

Supporting Information

C-H insertions in oxidative gold catalysis: Synthesis of polycyclic dihydropyran-3-ones via a relay strategy

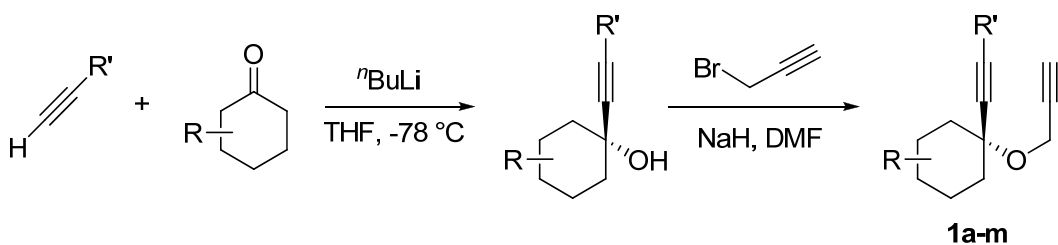
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General. Ethyl acetate (ACS grade), hexanes (ACS grade), dichloromethane (ACS grade) were purchased from Fisher Scientific and used without further purification. ACS grade 1,2-dichloroethane were purchased from Acros Organics and used directly. Commercially available reagents were used without further purification. Reactions were monitored by thin layer chromatography (TLC) using Silicycle precoated silica gel plates. Flash column chromatography was performed over Silicycle silica gel (230-400 mesh). ^1H NMR and ^{13}C NMR spectra were recorded on a Varian 400 MHz spectrometer, a Varian 500 MHz Unity plus spectrometer, and a Varian 600 MHz Unity plus spectrometer, using residue solvent peaks as internal standards (CDCl_3 , ^1H : 7.26 ppm; ^{13}C : 77.00 ppm). Infrared spectra were recorded with a Perkin Elmer FT-IR spectrum 2000 spectrometer and are reported in reciprocal centimeter (cm^{-1}). Mass spectra were recorded with Waters (Micromass) LCT premier #1, using electrospray method and TOF detector.

General Procedure A: preparation of substrates 1a-l

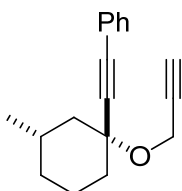


A terminal alkyne (6 mmol) and tetrahydrofuran (10 mL) was mixed in a flame-dried Schlenk flask under N_2 atmosphere. The mixture was cooled to $-78\text{ }^\circ\text{C}$ in a dry ice-acetone bath, to which $n\text{BuLi}$ (2.5 M in hexane, 2.4 mL) was added slowly. The mixture was stirred at the same temperature for 0.5 h before a cyclohexanone (5 mmol) was added in one portion. The reaction was allowed to warm to room temperature and stirred for an additional hour. Upon completion, the reaction was carefully quenched by 5 mL saturated NH_4Cl aqueous solution and the resulting mixture was extracted by diethyl ether. The organic layers are combined, washed with brine, dried with MgSO_4 , filtered and concentrated under vacuum. The crude residue was purified by silica gel chromatography, and the major diastereomer was collected.

The desired tertiary alcohol (1 mmol) thus obtained was dissolved in DMF (5 mL). The

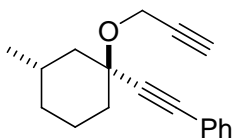
solution was cooled down to 0 °C, and NaH (50 mg, 60% dispersion in mineral oil) was added. The mixture was stirred at 0 °C for 20 min and propargyl bromide (1.2 mmol) was added in one portion. The mixture was then allowed to stir at room temperature until TLC showed completion of reaction. The reaction was poured into a mixture of ice and saturated NH₄Cl solution, and was subsequently extracted with diethyl ether. The organic layer was washed with water and brine, dried with MgSO₄, and concentrated under vacuum. The residue was purified by silica gel chromatography to give the substrates **1a-l**.

((*trans*-3-methyl-1-(prop-2-ynyloxy)cyclohexyl)ethynyl)benzene 1a



1a was synthesized following **General Procedure A** with an overall yield of 40%. ¹H NMR (500 MHz, CDCl₃) δ 7.50 – 7.39 (m, 2H), 7.34 – 7.28 (m, 3H), 4.41 (d, *J* = 2.4 Hz, 2H), 2.42 (dd, *J* = 4.9, 2.4 Hz, 1H), 2.22 – 2.12 (m, 2H), 1.85 – 1.61 (m, 4H), 1.46 (td, *J* = 12.8, 3.8 Hz, 1H), 1.22 (dd, *J* = 15.4, 9.1 Hz, 1H), 0.96 (d, *J* = 6.6 Hz, 3H), 0.92 – 0.82 (m, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 131.71, 128.34, 128.26, 122.69, 89.16, 87.68, 81.25, 76.56, 73.46, 51.76, 45.92, 37.36, 34.26, 30.30, 23.41, 22.16; IR (neat, cm⁻¹): 2930, 2861, 2119, 1490, 1457, 1443, 1303, 1166, 1063; ESI+ calculated for [C₁₈H₂₀NaO]⁺: 275.14, found 275.06.

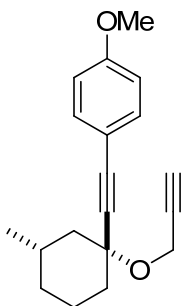
((*cis*-3-methyl-1-(prop-2-ynyloxy)cyclohexyl)ethynyl)benzene 1a'



1a' was synthesized following **General Procedure A** with an overall yield of 26%. ¹H NMR (500 MHz, CDCl₃) δ 7.42 (ddd, *J* = 4.6, 2.9, 1.3 Hz, 2H), 7.32 – 7.27 (m, 3H), 4.34 – 4.27 (m, 2H), 2.47 – 2.35 (m, 1H), 2.18 (d, *J* = 14.0 Hz, 2H), 1.84 (dd, *J* = 6.2, 3.0 Hz, 1H), 1.67 (dd, *J* = 18.0, 7.4 Hz, 2H), 1.61 – 1.56 (m, 1H), 1.32 (t, *J* = 13.1 Hz, 1H), 0.95 – 0.84 (m, 4H); ¹³C NMR (126 MHz, CDCl₃) δ 131.69, 128.25, 128.23, 122.70, 90.85, 84.85,

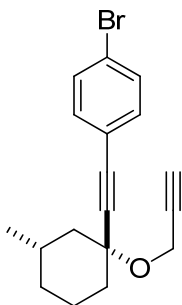
81.11, 73.67, 73.27, 51.93, 44.66, 35.71, 33.93, 26.92, 22.07, 20.88; IR (neat, cm^{-1}): 2949, 2865, 2118, 1491, 1456, 1444, 1352, 1262, 1152, 1066; ESI+ calculated for $[\text{C}_{18}\text{H}_{20}\text{NaO}]^+$: 275.14, found 275.11.

1-Methoxy-4-((*trans*-3-methyl-1-(prop-2-ynoxy)cyclohexyl)ethynyl)benzene 1b



1b was synthesized following **General Procedure A** with an overall yield of 45%. ^1H NMR (500 MHz, CDCl_3) δ 7.38 (d, $J = 8.8$ Hz, 2H), 6.84 (d, $J = 8.8$ Hz, 2H), 4.41 – 4.38 (m, 2H), 3.81 (s, 3H), 2.41 (t, $J = 2.2$ Hz, 1H), 2.15 (t, $J = 8.5$ Hz, 2H), 1.82 – 1.55 (m, 4H), 1.45 (td, $J = 12.8, 3.7$ Hz, 1H), 1.20 (t, $J = 12.2$ Hz, 1H), 0.93 (t, $J = 11.5$ Hz, 3H), 0.91 – 0.82 (m, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 159.60, 133.14, 114.75, 113.85, 87.61, 87.52, 81.29, 76.59, 73.38, 55.31, 55.26, 51.66, 45.93, 37.38, 34.25, 30.26, 23.40, 22.16; IR (neat, cm^{-1}): 3295, 2929, 2860, 2218, 1606, 1509, 1458, 1288, 1248, 1165, 1098, 1061, 1032; ESI+ calculated for $[\text{C}_{19}\text{H}_{22}\text{NaO}_2]^+$: 305.15, found 305.11.

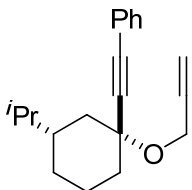
1-Bromo-4-((*trans*-3-methyl-1-(prop-2-ynoxy)cyclohexyl)ethynyl)benzene 1c



1c was synthesized following **General Procedure A** with an overall yield of 37%. ^1H NMR (500 MHz, CDCl_3) δ 7.44 (d, $J = 8.4$ Hz, 2H), 7.30 (d, $J = 8.4$ Hz, 2H), 4.38 (d, $J = 2.4$ Hz, 2H), 2.42 (t, $J = 2.4$ Hz, 1H), 2.15 (dd, $J = 12.9, 5.8$ Hz, 2H), 1.83 – 1.55 (m, 4H), 1.45 (td, $J = 12.7, 3.7$ Hz, 1H), 1.28 – 1.15 (m, 1H), 0.95 (d, $J = 6.6$ Hz, 3H), 0.92 – 0.82

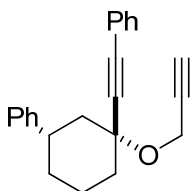
(m, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 133.15, 131.53, 122.59, 121.60, 90.44, 86.56, 81.12, 76.52, 73.56, 51.80, 45.78, 37.25, 34.20, 30.30, 23.39, 22.14; IR (neat, cm^{-1}): 3303, 2949, 2931, 2861, 2221, 2119, 1901, 1649, 1588, 1486, 1458, 1301, 1062, 823; ESI+ calculated for $[\text{C}_{18}\text{H}_{19}\text{BrNaO}]^+$: 353.05, 355.05, found 353.01, 355.01.

((*trans*-3-isopropyl-1-(prop-2-ynyloxy)cyclohexyl)ethynyl)benzene 1d



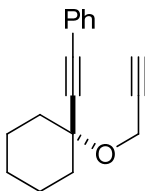
1d was synthesized following **General Procedure A** with an overall yield of 39%. ^1H NMR (500 MHz, CDCl_3) δ 7.45 (dt, $J = 7.3, 3.6$ Hz, 2H), 7.32 (dd, $J = 9.2, 5.6$ Hz, 3H), 4.41 (dt, $J = 14.4, 7.2$ Hz, 2H), 2.43 (t, $J = 2.4$ Hz, 1H), 2.17 (dd, $J = 16.2, 5.9$ Hz, 2H), 1.84 – 1.78 (m, 1H), 1.72 – 1.43 (m, 5H), 1.29 (t, $J = 12.1$ Hz, 1H), 1.01 – 0.86 (m, 7H); ^{13}C NMR (126 MHz, CDCl_3) δ 131.73, 128.34, 128.27, 122.74, 89.16, 87.77, 81.23, 77.01, 73.47, 51.77, 41.38, 41.23, 37.61, 32.45, 28.64, 23.41, 19.73, 19.63; IR (neat, cm^{-1}): 2928, 2853, 2223, 2117, 1490, 1444, 1331, 1072; ESI+ calculated for $[\text{C}_{20}\text{H}_{24}\text{NaO}]^+$: 303.17, found 303.12.

((*trans*-3-phenyl-1-(prop-2-ynyloxy)cyclohexyl)ethynyl)benzene 1e



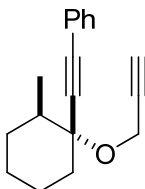
1e was synthesized following **General Procedure A** with an overall yield of 54%. ^1H NMR (500 MHz, CDCl_3) δ 7.53 – 7.44 (m, 2H), 7.38 – 7.29 (m, 5H), 7.27 – 7.19 (m, 3H), 4.49 – 4.38 (m, 2H), 3.01 – 2.93 (m, 1H), 2.45 – 2.36 (m, 2H), 2.29 (d, $J = 12.8$ Hz, 1H), 1.93 (d, $J = 12.0$ Hz, 2H), 1.89 – 1.74 (m, 2H), 1.67 – 1.59 (m, 1H), 1.46 – 1.36 (m, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 145.77, 131.82, 128.54, 128.46, 128.35, 126.92, 126.25, 122.55, 88.66, 88.29, 81.09, 76.69, 73.62, 51.92, 44.44, 41.67, 37.47, 33.70, 23.73; IR (neat, cm^{-1}): 3082, 3061, 3029, 2933, 2859, 2223, 2126, 1599, 1491, 1444, 1335, 1308, 1067, 1014; ESI+ calculated for $[\text{C}_{23}\text{H}_{22}\text{NaO}]^+$: 337.16, found 337.11.

((1-(Prop-2-ynoxy)cyclohexyl)ethynyl)benzene 1f



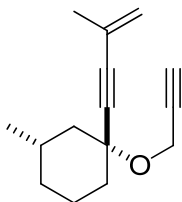
1f was synthesized following **General Procedure A** with an overall yield of 70%. ¹H NMR (500 MHz, CDCl₃) δ 7.45 (dt, *J* = 7.3, 3.6 Hz, 2H), 7.34 – 7.27 (m, 3H), 4.38 (d, *J* = 2.4 Hz, 2H), 2.42 (t, *J* = 2.4 Hz, 1H), 2.06 – 1.97 (m, 2H), 1.80 – 1.67 (m, 4H), 1.67 – 1.50 (m, 3H), 1.32 (dt, *J* = 12.7, 10.0 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 131.72, 128.32, 128.26, 122.72, 89.46, 87.03, 81.23, 75.52, 73.41, 51.77, 37.35, 25.38, 23.02; IR (neat, cm⁻¹): 2936, 2858, 2117, 1489, 1443, 1303, 1146; ESI+ calculated for [C₁₇H₁₈NaO]⁺: 261.13, found 261.09.

((cis-2-methyl-1-(prop-2-ynoxy)cyclohexyl)ethynyl)benzene 1g



1g was synthesized following **General Procedure A** with an overall yield of 35%. ¹H NMR (500 MHz, CDCl₃) δ 7.52 – 7.37 (m, 2H), 7.38 – 7.30 (m, 3H), 4.47 – 4.27 (m, 2H), 2.41 (t, *J* = 2.4 Hz, 1H), 2.29 (dt, *J* = 11.8, 2.8 Hz, 1H), 1.79 – 1.70 (m, 2H), 1.69 – 1.61 (m, 2H), 1.57 (tdd, *J* = 10.5, 6.8, 3.8 Hz, 1H), 1.53 – 1.45 (m, 1H), 1.44 – 1.35 (m, 1H), 1.30 – 1.21 (m, 1H), 1.10 (d, *J* = 6.5 Hz, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 131.74, 128.31, 128.27, 122.82, 89.14, 87.06, 81.53, 80.25, 73.22, 52.08, 41.79, 36.78, 32.23, 25.28, 23.84, 16.59; IR (neat, cm⁻¹): 2931, 2857, 2119, 1643, 1444, 1373, 1344, 1286, 1072; ESI+ calculated for [C₁₈H₂₀NaO]⁺: 275.14, found 275.08.

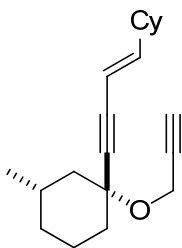
***trans*-3-Methyl-1-(3-methylbut-3-en-1-ynyl)-1-(prop-2-ynyloxy)cyclohexane 1h**



1h was synthesized following **General Procedure A** with an overall yield of 40%. ¹H NMR (500 MHz, CDCl₃) δ 5.30 – 5.27 (m, 1H), 5.24 – 5.21 (m, 1H), 4.34 – 4.30 (m, 2H), 2.42 – 2.38 (m, 1H), 2.11 – 2.04 (m, 2H), 1.90 (t, *J* = 1.3 Hz, 3H), 1.75 – 1.64 (m, 3H), 1.60 – 1.50 (m, 1H), 1.43 – 1.36 (m, 1H), 1.14 (t, *J* = 12.2 Hz, 1H), 0.93 (d, *J* = 6.5 Hz, 3H), 0.88 – 0.78 (m, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 126.32, 121.83, 88.90, 88.11, 81.26, 76.40, 73.36, 51.61, 45.85, 37.31, 34.23, 30.21, 23.54, 23.35, 22.13; IR (neat, cm⁻¹): 2930, 2867, 2118, 1457, 1373, 1305, 1099, 1063; ESI+ calculated for [C₁₅H₂₀NaO]⁺: 239.14, found 239.09.

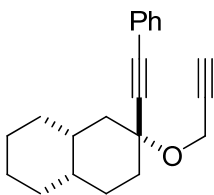
***trans*-1-((*E*)-4-Cyclohexylbut-3-en-1-ynyl)-3-methyl-1-(prop-2-ynyloxy)cyclohexane**

1i



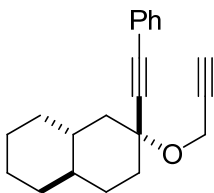
1i was synthesized following **General Procedure A** with an overall yield of 30%. ¹H NMR (500 MHz, CDCl₃) δ 6.09 (dt, *J* = 15.3, 7.7 Hz, 1H), 5.46 (dd, *J* = 16.1, 1.4 Hz, 1H), 4.32 (d, *J* = 2.5 Hz, 2H), 2.41 – 2.38 (m, 1H), 2.11 – 1.98 (m, 3H), 1.79 – 1.63 (m, 8H), 1.62 – 1.51 (m, 1H), 1.42 – 1.35 (m, 1H), 1.31 – 1.22 (m, 2H), 1.20 – 1.04 (m, 4H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.82 (ddd, *J* = 24.8, 12.8, 3.8 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 150.50, 106.59, 87.61, 86.55, 81.37, 76.56, 73.27, 51.57, 45.92, 41.20, 37.36, 34.26, 32.21, 30.17, 26.00, 25.82, 23.34, 22.12; IR (neat, cm⁻¹): 3021, 2927, 2852, 2201, 1448, 1332, 1295, 1278, 1184, 1098, 1064, 957; ESI+ calculated for [C₂₀H₂₈NaO]⁺: 307.20, found 307.16.

***trans*-2-(phenylethynyl)-2-(prop-2-ynyloxy)-*cis*-decahydronaphthalene 1j**



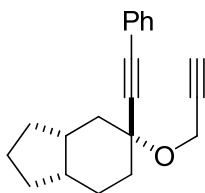
1j was synthesized following **General Procedure A** with an overall yield of 33%. ¹H NMR (500 MHz, CDCl₃) δ 7.48 – 7.39 (m, 2H), 7.35 – 7.28 (m, 3H), 4.41 (d, *J* = 2.4 Hz, 2H), 2.43 (dd, *J* = 5.5, 3.1 Hz, 1H), 2.13 – 2.05 (m, 1H), 1.97 – 1.80 (m, 3H), 1.80 – 1.65 (m, 4H), 1.65 – 1.53 (m, 3H), 1.47 – 1.40 (m, 1H), 1.39 – 1.19 (m, 4H); ¹³C NMR (126 MHz, CDCl₃) δ 131.73, 128.33, 128.26, 122.70, 89.18, 87.51, 81.20, 73.49, 51.72, 46.71, 44.65, 42.75, 40.09, 37.75, 37.31, 34.93, 33.56, 33.10, 32.52, 31.43, 31.02, 26.49, 21.17 (aliphatic carbon signals are messy due to rapid conformation change); IR (neat, cm⁻¹): 2924, 2857, 2126, 1489, 1443, 1371, 1304, 1275, 1144, 1068; ESI+ calculated for [C₂₁H₂₄NaO]⁺: 315.17, found 315.12.

***trans*-2-(phenylethynyl)-2-(prop-2-ynyloxy)-*trans*-decahydronaphthalene 1k**



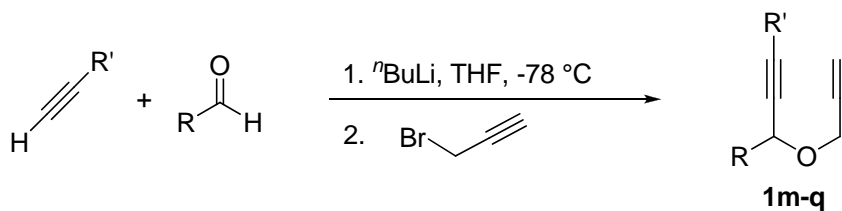
1k was synthesized following **General Procedure A** with an overall yield of 40%. ¹H NMR (500 MHz, CDCl₃) δ 7.45 (dt, *J* = 7.2, 3.0 Hz, 2H), 7.37 – 7.28 (m, 3H), 4.40 (d, *J* = 2.4 Hz, 2H), 2.42 (t, *J* = 2.4 Hz, 1H), 2.19 (ddd, *J* = 11.7, 6.0, 2.8 Hz, 1H), 2.13 – 2.03 (m, 1H), 1.75 – 1.55 (m, 6H), 1.46 – 1.18 (m, 5H), 1.09 – 0.85 (m, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 131.74, 128.34, 128.27, 122.74, 89.39, 87.47, 81.24, 76.49, 73.46, 51.80, 44.65, 42.75, 40.10, 37.76, 33.51, 33.11, 31.02, 26.49, 26.24; IR (neat, cm⁻¹): 2923, 2854, 2221, 2127, 1598, 1489, 1444, 1321, 1068; ESI+ calculated for [C₂₁H₂₄NaO]⁺: 315.17, found 315.12.

***trans*-5-(phenylethynyl)-5-(prop-2-ynyloxy)octahydro-1H-indene 11**



11 was synthesized following **General Procedure A** with an overall yield of 27%. ^1H NMR (500 MHz, CDCl_3) δ 7.47 – 7.41 (m, 2H), 7.32 (dd, $J = 5.0, 1.6$ Hz, 3H), 4.40 (d, $J = 2.4$ Hz, 2H), 2.42 (t, $J = 2.4$ Hz, 1H), 2.29 – 2.15 (m, 1H), 2.01 – 1.89 (m, 4H), 1.81 – 1.67 (m, 4H), 1.67 – 1.51 (m, 3H), 1.48 – 1.39 (m, 2H); ^{13}C NMR (500 MHz, CDCl_3) δ 80.88, 80.64, 80.63, 80.24, 77.82, 77.76, 77.30, 76.97, 76.75, 75.21, 74.28, 74.23, 74.23, 73.82, 73.79, 73.43, 73.27, 73.11; IR (neat, cm^{-1}): 2949, 2874, 2117, 1643, 1489, 1444, 1371, 1312, 1070; ESI+ calculated for $[\text{C}_{20}\text{H}_{22}\text{NaO}]^+$: 301.16, found 301.12.

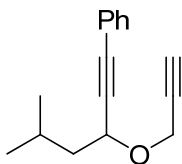
General Procedure B: preparation of substrates from aldehydes



A terminal alkyne (6 mmol) and tetrahydrofuran (10 mL) was mixed in a flame-dried Schlenk flask under N_2 atmosphere. The mixture was then cooled to -78 °C in a dry ice-acetone bath, and $n\text{BuLi}$ (2.5 M in hexane, 2.4 mL) was added slowly. The mixture was stirred at the same temperature for 0.5 h before the corresponding aldehyde (5 mmol) was added in one portion. The reaction was allowed to warm to room temperature and stirred for an additional hour. Upon completion, propargyl bromide (1.2 mmol) was added in one portion, and the mixture was stirred at room temperature. The reaction was monitored by TLC until the complete consumption of the tertiary alcohol. If no product is forming after the addition of propargyl bromide, tetrabutylammonium iodide (20 mol%) and DMF (1 mL) could be added to accelerate the reaction. Upon completion, the reaction was quenched by saturated NH_4Cl and extracted with diethyl ether. The combined organic layers were washed with water and brine, dried with MgSO_4 , and

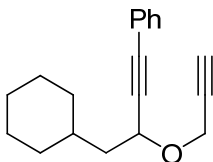
concentrated under vacuum. The residue was purified by silica gel chromatography to give the substrates **1n-r**.

(5-methyl-3-(prop-2-ynyloxy)hex-1-ynyl)benzene 1m



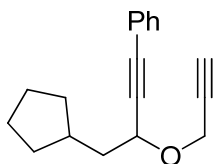
1m was synthesized following **General Procedure B** with an overall yield of 80%. ¹H NMR (500 MHz, CDCl₃) δ 7.50 – 7.41 (m, 2H), 7.37 – 7.28 (m, 3H), 4.57 (d, *J* = 6.8 Hz, 1H), 4.38 (ddd, *J* = 15.7, 10.8, 2.4 Hz, 2H), 2.45 – 2.44 (m, 1H), 2.00 – 1.90 (m, 1H), 1.80 (dt, *J* = 14.2, 7.2 Hz, 1H), 1.67 (dt, *J* = 13.7, 6.9 Hz, 1H), 0.98 (t, *J* = 6.3 Hz, 6H); ¹³C NMR (126 MHz, CDCl₃) δ 131.74, 128.38, 128.26, 122.63, 87.54, 86.15, 79.72, 74.33, 67.38, 55.73, 44.57, 24.72, 22.71, 22.35; IR (neat, cm⁻¹): 2958, 2870, 2118, 1644, 1490, 1468, 1443, 1331, 1127, 1081; ESI+ calculated for [C₁₆H₁₈NaO]⁺: 249.13, found 249.02.

(4-Cyclohexyl-3-(prop-2-ynyloxy)but-1-ynyl)benzene 1n



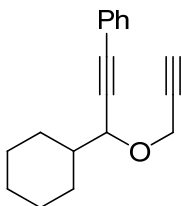
1n was synthesized following **General Procedure B** with an overall yield of 72%. ¹H NMR (500 MHz, CDCl₃) δ 7.45 (dtt, *J* = 5.5, 2.9, 1.4 Hz, 2H), 7.34 – 7.30 (m, 3H), 4.60 (dd, *J* = 7.8, 6.4 Hz, 1H), 4.37 (ddd, *J* = 15.7, 12.4, 2.4 Hz, 2H), 2.44 (t, *J* = 2.4 Hz, 1H), 1.88 – 1.75 (m, 3H), 1.75 – 1.60 (m, 5H), 1.34 – 1.22 (m, 2H), 1.22 – 1.15 (m, 1H), 1.02 – 0.92 (m, 2H); ¹³C NMR (126 MHz, CDCl₃) δ 131.75, 128.36, 128.30, 128.26, 122.67, 87.68, 86.11, 79.73, 74.32, 66.89, 55.73, 43.21, 34.03, 33.44, 32.99, 26.53, 26.21, 26.13; IR (neat, cm⁻¹): 3081, 3057, 3034, 2924, 2852, 2226, 2118, 1742, 1599, 1490, 1447, 1336, 1261, 1074; ESI+ calculated for [C₁₉H₂₂NaO]⁺: 289.16, found 289.12.

(4-Cyclopentyl-3-(prop-2-ynoxy)but-1-ynyl)benzene 1o



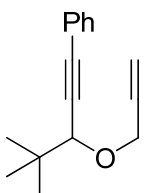
1o was synthesized following **General Procedure B** with an overall yield of 75%. ¹H NMR (600 MHz, CDCl₃) δ 7.45 – 7.41 (m, 2H), 7.34 – 7.27 (m, 3H), 4.50 (t, *J* = 6.9 Hz, 1H), 4.35 (qd, *J* = 15.7, 2.4 Hz, 2H), 2.43 (t, *J* = 2.4 Hz, 1H), 2.08 (dt, *J* = 15.3, 7.7 Hz, 1H), 1.92 – 1.79 (m, 4H), 1.65 – 1.58 (m, 2H), 1.58 – 1.48 (m, 2H), 1.21 – 1.11 (m, 2H); ¹³C NMR (126 MHz, CDCl₃) δ 131.74, 128.37, 128.25, 122.65, 87.61, 86.16, 79.74, 74.30, 68.44, 55.75, 41.95, 36.52, 32.72, 32.63, 25.12, 24.98; IR (neat, cm⁻¹): 2950, 2867, 2117, 1643, 1490, 1443, 1333, 1075; ESI+ calculated for [C₁₈H₂₀NaO]⁺: 275.14, found 275.10.

(3-Cyclohexyl-3-(prop-2-ynoxy)prop-1-ynyl)benzene 1p



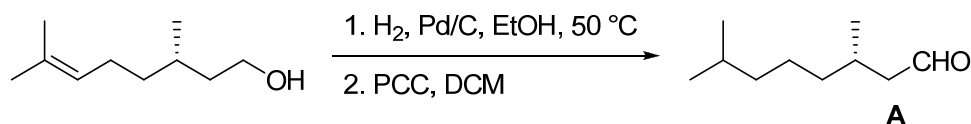
1p was synthesized following **General Procedure B** with an overall yield of 82%. ¹H NMR (500 MHz, CDCl₃) δ 7.46 (dd, *J* = 6.7, 3.1 Hz, 2H), 7.32 (dd, *J* = 4.6, 2.0 Hz, 3H), 4.46 – 4.29 (m, 3H), 2.44 (t, *J* = 2.3 Hz, 1H), 1.98 – 1.89 (m, 2H), 1.83 – 1.62 (m, 4H), 1.33 – 1.13 (m, 5H); ¹³C NMR (126 MHz, CDCl₃) δ 131.77, 128.32, 128.25, 122.74, 87.02, 86.45, 79.83, 74.22, 73.66, 55.95, 42.66, 29.06, 28.50, 26.43, 25.94, 25.91; IR (neat, cm⁻¹): 2927, 2853, 2117, 1643, 1490, 1444, 1331, 1261, 1072; ESI+ calculated for [C₁₈H₂₀NaO]⁺: 275.14, found 275.11.

(4,4-Dimethyl-3-(prop-2-ynoxy)pent-1-ynyl)benzene 1q

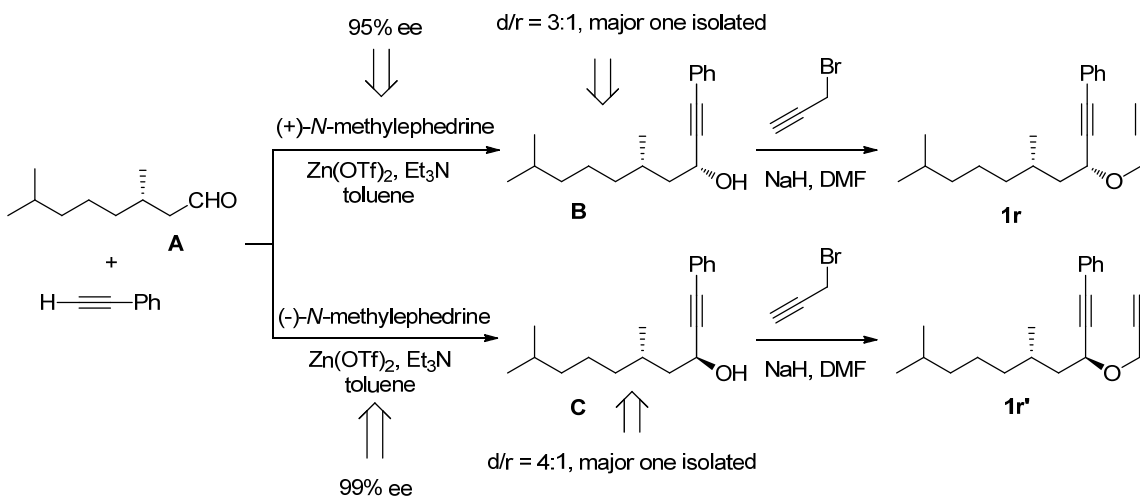


1q was synthesized following **General Procedure B** with an overall yield of 81%. ^1H NMR (500 MHz, CDCl_3) δ 7.48 – 7.42 (m, 2H), 7.34 – 7.28 (m, 3H), 4.38 (ddd, $J = 19.5, 16.0, 2.4$ Hz, 2H), 4.16 (s, 1H), 2.44 – 2.41 (m, 1H), 1.08 (s, 9H); ^{13}C NMR (126 MHz, CDCl_3) δ 131.75, 128.27, 128.25, 122.84, 86.81, 86.39, 79.87, 77.51, 74.16, 56.28, 35.56, 25.91 IR (neat, cm^{-1}): 3082, 3035, 2958, 2906, 2869, 2219, 2118, 1599, 1490, 1394, 1364, 1321, 1246, 1195, 1073; ESI+ calculated for $[\text{C}_{16}\text{H}_{18}\text{NaO}]^+$: 249.13, found 249.10.

Preparation of chiral substrates **1r**

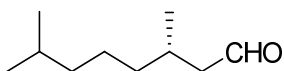


Pd/C (150 mg, 10% on carbon) was added to a dry round-bottom flask flushed with H_2 . To the flask was added ethanol (10 mL), (-)-citronellol (50 mmol, 95% ee), and concentrated HCl (12 M, 0.05 mL). The mixture was stirred at 50 °C under H_2 atmosphere and monitored by TLC. Upon completion, Pd/C was filtered off by a Celite plug, and the filtrate was concentrated under vacuum. The residue was dissolved in DCM (20 mL) and Celite was added to make the solution into a slurry. Pyridinium chlorochromate (60 mmol) was added to the slurry, and the mixture was stirred overnight at room temperature. The reaction mixture was then filtered through a silica pad to remove the chromium salts. The filtrate was concentrated under vacuum, and purified by silica gel chromatography to give the dihydrocitronellal **A**.



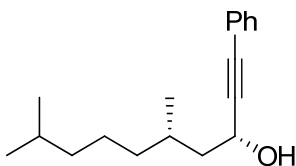
The asymmetric nucleophilic addition to **A** (2 mmol) was performed using Carreira's method.¹ (+)-*N*-methylephedrine (95% ee) and (-)-*N*-methylephedrine (99% ee) were used to prepare the chiral tertiary alcohols **B** and **C**, respectively. After careful column chromatography, **B** and **C** were isolated pure in 69% and 60% yield, respectively.

(*S*)-3,7-Dimethyloctanal A



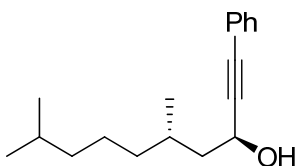
¹H NMR (500 MHz, CDCl₃) δ 9.76 (t, *J* = 2.3 Hz, 1H), 2.43 – 2.35 (m, 1H), 2.22 (ddd, *J* = 16.0, 7.8, 2.6 Hz, 1H), 2.06 (dt, *J* = 12.5, 5.7 Hz, 1H), 1.52 (dq, *J* = 13.3, 6.7 Hz, 1H), 1.36 – 1.12 (m, 6H), 0.96 (d, *J* = 6.7 Hz, 3H), 0.87 (d, *J* = 6.6 Hz, 6H); ¹³C NMR (126 MHz, CDCl₃) δ 203.11, 51.09, 38.98, 37.13, 28.18, 27.89, 24.65, 22.62, 22.54, 19.97; IR (neat, cm⁻¹): 2956, 2929, 2871, 2714, 1728, 1465, 1383, 1367, 1238, 1170, 1015; ESI+ calculated for [C₁₀H₂₀NaO]⁺: 179.14, found 179.07.

(3*R*, 5*S*)-5,9-dimethyl-1-phenyldec-1-yn-3-ol B



¹H NMR (500 MHz, CDCl₃) δ 7.45 – 7.40 (m, 2H), 7.30 (ddd, *J* = 10.8, 7.1, 2.7 Hz, 3H), 4.66 (dt, *J* = 8.0, 5.6 Hz, 1H), 1.89 – 1.82 (m, 1H), 1.82 – 1.73 (m, 2H), 1.61 – 1.55 (m, 1H), 1.53 – 1.49 (m, 1H), 1.39 – 1.25 (m, 3H), 1.21 – 1.13 (m, 3H), 0.97 (d, *J* = 6.6 Hz, 3H), 0.87 (d, *J* = 6.6 Hz, 6H); ¹³C NMR (126 MHz, CDCl₃) δ 131.63, 128.31, 128.25, 122.69, 90.61, 84.65, 61.23, 45.38, 39.20, 37.26, 29.34, 27.96, 24.54, 22.69, 22.58, 19.49; IR (neat, cm⁻¹): 2954, 2928, 2869, 1490, 1466, 1443, 1366, 1028; ESI+ calculated for [C₁₈H₂₆NaO]⁺: 281.19, found 281.04.

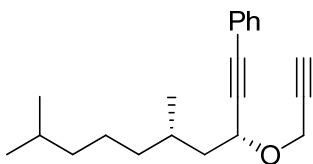
(3*S*, 5*S*)-5,9-dimethyl-1-phenyldec-1-yn-3-ol C



^1H NMR (500 MHz, CDCl_3) δ 7.46 – 7.40 (m, 2H), 7.36 – 7.29 (m, 3H), 4.70 – 4.65 (m, 1H), 1.84 – 1.74 (m, 2H), 1.69 – 1.62 (m, 1H), 1.54 – 1.51 (m, 1H), 1.41 – 1.25 (m, 4H), 1.22 – 1.14 (m, 2H), 0.97 (d, $J = 6.5$ Hz, 3H), 0.87 (d, $J = 6.6$ Hz, 6H); ^{13}C NMR (126 MHz, CDCl_3) δ 131.65, 128.32, 128.25, 122.70, 90.30, 84.94, 61.74, 45.25, 39.19, 37.23, 29.74, 27.96, 24.53, 22.68, 22.58, 19.82; IR (neat, cm^{-1}): 2958, 2932, 2100, 1643, 1489, 1465, 1445, 1382, 1364, 1140; ESI+ calculated for $[\text{C}_{18}\text{H}_{26}\text{NaO}]^+$: 281.19, found 281.15.

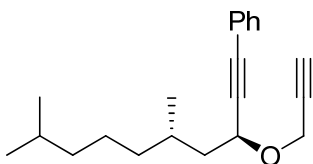
The chiral tertiary alcohol **B** or **C** (1 mmol) obtained from last step was dissolved in DMF (5 mL). The solution was cooled down to 0 °C and NaH (50 mg, 60% dispersion in mineral oil) was added. The mixture was stirred at 0 °C for 20 min and propargyl bromide (1.2 mmol) was added in one portion. The mixture was then allowed to stir at room temperature until TLC showed completion of reaction. The reaction was poured into a mixture of ice and saturated NH_4Cl solution, and was subsequently extracted with diethyl ether. The combined organic layers were washed with water and brine, dried with MgSO_4 , and concentrated under vacuum. The residue was purified by silica gel chromatography to give the chiral substrates **1s** and **1t**.

((3R, 5S)-5,9-dimethyl-3-(prop-2-ynyloxy)dec-1-ynyl)benzene (3R, 5S)-1r



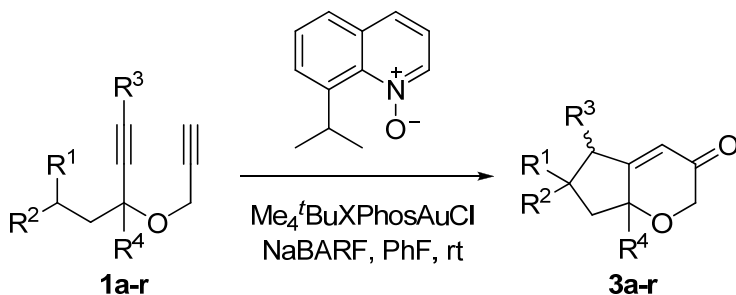
^1H NMR (500 MHz, CDCl_3) δ 7.49 – 7.40 (m, 2H), 7.34 – 7.30 (m, 3H), 4.60 (dd, $J = 8.4$, 5.5 Hz, 1H), 4.43 – 4.33 (m, 2H), 2.45 (t, $J = 2.4$ Hz, 1H), 1.92 (ddd, $J = 13.8$, 8.4, 5.4 Hz, 1H), 1.81 (h, $J = 6.6$ Hz, 1H), 1.63 – 1.56 (m, 1H), 1.54 – 1.50 (m, 1H), 1.36 – 1.26 (m, 3H), 1.22 – 1.14 (m, 3H), 0.97 (d, $J = 6.6$ Hz, 3H), 0.87 (d, $J = 6.6$ Hz, 6H); ^{13}C NMR (126 MHz, CDCl_3) δ 131.72, 128.35, 128.24, 122.65, 87.76, 86.00, 79.73, 74.30, 67.02, 55.71, 42.99, 39.24, 37.32, 29.22, 27.95, 24.53, 22.69, 22.58, 19.36; IR (neat, cm^{-1}): 2955, 2929, 2118, 1490, 1466, 1365, 1081; ESI+ calculated for $[\text{C}_{21}\text{H}_{28}\text{NaO}]^+$: 319.20, found 319.16.

((3*S*, 5*S*)-5,9-dimethyl-3-(prop-2-ynoxy)dec-1-ynyl)benzene (3*S*, 5*S*)-1r

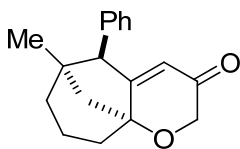


^1H NMR (500 MHz, CDCl_3) δ 7.47 – 7.41 (m, 2H), 7.35 – 7.28 (m, 3H), 4.58 (t, $J = 7.0$ Hz, 1H), 4.37 (qd, $J = 15.7, 2.4$ Hz, 2H), 2.44 (dd, $J = 2.4, 1.9$ Hz, 1H), 1.85 – 1.73 (m, 2H), 1.73 – 1.63 (m, 1H), 1.51 (dd, $J = 13.3, 6.6$ Hz, 1H), 1.40 – 1.24 (m, 3H), 1.19 – 1.12 (m, 3H), 0.96 (d, $J = 6.4$ Hz, 3H), 0.86 (d, $J = 6.6$ Hz, 6H); ^{13}C NMR (126 MHz, CDCl_3) δ 131.75, 128.37, 128.25, 122.64, 87.44, 86.32, 79.71, 74.31, 67.68, 55.76, 42.75, 39.19, 37.05, 29.59, 27.93, 24.45, 22.68, 22.59, 19.84; IR (neat, cm^{-1}): 2954, 2928, 2869, 2118, 1490, 1465, 1365, 1334, 1130, 1078; ESI+ calculated for $[\text{C}_{21}\text{H}_{28}\text{NaO}]^+$: 319.20, found 319.16.

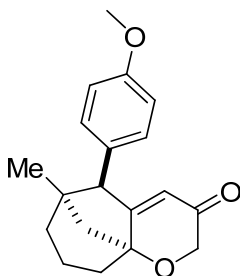
General procedure C: C-H insertion reactions



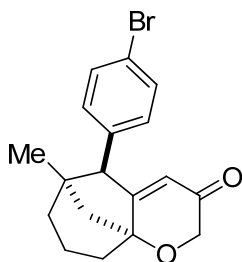
Substrate **1a-t** (0.05 mmol), 8-isopropylquinoline *N*-oxide (12.2 mg, 0.065 mmol), and fluorobenzene (1 mL) were mixed in a dry, clean vial with a magnetic string bar. The solution was stirred briefly before NaBARF (3.3 mg, 7.5 mol%) and $\text{Me}_4^t\text{BuXPhosAuCl}$ (1.8 mg, 5 mol%) were sequentially added. The reaction was then allowed to stir at room temperature until TLC showed complete consumption of the substrate. Fluorobenzene was then removed under vacuum and the residue was purified by silica gel chromatography to give the products **3a-r**.

3a

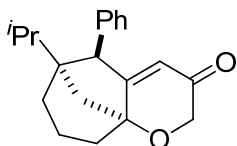
3a was prepared following **General Procedure C** in 70% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.39 – 7.30 (m, 4H), 7.30 – 7.26 (m, 1H), 5.99 (d, $J = 2.2$ Hz, 1H), 4.38 (dd, $J = 141.2, 17.9$ Hz, 2H), 3.89 (s, 1H), 2.64 – 2.36 (m, 1H), 1.96 (dt, $J = 10.8, 2.5$ Hz, 1H), 1.74 (d, $J = 10.7$ Hz, 1H), 1.70 – 1.64 (m, 3H), 1.30 – 1.22 (m, 4H), 1.18 – 1.10 (m, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 194.84, 175.07, 135.50, 128.87, 128.38, 127.11, 120.51, 80.59, 67.51, 58.16, 51.39, 44.88, 32.96, 32.88, 26.65, 19.78; IR (neat, cm^{-1}): 2954, 2874, 1669, 1495, 1450, 1386, 1317, 1260, 1186, 1105; HRMS (ES $^+$) calculated for $[\text{C}_{18}\text{H}_{20}\text{O}_2]^+$: 268.1463, found 268.1469.

3b

3b was prepared following **General Procedure C** in 62% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.24 (d, $J = 8.6$ Hz, 2H), 6.87 (d, $J = 8.8$ Hz, 2H), 5.97 (d, $J = 2.2$ Hz, 1H), 4.52 (d, $J = 17.9$ Hz, 1H), 4.23 (d, $J = 17.9$ Hz, 1H), 3.83 (s, 1H), 3.81 (s, 3H), 2.51 – 2.43 (m, 1H), 1.95 – 1.90 (m, 1H), 1.76 – 1.71 (m, 1H), 1.71 – 1.52 (m, 4H), 1.29 – 1.18 (m, 4H), 1.14 (dd, $J = 13.7, 4.6$ Hz, 1H); ^{13}C NMR (151 MHz, CDCl_3) δ 175.72, 158.53, 129.88, 127.34, 120.28, 113.71, 80.57, 67.54, 57.54, 55.23, 51.22, 44.74, 32.90, 32.87, 26.55, 19.80; IR (neat, cm^{-1}): 2932, 1731, 1696, 1511, 1456, 1236, 1172, 1103, 1030; HRMS (ES $^+$) calculated for $[\text{C}_{19}\text{H}_{22}\text{O}_3]^+$: 298.1569, found 298.1571.

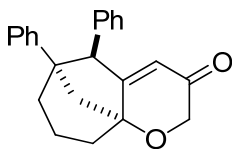
3c

3c was prepared following **General Procedure C** in 55% yield. ^1H NMR (600 MHz, CDCl_3) δ 7.47 (d, $J = 8.4$ Hz, 2H), 7.21 (d, $J = 8.4$ Hz, 2H), 5.95 (d, $J = 2.0$ Hz, 1H), 4.51 (d, $J = 17.9$ Hz, 1H), 4.24 (d, $J = 17.9$ Hz, 1H), 3.84 (s, 1H), 2.51 – 2.44 (m, 1H), 1.95 (d, $J = 10.8$ Hz, 1H), 1.77 – 1.64 (m, 3H), 1.62 – 1.56 (m, 1H), 1.31 – 1.20 (m, 4H), 1.12 (dd, $J = 14.1, 5.5$ Hz, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 194.69, 174.31, 134.55, 131.55, 130.48, 121.08, 120.62, 80.50, 67.48, 57.55, 51.29, 44.85, 32.90, 32.79, 26.56, 19.78; IR (neat, cm^{-1}): 2956, 2870, 1672, 1489, 1457, 1261, 1101, 1074, 1010; HRMS (ES+) calculated for $[\text{C}_{18}\text{H}_{19}\text{BrO}_2]^+$: 346.0568, found 346.0572.

3d

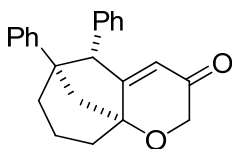
3d was prepared following **General Procedure C** in 64% yield. ^1H NMR (600 MHz, CDCl_3) δ 7.31 (t, $J = 4.3$ Hz, 4H), 7.27 – 7.25 (m, 1H), 5.94 (d, $J = 2.2$ Hz, 1H), 4.50 (d, $J = 17.8$ Hz, 1H), 4.22 (d, $J = 17.8$ Hz, 1H), 4.25 (s, 1H), 2.47 (dd, $J = 6.6, 3.2$ Hz, 1H), 1.96 – 1.88 (m, 2H), 1.70 – 1.59 (m, 5H), 1.10 (d, $J = 6.8$ Hz, 3H), 0.92 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 194.80, 175.69, 136.22, 128.98, 128.41, 126.97, 120.42, 80.65, 67.54, 53.17, 51.21, 43.24, 33.34, 32.25, 29.79, 19.77, 18.63, 17.54; IR (neat, cm^{-1}): 2962, 2877, 1671, 1497, 1449, 1372, 1317, 1261, 1112, 1087; HRMS (ES+) calculated for $[\text{C}_{20}\text{H}_{24}\text{O}_2]^+$: 296.1776, found 296.1779.

3e



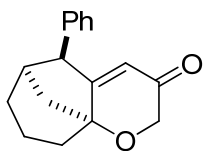
3e was prepared following **General Procedure C** in 53% yield. ^1H NMR (600 MHz, CDCl_3) δ 7.42 – 7.35 (m, 4H), 7.30 (dd, $J = 9.7, 4.3$ Hz, 1H), 7.22 – 7.13 (m, 3H), 6.86 (d, $J = 7.5$ Hz, 2H), 6.08 (d, $J = 1.9$ Hz, 1H), 4.54 (s, 1H), 4.56 (d, $J = 17.8$ Hz, 1H), 4.29 (d, $J = 17.8$ Hz, 1H), 2.63 (d, $J = 11.2$ Hz, 1H), 2.55 (s, 1H), 1.96 (d, $J = 11.1$ Hz, 1H), 1.76 (ddd, $J = 20.6, 18.4, 6.8$ Hz, 5H); ^{13}C NMR (126 MHz, CDCl_3) δ 194.71, 173.38, 145.00, 135.11, 128.62, 128.18, 128.13, 127.07, 126.81, 126.17, 121.25, 80.89, 67.46, 58.80, 51.62, 50.46, 32.72, 30.15, 19.38; IR (neat, cm^{-1}): 2962, 1669, 1501, 1446, 1259, 1156, 1102; HRMS (ES+) calculated for $[\text{C}_{23}\text{H}_{22}\text{O}_2]^+$: 330.1620, found 330.1625.

3e'



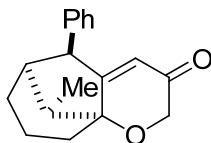
3e' was prepared following **General Procedure C** in 17% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.07 (dd, $J = 7.9, 7.5$ Hz, 2H), 7.05 – 6.94 (m, 6H), 6.88 (d, $J = 6.9$ Hz, 2H), 5.59 (d, $J = 1.9$ Hz, 1H), 4.48 (d, $J = 17.7$ Hz, 1H), 4.23 (d, $J = 17.7$ Hz, 1H), 4.06 (s, 1H), 2.89 (dt, $J = 11.0, 2.6$ Hz, 1H), 2.54 – 2.44 (m, 1H), 2.18 (dd, $J = 13.7, 2.5$ Hz, 1H), 2.14 (dd, $J = 11.1, 1.8$ Hz, 1H), 2.07 – 2.01 (m, 1H), 1.87 – 1.67 (m, 3H); ^{13}C (126 MHz, CDCl_3) δ 194.91, 176.78, 144.11, 140.85, 128.00, 127.72, 126.90, 126.42, 125.84, 121.66, 80.89, 67.25, 57.03, 51.89, 46.66, 42.68, 31.89, 20.35; IR (neat, cm^{-1}): 2958, 1671, 1494, 1452, 1314, 1263, 1121, 1097; HRMS (ES+) calculated for $[\text{C}_{23}\text{H}_{22}\text{O}_2]^+$: 330.1620, found 330.1625.

3f



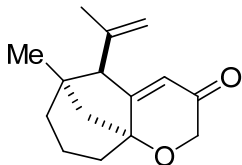
3f was prepared following **General Procedure C** in 50% yield. The product slightly decomposes on silica gel column. ^1H NMR (500 MHz, CDCl_3) δ 7.37 – 7.31 (m, 4H), 7.25 (d, $J = 2.5$ Hz, 1H), 6.17 (d, $J = 2.2$ Hz, 1H), 4.40 (dd, $J = 6.7, 1.8$ Hz, 1H), 4.52 (d, $J = 17.8$ Hz, 1H), 4.24 (d, $J = 17.8$ Hz, 1H), 2.84 (s, 1H), 2.46 (d, $J = 11.7$ Hz, 1H), 2.14 – 2.09 (m, 1H), 1.90 (d, $J = 10.7$ Hz, 1H), 1.69 (td, $J = 12.4, 5.8$ Hz, 1H), 1.54 – 1.50 (m, 1H), 1.43 – 1.26 (m, 6H); ^{13}C NMR (126 MHz, CDCl_3) δ 194.88, 172.60, 136.51, 128.53, 127.89, 126.67, 121.65, 81.36, 67.09, 51.09, 44.18, 39.53, 33.20, 26.00, 19.12; IR (neat, cm^{-1}): 2937, 2587, 1678, 1494, 1448, 1314, 1256, 1130, 1084, 1033; HRMS (ES+) calculated for $[\text{C}_{17}\text{H}_{18}\text{O}_2]^+$: 254.1307, found 254.1302.

3g



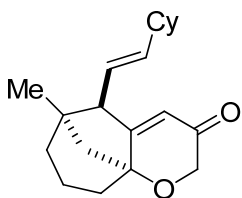
3g was prepared following **General Procedure C** in 40% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.35 – 7.30 (m, 4H), 7.25 – 7.20 (m, 1H), 6.24 (d, $J = 2.1$ Hz, 1H), 4.46 (d, $J = 7.1$ Hz, 1H), 4.50 (d, $J = 17.7$ Hz, 1H), 4.24 (d, $J = 17.7$ Hz, 1H), 2.54 – 2.43 (m, 2H), 2.12 (q, $J = 7.0$ Hz, 1H), 1.69 – 1.60 (m, 1H), 1.50 – 1.27 (m, 5H), 1.08 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 194.91, 171.80, 136.79, 128.43, 127.91, 126.45, 123.63, 83.44, 66.91, 48.04, 47.69, 45.71, 33.83, 27.55, 19.24, 13.31; IR (neat, cm^{-1}): 2930, 2860, 1674, 1494, 1449, 1264, 1134, 1099, 1023; HRMS (ES+) calculated for $[\text{C}_{18}\text{H}_{20}\text{O}_2]^+$: 268.1463, found 268.1465.

3h



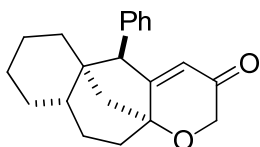
3h was prepared following **General Procedure C** in 50% yield. ^1H NMR (500 MHz, CDCl_3) δ 5.91 (d, $J = 2.1$ Hz, 1H), 5.11 – 5.04 (m, 1H), 4.96 (d, $J = 0.6$ Hz, 1H), 4.44 (d, $J = 17.7$ Hz, 1H), 4.17 (d, $J = 17.8$ Hz, 1H), 2.41 – 2.32 (m, 1H), 1.88 (d, $J = 0.6$ Hz, 3H), 1.86 – 1.82 (m, 1H), 1.70 – 1.55 (m, 5H), 1.40 – 1.34 (m, 1H), 1.24 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 194.94, 174.81, 139.49, 121.13, 115.21, 80.43, 67.25, 59.37, 52.17, 43.44, 33.82, 32.45, 27.58, 24.32, 19.83; IR (neat, cm^{-1}): 2952, 2872, 1668, 1457, 1316, 1286, 1267, 1214, 1099, 1028; HRMS (ES+) calculated for $[\text{C}_{15}\text{H}_{20}\text{O}_2]^+$: 232.1463, found 232.1463.

3i



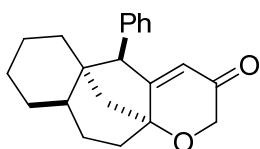
3i was prepared following **General Procedure C** in 54% yield. ^1H NMR (500 MHz, CDCl_3) δ 5.84 (d, $J = 2.2$ Hz, 1H), 5.60 (dt, $J = 15.9, 8.0$ Hz, 1H), 5.36 – 5.27 (m, 1H), 4.42 (d, $J = 17.9$ Hz, 1H), 4.17 (d, $J = 17.9$ Hz, 1H), 2.98 (d, $J = 8.1$ Hz, 1H), 2.30 (dd, $J = 7.3, 4.5$ Hz, 1H), 2.03 – 1.96 (m, 1H), 1.76 – 1.68 (m, 5H), 1.68 – 1.63 (m, 1H), 1.58 (d, $J = 6.3$ Hz, 1H), 1.56 – 1.51 (m, 1H), 1.44 – 1.24 (m, 4H), 1.19 – 1.07 (m, 3H), 1.04 (d, $J = 2.4$ Hz, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 195.00, 177.17, 143.03, 120.28, 118.87, 80.94, 67.55, 55.94, 50.57, 43.09, 40.92, 33.53, 33.21, 33.11, 32.68, 26.09, 25.93, 25.92, 25.31, 20.29; IR (neat, cm^{-1}): 2926, 2852, 1678, 1449, 1315, 1284, 1259, 1076; HRMS (ES+) calculated for $[\text{C}_{20}\text{H}_{28}\text{O}_2]^+$: 300.2089, found 300.2090.

3j



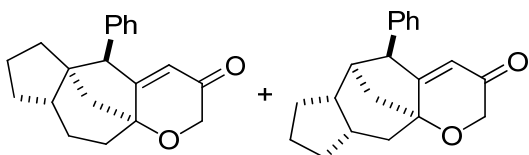
3j was prepared following **General Procedure C** in 64% yield. ^1H NMR (600 MHz, CDCl_3) δ 7.34 – 7.30 (m, 4H), 7.30 – 7.26 (m, 1H), 5.95 (d, $J = 1.7$ Hz, 1H), 4.50 (d, $J = 17.9$ Hz, 1H), 4.22 (d, $J = 17.9$ Hz, 1H), 3.79 (s, 1H), 2.37 – 2.29 (m, 2H), 1.94 (tt, $J = 13.8, 6.8$ Hz, 1H), 1.82 – 1.71 (m, 2H), 1.69 – 1.60 (m, 2H), 1.57 (dd, $J = 14.5, 10.6$ Hz, 1H), 1.53 – 1.49 (m, 2H), 1.40 – 1.24 (m, 3H), 1.11 – 0.99 (m, 2H); ^{13}C NMR (126 MHz, CDCl_3) δ 194.95, 175.19, 135.05, 129.42, 128.32, 127.25, 119.97, 81.27, 67.54, 60.52, 49.48, 43.40, 37.03, 35.53, 30.43, 29.59, 26.60, 26.11, 23.35; IR (neat, cm^{-1}): 2929, 2859, 1672, 1497, 1449, 1320, 1263, 1241, 1105, 1034; HRMS (ES $^+$) calculated for $[\text{C}_{21}\text{H}_{24}\text{O}_2]^+$: 308.1776, found 308.1781.

3k



3k was prepared following **General Procedure C** in 60% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.42 – 7.37 (m, 2H), 7.32 – 7.27 (m, 3H), 5.80 (d, $J = 2.5$ Hz, 1H), 4.49 (d, $J = 17.9$ Hz, 1H), 4.21 (d, $J = 17.9$ Hz, 1H), 2.59 – 2.53 (m, 1H), 1.92 (dd, $J = 10.7, 3.1$ Hz, 2H), 1.86 – 1.76 (m, 3H), 1.74 – 1.67 (m, 1H), 1.64 – 1.54 (m, 3H), 1.46 (dd, $J = 13.8, 4.6$ Hz, 3H), 1.15 – 1.05 (m, 2H), 0.61 – 0.51 (m, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 194.84, 177.84, 138.03, 129.03, 128.12, 127.41, 118.35, 79.92, 67.51, 60.65, 54.20, 48.53, 45.26, 38.65, 34.04, 28.20, 27.94, 26.50, 22.52; IR (neat, cm^{-1}): 2931, 2856, 1673, 1452, 1319, 1284, 1253, 1107, 102; HRMS (ES $^+$) calculated for $[\text{C}_{21}\text{H}_{24}\text{O}_2]^+$: 308.1776, found 308.1770.

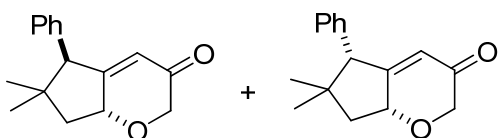
3l, 3l'



3l and **3l'** were prepared following **General Procedure C** as an inseparable 1:2 mixture, overall yield is 65%. ^1H NMR (500 MHz, CDCl_3) δ 7.36 – 7.28 (m, 3H), 7.25 – 7.21 (m, 2H), 6.11 (**3l'**, d, $J = 2.2$ Hz, 1H), 5.94 (**3l**, d, $J = 2.2$ Hz, 1H), 4.45 (**3l'**, dd, $J = 6.7, 1.7$ Hz,

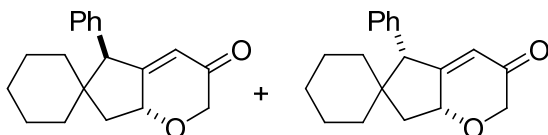
1H), 4.51 (**3l**, d, $J = 17.9$ Hz, 1H), 4.49 (**3l'**, d, $J = 17.9$ Hz, 1H), 4.23 (**3l**, d, $J = 17.8$ Hz, 1H), 4.22 (**3l'**, d, $J = 17.8$ Hz, 1H), 4.09 (**3l**, d, $J = 2.1$ Hz, 1H), 2.76 – 2.73 (**3l'**, m, 1H), 2.50 – 2.43 (**3l'**, m, 1H), 2.35 (ddd, $J = 11.9, 7.0, 3.7$ Hz, 1H), 2.04 (dd, $J = 12.4, 6.9$ Hz, 2H), 1.98 – 1.90 (m, 1H), 1.85 – 1.73 (m, 1H), 1.68 – 1.56 (m, 5H), 1.51 – 1.44 (m, 2H), 1.37 – 1.31 (m, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 195.02, 194.95, 175.85, 173.00, 136.88, 135.79, 129.32, 128.62, 128.40, 128.04, 127.22, 126.82, 121.55, 120.85, 81.30, 80.42, 67.75, 67.19, 55.91, 54.18, 53.42, 43.94, 43.56, 39.48, 38.81, 38.30, 37.84, 34.15, 34.12, 33.00, 31.37, 29.85, 28.72, 23.61, 22.45, 21.46; IR (neat, cm^{-1}): 2947, 2870, 1673, 1496, 1449, 1314, 1122, 1100; HRMS (ES+) calculated for $[\text{C}_{20}\text{H}_{22}\text{O}_2]^+$: 294.1620, found 294.1620.

