Supporting Information

Peptide diversification through addition reaction of free carboxylic acids to ynamides

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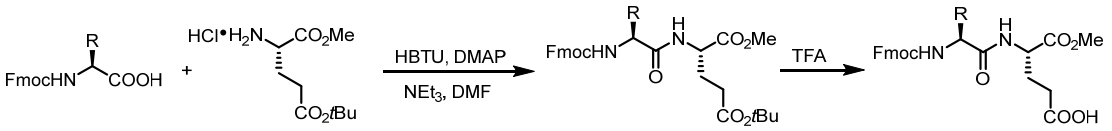
1. General Information

Ynamides **2a**[1], **2b**[2], **2d**[3], **2e**[4] and **2i**[1] were all prepared according to previous reports. All other reagents were used as received from commercial sources. Reactions were monitored through thin-layer chromatography (TLC) on 0.25-mm silica gel plates and visualized under UV light. Flash column chromatography (FCC) was performed using Flash silica gel (90-Å pore size, 200-300 μm). NMR spectra were recorded on a Bruker Avance-400 or -600 instrument, calibrated to $CD(H)Cl_3$ as the internal reference (7.26 and 77.0 ppm for ^1H and ^{13}C NMR spectra, respectively) and $CD(H)_3OD(H)$ as the internal reference (3.31 and 49.0 ppm for ^1H and ^{13}C NMR spectra, respectively). ^1H NMR spectral data were reported in terms of chemical shift (δ, ppm), multiplicity, coupling constant (Hz), and integration. ^{13}C NMR spectral data were reported in terms of chemical shift (δ, ppm). The following abbreviations indicated the multiplicities: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; and br, broad. High-resolution mass spectra were recorded using a SCIEX X500R LC-Q-TOF, ESI ion Source.

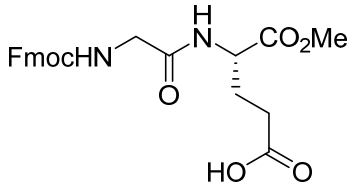
2. Substrate Preparation

Dipeptides were obtained using classical peptide synthesis in the liquid phase, by successive coupling and deprotection reactions.



General procedure for the formation of amide: The amine (2.0 mmol) was dissolved in dried DMF (4.0 mL) in a flask equipped with a magnetic stirring bar. The carboxylic acid (2.2 mmol) and NEt_3 (4 mmol) were added and the mixture was cooled to 0 °C. Then HBTU (4.4 mmol) was added portion-wise and the mixture was stirred at room temperature. Then DMAP (0.4 mmol) was added and the reaction was stirred for 24 h. The reaction mixture was poured over 5% aqueous HCl (20 mL) under vigorous stirring. Extractions with ethyl acetate (3 × 20 mL) followed. The organic layers were combined, washed with saturated aqueous $NaHCO_3$ (3 × 20 mL) and brine (20 mL), and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the reaction crude was purified by silica gel column chromatography (Petroleum ether/EtOAc or Methanol/Dichloromethane) to afford the products.

General procedure for the deprotection of *t*Bu group: The dipeptide (1.0 mmol) was dissolved in TFA (1 mL) and the solution was stirred at room temperature, monitored by TLC. After the dipeptide was consumed completely, the solvent was removed under reduced pressure and the reaction crude was purified by silica gel column chromatography (Petroleum ether/EtOAc or Methanol/Dichloromethane) to afford the products.



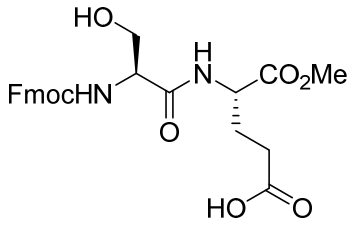
1a

396.1 mg, 45% (last step). Light yellow solid. R_f = 0.3 (Methanol/Dichloromethane, 1:10).

^{1}H NMR (400 MHz, CD_3OD) δ 7.79 (d, J = 7.6 Hz, 2H), 7.74 – 7.58 (m, 2H), 7.39 (t, J = 7.5 Hz, 2H), 7.31 (t, J = 7.5 Hz, 2H), 4.60 – 4.47 (m, 1H), 4.37 (d, J = 7.0 Hz, 2H), 4.23 (t, J = 7.0 Hz, 1H), 3.84 (s, 2H), 3.72 (s, 3H), 2.41 (t, J = 7.5 Hz, 2H), 2.25 – 2.21 (m, 1H), 2.04 – 1.90 (m, 1H).

^{13}C NMR (101 MHz, CD_3OD) δ 174.9, 172.1, 170.9, 157.6, 143.9, 141.2, 127.4, 126.8, 124.8, 119.5, 66.8, 51.7, 51.4, 46.9, 43.3, 29.7, 26.4.

HRMS (ESI, m/z) calcd for $C_{23}H_{25}N_2O_7$ $[M + H]^+$: 441.1657, found: 441.1658.



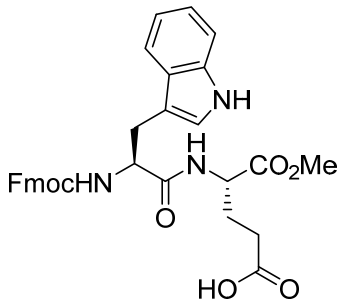
1b

470.2 mg, 73% (last step). Light yellow solid. R_f = 0.3 (Methanol/Dichloromethane, 1:10).

^{1}H NMR (400 MHz, CD_3OD) δ 7.81 (d, J = 7.6 Hz, 2H), 7.74 – 7.58 (m, 2H), 7.41 (t, J = 7.6 Hz, 2H), 7.33 (t, J = 7.6 Hz, 2H), 4.55 (t, J = 7.1 Hz, 1H), 4.41 (d, J = 6.9 Hz, 2H), 4.26 (d, J = 6.2 Hz, 2H), 3.80 (d, J = 5.3 Hz, 2H), 3.73 (s, 3H), 2.38 (d, J = 7.4 Hz, 2H), 2.26 – 2.13 (m, 1H), 2.09 – 1.88 (m, 1H).

^{13}C NMR (101 MHz, CD_3OD) δ 172.2, 171.7, 157.1, 143.9, 143.8, 141.2, 127.4, 126.8, 124.8, 124.8, 119.5, 66.8, 61.8, 57.0, 51.9, 51.5, 47.0, 29.3, 26.7.

HRMS (ESI, m/z) calcd for $C_{24}H_{26}N_2O_8$ $[M + H]^+$: 471.1762, found: 471.1757.



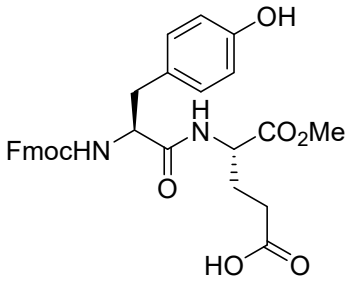
1c

459.4 mg, 40% (last step). Light yellow solid. R_f = 0.3 (Methanol/Dichloromethane, 1:10).

^{1}H NMR (600 MHz, CD_3OD) δ 7.76 (d, J = 7.6 Hz, 2H), 7.67 – 7.49 (m, 3H), 7.47 – 7.31 (m, 3H), 7.30 – 7.18 (m, 2H), 7.16 – 7.06 (m, 2H), 7.06 – 6.98 (m, 1H), 4.55 – 4.40 (m, 2H), 4.30 (dd, J = 10.6, 7.1 Hz, 1H), 4.23 (dd, J = 10.5, 6.8 Hz, 1H), 4.13 (t, J = 7.1 Hz, 1H), 3.64 (s, 3H), 3.28 (dd, J = 14.6, 6.0 Hz, 1H), 3.11 (dd, J = 14.6, 8.0 Hz, 1H), 2.39 – 2.20 (m, 2H), 2.17 – 2.03 (m, 1H), 1.96 – 1.83 (m, 1H).

^{13}C NMR (101 MHz, CD_3OD) δ 173.3, 172.1, 156.8, 143.8, 143.7, 141.1, 136.6, 127.4, 126.8, 124.9, 124.8, 123.4, 121.0, 119.5, 118.5, 117.9, 110.9, 109.4, 66.7, 55.8, 51.9, 51.4, 46.9, 27.7, 26.6.

HRMS (ESI, m/z) calcd for $C_{32}H_{31}N_3O_7$ $[M + H]^+$: 570.2235, found: 570.2219.



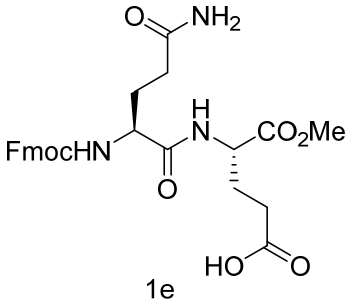
1d

327.7 mg, 30% (last step). Light yellow solid. R_f = 0.4 (Methanol/Dichloromethane, 1:10).

^{1}H NMR (400 MHz, CD_3OD) δ 7.79 (d, J = 7.6 Hz, 1H), 7.72 (d, J = 7.6 Hz, 1H), 7.68 – 7.50 (m, 2H), 7.48 – 7.36 (m, 2H), 7.35 – 7.22 (m, 2H), 7.08 (t, J = 6.7 Hz, 2H), 6.72 (d, J = 7.9 Hz, 2H), 4.61 – 4.43 (m, 2H), 4.40 – 4.13 (m, 3H), 3.70 (s, 3H), 3.08 – 2.96 (m, 1H), 2.87 – 2.74 (m, 1H), 2.37 (q, J = 7.6 Hz, 2H), 2.25 – 2.07 (m, 1H), 2.01 – 1.86 (m, 1H).

^{13}C NMR (101 MHz, CD_3OD) δ 173.0, 172.0, 156.8, 155.8, 143.9, 143.8, 141.1, 140.7, 130.0, 129.5, 127.7, 127.4, 126.8, 125.7, 124.9, 124.8, 119.6, 119.5, 114.8, 66.6, 64.8, 56.5, 51.8, 51.4, 36.9, 30.1, 26.6.

HRMS (ESI, m/z) calcd for $C_{30}H_{30}N_2O_8$ $[M + H]^+$: 547.2075, found: 547.2062.



1e

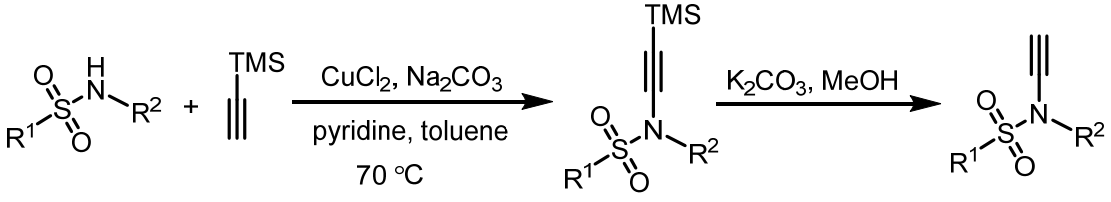
541.9 mg, 53% (last step). Light yellow solid. R_f = 0.3 (Methanol/Dichloromethane, 1:10).

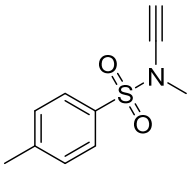
^{1}H NMR (400 MHz, CD_3OD) δ 7.81 (d, J = 7.6 Hz, 2H), 7.68 (t, J = 7.9 Hz, 2H), 7.40 (t, J = 7.6 Hz, 2H), 7.33 (t, J = 7.6 Hz, 2H), 4.53 – 4.45 (m, 1H), 4.44 – 4.33 (m, 2H), 4.29 – 4.12 (m, 2H), 3.73 (s, 3H), 2.43 (t, J = 7.6 Hz, 2H), 2.35 (t, J = 7.7 Hz, 2H), 2.26 – 2.03 (m, 2H), 2.03 – 1.86 (m, 2H).

^{13}C NMR (101 MHz, CD_3OD) δ 178.9, 175.5, 174.6, 159.4, 146.4, 146.2, 143.6, 129.8, 129.2, 127.3, 127.2, 121.9, 69.0, 56.8, 54.2, 53.8, 33.4, 32.2, 30.1, 28.6.

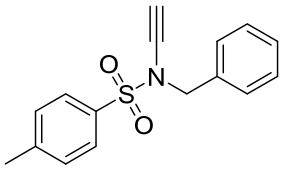
HRMS (ESI, m/z) calcd for $C_{26}H_{29}N_3O_8$ $[M + H]^+$: 512.2028, found: 512.2019.

The ynamides compounds **2a-2i**[1] were prepared using reported methods.

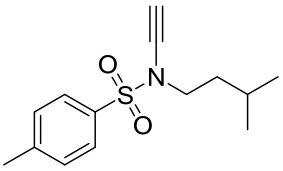




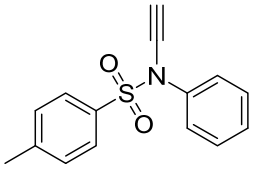
2a



2b



2c



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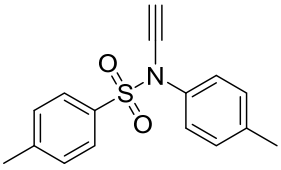
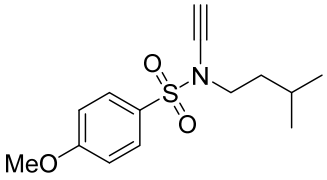
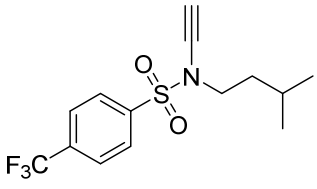


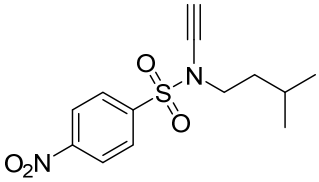
2e



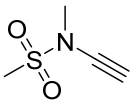
2f



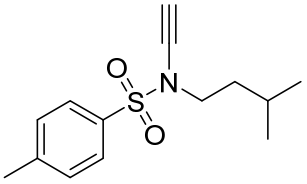
2g



2h



2i



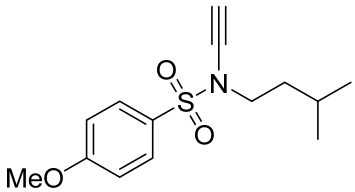
2c

253.2 mg, 63% (last step). Yellow oil. R_f = 0.5 (Petroleum ether/EtOAc, 8:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 8.1 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 3.32 (t, J = 7.5 Hz, 2H), 2.74 (s, 1H), 2.46 (s, 3H), 1.67 – 1.61 (m, 1H), 1.53 (q, J = 7.1 Hz, 2H), 0.90 (d, J = 6.5 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.7, 134.4, 129.7, 127.6, 76.0, 59.0, 49.5, 36.2, 25.1, 22.2, 21.6.

HRMS (ESI, m/z) calcd for $C_{14}H_{20}NO_2S$ $[M + H]^+$: 266.1209, found: 266.1210.



2f

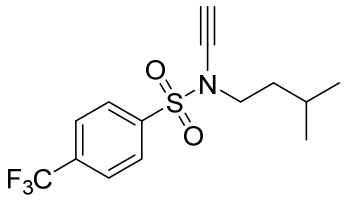
365.4 mg, 26% (last step). Yellow oil. R_f = 0.6 (Petroleum ether/EtOAc, 10:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 7.6 Hz, 2H), 7.03 (d, J = 7.7 Hz, 2H), 3.91 (s, 3H), 3.33 (t, J = 7.0 Hz, 2H), 2.75 (s, 1H), 1.66 (d, J = 15.4 Hz, 1H), 1.54 (q, J = 6.6

Hz, 2H), 0.91 (d, J = 5.7 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 163.7, 129.8, 129.2, 114.3, 76.3, 59.0, 55.7, 49.5, 36.4, 25.3, 22.3.

HRMS (ESI, m/z) calcd for $C_{14}H_{20}NO_3S$ $[M + H]^+$: 282.1159, found: 282.1162.



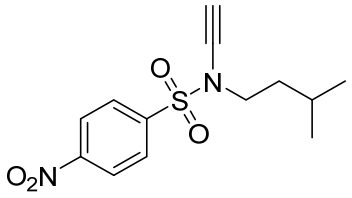
2g

1244.5 mg, 78% (last step). Brown solid. R_f = 0.6 (Petroleum ether/EtOAc, 15:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08 (d, J = 8.1 Hz, 2H), 7.86 (d, J = 8.2 Hz, 2H), 3.39 (t, J = 7.4 Hz, 2H), 2.78 (s, 1H), 1.69 – 1.64 (m, 1H), 1.57 (d, J = 7.3 Hz, 2H), 0.93 (d, J = 6.6 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 140.8, 135.3 (q, J = 33.2 Hz), 128.1, 126.4 (q, J = 3.8 Hz), 123.1 (q, J = 273.1 Hz), 75.2, 59.6, 49.9, 36.4, 25.2, 22.2.

HRMS (ESI, m/z) calcd for $C_{14}H_{17}F_3NO_2S$ $[M + H]^+$: 320.0927, found: 320.0928.



2h

1465.6 mg, 99% (last step). Yellow solid. R_f = 0.6 (Petroleum ether/EtOAc, 15:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, J = 8.9 Hz, 2H), 8.13 (d, J = 8.8 Hz, 2H), 3.44 – 3.36 (m, 2H), 2.79 (s, 1H), 1.7 – 1.62 (m, 1H), 1.56 (t, J = 7.1 Hz, 2H), 0.93 (d, J = 6.5 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 150.6, 142.7, 128.8, 124.4, 74.8, 59.9, 50.1, 36.3, 25.1, 22.2.

HRMS (ESI, m/z) calcd for $C_{13}H_{17}N_2O_4S$ $[M + H]^+$: 297.0904, found: 297.0910.

