Supplementary Information

PPh_3-catalyzed intramolecular cyclization of hydroxypropargylamides: Synthesis of structurally diverse morpholinone derivatives

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Received 31 January 2022; accepted (revised) 15 March 2022

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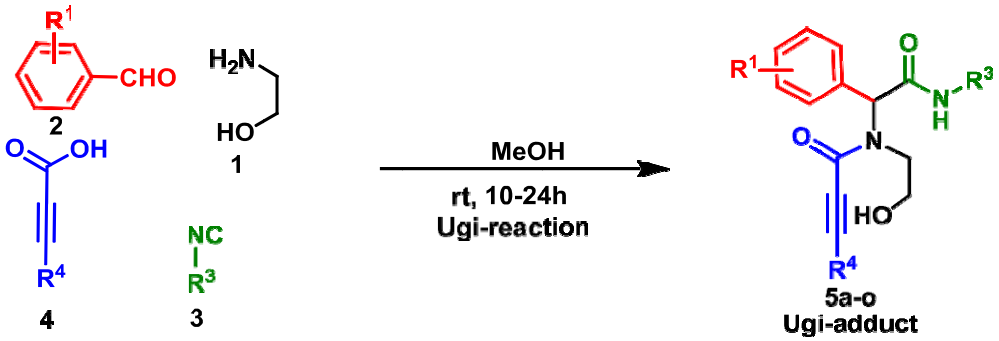
1. EXPERIMENTAL SECTION

General Experimental Information

Unless otherwise specified, all reactions were carried out under air atmosphere in oven-dried round-bottom flasks and the heating reactions were performed in oil bath. All commercially available reagents were purchased from commercial sources and were used without further purification. All reactions were monitored by thin layer chromatography over silica gel-coated TLC plates. Thin-layer chromatography (TLC) was performed on60 F254 silica gel, pre-coated on aluminum plates, and revealed with either a UV lamp(λ_{max} = 254 nm), a specific color reagent (iodine vapors). Silica gel 230-400 mesh was used for column chromatography. ^{1}H and^{13}C NMR spectra were recorded on Bruker AV400 MHz spectrometer. Chemical shifts δ are given in ppm relative to the residual signals of tetramethylsilane in $CDCl_3$ for ^{1}H and ^{13}CNMR. Coupling constants are given in hertz. The HRMS spectra were recorded as ESI-HRMS on Q-TOF masss pectrometer.

Commercially available grades of organic solvents of adequate purity are used in all reactions.

2. General Procedure for the Synthesis of 5a−0.

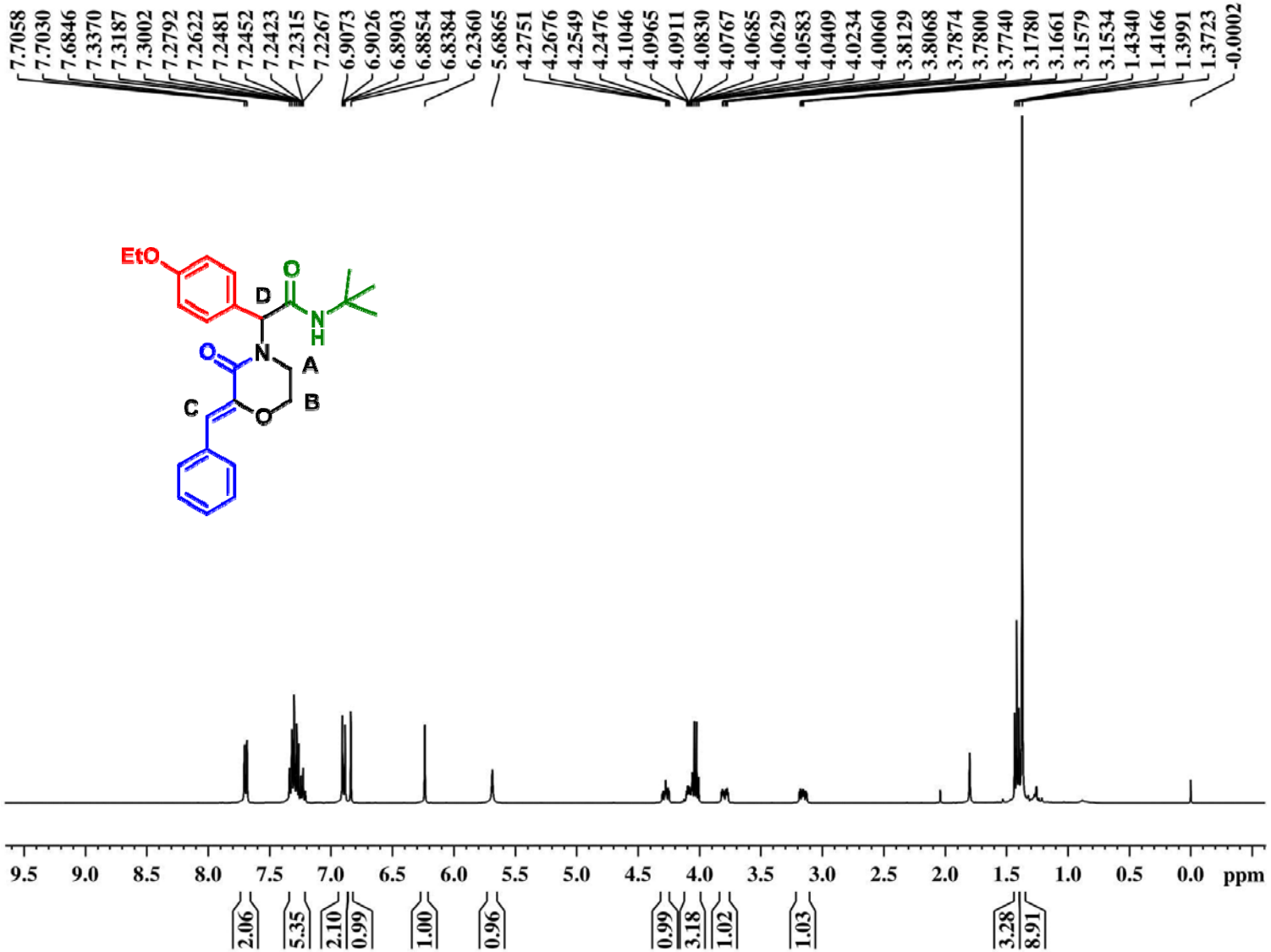


To solution of aromatic aldehyde **2** (1.0 mmol) in methanol (10 ml) were added with 1,2-aminoalcohol **(1**,1.0mmol) and the mixture was stirred at room temperature for 1h. Then, acid **4** (1mmol) was added, and stirring was continued, followed by addition of isocyanides **3** (1mmol). The mixture was stirred for 10-24 hrs at room temperature. Reaction was monitored by TLC (*n*-hexane/EtOAc 2:1). After the completion of the reaction, the reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (eluent:hexane/ EtOAc) to afford Ugi-hydroxypropargyl amides adduct (**5a-o)**.

3. General Procedure for the Synthesis of Compounds 6a−o.

To the stirred solution of Ugi- hydroxypropargylamides adduct **5a-o** (1 mmole) in 5 ml of EtOH was added triphenylphosphine (30%) at room temperature. The reaction mixture was stirred at80 °C for 10 h until the reaction reached completion as evidenced by TLC. After the completion of the reaction, the reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (eluent:hexane/EtOAc) to afford morpholinone products (**6a-o)**.

Spectral data and HRMS of **6a-6o**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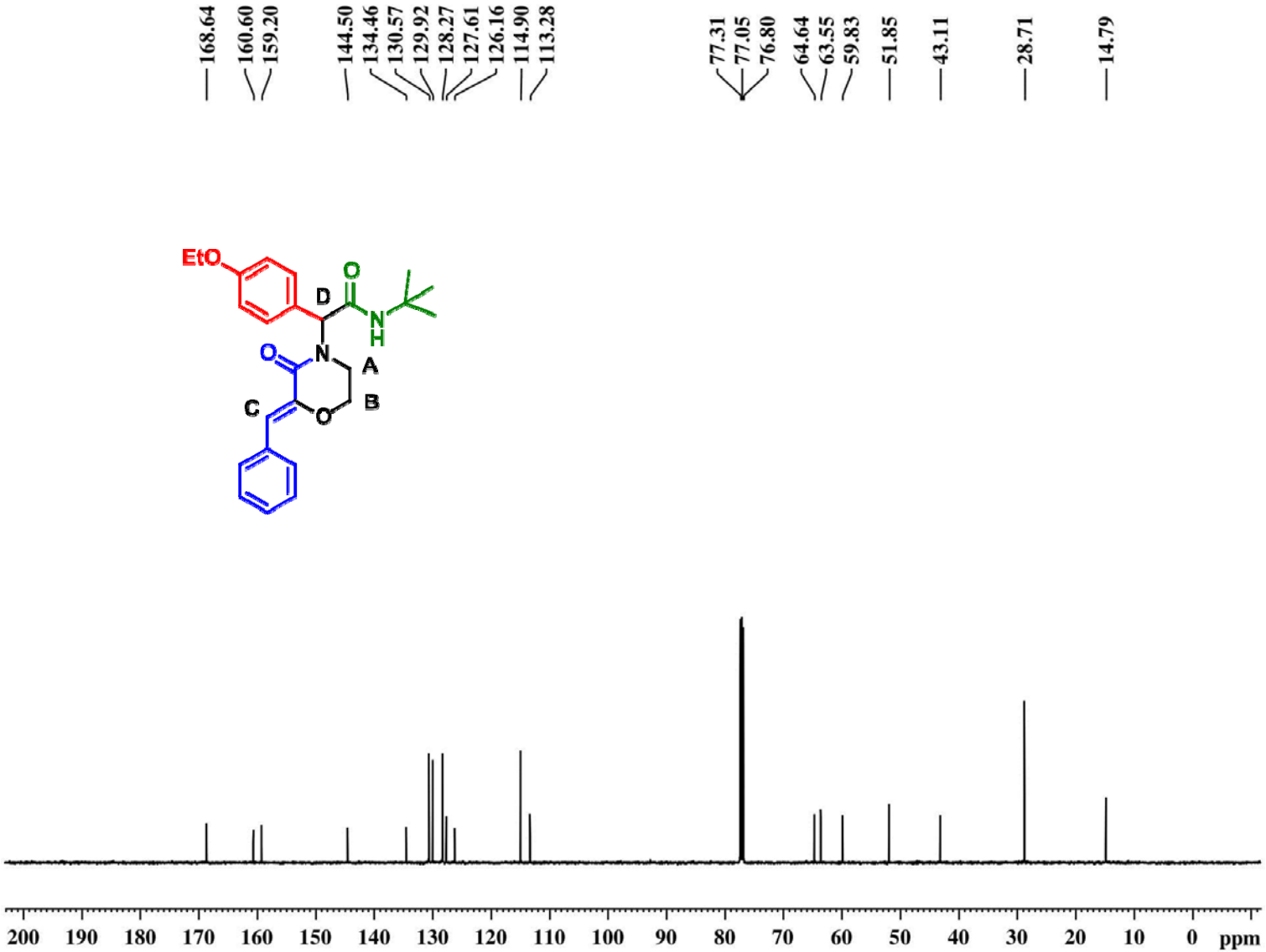
7.6846
7.3370
7.3187
7.3002
7.2792
7.2622
7.2481
7.2452
7.2423
7.2315
7.2267
6.9073
6.9026
6.8903
6.8854
6.8384
6.2360
5.6865
4.2751
4.2676
4.2549
4.2476
4.1046
4.0965
4.0911
4.0830
4.0767
4.0685
4.0629
4.0583
4.0409
4.0234
4.0060
3.8129
3.8068
3.7874
3.7800
3.7740
3.1780
3.1661
3.1579
3.1534
1.4340
1.4166
1.3991
1.3723
-0.0002
EtO
O
D
N
H
O
N
A
C
O
B
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
ppm
2.06
5.35
2.10
0.99
1.00
0.96
0.99
3.18
1.02
1.03
3.28
8.91

^{1}H NMR spectrum of **6a** (400MHz, $CDCl_3$)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159.20
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134.46
130.57
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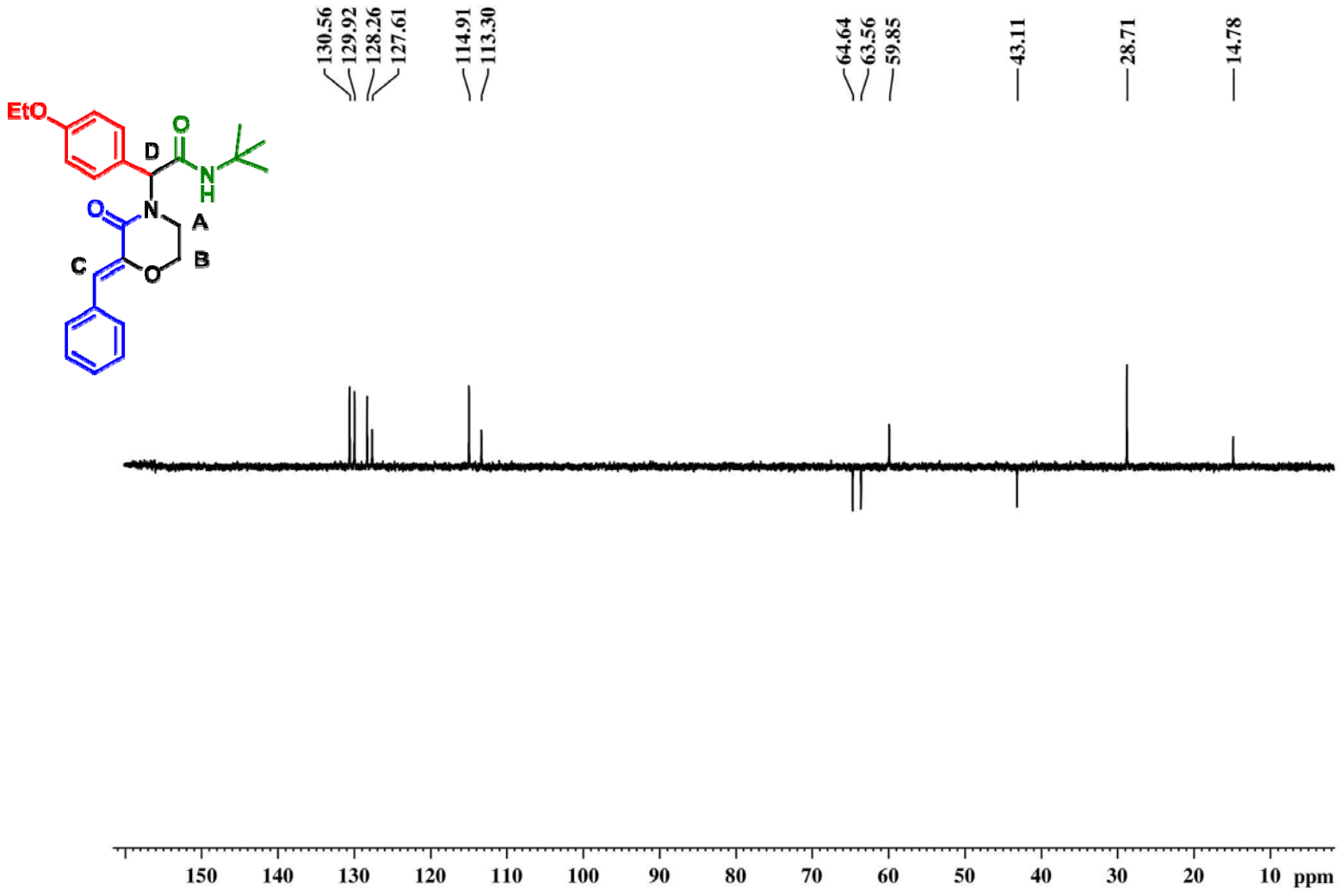
128.27
127.61
126.16
114.90
113.28
77.31
77.05
76.80
64.64
63.55
59.83
51.85
43.11
28.71
14.79
EtO
O
D
N
H
O
N
A
C
O
B
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR spectrum of **6a** (100MHz,MHz, $CDCl_3$)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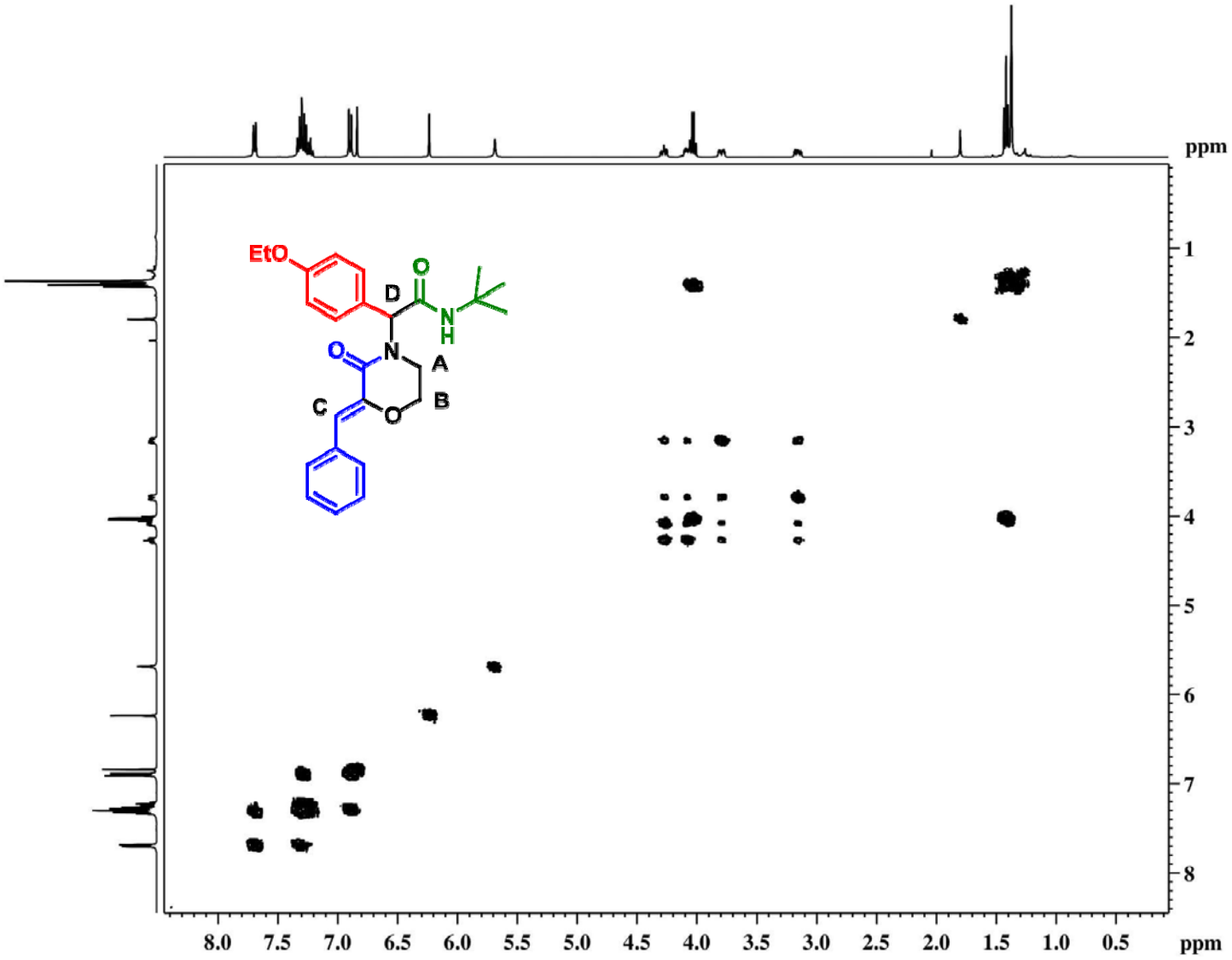
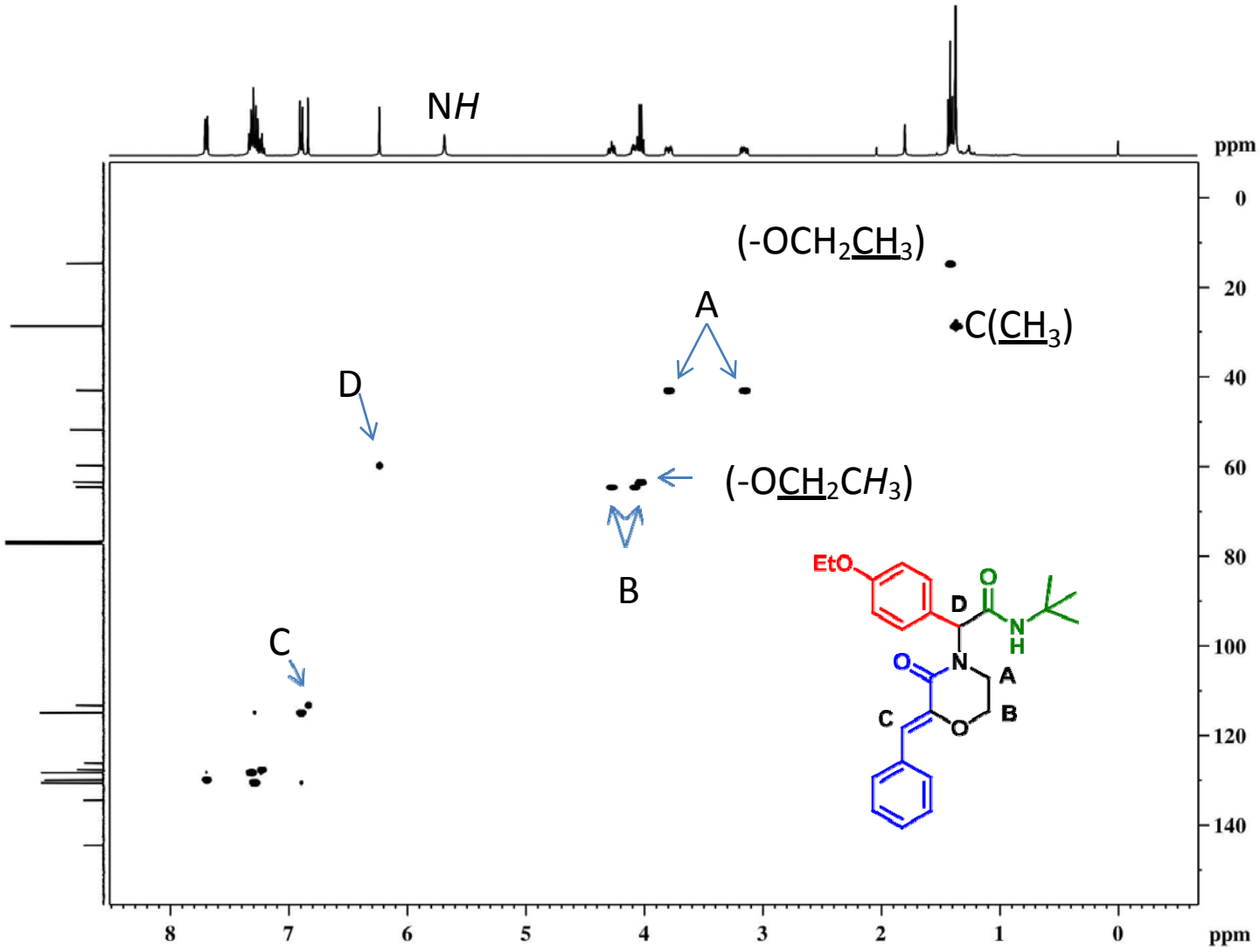
129.92
128.26
127.61
114.91
113.30
64.64
63.56
59.85
43.11
28.71
14.78
EtO
O
D
N
H
O
N
A
C
O
B
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

DEPT-135 spectrum of **6a** (100MHz,MHz,$CDCl_3$)


EtO
O
D
N
H
O
N
A
B
C
O
ppm
1
2
3
4
5
6
7
8
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
ppm

2D-COSY spectrum of **6a** (400MHz,MHz,$CDCl_3$)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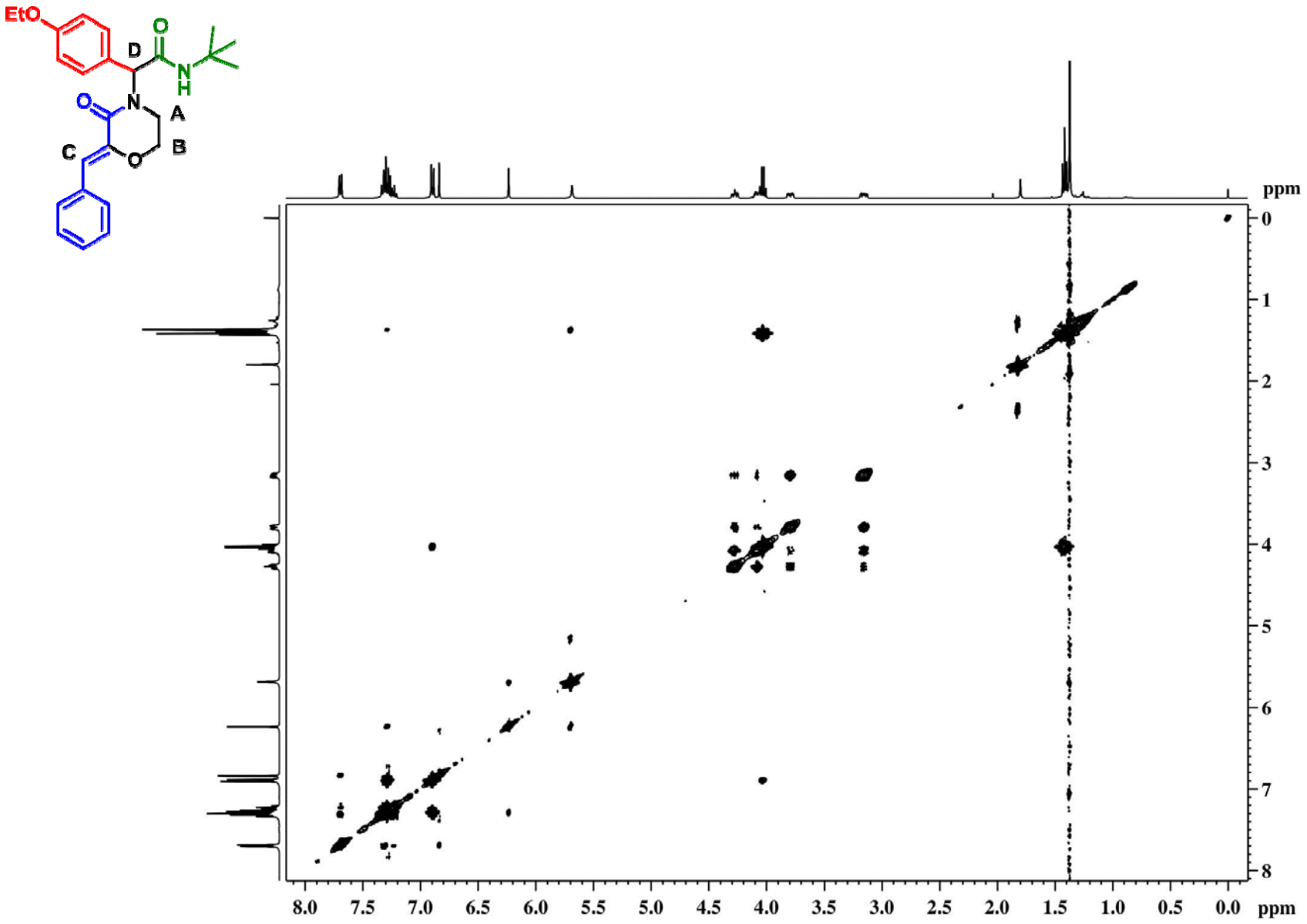
C(CH3)
A
D
(-OCH2CH3)
B
C
EtO
O
D
N
H
N
A
O
C
O
B
ppm
0
20
40
60
80
100
120
140
8
7
6
5
4
3
2
1
0
ppm

2D-HSQC spectrum of **6a** (400 MHz, MHz,$CDCl_3$)


EtO
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D
N
H
O
N
A
C
O
B
ppm
0
1
2
3
4
5
6
7
8
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
ppm

2D-NOESY spectrum of **6a** (400MHz, $CDCl_3$)

SAIF [HRMS Report]

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Vial:	CStk1-01:12	Injection Volume(µl):	1.00

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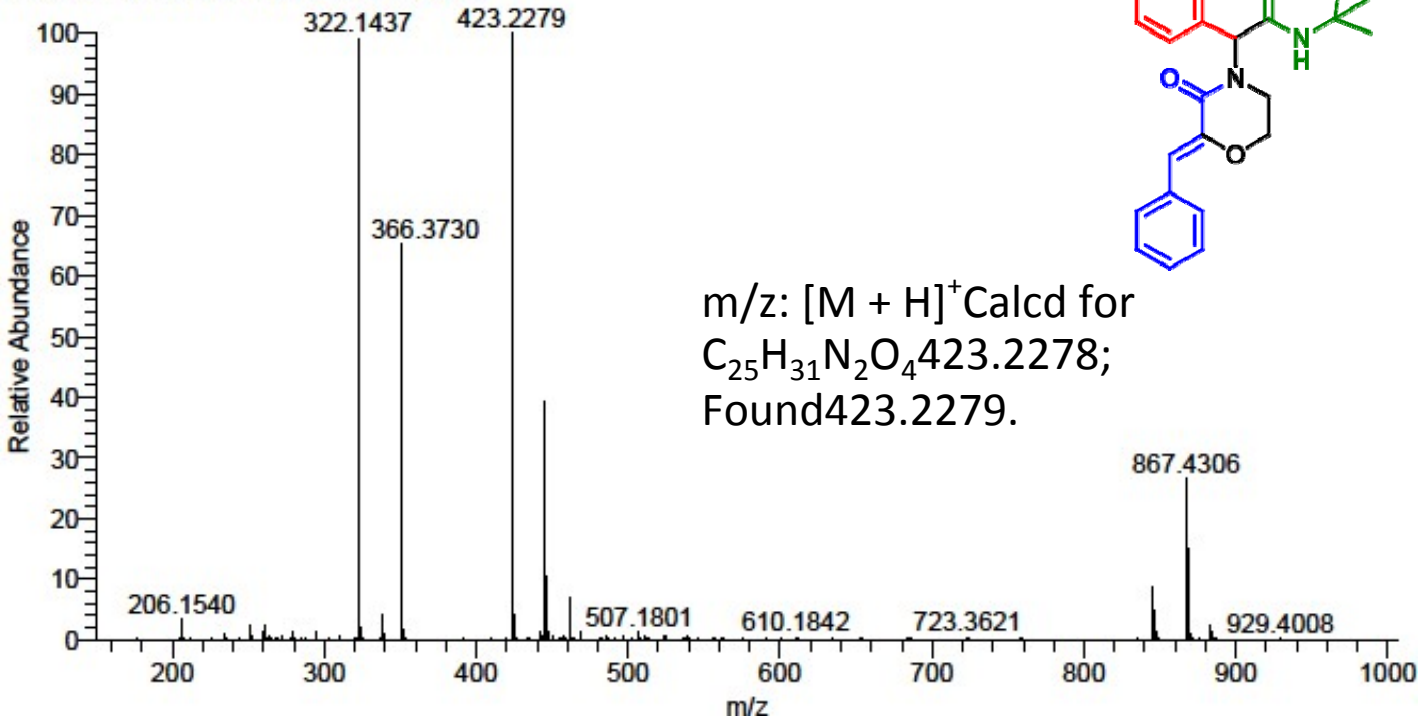
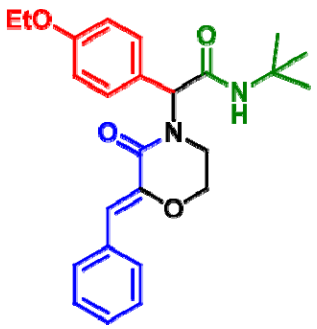
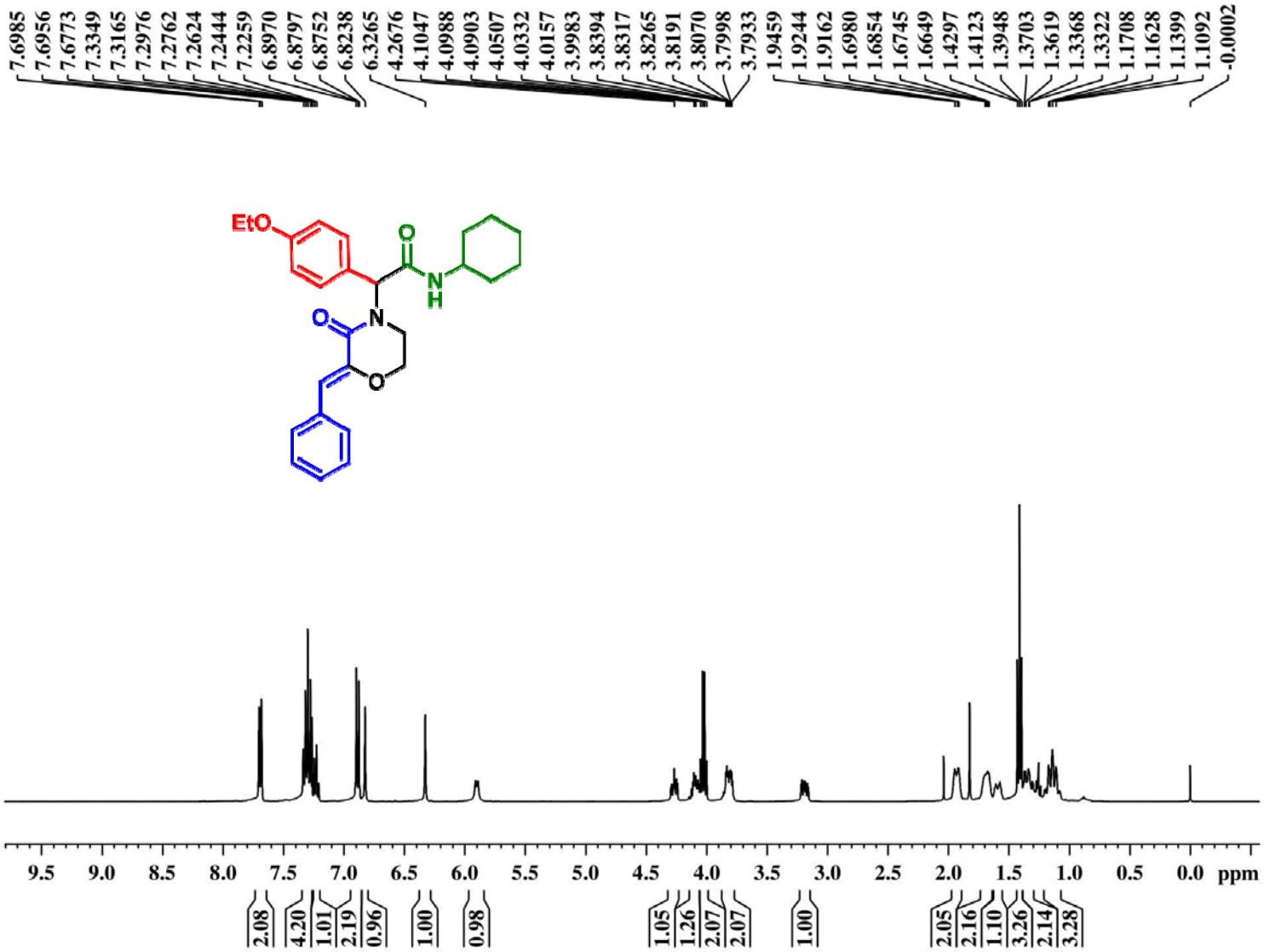




m/z: $[M + H]^+$Calcd for $C_{25}H_{31}N_2O_4$423.2278; Found423.2279.

HRMS of compound **6a**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6.8238
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4.1047
4.0988
4.0903
4.0507
4.0332
4.0157
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3.8317
3.8265
3.8191
3.8070
3.7998
3.7933
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1.9244
1.9162
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1.6854
1.6745
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1.4297
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1.3703
1.3619
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1.1628
1.1399
1.1092
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N
H
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N
O
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8.5
8.0
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7.0
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6.0
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2.0
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0.5
0.0
ppm
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4.20
1.01
2.19
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1.00
0.98
1.05
1.26
2.07
2.07
1.00
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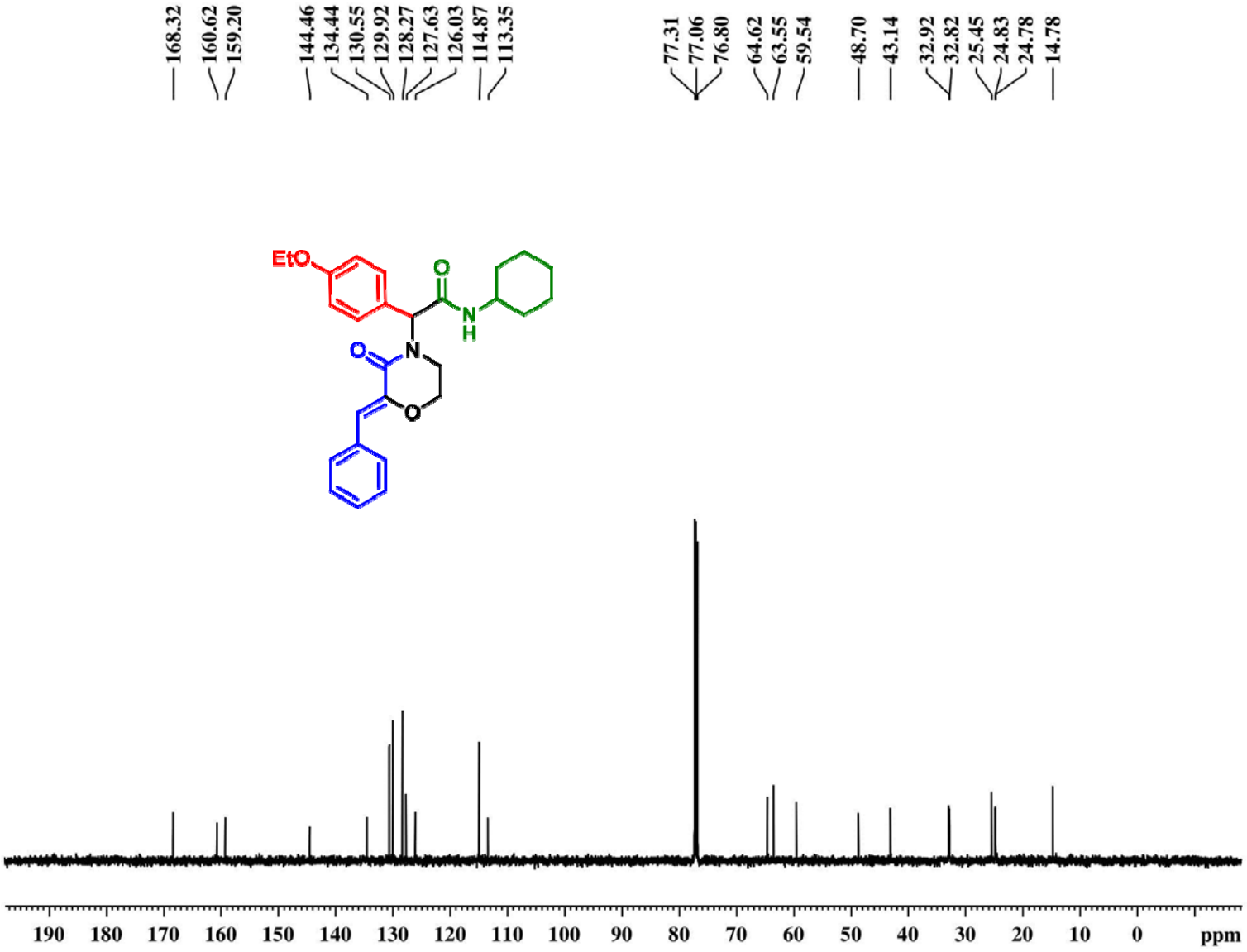
3.28

^{1}H NMR spectrum of **6b** (400MHz, $CDCl_3$)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159.20
144.46
134.44
130.55
129.92
128.27
127.63
126.03
114.87
113.35
77.31
77.06
76.80
64.62
63.55
59.54
48.70
43.14
32.92
32.82
25.45
24.83
24.78
14.78
EtO
O
N
H
O
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

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

SAIF [HRMS Report]

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Vial:	CStk1-01:9	Injection Volume(µl):	1.00

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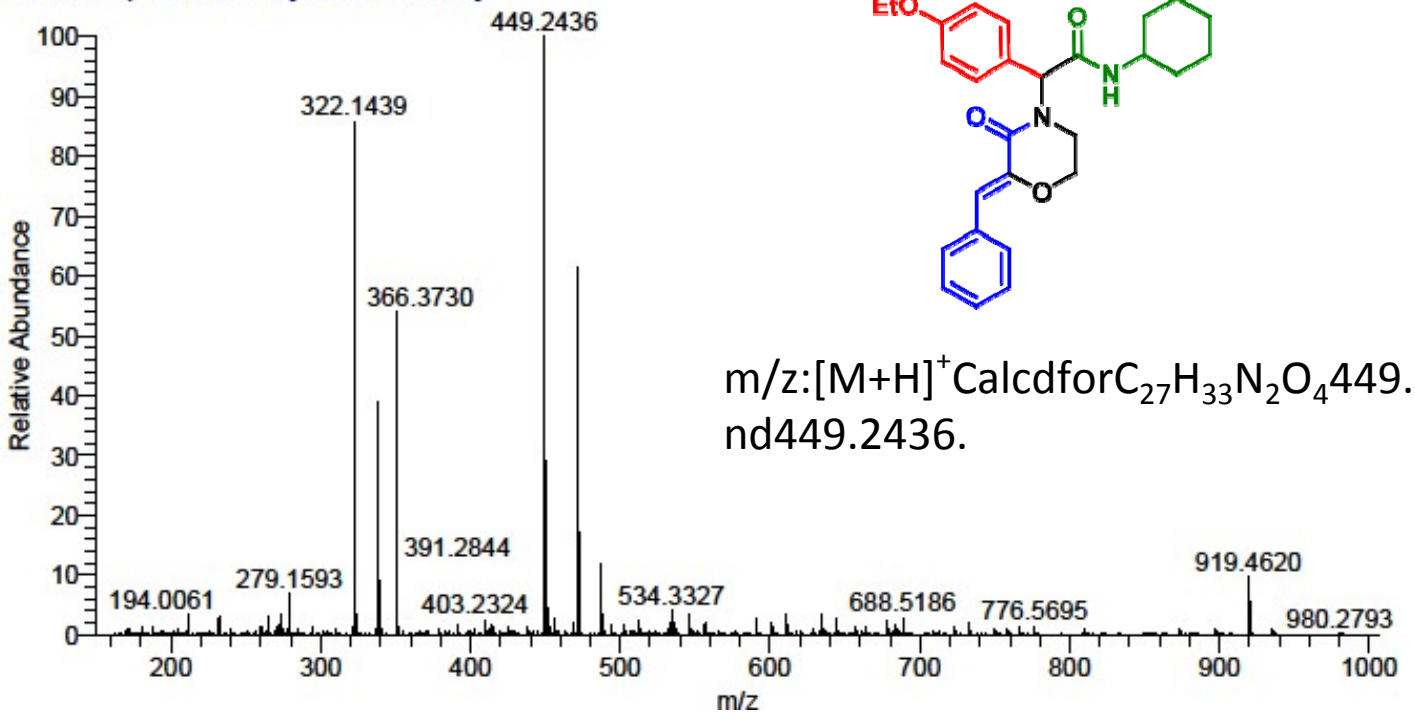
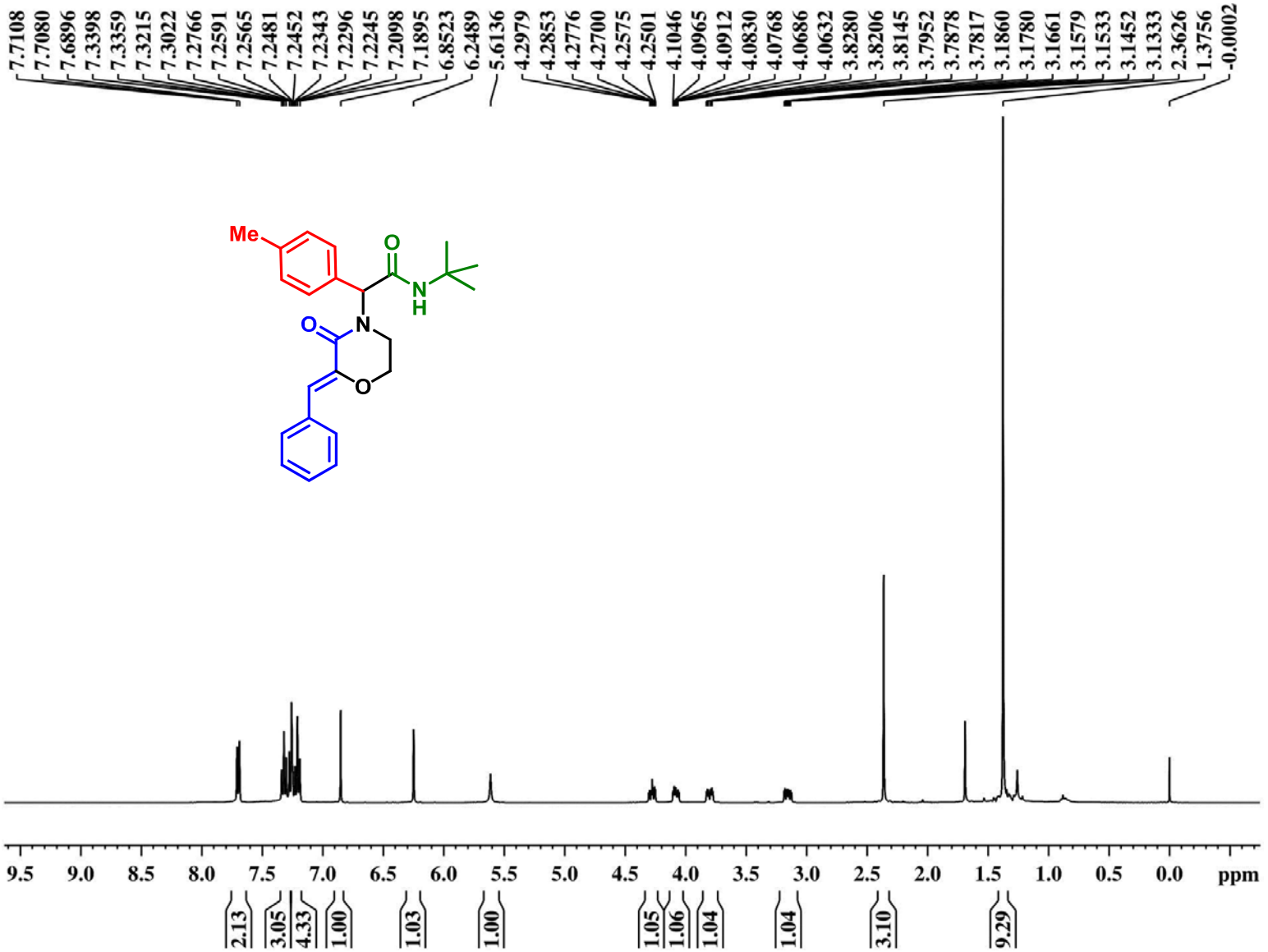


m/z:$[M+H]^+$CalcdforC$_{27}$H$_{33}$N$_2$O$_4$449.2435;Found449.2436.

