Supporting Information

Urotensin-II$^{(4-11)}$ Azasulfuryl Peptides: Synthesis and Biological Activity

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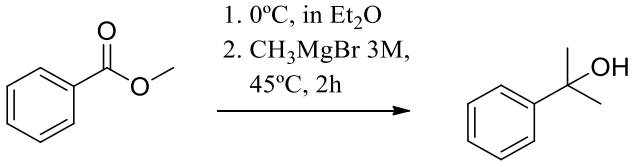
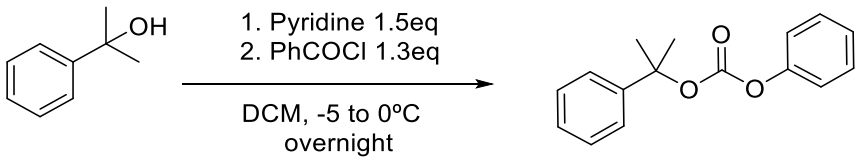
Synthesis of 2-phenylisopropyl alcohol (cumyl alcohol)



To a solution of methyl benzoate (630 μL, 5 mmol) in 5 mL of anhydrous ether, 5 mL of a solution of 3 M methyl magnesium bromide in ether was added drop-wise at 0 °C. The reaction was brought to 45 °C with a warm water bath. After 2 h, TLC (70:30 hexanes/EtOAc) demonstrated complete conversion of the ester to 2-phenyl-propan-2-ol. The reaction mixture was poured slowly into an ice-cooled saturated solution of NH_4Cl, and extracted with EtOAc (3 x 20 mL). The organic phase were washed with brine (2 x 10 mL), dried over $MgSO_4$, filtered and evaporated to afford 2-phenyl-propan-2-ol (98% yield): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.60 (6H, s), 1.75 (1H, s), 7.26 (1H, t, *J* = 7.1), 7.36 (2H, t, *J* = 7.5), 7.5 (2H, d, *J* = 7.4); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 150.0, 128.6, 127.1, 124.8, 72.5, 32.2.

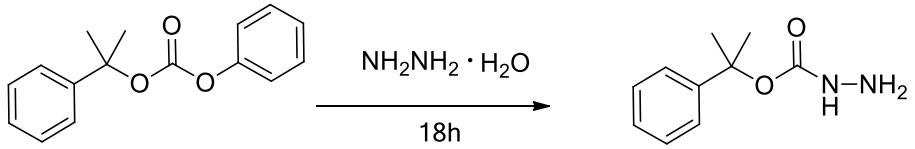
Synthesis of cumyl carbonate



To a stirred solution of cumyl alcohol (800 mg, 5.88 mmol) in DCM (5 mL) and pyridine (750 μL, 1.5 mol-equiv) at –5 °C, a solution of phenyl chloroformate (1 mL, 1.3 mol-equiv) in 4 mL of DCM was added drop-wise over 30 min. A thick paste formed gradually after the addition of the chloroformate, and the reaction mixture was further stirred overnight at 0 °C. The mixture was diluted with DCM (50 mL), washed with 1N HCl, 1N NaOH, H_2O and brine, dried over $MgSO_4$, filtered and evaporated to afford a quantitative yield of cumyl carbonate as colorless oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.80 (6H, s), 7.26 (2H, t, *J* = 7.1), 7.29 (2H, t, *J* = 7.5), 7.38 (2H, t, *J* = 7.5),

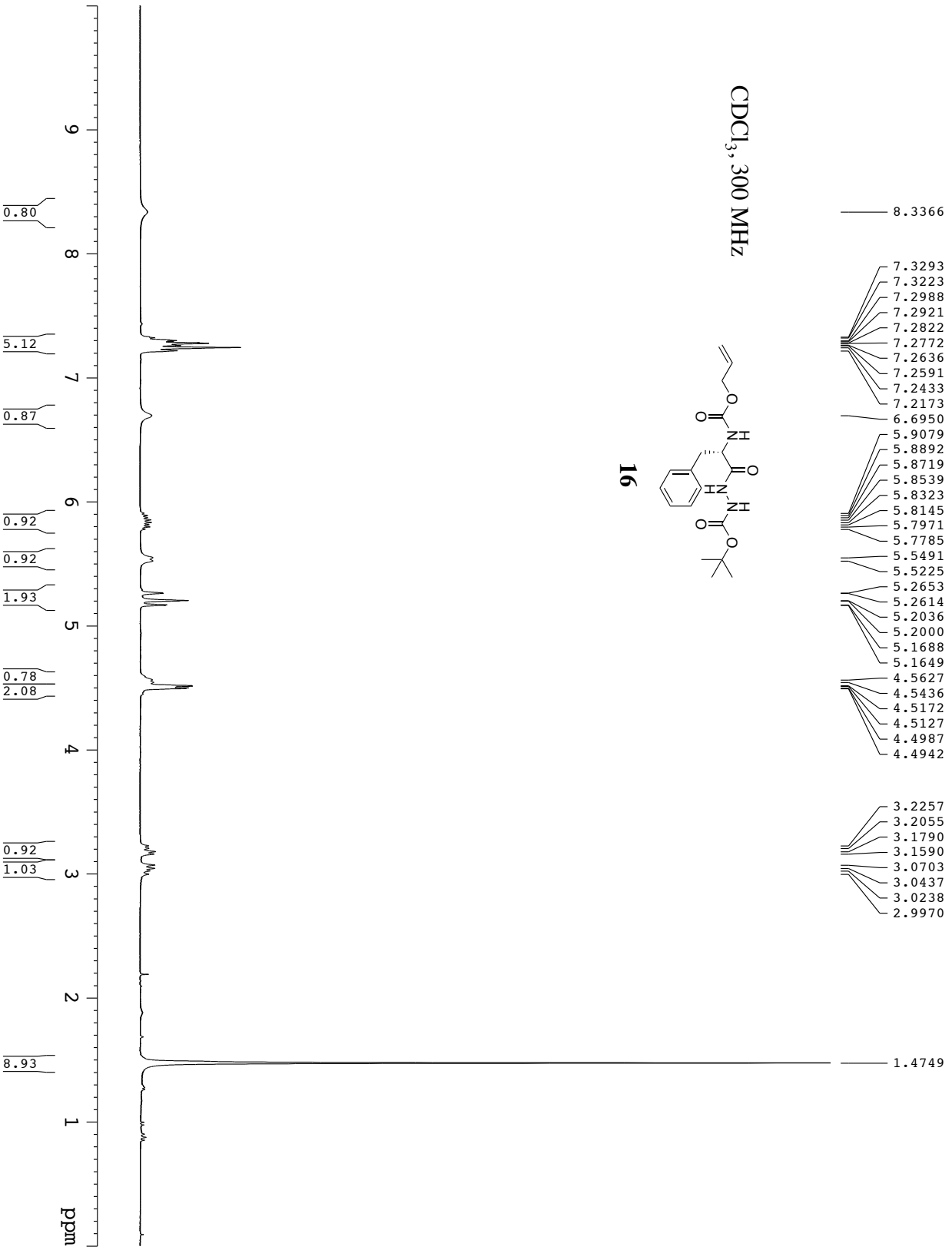
7.42 (2H, d, J = 7.4), 7.54 (2H, d, J = 7.4); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 151.6, 151.1, 144.7, 133.0, 129.4, 128.6, 125.1, 124.1, 121.8, 84.5, 28.2.

Synthesis of cumyl carbazate



Hydrazine hydrate (2.2 mL of a 50-60% solution in H_2O) was mixed with cumyl carbonate (1 g, 3.9 mmol) with vigorous stirring for 18 h. The mixture was poured into ice water, extracted with ethyl acetate (3 x 20 mL), and the combined organic phase was washed with NaOH 1N, H_2O, and brine, dried with sodium sulfate, filtered, and evaporated to a residue that was purified by trituration with hexane to afford cumyl carbazate as a pale yellow oil (88% yield): R_f = 0.15 (60:40 hexanes/EtOAc); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.60 (6H, s), 1.75 (1H, s), 3.73 (2H, s) 7.26 (1H, t, J = 7.1), 7.36 (2H, t, J = 7.5), 7.5 (2H, d, J = 7.4); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 145.7, 128.4, 127.5, 127.6, 124.3, 81.8, 29.3.

NMR spectra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7.3223
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7.2591
7.2433
7.2173
6.6950
5.9079
5.8892
5.8719
5.8539
5.8323
5.8145
5.7971
5.7785
5.5491
5.5225
5.2653
5.2614
5.2036
5.2000
5.1688
5.1649
4.5627
4.5436
4.5172
4.5127
4.4987
4.4942
3.2257
3.2055
3.1790
3.1590
3.0703
3.0437
3.0238
2.9970
1.4749
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0.92
0.92
1.93
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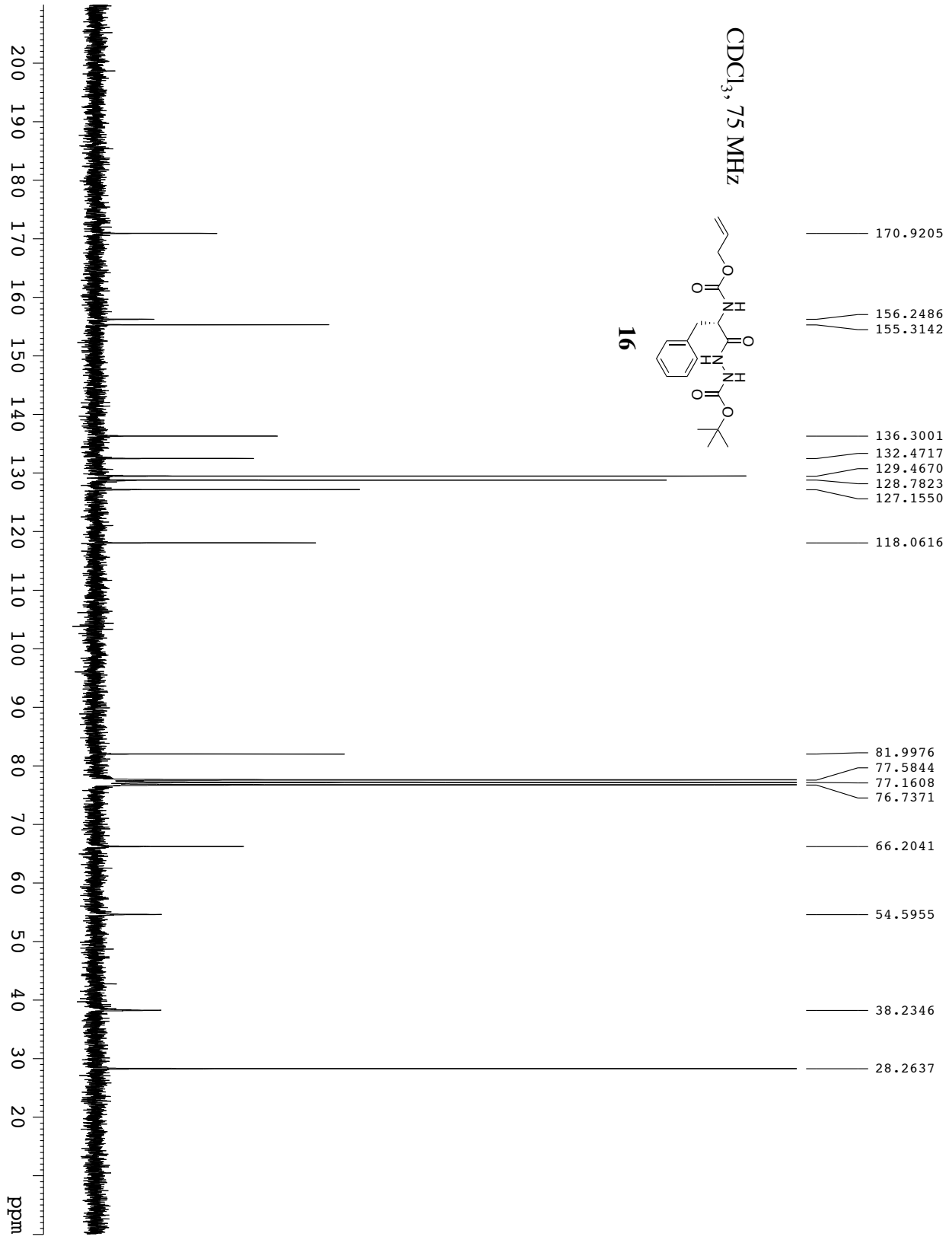
2.08
0.92
1.03
8.93
ppm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132.4717
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128.7823
127.1550
118.0616
81.9976
77.5844
77.1608
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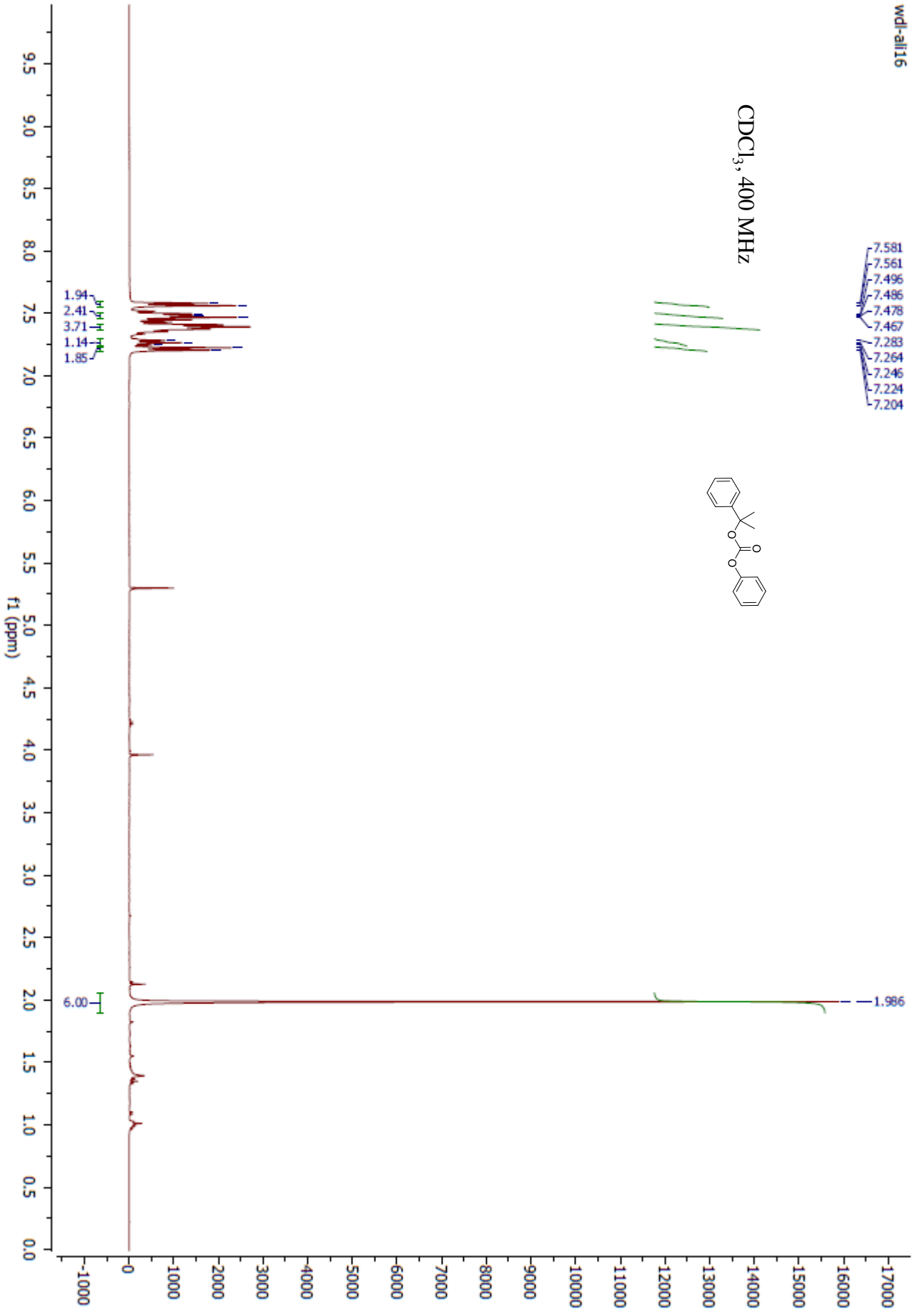
ppm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7.496
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7.283
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7.204
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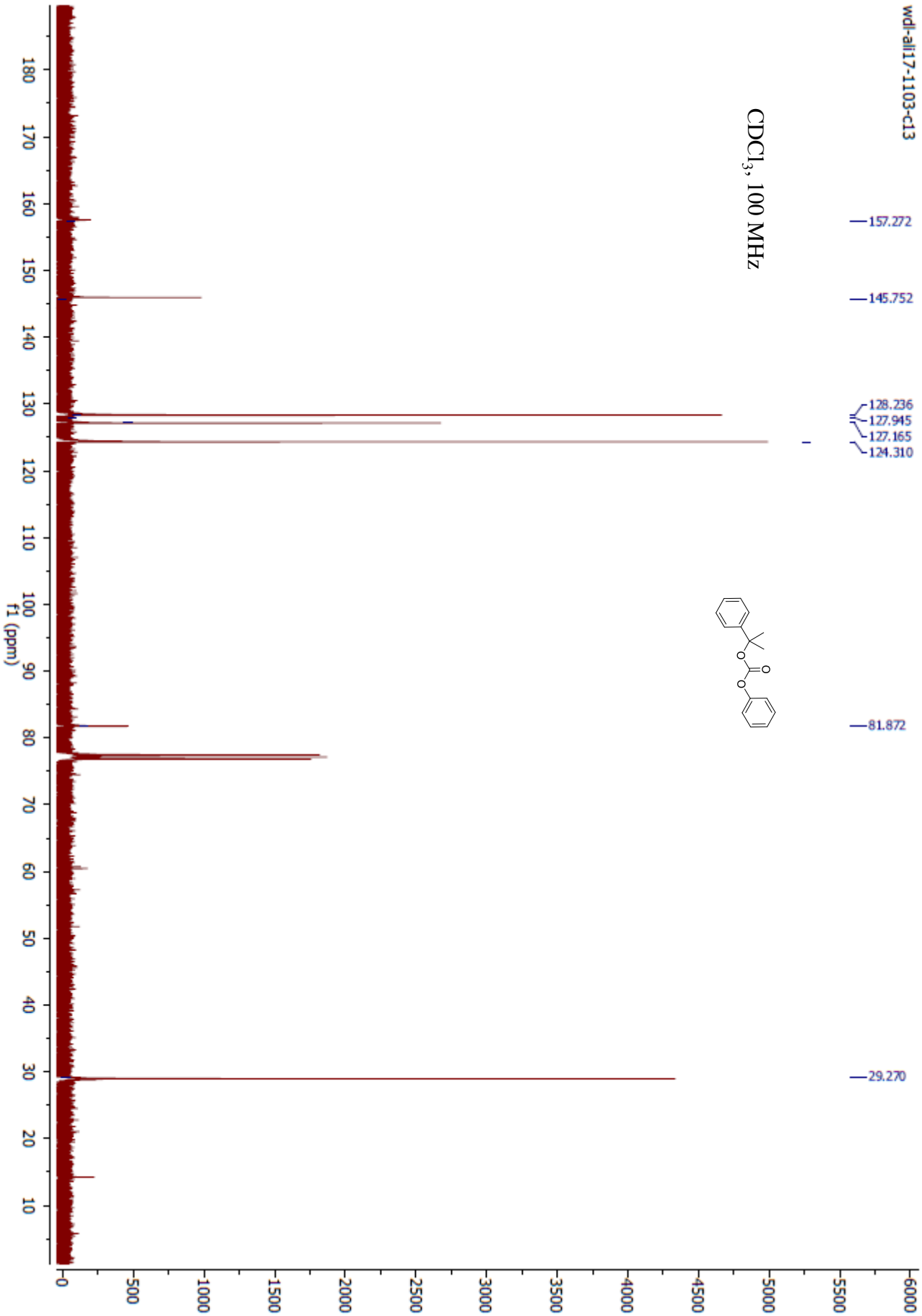
2.41
3.71
1.14
1.85
6.00
1.986
f1 (ppm)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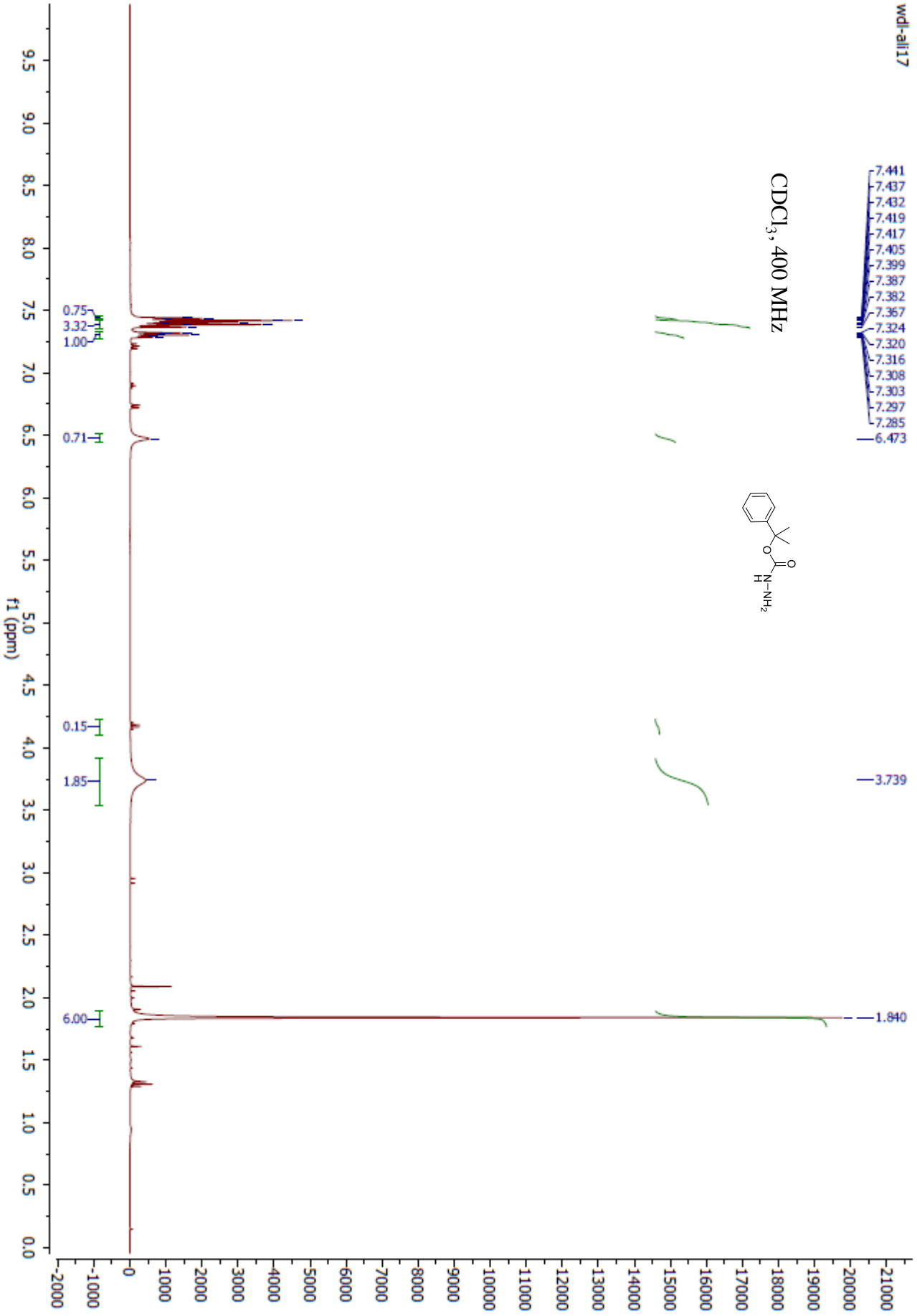
127.165
124.310
81.872
29.270
f1 (ppm)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CDCl3, 400 MHz
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7.432
7.419
7.417
7.405
7.399
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7.367
7.324
7.320
7.316
7.308
7.303
7.297
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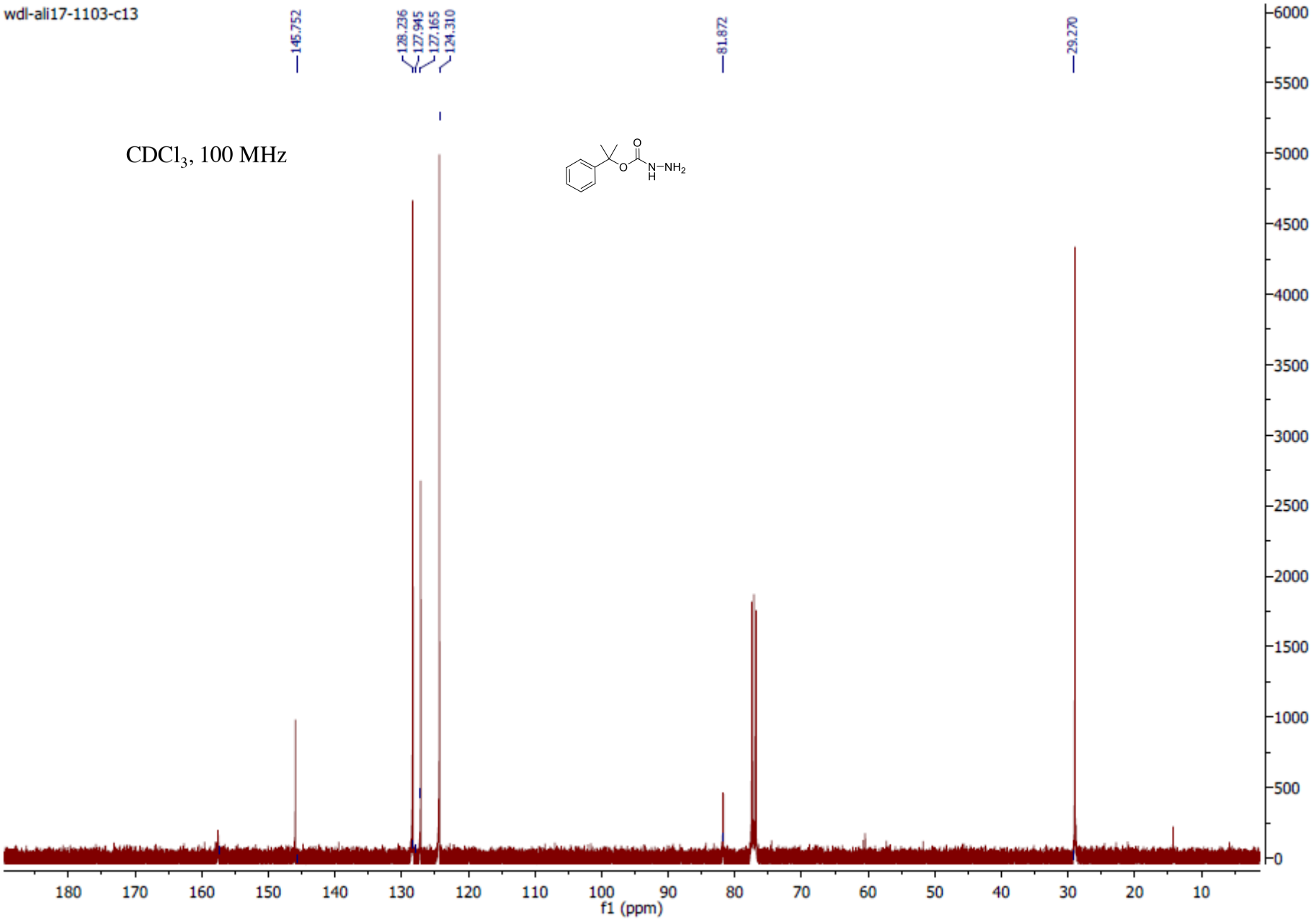
0.15
1.85
6.00
f1 (ppm)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145.752
128.236
127.945
127.165
124.310
81.872
29.270
CDCl3, 100 MHz
O
O
N
H
NH2
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
180
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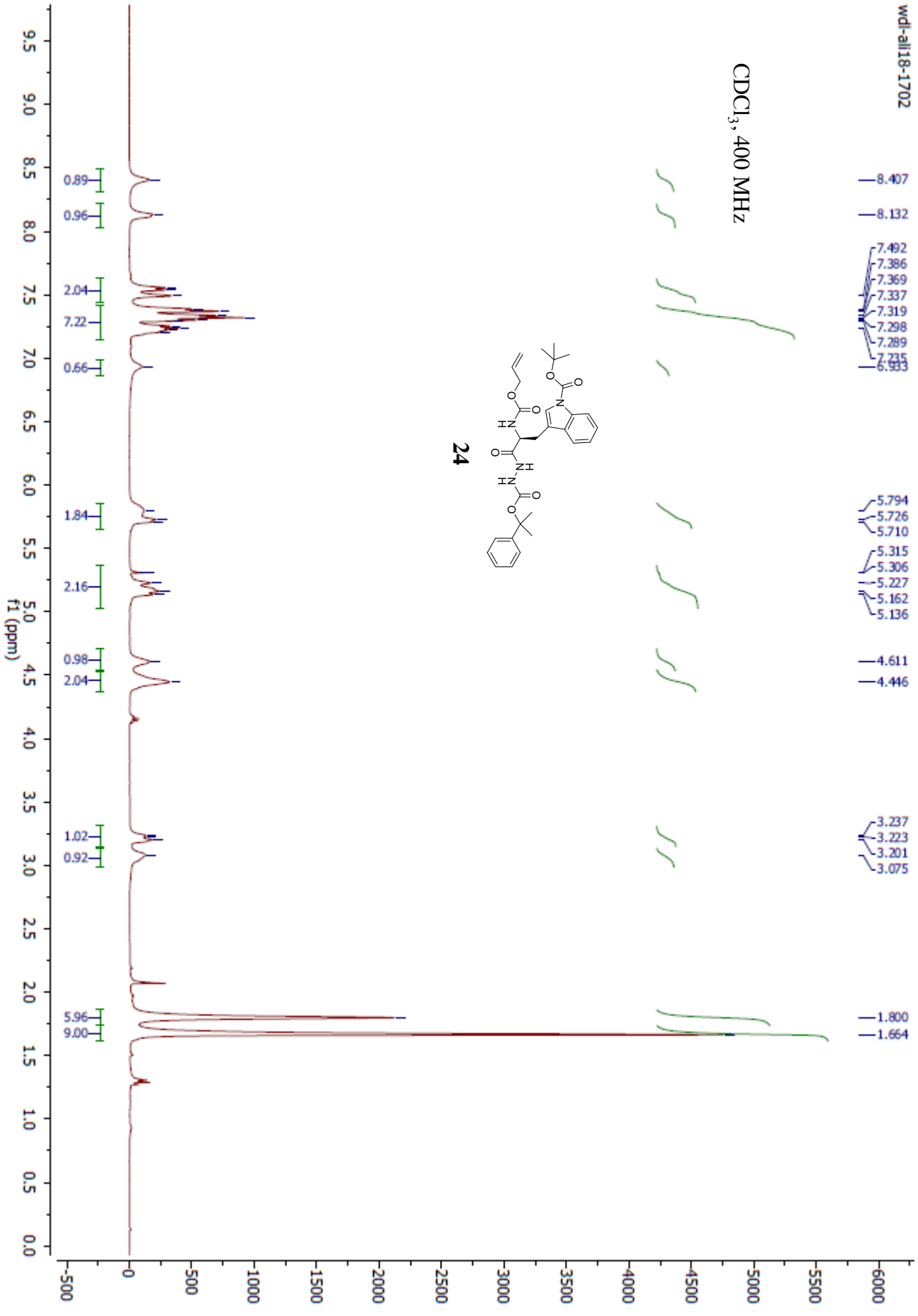
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.337
7.319
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7.289
7.235
6.933
5.794
5.726
5.710
5.315
5.306
5.227
5.162
5.136
4.611
4.446
3.237
3.223
3.201
3.075
1.800
1.664
0.89
0.96
2.04
7.22
0.66
1.84
2.16
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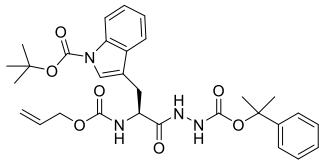
f1 (ppm)

