Supporting Information

$Cu(OTf)_2$-Catalyzed C3 *Aza*-Friedel-Crafts Alkylation of Indoles with *N*, *O*-Acetals

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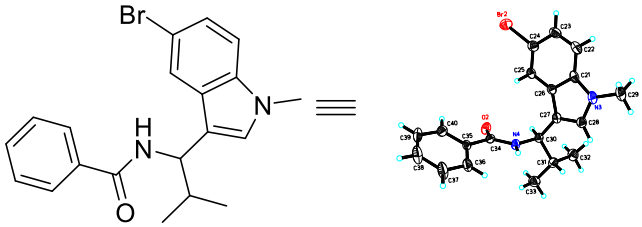
I. X-Ray Crystallographic Structure for compounds 6k and 6y

ORTEP drawing of the X-ray crystallographic structure of **6k** (50% probability ellipsoids, **dd21044** is the original test number for **6k**): The single crystal of **6k** was prepared from its solution in ethylacetate /methanol (6:1) by slow evaporation of the solvent (**Table S1**)



6k **6k**

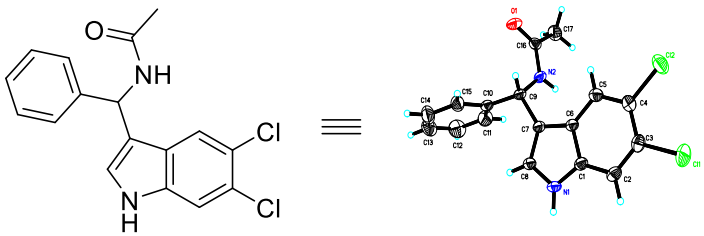
Table S1. Crystal data and structure refinement for **6k.**

Identification code	**6k**
Empirical formula	$C_{20}H_{21}BrN_2O$
Formula weight	385.30
Temperature	213(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P -1
Unit cell dimensions	a = 9.7774(6) Å α= 91.317(2)°
	b = 10.6374(7) Å β= 95.348(2)°
	c = 18.9895(14) Å γ= 108.325(2)°
Volume	1863.8(2) $Å^3$
Z	4
Density (calculated)	1.373 Mg/m^3
Absorption coefficient	2.214 mm^{-1}
F(000)	792
Crystal size	0.160 x 0.110 x 0.100 mm^3
Theta range for data collection	2.342 to 26.000°
Index ranges	-12<=h<=12, -13<=k<=13, -23<=l<=23
Reflections collected	36864
Independent reflections	7312 [R(int) = 0.0659]
Completeness to theta = 25.242°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.5427
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7312 / 0 / 439
Goodness-of-fit on F^2	1.006
Final R indices [I>2sigma(I)]	R1 = 0.0452, wR2 = 0.0984
R indices (all data)	R1 = 0.0854, wR2 = 0.1174
Extinction coefficient	n/a
Largest diff. peak and hole	0.568 and -0.544 e. $Å^{-3}$

CCDC-2117863. For detailed crystallographic data, please refer to the Cambridge

Crystallographic Data Centre at http://ccdc.cam.ac.uk.\

ORTEP drawing of the X-ray crystallographic structure of **6y** (50% probability ellipsoids, **dd21019** is the original test number for **6y**): The single crystal of **6y** was prepared from its solution in petroleum ether/ethylacetate (1:1) by slow evaporation of the solvent (**Table S2**)



6y **6y**

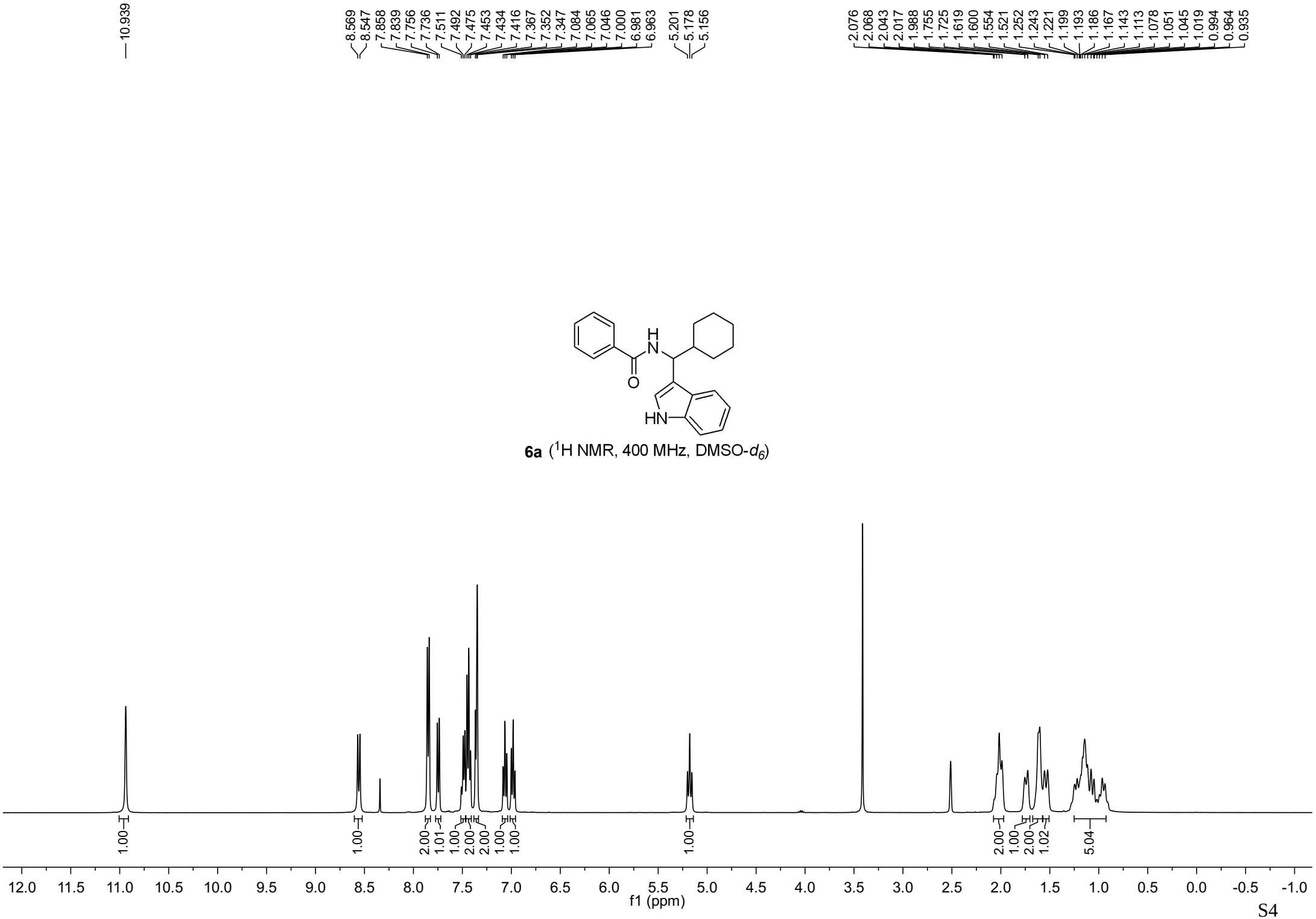
Table S2. Crystal data and structure refinement for **6y**.

Identification code	**6y**
Empirical formula	$C_{21}H_{22}Cl_2N_2O_3$
Formula weight	421.30
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P -1
Unit cell dimensions	a = 9.2206(5) Å α= 112.905(2)°
	b = 11.1857(6) Å β= 103.286(2)°
	c = 11.8704(7) Å γ= 96.447(2)°
Volume	1069.43(10) $Å^3$
Z	2
Density (calculated)	1.308 Mg/m^3
Absorption coefficient	0.327 mm^{-1}
F(000)	440
Crystal size	0.160 x 0.140 x 0.110 mm^3
Theta range for data collection	2.127 to 25.997°
Index ranges	-11<=h<=11, -13<=k<=13, -14<=l<=14
Reflections collected	20962
Independent reflections	4192 [R(int) = 0.0337]
Completeness to theta = 25.242°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.7061
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4192 / 0 / 257
Goodness-of-fit on F^2	1.018

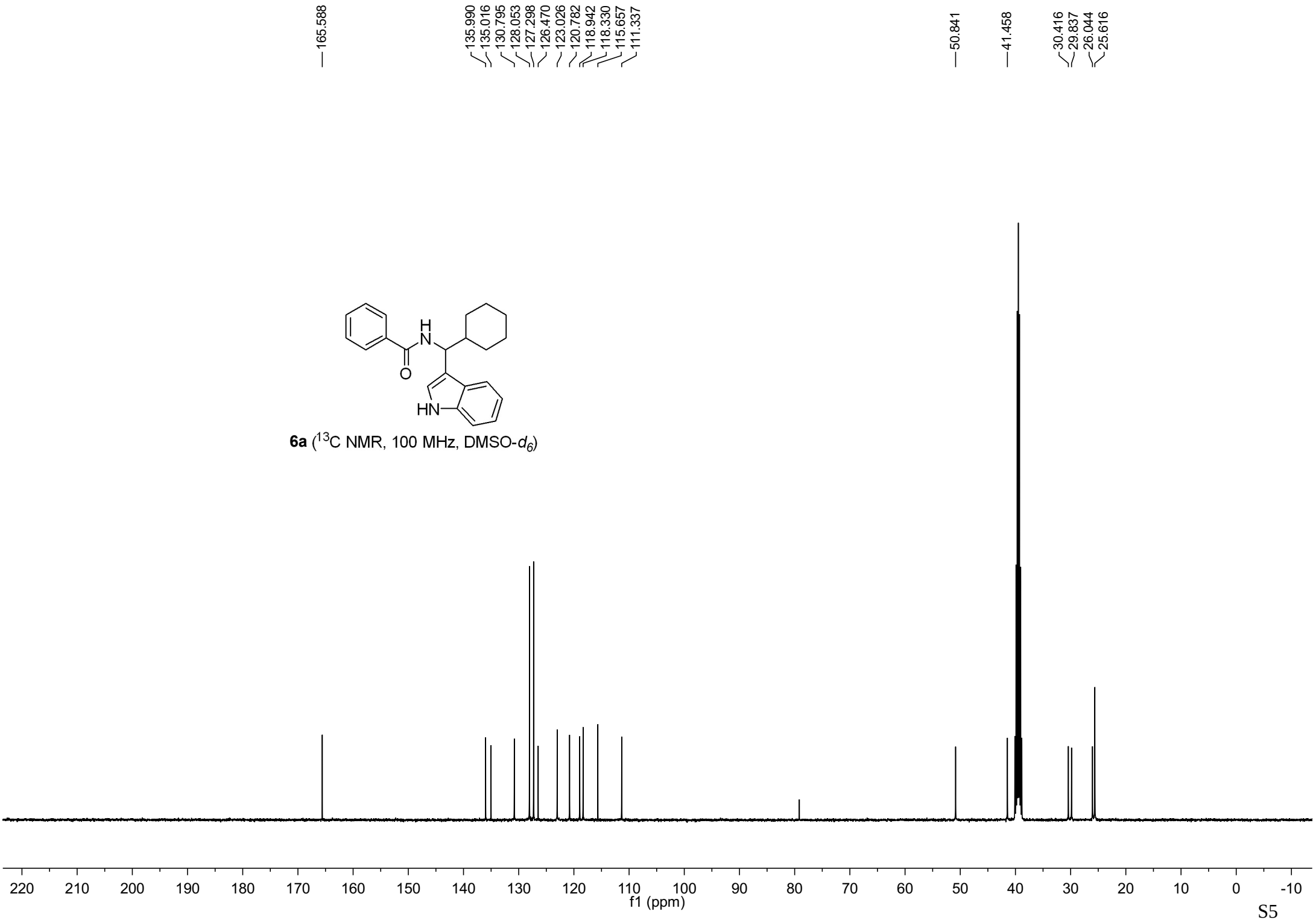
Final R indices [I>2sigma(I)]	R1 = 0.0435, wR2 = 0.1076
R indices (all data)	R1 = 0.0583, wR2 = 0.1191
Extinction coefficient	0.019(4)
Largest diff. peak and hole	0.208 and -0.258 e. $Å^{-3}$

CCDC-2117862. For detailed crystallographic data, please refer to the Cambridge Crystallographic Data Centre at http://ccdc.cam.ac.uk.\

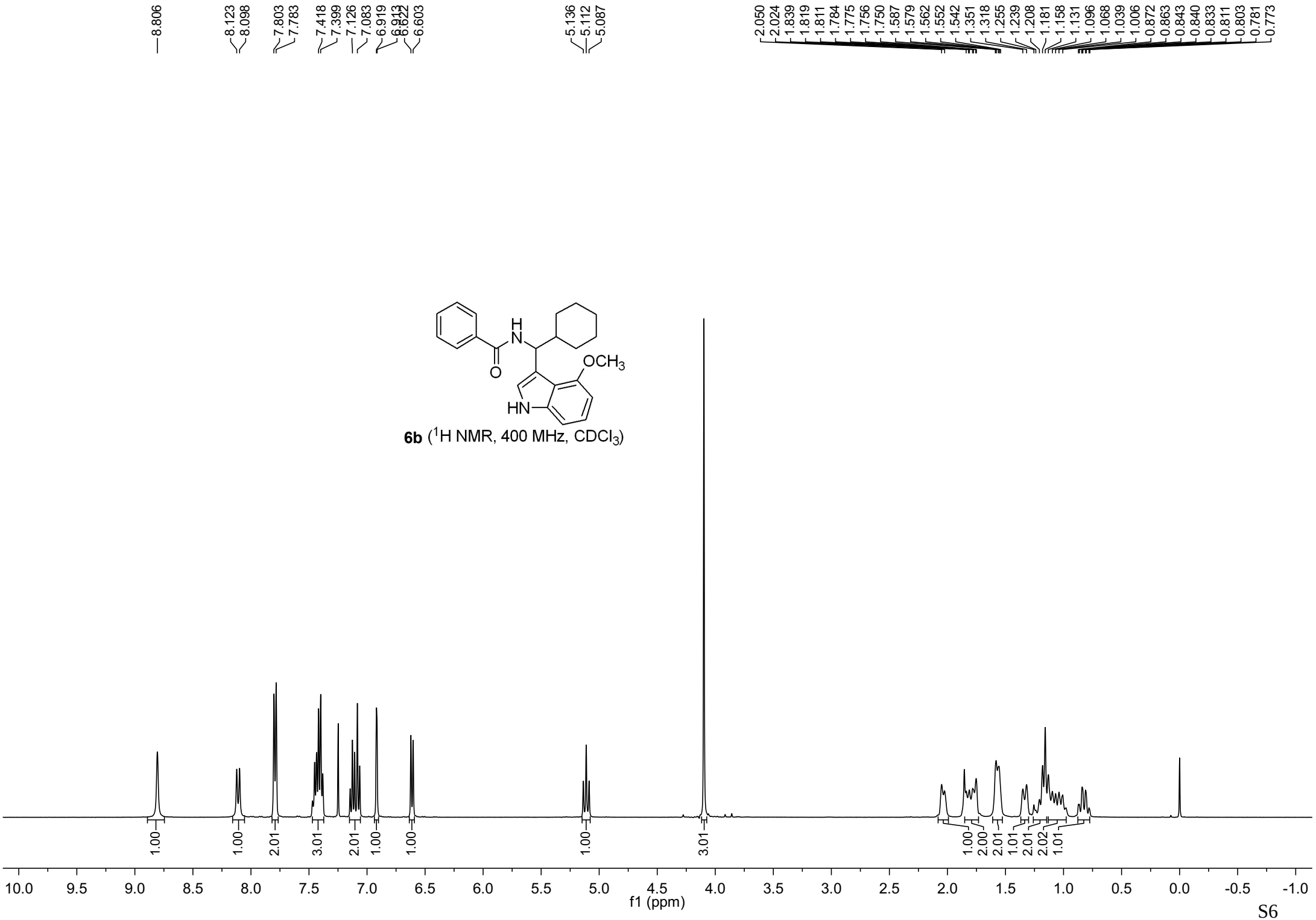
II. Copies of ^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectrum



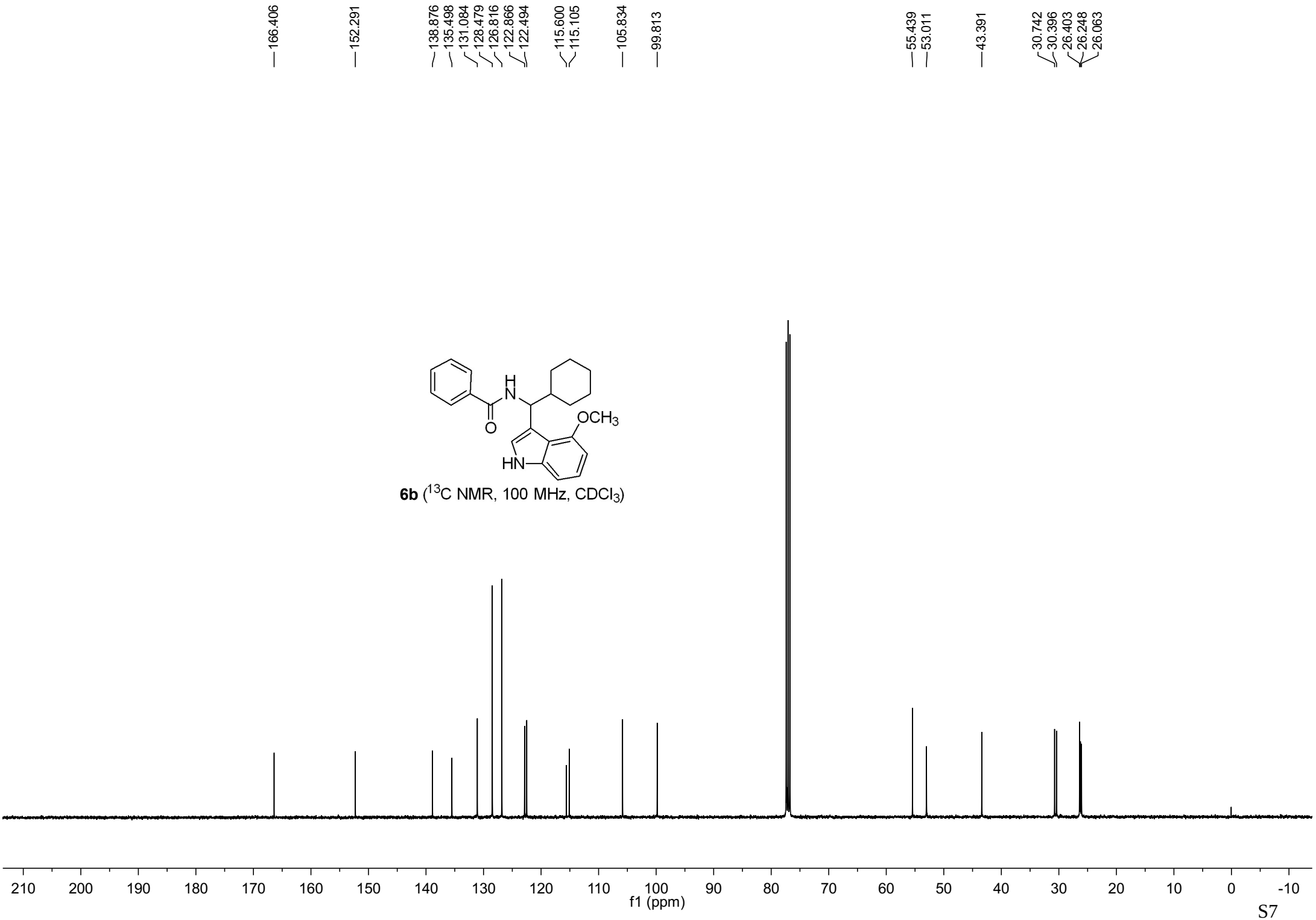
6a (^{1}H NMR, 400 MHz, DMSO-d_6)



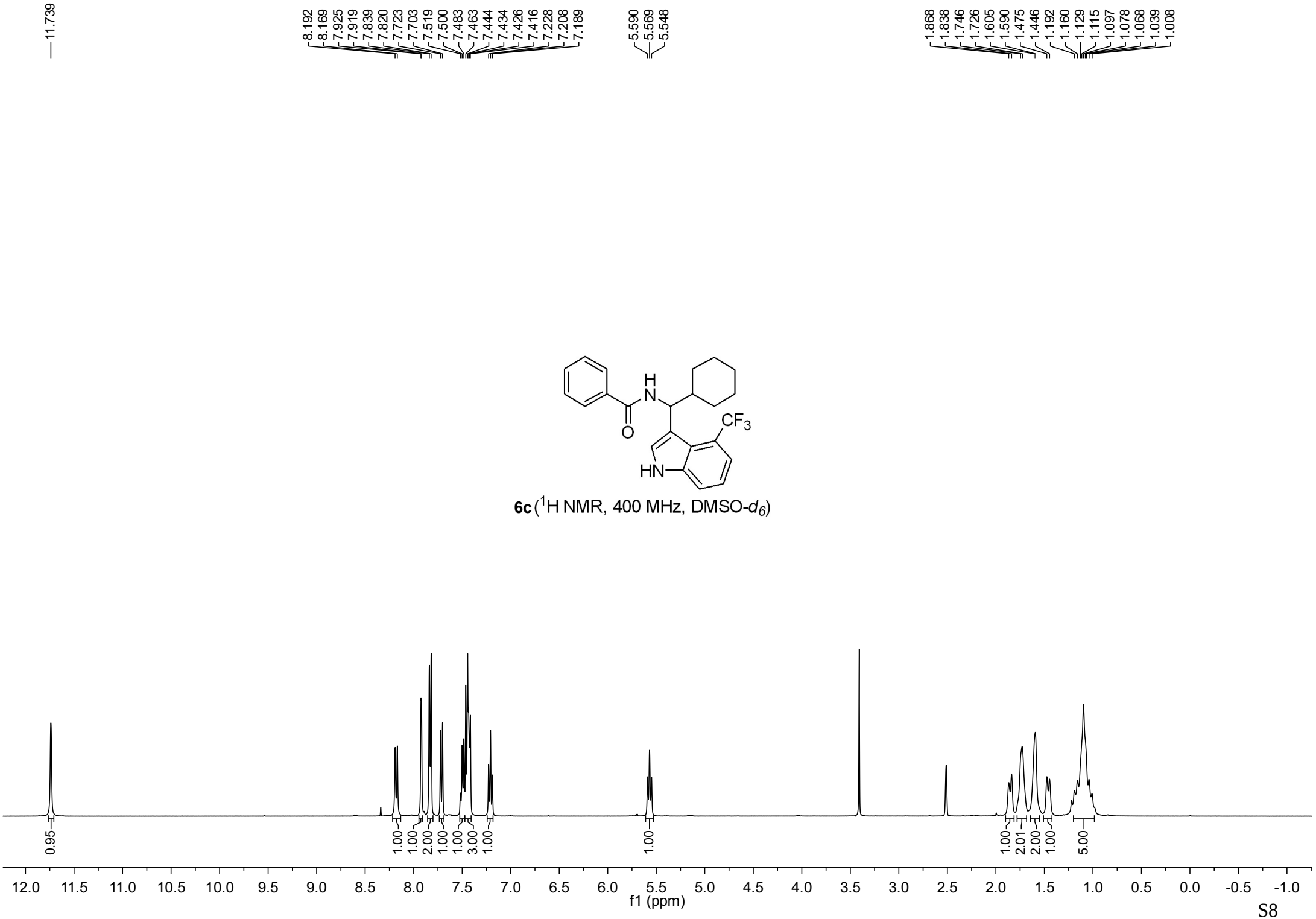
6a (^{13}C NMR, 100 MHz, DMSO-d_6)



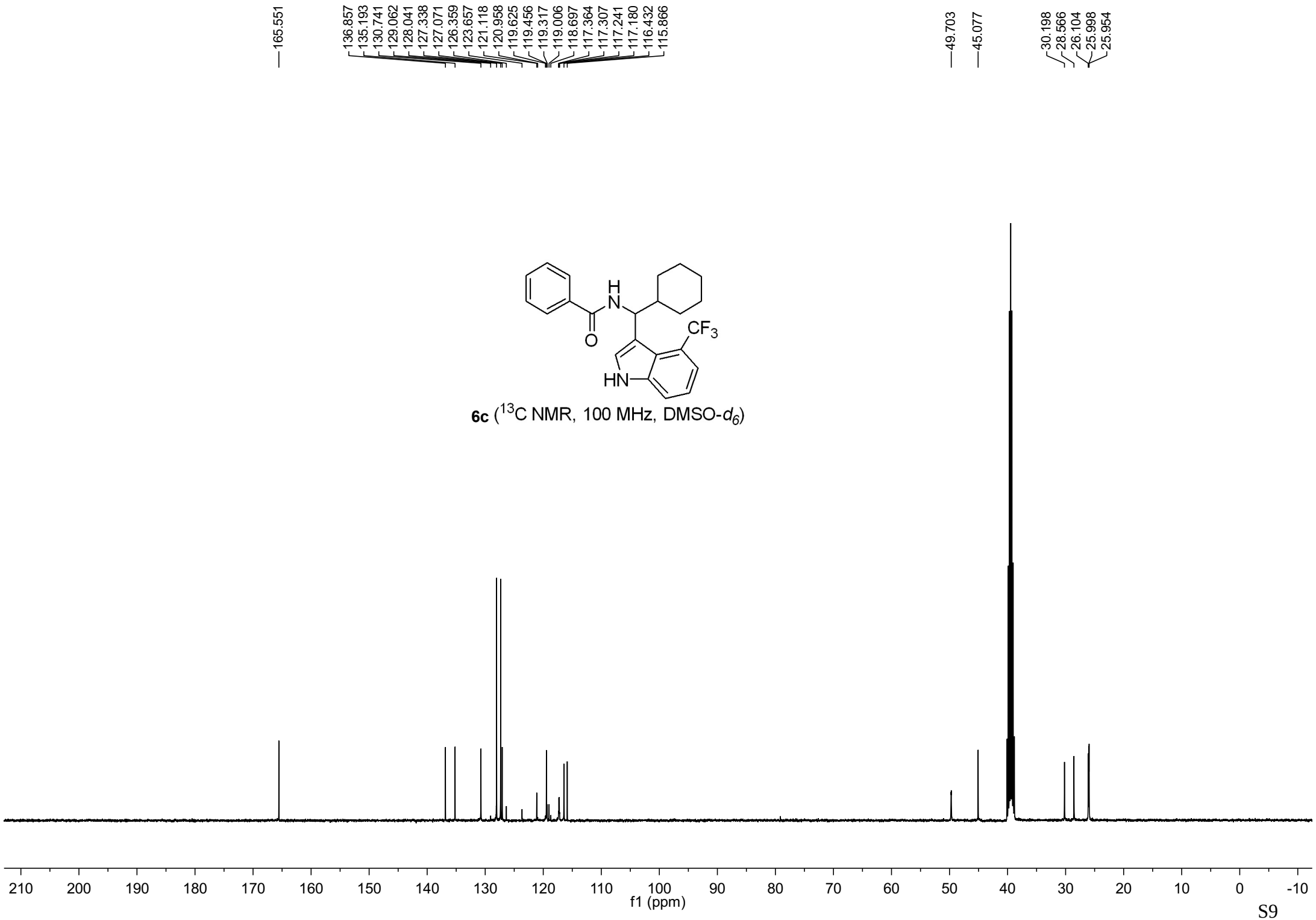
6b (^{1}H NMR, 400 MHz, $CDCl_3$)



6b (^{13}C NMR, 100 MHz, $CDCl_3$)

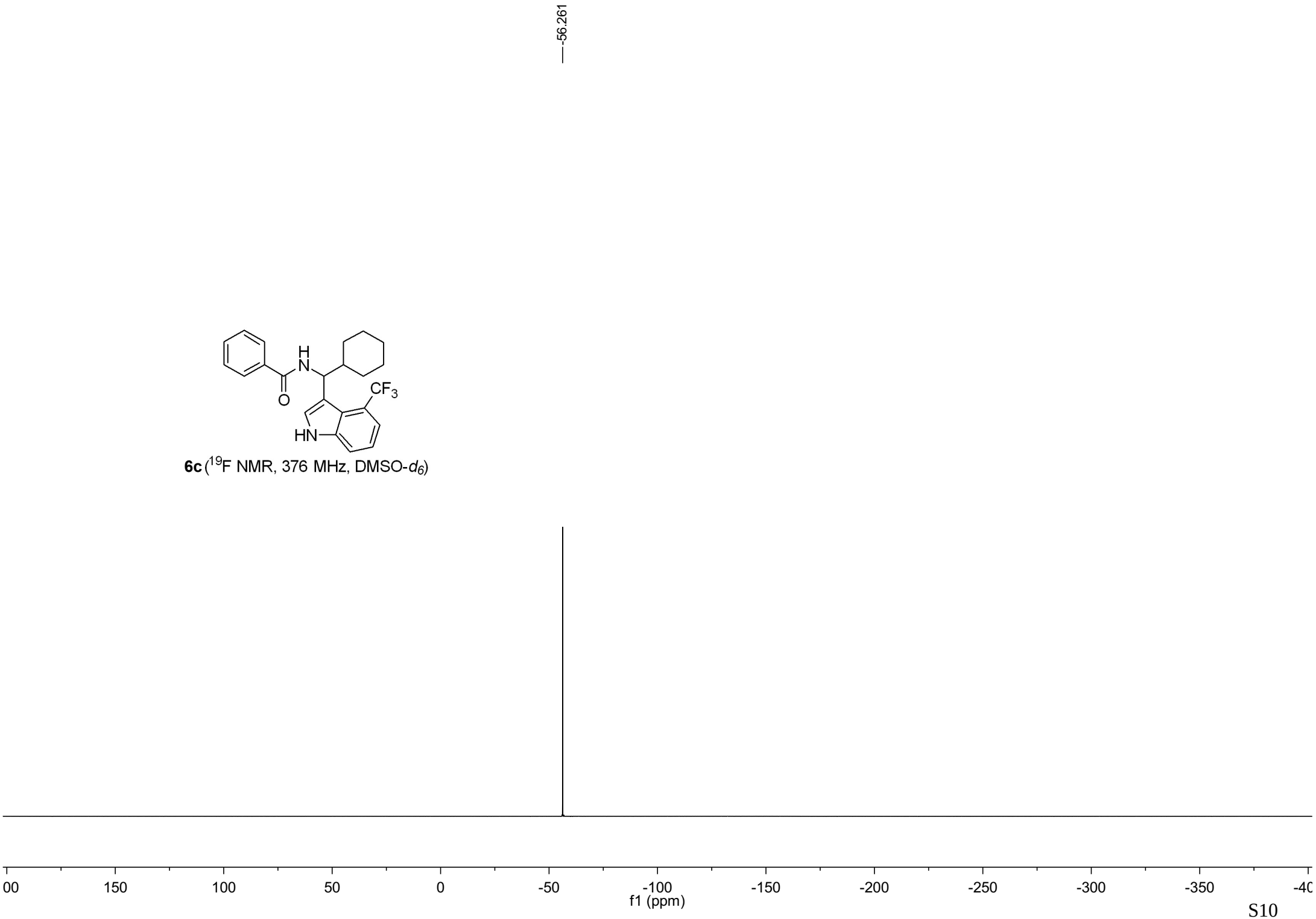


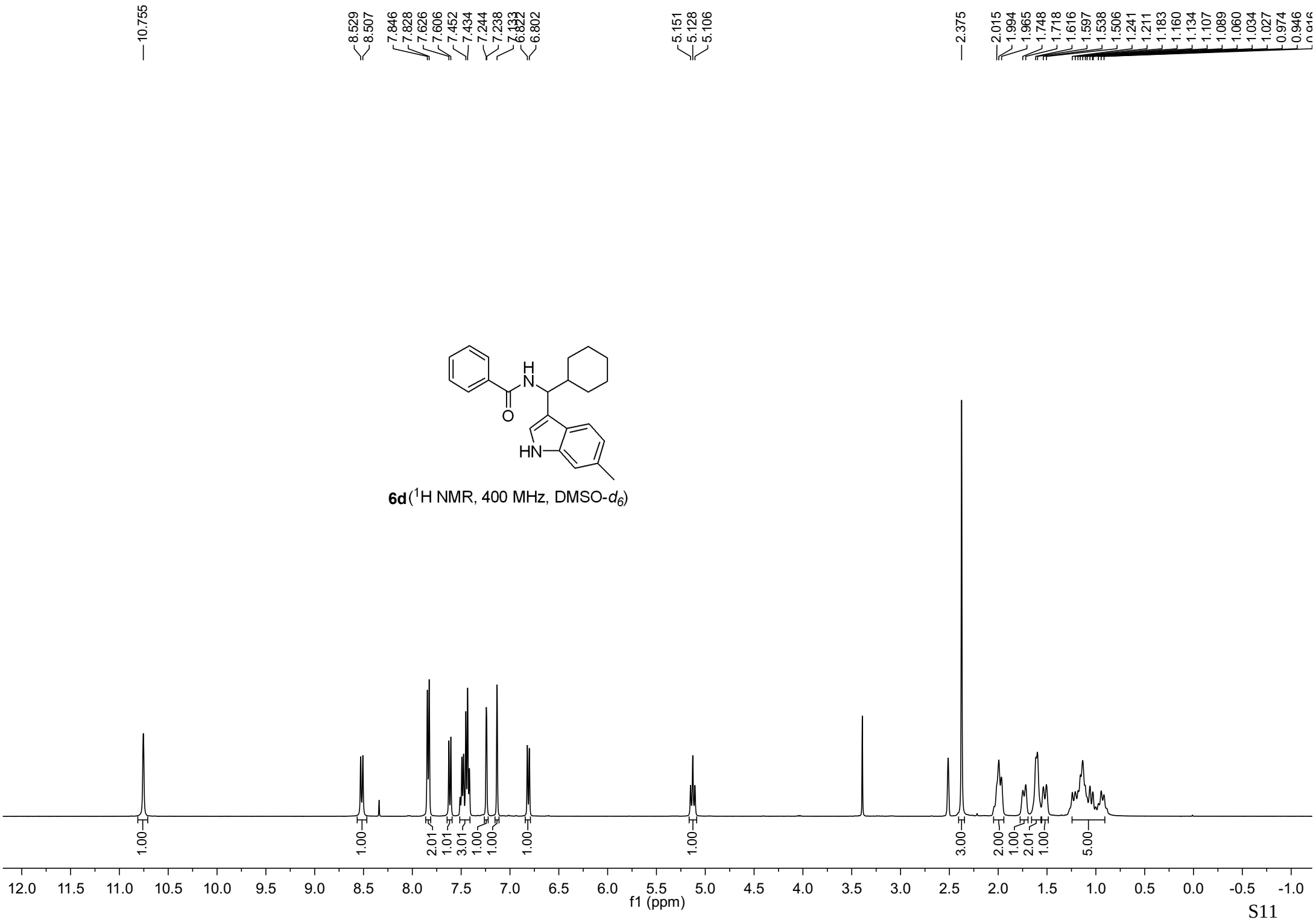
6c (^{1}H NMR, 400 MHz, DMSO-d_6)



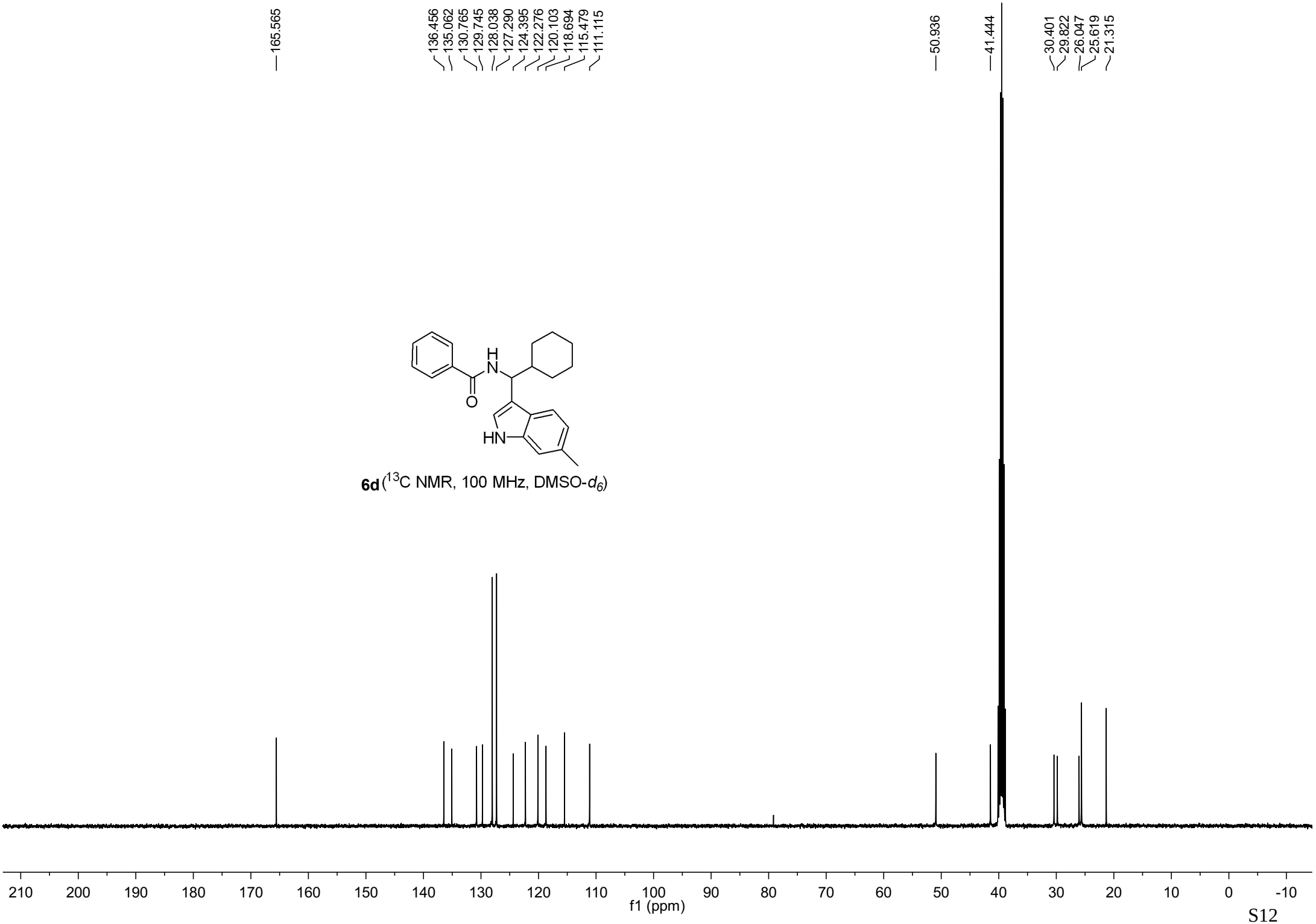
6c (^{13}C NMR, 100 MHz, DMSO-d_6)

6c (^{19}F NMR, 376 MHz, DMSO-d_6)

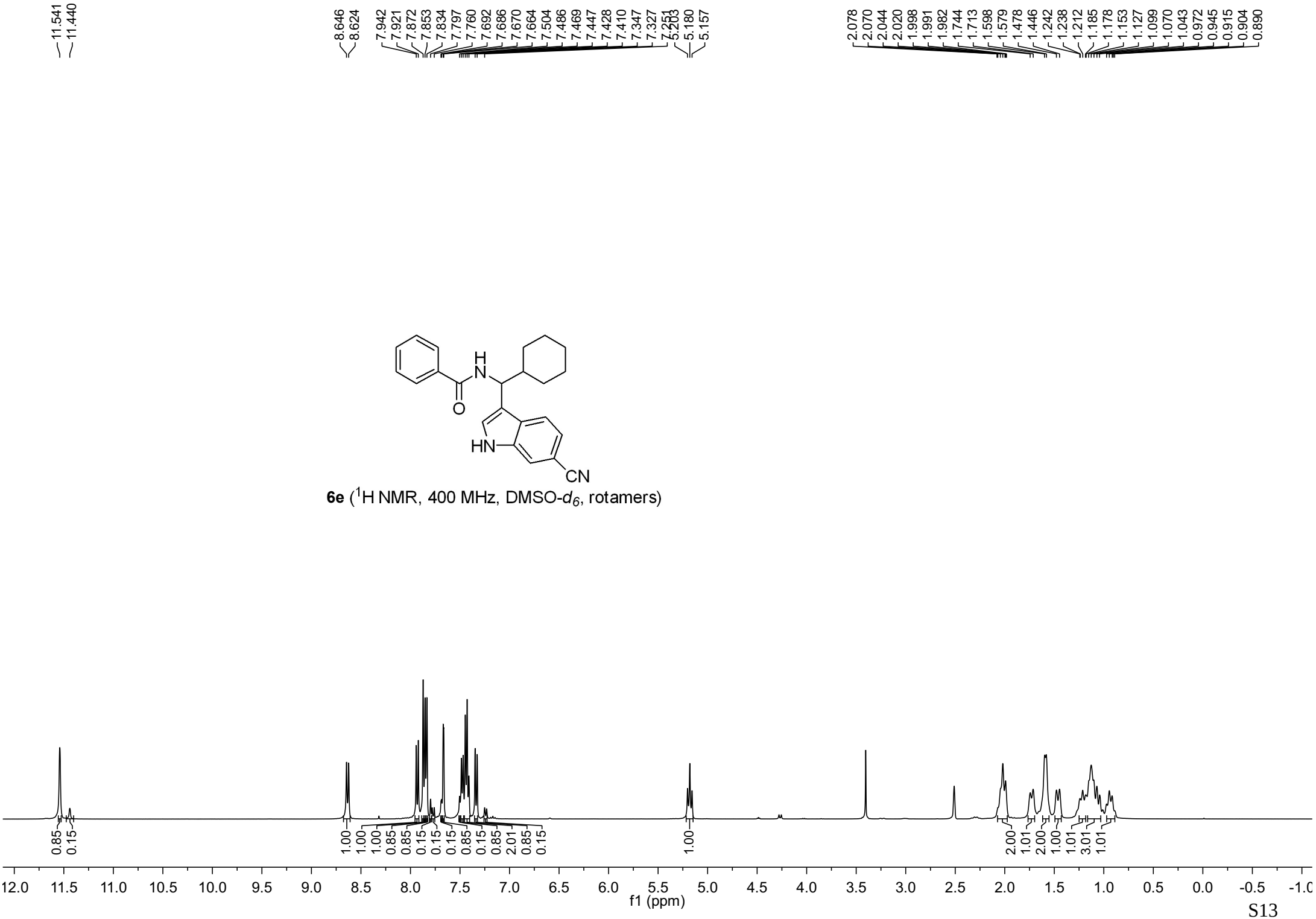




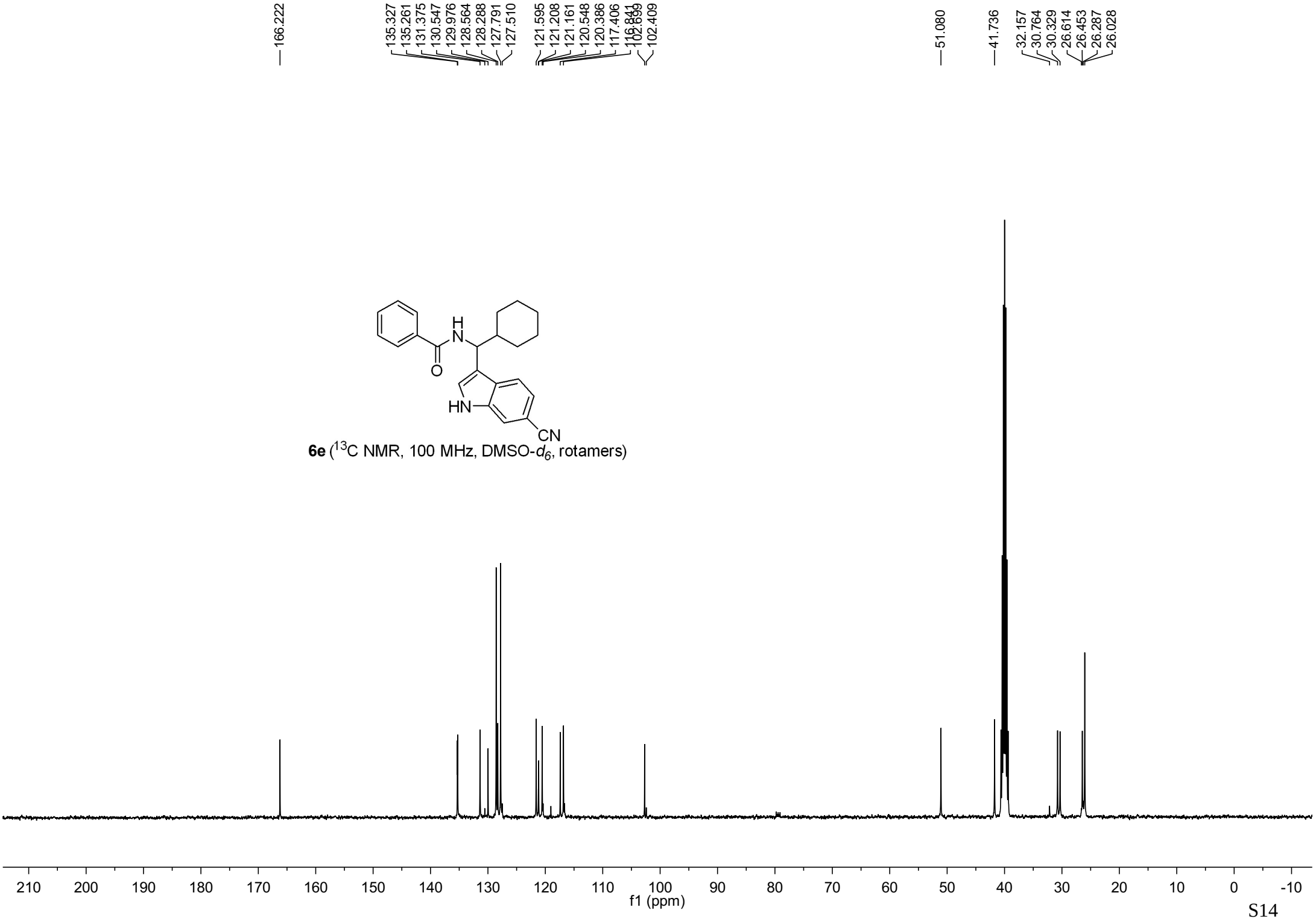
6d (^{1}H NMR, 400 MHz, DMSO-d_6)



