

Supporting Information

Intramolecular 1,5-C(sp³)-H Radical Amination via Co(II)-Based Metalloradical Catalysis for Five-Membered Cyclic Sulfamides

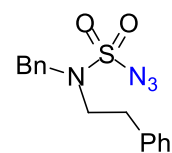
Hongjian Lu,^{*#†‡} Kai Lang,^{#§†} Huiling Jiang,[†] Lukasz Wojtas[†] and X. Peter Zhang^{*§†}

[§]Department of Chemistry, Merkert Chemistry Center, Boston College, Chestnut Hill, Massachusetts 02467, United States

[†]Department of Chemistry, University of South Florida, Tampa, Florida 33620, United States

[‡]The Institute of Chemistry & Biomedical Sciences, Nanjing University, Nanjing, 210093, P. R. China

500 MHz, CDCl₃



1a

7.425
7.421
7.408
7.404
7.393
7.386
7.383
7.373
7.353
7.349
7.337
7.311
7.298
7.282
7.272
7.253
7.238
7.223
7.109
7.095

4.432

3.457
3.441
3.425
2.865
2.849
2.833

2500

2000

1500

1000

500

0

5.0

ppm (f1)

SII

2

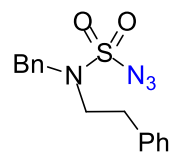
7.41
1.81

2.00

2.00

2.00

125 MHz, CDCl₃



1a

137.513
134.410
128.867
128.662
128.629
128.556
128.483
126.737

77.254
77.000
76.745

52.884
49.911

34.283

10000

5000

0

ppm (f1)

150

100

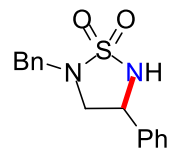
50

0

SII

3

500 MHz, CDCl₃



2a

7.402
7.398
7.394
7.383
7.378
7.375
7.362
7.356
7.351
7.340
7.337
7.332
7.323
7.314
7.260

4.810
4.799
4.787
4.388
4.361
4.012
3.985
3.576
3.561
3.557
3.547
3.542
3.140
3.123
3.120
3.104

9.14

1.86

0.99

1.00

1.00

1.00

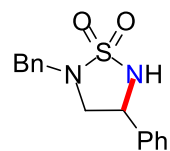
5.0

ppm (f1)

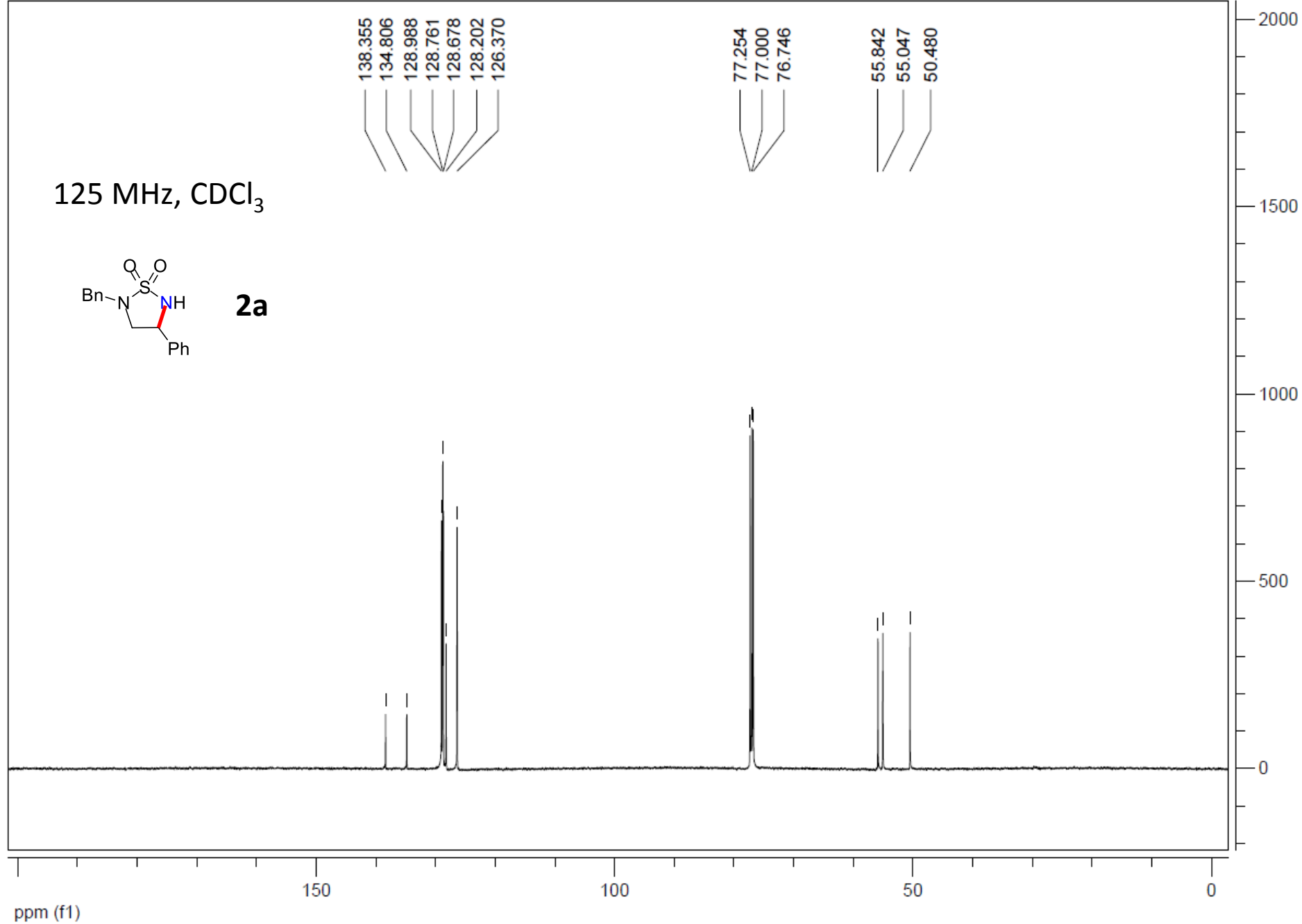
SII

4

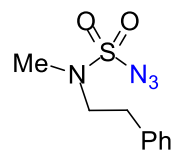
125 MHz, CDCl₃



2a



400 MHz, CDCl₃



1b

7.330
7.312
7.293
7.256
7.238
7.232
7.215
7.212
7.194

3.486
3.467
3.447
2.937
2.916
2.907
2.899

4.38

2.00

4.86

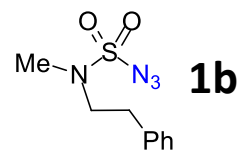
5.0

SII

ppm (f1)

6

100 MHz, CDCl₃



137.399

128.720

126.851

77.318

77.000

76.682

53.005

36.322

34.134

5000

4000

3000

2000

1000

0

ppm (f1)

150

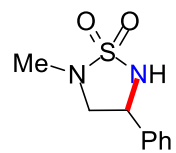
100 SII

50

0

7

400 MHz, CDCl₃



2b

7.440
7.422
7.407
7.390
7.371
7.363
7.345
7.260

4.869
4.851
4.834
4.816
4.728
4.715
3.716
3.697
3.692
3.674
3.254
3.232
3.210
2.754

4.78

0.91
0.99

1.01

1.00

3.03

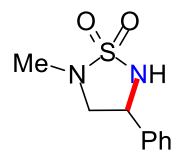
ppm (f1)

5.0

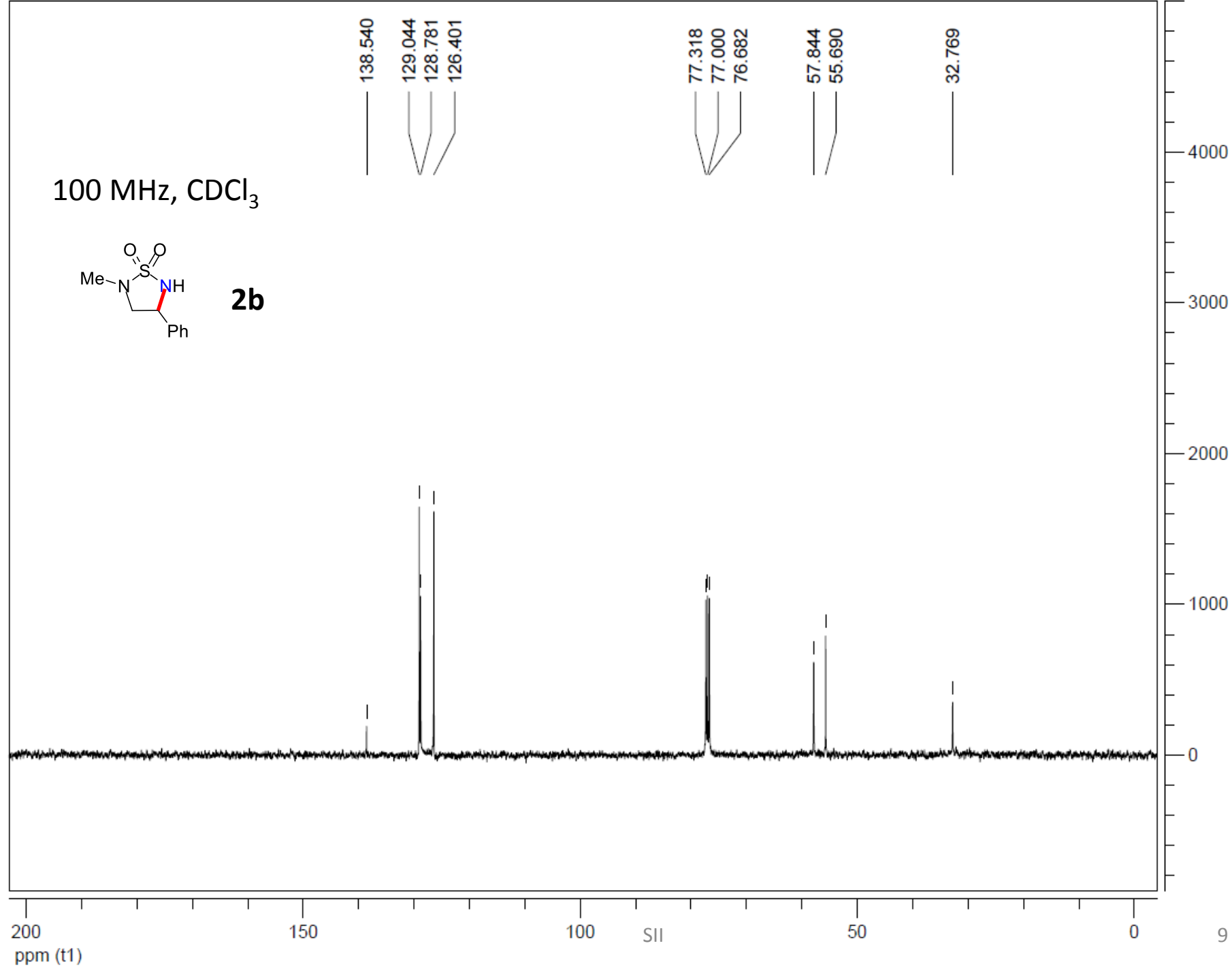
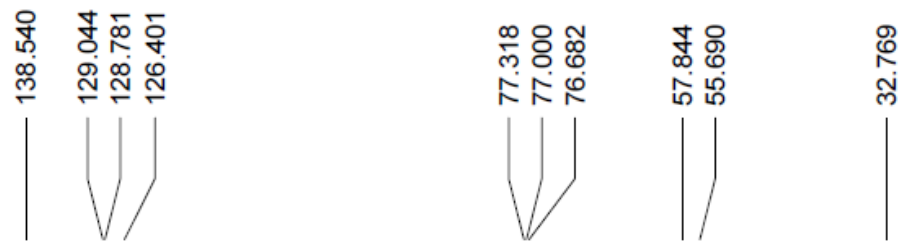
SII

8

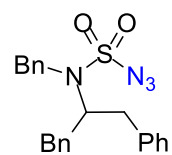
100 MHz, CDCl₃



2b



500 MHz, CDCl₃



1c

7.340
7.338
7.327
7.320
7.281
7.271
7.252
7.235
7.221
7.208
7.032
7.018

4.240
4.002
3.989
3.976
3.080
3.065
3.052
3.037
2.833
2.819
2.806
2.791

2.985
3.80

2.01
0.98

2.00
2.04

ppm (f1)

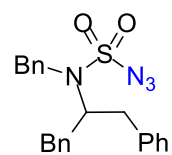
5.0

0.0

SII

10

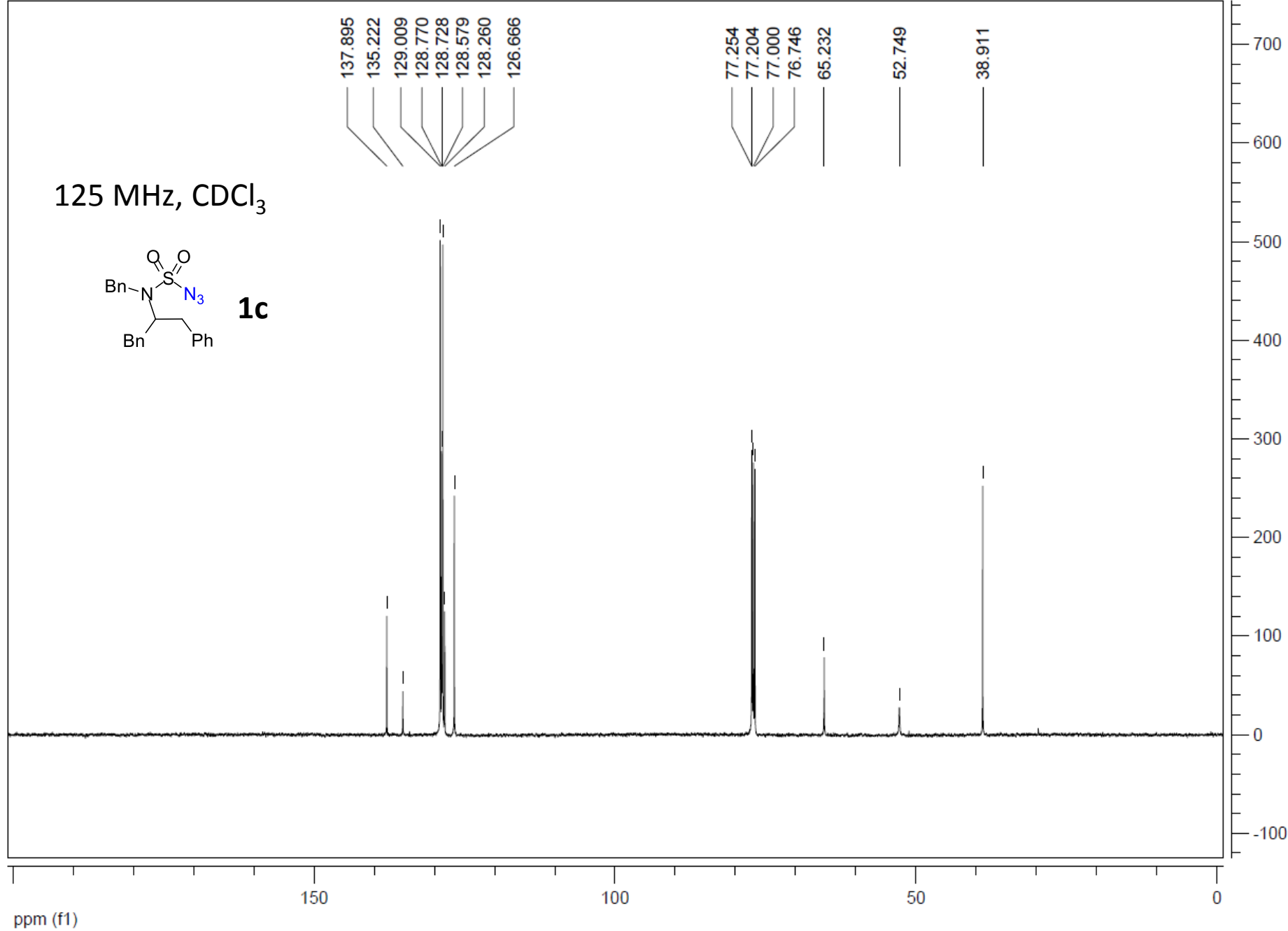
125 MHz, CDCl₃



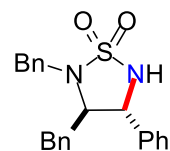
1c

137.895
135.222
129.009
128.770
128.728
128.579
128.260
126.666

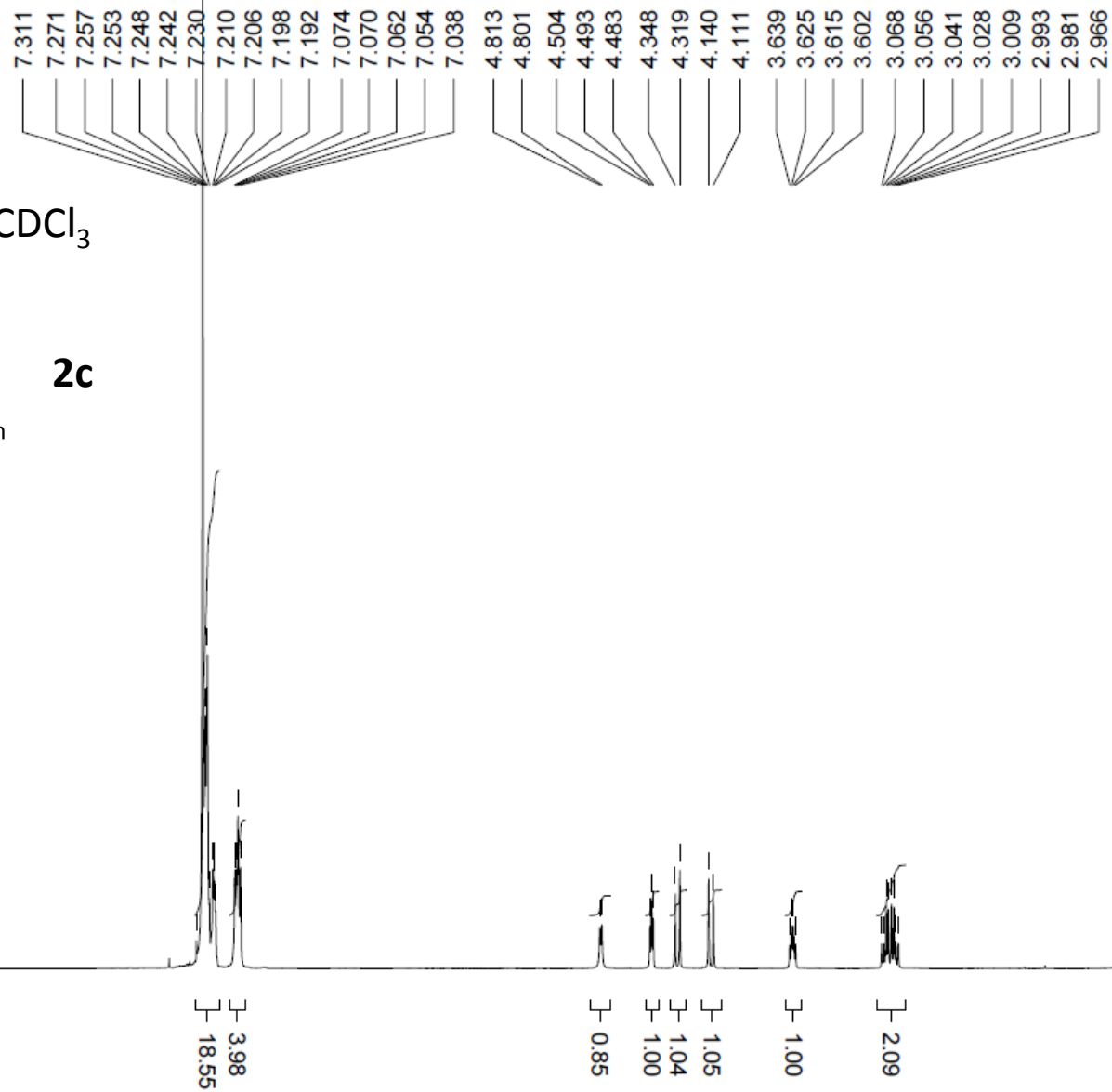
77.254
77.204
77.000
76.746
65.232
52.749
38.911



500 MHz, CDCl₃



2c



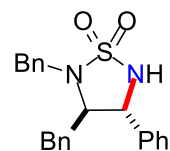
ppm (f1)

5.0

SII

12

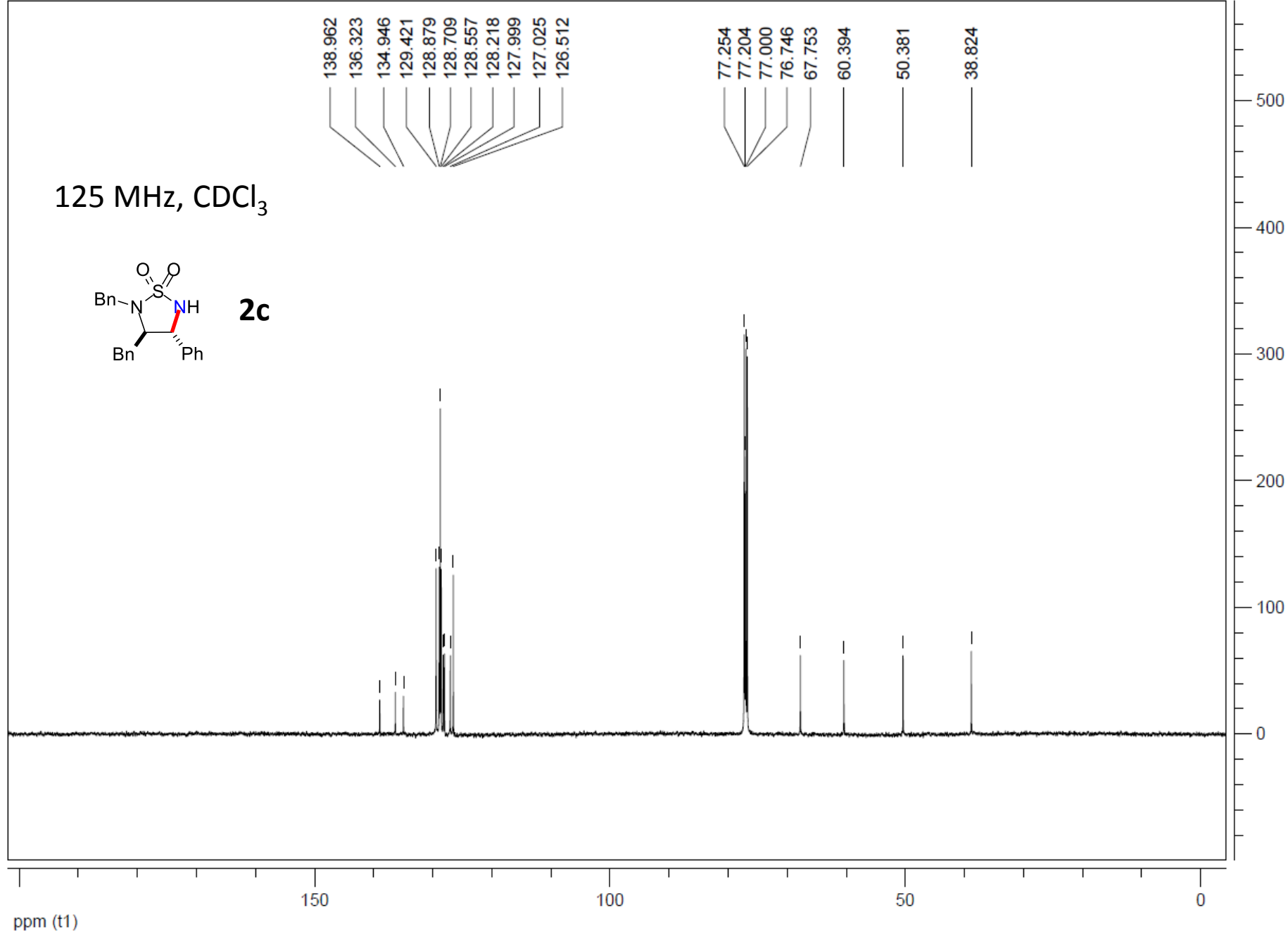
125 MHz, CDCl₃

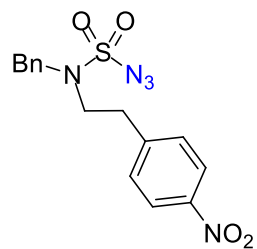


2c

138.962
136.323
134.946
129.421
128.879
128.709
128.557
128.218
127.999
127.025
126.512

77.254
77.204
77.000
76.746
67.753
60.394
50.381
38.824





1d

500 MHz, CDCl₃

8.105
8.087
7.380
7.372
7.367
7.311
7.304
7.297
7.260
7.184
7.167

4.415

3.467
3.452
3.436
2.913
2.897
2.882

1.76

1.88
1.89

2.00

2.00

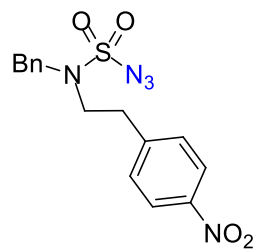
2.01

ppm (f1)

5.0

0.0

125 MHz, CDCl₃



146.903
145.134
134.075
129.612
129.031
128.759
128.683
123.832

77.254
77.000
76.746

53.618
49.643

34.409

200
ppm (f1)

150

100

50

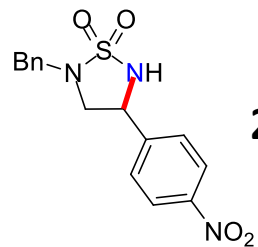
0

SII

15

600
500
400
300
200
100
0
-100

500 MHz, CD₃OD



2d

8.255
8.238
7.715
7.698
7.406
7.392
7.382
7.367
7.352
7.335
7.321

4.985
4.970
4.954
4.892
4.324
4.297
4.001
3.974
3.818
3.799
3.783
3.340
3.020
3.001
2.985

1.73

1.84

4.32

0.96

1.00

1.01

1.04

1.04

10.0
ppm (f1)

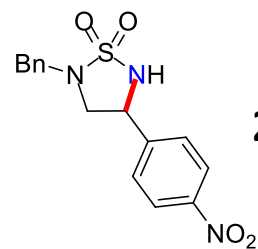
5.0

0.0

SII

16

125 MHz, CD₃OD



147.676
147.550
135.312
128.353
128.308
127.684
127.164
123.361

54.341
54.165
49.977
48.100
47.830
47.759
47.589
47.419
47.248
47.078

2000

1500

1000

500

0

ppm (t1)

150

100

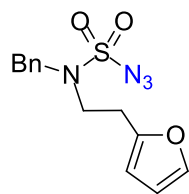
50

0

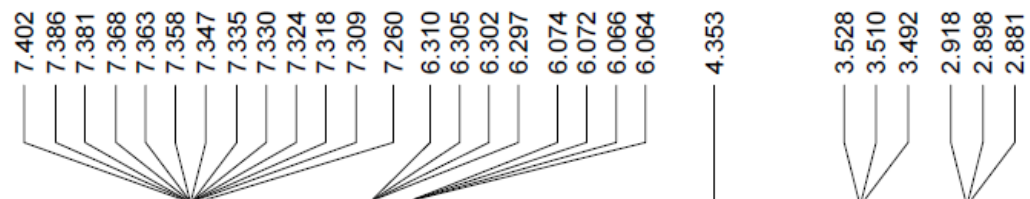
SII

17

400 MHz, CDCl₃



1e



5.37

0.84
0.86

2.00

2.01

2.01

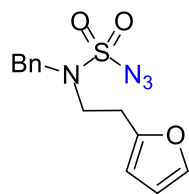
ppm (f1)

5.0

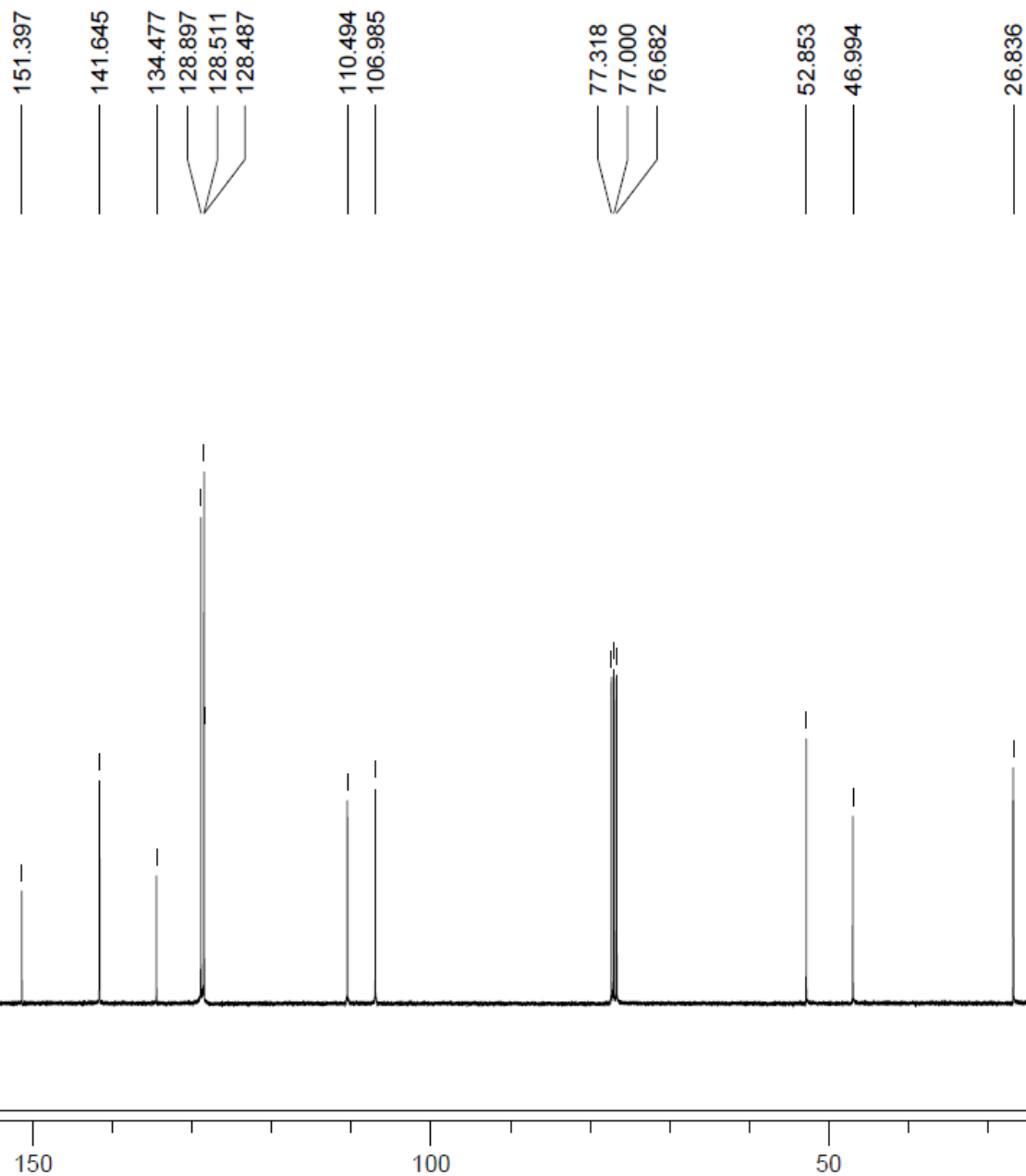
SII

18

100 MHz, CDCl₃

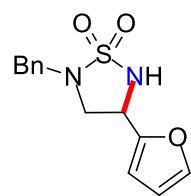


1e

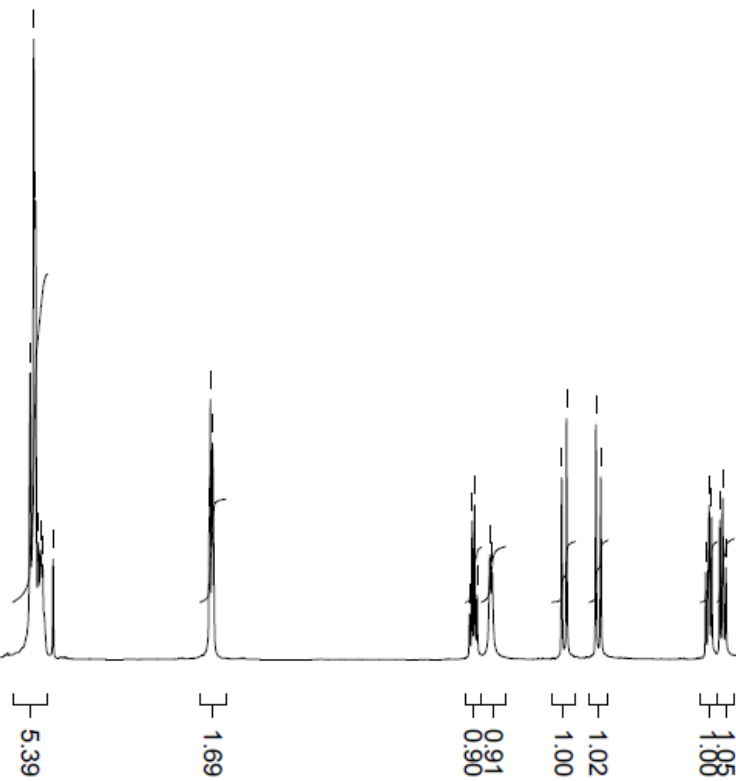
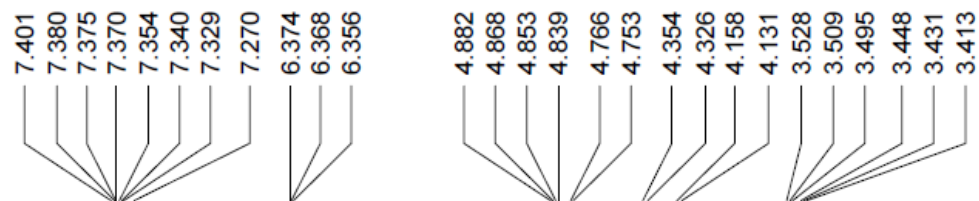


ppm (f1)

500 MHz, CDCl₃



2e



ppm (f1)

5.0

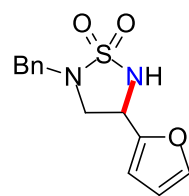
SII

20

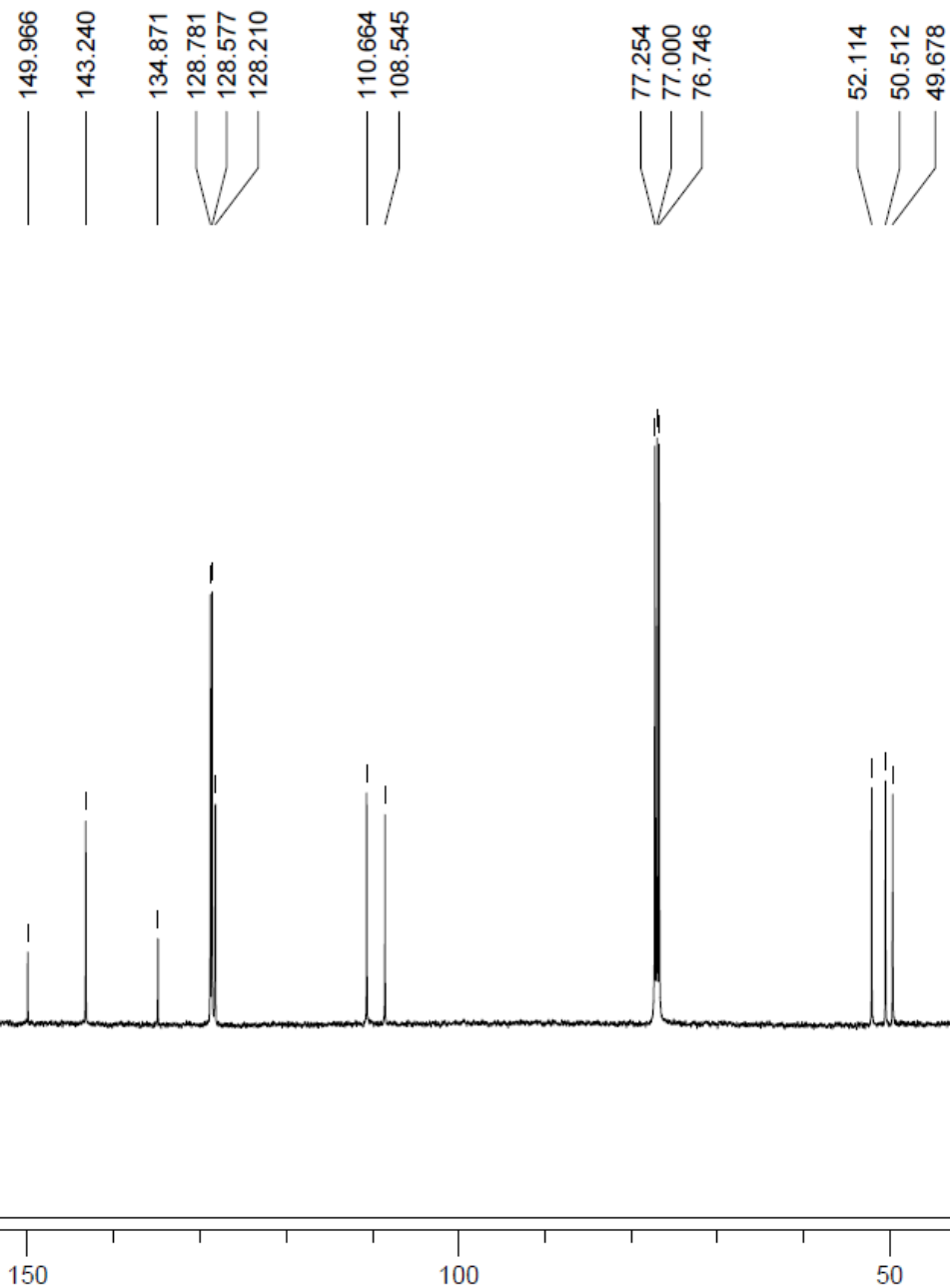
500

0

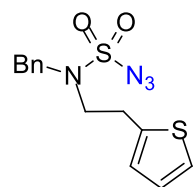
125 MHz, CDCl₃



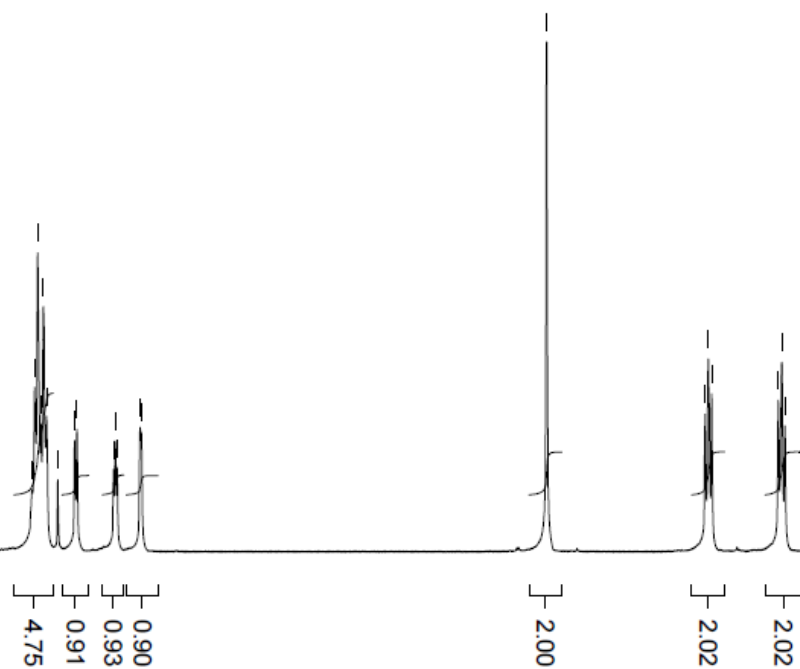
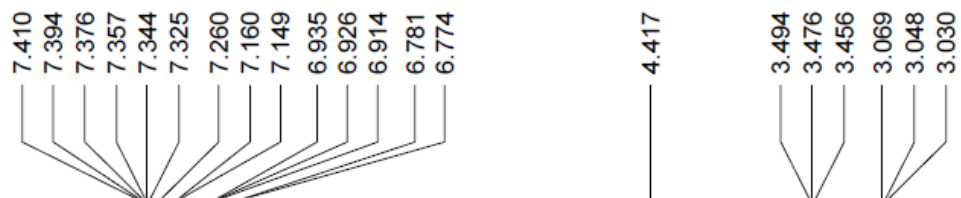
2e



400 MHz, CDCl₃



1f



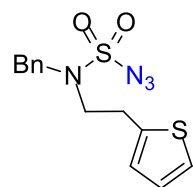
ppm (f1)

5.0

SII

22

100 MHz, CDCl₃



1f

139.569
134.381
128.961
128.614
128.588
127.107
125.720
124.216

77.318
77.000
76.682

53.227
50.043

28.480

10000

5000

0

ppm (f1)

150

100

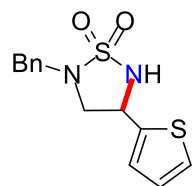
50

0

SII

23

500 MHz, CDCl₃



2f

7.393
7.378
7.364
7.349
7.335
7.330
7.322
7.308
7.297
7.287
7.260
7.058
7.051
6.968
6.960
6.951
5.078
5.064
5.051
5.037
4.786
4.775
4.334
4.307
4.133
4.105
3.595
3.581
3.575
3.562
3.316
3.300
3.296
3.281

1000

5.21

0.89

0.89

26.0

0.89

1.00

1.02

1.01

1.01

5.0

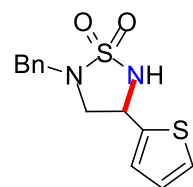
0.0

ppm (f1)

