Supporting Information

Copper(II) carboxymethylcellulose (CMC-Cu^{II}) as an efficient catalyst for Aldehyde-Alkyne-Amine coupling under solvent-free conditions

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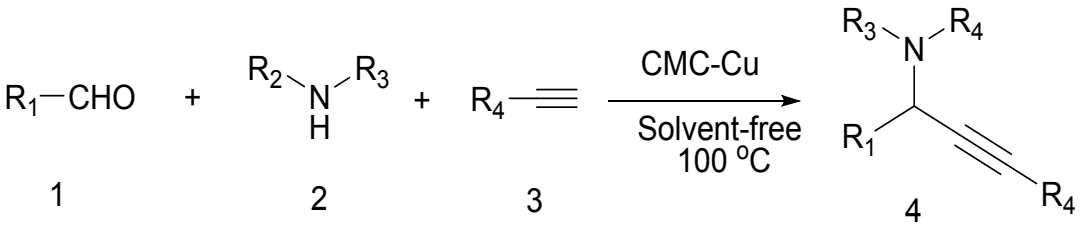

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1. General Considerations

FT-IR spectra were recorded on a Nicolet 6700 spectrometer as KBr pellets in the range 4000−400 cm^{-1}. 1H NMR (300MHz) and 13C NMR (75MHz) spectra were obtained with a Bruker Avance instrument with $CDCl_3$ as solvent and TMS as internal standard. HRMS data were recorded using AB SCIEX Triple TOF 5600+ detection. The elemental copper content of polymeric catalysts was determined by PerkinElmer Optima 2000DV inductively coupled plasma-atomic emission spectrometry (ICP-AES). X-ray photoelectron spectroscopy (XPS) measurements were performed on a Kratos Axis Ultra ^{DLD} X-ray photoelectron spectrometer with an Al Kα excitation. Scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) were performed with JEM-2100F instrument. All chemicals were obtained from commercial sources and used as received. All chemicals were obtained from commercial sources and used as received.

2. Preparation of copper (II) carboxymethylcellulose (CMC-Cu^{II})

The 1 wt % aqueous solution of sodium carboxymethylcellulose was slowly added dropwise to an aqueous solution containing 10 wt % $CuSO_4{\cdot}5H_2O$ with constantly stirring at room temperature. The blue solid was precipitated immediately and further left equilibrate in solution for 6 h. The resulting solid was separated from the solution by suction and washed thoroughly with distilled water, then dried in vacuum to constant weight to provide the CMC-Cu^{II} as blue powder. The Cu content was determined to be 2.146 mmol/g by ICP-AES.

3. General procedure for A3-coupling reaction

A mixture of aldehyde (1.0mmol), amine (1.2mmol) and phenylacetylene (1.5mmol) and a catalytic amount of CMC-Cu^{II} (5 mol %) was stirred in neat at 100°C under air for 15h. After the completion of reaction, 30 mL EtOAc was added to dissolve the product. The catalyst was separated by filtration and the filtrate was washed with brine and dried over anhydrous Na_2SO_4. The product was obtained after removal of the organic fractions using a rotary evaporator. The desired pure products were further purified by preparative TLC. All the products except 4i, 4r, 4s, 4w, 4x, 4y and 4z are known, and their ^{1}H NMR data were found to be identical to those reported in the literature. The new compounds were fully characterized by FT-IR, ^{1}H NMR, ^{13}C NMR, Ms, and HR-Ms.

4. FT-IR of CMC-Cu^{II}

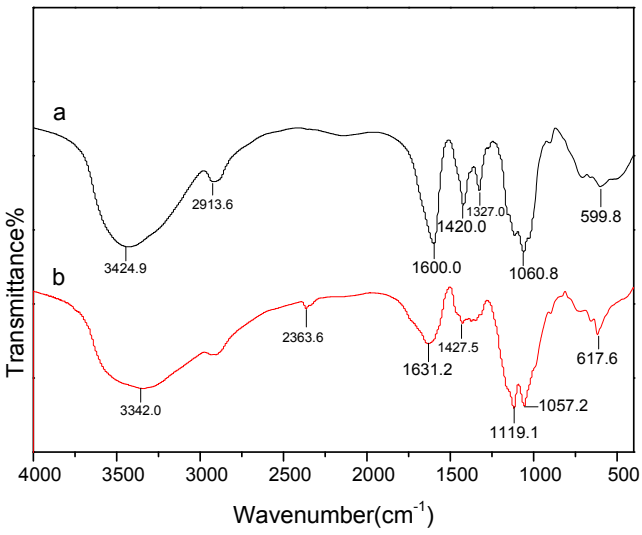
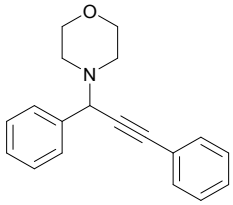
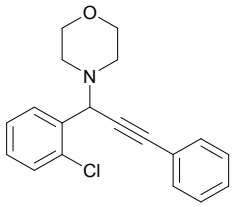


Figure 1. FT-IR spectra of CMC-Na (a), CMC-Cu^{II} (b)

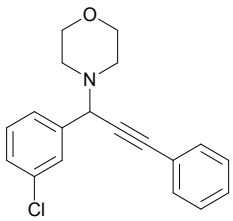
5. Characterization data for A3-coupling products



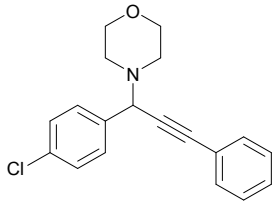
4-(1, 3-diphenylprop-2-yn-1-yl)morpholine (4a). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.4 Hz, 2H), 7.53 (dd, *J* = 5.9, 3.0 Hz, 2H), 7.37 (dd, *J* = 15.0, 6.1 Hz, 6H), 4.81 (s, 1H), 3.75 (q, *J* = 4.1 Hz, 4H), 2.66 (t, J = 4.7 Hz, 4H).



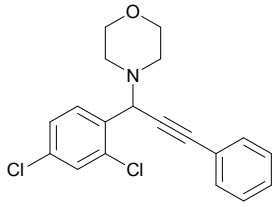
4-(1-(2-chlorophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4b). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83 – 7.76 (m, 1H), 7.53 (ddt, *J* = 5.6, 3.0, 1.6 Hz, 2H), 7.46 – 7.42 (m, 1H), 7.36 (ddt, *J* = 5.9, 4.2, 2.3 Hz, 3H), 7.33 – 7.26 (m, 2H), 5.17 (s, 1H), 3.74 (q, *J* = 4.6 Hz, 4H), 2.71 (t, *J* = 4.7 Hz, 4H).



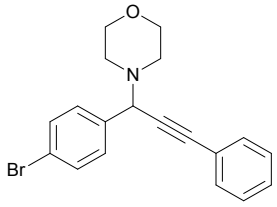
4-(1-(3-chlorophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4c). ^{1}H NMR(300 MHz, $CDCl_3$) δ 7.66 (p, *J* = 1.1 Hz, 1H), 7.57 – 7.50 (m, 3H), 7.37 – 7.32 (m, 3H), 7.32 – 7.26 (m, 2H), 4.78 (s, 1H), 3.75 (td, *J* = 4.3, 2.9 Hz, 4H), 2.64 (t, *J* = 4.7 Hz, 4H).



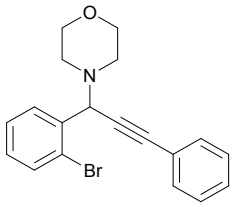
4-(1-(4-chlorophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4d). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 – 7.55 (m, 2H), 7.52 (dtd, *J* = 5.7, 4.1, 2.5 Hz, 2H), 7.40 – 7.29 (m, 5H), 4.77 (s, 1H), 3.74 (q, *J* = 4.2 Hz, 4H), 2.63 (t, *J* = 4.7 Hz, 4H).



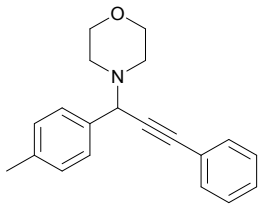
4-(1-(2, 4-dichlorophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4e). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.3 Hz, 1H), 7.55 – 7.48 (m, 2H), 7.46 (d, *J* = 2.1 Hz, 1H), 7.40 – 7.33 (m, 3H), 7.29 (dd, *J* = 8.3, 2.2 Hz, 1H), 5.08 (s, 1H), 3.73 (q, *J* = 4.9 Hz, 4H), 2.68 (t, *J* = 4.7 Hz, 4H).



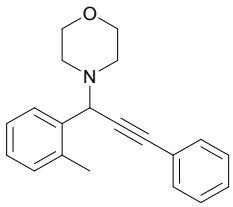
4-(1-(4-bromophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4f). ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.56 – 7.47 (m, 6H), 7.38 – 7.31 (m, 3H), 4.75 (s, 1H), 3.73 (q, *J* = 4.2 Hz, 4H), 2.62 (t, *J* = 4.6 Hz, 4H).



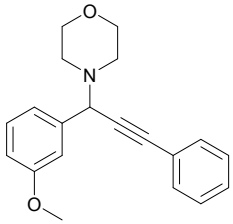
4-(1-(2-bromophenyl)-3-phenylprop-2-yn-1-yl)morpholine (4g). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.61 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.54 – 7.48 (m, 2H), 7.37 – 7.31 (m, 4H), 7.18 (td, *J* = 7.6, 1.8 Hz, 1H), 5.08 (s, 1H), 3.77 – 3.65 (m, 4H), 2.74 – 2.63 (m, 4H).



4-(3-phenyl-1-(p-tolyl) prop-2-yn-1-yl)morpholine (4h). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55 – 7.50 (m, 4H), 7.34 (dp, *J* = 4.6, 1.7 Hz, 3H), 7.19 (d, *J* = 7.9 Hz, 2H), 4.77 (s, 1H), 3.81 – 3.70 (m, 4H), 2.66 (tq, *J* = 5.2, 2.9 Hz, 4H), 2.37 (s, 3H).

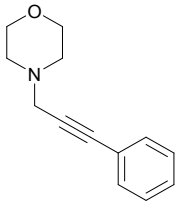


4-(3-phenyl-1-(o-tolyl) prop-2-yn-1-yl)morpholine(4i). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (dd, *J* = 7.3, 2.5 Hz, 1H), 7.57 – 7.50 (m, 2H), 7.38 – 7.31 (m, 3H), 7.22 (dd, *J* = 5.7, 3.4 Hz, 3H), 4.89 (s, 1H), 3.71 (p, *J* = 5.8, 5.2 Hz, 4H), 2.65 (t, *J* = 4.7 Hz, 4H), 2.50 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.63 , 135.88 , 131.90 , 130.83 , 129.14 , 128.42 , 128.30 , 127.95 , 125.47 , 123.20 , 88.71 , 85.14 , 67.34 , 59.95 , 49.84 , 19.21 . IR (cm^{-1}):3057.8, 3023.7, 2956.1, 2918.6, 2853.1, 1489.3, 1116.1, 752.8. LRMS (ESI) ($[M]^+$) Calcd. for $C_{20}H_{21}NO$: 291.20, HRMS (ESI) ($[M]^+$)Found: 291.1623.

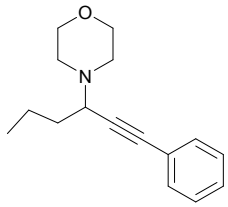


4-(1-(3-methoxyphenyl)-3-phenylprop-2-yn-1-yl)morpholine (4j). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.57 – 7.47 (m, 2H), 7.37 – 7.32 (m, 3H), 7.30 – 7.22 (m, 3H), 6.90 – 6.81 (m, 1H), 4.78 (s, 1H),

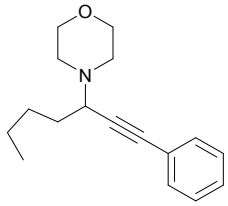
3.84 (s, 3H), 3.76 (td, *J* = 5.2, 2.4 Hz, 4H).



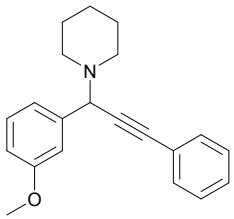
4-(3-phenylprop-2-yn-1-yl)morpholine (4m). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43 (ddd, *J* = 6.7, 3.4, 1.6 Hz, 2H), 7.29 (dt, *J* = 4.9, 1.5 Hz, 3H), 3.80 – 3.71 (m, 4H), 3.50 (s, 2H), 2.69 – 2.58 (m, 4H).



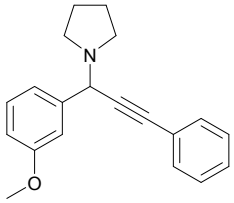
4-(1-phenylhex-1-yn-3-yl)morpholine (4n). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 – 7.38 (m, 2H), 7.35 – 7.26 (m, 3H), 3.82 – 3.69 (m, 4H), 3.51 (dd, *J* = 8.4, 6.3 Hz, 1H), 2.66 (dddd, *J* = 53.0, 11.3, 6.0, 3.7 Hz, 4H), 1.76 – 1.44 (m, 4H), 0.97 (t, *J* = 7.2 Hz, 3H).



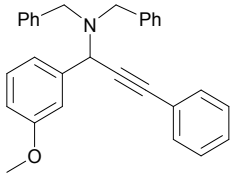
4-(1-phenylhept-1-yn-3-yl)morpholine (4o). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 – 7.39 (m, 2H), 7.29 (h, *J* = 2.6 Hz, 3H), 3.85 – 3.67 (m, 4H), 3.49 (t, *J* = 7.4 Hz, 1H), 2.66 (dddd, *J* = 53.4, 11.4, 6.0, 3.6 Hz, 4H), 1.78 – 1.65 (m, 2H), 1.46 – 1.30 (m, 4H), 0.94 (d, *J* = 7.1 Hz, 3H).



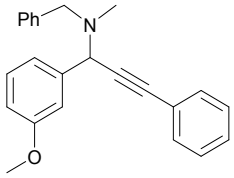
1-(1-(3-methoxyphenyl)-3-phenylprop-2-yn-1-yl)piperidine (4p). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56 – 7.51 (m, 2H), 7.36 – 7.32 (m, 3H), 7.30 – 7.24 (m, 3H), 6.85 (dt, *J* = 7.4, 2.2 Hz, 1H), 4.81 (s, 1H), 3.84 (s, 3H), 2.60 (t, *J* = 5.6 Hz, 4H), 1.67 – 1.57 (m, 4H), 1.47 (q, *J* = 5.7 Hz, 2H).



