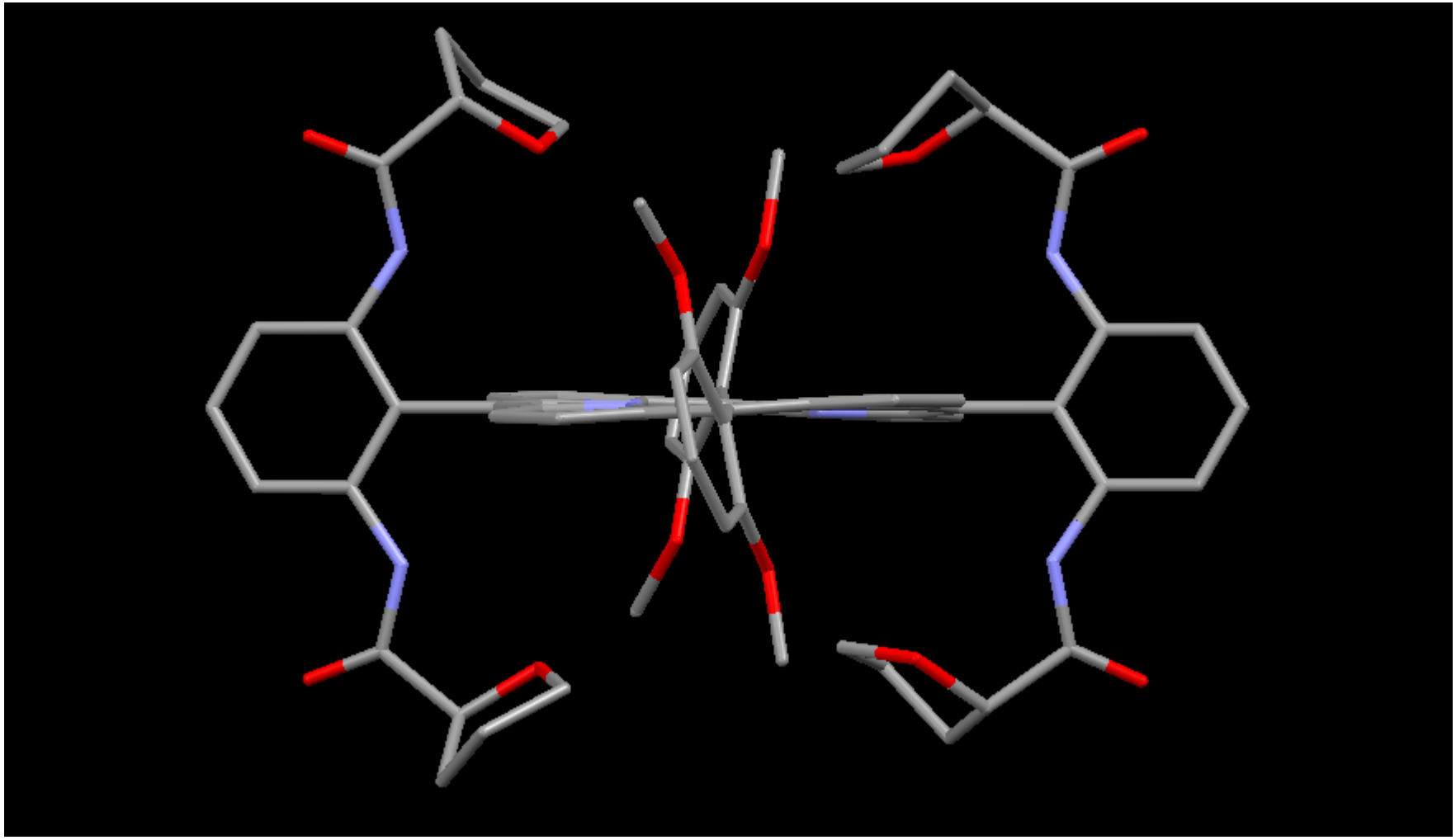
[H_2(**P6**)]


171.679
160.540
138.679
132.259
130.981
130.715
122.125
118.493
116.692
113.925
107.185
104.170
78.009
77.594
77.277
76.958
67.991
55.919
29.989
24.346
MeO
OMe
MeO
OMe
N–H
H–N
N–H
H–N
[H2(P6)]
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm (f1)

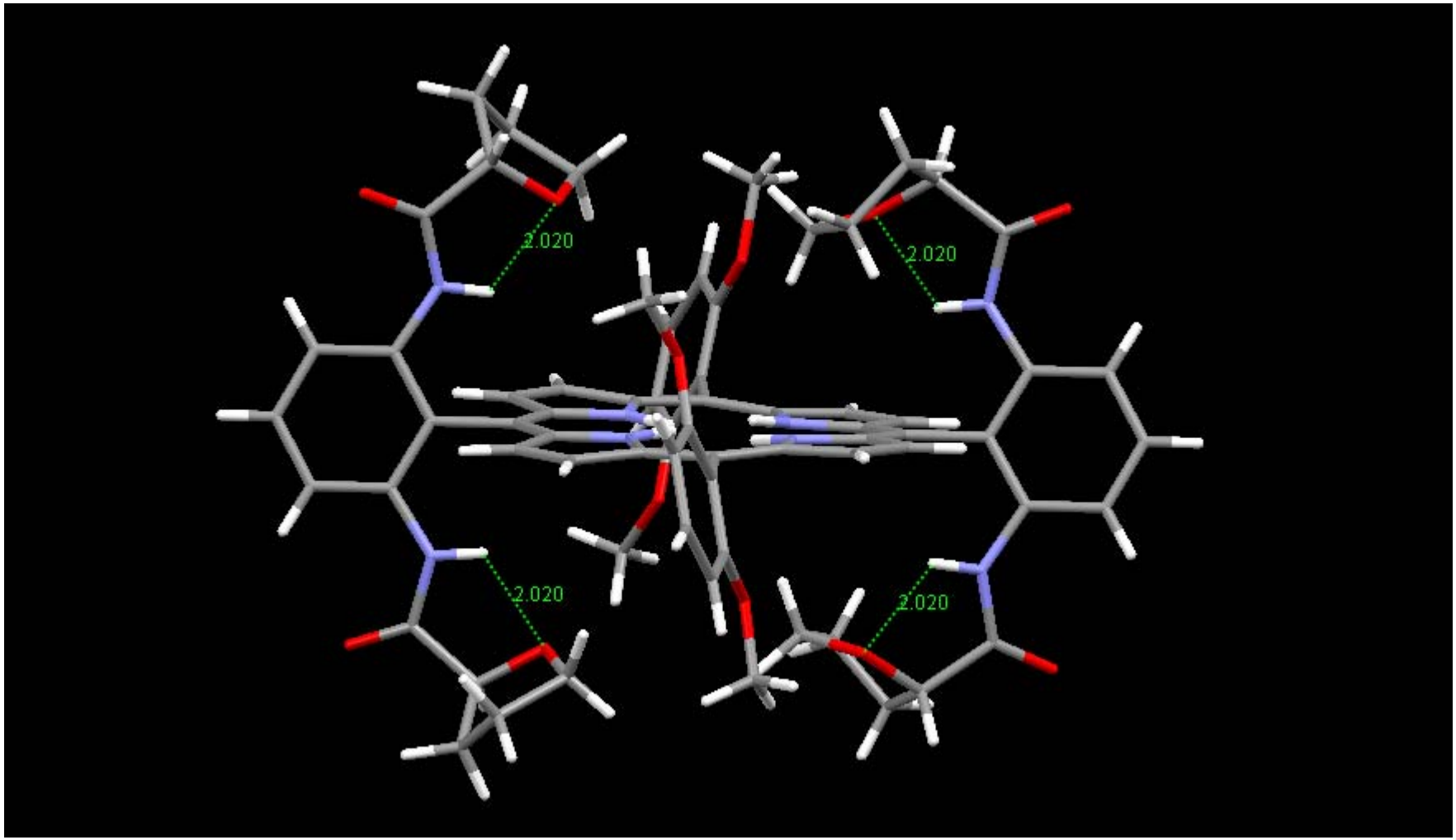


X-Ray structure of [H2(P6)]

SS-9

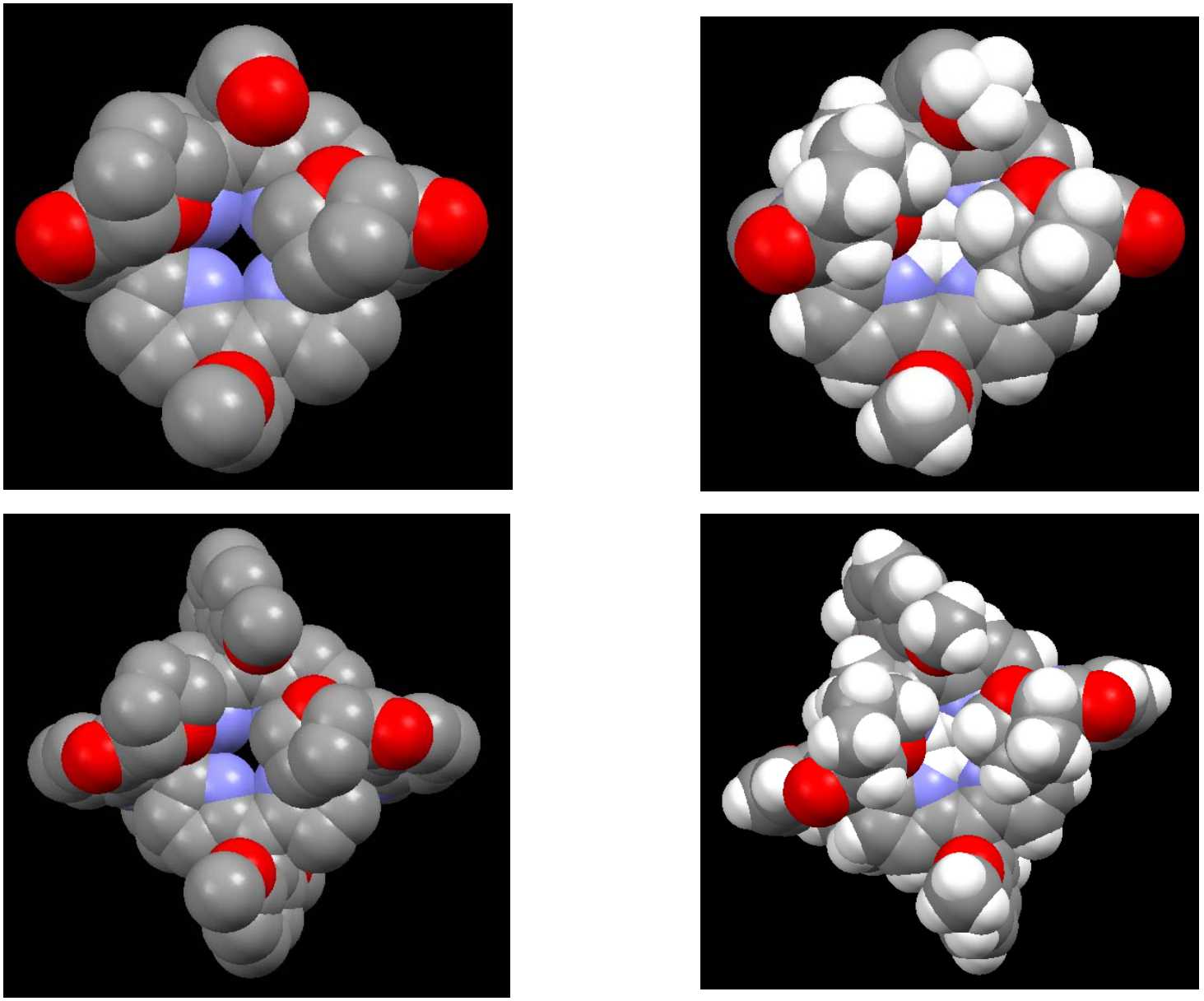


X-Ray structure of [H2(P6)], Hydrogen omitted



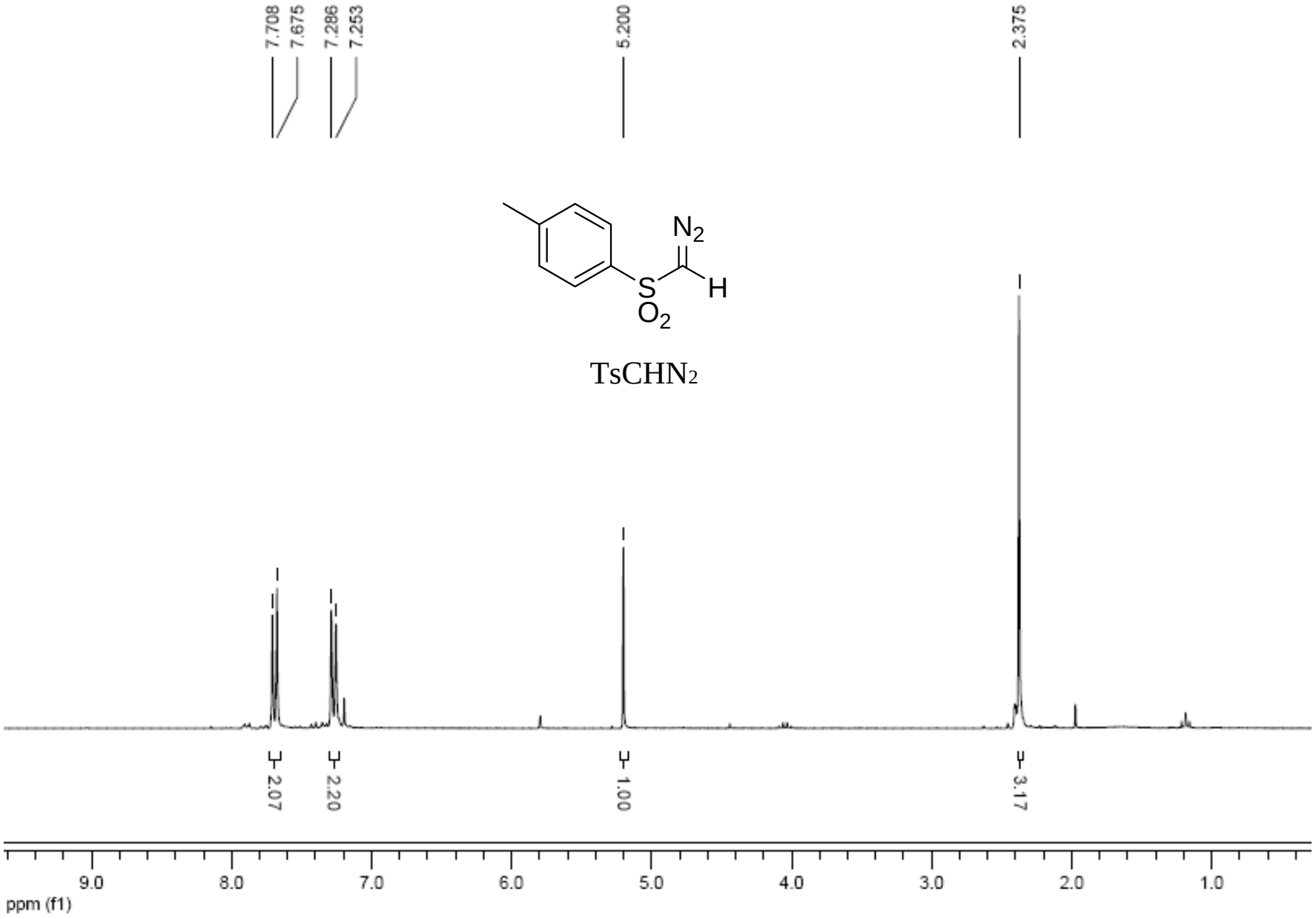
X-Ray structure of [H2(P6)]

Hydrogen Omitted

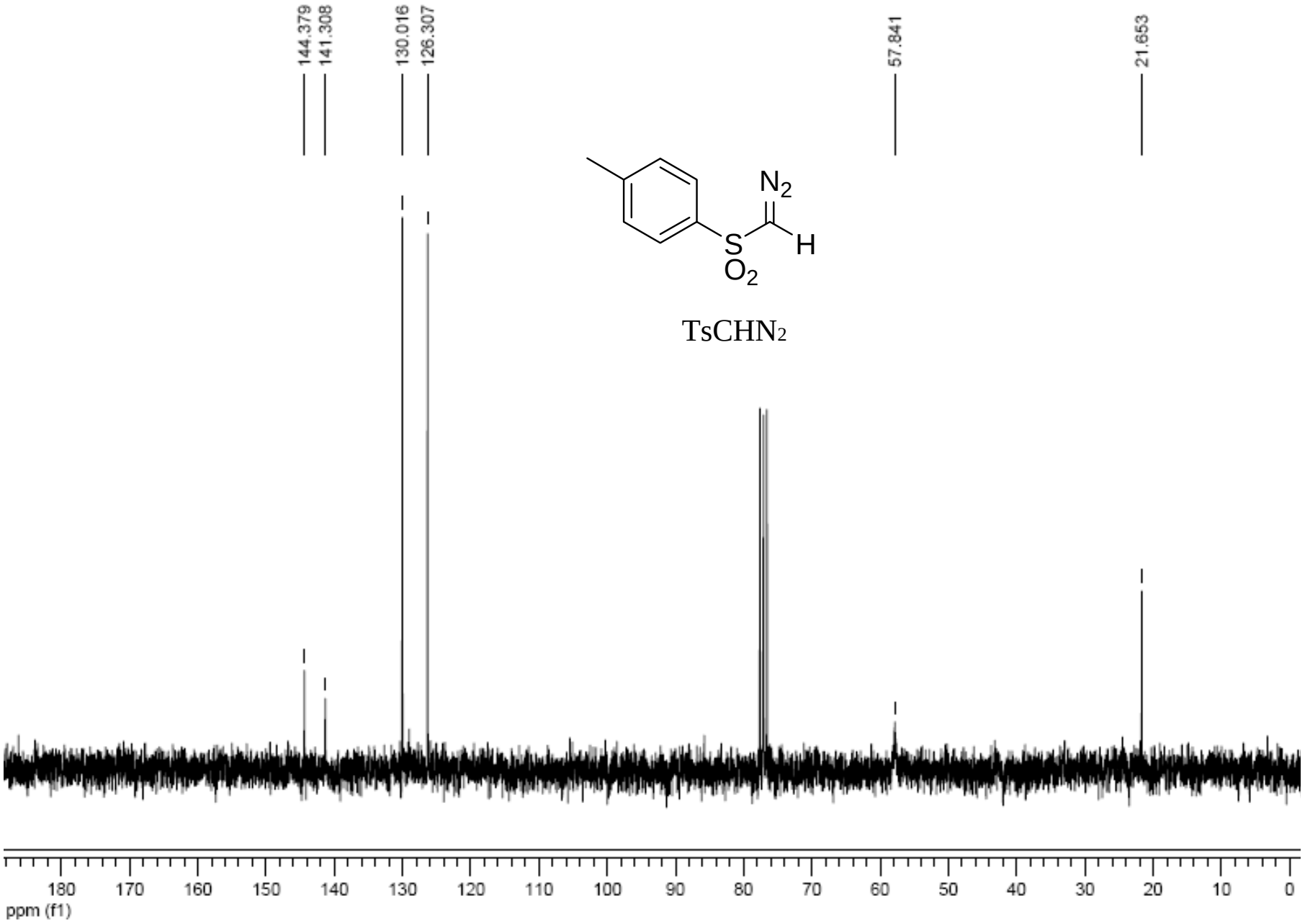


Top View of [H2(P6)]

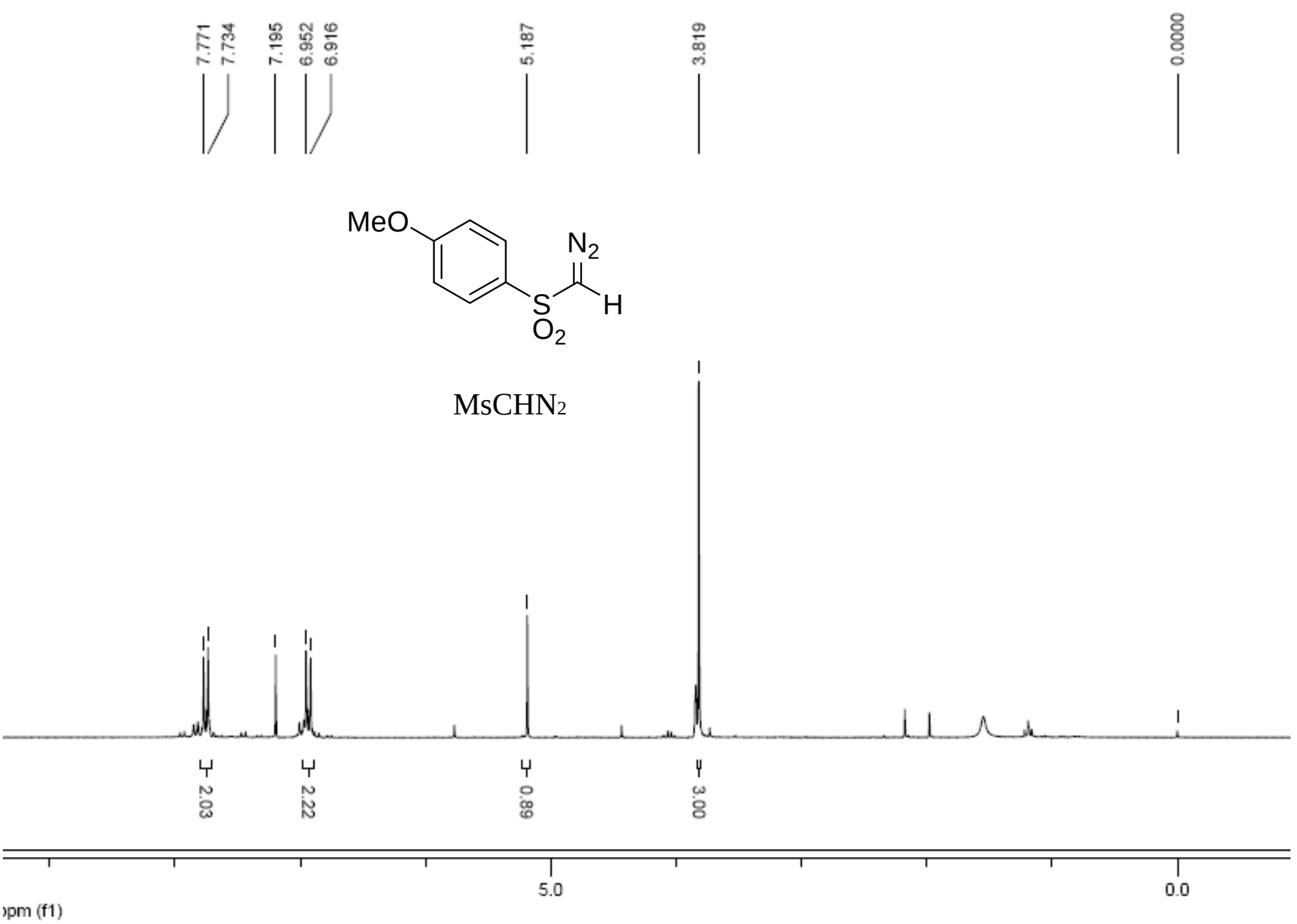
SS-12

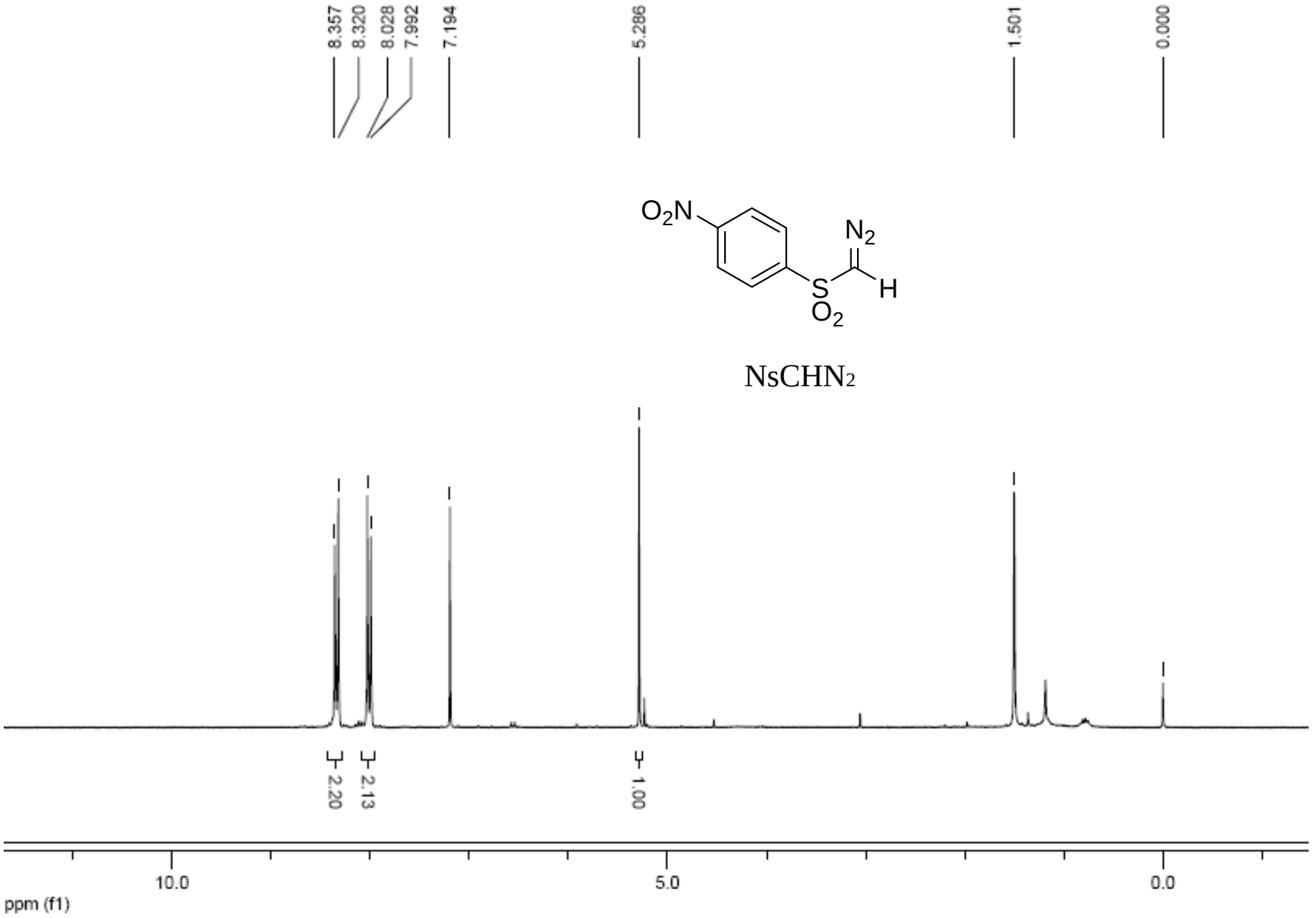

7.708
7.675
7.286
7.253
5.200
2.375
N2
S
O2
H
2.07
2.20
1.00
3.17
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

