

Supporting Information

Iron-Catalyzed Alkynylation of Aliphatic Tertiary Amines with 1-iodoalkynes to Propargylamines

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Table of Contents

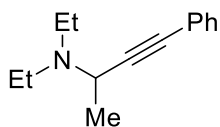
1. General information	S2
2. General procedure and characterization data for product 3	S2
3. References	S9
3. Copies of ^1H , ^{13}C NMR spectra for new compounds	S9

1. General Information

^1H NMR spectra were recorded on Bruker 500 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for CDCl_3 . The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, J , are reported in Hertz (Hz). ^{13}C NMR spectra were obtained at Bruker 125 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in CDCl_3). The NMR yield was determined by ^1H NMR using CH_2Br_2 as an internal standard. APEX II (Bruker Inc.) was used for ESI-HRMS. IR spectra were recorded by a Nicolet 5MX-S infrared spectrometer. Flash column chromatography was performed over silica gel 200-300. All reagents were weighed and handled in air at room temperature. All chemical reagents were purchased from Alfa, Acros, Aldrich, and TCI, J&K and used without further purification.

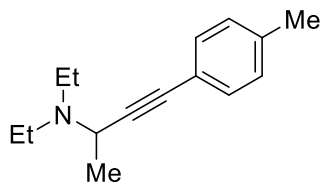
2. General procedure and characterization data for product 3

To a mixture of tertiary aliphatic amine **1** (1.0 mmol), 1-iodoalkynes **2** (0.20 mmol) and FeCl_2 (2.6 mg, 10 mol%), MeCN (1.0 mL) was added under nitrogen at room temperature. *tert*-Butyl hydroperoxide (TBHP-decan) (1.0 mmol) was dropped into the mixture under nitrogen at room temperature. The resulting mixture was stirred at 120 $^\circ\text{C}$ for 12 h. After the mixture was cooled to room temperature, the solvent was removed under reduced pressure. The crude product was purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether) to give the product **3** as yellow oil.

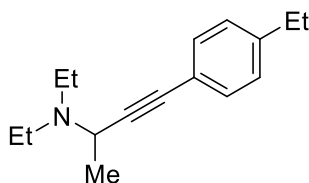


N,N-diethyl-4-phenylbut-3-yn-2-amine (**3a**)^[1]. (35 mg, 86%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3); IR (neat): ν_{max} 2972, 2931, 2871, 2817, 1489, 1443, 1381, 1302, 1184, 1095, 1069, 1049, 765, 691 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.38–7.27 (m, 2H), 7.24–7.14 (m, 3H), 3.81 (q, J = 7.0 Hz, 1H), 2.72–2.58 (m, 2H), 2.49–2.36 (m, 2H), 1.33 (d, J = 7.0 Hz, 3H), 1.03 (t, J = 7.0 Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 131.5, 128.1, 127.6, 123.4, 89.4, 84.0,

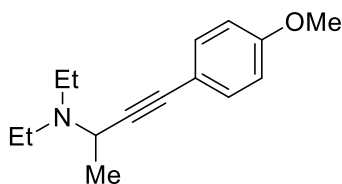
48.2, 44.6, 20.1, 13.6; HRMS (ESI) calcd for C₁₄H₁₉N [M +H⁺], 202.1590; found: 202.1598.



***N,N*-diethyl-4-(*p*-tolyl)but-3-yn-2-amine (3b).** (35 mg, 81%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3); IR (neat): ν_{\max} 2989, 2956, 1656, 1509, 1467, 1302, 1255, 1186, 1095, 815, 732, 525 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.30 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 3.89 (q, *J* = 7.0 Hz, 1H), 2.78–2.67 (m, 2H), 2.56–2.46 (m, 2H), 2.33 (s, 3H), 1.41 (d, *J* = 7.0 Hz, 3H), 1.11 (t, *J* = 7.0 Hz, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 137.7, 131.5, 128.9, 120.4, 88.7, 84.1, 48.3, 44.6, 21.4, 20.1, 13.6; HRMS (ESI) calcd for C₁₅H₂₁N [M +H⁺], 216.1747; found: 216.1754.

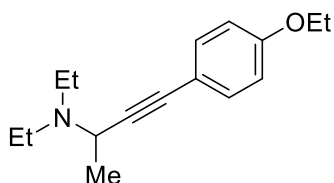


***N,N*-diethyl-4-(4-ethylphenyl)but-3-yn-2-amine (3c).** (35 mg, 76%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3); IR (neat): ν_{\max} 2966, 1661, 1509, 1456, 1379, 1302, 1185, 1095, 832, 731, 539 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.33 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 3.89 (q, *J* = 7.0 Hz, 1H), 2.79–2.67 (m, 2H), 2.63 (q, *J* = 7.5 Hz, 2H), 2.57–2.45 (m, 2H), 1.41 (d, *J* = 7.0 Hz, 3H), 1.21 (t, *J* = 7.5 Hz, 3H), 1.11 (t, *J* = 7.0 Hz, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 144.1, 131.6, 127.7, 120.6, 88.7, 84.1, 48.3, 44.6, 28.7, 20.2, 15.4, 13.6; HRMS (ESI) calcd for C₁₆H₂₃N [M +H⁺], 230.1903; found: 230.1911.

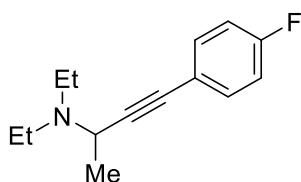


***N,N*-diethyl-4-(4-methoxyphenyl)but-3-yn-2-amine (3d).** (36 mg, 78%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3); IR

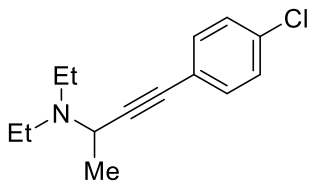
(neat): ν_{\max} 2969, 2930, 2836, 1607, 1509, 1465, 1379, 1289, 1246, 1172, 1095, 1034, 831, 792 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.34 (d, $J = 8.5$ Hz, 2H), 6.81 (d, $J = 8.5$ Hz, 2H), 3.88 (q, $J = 7.0$ Hz, 1H), 3.79 (s, 3H), 2.77–2.67 (m, 2H), 2.56–2.45 (m, 2H), 1.40 (d, $J = 7.0$ Hz, 3H), 1.10 (t, $J = 7.0$ Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 159.2, 133.0, 115.6, 113.8, 87.9, 83.7, 55.2, 48.3, 44.6, 20.2, 13.6; HRMS (ESI) calcd for $\text{C}_{15}\text{H}_{21}\text{NO}$ [$\text{M} + \text{H}^+$], 232.1696; found: 232.1702.



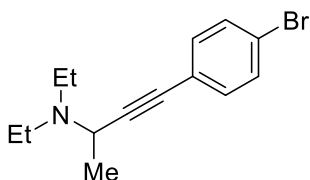
4-(4-ethoxyphenyl)-*N,N*-diethylbut-3-yn-2-amine (3e). (36 mg, 73%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, $R_f = 0.3$); IR (neat): ν_{\max} 2975, 2929, 1606, 1508, 1476, 1380, 1286, 1245, 1172, 1115, 1094, 1048, 922, 834, 534 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.32 (d, $J = 8.5$ Hz, 2H), 6.80 (d, $J = 8.5$ Hz, 2H), 4.01 (q, $J = 7.0$ Hz, 2H), 3.88 (q, $J = 7.0$ Hz, 1H), 2.79–2.64 (m, 2H), 2.56–2.42 (m, 2H), 1.40 (t, $J = 7.0$ Hz, 6H), 1.10 (t, $J = 7.0$ Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 158.5, 133.0, 115.5, 114.3, 87.8, 83.8, 63.4, 48.3, 44.6, 20.2, 14.7, 13.7; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{23}\text{NO}$ [$\text{M} + \text{H}^+$], 246.1852; found: 246.1861.



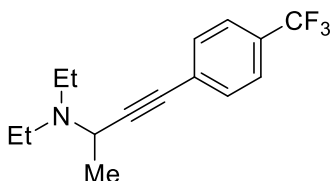
***N,N*-diethyl-4-(4-fluorophenyl)but-3-yn-2-amine (3f).** (33 mg, 76%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, $R_f = 0.3$); IR (neat): ν_{\max} 2925, 1601, 1507, 1468, 1379, 1303, 1231, 1155, 1093, 835, 527 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.42–7.33 (m, 2H), 7.01–6.93 (m, 2H), 3.88 (q, $J = 7.0$ Hz, 1H), 2.79–2.65 (m, 2H), 2.55–2.43 (m, 2H), 1.40 (d, $J = 7.0$ Hz, 3H), 1.10 (t, $J = 7.0$ Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 162.2 (d, $J = 246.2$ Hz), 133.4 (d, $J = 8.8$ Hz), 119.6, 115.4 (d, $J = 21.2$ Hz), 89.2, 82.9, 48.2, 44.6, 20.0, 13.7; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{18}\text{FN}$ [$\text{M} + \text{H}^+$], 220.1496; found: 220.1505.



4-(4-chlorophenyl)-*N,N*-diethylbut-3-yn-2-amine (3g). (41 mg, 87%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, $R_f = 0.3$); IR (neat): ν_{\max} 2971, 2928, 1488, 1467, 1380, 1301, 1251, 1185, 1091, 1014, 827, 751, 521 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.32 (d, $J = 8.5$ Hz, 2H), 7.25 (d, $J = 8.5$ Hz, 2H), 3.88 (q, $J = 7.0$ Hz, 1H), 2.77–2.65 (m, 2H), 2.53–2.44 (m, 2H), 1.40 (d, $J = 7.0$ Hz, 3H), 1.10 (t, $J = 7.0$ Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 133.7, 132.8, 128.5, 122.0, 90.6, 82.9, 48.3, 44.6, 19.9, 13.6; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{18}\text{ClN}$ [$\text{M} + \text{H}^+$], 236.1201; found: 236.1208.

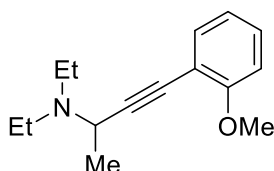


4-(4-bromophenyl)-*N,N*-diethylbut-3-yn-2-amine (3h). (37 mg, 66%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, $R_f = 0.3$); IR (neat): ν_{\max} 2971, 2929, 1486, 1379, 1301, 1251, 1185, 1095, 1069, 822, 744, 521 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.41 (d, $J = 8.5$ Hz, 2H), 7.26 (d, $J = 8.5$ Hz, 2H), 3.88 (q, $J = 7.0$ Hz, 1H), 2.77–2.63 (m, 2H), 2.54–2.42 (m, 2H), 1.40 (d, $J = 7.0$ Hz, 3H), 1.10 (t, $J = 7.0$ Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 133.1, 131.4, 122.4, 121.9, 90.9, 83.0, 48.3, 44.7, 20.0, 13.7; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{18}\text{BrN}$ [$\text{M} + \text{H}^+$], 280.0695; found: 280.0707.

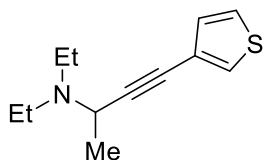


4-(4-ethoxyphenyl)-*N,N*-diethylbut-3-yn-2-amine (3i). (36 mg, 66%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, $R_f = 0.3$); IR (neat): ν_{\max} 2921, 2851, 1632, 1469, 1323, 1168, 1131, 1066, 841 cm^{-1} ; ^1H NMR (500 MHz,

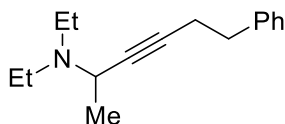
CDCl₃) δ 7.54 (d, $J = 8.0$ Hz, 2H), 7.50 (d, $J = 8.0$ Hz, 2H), 3.91 (q, $J = 7.0$ Hz, 1H), 2.79–2.67 (m, 2H), 2.56–2.44 (m, 2H), 1.42 (d, $J = 7.0$ Hz, 3H), 1.11 (t, $J = 7.0$ Hz, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 131.9, 129.5(q, $J = 32.5$ Hz), 127.7, 125.1(q, $J = 3.8$ Hz), 124.0 (q, $J = 270.3$ Hz), 92.4, 82.9, 48.3, 44.7, 19.9, 13.7; HRMS (ESI) calcd for C₁₅H₁₈F₃N [M + H⁺], 270.1464; found: 270.1477.



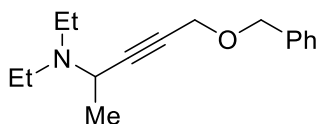
***N,N*-diethyl-4-(2-methoxyphenyl)but-3-yn-2-amine (3j).** (36 mg, 78%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, $R_f = 0.3$); IR (neat): ν_{\max} 2968, 2928, 2850, 1633, 1596, 1492, 1463, 1433, 1379, 1293, 1260, 1182, 1095, 1026, 751, 621 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.41–7.33 (m, 1H), 7.29–7.20 (m, 1H), 6.92–6.81 (m, 2H), 3.95 (q, $J = 7.0$ Hz, 1H), 3.85 (s, 3H), 2.79–2.67 (m, 2H), 2.60–2.48 (m, 2H), 1.43 (d, $J = 7.0$ Hz, 3H), 1.11 (t, $J = 7.0$ Hz, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 159.9, 133.5, 129.1, 120.3, 112.7, 110.7, 93.8, 80.1, 55.7, 48.6, 44.6, 20.2, 13.7; HRMS (ESI) calcd for C₁₅H₂₁NO [M + H⁺], 232.1696; found: 232.1705.



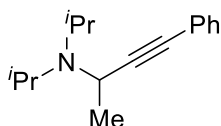
***N,N*-diethyl-4-(thiophen-3-yl)but-3-yn-2-amine (3k).** (30 mg, 72%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.3$); IR (neat): ν_{\max} 2969, 2927, 1660, 1519, 1468, 1379, 1261, 1186, 1095, 959, 779, 626 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.42–7.30 (m, 1H), 7.30–7.17 (m, 1H), 7.13–7.02 (m, 1H), 3.88 (q, $J = 7.0$ Hz, 1H), 2.78–2.66 (m, 2H), 2.56–2.42 (m, 2H), 1.40 (d, $J = 7.0$ Hz, 3H), 1.10 (t, $J = 7.0$ Hz, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 130.0, 127.9, 125.0, 122.4, 89.0, 78.9, 48.3, 44.6, 20.0, 13.7.; HRMS (ESI) calcd for C₁₂H₁₇NS [M + H⁺], 208.1154; found: 208.1161.



***N,N*-diethyl-6-phenylhex-3-yn-2-amine (3l).** (36 mg, 78%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3); IR (neat): ν_{\max} 2966, 2922, 2850, 1632, 1455, 1383, 1186, 1086, 747, 697 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.33–7.24 (m, 2H), 7.26–7.15 (m, 3H), 3.63 (q, J = 7.0 Hz, 1H), 2.84–2.76 (m, 2H), 2.64–2.54 (m, 2H), 2.52–2.46 (m, 2H), 2.38–2.26 (m, 2H), 1.27 (d, J = 7.0 Hz, 3H), 1.03 (t, J = 7.0 Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 140.8, 128.5, 128.2, 126.1, 83.0, 80.4, 47.7, 44.4, 35.5, 20.8, 20.2, 13.6; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{23}\text{N}$ [$\text{M} + \text{H}^+$], 230.1903; found: 230.1911.

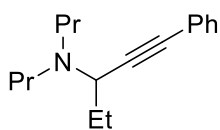


5-(benzyloxy)-*N,N*-diethylpent-3-yn-2-amine (3m). (32 mg, 65%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:1, R_f = 0.3); IR (neat): ν_{\max} 2969, 2925, 2851, 1632, 1455, 1381, 1302, 1261, 1202, 1073, 1027, 736, 698 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.40–7.27 (m, 5H), 4.60 (s, 2H), 4.20 (s, 2H), 3.75 (q, J = 7.0 Hz, 1H), 2.75–2.62 (m, 2H), 2.48–2.36 (m, 2H), 1.34 (d, J = 7.0 Hz, 3H), 1.08 (t, J = 7.0 Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 137.6, 128.4, 128.1, 127.8, 86.4, 79.4, 71.3, 57.5, 47.8, 44.6, 20.1, 13.7; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{23}\text{NO}$ [$\text{M} + \text{H}^+$], 246.1852; found: 246.1859.