HRMS of compound **6b**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7.2452
7.2343
7.2296
7.2245
7.2098
7.1895
6.8523
6.2489
5.6136
4.2979
4.2853
4.2776
4.2700
4.2575
4.2501
4.1046
4.0965
4.0912
4.0830
4.0768
4.0686
4.0632
3.8280
3.8206
3.8145
3.7952
3.7878
3.7817
3.1860
3.1780
3.1661
3.1579
3.1533
3.1452
3.1333
2.3626
1.3756
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Me
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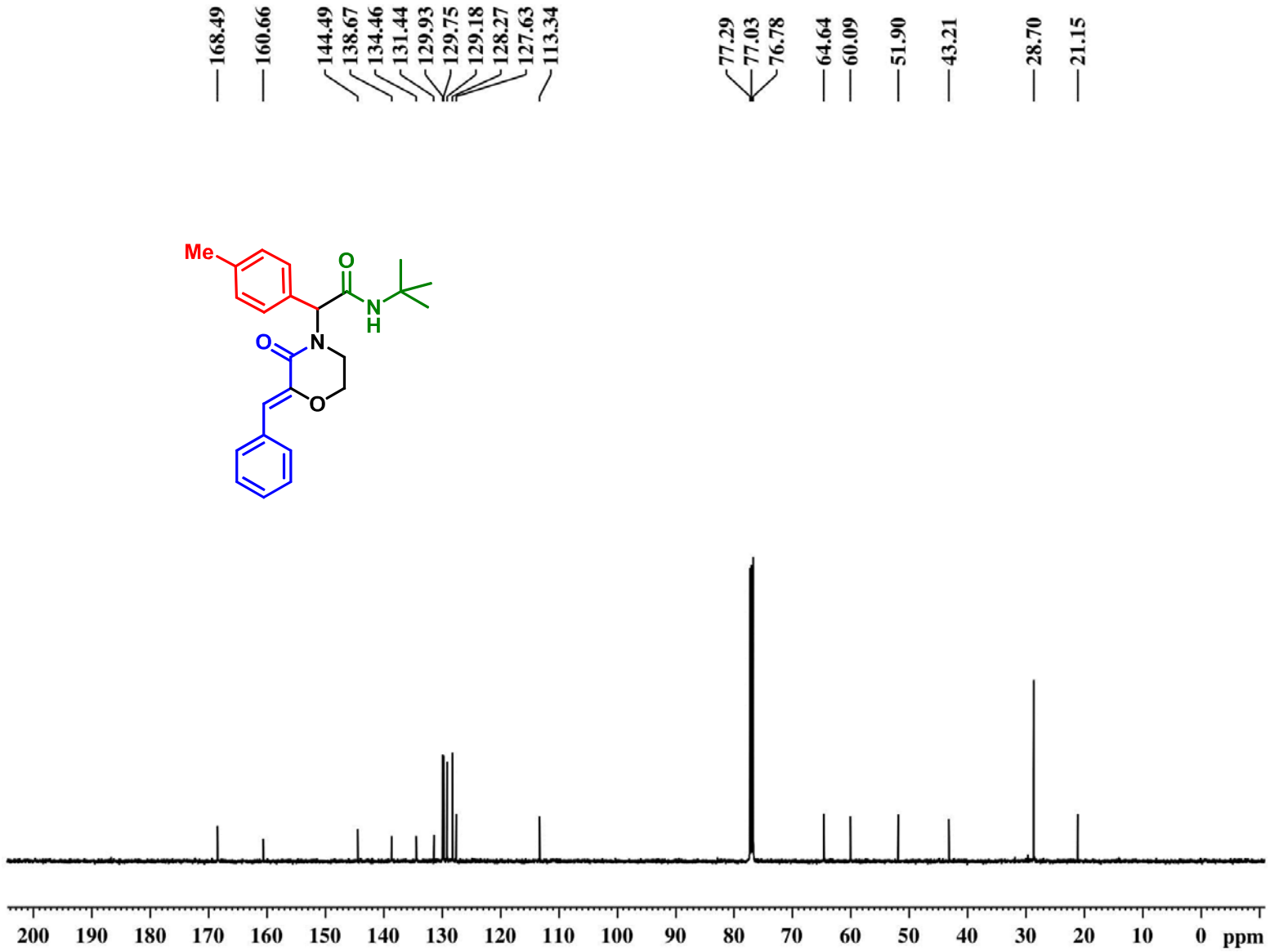
N
H
O
N
O
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.13
3.05
4.33
1.00
1.03
1.00
1.05
1.06
1.04
1.04
3.10
9.29

^{1}H NMR spectrum of **6c** (400MHz, $CDCl_3$)


168.49
160.66
144.49
138.67
134.46
131.44
129.93
129.75
129.18
128.27
127.63
113.34
77.29
77.03
76.78
64.64
60.09
51.90
43.21
28.70
21.15
Me
O
N
H
O
N
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR spectrum of **6c** (100 MHz, MHz, $CDCl_3$)

SAIF [HRMS Report]

Data File:	HRMS21I17MAR39	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
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Vial:	CStk1-01:39	Injection Volume(µl):	1.00

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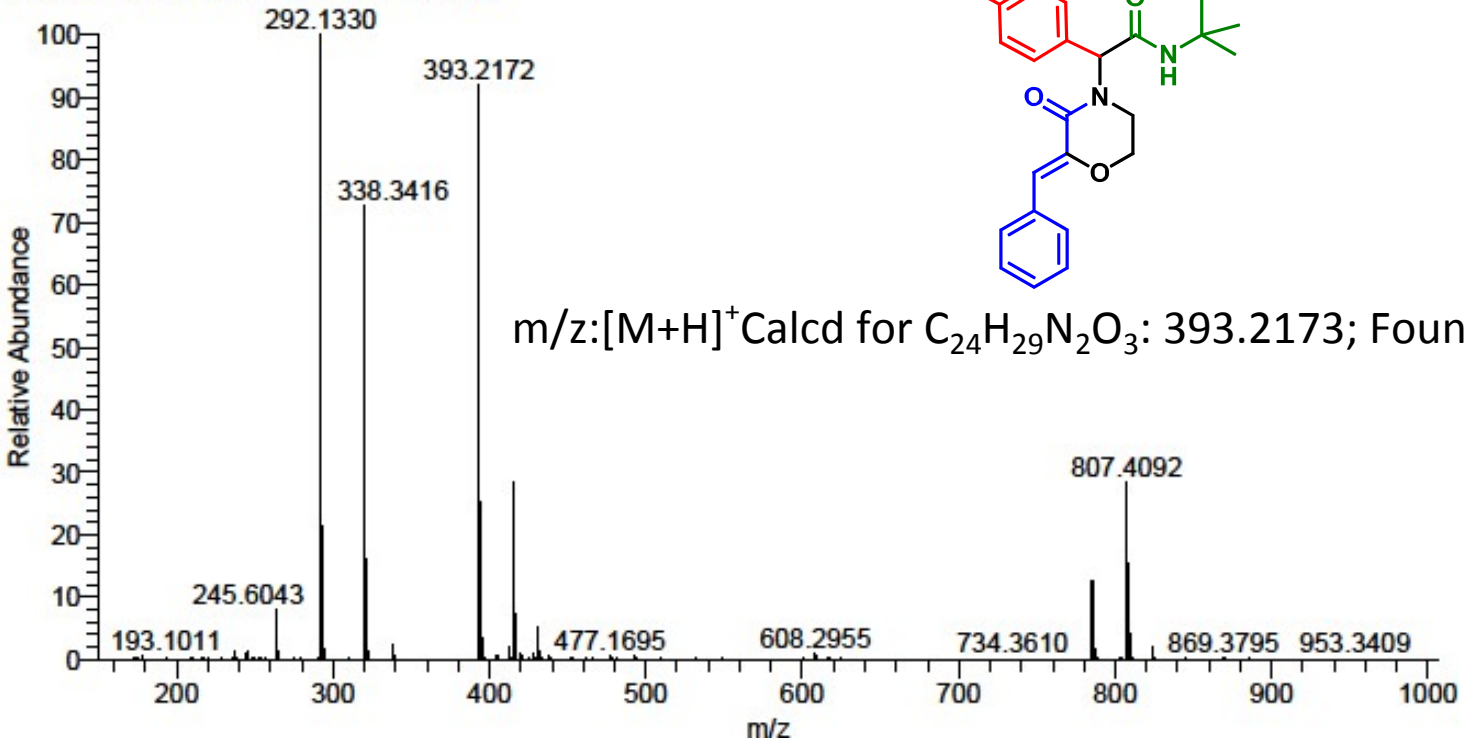
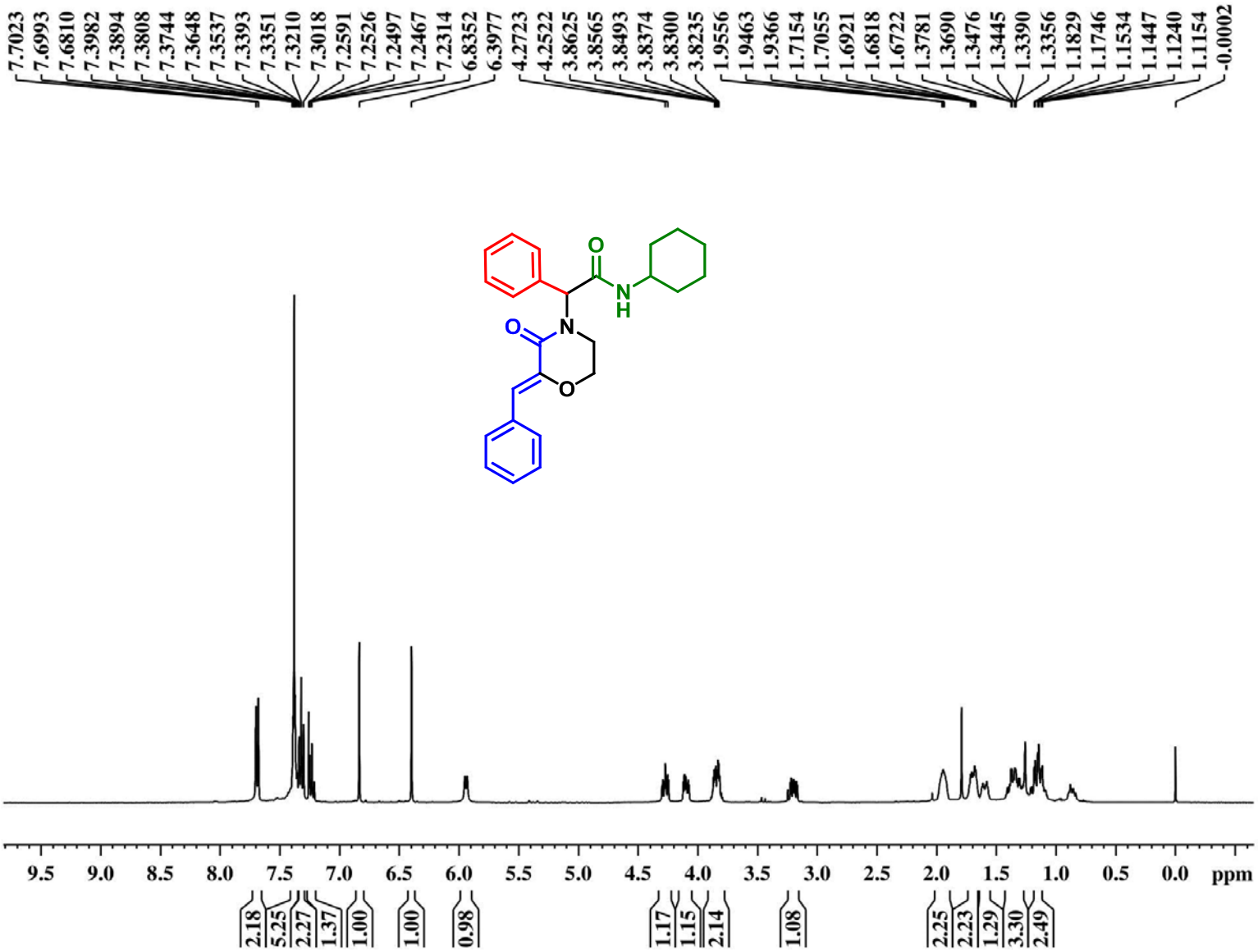


m/z:$[M+H]^+$Calcd for $C_{24}H_{29}N_2O_3$: 393.2173; Found: 393.2172.

HRMS of compound **6c**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7.6810
7.3982
7.3894
7.3808
7.3744
7.3648
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7.3351
7.3210
7.3018
7.2591
7.2526
7.2497
7.2467
7.2314
6.8352
6.3977
4.2723
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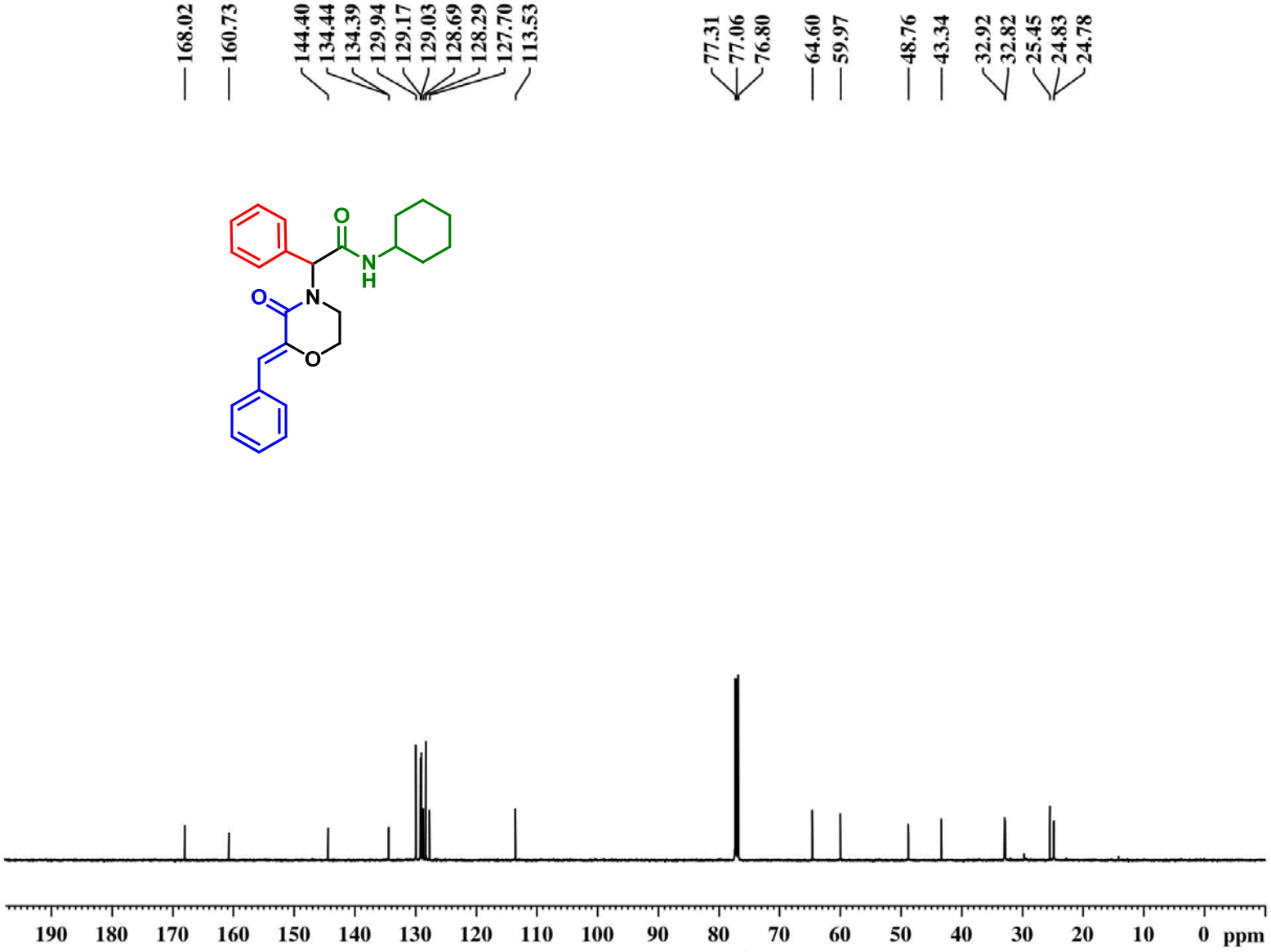
3.8625
3.8565
3.8493
3.8374
3.8300
3.8235
1.9556
1.9463
1.9366
1.7154
1.7055
1.6921
1.6818
1.6722
1.3781
1.3690
1.3476
1.3445
1.3390
1.3356
1.1829
1.1746
1.1534
1.1447
1.1240
1.1154
-0.0002
O
N
H
O
N
O
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.18
5.25
2.27
1.37
1.00
1.00
0.98
1.17
1.15
2.14
1.08
2.25
2.23
1.29
3.30
2.49

^{1}H NMR spectrum of **6d** (400MHz,$CDCl_3$)


168.02
160.73
144.40
134.44
134.39
129.94
129.17
129.03
128.69
128.29
127.70
113.53
77.31
77.06
76.80
64.60
59.97
48.76
43.34
32.92
32.82
25.45
24.83
24.78
O
N
H
O
N
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR spectrum of **6d** (100 MHz, MHz, $CDCl_3$)

SAIF [HRMS Report]

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Vial:	CStk1-01:38	Injection Volume(µl):	1.00

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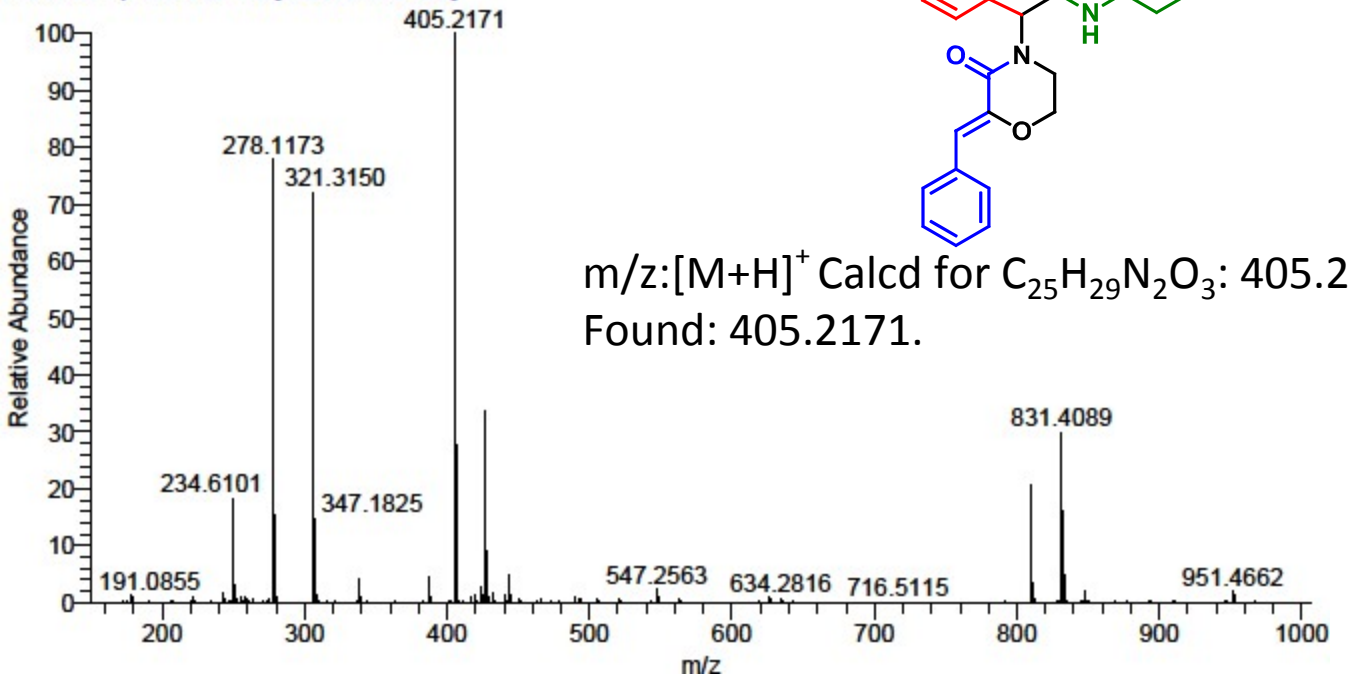
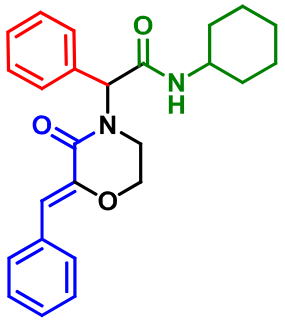
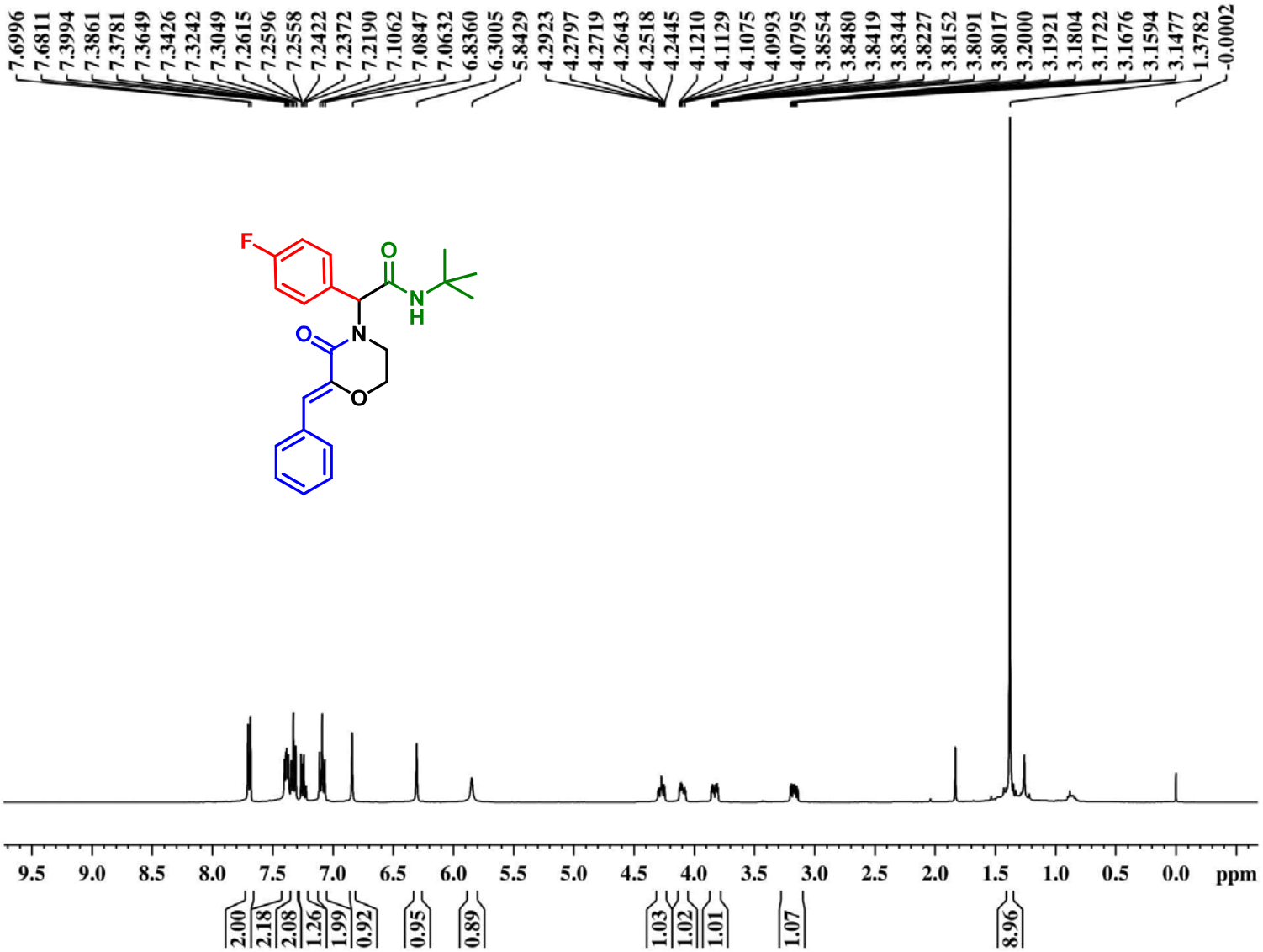




m/z:$[M+H]^+$ Calcd for $C_{25}H_{29}N_2O_3$: 405.2173; Found: 405.2171.

HRMS of compound **6d**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7.2558
7.2422
7.2372
7.2190
7.1062
7.0847
7.0632
6.8360
6.3005
5.8429
4.2923
4.2797
4.2719
4.2643
4.2518
4.2445
4.1210
4.1129
4.1075
4.0993
4.0795
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3.8480
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3.8091
3.8017
3.2000
3.1921
3.1804
3.1722
3.1676
3.1594
3.1477
1.3782
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F
O
N
H
O
N
O
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
ppm
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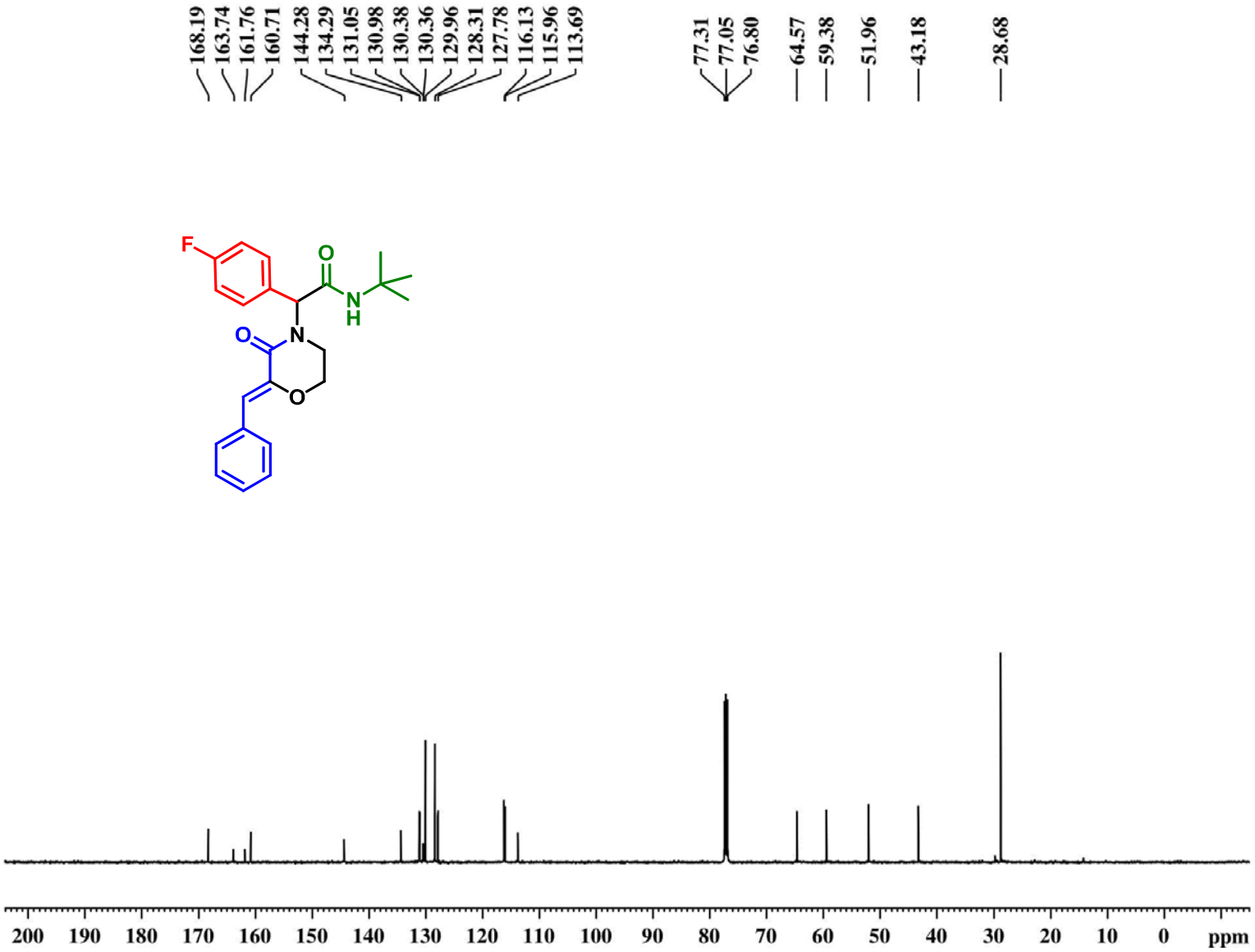
2.08
1.26
1.99
0.92
0.95
0.89
1.03
1.02
1.01
1.07
8.96

^{1}H NMR spectrum of **6e** (400MHz,$CDCl_3$)

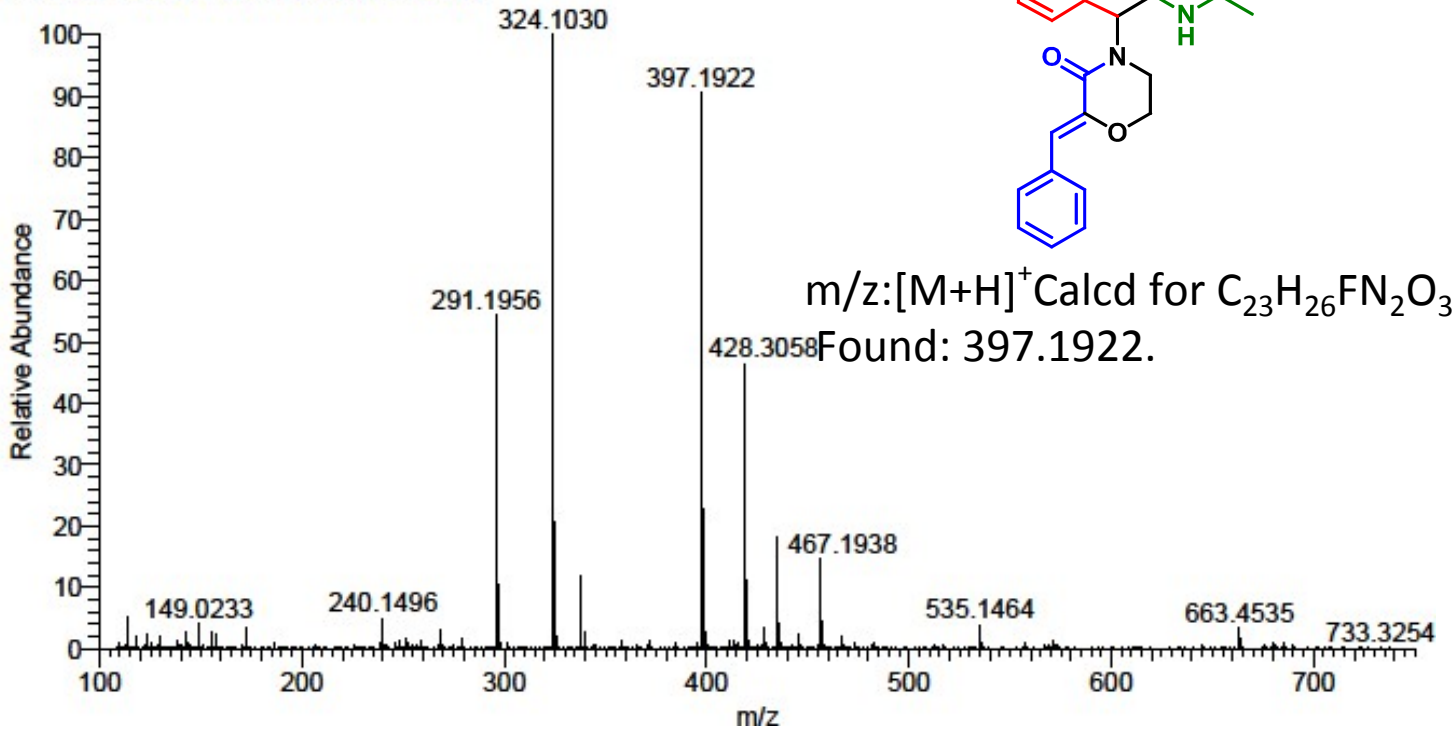

168.19
163.74
161.76
160.71
144.28
134.29
131.05
130.98
130.38
130.36
129.96
128.31
127.78
116.13
115.96
113.69
77.31
77.05
76.80
64.57
59.38
51.96
43.18
28.68
F
O
N
H
O
N
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

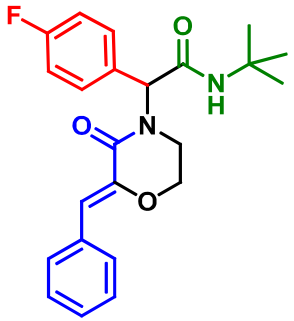
^{13}C NMR spectrum of **6e** (100MHz, $CDCl_3$)

SAIF [HRMS Report]

Data File:	HRMS21I17MAR37	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-10R	Sample Name:	
Acquisition Date:	03/17/21 12:15:34 PM	Run Time(min):	0.00
Vial:	CStk1-01:37	Injection Volume(µl):	1.00

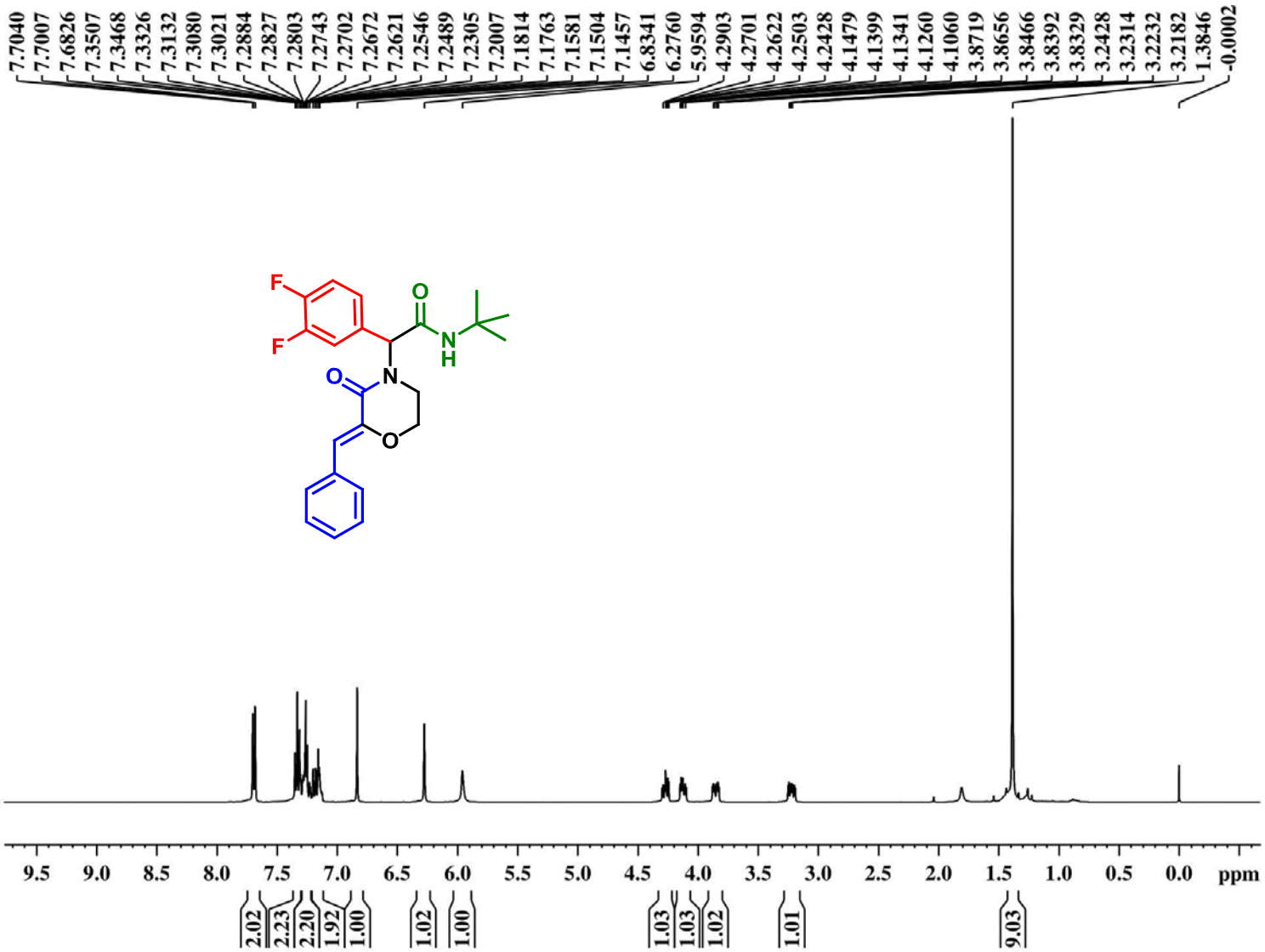
HRMS21I17MAR37 #32-65 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 1.89E6
T: FTMS + c ESI Full ms [100.00-750.00]



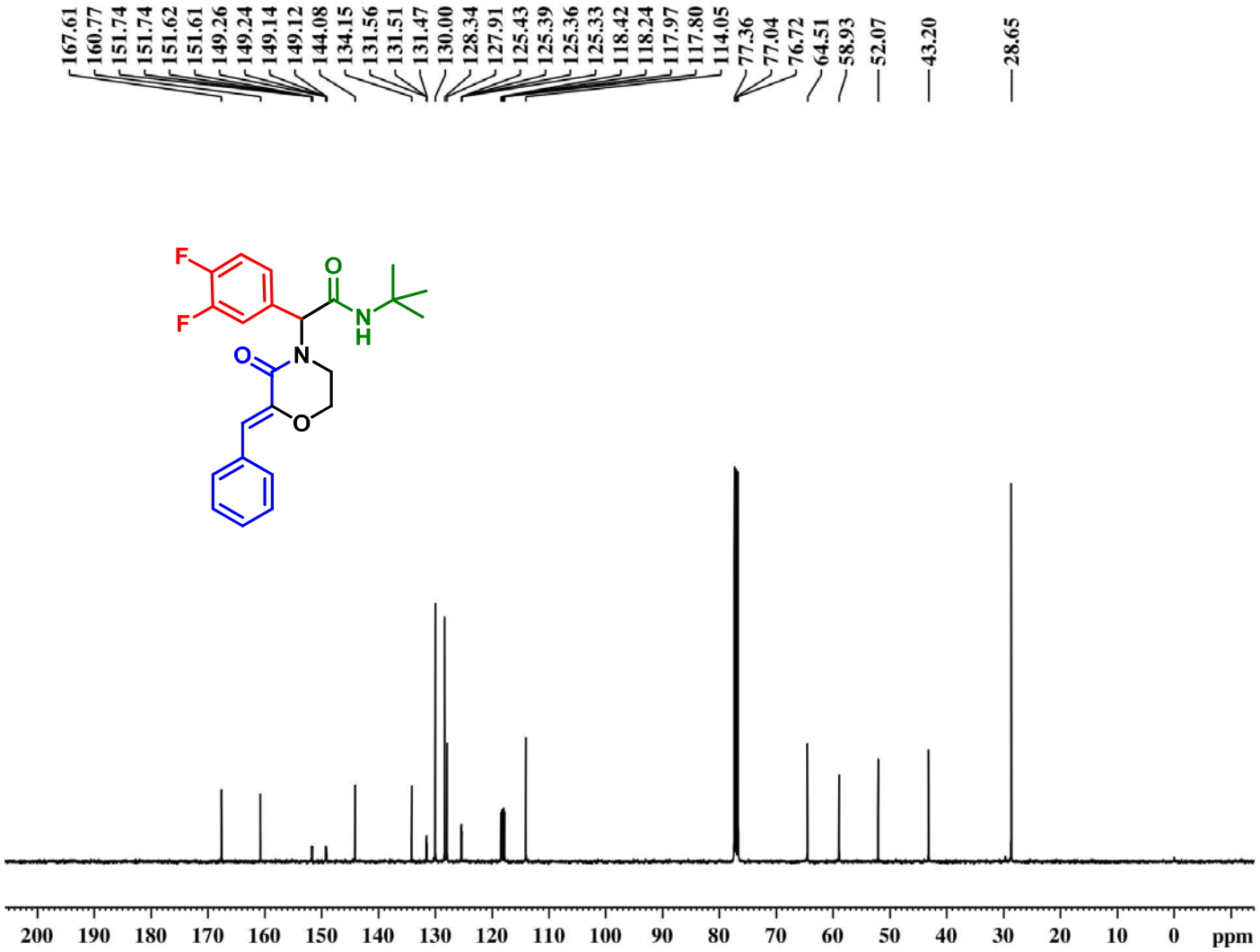


m/z:$[M+H]^+$Calcd for $C_{23}H_{26}FN_2O_3$: 397.1922; Found: 397.1922.