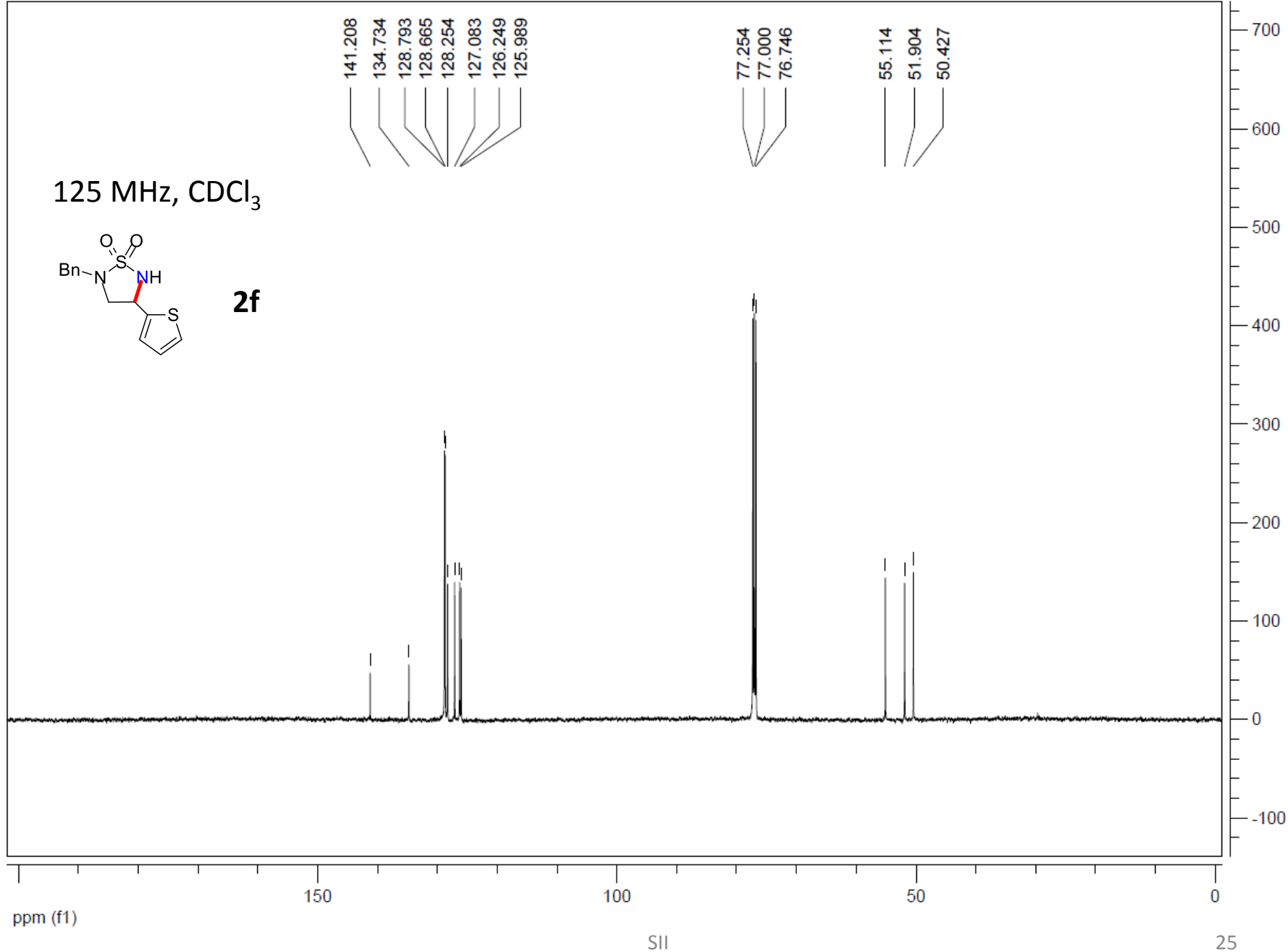
SII

24

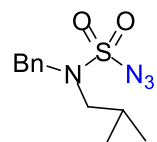
125 MHz, CDCl₃



2f



500 MHz, CDCl₃



1g

7.397
7.380
7.369
7.362
7.351
7.335
7.331
7.260

4.473

3.042
3.027

1.906
1.892
1.878
1.864
1.851
1.837

0.885
0.871

4.67

2.06

2.16

1.00

6.60

ppm (f1)

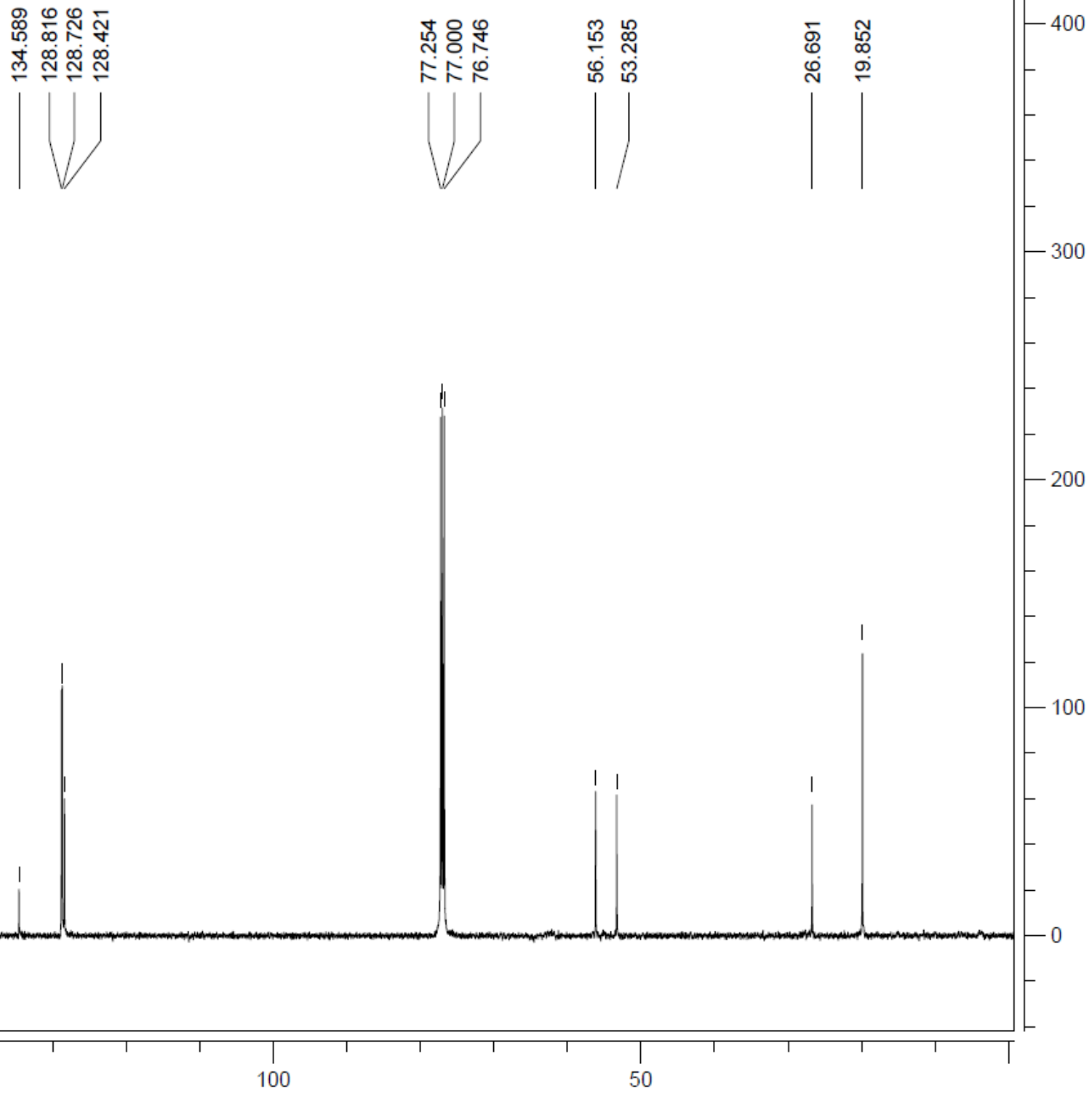
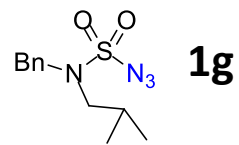
5.0

0.0

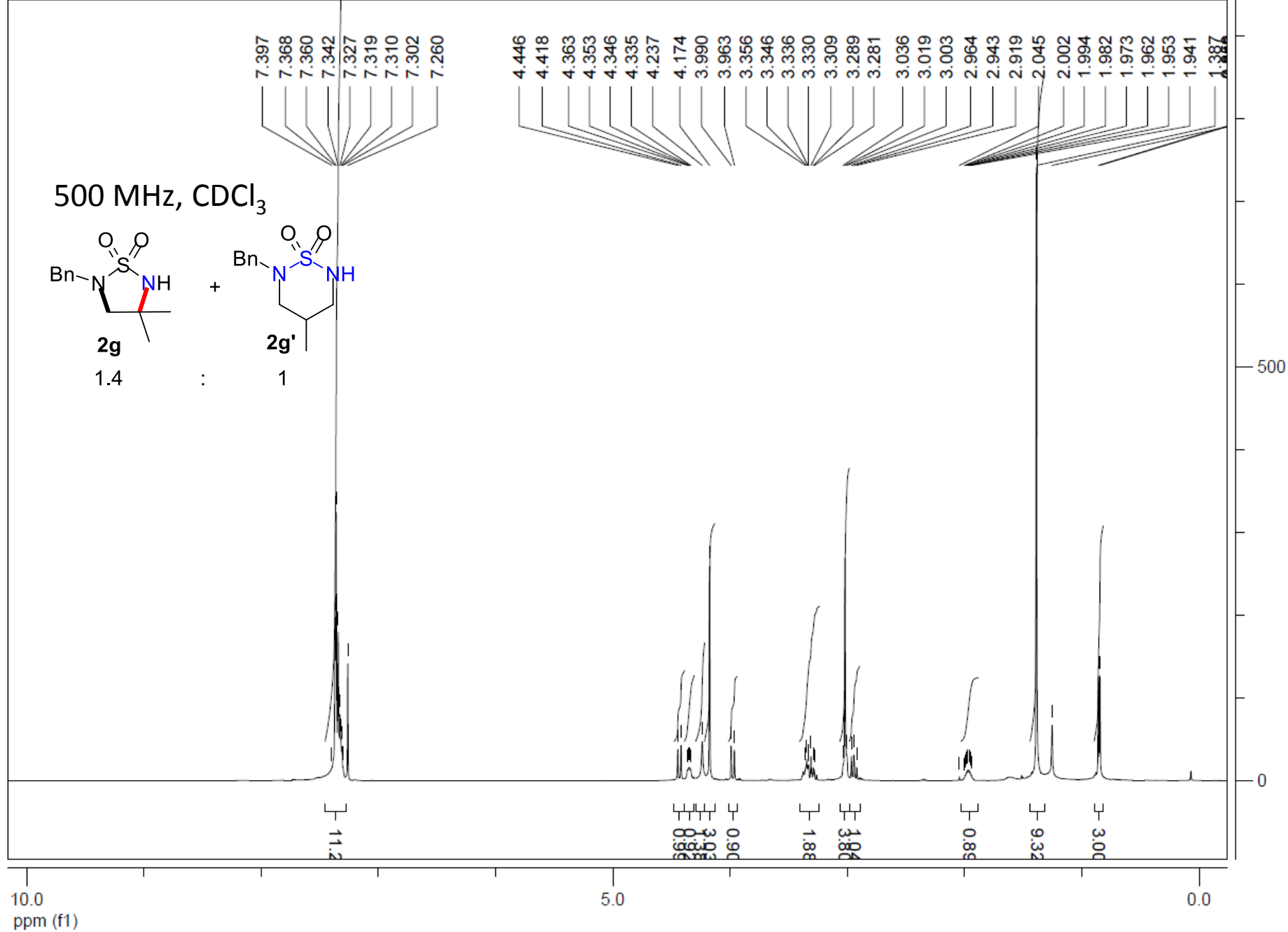
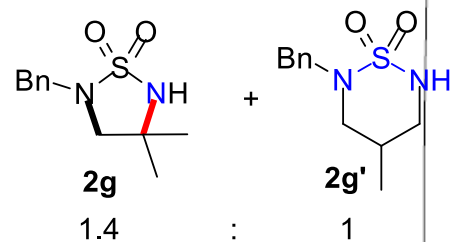
SII

26

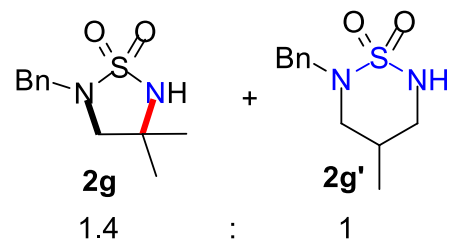
125 MHz, CDCl₃



500 MHz, CDCl₃



125 MHz, CDCl₃



135.393
135.185
128.737
128.618
128.424
128.062
127.899

77.254
77.000
76.746

59.928
55.722
54.840

51.989
51.270
49.862

29.040
28.452

14.803

1500

1000

500

0

ppm (t1)

150

100

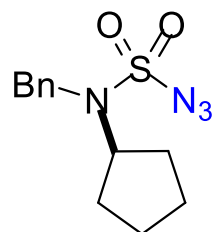
50

0

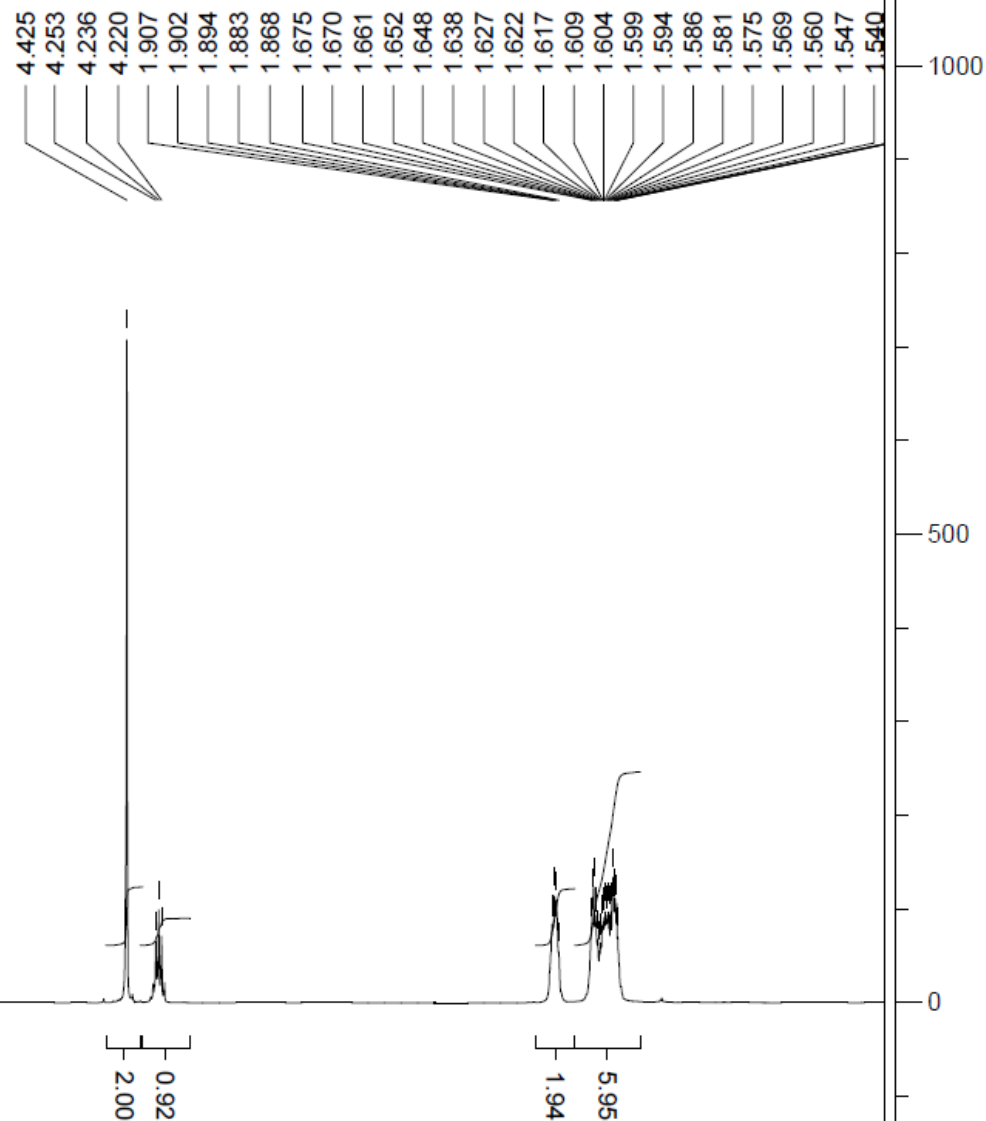
SII

29

500 MHz, CDCl₃



1h



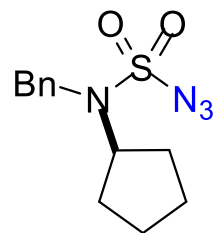
ppm (f1)

5.0

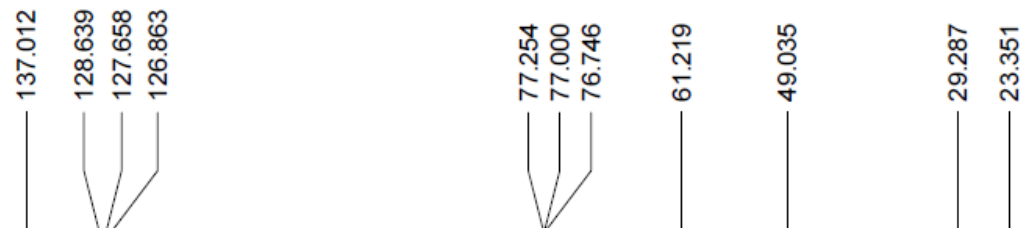
SII

30

125 MHz, CDCl₃



1h



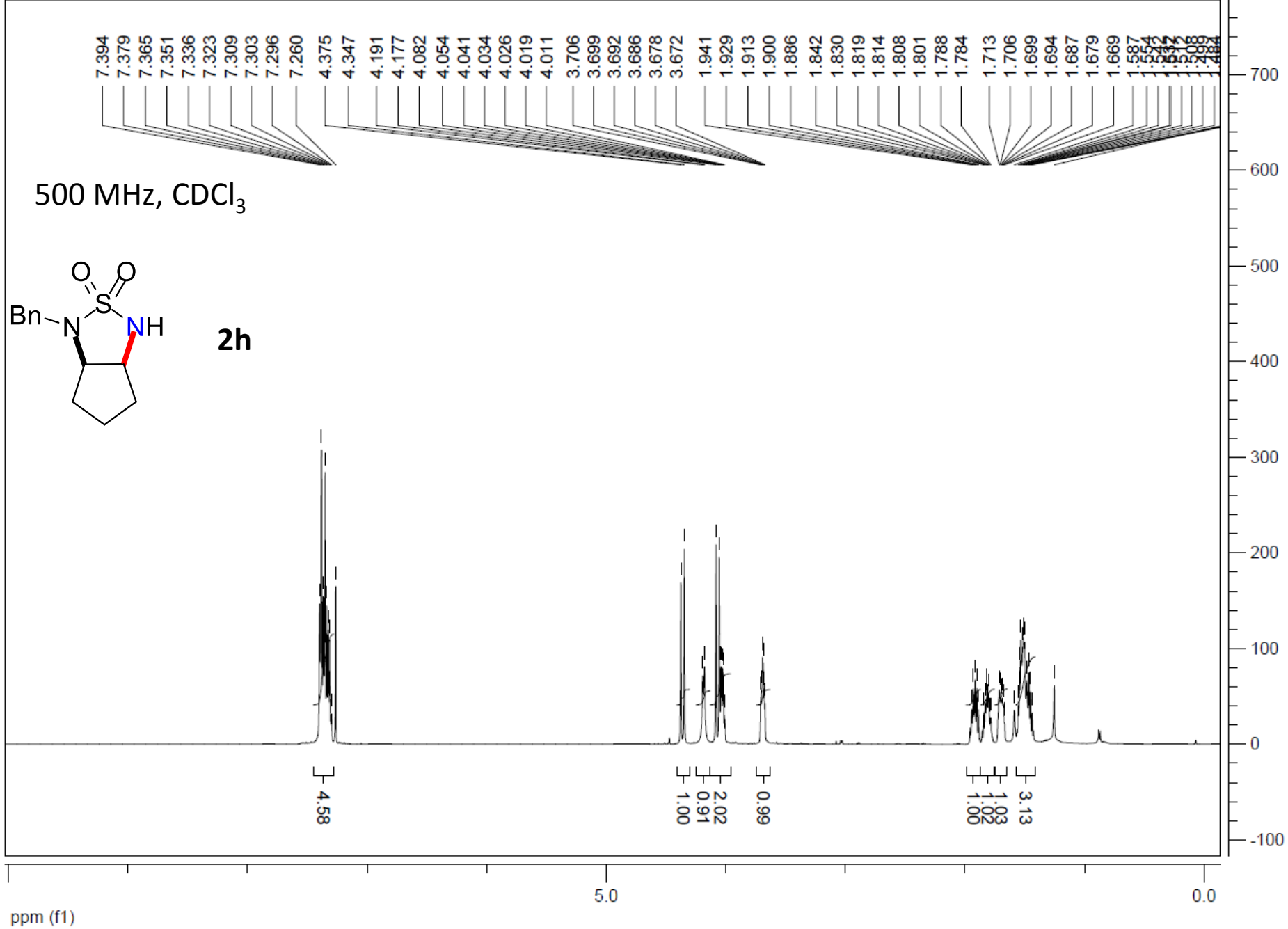
ppm (t1)

150

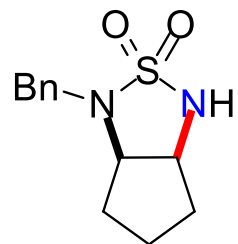
100

50

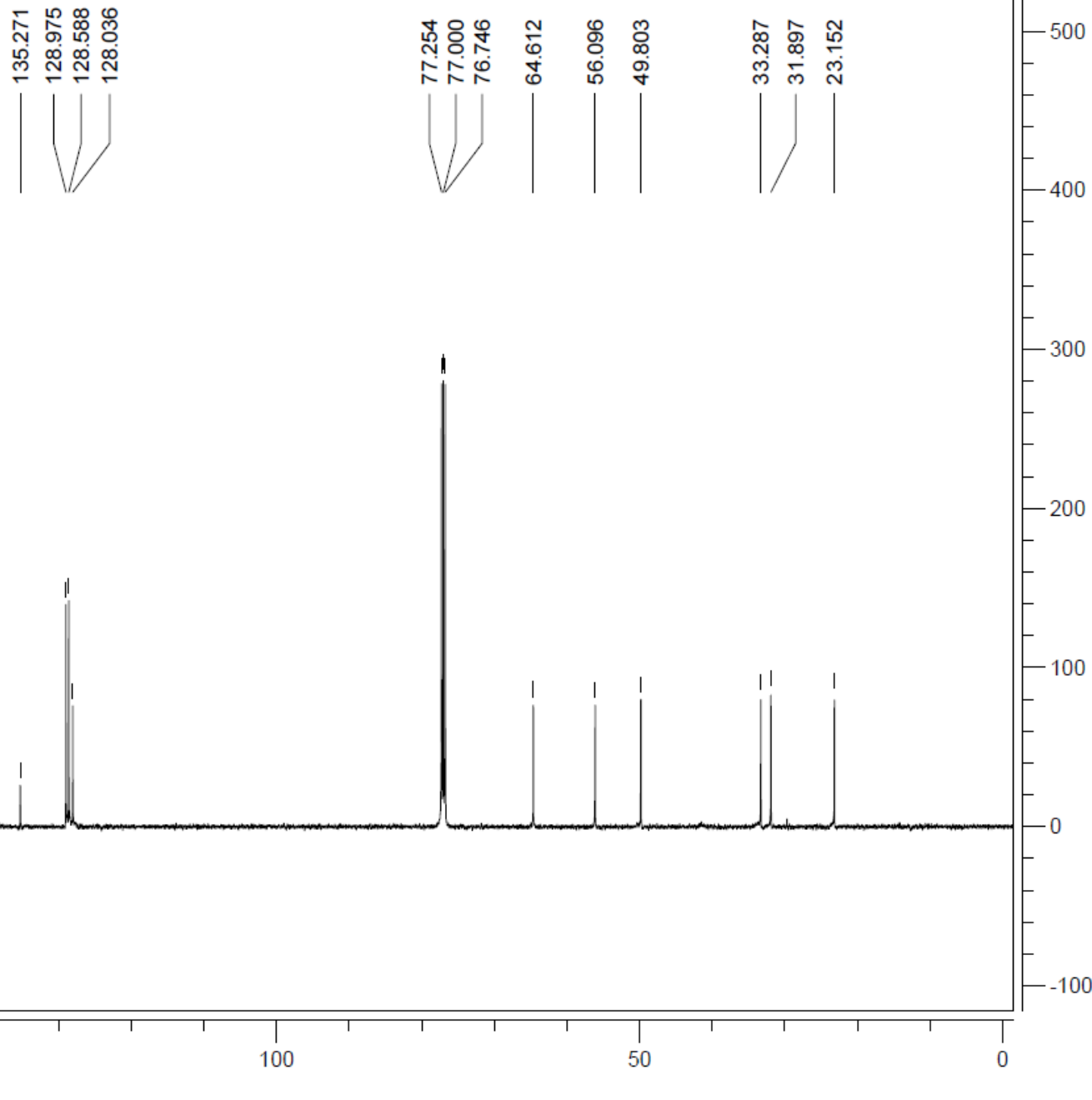
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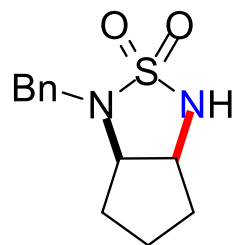


125 MHz, CDCl₃

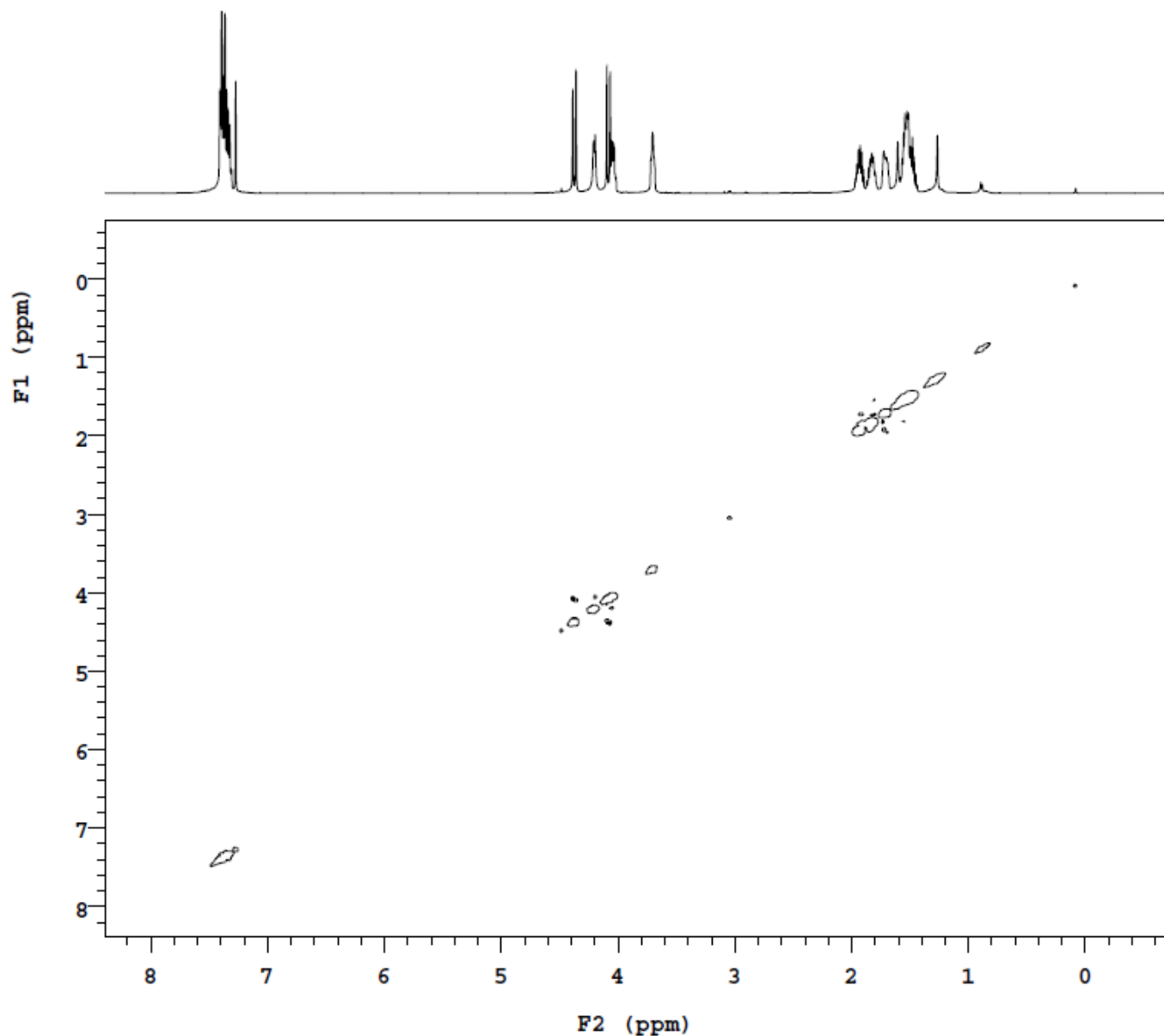
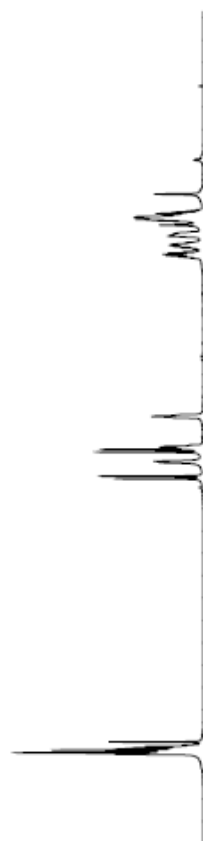


2h

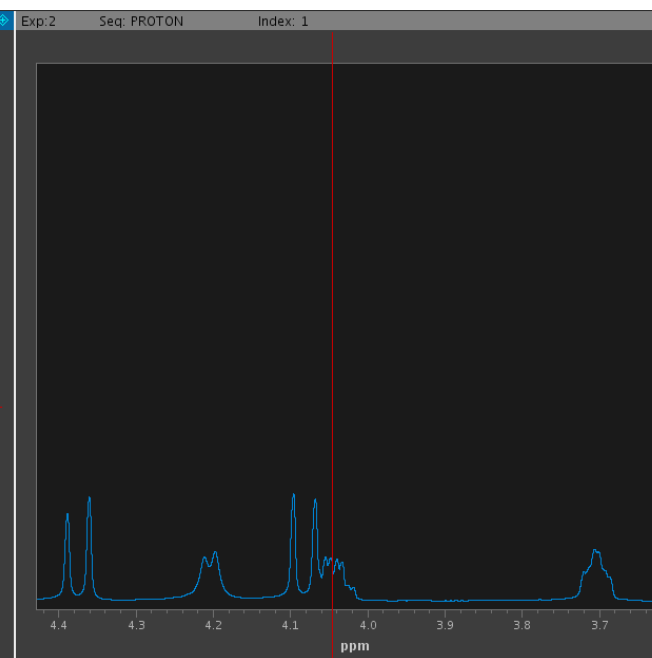
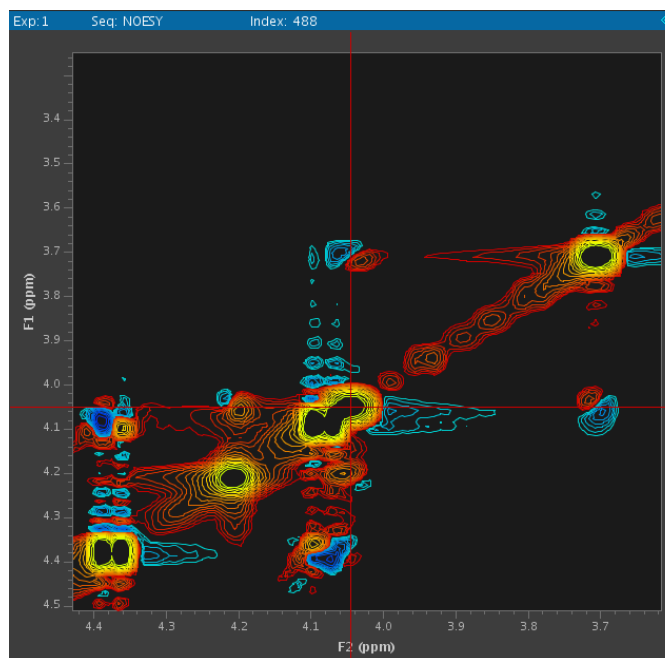
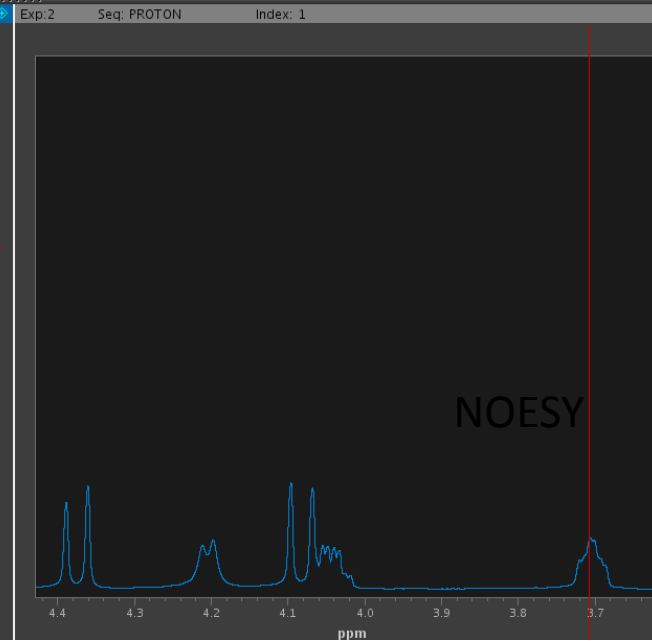
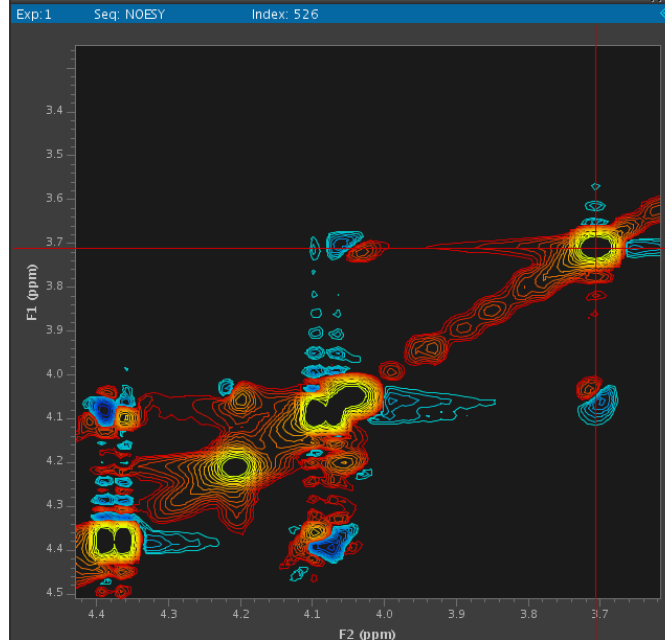
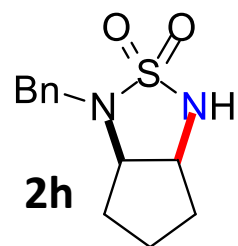
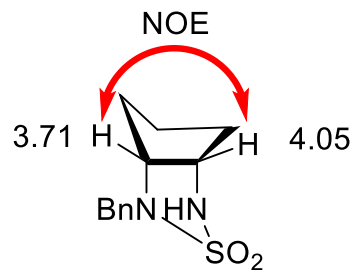


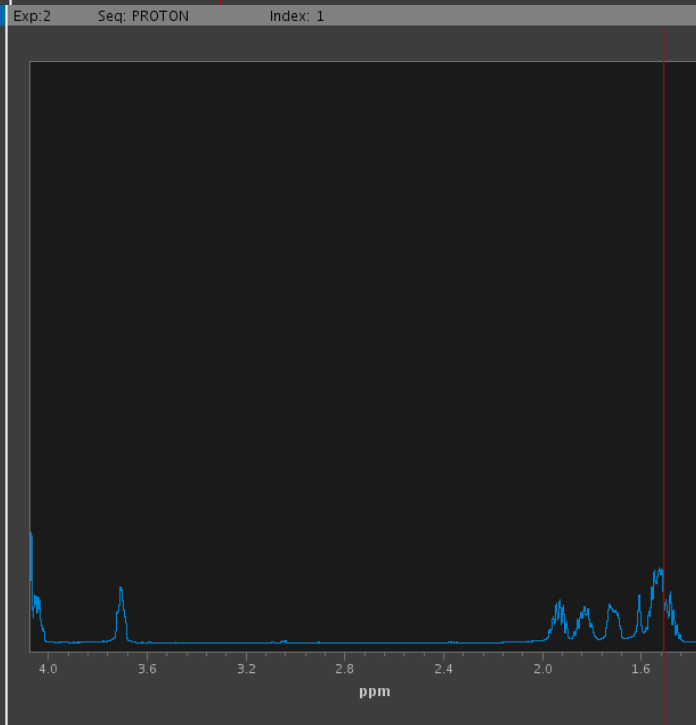
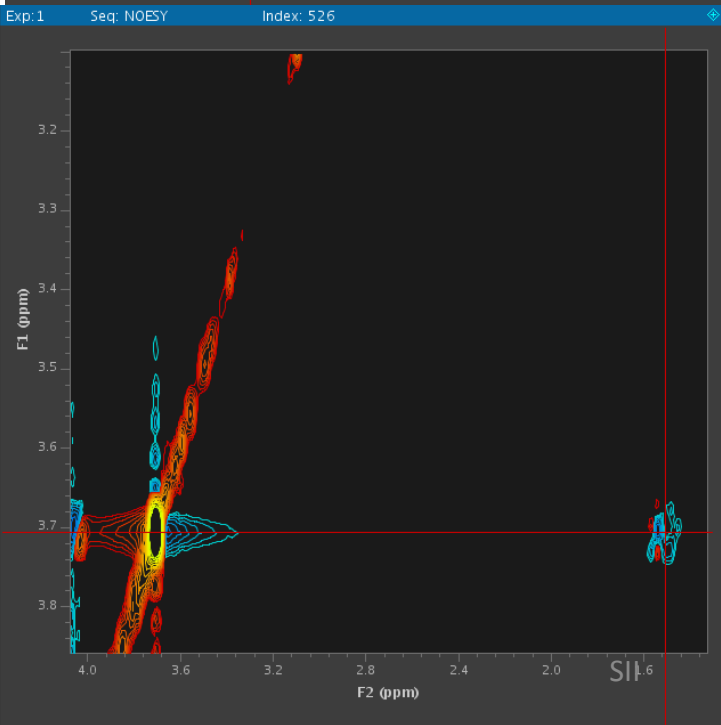
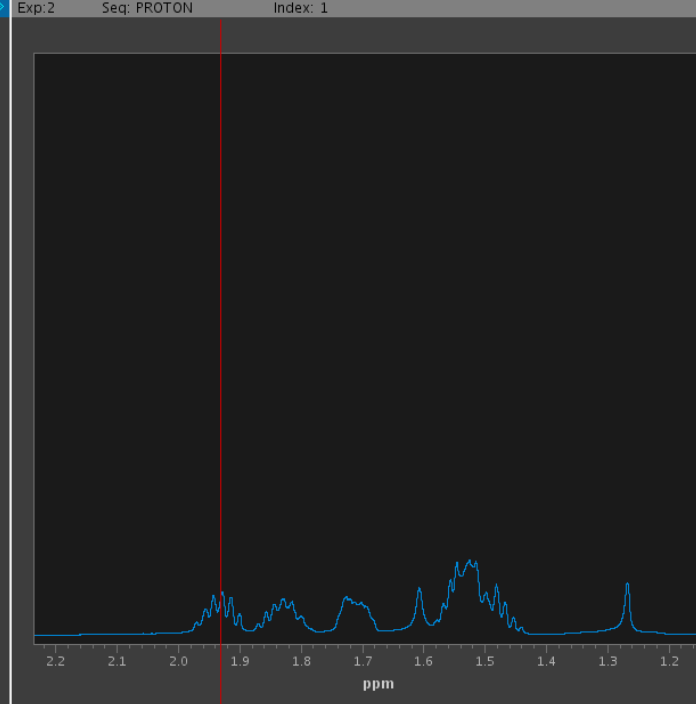
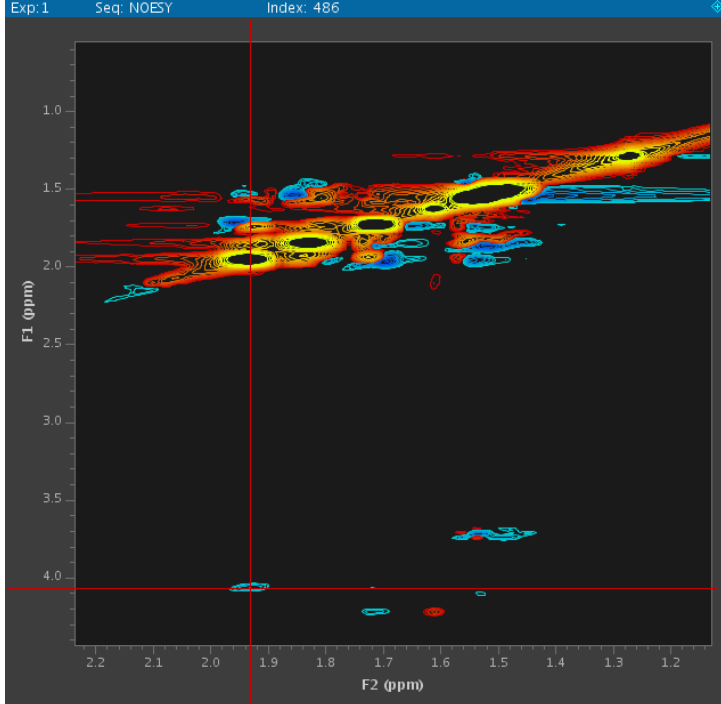
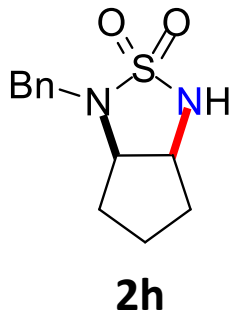
500 MHz, CDCl₃

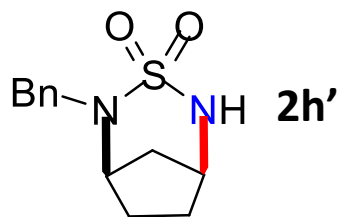
2h



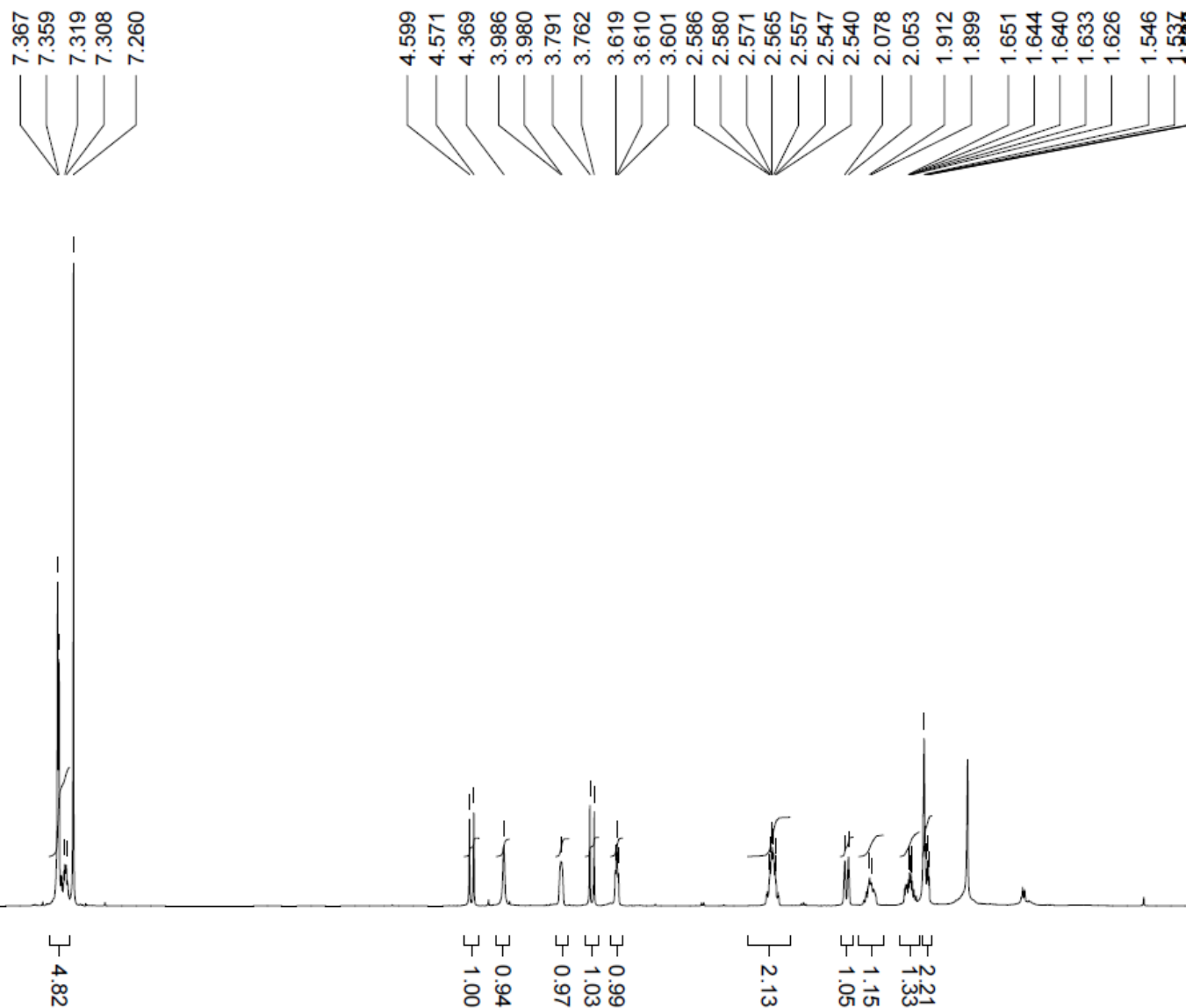
500 MHz, CDCl₃







500 MHz, CDCl₃



5.0

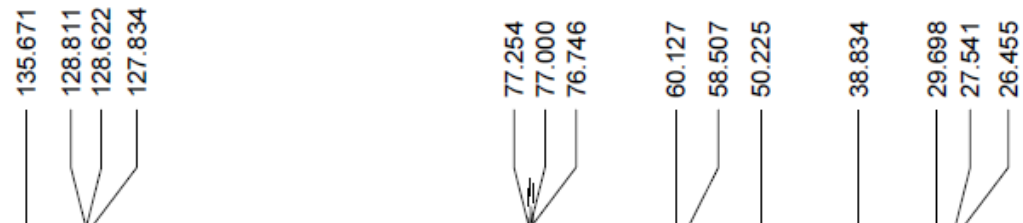
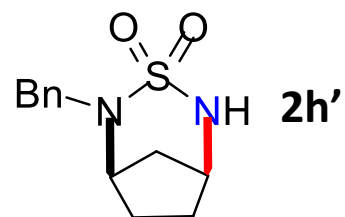
0.0

ppm (f1)