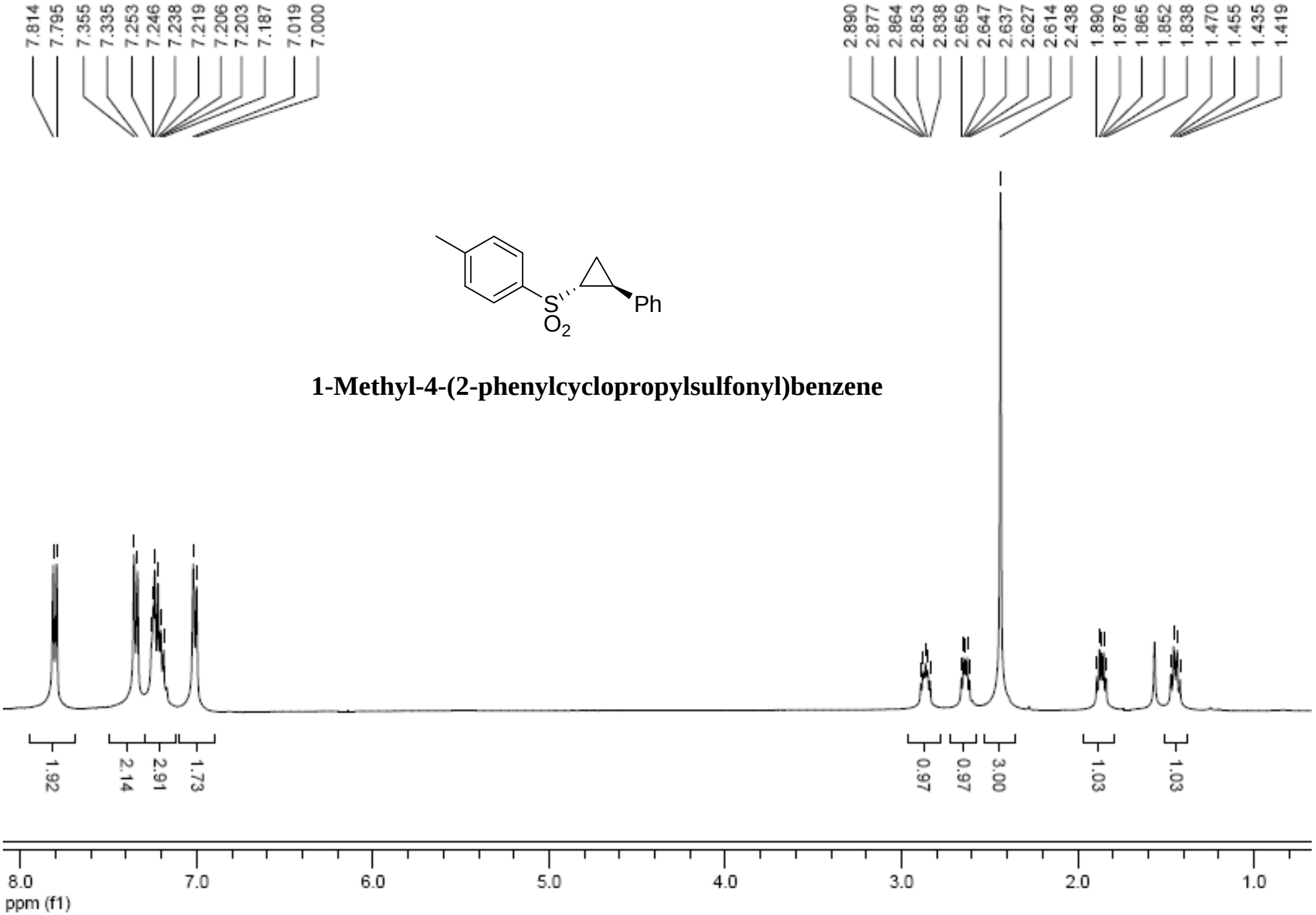
$TsCHN_2$


144.379
141.308
130.016
126.307
57.841
21.653
N2
S
O2
H
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm (f1)

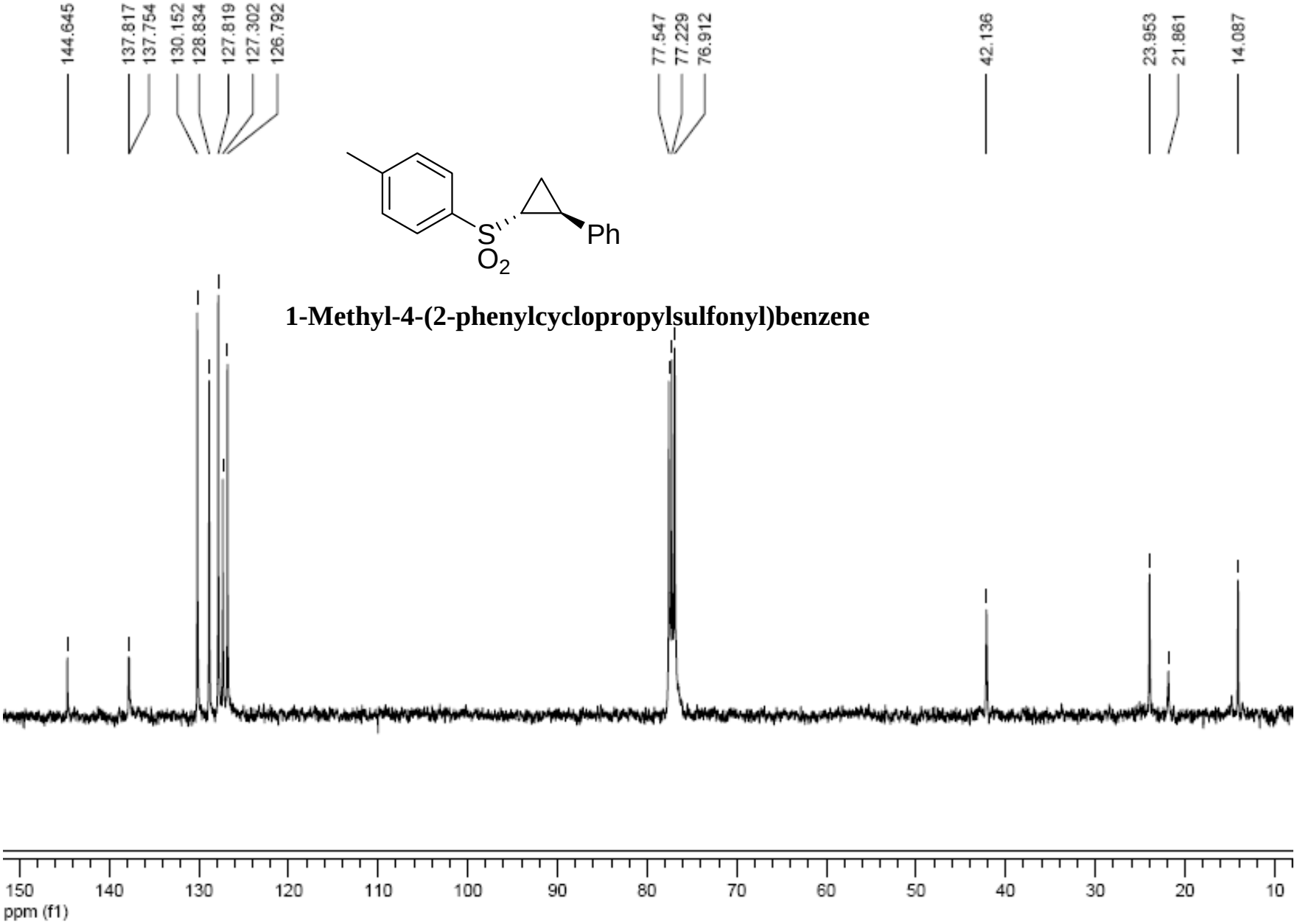
$TsCHN_2$


7.771
7.734
7.195
6.952
6.916
5.187
3.819
0.0000
MeO
N2
S
O2
H
MsCHN2
2.03
2.22
0.89
3.00
5.0
0.0
ppm (f1)

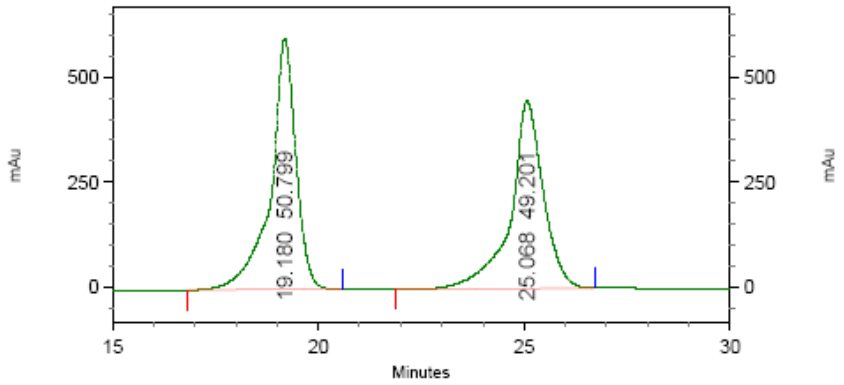

8.357
8.320
8.028
7.992
7.194
5.286
1.501
0.000
O2N
N2
S
O2
H
NsCHN2
2.20
2.13
1.00
10.0
5.0
0.0
ppm (f1)

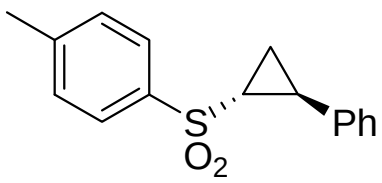


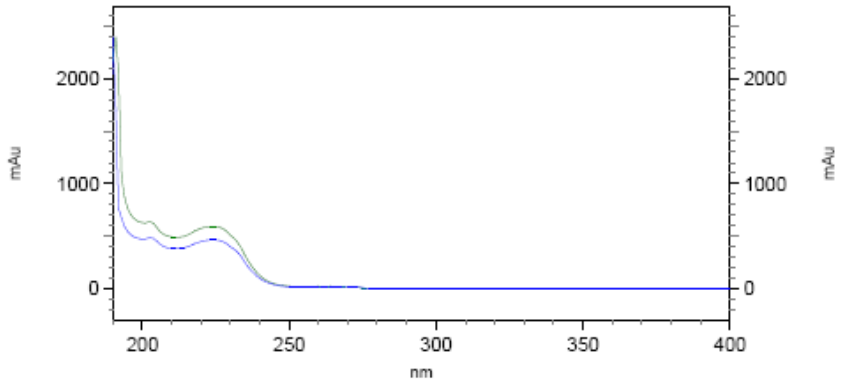
1-Methyl-4-(2-phenylcyclopropylsulfonyl)benzene



1-Methyl-4-(2-phenylcyclopropylsulfonyl)benzene



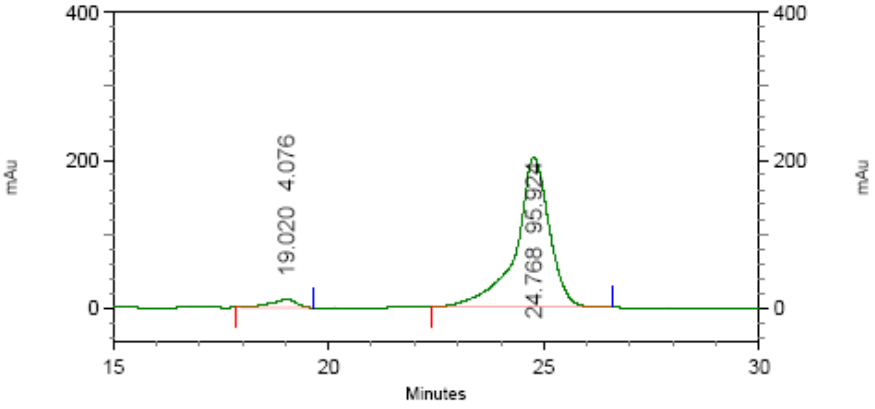


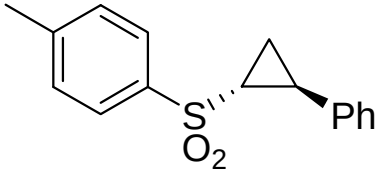


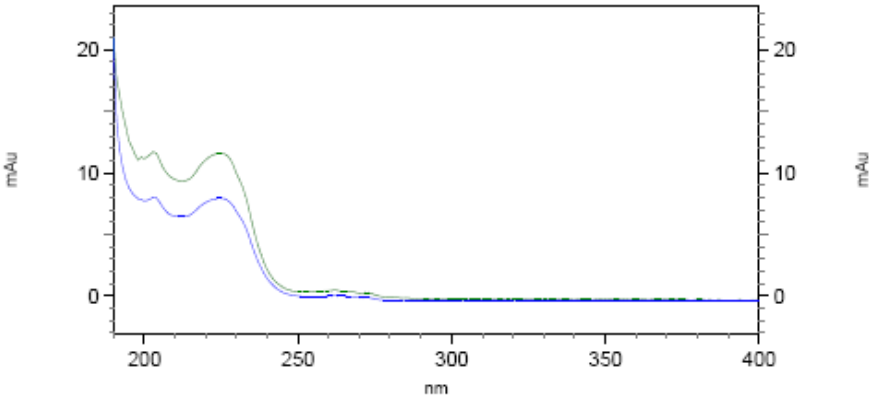
2: 225 nm, 4 nm

Results

Pk #	Name	Retention Time	Area Percent
1		19.180	50.799
2		25.068	49.201
Totals			100.000

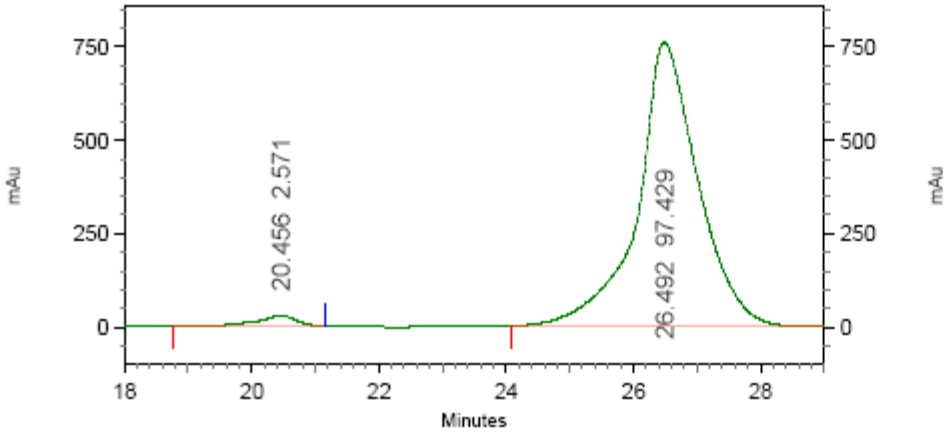


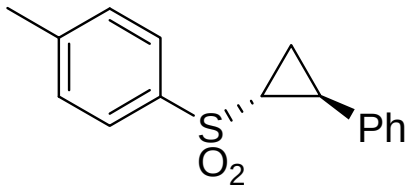


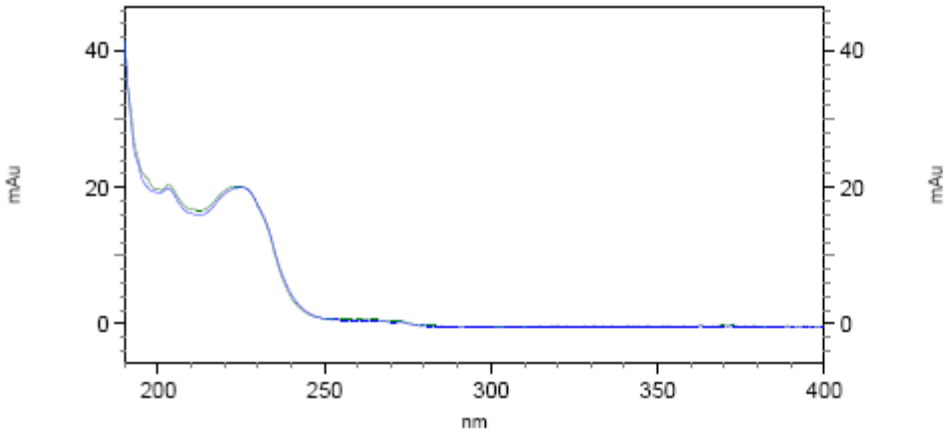


2: 225 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		19.020	4.076
2		24.768	95.924
Totals			100.000



