Supporting information for

Aldehyde-Alkyne-Amine (A^3) Coupling Catalyzed by a Highly Efficient Dicopper Complex

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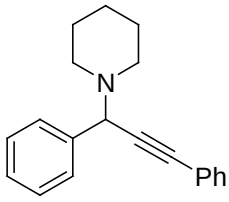
I. General methods

Unless otherwise noted, reagents and solvents were commercially available and used as received without any further purification. Toluene was dried over calcium hydride prior to use. All the A^3-coupling reactions were performed in flame-dried glassware under nitrogen atmosphere. ^1H and ^{13}C NMR spectra were determined in deuterated solvents on a Bruker av400 NMR spectrometer. Chemical shifts were reported in delta (δ) units, parts per million (ppm) downfield from TMS. High resolution mass spectra (HRMS) were recorded on a Bruker Apex ultra 7.0T FT-MS.

II. Experimental procedure and characterization data

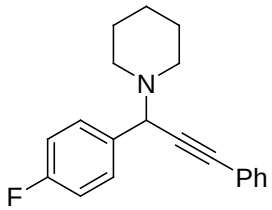
General procedure for A^3-coupling: To a solution of $Cu^I_2(pip)_2$ (2.0 mg, 0.4% mmol) in toluene (3 mL) was added alkyne (1.2 mmol), aldehyde (1.0 mmol) and amine (1.0 mmol) under nitrogen atmosphere. The reaction mixture was heated at 110 ^oC for 2 hrs, cooled, and then subjected to column chromatography on silica gel (300-400 mesh) eluting with petroleum ether-ethyl acetate to which give the desired propargylamine.

1-(1,3-diphenylprop-2-ynyl)piperidine (Table 2, entry 1)



Pale yellow oil, 270 mg, 98%; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.69-7.63 (m, 2H), 7.57-7.51 (m, 2H), 7.42-7.29 (m, 6H), 4.83 (s, 1H), 2.59 (t, J = 5.14 Hz, 4H), 1.69-1.53 (m, 4H), 1.51-1.39 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 138.6, 131.9, 128.6, 128.3, 128.1, 127.5, 123.4, 87.9, 86.1, 62.4, 50.7, 26.2, 24.5; HRMS (ESI): *m/z* calcd for $C_{20}H_{21}N$ $[M+H]^+$ 276.1747, found 276.1748.

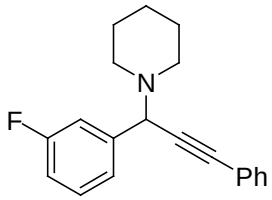
1-(1-(4-fluorophenyl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 2)



Pale yellow oil, 284 mg, 97%; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.66-7.59

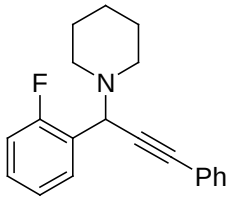
(m, 2H), 7.56-7.51 (m, 2H), 7.38-7.31 (m, 3H), 7.09-7.00 (m, 2H), 4.79 (s, 1H), 2.65-2.47 (m, 4H), 1.72-1.51 (m, 4H), 1.50-1.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.2 (d, J_{CF} = 244.1 Hz), 134.4 (d, J_{CF} = 3.0 Hz), 131.8, 130.0 (d, J_{CF} = 8.0 Hz), 128.3, 128.2, 123.2, 114.8 (d, J_{CF} = 21.2 Hz), 88.1, 85.7, 61.7, 50.6, 29.7, 26.2, 24.4; HRMS (ESI): *m/z* calcd for $C_{20}H_{20}FN$ $[M+H]^+$ 294.1653, found 294.1654.

1-(1-(3-fluorophenyl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 3)



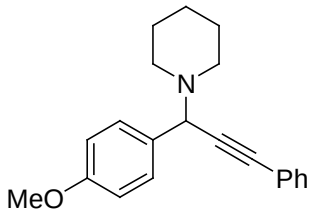
Pale yellow oil, 288 mg, 98%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.58-7.51 (m, 2H), 7.49-7.30 (m, 6H), 7.00 (td, *J* = 2.60, 8.28 Hz, 1H), 4.81 (s, 1H), 2.65-2.48 (m, 4H), 1.71-1.54 (m, 4H), 1.53-1.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.8 (d, J_{CF} = 243.6 Hz), 141.5 (d, J_{CF} = 7.0 Hz), 131.8, 129.4 (d, J_{CF} = 8.0 Hz), 128.3, 128.2, 124.0 (d, J_{CF} = 2.7 Hz), 123.1, 115.3 (d, J_{CF} = 22.3 Hz), 114.3 (d, J_{CF} = 21.1 Hz), 88.2, 85.3, 61.9 (d, J_{CF} = 2.0 Hz), 50.7, 26.2, 24.4; HRMS (ESI): *m/z* calcd for $C_{20}H_{20}FN$ $[M+H]^+$ 294.1653, found 294.1648.

1-(1-(2-fluorophenyl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 4)



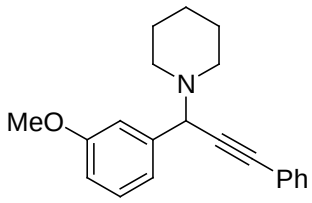
Pale yellow oil, 287 mg, 98%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.69 (td, *J* = 1.80, 7.52 Hz, 1H), 7.53-7.48 (m, 2H), 7.37-7.26 (m, 4H), 7.17 (td, *J* = 1.20, 7.56 Hz, 1H), 7.08 (m, 1H), 5.10 (s, 1H), 2.62 (t, *J* = 5.16 Hz, 4H), 1.67-1.54 (m, 4H), 1.47-1.37 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.8 (d, J_{CF} = 246.9 Hz), 131.8, 130.8 (d, J_{CF} = 3.6 Hz), 129.4 (d, J_{CF} = 8.3 Hz), 128.3, 128.2, 125.3 (d, J_{CF} = 13.3 Hz), 123.5 (d, J_{CF} = 3.6 Hz), 123.2, 115.5 (d, J_{CF} = 22.1 Hz), 86.9, 86.0, 55.7 (d, J_{CF} = 2.4 Hz), 50.8, 26.1, 24.4; HRMS (ESI): *m/z* calcd for $C_{20}H_{20}FN$ $[M+H]^+$ 294.1653, found 294.1648.

1-(1-(4-methoxyphenyl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 5)



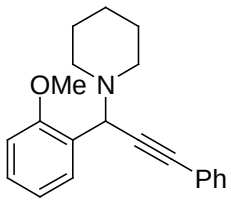
Yellow solid, 293 mg, 96%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.59-7.49 (m, 4H), 7.38-7.31 (m, 3H), 6.94-6.88 (m, 2H), 4.76 (s, 1H), 3.83 (s, 3H), 2.63-2.48 (m, 4H), 1.66-1.51 (m, 4H), 1.50-1.38 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.0, 131.8, 130.8, 129.7, 128.3, 128.0, 123.4, 113.4, 87.7, 86.5, 61.8, 55.3, 50.7, 26.3, 24.5; HRMS (ESI): *m/z* calcd for $C_{21}H_{23}NO$ $[M+H]^+$ 306.1852, found 306.1847.

1-(1-(3-methoxyphenyl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 6)



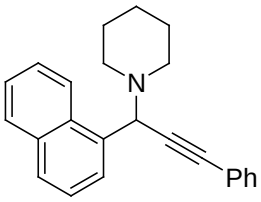
Yellow oil, 296 mg, 97%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.57-7.49 (m, 2H), 7.37-7.28 (m, 4H), 7.27-7.23 (m, 2H), 6.86 (ddd, *J* = 1.56, 2.32, 7.80 Hz, 1H), 4.79 (s, 1H), 3.85 (s, 3H), 2.66-2.48 (m, 4H), 1.70-1.53 (m, 4H), 1.50-1.41 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.6, 140.4, 131.9, 129.0, 128.3, 128.1, 123.4, 120.9, 114.2, 112.8, 87.8, 86.1, 62.4, 55.3, 50.8, 26.3, 24.5; HRMS (ESI): *m/z* calcd for $C_{21}H_{23}NO$ $[M+H]^+$ 306.1852, found 306.1859.

1-(1-(2-methoxyphenyl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 7)



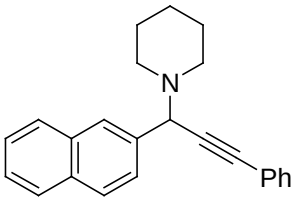
Yellow oil, 297 mg, 97%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.65 (dd, *J* = 1.76, 7.60 Hz, 1H), 7.51-7.45 (m, 2H), 7.34-7.28 (m, 4H), 6.99 (td, *J* = 1.04, 7.48 Hz, 1H), 6.93 (dd, *J* = 0.96, 8.24 Hz, 1H), 5.22 (s, 1H), 3.88 (s, 3H), 2.74-2.51 (m, 4 H), 1.67-1.52 (m, 4H), 1.47-1.37 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.4, 132.4, 129.9, 128.4, 127.3, 122.7, 120.8, 114.0, 86.8, 80.9, 56.1, 50.8, 44.9, 25.8, 25.3; HRMS (ESI): *m/z* calcd for $C_{21}H_{23}NO$ $[M+H]^+$ 306.1852, found 306.1849.

1-(1-(naphthalen-1-yl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 8)



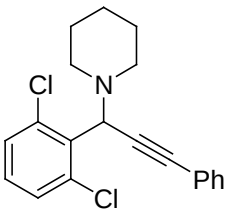
Yellow solid, 309 mg, 95%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.43 (d, J = 8.36 Hz, 1H), 7.95 (d, J = 7.04 Hz, 1H), 7.88 (d, J = 7.80 Hz, 1H), 7.83 (d, J = 8.20 Hz, 1H), 7.61-7.43 (m, 5H), 7.41-7.31 (m, 3H), 5.45 (s, 1H), 2.77-2.54 (m, 4H), 1.63-1.51 (m, 4H), 1.49-1.43 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 134.3, 134.1, 132.0, 131.9, 128.6, 128.5, 128.4, 128.1, 126.9, 125.8, 125.6, 125.1, 124.8, 123.5, 88.6, 86.0, 60.6, 50.8, 26.4, 24.7; HRMS (ESI): *m/z* calcd for $C_{24}H_{23}N$ $[M+H]^+$ 326.1903, found 326.1904.

1-(1-(naphthalen-2-yl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 9)



Pale yellow solid, 316 mg, 97%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.11 (s, 1H), 7.95-7.75 (m, 4H), 7.64-7.55 (m, 2H), 7.54-7.45 (m, 2H), 7.43-7.32 (m, 3H), 4.97 (s, 1H), 2.63 (t, J = 5.32 Hz, 4H), 1.79-1.54 (m, 4H), 1.54-1.39 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 136.4, 133.2, 133.1, 131.9, 128.4, 128.2, 128.2, 127.8, 127.7, 127.4, 126.8, 126.0, 125.9, 123.5, 88.2, 86.1, 62.6, 50.9, 26.3, 24.6; HRMS (ESI): *m/z* calcd for $C_{24}H_{23}N$ $[M+H]^+$ 326.1903, found 326.1900.

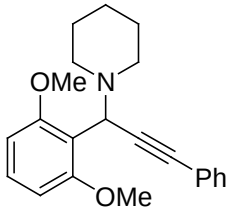
1-(1-(2,6-dichlorophenyl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 10)



Pale yellow oil, 302 mg, 88%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.50-7.44 (m, 2H), 7.34 (d, J = 4.52 Hz, 2H), 7.32-7.28 (m, 3H), 7.16 (dd, J = 7.64, 8.44 Hz, 1H), 5.26 (s, 1H), 2.93-2.74 (br, 2H), 2.52-2.38 (m, 2H), 1.66-1.53 (m, 4H), 1.50-1.39 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 136.5, 134.5, 131.8, 129.4, 128.9, 128.2, 128.1, 123.4, 86.3, 86.2, 58.4, 52.1, 26.2,

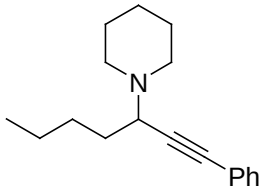
24.5; HRMS (ESI): *m/z* calcd for $C_{20}H_{19}Cl_2N$ $[M+H]^+$ 344.0967, found 344.0968.

1-(1-(2,6-dimethoxyphenyl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 11)



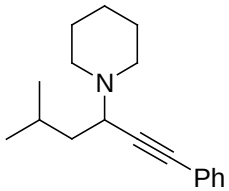
Yellow solid, 329 mg, 98%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.48-7.43 (m, 2H), 7.33-7.22 (m, 4H), 6.61 (d, *J* = 8.32 Hz, 2H), 5.37 (s, 1H), 3.96 (s, 6H), 2.79-2.62 (m, 4H), 2.52-2.38 (m, 2H), 1.69-1.52 (m, 4H), 1.46-1.36 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.2, 131.7, 129.2, 128.1, 127.4, 124.4, 114.9, 105.1, 89.0, 83.1, 56.2, 51.7, 51.4, 26.0, 24.3; HRMS (ESI): *m/z* calcd for $C_{22}H_{25}NO_2$ $[M+H]^+$ 336.1958, found 336.1959.

1-(1-phenylhept-1-yn-3-yl)piperidine (Table 2, entry 12)



Brown red oil, 253 mg, 99%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.48-7.43 (m, 2H), 7.34-7.28 (m, 3H), 3.49 (dd, *J* = 1.88, 9.08 Hz, 1H), 2.76-2.64 (m, 2H), 2.56-2.44 (m, 2H), 1.78-1.70 (m, 2H), 1.68-1.54 (m, 5H), 1.52-1.37 (m, 5H), 0.94 (t, *J* = 7.20 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 131.7, 128.2, 127.8, 123.6, 88.1, 85.7, 58.6, 50.7, 33.2, 29.2, 26.2, 24.6, 22.5, 14.1; HRMS (ESI): *m/z* calcd for $C_{18}H_{25}N$ $[M+H]^+$ 256.2060, found 256.2066.

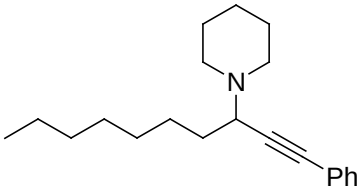
1-(5-methyl-1-phenylhex-1-yn-3-yl)piperidine (Table 2, entry 13)



Yellow oil, 252 mg, 99%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.47-7.42 (m, 2H), 7.34-7.28 (m, 3H), 3.59 (dd, *J* = 5.64, 9.76 Hz, 1H), 2.77-2.63 (m, 2H), 2.58-2.44 (m, 2H), 1.96-1.84 (m, 1H), 1.74-1.55 (m, 6H), 1.51-1.44 (m, 2H), 0.99 (d, *J* = 6.68 Hz, 3H), 0.96 (d, *J* = 6.60 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 131.7, 128.2, 127.7, 123.7, 88.2, 85.7, 56.7, 50.6, 42.3, 26.2, 25.4, 24.6, 23.2, 22.1; HRMS (ESI): *m/z* calcd for $C_{18}H_{25}N$ $[M+H]^+$ 256.2060, found

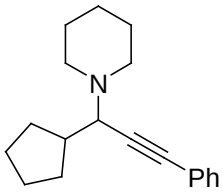
256.2058.

1-(1-phenyldec-1-yn-3-yl)piperidine (Table 2, entry 14)



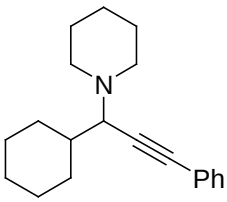
Yellow oil, 289 mg, 97%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.48-7.43 (m, 2H), 7.34-7.28 (m, 3H), 3.49 (dd, *J* = 5.64, 9.24 Hz, 1H), 2.76-2.65 (m, 2H), 2.58-2.44 (m, 2H), 1.79-1.53 (m, 8H), 1.51-1.43 (m, 3H), 1.36-1.26 (m, 7H), 0.90 (t, *J* = 7.04 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 131.7, 128.2, 127.7, 123.6, 88.2, 85.6, 58.7, 50.6, 33.5, 31.9, 29.4, 29.2, 26.9, 26.2, 24.6, 22.7, 14.1; HRMS (ESI): *m/z* calcd for $C_{21}H_{31}N$ $[M+H]^+$ 298.2529, found 298.2527.

1-(1-cyclopentyl-3-phenylprop-2-ynyl)piperidine (Table 2, entry 15)



Pale yellow oil, 243 mg, 91%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.48-7.43 (m, 2H), 7.35-7.28 (m, 3H), 3.24 (d, *J* = 9.32 Hz, 1H), 2.74-2.63 (m, 2H), 2.51-2.39 (m, 2H), 2.32-2.17 (m, 1H), 1.96-1.84 (m, 1H), 1.83-1.72 (m, 1H), 1.68-1.56 (m, 7H), 1.55-1.43 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 131.7, 128.2, 127.7, 123.8, 88.0, 85.6, 63.7, 50.8, 42.4, 30.8, 30.5, 26.2, 25.5, 25.3, 24.7; HRMS (ESI): *m/z* calcd for $C_{19}H_{25}N$ $[M+H]^+$ 268.2060, found 268.2060.

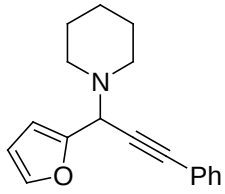
1-(1-cyclohexyl-3-phenylprop-2-ynyl)piperidine (Table 2, entry 16)



Pale yellow oil, 264 mg, 94%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.51-7.40 (m, 2H), 7.37-7.23 (m, 3H), 3.13 (d, *J* = 9.80 Hz, 1H), 2.71-2.57 (m, 2H), 2.49-2.32 (m, 2H), 2.20-1.99 (m, 2H), 1.86-1.72 (m, 2H), 1.72-1.50 (m, 6H), 1.50-1.39 (m, 2H), 1.36-1.12 (m, 3H),

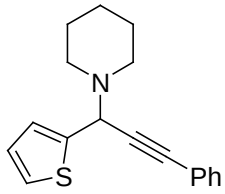
1.10-0.86 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 131.7, 128.2, 127.6, 123.9, 87.8, 86.2, 64.4, 50.8, 39.6, 31.4, 30.5, 26.9, 26.3(4), 26.3(1), 26.2, 24.8; HRMS (ESI): *m/z* calcd for $C_{20}H_{27}N$ $[M+H]^+$ 282.2216, found 282.2222.

1-(1-(furan-2-yl)-3-phenylprop-2-ynyl)piperidine (Table 2, entry 17)



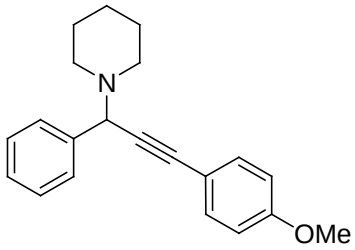
Brown oil, 236 mg, 89%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.54-7.49 (m, 2H),7.44 (dd, *J* = 0.84, 1.72 Hz, 1H), 7.37-7.31 (m,3H), 6.50 (dt, *J* = 0.84, 3.20 Hz, 1H), 6.36 (dd, *J* = 1.84, 3.20 Hz ,1H), 4.89 (s, 1H), 2.68-2.52 (m, 4H), 1.75-1.55 (m, 4H), 1.53-1.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 151.6, 142.6, 131.9, 128.3, 122.9, 110.0, 109.3, 86.4, 83.8, 56.6, 50.5, 26.0, 24.3; HRMS (ESI): *m/z* calcd for $C_{18}H_{19}NO$ $[M+Na]^+$ 288.1359, found 288.1360.

1-(3-phenyl-1-(thiophen-2-yl)prop-2-ynyl)piperidine (Table 2, entry 18)



Brown solid, 228 mg, 81%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.57-7.51 (m, 2H), 7.38-7.33 (m, 3H), 7.29 (ddd, *J* = 0.40, 1.20, 5.08 Hz, 1H), 7.24 (dt, *J* = 1.24, 3.48 Hz, 1H), 6.98 (dd, *J* = 3.52, 9.16 Hz, 1H), 5.01 (s, 1H), 2.73-2.53 (m, 4H), 1.71-1.55 (m, 4H), 1.52- 1.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 144.1, 131.9, 128.4, 128.3, 126.2, 125.9, 125.4, 123.1, 87.0, 85.4, 58.3, 50.7, 26.2, 24.5; HRMS (ESI): *m/z* calcd for $C_{18}H_{19}NS$ $[M+H]^+$ 282.1311, found 282.1316.

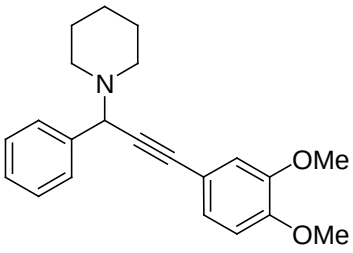
1-(3-(4-methoxyphenyl)-1-phenylprop-2-ynyl)piperidine (Table 3, entry 1)



Yellow solid, 296 mg, 97%; ^{1}H NMR ($CDCl_3$, 400 MHz)

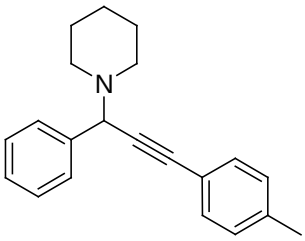
δ 7.70-7.59 (m, 2H), 7.52-7.42 (m, 2H), 7.40-7.23 (m, 3H), 6.92-6.82 (m, 2H), 4.79 (s, 1H), 3.84 (s, 3H), 2.57 (t, *J* = 5.36 Hz, 4H), 1.69-1.52 (m, 4H), 1.50-1.38 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.5, 138.9, 133.2, 128.6, 128.1, 127.4, 115.6, 113.9, 87.7, 84.6, 62.5, 55.3, 50.7, 26.3, 24.5; HRMS (ESI): *m/z* calcd for $C_{21}H_{23}NO$ $[M+H]^+$ 306.1852, found 306.1856.

1-(3-(3,4-dimethoxyphenyl)-1-phenylprop-2-ynyl)piperidine (Table 3, entry 2)



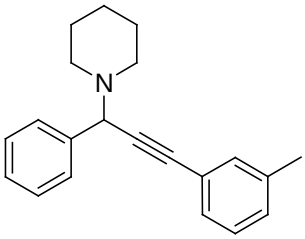
Pale yellow oil, 319 mg, 95%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.68-7.62 (m, 2H), 7.41-7.35 (m, 2H), 7.34-7.29 (m, 1H), 7.13 (dd, *J* = 1.80, 8.24 Hz, 1H), 7.02 (d, *J* = 1.80 Hz, 1H), 6.83 (d, *J* = 8.32 Hz, 1H), 4.79 (s, 1H), 3.92 (s, 3H), 3.91 (s, 3H), 2.58 (t, *J* = 5.04 Hz, 4H), 1.68-1.54 (m, 4H), 1.50-1.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.3, 148.6, 138.7, 128.6, 128.1, 127.5, 125.1, 115.6, 114.5, 111.0, 87.7, 84.5, 62.5, 55.9(4), 55.9(1), 50.8, 26.2, 24.4; HRMS (ESI): *m/z* calcd for $C_{22}H_{25}NO_2$ $[M+Na]^+$ 357.1778, found 358.1776.

1-(1-phenyl-3-*p*-tolylprop-2-ynyl)piperidine (Table 3, entry 3)



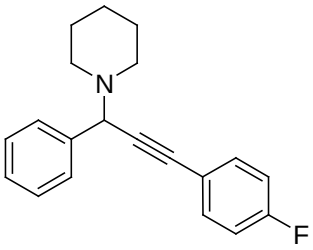
Pale yellow oil, 280 mg, 97%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.69-7.63 (m, 2H), 7.46-7.42 (m, 2H), 7.41-7.35 (m, 2H), 7.34-7.29 (m, 1H), 7.19-7.13 (m, 2H), 4.81 (s, 1H), 2.58 (t, *J* = 5.16 Hz, 4H), 2.38 (s, 3H), 1.68-1.54 (m, 4H), 1.50-1.42 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 138.8, 138.1, 131.8, 129.1, 128.6, 128.1, 127.5, 120.4, 88.0, 85.4, 62.5, 50.7, 26.3, 24.5, 21.5; HRMS (ESI): *m/z* calcd for $C_{21}H_{23}N$ $[M+H]^+$ 290.1903, found 290.1906.

1-(1-phenyl-3-*m*-tolylprop-2-ynyl)piperidine (Table 3, entry 4)



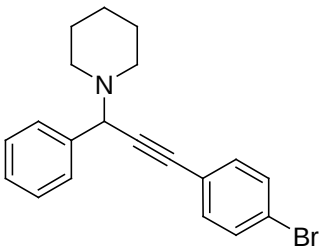
Pale yellow oil, 283 mg, 98%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.69-7.64 (m, 2H), 7.42-7.29 (m, 5H), 7.27-7.21 (m, 1H), 7.18-7.13 (m, 1H), 4.82 (s, 1H), 2.59 (t, J = 5.04 Hz, 4H), 2.37 (s, 3H), 1.71-1.53 (m, 4H), 1.50-1.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 138.8, 138.0, 132.4, 129.0, 128.9, 128.6, 128.2, 128.1, 127.5, 123.2, 88.1, 85.7, 62.4, 50.8, 26.3, 24.5, 21.3; HRMS (ESI): *m/z* calcd for $C_{21}H_{23}N$ $[M+H]^+$ 290.1903, found 290.1901.

1-(3-(4-fluorophenyl)-1-phenylprop-2-ynyl)piperidine (Table 3, entry 5)



Pale yellow oil, 273 mg, 93%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.66-7.61 (m, 2H), 7.55-7.47 (m,2H), 7.42-7.35 (m, 2H), 7.34-7.28 (m, 1H), 7.08-7.00 (m, 2H), 4.80 (s, 1H), 2.57 (t, J = 5.40 Hz, 4H), 1.71-1.52 (m, 4H), 1.51-1.38 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.4 (d, J_{CF} = 247.5 Hz), 138.5, 133.6 (d, J_{CF} = 8.2 Hz), 128.5, 128.1, 127.5, 119.4 (d, J_{CF} = 3.5 Hz), 115.5 (d, J_{CF} = 21.8 Hz), 86.8, 85.8, 62.4, 50.8, 26.2, 24.5; HRMS (ESI): *m/z* calcd for $C_{20}H_{20}FN$ $[M+H]^+$ 294.1653, found 294.1654.

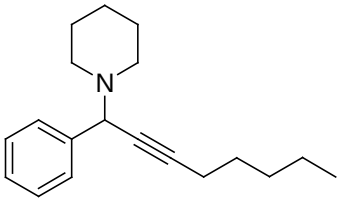
1-(3-(4-bromophenyl)-1-phenylprop-2-ynyl)piperidine (Table 3, entry 6)



Yellow solid, 346 mg, 98%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.65-7.60 (m, 2H), 7.50-7.45 (m, 2H), 7.42-7.35 (m, 4H), 7.34-7.29 (m, 1H), 4.79 (s, 1H), 2.56 (t, J = 5.20 Hz, 4H), 1.68-1.53 (m, 4H), 1.50-1.39 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 138.4, 133.3, 131.6, 128.5, 128.2, 127.6, 122.3, 122.2, 87.5, 86.8, 62.5, 50.8, 26.2, 24.5; HRMS (ESI):

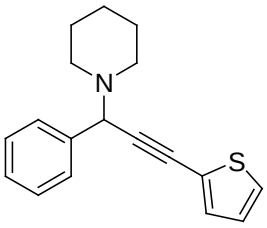
m/z calcd for $C_{20}H_{20}BrN$ $[M+H]^+$ 354.0852, found 354.0854.

1-(1-phenyloct-2-ynyl)piperidine (Table 3, entry 7)



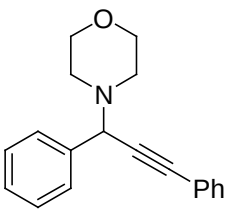
Yellow oil, 264 mg, 98%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.60-7.55 (m, 2H), 7.38-7.32 (m, 2H), 7.31-7.25 (m, 1H), 4.55 (s, 1H), 2.48 (t, *J* = 5.08 Hz, 4H), 2.33 (td, *J* = 2.16, 7.04 Hz, 2H), 1.66-1.52 (m, 6H), 1.50-1.29 (m, 6H), 0.94 (t, *J* = 7.22 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 139.3, 128.5, 127.9, 127.2, 87.9, 76.3, 62.0, 50.6, 31.2, 28.8, 26.2, 24.6, 22.2, 18.8, 14.1; HRMS (ESI): *m/z* calcd for $C_{19}H_{27}N$ $[M+H]^+$ 270.2216, found 270.2222.

1-(1-phenyl-3-(thiophen-2-yl)prop-2-ynyl)piperidine (Table 3, entry 8)



Yellow solid, 239 mg, 85%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.65-7.60 (m, 2H), 7.41-7.35 (m, 2H), 7.41-7.35 (m, 1H), 7.28-7.24 (m, 2H), 7.00 (dd, *J* = 3.68, 4.92 Hz, 1H), 4.83 (s, 1H), 2.57 (t, *J* = 5.40 Hz, 4H), 1.70-1.52 (m, 4H), 1.50-1.37 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 138.3, 131.8, 128.5, 128.1, 127.6, 126.9, 126.6, 123.3, 90.3, 80.9, 62.6, 50.8, 26.2, 24.4; HRMS (ESI): *m/z* calcd for $C_{18}H_{19}NS$ $[M+H]^+$ 282.1311, found 282.1316.

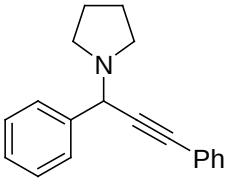
4-(1,3-diphenylprop-2-ynyl)morpholine (Table 4, entry 1)



Pale yellow oil, 266 mg, 96%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.69-7.63 (m, 2H), 7.57-7.50 (m, 2H), 7.43-7.30 (m, 6H), 4.92 (s, 1H), 3.84-3.68 (m, 4H), 2.72-2.60 (m, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 137.9, 131.9, 128.7, 128.3(8), 128.3(2), 128.3(0), 127.8, 123.0, 88.6, 85.1, 67.2, 62.1, 49.9; HRMS (ESI): *m/z* calcd for $C_{19}H_{19}NO$ $[M+H]^+$ 278.1539,

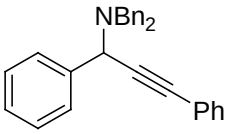
found 278.1539.