SII

37

125 MHz, CDCl₃



1500

1000

500

0

ppm (f1)

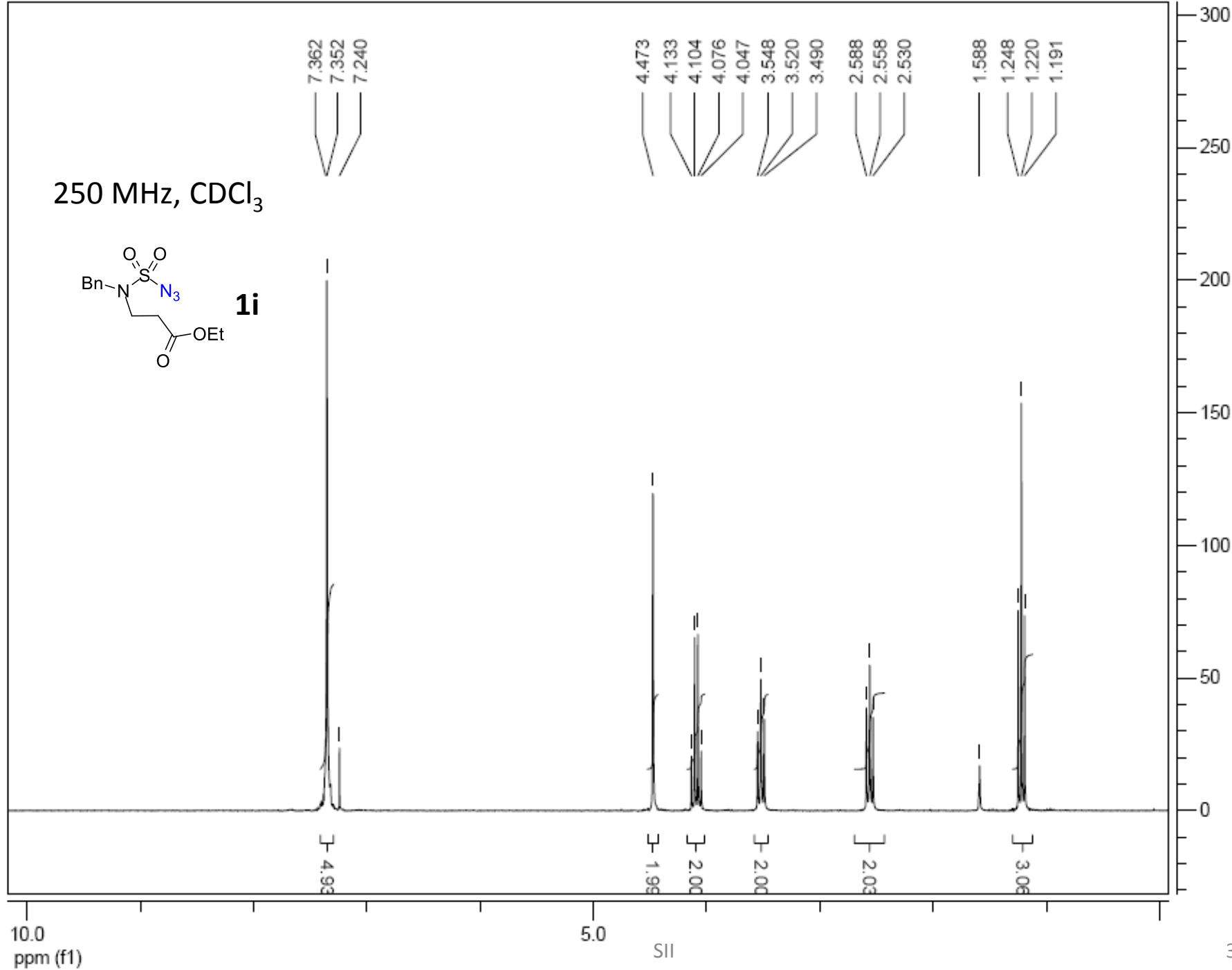
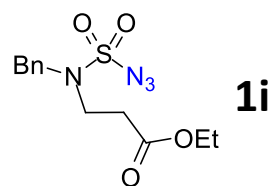
150

100

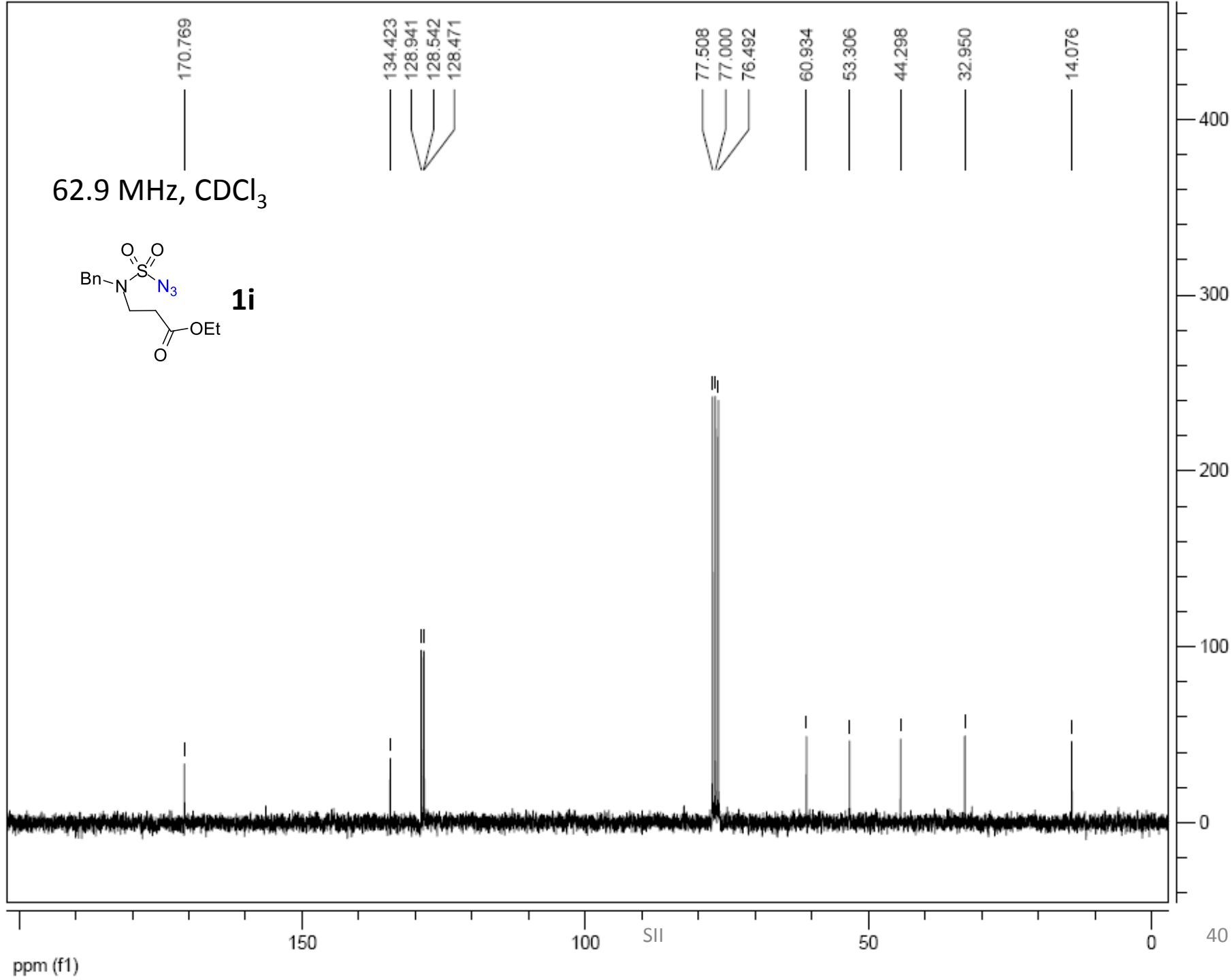
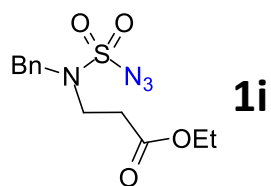
50

0

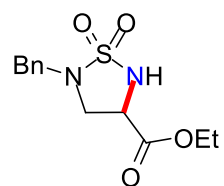
250 MHz, CDCl₃



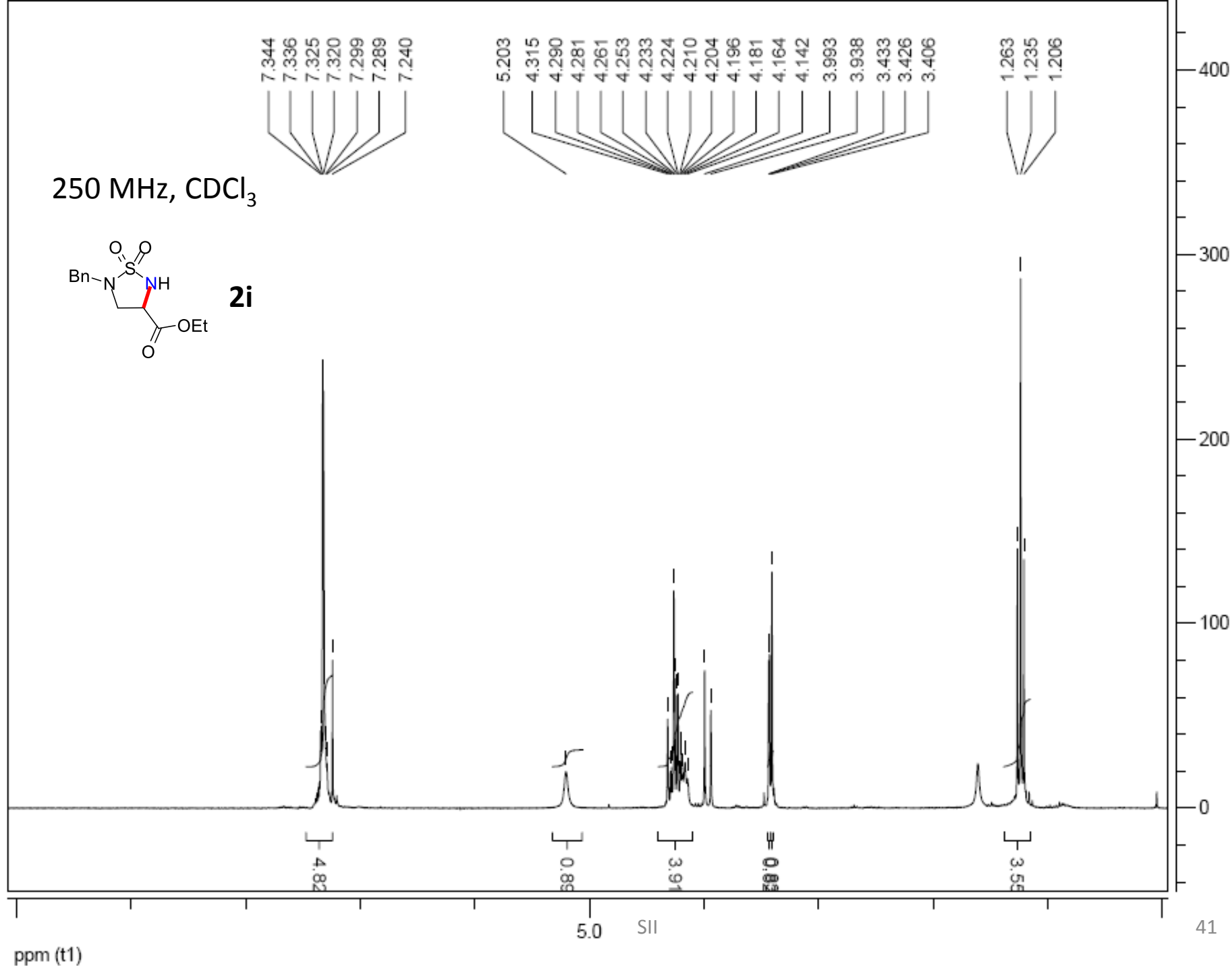
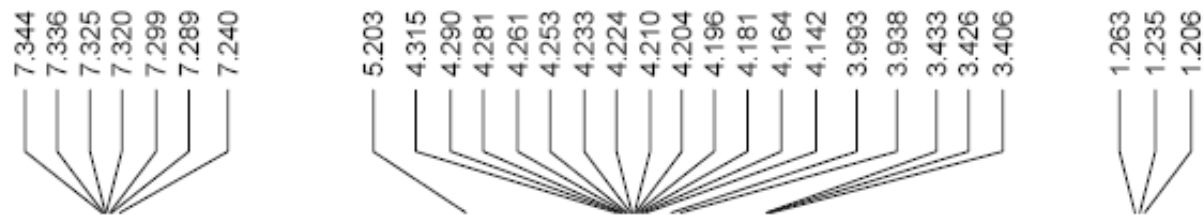
62.9 MHz, CDCl₃



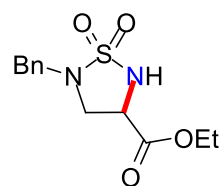
250 MHz, CDCl₃



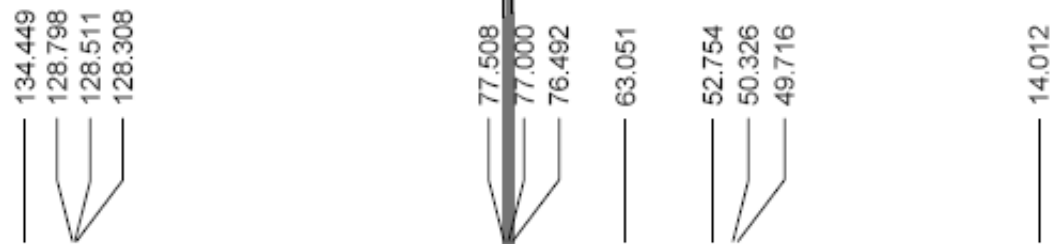
2i



62.9 MHz, CDCl₃



2i



1000

500

0

42

SII

ppm (f1)

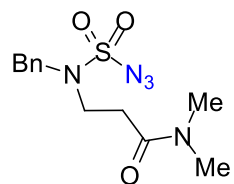
150

100

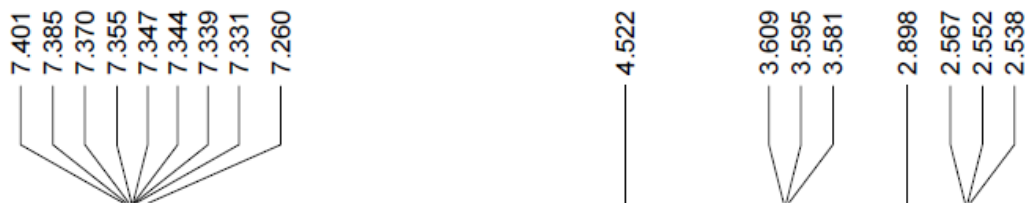
50

0

500 MHz, CDCl₃



1j



4.61

2.02

2.00

5.72

2.01

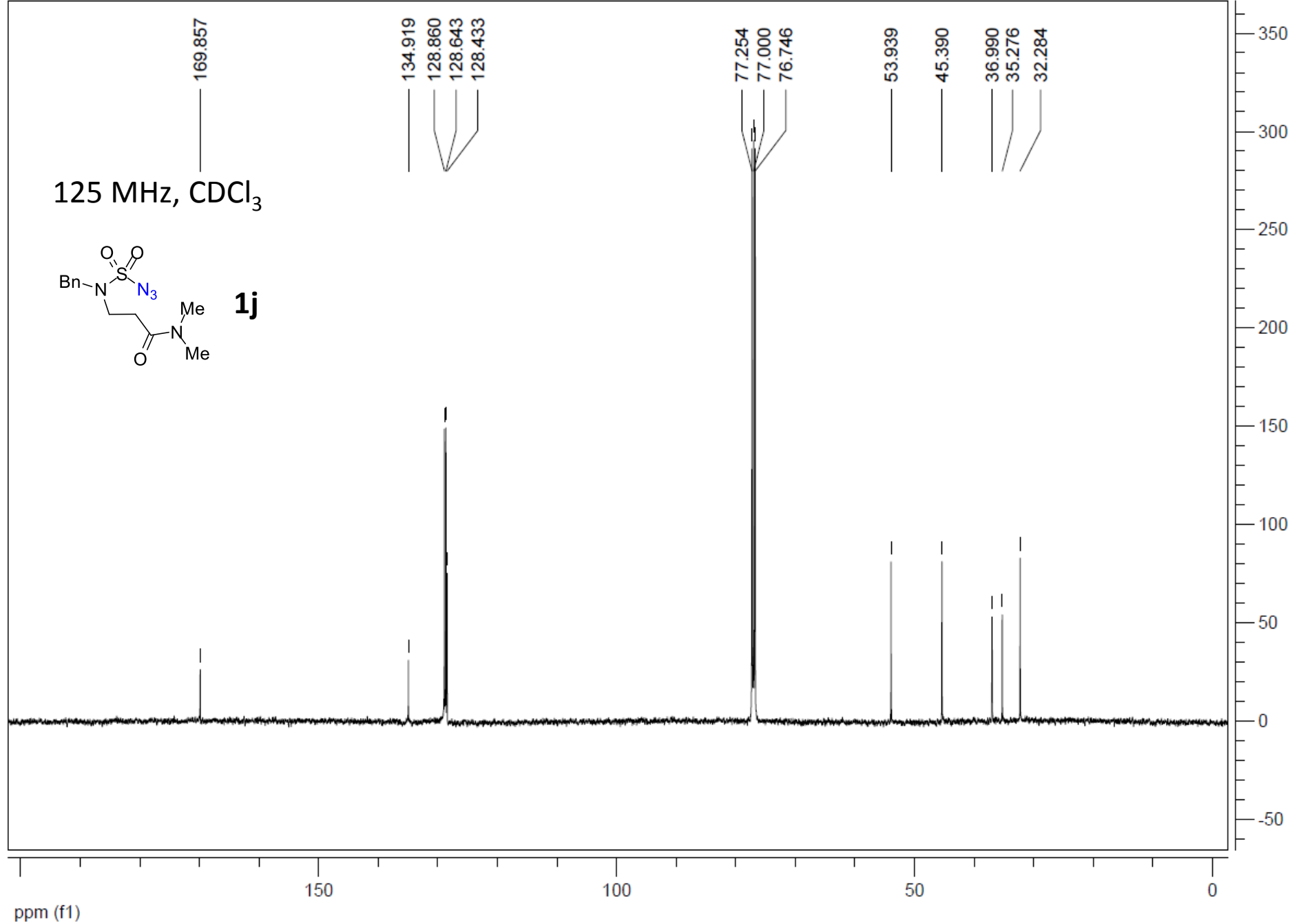
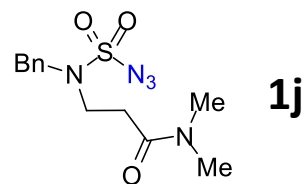
ppm (f1)

5.0

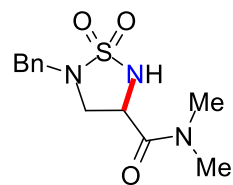
SII

43

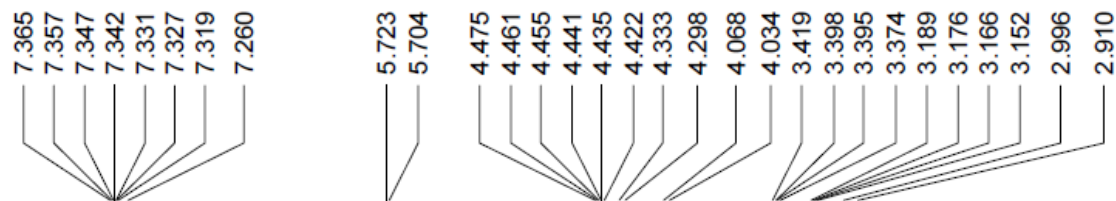
125 MHz, CDCl₃



400 MHz, CDCl₃



2j



5.08

0.83

0.94

1.04

1.04

1.02

3.23

3.21

3.19

3.17

3.15

2.99

2.91

5.0

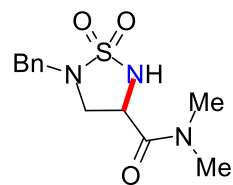
0.0

ppm (f1)

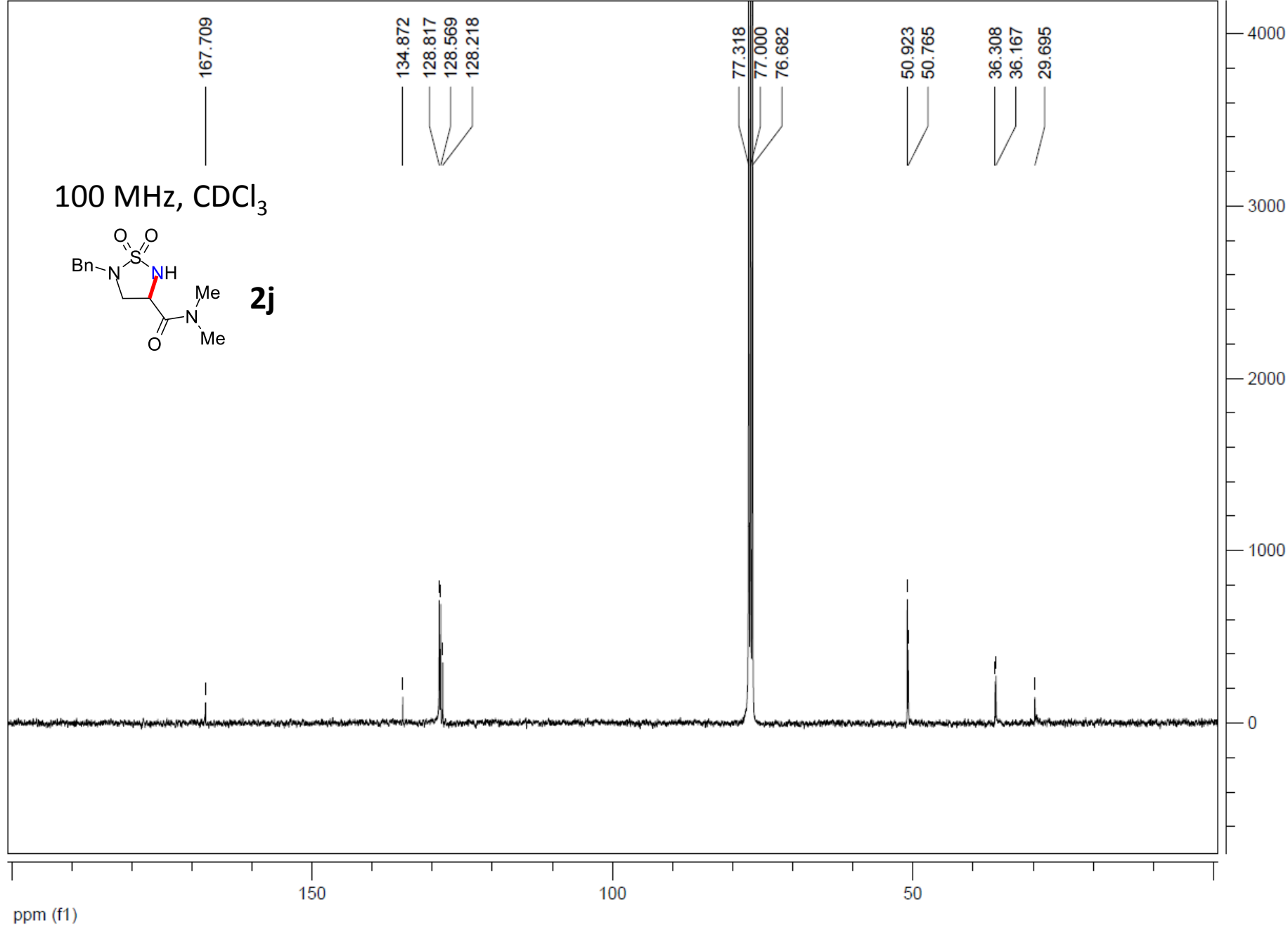
SII

45

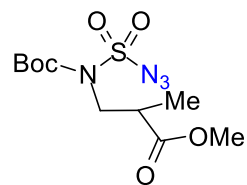
100 MHz, CDCl₃



2j



400 MHz, CDCl₃



1k

7.242

4.028
4.009
3.991
3.972
3.784
3.766
3.747
3.729
3.666
2.925
2.902
2.884
2.866
2.848
2.829
2.811
1.534
1.171
1.153

4000

3000

2000

1000

0

1.00

2.81

0.96

8.85

2.97

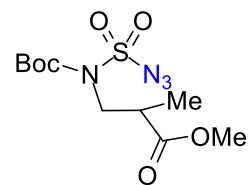
5.0

ppm (f1)

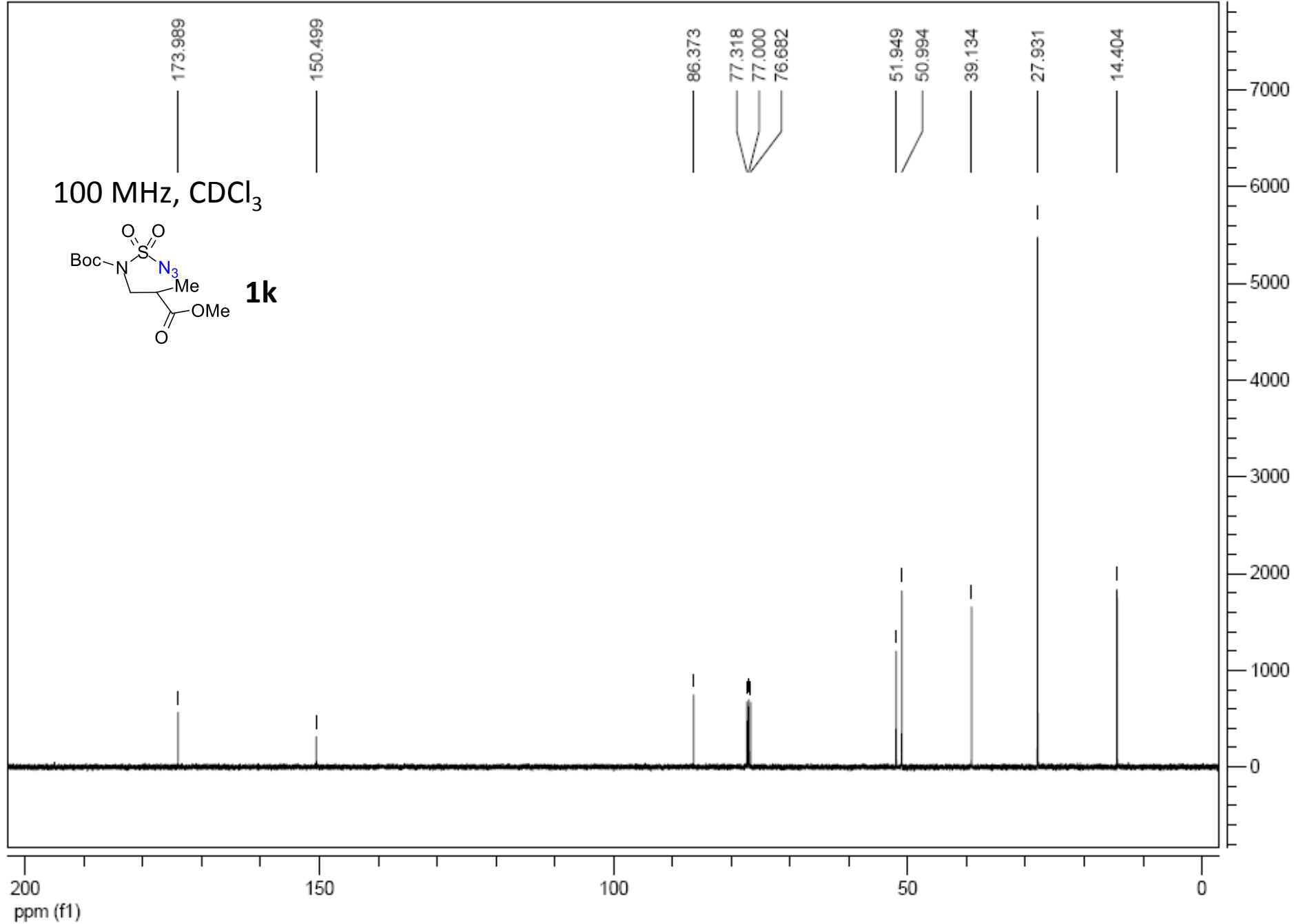
SII

47

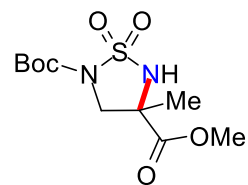
100 MHz, CDCl₃



1k



400 MHz, CDCl₃



2k

7.240

5.544

4.137

4.111

3.838

3.757

3.731

1.647

1.592

1500

1000

500

0

5.0

ppm (f1)

SII

49

1.00

1.11

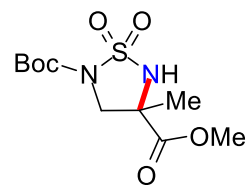
3.08

1.19

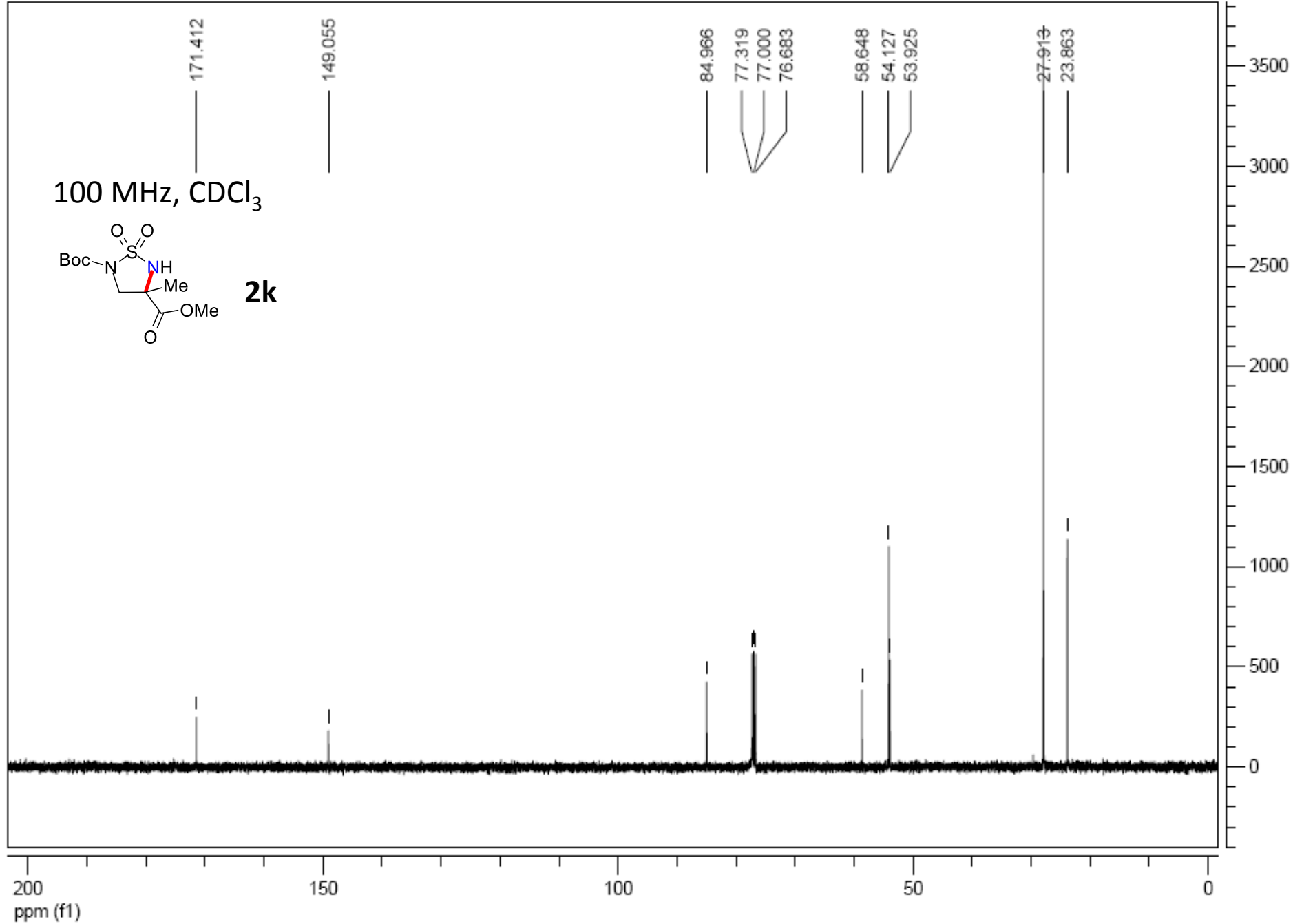
3.47

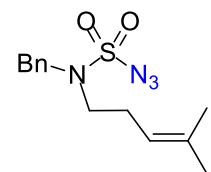
9.78

100 MHz, CDCl₃



2k





250 MHz, CDCl₃

1l

7.365
7.359
7.348
7.345
7.334
7.323
7.316
7.240

4.985
4.980
4.974
4.968
4.961
4.956
4.950
4.945
4.933
4.927
4.921
4.916
4.456

3.196
3.172
3.166
3.156
3.133

2.262
2.232
2.200
2.170
1.646
1.527

5.17

1.00

2.00

2.06

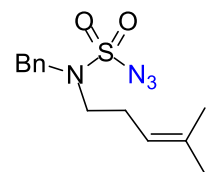
2.04

6.44

5.0 SII

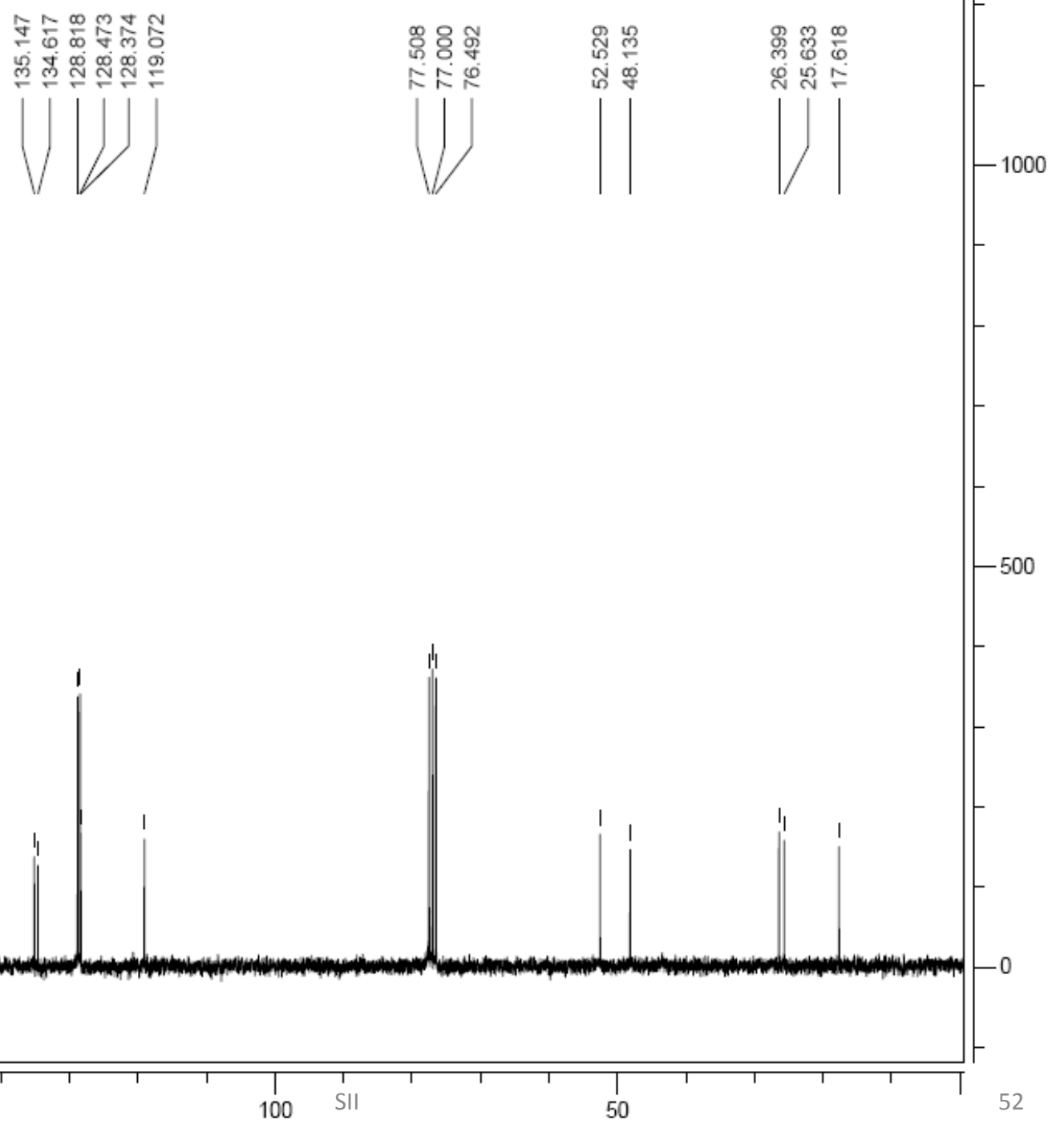
ppm (f1)

51

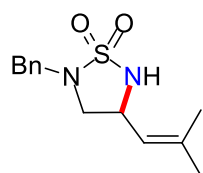


1l

62.9 MHz, CDCl₃



400 MHz, CDCl₃



21

7.351
7.344
7.320
7.318
7.308
7.300
7.240

5.146
5.124
4.485
4.468
4.449
4.431
4.411
4.253
4.218
4.096
4.062
3.286
3.268
3.263
3.246
2.971
2.952
2.928
1.694
1.643

4.66

0.96

1.00

1.96

1.00

1.01

1.00

6.94

ppm (f1)

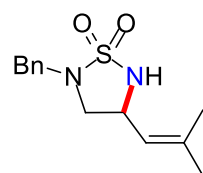
5.0

SII

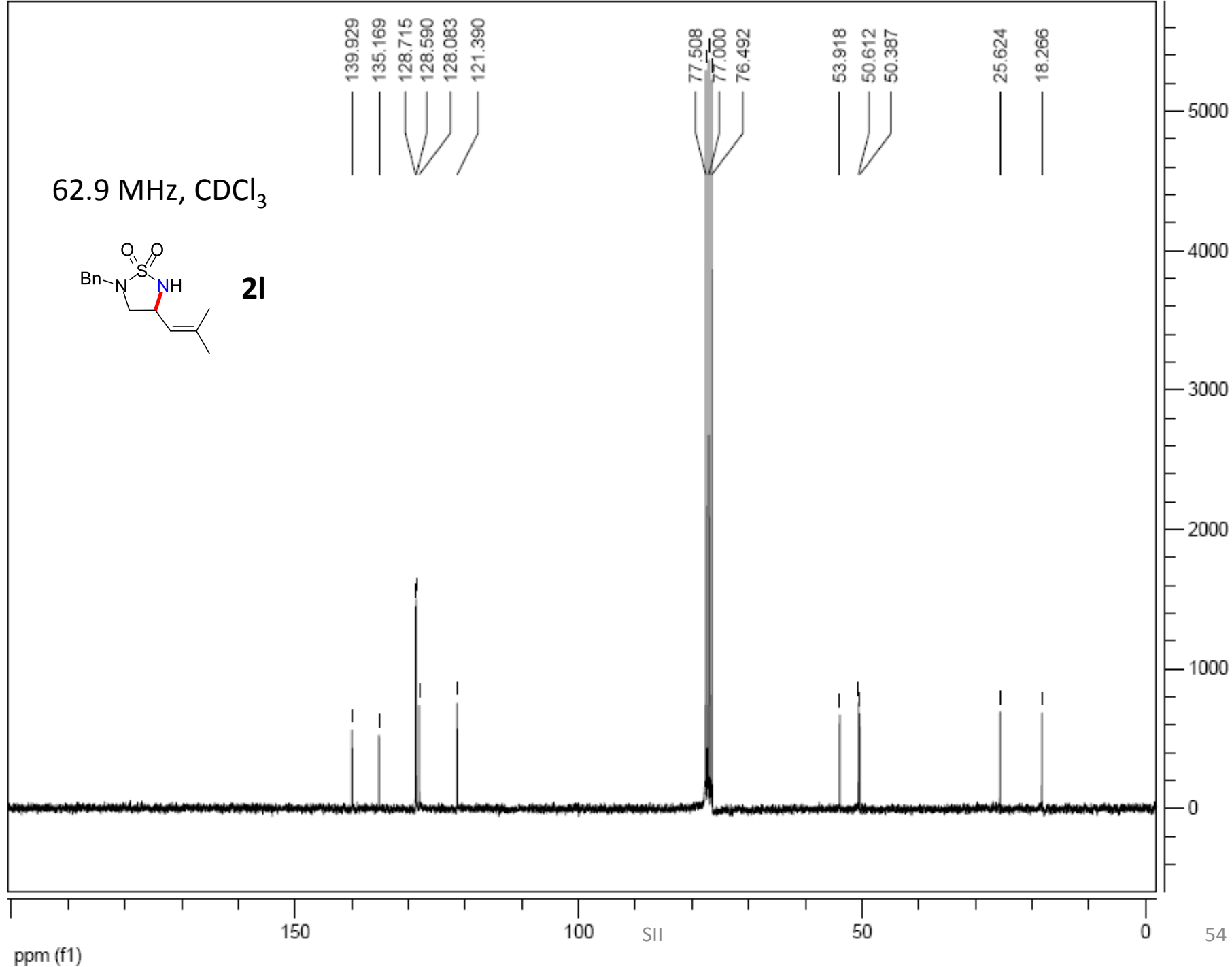
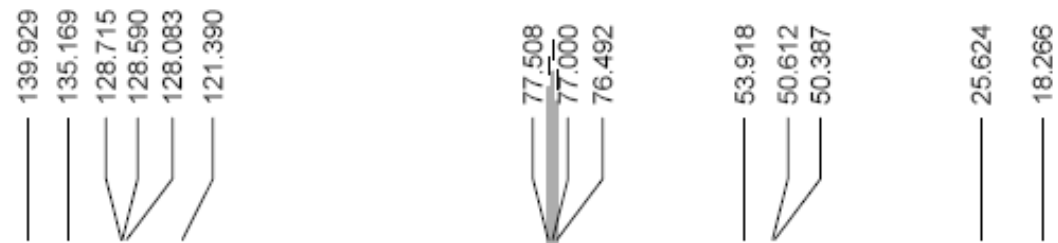
0.0