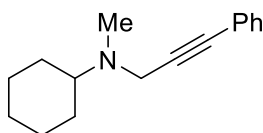


***N,N*-diisopropyl-4-phenylbut-3-yn-2-amine (3n)** ^[1]. (36 mg, 79%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3); ^1H NMR (500 MHz, CDCl_3) δ 7.33–7.27 (m, 2H), 7.24–7.16 (m, 3H), 3.82 (q, J = 7.0 Hz, 1H), 3.24–3.13 (m, 2H), 1.29 (d, J = 7.0 Hz, 3H), 1.11 (d, J = 6.5 Hz, 6H), 0.98 (d, J = 6.5 Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 131.3, 128.2, 127.4, 124.1, 94.5, 81.9, 46.0,

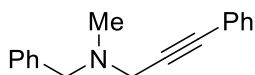
41.6, 24.1, 23.7, 20.5;



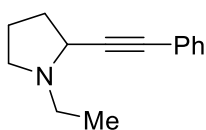
1-phenyl-*N,N*-dipropylpent-1-yn-3-amine (3o)^[1]. (32 mg, 66%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3); ^1H NMR (500 MHz, CDCl_3) δ 7.45–7.37 (m, 2H), 7.32–7.21 (m, 3H), 3.52 (t, J = 7.5 Hz, 1H), 2.60–2.47 (m, 2H), 2.48–2.36 (m, 2H), 1.78–1.63 (m, 2H), 1.59–1.37 (m, 4H), 1.04 (t, J = 7.0 Hz, 3H), 0.90 (t, J = 7.0 Hz, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 131.7, 128.2, 127.6, 123.7, 89.2, 84.4, 56.0, 53.6, 27.4, 21.7, 12.0, 11.4;



***N*-methyl-*N*-(3-phenylprop-2-yn-1-yl)cyclohexanamine (3p)**^[1]. (30 mg, 66%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3); ^1H NMR (500 MHz, CDCl_3) δ 7.47–7.38 (m, 2H), 7.34–7.26 (m, 3H), 3.63 (s, 2H), 2.49–2.36 (m, 4H), 2.01–1.92 (m, 2H), 1.83–1.74 (m, 2H), 1.66–1.56 (m, 1H), 1.34–1.11 (m, 5H); ^{13}C NMR (125 MHz, CDCl_3) δ 131.6, 128.2, 127.9, 123.4, 85.6, 84.8, 61.0, 43.7, 38.5, 29.8, 26.1, 25.5;



***N*-benzyl-*N*-methyl-3-phenylprop-2-yn-1-amine (3q)**^[2]. (25 mg, 53%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.6); ^1H NMR (500 MHz, CDCl_3) δ 7.54–7.24 (m, 10H), 3.65 (s, 2H), 3.53 (s, 2H), 2.42 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 138.4, 131.7, 129.2, 128.4, 128.3, 128.0, 127.2, 123.3, 85.7, 84.4, 60.2, 45.7, 42.0;



1-ethyl-2-(phenylethynyl)pyrrolidine (3r). (26 mg, 66%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3); IR (neat): ν_{max} 2922,

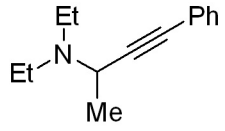
2850, 1631, 1468, 755, 532 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.47–7.38 (m, 2H), 7.33–7.21 (m, 3H), 3.64–3.53 (m, 1H), 3.03–2.92 (m, 1H), 2.93–2.84 (m, 1H), 2.57–2.38 (m, 2H), 2.25–2.14 (m, 1H), 2.11–1.89 (m, 2H), 1.89–1.77 (m, 1H), 1.18 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 131.7, 128.2, 127.9, 123.3, 88.6, 84.4, 54.9, 51.5, 47.4, 31.8, 22.0, 13.7; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{17}\text{N}$ [$\text{M} + \text{H}^+$], 200.1434; found: 200.1443.

3. References

- (1) J. Xie, S. Shi, T. Zhang, N. Mehrkens, M. Rudolph, A. S. K. Hashmi, *Angew. Chem. Int. Ed.* 2015, **54**, 6046.
- (2) Z.-P. Li, C.-J. Li, *J. Am. Chem. Soc.* 2004, **126**, 11810.

4. Copies of ^1H , ^{13}C NMR spectra for new compounds

PROTON_01
M-3ap



7.33
7.32
7.32
7.31
7.21
7.21
7.20
7.19
7.19
7.18
7.18

3.83
3.82
3.80
3.79
2.67
2.65
2.64
2.63
2.61
2.44
2.43
2.42
1.33
1.32
1.04
1.03
1.01



E (m)
7.18
D (m)
7.32
HH



A (q)
3.81
H



C (m)
2.42
B (m)
2.65
HH



G (t)
1.03
F (d)
1.33
H H

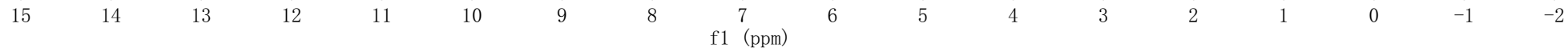


1.98
2.98

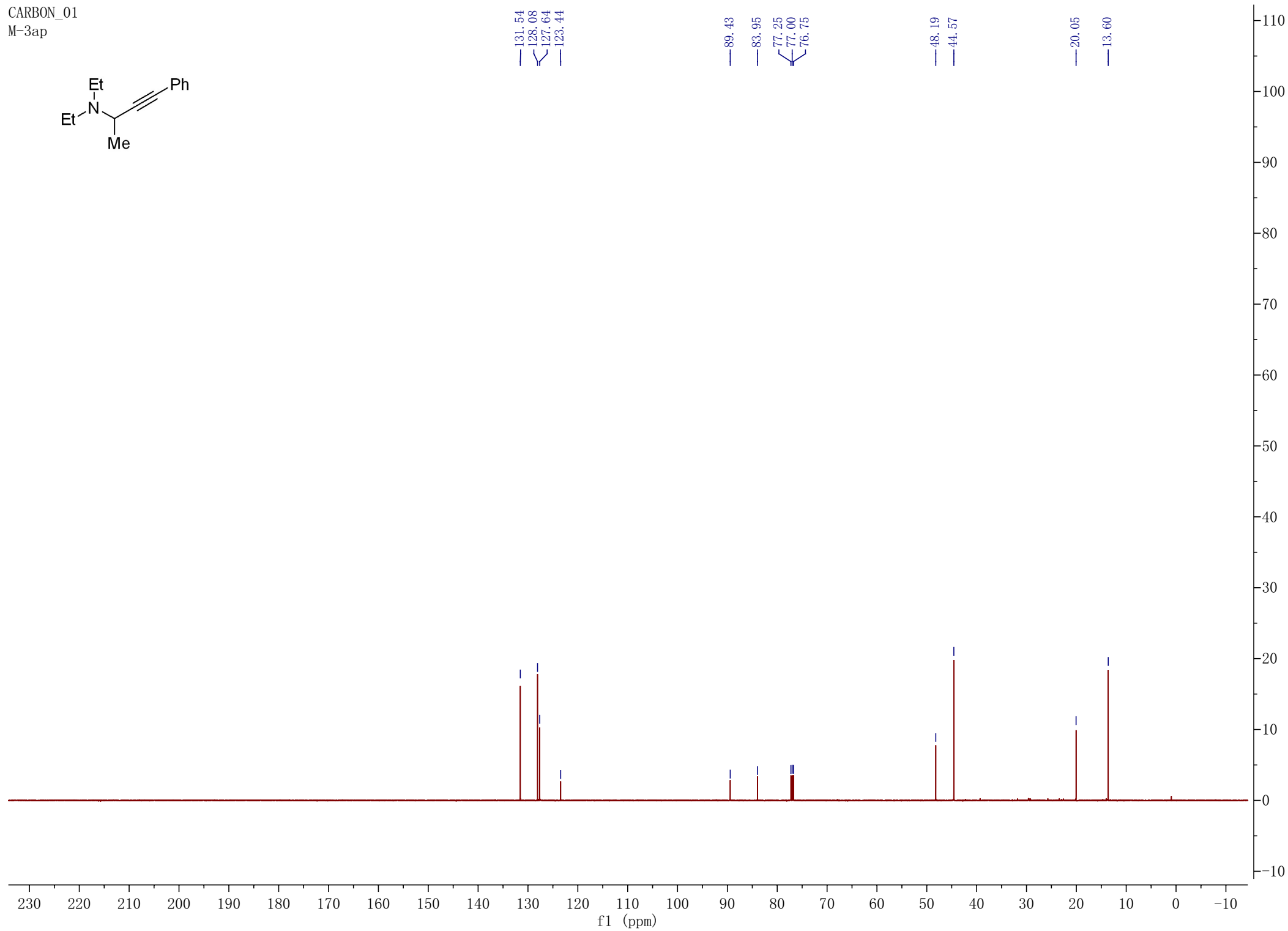
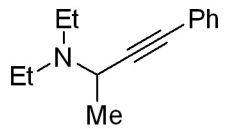
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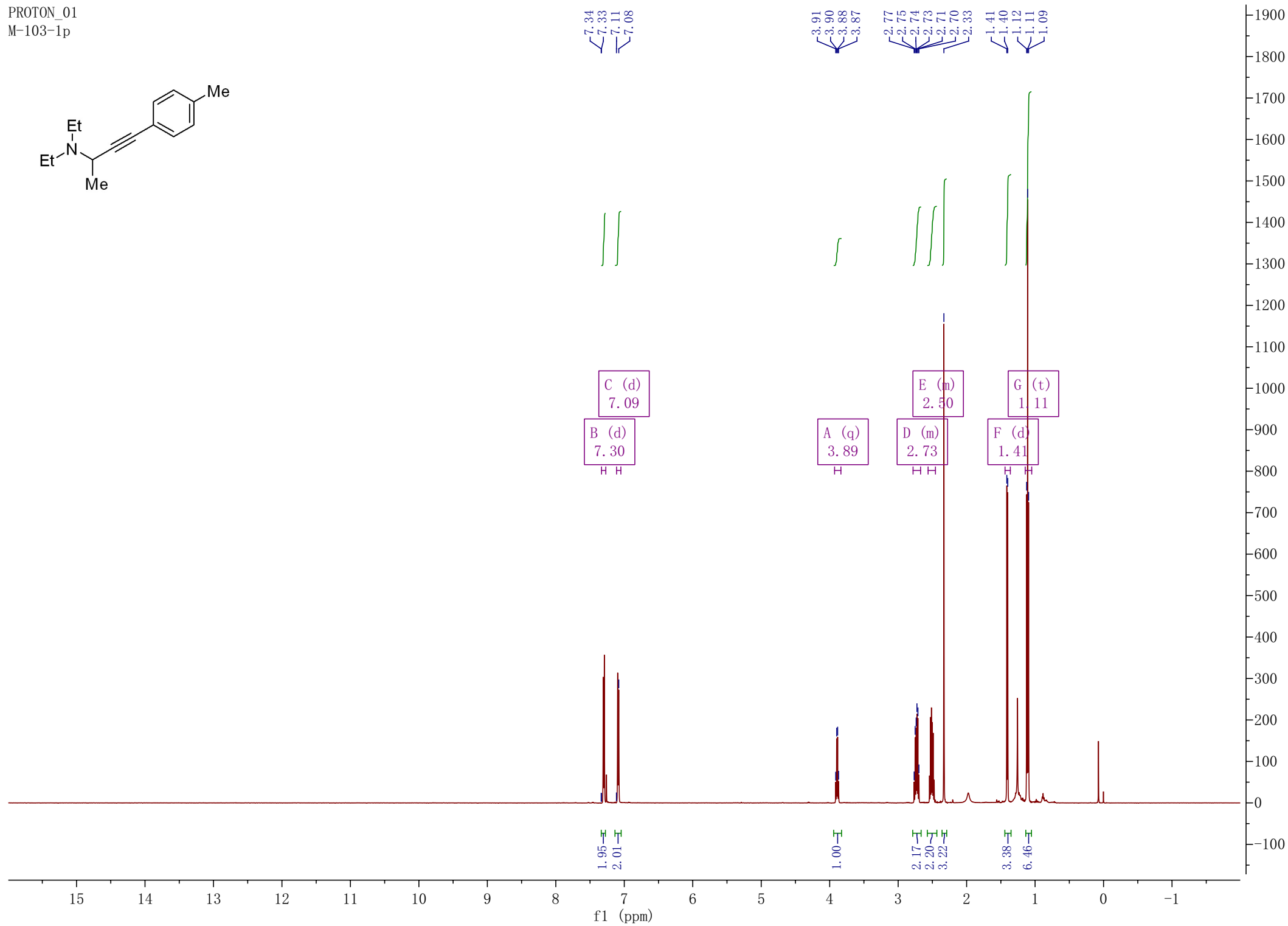
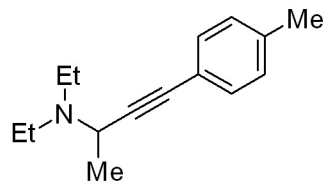
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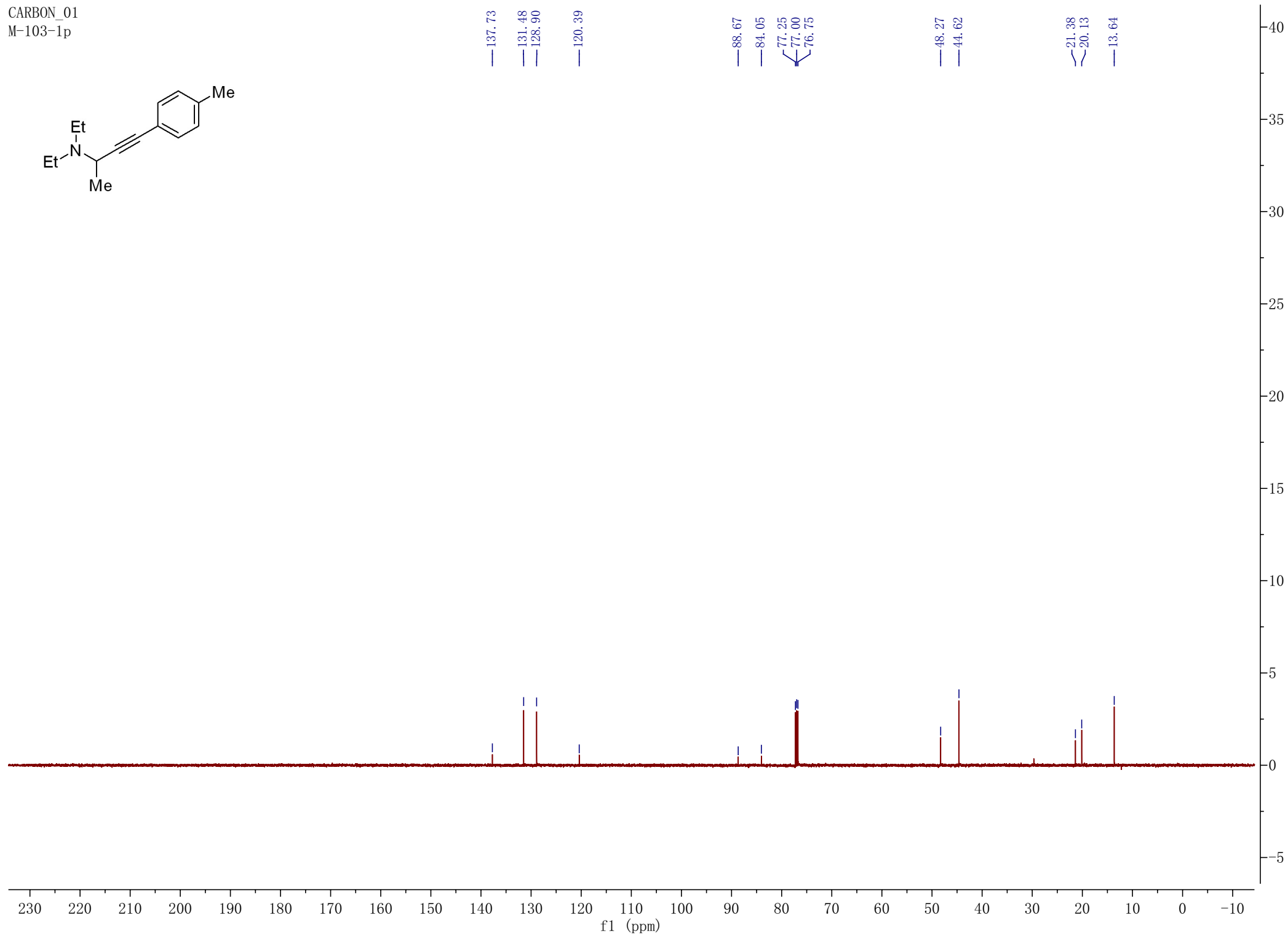
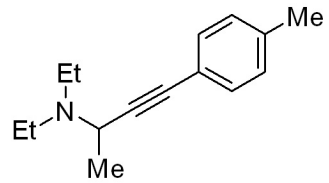
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M-3ap



