Supporting Information

Cobalt-Catalyzed selective hydroacylation of alkynes

Jian Wu, Wen-Xia Gao, Xiao-Bo Huang, Yun-Bing Zhou,* Miao-Chang Liu* and Hua-Yue Wu

College of Chemistry and Materials Engineering, Wenzhou University, Wenzhou, 325035, P. R. of China

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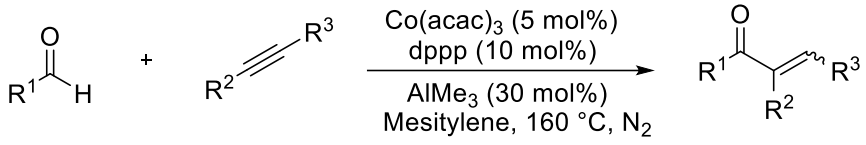
1. General Information

All reagents and solvents were purchased from TCI, Sigma-Aldrich, Alfa Aesar, Acros and Meryer. All reactions were conducted using standard Schlenk techniques. Column chromatography was performed using EM silica gel 60 (300−400 mesh). ^{1}H NMR and ^{13}C NMR spectra were measured on a 500 MHz Bruker AVANCE spectrometer (500 MHz for ^{1}H, 125 MHz for ^{13}C), using $CDCl_3$ as the solvent with tetramethylsilane (TMS) as the internal standard at room temperature. Chemical shifts were reported in ppm. ^{1}H NMR spectra were referenced to $CDCl_3$ (7.26 ppm), and ^{13}C-NMR spectra were referenced to $CDCl_3$ (77.0 ppm) spectra were referenced to $CDCl_3$. Peak multiplicities were designated by the following abbreviations: s, singlet; d, doublet; t, triplet; m, multiplet. Chemical shifts are given in δ relative to TMS, the coupling constants *J* are given in Hz. Analysis of crude reaction mixture was done on the Varian 4000 GC/MS and Agilent 7890A/5975C. High-resolution mass spectra were recorded on a micrOTOF-Q II 10410 mass spectrometer. Unless otherwise noted, all reagents and solvents were obtained commercially and used without further purification.

2. General Experimental Procedures

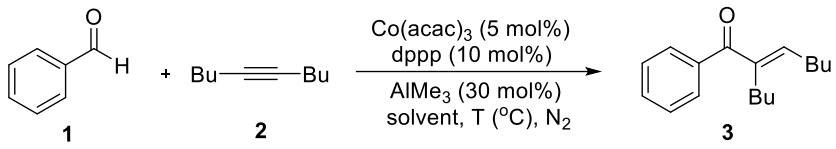


Scheme S1

A 10 mL pressure tube equipped with a stir bar was charged with aldehyde (0.30 mmol), alkyne (0.45 mmol), $Co(acac)_3$ (5.4 mg, 0.015 mmol), dppp (12.3 mg, 0.03 mmol), $AlMe_3$ (2.0 M in hexane, 0.09 mmol) and mesitylene (1.5 mL). The reaction mixture was stirred in a heating mantle preheated to 160 °C under N_2 atmosphere for 5 h. Upon cooling to room temperature, the reaction mixture was diluted with Ethyl acetate. The organic solution was purified by flash chromatography on silica gel to afford the desired product.

3. Screening of Solvent

Table S1. Screening of solvent



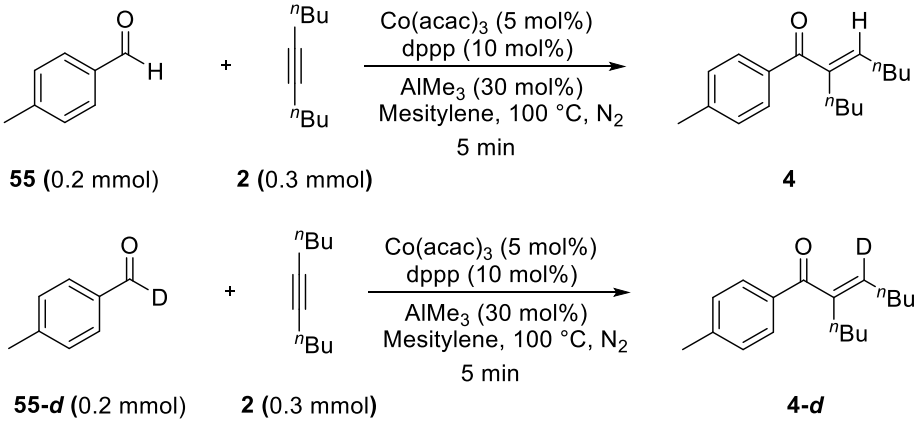
1 2 3

entry	solvent	T (°C)	yield (%)
1	cyclohexane	120	28
2	heptane	120	30
3	toluene	120	62
4	mesitylene	120	60
5	DCE	120	trace
6	THF	120	33
7	dioxane	120	21
8	heptane	160	49
9	toluene	160	57
10	mesitylene	160	82

Reaction conditions: **1** (0.3 mmol), **2** (0.45 mmol), $Co(acac)_3$ (5 mol%), dppp (10 mol%), $AlMe_3$ (30 mol%), solvent (1.5 mL), N_2 atmosphere, 5 h, isolated yield.

4. Mechanistic Experiments

a) Kinetic Isotope Effect (KIE) Experiments



Scheme S2

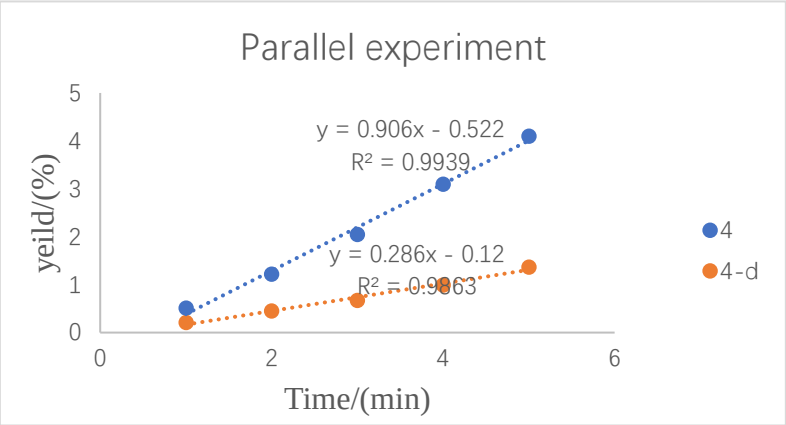
Individual reactions of **55** (0.2 mmol) and **55-*d*** (0.2 mmol) with **2** (0.3 mmol) were set up side by side according to the general procedure in the presence of 1,2-

diphenylethyne as an internal standard for GC analysis. The progress of each reaction was monitored by GC analysis of periodically taken aliquots (0–5 min). The results of this experiment are summarized in Table S1/Figure S1. From the thus determined initial rates, the H/D KIE was calculated to be 3.2.

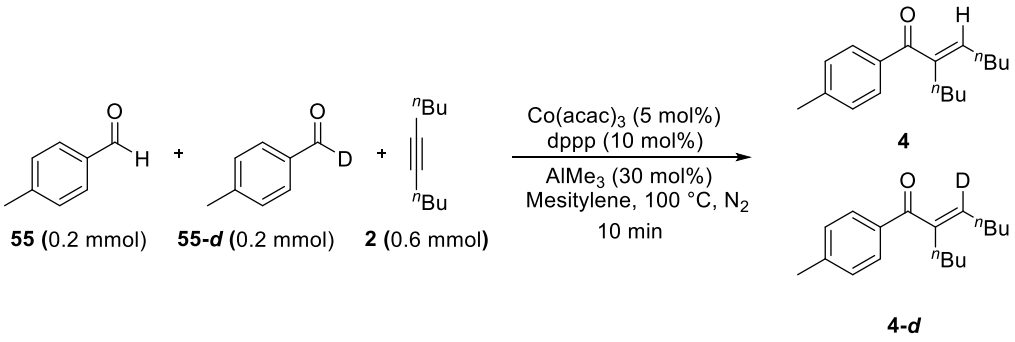
Table S2. Progress of individual reactions of **55** and **55-*d*** with **2**

***t*/min**	1	2	3	4	5
Yield of 4/%	0.51	1.22	2.05	3.1	4.1
Yield of 4-*d*/%	0.21	0.45	0.67	0.99	1.37

Figure S1. Plot of yield (%) against reaction time



b) Intermolecular Competition Reaction

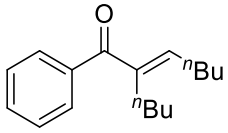


Scheme S3

The reaction of an equimolar mixture of **55** (0.2 mmol) and **55-*d*** (0.2 mmol) with 2 (0.6 mmol) was performed according to the general procedure. The overall yield was determined by GC analysis of the crude product using 1,2-diphenylethyne as an internal standard.

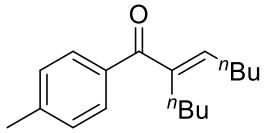
5. Characterization of Products in Details

(*E*)-2-butyl-1-phenylhept-2-en-1-one (3)[6]



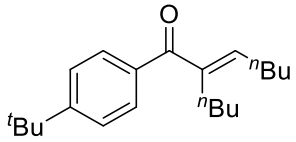
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (60.0 mg, 82%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.0 Hz, 2H), 7.48 (t, *J* = 7.0 Hz, 1H), 7.42-7.36 (m, 2H), 6.18 (t, *J* = 7.5 Hz, 1H), 2.48 (t, *J* = 7.0 Hz, 2H), 2.28 (q, *J* = 7.5 Hz, 2H), 1.44-1.30 (m, 8H), 0.95-0.86 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 198.9, 145.6, 141.2, 139.0, 131.3, 129.3, 127.9, 31.1, 31.0, 28.5, 26.4, 22.7, 22.4, 13.9, 13.8.

(*E*)-2-butyl-1-(p-tolyl)hept-2-en-1-one (4)



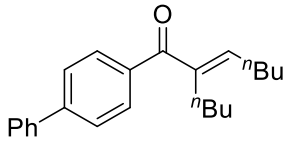
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (65.8 mg, 85%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.21 (d, *J* = 8.0 Hz, 2H), 6.14 (t, *J* = 7.5 Hz, 1H), 2.47 (t, *J* = 7.5 Hz, 2H), 2.39 (s, 3H), 2.27 (q, *J* = 7.5 Hz, 2H), 1.44-1.32 (m, 8H), 0.93-0.88 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 198.7, 144.5, 142.0, 141.2, 136.2, 129.6, 128.6, 31.2, 31.1, 28.4, 26.6, 22.8, 22.5, 21.4, 13.9, 13.8. HRMS: (ESI) calculated for $C_{18}H_{26}ONa$ $[M+Na]^+$ 281.1882, found 281.1887.

(*E*)-2-butyl-1-(4-(tert-butyl)phenyl)hept-2-en-1-one (5)



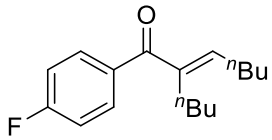
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (66.6 mg, 74%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.64-7.61 (m, 2H), 7.45-7.41 (m, 2H), 6.20-6.16 (m, 1H), 2.49-2.45 (m, 2H), 2.29-2.26 (m, 2H), 1.41-1.33 (m, 17H), 0.93-0.90 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 198.7, 155.0, 144.5, 141.1, 136.1, 129.4, 124.9, 34.9, 31.2, 31.1, 31.0, 28.5, 26.6, 22.8, 22.5, 14.0, 13.9. HRMS: (ESI) calculated for $C_{21}H_{32}ONa$ $[M+Na]^+$ 323.2351, found 323.2355.

(*E*)-1-([1,1'-biphenyl]-4-yl)-2-butylhept-2-en-1-one (6)



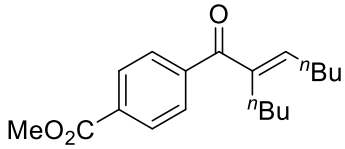
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (68.2 mg, 71%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.67-7.62 (m, 4H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.39 (t, *J* = 7.5 Hz, 1H), 6.25 (t, J = 7.5 Hz, 1H), 2.52 (t, *J* = 7.5 Hz, 2H), 2.32 (q, *J* = 7.5 Hz, 2H), 1.50-1.35 (m, 8H), 0.98-0.91 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 198.6, 145.2, 144.2, 141.3, 140.1, 137.7, 130.0, 128.9, 127.9, 127.2, 126.7, 31.2, 31.1, 28.5, 26.6, 22.8, 22.5, 14.0, 13.9. HRMS: (ESI) calculated for $C_{23}H_{29}O$ $[M+H]^+$ 321.2213, found 321.2218.

(*E*)-2-butyl-1-(4-fluorophenyl)hept-2-en-1-one (7)



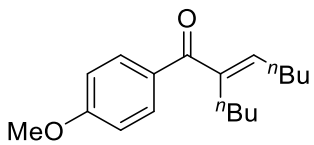
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (63.7 mg, 81%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70-7.65 (m, 2H), 7.12-7.05 (m, 2H), 6.13 (t, *J* = 7.5 Hz, 1H), 2.49-2.43 (m, 2H), 2.31-2.24 (m, 2H), 1.42-1.34 (m, 8H), 0.93-0.88 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 197.5, 164.8 (d, *J* = 250.0 Hz), 145.0, 141.2, 135.2 (d, *J* = 2.5 Hz), 131.8 (d, *J* = 8.8 Hz), 115.1 (d, *J* = 21.3 Hz), 31.2, 31.1, 28.5, 26.6, 22.8, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{17}H_{23}OFNa$ $[M+Na]^+$ 285.1631, found 285.1636.

methyl (*E*)-4-(2-butylhept-2-enoyl)benzoate (8)



Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (34.4 mg, 38%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.0 Hz, 2H), 7.64 (d, *J* = 8.4 Hz, 2H), 6.18 (t, *J* = 7.2 Hz, 1H), 3.94 (s, 3H), 2.49-2.44 (m, 2H), 2.32-2.26 (m, 2H), 1.41-1.31 (m, 8H), 0.95-0.88 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 198.2, 166.5, 147.5, 143.2, 141.4, 132.3, 129.3, 129.0, 52.3, 31.2, 31.0, 28.7, 26.2, 22.8, 22.5, 14.0, 13.9. HRMS: (ESI) calculated for $C_{19}H_{26}O_3Na$ $[M+Na]^+$ 325.1780, found 325.1789.

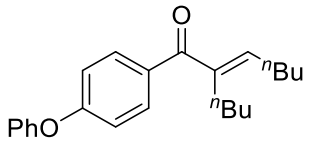
(*E*)-2-butyl-1-(4-methoxyphenyl)hept-2-en-1-one (12)



Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (73.1 mg, 89%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 6.08 (t, *J* = 7.2 Hz, 1H), 3.85 (s, 3H), 2.46 (t, *J* = 7.2 Hz, 2H), 2.30-2.23 (m, 2H), 1.41-1.33 (m, 8H), 0.92-0.87 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 198.0, 162.5, 143.1, 141.1, 131.7, 131.4, 113.3, 55.4, 31.2, 31.1, 28.3,

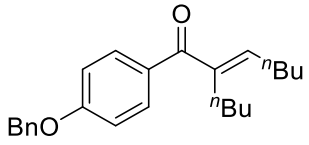
26.9, 22.8, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{18}H_{26}O_2Na$ $[M+Na]^+$ 297.1831, found 297.1847.

(*E*)-2-butyl-1-(4-phenoxyphenyl)hept-2-en-1-one (13)



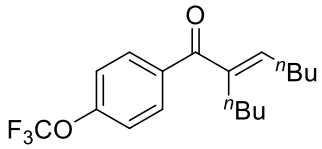
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (85.6 mg, 85%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71-7.66 (m, 2H), 7.41-7.35 (m, 2H), 7.19-7.15 (m, 1H), 7.08-7.05 (m, 2H), 6.99-6.96 (m, 2H), 6.15 (t, *J* = 7.2 Hz, 1H), 2.50-2.45 (m, 2H), 2.31-2.24 (m, 2H), 1.43-1.34 (m, 8H), 0.94-0.90 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 197.8, 160.8, 155.9, 143.9, 141.2, 133.4, 131.7, 129.9, 124.3, 120.0, 117.1, 31.2, 31.1, 28.5, 26.8, 22.8, 22.6, 14.0, 13.9. HRMS: (ESI) calculated for $C_{23}H2_8O_2Na$ $[M+Na]^+$ 359.1987, found 359.1991.

(*E*)-1-(4-(benzyloxy)phenyl)-2-butylhept-2-en-1-one (14)



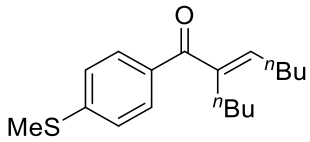
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (78.7mg, 75%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.5 Hz, 2H), 7.46-7.37 (m, 4H), 7.36-7.32 (m, 1H), 7.00 (d, *J* = 9.0 Hz, 2H), 6.12 (t, *J* = 7.5 Hz, 1H), 2.50 (t, *J* = 7.0 Hz, 2H), 2.29 (q, *J* = 7.5 Hz, 2H), 1.45-1.35 (m, 8H), 0.96-0.91 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 197.8, 161.7, 143.1, 141.1, 136.3, 131.7, 131.5, 128.6, 128.1, 127.4, 114.1, 70.0, 31.2, 31.1, 28.3, 26.8, 22.7, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{24}H_{30}O_2Na$ $[M+Na]^+$ 373.2144, found 373.2145.

(*E*)-2-butyl-1-(4-(trifluoromethoxy)phenyl)hept-2-en-1-one (15)



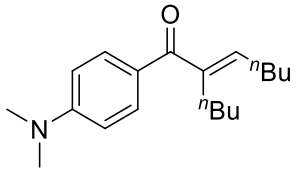
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (83.6 mg, 85%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.73-7.67 (m, 2H), 7.28-7.22 (m, 2H), 6.18 (t, *J* = 8.0 Hz, 1H), 2.51-2.44 (m, 2H), 2.33-2.26 (m, 2H), 1.45-1.32 (m, 8H), 0.95-0.89 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 197.4, 151.5, 151.4 146.0, 141.2, 137.4, 131.1, 120.1, 31.2, 31.1, 28.6, 26.5, 22.8, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{18}H_{24}O_2F_3$ $[M+H]^+$ 329.1723, found 329.1727.

(*E*)-2-butyl-1-(4-(methylthio)phenyl)hept-2-en-1-one (16)



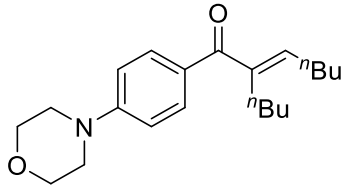
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (73.9 mg, 85%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 6.0 Hz, 2H), 7.22 (d, *J* = 6.4 Hz, 2H), 6.12 (t, *J* = 6.0 Hz, 1H), 2.49 (s, 3H), 2.48-2.43 (m, 2H), 2.29-2.23 (m, 2H), 1.41-1.31 (m, 8H), 0.93-0.87 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 198.0, 144.2, 143.7, 141.1, 135.1, 129.9, 124.8, 31.1, 28.4, 26.7, 22.7, 22.5, 14.9, 13.9, 13.8, 13.7. HRMS: (ESI) calculated for $C_{18}H_{26}OSNa$ $[M+Na]^+$ 313.1602, found 313.1602.

(*E*)-2-butyl-1-(4-(dimethylamino)phenyl)hept-2-en-1-one (17)



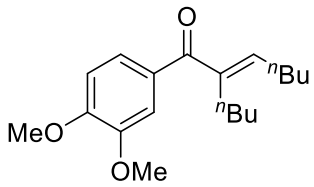
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (74.0 mg, 86%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77-7.68 (m, 2H), 6.69-6.59 (m, 2H), 6.02-5.95 (m, 1H), 3.10-2.99 (m, 6H), 2.49-2.42 (m, 2H), 2.31-2.18 (m, 2H), 1.45-1.30 (m, 8H), 0.97-0.84 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 197.9, 152.9, 141.0, 140.1, 132.0, 125.9, 110.5, 40.0, 31.4, 31.1, 28.1, 27.4, 22.8, 22.5, 14.0, 13.9. HRMS: (ESI) calculated for $C_{19}H_{29}ONNa$ $[M+Na]^+$ 310.2147, found 310.2151.

(E)-2-butyl-1-(4-morpholinophenyl)hept-2-en-1-one (18)



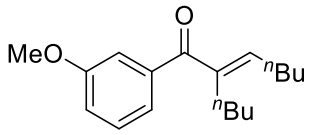
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (92.7 mg, 94%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.0 Hz, 2H), 6.04 (t, *J* = 7.5 Hz, 1H), 3.86-3.76 (m, 4H), 3.30-3.21 (m, 4H), 2.45 (t, *J* = 8.0 Hz, 2H), 2.24 (q, *J* = 7.5 Hz, 2H), 1.43-1.30 (m, 8H), 0.93-0.85 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 197.7, 153.5, 141.7, 141.0, 131.6, 129.1, 113.2, 66.5, 47.8, 31.2, 31.1, 28.2, 27.0, 22.7, 22.4, 13.9, 13.8. HRMS: (ESI) calculated for $C_{21}H_{31}ON_2Na$ $[M+Na]^+$ 352.2253, found 352.2258.

(E)-2-butyl-1-(3,4-dimethoxyphenyl)hept-2-en-1-one (19)



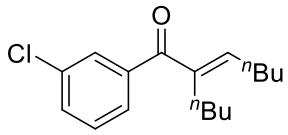
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (64.7 mg, 71%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37-7.28 (m, 2H), 6.90-6.82 (m, 1H), 6.14-6.06 (m, 1H), 3.95-3.87 (m, 6H), 2.51-2.42 (m, 2H), 2.33-2.22 (m, 2H), 1.47-1.28 (m, 8H), 0.95-0.85 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 197.8, 152.2, 148.6, 142.8, 140.8, 131.3, 124.1, 111.9, 109.5, 55.8, 55.7, 31.1, 31.0, 28.2, 26.9, 22.7, 22.4, 13.9, 13.8. HRMS: (ESI) calculated for $C_{19}H_{28}O_3Na$ $[M+Na]^+$ 327.1936, found 327.1933.

(E)-2-butyl-1-(3-methoxyphenyl)hept-2-en-1-one (20)



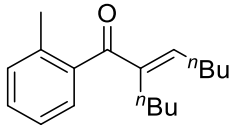
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (50.1 mg, 61%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33-7.30 (m, 1H), 7.21-7.18 (m, 2H), 7.05-7.01 (m, 1H), 6.20 (t, *J* = 7.5 Hz, 1H), 3.83 (s, 3H), 2.48 (t, *J* = 7.5 Hz, 2H), 2.28 (q, *J* = 7.0 Hz, 2H), 1.43-1.30 (m, 8H), 0.93-0.88 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 198.7, 159.3, 145.7, 141.1, 140.4, 128.9, 121.9, 117.6, 113.9, 55.3, 31.2, 31.1, 28.5, 26.5, 22.8, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{18}H_{26}O_2Na$ $[M+Na]^+$ 297.1831, found 297.1847.

(E)-2-butyl-1-(3-chlorophenyl)hept-2-en-1-one (21)



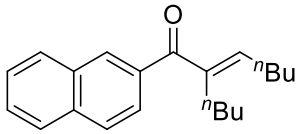
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (60.0 mg, 98%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61-7.58 (m, 1H), 7.50-7.44 (m, 2H), 7.37-7.31 (m, 1H), 6.20-6.15 (m, 1H), 2.49-2.43 (m, 2H), 2.32-2.25 (m, 2H), 1.43-1.32 (m, 8H), 0.95-0.88 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 197.3, 146.7, 141.1, 140.8, 134.2, 131.3, 129.3, 129.2, 127.3, 31.2, 31.0, 28.6, 26.4, 22.8, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{17}H_{23}ClONa$ $[M+Na]^+$ 301.1335, found 301.1328.

(E)-2-butyl-1-(o-tolyl)hept-2-en-1-one (22)



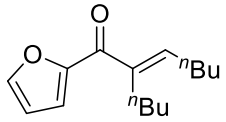
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (28.6 mg, 37%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.32-7.24 (m, 1H), 7.21-7.12 (m, 3H), 6.18 (t, *J* = 7.5 Hz, 1H), 2.50-2.41 (m, 2H), 2.28-2.18 (m, 5H), 1.44-1.24 (m, 8H), 0.98-0.83 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 200.7, 149.1, 142.8, 140.2, 135.6, 130.5, 129.1, 127.5, 125.0, 31.4, 30.9, 28.8, 25.3, 22.9, 22.5, 19.5, 14.0, 13.8. HRMS: (ESI) calculated for $C_{15}H_8OS_2F_2Na$ $[M+Na]^+$ 328.9883, found 328.9895.

(*E*)-2-butyl-1-(naphthalen-2-yl)hept-2-en-1-one (25)



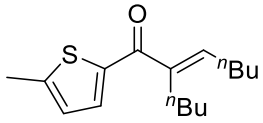
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (44.9 mg, 51%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.94-7.86 (m, 3H), 7.80-7.76 (m, 1H), 7.60-7.51 (m, 2H), 6.26 (t, *J* = 7.5 Hz, 1H), 2.55 (t, *J* = 7.0 Hz, 2H), 2.33 (q, *J* = 7.5 Hz, 2H), 1.52-1.34 (m, 8H), 0.99-0.95 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 199.0, 145.5, 141.4, 136.3, 134.8, 132.3, 130.4, 129.1, 127.9, 127.7, 126.5, 125.8, 31.3, 31.0, 28.6, 26.7, 22.9, 22.5, 14.0, 13.9. HRMS: (ESI) calculated for $C_{21}H_{26}ONa$ $[M+Na]^+$ 317.1882, found 317.1885.

(*E*)-2-butyl-1-(furan-2-yl)hept-2-en-1-one (26)



Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (50.5 mg, 72%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 (s, 1H), 7.02-6.98 (m, 1H), 6.51-6.44 (m, 2H), 2.48-2.39 (m, 2H), 2.33-2.24 (m, 2H), 1.47-1.28 (m, 8H), 0.97-0.84 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 185.1, 152.4, 146.4, 142.5, 140.9, 118.9, 111.5, 31.2, 31.1, 28.4. 26.7, 22.7, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{15}H_{22}O_2Na$ $[M+Na]^+$ 257.1518, found 257.1508.

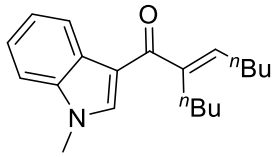
(*E*)-2-butyl-1-(5-methylthiophen-2-yl)hept-2-en-1-one (27)



Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (66.5 mg, 84%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 4.5 Hz, 1H),6.77-6.72 (m, 1H), 6.33-6.24 (m, 1H), 2.51 (s, 3H), 2.45-2.39 (m, 2H), 2.30-2.20 (m, 2H), 1.46-1.28 (m, 8H), 0.96-0.83 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 190.3, 148.9, 142.2, 141.2, 140.8, 133.8, 126.2, 31.3, 31.1, 28.2, 27.2, 22.7, 22.5, 15.8,

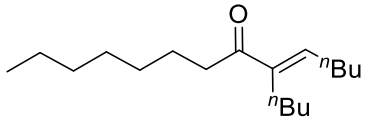
14.0, 13.9. HRMS: (ESI) calculated for $C_{16}H_{24}OSNa$ $[M+Na]^+$ 287.1446, found 287.1444.