1-(1,3-diphenylprop-2-ynyl)pyrrolidine (Table 4, entry 2)

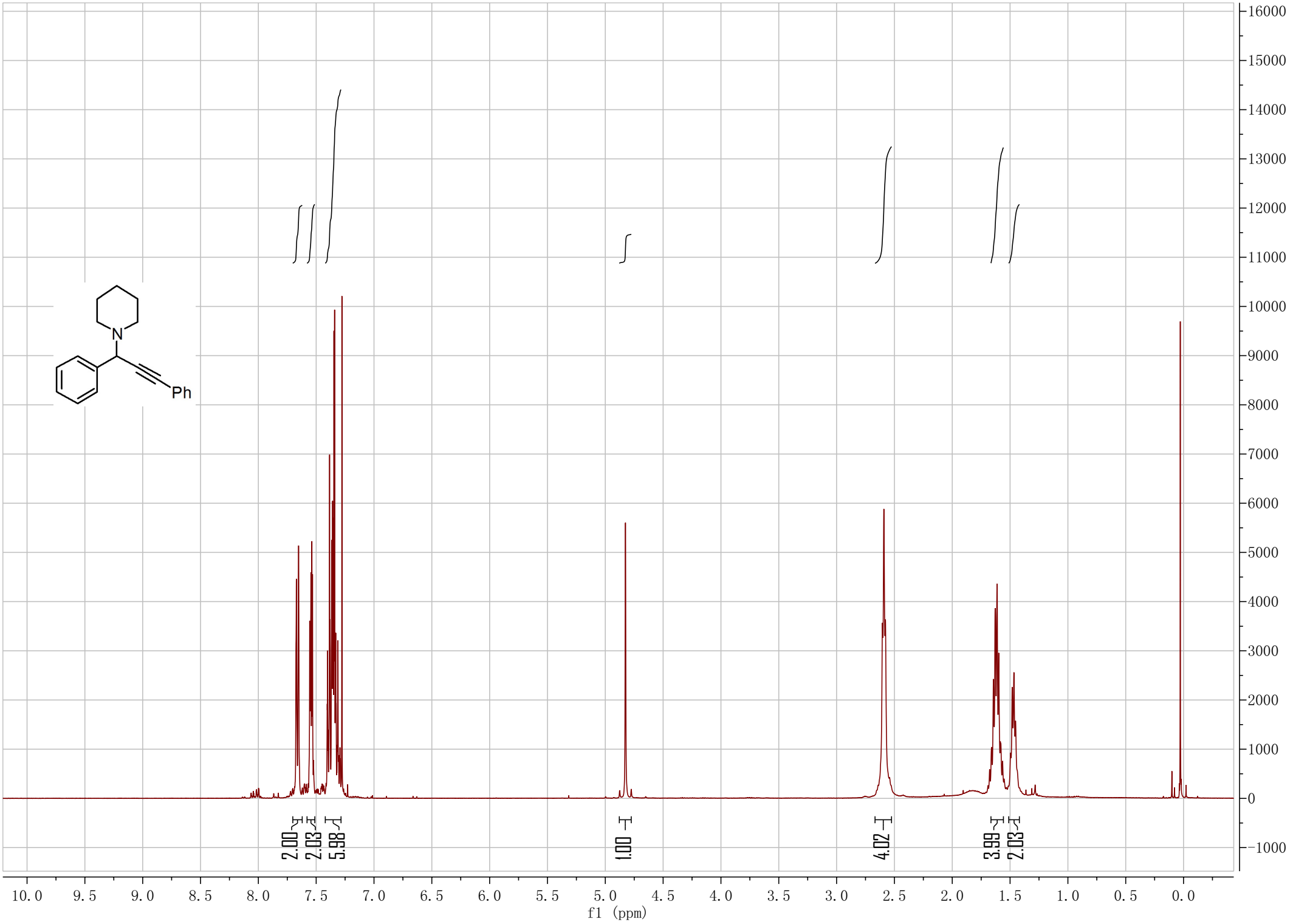


Pale yellow oil, 228 mg, 87%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.66-7.61 (m, 2H), 7.54-7.48 (m, 2H), 7.42-7.29 (m, 6H), 4.92 (s, 1H), 2.80-2.64 (m, 4H), 1.89-1.76 (m, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 139.5, 131.8, 128.3(4), 128.3(1), 128.1, 127.6, 123.3, 87.0, 86.7, 59.2, 50.3, 23.5; HRMS (ESI): *m/z* calcd for $C_{19}H_{19}N$ $[M+H]^+$ 262.1590, found 262.1590.

***N,N*-dibenzyl-1,3-diphenylprop-2-yn-1-amine** (Table 4, entry 3)



Colorless solid, 376 mg, 97%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.80-7.74 (m, 2H), 7.70-7.64 (m, 2H), 7.51-7.23 (m, 16H), 4.97 (s, 1H), 3.83 (d, J = 13.44 Hz, 2H), 3.58 (d, J = 13.48 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 139.7, 139.3, 132.1, 129.1, 128.6, 128.5, 128.40 128.3, 127.6, 127.2, 123.4, 88.8, 84.9, 56.2, 54.8; HRMS (ESI): *m/z* calcd for $C_{29}H_{25}N$ $[M+H]^+$ 388.2060, found 388.2059.


N
Ph
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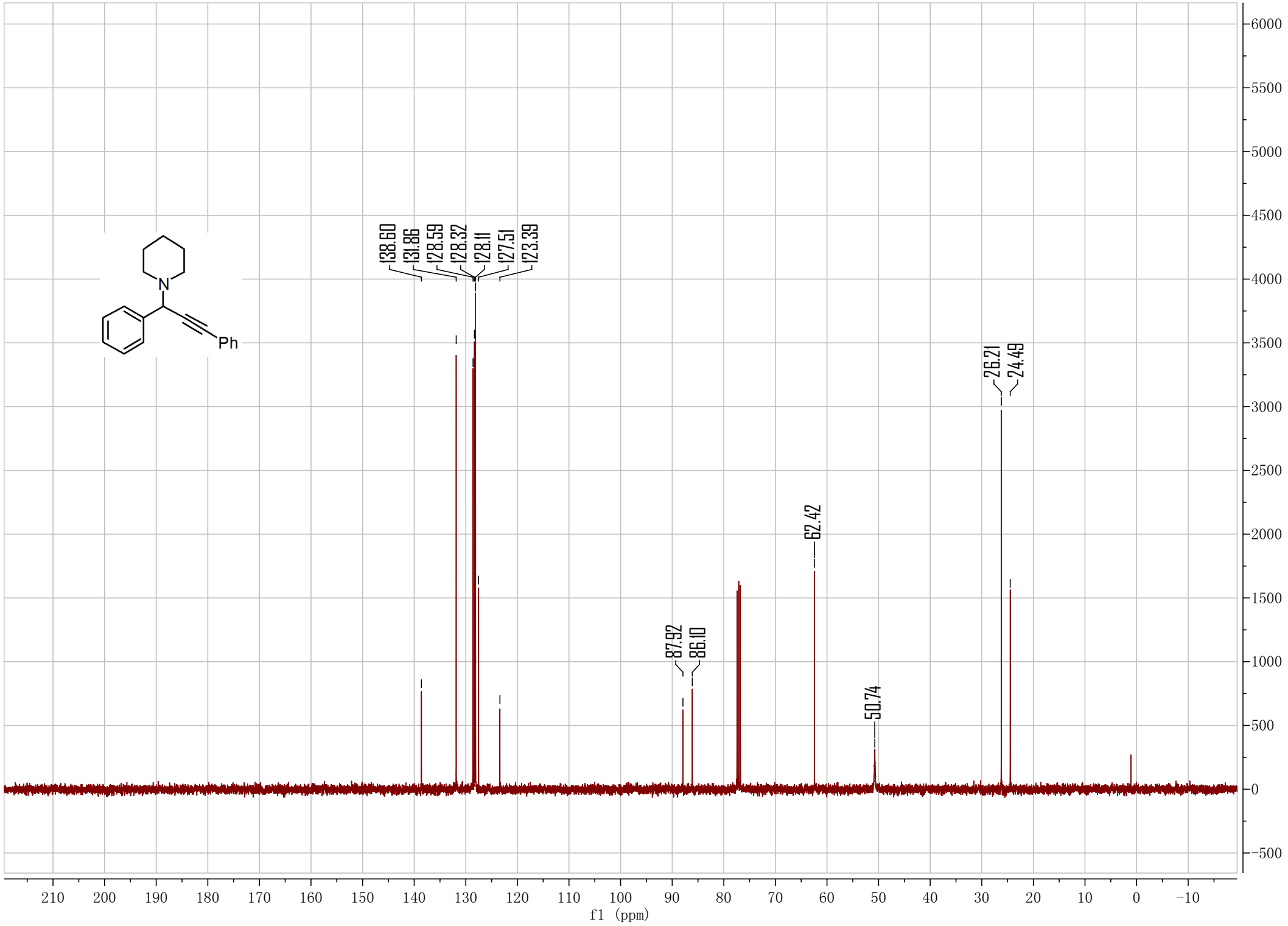
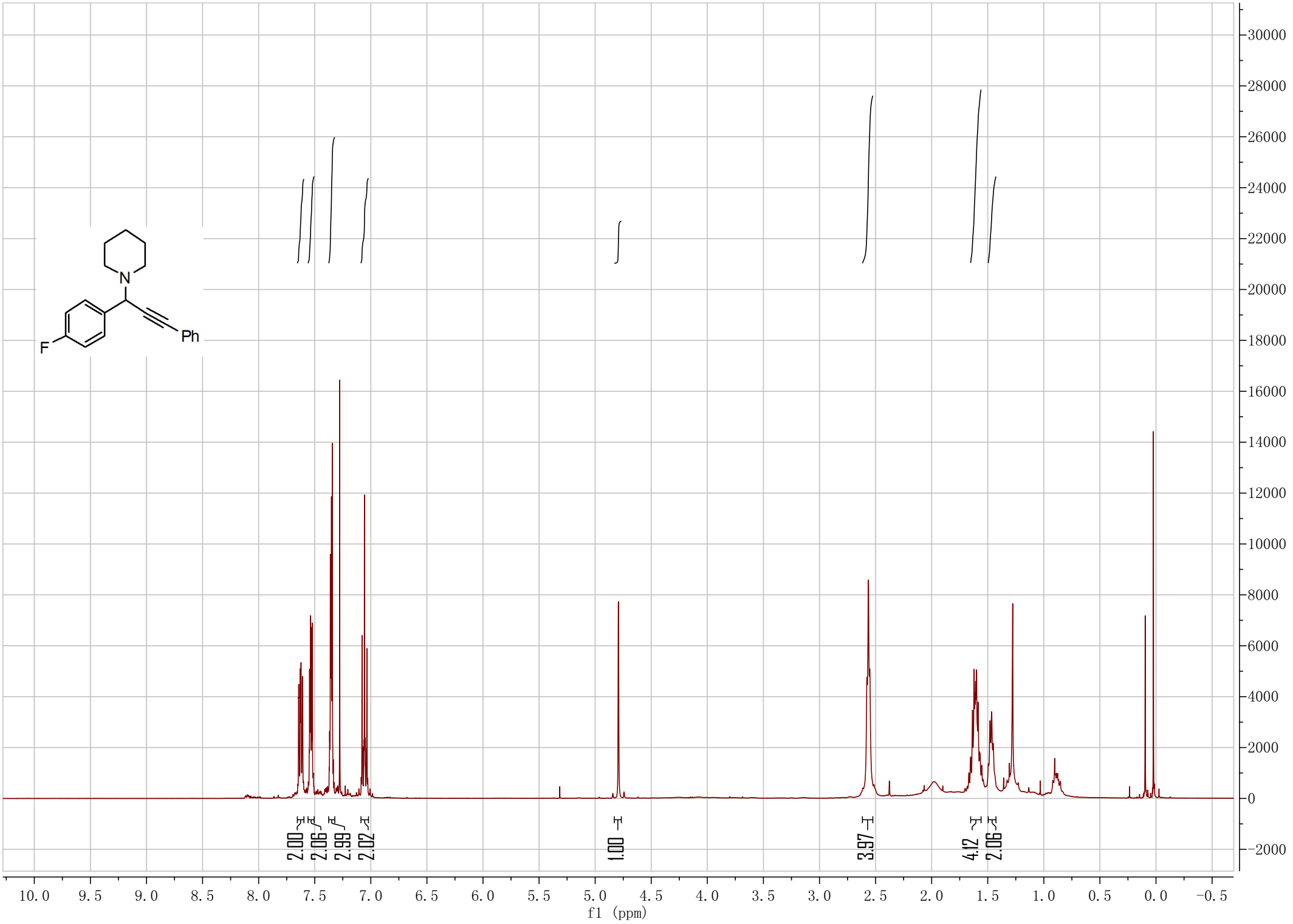
2.03
5.98
1.00
4.02
3.99
2.03
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


138.60
131.86
128.59
128.32
128.11
127.51
123.39
87.92
86.10
62.42
50.74
26.21
24.49
N
Ph
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
−500
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)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F
Ph
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2.02
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3.97
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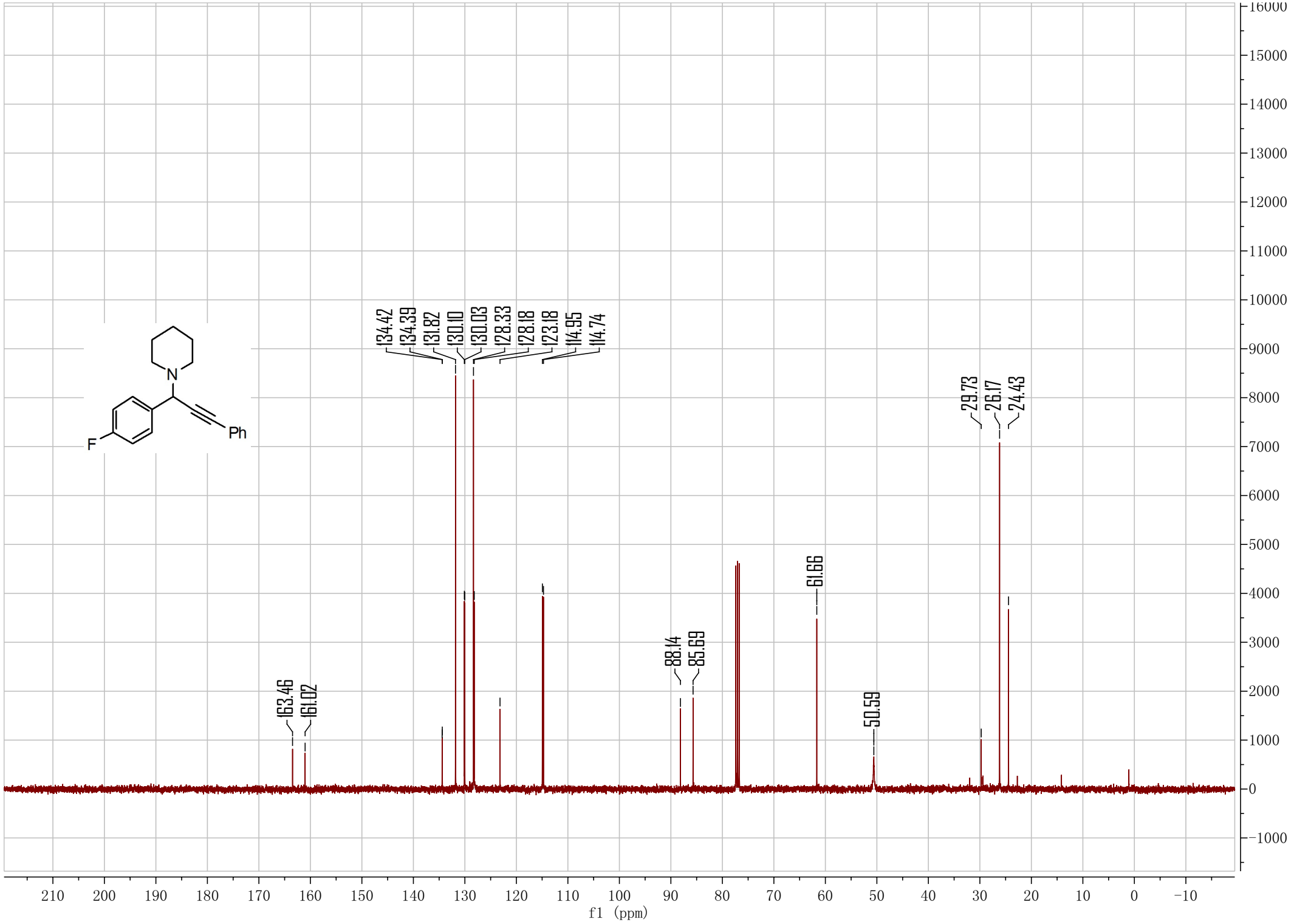
2.06
f1 (ppm)


134.42
134.39
131.82
130.10
130.03
128.33
128.18
123.18
114.95
114.74
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161.02
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85.69
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50.59
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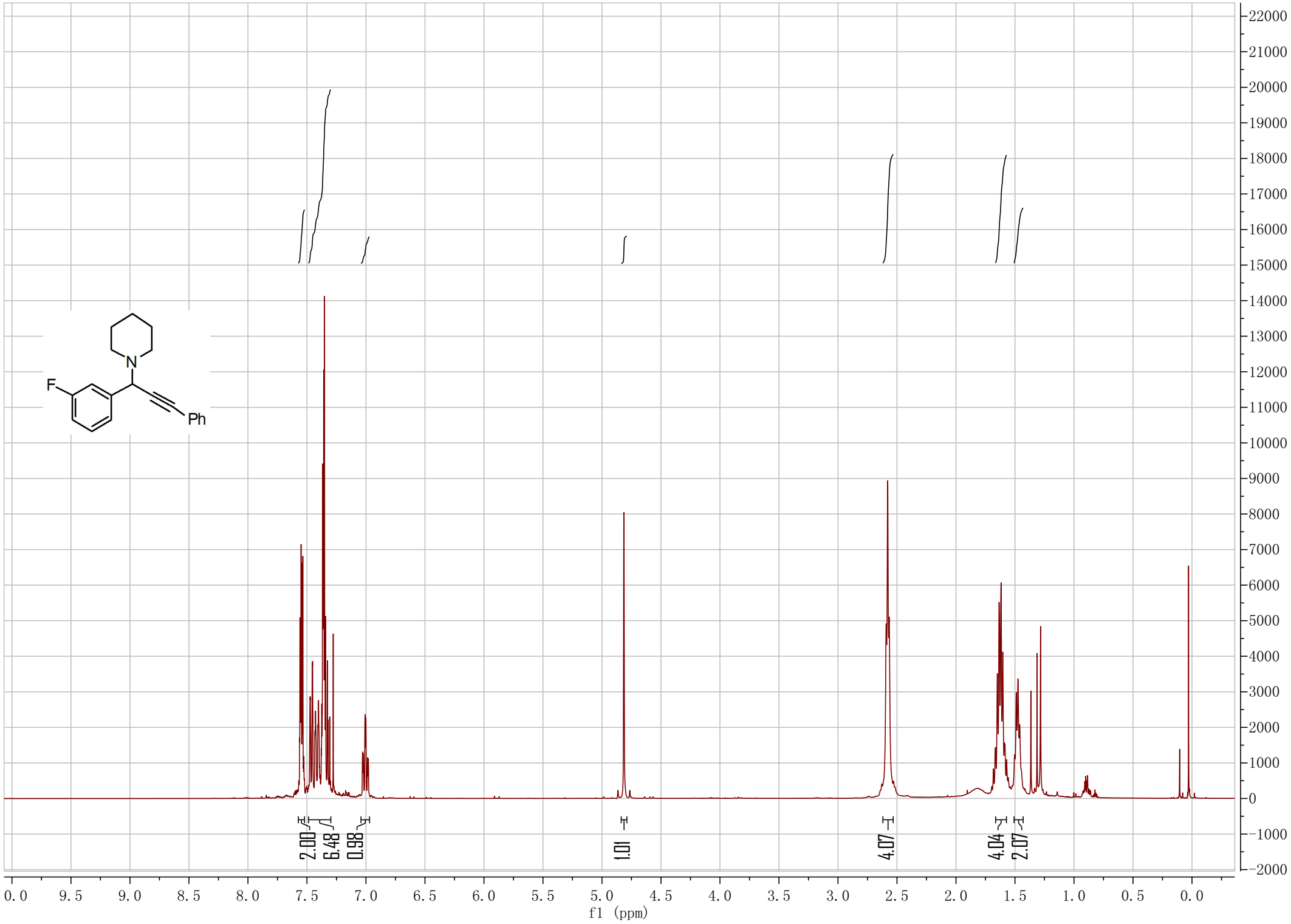
26.17
24.43
N
F
Ph
f1 (ppm)


F
N
Ph
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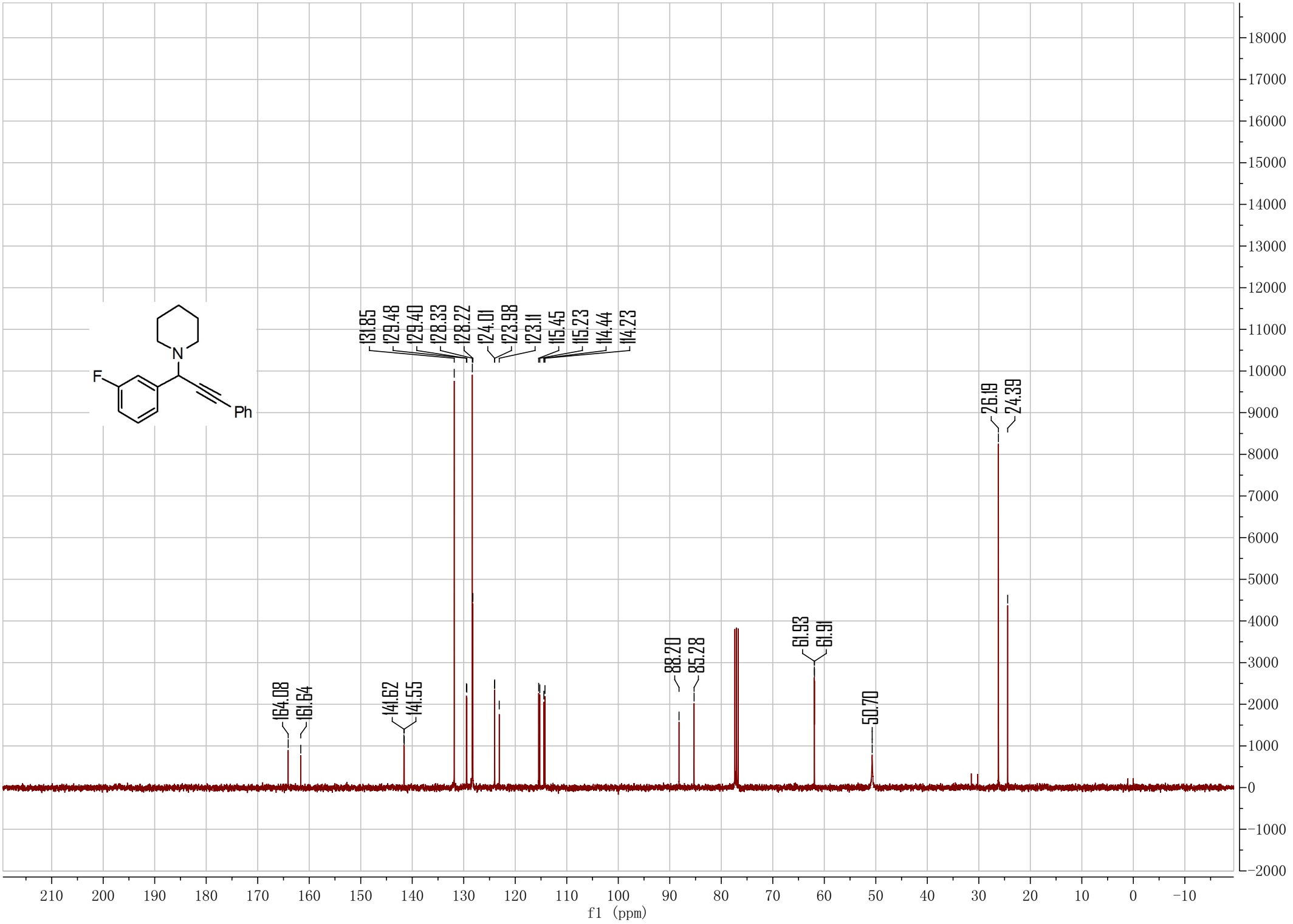
1.01
4.07
4.04
2.07
f1 (ppm)


F
N
Ph
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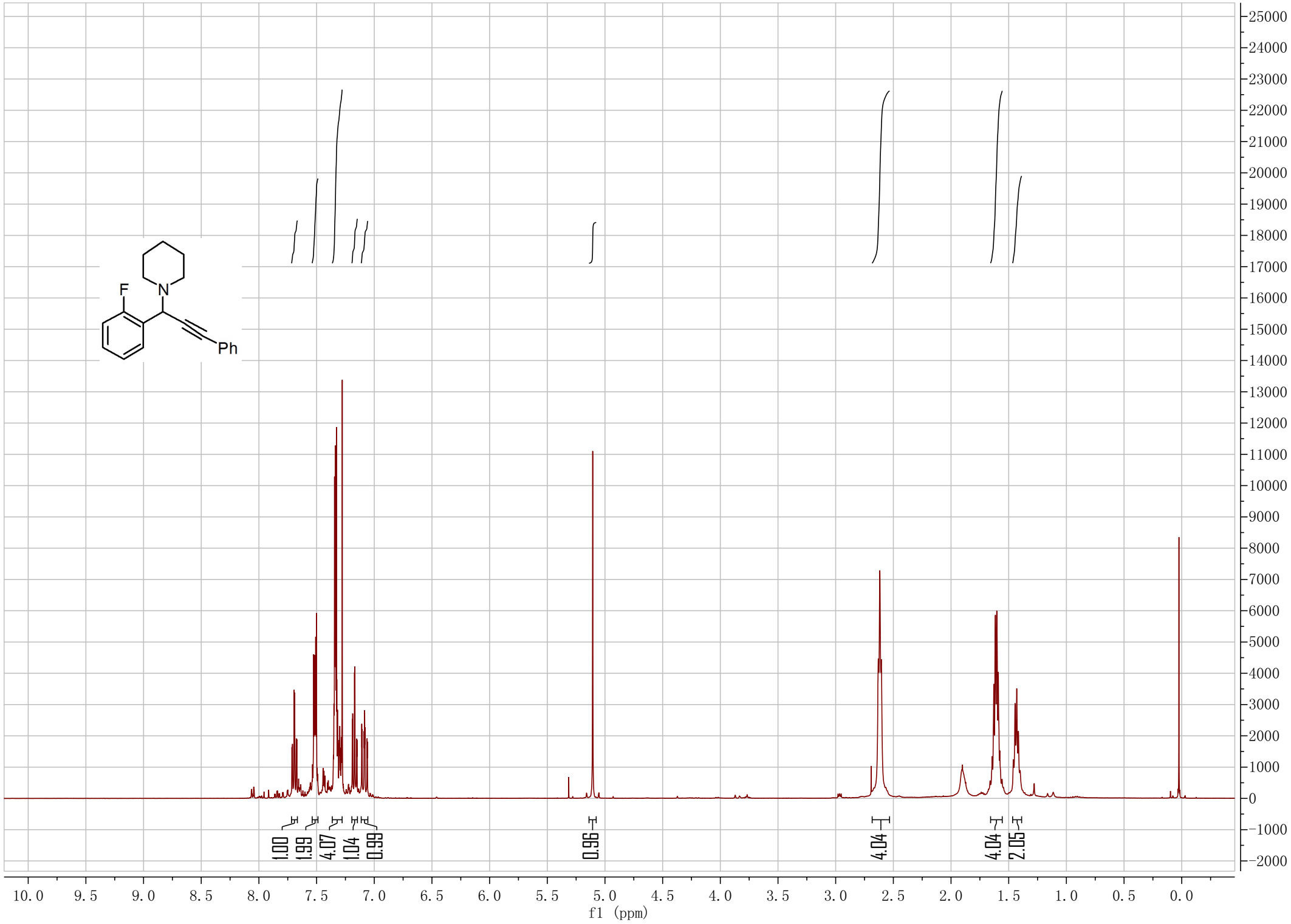
129.48
129.40
128.33
128.22
124.01
123.98
123.11
115.45
115.23
114.44
114.23
164.08
161.64
141.62
141.55
88.20
85.28
61.93
61.91
50.70
26.19
24.39
f1 (ppm)


F
N
Ph
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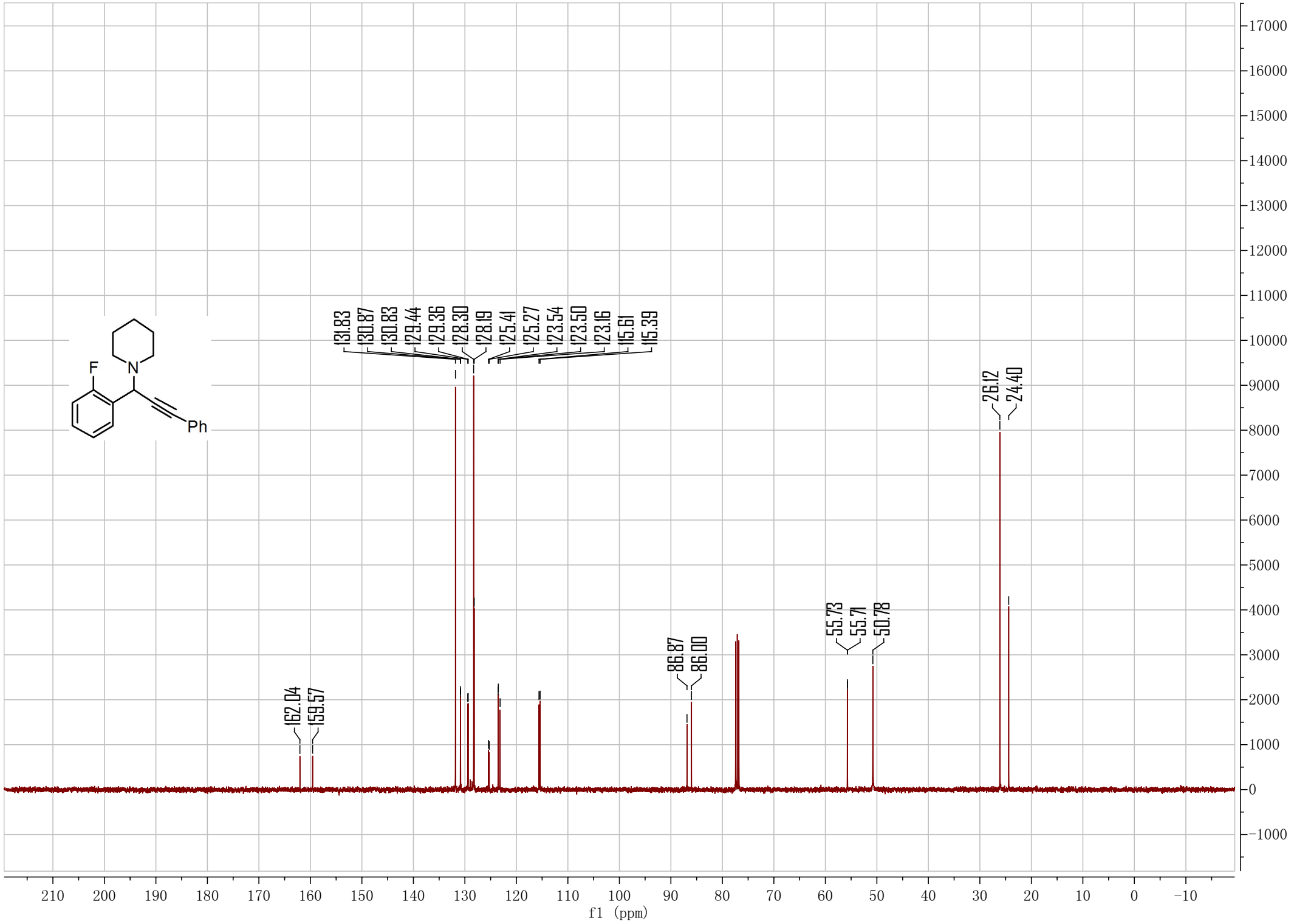
1.99
4.07
1.04
0.99
0.96
4.04
4.04
2.05
f1 (ppm)