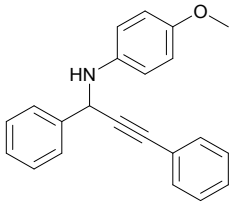
1-(1-(3-methoxyphenyl)-3-phenylprop-2-yn-1-yl)pyrrolidine (4q). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 – 7.45 (m, 2H), 7.32 – 7.28 (m, 3H), 7.25 – 7.18 (m, 3H), 6.83 (ddd, *J* = 8.0, 2.6, 1.4 Hz, 1H), 4.92 (s, 1H), 3.80 (s, 3H), 2.75 (p, *J* = 3.9 Hz, 4H), 1.79 (d, *J* = 1.4 Hz, 4H).



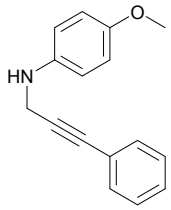
N, N-dibenzyl-1-(3-methoxyphenyl)-3-phenylprop-2-yn-1-amine (4r). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 – 7.65 (m, 2H), 7.50 (d, *J* = 7.1 Hz, 4H), 7.45 – 7.41 (m, 3H), 7.38 (dt, *J* = 5.6, 1.9 Hz, 5H), 7.34 (d, *J* = 7.6 Hz, 2H), 7.32 – 7.27 (m, 2H), 6.88 – 6.83 (m, 1H), 4.97 (s, 1H), 3.89 (s, 1H), 3.87 (s, 3H), 3.85 (s, 1H), 3.60 (d, *J* = 13.5 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.63 , 141.06 , 139.65 , 132.10 , 129.20 , 129.03 , 128.53 , 128.44 , 128.39 , 127.16 , 120.84 , 114.36 , 112.75 , 56.20 , 55.34 , 54.84 . IR (cm^{-1}): 3453.6, 3023.6, 2821.2, 1945.0, 1803.5, 1598.0, 1488.0, 1323.1. LRMS (ESI) ([M]$^+$) Calcd. for $C_{30}H_{27}NO$:417.26. HRMS (ESI) ([M]$^+$)Found: 417.2093.



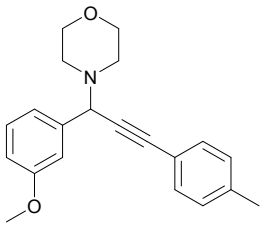
N-benzyl-1-(3-methoxyphenyl)-N-methyl-3-phenylprop-2-yn-1-amine (4s). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.63 – 7.58 (m, 2H), 7.48 – 7.44 (m, 2H), 7.41 – 7.28 (m, 9H), 6.86 (ddt, *J* = 7.7, 4.5, 2.5 Hz, 1H), 4.94 (s, 1H), 3.86 (s, 3H), 3.80 – 3.64 (m, 2H), 2.30 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.68 , 140.84 , 139.35 , 132.02 , 129.22 , 129.14 , 128.48 , 128.44 , 128.32 , 127.21 , 120.88 , 114.26 , 112.97 , 84.79 , 59.72 , 58.99 , 55.37 , 38.26 . IR (cm^{-1}): 3059.0, 2944.0, 2836.5, 2793.6, 2360.5, 1950.1, 1602.1, 1279.9, 1048.2. LRMS (ESI) ([M]$^+$) Calcd. for $C_{24}H_{23}NO$:341.02. HRMS (ESI) ([M]$^+$)Found: 341.1780.



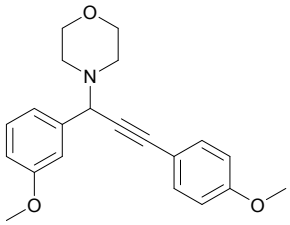
N-(1-(2-bromophenyl)-3-phenylprop-2-yn-1-yl)-4-methoxyaniline (4u). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 – 7.64 (m, 2H), 7.41 (td, *J* = 5.6, 2.7 Hz, 4H), 7.36 (t, *J* = 1.5 Hz, 1H), 7.34 (d, *J* = 2.2 Hz, 1H), 7.29 (s, 2H), 7.29 – 7.27 (m, 1H), 6.79 (d, *J* = 6.0 Hz, 3H), 5.42 (s, 1H), 3.85 – 3.82 (m, 1H), 3.76 (s, 3H).



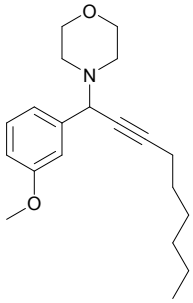
N-(3-(3-methoxyphenyl) prop-2-yn-1-yl)aniline (4v). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 – 7.37 (m, 2H), 7.32 – 7.27 (m, 3H), 6.86 – 6.80 (m, 2H), 6.76 – 6.70 (m, 2H), 4.11 (s, 2H), 3.77 (s, 3H).



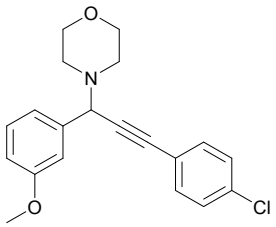
4-(1-(3-methoxyphenyl)-3-(p-tolyl) prop-2-yn-1-yl)morpholine (4w). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45 – 7.39 (m, 2H), 7.31 – 7.22 (m, 3H), 7.15 (d, *J* = 7.9 Hz, 2H), 6.86 (ddd, *J* = 7.7, 2.6, 1.6 Hz, 1H), 4.77 (s, 1H), 3.84 (s, 3H), 3.76 (dq, *J* = 5.1, 2.5 Hz, 4H), 2.72 – 2.60 (m, 4H), 2.38 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.70 , 139.52 , 138.48 , 131.80 , 129.27 , 129.16 , 121.13 , 114.45 , 113.25 , 84.23 , 67.20 , 62.10 , 55.37 , 49.99 , 21.59 . IR (cm^{-1}): 3423.0, 3026.6, 2956.9, 2751.2, 2691.2, 2219.6, 1717.6, 1509.0. LRMS (ESI) ($[M]^+$) Calcd. for $C_{21}H_{23}NO_2$:321.20. HRMS (ESI) ($[M]^+$)Found: 321.1729.



4-(1-(3-methoxyphenyl)-3-(4-methoxyphenyl) prop-2-yn-1-yl)morpholine (4x). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 – 7.36 (m, 2H), 7.25 – 7.15 (m, 3H), 6.85 – 6.77 (m, 3H), 4.69 (s, 1H), 3.78 (s, 3H), 3.77 (s, 3H), 3.69 (td, *J* = 5.0, 2.3 Hz, 4H), 2.59 (q, *J* = 4.5 Hz, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.69 , 139.58 , 133.31 , 129.26 , 121.13 , 115.13 , 114.46 , 114.03 , 113.21 , 83.46 , 67.18 , 62.11 , 55.41 , 55.37 , 49.98 . IR (cm^{-1}): 3391.3, 3000.9, 2835.7, 2052.7, 1715.0, 1604.2, 1487.5, 1266.6, 833.6. LRMS (ESI) ($[M]^+$) Calcd. for $C_{21}H_{23}NO_3$:337.19. HRMS (ESI) ($[M]^+$)Found: 337.1678.

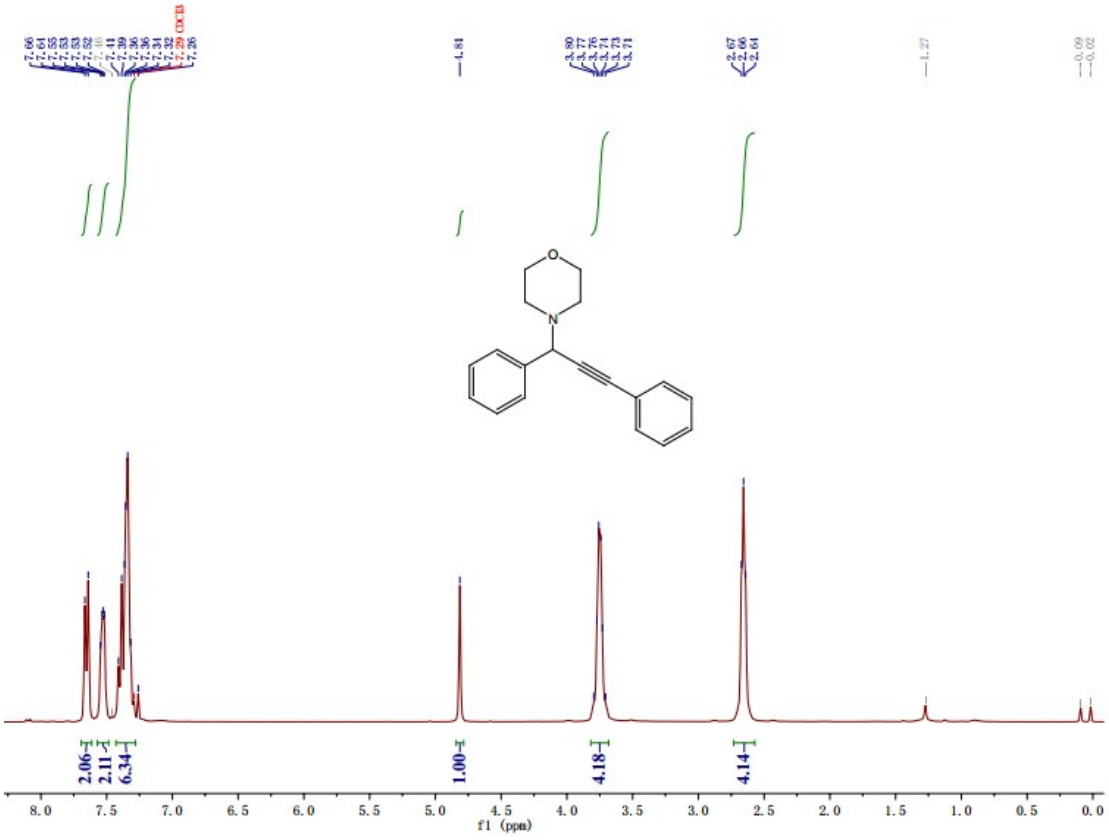


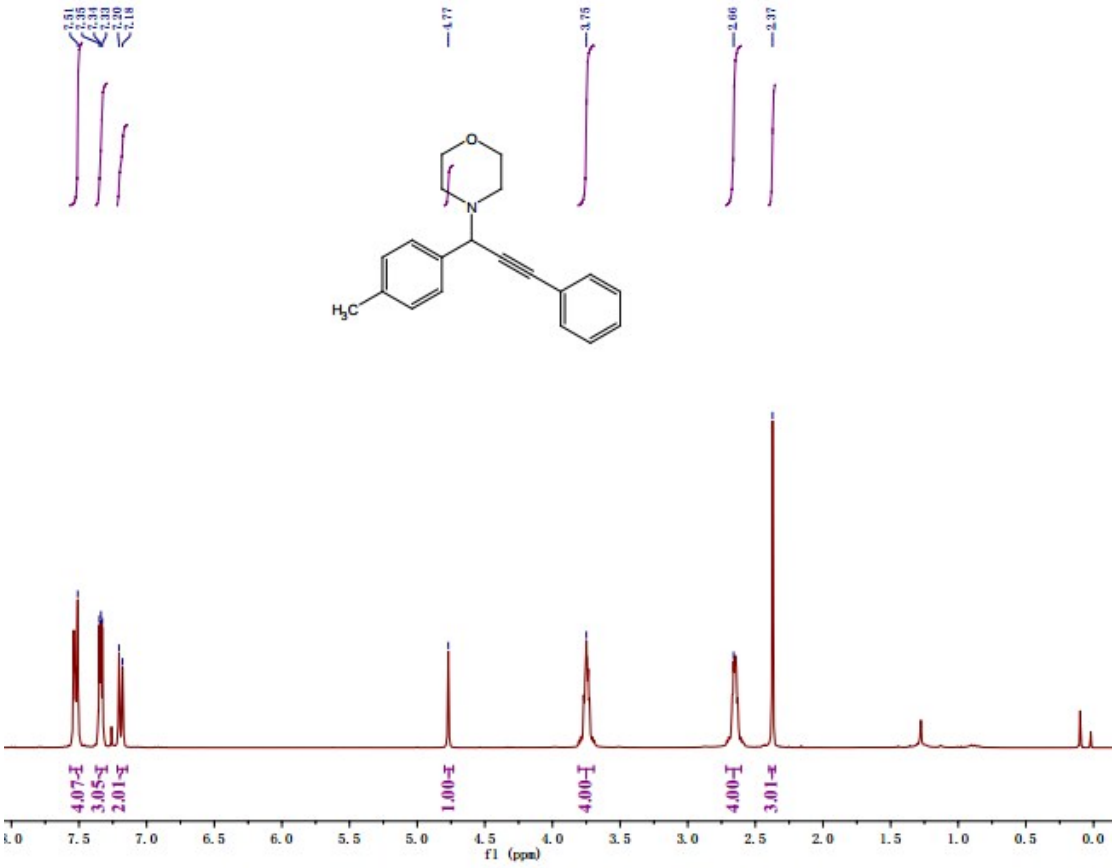
4-(1-(3-methoxyphenyl) non-2-yn-1-yl)morpholine (4y). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.0 Hz, 1H), 7.14 (ddd, *J* = 5.4, 2.0, 1.0 Hz, 2H), 6.80 (ddd, *J* = 8.1, 2.6, 1.1 Hz, 1H), 4.48 (s, 1H), 3.80 (s, 3H), 3.69 (td, *J* = 4.9, 2.1 Hz, 4H), 2.52 (q, *J* = 4.6 Hz, 4H), 2.29 (td, *J* = 7.0, 2.1 Hz, 2H), 1.60 – 1.51 (m, 2H), 1.48 – 1.38 (m, 2H), 1.30 (tt, *J* = 7.3, 3.0 Hz, 4H), 0.92 – 0.85 (m, 3H). ^{13}C NMR (300 MHz, $CDCl_3$) δ 159.63 , 140.13 , 129.14 , 121.10 , 114.38 , 113.09 , 75.39 , 67.24 , 61.75 , 55.36 , 49.91 , 31.46 , 29.11 , 28.77 , 22.70 , 18.93 , 14.18 . IR (cm^{-1}): 3415.3, 2930.7, 2854.9, 2750.3, 2689.1, 1724.4, 1603.6, 1487.5. LRMS (ESI) ($[M]^+$) Calcd. for $C_{20}H_{29}NO_2$:315.18. HRMS (ESI) ($[M]^+$)Found:315.2198.