HRMS of compound **6e**


7.7040
7.7007
7.6826
7.3507
7.3468
7.3326
7.3132
7.3080
7.3021
7.2884
7.2827
7.2803
7.2743
7.2702
7.2672
7.2621
7.2546
7.2489
7.2305
7.2007
7.1814
7.1763
7.1581
7.1504
7.1457
6.8341
6.2760
5.9594
4.2903
4.2701
4.2622
4.2503
4.2428
4.1479
4.1399
4.1341
4.1260
4.1060
3.8719
3.8656
3.8466
3.8392
3.8329
3.2428
3.2314
3.2232
3.2182
1.3846
-0.0002
F
F
O
N
H
O
N
O
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
ppm
2.02
2.23
2.20
1.92
1.00
1.02
1.00
1.03
1.03
1.02
1.01
9.03

^{1}H NMR spectrum of **6f** (400MHz, $CDCl_3$)

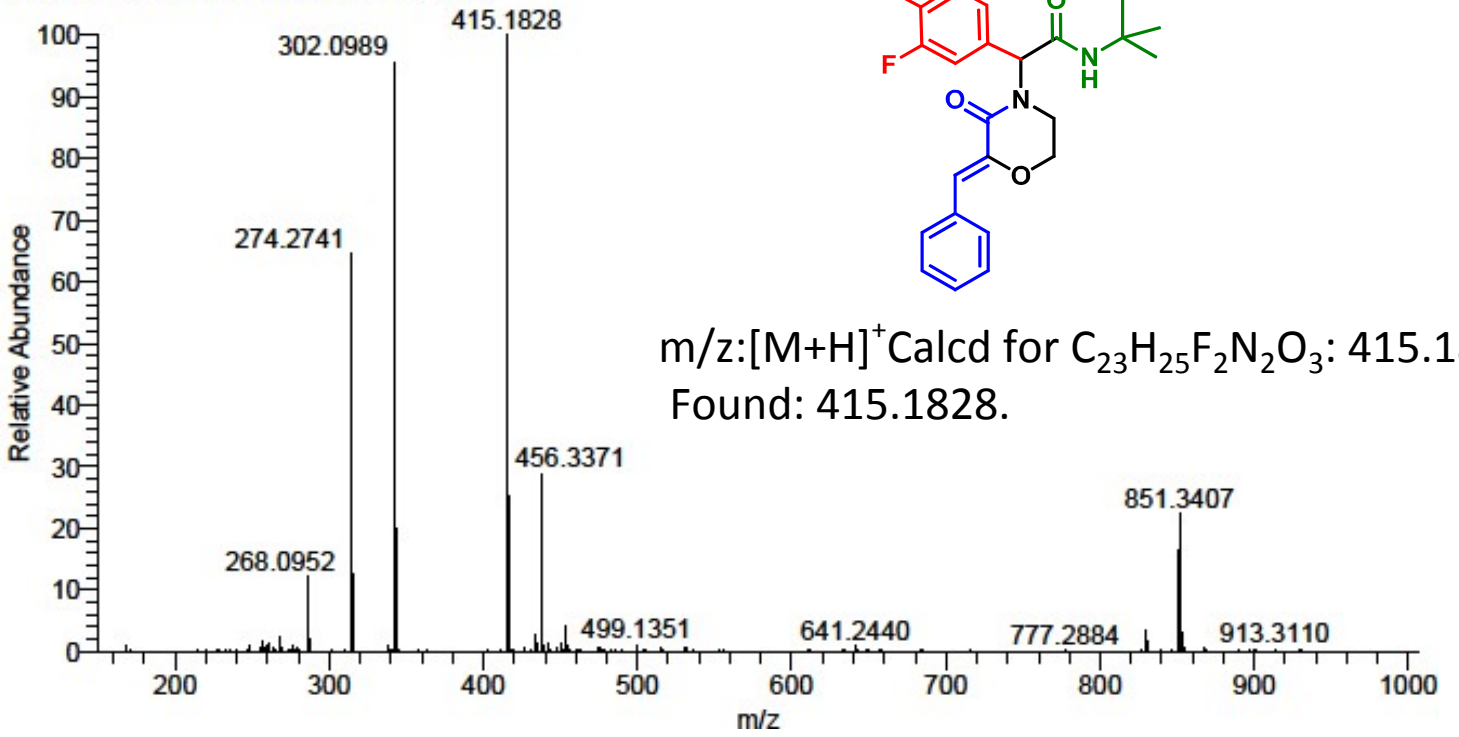

167.61
160.77
151.74
151.74
151.62
151.61
149.26
149.24
149.14
149.12
144.08
134.15
131.56
131.51
131.47
130.00
128.34
127.91
125.43
125.39
125.36
125.33
118.42
118.24
117.97
117.80
114.05
77.36
77.04
76.72
64.51
58.93
52.07
43.20
28.65
F
F
O
N
H
O
N
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

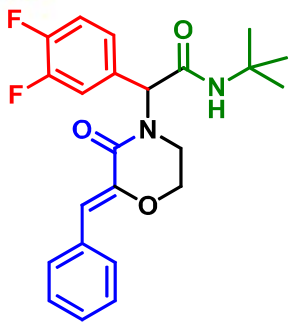
^{13}C NMR spectrum of **6f** (100MHz,MHz, $CDCl_3$)

SAIF [HRMS Report]

Data File:	HRMS21I19MAR10	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-21R	Sample Name:	
Acquisition Date:	03/19/21 11:20:10 AM	Run Time(min):	0.00
Vial:	CStk1-01:10	Injection Volume(ul):	1.00

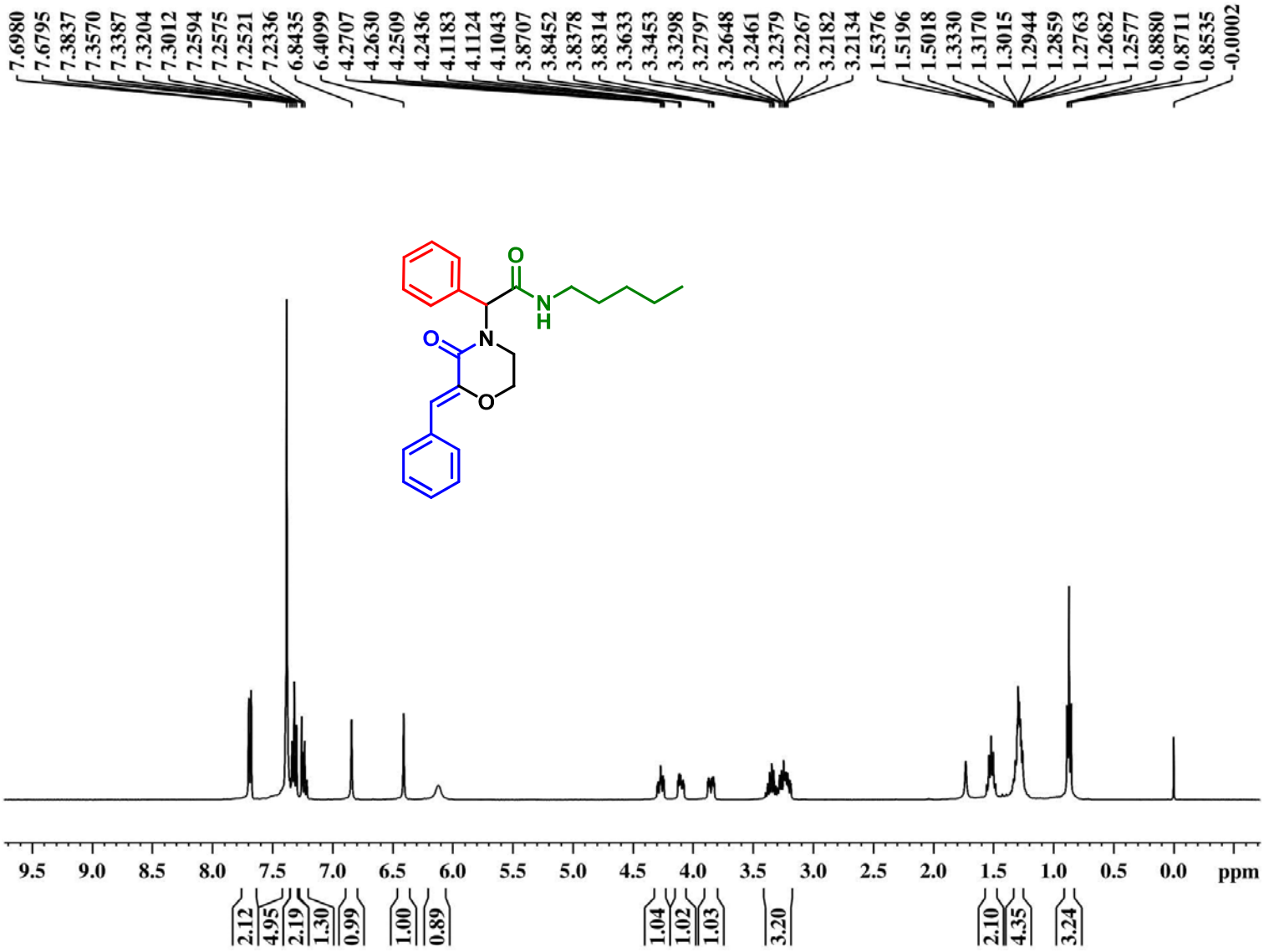
HRMS21I19MAR10 #32-66 RT: 0.25-0.50 AV: 35 SB: 1 0.00 NL: 7.01E6
T: FTMS + p ESI Full ms [150.00-1000.00]



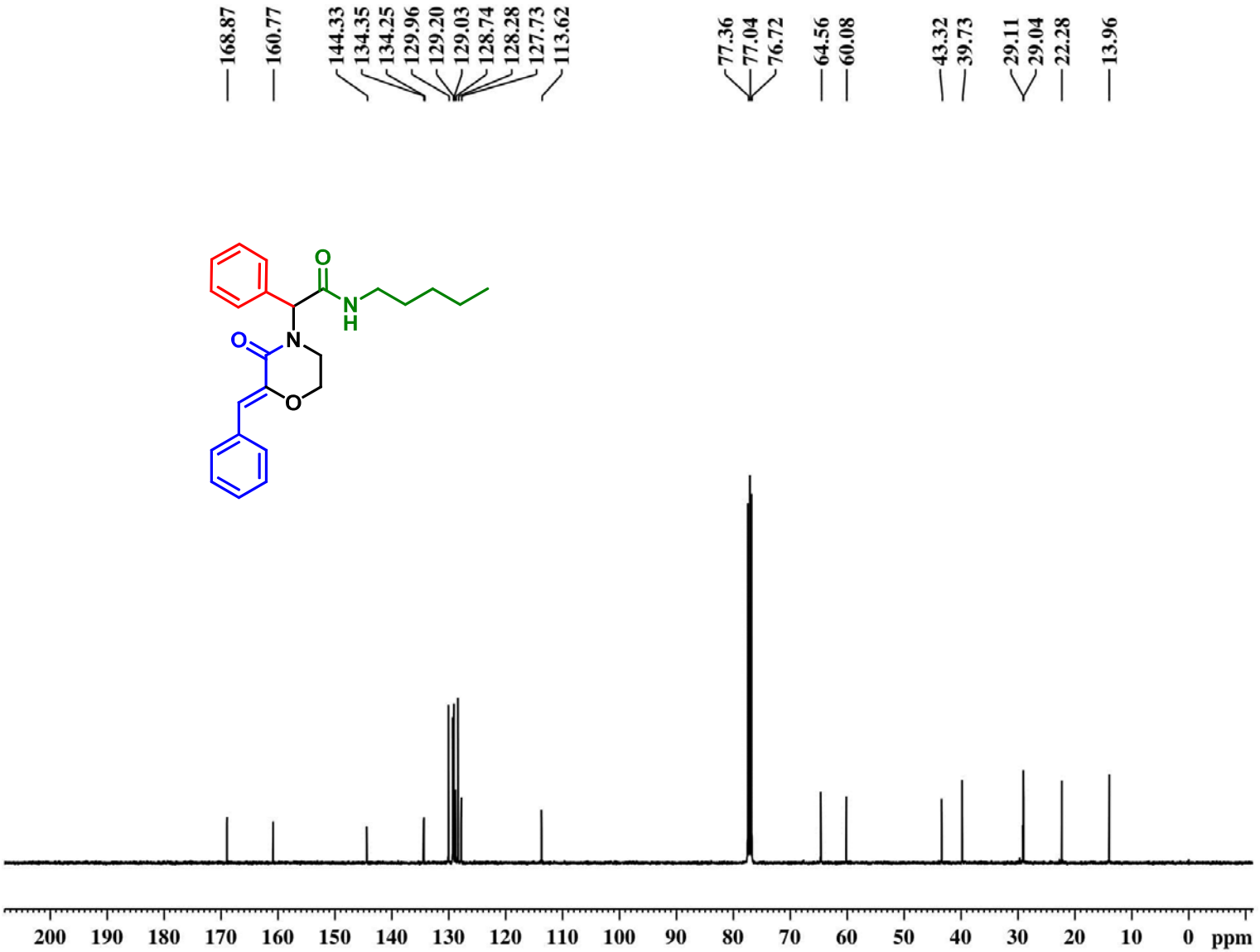


m/z:$[M+H]^+$Calcd for $C_{23}H_{25}F_2N_2O_3$: 415.1828;
Found: 415.1828.

HRMS of compound **6f**


7.6980
7.6795
7.3837
7.3570
7.3387
7.3204
7.3012
7.2594
7.2575
7.2521
7.2336
6.8435
6.4099
4.2707
4.2630
4.2509
4.2436
4.1183
4.1124
4.1043
3.8707
3.8452
3.8378
3.8314
3.3633
3.3453
3.3298
3.2797
3.2648
3.2461
3.2379
3.2267
3.2182
3.2134
1.5376
1.5196
1.5018
1.3330
1.3170
1.3015
1.2944
1.2859
1.2763
1.2682
1.2577
0.8880
0.8711
0.8535
-0.0002
O
N
H
O
N
O
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
ppm
2.12
4.95
2.19
1.30
0.99
1.00
0.89
1.04
1.02
1.03
3.20
2.10
4.35
3.24

^{1}H NMR spectrum of **6g** (400MHz, $CDCl_3$)

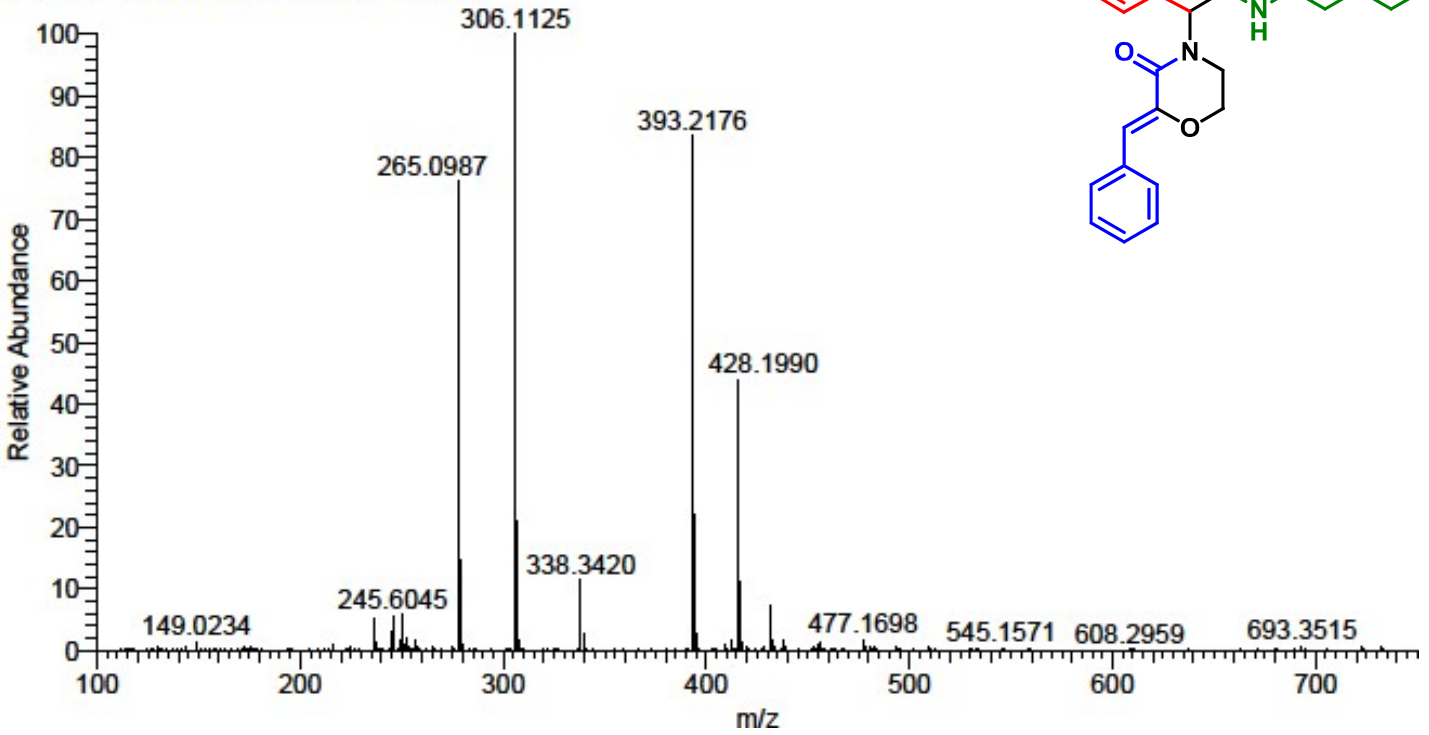

168.87
160.77
144.33
134.35
134.25
129.96
129.20
129.03
128.74
128.28
127.73
113.62
77.36
77.04
76.72
64.56
60.08
43.32
39.73
29.11
29.04
22.28
13.96
O
N
H
O
N
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

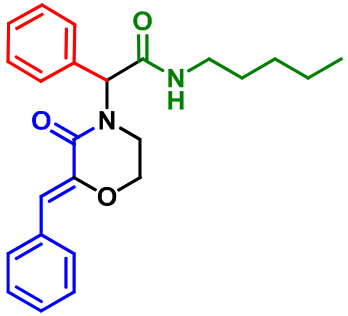
^{13}C NMR spectrum of **6g** (100MHz,MHz, $CDCl_3$)

SAIF [HRMS Report]

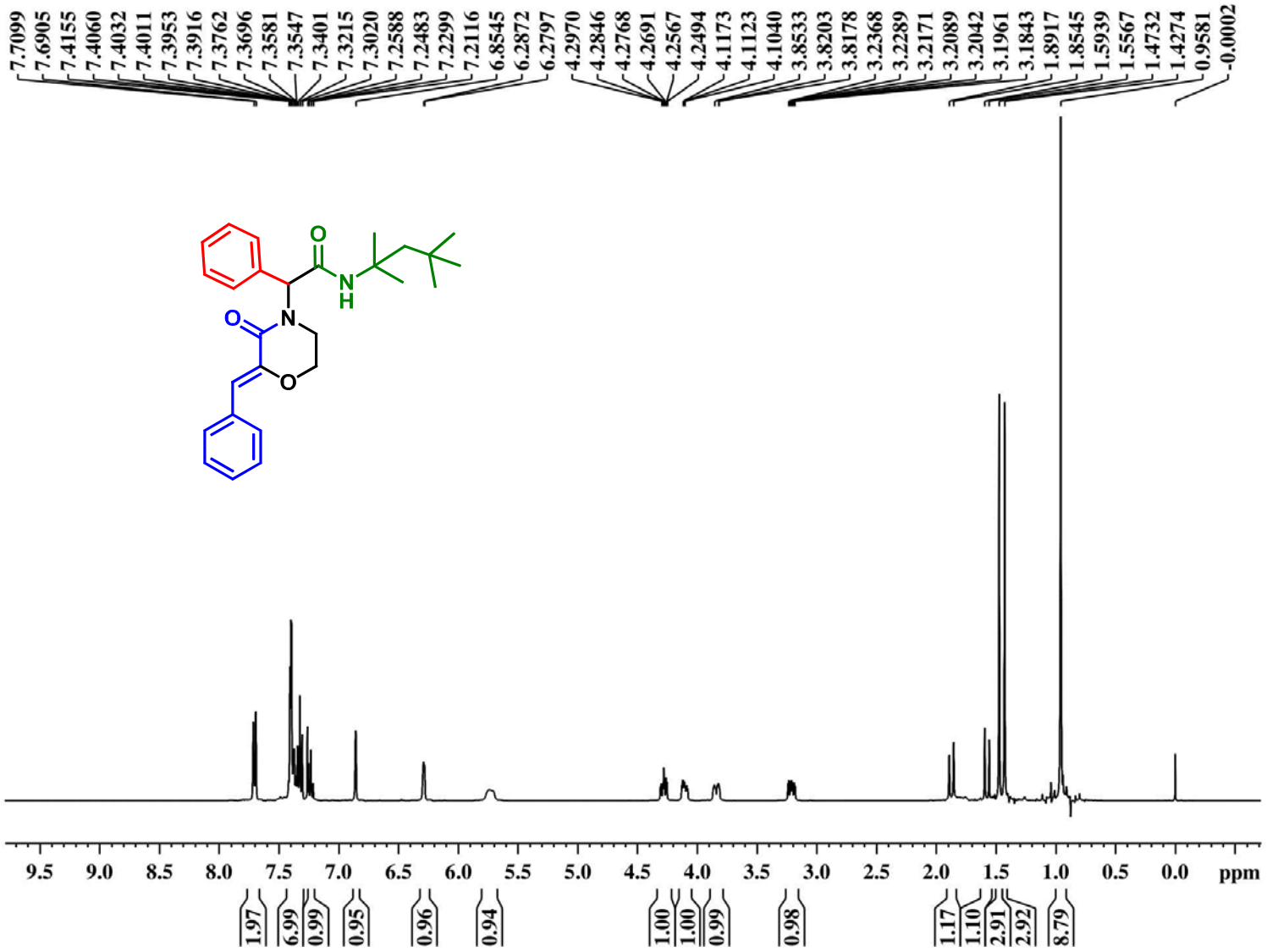
Data File:	HRMS21I22MAR11	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-20	Sample Name:	
Acquisition Date:	03/22/21 01:03:22 PM	Run Time(min):	0.00
Vial:	CStk1-01:11	Injection Volume(µl):	1.00

HRMS21I22MAR11 #33-66 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 1.55E6
T: FTMS + c ESI Full ms [100.00-750.00]

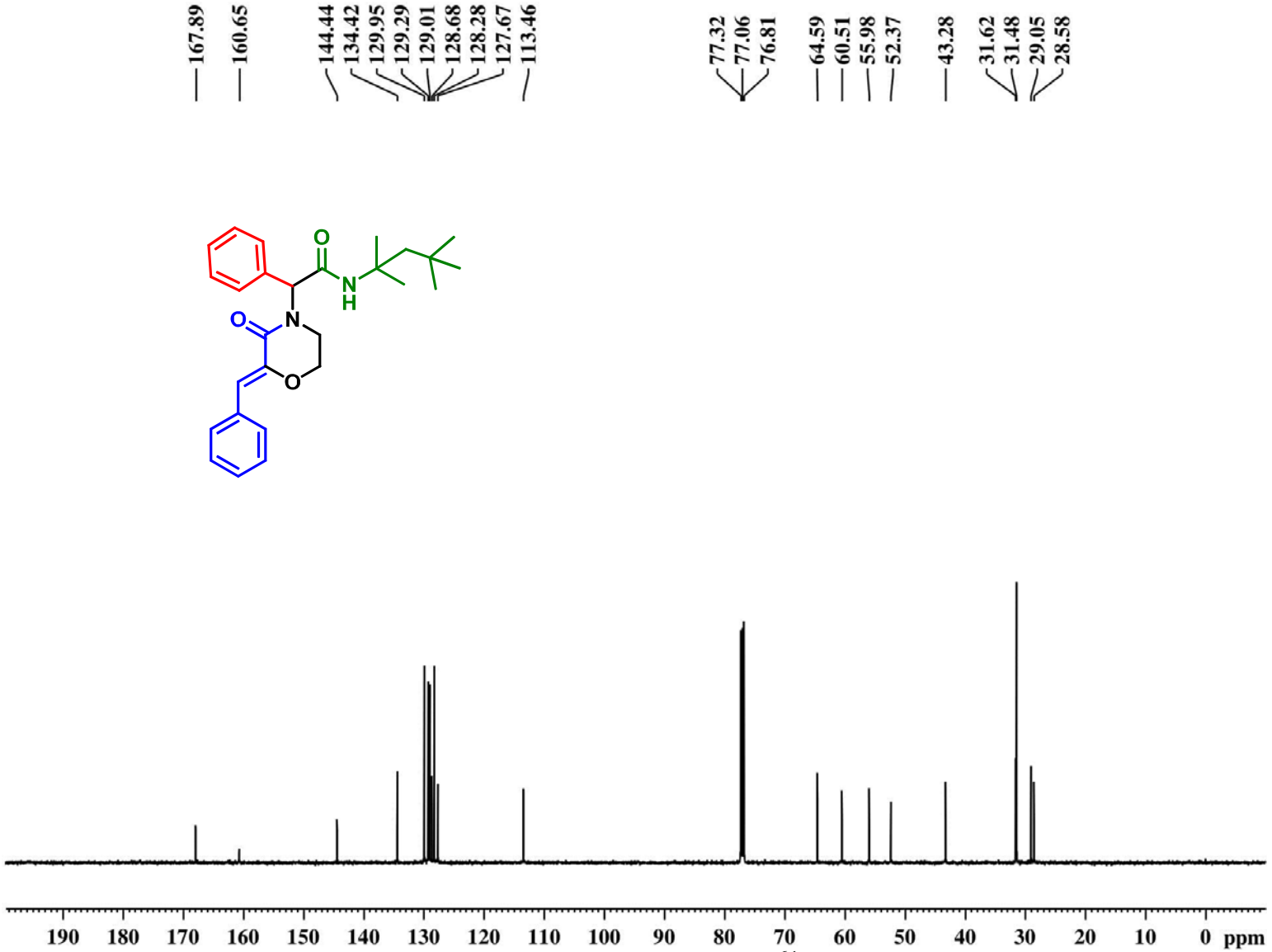




HRMS of compound **6g**


7.7099
7.6905
7.4155
7.4060
7.4032
7.4011
7.3953
7.3916
7.3762
7.3696
7.3581
7.3547
7.3401
7.3215
7.3020
7.2588
7.2483
7.2299
7.2116
6.8545
6.2872
6.2797
4.2970
4.2846
4.2768
4.2691
4.2567
4.2494
4.1173
4.1123
4.1040
3.8533
3.8203
3.8178
3.2368
3.2289
3.2171
3.2089
3.2042
3.1961
3.1843
1.8917
1.8545
1.5939
1.5567
1.4732
1.4274
0.9581
-0.0002
O
N
H
O
N
O
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
ppm
1.97
6.99
0.99
0.95
0.96
0.94
1.00
1.00
0.99
0.98
1.17
1.10
2.91
2.92
8.79

^{1}H NMR spectrum of **6h** (400MHz,$CDCl_3$)

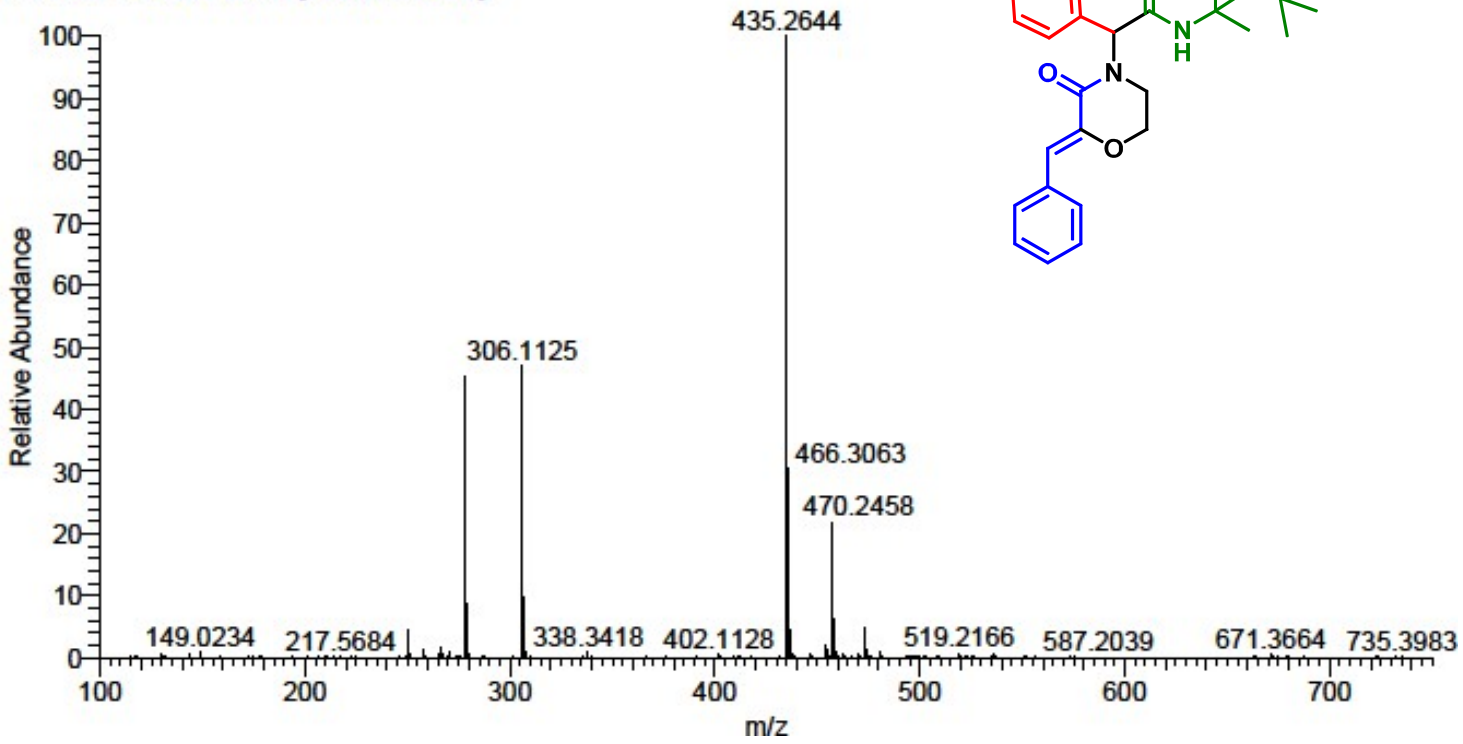

167.89
160.65
144.44
134.42
129.95
129.29
129.01
128.68
128.28
127.67
113.46
77.32
77.06
76.81
64.59
60.51
55.98
52.37
43.28
31.62
31.48
29.05
28.58
O
N
H
O
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

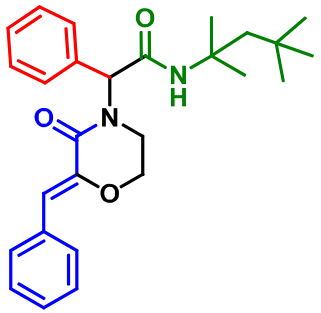
^{13}C NMR spectrum of **6h** (100 MHz, $CDCl_3$)

SAIF [HRMS Report]

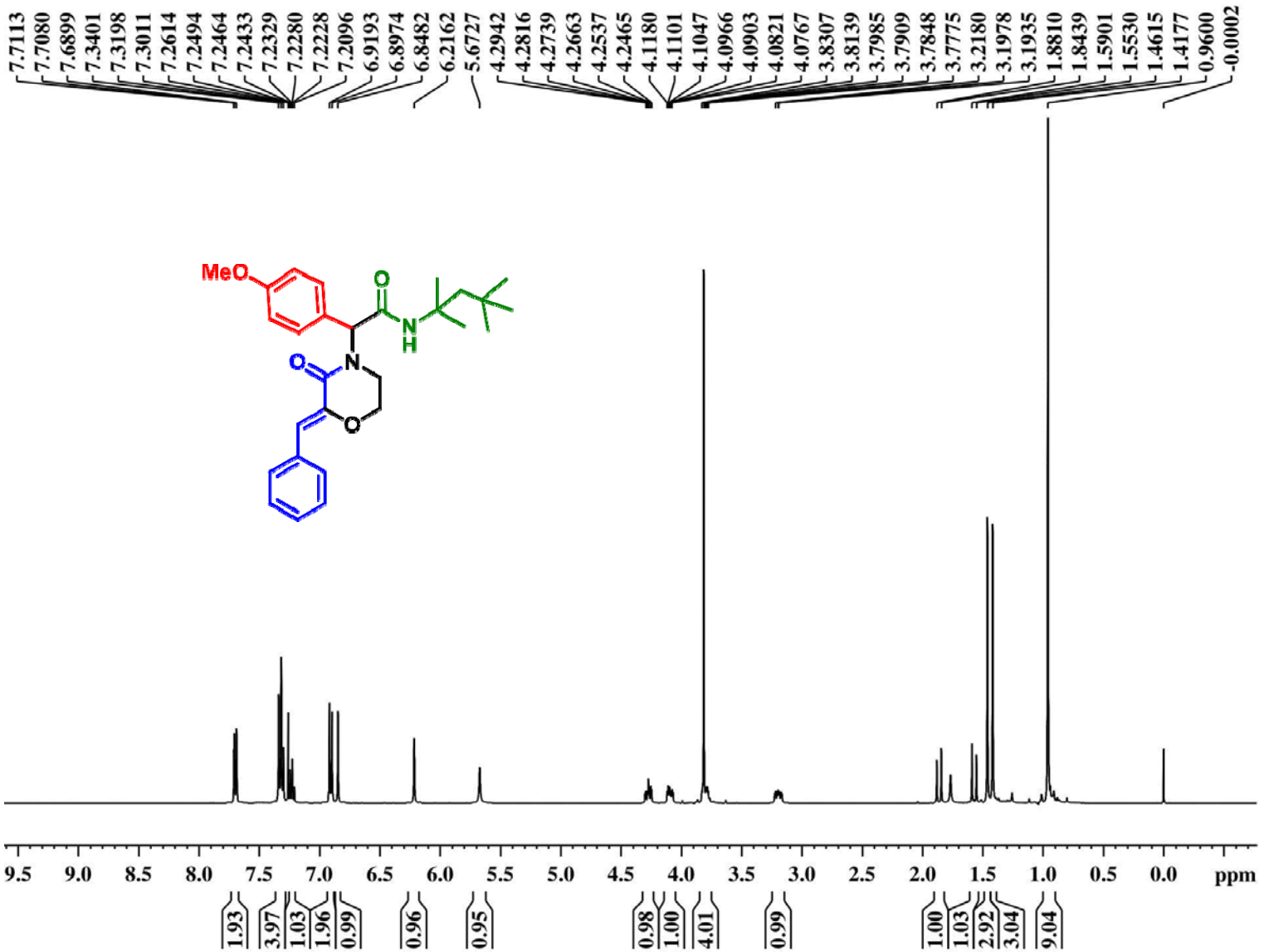
Data File:	HRMS21I22MAR09	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-15R	Sample Name:	
Acquisition Date:	03/22/21 12:59:26 PM	Run Time(min):	0.00
Vial:	CStk1-01:9	Injection Volume(µl):	1.00

HRMS21I22MAR09 #33-67 RT: 0.25-0.50 AV: 35 SB: 1 0.01 NL: 8.96E6
T: FTMS + c ESI Full ms [100.00-750.00]

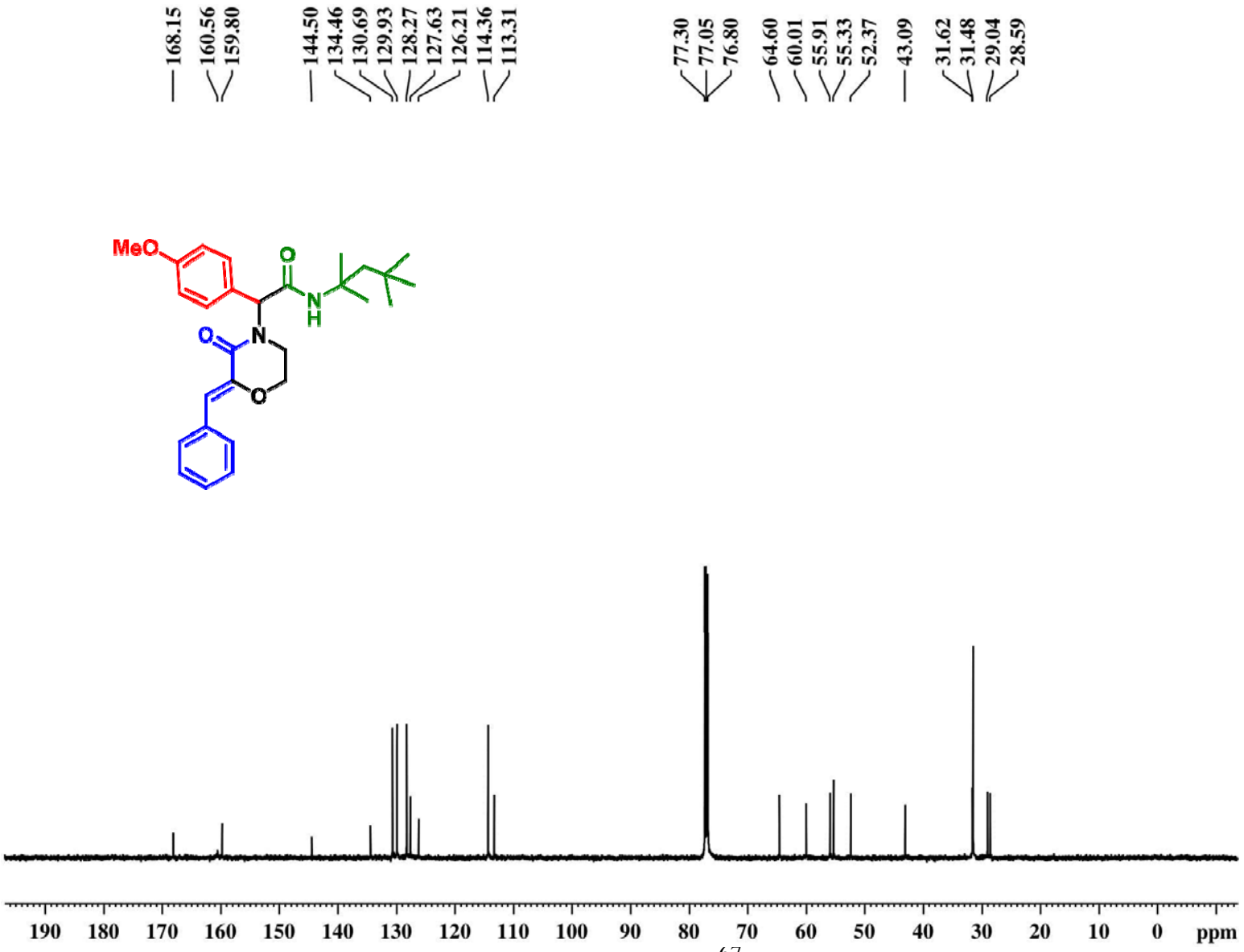




HRMS of compound **6h**


7.7113
7.7080
7.6899
7.3401
7.3198
7.3011
7.2614
7.2494
7.2464
7.2433
7.2329
7.2280
7.2228
7.2096
6.9193
6.8974
6.8482
6.2162
5.6727
4.2942
4.2816
4.2739
4.2663
4.2537
4.2465
4.1180
4.1101
4.1047
4.0966
4.0903
4.0821
4.0767
3.8307
3.8139
3.7985
3.7909
3.7848
3.7775
3.2180
3.1978
3.1935
1.8810
1.8439
1.5901
1.5530
1.4615
1.4177
0.9600
-0.0002
MeO
O
N
H
O
N
O
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 ppm
1.93
3.97
1.03
1.96
0.99
0.96
0.95
0.98
1.00
4.01
0.99
1.00
1.03
2.92
3.04
9.04

^{1}H NMR spectrum of **6i** (400MHz, $CDCl_3$)

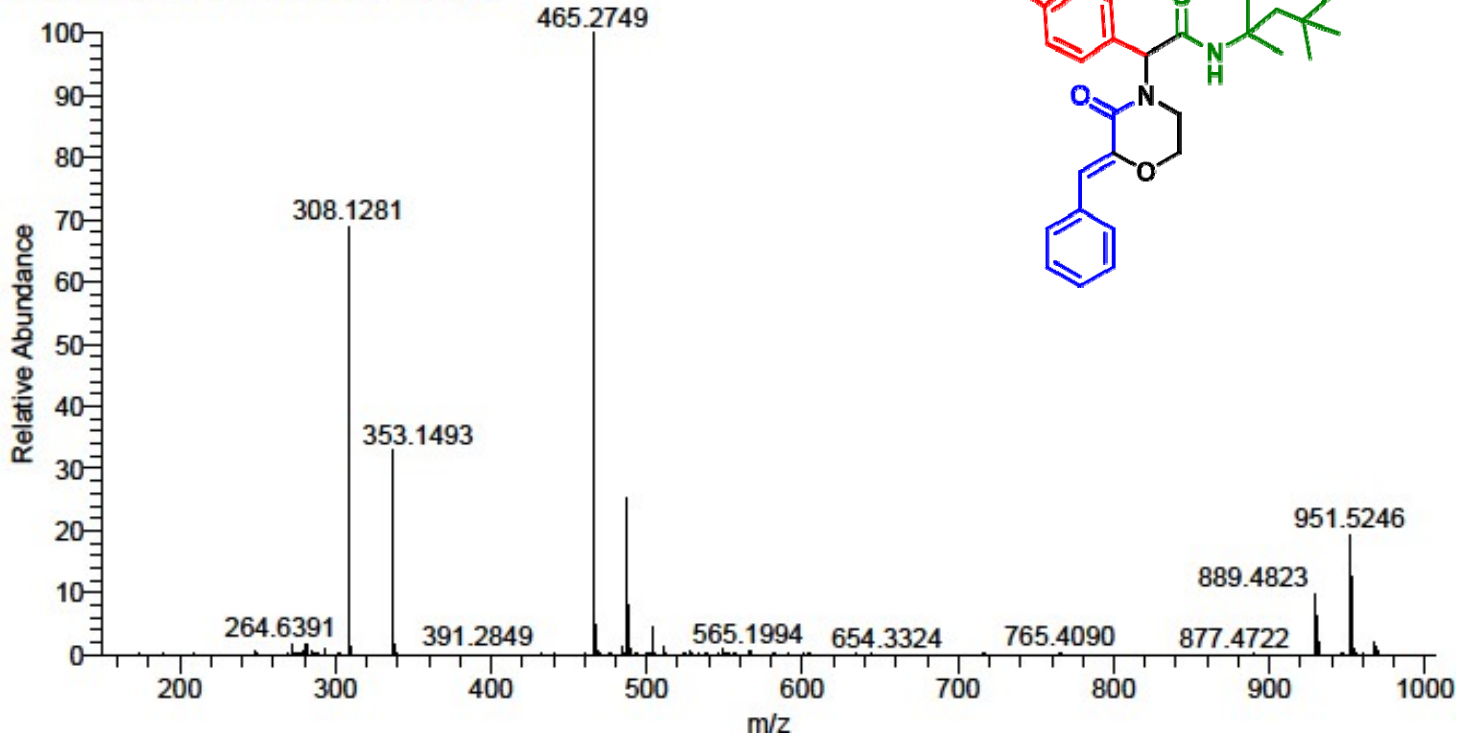

168.15
160.56
159.80
144.50
134.46
130.69
129.93
128.27
127.63
126.21
114.36
113.31
77.30
77.05
76.80
64.60
60.01
55.91
55.33
52.37
43.09
31.62
31.48
29.04
28.59
MeO
O
N
H
O
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