53

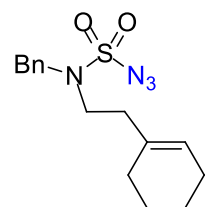
62.9 MHz, CDCl₃



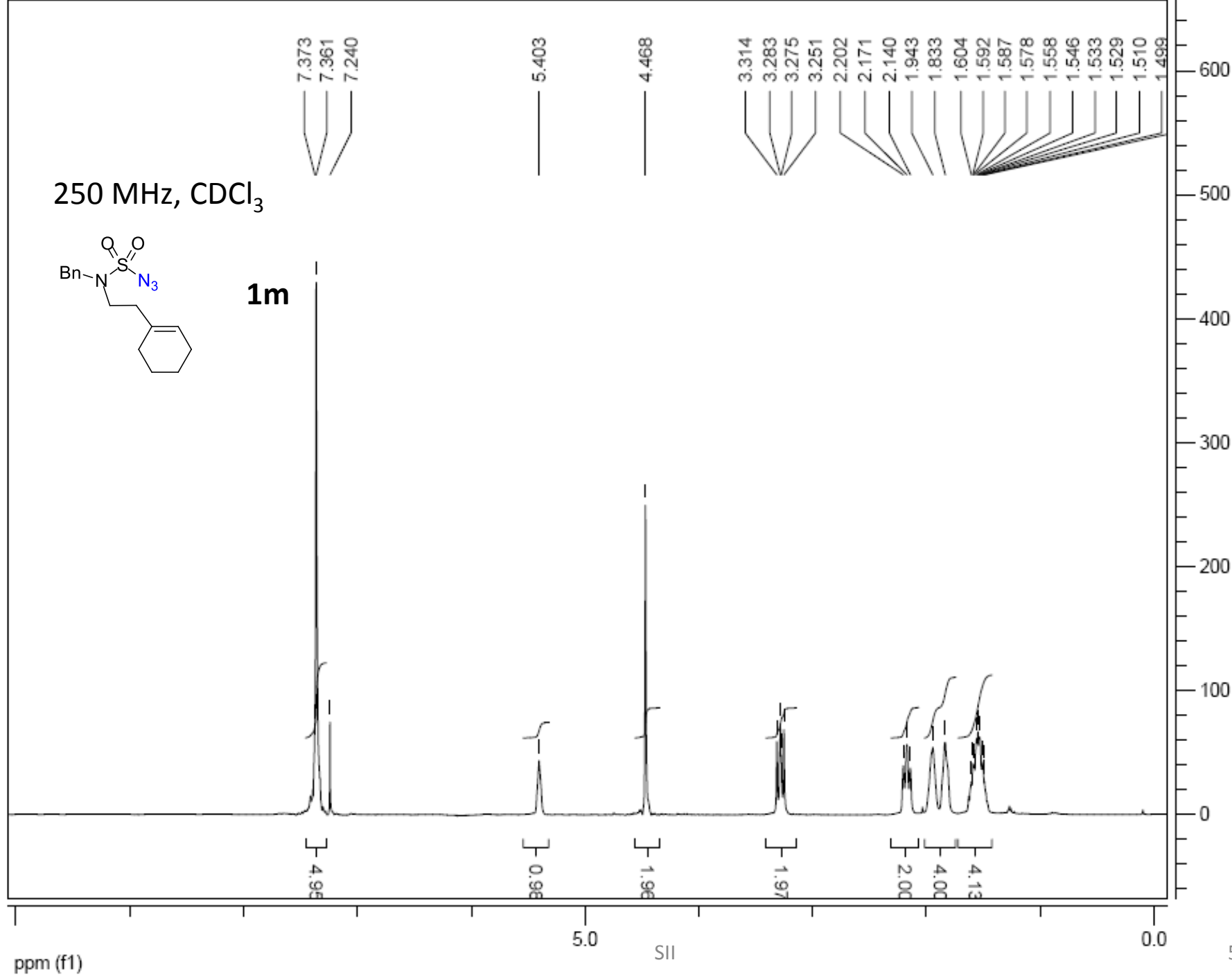
2l



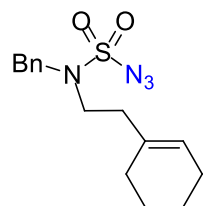
250 MHz, CDCl₃



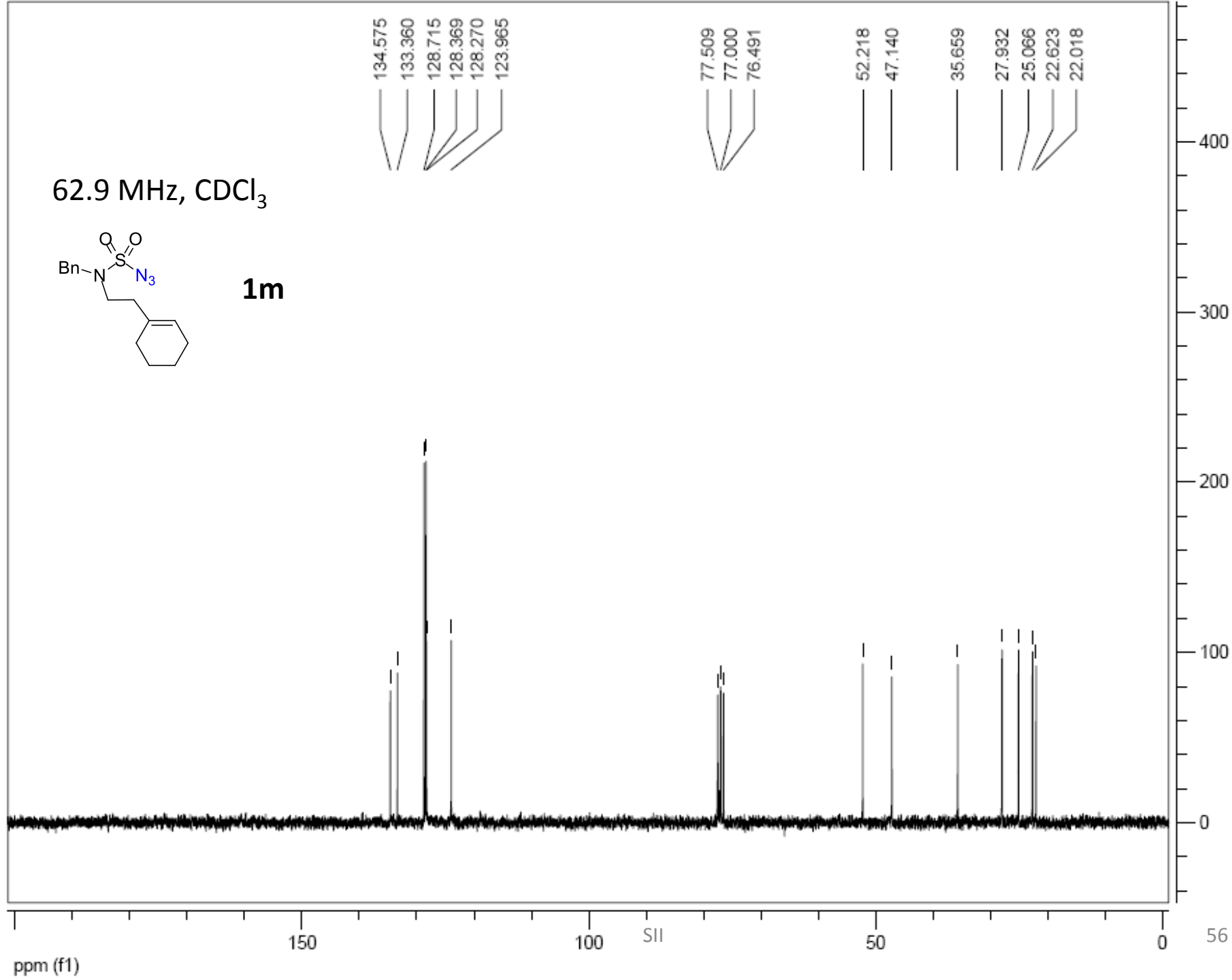
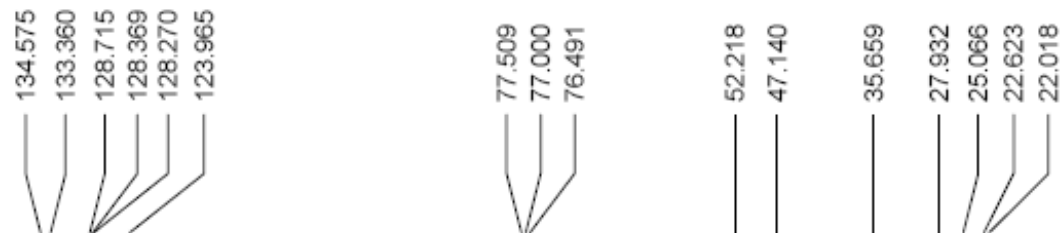
1m



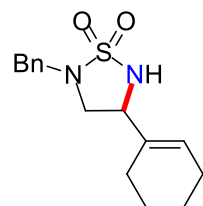
62.9 MHz, CDCl₃



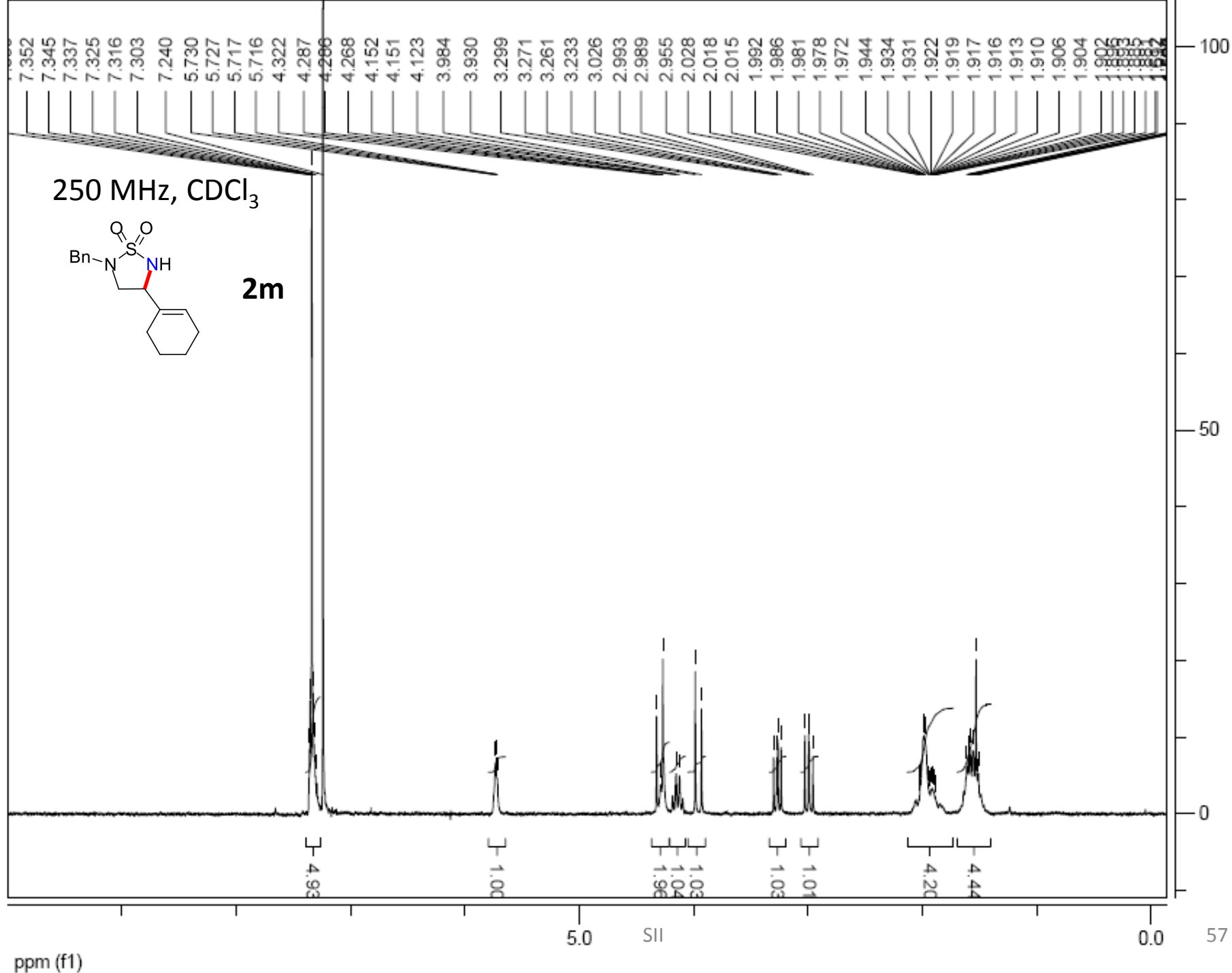
1m



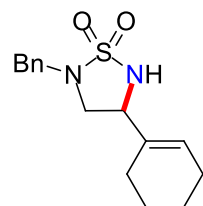
250 MHz, CDCl₃



2m



100 MHz, CDCl₃



2m

135.076
133.870
128.672
128.554
128.045
126.058

77.317
77.000
76.680

57.687
51.739
50.378

24.877
23.675
22.230
22.051

1500

1000

500

0

58

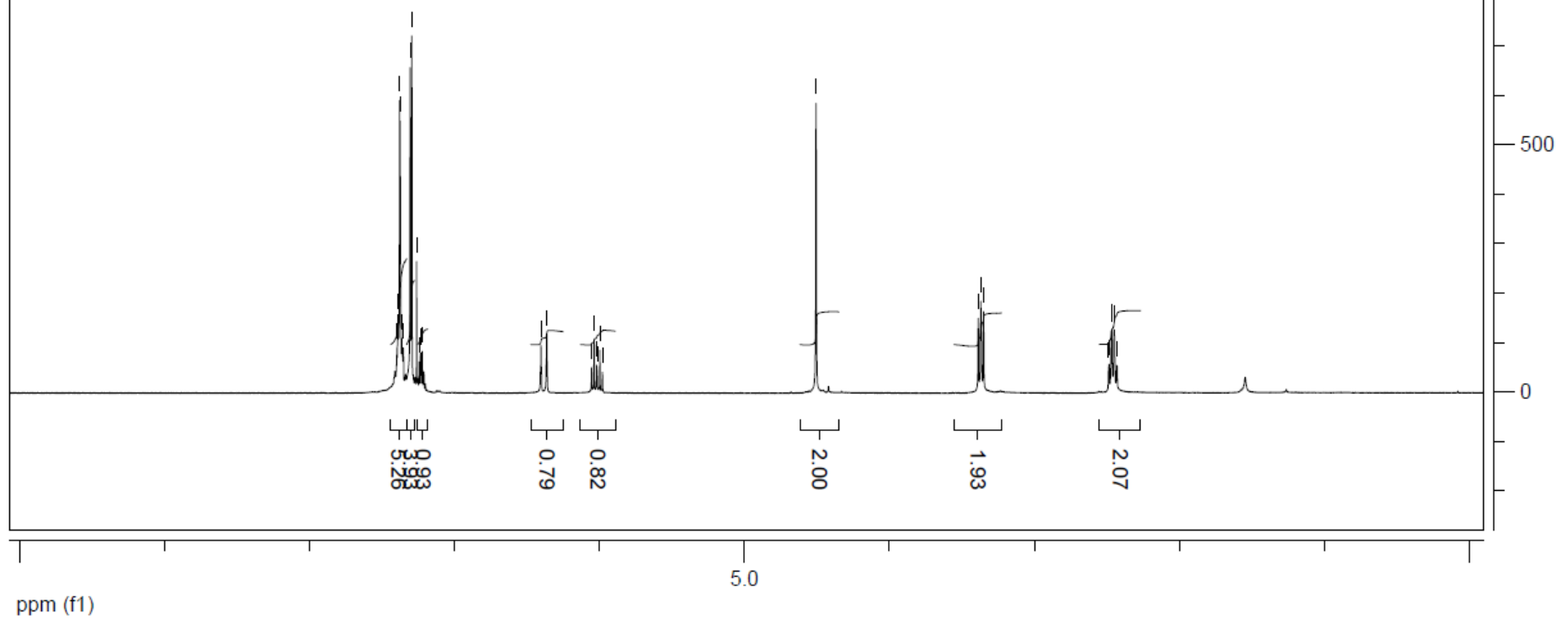
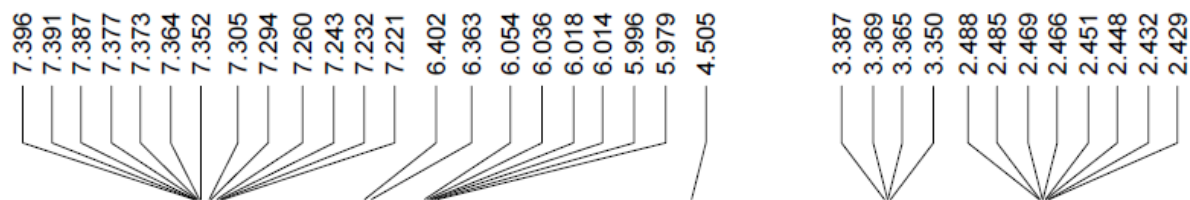
SII

100

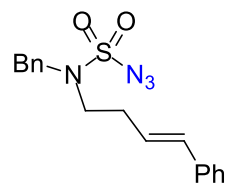
150

ppm (f1)

 **1n**



100 MHz, CDCl₃



1n

136.892
134.536
132.859
128.923
128.539
128.507
127.447
126.077
125.191

77.318
77.000
76.682

52.891
48.339

31.365

15000

10000

5000

0

ppm (f1)

150

100

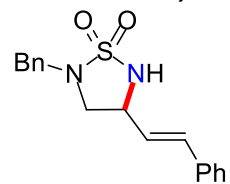
50

0

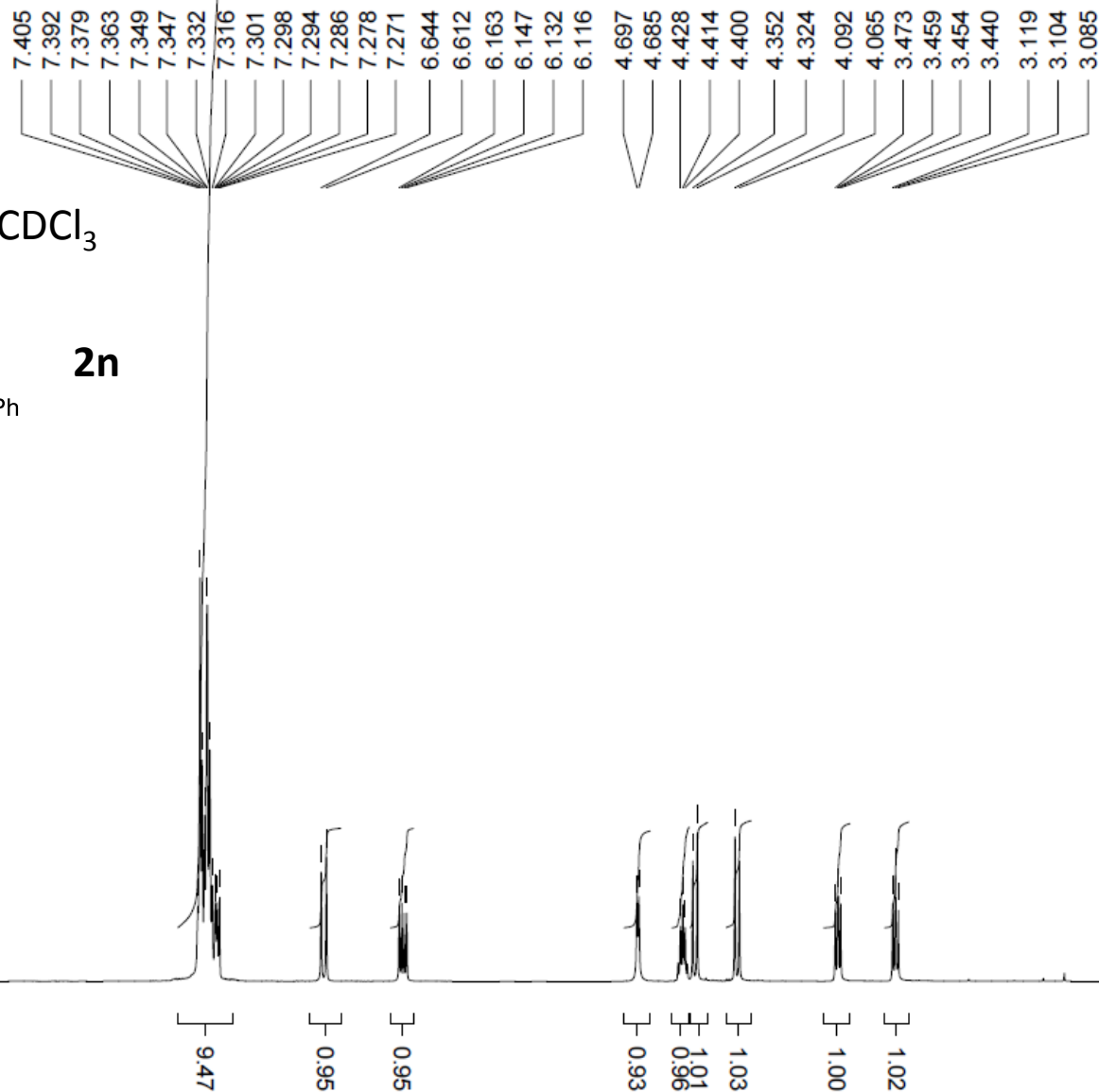
SII

60

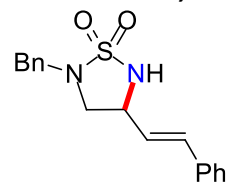
500 MHz, CDCl₃



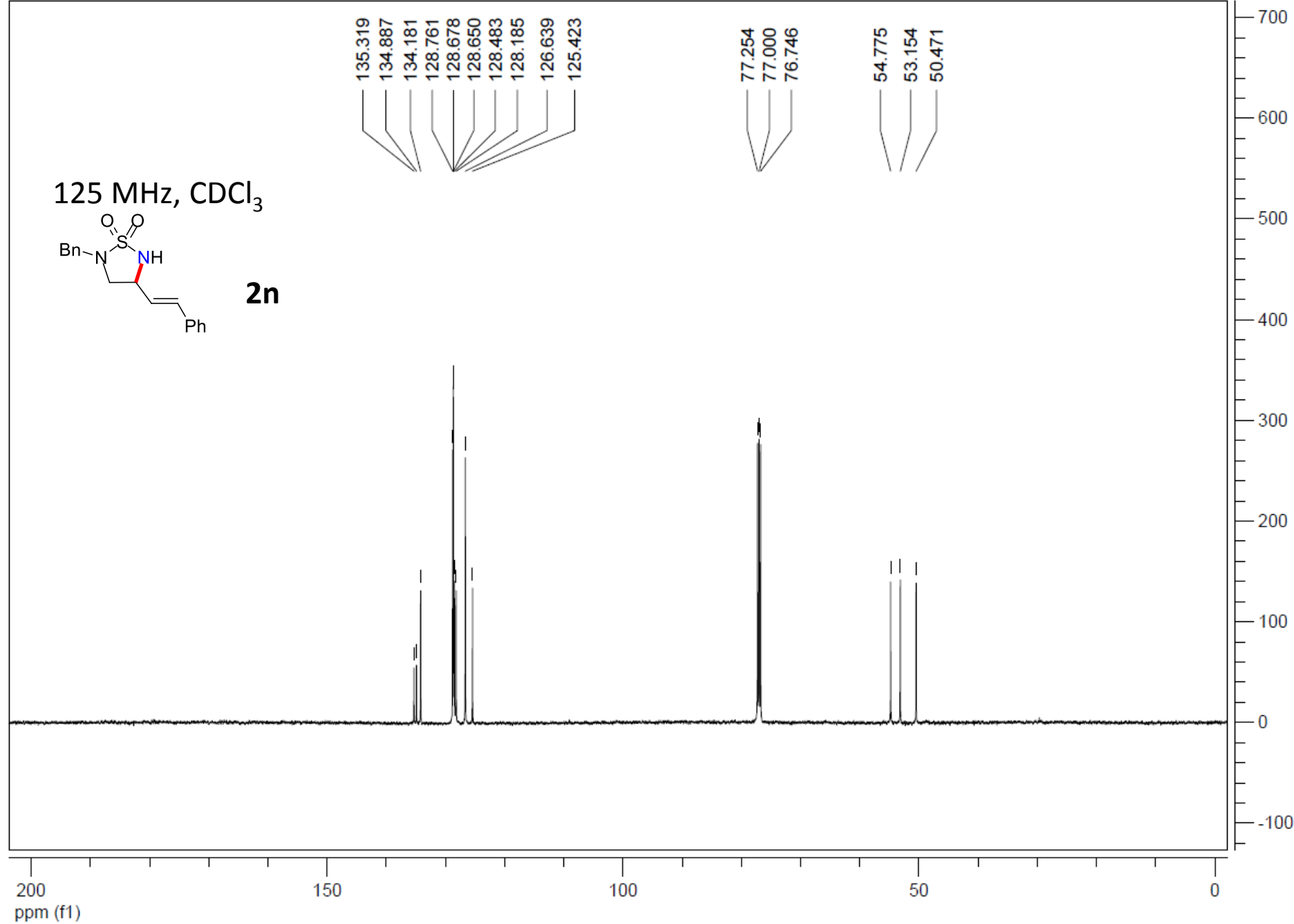
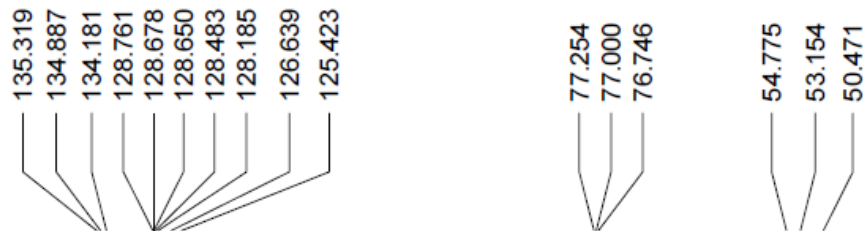
2n



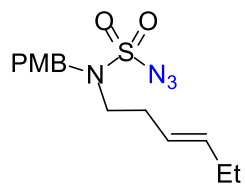
125 MHz, CDCl₃



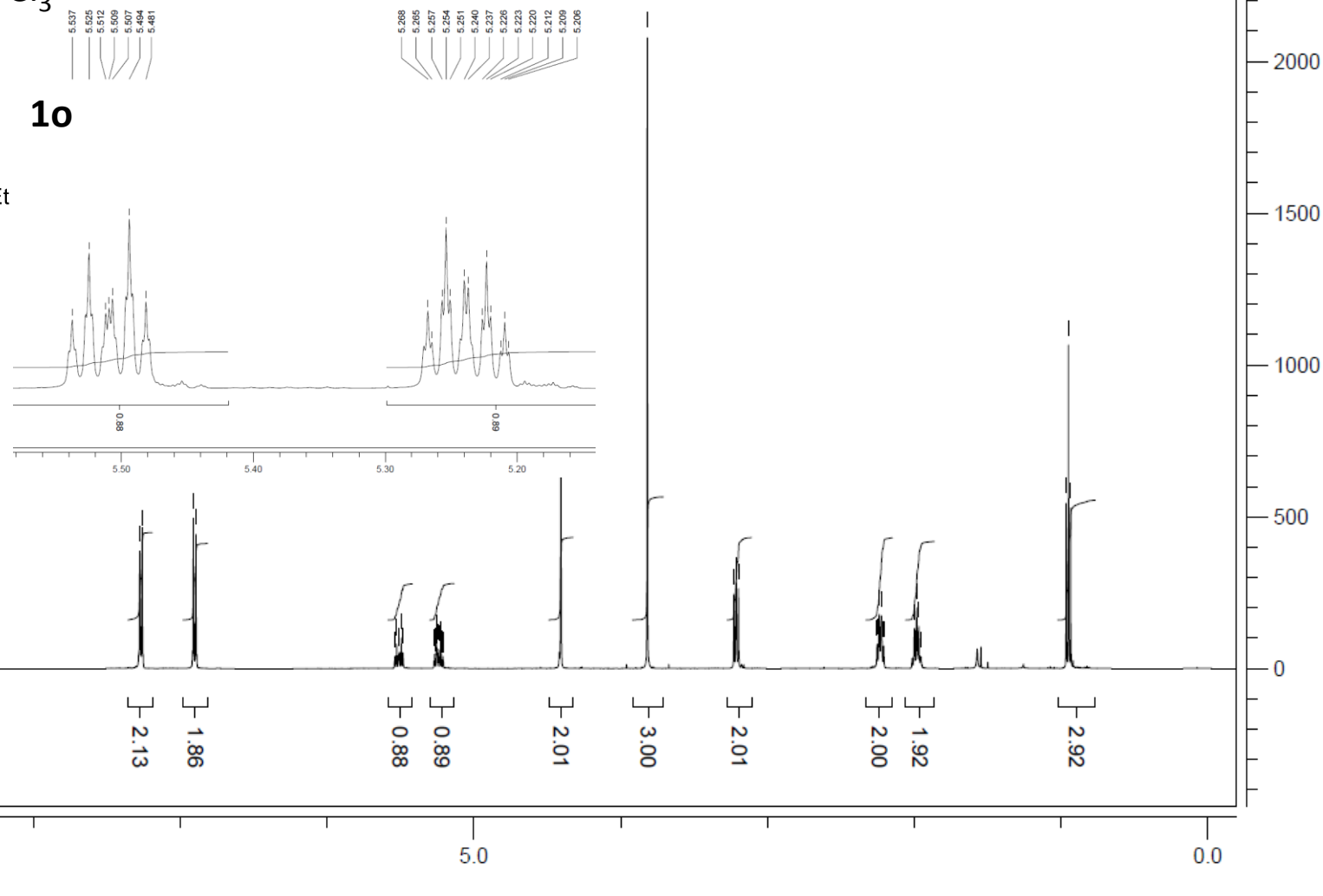
2n

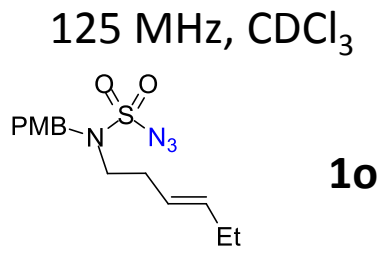


500 MHz, CDCl₃

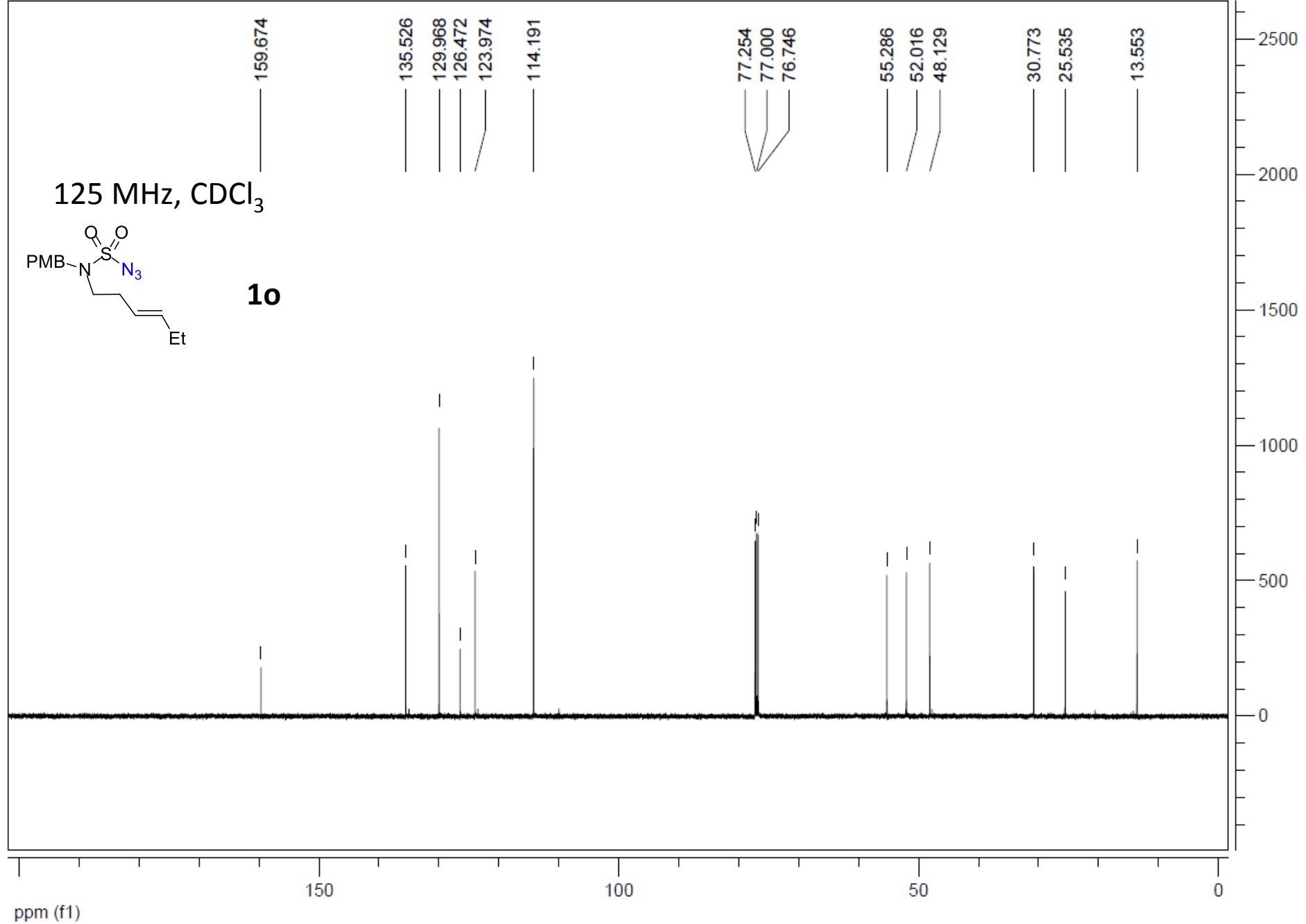


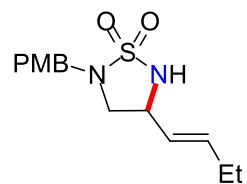
1o





159.674
135.526
129.968
126.472
123.974
114.191
77.254
77.000
76.746
55.286
52.016
48.129
30.773
25.535
13.553

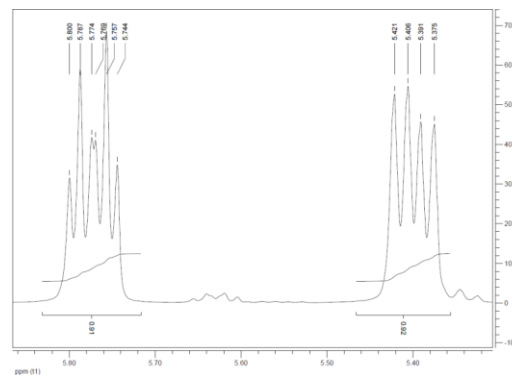
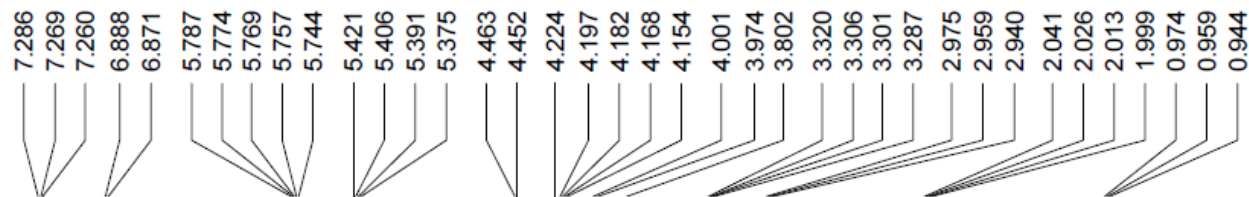




500 MHz, CDCl₃

2o

500M



Integration values for the peaks in the main spectrum:

- 1.95, 1.92, 0.91, 0.92, 0.87, 1.94, 0.99, 2.96, 1.00, 1.00, 1.97, 3.08

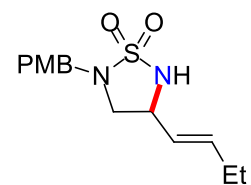
10.0
ppm (f1)

5.0

0.0

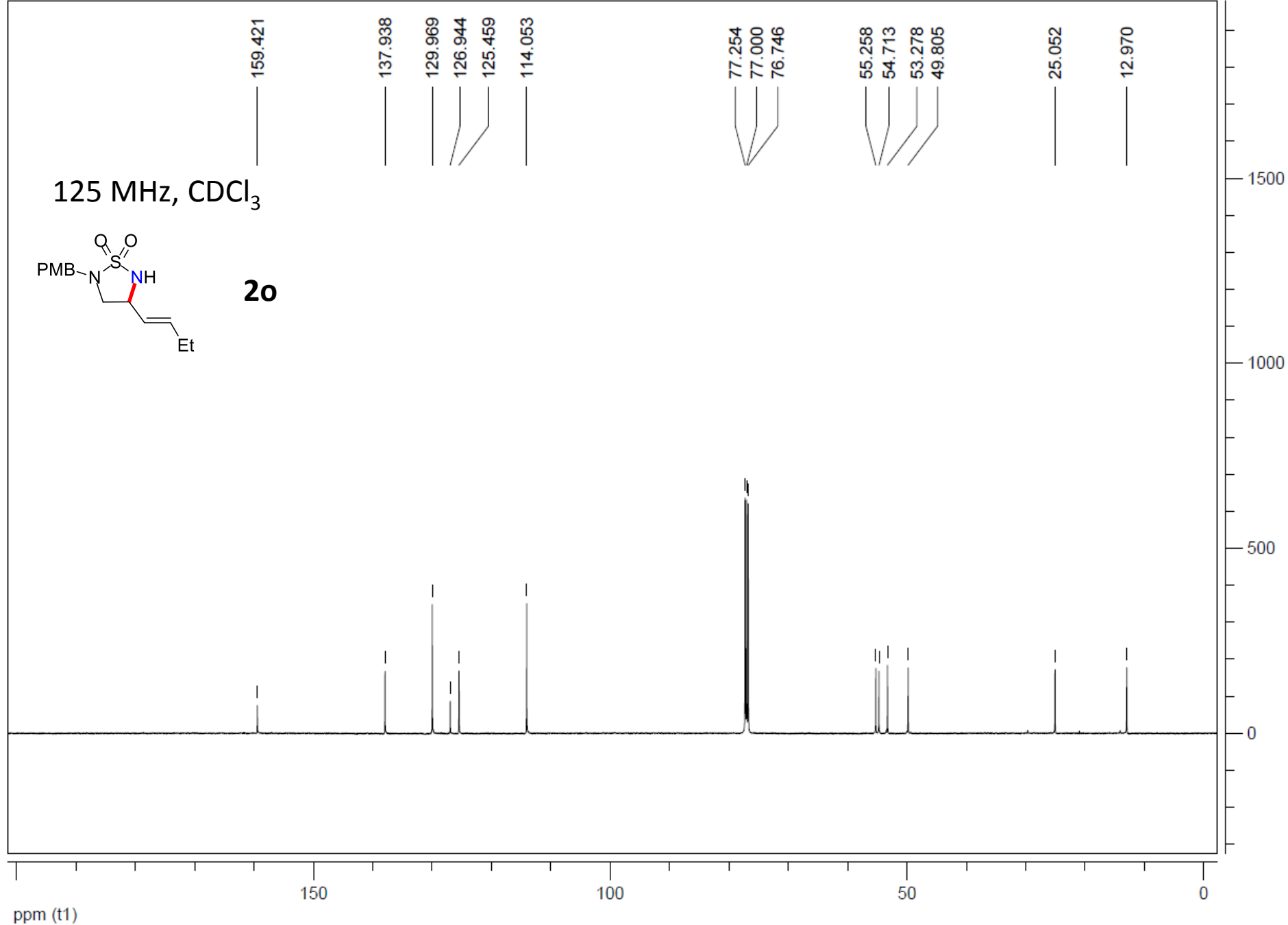
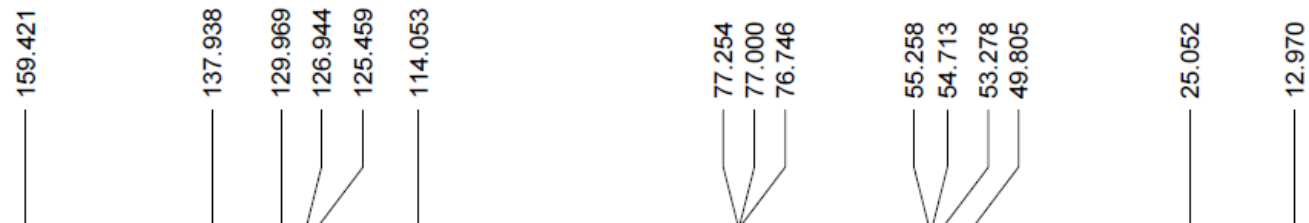
SII