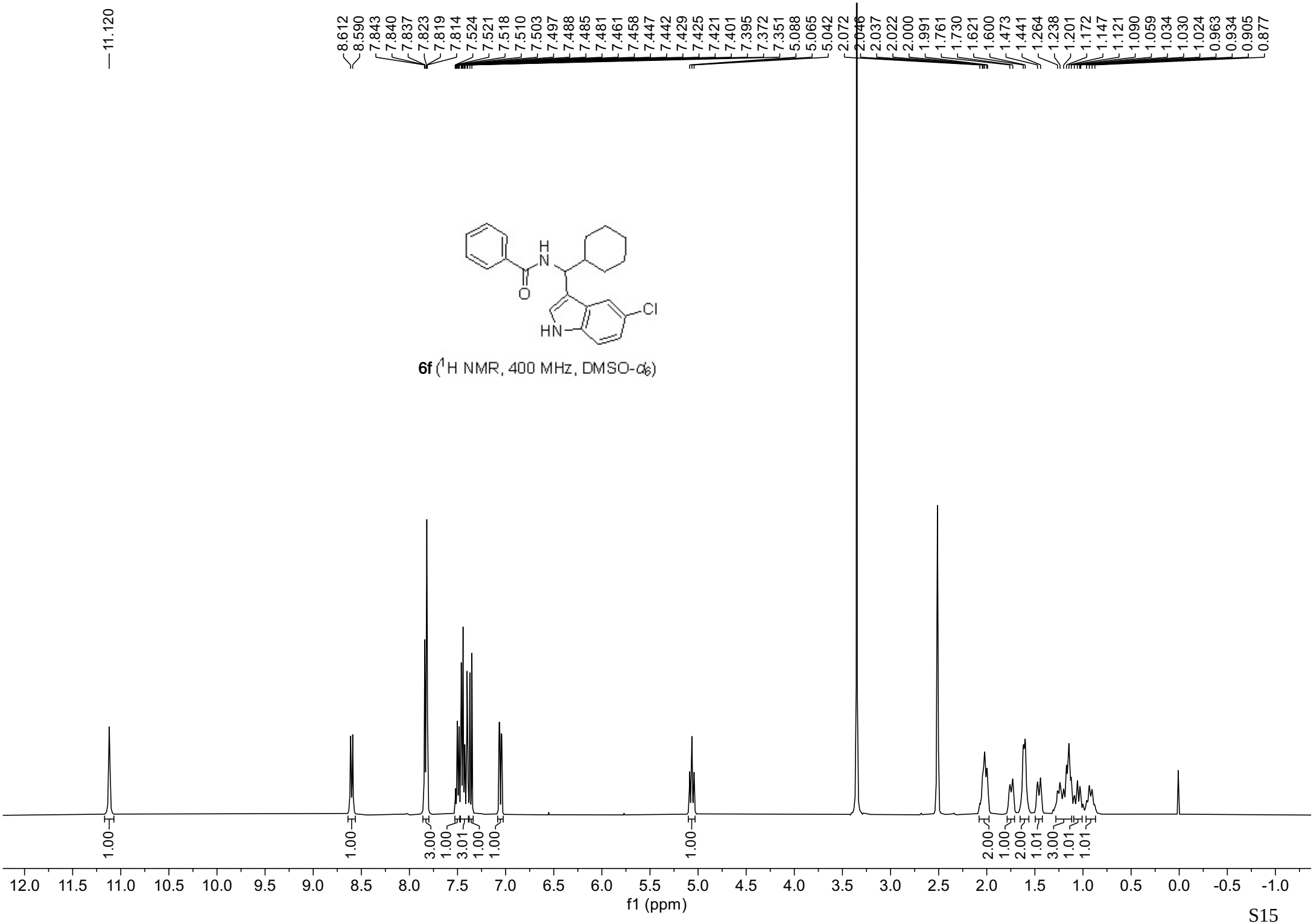
6d (^{13}C NMR, 100 MHz, DMSO-d_6)



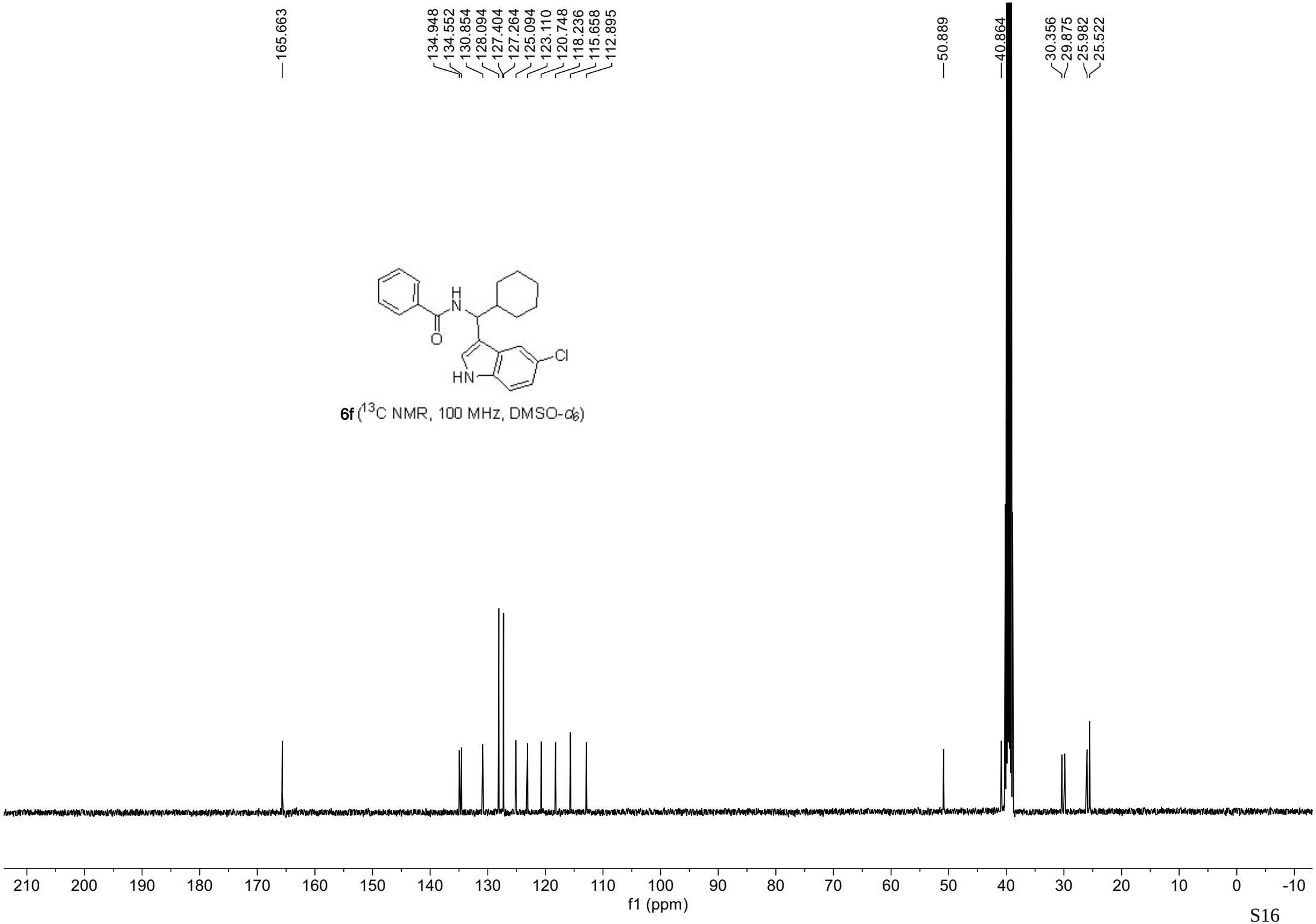
6e (^{1}H NMR, 400 MHz, DMSO-d_6, rotamers)



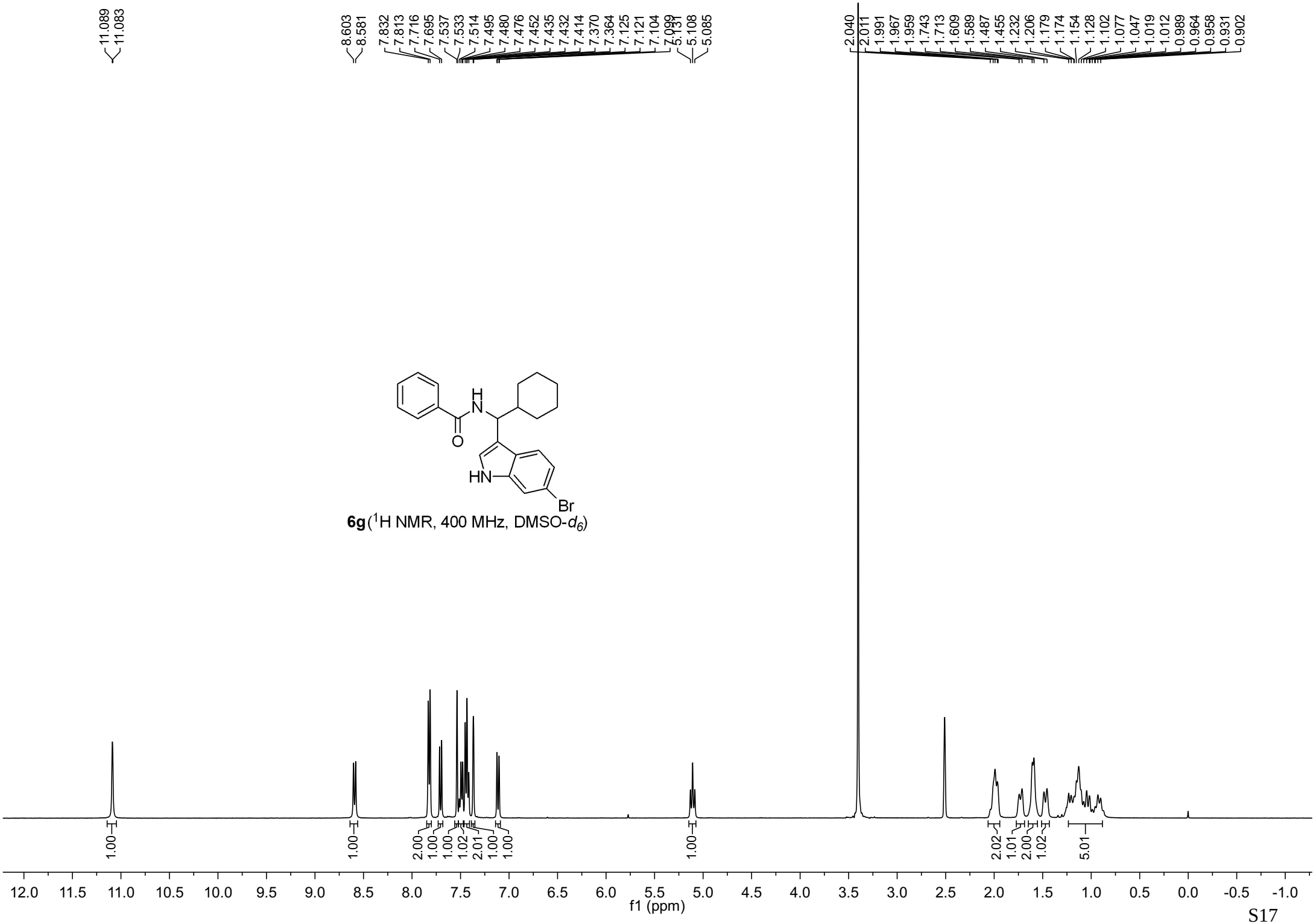
6e (^{13}C NMR, 100 MHz, DMSO-d_6, rotamers)



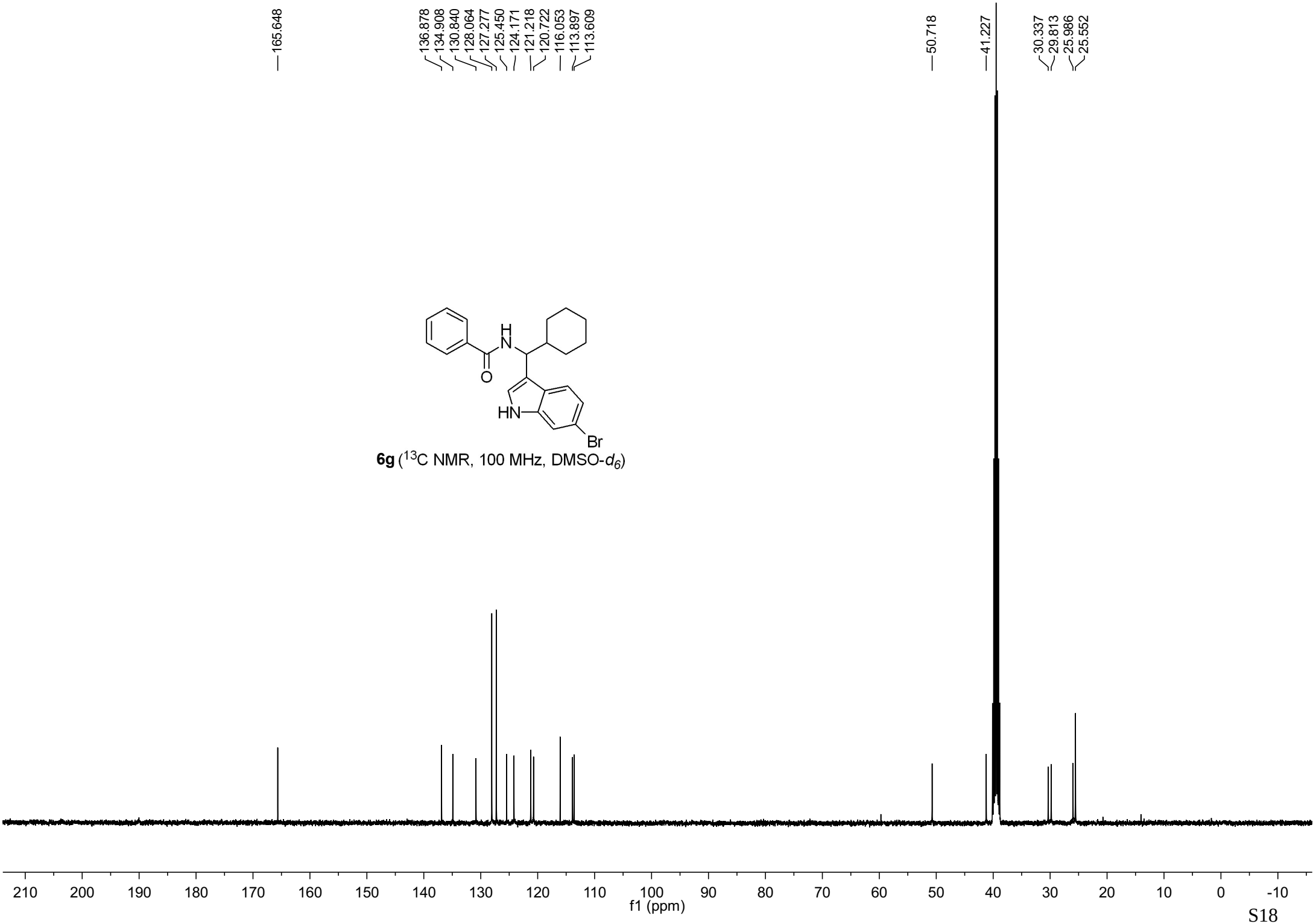
6f (^{1}H NMR, 400 MHz, DMSO-d_6)



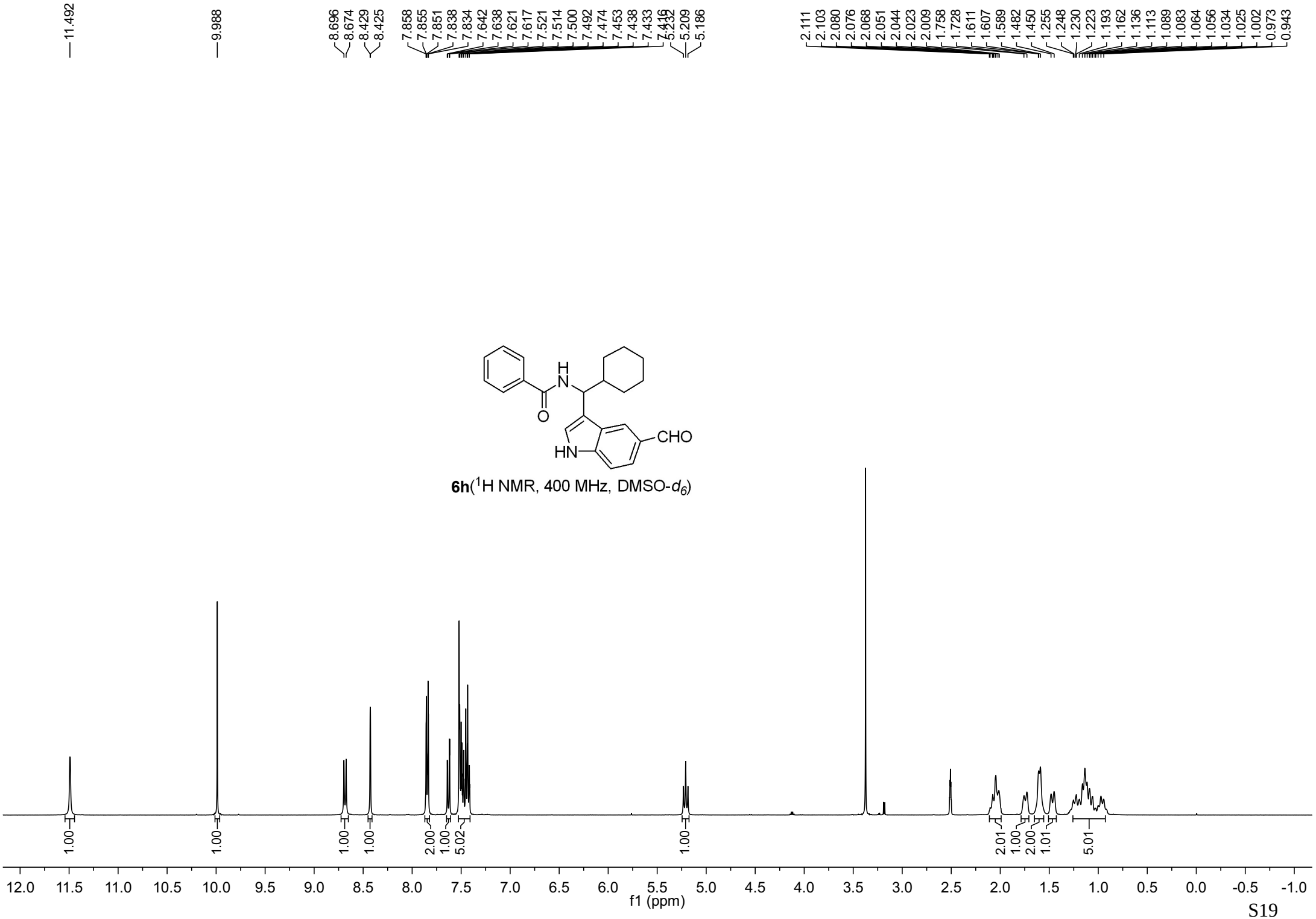
6f (^{13}C NMR, 100 MHz, DMSO-d_6)

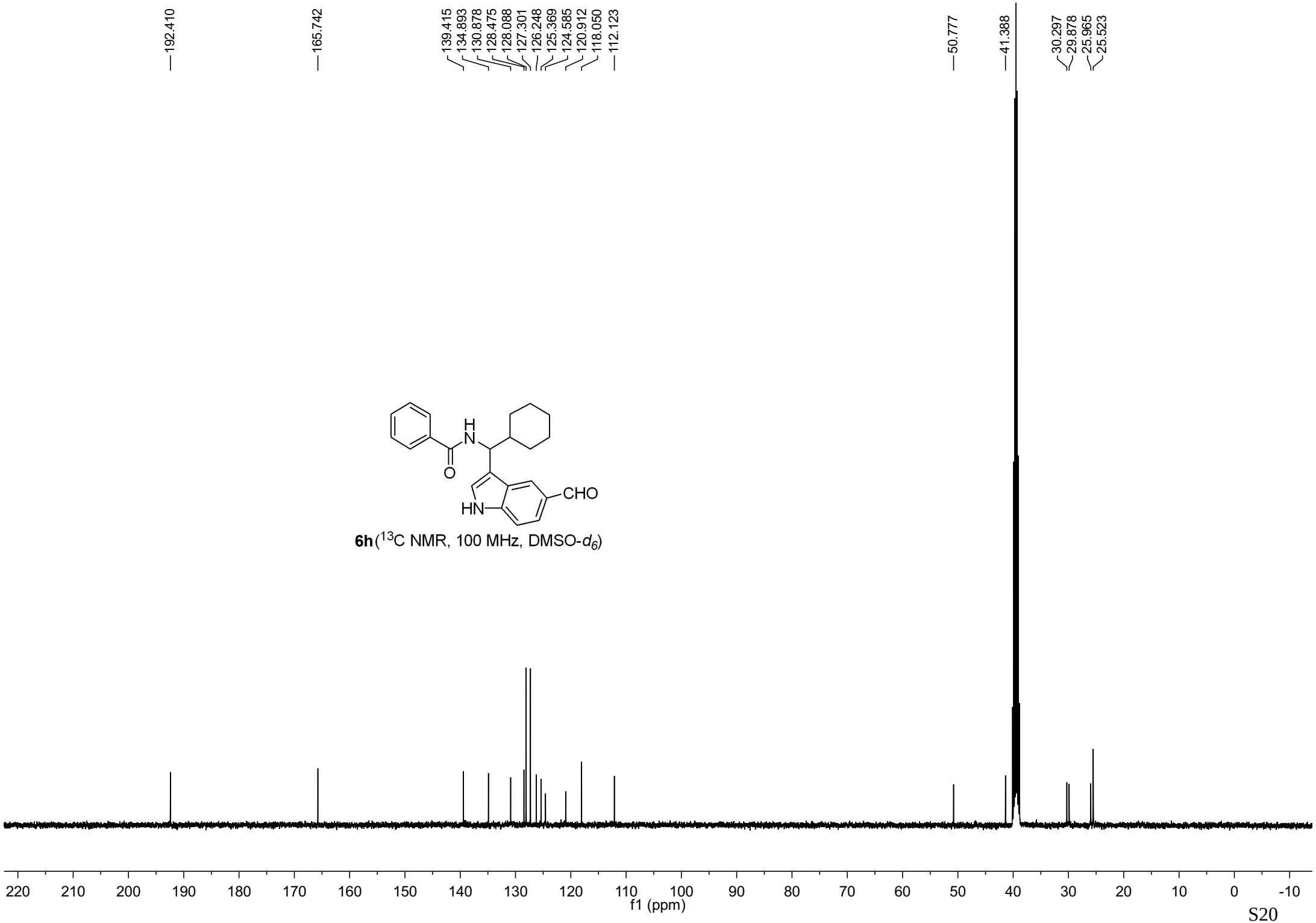


6g (^{1}H NMR, 400 MHz, DMSO-d_6)

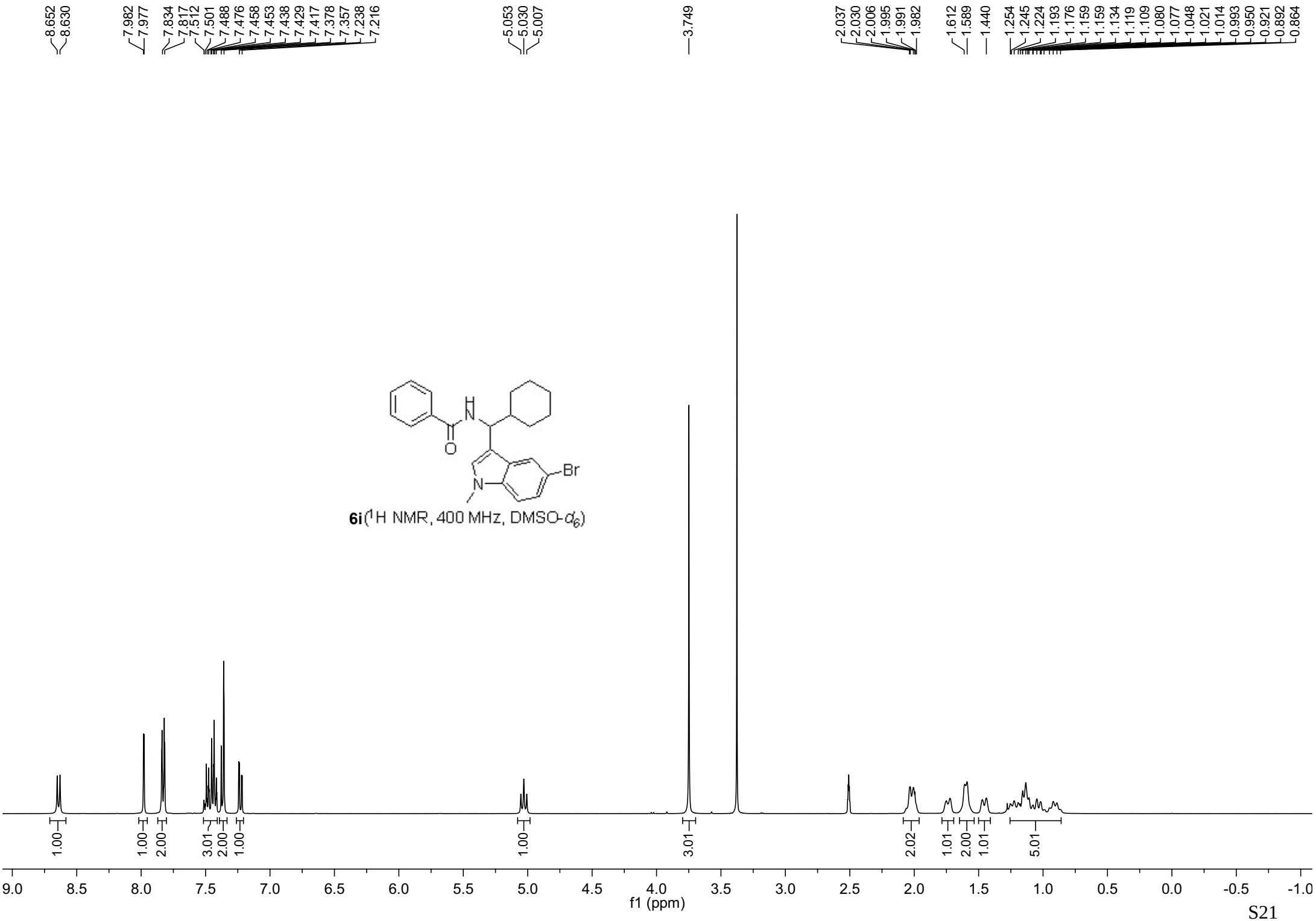


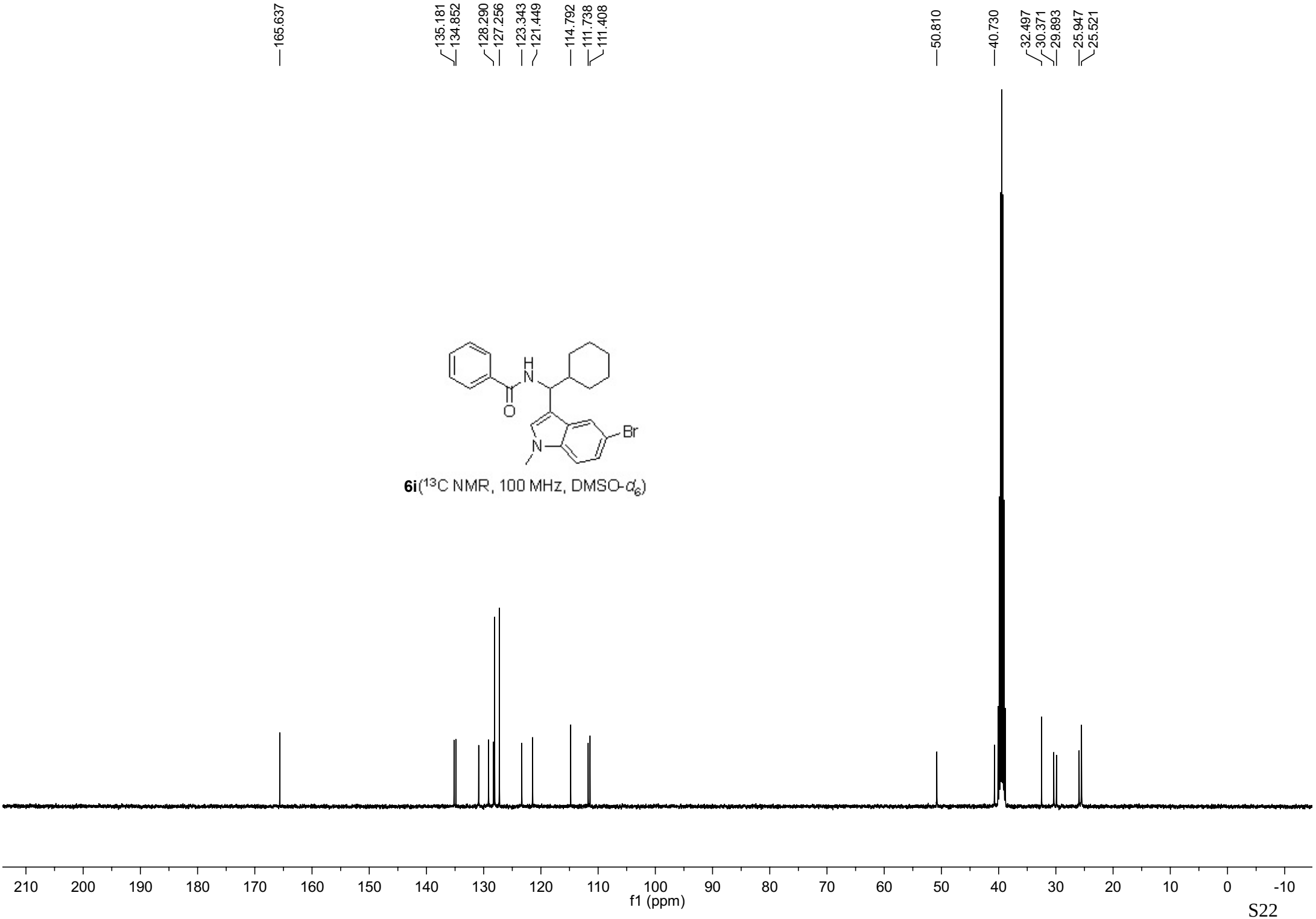
6g (^{13}C NMR, 100 MHz, DMSO-d_6)


11.492
9.988
8.696
8.674
8.429
8.425
7.858
7.855
7.851
7.838
7.834
7.642
7.638
7.621
7.617
7.521
7.514
7.500
7.492
7.474
7.453
7.438
7.433
7.416
5.232
5.209
5.186
2.111
2.103
2.080
2.076
2.068
2.051
2.044
2.023
2.009
1.758
1.728
1.611
1.607
1.589
1.482
1.450
1.255
1.248
1.230
1.223
1.193
1.162
1.136
1.113
1.089
1.083
1.064
1.056
1.034
1.025
1.002
0.973
0.943
H
N
O
CHO
HN
6h(1H NMR, 400 MHz, DMSO-d6)
1.00
1.00
1.00
1.00
2.00
1.00
5.02
1.00
2.01
1.00
2.00
1.01
5.01
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

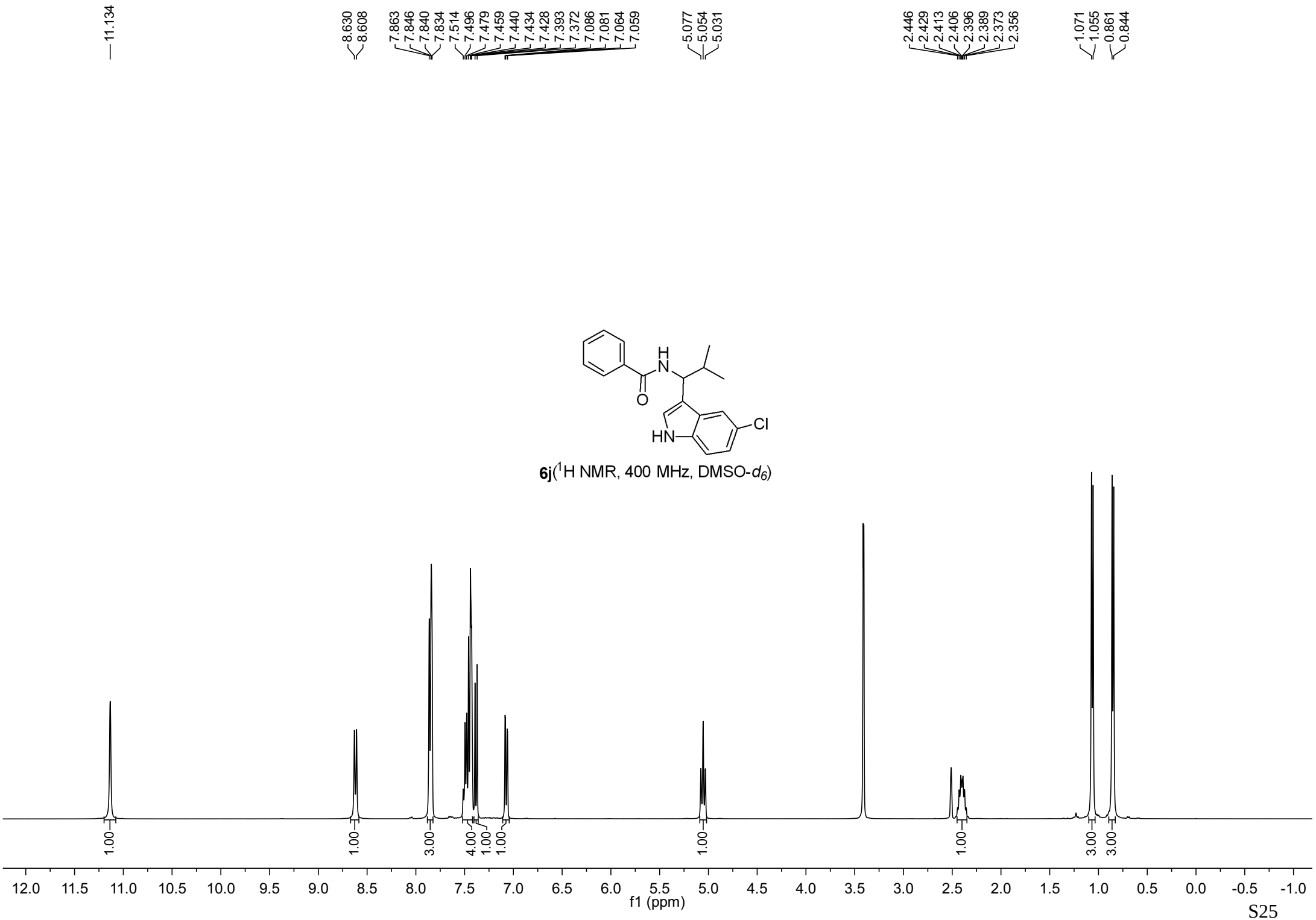


6h(^{13}C NMR, 100 MHz, DMSO-d_6)

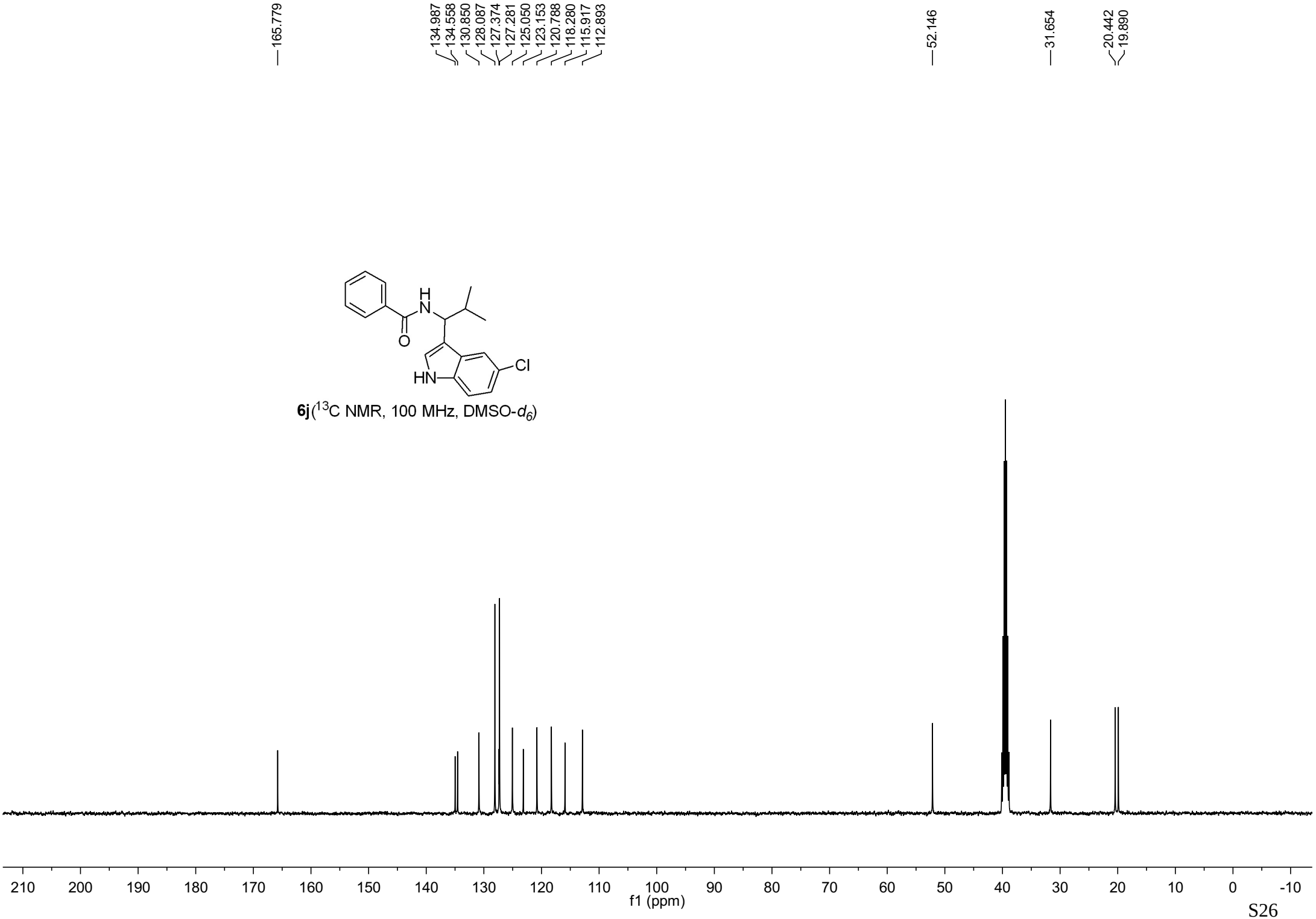

8.652
8.630
7.982
7.977
7.834
7.817
7.512
7.501
7.488
7.476
7.458
7.453
7.438
7.429
7.417
7.378
7.357
7.238
7.216
5.053
5.030
5.007
3.749
2.037
2.030
2.006
1.995
1.991
1.982
1.612
1.589
1.440
1.254
1.245
1.224
1.193
1.176
1.159
1.159
1.134
1.119
1.109
1.080
1.077
1.048
1.021
1.014
0.993
0.950
0.921
0.892
0.864
H
N
O
Br
N
6i(1H NMR, 400 MHz, DMSO-d6)
1.00
1.00
2.00
3.01
2.00
1.00
1.00
3.01
2.02
1.01
2.00
1.01
5.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



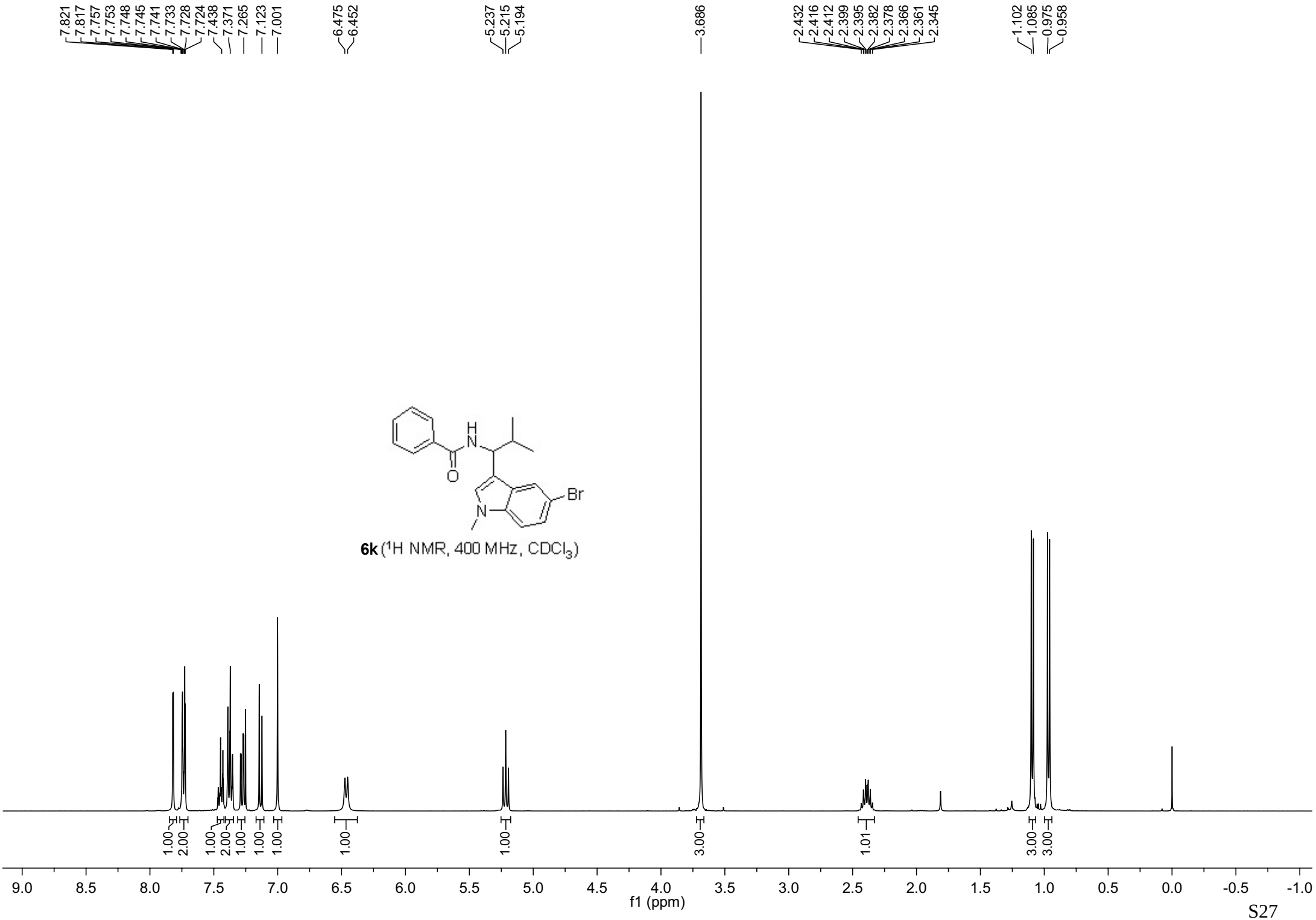
6i(^{13}C NMR, 100 MHz, DMSO-d_6)