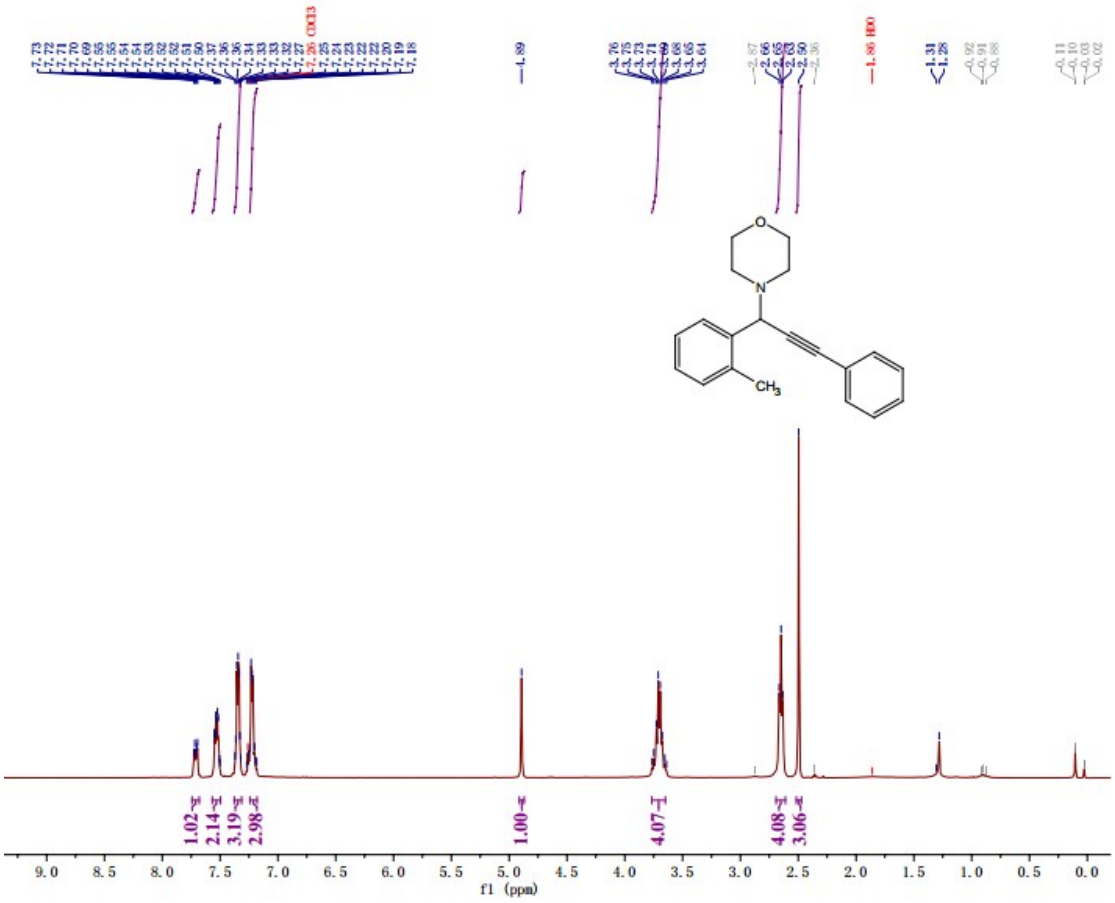


4-(3-(4-chlorophenyl)-1-(3-methoxyphenyl) prop-2-yn-1-yl)morpholine (4z). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 – 7.40 (m, 2H), 7.33 – 7.26 (m, 3H), 7.24 – 7.18 (m, 2H), 6.88 – 6.83 (m, 1H), 4.75 (s, 1H), 3.83 (s, 3H), 3.74 (td, *J* = 5.0, 2.3 Hz, 4H), 2.69 – 2.57 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.75 , 139.28 , 134.41 , 133.15 , 129.36 , 128.76 , 121.51 , 121.01 , 114.48 , 113.20 , 87.44 , 86.20 , 67.22 , 62.10 , 55.39 , 50.07 . IR (cm^{-1}): 3454.8, 3065.9, 2997.7, 2855.2, 2822.4, 2739.5, 2569.8, 1679.4. LRMS (ESI) ($[M]^+$) Calcd. for $C_{20}H_{20}ClNO_2$:340.93. HRMS (ESI) ($[M]^+$)Found: 341.1183.

6.^{1}H NMR and ^{13}C NMR spectra



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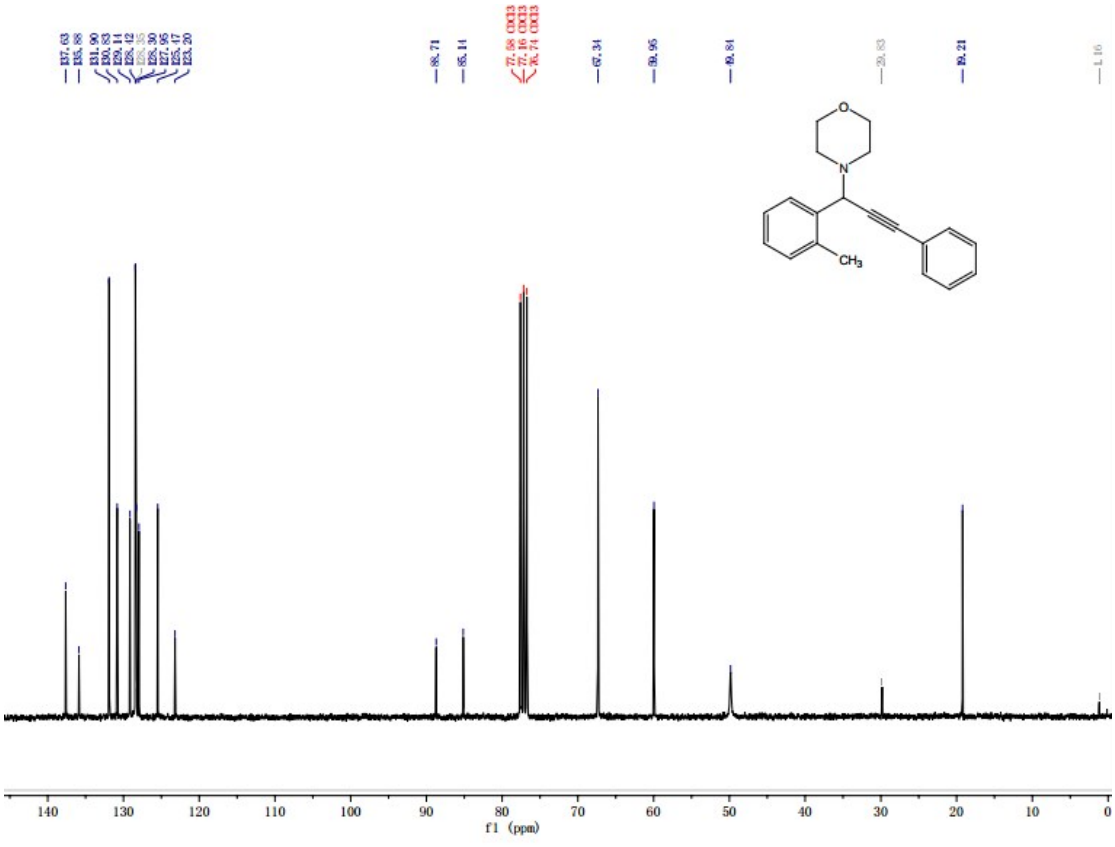
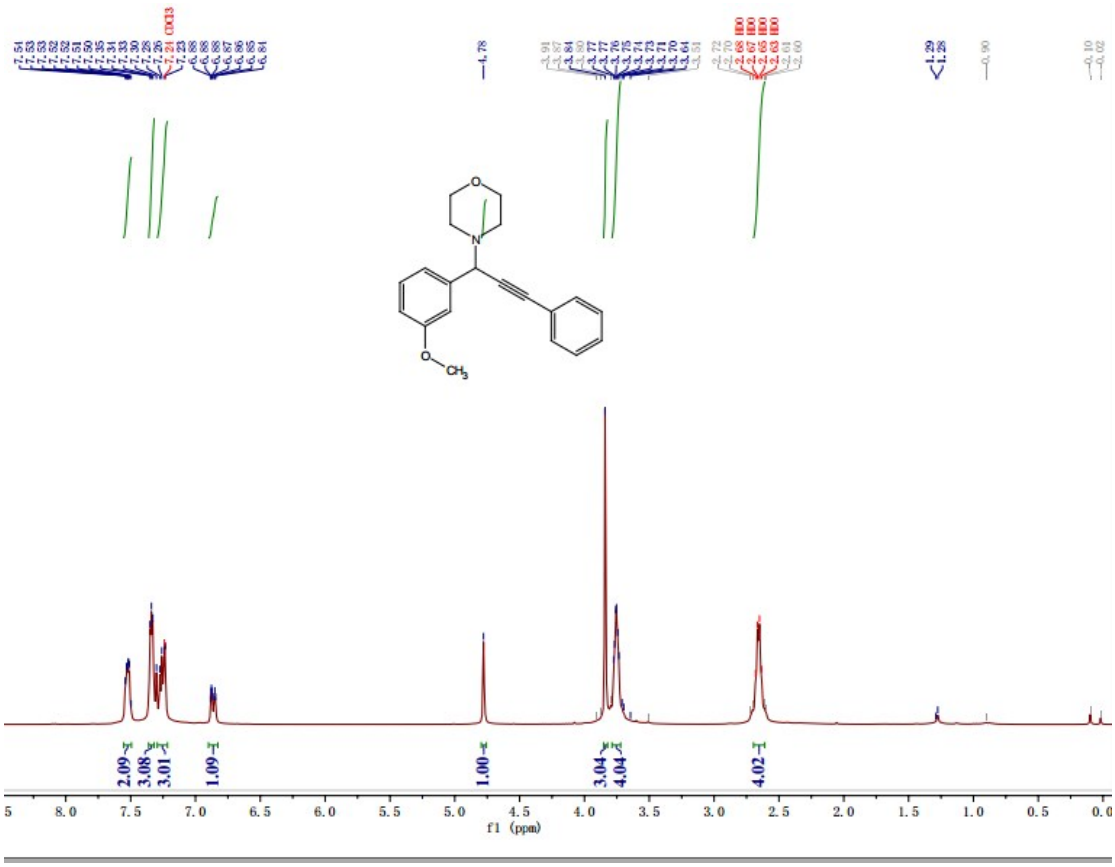
3.19
2.98
1.00
4.07
4.08
3.06
f1 (ppm)