(*E*)-2-butyl-1-(1-methyl-1H-indol-3-yl)hept-2-en-1-one (28)



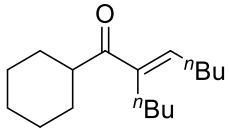
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (41.0 mg, 46%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.45-8.38 (m, 1H), 7.62-7.51 (m, 1H), 7.41-7.33 (m, 1H), 2.51-2.44 (m, 2H), 2.33-2.26 (m, 2H), 1.45-1.32 (m, 8H), 0.95-0.89 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 193.6, 143.1, 138.0, 137.5, 136.5, 127.1, 123.2, 122.6, 122.2, 116.1, 109.4, 33.4, 31.5, 31.2, 28.1, 27.3, 22.9, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{20}H_{27}NONa$ $[M+Na]^+$ 320.1991, found 320.1995.

(*E*)-6-butyltetradec-5-en-7-one (29)



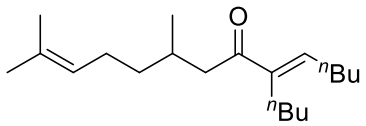
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (55.0 mg, 69%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.57-6.51 (m, 1H), 2.63-2.57 (m, 2H), 2.28-2.20 (m, 4H), 1.44-1.26 (m, 18H), 0.95-0.84 (m, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 220.2, 142.3, 142.0, 37.4, 31.7, 31.5, 31.2, 29.4, 29.1, 28.6, 25.5, 25.1, 22.9, 22.6, 22.5, 14.0, 13.9, 13.8. HRMS: (ESI) calculated for $C_{18}H_{34}ONa$ $[M+Na]^+$ 289.2508, found 289.2501.

(*E*)-2-butyl-1-cyclohexylhept-2-en-1-one (30)



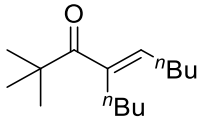
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (59.2 mg, 79%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 6.48 (t, J = 7.0 Hz, 1H), 2.97-2.90 (m, 1H), 2.25-2.17 (m, 4H), 1.76-1.70 (m, 2H), 1.69-1.60 (m, 3H), 1.46-1.13 (m, 13H), 0.90-0.78 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 205.4, 141.1, 141.0, 44.3, 31.4, 31.2, 29.8, 28.5, 25.9, 25.8, 25.5, 22.8, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{17}H_{31}O$ $[M+H]^+$ 251.2370, found 251.2363.

(*E*)-6-butyl-9,13-dimethyltetradeca-5,12-dien-7-one (31)



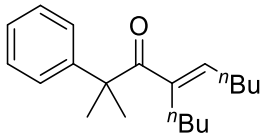
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (48.1 mg, 55%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.60-6.46 (m, 1H), 5.49-5.20 (m, 1H), 2.66-2.56 (m, 1H), 2.47-2.37 (m, 4H), 2.32-2.19 (m, 4H), 2.09-1.90 (m, 4H), 1.73-1.53 (m, 6H), 1.44-1.18 (m, 9H), 0.98-0.83 (m, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ202.0, 142.6, 142.4, 131.3, 124.5, 44.7, 37.2, 31.5, 31.2, 30.1, 28.6, 28.5, 25.7, 22.9, 22.5, 19.9, 17.6, 13.9, 13.8. HRMS: (ESI) calculated for $C_{20}H_{36}ONa$ $[M+Na]^+$ 315.2664, found 315.2665.

(*E*)-4-butyl-2,2-dimethylnon-4-en-3-one (32)



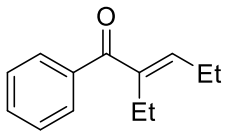
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (30.2 mg, 45%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 6.11-5.94 (m, 1H), 2.34-2.07 (m, 4H), 1.44-1.14 (m, 17H), 0.99-0.75 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 211.4, 140.9, 135.4, 43.8, 31.4, 31.3, 28.6, 27.9, 27.8, 22.8, 22.5, 13.9, 13.8. HRMS: (ESI) calculated for $C_{15}H_{28}ONa$ $[M+Na]^+$ 247.2038, found 247.2031.

(*E*)-4-butyl-2-methyl-2-phenylnon-4-en-3-one (33)



Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (50.6 mg, 59%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ7.35-7.29 (m, 3H), 7.24-7.20 (m, 2H), 5.83-5.80 (m, 1H), 2.26-2.20 (m, 2H), 2.00-1.95 (m, 2H), 1.55-1.52 (m, 6H), 1.32-1.24 (m, 4H), 1.10-1.04 (m, 4H), 0.93-0.90 (m, 3H), 0.82-0.76 (m, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 205.3, 146.9, 144.0, 138.6, 128.6, 126.1, 125.3, 51.0, 31.4, 30.8, 28.6, 28.0, 26.7, 22.9, 22.0, 13.9, 13.8. HRMS: (ESI) calculated for $C_{20}H_{30}ONa$ $[M+Na]^+$ 309.2195, found 309.2191.

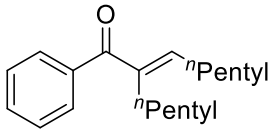
(*E*)-2-ethyl-1-phenylpent-2-en-1-one (34)[7]



Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (40.0 mg, 71%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.65-7.61 (m, 2H), 7.52-7.47 (m, 1H), 7.44-7.38 (m, 2H), 6.17 (t, *J* = 7.2 Hz, 1H), 2.48 (q, *J* = 7.6 Hz,

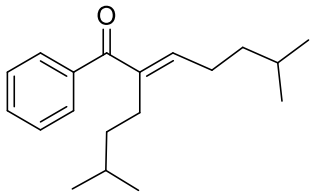
2H), 2.34-2.25 (m, 2H), 1.08-1.01 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 199.0, 146.7, 142.0, 139.2, 131.3, 129.3, 128.0, 22.0, 19.9, 13.7, 13.5.

(*E*)-2-Pentyl-1-phenylhept-2-en-1-one (35)



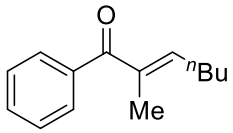
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (61.5 mg, 82%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.67-7.62 (m, 2H), 7.52-7.46 (m, 1H), 7.44-7.37 (m, 2H), 6.18 (t, *J* = 7.2 Hz, 1H), 2.51-2.42 (m, 2H), 2.32-2.23 (m, 2H), 1.48-1.39 (m, 4H), 1.38-1.25 (m, 8H), 0.95-0.84 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ199.0, 145.7, 141.2, 139.1, 131.3, 129.3, 127.9, 31.9, 31.6, 28.8, 28.6, 26.7, 22.5, 22.4, 22.3, 14.0, 13.9. HRMS: (ESI) calculated for $C_{19}H_{28}ONa$ $[M+Na]^+$ 295.2038, found 295.2035.

(*E*)-2-isopentyl-6-methyl-1-phenylhept-2-en-1-one (36)



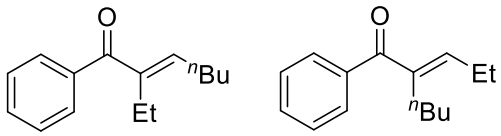
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (67.7 mg, 83%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.65-7.62 (m, 2H), 7.51-7.47 (m, 1H), 7.43-7.37 (m, 2H), 6.16 (t, *J* = 7.5 Hz, 1H), 2.50-2.45 (m, 2H), 2.27 (q, *J* = 8.0 Hz, 2H), 1.64-1.55 (m, 2H), 1.34-1.28 (m, 4H), 0.94-0.90 (m, 12H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ198.9, 145.4, 141.4, 139.1, 131.3, 129.3, 128.0, 38.2, 38.1, 28.3, 27.9, 26.7, 24.7, 22.5, 22.4. HRMS: (ESI) calculated for $C_{19}H_{28}ONa$ $[M+Na]^+$ 295.2038, found 295.2033.

(*E*)-2-methyl-1-phenylhept-2-en-1-one (37)[7]



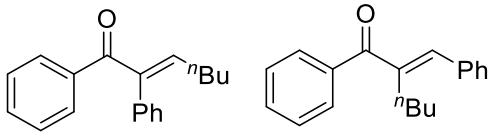
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (67.7 mg, 83%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.63-7.60 (m, 2H), 7.50-7.42 (m, 1H), 7.42-7.38 (m, 2H), 6.31-6.27 (m, 1H), 2.29-2.24 (m, 2H), 2.00 (s, 3H), 1.41-1.33 (m, 4H), 0.94-0.89 (m, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ199.0, 146.8, 138.9, 136.4, 131.2, 129.2, 128.0, 30.7, 28.8, 22.5, 13.8, 12.4.

(*E*)-1-phenyl-2-propylidenehexan-1-one and (*E*)-2-ethyl-1-phenylhept-2-en-1-one (39 and 40)



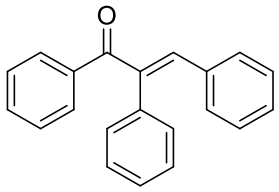
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (43.4 mg, 67%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.69-7.60 (m, 2H), 7.52-7.37 (m, 3H), 6.23-6.91 (m, 1H), 2.55-2.44 (m, 2H), 2.35-2.24 (m, 2H), 1.48-1.27 (m, 4H), 1.10-0.99 (m, 3H), 0.96-0.85 (m, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ199.0, 198.9, 146.9, 145.5, 142.5, 140.7, 139.2, 131.4, 131.3, 129.2, 128.0, 31.3, 31.1, 28.4, 26.4, 22.8, 22.5, 22.2, 20.0, 13.9, 13.8, 13.6, 13.5. HRMS: (ESI) calculated for $C_{15}H_{20}ONa$ $[M+Na]^+$ 239.1412, found 239.1415.

(*E*)-1,2-diphenylhept-2-en-1-one and (*E*)-2-benzylidene-1-phenylhexan-1-one (41 and 42)[8]



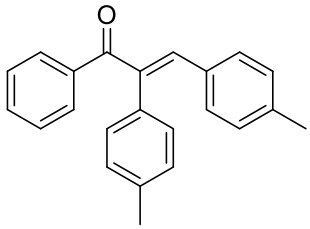
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (51.4 mg, 65%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.98-7.93 (m, 2H), 7.89-7.84 (m, 2H),7.55-7.49 (m, 1H), 7.43-7.38 (m, 4H), 7.34-7.22 (m, 10H), 7.13-7.03 (m, 5H), 6.72 (s, 1H), 6.26 (t, *J* = 7.5 Hz, 1H), 2.49 (t, *J* = 7.5 Hz, 2H), 2.08 (q, *J* = 7.5 Hz, 2H), 1.44-1.35 (m, 4H), 1.32-1.23 (m, 4H), 0.91 (t, *J* = 7.5 Hz, 3H), 0.82 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ200.9, 198.6, 141.9, 140.8, 137.4, 137.0, 135.9, 135.8, 133.4, 133.1, 132.6, 129.6, 129.4, 128.7, 128.6, 128.5, 128.4, 128.1, 127.6, 127.3, 126.0, 36.5, 31.5, 30.3, 29.6, 22.4, 22.2, 13.8, 13.7.

(*E*)-1,2,3-triphenylprop-2-en-1-one(43)[9]



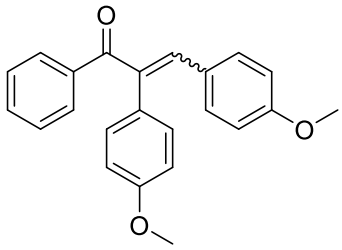
Following the general procedure, using 15 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (37.5 mg, 44%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86 (d, J = 5.6 Hz, 2H), 7.56-7.51 (m, 1H), 7.48-7.42 (m, 2H), 7.38-7.31 (m, 3H), 7.30-7.25 (m, 2H), 7.23-7.15 (m, 4H), 7.11-7.06 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ197.5, 140.8, 140.0, 138.2, 136.5, 134.8, 132.1, 130.3, 129.8, 129.6, 128.9, 128.7, 128.3, 128.2, 127.9.

(*E*)-1-phenyl-2,3-di-p-tolylprop-2-en-1-one (44)[9]



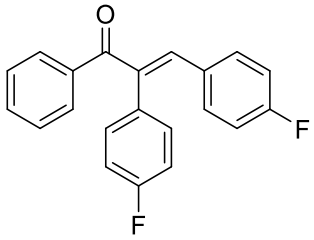
Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (27.3 mg, 43%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86 (d, J = 6.0 Hz, 2H), 7.53-7.48 (m, 1H), 7.45-7.39 (m, 2H), 7.21-7.17 (m, 1H), 7.17-7.14 (m, 4H), 7.03-6.96 (m, 4H), 2.36 (s, 3H), 2.28 (s, 3H) ; **^{13}C NMR** (125 MHz, $CDCl_3$) δ197.8, 140.2, 140.0, 139.1, 138.5, 137.5, 133.7, 132.1, 131.9, 130.3, 129.7, 129.5, 129.4, 129.0, 128.2, 21.3, 21.2.

(*E*/*Z*)-2,3-bis(4-methoxyphenyl)-1-phenylprop-2-en-1-one (45)[9]



Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (49.5 mg, 48%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.06-8.00 (m, 1H), 7.88-7.81 (m, 1H), 7.57-7.36 (m, 5H), 7.27-7.20 (m, 2H), 7.12-7.08 (m, 1H), 6.97-6.86 (m, 2H), 6.78-6.71 (m, 2H), 3.88-3.74 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ199.9, 197.9, 160.2, 159.5, 159.2, 159.1, 140.6, 138.8, 138.4, 138.1, 136.5, 133.5, 132.1, 131.7, 131.0, 130.1, 129.7, 129.6, 128.7, 128.1, 128.0, 127.4, 114.2, 113.9, 113.7, 55.3, 55.2, 55.1, 55.0.

(*E*)-2,3-bis(4-fluorophenyl)-1-phenylprop-2-en-1-one (46)[9]



Following the general procedure, using 20 / 1 petroleum ether / EtOAc as the eluant afford a colorless liquid (43.2 mg, 98%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.92-7.85 (m, 2H), 7.65-7.57 (m, 1H), 7.56-7.48 (m, 2H), 7.34-7.26 (m, 3H), 7.17-7.08 (m, 4H), 6.99-6.90 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ197.2, 162.9 (d, J = 250.0 Hz), 162.5 (d, J = 250.0 Hz), 139.5, 139.4, 138.0, 132.3, 132.2, 132.1, 132.0, 130.5 (d, J = 8.8 Hz), 130.8, 130.7, 129.7, 128.3, 116.0 (d, J = 21.3 Hz), 115.5 (d, J = 22.5 Hz).

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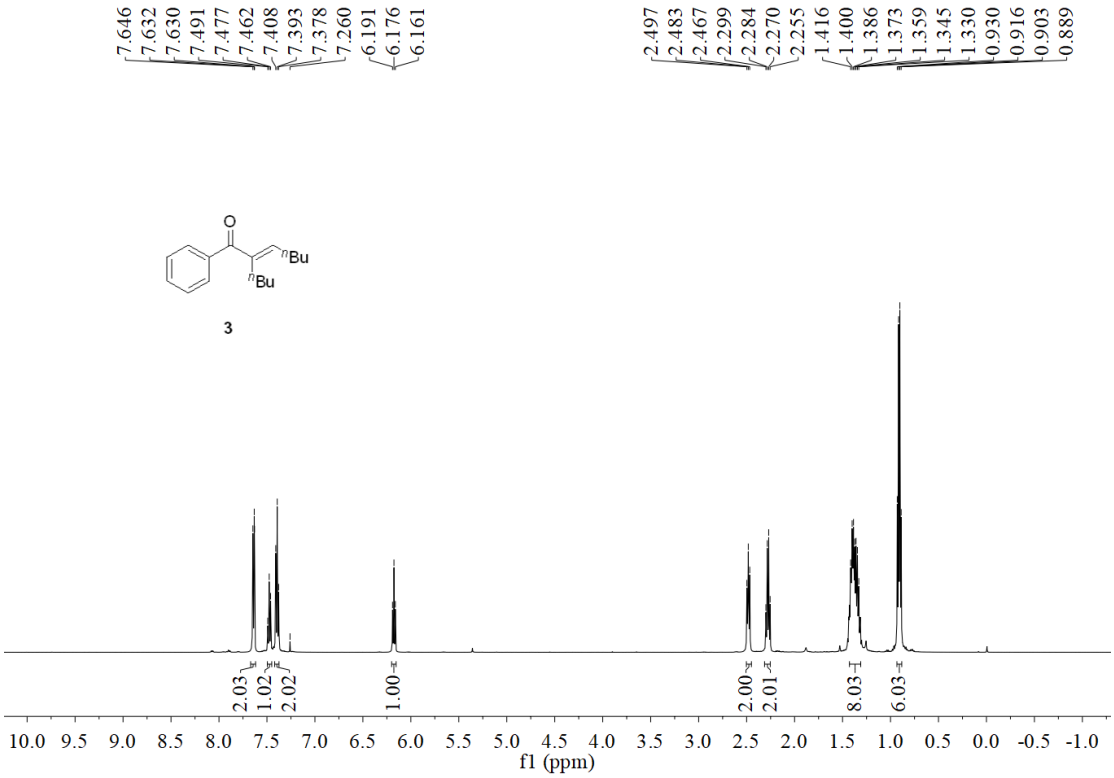
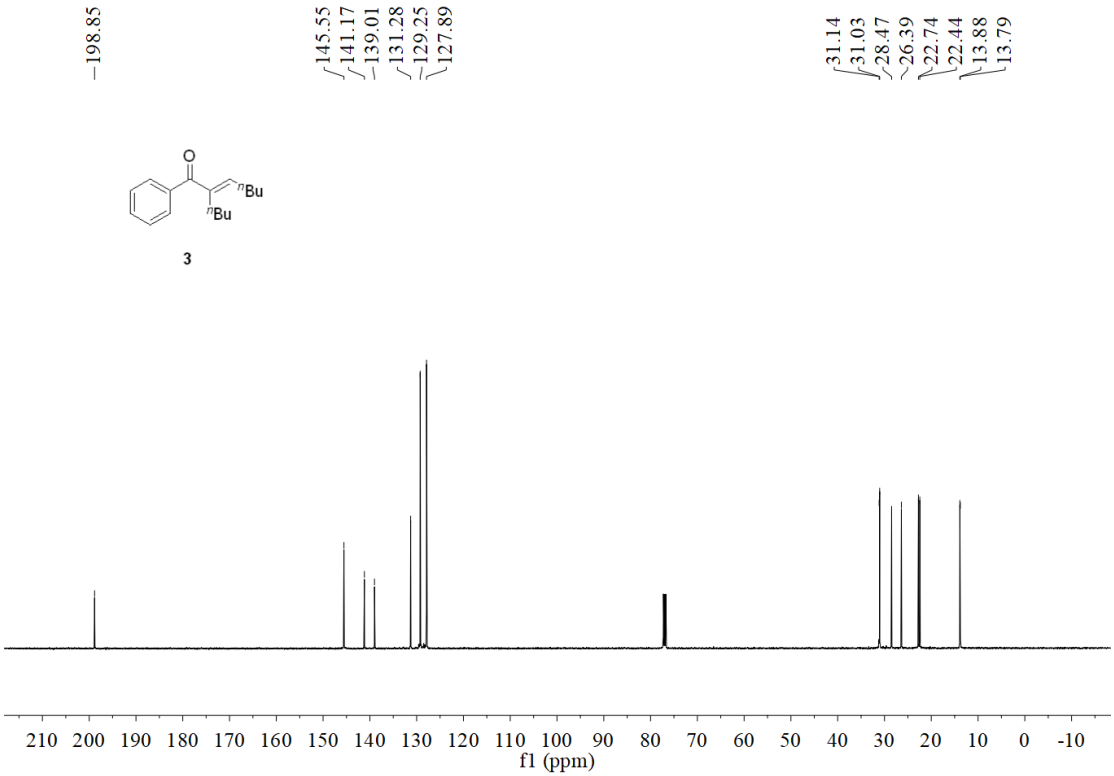
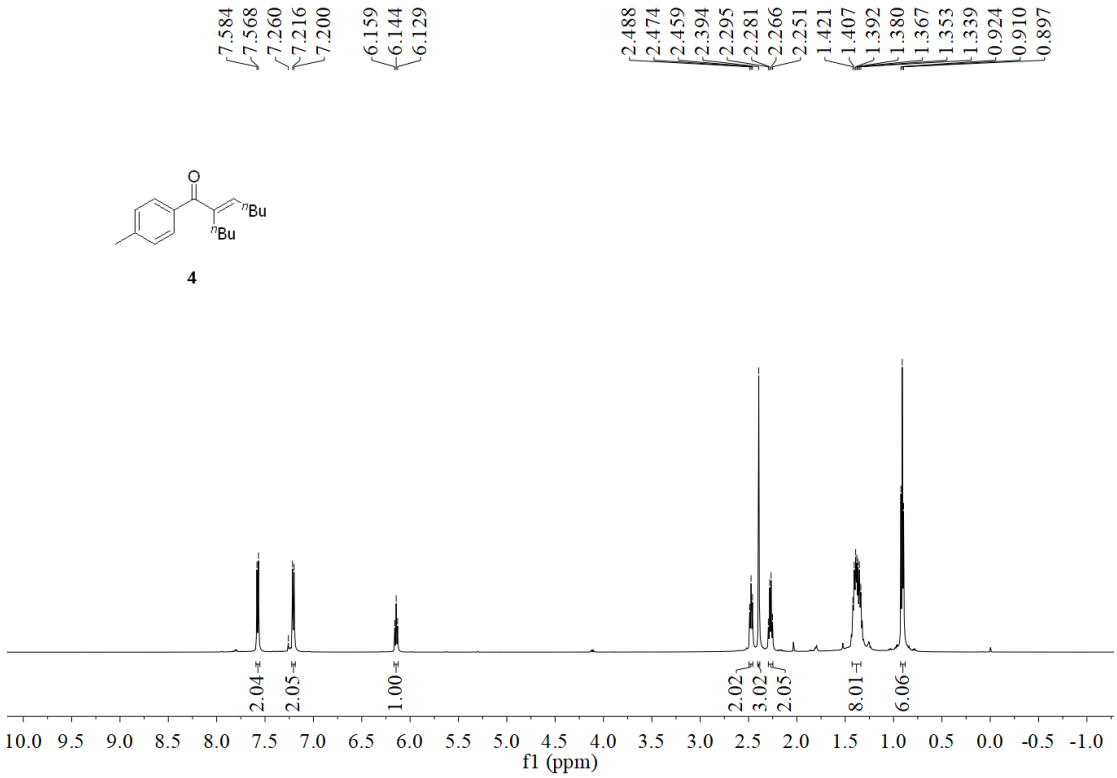
6. NMR Spectra



(500 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



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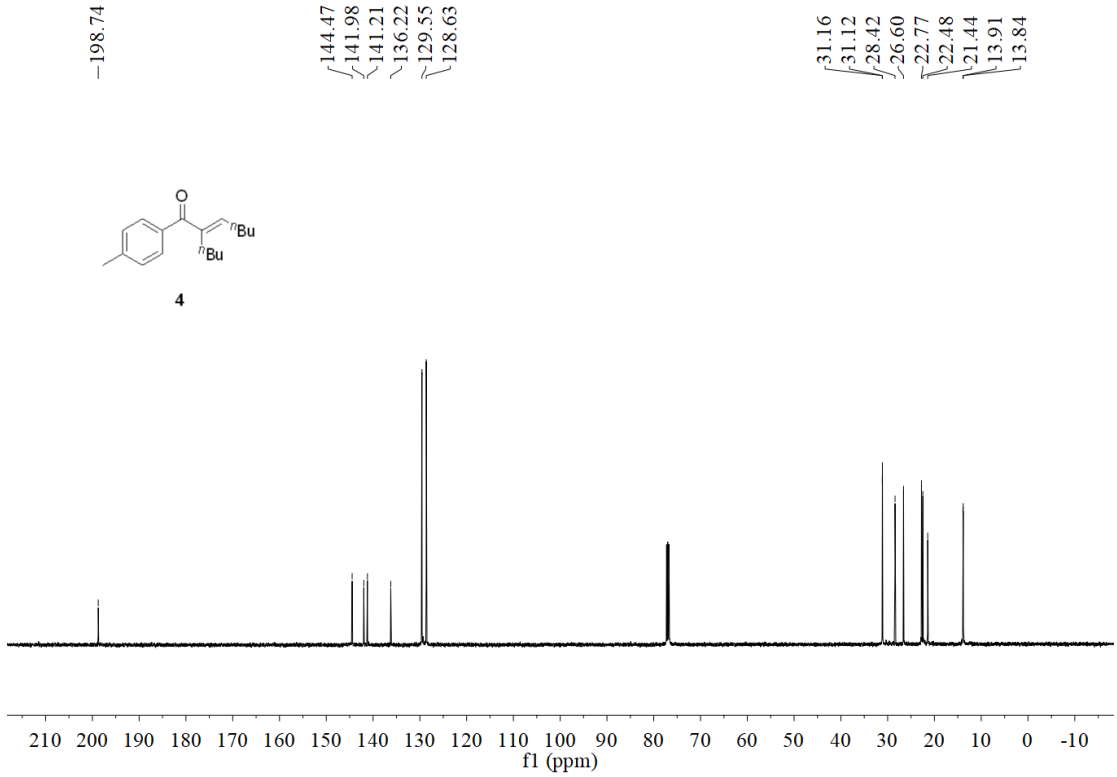
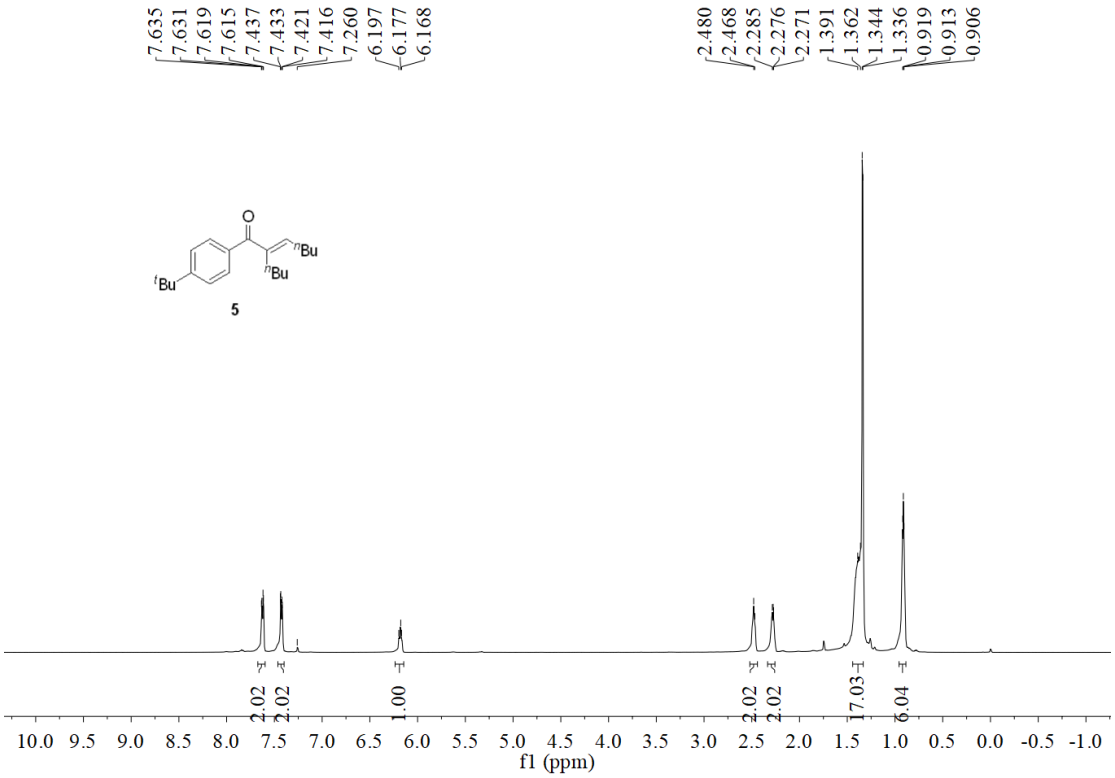
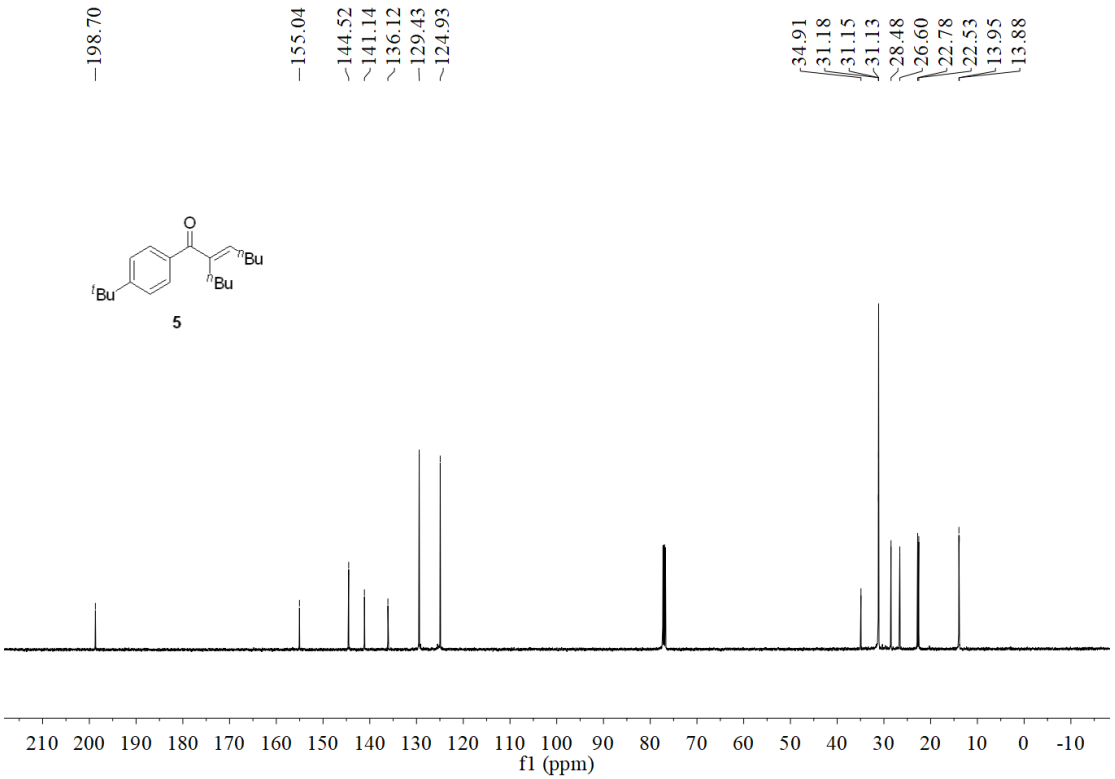