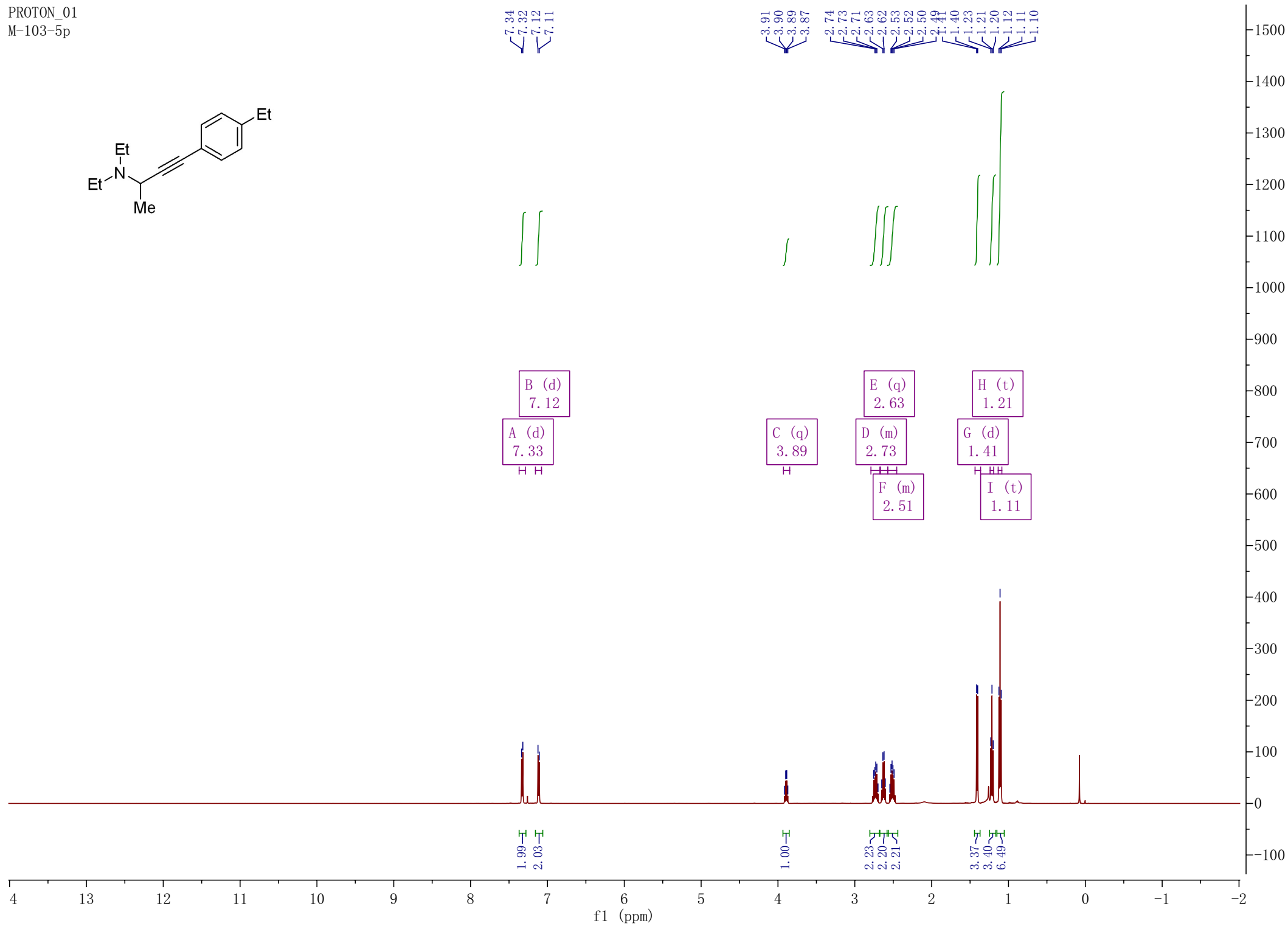
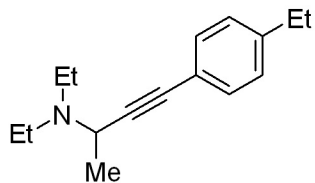
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M-103-1p



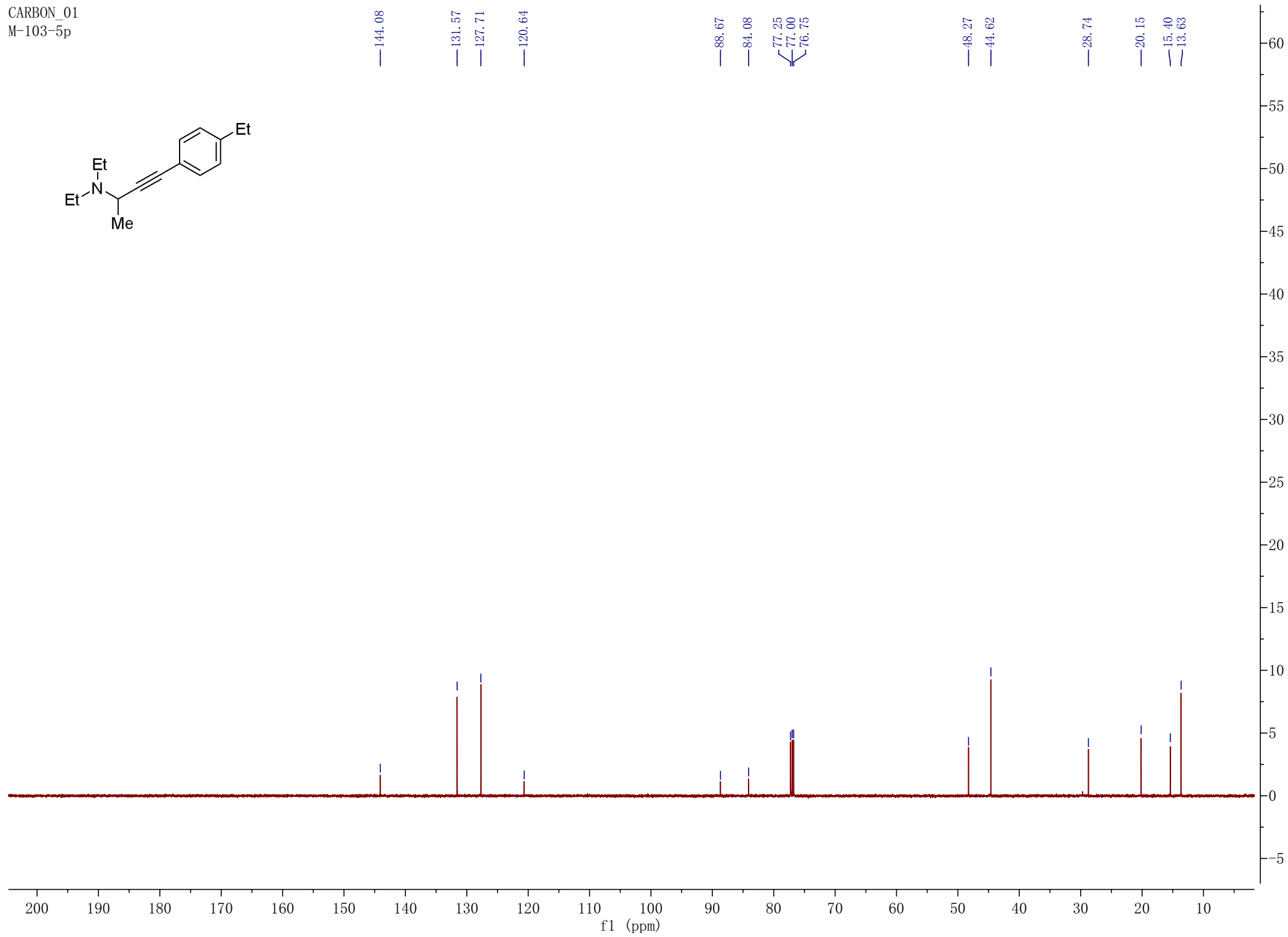
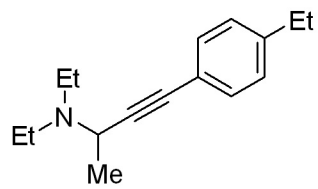
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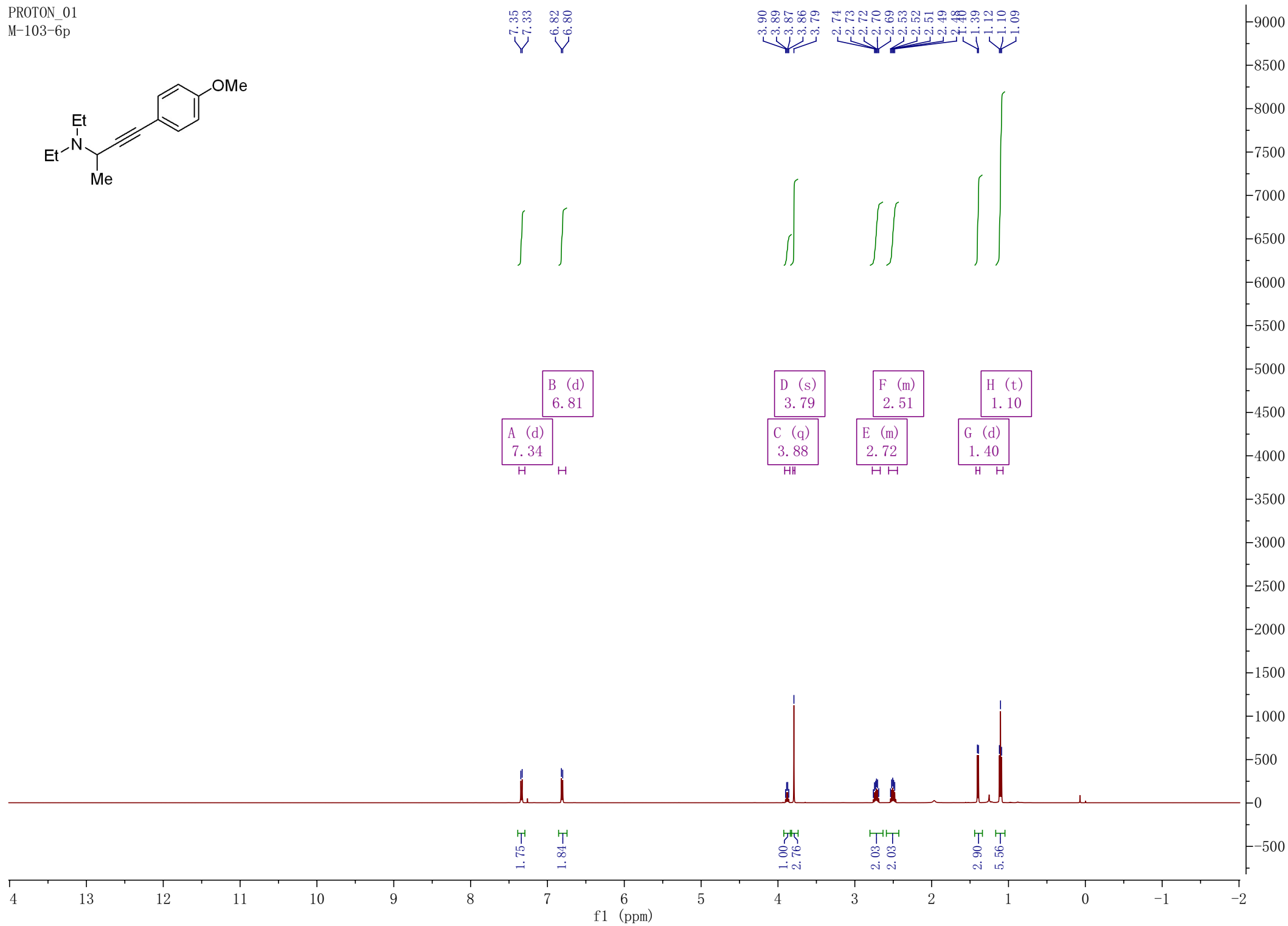
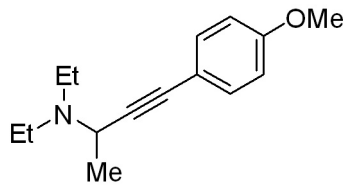
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M-103-5p



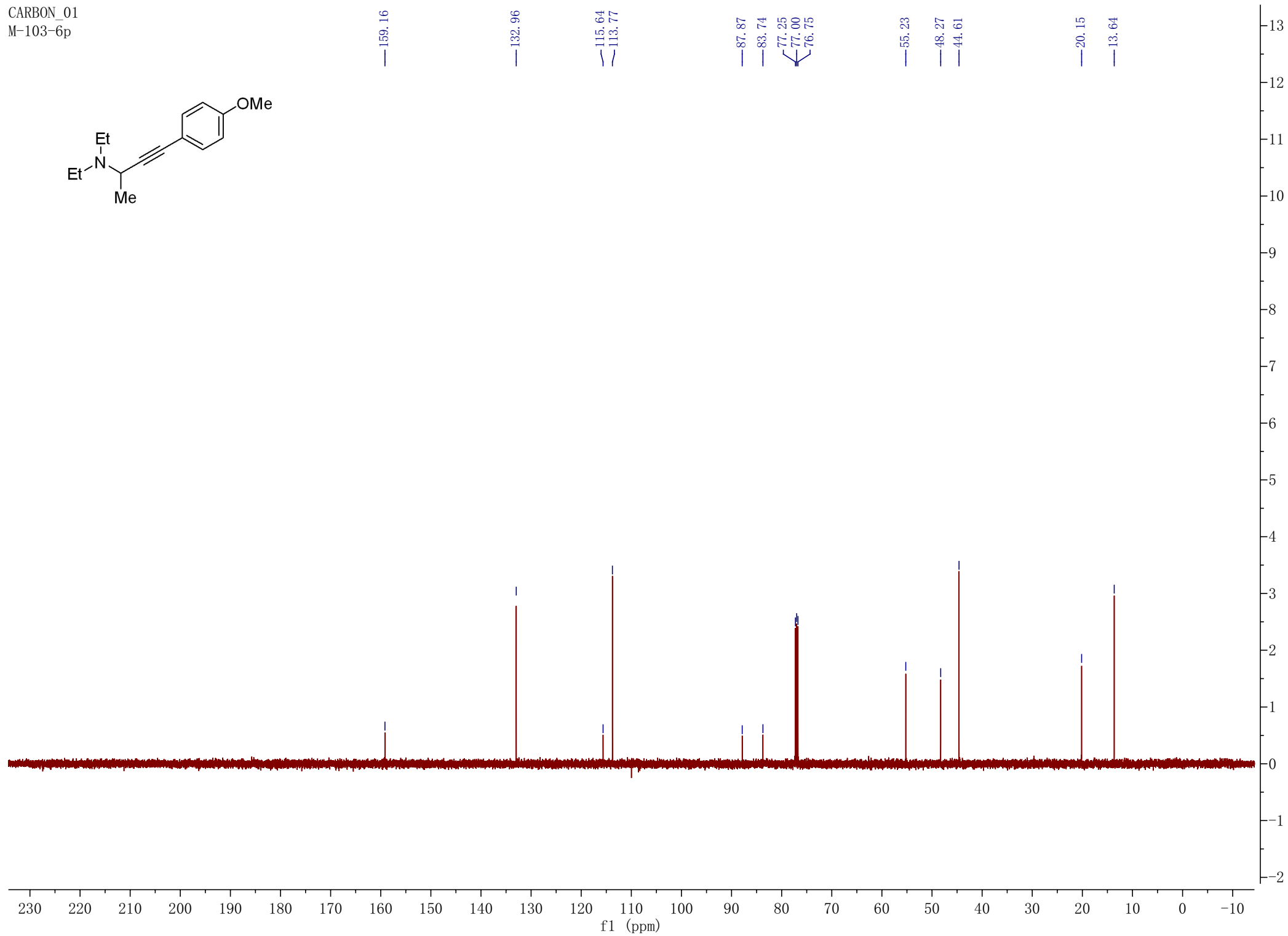
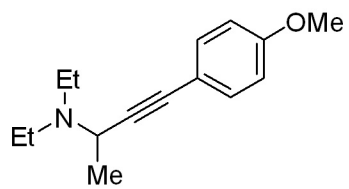
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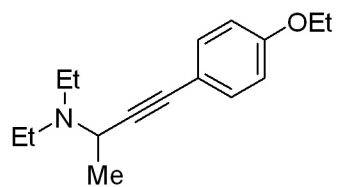
PROTON_01
M-103-6p