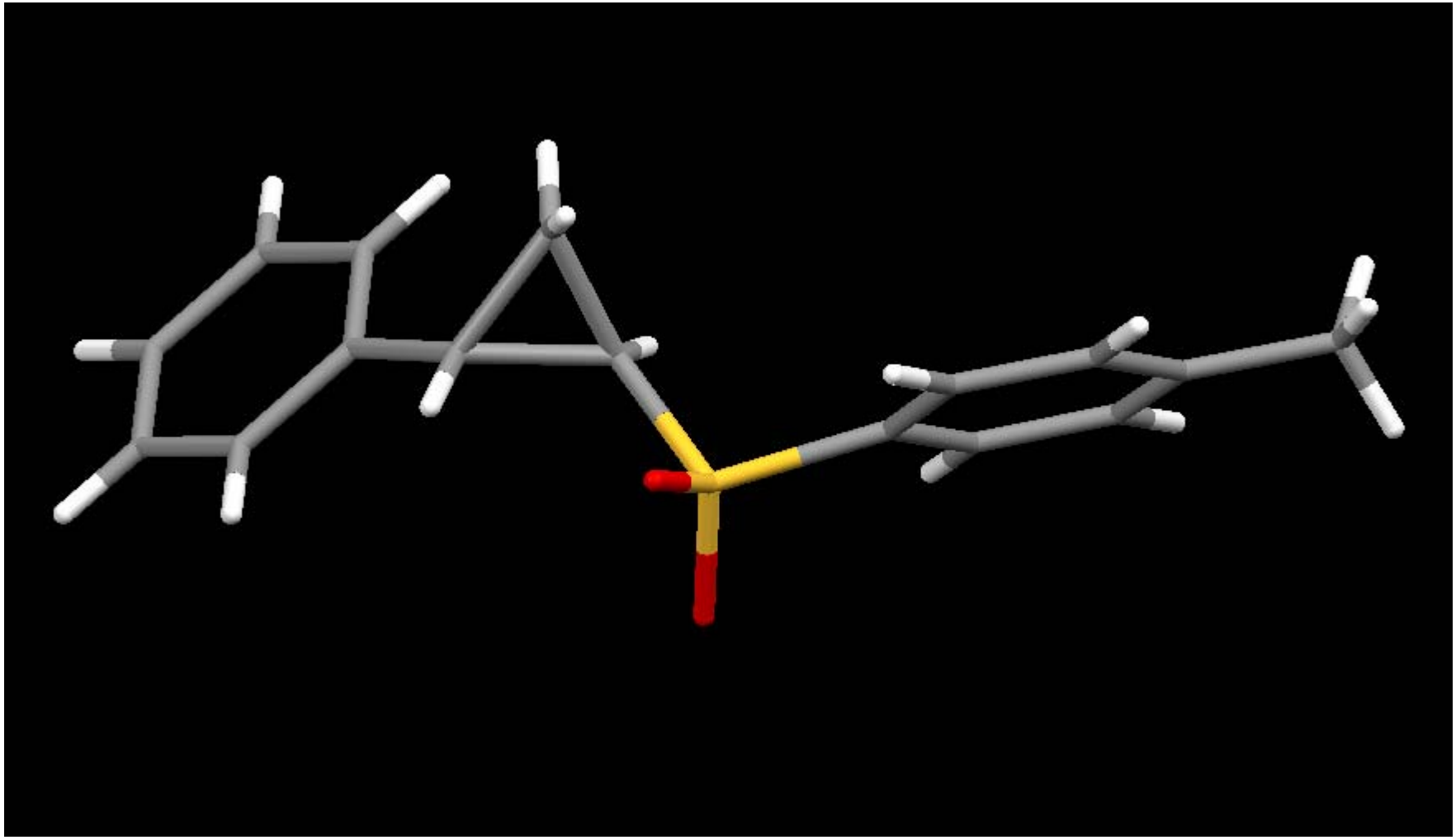




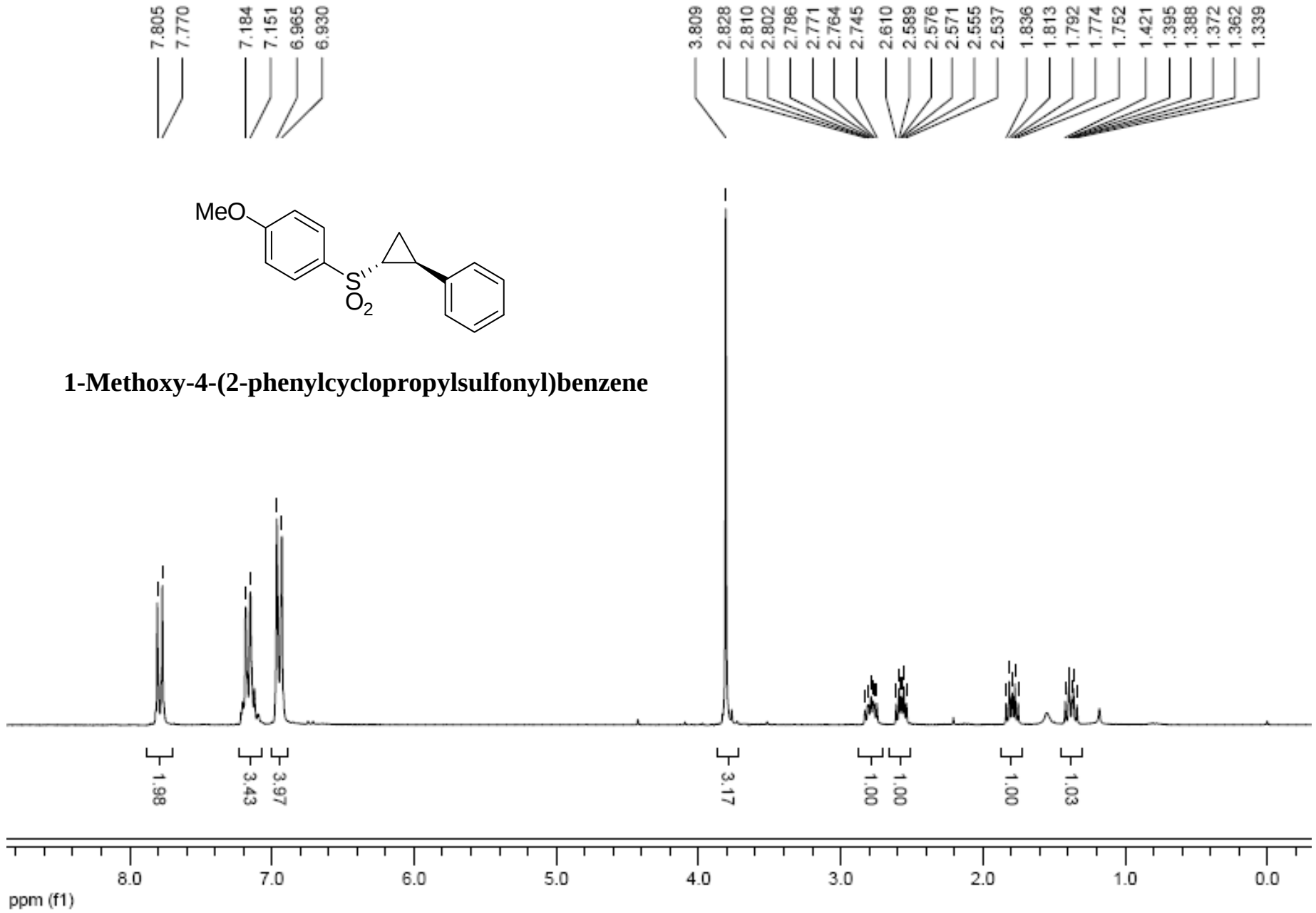
2: 225 nm, 4 nm

Results

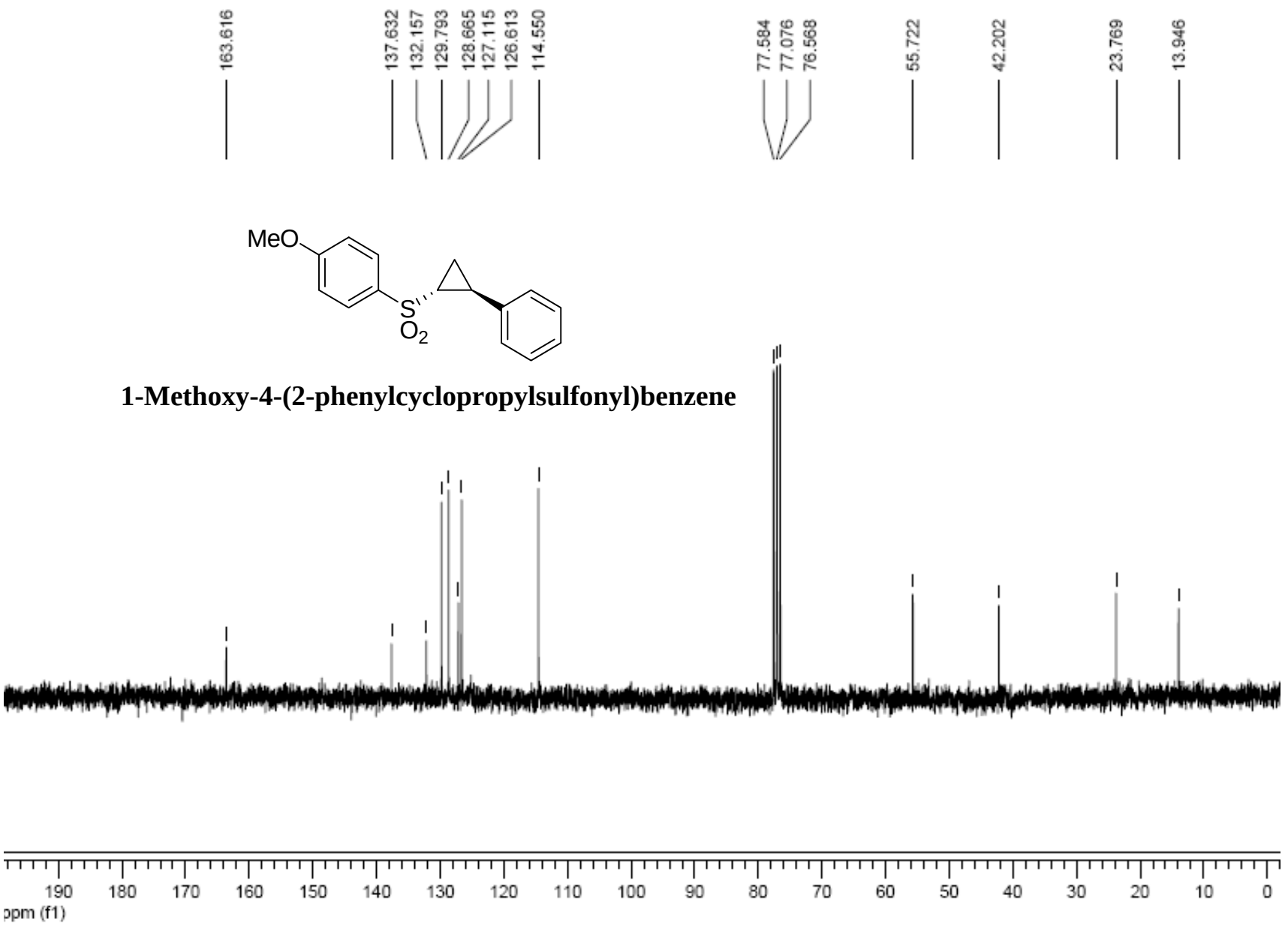
Pk #	Name	Retention Time	Area Percent
1		20.456	2.571
2		26.492	97.429
Totals			100.000



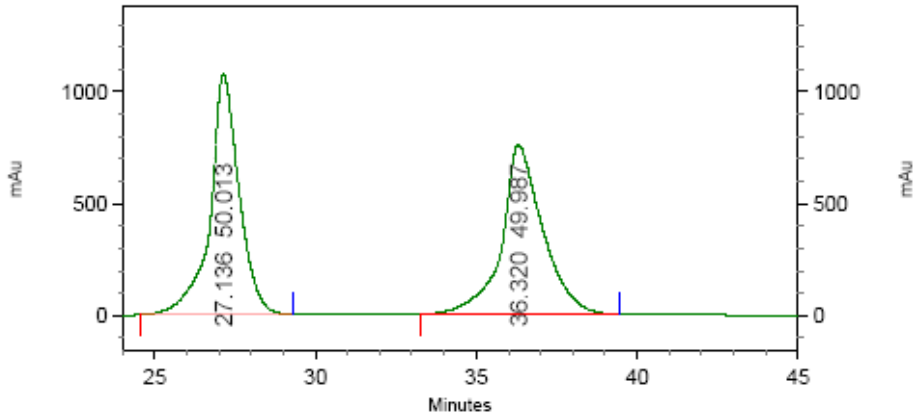
X-Ray structural of 1-Methyl-4-(2-phenylcyclopropylsulfonyl)benzene

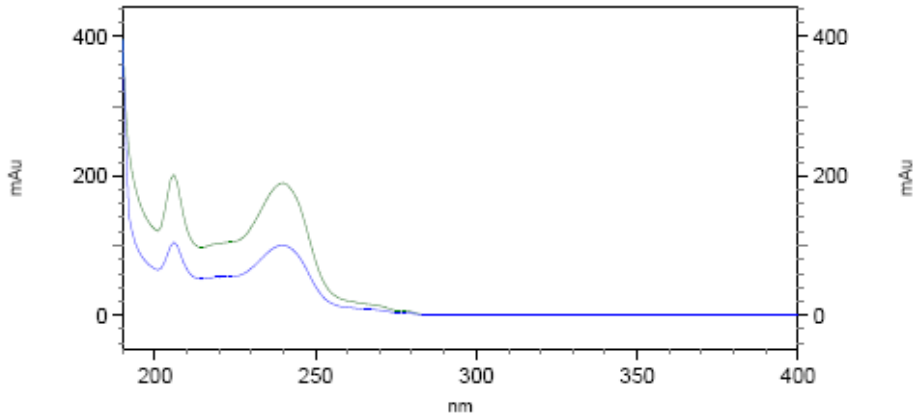


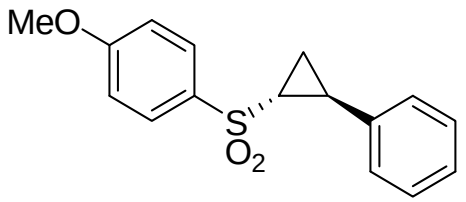
1-Methoxy-4-(2-phenylcyclopropylsulfonyl)benzene



1-Methoxy-4-(2-phenylcyclopropylsulfonyl)benzene



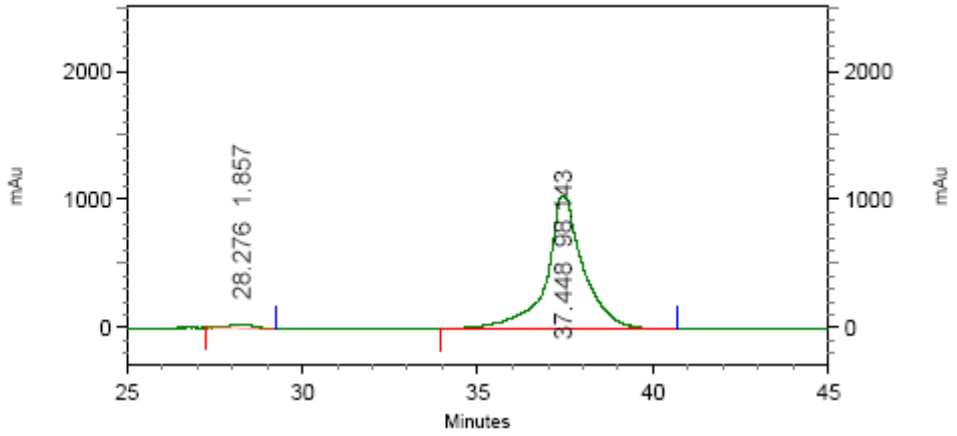


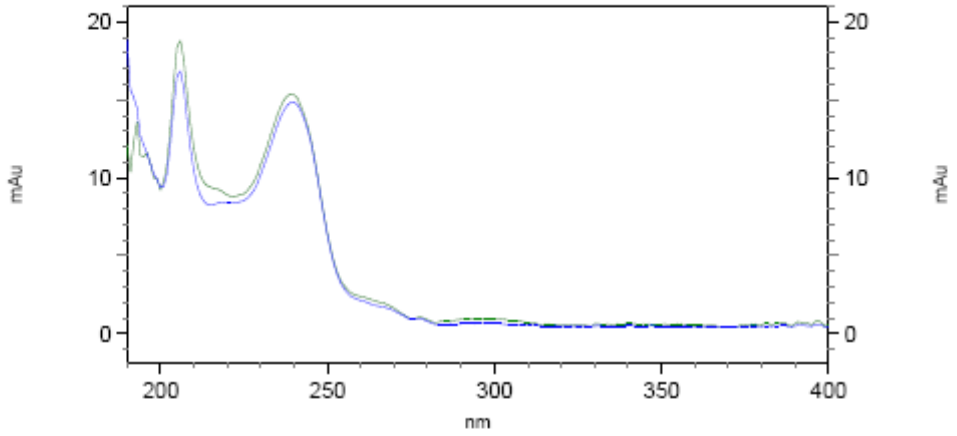


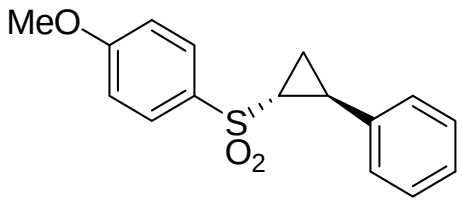
4: 223 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		27.136	50.013
2		36.320	49.987

Totals			100.000



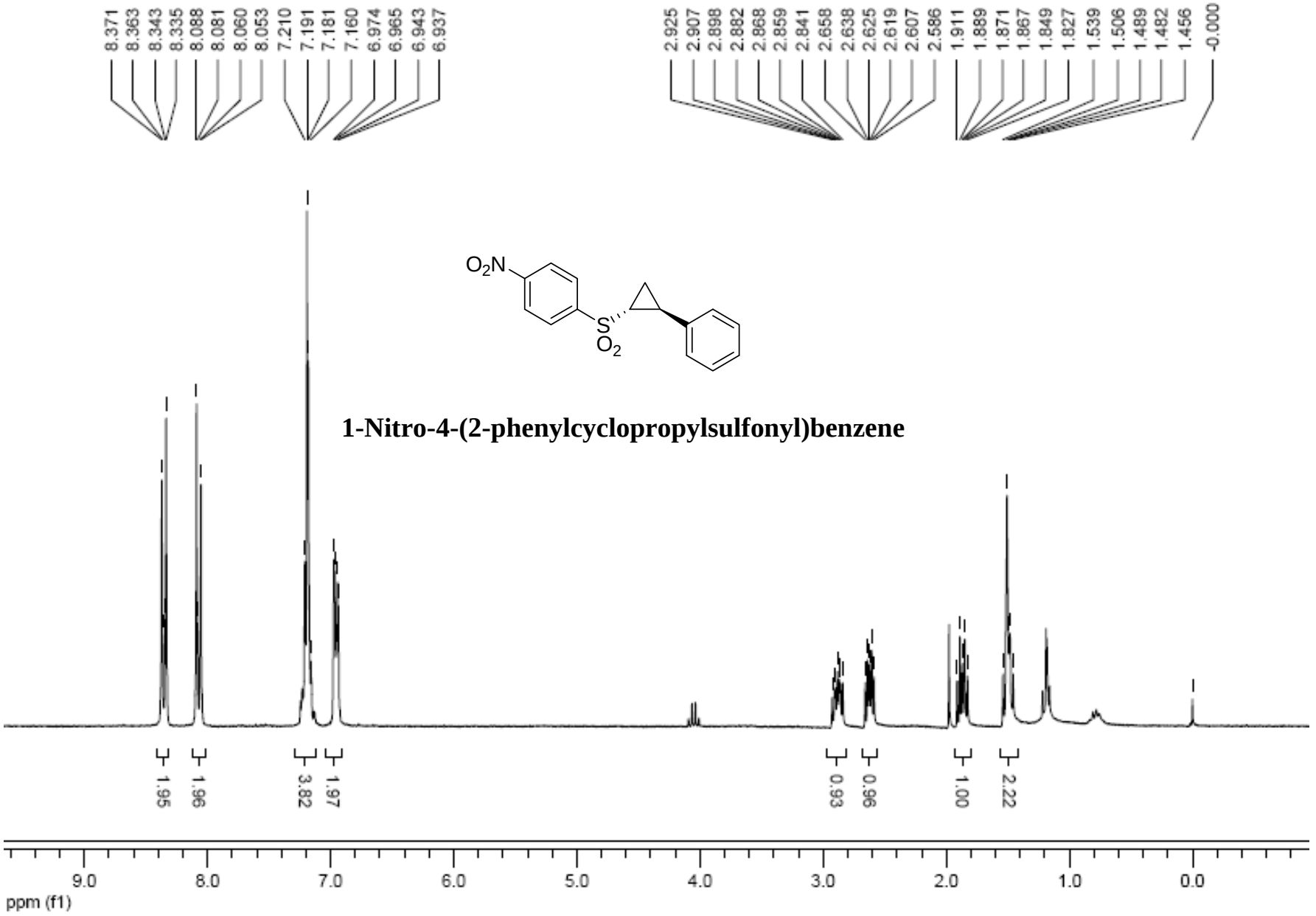




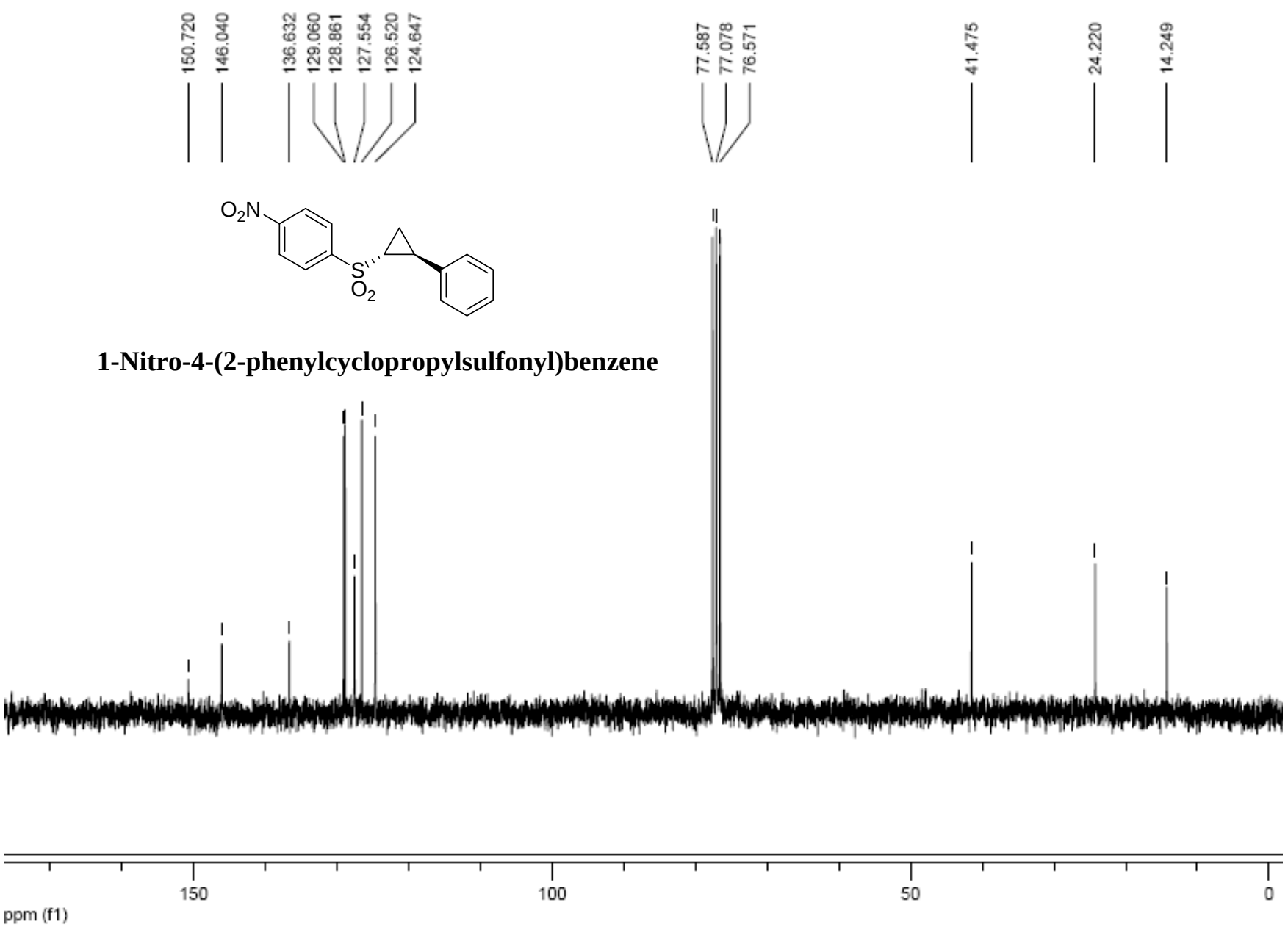
5: 192 nm, 4 nm

Results

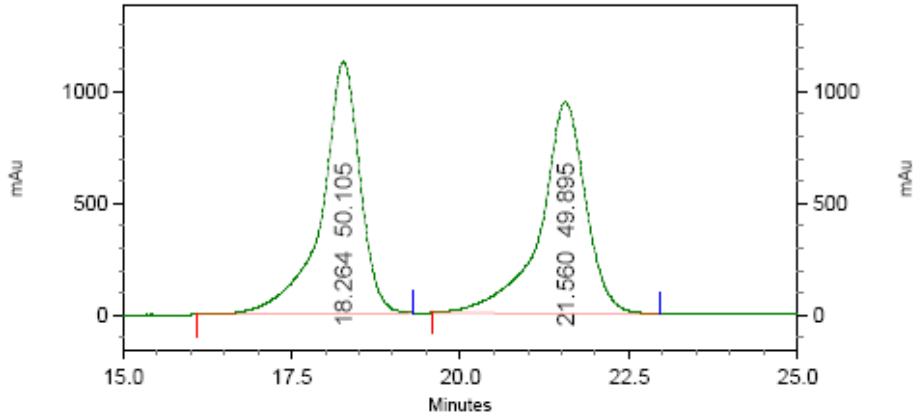
Pk #	Name	Retention Time	Area Percent
1		28.276	1.857
2		37.448	98.143
Totals			100.000

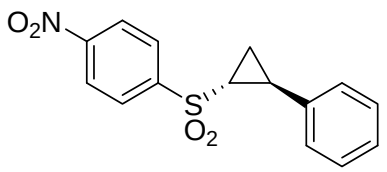


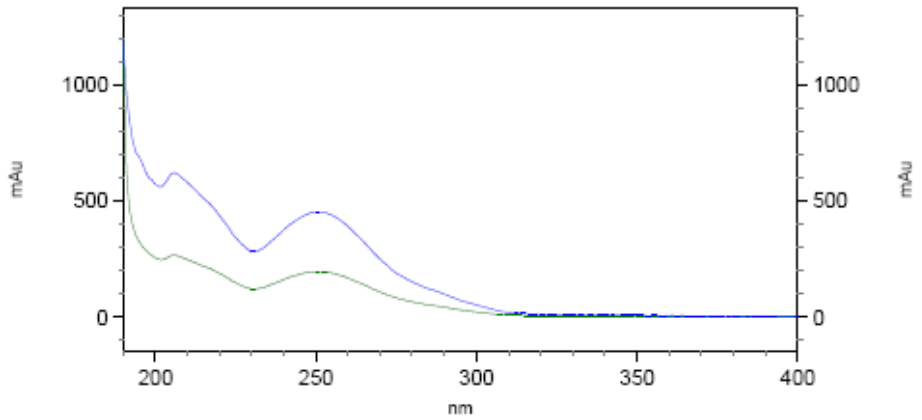
1-Nitro-4-(2-phenylcyclopropylsulfonyl)benzene



1-Nitro-4-(2-phenylcyclopropylsulfonyl)benzene



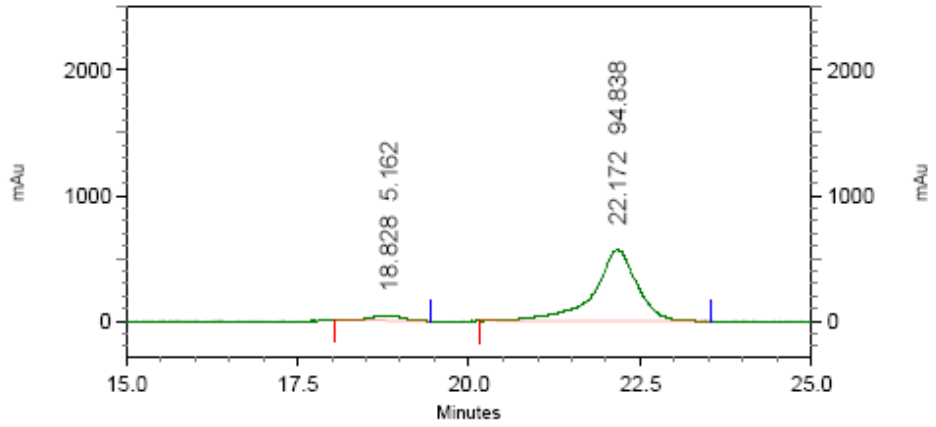


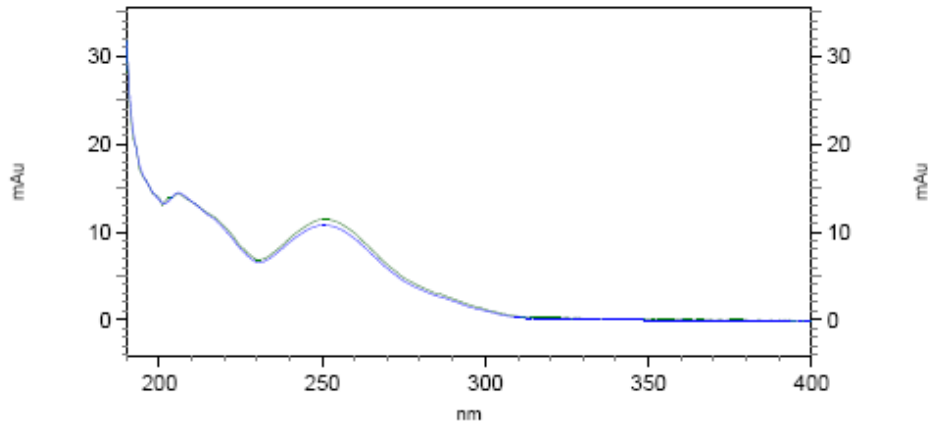


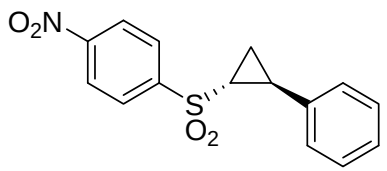
4: 223 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		18.264	50.105
2		21.560	49.895