wdl-ali18-1702-c13

$CDCl_3$, 100 MHz



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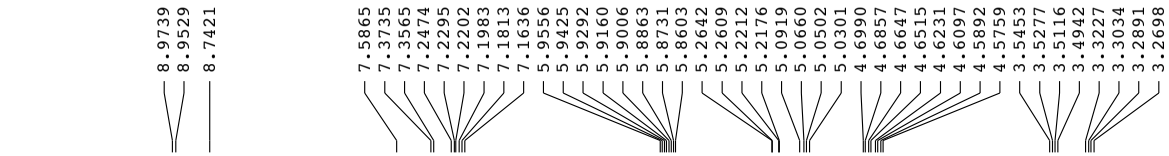
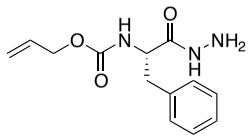


pyridine-d_5, 400 MHz



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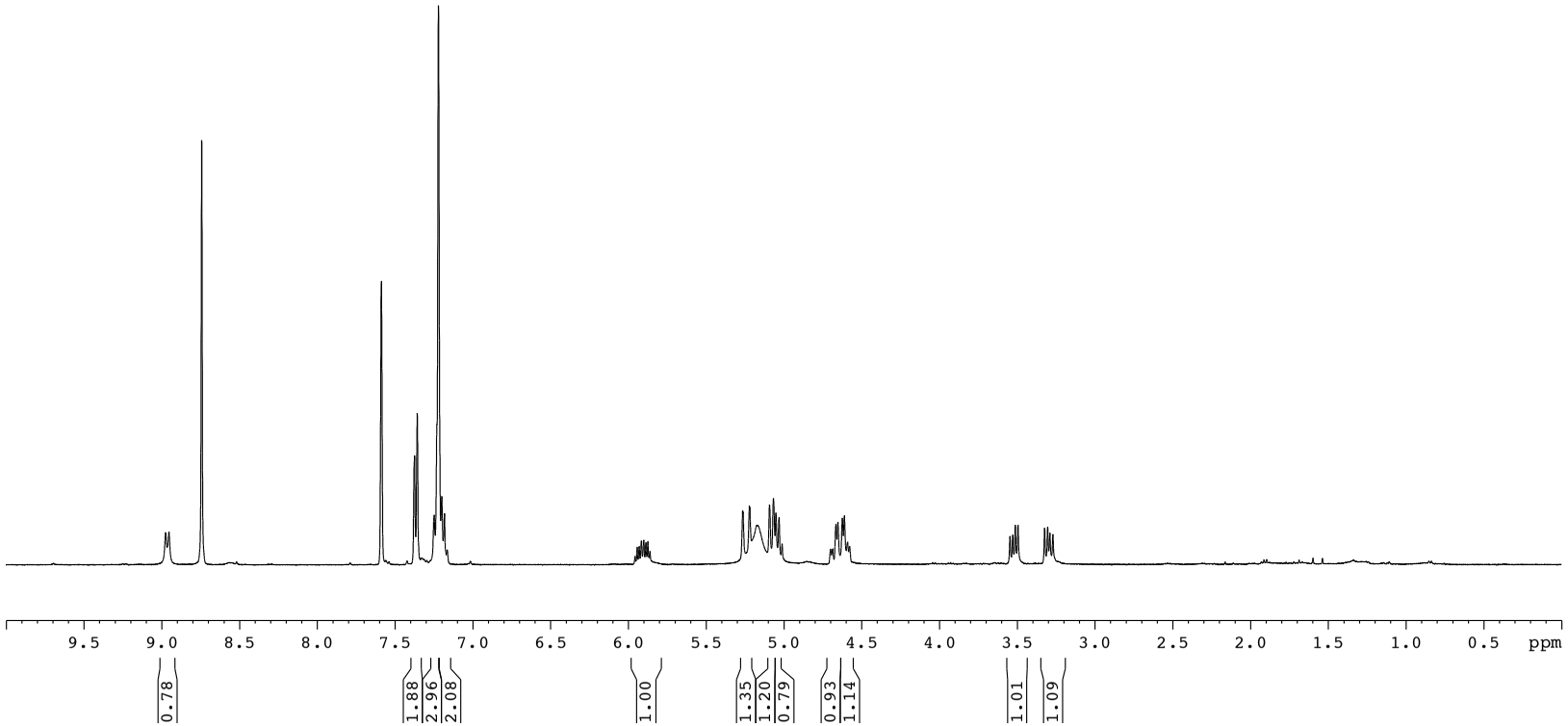