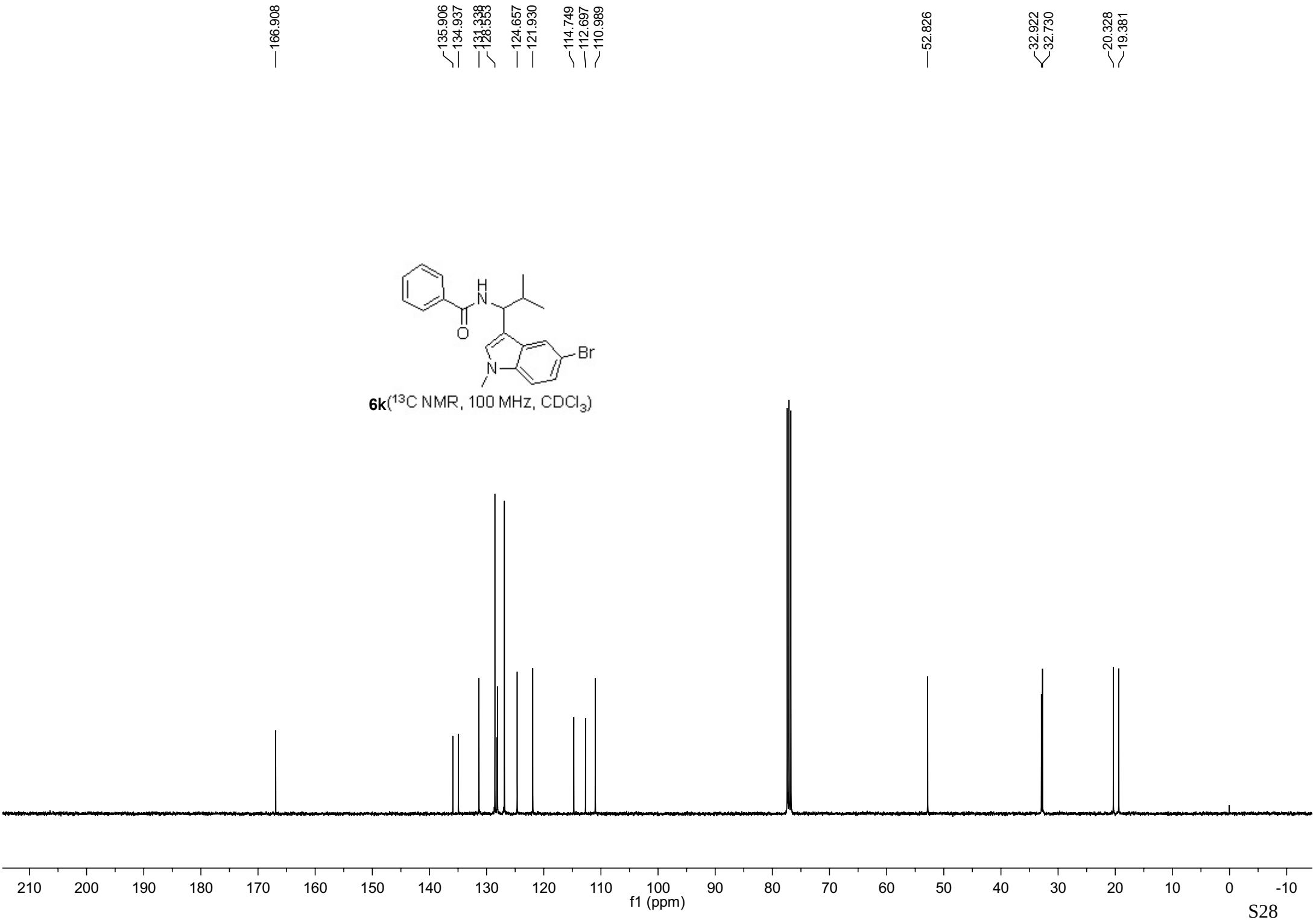


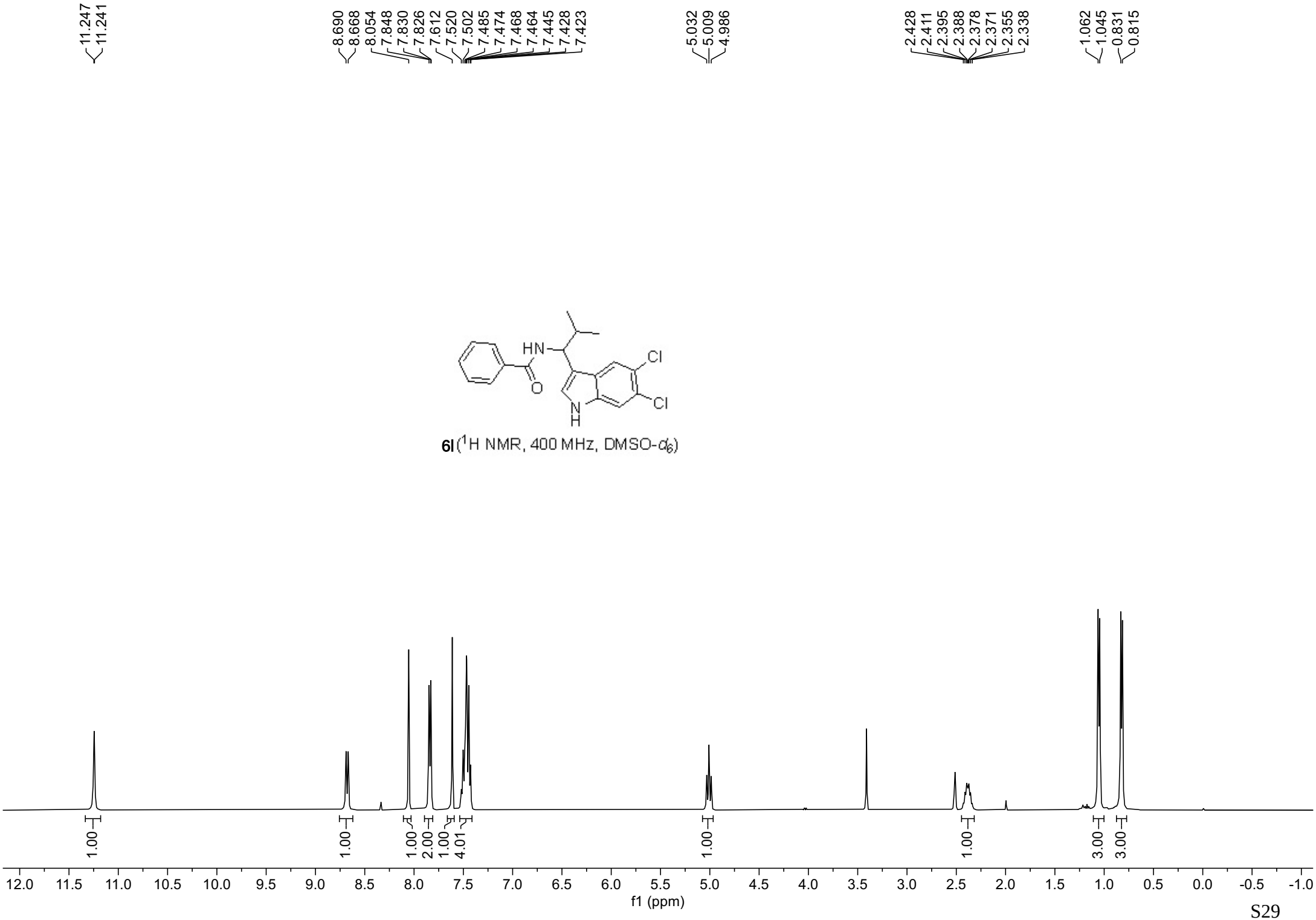
6j(^{1}H NMR, 400 MHz, DMSO-d_6)



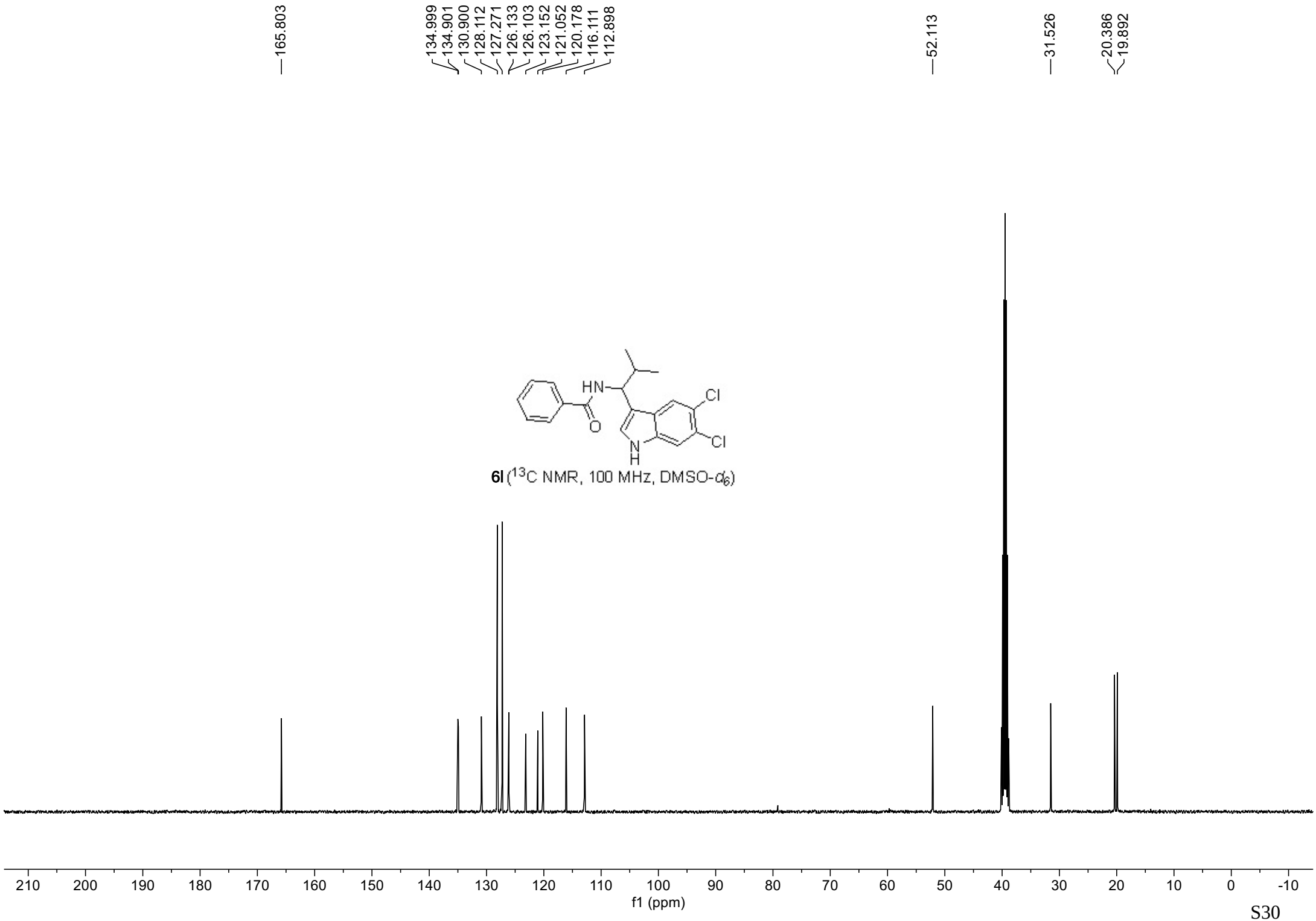
6j (^{13}C NMR, 100 MHz, DMSO-d_6)


7.821
7.817
7.757
7.753
7.748
7.745
7.741
7.733
7.728
7.724
7.438
7.371
7.265
7.123
7.001
6.475
6.452
5.237
5.215
5.194
3.686
2.432
2.416
2.412
2.399
2.395
2.382
2.378
2.366
2.361
2.345
1.102
1.085
0.975
0.958
H
N
O
N
Br
6k (1H NMR, 400 MHz, CDCl3)
1.00
2.00
1.00
2.00
1.00
1.00
1.00
1.00
1.00
3.00
1.01
3.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

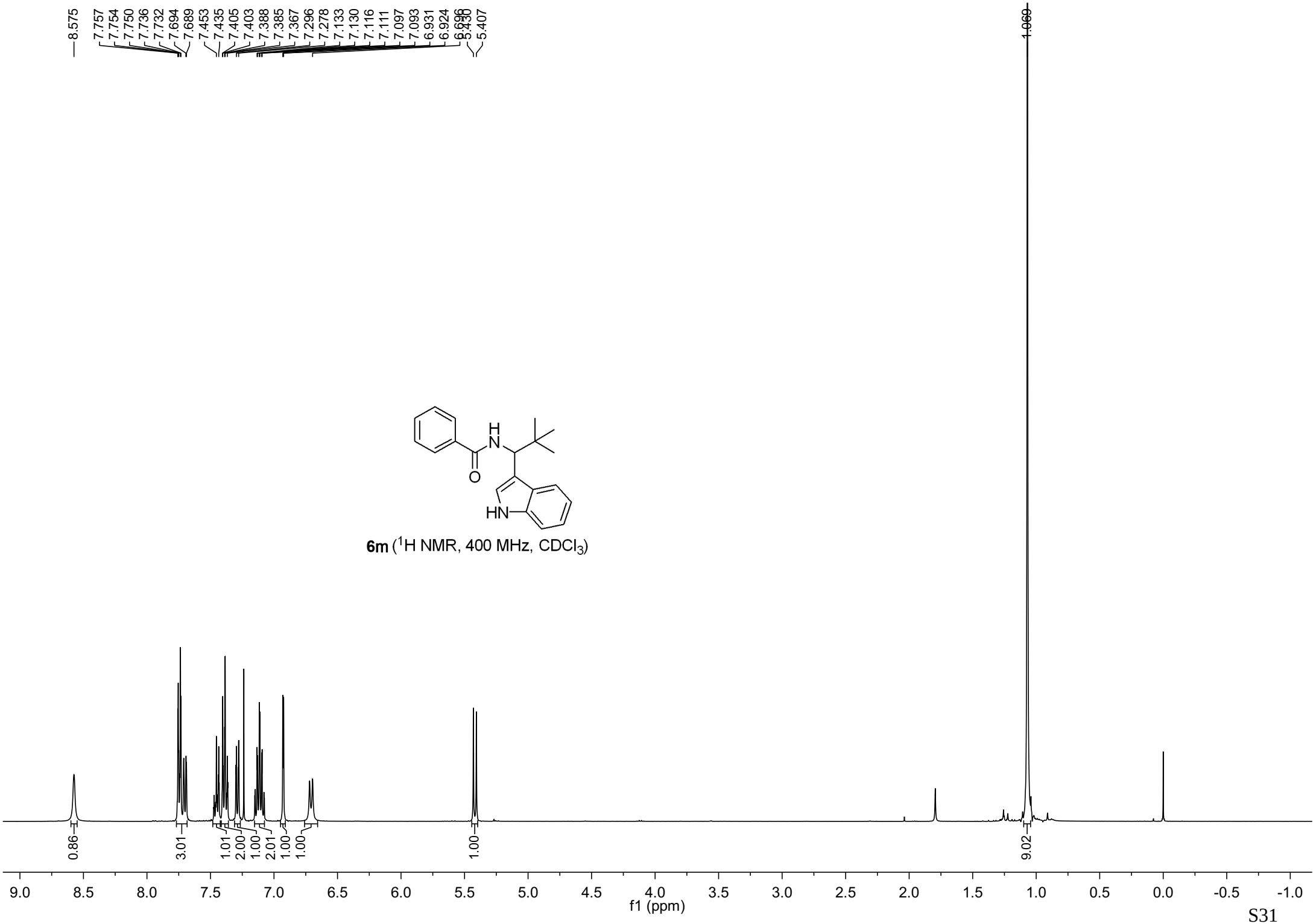

166.908
135.906
134.937
131.338
128.553
124.657
121.930
114.749
112.697
110.989
52.826
32.922
32.730
20.328
19.381
H
N
O
N
Br
6k(13C NMR, 100 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



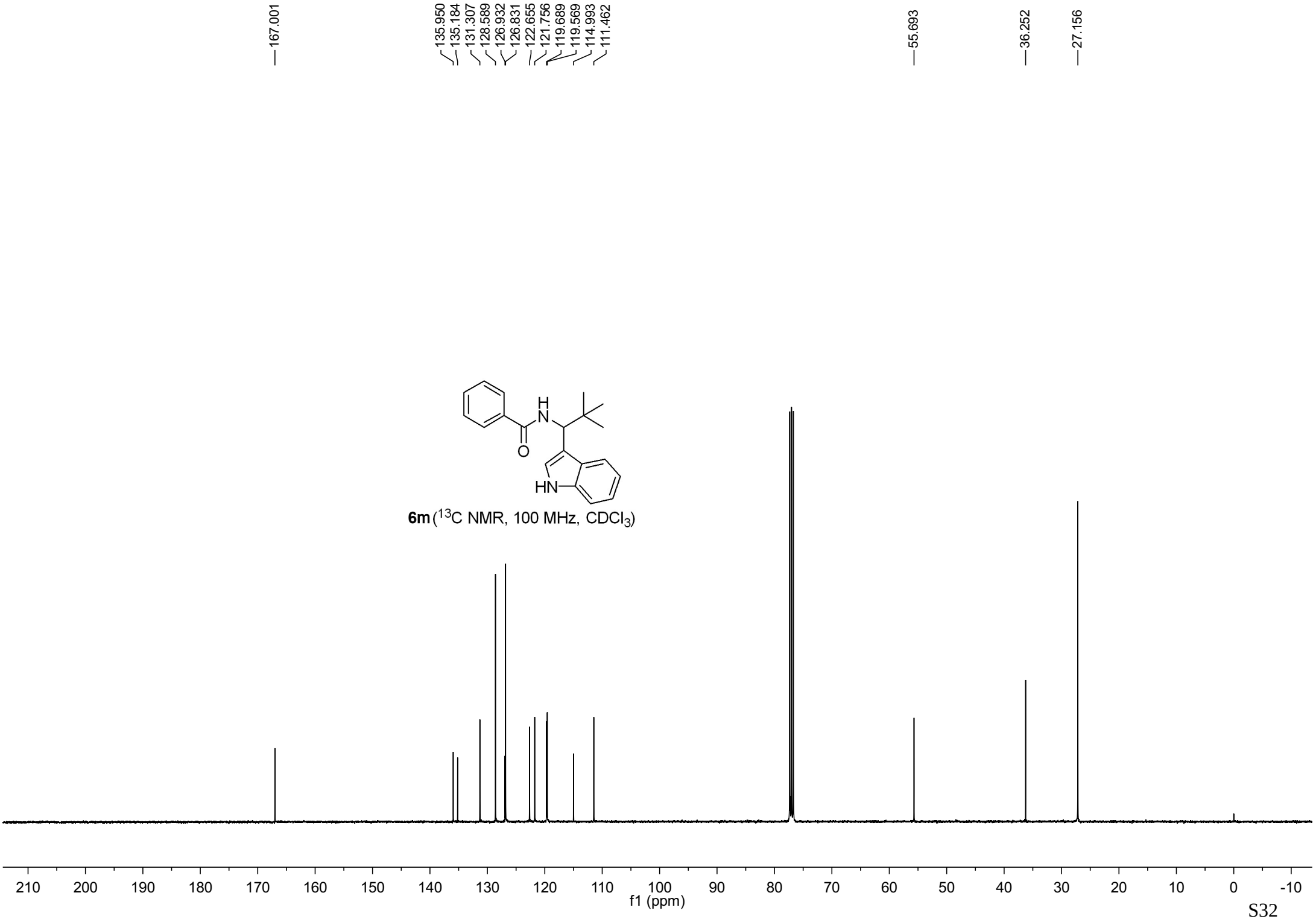
6l (^{1}H NMR, 400 MHz, DMSO-d_6)



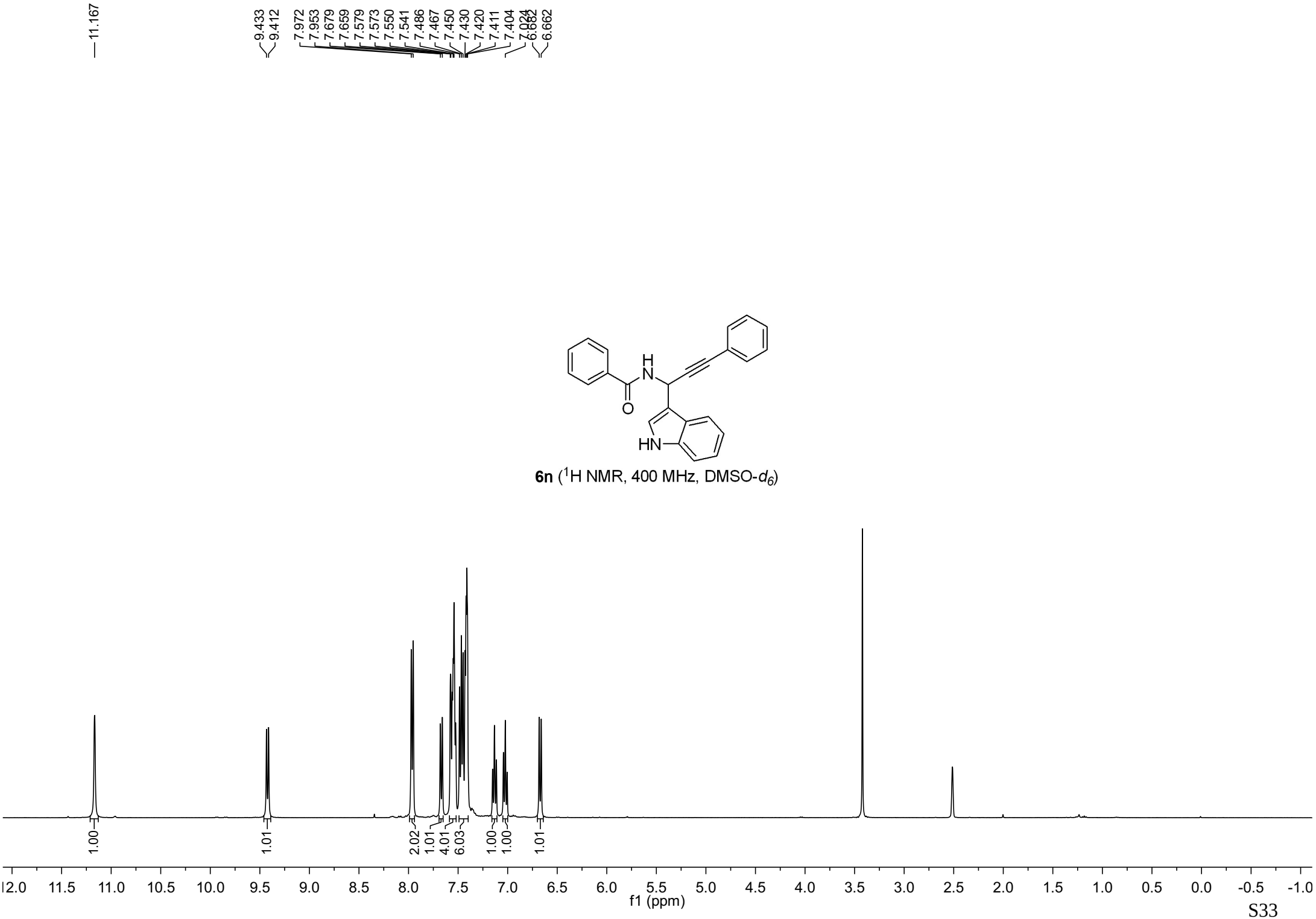
6l (^{13}C NMR, 100 MHz, DMSO-d_6)



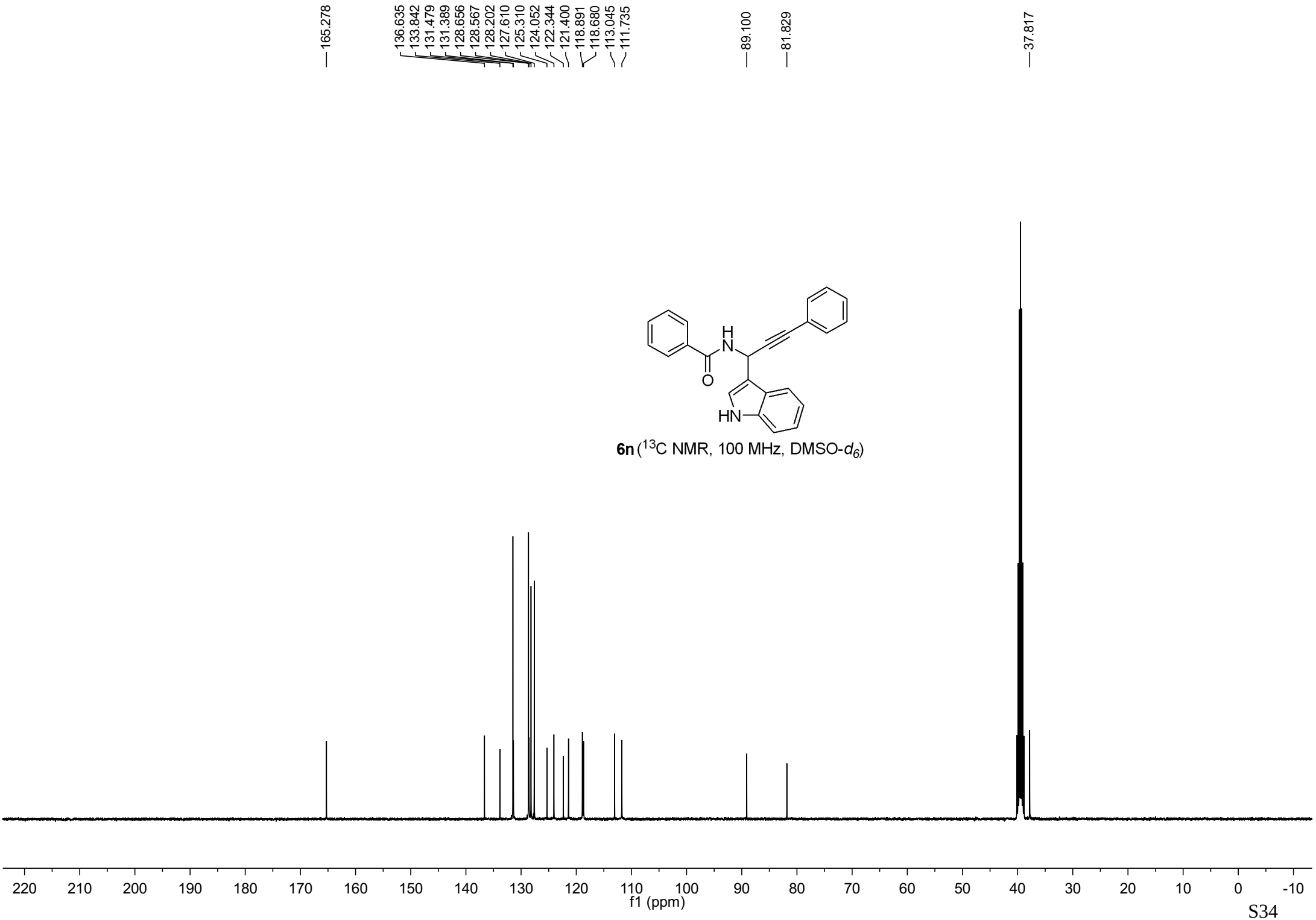
6m (^{1}H NMR, 400 MHz, $CDCl_3$)



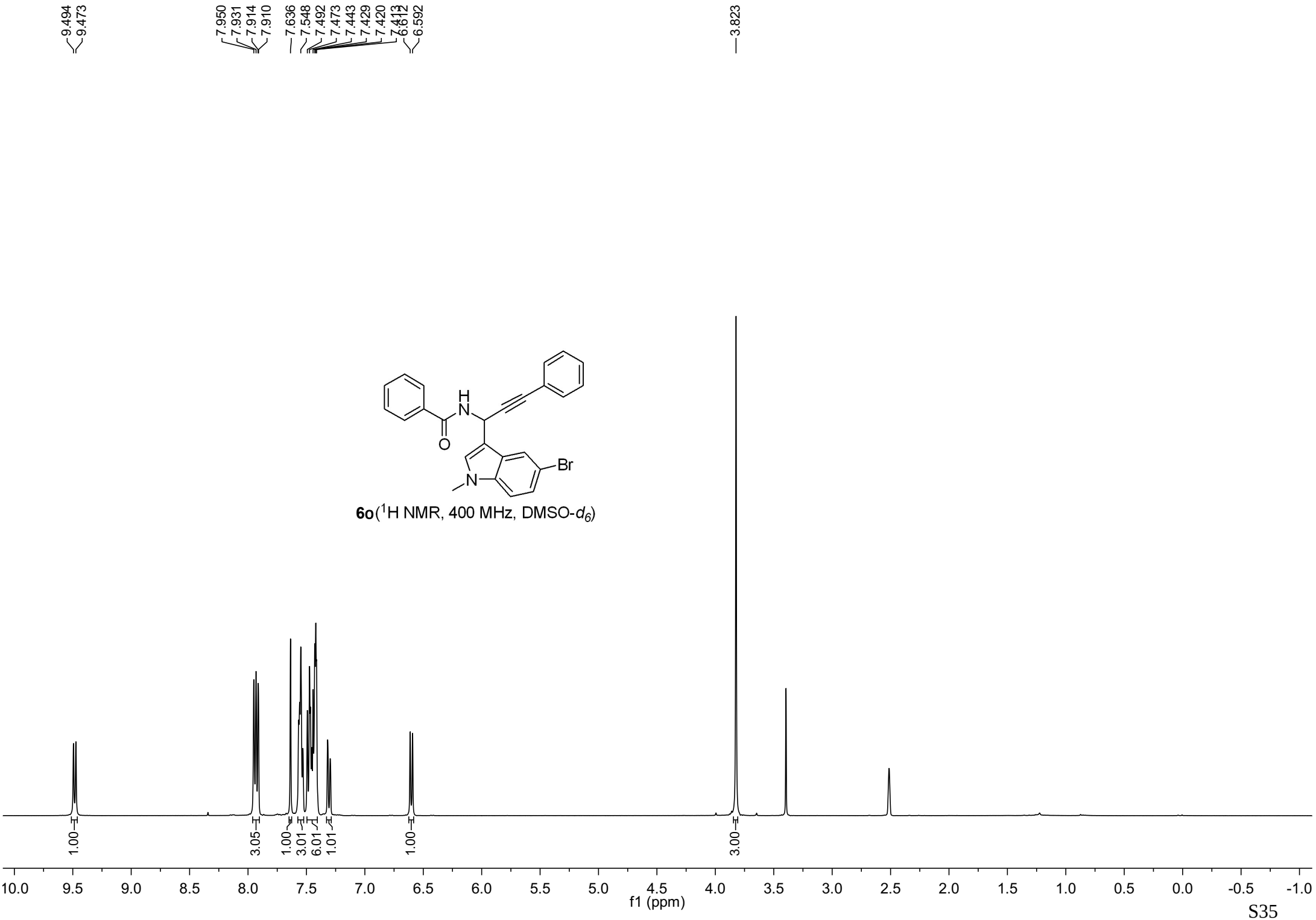
6m (^{13}C NMR, 100 MHz, $CDCl_3$)



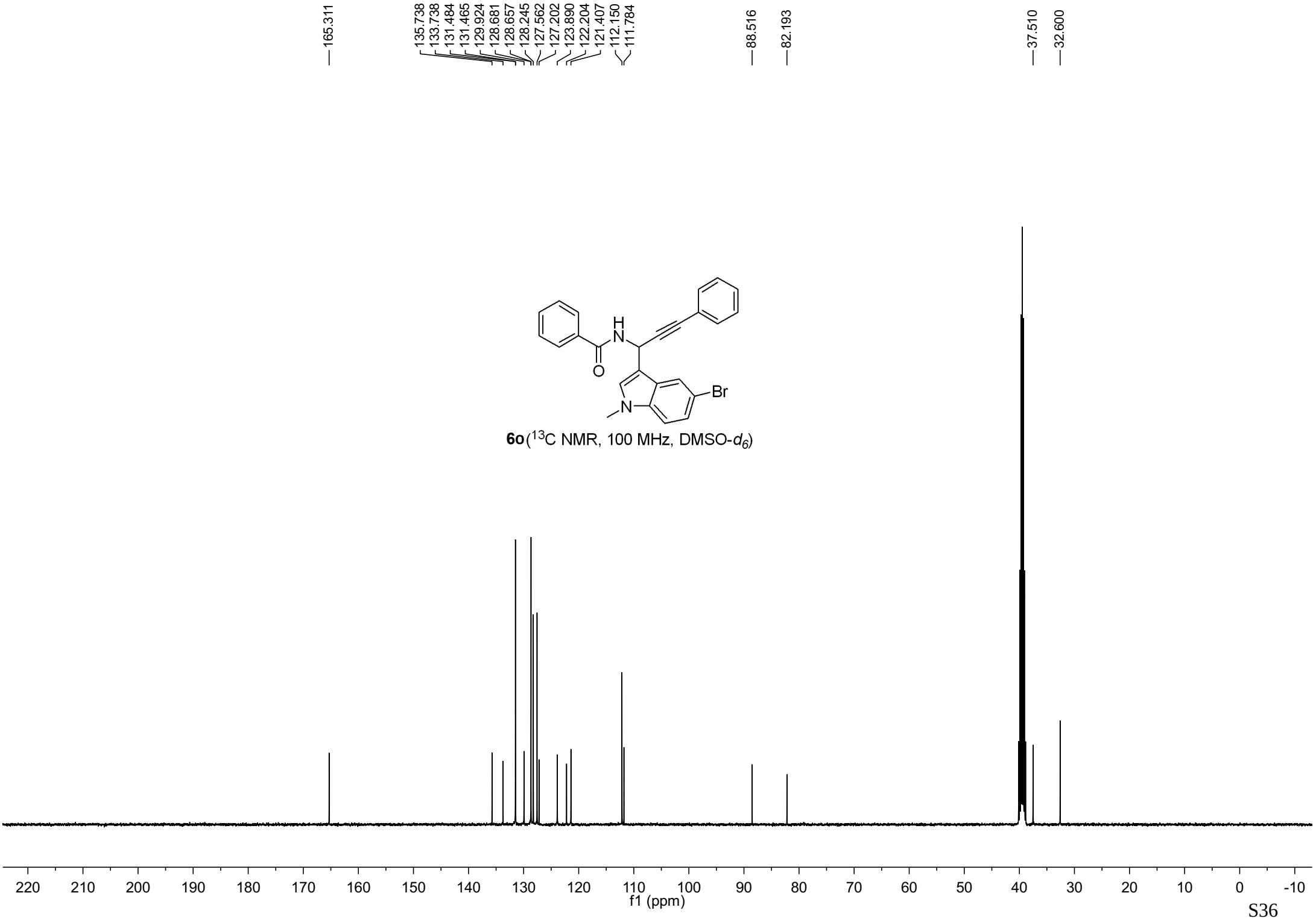
6n (^{1}H NMR, 400 MHz, DMSO-d_6)



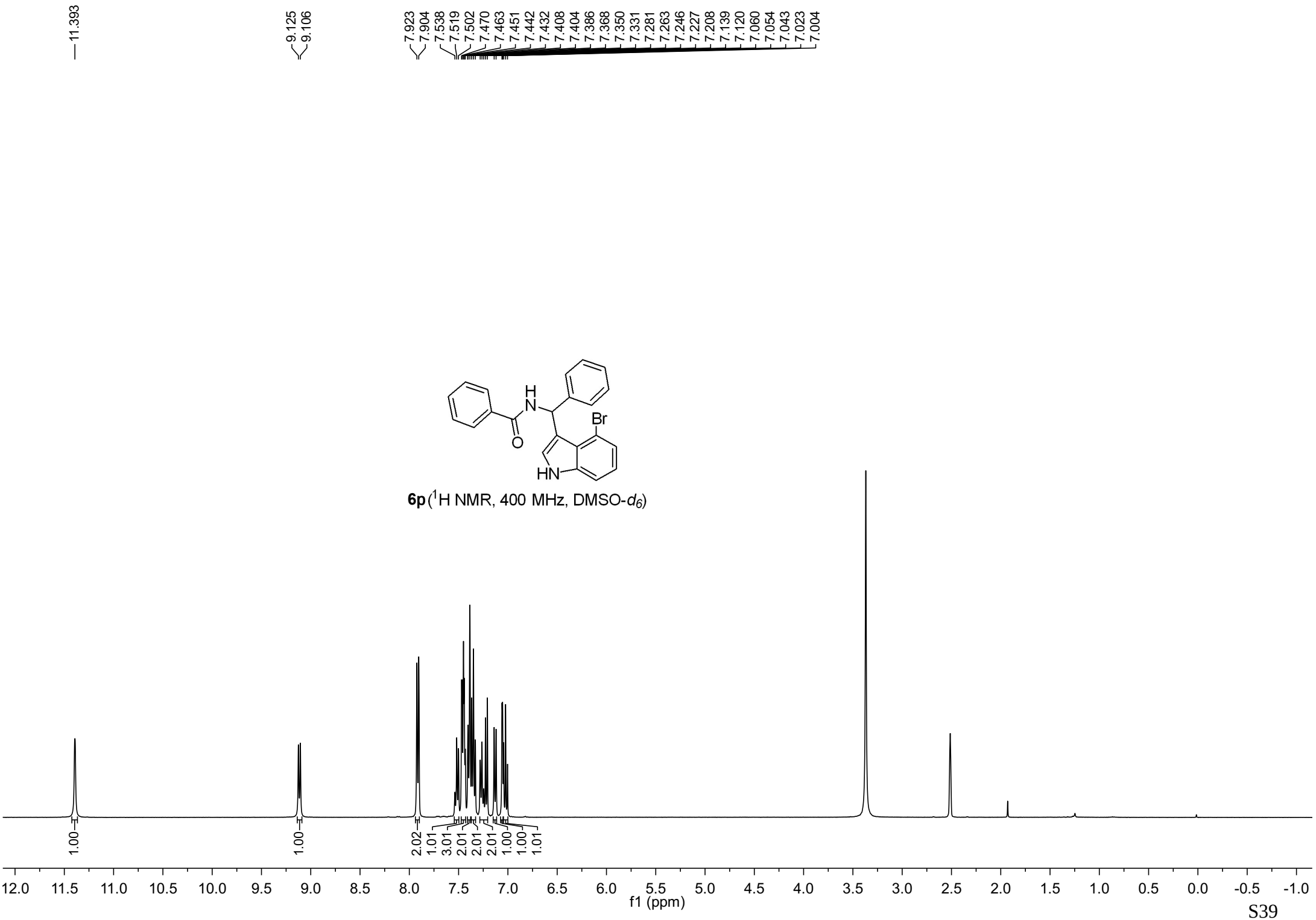
6n (^{13}C NMR, 100 MHz, DMSO-d_6)



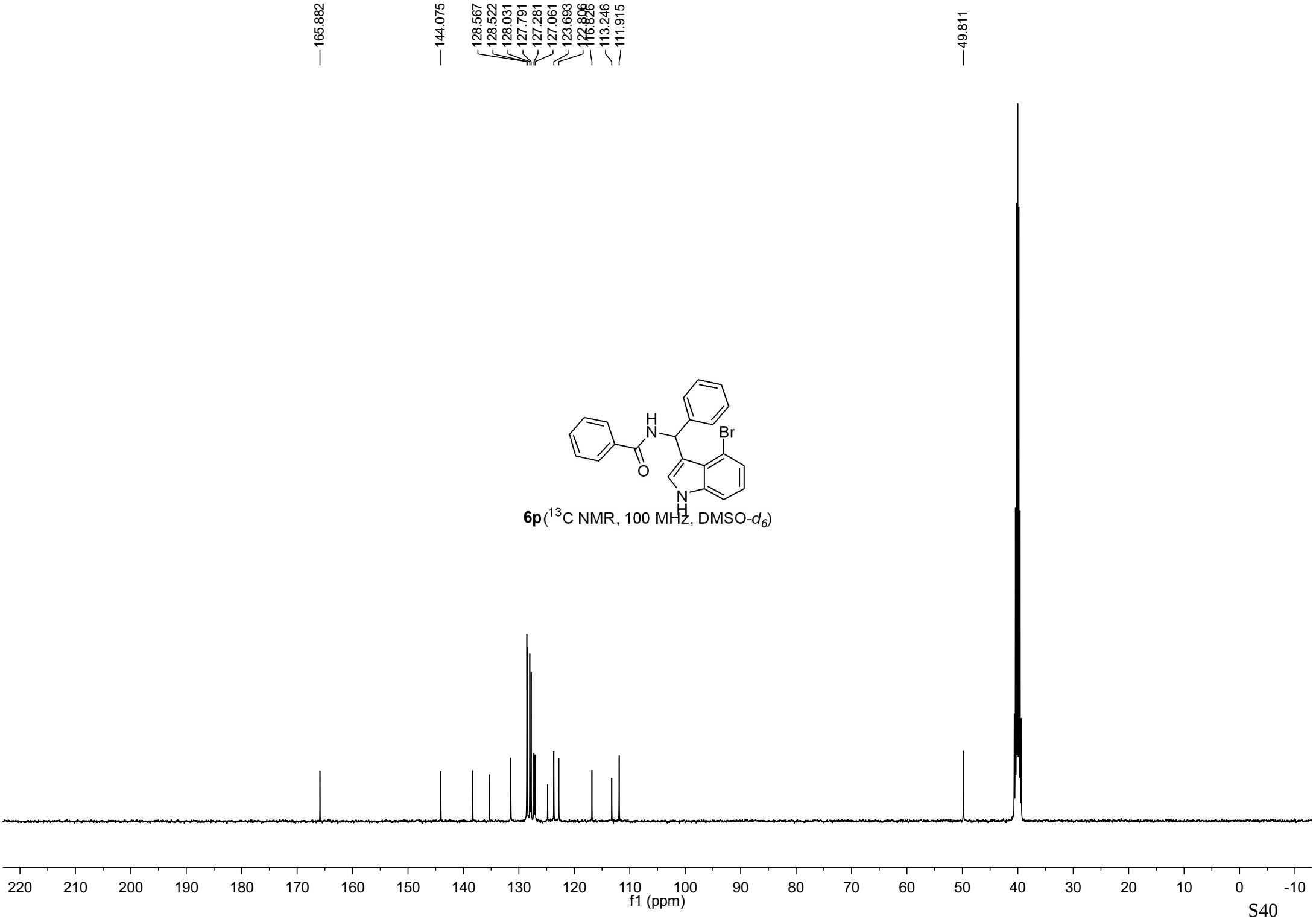
6o(^{1}H NMR, 400 MHz, DMSO-d_6)



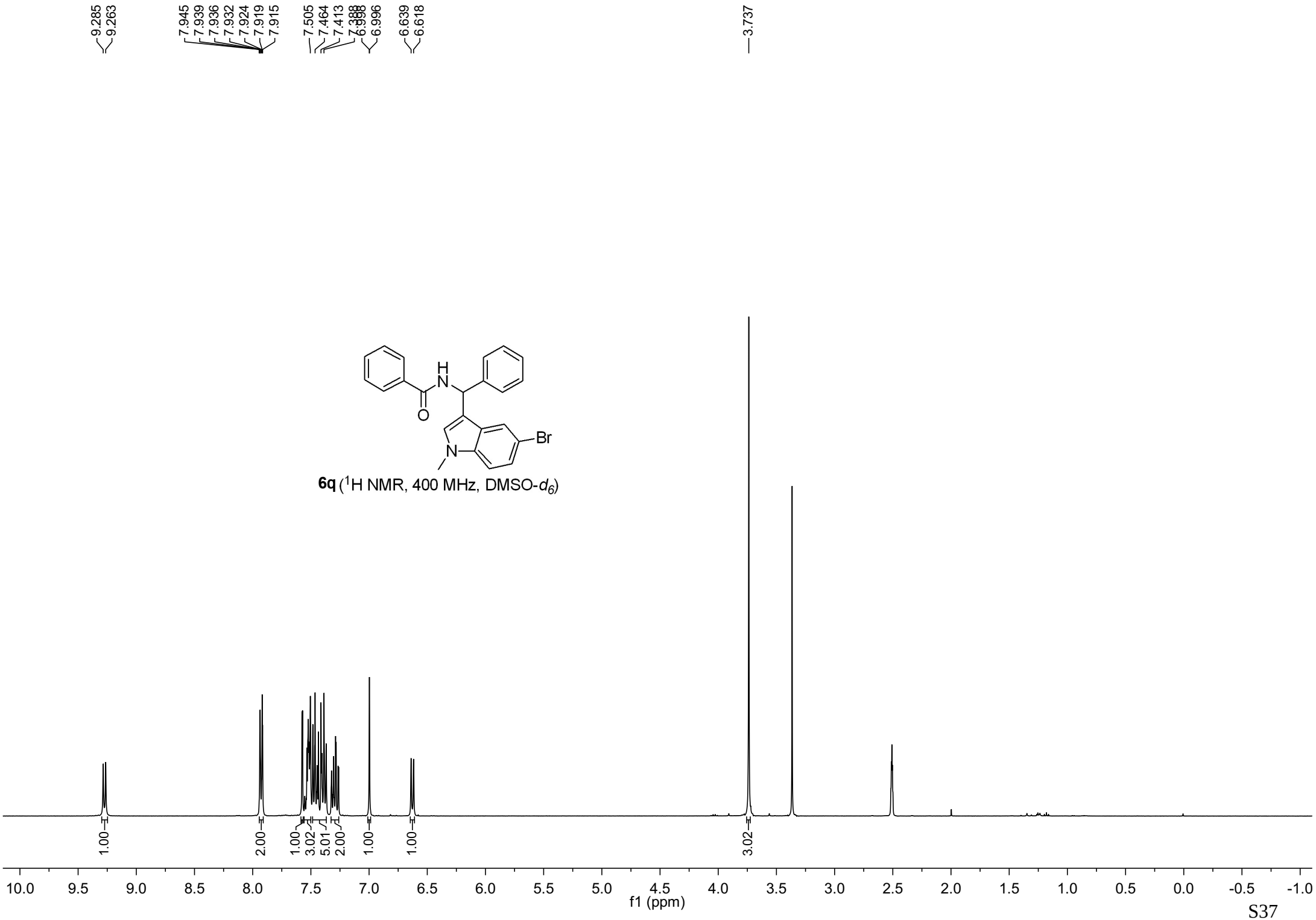
6o(^{13}C NMR, 100 MHz, DMSO-d_6)