CARBON_01
M-103-6p



PROTON_01
M-103-7p



7.33
7.32

6.81
6.79

4.03
4.02
4.01
3.99
3.90
3.88
3.87
3.86

2.76
2.74
2.73
2.72
2.70
2.69
2.53
2.52
2.50
2.49
2.48
2.46

1.41
1.40
1.40
1.39
1.12
1.10
1.09



A (d)
7.32

B (d)
6.80

C (q)
4.01

D (q)
3.88

E (m)
2.71

F (m)
2.50

G (t)
1.40

H (t)
1.10

1.79

1.89

1.92

1.00

2.05

2.11

5.50

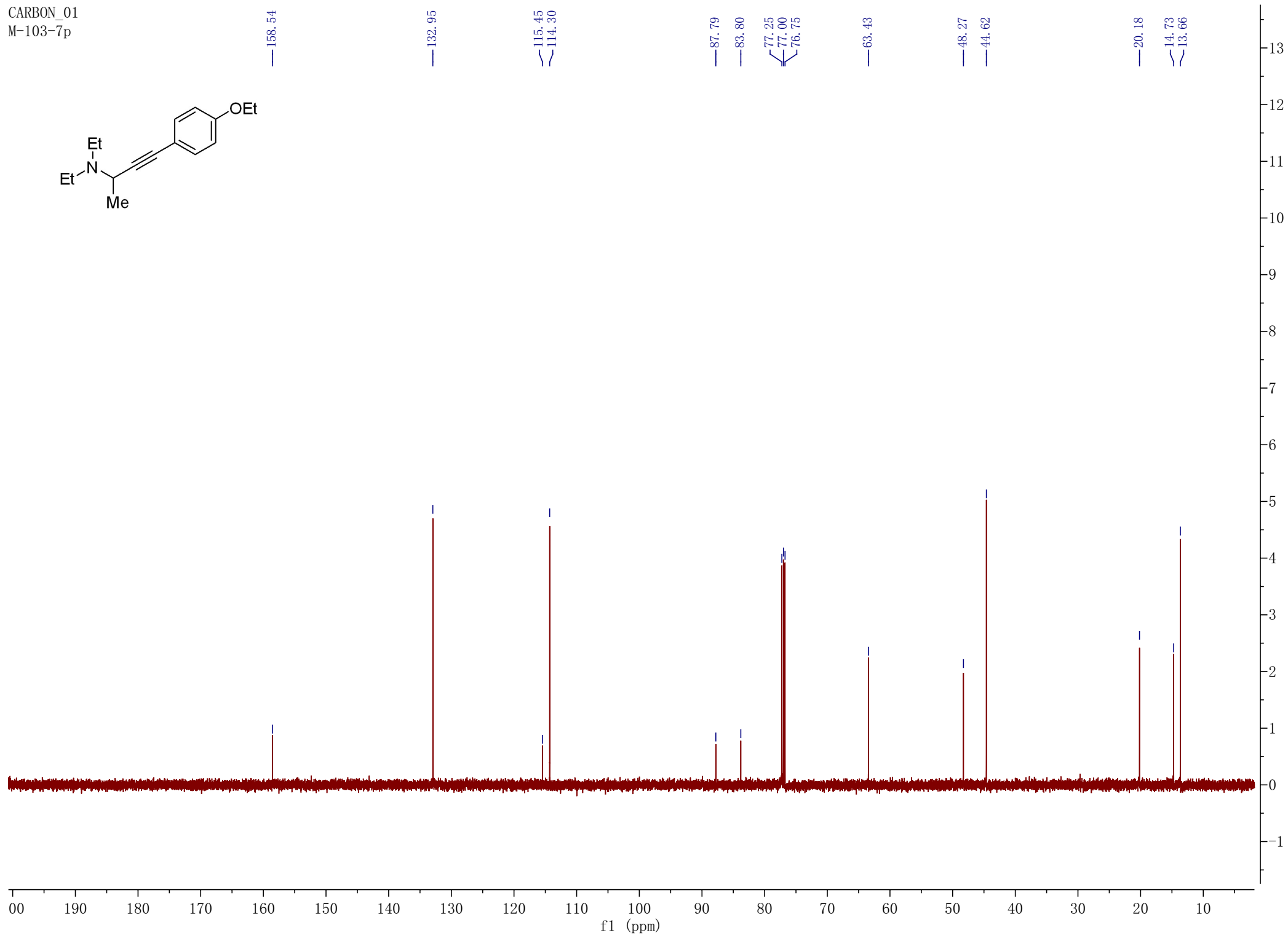
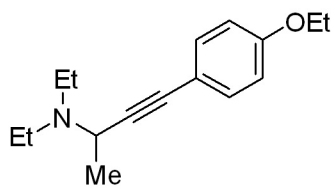
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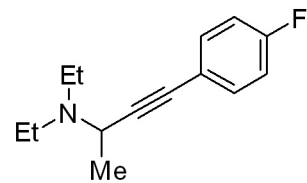
f1 (ppm)

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4000
3500
3000
2500
2000
1500
1000
500
0

CARBON_01
M-103-7p



PROTON_01
M-103-2p



7.39
7.38
7.37
7.36
7.00
6.99
6.97
6.95

3.90
3.89
3.87
3.86

2.73
2.72
2.70
2.51
2.50
2.49
2.47

1.41
1.39
1.12
1.10
1.09



A (m)
7.37
B (m)
6.98

C (q)
3.88

D (m)
2.73
E (m)
2.50

F (d)
1.40
G (t)
1.10

1.75

1.77

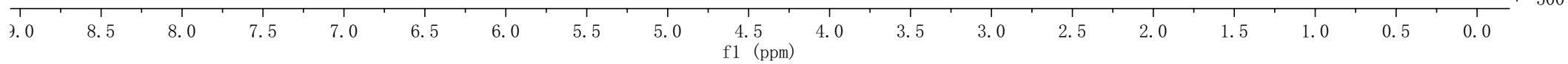
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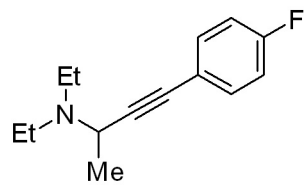
2.14

3.12

5.98



CARBON_01
M-103-2p



163.14
161.17

133.45
133.38

119.56
115.46
115.29

89.19

82.92

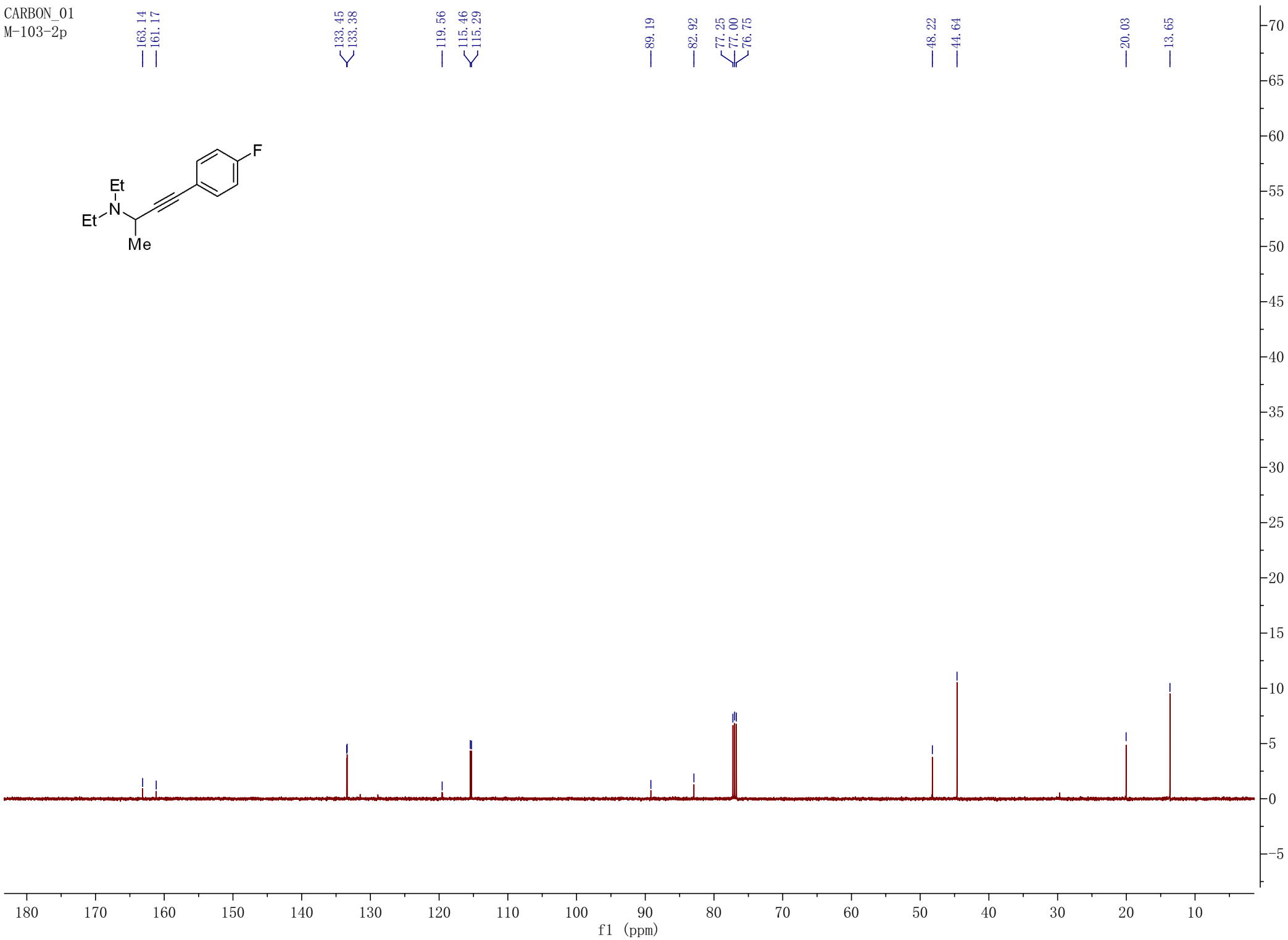
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77.00
76.75

48.22

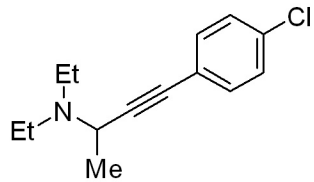
44.64

20.03

13.65



PROTON_01
M-103-3p



7.33
7.31
7.26
7.24
7.21

3.90
3.89
3.87
3.86

2.75
2.74
2.72
2.71
2.70
2.68
2.52
2.51
2.49
2.48
2.47
2.45

1.40
1.39
1.12
1.10
1.09



B (d)
7.25
A (d)
7.32

C (q)
3.88

E (m)
2.49
D (m)
2.72

G (t)
1.10
F (d)
1.40

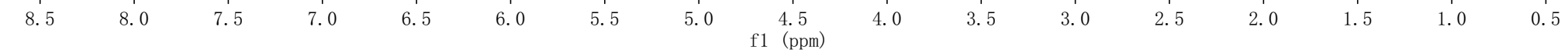
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1.96

1.00

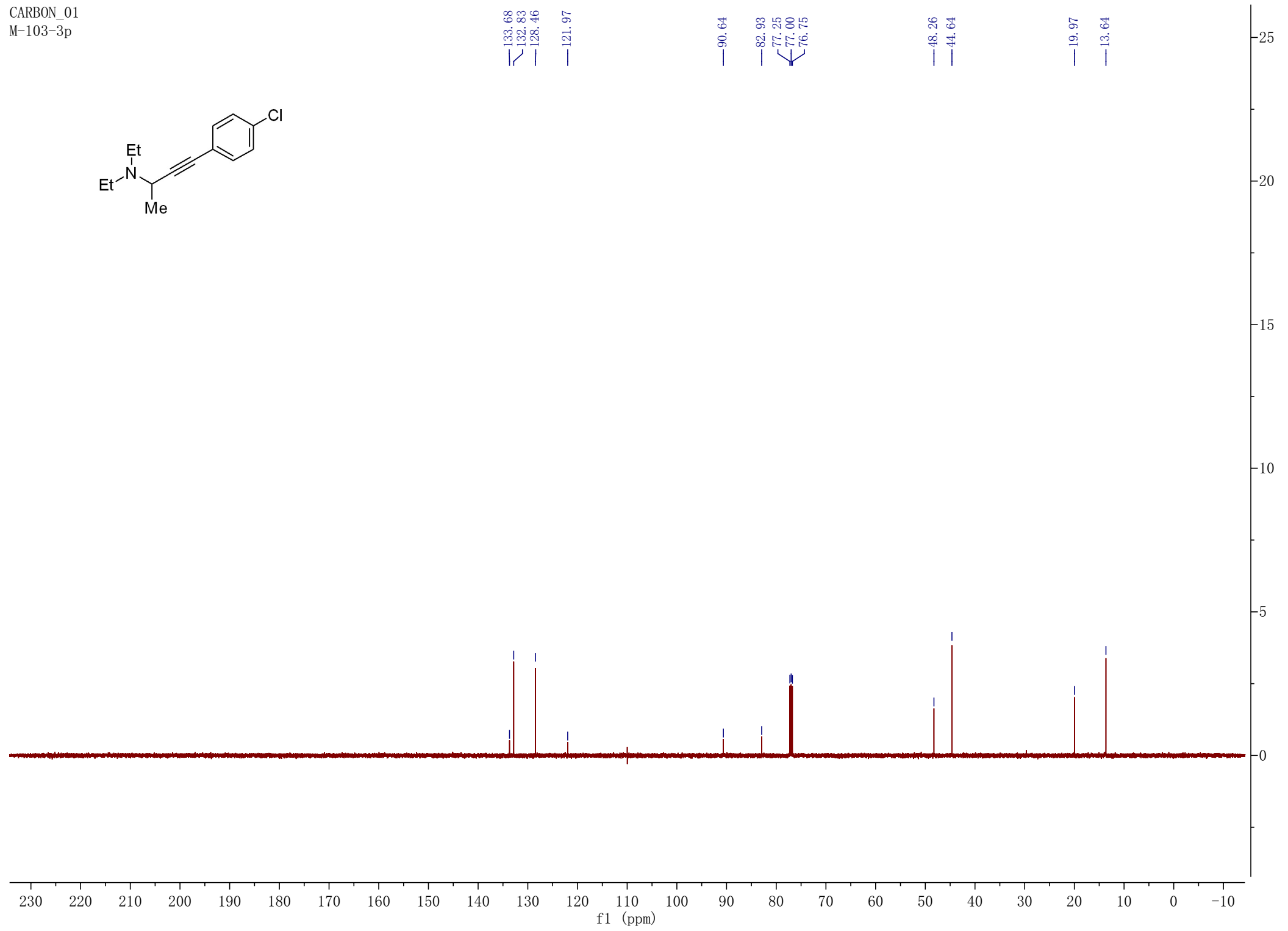
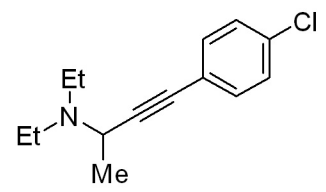
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3.18

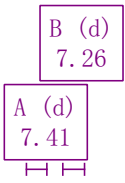
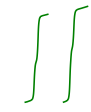
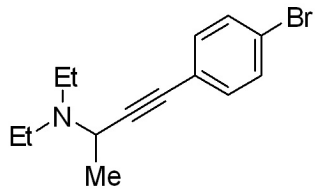
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CARBON_01
M-103-3p