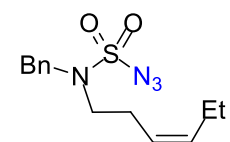
65



2o

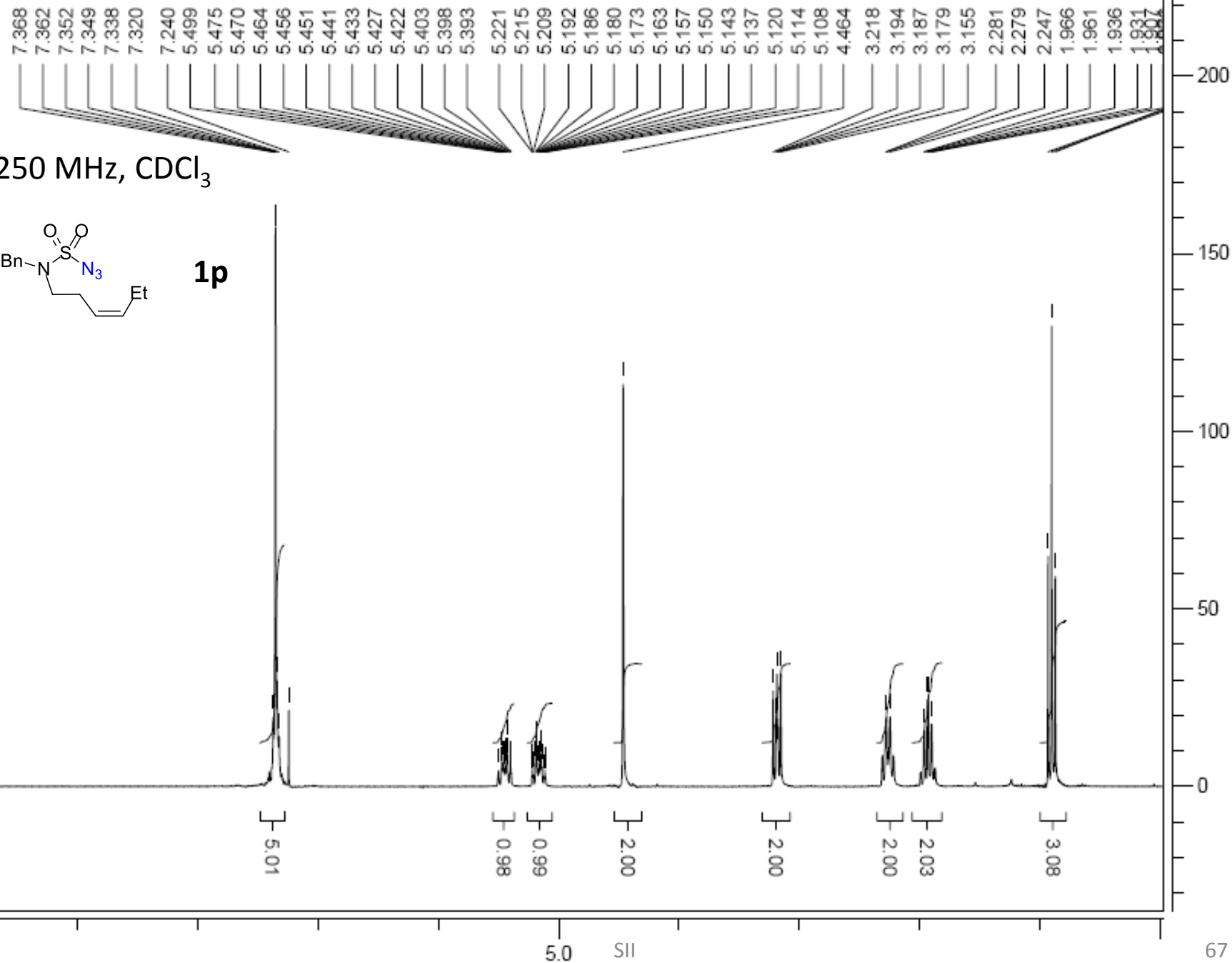
125 MHz, CDCl₃



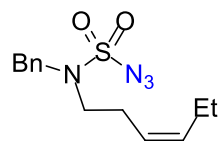


250 MHz, CDCl₃

1p



62.9 MHz, CDCl₃



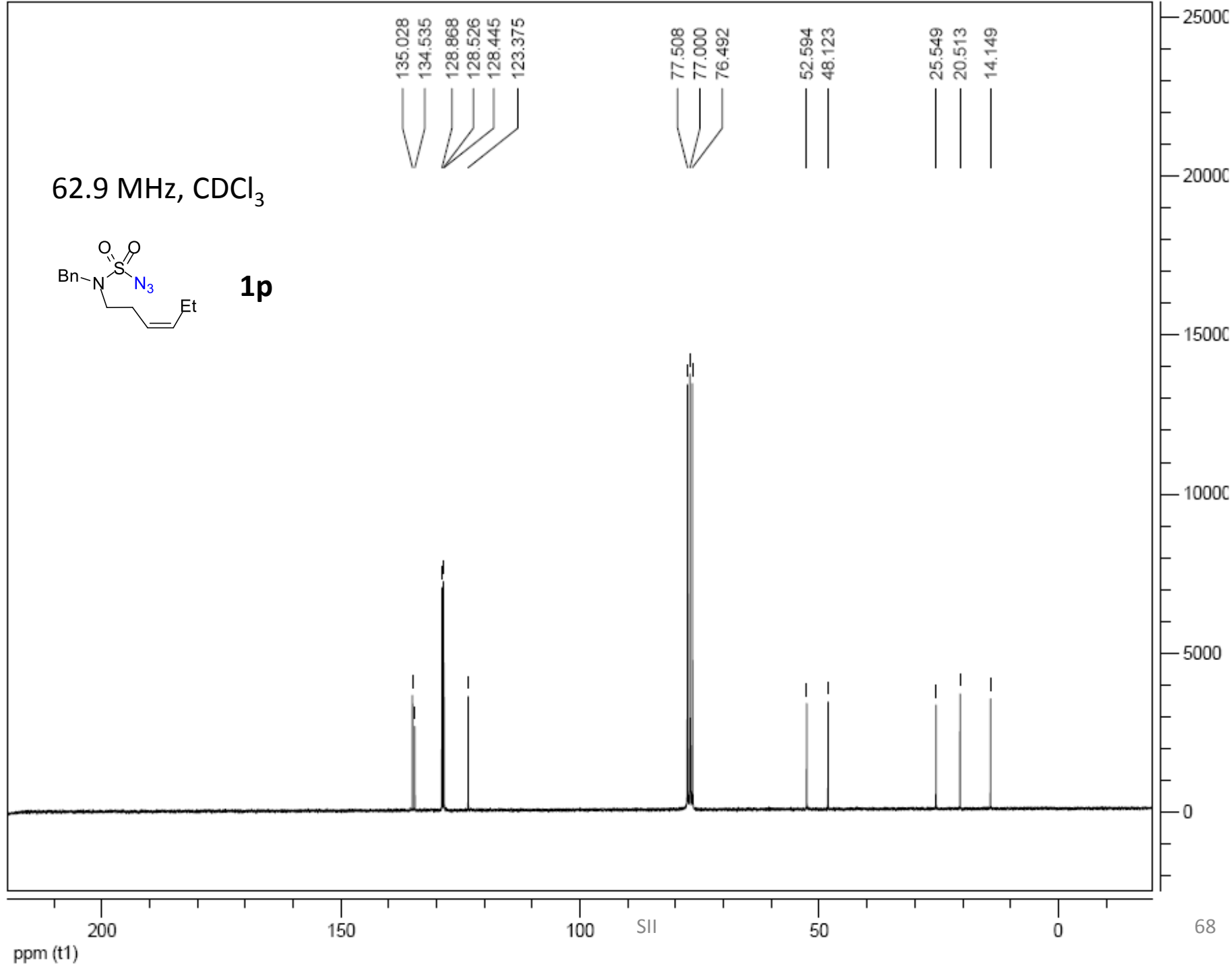
1p

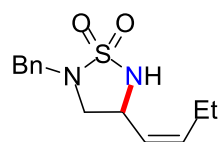
135.028
134.535
128.868
128.526
128.445
123.375

77.508
77.000
76.492

52.594
48.123

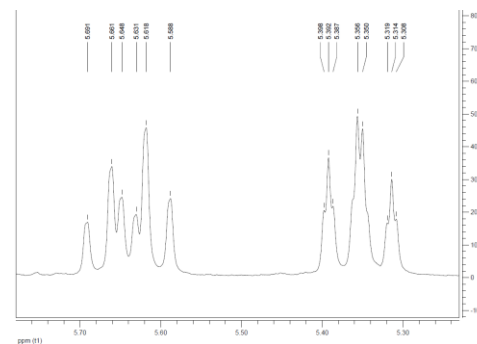
25.549
20.513
14.149





2p

250 MHz, CDCl₃



250M

500

0

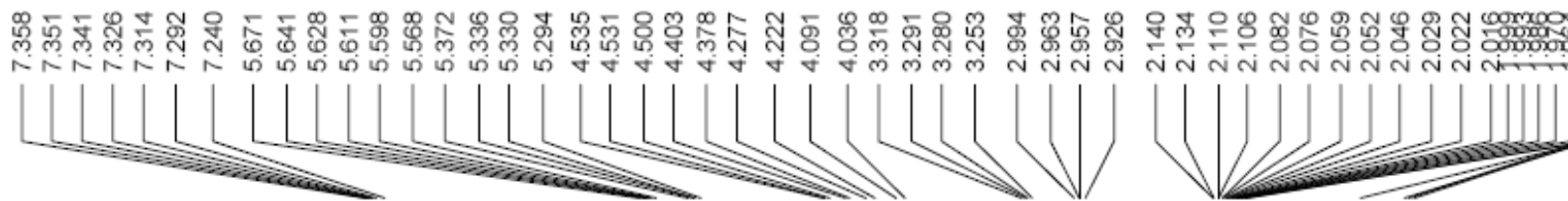
69

5.0

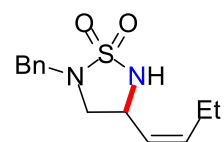
SII

0.0

ppm (t1)



62.9 MHz, CDCl₃



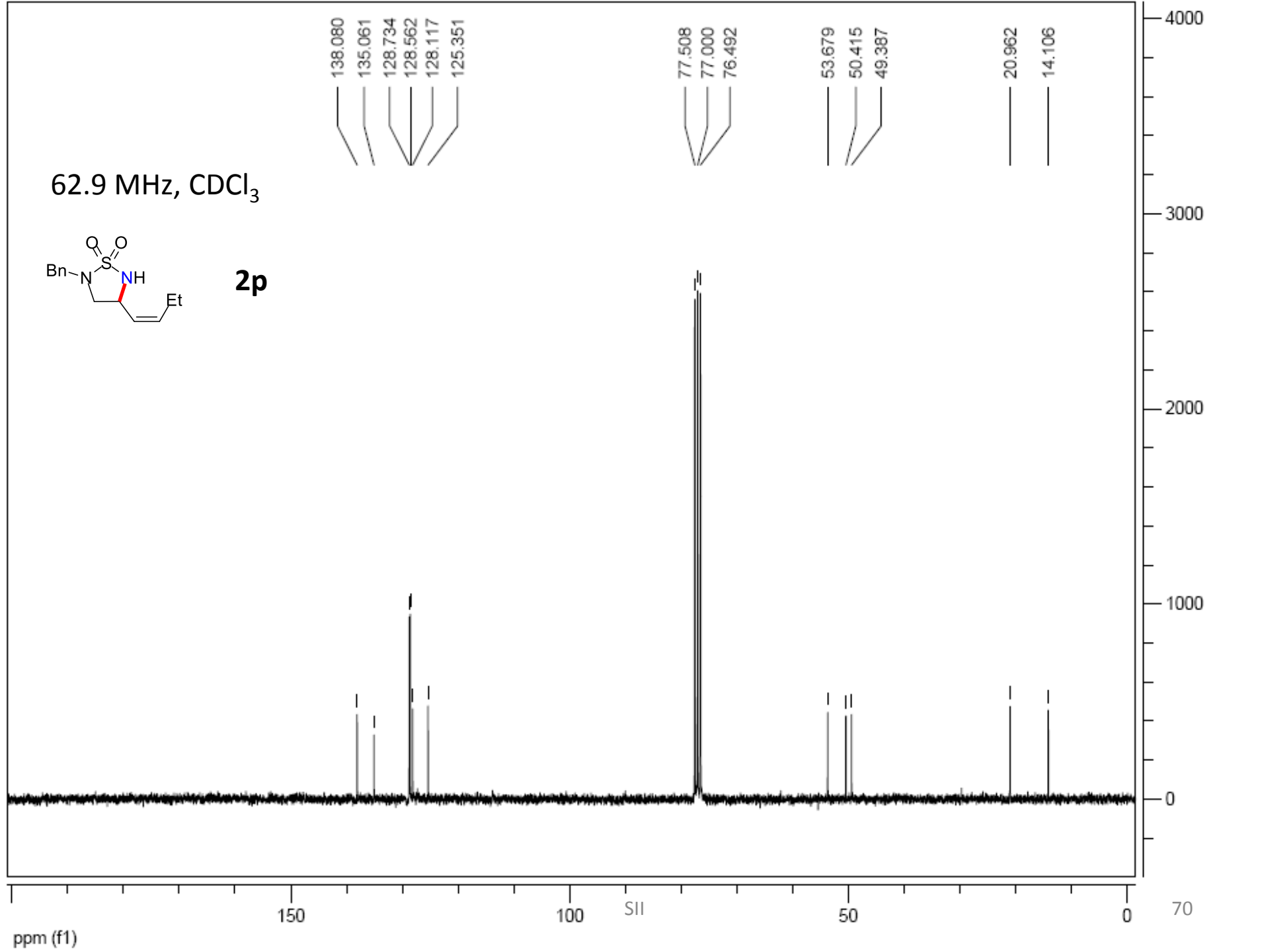
2p

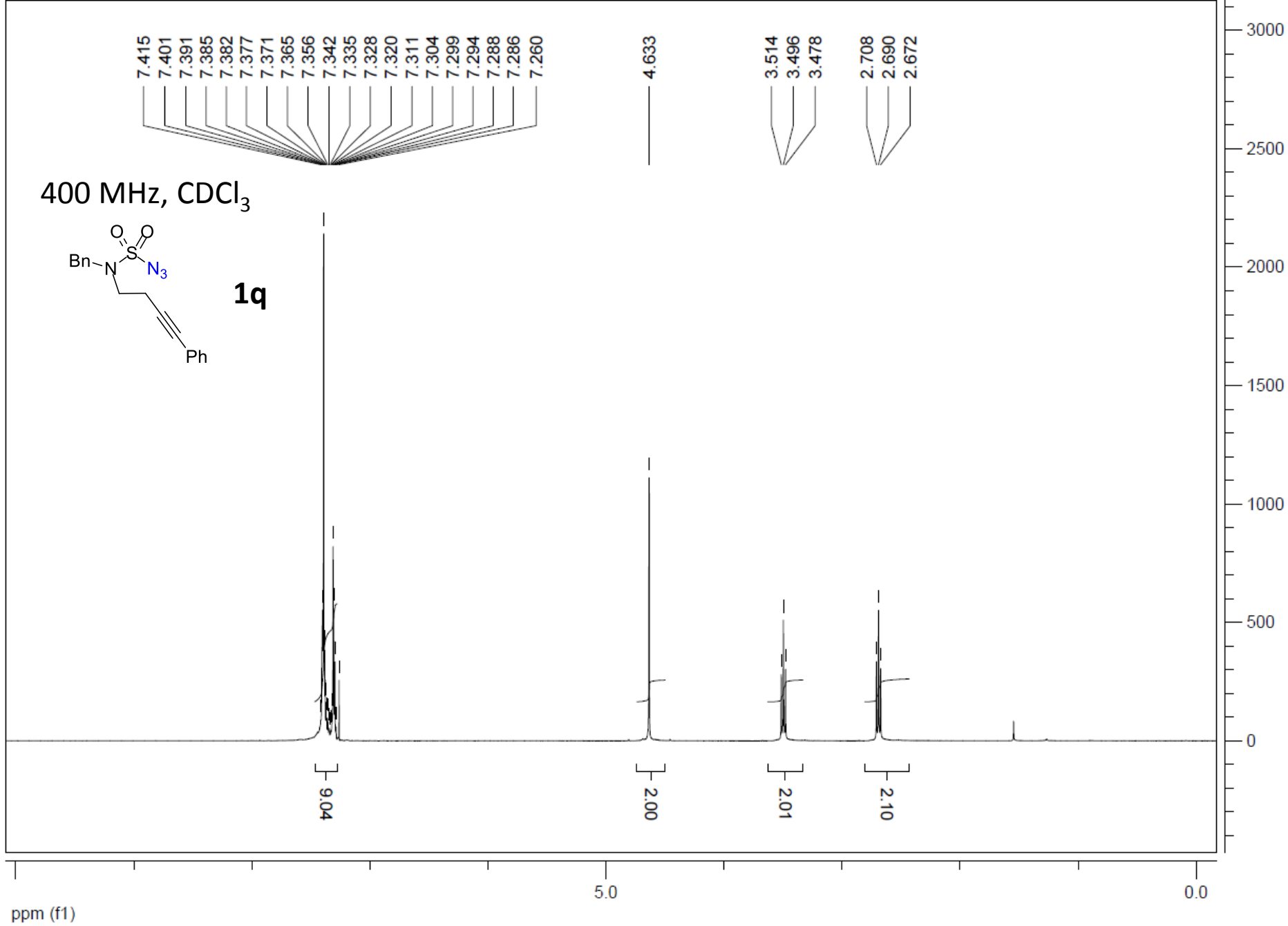
138.080
135.061
128.734
128.562
128.117
125.351

77.508
77.000
76.492

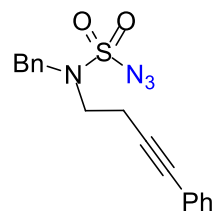
53.679
50.415
49.387

20.962
14.106





100 MHz, CDCl₃



1q

134.462
131.522
128.975
128.562
128.514
128.300
128.124
122.988

85.575
82.796
77.318
77.000
76.682

53.190
47.107

19.163

20000

15000

10000

5000

0

150

100

50

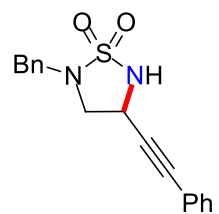
0

ppm (f1)

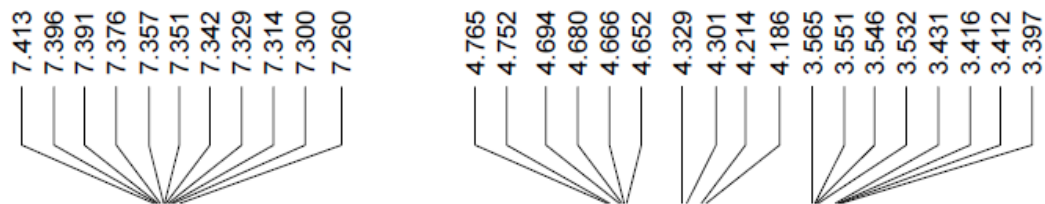
SII

72

500 MHz, CDCl₃



2q



9.15

0.95

1.00

1.05

ppm (f1)

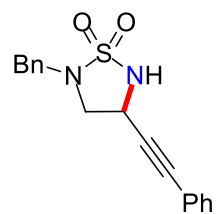
5.0

0.0

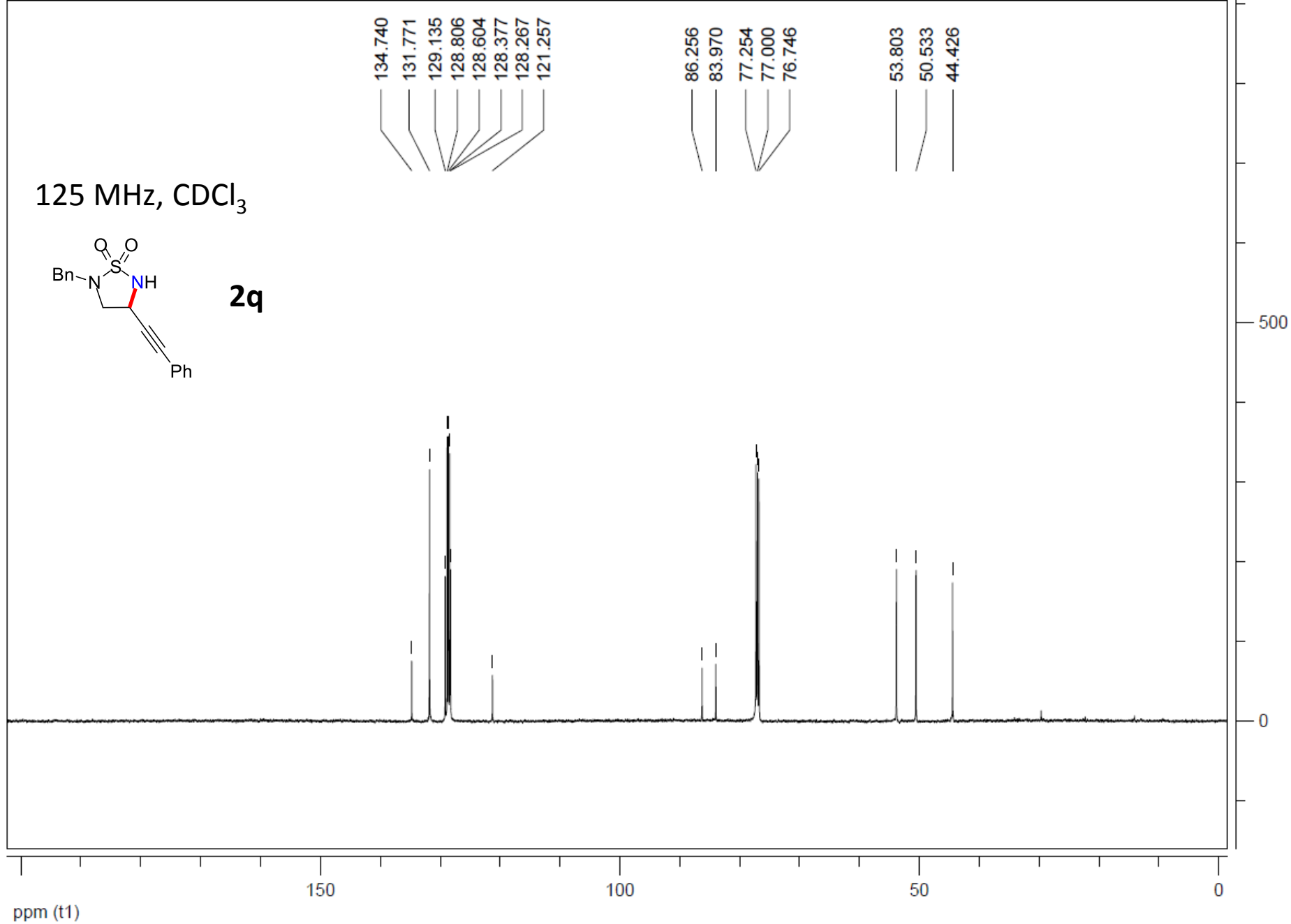
SII

73

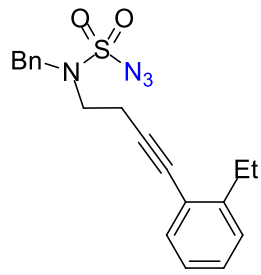
125 MHz, CDCl₃



2q



500 MHz, CDCl₃



7.401
7.392
7.386
7.378
7.369
7.361
7.350
7.265
7.260
7.250
7.235
7.233
7.210
7.197
7.146
7.143
7.131
7.129
7.116
7.114

4.631

3.521
3.507
3.492
2.798
2.783
2.767
2.752
2.747
2.733
2.718

1.245
1.230
1.215

5.42
2.81
2.84

2.00

1.98

4.87
4.89

2.92

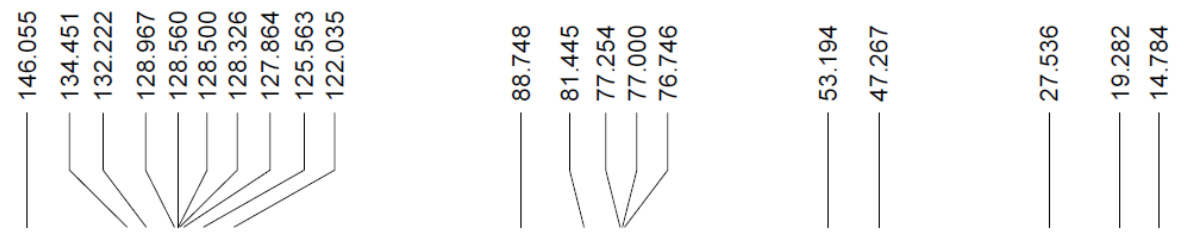
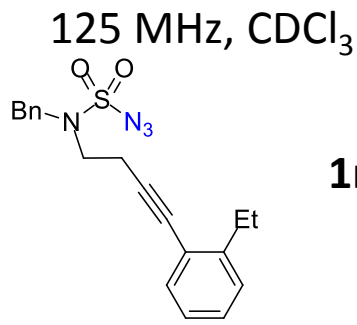
ppm (f1)

5.0

0.0

SII

75

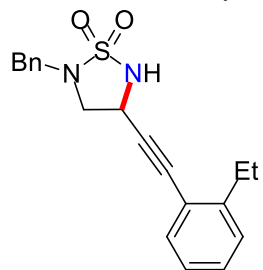


ppm (f1)

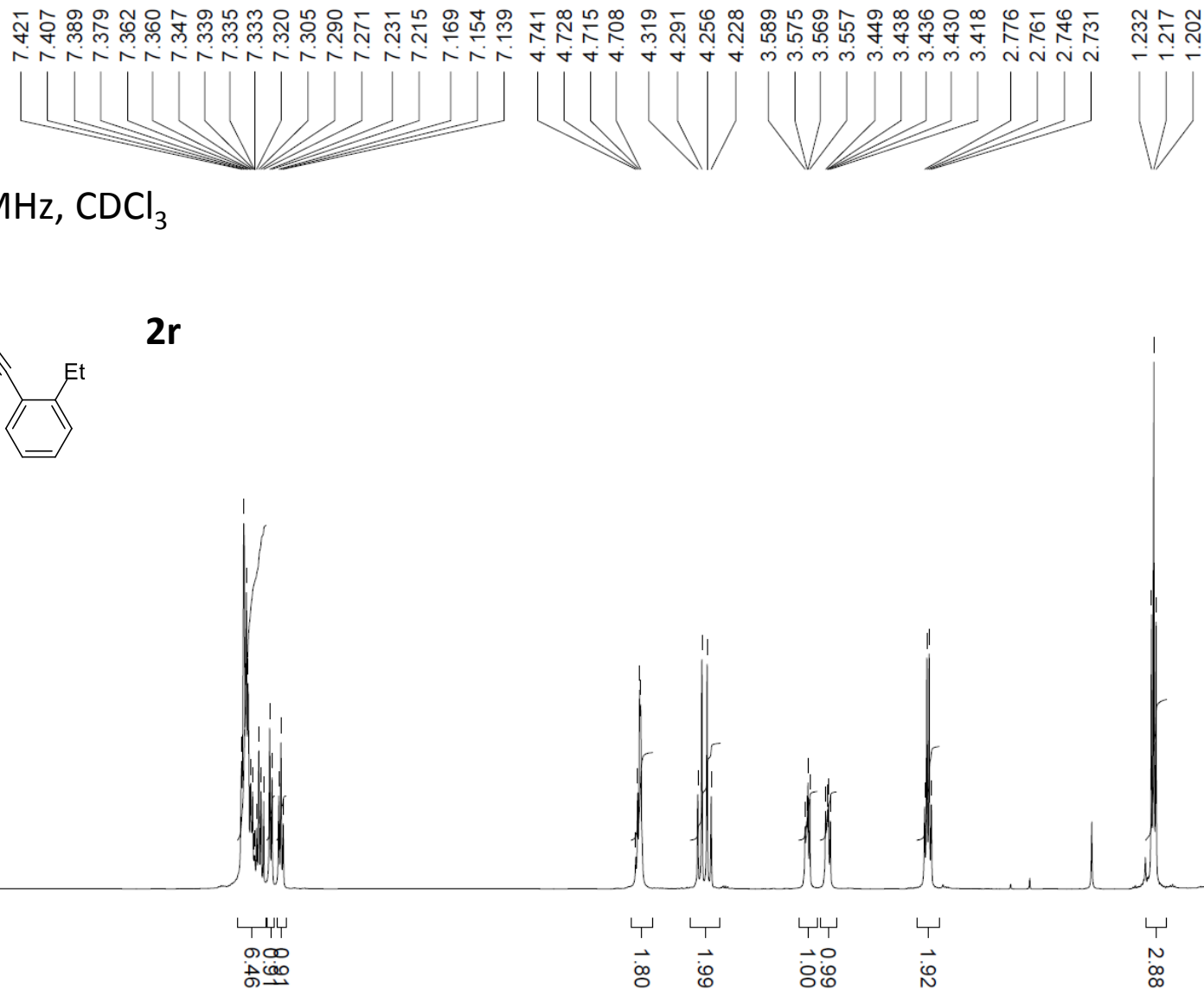
SII

76

500 MHz, CDCl₃



2r



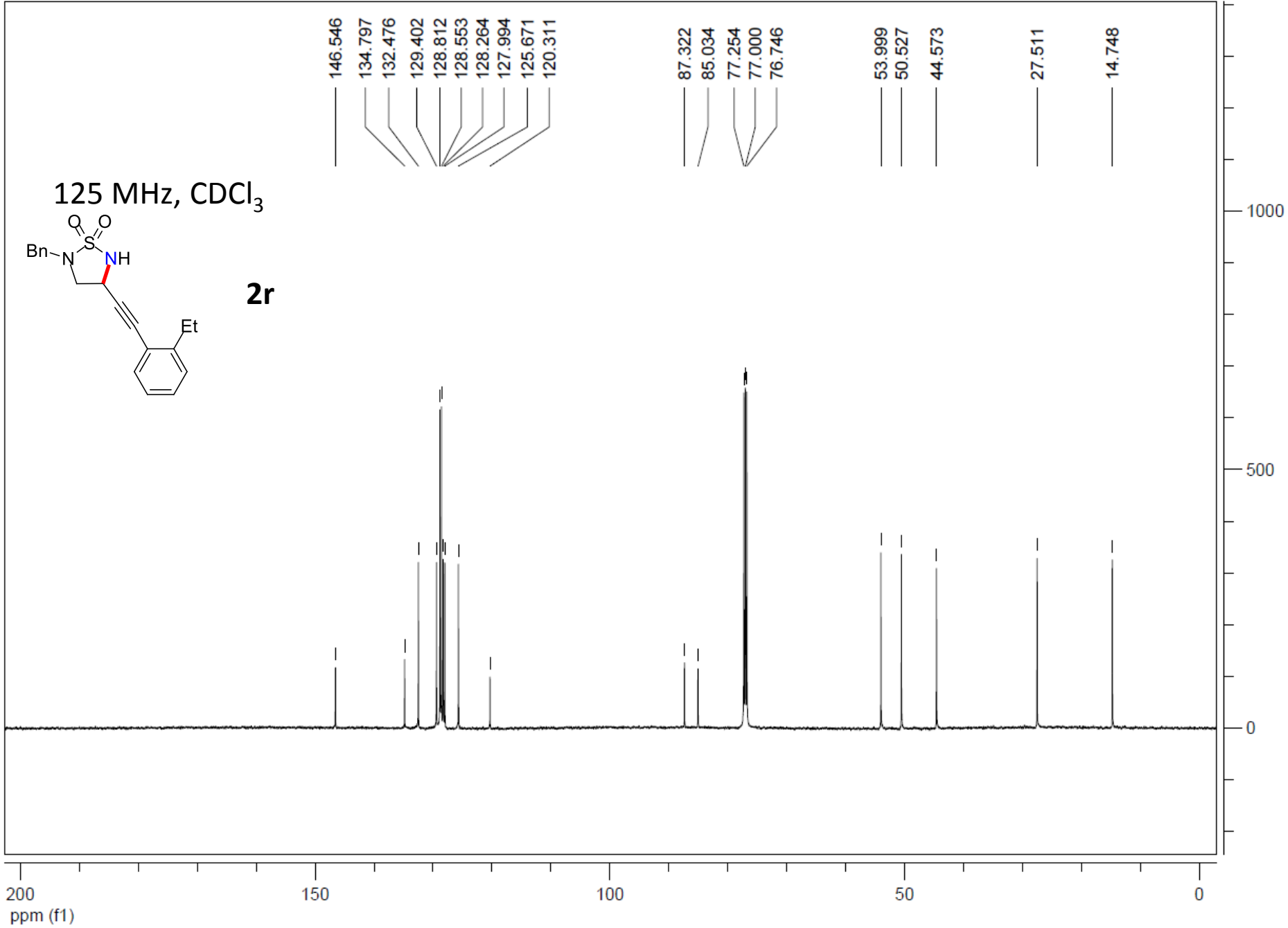
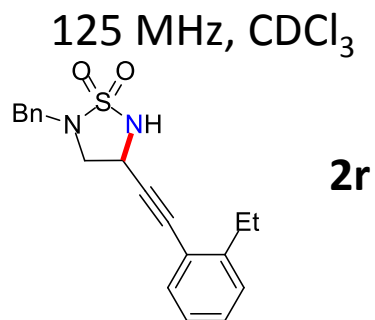
ppm (f1)

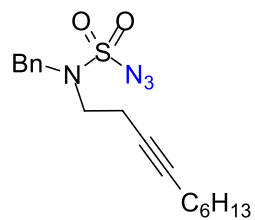
5.0

0.0

SII

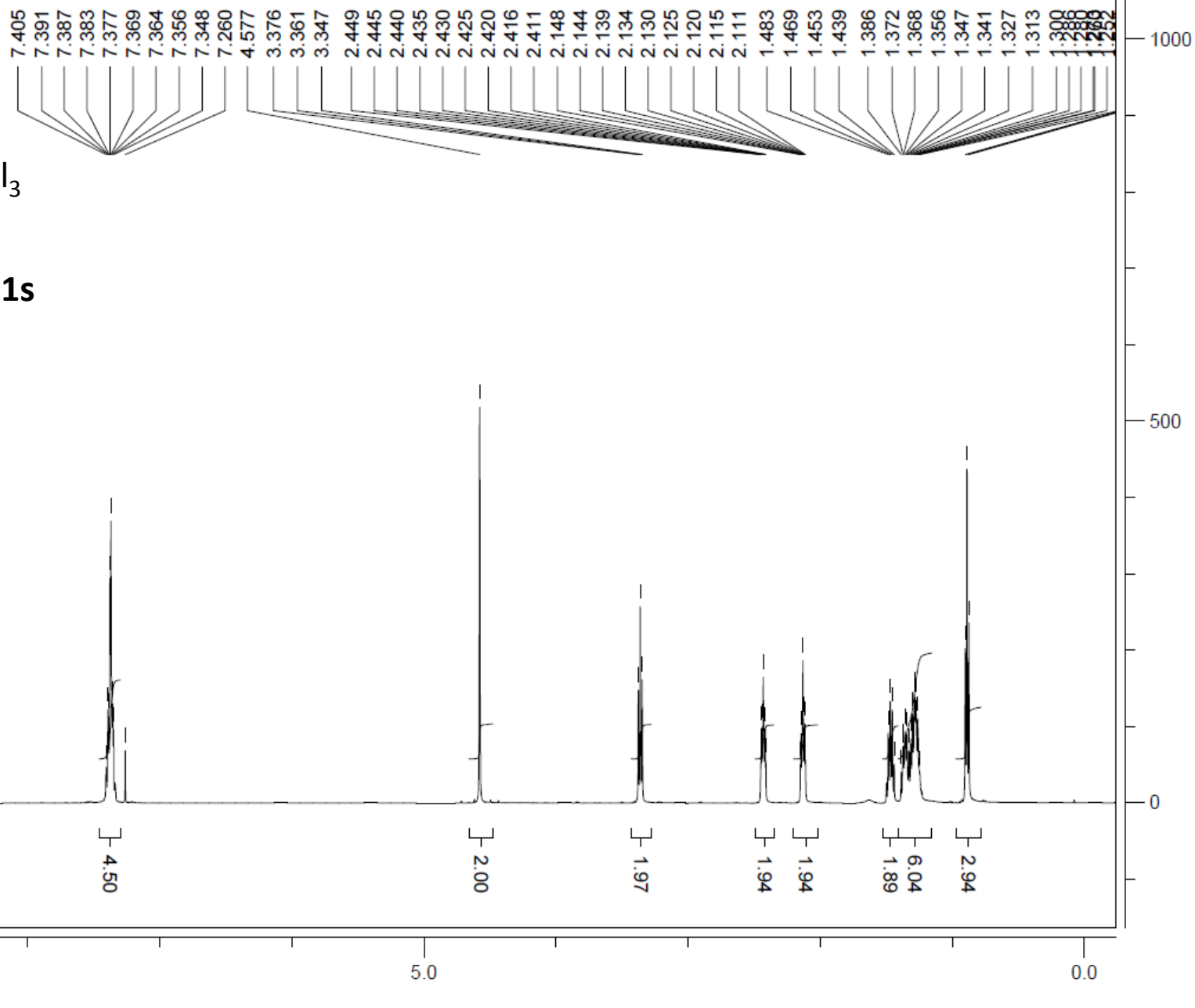
77



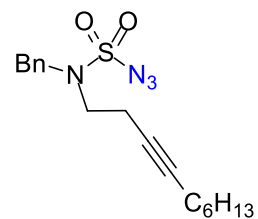


1s

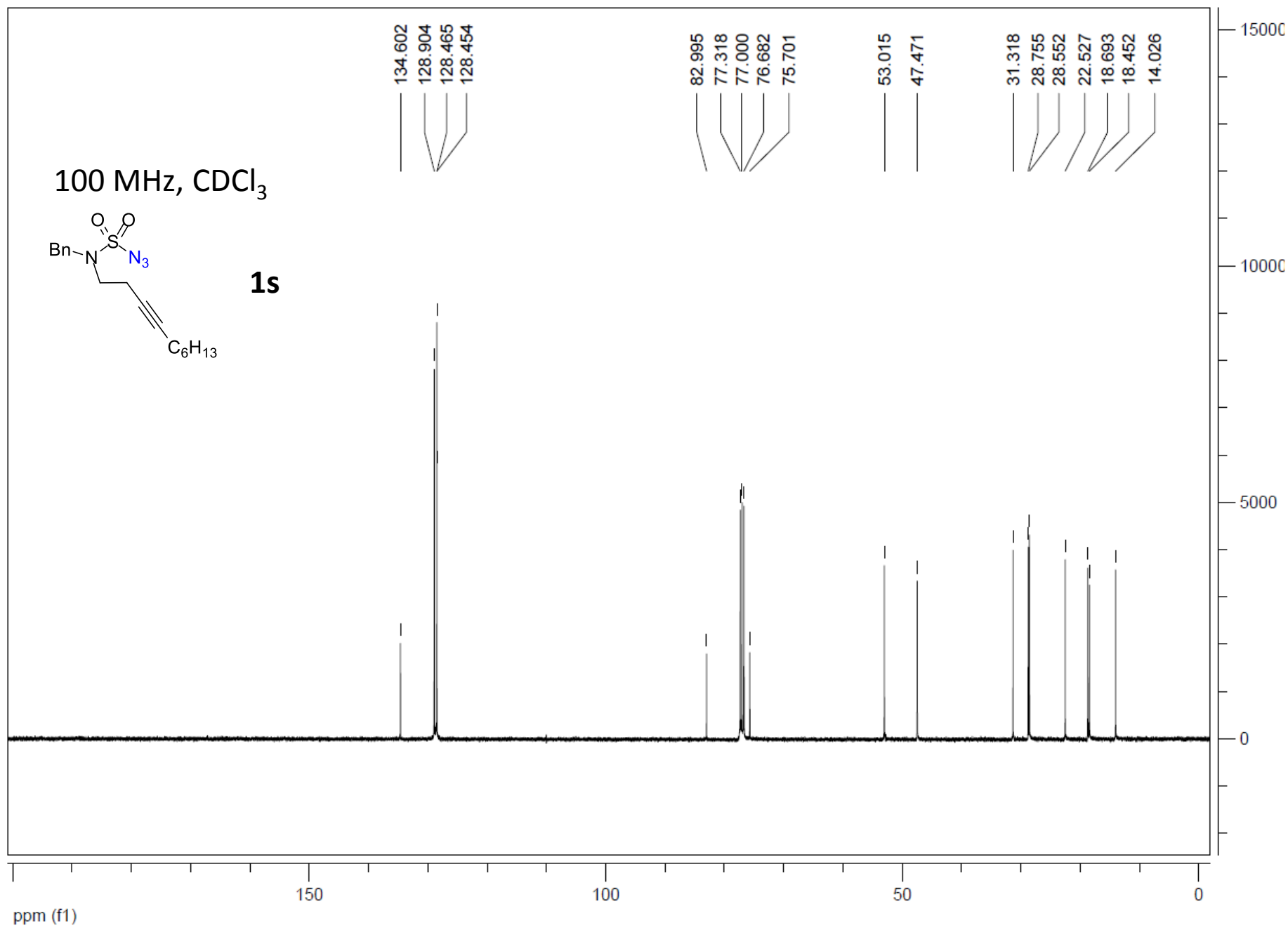
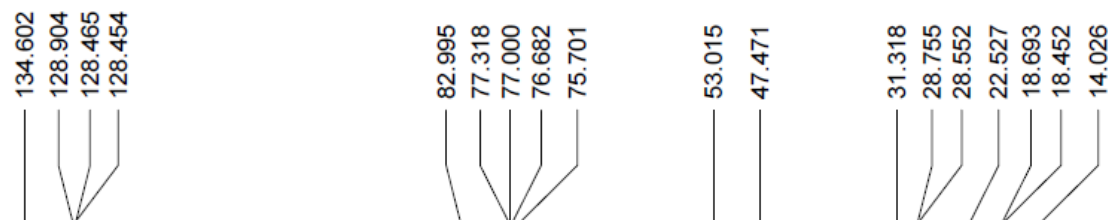
500 MHz, CDCl₃



100 MHz, CDCl₃



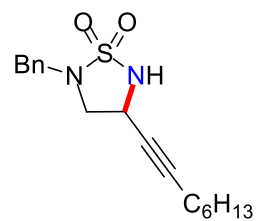
1s



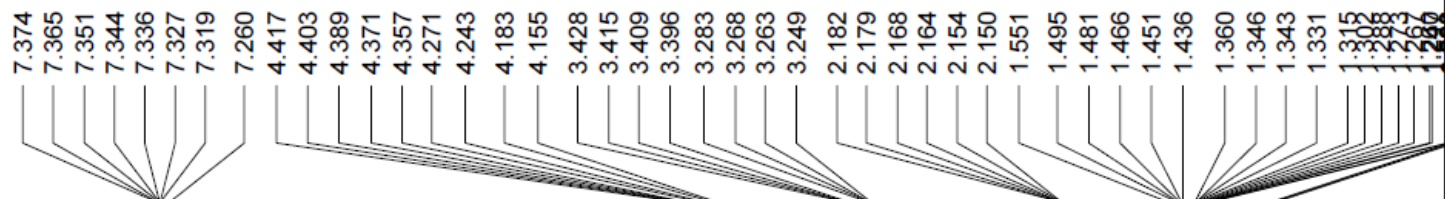
SII

80

500 MHz, CDCl₃



2s



4.42

1.75

1.97

1.00

1.94

1.95

6.01

2.74

5.0

500

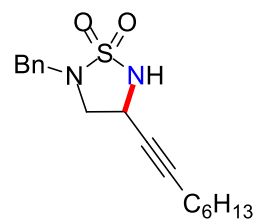
0

ppm (f1)

SII

81

125 MHz, CDCl₃



2s

134.902
128.778
128.563
128.215

87.773
77.254
77.000
76.746
75.224

54.322
50.442
44.241

31.213
28.451
28.164
22.465
18.557
14.013

400

300

200

100

0

150

100

50

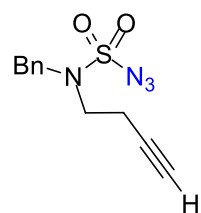
0

ppm (f1)