131.83
130.87
130.83
129.44
129.36
128.30
128.19
125.41
125.27
123.54
123.50
123.16
115.61
115.39
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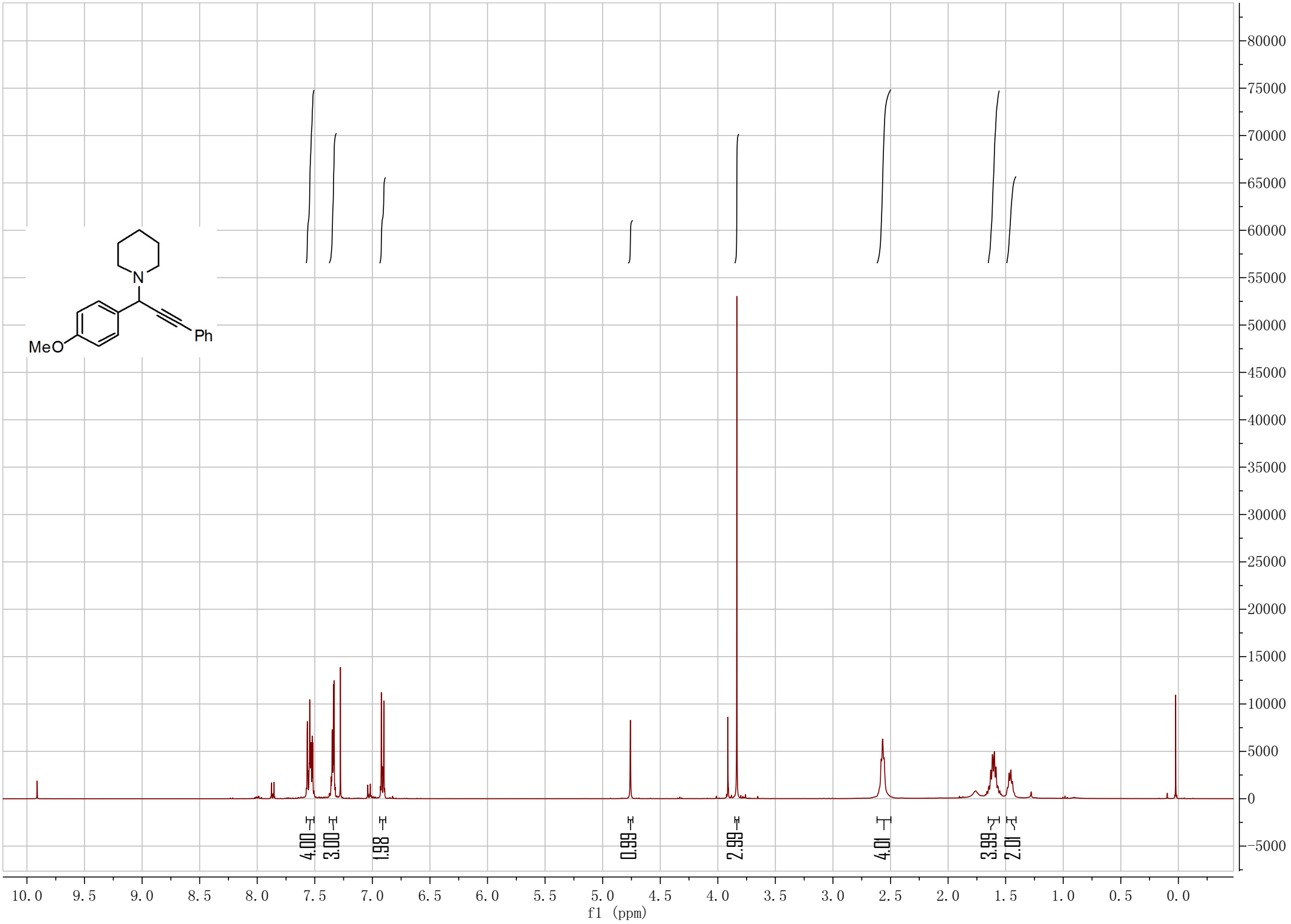
26.12
24.40
F
N
Ph
f1 (ppm)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MeO
Ph
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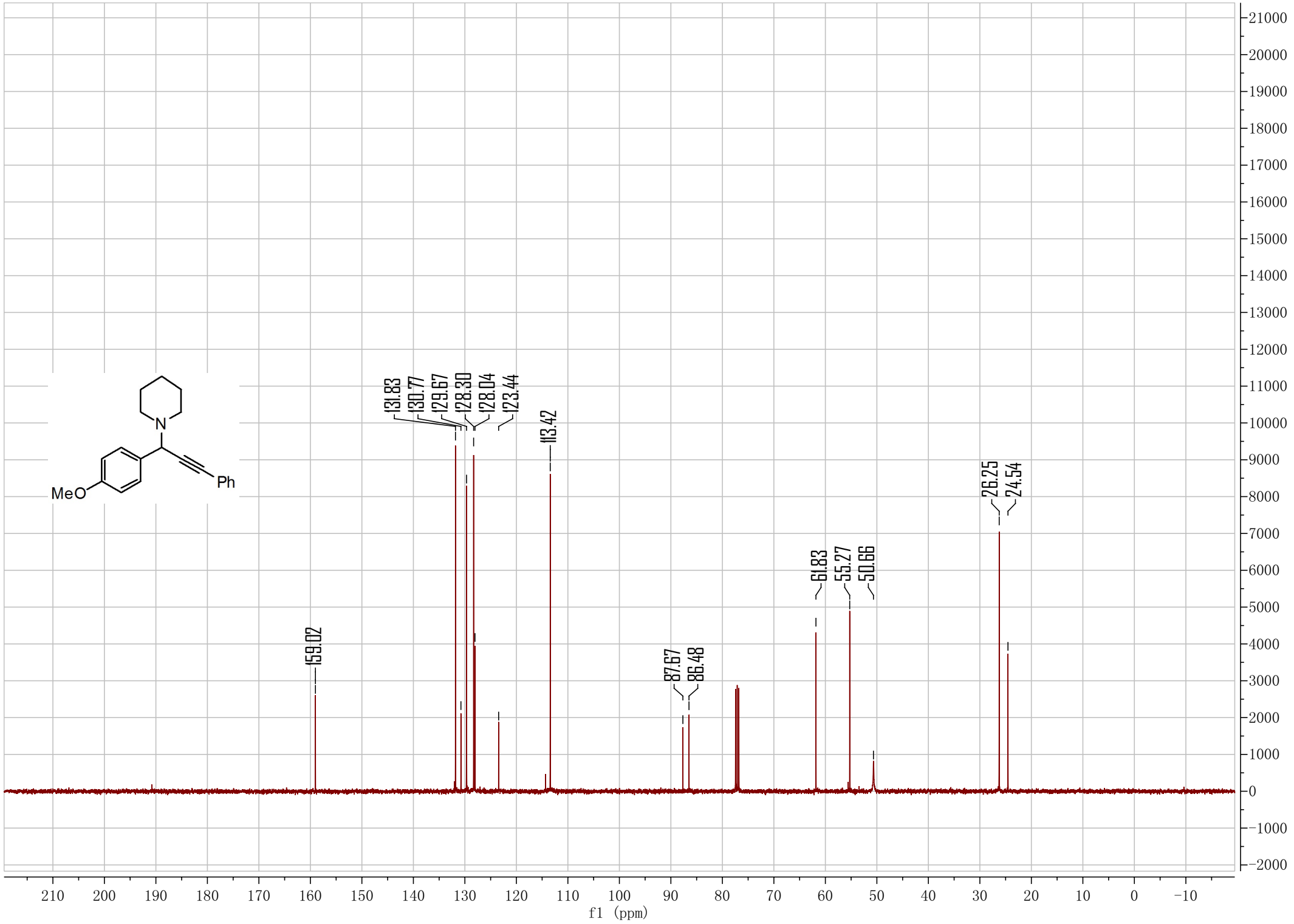
1.98
0.99
2.99
4.01
3.99
2.01
f1 (ppm)


MeO
N
Ph
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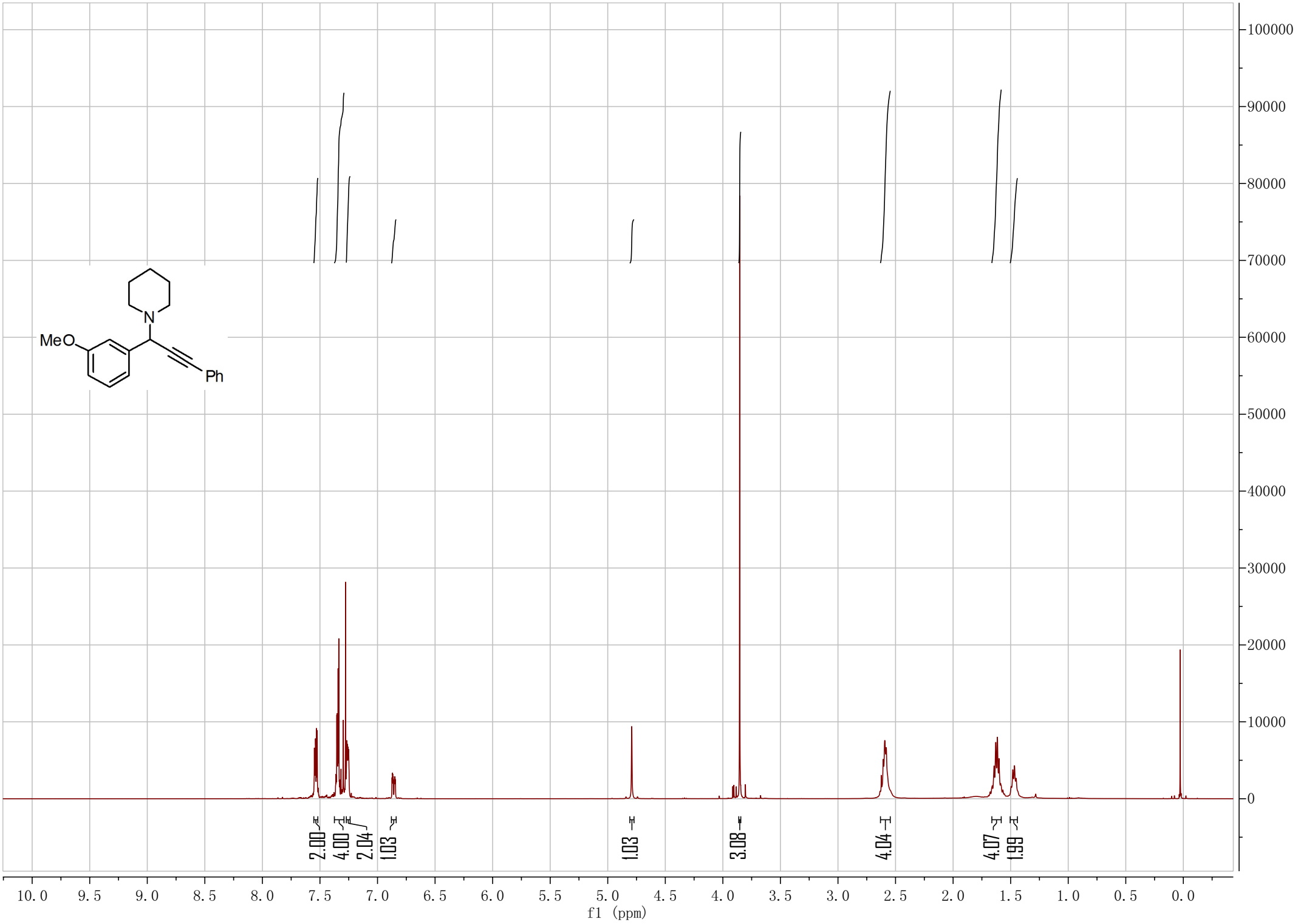
50.66
26.25
24.54
f1 (ppm)


MeO
N
Ph
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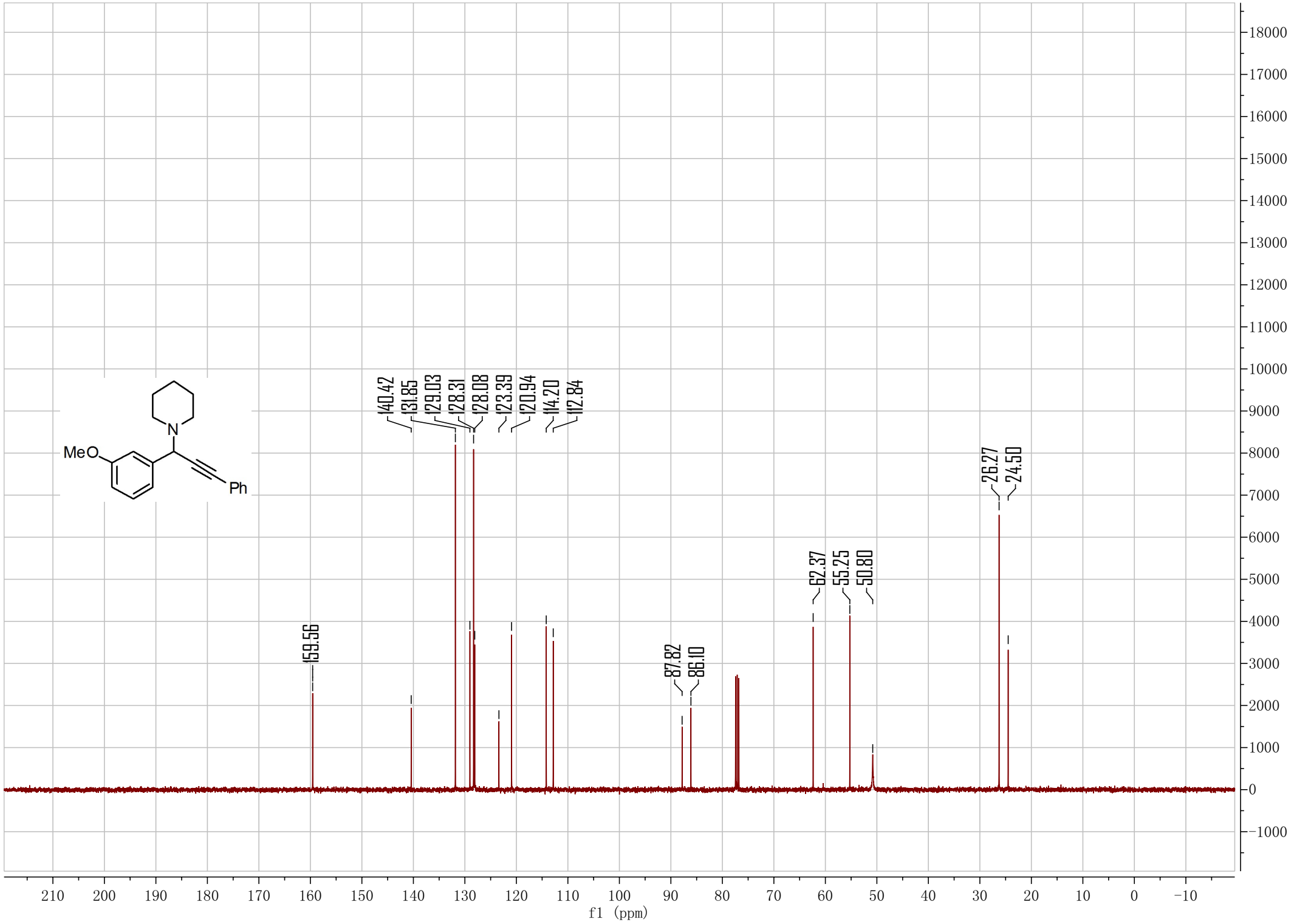
4.00
2.04
1.03
1.03
3.08
4.04
4.07
1.99
f1 (ppm)


MeO
N
Ph
140.42
131.85
129.03
128.31
128.08
123.39
120.94
114.20
112.84
159.56
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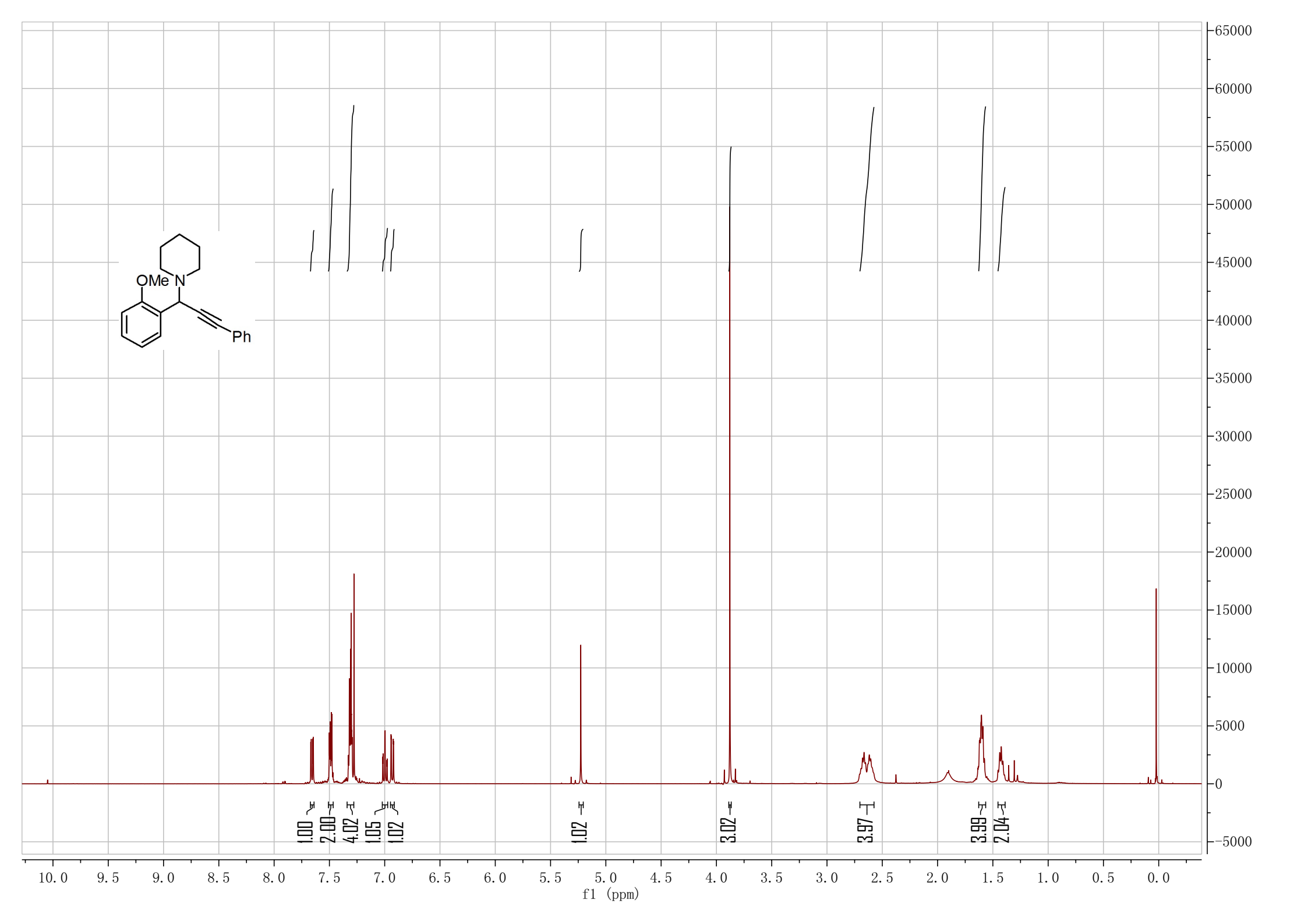
55.25
50.80
26.27
24.50
f1 (ppm)


OMe
N
Ph
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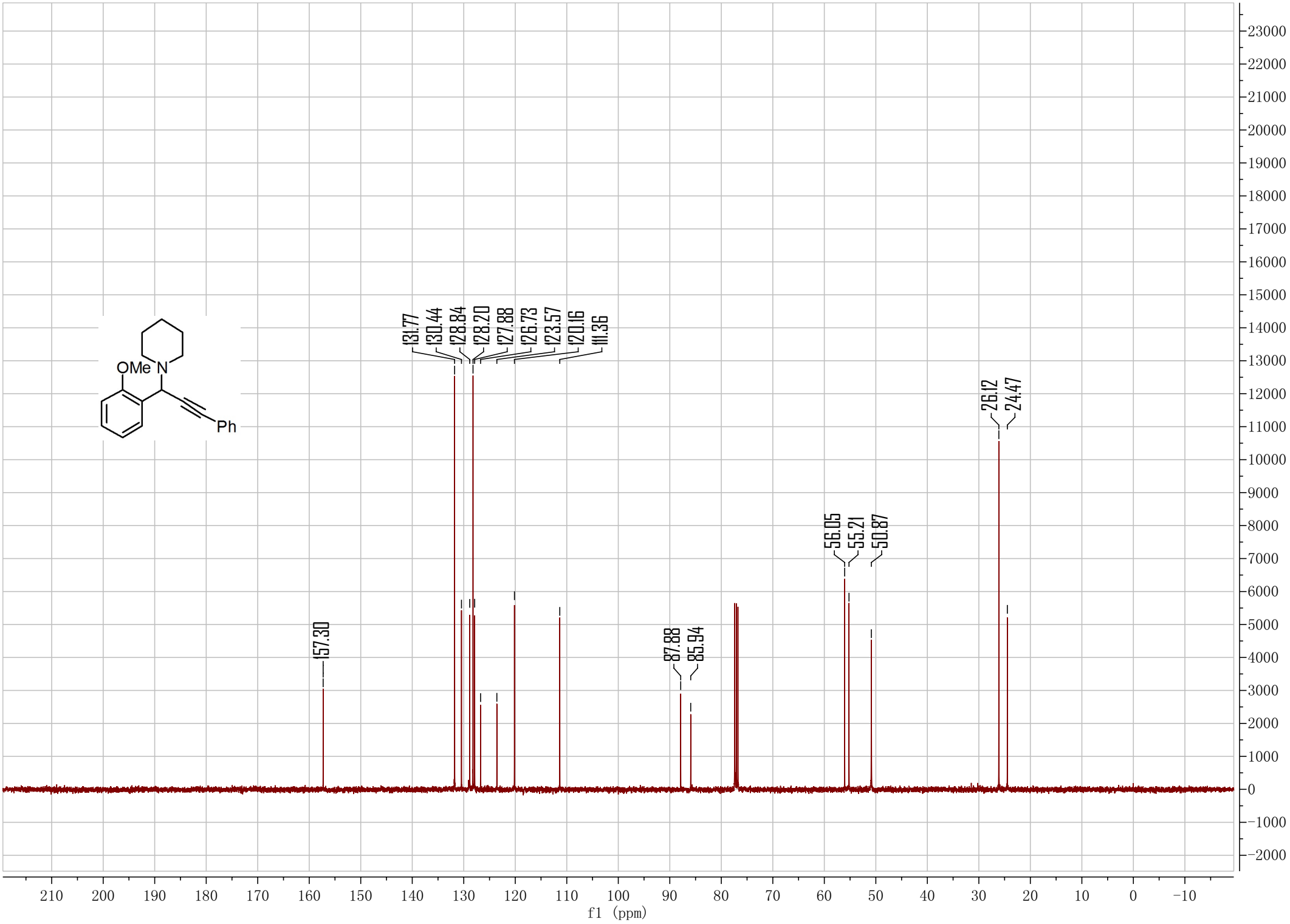
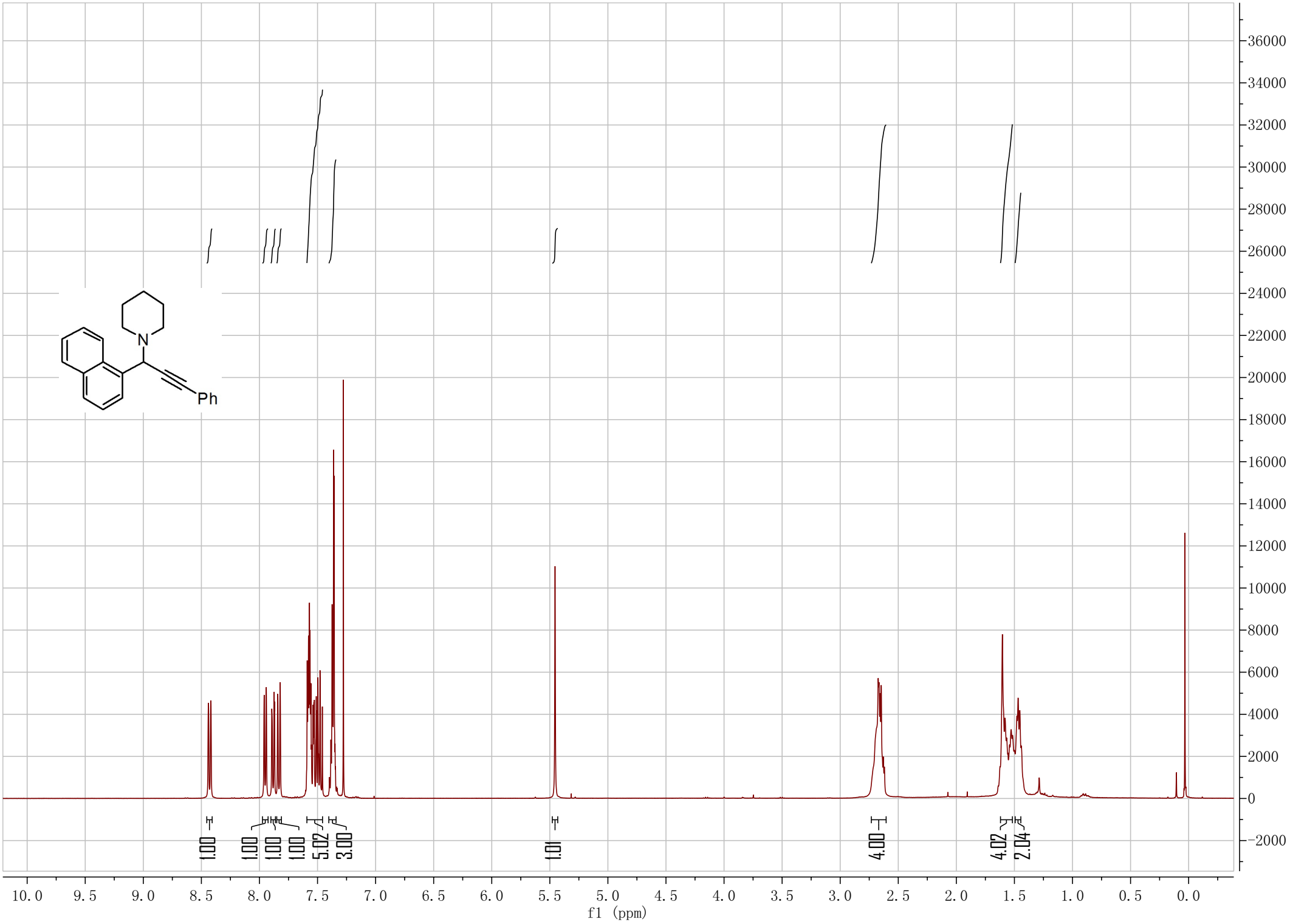
1.05
1.02
1.02
3.02
3.97
3.99
2.04
f1 (ppm)


OMe
N
Ph
131.77
130.44
128.84
128.20
127.88
126.73
123.57
120.16
111.36
157.30
87.88
85.94
56.05
55.21
50.87
26.12
24.47
f1 (ppm)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Ph
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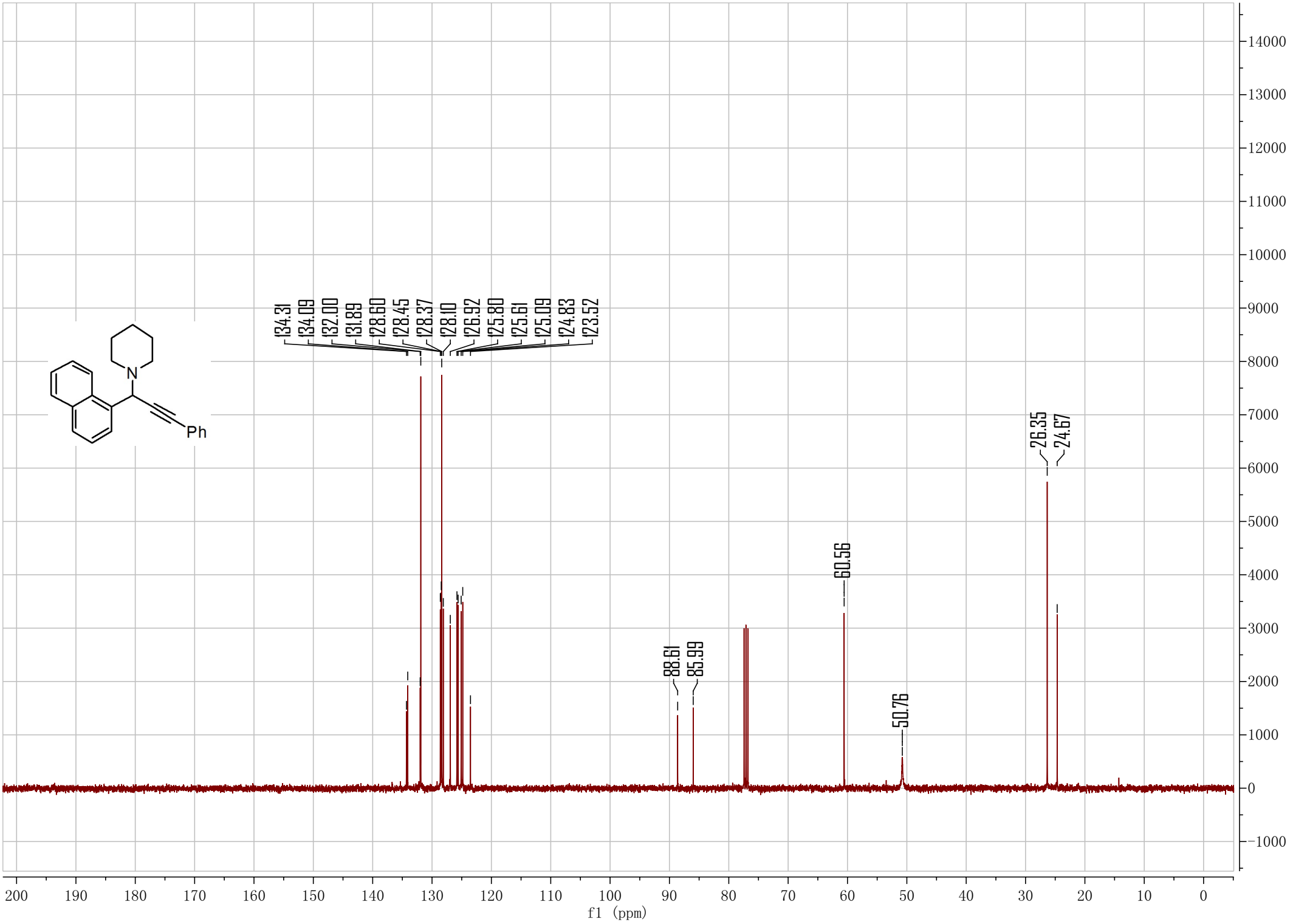
1.00
1.00
1.00
5.02
3.00
1.01
4.00
4.02
2.04
36000
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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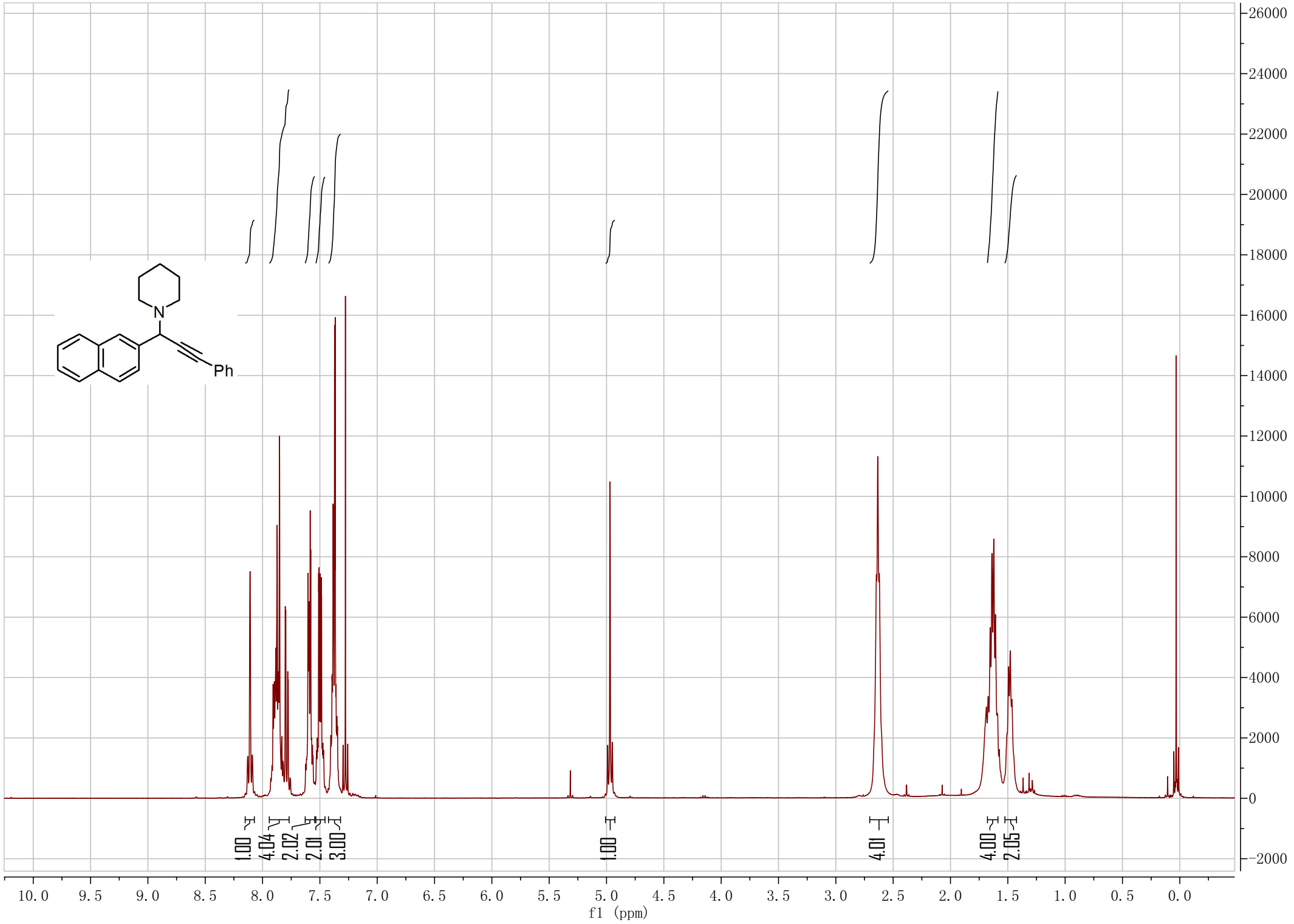
134.09
132.00
131.89
128.60
128.45
128.37
128.10
126.92
125.80
125.61
125.09
124.83
123.52
88.61
85.99
60.56
50.76
26.35
24.67
N
Ph
f1 (ppm)