f1 (ppm)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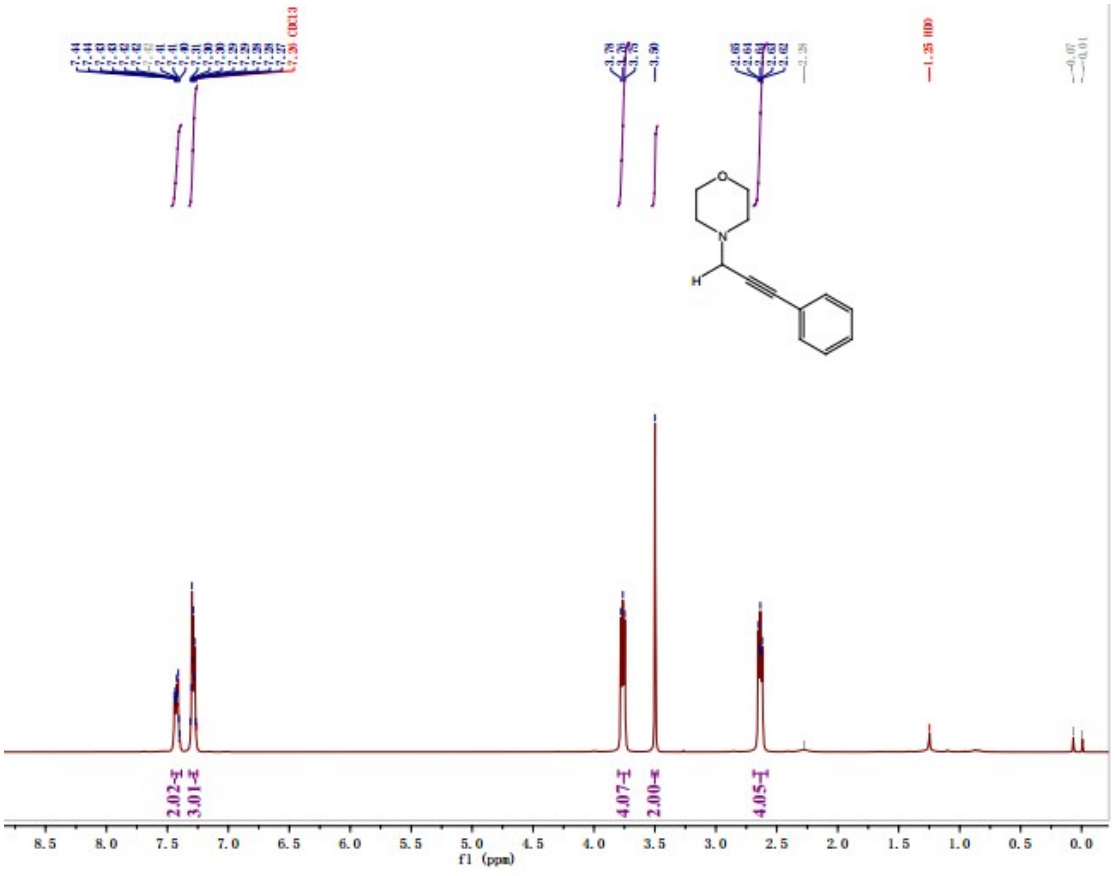
3.01
1.09
1.00
3.04
4.04
4.02
f1 (ppm)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2.00
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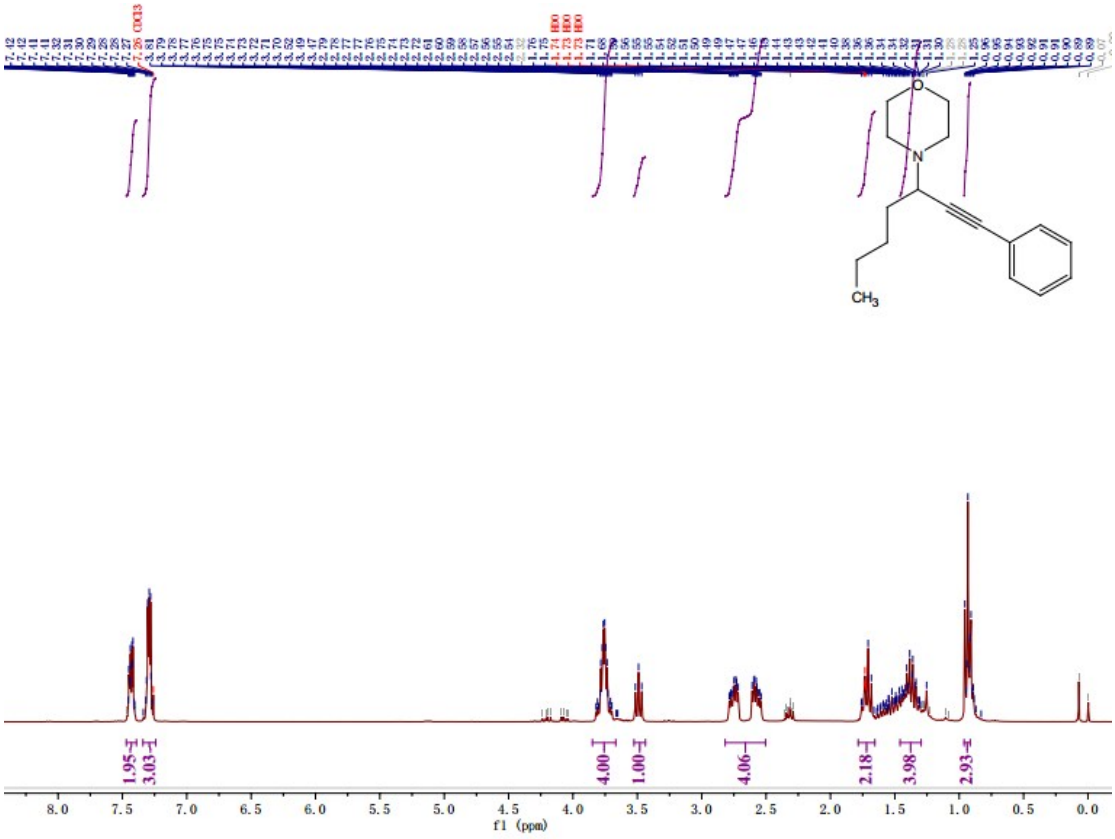
f1 (ppm)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1.00
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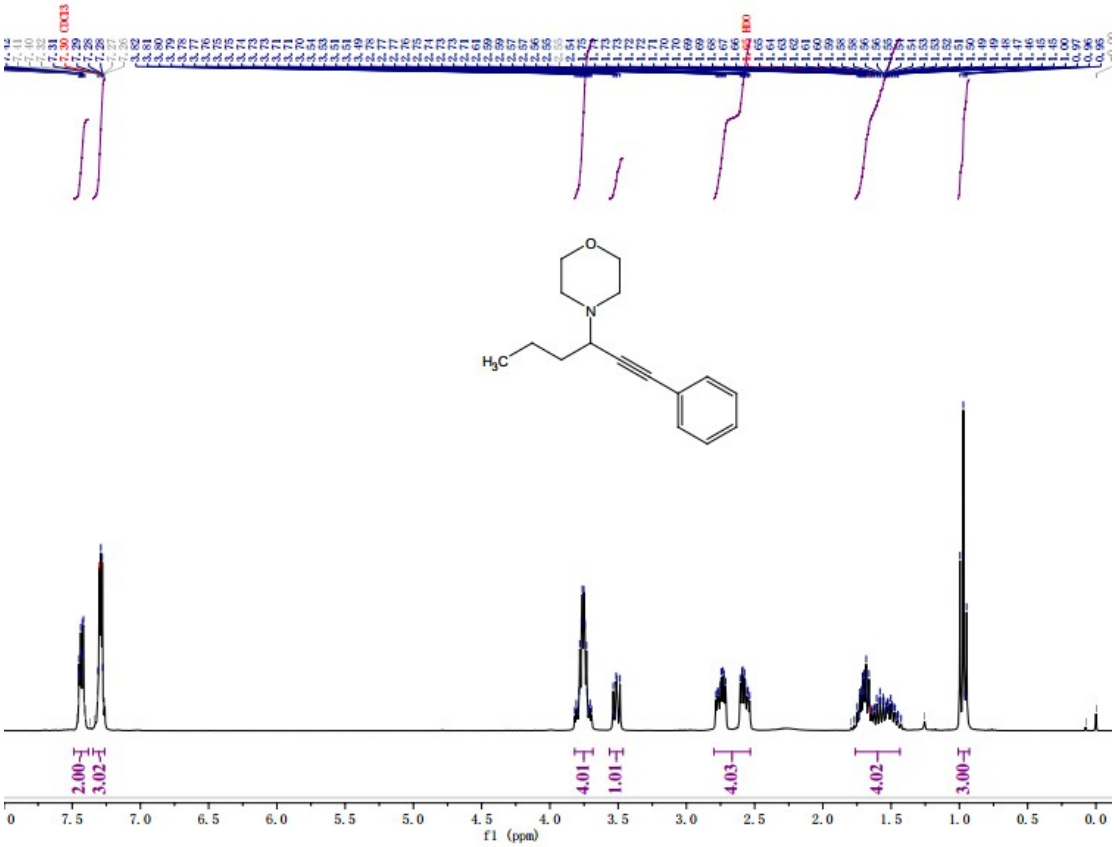
2.18
3.98
2.93
f1 (ppm)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H3C
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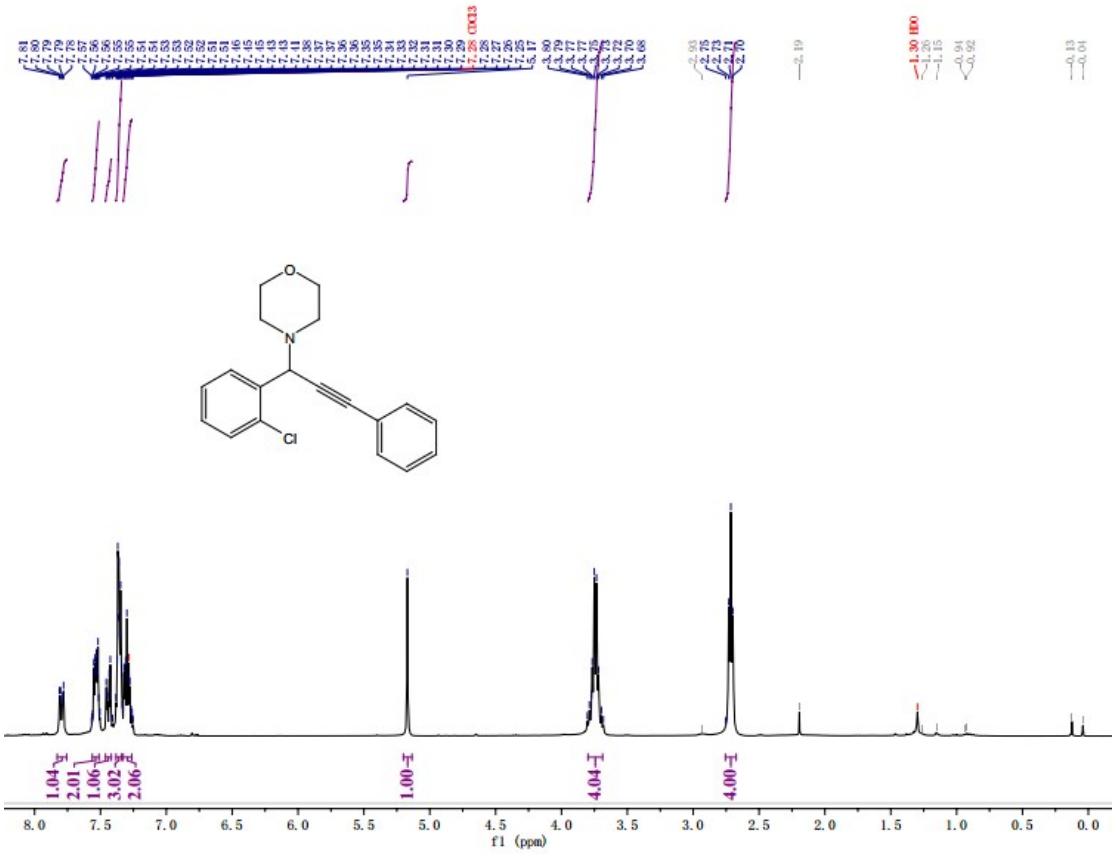
4.01
1.01
4.03
4.02
3.00
f1 (ppm)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Cl
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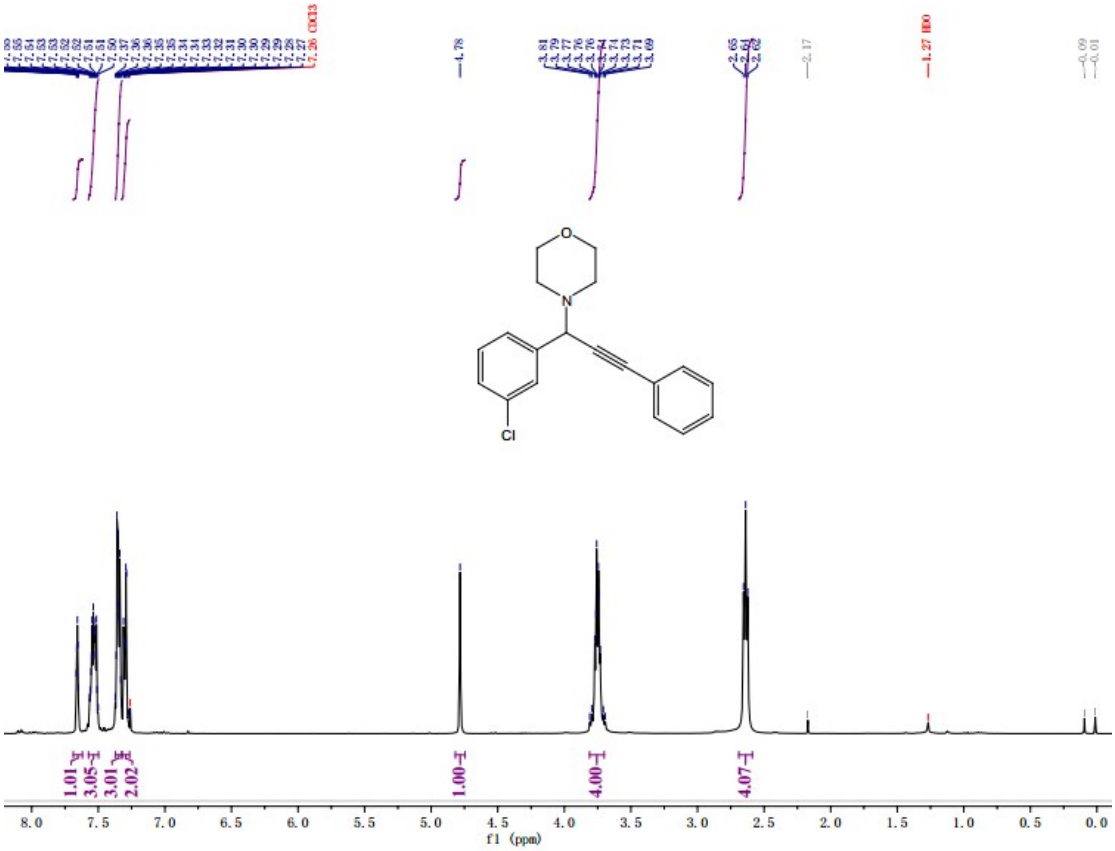
1.06
3.02
2.06
1.00
4.04
4.00
f1 (ppm)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Cl
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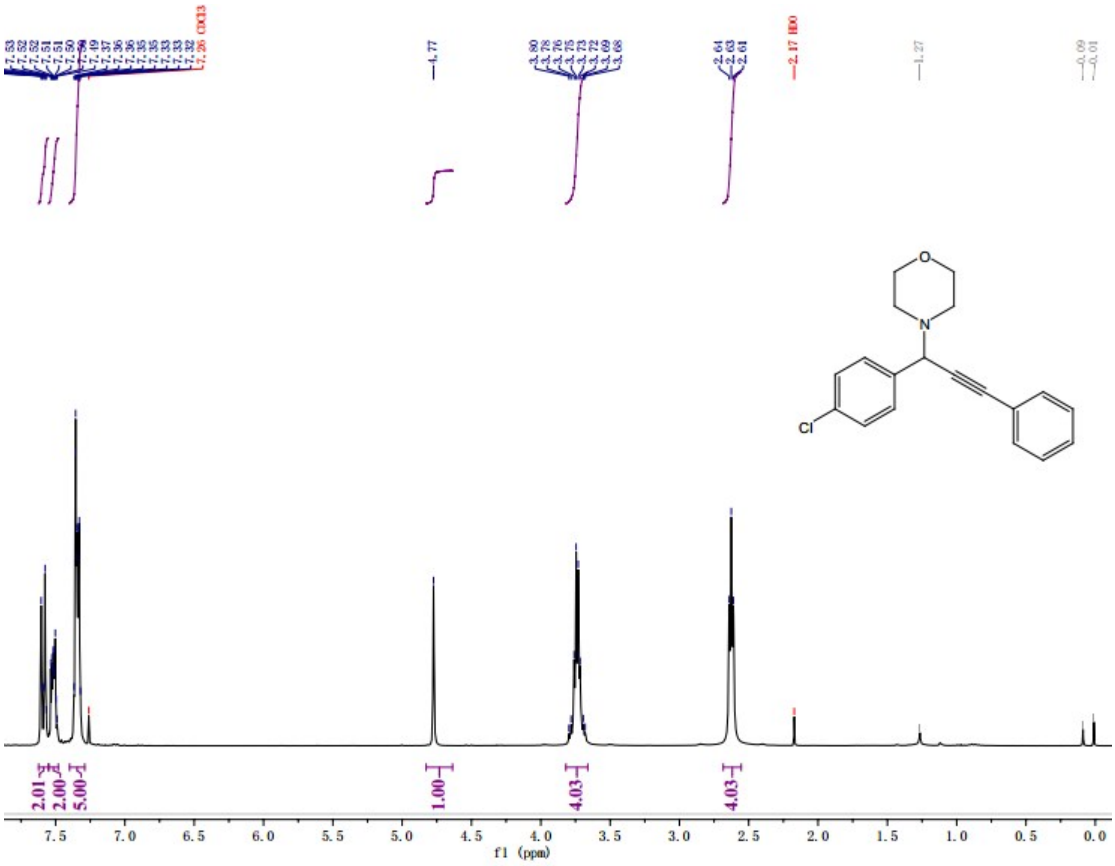
3.01
2.02
1.00
4.00
4.07
f1 (ppm)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7.52
7.52
7.51
7.51
7.50
7.50
7.37
7.36