3. Reference

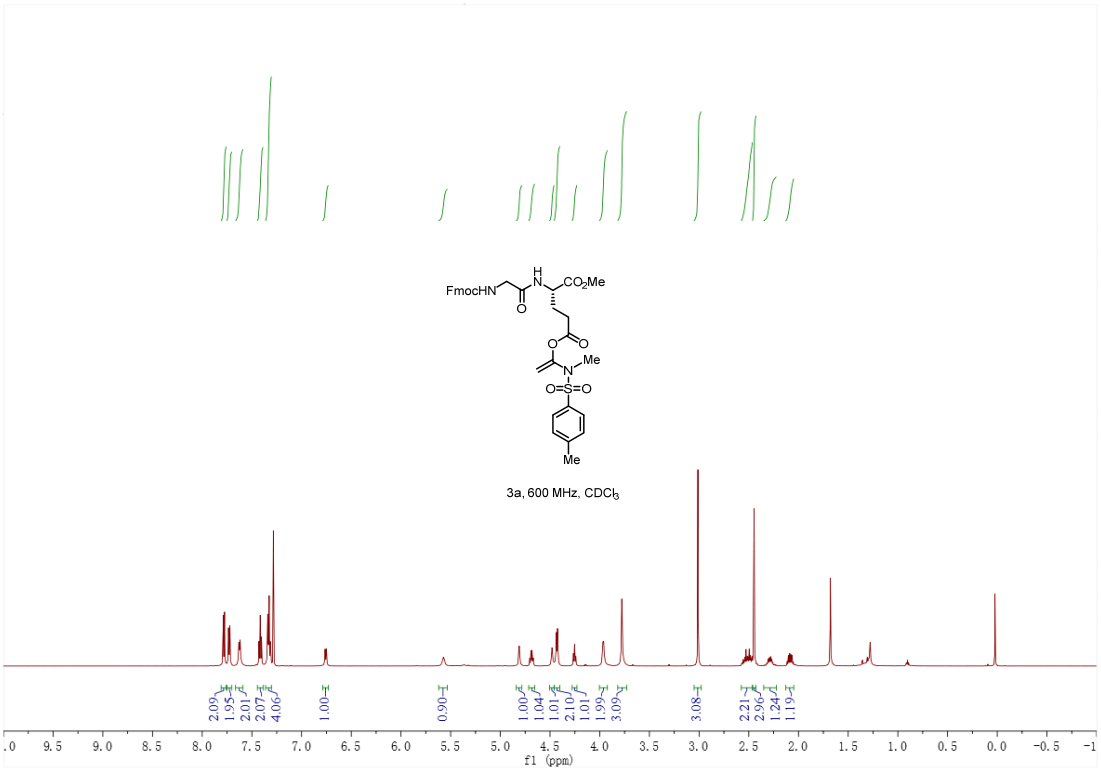
[1] L. Hu, S. Xu, Z. Zhao, Y. Yang, Z. Peng, M. Yang, C. Wang, J. Zhao, *J Am Chem Soc* **2016**, *138*, 13135–13138.

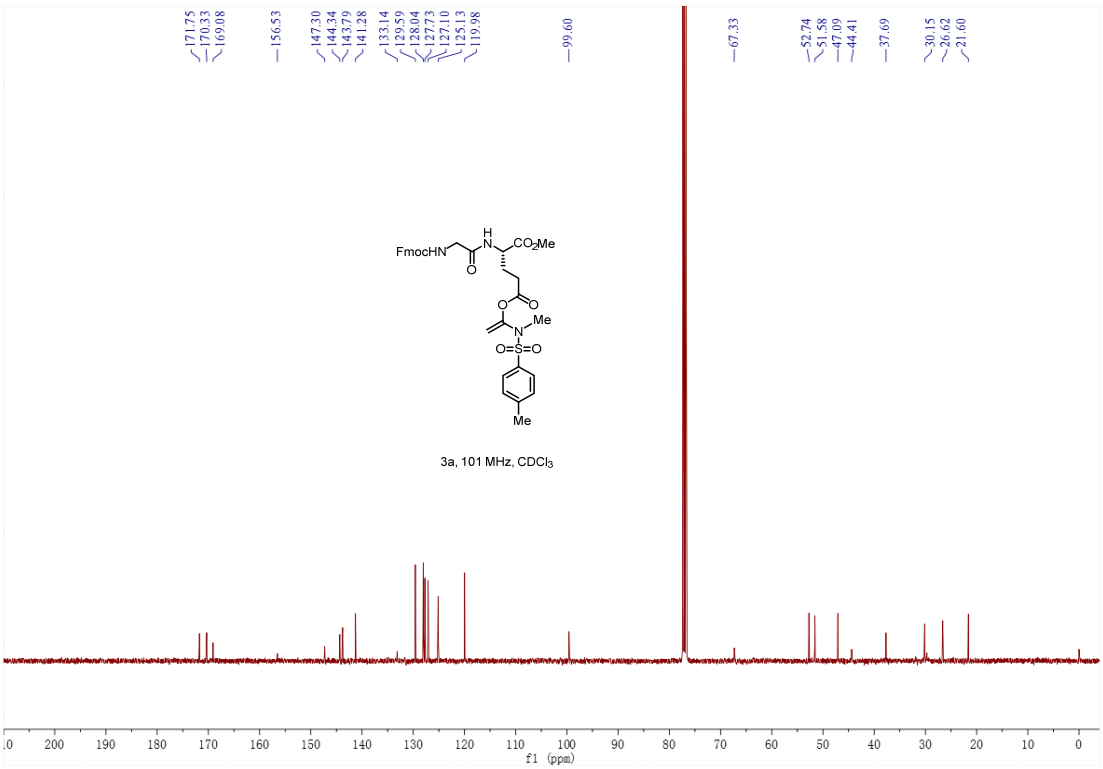
[2] Y. Zheng, B. Moegle, S. Ghosh, A. Perfetto, D. Luise, I. Ciofini, L. Miesch, *Chemistry - A European Journal* **2022**, *28*, DOI 10.1002/chem.202103598.

[3] F. Schlimpen, T. Ast, V. Bénéteau, P. Pale, S. Chassaing, *Green Chemistry* **2022**, *24*, 6467–6475.

[4] M. Yudasaka, D. Shimbo, T. Maruyama, N. Tada, A. Itoh, *Org Lett* **2019**, *21*, 1098–1102.

4. Spectra


FmocHN
CO2Me
Me
Me
3a, 600 MHz, CDCl3
2.09
1.95
2.01
2.07
4.06
1.00
0.90
1.00
1.04
1.01
2.10
1.01
1.99
3.09
3.08
2.21
2.96
1.24
1.19
f1 (ppm)


171.75
170.33
169.08
156.53
147.30
144.34
143.79
141.28
133.14
129.59
128.04
127.73
127.10
125.13
119.98
99.60
67.33
52.74
51.58
47.09
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37.69
30.15
26.62
21.60
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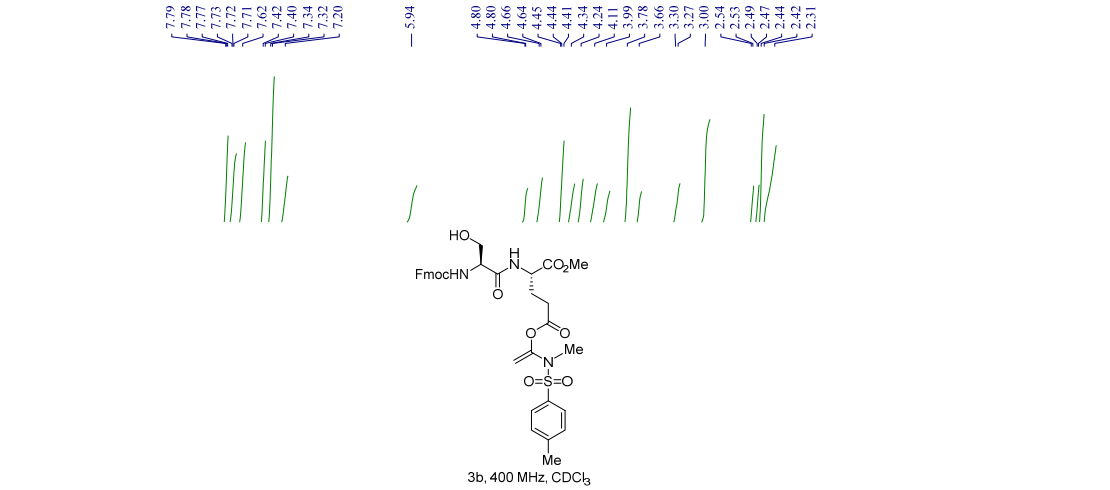
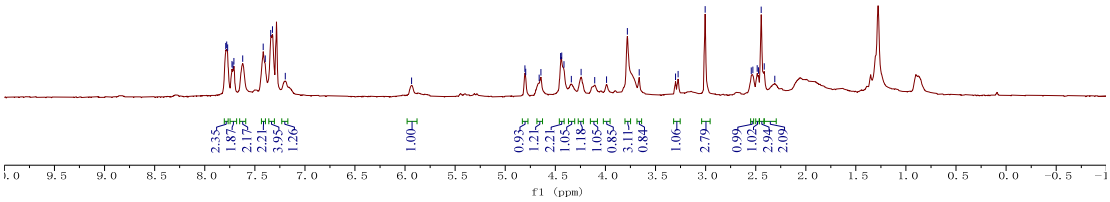
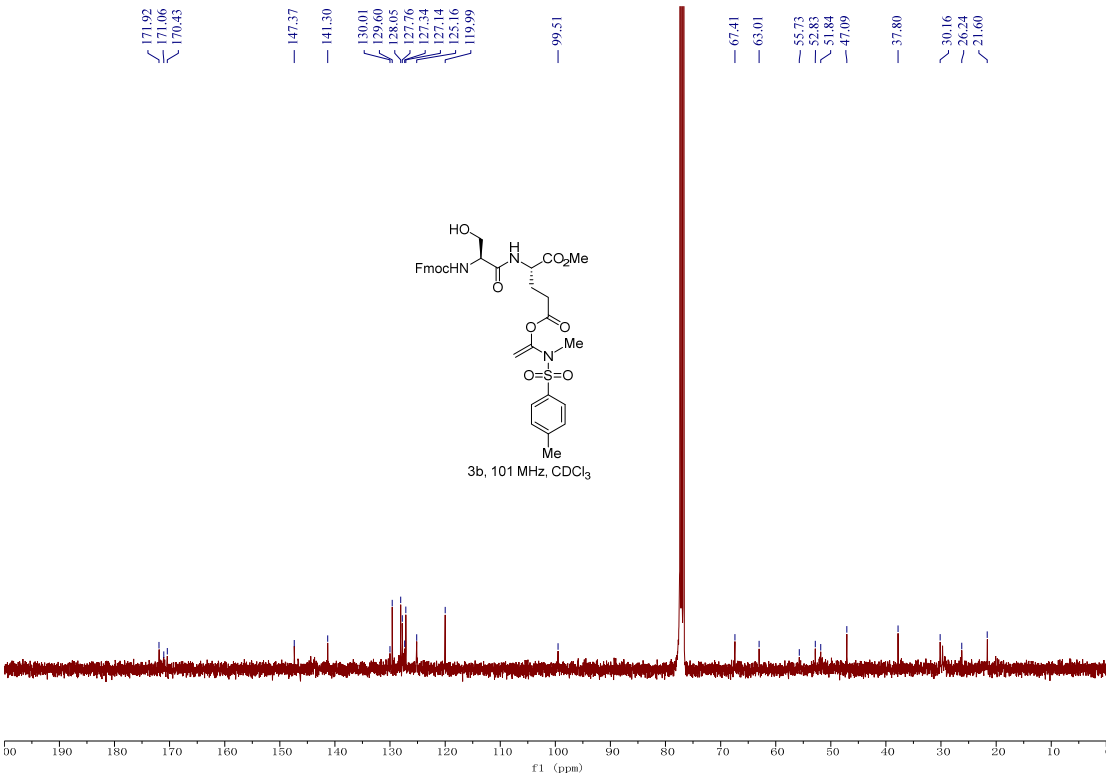
CO2Me
Me
Me
3a, 101 MHz, CDCl3
f1 (ppm)