3m+3m'



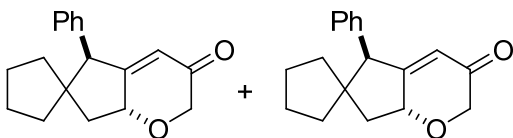
3m and **3m'** were prepared following **General Procedure C** as a 5:1 mixture, overall yield is 60%. ^1H NMR (500 MHz, CDCl_3) δ 7.36 – 7.28 (m, 3H), 7.16 – 7.12 (**3m**, m, 2H), 7.12 – 7.08 (**3m'**, m, 2H), 5.90 (**3m**, d, $J = 1.9$ Hz, 1H), 5.30 (**3m'**, s, 1H), 4.84 – 4.78 (**3m'**, m, 1H), 4.78 – 4.72 (**3m**, m, 1H), 4.33 (**3m'**, d, $J = 16.6$ Hz, 1H), 4.32 (**3m**, d, $J = 9.6$ Hz, 1H), 4.15 (**3m'**, dd, $J = 16.8, 1.8$ Hz, 1H), 4.09 (**3m**, dd, $J = 16.7, 1.7$ Hz, 2H), 3.89 – 3.87 (**3m'**, m, 1H), 3.59 (**3m**, d, $J = 2.3$ Hz, 1H), 2.19 (dt, $J = 11.6, 7.6$ Hz, 1H), 1.84 (**3m**, dd, $J = 13.7, 6.1$ Hz, 1H), 1.77 (**3m'**, t, $J = 11.5$ Hz, 1H), 1.22 (**3m'**, s, 3H), 1.14 (**3m**, s, 3H), 0.82 (**3m**, s, 3H), 0.68 (**3m'**, s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 195.43, 195.06, 174.14, 173.20, 137.70, 136.50, 130.25, 129.15, 128.36, 128.24, 127.28, 127.14, 122.33, 121.14, 78.02, 77.20, 72.91, 72.89, 60.12, 59.94, 44.79, 44.10, 42.54, 41.34, 29.63, 28.66, 25.84, 24.45; IR (neat, cm^{-1}): 2961, 2867, 1683, 1494, 1451, 1334, 1289, 1260, 1236, 1110, 1026; HRMS (ES+) calculated for $[\text{C}_{16}\text{H}_{18}\text{O}_2]^+$: 242.1307, found 242.1308.

3n+3n'

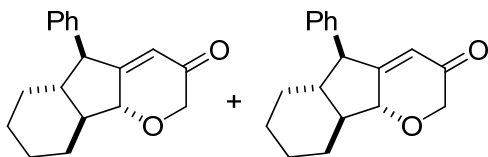


3n and **3n'** were prepared following **General Procedure C** as an inseparable 2.8:1 mixture, overall yield is 66%. ¹H NMR (500 MHz, CDCl₃) δ 7.37 – 7.26 (m, 3H), 7.13 (**3n'**, d, *J* = 7.4 Hz, 2H), 7.06 (**3n**, d, *J* = 7.2 Hz, 2H), 5.88 (**3n**, s, 1H), 5.84 (**3n'**, s, 1H), 4.81 – 4.69 (m, 1H), 4.32 (**3n**, d, *J* = 16.7 Hz, 1H), 4.30 (**3n'**, d, *J* = 16.8 Hz, 1H), 4.13 (**3n**, dd, *J* = 16.8, 2.0 Hz, 1H), 4.08 (**3n'**, dd, *J* = 16.7, 1.8 Hz, 1H), 3.79 (**3n**, t, *J* = 2.3 Hz, 1H), 3.59 (**3n'**, d, *J* = 2.2 Hz, 1H), 2.65 (**3n**, dd, *J* = 12.3, 7.7 Hz, 1H), 2.27 (**3n'**, dd, *J* = 13.7, 9.2 Hz, 1H), 1.92 (**3n'**, dd, *J* = 13.7, 7.2 Hz, 1H), 1.67 – 1.22 (m, 10H), 1.00 – 0.92 (m, 1H), 0.60 (**3n**, td, *J* = 13.3, 3.9 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 195.37, 195.15, 174.20, 137.41, 130.69, 129.60, 128.29, 128.12, 127.21, 127.12, 121.91, 121.07, 120.70, 115.26, 77.93, 76.68, 72.87, 72.66, 61.49, 60.37, 45.87, 45.25, 38.79, 38.59, 38.14, 33.94, 31.78, 25.53, 25.51, 23.56, 23.40, 22.40, 22.27; IR (neat, cm⁻¹): 2928, 2854, 1667, 1497, 1452, 1424, 1332, 1258, 1232, 1118, 1014; HRMS (ES⁺) calculated for [C₁₉H₂₂O₂]⁺: 282.1620, found 282.1616.

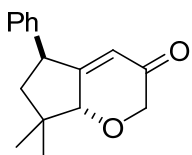
3o+3o'



3o and **3o'** were prepared following **General Procedure C** as an inseparable 4:1 mixture, overall yield is 62%. ¹H NMR (400 MHz, CDCl₃) δ 7.34 – 7.25 (m, 3H), 7.08 (t, *J* = 5.8 Hz, 2H), 5.86 (**3o**, s, 1H), 5.74 (**3o'**, s, 1H), 4.77 – 4.66 (m, 1H), 4.31 (**3o**, d, *J* = 16.7 Hz, 1H), 4.28 (**3o'**, d, *J* = 16.7 Hz, 1H), 4.13 (**3o**, dd, *J* = 16.7, 1.9 Hz, 1H), 4.08 (**3o'**, dd, *J* = 16.7, 1.8 Hz, 1H), 4.05 (**3o**, s, 1H), 3.68 (**3o'**, s, 1H), 2.27 (**3o**, dd, *J* = 11.8, 7.4 Hz, 1H), 2.14 (**3o'**, dd, *J* = 12.7, 8.2 Hz, 1H), 1.96 (**3o'**, dd, *J* = 12.7, 9.6 Hz, 1H), 1.85 – 1.76 (m, 1H), 1.73 – 1.42 (m, 5H), 1.27 – 1.08 (m, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 195.24, 195.04, 174.61, 174.27, 139.59, 138.15, 129.99, 129.54, 128.39, 128.34, 127.13, 127.06, 121.81, 121.80, 78.32, 77.11, 72.75, 72.42, 57.50, 57.48, 52.90, 52.25, 42.96, 42.55, 41.02, 38.38, 34.96, 33.34, 24.46, 23.48, 23.47, 22.97; IR (neat, cm⁻¹): 3029, 2957, 2867, 1675, 1494, 1452, 1333, 1259, 1233, 1112, 1049, 1032; HRMS (ES⁺) calculated for [C₁₈H₂₀O₂]⁺: 268.1463, found 268.1461.

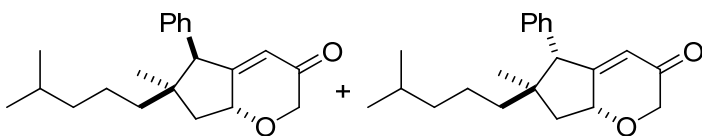
3p+3p'

3p and **3p'** were prepared following **General Procedure C** as an inseparable 4:1 mixture, overall yield is 71%. ^1H NMR (500 MHz, CDCl_3) δ 7.33 (dd, $J = 10.2, 4.7$ Hz, 2H), 7.28 – 7.24 (m, 1H), 7.14 – 7.09 (m, 2H), 5.93 (**3p'**, s, 1H), 5.86 (**3p**, t, $J = 2.0$ Hz, 1H), 4.79 – 4.74 (**3p'**, m, 1H), 4.31 (d, $J = 16.6$ Hz, 1H), 4.29 – 4.26 (**3p**, m, 1H), 4.11 (**3p'**, dd, $J = 16.5, 2.2$ Hz, 1H), 4.10 (**3p**, dd, $J = 16.6, 1.9$ Hz, 1H), 3.90 (**3p'**, d, $J = 11.0$ Hz, 1H), 3.51 (**3p**, dt, $J = 10.9, 2.5$ Hz, 1H), 2.42 (**3p'**, td, $J = 11.9, 5.4$ Hz, 1H), 2.25 (**3p'**, dd, $J = 13.8, 9.1$ Hz, 1H), 2.20 (**3p**, dd, $J = 8.1, 4.0$ Hz, 1H), 1.93 – 1.88 (**3p**, m, 1H), 1.88 – 1.73 (m, 2H), 1.59 – 1.45 (m, 2H), 1.31 – 1.16 (m, 4H); ^{13}C NMR (126 MHz, CDCl_3) δ 195.18, 194.89, 174.30, 173.25, 141.79, 140.58, 128.82, 128.76, 128.21, 128.07, 127.02, 126.97, 123.64, 122.32, 82.95, 81.97, 72.37, 72.22, 54.18, 49.32, 48.59, 48.47, 43.54, 41.02, 29.74, 29.36, 25.78, 25.39, 24.25, 23.75, 21.98, 20.43; IR (neat, cm^{-1}): 3085, 3061, 3028, 2928, 2855, 1682, 1495, 1450, 1318, 1271, 1259, 1235, 1154, 1012; HRMS (ES+) calculated for $[\text{C}_{18}\text{H}_{20}\text{O}_2]^+$: 268.1463, found 268.1464.

3q

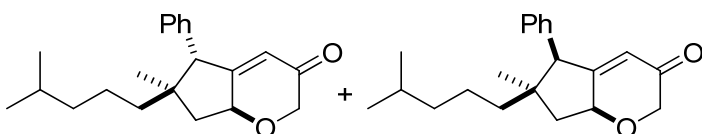
3q was prepared following **General Procedure C** in 53% yield. ^1H NMR (600 MHz, CDCl_3) δ 7.30 (t, $J = 6.7$ Hz, 2H), 7.22 (t, $J = 7.3$ Hz, 1H), 7.13 (d, $J = 7.3$ Hz, 2H), 5.93 (s, 1H), 4.30 (d, $J = 2.5$ Hz, 1H), 4.29 (d, $J = 16.3$ Hz, 1H), 4.08 (dd, $J = 16.4, 2.0$ Hz, 1H), 4.00 (dd, $J = 10.7, 8.7$ Hz, 1H), 2.15 (dd, $J = 13.2, 8.7$ Hz, 1H), 1.74 – 1.69 (m, 1H), 1.24 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 194.94, 173.16, 142.18, 128.85, 127.41, 126.89, 123.30, 86.06, 71.85, 46.56, 45.28, 40.56, 26.20, 20.15; IR (neat, cm^{-1}): 2962, 2868, 1675, 1495, 1455, 1268, 1228, 1136, 1031; HRMS (ES+) calculated for $[\text{C}_{16}\text{H}_{18}\text{O}_2]^+$: 242.1307, found 242.1306.

(5S, 6R, 7aR)-3r + (5R, 6R, 7aR)-3r



^1H NMR (500 MHz, CDCl_3) δ 7.37 – 7.27 (m, 3H), 7.14 (**5R**, d, $J = 7.5$ Hz, 2H), 7.11 (**5S**, d, $J = 7.5$ Hz, 2H), 5.92 (**5S**, s, 1H), 5.89 (**5R**, s, 1H), 4.75 – 4.67 (m, 1H), 4.34 (**5S**, d, $J = 8.0$ Hz, 1H), 4.30 (**5R**, d, $J = 8.0$ Hz, 1H), 4.14 (**5S**, d, $J = 16.7$ Hz, 1H), 4.08 (**5R**, d, $J = 16.7$ Hz, 1H), 3.91 (**5S**, s, 1H), 3.64 (**5R**, s, 1H), 2.38 (**5S**, dd, $J = 12.3, 7.5$ Hz, 1H), 2.27 (**5R**, dd, $J = 13.9, 9.5$ Hz, 1H), 1.74 (**5R**, dd, $J = 13.9, 6.0$ Hz, 1H), 1.61 – 1.50 (m, 2H), 1.34 (pt, $J = 13.9, 6.1$ Hz, 3H), 1.15 (p, $J = 6.5, 6.1$ Hz, 1H), 1.09 – 0.92 (m, 1H), 0.87 (**5S**, dd, $J = 6.7, 3.0$ Hz, 6H), 0.81 (s, 3H), 0.79 (**5R**, t, $J = 7.1$ Hz, 6H), 0.63 (**5S**, t, $J = 12.2$ Hz, 1H); ^{13}C NMR (126 MHz, CDCl_3) δ 195.43, 195.12, 173.97, 173.23, 137.01, 136.39, 130.40, 129.33, 128.35, 128.22, 127.25, 127.16, 122.16, 121.01, 77.71, 76.58, 72.96, 72.86, 61.86, 59.06, 45.76, 44.30, 42.46, 41.87, 40.71, 39.63, 39.60, 35.98, 27.95, 27.88, 25.58, 23.38, 22.64, 22.62, 22.55, 22.47, 22.43, 21.98; IR (neat, cm^{-1}): 2956, 2932, 2868, 1678, 1495, 1453, 1381, 1333, 1259, 1233, 1115; HRMS (ES⁺) calculated for $[\text{C}_{21}\text{H}_{28}\text{O}_2]^+$: 312.2089, found 312.2086.

(5R, 6R, 7aS)-3r + (5S, 6R, 7aS)-3r



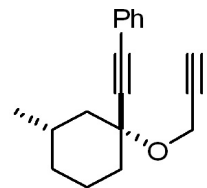
^1H NMR (500 MHz, CDCl_3) δ 7.30 (q, $J = 9.5, 8.3$ Hz, 3H), 7.12 (**5S**, d, $J = 7.7$ Hz, 2H), 7.08 (**5R**, d, $J = 7.5$ Hz, 2H), 5.89 (**5R**, s, 1H), 5.84 (**5S**, s, 1H), 4.84 – 4.74 (m, 1H), 4.33 (**5R**, d, $J = 16.7$ Hz, 1H), 4.31 (**5S**, d, $J = 16.8$ Hz, 1H), 4.14 (**5R**, d, $J = 16.7$ Hz, 1H), 4.09 (**5S**, d, $J = 16.7$ Hz, 1H), 3.92 (**5R**, d, $J = 2.9$ Hz, 1H), 3.64 (**5S**, d, $J = 2.3$ Hz, 1H), 2.16 (**5R**, dd, $J = 11.9, 7.5$ Hz, 1H), 2.09 (**5S**, dd, $J = 13.4, 8.8$ Hz, 1H), 1.92 (**5S**, dd, $J = 13.4, 7.7$ Hz, 1H), 1.74 (**5R**, t, $J = 11.4$ Hz, 1H), 1.54 (dq, $J = 21.6, 7.6$ Hz, 2H), 1.46 – 1.38 (m, 1H), 1.32 (p, $J = 7.6$ Hz, 2H), 1.25 – 1.15 (m, 2H), 0.88 (**5R**, dd, $J = 6.8, 2.8$ Hz, 6H), 0.77 (**5S**, d, $J = 6.6$ Hz, 6H), 0.68 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 195.06, 195.05, 174.20, 171.10, 137.90, 137.43, 130.45, 129.39, 128.29, 128.17, 127.16, 127.08, 122.34, 121.61, 89.23, 78.08, 72.89, 72.73, 60.72, 60.37, 59.37, 45.12, 44.47, 42.05, 41.99, 41.51,

39.61, 39.58, 38.23, 27.89, 27.23, 22.64, 22.54, 22.51, 22.38, 22.08, 21.03, 14.19; IR (neat, cm^{-1}): 2958, 2868, 1674, 1495, 1453, 1383, 1334, 1287, 1259, 1118; HRMS (ES+) calculated for $[\text{C}_{21}\text{H}_{28}\text{O}_2]^+$: 312.2089, found 312.2089.

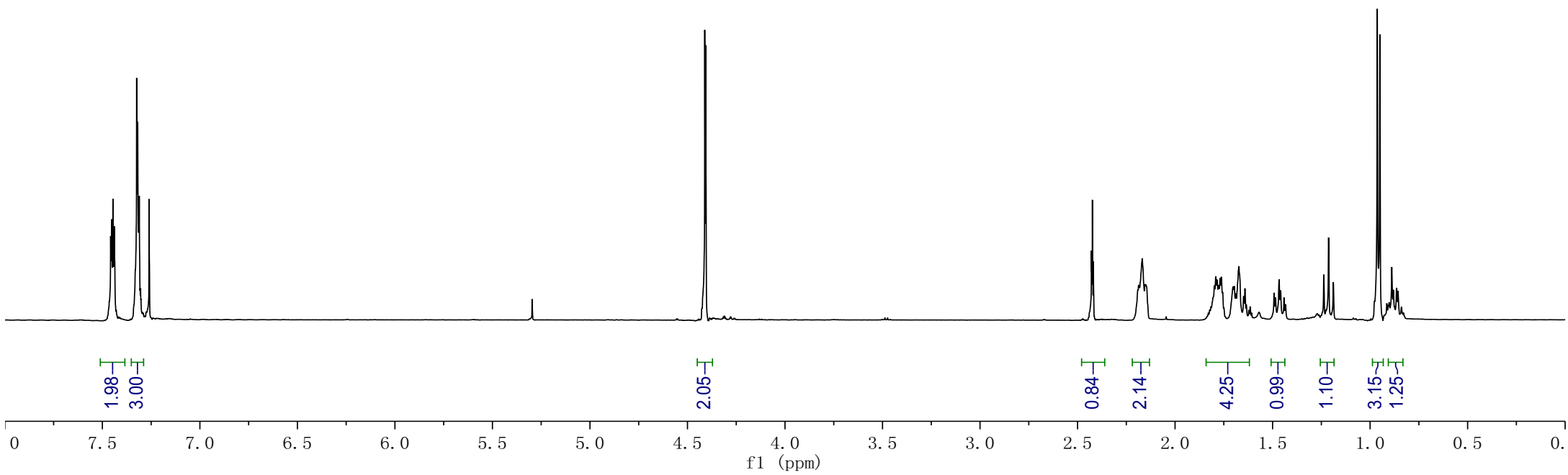
Reference

1. (a) D.E. Frantz, R. Fässler, E.M. Carreira *J. Am. Chem. Soc.* **2000**, *122*, 1806; (b) D. Boyall, D. Frantz, E.M. Carreira *Org. Lett.* **2002**, *4*, 2605.

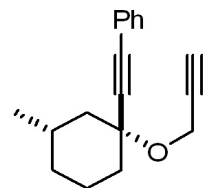
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2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



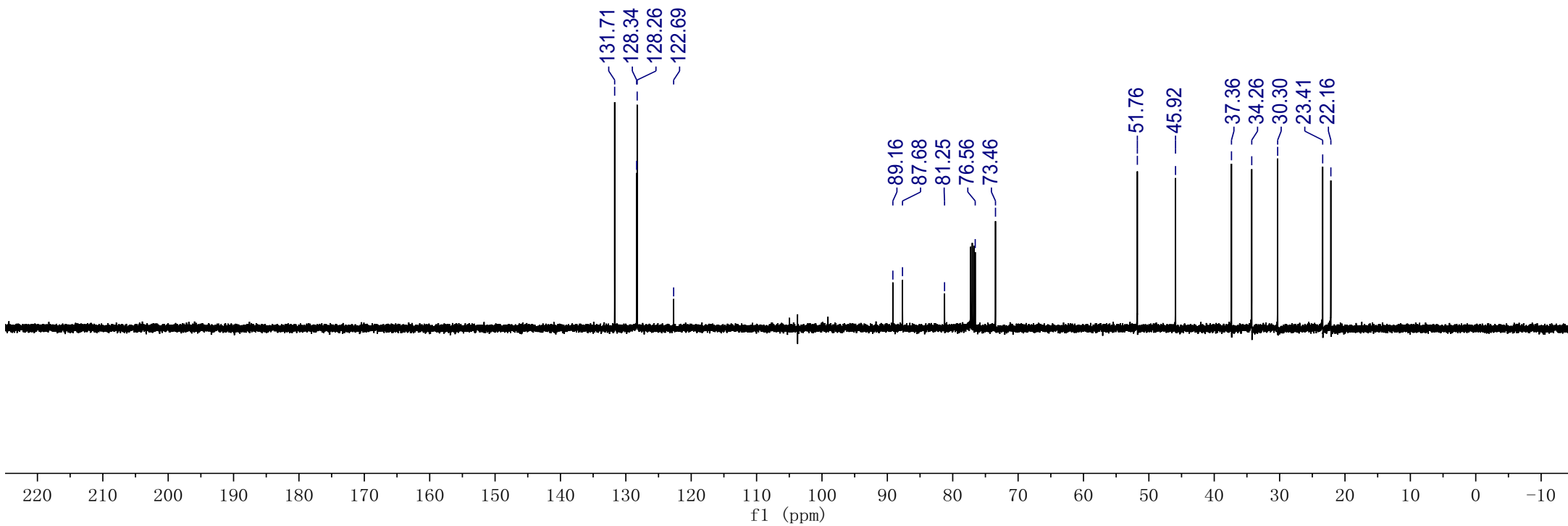
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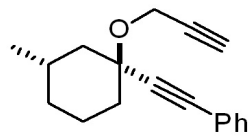
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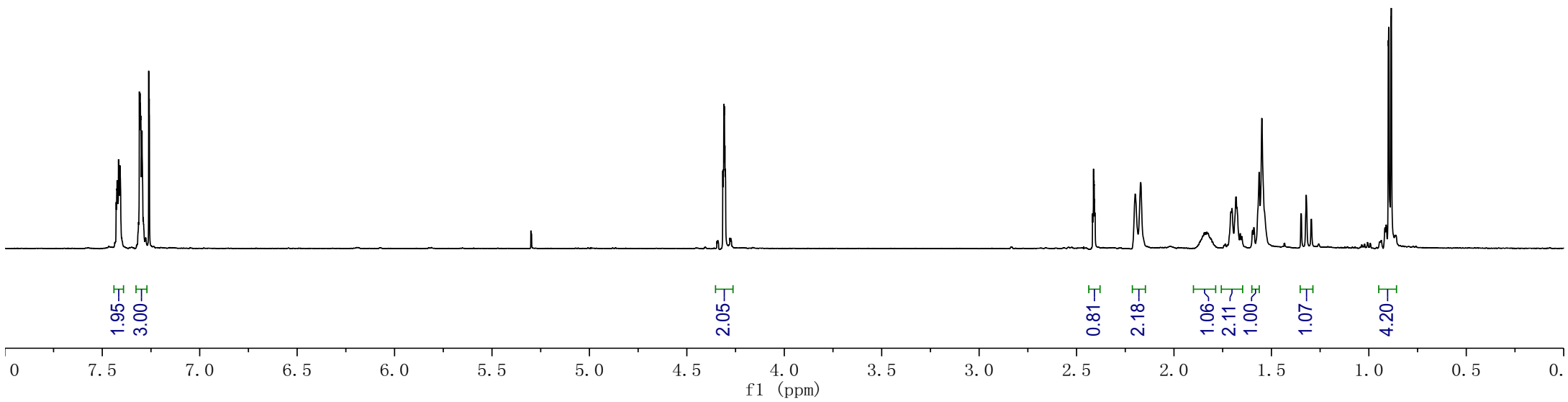
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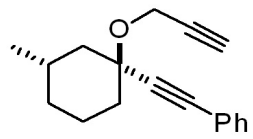
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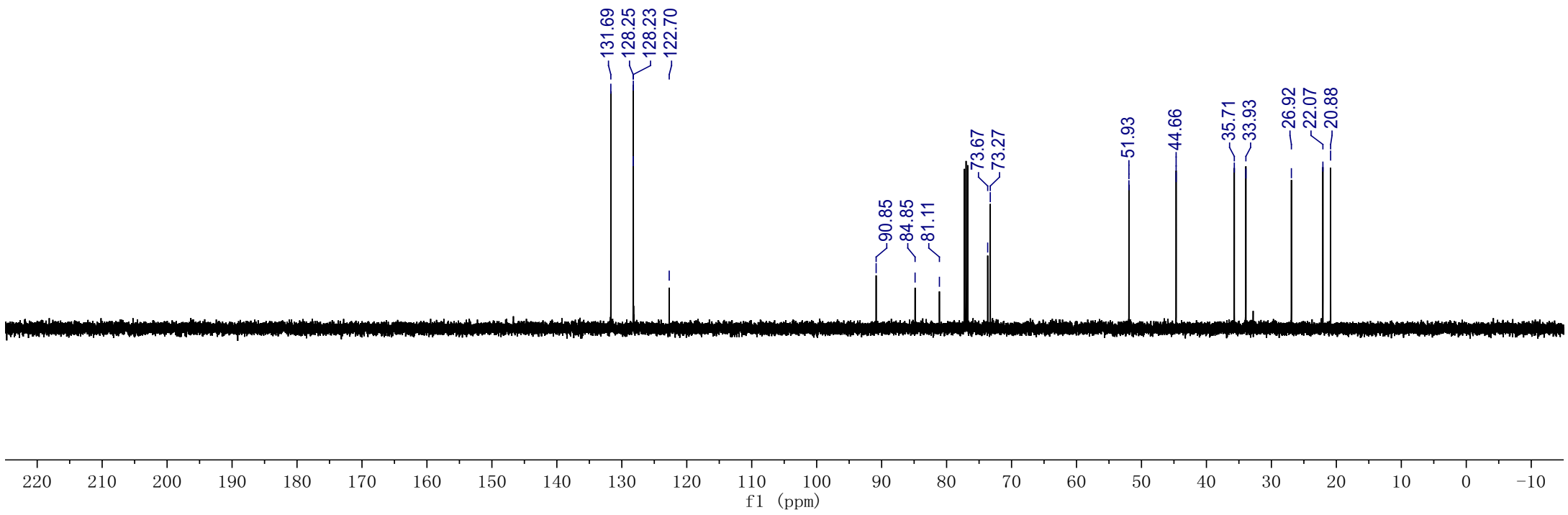
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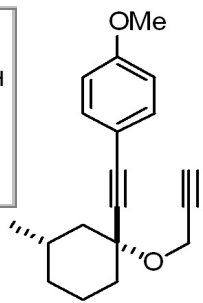
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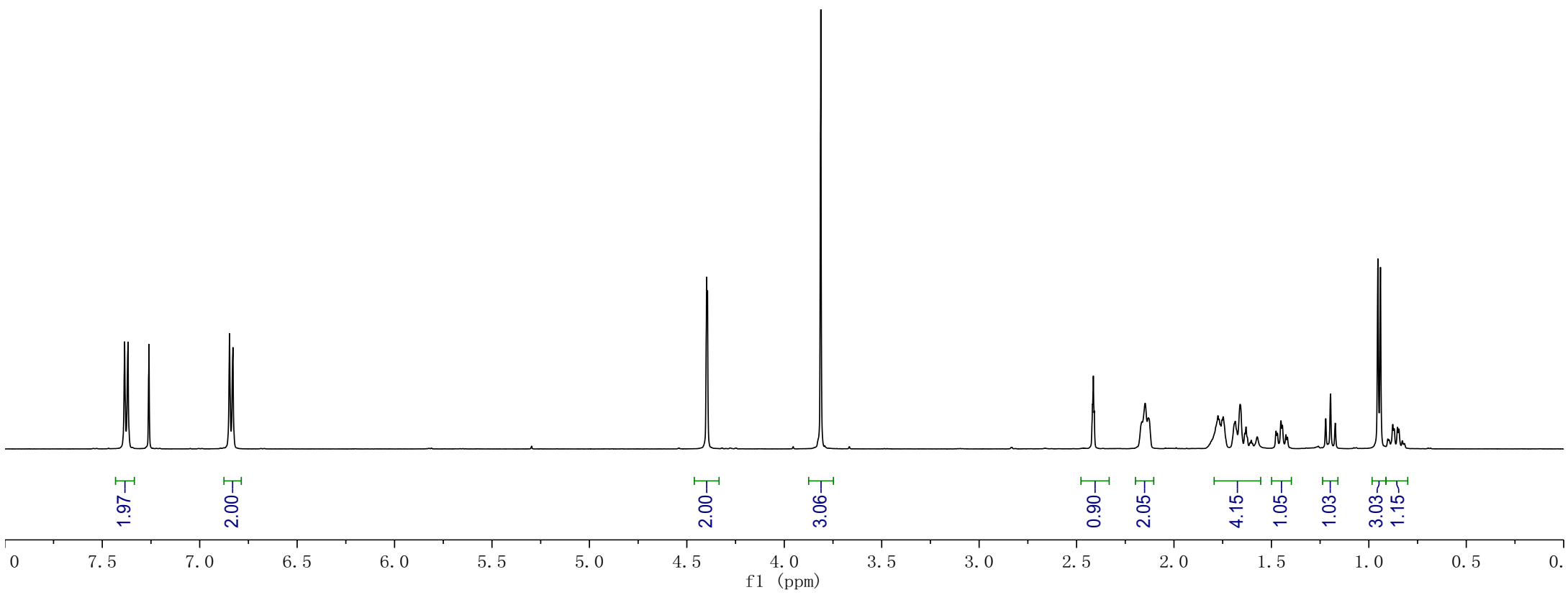
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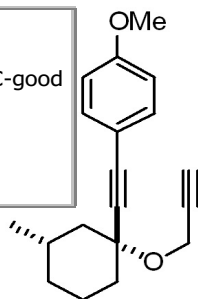
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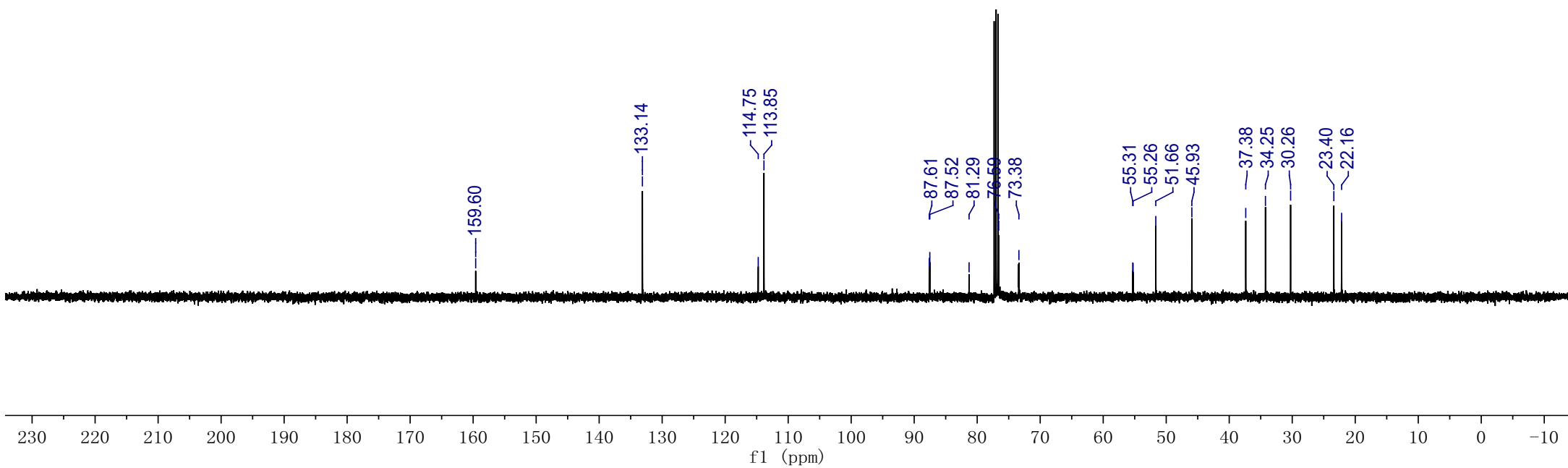
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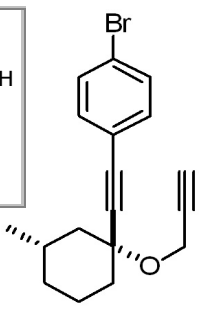
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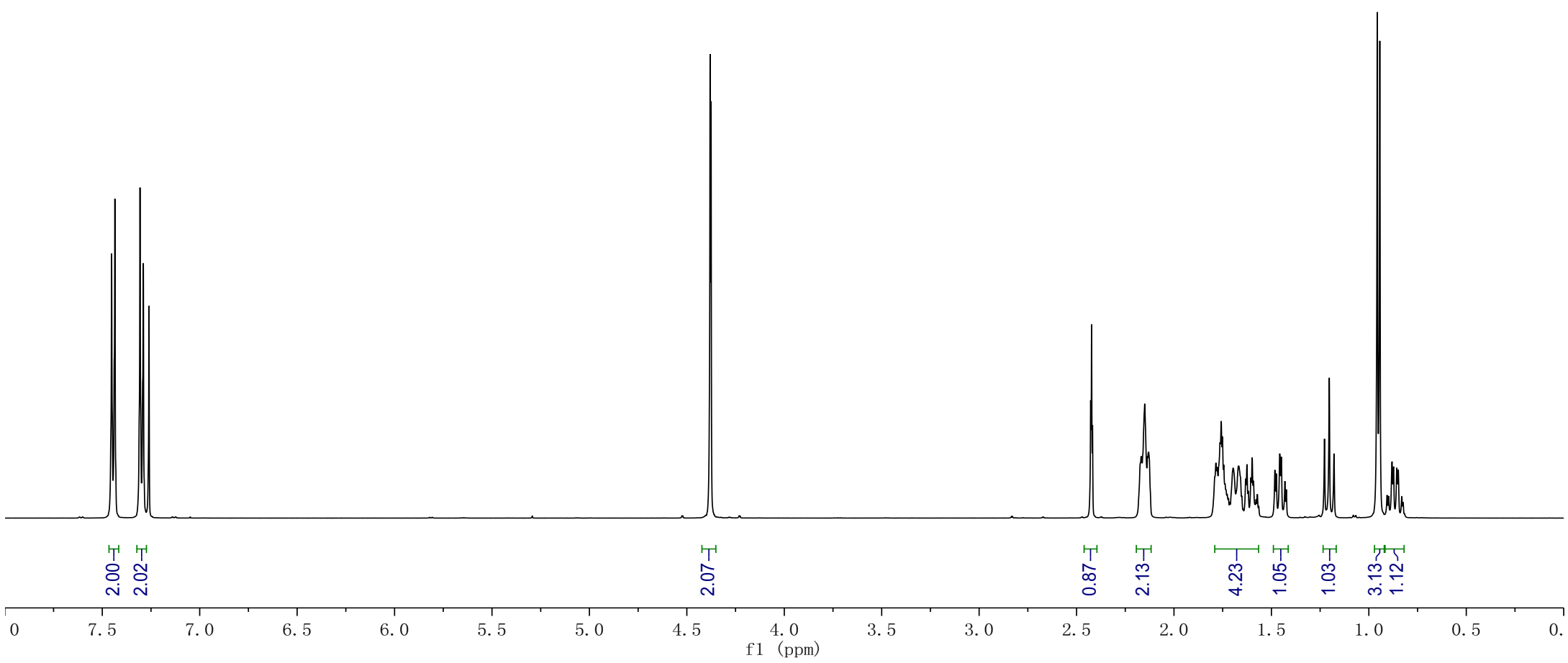
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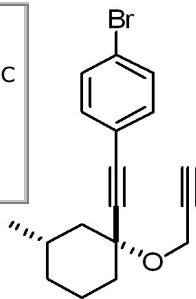
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4 Spectrometer Frequency	499.86



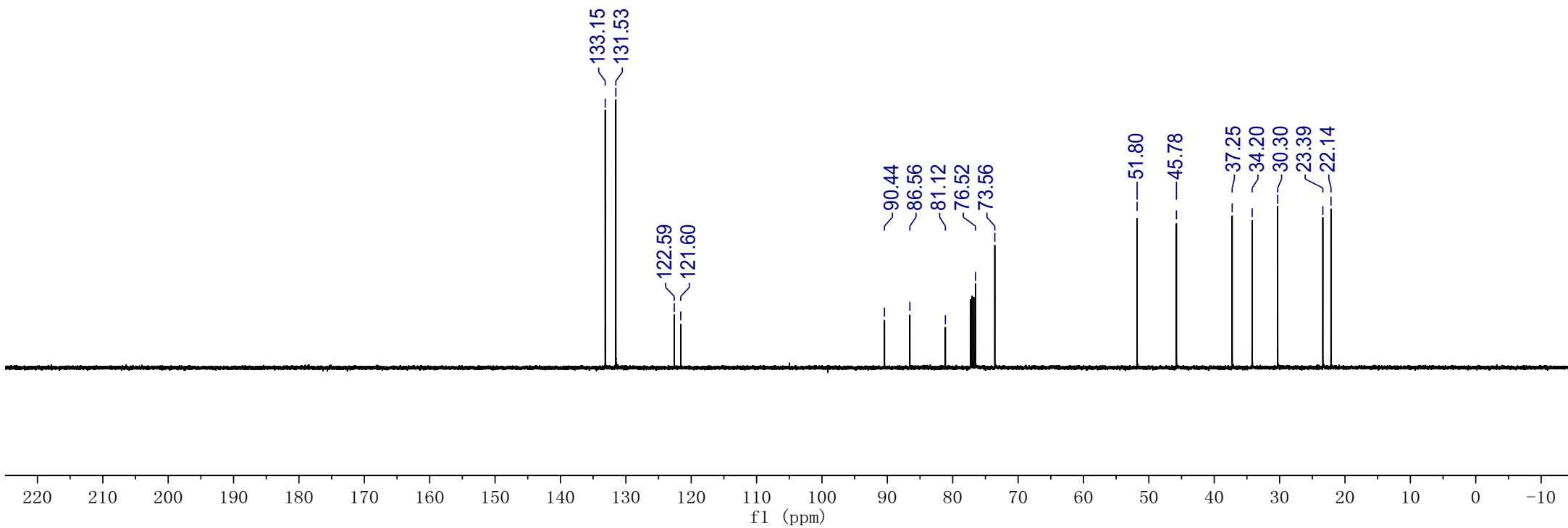
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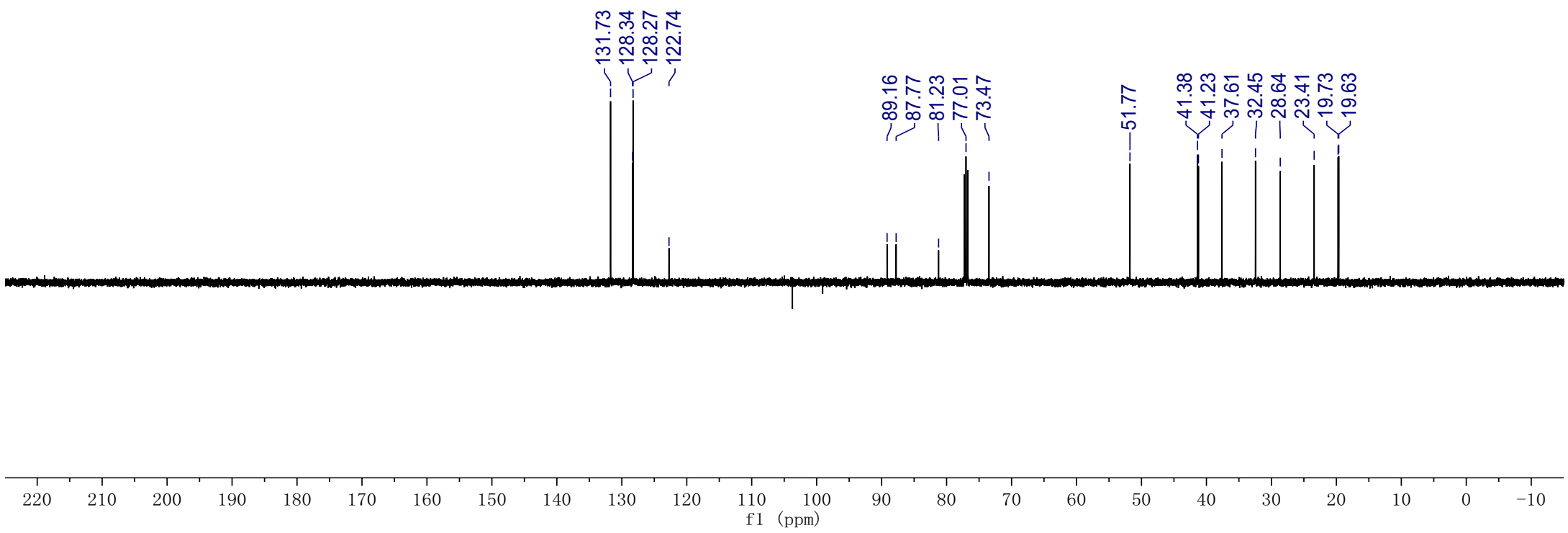
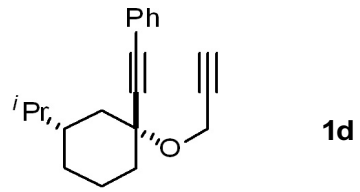
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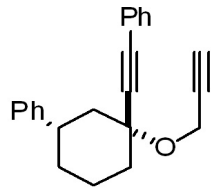
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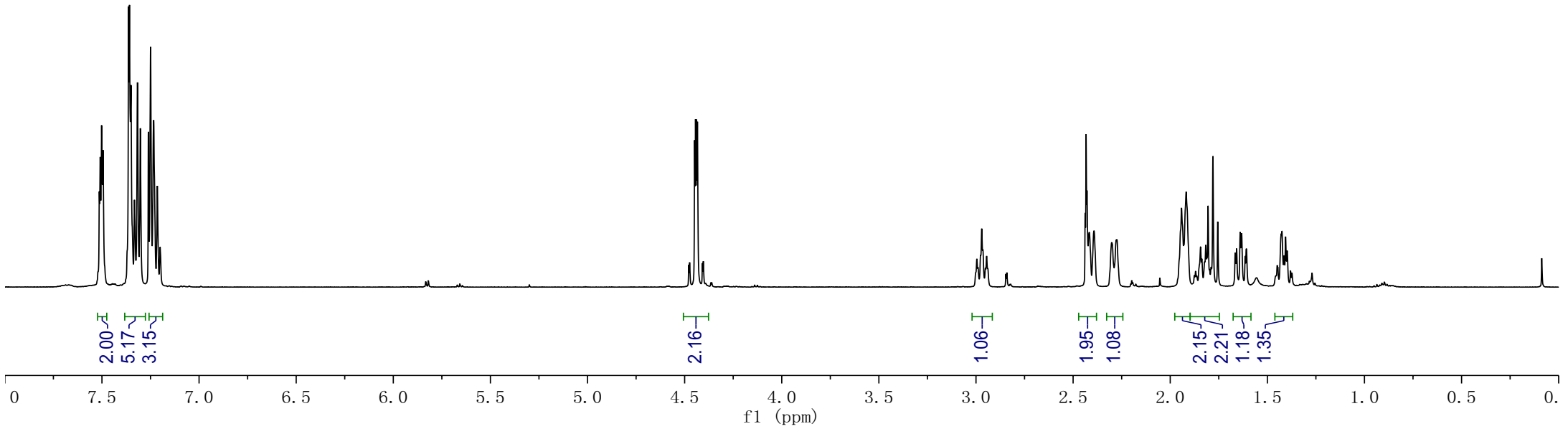
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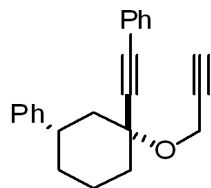
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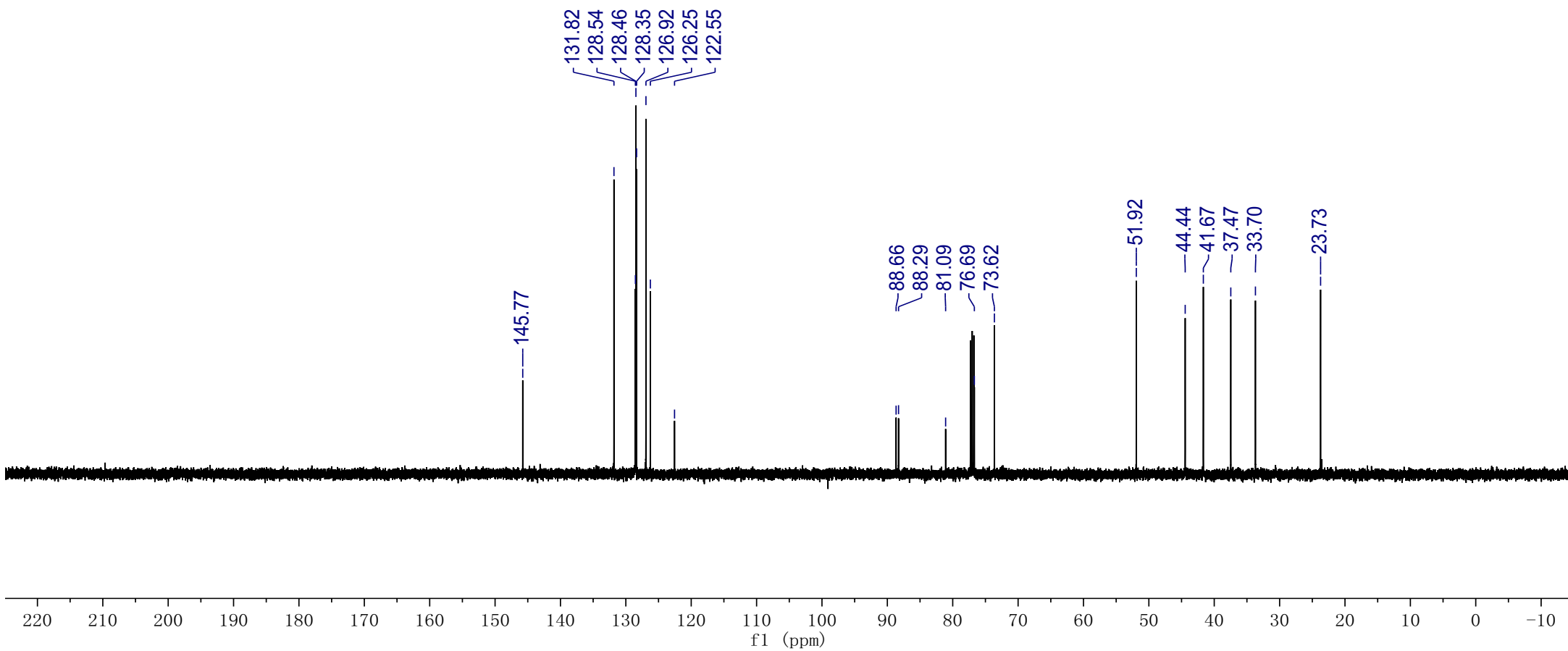
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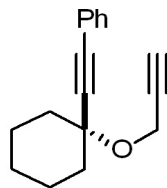
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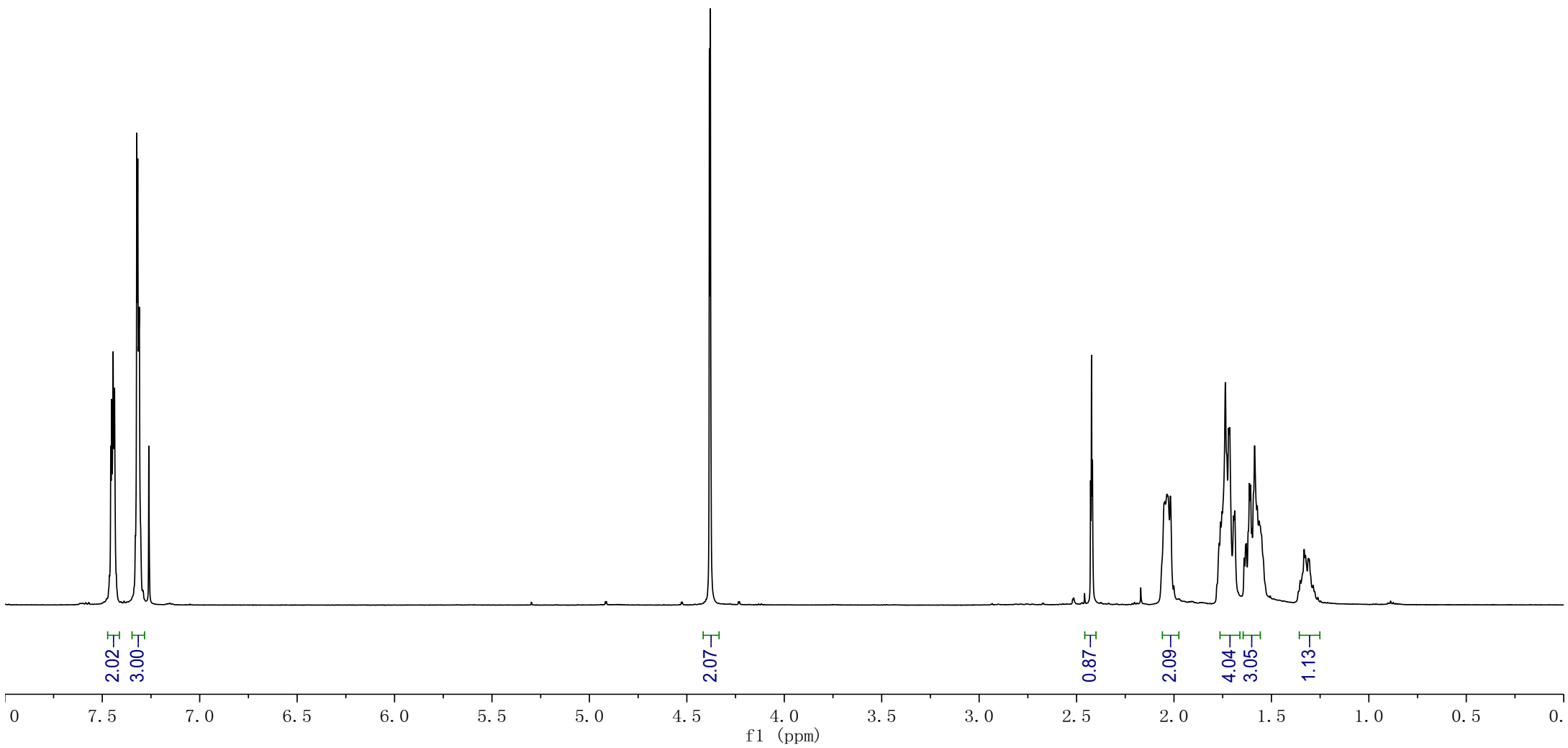
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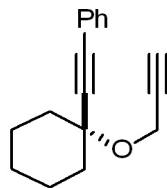
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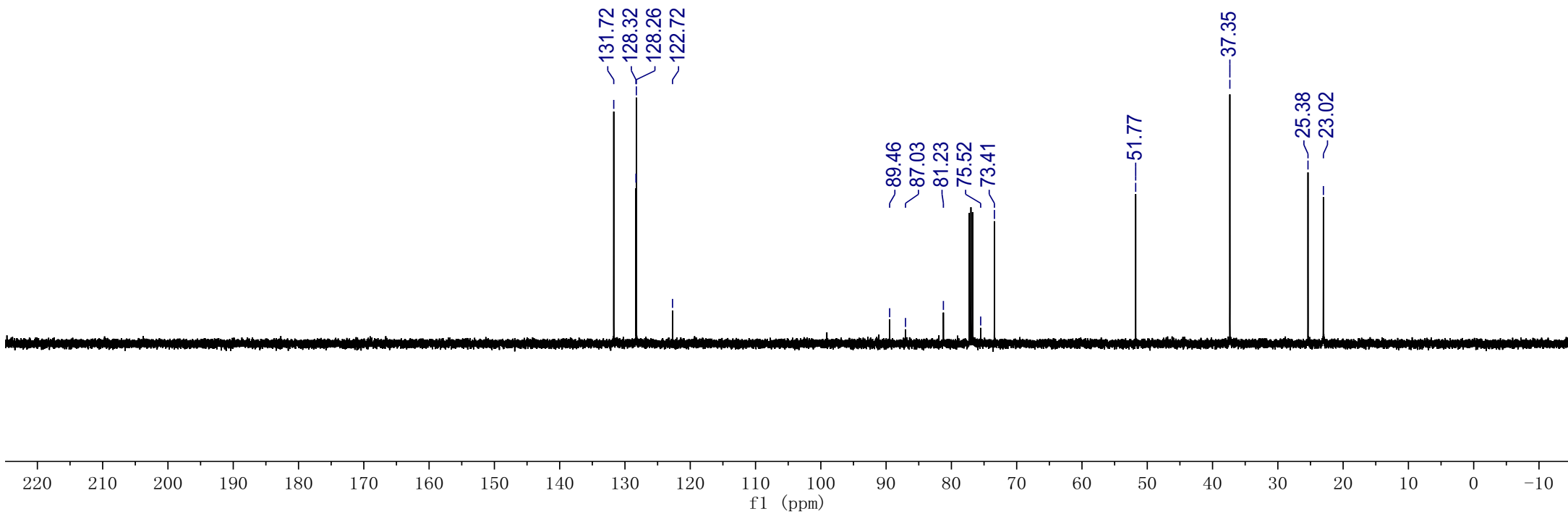
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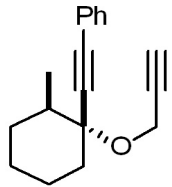
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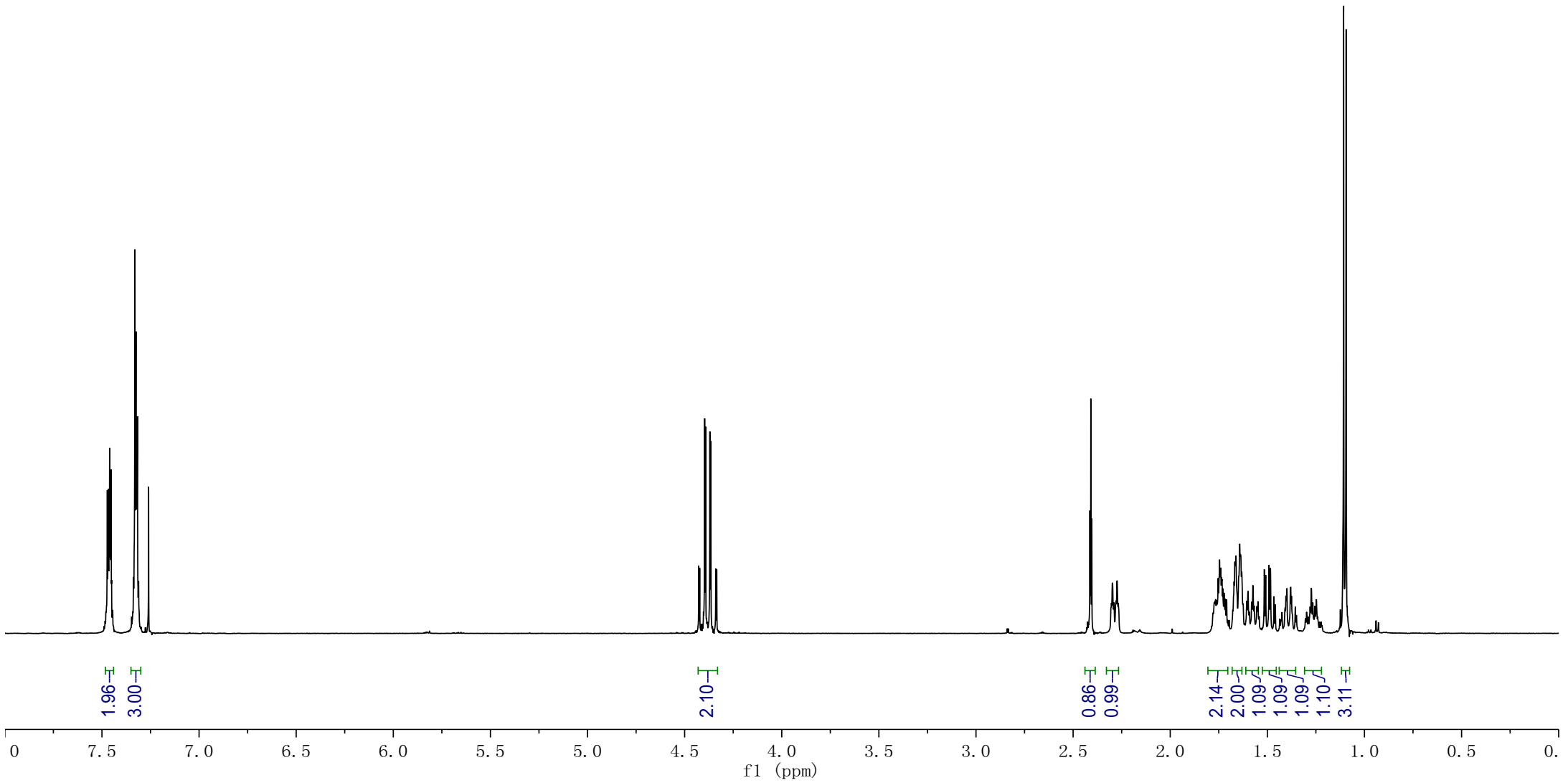
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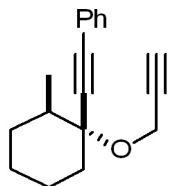
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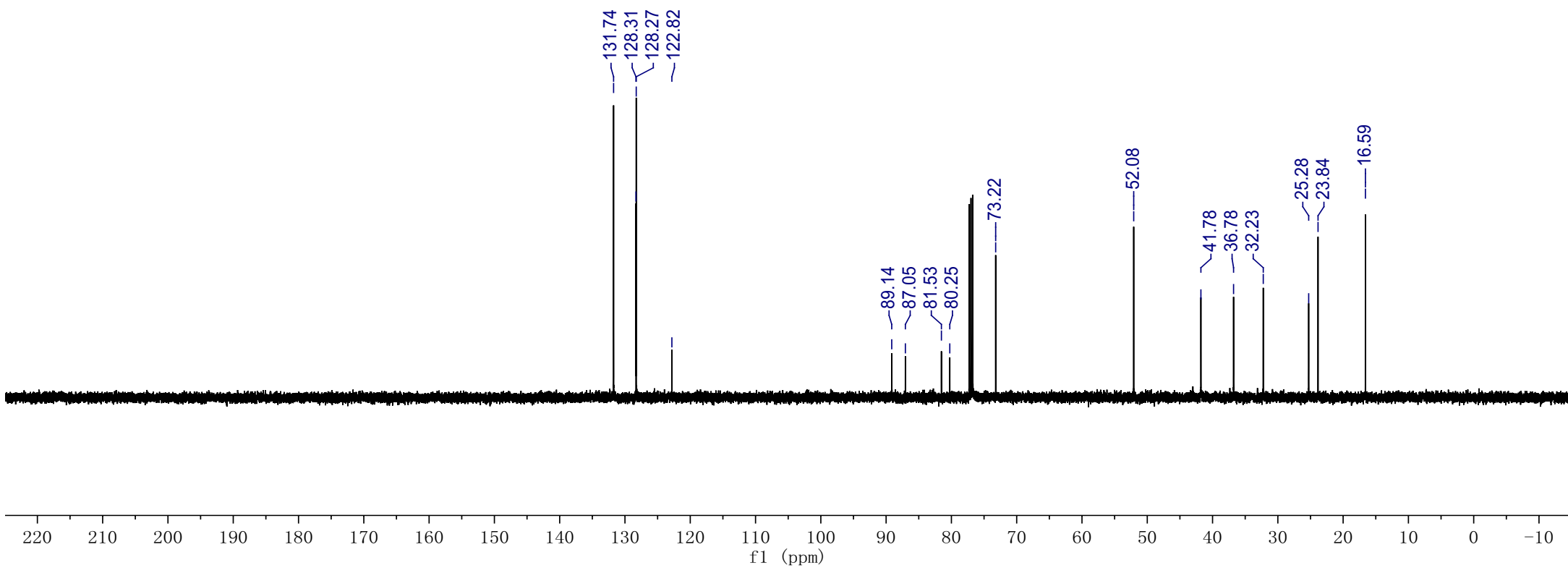
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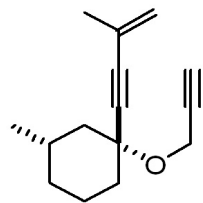
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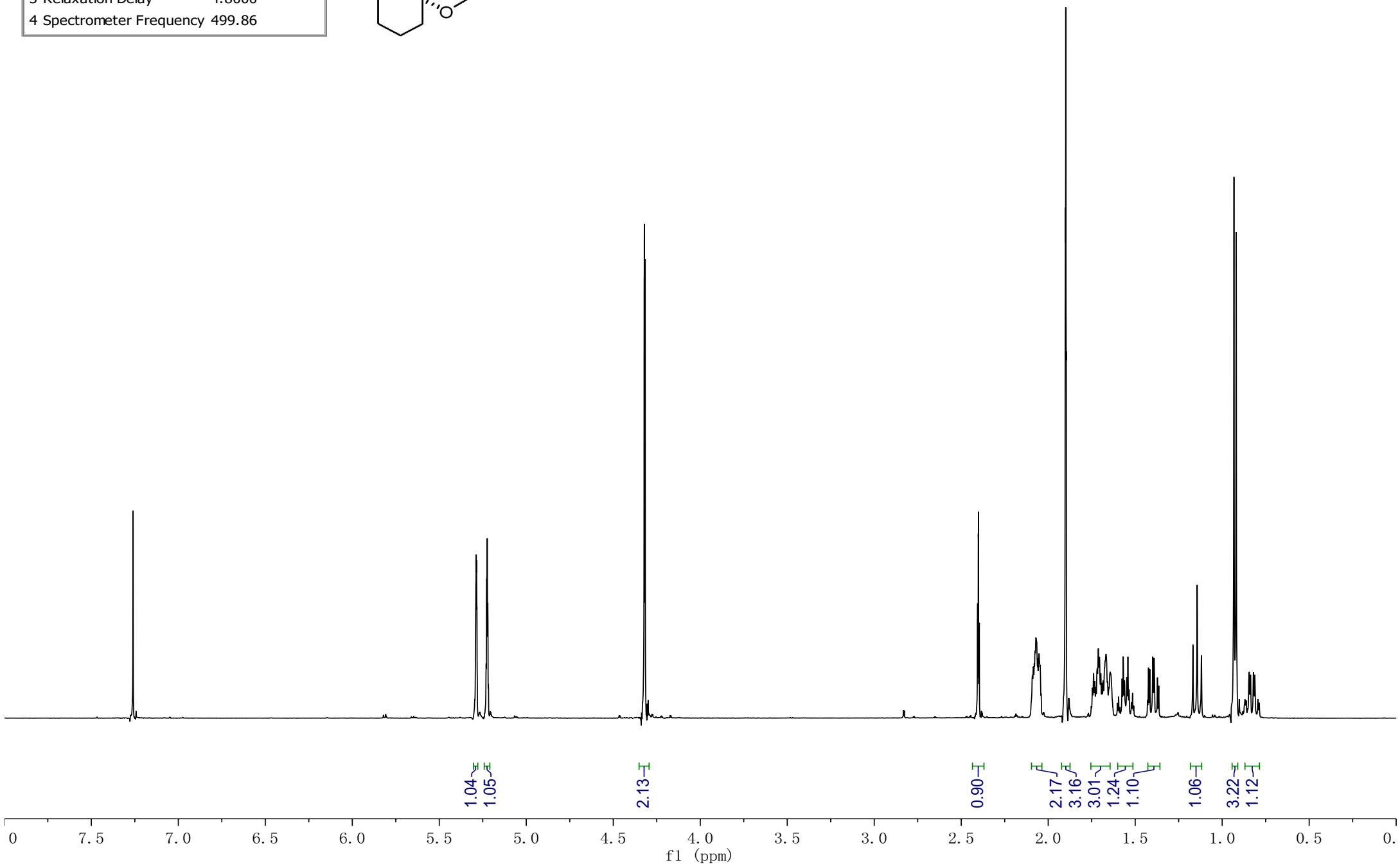
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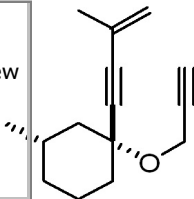
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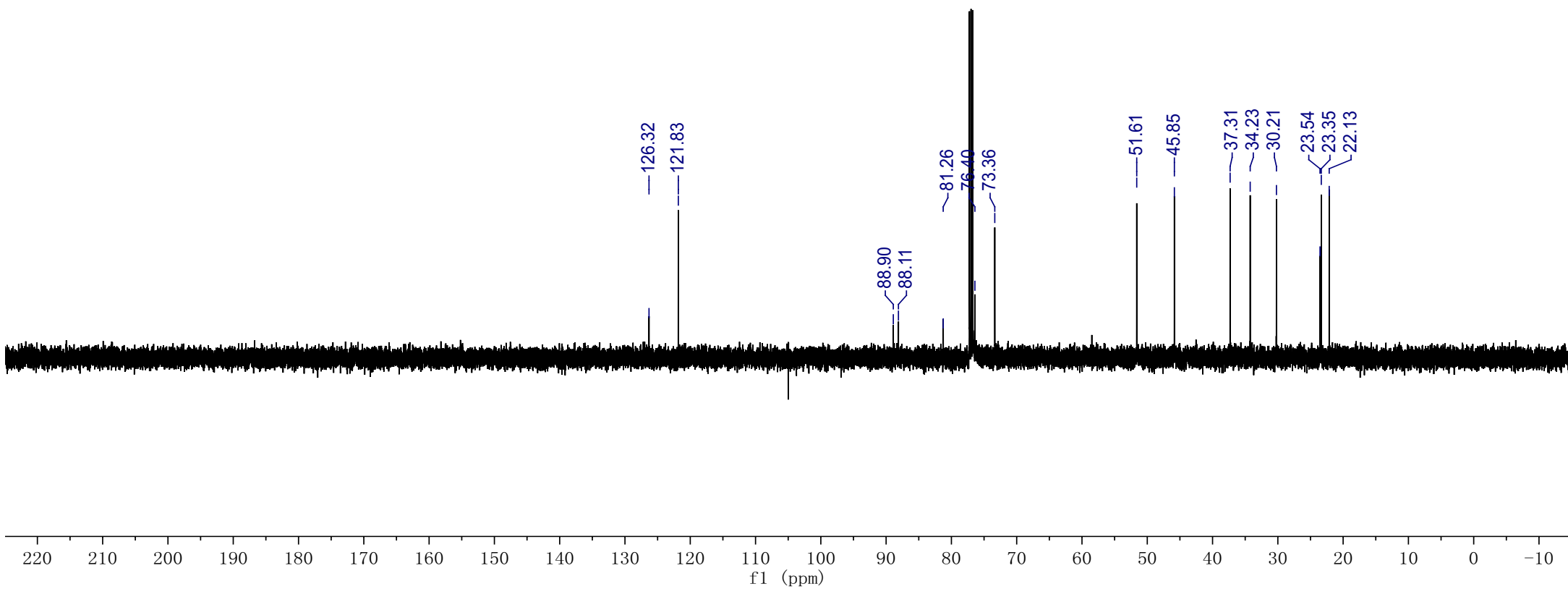
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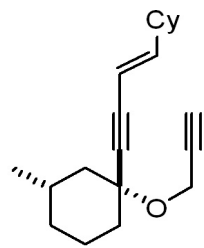
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4 Nucleus	13C