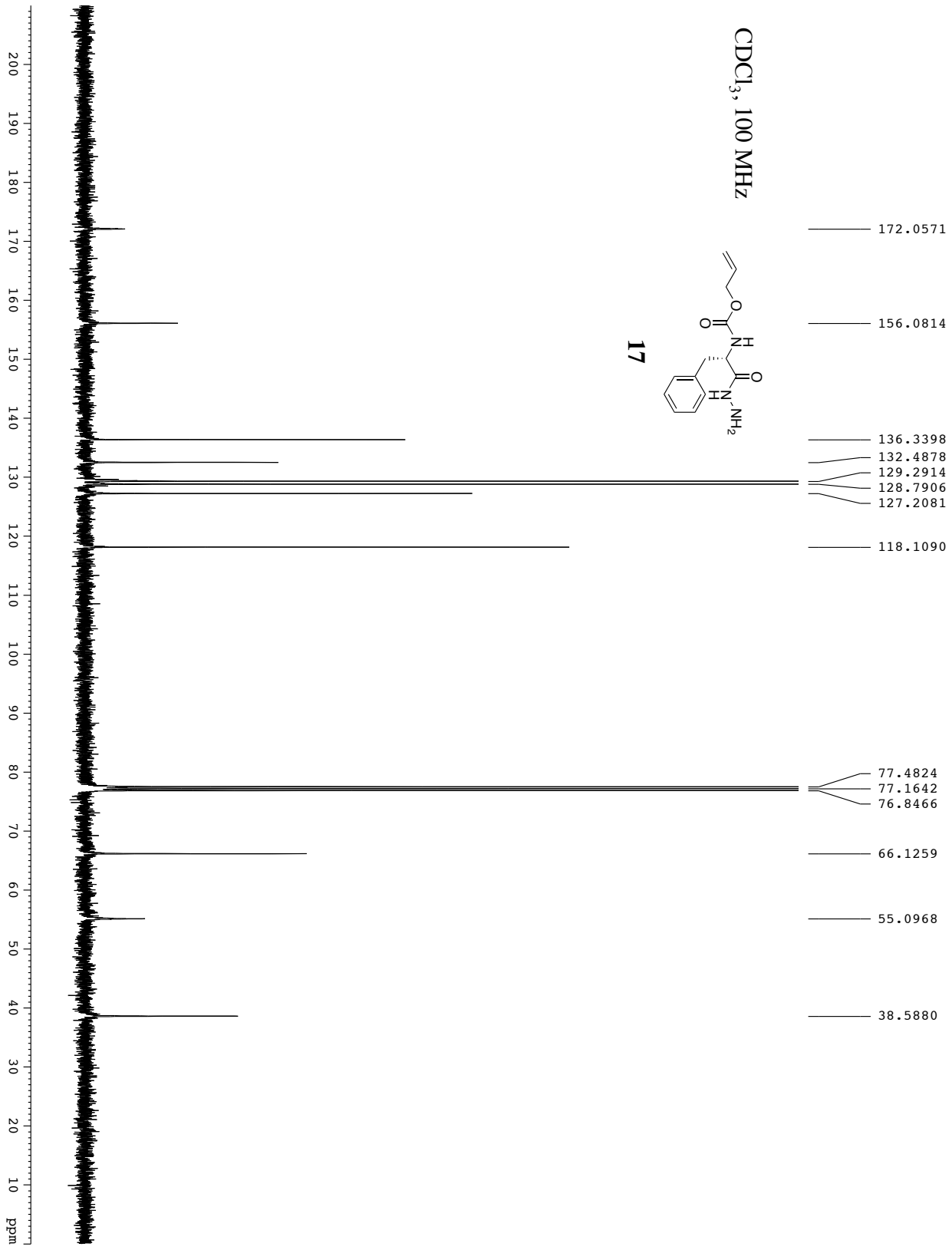
CDCl3, 100 MHz
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172.0571
156.0814
136.3398
132.4878
129.2914
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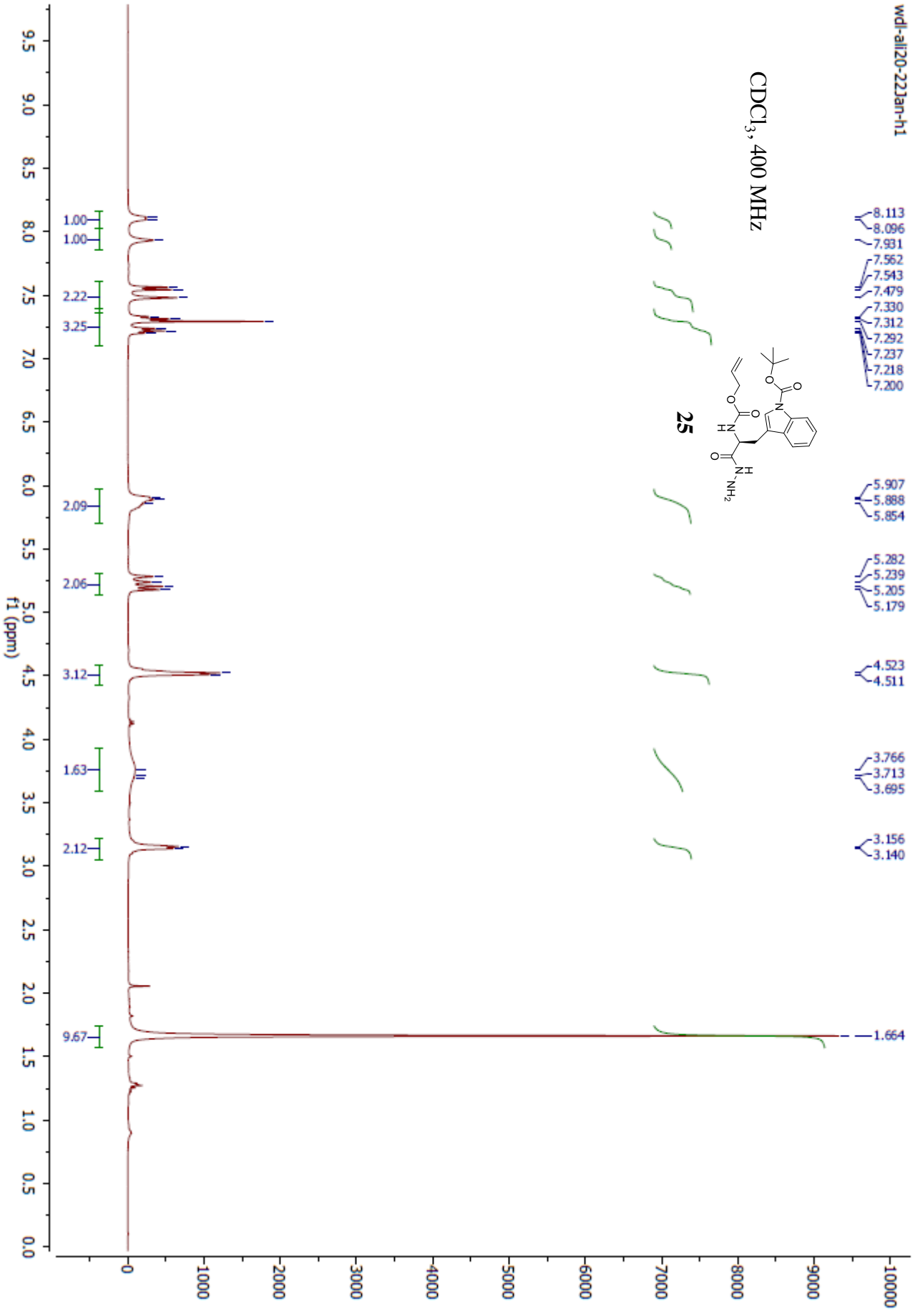
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7.312
7.292
7.237
7.218
7.200
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5.888
5.854
5.282
5.239
5.205
5.179
4.523
4.511
3.766
3.713
3.695
3.156
3.140
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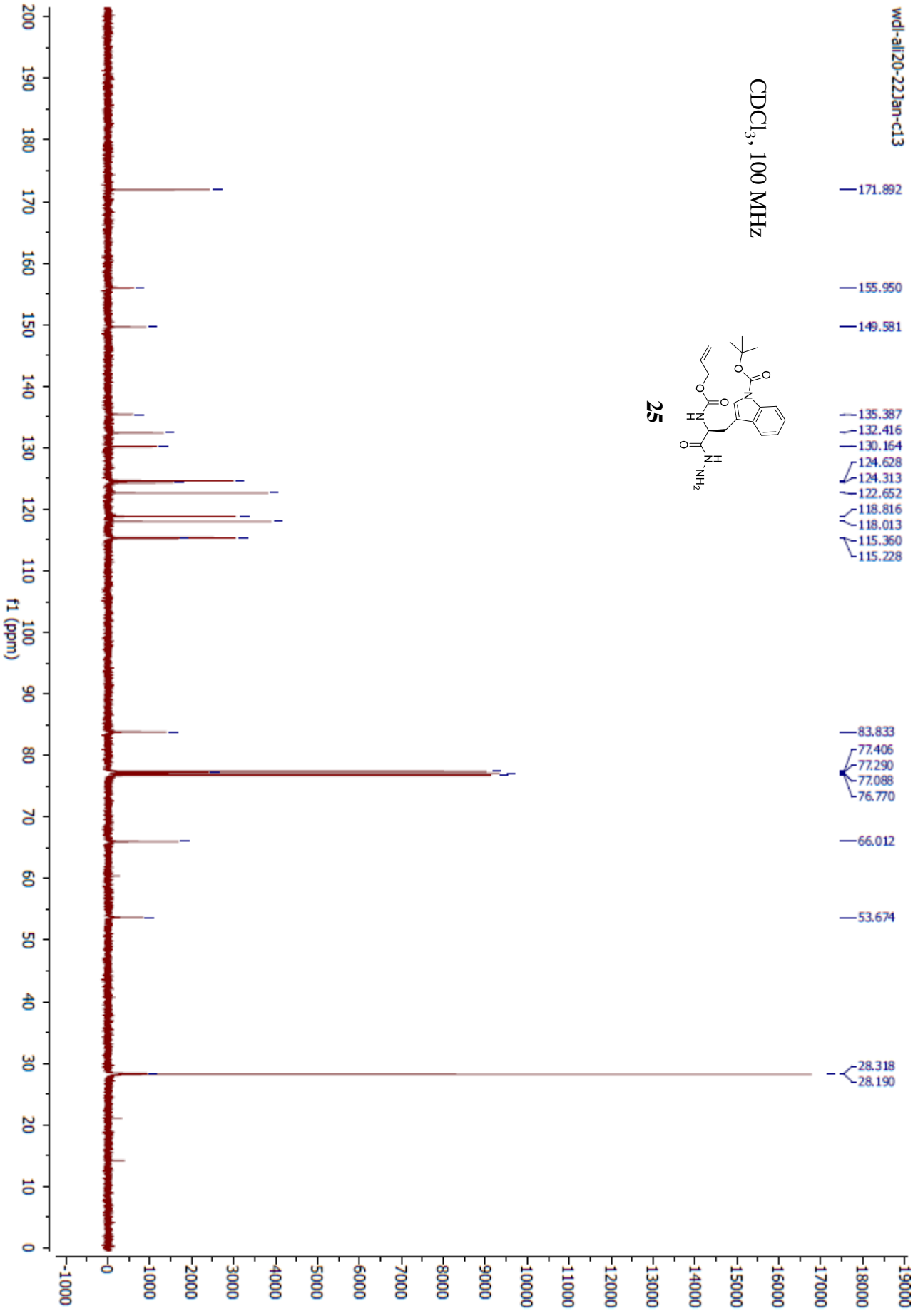
f1 (ppm)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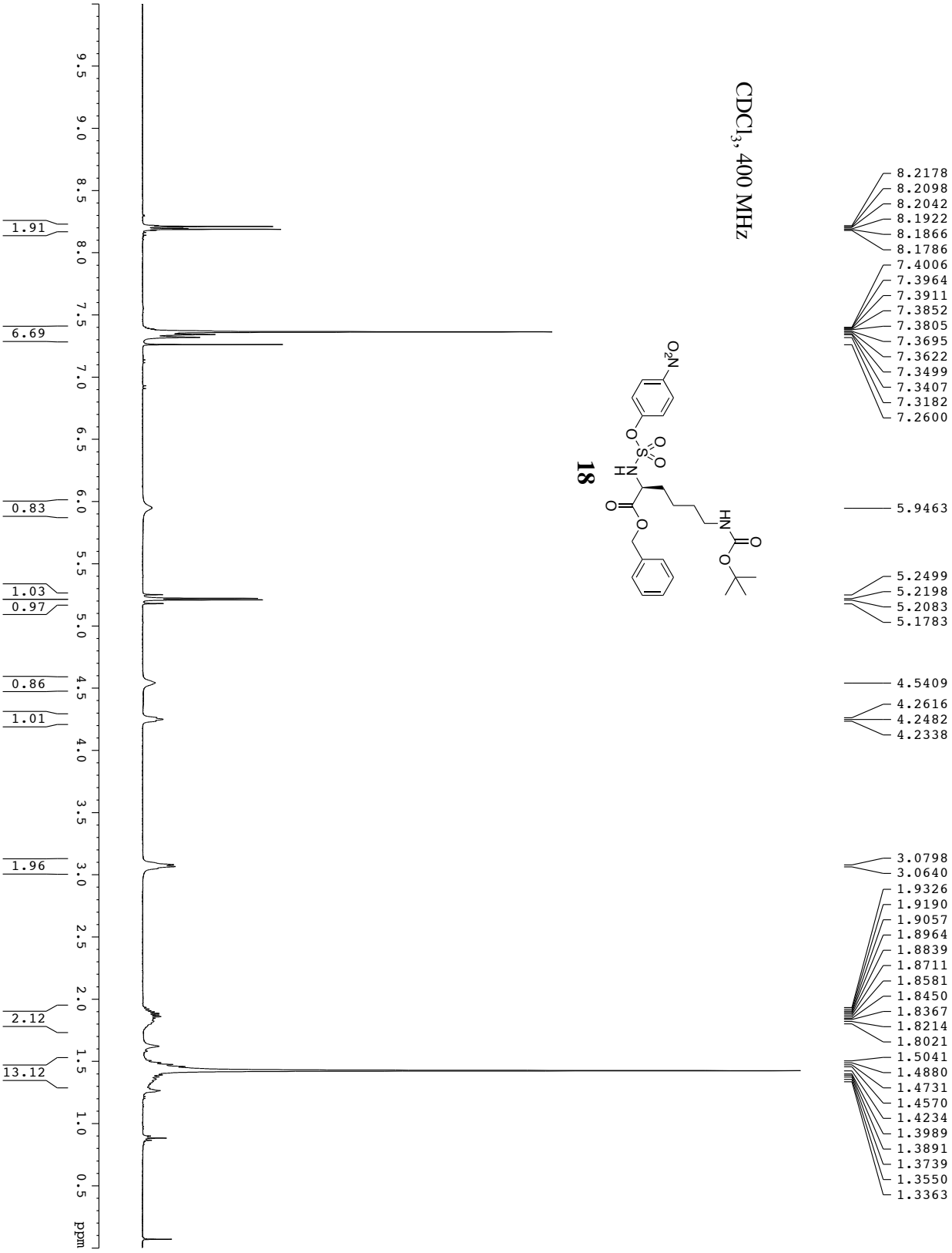
f1 (ppm)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5.2198
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5.1783
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4.2482
4.2338
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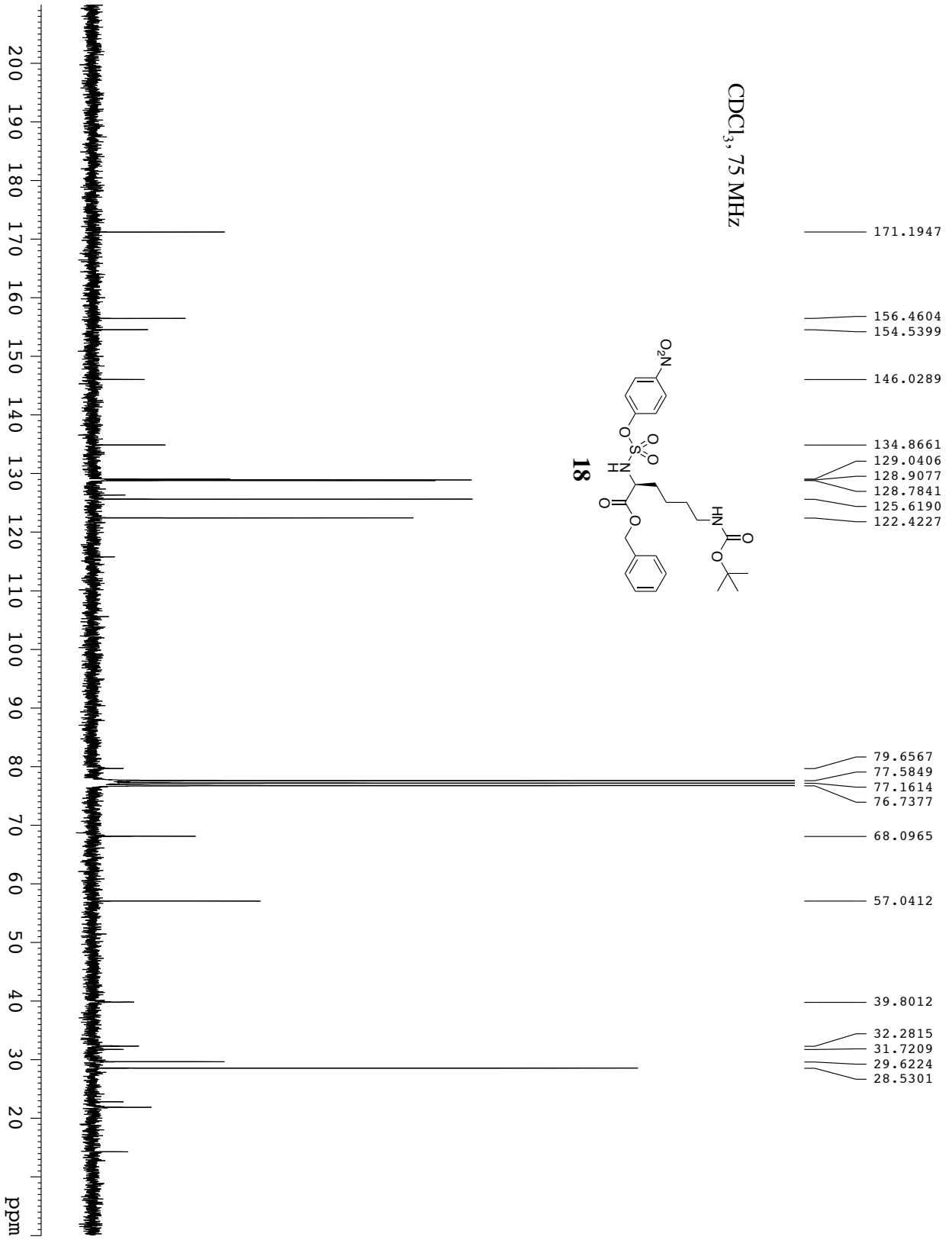
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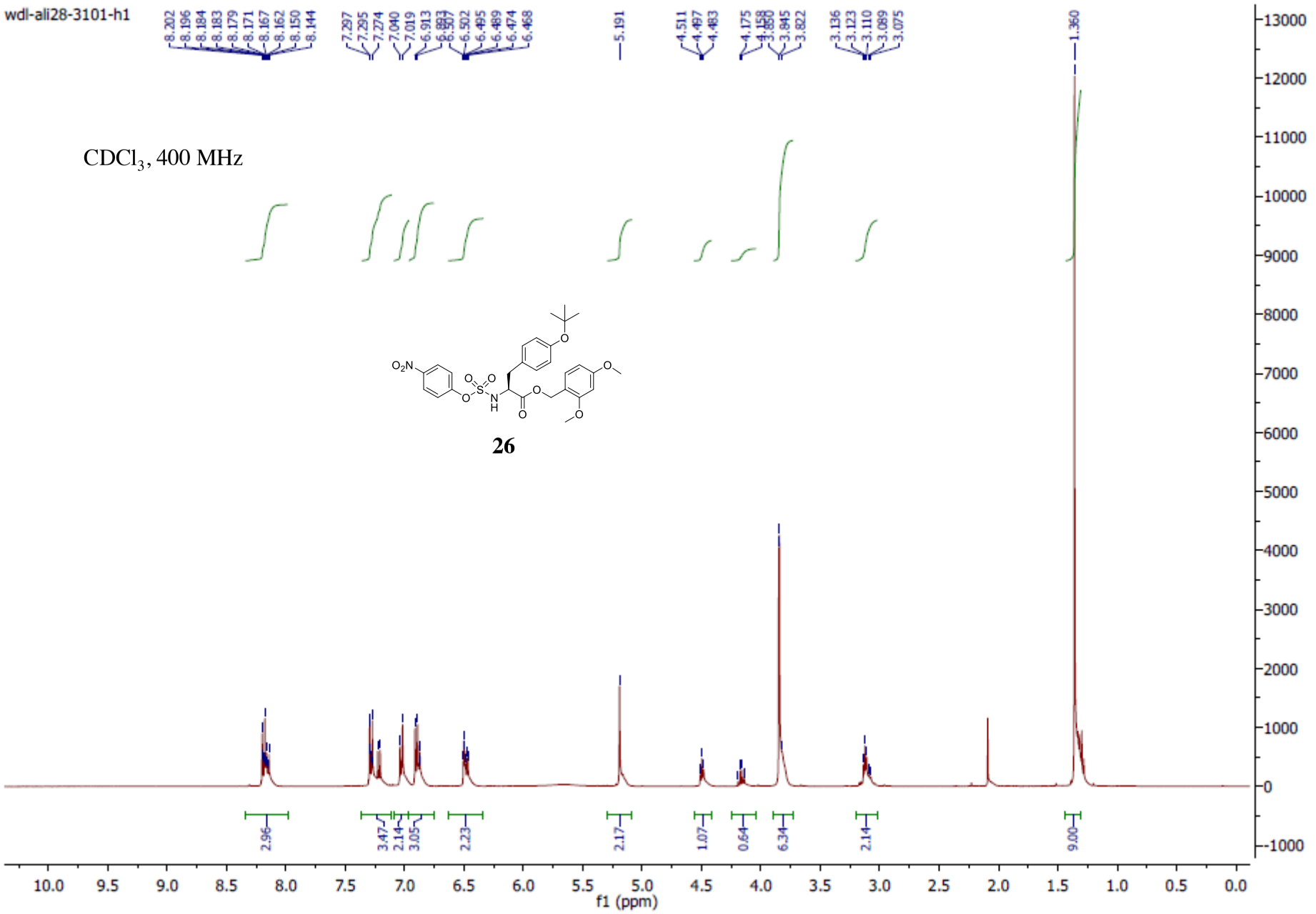
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6.474
6.468
5.191
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4.497
4.483
4.175
3.850
3.845
3.822
3.136
3.123
3.110
3.089
3.075
1.360
CDCl3, 400 MHz
O2N
O
S
N
H
O
O
O
O
O
O
26
2.96
3.47
2.14
3.05
2.23
2.17
1.07
0.64
6.34
2.14
9.00
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
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9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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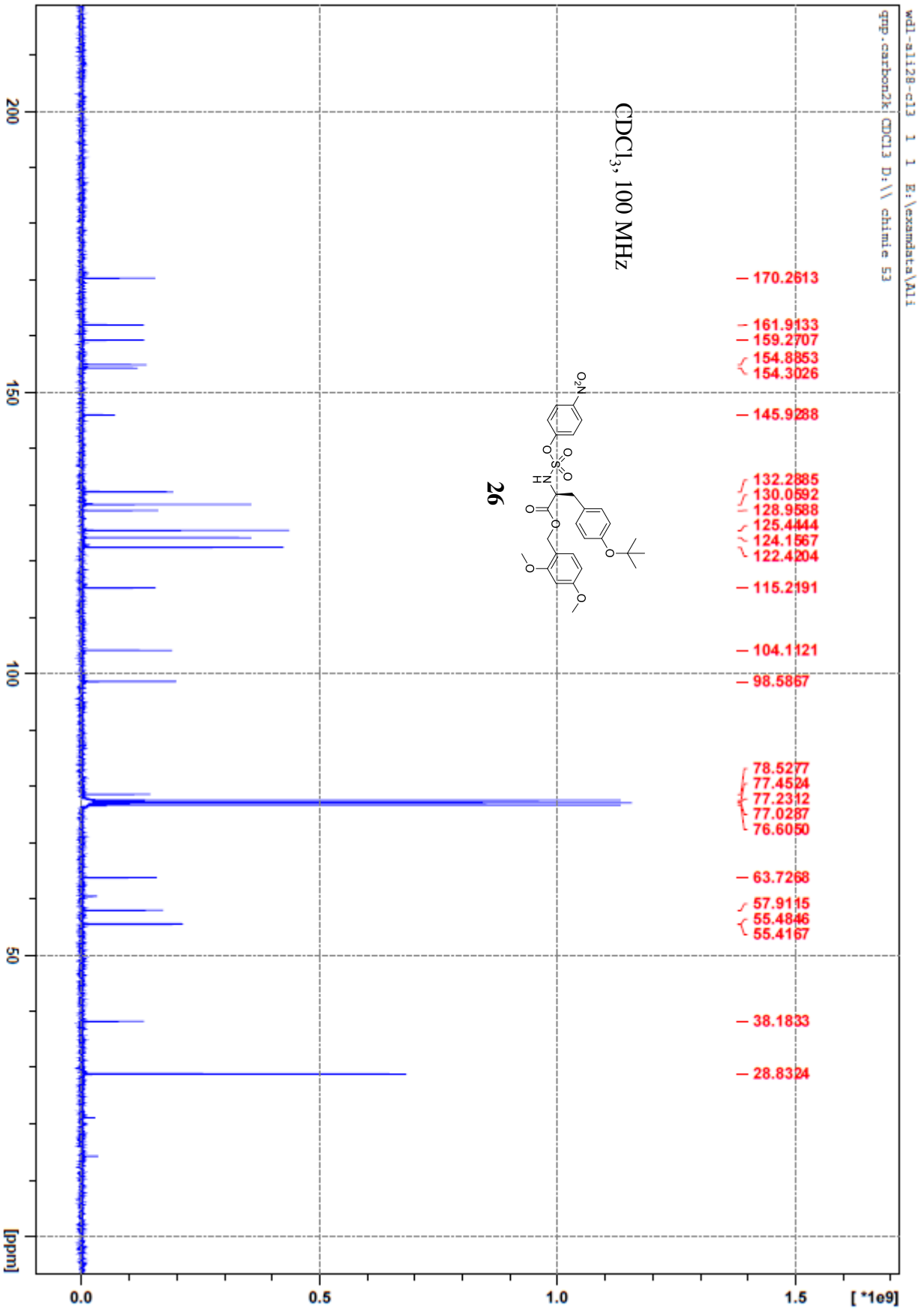
f1 (ppm)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CDCl3, 100 MHz
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130.0592
128.9588
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122.4204
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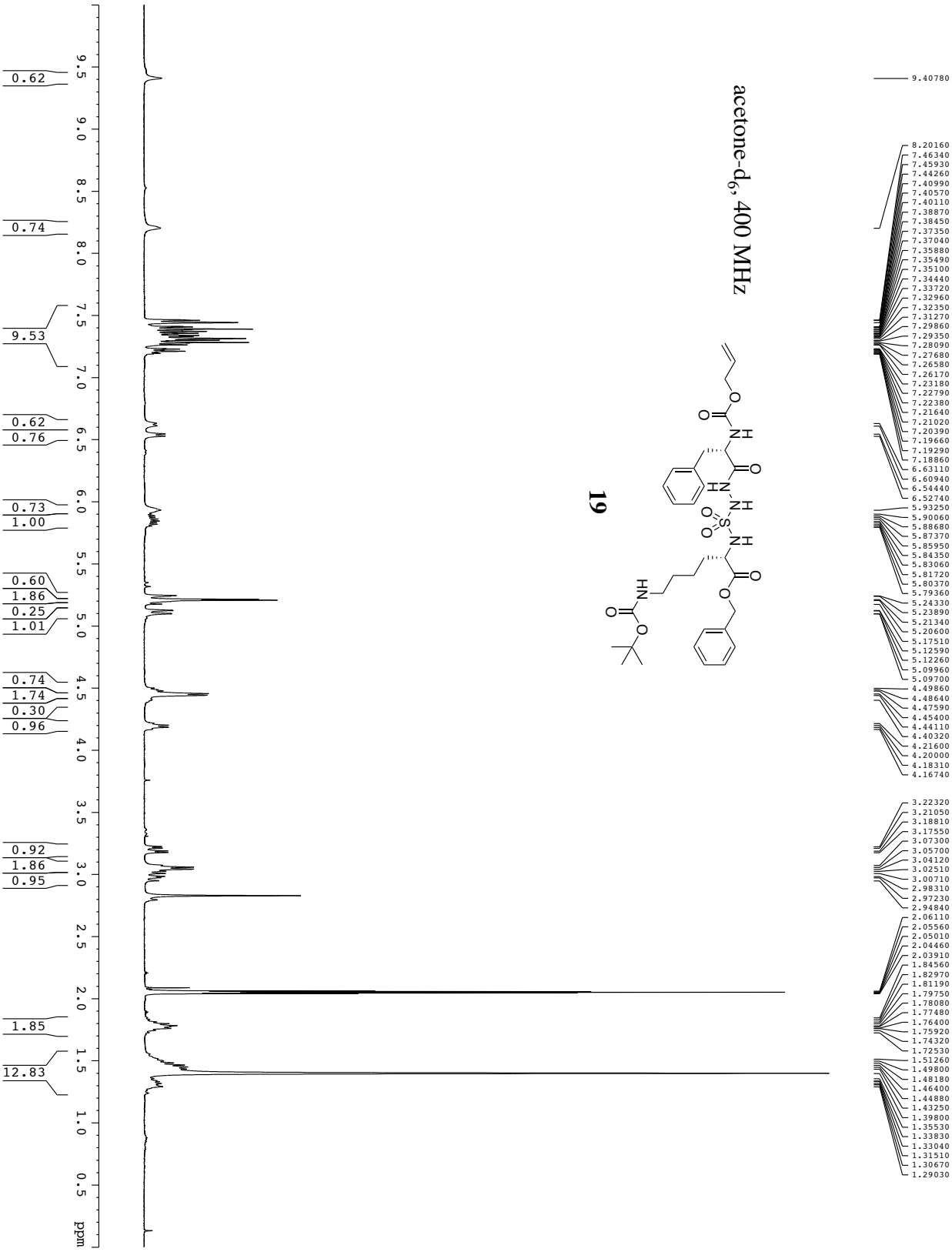
acetone-d6, 400 MHz
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0.74
9.53
0.62
0.76
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1.86
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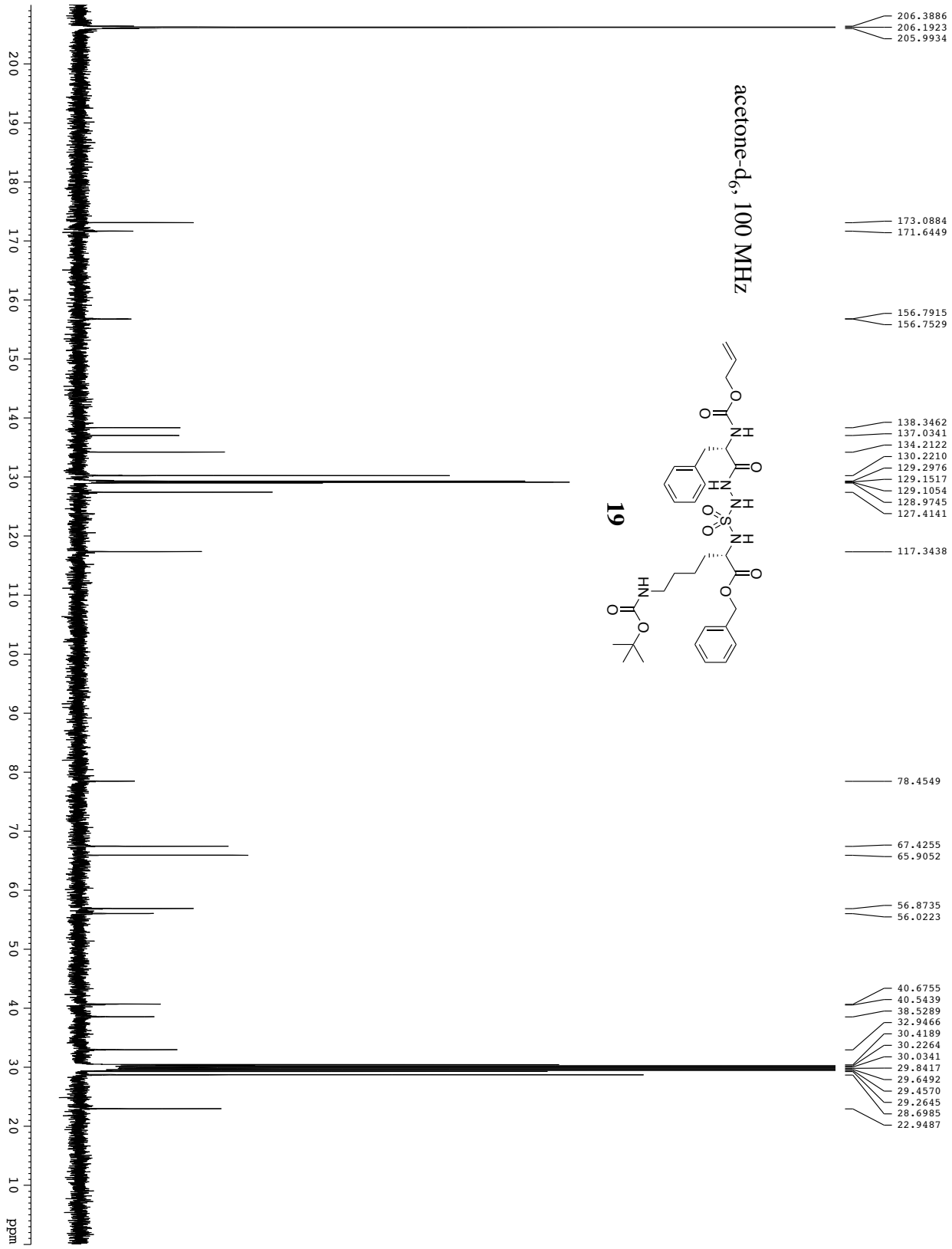
1.86
0.95
1.85
12.83
ppm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129.2976
129.1517
129.1054
128.9745
127.4141
117.3438
78.4549
67.4255
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56.8735
56.0223
40.6755
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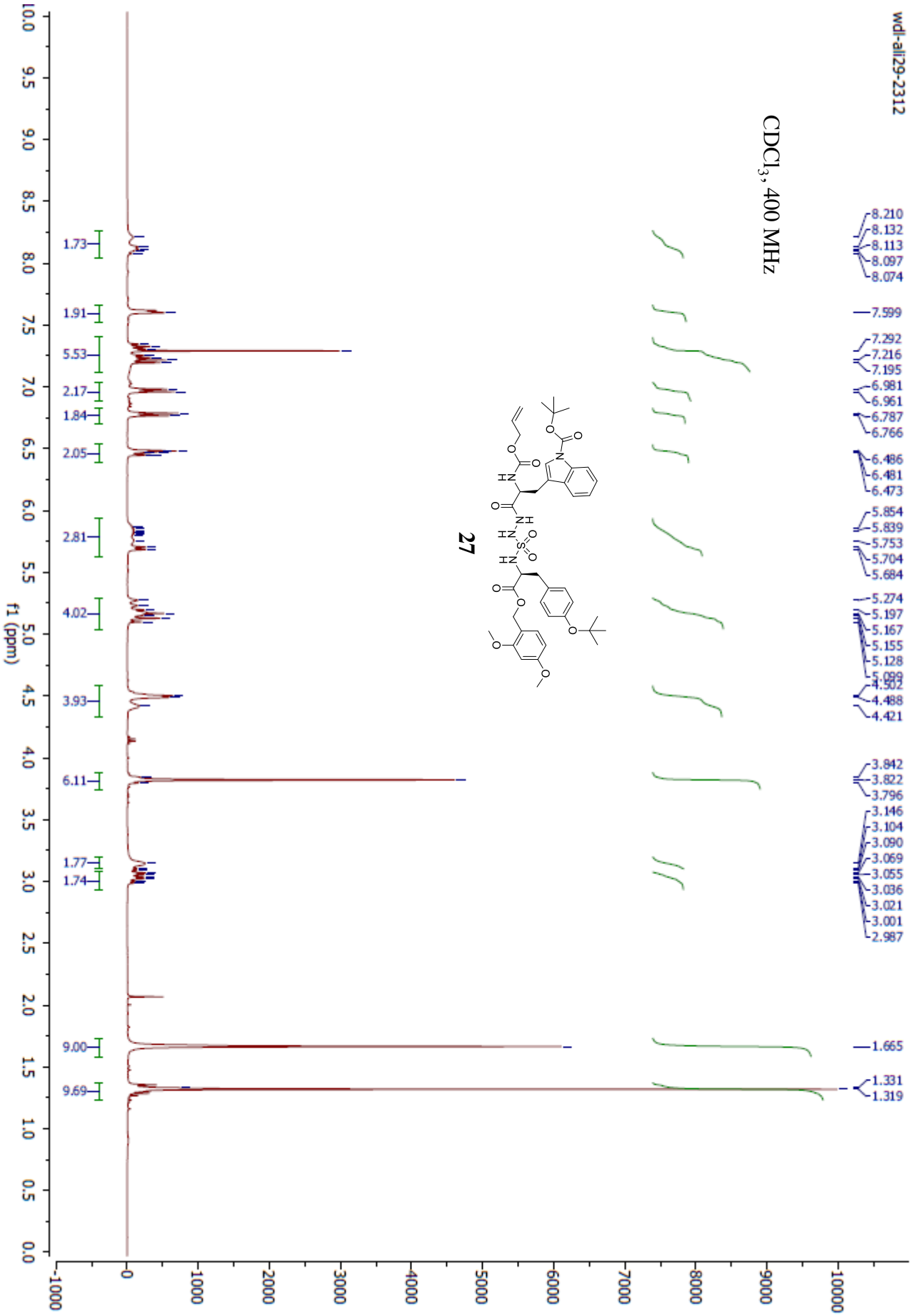
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CDCl3, 400 MHz
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f1 (ppm)
1.73
1.91
5.53
2.17
1.84
2.05
2.81
4.02
3.93
6.11
1.77
1.74
9.00
9.69
8.210
8.132
8.113
8.097
8.074
7.599
7.292
7.216
7.195
6.981
6.961
6.787
6.766
6.486
6.481
6.473
5.854
5.839
5.753
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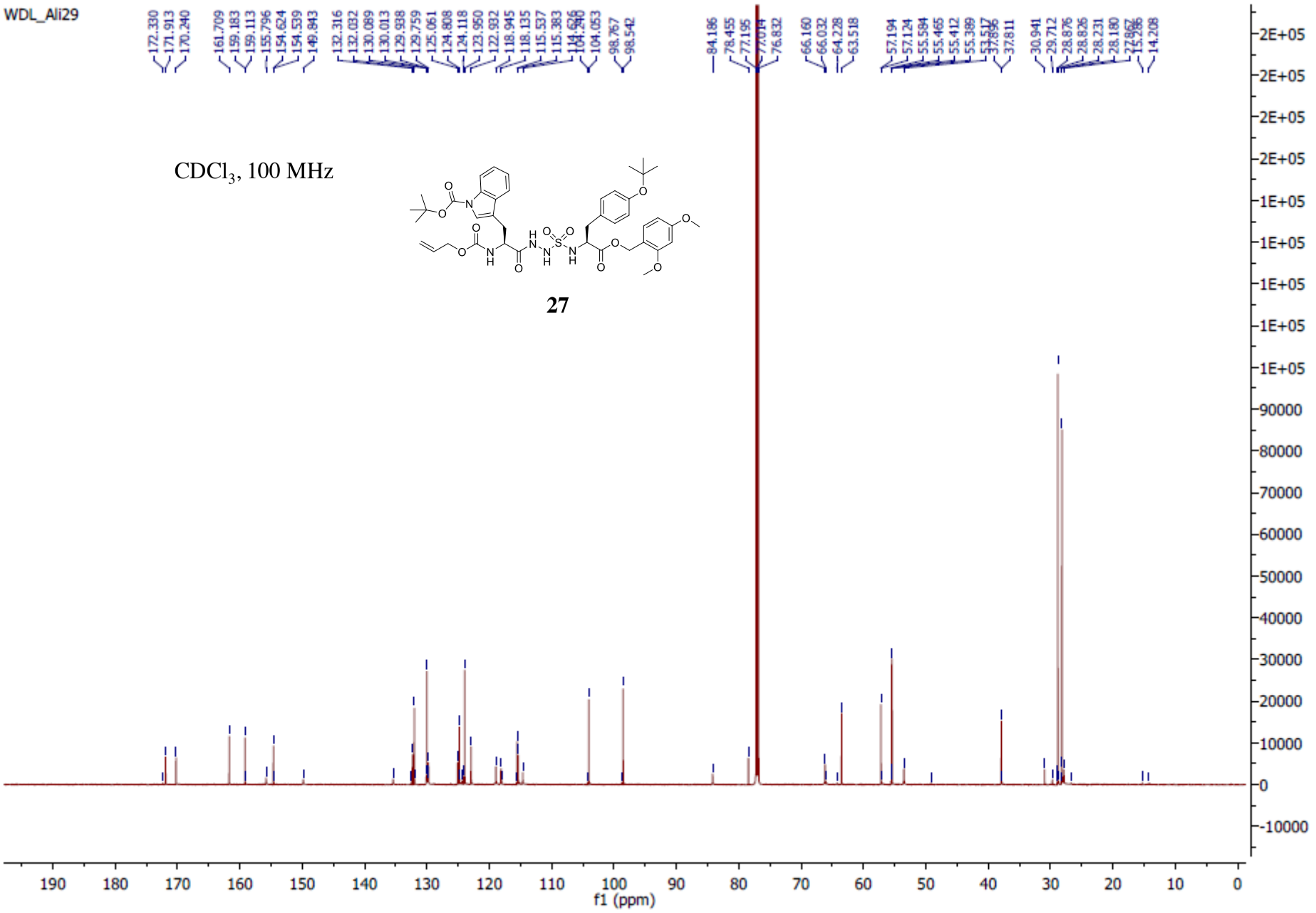
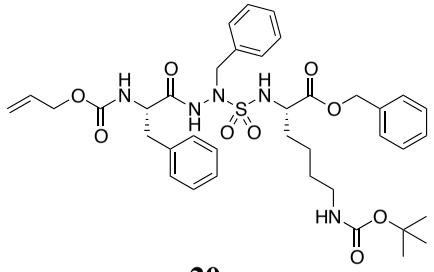
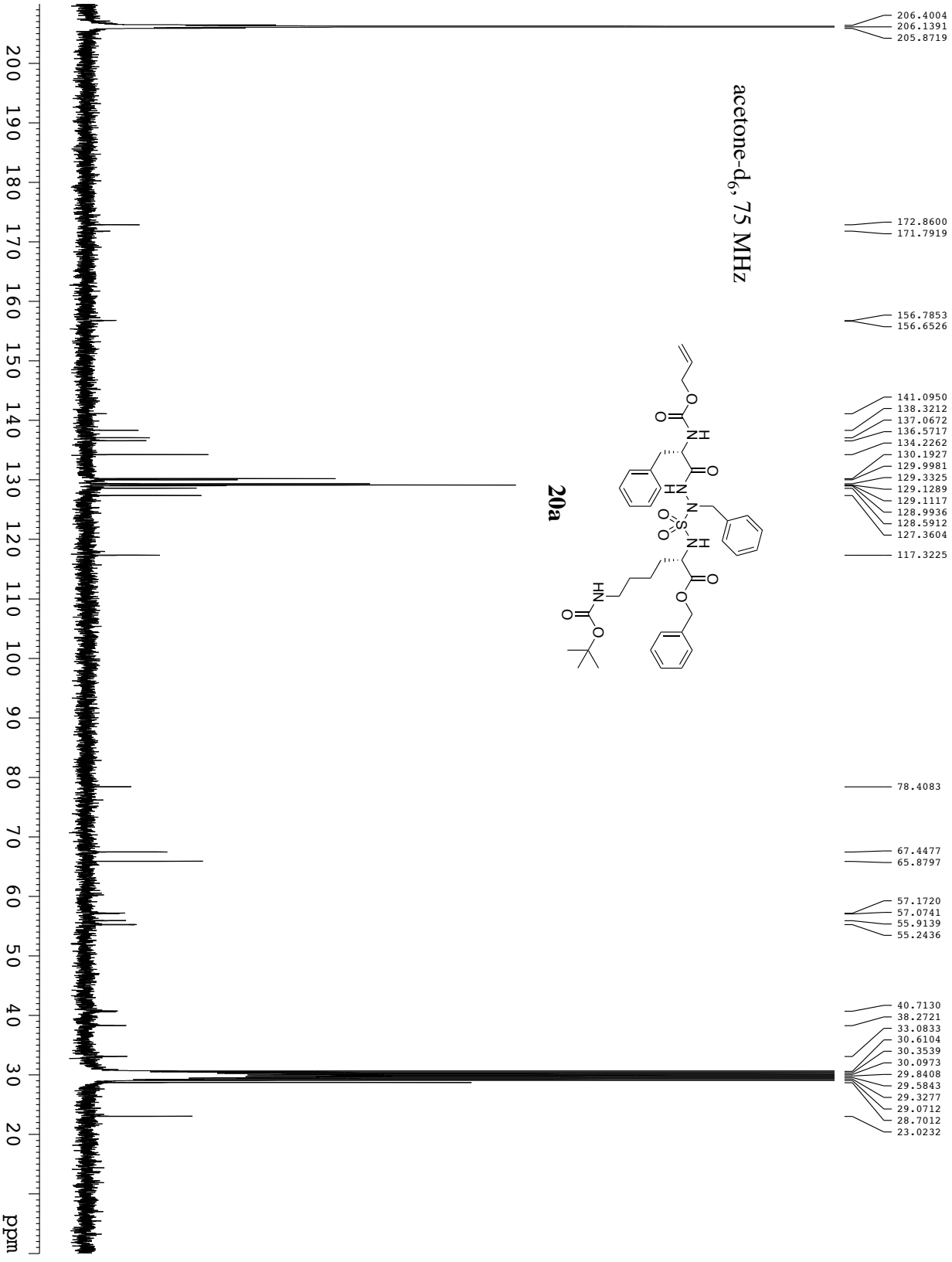
3.146
3.104
3.090
3.069
3.055
3.036
3.021
3.001
2.987
1.665
1.331
1.319

