Supporting Information

DBU-Promoted Carbonylative Synthesis of 1,3-Oxathiolan-2-ones from Propargylic Alcohols with TFBen as the CO source

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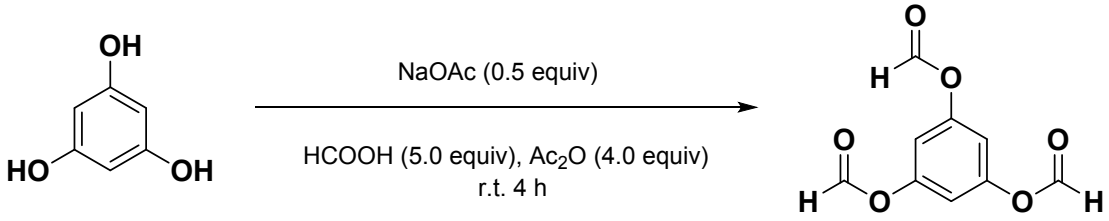
1. General Information

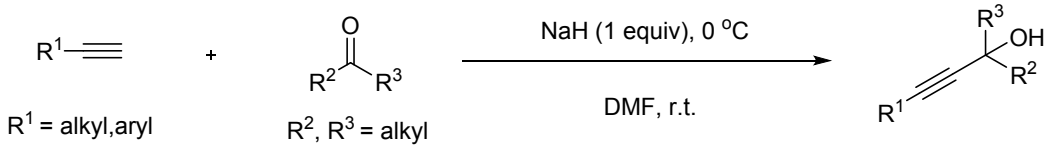
All reactions were performed under nitrogen protection unless otherwise noted. All reagents were obtained from commercial sources and used as received without further purification. Column chromatography was performed on silica gel (200−300 mesh) using petroleum ether (bp 60−90 °C) and ethyl acetate as eluent. Reactions were followed with TLC (0.25 mm silica gel 20 cm×20 cm). Vizualisation was accomplished with UV light.^{1}H and ^{13}C NMR spectra were taken on 400 MHz instruments, and spectral data were reported in ppm relative to tetramethylsilane (TMS) as internal standard and $CDCl_3$ as solvent.

2. Preparation of TFBen



Formic acid (8.4 mL, 222.8 mmol, 5.0 equiv) was added to acetic anhydride (16.8 mL, 178.2 mmol, 4.0 equiv) at rt. The mixture was stirred at 60 ^{o}C for 1 h and cooled to rt. The resulting solution was poured into a flask containing 1,3,5-trihydroxybenzene (5.62 g, 44.6 mmol, 1.0 equiv) and NaOAc (1.83 g, 22.3 mmol, 0.5 equiv) . The mixture was stirred for 4 h in a water bath and then diluted with toluene (100 mL), washed with H_2O (50 mL) twice. Keep the organic phase in fridge (2 - 8 ^{o}C) overnight. Then filtered and dried in vacuo to afford the desired product benzene-1,3,5-triyl triformate (TFBen) (5.1 g, 55%) as a white solid.

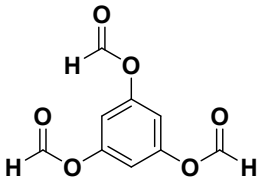
3. Procedure for the Synthesis of Propargylic Alcohols[1]



To a 50 mL round-bottom flask was added an alkyne (7 mmol) in DMF (5 mL). The mixture was cooled to 0 ^{o}C and stirred for 10 min. Then sodium hydride (7 mmol, 1 equiv) was added and the reaction continued at 0 ^{o}C for 4 - 6 h. A ketone (1.2 equiv) was added and the system was warmed to room temperature for 10 h. After the reaction was completed, the reaction mixture was diluted with

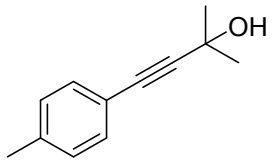
saturated sodium bicarbonate solution (60 mL) and extracted with ethyl acetate (40 mL) three times. The combined organic phases were dried with anhydrous Na_2SO_4, concentrated and purified by silica gel column chromatography (PE/EtOAc = 10/1) to obtain the desired propargylic alcohols.

4. Characterization of TFBen and Substrates



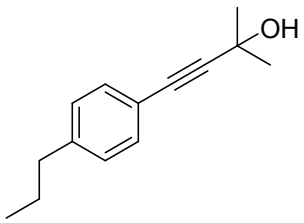
benzene-1,3,5-triyl triformate, TFBen[2]

5.1 g, white solid, mp 53.2–55.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.24 (s, 3H), 6.97 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 158.06, 150.30, 112.62.



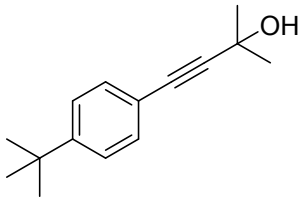
2-methyl-4-(*p*-tolyl)but-3-yn-2-ol, 1b

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.29 (d, *J* = 8.1 Hz, 2H), 7.07 (d, *J* = 7.9 Hz, 2H), 2.57 (s, 1H), 2.32 (s, 3H), 1.60 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 138.15, 131.43, 128.89, 119.59, 93.10, 82.10, 65.49, 31.42, 21.32.



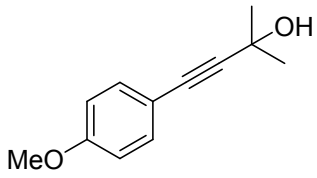
2-methyl-4-(4-propylphenyl)but-3-yn-2-ol, 1c

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.32 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 2.56 (t, *J* = 7.6 Hz, 2H), 2.21 (s, 1H), 1.64 – 1.58 (m, 8H), 0.91 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 143.01, 131.46, 128.35, 119.83, 93.10, 82.20, 65.56, 37.83, 31.47, 24.25, 13.65.



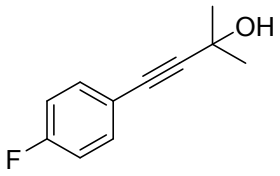
4-(4-(*tert*-butyl)phenyl)-2-methylbut-3-yn-2-ol, 1d

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.33 (q, *J* = 8.5 Hz, 4H), 1.61 (s, 6H), 1.30 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 151.47, 131.32, 125.21, 119.66, 93.09, 82.19, 65.63, 34.7, 31.52, 31.14.



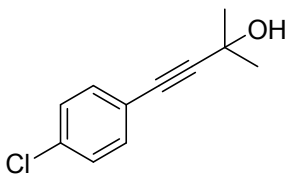
4-(4-methoxyphenyl)-2-methylbut-3-yn-2-ol, 1e

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.35 – 7.33 (m, 2H), 6.81 (d, *J* = 8.7 Hz, 2H), 3.78 (s, 3H), 2.51 (s, 1H), 1.60 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 159.39, 132.97, 114.78, 113.78, 92.43, 81.85, 65.49, 55.15, 31.46.



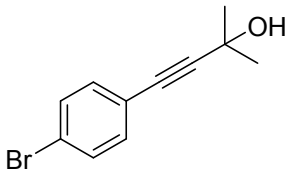
4-(4-fluorophenyl)-2-methylbut-3-yn-2-ol, 1f

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 (dd, *J* = 8.7, 5.4 Hz, 2H), 6.98 (t, *J* = 8.7 Hz, 2H), 2.37 (s, 1H), 1.61 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 162.385 (d, *J* = 247 Hz), 133.45 (d, *J* = 8 Hz),118.76 (d, *J* = 3 Hz), 115.44 (d, *J* = 22 Hz), 93.46, 81.01, 65.51, 31.38.



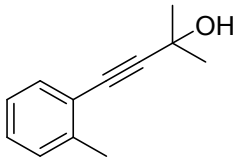
4-(4-chlorophenyl)-2-methylbut-3-yn-2-ol, 1g

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.33 (d, *J* = 8.5 Hz, 2H), 7.26 (d, *J* = 8.6 Hz, 2H), 2.31 (s, 1H), 1.61 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 134.21, 132.81, 128.53, 121.19, 94.70, 81.00, 65.55, 31.36.



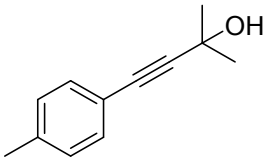
4-(4-bromophenyl)-2-methylbut-3-yn-2-ol, 1h

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.43 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.3 Hz, 2H), 2.16 (s, 1H), 1.61 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 133.01, 131.43, 122.39, 121.61, 94.85, 81.03, 65.53, 31.31.



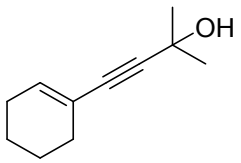
2-methyl-4-(*o*-tolyl)but-3-yn-2-ol, 1i

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.37 (d, *J* = 7.6 Hz, 1H), 7.22 – 7.17 (m, 2H), 7.13 – 7.09 (m, 1H), 2.41 (s, 3H), 1.63 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 140.08, 131.77, 129.32, 128.23, 125.44, 122.39, 97.86, 80.97, 65.69, 31.56, 20.54.



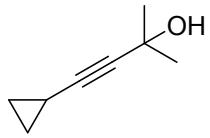
2-methyl-4-(*p*-tolyl)but-3-yn-2-ol, 1j

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.29 (d, *J* = 8.1 Hz, 2H), 7.07 (d, *J* = 7.9 Hz, 2H), 2.57 (s, 1H), 2.32 (s, 3H), 1.60 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 138.15, 131.43, 128.89, 119.59, 93.10, 82.10, 65.49, 31.42, 21.32.



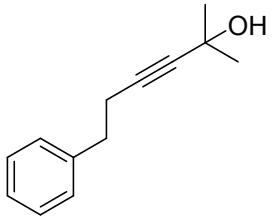
4-(cyclohex-1-en-1-yl)-2-methylbut-3-yn-2-ol, 1k

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.07 (s, 1H), 2.81 (s, 1H), 2.08 (s, 4H), 1.63 – 1.57 (m, 4H), 1.53 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 134.60, 120.00, 91.13, 83.59, 65.28, 31.41, 29.04, 25.41, 22.11, 21.31.



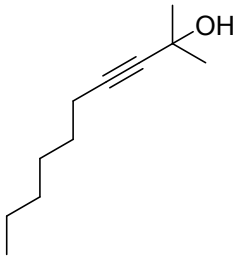
4-cyclopropyl-2-methylbut-3-yn-2-ol, 1i

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.13 (s, 1H), 1.48 (s, 6H), 1.23 (ddd, *J* = 10.1, 8.2, 5.0 Hz, 1H), 0.78 – 0.73 (m, 2H), 0.67 – 0.63 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 85.51, 80.26, 65.16, 31.65, 8.10, -0.77.



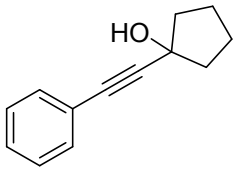
2-methyl-6-phenylhex-3-yn-2-ol, 1m

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.28 (q, *J* = 7.9 Hz, 2H), 7.22 – 7.20 (m, 1H), 2.80 (t, *J* = 7.5 Hz, 2H), 2.46 (t, *J* = 7.5 Hz, 2H), 1.46 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 140.62, 128.47, 128.26, 126.22, 85.96, 81.73, 65.17, 35.05, 31.59, 20.80.



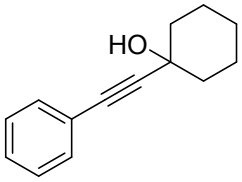
2-methyldec-3-yn-2-ol, 1n

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.16 (dd, *J* = 14.5, 7.3 Hz, 3H), 1.52 – 1.45 (m, 8H), 1.40 – 1.32 (m, 6H), 0.89 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 85.03, 82.53, 65.20, 31.67, 31.24, 28.58, 28.43, 22.47, 18.50, 13.98.



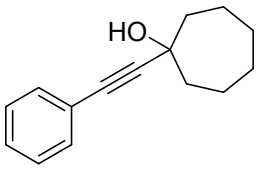
1-(phenylethynyl)cyclopentan-1-ol, 1o

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.43 – 7.39 (m, 2H), 7.29 – 7.25 (m, 3H), 2.23 (s, 1H), 2.08 – 2.03 (m, 4H), 1.90 - 1.73 (dddd, *J* = 15.2, 12.2, 11.4, 7.5 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.53, 128.16, 128.08, 122.82, 92.87, 82.99, 74.81, 42.43, 23.43.



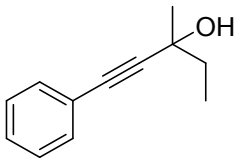
1-(phenylethynyl)cyclohexan-1-ol, 1p

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.43 – 7.41 (m, 2H), 7.29 (dd, *J* = 6.5, 3.6 Hz, 3H), 2.38 (s, 1H), 2.03 – 1.99 (m, 2H), 1.74 – 1.54 (m, 7H), 1.29 – 1.26 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.56, 128.12, 128.05, 122.83, 92.81, 84.24, 68.99, 39.93, 25.11, 23.32.



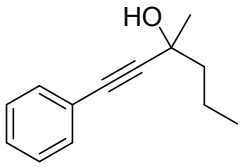
1-(phenylethynyl)cycloheptan-1-ol, 1q

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.43 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.30 – 7.28 (m, 3H), 2.12 (dd, *J* = 13.9, 7.4 Hz, 3H), 1.95 – 1.88 (m, 2H), 1.62 (dd, *J* = 11.5, 7.7 Hz, 7H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.58, 128.17, 128.10, 122.87, 93.77, 83.52, 72.17, 43.11, 27.90, 22.25.



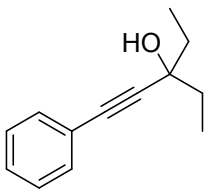
3-methyl-1-phenylpent-1-yn-3-ol, 1r

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.40 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.26 – 7.25 (m, 3H), 2.86 (s, 1H), 1.85 – 1.71 (m, 2H), 1.56 (s, 3H), 1.09 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 131.48, 128.05, 127.99, 122.72, 92.73, 83.15, 68.93, 36.48, 29.09, 8.99.



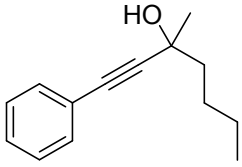
3-methyl-1-phenylhex-1-yn-3-ol, 1s

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.40 (dd, *J* = 5.0, 2.2 Hz, 2H), 7.29 – 7.27 (m, 3H), 2.40 (s, 1H), 1.76 – 1.70 (m, 2H), 1.62 – 1.57 (m, 5H), 0.98 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.55, 128.14, 128.10, 122.73, 92.94, 83.15, 68.50, 45.93, 29.74, 18.06, 14.18.



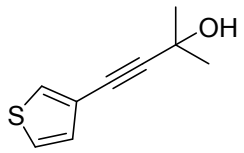
3-ethyl-1-phenylpent-1-yn-3-ol, 1t

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42 (dd, *J* = 6.5, 2.9 Hz, 2H), 7.29 – 7.27 (m, 3H), 2.26 (s, 1H), 1.83 – 1.69 (m, 4H), 1.10 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.61, 128.15, 128.10, 122.82, 91.64, 84.38, 72.51, 34.38, 8.62.



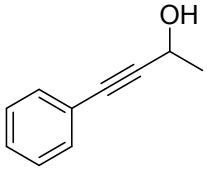
3-methyl-1-phenylhept-1-yn-3-ol, 1u

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.28 (dd, *J* = 6.4, 3.6 Hz, 3H), 2.52 (s, 1H), 1.861– 1.69 (m, 2H), 1.60 – 1.48 (m, 5H), 1.42 – 1.33 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.54, 128.12, 128.07, 122.75, 93.00, 83.12, 68.51, 43.43, 29.70, 26.87, 22.74, 13.98.



2-methyl-4-(thiophen-3-yl)but-3-yn-2-ol, 1v

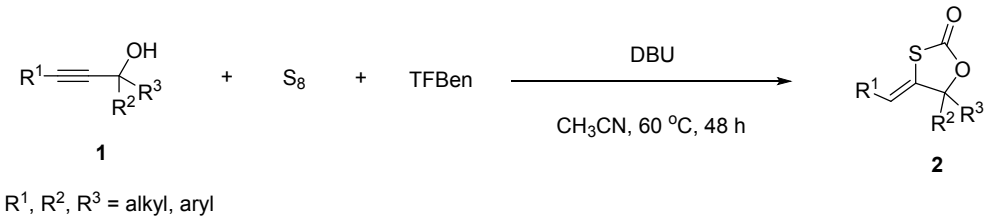
Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42 – 7.41 (m, 1H), 7.25 – 7.24 (m, 1H), 7.09 – 7.08 (m, 1H), 1.61 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 129.76, 128.54, 125.17, 121.62, 93.35, 77.17, 65.49, 31.35.



4-phenylbut-3-yn-2-ol, 2x

Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.43 (dd, *J* = 6.2, 2.6 Hz, 2H), 7.31 – 7.27 (m, 2H), 4.76 (dd, *J* = 13.1, 6.5 Hz, 1H), 2.29 (s, 1H), 1.55 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 131.49, 128.14, 128.10, 122.53, 91.04, 83.73, 58.49, 24.18.

5. General Procedure for Reaction of Propargylic Alcohols with S_8 and TFBen

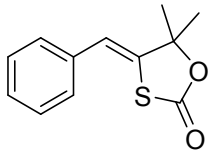


1 **2**

R^1, R^2, R^3 = alkyl, aryl

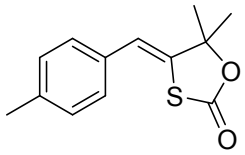
S_8 (4 equiv), TFBen (1.0 equiv) and DBU (2.0 equiv) were added to a 15 mL tube equipped with a magnetic stirrer which was then placed under vacuum and refilled with nitrogen three times. A solution of the propargylic alcohol **1** (0.5 mmol) in CH_3CN (2.0 mL) was added to the reaction tube, then the tube was sealed and the mixture was stirred at 60 °C for 48 h. After the reaction was completed, the reaction mixture was diluted with 50 mL water and extracted with 30 mL EtOAc three times. The combined organic phases were dried with anhydrous Na_2SO_4, concentrated and purified by silica gel column chromatography (PE/EtOAc = 50/1) to obtain the desired products **2**.

6. Characterization of Products



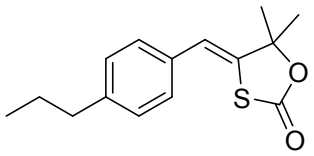
(*Z*)-4-benzylidene-5,5-dimethyl-1,3-oxathiolan-2-one, 2a

95.7 mg, 87% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 (dd, *J* = 9.9, 5.4 Hz, 2H), 7.28 (dd, *J* = 13.0, 5.7 Hz, 3H), 6.51 (s, 1H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.30, 138.65, 135.09, 128.78, 127.89, 127.86, 120.05, 90.80, 28.34. HRMS (ESI): [M+H$^+$] calcd. for $C_{12}H_{13}O_2S^+$, 221.0558; found, 221.0561.



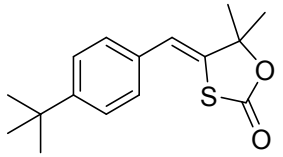
(*Z*)-5,5-dimethyl-4-(4-methylbenzylidene)-1,3-oxathiolan-2-one, 2b

108.8 mg, 93% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.19 (s, 4H), 6.48 (s, 1H), 2.35 (s, 3H), 1.73 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.52, 137.84, 137.26, 132.18, 129.42, 127.78, 119.97, 90.79, 28.27, 21.18. HRMS (ESI): [M+H$^+$] calcd. for $C_{13}H_{15}O_2S^+$, 235.0787; found, 235.0776.



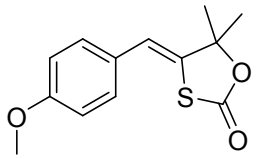
(*Z*)-5,5-dimethyl-4-(4-propylbenzylidene)-1,3-oxathiolan-2-one, 2c

115.3 mg, 88% yield, red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.21 (d, *J* = 3.7 Hz, 4H), 6.49 (s, 1H), 2.62 – 2.57 (m, 2H), 1.73 (d, *J* = 3.7 Hz, 6H), 1.67 - 1.61 (dd, *J* = 14.9, 7.4 Hz, 2H), 0.96 -0.92 (td, *J* = 7.3, 3.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.52, 142.68, 137.29, 132.43, 128.88, 127.81, 120.00, 90.81, 37.67, 28.31, 24.34, 13.71. HRMS (ESI): [M+H$^+$] calcd. for $C_{15}H_{19}O_2S^+$, 263.1100; found, 263.1094.



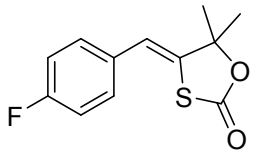
(*Z*)-4-(4-(*tert*-butyl)benzylidene)-5,5-dimethyl-1,3-oxathiolan-2-one, 2d

131.1 mg, 95% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 6.49 (s, 1H), 1.73 (s, 6H), 1.34 – 1.26 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.57, 151.09, 137.43, 132.16, 127.67, 125.73, 119.83, 90.85, 34.66, 31.16, 28.34.HRMS (ESI): [M+H$^+$] calcd. for $C_{16}H_{21}O_2S^+$, 277.1257; found, 277.1267.



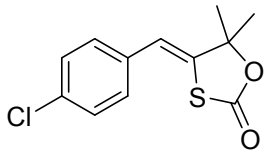
(*Z*)-4-(4-methoxybenzylidene)-5,5-dimethyl-1,3-oxathiolan-2-one, 2e

97.5 mg, 78% yield, yellow solid. Mp 85 – 87 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.23 (d, *J* = 8.7 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 6.46 (s, 1H), 3.83 (s, 3H), 1.73 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.56, 159.10, 135.89, 129.25, 127.64, 119.60, 114.18, 90.81, 55.28, 28.27. HRMS (ESI): [M+H$^+$] calcd. for $C_{13}H_{15}O_3S^+$, 251.0736; found, 251.0737.



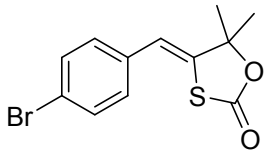
(*Z*)-4-(4-fluorobenzylidene)-5,5-dimethyl-1,3-oxathiolan-2-one, 2f

80.9 mg, 68% yield, white solid. Mp 87 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.27 (dd, *J* = 8.5, 5.3 Hz, 2H), 7.09 (t, *J* = 8.5 Hz, 2H), 6.49 (s, 1H), 1.74 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.01, 161.91 (d, *J* = 247.0 Hz), 138.44 (d, *J* = 2.0 Hz), 131.24 (d, *J* = 3 Hz), 129.59 (d, *J* = 8 Hz), 118.92, 115.80 (d, *J* = 22 Hz), 90.79, 28.26. HRMS (ESI): [M+H$^+$] calcd. for $C_{12}H_{12}FO_2S^+$, 239.0537; found, 239.0530.



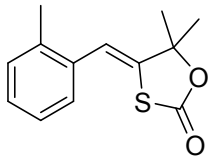
(*Z*)-4-(4-chlorobenzylidene)-5,5-dimethyl-1,3-oxathiolan-2-one, 2g

106.6 mg, 84% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36 (d, *J* = 8.5 Hz, 2H), 7.22 (d, *J* = 8.4 Hz, 2H), 6.46 (s, 1H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.76, 139.54, 133.57, 129.09, 128.95, 118.84, 118.82, 90.81, 28.29. HRMS (ESI): [M+H$^+$] calcd. for $C_{12}H_{12}ClO_2S^+$, 255.0241; found, 255.0245.



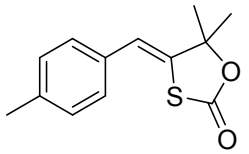
(*Z*)-4-(4-bromobenzylidene)-5,5-dimethyl-1,3-oxathiolan-2-one, 2h

104.6 mg, 70% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.51 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 6.44 (s, 1H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.79, 139.68, 133.99, 131.91, 129.35, 121.73, 118.87, 90.86, 28.29. HRMS (ESI): [M+H$^+$] calcd. for $C_{12}H_{12}BrO_2S^+$, 298.9736; found, 298.9738.



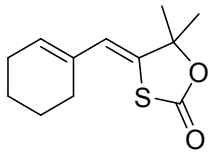
(*Z*)-5,5-dimethyl-4-(2-methylbenzylidene)-1,3-oxathiolan-2-one, 2i

99.4 mg, 85% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.25 – 7.22 (m, 2H), 7.20 (s, 2H), 6.59 (s, 1H), 2.29 (s, 3H), 1.76 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.55, 140.79, 136.10, 134.60, 130.27, 128.16, 126.80, 126.19, 118.64, 90.15, 28.31, 19.64. HRMS (ESI): [M+H$^+$] calcd. for $C_{13}H_{15}O_2S^+$, 235.0787; found, 235.0796.



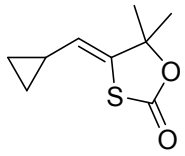
(*Z*)-5,5-dimethyl-4-(4-methylbenzylidene)-1,3-oxathiolan-2-one, 2j

99.4 mg, 85% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.19 (s, 4H), 6.48 (s, 1H), 2.35 (s, 3H), 1.73 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.52, 137.84, 137.26, 132.18, 129.42, 127.78, 119.97 , 90.79, 28.27, 21.18. HRMS (ESI): [M+H$^+$] calcd. for $C_{13}H_{15}O_2S^+$, 235.0787; found, 235.0776.



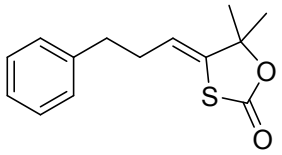
(*Z*)-4-(cyclohex-1-en-1-ylmethylene)-5,5-dimethyl-1,3-oxathiolan-2-one, 2k

97.4 mg, 87% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.94 (s, 1H), 5.78 (s, 1H), 2.18 – 2.15 (m, 4H), 1.70 - 1.66 (dd, *J* = 8.3, 4.4 Hz, 2H), 1.63 (s, 6H), 1.60 – 1.57 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 169.28, 133.97, 133.61, 131.33, 123.38, 90.64, 28.22, 27.32, 25.79, 22.49, 21.66. HRMS (ESI): [M+H$^+$] calcd. for $C_{12}H_{17}O_2S^+$, 225.0944; found, 225.0936.



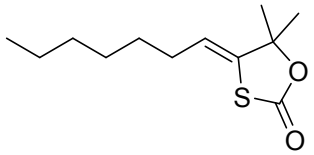
(*Z*)-4-(cyclopropylmethylene)-5,5-dimethyl-1,3-oxathiolan-2-one, 2i

71.7 mg, 78% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 4.99 (d, *J* = 8.9 Hz, 1H), 1.59 (s, 6H), 1.18 – 1.12 (m, 1H), 0.87 (dt, *J* = 6.4, 4.7 Hz, 2H), 0.49 – 0.45 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.77, 136.66, 124.20, 89.40, 28.03, 13.60, 7.28. HRMS (ESI): [M+H$^+$] calcd. for $C_9H_{13}O_2S^+$, 185.0631; found, 185.0645.



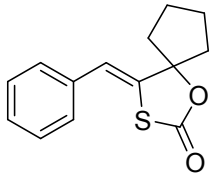
(*Z*)-5,5-dimethyl-4-(3-phenylpropylidene)-1,3-oxathiolan-2-one, 2m

90.5 mg, 73% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.29 (t, *J* = 7.2 Hz, 2H), 7.21 (dd, *J* = 10.4, 3.9 Hz, 1H), 7.15 (d, *J* = 6.8 Hz, 2H), 5.50– 5.47 (m, 1H), 2.73 (t, *J* = 7.3 Hz, 2H), 2.41 – 2.22 (m, 2H), 1.56 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.33, 140.45, 139.88, 128.40, 128.38, 126.20, 119.56, 89.33, 34.66, 33.58, 28.01. HRMS (ESI): [M+Na$^+$] calcd. for $C_{14}H_{17}O_2S^+$, 271.0763; found, 271.0765.



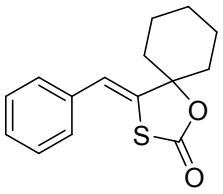
(*Z*)-4-heptylidene-5,5-dimethyl-1,3-oxathiolan-2-one, 2n

91.2 mg, 80% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.49 (t, *J* = 7.2 Hz, 1H), 2.03 – 1.97 (m, 2H), 1.61 (d, *J* = 2.4 Hz, 6H), 1.41 (dd, *J* = 13.9, 6.8 Hz, 2H), 1.44 – 1.38 (m, 6H), 0.89 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.60, 138.81, 120.91, 89.35, 31.97, 31.51, 28.64, 28.59, 28.10 , 22.51, 14.00. HRMS (ESI): [M+Na$^+$] calcd. for $C_{12}H_{21}O_2S^+$, 244.1492; found, 244.1484.



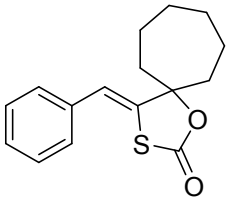
(*Z*)-4-benzylidene-1-oxa-3-thiaspiro[4.4]nonan-2-one, 2o

97.1 mg, 79% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42 – 7.38 (m, 1H), 7.30 – 7.27 (m, 2H), 6.52 (s, 1H), 1.74 (s, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.70, 137.32, 135.20, 128.78, 127.86, 127.78, 119.92, 93.63, 34.43, 27.12, 7.88. HRMS (ESI): [M+H$^+$] calcd. for $C_{14}H_{15}O_2S^+$, 247.0787; found, 247.0804.



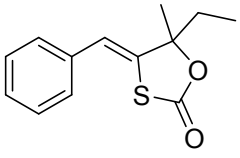
(*Z*)-4-benzylidene-1-oxa-3-thiaspiro[4.5]decan-2-one, 2p

127.4 mg, 98% yield, yellow solid, Mp 120 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.40 – 7.36 (m, 2H), 7.30 – 7.25 (m, 3H), 6.49 (s, 1H), 2.14 – 2.12 (m, 2H), 1.79 - 1.75 (t, *J* = 7.5 Hz, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.36, 138.56, 135.17, 128.68, 127.87, 127.65, 119.98, 92.59, 37.33, 24.56, 22.32. HRMS (ESI): [M+H$^+$] calcd. for $C_{15}H_{17}O_2S^+$, 261.0871; found, 261.0869.



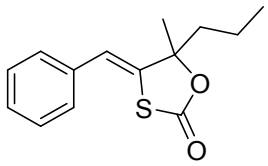
(*Z*)-4-benzylidene-1-oxa-3-thiaspiro[4.6]undecan-2-one, 2q

123.3 mg, 90% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 – 7.38 (m, 2H), 7.31 – 7.22 (m, 3H), 6.54 (s, 1H), 2.35 - 2.25 (dd, *J* = 13.2, 4.5 Hz, 2H), 2.13 – 2.06 (m, 2H), 2.03 – 1.82 (m, 6H), 1.33 – 1.25 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.59, 140.32, 135.28, 128.71, 127.94, 127.70, 119.80, 96.05, 40.90, 28.70, 22.59. HRMS (ESI): [M+H$^+$] calcd. for $C_{16}H_{19}O_2S^+$, 275.1100; found, 275.1088.



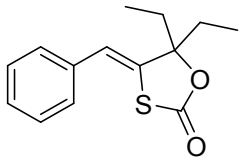
(*Z*)-4-benzylidene-5-ethyl-5-methyl-1,3-oxathiolan-2-one, 2r

94.7 mg, 81% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.40 (t, *J* = 7.7 Hz, 2H), 7.31 (t, *J* = 7.2 Hz, 3H), 6.44 (s, 1H), 2.08 – 1.94 (m, 2H), 1.70 (s, 3H), 1.02 (dd, *J* = 7.7, 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.66, 137.17, 135.13, 128.73, 127.81, 127.72, 119.90, 93.60, 34.36, 27.05, 7.83. HRMS (ESI): [M+H$^+$] calcd. for $C_{13}H_{15}O_2S^+$, 235.0787; found, 235.0791.



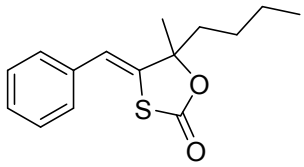
(*Z*)-4-benzylidene-5-methyl-5-propyl-1,3-oxathiolan-2-one, 2s

104.1 mg, 84% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.40 (t, *J* = 7.6 Hz, 2H), 7.30 (dd, *J* = 7.9, 2.5 Hz, 3H), 6.44 (s, 1H), 2.01 – 1.85 (m, 2H), 1.70 (s, 3H), 1.52 - 1.43 (dtd, *J* = 10.6, 7.3, 3.5 Hz, 2H), 0.95 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.68, 137.63, 135.18, 128.78, 127.85, 127.77, 119.83, 93.32, 43.70, 27.42, 16.82, 13.97. HRMS (ESI): [M+H$^+$] calcd. for $C_{14}H_{17}O_2S^+$, 249.0944; found, 249.0948.



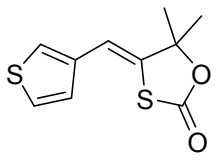
(*Z*)-4-benzylidene-5,5-diethyl-1,3-oxathiolan-2-one, 2t

115.3 mg, 93% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 (t, *J* = 7.6 Hz, 2H), 7.32 – 7.27 (m, 3H), 6.39 (s, 1H), 2.06 (dd, *J* = 14.5, 7.3 Hz, 2H), 1.89 (dd, *J* = 14.5, 7.3 Hz, 2H), 1.00 (t, *J* = 7.3 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 169.11, 135.64, 135.26, 128.71, 127.78, 127.64, 119.64, 96.66, 33.46, 7.40. HRMS (ESI): [M+Na$^+$] calcd. for $C_{14}H_{17}O_2S^+$, 271.0763; found, 271.0777.



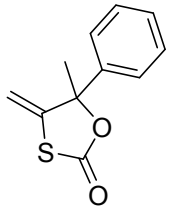
(*Z*)-4-benzylidene-5-butyl-5-methyl-1,3-oxathiolan-2-one, 2u

127.1 mg, 97% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42 - 7.38 (t, *J* = 7.7 Hz, 2H), 7.31 – 7.28 (m, 3H), 6.44 (s, 1H), 2.04 – 1.87 (m, 2H), 1.70 (s, 3H), 1.45 – 1.31 (m, 4H), 0.93 - 0.89 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.66, 137.59, 135.17, 128.76, 127.84, 127.74 , 119.81, 93.34, 41.24, 27.46, 25.46, 22.56, 13.85. HRMS (ESI): [M+Na$^+$] calcd. for $C_{15}H_{19}O_2S^+$, 285.0920; found, 285.0913.



(*Z*)-5,5-dimethyl-4-(thiophen-3-ylmethylene)-1,3-oxathiolan-2-one, 2v

89.2 mg, 79% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.22 (d, *J* = 1.7 Hz, 1H), 7.12 (dd, *J* = 5.0, 1.0 Hz, 1H), 6.54 (s, 1H), 1.72 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.89, 137.59, 136.55, 127.09, 126.27, 123.52, 114.38, 90.53, 28.24. HRMS (ESI): [M+H$^+$] calcd. for $C_{10}H_{11}O_2S_2^+$, 227.0195; found, 227.0192.