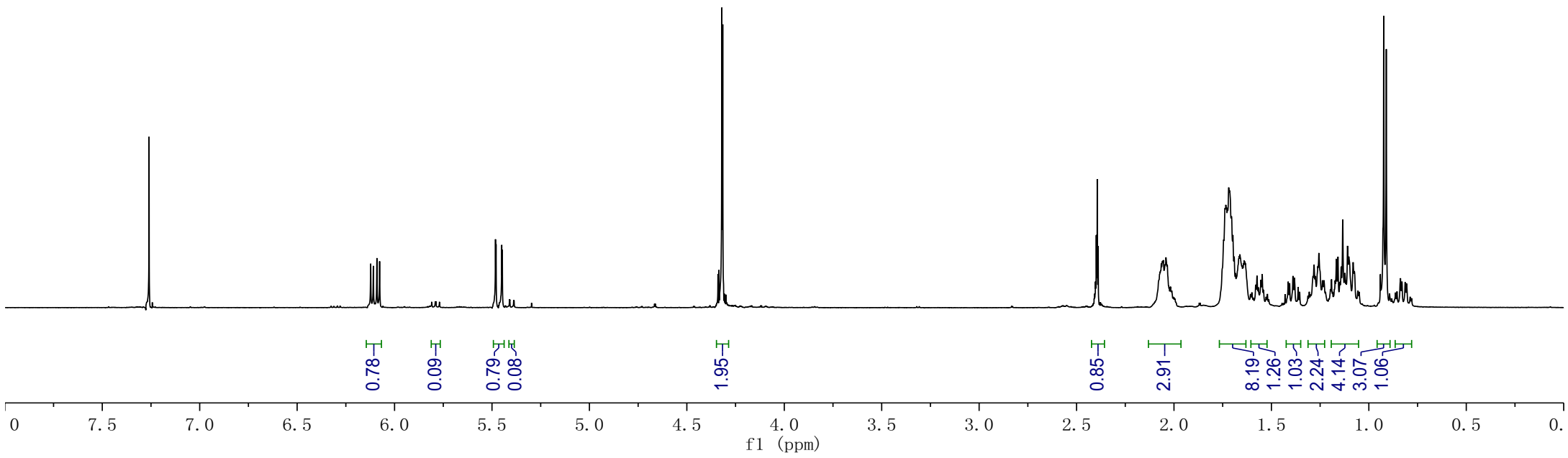
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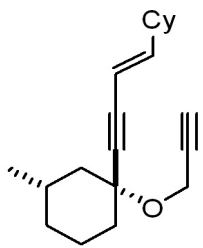
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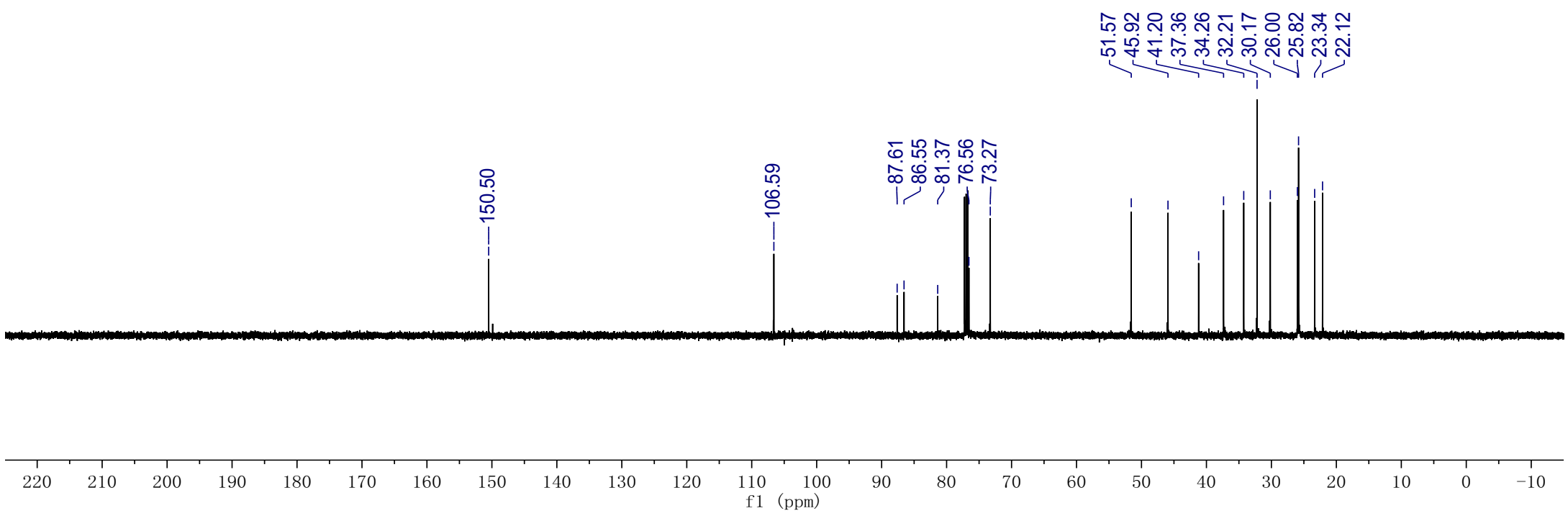
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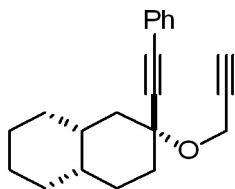
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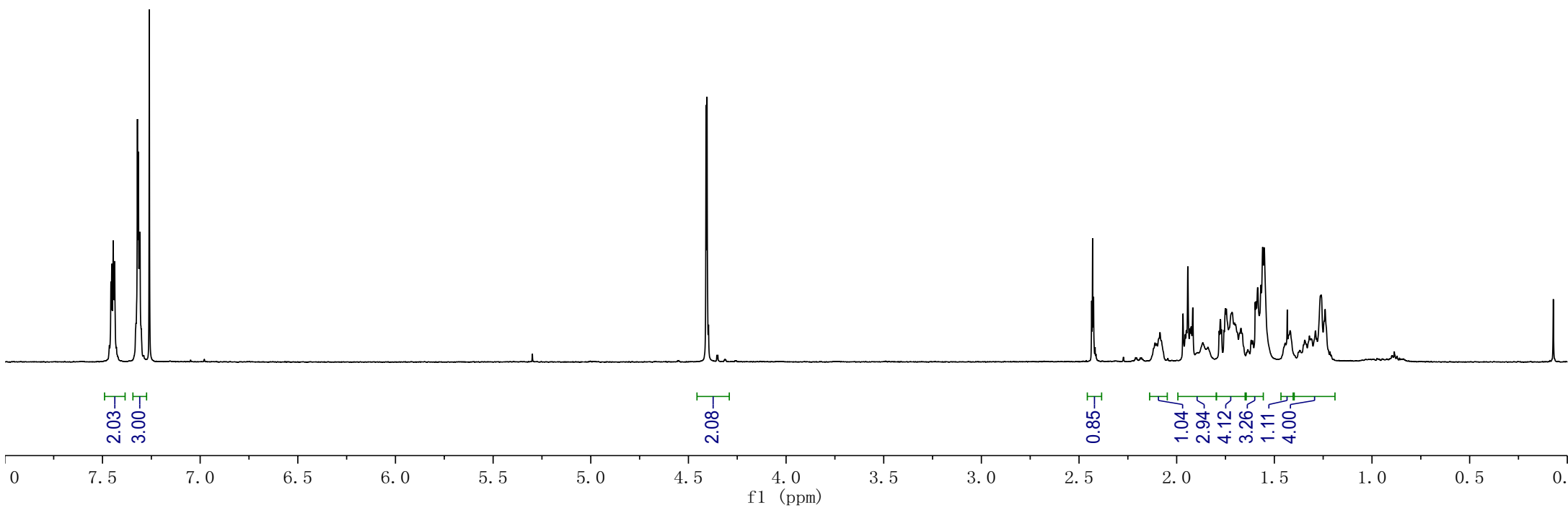
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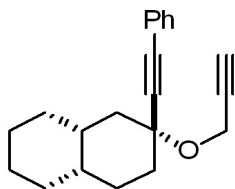
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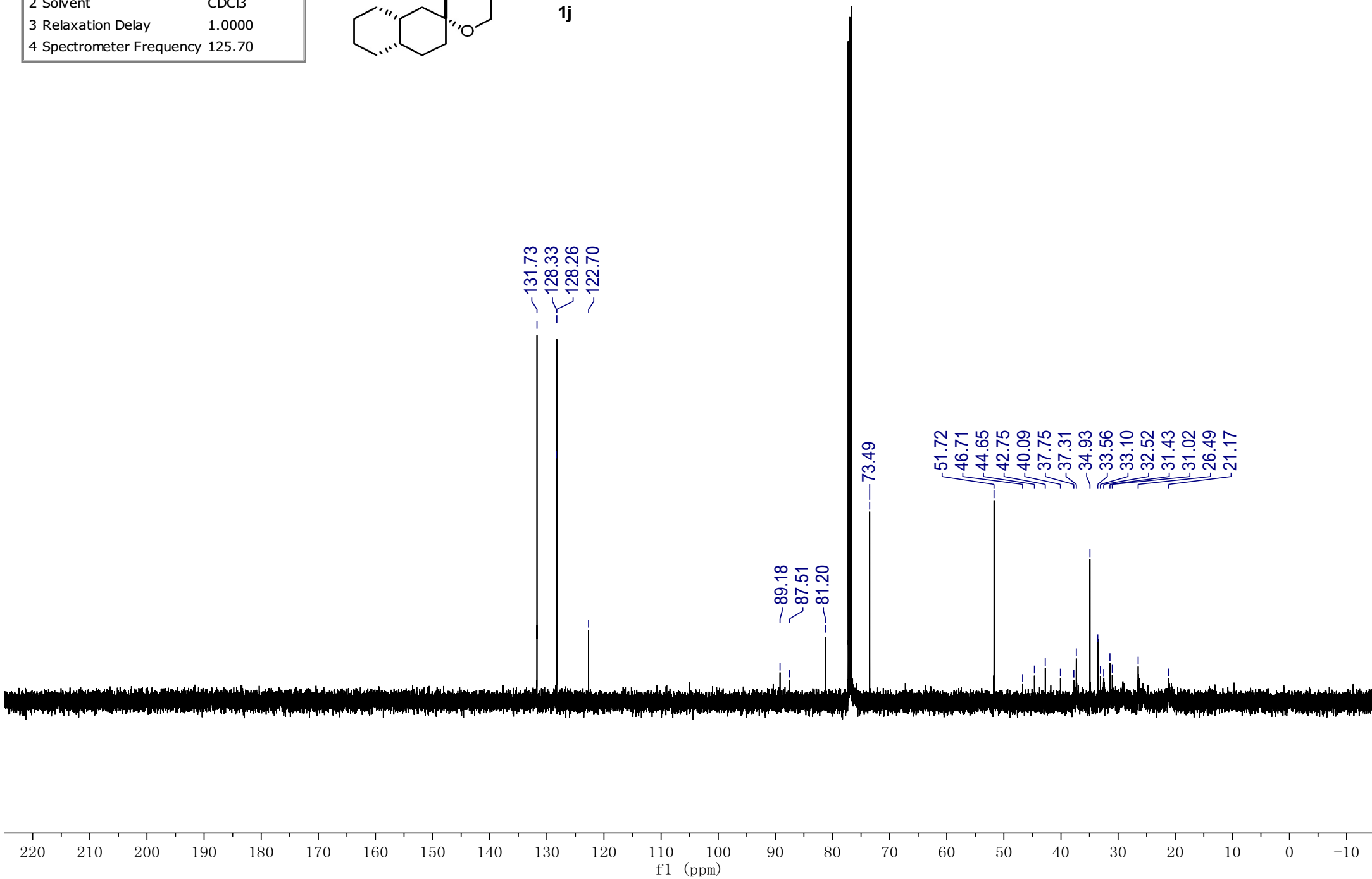
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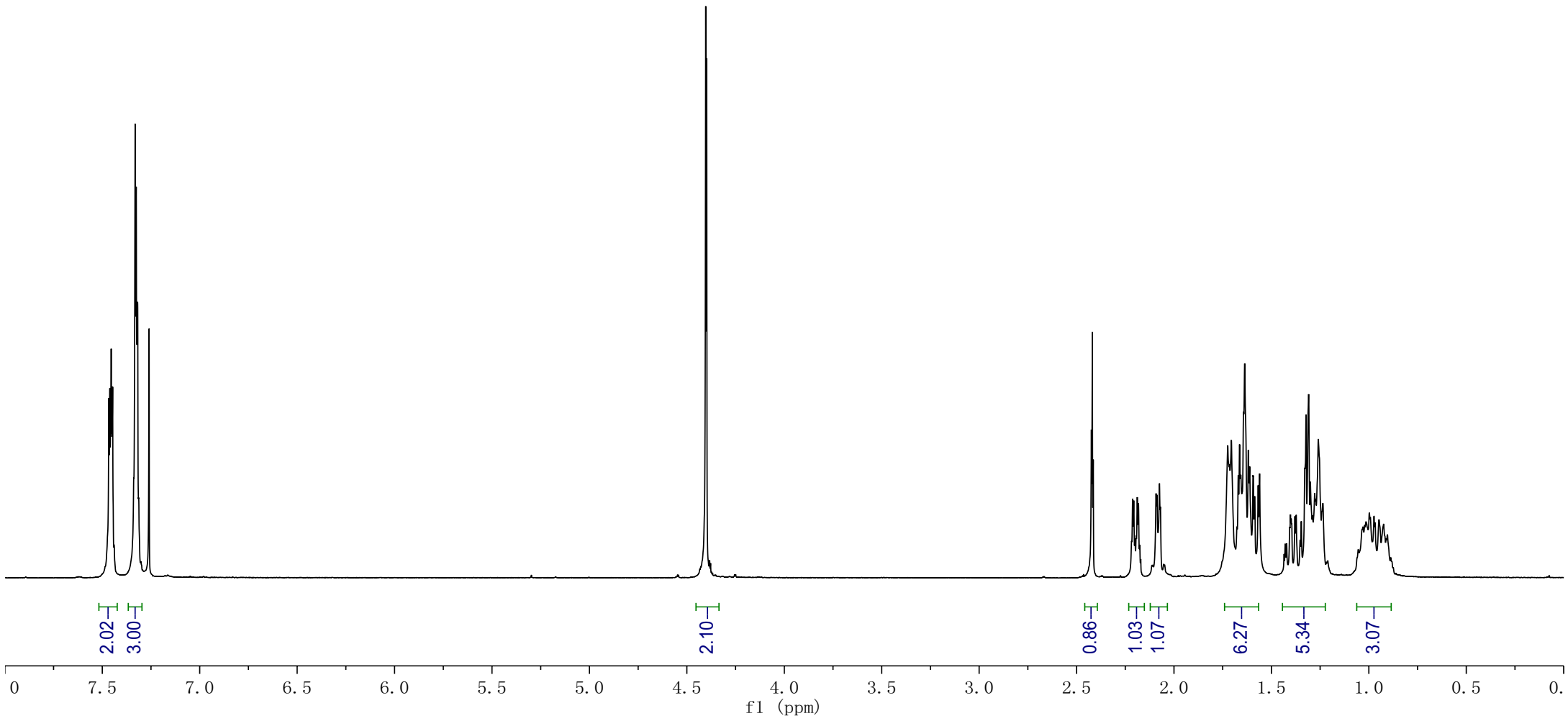
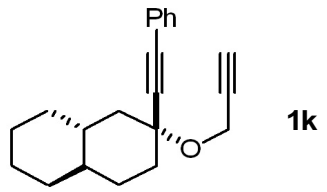
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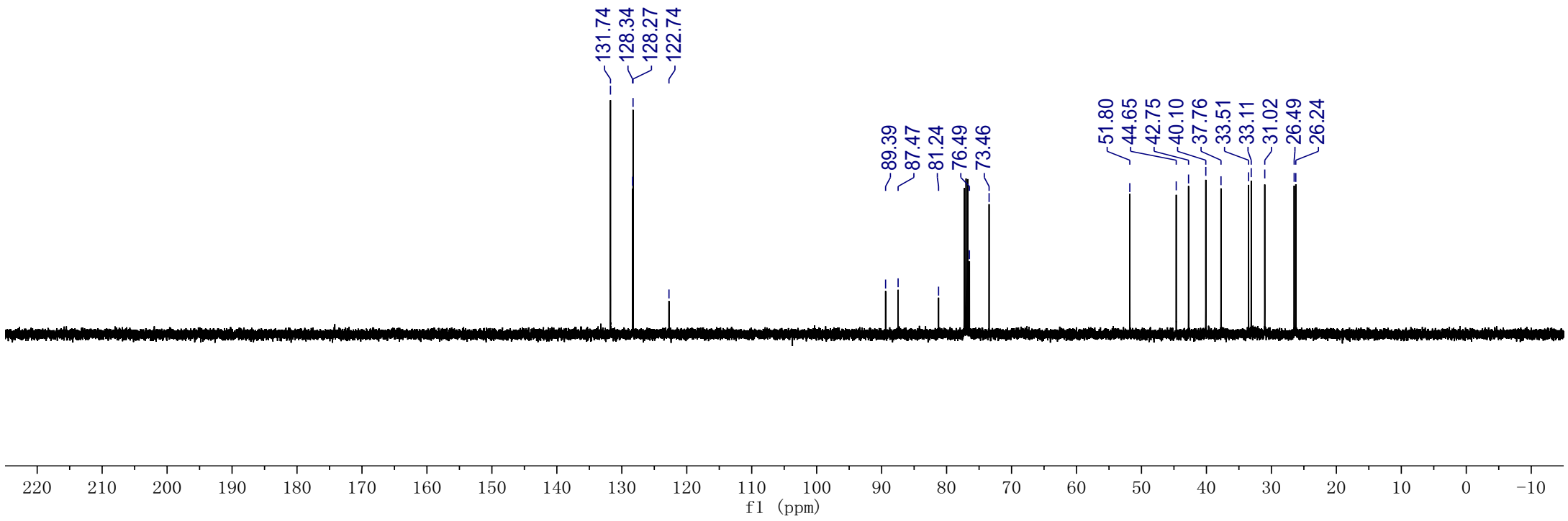
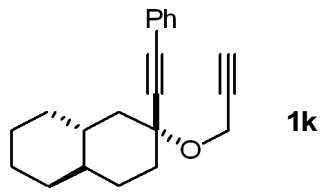
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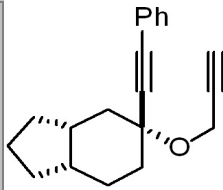
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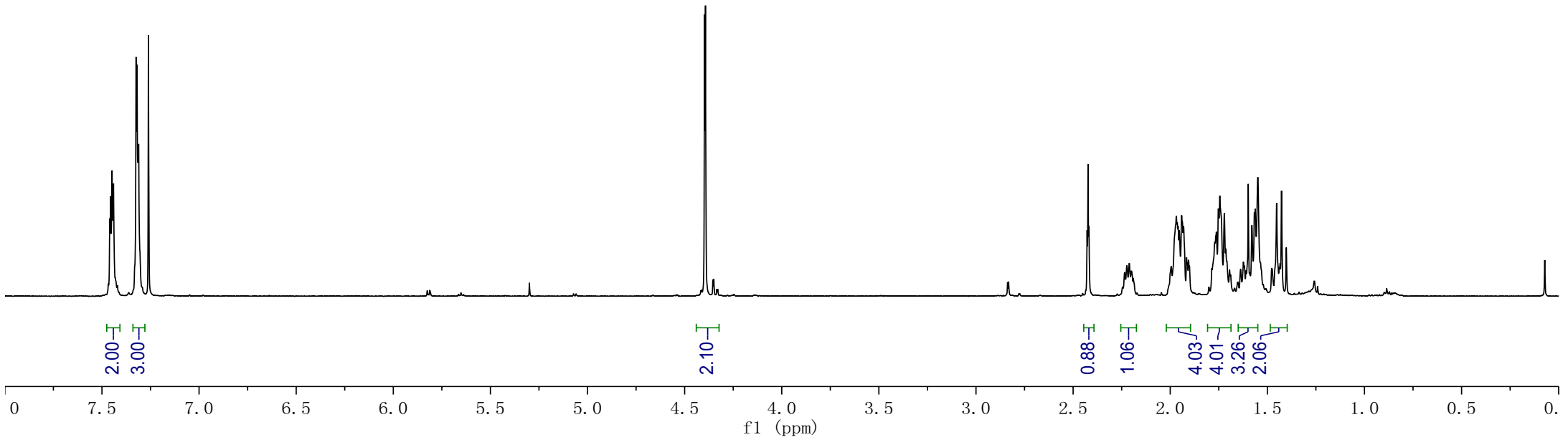
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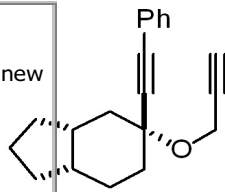
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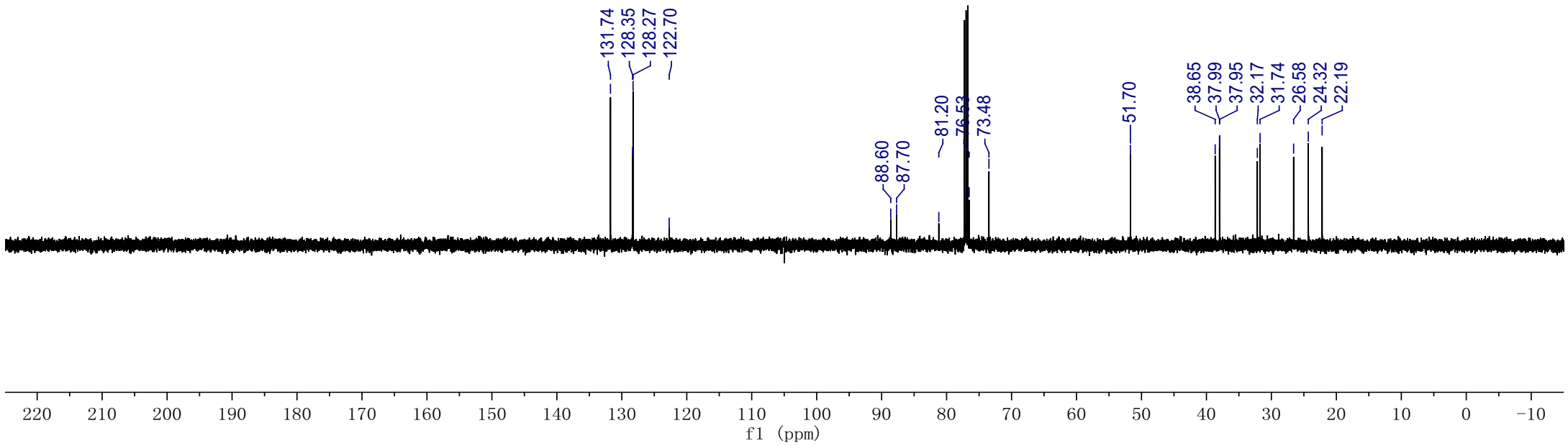
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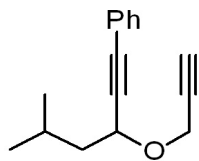
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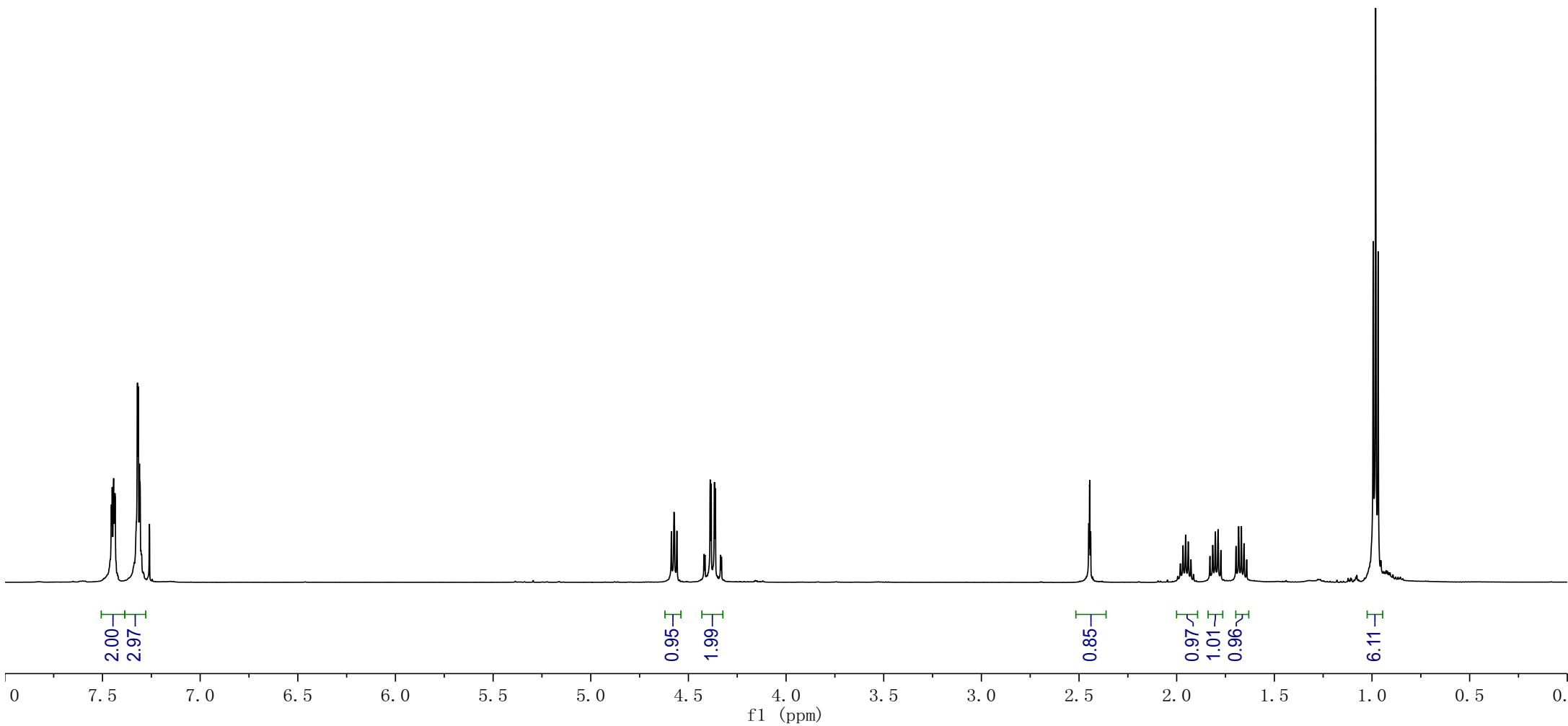
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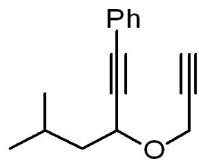
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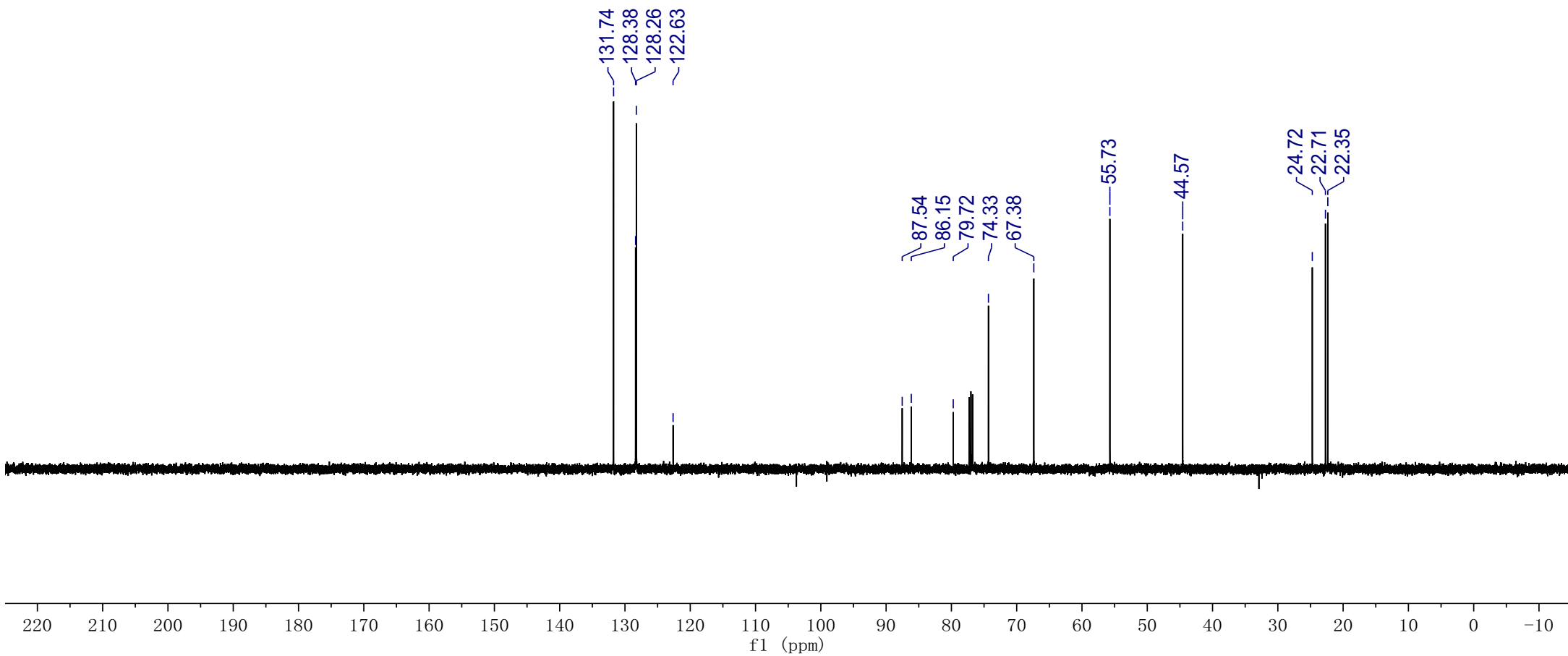
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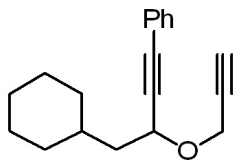
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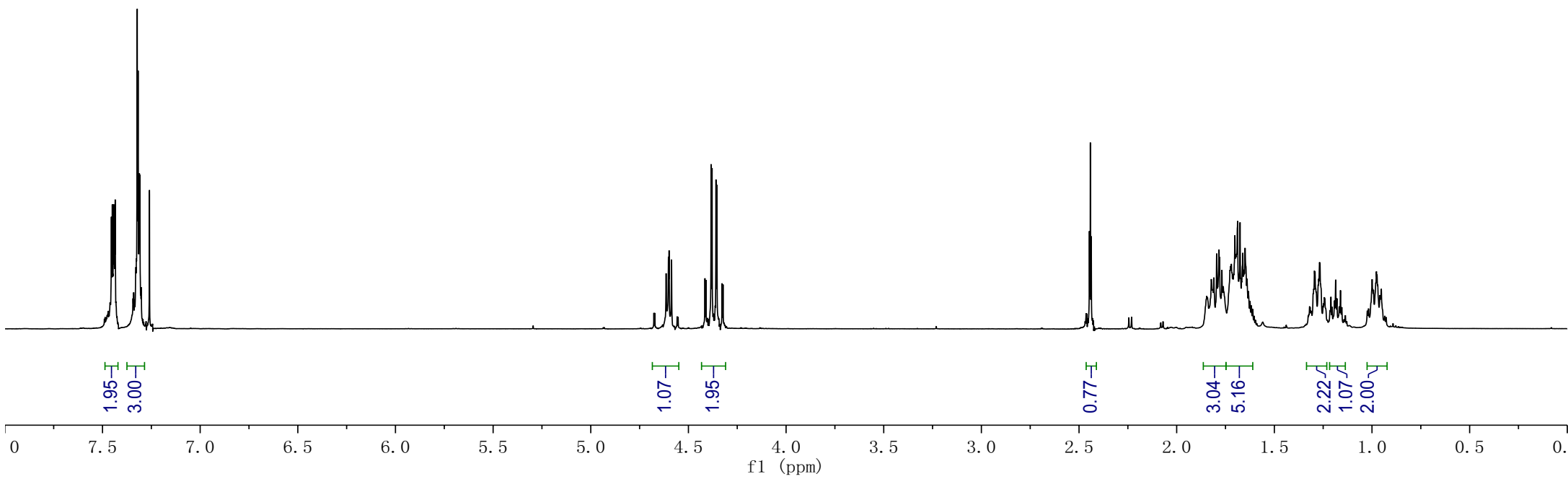
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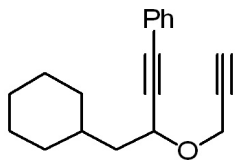
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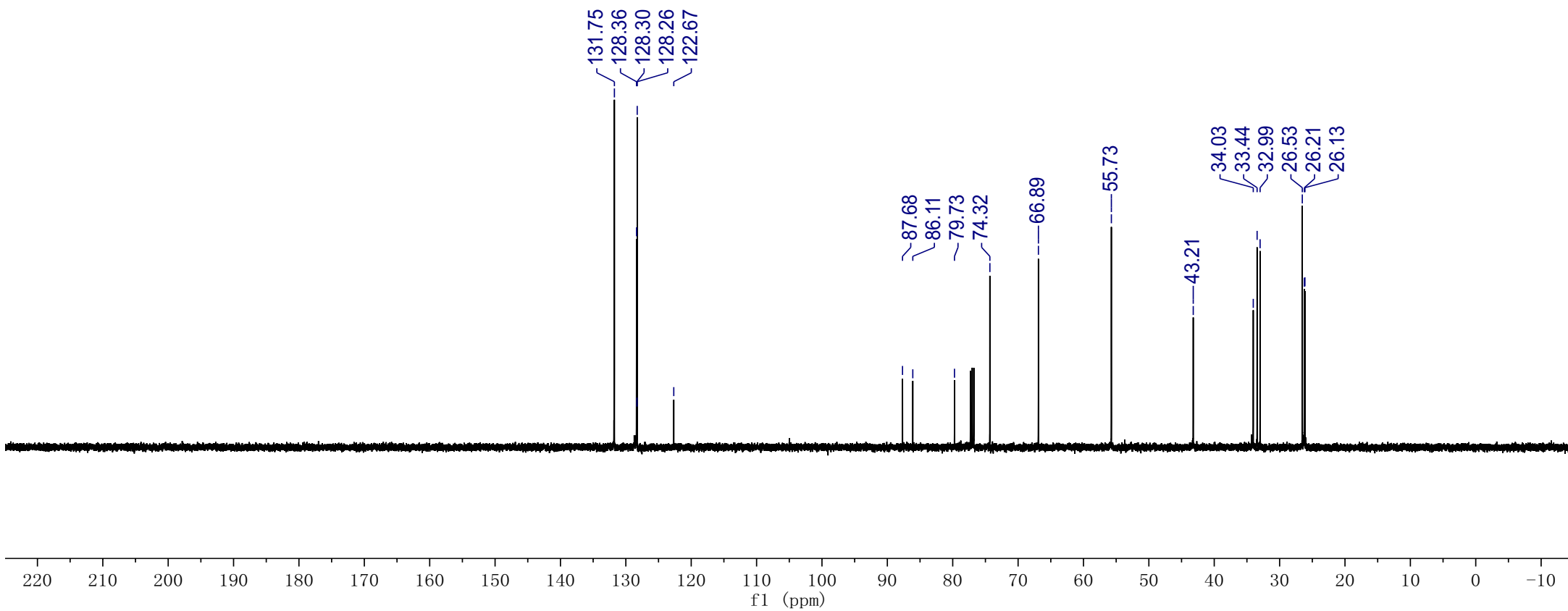
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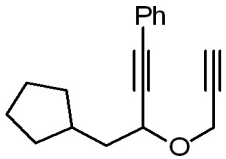
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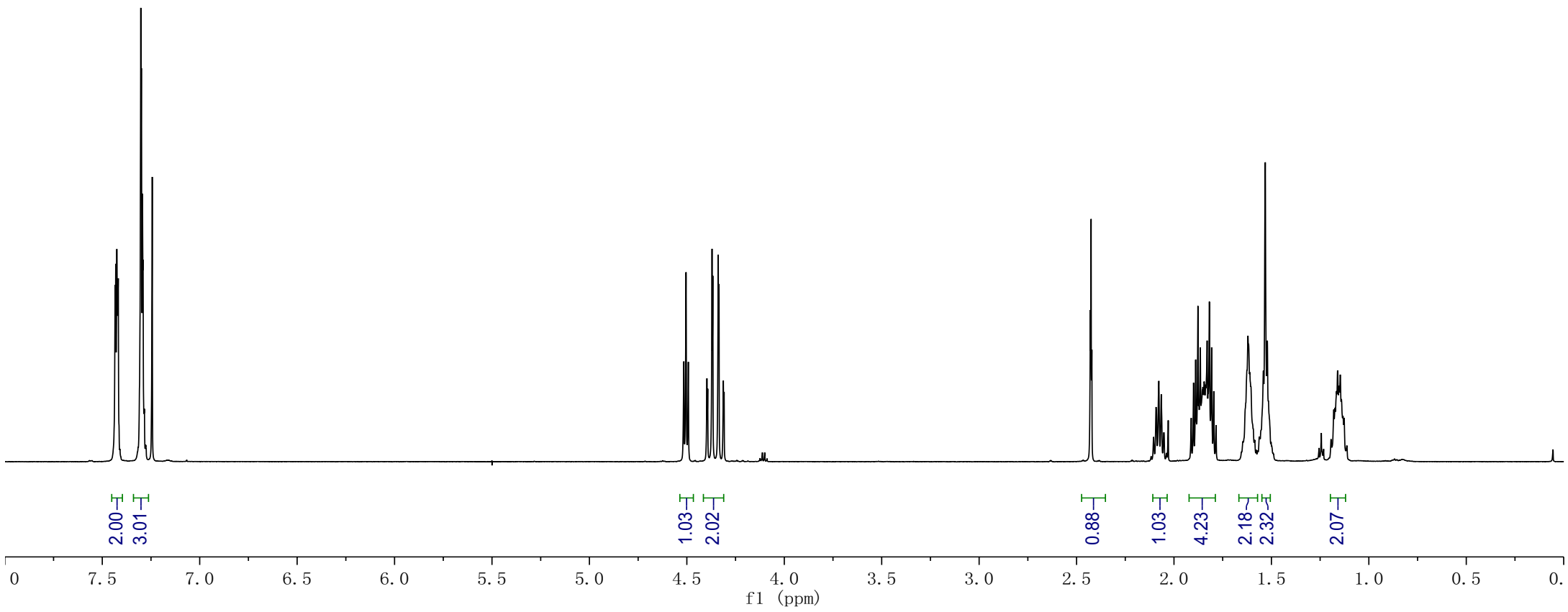
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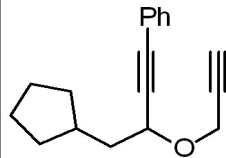
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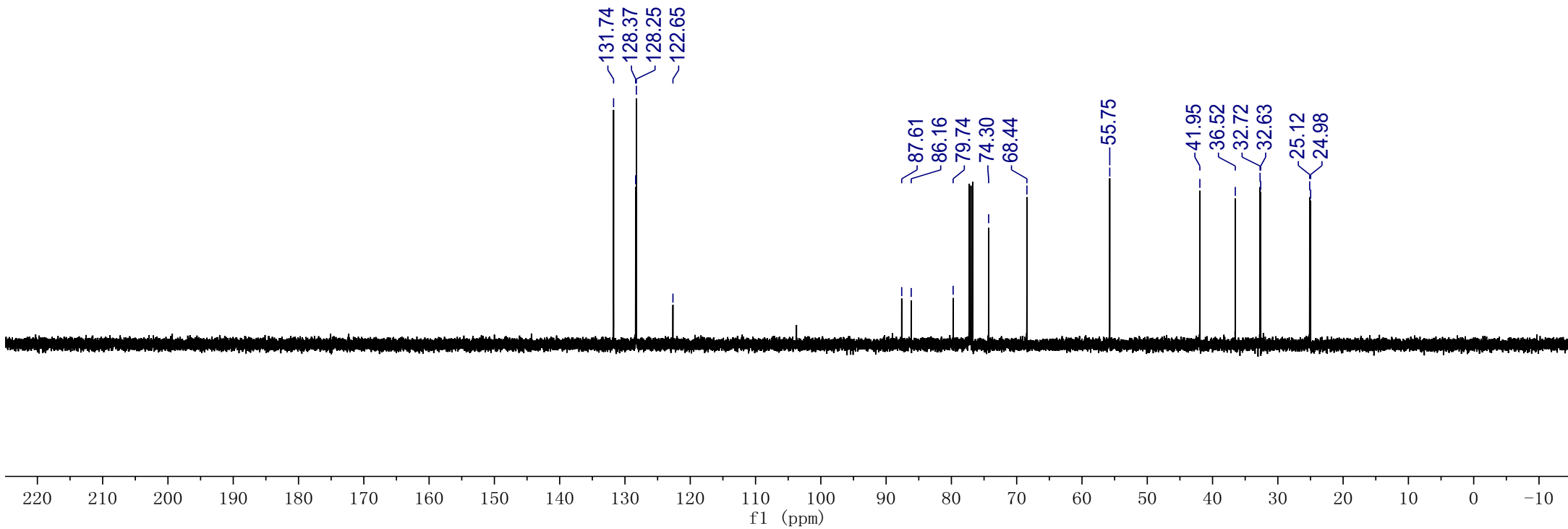
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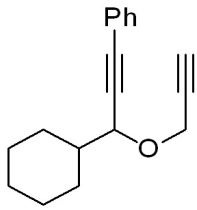
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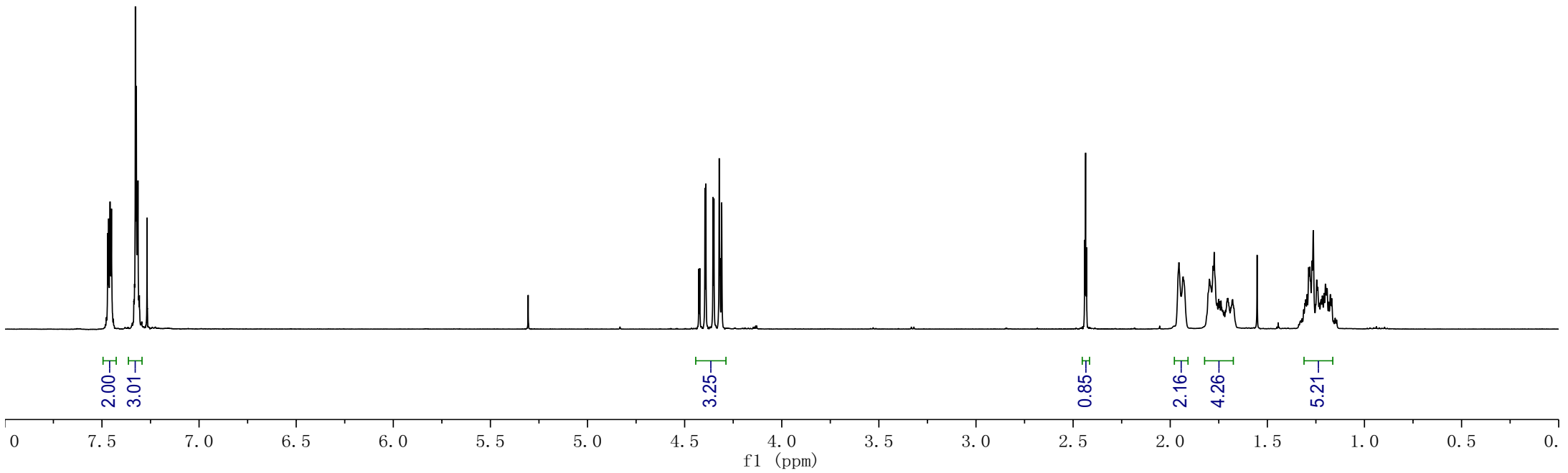
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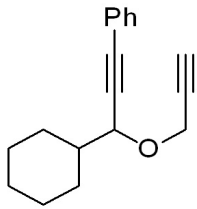
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4 Nucleus	1H



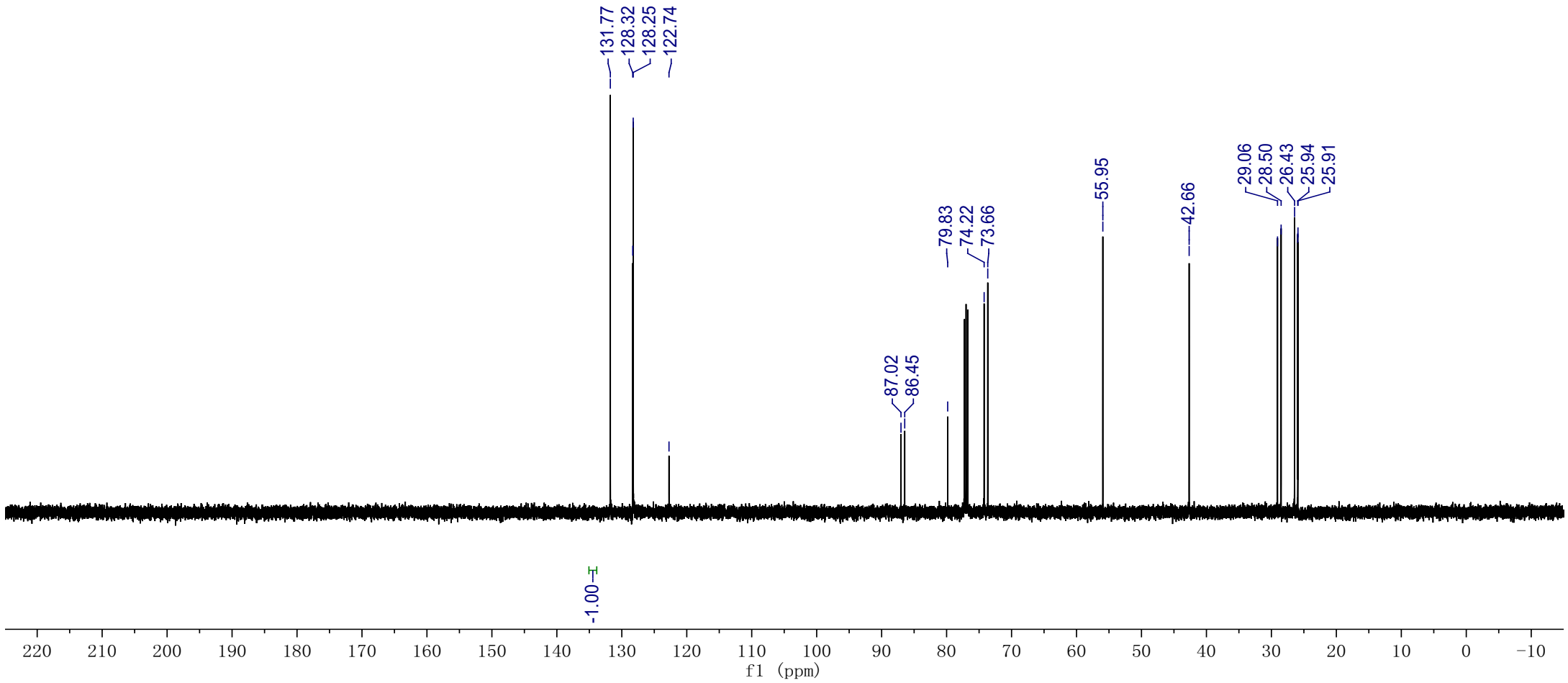
1p



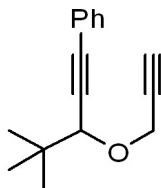
Parameter	Value
1 Title	zzt-18-4-SM-C
2 Solvent	CDCl3
3 Spectrometer Frequency	125.70
4 Nucleus	13C



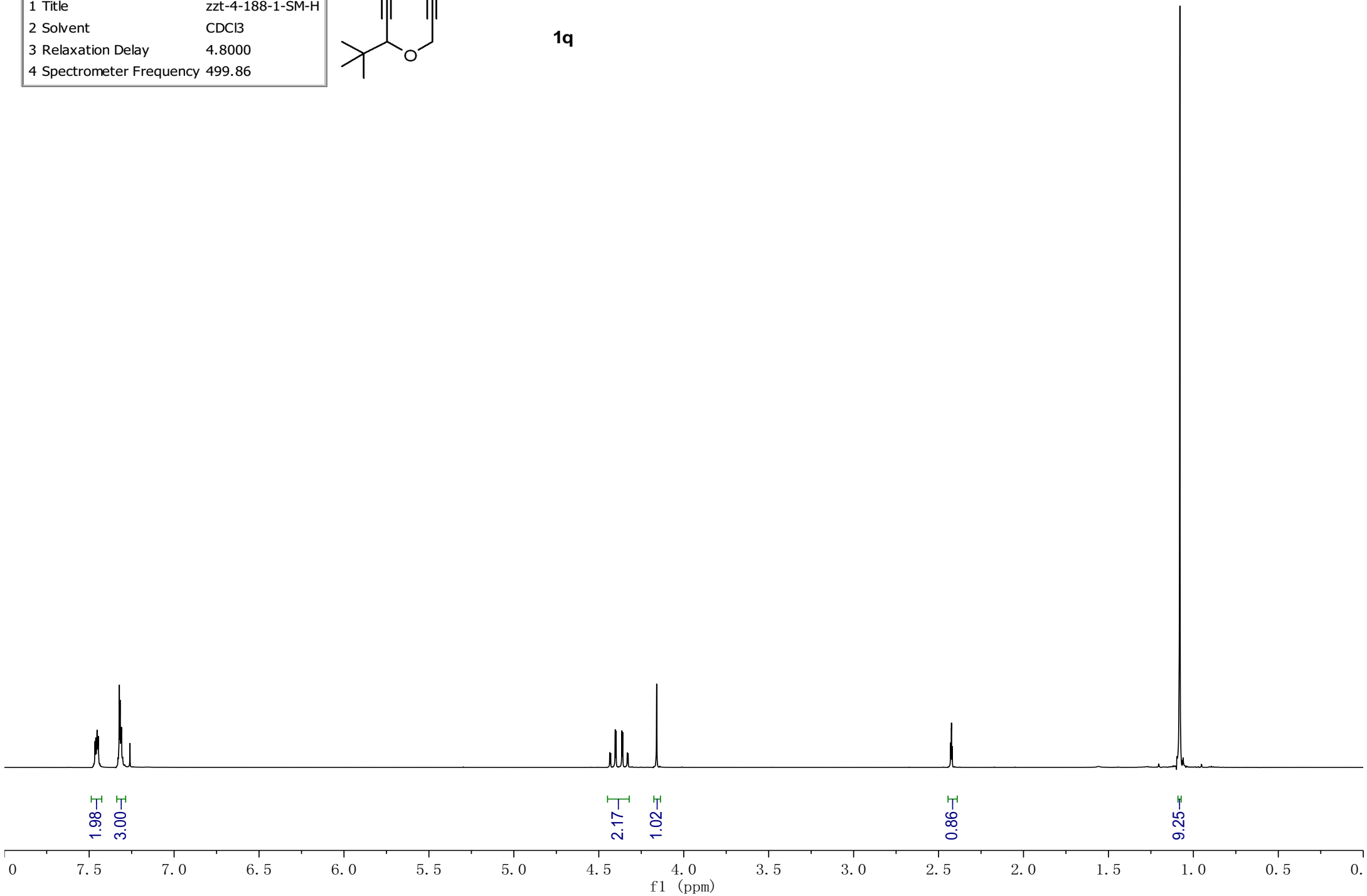
1p



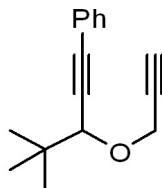
Parameter	Value
1 Title	zzt-4-188-1-SM-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



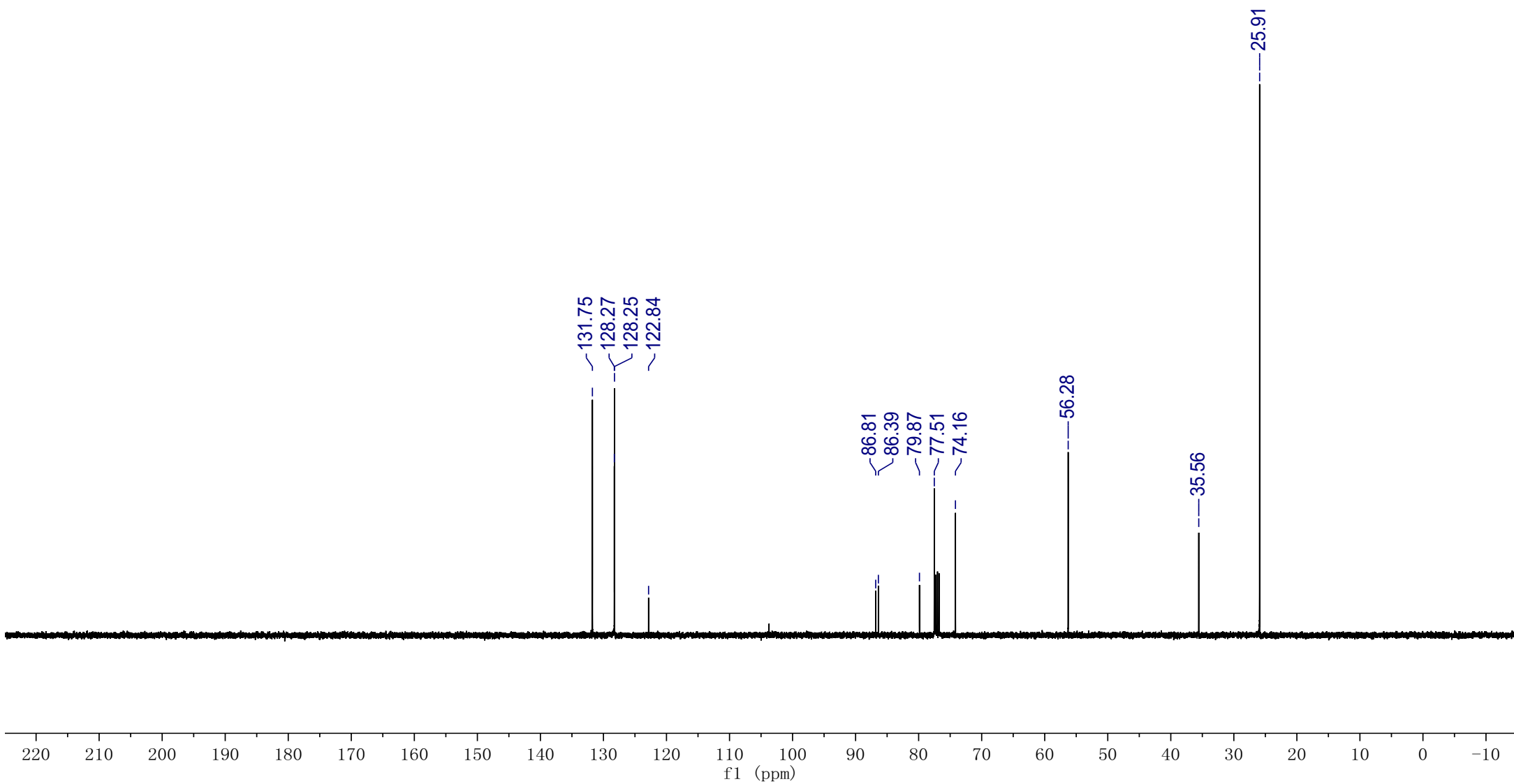
1q



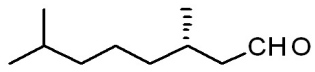
Parameter	Value
1 Title	zzt-4-188-1-SM-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