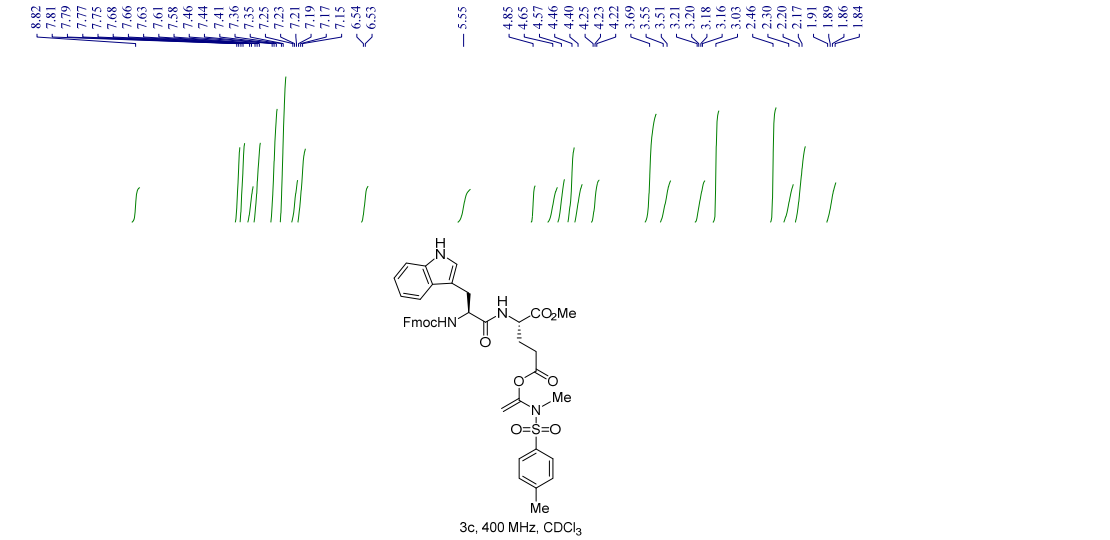


3b, 400 MHz, $CDCl_3$

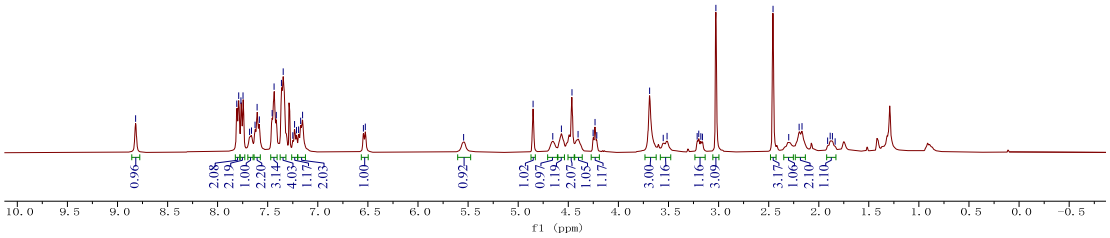


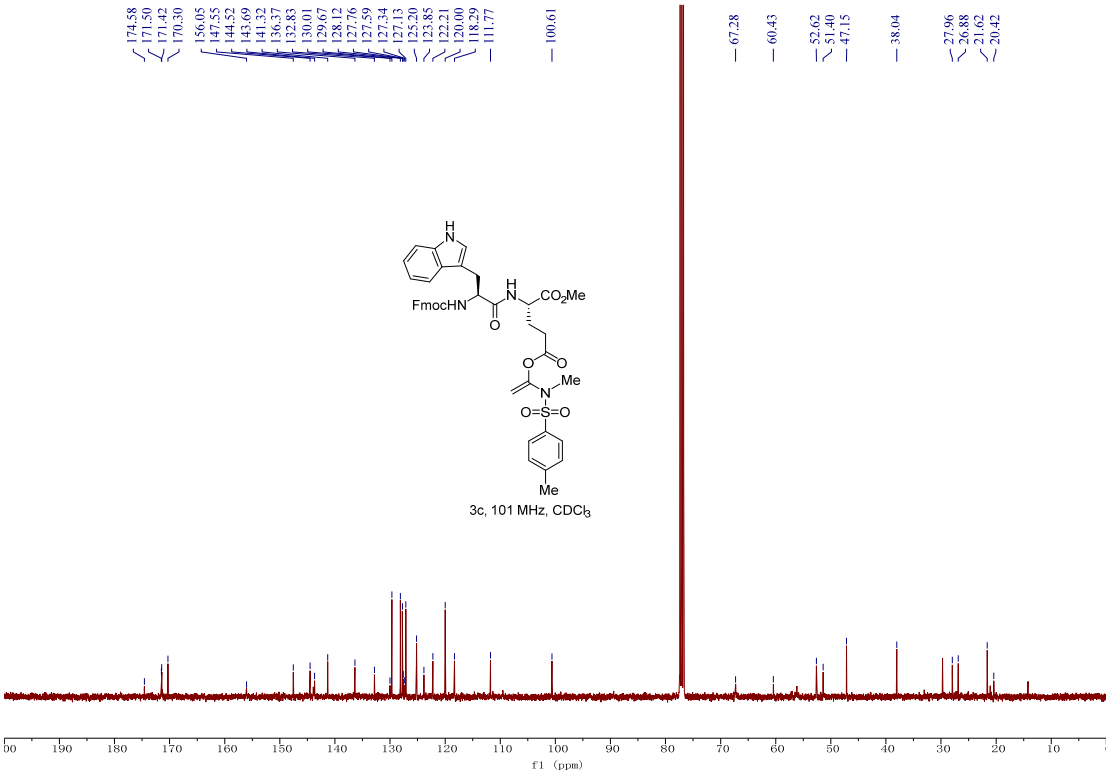


3b, 101 MHz, $CDCl_3$

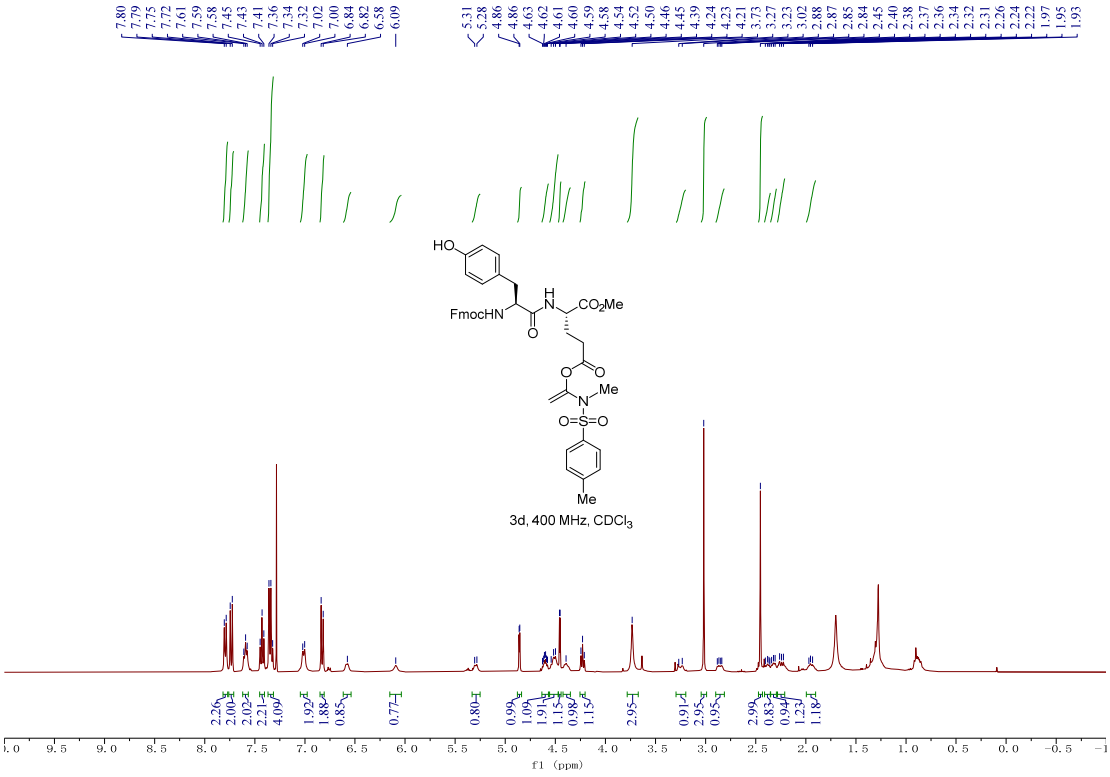


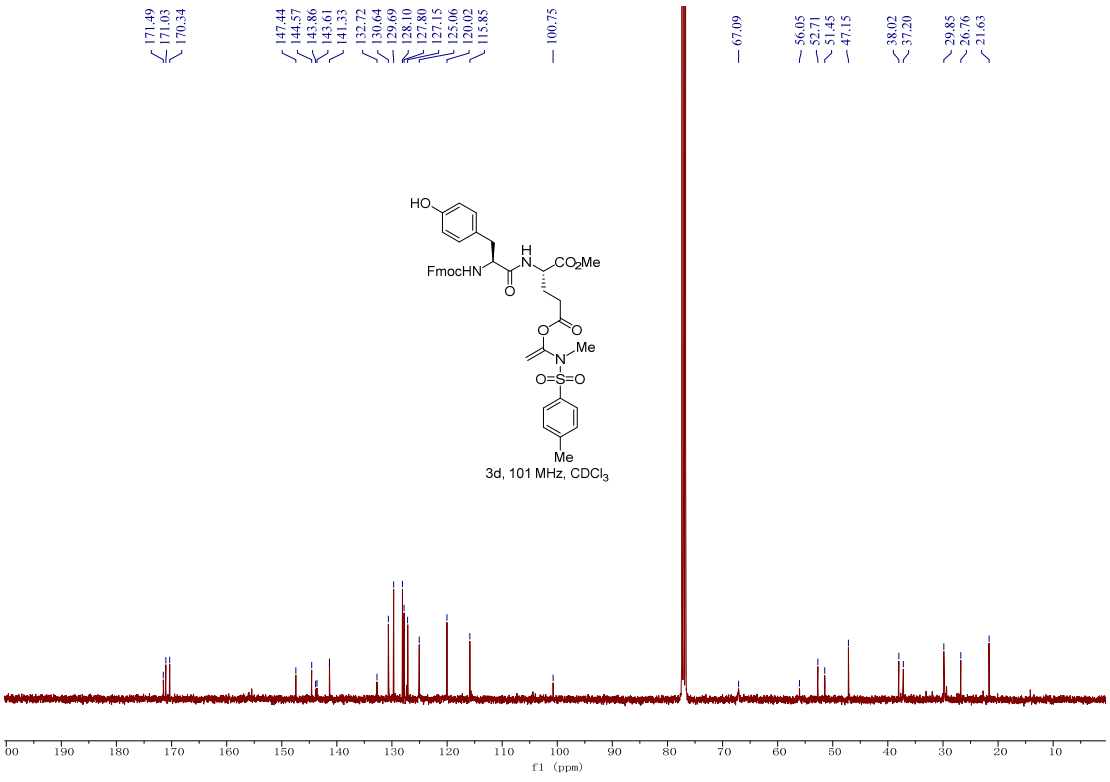
3c, 400 MHz, $CDCl_3$

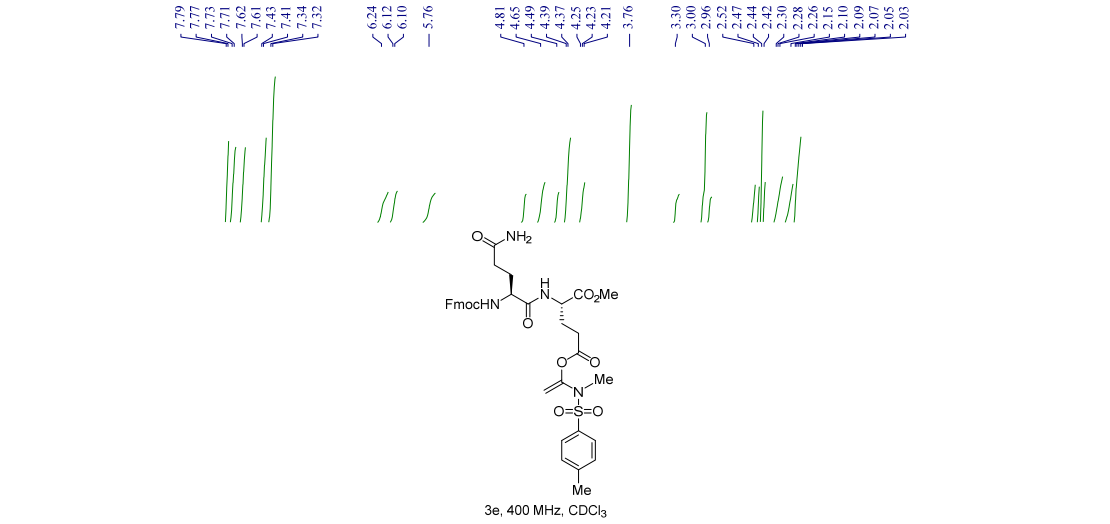




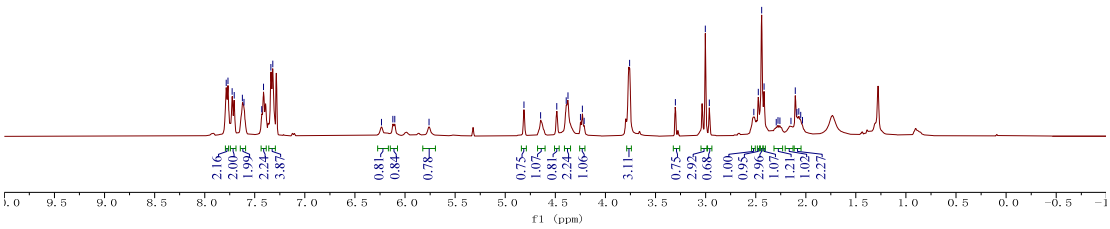
3c, 101 MHz, $CDCl_3$

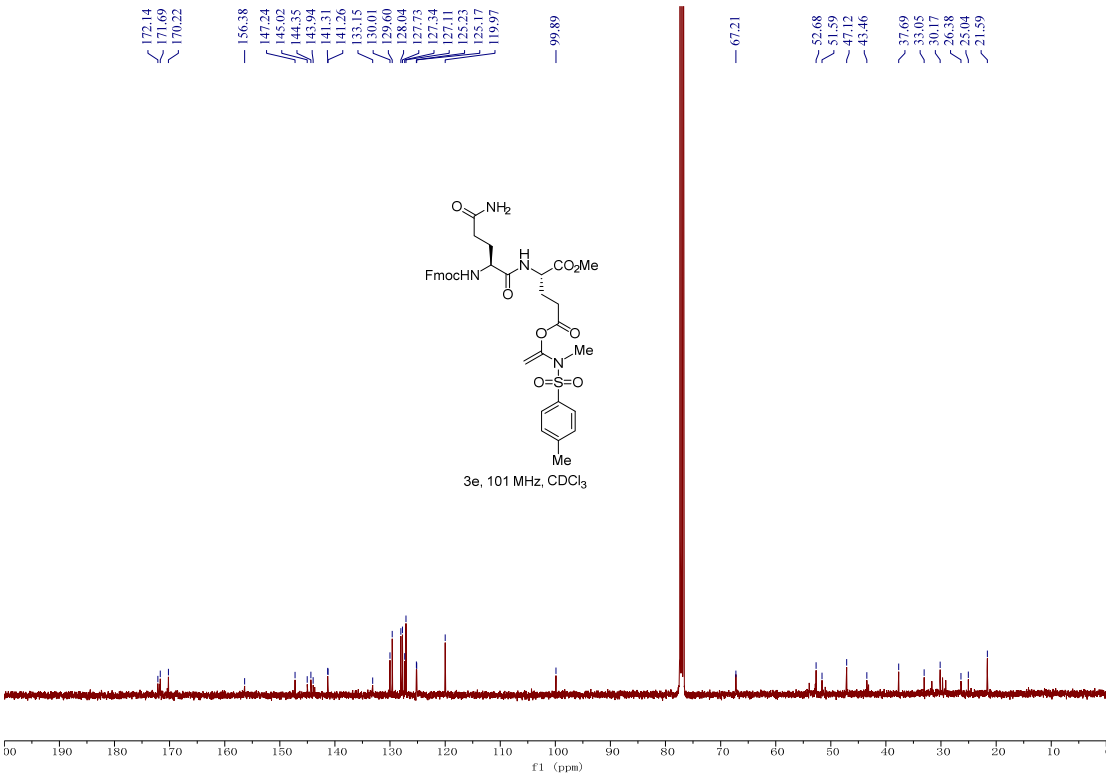

7.80 7.79 7.75 7.72 7.61 7.59 7.58 7.45 7.43 7.41 7.36 7.34 7.32 7.02 7.00 6.84 6.82 6.58 6.09
5.31 5.28 4.86 4.86 4.63 4.62 4.61 4.60 4.59 4.58 4.54 4.52 4.50 4.46 4.45 4.39 4.24 4.23 4.21 3.73 3.27 3.23 3.02 2.88 2.87 2.85 2.84 2.45 2.40 2.38 2.37 2.36 2.34 2.32 2.31 2.26 2.24 2.22 1.97 1.95 1.93
HO
FmocHN
H
N
O
CO2Me
O
O
N
Me
O=S=O
Me
3d, 400 MHz, CDCl3
2.26 2.00 2.02 2.21 4.09 1.92 1.88 0.85 0.77 0.80 0.99 1.09 1.91 1.15 0.98 1.15 2.95 0.91 2.95 0.95 2.99 0.83 0.94 1.29 1.18
f1 (ppm)


171.49 171.03 170.34 147.44 144.57 143.86 143.61 141.33 132.72 130.64 129.69 128.10 127.80 127.15 125.06 120.02 115.85 100.75 67.09 56.05 52.71 51.45 47.15 38.02 37.20 29.85 26.76 21.63
HO
FmocHN
H
N
O
CO2Me
O
O
N
Me
O=S=O
Me
3d, 101 MHz, CDCl3
f1 (ppm)