5-methyl-4-methylene-5-phenyl-1,3-oxathiolan-2-one, 2w

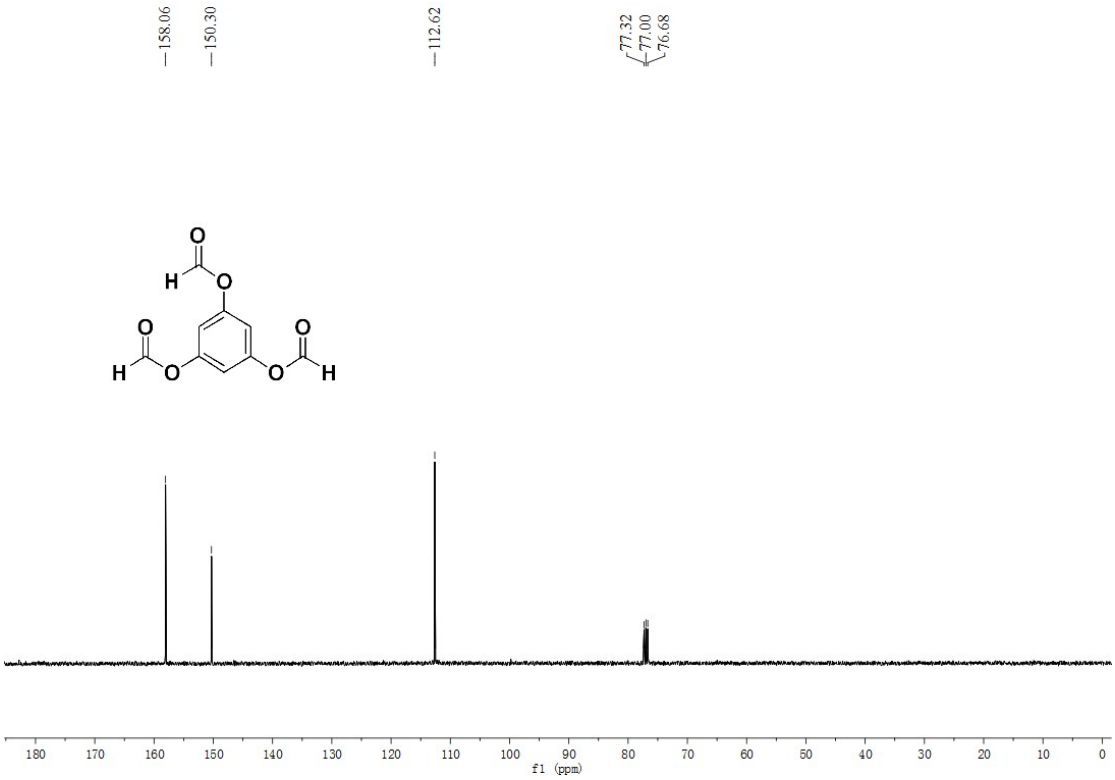
87.5 mg, 85% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.51- 7.48 (dd, *J* = 8.2, 1.5 Hz, 2H), 7.38 (t, *J* = 7.6 Hz, 3H), 5.32 (s, 2H), 1.98 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.44, 146.76, 140.47, 128.64, 124.91, 108.62, 99.92, 91.99, 27.01. HRMS (ESI): [M+H$^+$] calcd. for $C_{11}H_{11}O_2S^+$, 207.0402; found, 207.0405.

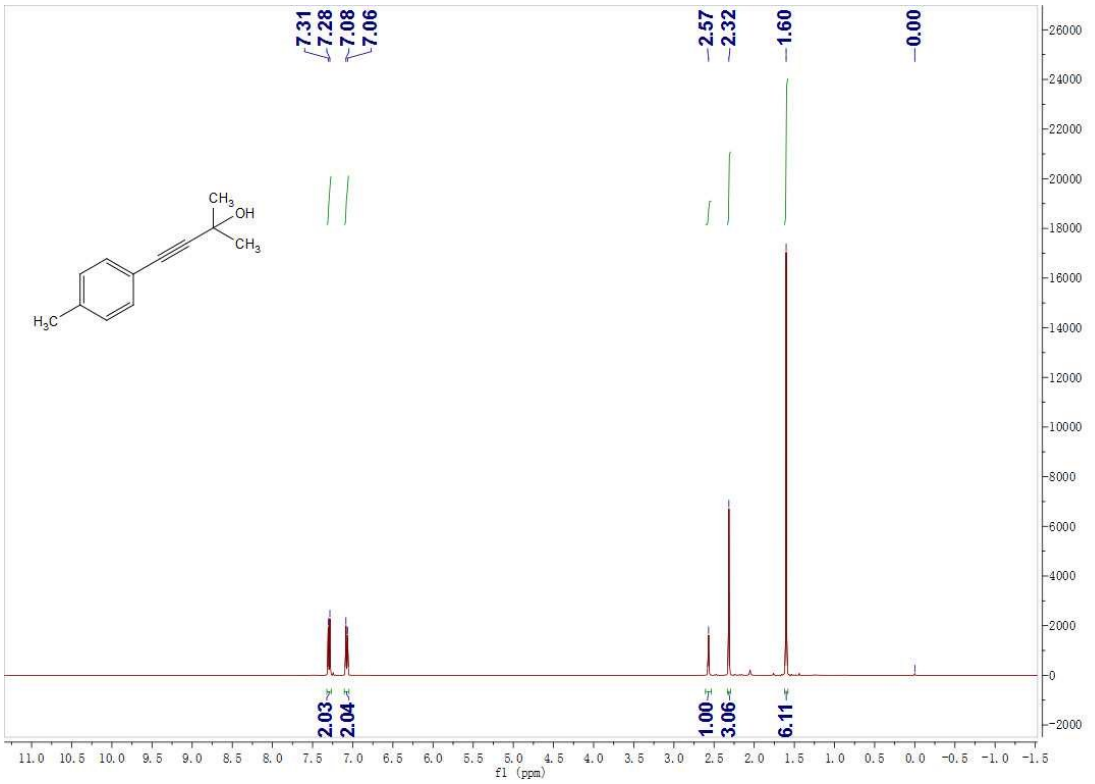
7. References

1 X. Wang, M. Emoto, A. Sugimoto, Y. Miyake, K. Itto, M. Amasaka, S. Xu, H. Hirata, H. Fujii, H. Arimoto, *Tetrahedron.*, 2014, **55**, 2146-2149.

2 (a) L. Jiang, X. Qi, X. –F. Wu, *Tetrahedron.* 2016**, 57**, 3368-3370. (b) L. Jiang, R. Li, C. Zhou, X. Qi, J. –B. Peng, X. –F. Wu, *Molecular Catalysis*, 2017**, 433**, 8-11.