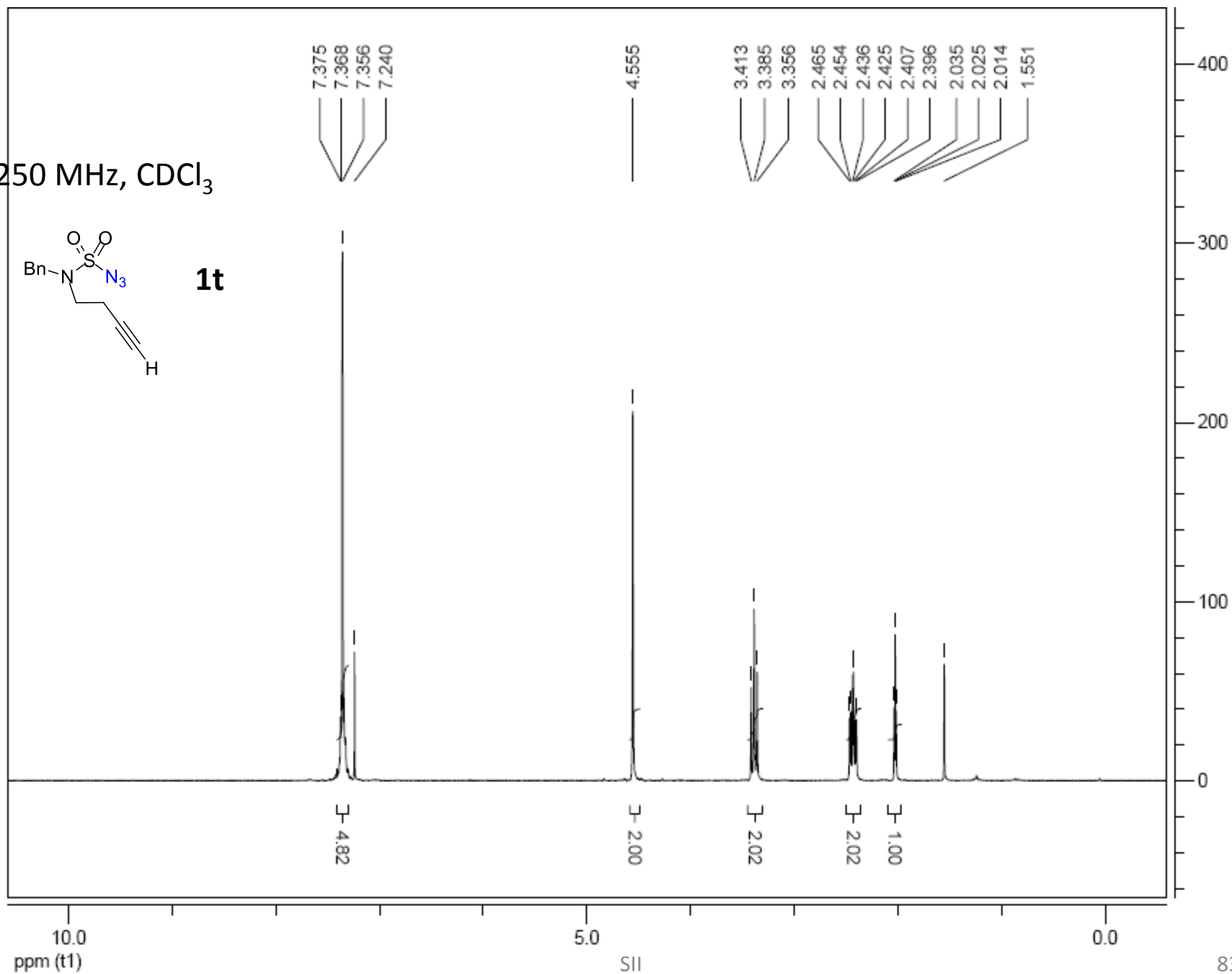
SII

82

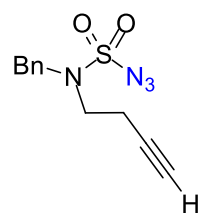
250 MHz, CDCl₃



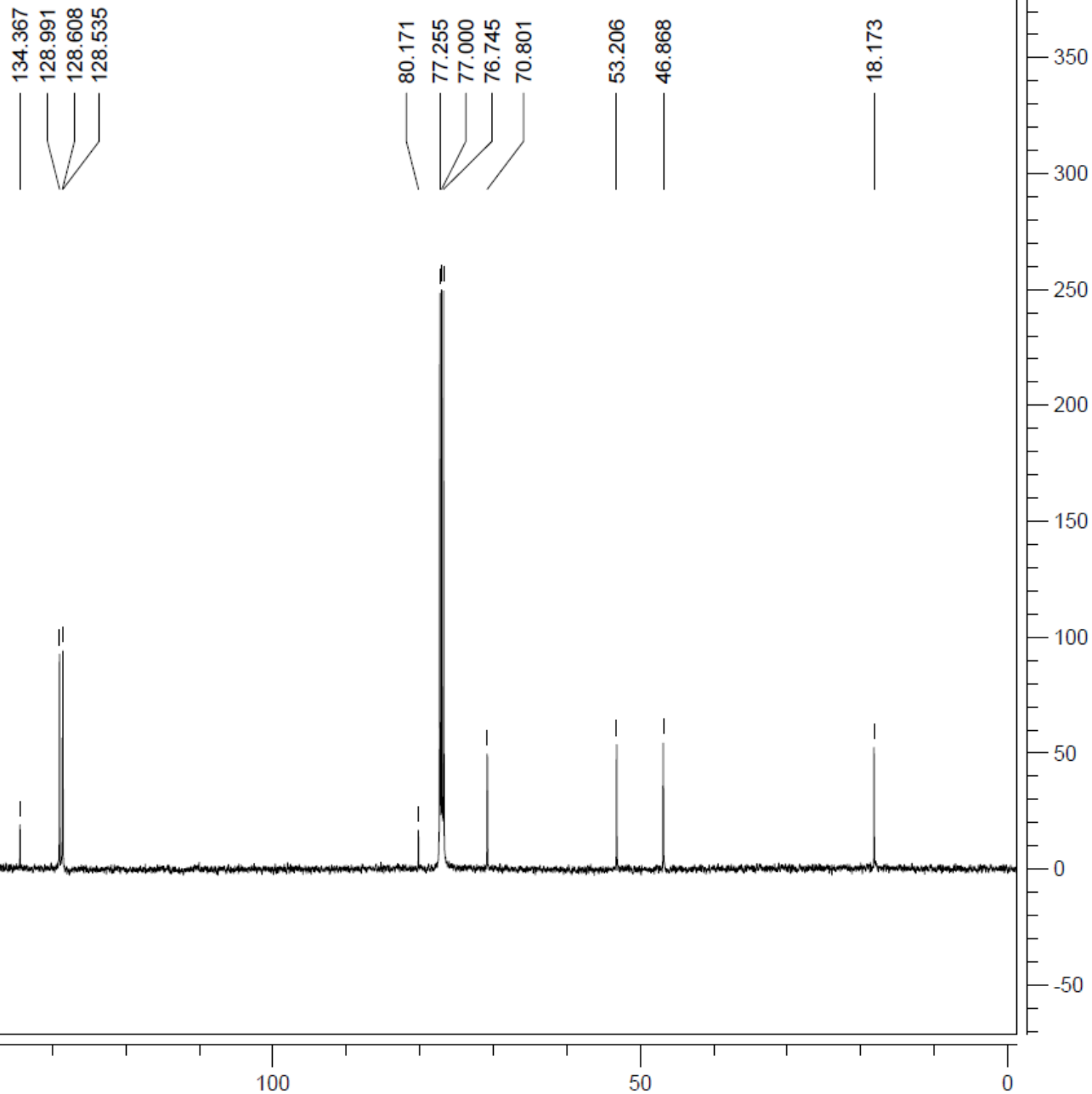
1t

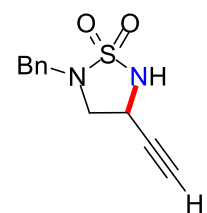


125 MHz, CDCl₃



1t





500 MHz, CDCl₃

2t

7.379
7.370
7.352
7.345
7.334
7.325
7.260

4.583
4.571
4.446
4.442
4.433
4.428
4.419
4.415
4.405
4.401
4.281
4.254
4.186
4.159
3.500
3.486
3.481
3.467
3.346
3.332
3.327
3.313
2.502
2.497

4.53

0.88
0.90
1.01
1.01

1.00
1.01

0.80

5.0

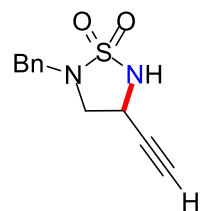
0.0

ppm (f1)

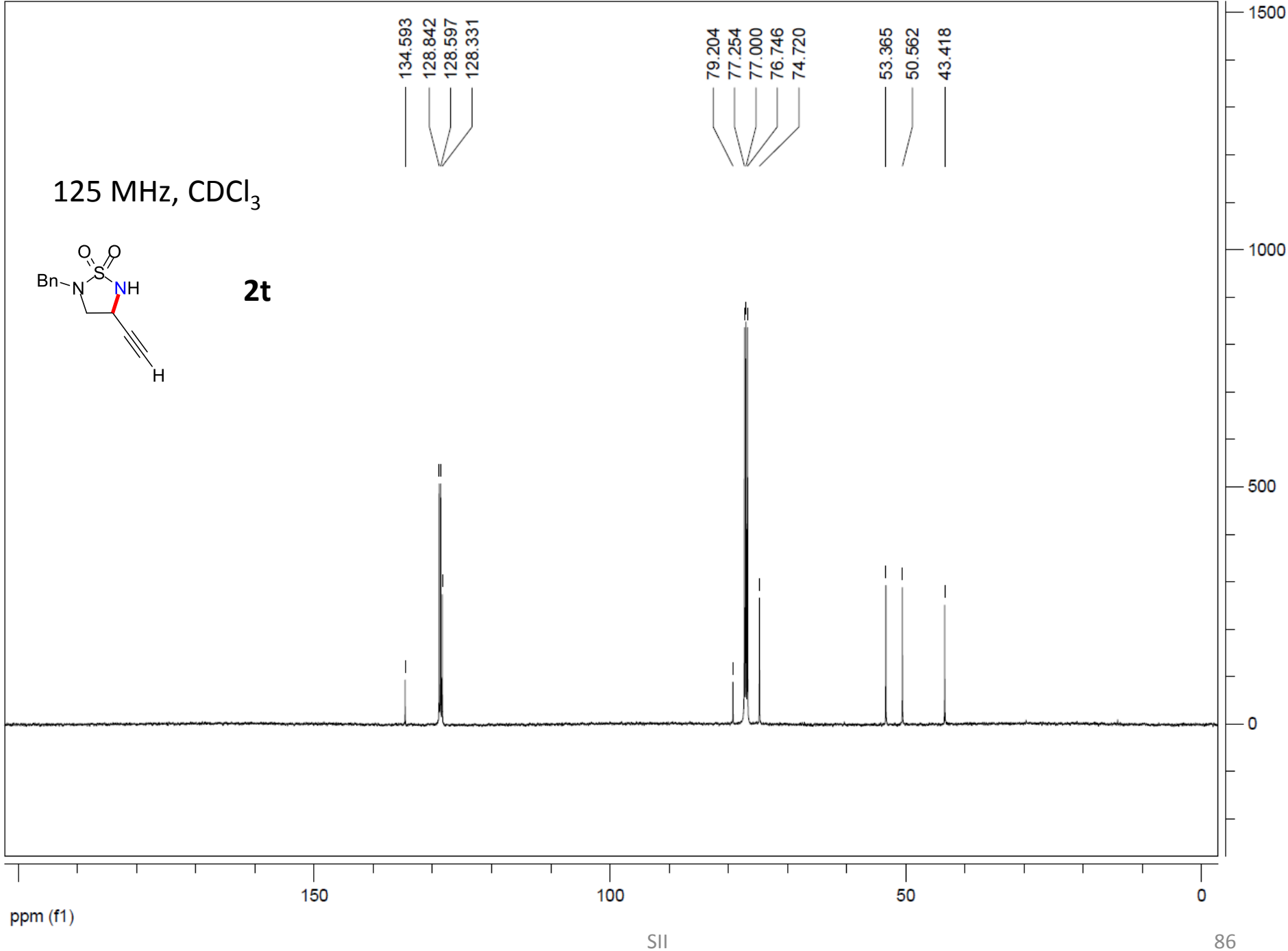
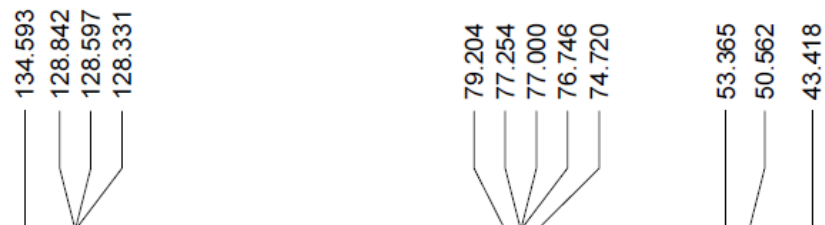
SII

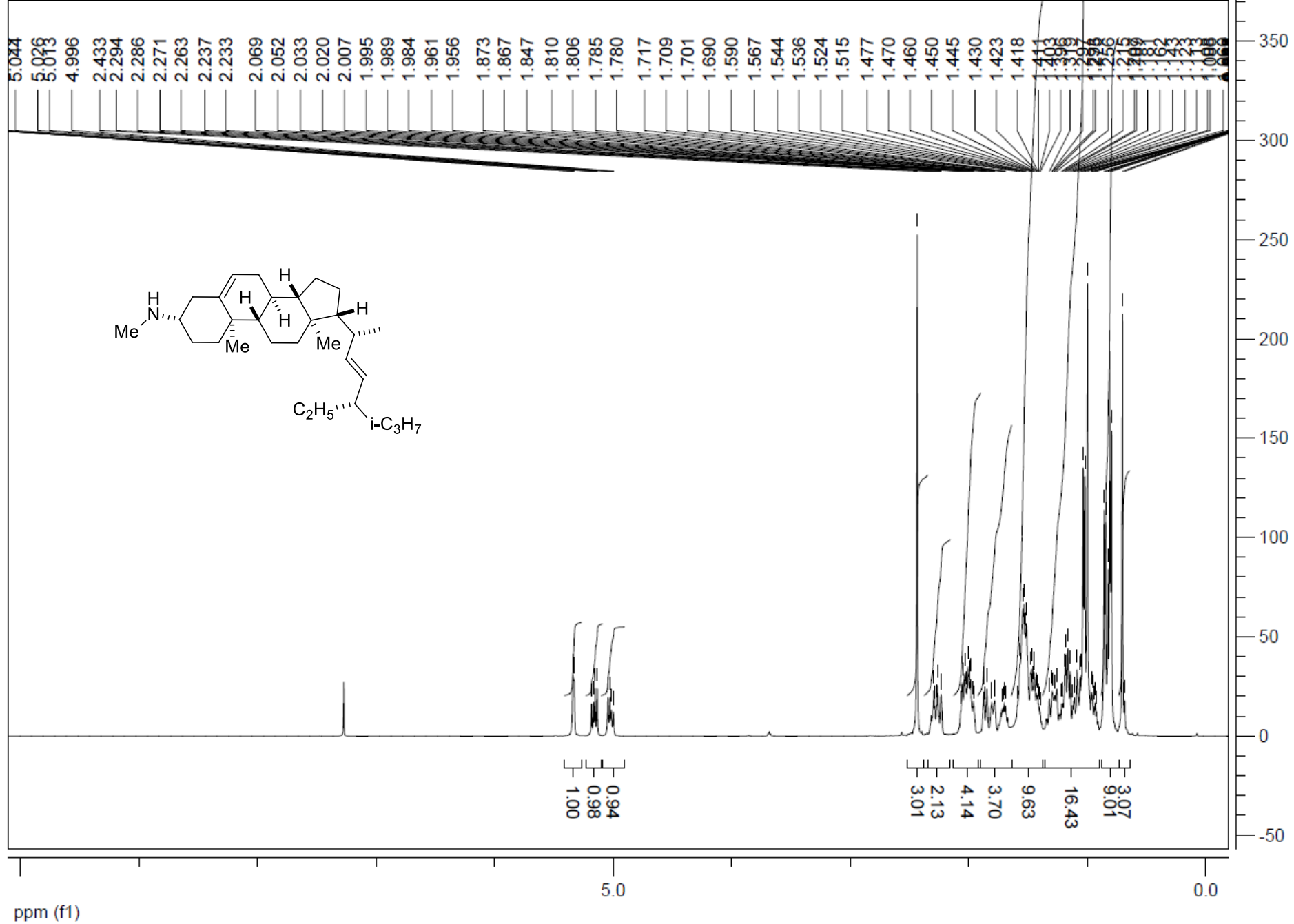
85

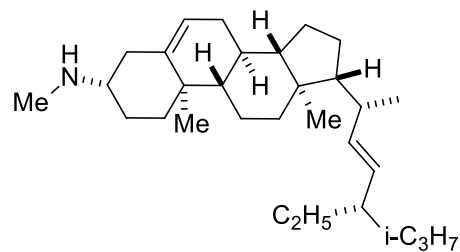
125 MHz, CDCl₃



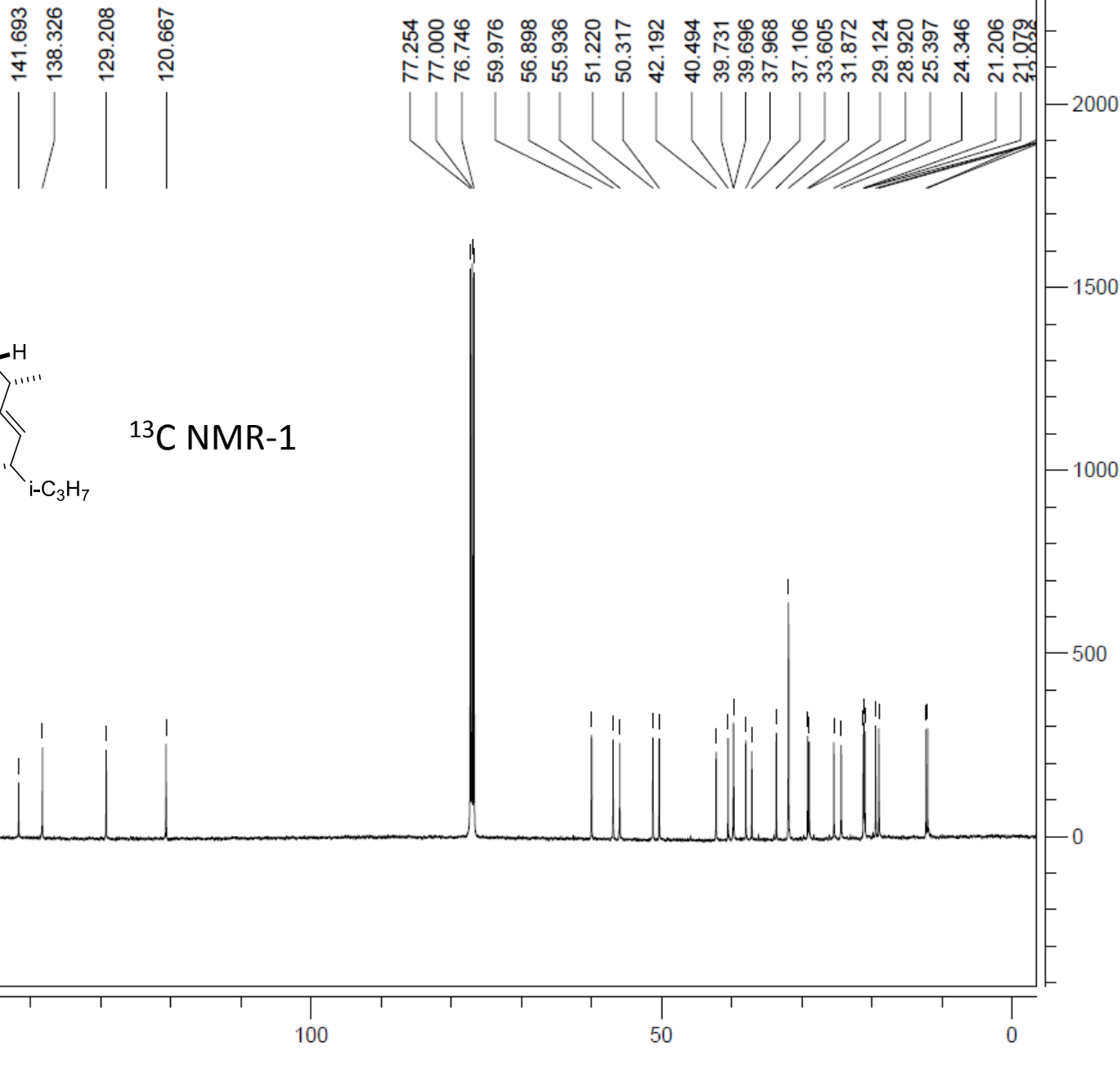
2t

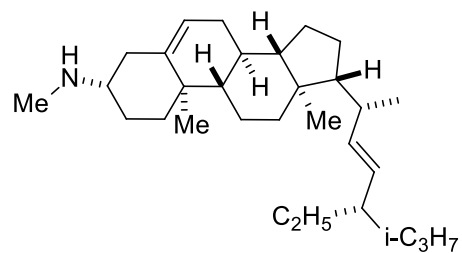




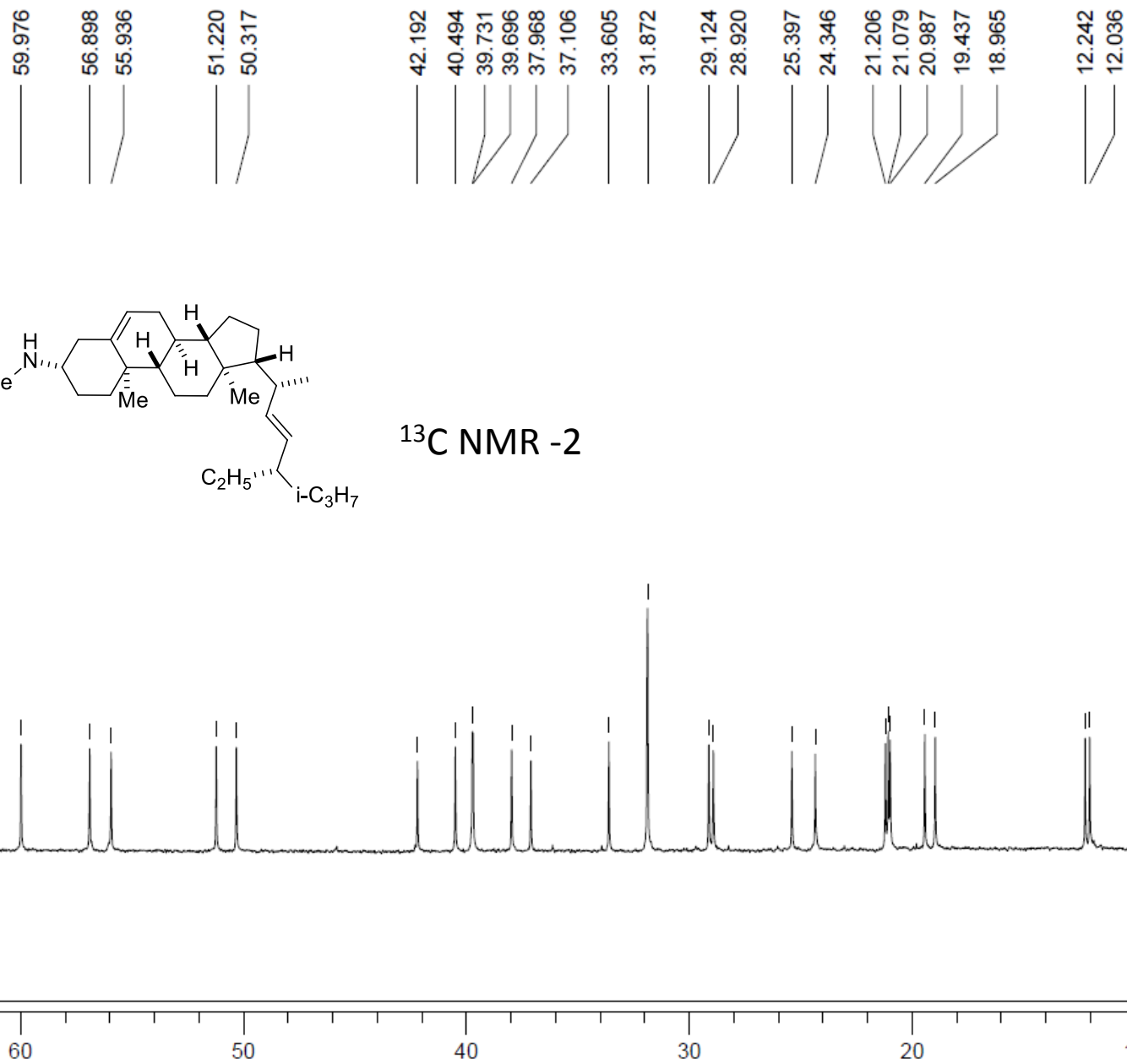


¹³C NMR-1

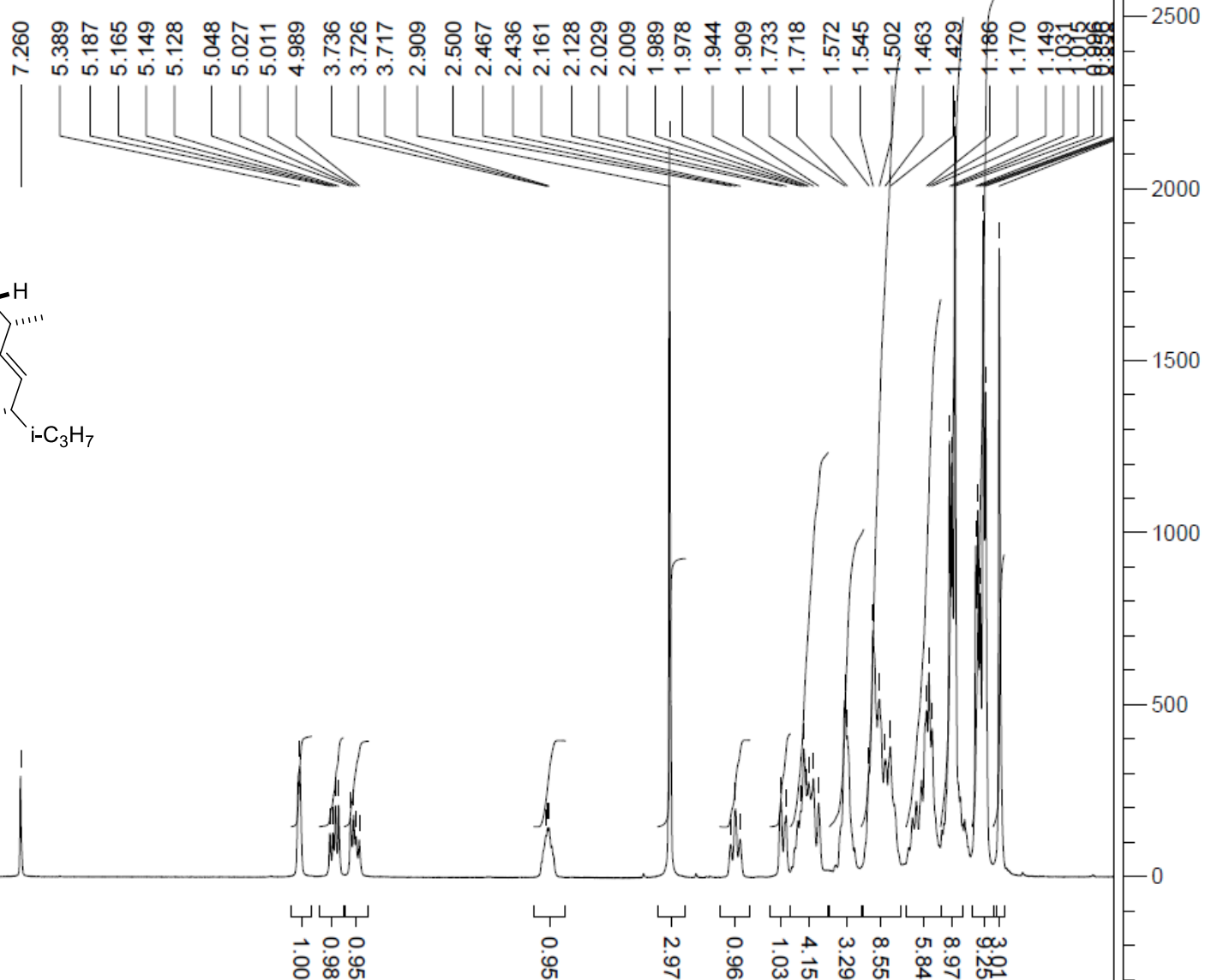
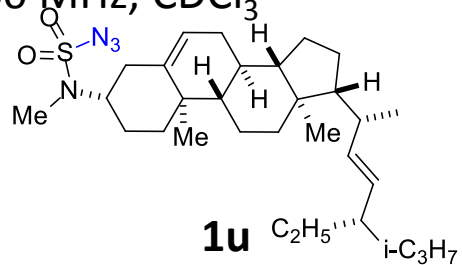




¹³C NMR -2



400 MHz, CDCl₃



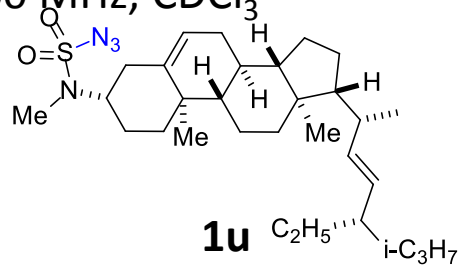
5.0

ppm (f1)

SII

90

100 MHz, CDCl₃



¹³C NMR -1

139.392
138.243
129.311
122.935

77.317
77.000
76.682
59.498
56.716
55.907
51.225
50.045
42.189
40.477
39.558
37.774
36.500
35.535
31.876
31.832
31.790
30.024
28.885
25.610
25.402
24.332
21.220
21.000

10000

5000

0

ppm (f1)

150

100

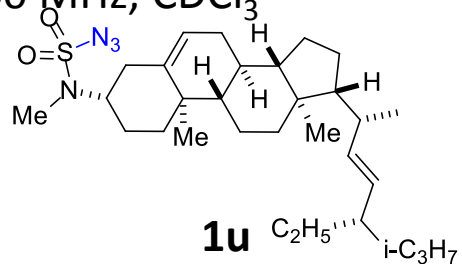
50

0

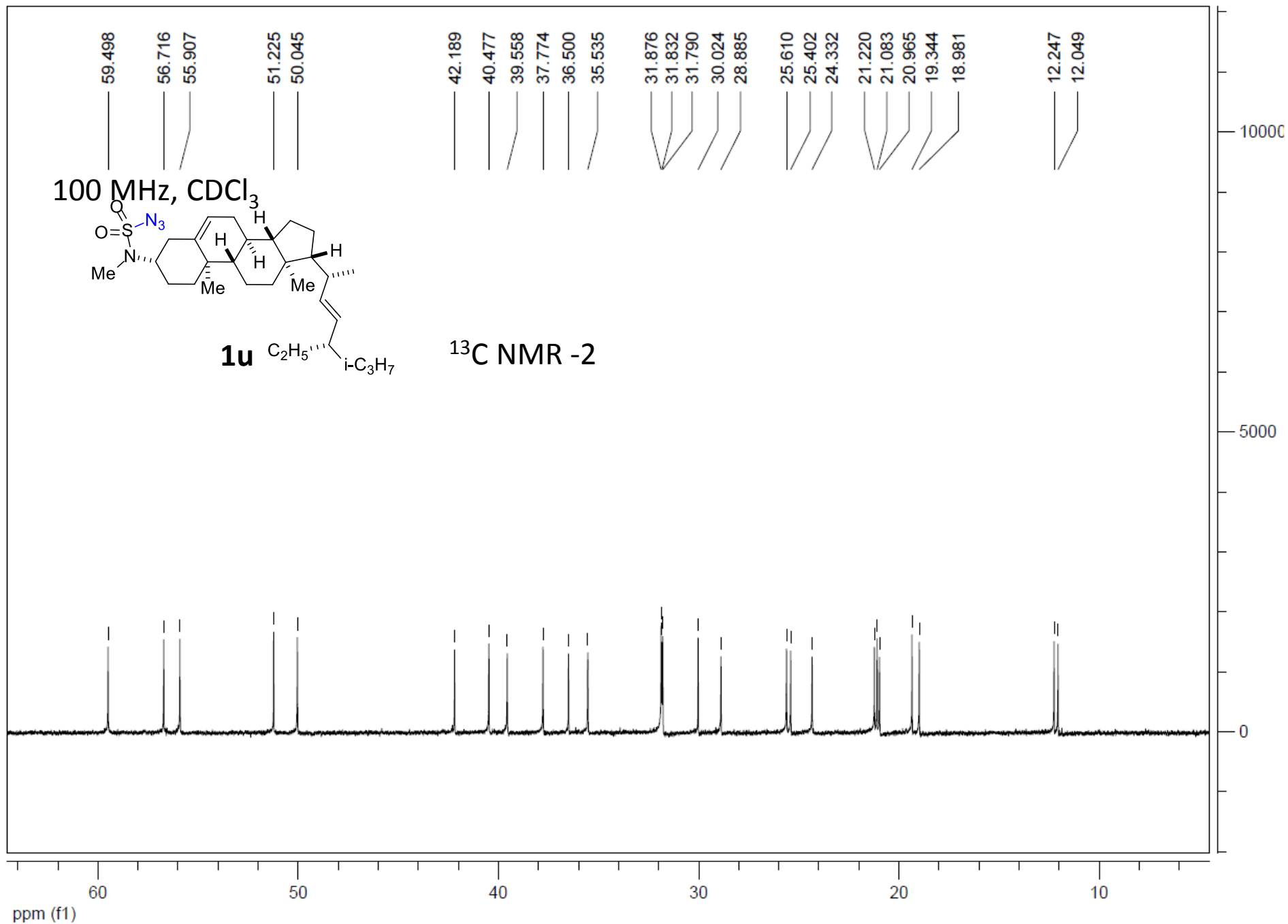
SII

91

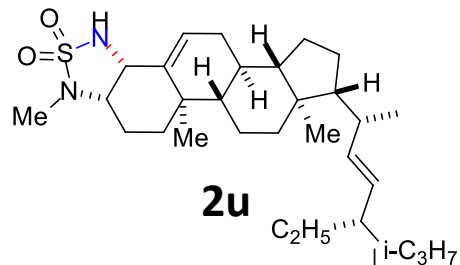
100 MHz, CDCl₃



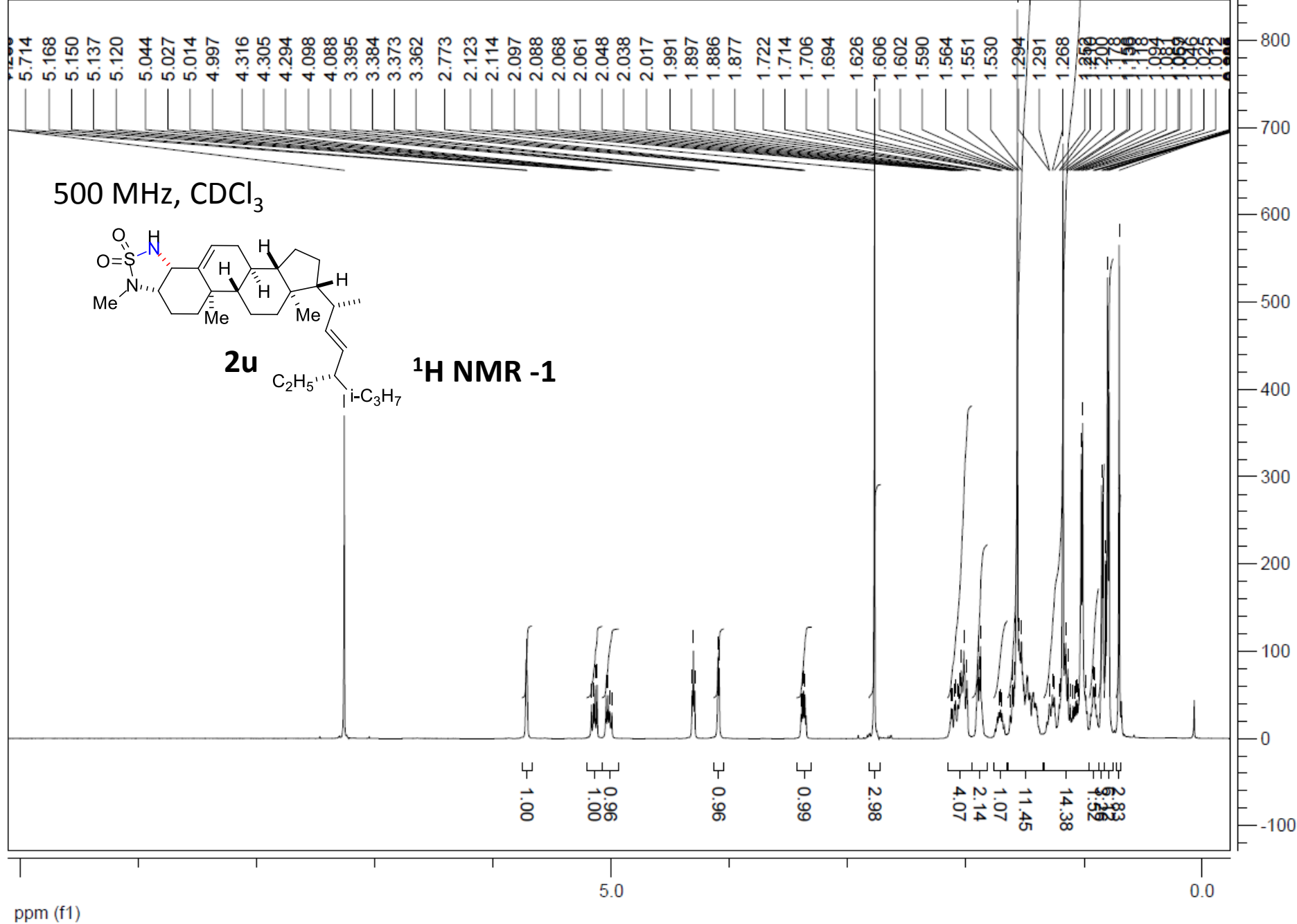
¹³C NMR -2



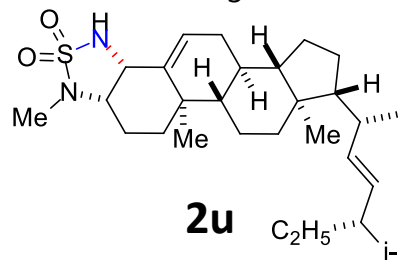
500 MHz, CDCl₃



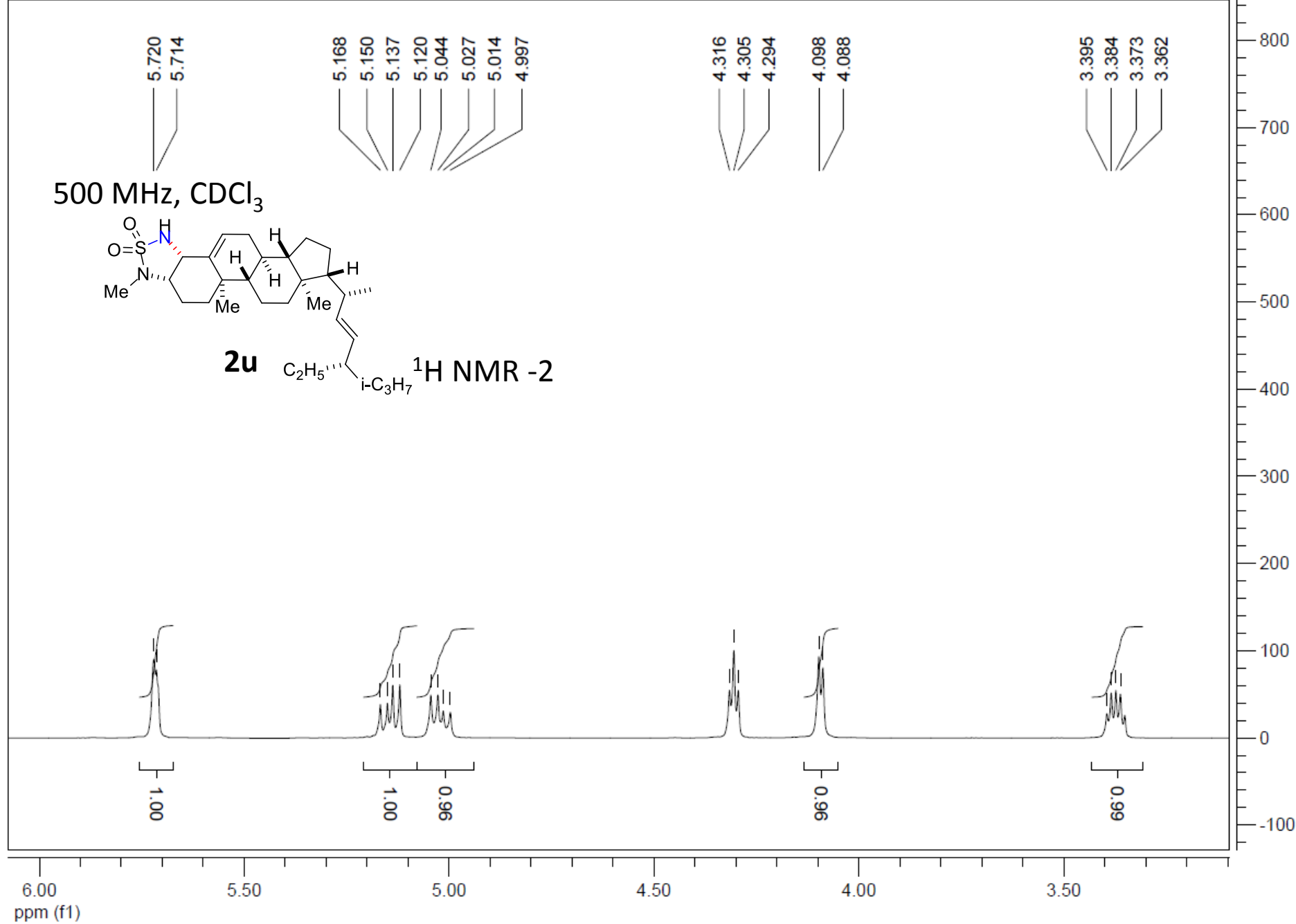
¹H NMR -1

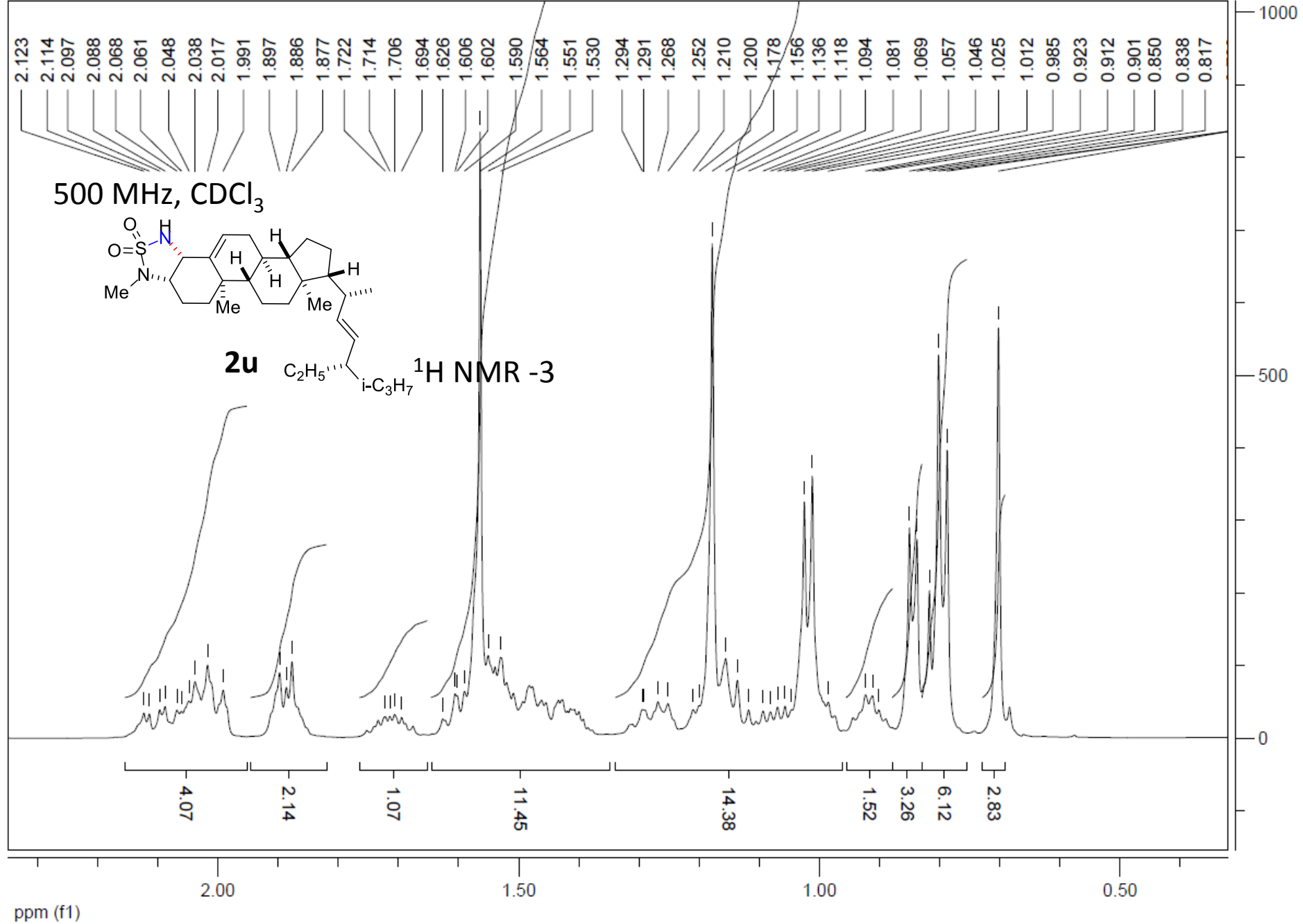


500 MHz, CDCl₃

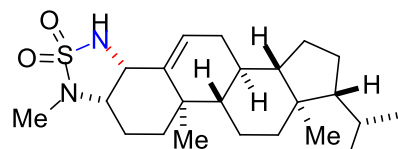


¹H NMR -2





100 MHz, CDCl₃



2u

C₂H₅

i-C₃H₇

¹³C NMR -1

138.145
137.252
131.634
129.410

77.318
77.000
76.682
62.107
59.794
56.956
55.876
51.236
49.515
42.188
40.440
39.457
36.391
34.254
32.070
31.861
31.537
29.149
28.862
25.390
24.227
21.202
21.082