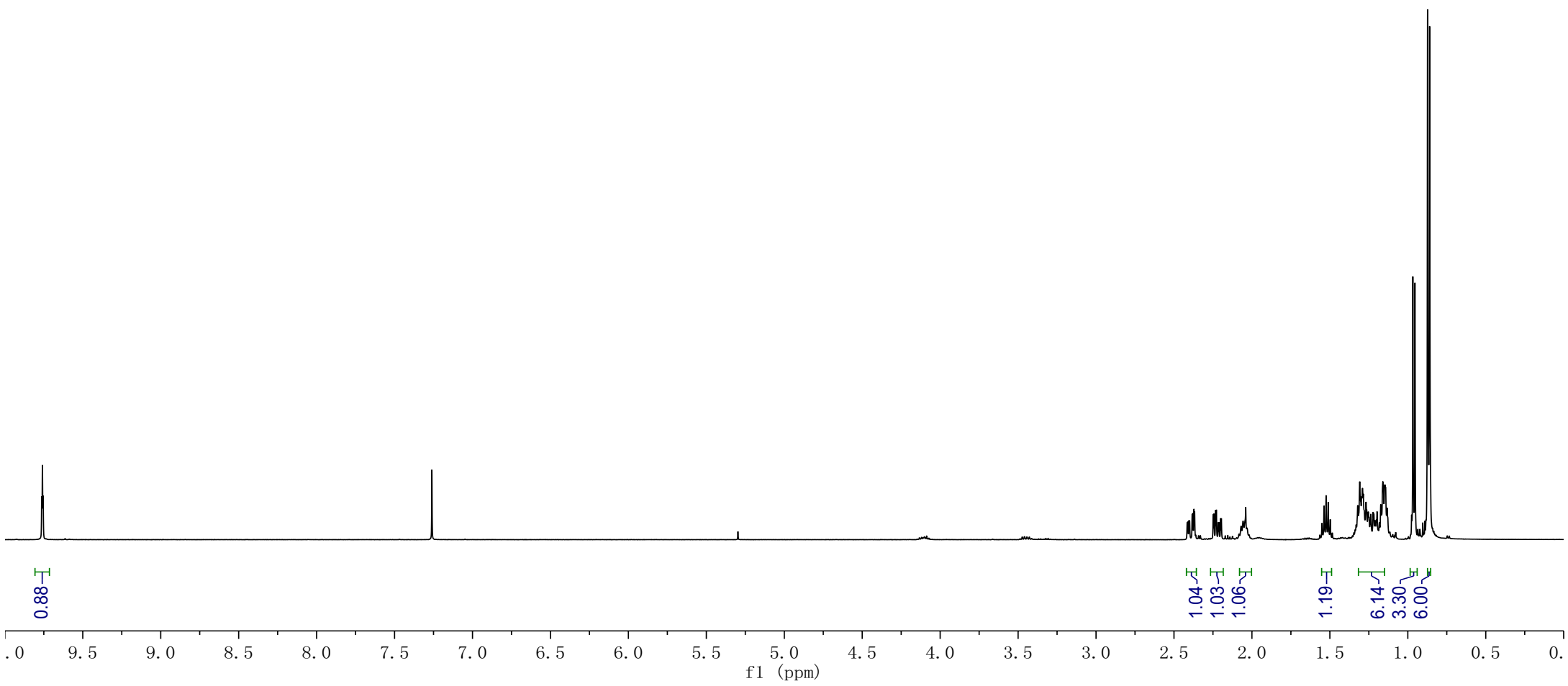
1q



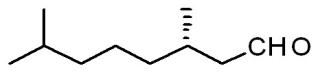
Parameter	Value
1 Title	zzt-5-8-2-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



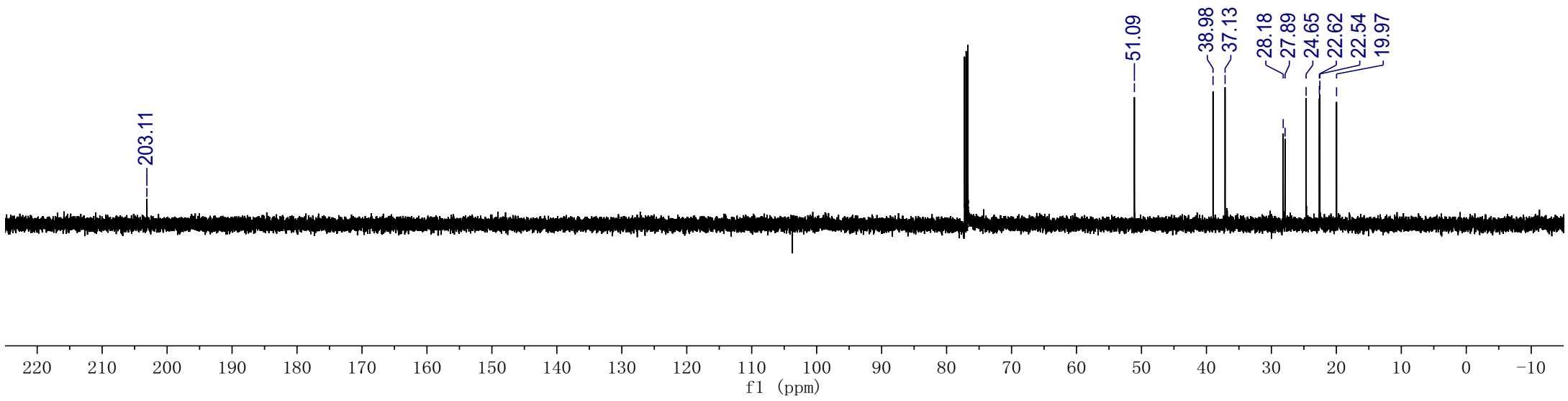
A



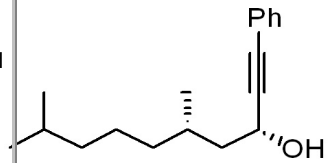
Parameter	Value
1 Title	zzt-5-8-2-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



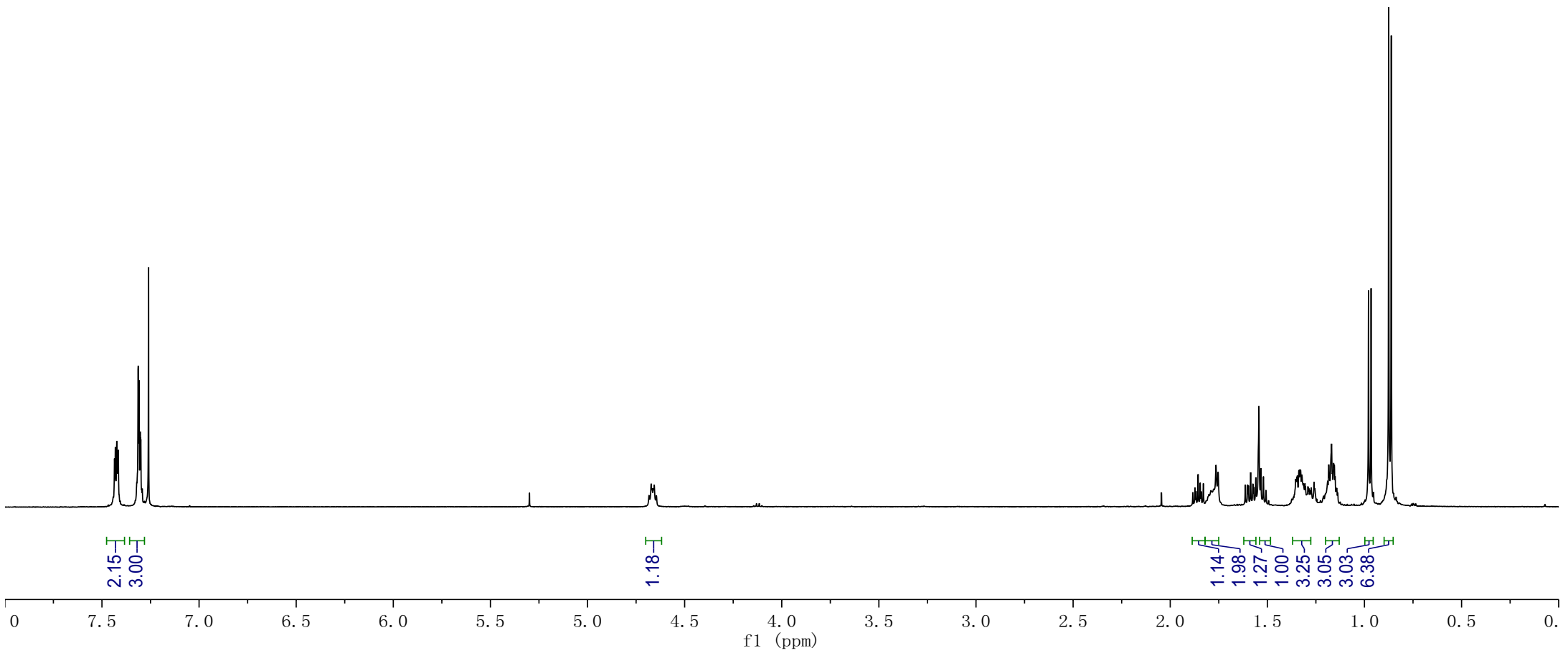
A



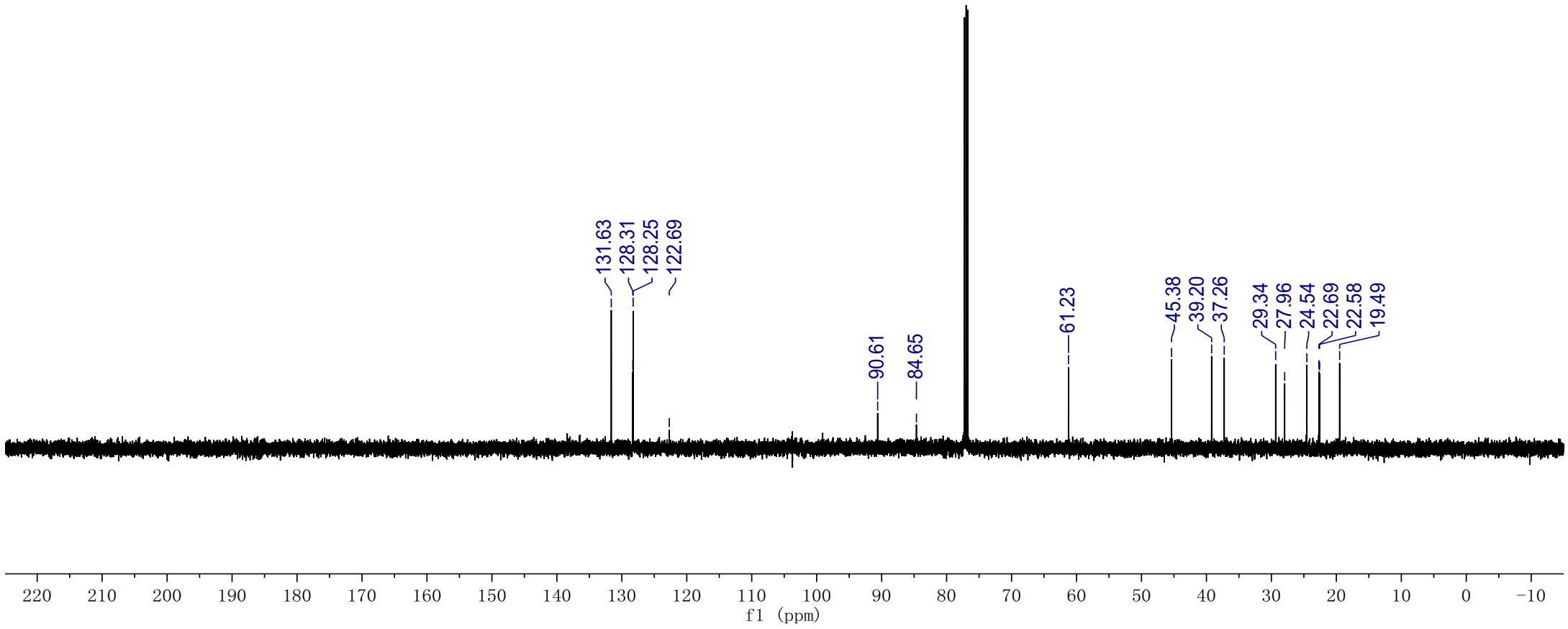
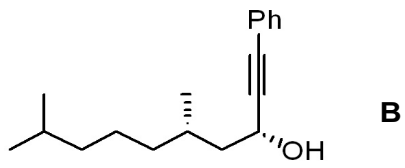
Parameter	Value
1 Title	zzt-5-8-3-H-good
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



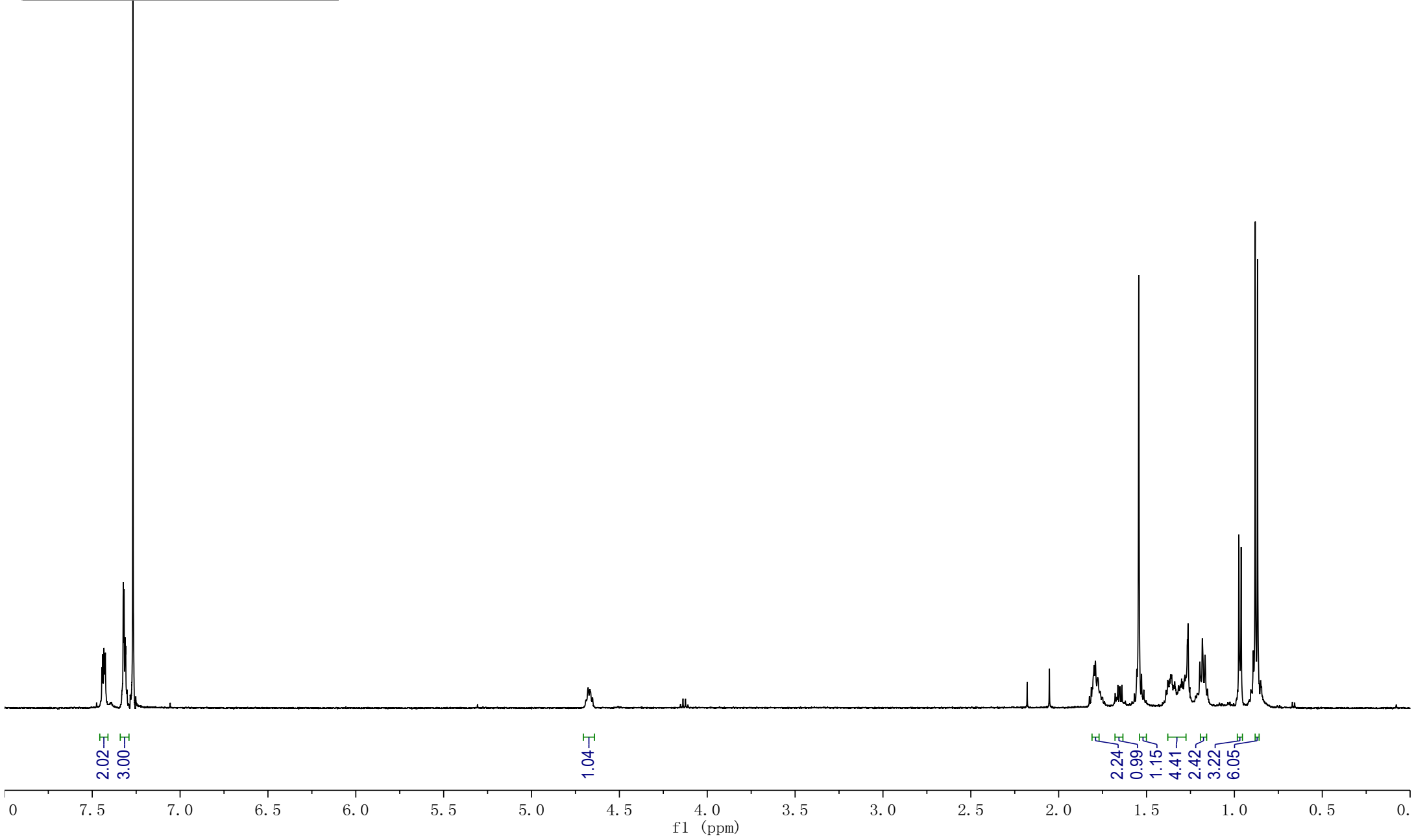
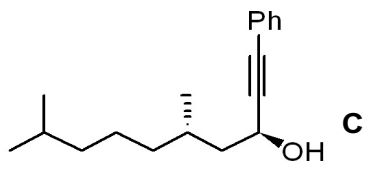
B



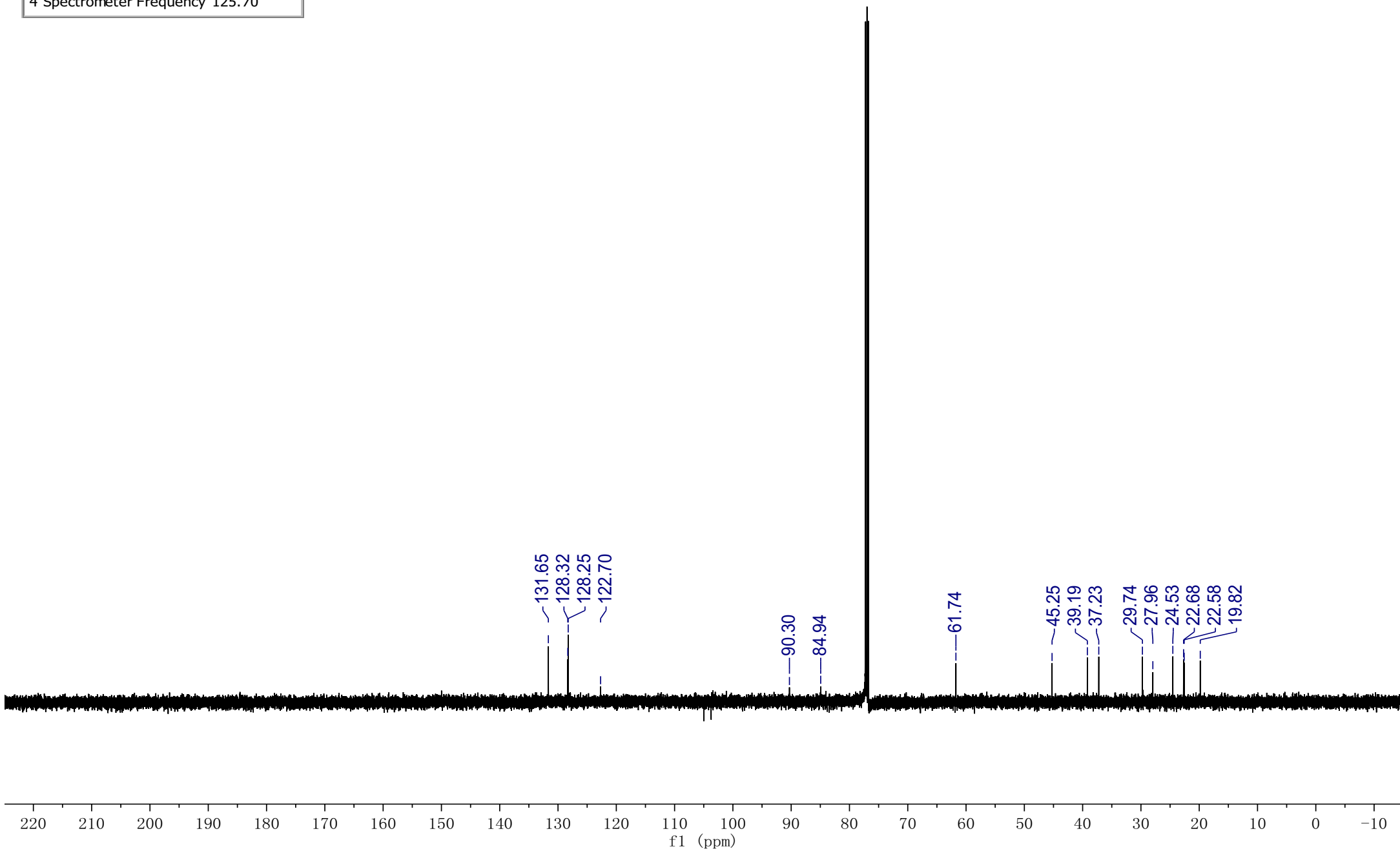
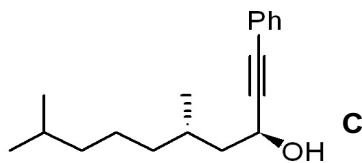
Parameter	Value
1 Title	zzt-5-8-3-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



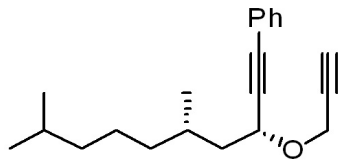
Parameter	Value
1 Title	zzt-5-12-2-H-new
2 Solvent	CDCl3
3 Spectrometer Frequency	499.86
4 Nucleus	1H



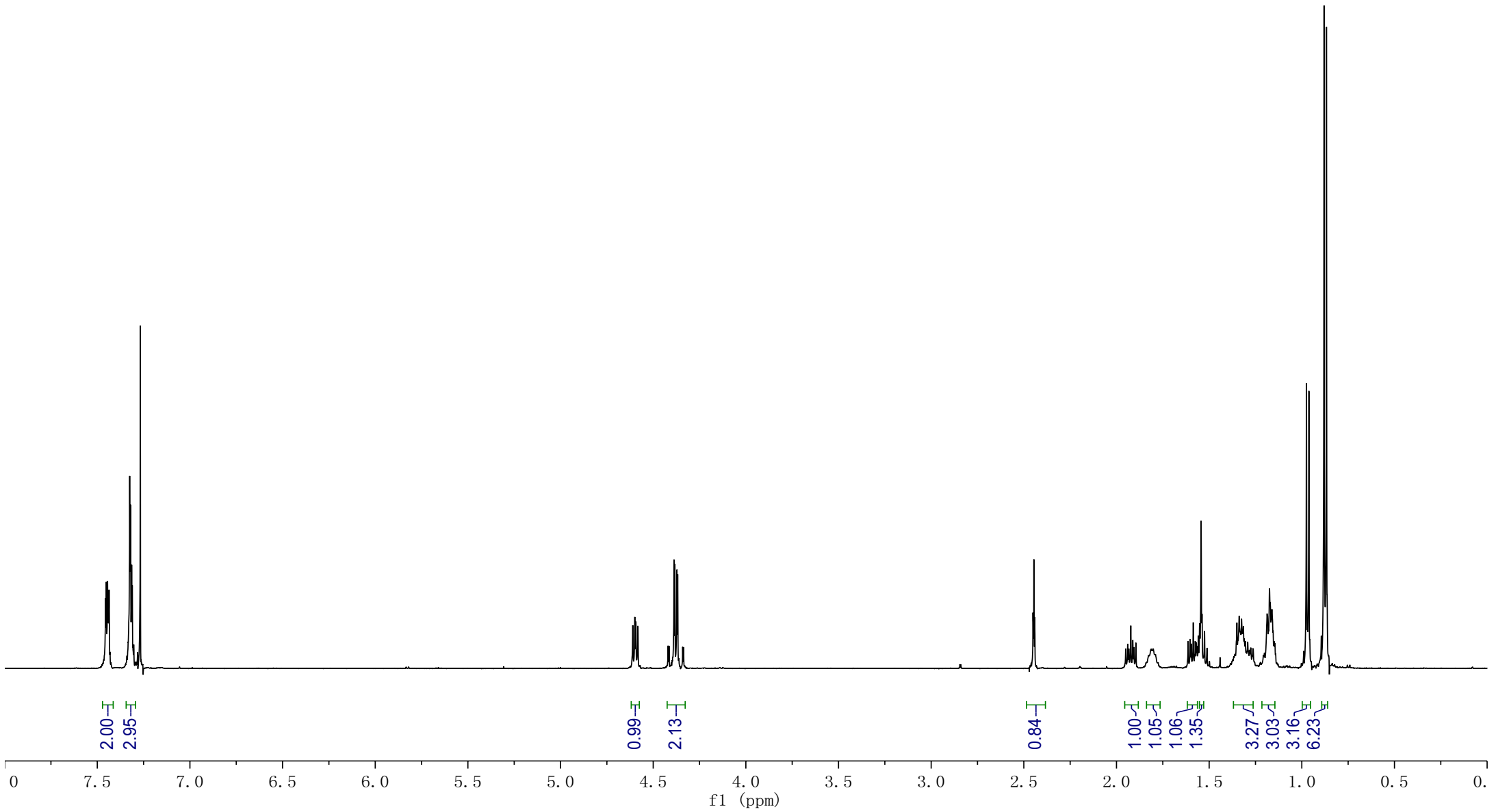
Parameter	Value
1 Title	zzt-5-12-2-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



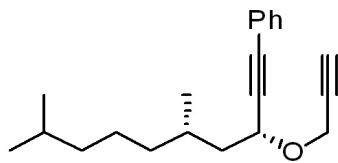
Parameter	Value
1 Title	zzt-5-10-2-H-new
2 Solvent	CDCl3
3 Spectrometer Frequency	499.86
4 Nucleus	1H



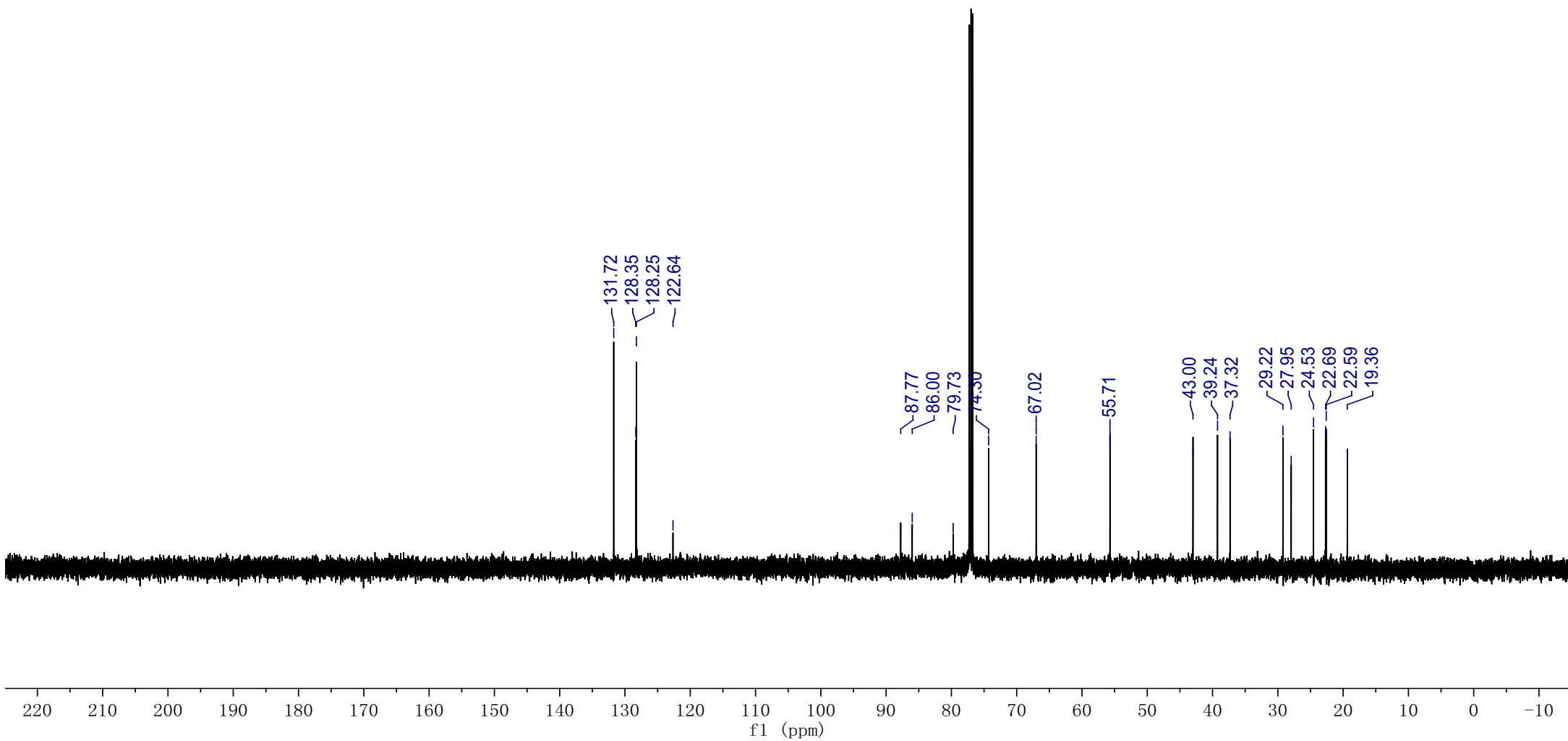
(3R, 5S)-1r



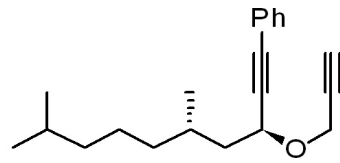
Parameter	Value
1 Title	zzt-5-10-2-C-new
2 Solvent	CDCl3
3 Spectrometer Frequency	125.70
4 Nucleus	13C



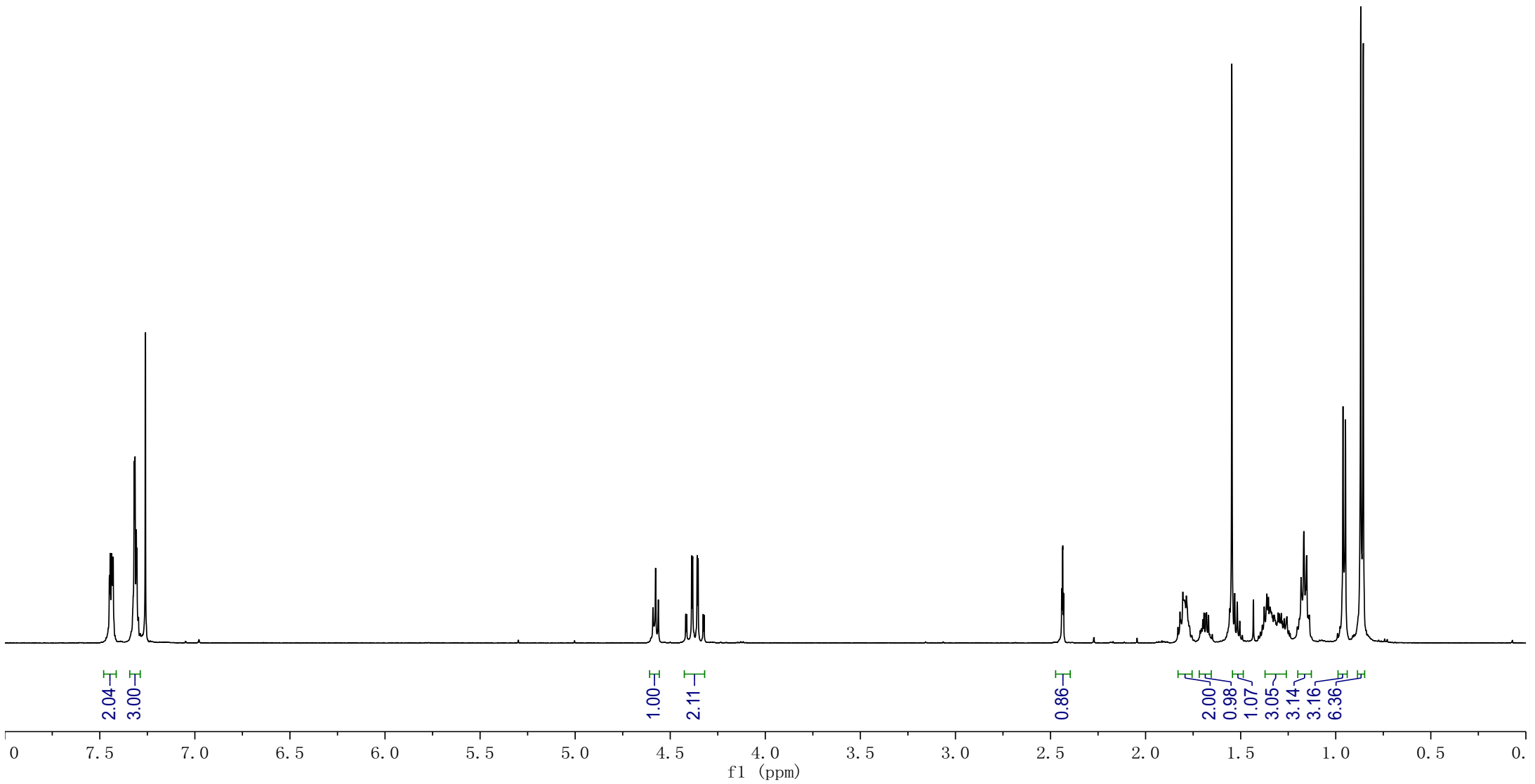
(3R, 5S)-1r



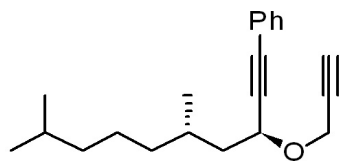
Parameter	Value
1 Title	zzt-5-12-3-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



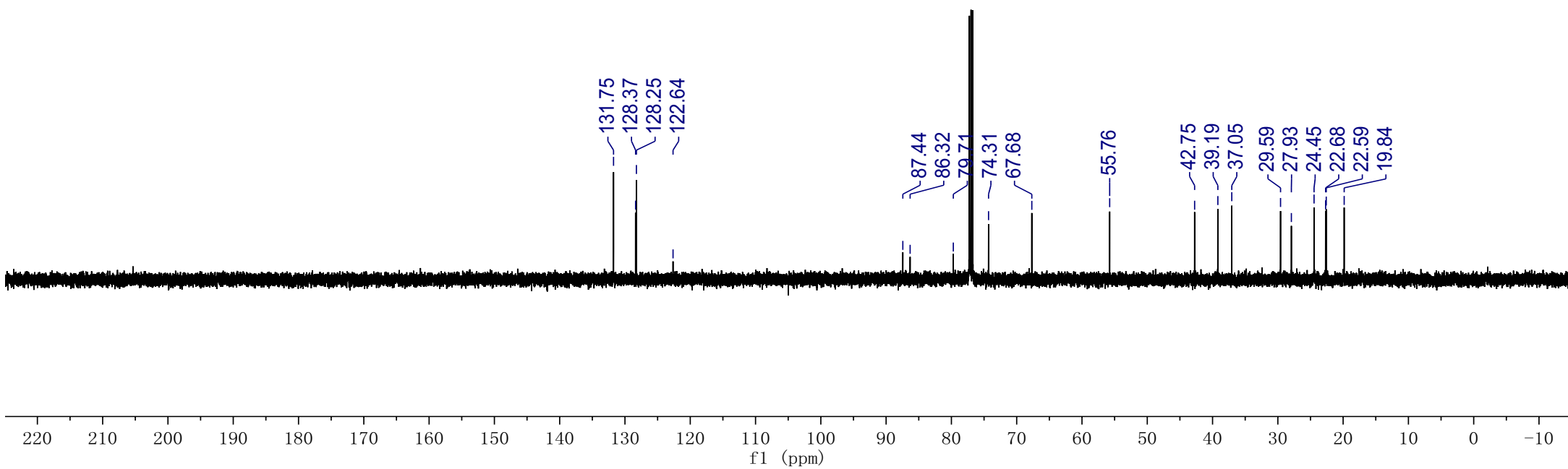
(3S, 5S)-1r



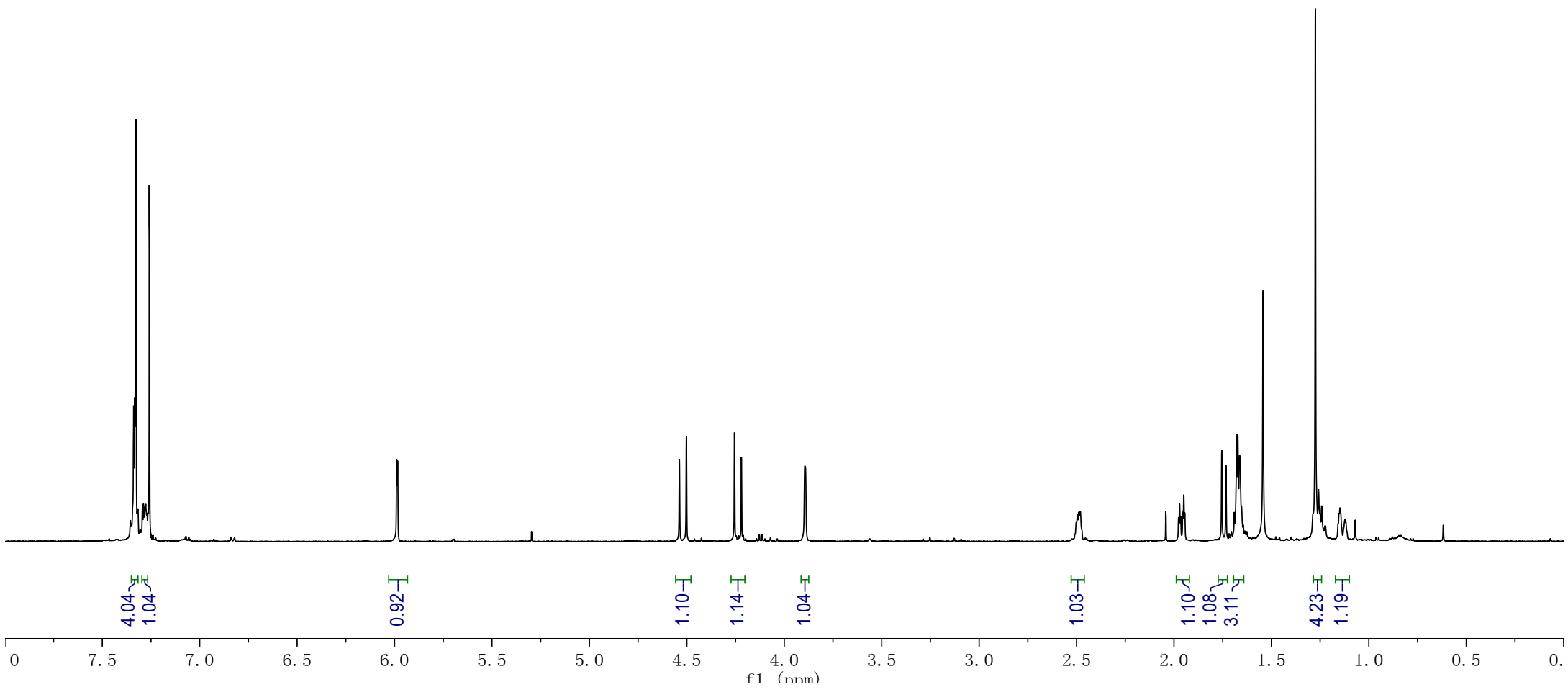
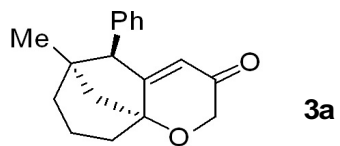
Parameter	Value
1 Title	zzt-5-12-3-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



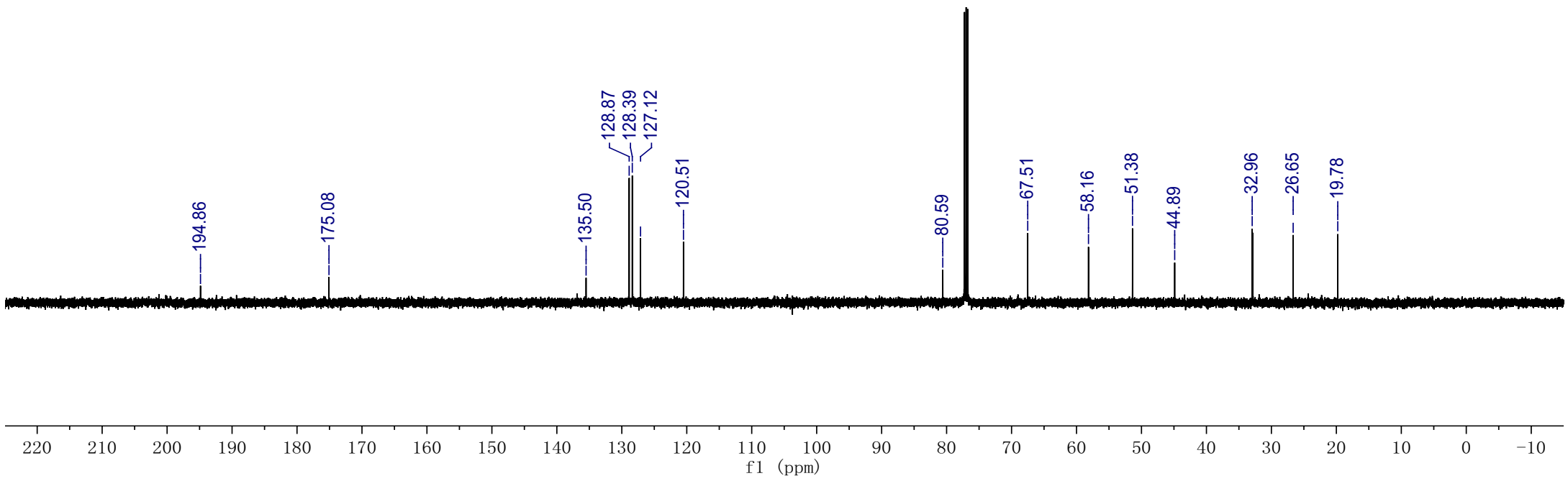
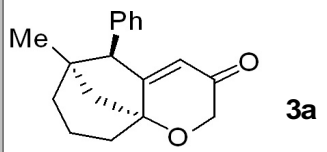
(3S, 5S)-1r



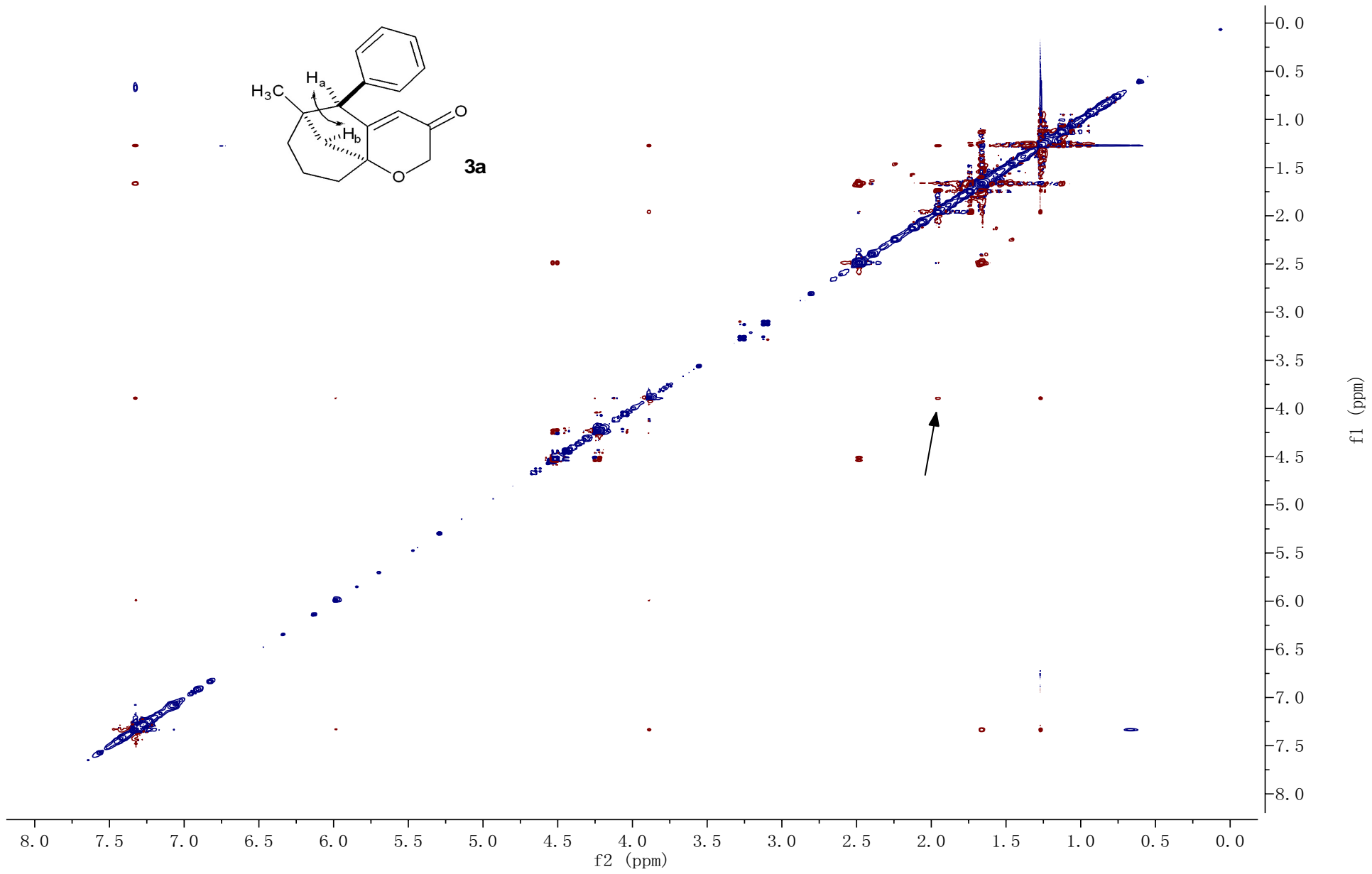
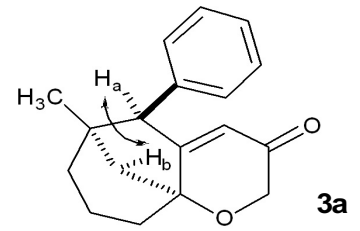
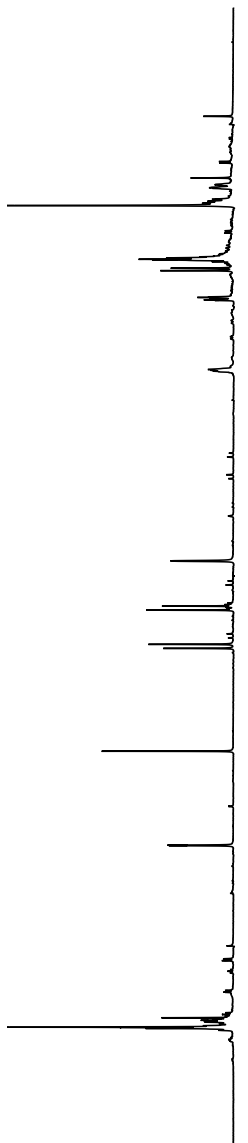
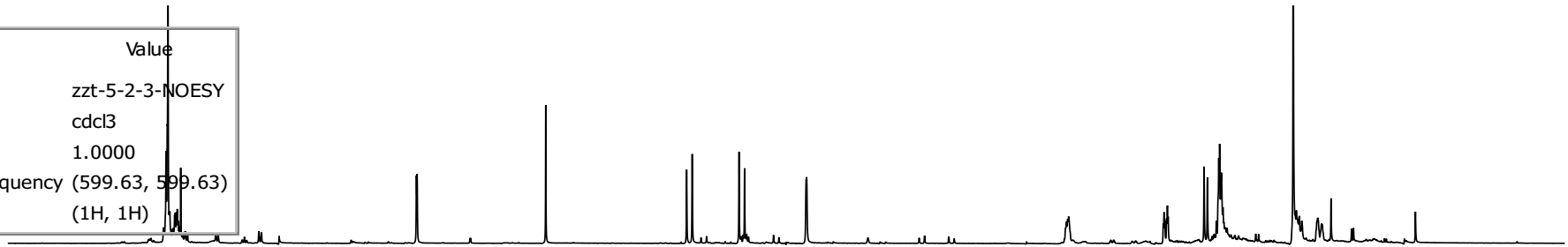
Parameter	Value
1 Title	zzt-5-2-3-pure-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



Parameter	Value
1 Title	zzt-5-2-3-C-new
2 Solvent	CDCl3
3 Spectrometer Frequency	125.70
4 Nucleus	13C



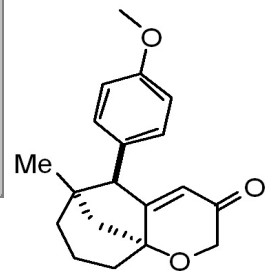
Parameter	Value
1 Title	zzt-5-2-3-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



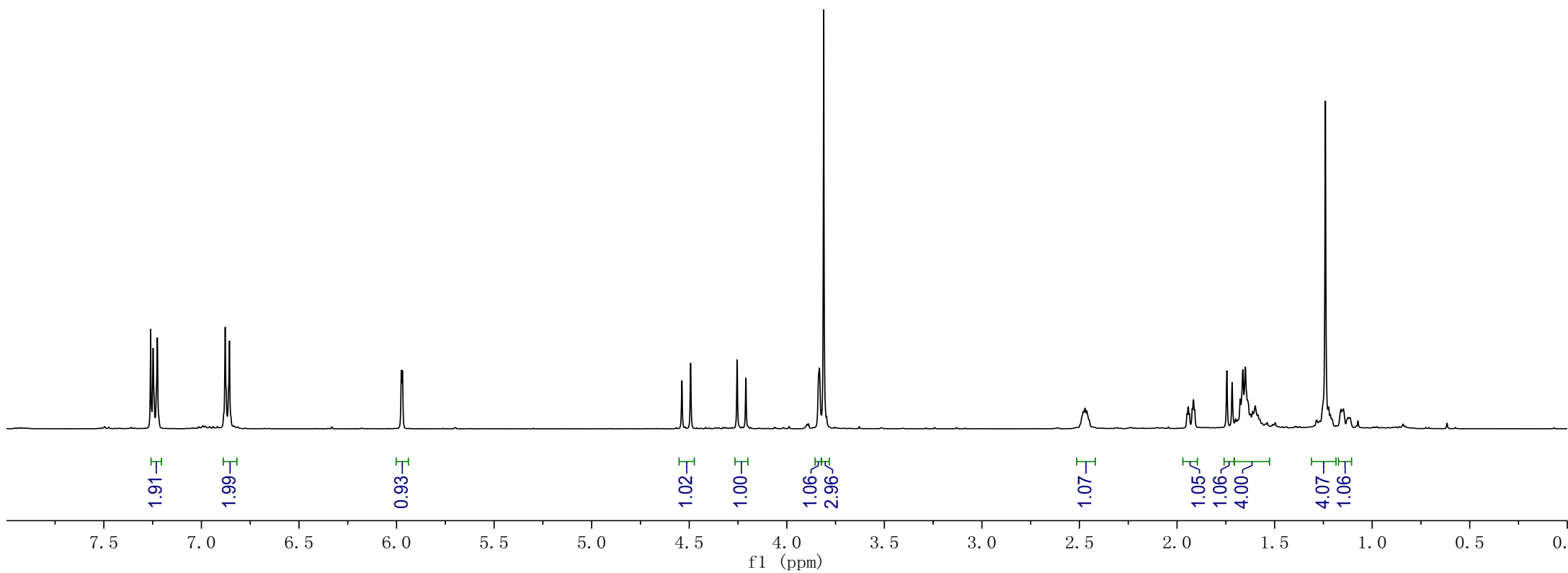
f1 (ppm)

f2 (ppm)

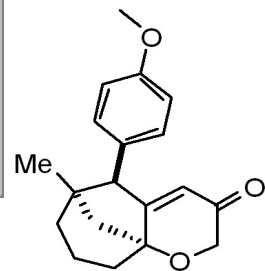
Parameter	Value
1 Title	zzt-4-151-1-H
2 Solvent	cdcl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	399.78



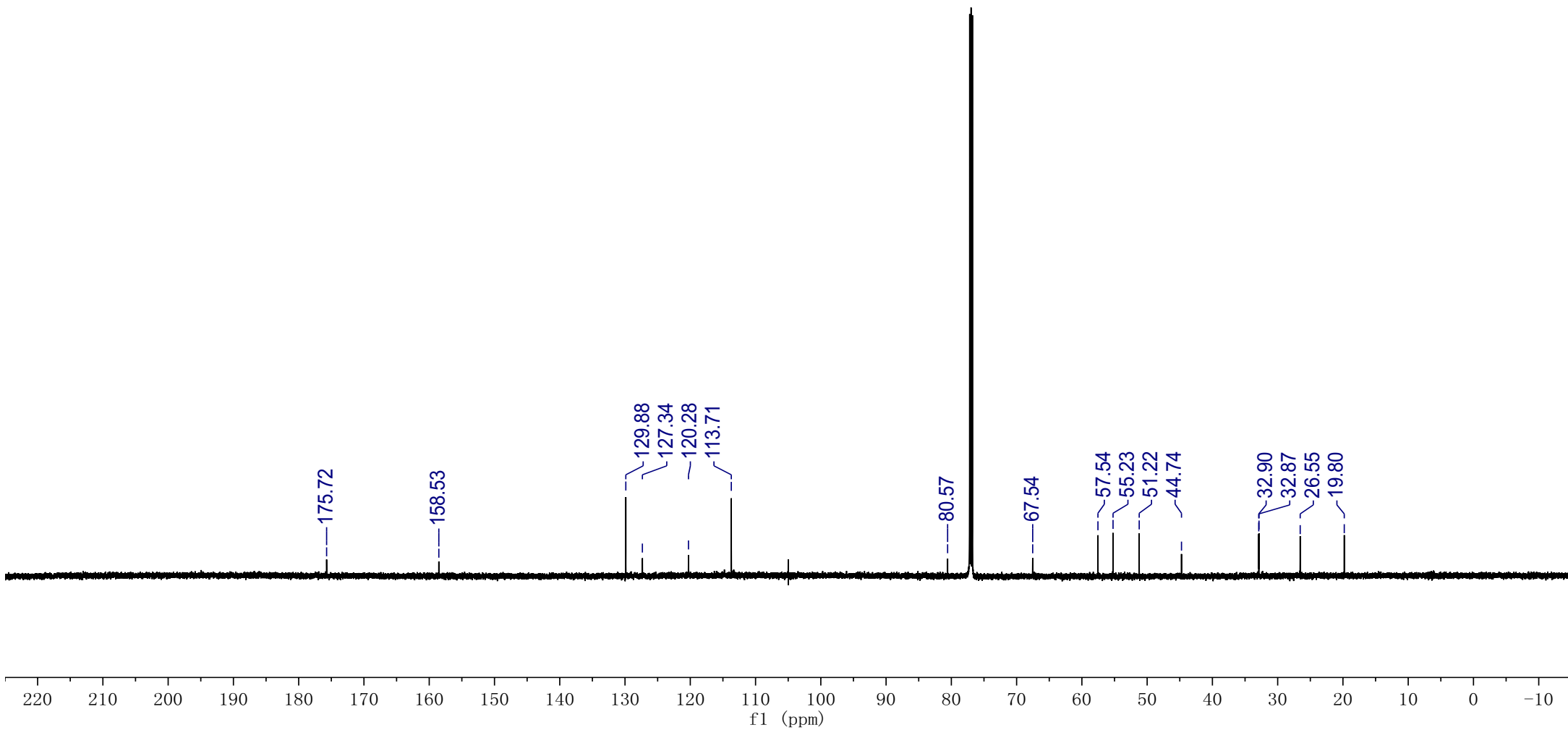
3b



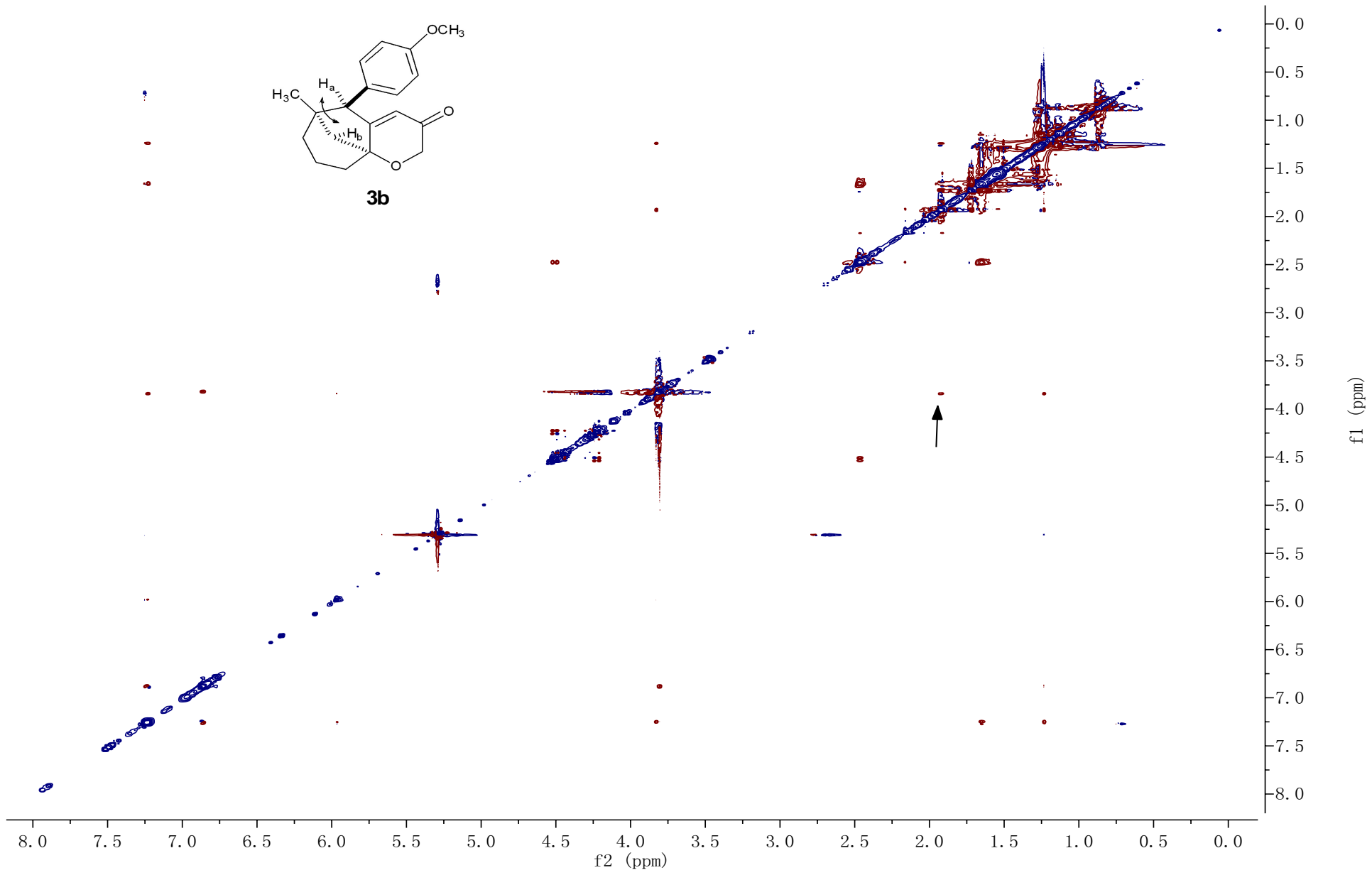
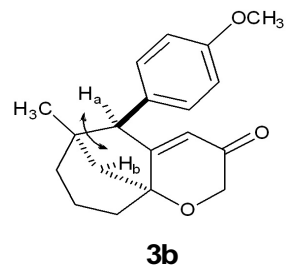
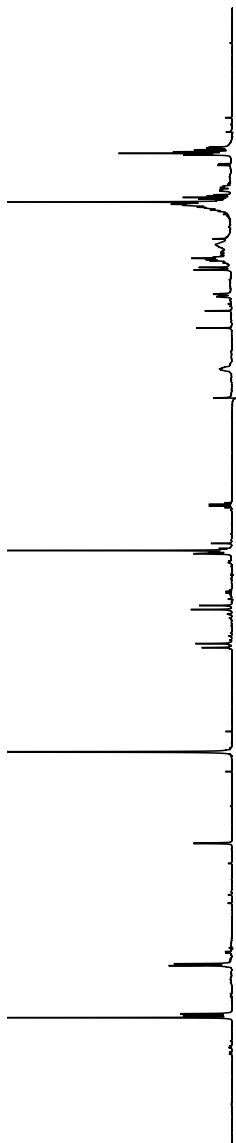
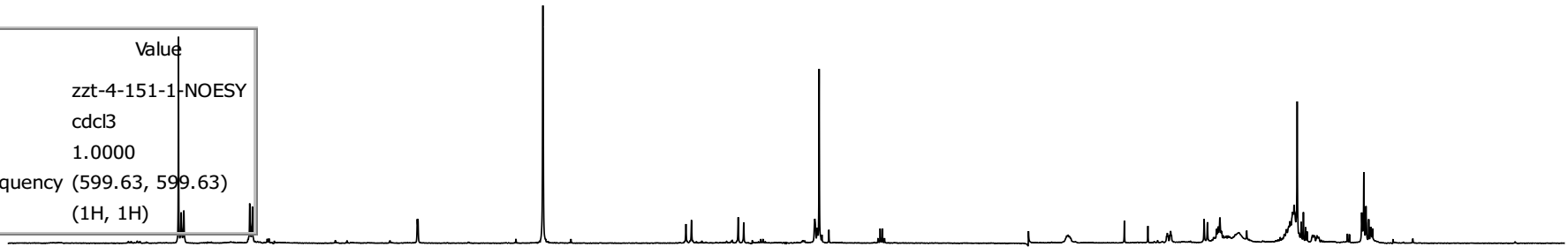
Parameter	Value
1 Title	zzt-4-151-1-C
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	150.79