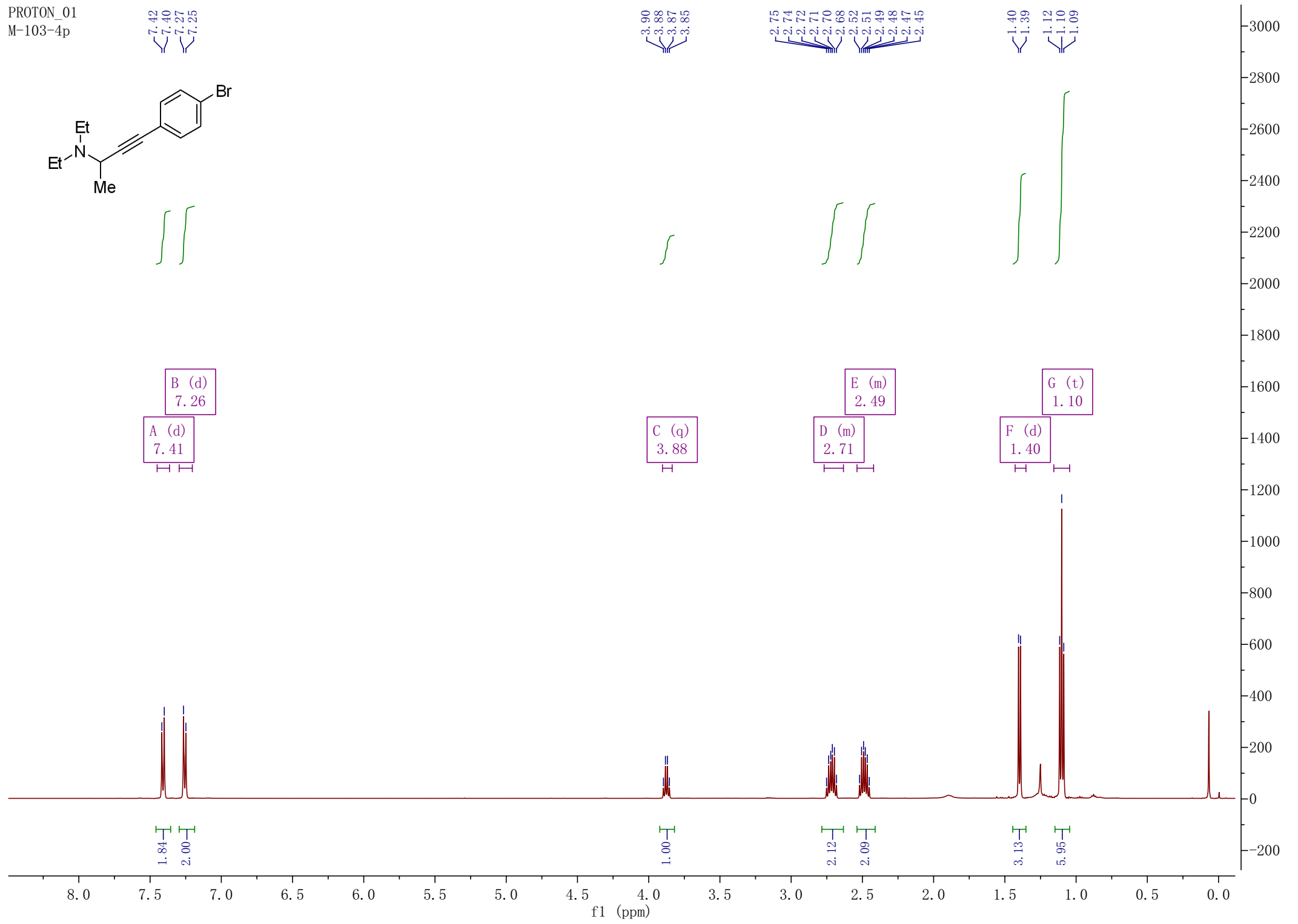
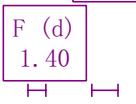
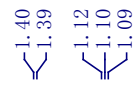
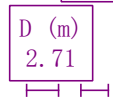
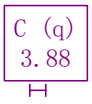
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M-103-4p



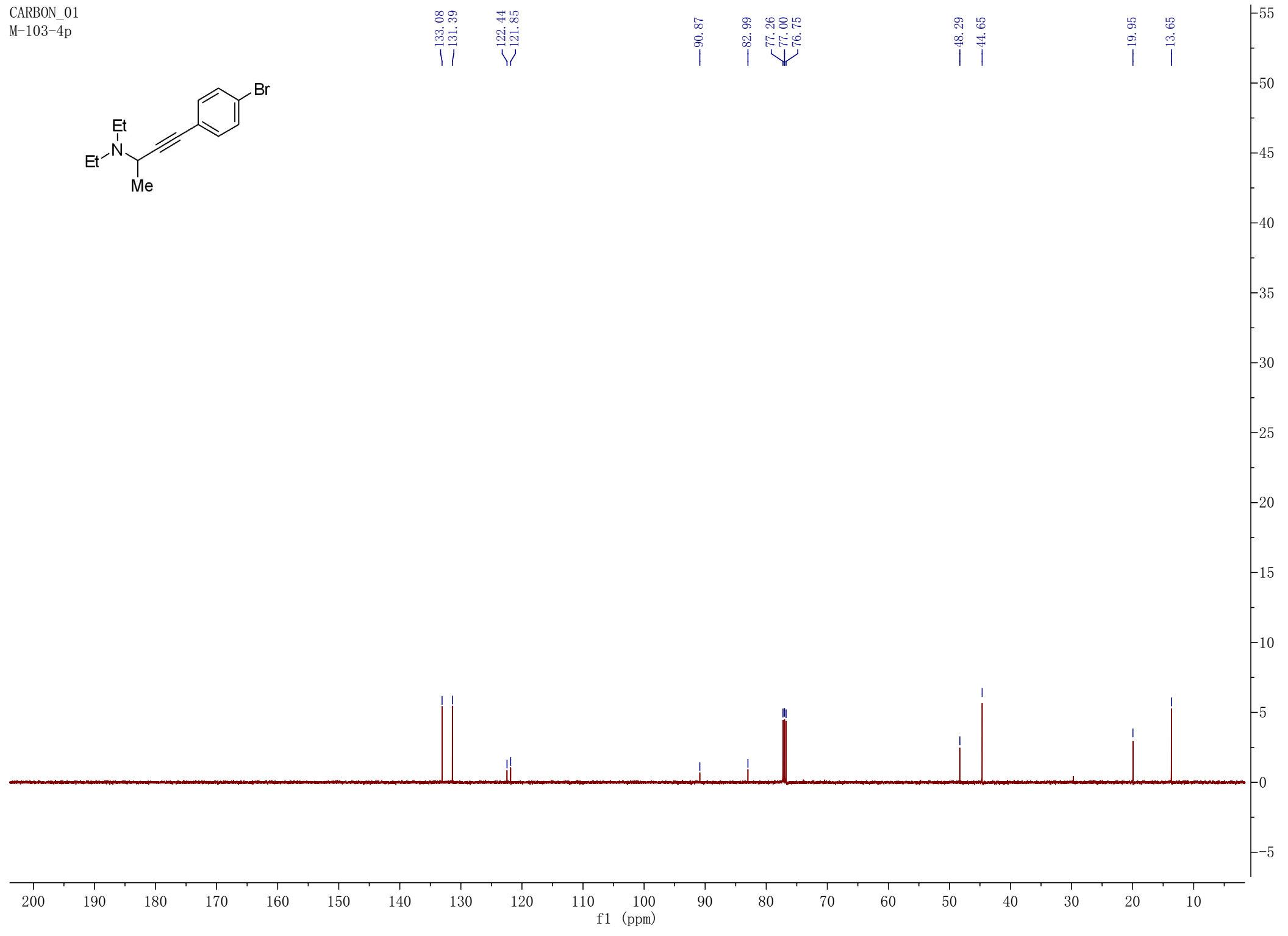
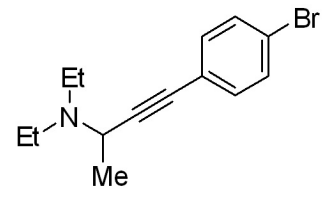
3.90
3.88
3.87
3.85

2.75
2.74
2.72
2.71
2.70
2.68
2.52
2.51
2.49
2.48
2.47
2.45

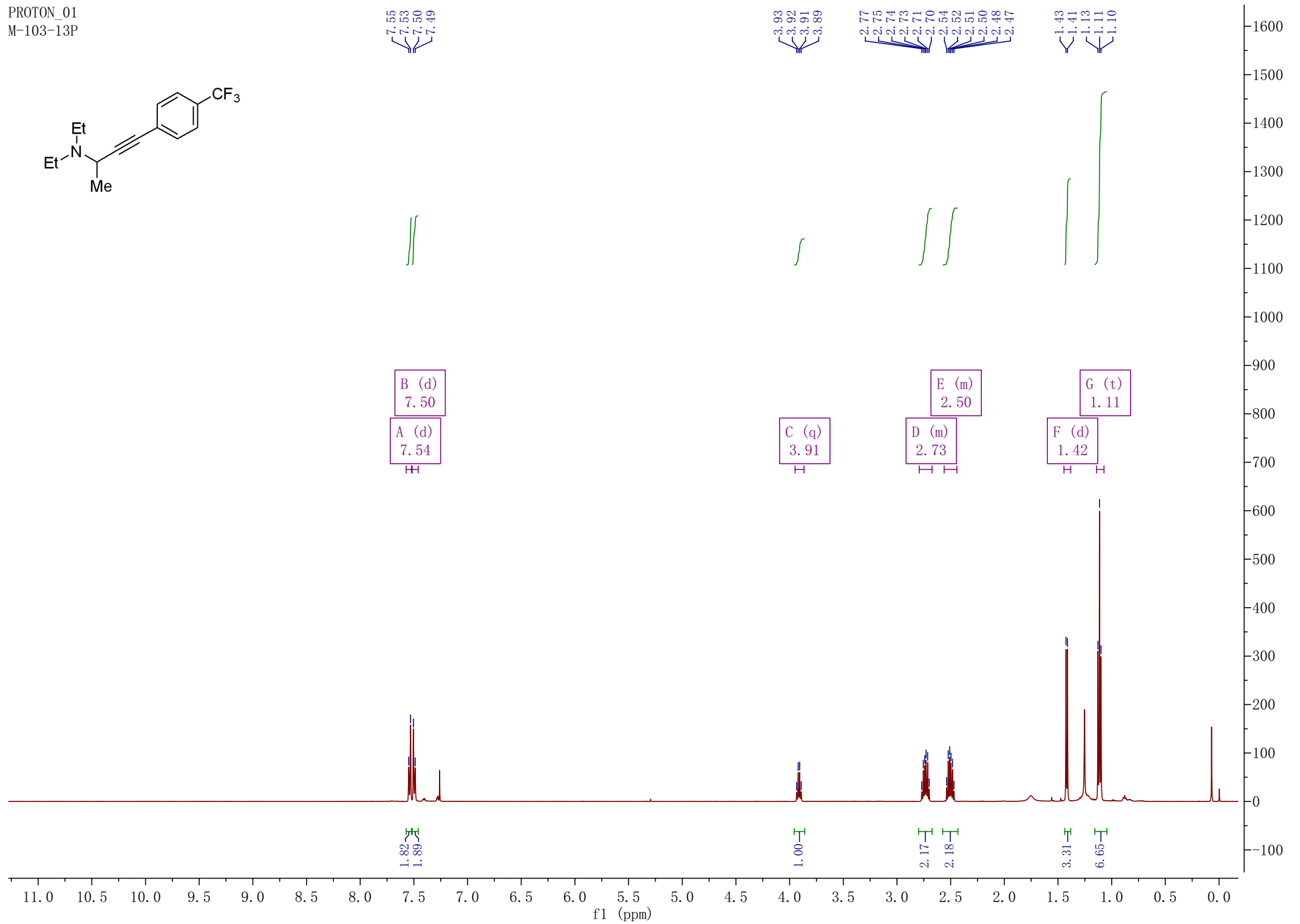
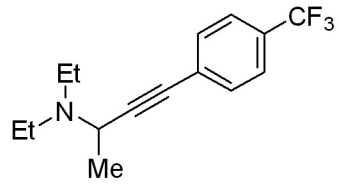
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1.39
1.12
1.10
1.09



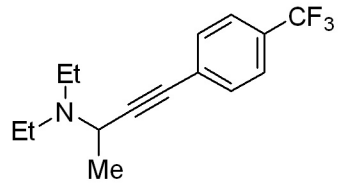
CARBON_01
M-103-4p



PROTON_01
M-103-13P



CARBON_01
M-103-13P



131.86
131.63
129.66
129.40
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127.76
127.34
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125.12
125.09
125.06

92.43

82.88

77.25

77.00

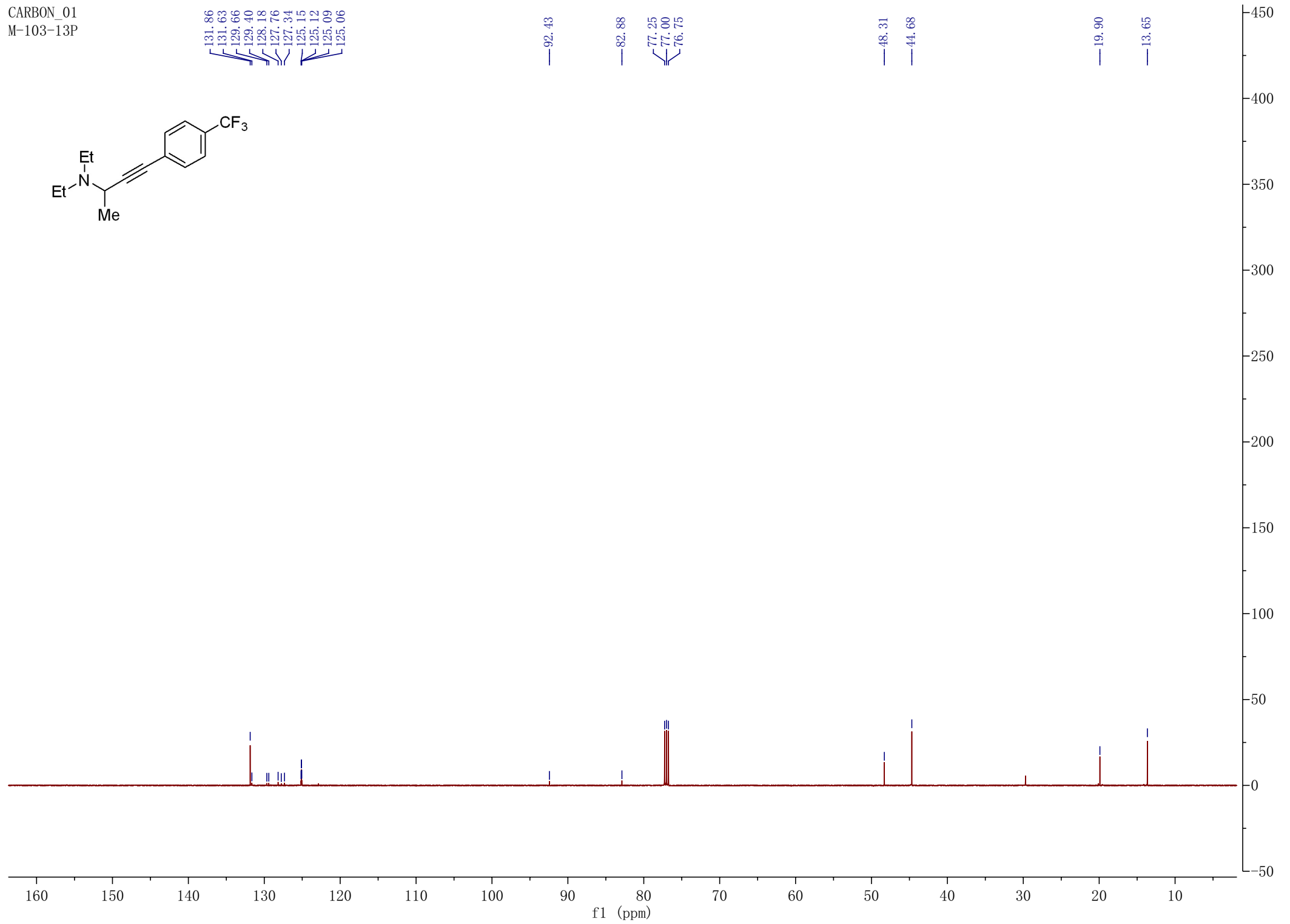
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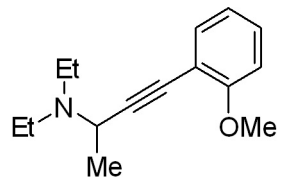
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19.90