N
Ph
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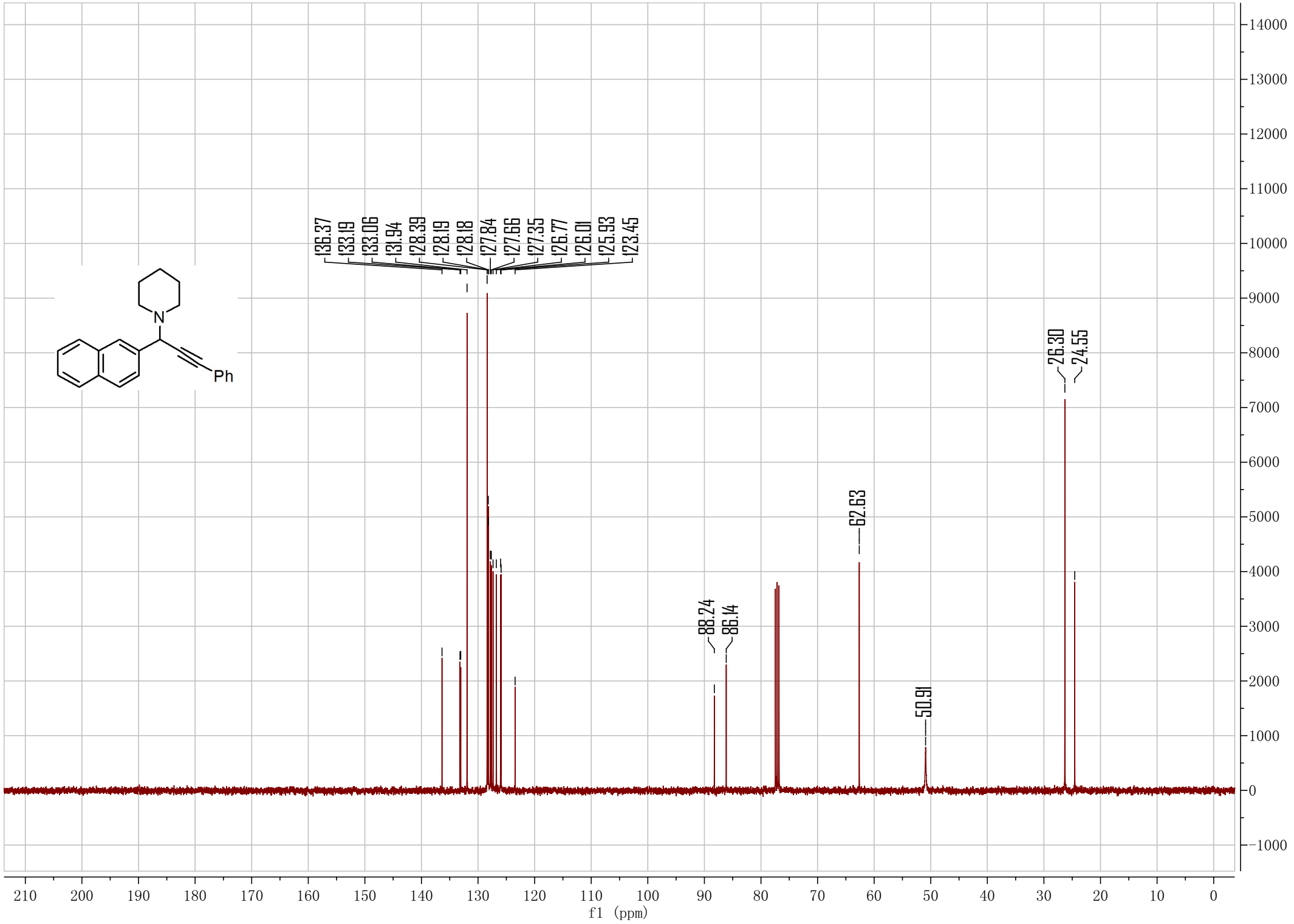
4.04
2.02
2.01
3.00
1.00
4.01
4.00
2.05
f1 (ppm)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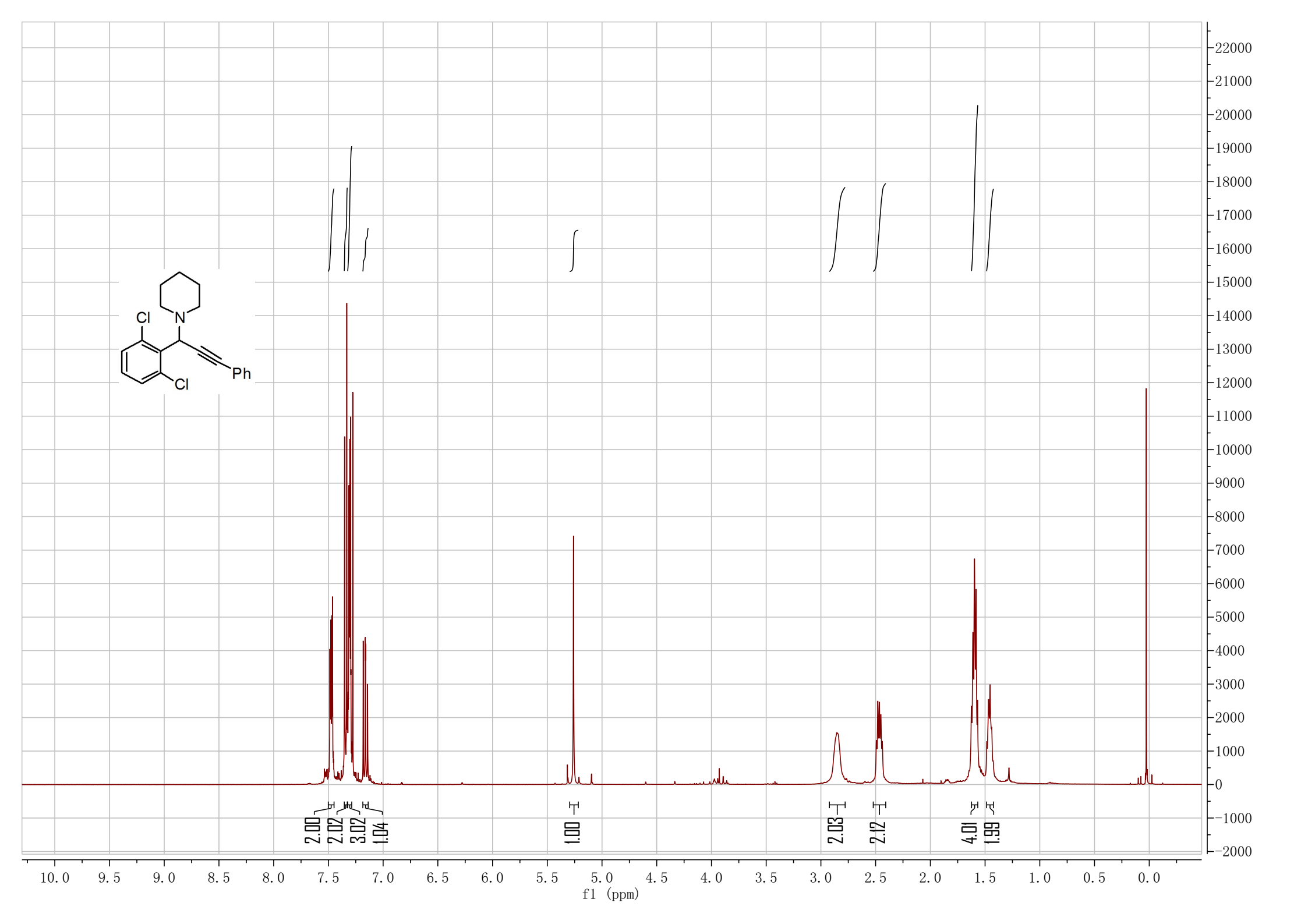
133.19
133.06
131.94
128.39
128.19
128.18
127.84
127.66
127.35
126.77
126.01
125.93
123.45
88.24
86.14
62.63
50.91
26.30
24.55
N
Ph
f1 (ppm)


Cl
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Cl
Ph
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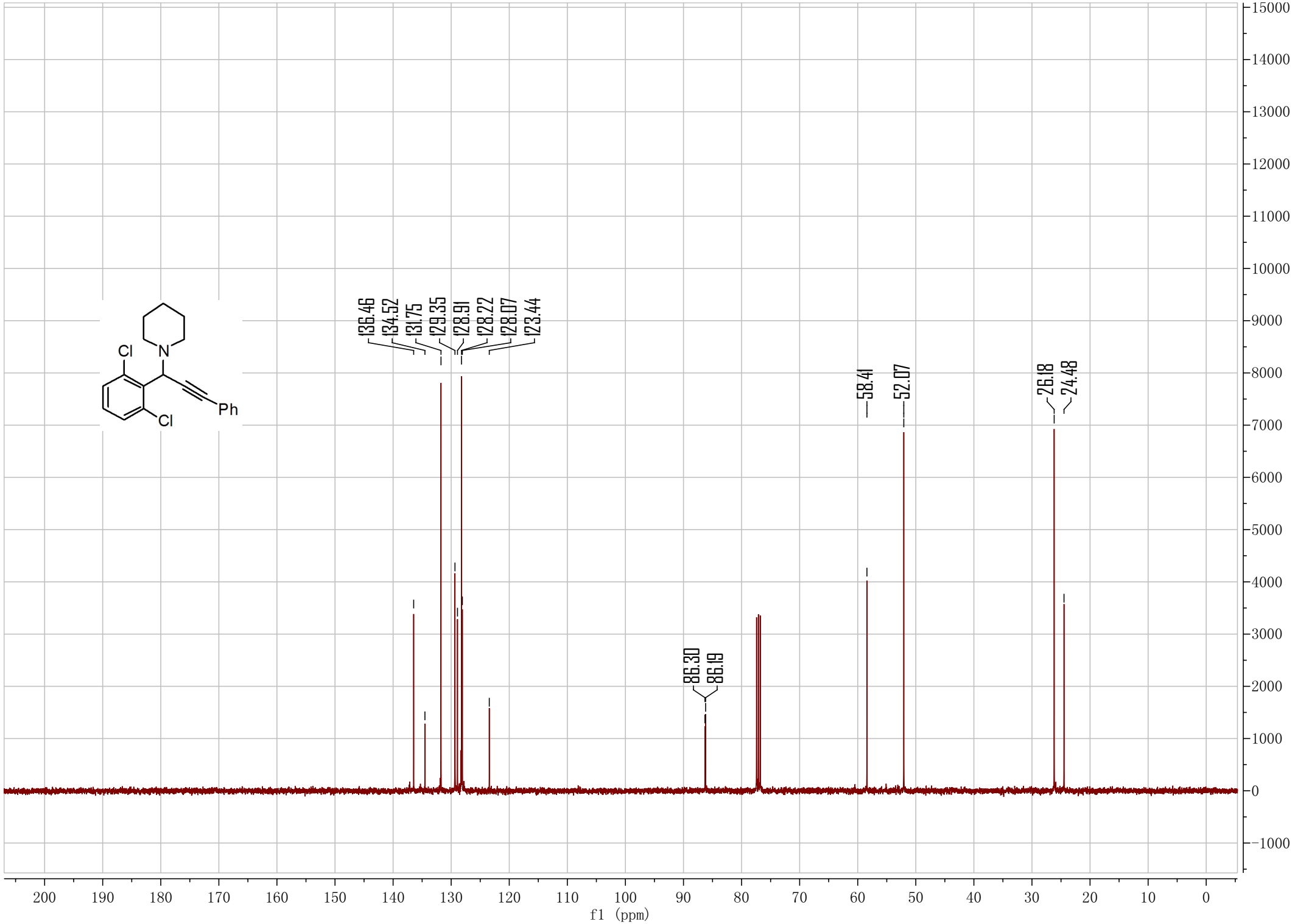
2.02
3.02
1.04
1.00
2.03
2.12
4.01
1.99
f1 (ppm)


136.46
134.52
131.75
129.35
128.91
128.22
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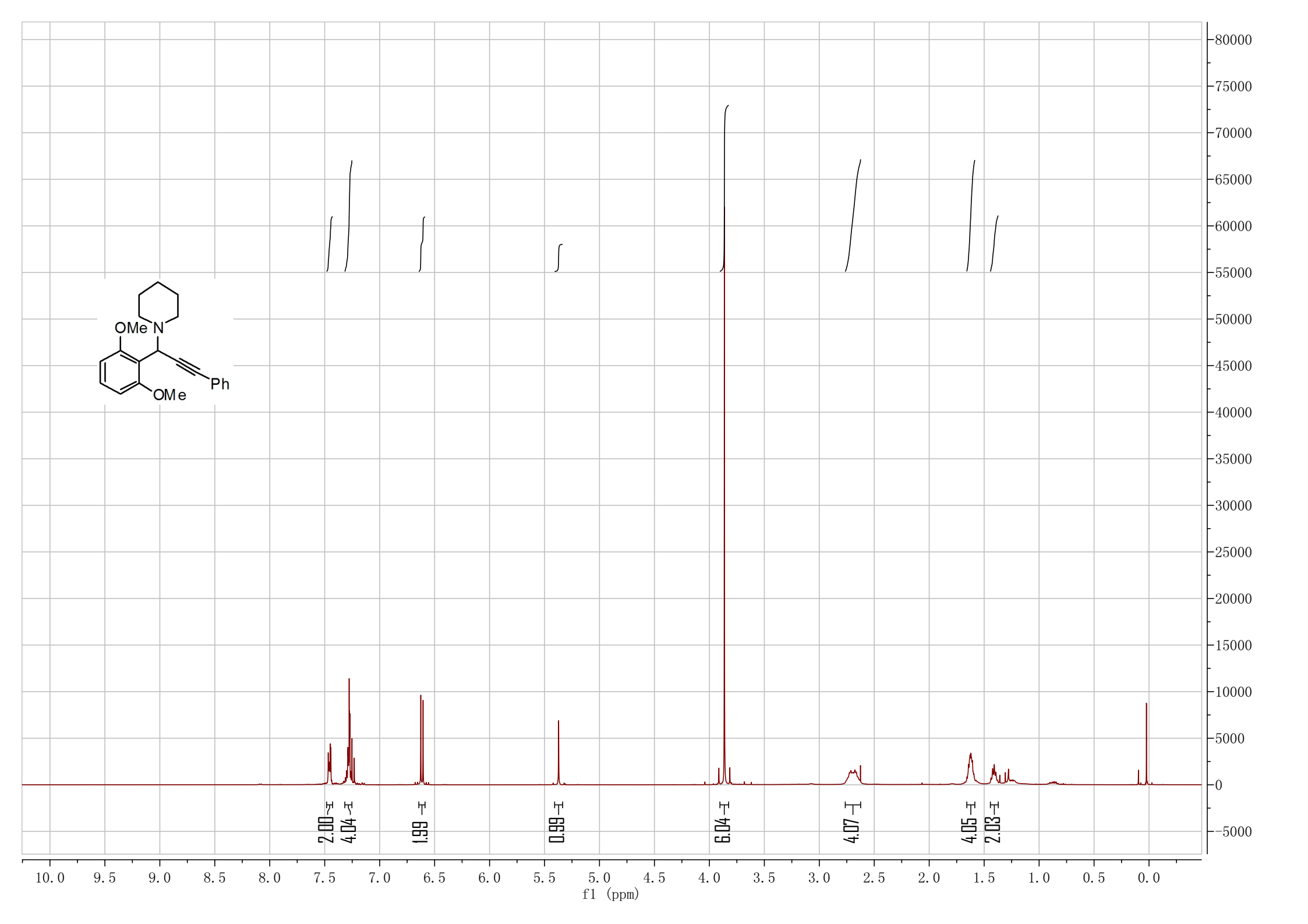
58.41
52.07
26.18
24.48
86.30
86.19
Cl
N
Cl
Ph
f1 (ppm)


OMe
N
OMe
Ph
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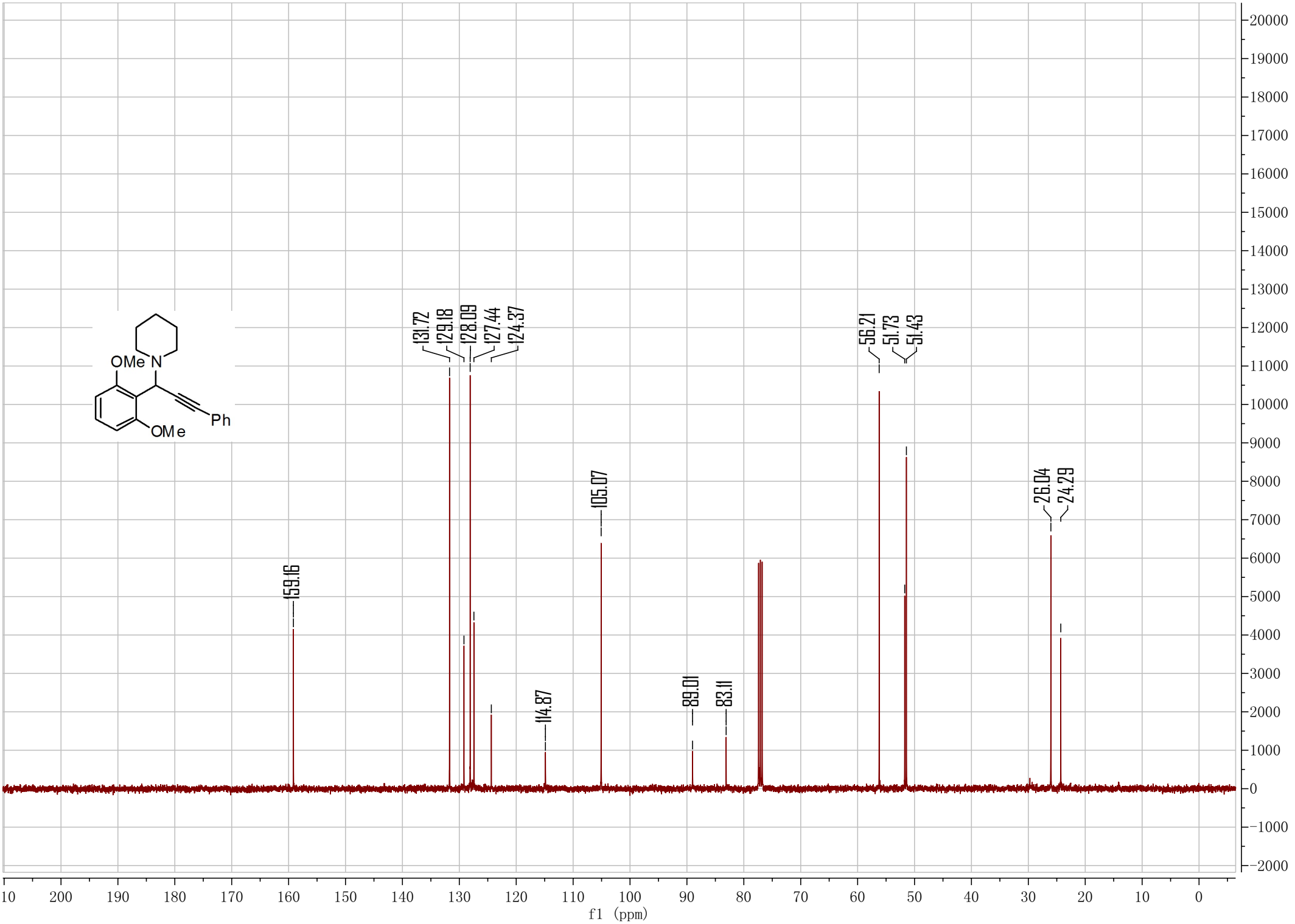
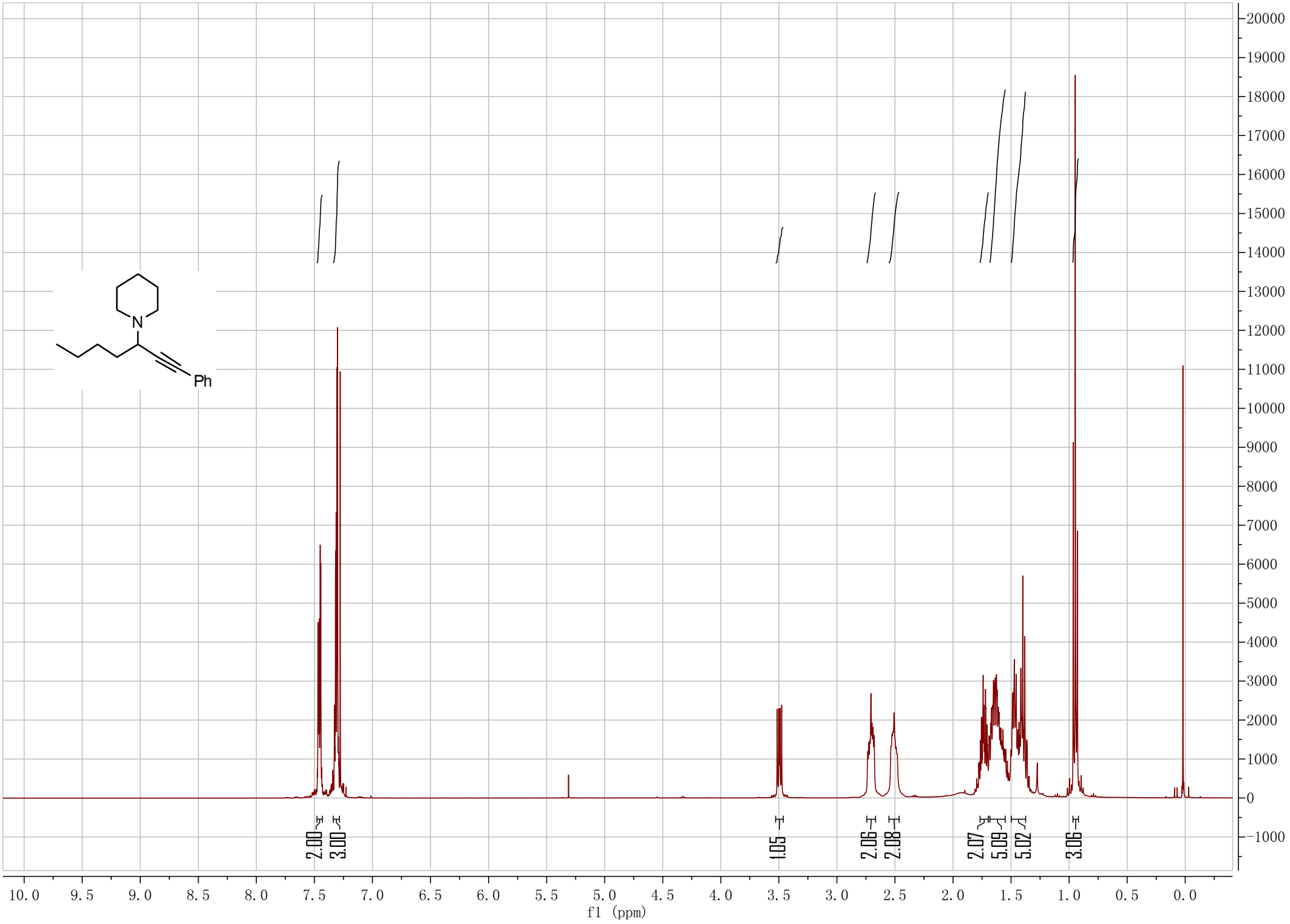
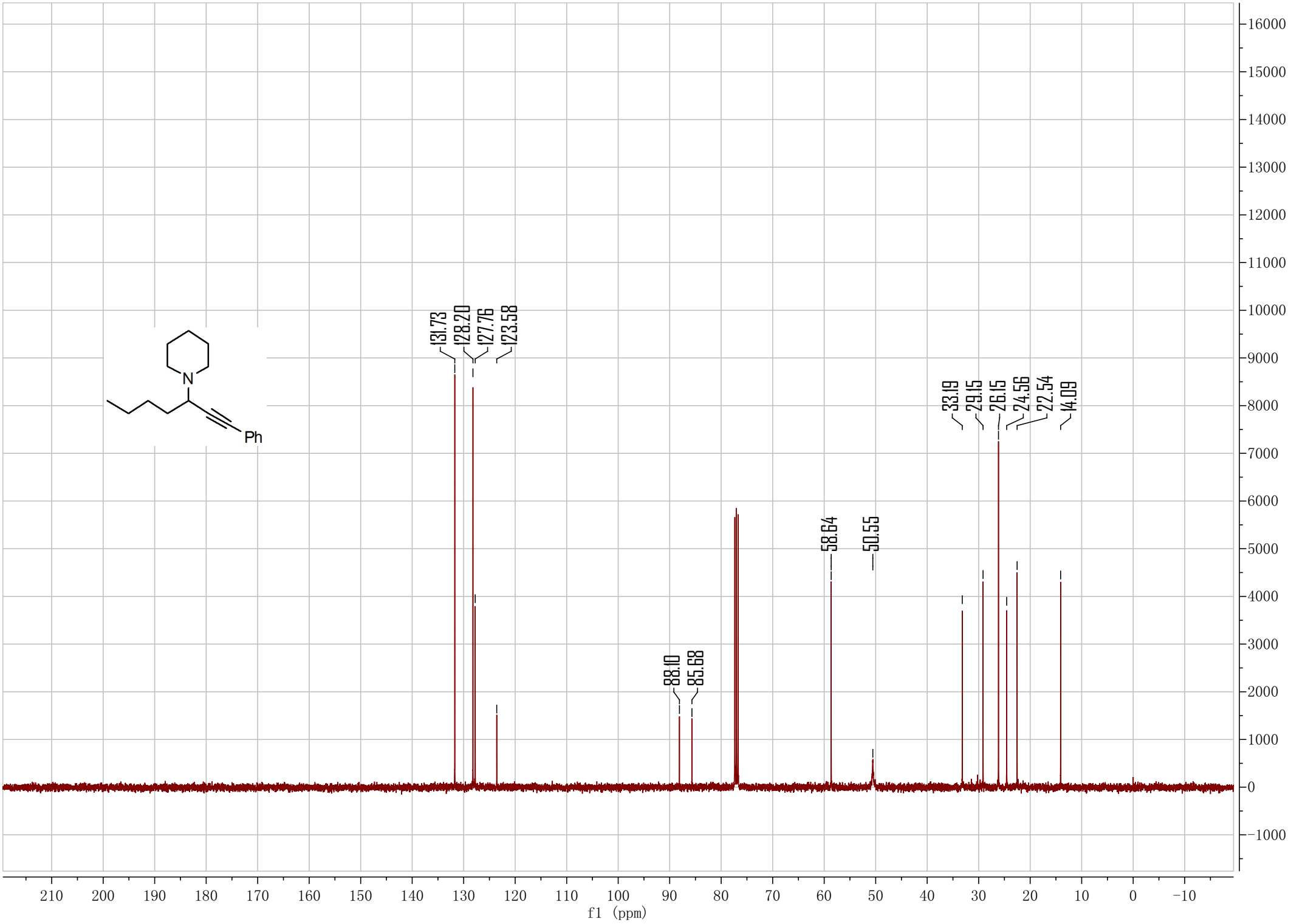
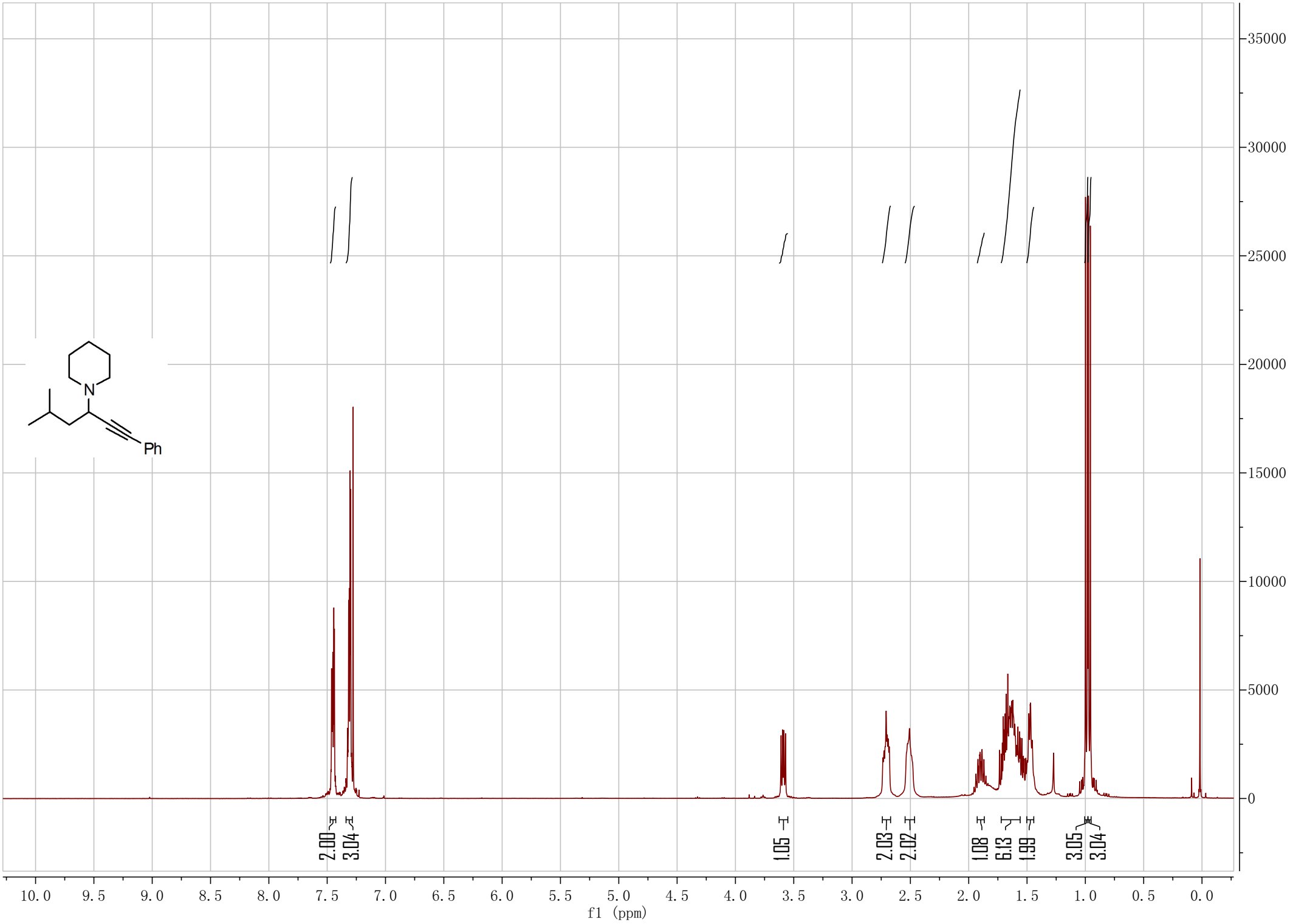
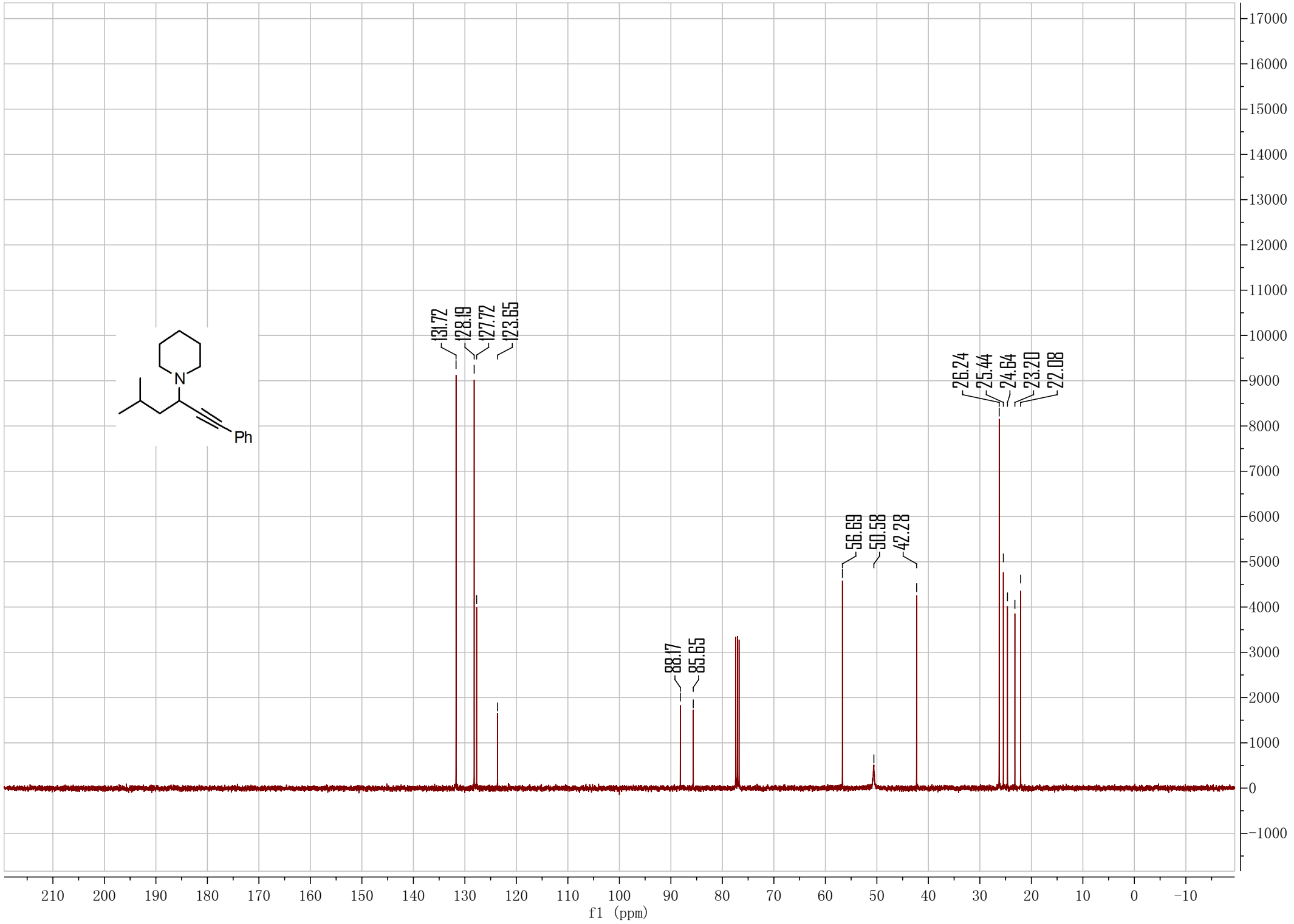
4.04
1.99
0.99
6.04
4.07
4.05
2.03
f1 (ppm)