8. Spectrum of TFBen and Substrates

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2.04
1.00
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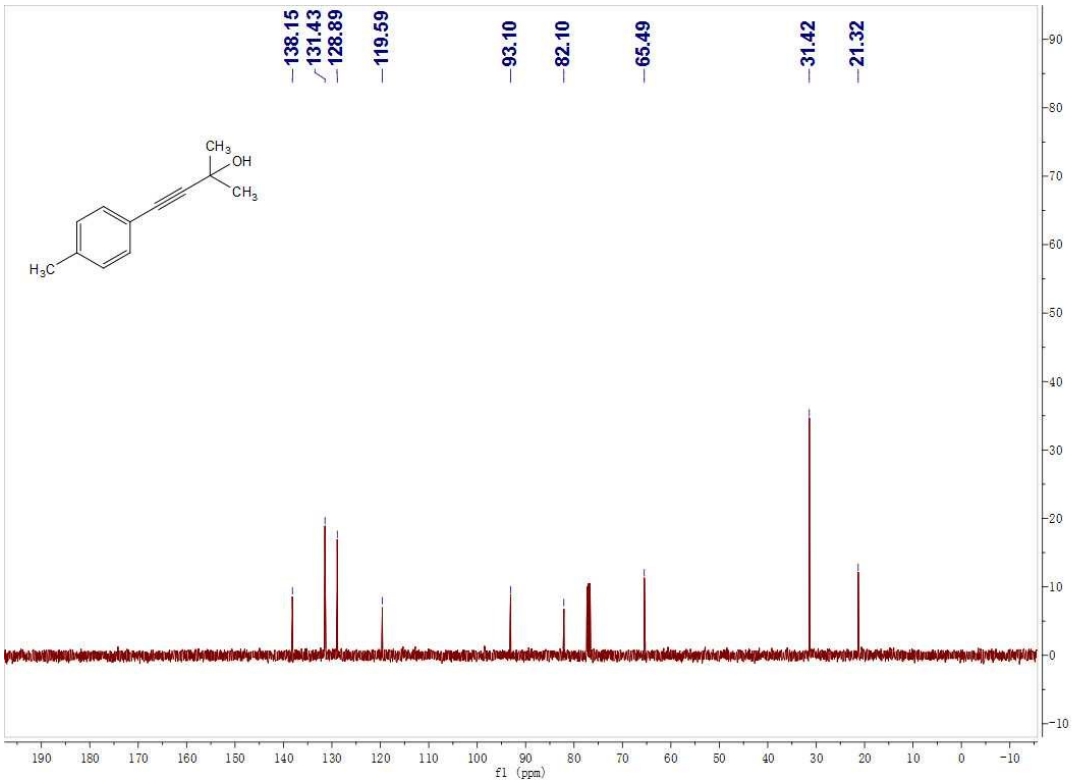
6.11
f1 (ppm)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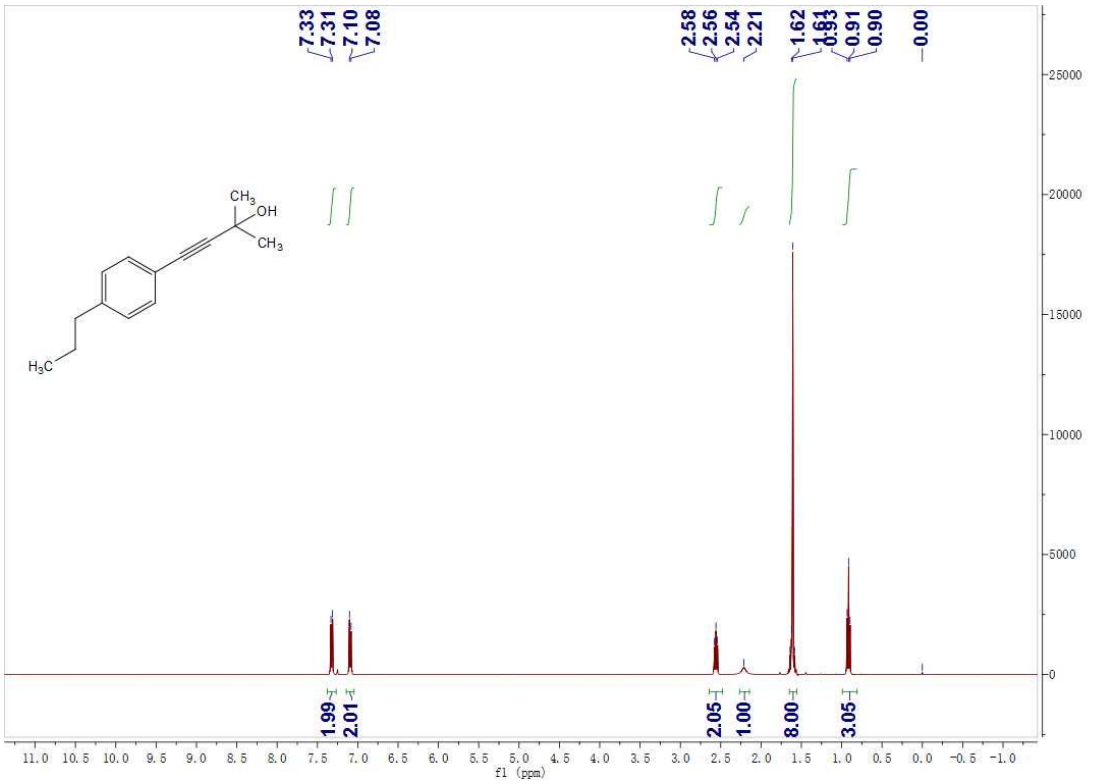
f1 (ppm)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CH3
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1.99
2.01
2.05
1.00
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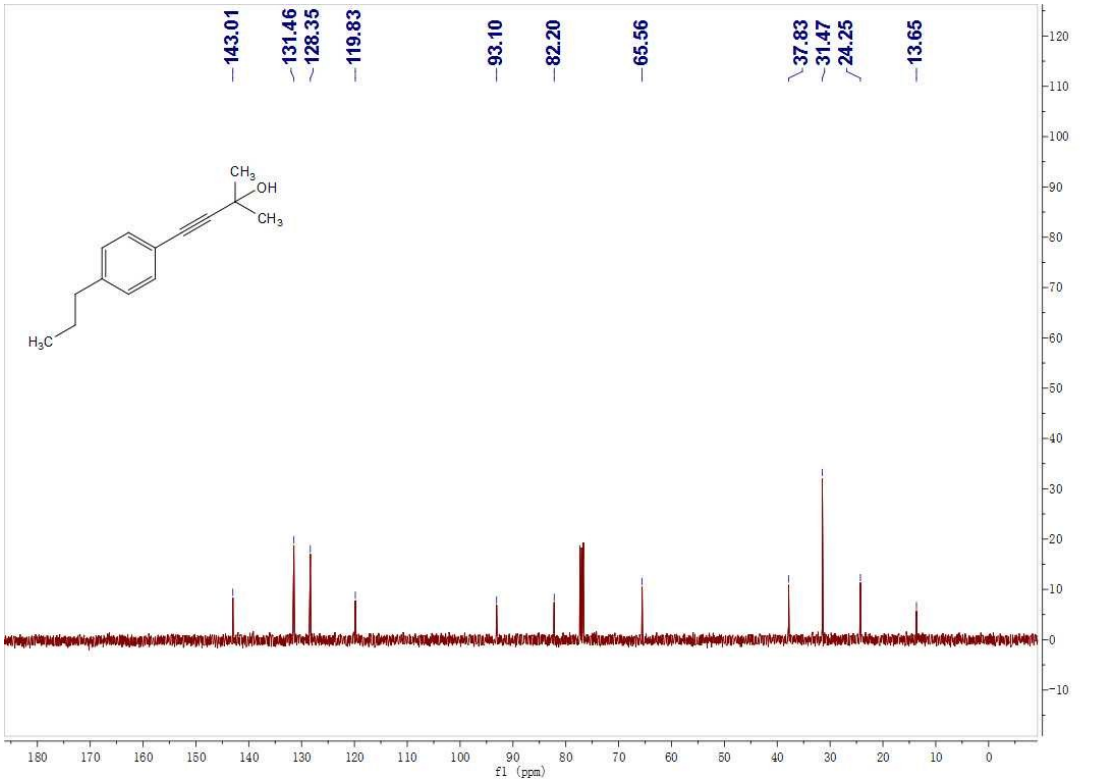
3.05
f1 (ppm)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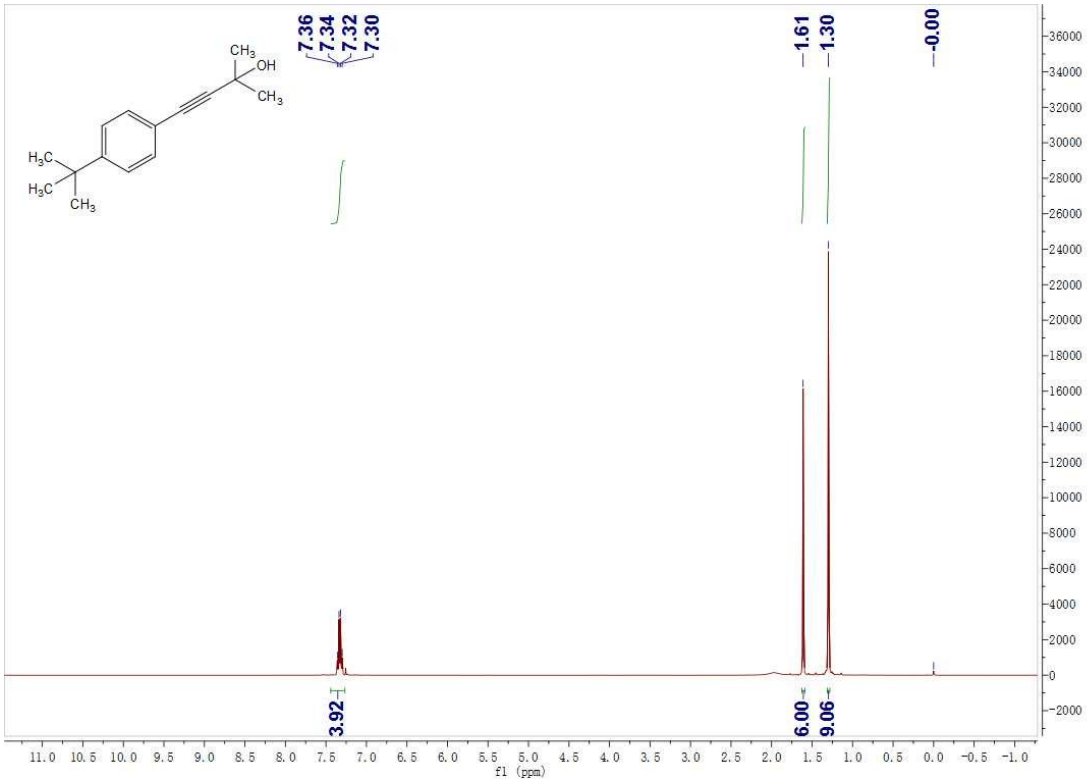
f1 (ppm)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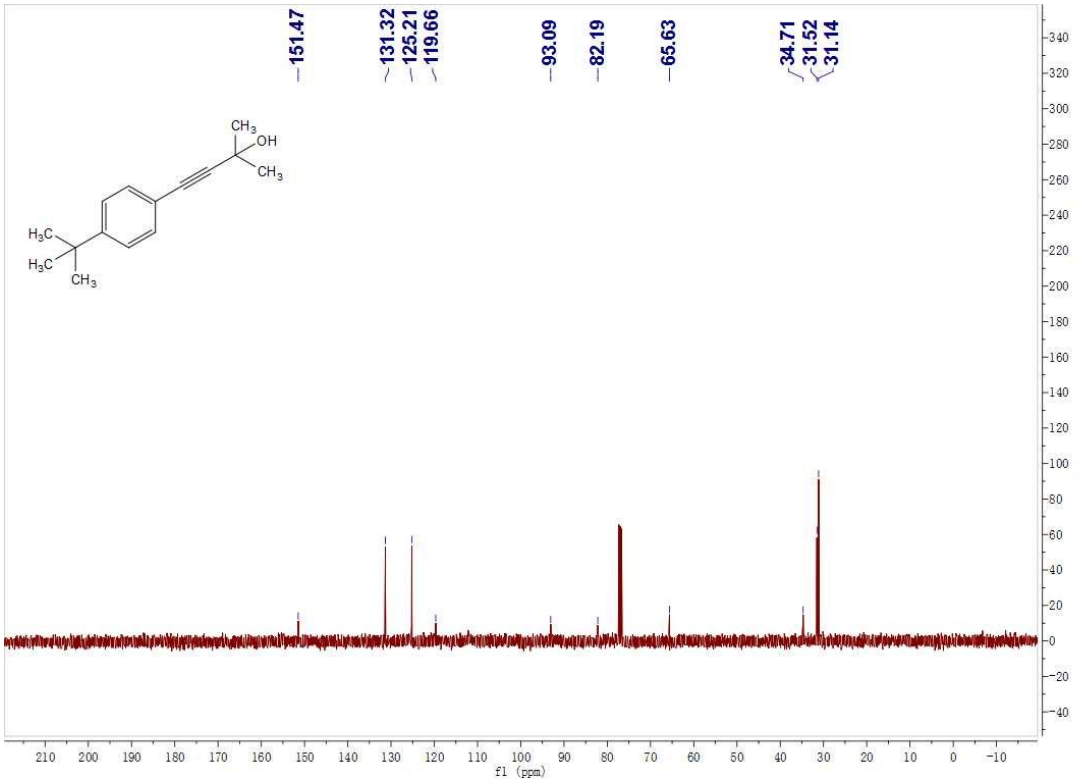
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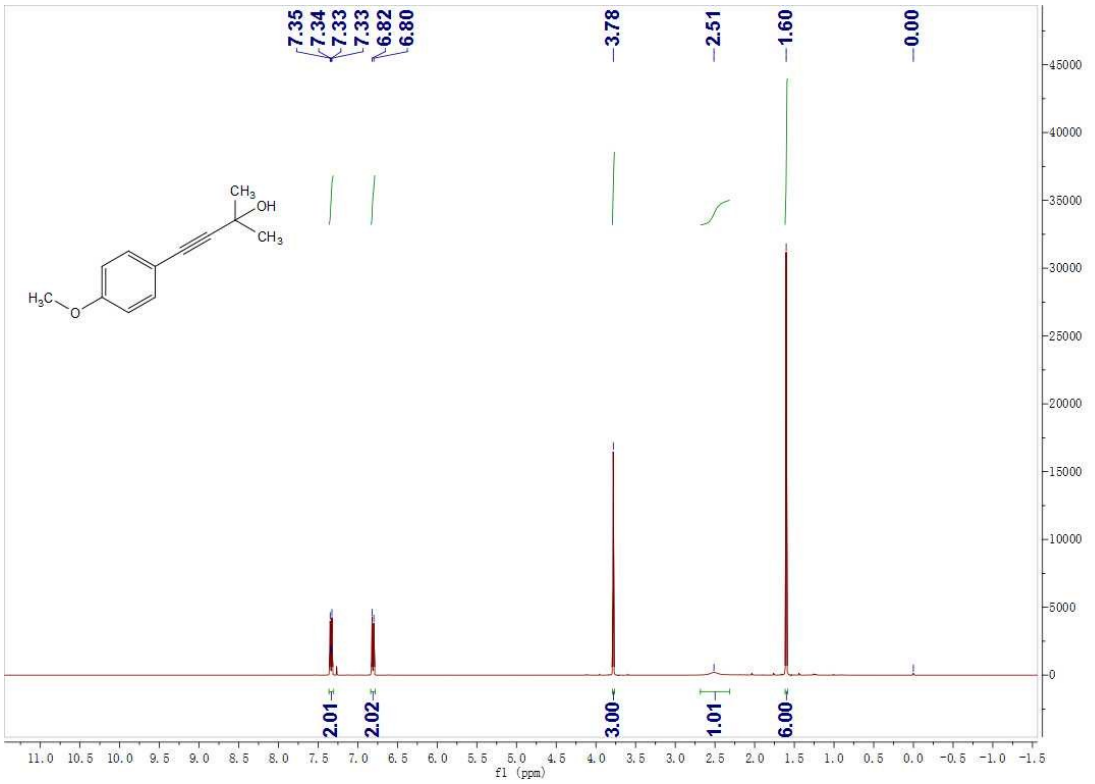
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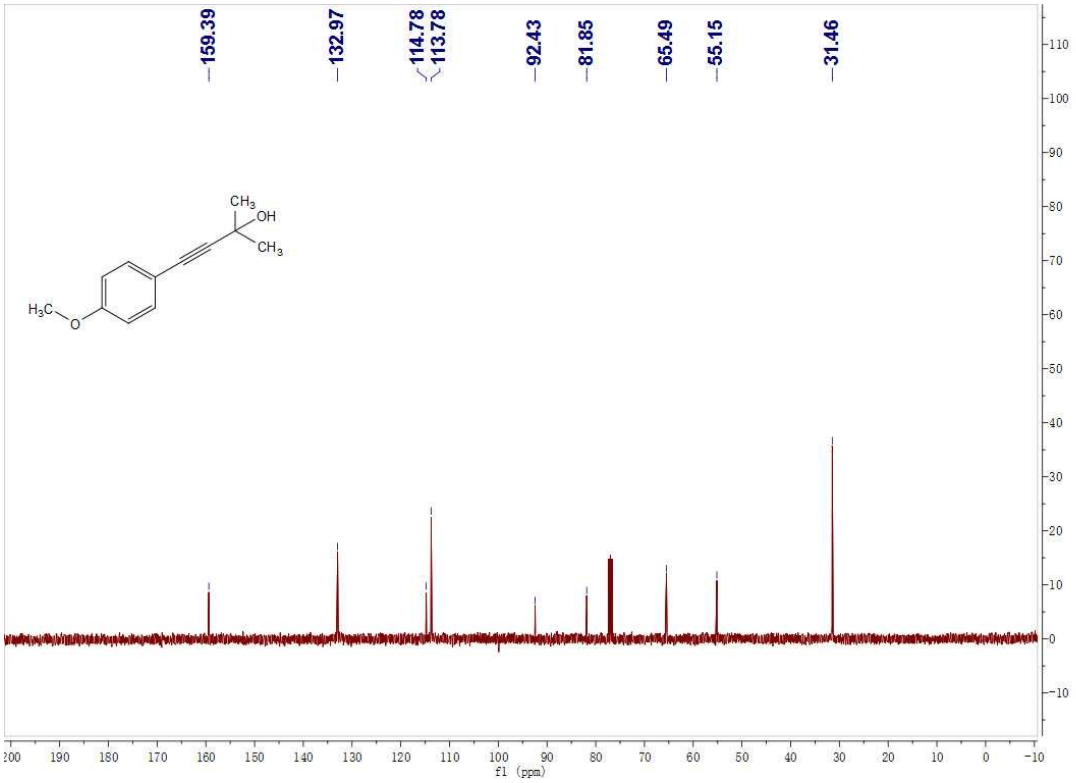
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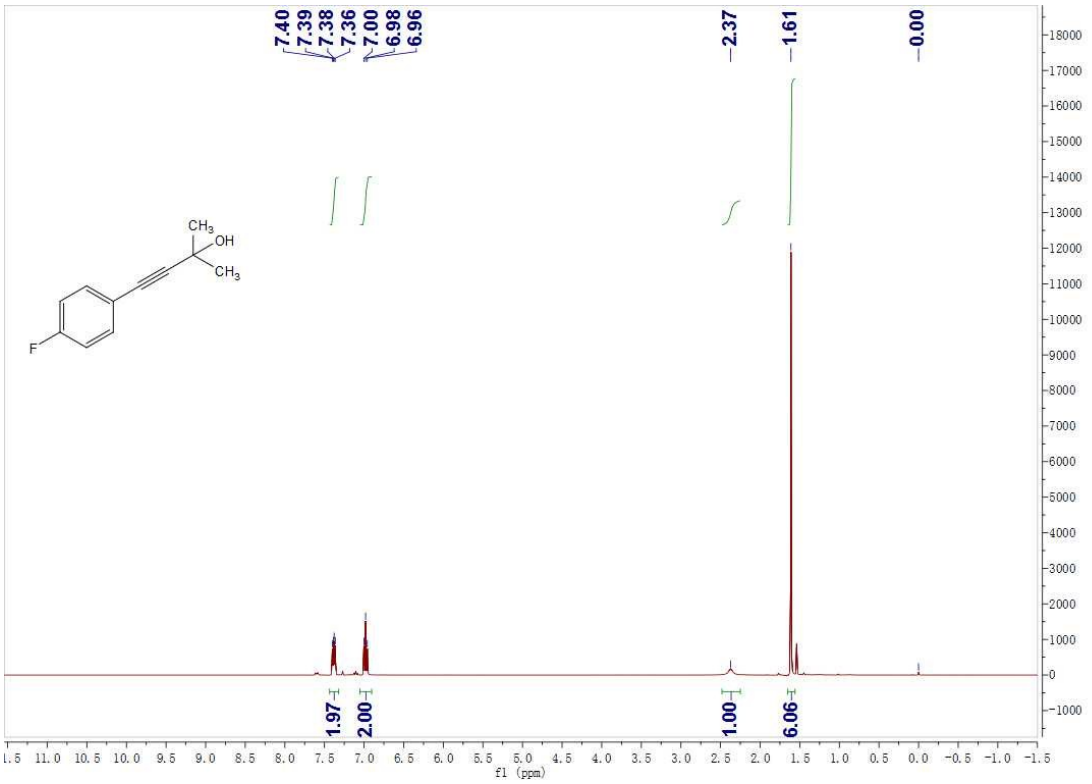
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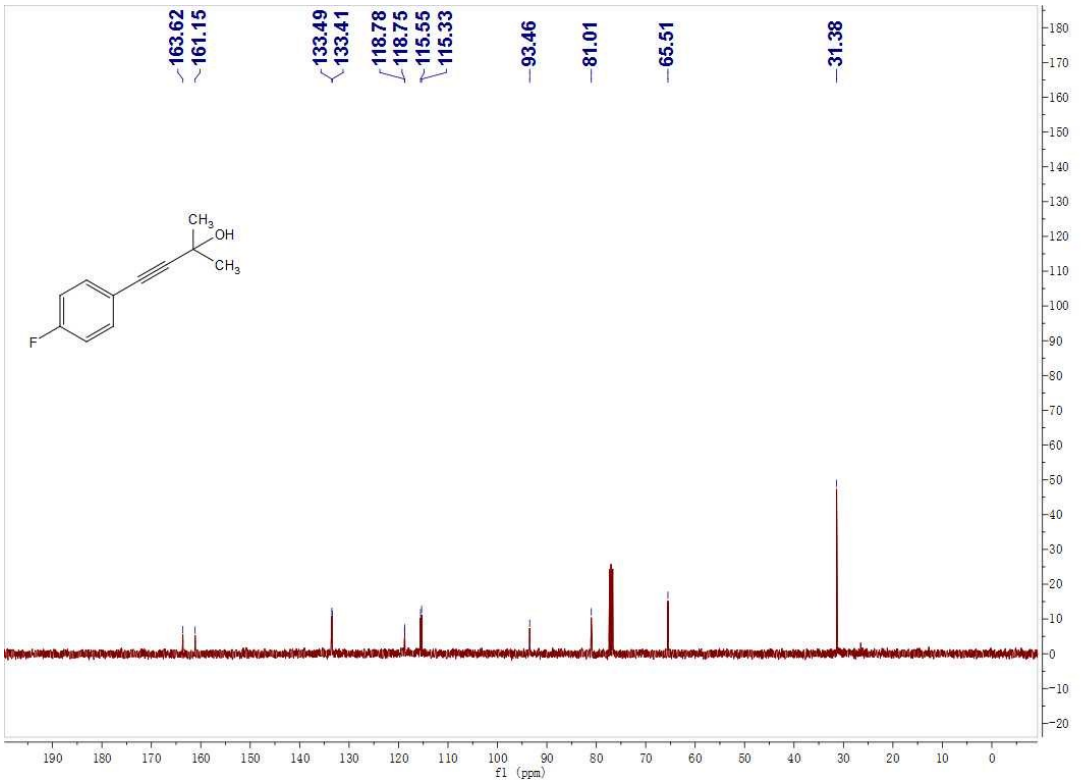
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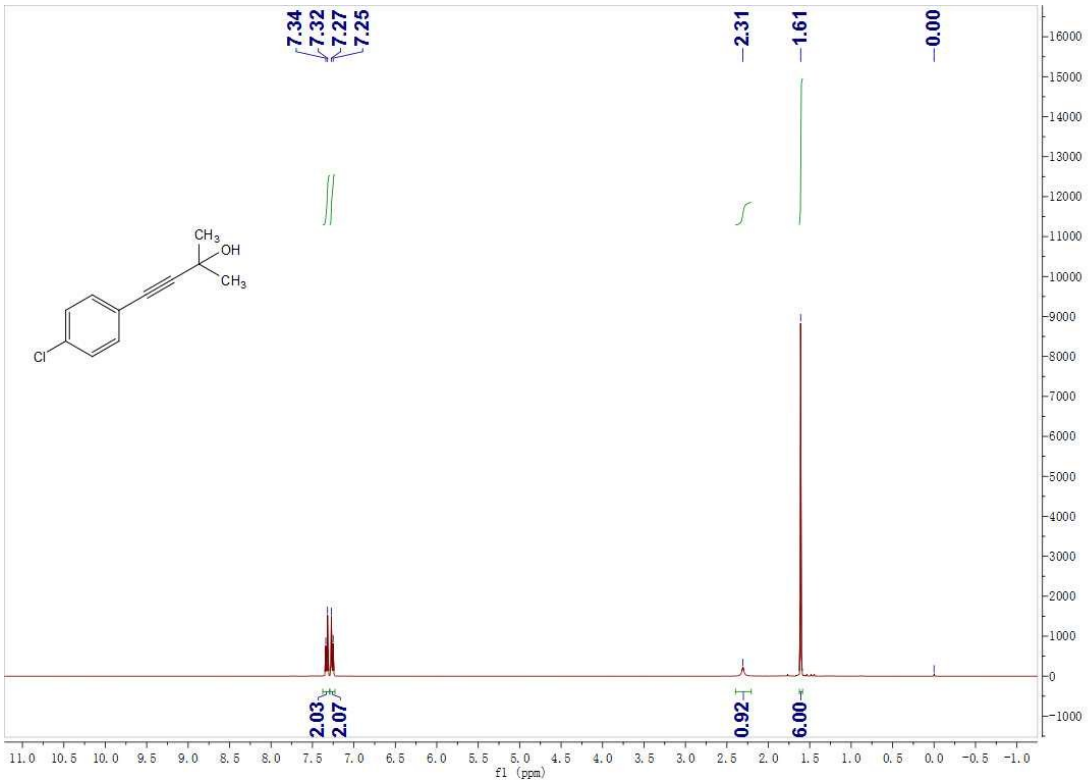
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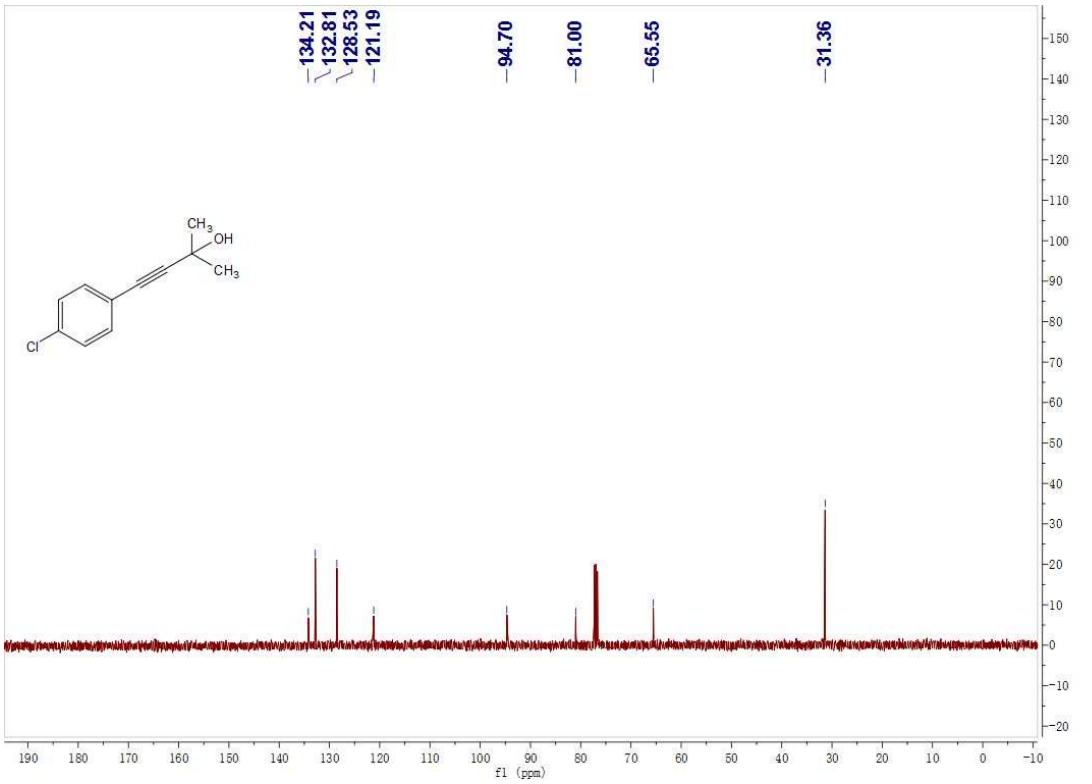
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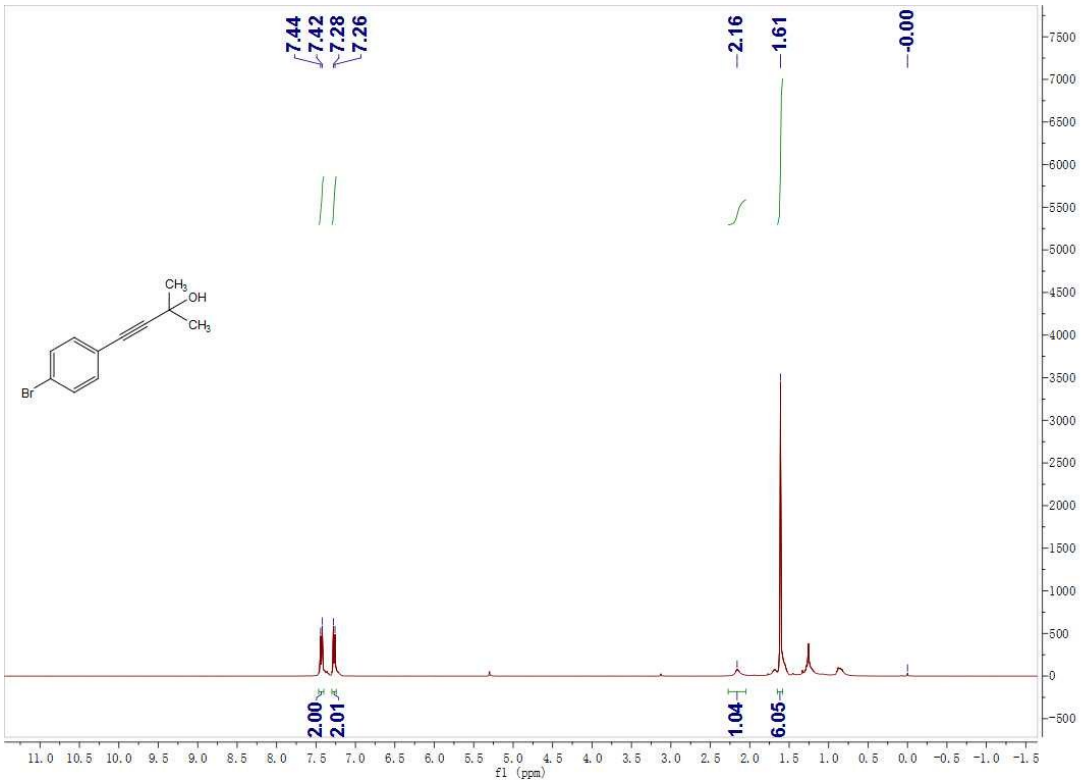
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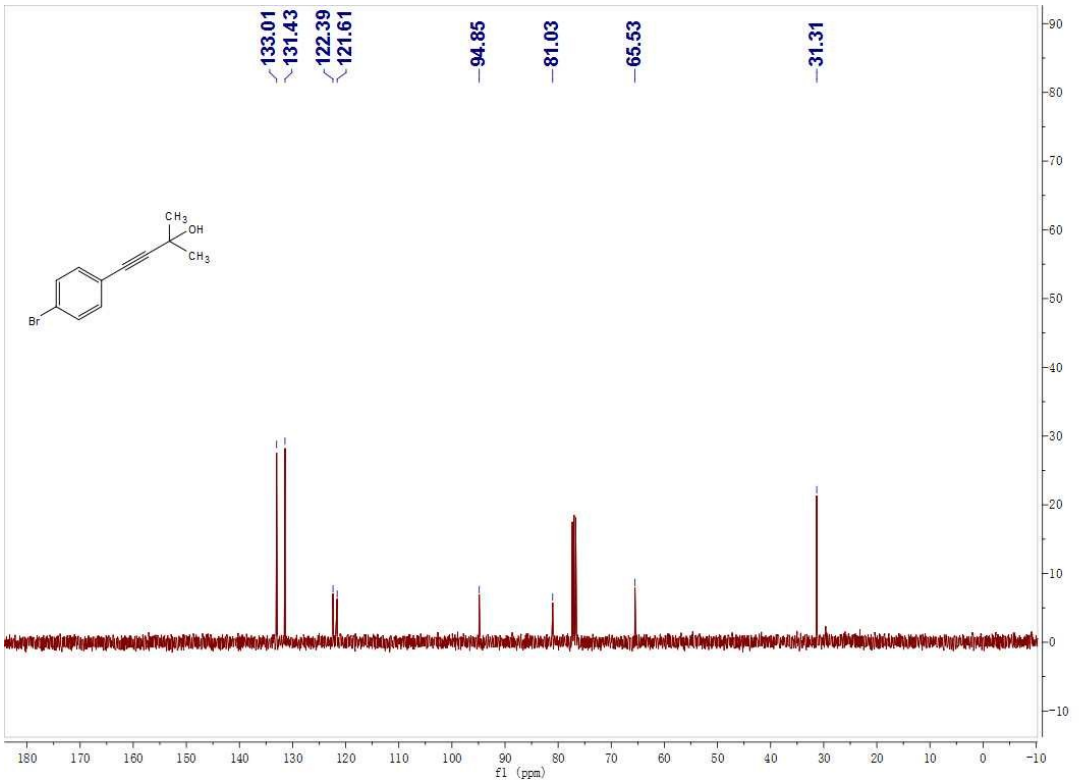
f1 (ppm)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Br
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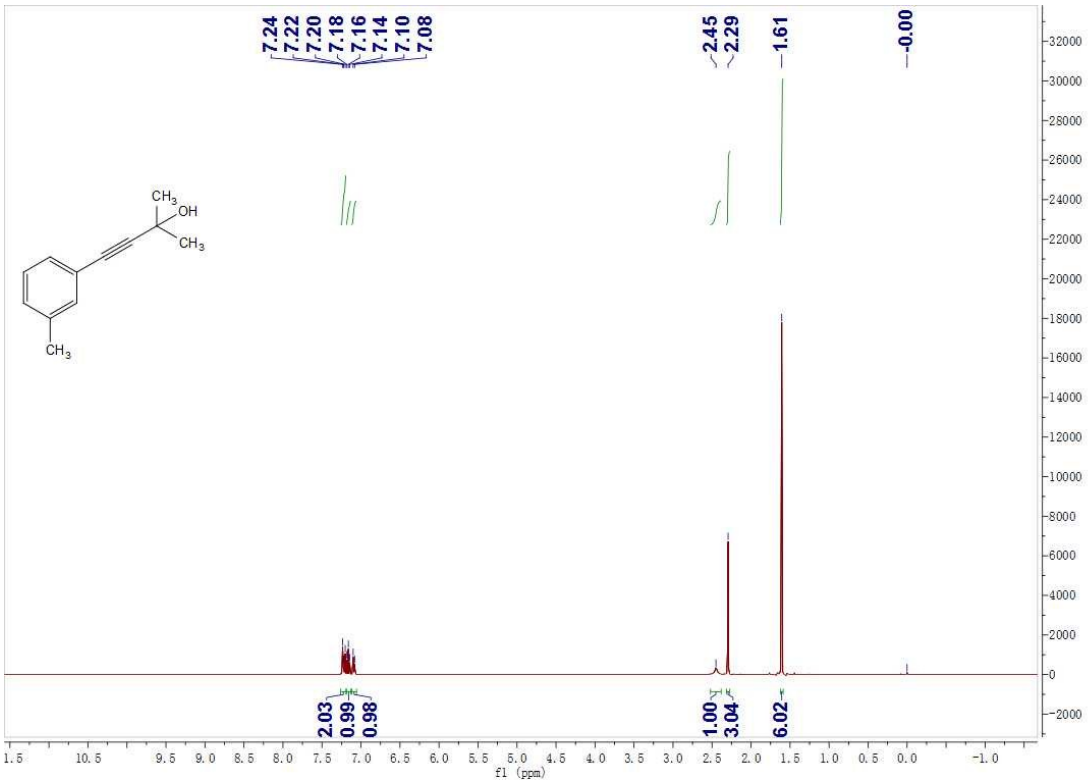
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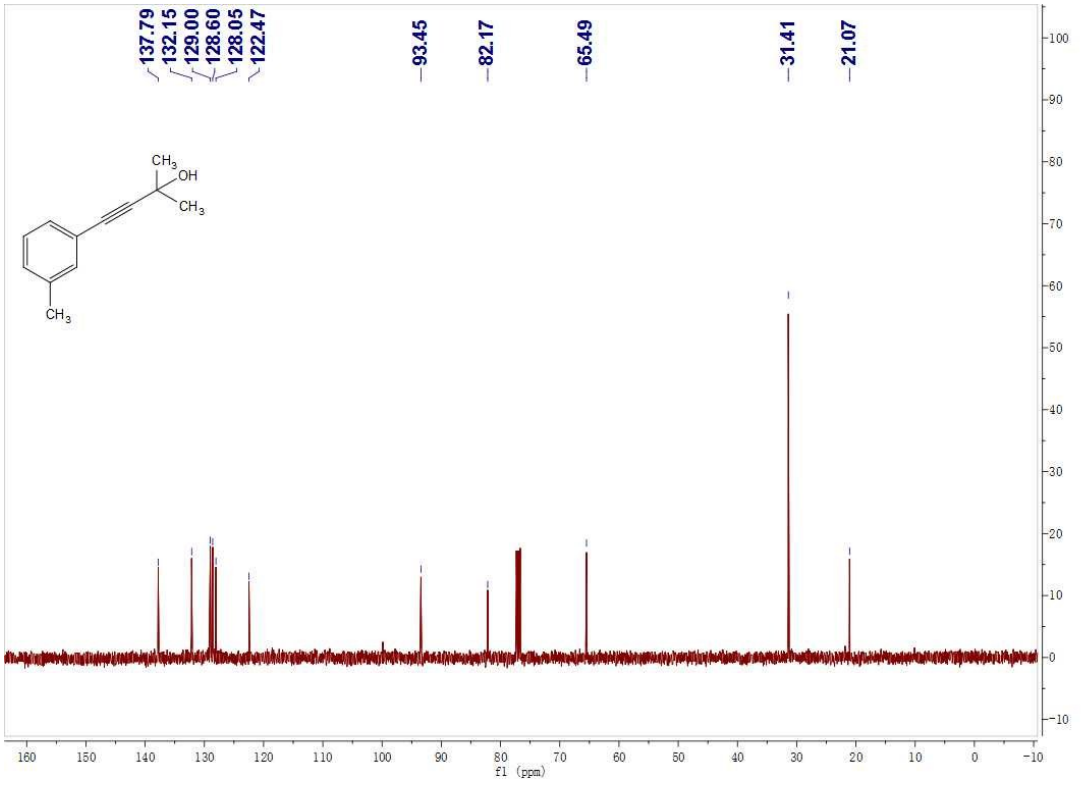
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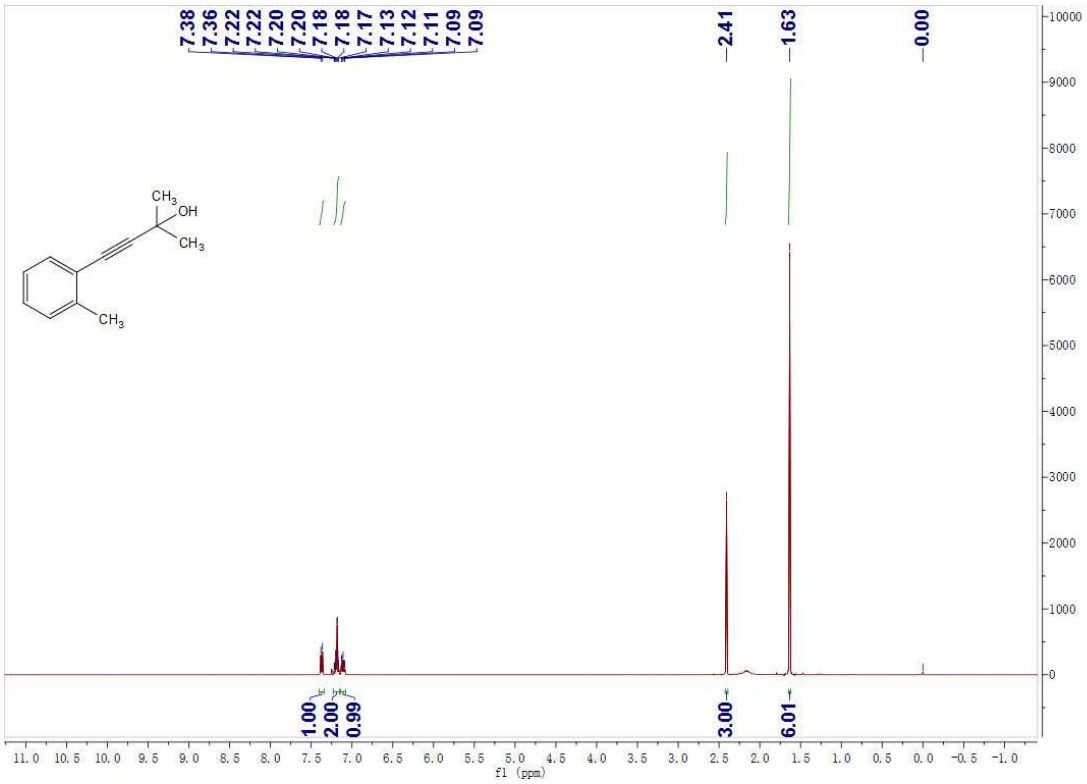
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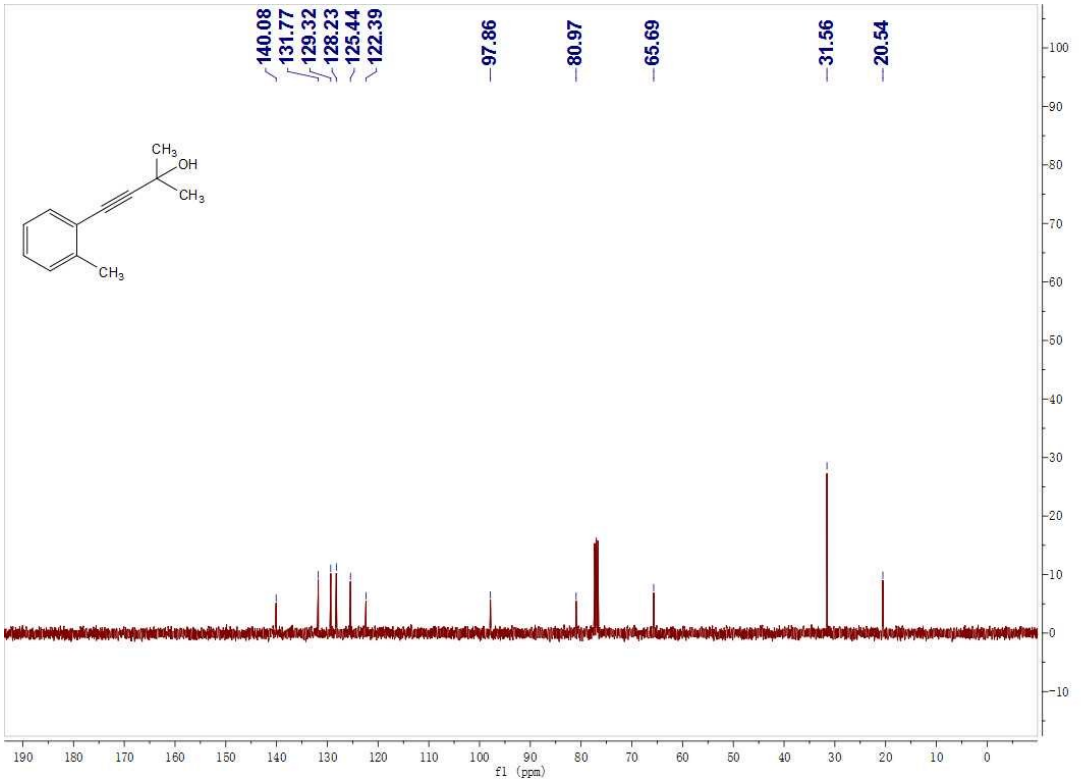
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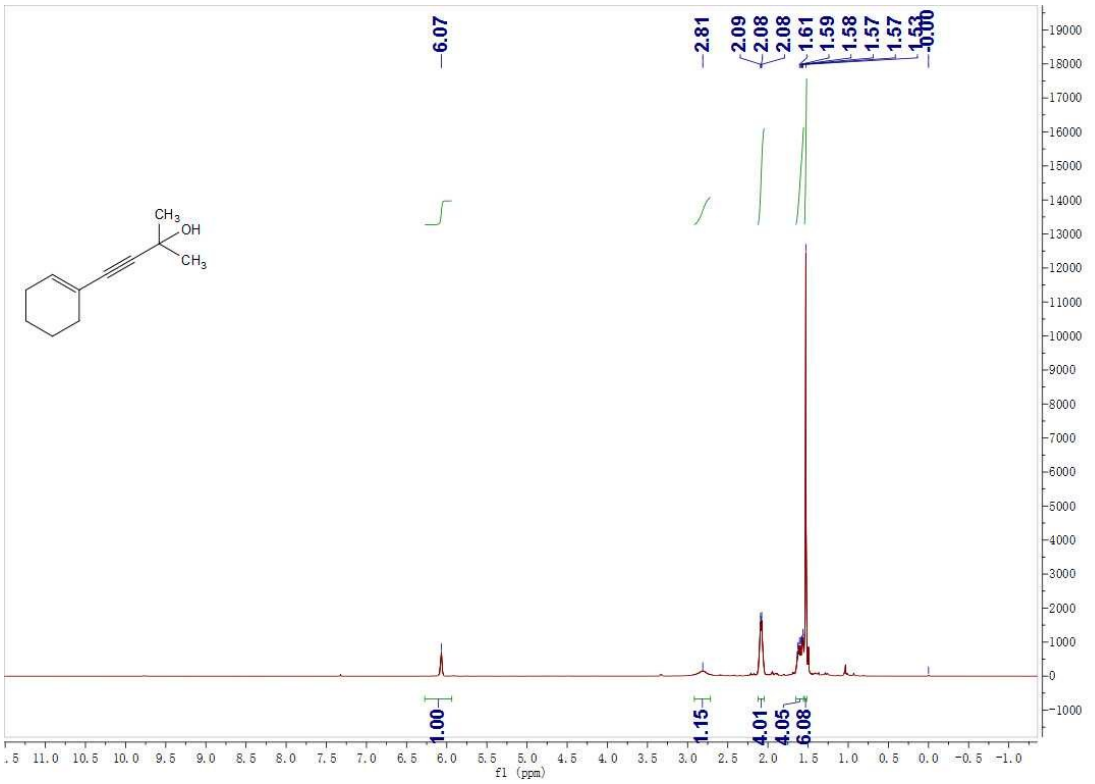
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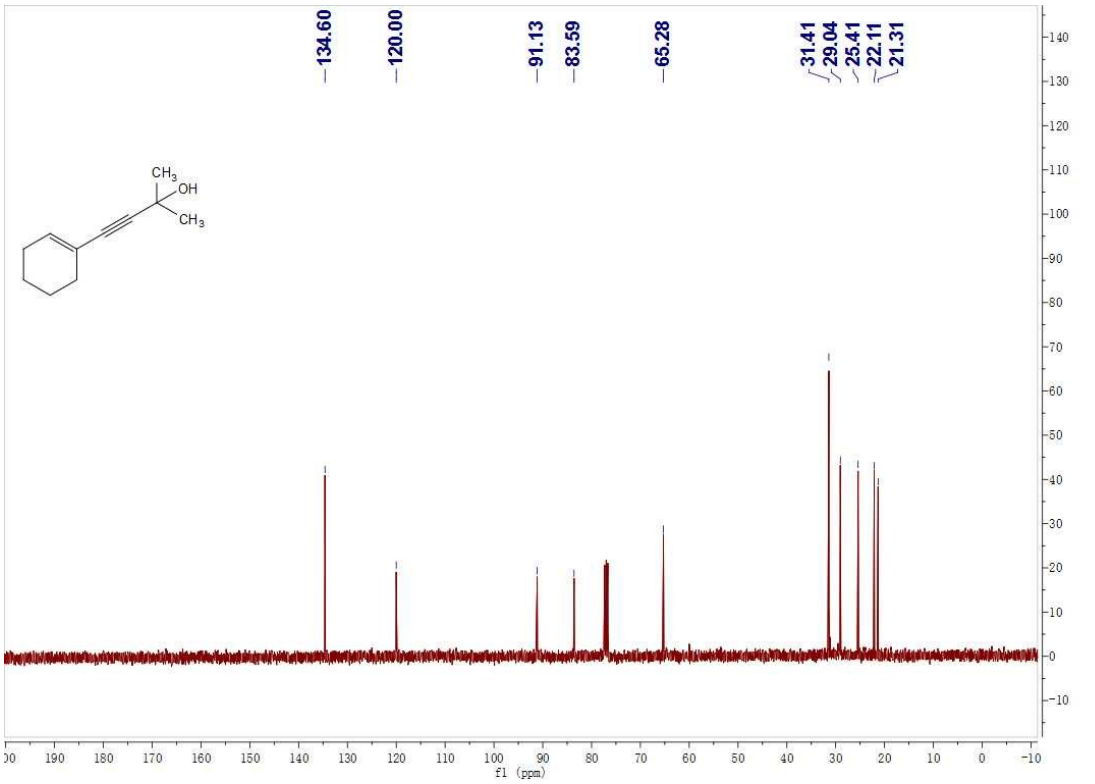
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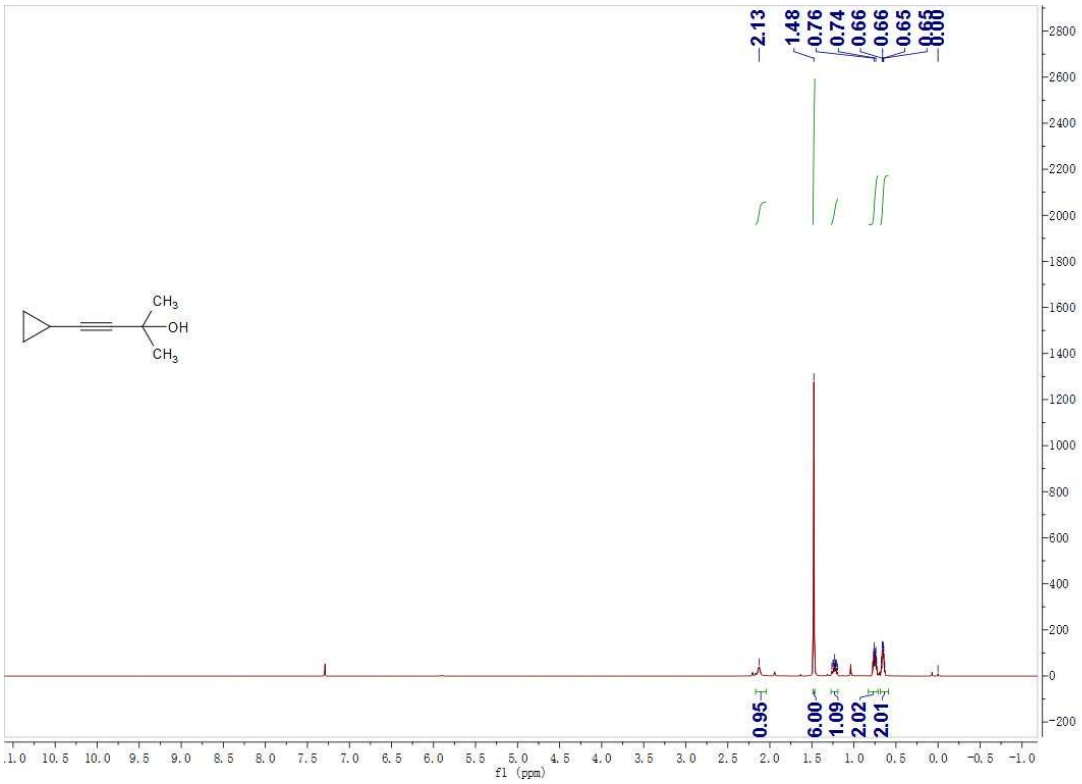
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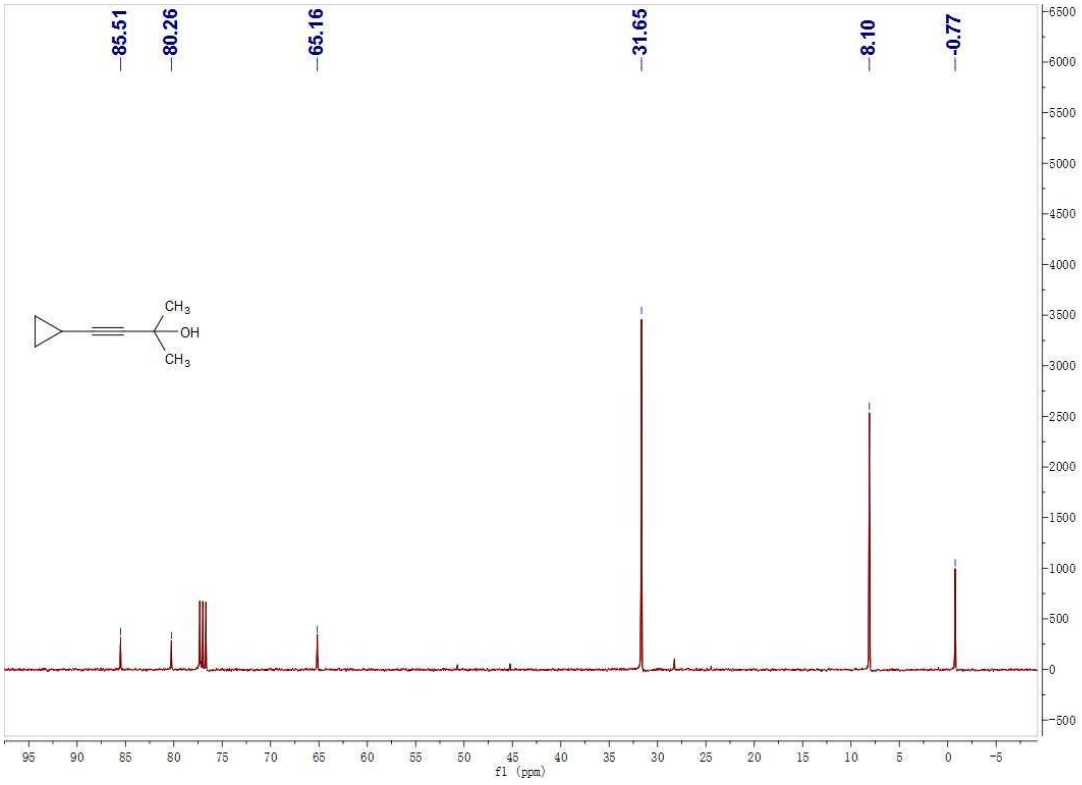
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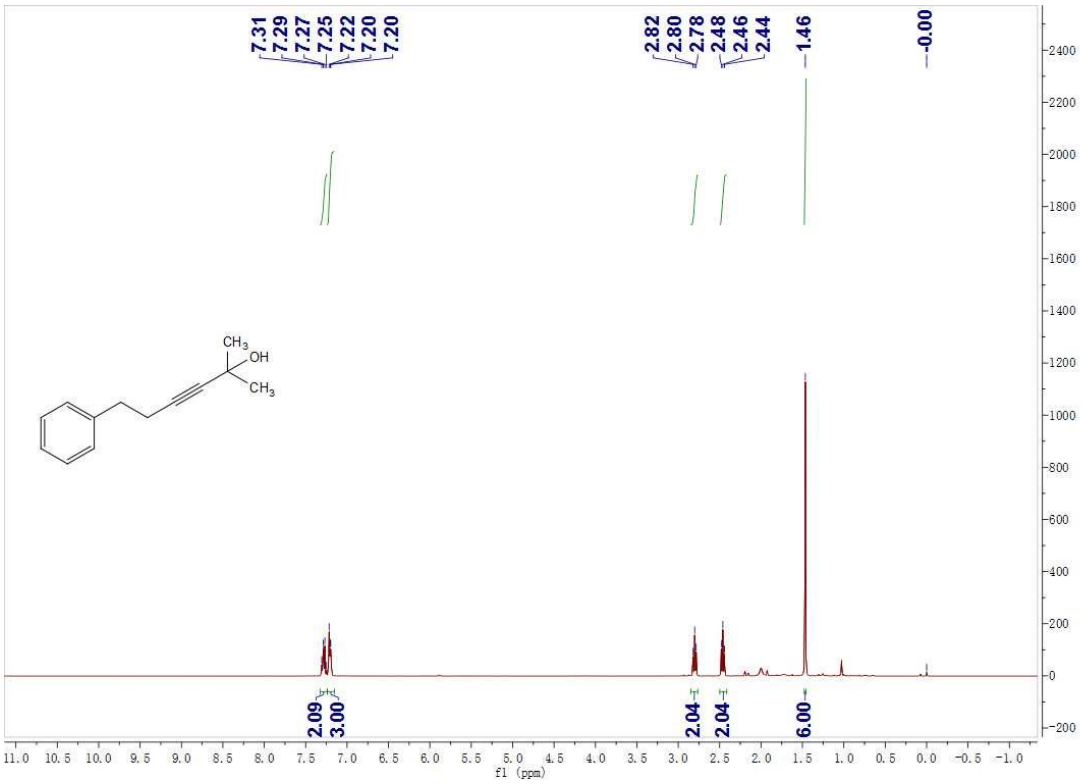
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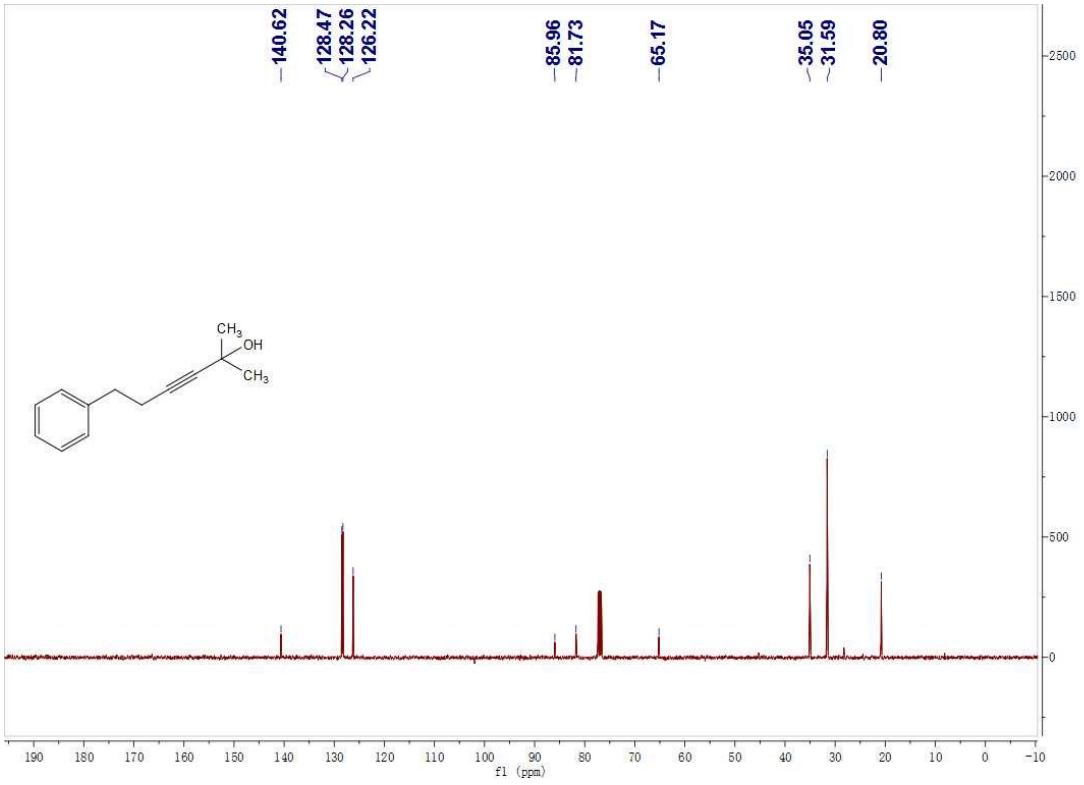
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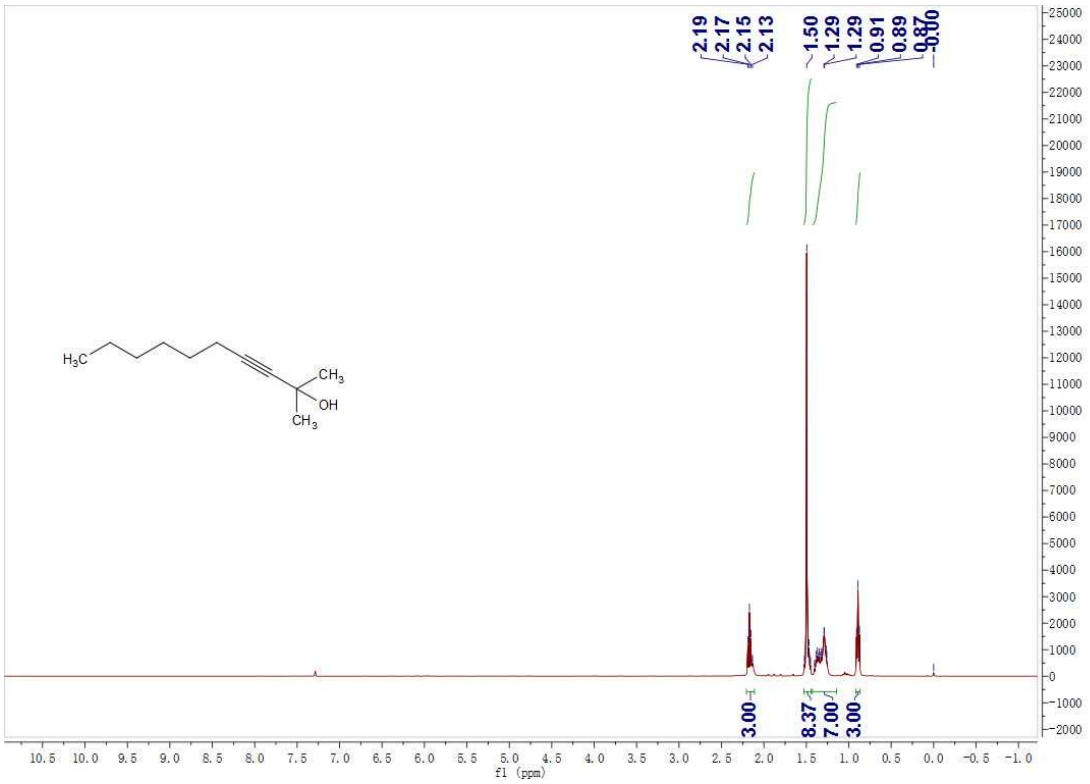
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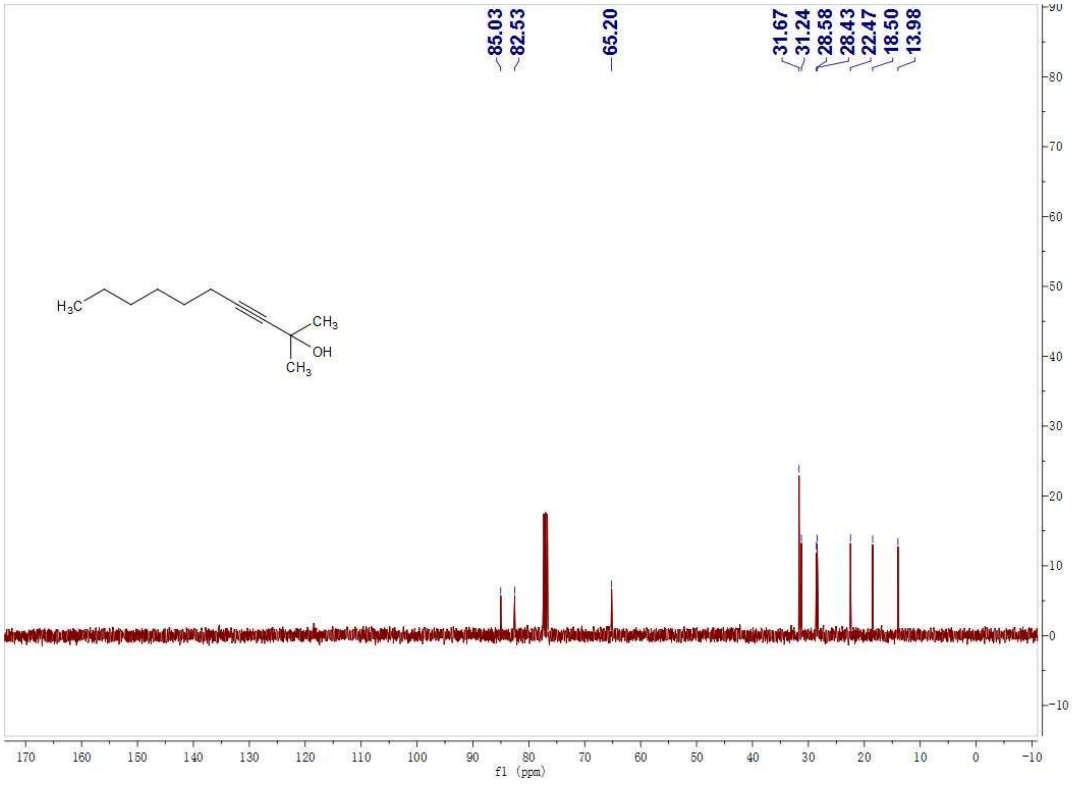
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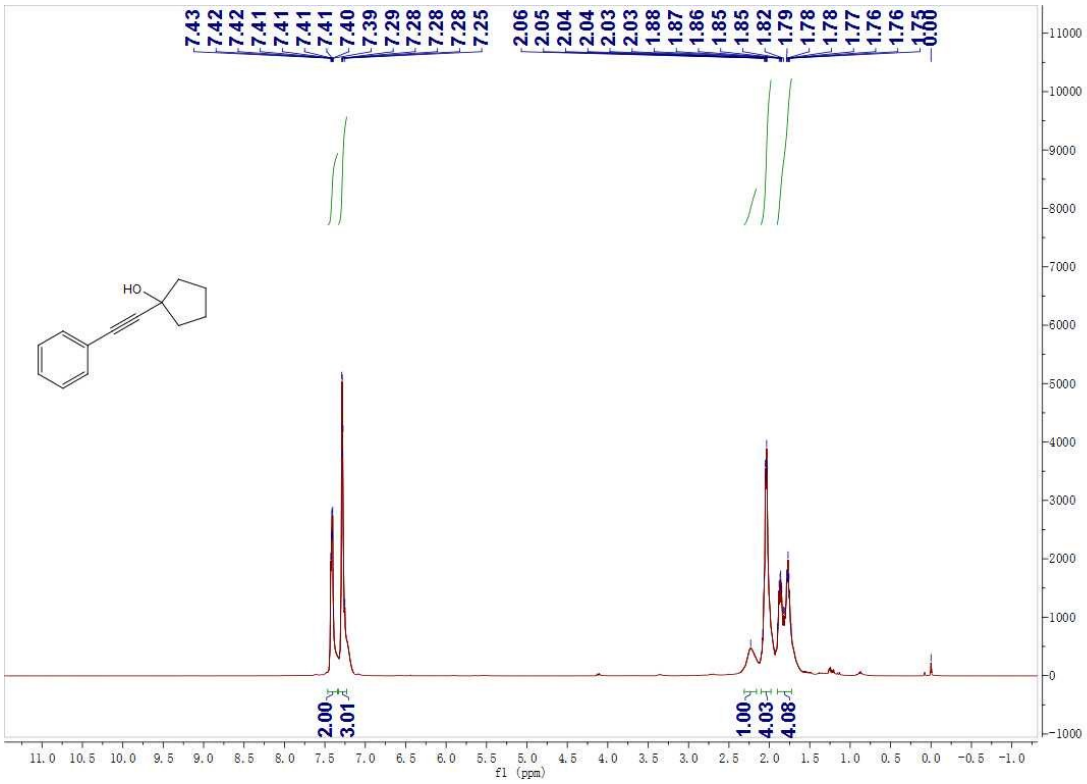
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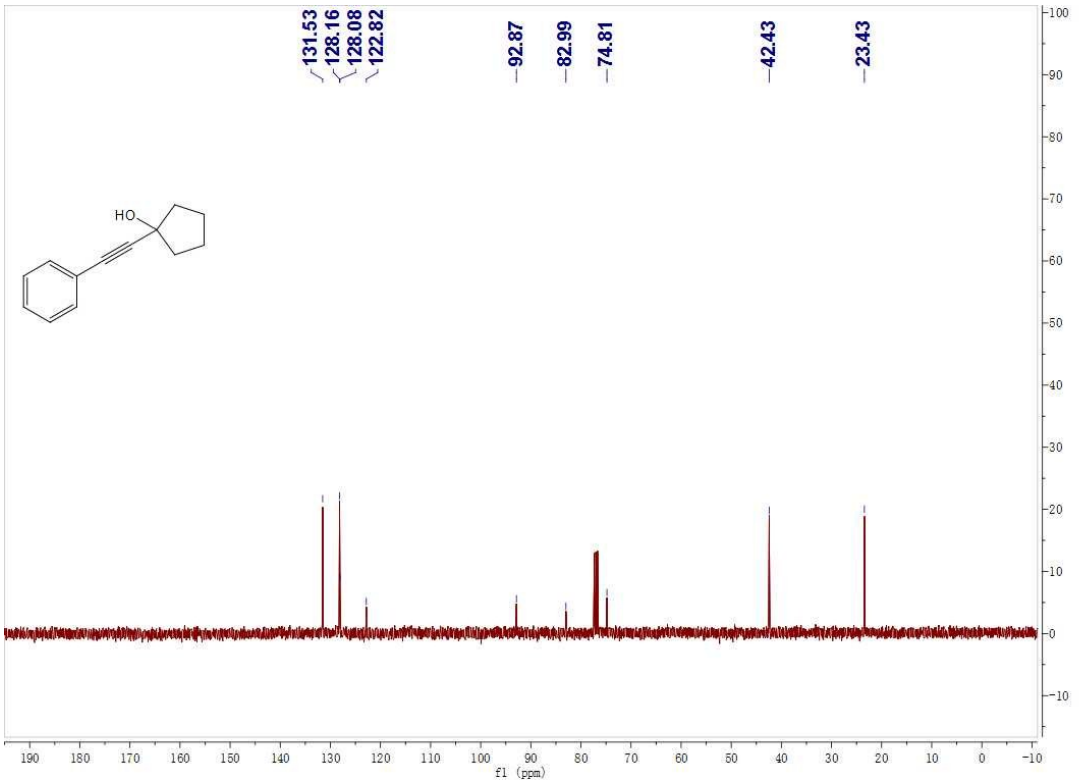
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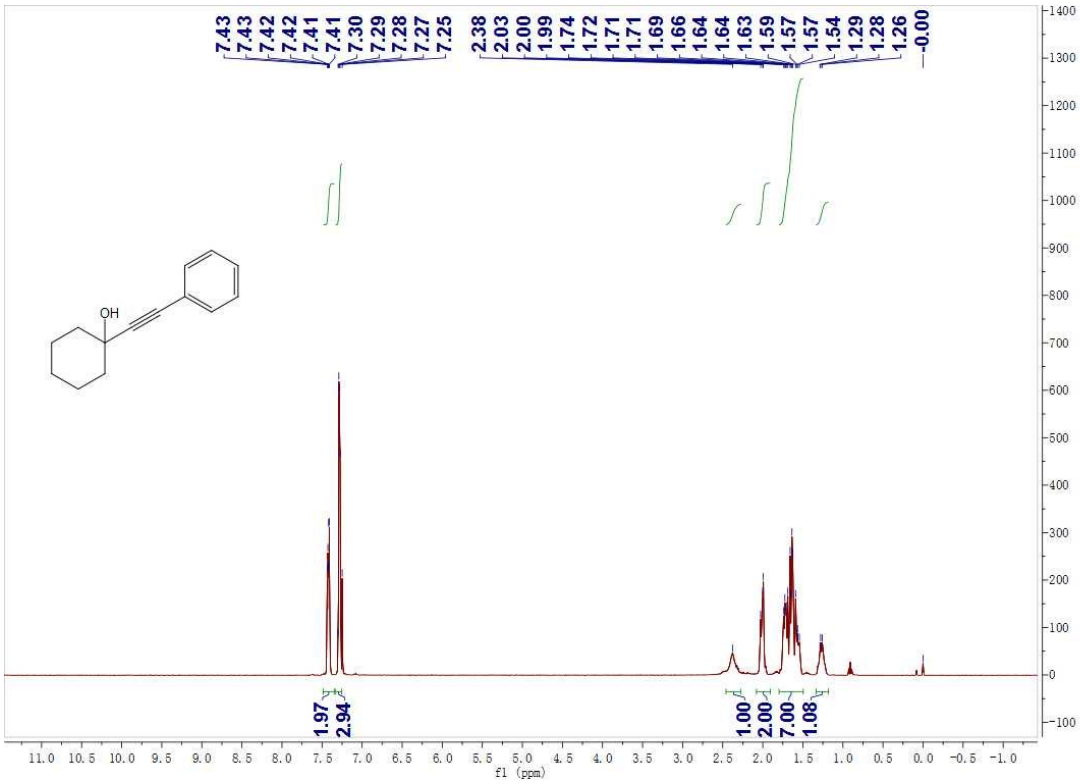
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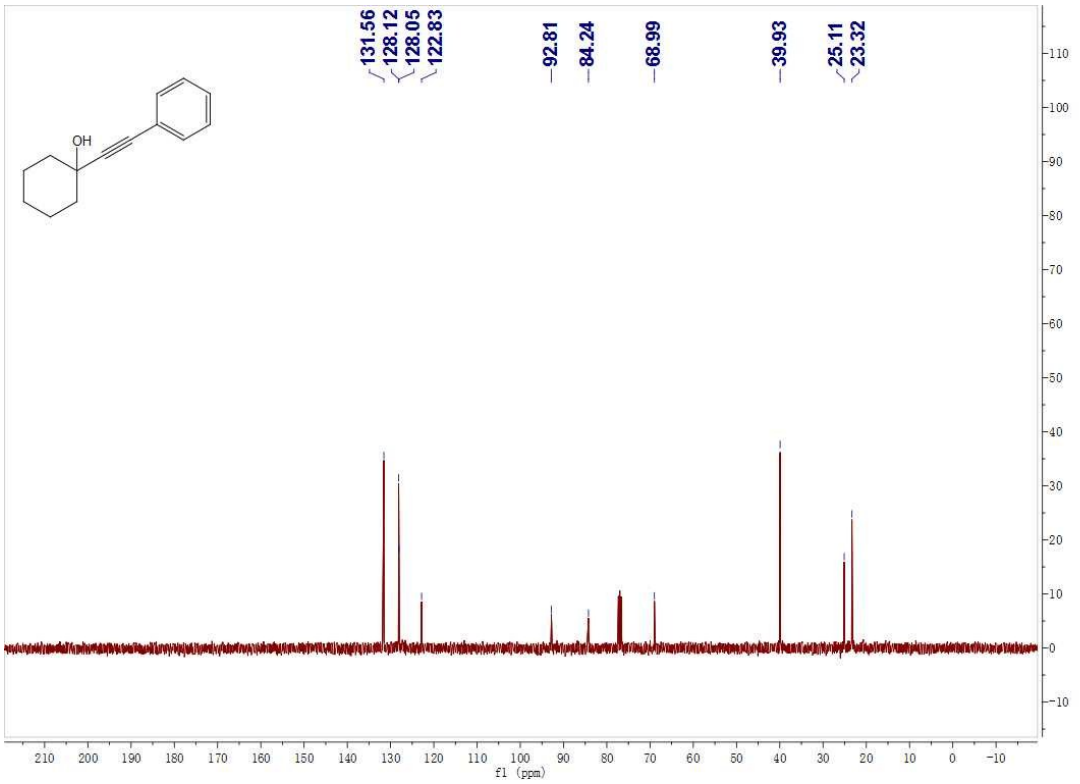
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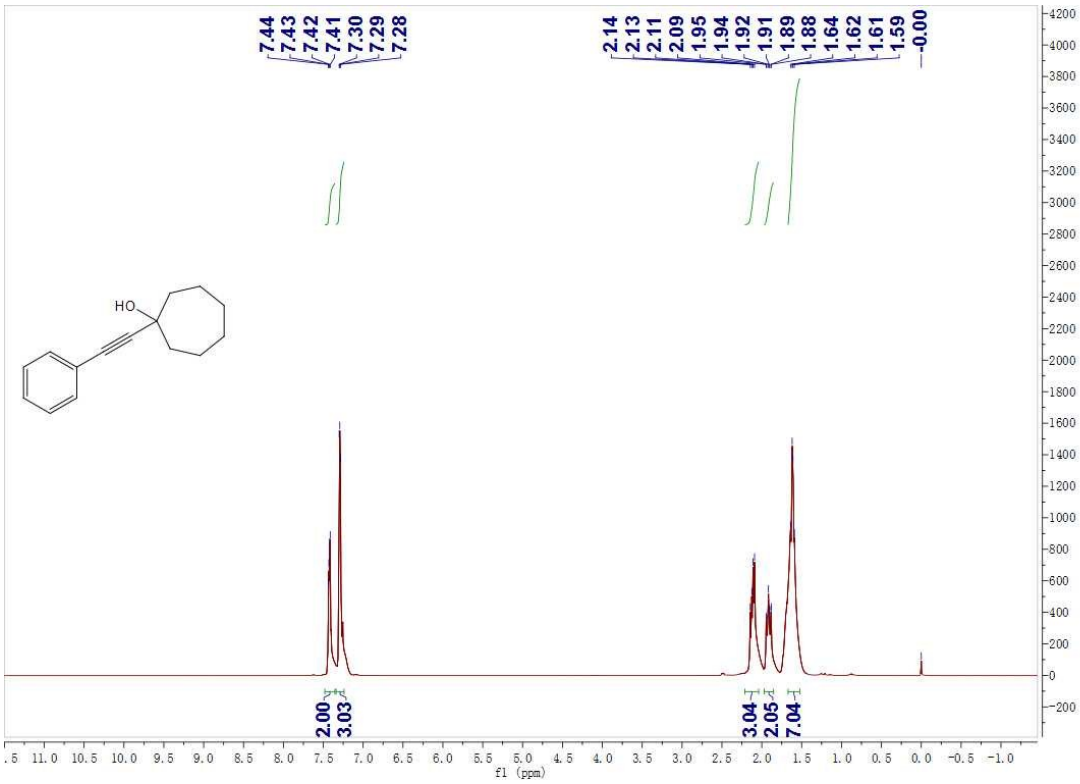
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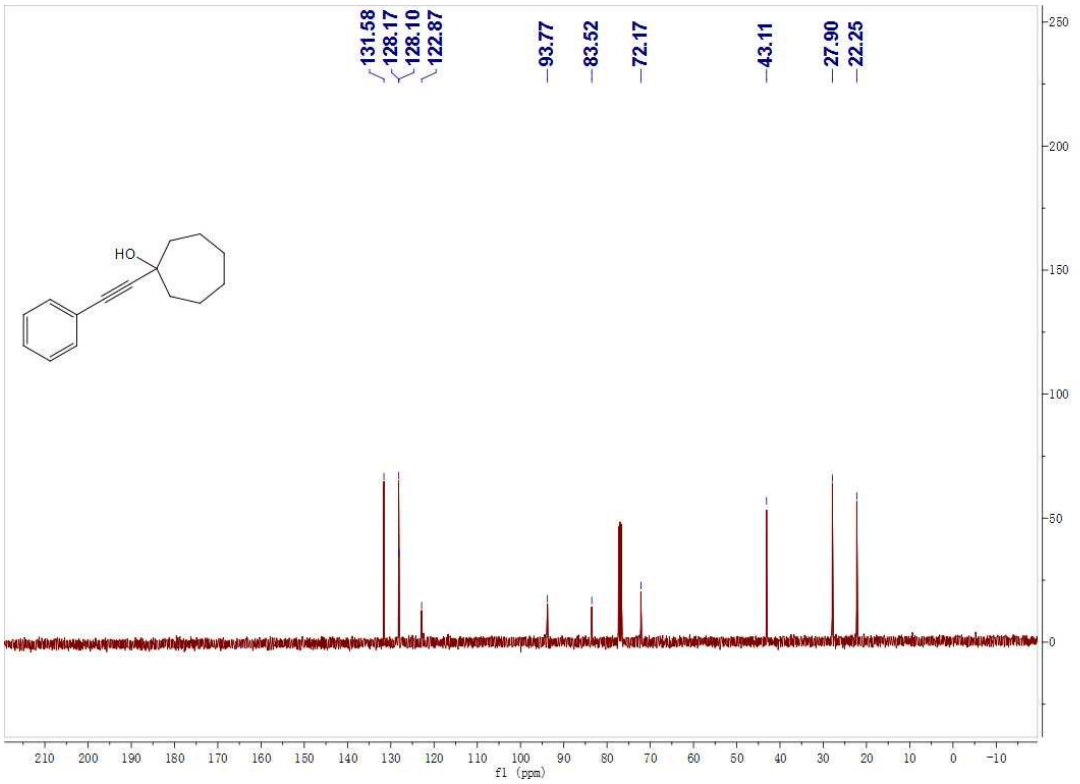
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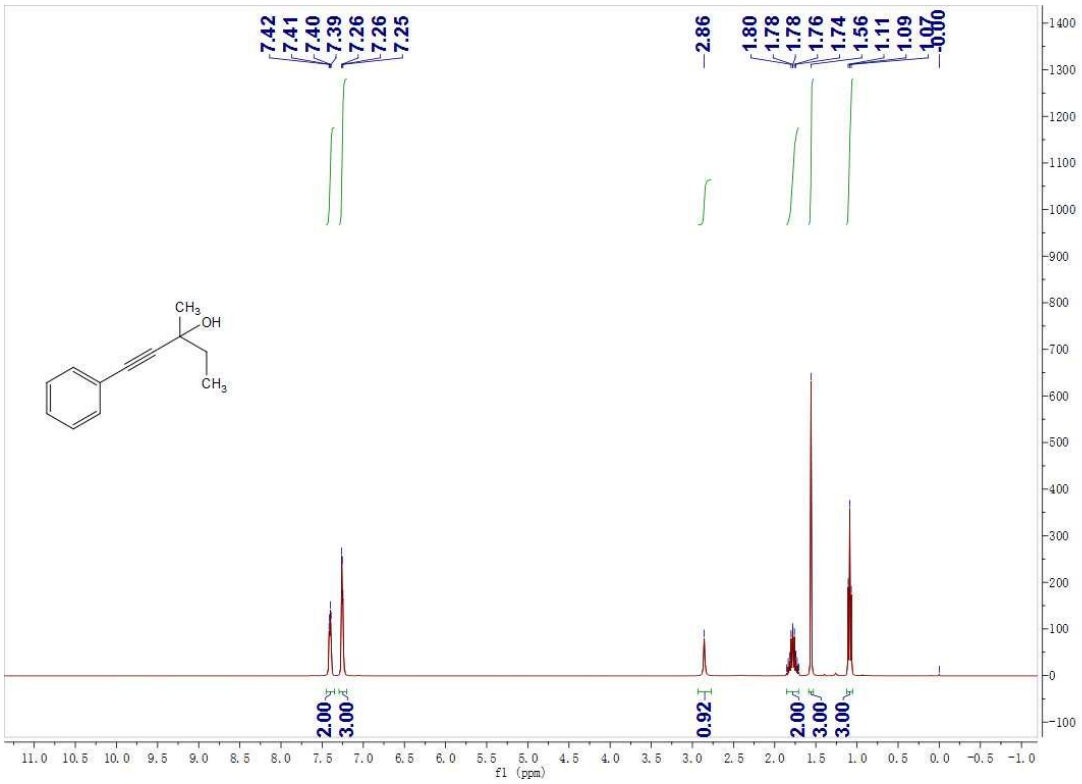
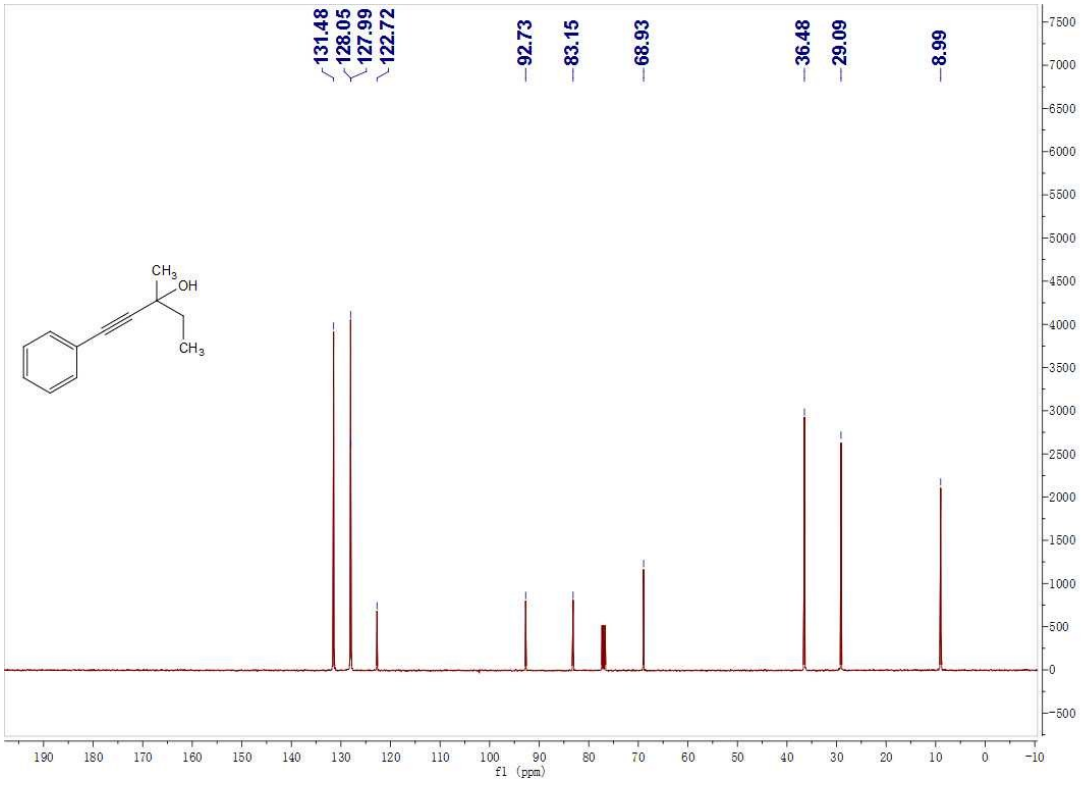
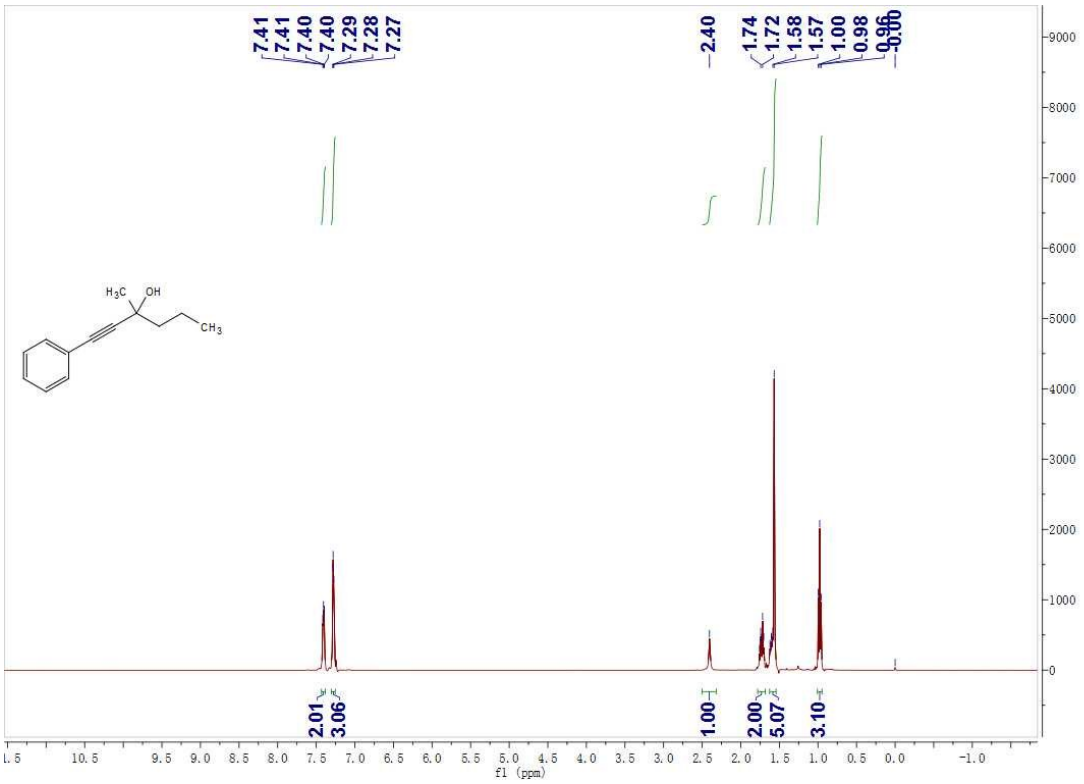
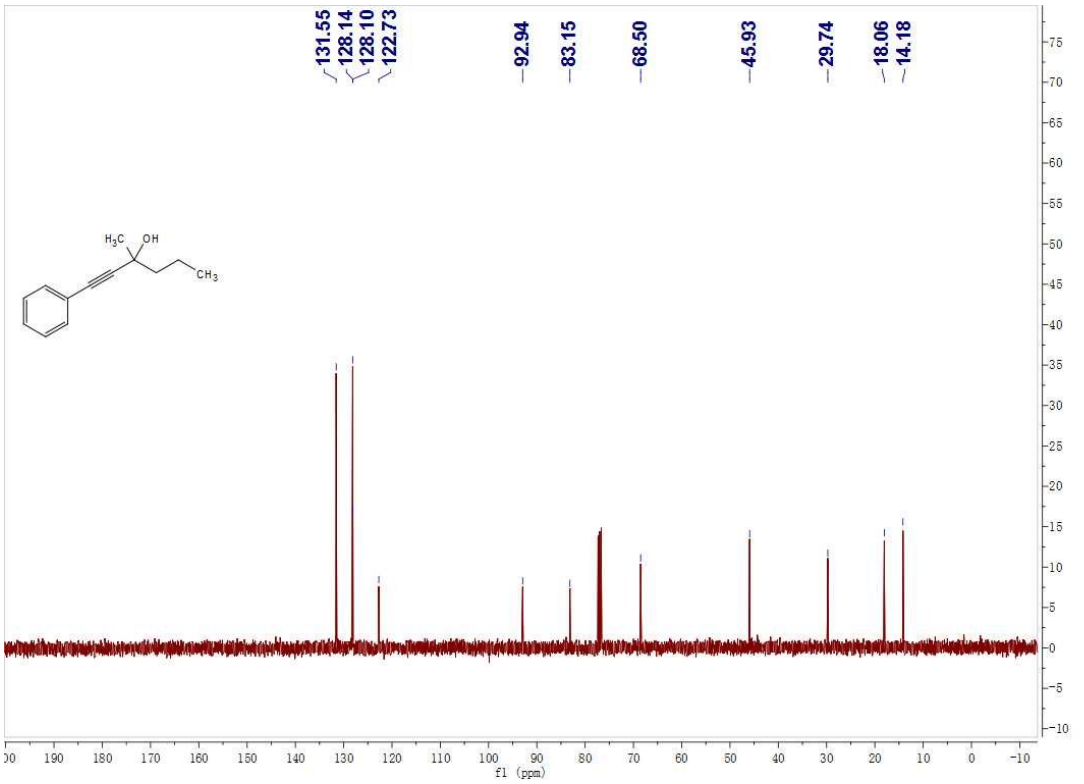
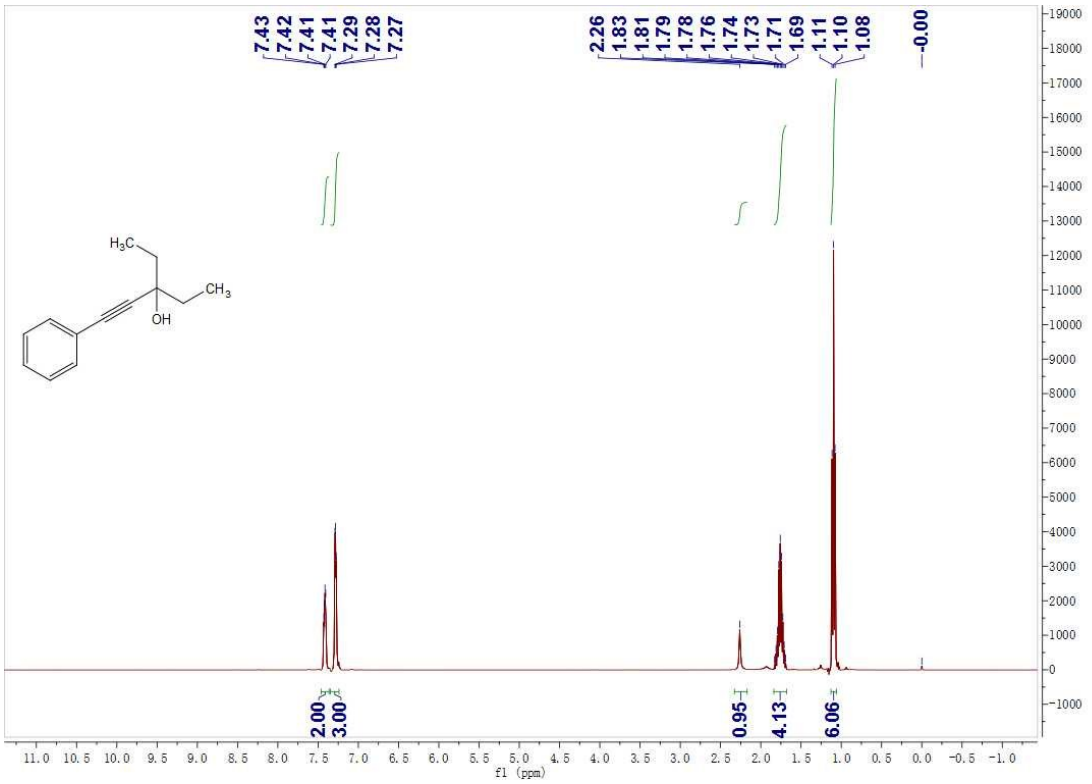
HO
f1 (ppm)