(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



(500 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



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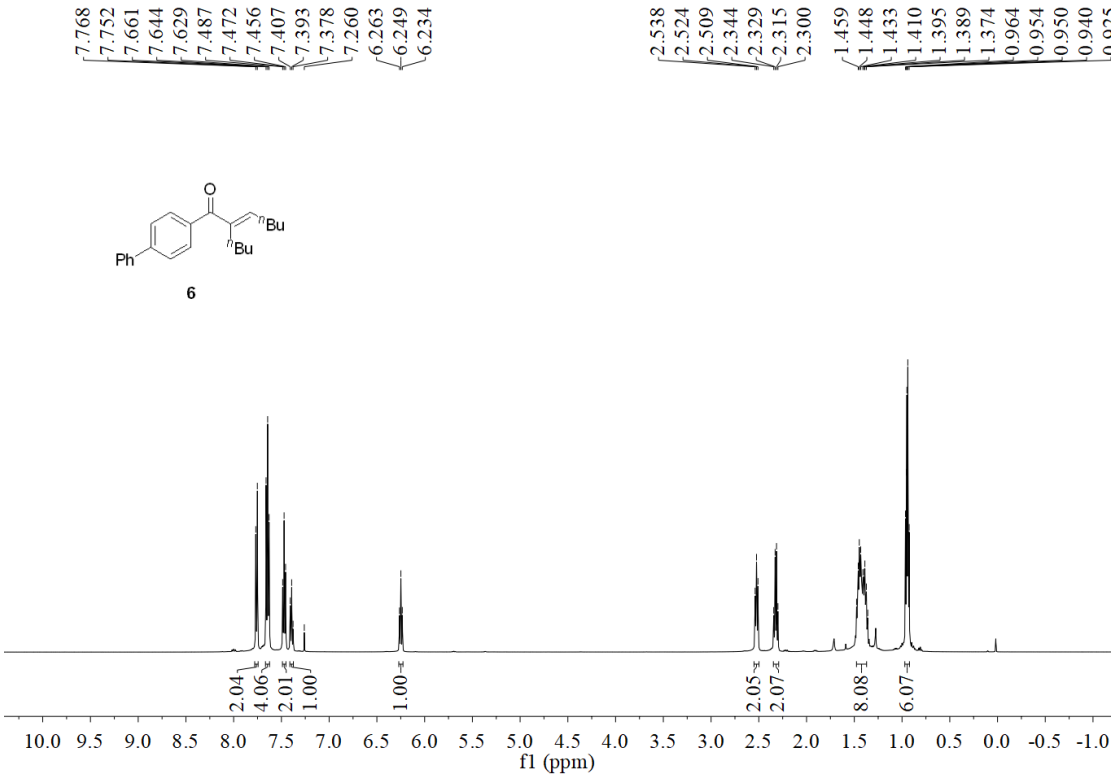

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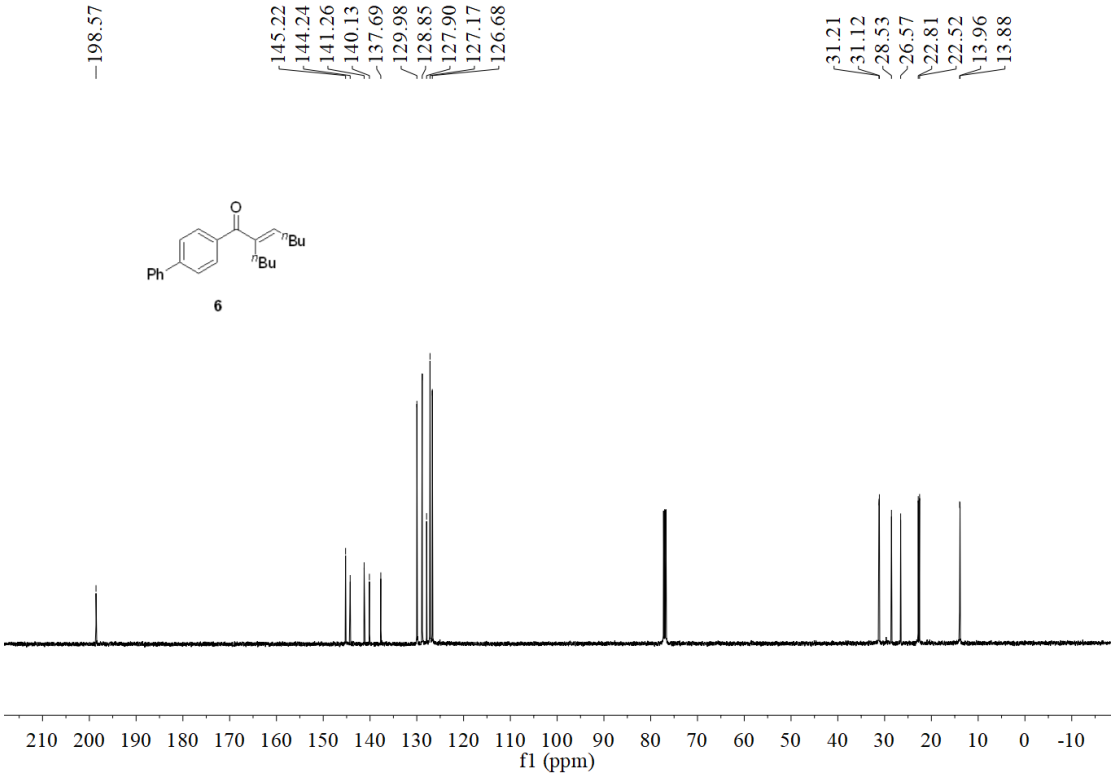
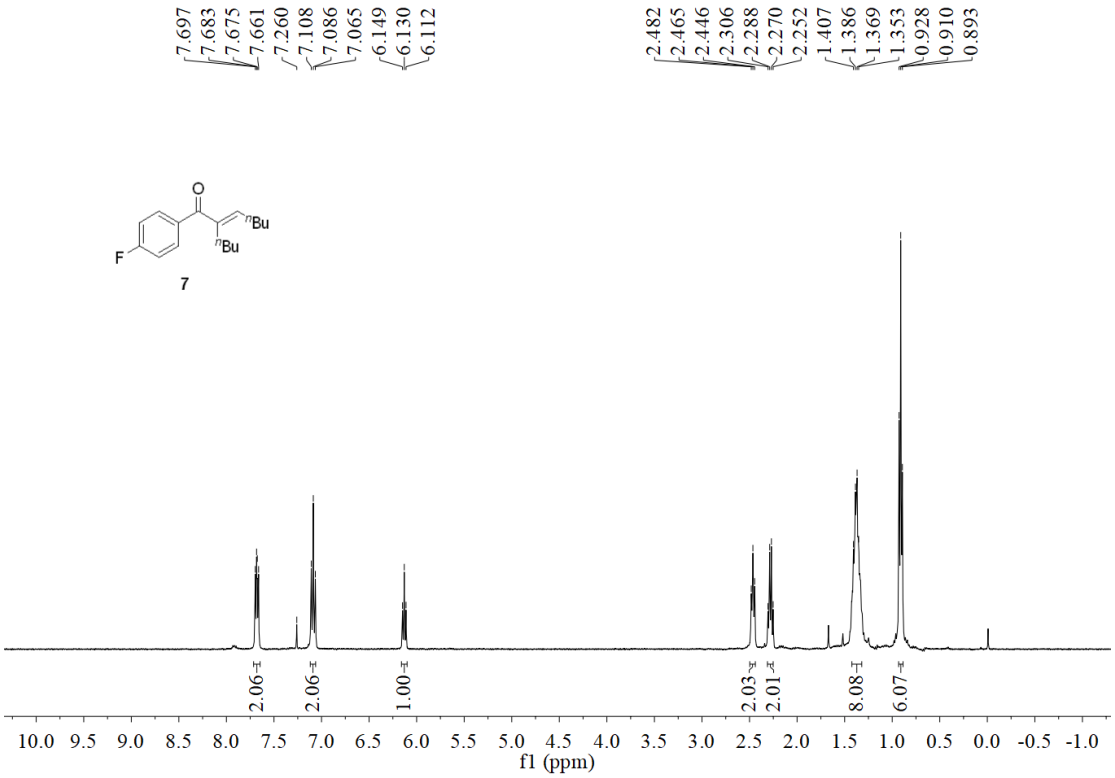


(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



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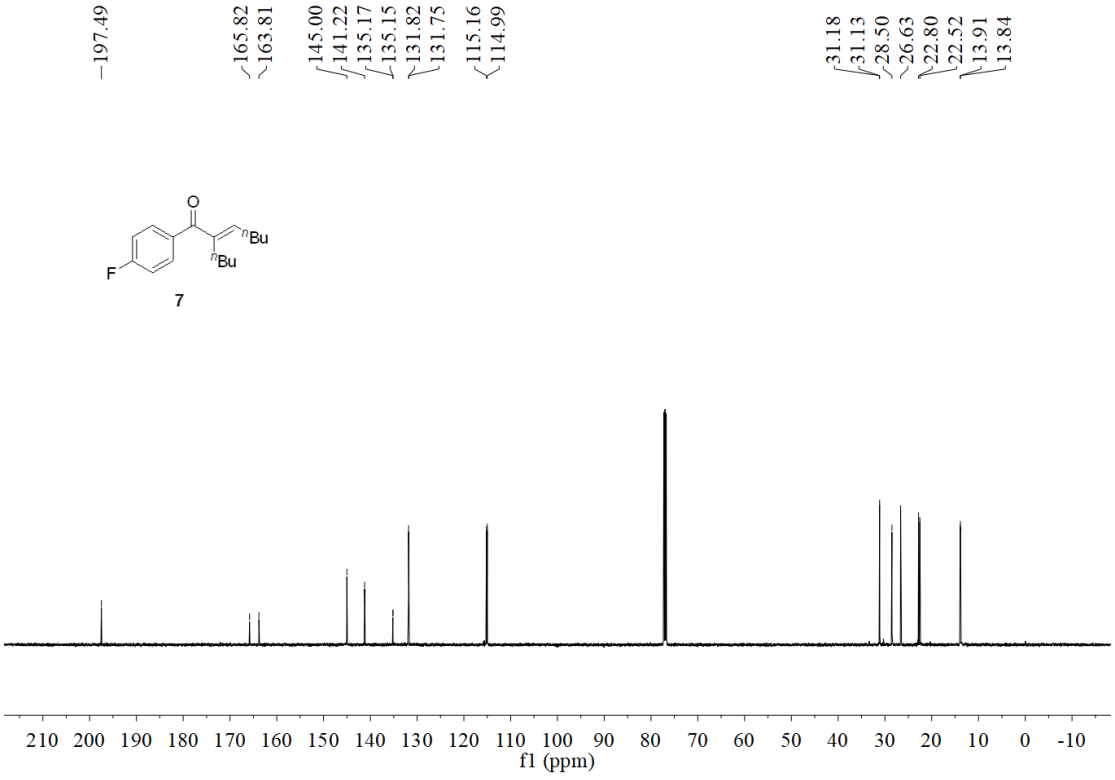
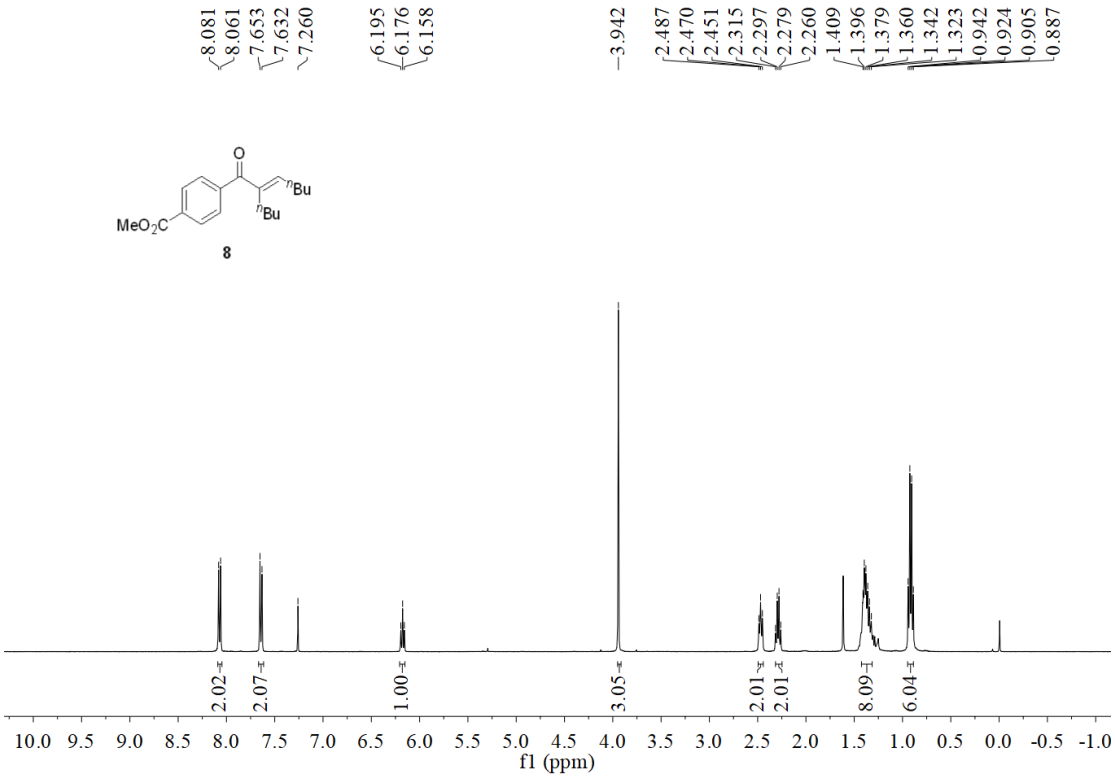
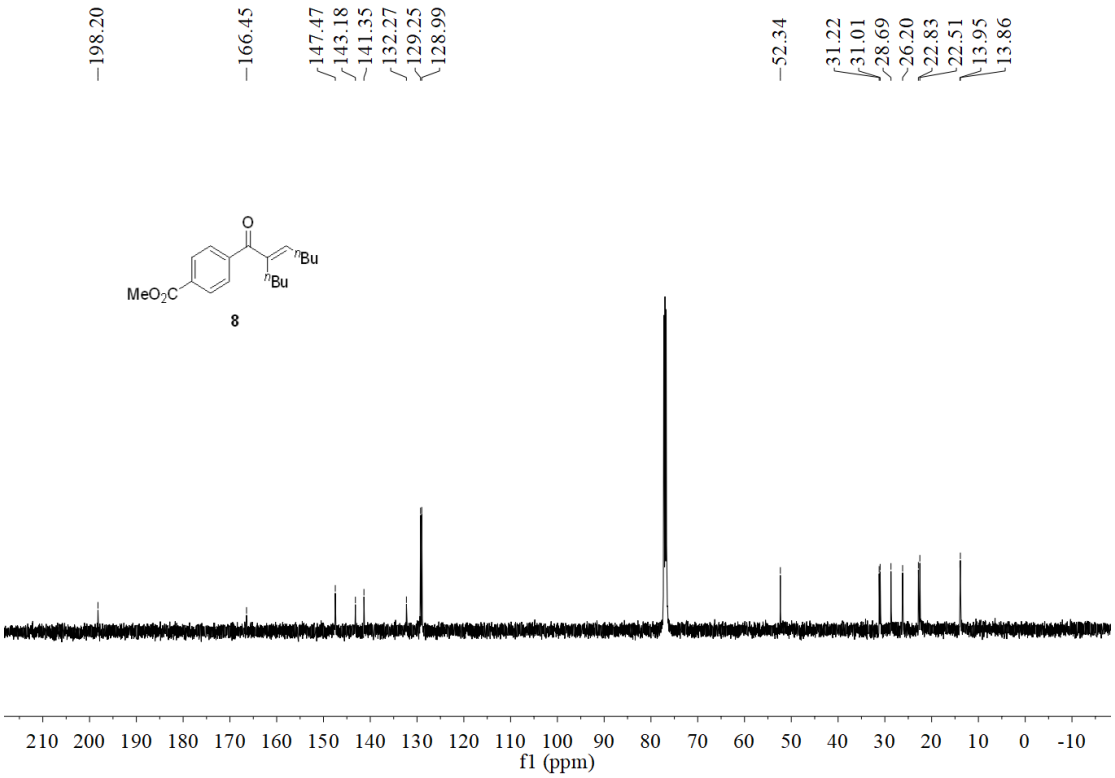


(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



(400 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



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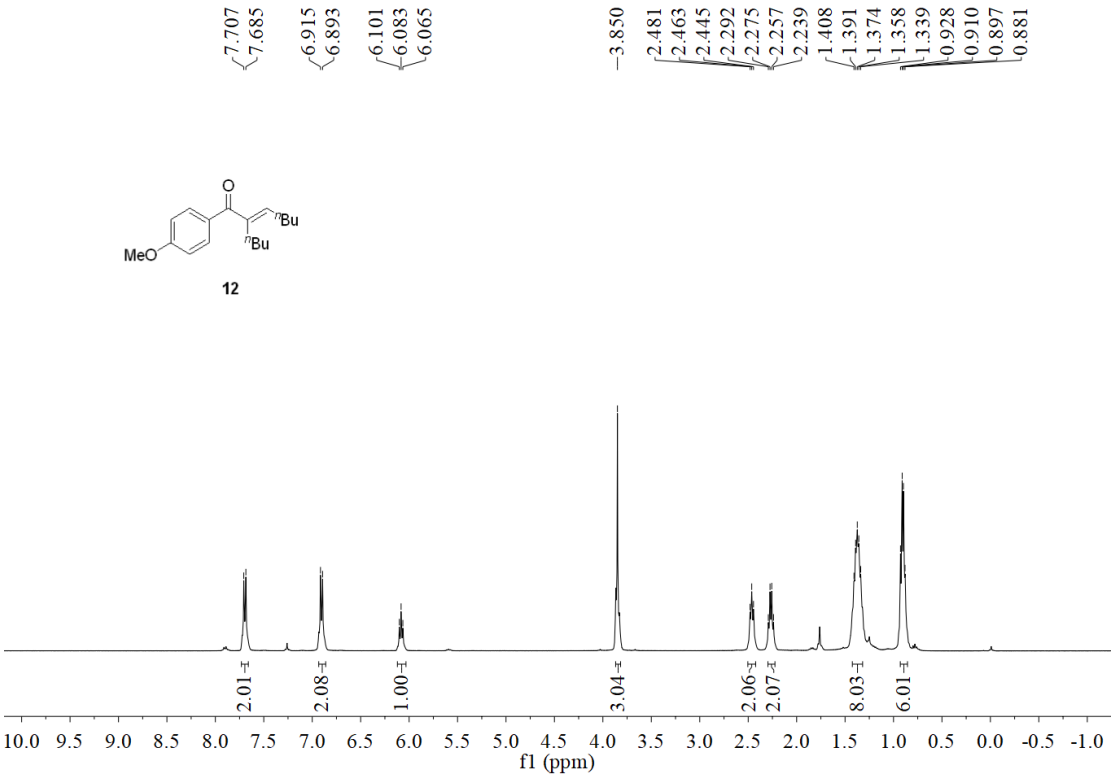

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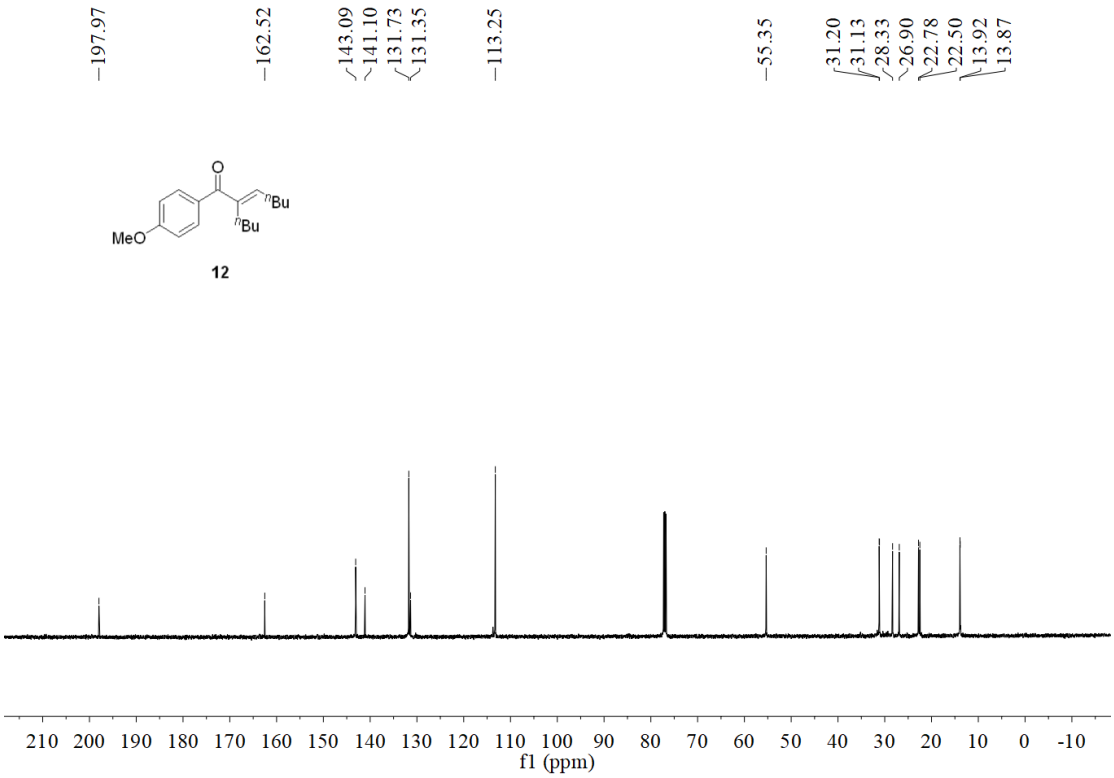

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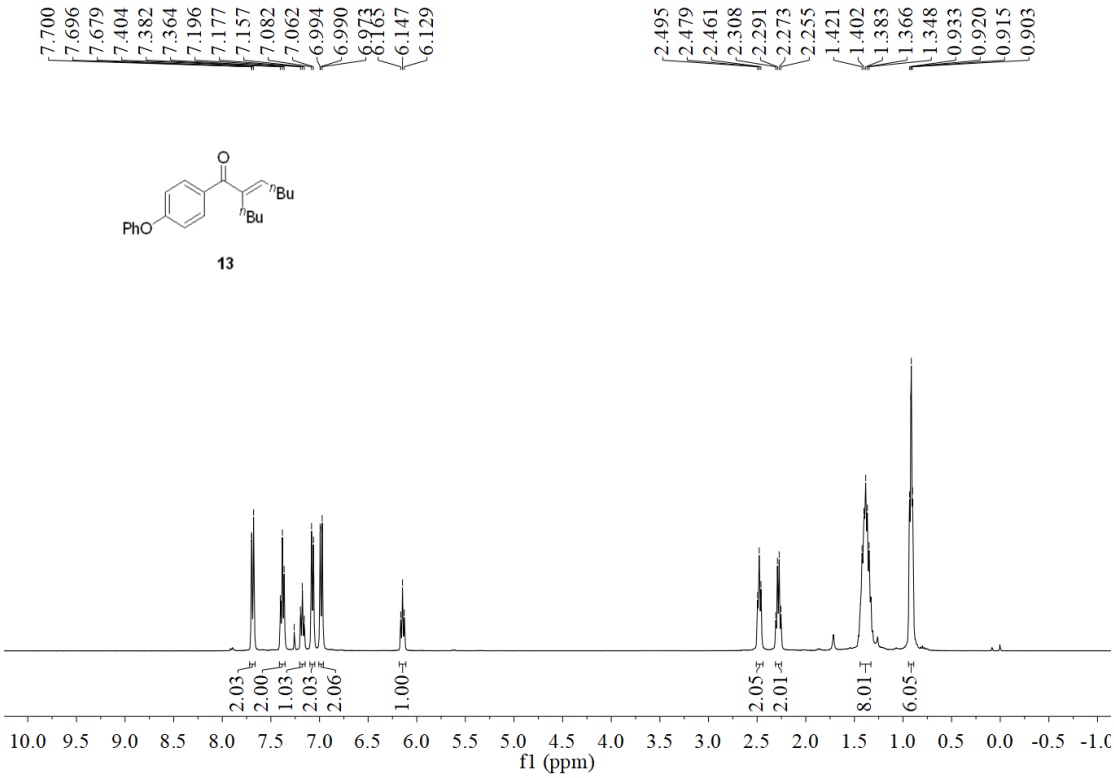