^{13}C NMR spectrum of **6i** (100 MHz, $CDCl_3$)

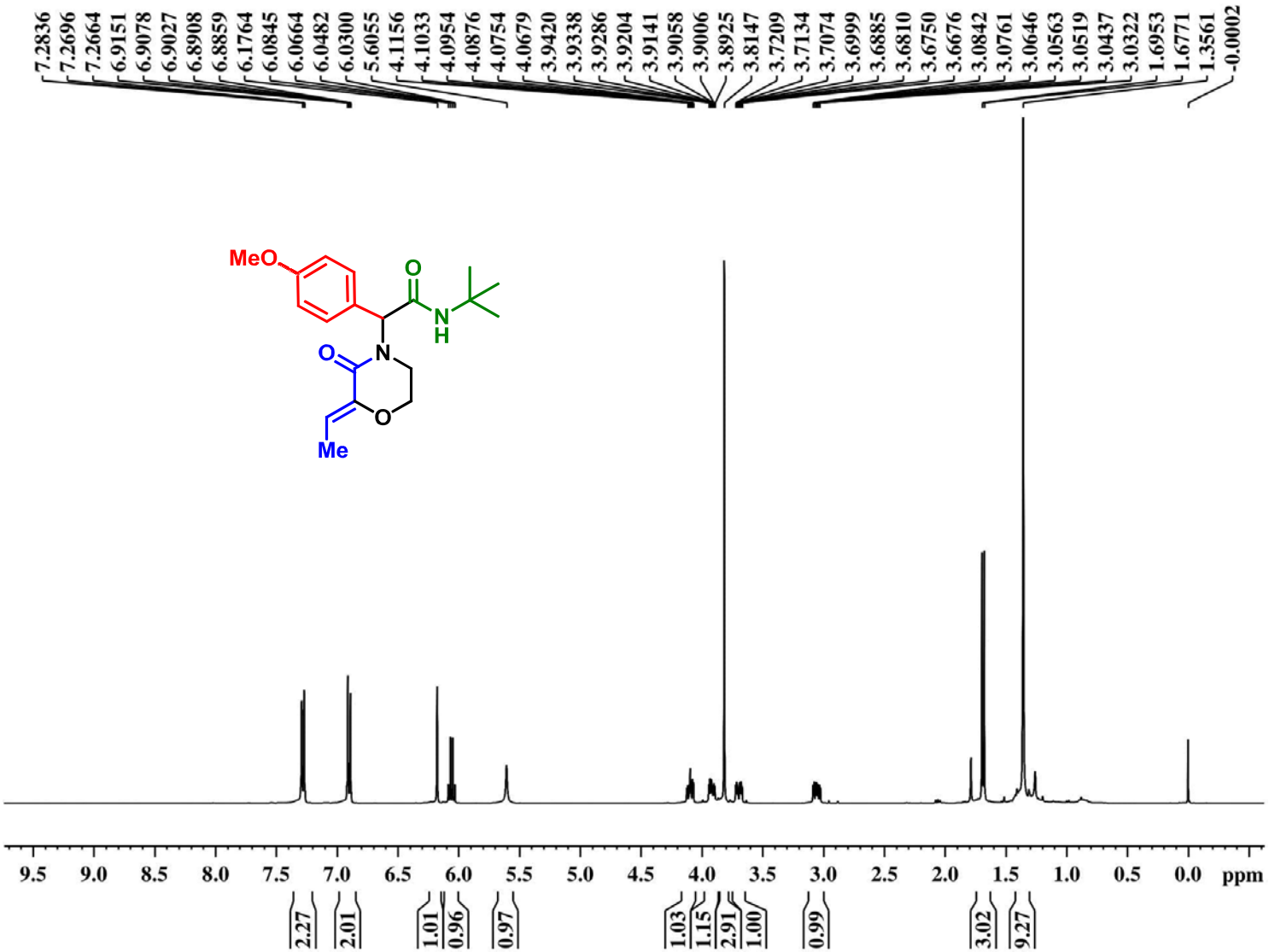
SAIF [HRMS Report]

Data File:	HRMS21I22MAR10	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-16R	Sample Name:	
Acquisition Date:	03/22/21 01:01:25 PM	Run Time(min):	0.00
Vial:	CStk1-01:10	Injection Volume(µl):	1.00

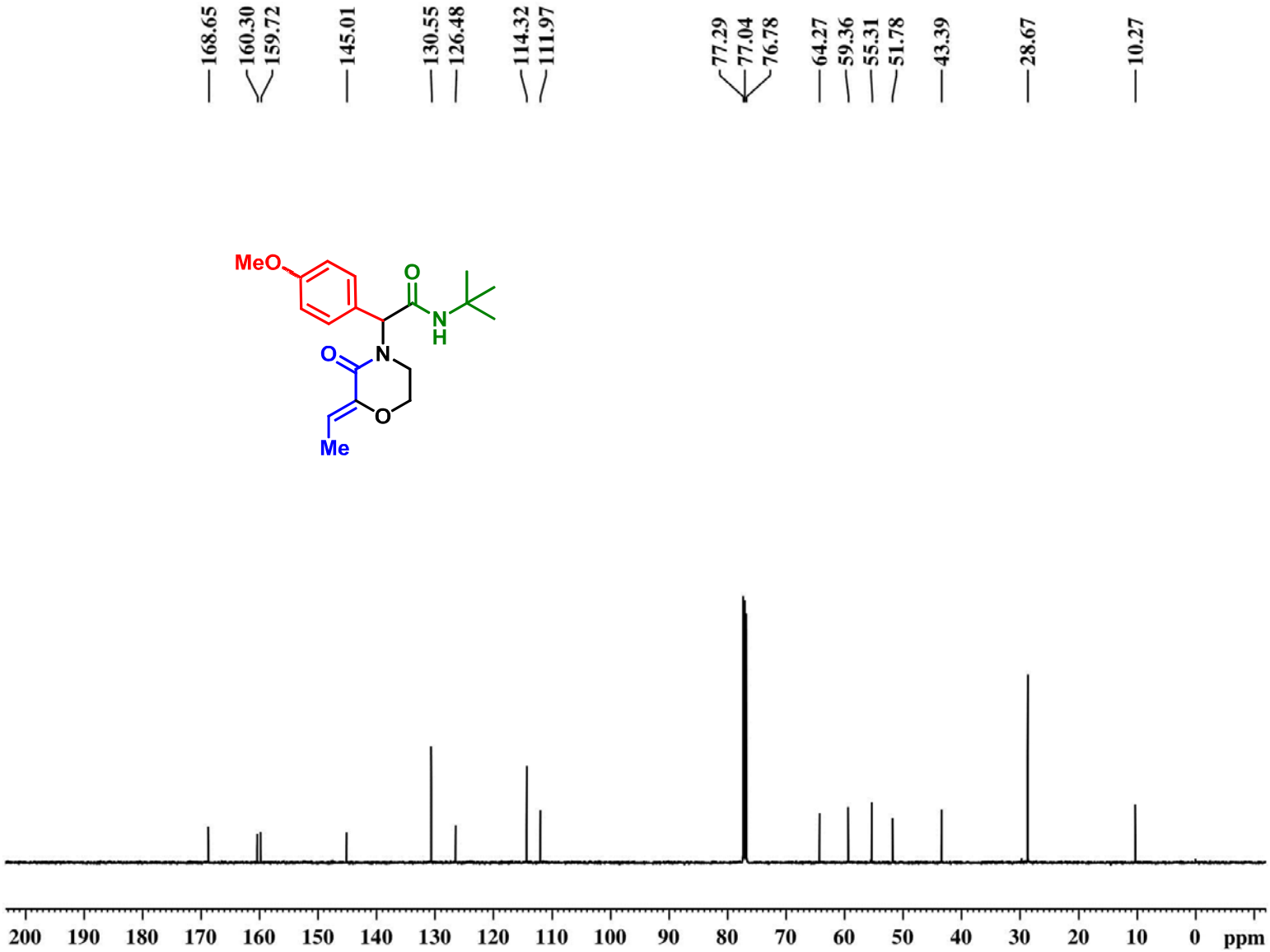
HRMS21I22MAR10 #33-67 RT: 0.25-0.50 AV: 35 SB: 1 0.00 NL: 5.23E6
T: FTMS + p ESI Full ms [150.00-1000.00]



HRMS of compound **6i**


7.2836
7.2696
7.2664
6.9151
6.9078
6.9027
6.8908
6.8859
6.1764
6.0845
6.0664
6.0482
6.0300
5.6055
4.1156
4.1033
4.0954
4.0876
4.0754
4.0679
3.9420
3.9338
3.9286
3.9204
3.9141
3.9058
3.9006
3.8925
3.8147
3.7209
3.7134
3.7074
3.6999
3.6885
3.6810
3.6750
3.6676
3.0842
3.0761
3.0646
3.0563
3.0519
3.0437
3.0322
1.6953
1.6771
1.3561
-0.0002
MeO
O
N
H
O
N
O
Me
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
ppm
2.27
2.01
1.01
0.96
0.97
1.03
1.15
2.91
1.00
0.99
3.02
9.27

^{1}H NMR spectrum of **6j** (400MHz, $CDCl_3$)

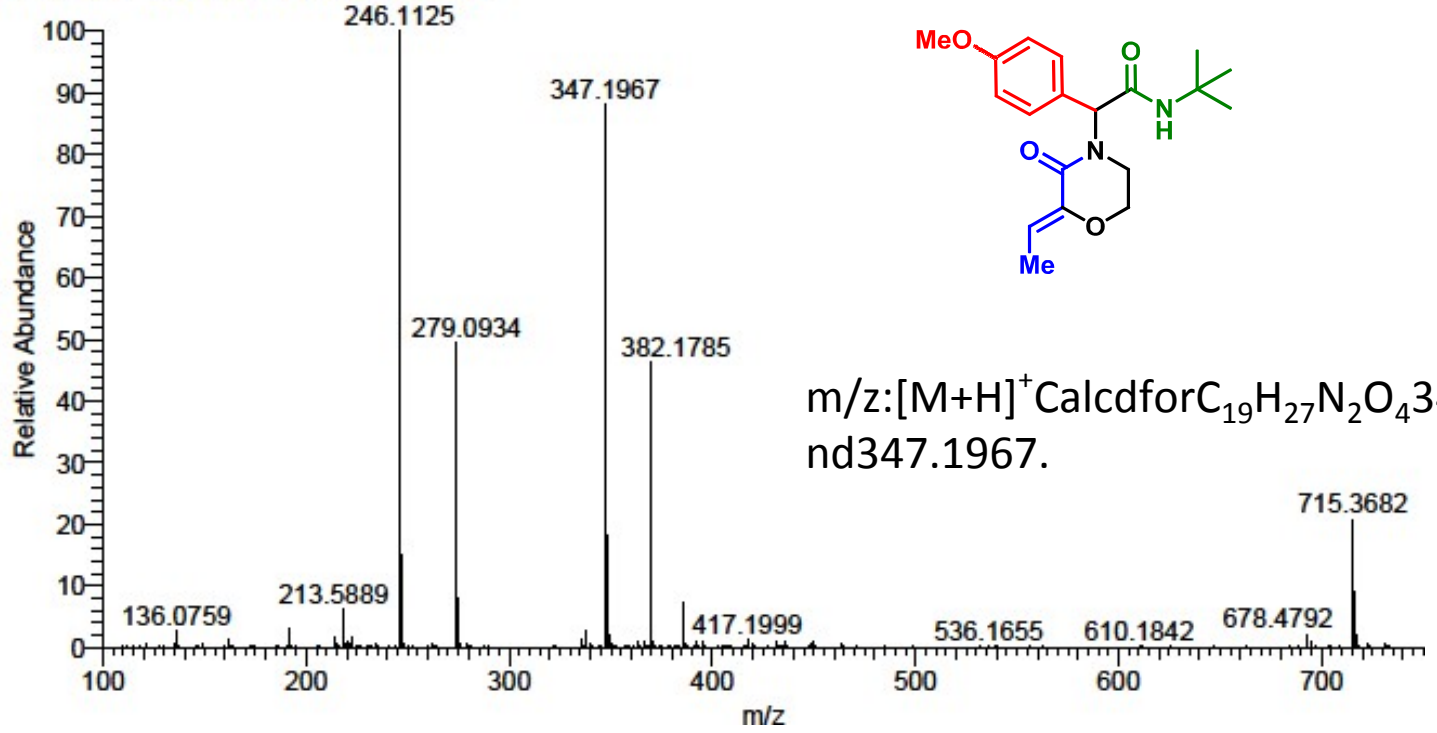

168.65
160.30
159.72
145.01
130.55
126.48
114.32
111.97
77.29
77.04
76.78
64.27
59.36
55.31
51.78
43.39
28.67
10.27
MeO
O
N
H
O
N
O
Me
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

^{13}C NMR spectrum of **6j** (100MHz, $CDCl_3$)

SAIF [HRMS Report]

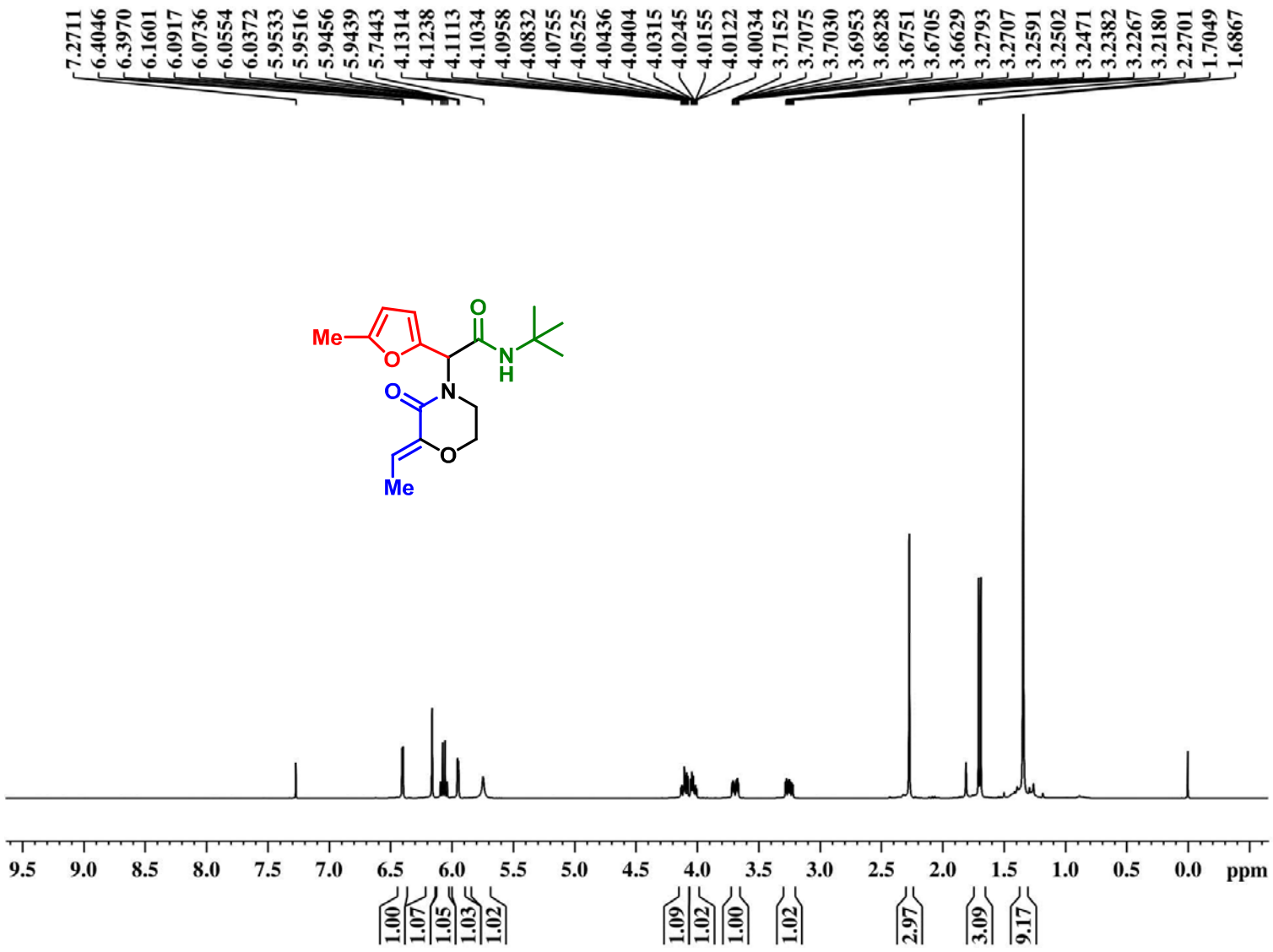
Data File:	HRMS21I23MAR10	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-24R	Sample Name:	
Acquisition Date:	03/23/21 11:48:40 AM	Run Time(min):	0.00
Vial:	CStk1-01:10	Injection Volume(µl):	1.00

HRMS21I23MAR10 #33-66 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 4.39E6
T: FTMS + c ESI Full ms [100.00-750.00]

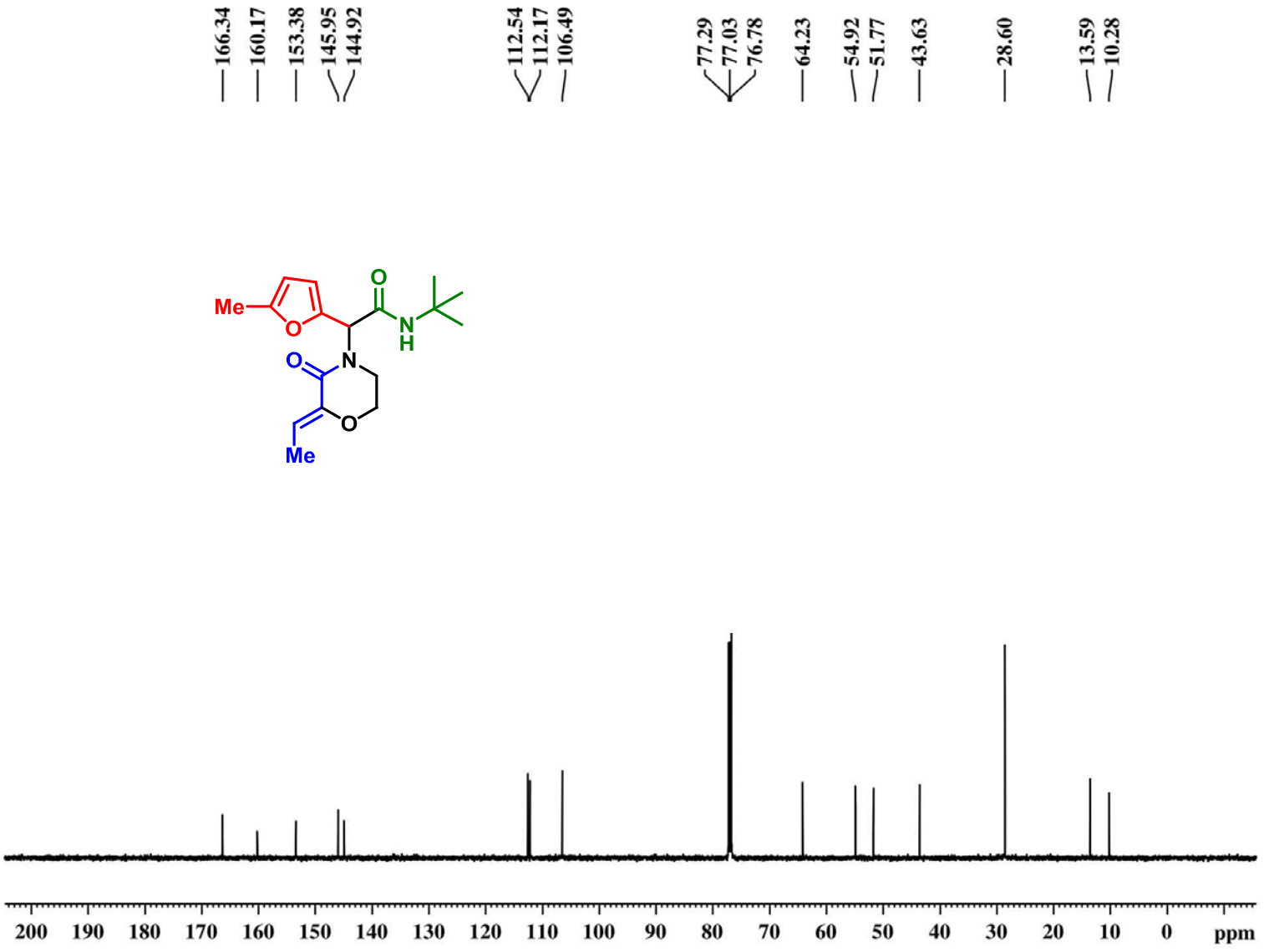


m/z:$[M+H]^+$CalcdforC$_{19}$H$_{27}$N$_2$O$_4$347.1965;Found347.1967.

HRMS of compound **6j**


7.2711
6.4046
6.3970
6.1601
6.0917
6.0736
6.0554
6.0372
5.9533
5.9516
5.9456
5.9439
5.7443
4.1314
4.1238
4.1113
4.1034
4.0958
4.0832
4.0755
4.0525
4.0436
4.0404
4.0315
4.0245
4.0155
4.0122
4.0034
3.7152
3.7075
3.7030
3.6953
3.6828
3.6751
3.6705
3.6629
3.2793
3.2707
3.2591
3.2502
3.2471
3.2382
3.2267
3.2180
2.2701
1.7049
1.6867
Me
O
O
N
H
O
N
O
Me
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
ppm
1.00
1.07
1.05
1.03
1.02
1.09
1.02
1.00
1.02
2.97
3.09
9.17

^{1}H NMR spectrum of **6k** (400MHz, $CDCl_3$)

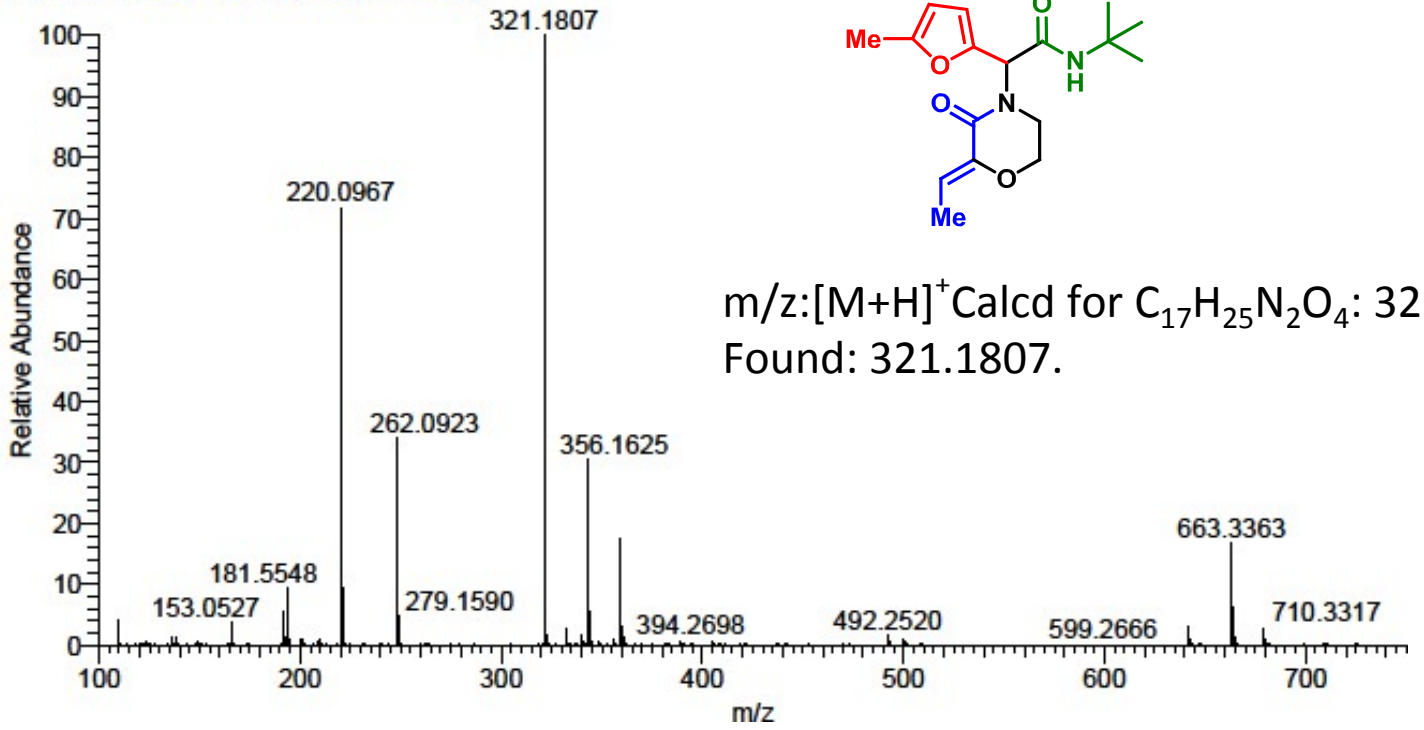

166.34
160.17
153.38
145.95
144.92
112.54
112.17
106.49
77.29
77.03
76.78
64.23
54.92
51.77
43.63
28.60
13.59
10.28
Me
O
O
N
H
O
N
O
Me
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

^{13}C NMR spectrum of **6k** (100MHz, $CDCl_3$)

SAIF [HRMS Report]

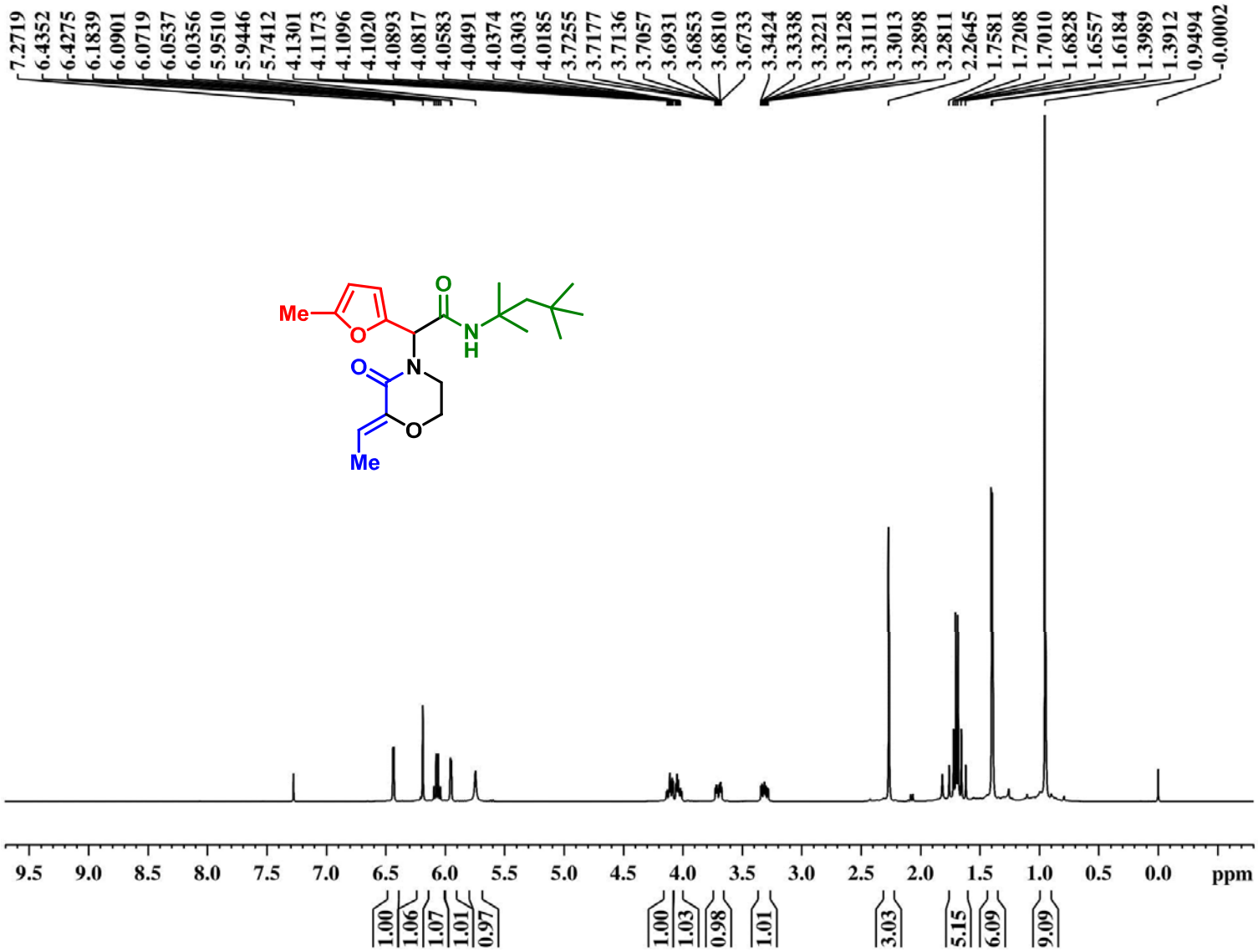
Data File:	HRMS21I19MAR11	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-27R	Sample Name:	
Acquisition Date:	03/19/21 11:22:09 AM	Run Time(min):	0.00
Vial:	CStk1-01:11	Injection Volume(µl):	1.00

HRMS21I19MAR11 #33-67 RT: 0.25-0.50 AV: 35 SB: 1 0.01 NL: 2.06E7
T: FTMS + c ESI Full ms [100.00-750.00]

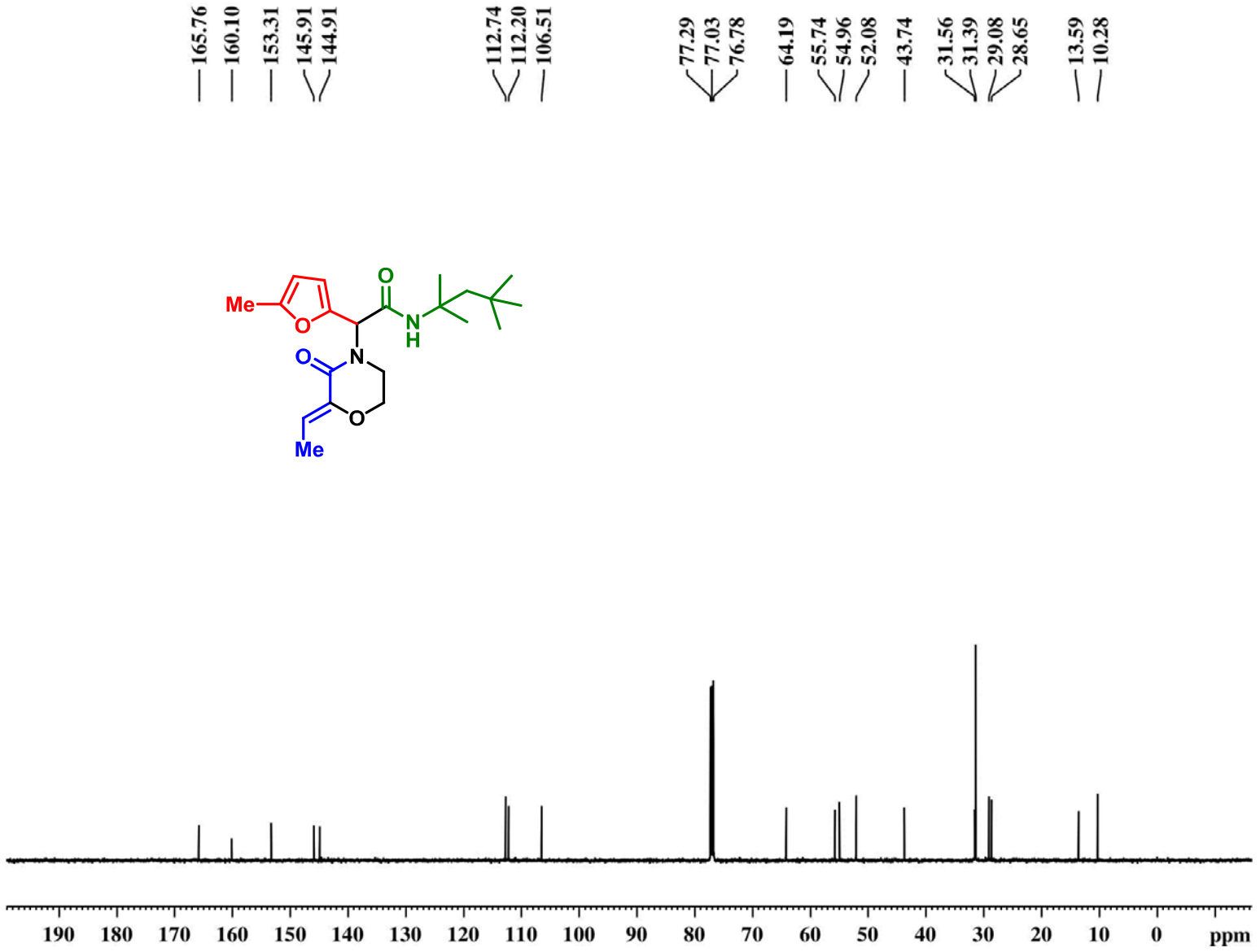


m/z:$[M+H]^+$Calcd for $C_{17}H_{25}N_2O_4$: 321.1801; Found: 321.1807.

HRMS of compound **6k**


7.2719
6.4352
6.4275
6.1839
6.0901
6.0719
6.0537
6.0356
5.9510
5.9446
5.7412
4.1301
4.1173
4.1096
4.1020
4.0893
4.0817
4.0583
4.0491
4.0374
4.0303
4.0185
3.7255
3.7177
3.7136
3.7057
3.6931
3.6853
3.6810
3.6733
3.3424
3.3338
3.3221
3.3128
3.3111
3.3013
3.2898
3.2811
2.2645
1.7581
1.7208
1.7010
1.6828
1.6557
1.6184
1.3989
1.3912
0.9494
-0.0002
Me
O
N
H
O
N
O
Me
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
ppm
1.00
1.06
1.07
1.01
0.97
1.00
1.03
0.98
1.01
3.03
5.15
6.09
9.09

^{1}H NMR spectrum of **6l** (400MHz, $CDCl_3$)

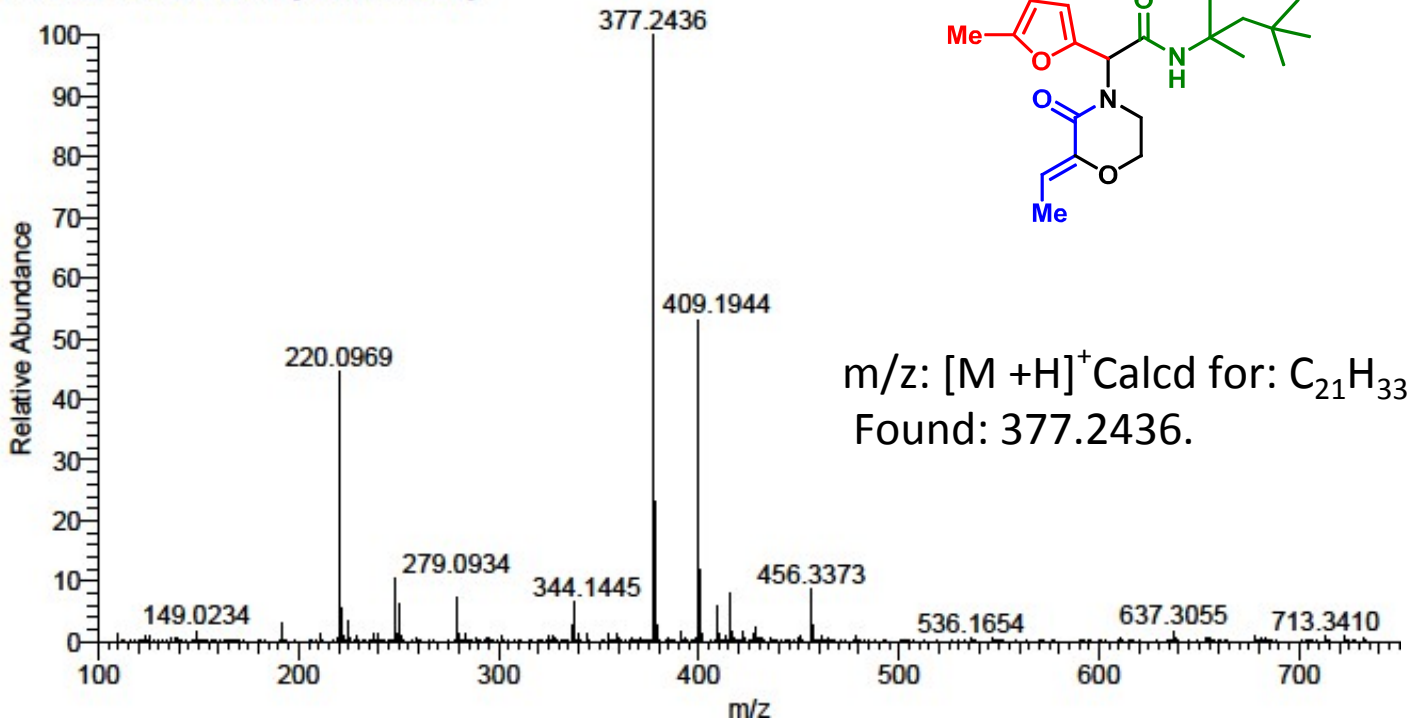

165.76
160.10
153.31
145.91
144.91
112.74
112.20
106.51
77.29
77.03
76.78
64.19
55.74
54.96
52.08
43.74
31.56
31.39
29.08
28.65
13.59
10.28
Me
O
N
H
O
N
O
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

^{13}C NMR spectrum of **6l** (100 MHz, $CDCl_3$)

SAIF [HRMS Report]

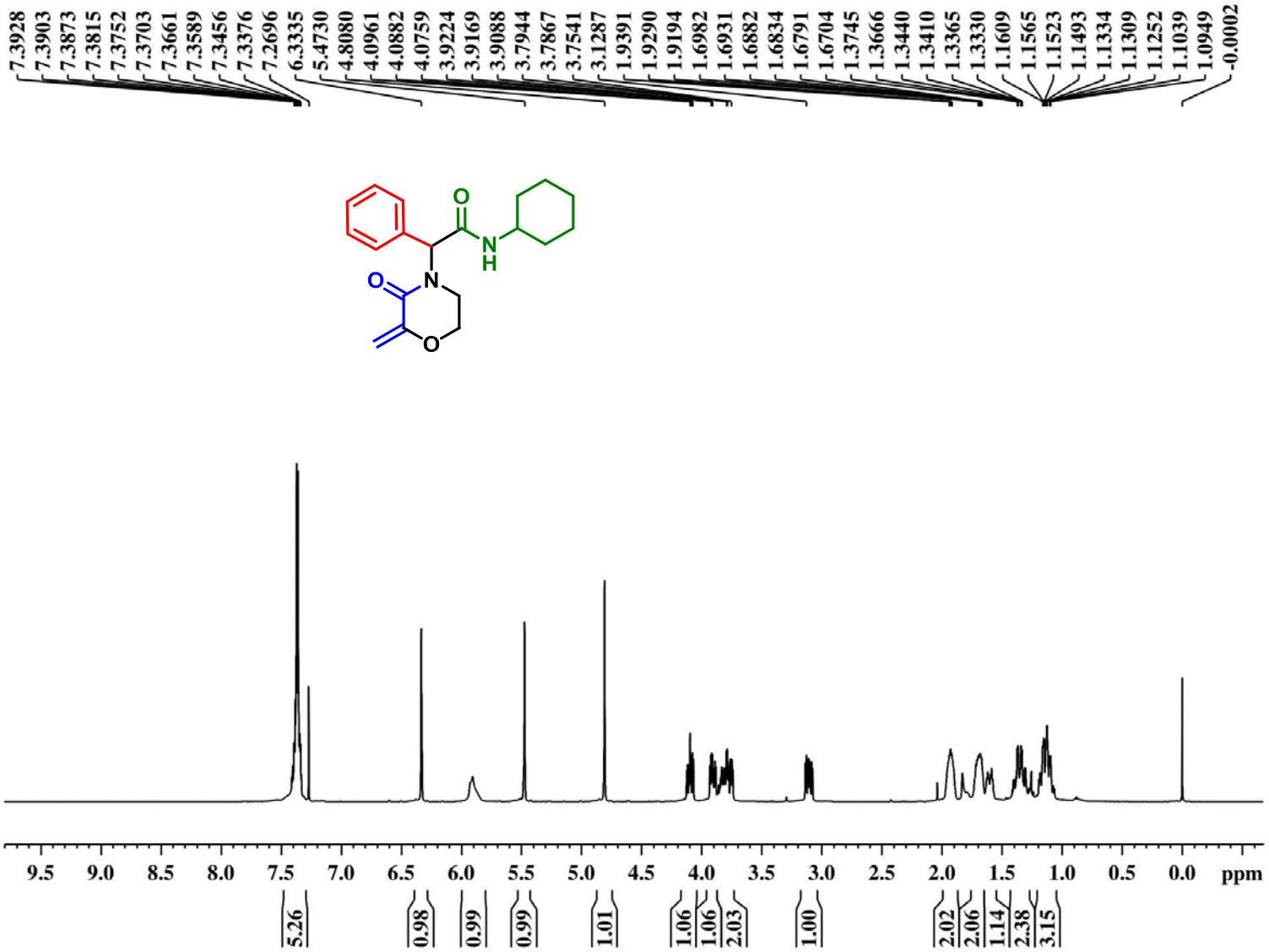
Data File:	HRMS21I23MAR11	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-26R	Sample Name:	
Acquisition Date:	03/23/21 11:50:41 AM	Run Time(min):	0.00
Vial:	CStk1-01:11	Injection Volume(µl):	1.00

HRMS21I23MAR11 #33-66 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 2.34E6
T: FTMS + c ESI Full ms [100.00-750.00]

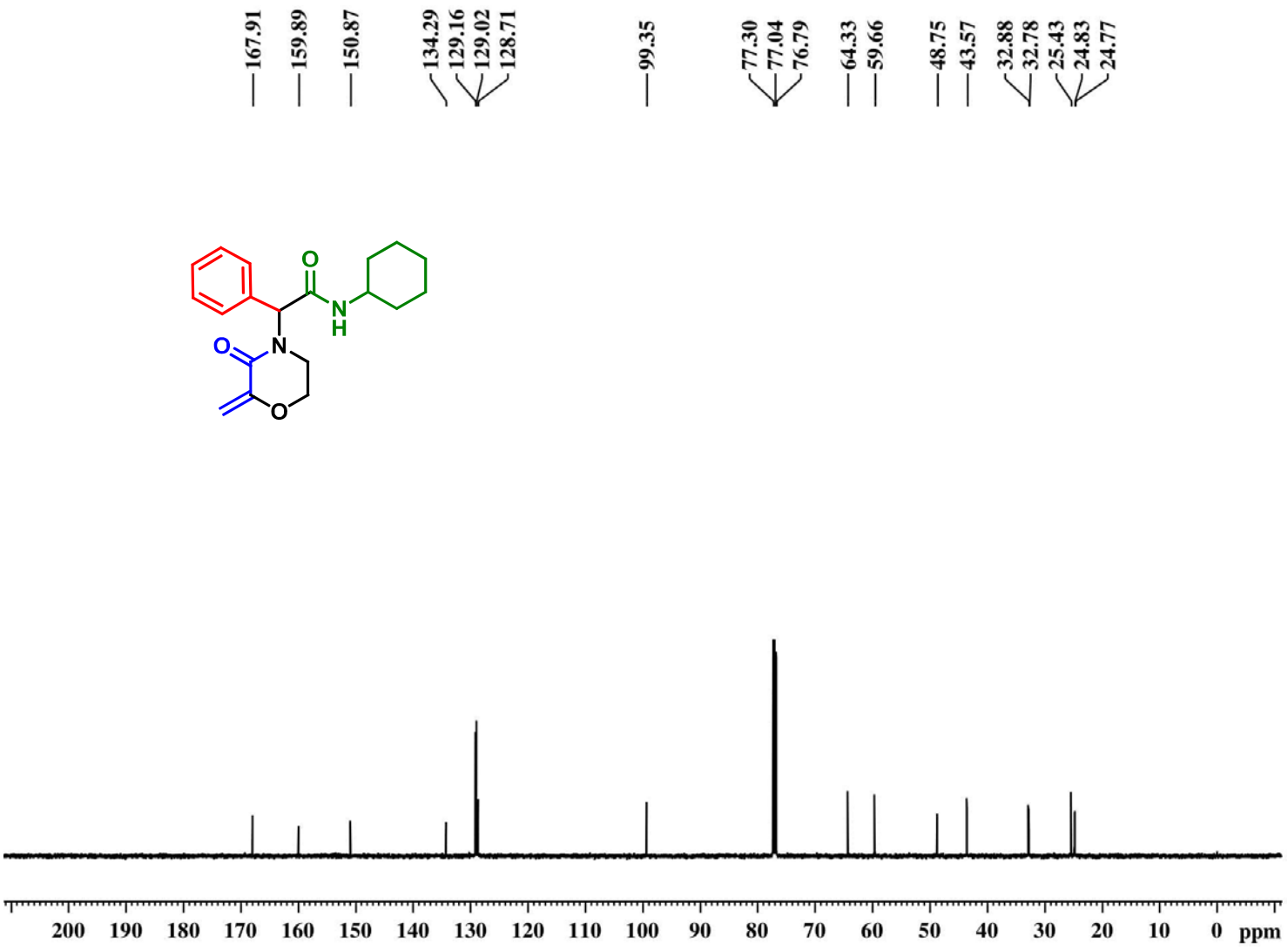


m/z: $[M+H]^+$Calcd for: $C_{21}H_{33}N_2O_4$377.2435;
Found: 377.2436.