OMe
N
OMe
Ph
131.72
129.18
128.09
127.44
124.37
159.16
105.07
114.87
89.01
83.11
56.21
51.73
51.43
26.04
24.29
f1 (ppm)


N
Ph
2.00
3.00
1.05
2.06
2.08
2.07
5.09
5.02
3.06
f1 (ppm)


131.73
128.20
127.76
123.58
88.10
85.68
58.64
50.55
33.19
29.15
26.15
24.56
22.54
14.09
N
Ph
f1 (ppm)


N
Ph
2.00
3.04
1.05
2.03
2.02
1.08
6.13
1.99
3.05
3.04
35000
30000
25000
20000
15000
10000
5000
0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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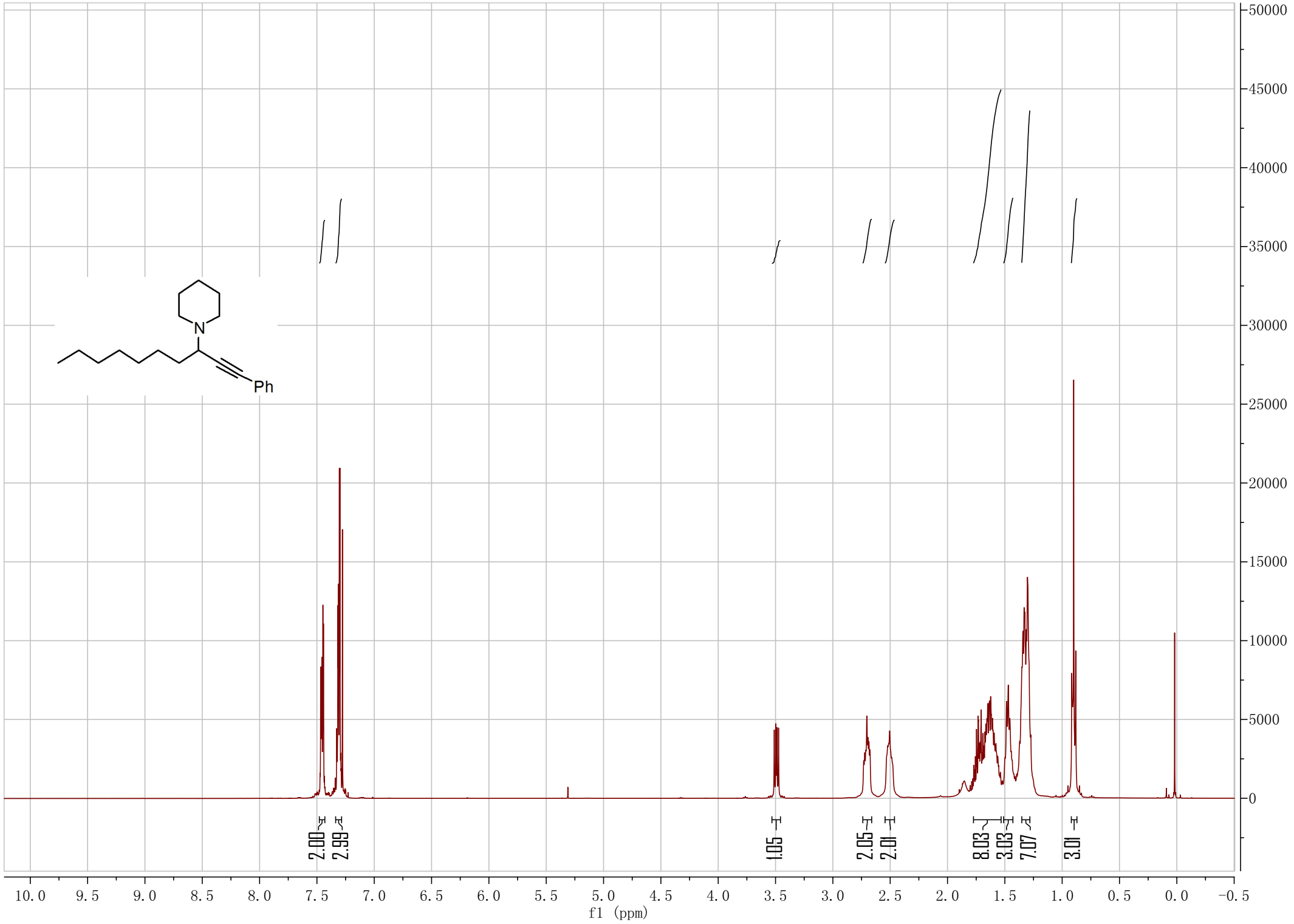
128.19
127.72
123.65
88.17
85.65
56.69
50.58
42.28
26.24
25.44
24.64
23.20
22.08
N
Ph
f1 (ppm)


N
Ph
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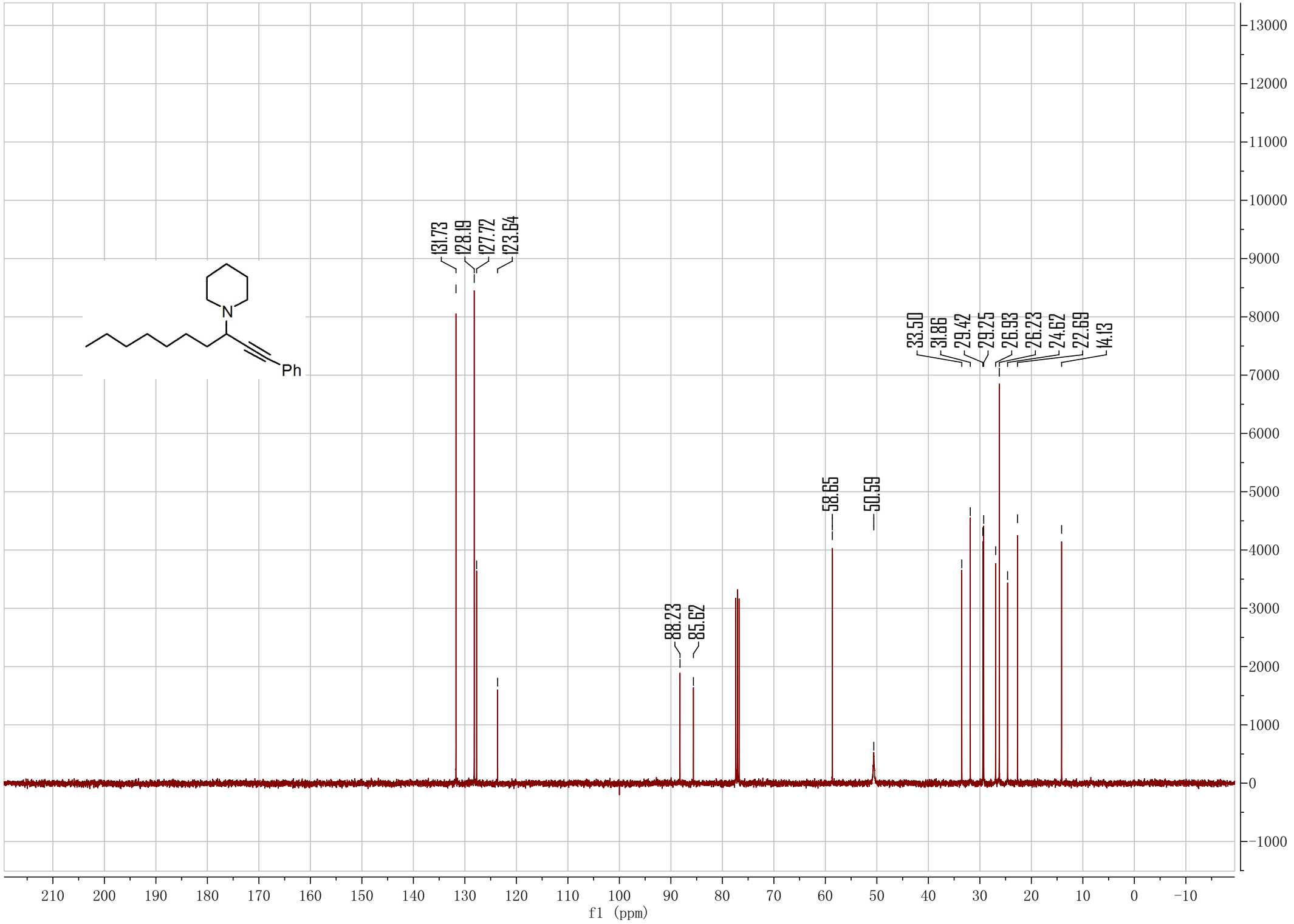
2.99
1.05
2.05
2.01
8.03
3.03
7.07
3.01
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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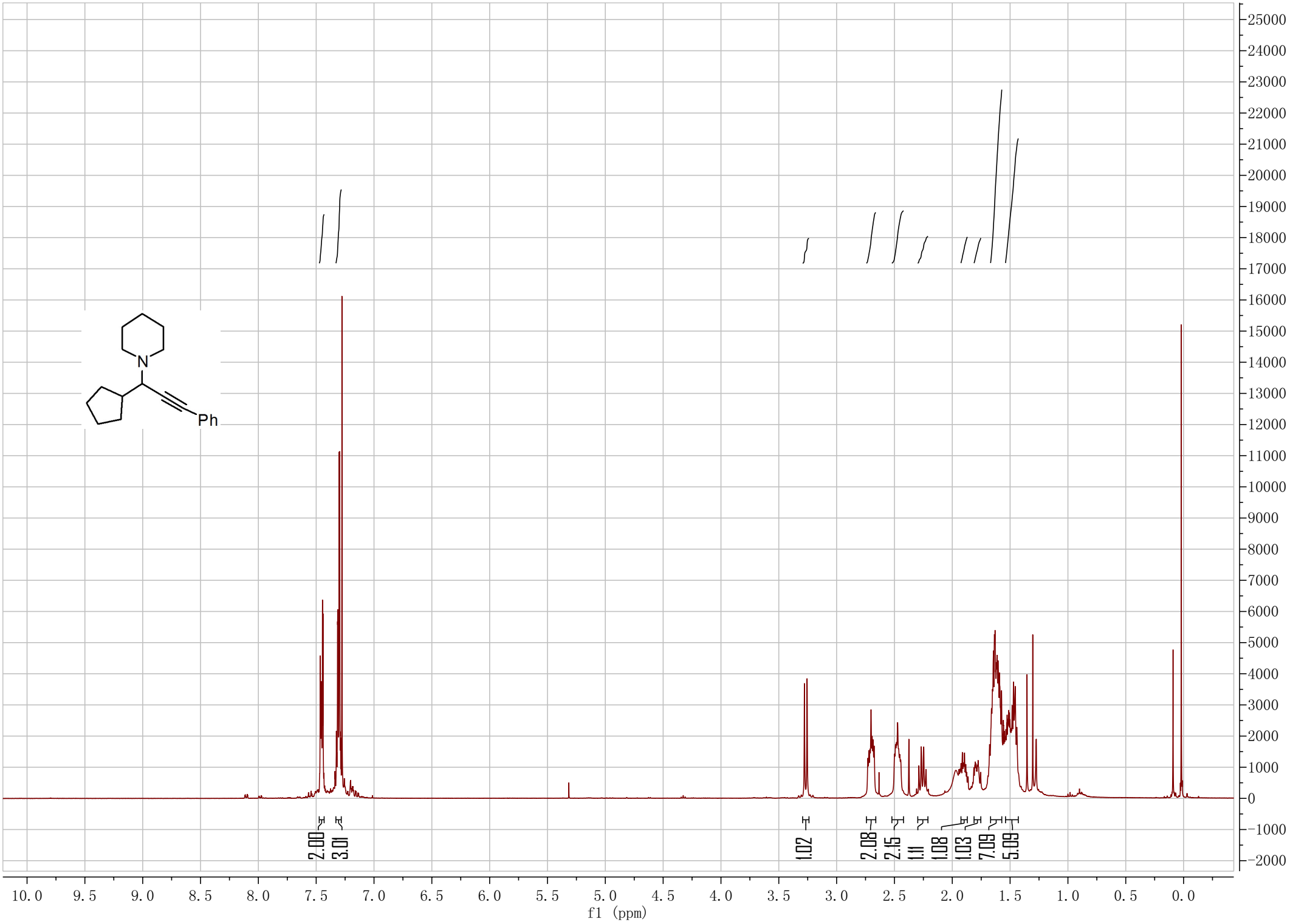
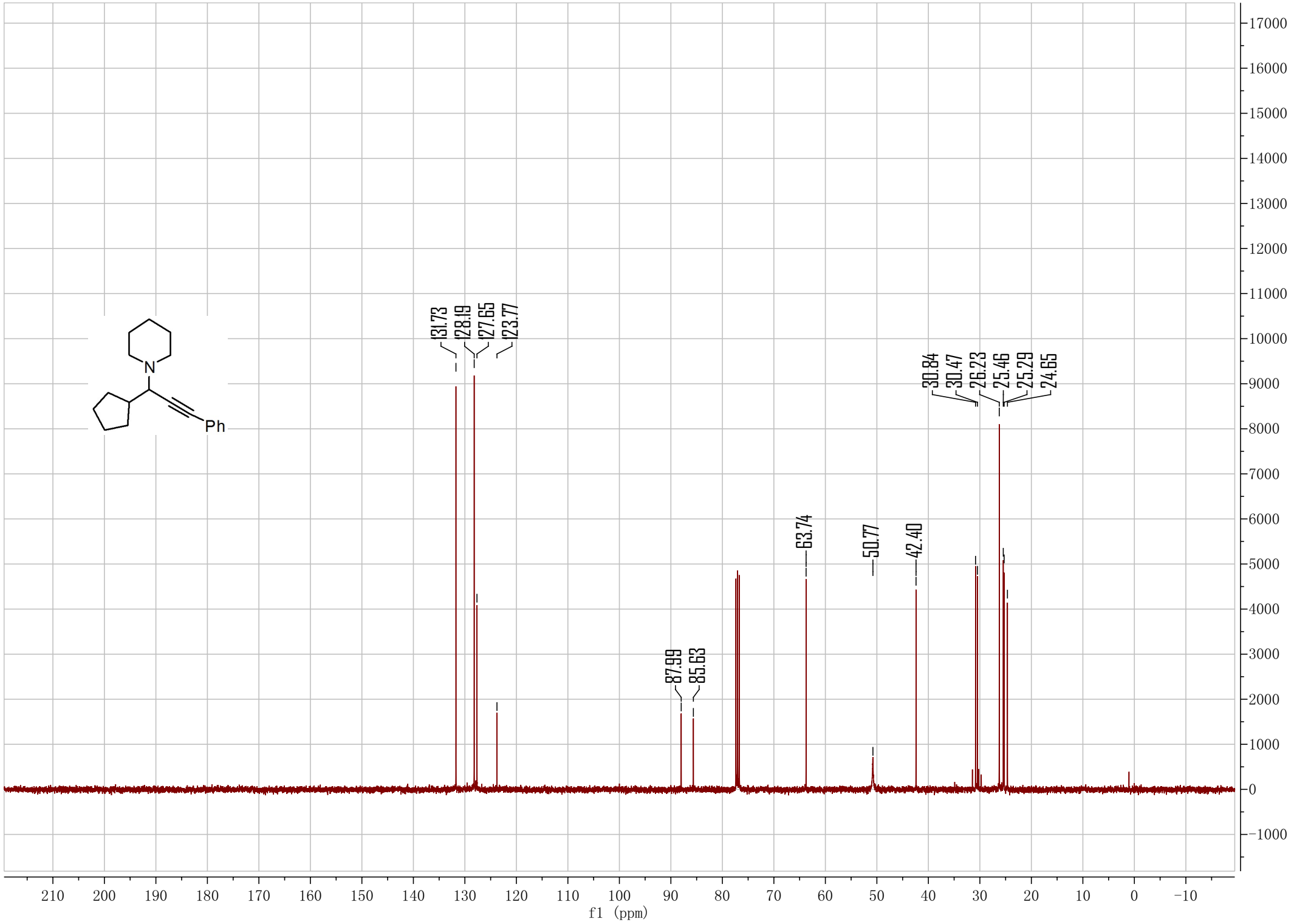
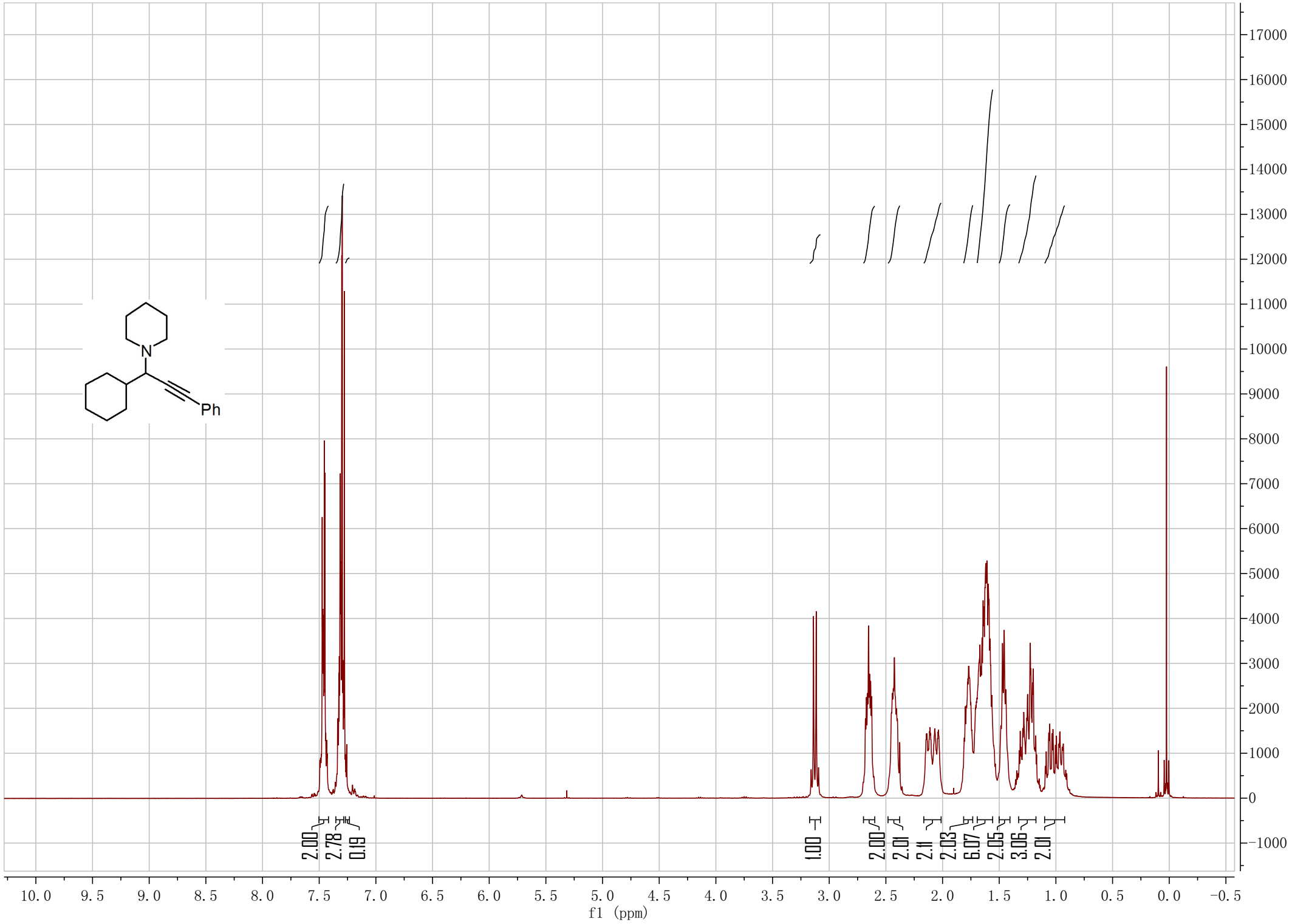
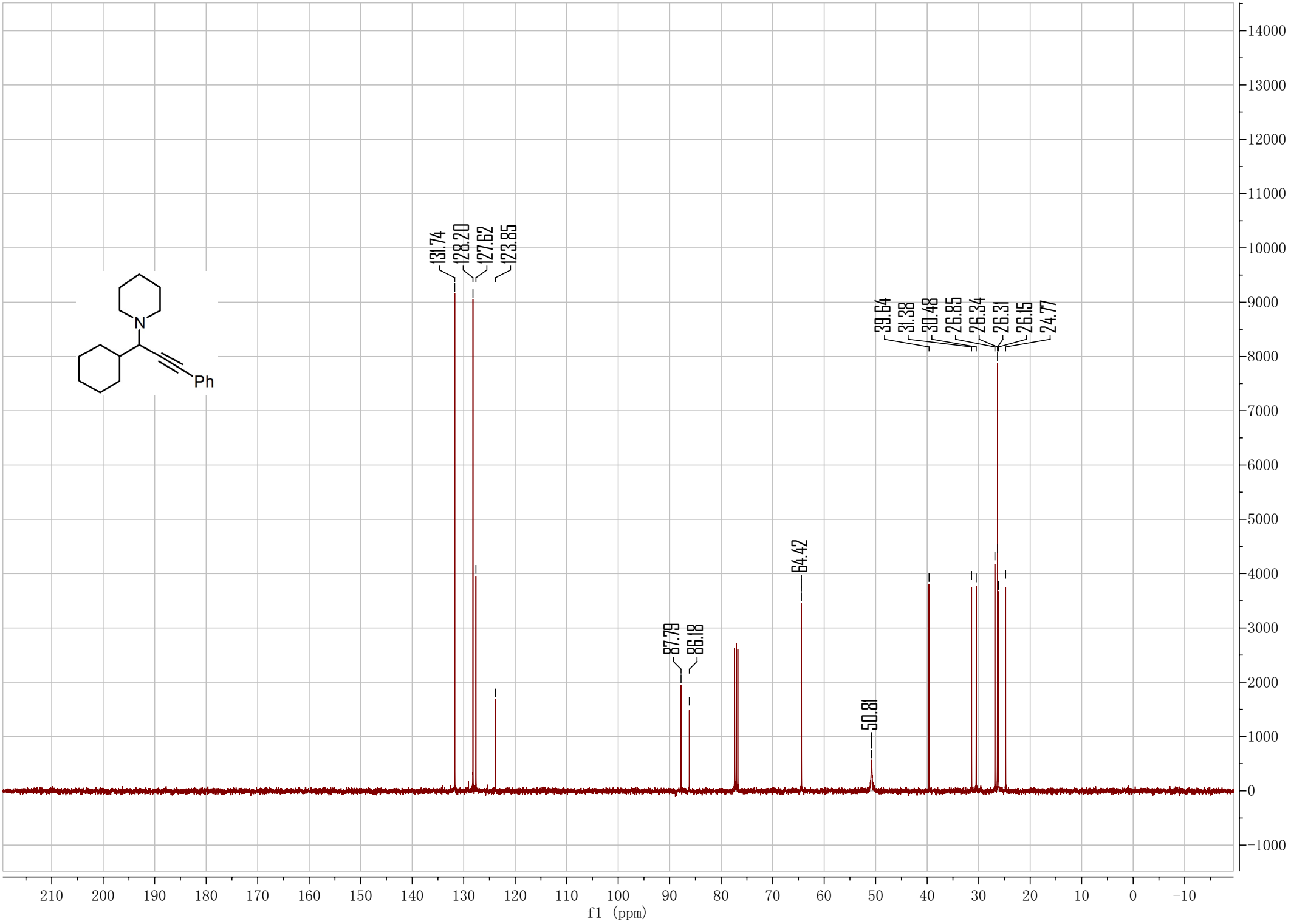
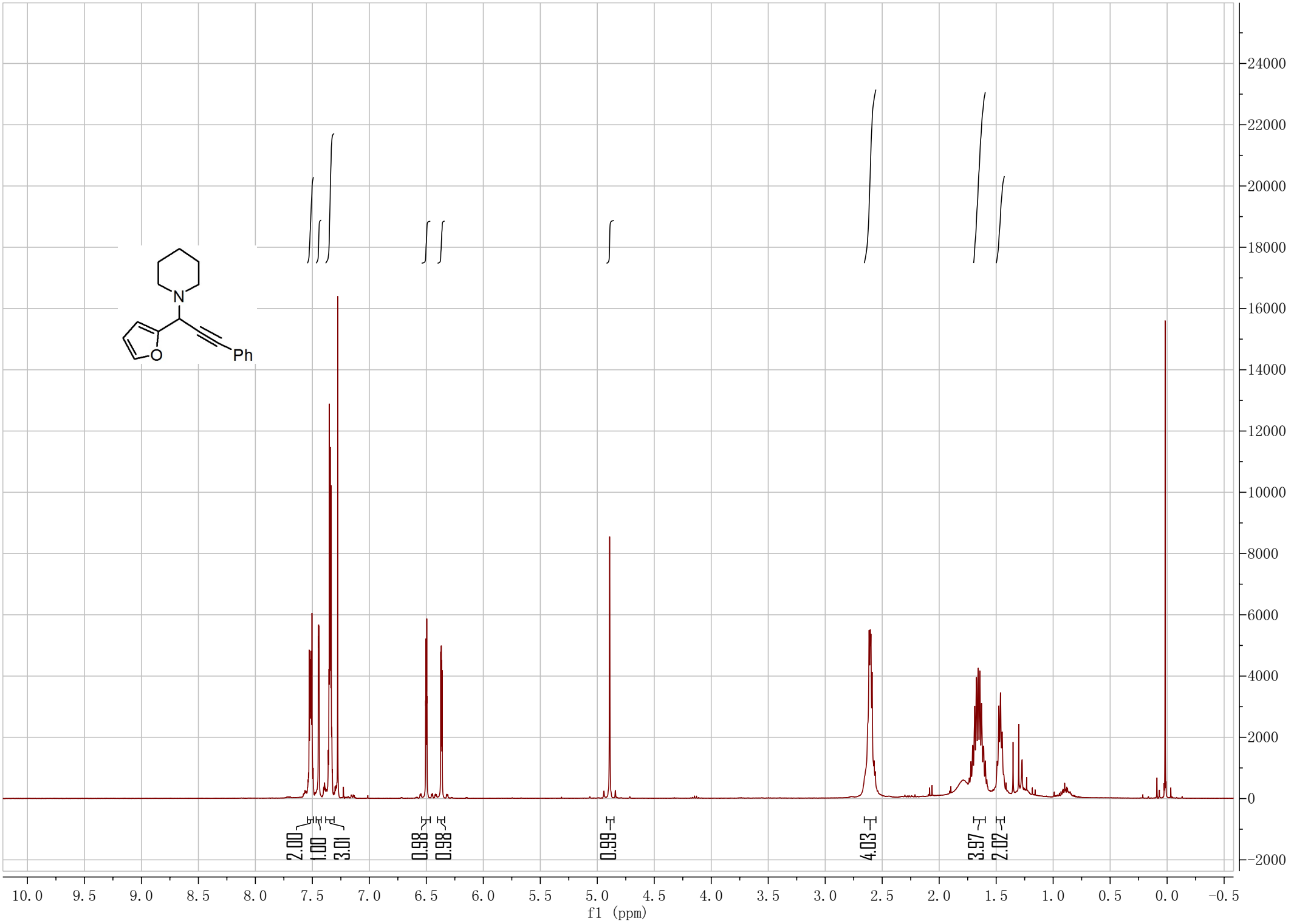
128.19
127.72
123.64
88.23
85.62
58.65
50.59
33.50
31.86
29.42
29.25
26.93
26.23
24.62
22.69
14.13
N
Ph
f1 (ppm)