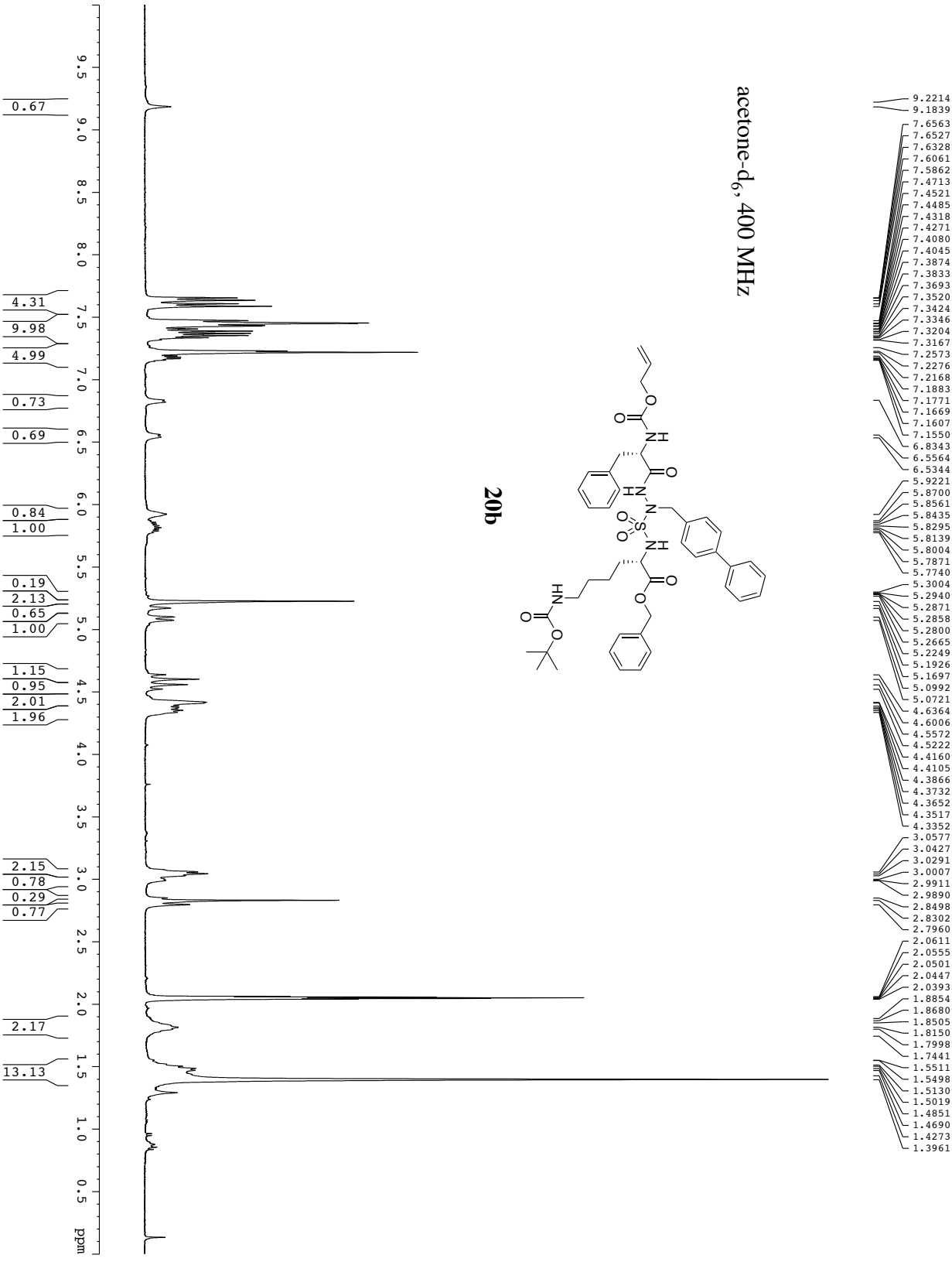

WDL_Ali29
172.330
171.913
170.240
161.709
159.183
159.113
155.796
154.624
154.539
149.843
132.316
132.032
130.089
130.013
129.938
129.759
125.061
124.808
124.118
123.950
122.932
118.945
118.135
115.537
115.383
104.053
98.767
98.542
84.186
78.455
77.195
76.832
66.160
66.032
64.228
63.518
57.194
57.124
55.594
55.465
55.412
55.389
37.811
30.941
29.712
28.876
28.826
28.231
28.180
14.208
CDCl3, 100 MHz
27
2E+05
1E+05
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

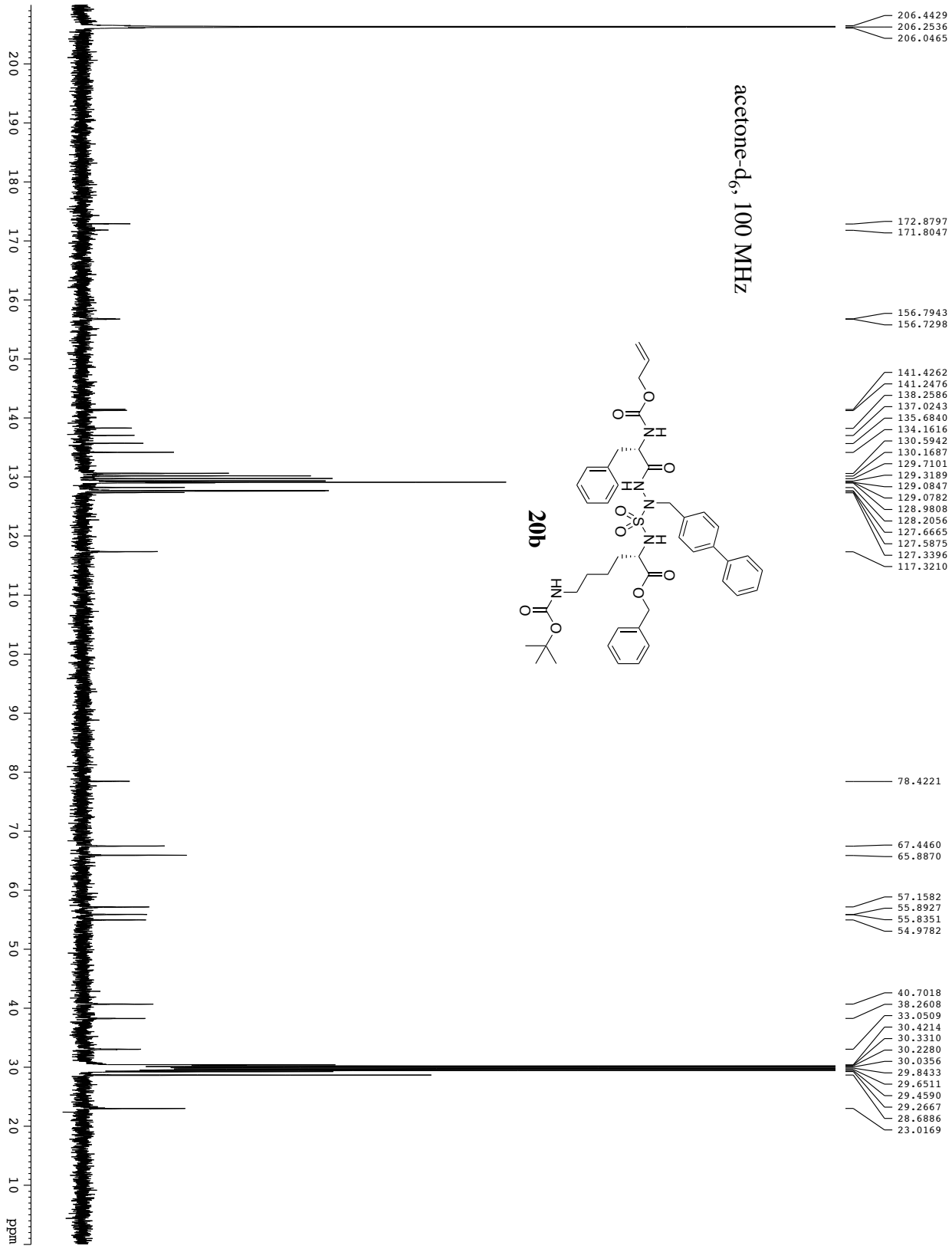
acetone-d_6, 400 MHz

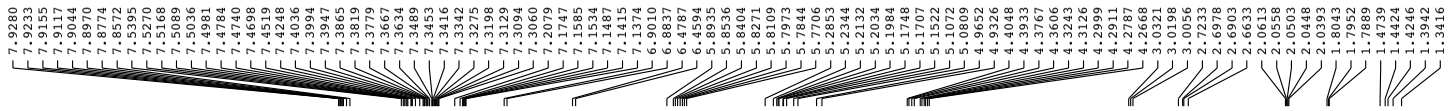


20a

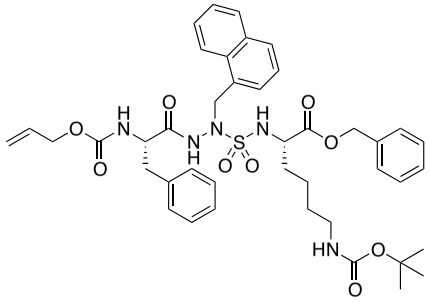

acetone-d6, 75 MHz
20a
206.4004
206.1391
205.8719
172.8600
171.7919
156.7853
156.6526
141.0950
138.3212
137.0672
136.5717
134.2262
130.1927
129.9981
129.3325
129.1289
129.1117
128.9936
128.5912
127.3604
117.3225
78.4083
67.4477
65.8797
57.1720
57.0741
55.9139
55.2436
40.7130
38.2721
33.0833
30.6104
30.3539
30.0973
29.8408
29.5843
29.3277
29.0712
28.7012
23.0232
ppm


acetone-d6, 400 MHz
20b
9.2214
9.1839
7.6563
7.6527
7.6328
7.6061
7.5862
7.4713
7.4521
7.4485
7.4318
7.4271
7.4080
7.4045
7.3874
7.3833
7.3693
7.3520
7.3424
7.3346
7.3204
7.3167
7.2573
7.2276
7.2168
7.1883
7.1771
7.1669
7.1607
7.1550
6.8343
6.5564
6.5344
5.9221
5.8700
5.8561
5.8435
5.8295
5.8139
5.8004
5.7871
5.7740
5.3004
5.2940
5.2871
5.2858
5.2800
5.2665
5.2249
5.1926
5.1697
5.0992
5.0721
4.6364
4.6006
4.5572
4.5222
4.4160
4.4105
4.3866
4.3732
4.3652
4.3517
4.3352
3.0577
3.0427
3.0291
3.0007
2.9911
2.9890
2.8498
2.8302
2.7960
2.0611
2.0555
2.0501
2.0447
2.0393
1.8854
1.8680
1.8505
1.8150
1.7998
1.7441
1.5511
1.5498
1.5130
1.5019
1.4851
1.4690
1.4273
1.3961
0.67
4.31
9.98
4.99
0.73
0.69
0.84
1.00
0.19
2.13
0.65
1.00
1.15
0.95
2.01
1.96
2.15
0.78
0.29
0.77
2.17
13.13
ppm