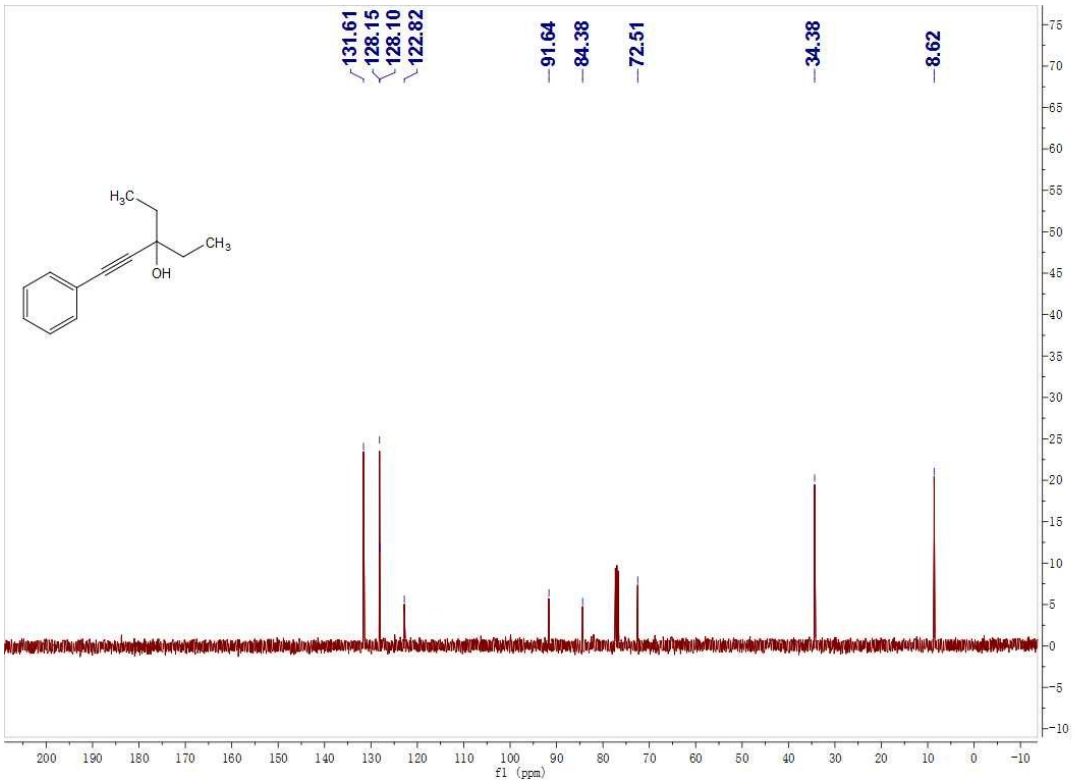

7.42
7.41
7.40
7.39
7.26
7.26
7.25
2.86
1.80
1.78
1.78
1.76
1.74
1.56
1.11
1.09
1.07
0.00
CH3
OH
CH3
2.00
3.00
0.92
2.00
3.00
3.00
f1 (ppm)

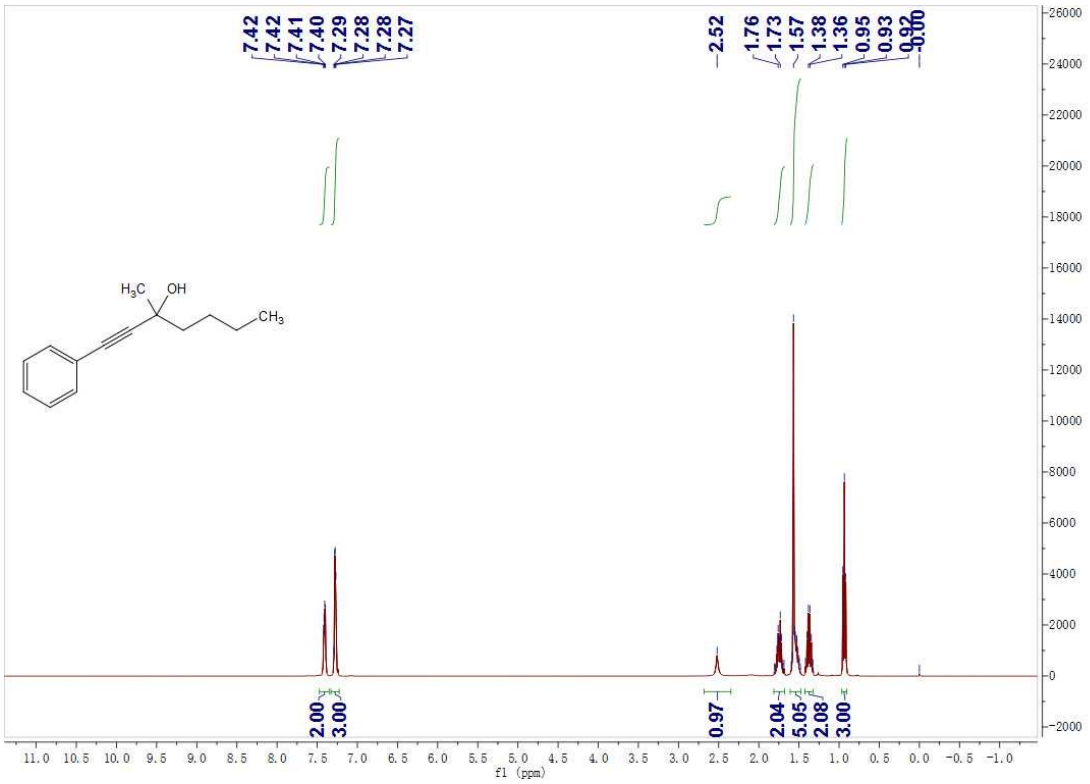

131.48
128.05
127.99
122.72
92.73
83.15
68.93
36.48
29.09
8.99
CH3
OH
CH3
f1 (ppm)