Totals			100.000

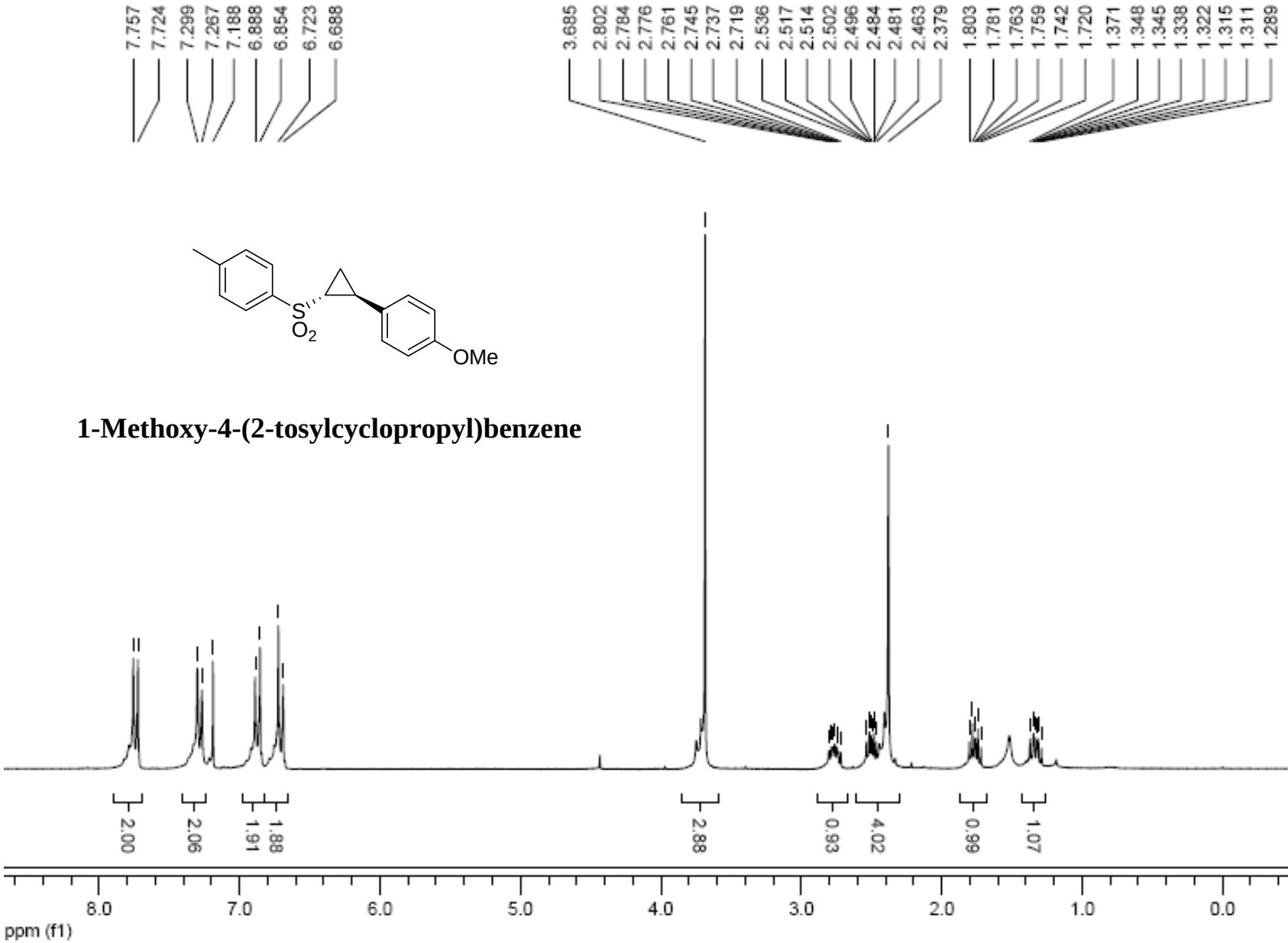




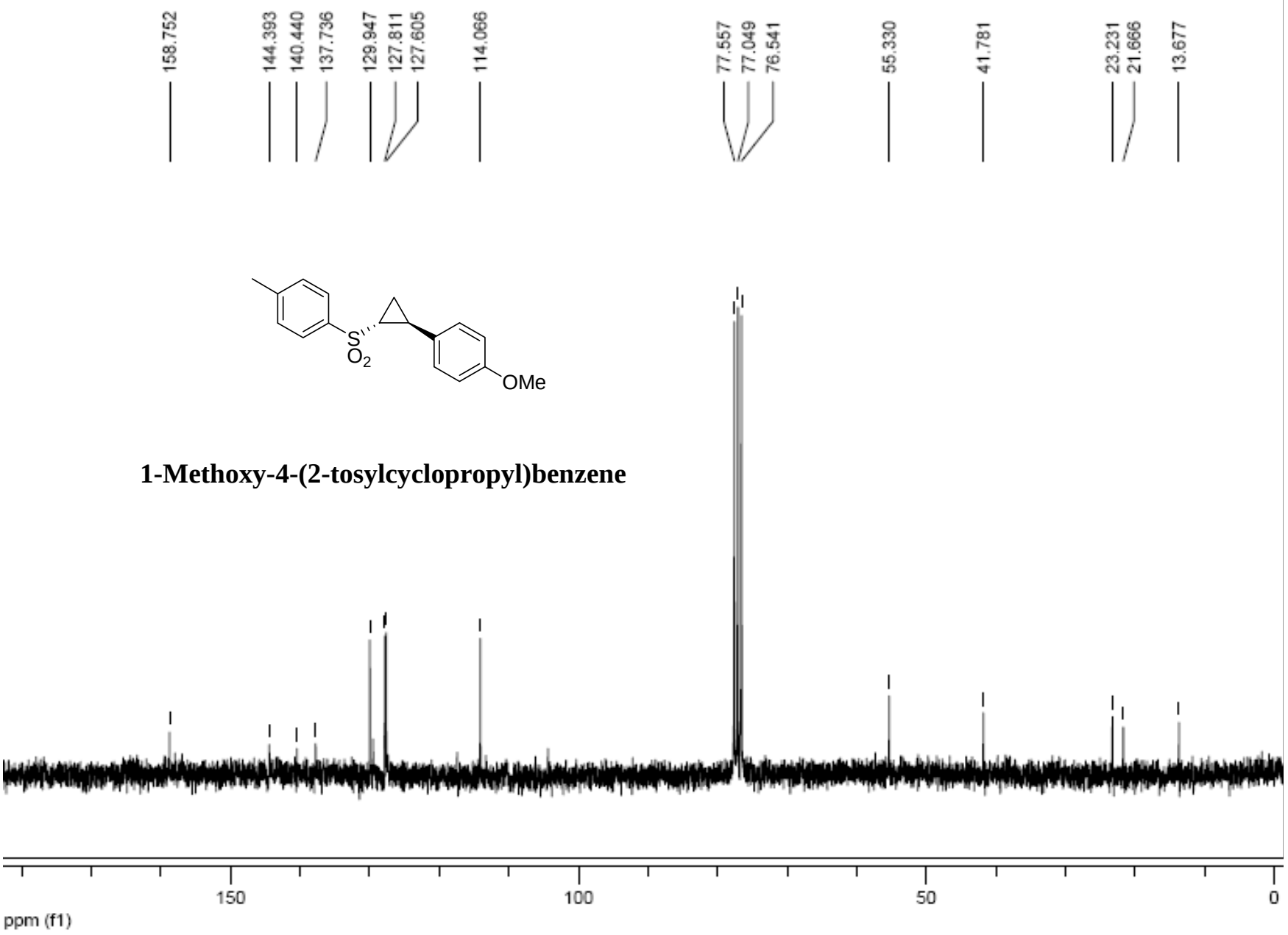


5: 192 nm, 4 nm
Results

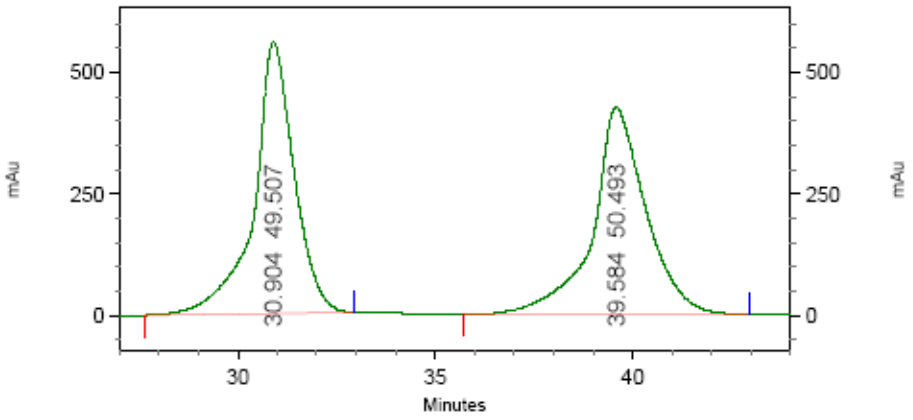
Pk #	Name	Retention Time	Area Percent
1		18.828	5.162
2		22.172	94.838
Totals			100.000

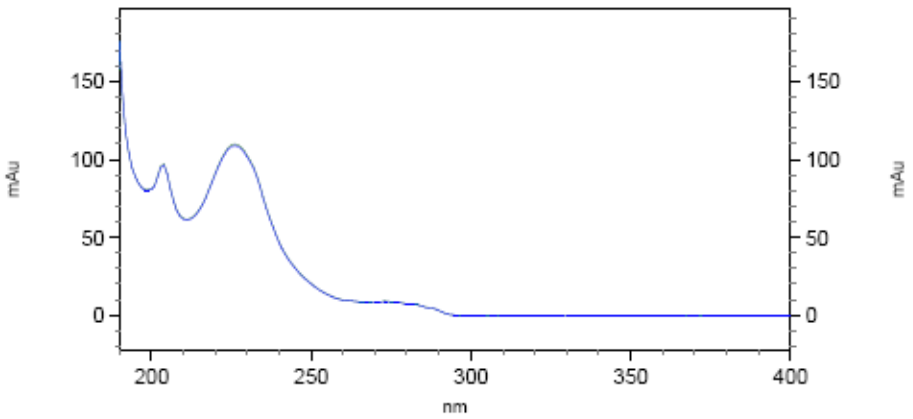


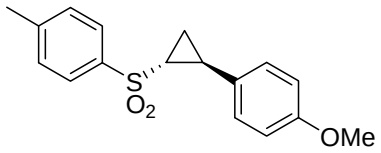
1-Methoxy-4-(2-tosylcyclopropyl)benzene



1-Methoxy-4-(2-tosylcyclopropyl)benzene



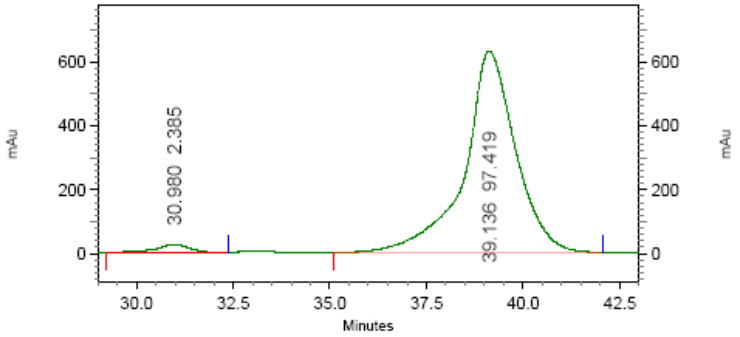


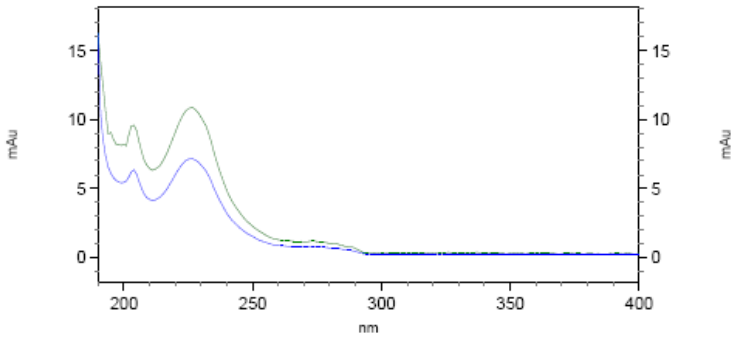


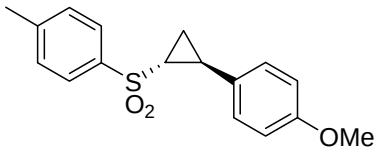
2: 225 nm, 4 nm

Results

Pk #	Name	Retention Time	Area Percent
1		30.904	49.507
2		39.584	50.493
Totals			100.000

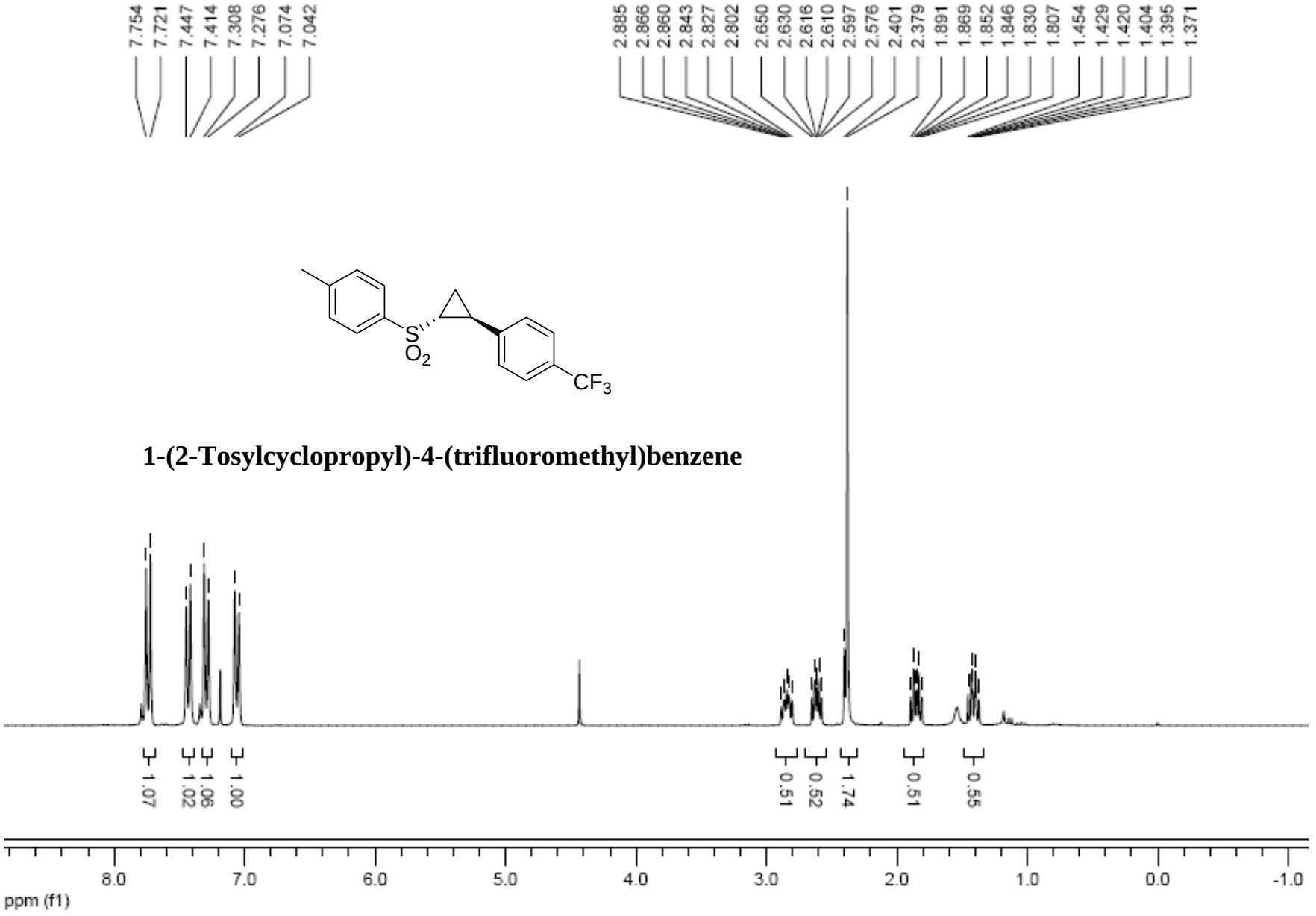




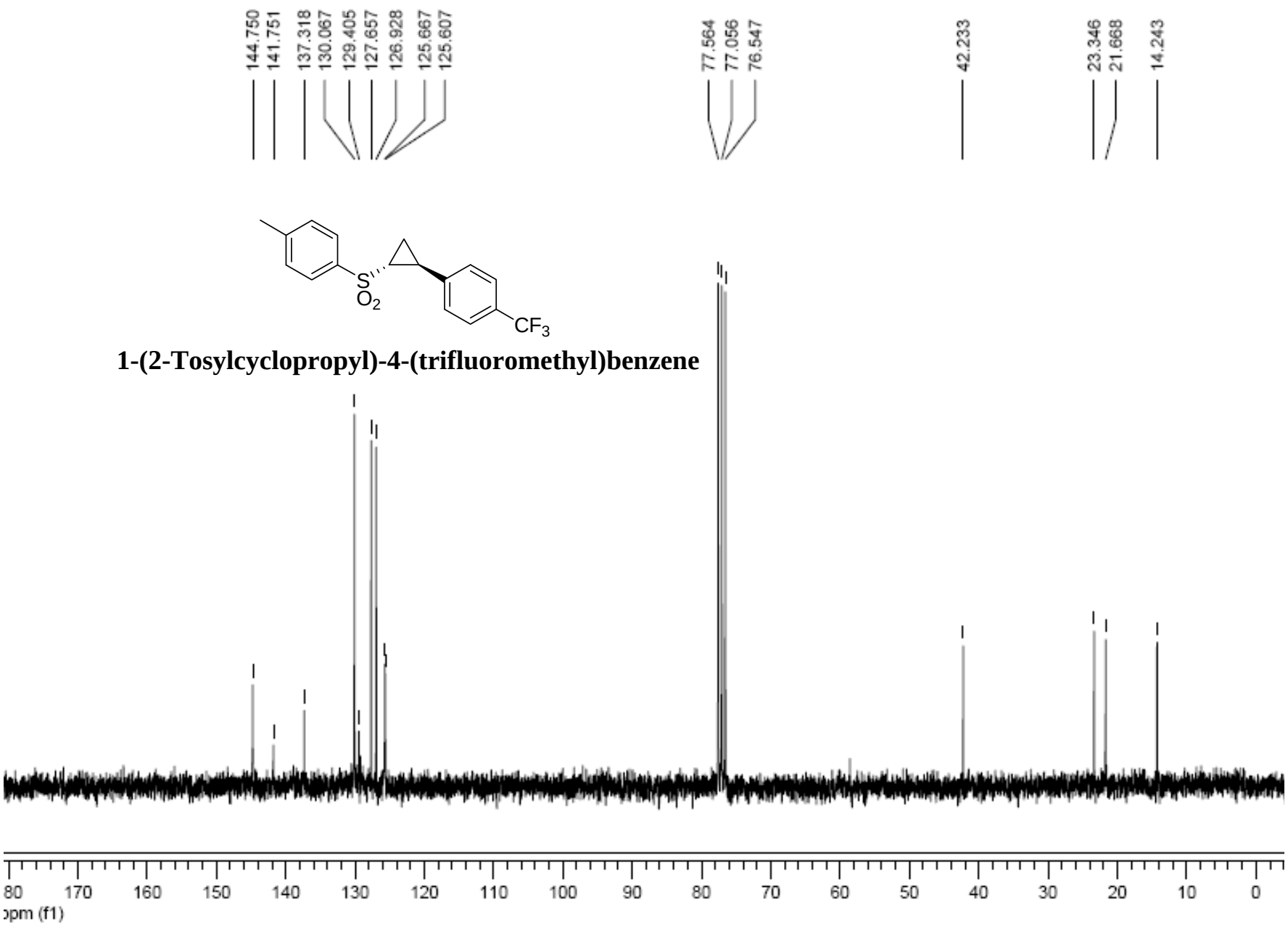


3: 203 nm, 4 nm
Results

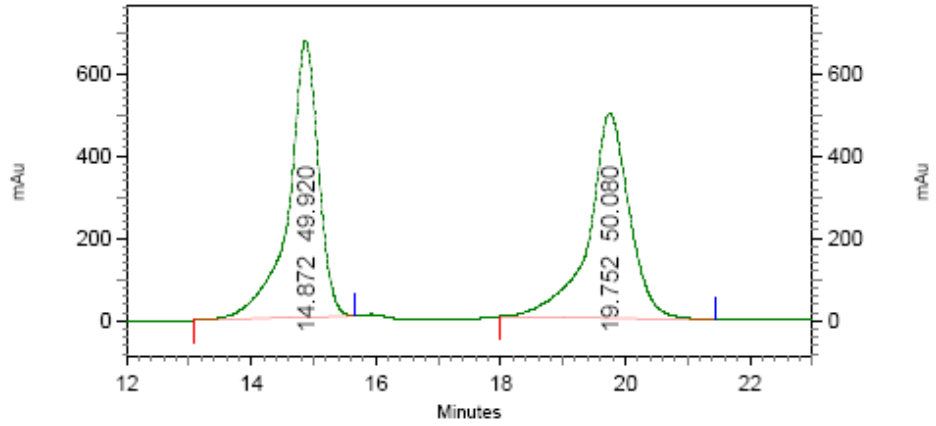
Pk #	Name	Retention Time	Area Percent
1		20.284	0.196
2		30.980	2.385
3		39.136	97.419
Totals			100.000

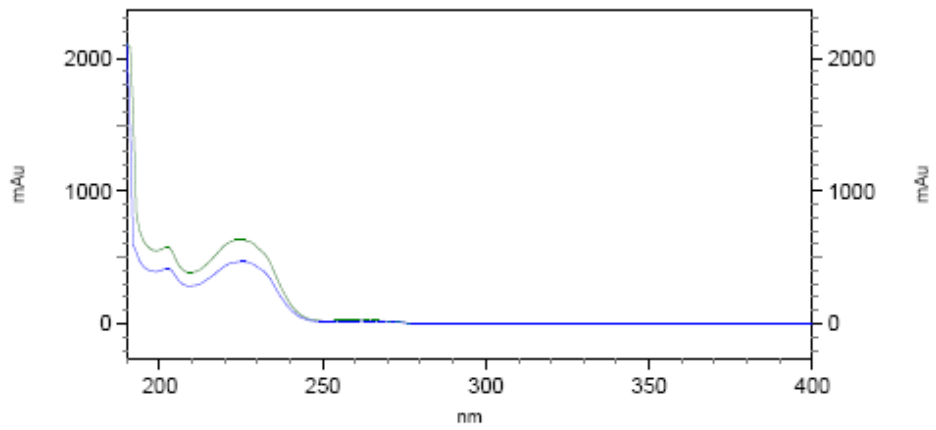


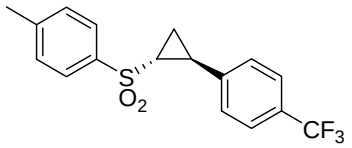
1-(2-Tosylcyclopropyl)-4-(trifluoromethyl)benzene