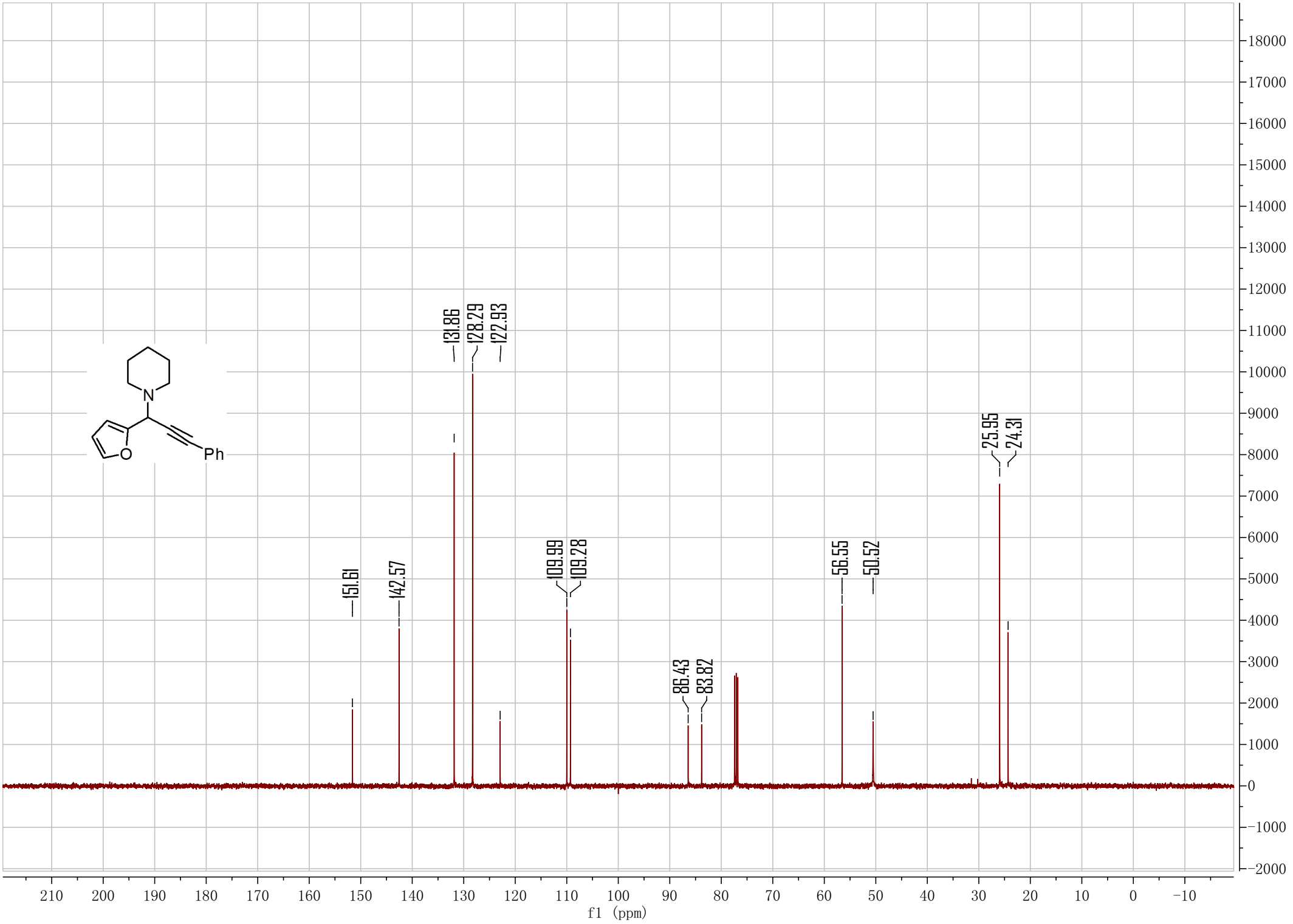

N
Ph
2.00
3.01
1.02
2.08
2.15
1.11
1.08
1.03
7.09
5.09
f1 (ppm)

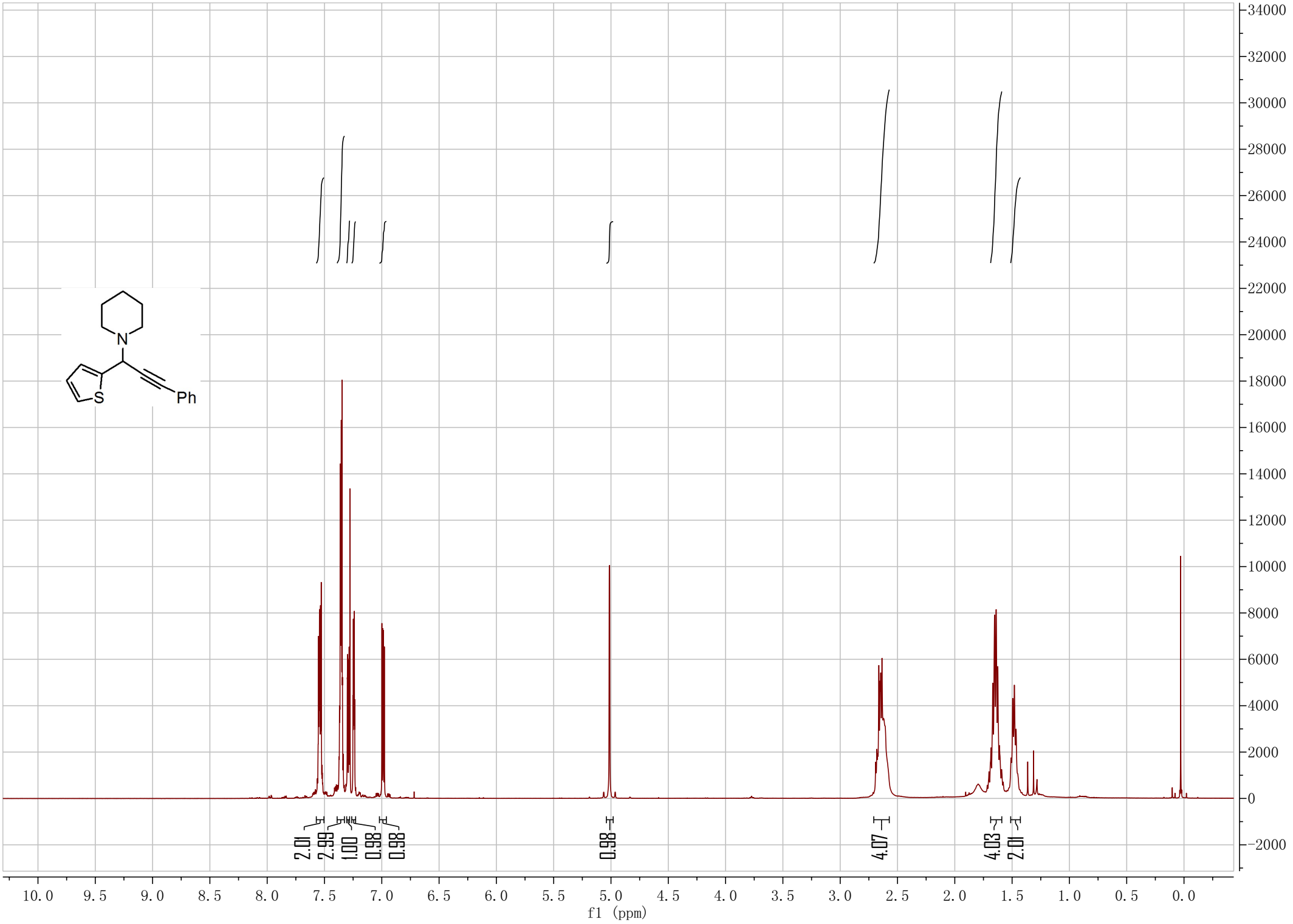

N
Ph
131.73
128.19
127.65
123.77
87.99
85.63
63.74
50.77
42.40
30.84
30.47
26.23
25.46
25.29
24.65
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


N
Ph
2.00
2.78
0.19
1.00
2.00
2.01
2.11
2.03
6.07
2.05
3.06
2.01
f1 (ppm)


131.74
128.20
127.62
123.85
87.79
86.18
64.42
50.81
39.64
31.38
30.48
26.85
26.34
26.31
26.15
24.77
N
Ph
f1 (ppm)


N
O
Ph
2.00
1.00
3.01
0.98
0.98
0.99
4.03
3.97
2.02
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


N
O
Ph
151.61
142.57
131.86
128.29
122.93
109.99
109.28
86.43
83.82
56.55
50.52
25.95
24.31
f1 (ppm)


N
S
Ph
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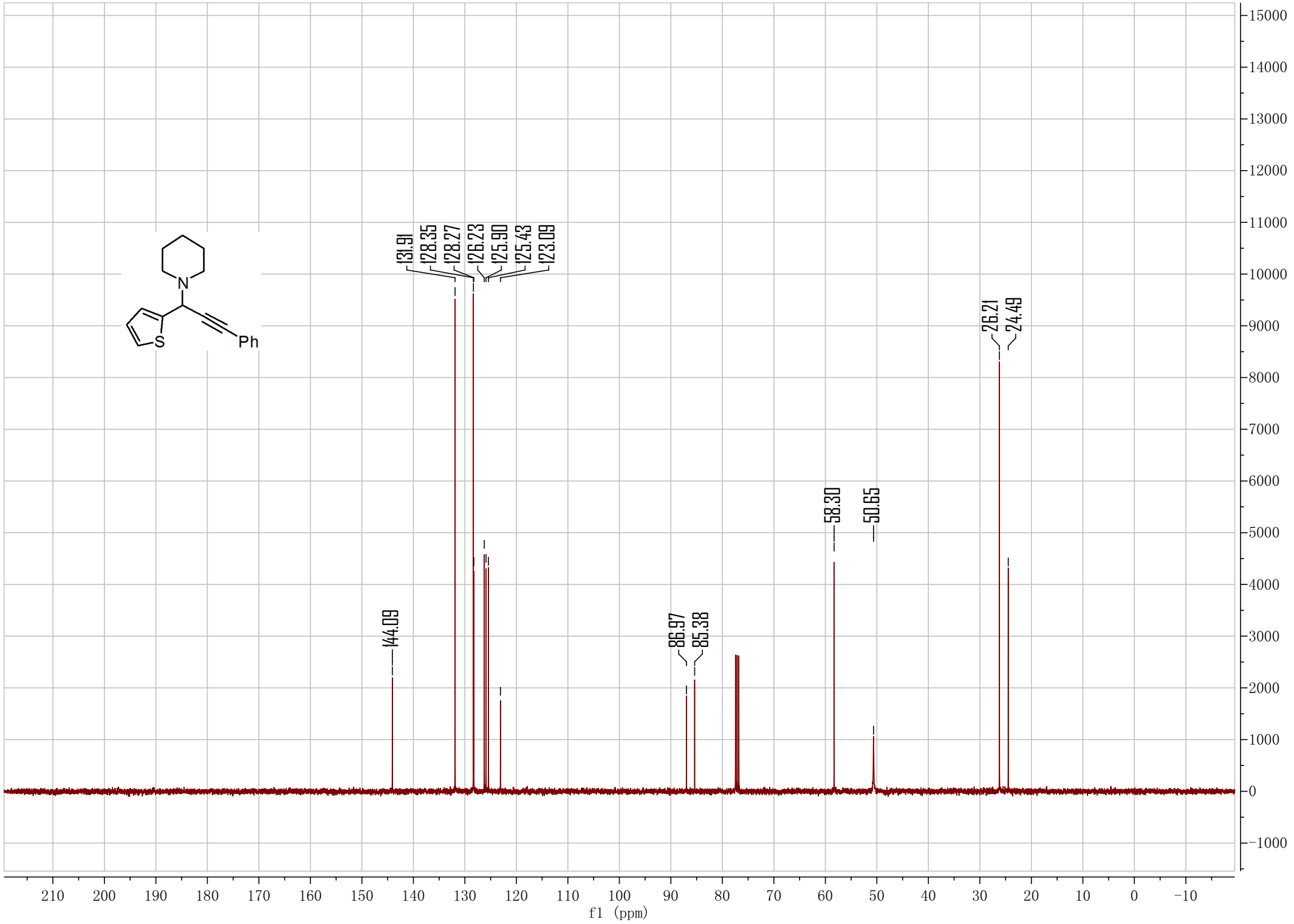
2.99
1.00
0.98
0.98
0.98
4.07
4.03
2.01
f1 (ppm)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128.27
126.23
125.90
125.43
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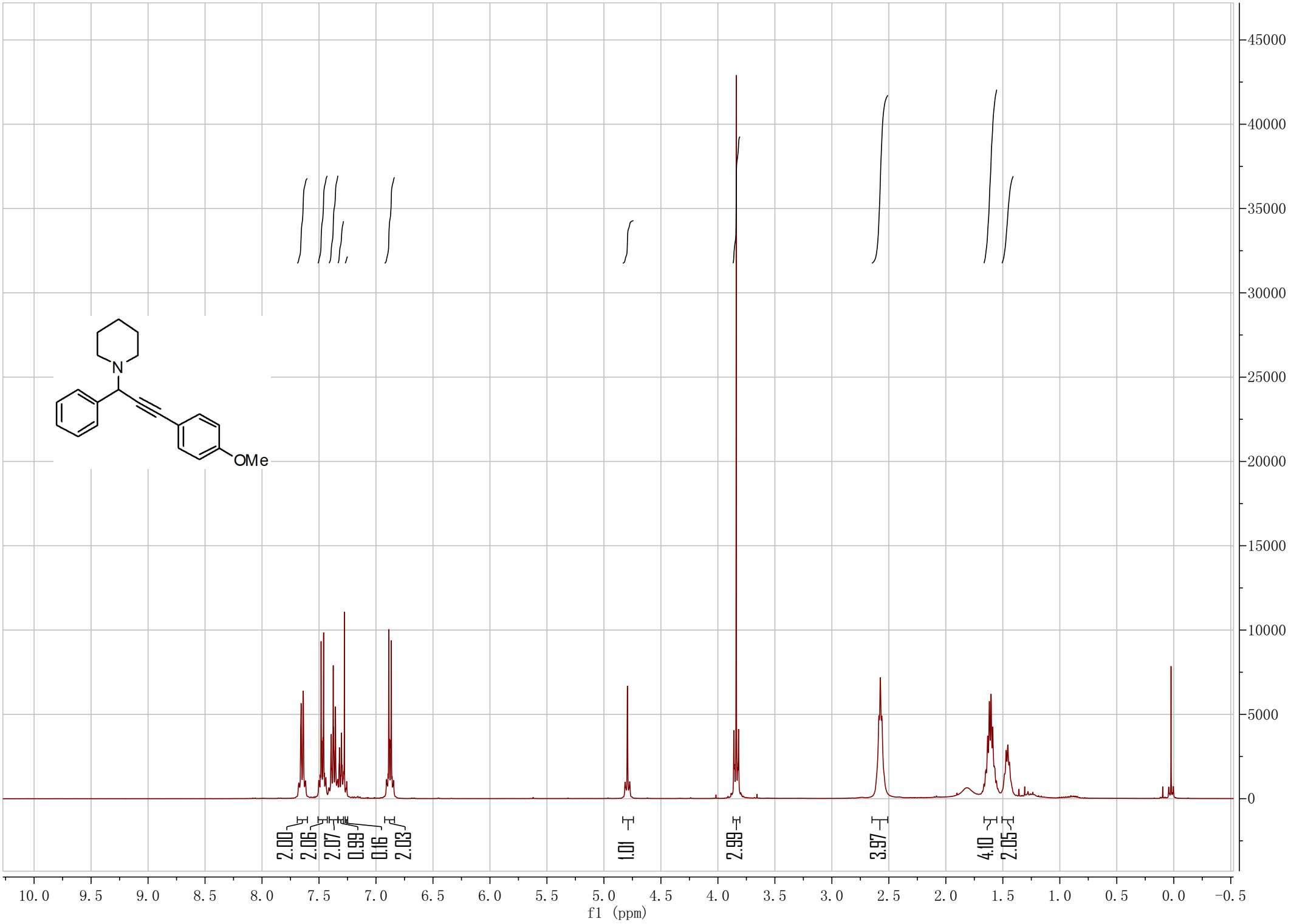
144.09
86.97
85.38
58.30
50.65
26.21
24.49
N
S
Ph
f1 (ppm)


N
OMe
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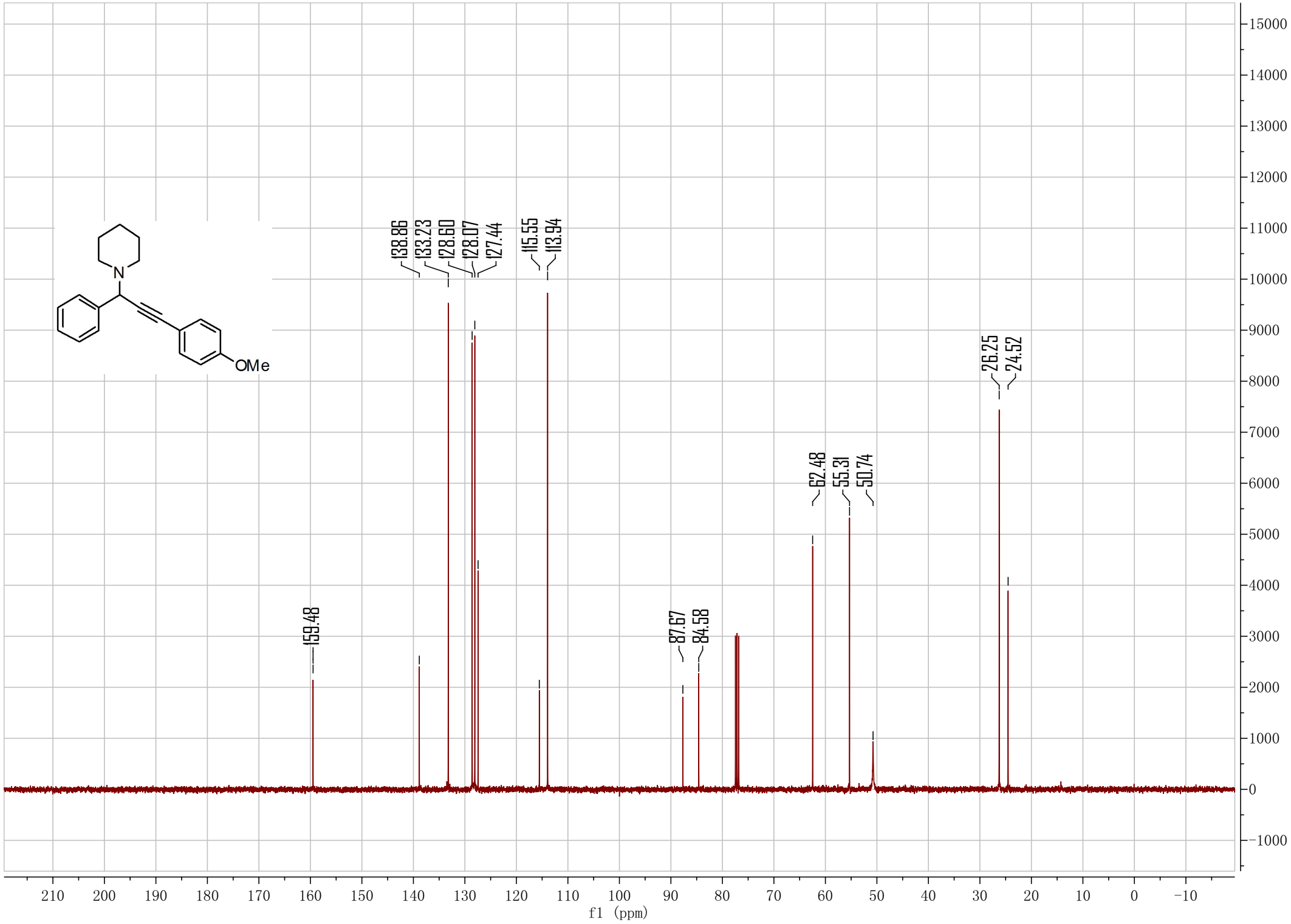
2.06
2.07
0.99
0.16
2.03
1.01
2.99
3.97
4.10
2.05
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


N
OMe
159.48
138.86
133.23
128.60
128.07
127.44
115.55
113.94
87.67
84.58
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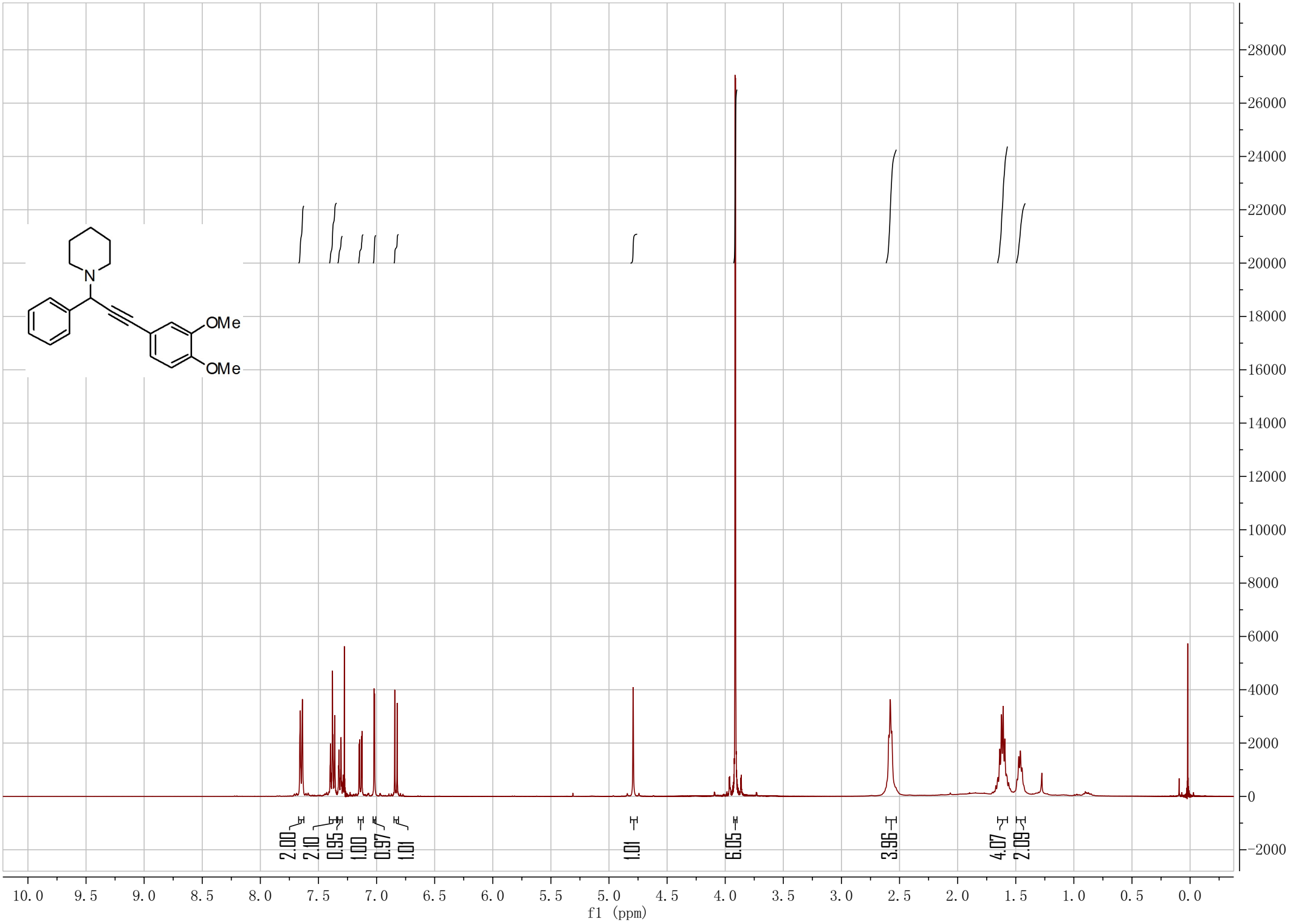
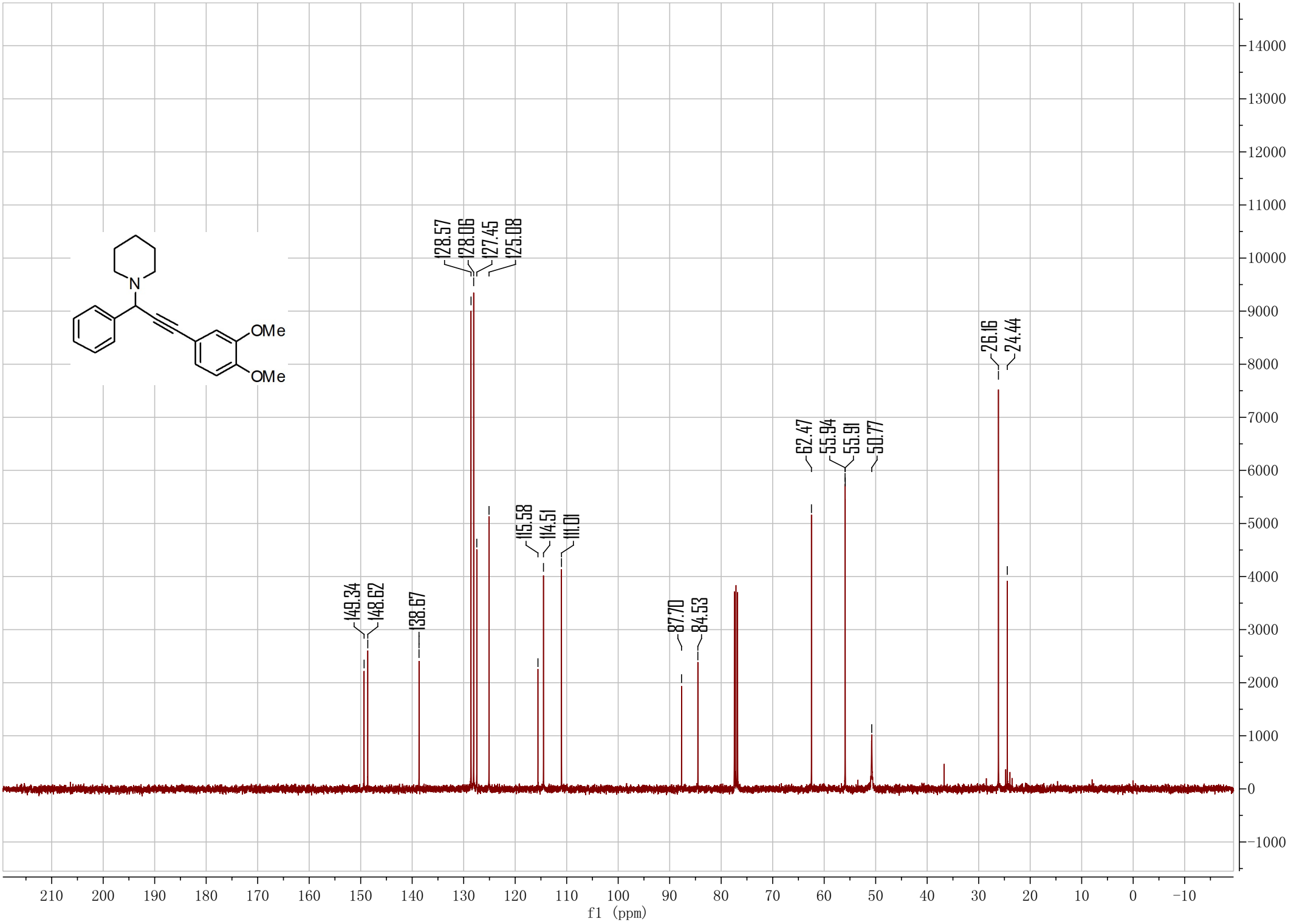
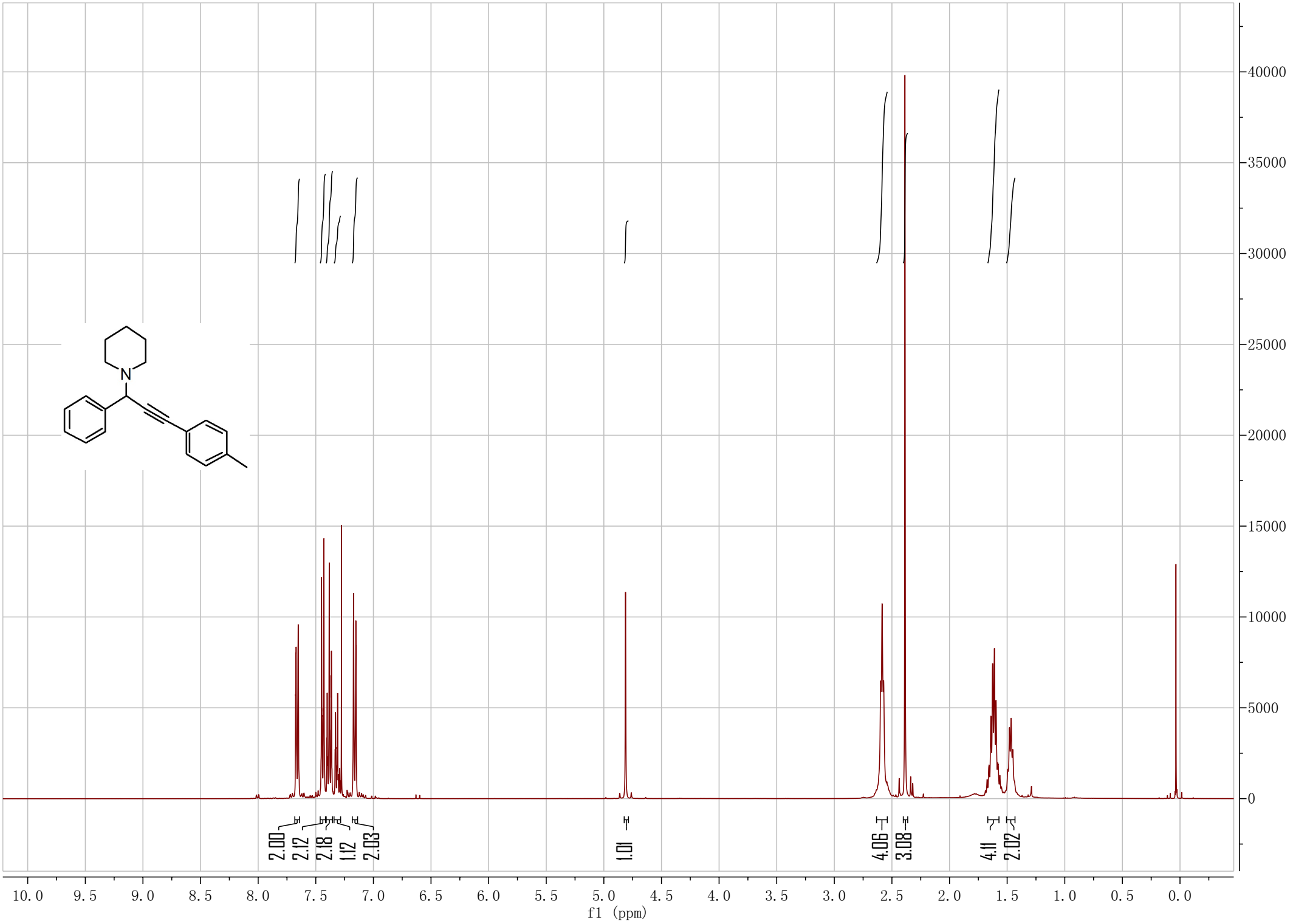
55.31
50.74
26.25
24.52
f1 (ppm)


N
OMe
OMe
2.00
2.10
0.95
1.00
0.97
1.01
1.01
6.05
3.96
4.07
2.09
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


N
OMe
OMe
128.57
128.06
127.45
125.08
149.34
148.62
138.67
115.58
114.51
111.01
87.70
84.53
62.47
55.94
55.91
50.77
26.16
24.44
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