13.65



PROTON_01
M-103-8p

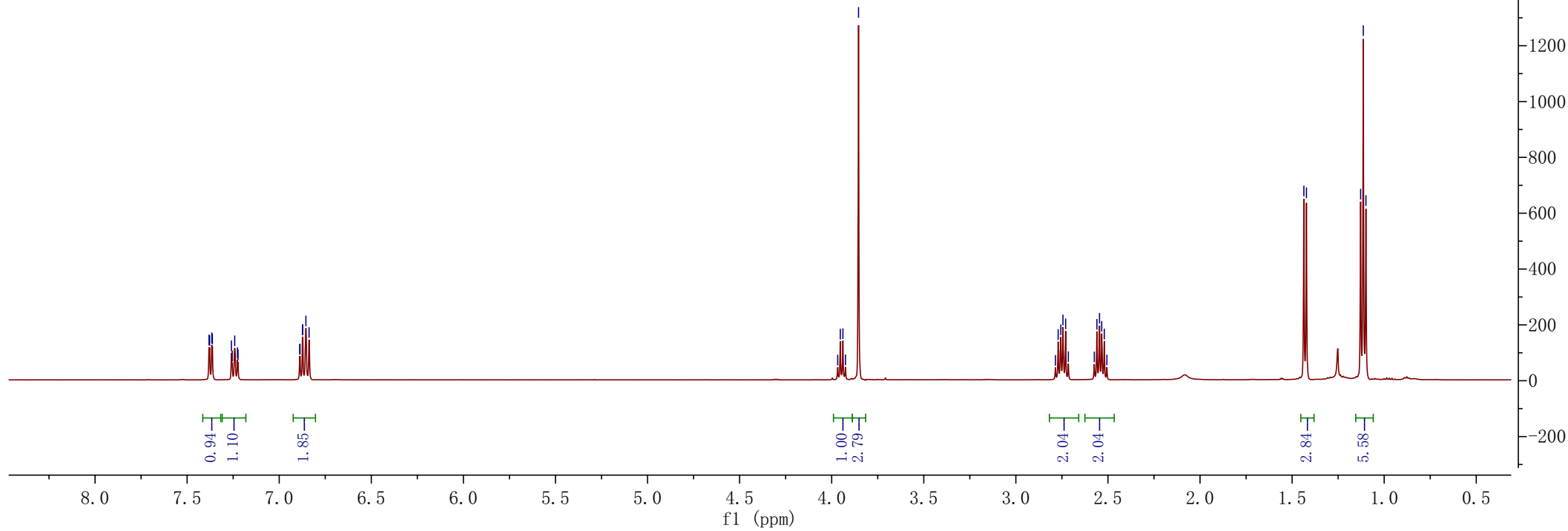
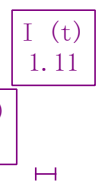
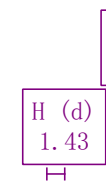
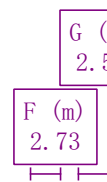
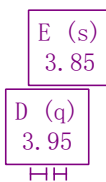
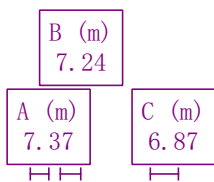


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7.38
7.37
7.36
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7.24
7.24
7.23
7.22
6.89
6.89
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6.87
6.86
6.84

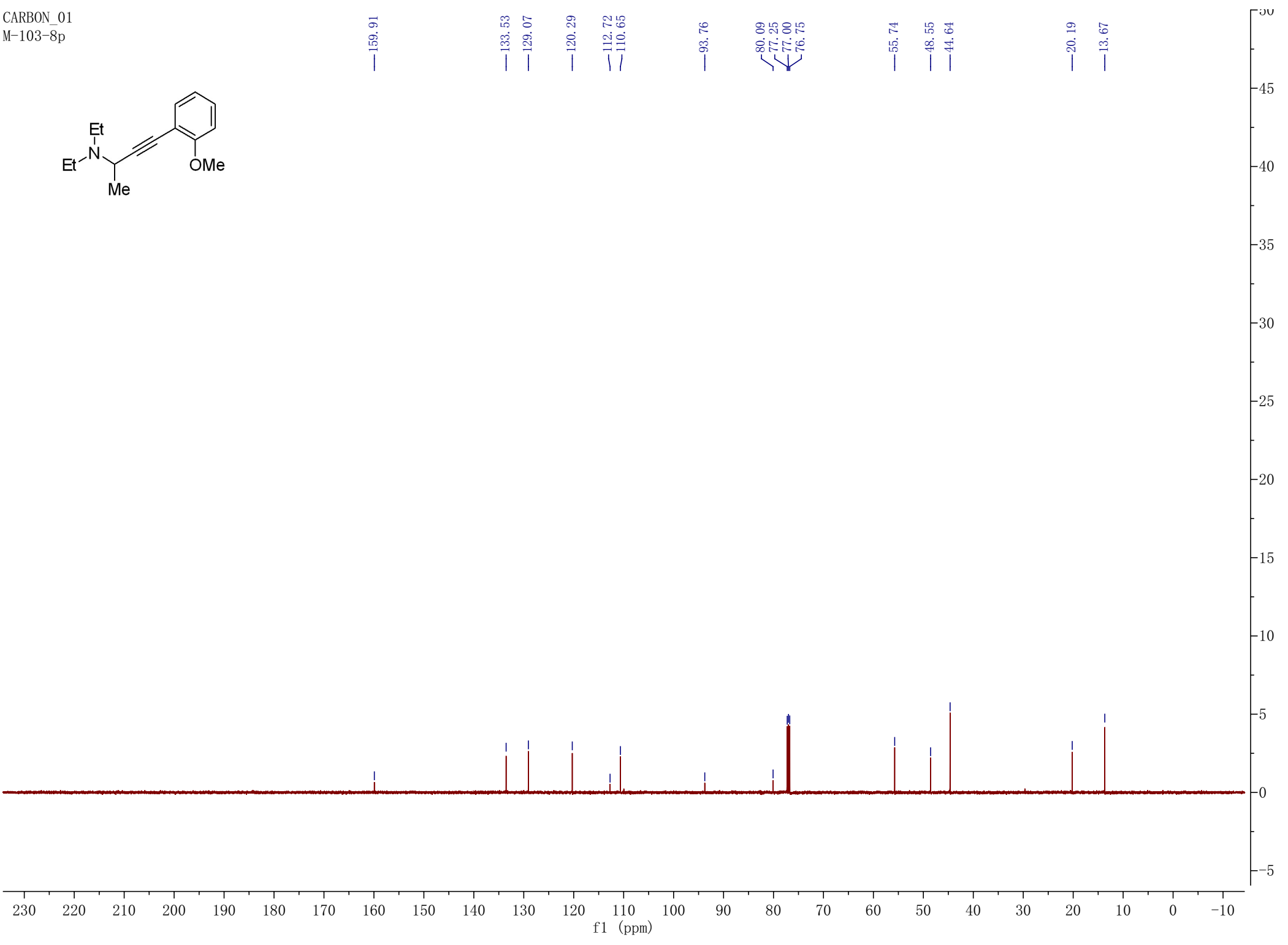
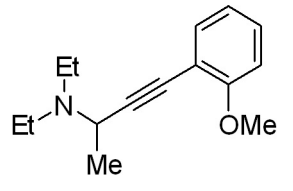
3.97
3.95
3.94
3.93
3.85

2.78
2.77
2.76
2.74
2.73
2.72
2.57
2.56
2.55
2.53
2.52
2.51

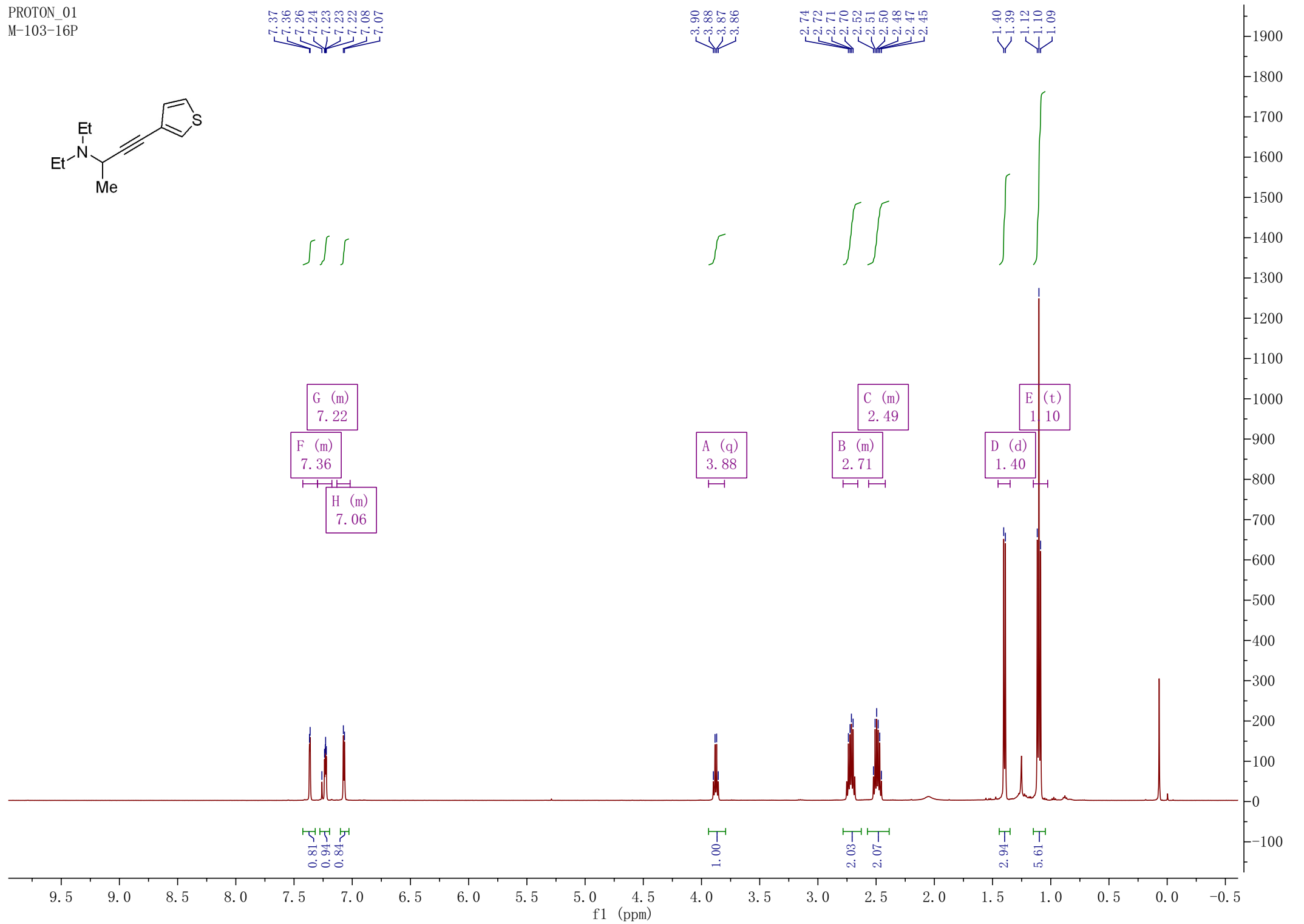
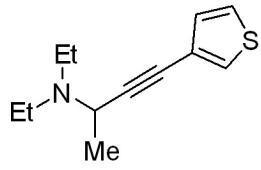
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1.42
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1.11
1.10



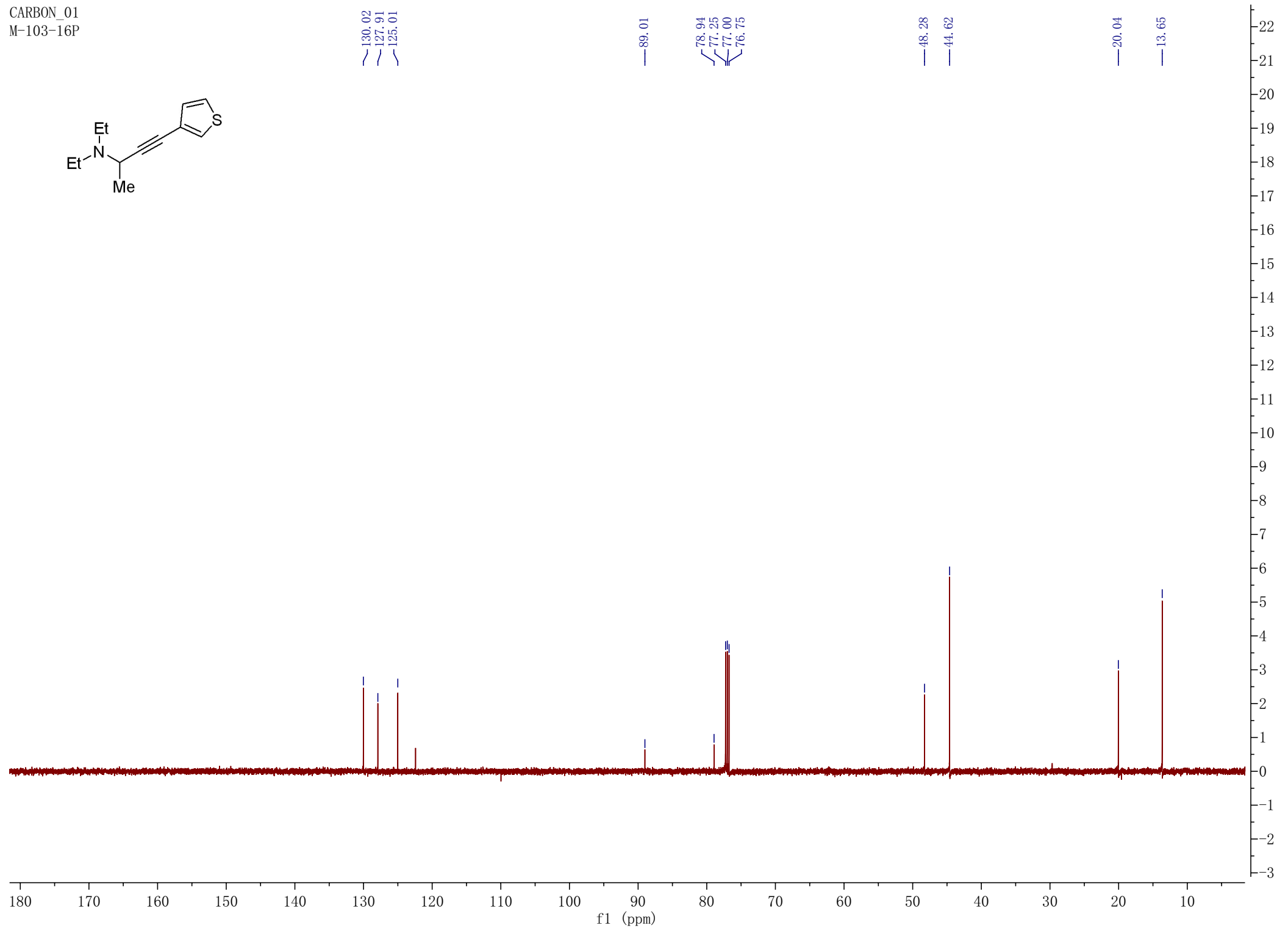
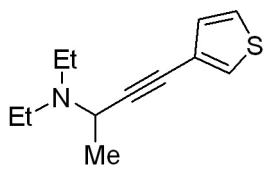
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M-103-8p



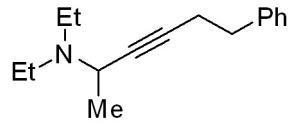
PROTON_01
M-103-16P



CARBON_01
M-103-16P



PROTON_01
m-103-19P



7.30
7.28
7.27
7.26
7.23
7.21
7.20
7.19

3.65
3.64
3.64
3.63
3.63
3.62
3.61
2.82
2.81
2.79
2.62
2.60
2.59
2.58
2.56
2.55
2.50
2.50
2.35
2.33
2.32
2.31

1.27
1.26
1.05
1.03
1.02



B (m)
7.21
A (m)
7.27

C (q)
3.63

E (m)
2.58
D (m)
2.81
G (m)
2.33
F (m)
2.49

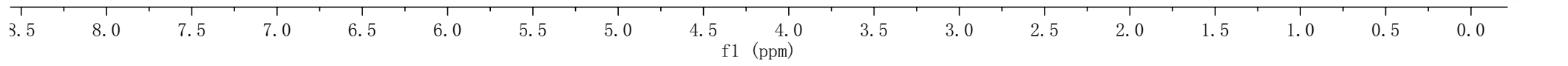
I (t)
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H (d)
1.27