1-(2-Tosylcyclopropyl)-4-(trifluoromethyl)benzene

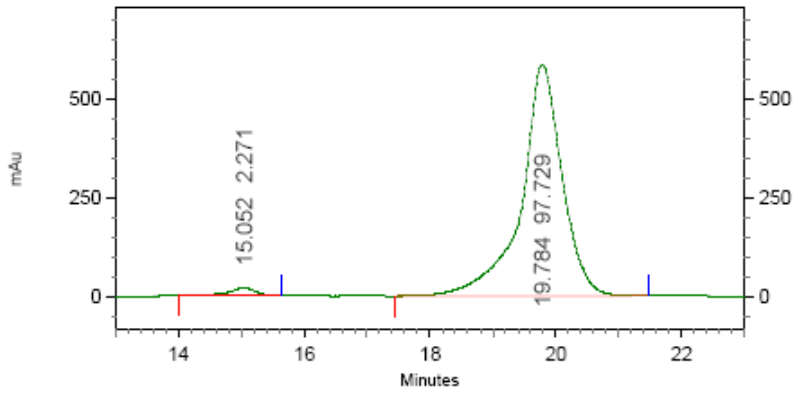


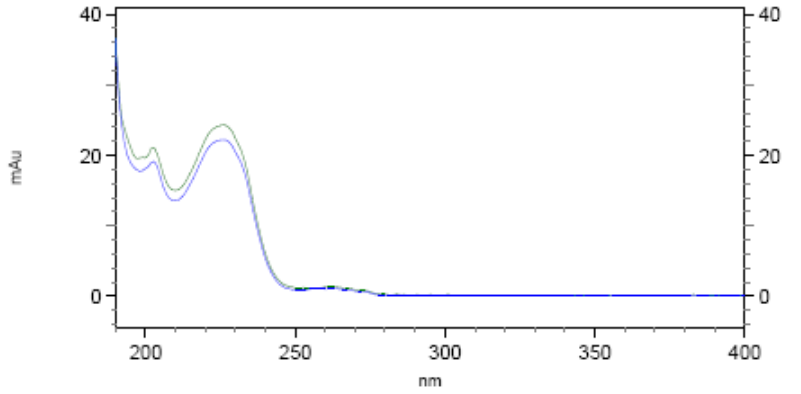


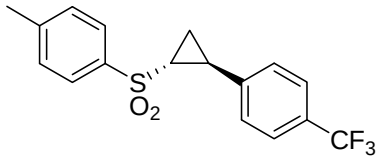


2: 225 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		14.872	49.920
2		19.752	50.080
Totals			100.000

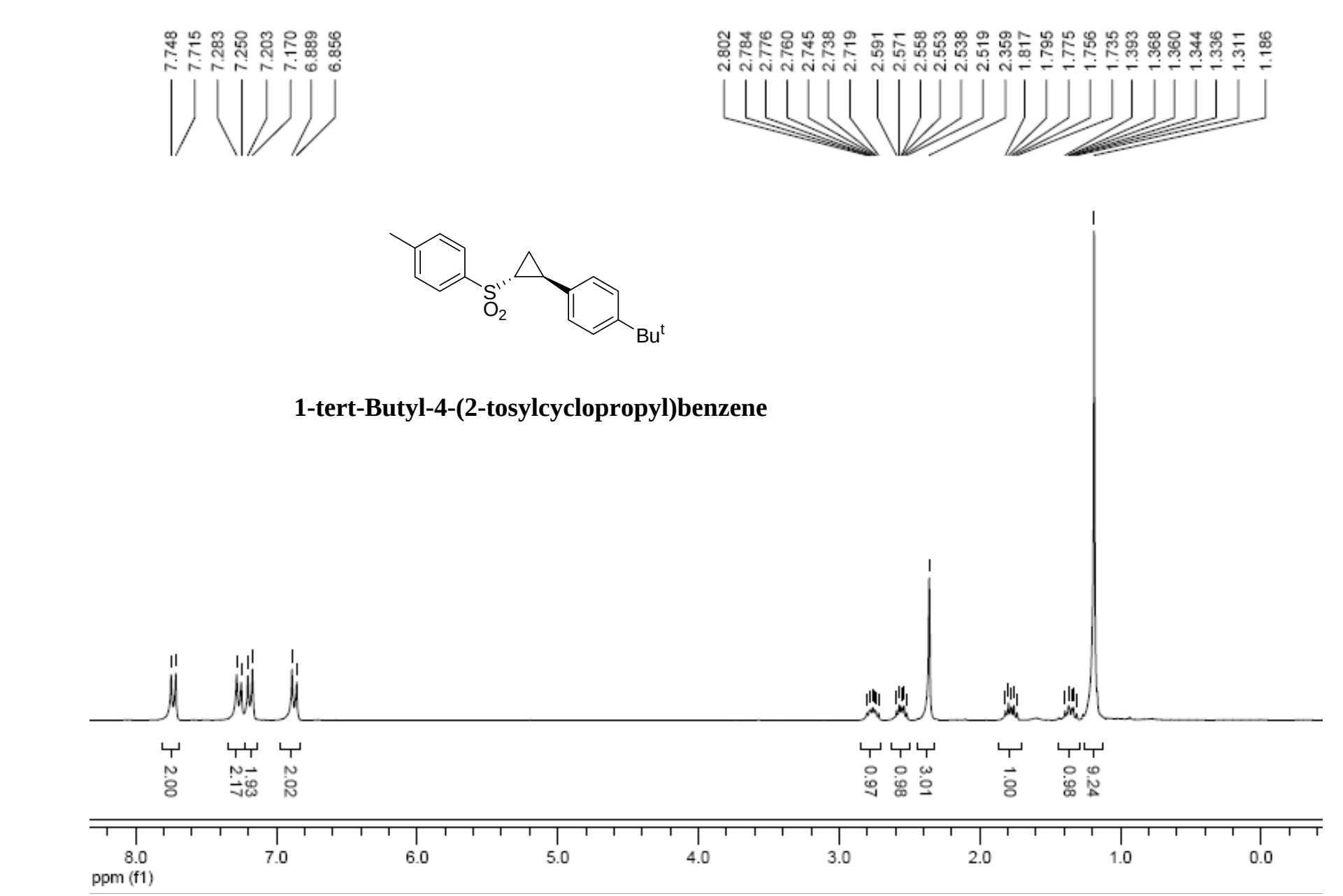




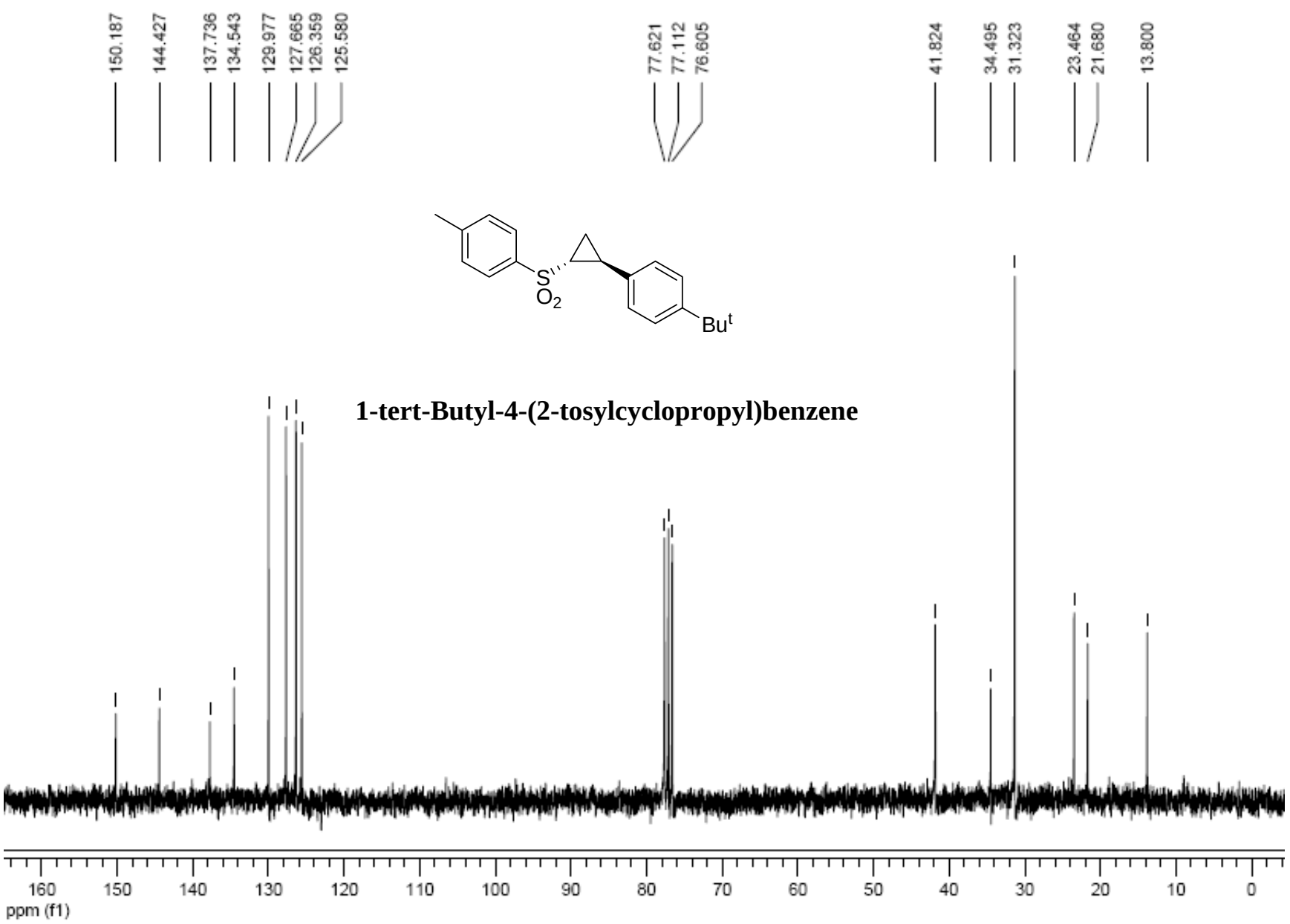


3: 203 nm, 4 nm
Results

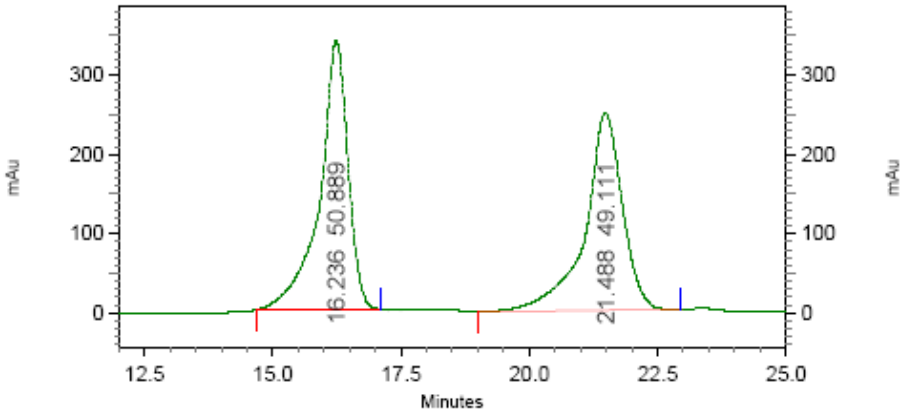
Pk #	Name	Retention Time	Area Percent
1		15.052	2.271
2		19.784	97.729
Totals			100.000

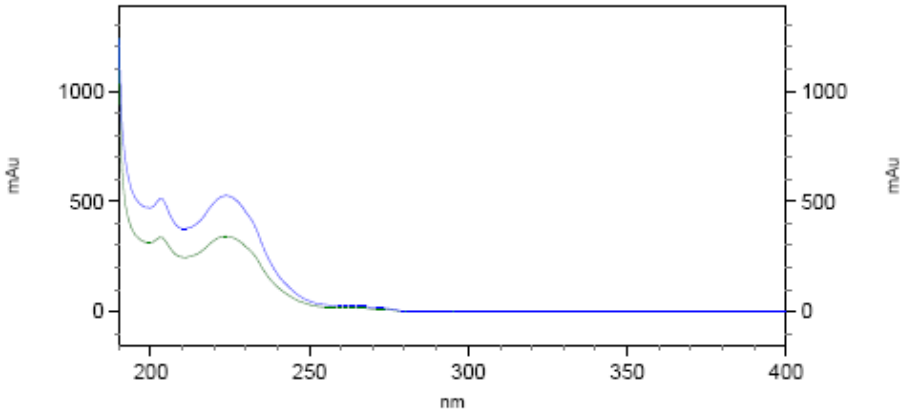


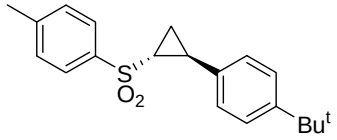
1-tert-Butyl-4-(2-tosylcyclopropyl)benzene



1-tert-Butyl-4-(2-tosylcyclopropyl)benzene



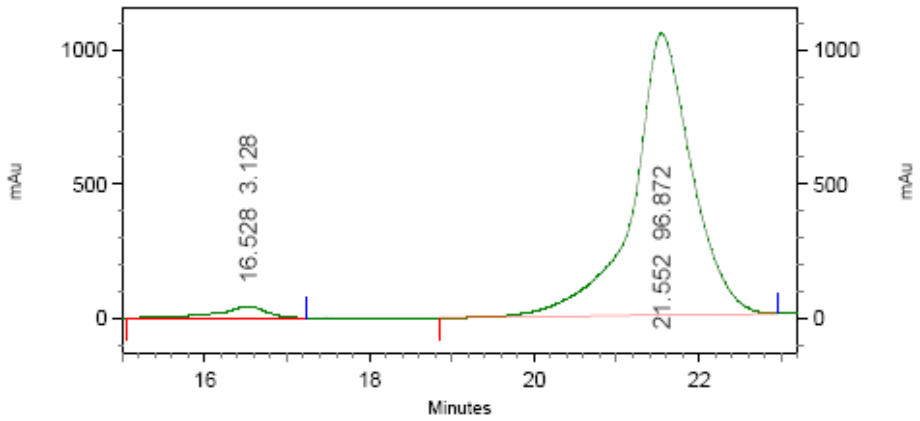


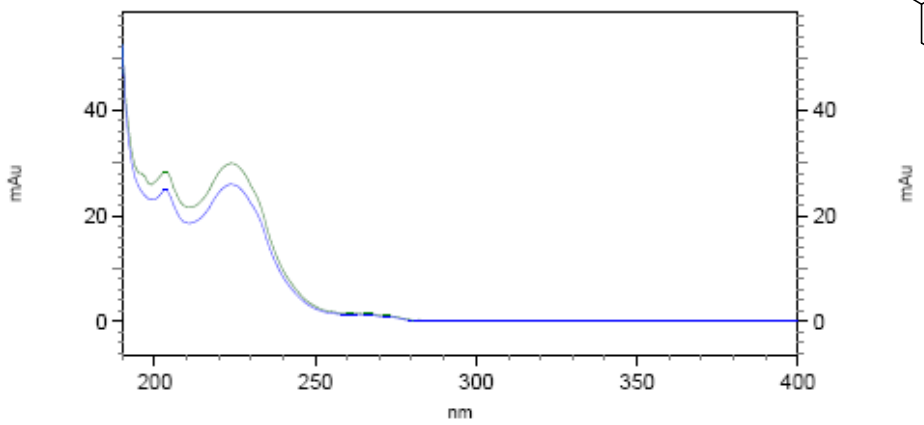


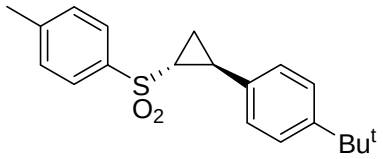
2: 225 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		16.236	50.889
2		21.488	49.111

Totals			100.000

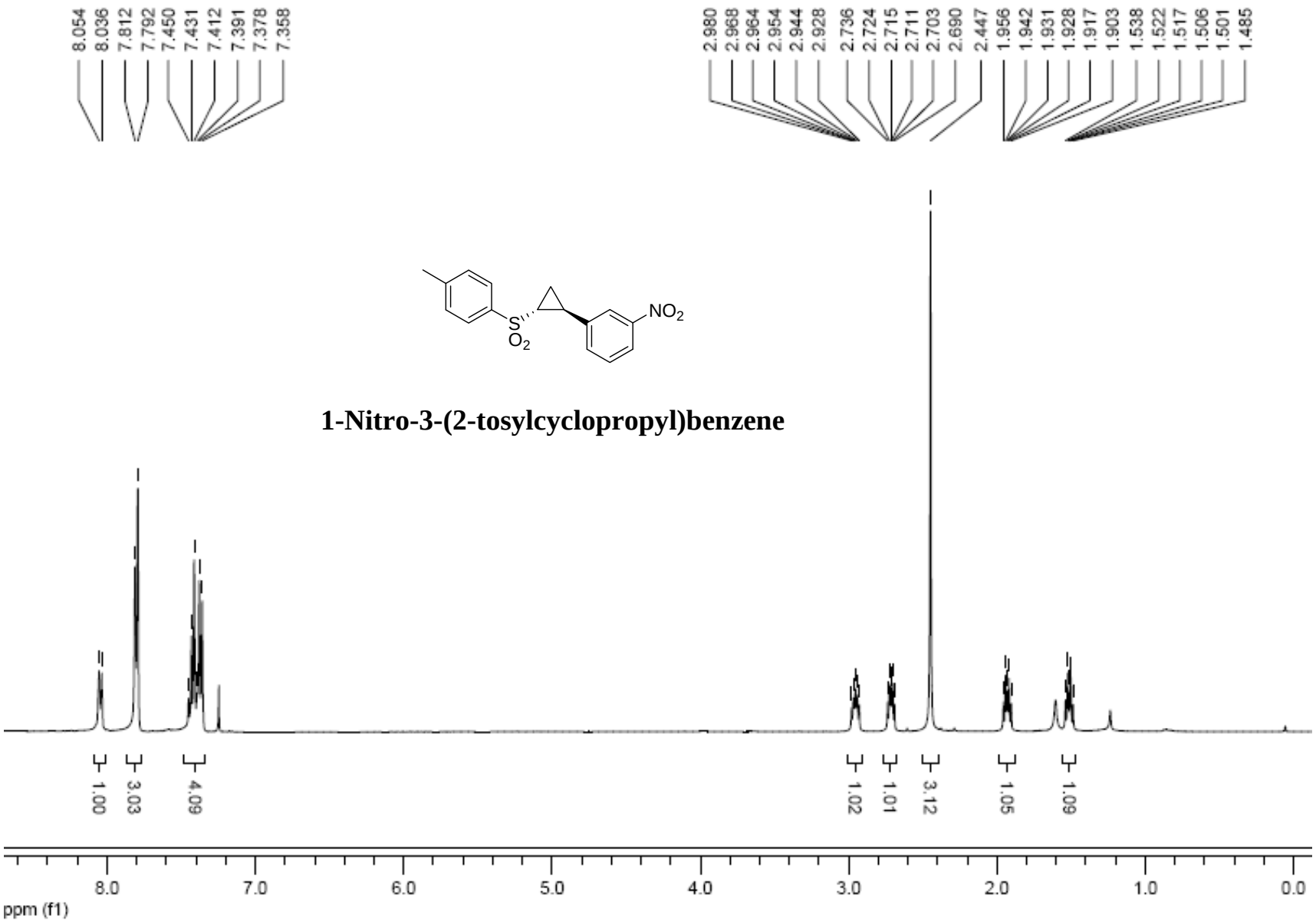




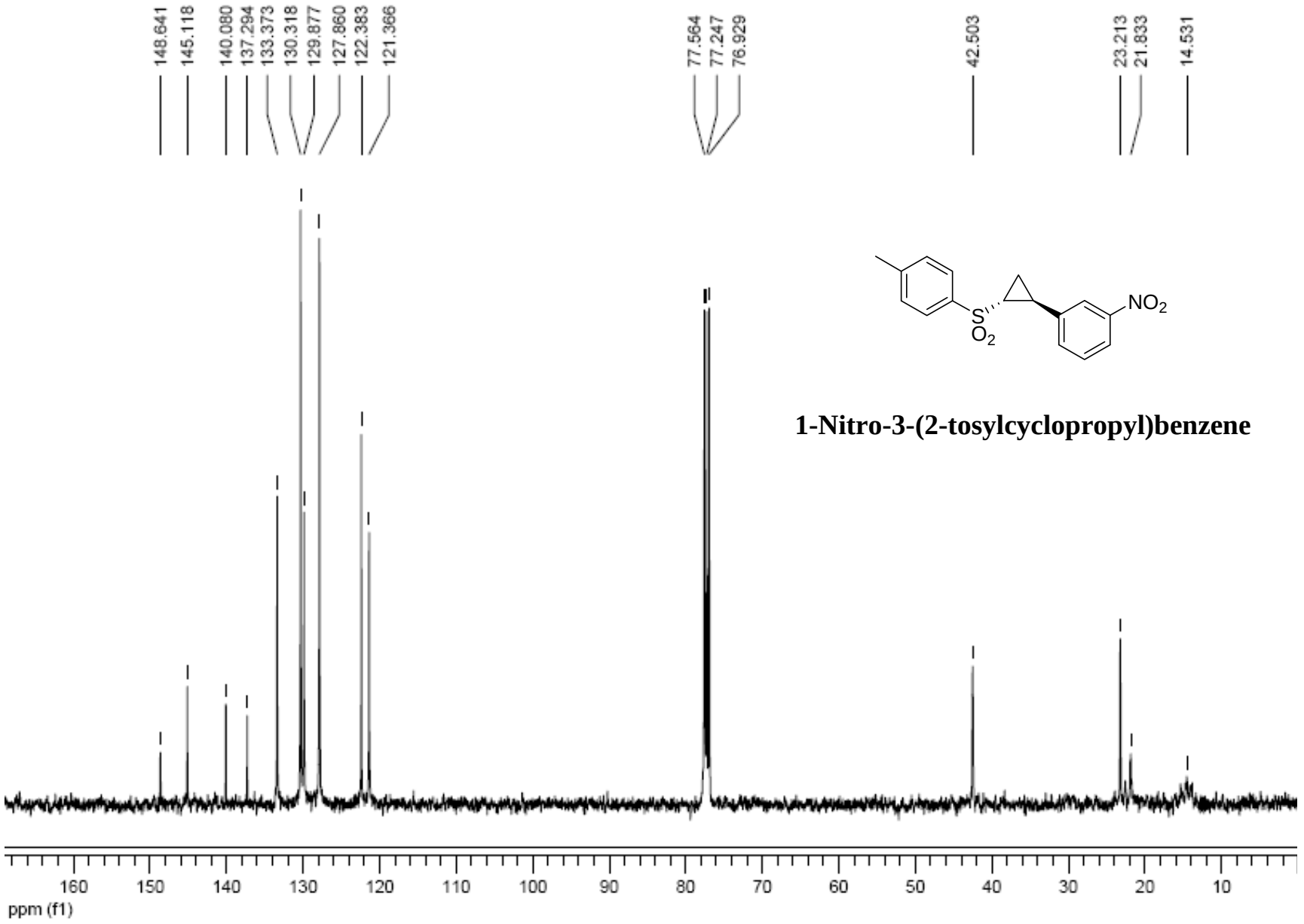


3: 203 nm, 4 nm
Results

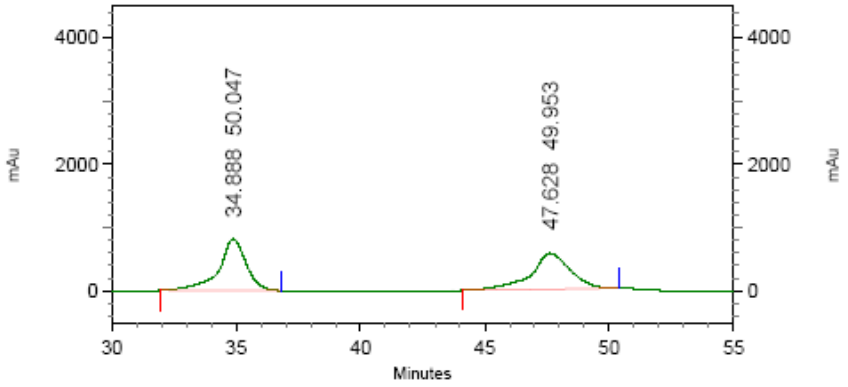
Pk #	Name	Retention Time	Area Percent
1		16.528	3.128
2		21.552	96.872
Totals			100.000