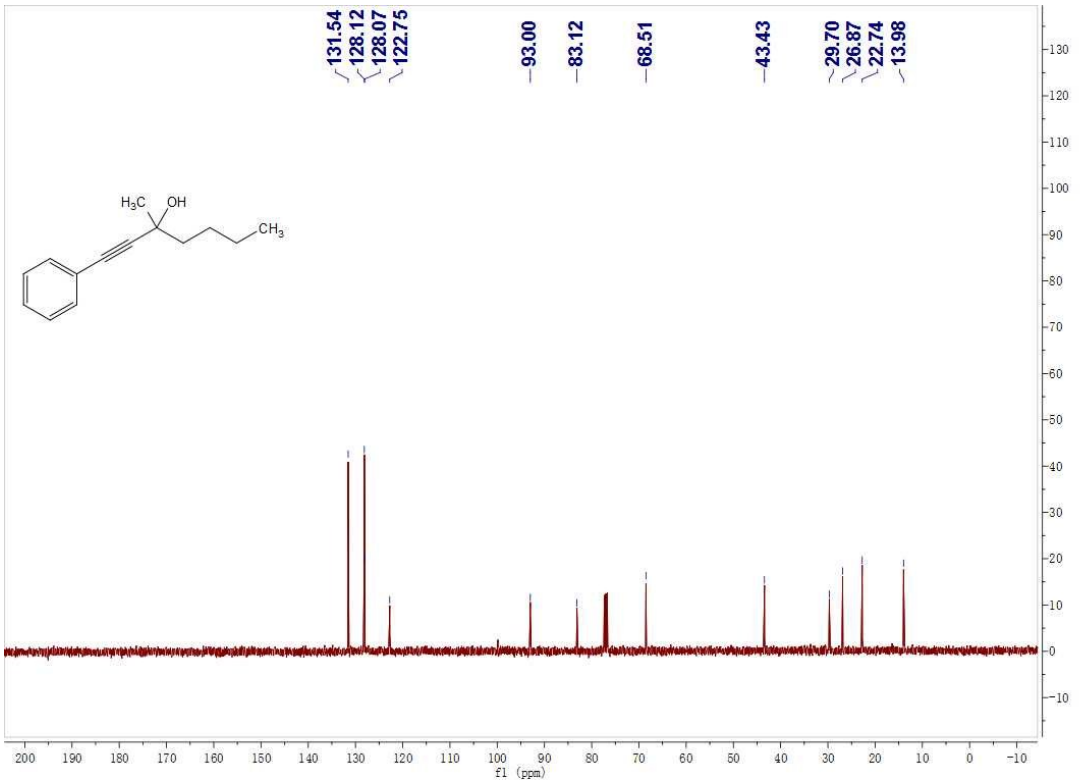

7.41
7.41
7.40
7.40
7.29
7.28
7.27
2.40
1.74
1.72
1.58
1.57
1.00
0.98
0.96
0.00
H3C
OH
CH3
2.01
3.06
1.00
2.00
5.07
3.10
f1 (ppm)

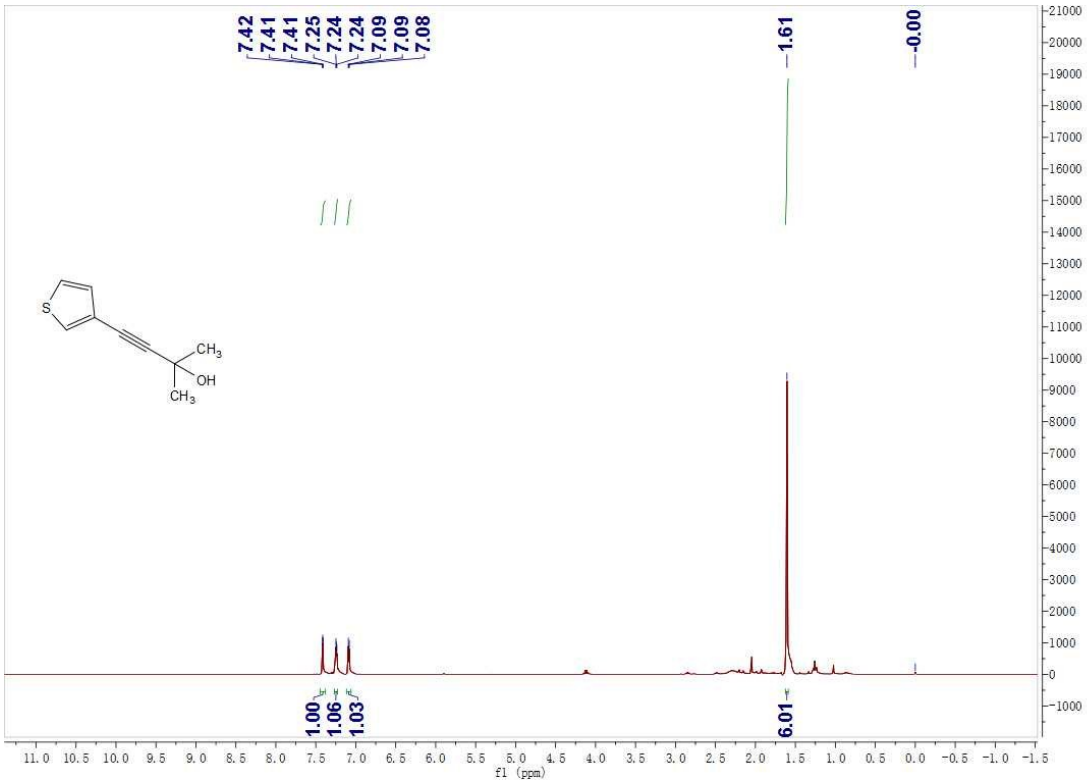

131.55
128.14
128.10
122.73
92.94
83.15
68.50
45.93
29.74
18.06
14.18
H3C
OH
CH3
f1 (ppm)

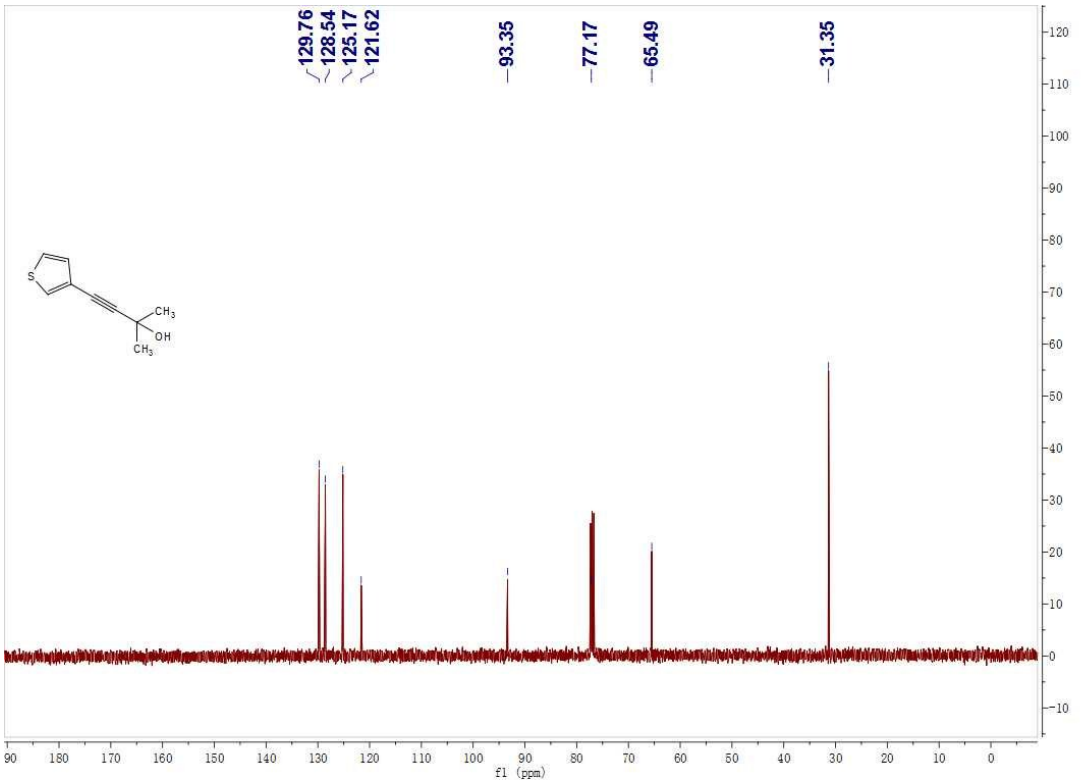

7.43
7.42
7.41
7.41
7.29
7.28
7.27
2.26
1.83
1.81
1.79
1.78
1.76
1.74
1.73
1.71
1.69
1.11
1.10
1.08
-0.00
H3C
CH3
OH
2.00
3.00
0.95
4.13
6.06
f1 (ppm)

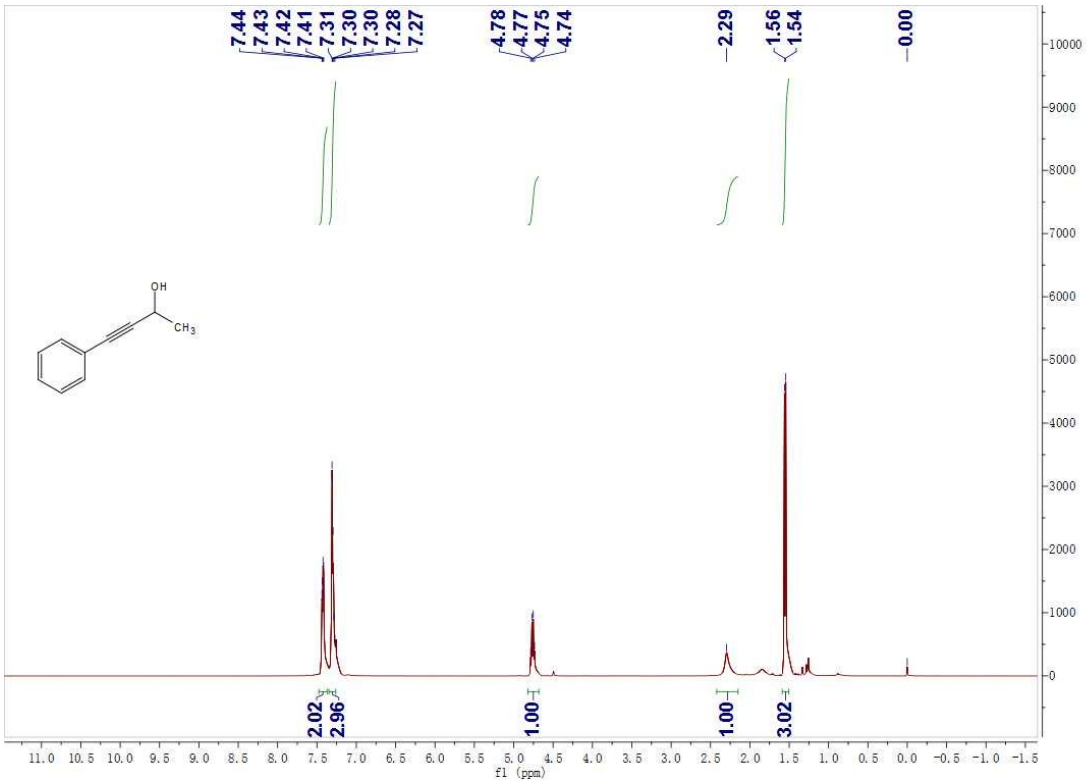

131.61
128.15
128.10
122.82
91.64
84.38
72.51
34.38
8.62
H3C
CH3
OH
f1 (ppm)

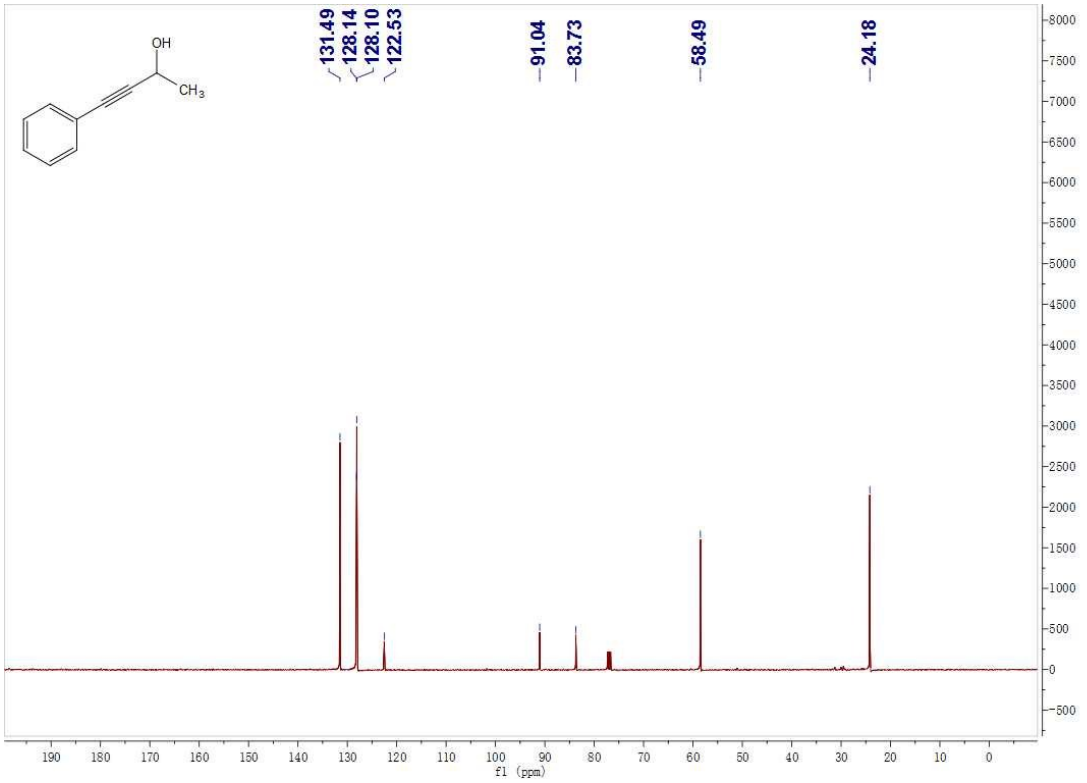

7.42
7.42
7.41
7.40
7.29
7.28
7.28
7.27
2.52
1.76
1.73
1.57
1.38
1.36
0.95
0.93
0.92
0.00
H3C
OH
CH3
2.00
3.00
0.97
2.04
5.05
2.08
3.00
f1 (ppm)


131.54
128.12
128.07
122.75
93.00
83.12
68.51
43.43
29.70
26.87
22.74
13.98
H3C
OH
CH3
f1 (ppm)

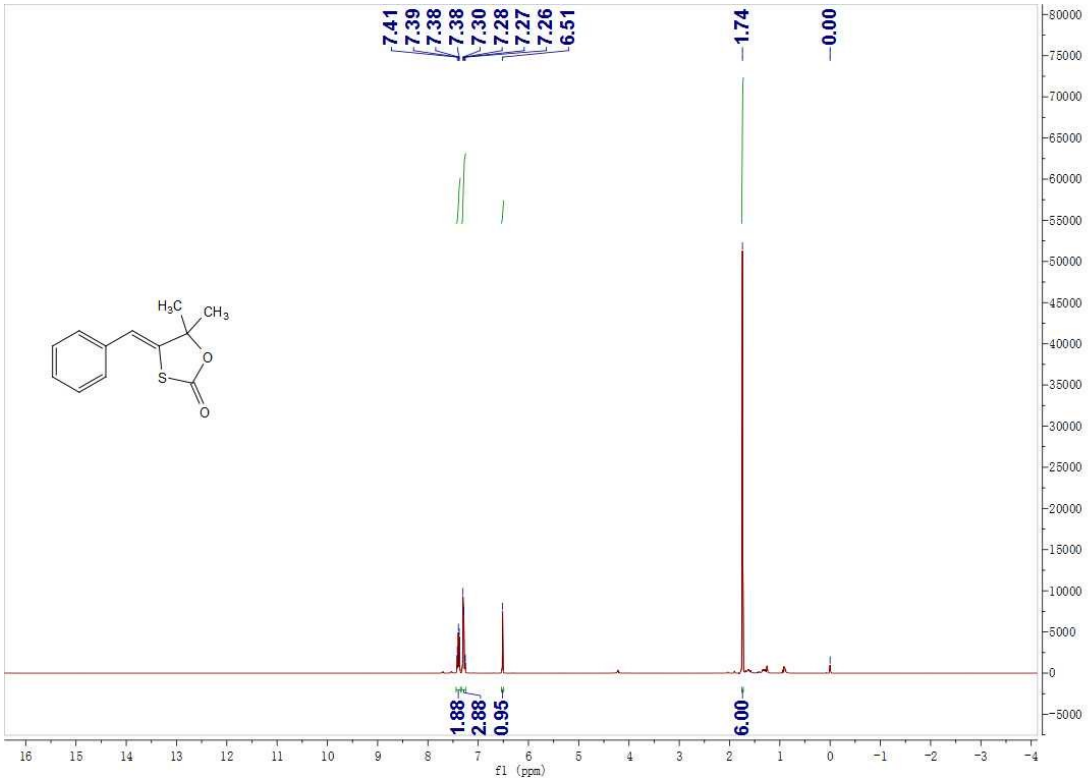

7.42
7.41
7.41
7.25
7.24
7.24
7.09
7.09
7.08
1.61
-0.00
1.00
1.06
1.03
6.01
f1 (ppm)

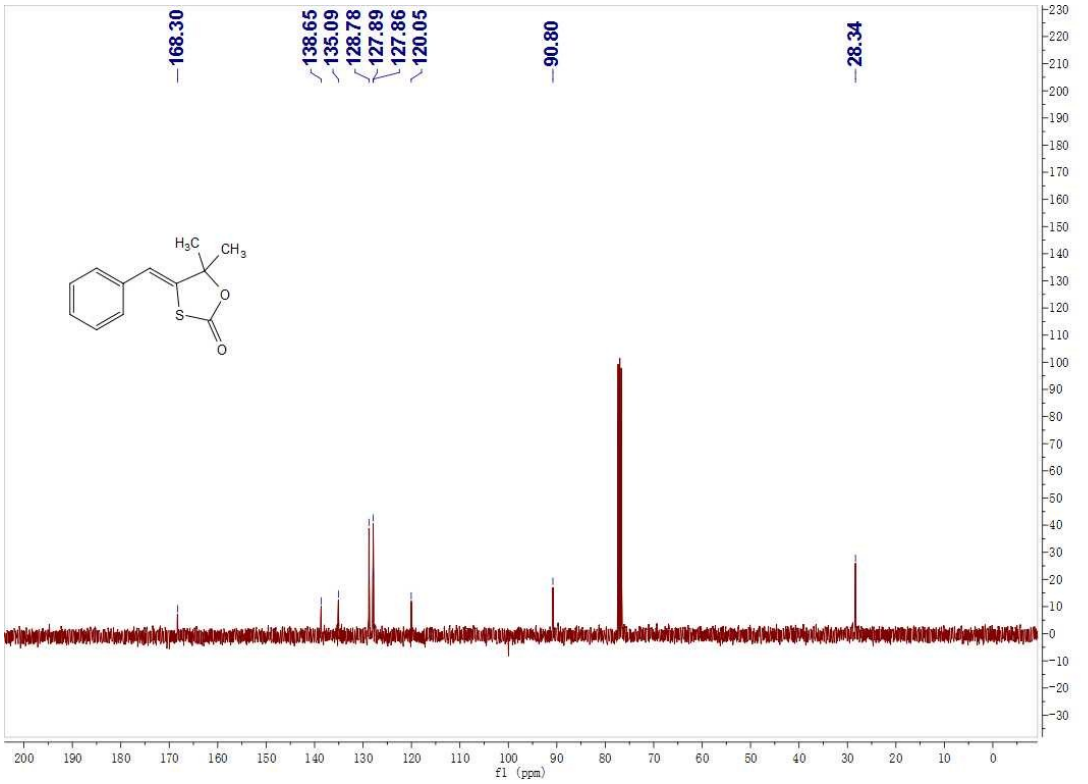

129.76
128.54
125.17
121.62
93.35
77.17
65.49
31.35
f1 (ppm)

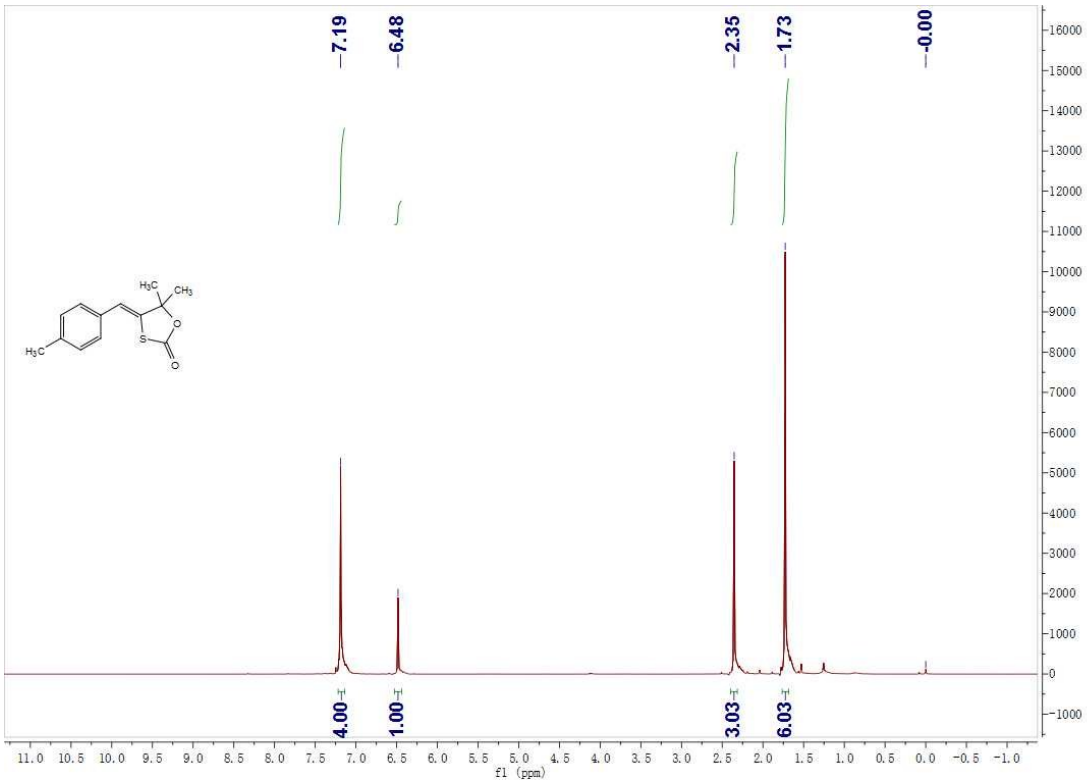

7.44
7.43
7.42
7.41
7.31
7.30
7.30
7.28
7.27
4.78
4.77
4.75
4.74
2.29
1.56
1.54
0.00
OH
CH3
2.02
2.96
1.00
1.00
3.02
f1 (ppm)

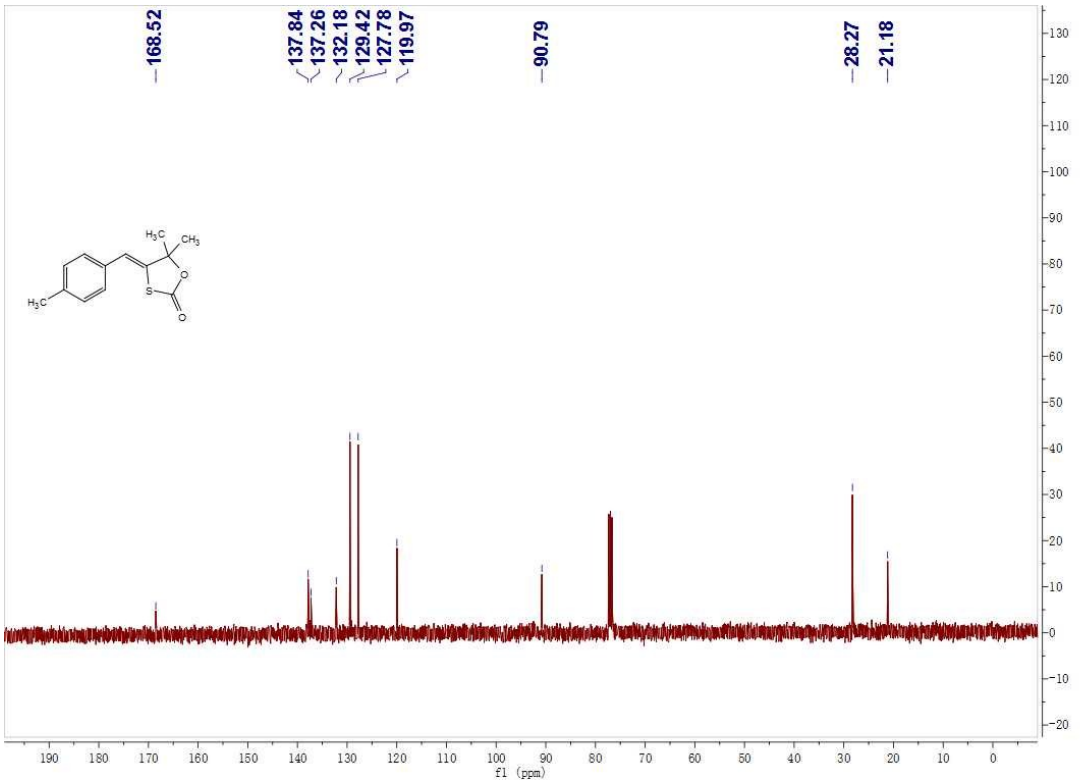

131.49
128.14
128.10
122.53
91.04
83.73
58.49
24.18
OH
CH3
f1 (ppm)

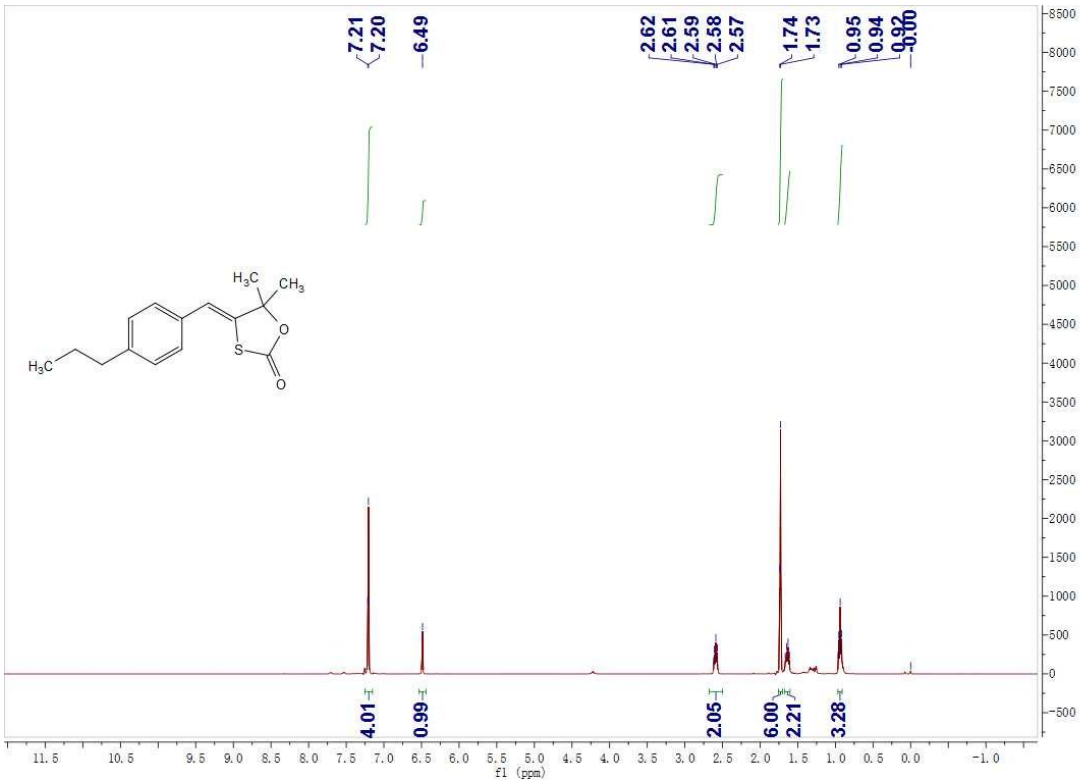
9. Spectrum of Products

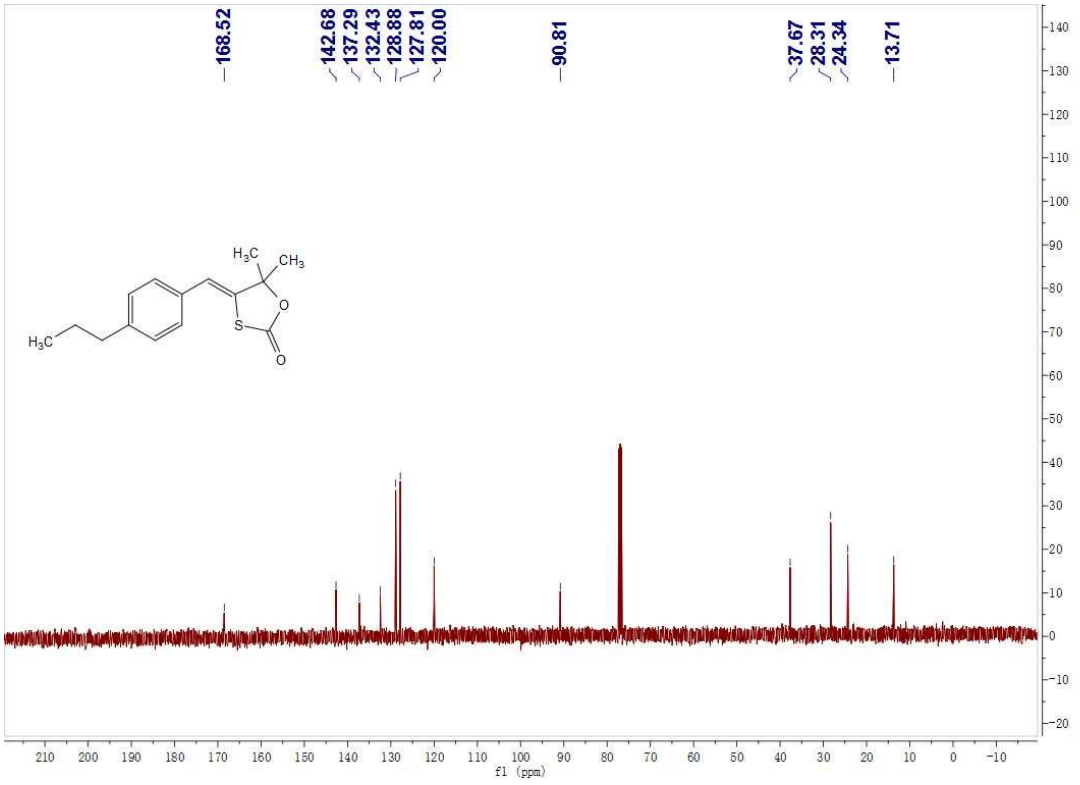


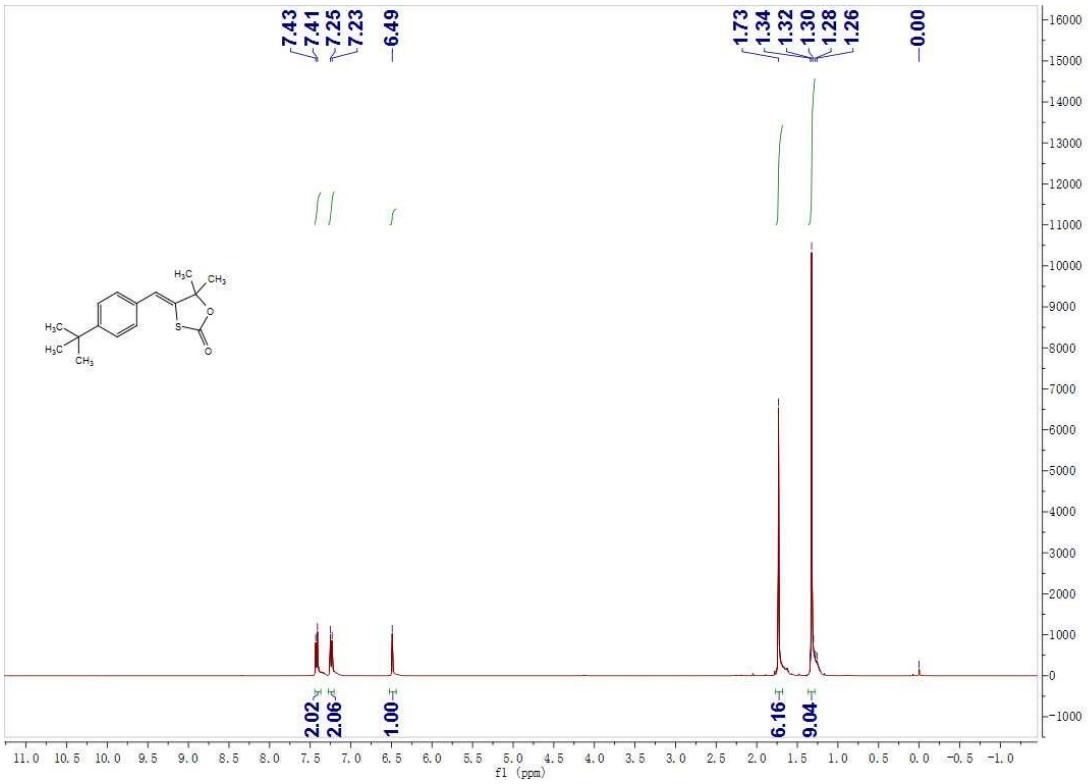


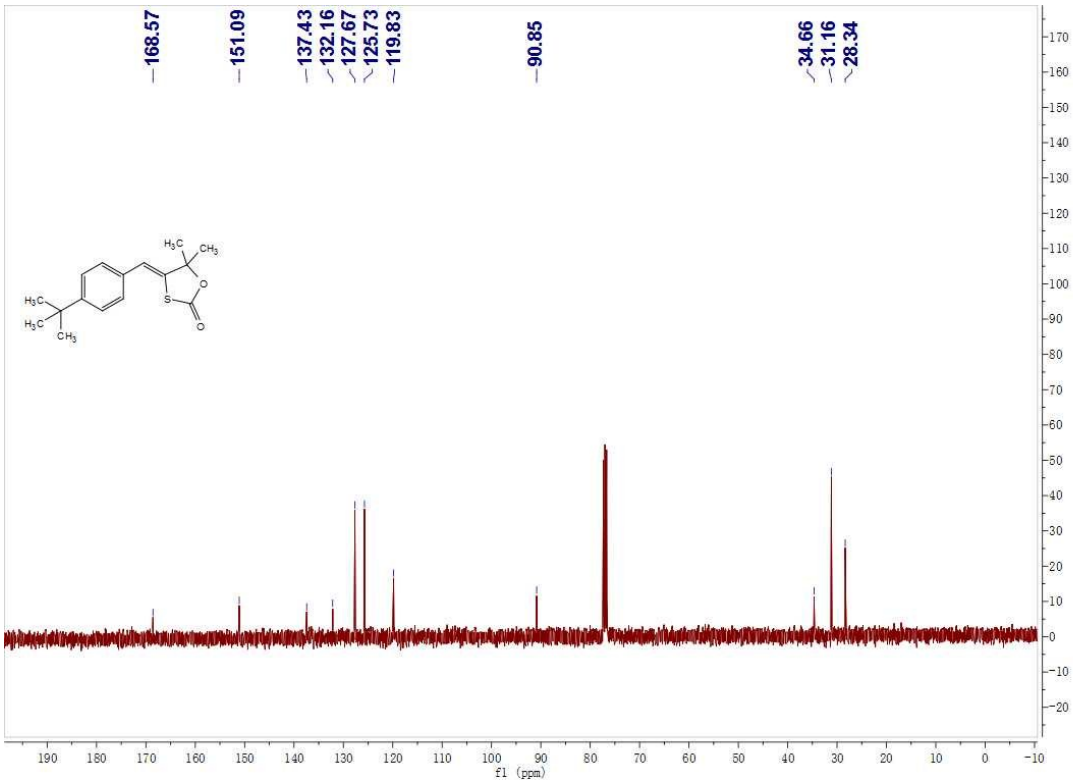

7.19
6.48
2.35
1.73
-0.00
4.00
1.00
3.03
6.03
f1 (ppm)