1.86
2.54

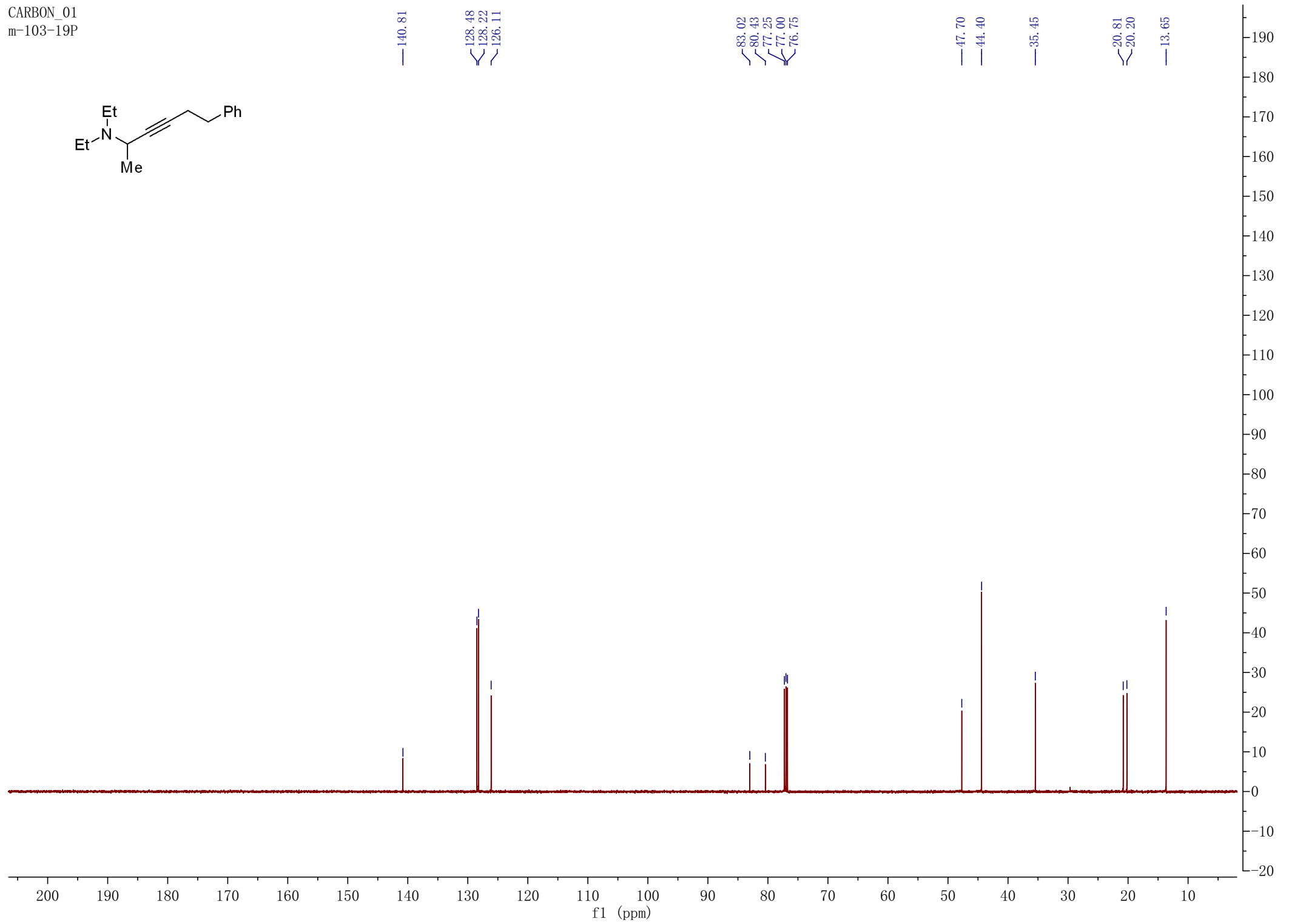
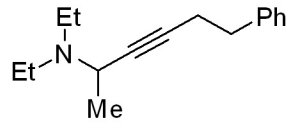
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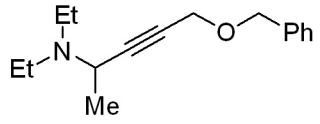
3.13
5.48



CARBON_01
m-103-19P



PROTON_01
m-103-18P



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7.34
7.33
7.33
7.32
7.31
7.30
7.30
7.29
7.29
7.29
7.28
7.26

4.60

4.20
4.20

3.76
3.75
3.75
3.74
3.74
3.73

2.70
2.68
2.67
2.66
2.64

2.46
2.45
2.44
2.42
2.41
2.40

1.35
1.34

1.09
1.08
1.07

A (m)
7.31

B (s)
4.60

C (s)
4.20

D (q)
3.75

E (m)
2.68

F (m)
2.43

G (d)
1.34

H (t)
1.08

4.67

2.00

1.99

1.06

2.10

2.07

3.06

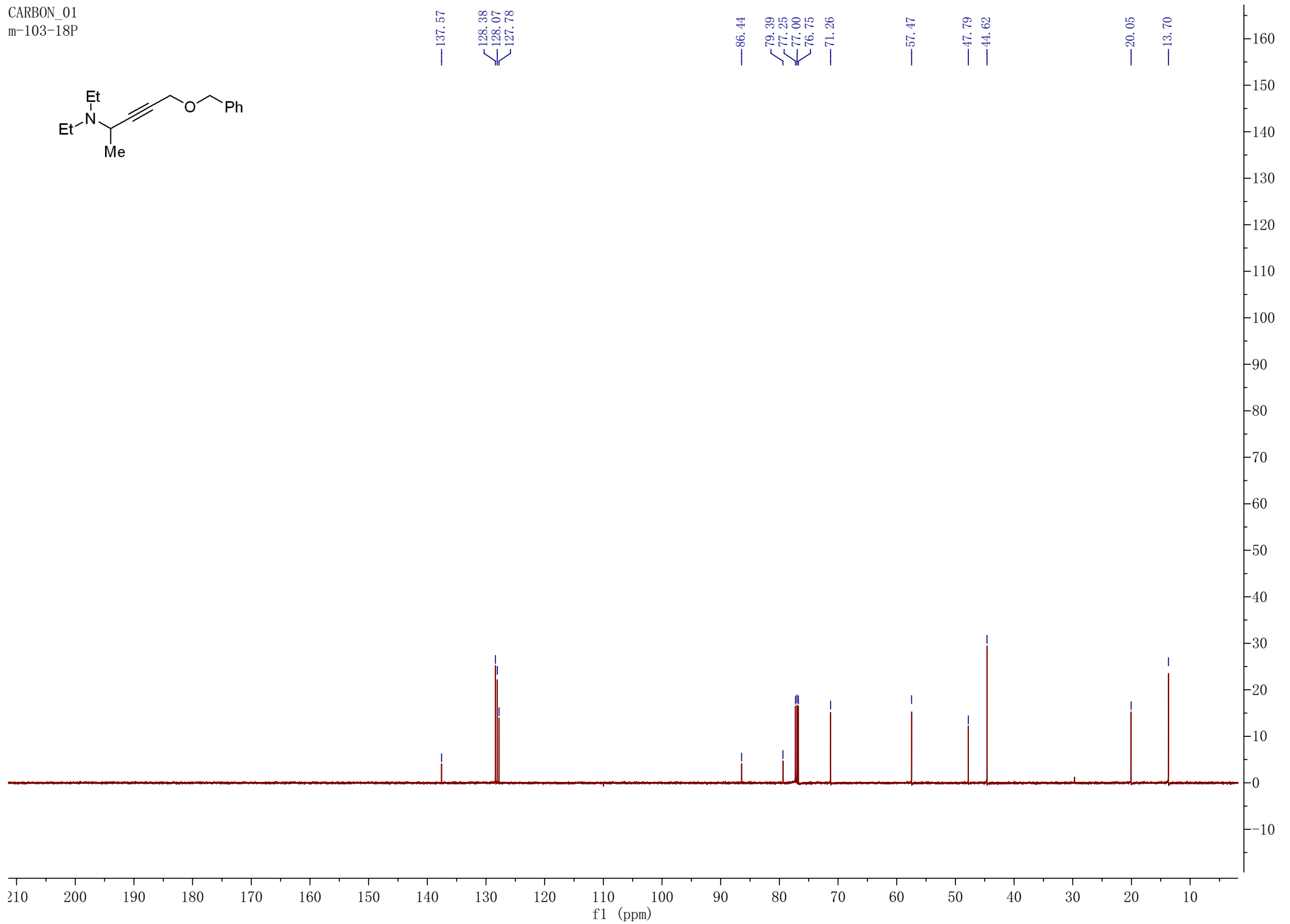
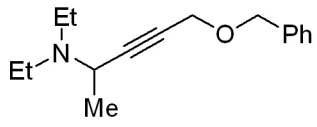
5.90

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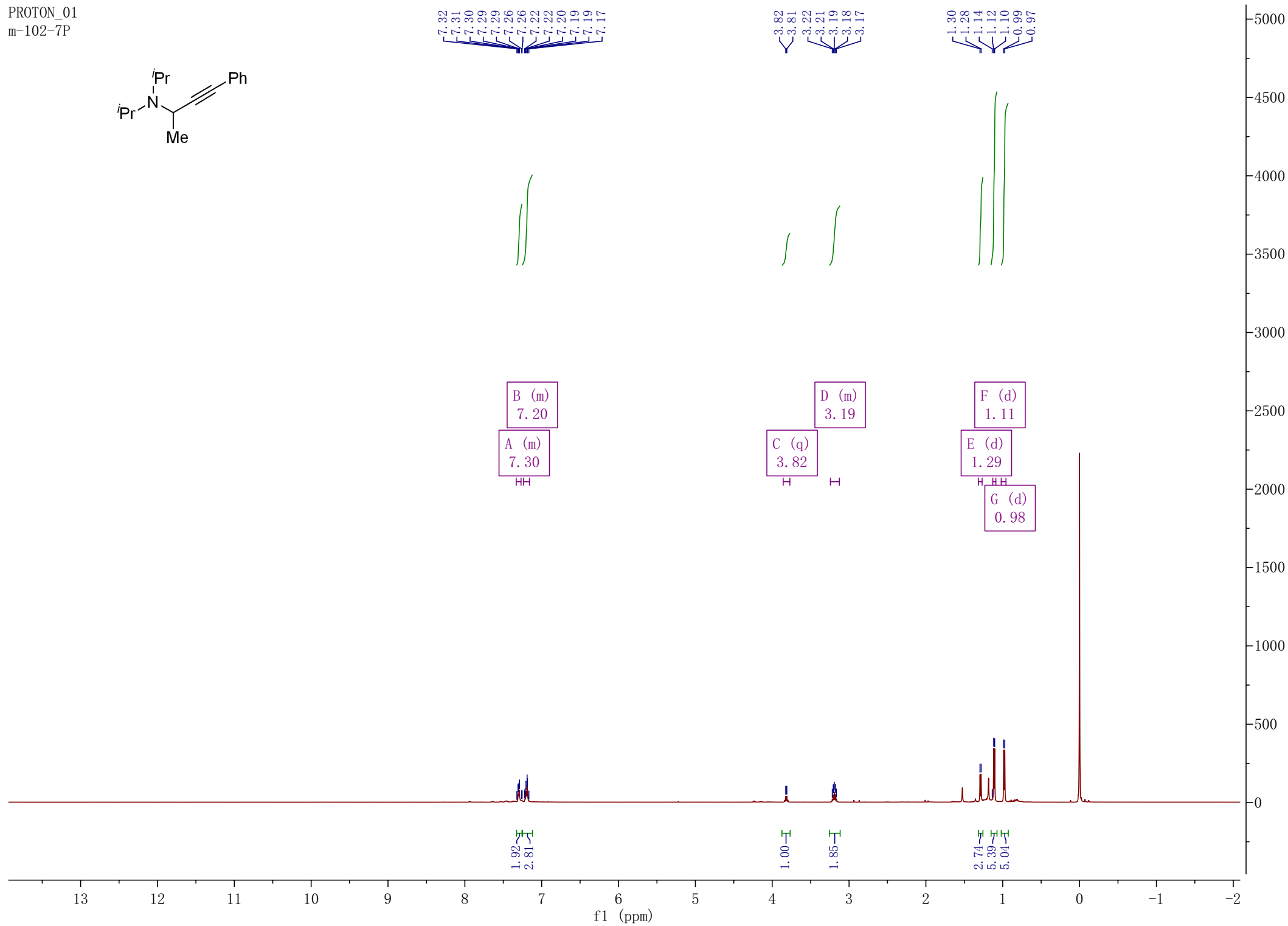
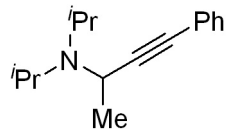
f1 (ppm)

6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500

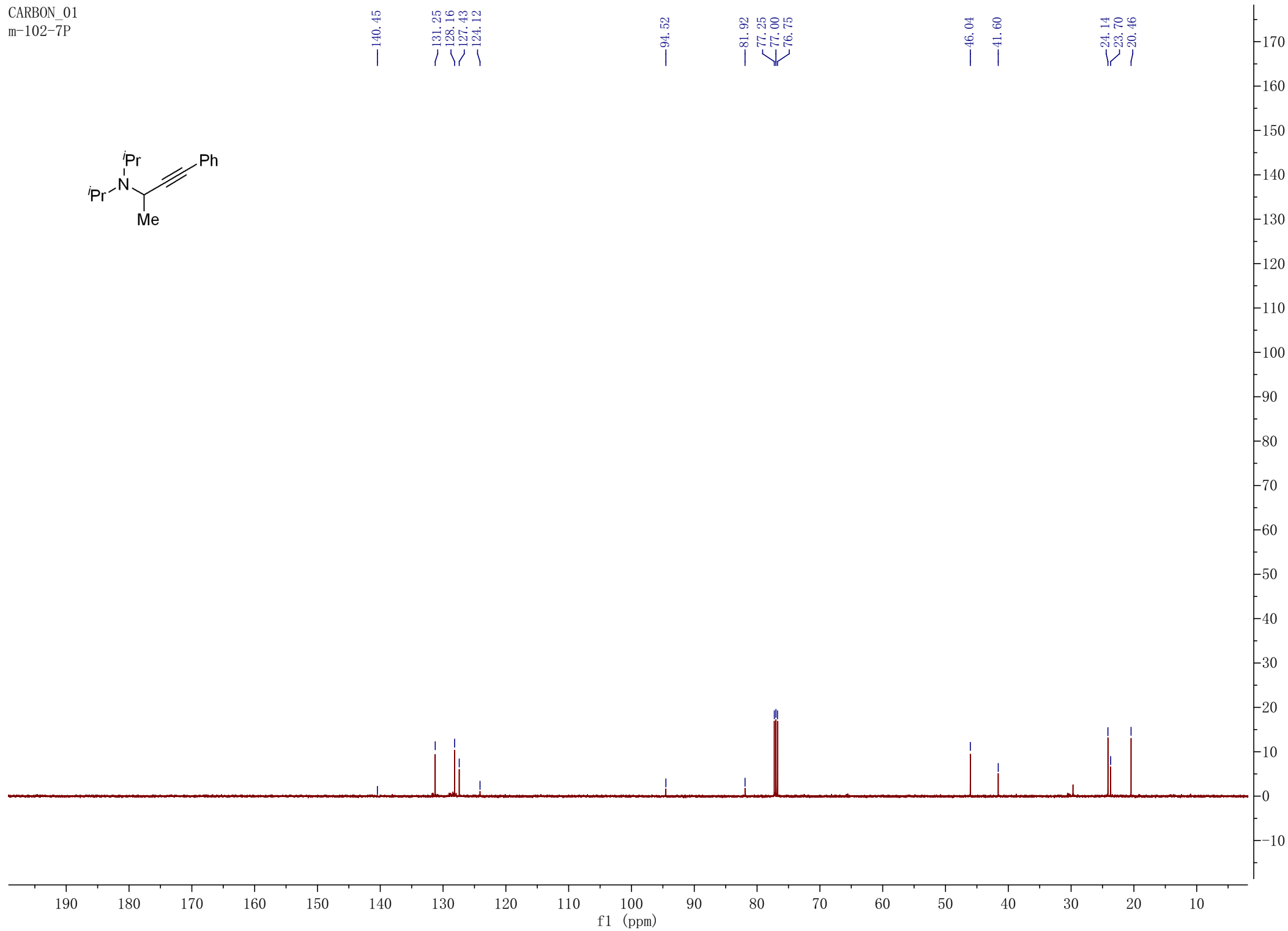
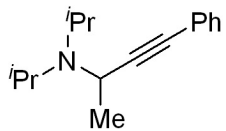
CARBON_01
m-103-18P