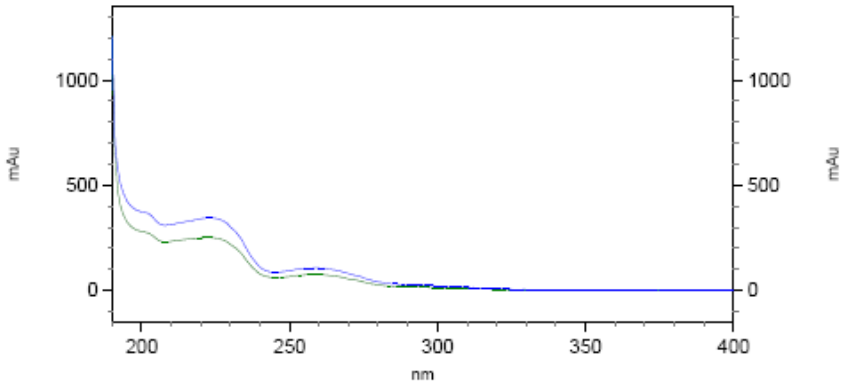


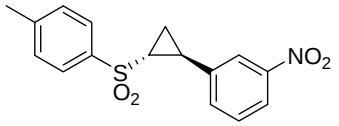
1-Nitro-3-(2-tosylcyclopropyl)benzene



1-Nitro-3-(2-tosylcyclopropyl)benzene

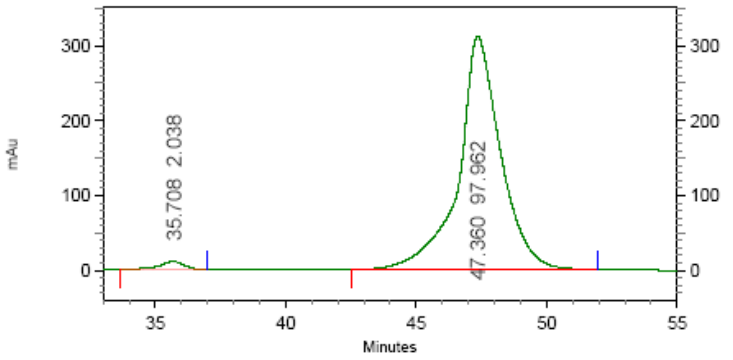


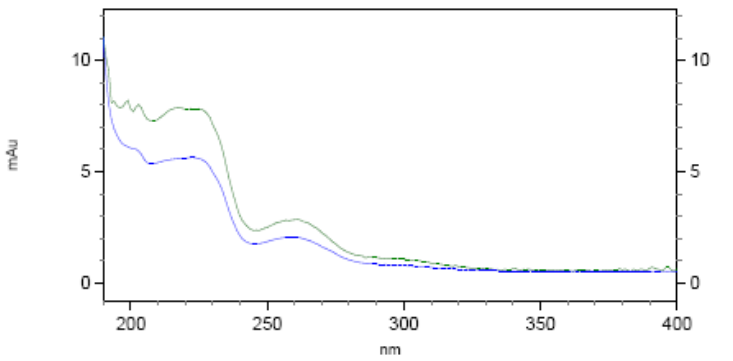


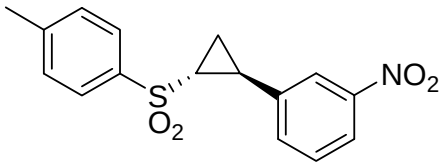


5: 240 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		34.888	50.047
2		47.628	49.953
Totals			100.000

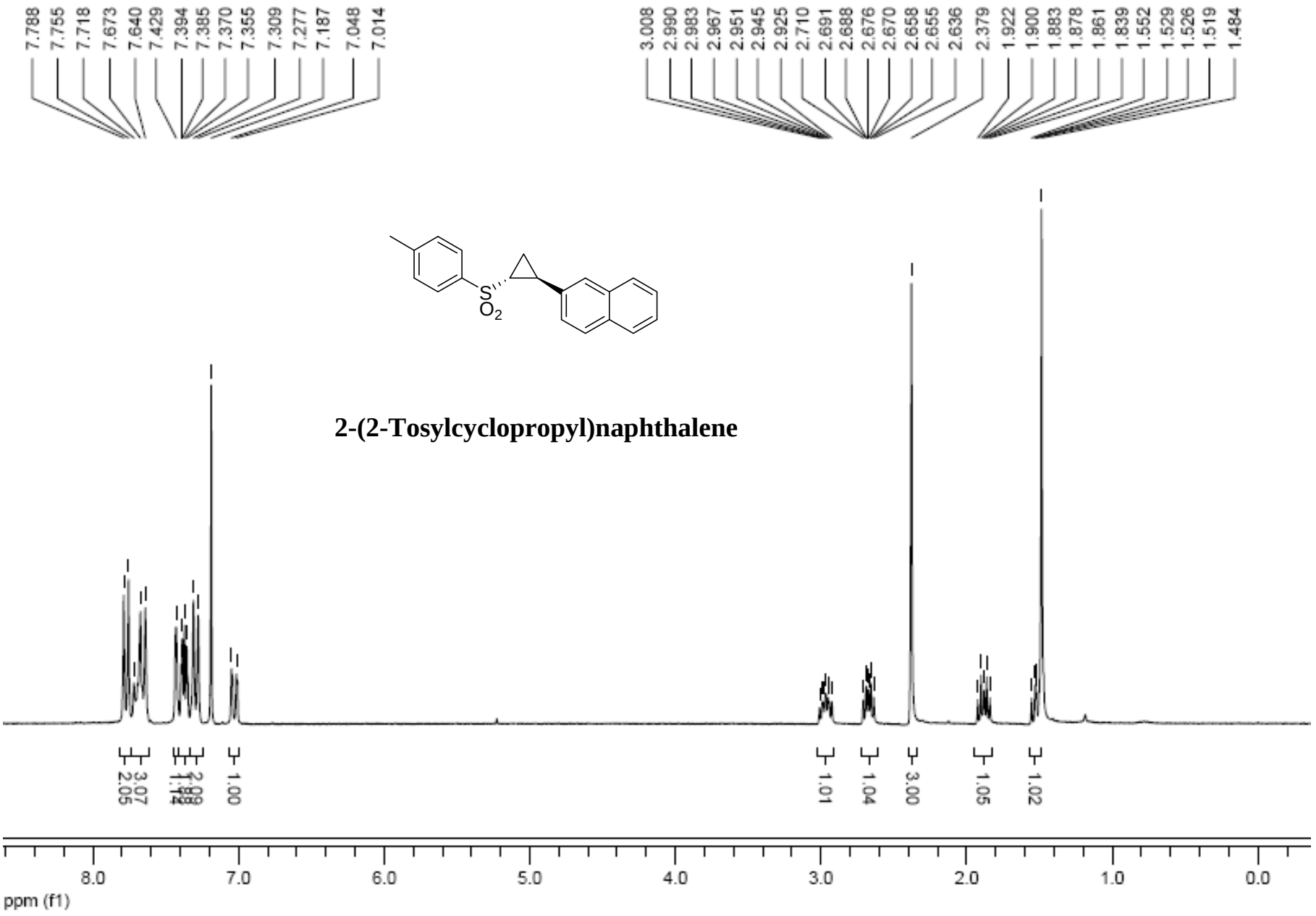




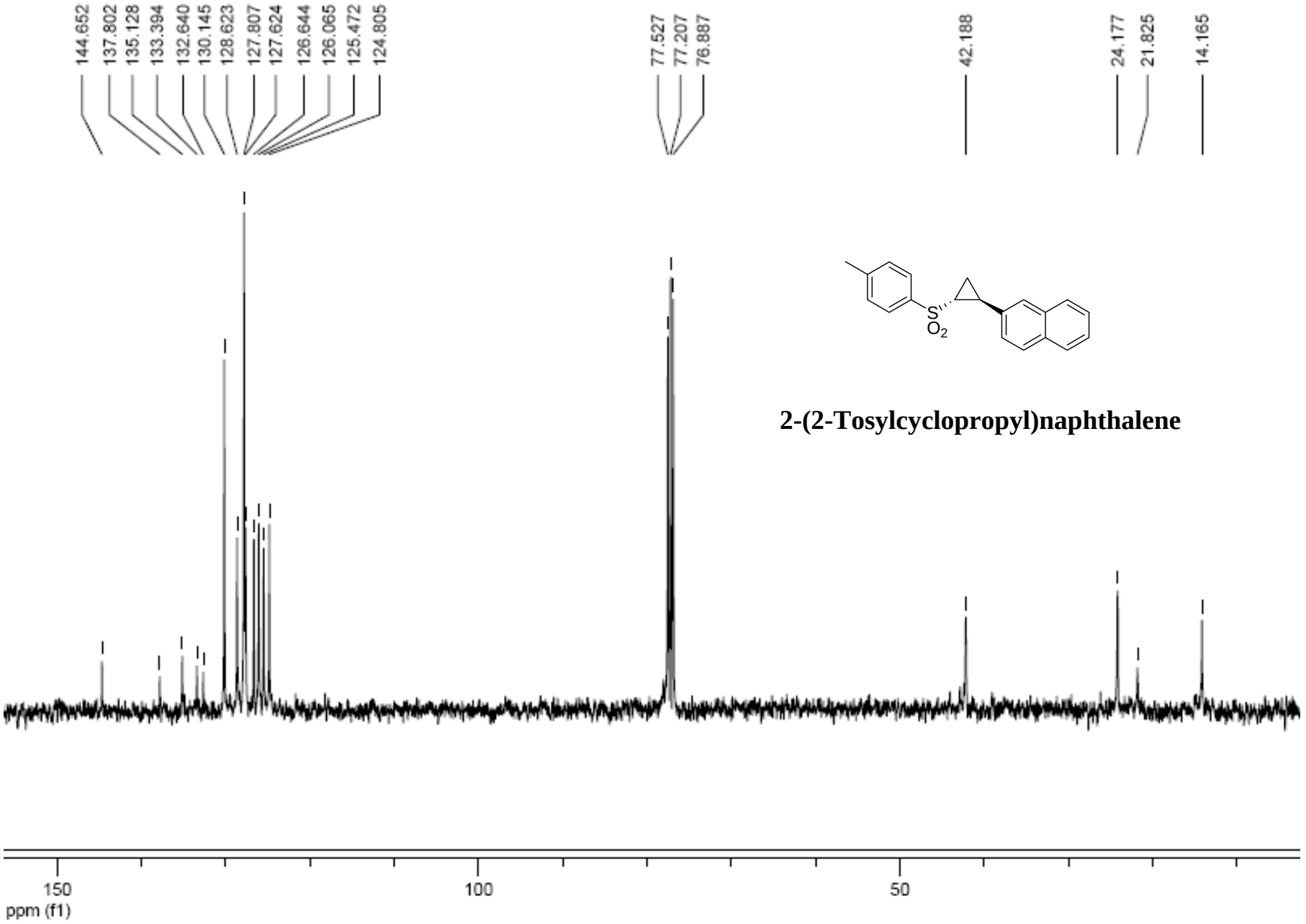


2: 225 nm, 4 nm
Results

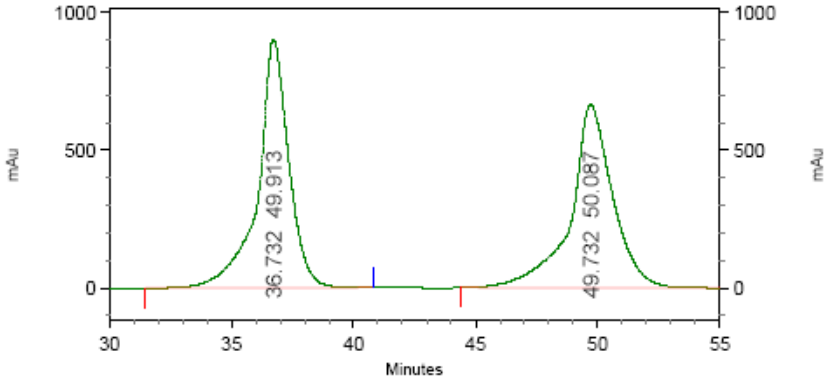
Pk #	Name	Retention Time	Area Percent
1		35.708	2.038
2		47.360	97.962
Totals			100.000

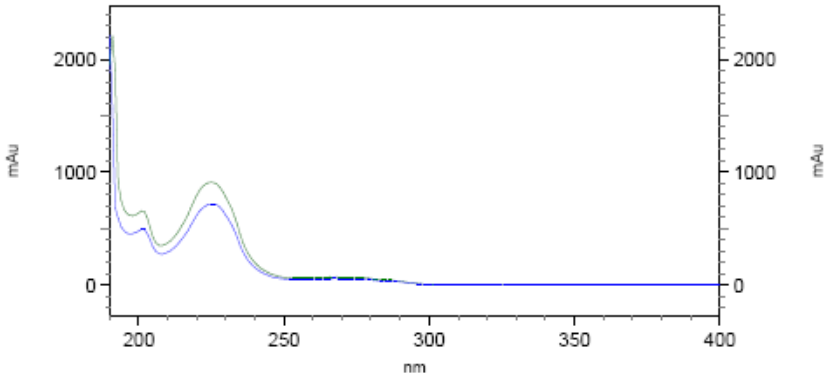


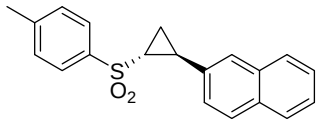
2-(2-Tosylcyclopropyl)naphthalene



2-(2-Tosylcyclopropyl)naphthalene

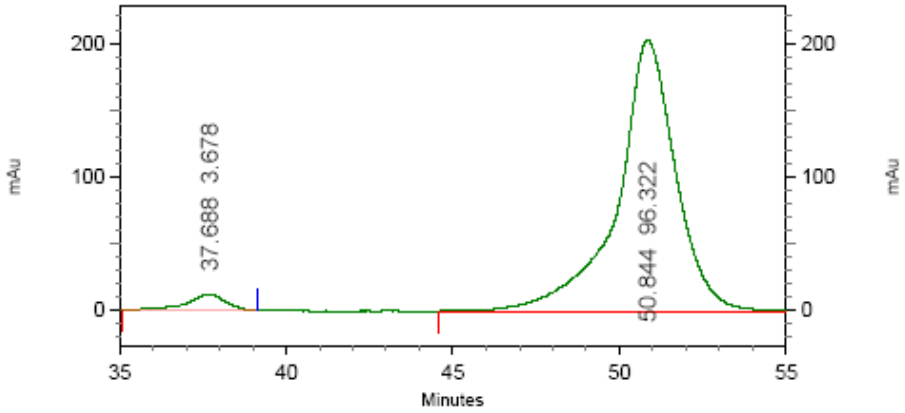


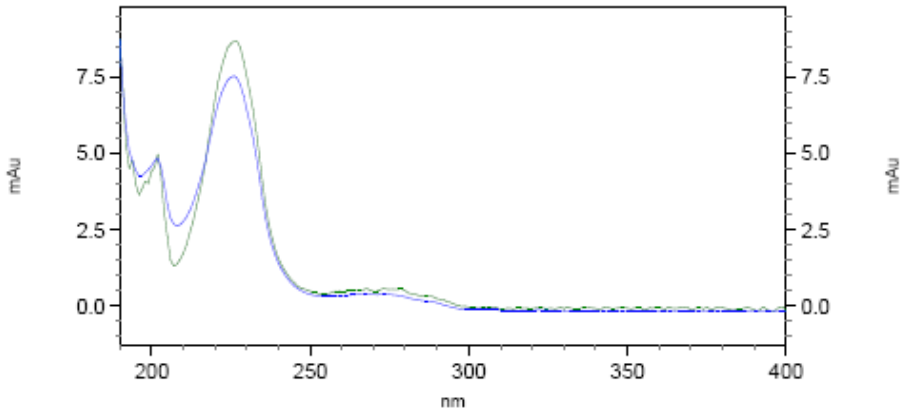


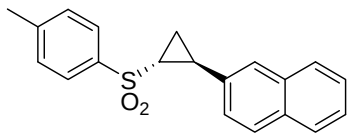


2: 225 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		36.732	49.913
2		49.732	50.087
Totals			100.000



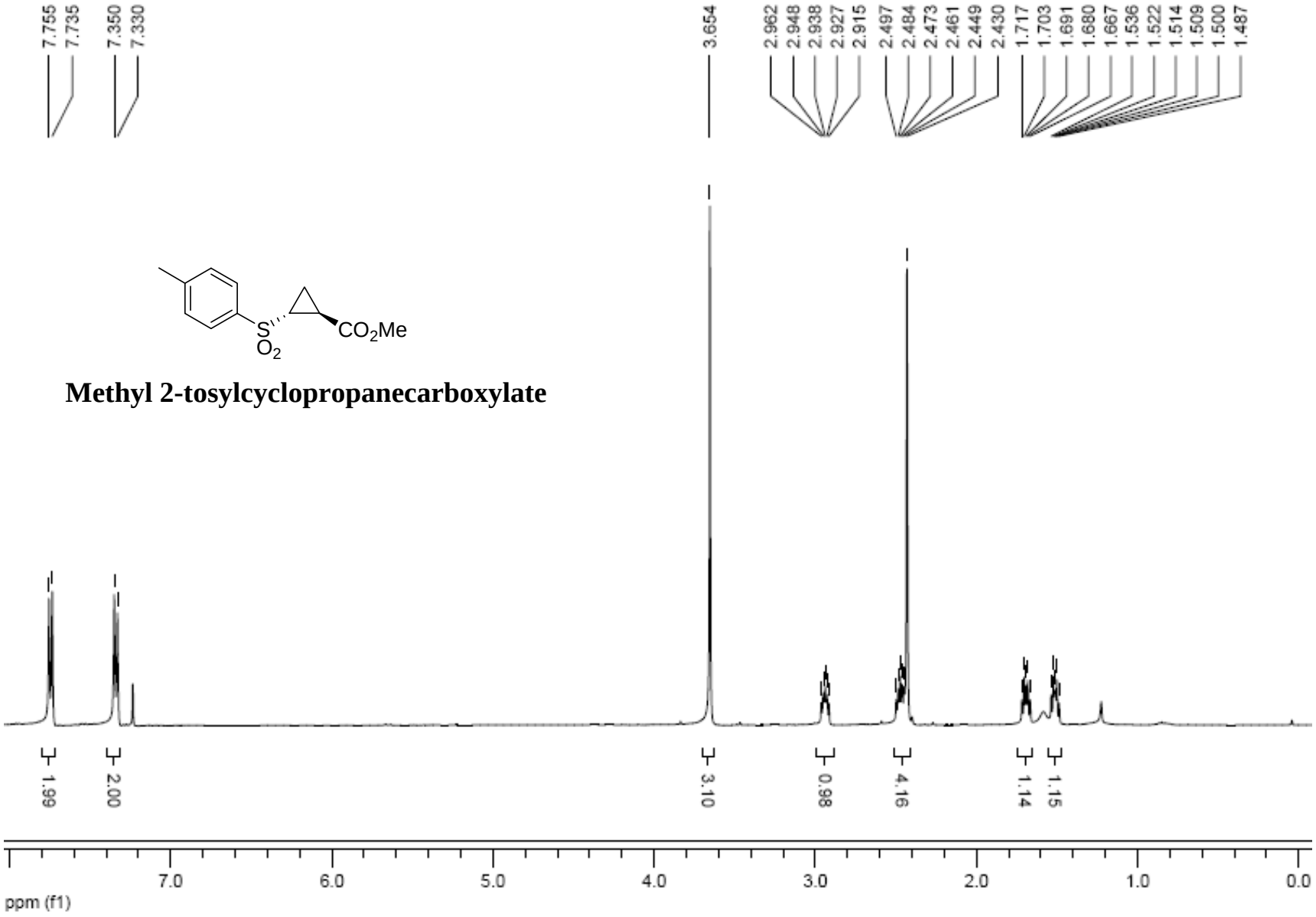




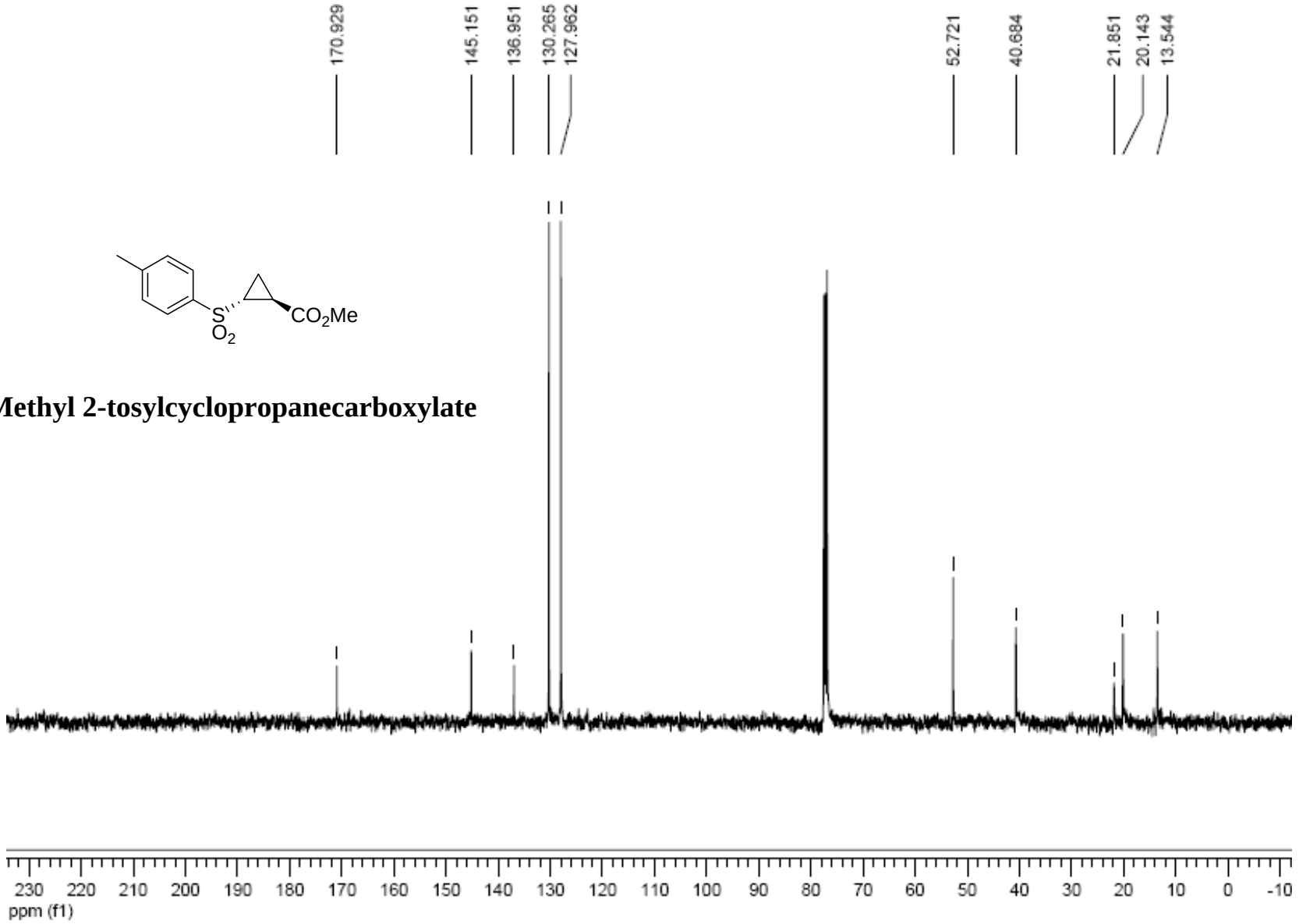
2: 225 nm, 4 nm

Results

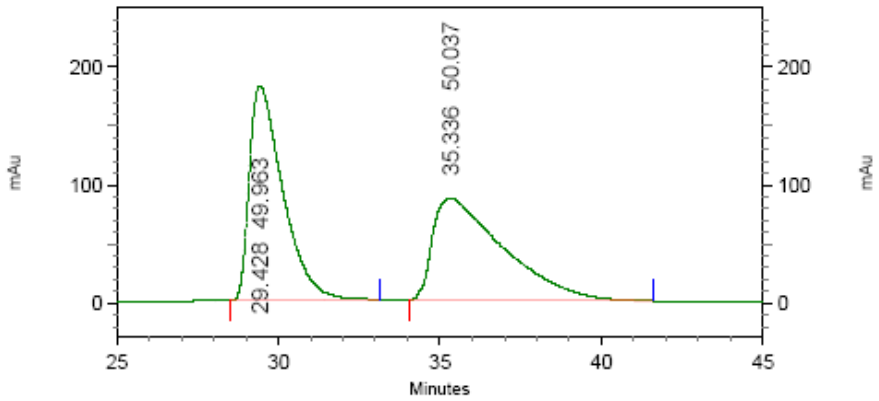
Pk #	Name	Retention Time	Area Percent
1		37.688	3.678
2		50.844	96.322
Totals			100.000