7.35
7.35
7.33
7.33
7.32
7.26 CDCl3
4.77
3.80
3.78
3.76
3.75
3.73
3.72
3.69
3.68
2.64
2.63
2.04
2.17 HDO
1.27
0.09
0.01
O
N
Cl
2.01
2.00
5.00
1.00
4.03
4.03
7.5
7.0
6.5
6.0
5.5
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4.5
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2.5
2.0
1.5
1.0
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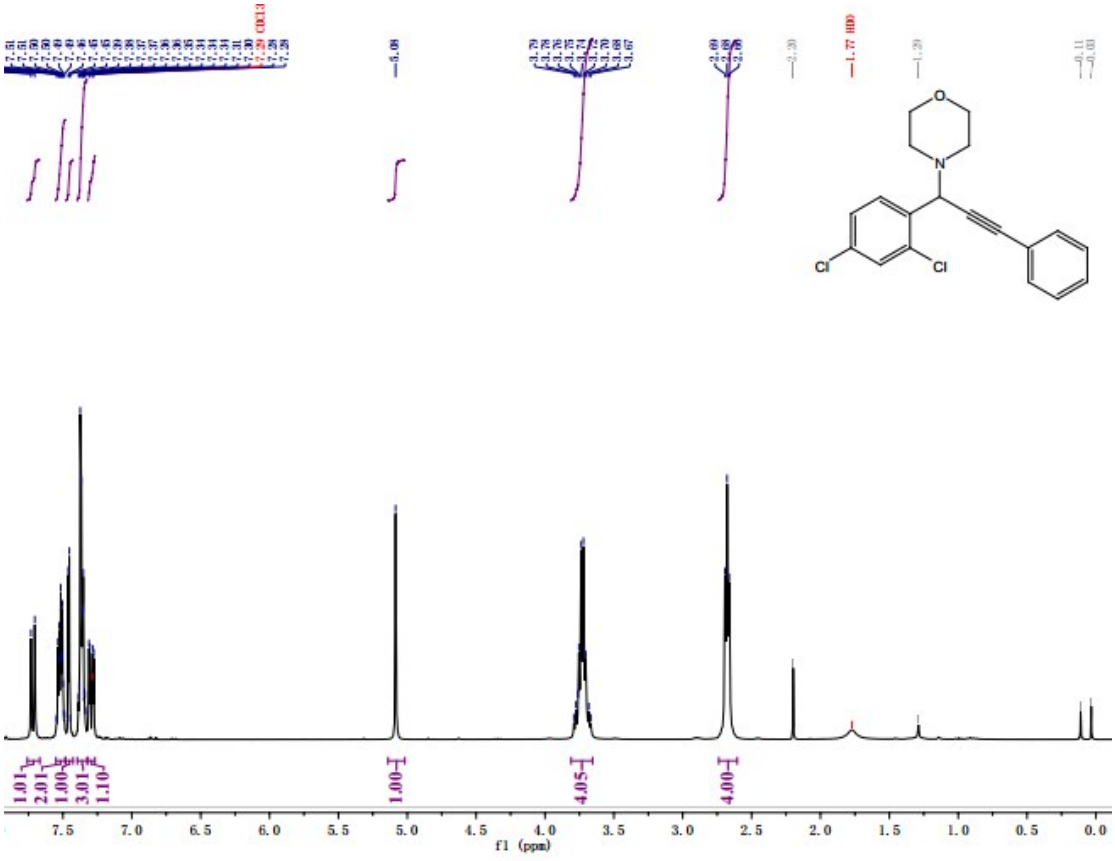
0.0
f1 (ppm)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7.51
7.50
7.50
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7.49
7.46
7.45
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7.38
7.37
7.37
7.36
7.36
7.35
7.35
7.34
7.34
7.31
7.31
7.30
7.30
7.29 CDCl3
7.28
7.28
5.08
3.79
3.78
3.76
3.75
3.74
3.72
3.70
3.68
3.67
2.69
2.68
2.66
2.65
2.30
1.77 HDO
1.29
0.11
0.01
O
N
Cl
Cl
1.01
2.01
1.00
3.01
1.10
1.00
4.05
4.00
7.5
7.0
6.5
6.0
5.5
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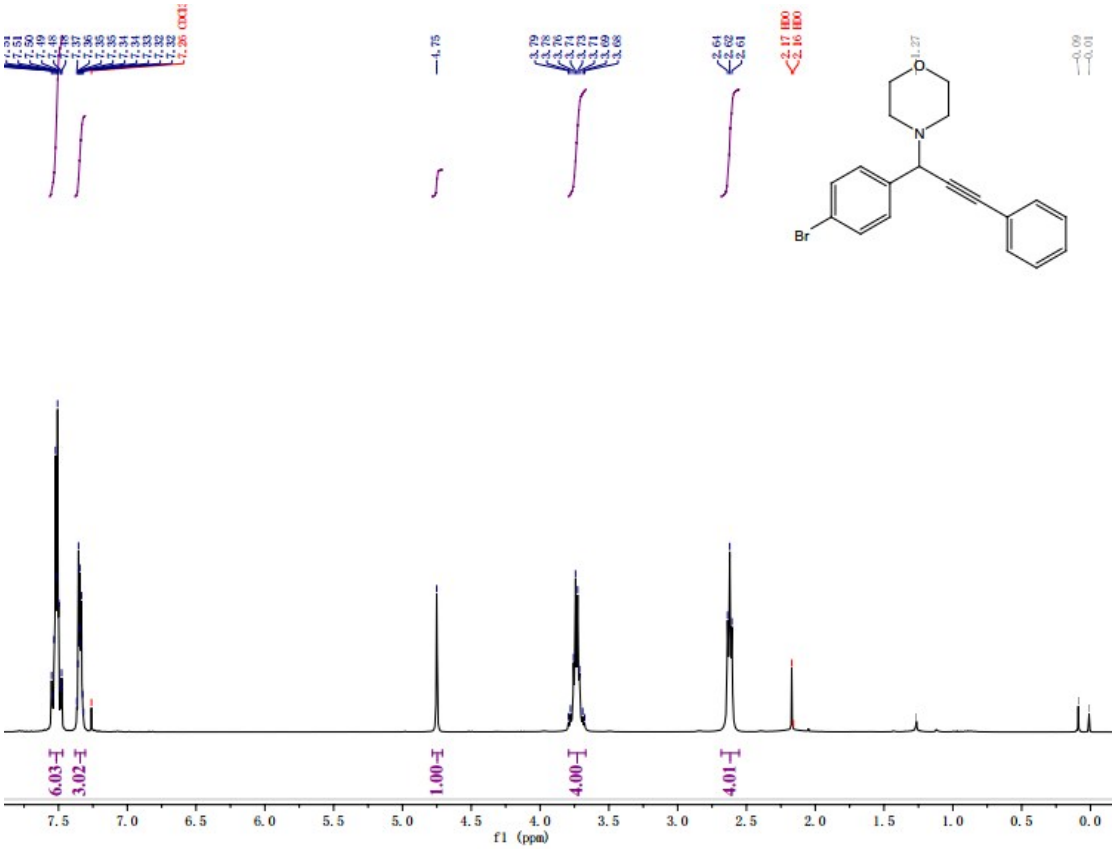
f1 (ppm)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Br
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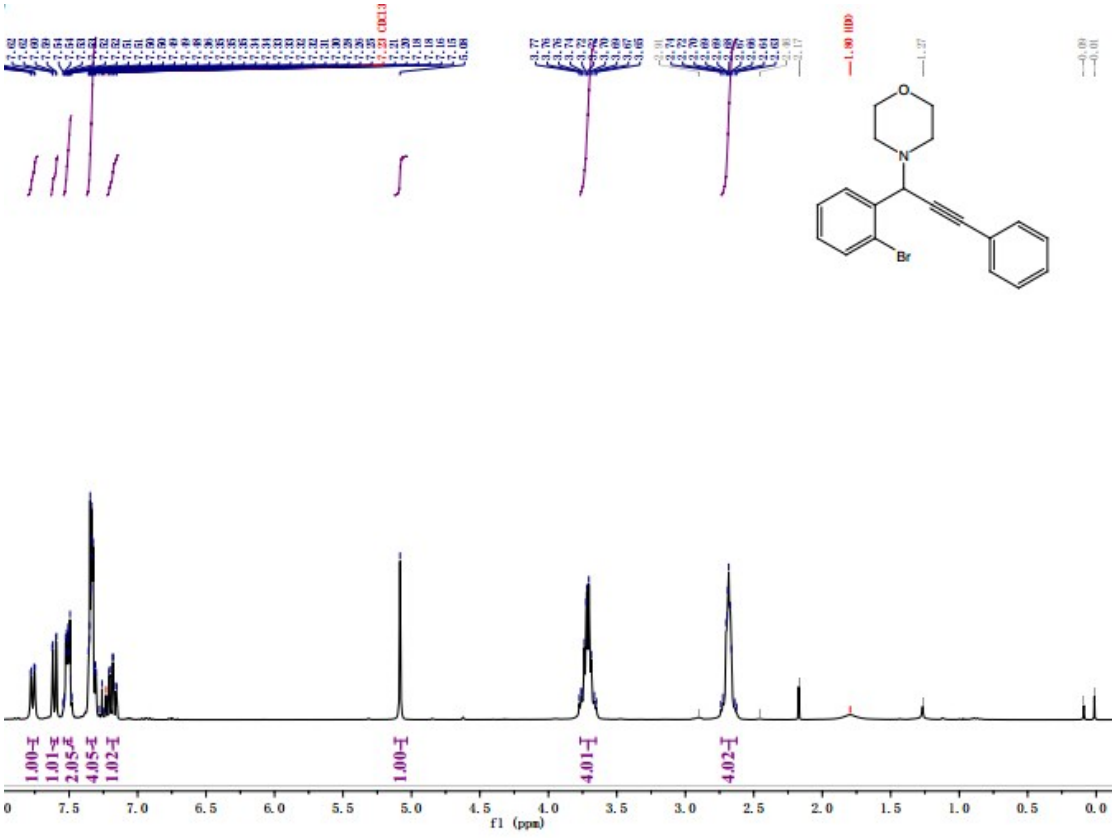
4.00
4.01
f1 (ppm)