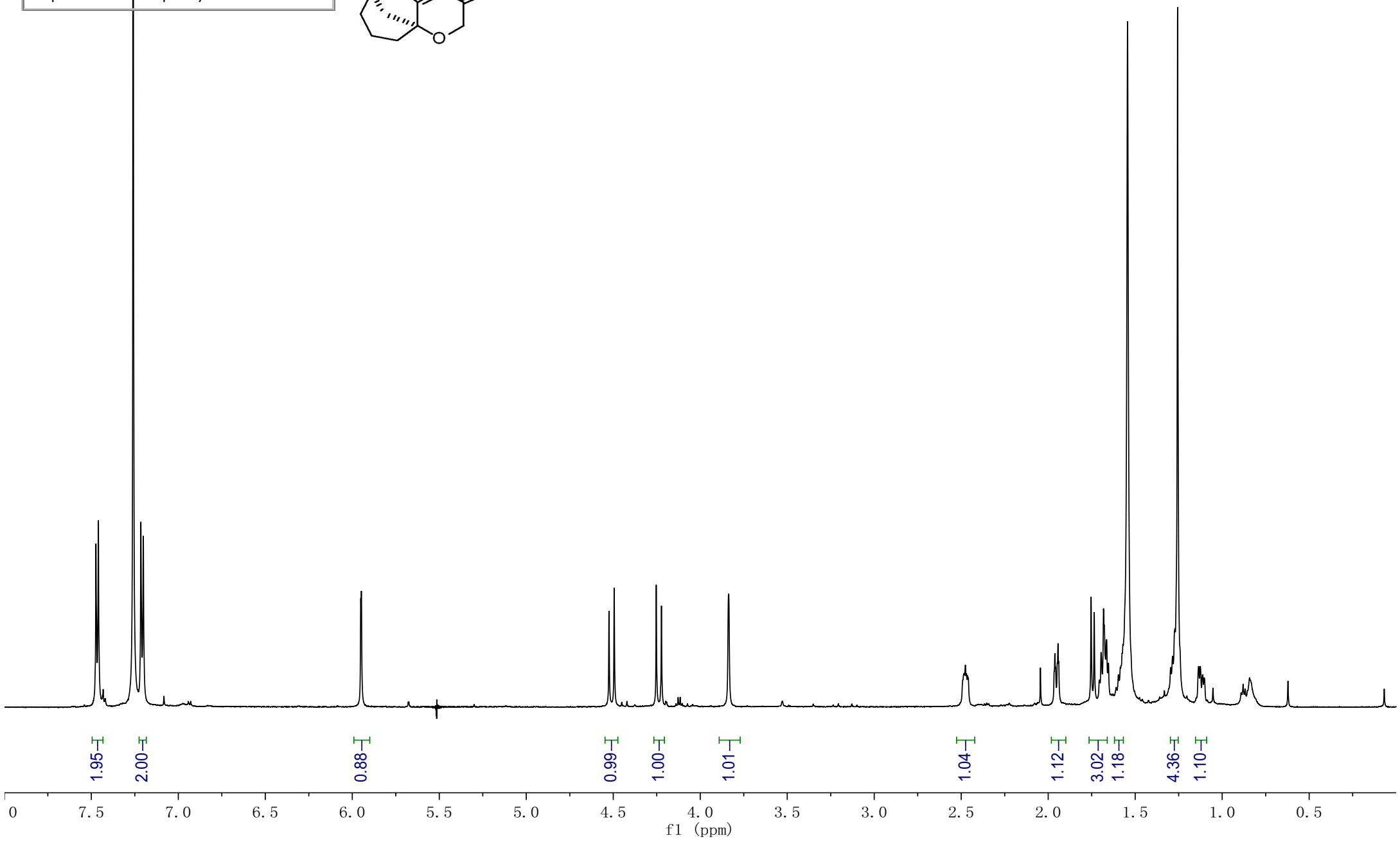
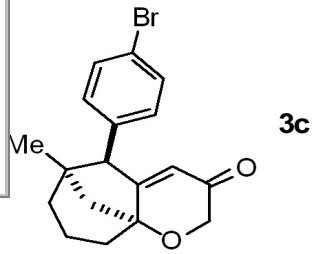
3b



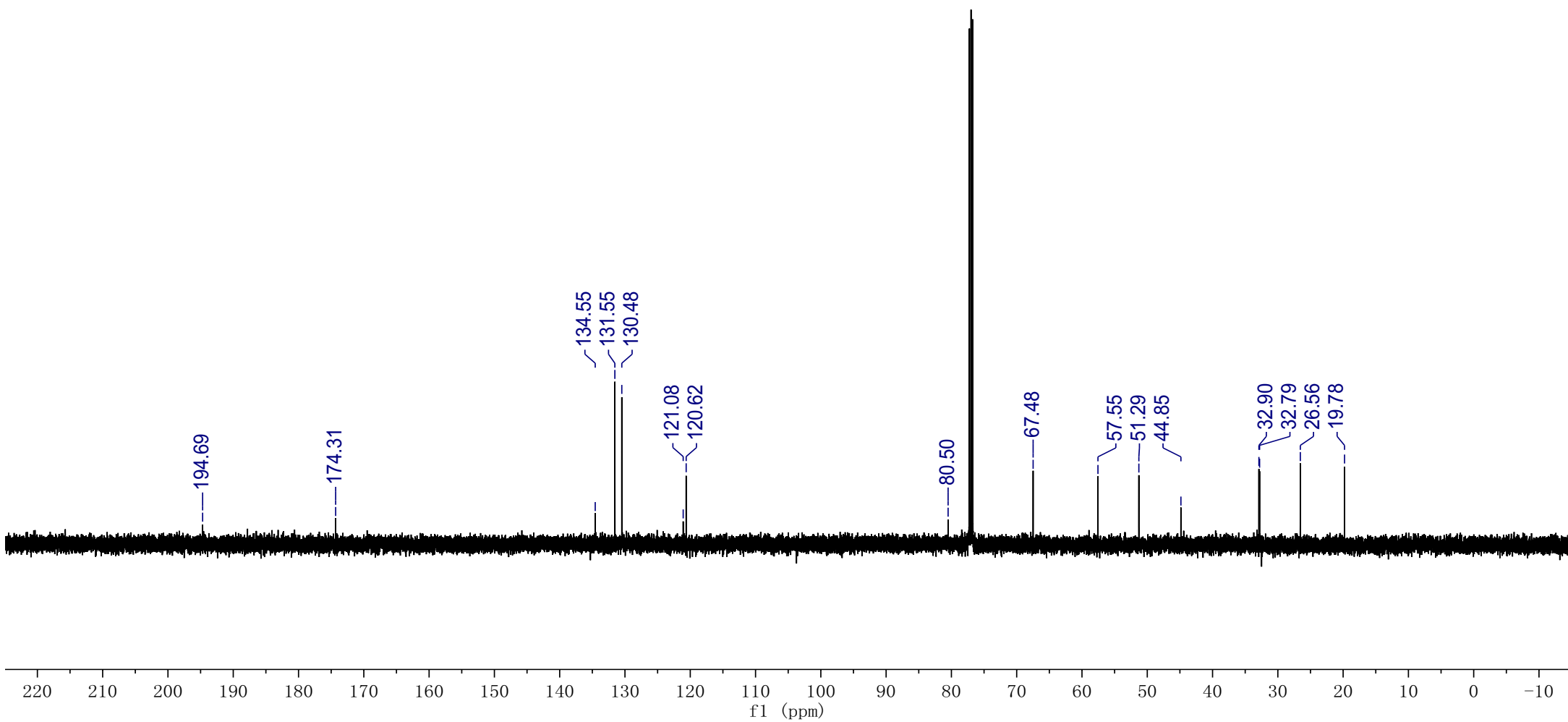
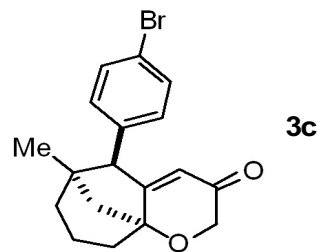
Parameter	Value
1 Title	zzt-4-151-1-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



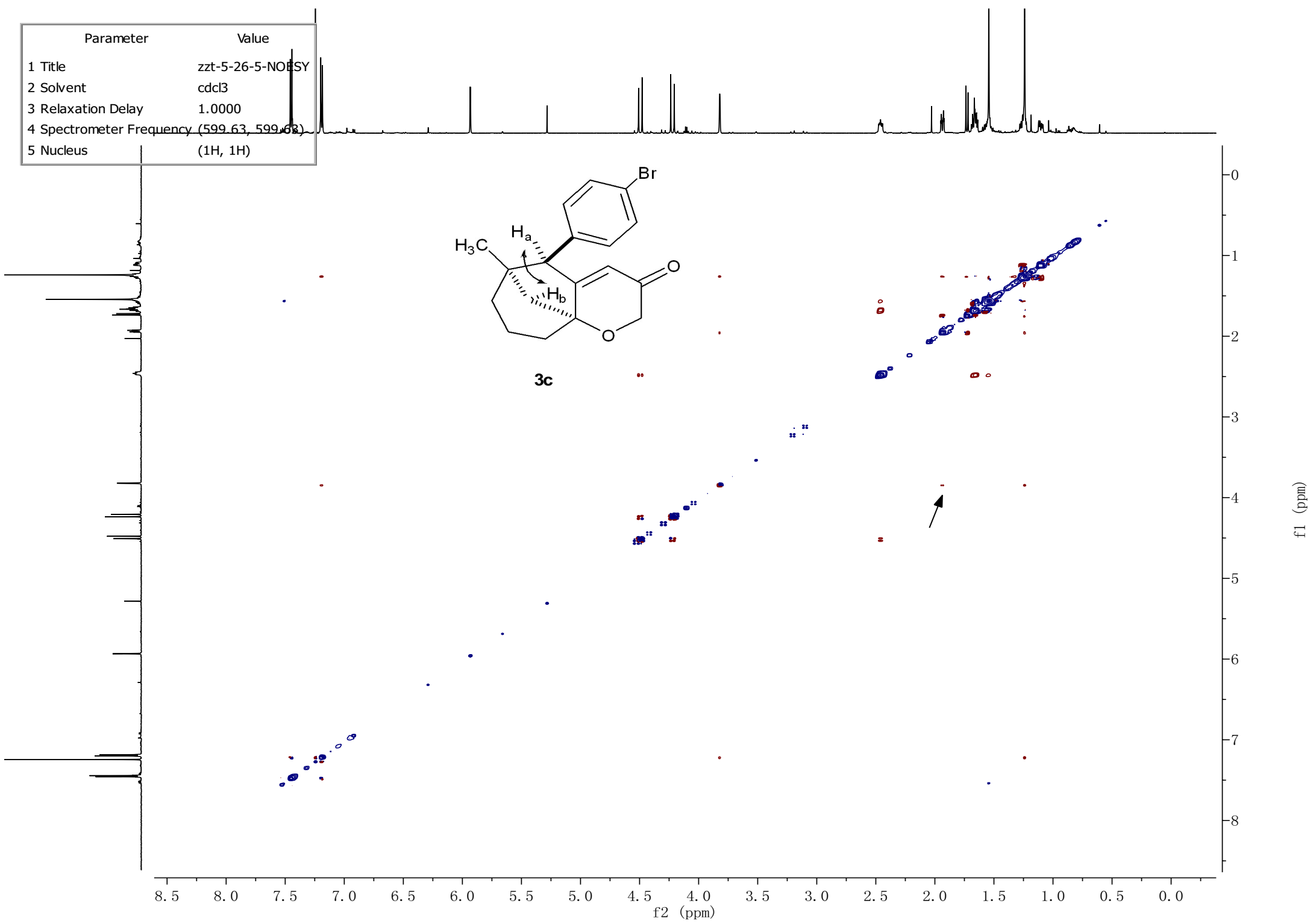
Parameter	Value
1 Title	zzt-5-26-5-H-new
2 Solvent	cdcl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	599.64



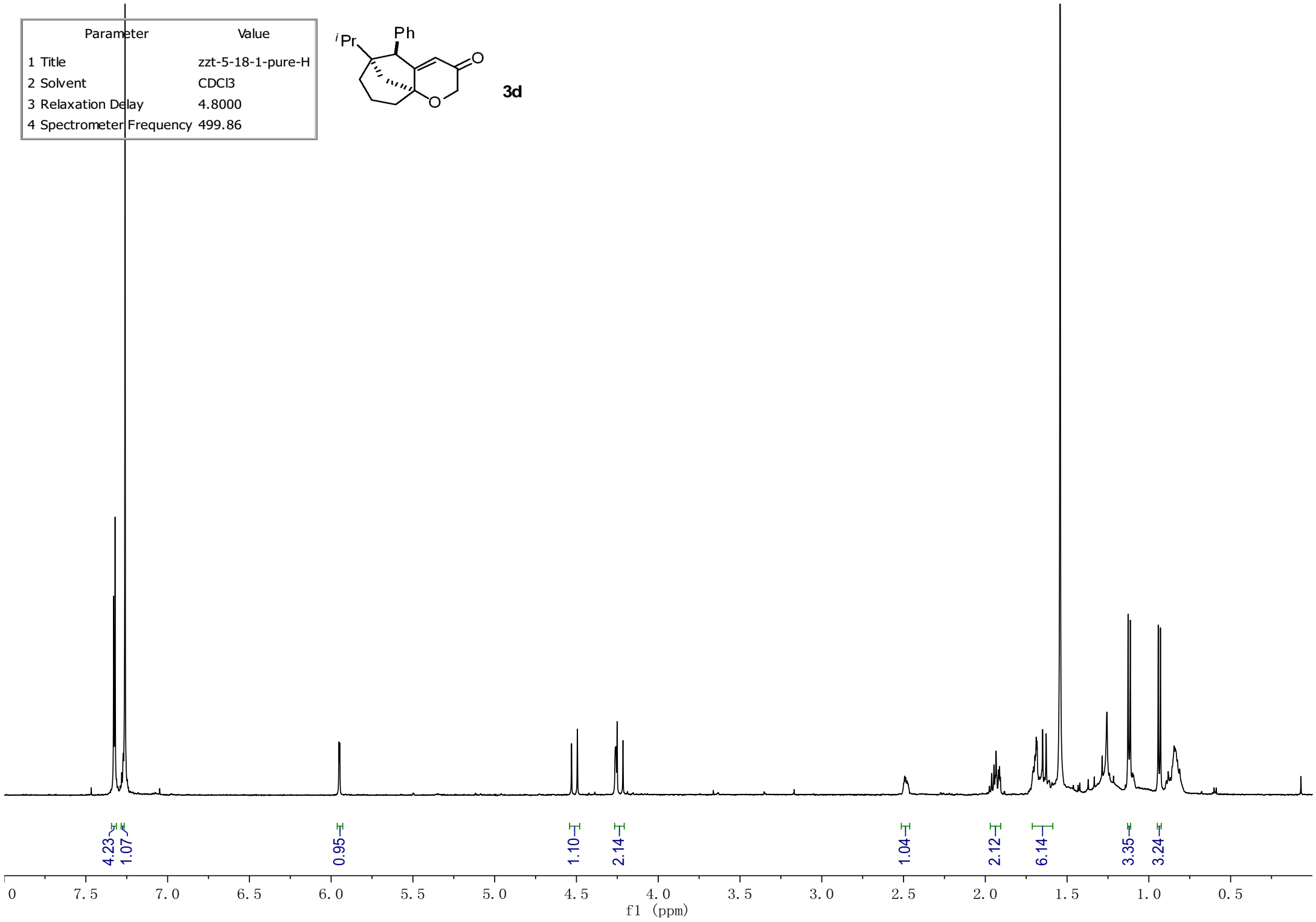
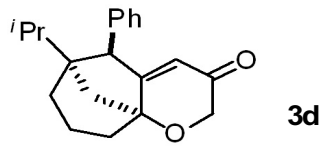
Parameter	Value
1 Title	zzt-5-26-5-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



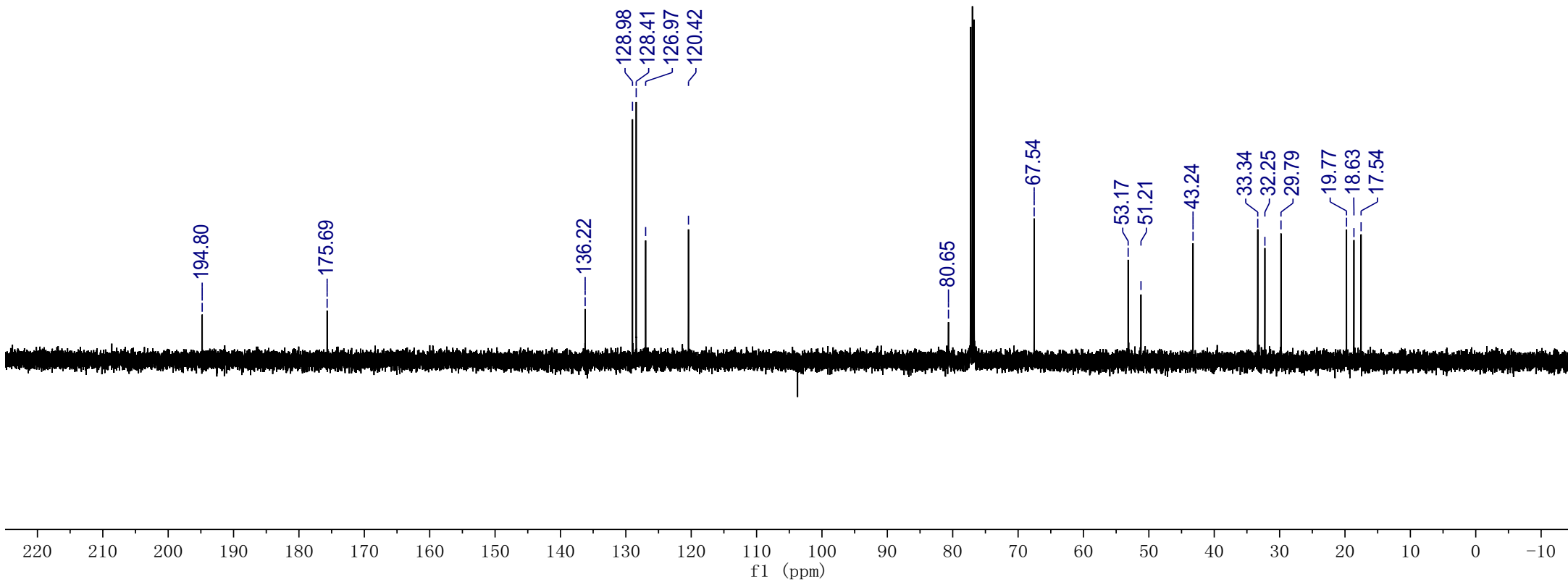
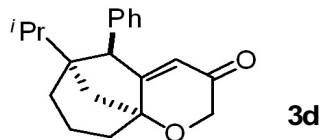
Parameter	Value
1 Title	zzt-5-26-5-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



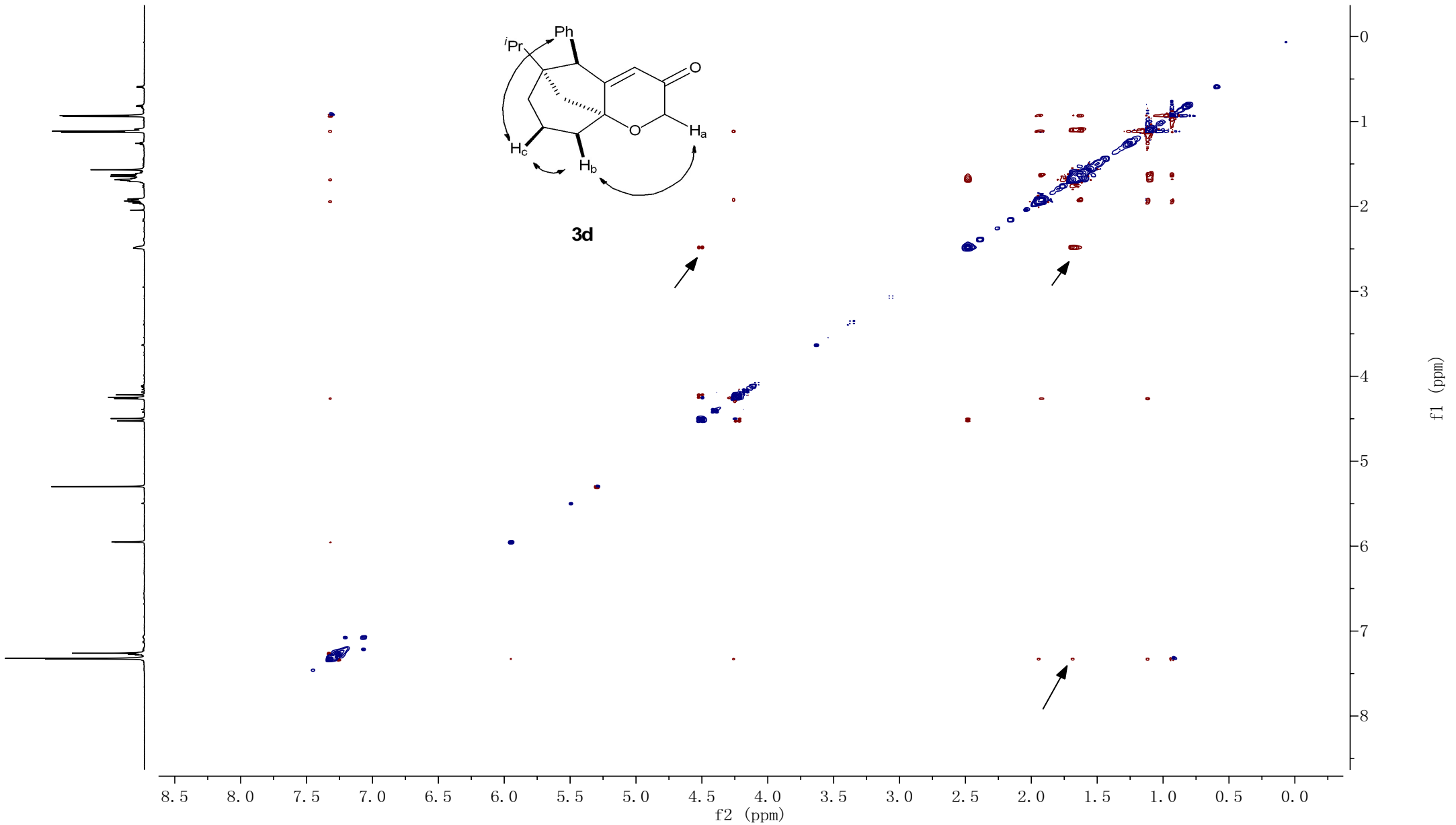
Parameter	Value
1 Title	zzt-5-18-1-pure-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



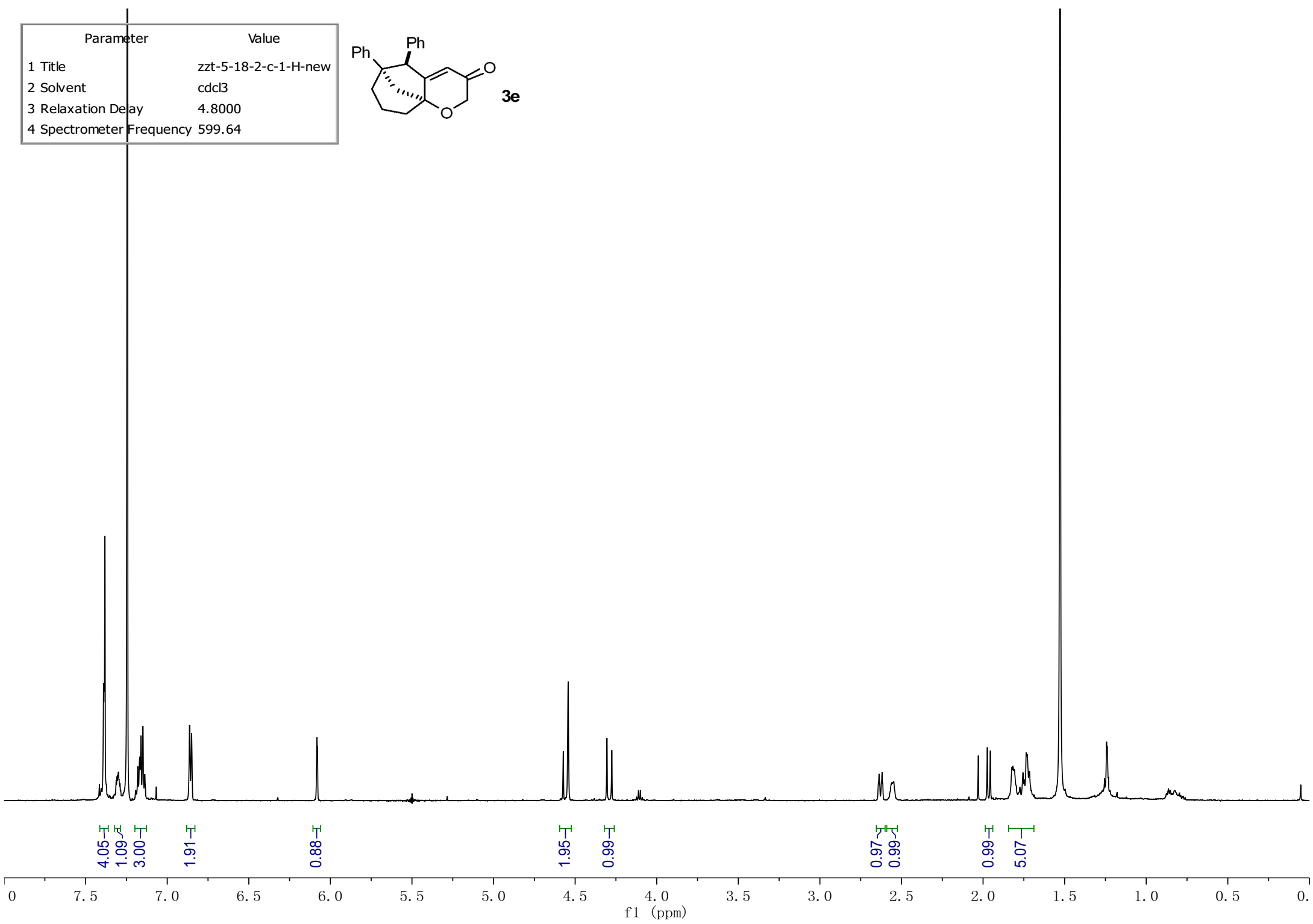
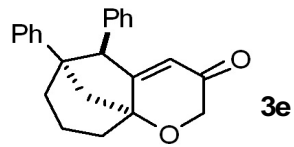
Parameter	Value
1 Title	zzt-5-18-1-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



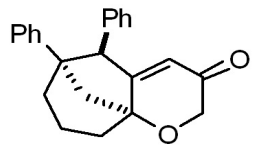
Parameter	Value
1 Title	zzt-5-18-1-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



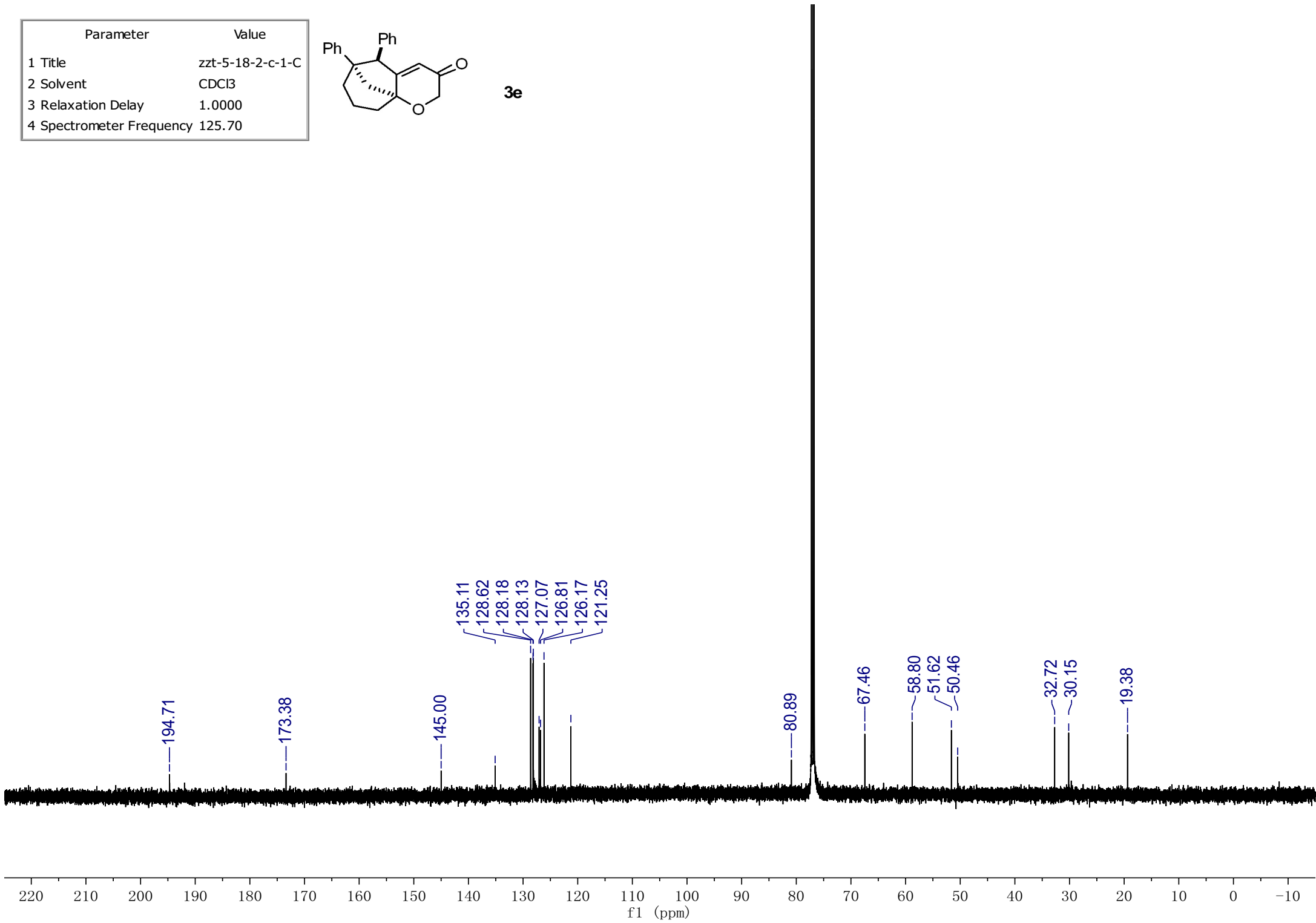
Parameter	Value
1 Title	zzt-5-18-2-c-1-H-new
2 Solvent	cdcl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	599.64



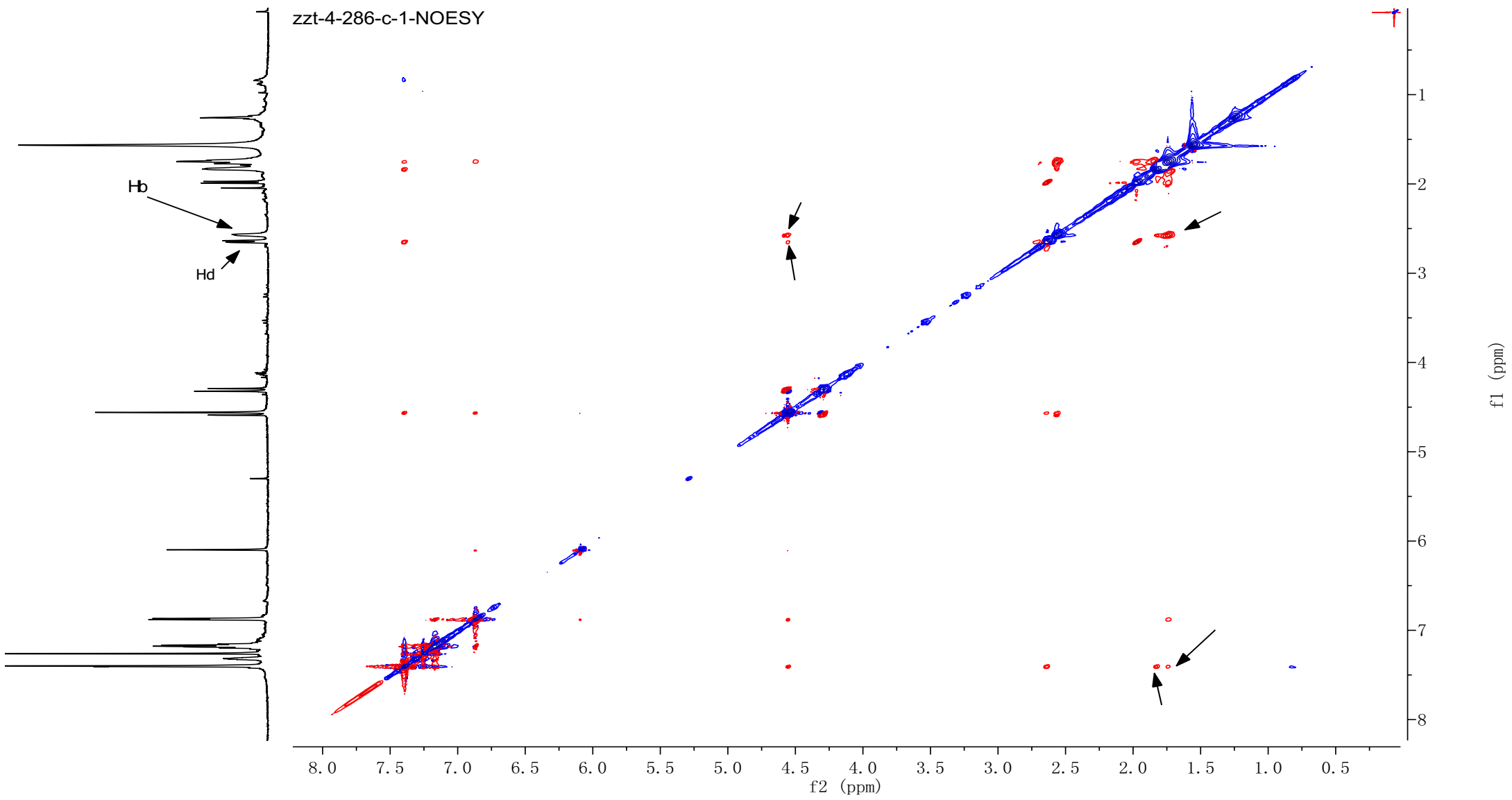
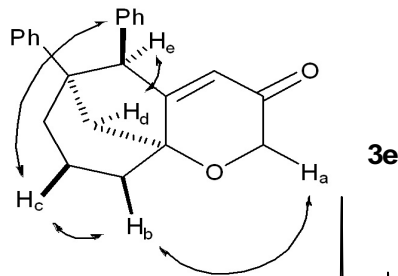
Parameter	Value
1 Title	zzt-5-18-2-c-1-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



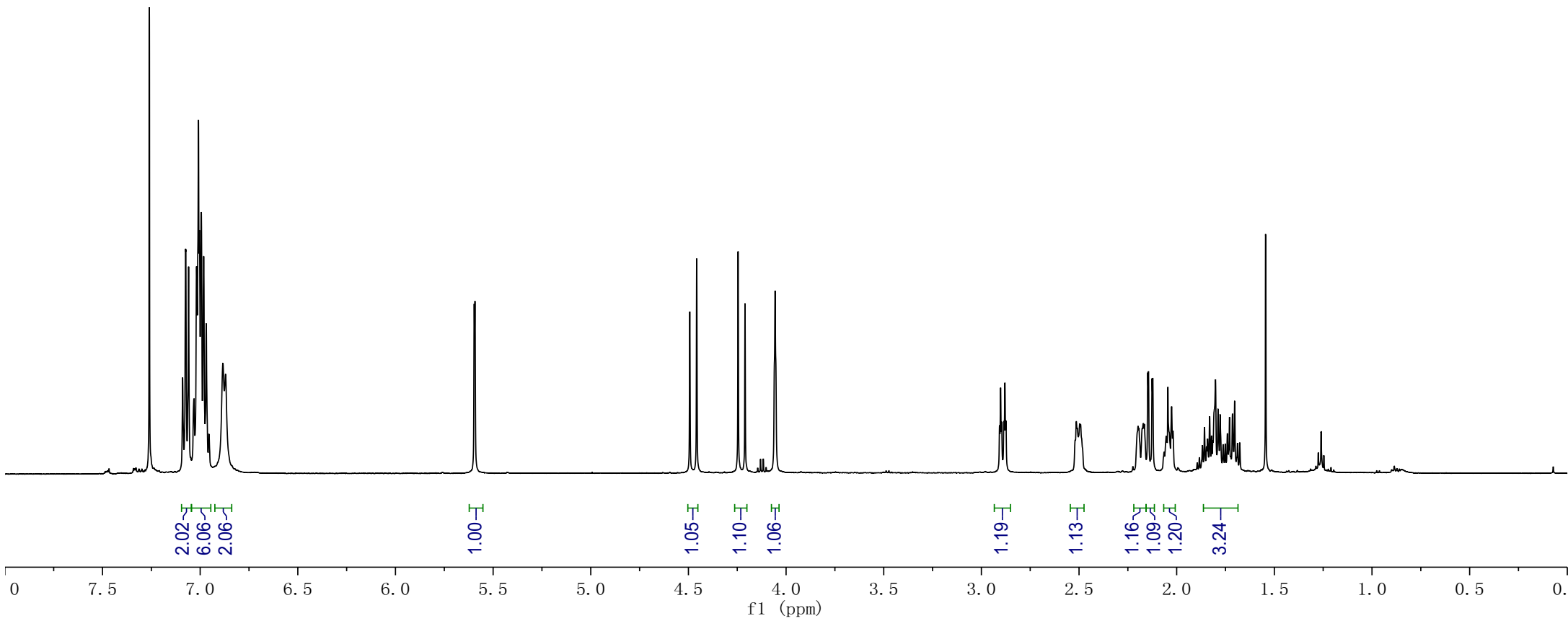
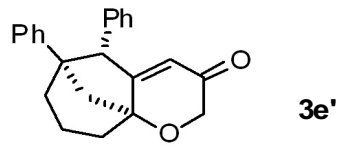
3e



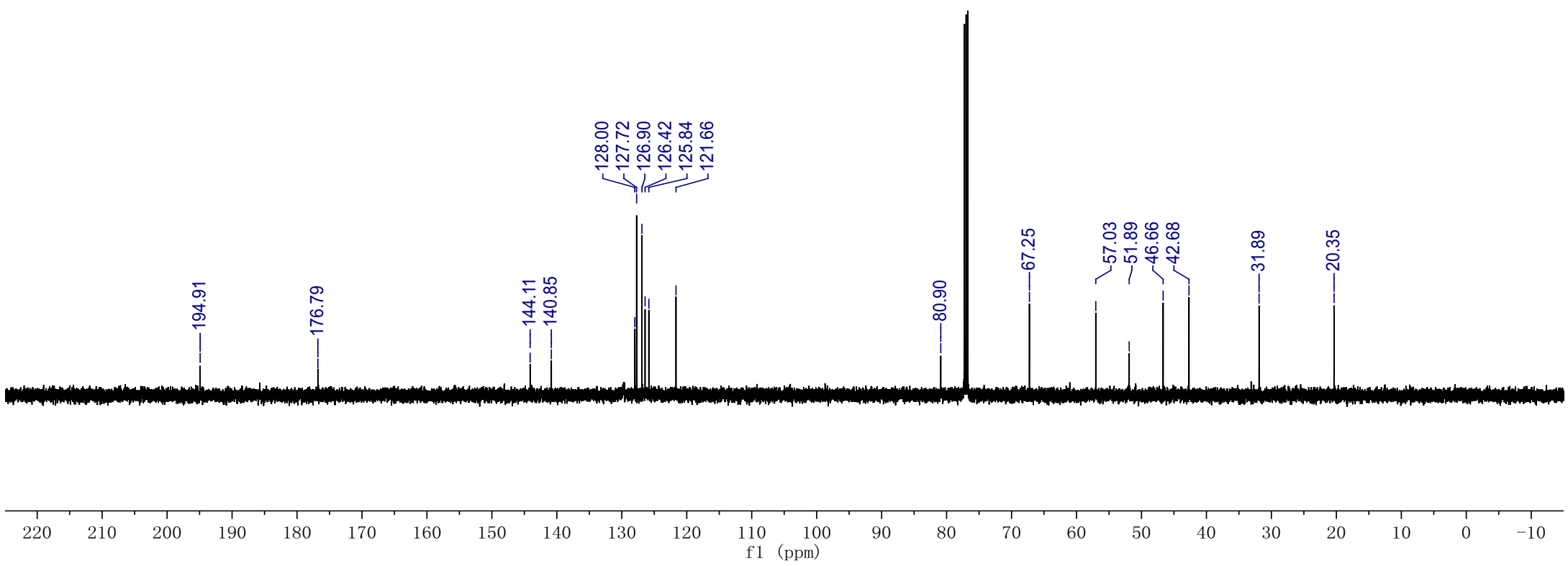
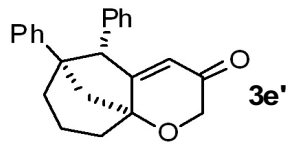
Parameter	Value
1 Title	zzt-4-286-c-1-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



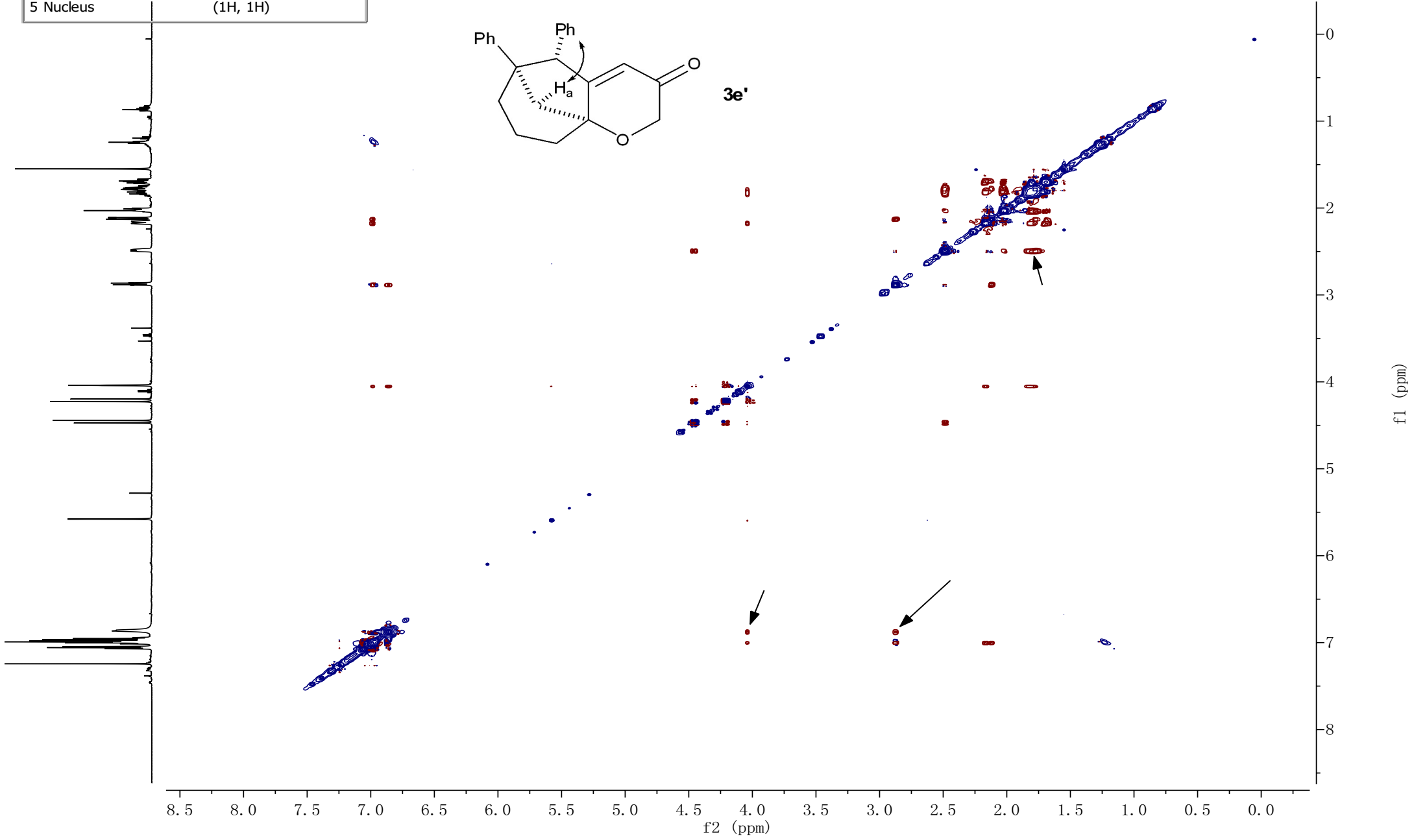
Parameter	Value
1 Title	zzt-4-18-2-c-2-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



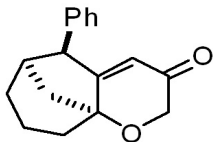
Parameter	Value
1 Title	zzt-5-18-2-c-2-Cnew
2 Solvent	CDCl3
3 Spectrometer Frequency	125.70
4 Nucleus	13C



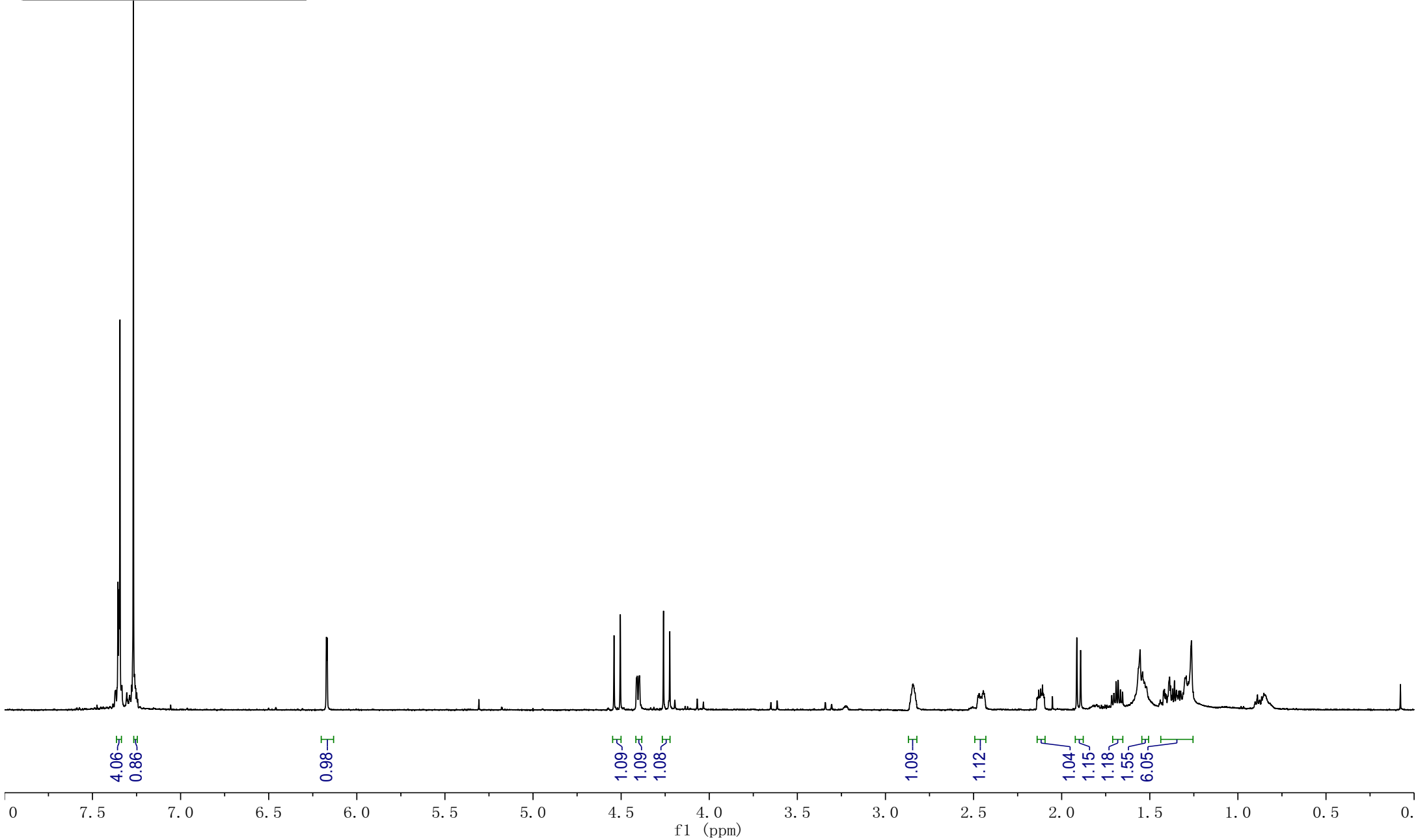
Parameter	Value
1 Title	zzt-5-18-2-c-2-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	(599.63, 599.63)
5 Nucleus	(1H, 1H)



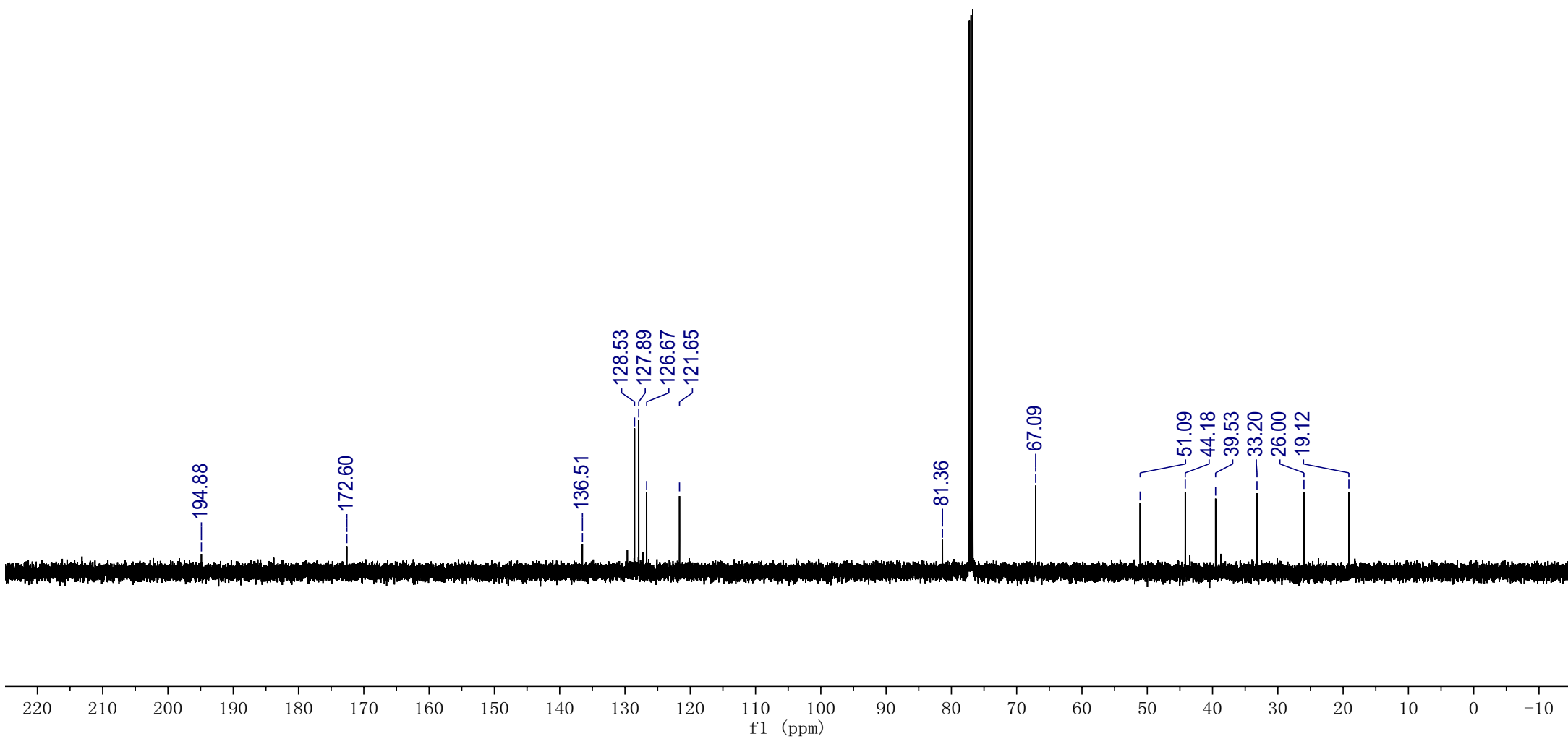
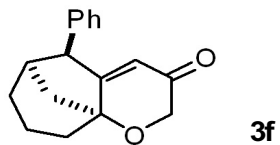
Parameter	Value
1 Title	zzt-5-26-3-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



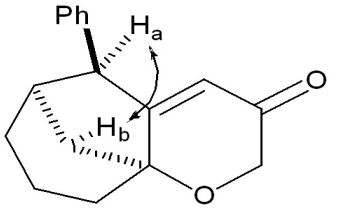
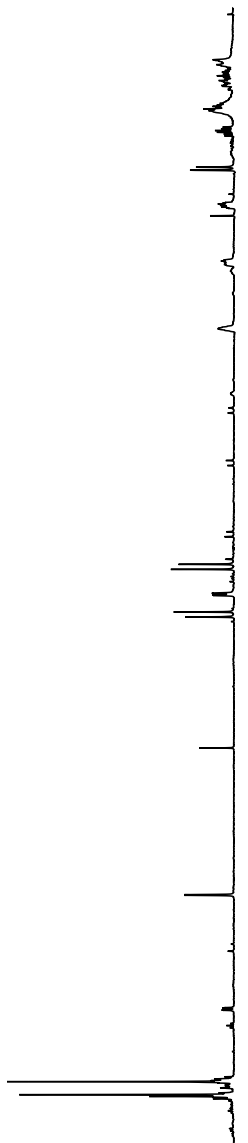
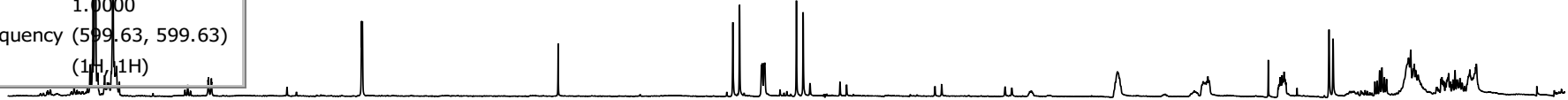
3f



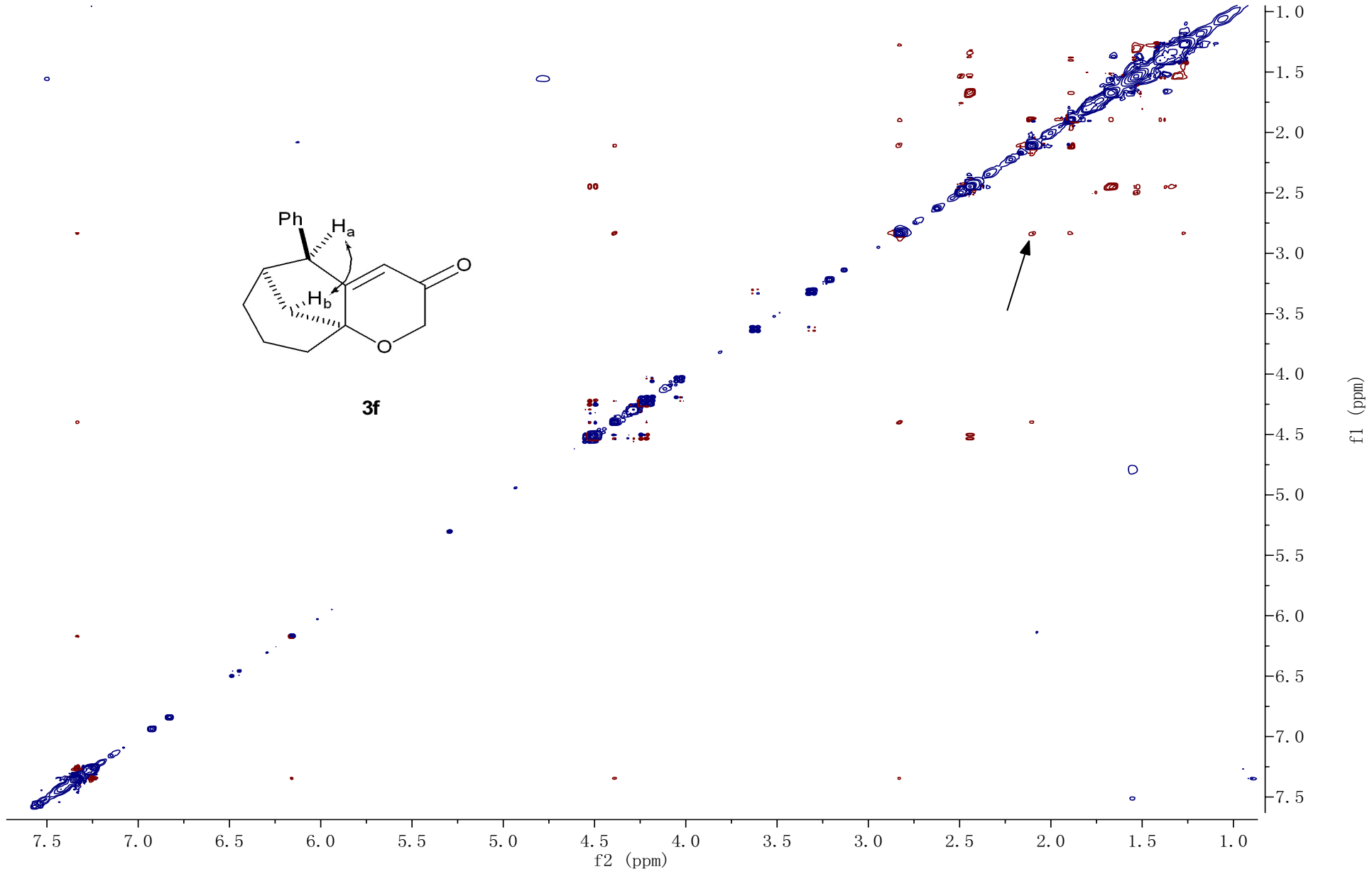
Parameter	Value
1 Title	zzt-5-26-3-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



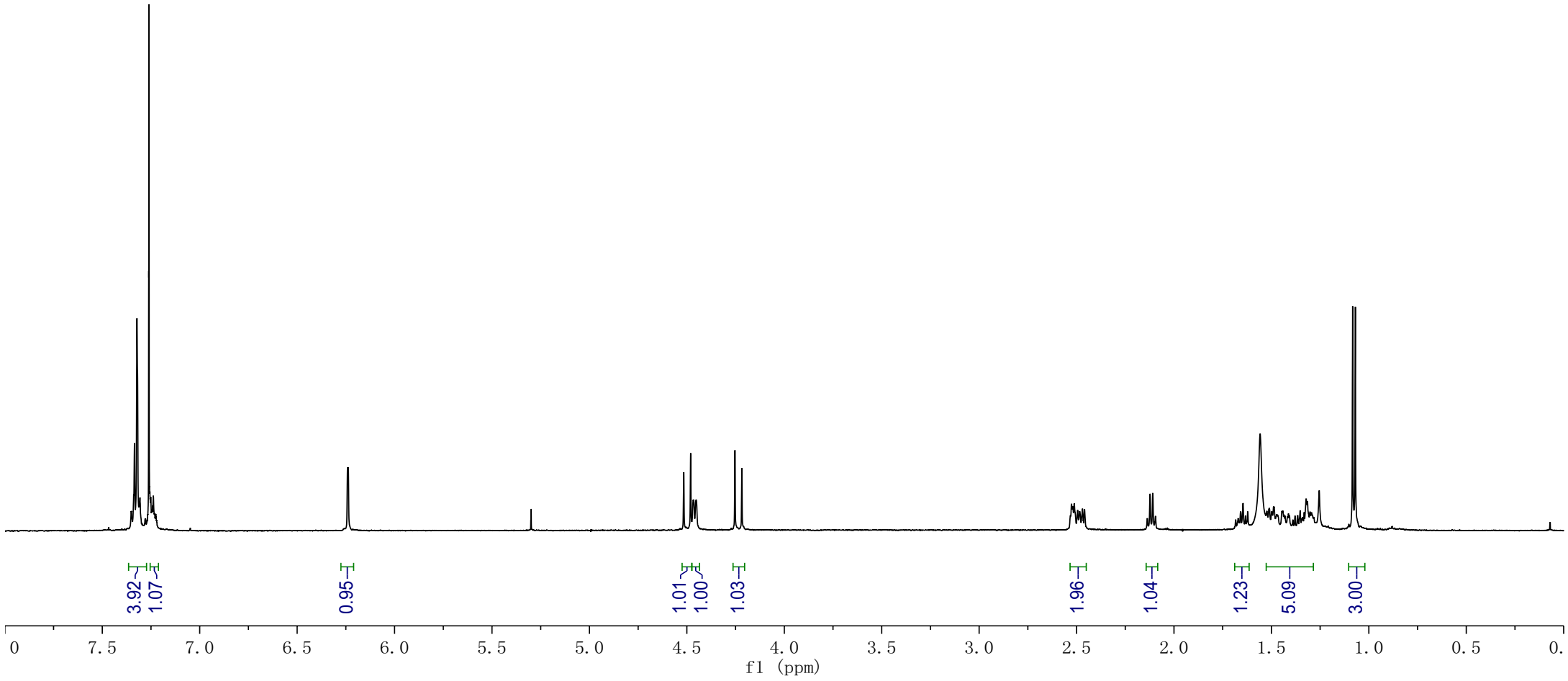
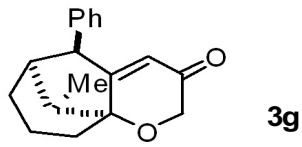
Parameter	Value
1 Title	zz1-5-26-3-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H,1H)