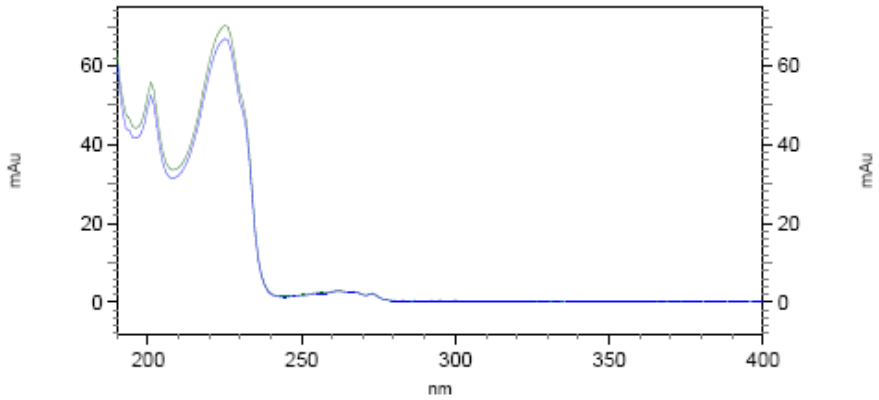


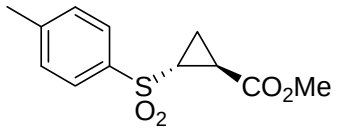
Methyl 2-tosylcyclopropanecarboxylate



Methyl 2-tosylcyclopropanecarboxylate



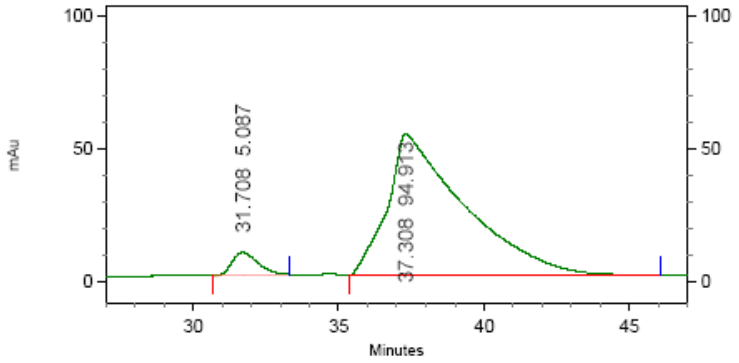


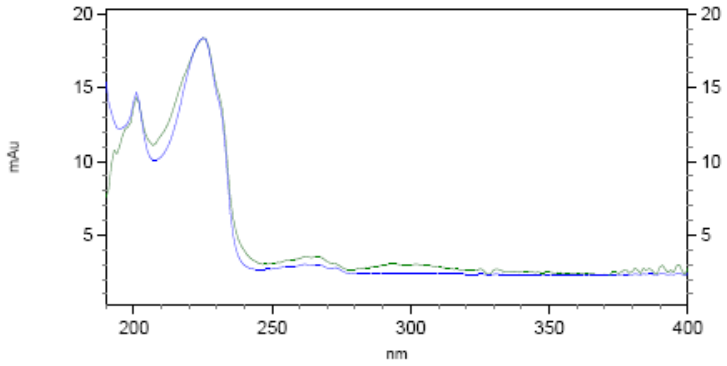


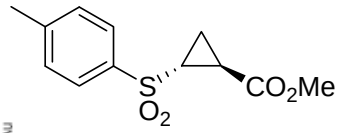
2: 225 nm, 4 nm

Results

Pk #	Name	Retention Time	Area Percent
1		29.428	49.963
2		35.336	50.037
Totals			100.000



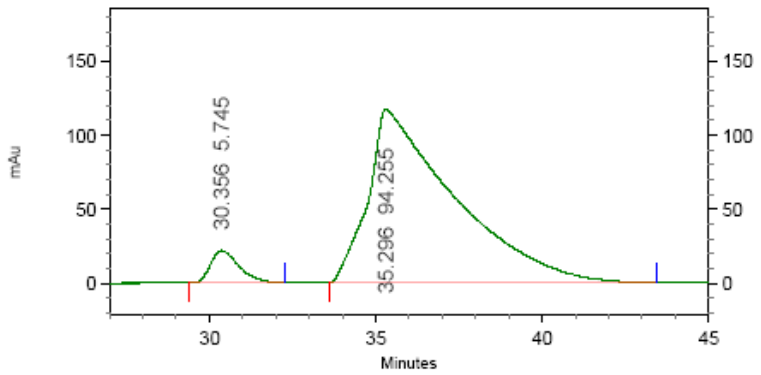


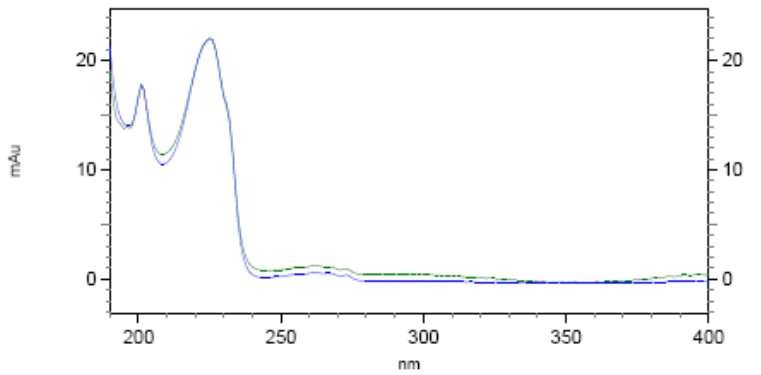


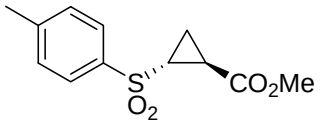
2: 225 nm, 4 nm

Results

Name	Retention Time	Area Percent	Pk #
	31.708	5.087	1
	37.308	94.913	2
Totals		100.000	

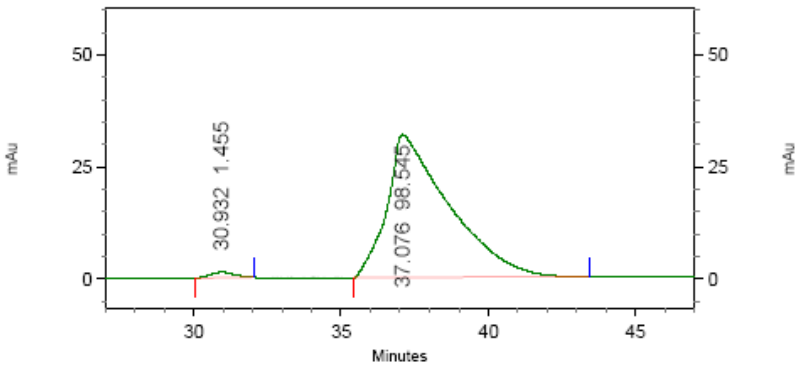


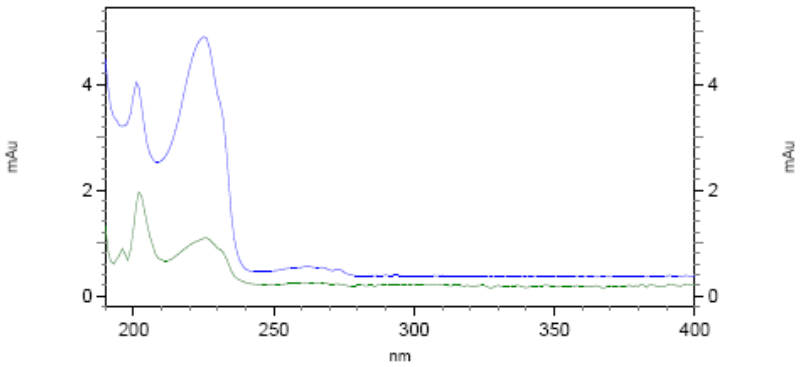


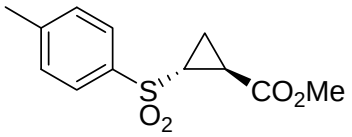


2: 225 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		30.356	5.745
2		35.296	94.255
Totals			100.000



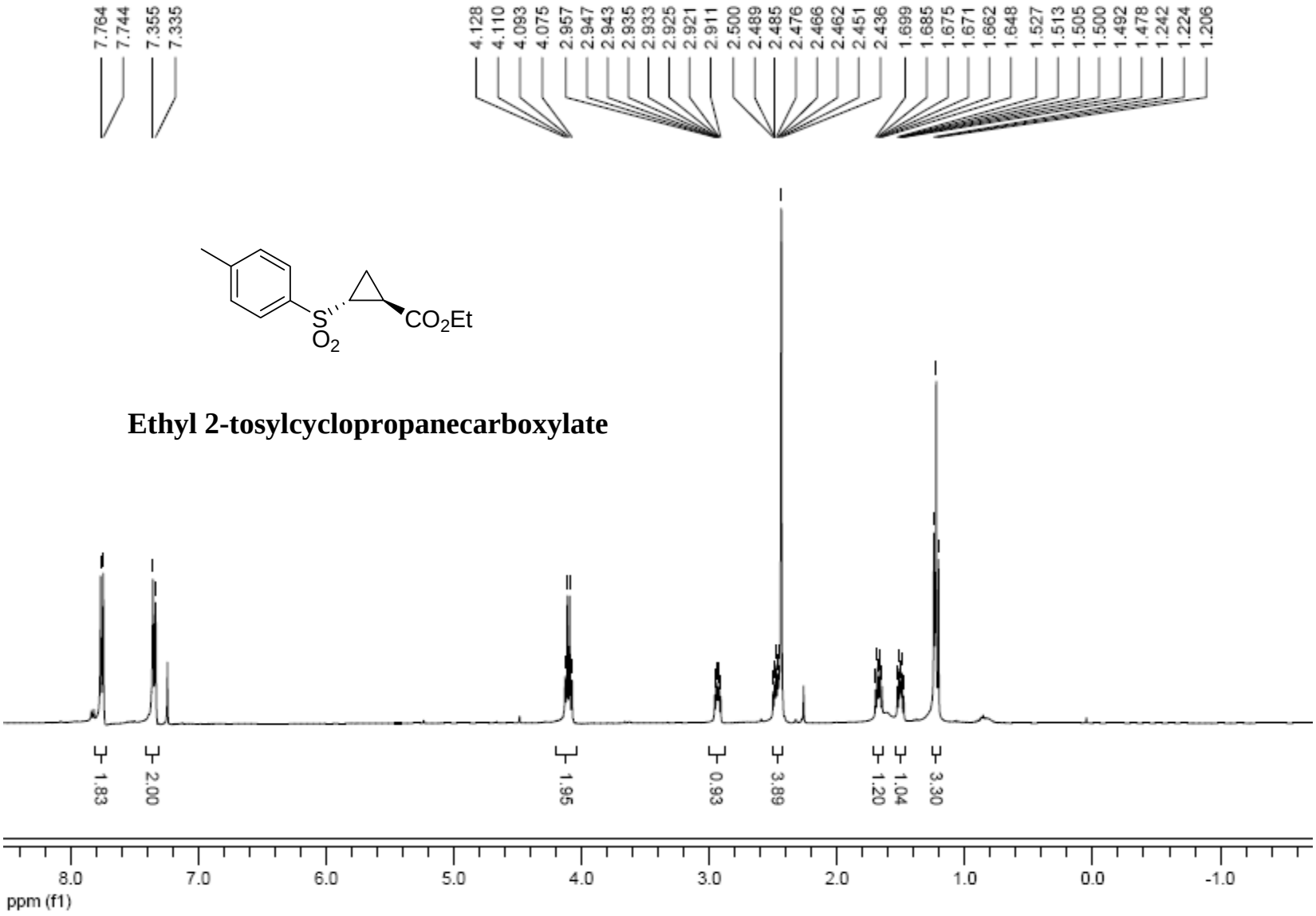




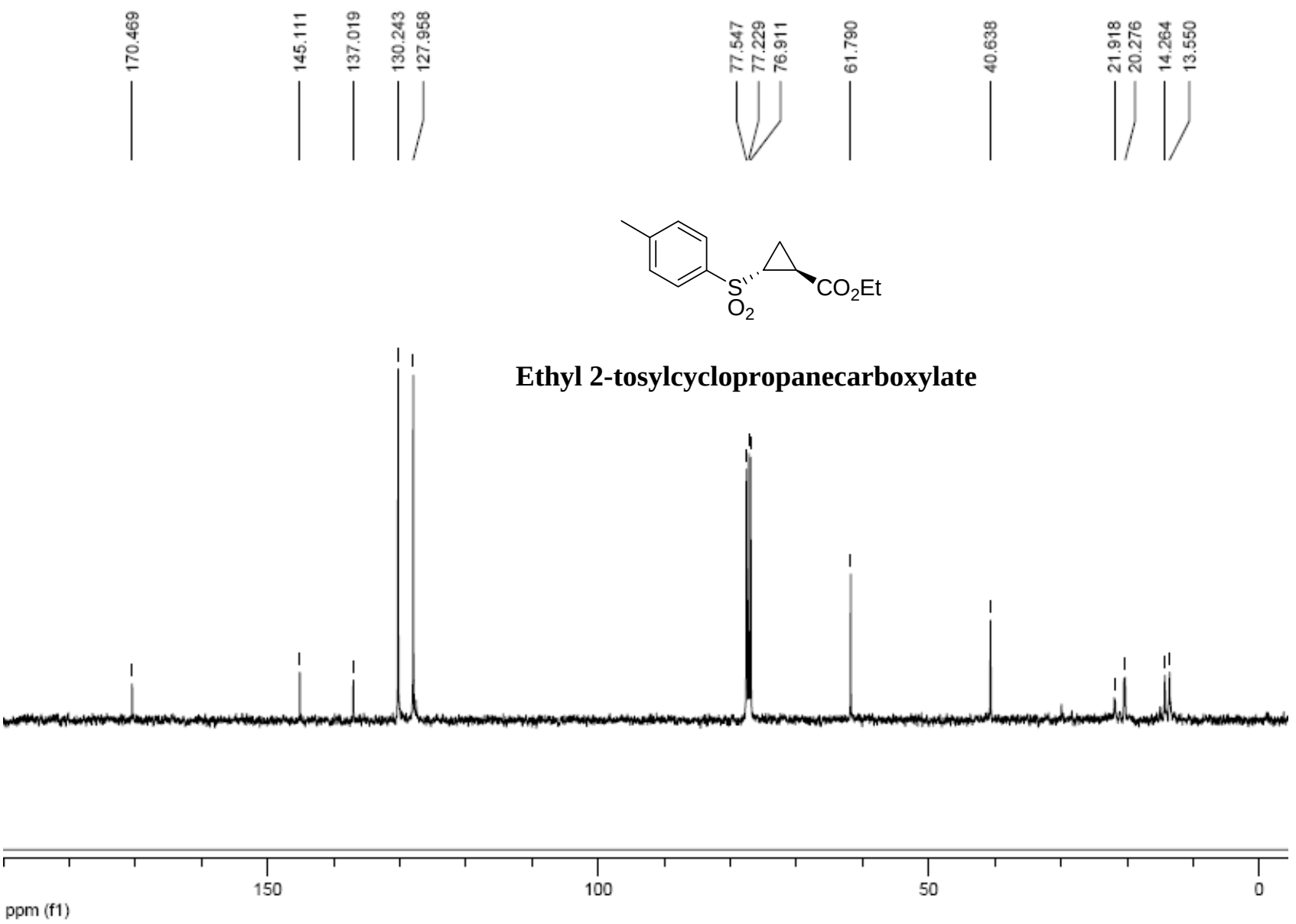
2: 225 nm, 4 nm

Results

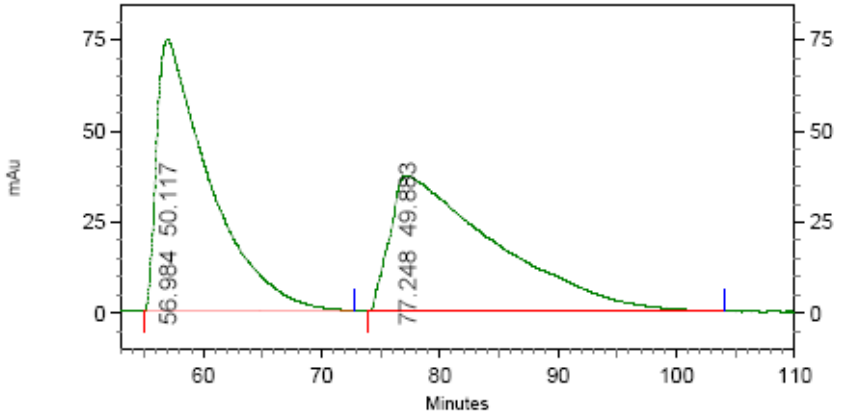
Name	Retention Time	Area Percent	Pk #
	30.932	1.455	1
	37.076	98.545	2
Totals		100.000	

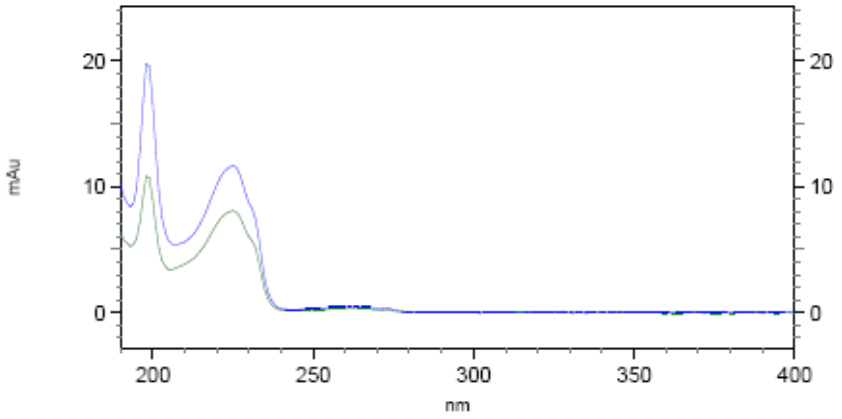


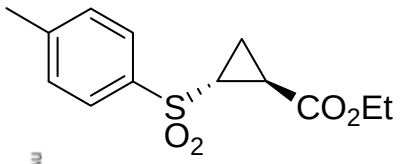
Ethyl 2-tosylcyclopropanecarboxylate



Ethyl 2-tosylcyclopropanecarboxylate

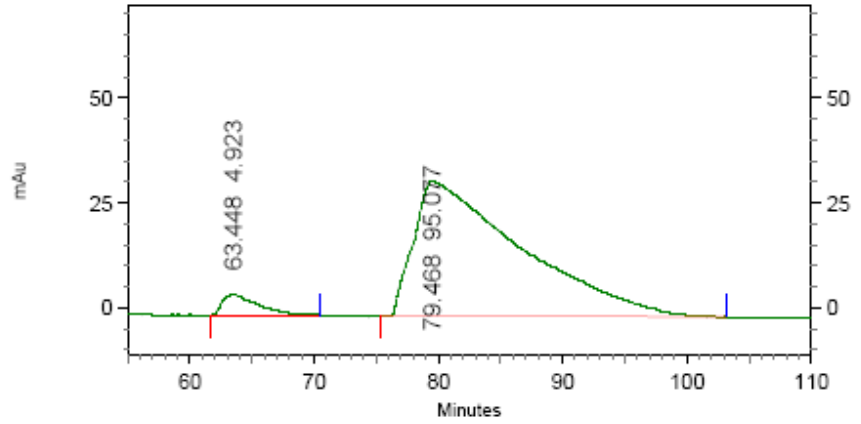


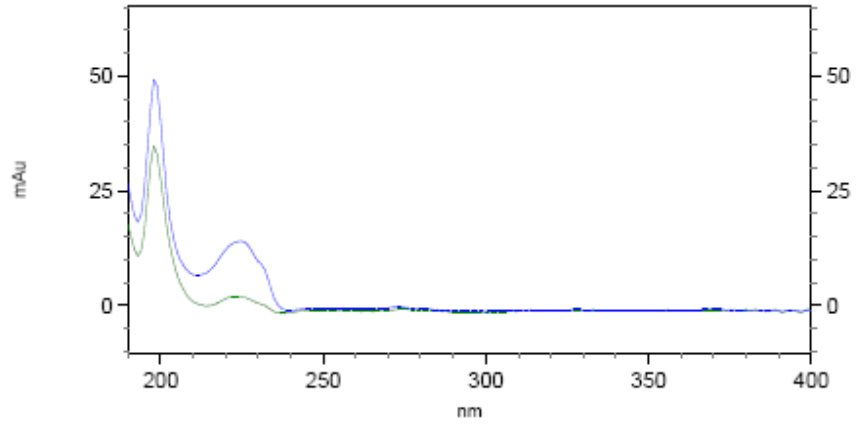


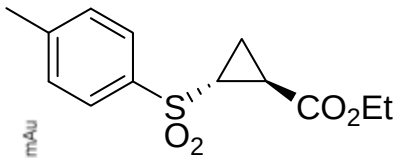


2: 225 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		56.984	50.117
2		77.248	49.883
Totals			100.000