O
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Br
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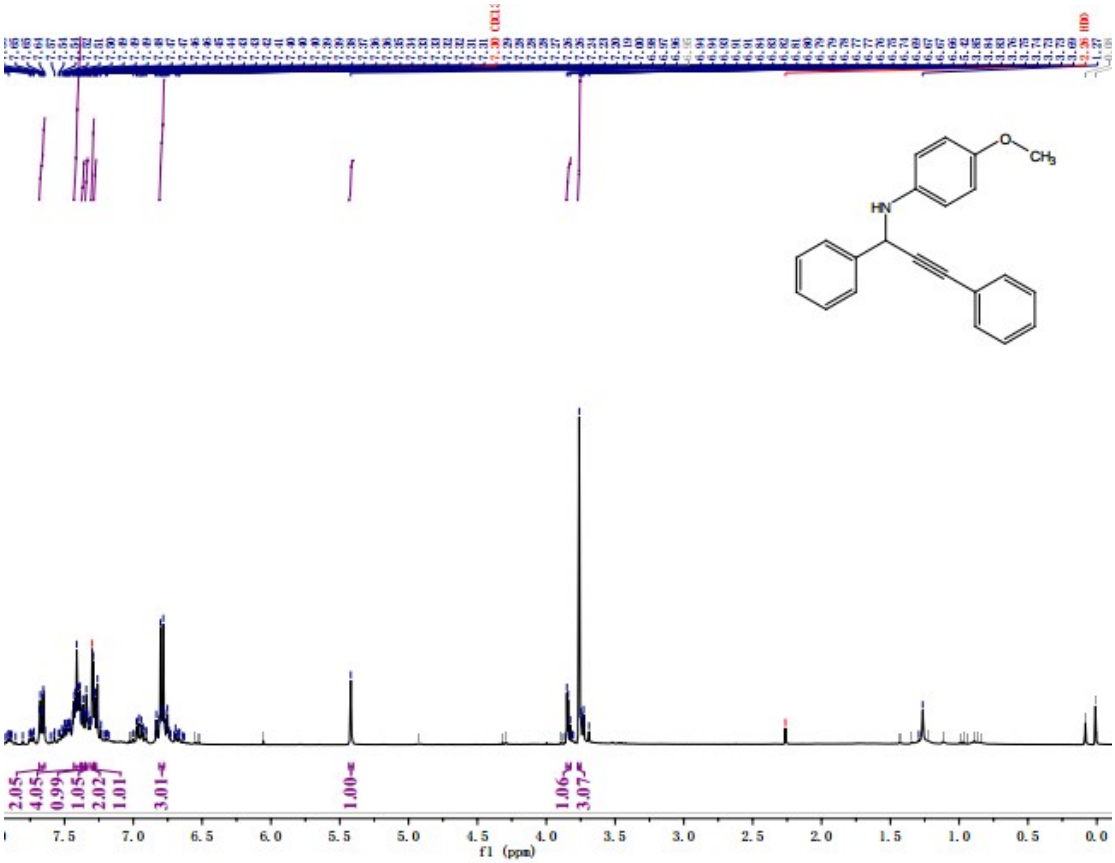
2.05
4.05
1.02
1.00
4.01
4.02
f1 (ppm)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O
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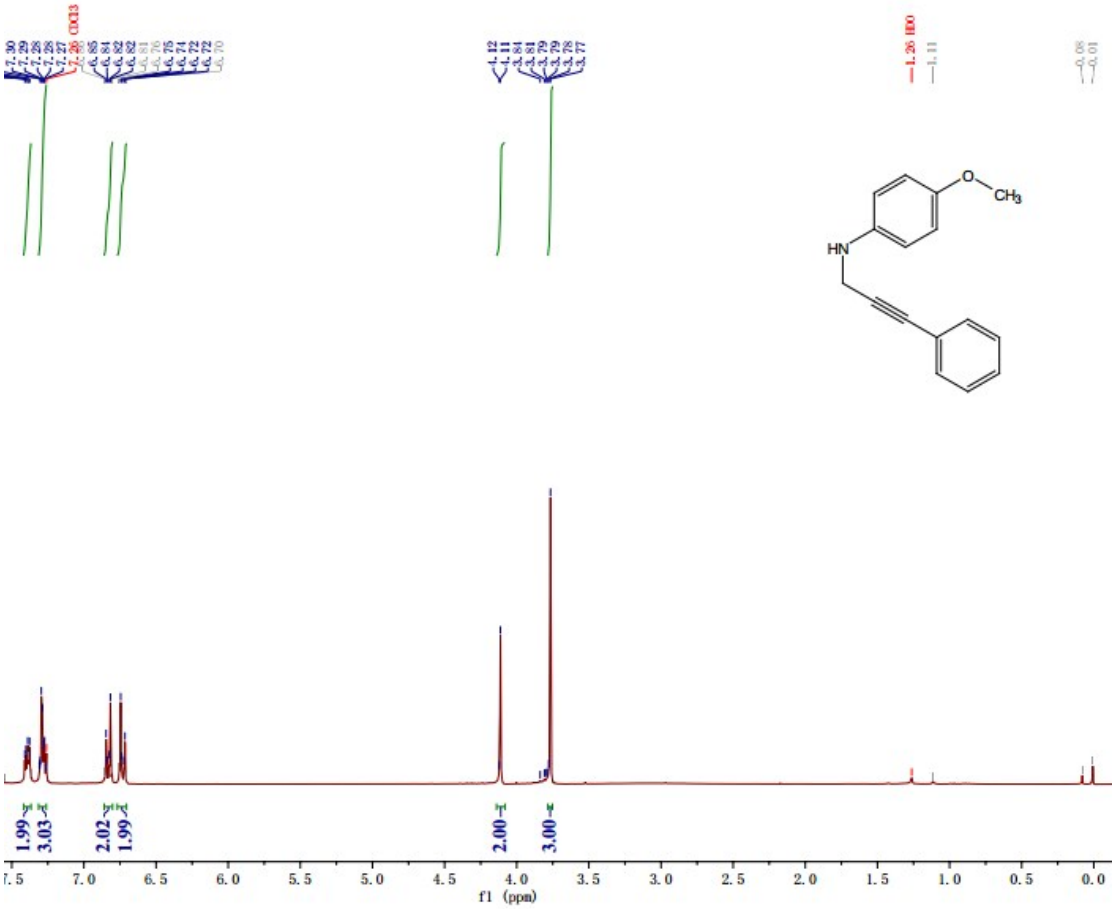
2.02
1.01
3.01
1.00
1.06
3.07
f1 (ppm)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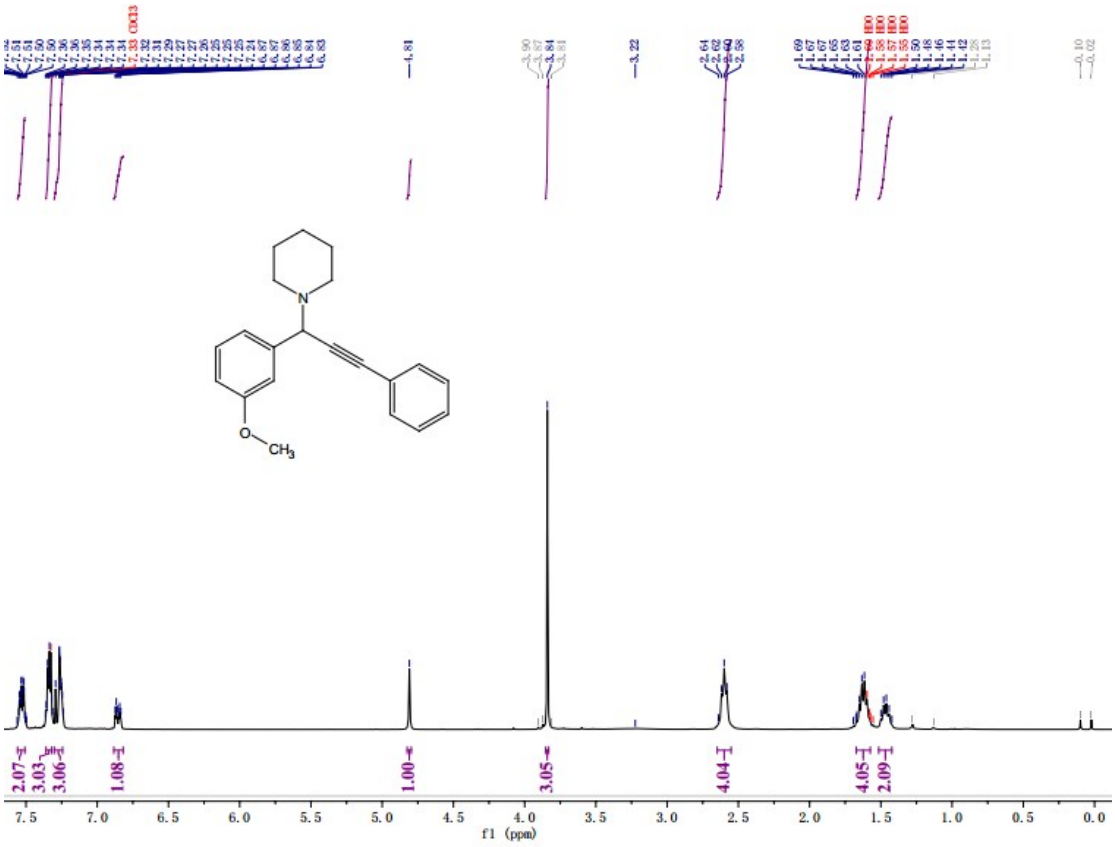
O
CH3
1.99
3.03
2.02
1.99
2.00
3.00
f1 (ppm)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O
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1.08
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3.05
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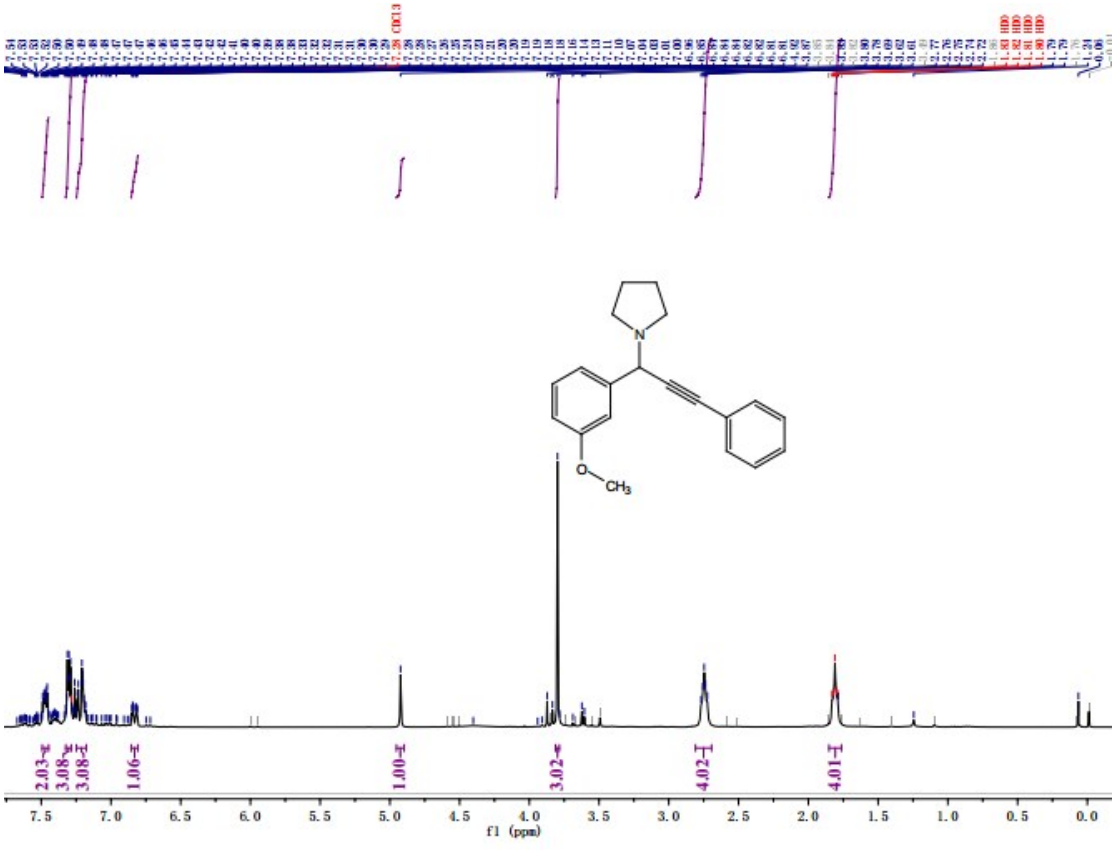
4.05
2.09
f1 (ppm)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O