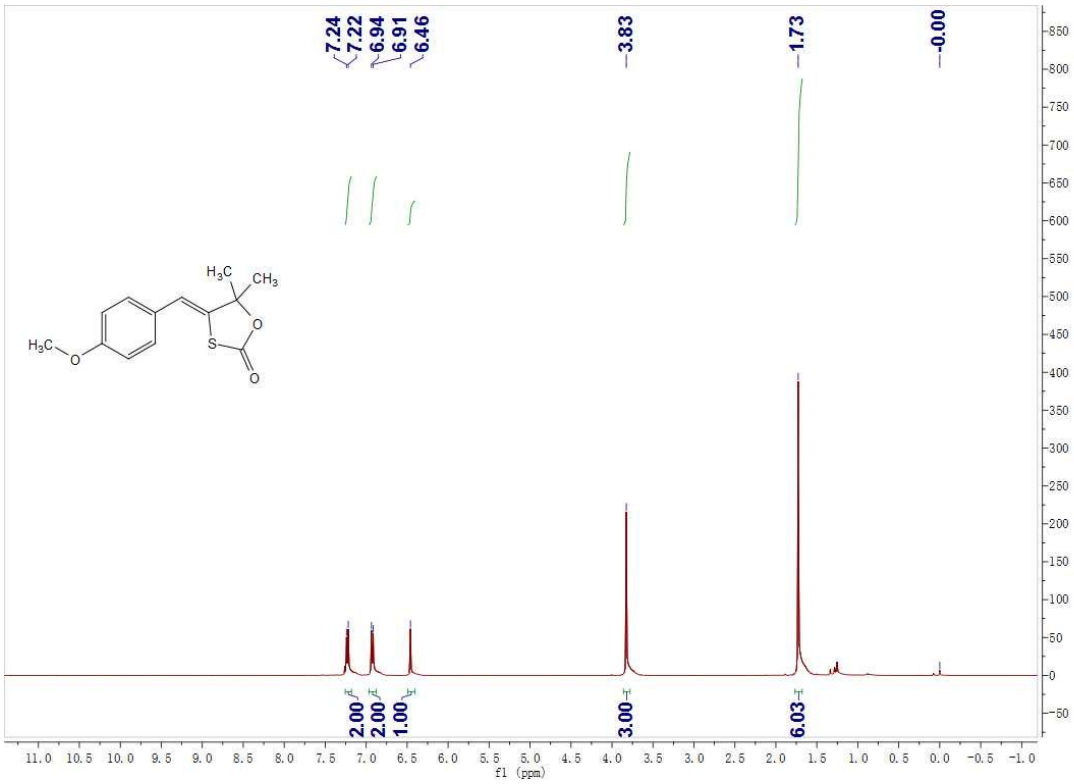

168.52
137.84
137.26
132.18
129.42
127.78
119.97
90.79
28.27
21.18
f1 (ppm)

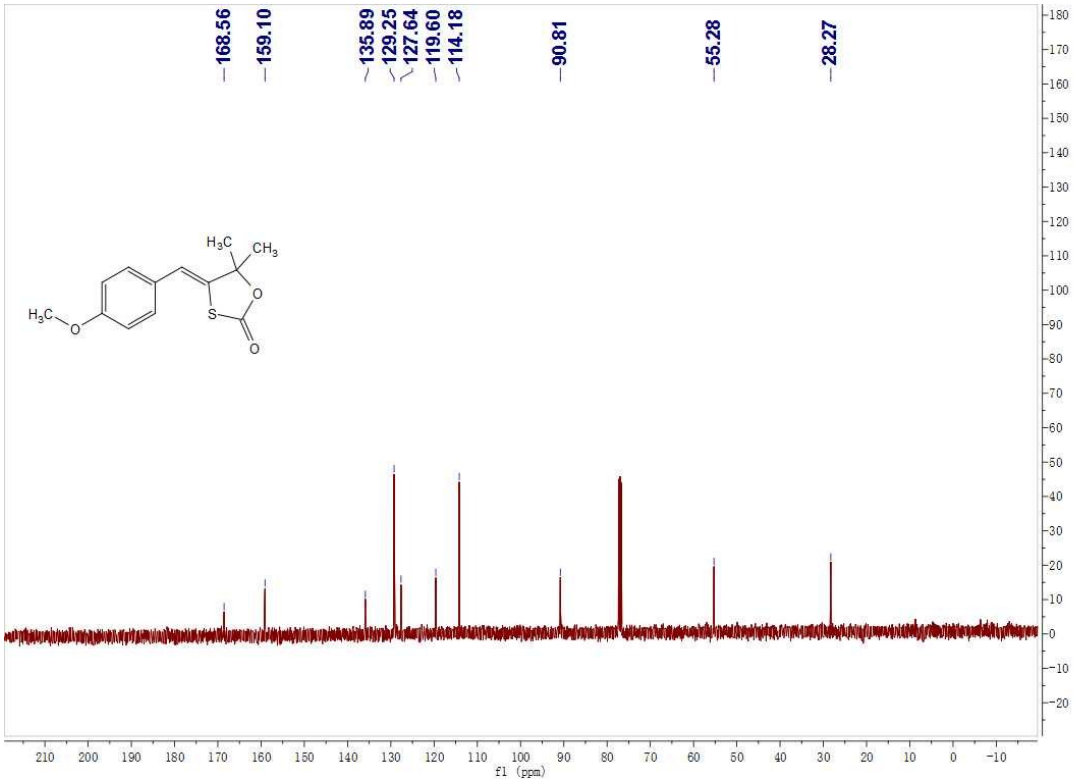

7.21
7.20
6.49
2.62
2.61
2.59
2.58
2.57
1.74
1.73
0.95
0.94
0.92
0.00
4.01
0.99
2.05
6.00
2.21
3.28
f1 (ppm)

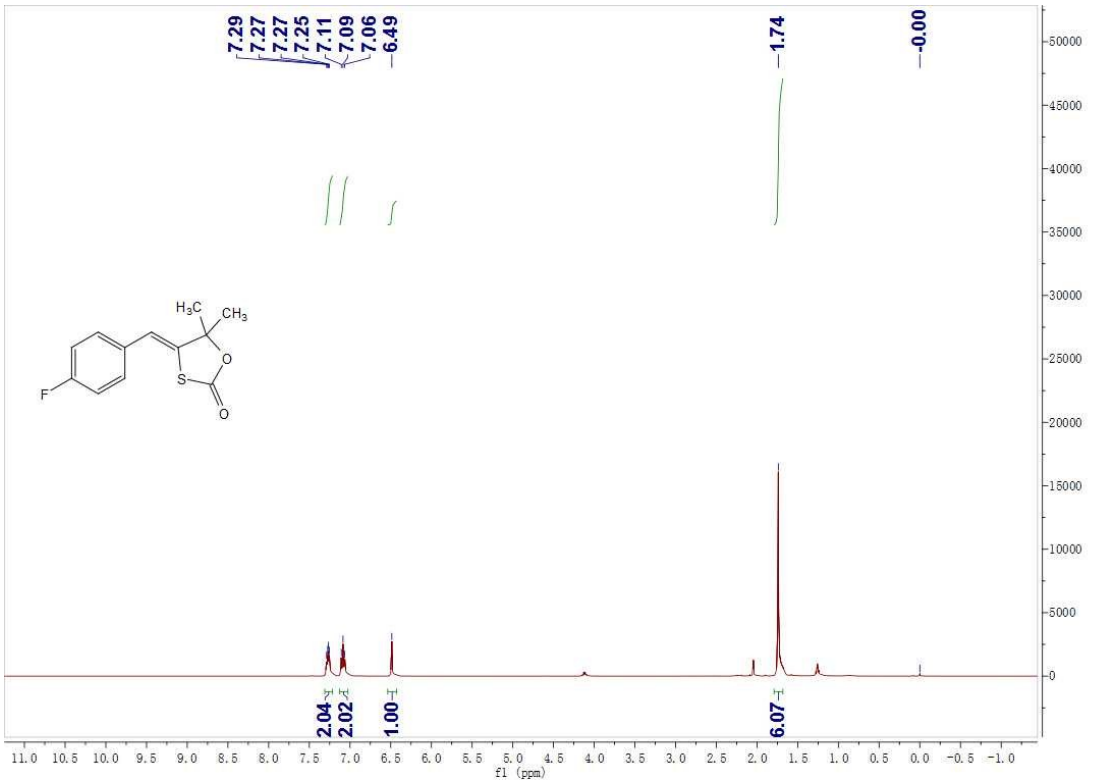

168.52
142.68
137.29
132.43
128.88
127.81
120.00
90.81
37.67
28.31
24.34
13.71
f1 (ppm)

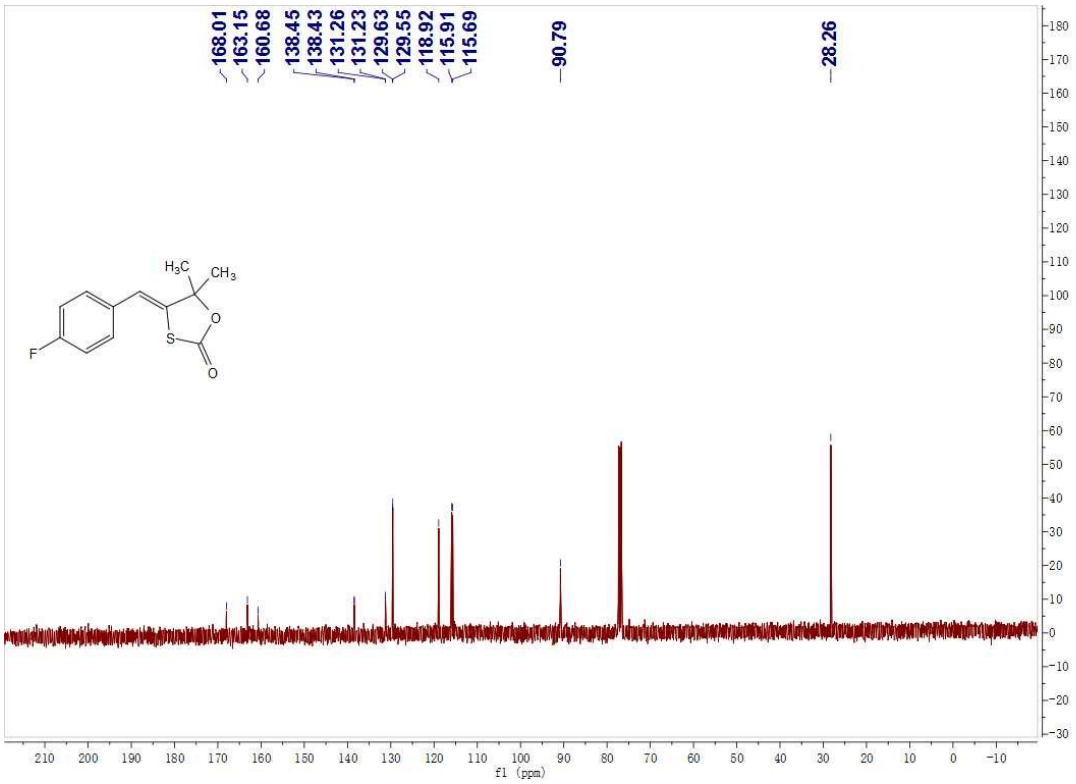

7.43
7.41
7.25
7.23
6.49
1.73
1.34
1.32
1.30
1.28
1.26
0.00
2.02
2.06
1.00
6.16
9.04
f1 (ppm)

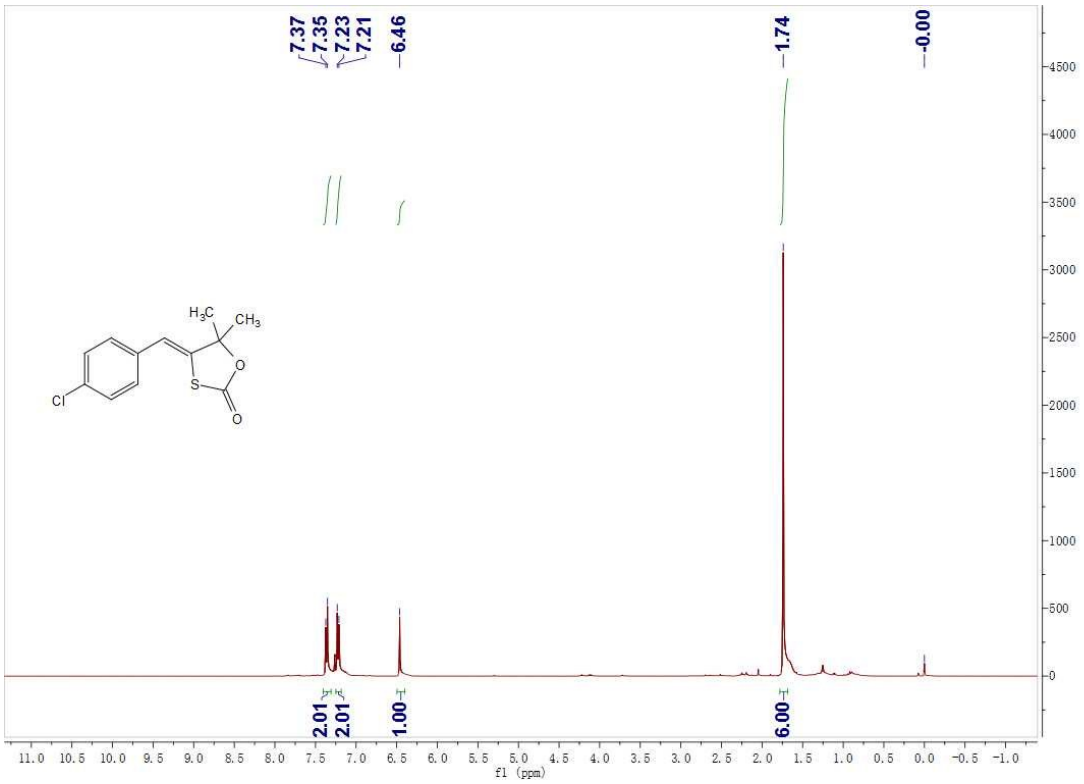

168.57
151.09
137.43
132.16
127.67
125.73
119.83
90.85
34.66
31.16
28.34
f1 (ppm)

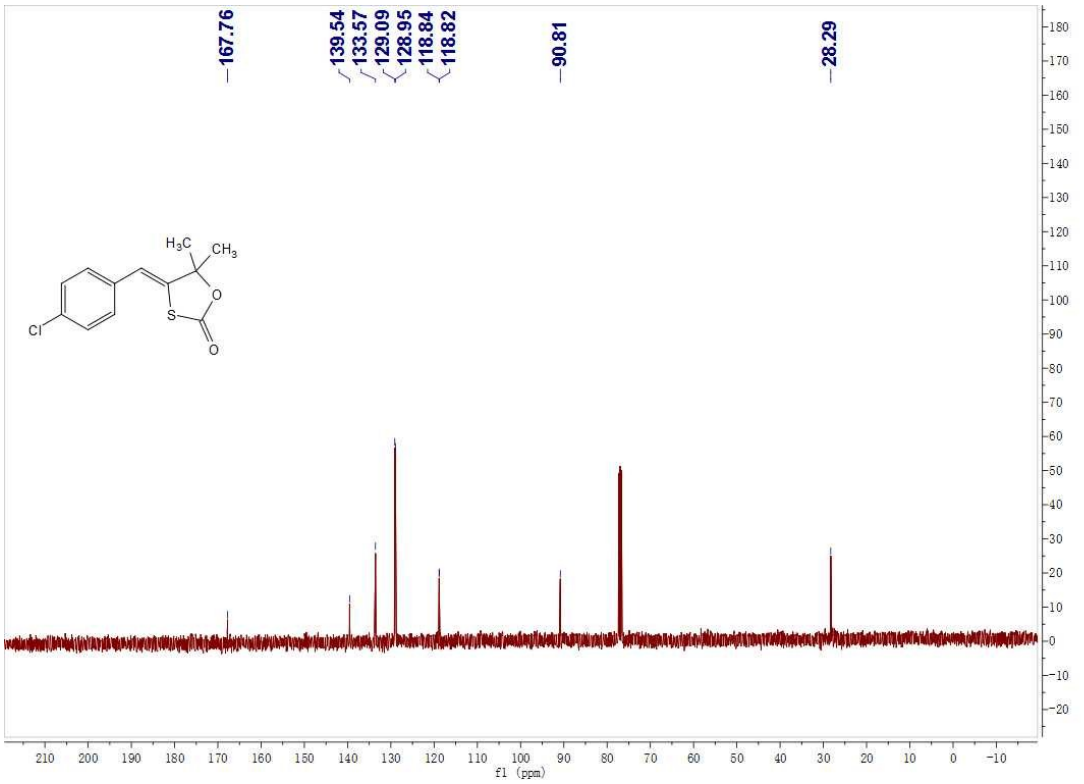

7.24
7.22
6.94
6.91
6.46
3.83
1.73
-0.00
2.00
2.00
1.00
3.00
6.03
f1 (ppm)


168.56
159.10
135.89
129.25
127.64
119.60
114.18
90.81
55.28
28.27
f1 (ppm)


7.29
7.27
7.27
7.25
7.11
7.09
7.06
6.49
1.74
0.00
2.04
2.02
1.00
6.07
f1 (ppm)


168.01
163.15
160.68
138.45
138.43
131.26
131.23
129.63
129.55
118.92
115.91
115.69
90.79
28.26
f1 (ppm)


7.37
7.35
7.23
7.21
6.46
1.74
0.00
2.01
2.01
1.00
6.00
f1 (ppm)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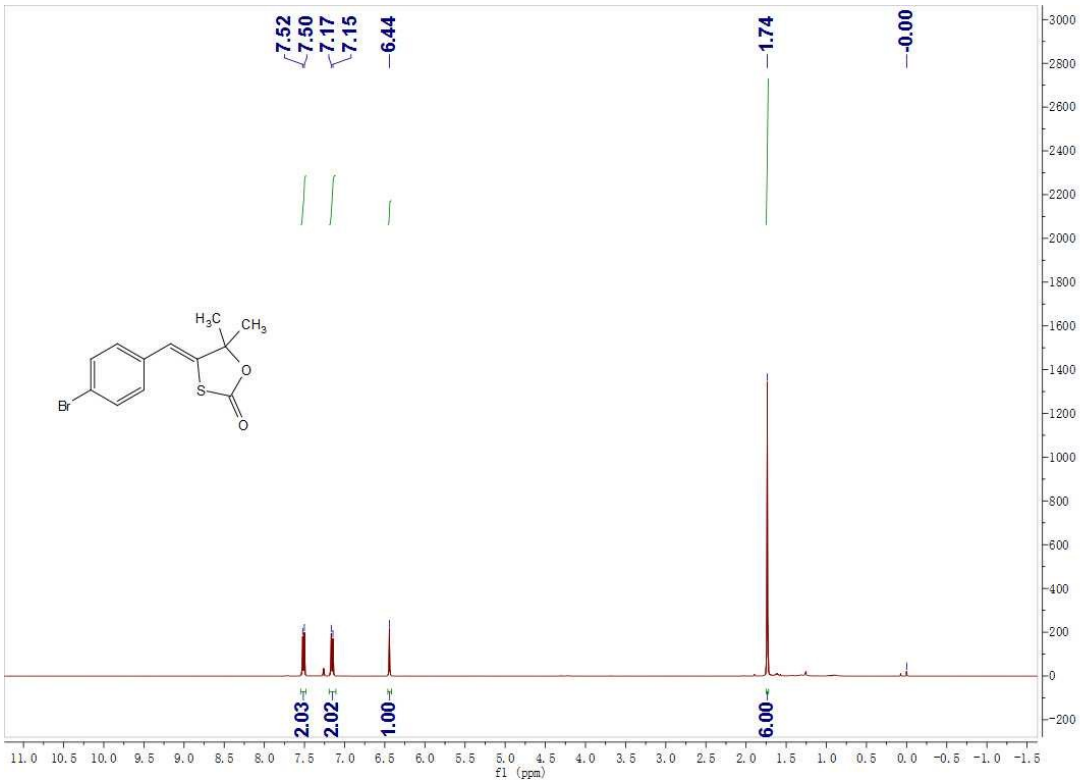
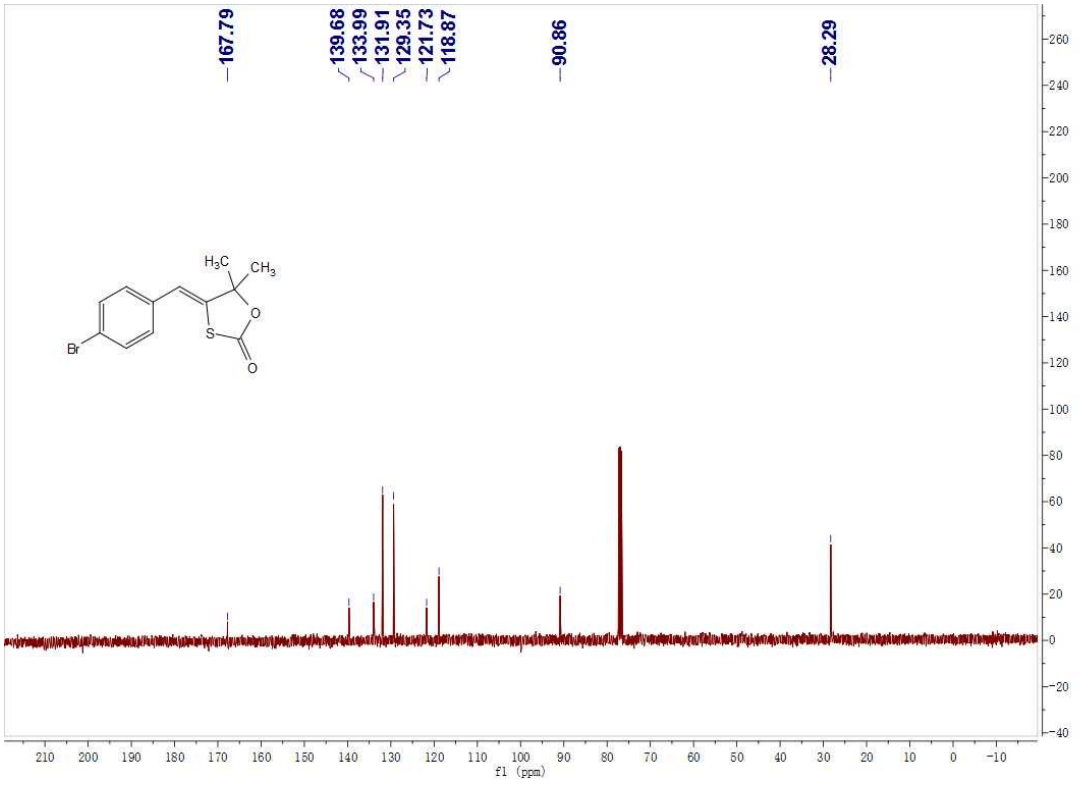
139.54
133.57
129.09
128.95
118.84
118.82
90.81
28.29
f1 (ppm)


7.52
7.50
7.17
7.15
6.44
1.74
0.00
2.03
2.02
1.00
6.00
f1 (ppm)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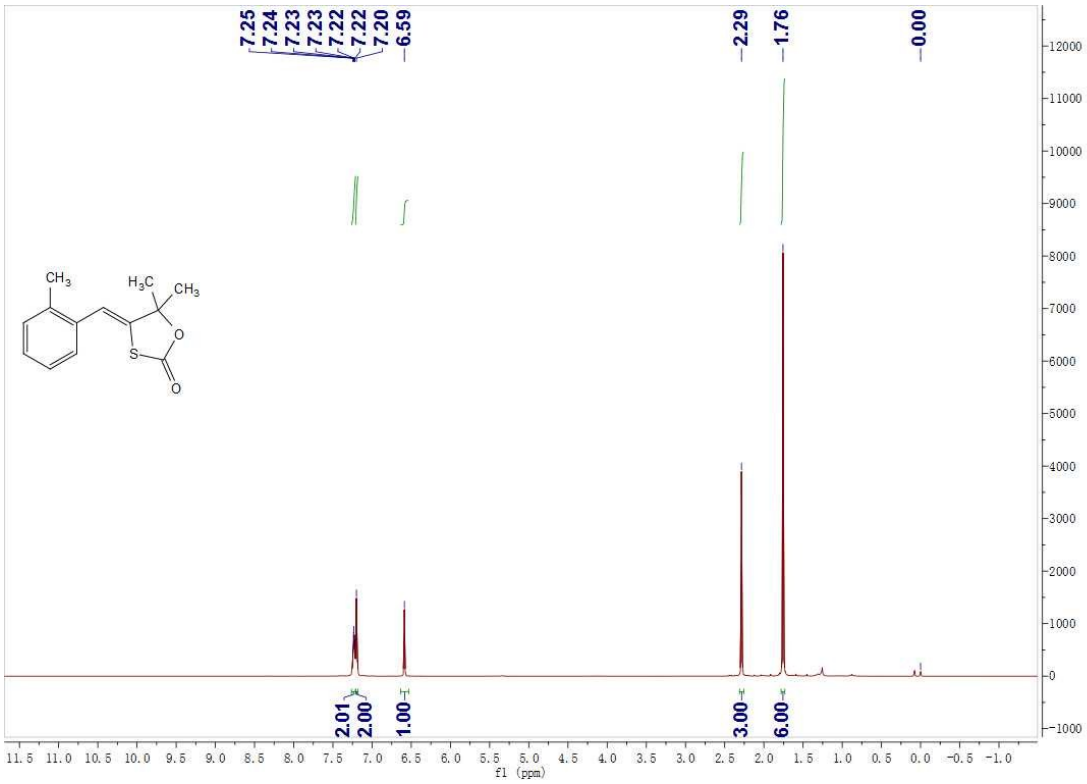
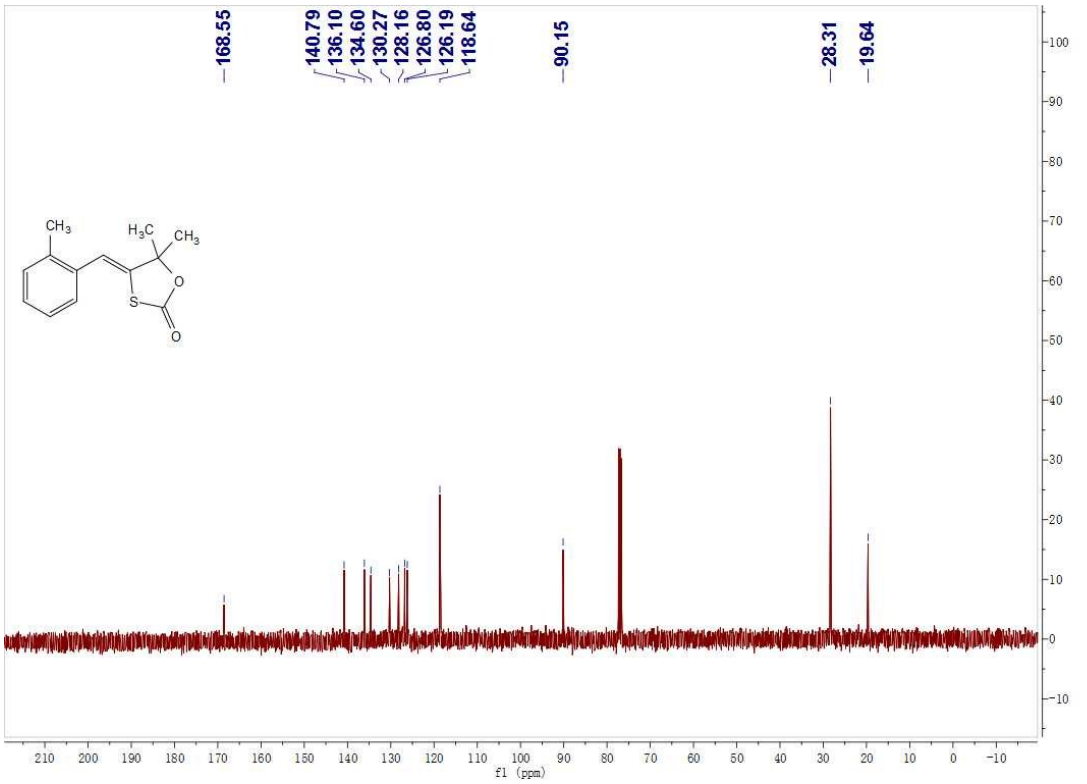
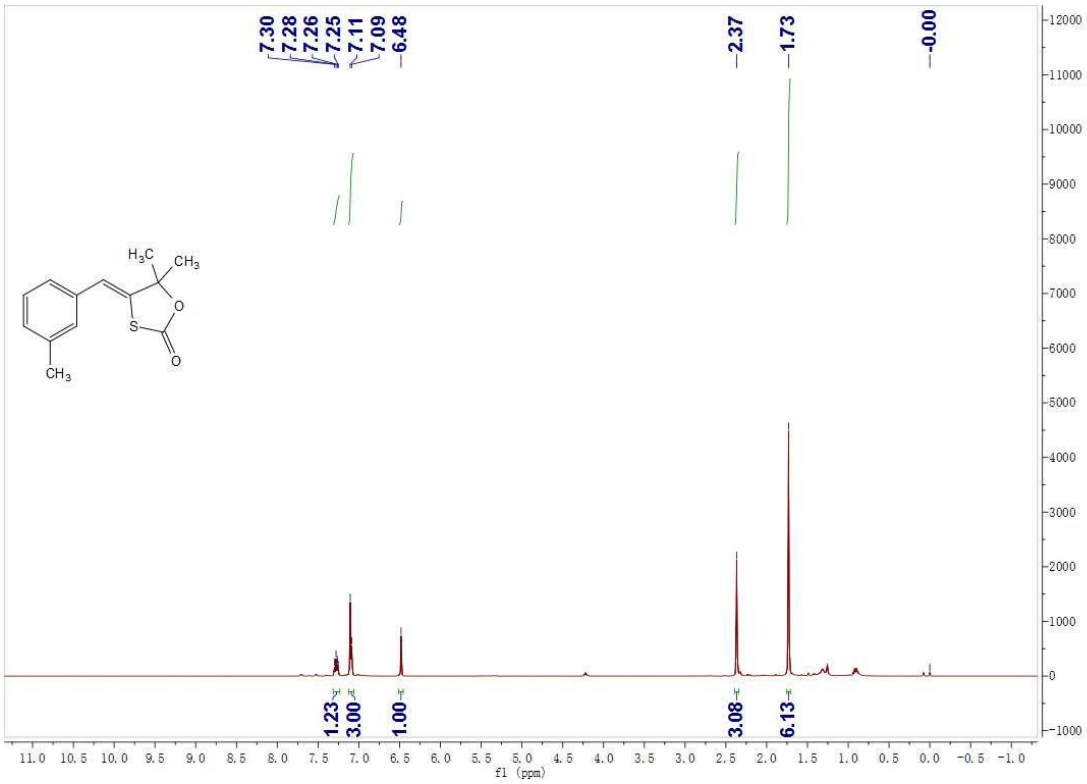
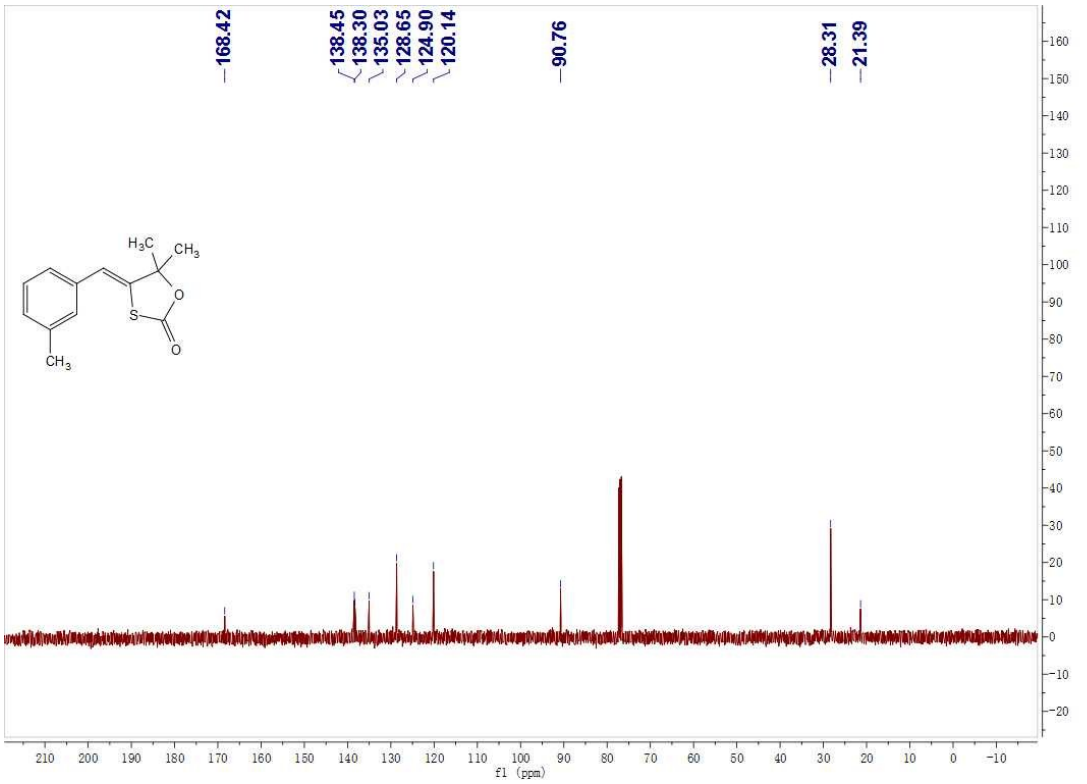
139.68
133.99
131.91
129.35
121.73
118.87
90.86
28.29
f1 (ppm)


7.25
7.24
7.23
7.23
7.22
7.22
7.20
6.59
2.29
1.76
0.00
2.01
2.00
1.00
3.00
6.00
f1 (ppm)


168.55
140.79
136.10
134.60
130.27
128.16
126.80
126.19
118.64
90.15
28.31
19.64
f1 (ppm)


7.30
7.28
7.26
7.25
7.11
7.09
6.48
2.37
1.73
0.00
1.23
3.00
1.00
3.08
6.13
f1 (ppm)