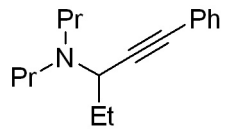
PROTON_01
m-102-7P



CARBON_01
m-102-7P



PROTON_01
m-102-8P



7.43
7.43
7.42
7.29
7.28
7.28

3.54
3.52
3.51
2.53
2.52
2.51
2.50
2.49
2.45
2.44
2.43
2.42
2.41
2.40
2.39
1.73
1.71
1.70
1.68
1.67
1.47
1.46
1.04
1.03
0.92
0.90
0.89



A (m)
7.42

B (m)
7.28

C (t)
3.52

D (m)
2.51

E (m)
2.42

F (m)
1.69

G (m)
1.49

H (t)
1.04

I (t)
0.90

1.73
2.67

1.00

1.97
1.96

2.56
3.99

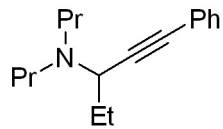
2.82
5.62

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

f1 (ppm)

4500
4000
3500
3000
2500
2000
1500
1000
500
0

CARBON_01
m-102-8P



131.67
128.15
127.60
123.73

89.20

84.35

77.25

77.00

76.75

56.00

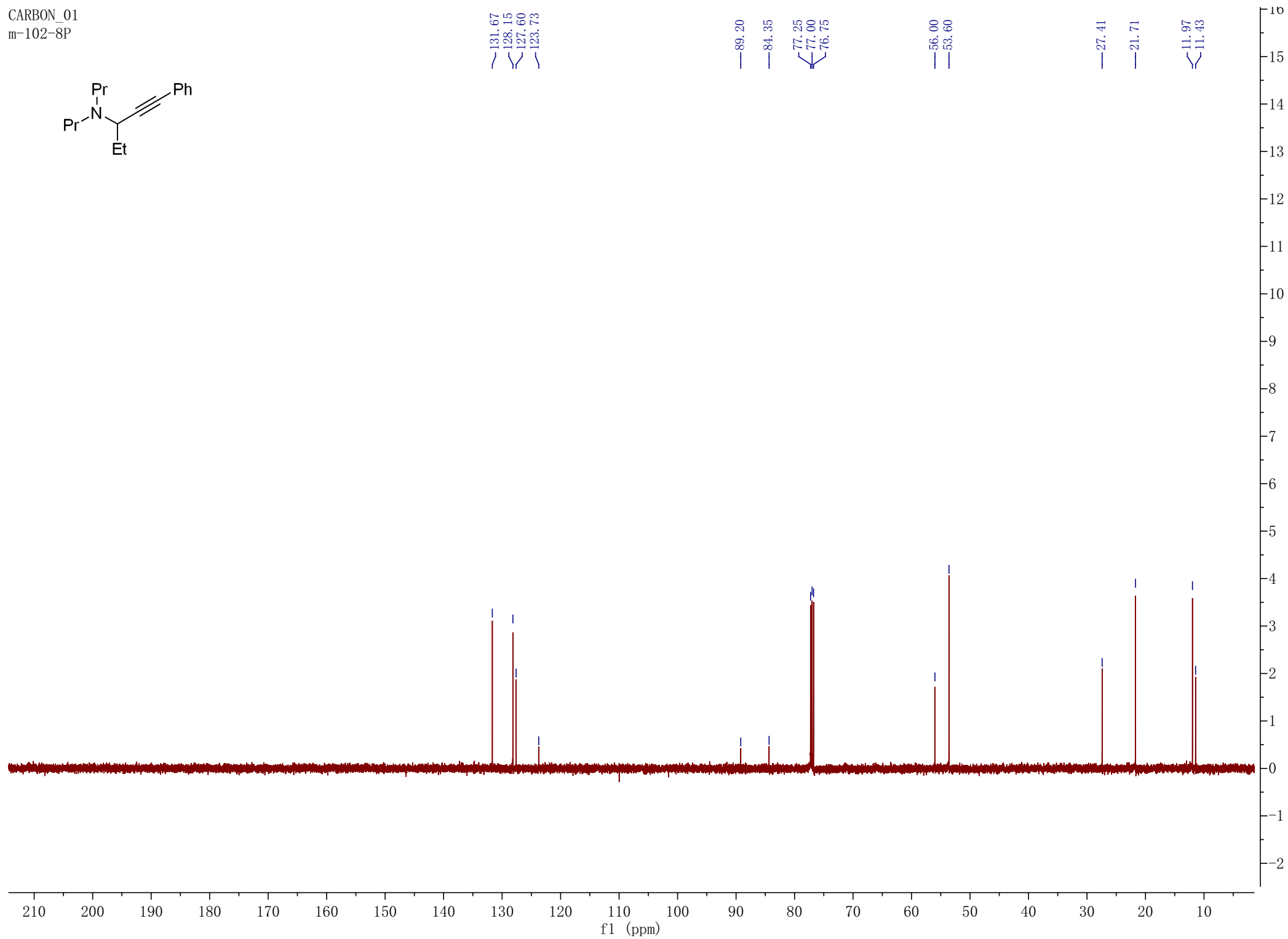
53.60

27.41

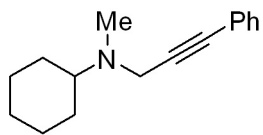
21.71

11.97

11.43



PROTON_01
m-102-6P



7.43
7.42
7.42
7.41
7.31
7.30
7.29
7.28
7.26

3.63

2.47
2.46
2.45
2.45
2.44
2.42
1.98
1.95
1.86
1.80
1.78
1.63
1.61
1.33
1.31
1.29
1.25
1.24
1.22
1.20
1.17
1.15
1.14
1.12
1.12
1.10



B (m)
7.29

A (m)
7.42

HH

C (s)
3.63

H

D (m)
2.45

HH

E (m)
1.95

HH

F (m)
1.78

HH

H (m)
1.21

HH

G (m)
1.62

HH

1.85

2.91

2.00

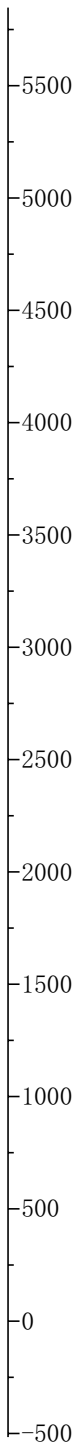
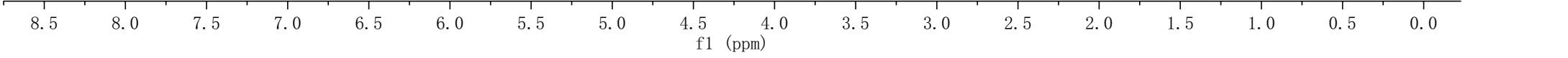
4.06

2.15

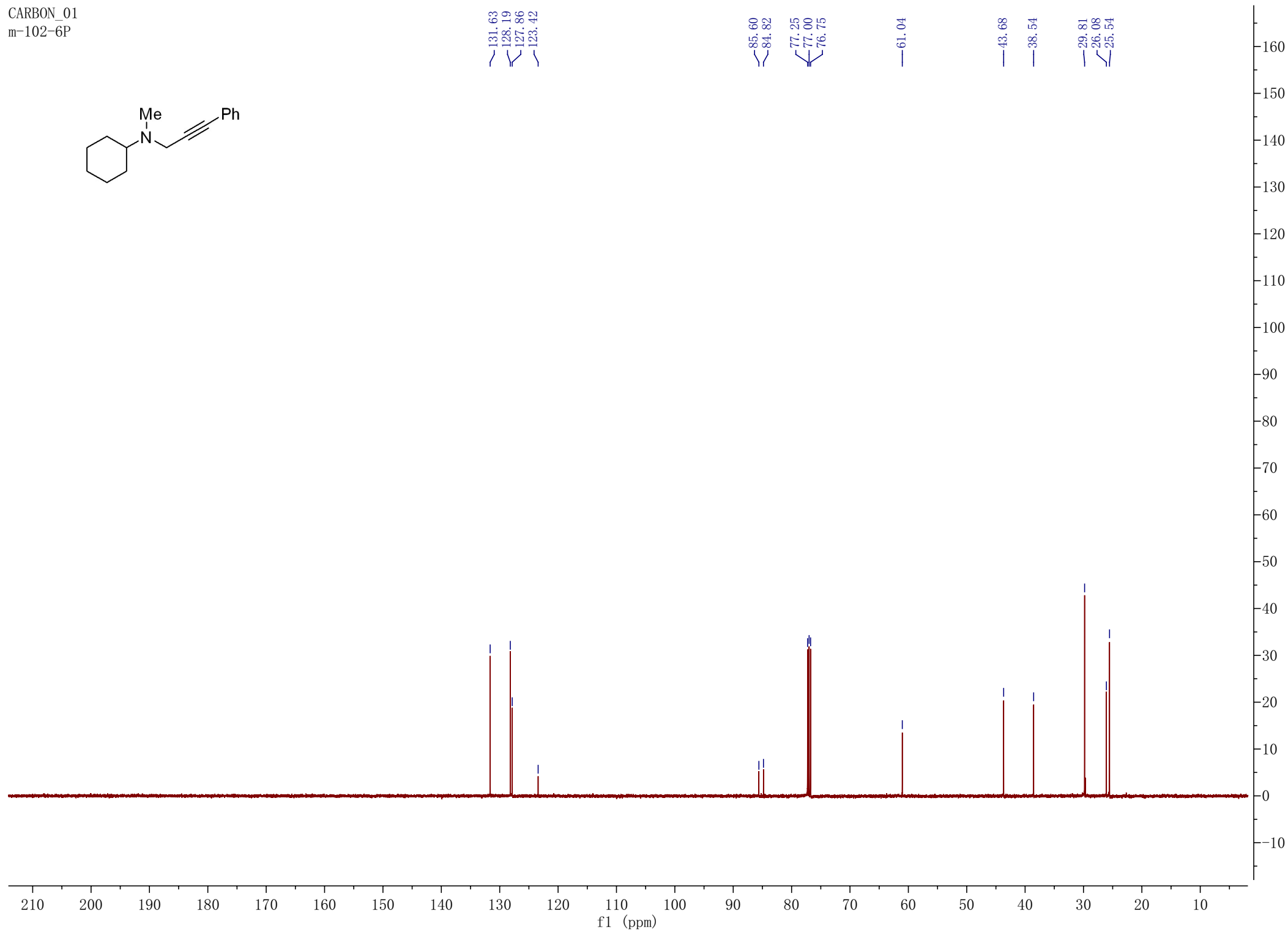
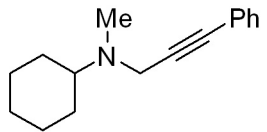
2.18

1.23

6.25

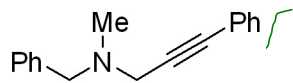


CARBON_01
m-102-6P



PROTON_01
m-102-4P

7.49
7.48
7.47
7.47
7.39
7.37
7.36
7.34
7.33
7.32
7.32
7.29
7.28
7.26



A (m)
7.37

B (s)
3.65

C (s)
3.53

D (s)
2.42

9.10

2.00
2.07

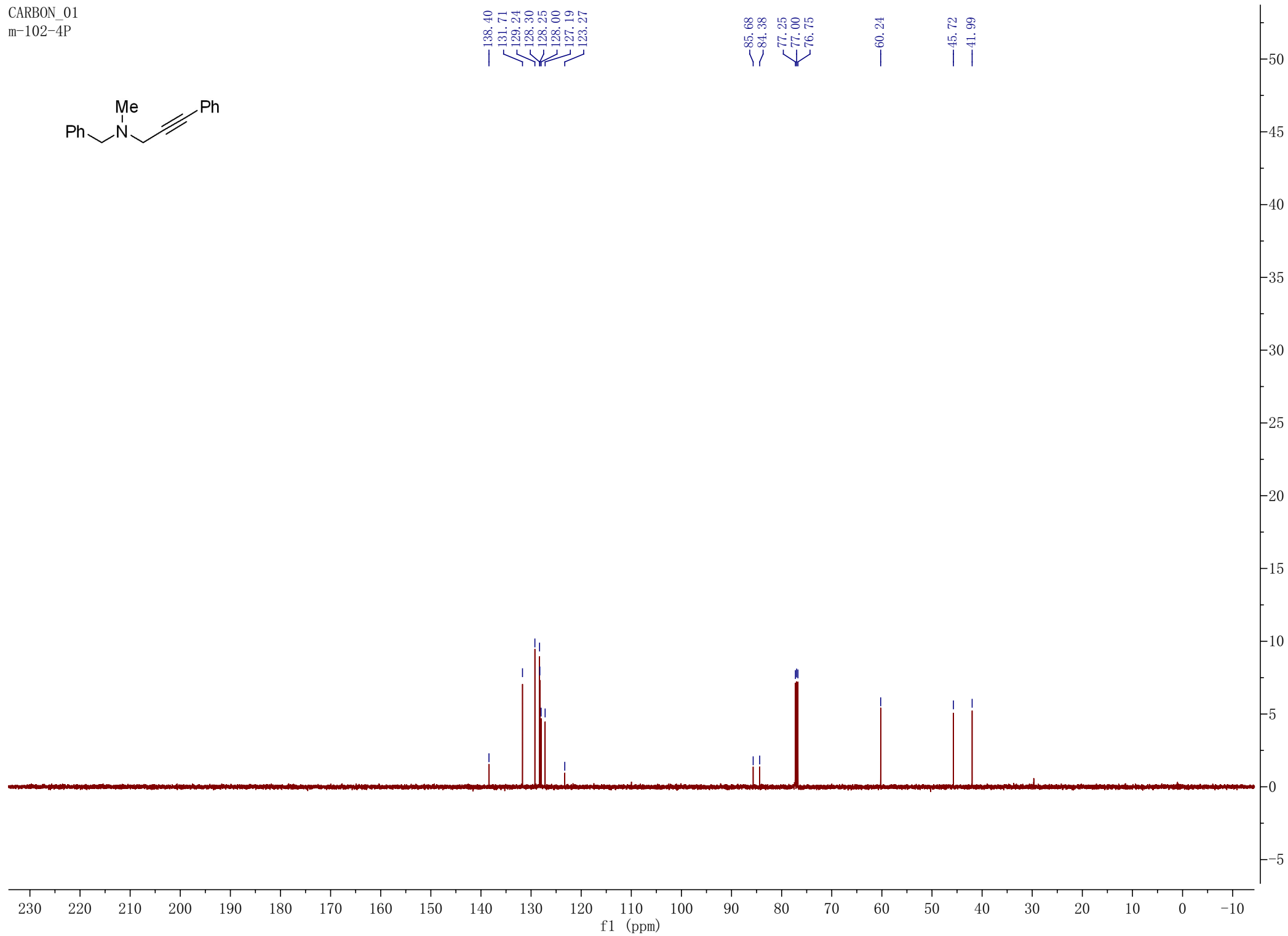
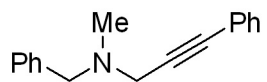
2.98

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

f1 (ppm)

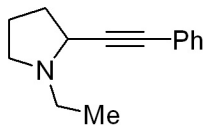
4500
4000
3500
3000
2500
2000
1500
1000
500
0

CARBON_01
m-102-4P

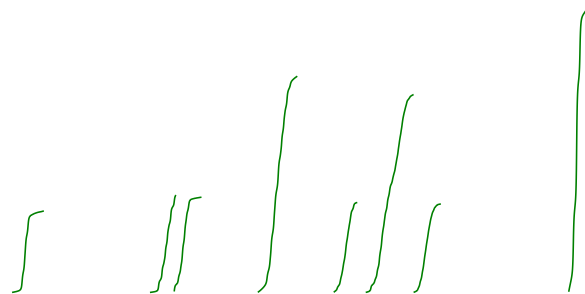


PROTON_01
M-102-2P

7.43
7.42
7.41
7.41
7.29
7.28
7.27
7.26



3.60
3.59
3.58
3.01
2.99
2.98
2.97
2.95
2.90
2.89
2.51
2.50
2.48
2.19
2.04
2.03
2.02
2.01
1.96
1.94
1.85
1.84
1.18
1.16



B (m)
7.28
A (m)
7.42

E (m)
2.90
D (m)
2.97
G (m)
2.19
F (m)
2.48
H (m)
1.99
I (m)
1.83
J (t)
1.18

1.84
3.04

1.00
1.19
1.17
2.65
1.10
2.43
1.09
3.51

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

f1 (ppm)

2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200

CARBON_01
M-102-2P