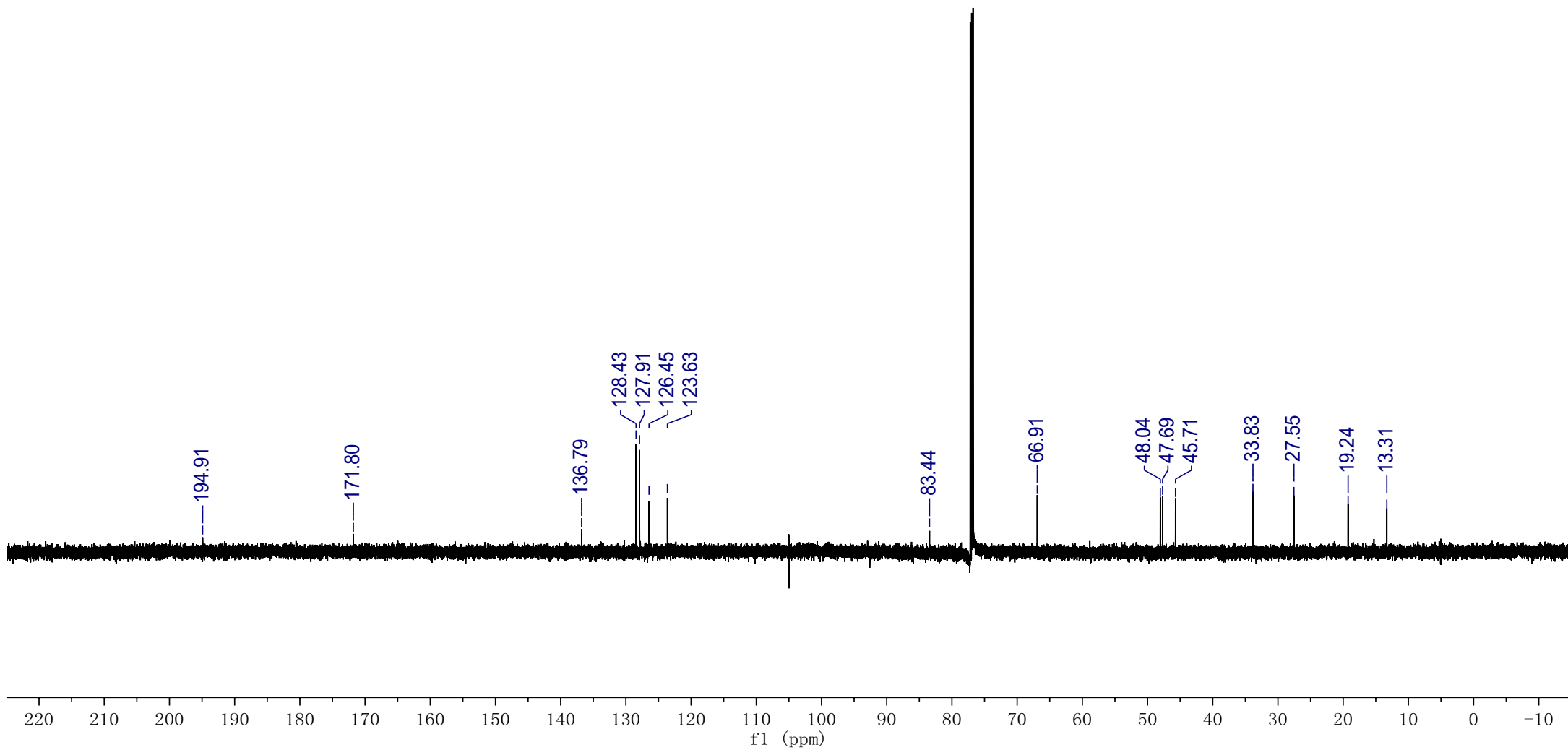
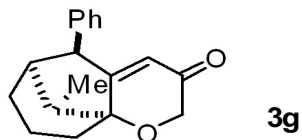
3f



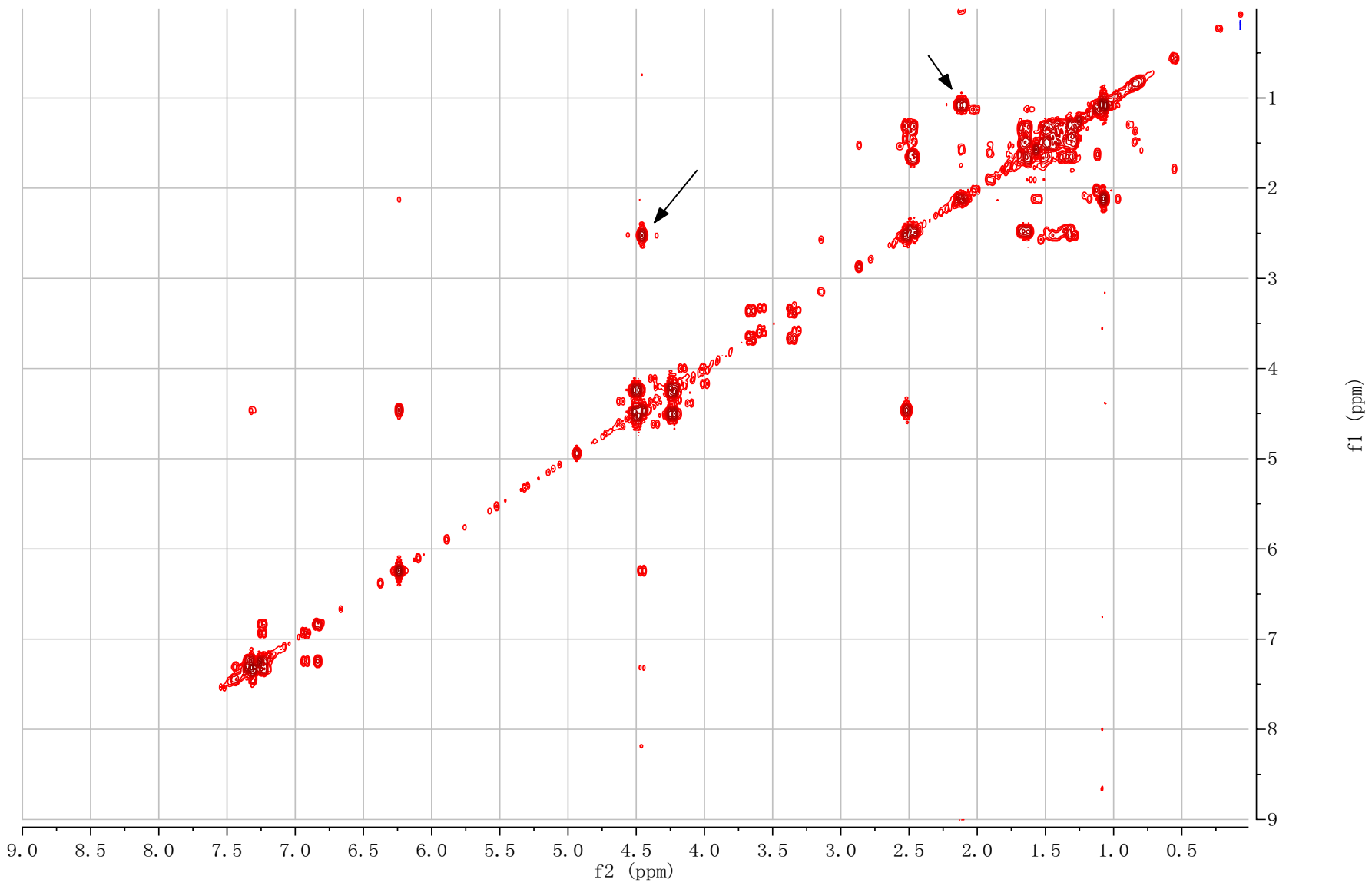
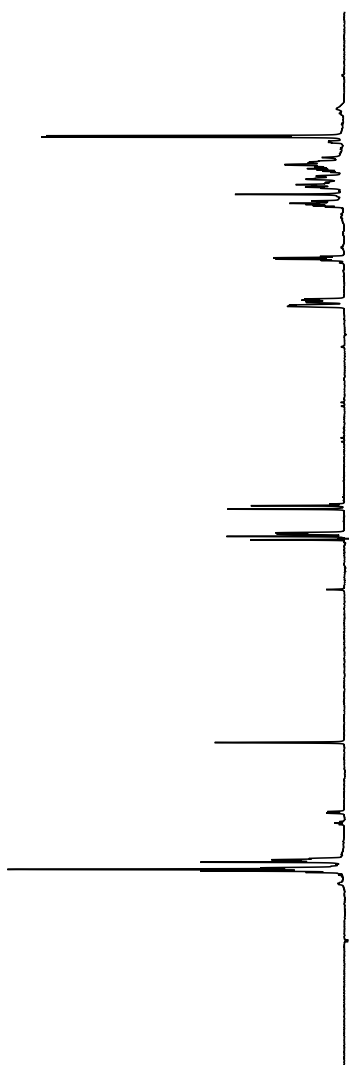
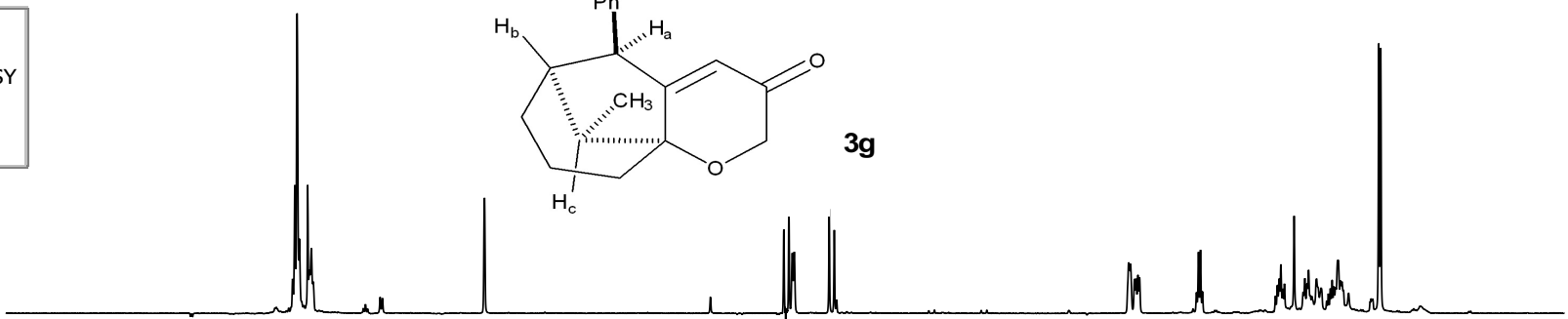
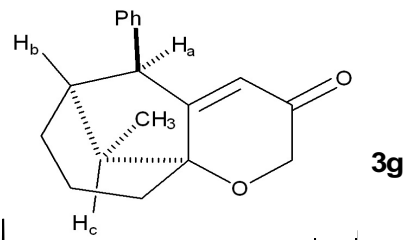
Parameter	Value
1 Title	zzt-4-170-1-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



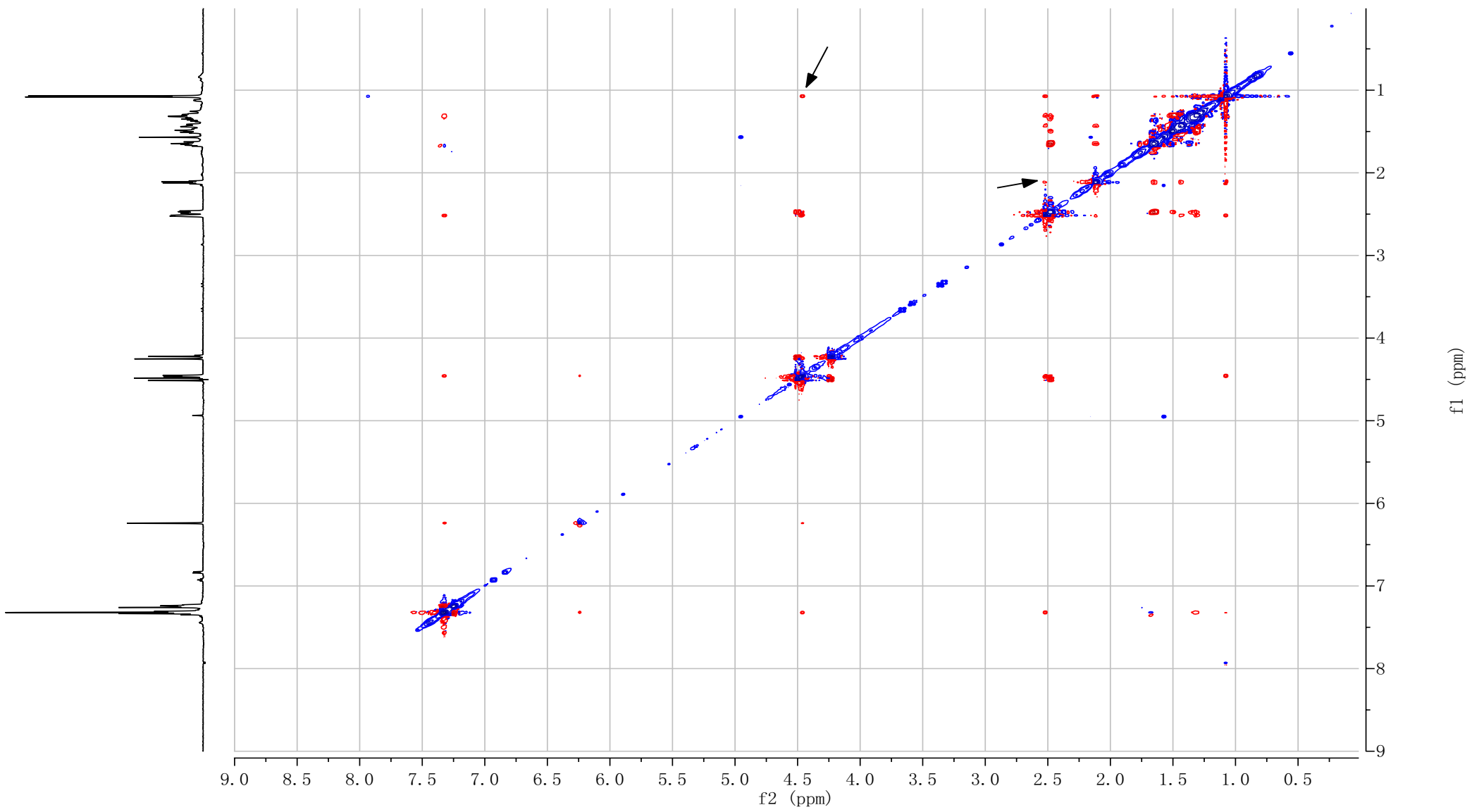
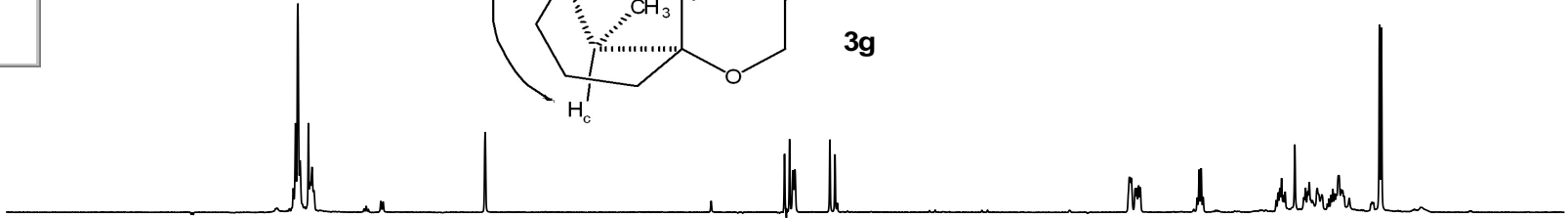
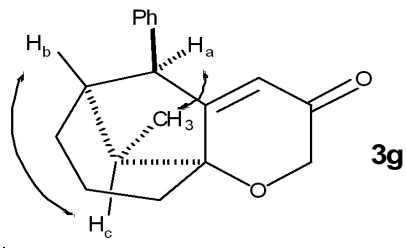
Parameter	Value
1 Title	zzt-4-170-1-C
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	150.79



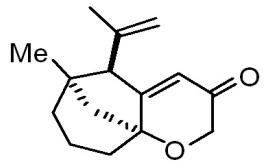
Parameter	Value
1 Title	zzt-4-170-1-COSY
2 Solvent	"cdcl3"
3 Relaxation Delay	1.0000



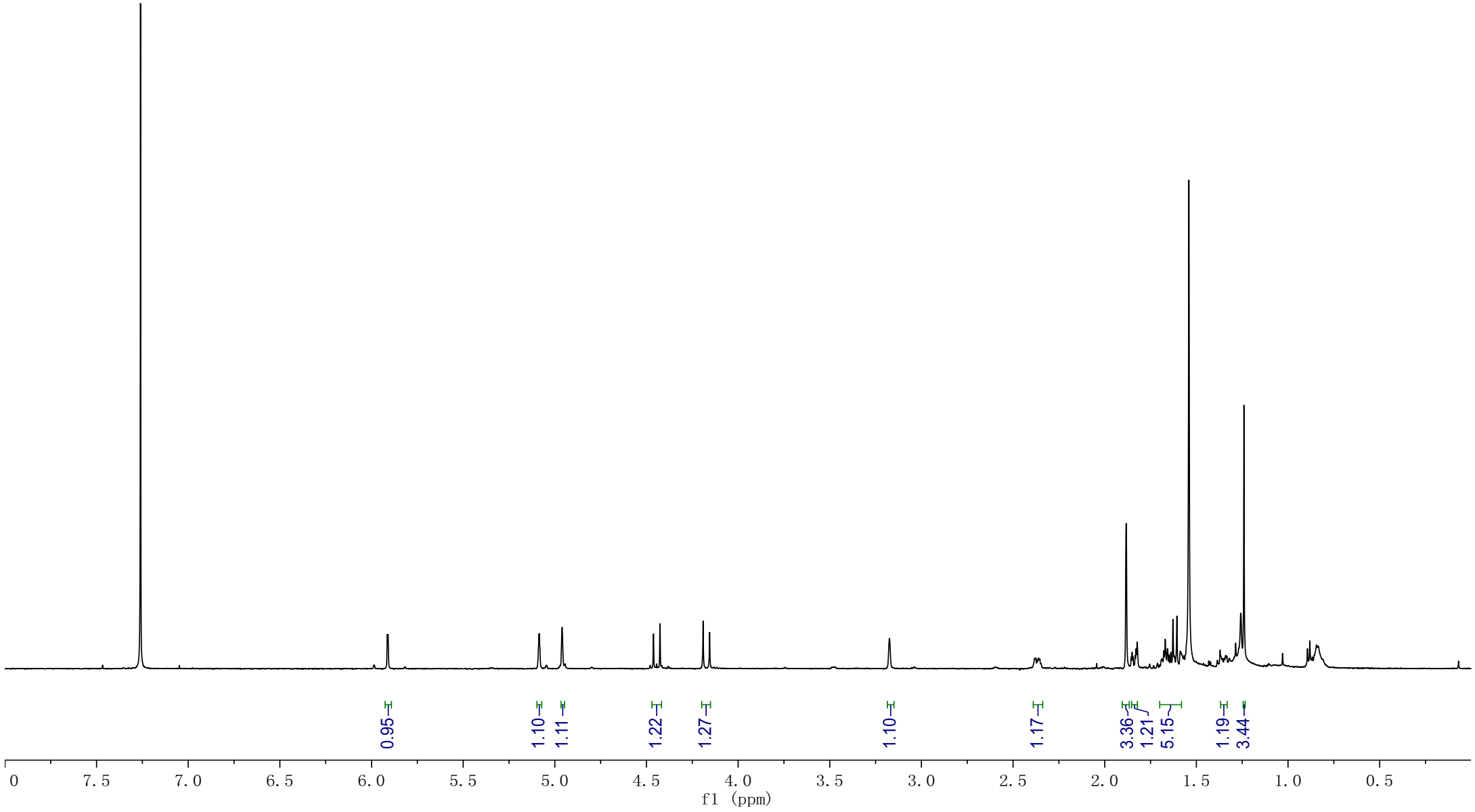
Parameter	Value
1 Title	zzt-4-170-1-NOESY
2 Solvent	"cdcl3"
3 Relaxation Delay	1.0000



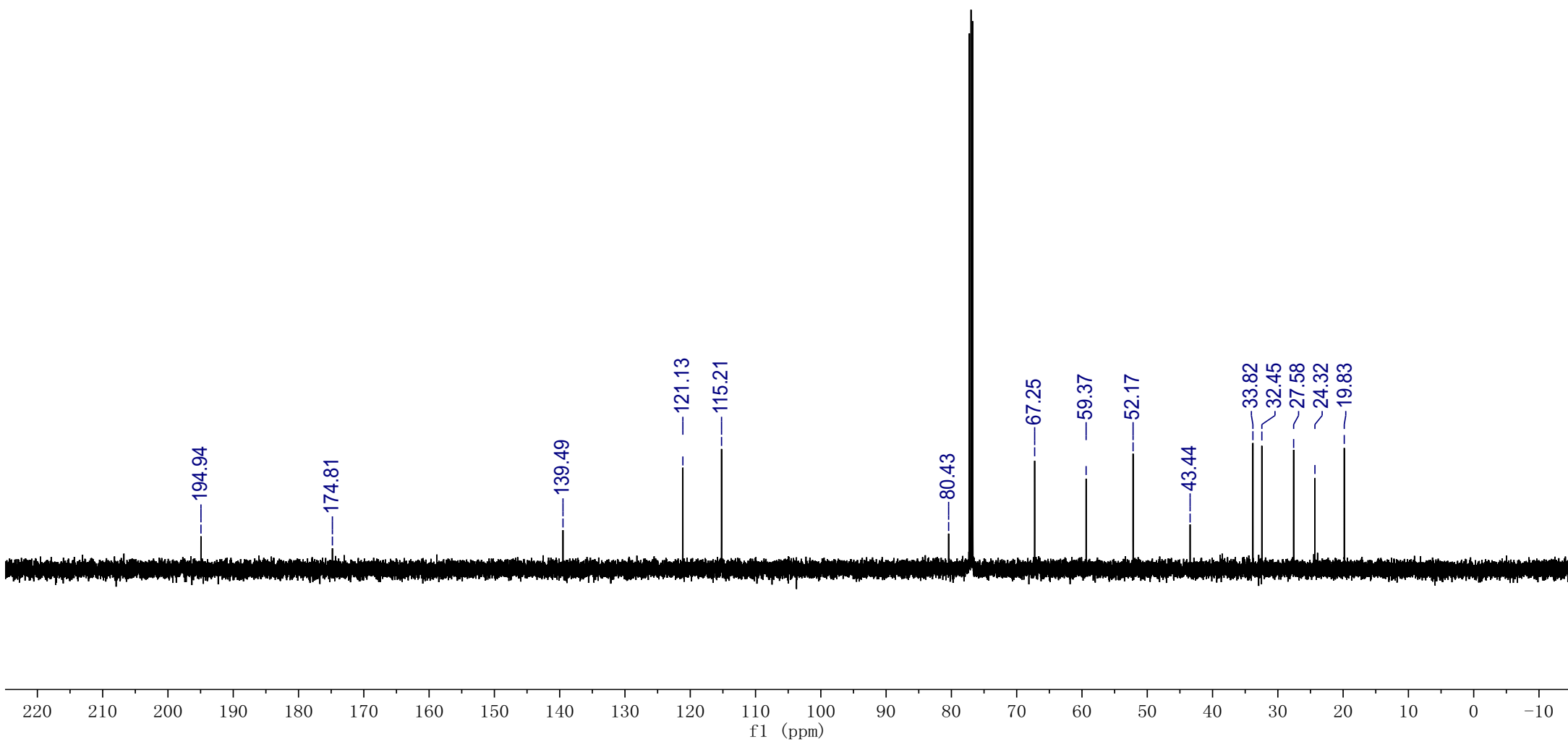
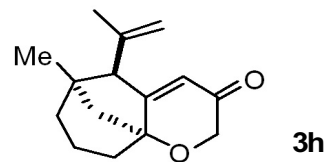
Parameter	Value
1 Title	zzt-5-26-2-pure-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



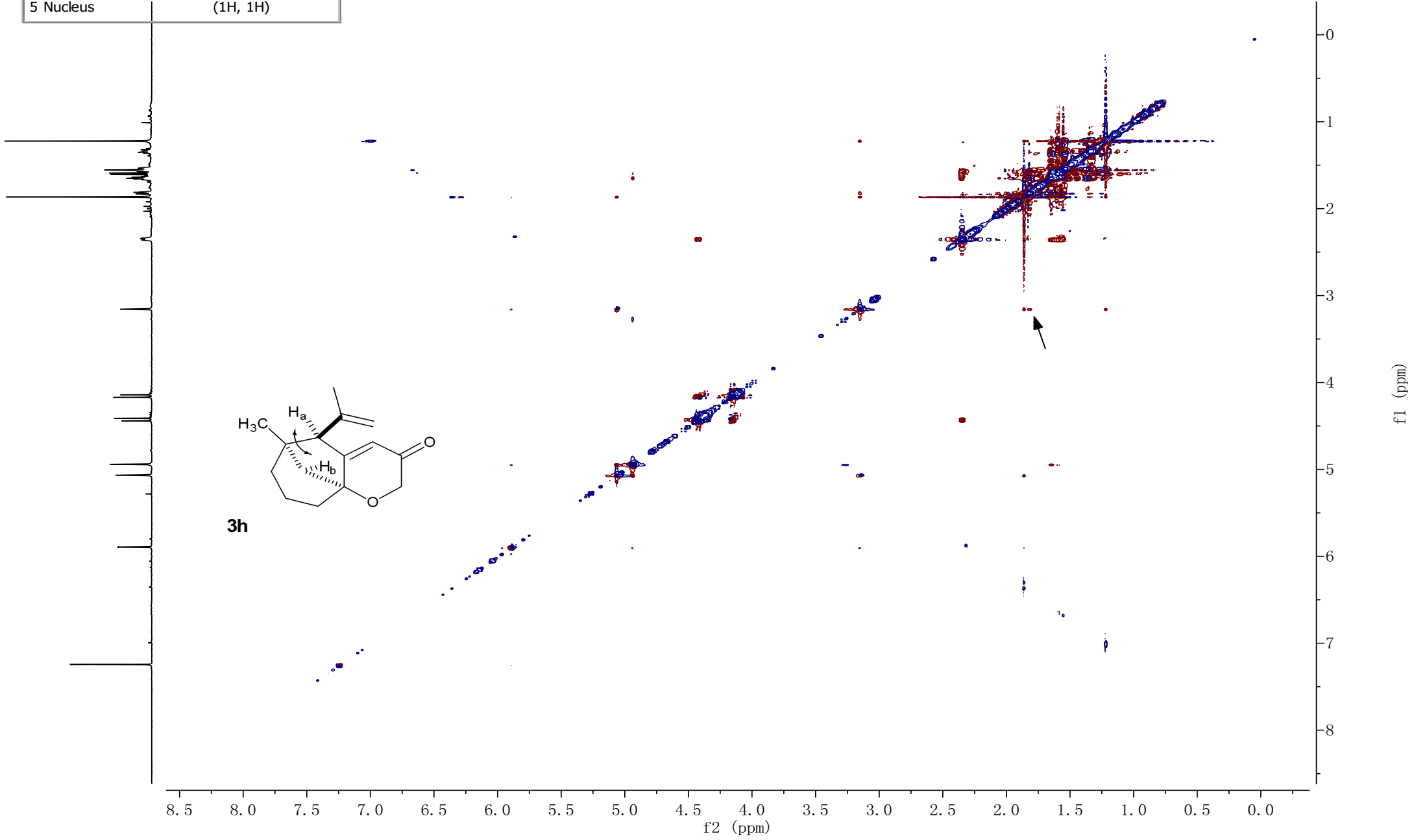
3h



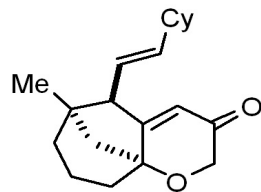
Parameter	Value
1 Title	zzt-5-26-2-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



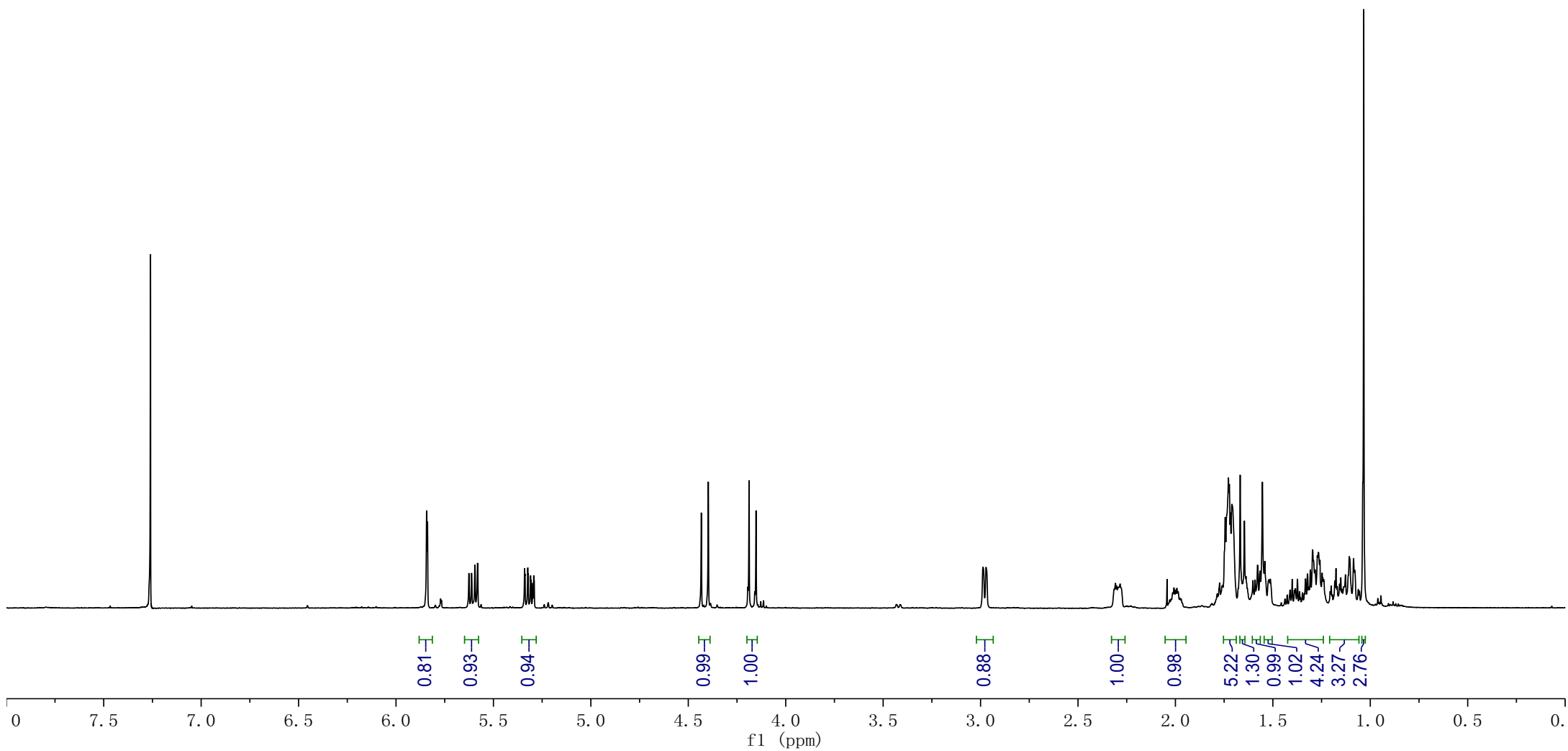
Parameter	Value
1 Title	zzt-5-26-2-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



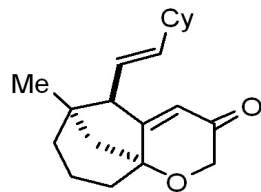
Parameter	Value
1 Title	zzt-5-26-1-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



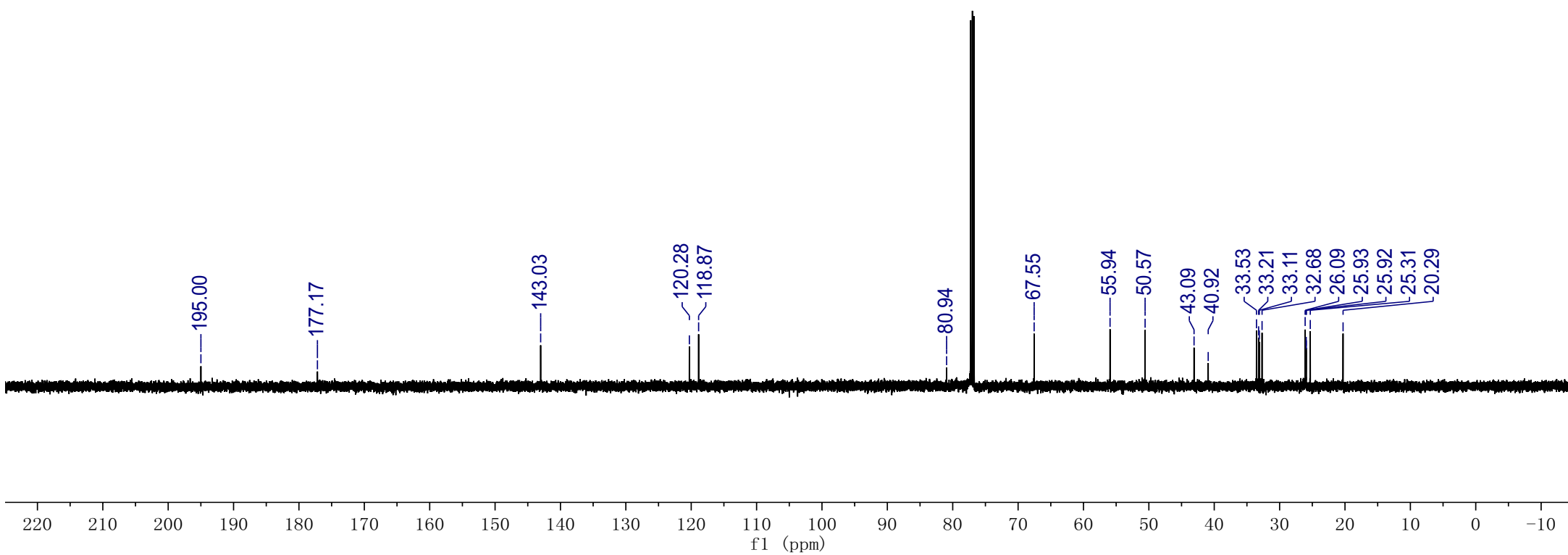
3i



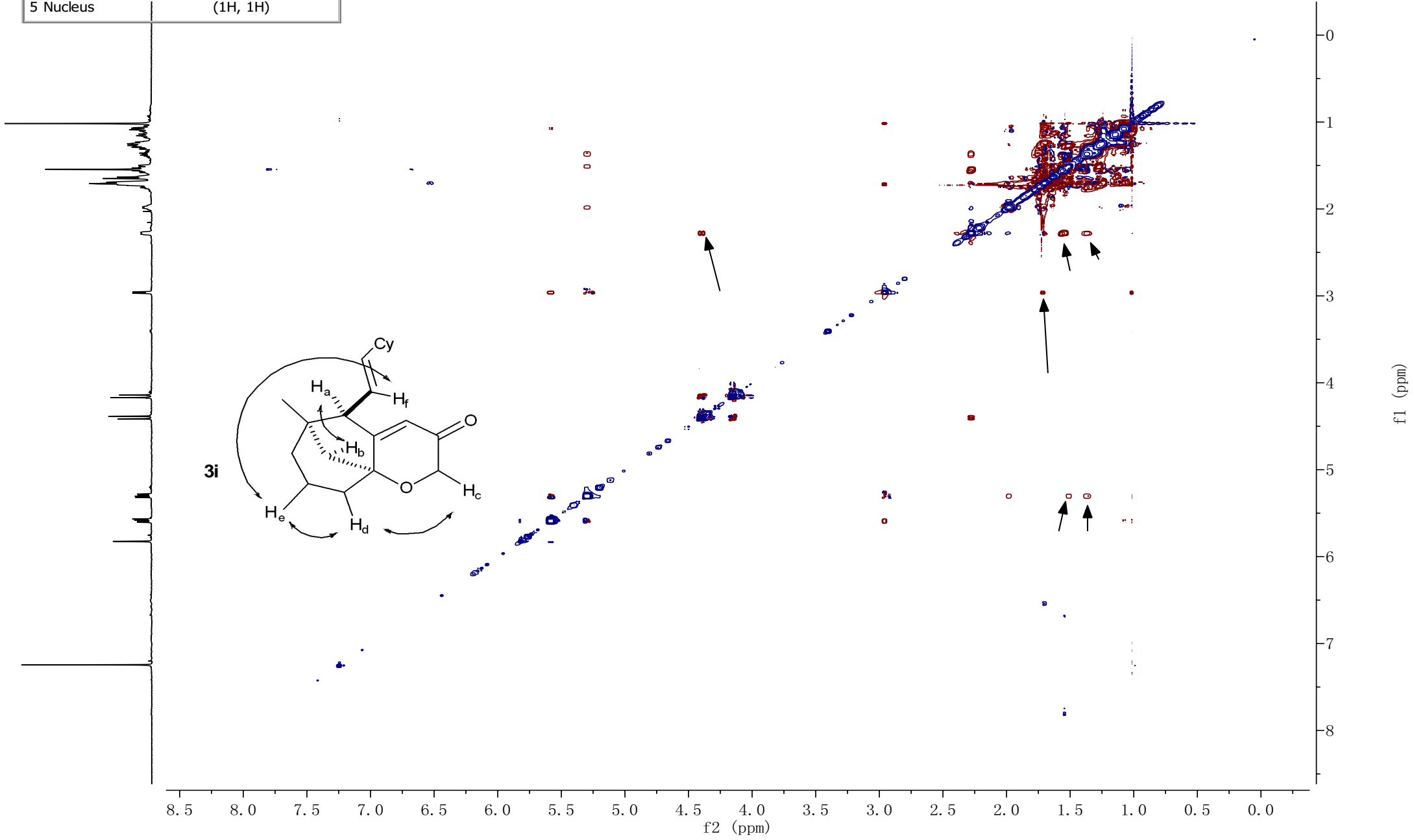
Parameter	Value
1 Title	zzt-5-26-1-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



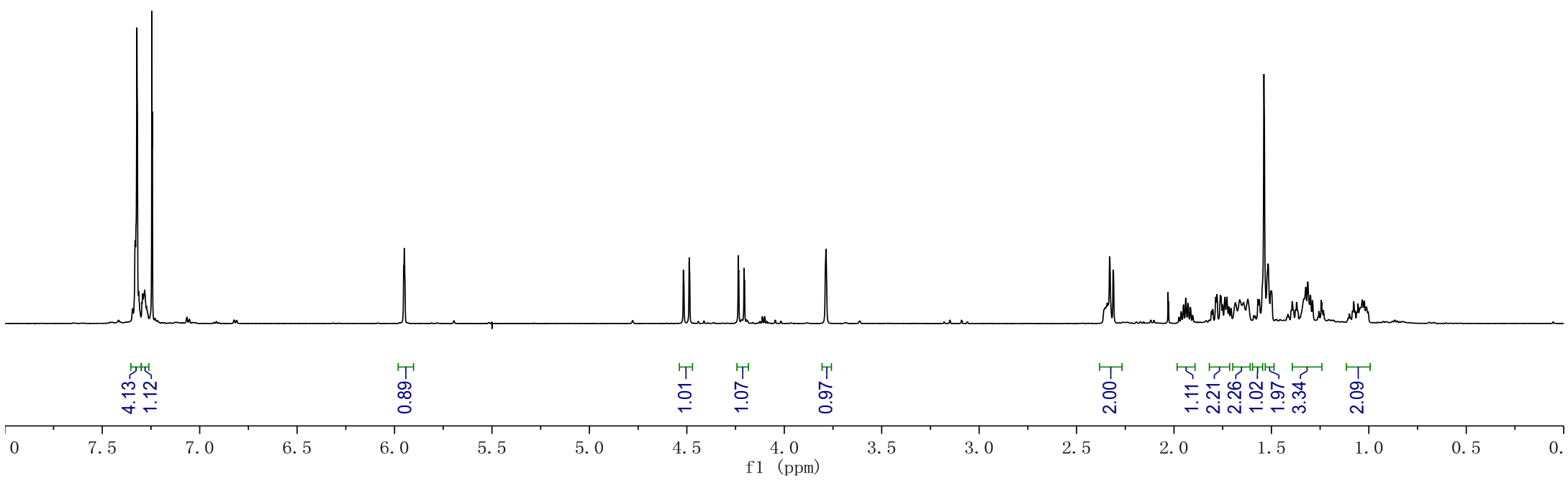
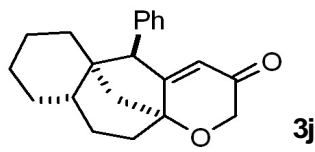
3i



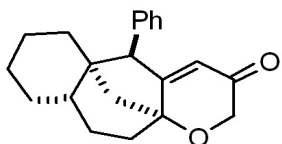
Parameter	Value
1 Title	zzt-5-26-1-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	(599.63, 599.63)
5 Nucleus	(1H, 1H)



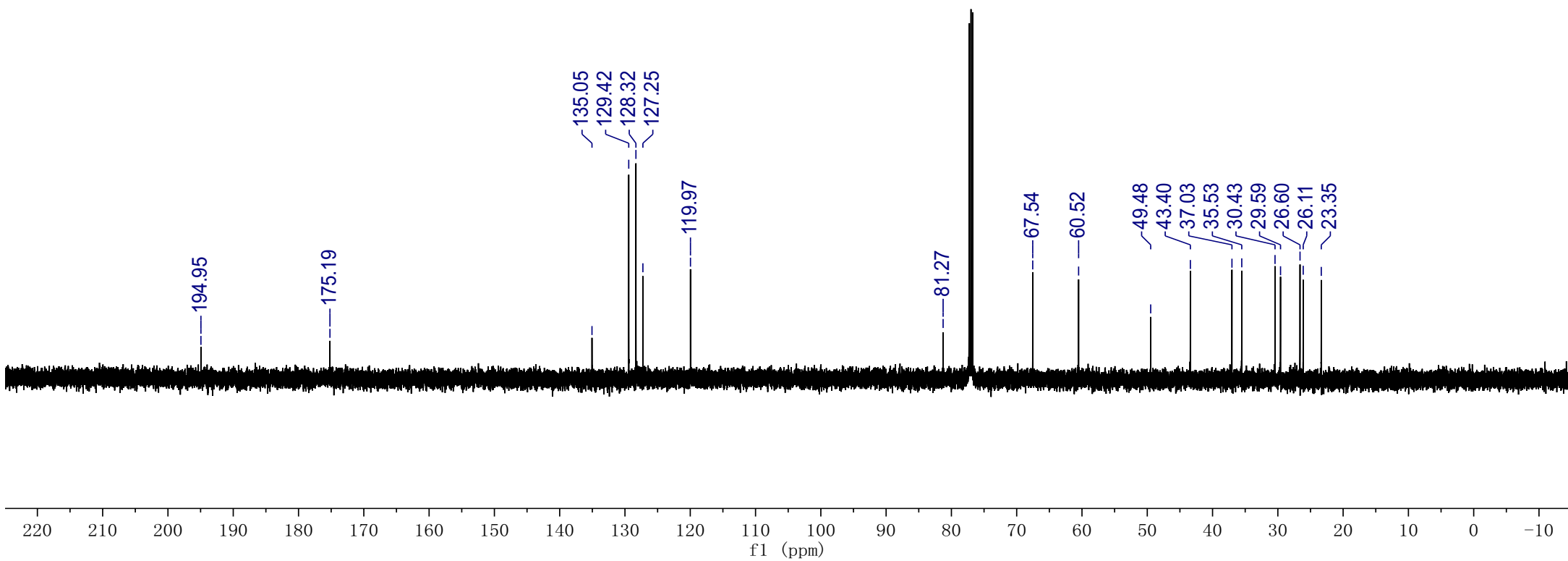
Parameter	Value
1 Title	zzt-5-26-6-H-new
2 Solvent	cdcl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	599.64



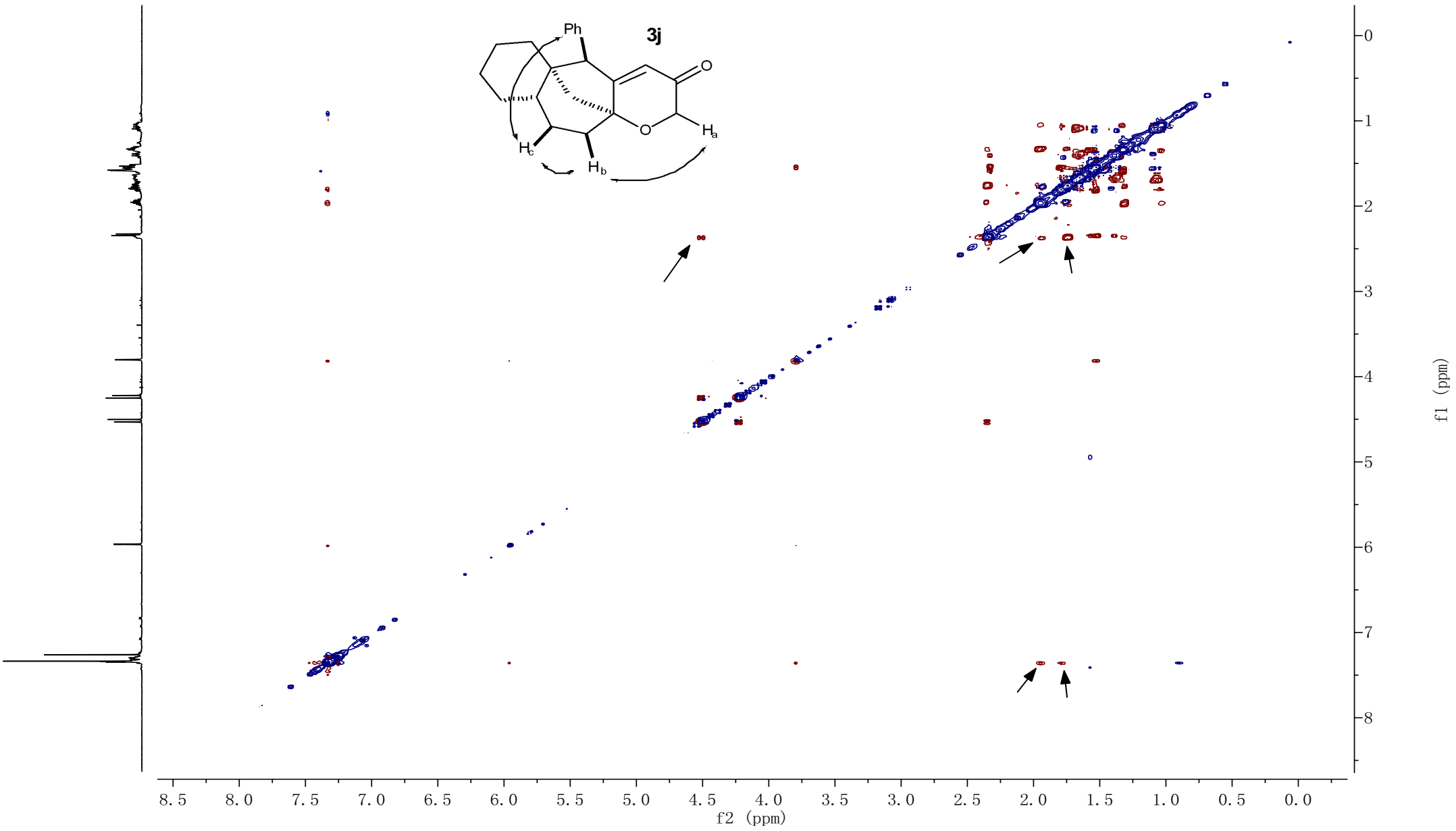
Parameter	Value
1 Title	zzt-5-26-6-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



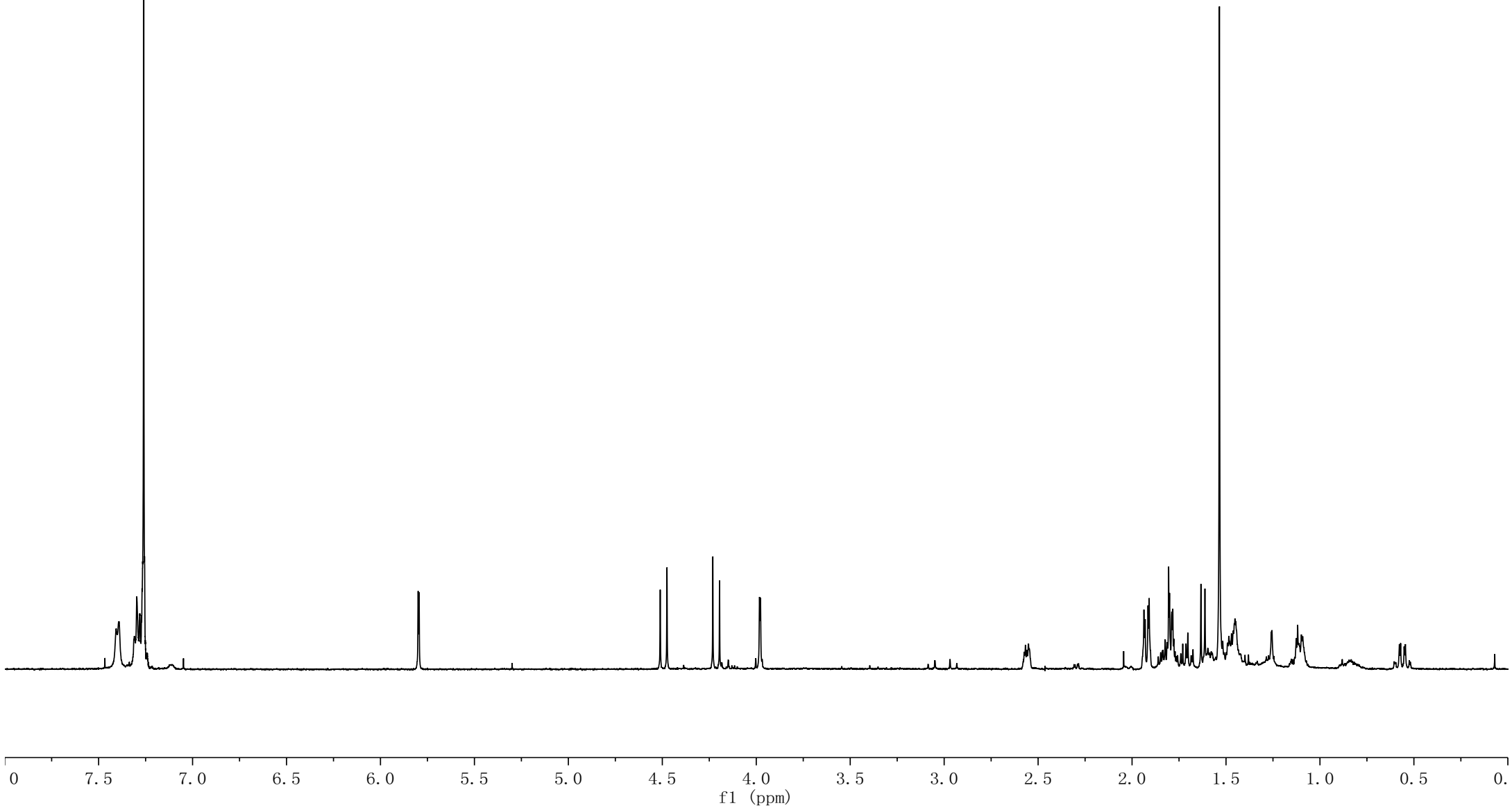
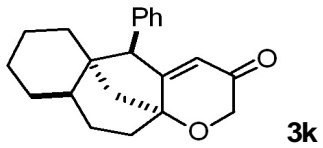
3j



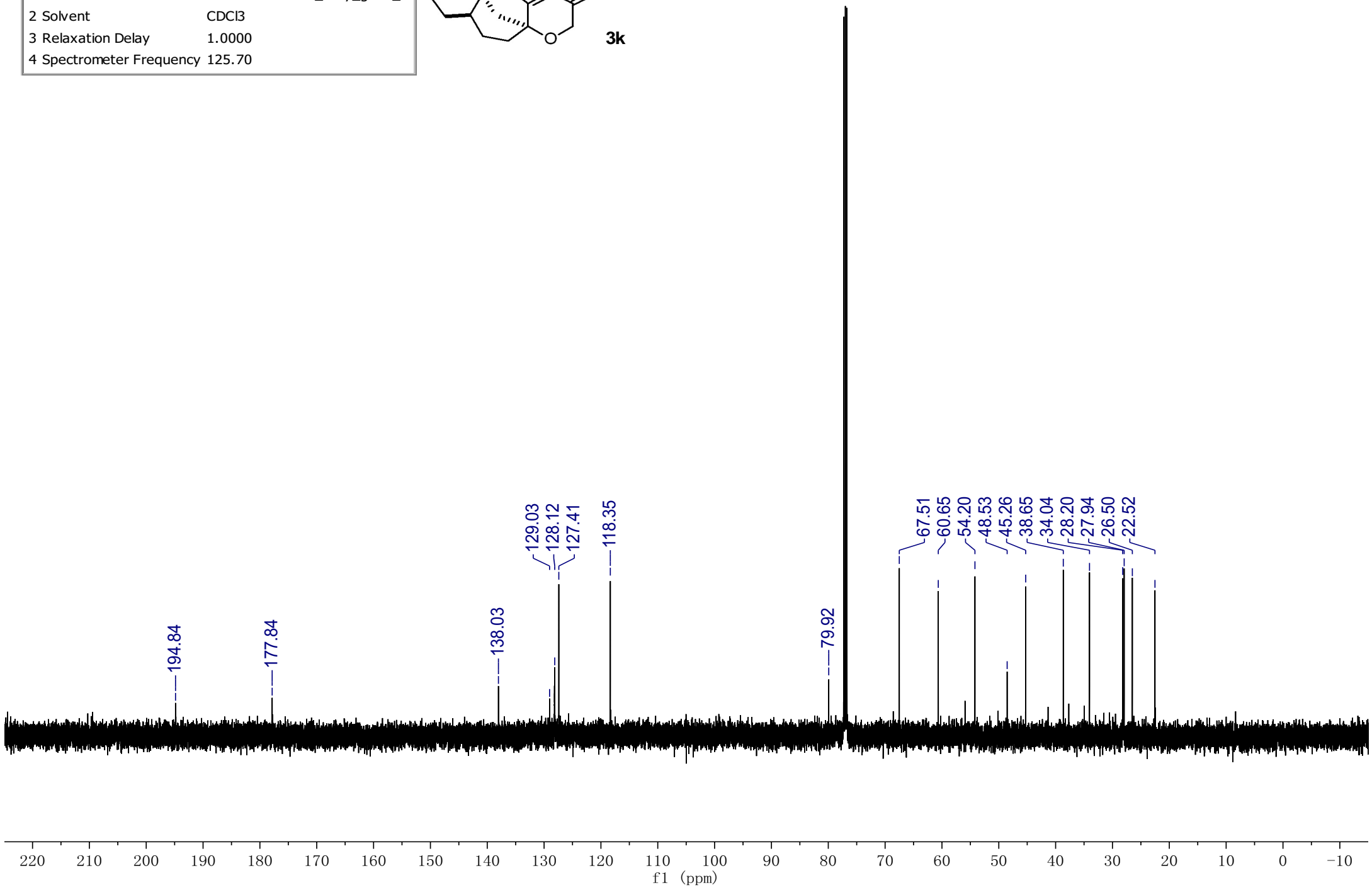
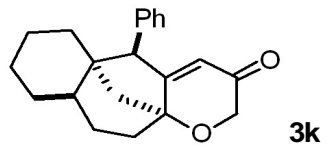
Parameter	Value
1 Title	zzt-5-26-6-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



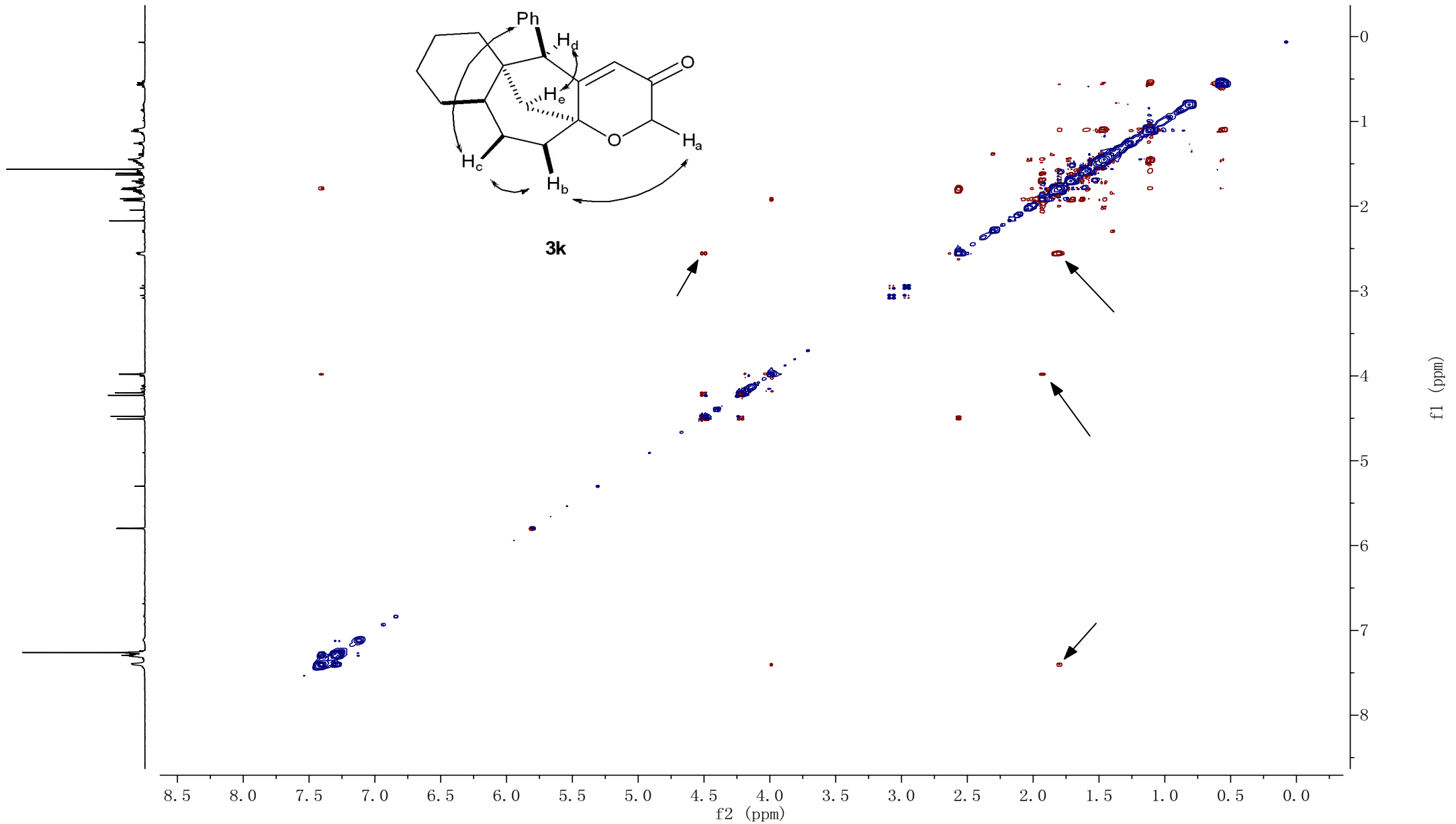
Parameter	Value
1 Title	zzt-5-18-3-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



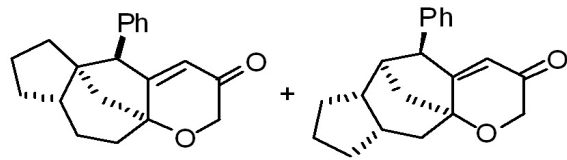
Parameter	Value
1 Title	zzt-5-18-3-C-not_very_good_2
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