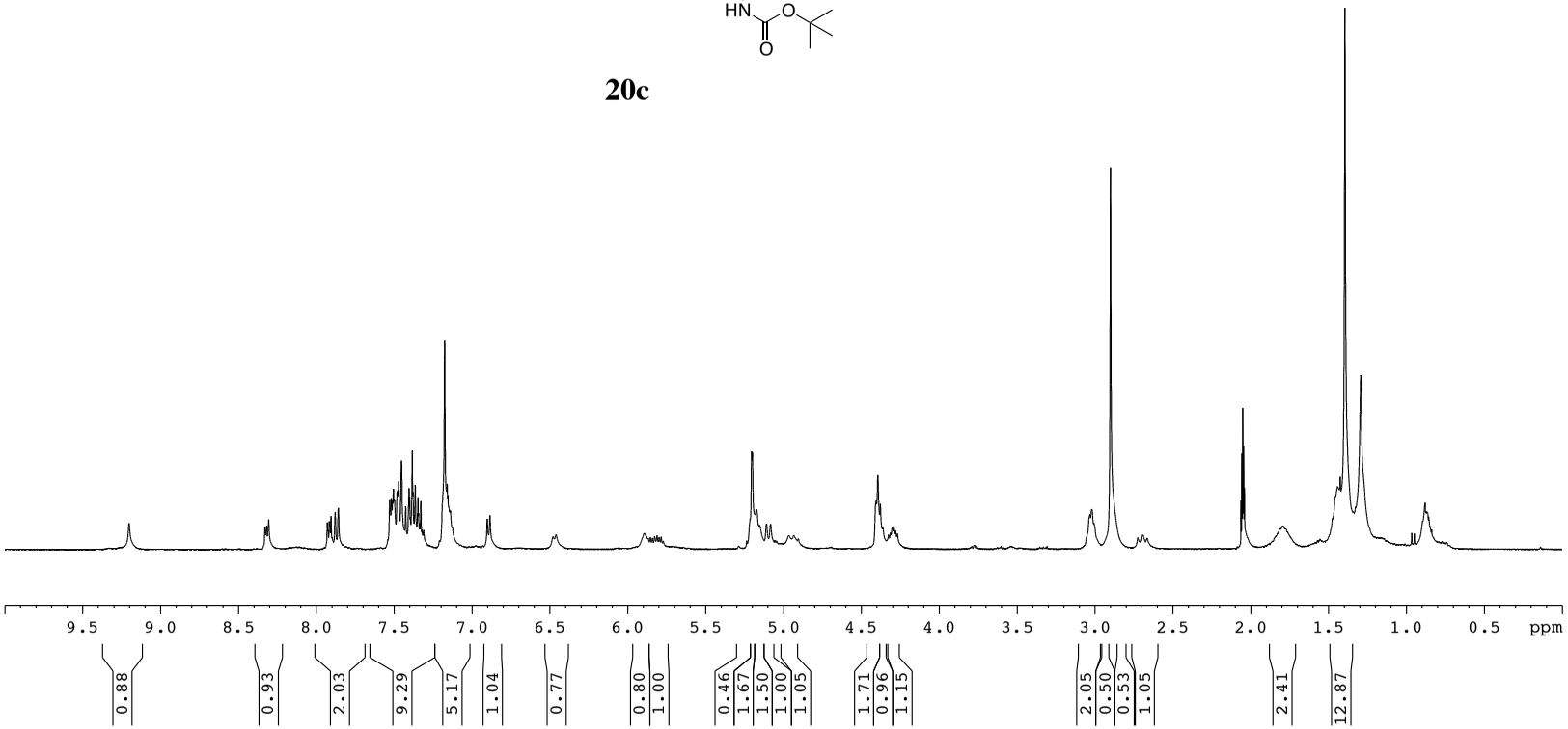

acetone-d6, 100 MHz
20b
206.4429
206.2536
206.0465
172.8797
171.8047
156.7943
156.7298
141.4262
141.2476
138.2586
137.0243
135.6840
134.1616
130.5942
130.1687
129.7101
129.3189
129.0847
129.0782
128.9808
128.2056
127.6665
127.5875
127.3396
117.3210
78.4221
67.4460
65.8870
57.1582
55.8927
55.8351
54.9782
40.7018
38.2608
33.0509
30.4214
30.3310
30.2280
30.0356
29.8433
29.6511
29.4590
29.2667
28.6886
23.0169
ppm

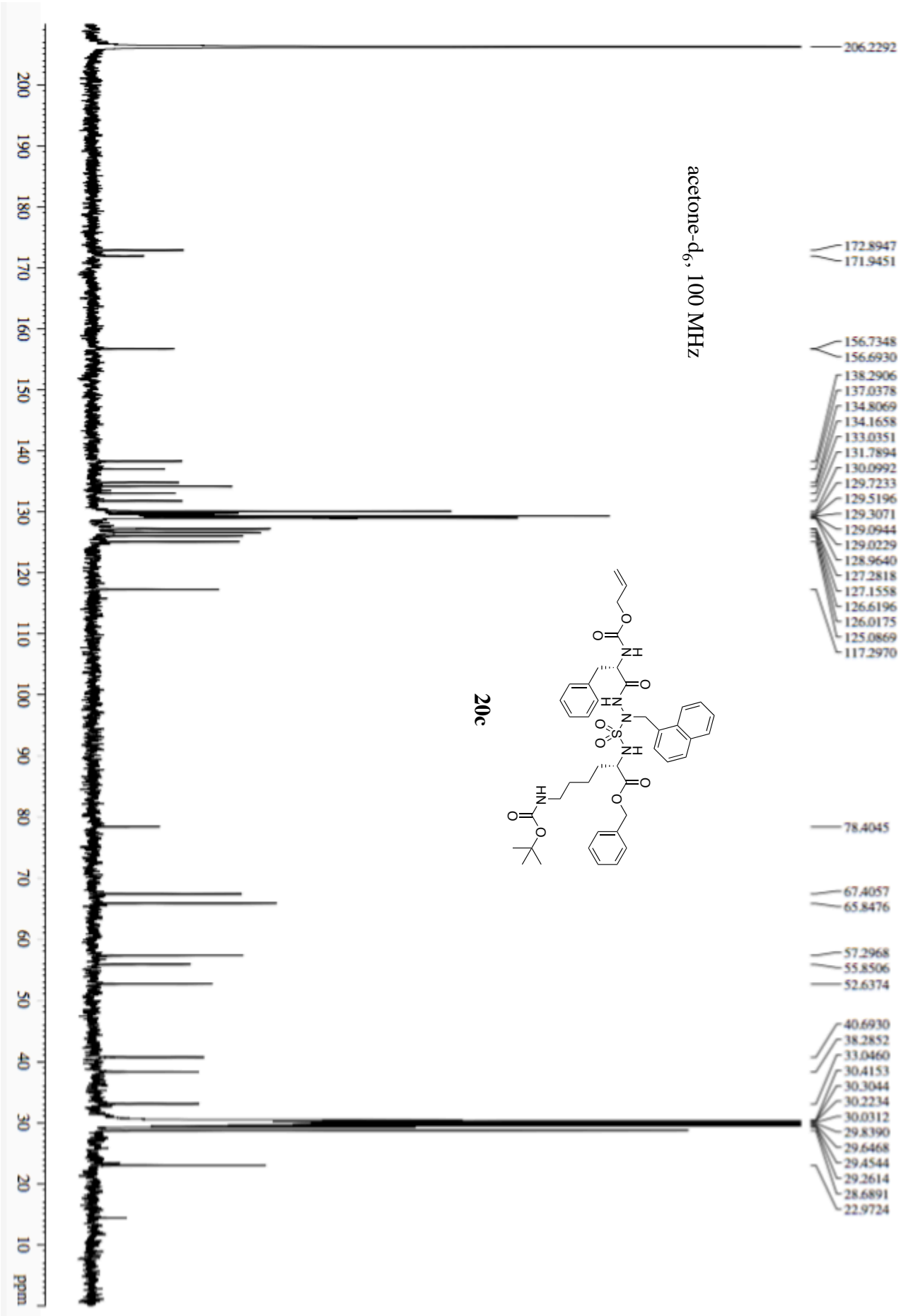


acetone-d_6, 400 MHz

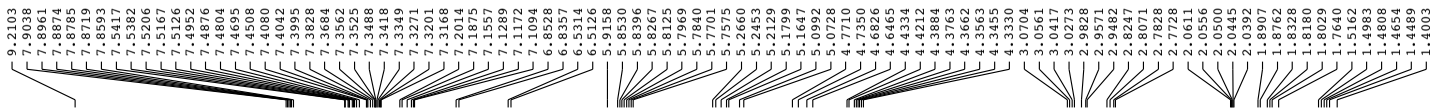


20c

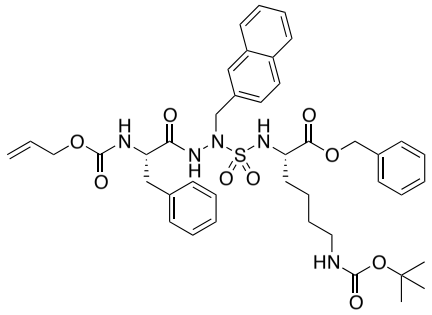



acetone-d6, 100 MHz
206.2292
172.8947
171.9451
156.7348
156.6930
138.2906
137.0378
134.8069
134.1658
133.0351
131.7894
130.0992
129.7233
129.5196
129.3071
129.0944
129.0229
128.9640
127.2818
127.1558
126.6196
126.0175
125.0869
117.2970
78.4045
67.4057
65.8476
57.2968
55.8506
52.6374
40.6930
38.2852
33.0460
30.4153
30.3044
30.2234
30.0312
29.8390
29.6468
29.4544
29.2614
28.6891
22.9724
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

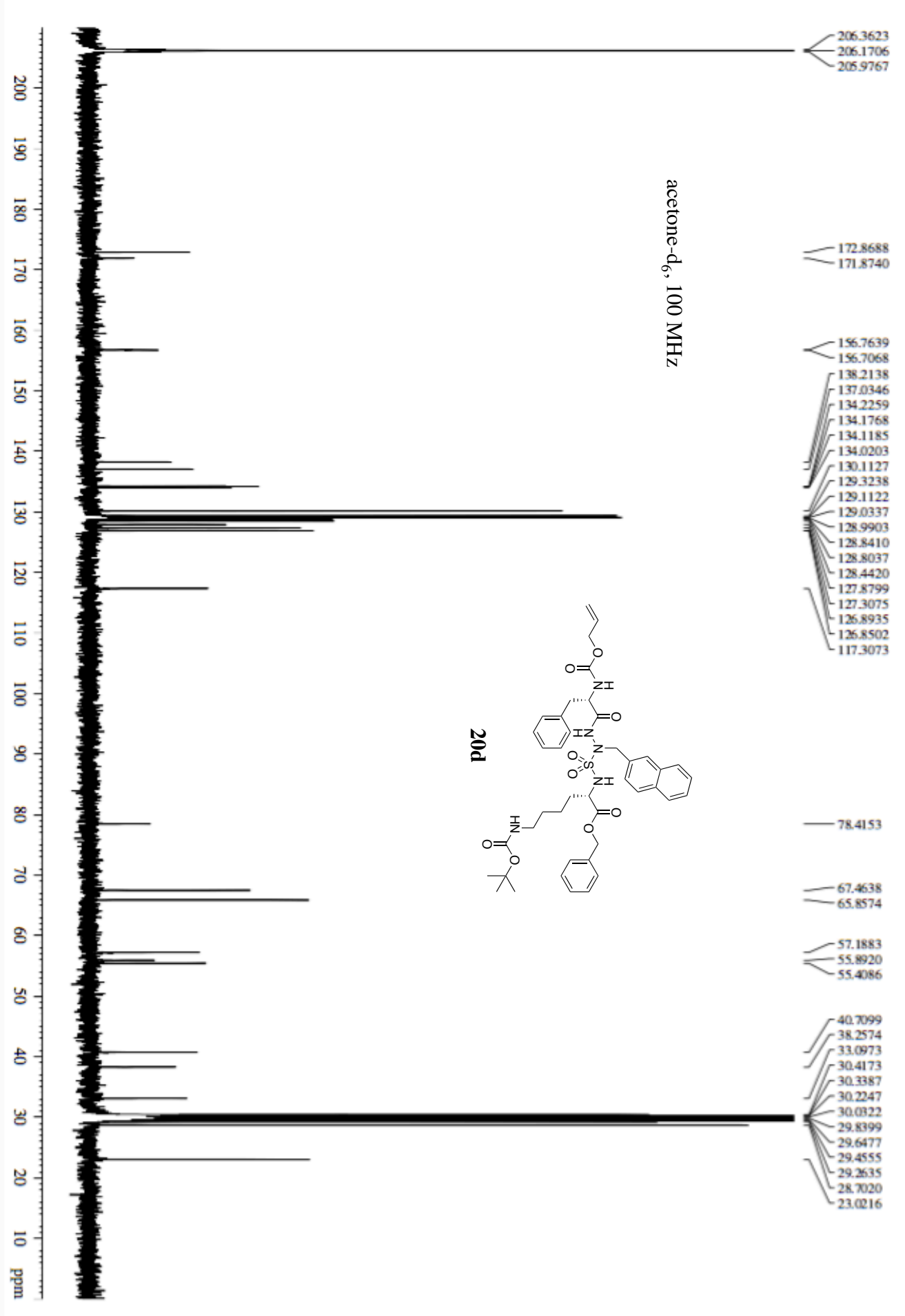
20c

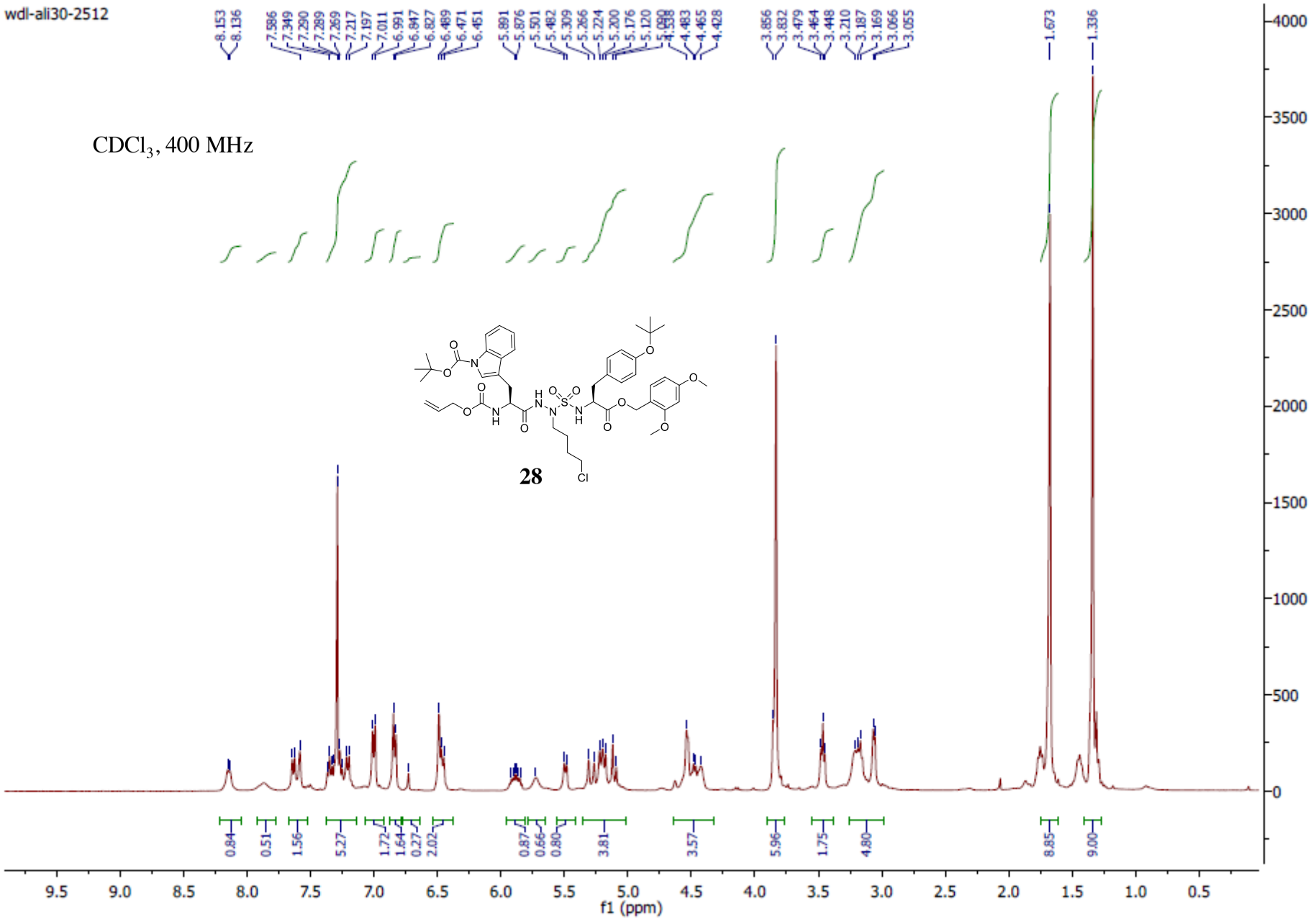


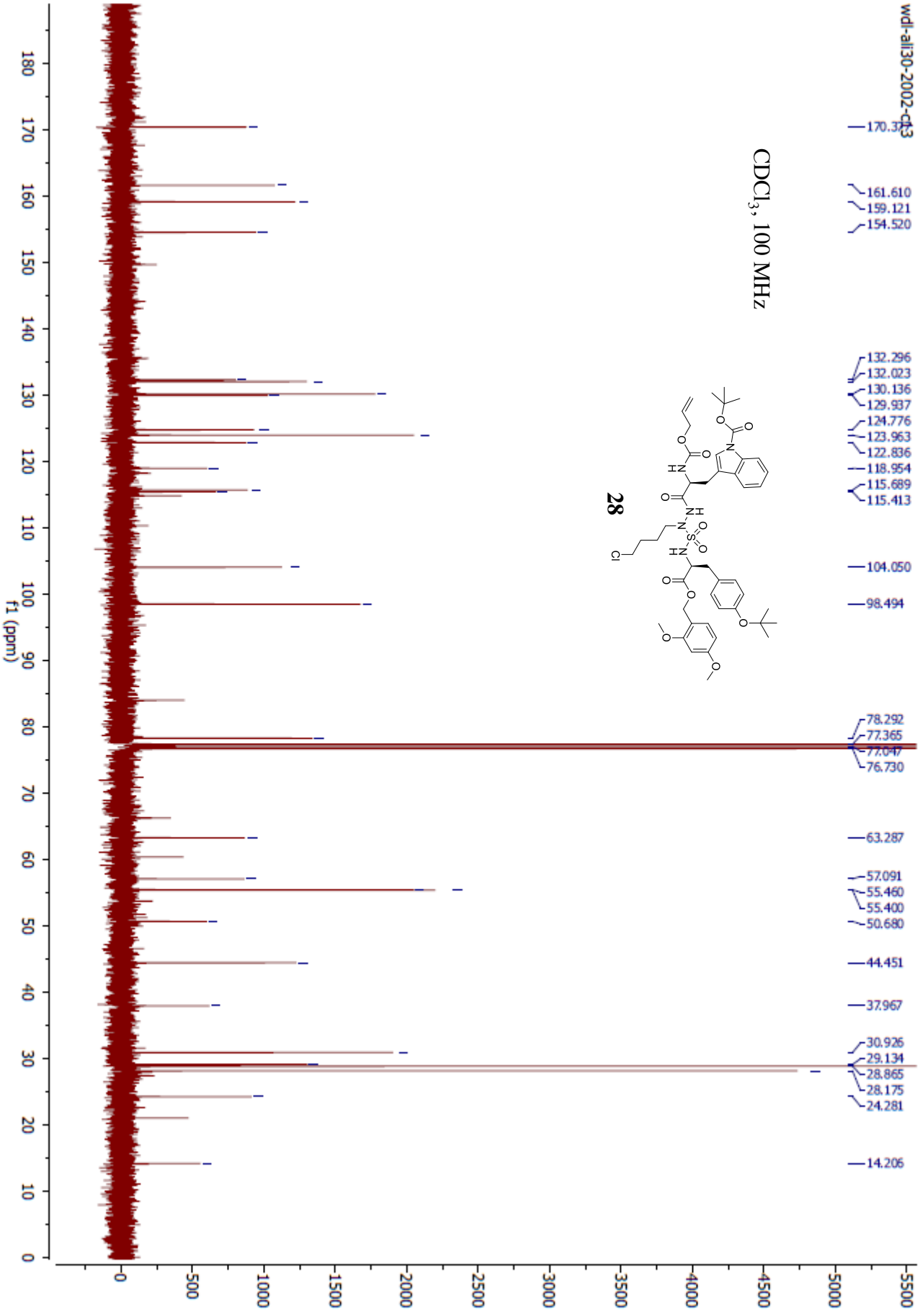
acetone-d_6, 400 MHz

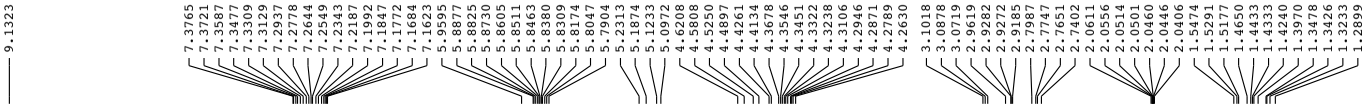


20d

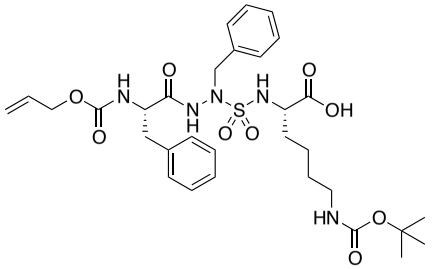

acetone-d6, 100 MHz
20d
206.3623
206.1706
205.9767
172.8688
171.8740
156.7639
156.7068
138.2138
137.0346
134.2259
134.1768
134.1185
134.0203
130.1127
129.3238
129.1122
129.0337
128.9903
128.8410
128.8037
128.4420
127.8799
127.3075
126.8935
126.8502
117.3073
78.4153
67.4638
65.8574
57.1883
55.8920
55.4086
40.7099
38.2574
33.0973
30.4173
30.3387
30.2247
30.0322
29.8399
29.6477
29.4555
29.2635
28.7020
23.0216
ppm


wdl-ali30-2512
8.153
8.136
7.586
7.349
7.290
7.289
7.269
7.217
7.197
7.011
6.991
6.847
6.827
6.489
6.471
6.451
5.891
5.876
5.501
5.482
5.309
5.266
5.224
5.200
5.176
5.120
4.538
4.483
4.465
4.428
3.856
3.832
3.479
3.464
3.448
3.210
3.187
3.169
3.066
3.055
1.673
1.336
CDCl3, 400 MHz
28
0.84
0.51
1.56
5.27
1.72
1.64
0.27
2.02
0.87
0.66
0.80
3.81
3.57
5.96
1.75
4.80
8.85
9.00
f1 (ppm)