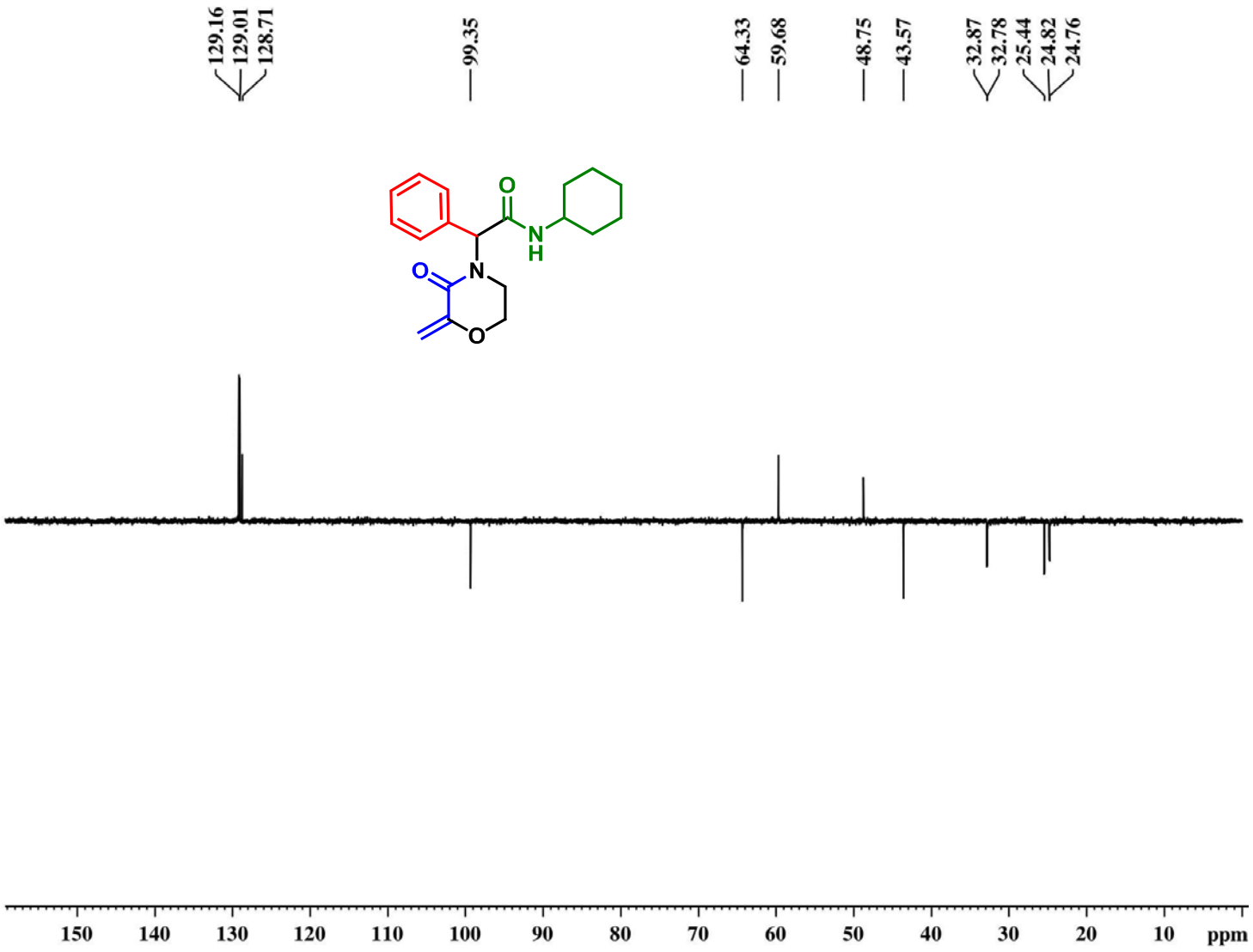
HRMS of compound **6l**


7.3928
7.3903
7.3873
7.3815
7.3752
7.3703
7.3661
7.3589
7.3456
7.3376
7.2696
6.3335
5.4730
4.8080
4.0961
4.0882
4.0759
3.9224
3.9169
3.9088
3.7944
3.7867
3.7541
3.1287
1.9391
1.9290
1.9194
1.6982
1.6931
1.6882
1.6834
1.6791
1.6704
1.3745
1.3666
1.3440
1.3410
1.3365
1.3330
1.1609
1.1565
1.1523
1.1493
1.1334
1.1309
1.1252
1.1039
1.0949
-0.0002
O
N
H
O
N
O
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
ppm
5.26
0.98
0.99
0.99
1.01
1.06
1.06
2.03
1.00
2.02
2.06
1.14
2.38
3.15

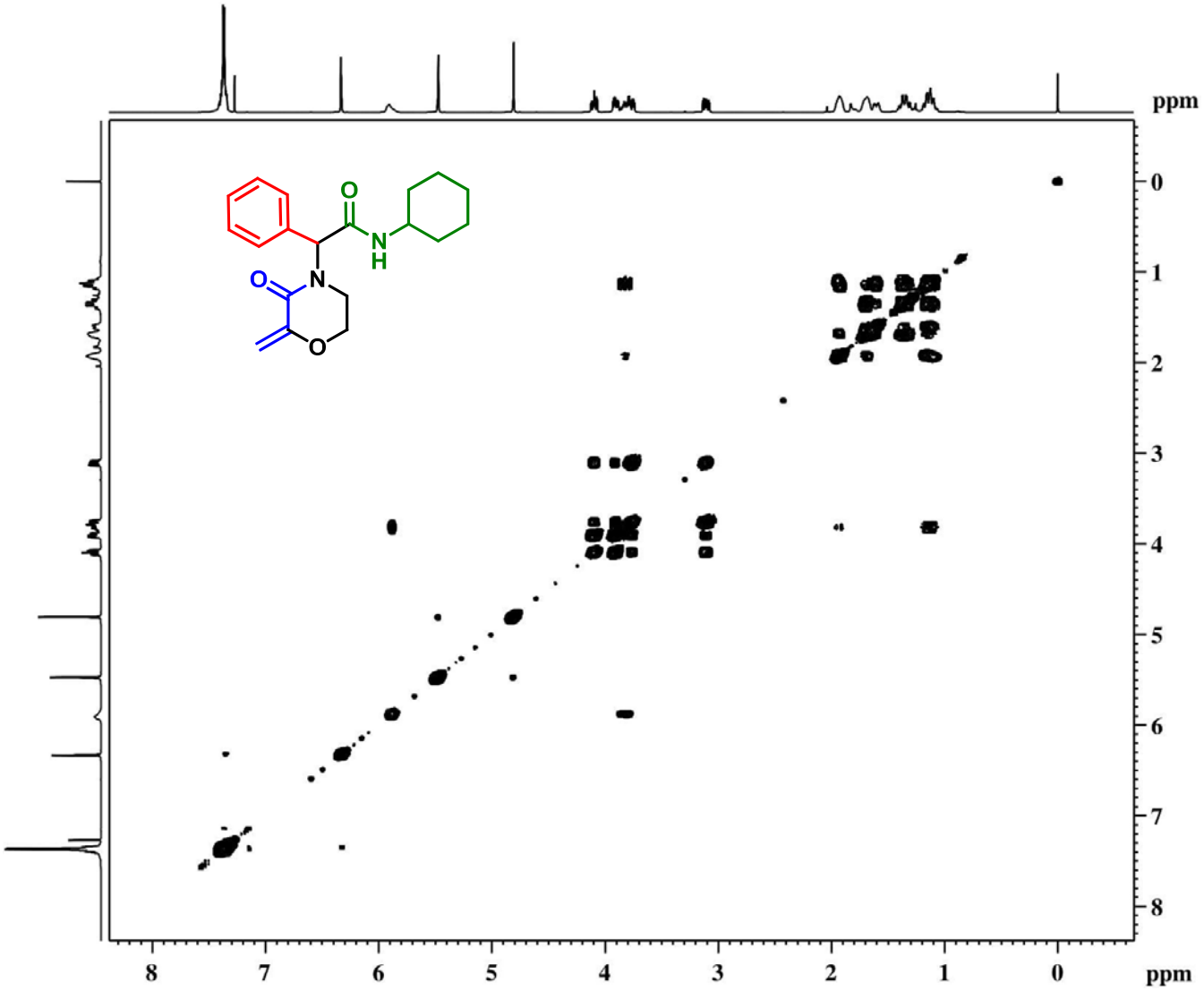
^{1}H NMR spectrum of **6m** (400MHz, $CDCl_3$)


167.91
159.89
150.87
134.29
129.16
129.02
128.71
99.35
77.30
77.04
76.79
64.33
59.66
48.75
43.57
32.88
32.78
25.43
24.83
24.77
O
N
H
O
N
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

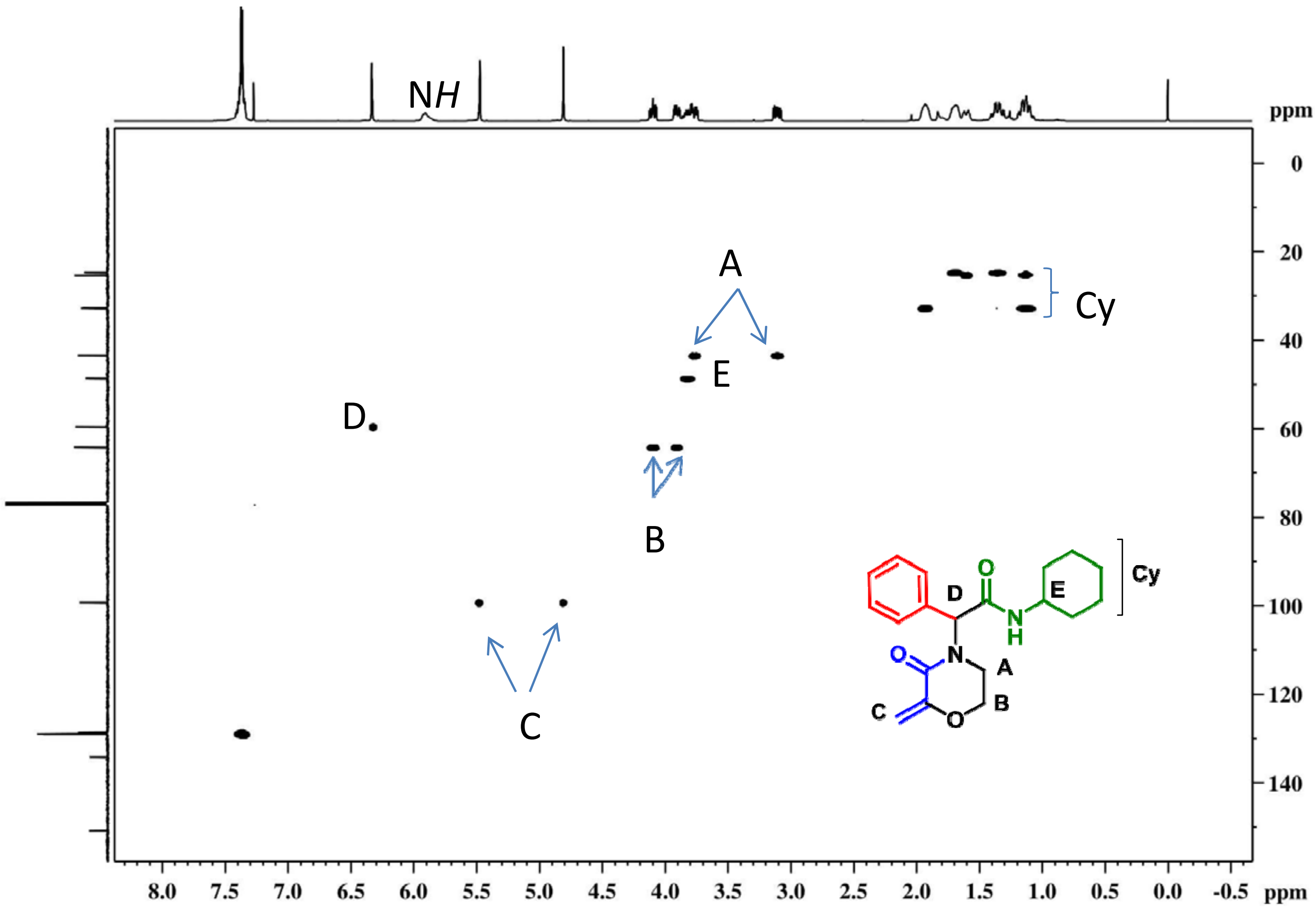
^{13}C NMR spectrum of **6m** (100MHz, $CDCl_3$)


129.16
129.01
128.71
99.35
64.33
59.68
48.75
43.57
32.87
32.78
25.44
24.82
24.76
O
N
H
O
N
O
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

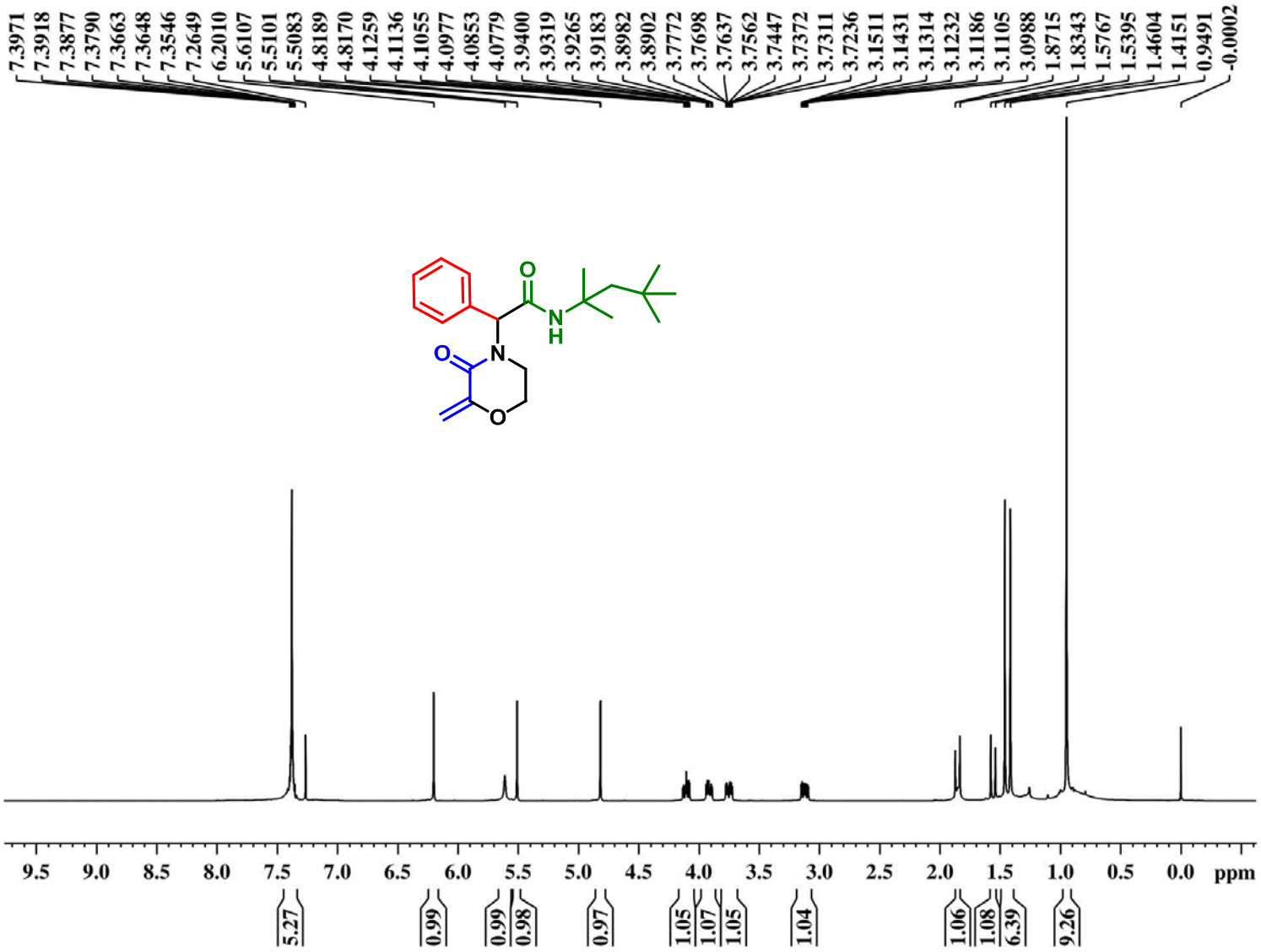
DEPT-135 spectrum of **6m** (100MHz, $CDCl_3$)


O
N
H
O
N
O
ppm
0
1
2
3
4
5
6
7
8
8
7
6
5
4
3
2
1
0
ppm

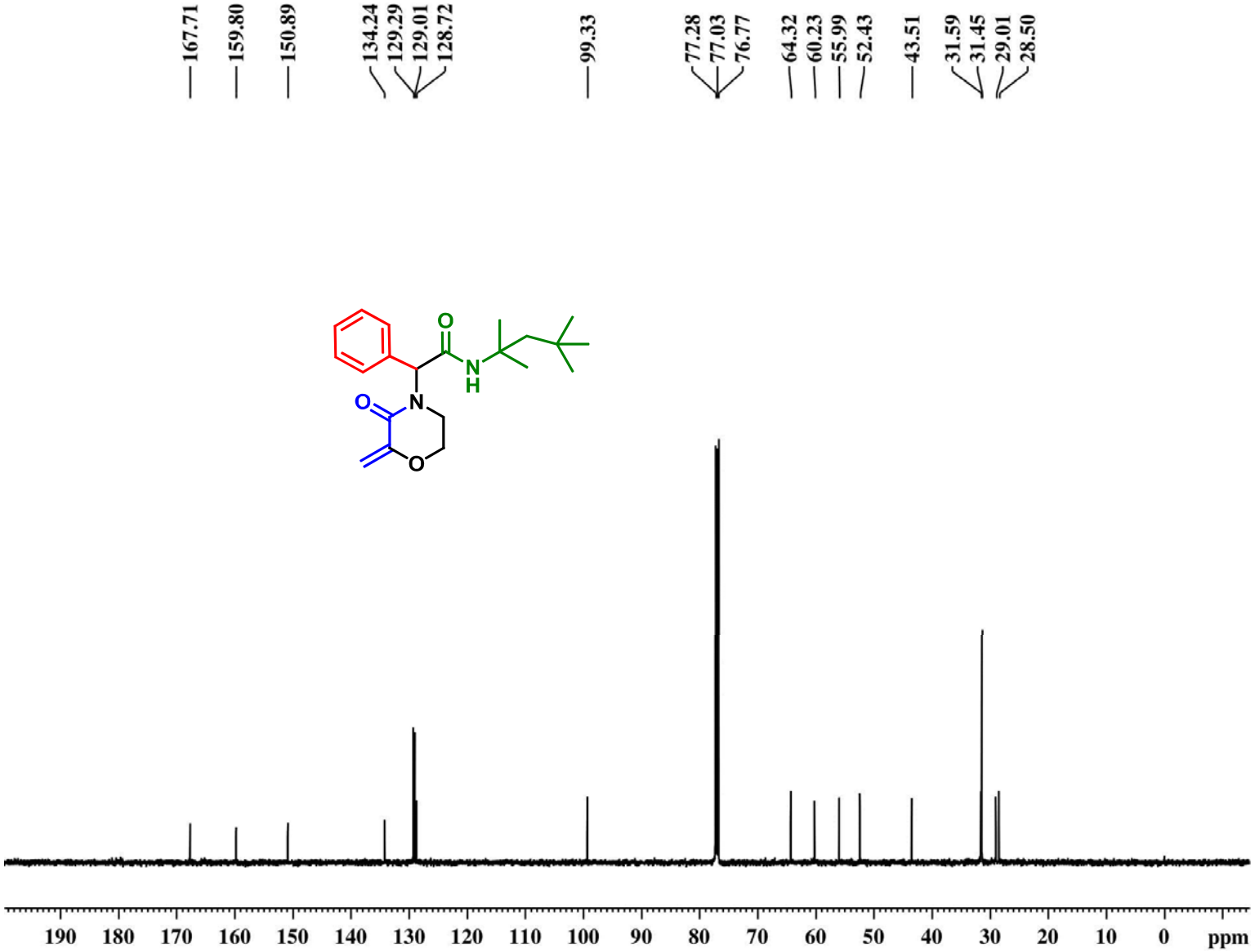
2D-COSY spectrum of **6m** (400 MHz, $CDCl_3$)


NH
ppm
A
E
D
B
C
Cy
D
O
N
H
E
Cy
O
N
A
C
O
B
0
20
40
60
80
100
120
140
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
ppm

2D-HSQC spectrum of **6m** (400MHz, $CDCl_3$)


7.3971
7.3918
7.3877
7.3790
7.3663
7.3648
7.3546
7.2649
6.2010
5.6107
5.5101
5.5083
4.8189
4.8170
4.1259
4.1136
4.1055
4.0977
4.0853
4.0779
3.9400
3.9319
3.9265
3.9183
3.8982
3.8902
3.7772
3.7698
3.7637
3.7562
3.7447
3.7372
3.7311
3.7236
3.1511
3.1431
3.1314
3.1232
3.1186
3.1105
3.0988
1.8715
1.8343
1.5767
1.5395
1.4604
1.4151
0.9491
-0.0002
O
N
H
O
N
O
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
ppm
5.27
0.99
0.99
0.98
0.97
1.05
1.07
1.05
1.04
1.06
1.08
6.39
9.26

^{1}H NMR spectrum of **6n** (400MHz, $CDCl_3$)

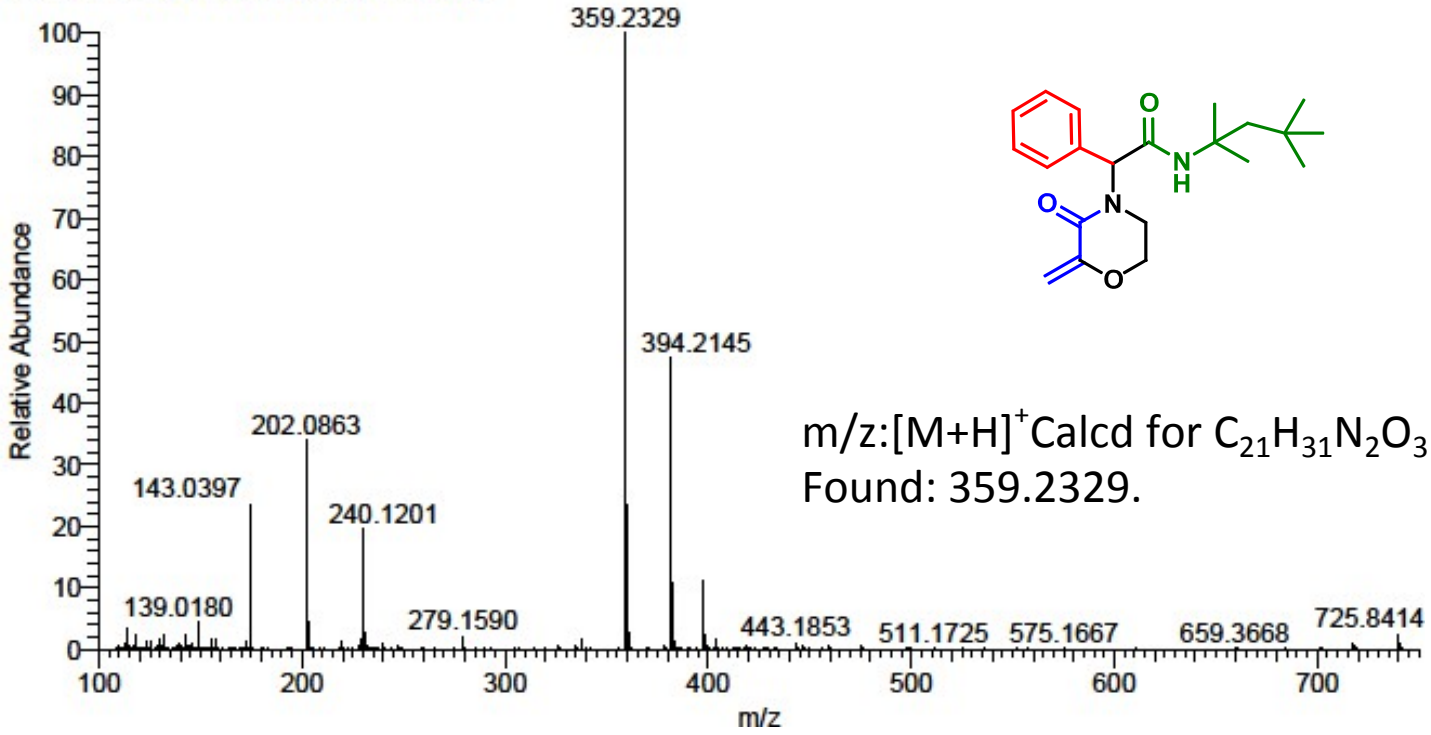

167.71
159.80
150.89
134.24
129.29
129.01
128.72
99.33
77.28
77.03
76.77
64.32
60.23
55.99
52.43
43.51
31.59
31.45
29.01
28.50
O
N
H
O
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

^{13}C NMR spectrum of **6n** (100 MHz, $CDCl_3$)

SAIF [HRMS Report]

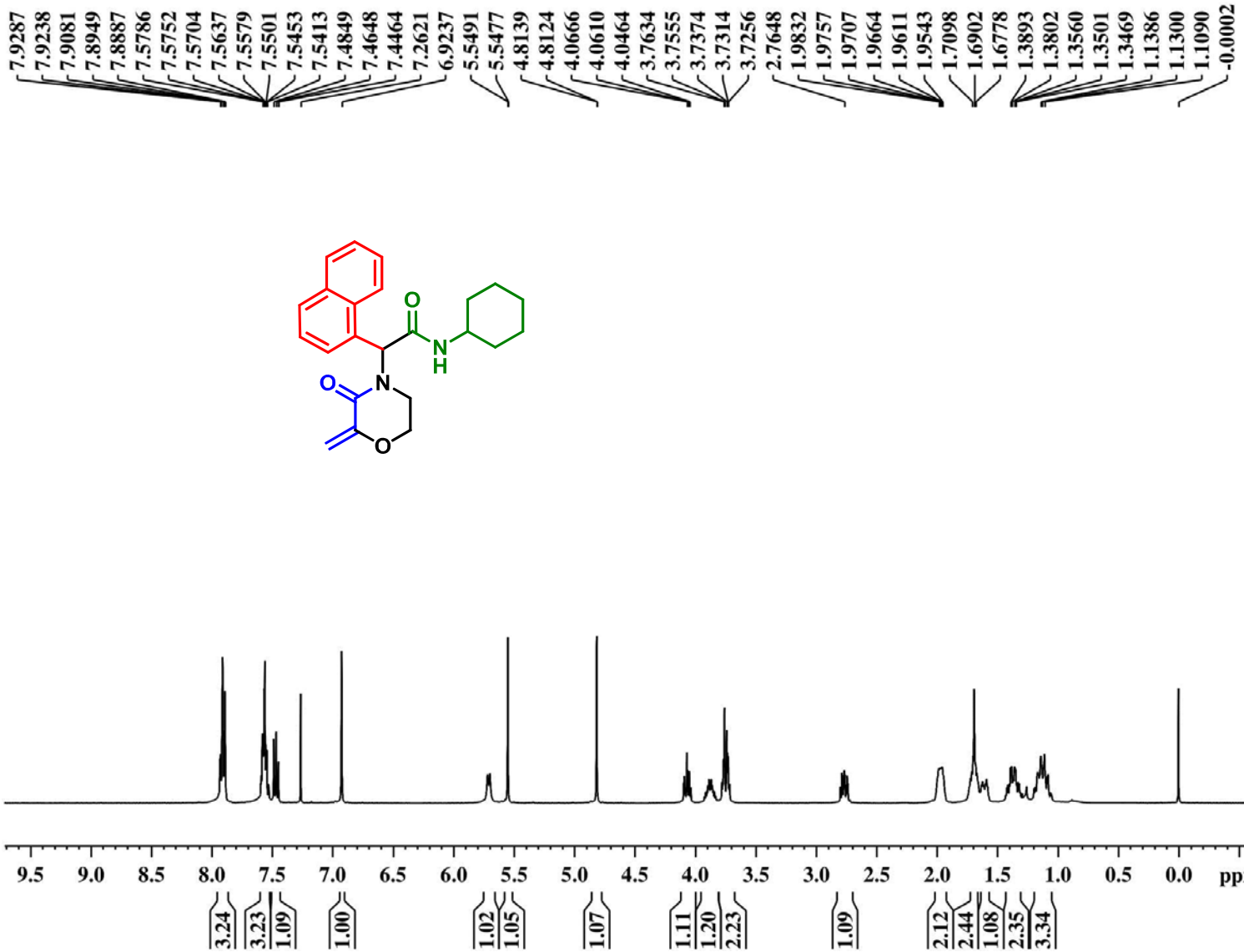
Data File:	HRMS21I19MAR12	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-28R	Sample Name:	
Acquisition Date:	03/19/21 11:24:08 AM	Run Time(min):	0.00
Vial:	CStk1-01:12	Injection Volume(µl):	1.00

HRMS21I19MAR12 #32-65 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 3.23E6
T: FTMS + c ESI Full ms [100.00-750.00]

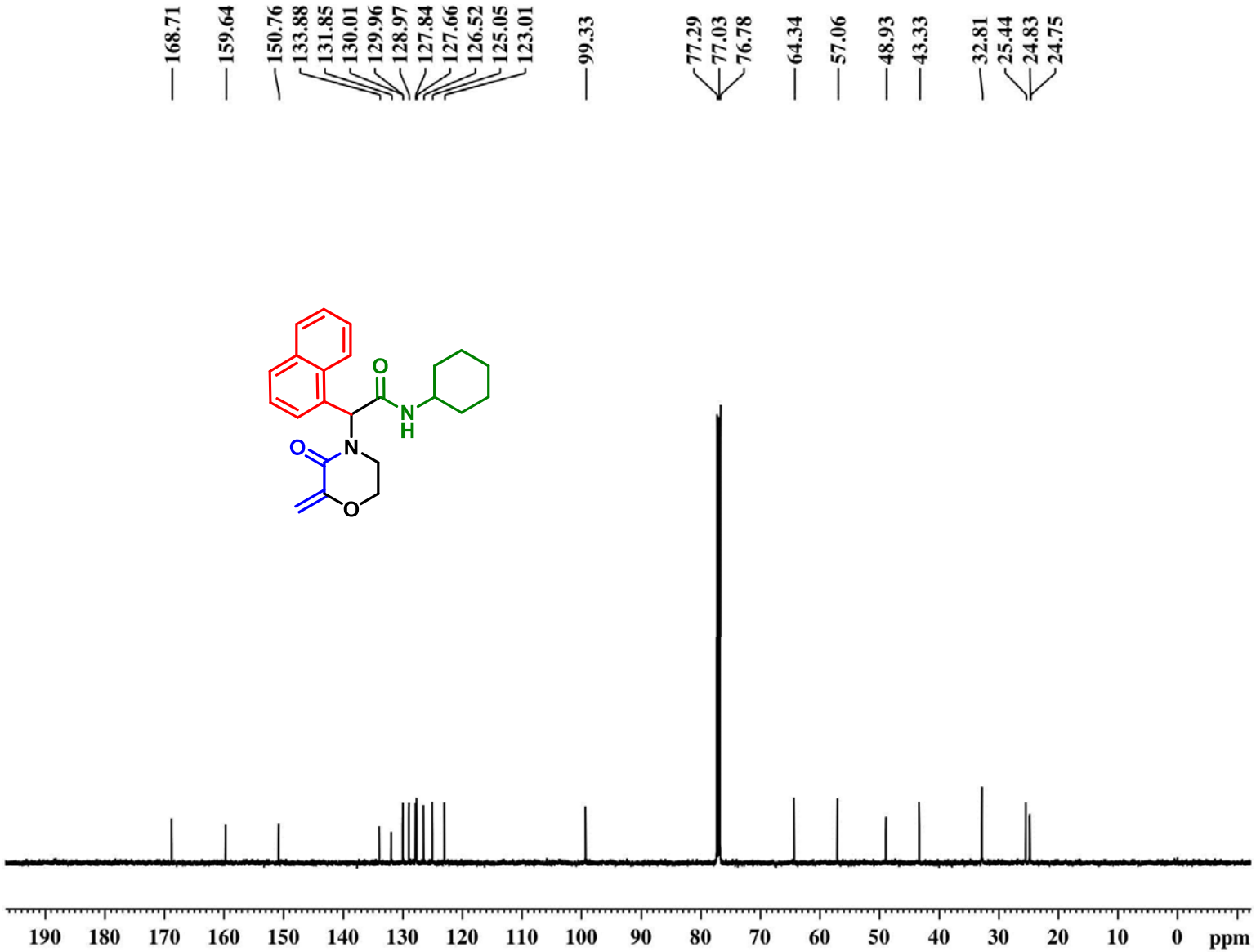


m/z:$[M+H]^+$Calcd for $C_{21}H_{31}N_2O_3$: 359.2329; Found: 359.2329.

HRMS of compound **6n**


7.9287
7.9238
7.9081
7.8949
7.8887
7.5786
7.5752
7.5704
7.5637
7.5579
7.5501
7.5453
7.5413
7.4849
7.4648
7.4464
7.2621
6.9237
5.5491
5.5477
4.8139
4.8124
4.0666
4.0610
4.0464
3.7634
3.7555
3.7374
3.7314
3.7256
2.7648
1.9832
1.9757
1.9707
1.9664
1.9611
1.9543
1.7098
1.6902
1.6778
1.3893
1.3802
1.3560
1.3501
1.3469
1.1386
1.1300
1.1090
-0.0002
O
N
H
O
N
O
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
ppm
3.24
3.23
1.09
1.00
1.02
1.05
1.07
1.11
1.20
2.23
1.09
2.12
2.44
1.08
2.35
3.34

^{1}H NMR spectrum of **6o** (400MHz, $CDCl_3$)

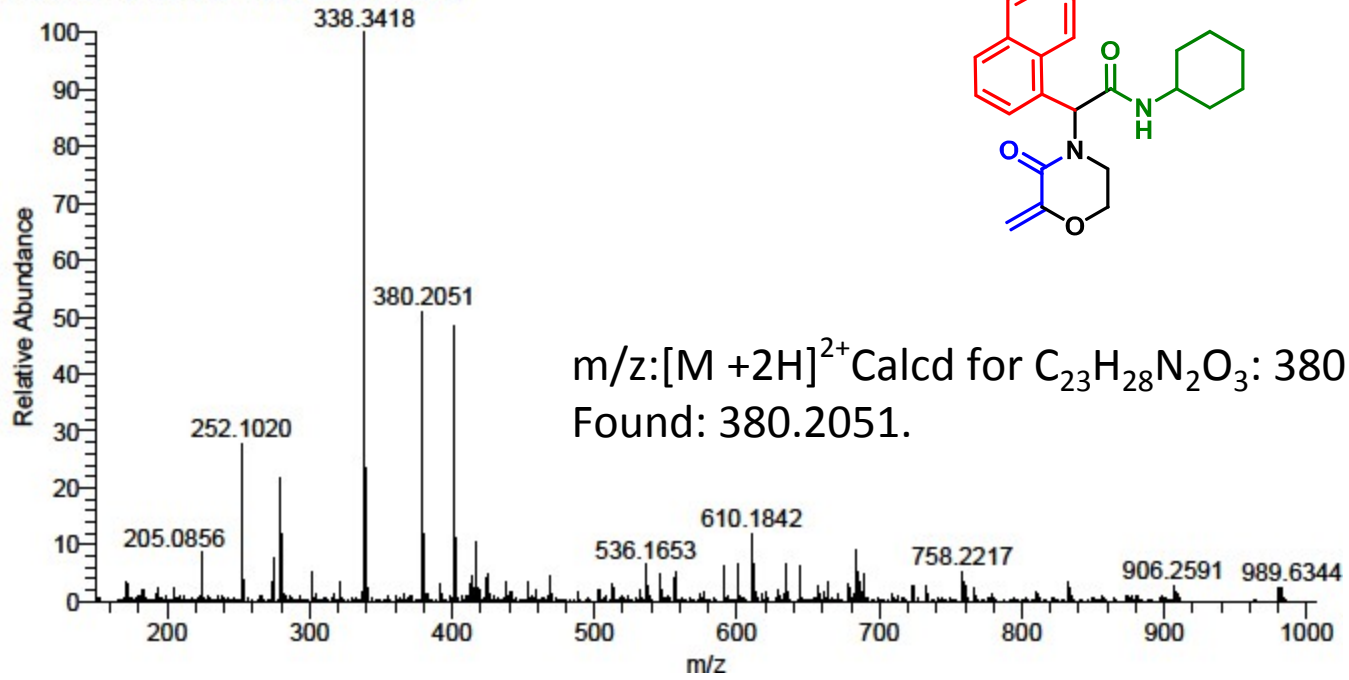

168.71
159.64
150.76
133.88
131.85
130.01
129.96
128.97
127.84
127.66
126.52
125.05
123.01
99.33
77.29
77.03
76.78
64.34
57.06
48.93
43.33
32.81
25.44
24.83
24.75
O
N
H
O
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

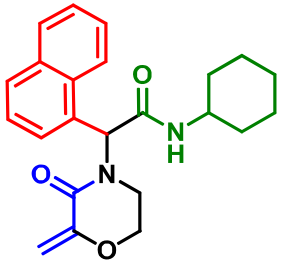
^{13}C NMR spectrum of **6o** (100 MHz, $CDCl_3$)

SAIF [HRMS Report]

Data File:	HRMS21I23MAR12	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-29R	Sample Name:	
Acquisition Date:	03/23/21 11:52:38 AM	Run Time(min):	0.00
Vial:	CStk1-01:12	Injection Volume(ul):	1.00

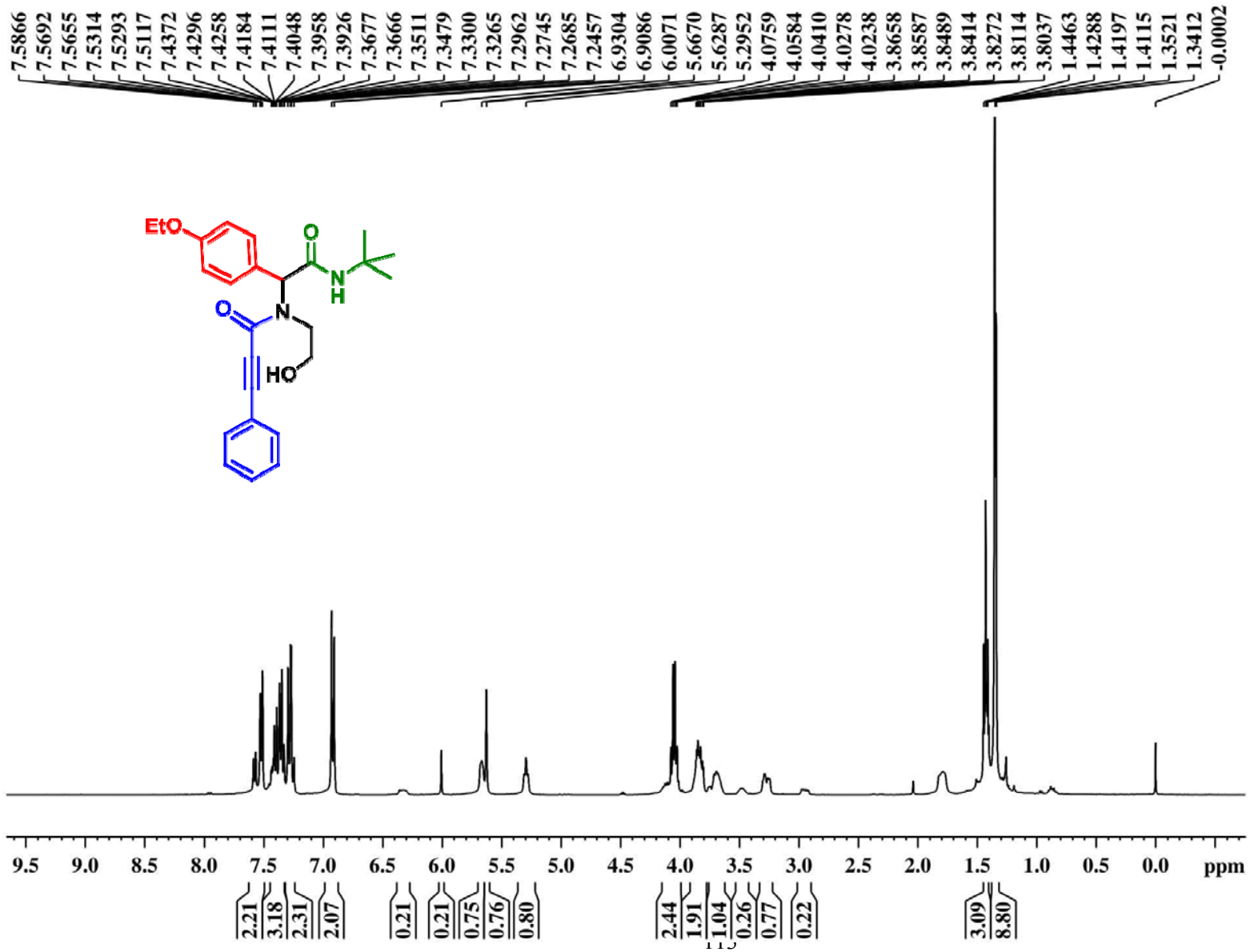
HRMS21I23MAR12 #32-63 RT: 0.25-0.50 AV: 32 SB: 1 0.00 NL: 1.23E5
T: FTMS + p ESI Full ms [150.00-1000.00]



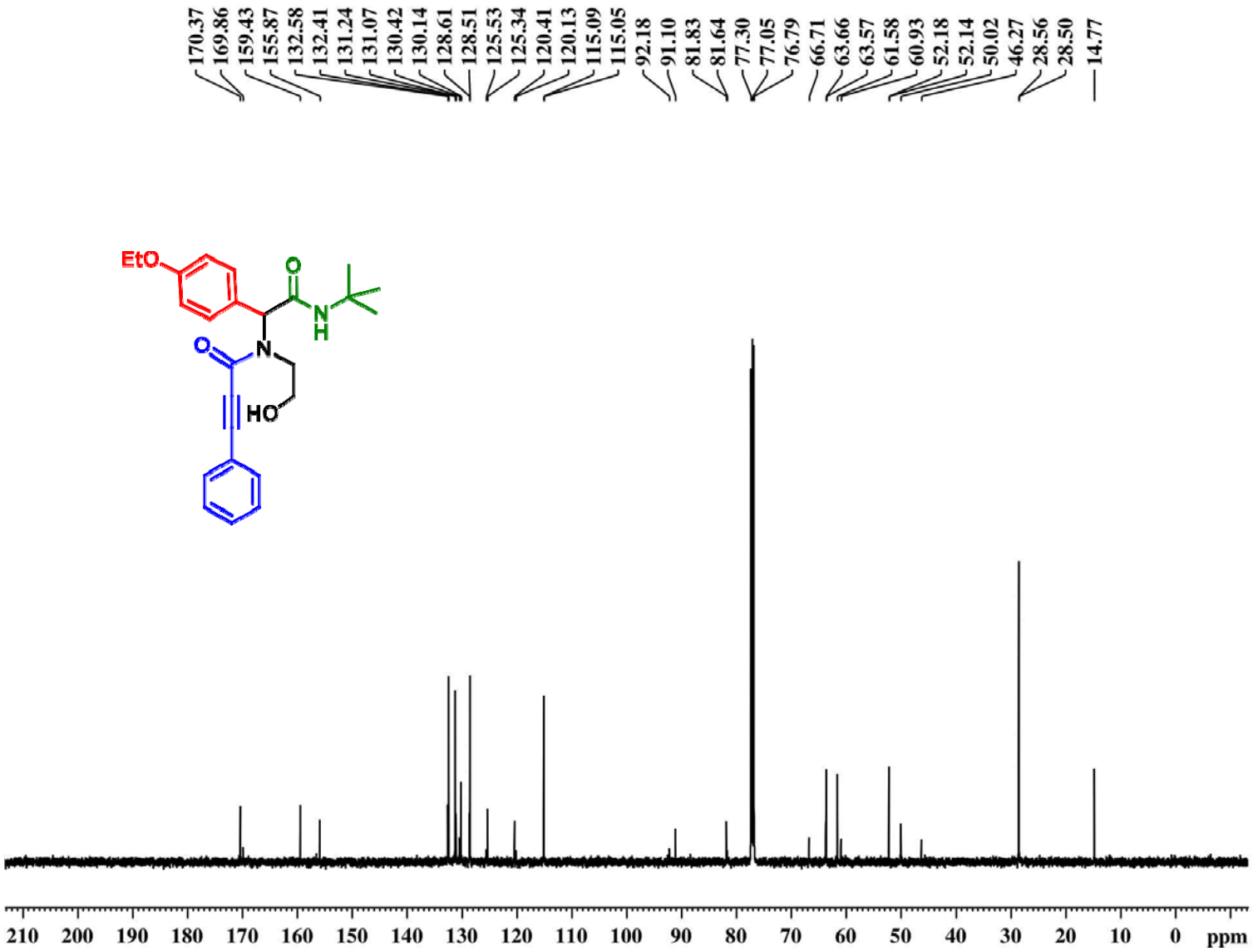


m/z:$[M+2H]^{2+}$Calcd for $C_{23}H_{28}N_2O_3$: 380.2049; Found: 380.2051.