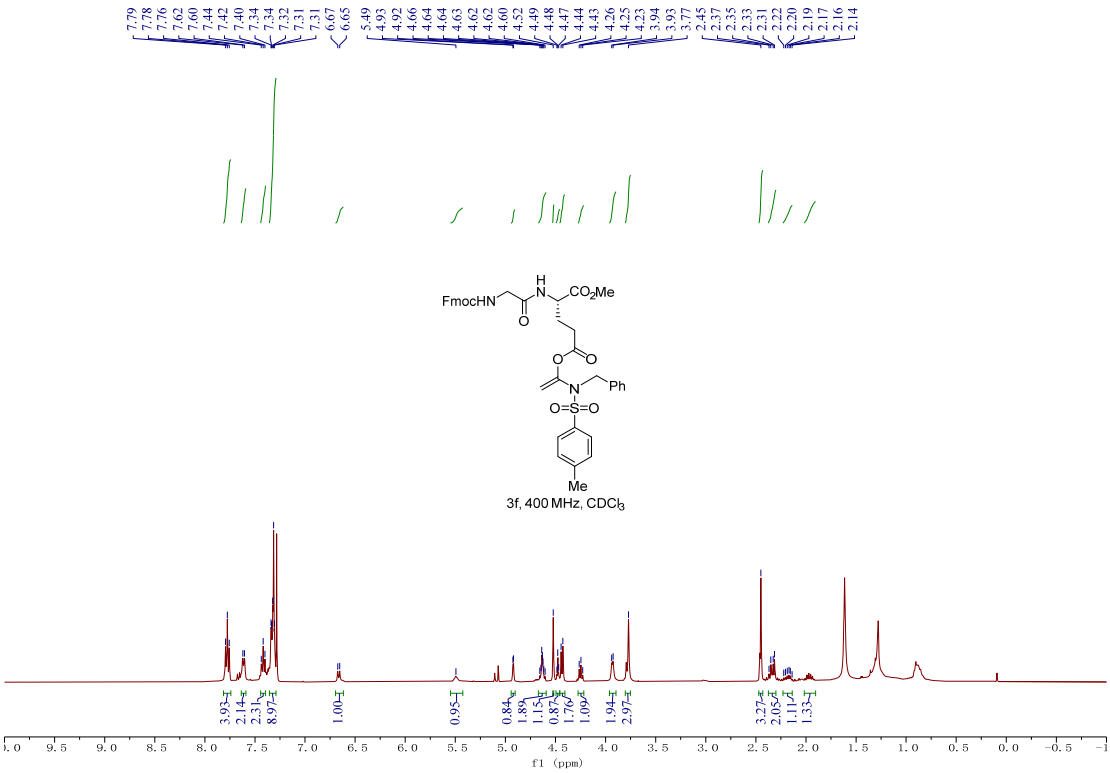


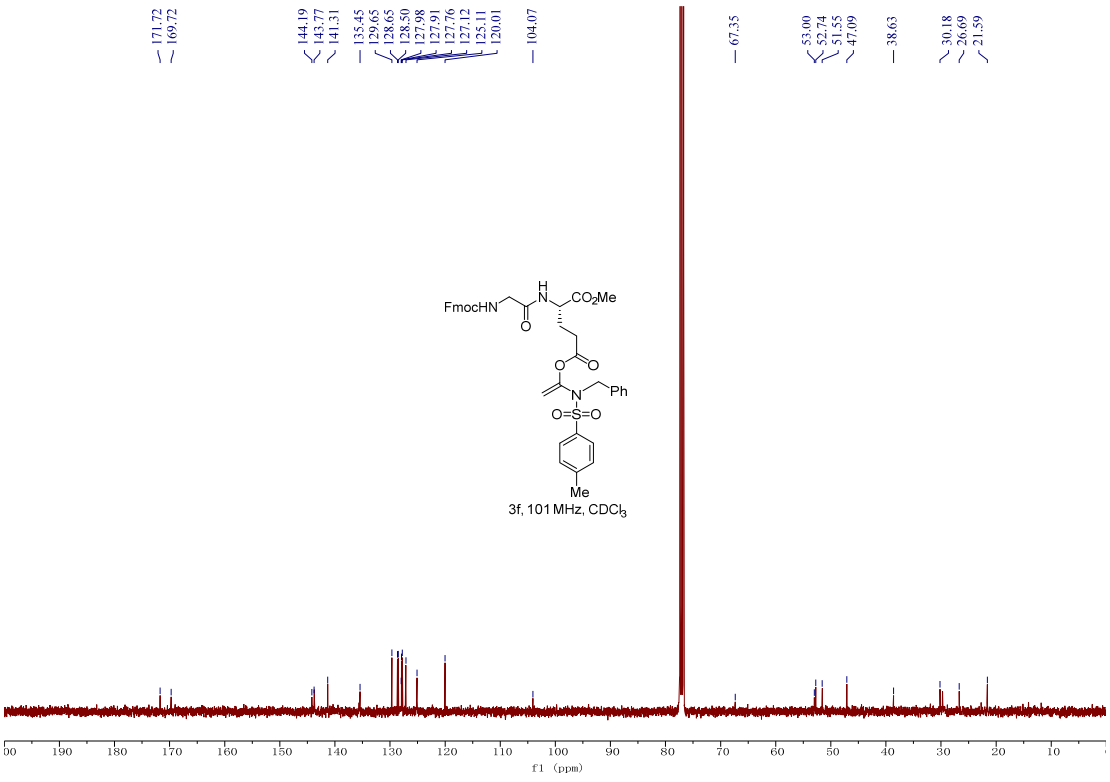
3e, 400 MHz, $CDCl_3$

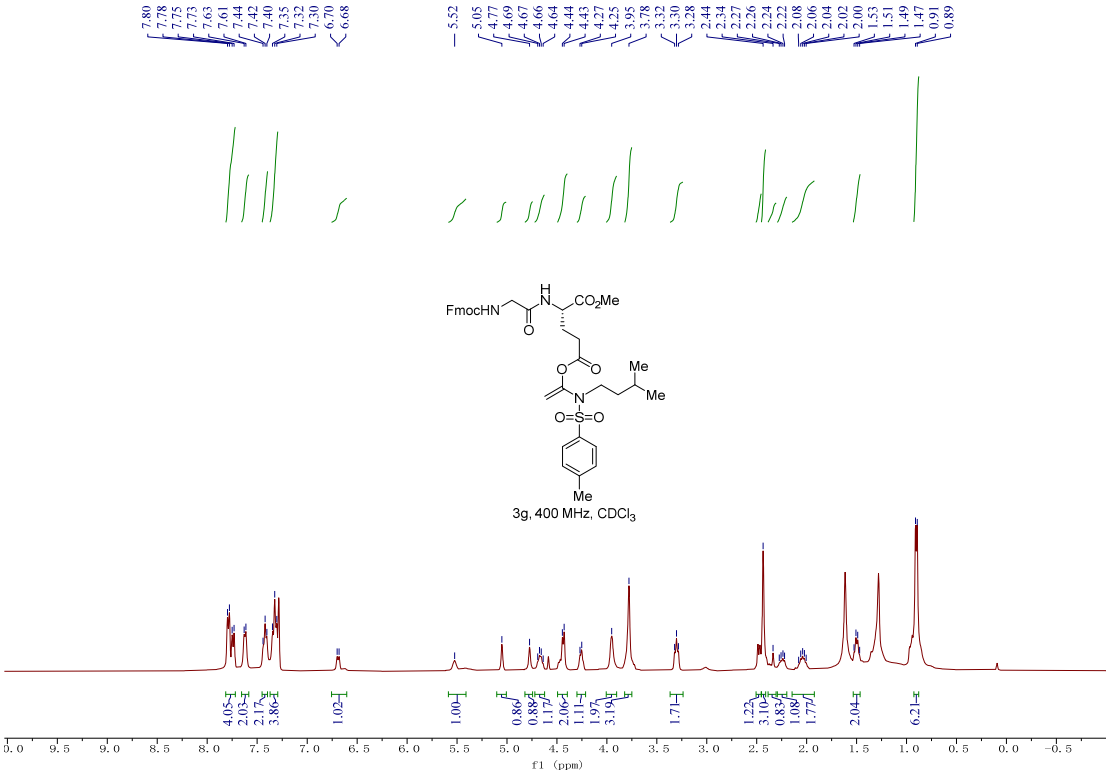


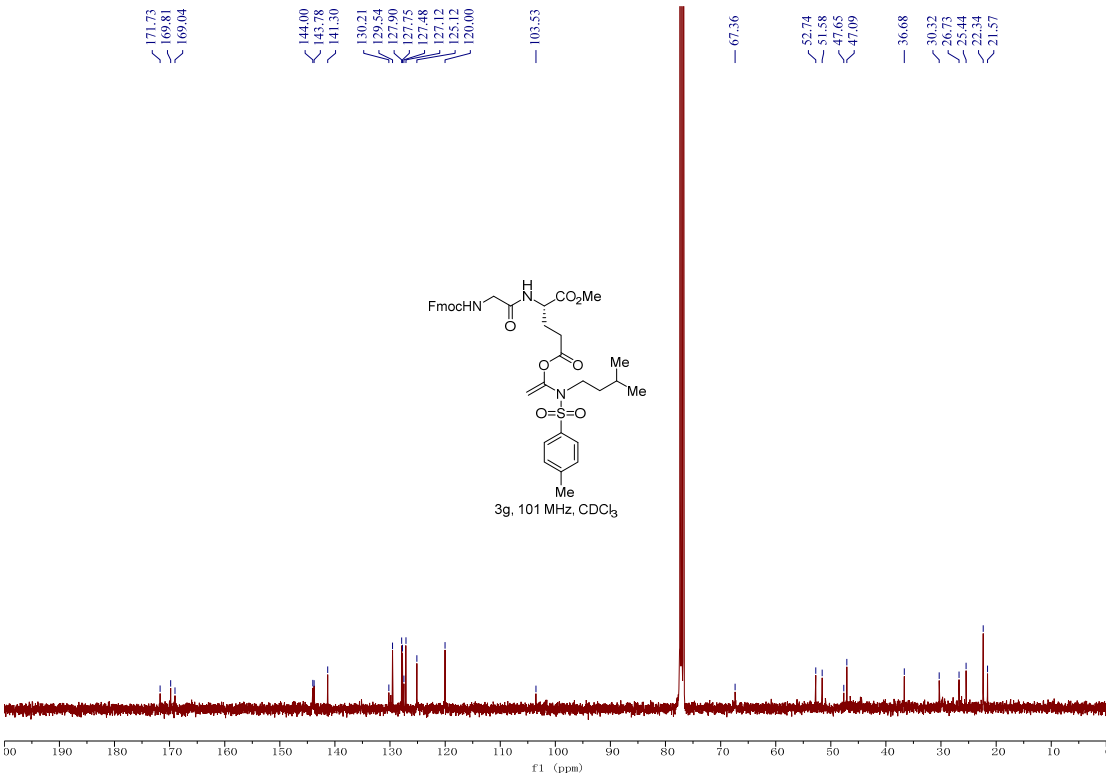


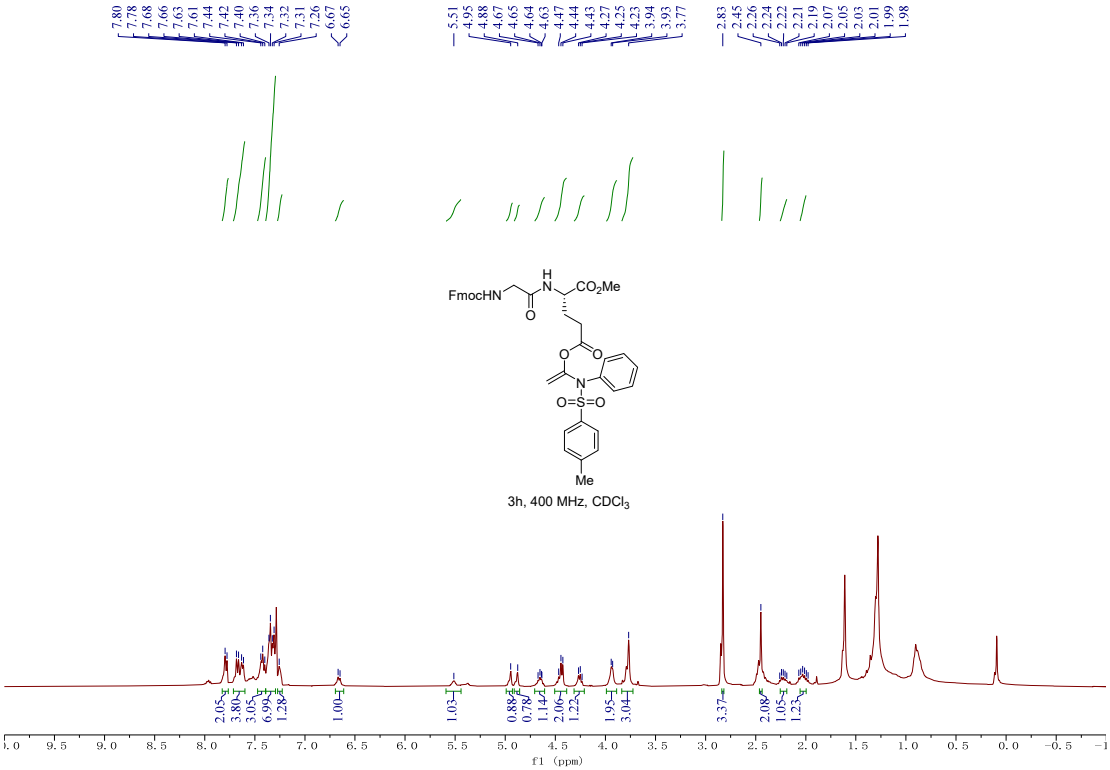
3e, 101 MHz, $CDCl_3$

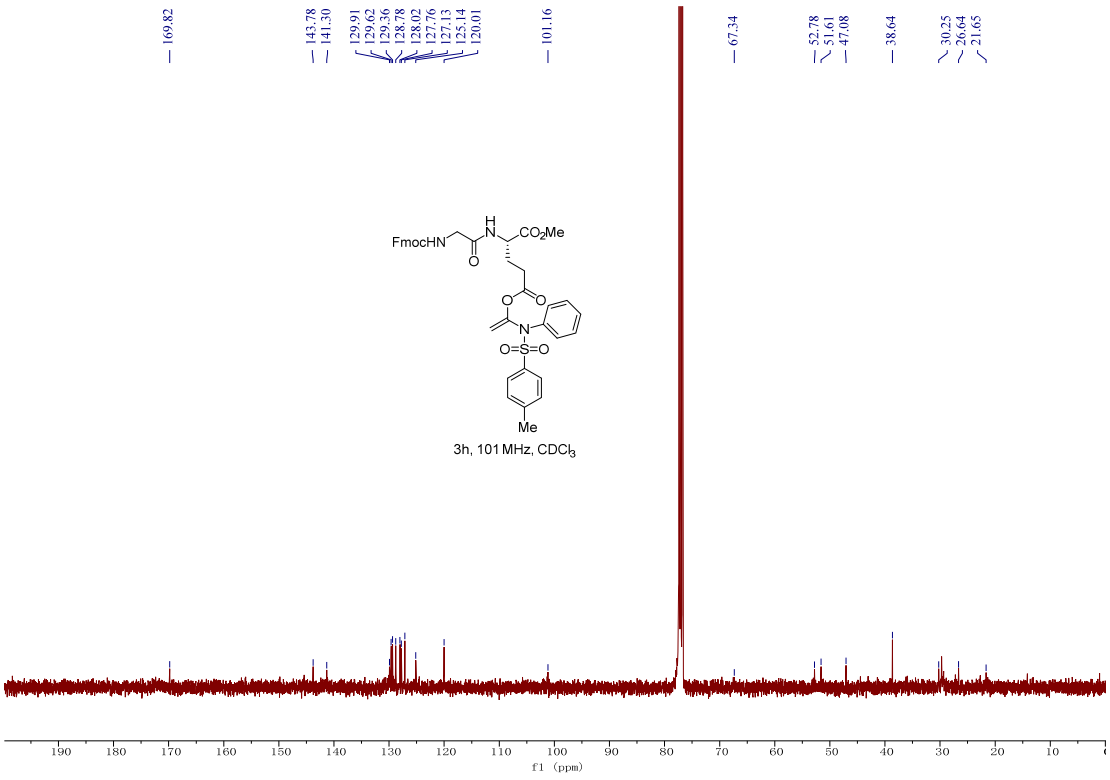

7.79
7.78
7.76
7.62
7.60
7.44
7.42
7.40
7.34
7.34
7.32
7.31
7.31
6.67
6.65
5.49
4.93
4.92
4.66
4.64
4.64
4.63
4.62
4.62
4.62
4.60
4.52
4.49
4.48
4.47
4.44
4.43
4.26
4.25
4.23
3.94
3.93
3.77
2.45
2.37
2.35
2.33
2.31
2.22
2.20
2.19
2.17
2.16
2.14
FmocHN
H
N
CO2Me
O
O
O
N
Ph
O=S=O
Me
3f, 400 MHz, CDCl3
3.93
2.14
2.31
8.97
1.00
0.95
0.84
1.89
1.15
0.87
1.76
1.09
1.94
2.97
3.27
2.05
1.11
1.33
f1 (ppm)

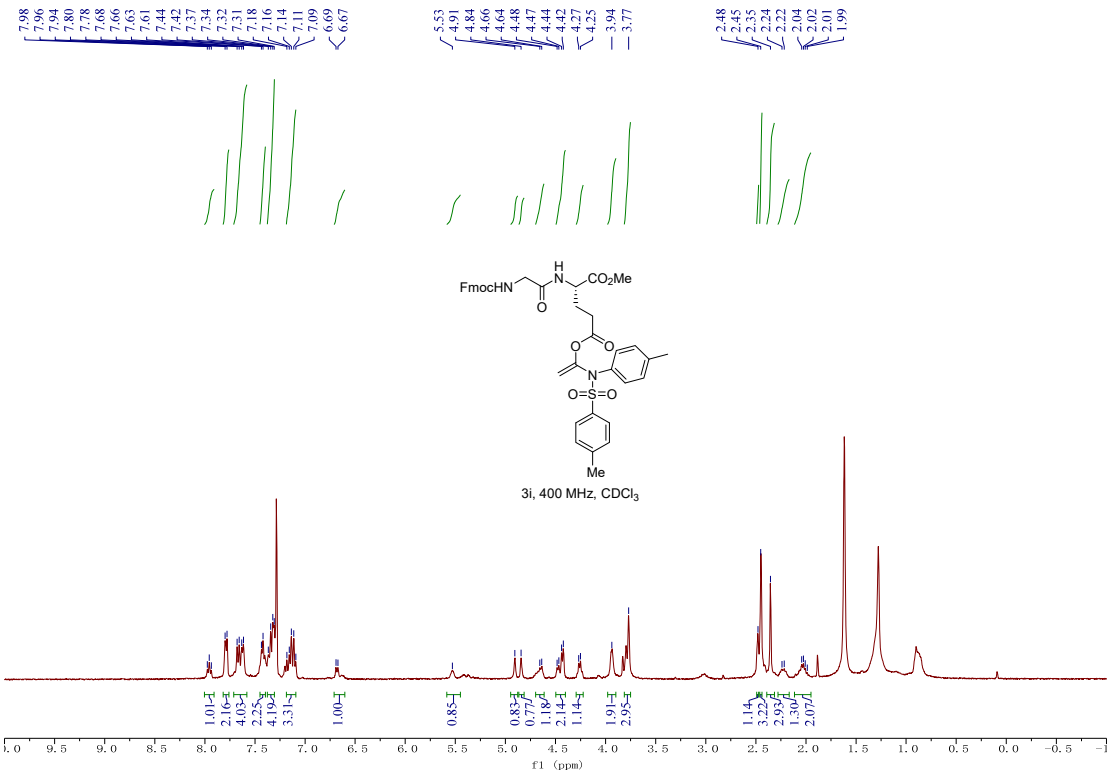

171.72
169.72
144.19
143.77
141.31
135.45
129.65
128.65
128.59
127.98
127.91
127.76
127.12
125.11
120.01
104.07
67.35
53.00
52.74
51.55
47.09
38.63
30.18
26.69
21.59
FmocHN
H
N
CO2Me
O
O
O
N
Ph
O=S=O
Me
3f, 101 MHz, CDCl3
f1 (ppm)

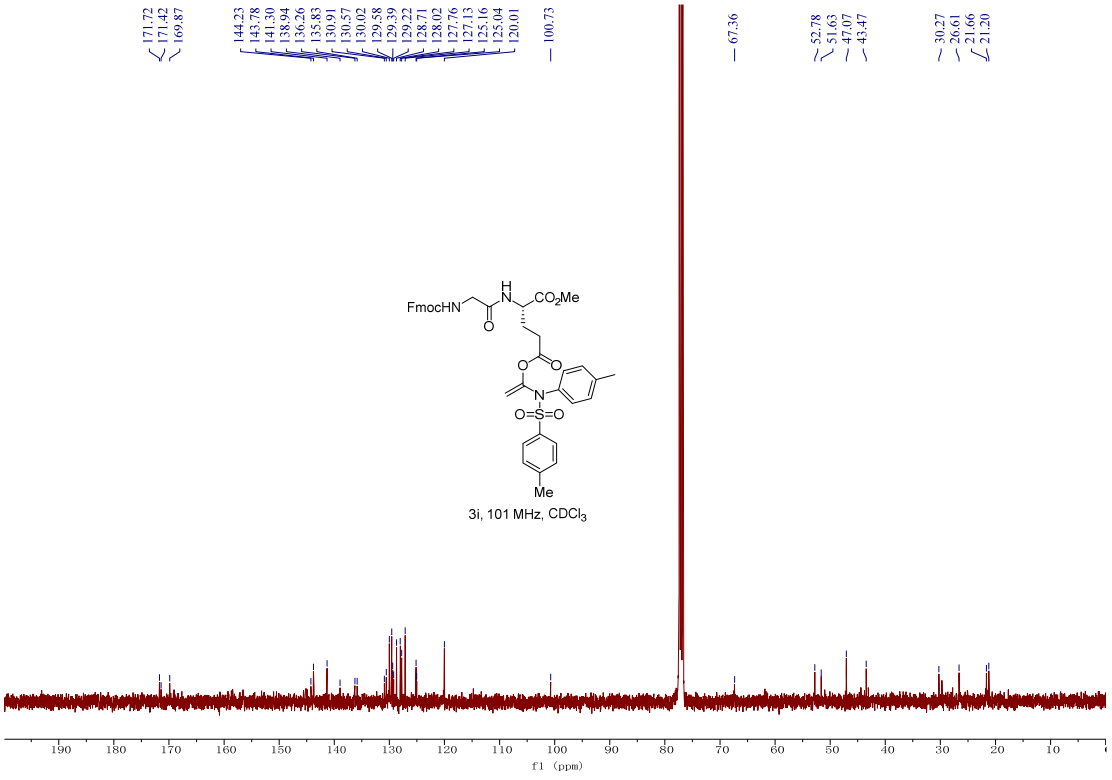

7.80 7.78 7.75 7.73 7.63 7.61 7.44 7.42 7.40 7.35 7.32 7.30 6.70 6.68
5.52 5.05 4.77 4.69 4.67 4.66 4.64 4.44 4.43 4.27 4.25 3.95 3.78 3.32 3.30 3.28 2.44 2.34 2.27 2.26 2.24 2.22 2.08 2.06 2.04 2.02 2.00 1.53 1.51 1.49 1.47 0.91 0.89
FmocHN
CO2Me
Me
Me
O=S=O
Me
3g, 400 MHz, CDCl3
4.05 2.03 2.17 3.86 1.02 1.00 0.86 0.88 1.17 2.06 1.11 1.97 3.19 1.71 1.22 3.10 0.83 1.08 1.77 2.04 6.21
f1 (ppm)

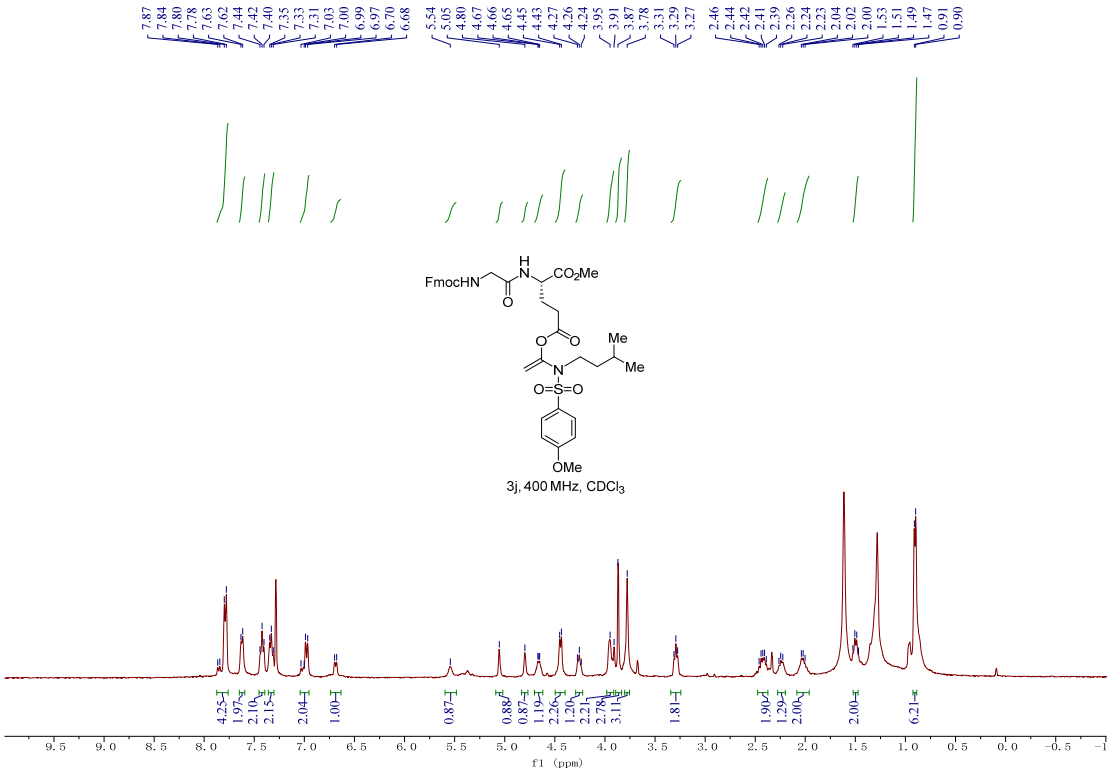

171.73 169.81 169.04
144.00 143.78 141.30 130.21 129.54 127.90 127.75 127.48 127.12 125.12 120.00
103.53
67.36
52.74 51.58 47.65 47.09
36.68
30.32 26.73 25.44 22.34 21.57
FmocHN
CO2Me
Me
Me
O=S=O
Me
3g, 101 MHz, CDCl3
f1 (ppm)