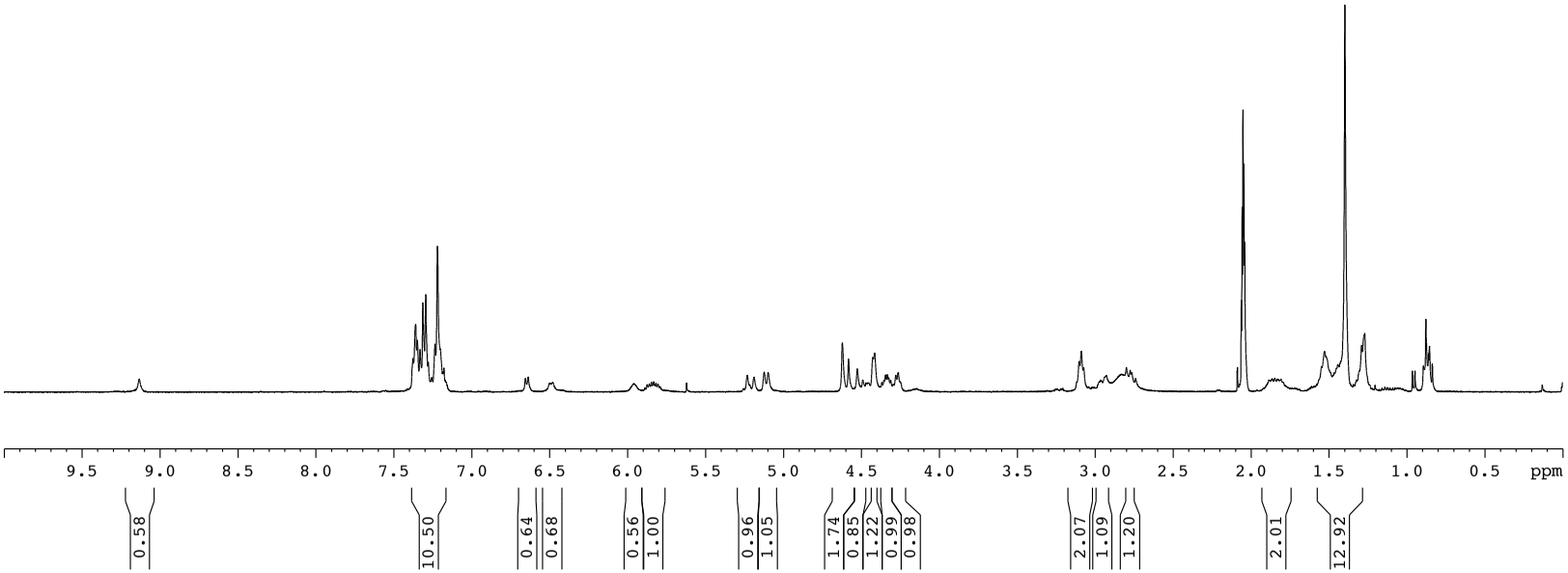

wdl-ali30-2002-cdcl3
CDCl3, 100 MHz
28
170.333
161.610
159.121
154.520
132.296
132.023
130.136
129.937
124.776
123.963
122.836
118.954
115.689
115.413
104.050
98.494
78.292
77.365
77.047
76.730
63.287
57.091
55.460
55.400
50.680
44.451
37.967
30.926
29.134
28.865
28.175
24.281
14.206
f1 (ppm)

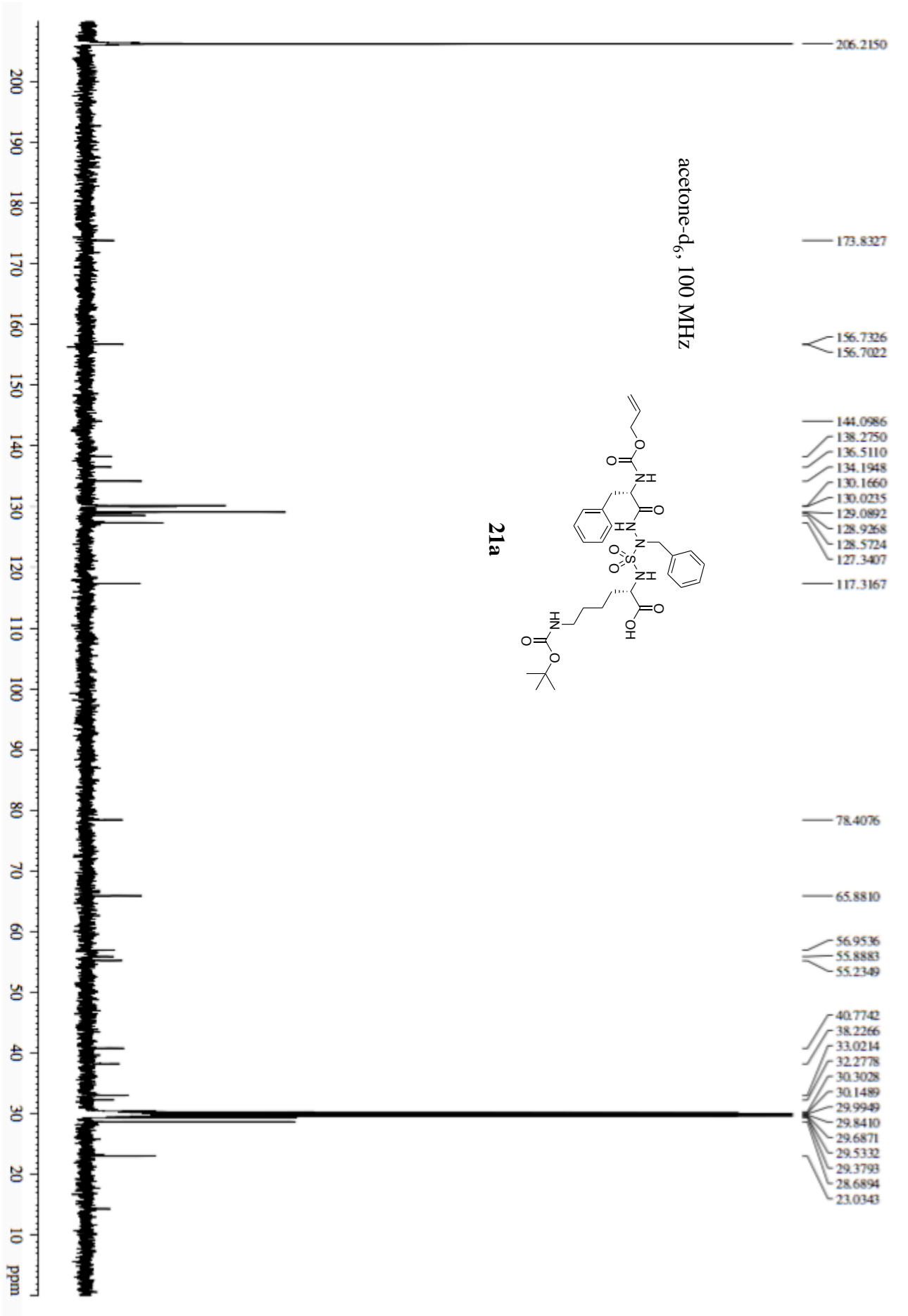


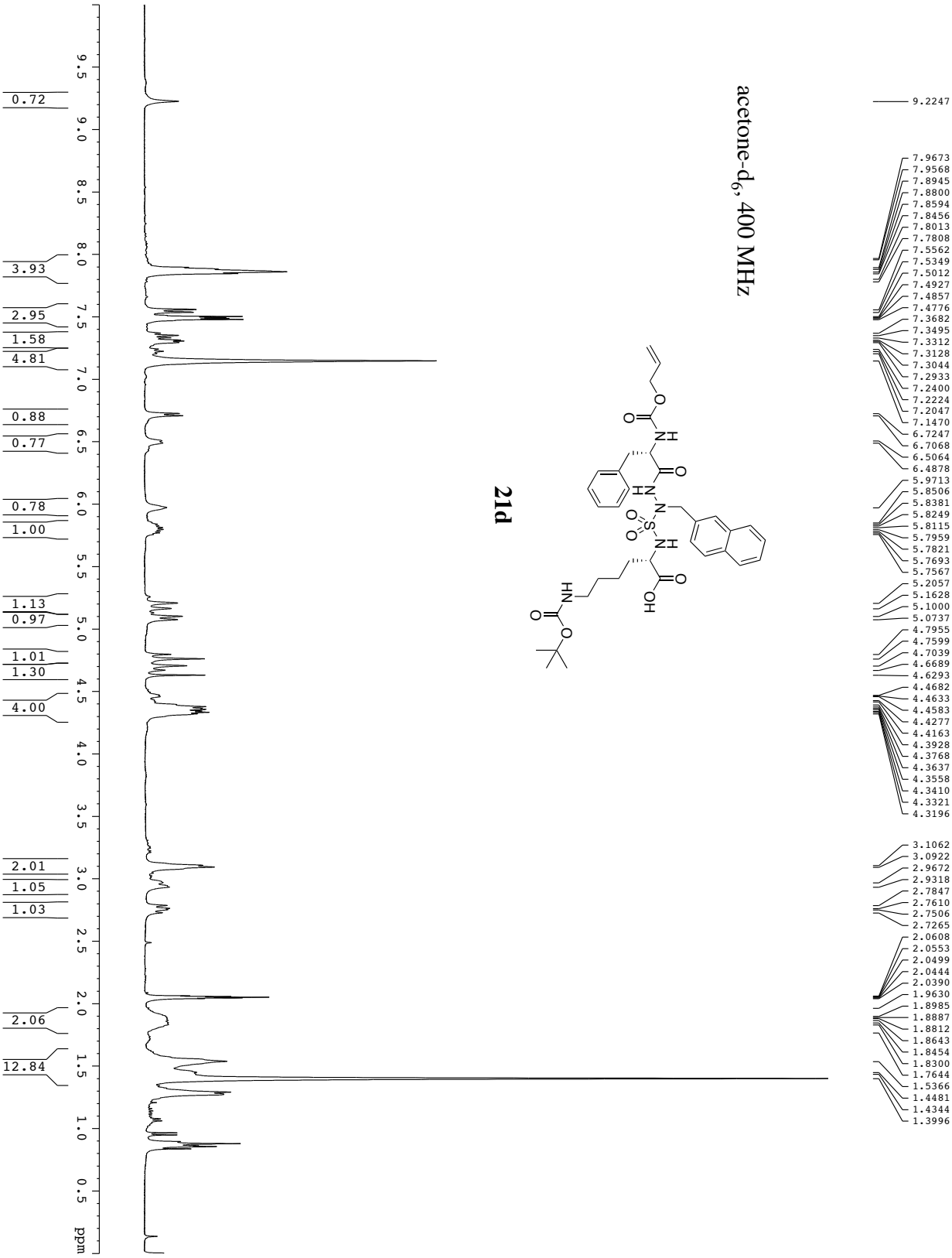
acetone-d_6, 400 MHz

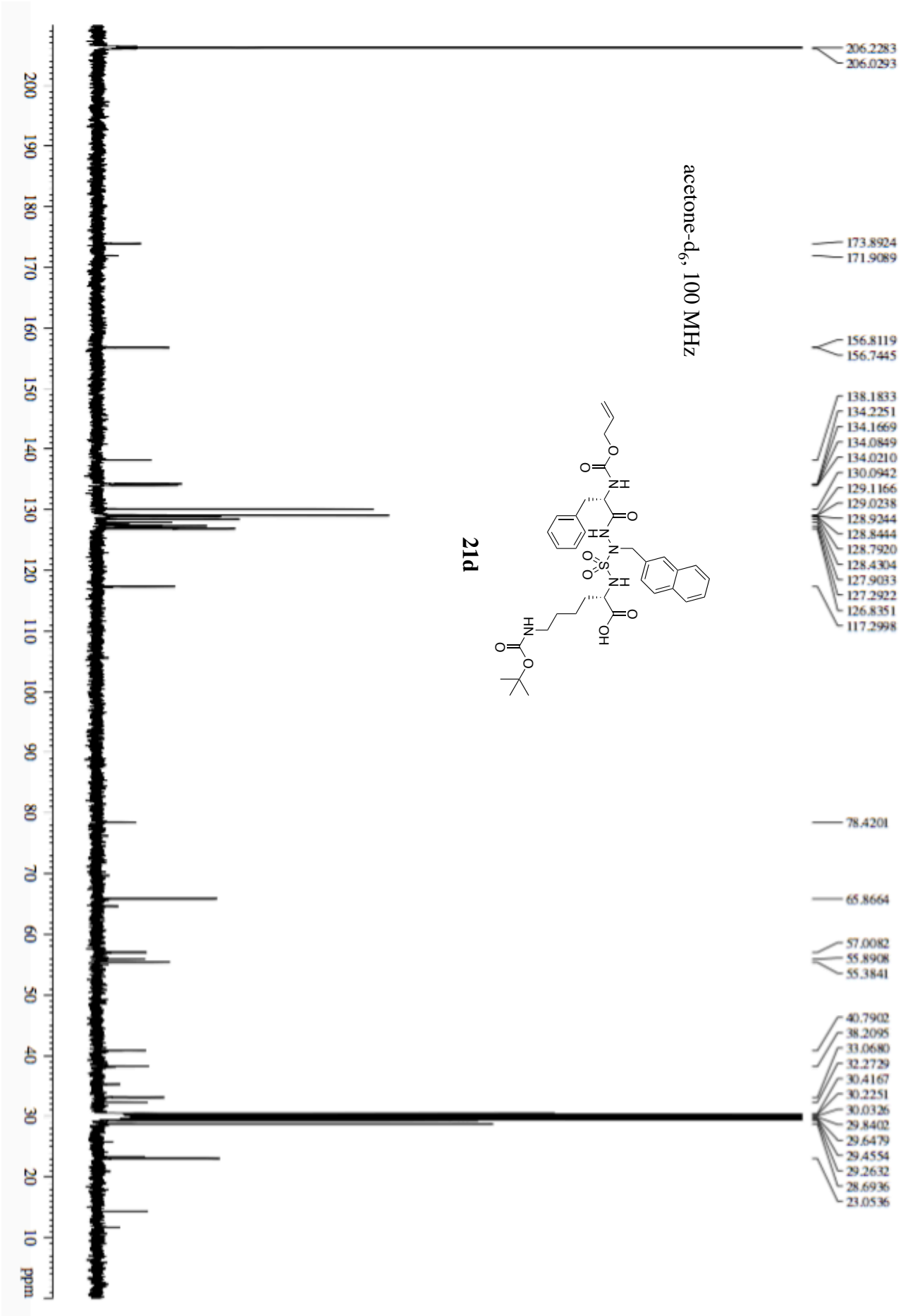


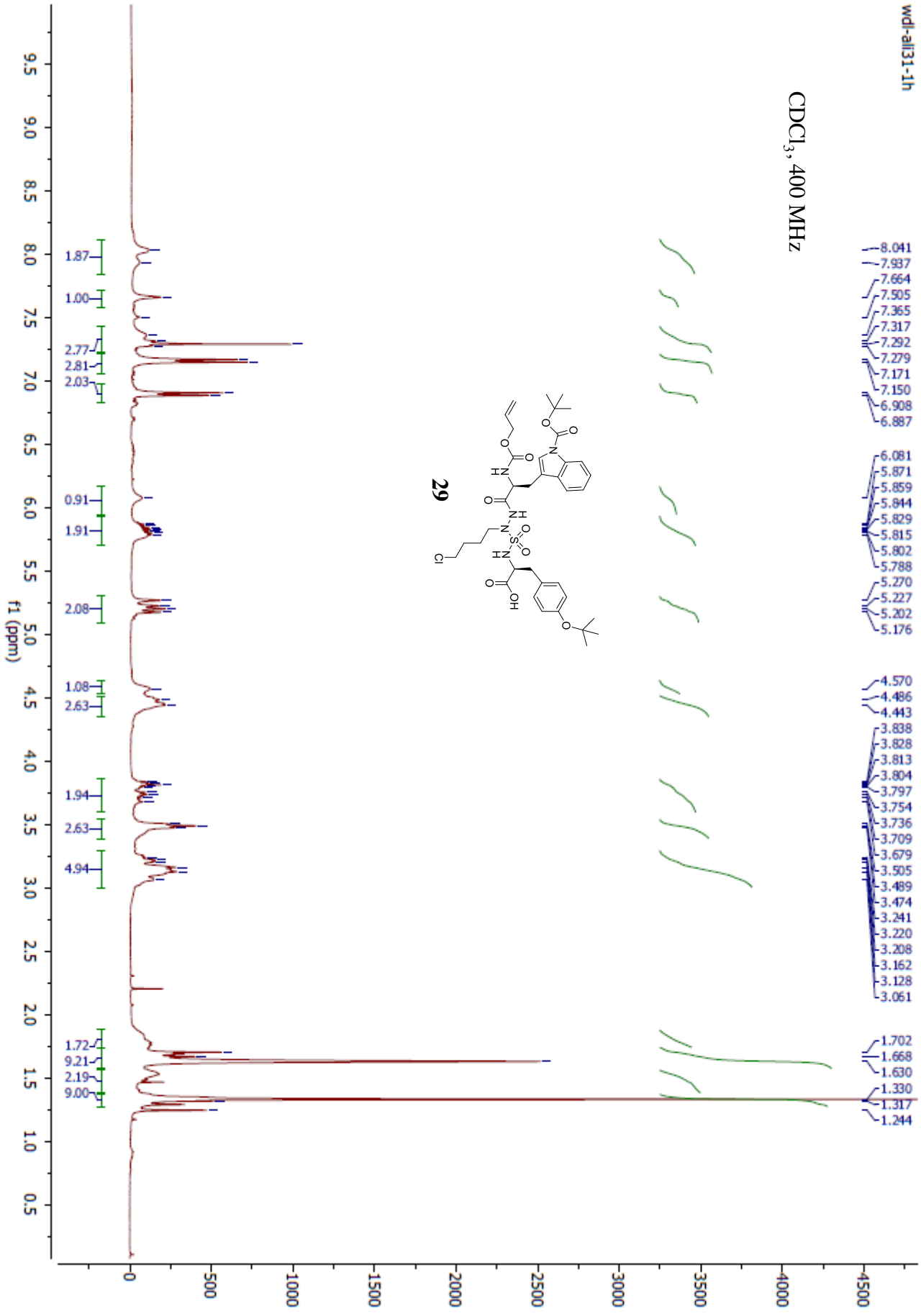
21a

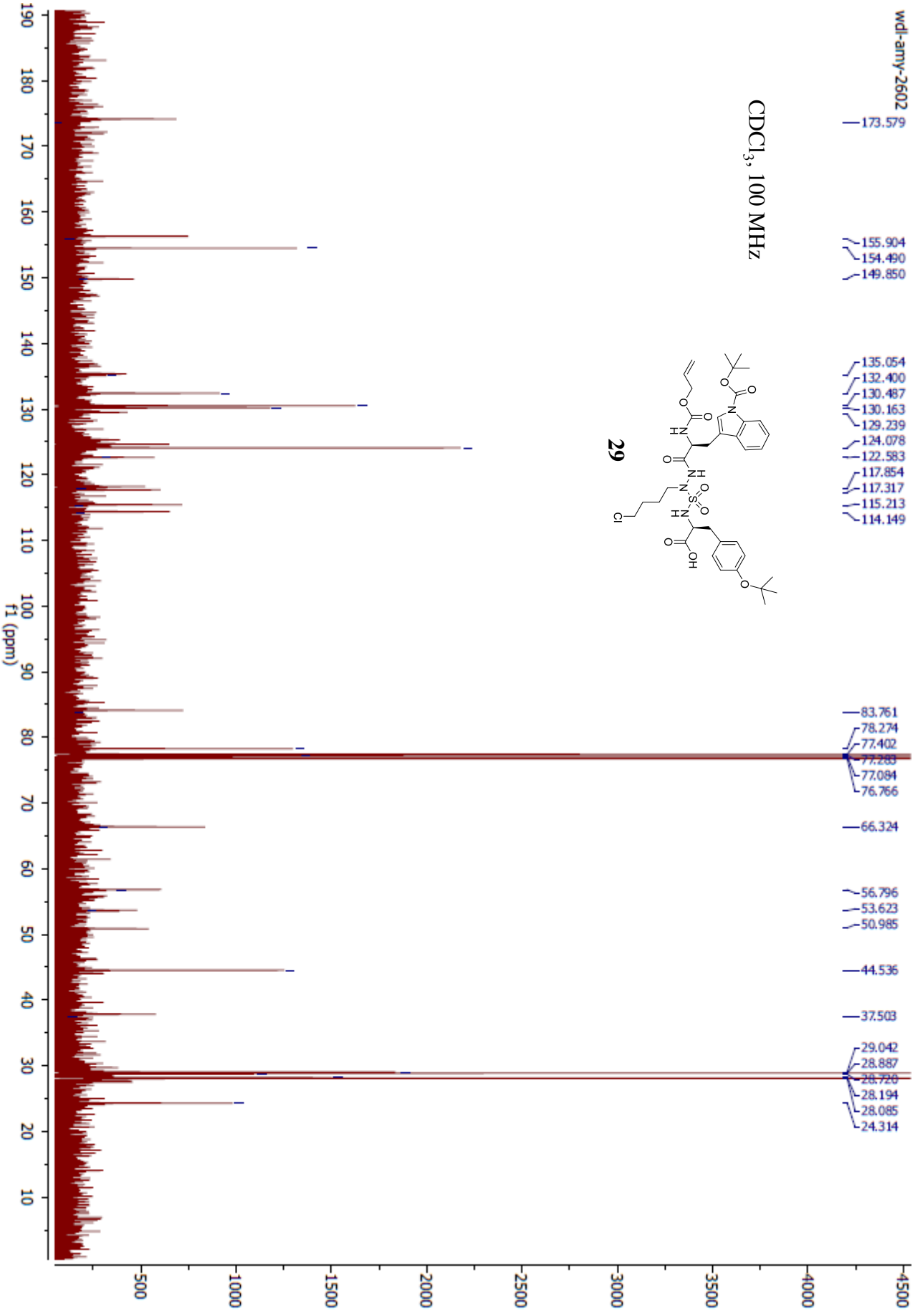



acetone-d6, 100 MHz
21a
206.2150
173.8327
156.7326
156.7022
144.0986
138.2750
136.5110
134.1948
130.1660
130.0235
129.0892
128.9268
128.5724
127.3407
117.3167
78.4076
65.8810
56.9536
55.8883
55.2349
40.7742
38.2266
33.0214
32.2778
30.3028
30.1489
29.9949
29.8410
29.6871
29.5332
29.3793
28.6894
23.0343
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


acetone-d6, 400 MHz
21d
9.2247
7.9673
7.9568
7.8945
7.8800
7.8594
7.8456
7.8013
7.7808
7.5562
7.5349
7.5012
7.4927
7.4857
7.4776
7.3682
7.3495
7.3312
7.3128
7.3044
7.2933
7.2400
7.2224
7.2047
7.1470
6.7247
6.7068
6.5064
6.4878
5.9713
5.8506
5.8381
5.8249
5.8115
5.7959
5.7821
5.7693
5.7567
5.2057
5.1628
5.1000
5.0737
4.7955
4.7599
4.7039
4.6689
4.6293
4.4682
4.4633
4.4583
4.4277
4.4163
4.3928
4.3768
4.3637
4.3558
4.3410
4.3321
4.3196
3.1062
3.0922
2.9672
2.9318
2.7847
2.7610
2.7506
2.7265
2.0608
2.0553
2.0499
2.0444
2.0390
1.9630
1.8985
1.8887
1.8812
1.8643
1.8454
1.8300
1.7644
1.5366
1.4481
1.4344
1.3996
0.72
3.93
2.95
1.58
4.81
0.88
0.77
0.78
1.00
1.13
0.97
1.01
1.30
4.00
2.01
1.05
1.03
2.06
12.84
ppm


acetone-d6, 100 MHz
21d
206.2283
206.0293
173.8924
171.9089
156.8119
156.7445
138.1833
134.2251
134.1669
134.0849
134.0210
130.0942
129.1166
129.0238
128.9244
128.8444
128.7920
128.4304
127.9033
127.2922
126.8351
117.2998
78.4201
65.8664
57.0082
55.8908
55.3841
40.7902
38.2095
33.0680
32.2729
30.4167
30.2251
30.0326
29.8402
29.6479
29.4554
29.2632
28.6936
23.0536
ppm


wdl-all31-1h
CDCl3, 400 MHz
29
f1 (ppm)


wdl-amy-2602
CDCl3, 100 MHz
29
173.579
155.904
154.490
149.850
135.054
132.400
130.487
130.163
129.239
124.078
122.583
117.854
117.317
115.213
114.149
83.761
78.274
77.402
77.283
77.084
76.766
66.324
56.796
53.623
50.985
44.536
37.503
29.042
28.887
28.720
28.194
28.085
24.314
f1 (ppm)