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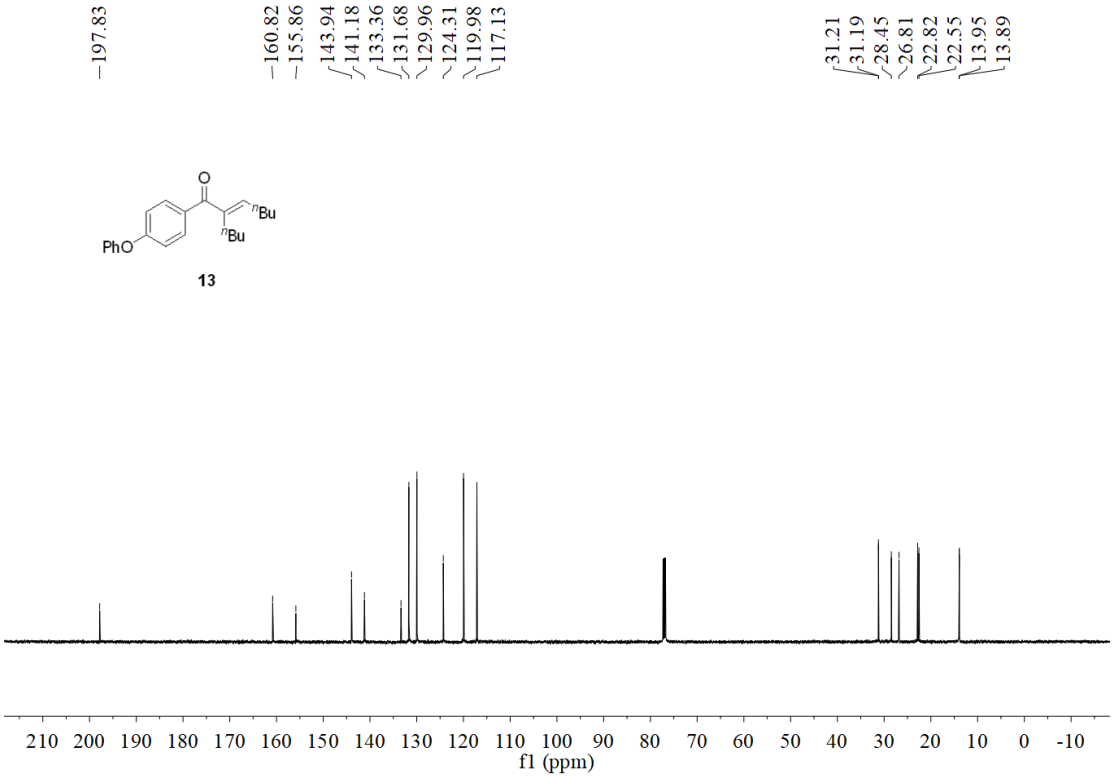
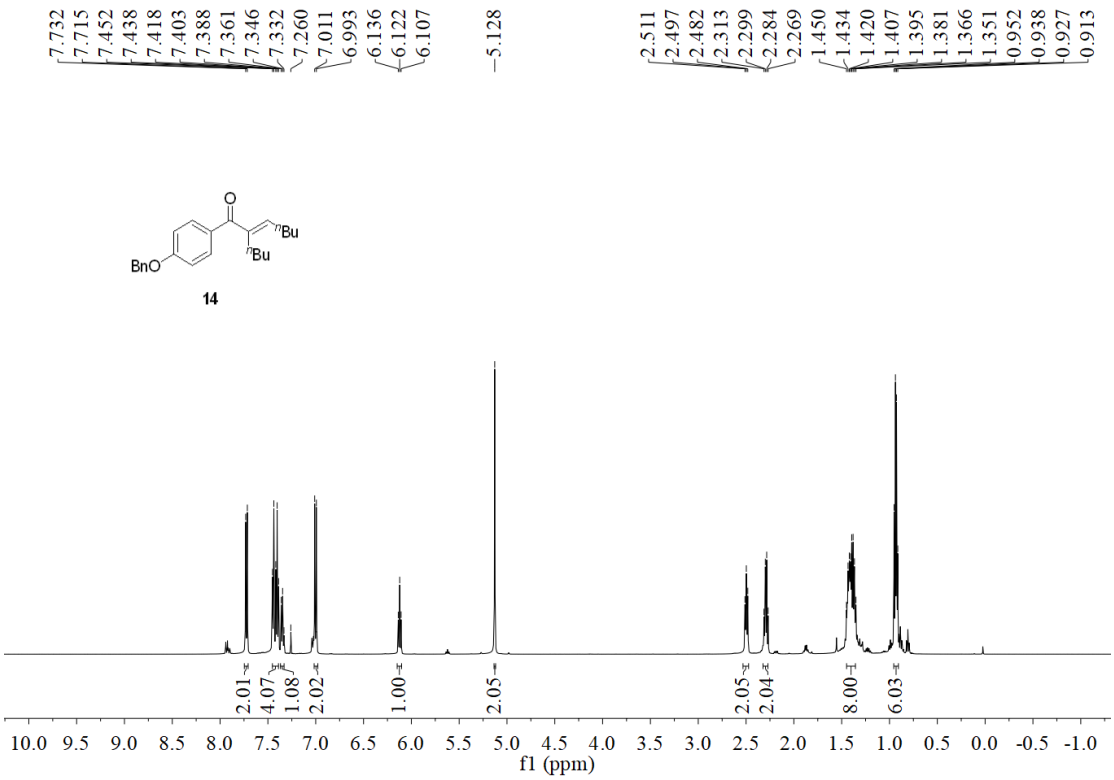


(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



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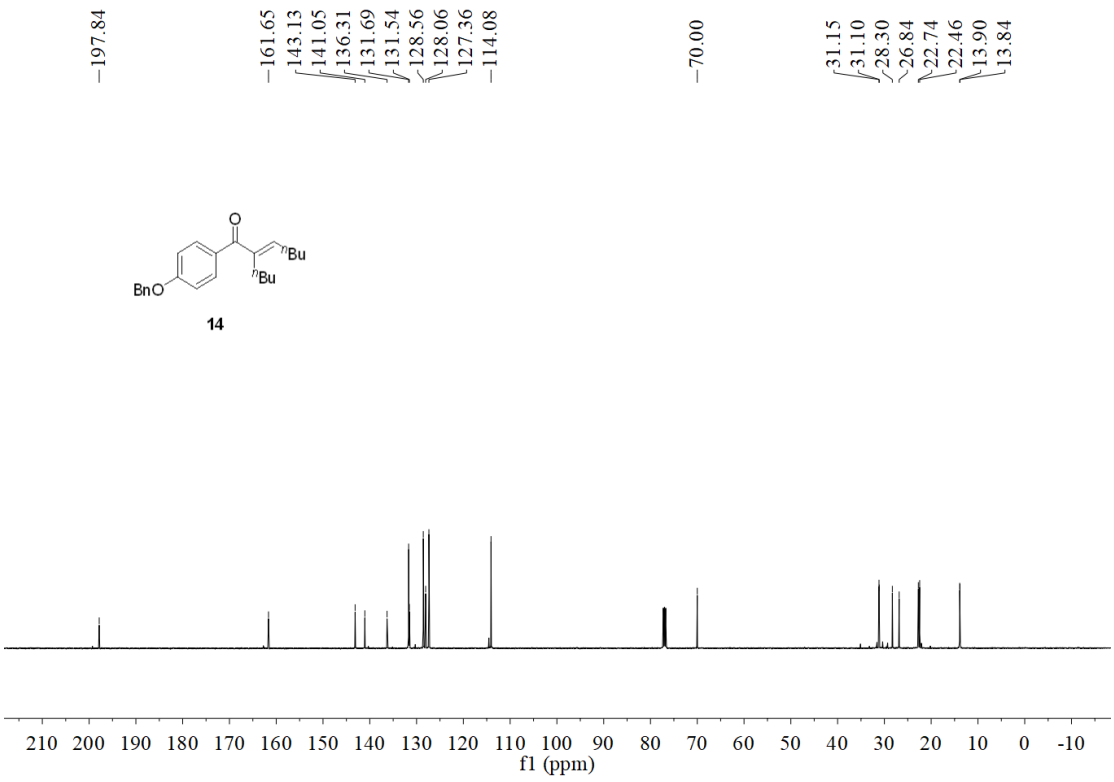

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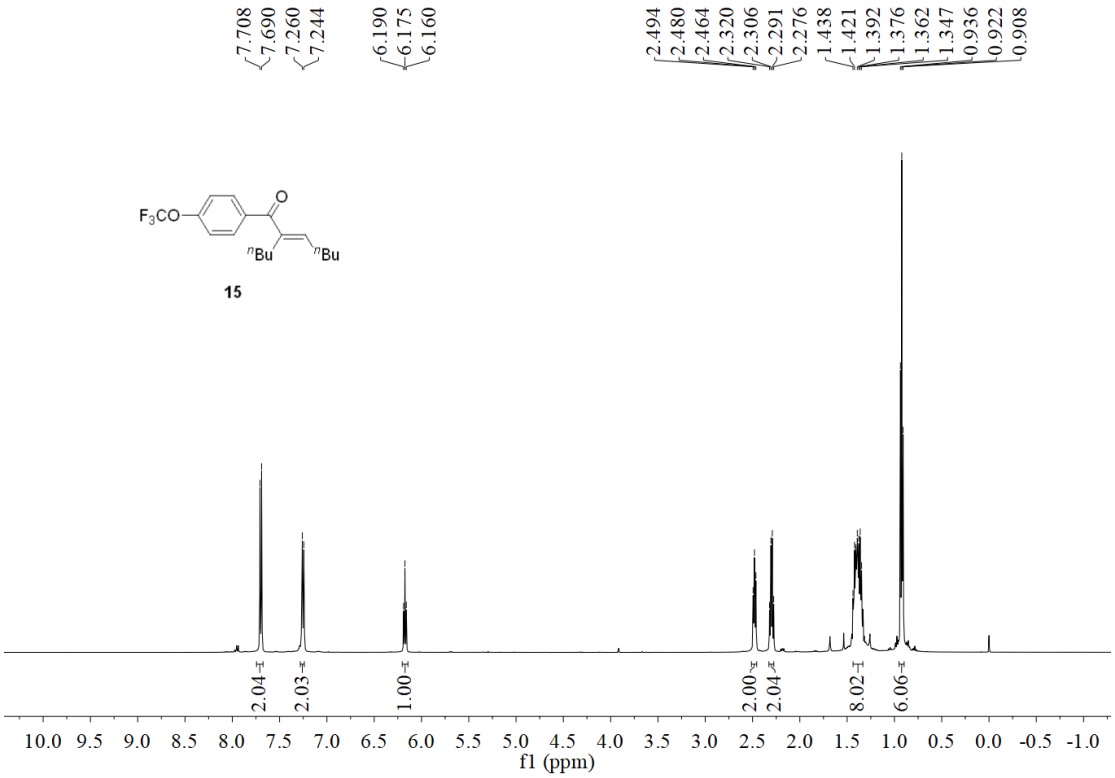

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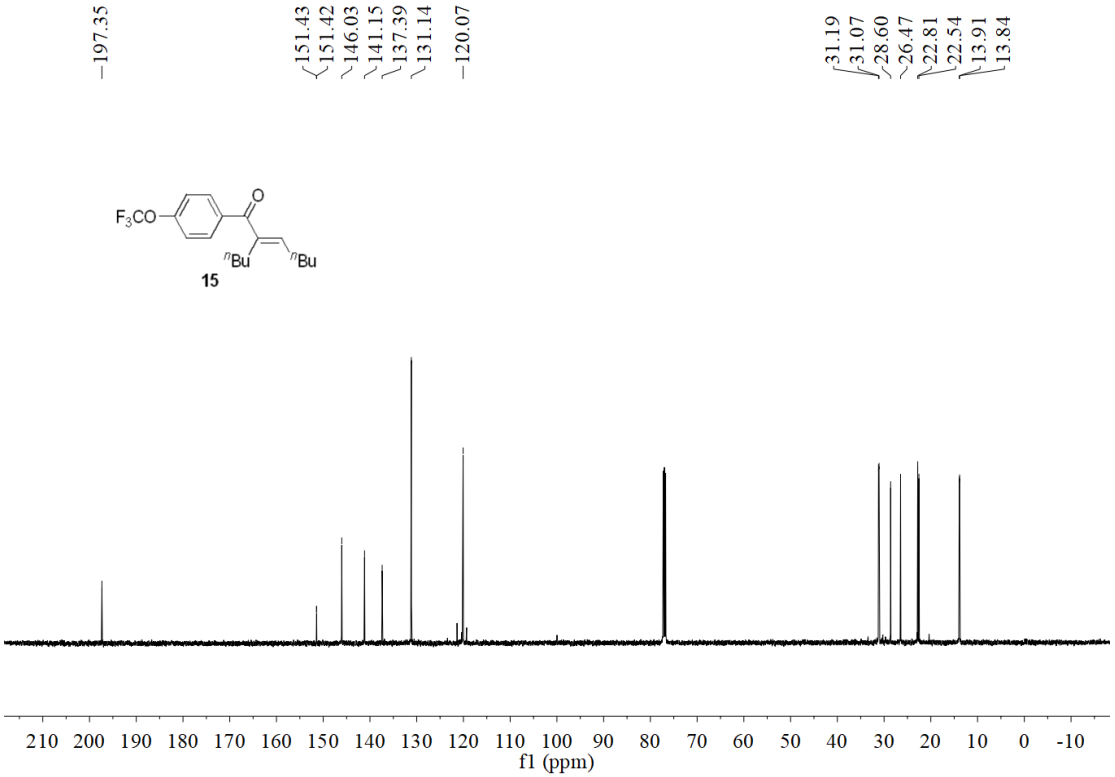

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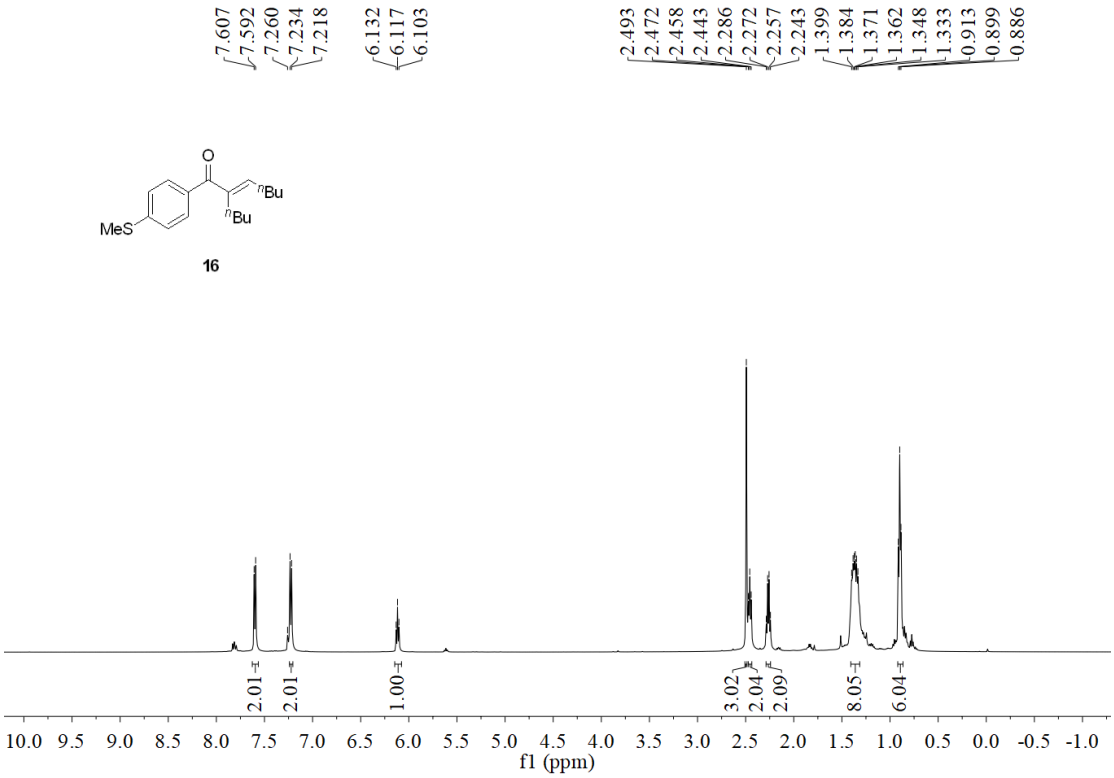

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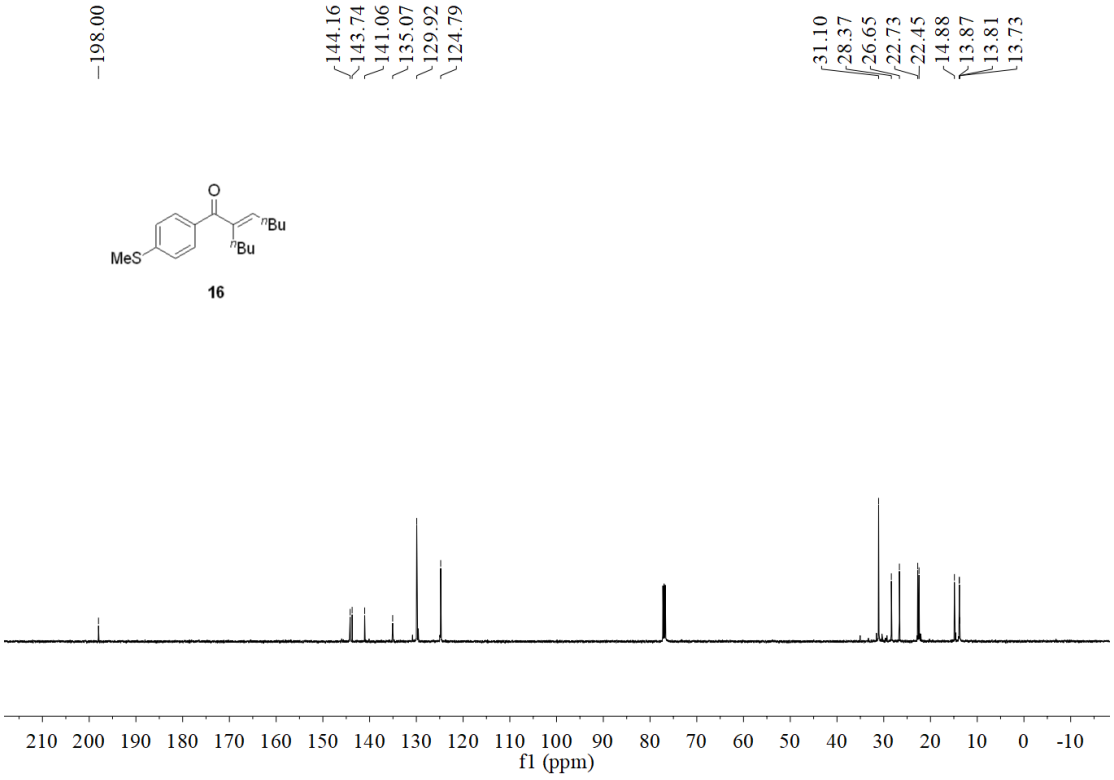

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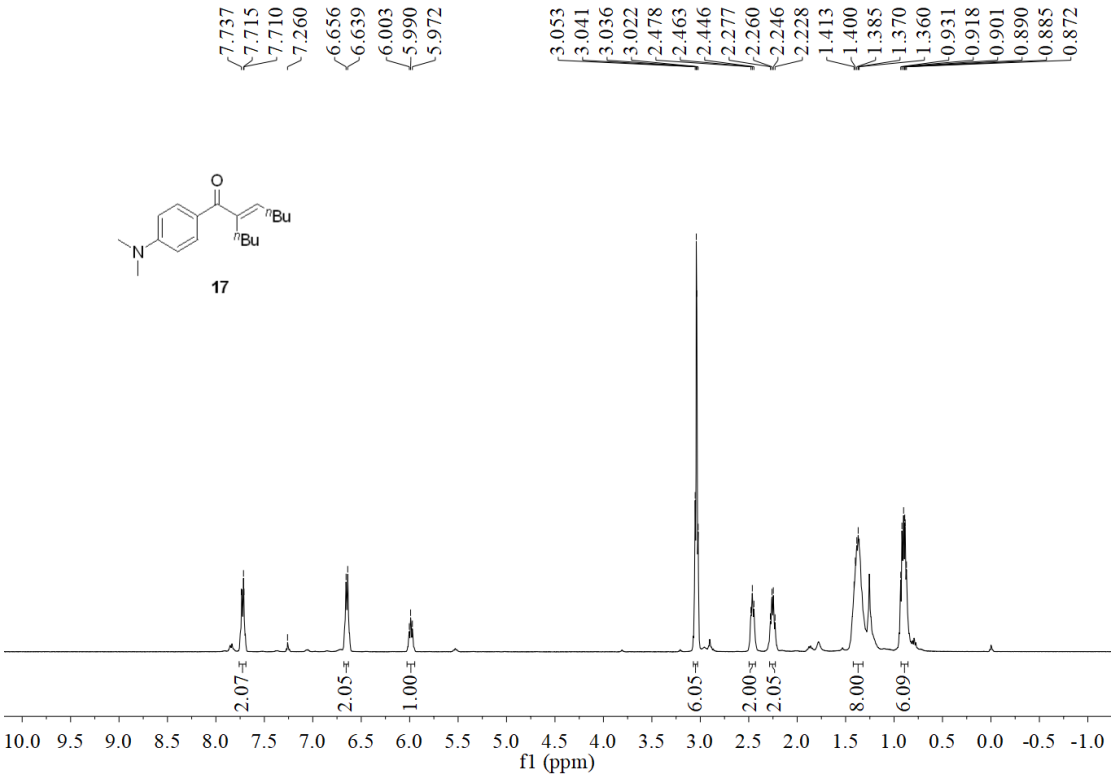

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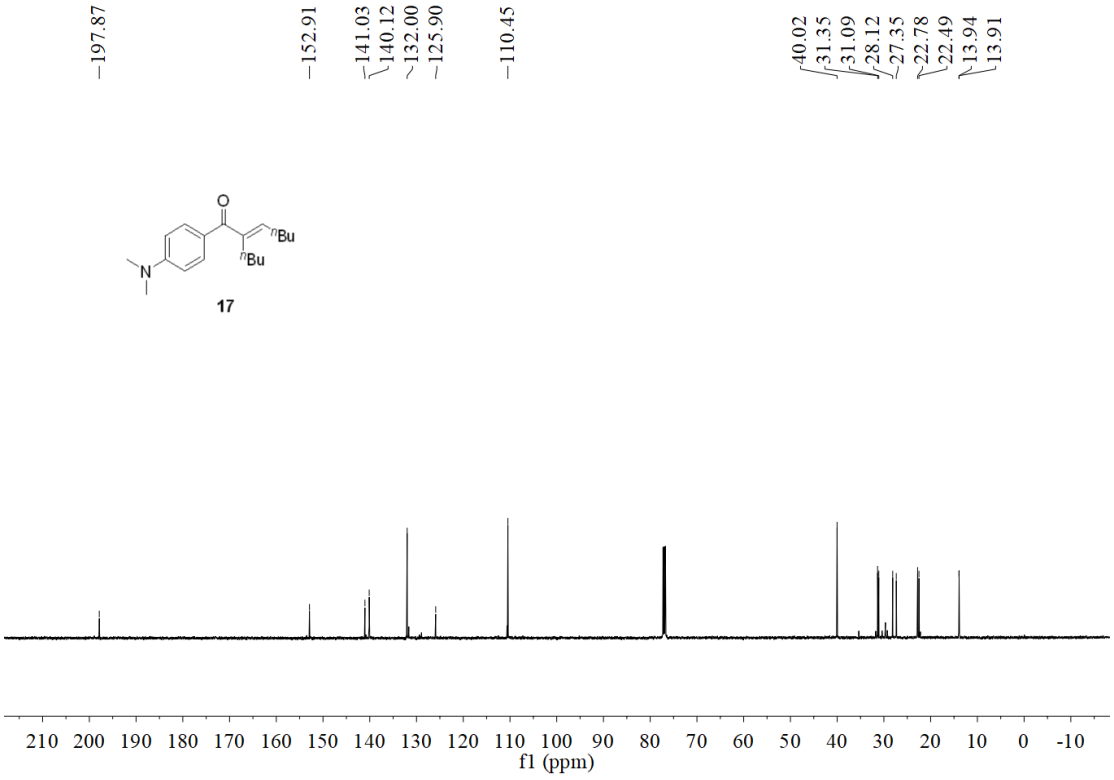

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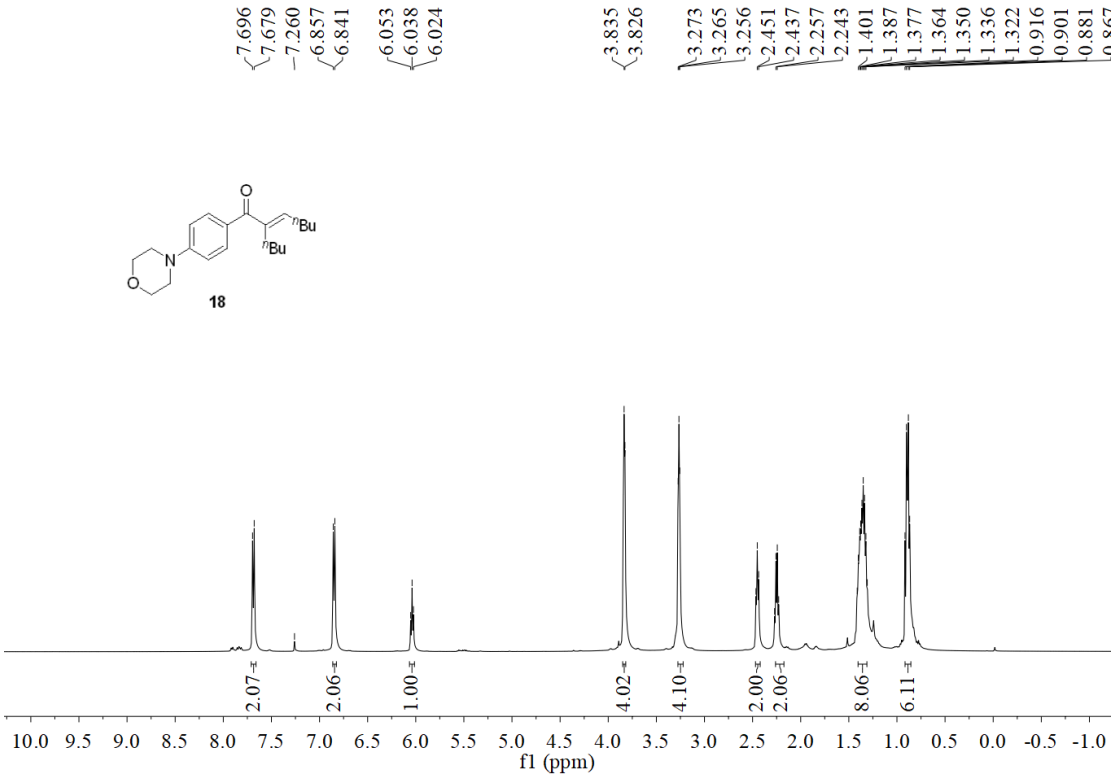