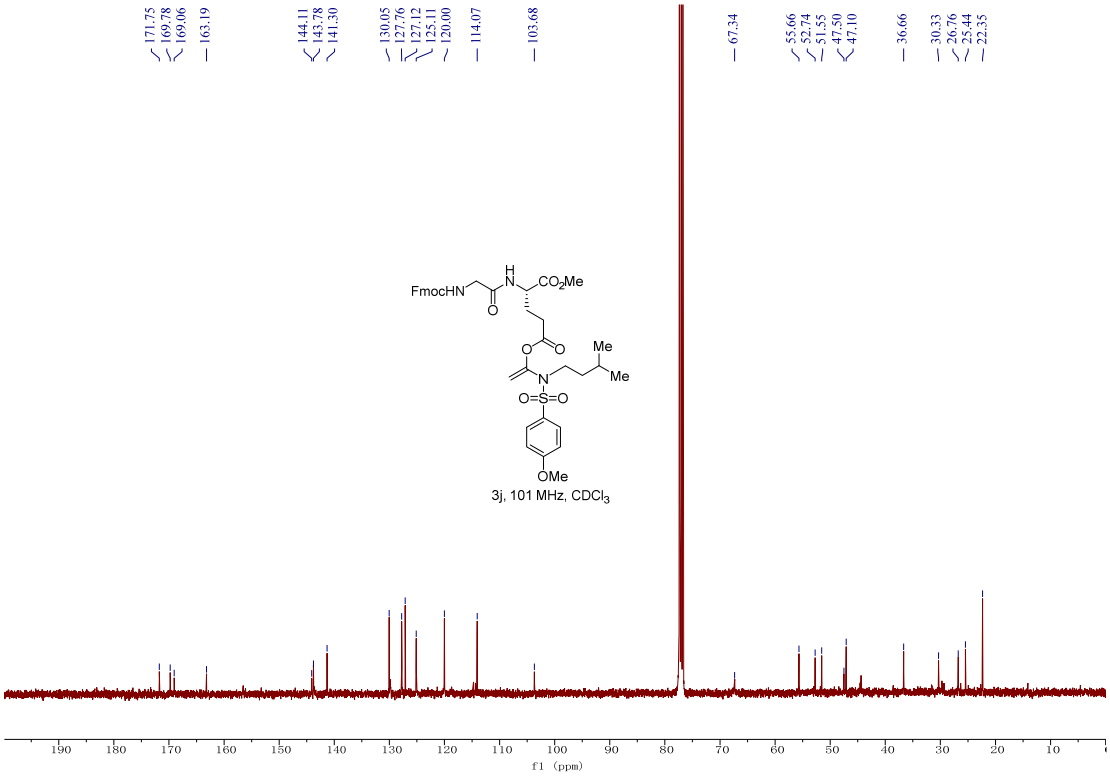

7.80
7.78
7.68
7.66
7.63
7.61
7.44
7.42
7.40
7.36
7.34
7.32
7.31
7.26
6.67
6.65
5.51
4.95
4.88
4.67
4.65
4.64
4.63
4.47
4.44
4.43
4.27
4.25
4.23
3.94
3.93
3.77
2.83
2.45
2.26
2.24
2.22
2.21
2.19
2.07
2.05
2.03
2.01
1.99
1.98
FmocHN
H
N
CO2Me
O
O
O
O
N
O=S=O
Me
3h, 400 MHz, CDCl3
2.05
3.80
3.05
6.99
1.28
1.00
1.03
0.88
0.78
1.14
2.06
1.22
1.95
3.04
3.37
2.08
1.05
1.23
f1 (ppm)

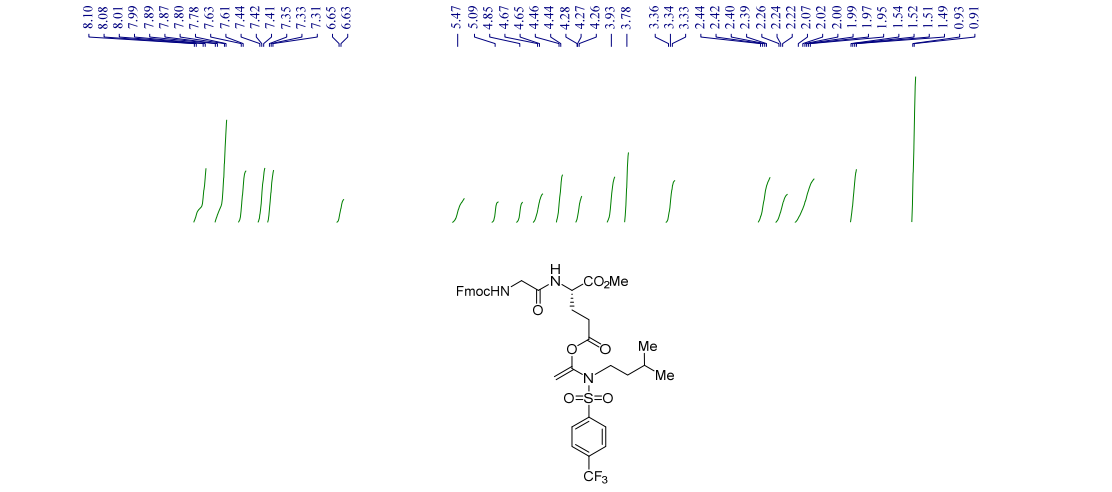

169.82
143.78
141.30
129.91
129.62
129.36
128.78
128.02
127.76
127.13
125.14
120.01
101.16
67.34
52.78
51.61
47.08
38.64
30.25
26.64
21.65
FmocHN
H
N
CO2Me
O
O
O
O
N
O=S=O
Me
3h, 101 MHz, CDCl3
f1 (ppm)


7.98
7.96
7.94
7.80
7.78
7.68
7.66
7.63
7.61
7.44
7.42
7.37
7.34
7.32
7.31
7.18
7.16
7.14
7.11
7.09
6.69
6.67
5.53
4.91
4.84
4.66
4.64
4.48
4.47
4.44
4.42
4.27
4.25
3.94
3.77
2.48
2.45
2.35
2.24
2.22
2.04
2.02
2.01
1.99
FmocHN
H
N
CO2Me
O
O
O
O
N
O=S=O
Me
3i, 400 MHz, CDCl3
1.01
2.16
4.03
2.25
4.19
3.31
1.00
0.85
0.83
0.77
1.18
2.14
1.14
1.91
2.95
1.14
3.22
2.93
1.30
2.07
f1 (ppm)

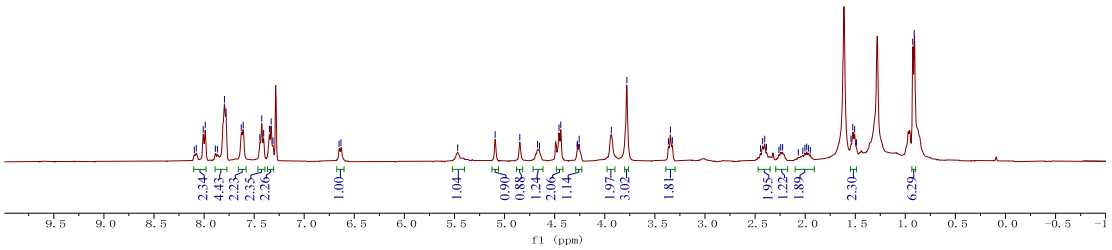

171.72
171.42
169.87
144.23
143.78
141.30
138.94
136.26
135.83
130.91
130.57
130.02
129.58
129.39
129.22
128.71
128.02
127.76
127.13
125.16
125.04
120.01
100.73
67.36
52.78
51.63
47.07
43.47
30.27
26.61
21.66
21.20
FmocHN
H
N
CO2Me
O
O
O
O
N
O=S=O
Me
3i, 101 MHz, CDCl3
f1 (ppm)

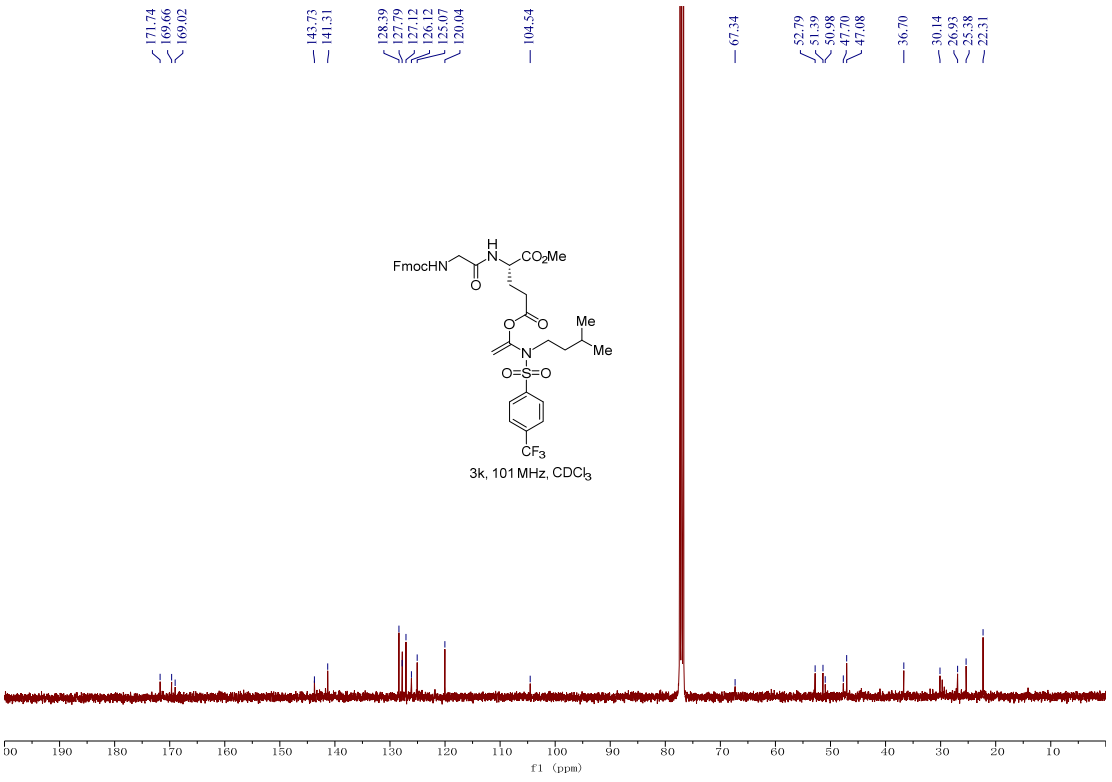

7.87 7.84 7.80 7.78 7.63 7.62 7.44 7.42 7.40 7.35 7.33 7.31 7.03 7.00 6.99 6.97 6.70 6.68
5.54 5.05 4.80 4.67 4.66 4.65 4.45 4.43 4.27 4.26 4.24 3.95 3.91 3.87 3.78 3.31 3.29 3.27
2.46 2.44 2.42 2.41 2.39 2.26 2.24 2.23 2.04 2.02 2.00 1.53 1.51 1.49 1.47 0.91 0.90
FmocHN
CO2Me
Me
Me
O=S=O
OMe
3j, 400 MHz, CDCl3
4.25 1.97 2.10 2.15 2.04 1.00 0.87 0.88 0.87 1.19 2.26 1.20 2.21 2.78 3.11 1.81 1.90 1.29 2.00 2.00 6.21
f1 (ppm)


171.75 169.78 169.06 163.19 144.11 143.78 141.30 130.05 127.76 127.12 125.11 120.00 114.07 103.68 67.34 55.66 52.74 51.55 47.50 47.10 36.66 30.33 26.76 25.44 22.35
FmocHN
CO2Me
Me
Me
O=S=O
OMe
3j, 101 MHz, CDCl3
f1 (ppm)