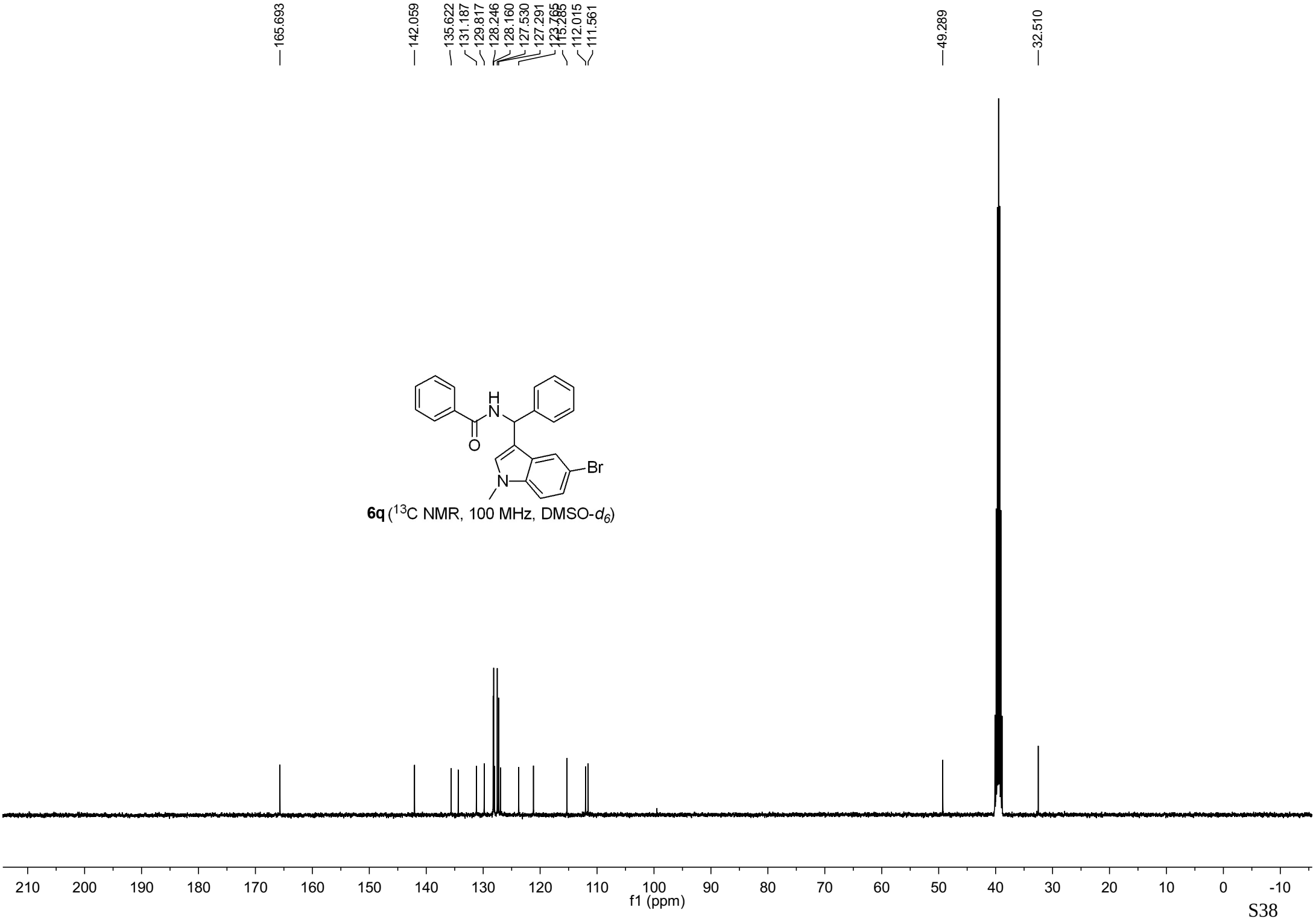
6p (^{1}H NMR, 400 MHz, DMSO-d_6)



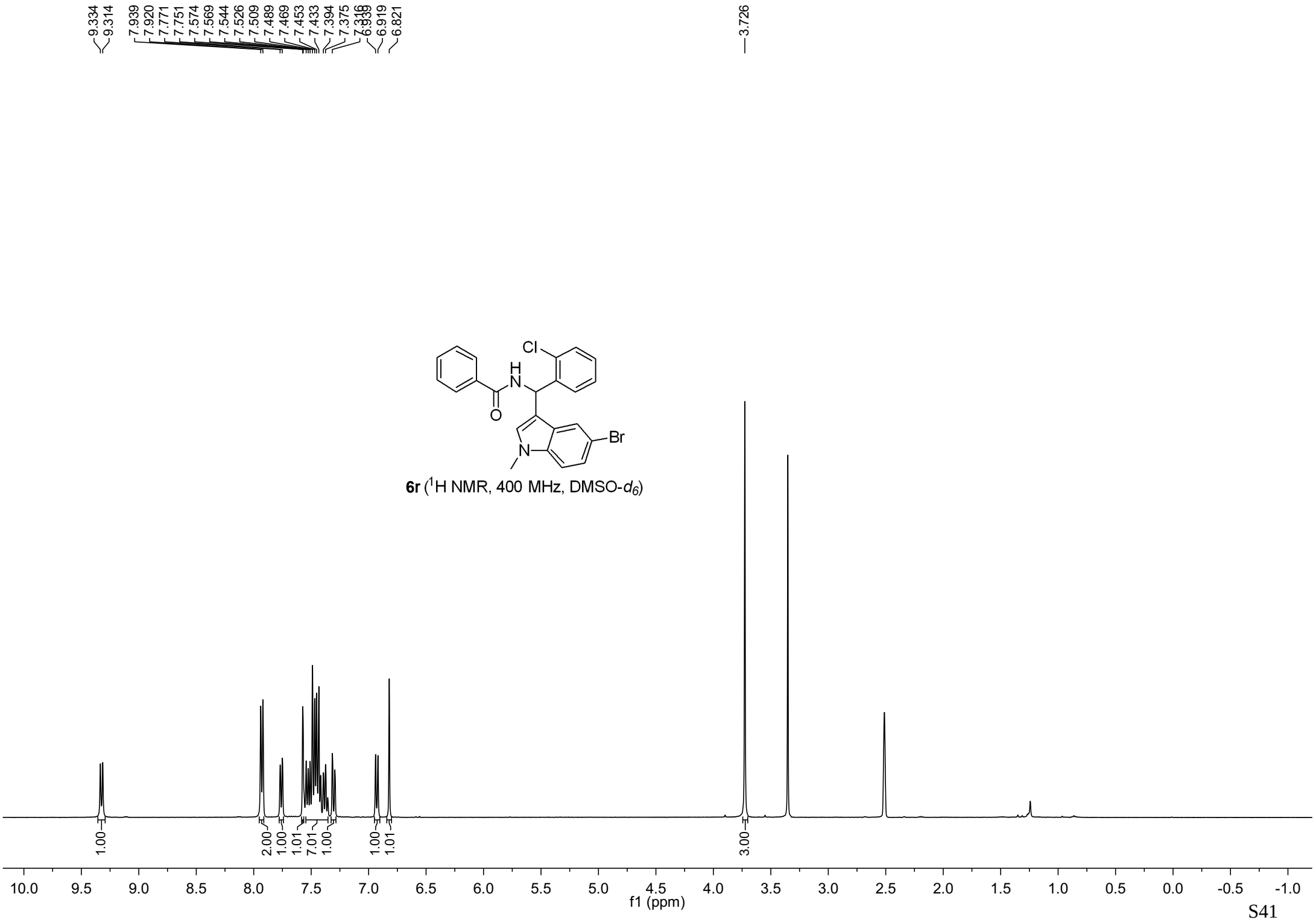
6p(^{13}C NMR, 100 MHz, DMSO-d_6)



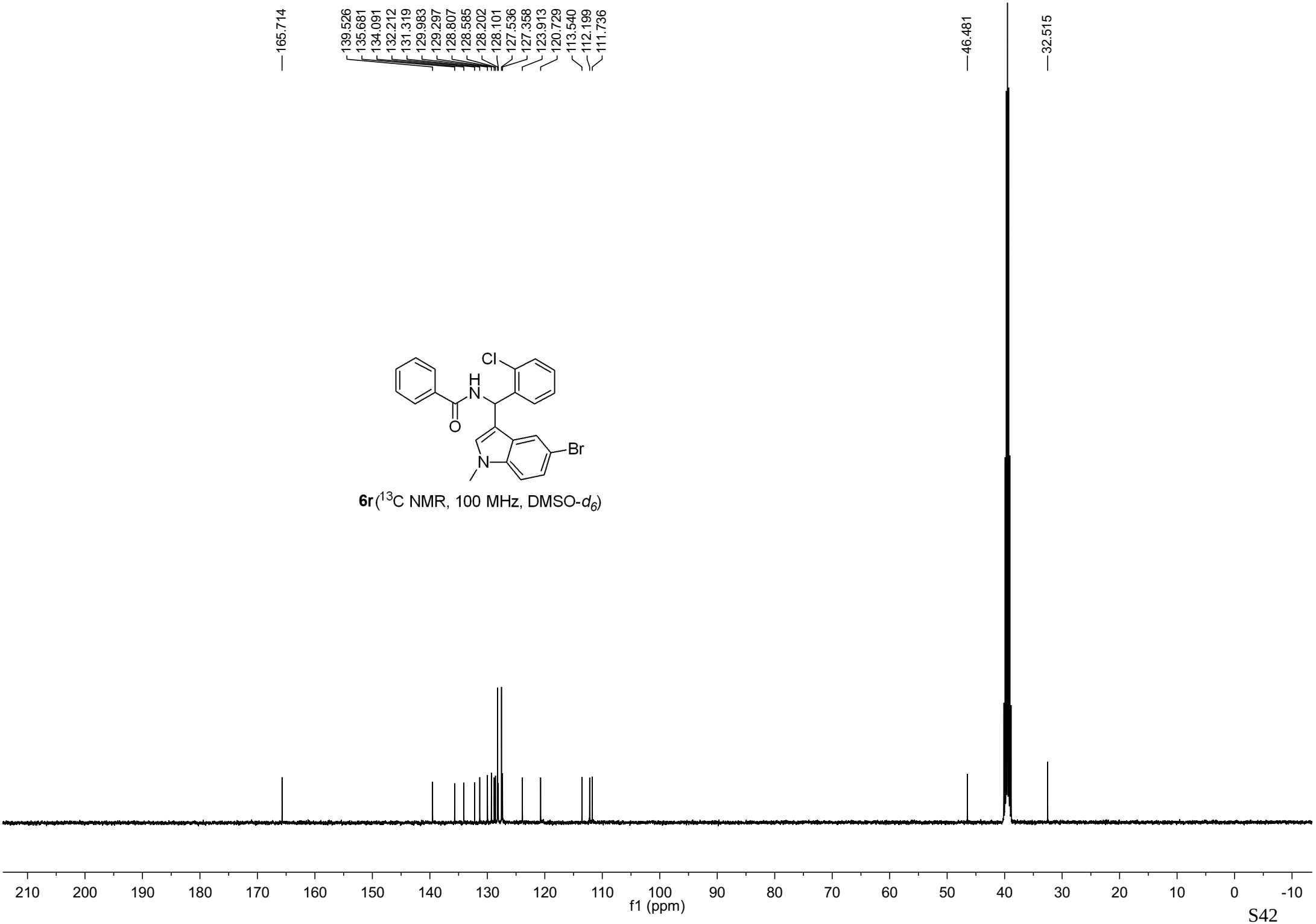
6q (^{1}H NMR, 400 MHz, DMSO-d_6)



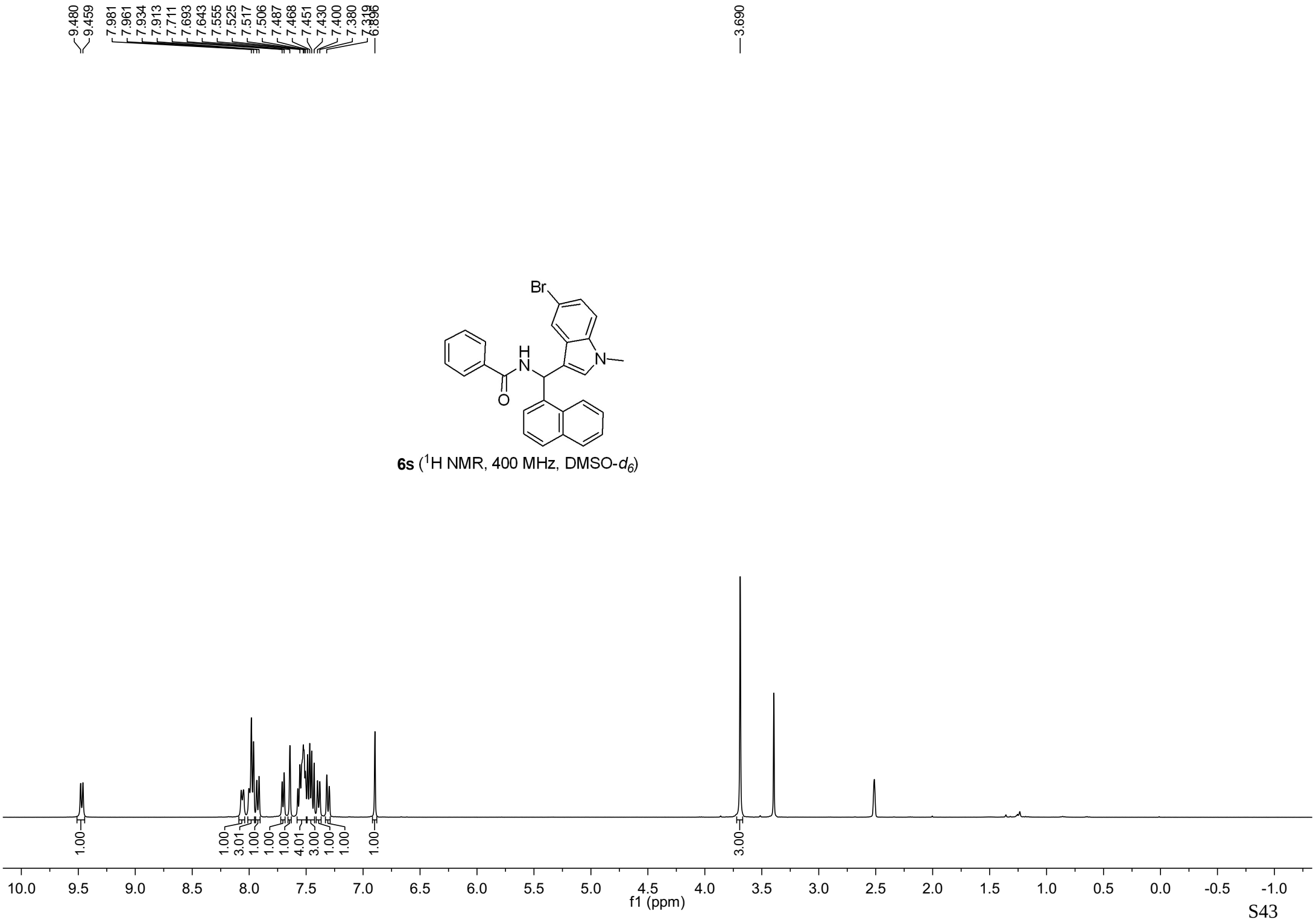
6q (^{13}C NMR, 100 MHz, DMSO-d_6)



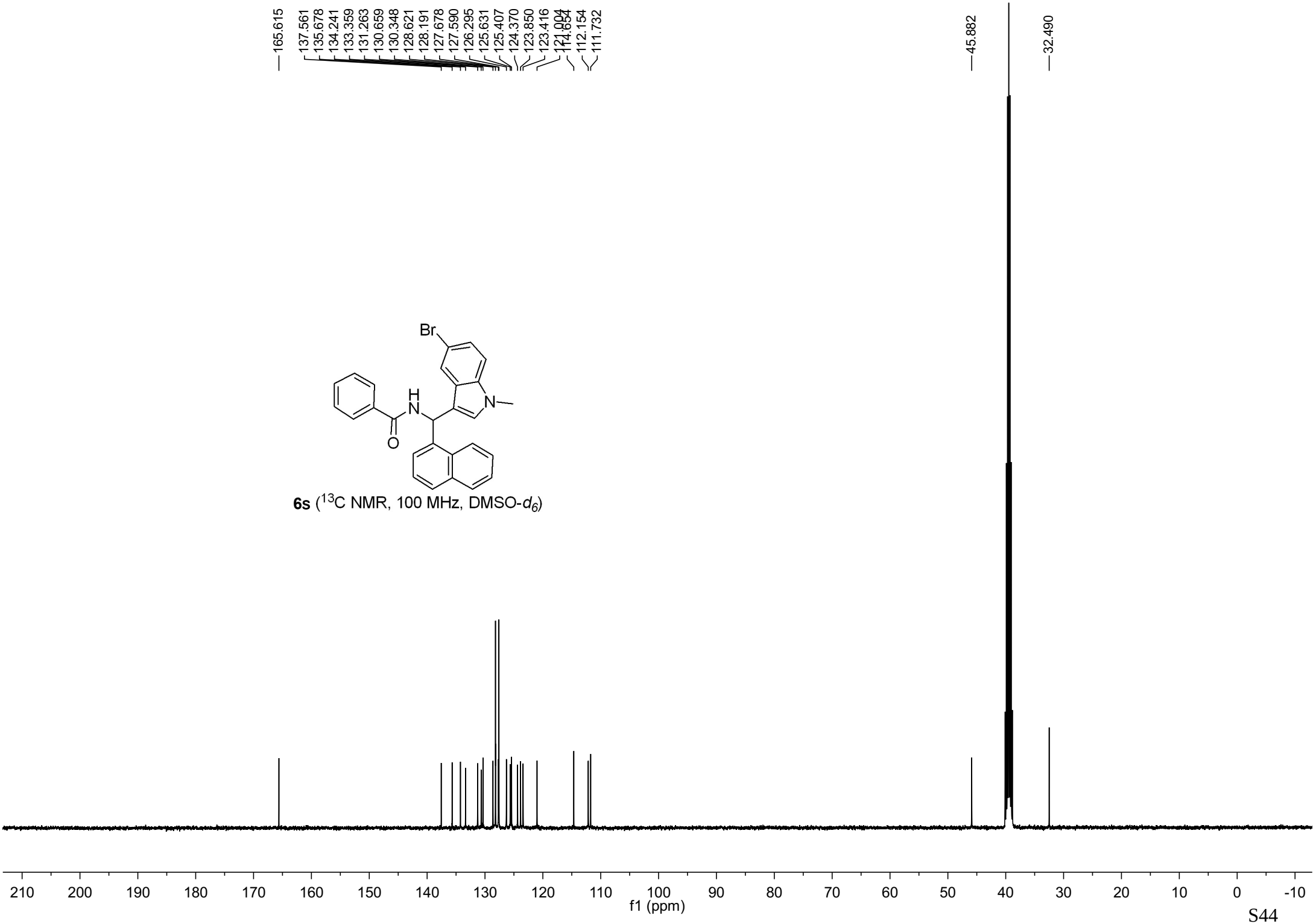
6r (^{1}H NMR, 400 MHz, DMSO-d_6)



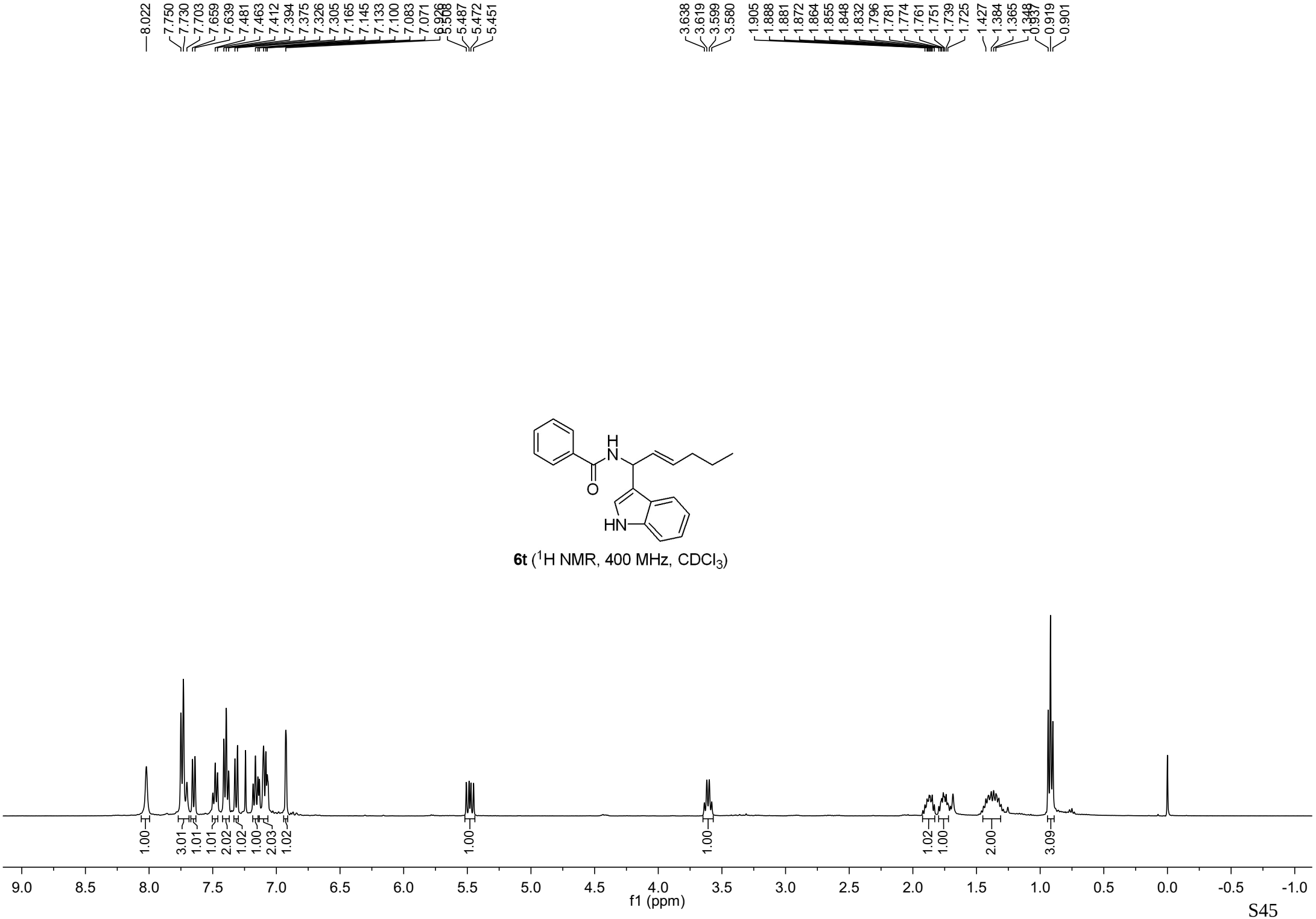
6r (^{13}C NMR, 100 MHz, DMSO-d_6)



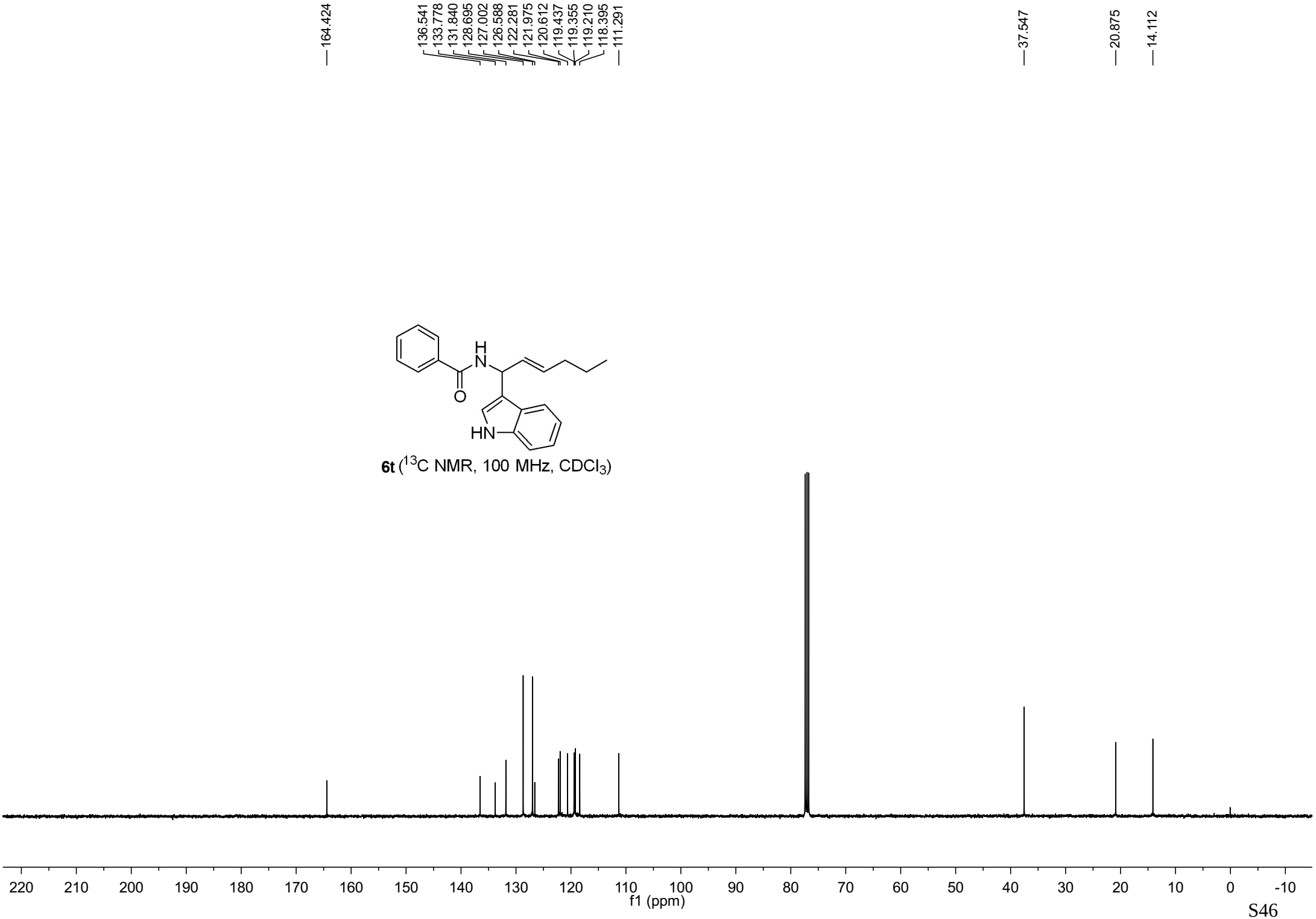
6s (^{1}H NMR, 400 MHz, DMSO-d_6)



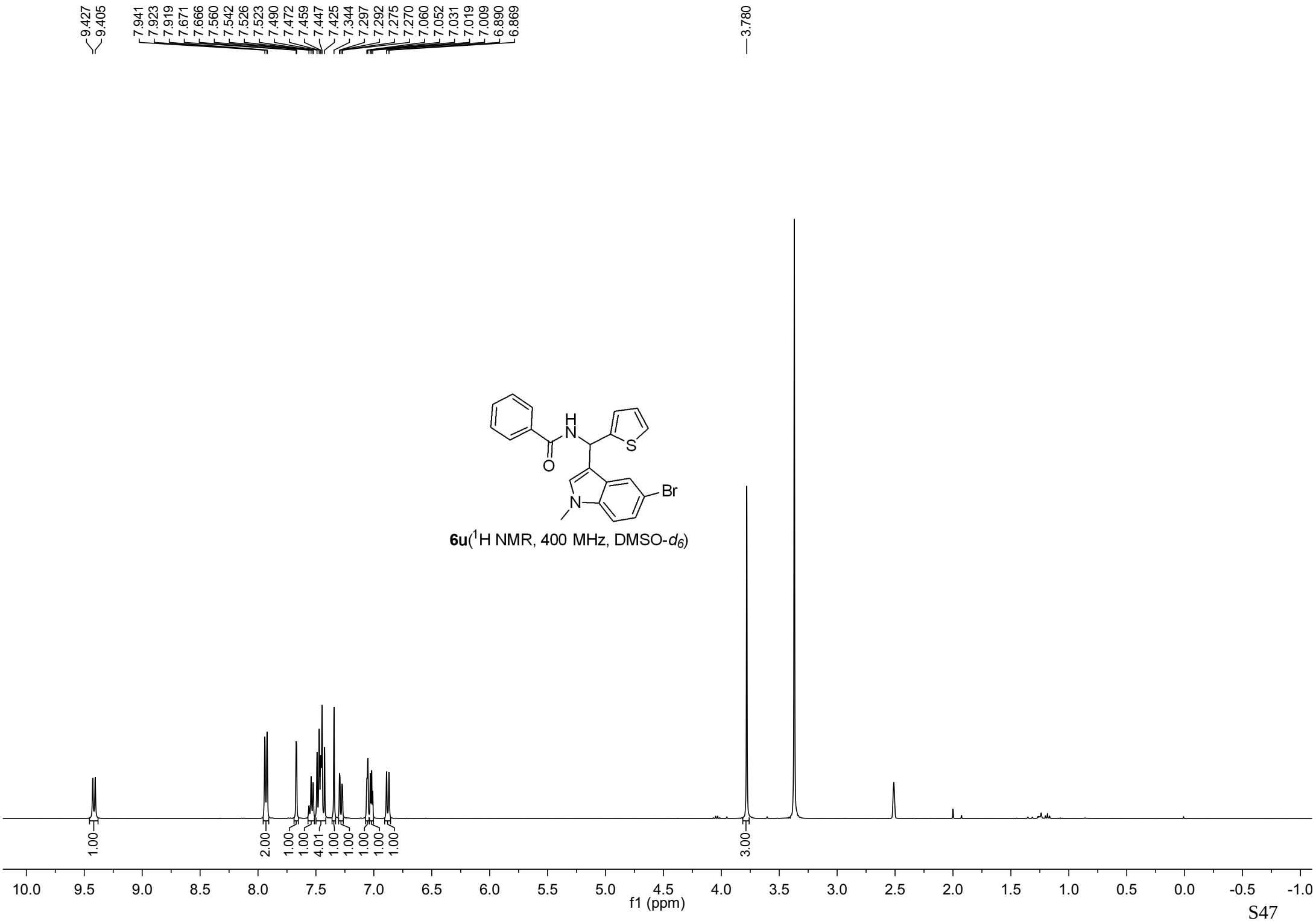
6s (^{13}C NMR, 100 MHz, DMSO-d_6)



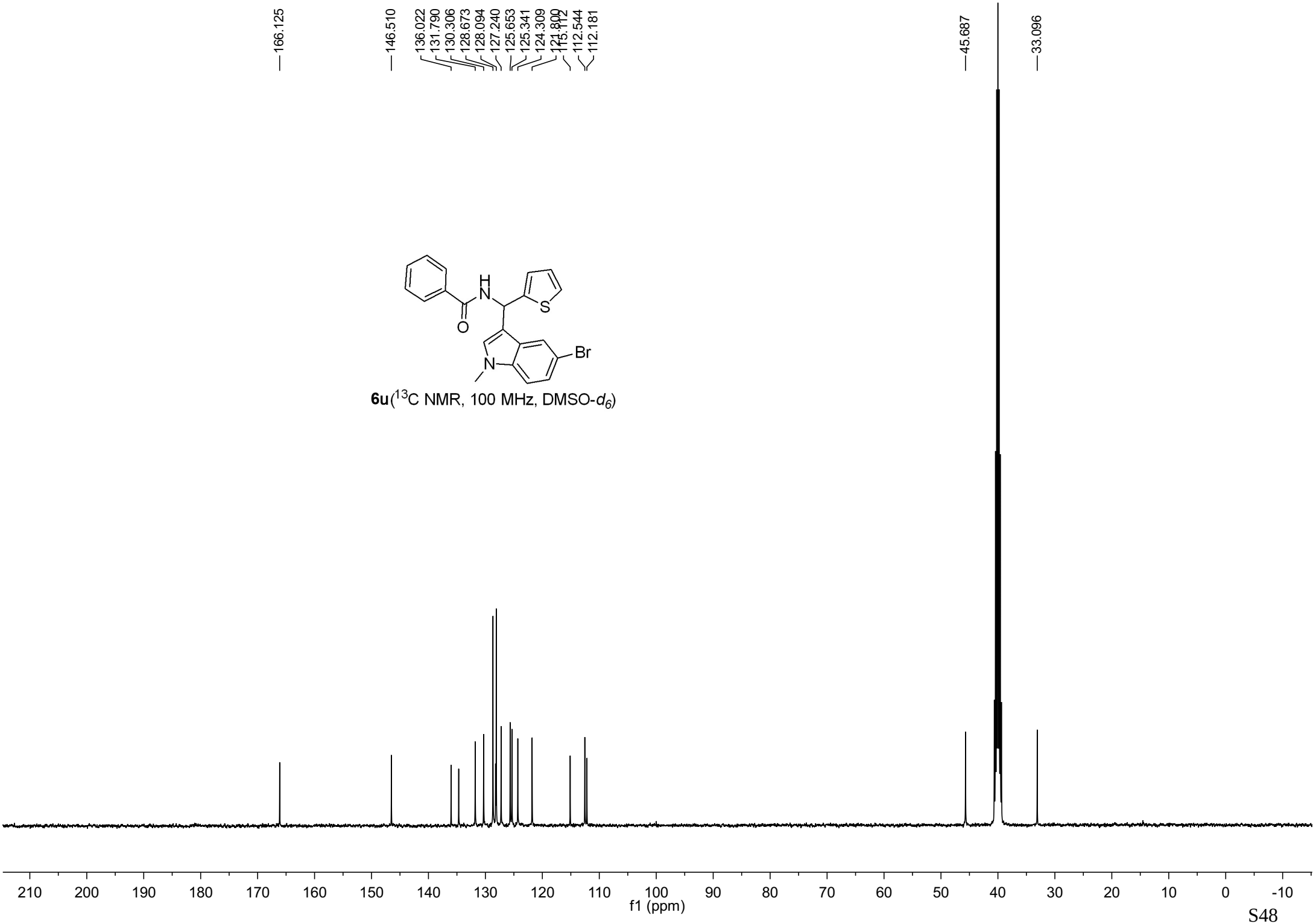
6t (^{1}H NMR, 400 MHz, $CDCl_3$)



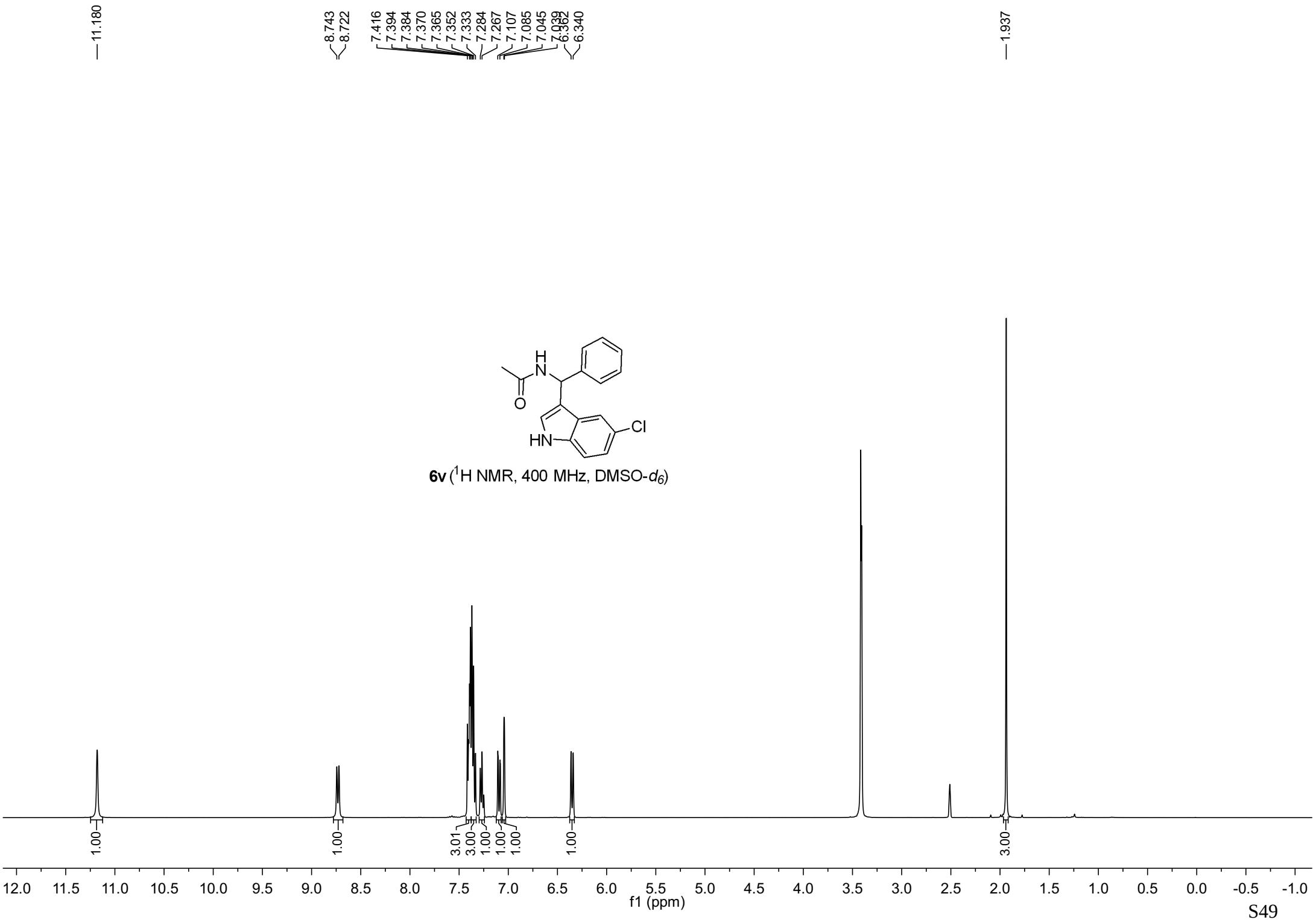
6t (^{13}C NMR, 100 MHz, $CDCl_3$)



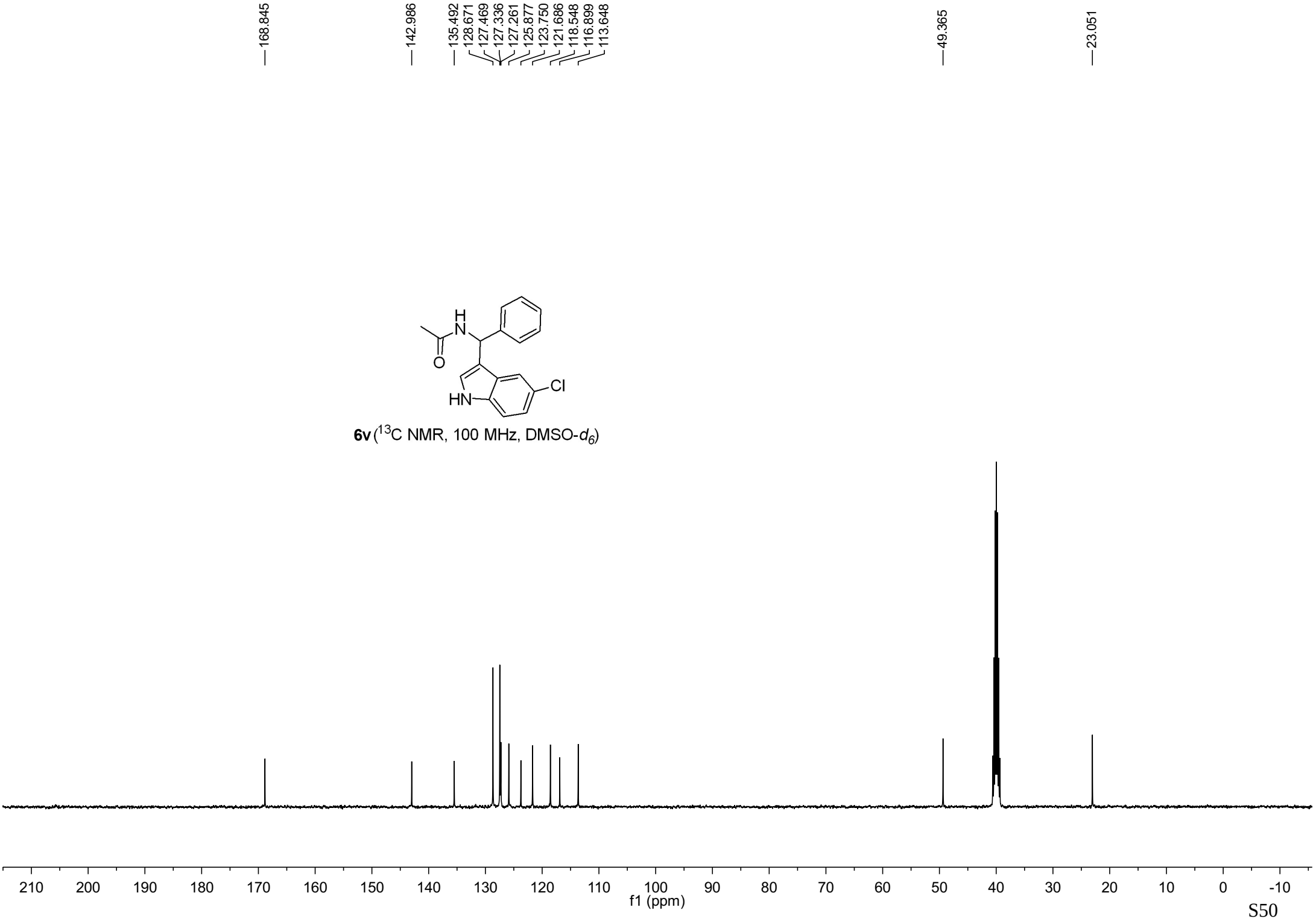
6u(^{1}H NMR, 400 MHz, DMSO-d_6)



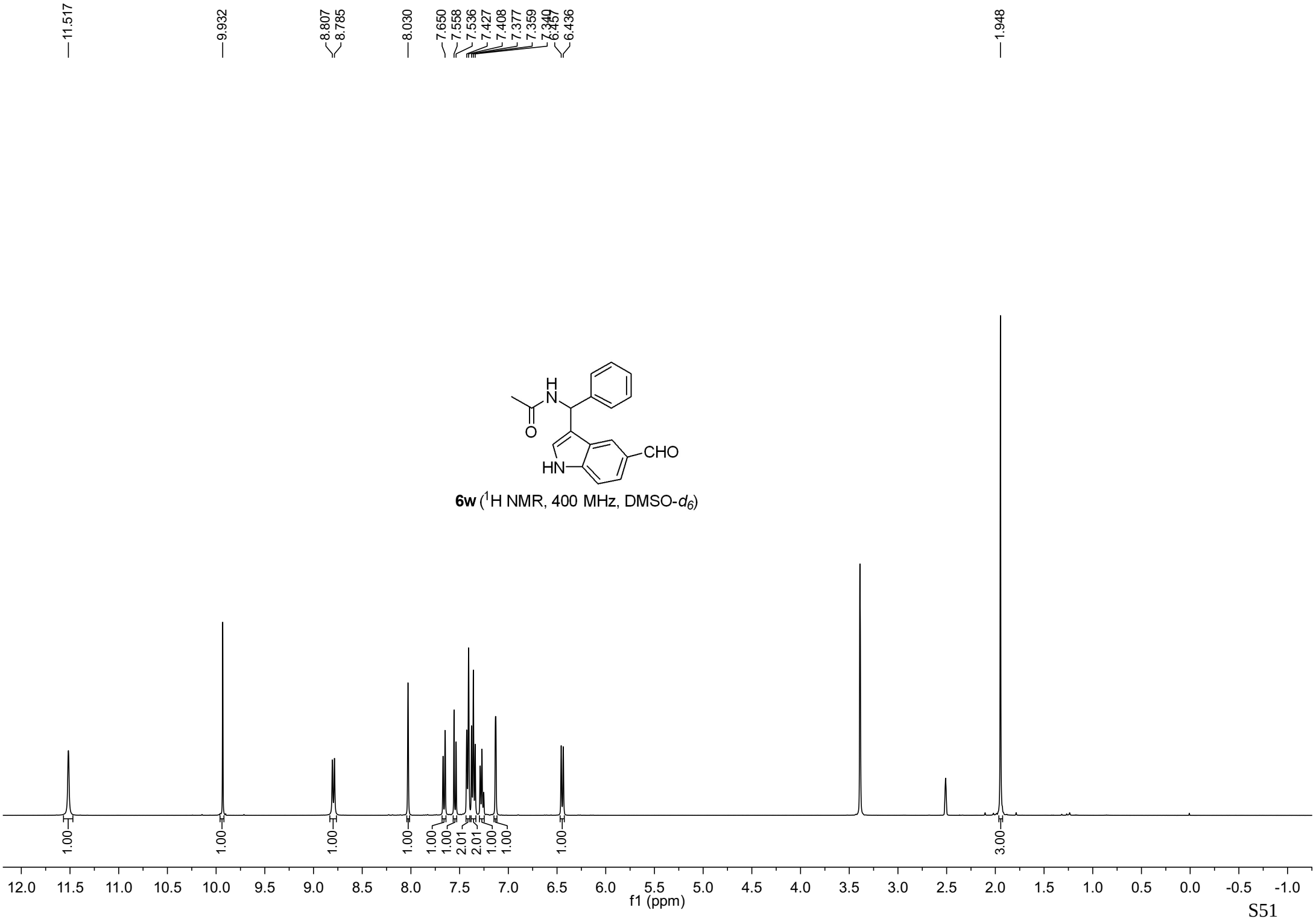
6u(^{13}C NMR, 100 MHz, DMSO-d_6)