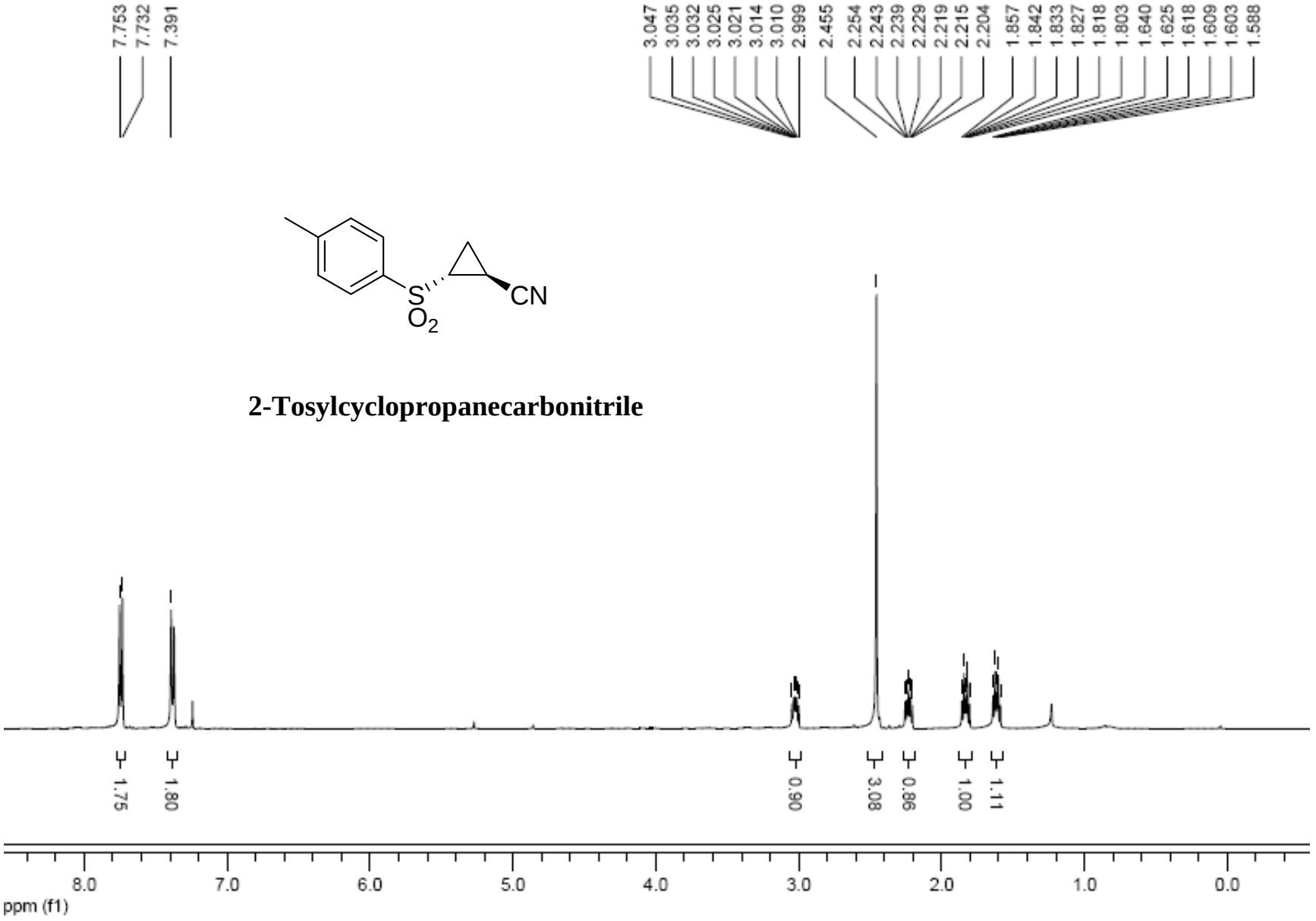




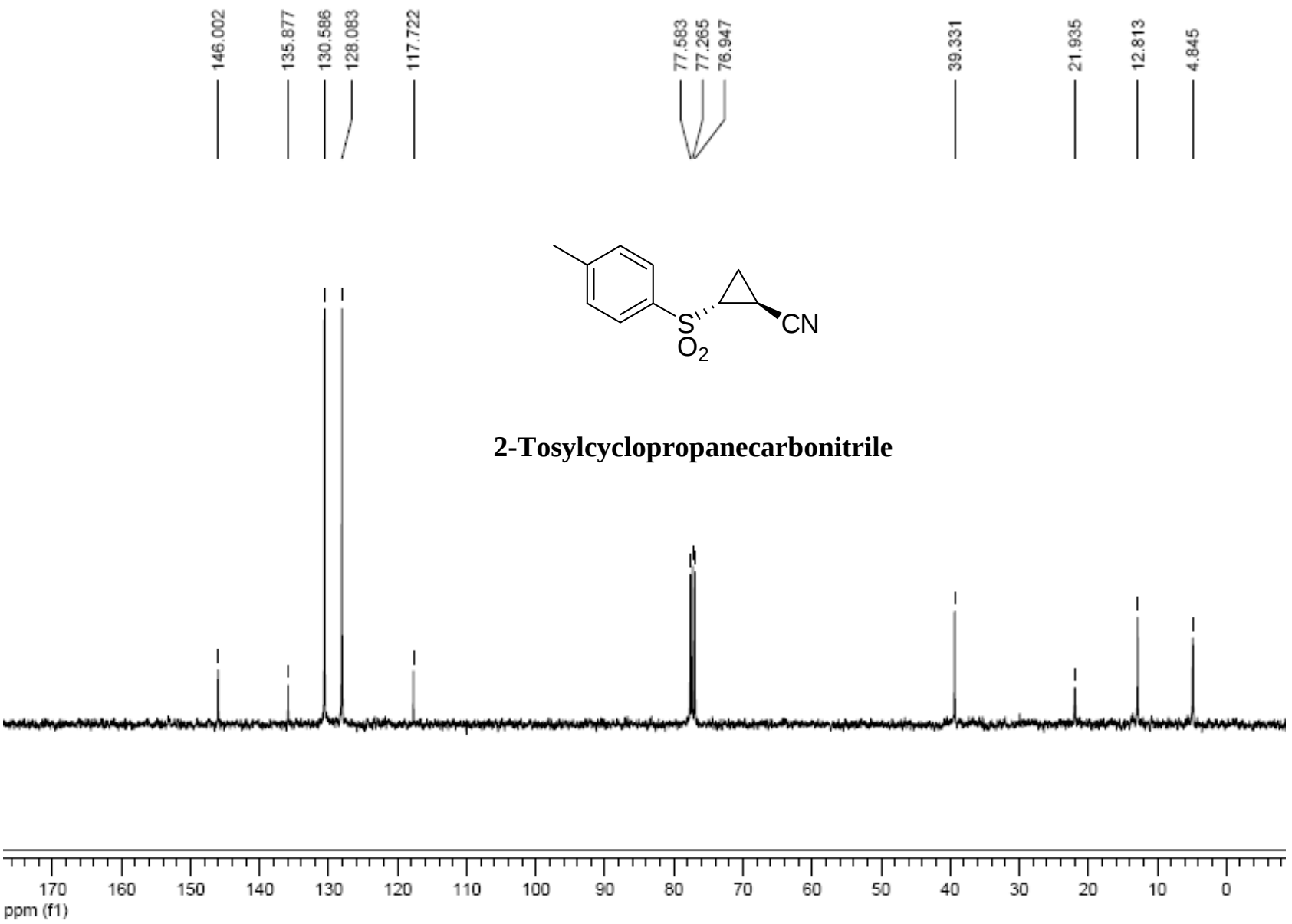
2: 225 nm, 4 nm

Results

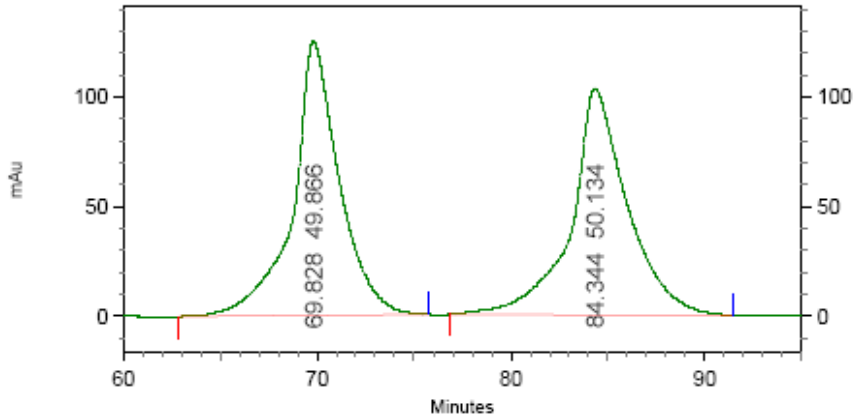
Pk #	Name	Retention Time	Area Percent
1		63.448	4.923
2		79.468	95.077
Totals			100.000

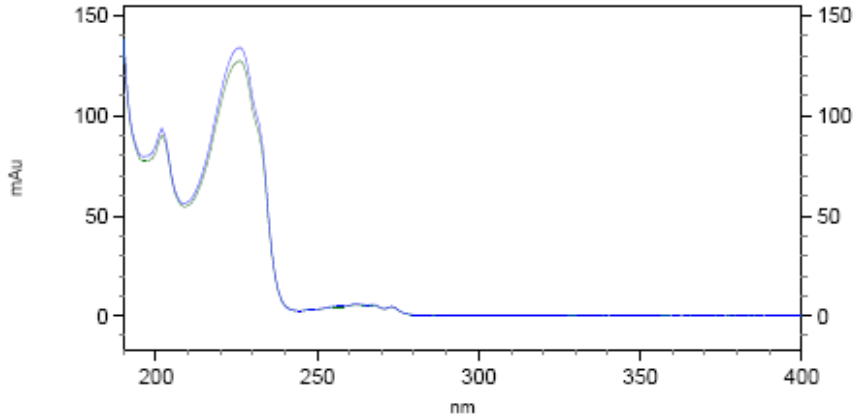


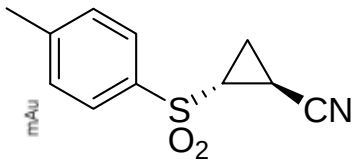
2-Tosylcyclopropanecarbonitrile



2-Tosylcyclopropanecarbonitrile

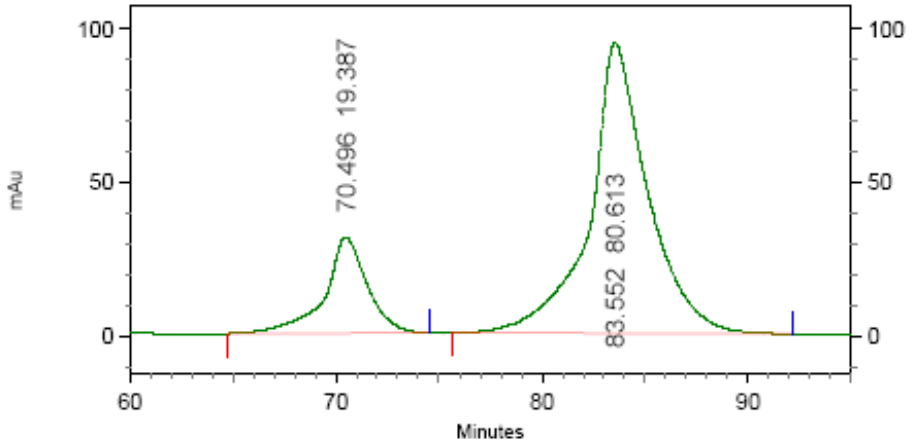


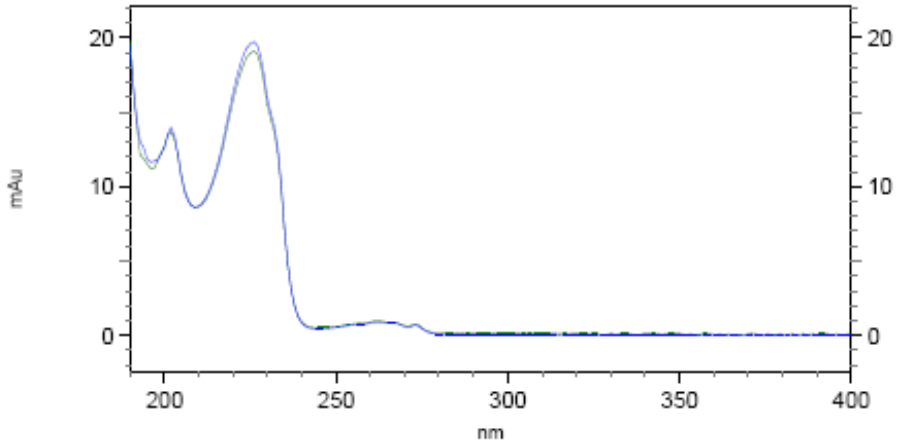


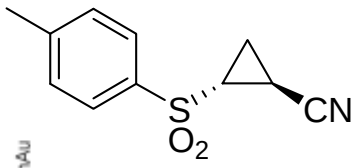


2: 225 nm, 4 nm
Results

Pk #	Name	Retention Time	Area Percent
1		69.828	49.866
2		84.344	50.134
Totals			100.000

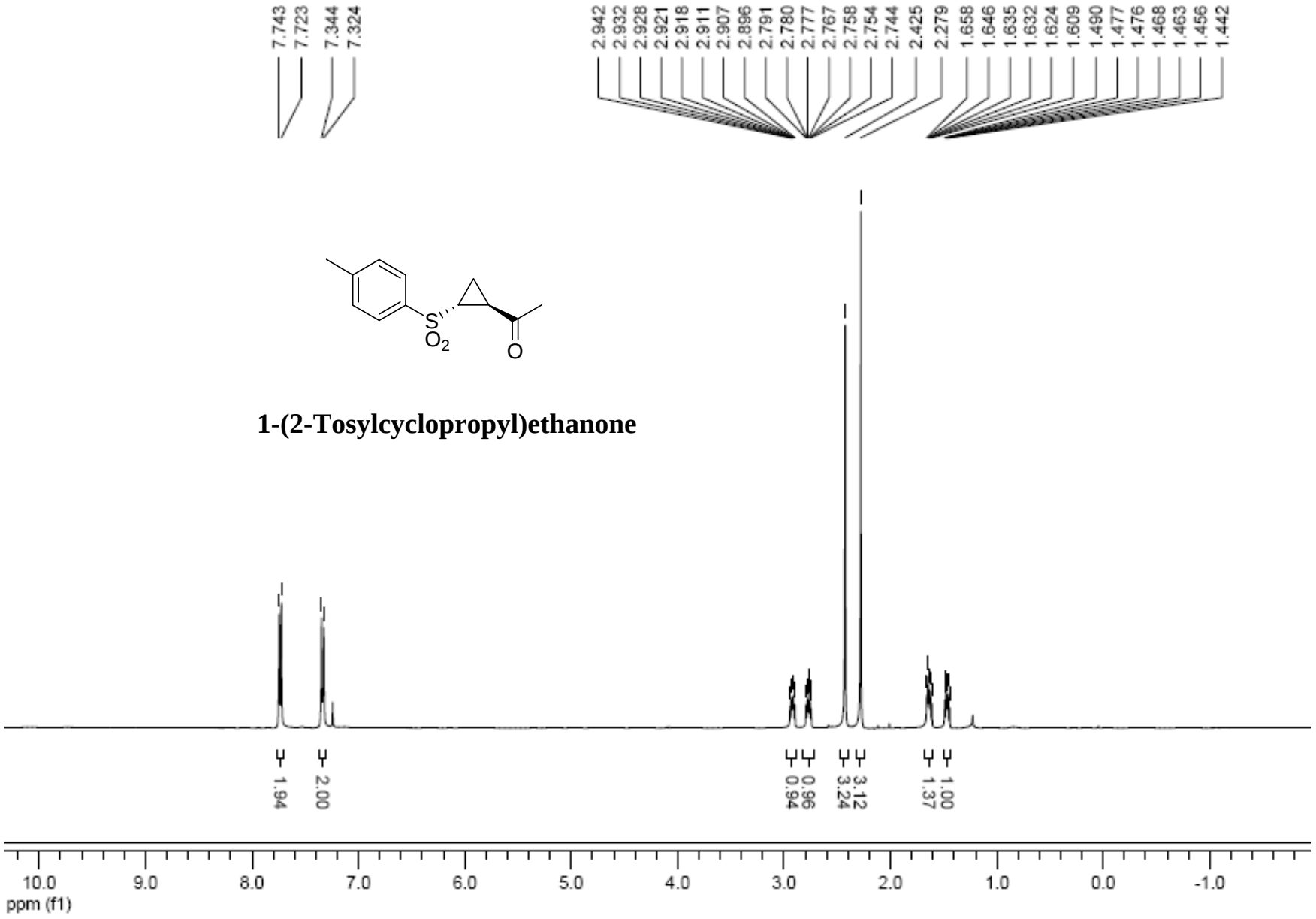




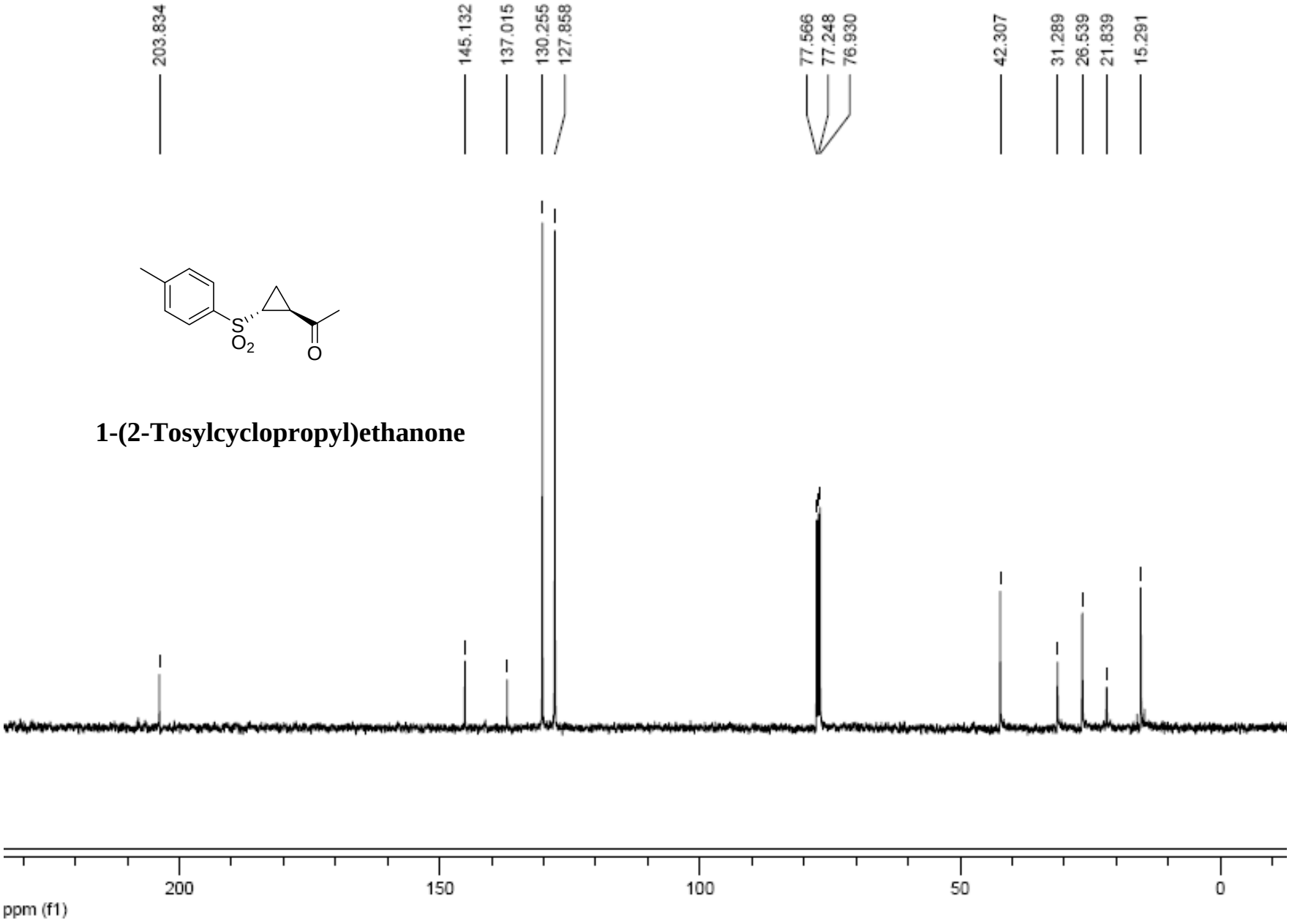


2: 225 nm, 4 nm
Results

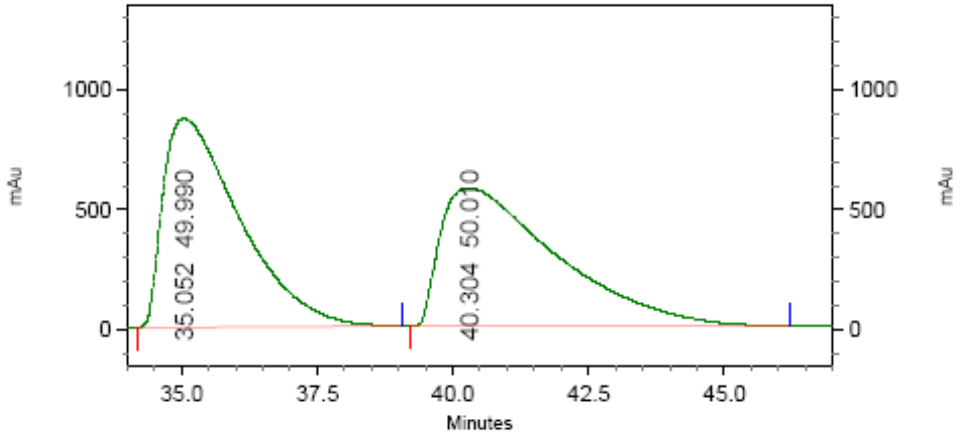
Pk #	Name	Retention Time	Area Percent
1		70.496	19.387
2		83.552	80.613
Totals			100.000

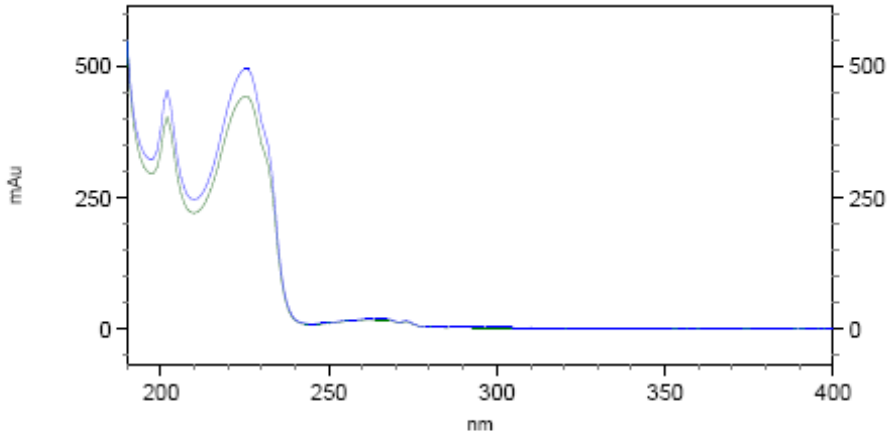


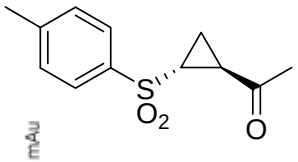
1-(2-Tosylcyclopropyl)ethanone



1-(2-Tosylcyclopropyl)ethanone



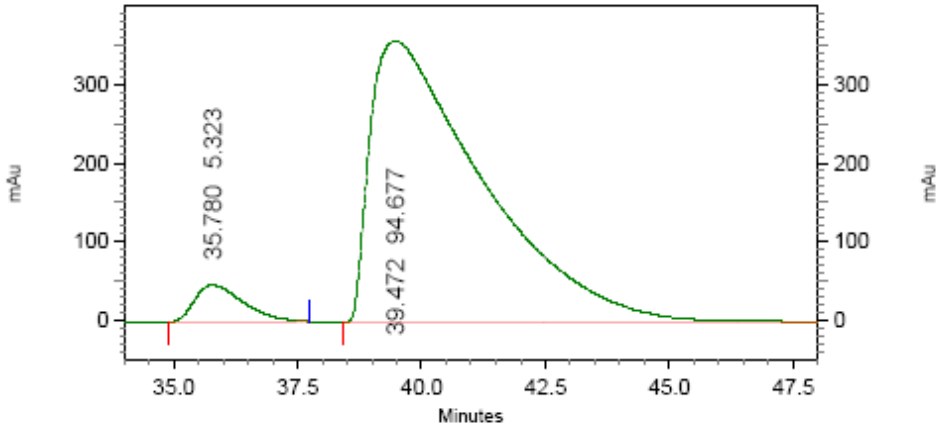


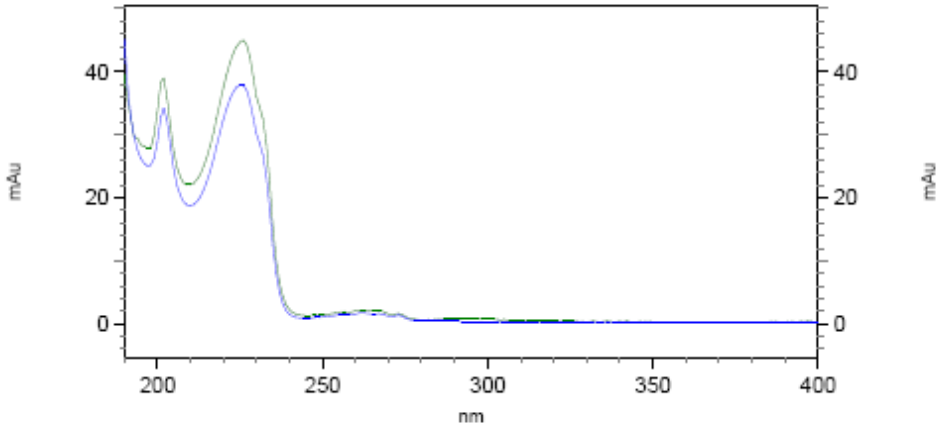


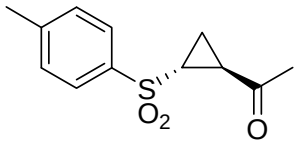
2: 225 nm, 4 nm

Results

Pk #	Name	Retention Time	Area Percent
1		35.052	49.990
2		40.304	50.010
Totals			100.000







2: 225 nm, 4 nm

Results

Pk #	Name	Retention Time	Area Percent
1		35.780	5.323
2		39.472	94.677
Totals			100.000