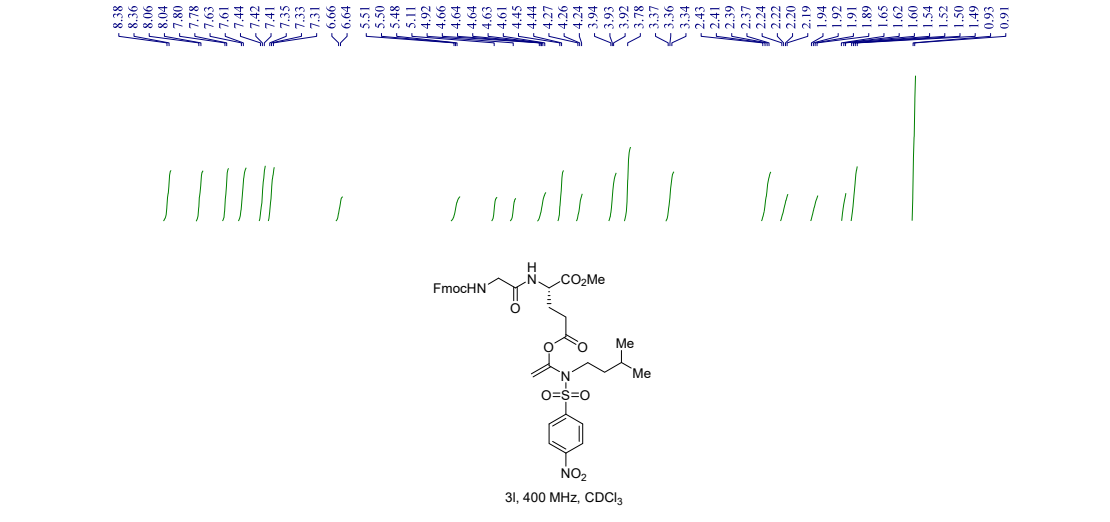


3k, 600 MHz, $CDCl_3$

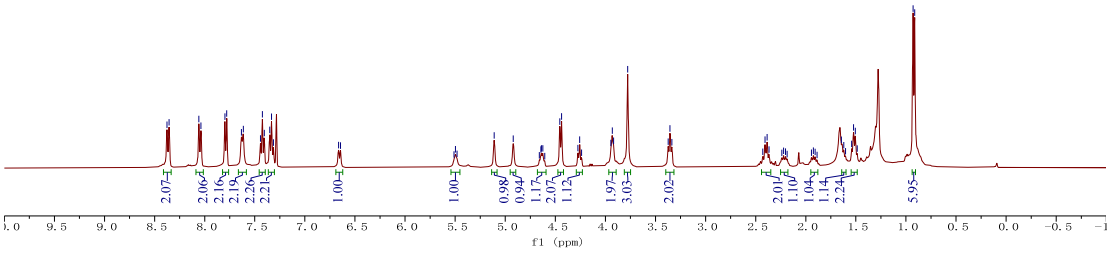


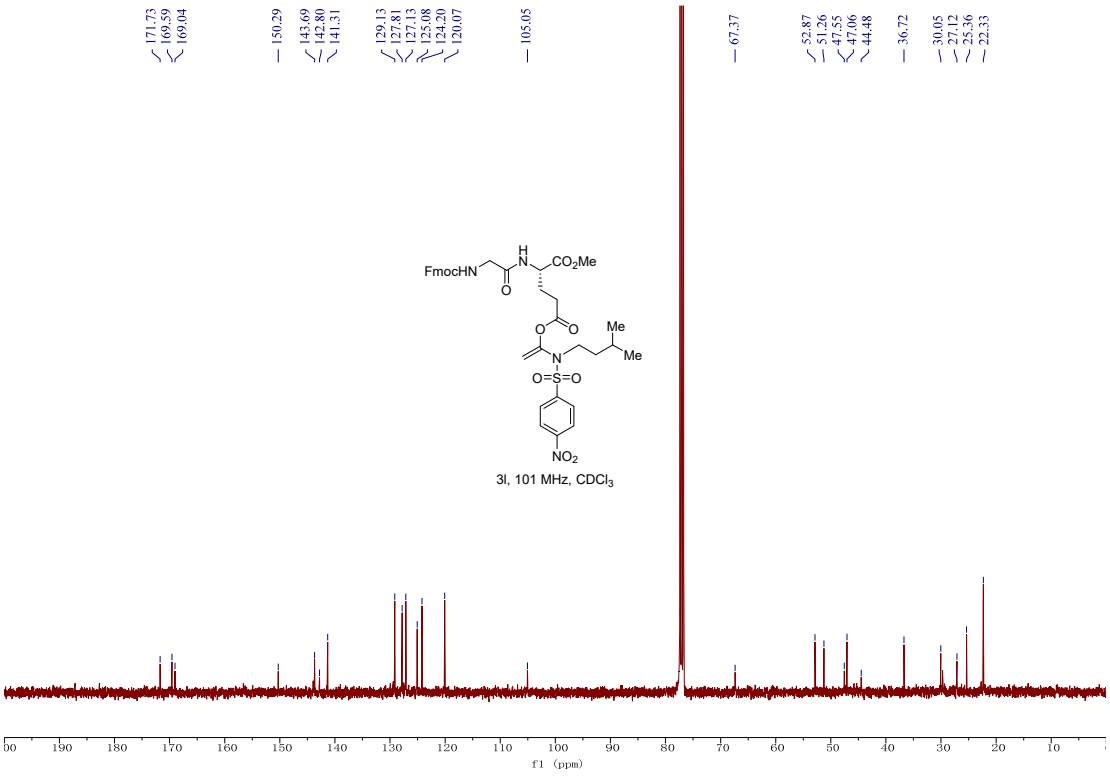


3k, 101 MHz, $CDCl_3$

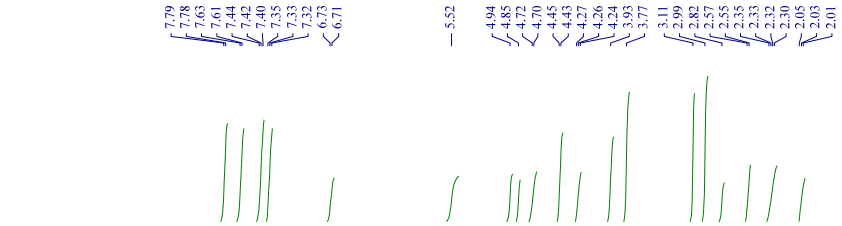


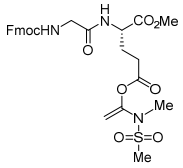
3l, 400 MHz, $CDCl_3$



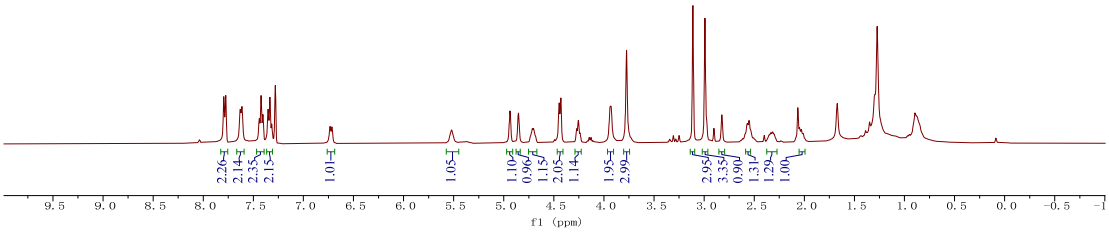


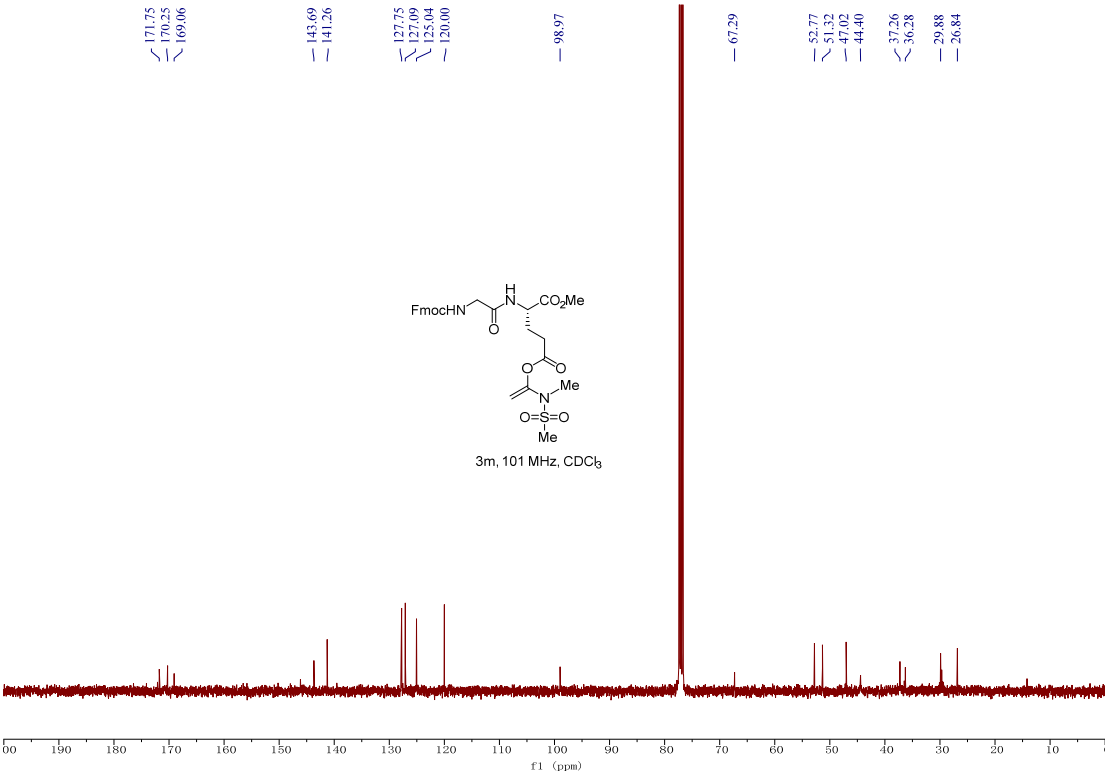
3l, 101 MHz, $CDCl_3$



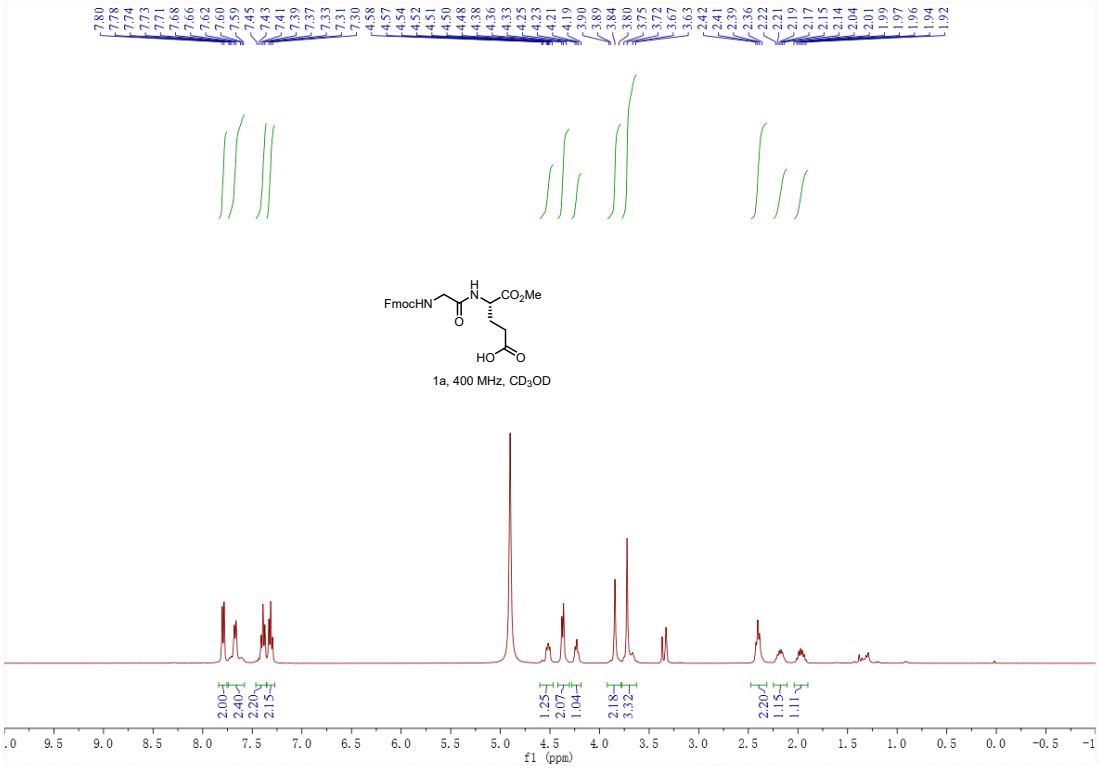


3m, 400 MHz, $CDCl_3$

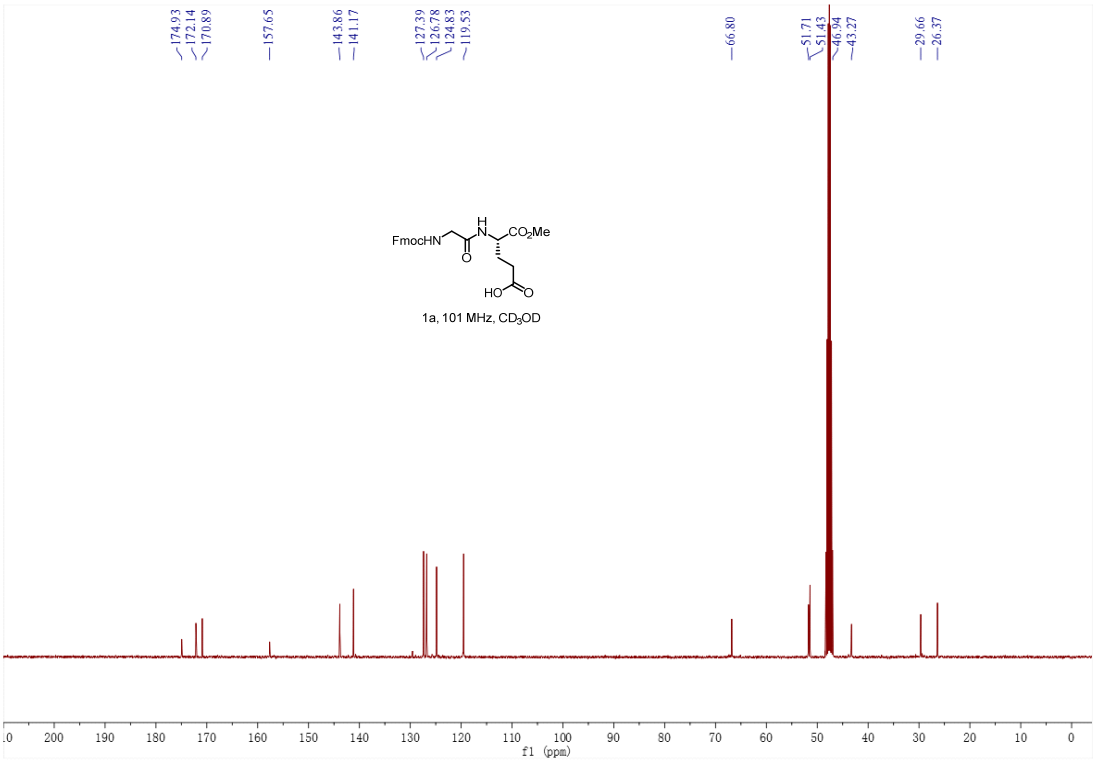




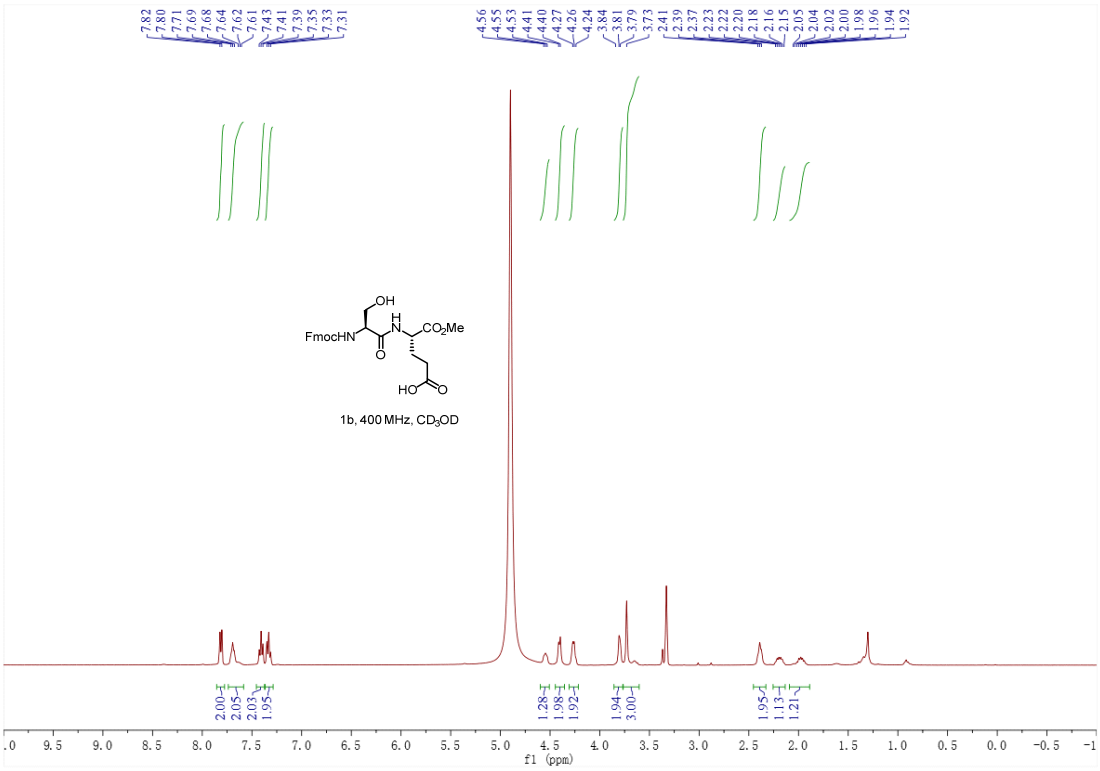
3m, 101 MHz, $CDCl_3$

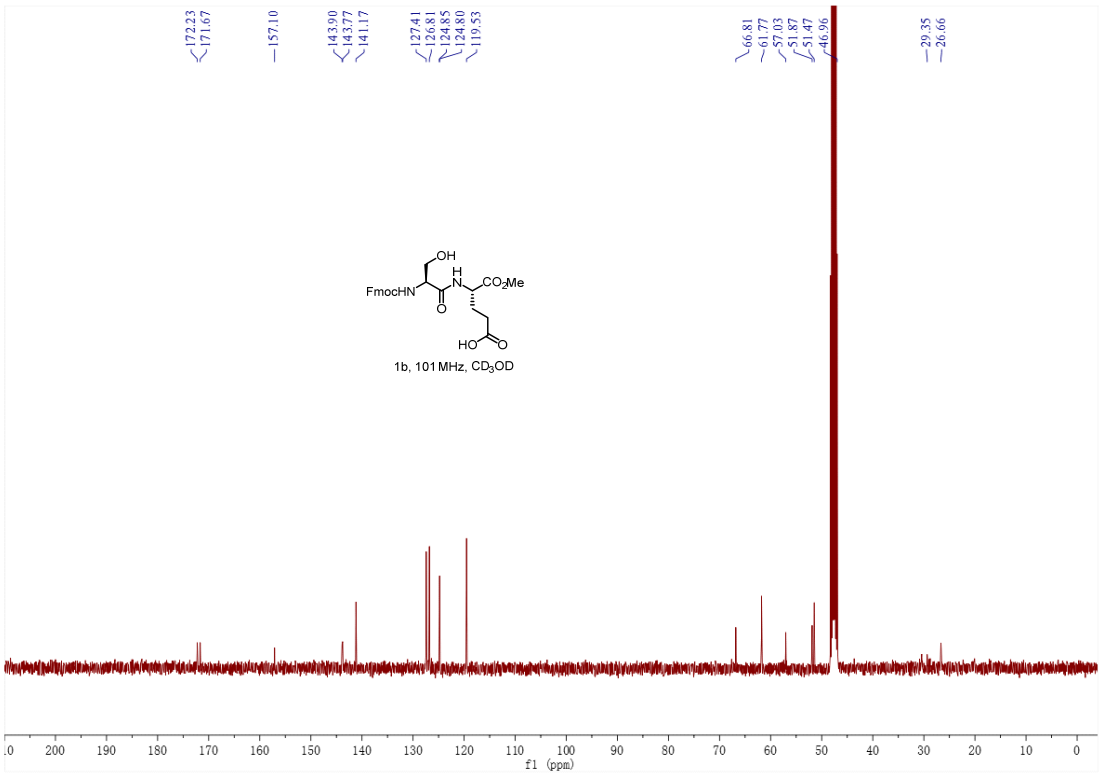


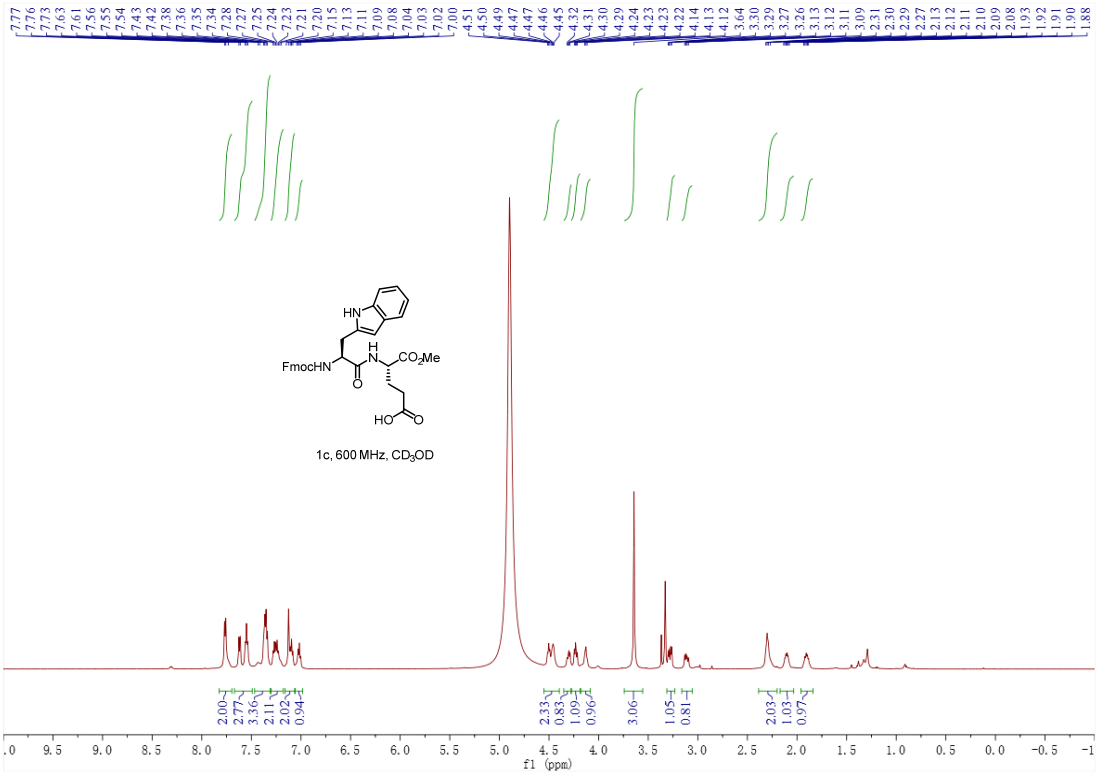
1a, 400 MHz, CD_3OD

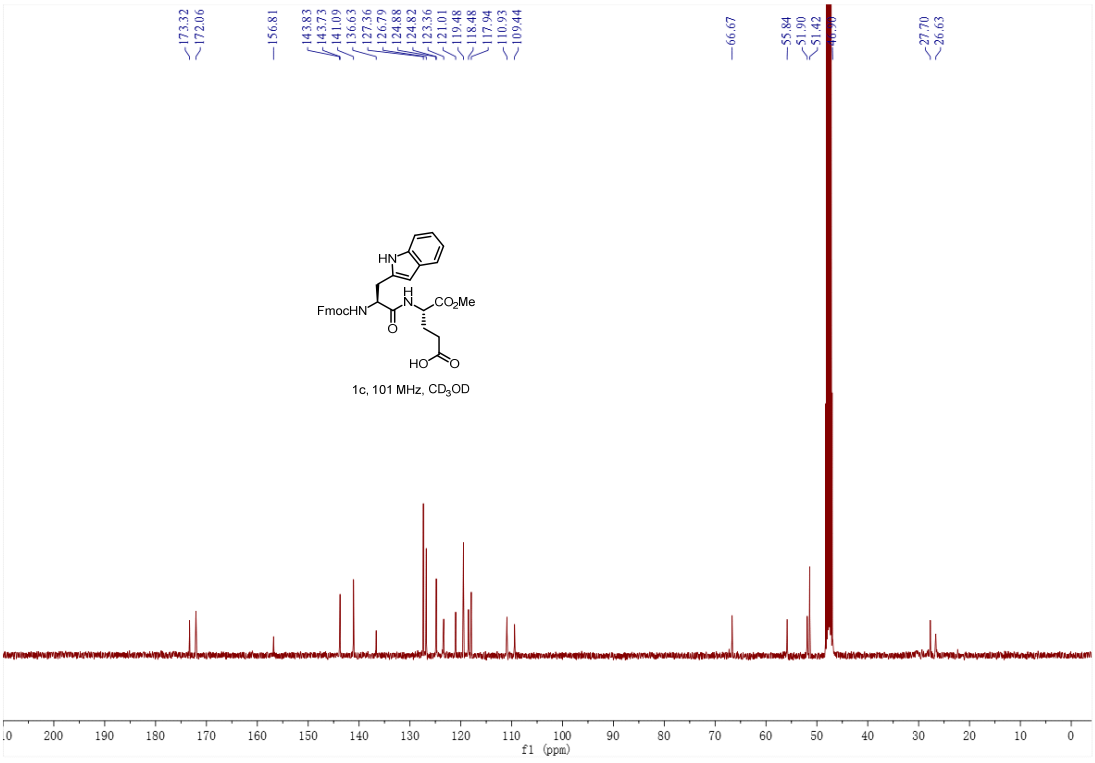


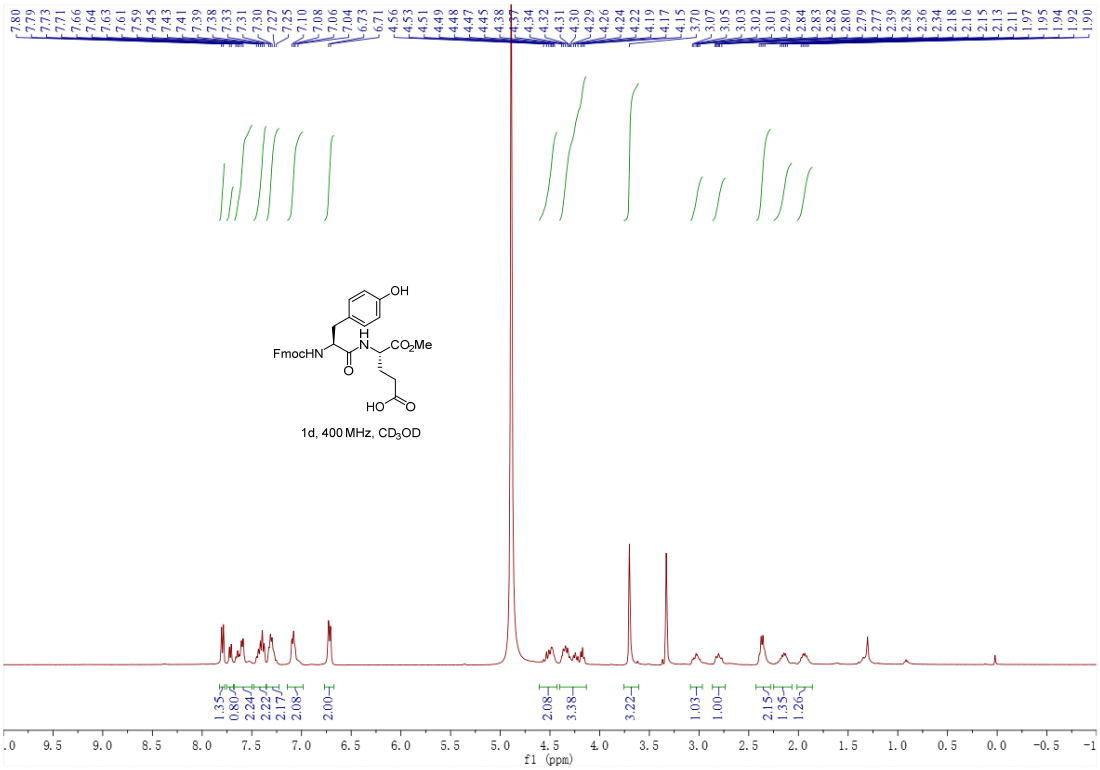
1a, 101 MHz, CD_3OD

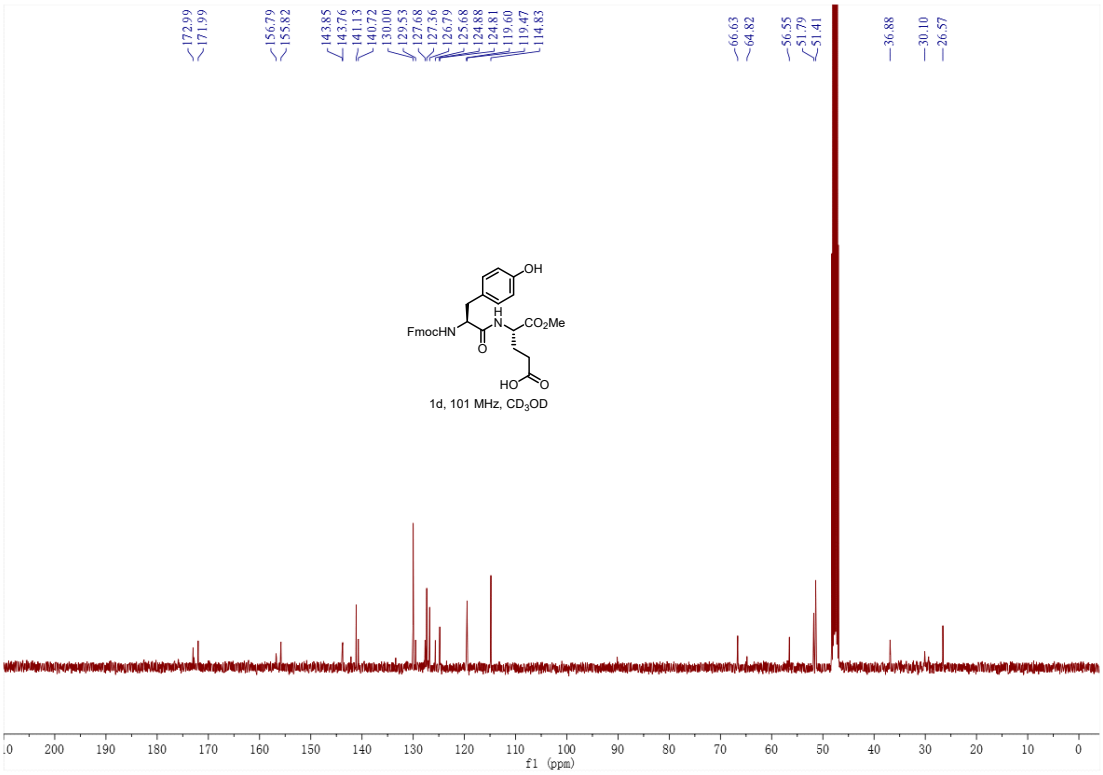

7.82
7.80