Spectral data of **5a-5o**


7.5866
7.5692
7.5655
7.5314
7.5293
7.5117
7.4372
7.4296
7.4258
7.4184
7.4111
7.4048
7.3958
7.3926
7.3677
7.3666
7.3511
7.3479
7.3300
7.3265
7.2962
7.2745
7.2685
7.2457
6.9304
6.9086
6.0071
5.6670
5.6287
5.2952
4.0759
4.0584
4.0410
4.0278
4.0238
3.8658
3.8587
3.8489
3.8414
3.8272
3.8114
3.8037
1.4463
1.4288
1.4197
1.4115
1.3521
1.3412
-0.0002
EtO
O
N
H
O
N
HO
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
ppm
2.21
3.18
2.31
2.07
0.21
0.21
0.75
0.76
0.80
2.44
1.91
1.04
0.26
0.77
0.22
3.09
8.80

^{1}H NMR spectrum of **5a** (400 MHz, $CDCl_3$)


170.37
169.86
159.43
155.87
132.58
132.41
131.24
131.07
130.42
130.14
128.61
128.51
125.53
125.34
120.41
120.13
115.09
115.05
92.18
91.10
81.83
81.64
77.30
77.05
76.79
66.71
63.66
63.57
61.58
60.93
52.18
52.14
50.02
46.27
28.56
28.50
14.77
EtO
O
N
H
O
N
HO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR spectrum of **5a** (100MHz,MHz,$CDCl_3$)

SAIF [HRMS Report]

Data File:	HRMS21I23MAR08	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	PKM-M-22R	Sample Name:	
Acquisition Date:	03/23/21 11:44:45 AM	Run Time(min):	0.00
Vial:	CStk1-01:8	Injection Volume(µl):	1.00

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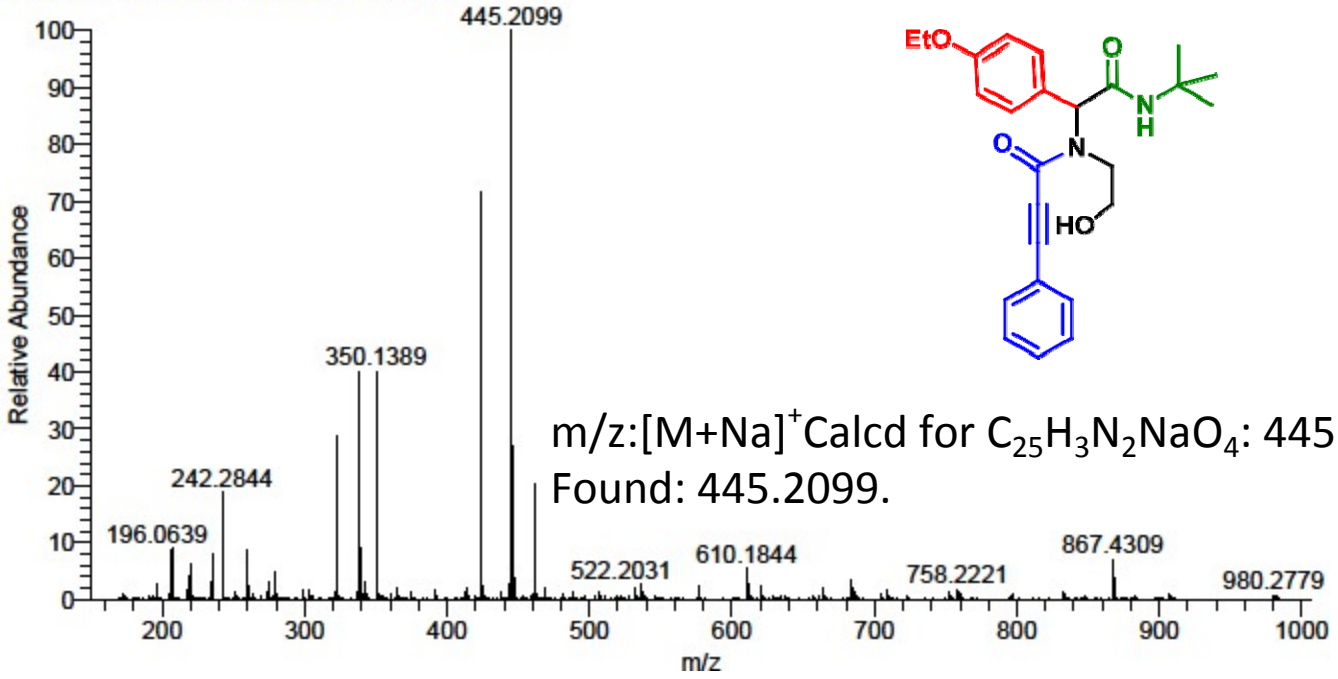
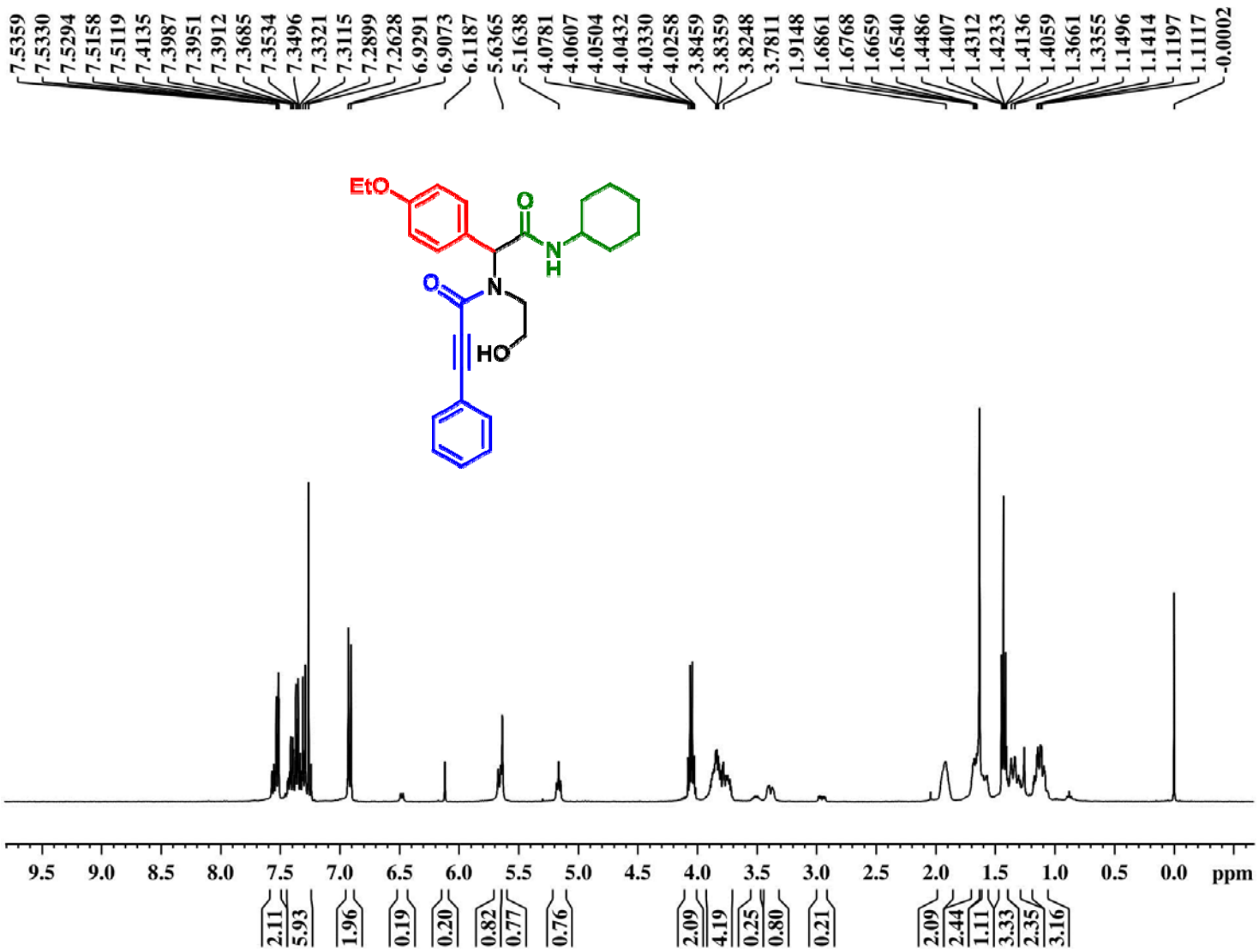
T: FTMS + p ESI Full ms [150.00-1000.00]



m/z:$[M+Na]^+$Calcd for $C_{25}H_3N_2NaO_4$: 445.2098; Found: 445.2099.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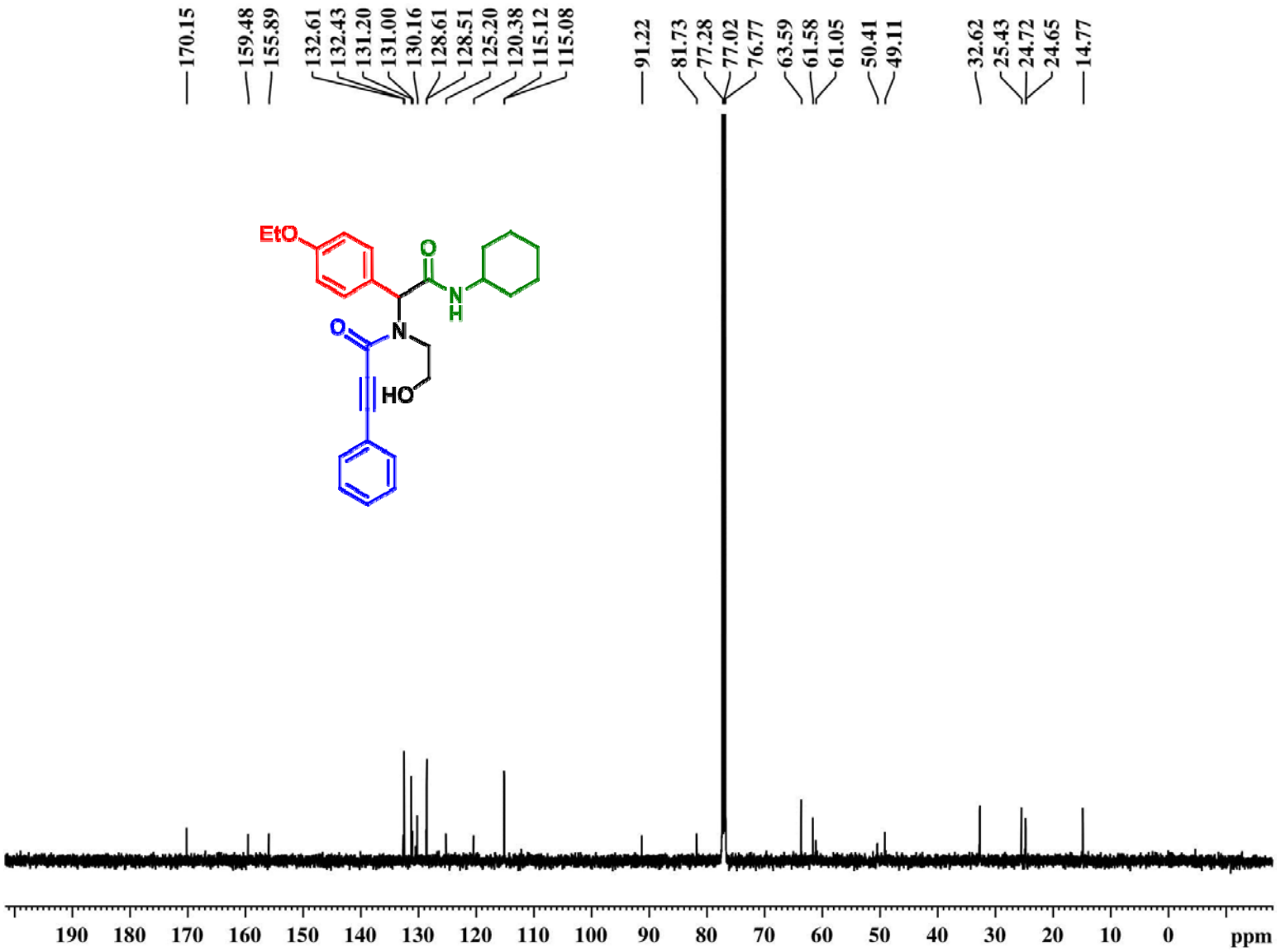
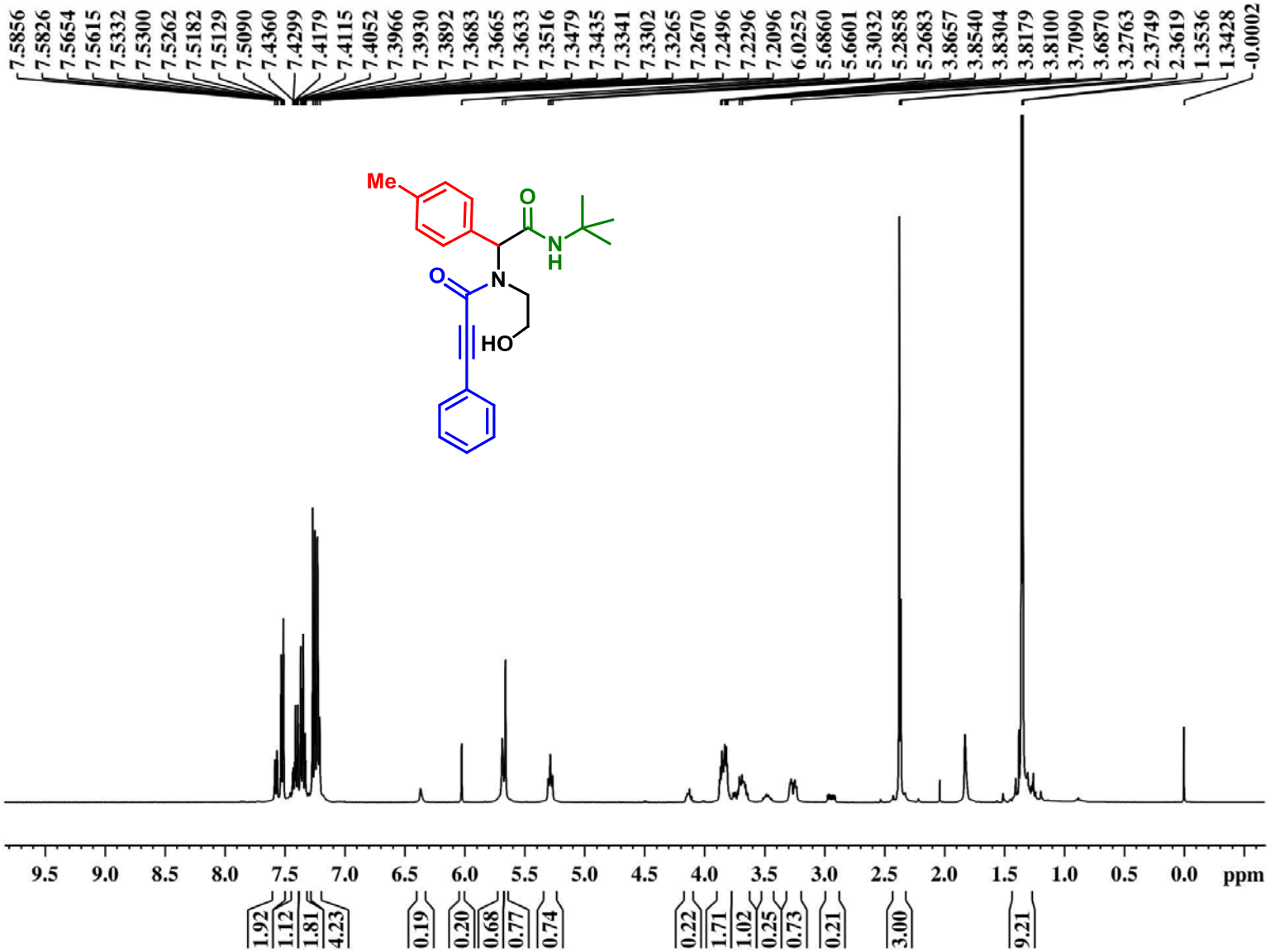
7.5158
7.5119
7.4135
7.3987
7.3951
7.3912
7.3685
7.3534
7.3496
7.3321
7.3115
7.2899
7.2628
6.9291
6.9073
6.1187
5.6365
5.1638
4.0781
4.0607
4.0504
4.0432
4.0330
4.0258
3.8459
3.8359
3.8248
3.7811
1.9148
1.6861
1.6768
1.6659
1.6540
1.4486
1.4407
1.4312
1.4233
1.4136
1.4059
1.3661
1.3355
1.1496
1.1414
1.1197
1.1117
-0.0002
EtO
O
N
H
O
N
HO
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 ppm
2.11 5.93 1.96 0.19 0.20 0.82 0.77 0.76 2.09 4.19 0.25 0.80 0.21 2.09 2.44 1.11 3.33 2.35 3.16

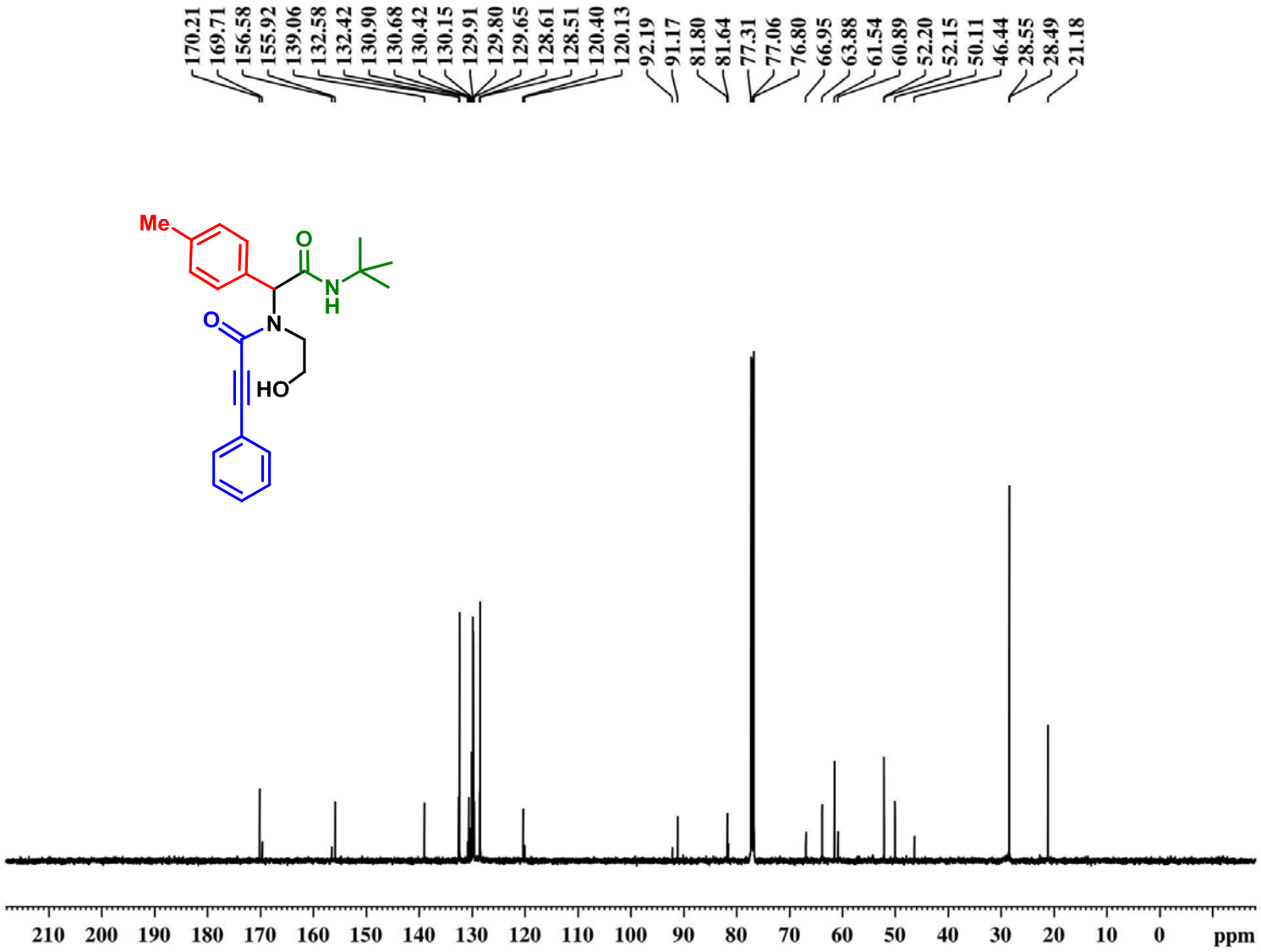
^{1}H NMR spectrum of **5b** (400MHz, $CDCl_3$)


170.15
159.48
155.89
132.61
132.43
131.20
131.00
130.16
128.61
128.51
125.20
120.38
115.12
115.08
91.22
81.73
77.28
77.02
76.77
63.59
61.58
61.05
50.41
49.11
32.62
25.43
24.72
24.65
14.77
EtO
O
N
H
O
N
HO
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

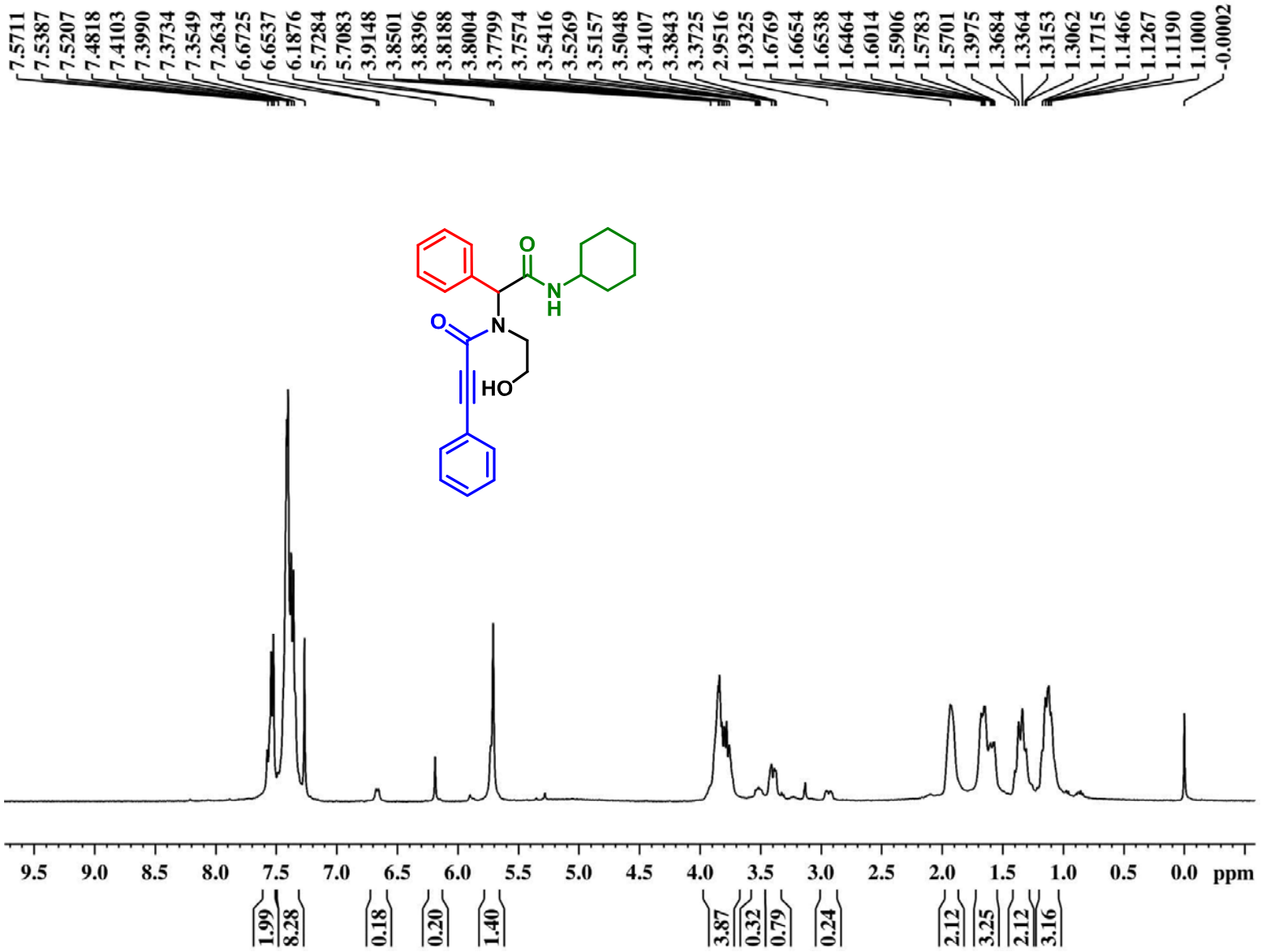
^{13}C NMR spectrum of **5b** (100MHz, $CDCl_3$)


7.5856
7.5826
7.5654
7.5615
7.5332
7.5300
7.5262
7.5182
7.5129
7.5090
7.4360
7.4299
7.4179
7.4115
7.4052
7.3966
7.3930
7.3892
7.3683
7.3665
7.3633
7.3516
7.3479
7.3435
7.3341
7.3302
7.3265
7.2670
7.2496
7.2296
7.2096
6.0252
5.6860
5.6601
5.3032
5.2858
5.2683
3.8657
3.8540
3.8304
3.8179
3.8100
3.7090
3.6870
3.2763
2.3749
2.3619
1.3536
1.3428
-0.0002
Me
O
N
H
O
N
HO
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
ppm
1.92
1.12
1.81
4.23
0.19
0.20
0.68
0.77
0.74
0.22
1.71
1.02
0.25
0.73
0.21
3.00
9.21

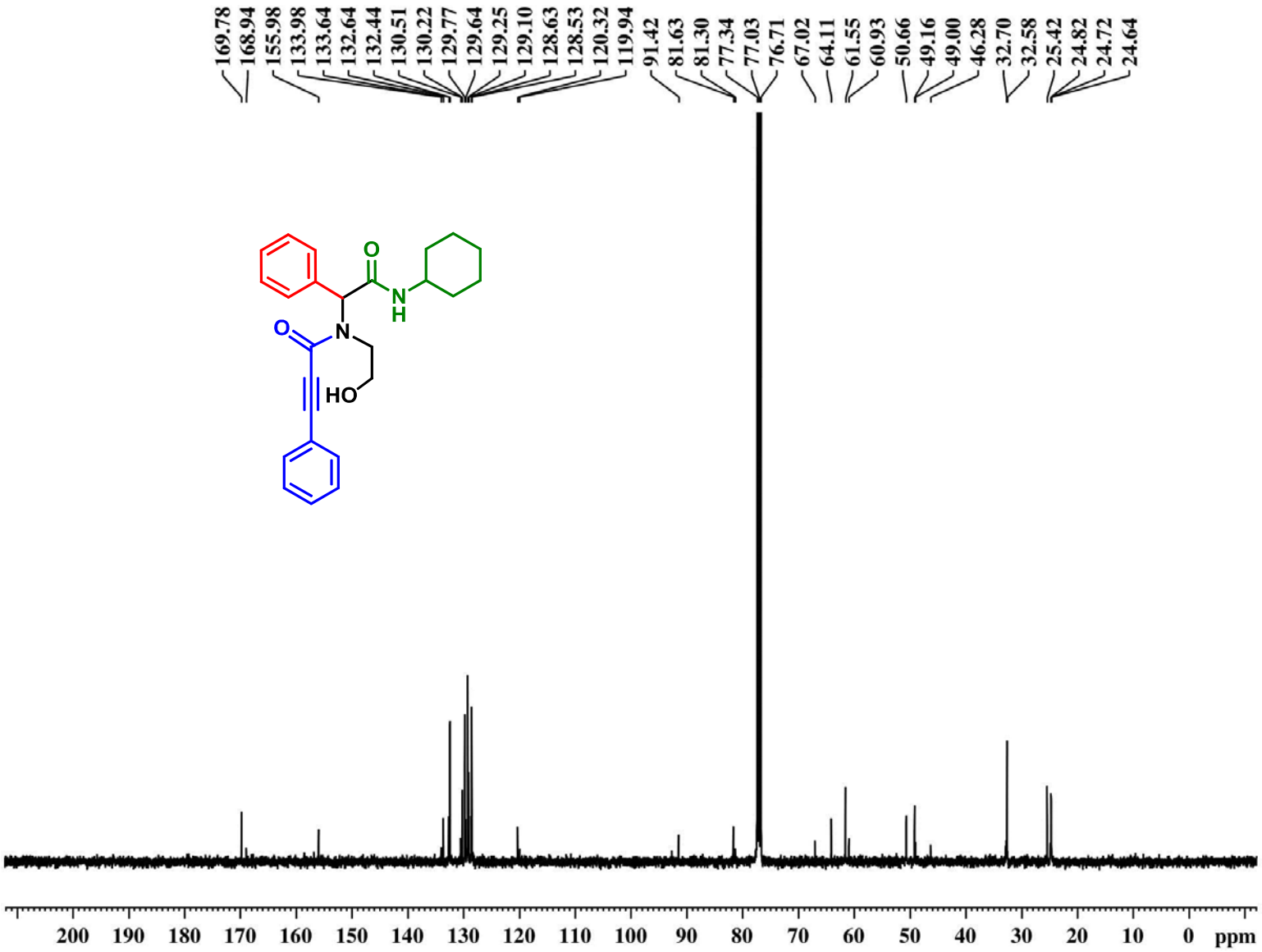
^{1}H NMR spectrum of **5c** (400 MHz, $CDCl_3$)


170.21
169.71
156.58
155.92
139.06
132.58
132.42
130.90
130.68
130.42
130.15
129.91
129.80
129.65
128.61
128.51
120.40
120.13
92.19
91.17
81.80
81.64
77.31
77.06
76.80
66.95
63.88
61.54
60.89
52.20
52.15
50.11
46.44
28.55
28.49
21.18
Me
O
N
H
O
N
HO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

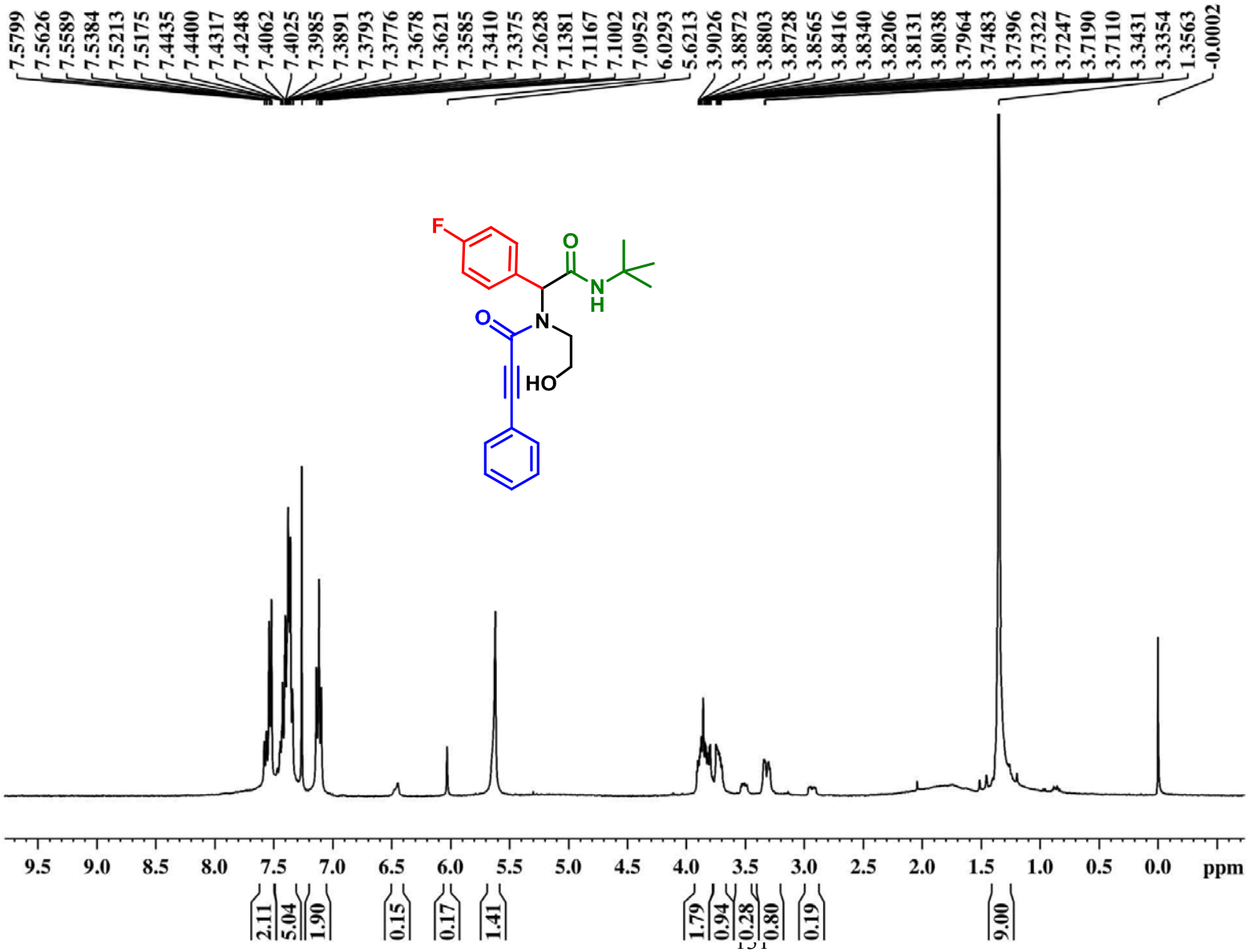
^{13}C NMR spectrum of **5c** (100 MHz, $CDCl_3$)


7.5711
7.5387
7.5207
7.4818
7.4103
7.3990
7.3734
7.3549
7.2634
6.6725
6.6537
6.1876
5.7284
5.7083
3.9148
3.8501
3.8396
3.8188
3.8004
3.7799
3.7574
3.5416
3.5269
3.5157
3.5048
3.4107
3.3843
3.3725
2.9516
1.9325
1.6769
1.6654
1.6538
1.6464
1.6014
1.5906
1.5783
1.5701
1.3975
1.3684
1.3364
1.3153
1.3062
1.1715
1.1466
1.1267
1.1190
1.1000
-0.0002
O
N
H
O
N
HO
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 ppm
1.99
8.28
0.18
0.20
1.40
3.87
0.32
0.79
0.24
2.12
3.25
2.12
3.16

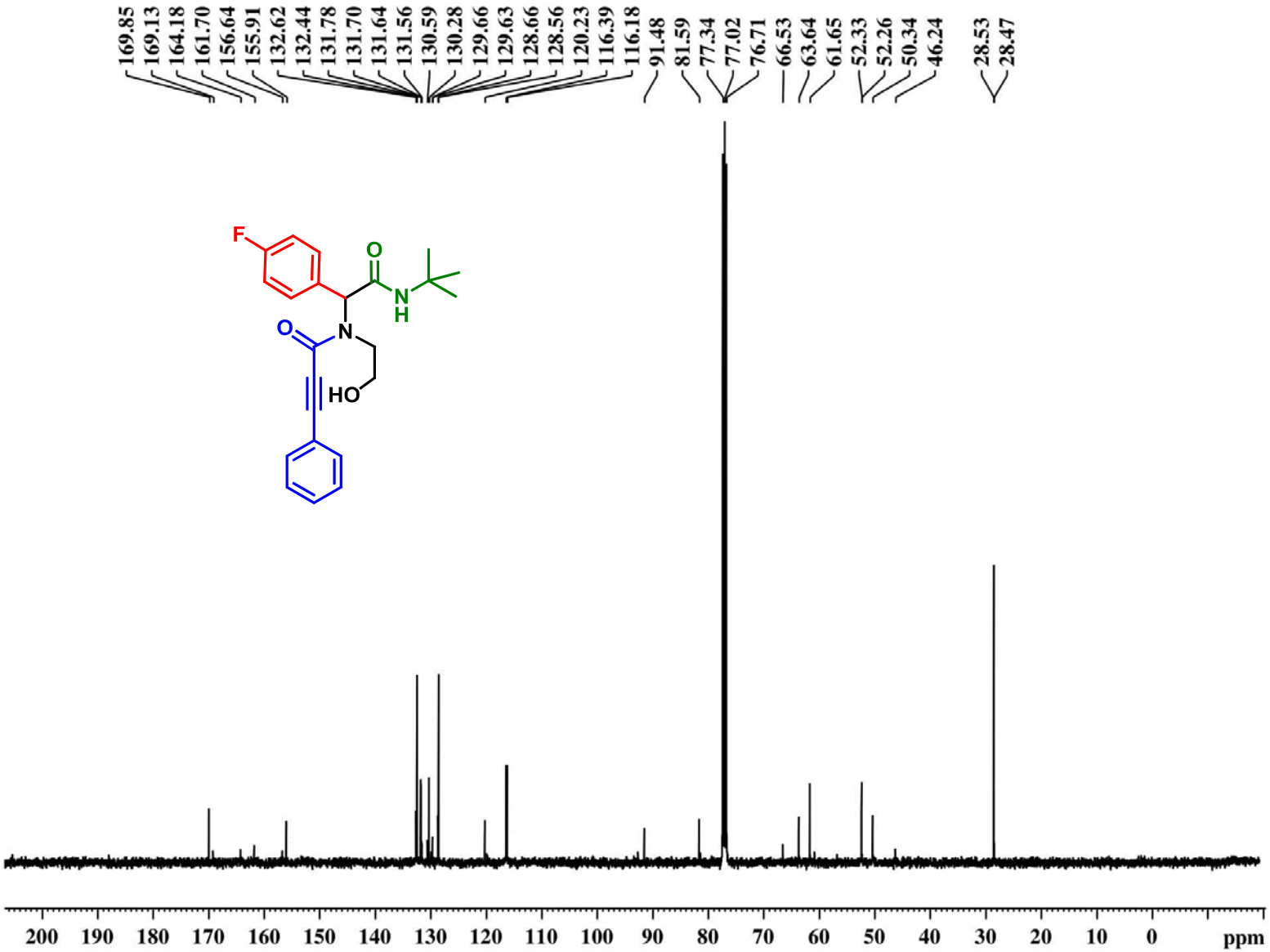
^{1}H NMR spectrum of **5d** (400MHz, $CDCl_3$)


169.78
168.94
155.98
133.98
133.64
132.64
132.44
130.51
130.22
129.77
129.64
129.25
129.10
128.63
128.53
120.32
119.94
91.42
81.63
81.30
77.34
77.03
76.71
67.02
64.11
61.55
60.93
50.66
49.16
49.00
46.28
32.70
32.58
25.42
24.82
24.72
24.64
O
N
H
O
N
HO
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