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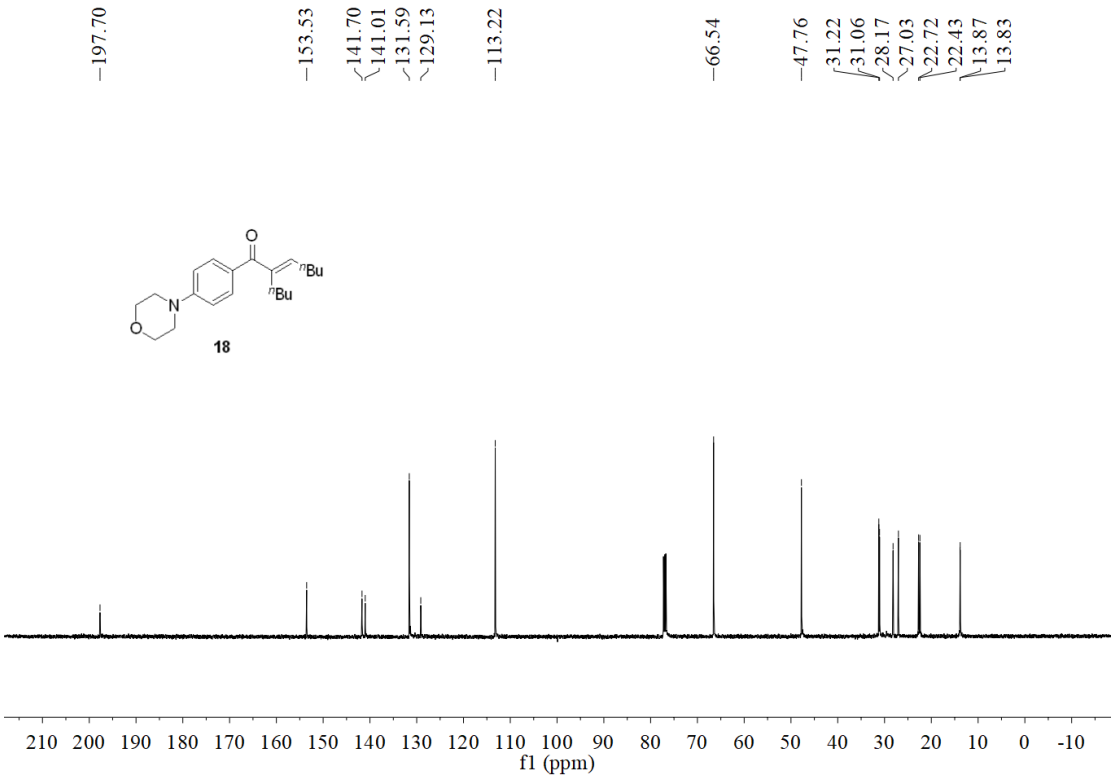
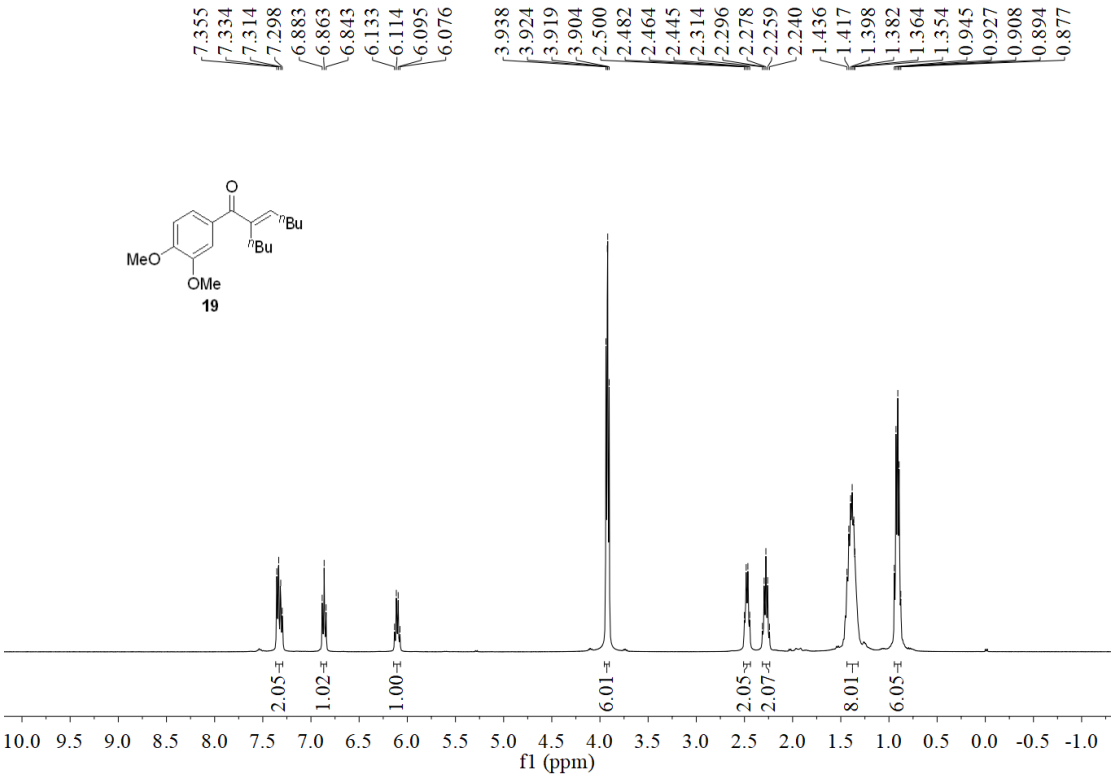


(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



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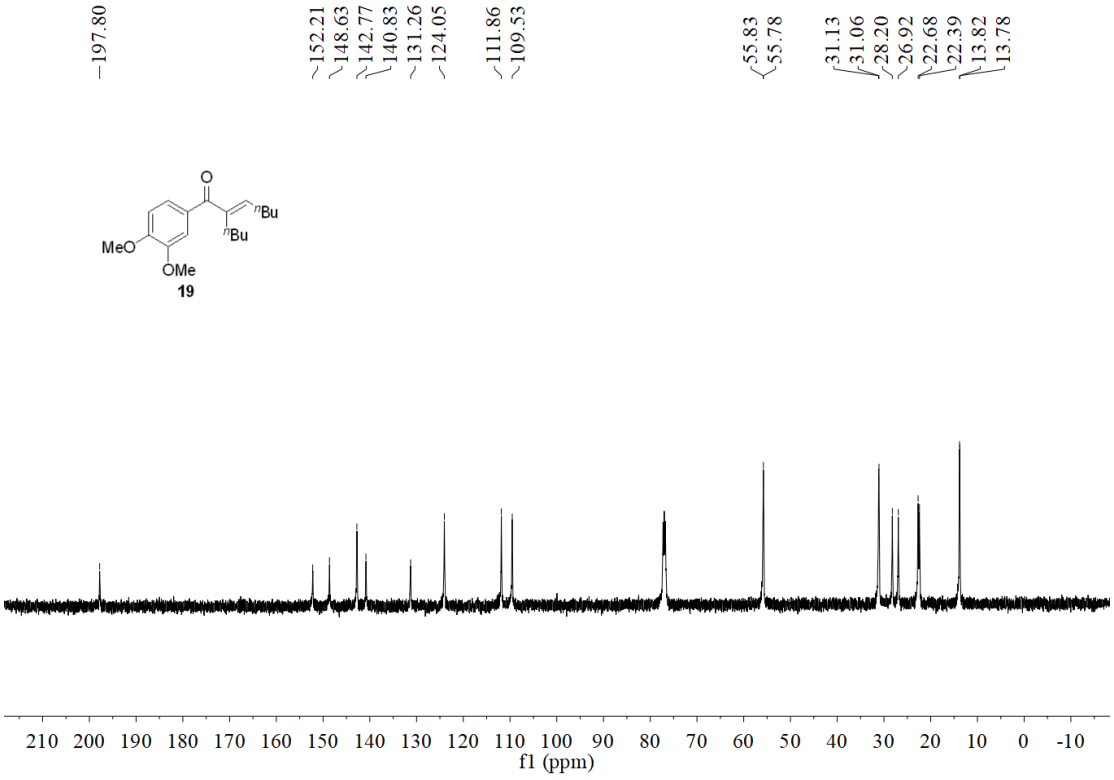

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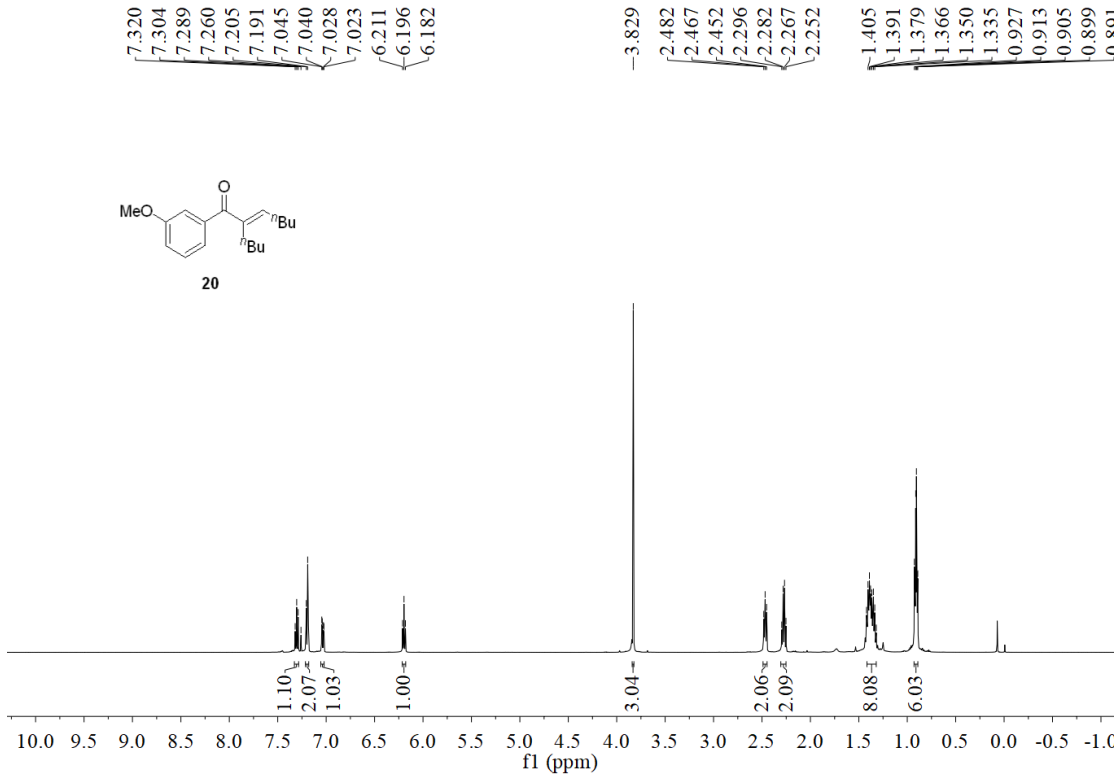

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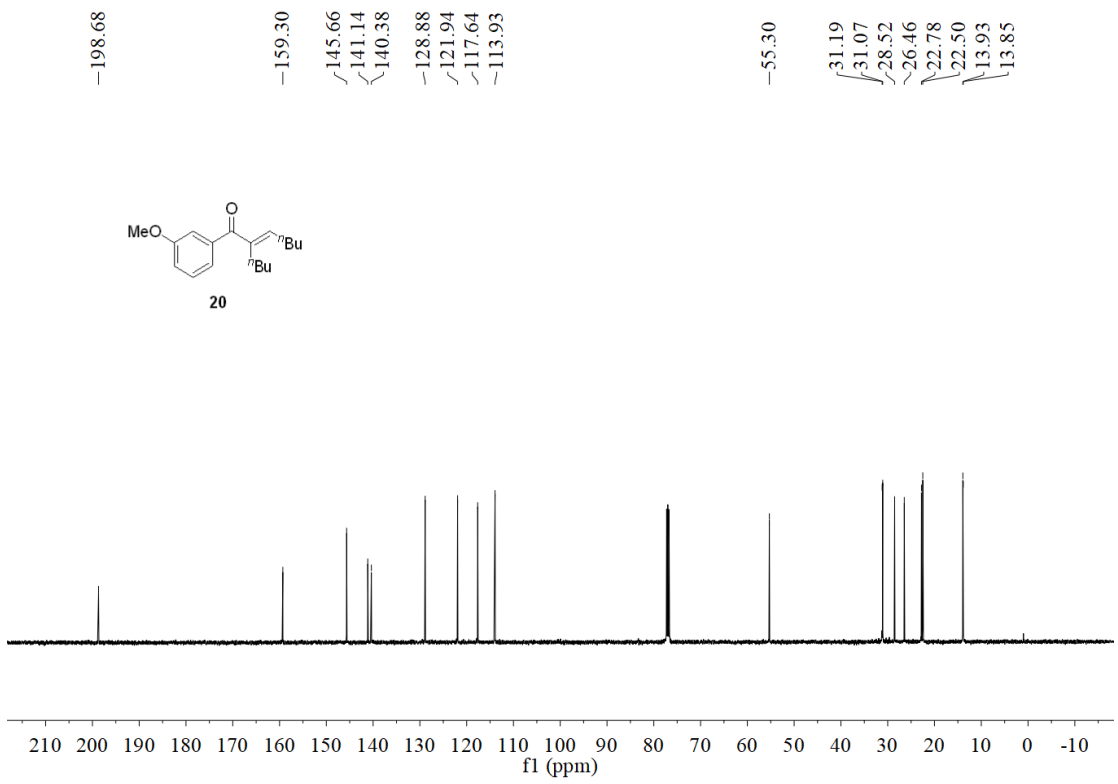

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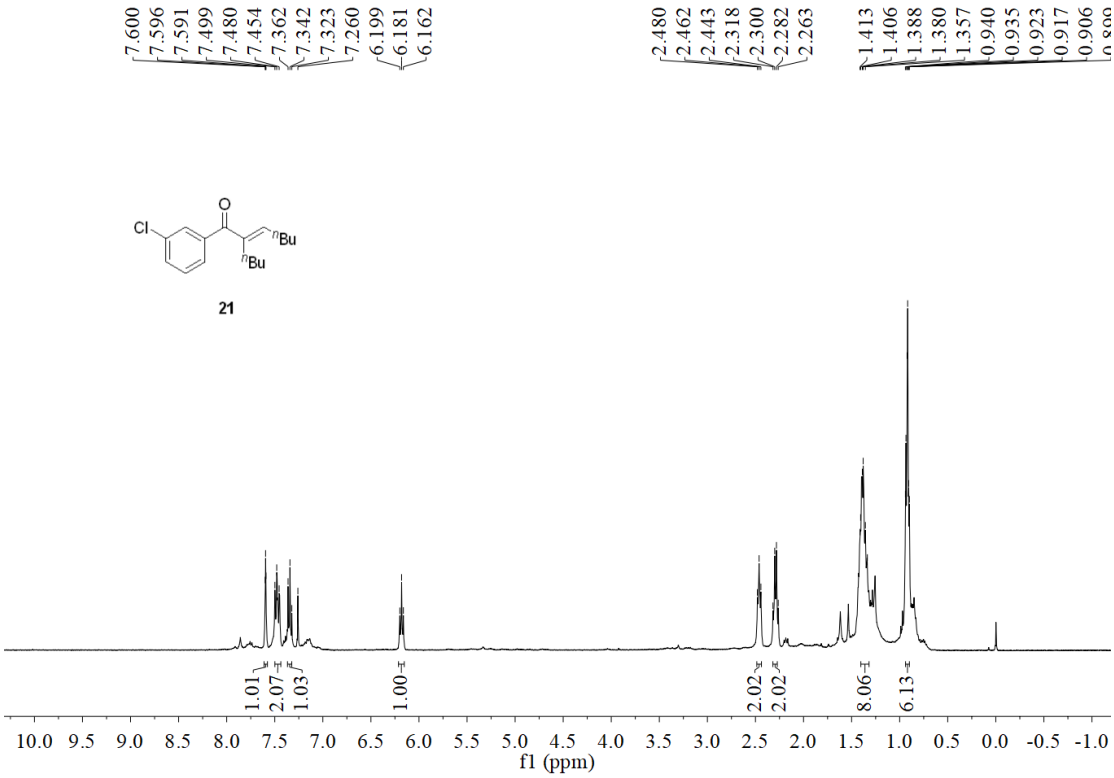
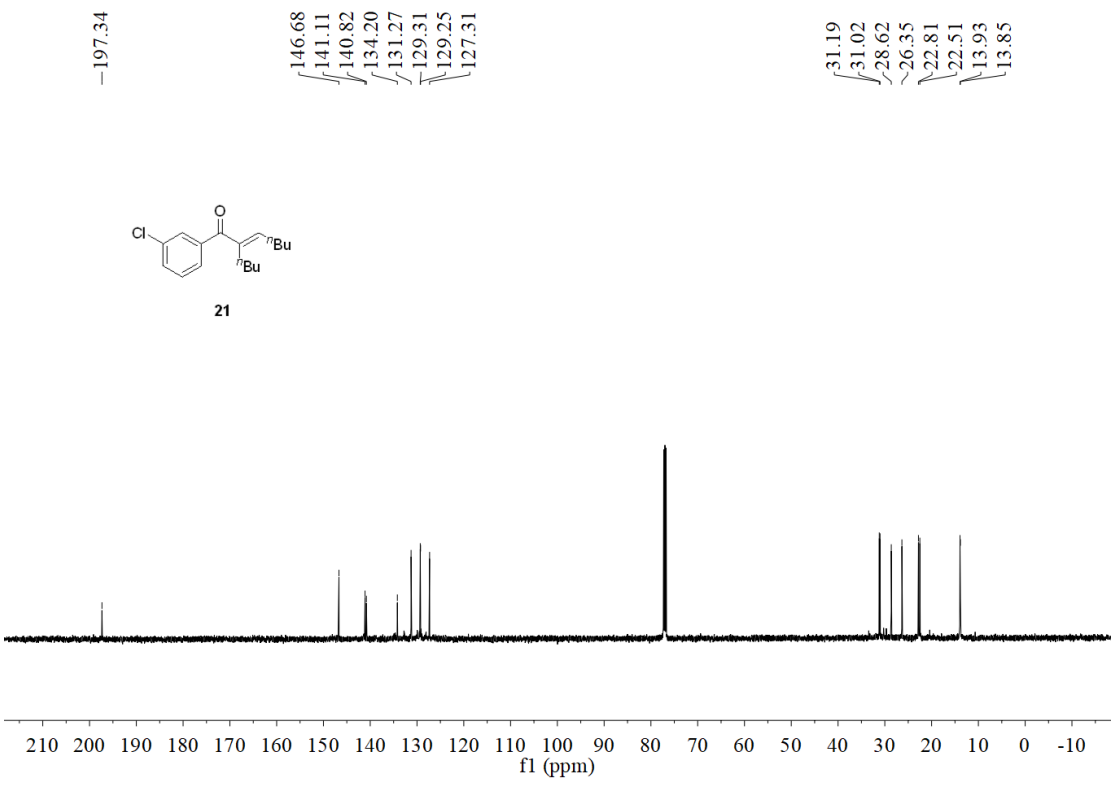


(400 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



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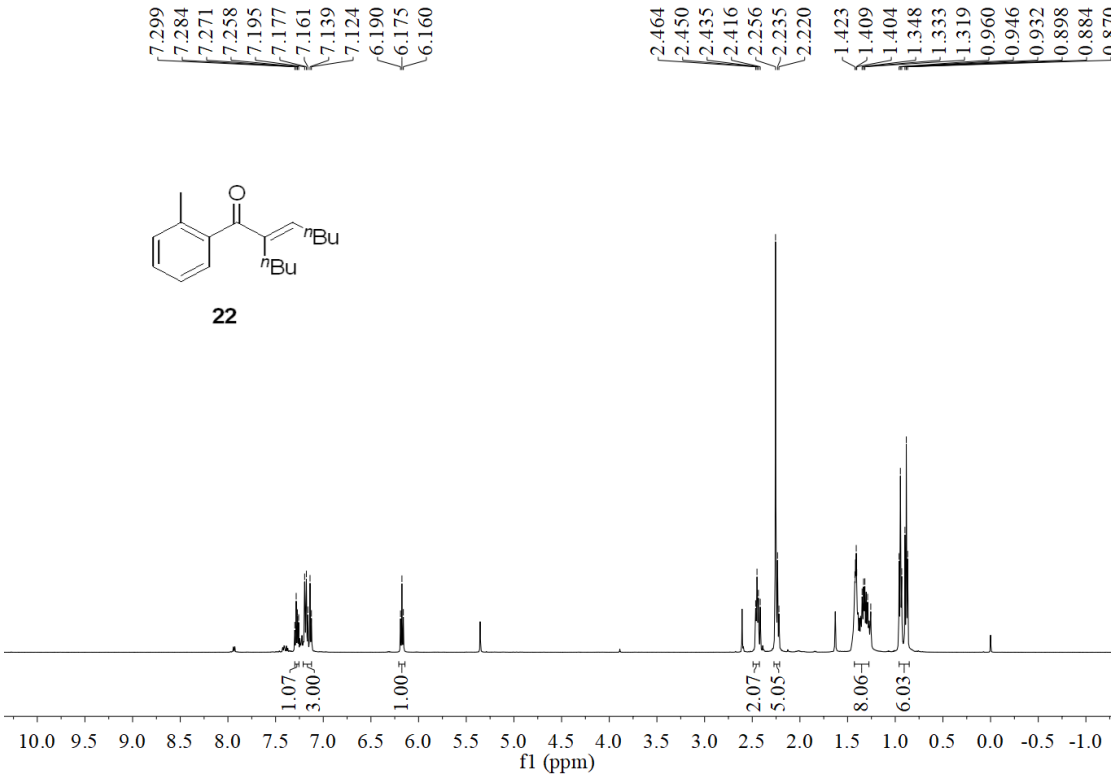
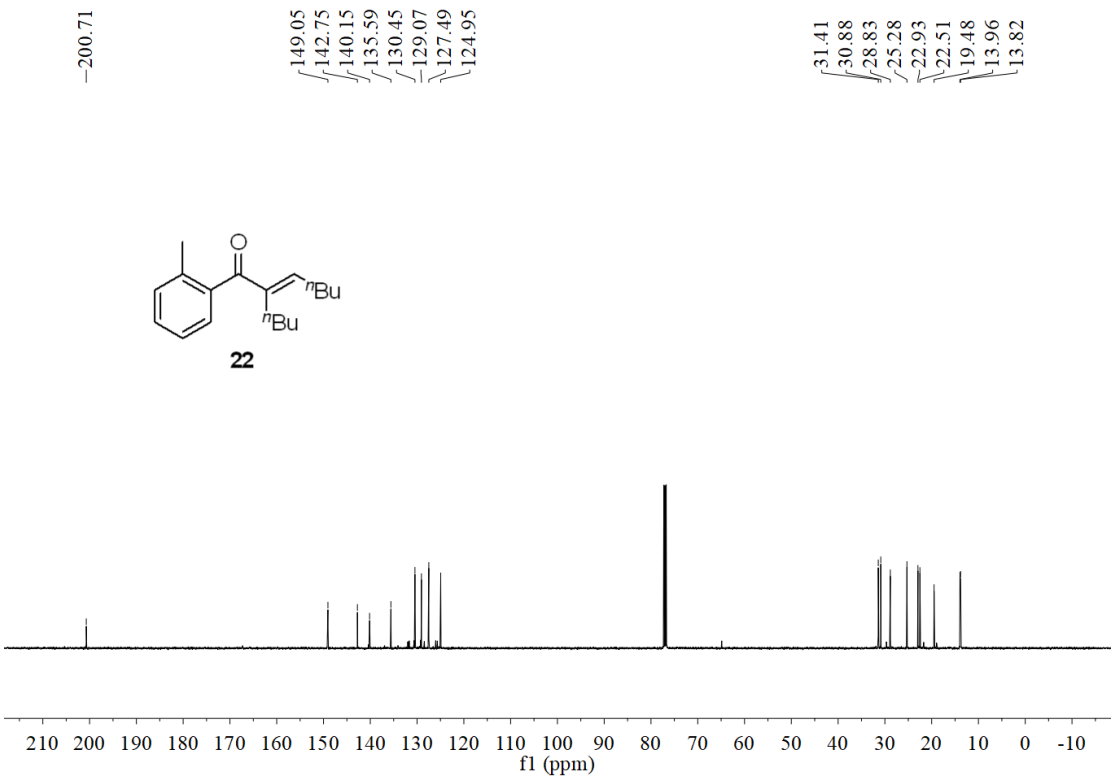
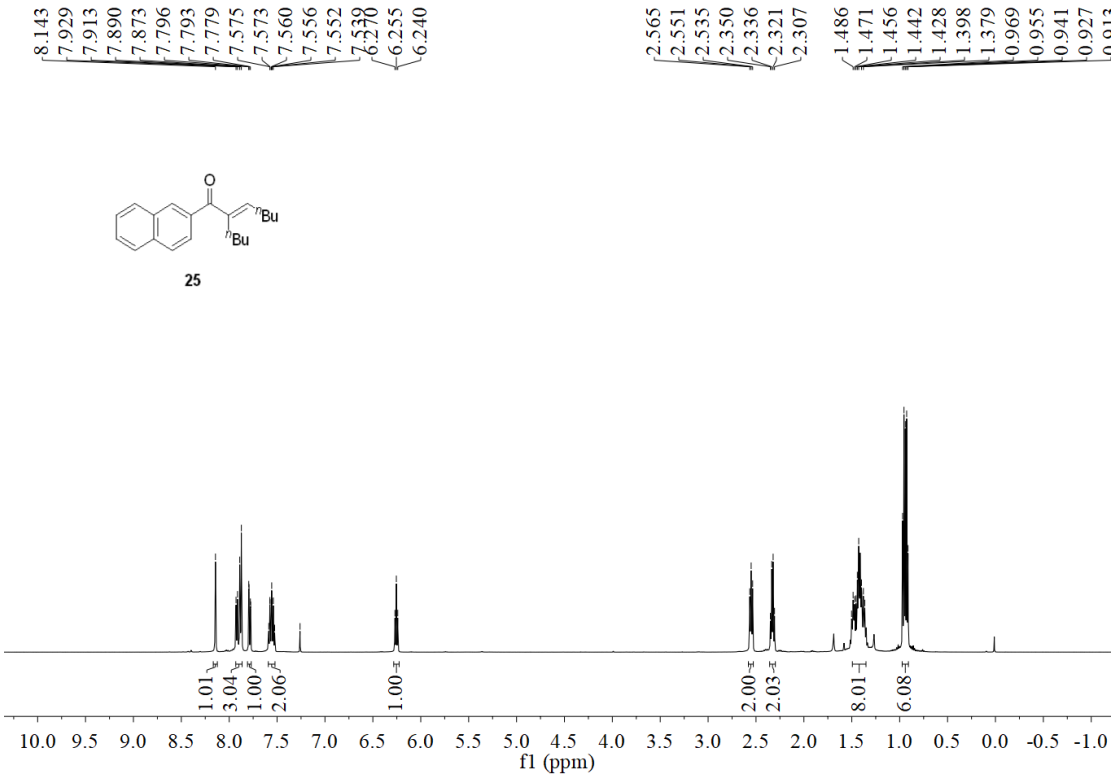


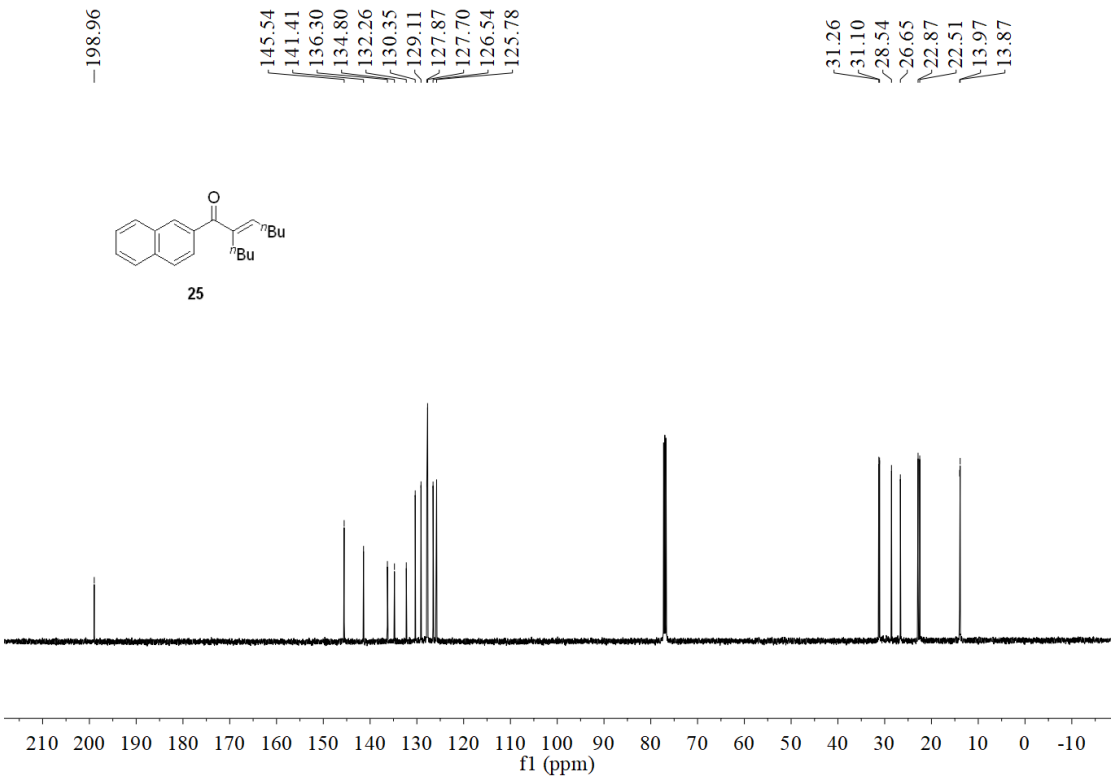
(500 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