7.71
7.69
7.68
7.64
7.62
7.61
7.43
7.41
7.39
7.35
7.33
7.31
4.56
4.55
4.53
4.41
4.40
4.27
4.26
4.24
3.84
3.81
3.79
3.73
2.41
2.39
2.37
2.23
2.22
2.20
2.18
2.16
2.15
2.05
2.04
2.02
2.00
1.98
1.96
1.94
1.92
OH
FmocHN
H
N
CO2Me
O
HO
O
1b, 400 MHz, CD3OD
2.00
2.05
2.03
1.95
1.28
1.98
1.92
1.94
3.00
1.95
1.13
1.21
f1 (ppm)

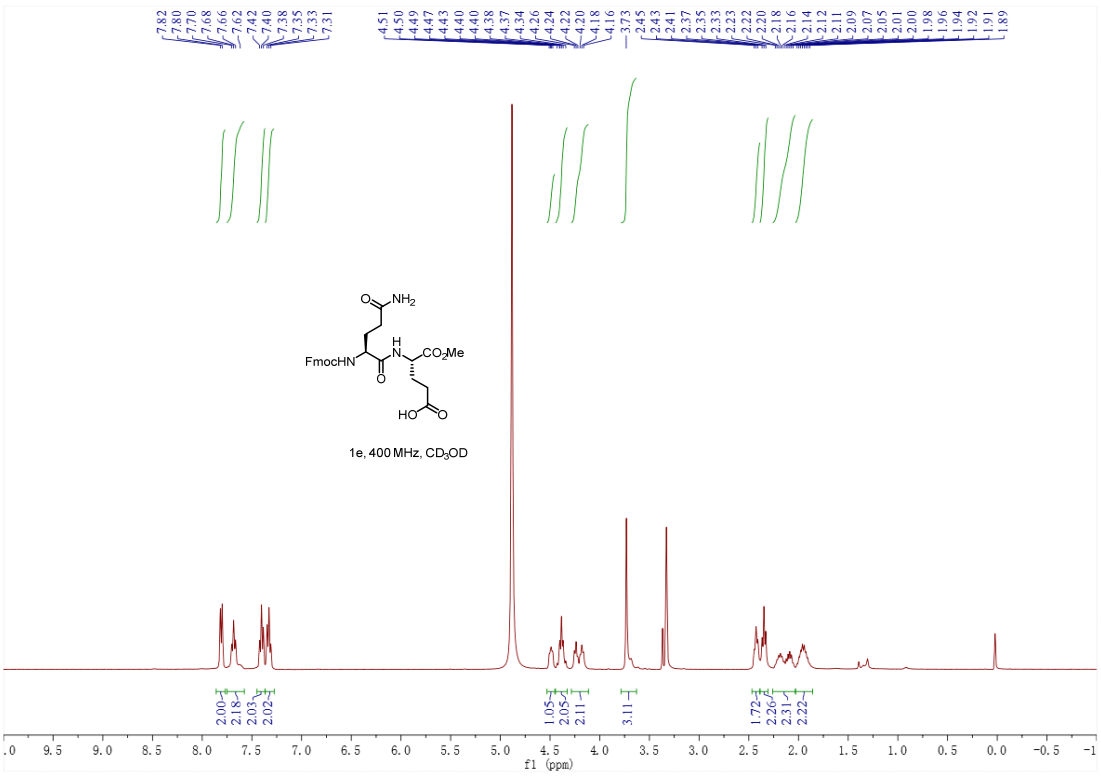

172.23
171.67
157.10
143.90
143.77
141.17
127.41
126.81
124.85
124.80
119.53
66.81
61.77
57.03
51.87
51.47
46.96
29.35
26.66
OH
FmocHN
H
N
CO2Me
O
HO
O
1b, 101 MHz, CD3OD
f1 (ppm)

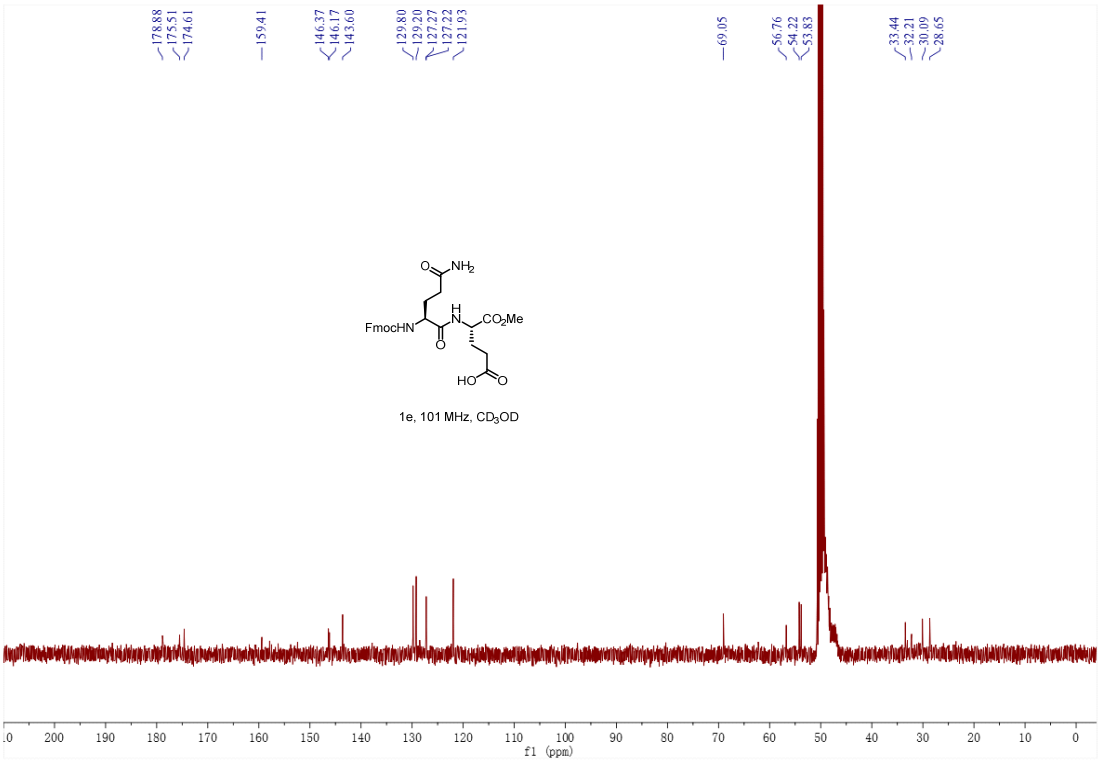

FmocHN
HN
CO2Me
HO
O
1c, 600 MHz, CD3OD
f1 (ppm)

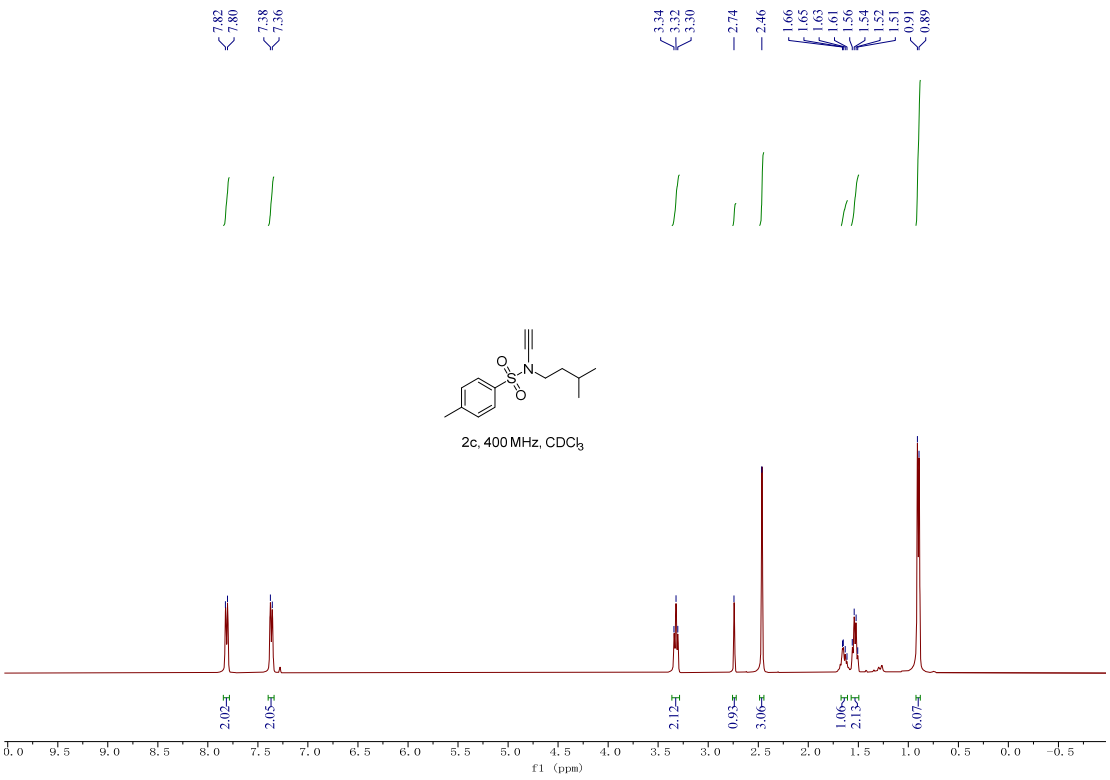

FmocHN
HN
CO2Me
HO
O
1c, 101 MHz, CD3OD
f1 (ppm)

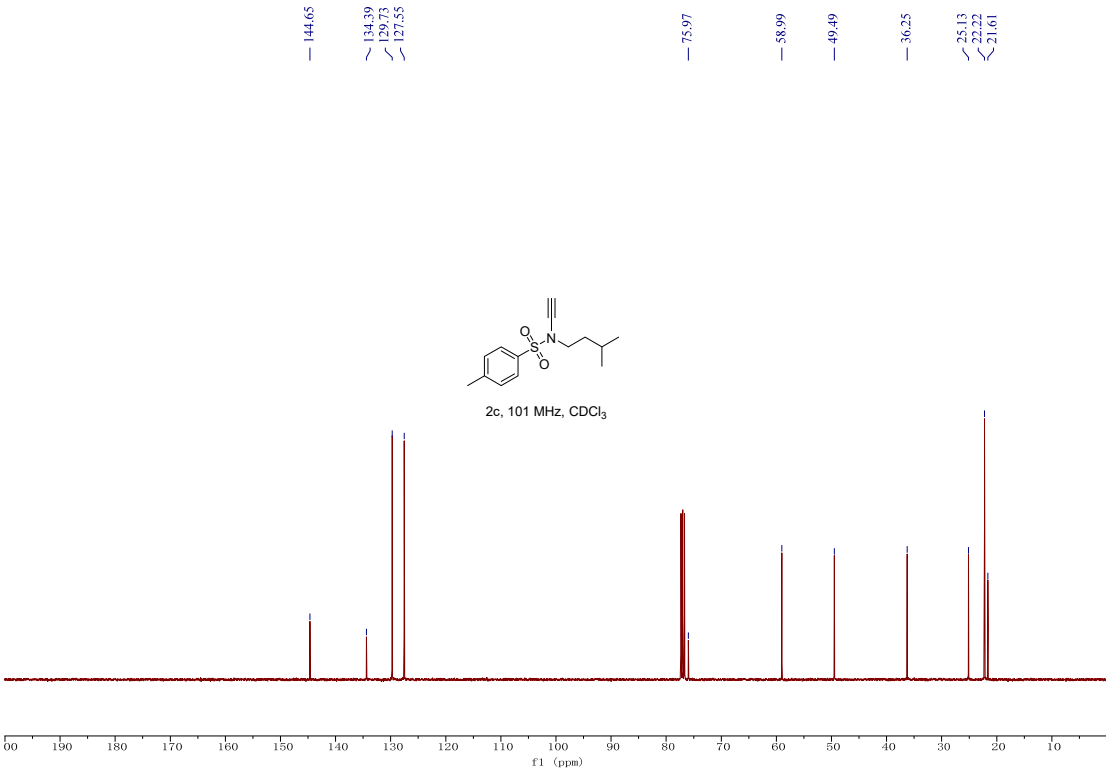

OH
FmocHN
H
N
CO2Me
O
HO
O
1d, 400 MHz, CD3OD
f1 (ppm)