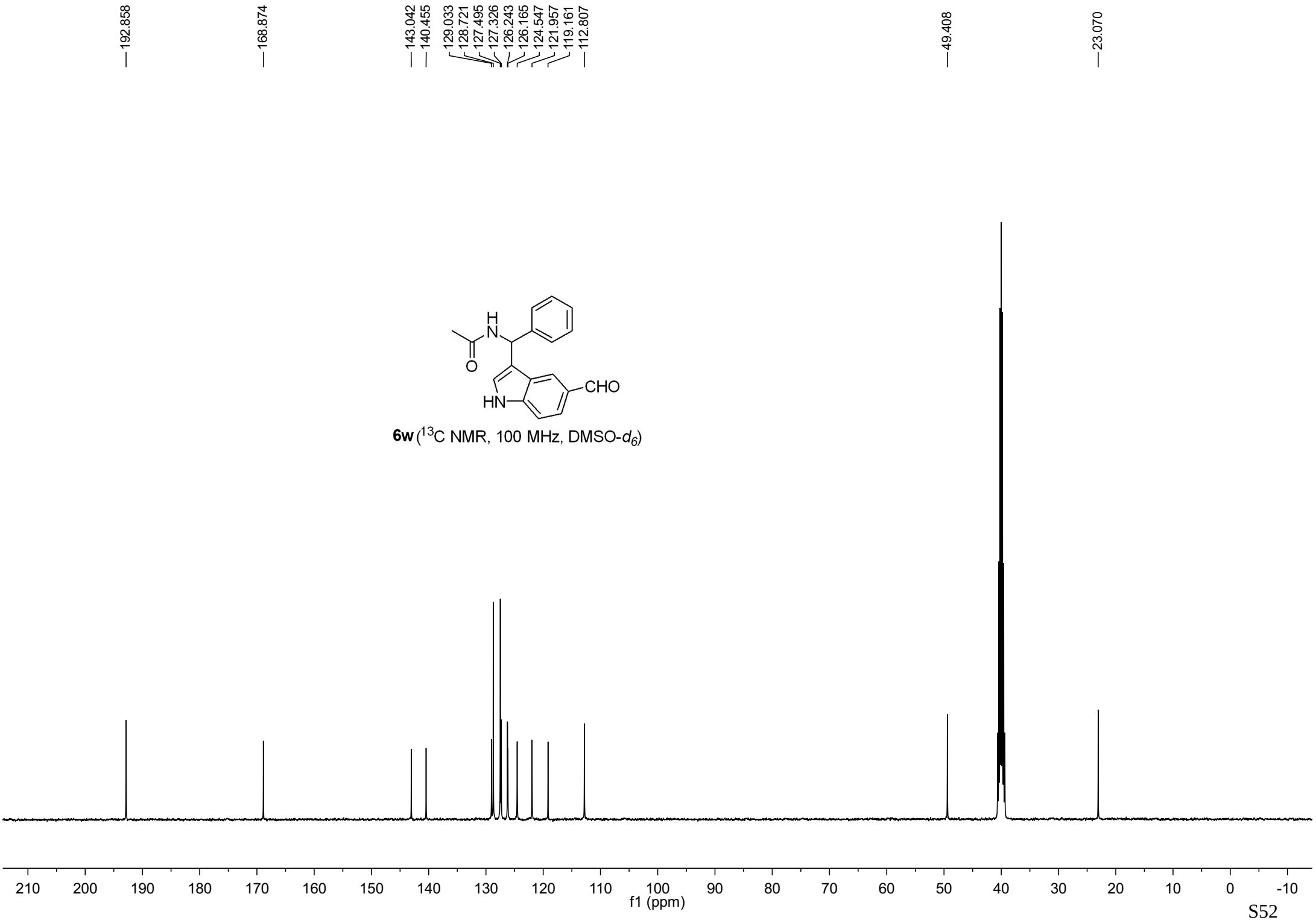
6v (^{1}H NMR, 400 MHz, DMSO-d_6)



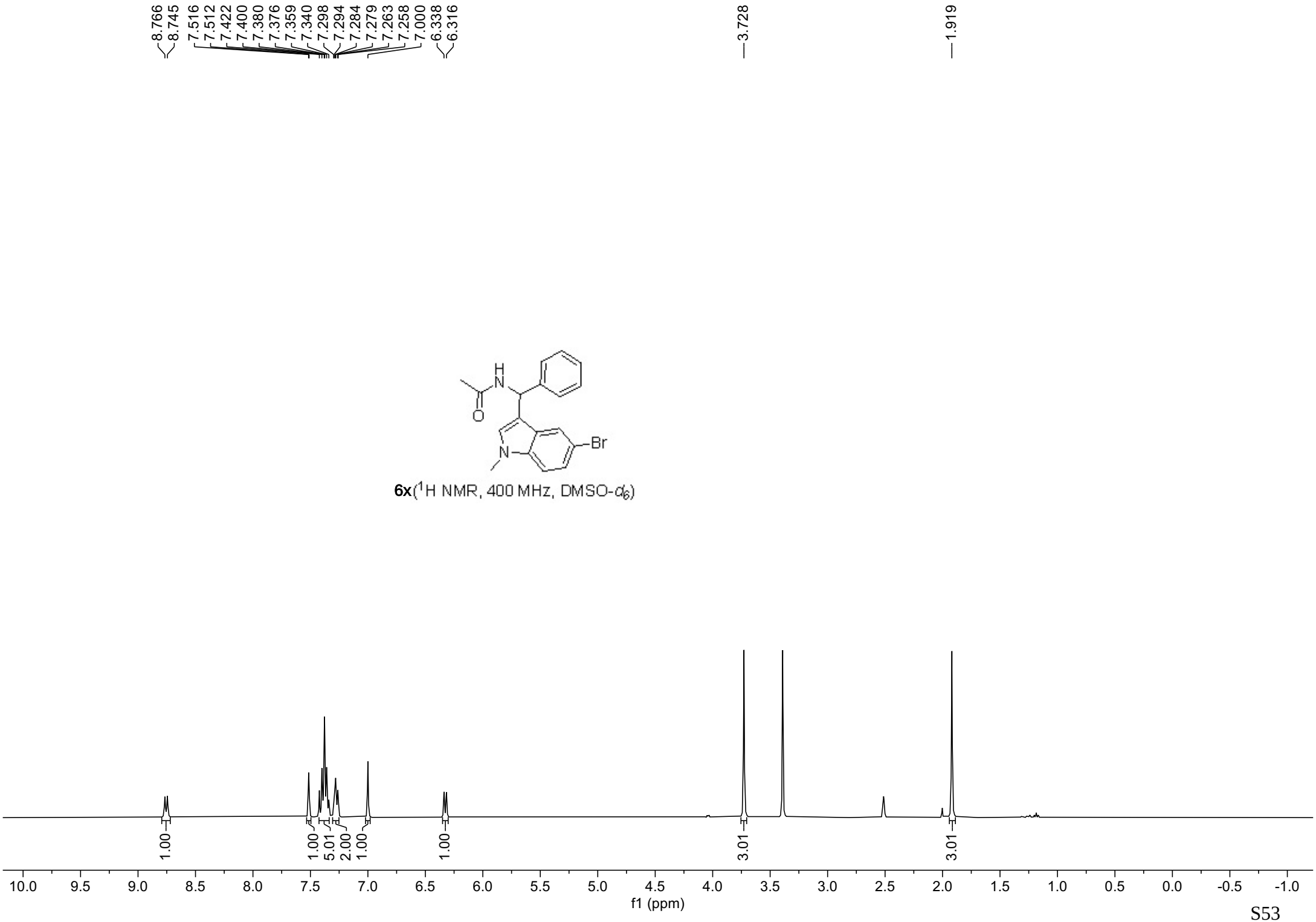
6v (^{13}C NMR, 100 MHz, DMSO-d_6)



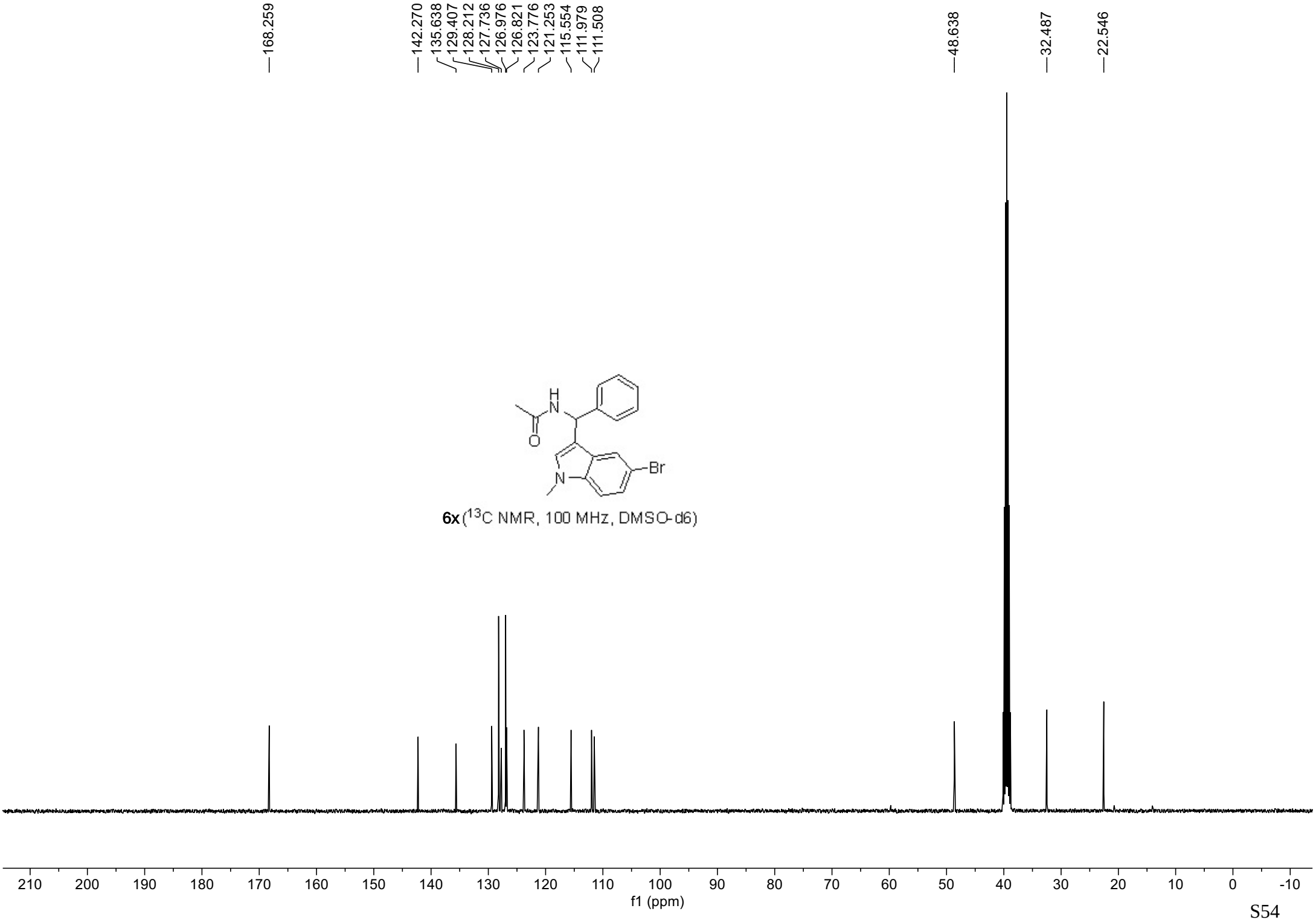
6w (^{1}H NMR, 400 MHz, DMSO-d_6)



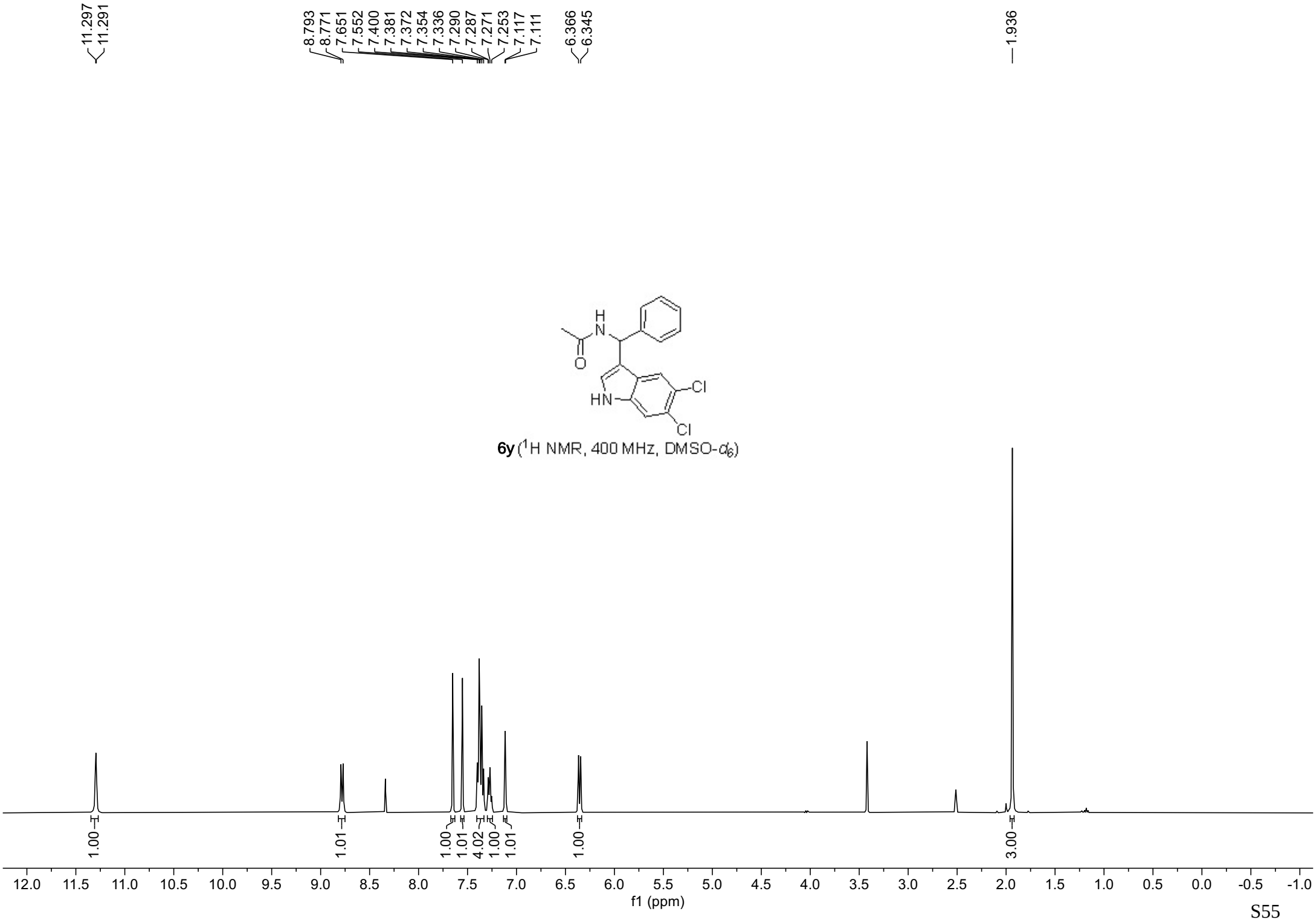
6w (^{13}C NMR, 100 MHz, DMSO-d_6)



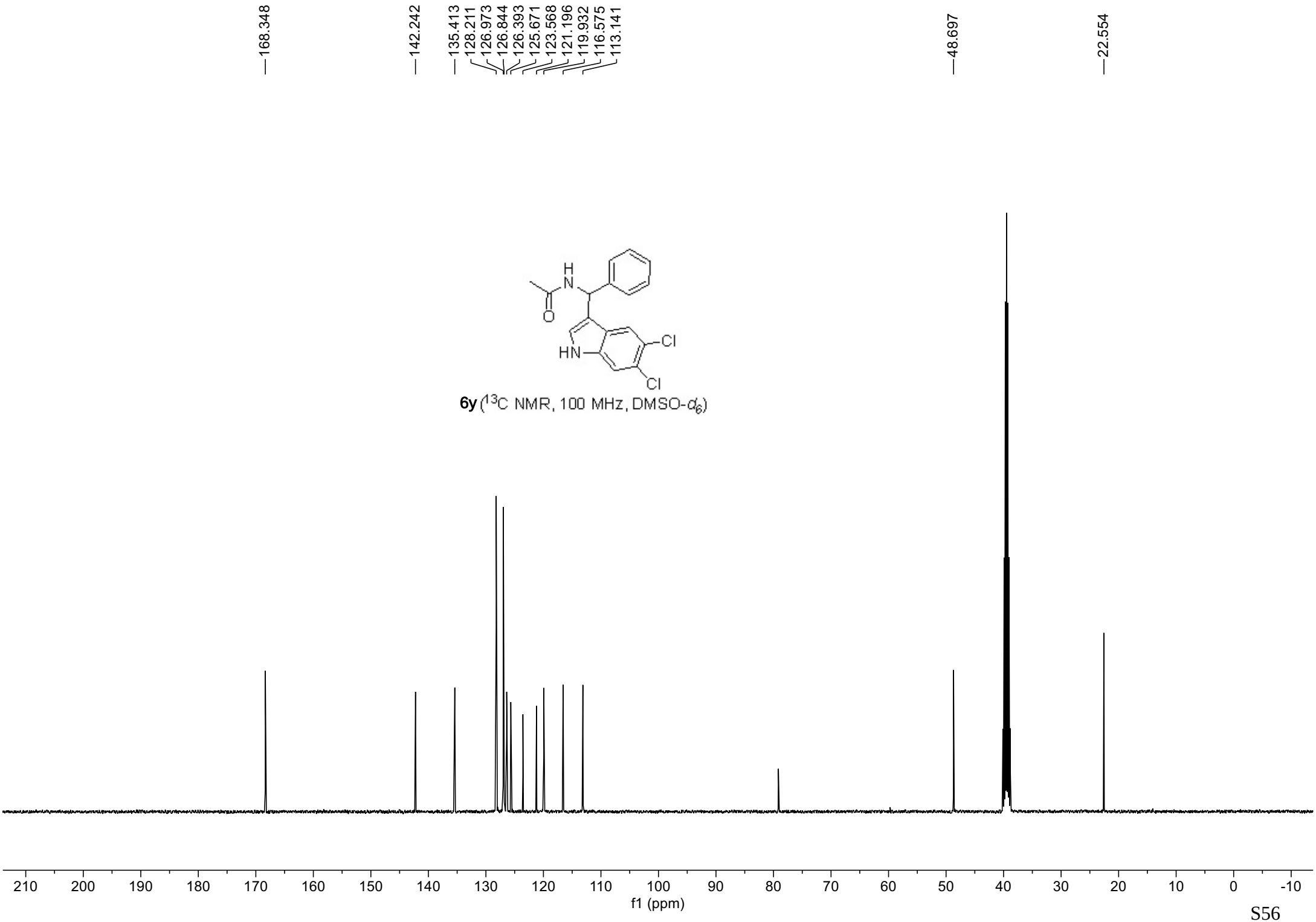
6x(^{1}H NMR, 400 MHz, DMSO-d_6)

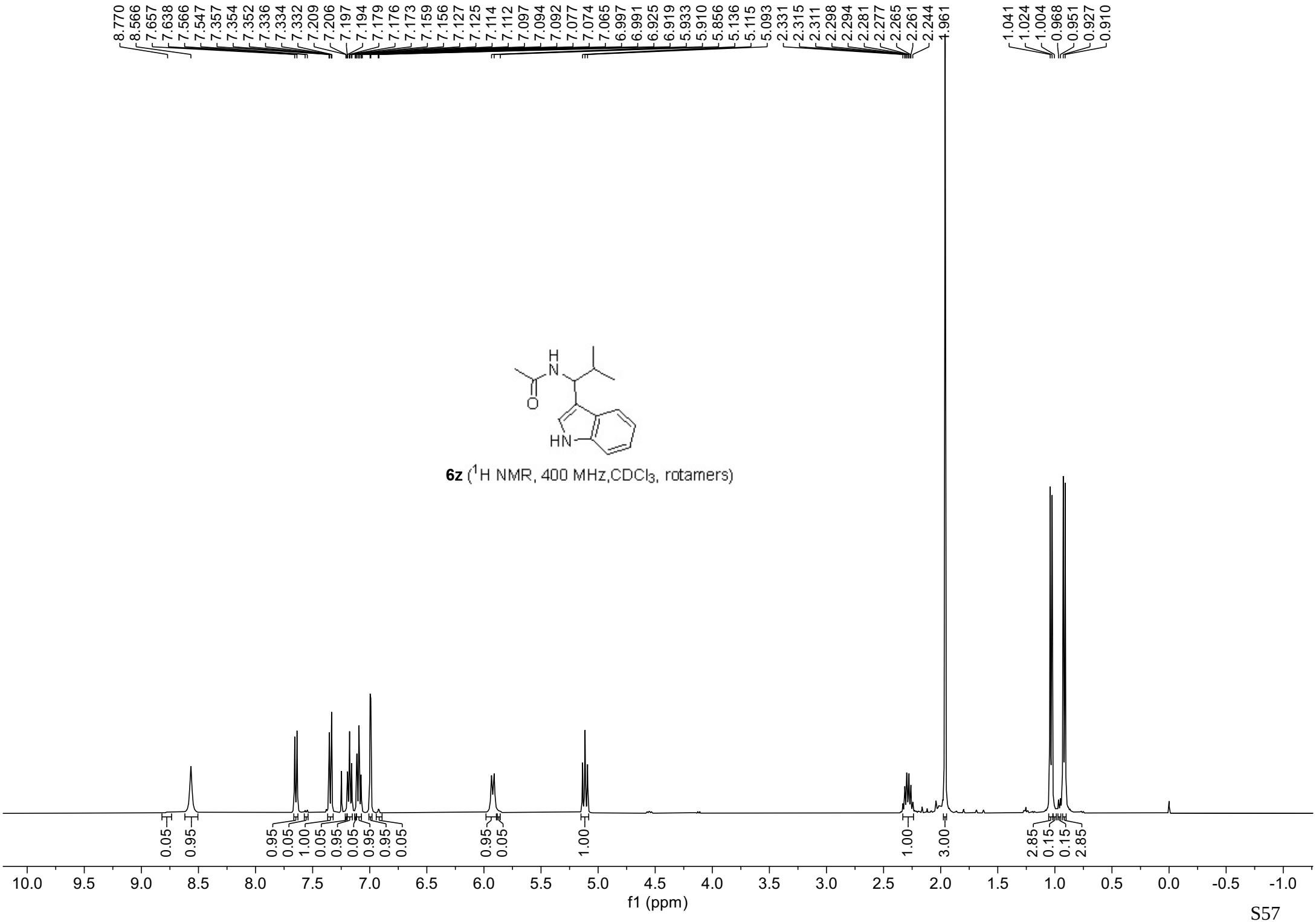


6x (^{13}C NMR, 100 MHz, DMSO-d6)

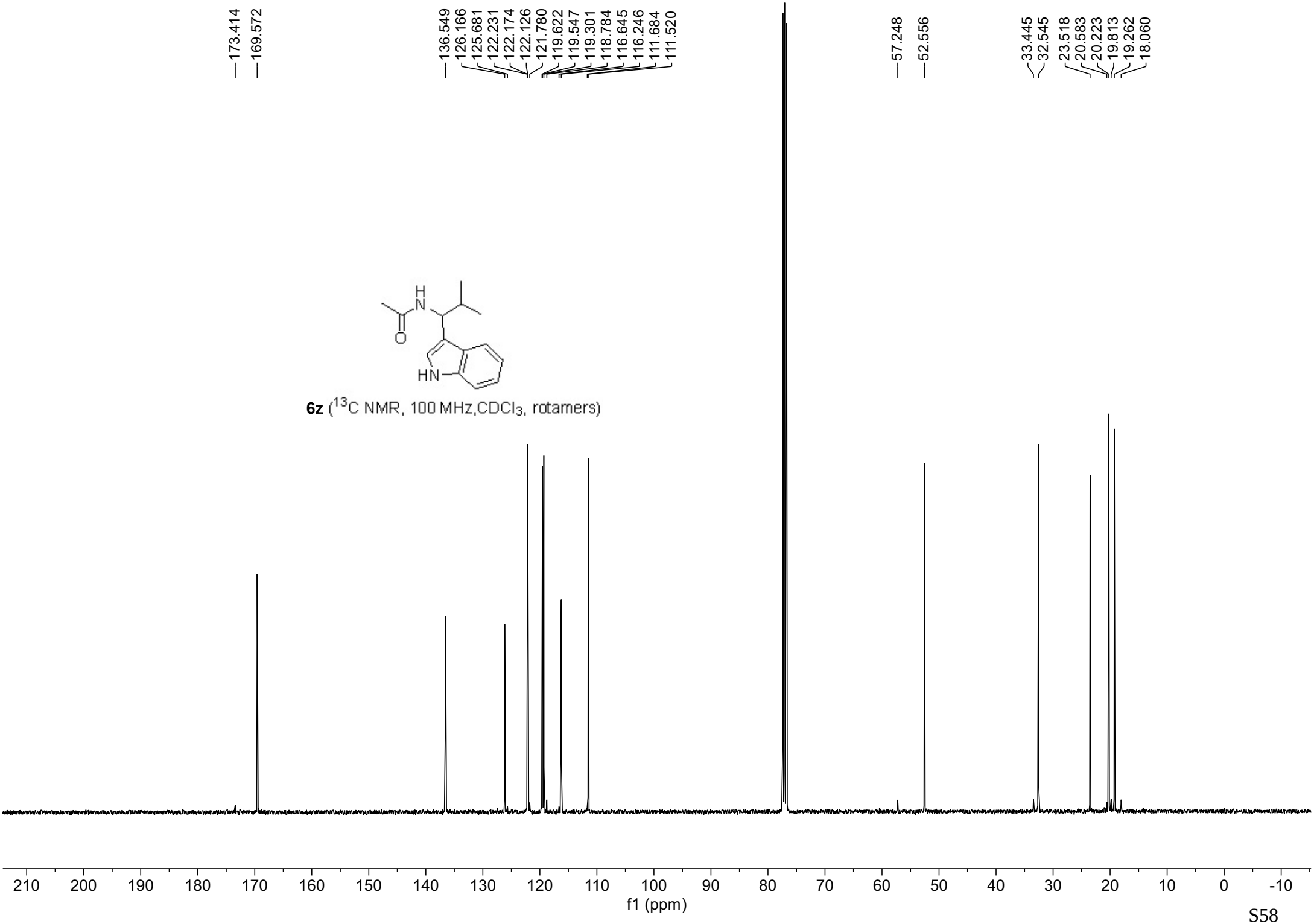


6y (^{1}H NMR, 400 MHz, DMSO-d_6)

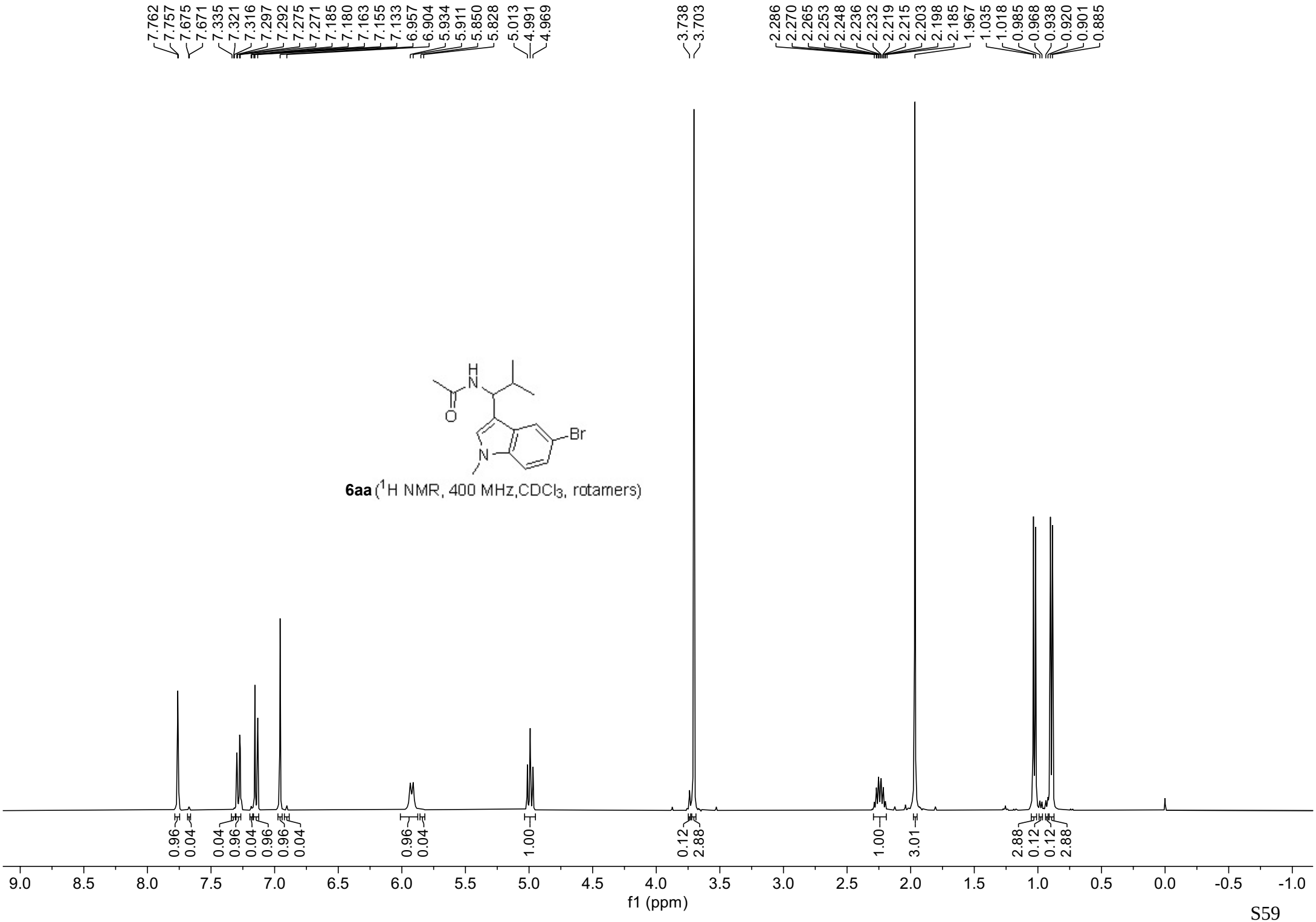

168.348
142.242
135.413
128.211
126.973
126.844
126.393
125.671
123.568
121.196
119.932
116.575
113.141
48.697
22.554
H
N
O
HN
Cl
Cl
6y (13C NMR, 100 MHz, DMSO-d6)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



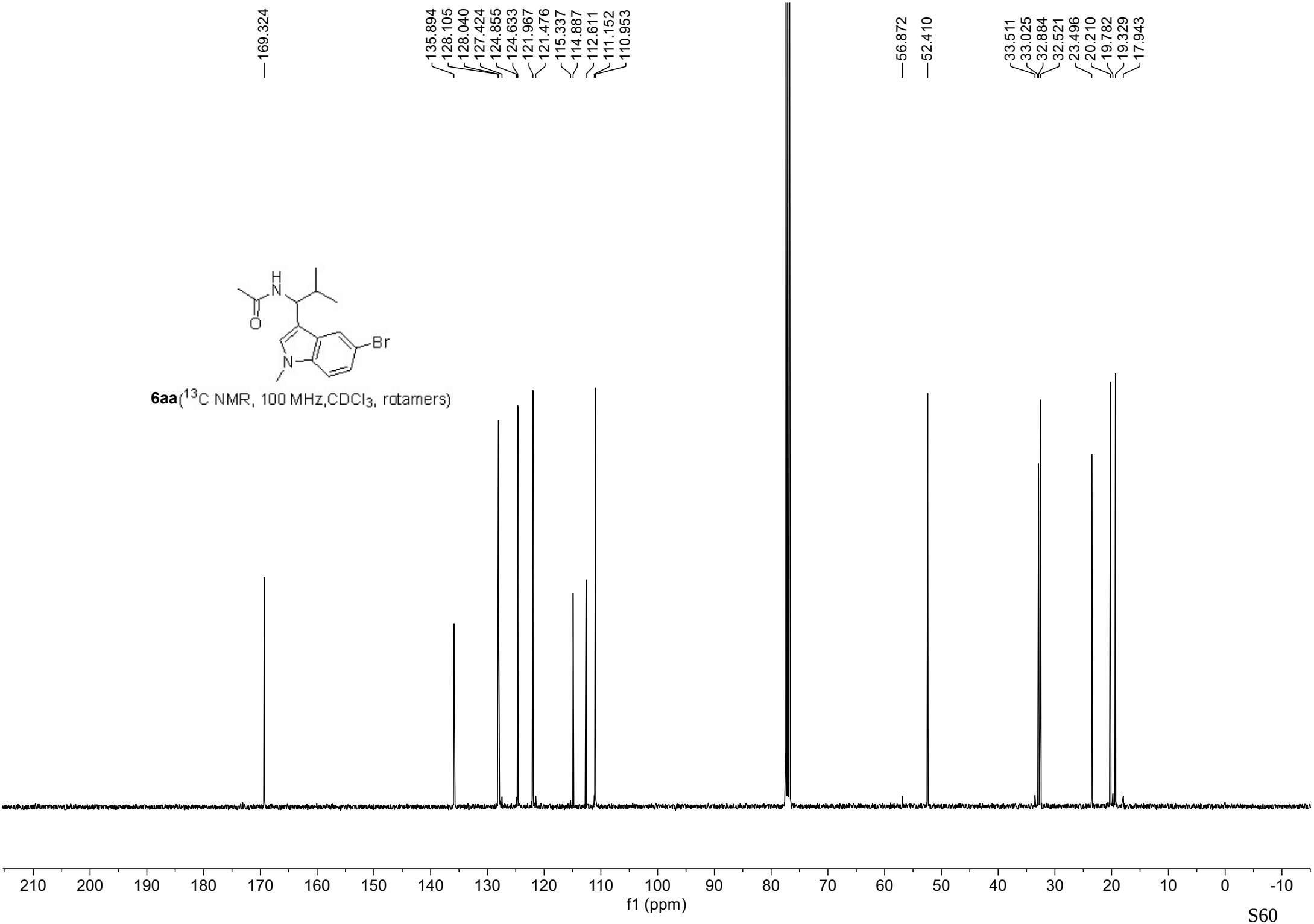
6z (^{1}H NMR, 400 MHz,$CDCl_3$, rotamers)



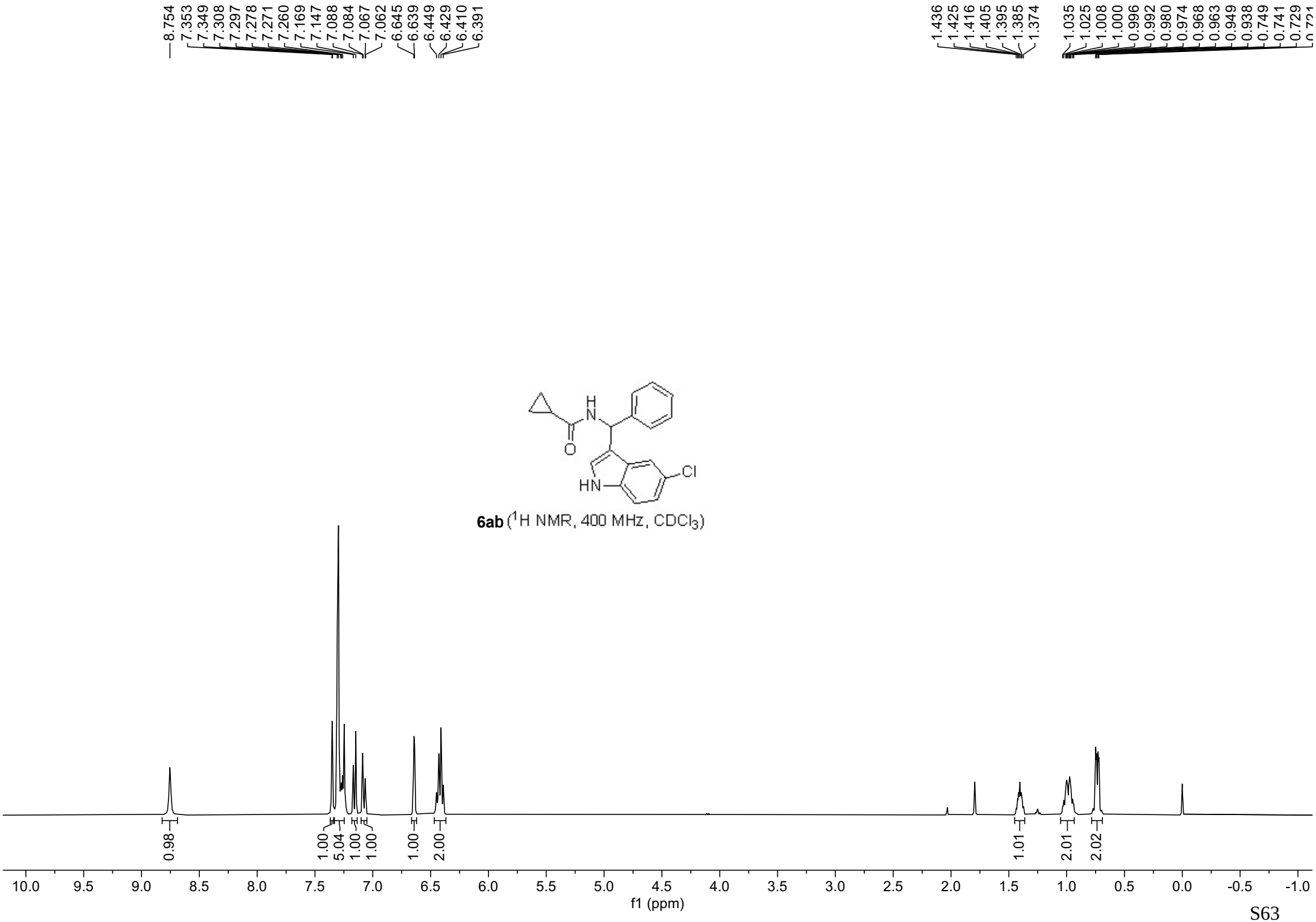
6z (^{13}C NMR, 100 MHz,$CDCl_3$, rotamers)

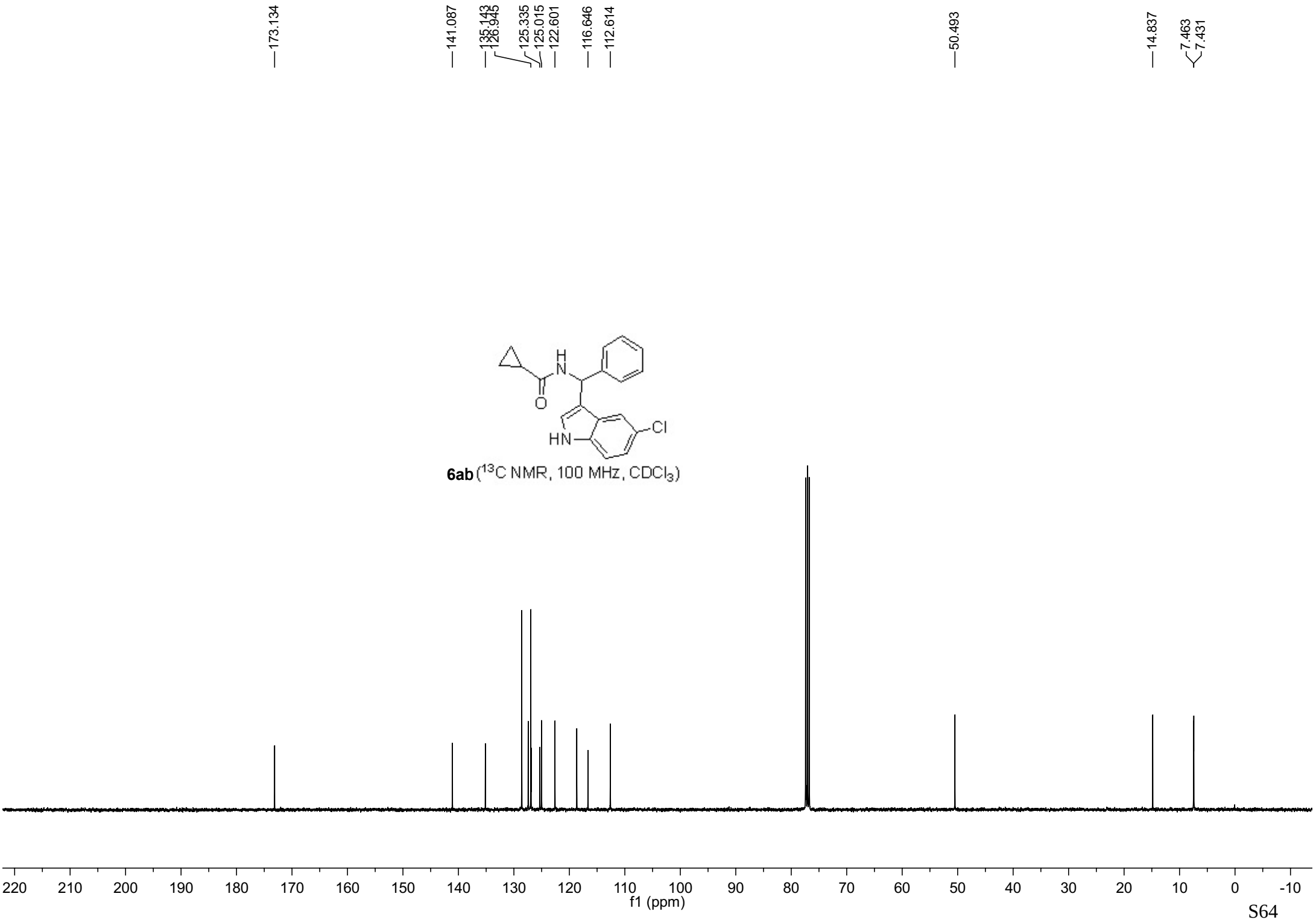


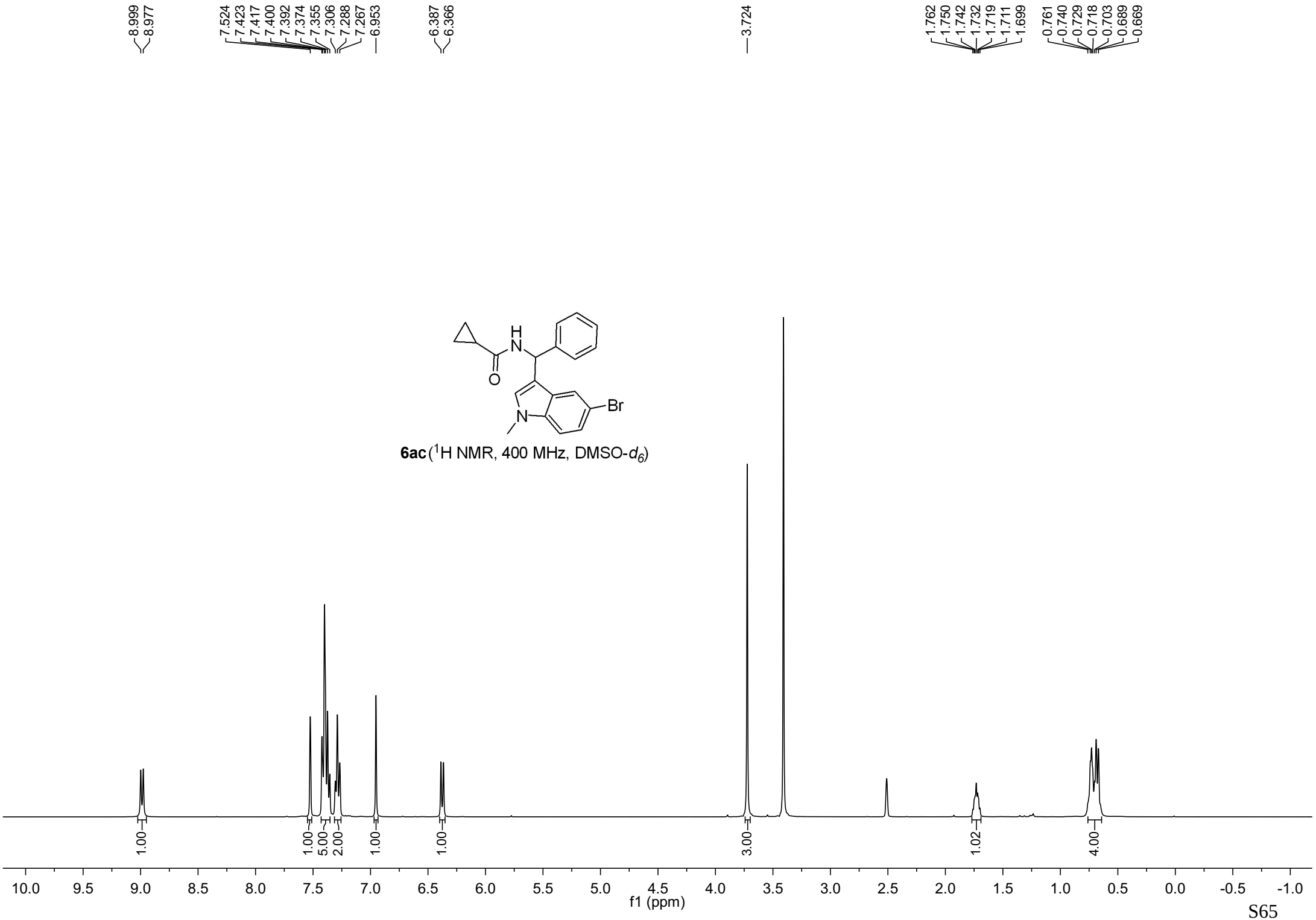
6aa (^{1}H NMR, 400 MHz,$CDCl_3$, rotamers)