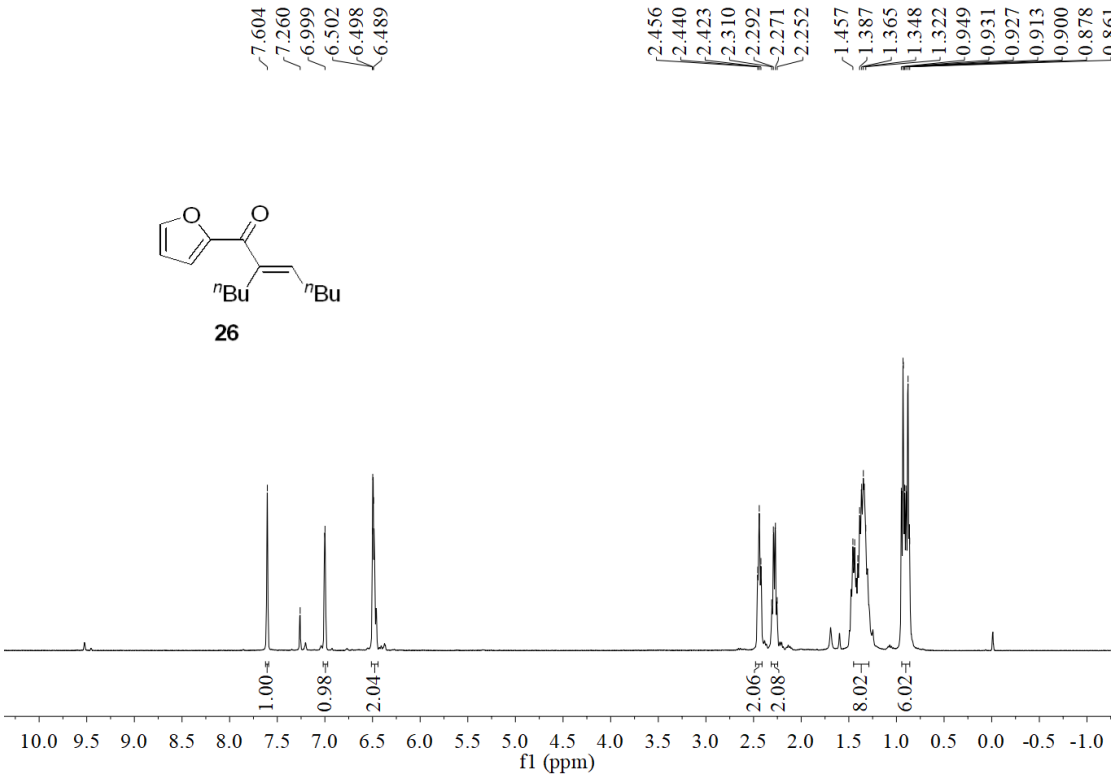
(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



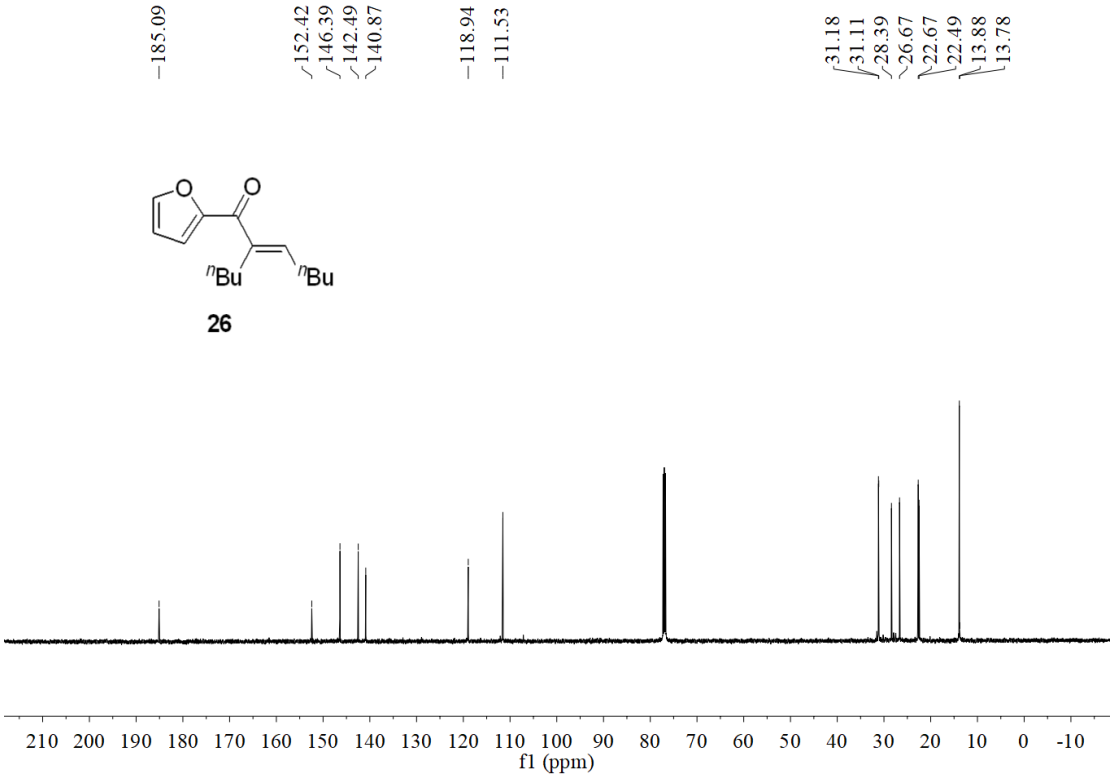
(500 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



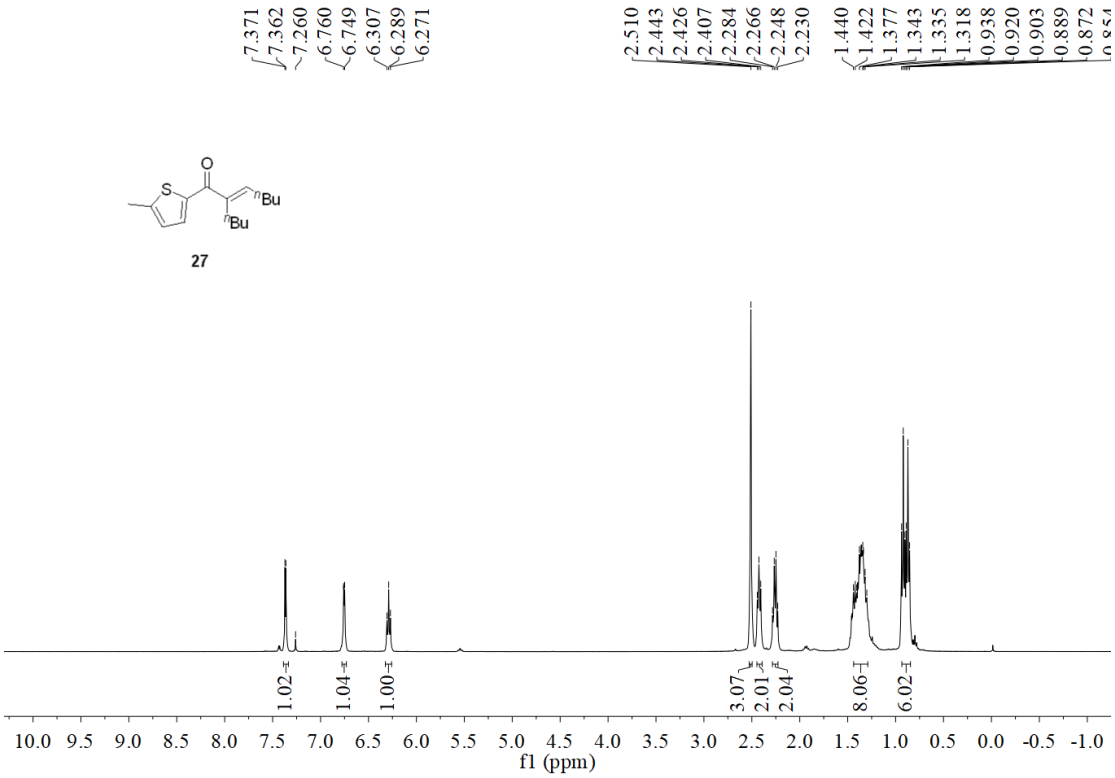
(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



(400 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



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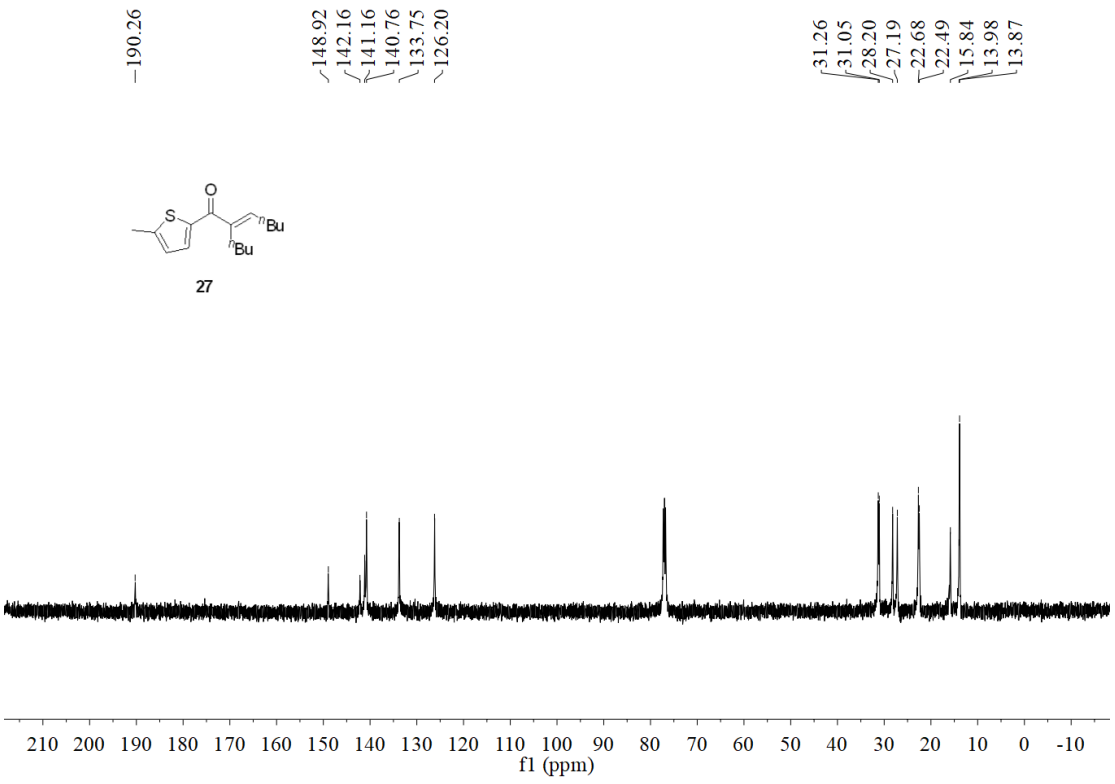
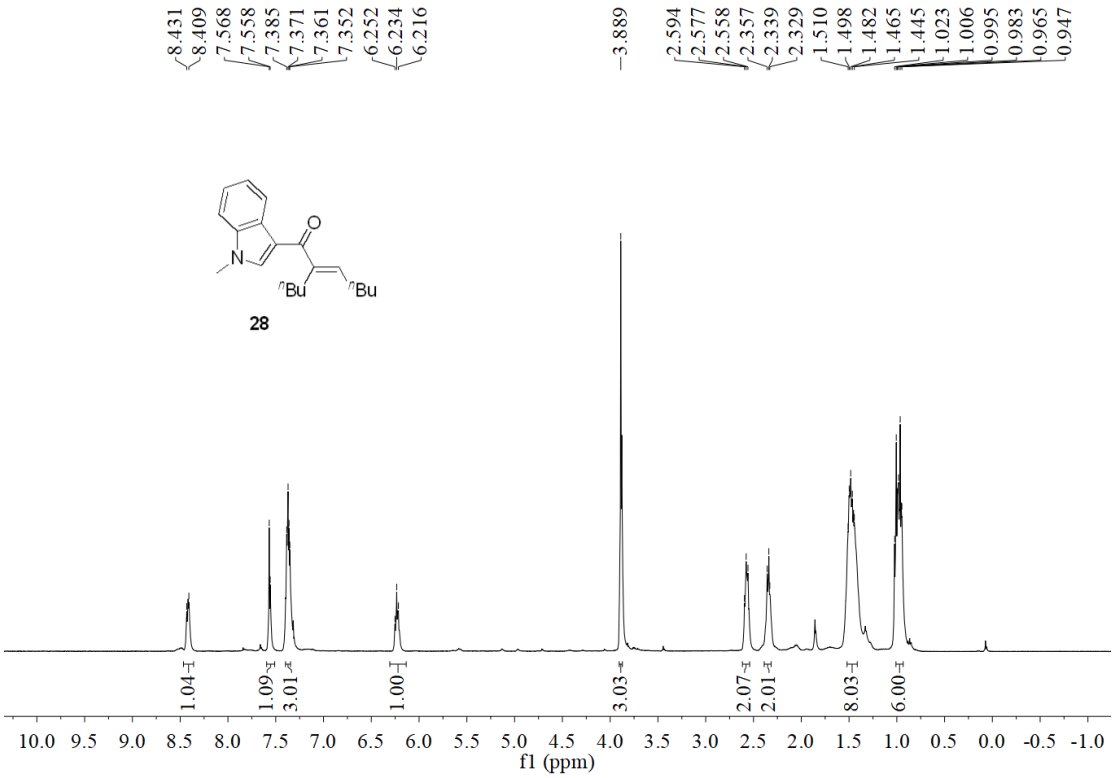
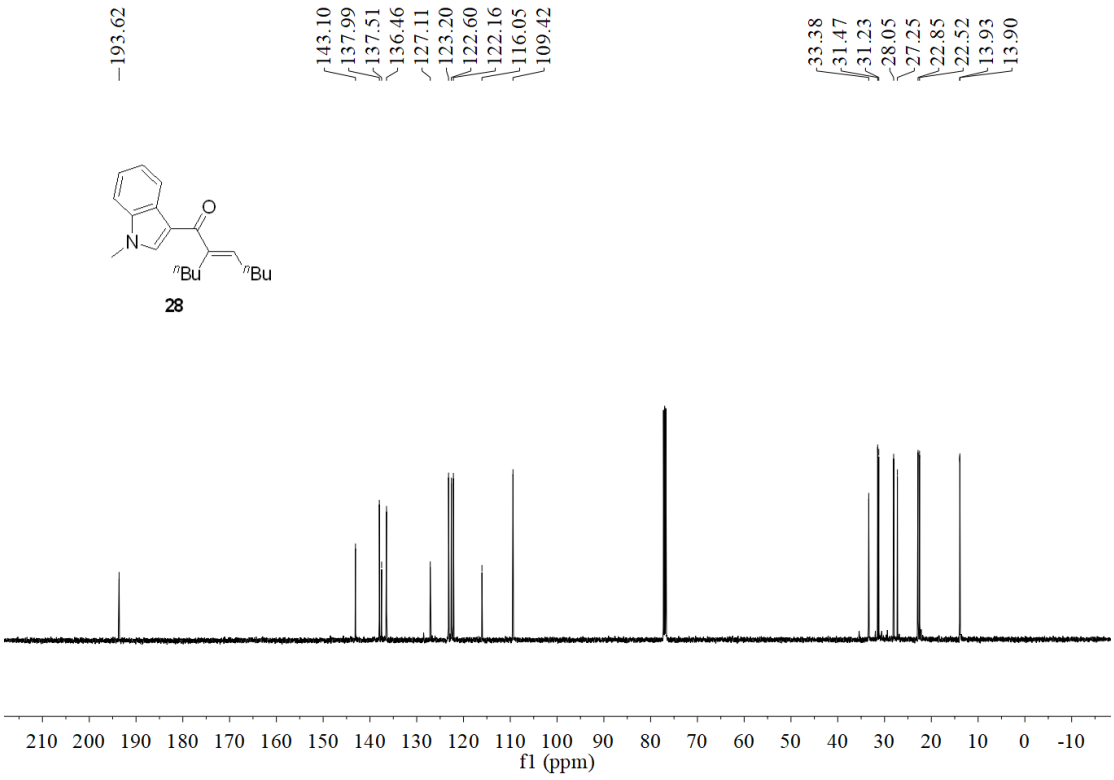


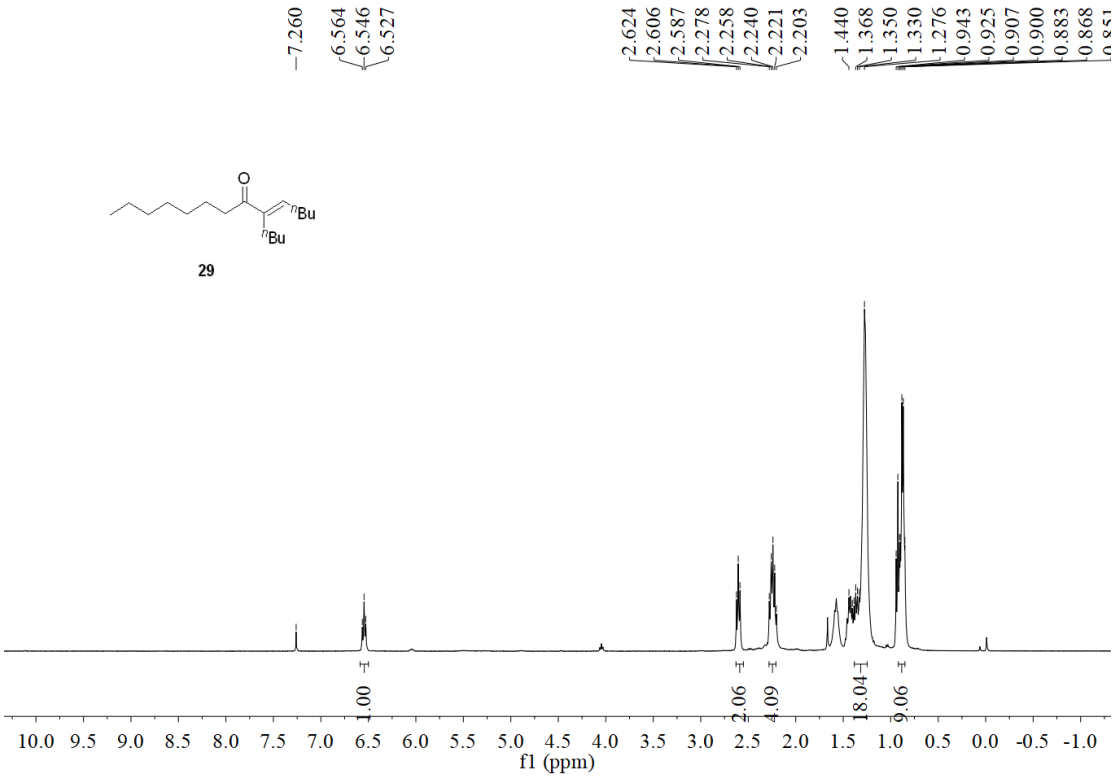
(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



(400 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



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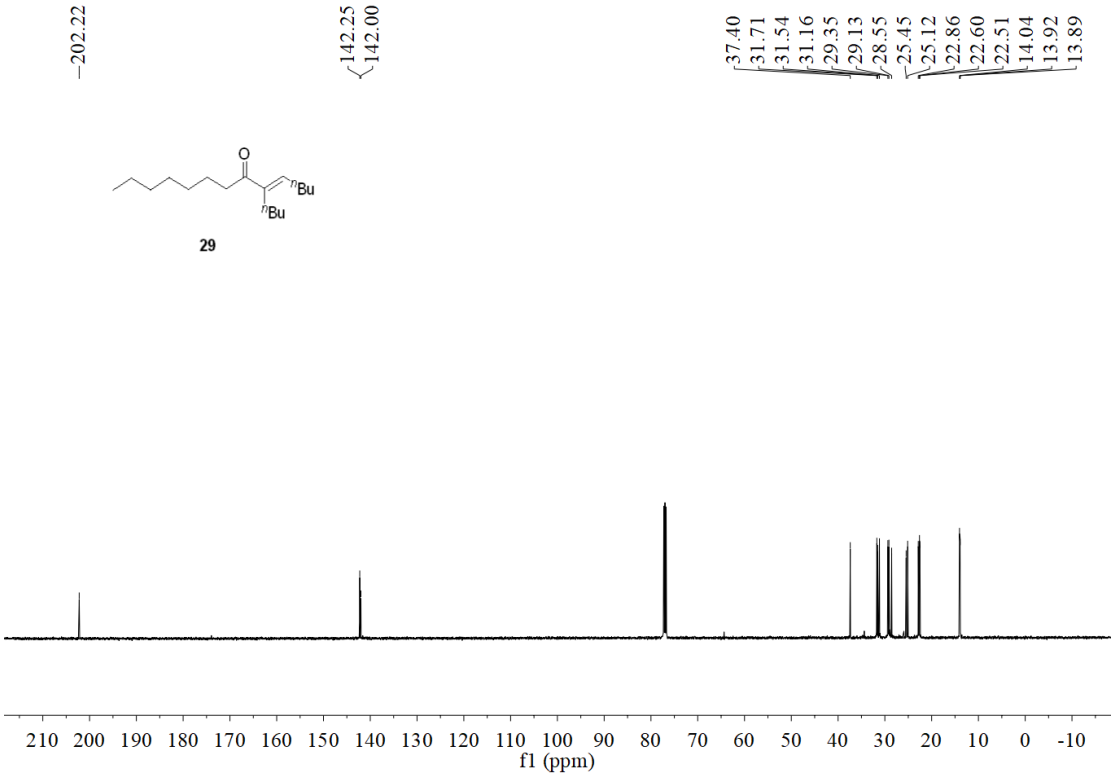

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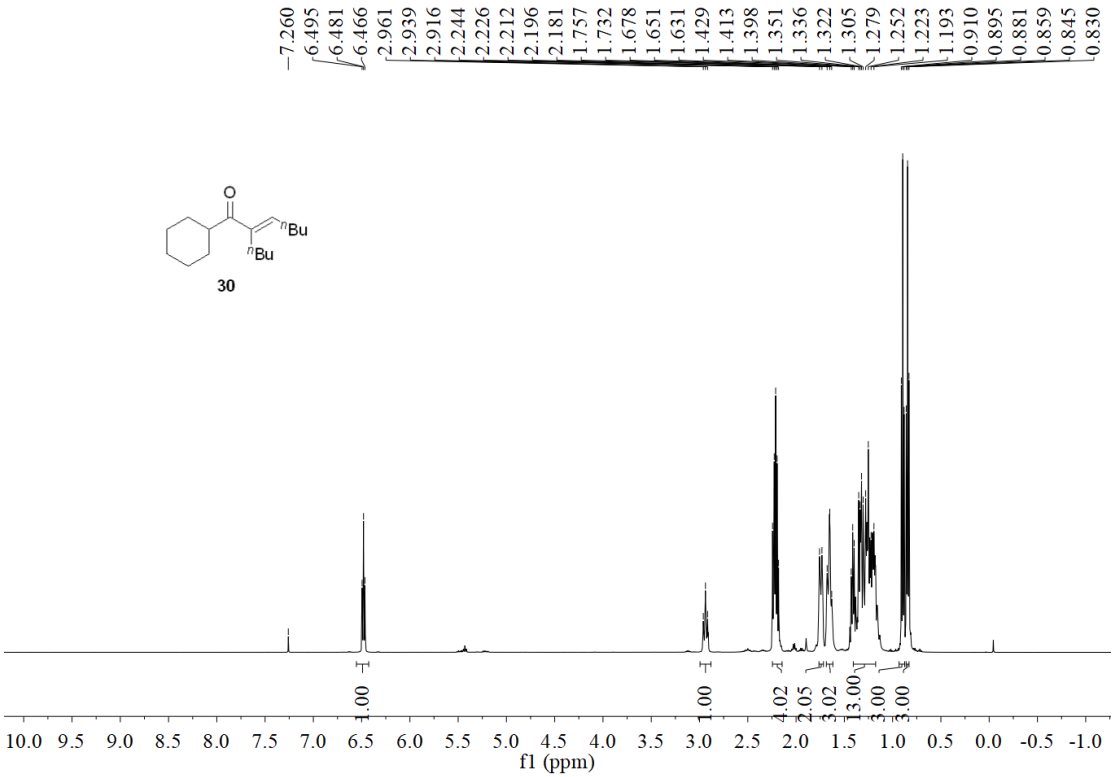

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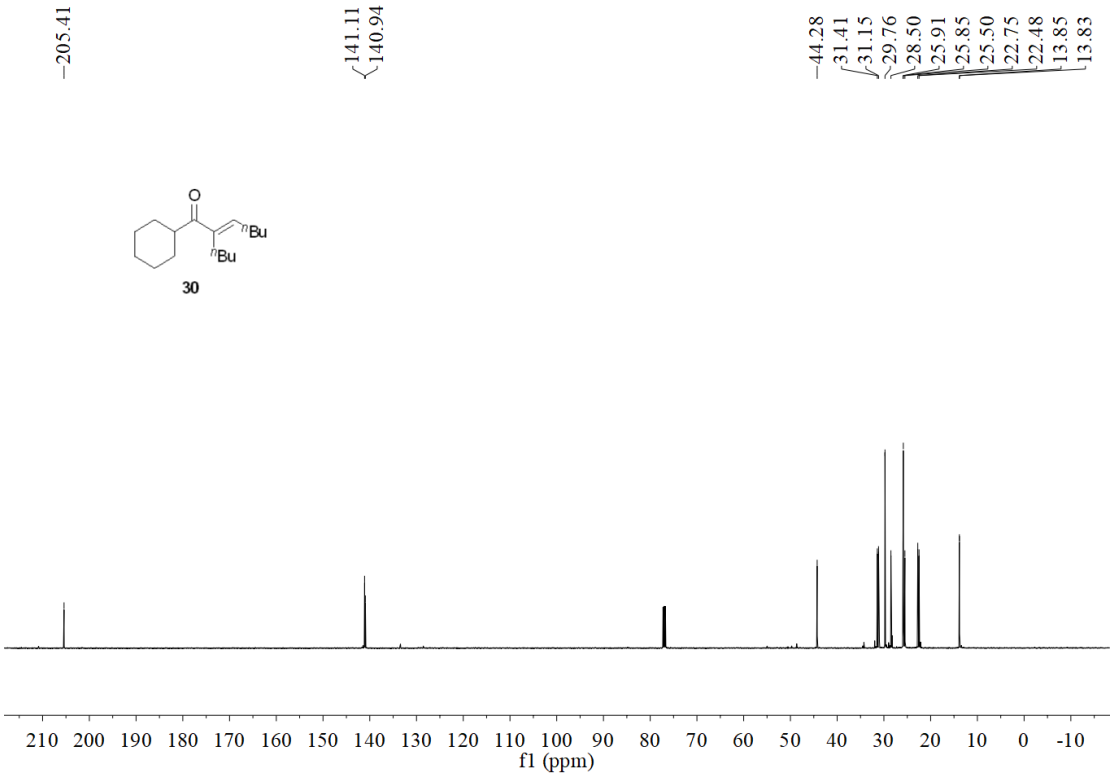