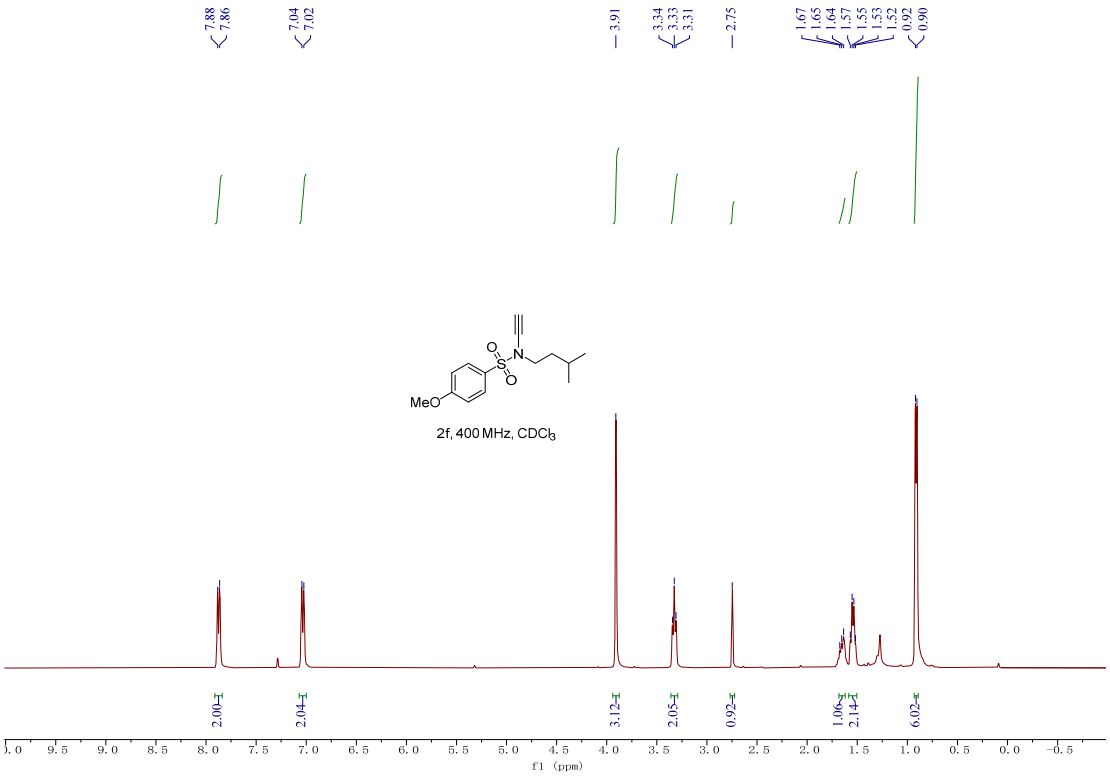

OH
FmocHN
H
N
CO2Me
O
HO
O
1d, 101 MHz, CD3OD
f1 (ppm)


7.82
7.80
7.70
7.68
7.66
7.62
7.42
7.40
7.38
7.35
7.33
7.31
4.51
4.50
4.49
4.47
4.43
4.40
4.40
4.38
4.37
4.34
4.26
4.24
4.22
4.20
4.18
4.16
3.73
2.45
2.43
2.41
2.37
2.35
2.33
2.23
2.22
2.20
2.18
2.16
2.14
2.12
2.11
2.09
2.07
2.05
2.01
2.00
1.98
1.96
1.94
1.92
1.91
1.89
NH2
O
H
N
CO2Me
FmocHN
HO
O
1e, 400 MHz, CD3OD
2.00
2.18
2.03
2.02
1.05
2.05
2.11
3.11
1.72
2.26
2.31
2.22
f1 (ppm)

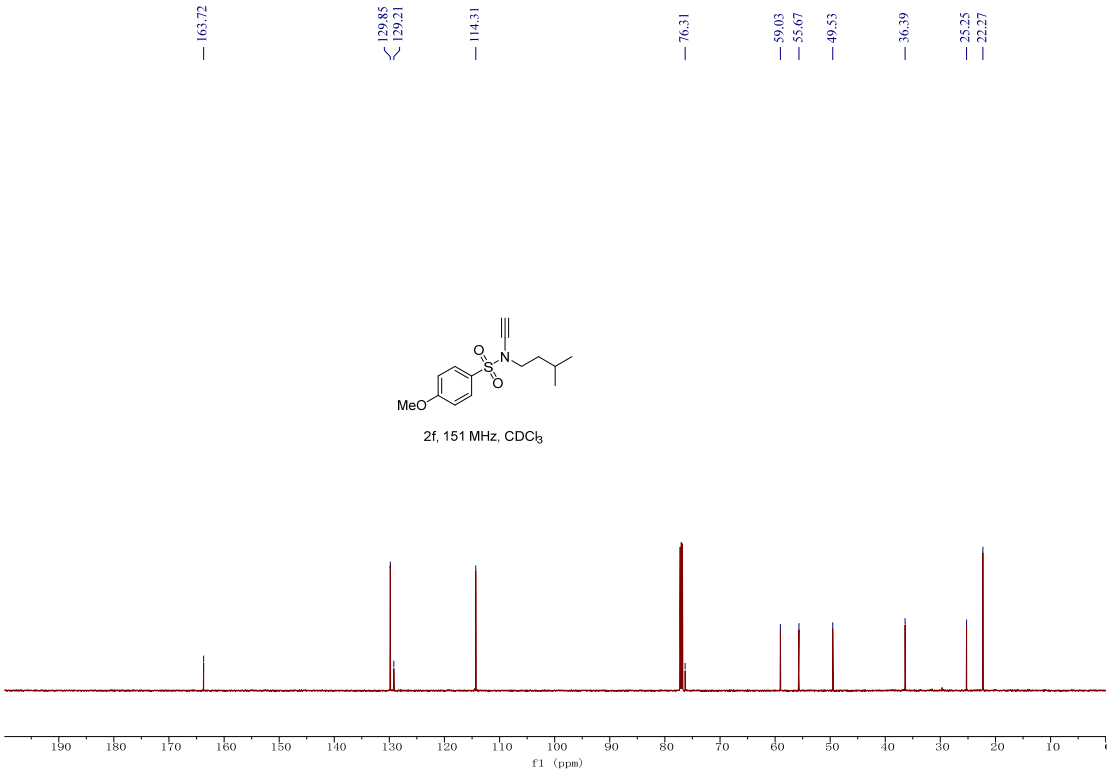

178.88
175.51
174.61
159.41
146.37
146.17
143.60
129.80
129.20
127.27
127.22
121.93
69.05
56.76
54.22
53.83
33.44
32.21
30.09
28.65
NH2
O
H
N
CO2Me
FmocHN
HO
O
1e, 101 MHz, CD3OD
f1 (ppm)


7.82
7.80
7.38
7.36
3.34
3.32
3.30
2.74
2.46
1.66
1.65
1.63
1.61
1.56
1.54
1.52
1.51
0.91
0.89
2c, 400 MHz, CDCl3
2.02
2.05
2.12
0.93
3.06
1.06
2.13
6.07
f1 (ppm)

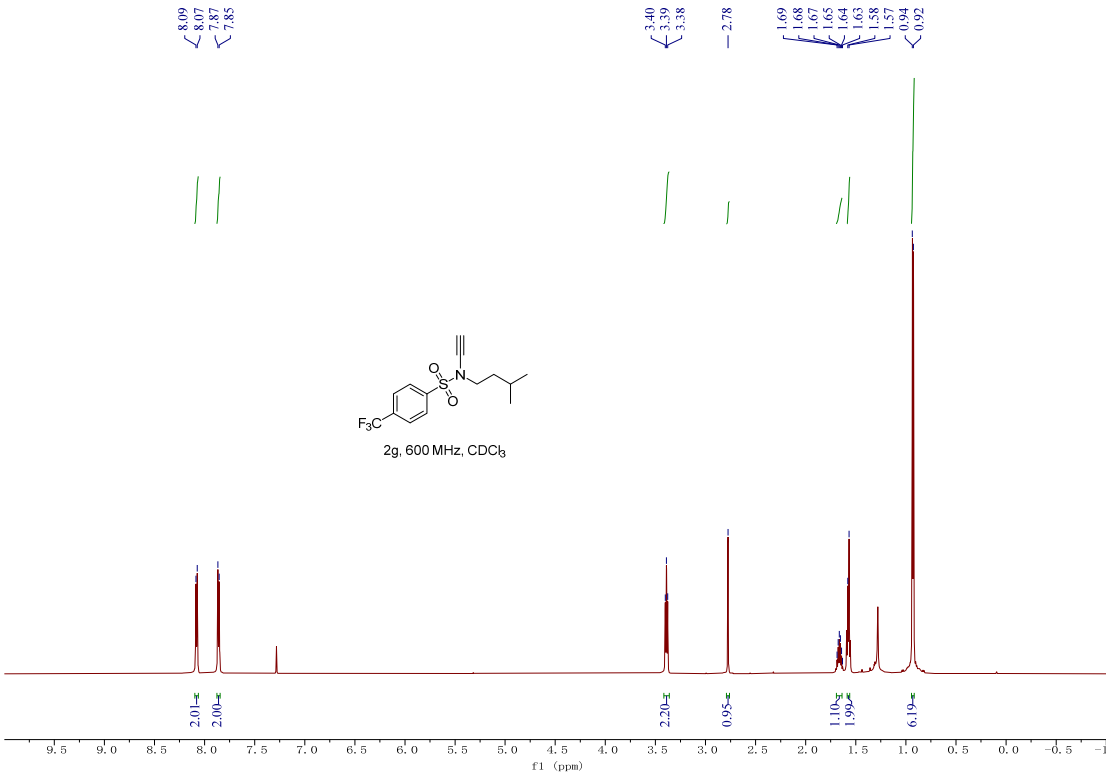

144.65
134.39
129.73
127.55
75.97
58.99
49.49
36.25
25.13
22.22
21.61
2c, 101 MHz, CDCl3
f1 (ppm)

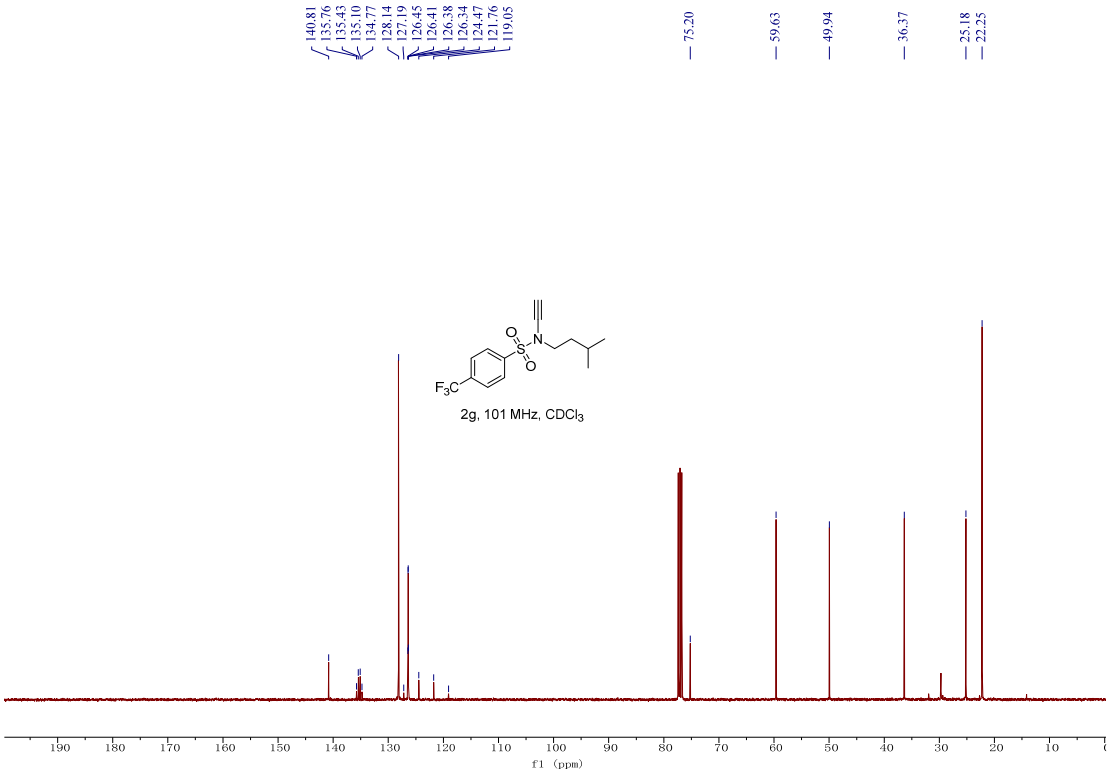


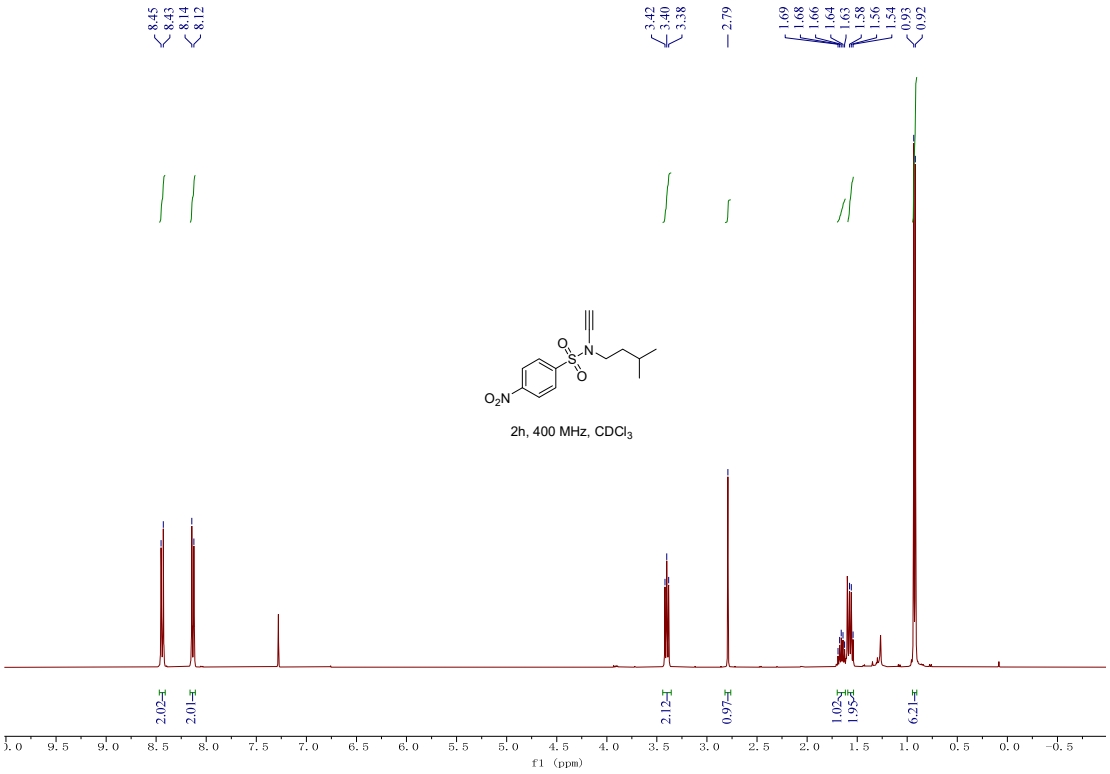
2f, 400 MHz, $CDCl_3$



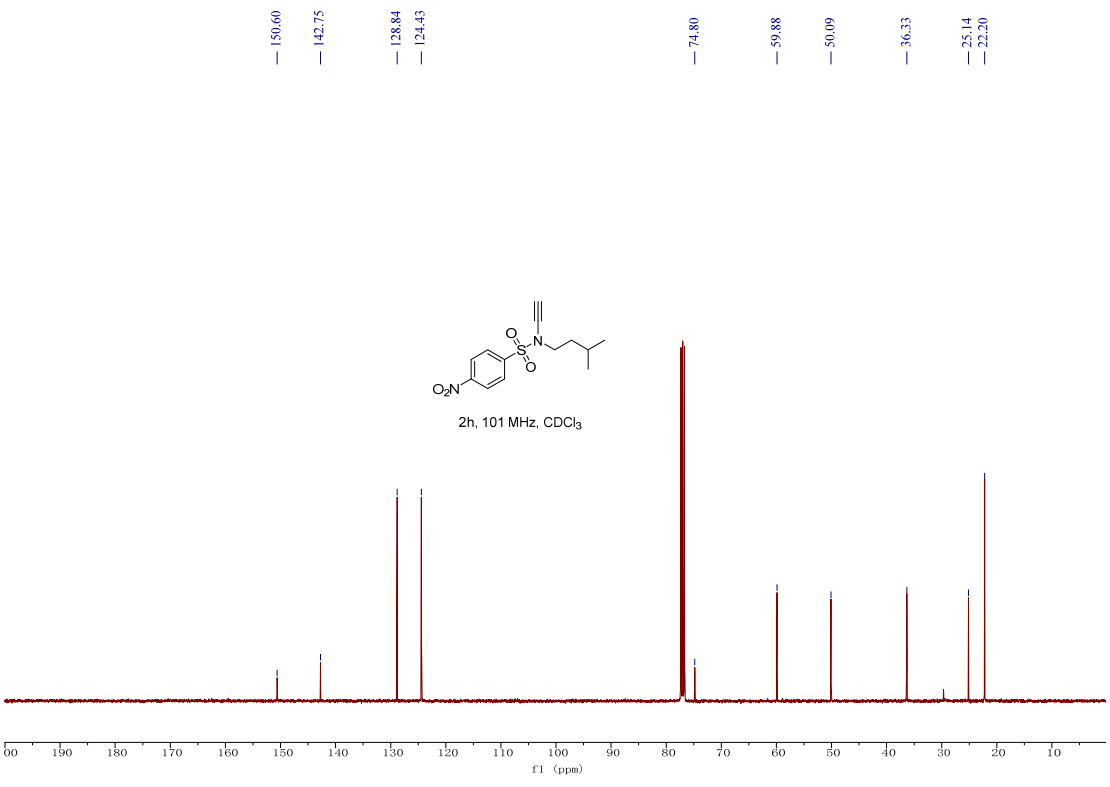
2f, 151 MHz, $CDCl_3$


8.09
8.07
7.87
7.85
3.40
3.39
3.38
2.78
1.69
1.68
1.67
1.65
1.64
1.63
1.58
1.57
0.94
0.92
F3C
2g, 600 MHz, CDCl3
2.01
2.00
2.20
0.95
1.10
1.99
6.19
f1 (ppm)


140.81
135.76
135.43
135.10
134.77
128.14
127.19
126.45
126.41
126.38
126.34
124.47
121.76
119.05
75.20
59.63
49.94
36.37
25.18
22.25
F3C
2g, 101 MHz, CDCl3
f1 (ppm)



2h, 400 MHz, $CDCl_3$



2h, 101 MHz, $CDCl_3$