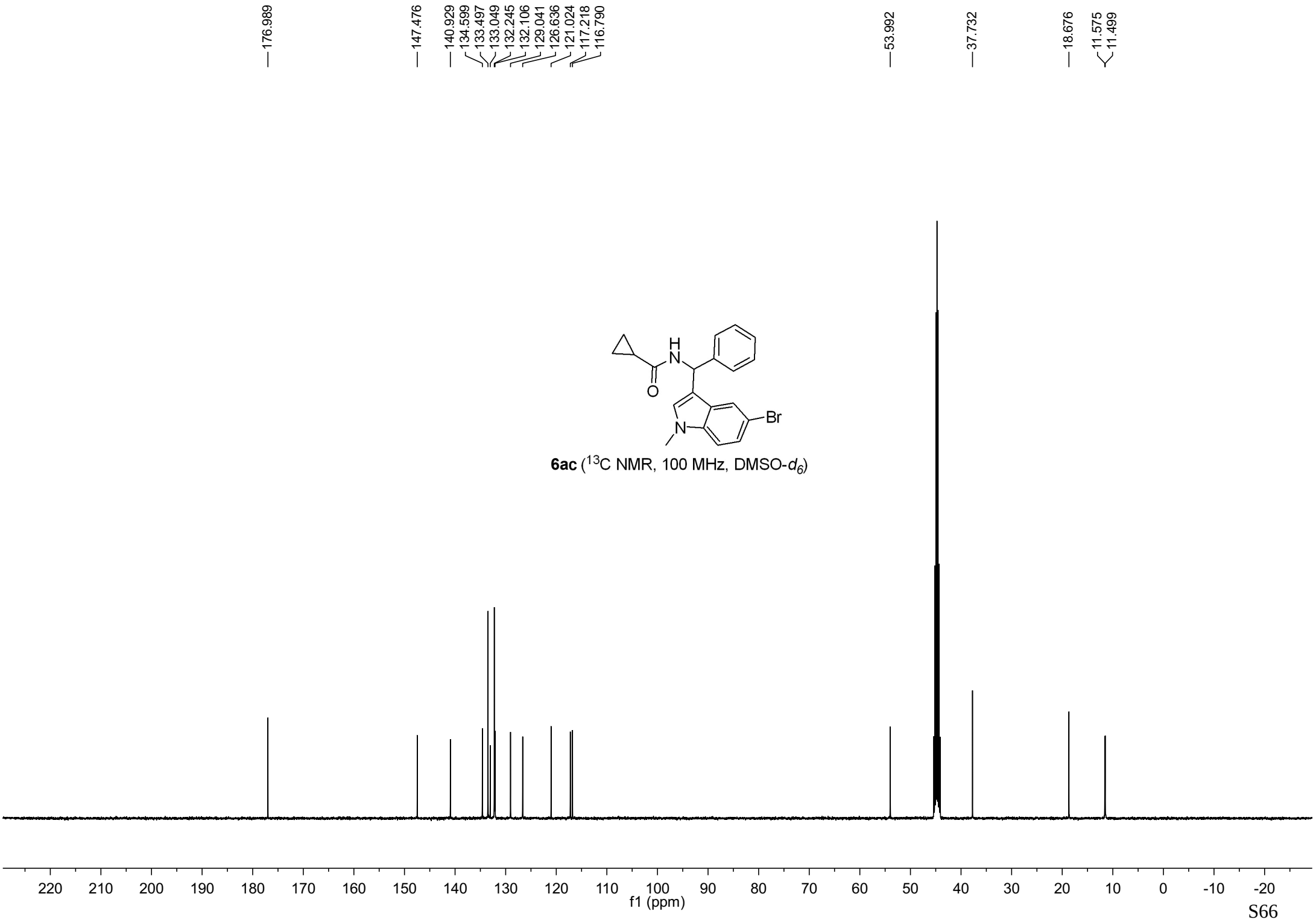


6aa(^{13}C NMR, 100 MHz,$CDCl_3$, rotamers)

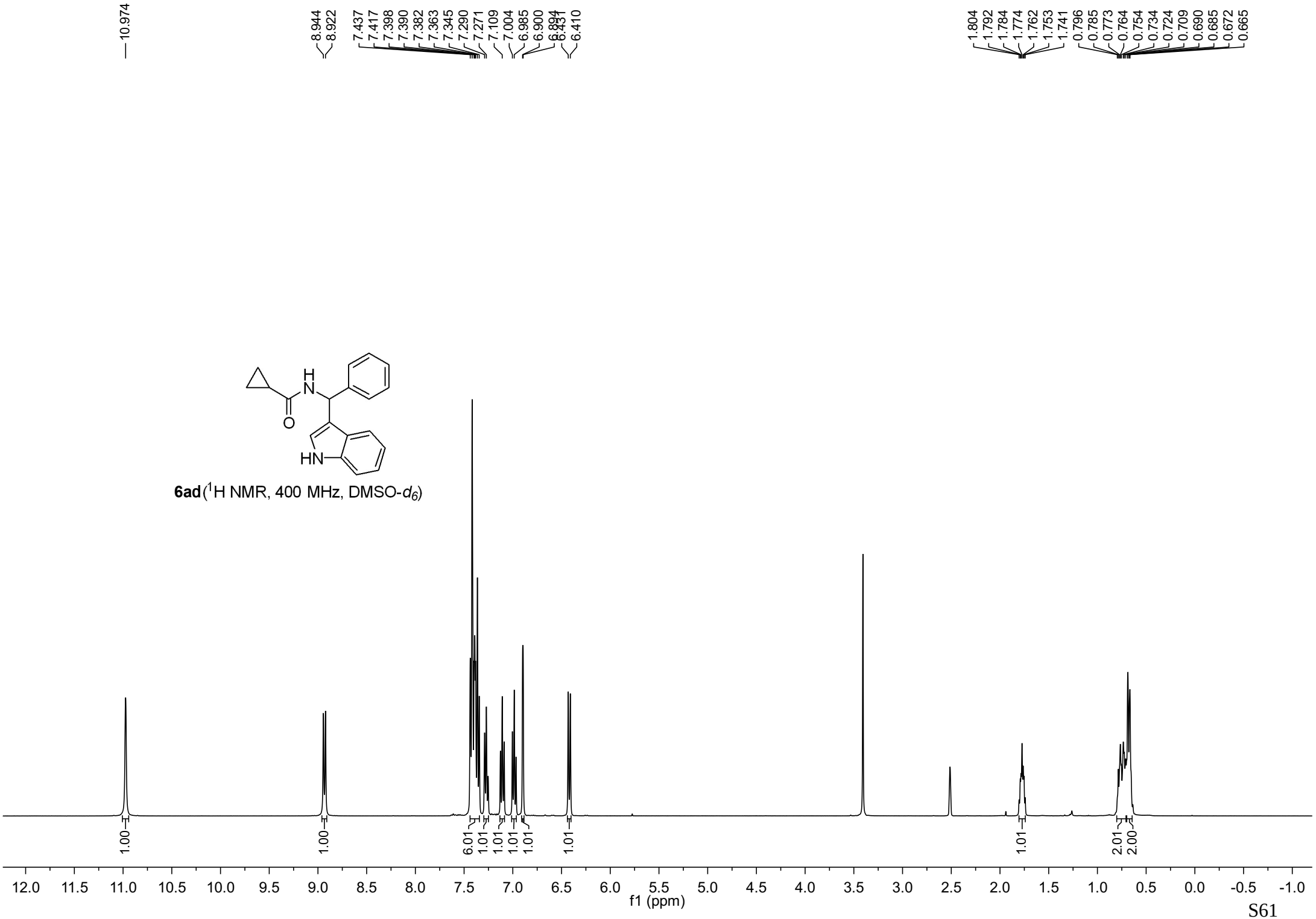

8.754
7.353
7.349
7.308
7.297
7.278
7.271
7.260
7.169
7.147
7.088
7.084
7.067
7.062
6.645
6.639
6.449
6.429
6.410
6.391
1.436
1.425
1.416
1.405
1.395
1.385
1.374
1.035
1.025
1.008
1.000
0.996
0.992
0.980
0.974
0.968
0.963
0.949
0.938
0.749
0.741
0.729
H
N
O
HN
Cl
6ab (1H NMR, 400 MHz, CDCl3)
0.98
1.00
5.04
1.00
1.00
1.00
2.00
1.01
2.01
2.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

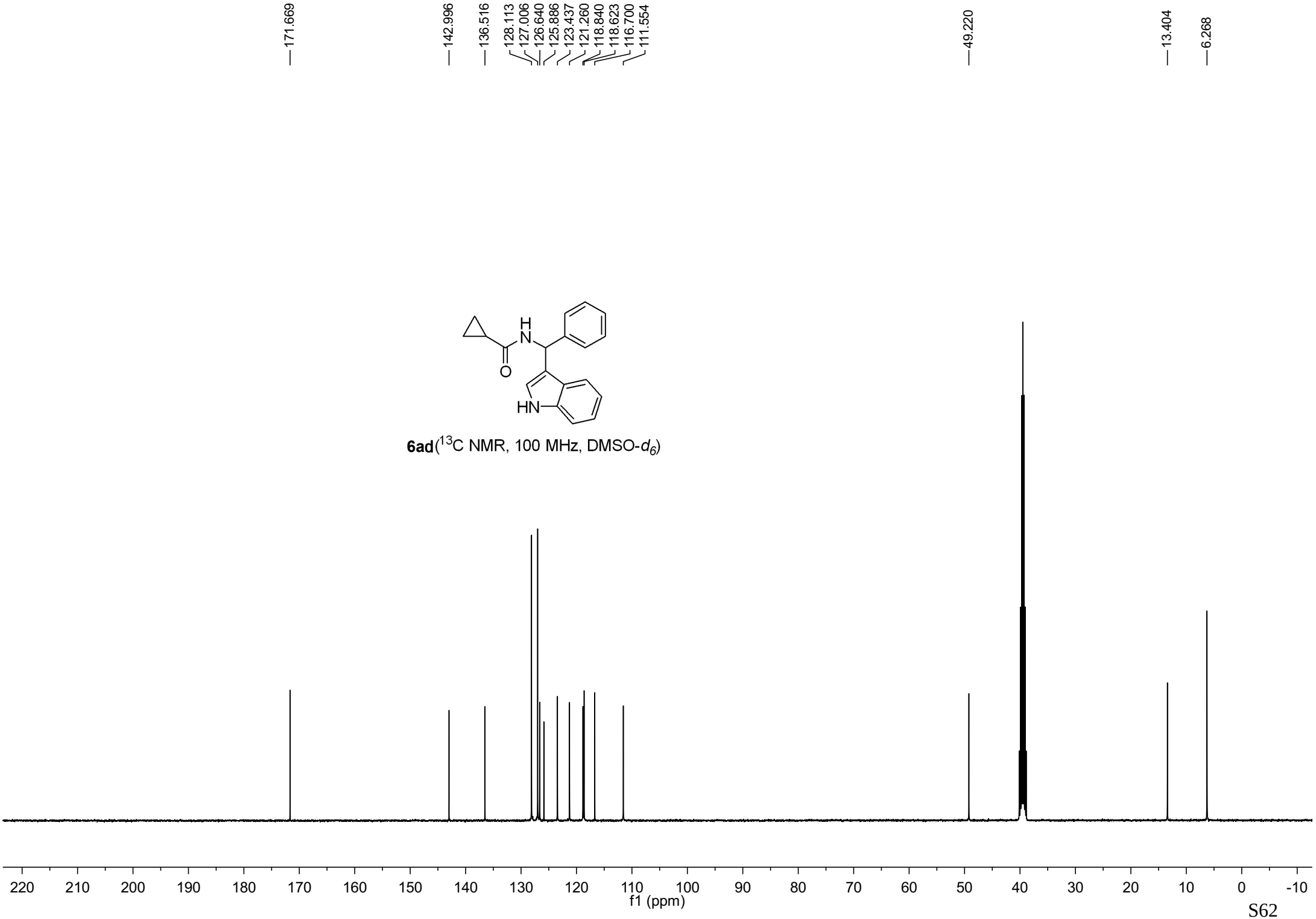

173.134
141.087
135.143
126.945
125.335
125.015
122.601
116.646
112.614
50.493
14.837
7.463
7.431
H
N
O
HN
Cl
6ab (13C NMR, 100 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.999
8.977
7.524
7.423
7.417
7.400
7.392
7.374
7.355
7.306
7.288
7.267
6.953
6.387
6.366
3.724
1.762
1.750
1.742
1.732
1.719
1.711
1.699
0.761
0.740
0.729
0.718
0.703
0.689
0.669
H
N
O
N
Br
6ac (1H NMR, 400 MHz, DMSO-d6)
1.00
1.00
5.00
2.00
1.00
1.00
3.00
1.02
4.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

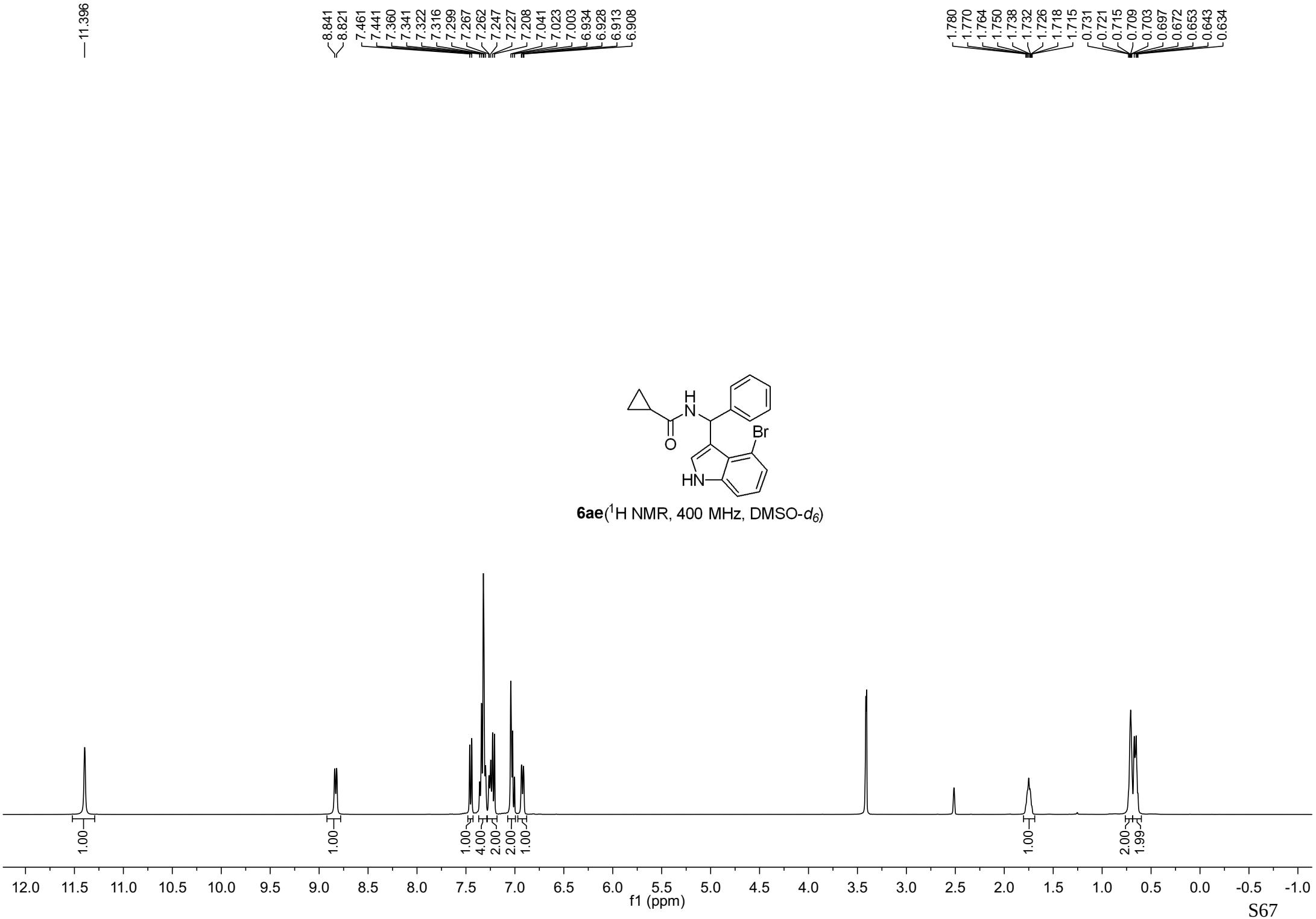


6ac (^{13}C NMR, 100 MHz, DMSO-d_6)

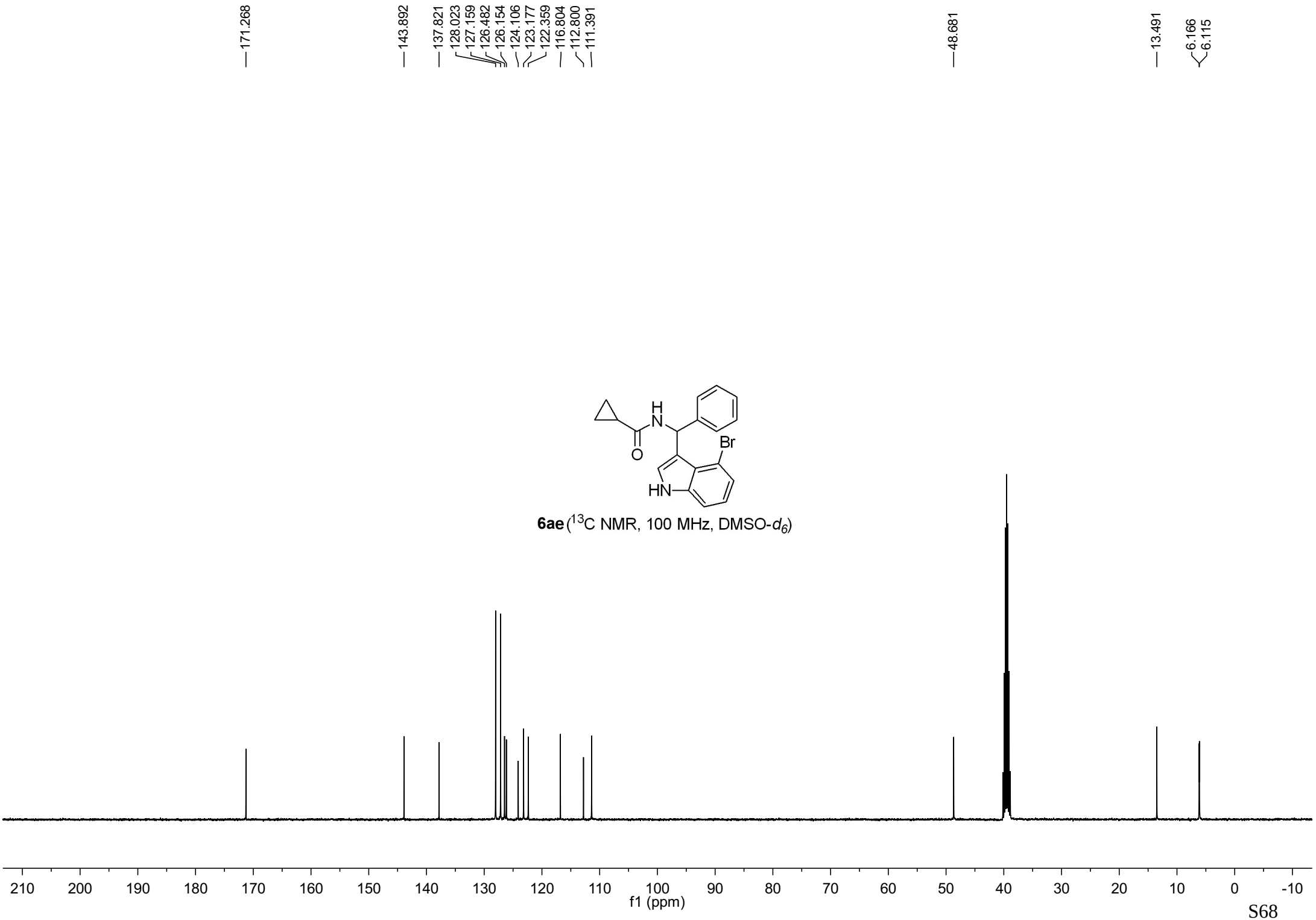

10.974
8.944
8.922
7.437
7.417
7.398
7.390
7.382
7.363
7.345
7.290
7.271
7.109
7.004
6.985
6.900
6.410
1.804
1.792
1.784
1.774
1.762
1.753
1.741
0.796
0.785
0.773
0.764
0.754
0.734
0.724
0.709
0.690
0.685
0.672
0.665
H
N
O
HN
6ad (1H NMR, 400 MHz, DMSO-d6)
1.00
1.00
6.01
1.01
1.01
1.01
1.01
1.01
2.01
2.00
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



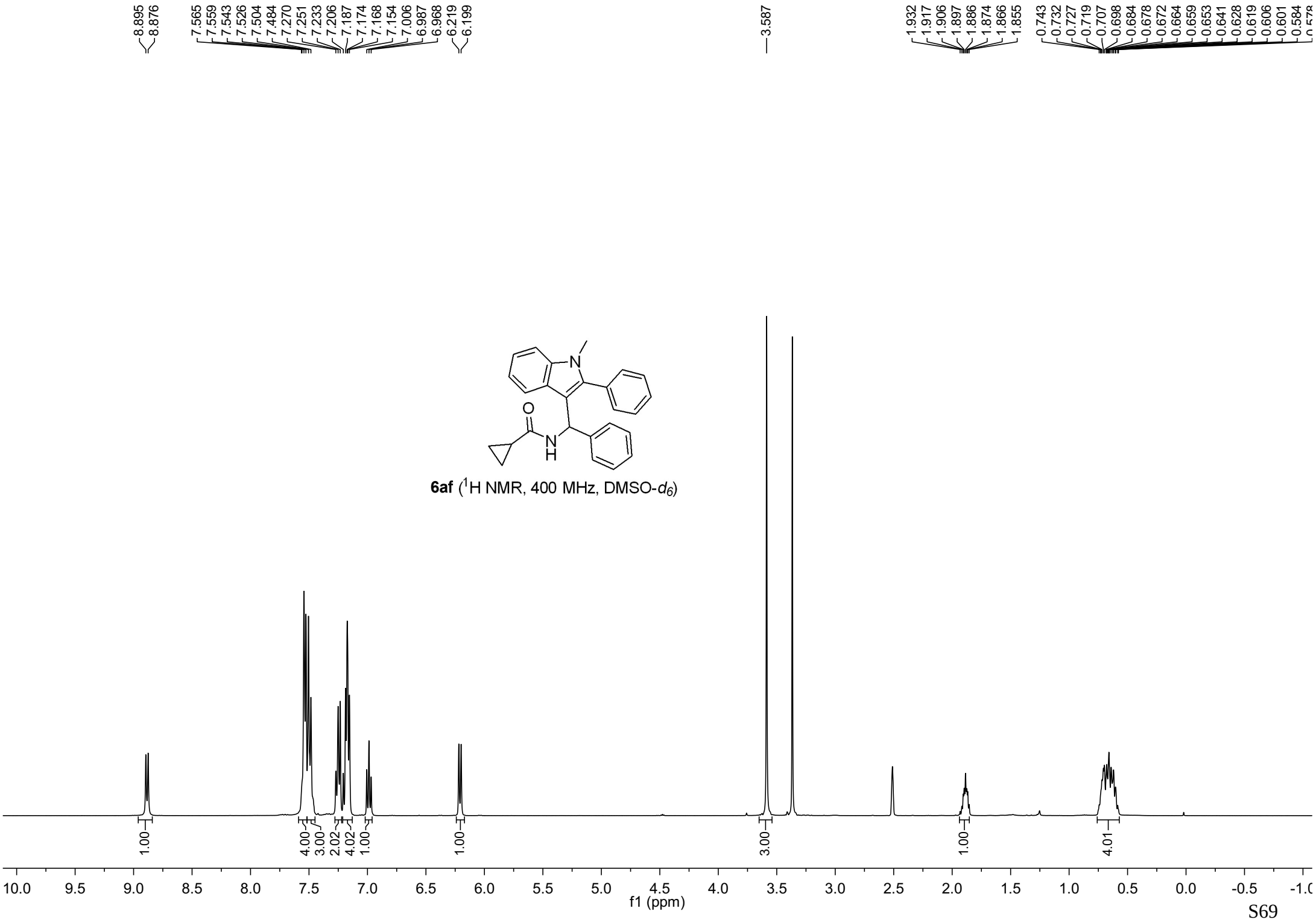
6ad(^{13}C NMR, 100 MHz, DMSO-d_6)



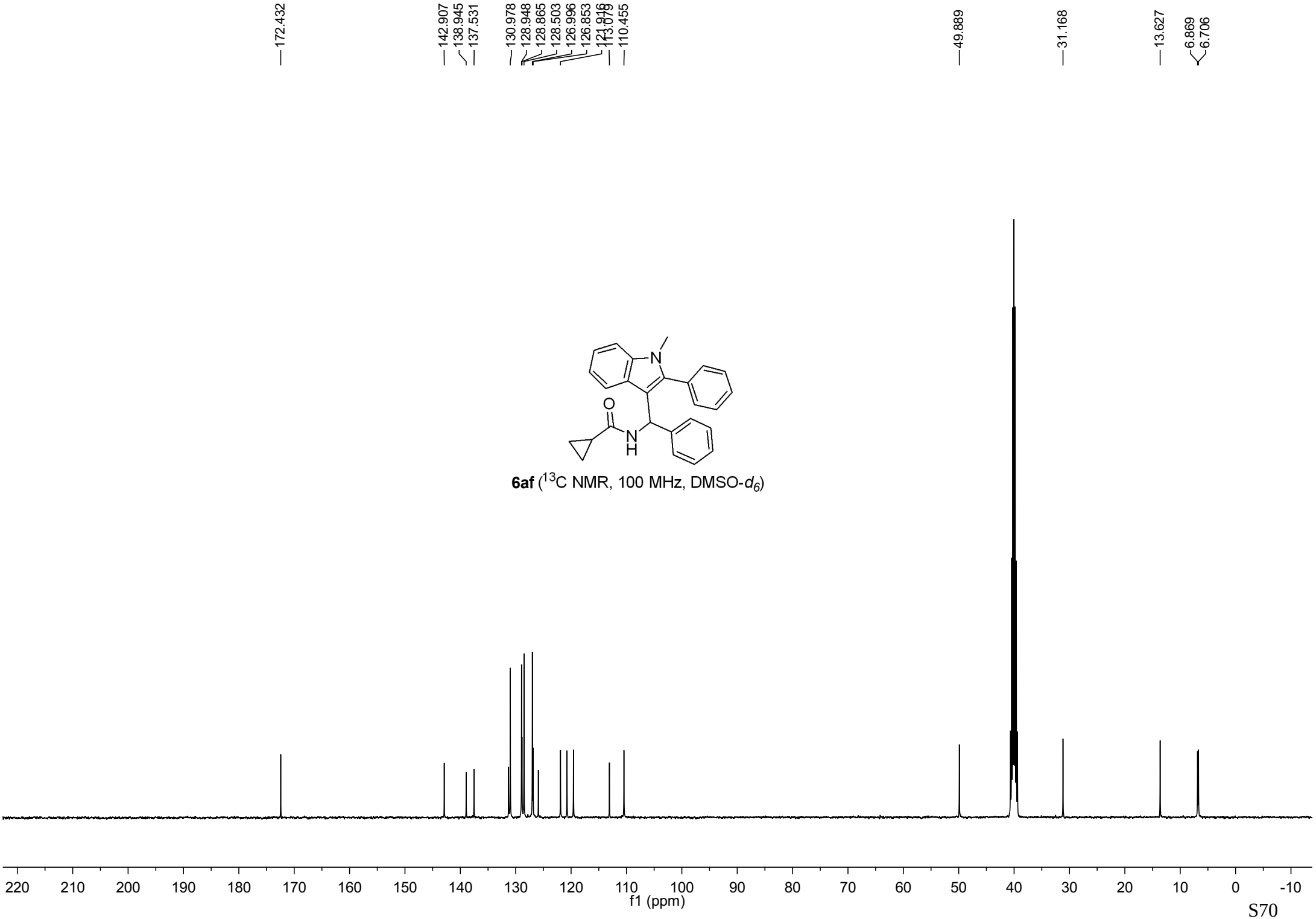
6ae(^{1}H NMR, 400 MHz, DMSO-d_6)



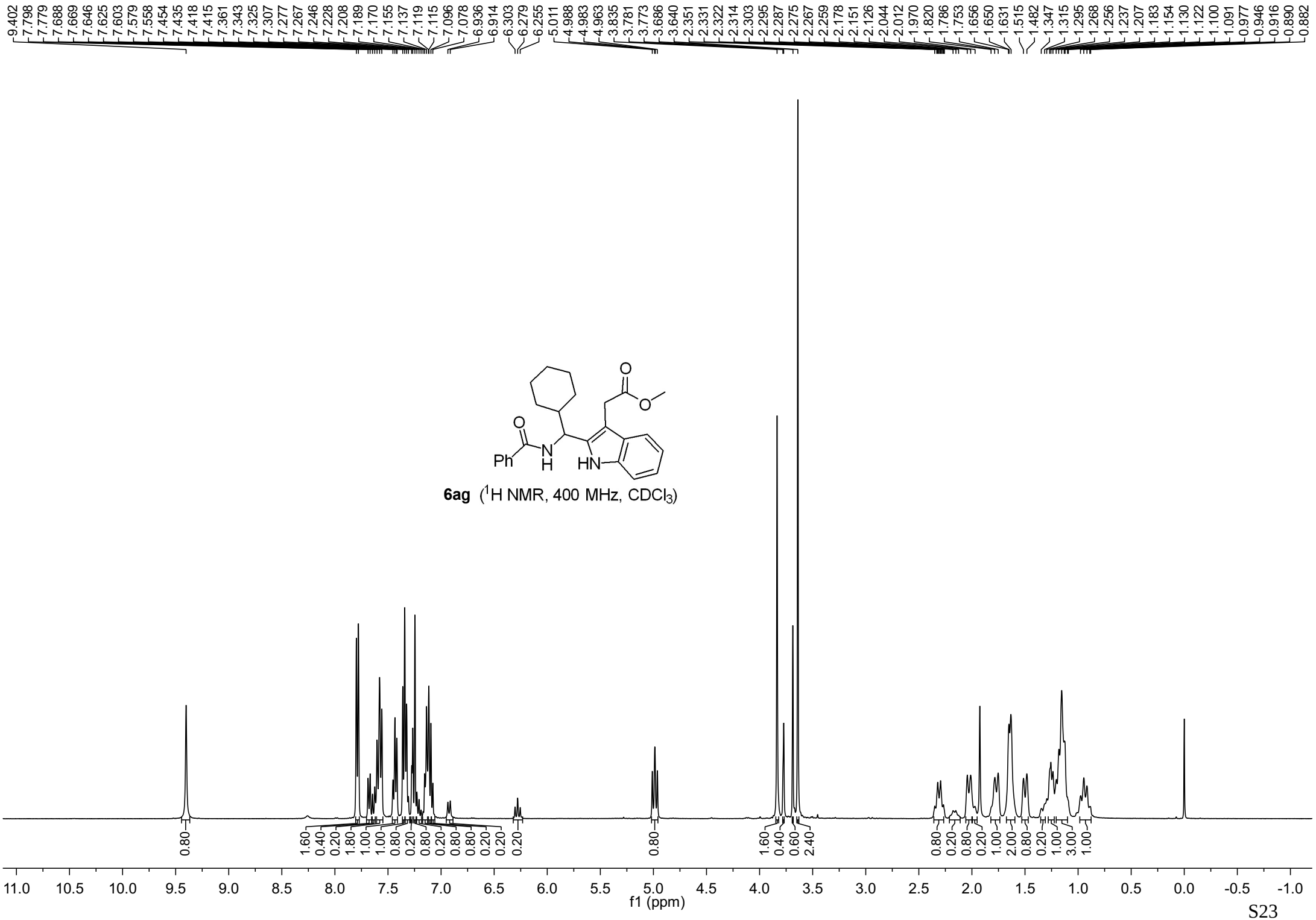
6ae (^{13}C NMR, 100 MHz, DMSO-d_6)

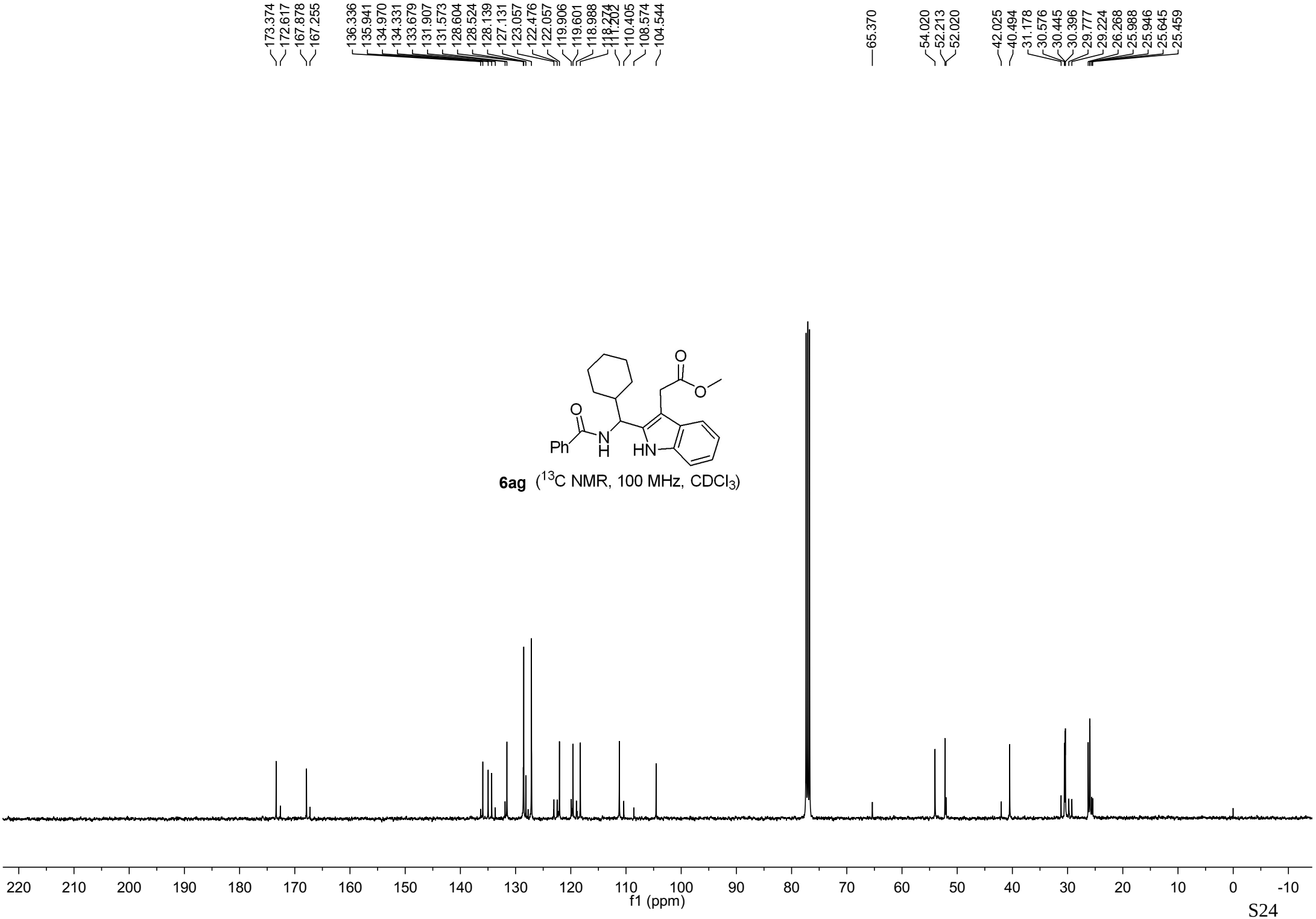


6af (^{1}H NMR, 400 MHz, DMSO-d_6)

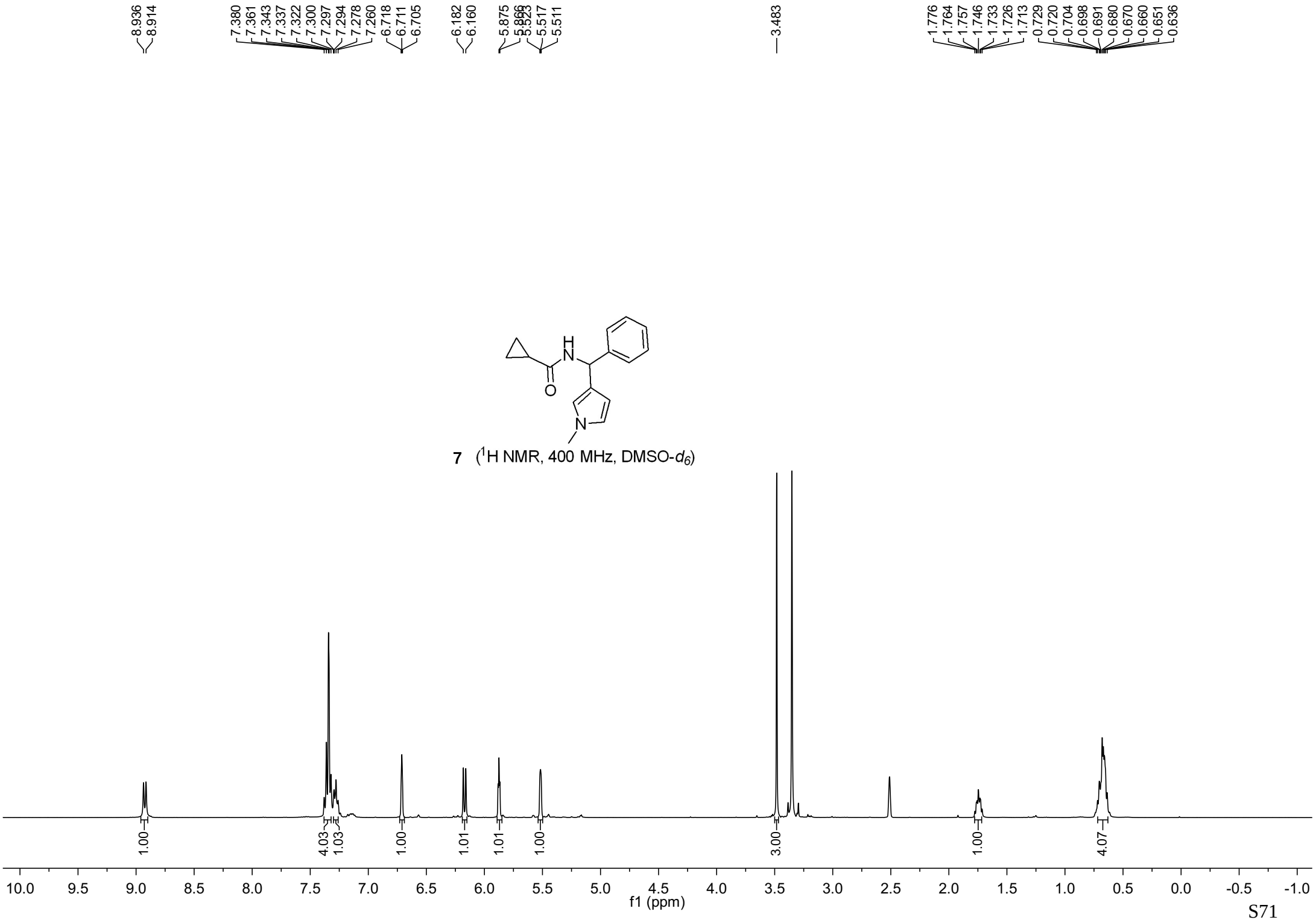


6af (^{13}C NMR, 100 MHz, DMSO-d_6)

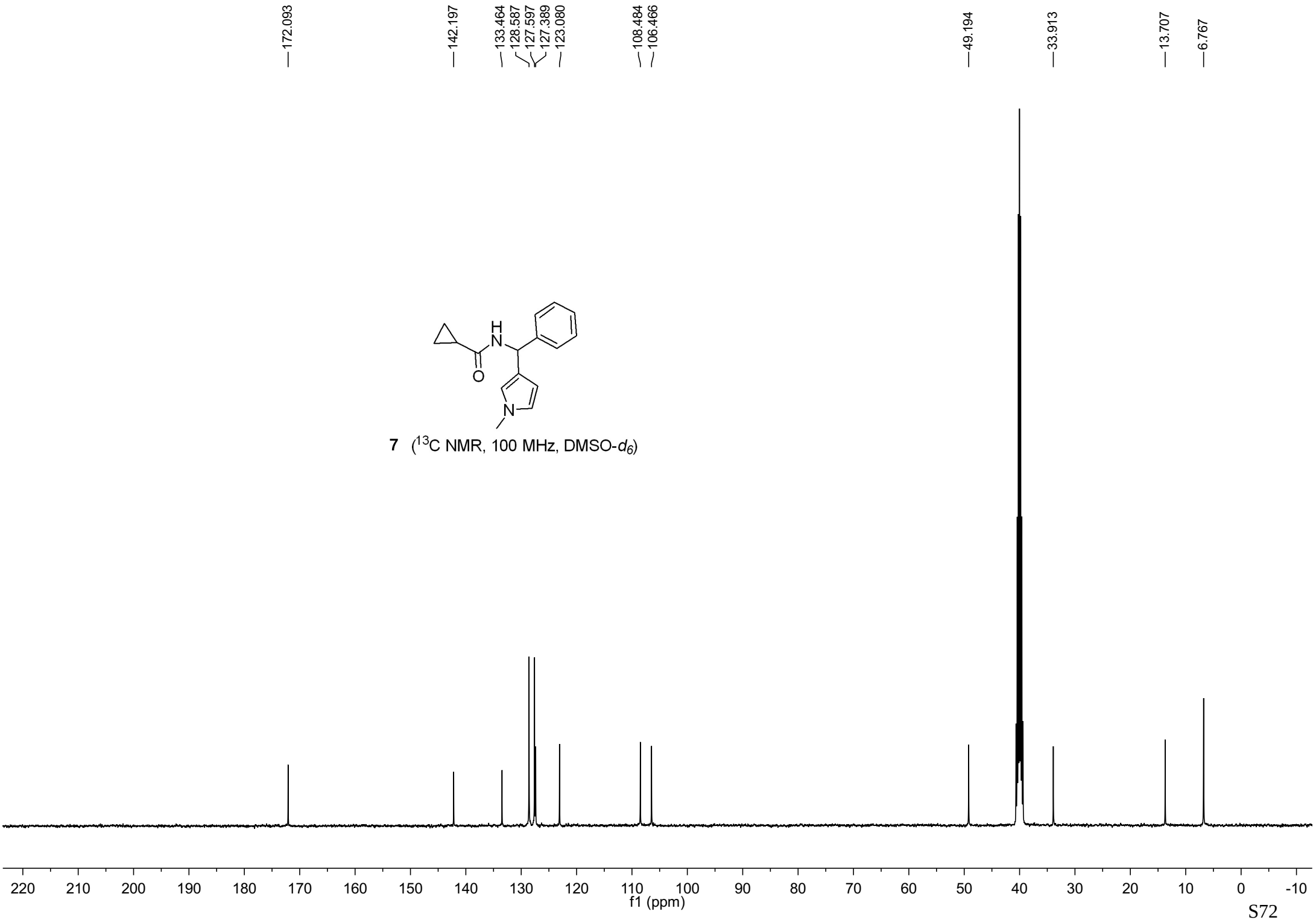

9.402 7.798 7.779 7.688 7.669 7.646 7.625 7.603 7.579 7.558 7.454 7.435 7.418 7.415 7.361 7.343 7.325 7.307 7.277 7.267 7.246 7.228 7.208 7.189 7.170 7.155 7.137 7.119 7.115 7.096 7.078 6.936 6.914 6.303 6.279 6.255 5.011 4.988 4.983 4.963 3.835 3.781 3.773 3.686 3.640 2.351 2.331 2.322 2.314 2.303 2.295 2.287 2.275 2.267 2.259 2.178 2.151 2.126 2.044 2.012 1.970 1.820 1.786 1.753 1.656 1.650 1.631 1.515 1.482 1.347 1.315 1.295 1.268 1.256 1.237 1.207 1.183 1.154 1.130 1.122 1.100 1.091 0.977 0.946 0.916 0.890 0.882
O
O
O
N
H
Ph
HN
6ag (1H NMR, 400 MHz, CDCl3)
0.80
1.60 0.40 0.20 1.80 1.00 1.00 0.80 0.20 0.80 0.20 0.80 0.20 0.20
0.20
0.80
1.60 0.40 0.60 2.40
0.80 0.20 0.80 0.20 1.00 2.00 0.80 0.20 1.00 3.00 1.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)



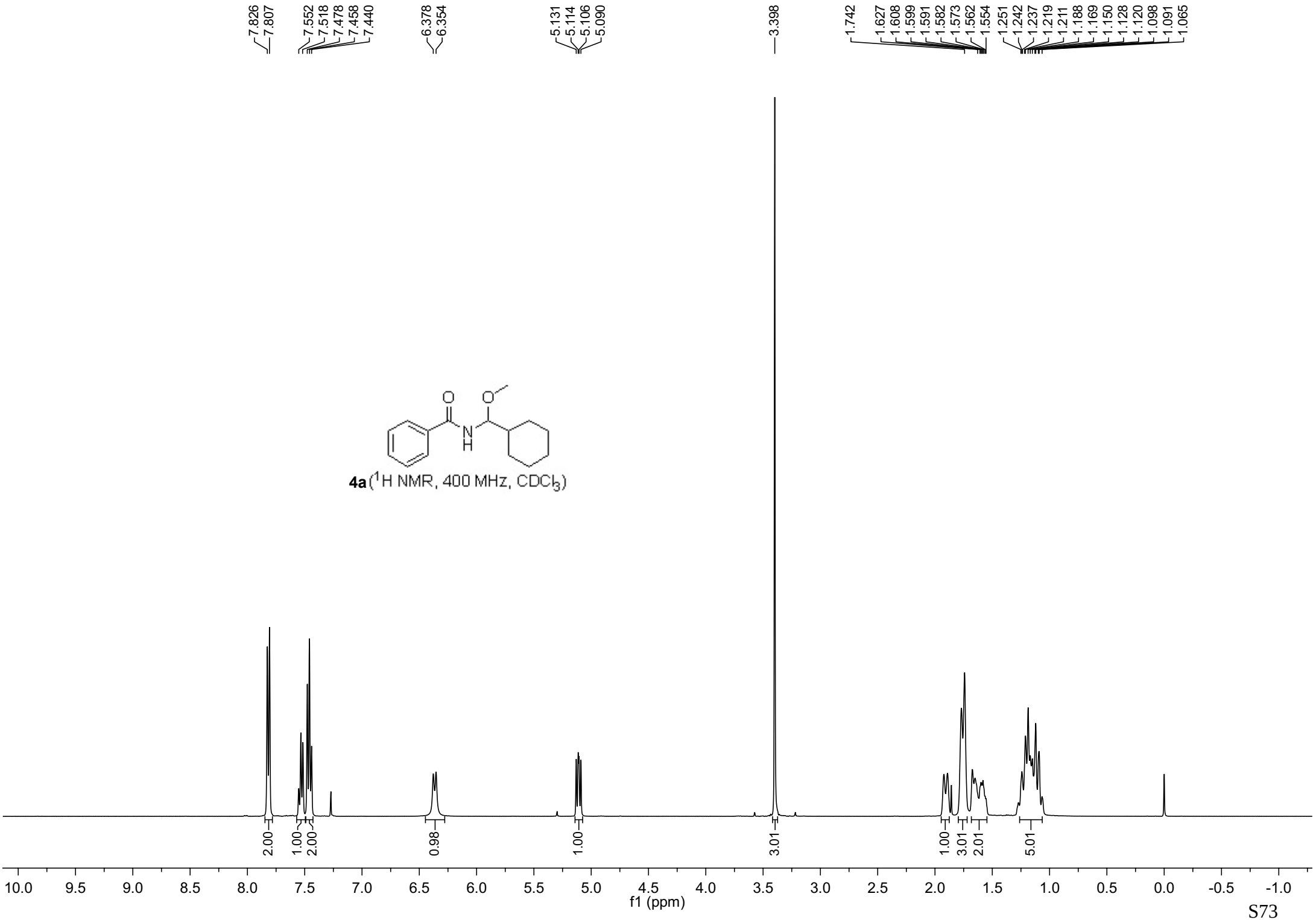
6ag (^{13}C NMR, 100 MHz, $CDCl_3$)