CH3
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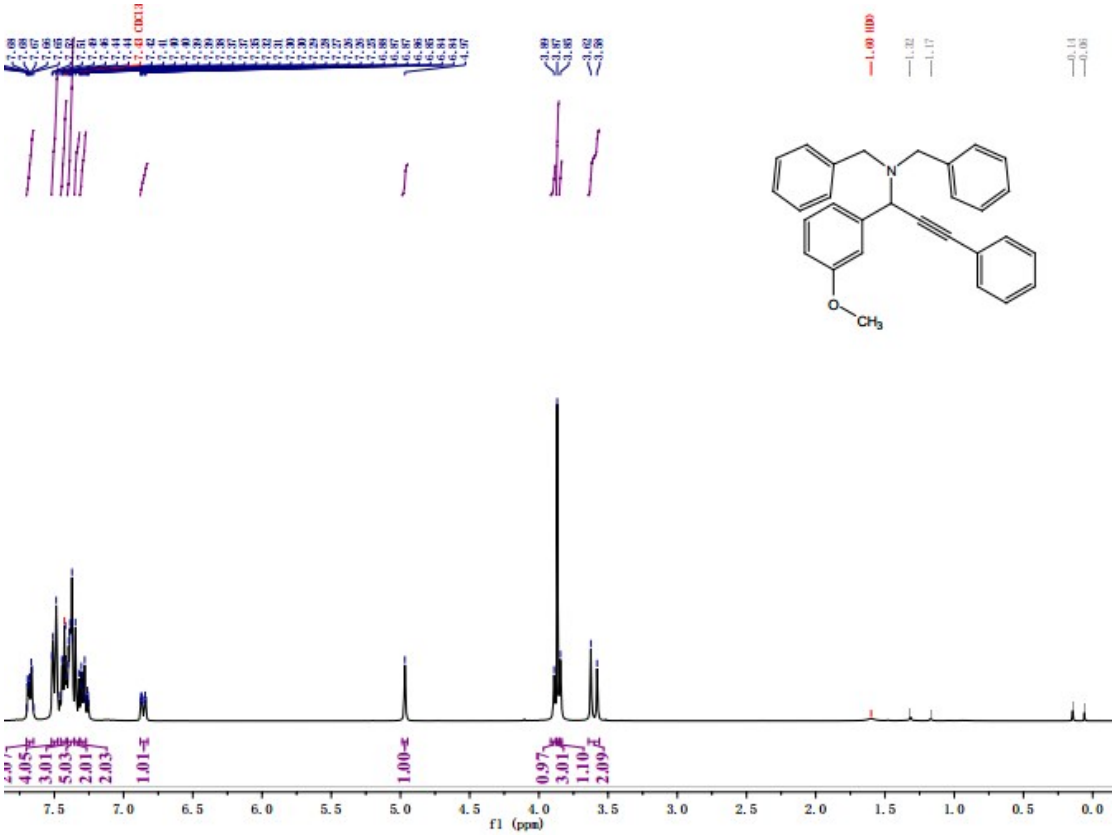
3.08
1.06
1.00
3.02
4.02
4.01
f1 (ppm)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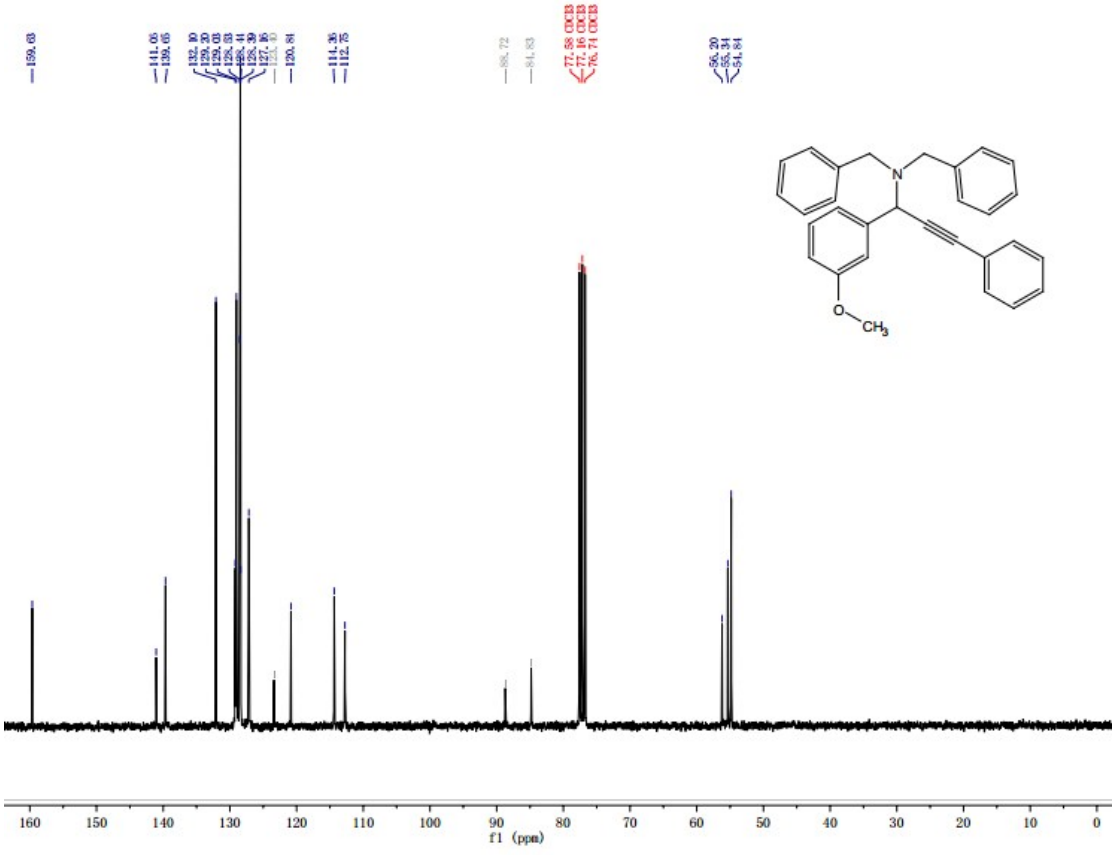
2.09
f1 (ppm)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132.10
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129.03
128.33
128.14
128.09
127.85
121.10
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114.36
112.75
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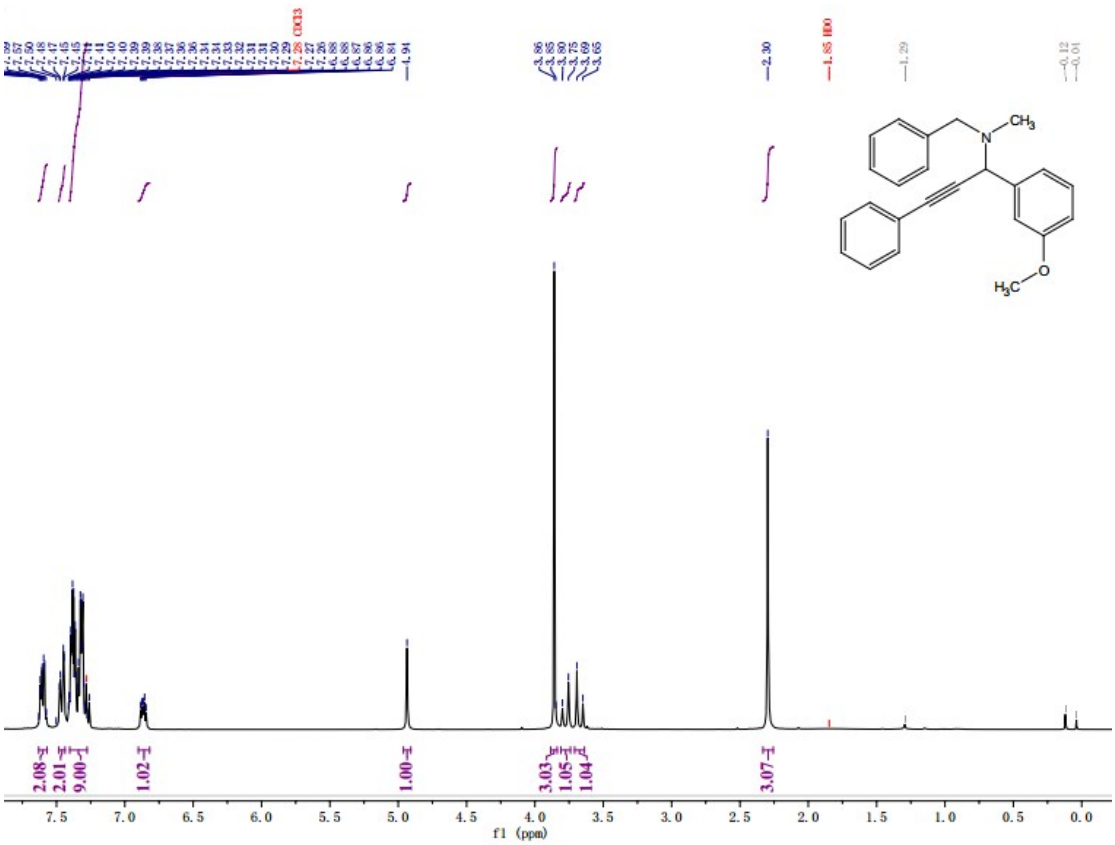
77.58 CDCl3
77.16 CDCl3
76.71 CDCl3
56.20
55.34
54.84
f1 (ppm)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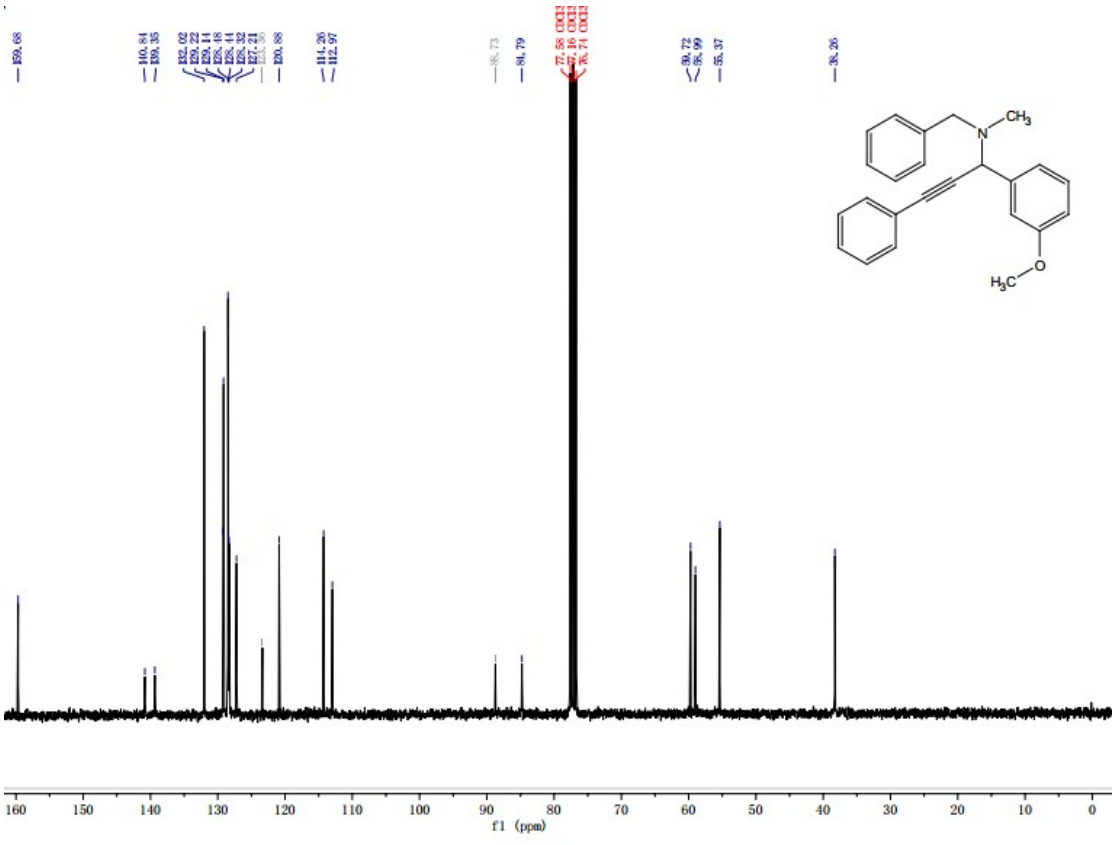
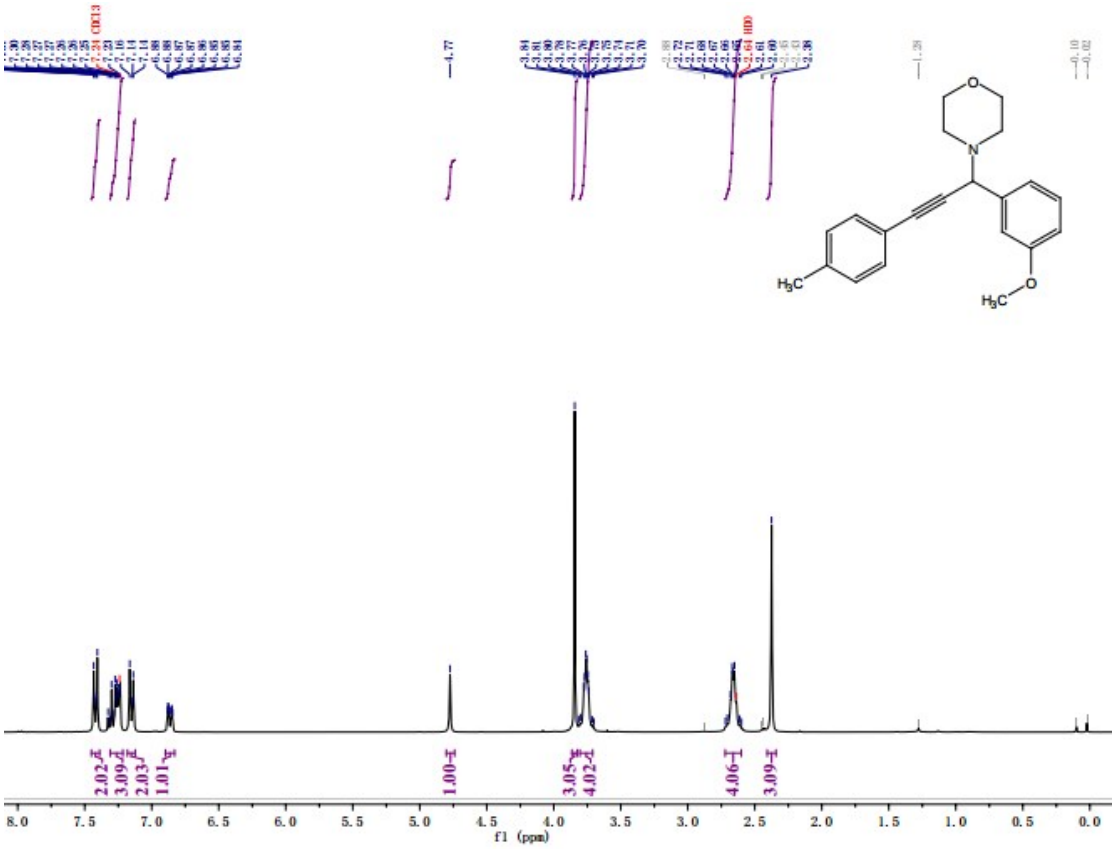
1.00
3.03
1.05
1.04
3.07
f1 (ppm)