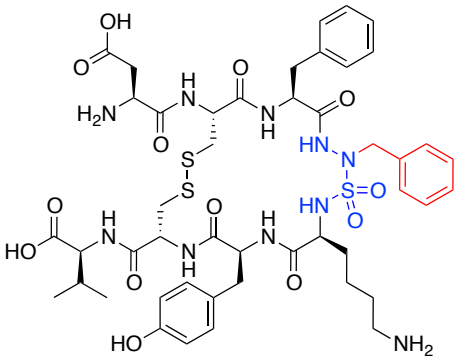
Characterization of azasulfuryl peptides mimics of UII$^{(4\text{-}11)}$

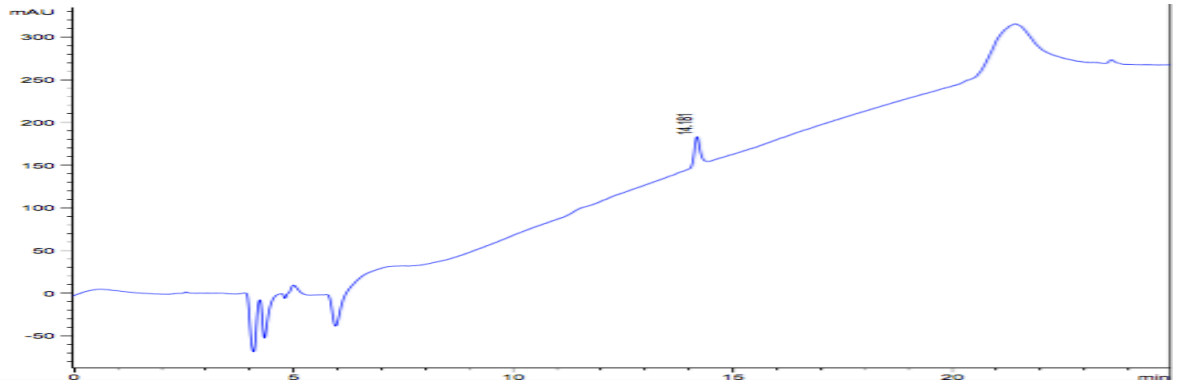
Table S1. Characterization of synthesized peptides **6-11**.

Peptide	% crude purity[a]	% isolated purity[a] (yield[b])	HRMS *m*/*z* calcd (*m*/*z* observed)
[AsF^{7}]UII$^{(4\text{-}11)}$ **(6)**	43	>99 (1%)	1059.3733 (1059.3706)
[AsBip^{7}]UII$^{(4\text{-}11)}$ **(7)**	78	>99 (2%)	1135.4046 (1135.4064)
[AsNal(1')^{7}]UII$^{(4\text{-}11)}$ **(8)**	58	>99 (4%)	1109.3889 (1109.3879)
[AsNal(2')^{7}]UII$^{(4\text{-}11)}$ **(9)**	80	>99 (9%)	1109.3889 (1109.3887)
[AsK^{8}]UII$^{(4\text{-}11)}$ **(10)**	51	>99 (1%)	550.6172[c] (550.6153)
[AsK(N,N^{ε}-Me$_2$)^{8}]UII$^{(4\text{-}11)}$ **(11)**	44	>99 (2%)	564.7192[c] (564.7173)

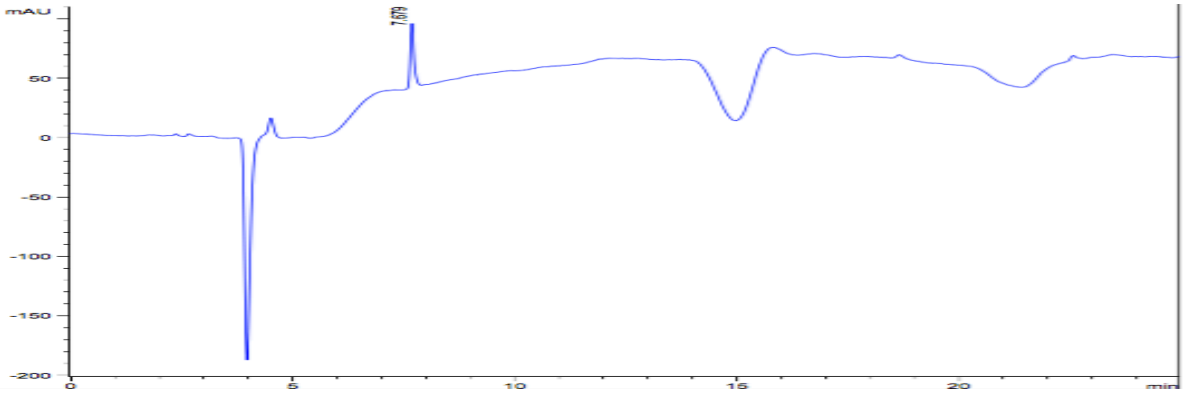
[a]Crude and isolated purity were assessed by LC-MS analysis at 214 nm and 254 nm using H_2O (0.1% formic acic)/MeOH (0.1% formic acid) and H_2O (0.1% formic acid)/MeCN (0.1% formic acid) as eluents. [b]Isolated yields calculated from resin loading. [c]HRMS indicate $[M+2H]^{2+}$.

Peptide [AsF^{7}]UII$^{4-11}$ **(6)**



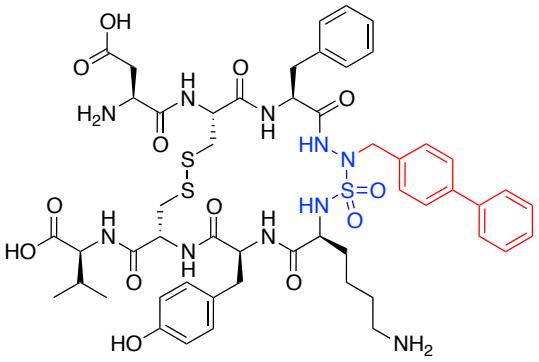


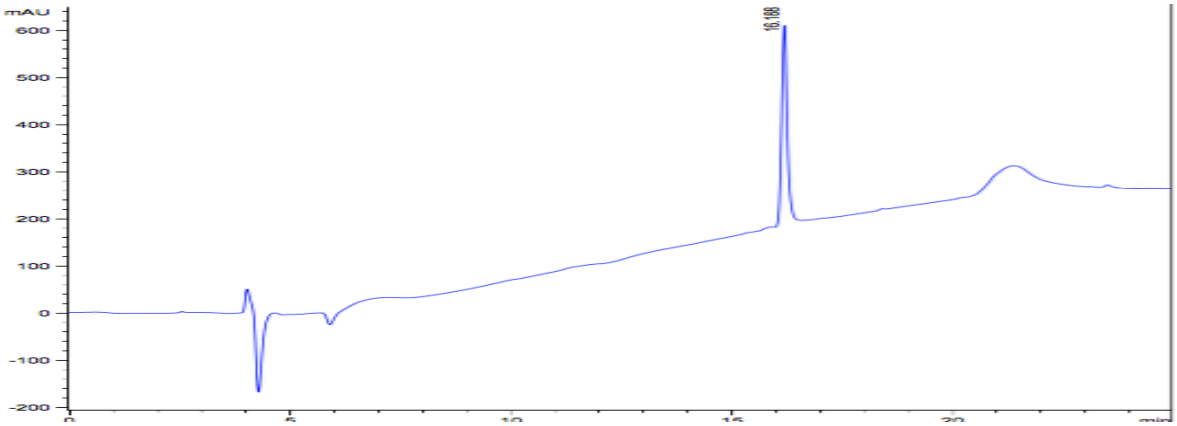
Purity check/Gradient #1 - purity: >99%, t_R: 14.18 min [analytical HPLC, 20 to 80% methanol in water (0.1% formic acid) over 15 min + 90% methanol in water (0.1% formic acid) over 5 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).



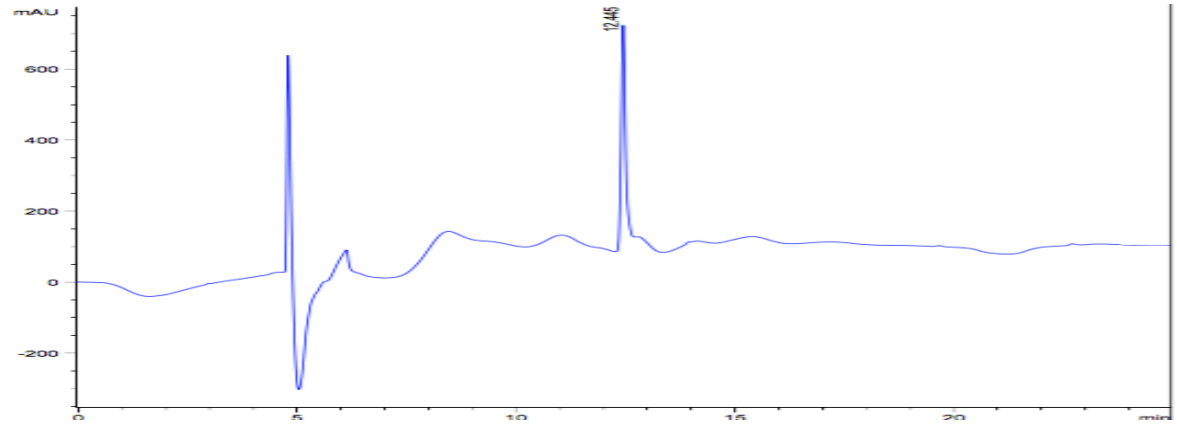
Purity check/Gradient #2 - purity: >99%, t_R: 7.68 min [analytical HPLC, 20 to 80% acetonitrile in water (0.1% formic acid) over 15 min + 90% acetonitrile in water (0.1% formic acid) over 5 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).

Peptide [AsBip^{7}]UII$^{(4\text{-}11)}$ **(7)**



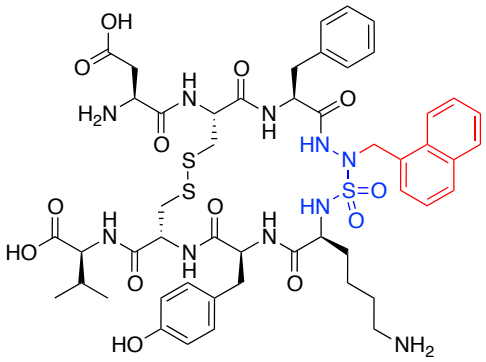


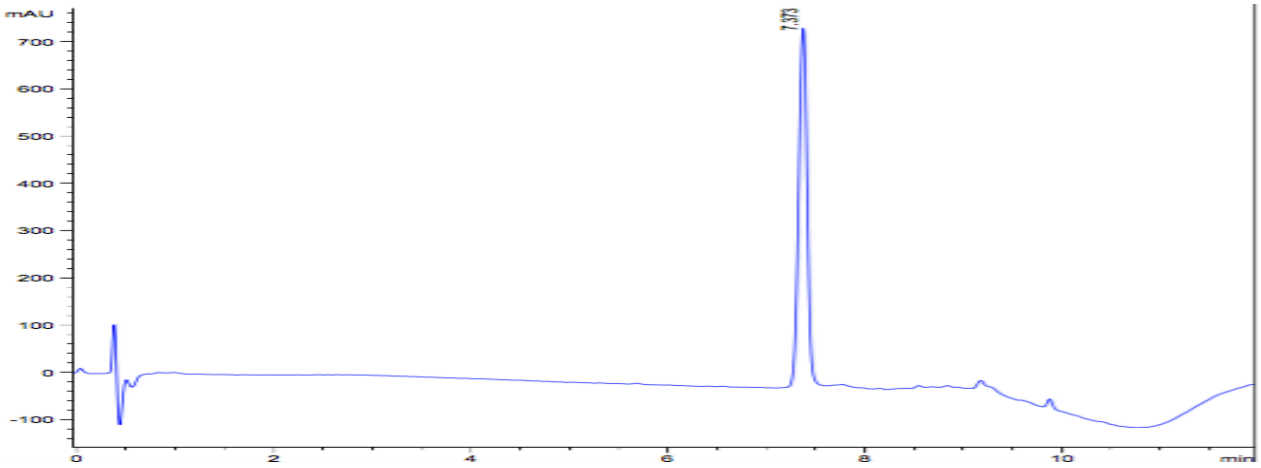
Purity check: Gradient #1 - purity: >99%, t_R: 16.19 min [analytical HPLC, 20 to 80% methanol in water (0.1% formic acid) over 15 min + 90% methanol in water (0.1% formic acid) over 5 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).



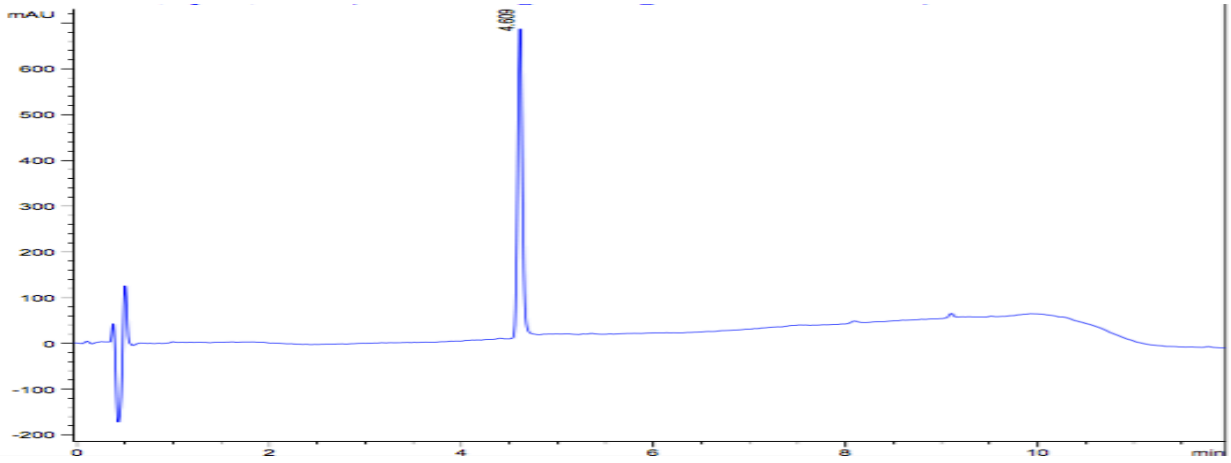
Purity check/Gradient #2 - purity: >99%, t_R: 12.44 min [analytical HPLC, 20 to 80% acetonitrile in water (0.1% formic acid) over 15 min + 90% acetonitrile in water (0.1% formic acid) over 5 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).

Peptide [As(1')Nal^{7}]UII$^{(4-11)}$ **(8)**



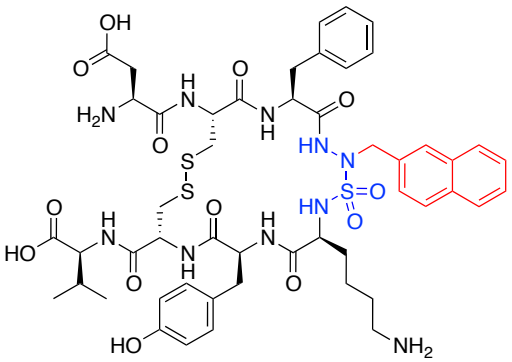


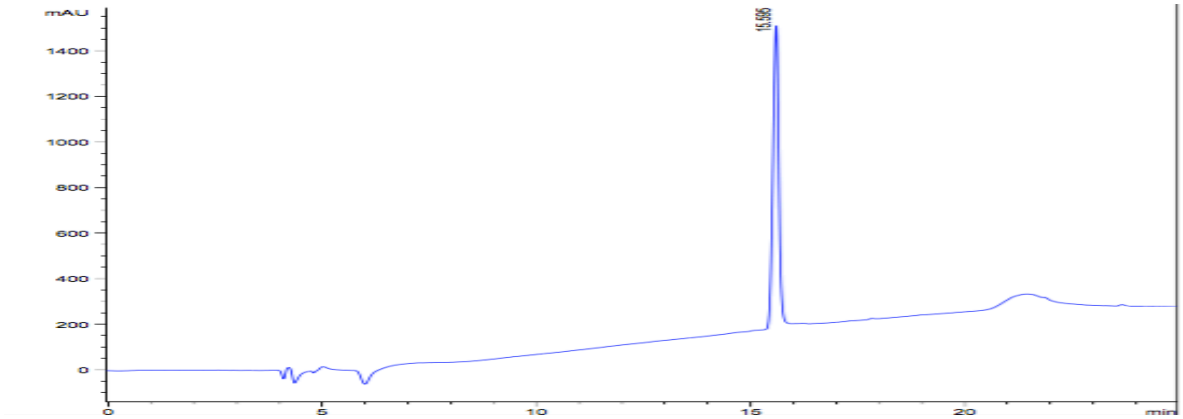
Purity check: Gradient #1 - purity: >99%; t_R: 7.37 min [analytical HPLC, 20 to 60% methanol in water (0.1% formic acid) over 6 min + 90% methanol in water (0.1% formic acid) over 2 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).



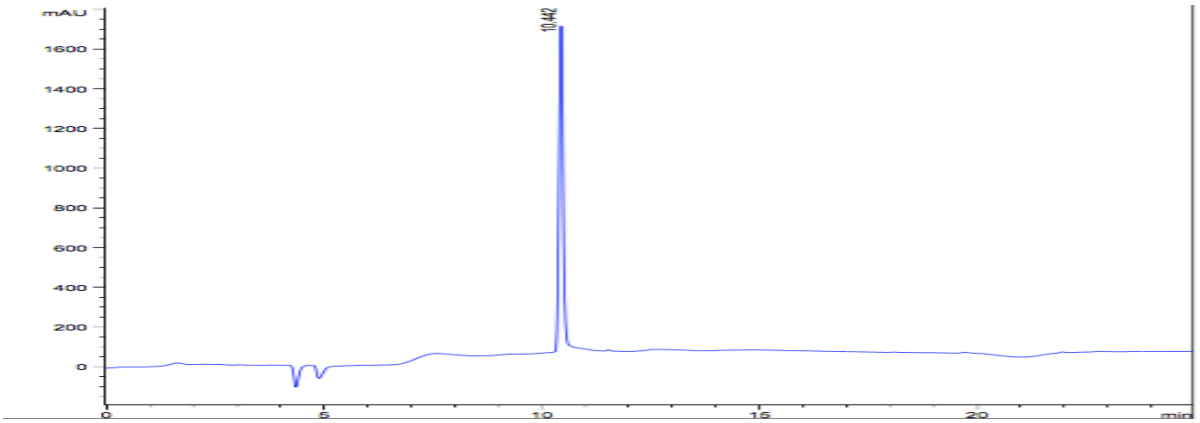
Purity check: Gradient #2 - purity: >99%, t_R: 4.61 min [analytical HPLC, 20 to 60% acetonitrile in water (0.1% formic acid) over 6 min + 90% acetonitrile in water (0.1% formic acid) over 2 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).

Peptide [As(2')Nal^{7}]UII$^{(4-11)}$ **(9)**



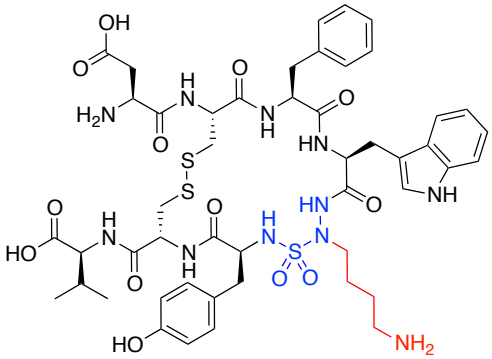


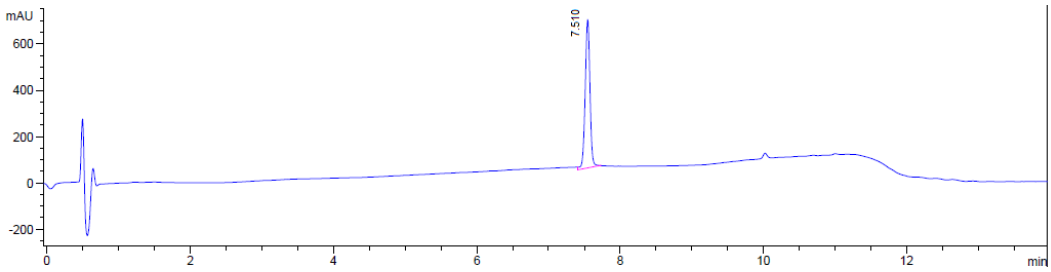
Purity check/Gradient #1 - purity: >99%, t_R: 15.60 min [analytical HPLC, 20 to 80% methanol in water (0.1% formic acid) over 15 min + 90% methanol in water (0.1% formic acid) over 5 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).



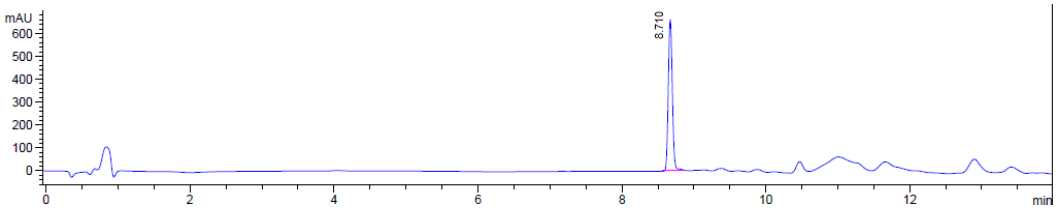
Purity check/Gradient #2 - purity: >99%, t_R: 10.44 min [analytical HPLC, 20 to 80% acetonitrile in water (0.1% formic acid) over 15 min + 90% acetonitrile in water (0.1% formic acid) over 5 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).