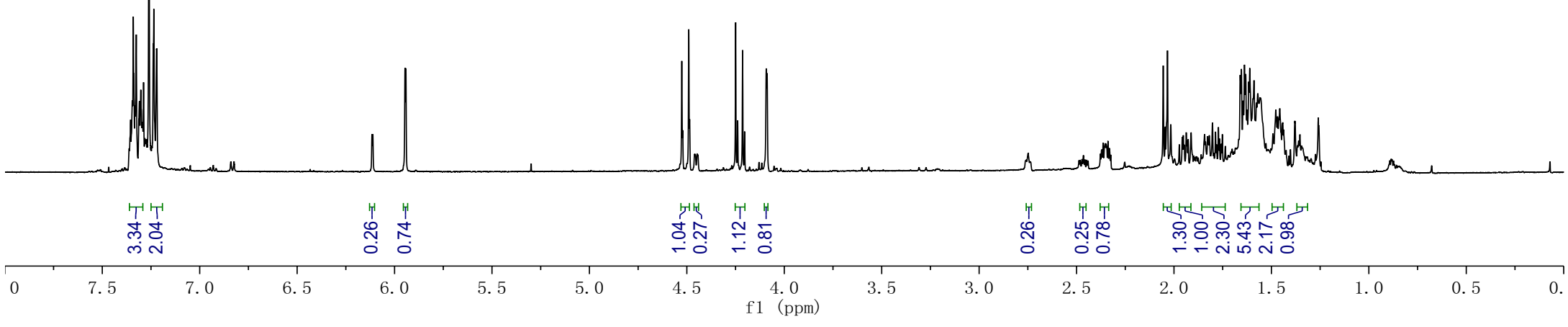
Parameter	Value
1 Title	zzt-5-18-3-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



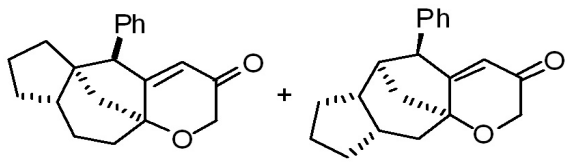
Parameter	Value
1 Title	zzt-5-26-4-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



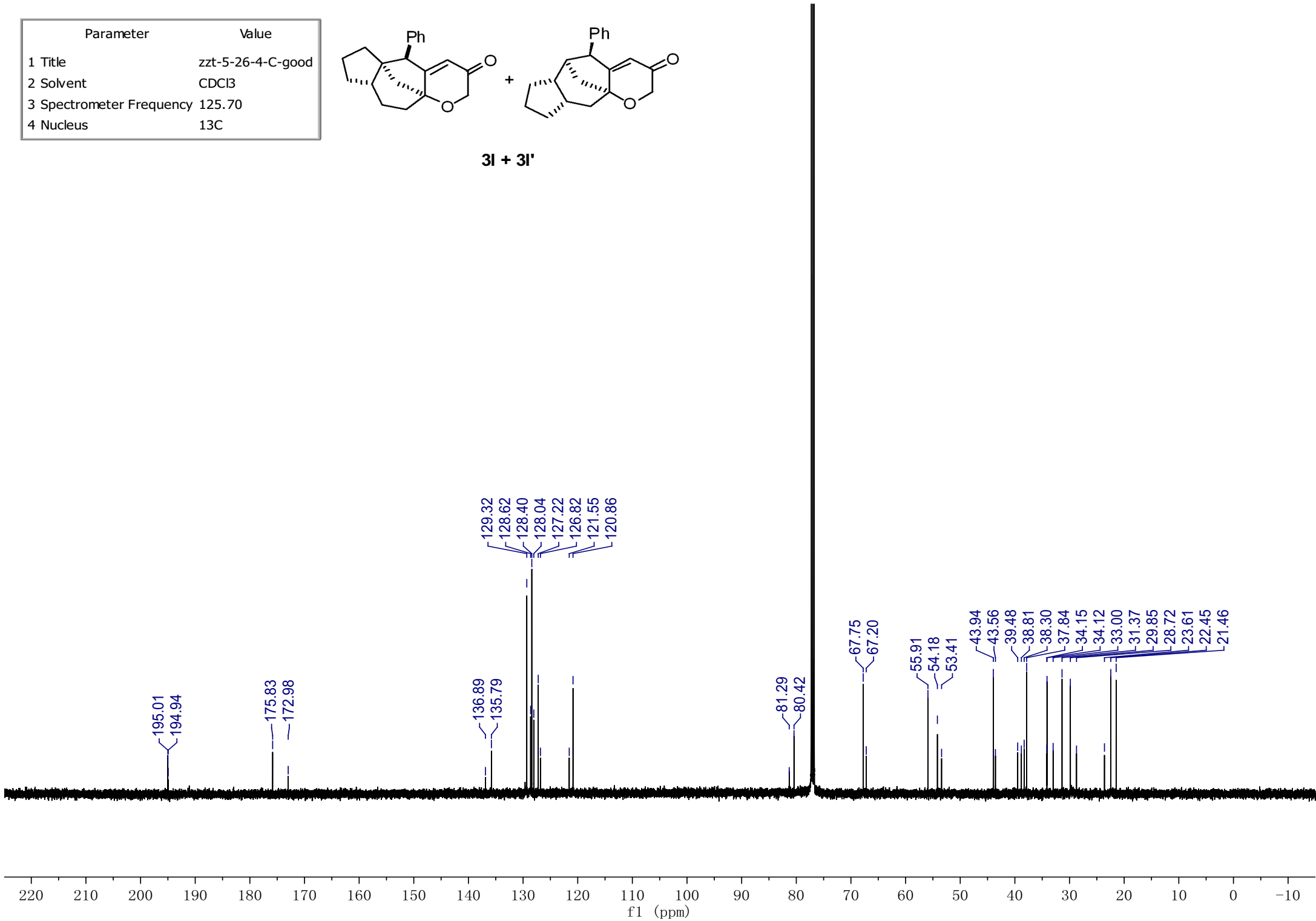
3I + 3I'



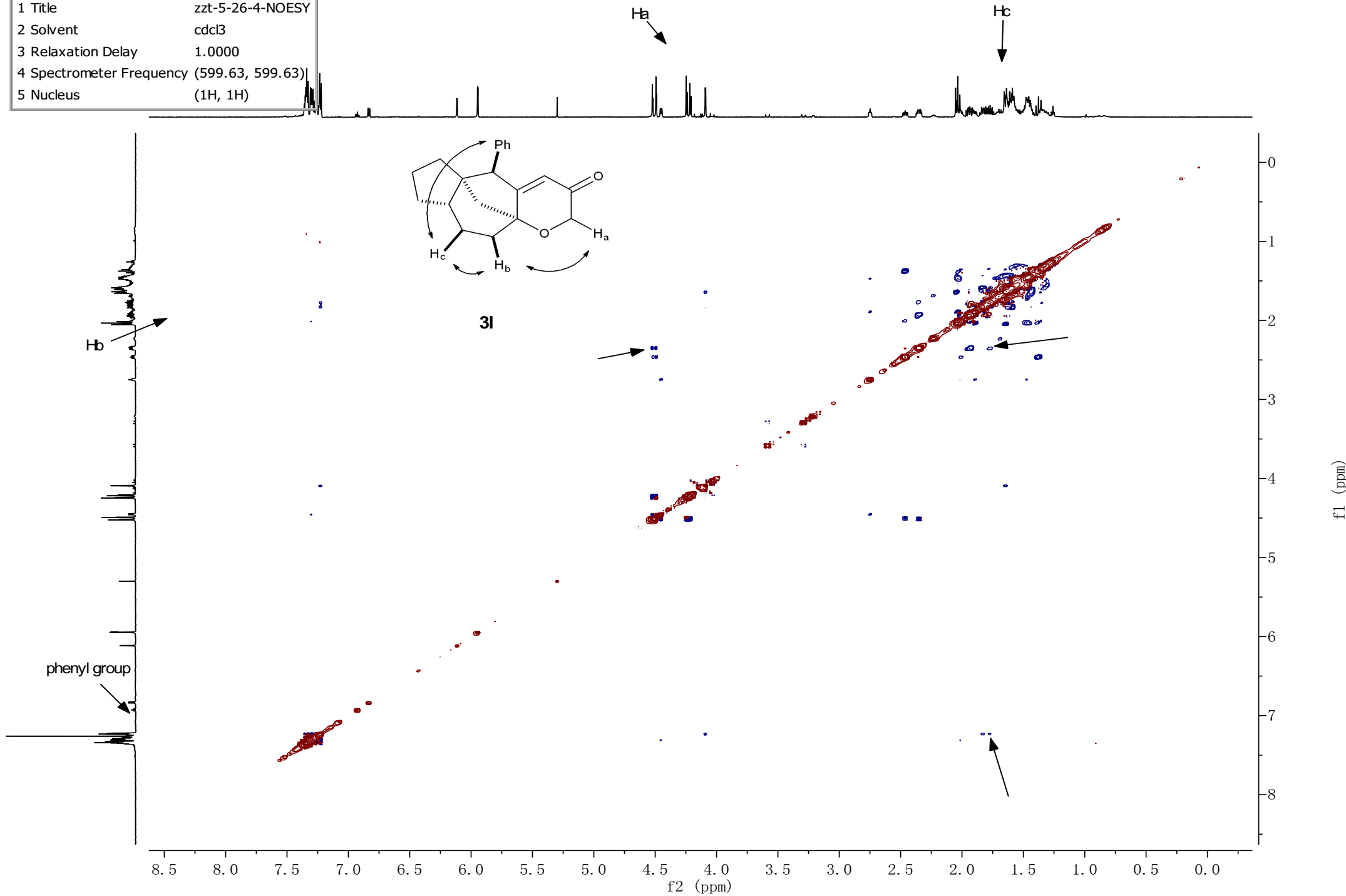
Parameter	Value
1 Title	zzt-5-26-4-C-good
2 Solvent	CDCl ₃
3 Spectrometer Frequency	125.70
4 Nucleus	13C



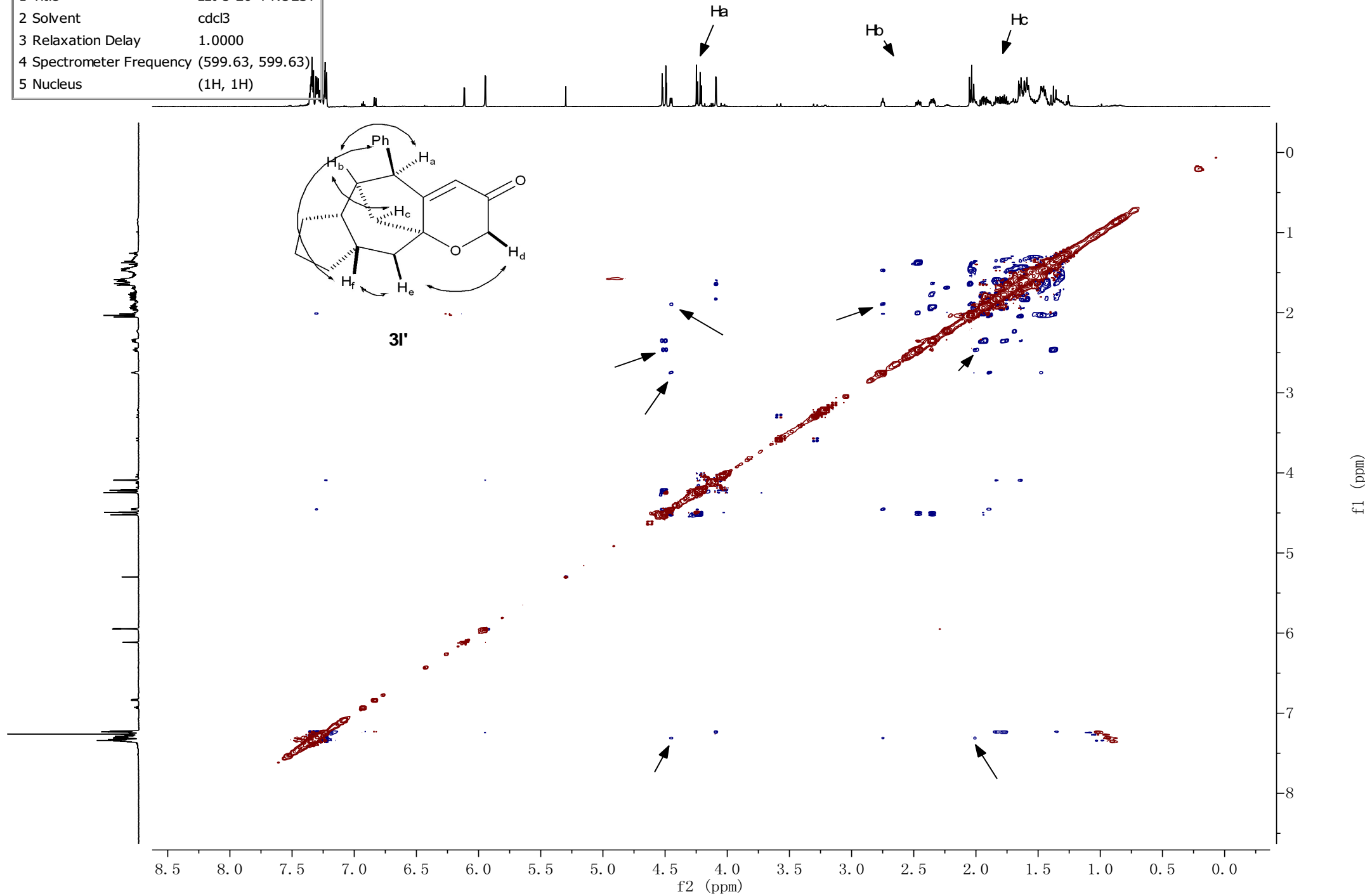
3I + 3I'



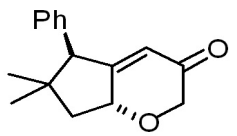
Parameter	Value
1 Title	zzt-5-26-4-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



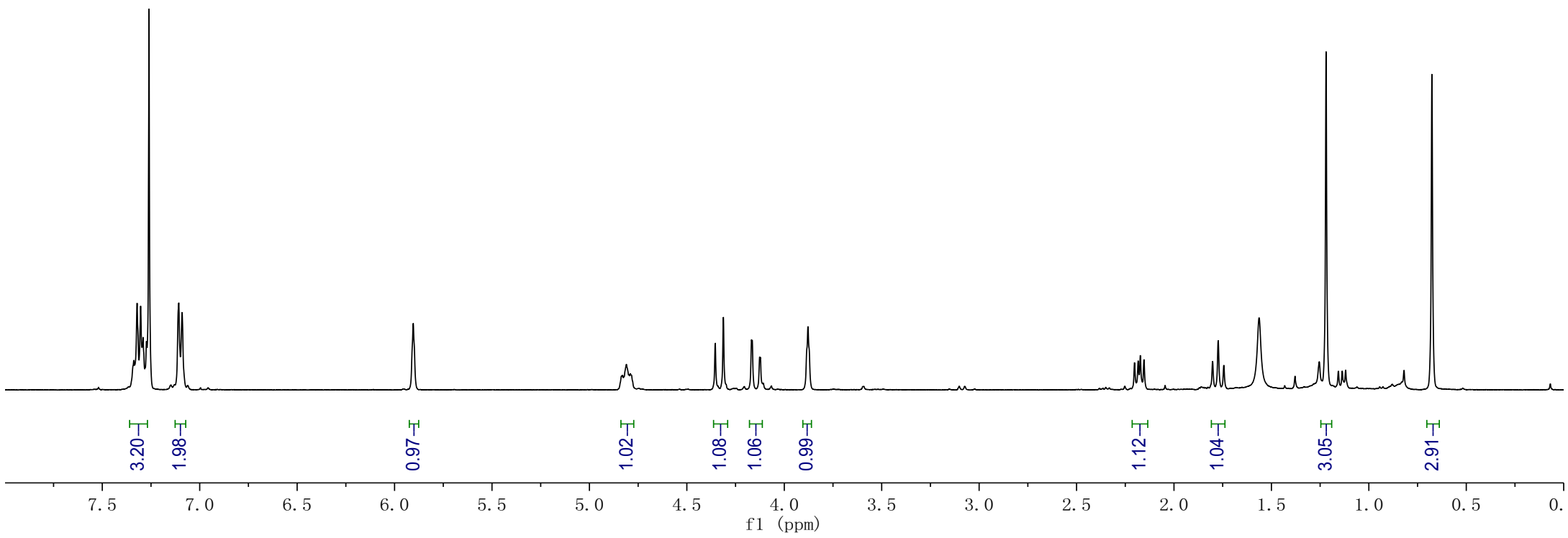
Parameter	Value
1 Title	zzt-5-26-4-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



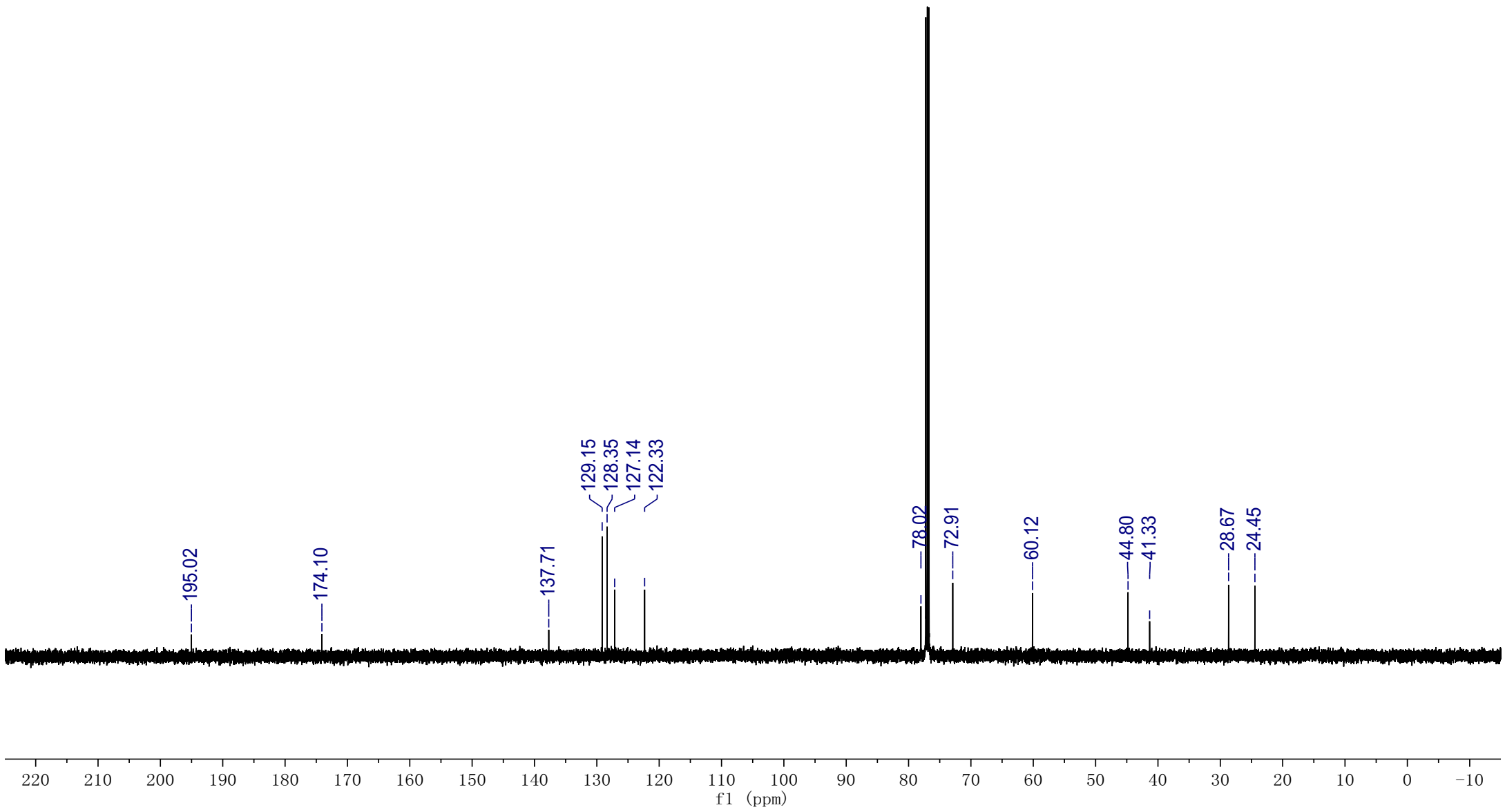
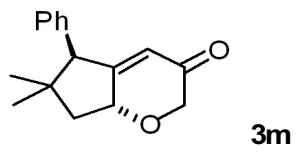
Parameter	Value
1 Title	zzt-4-151-2-F-H
2 Solvent	cdcl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	399.78



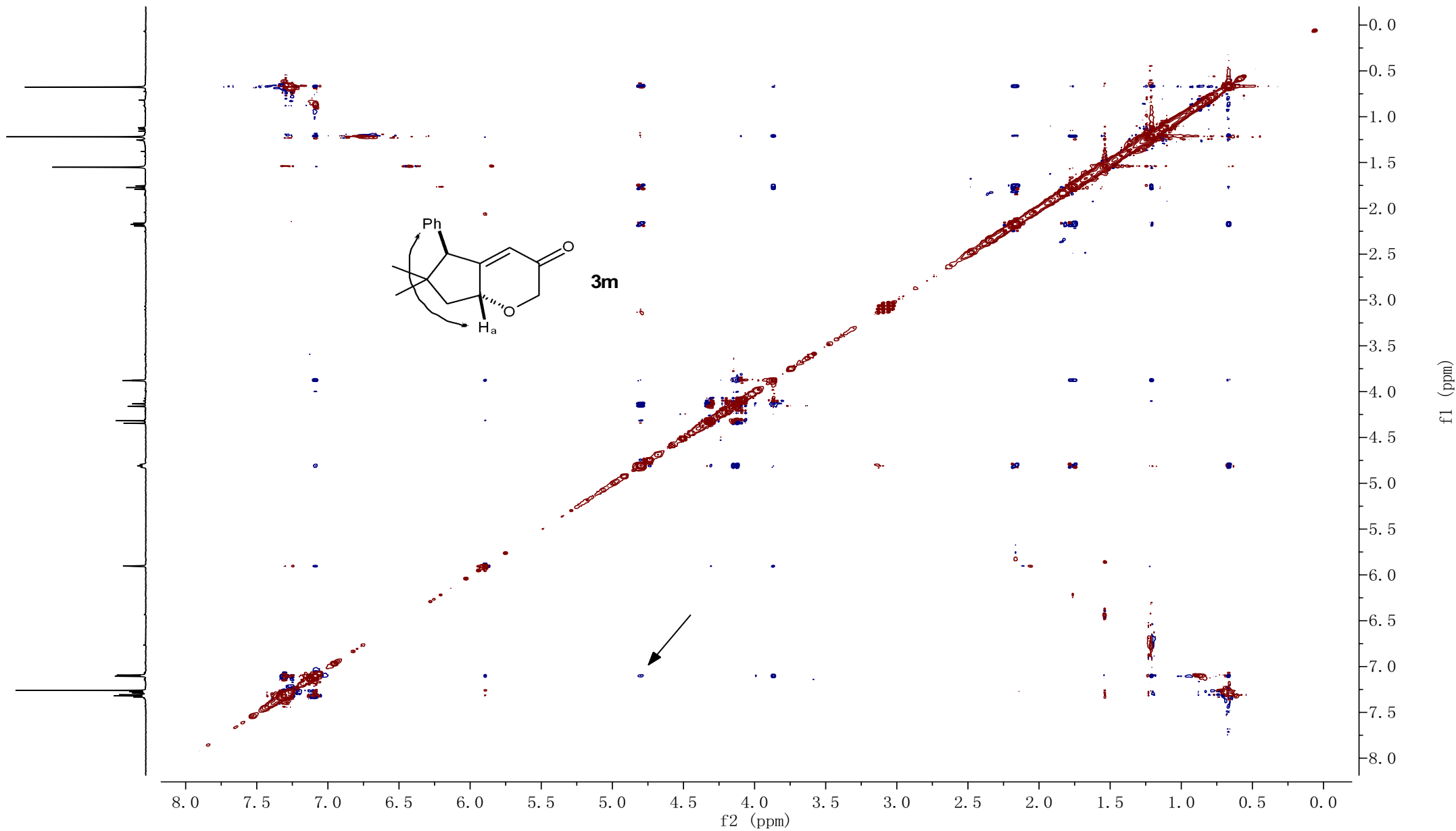
3m



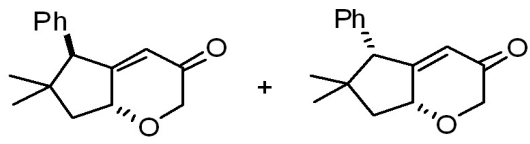
Parameter	Value
1 Title	zzt-4-151-2-F-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



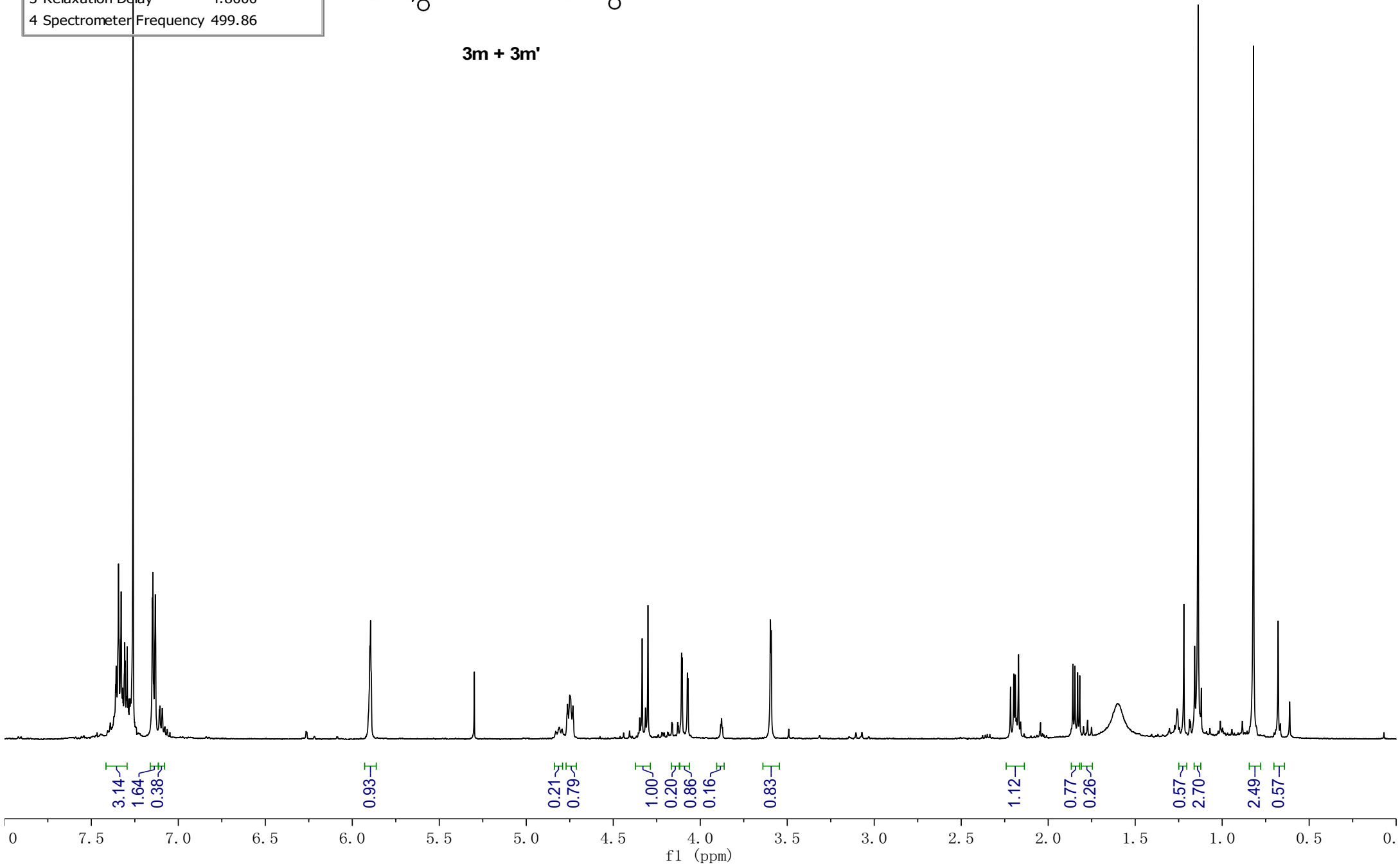
Parameter	Value
1 Title	zzt-4-151-2-F-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



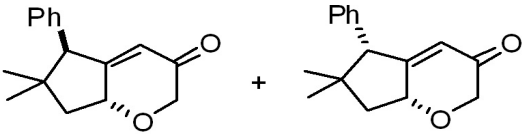
Parameter	Value
1 Title	zzt-4-151-2-B-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



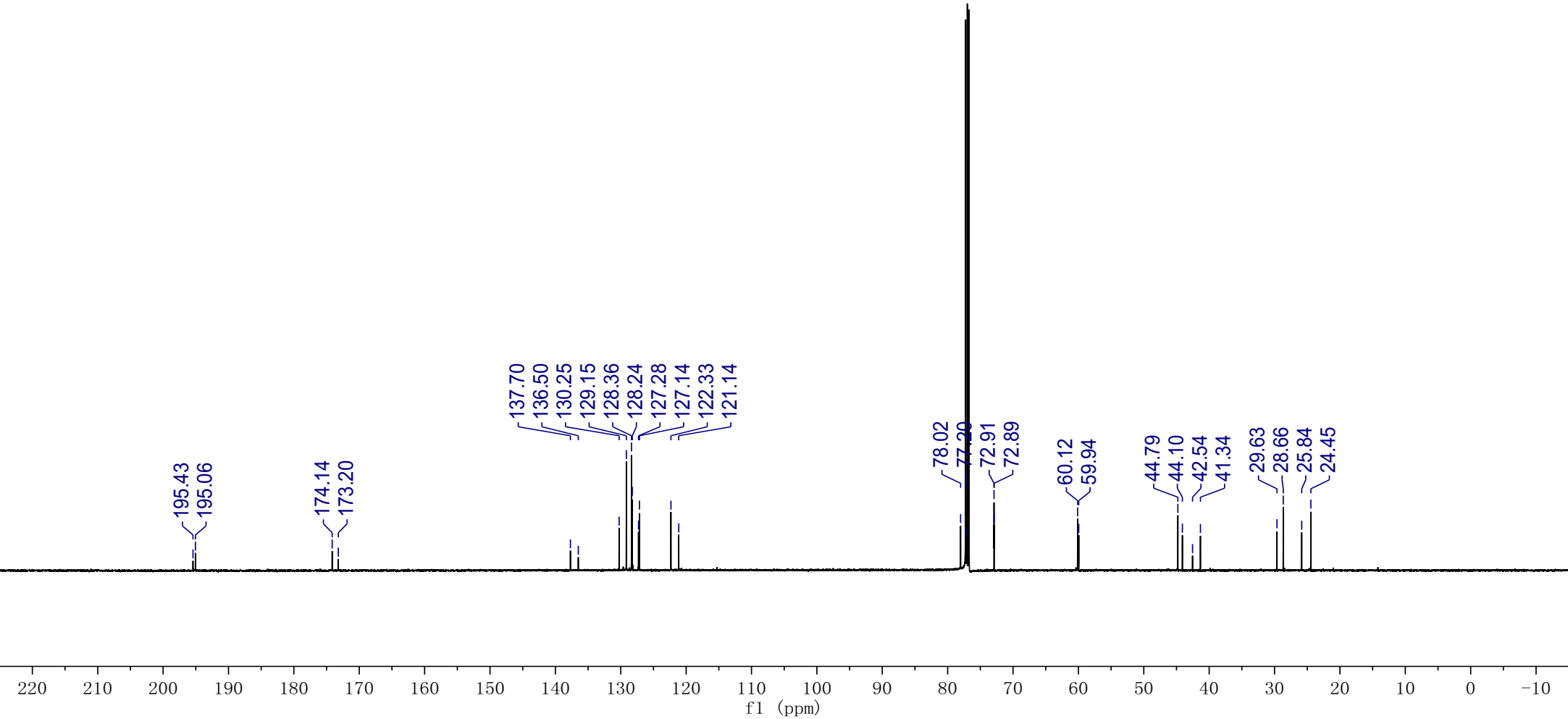
3m + 3m'



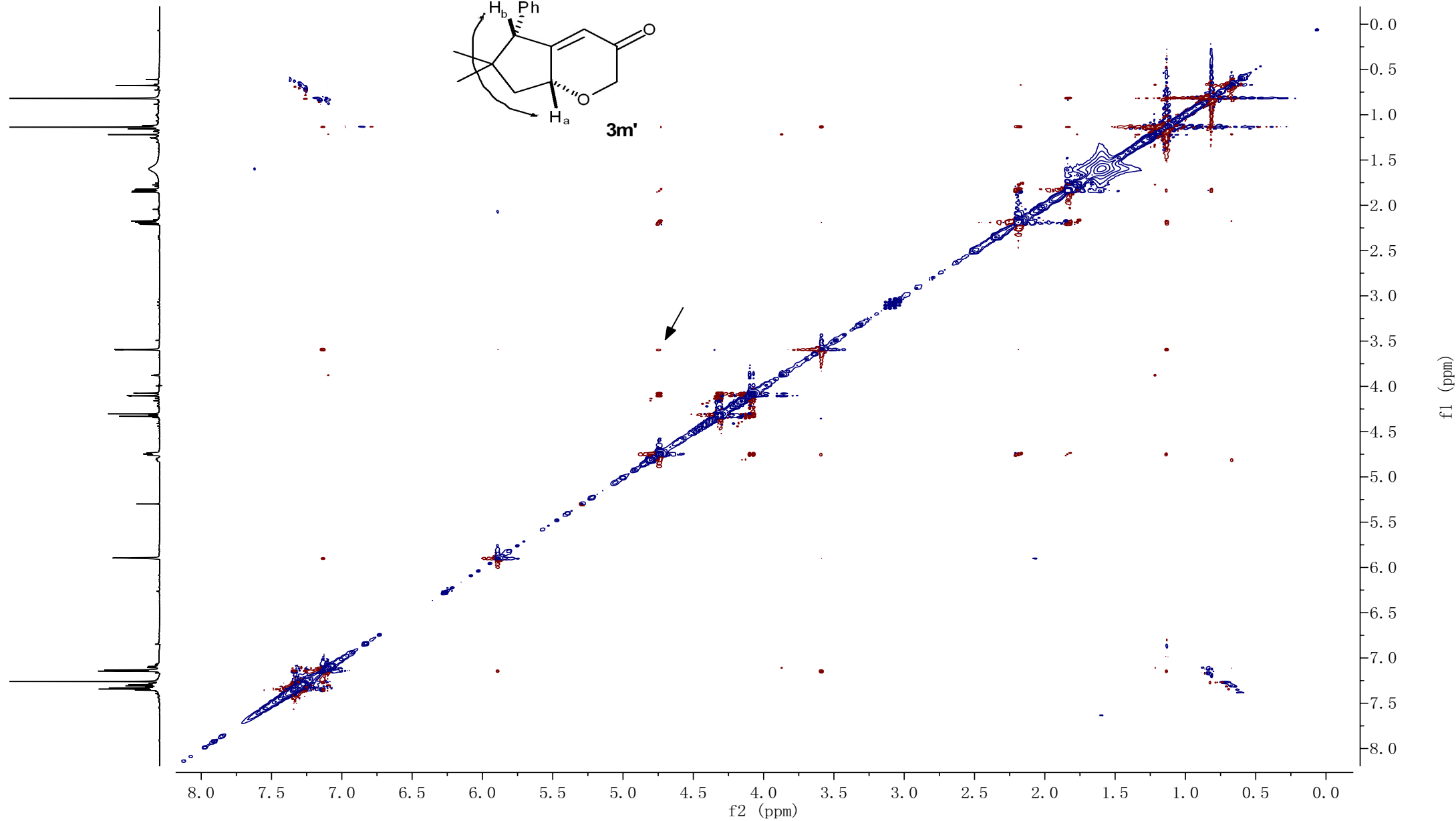
Parameter	Value
1 Title	zzt-4-151-2-B-C-with_diastereomer
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



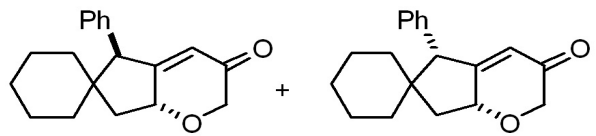
3m + 3m'



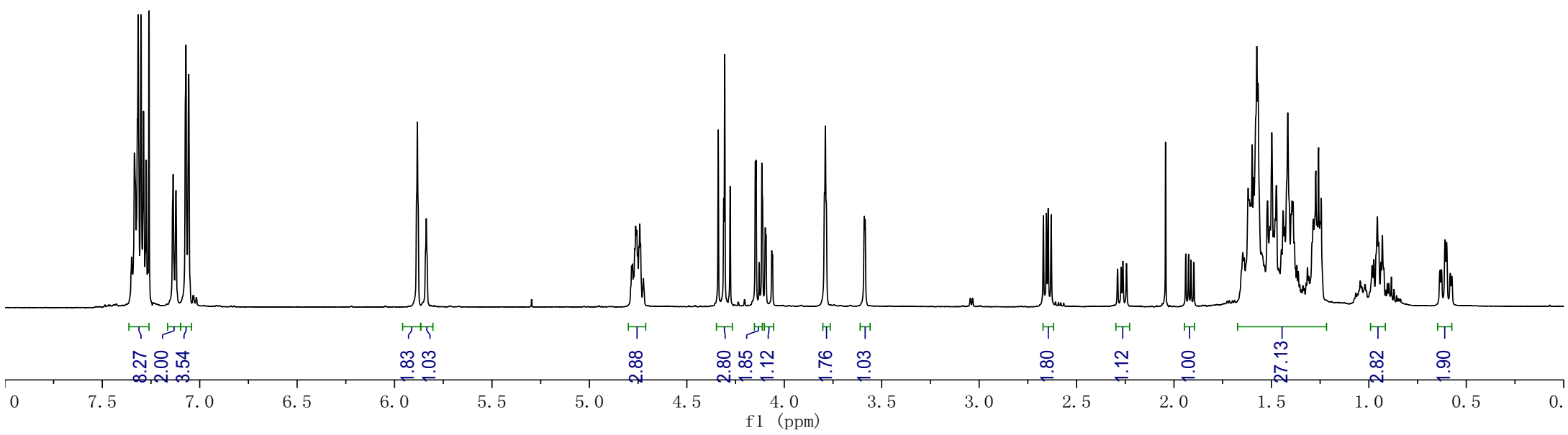
Parameter	Value
1 Title	zzt-4-151-2-B-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



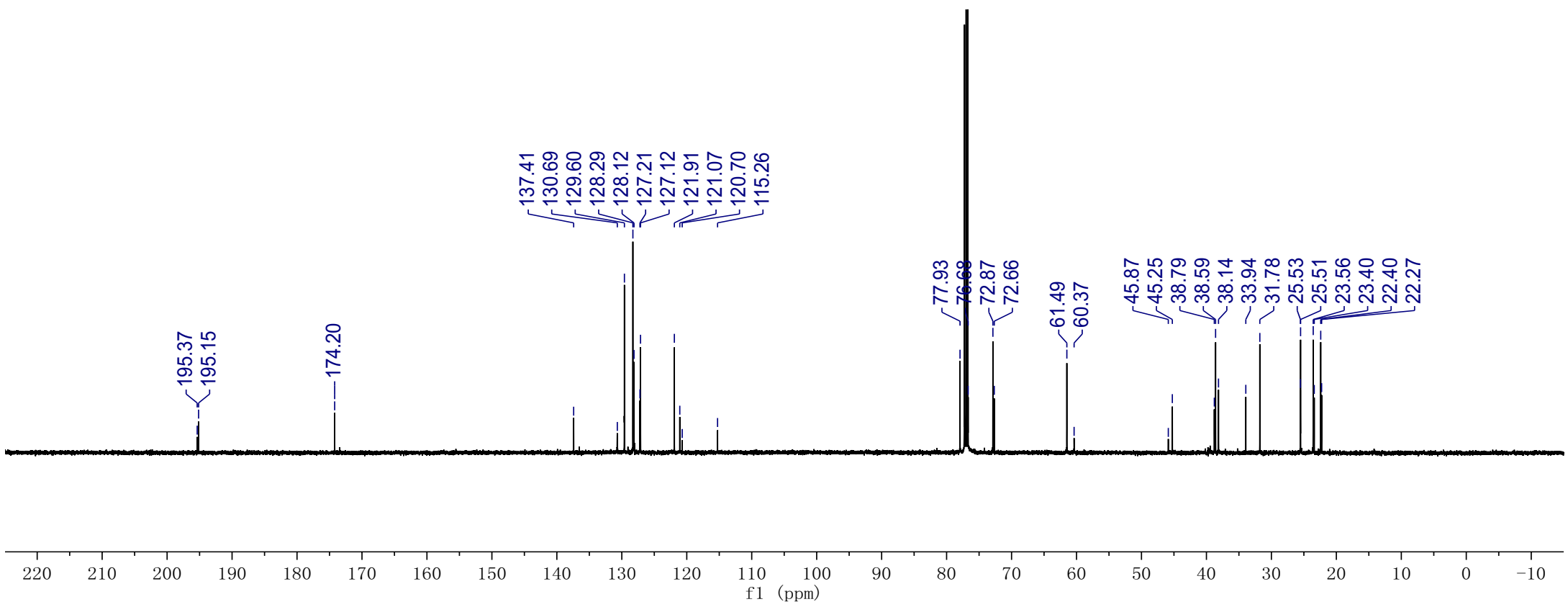
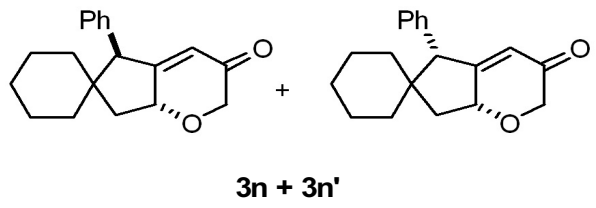
Parameter	Value
1 Title	zzt-5-9-2-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



3n + 3n'



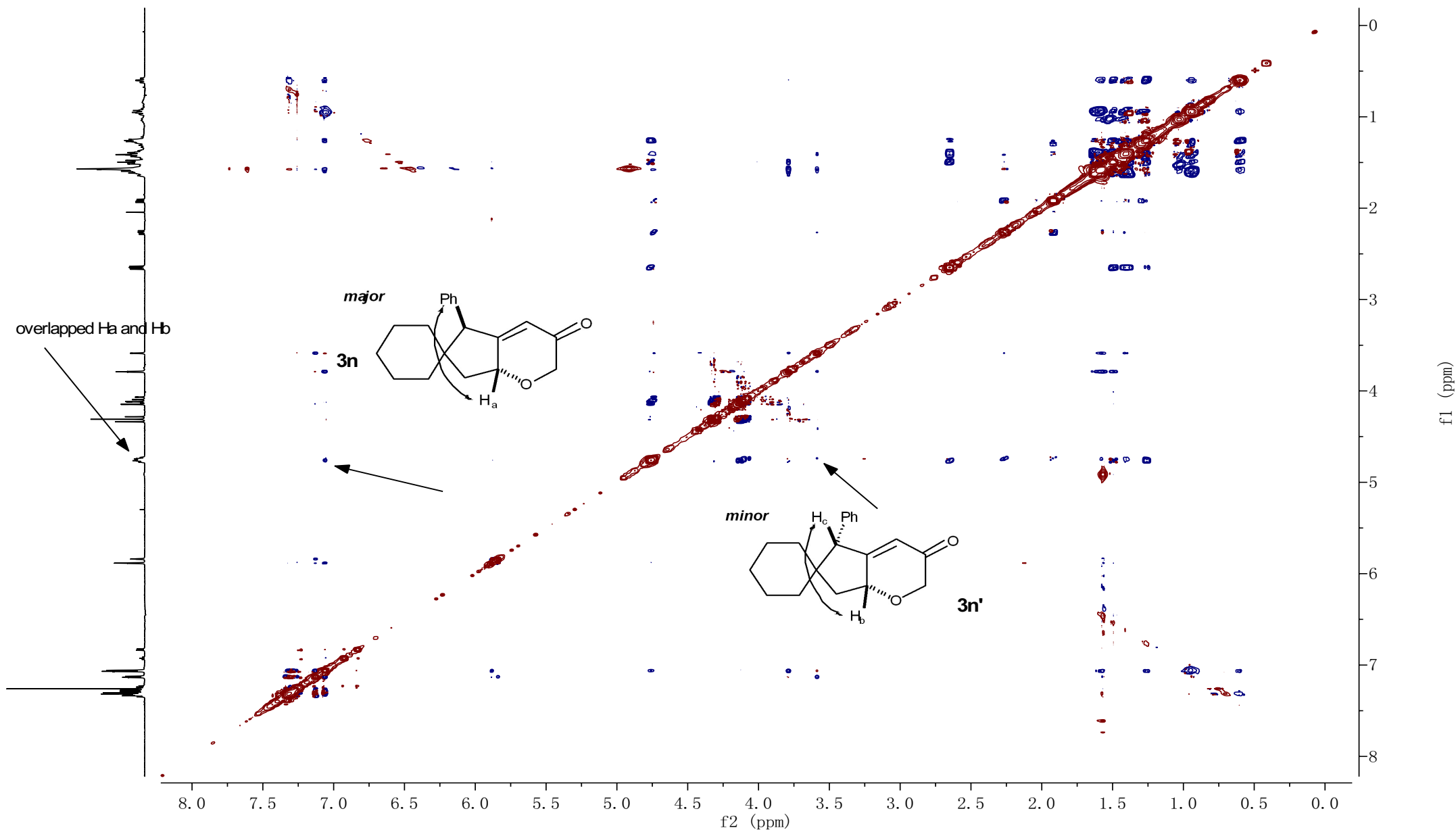
Parameter	Value
1 Title	zzt-5-9-2-C-final
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



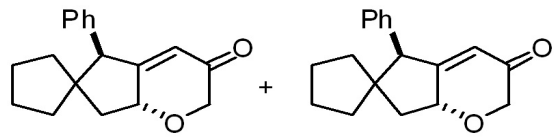
Parameter	Value
1 Title	zzt-5-9-2-NOESY_again
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)

phenyl group in major product

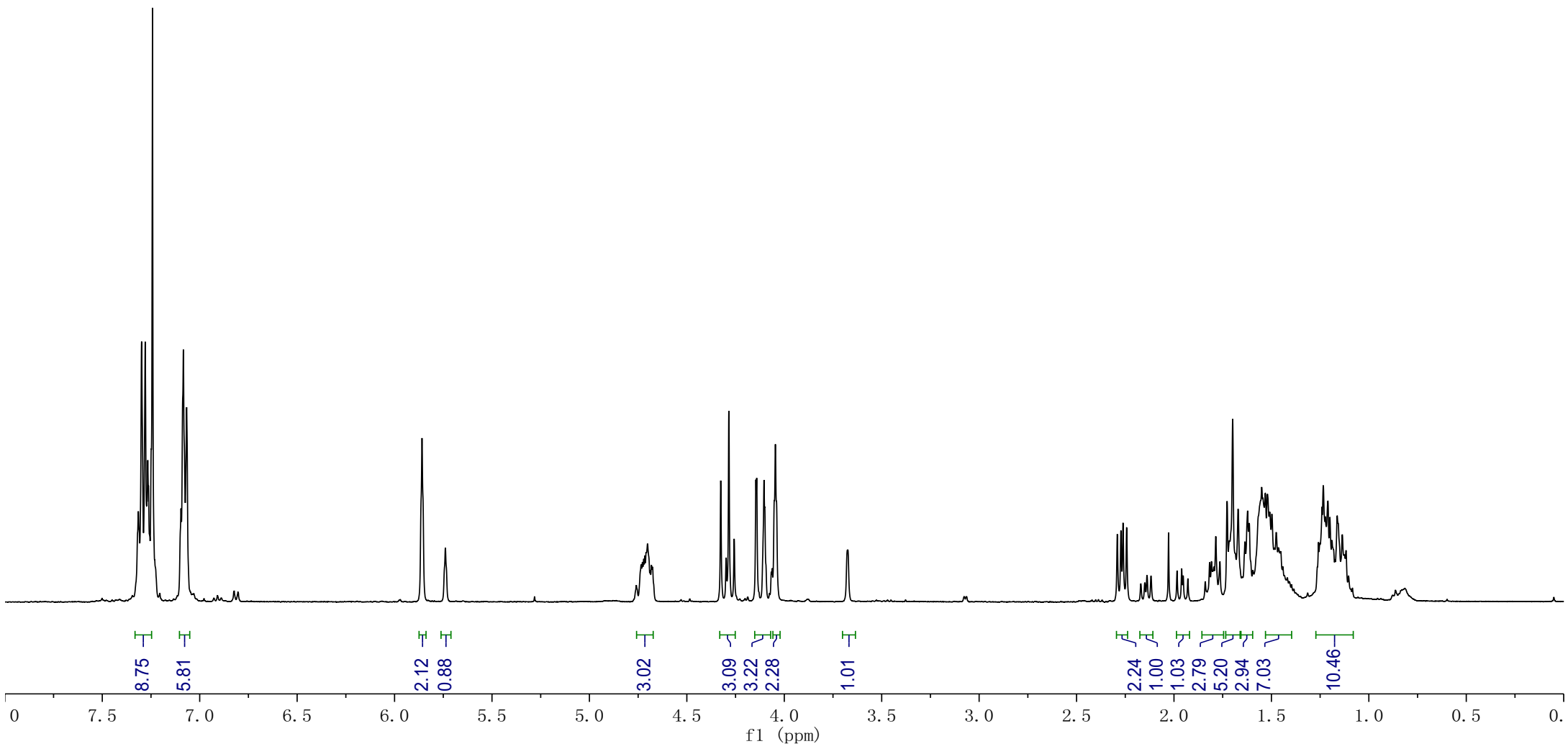
Hc in minor product



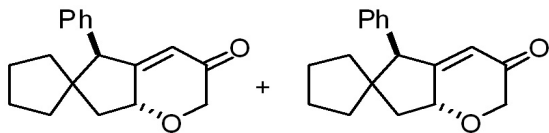
Parameter	Value
1 Title	zzt-5-9-3-H_good
2 Solvent	cdcl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	399.78



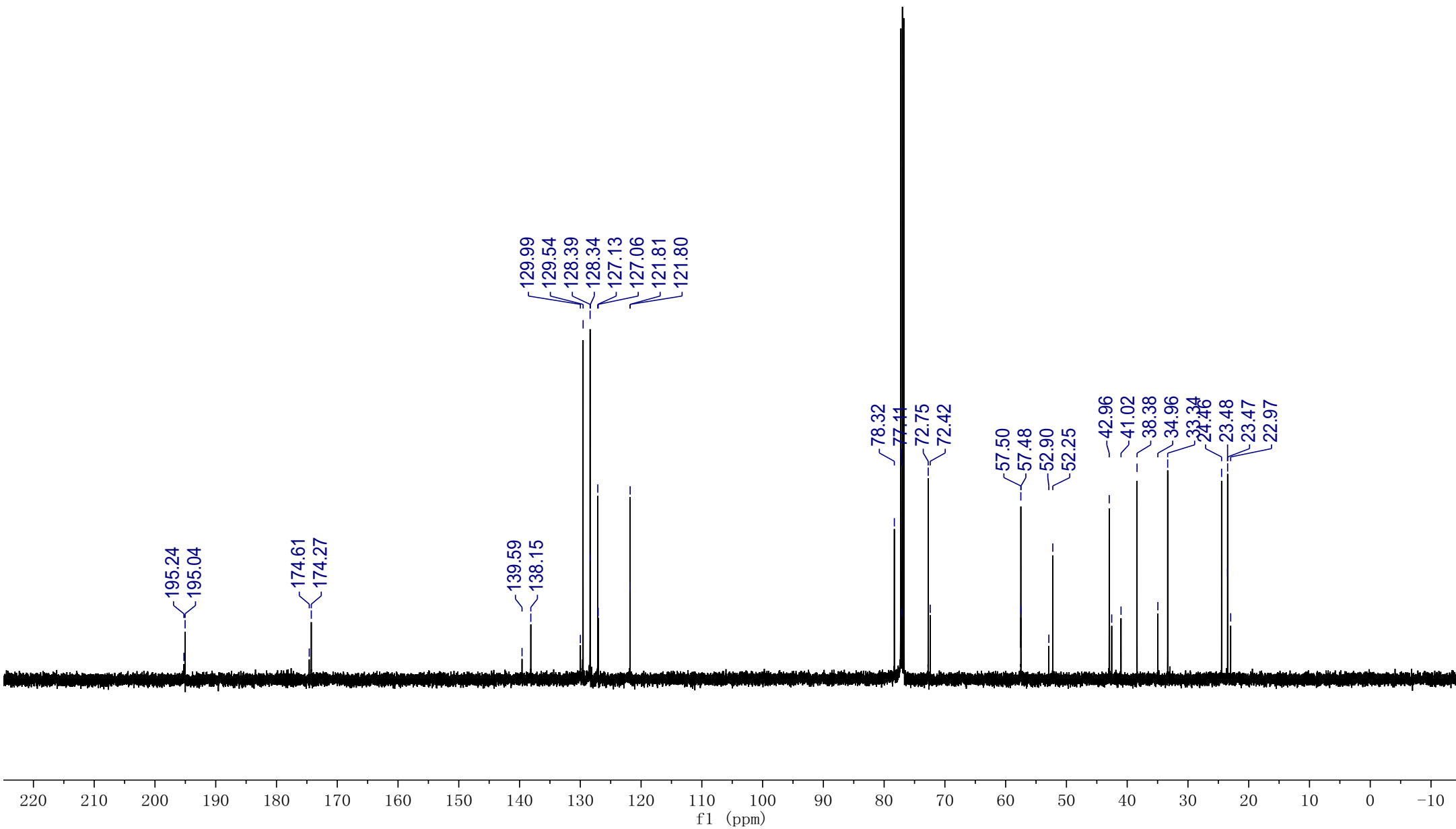
3o + 3o'



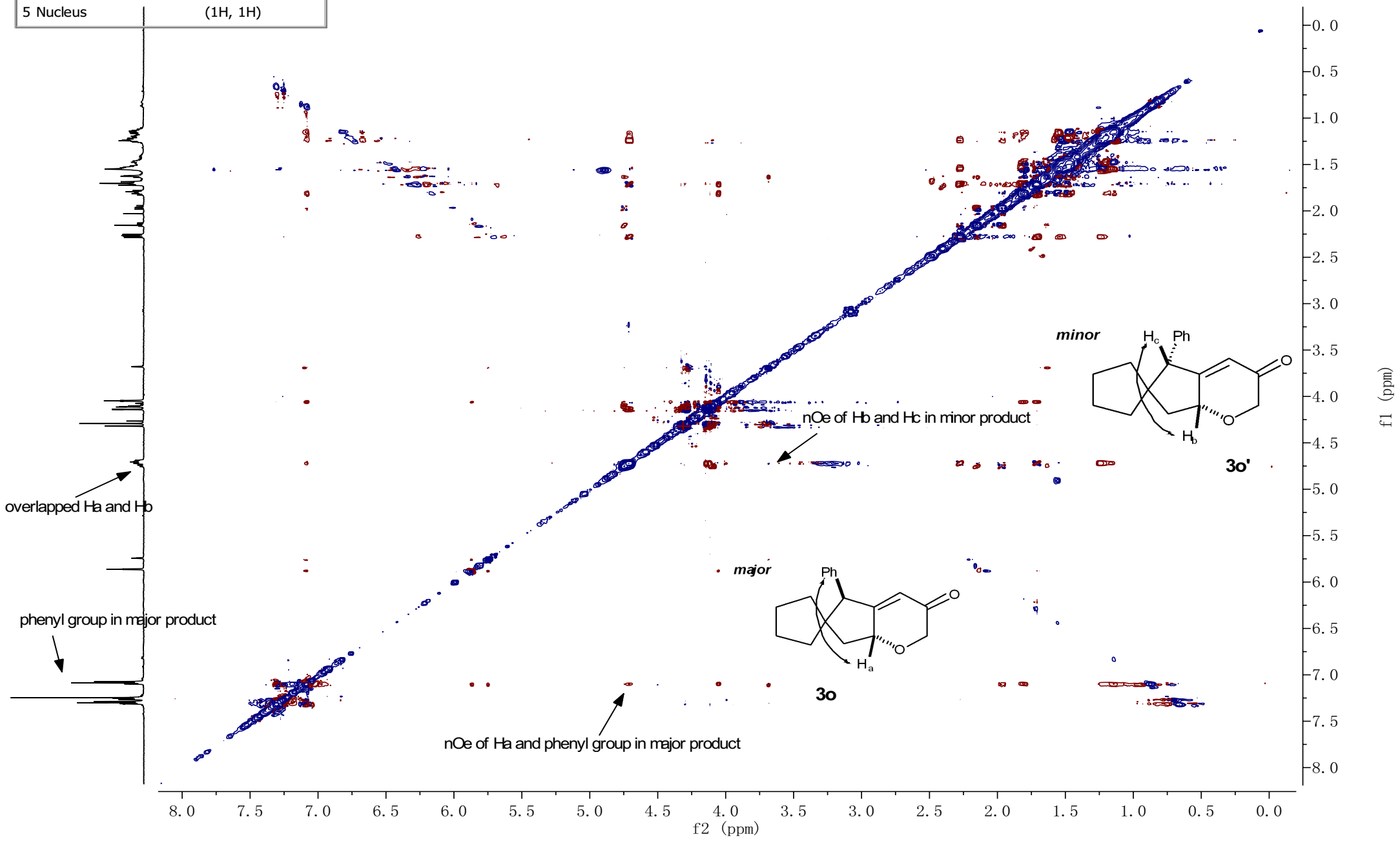
Parameter	Value
1 Title	zzt-5-9-3-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



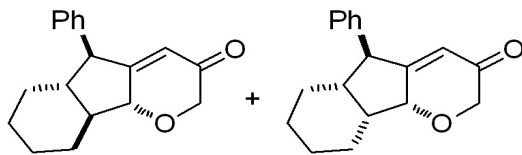
3o + 3o'



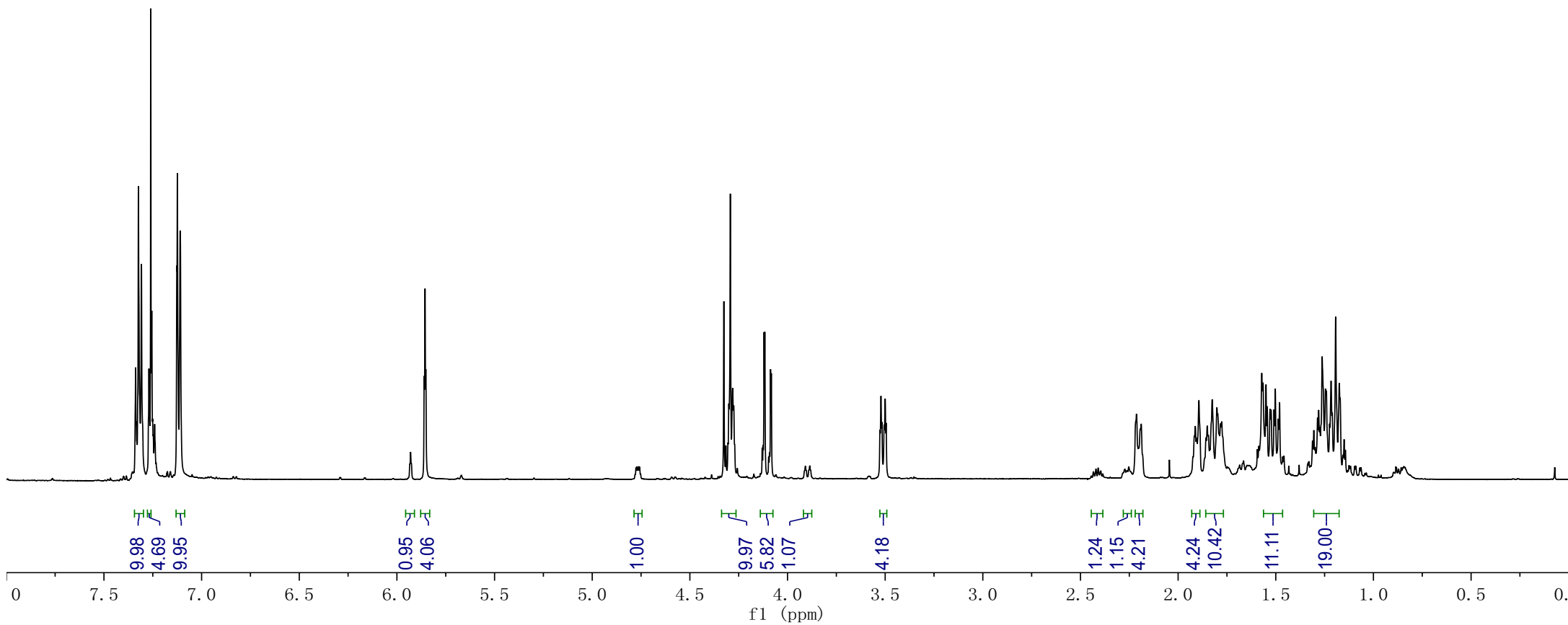
Parameter	Value
1 Title	zzt-5-9-3-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	(599.63, 599.63)
5 Nucleus	(1H, 1H)



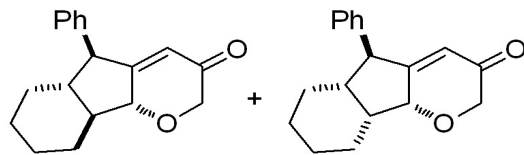
Parameter	Value
1 Title	zzt-5-18-4-H
2 Solvent	CDCl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	499.86