168.42
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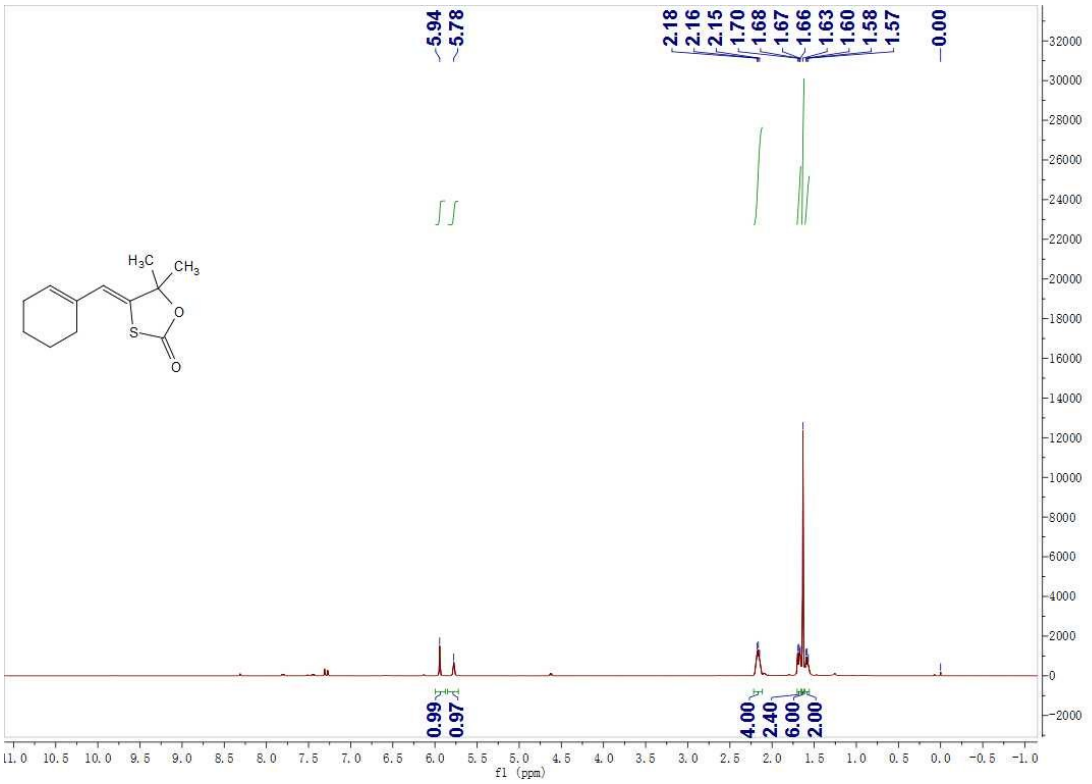
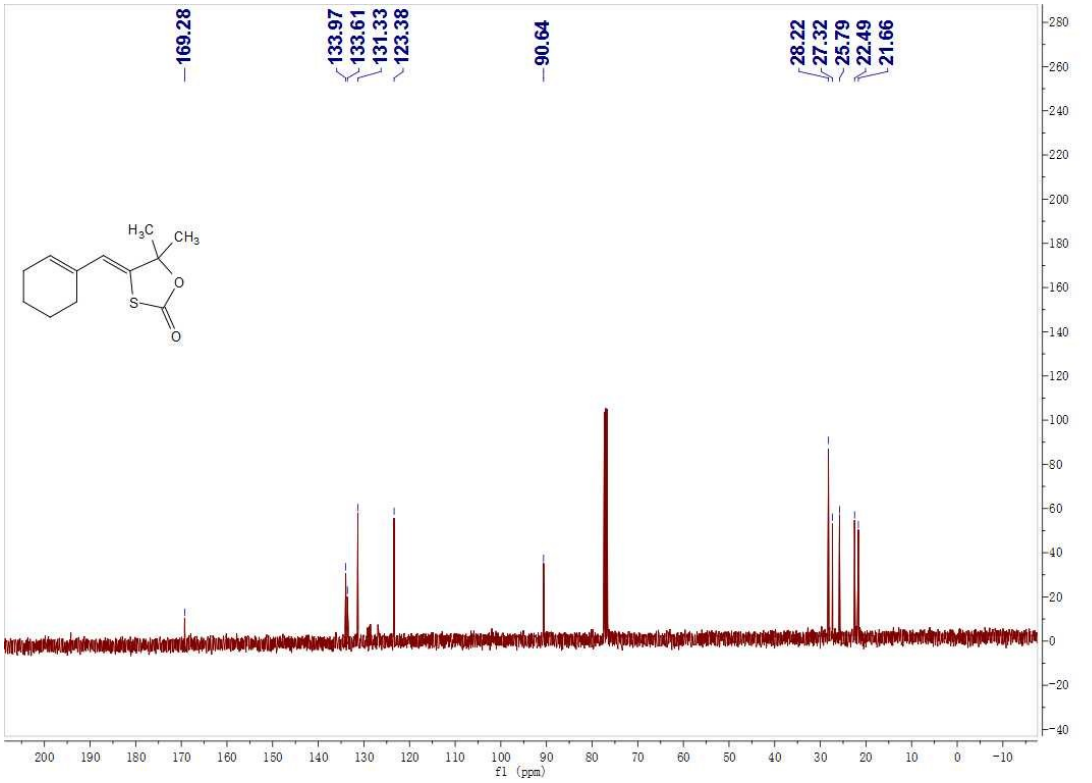
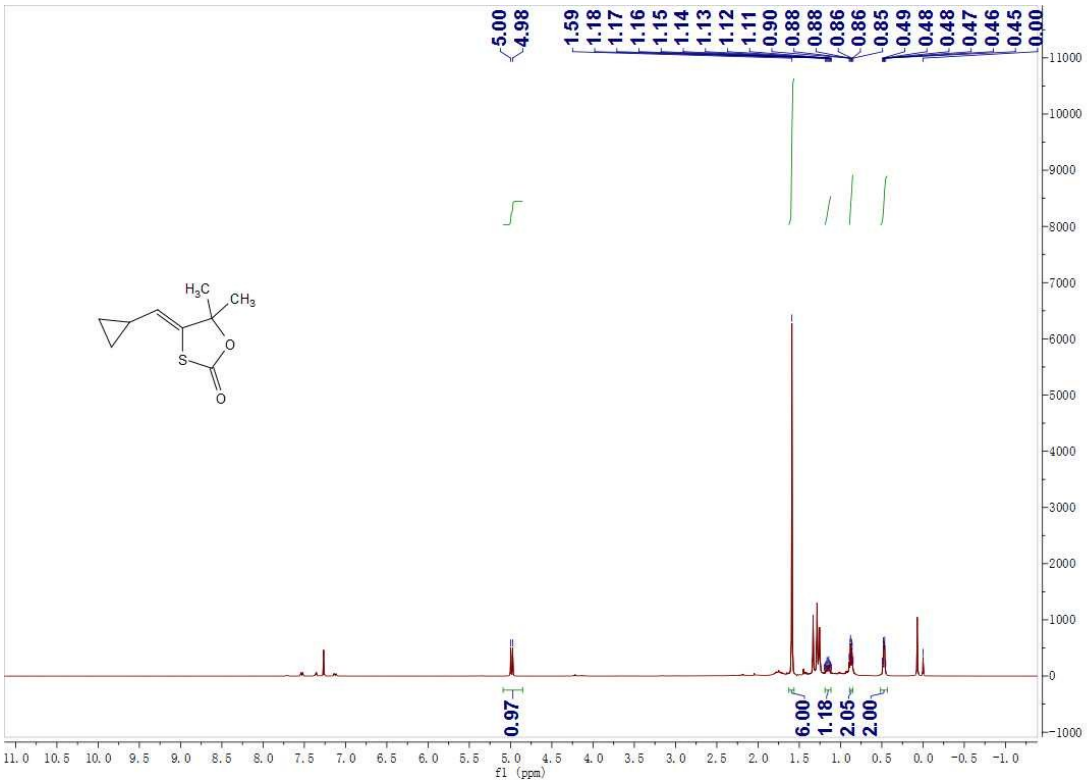
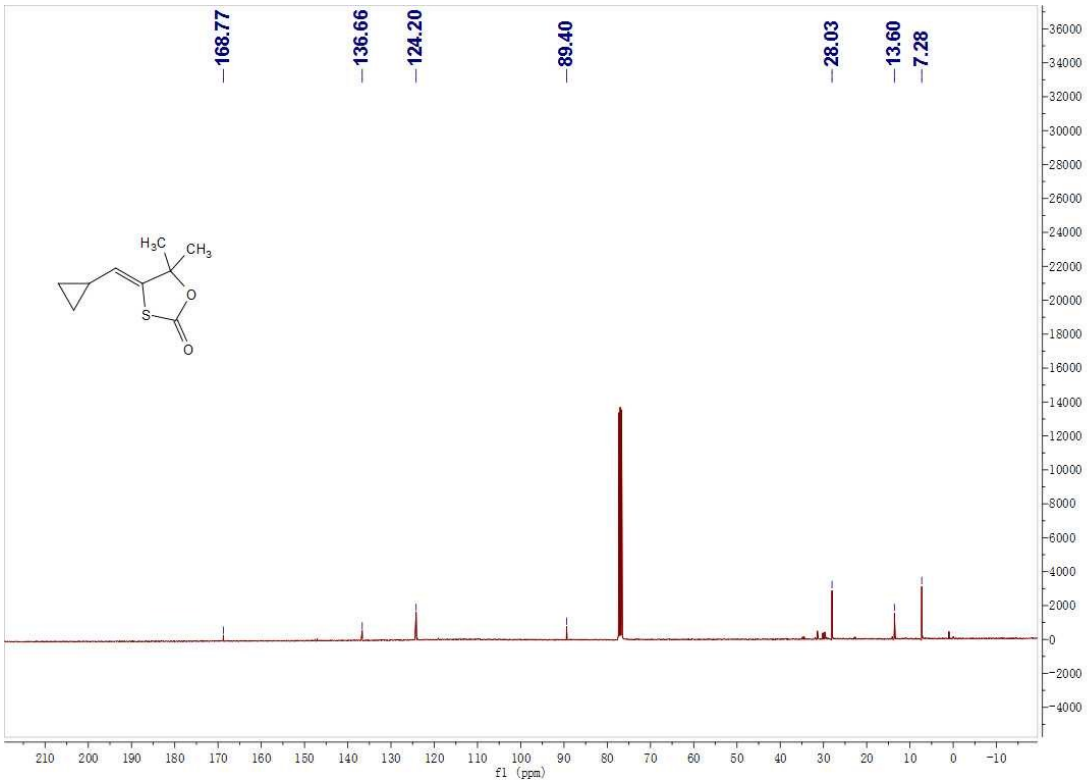
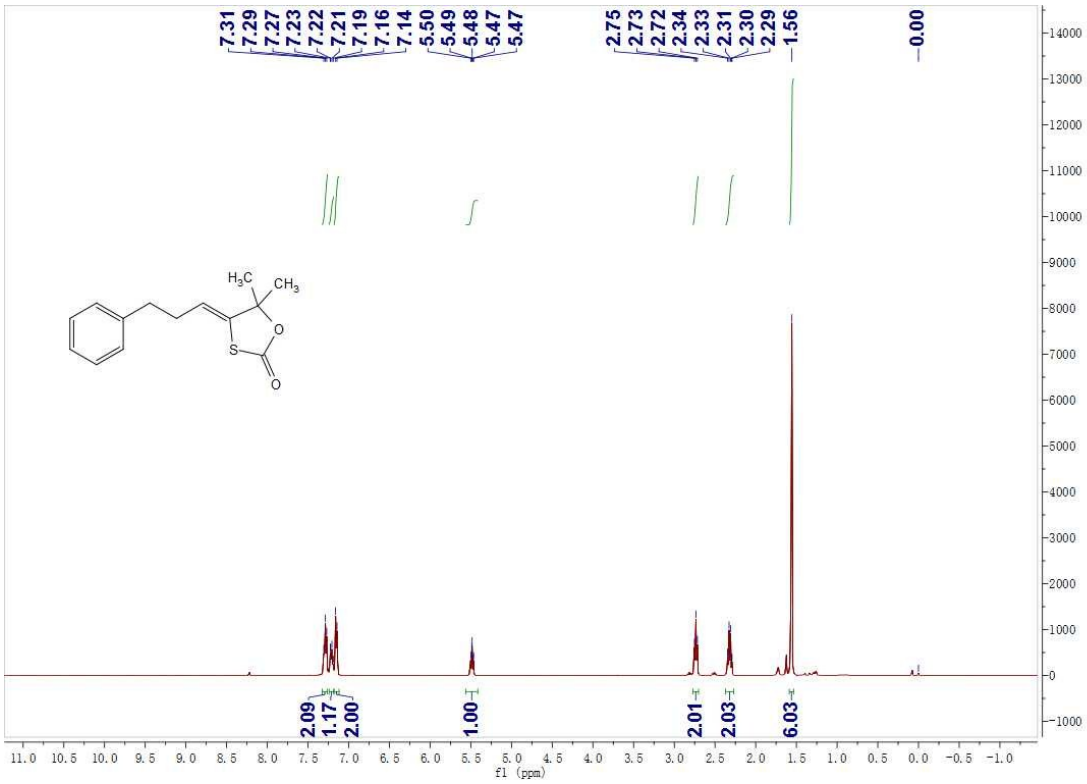
135.03
128.65
124.90
120.14
90.76
28.31
21.39
f1 (ppm)

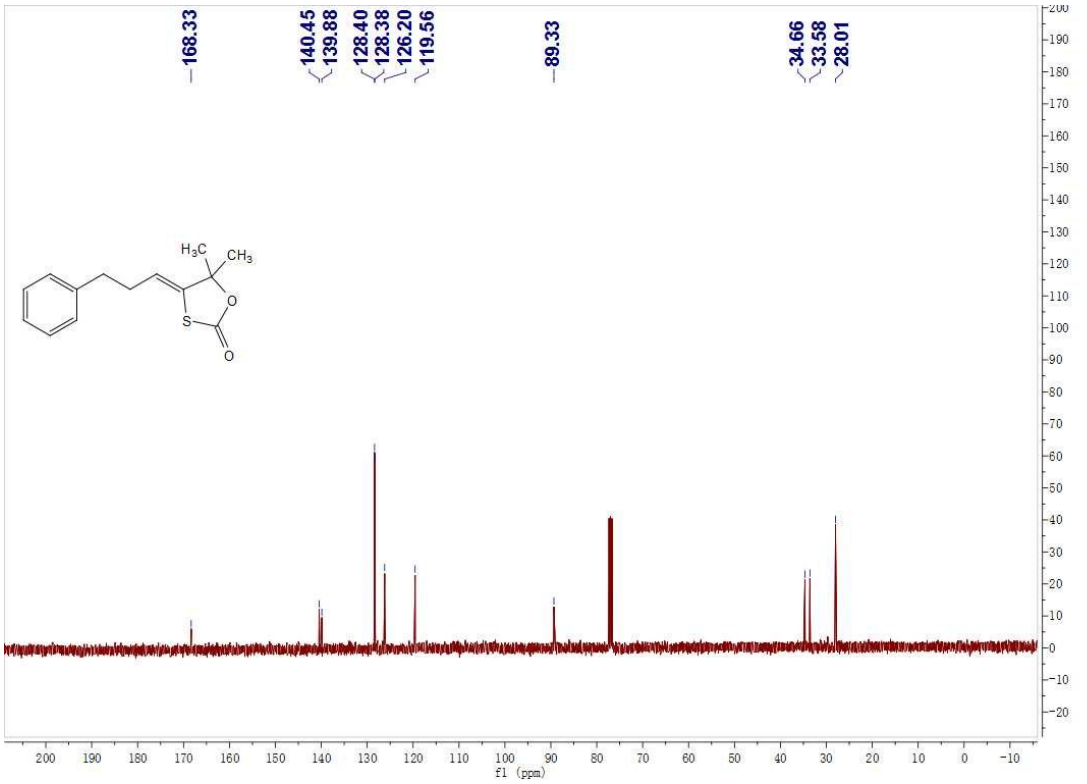

5.94
5.78
2.18
2.16
2.15
1.70
1.68
1.67
1.66
1.63
1.60
1.58
1.57
0.00
0.99
0.97
4.00
2.40
6.00
2.00
f1 (ppm)


169.28
133.97
133.61
131.33
123.38
90.64
28.22
27.32
25.79
22.49
21.66
f1 (ppm)


5.00
4.98
1.59
1.18
1.17
1.16
1.15
1.14
1.13
1.12
1.11
0.90
0.88
0.88
0.86
0.86
0.85
0.49
0.48
0.48
0.47
0.46
0.45
0.00
0.97
6.00
1.18
2.05
2.00
f1 (ppm)


168.77
136.66
124.20
89.40
28.03
13.60
7.28
f1 (ppm)


7.31
7.29
7.27
7.23
7.22
7.21
7.19
7.16
7.14
5.50
5.49
5.48
5.47
5.47
2.75
2.73
2.72
2.34
2.33
2.31
2.30
2.29
1.56
0.00
2.09
1.17
2.00
1.00
2.01
2.03
6.03
f1 (ppm)


168.33
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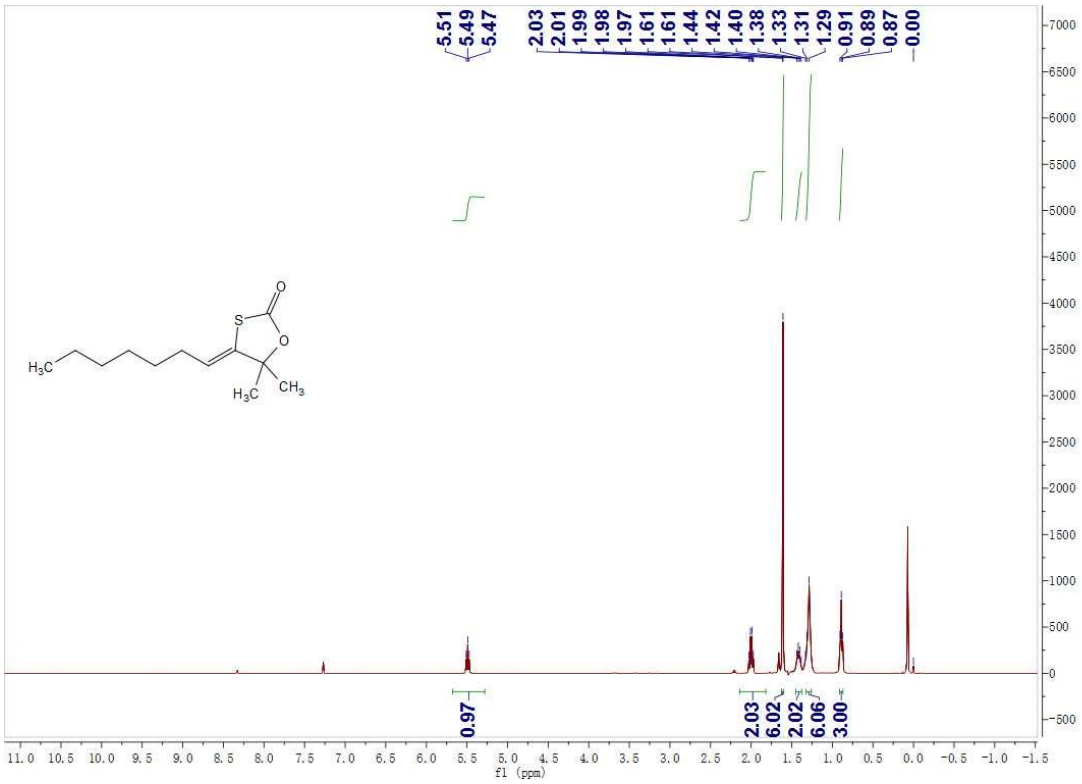
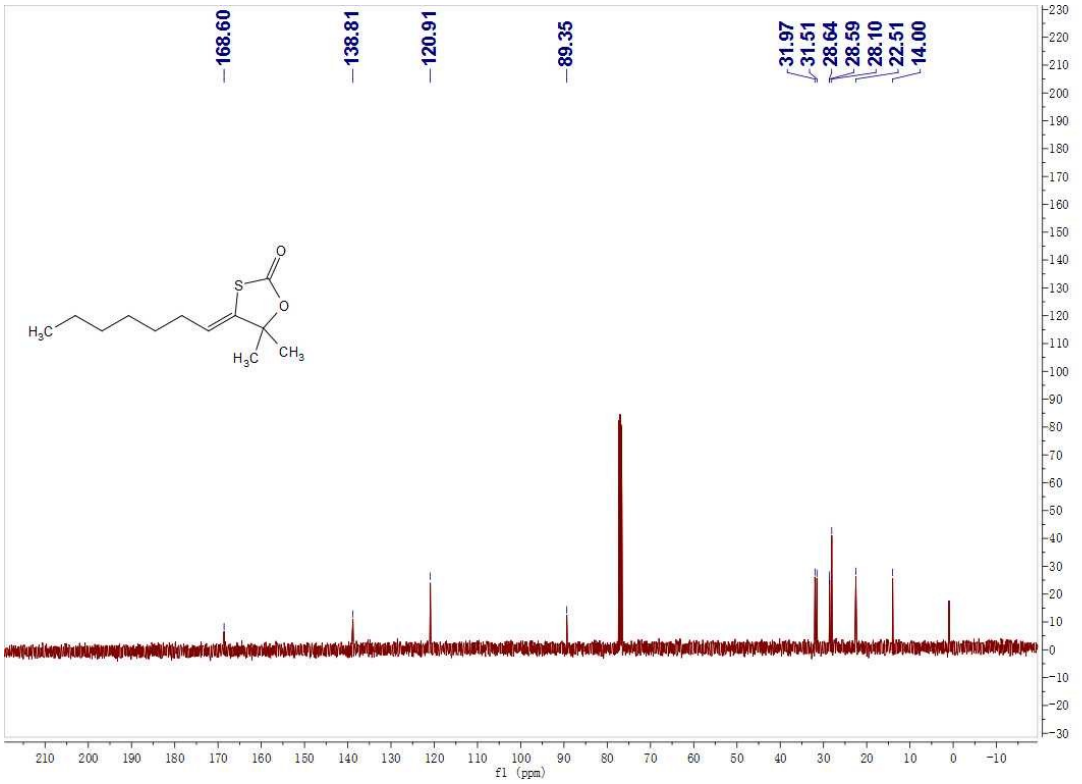
139.88
128.40
128.38
126.20
119.56
89.33
34.66
33.58
28.01
f1 (ppm)


5.51
5.49
5.47
2.03
2.01
1.99
1.98
1.97
1.61
1.61
1.44
1.42
1.40
1.38
1.33
1.31
1.29
0.91
0.89
0.87
0.00
0.97
2.03
6.02
2.02
6.06
3.00
f1 (ppm)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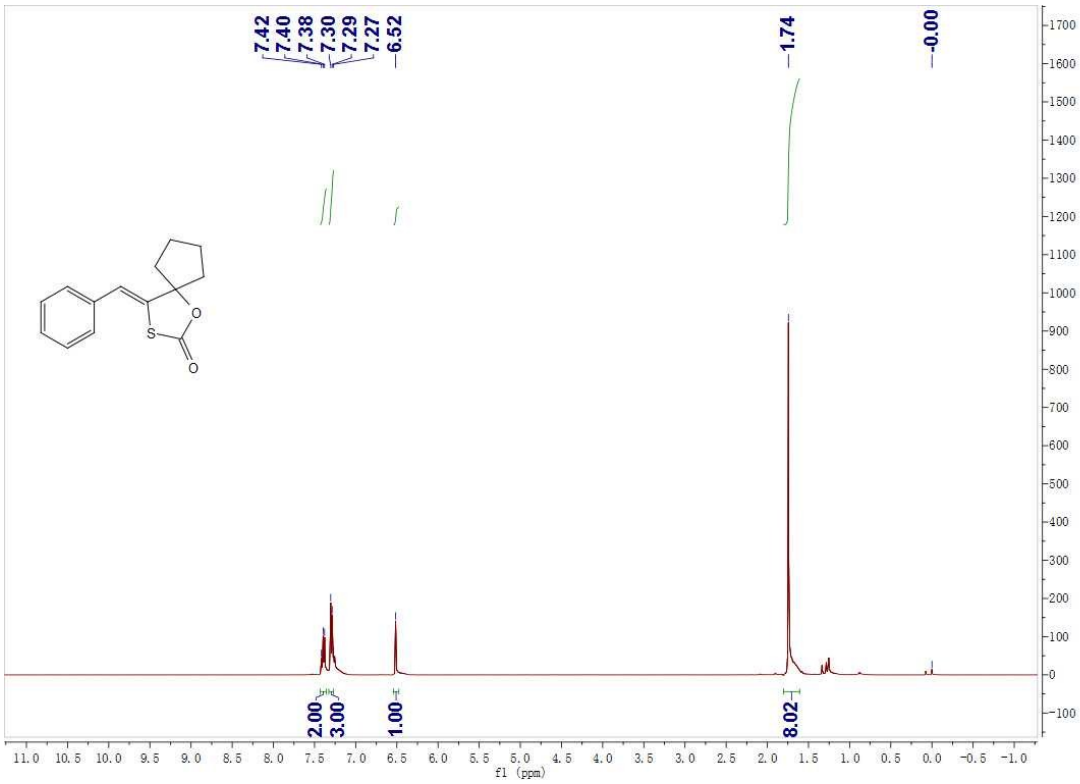
31.51
28.64
28.59
28.10
22.51
14.00
f1 (ppm)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7.27
6.52
1.74
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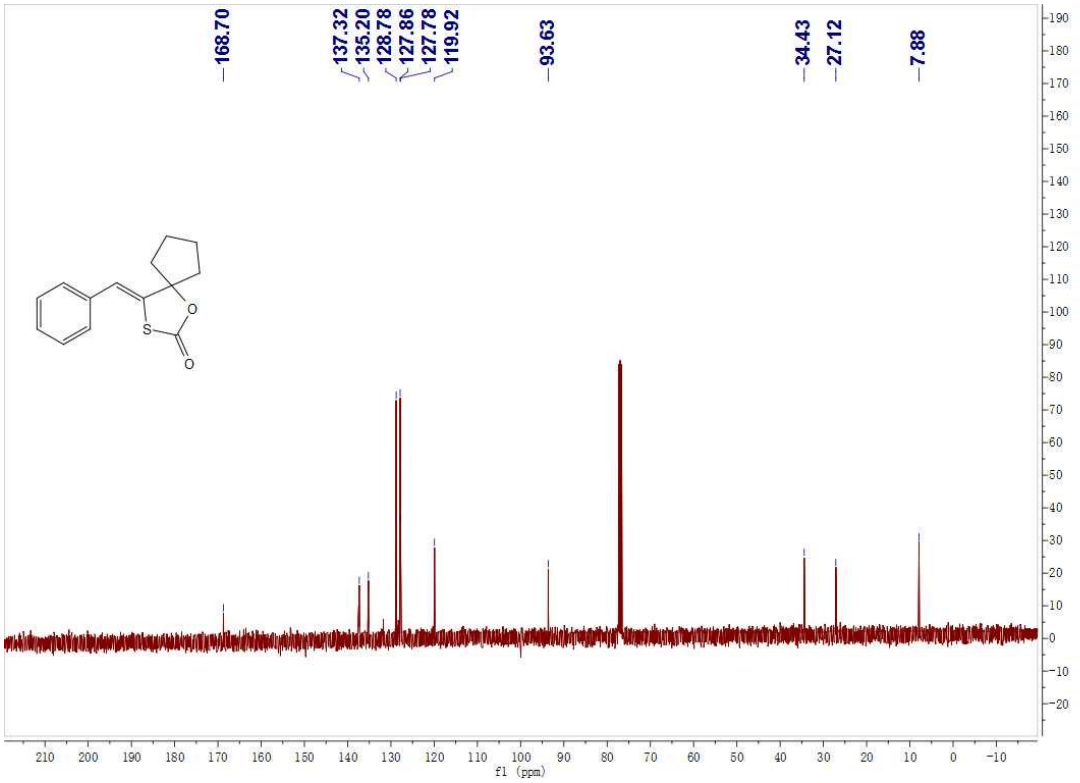
3.00
1.00
8.02
f1 (ppm)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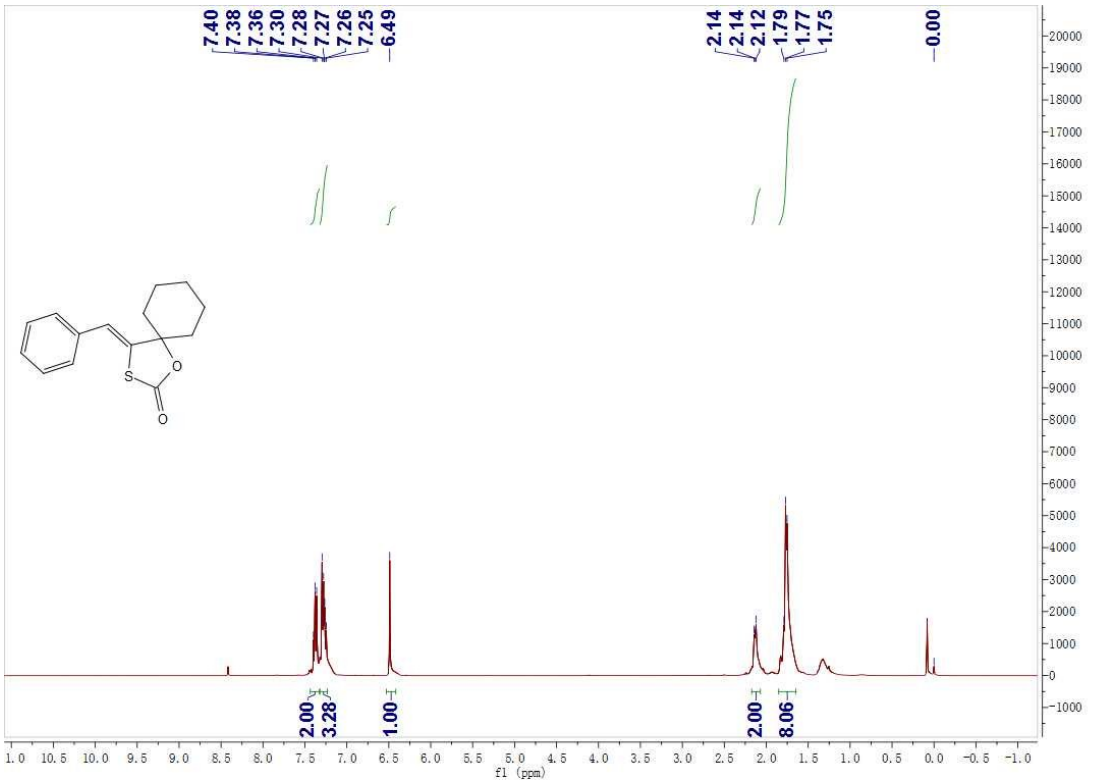
7.88
f1 (ppm)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7.27
7.26
7.25
6.49
2.14
2.14
2.12
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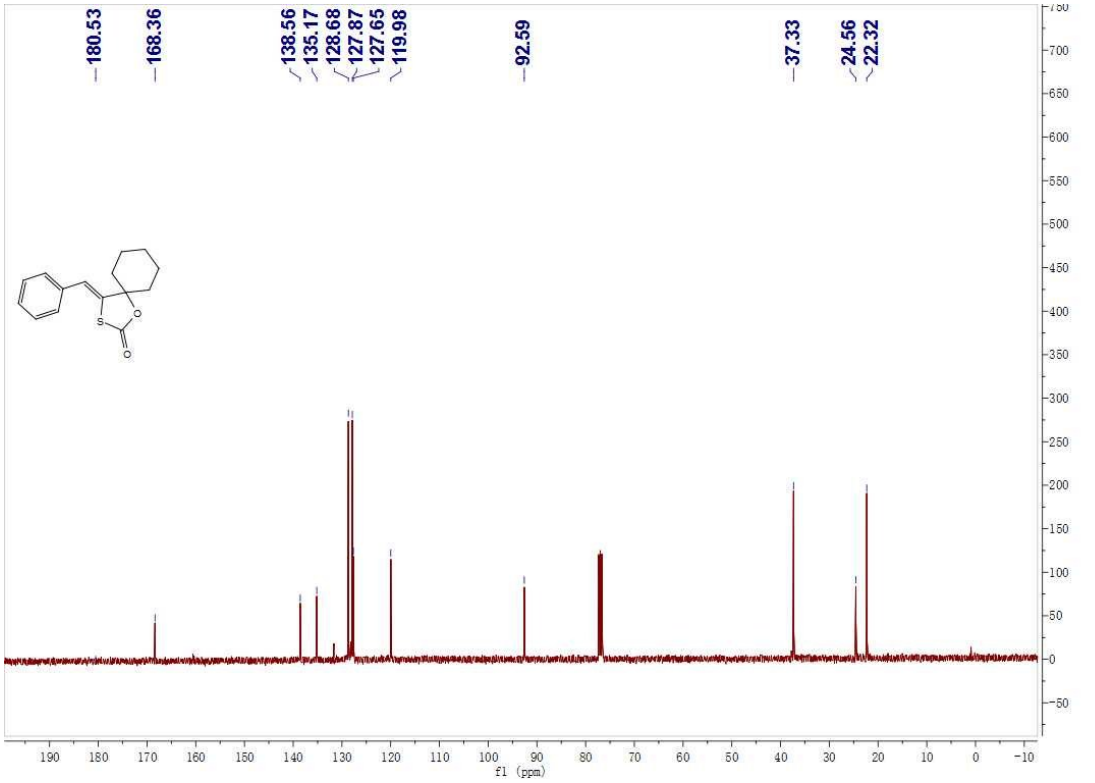
1.77
1.75
0.00
2.00
3.28
1.00
2.00
8.06
f1 (ppm)