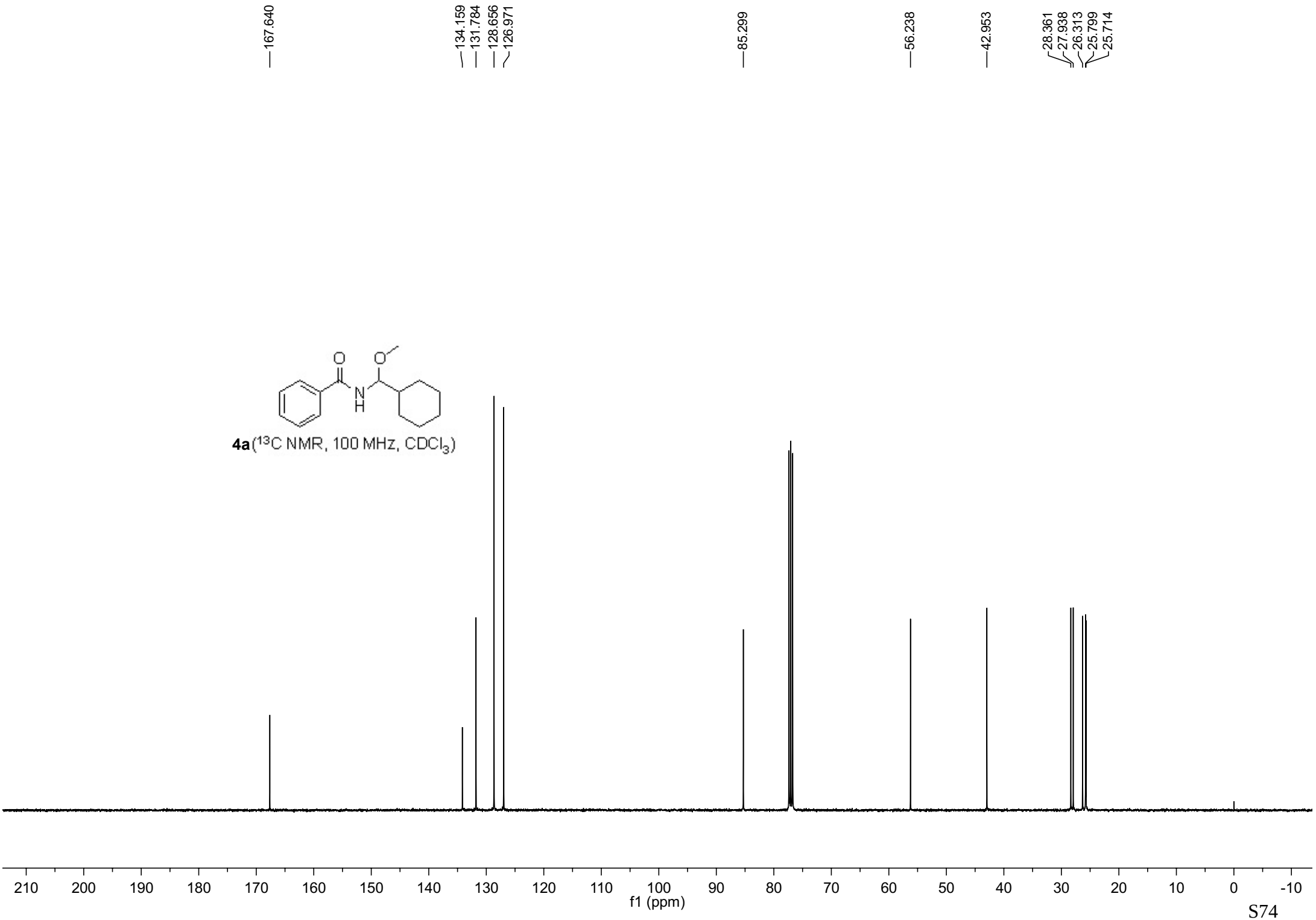


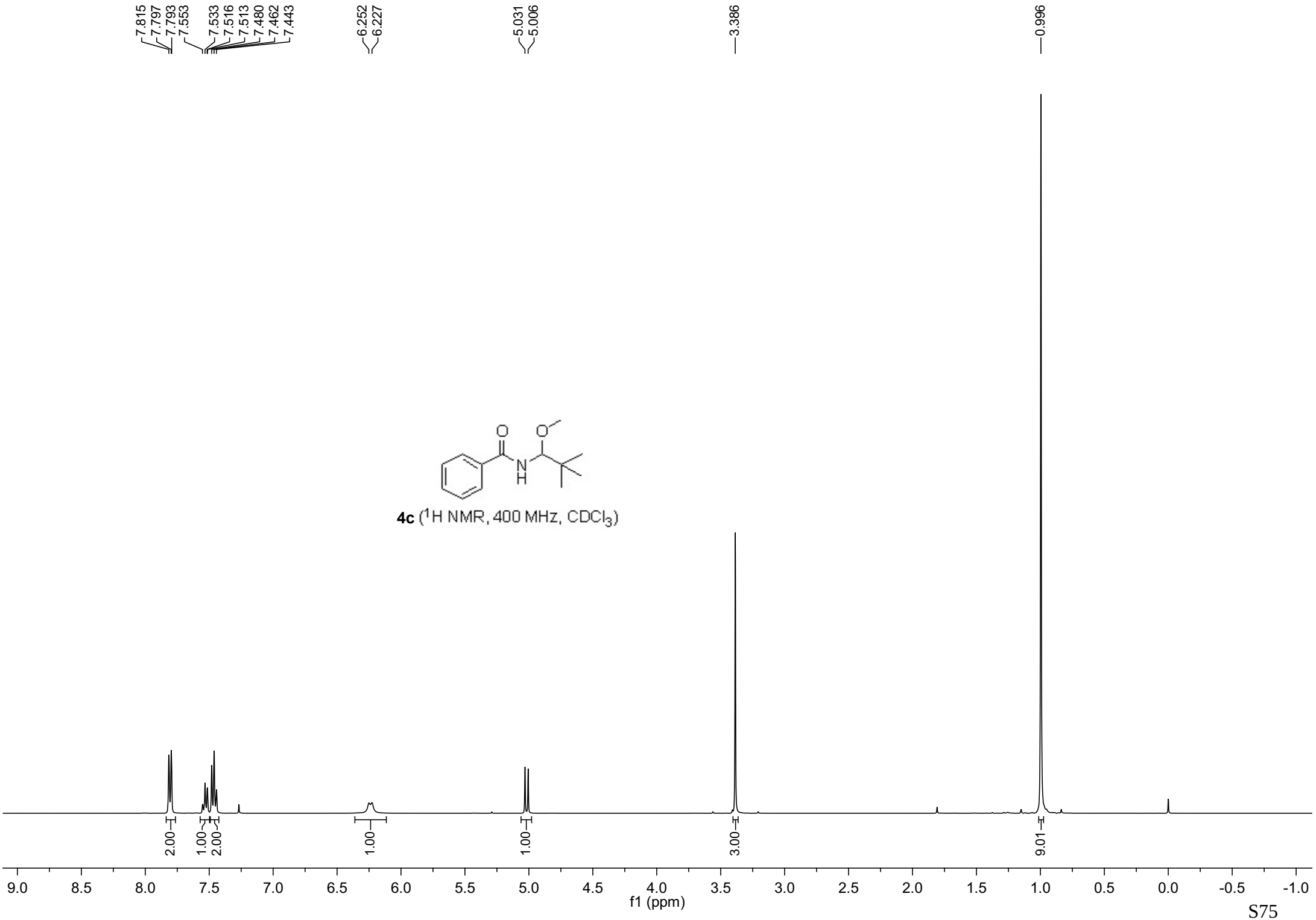
7 (^{1}H NMR, 400 MHz, DMSO-d_6)



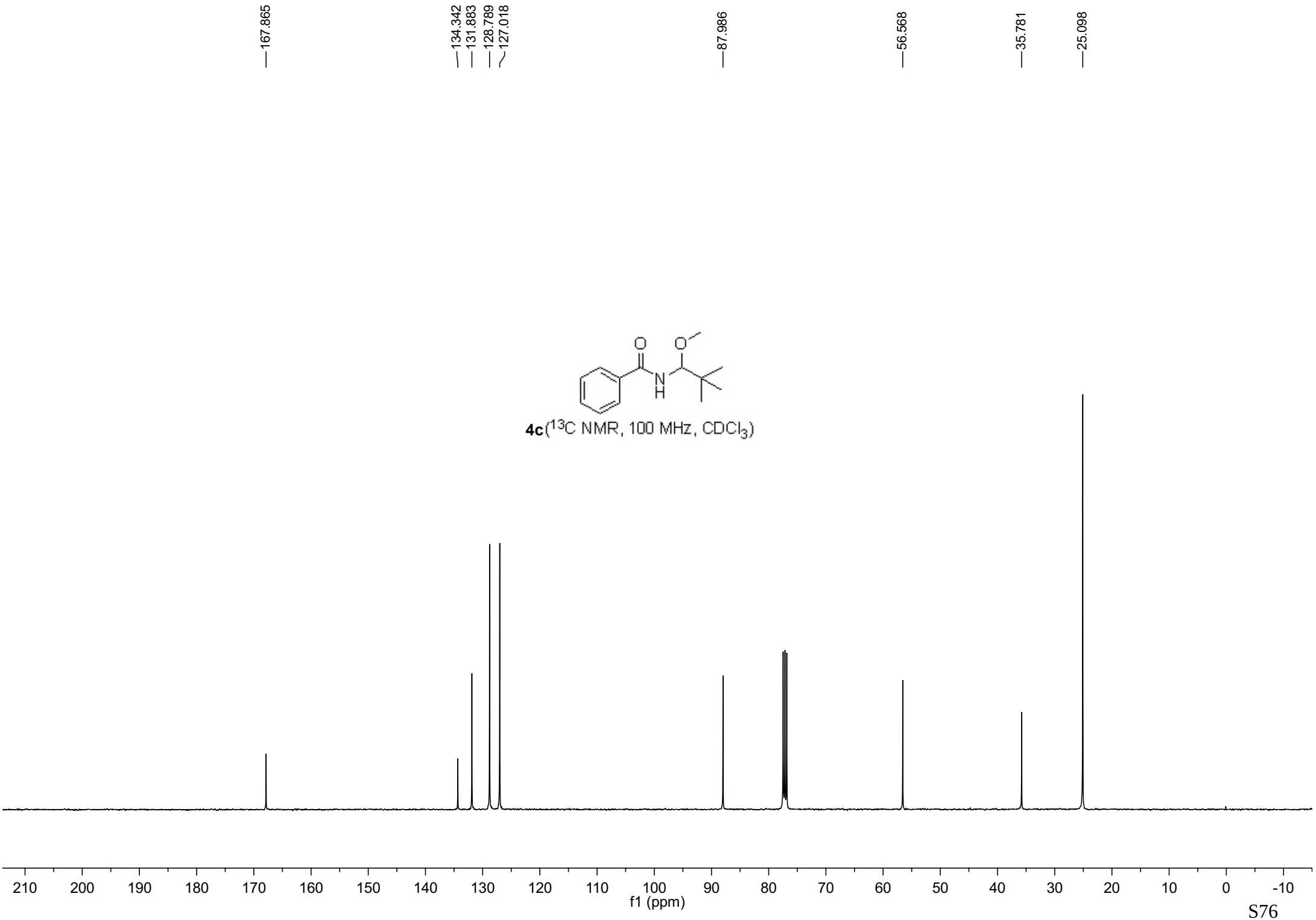
7 (^{13}C NMR, 100 MHz, DMSO-d_6)

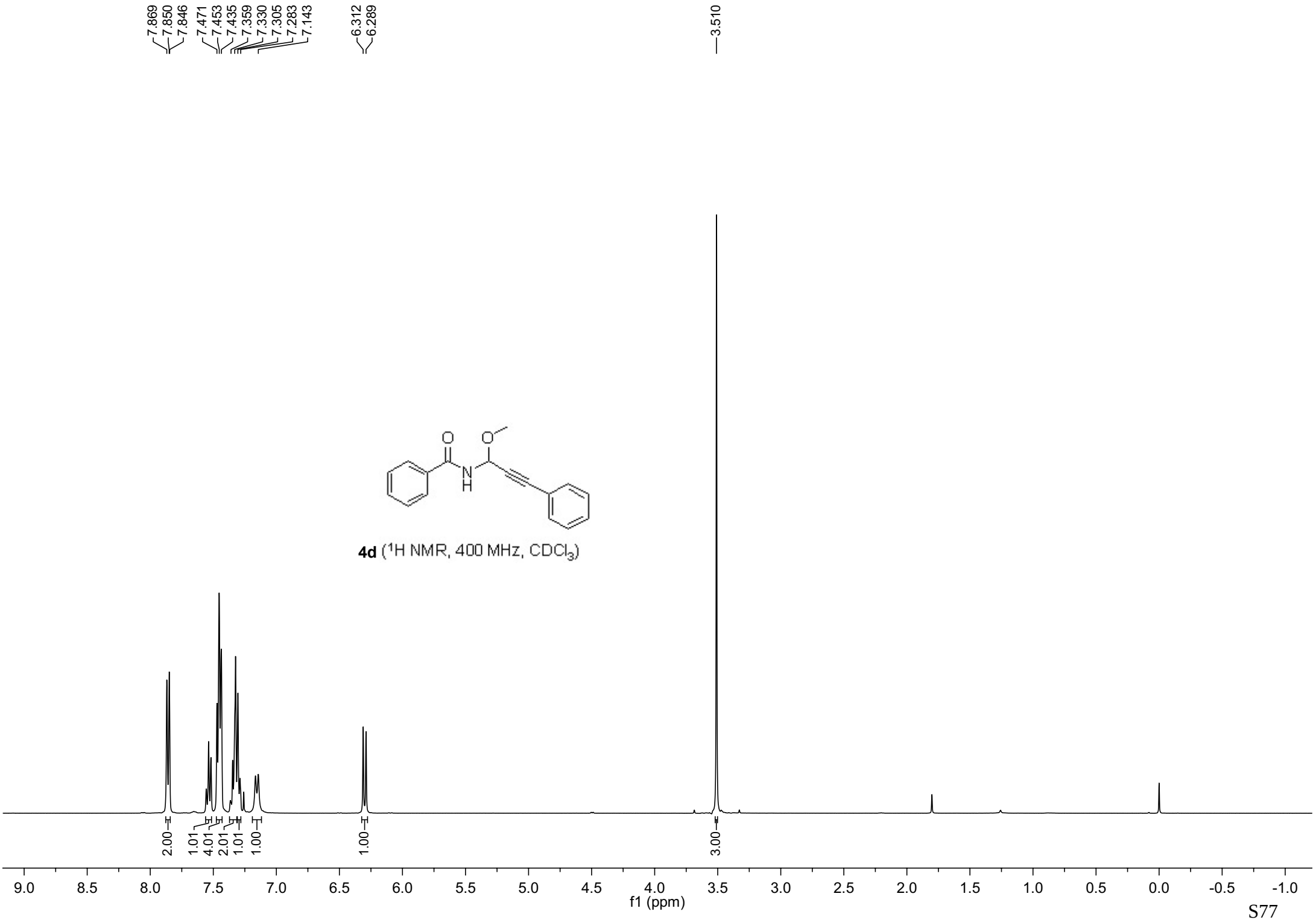

7.826
7.807
7.552
7.518
7.478
7.458
7.440
6.378
6.354
5.131
5.114
5.106
5.090
3.398
1.742
1.627
1.608
1.599
1.591
1.582
1.573
1.562
1.554
1.251
1.242
1.237
1.219
1.211
1.188
1.169
1.150
1.128
1.120
1.098
1.091
1.065
O
O
N
H
4a (1H NMR, 400 MHz, CDCl3)
2.00
1.00
2.00
0.98
1.00
3.01
1.00
3.01
2.01
5.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


167.640
134.159
131.784
128.656
126.971
85.299
56.238
42.953
28.361
27.938
26.313
25.799
25.714
O
O
N
H
4a(13C NMR, 100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

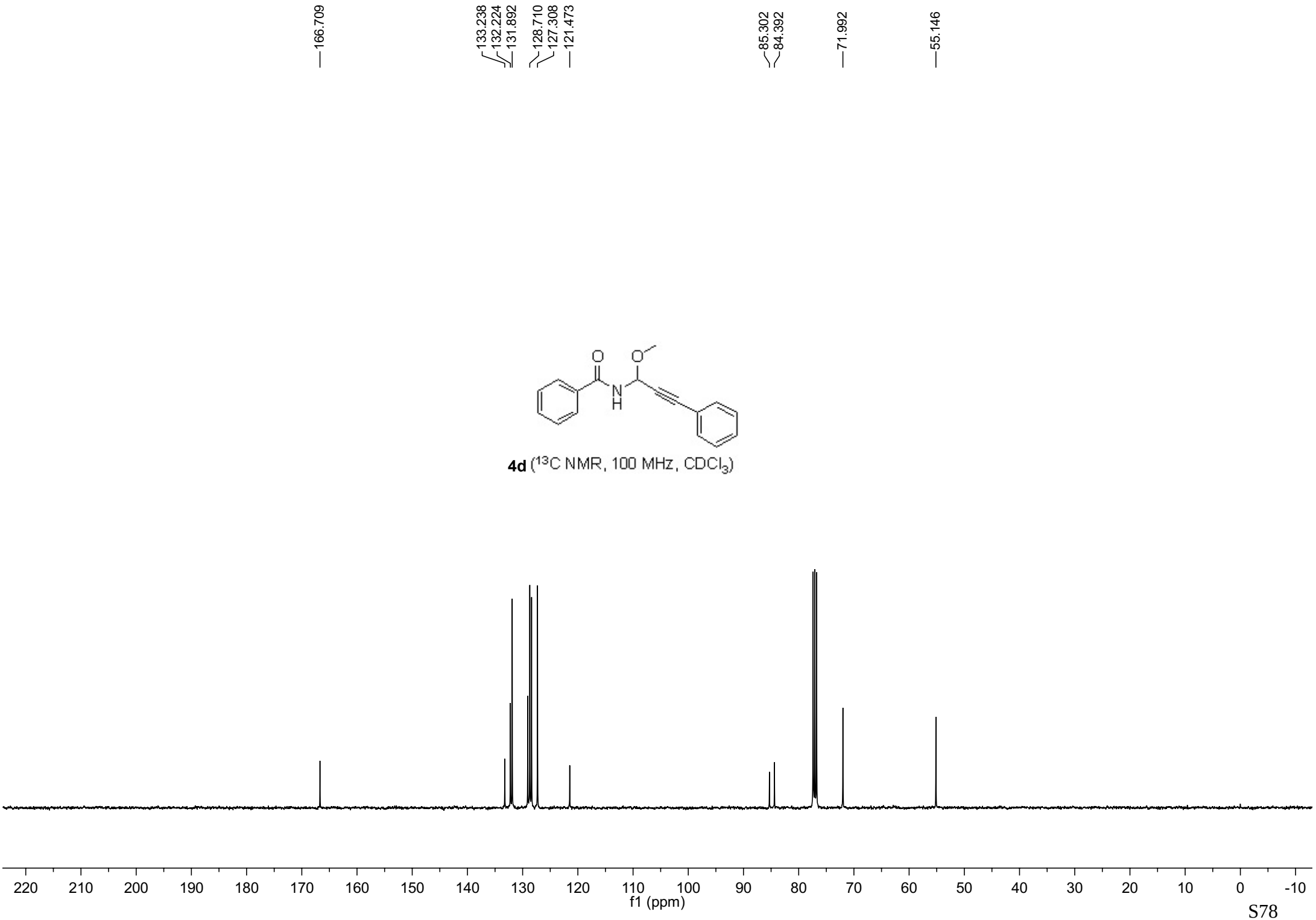


4c (^{1}H NMR, 400 MHz, $CDCl_3$)

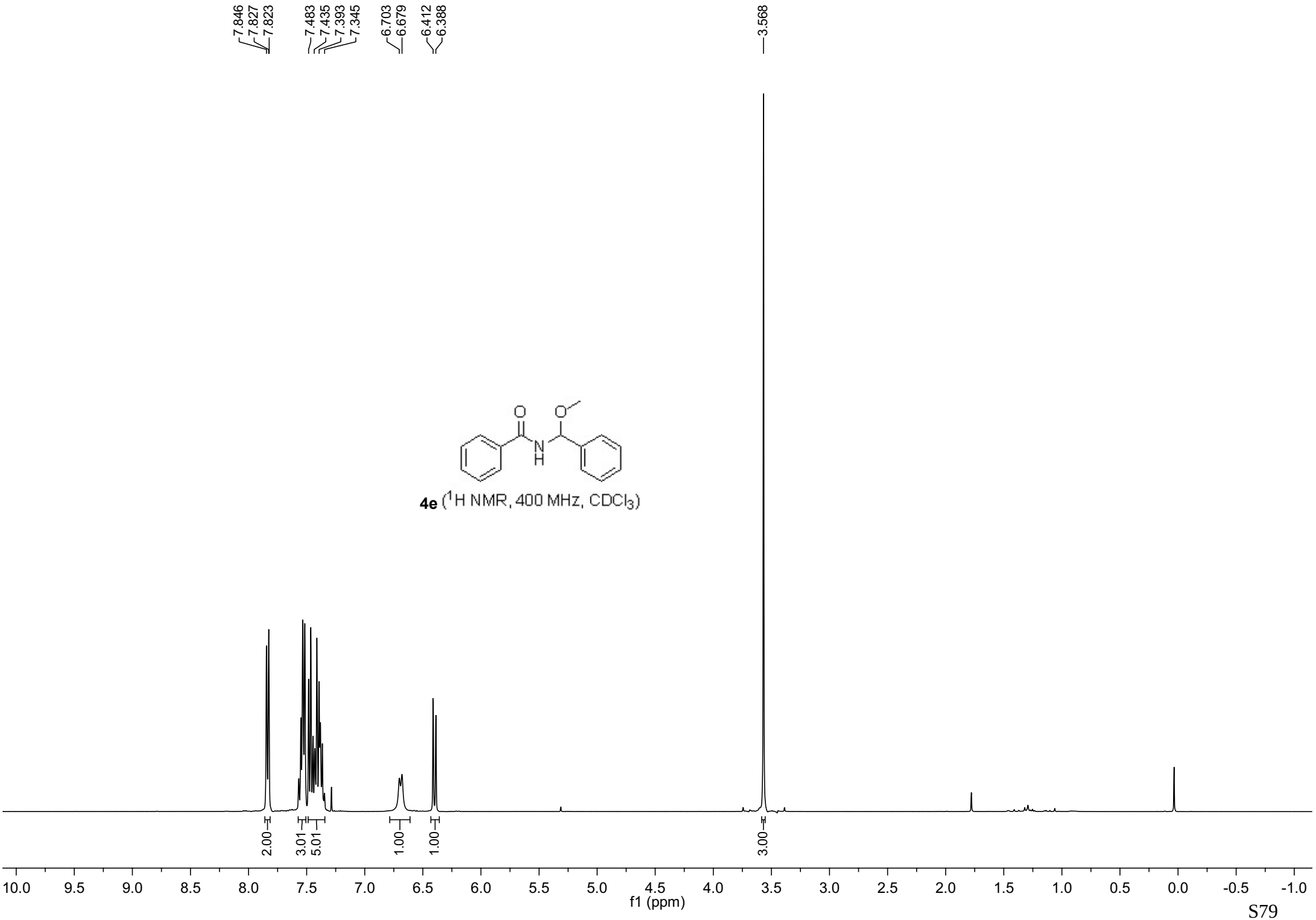

167.865
134.342
131.883
128.789
127.018
87.986
56.568
35.781
25.098
O
O
N
H
4c(13C NMR, 100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



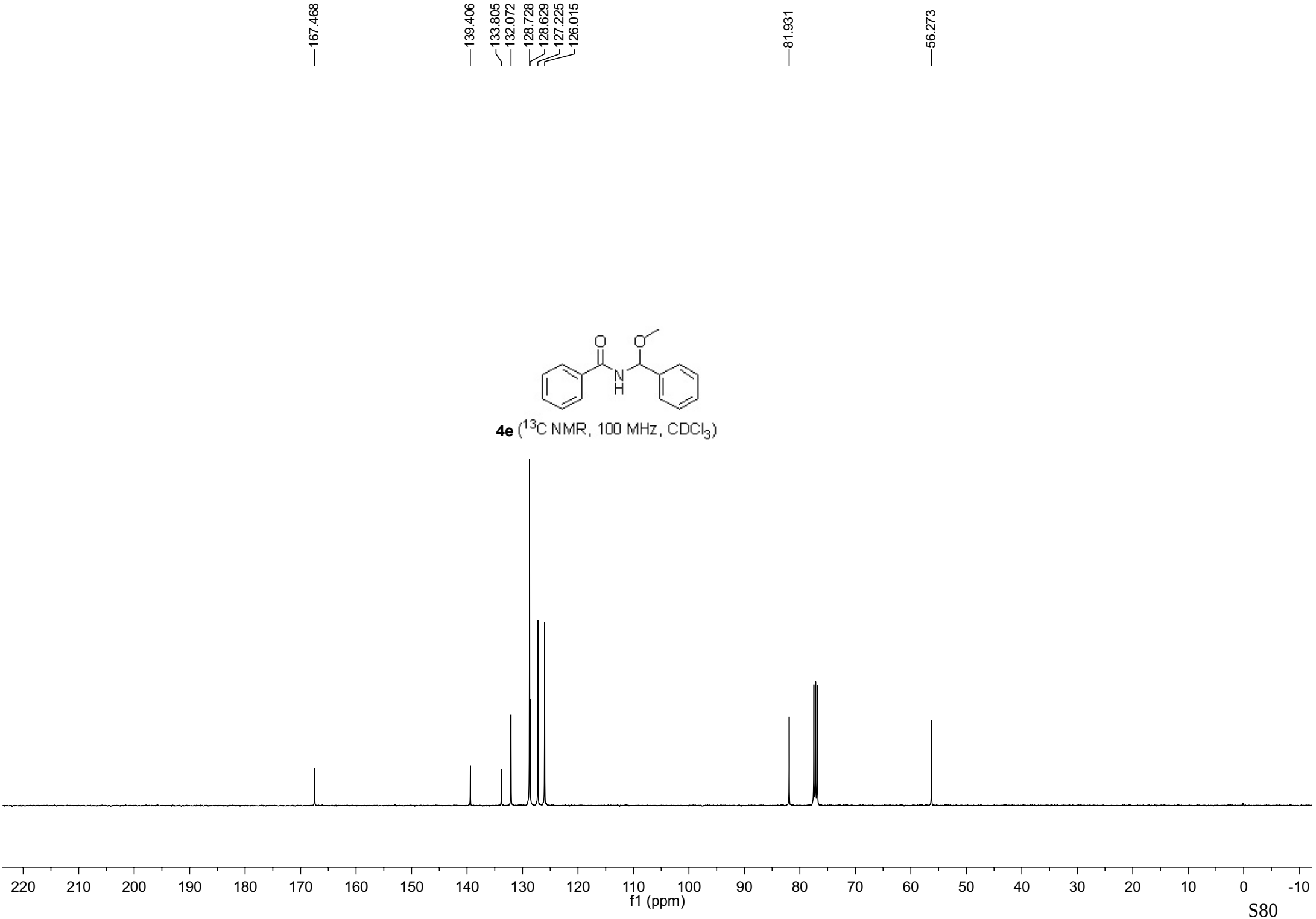
4d (^{1}H NMR, 400 MHz, $CDCl_3$)

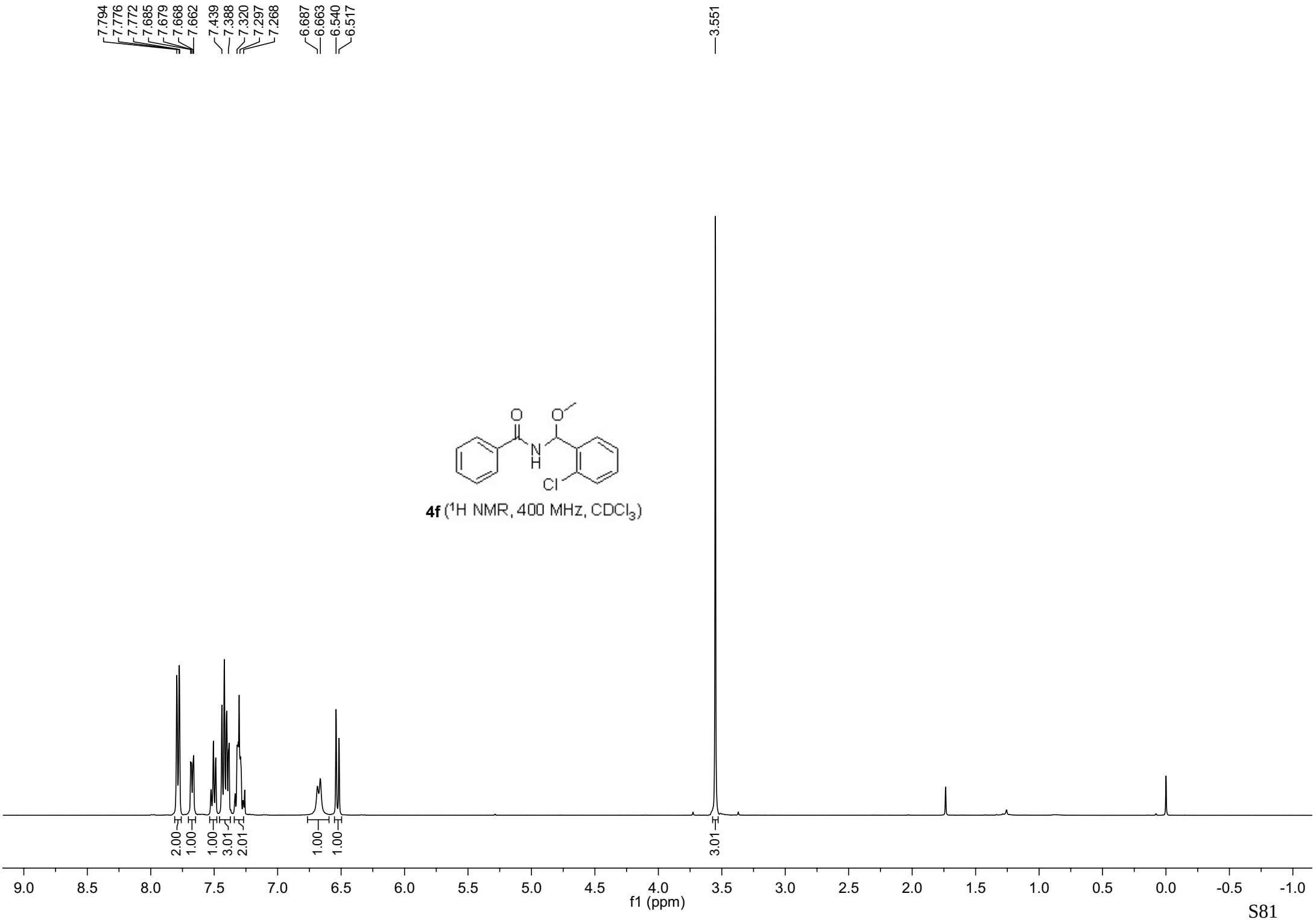


4d (^{13}C NMR, 100 MHz, $CDCl_3$)

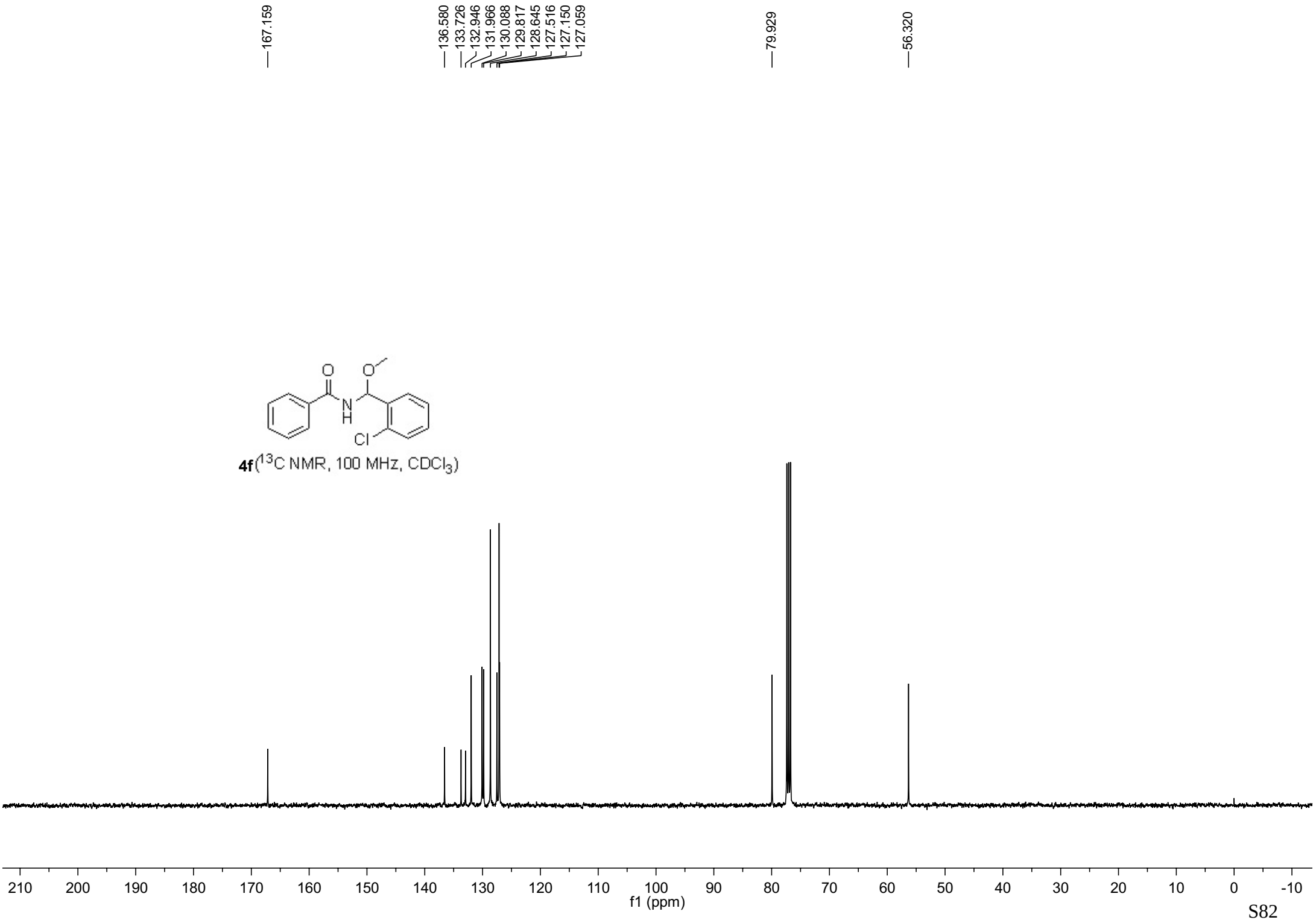


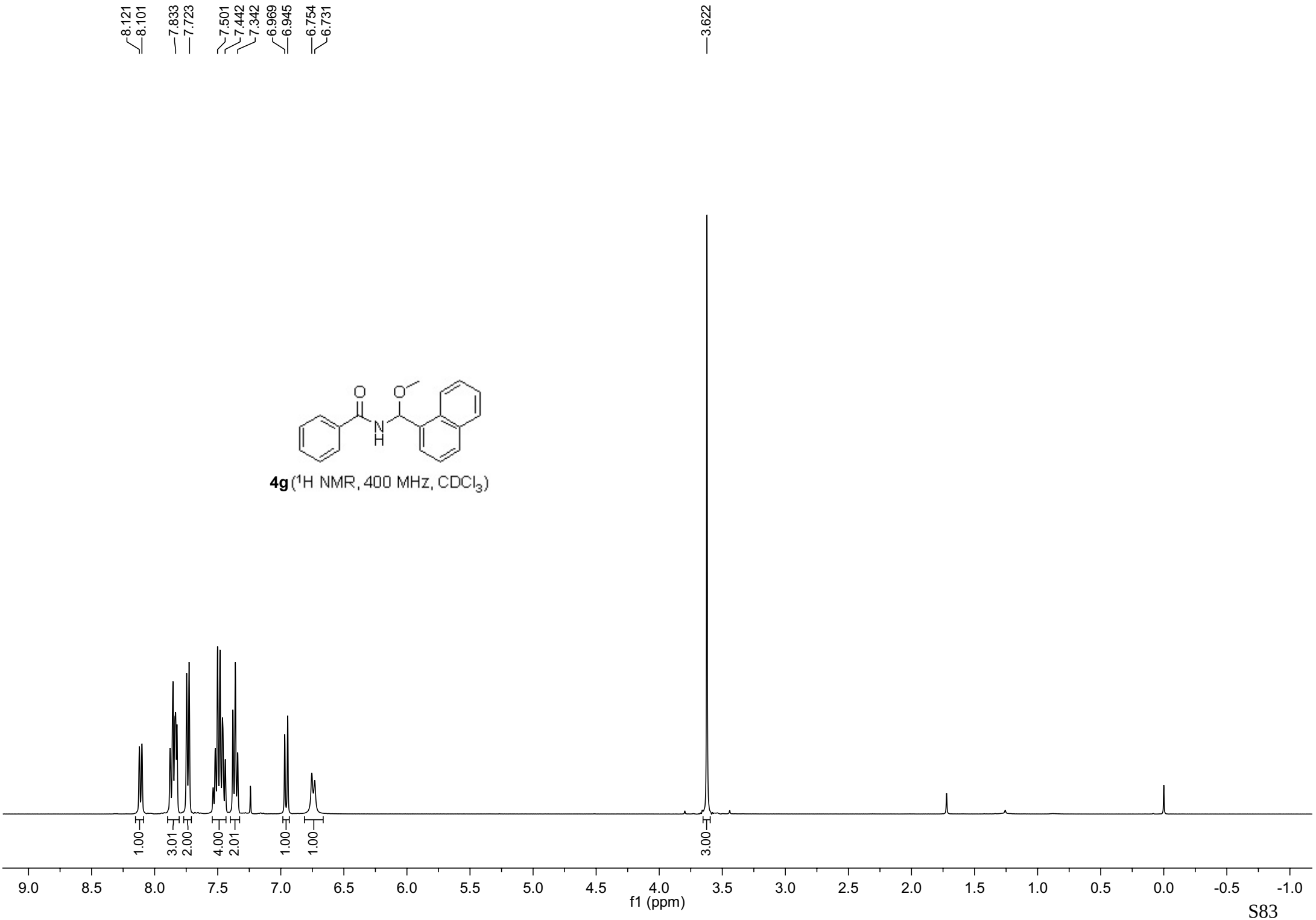
4e (^{1}H NMR, 400 MHz, $CDCl_3$)

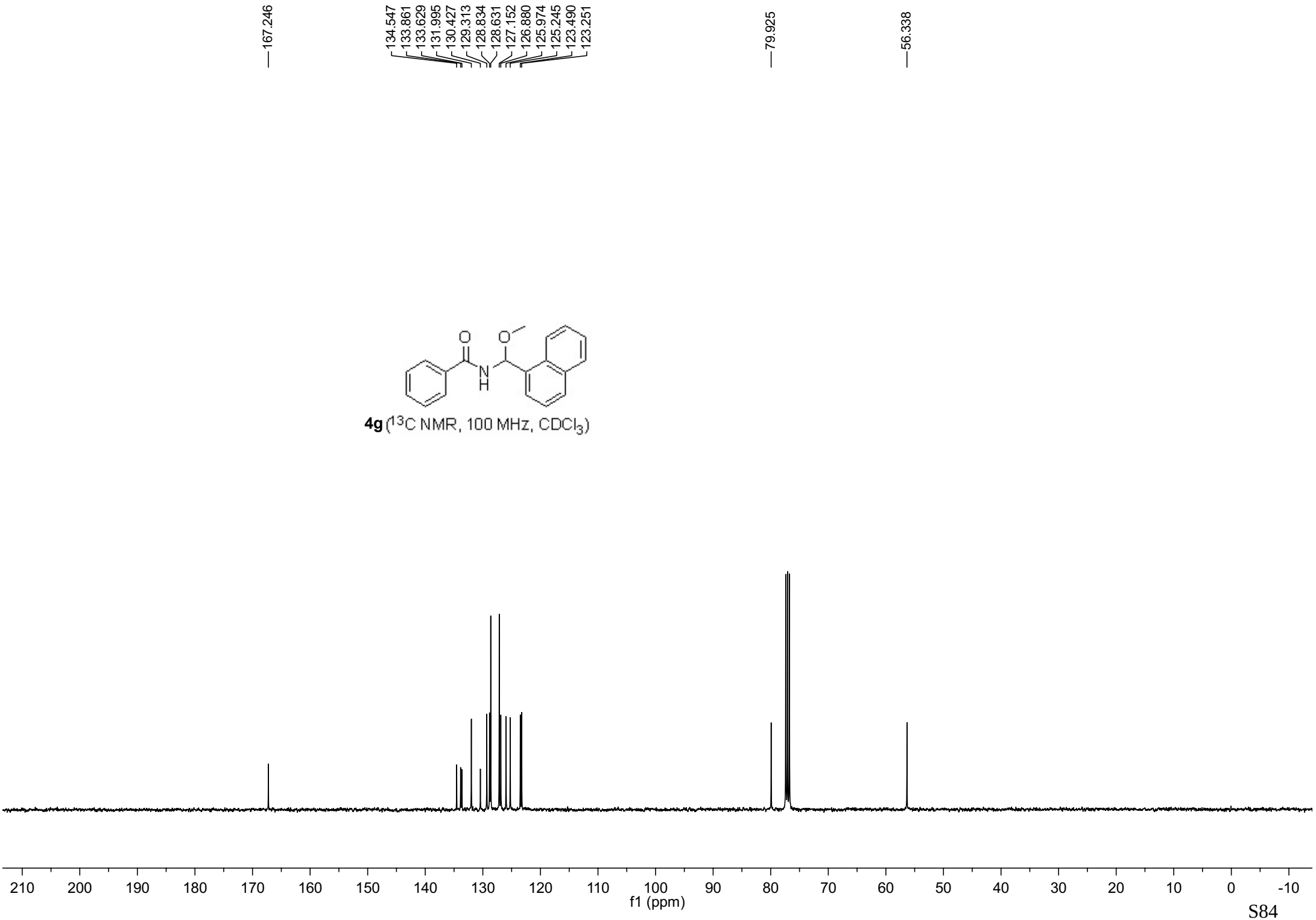

167.468
139.406
133.805
132.072
128.728
128.629
127.225
126.015
81.931
56.273
O
O
N
H
4e (13C NMR, 100 MHz, CDCl3)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



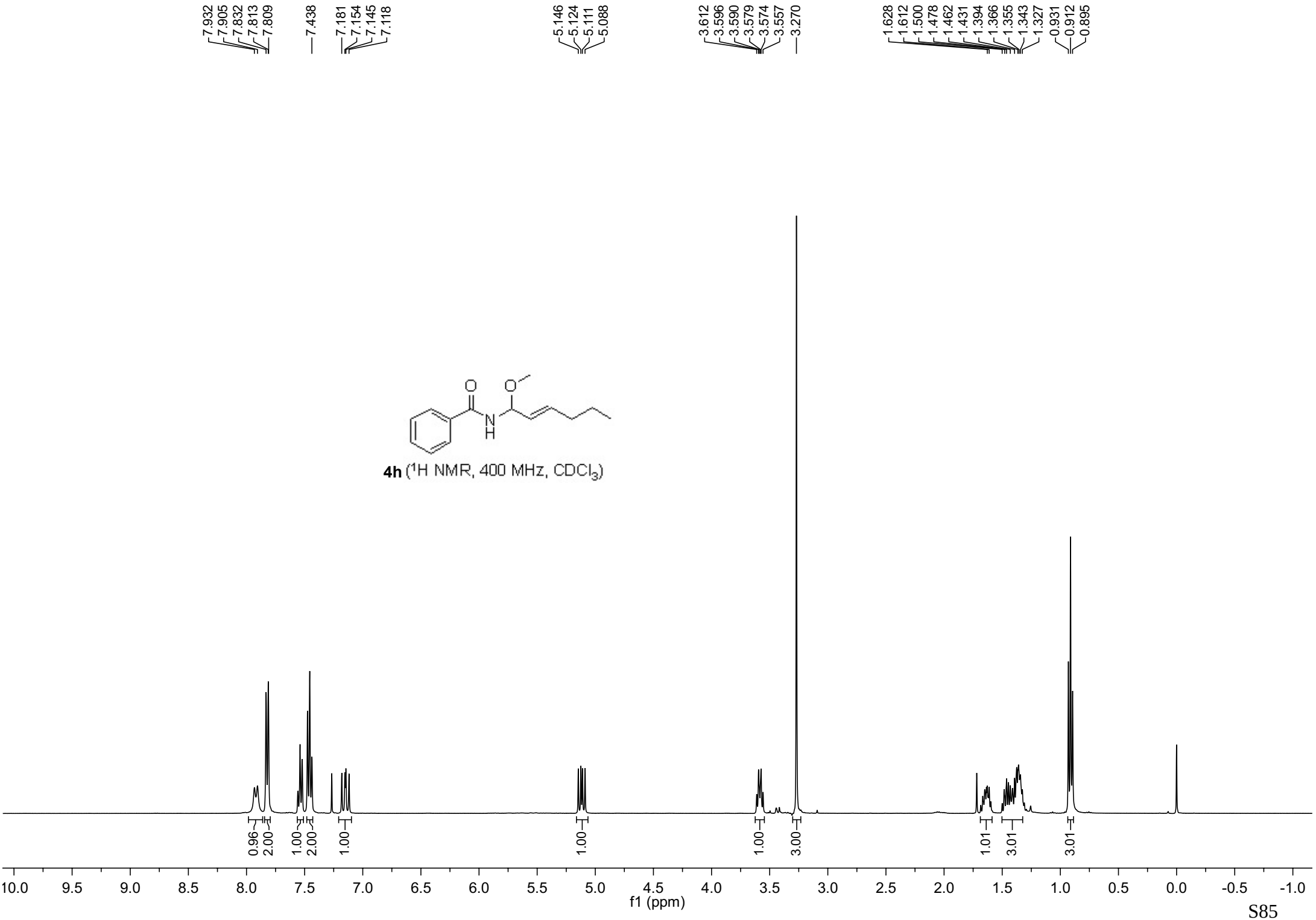
4f (^{1}H NMR, 400 MHz, $CDCl_3$)