N
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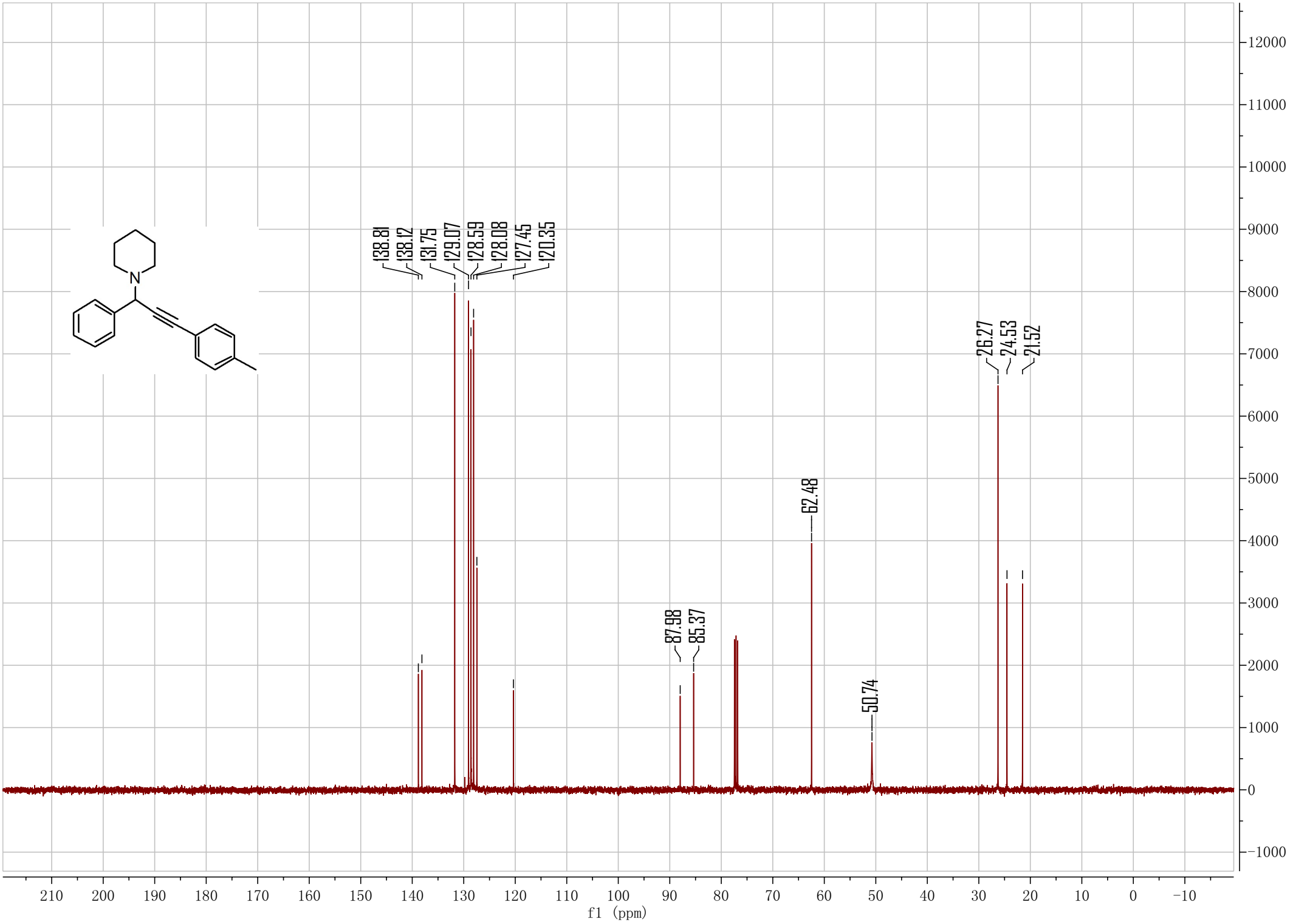
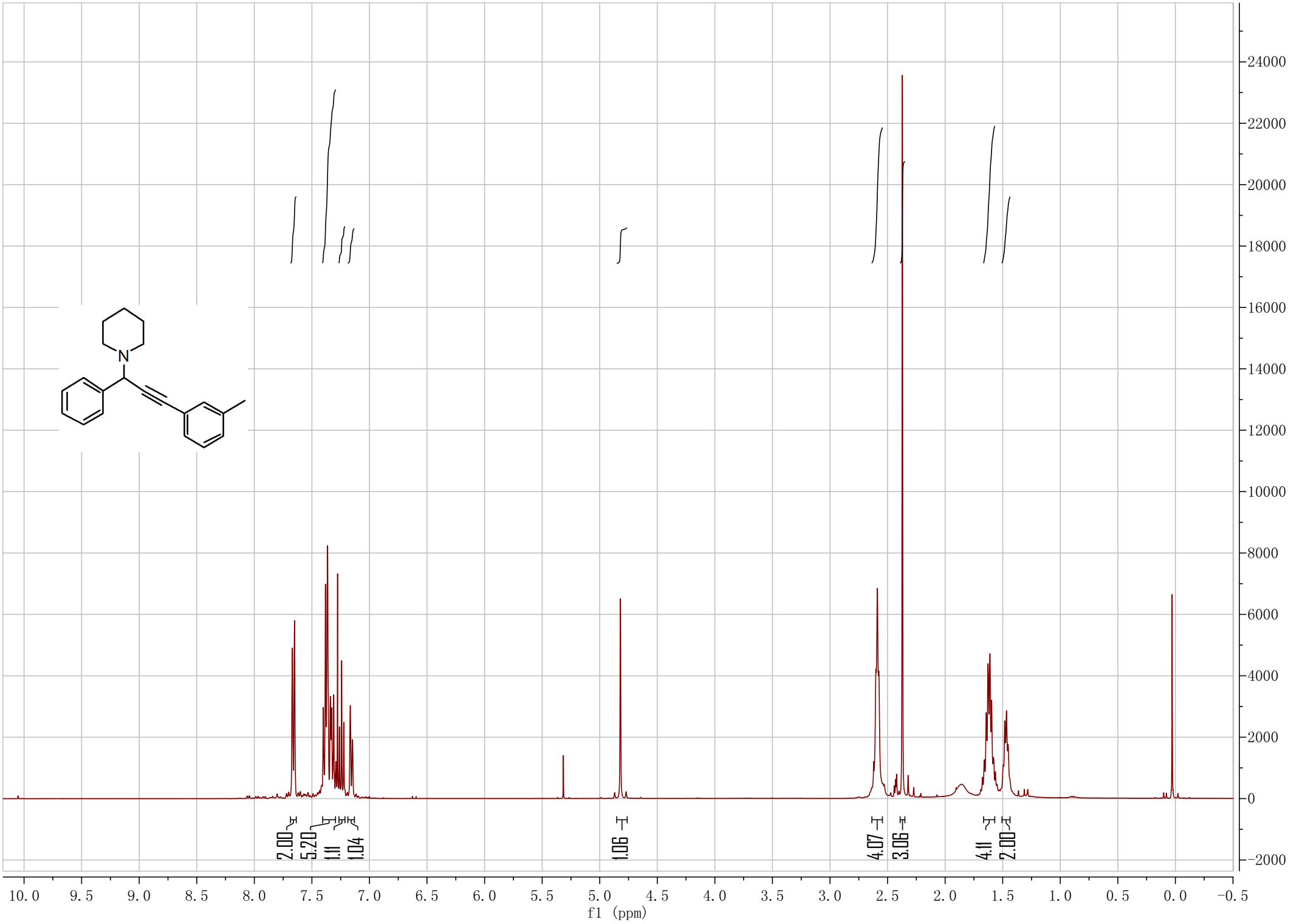
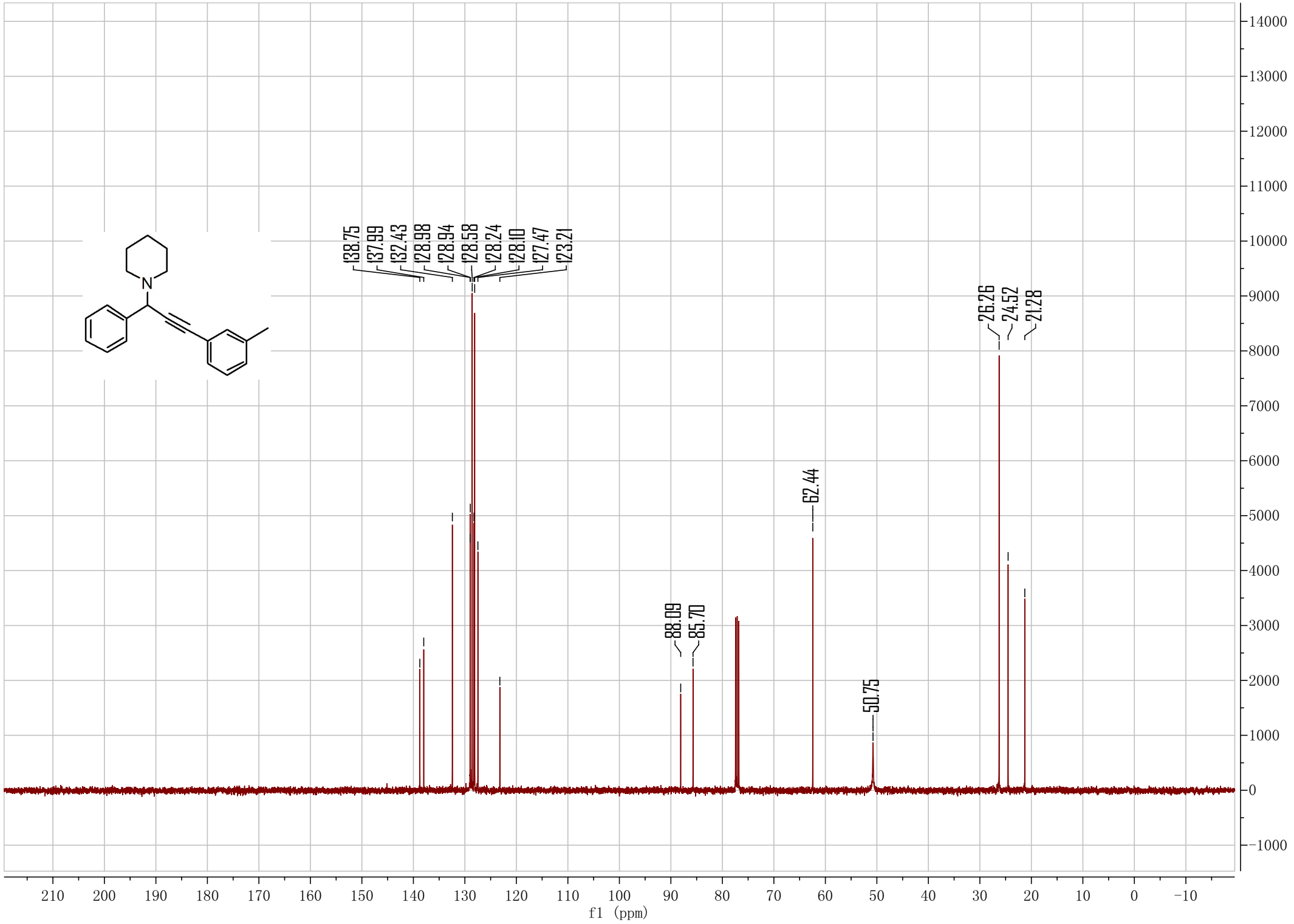
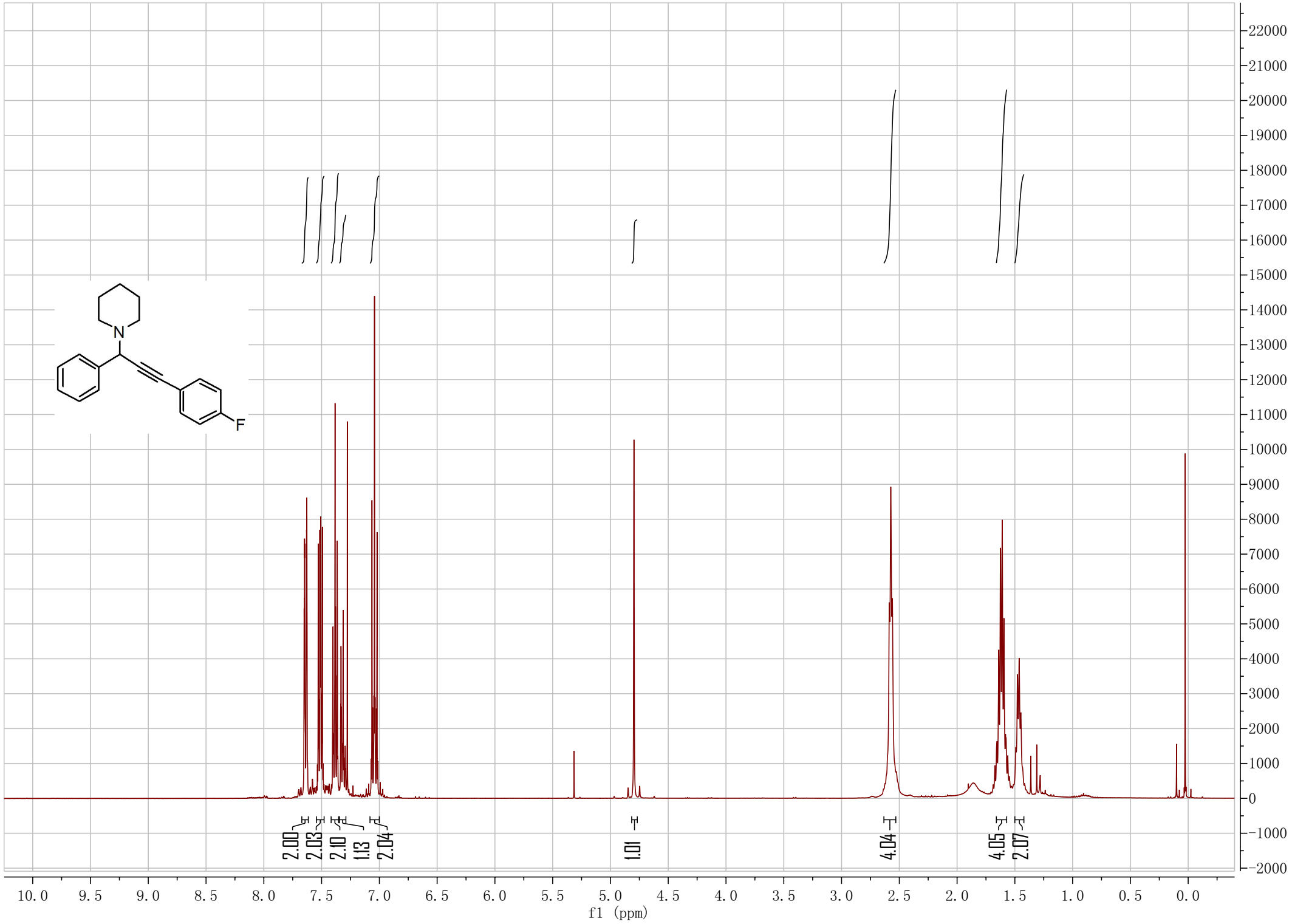
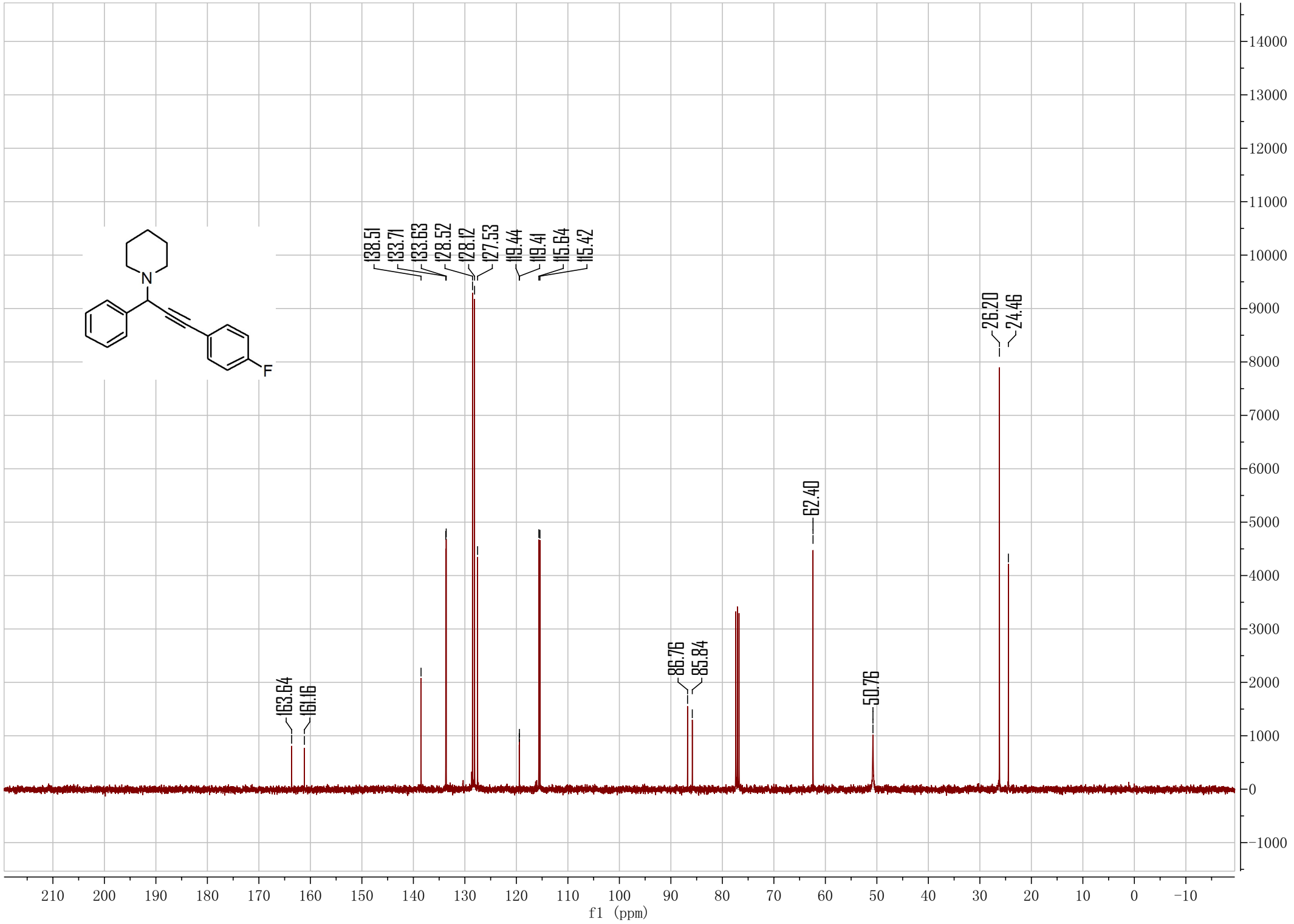
2.12
2.18
1.12
2.03
1.01
4.06
3.08
4.11
2.02
40000
35000
30000
25000
20000
15000
10000
5000
0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

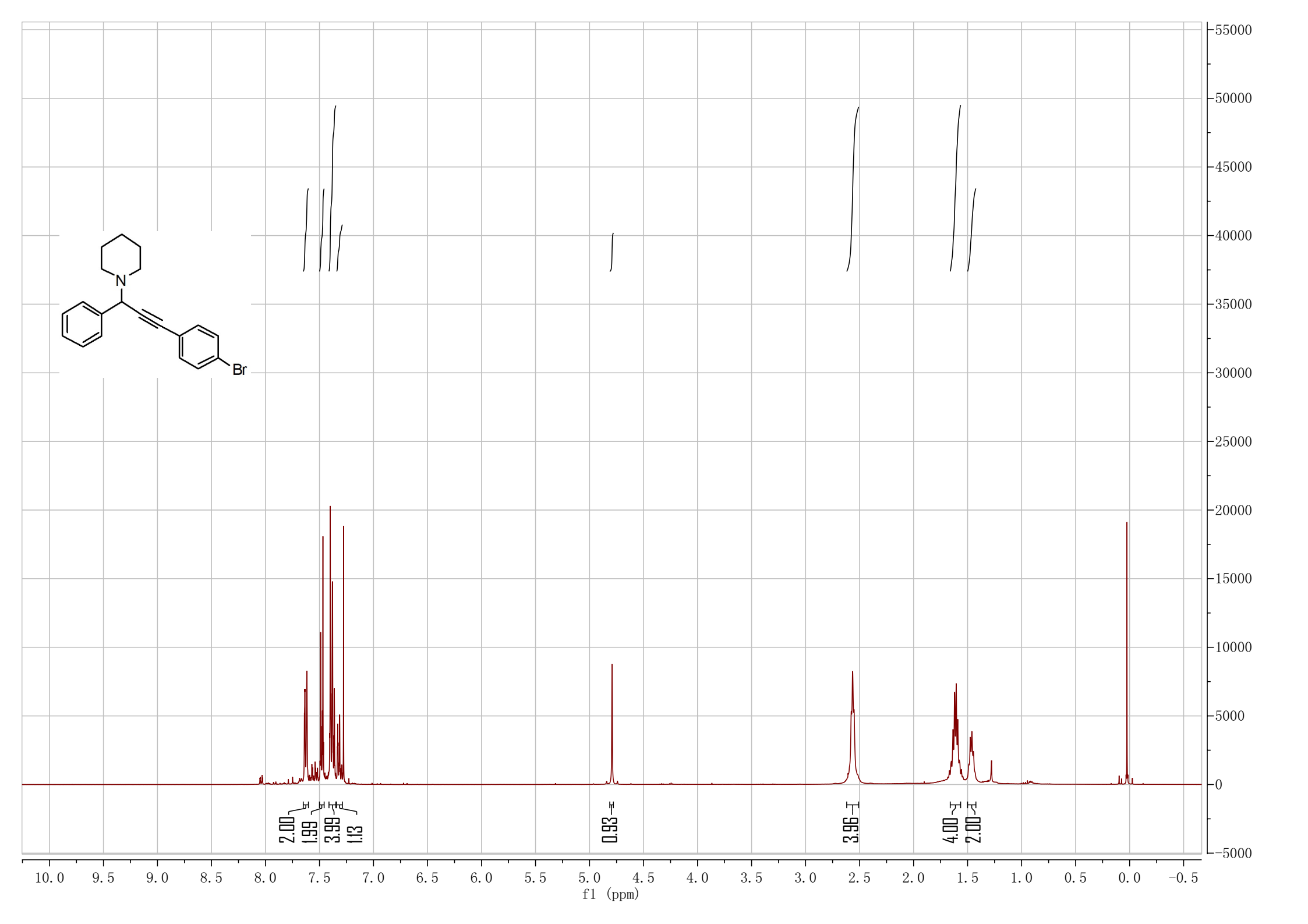

138.81
138.12
131.75
129.07
128.59
128.08
127.45
120.35
87.98
85.37
62.48
50.74
26.27
24.53
21.52
N
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

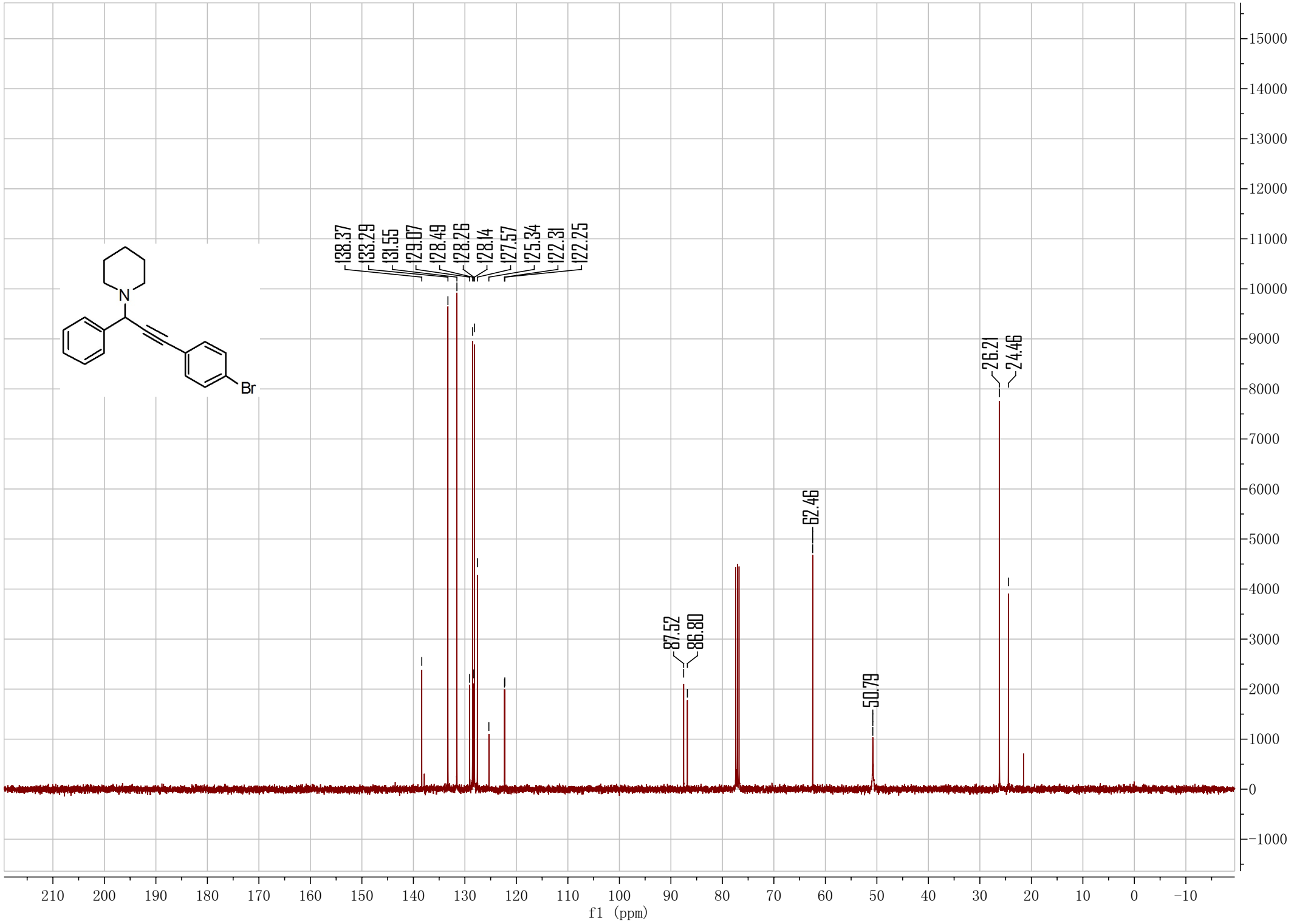

N
2.00
5.20
1.11
1.04
1.06
4.07
3.06
4.11
2.00
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

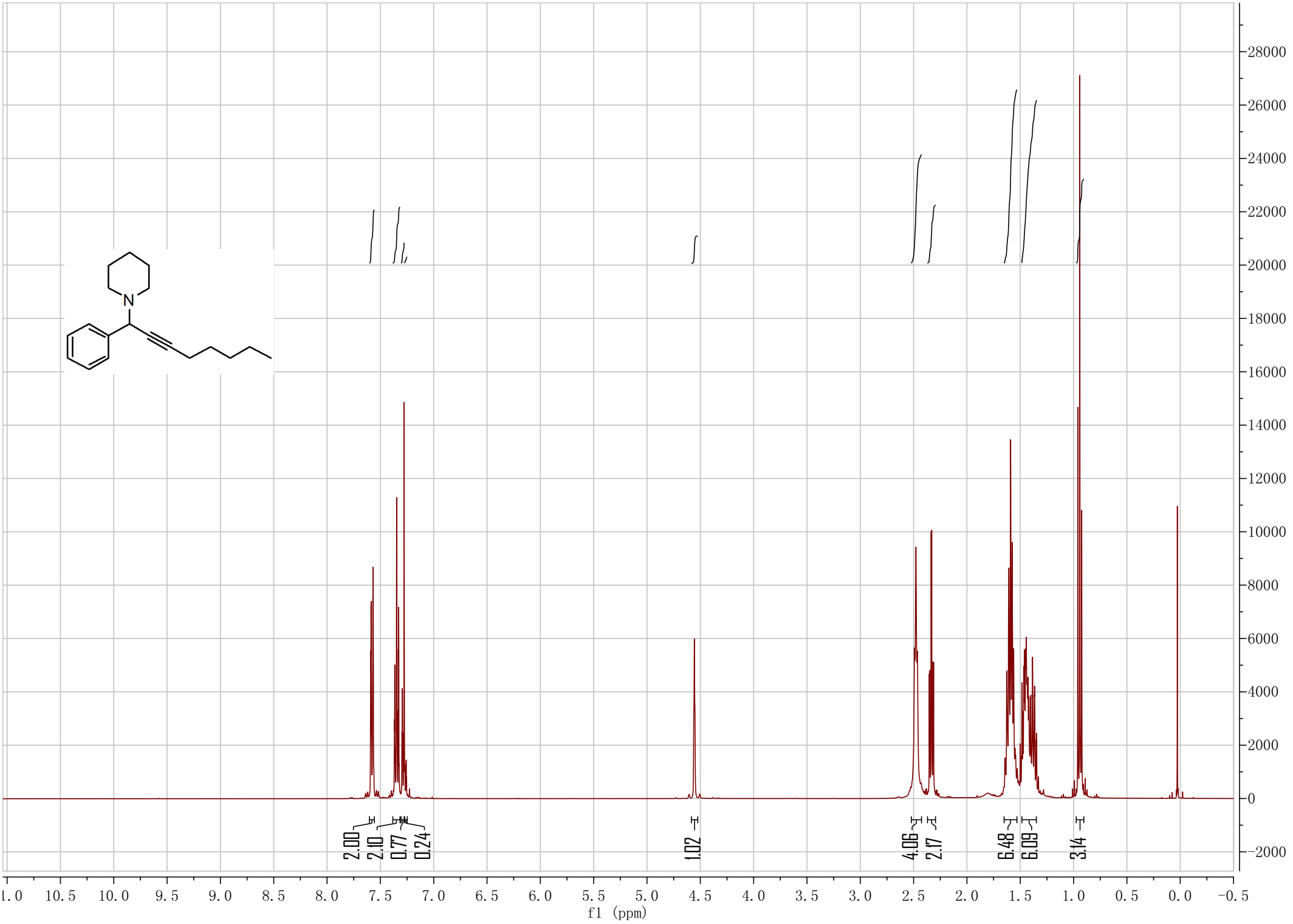

138.75
137.99
132.43
128.98
128.94
128.58
128.24
128.10
127.47
123.21
88.09
85.70
62.44
50.75
26.26
24.52
21.28
f1 (ppm)

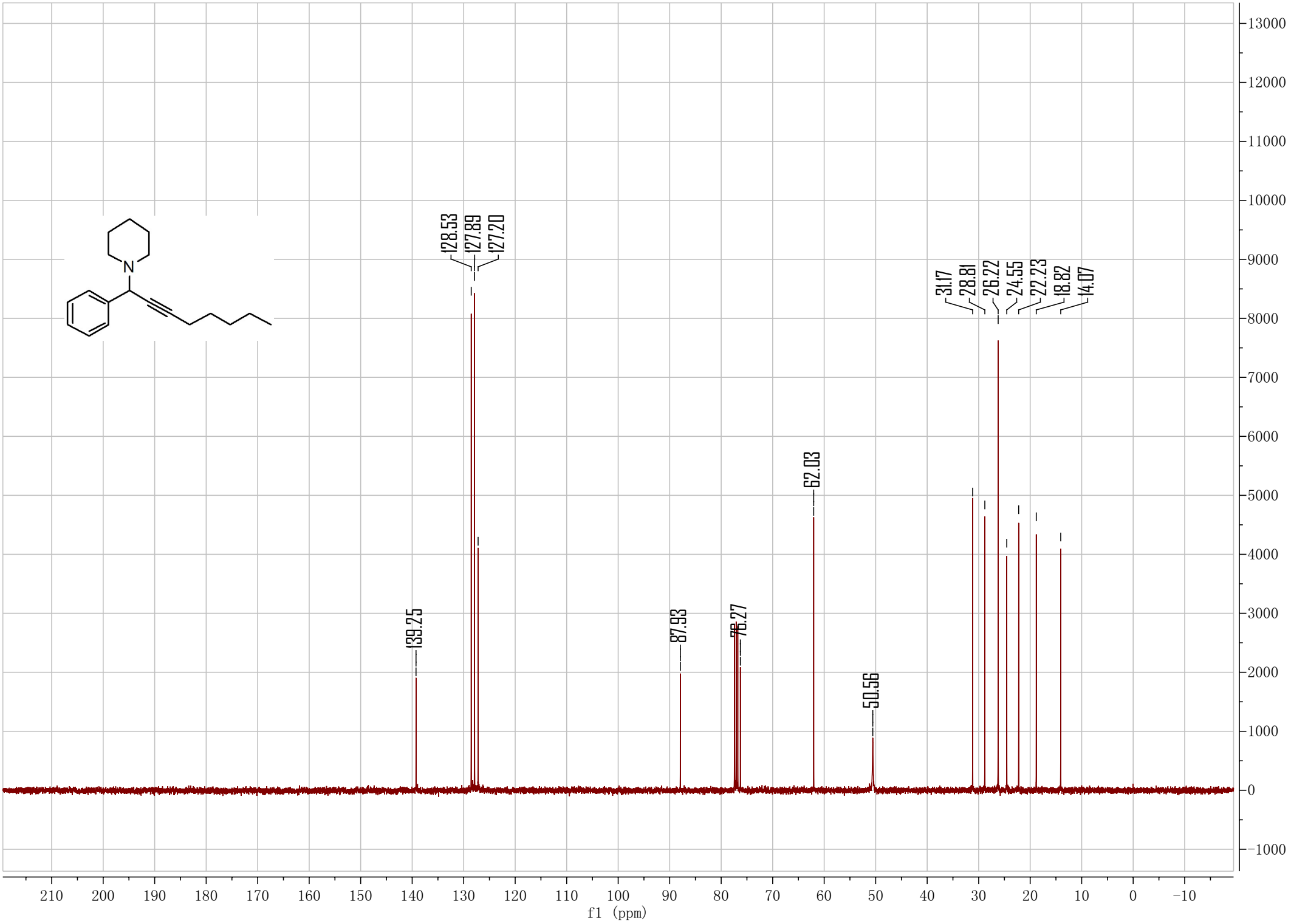

N
F
2.00
2.03
2.10
1.13
2.04
1.01
4.04
4.05
2.07
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