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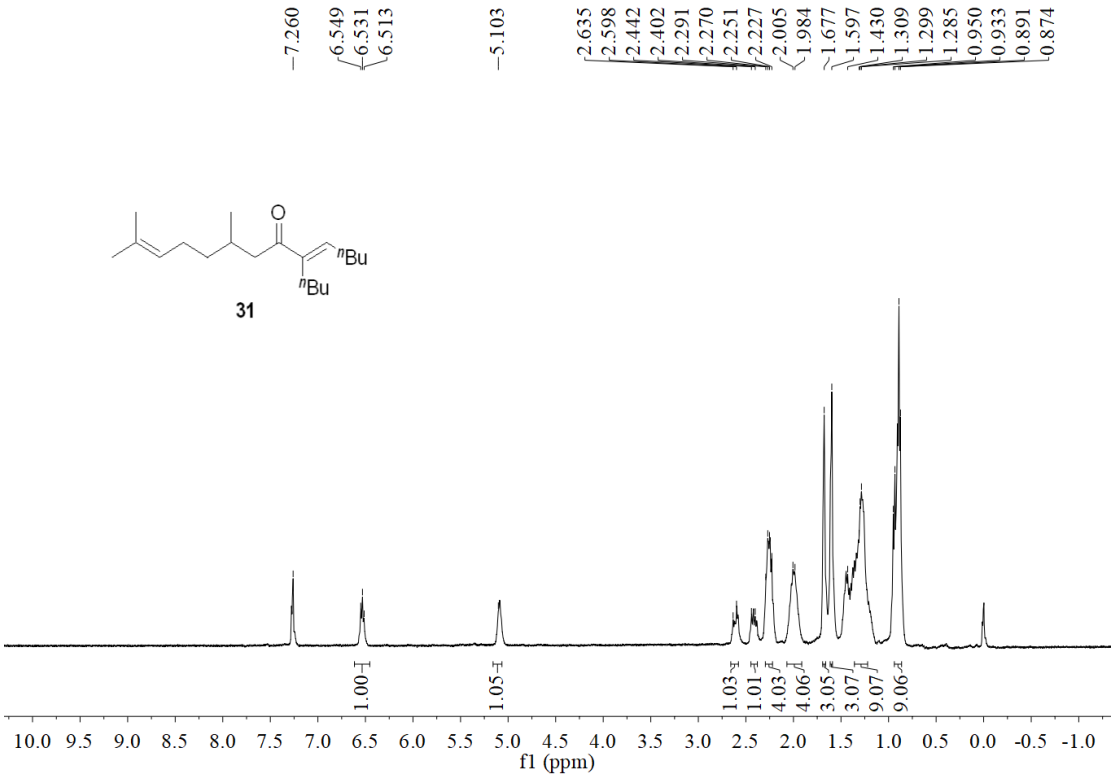

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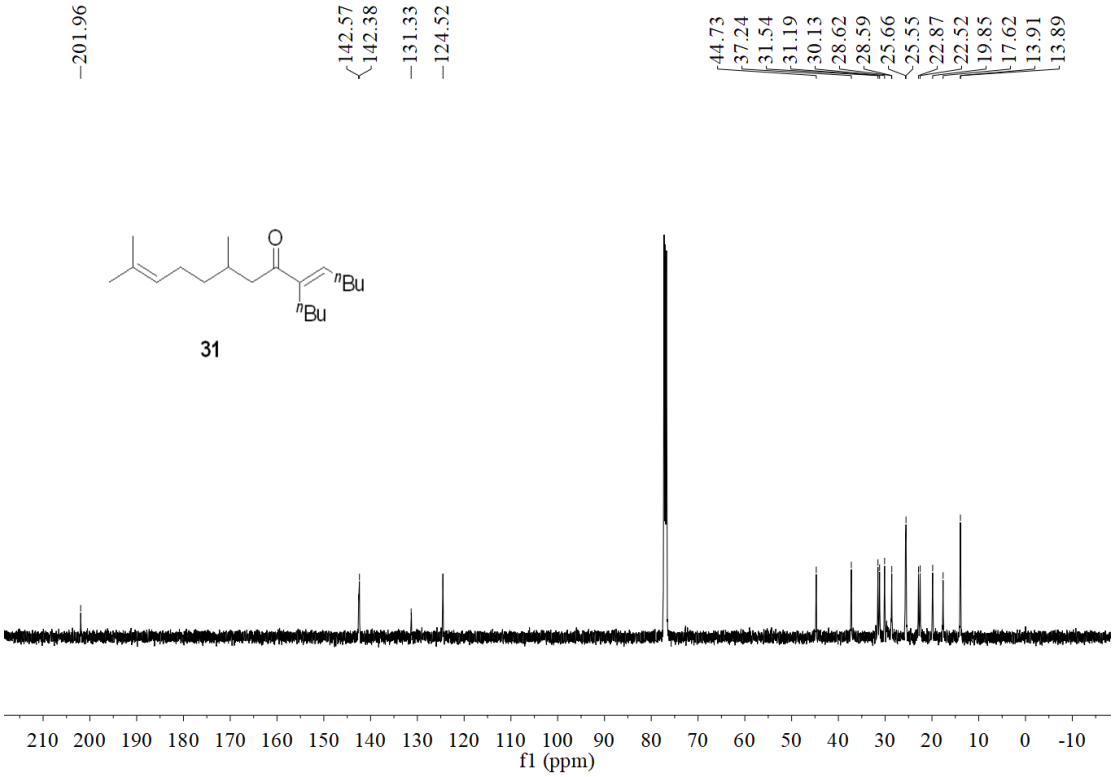
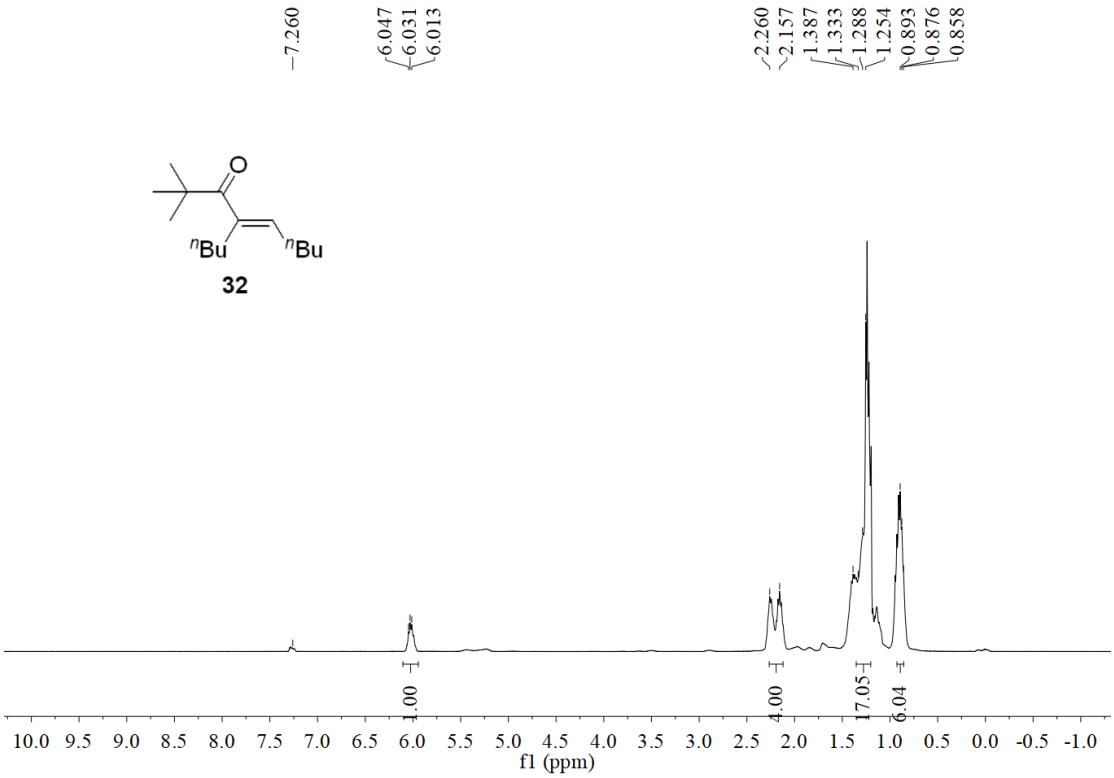
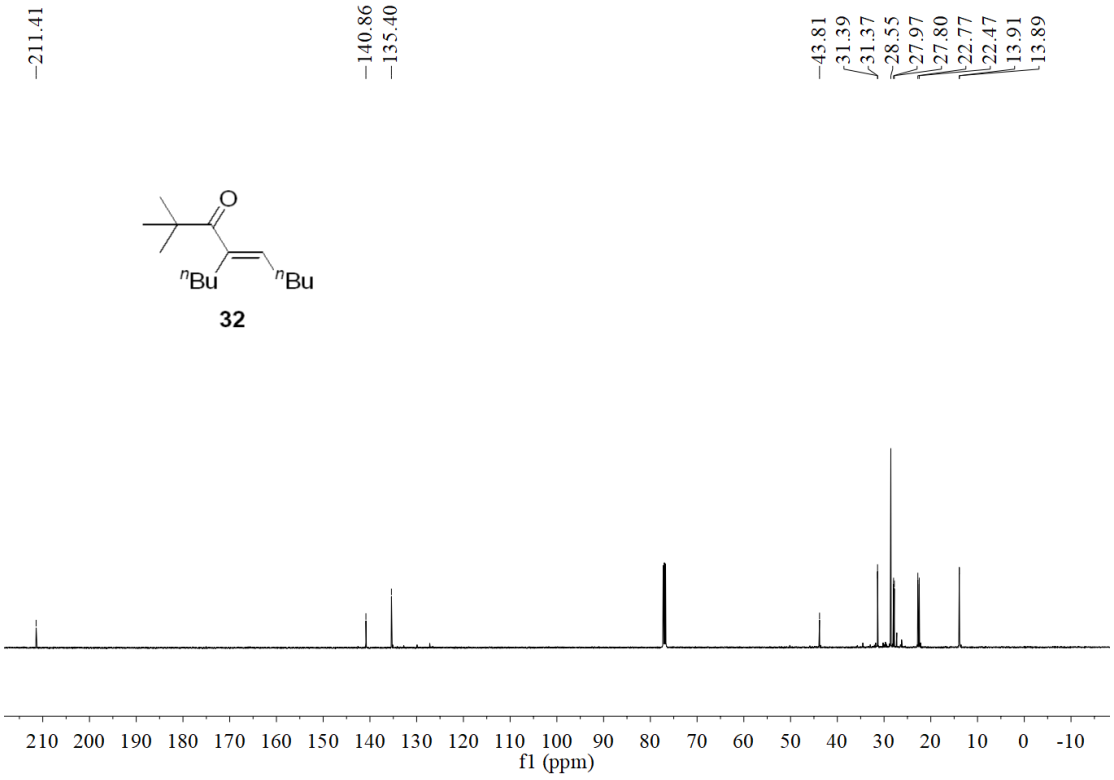


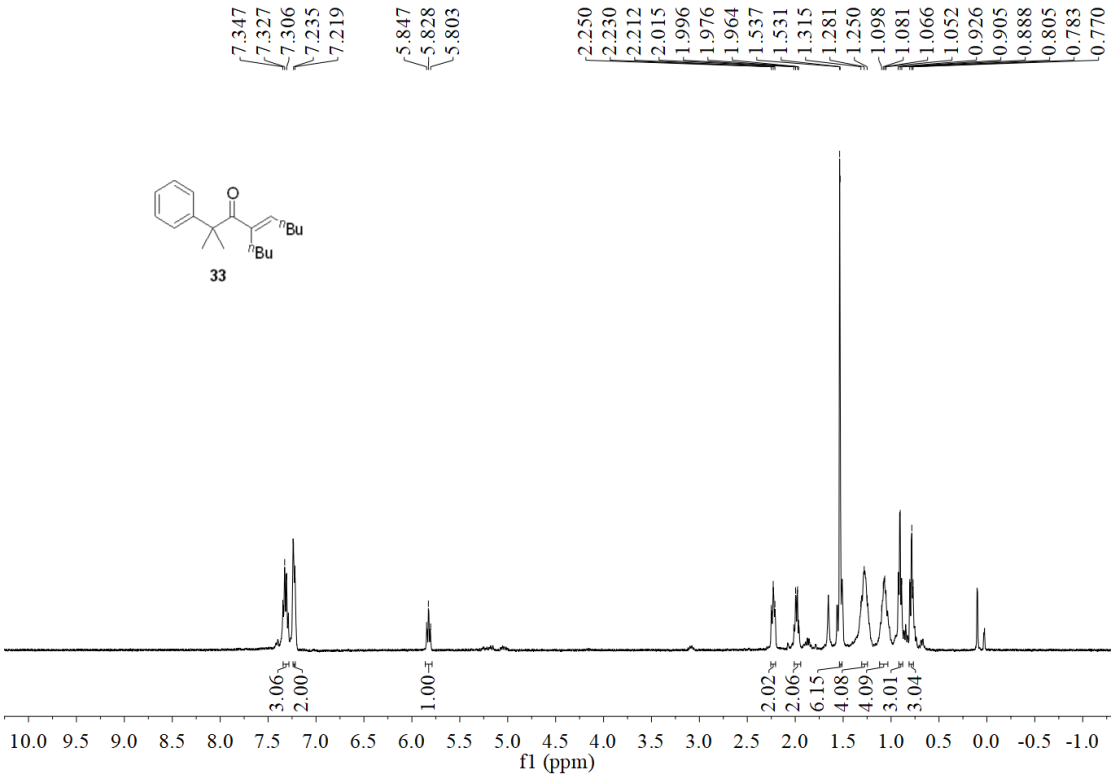
(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



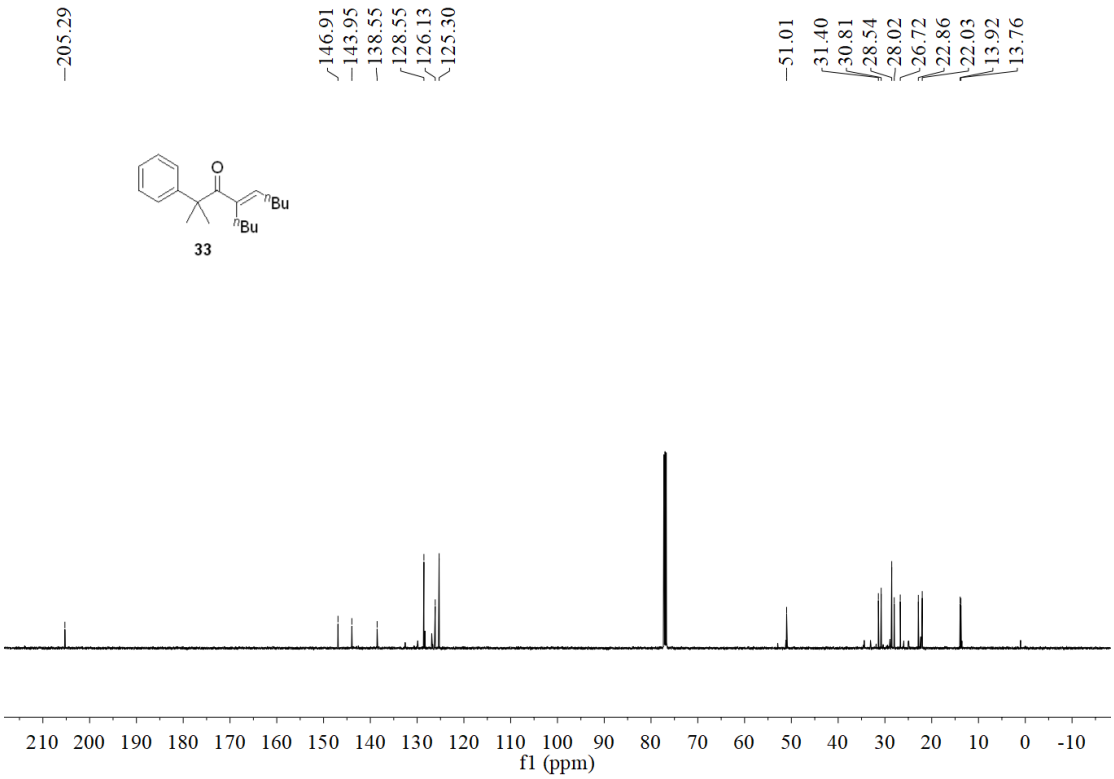
(400 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



(400 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



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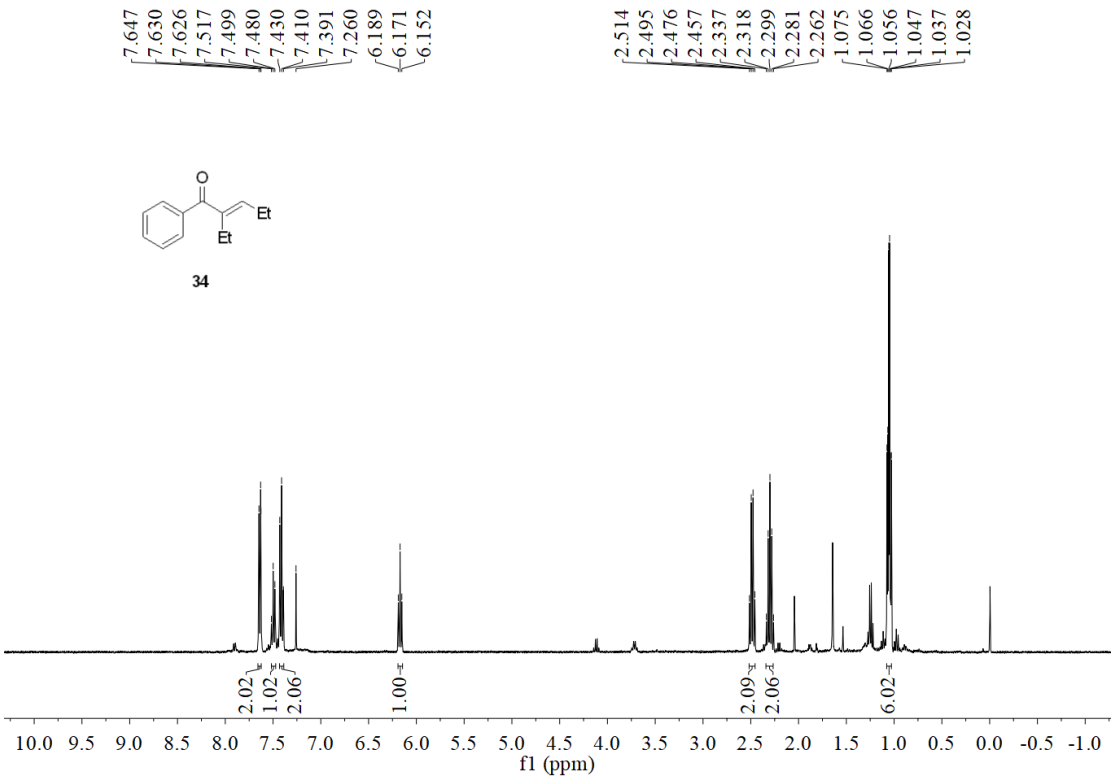

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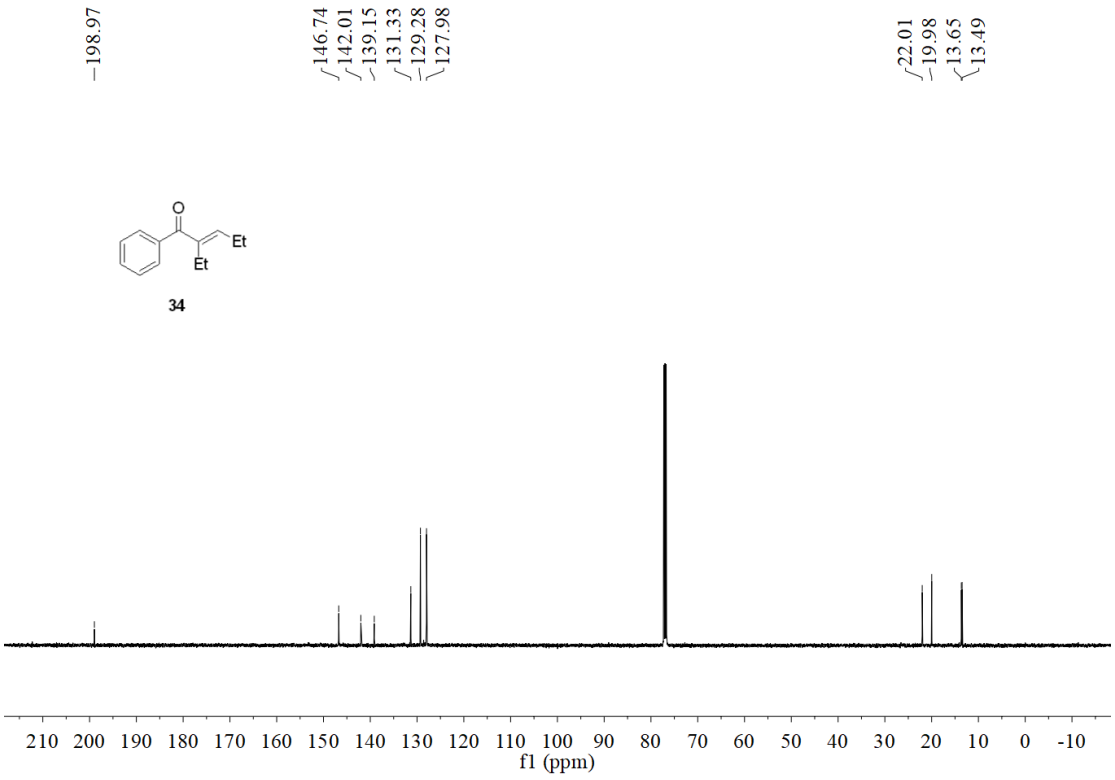

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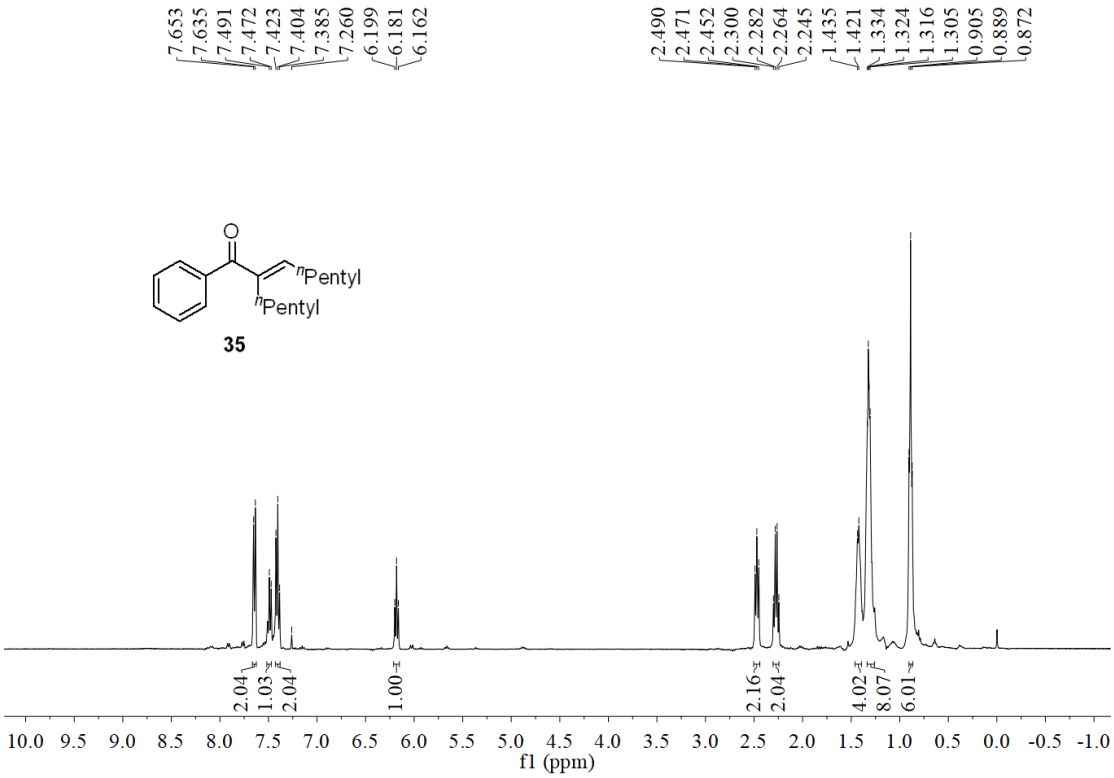

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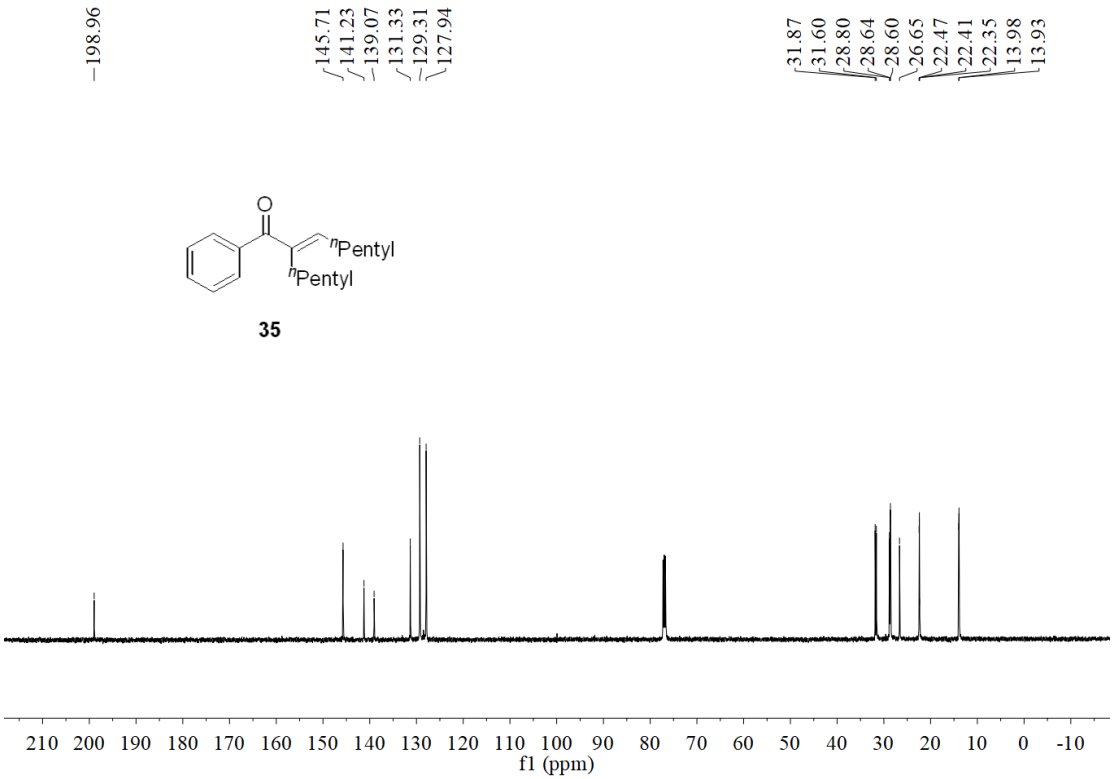