Peptide [asK^{8}]UII$^{(4\text{-}11)}$ **(10)**



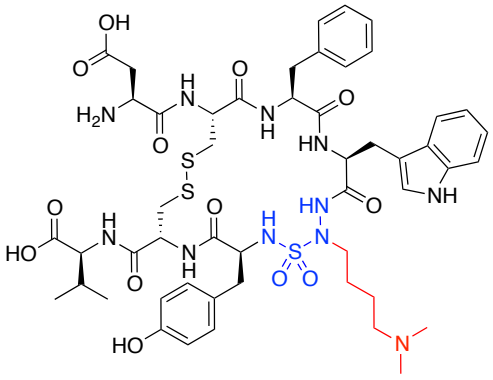


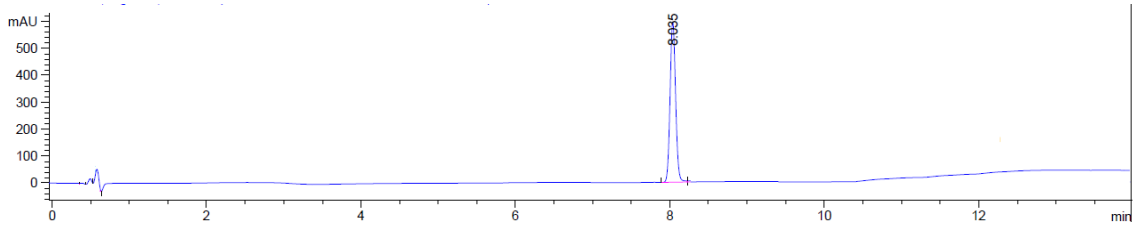
Purity check/Gradient #1 - purity: >99%, t_R: 7.51 min [analytical HPLC, 10-90% acetonitrile in water (0.1% formic acid) over 8 min + 90% acetonitrile in water (0.1% formic acid) over 2 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).



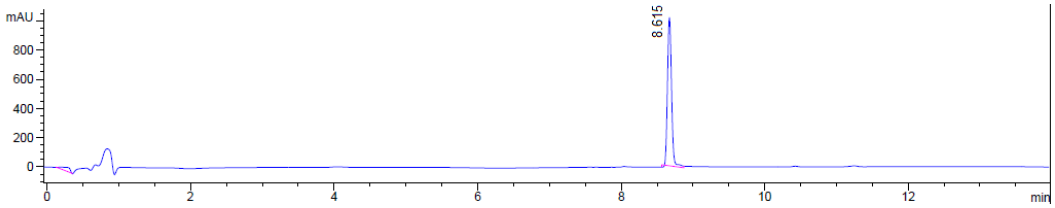
Purity check/Gradient #2 - purity: >99%, t_R: 8.71 min [analytical HPLC, 10 to 90% methanol in water (0.1% formic acid) over 8 min + 90% methanol in water (0.1% formic acid) over 2 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm X 100 mm).

Peptide [As(*N*,*N*-Me_2)K^8]$UII^{(4\text{-}11)}$ **(11)**





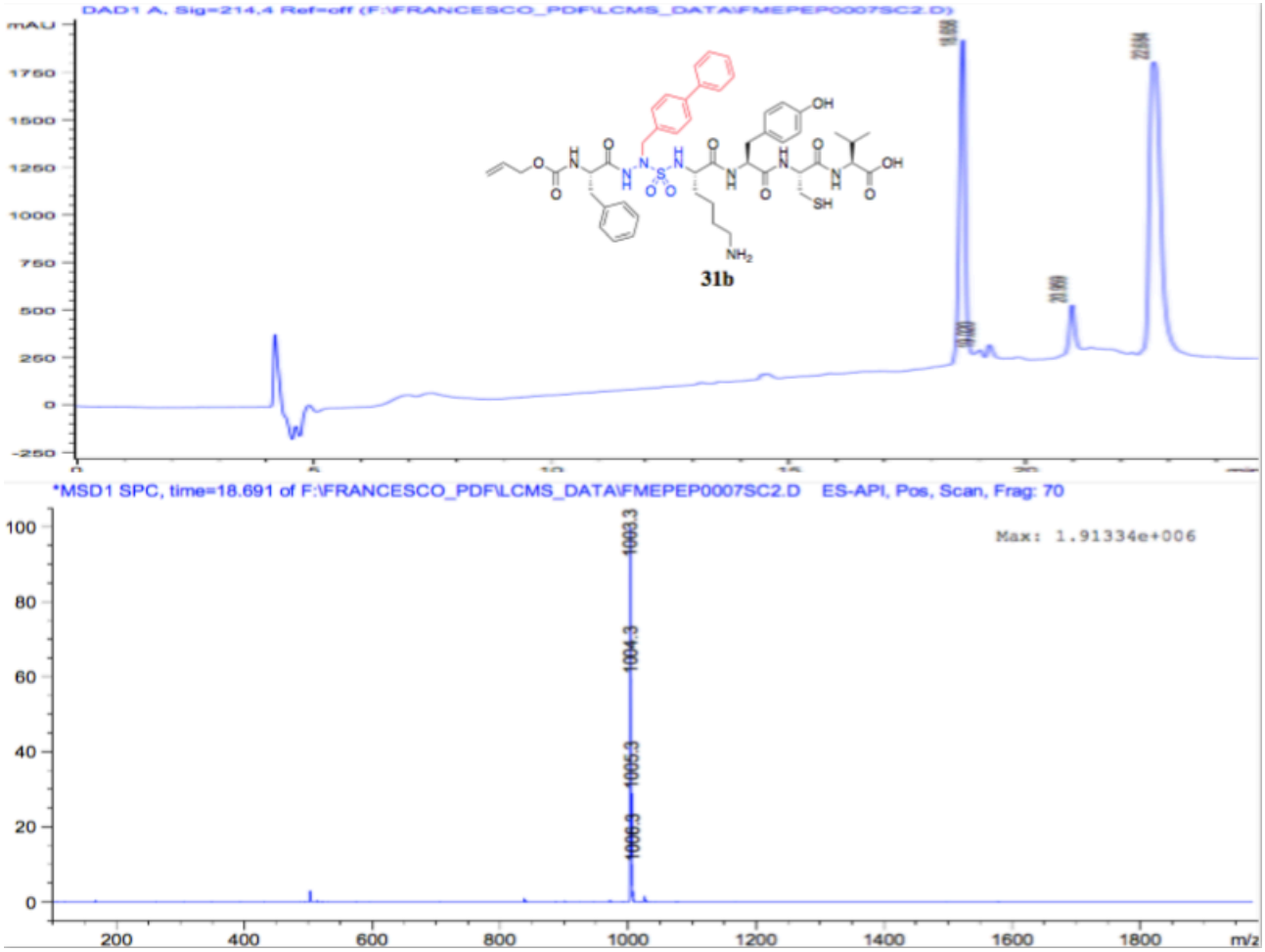
Purity check/Gradient #1 - purity: >99%, t_R: 8.04 min [analytical HPLC, 10 to 90% acetonitrile in water (0.1% formic acid) over 8 min + 90% acetonitrile in water (0.1% formic acid) over 2 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100 Å, 3.5 µm, 4.6 mm X 100 mm).



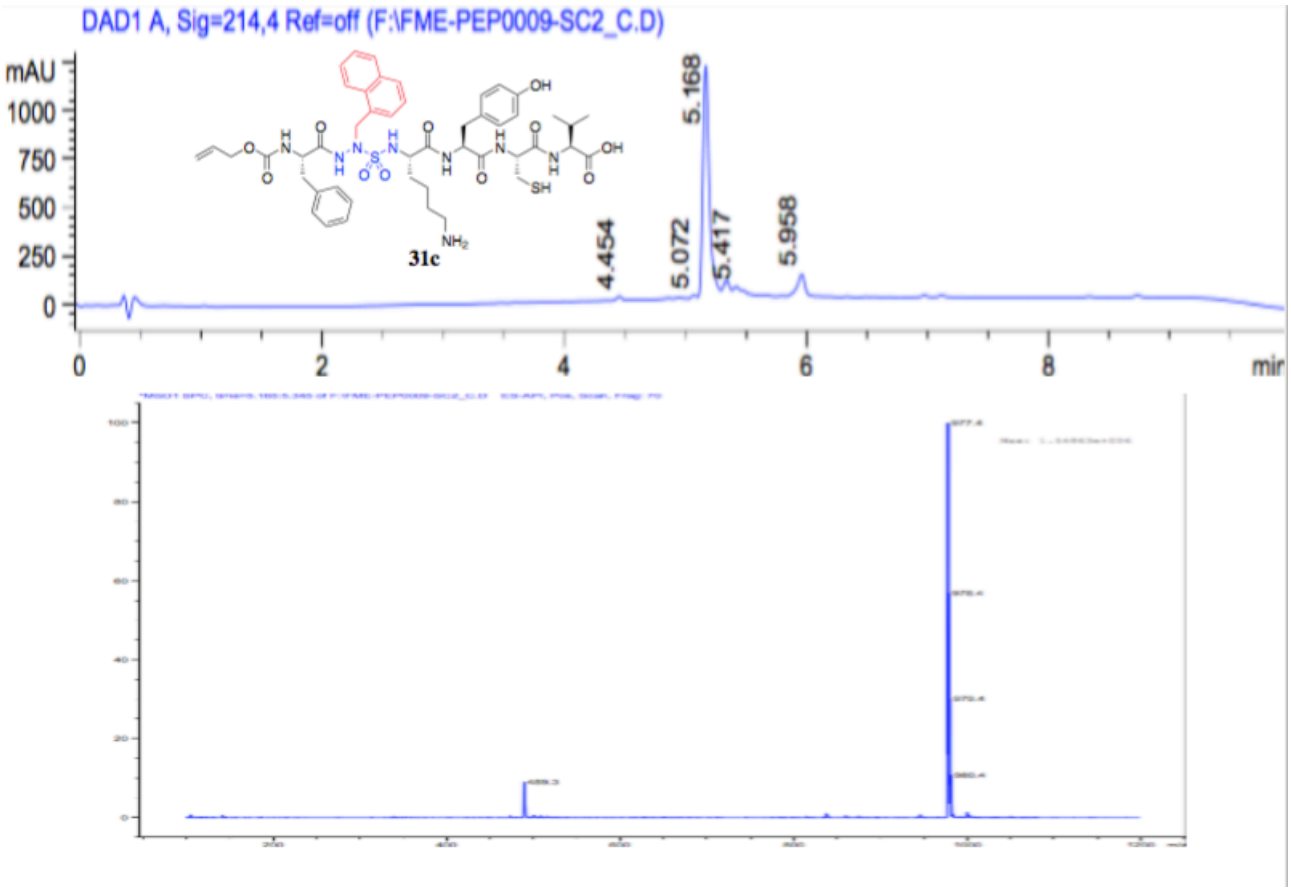
Purity check/Gradient #2 - Purity: >99%, t_R: 8.61 min [analytical HPLC, 10 to 90% methanol in water (0.1% formic acid) over 8 min + 90% methanol in water (0.1% formic acid) over 2 min, flow rate of 0.5 mL/min] on a Sunfire C18 analytical column (100 Å, 3.5 µm, 4.6 mm X 100 mm).

LC-MS Analyses

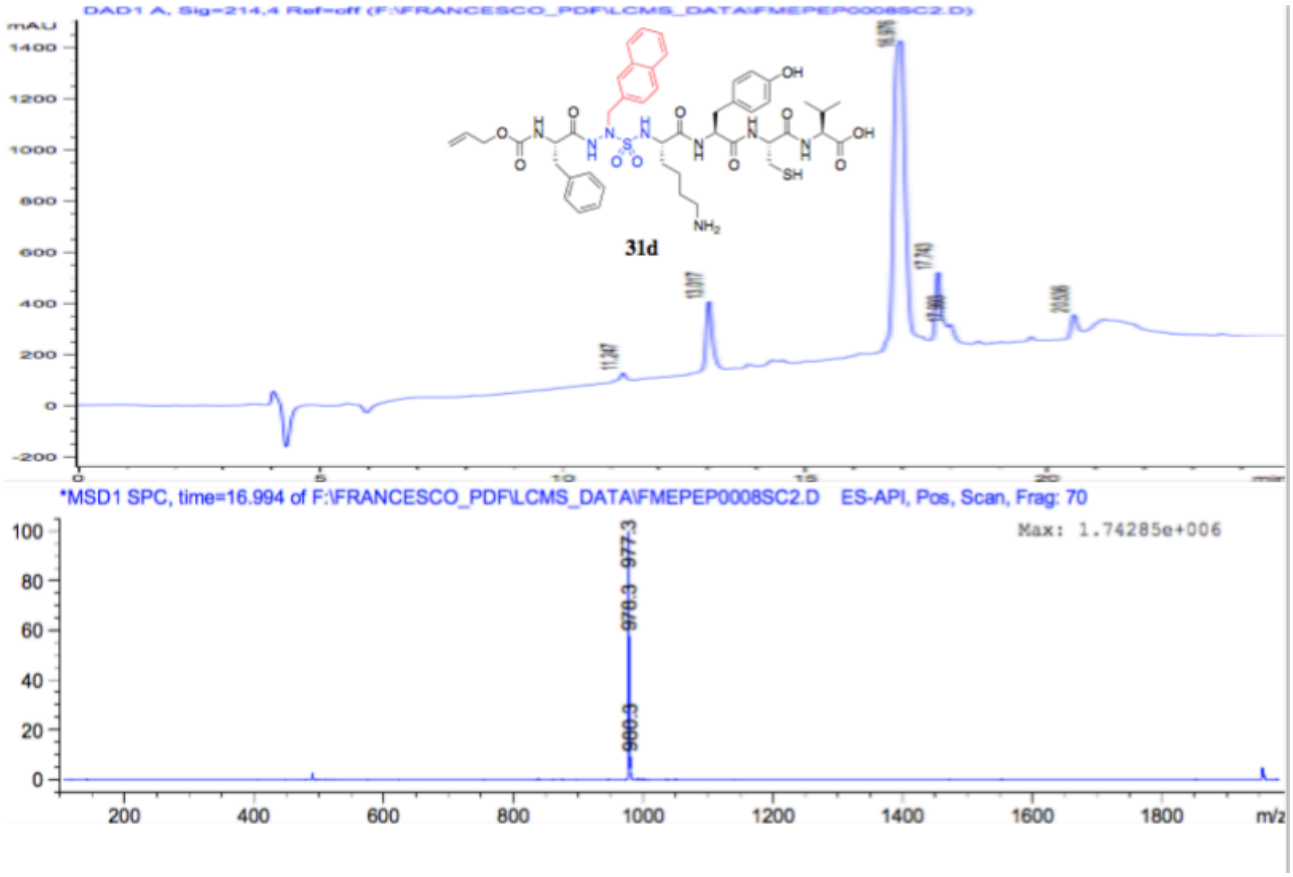
LC-MS chromatogram (10 to 80% MeOH over 15 min, t_R: 17.40 min) of small amount of resin **31a** conveniently cleaved and analyzed on Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm x 100 mm).



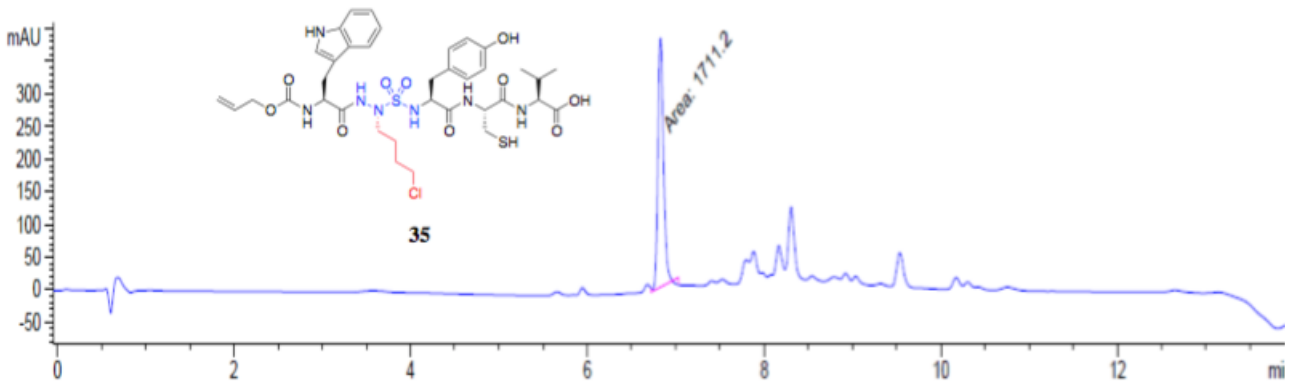
LC-MS chromatogram (10 to 80% MeOH over 15 min, t_R: 18.69 min) of resin **31b** aliquot cleaved and analyzed on Sunfire C18 analytical column (100Å, 3.5 μm, 4.6 mm x 100 mm).



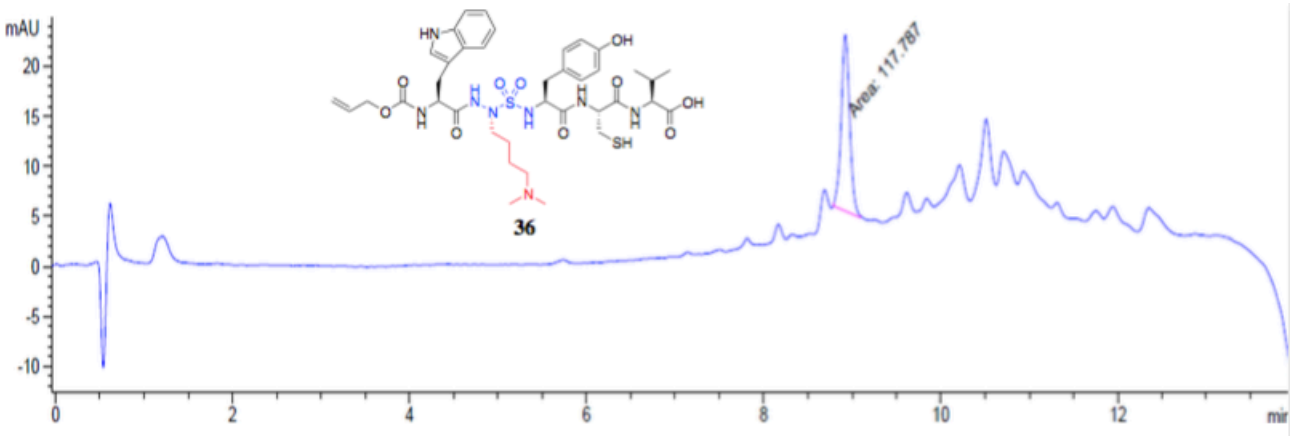
LC-MS chromatogram (15 to 80% MeOH over 8 min, t_R: 5.17 min) of resin **31c** aliquot cleaved and analyzed on Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm x 100 mm).



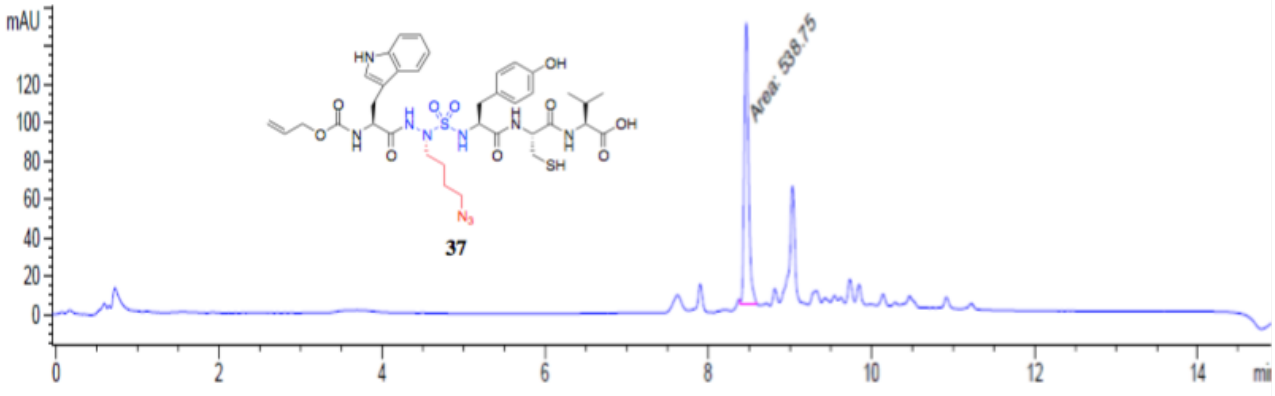
LC-MS chromatogram (20 to 80% MeOH over 15 min, t_R: 16.99 min) of resin **31d** aliquot cleaved and analyzed on Sunfire C18 analytical column (100Å, 3.5 μm, 4.6 mm x 100 mm).



LC-MS chromatogram of **35** (20-90% MeOH, 14 min, t_R = 6.8 min), on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm x 100 mm).

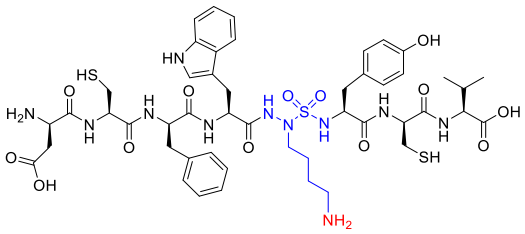


LC-MS chromatogram of **36** (10-90% MeOH, 14 min, t_R = 8.9 min), on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm x 100 mm).

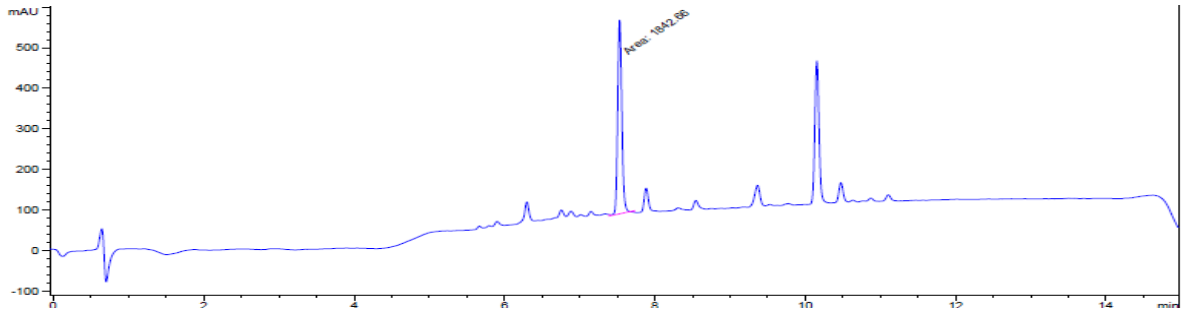


LC-MS chromatogram of **37** (20-90% MeOH, 14 min, t_R = 8.3 min), on a Sunfire C18 analytical column (100Å, 3.5 µm, 4.6 mm x 100 mm).

Azide reduction on solid support



(**42**)



LC-MS chromatogram of **42** (10-90% MeOH, 15 min, t_R = 7.6 min), on a Sunfire C18 analytical column (100Å, 3.5 μm, 4.6 mm X 100 mm).