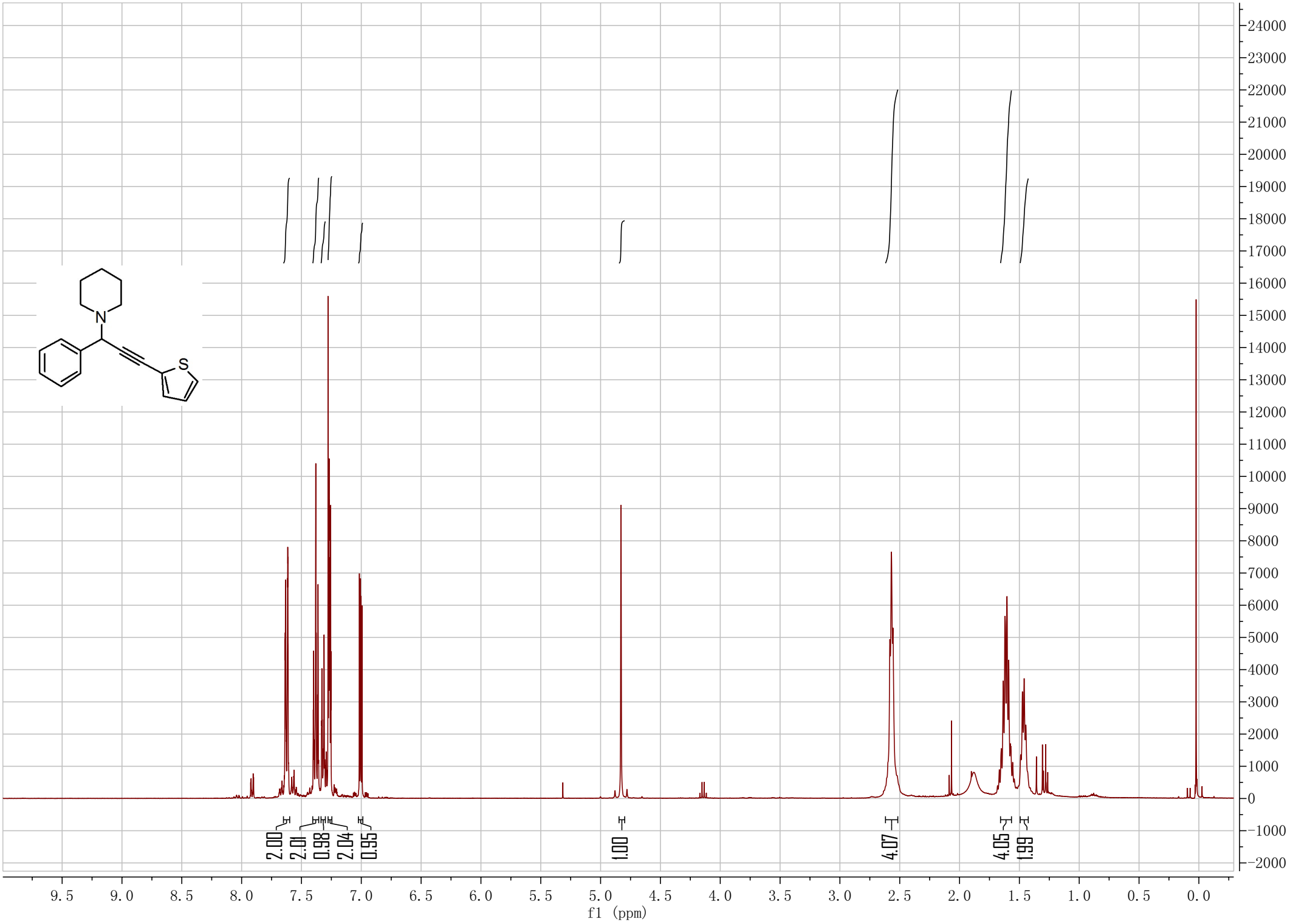

N
F
138.51
133.71
133.63
128.52
128.12
127.53
119.44
119.41
115.64
115.42
163.64
161.16
86.76
85.84
62.40
50.76
26.20
24.46
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

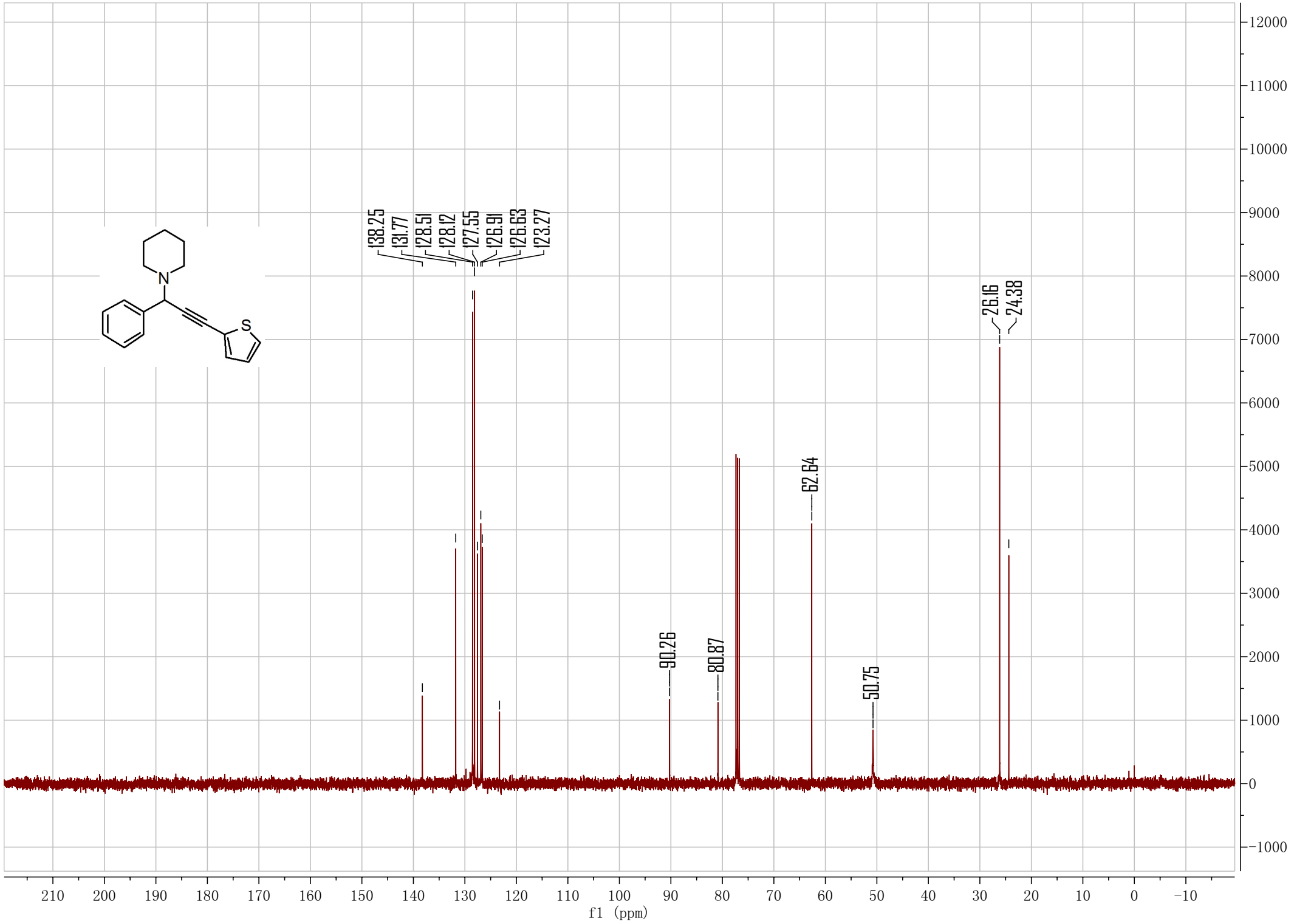

N
Br
2.00
1.99
3.99
1.13
0.93
3.96
4.00
2.00
f1 (ppm)

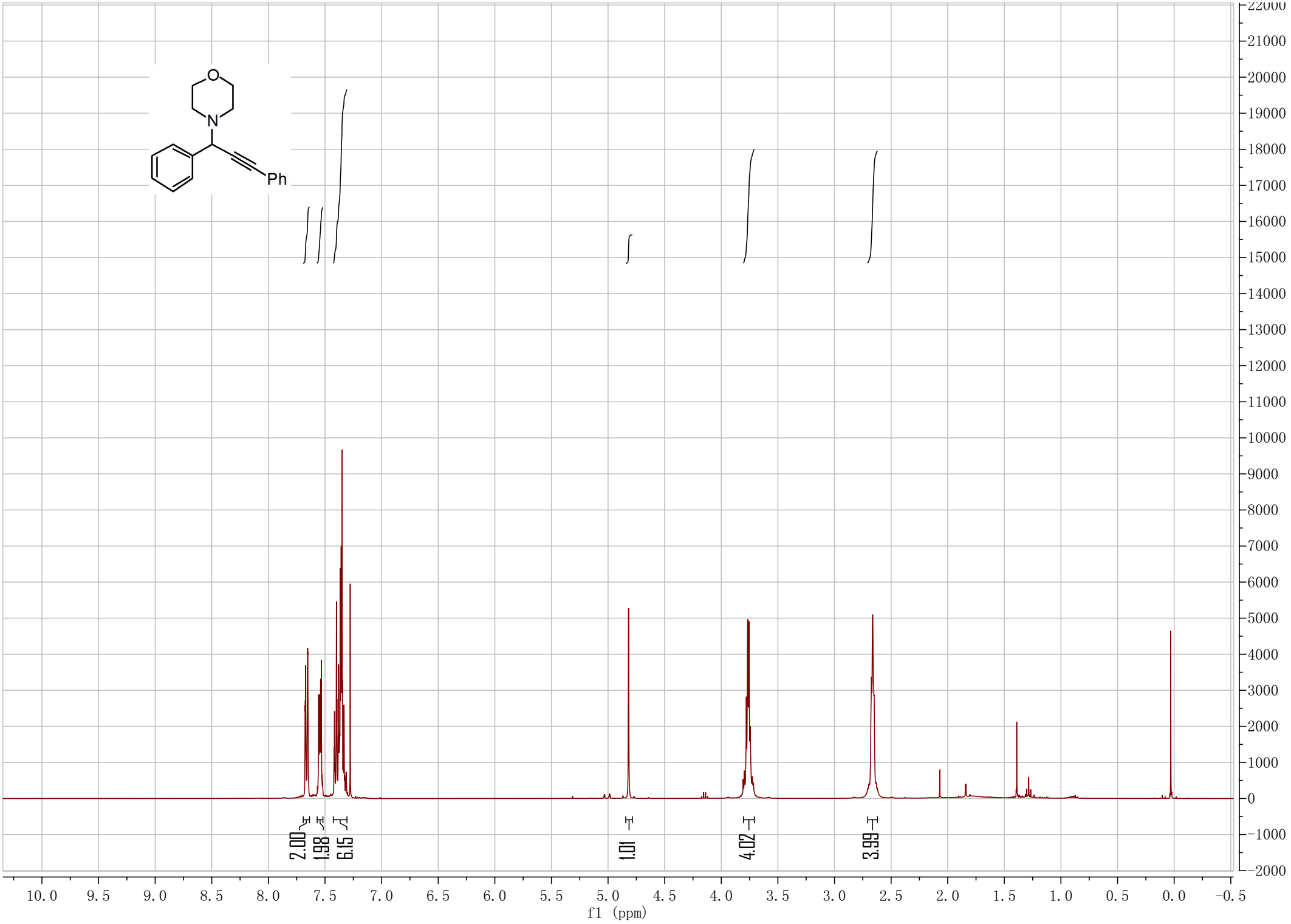

138.37
133.29
131.55
129.07
128.49
128.26
128.14
127.57
125.34
122.31
122.25
87.52
86.80
62.46
50.79
26.21
24.46
N
Br
f1 (ppm)

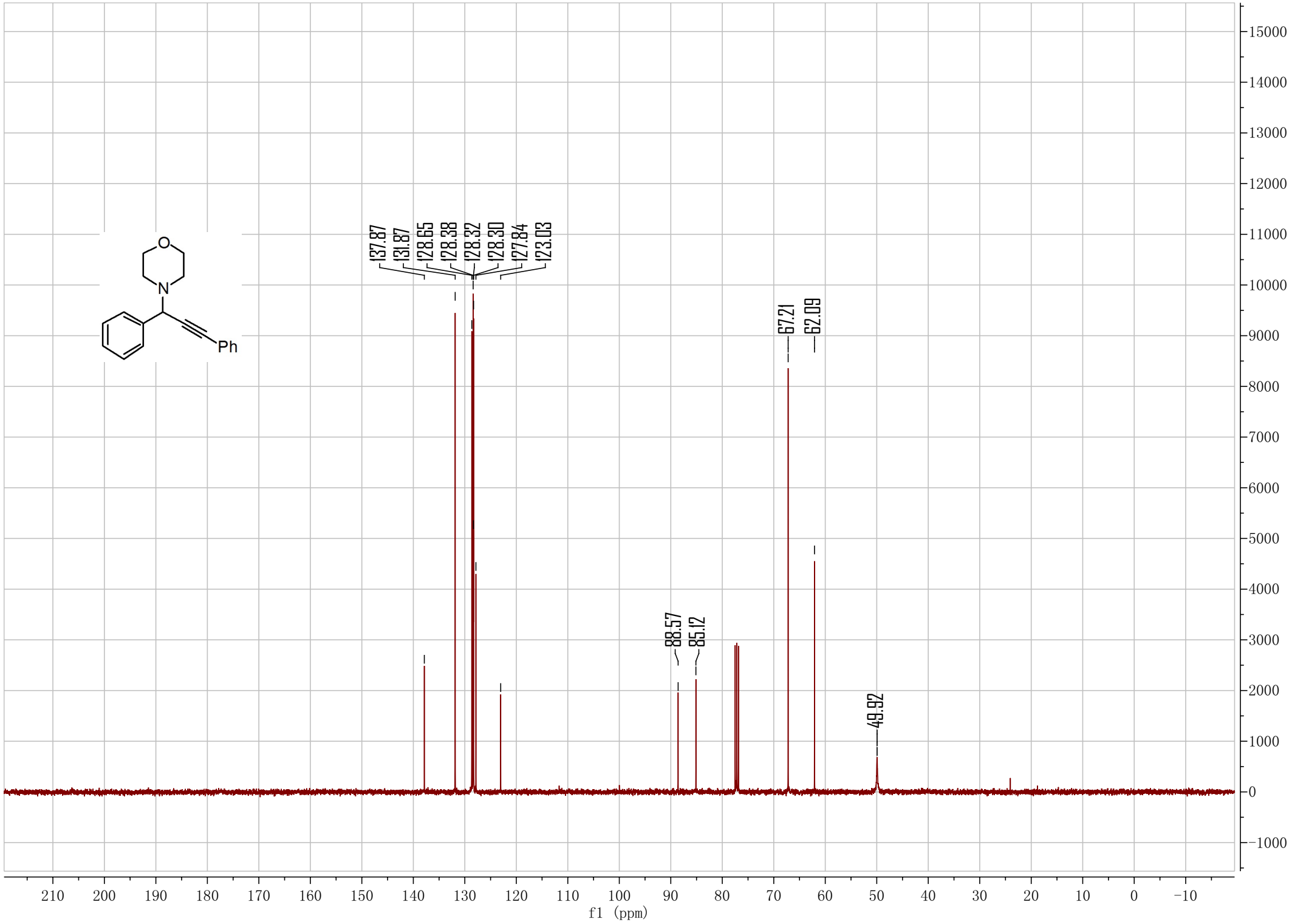

N
2.00
2.10
0.77
0.24
1.02
4.06
2.17
6.48
6.09
3.14
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

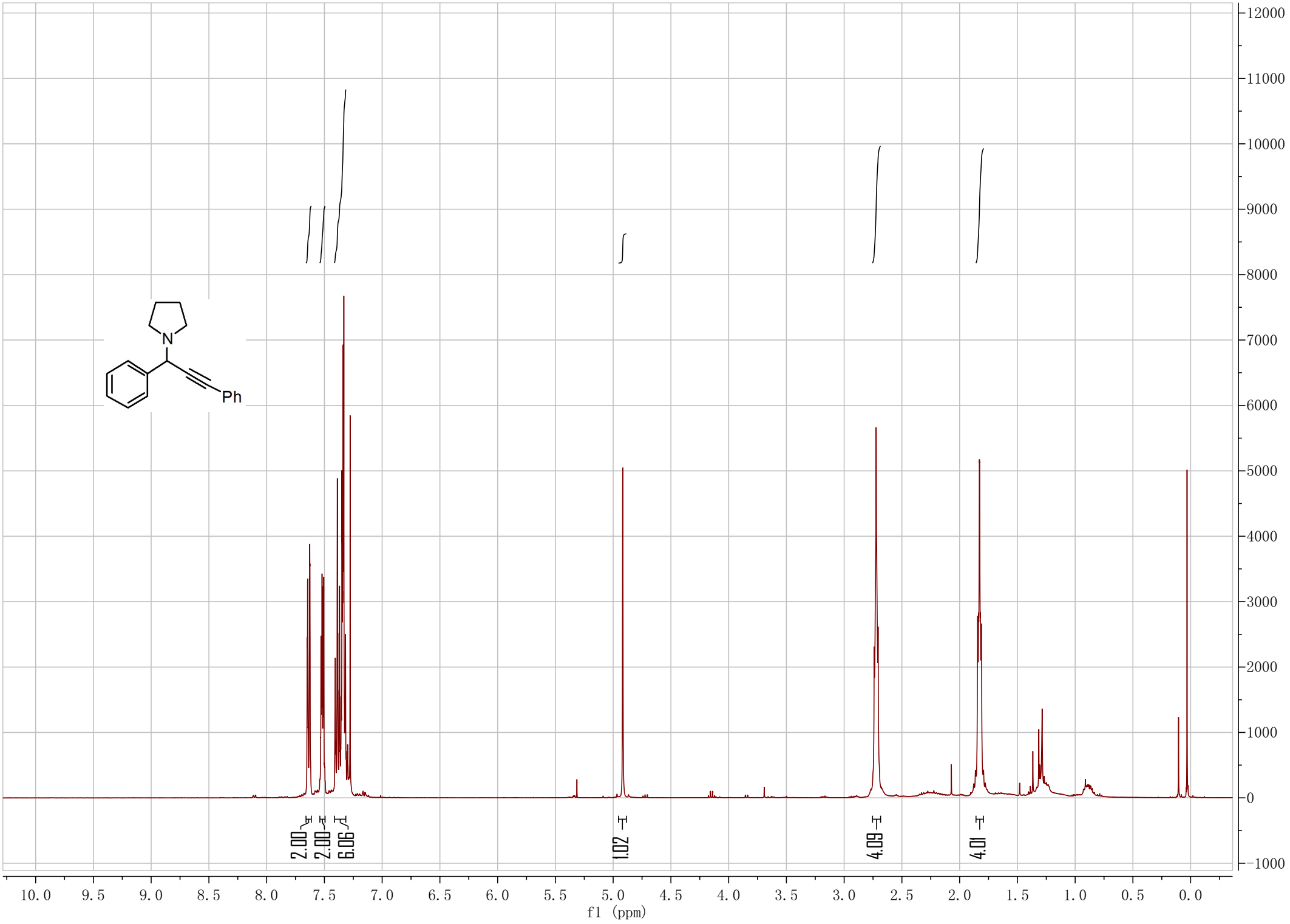

128.53
127.89
127.20
139.25
87.93
76.27
62.03
50.56
31.17
28.81
26.22
24.55
22.23
18.82
14.07
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

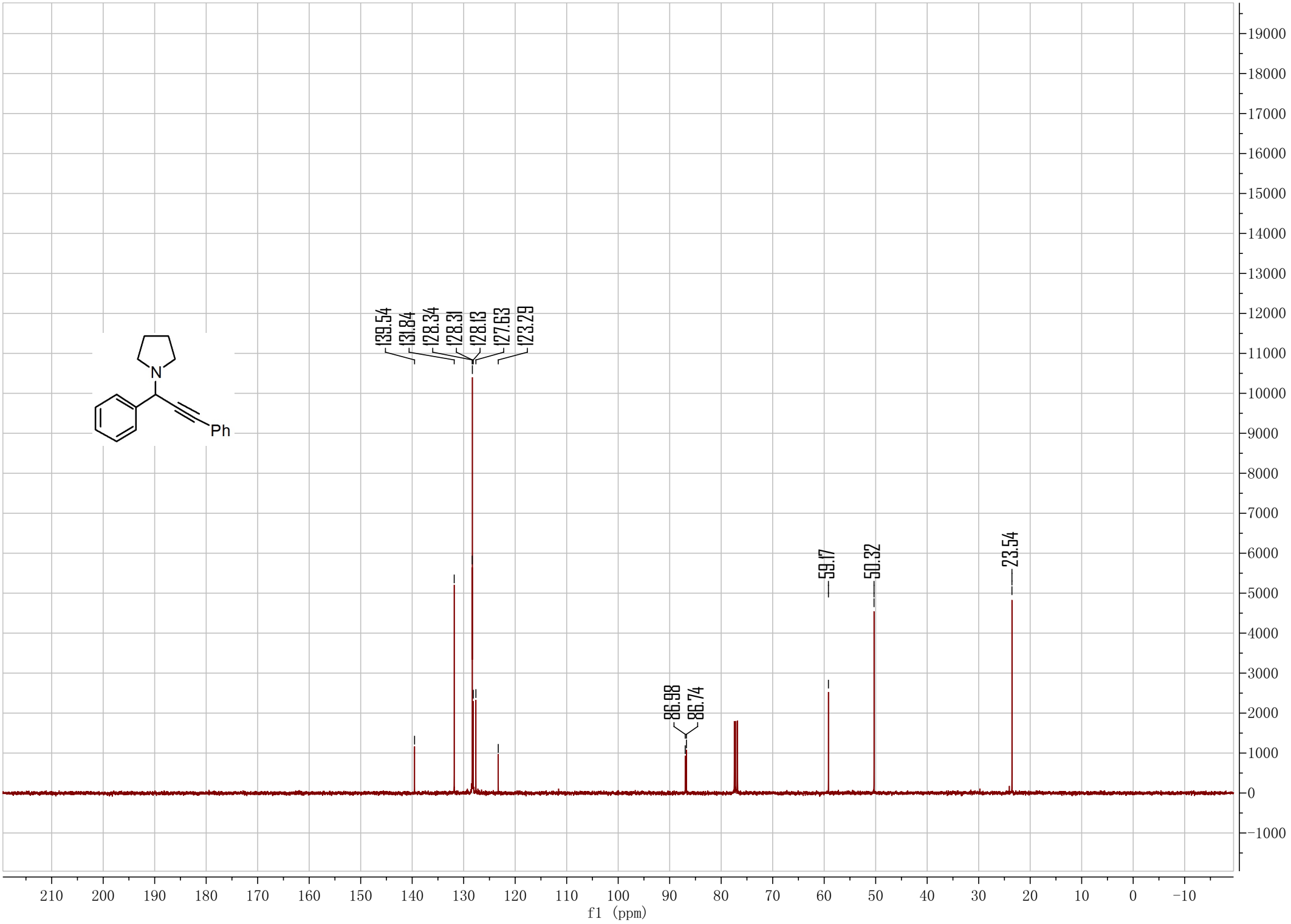

N
S
2.00
2.01
0.98
2.04
0.95
1.00
4.07
4.05
1.99
f1 (ppm)

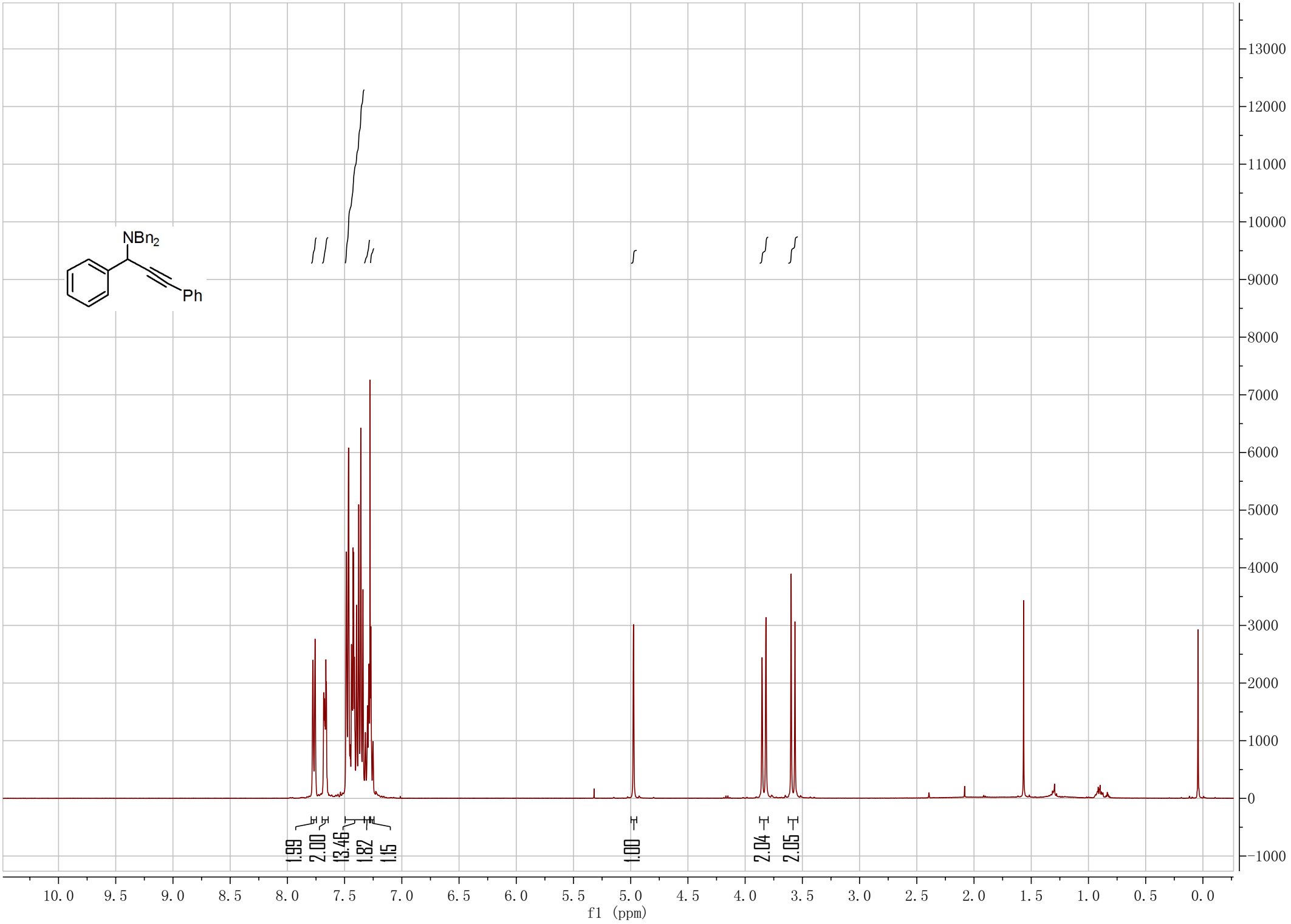

138.25
131.77
128.51
128.12
127.55
126.91
126.63
123.27
90.26
80.87
62.64
50.75
26.16
24.38
N
S
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

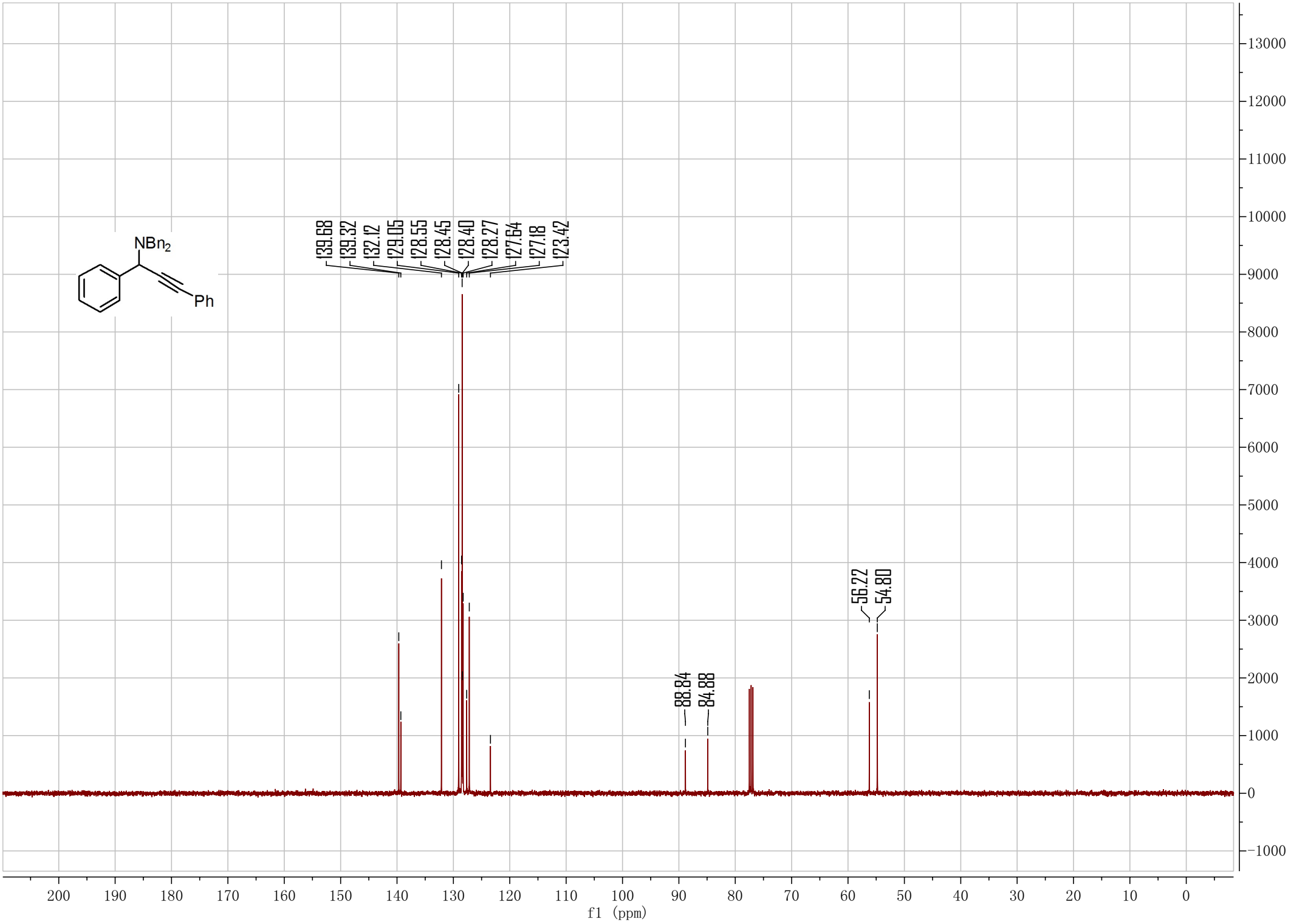

O
N
Ph
2.00
1.98
6.15
1.01
4.02
3.99
f1 (ppm)


137.87
131.87
128.65
128.38
128.32
128.30
127.84
123.03
67.21
62.09
88.57
85.12
49.92
O
N
Ph
f1 (ppm)


N
Ph
2.00
2.00
6.06
1.02
4.09
4.01
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


N
Ph
139.54
131.84
128.34
128.31
128.13
127.63
123.29
86.98
86.74
59.17
50.32
23.54
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


NBn2
Ph
1.99
2.00
13.46
1.82
1.15
1.00
2.04
2.05
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


NBn2
Ph
139.68
139.32
132.12
129.05
128.55
128.45
128.40
128.27
127.64
127.18
123.42
88.84
84.88
56.22
54.80
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)