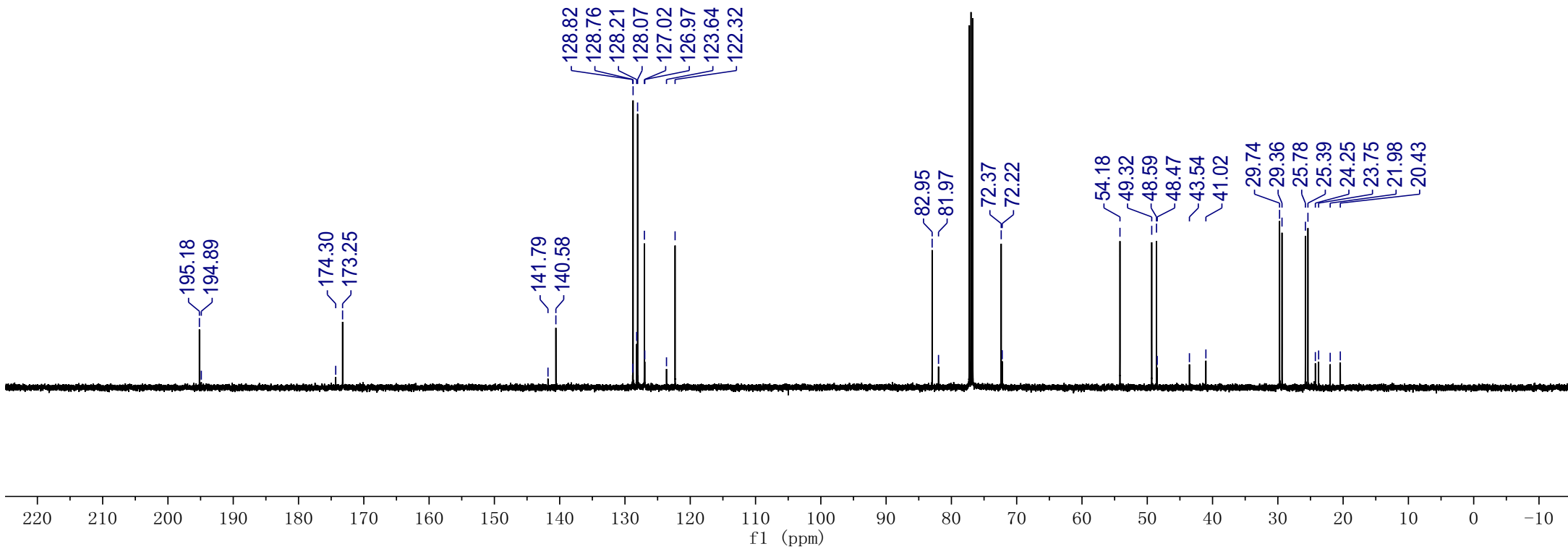
3p + 3p'



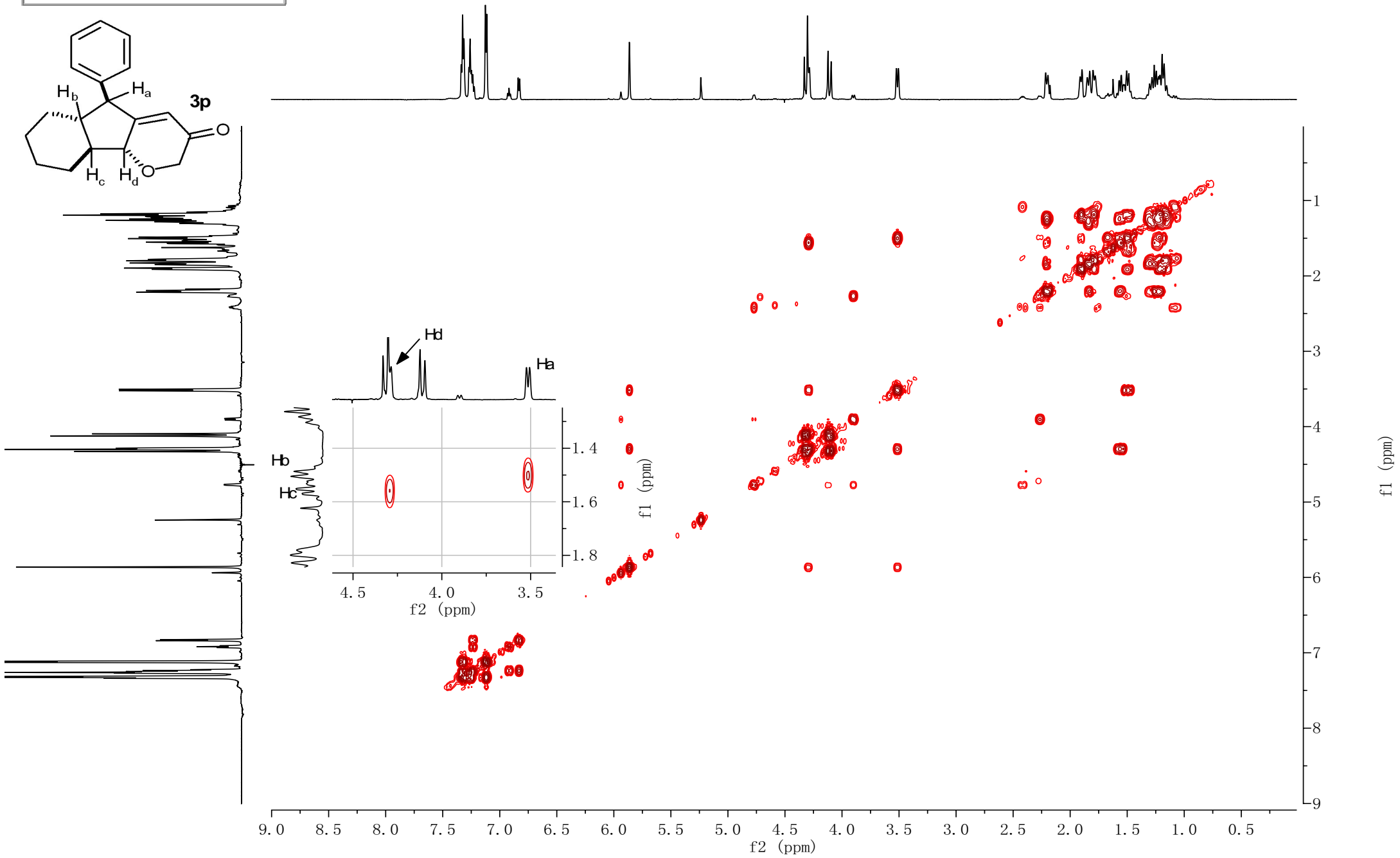
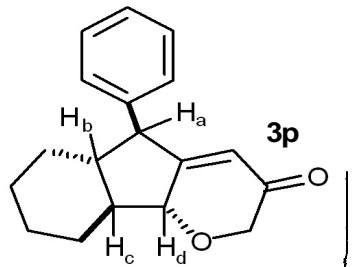
Parameter	Value
1 Title	zzt-5-18-4-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



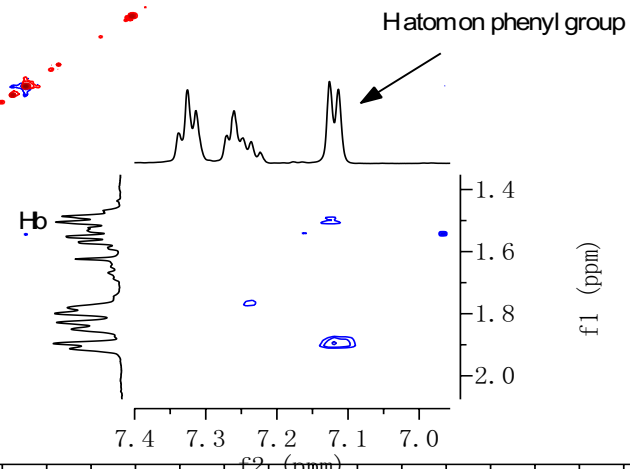
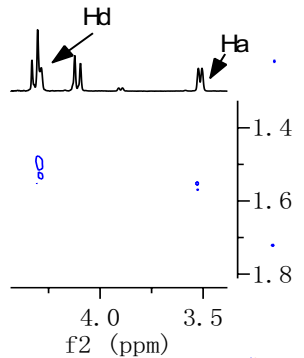
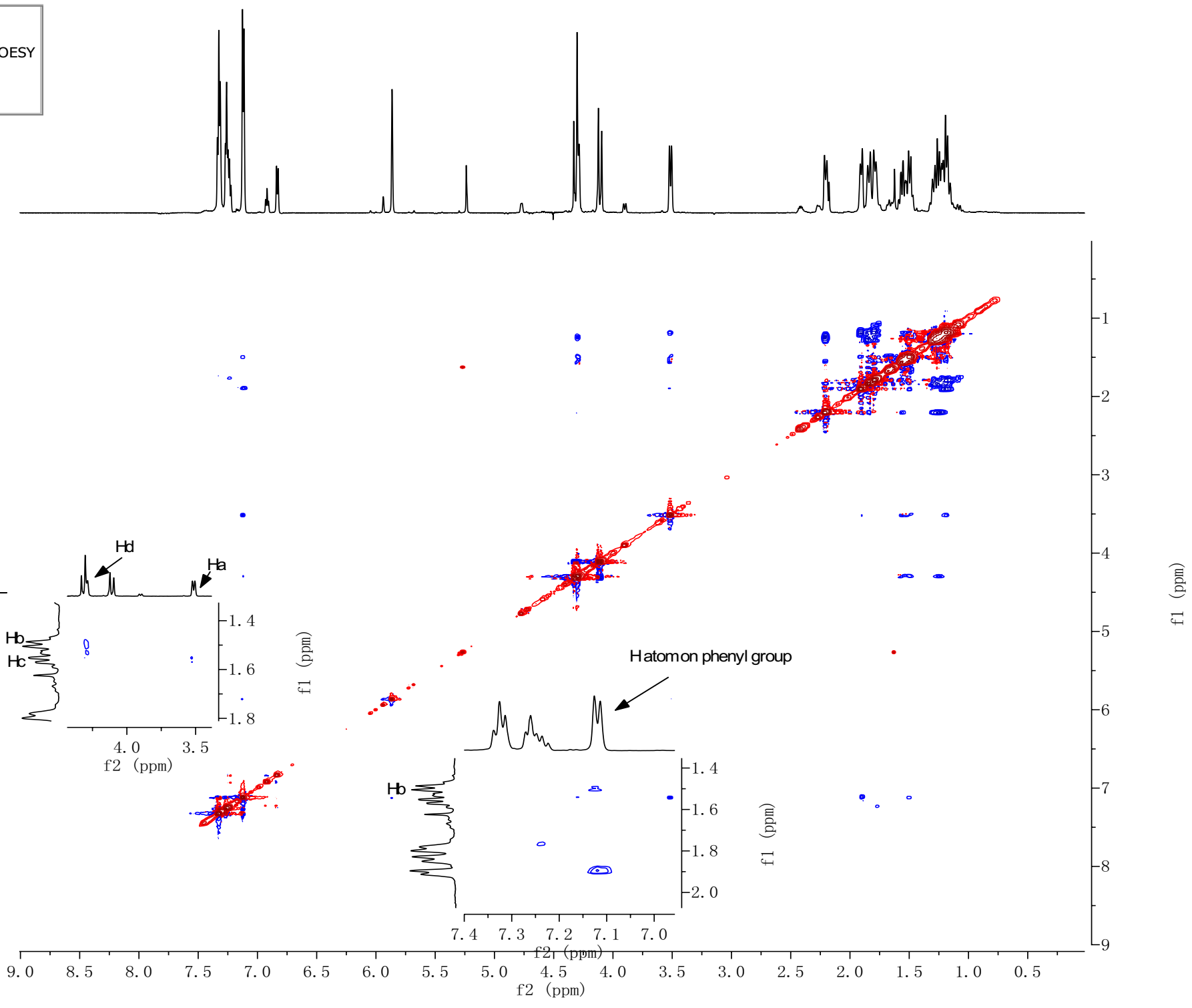
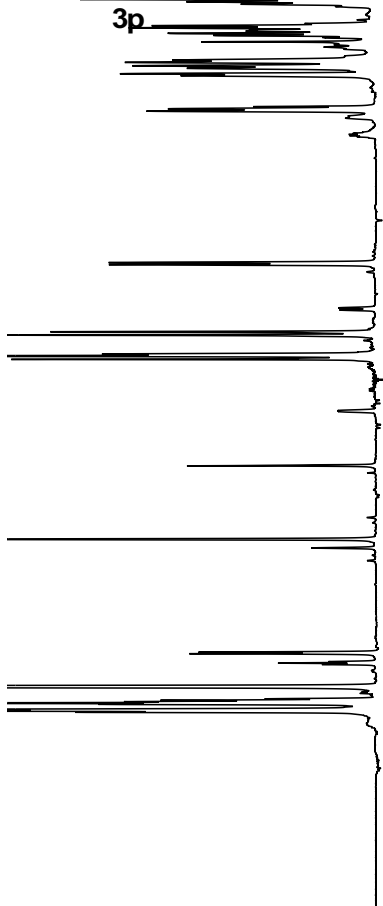
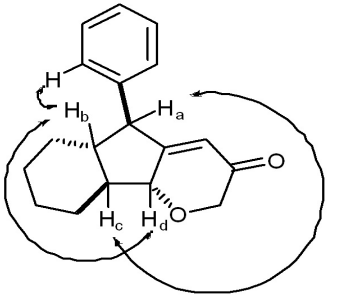
3p + 3p'



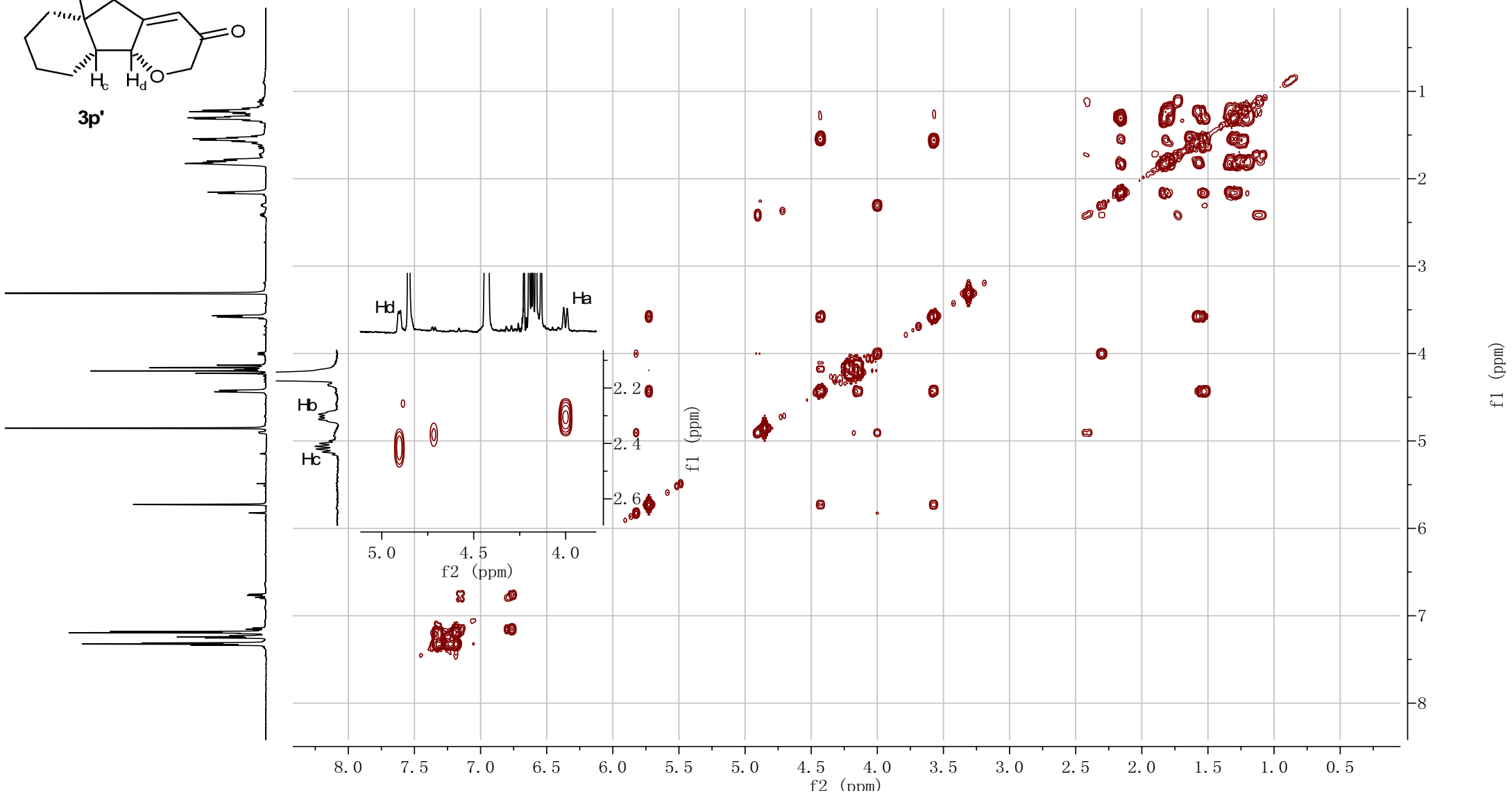
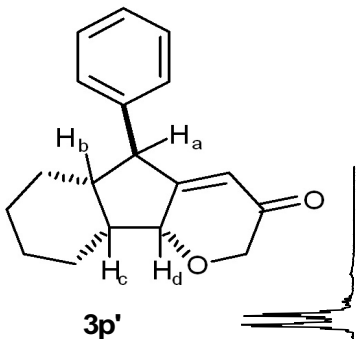
Parameter	Value
1 Title	zzt-4-170-2-COSY
2 Solvent	"cdcl3"
3 Relaxation Delay	1.0000



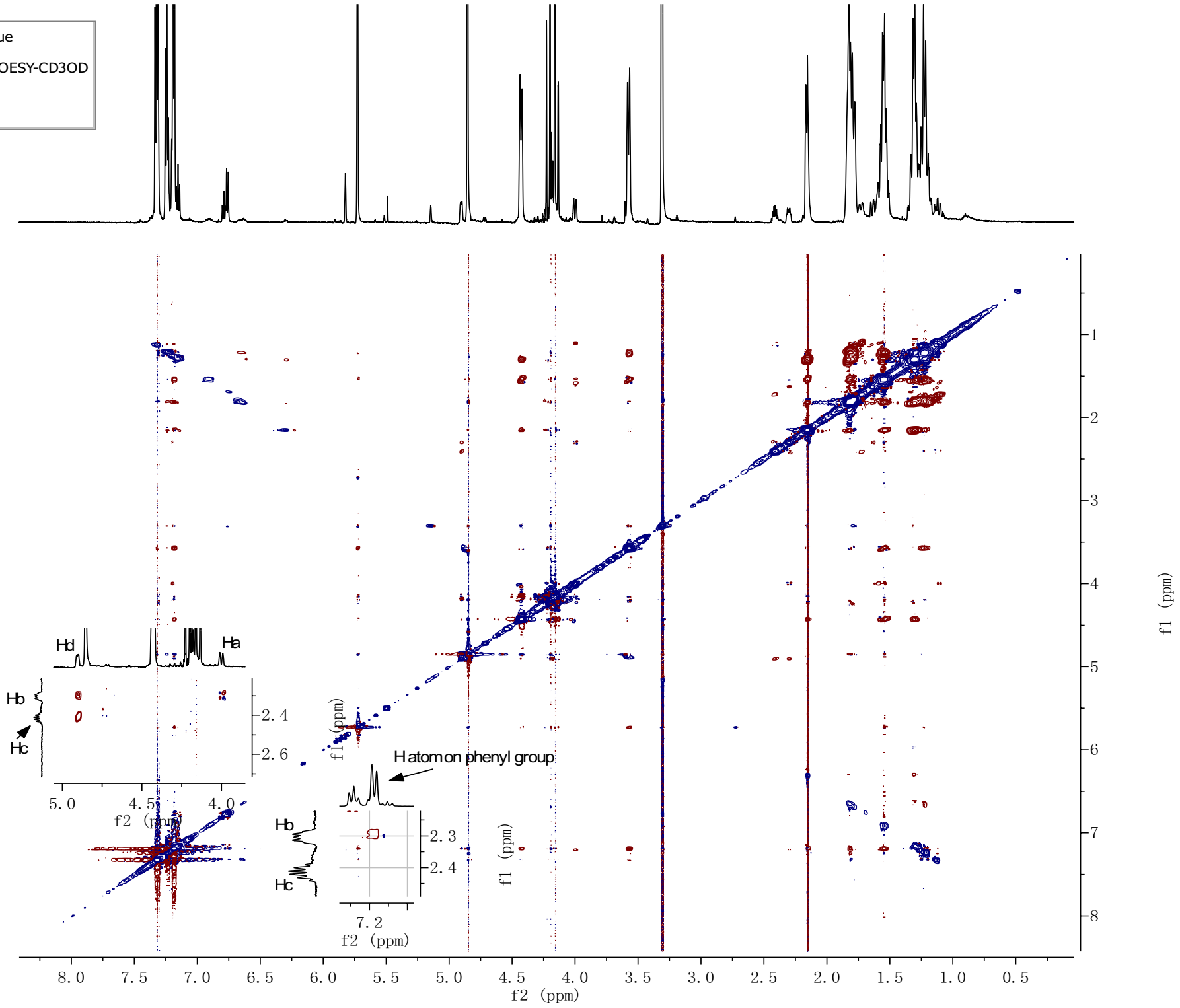
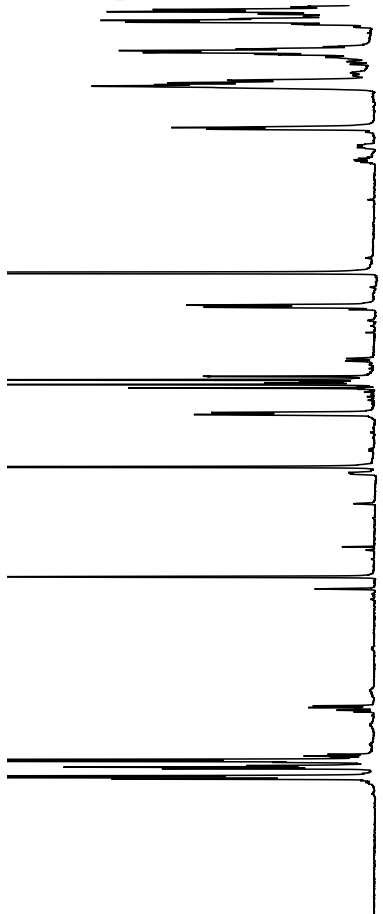
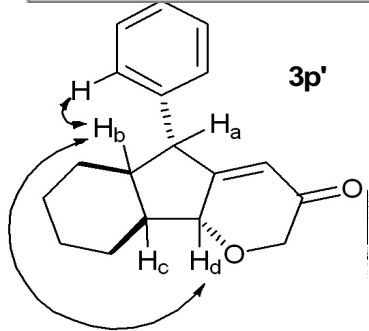
Parameter	Value
1 Title	zzt-4-170-2-NOESY
2 Solvent	"cdcl3"
3 Relaxation Delay	1.0000



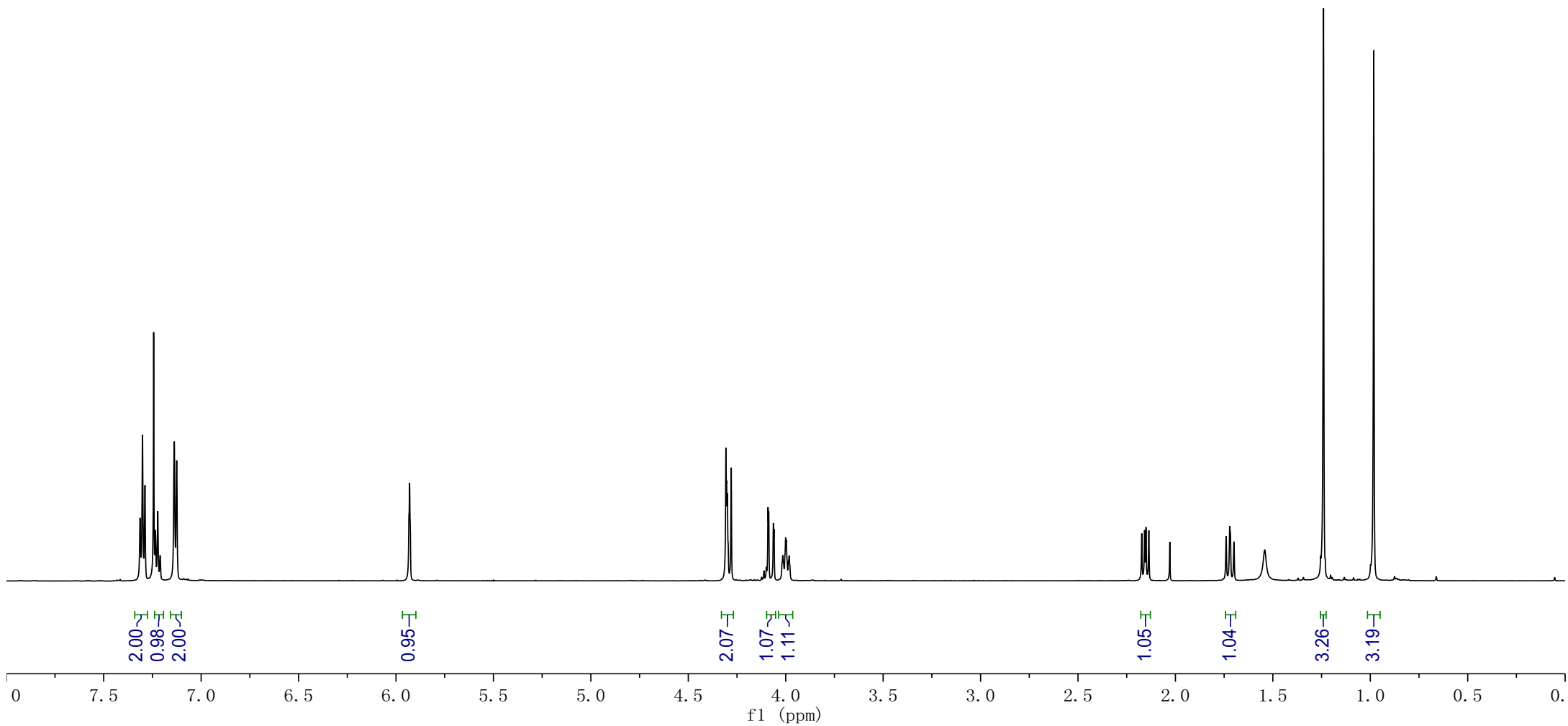
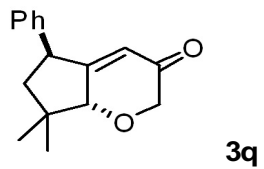
Parameter	Value
1 Title	zzt-4-170-2-COSY-CD3OD
2 Solvent	"cd3od"
3 Relaxation Delay	1.0000



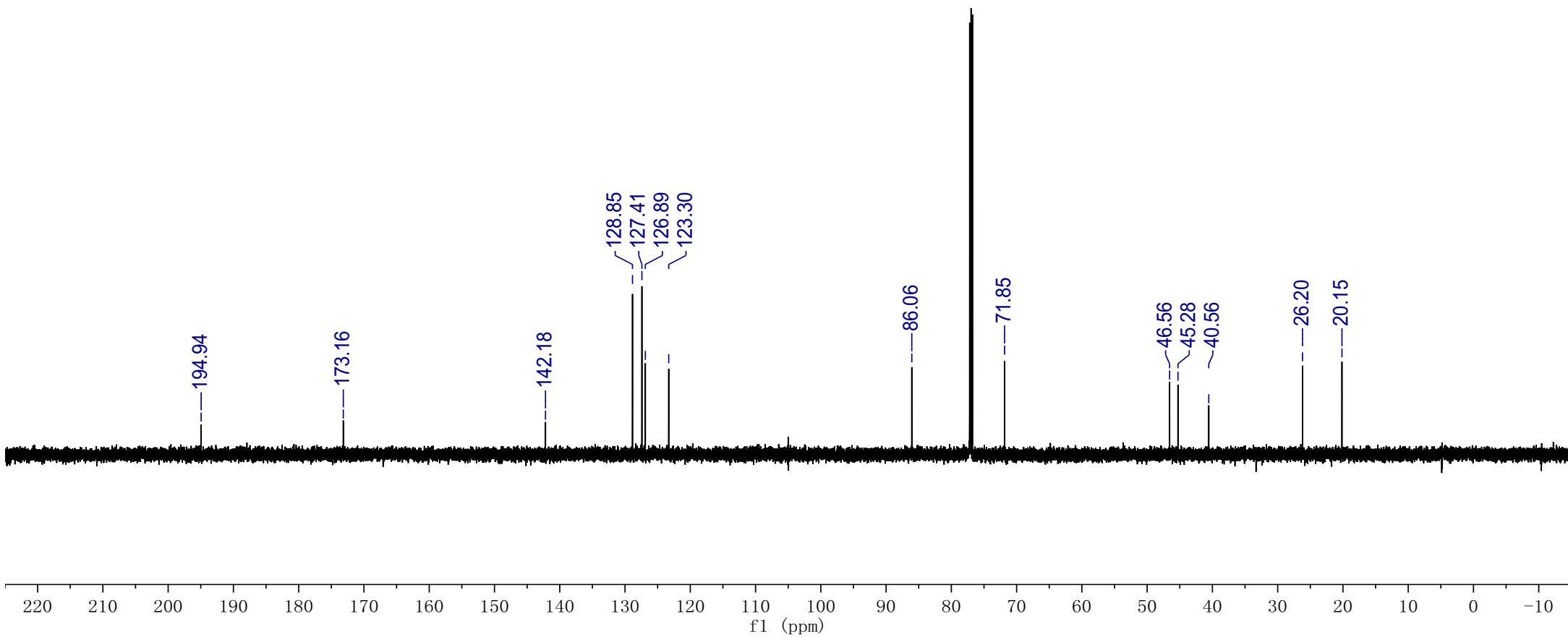
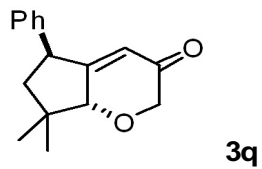
Parameter	Value
1 Title	zzt-4-170-2-NOESY-CD3OD
2 Solvent	"cd3od"
3 Relaxation Delay	1.0000



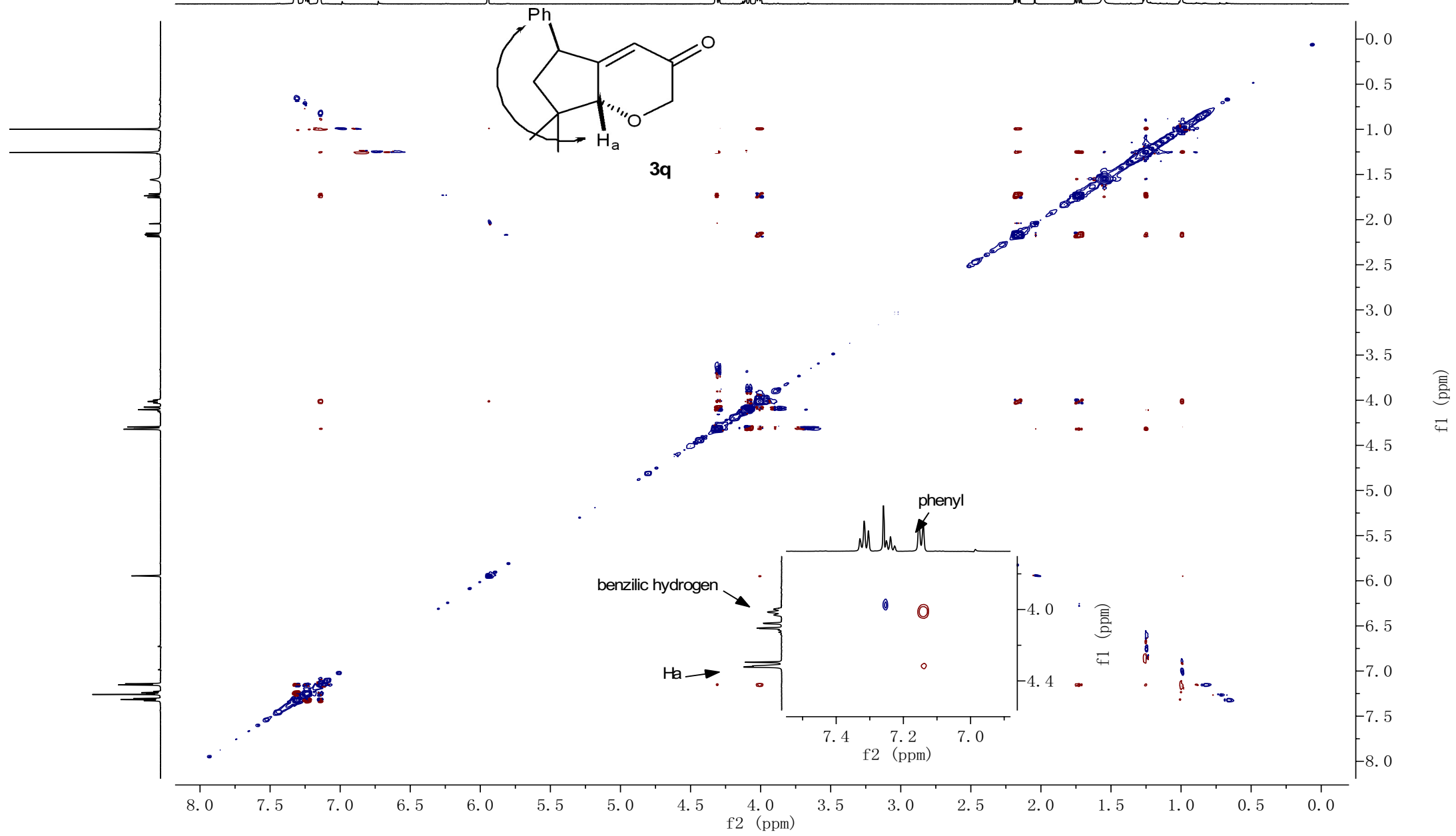
Parameter	Value
1 Title	zzt-4-188-1-H
2 Solvent	cdcl3
3 Relaxation Delay	4.8000
4 Spectrometer Frequency	599.64



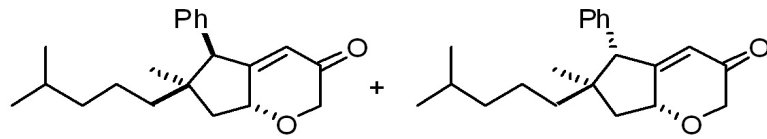
Parameter	Value
1 Title	zzt-4-188-1-C
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	150.79



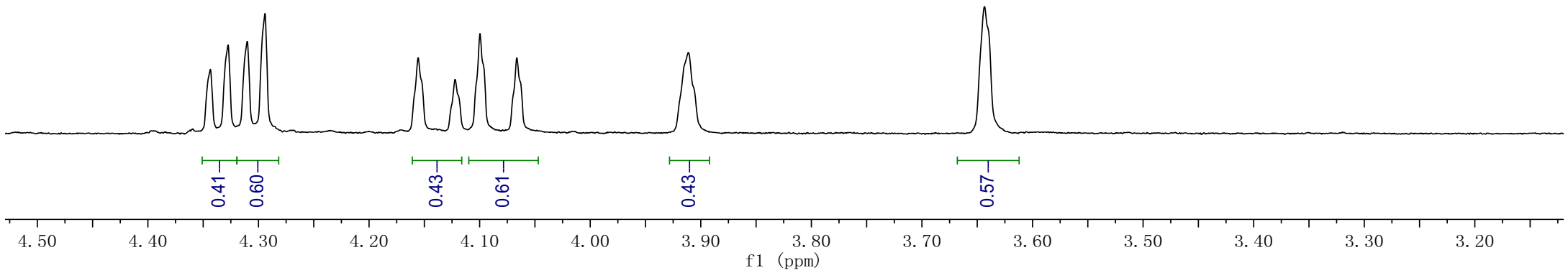
Parameter	Value
1 Title	zzt-4-188-1-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



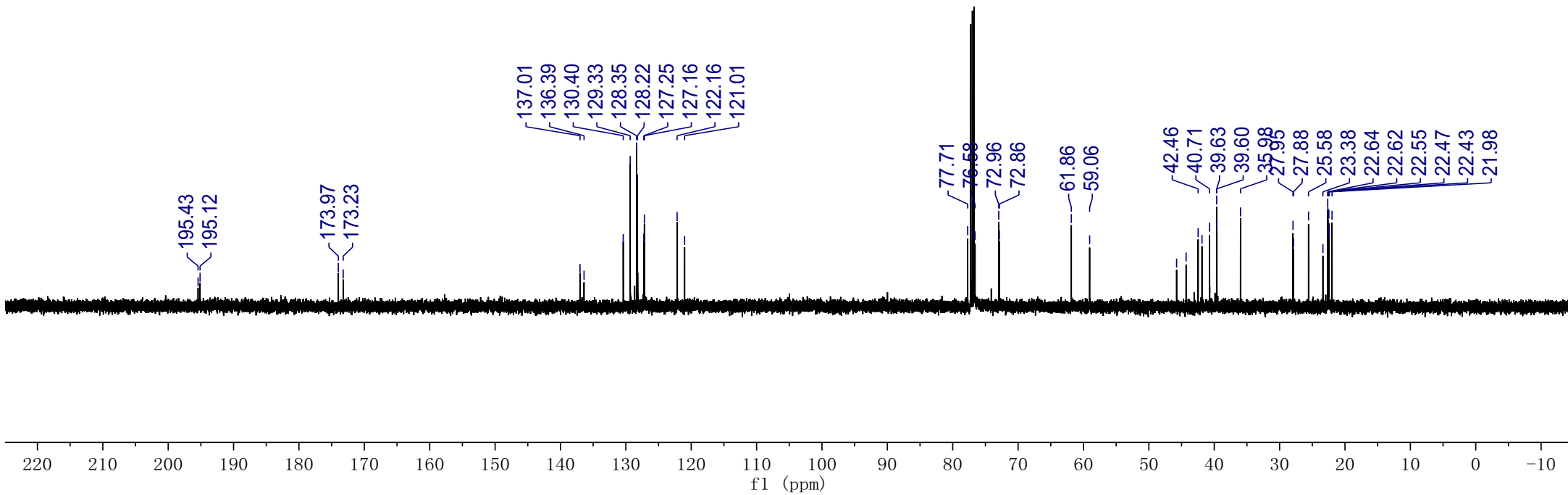
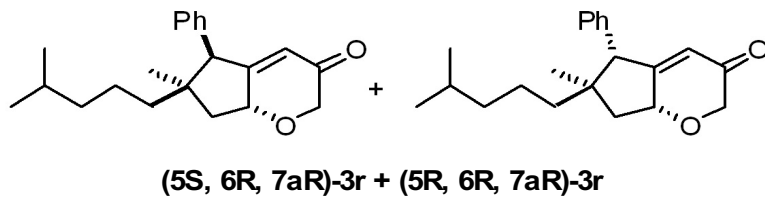
Parameter	Value
1 Title	zzt-5-30-1-H-again
2 Solvent	CDCl3
3 Spectrometer Frequency	499.86
4 Nucleus	1H



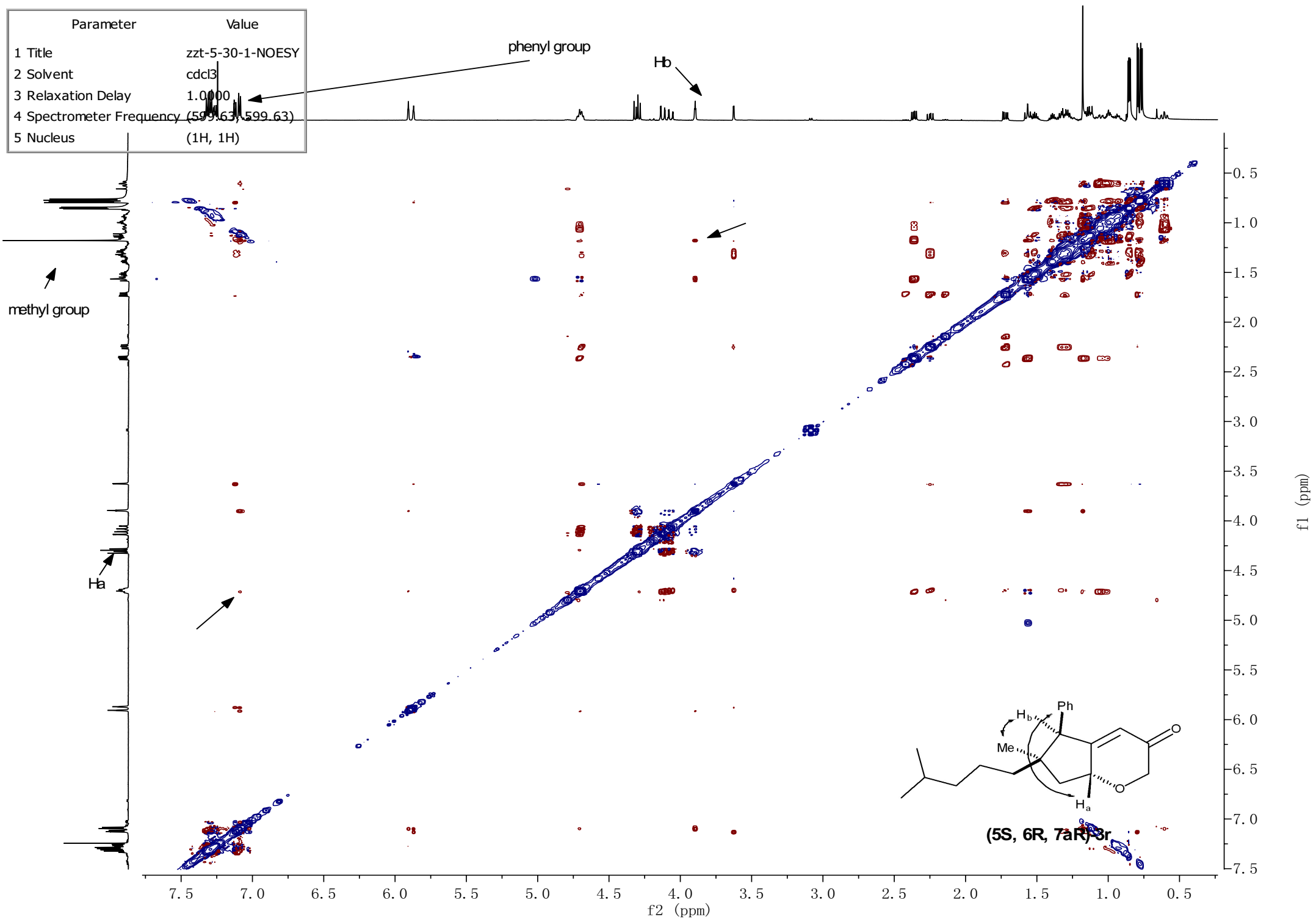
(5S, 6R, 7aR)-3r + (5R, 6R, 7aR)-3r



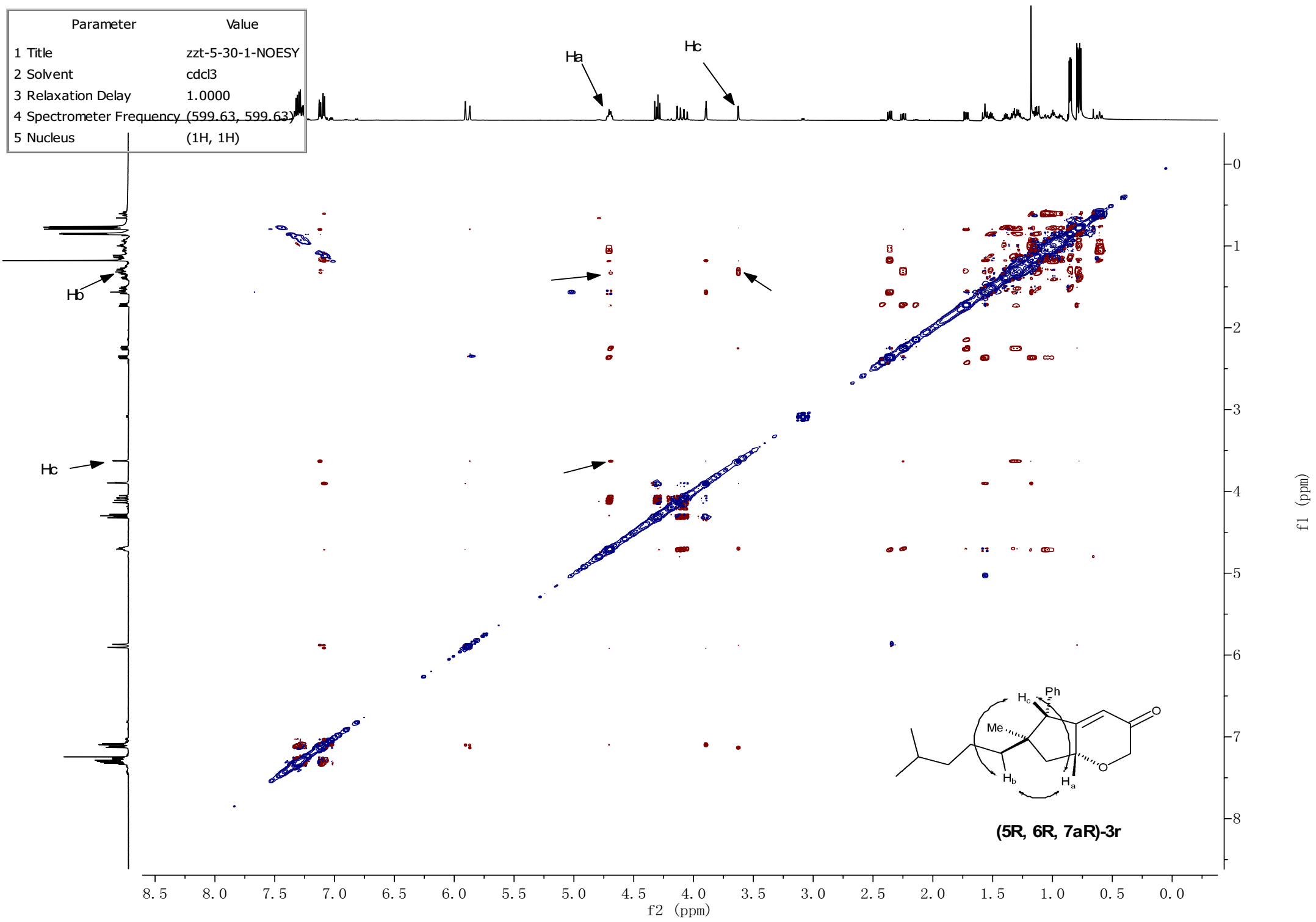
Parameter	Value
1 Title	zzt-5-30-1-C
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



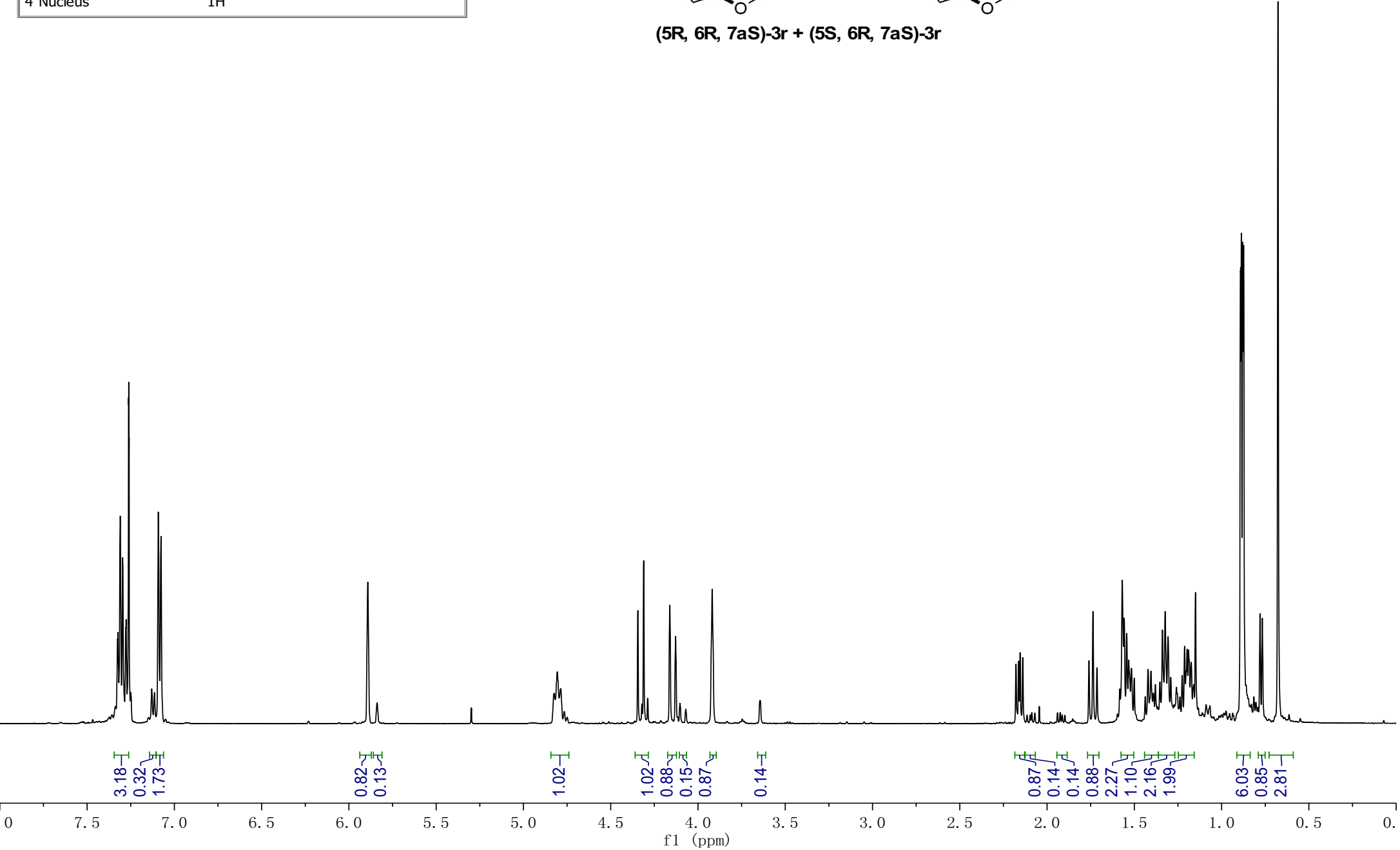
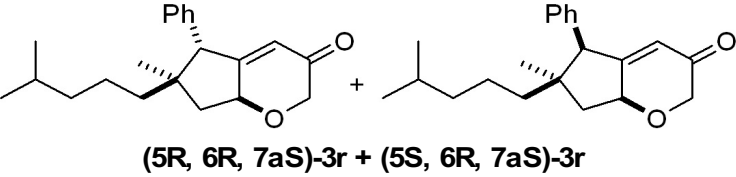
Parameter	Value
1 Title	zzt-5-30-1-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	(599.63, 599.63)
5 Nucleus	(1H, 1H)



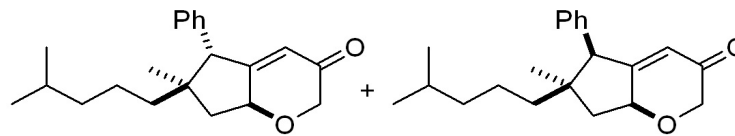
Parameter	Value
1 Title	zzt-5-30-1-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	(599.63, 599.63)
5 Nucleus	(1H, 1H)



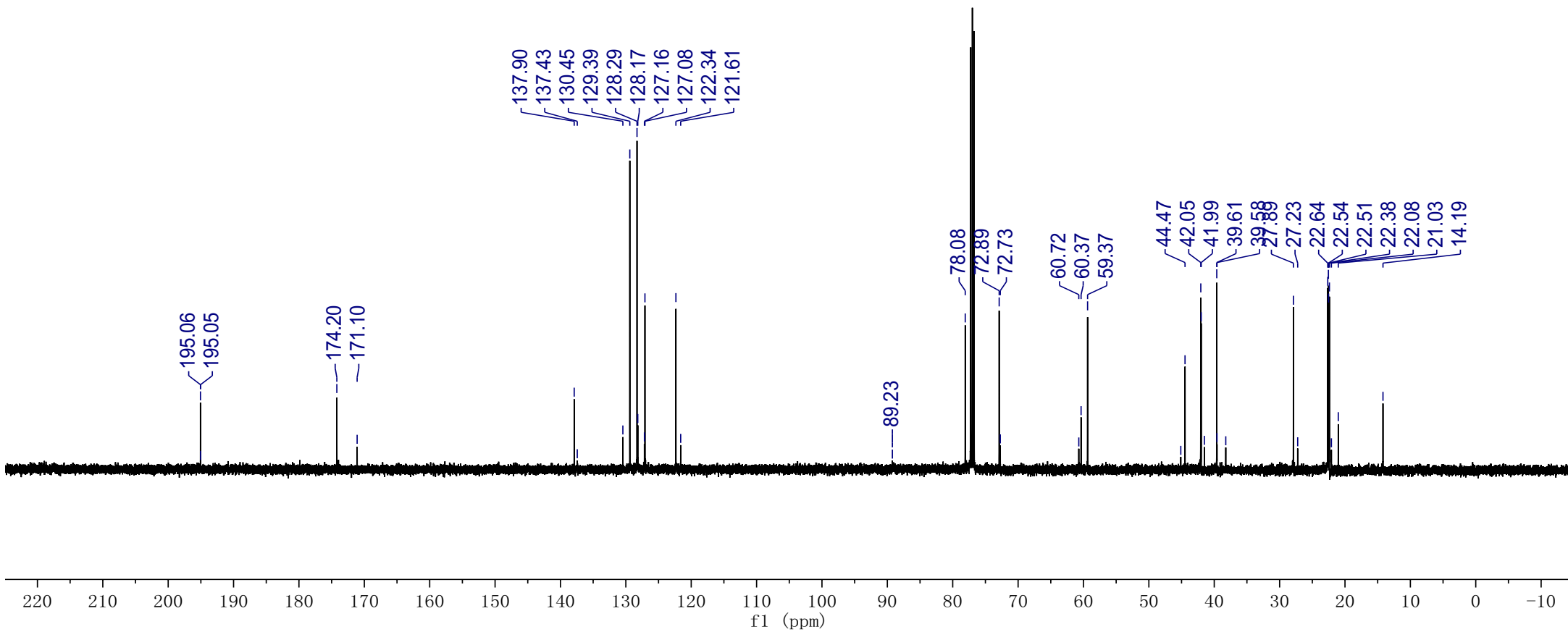
Parameter	Value
1 Title	(5 <i>R</i> , 6 <i>R</i> , 7 <i>aS</i>)-3r + (5 <i>S</i> , 6 <i>R</i> , 7 <i>aS</i>)-3r-H
2 Solvent	CDCl ₃
3 Spectrometer Frequency	499.86
4 Nucleus	¹ H



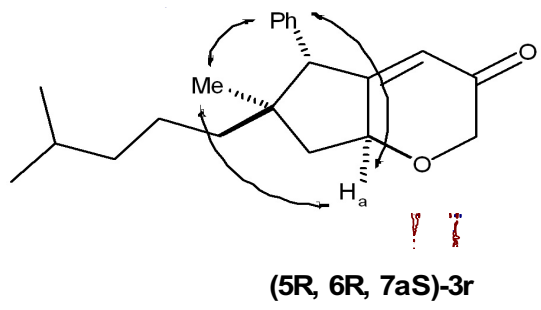
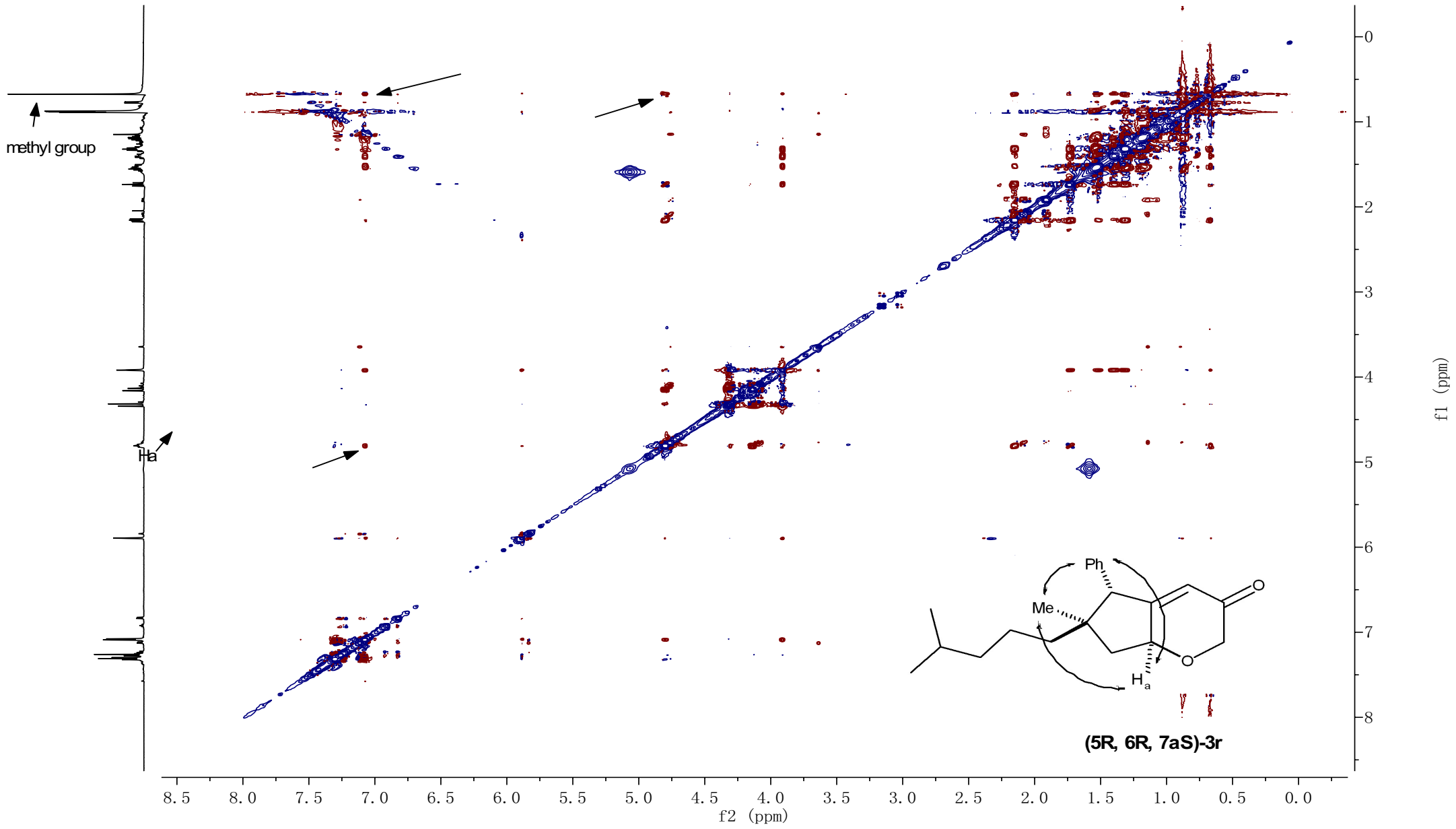
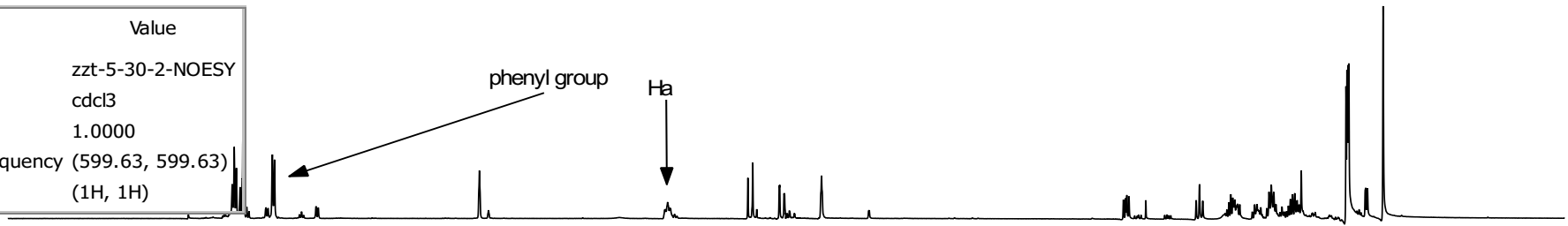
Parameter	Value
1 Title	zzt-5-30-2-C-maybe_overlapped
2 Solvent	CDCl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency	125.70



(5R, 6R, 7aS)-3r + (5S, 6R, 7aS)-3r



Parameter	Value
1 Title	zzt-5-30-2-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)



Parameter	Value
1 Title	zzt-5-30-2-NOESY
2 Solvent	cdcl3
3 Relaxation Delay	1.0000
4 Spectrometer Frequency (599.63, 599.63)	
5 Nucleus	(1H, 1H)