7000

6000

5000

4000

3000

2000

1000

0

-1000

150

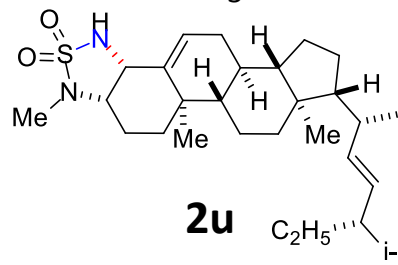
100

50

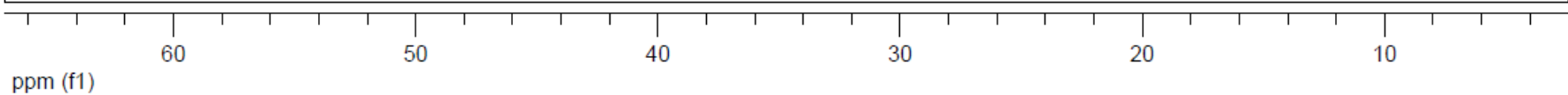
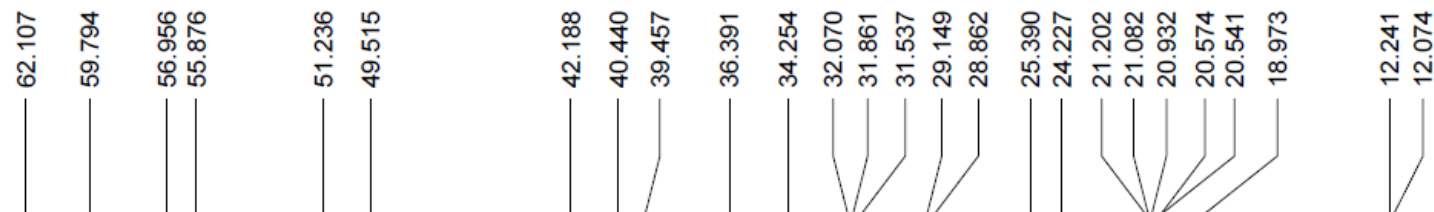
0

ppm (f1)

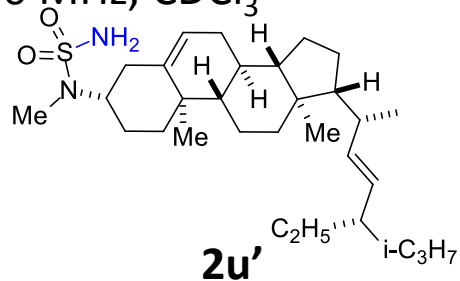
100 MHz, CDCl₃



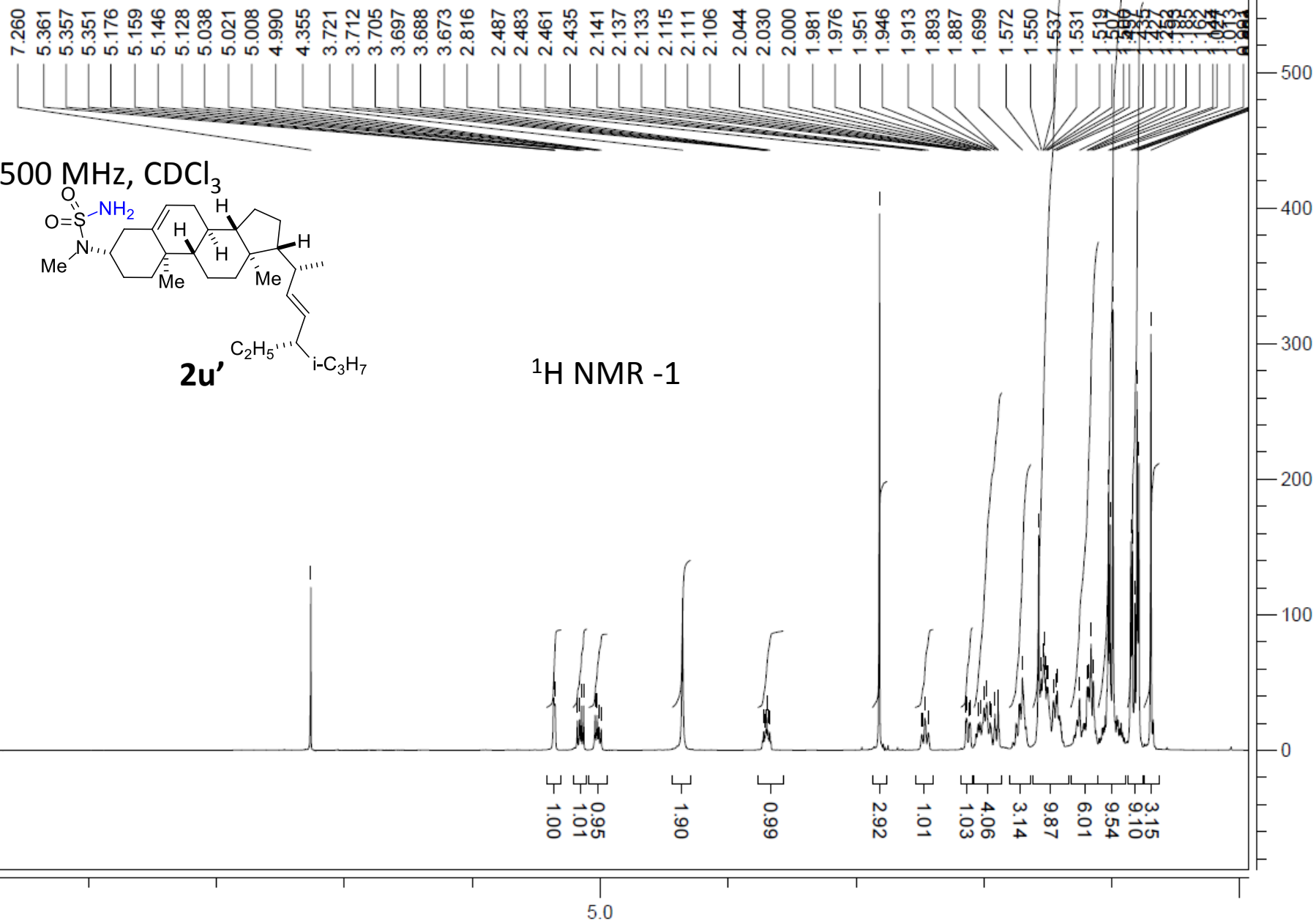
¹³C NMR -2



500 MHz, CDCl₃

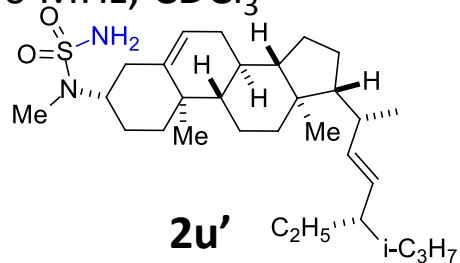


¹H NMR -1

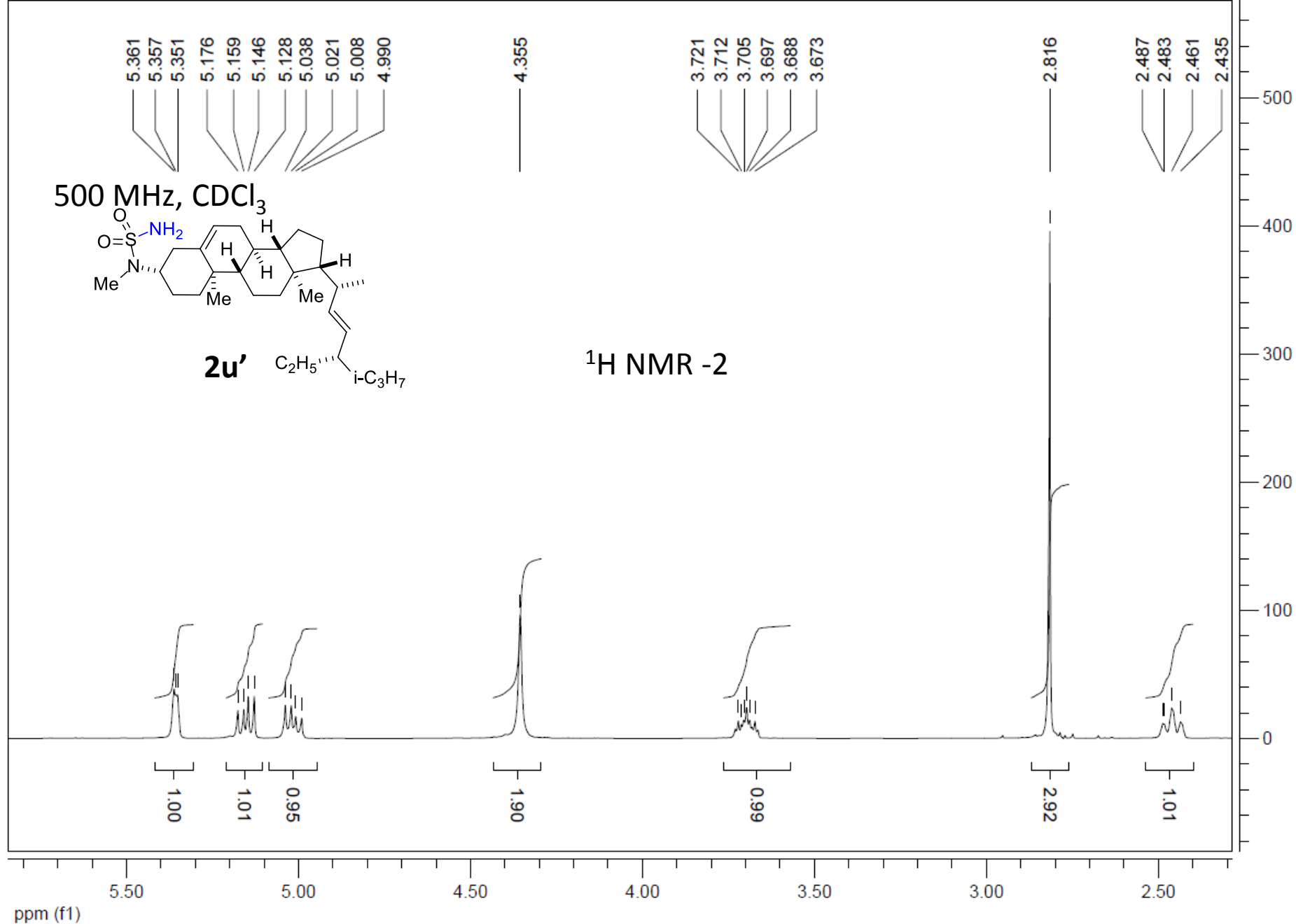


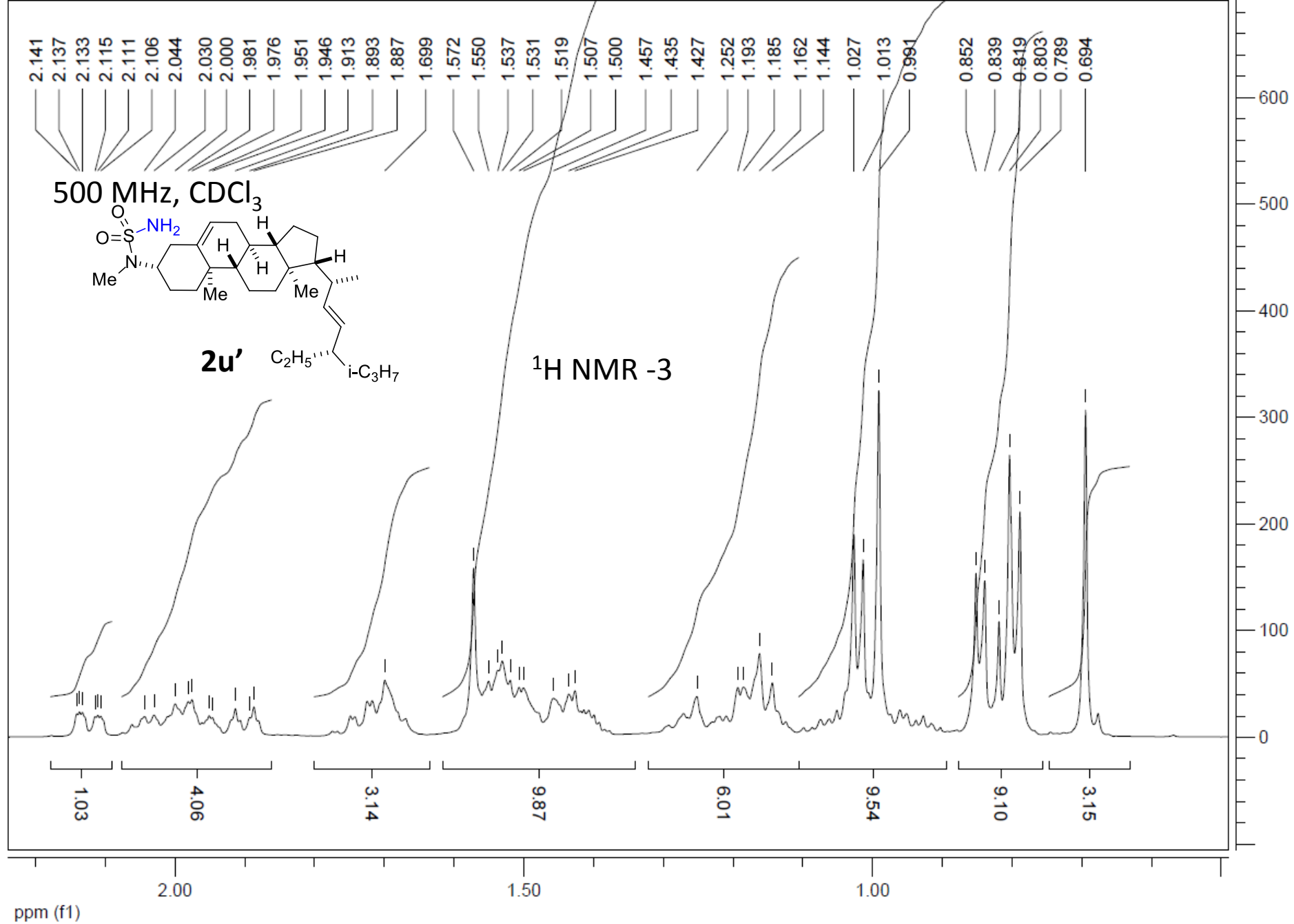
ppm (f1)

500 MHz, CDCl₃

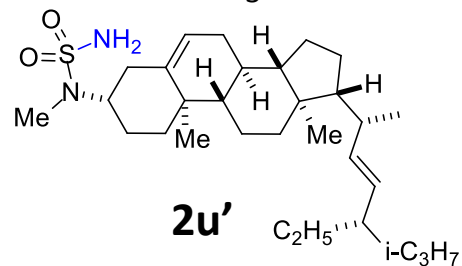


¹H NMR -2





100 MHz, CDCl₃



¹³C NMR -1

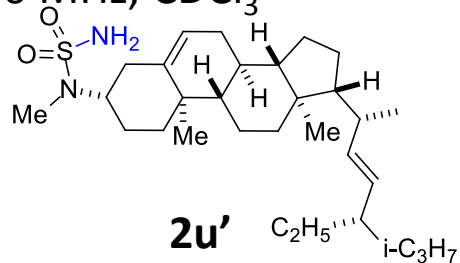


ppm (f1)

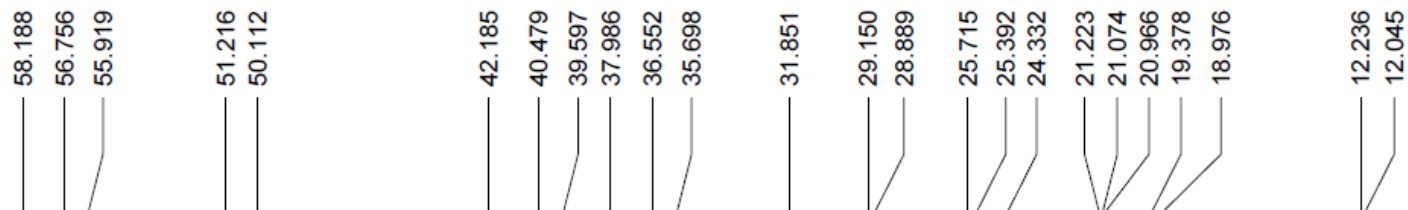
SII

101

500 MHz, CDCl₃



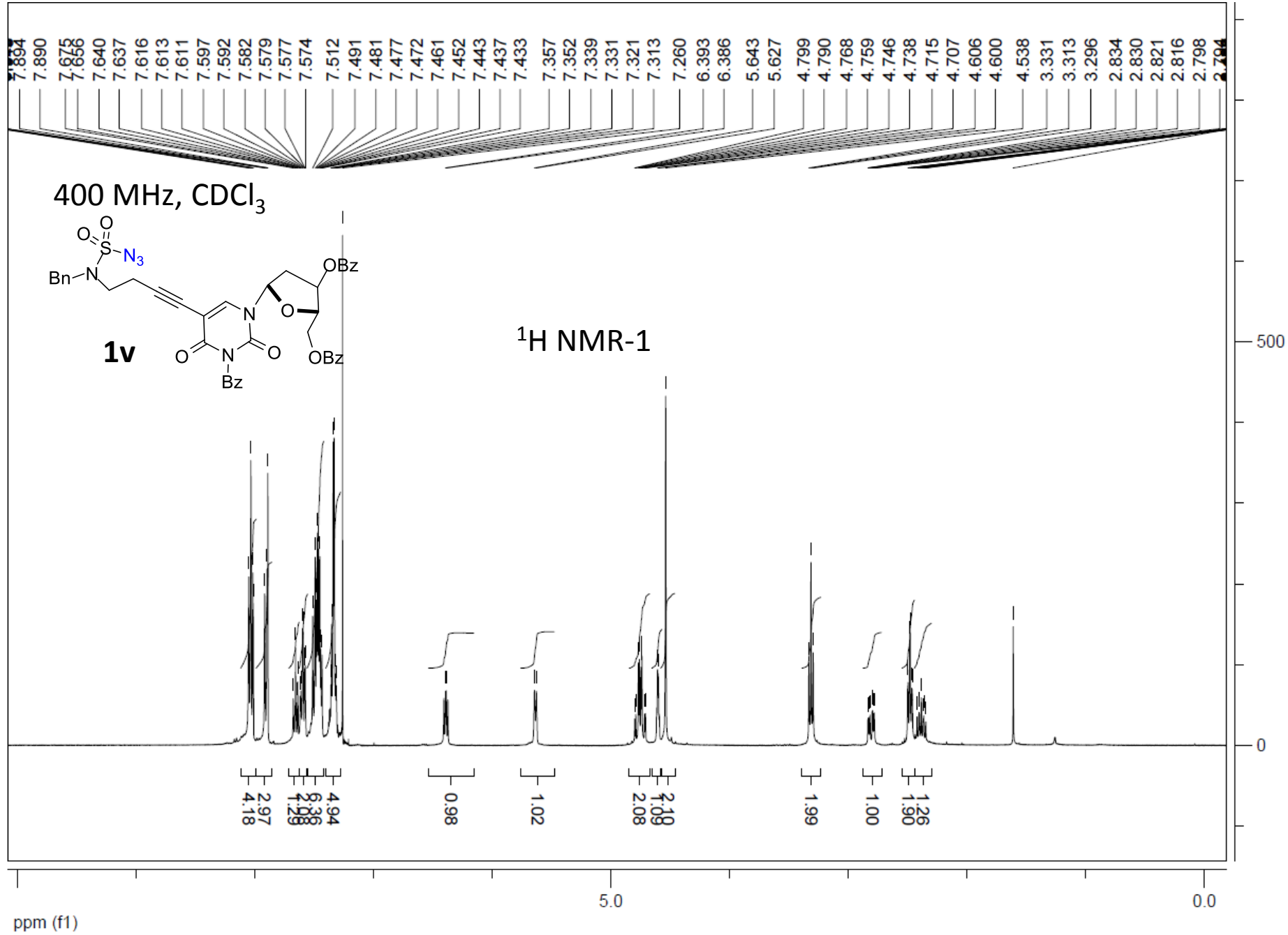
¹³C NMR -2

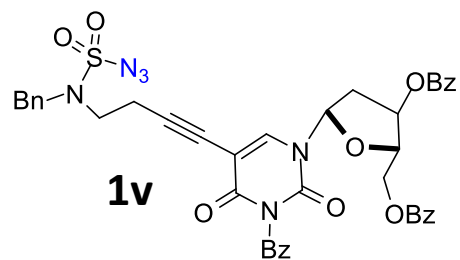


ppm (f1)

SII

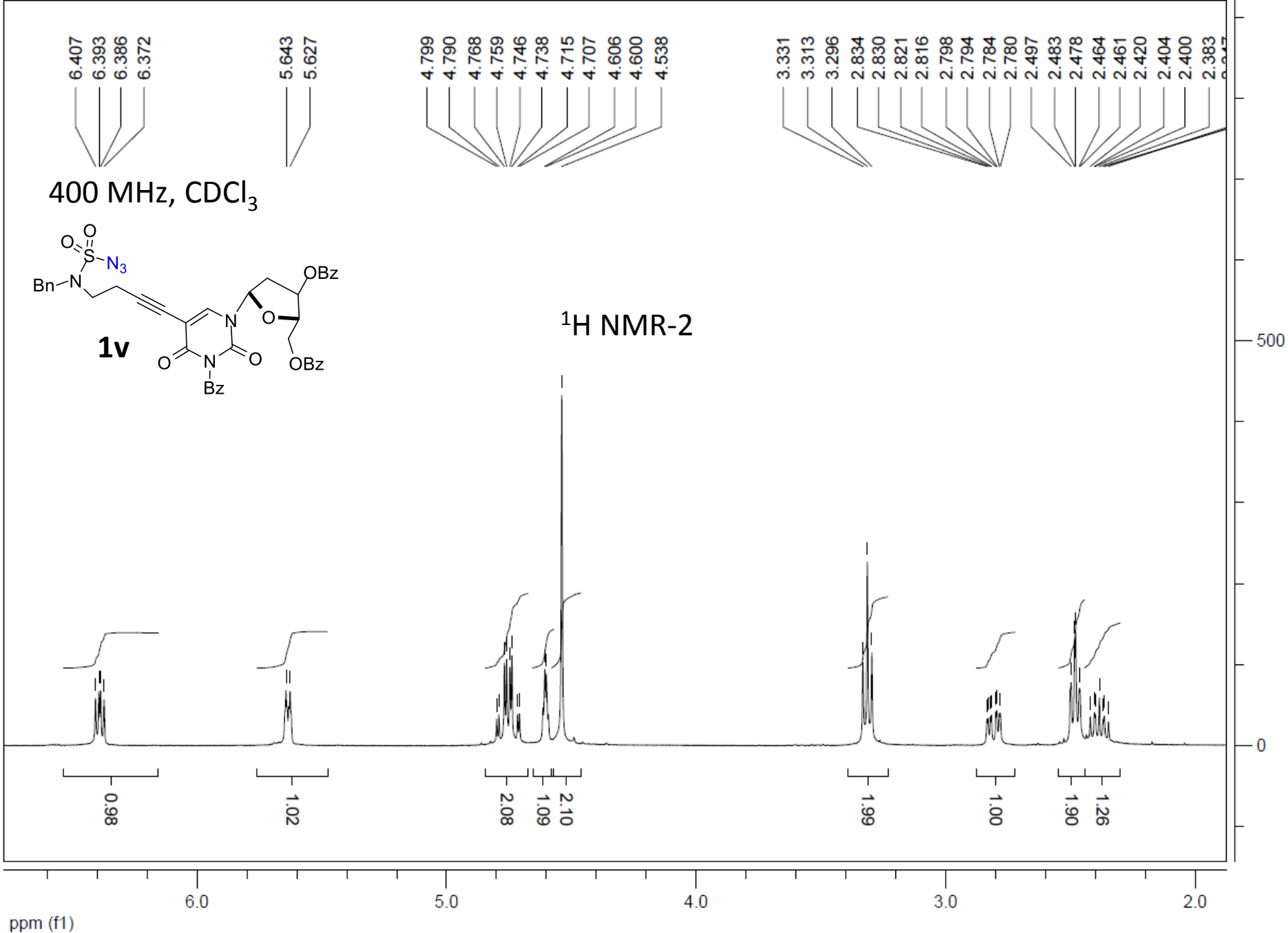
102





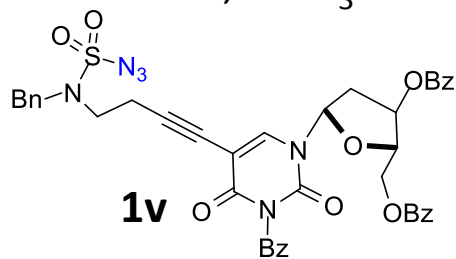
400 MHz, CDCl₃

¹H NMR-2



167.592
165.936
165.805
160.316
148.157
140.853
135.275
134.411
133.727
133.615
131.042
130.459
129.706
129.497
129.178
129.099
128.910
128.820
128.735
128.552
128.466
100.711
91.127
86.076
83.258
77.318
77.000
76.682
74.785
72.786
64.247
53.158
46.731
38.597
19.251

100 MHz, CDCl₃



5000

0

150

100

50

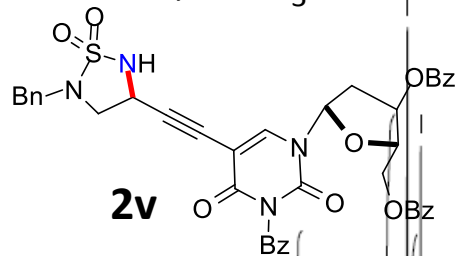
0

ppm (f1)