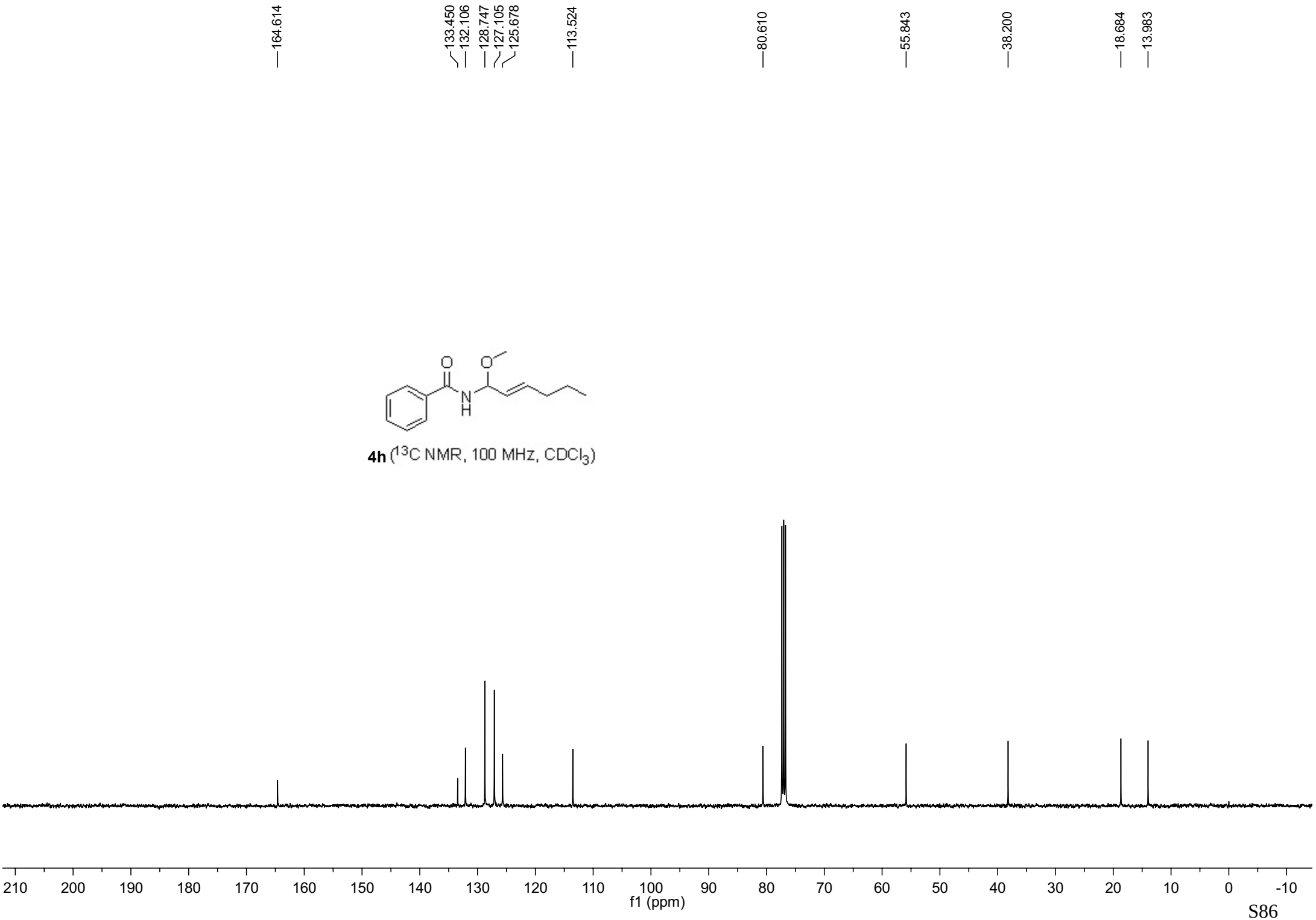

167.159
136.580
133.726
132.946
131.966
130.088
129.817
128.645
127.516
127.150
127.059
79.929
56.320
O
O
N
H
Cl
4f(13C NMR, 100 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.121
8.101
7.833
7.723
7.501
7.442
7.342
6.969
6.945
6.754
6.731
3.622
O
O
N
H
4g (1H NMR, 400 MHz, CDCl3)
1.00
3.01
2.00
4.00
2.01
1.00
1.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

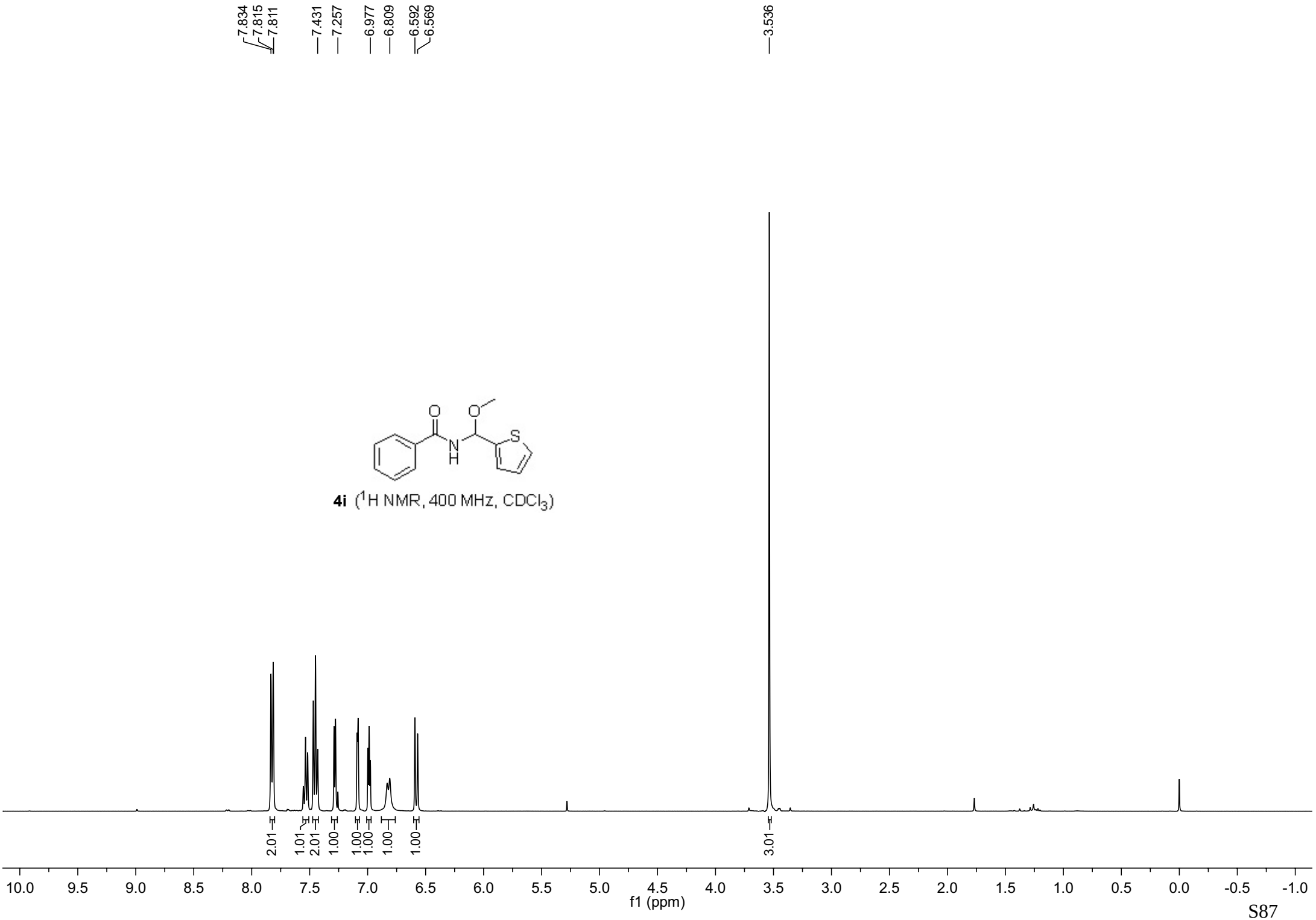


4g (^{13}C NMR, 100 MHz, $CDCl_3$)

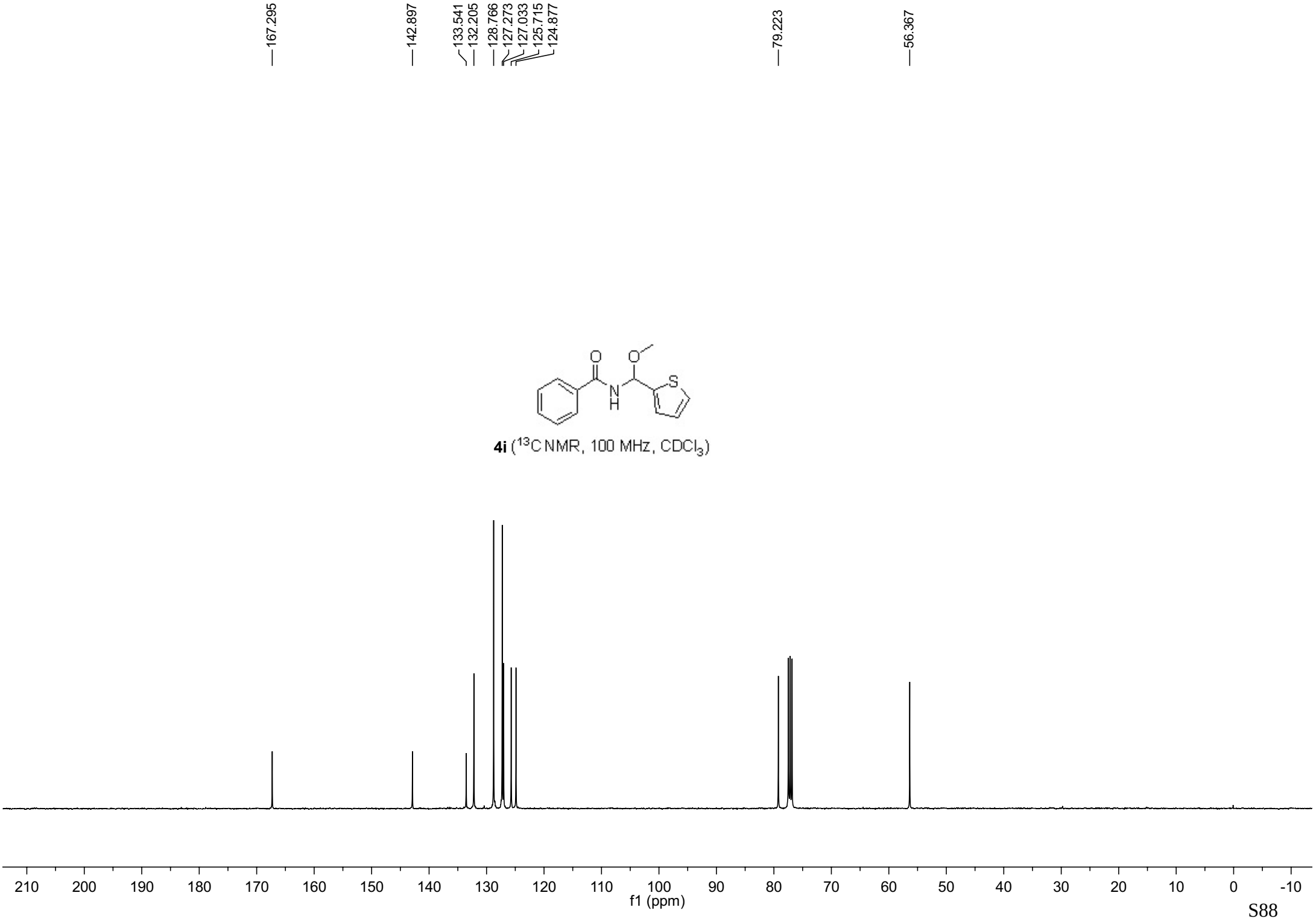

7.932
7.905
7.832
7.813
7.809
7.438
7.181
7.154
7.145
7.118
5.146
5.124
5.111
5.088
3.612
3.596
3.590
3.579
3.574
3.557
3.270
1.628
1.612
1.500
1.478
1.462
1.431
1.394
1.366
1.355
1.343
1.327
0.931
0.912
0.895
O
O
N
H
4h (1H NMR, 400 MHz, CDCl3)
0.96
2.00
1.00
2.00
1.00
1.00
1.00
3.00
1.01
3.01
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

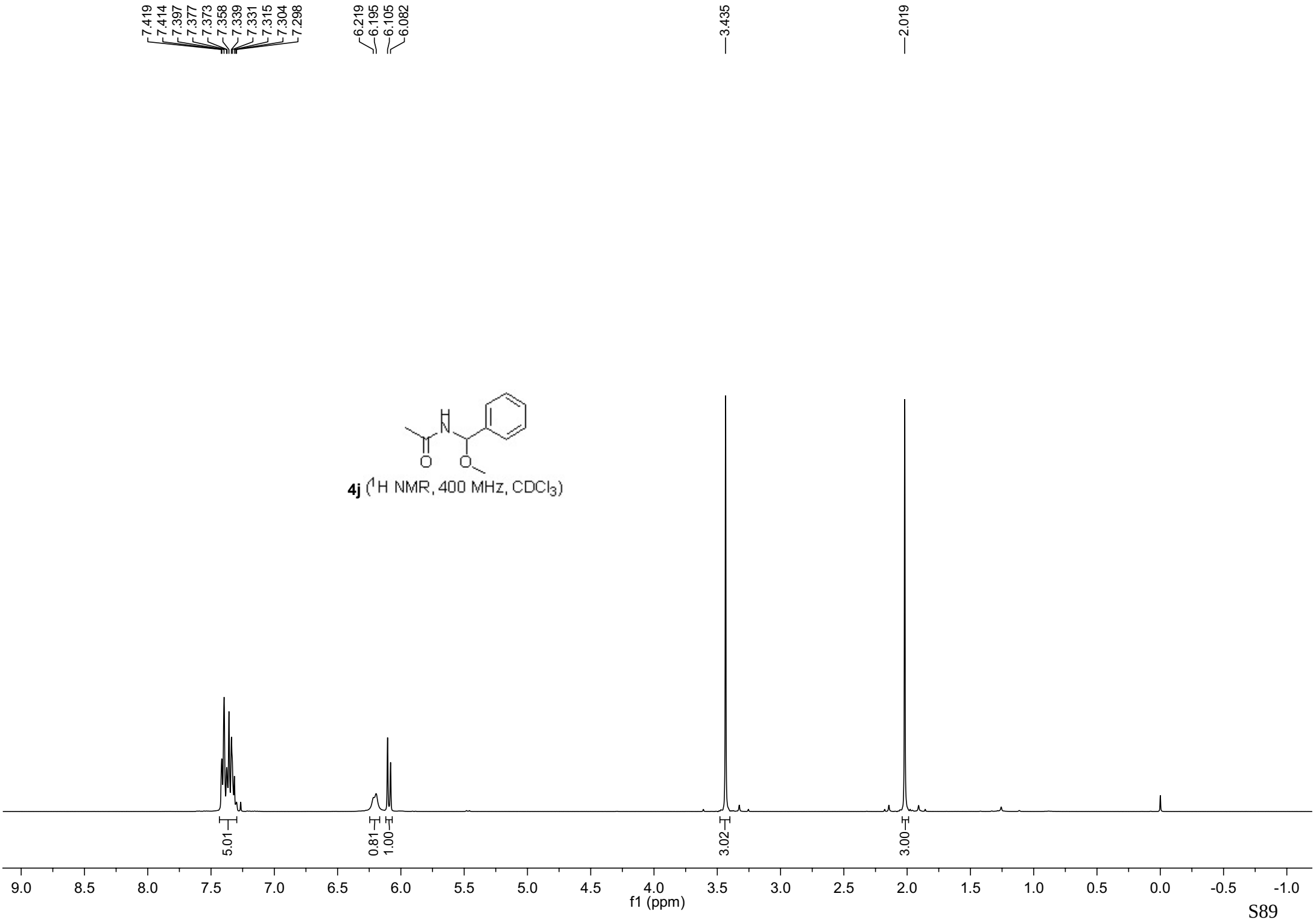


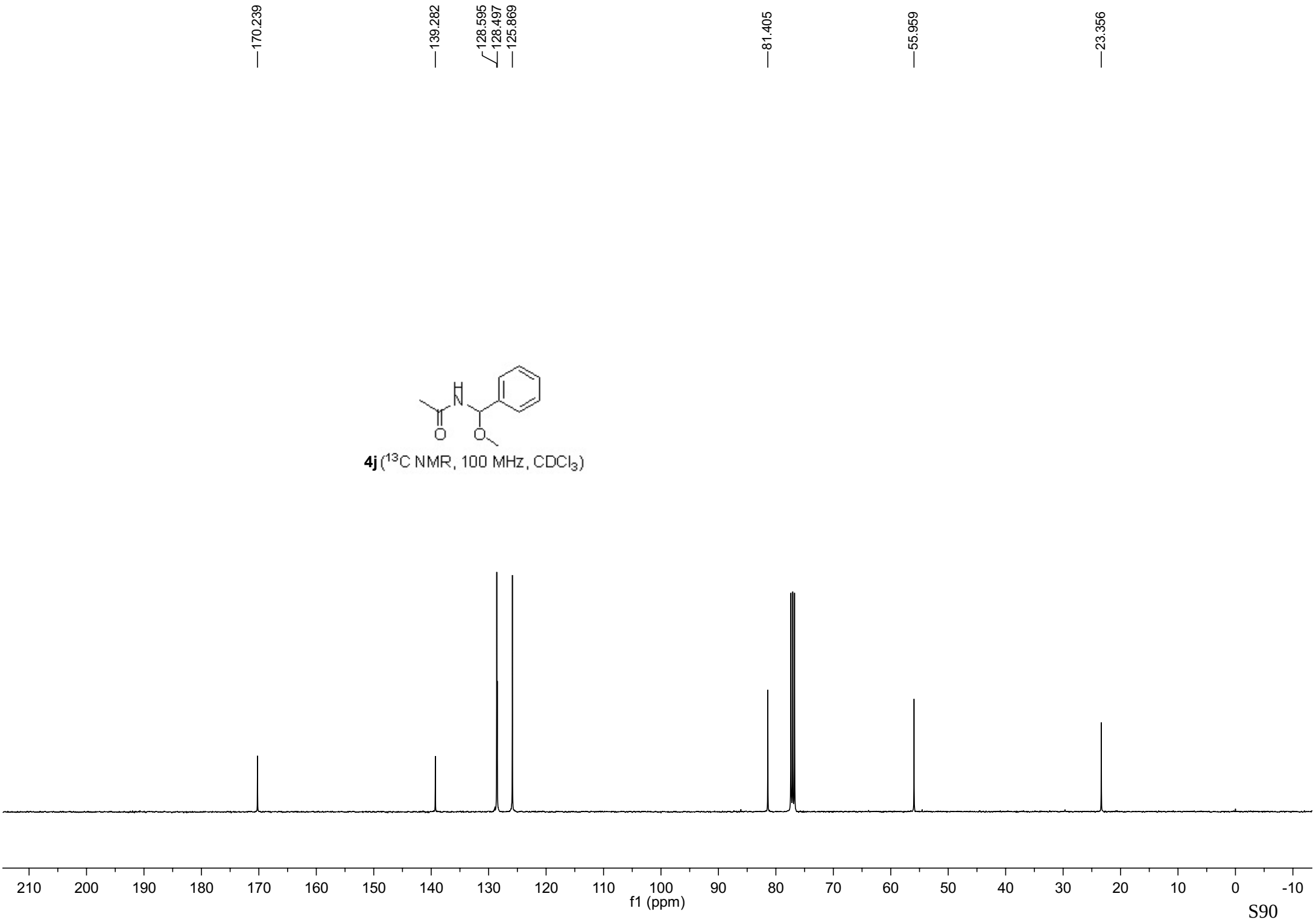
4h (^{13}C NMR, 100 MHz, $CDCl_3$)

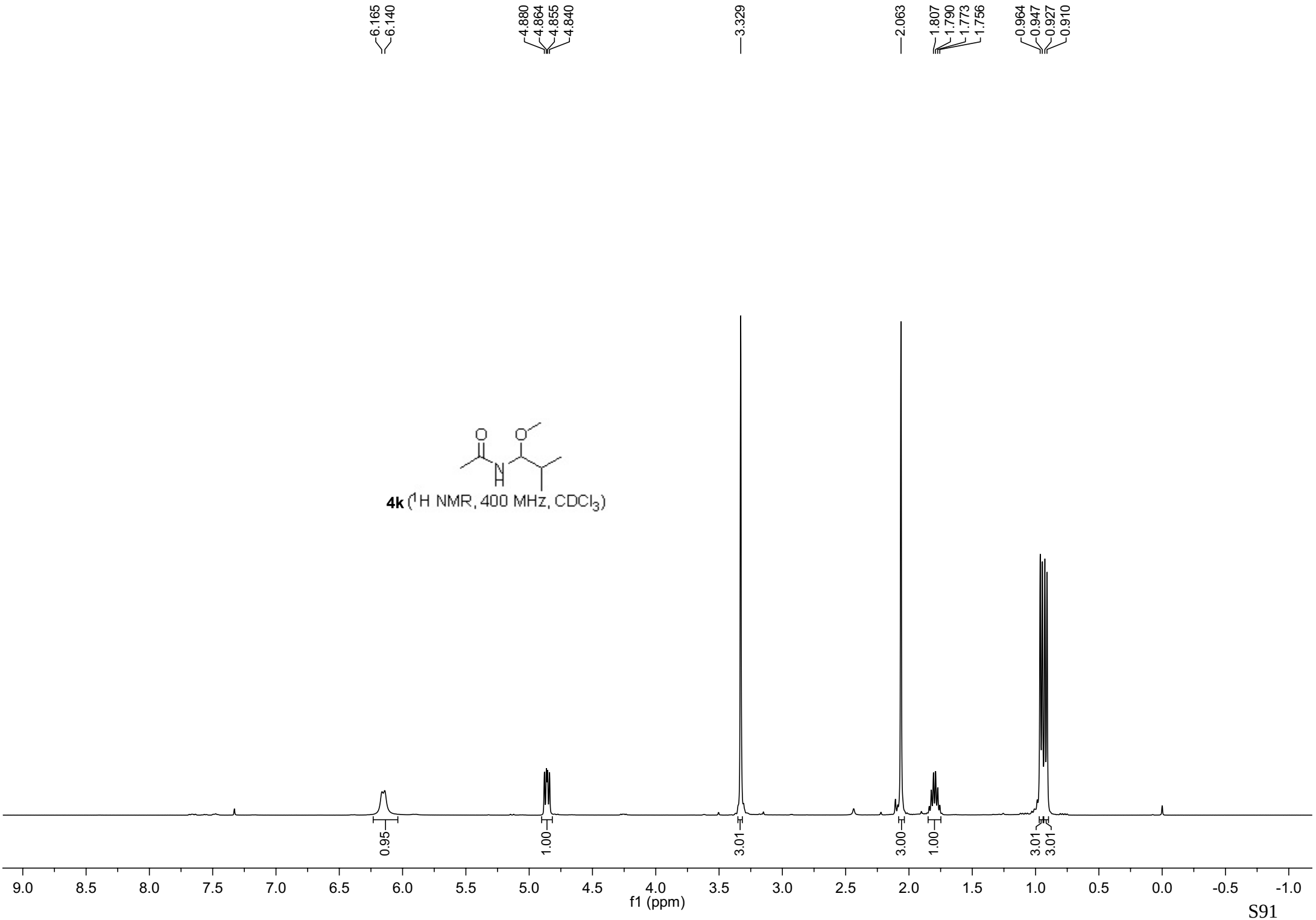


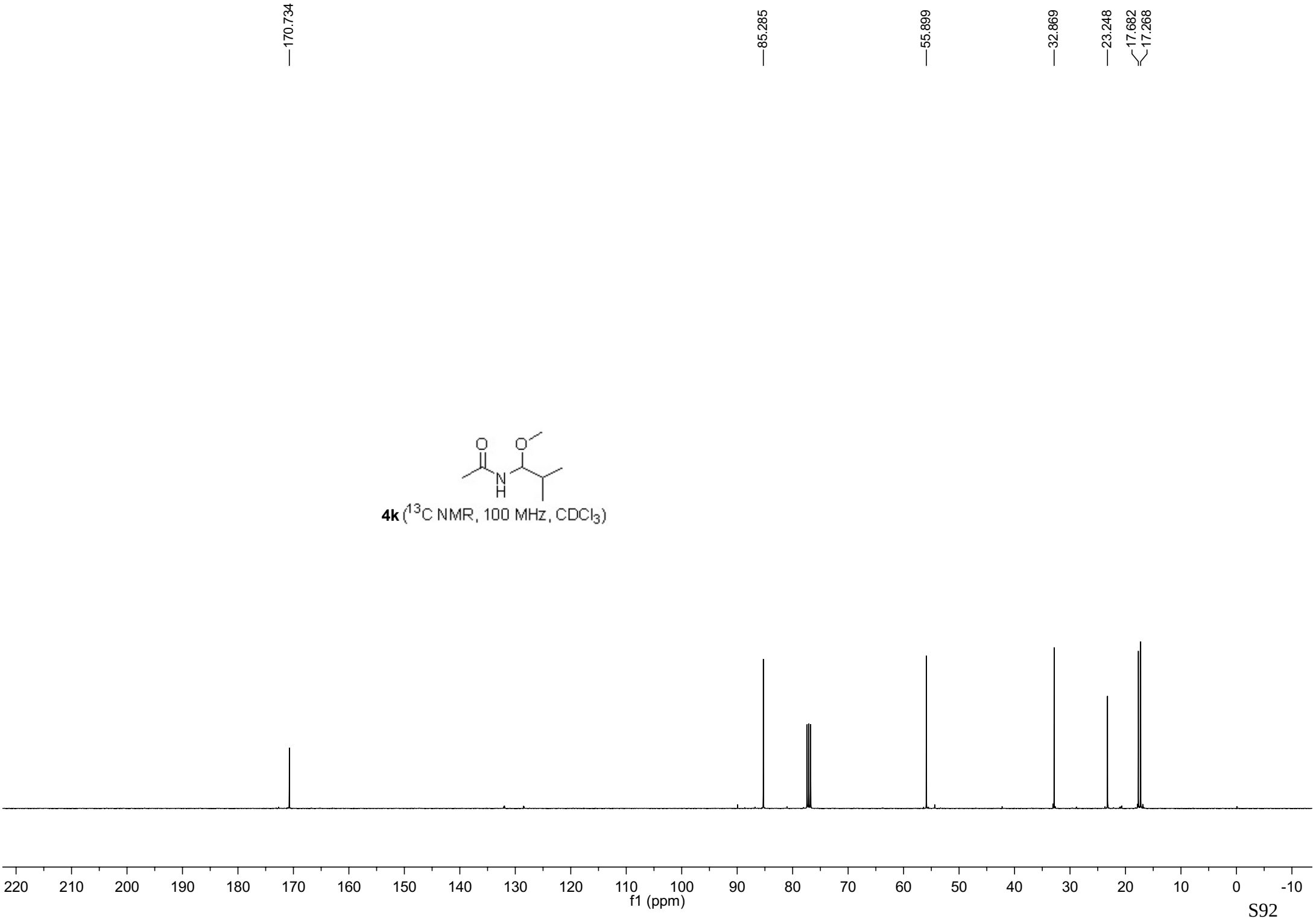
4i (^{1}H NMR, 400 MHz, $CDCl_3$)


167.295
142.897
133.541
132.205
128.766
127.273
127.033
125.715
124.877
79.223
56.367
O
O
N
H
S
4i (13C NMR, 100 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

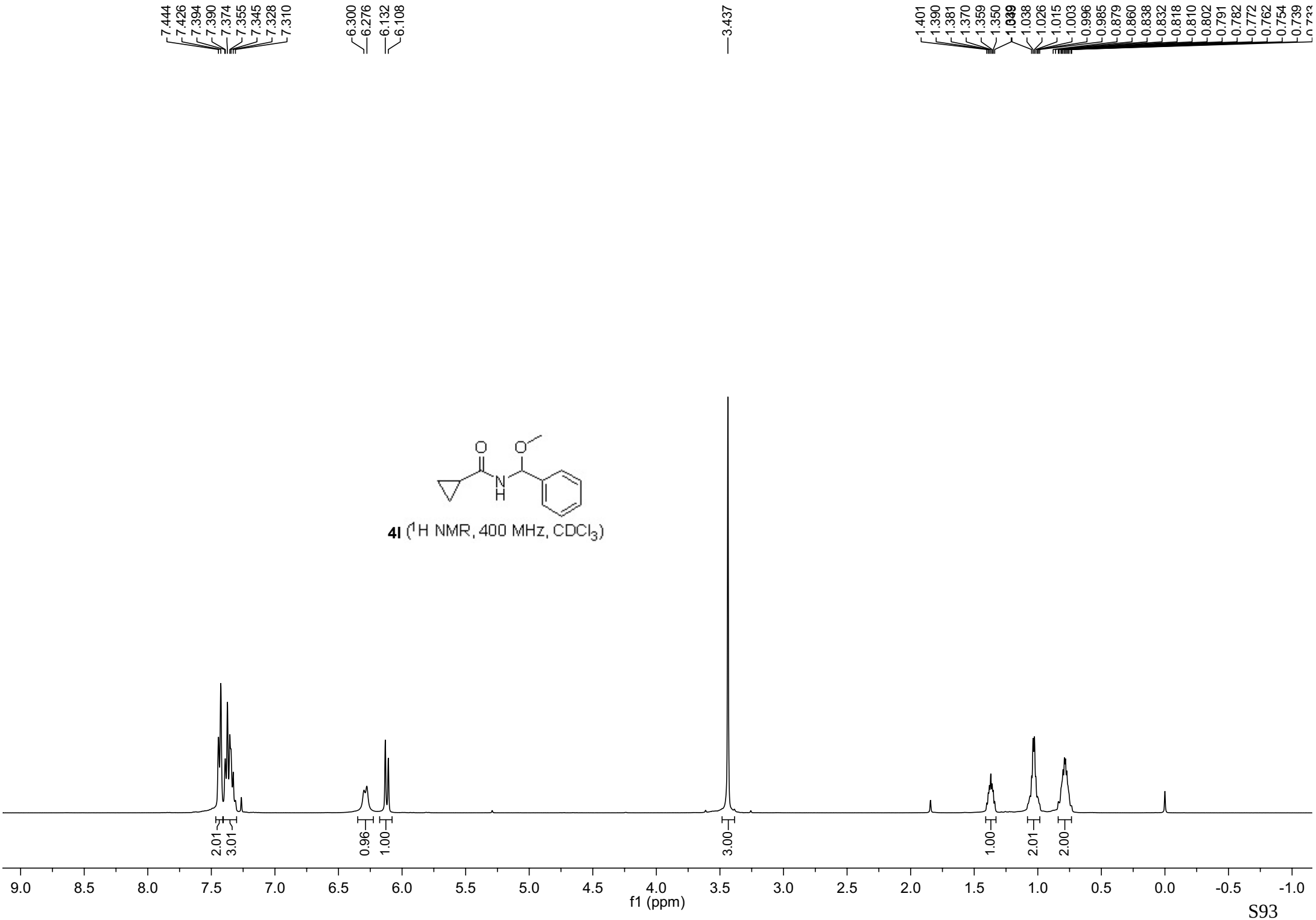

7.419
7.414
7.397
7.377
7.373
7.358
7.339
7.331
7.315
7.304
7.298
6.219
6.195
6.105
6.082
3.435
2.019
4j (1H NMR, 400 MHz, CDCl3)
5.01
0.81
1.00
3.02
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

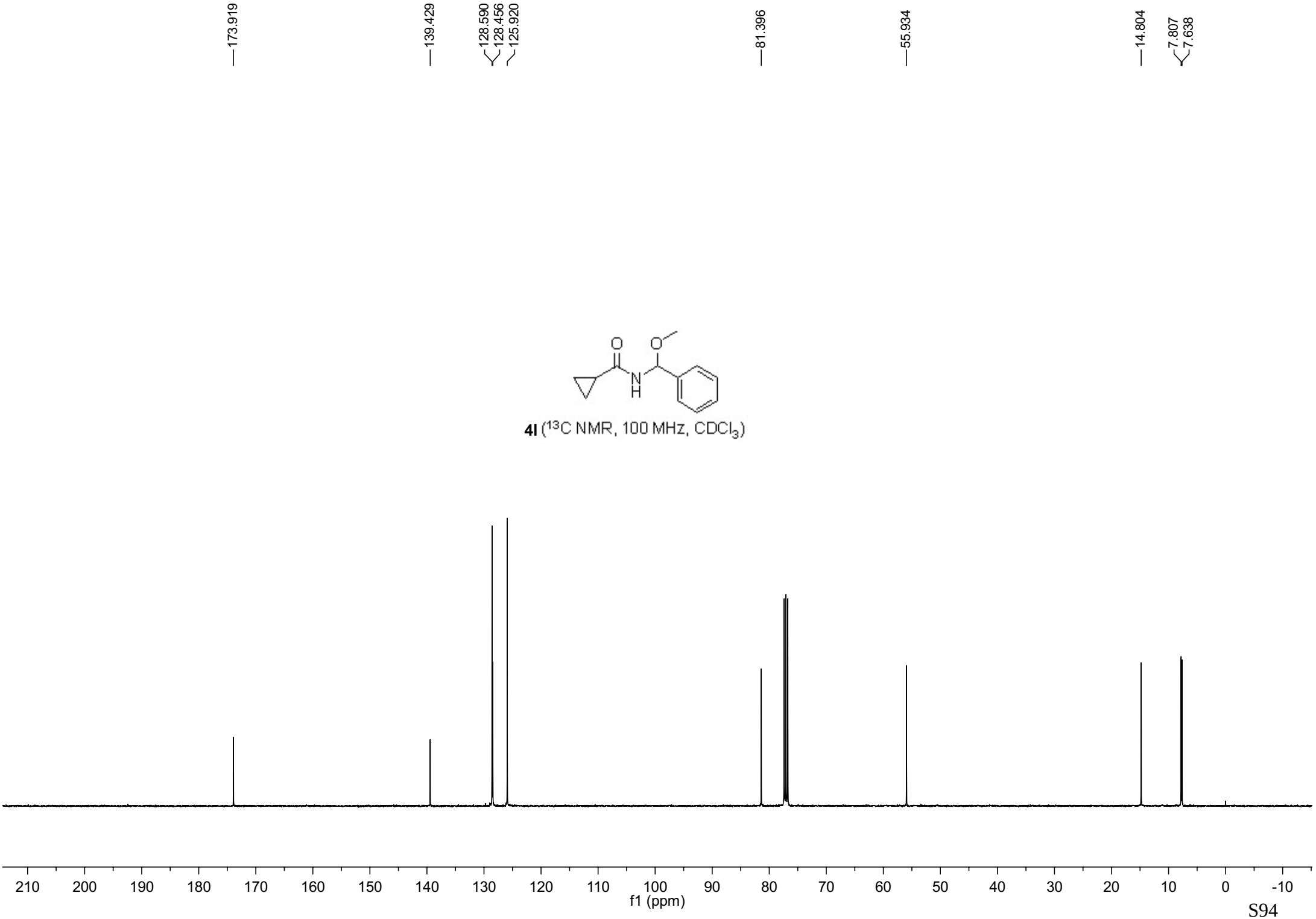

170.239
139.282
128.595
128.497
125.869
81.405
55.959
23.356
H
N
O
O
4j (13C NMR, 100 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


6.165
6.140
4.880
4.864
4.855
4.840
3.329
2.063
1.807
1.790
1.773
1.756
0.964
0.947
0.927
0.910
4k (1H NMR, 400 MHz, CDCl3)
0.95
1.00
3.01
3.00
1.00
3.01
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



4k (^{13}C NMR, 100 MHz, $CDCl_3$)


7.444
7.426
7.394
7.390
7.374
7.355
7.345
7.328
7.310
6.300
6.276
6.132
6.108
3.437
1.401
1.390
1.381
1.370
1.359
1.350
1.038
1.026
1.015
1.003
0.996
0.985
0.879
0.860
0.838
0.832
0.818
0.810
0.802
0.791
0.782
0.772
0.762
0.754
0.739
O
O
N
H
4l (1H NMR, 400 MHz, CDCl3)
2.01
3.01
0.96
1.00
3.00
1.00
2.01
2.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



4l (^{13}C NMR, 100 MHz, $CDCl_3$)

Analysis Report

<Sample Information>

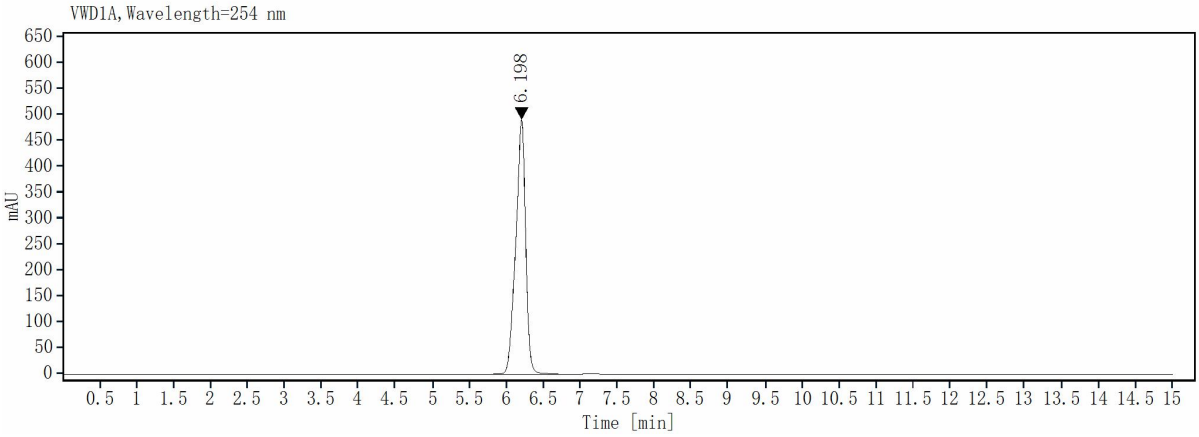
Sample Name: **6e** Injection Volume: 3.000

Sample Vial Number: P1-A3

Sequence Acquired Date: 2022-01-19 18:44:49+08:00

Injection Acq Method Name: Methods-2.amx

Injection Acquired By: System Administrator



Peak #	Ret.Time	Area	Height	Area%
1	6.198	4575.21	491.37	100%
Total		4575.21	491.37	100%

Analysis Report

<Sample Information>

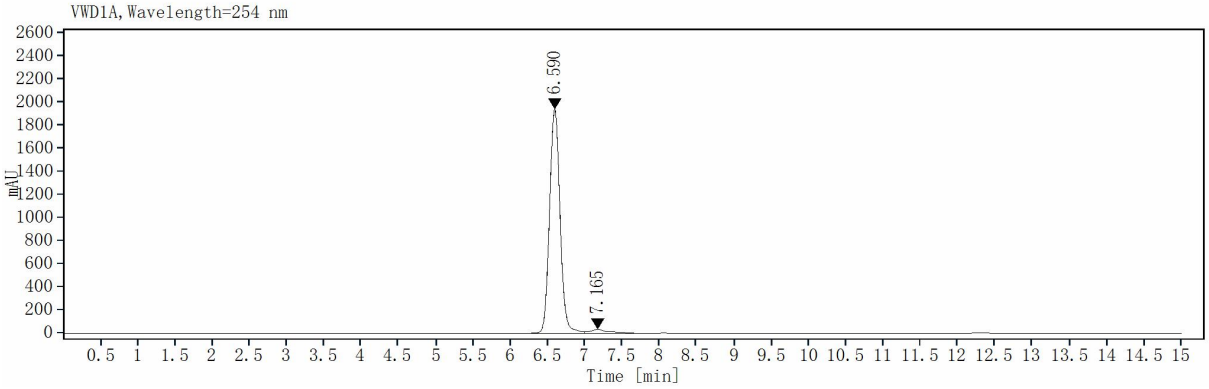
Sample Name: **6z** Injection Volume: 3.000

Sample Vial Number: P1-A1

Sequence Acquired Date: 2022-01-19 16:44:59+08:00

Injection Acq Method Name: Methods-1.amx

Injection Acquired By: System Administrator



Peak #	Ret.Time	Area	Height	Area%
1	6.590	19628.00	1944.58	98.92%
2	7.165	241.30	35.54	1.08%
Total		19842.30	1980.12	100%

Analysis Report

<Sample Information>

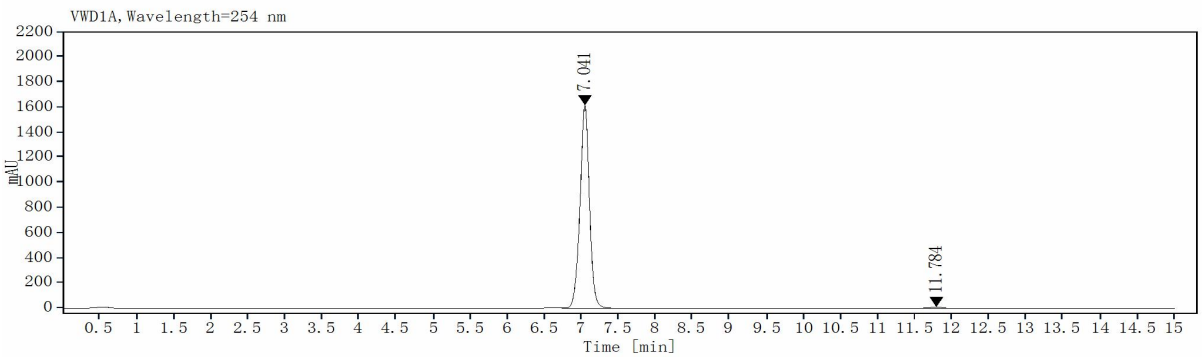
Sample Name: **6aa** Injection Volume: 3.000

Sample Vial Number: P1-A2

Sequence Acquired Date: 2022-01-19 17:19:52+08:00

Injection Acq Method Name: Methods-1.amx

Injection Acquired By: System Administrator



Peak #	Ret.Time	Area	Height	Area%
1	7.041	14501.50	1615.51	99.32%
2	11.784	99.29	11.01	0.68%
Total		14600.79	1626.52	100%

Analysis Report

<Sample Information>

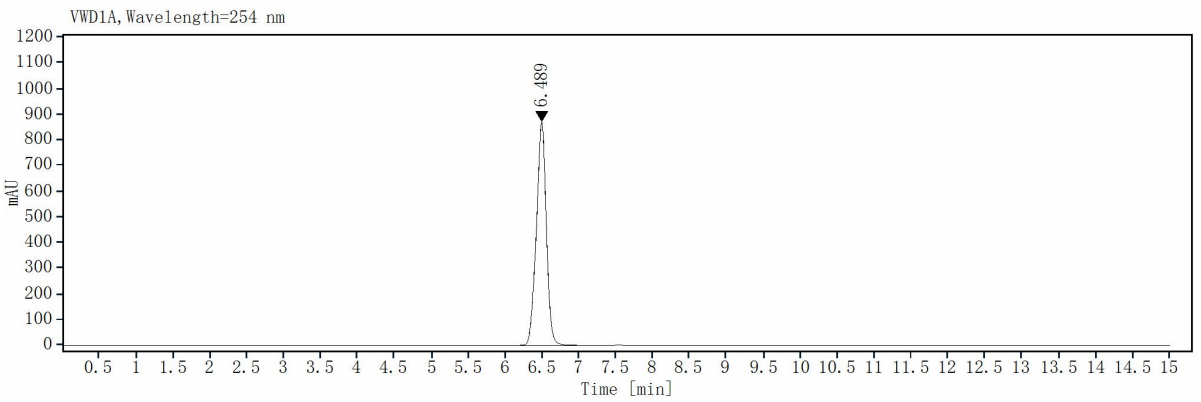
Sample Name: **6ag** Injection Volume: 3.000

Sample Vial Number: P1-A4

Sequence Acquired Date: 2022-01-19 19:07:05+08:00

Injection Acq Method Name: Methods-2.amx

Injection Acquired By: System Administrator



Peak #	Ret.Time	Area	Height	Area%
1	6.489	8205.32	870.13	100%
Total		8205.32	870.13	100%