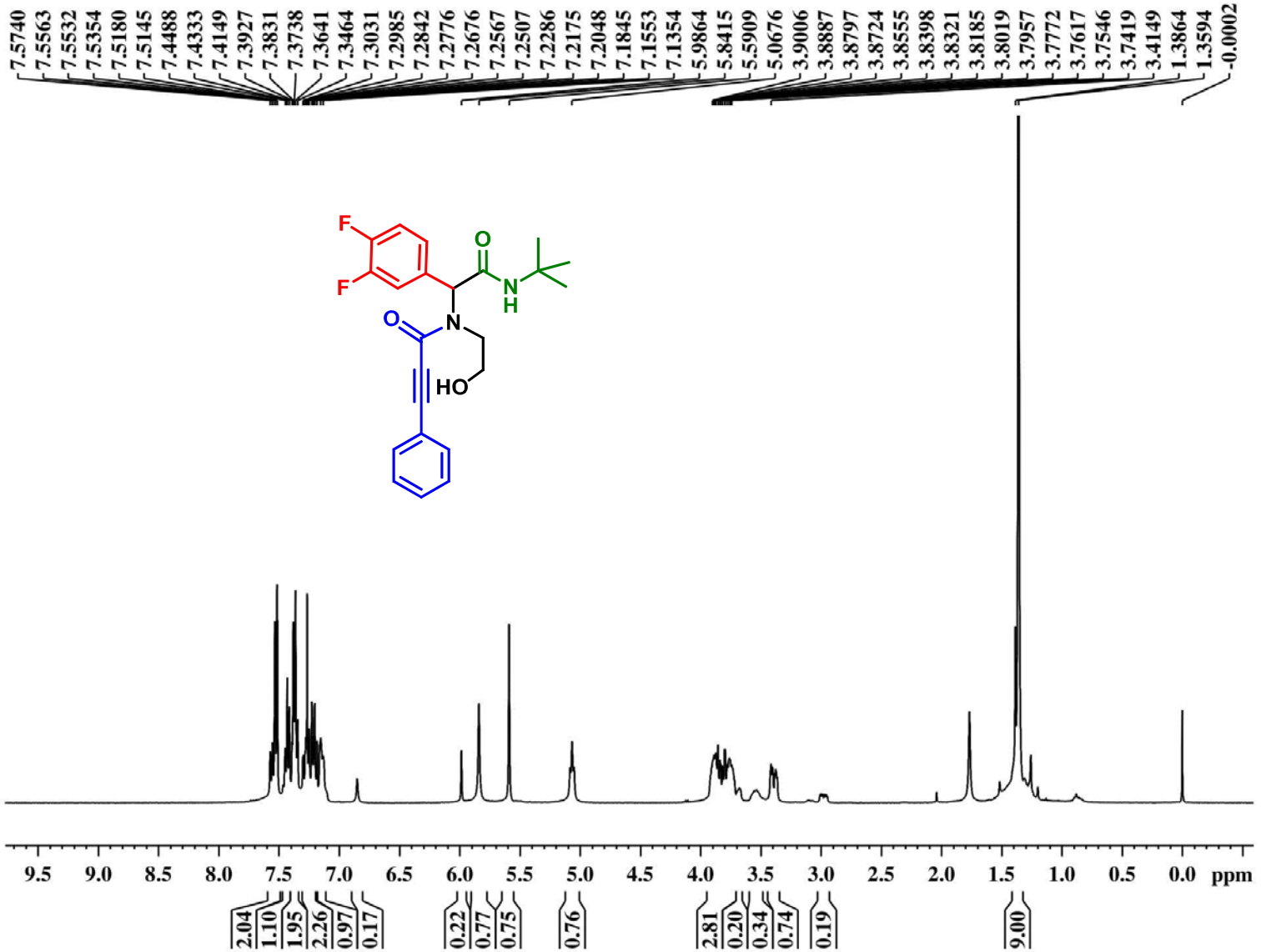
^{13}C NMR spectrum of **5d** (100MHz, $CDCl_3$)


7.5799
7.5626
7.5589
7.5384
7.5213
7.5175
7.4435
7.4400
7.4317
7.4248
7.4062
7.4025
7.3985
7.3891
7.3793
7.3776
7.3678
7.3621
7.3585
7.3410
7.3375
7.2628
7.1381
7.1167
7.1002
7.0952
6.0293
5.6213
3.9026
3.8872
3.8803
3.8728
3.8565
3.8416
3.8340
3.8206
3.8131
3.8038
3.7964
3.7483
3.7396
3.7322
3.7247
3.7190
3.7110
3.3431
3.3354
1.3563
-0.0002
F
O
N
H
O
N
HO
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
ppm
2.11
5.04
1.90
0.15
0.17
1.41
1.79
0.94
0.28
0.80
0.19
9.00

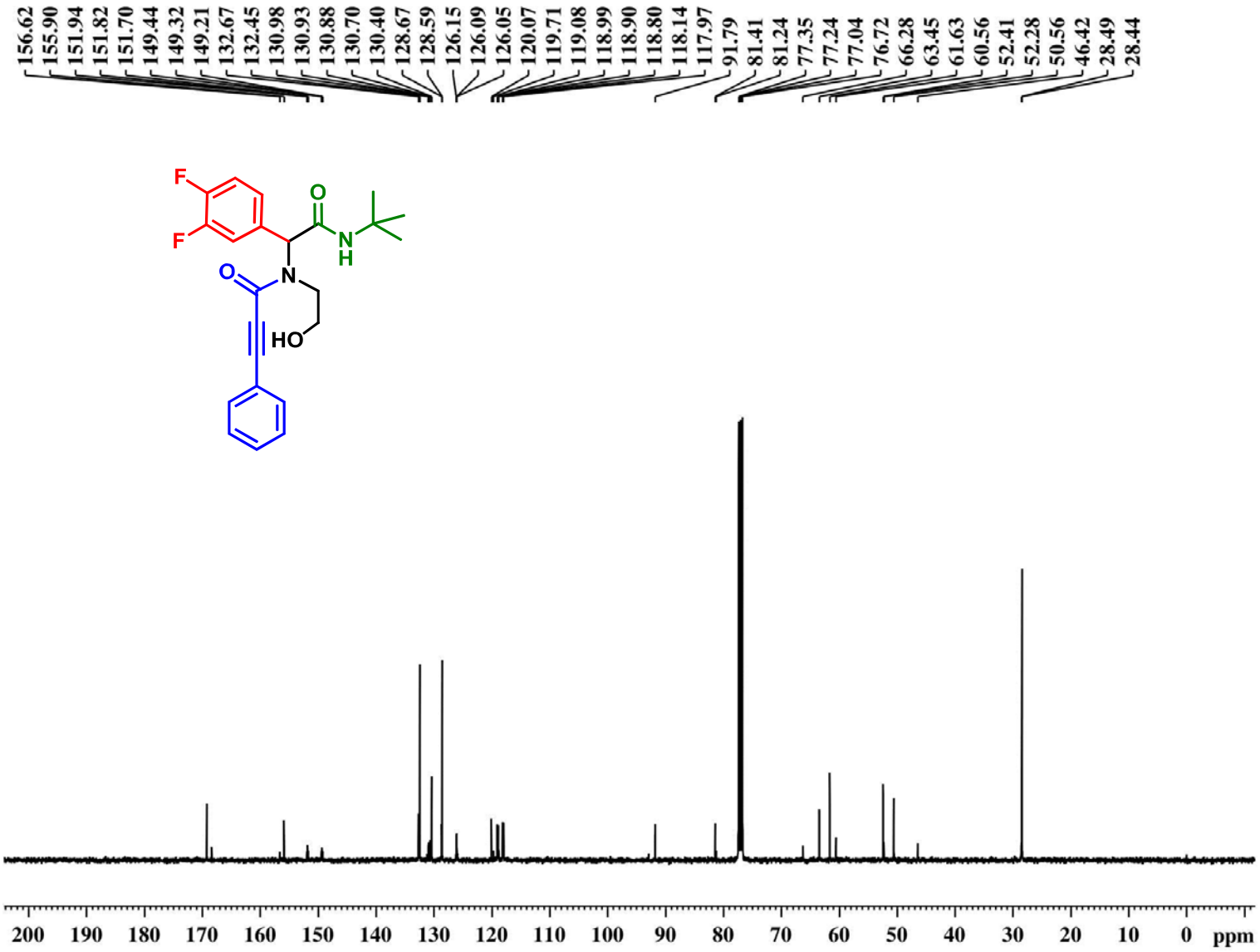
^{1}H NMR spectrum of **5e** (400 MHz, $CDCl_3$)


169.85
169.13
164.18
161.70
156.64
155.91
132.62
132.44
131.78
131.70
131.64
131.56
130.59
130.28
129.66
129.63
128.66
128.56
120.23
116.39
116.18
91.48
81.59
77.34
77.02
76.71
66.53
63.64
61.65
52.33
52.26
50.34
46.24
28.53
28.47
F
O
N
H
O
N
HO
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

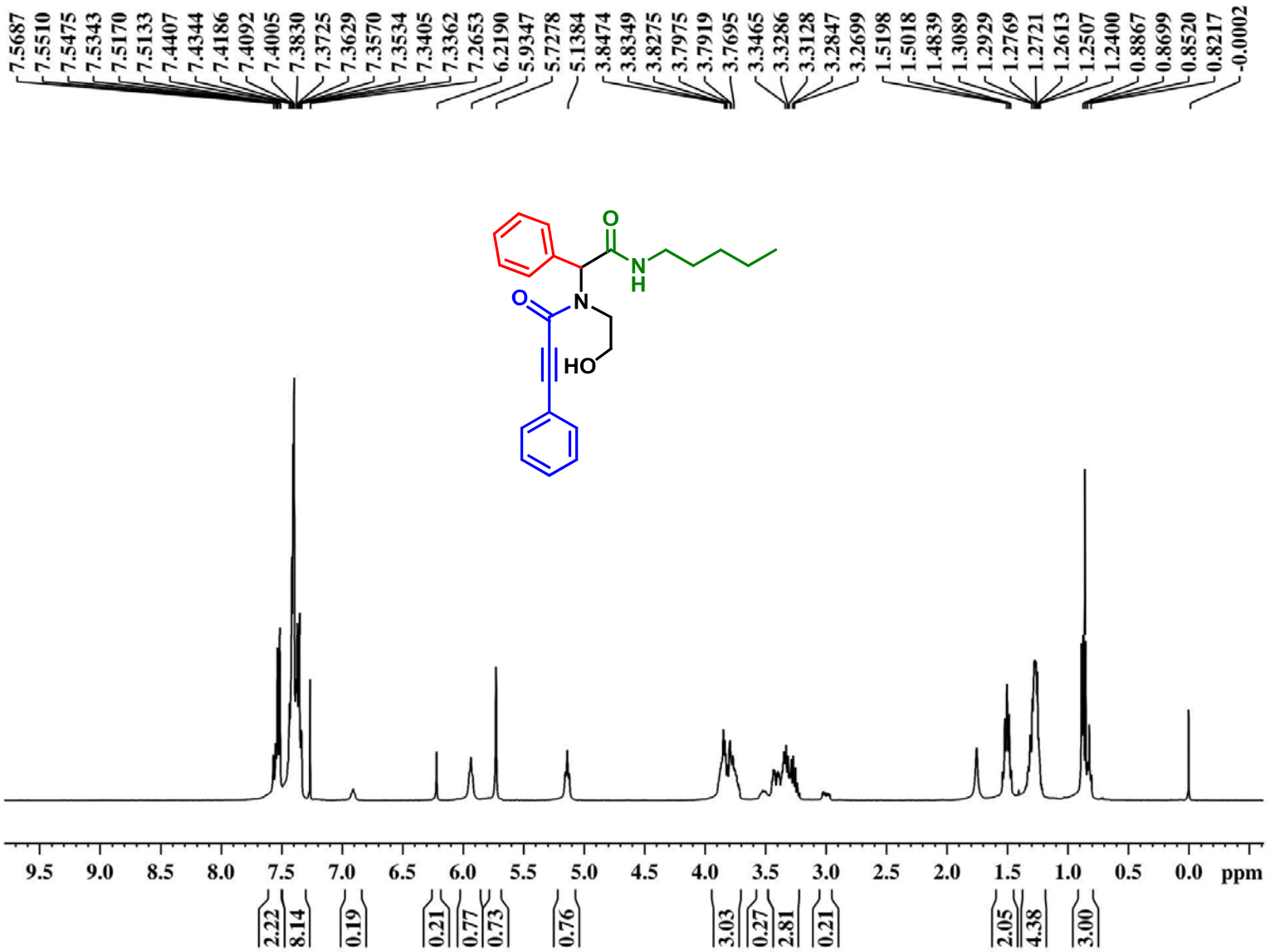
^{13}C NMR spectrum of **5e** (100MHz, $CDCl_3$)


7.5740
7.5563
7.5532
7.5354
7.5180
7.5145
7.4488
7.4333
7.4149
7.3927
7.3831
7.3738
7.3641
7.3464
7.3031
7.2985
7.2842
7.2776
7.2676
7.2567
7.2507
7.2286
7.2175
7.2048
7.1845
7.1553
7.1354
5.9864
5.8415
5.5909
5.0676
3.9006
3.8887
3.8797
3.8724
3.8555
3.8398
3.8321
3.8185
3.8019
3.7957
3.7772
3.7617
3.7546
3.7419
3.4149
1.3864
1.3594
-0.0002
F
F
O
N
H
O
N
HO
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
ppm
2.04
1.10
1.95
2.26
0.97
0.17
0.22
0.77
0.75
0.76
2.81
0.20
0.34
0.74
0.19
9.00

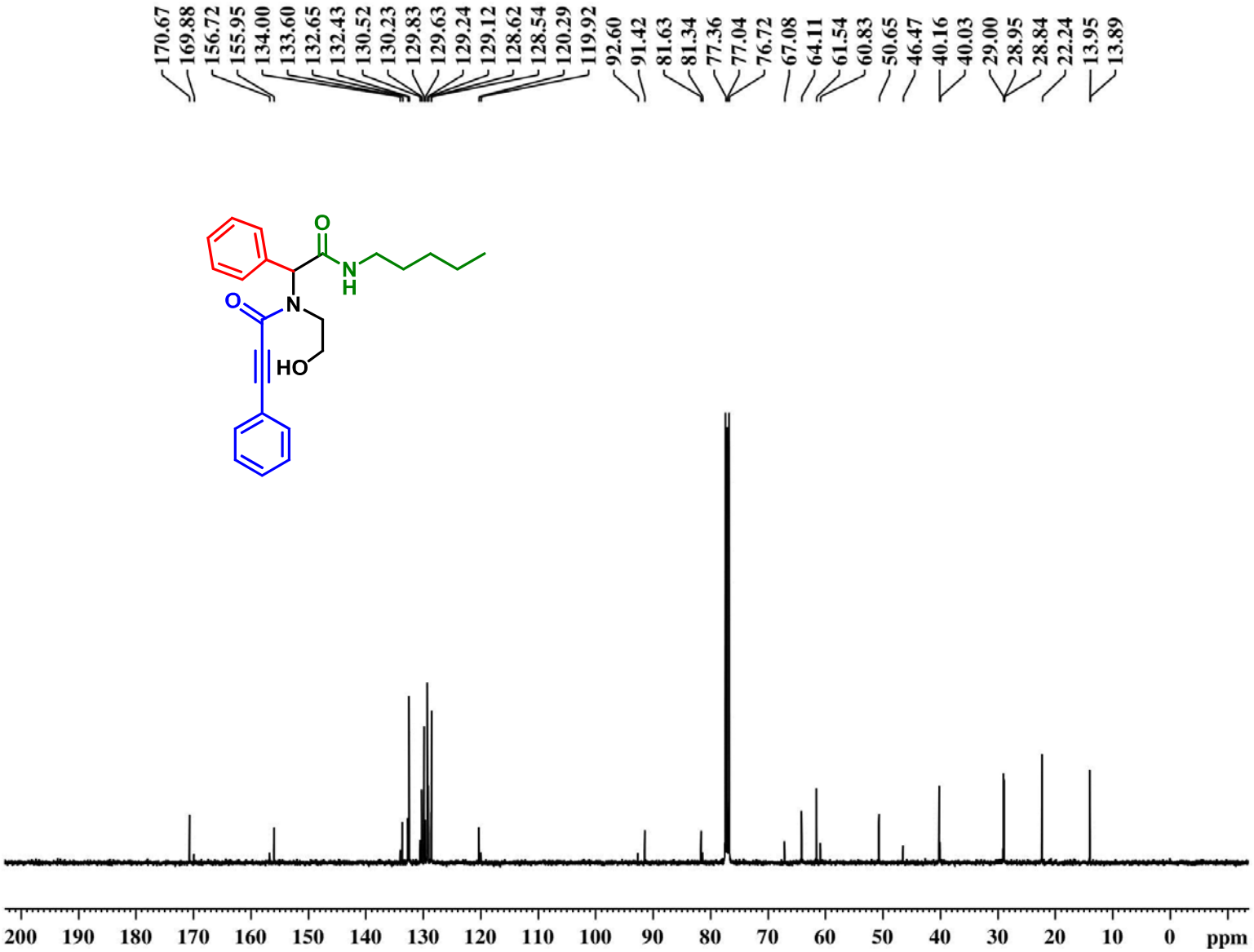
^{1}H NMR spectrum of **5f** (400 MHz, $CDCl_3$)


156.62
155.90
151.94
151.82
151.70
149.44
149.32
149.21
132.67
132.45
130.98
130.93
130.88
130.70
130.40
128.67
128.59
126.15
126.09
126.05
120.07
119.71
119.08
118.99
118.90
118.80
118.14
117.97
91.79
81.41
81.24
77.35
77.24
77.04
76.72
66.28
63.45
61.63
60.56
52.41
52.28
50.56
46.42
28.49
28.44
F
F
O
N
H
O
N
HO
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

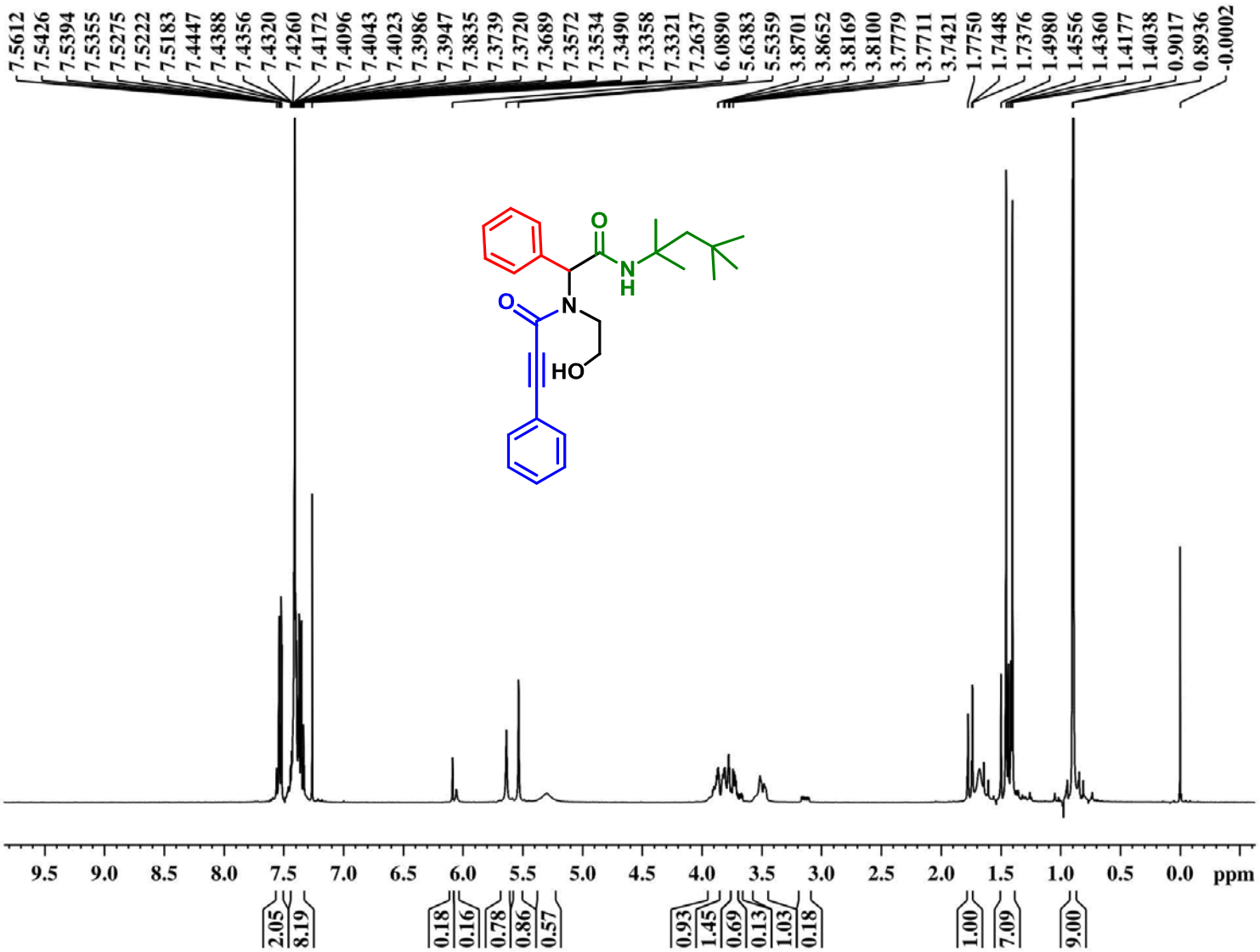
^{13}C NMR spectrum of **5f** (100MHz, $CDCl_3$)


7.5687
7.5510
7.5475
7.5343
7.5170
7.5133
7.4407
7.4344
7.4186
7.4092
7.4005
7.3830
7.3725
7.3629
7.3570
7.3534
7.3405
7.3362
7.2653
6.2190
5.9347
5.7278
5.1384
3.8474
3.8349
3.8275
3.7975
3.7919
3.7695
3.3465
3.3286
3.3128
3.2847
3.2699
1.5198
1.5018
1.4839
1.3089
1.2929
1.2769
1.2721
1.2613
1.2507
1.2400
0.8867
0.8699
0.8520
0.8217
-0.0002
O
N
H
O
N
HO
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
ppm
2.22
8.14
0.19
0.21
0.77
0.73
0.76
3.03
0.27
2.81
0.21
2.05
4.38
3.00

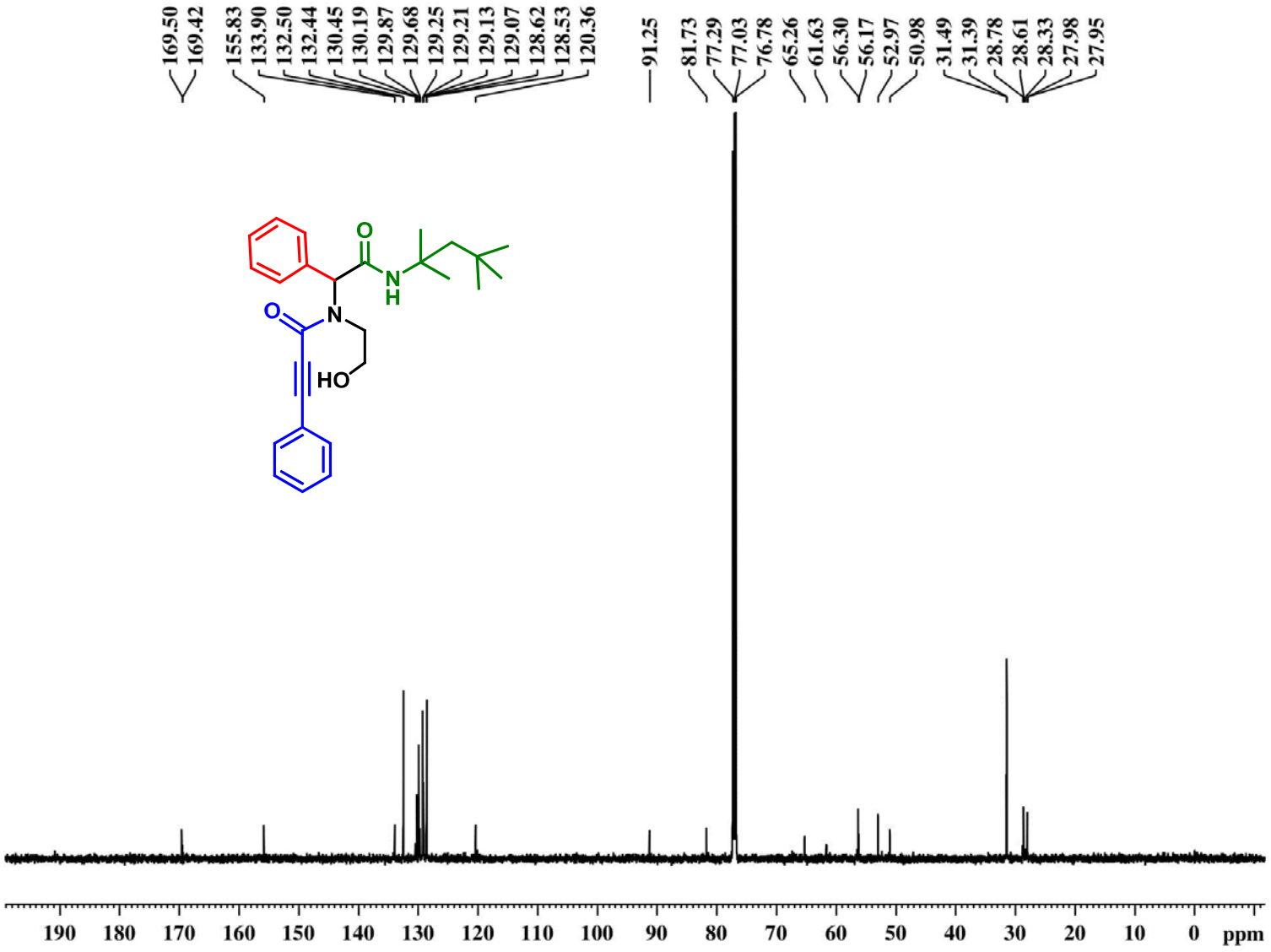
^{1}H NMR spectrum of **5g** (400 MHz, $CDCl_3$)


170.67
169.88
156.72
155.95
134.00
133.60
132.65
132.43
130.52
130.23
129.83
129.63
129.24
129.12
128.62
128.54
120.29
119.92
92.60
91.42
81.63
81.34
77.36
77.04
76.72
67.08
64.11
61.54
60.83
50.65
46.47
40.16
40.03
29.00
28.95
28.84
22.24
13.95
13.89
O
N
H
O
N
HO
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

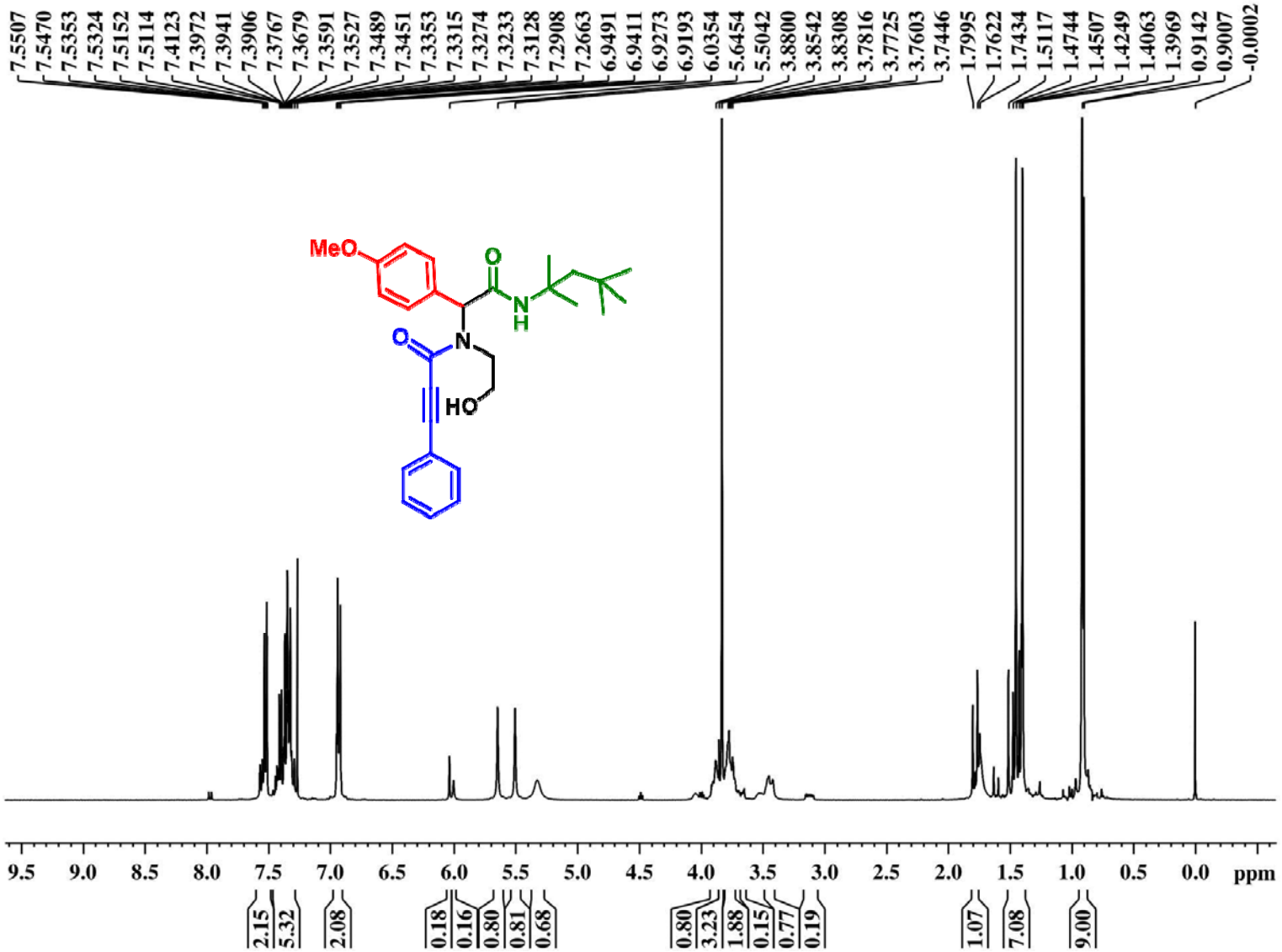
^{13}C NMR spectrum of **5g** (100MHz, $CDCl_3$)


7.5612
7.5426
7.5394
7.5355
7.5275
7.5222
7.5183
7.4447
7.4388
7.4356
7.4320
7.4260
7.4172
7.4096
7.4043
7.4023
7.3986
7.3947
7.3835
7.3739
7.3720
7.3689
7.3572
7.3534
7.3490
7.3358
7.3321
7.2637
6.0890
5.6383
5.5359
3.8701
3.8652
3.8169
3.8100
3.7779
3.7711
3.7421
1.7750
1.7448
1.7376
1.4980
1.4556
1.4360
1.4177
1.4038
0.9017
0.8936
-0.0002
O
N
H
O
N
HO
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
ppm
2.05
8.19
0.18
0.16
0.78
0.86
0.57
0.93
1.45
0.69
0.13
1.03
0.18
1.00
7.09
9.00

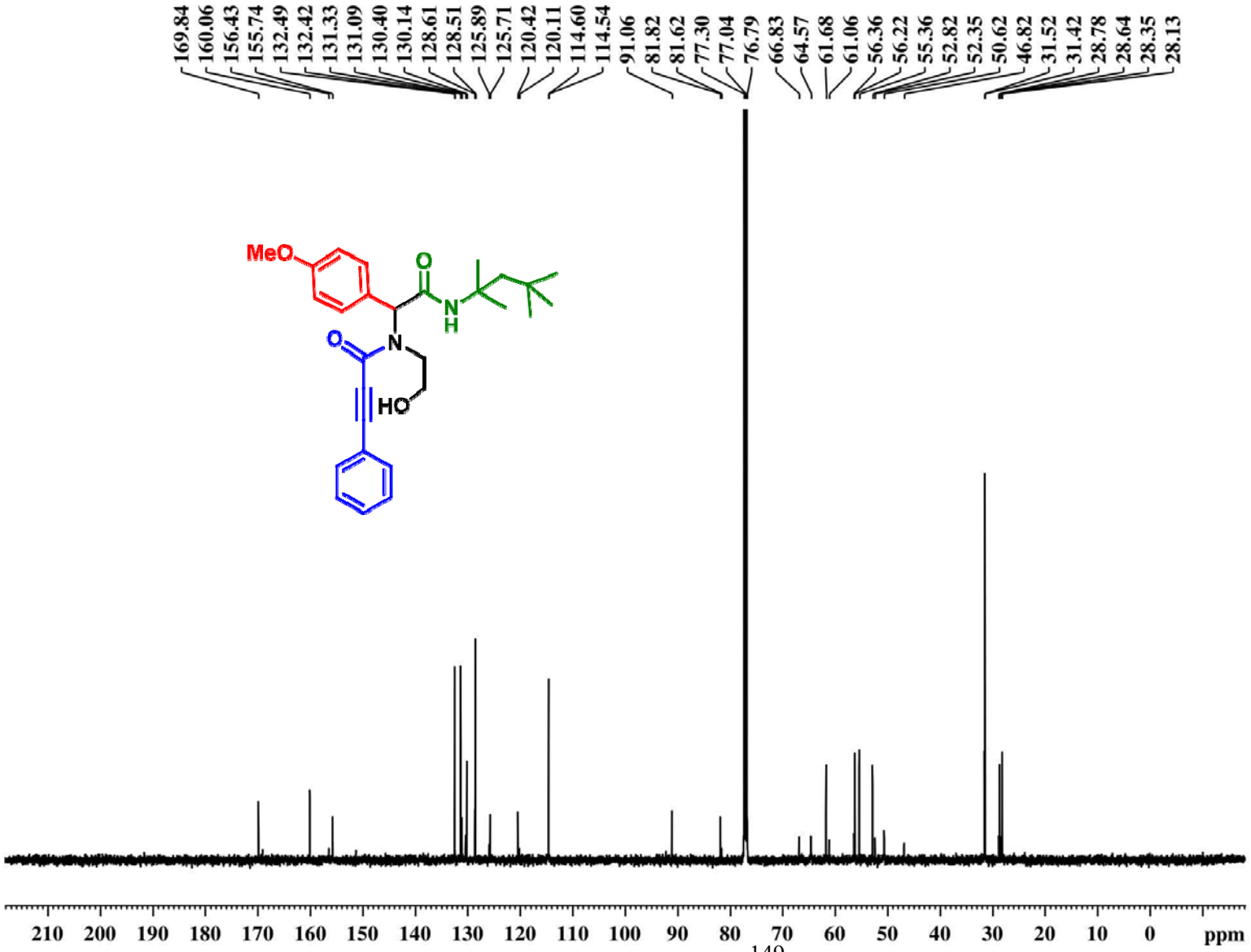
^{1}H NMR spectrum of **5h** (400MHz, $CDCl_3$)


169.50
169.42
155.83
133.90
132.50
132.44
130.45
130.19
129.87
129.68
129.25
129.21
129.13
129.07
128.62
128.53
120.36
91.25
81.73
77.29
77.03
76.78
65.26
61.63
56.30
56.17
52.97
50.98
31.49
31.39
28.78
28.61
28.33
27.98
27.95
O
N
H
O
N
HO
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

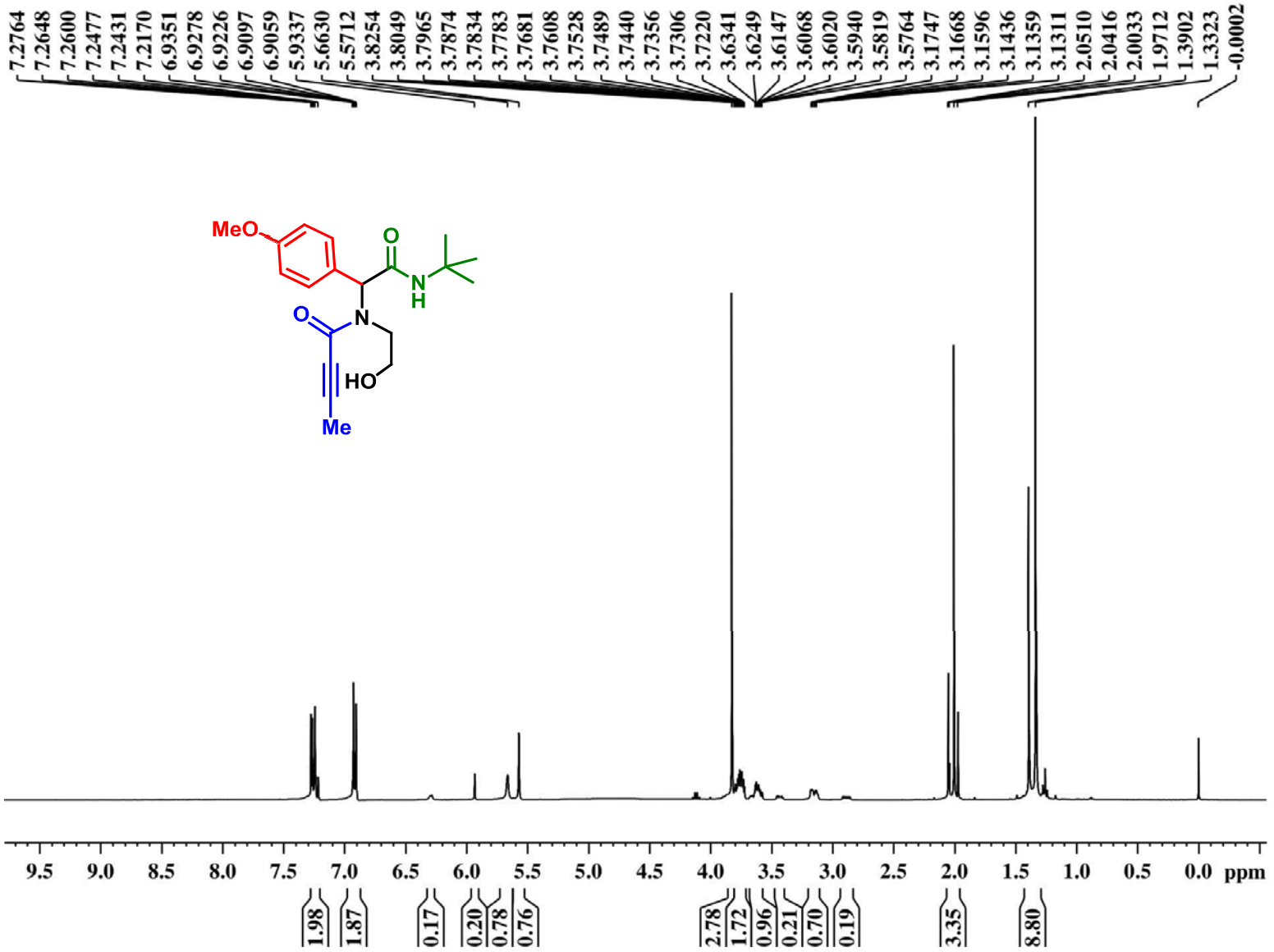
^{13}C NMR spectrum of **5h** (100MHz, $CDCl_3$)


7.5507
7.5470
7.5353
7.5324
7.5152
7.5114
7.4123
7.3972
7.3941
7.3906
7.3767
7.3679
7.3591
7.3527
7.3489
7.3451
7.3353
7.3315
7.3274
7.3233
7.3128
7.2908
7.2663
6.9491
6.9411
6.9273
6.9193
6.0354
5.6454
5.5042
3.8800
3.8542
3.8308
3.7816
3.7725
3.7603
3.7446
1.7995
1.7622
1.7434
1.5117
1.4744
1.4507
1.4249
1.4063
1.3969
0.9142
0.9007
-0.0002
MeO
O
N
H
O
N
HO
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
ppm
2.15
5.32
2.08
0.18
0.16
0.80
0.81
0.68
0.80
3.23
1.88
0.15
0.77
0.19
1.07
7.08
9.00

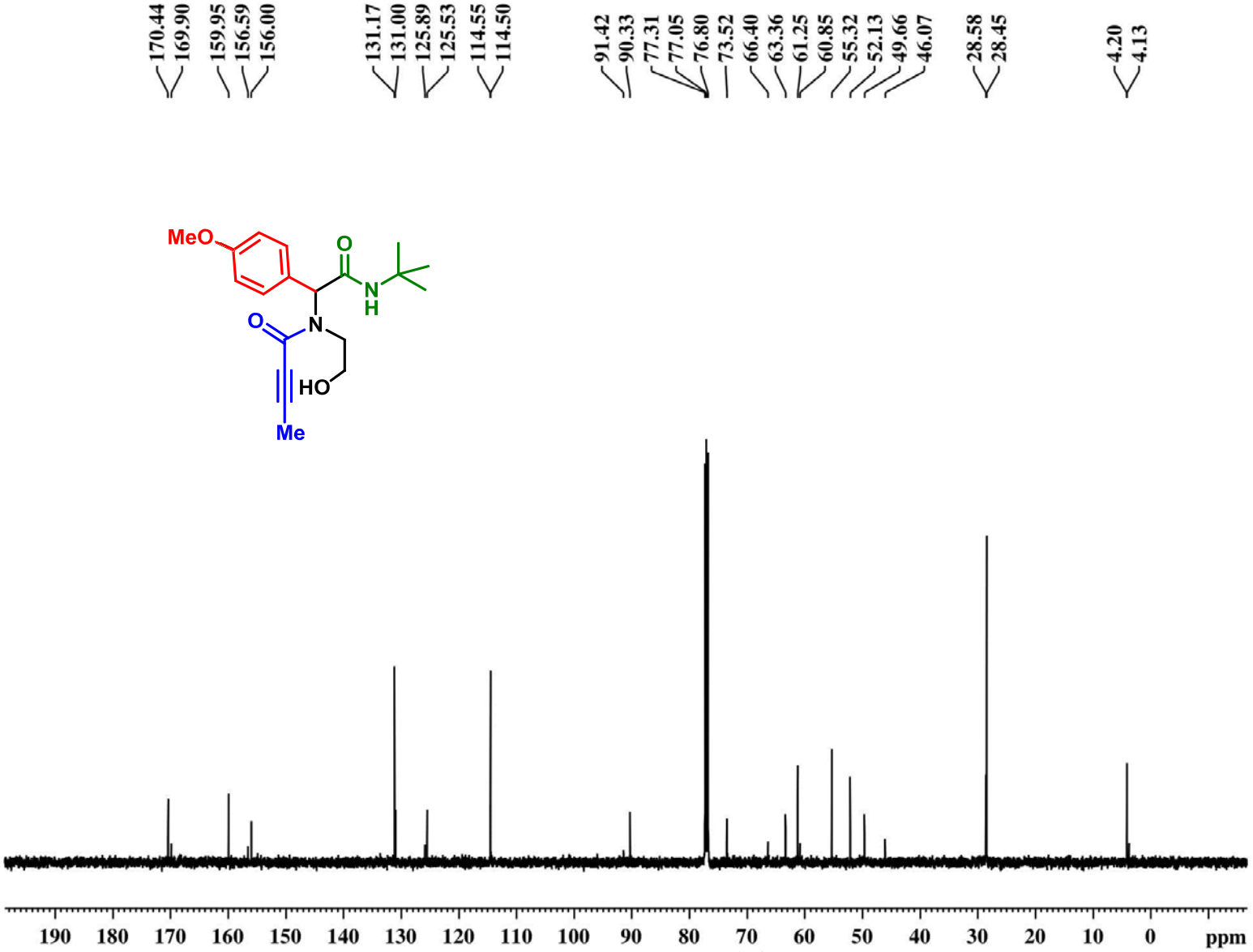
^{1}H NMR spectrum of **5i** (400 MHz, $CDCl_3$)


169.84
160.06
156.43
155.74
132.49
132.42
131.33
131.09
130.40
130.14
128.61
128.51
125.89
125.71
120.42
120.11
114.60
114.54
91.06
81.82
81.62
77.30
77.04
76.79
66.83
64.57
61.68
61.06
56.36
56.22
55.36
52.82
52.35
50.62
46.82
31.52
31.42
28.78
28.64
28.35
28.13
MeO
O
N
H
O
N
HO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