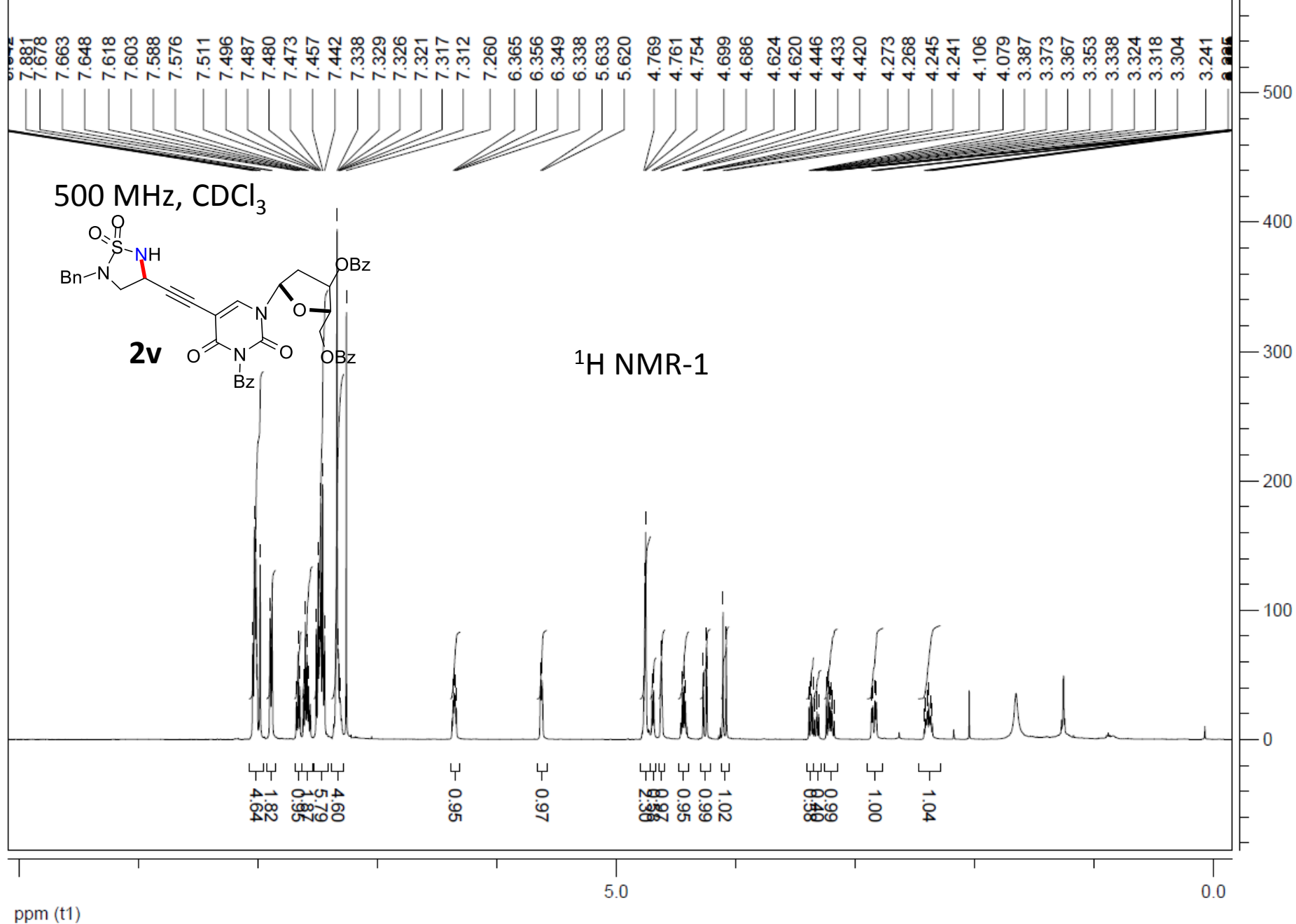
SII

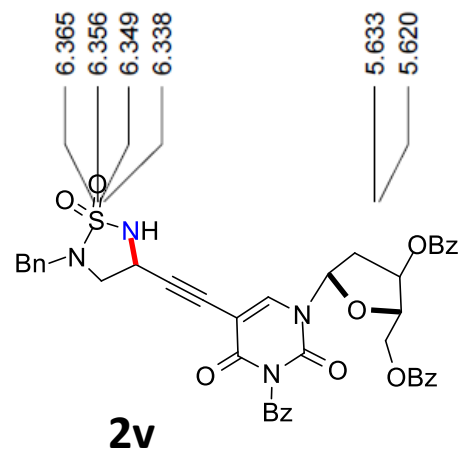
105

500 MHz, CDCl₃

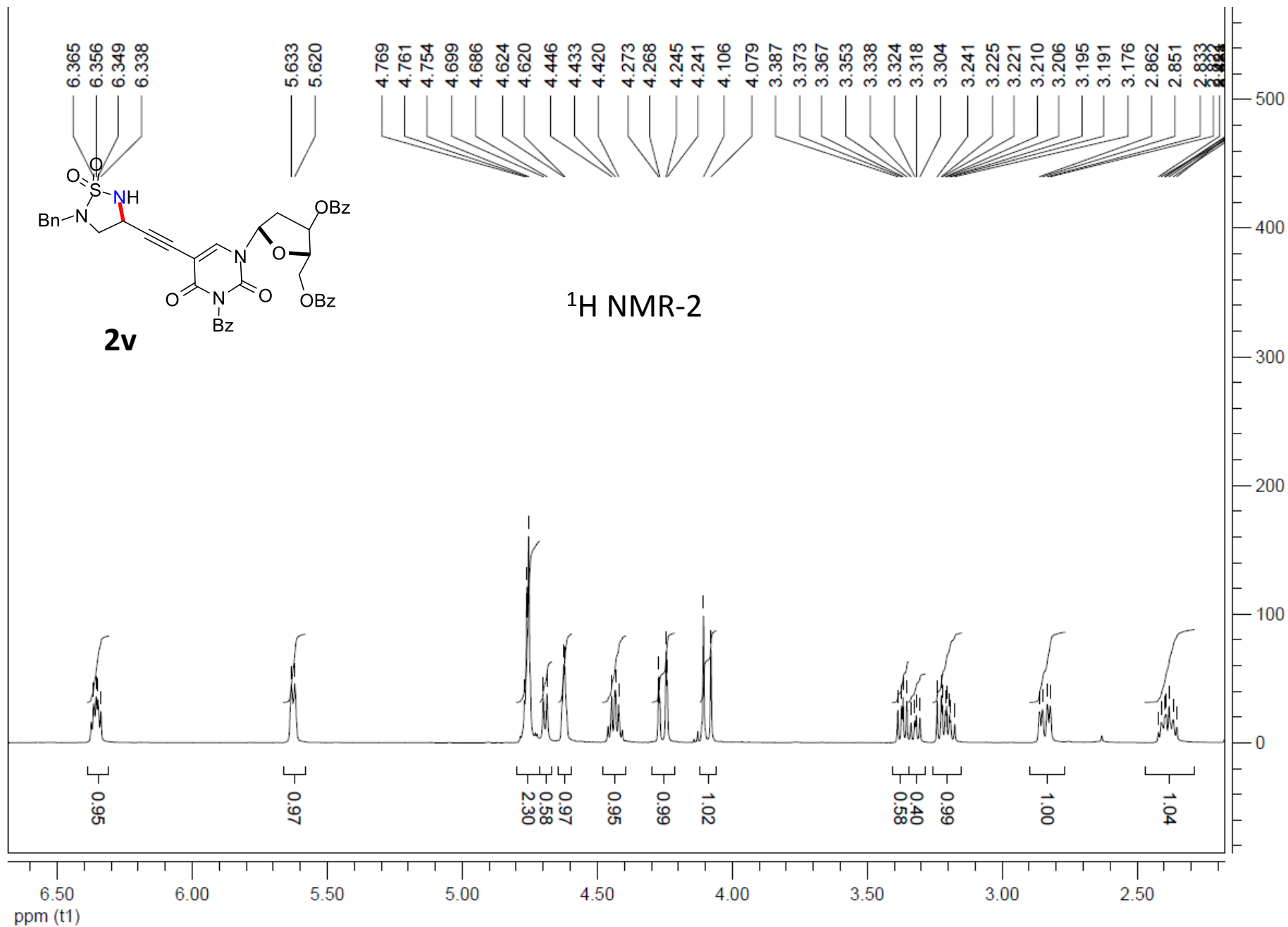


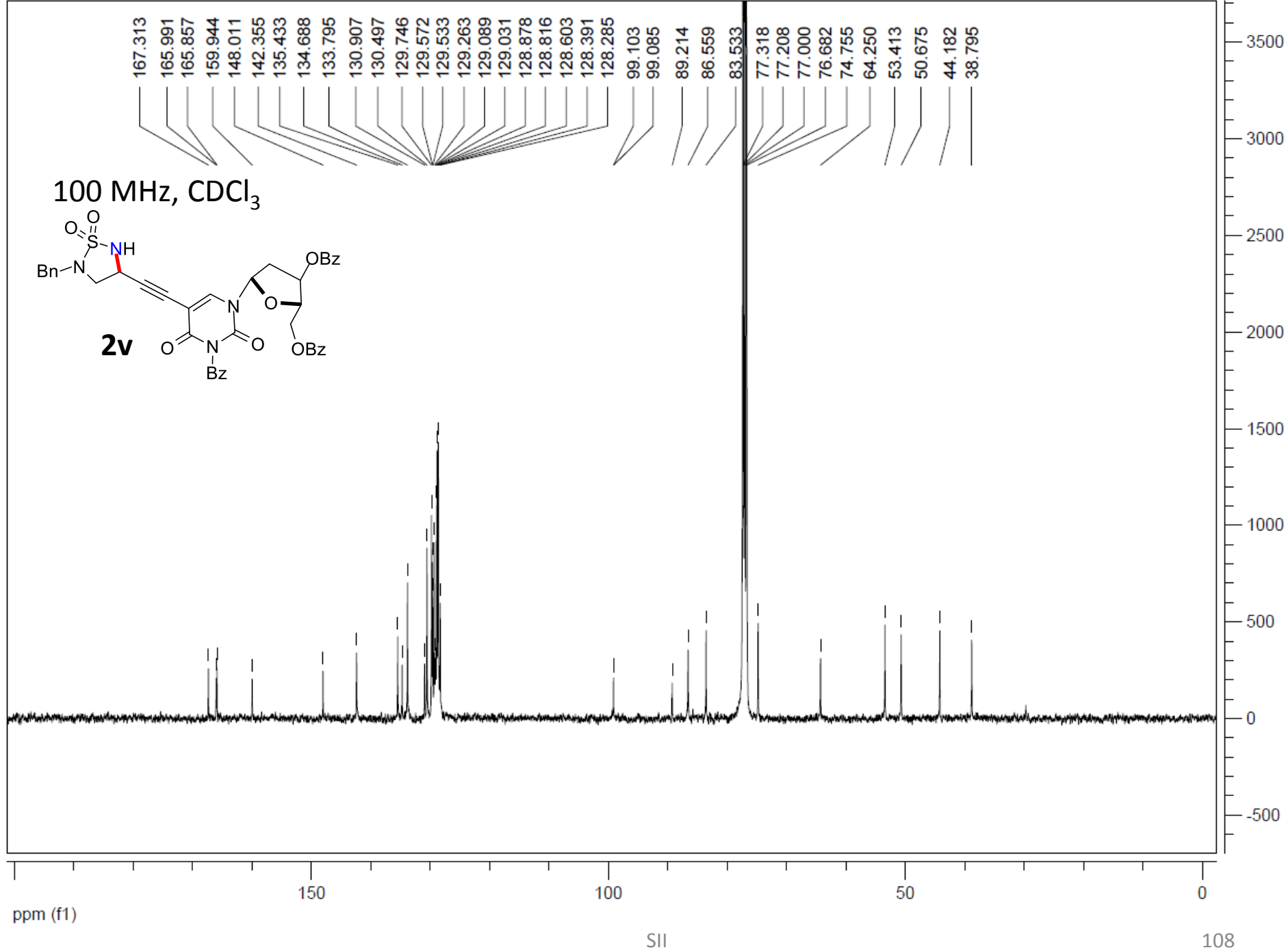
¹H NMR-1



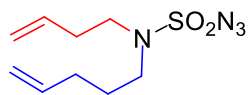


^1H NMR-2

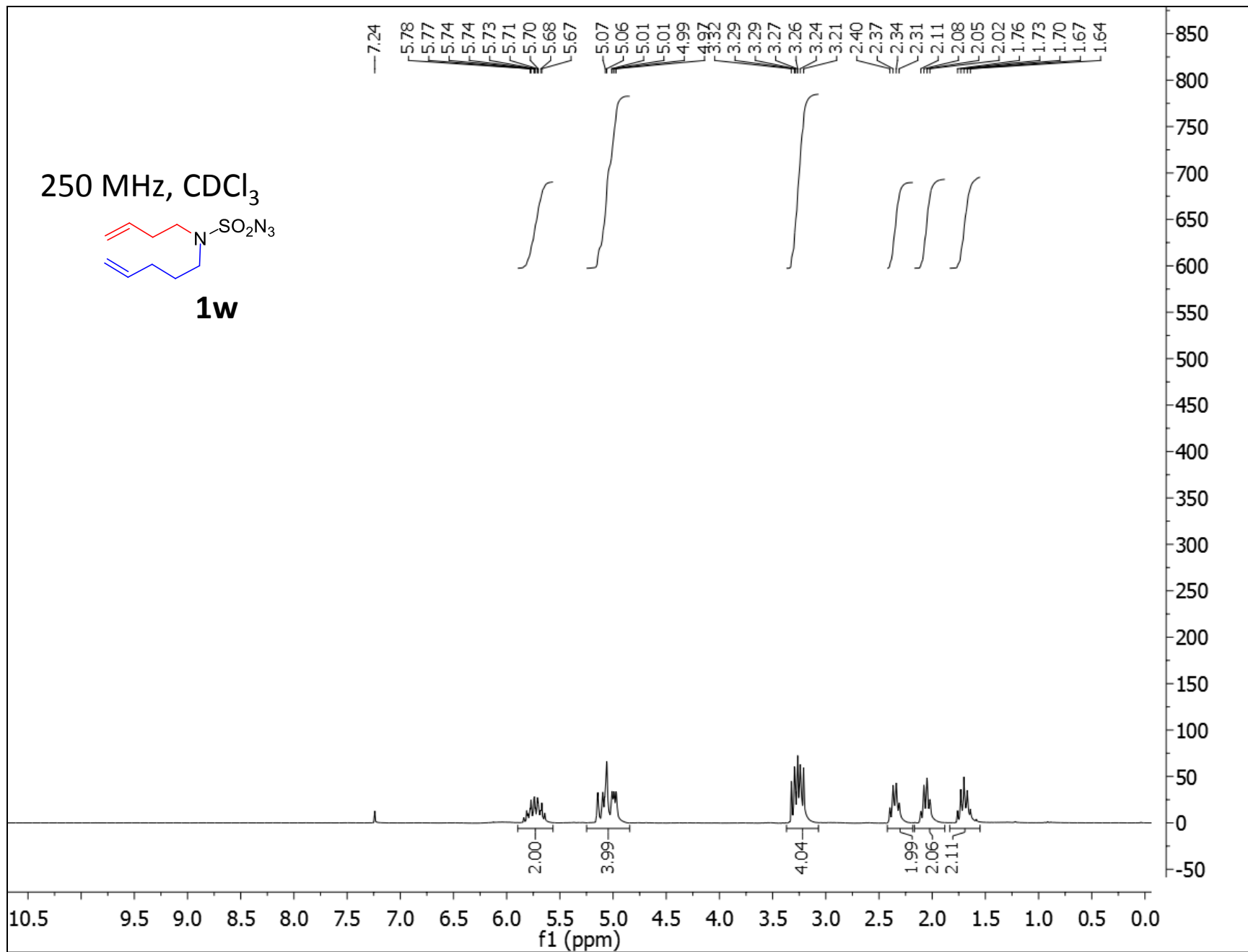




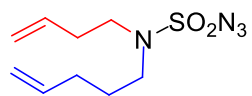
250 MHz, CDCl₃



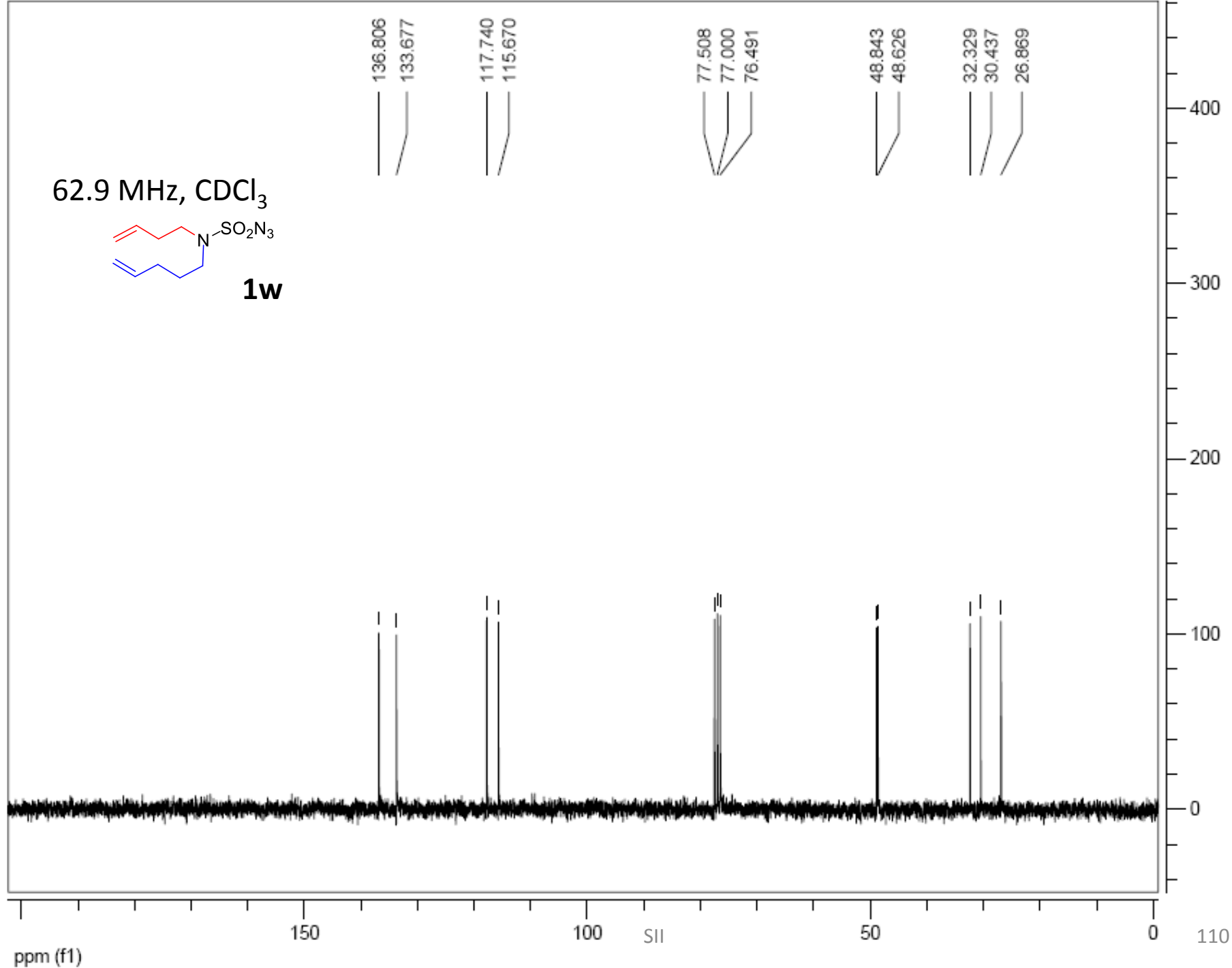
1w

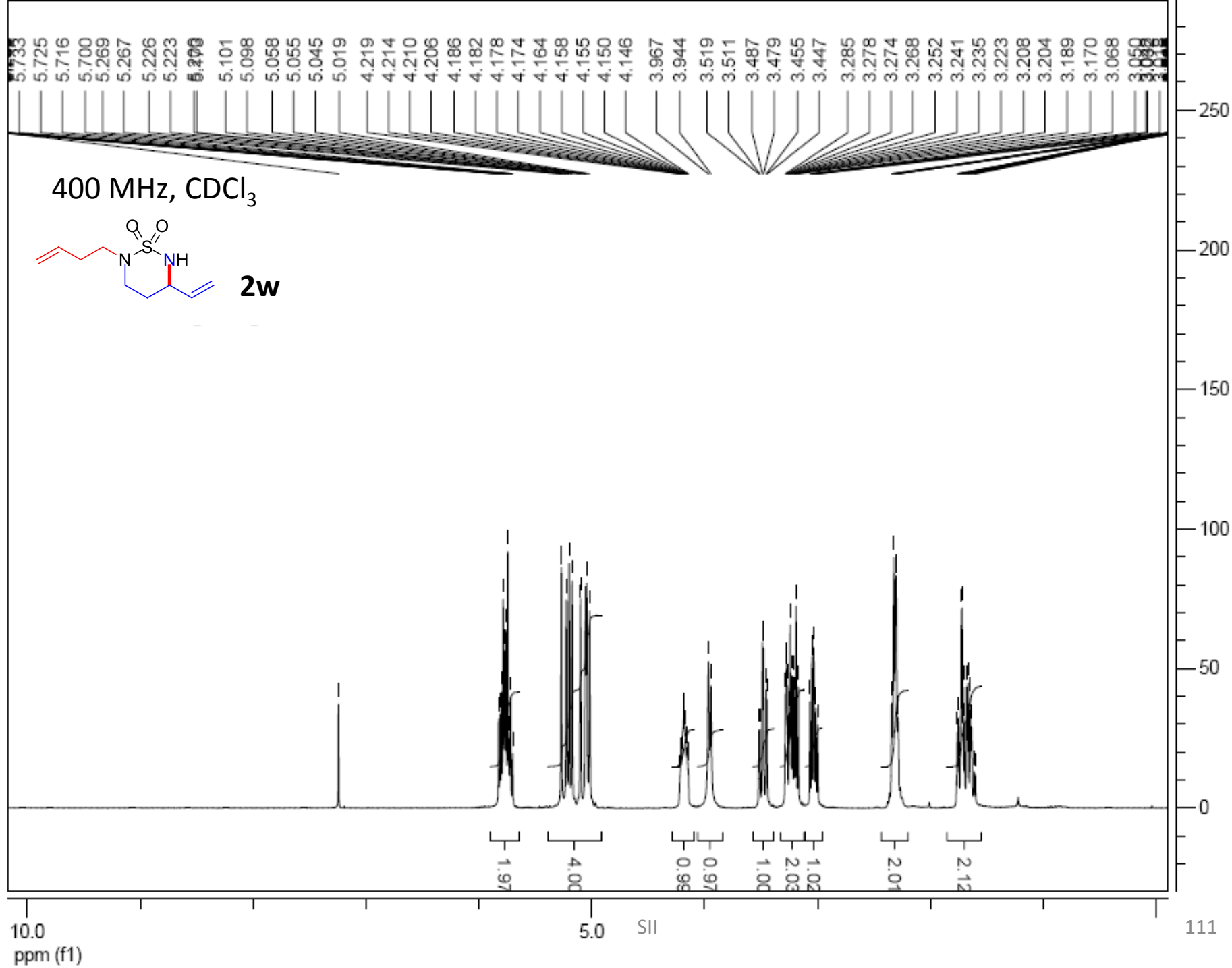


62.9 MHz, CDCl₃

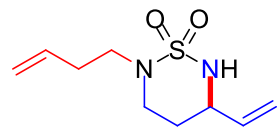


1w



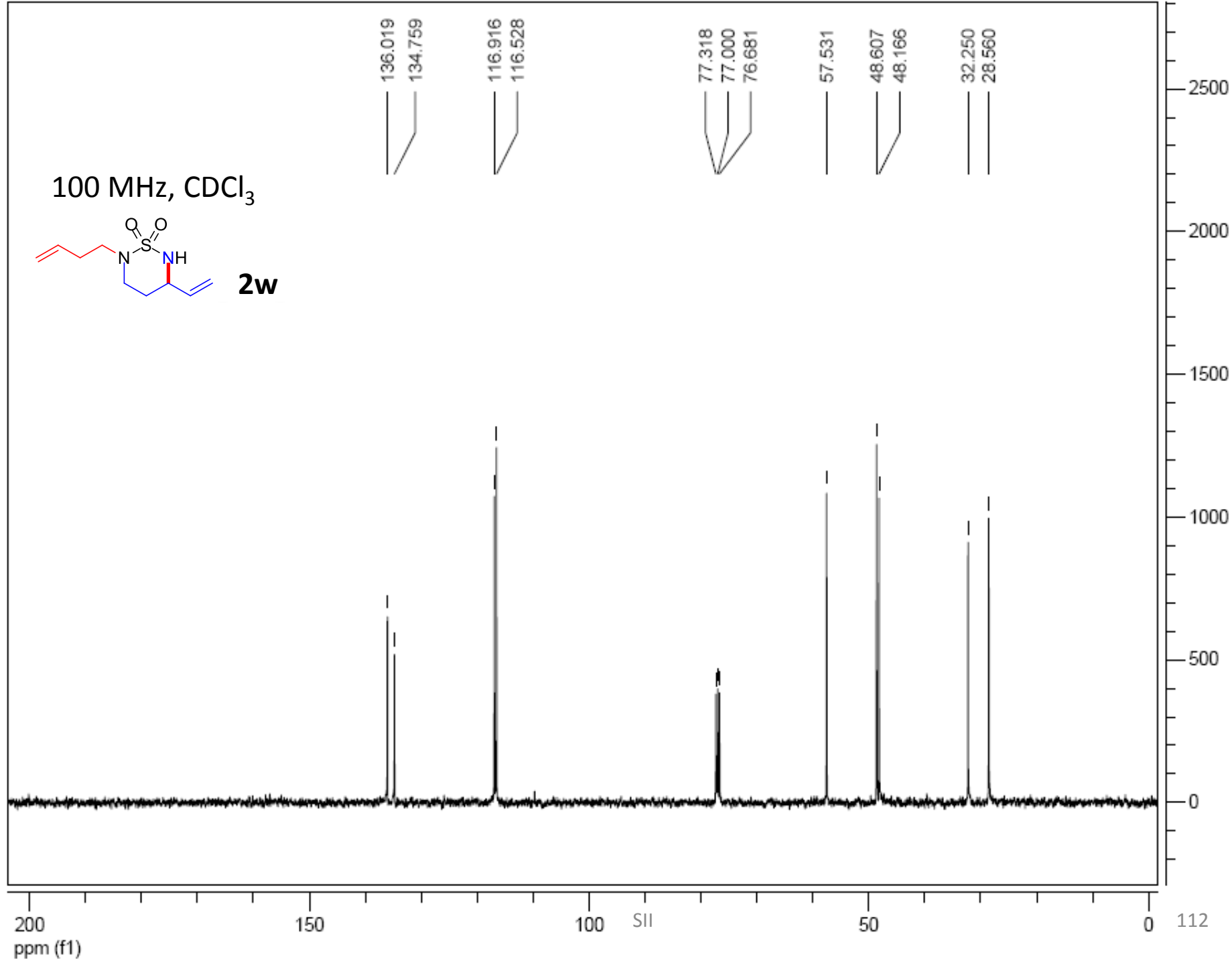


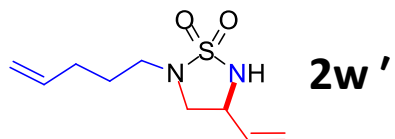
100 MHz, CDCl₃



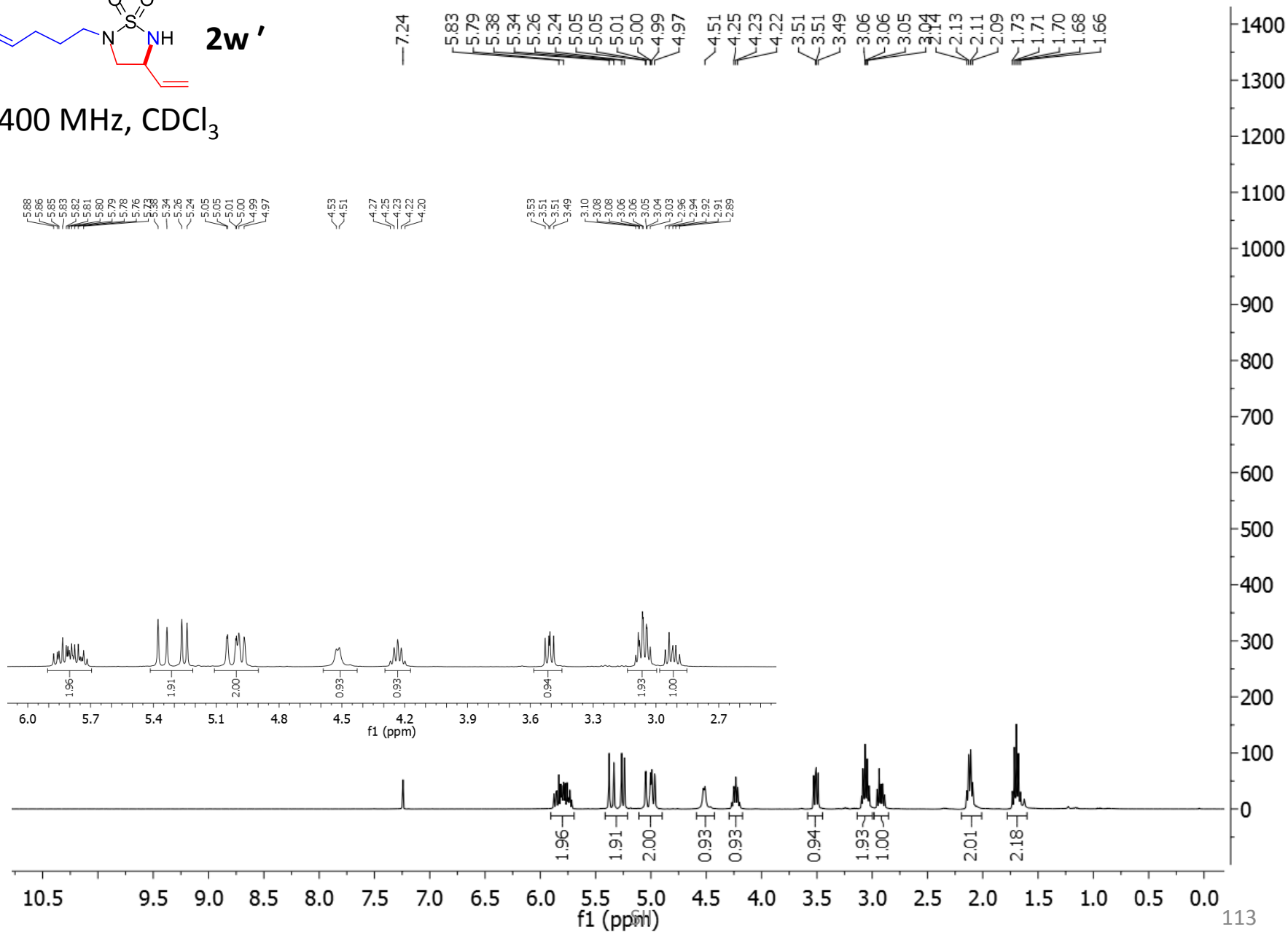
2w

136.019 134.759 116.916 116.528 77.318 77.000 76.681 57.531 48.607 48.166 32.250 28.560

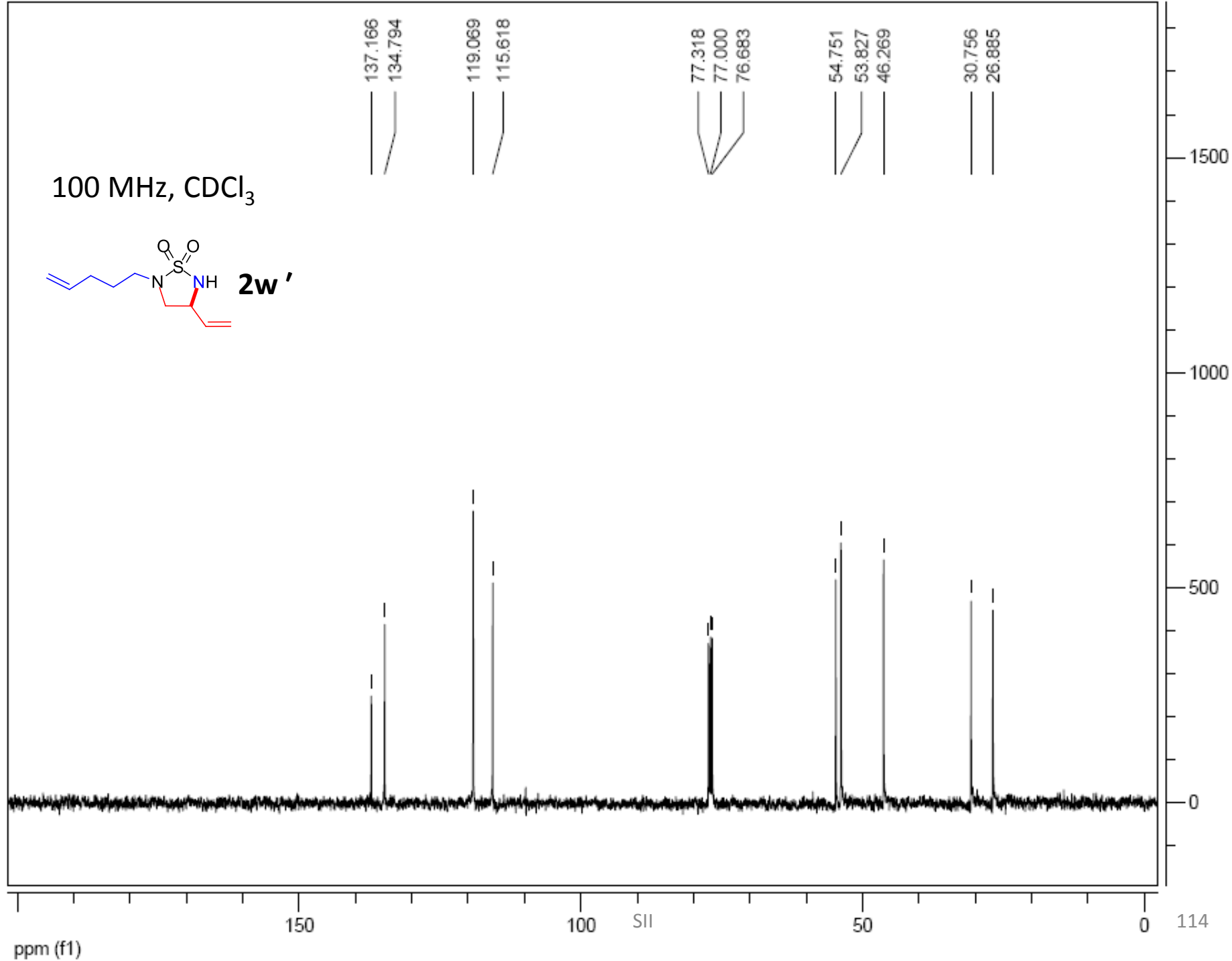
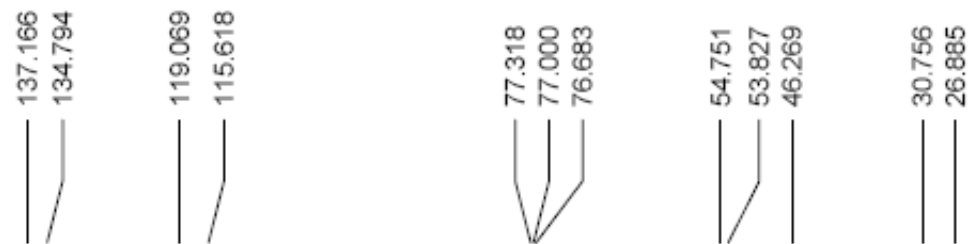
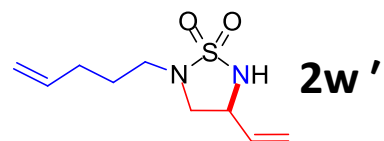




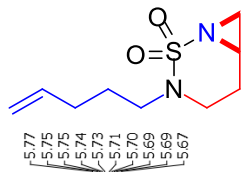
400 MHz, CDCl₃



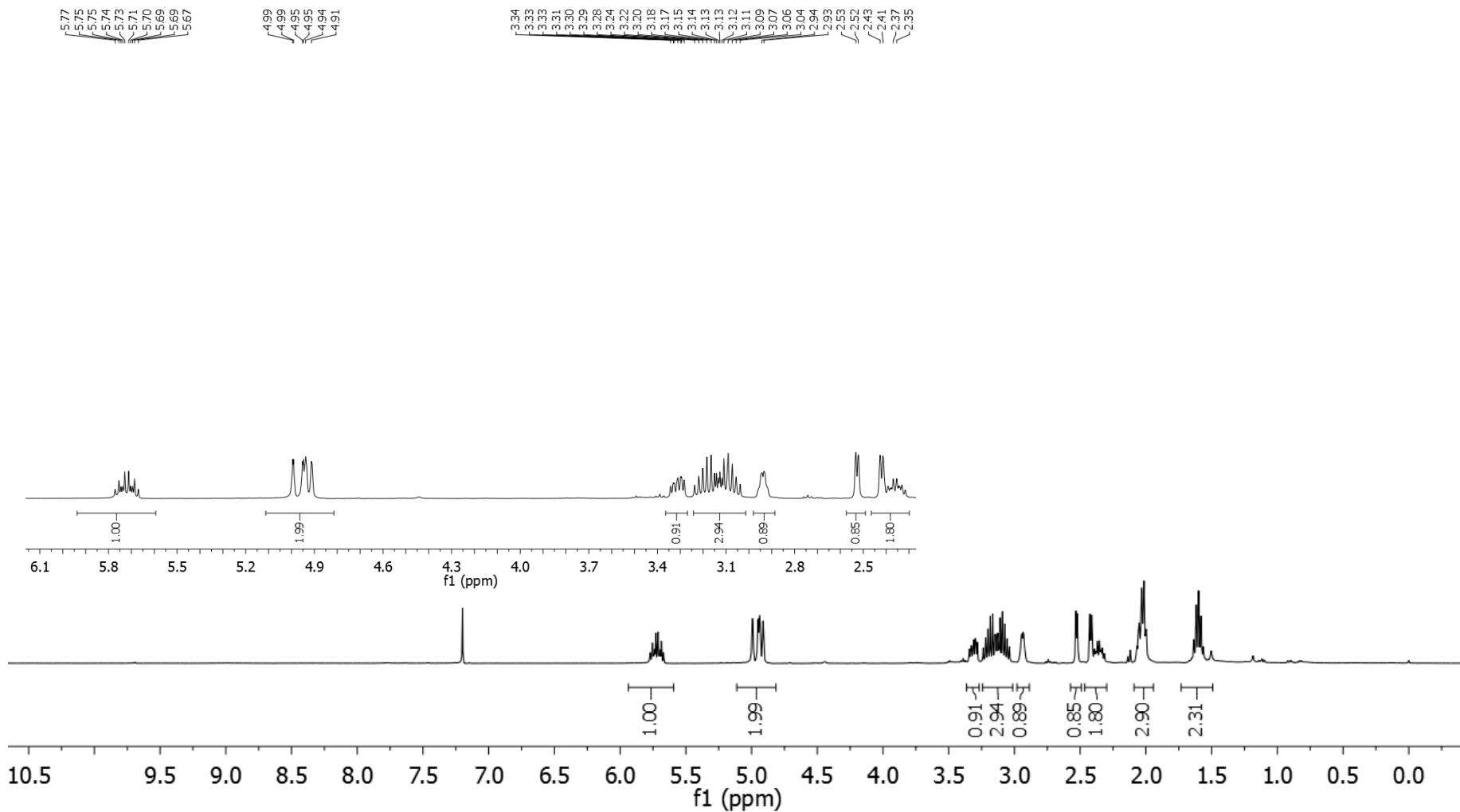
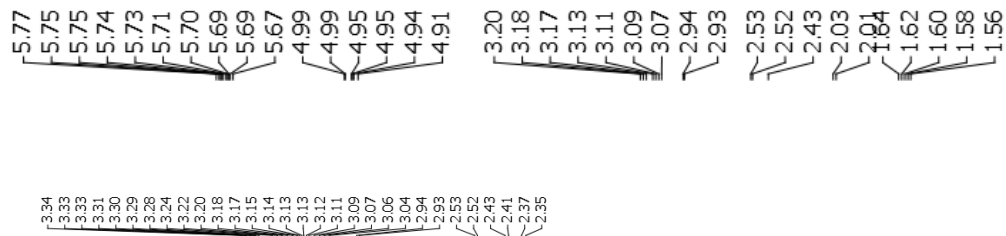
100 MHz, CDCl₃



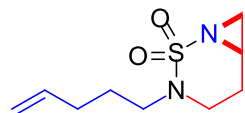
400 MHz, CDCl₃



2w''



100 MHz, CDCl₃



2w''

137.273

115.436

77.318

77.000

76.682

48.964

43.773

40.213

32.893

30.552

27.123

17.278

2500

2000

1500

1000

500

0

116

ppm (f1)

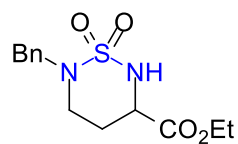
200

150

100

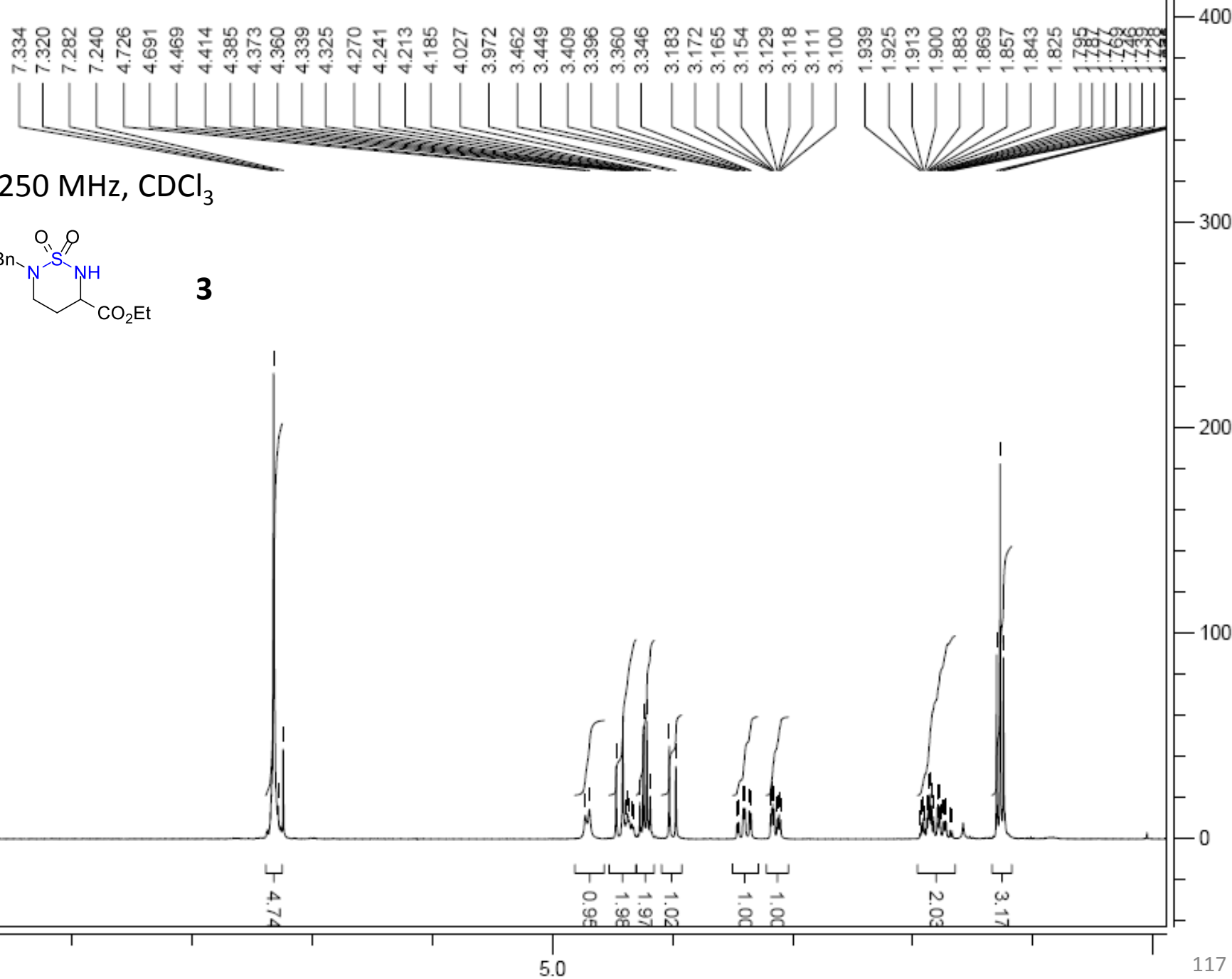
50

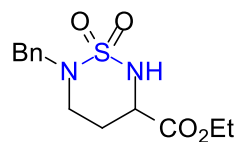
0



250 MHz, CDCl₃

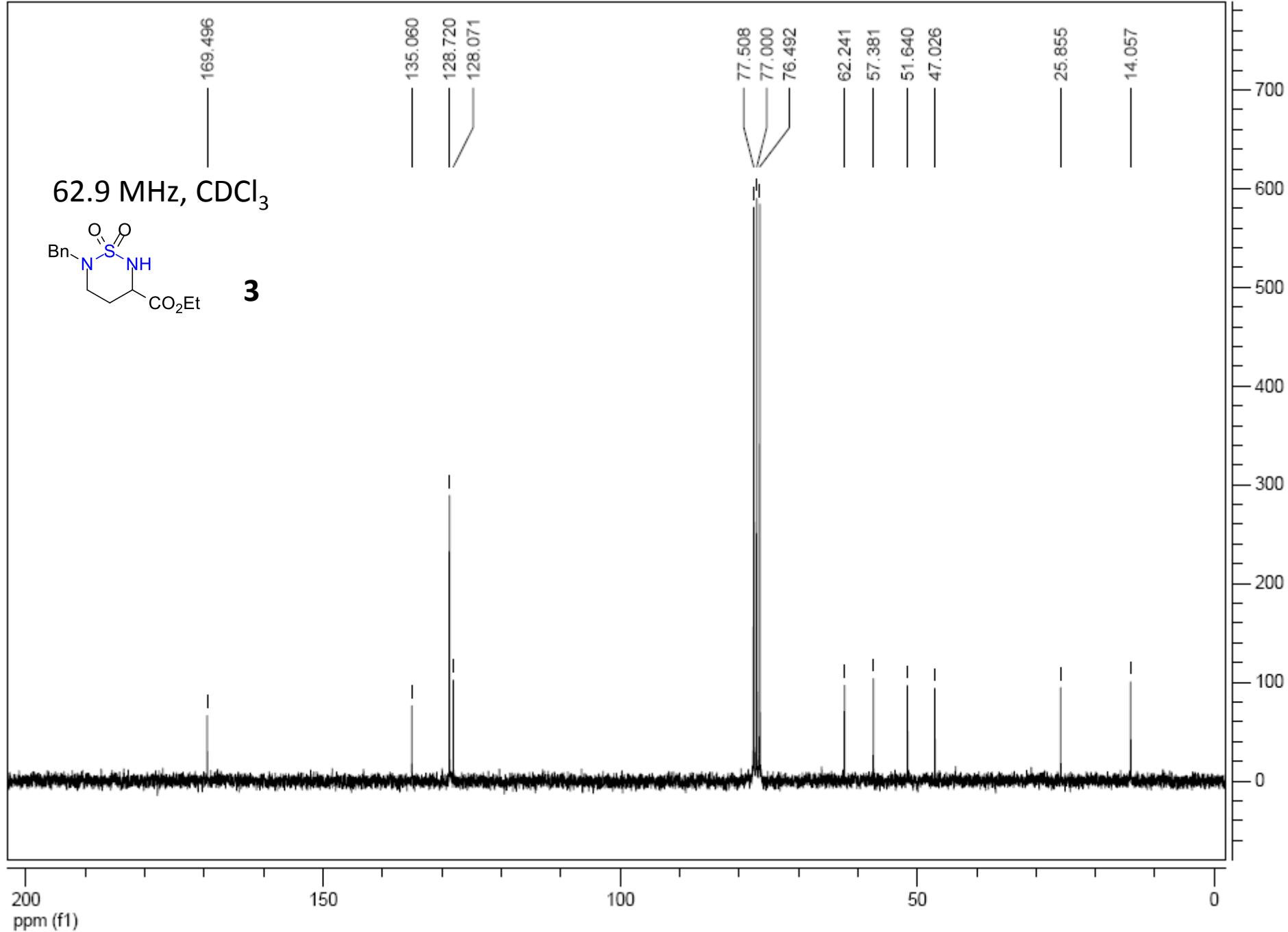
3

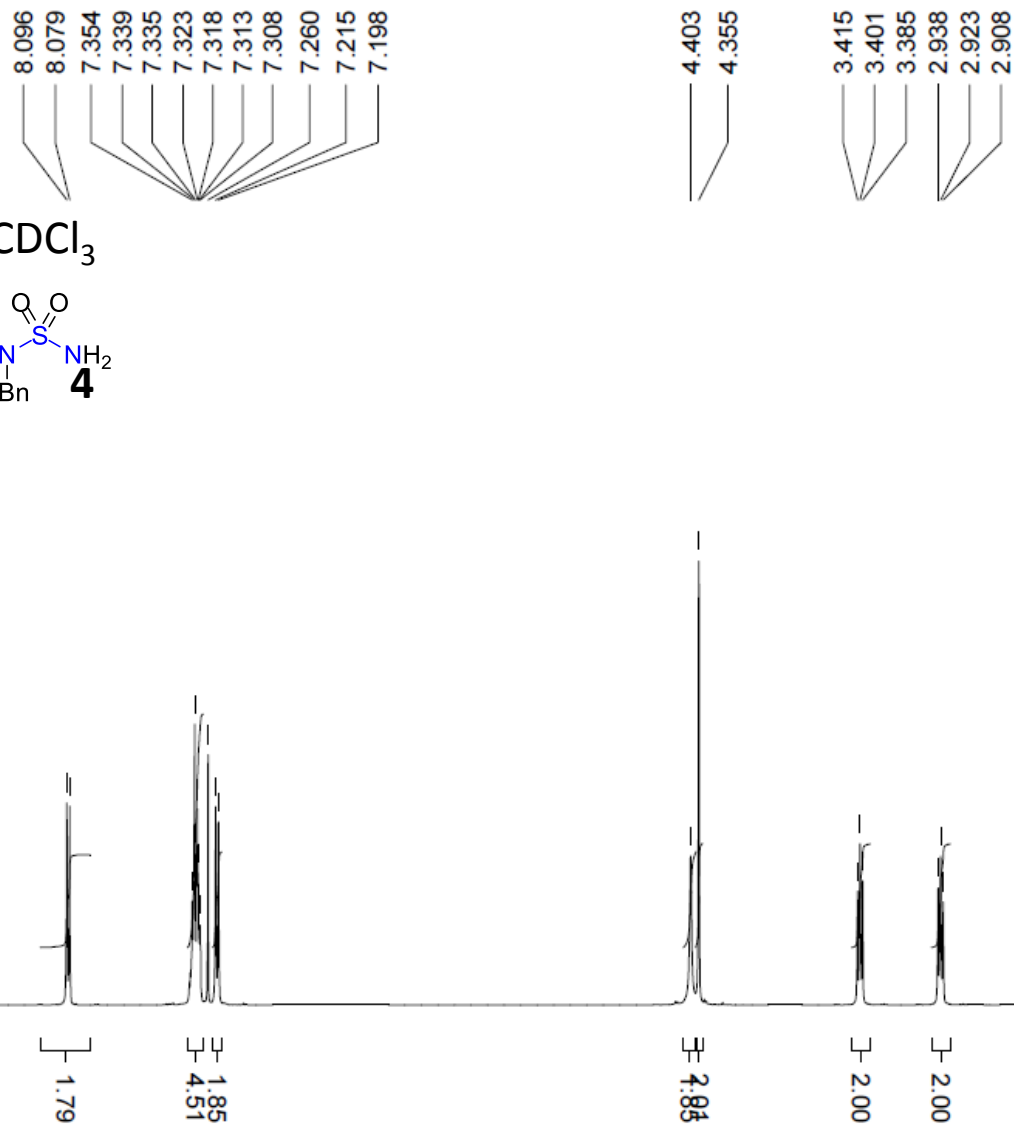
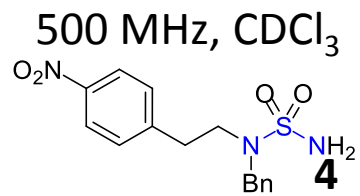




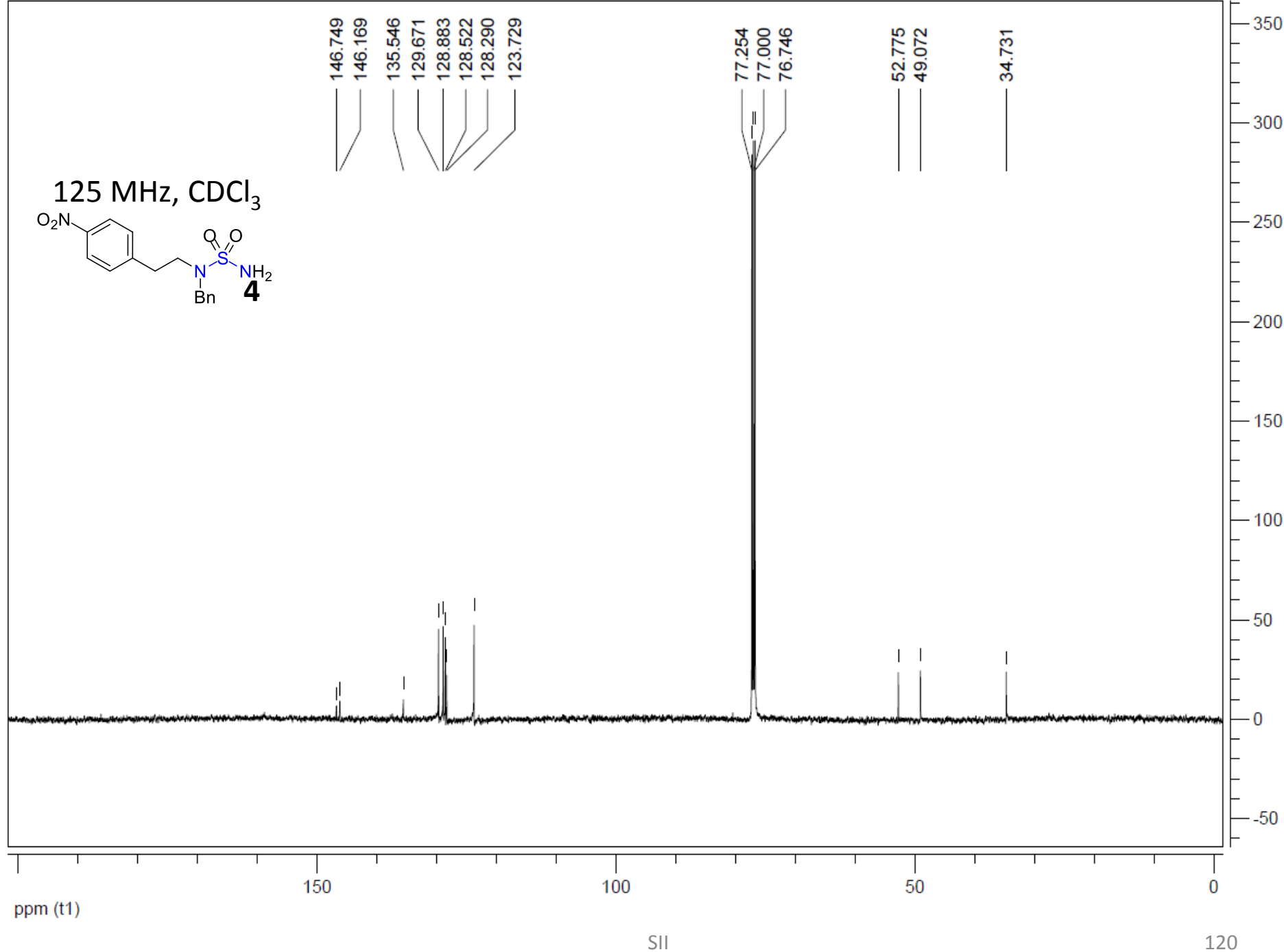
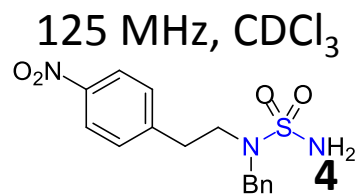
3

62.9 MHz, CDCl₃

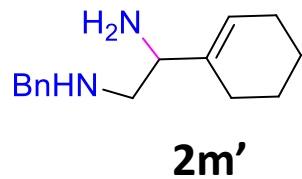




ppm (f1)



400 MHz, CDCl₃



7.25
7.24
7.19

3.25
3.24
3.22

5.55

3.73

3.25
3.24
3.22

2.60
2.59

2.49

2.47

1.94

1.78

1.54

1.53

1.52

1.50

1.49

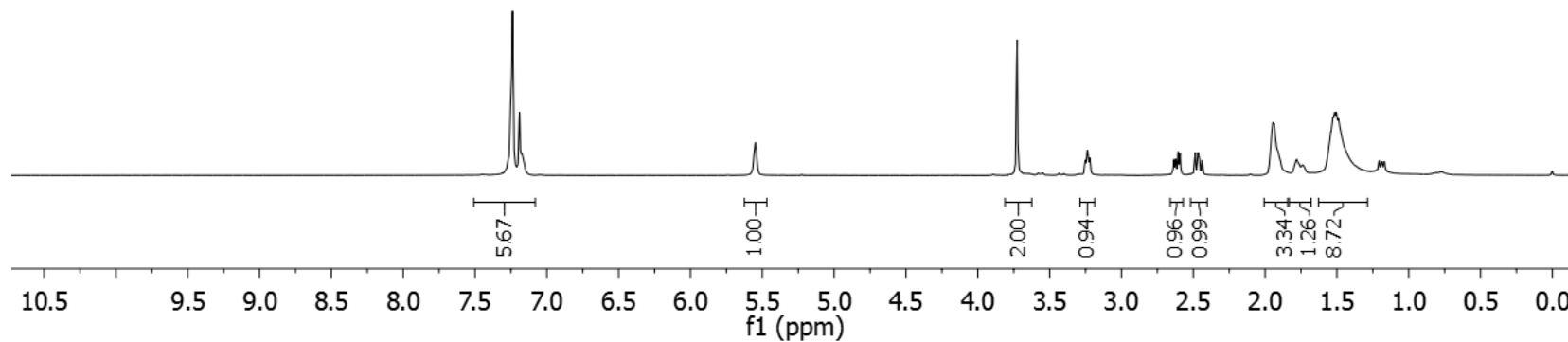
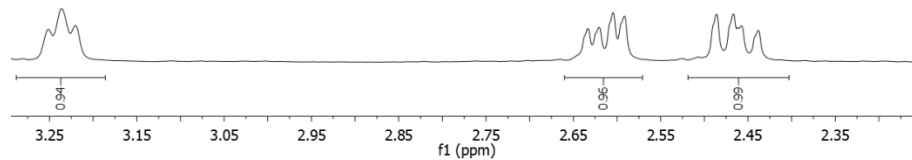
1.21

1.19

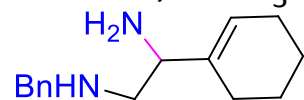
1.17

2.63
2.62
2.60
2.59

2.49
2.47
2.46
2.44



62.9 MHz, CDCl₃



2m'

140.501
139.675
128.340
128.037
126.841
122.293
77.508
77.000
76.492
57.190
53.854
53.643
25.036
24.629
22.799
22.663

1500

1000

500

0

122

SII

ppm (f1)