180.53
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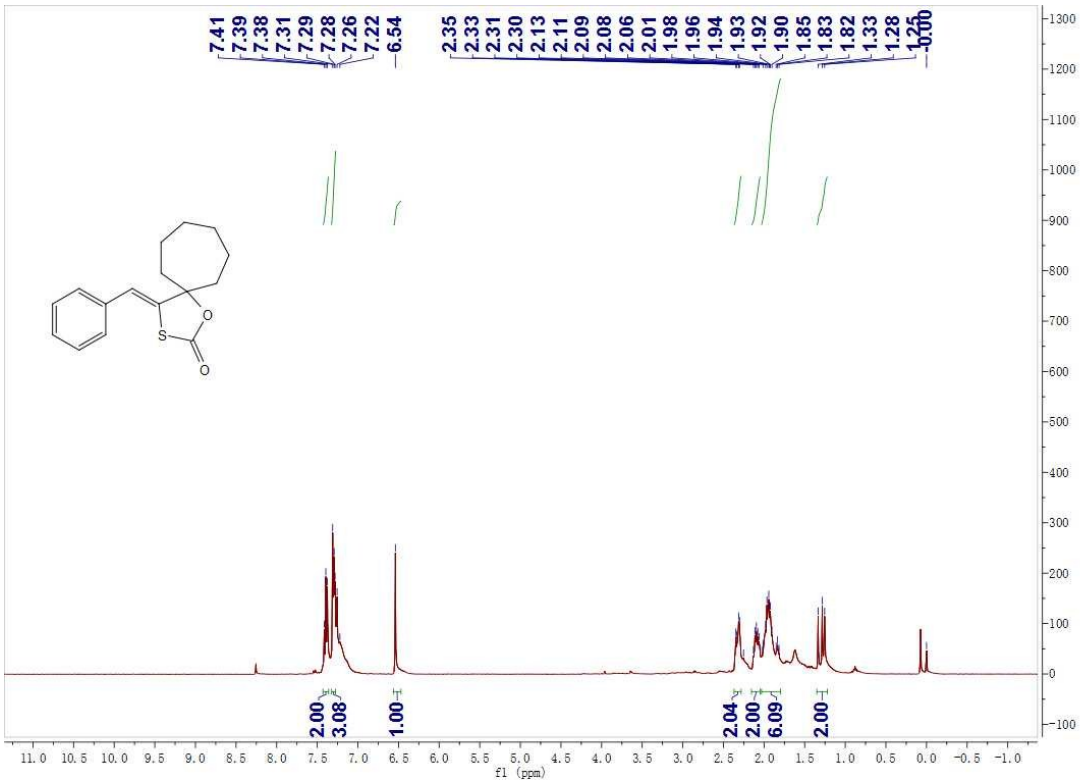
127.87
127.65
119.98
92.59
37.33
24.56
22.32
f1 (ppm)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7.26
7.22
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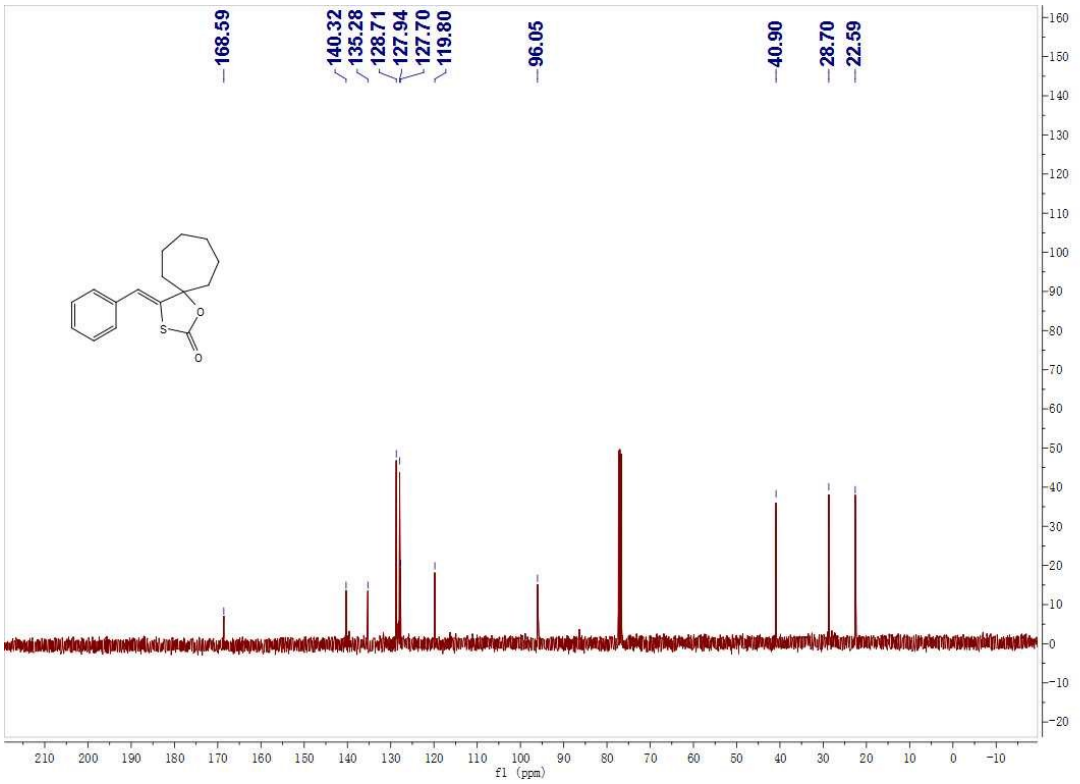
2.33
2.31
2.30
2.13
2.11
2.09
2.08
2.06
2.01
1.98
1.96
1.94
1.93
1.92
1.90
1.85
1.83
1.82
1.33
1.28
1.25
0.00
2.00
3.08
1.00
2.04
2.00
6.09
2.00
f1 (ppm)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119.80
96.05
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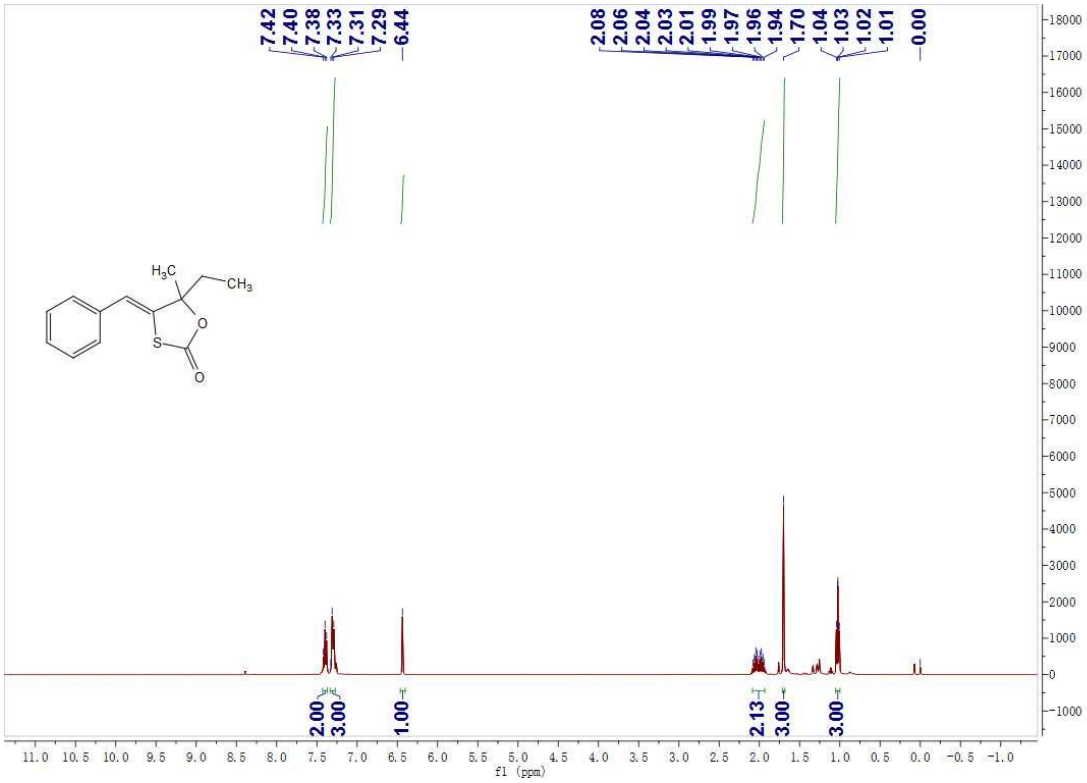
28.70
22.59
f1 (ppm)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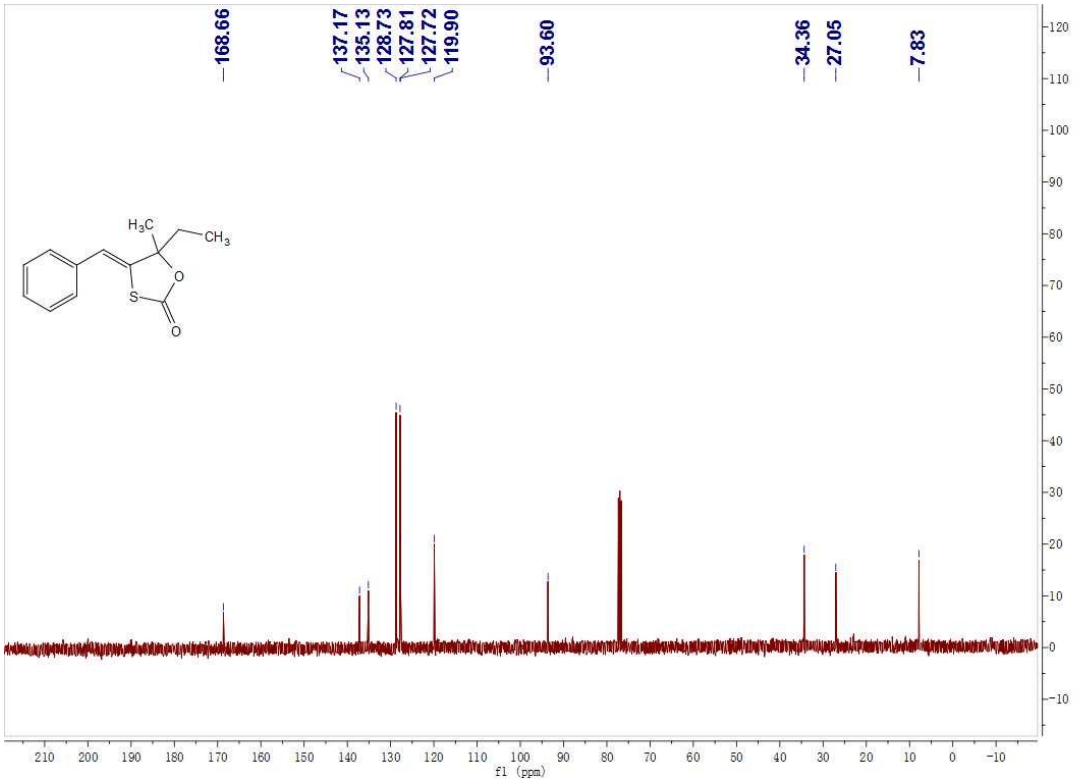
2.04
2.03
2.01
1.99
1.97
1.96
1.94
1.70
1.04
1.03
1.02
1.01
0.00
2.00
3.00
1.00
2.13
3.00
3.00
f1 (ppm)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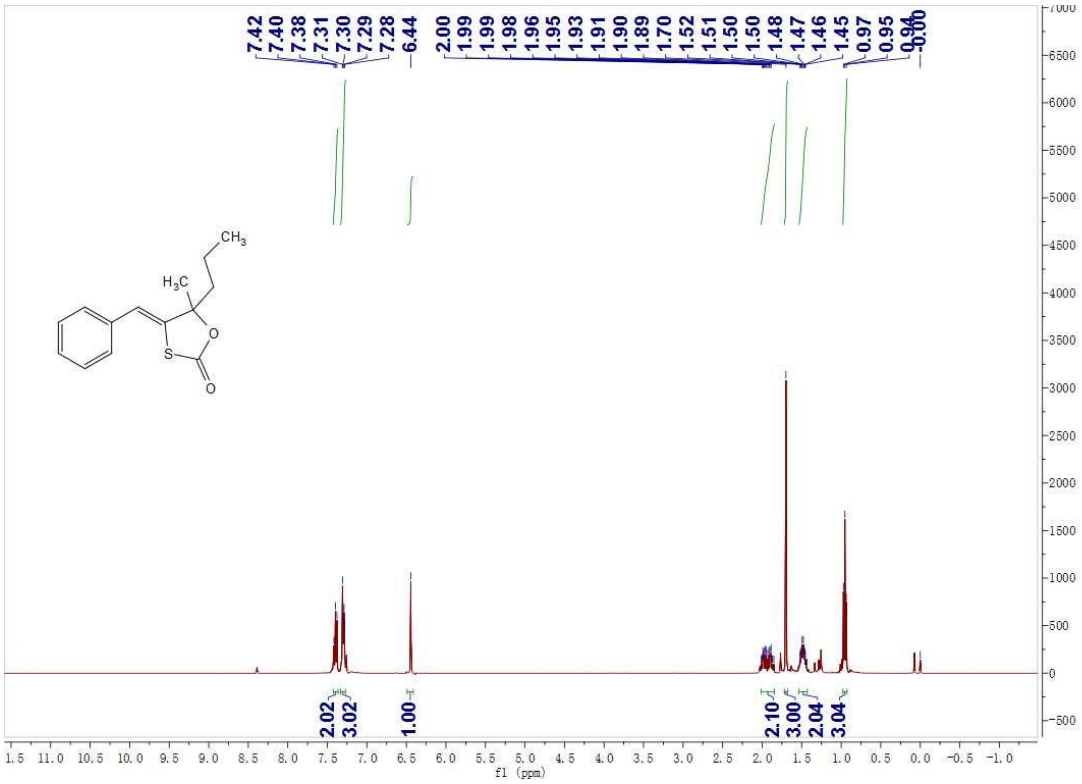
7.83
f1 (ppm)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7.29
7.28
6.44
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1.99
1.98
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1.91
1.90
1.89
1.70
1.52
1.51
1.50
1.50
1.48
1.47
1.46
1.45
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0.95
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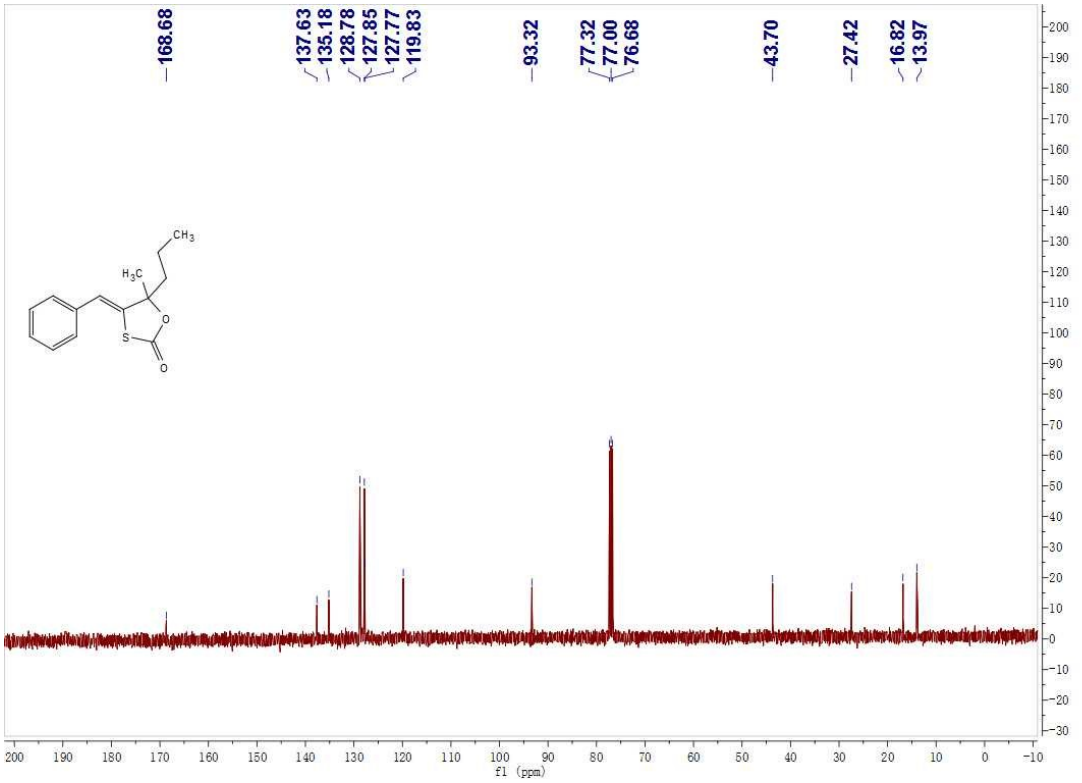
0.00
2.02
3.02
1.00
2.10
3.00
2.04
3.04
f1 (ppm)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77.32
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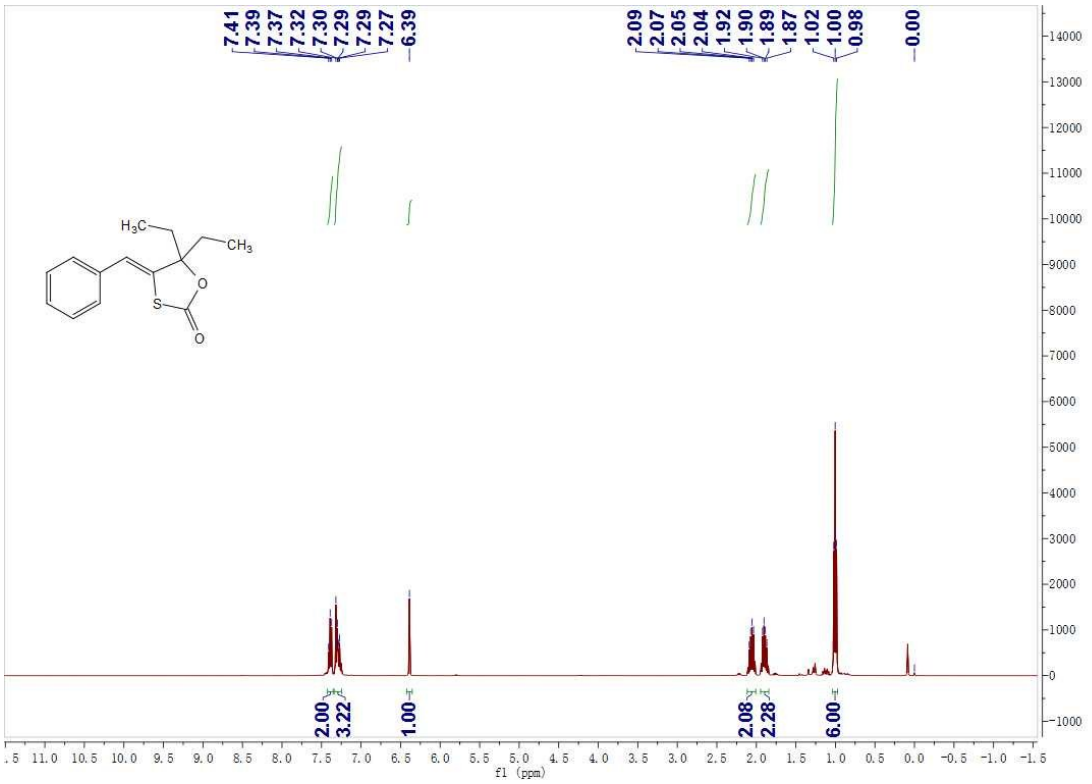
76.68
43.70
27.42
16.82
13.97
f1 (ppm)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7.27
6.39
2.09
2.07
2.05
2.04
1.92
1.90
1.89
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1.02
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0.98
0.00
2.00
3.22
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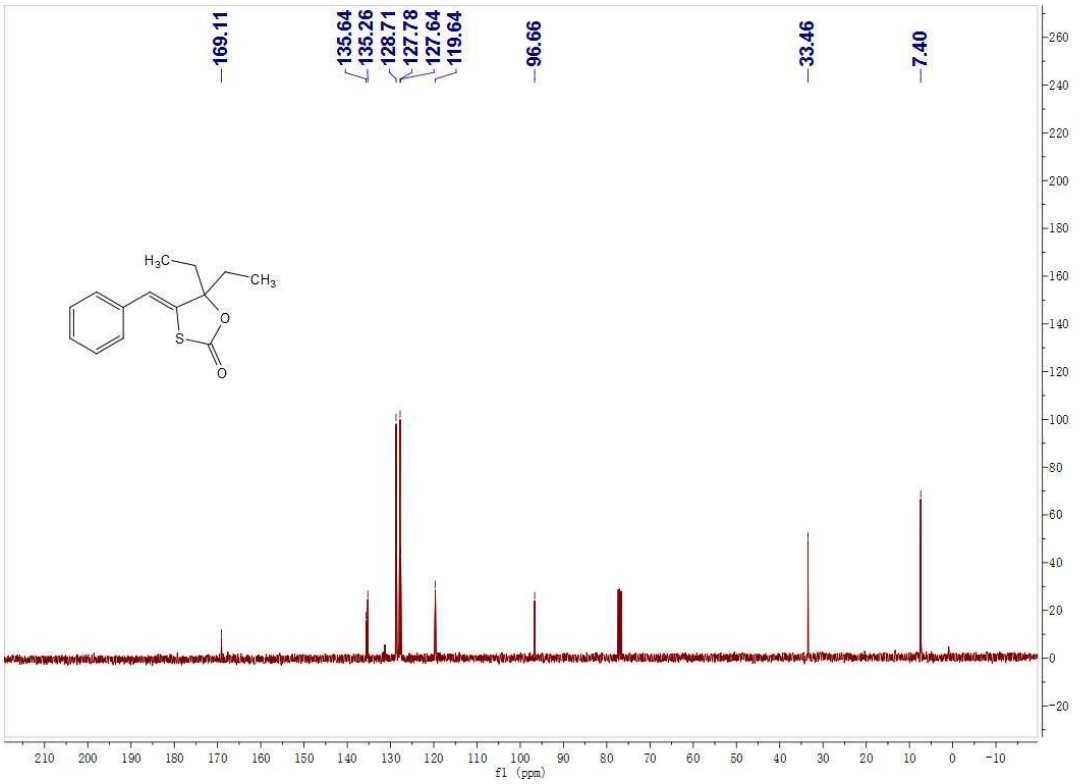
2.08
2.28
6.00
f1 (ppm)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119.64
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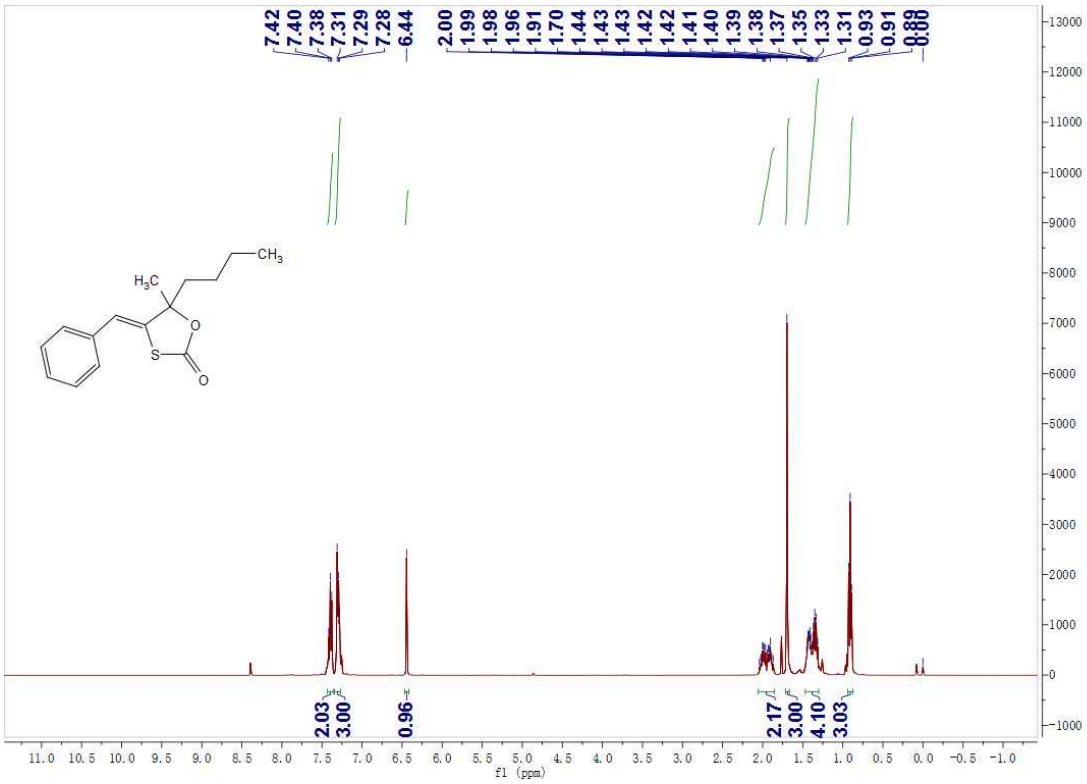
7.40
f1 (ppm)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7.28
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1.96
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1.43
1.42
1.42
1.41
1.40
1.39
1.38
1.37
1.35
1.33
1.31
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0.91
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0.88
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3.00
0.96
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3.00
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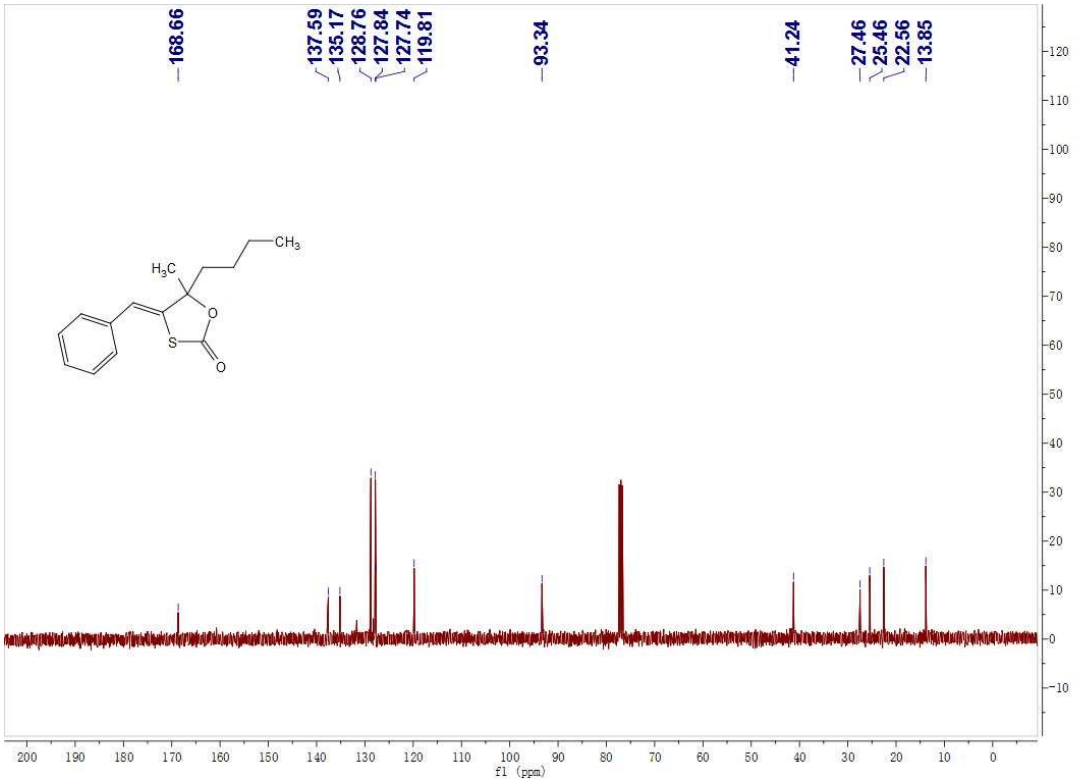
3.03
f1 (ppm)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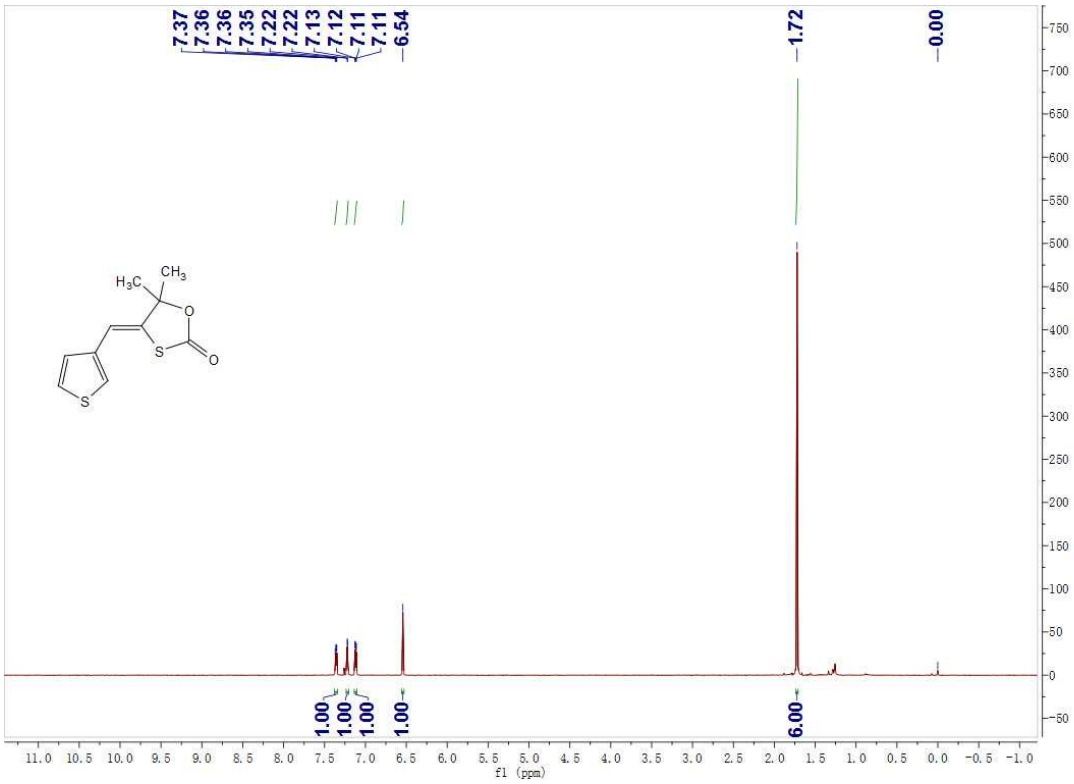
13.85
f1 (ppm)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7.36
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7.22
7.22
7.13
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7.11
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1.00
1.00
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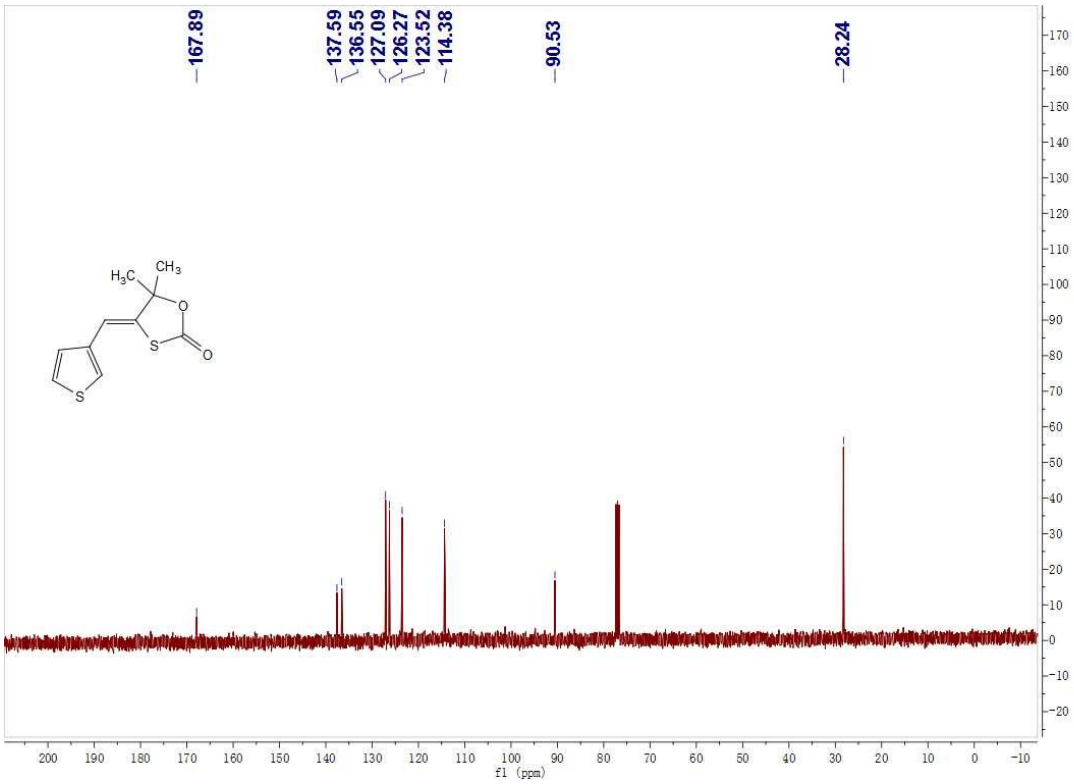
1.00
6.00
f1 (ppm)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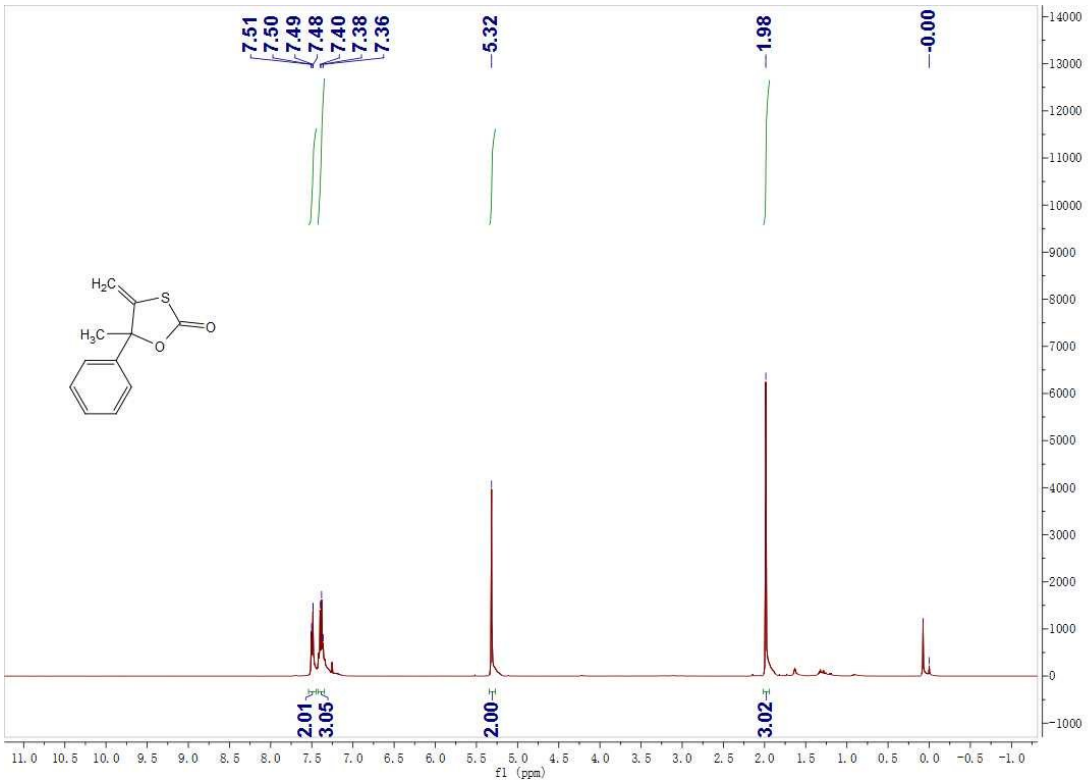
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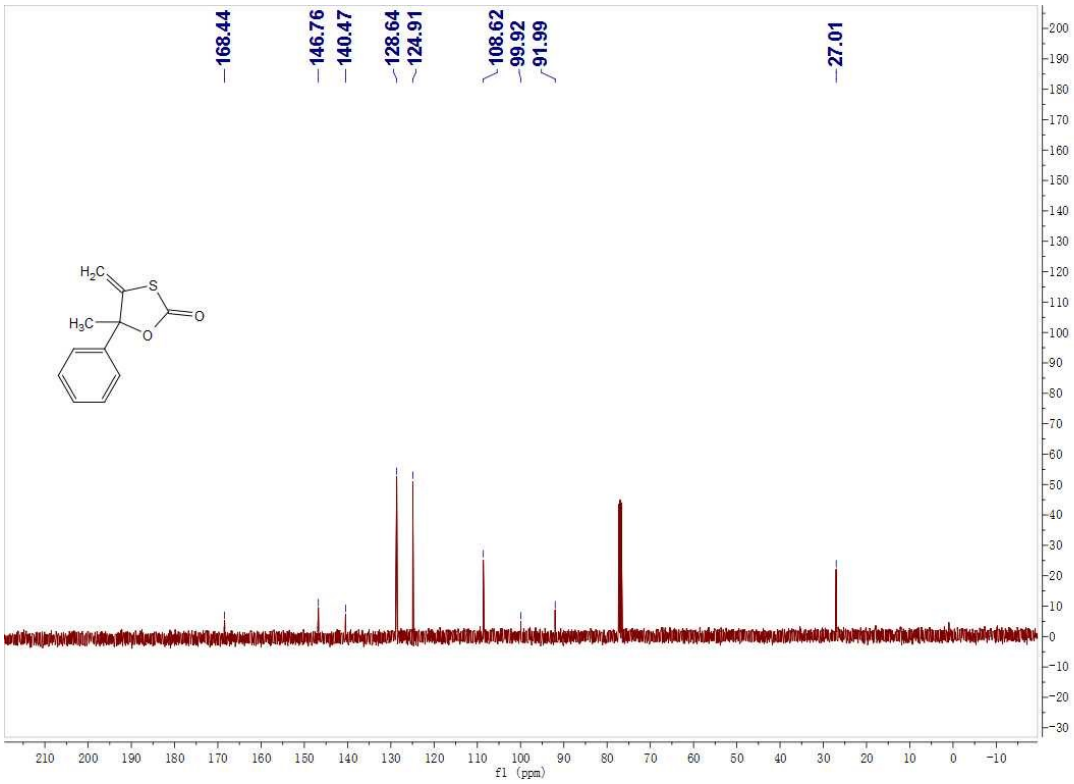
7.36
5.32
1.98
0.00
2.01
3.05
2.00
3.02
f1 (ppm)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99.92
91.99
27.01
f1 (ppm)