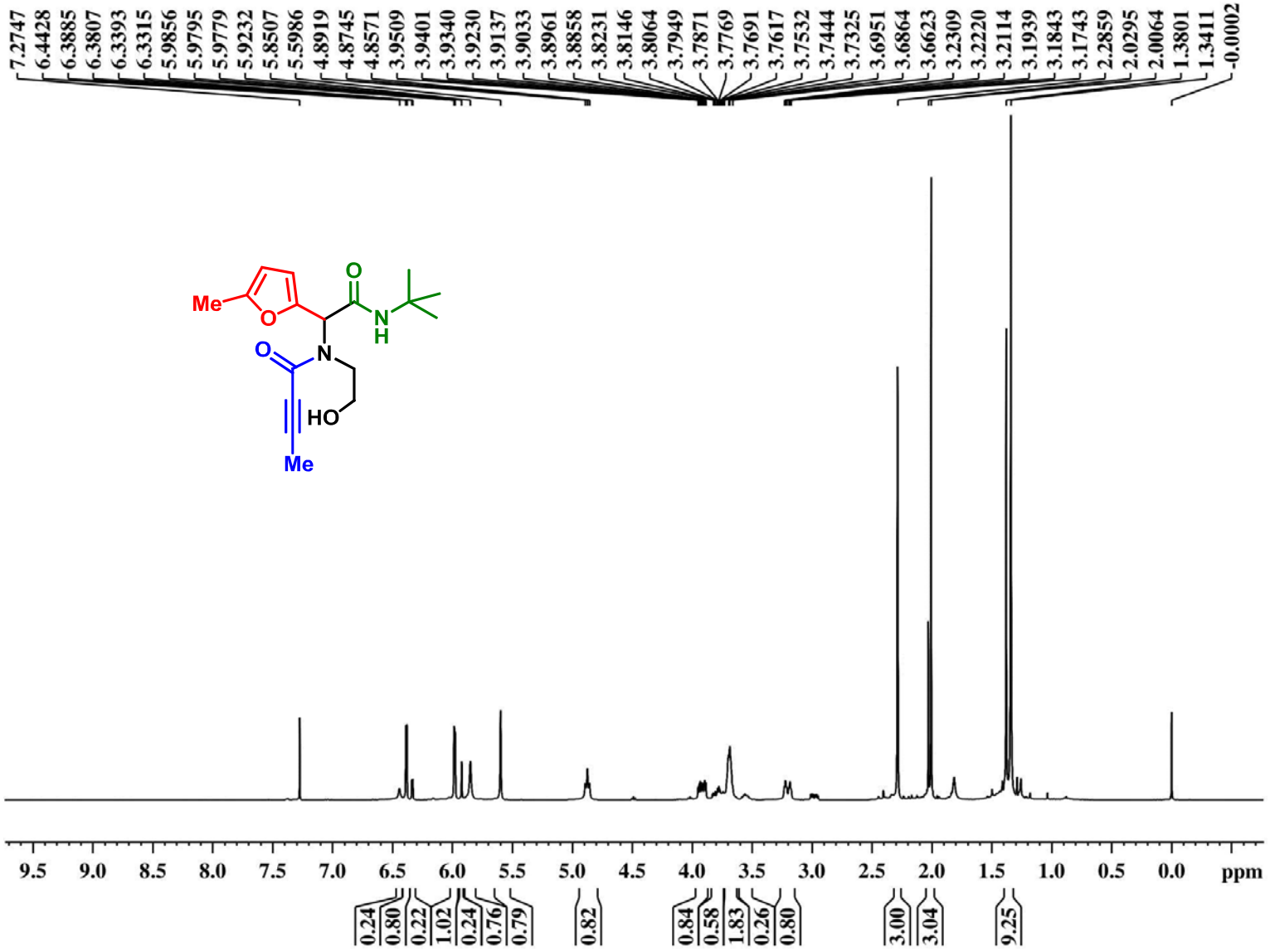
^{13}C NMR spectrum of **5i** (100MHz, $CDCl_3$)


7.2764
7.2648
7.2600
7.2477
7.2431
7.2170
6.9351
6.9278
6.9226
6.9097
6.9059
5.9337
5.6630
5.5712
3.8254
3.8049
3.7965
3.7874
3.7834
3.7783
3.7681
3.7608
3.7528
3.7489
3.7440
3.7356
3.7306
3.7220
3.6341
3.6249
3.6147
3.6068
3.6020
3.5940
3.5819
3.5764
3.1747
3.1668
3.1596
3.1436
3.1359
3.1311
2.0510
2.0416
2.0033
1.9712
1.3902
1.3323
-0.0002
MeO
O
N
H
O
N
HO
Me
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
ppm
1.98
1.87
0.17
0.20
0.78
0.76
2.78
1.72
0.96
0.21
0.70
0.19
3.35
8.80

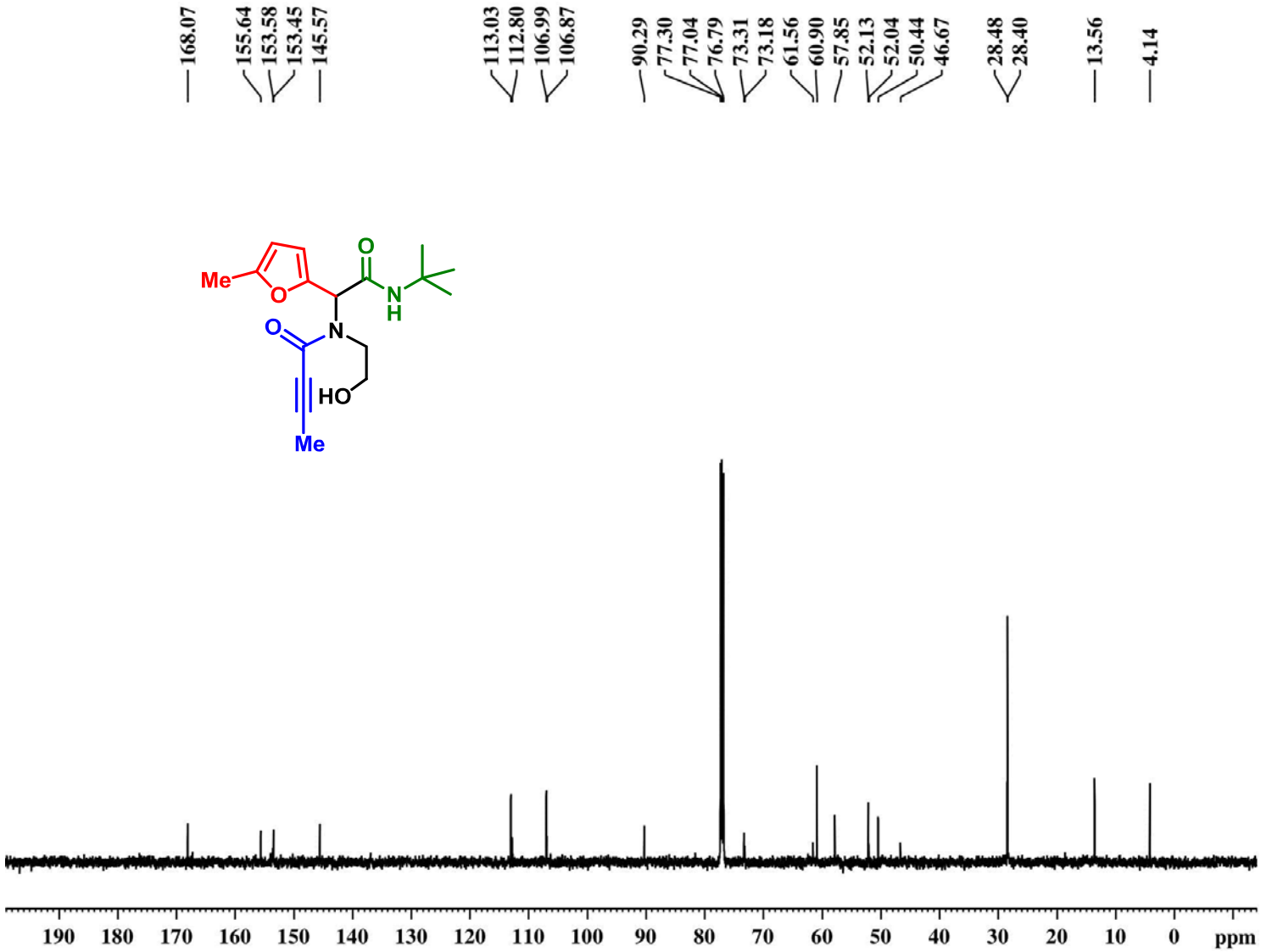
^{1}H NMR spectrum of **5j** (400 MHz, $CDCl_3$)


170.44
169.90
159.95
156.59
156.00
131.17
131.00
125.89
125.53
114.55
114.50
91.42
90.33
77.31
77.05
76.80
73.52
66.40
63.36
61.25
60.85
55.32
52.13
49.66
46.07
28.58
28.45
4.20
4.13
MeO
O
N
H
O
N
HO
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

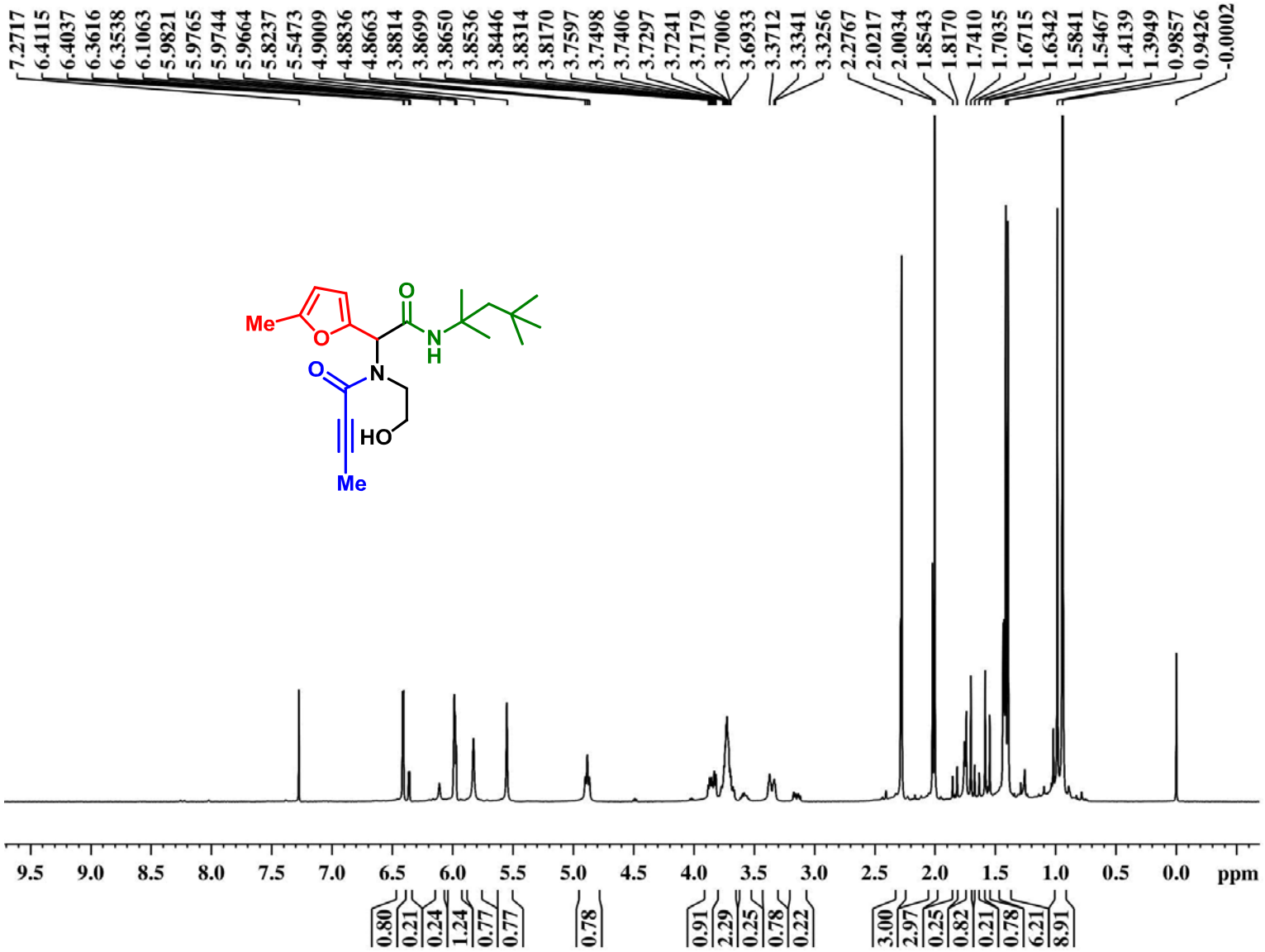
^{13}C NMR spectrum of **5j** (100MHz, $CDCl_3$)


7.2747
6.4428
6.3885
6.3807
6.3393
6.3315
5.9856
5.9795
5.9779
5.9232
5.8507
5.5986
4.8919
4.8745
4.8571
3.9509
3.9401
3.9340
3.9230
3.9137
3.9033
3.8961
3.8858
3.8231
3.8146
3.8064
3.7949
3.7871
3.7769
3.7691
3.7617
3.7532
3.7444
3.7325
3.6951
3.6864
3.6623
3.2309
3.2220
3.2114
3.1939
3.1843
3.1743
2.2859
2.0295
2.0064
1.3801
1.3411
-0.0002
Me
O
O
N
H
O
N
HO
Me
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
ppm
0.24
0.80
0.22
1.02
0.24
0.76
0.79
0.82
0.84
0.58
1.83
0.26
0.80
3.00
3.04
9.25

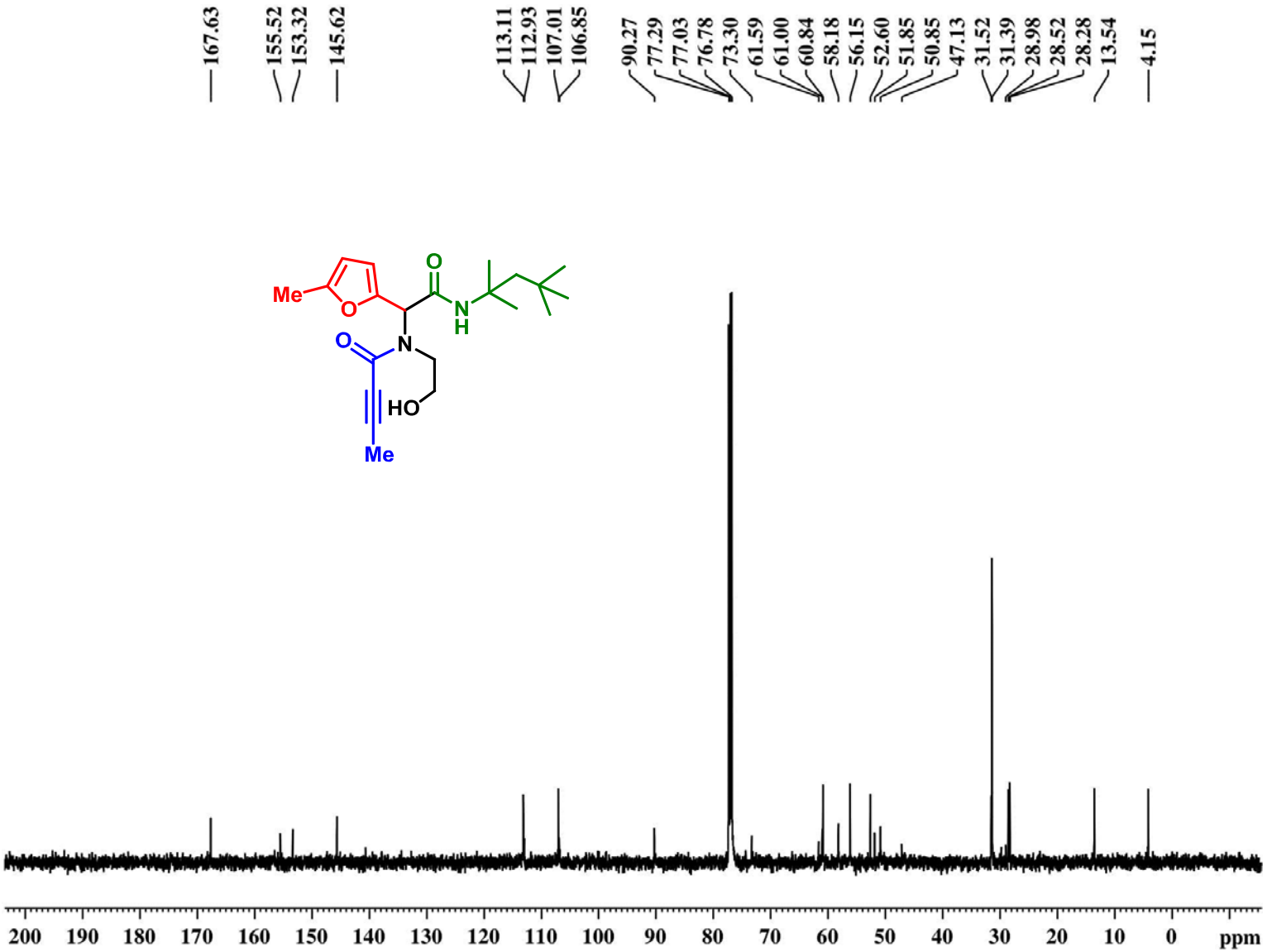
^{1}H NMR spectrum of **5k** (400MHz, $CDCl_3$)


168.07
155.64
153.58
153.45
145.57
113.03
112.80
106.99
106.87
90.29
77.30
77.04
76.79
73.31
73.18
61.56
60.90
57.85
52.13
52.04
50.44
46.67
28.48
28.40
13.56
4.14
Me
O
O
N
H
O
N
HO
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

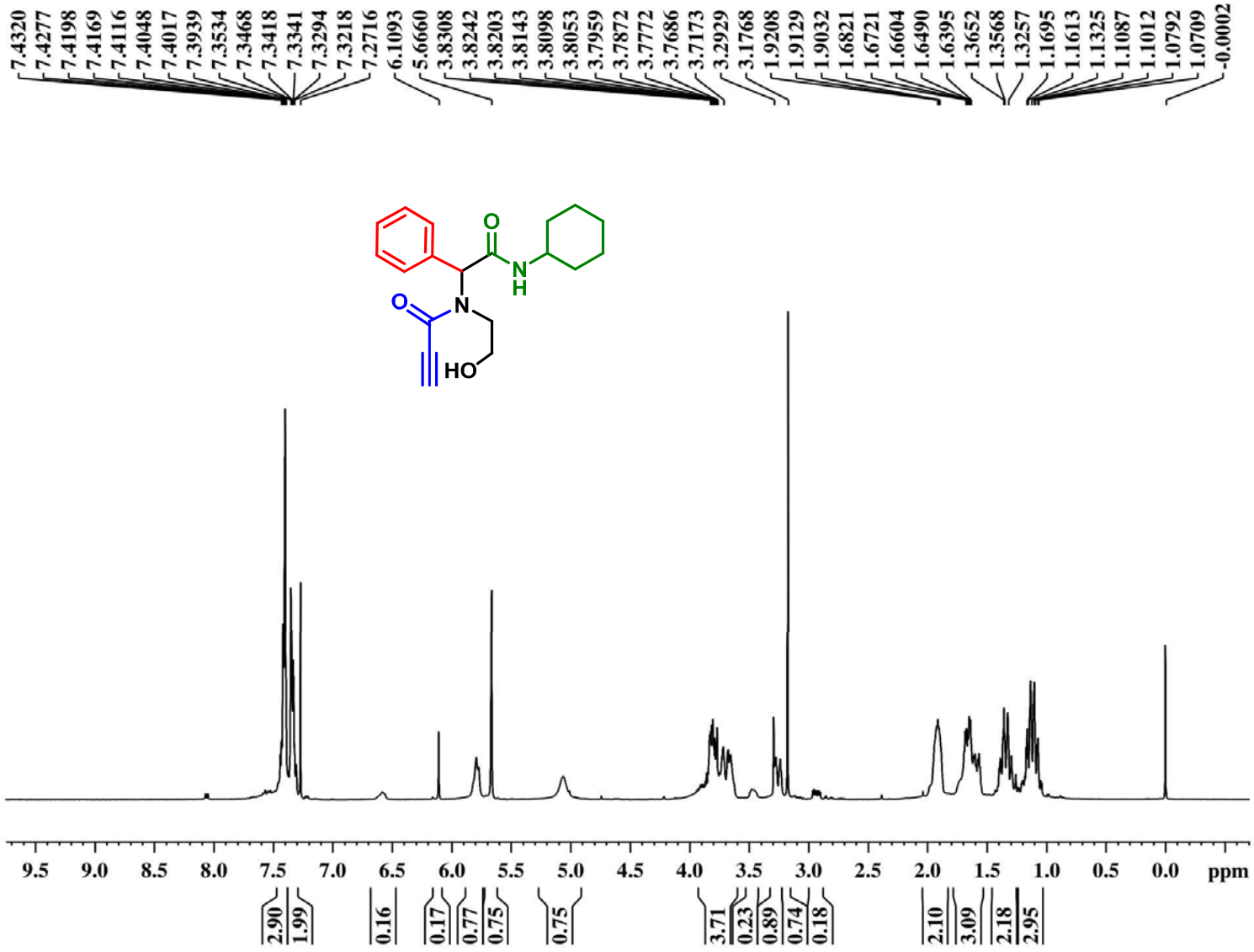
^{13}C NMR spectrum of **5k** (100MHz, $CDCl_3$)


7.2717
6.4115
6.4037
6.3616
6.3538
6.1063
5.9821
5.9765
5.9744
5.9664
5.8237
5.5473
4.9009
4.8836
4.8663
3.8814
3.8699
3.8650
3.8536
3.8446
3.8314
3.8170
3.7597
3.7498
3.7406
3.7297
3.7241
3.7179
3.7006
3.6933
3.3712
3.3341
3.3256
2.2767
2.0217
2.0034
1.8543
1.8170
1.7410
1.7035
1.6715
1.6342
1.5841
1.5467
1.4139
1.3949
0.9857
0.9426
-0.0002
Me
O
O
N
H
O
N
HO
Me
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
ppm
0.80
0.21
0.24
1.24
0.77
0.77
0.78
0.91
2.29
0.25
0.78
0.22
3.00
2.97
0.25
0.82
0.21
0.78
6.21
8.91

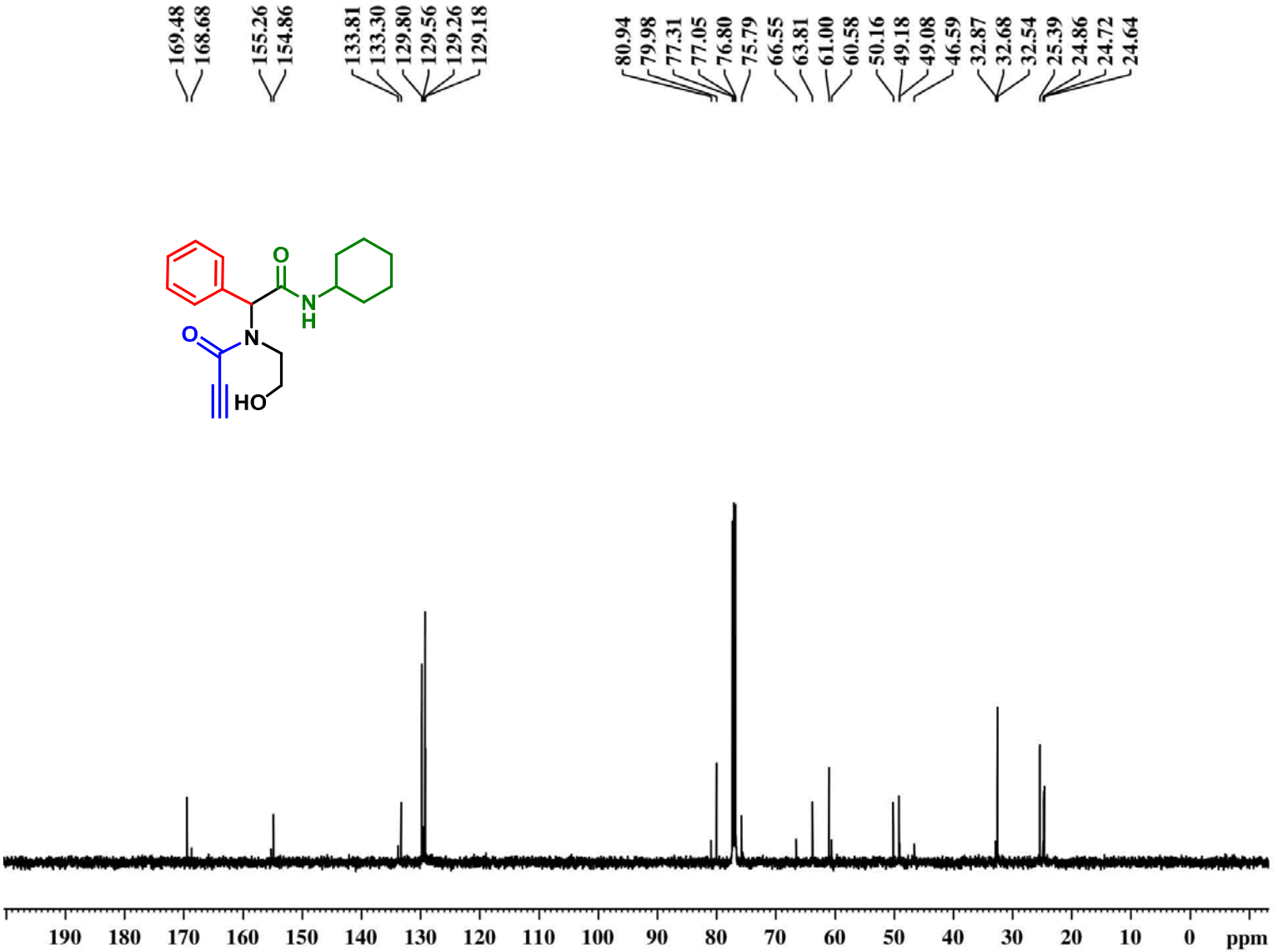
^{1}H NMR spectrum of **5l** (400 MHz, $CDCl_3$)


167.63
155.52
153.32
145.62
113.11
112.93
107.01
106.85
90.27
77.29
77.03
76.78
73.30
61.59
61.00
60.84
58.18
56.15
52.60
51.85
50.85
47.13
31.52
31.39
28.98
28.52
28.28
13.54
4.15
Me
O
O
N
H
O
N
HO
Me
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

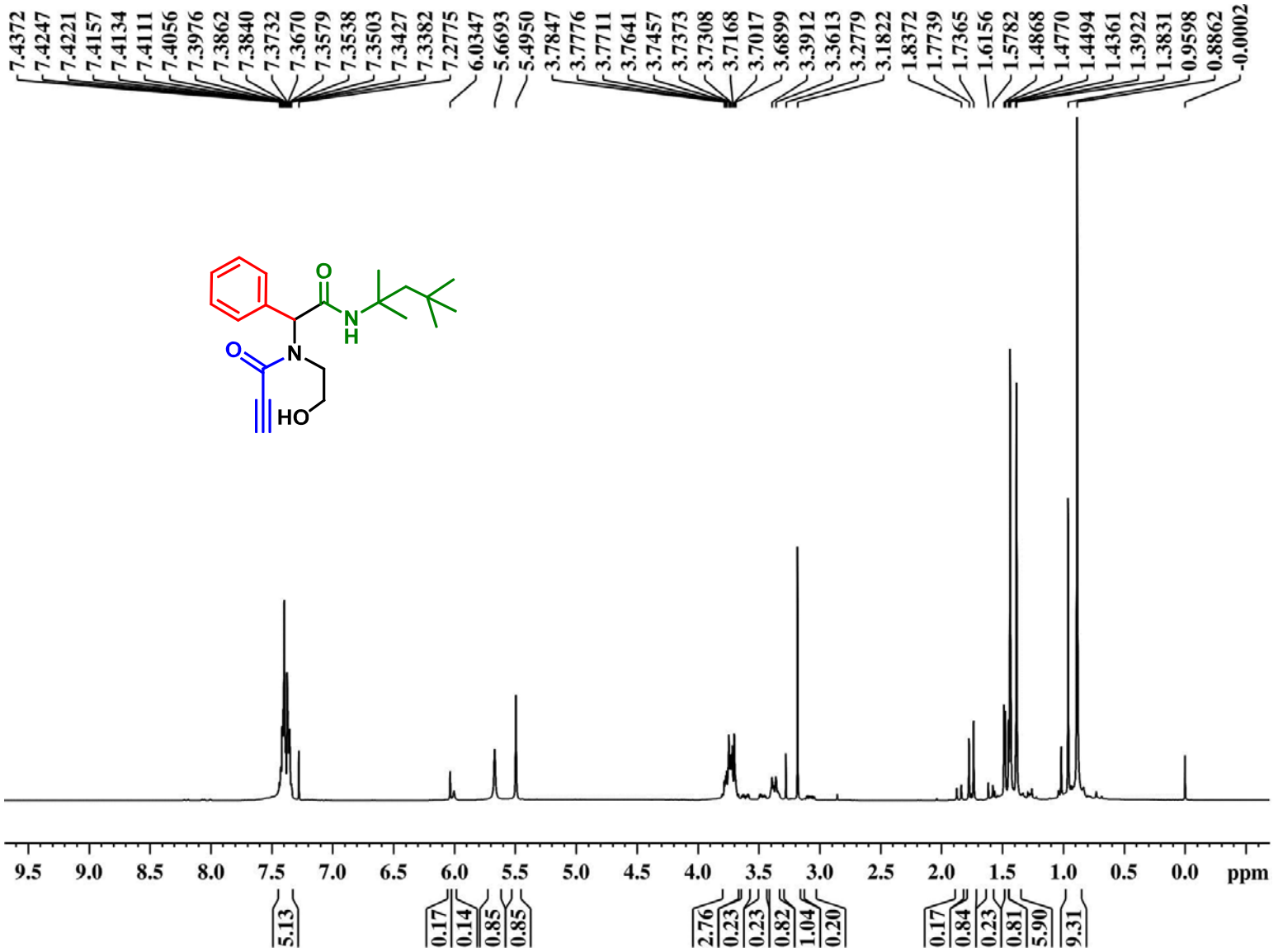
^{13}C NMR spectrum of **5l** (100MHz, $CDCl_3$)


7.4320
7.4277
7.4198
7.4169
7.4116
7.4048
7.4017
7.3939
7.3534
7.3468
7.3418
7.3341
7.3294
7.3218
7.2716
6.1093
5.6660
3.8308
3.8242
3.8203
3.8143
3.8098
3.8053
3.7959
3.7872
3.7772
3.7686
3.7173
3.2929
3.1768
1.9208
1.9129
1.9032
1.6821
1.6721
1.6604
1.6490
1.6395
1.3652
1.3568
1.3257
1.1695
1.1613
1.1325
1.1087
1.1012
1.0792
1.0709
-0.0002
O
N
H
O
N
HO
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 ppm
2.90 1.99 0.16 0.17 0.77 0.75 0.75 3.71 0.23 0.89 0.74 0.18 2.10 3.09 2.18 2.95

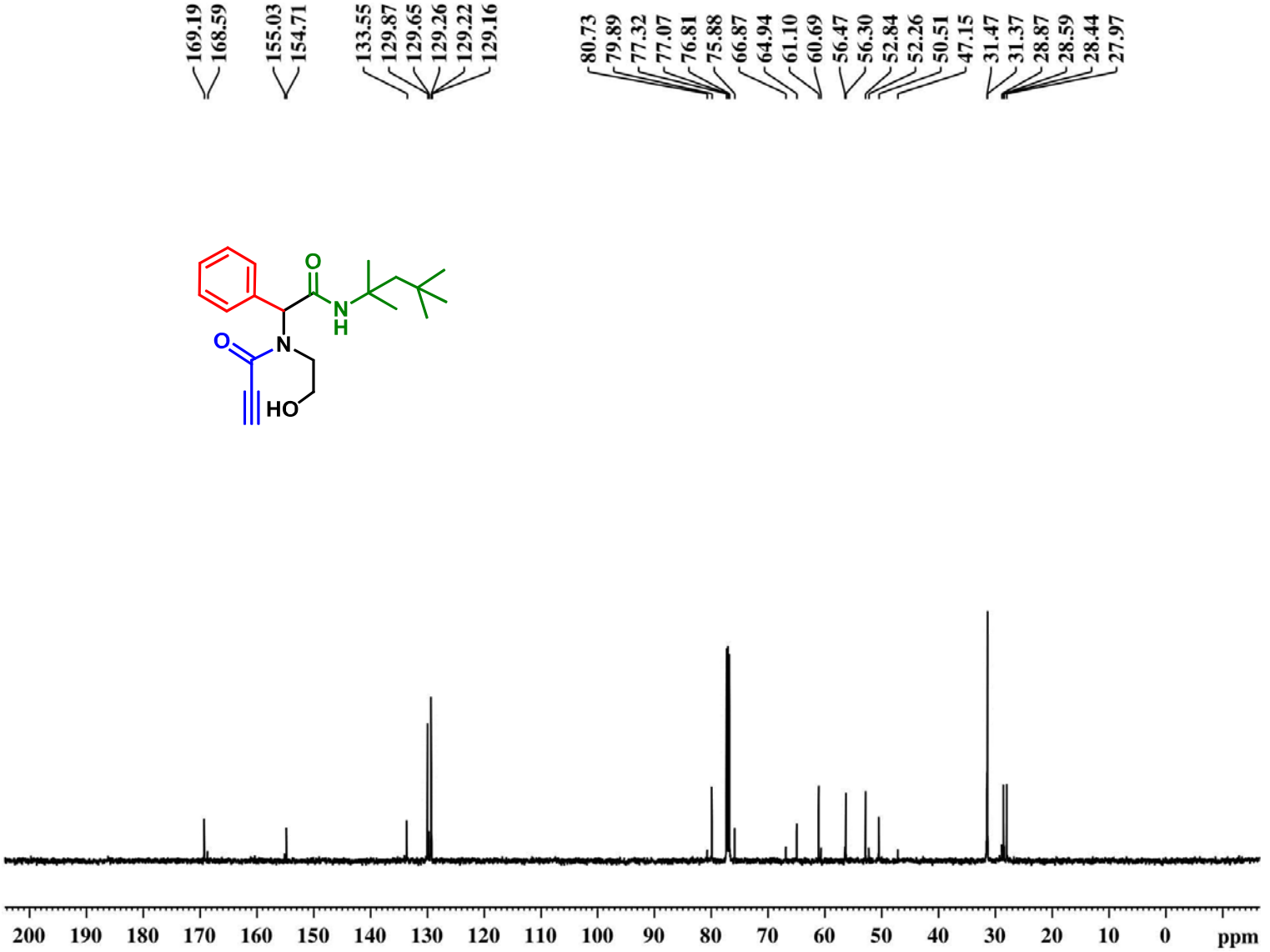
^{1}H NMR spectrum of **5m** (400MHz, $CDCl_3$)


169.48
168.68
155.26
154.86
133.81
133.30
129.80
129.56
129.26
129.18
80.94
79.98
77.31
77.05
76.80
75.79
66.55
63.81
61.00
60.58
50.16
49.18
49.08
46.59
32.87
32.68
32.54
25.39
24.86
24.72
24.64
O
N
H
O
N
HO
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

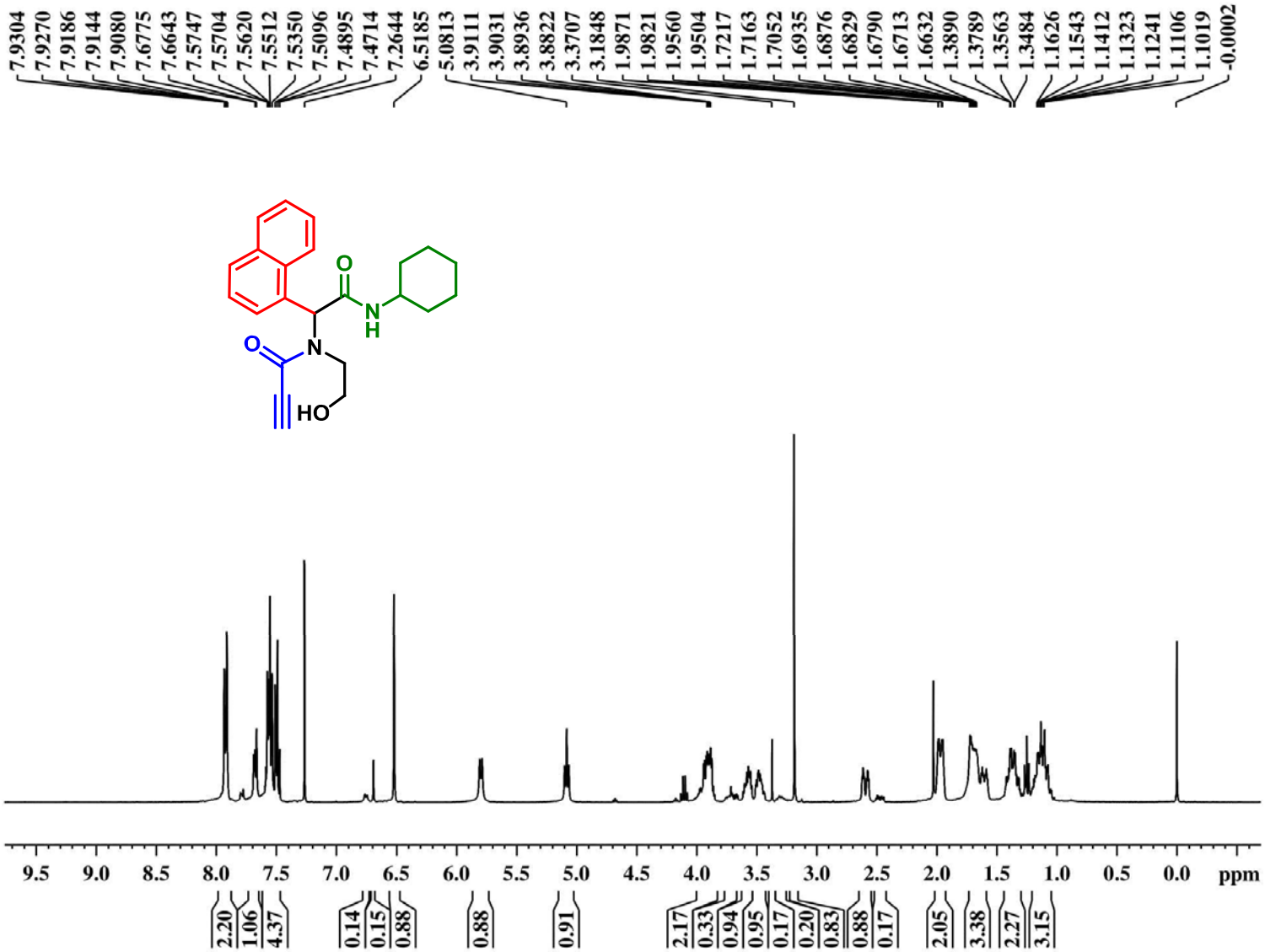
^{13}C NMR spectrum of **5m** (100MHz, $CDCl_3$)


7.4372
7.4247
7.4221
7.4157
7.4134
7.4111
7.4056
7.3976
7.3862
7.3840
7.3732
7.3670
7.3579
7.3538
7.3503
7.3427
7.3382
7.2775
6.0347
5.6693
5.4950
3.7847
3.7776
3.7711
3.7641
3.7457
3.7373
3.7308
3.7168
3.7017
3.6899
3.3912
3.3613
3.2779
3.1822
1.8372
1.7739
1.7365
1.6156
1.5782
1.4868
1.4770
1.4494
1.4361
1.3922
1.3831
0.9598
0.8862
-0.0002
O
N
H
O
N
HO
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
ppm
5.13
0.17
0.14
0.85
0.85
2.76
0.23
0.23
0.82
1.04
0.20
0.17
0.84
0.23
0.81
5.90
9.31

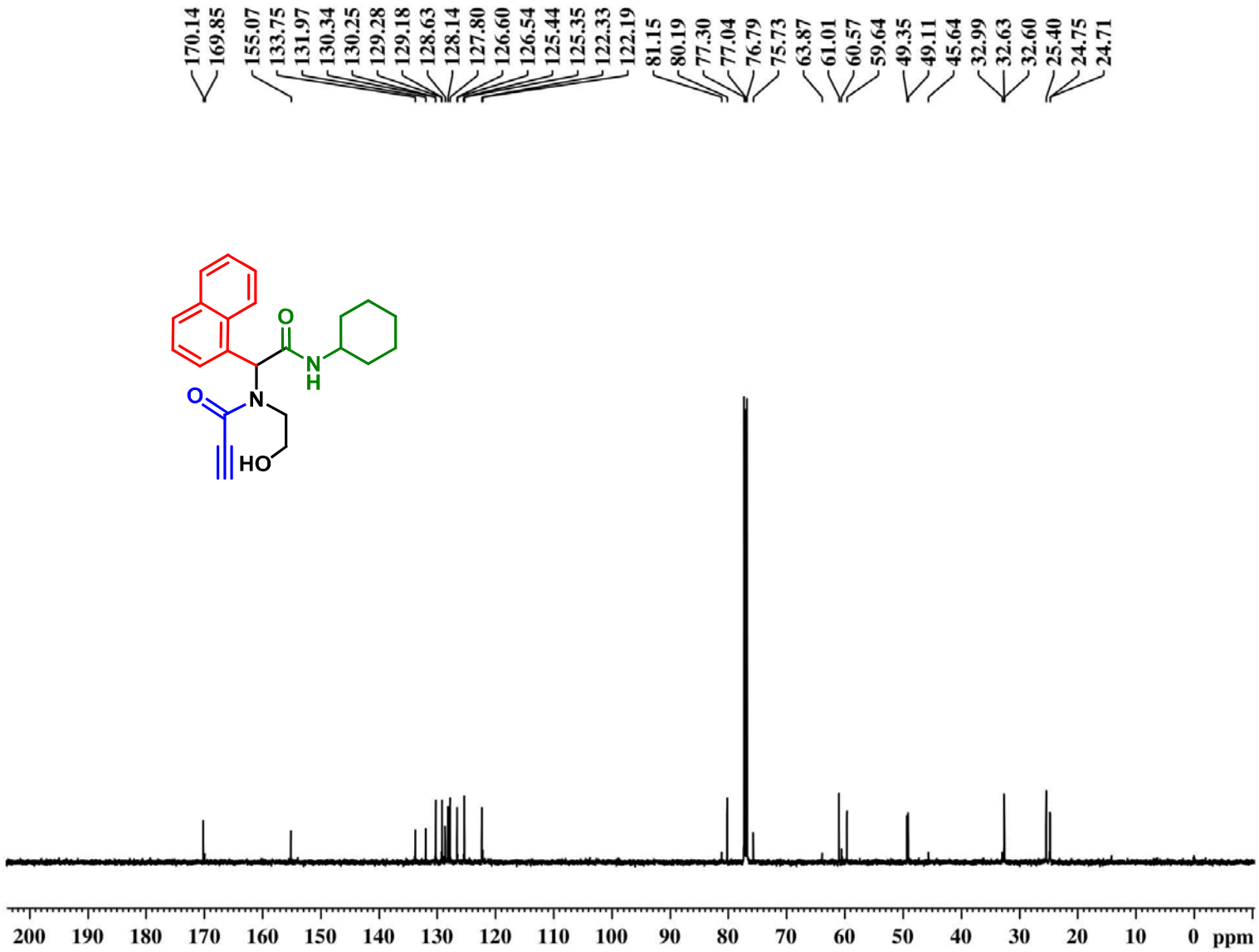
^{1}H NMR spectrum of **5n** (400MHz, $CDCl_3$)


169.19
168.59
155.03
154.71
133.55
129.87
129.65
129.26
129.22
129.16
80.73
79.89
77.32
77.07
76.81
75.88
66.87
64.94
61.10
60.69
56.47
56.30
52.84
52.26
50.51
47.15
31.47
31.37
28.87
28.59
28.44
27.97
O
N
H
O
N
HO
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

^{13}C NMR spectrum of **5n** (100MHz, $CDCl_3$)


7.9304
7.9270
7.9186
7.9144
7.9080
7.6775
7.6643
7.5747
7.5704
7.5620
7.5512
7.5350
7.5096
7.4895
7.4714
7.2644
6.5185
5.0813
3.9111
3.9031
3.8936
3.8822
3.3707
3.1848
1.9871
1.9821
1.9560
1.9504
1.7217
1.7163
1.7052
1.6935
1.6876
1.6829
1.6790
1.6713
1.6632
1.3890
1.3789
1.3563
1.3484
1.1626
1.1543
1.1412
1.1323
1.1241
1.1106
1.1019
-0.0002
O
N
H
O
N
HO
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
ppm
2.20
1.06
4.37
0.14
0.15
0.88
0.88
0.91
2.17
0.33
0.94
0.95
0.17
0.20
0.83
0.88
0.17
2.05
3.38
2.27
3.15

^{1}H NMR spectrum of **5o** (400MHz, $CDCl_3$)


170.14
169.85
155.07
133.75
131.97
130.34
130.25
129.28
129.18
128.63
128.14
127.80
126.60
126.54
125.44
125.35
122.33
122.19
81.15
80.19
77.30
77.04
76.79
75.73
63.87
61.01
60.57
59.64
49.35
49.11
45.64
32.99
32.63
32.60
25.40
24.75
24.71
O
N
H
O
N
HO
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR spectrum of **5o** (100 MHz, $CDCl_3$)