CDCl3
f1 (ppm)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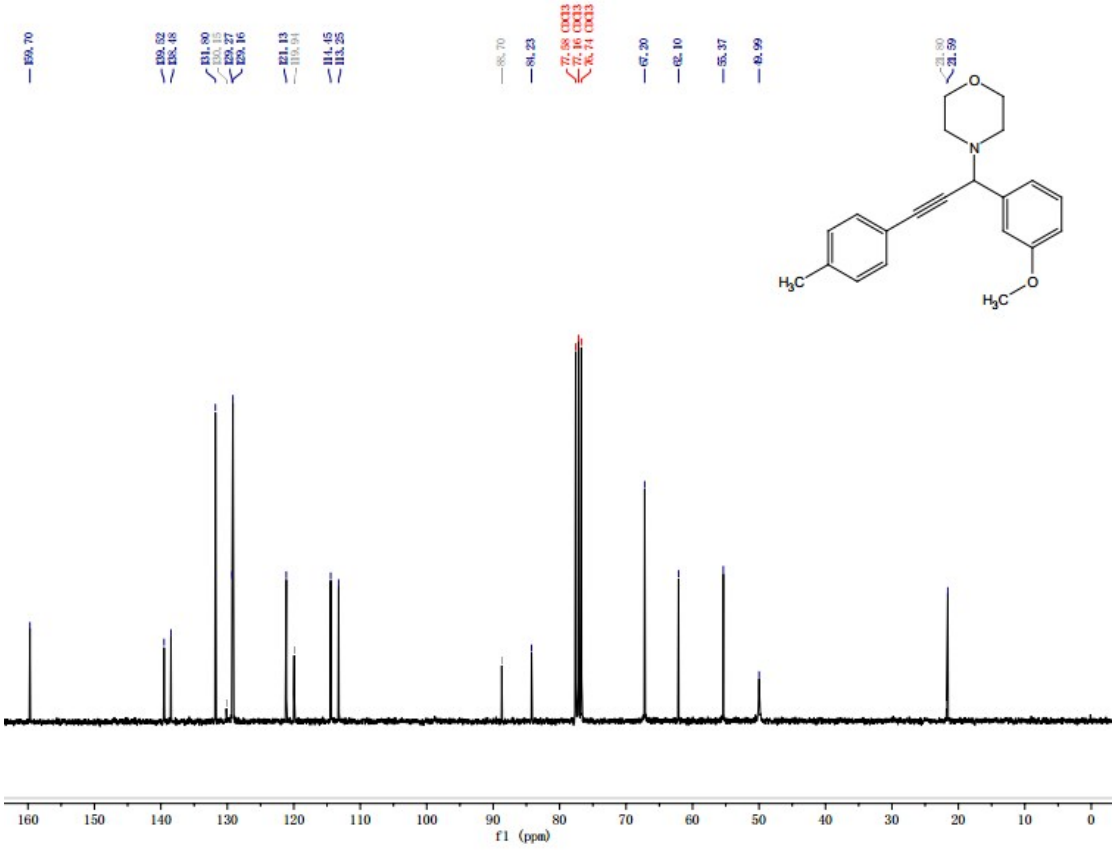
f1 (ppm)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129.16
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113.25
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81.23
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76.74 CDCl3
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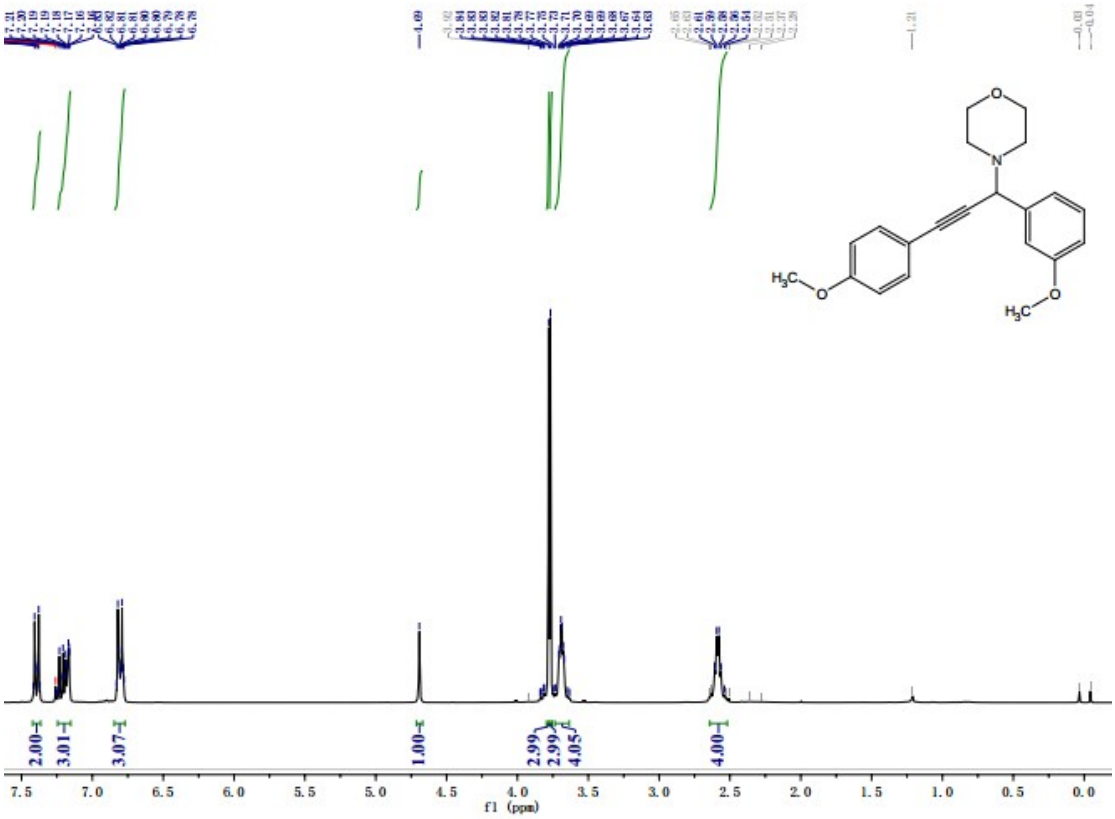
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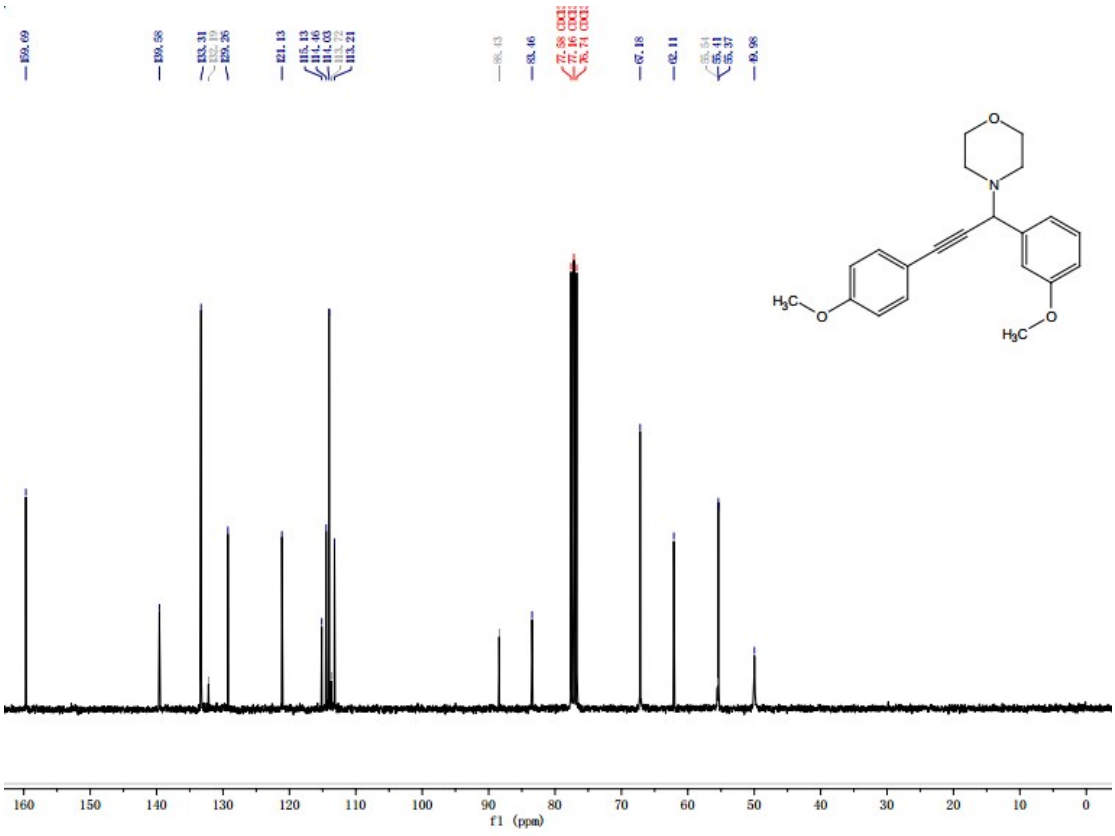
4.00
f1 (ppm)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115.13
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111.03
113.22
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55.41
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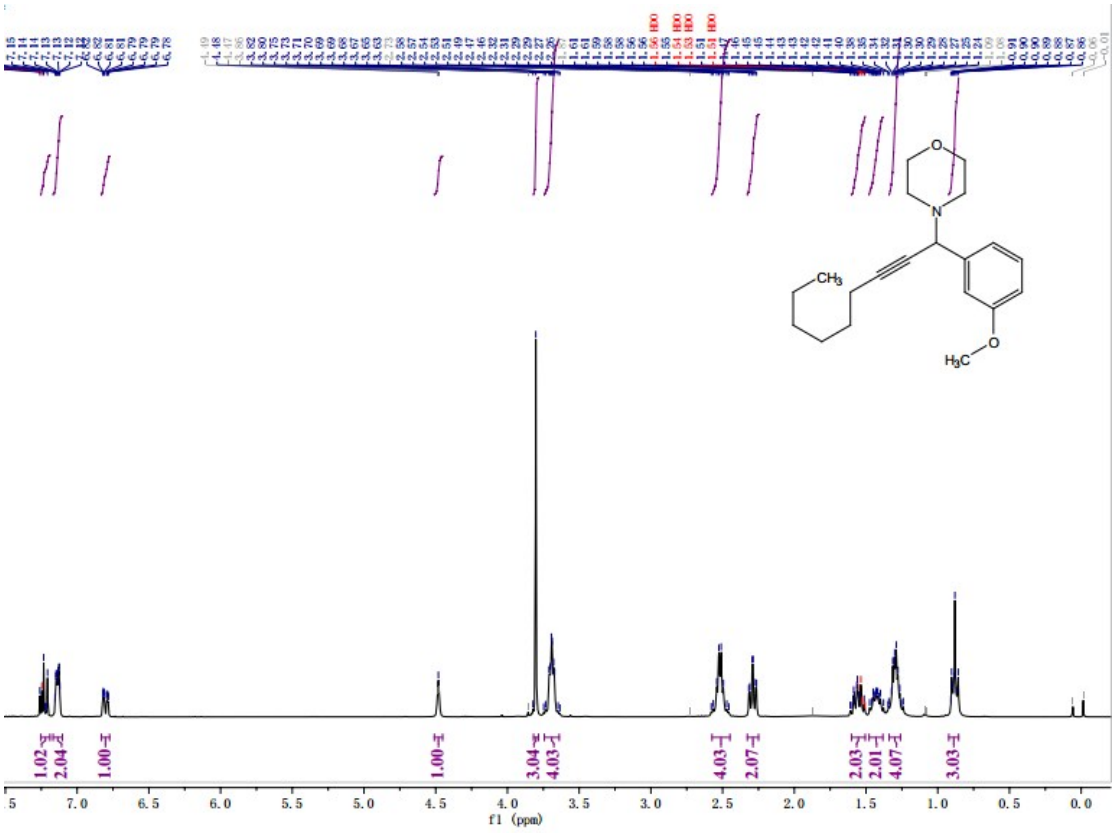
f1 (ppm)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2.03
2.01
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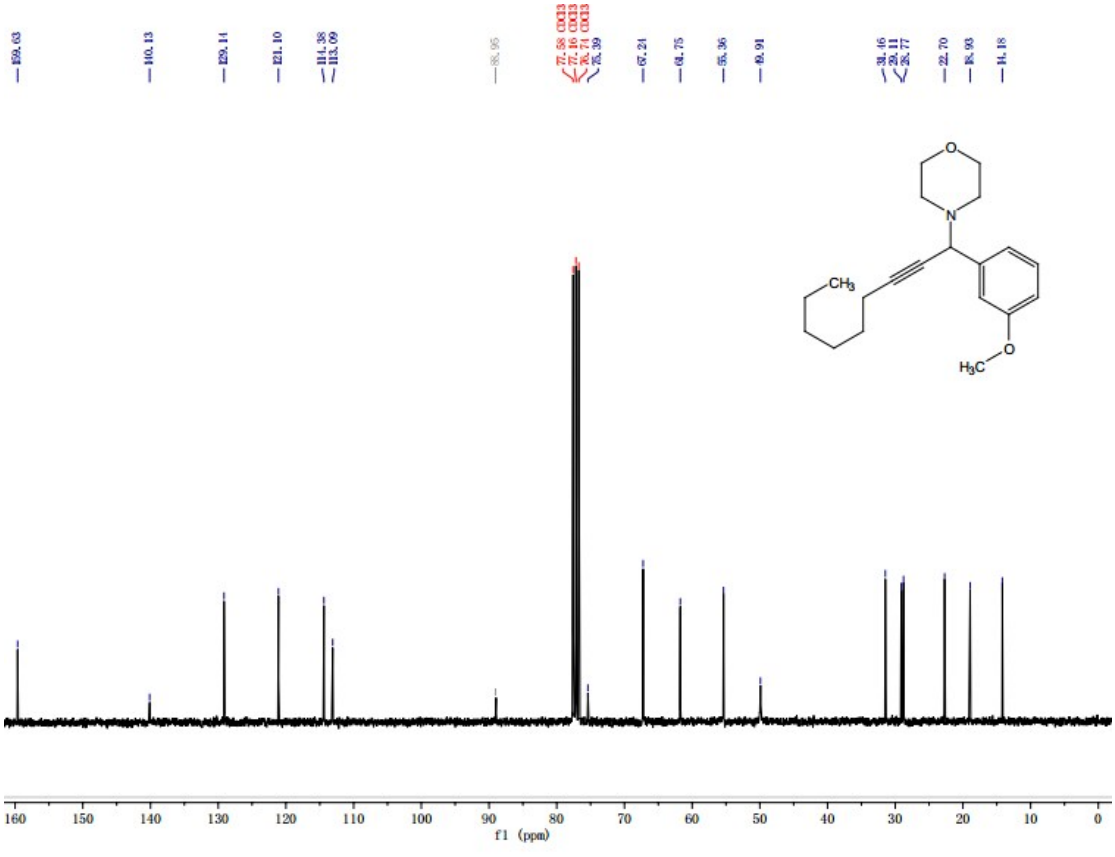
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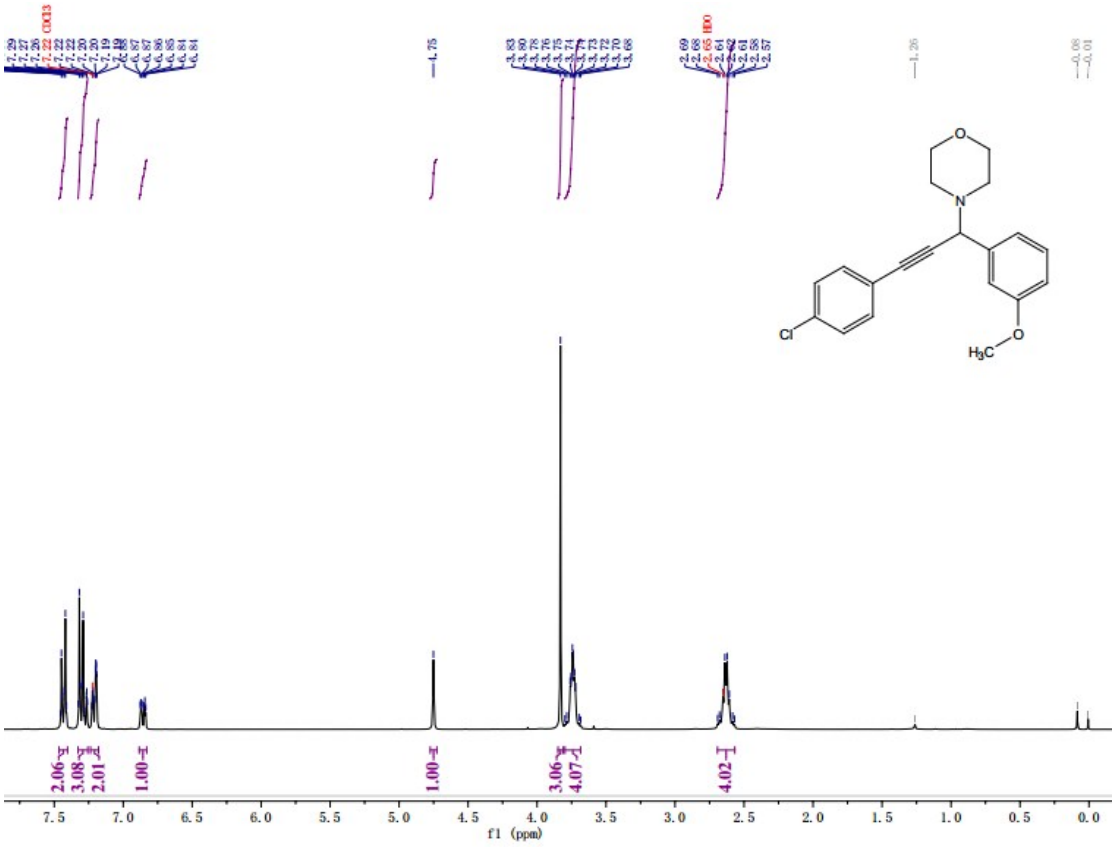
f1 (ppm)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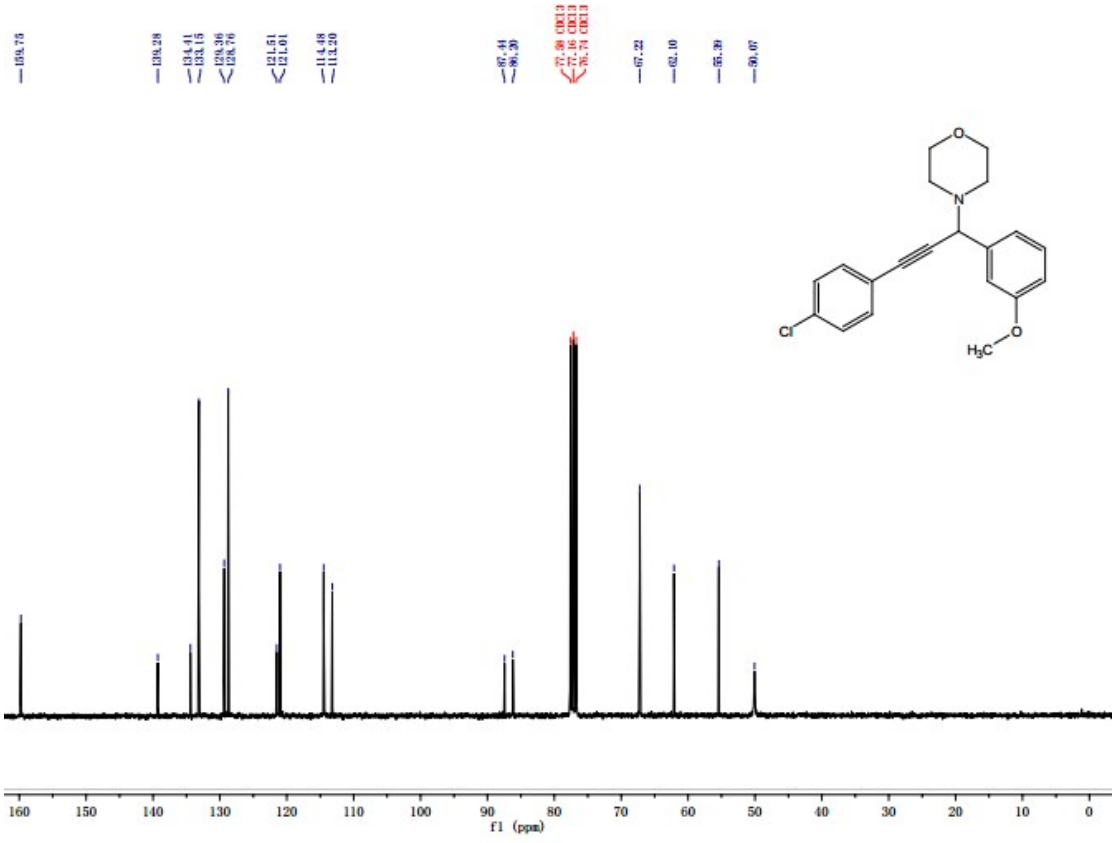
f1 (ppm)