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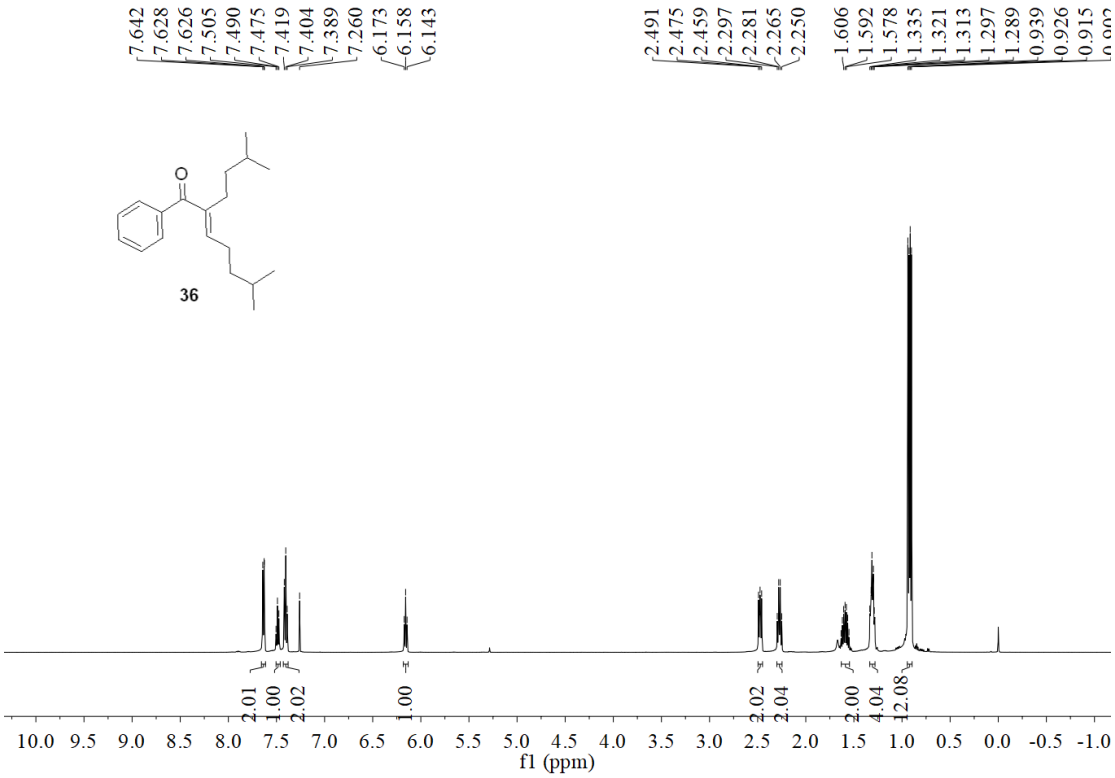

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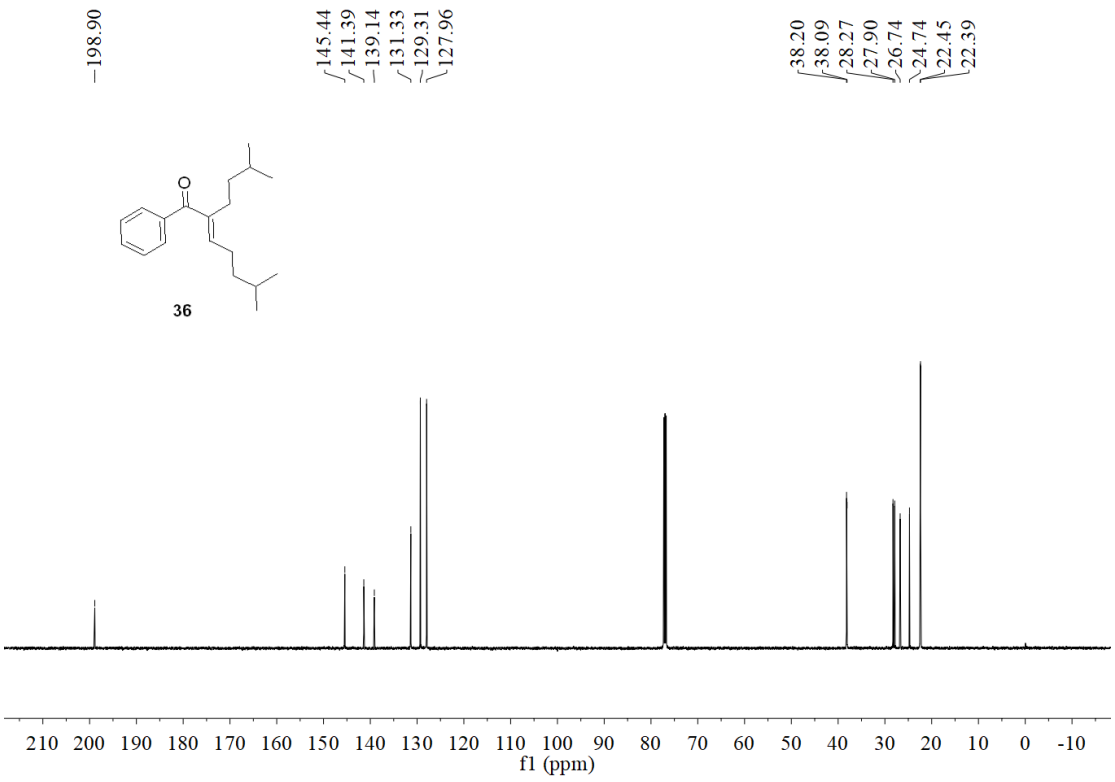
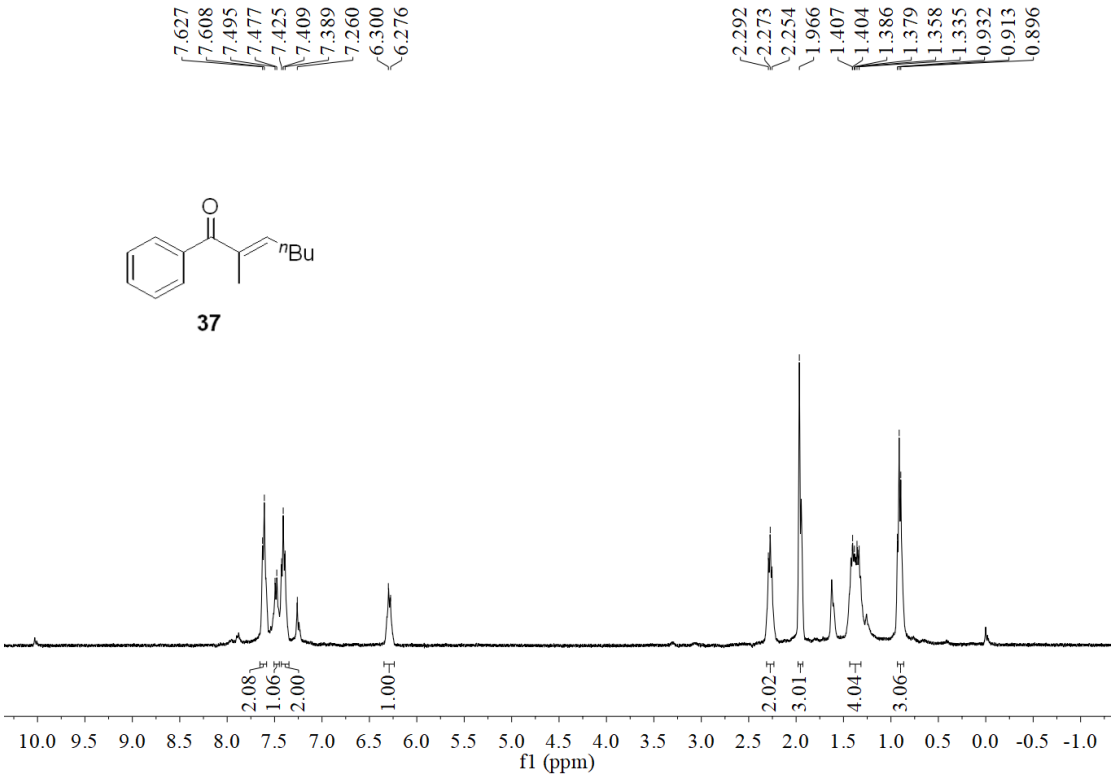
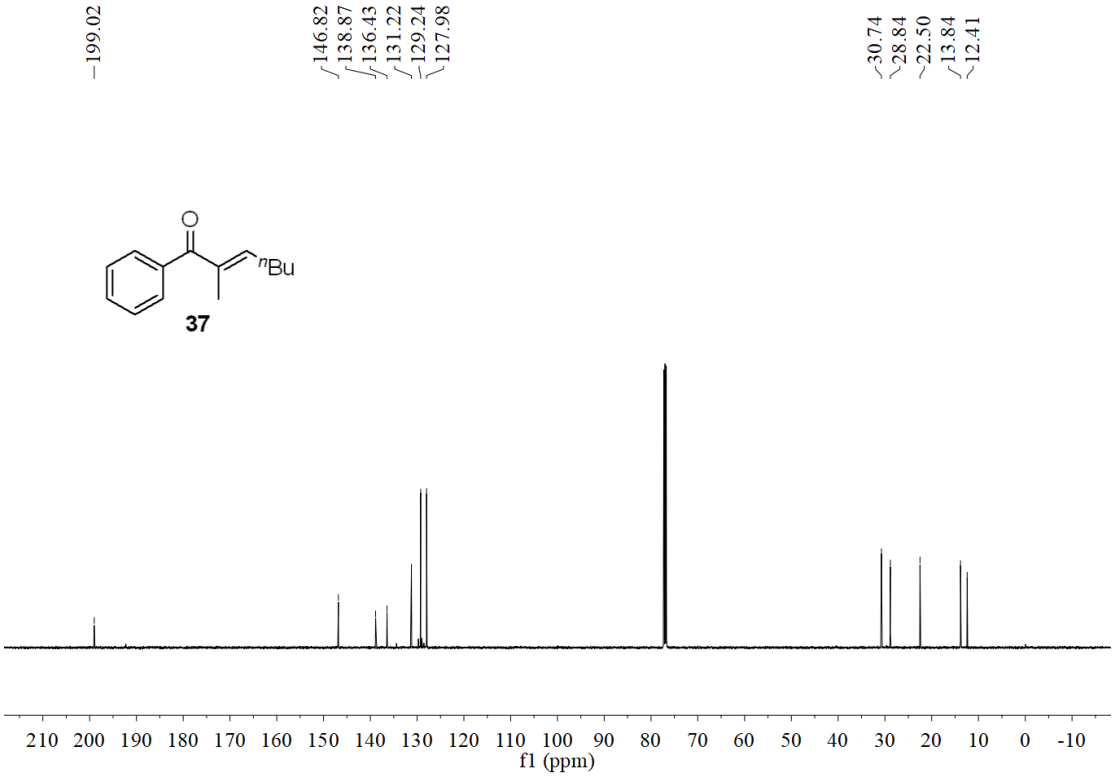


(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)



(400 MHz for ^{1}H NMR with $CDCl_3$ as solvent)



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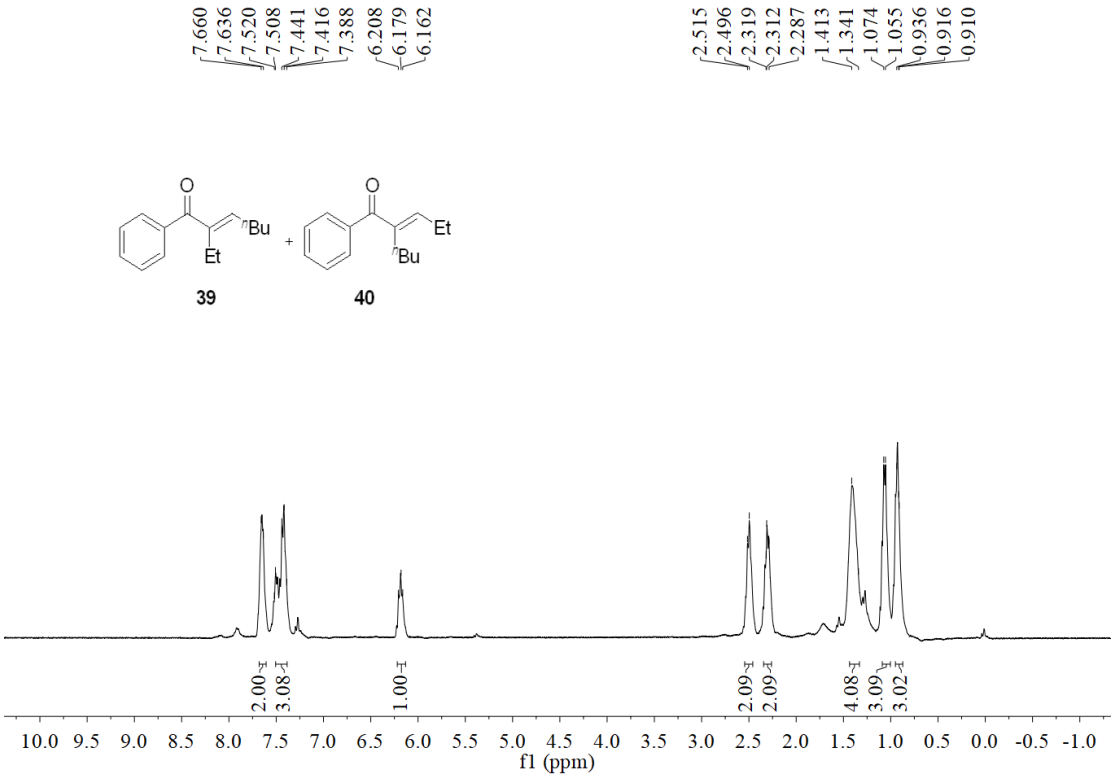

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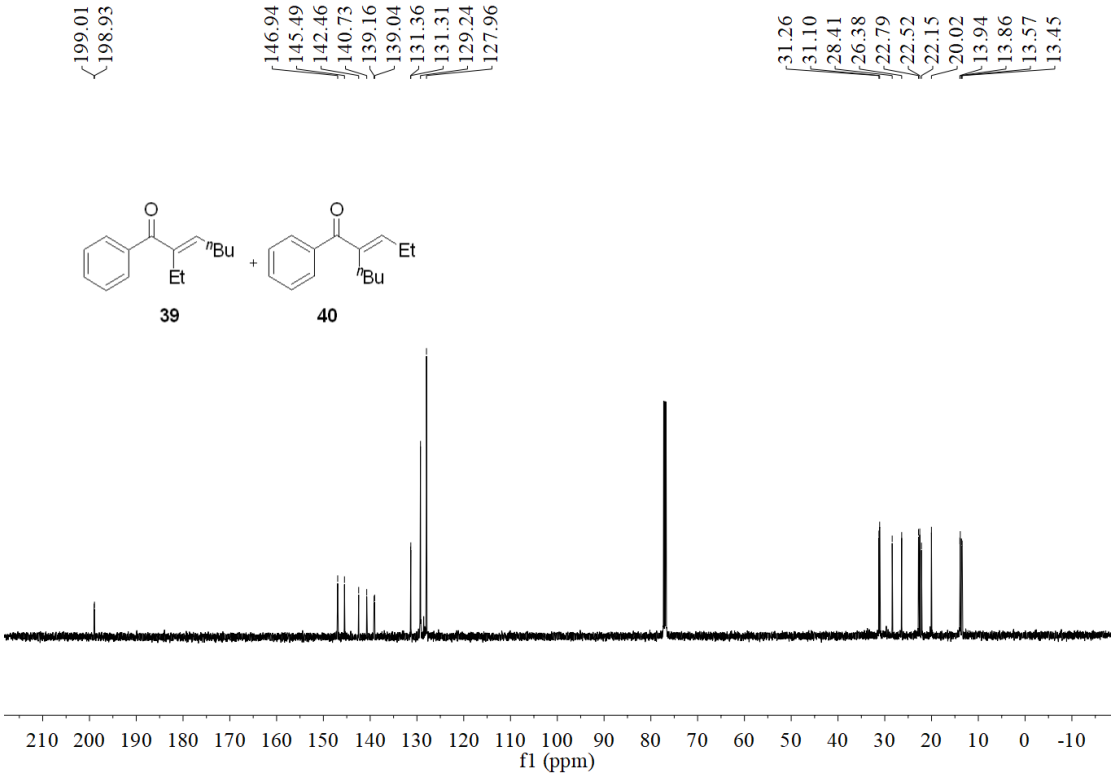

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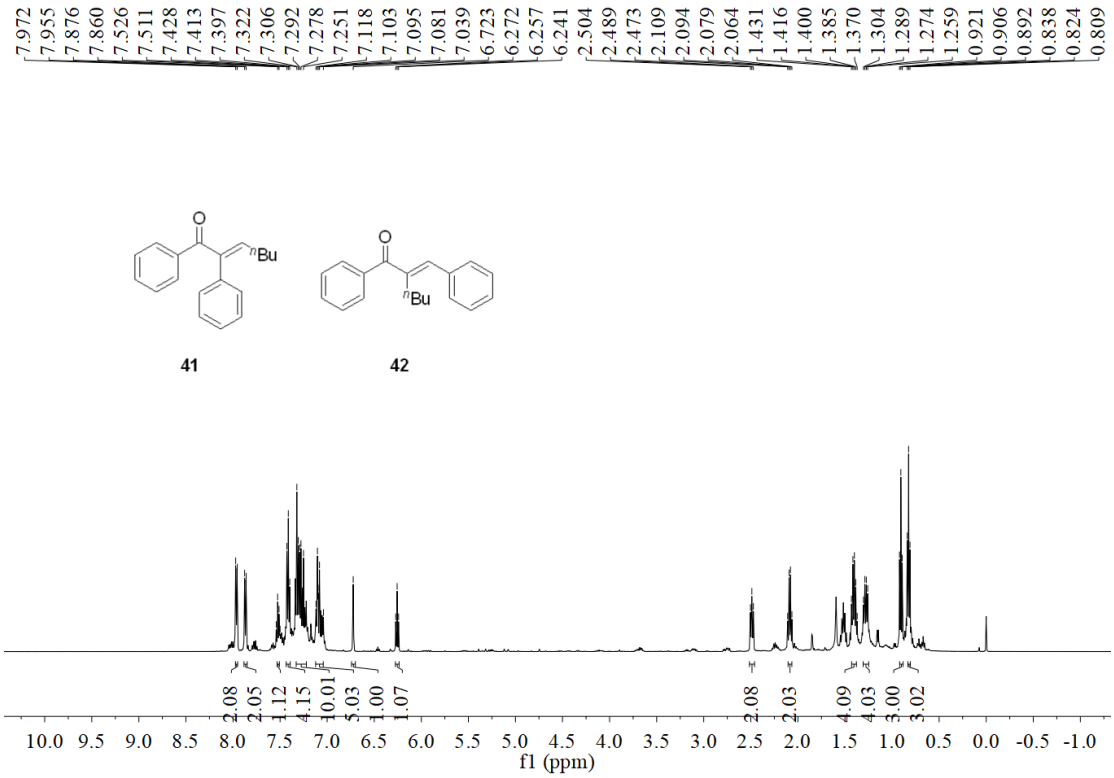

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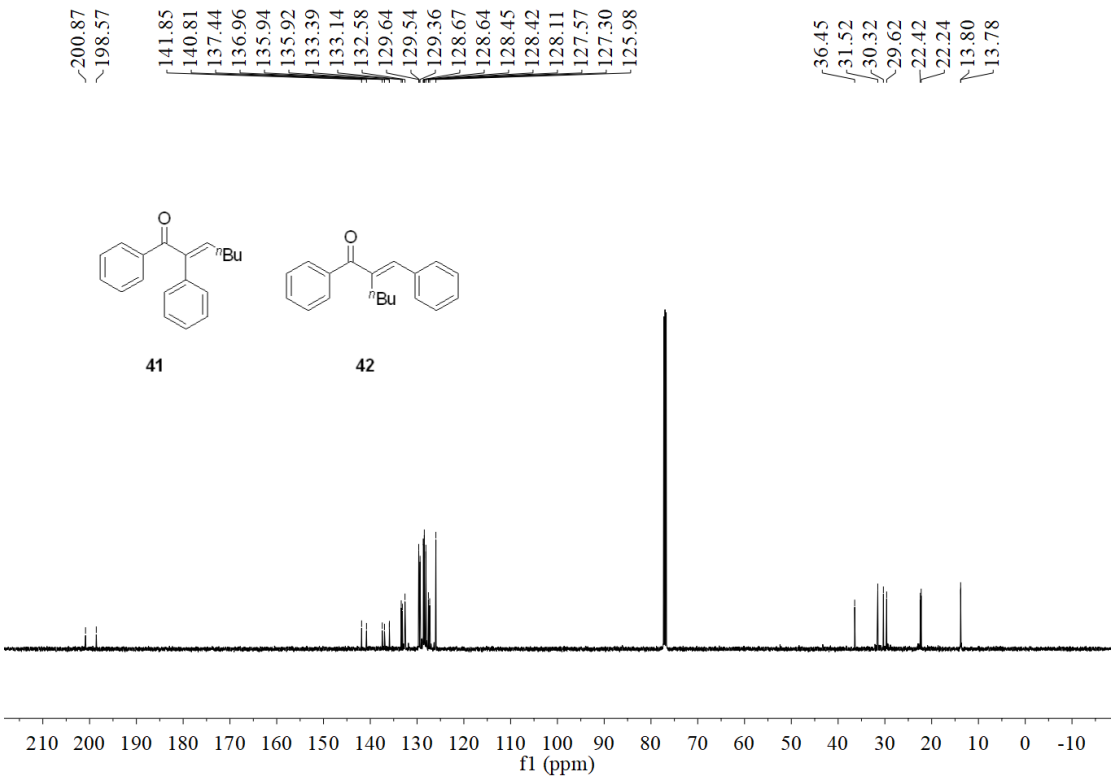

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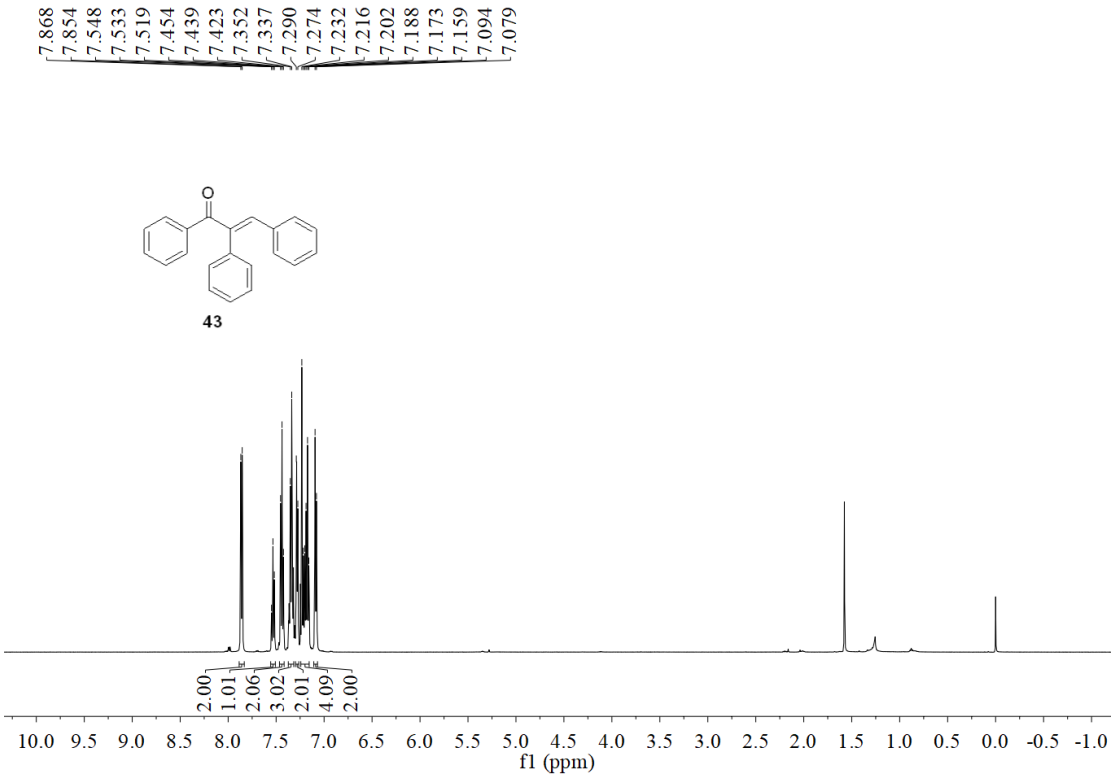

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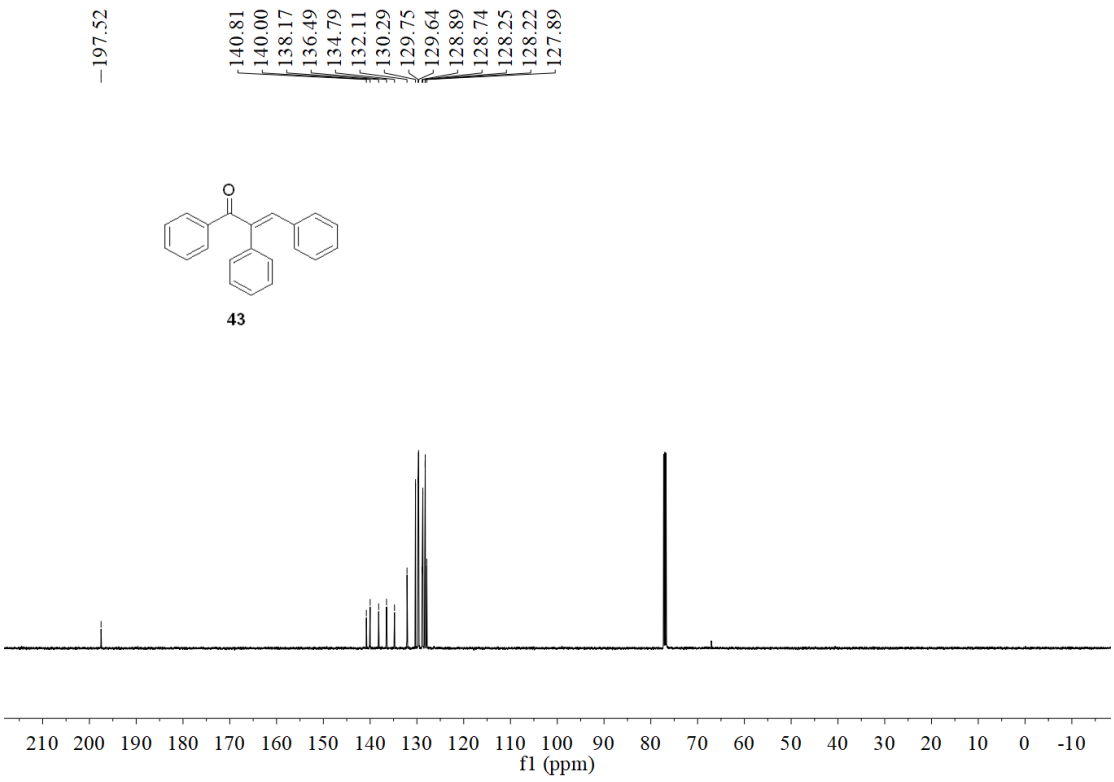

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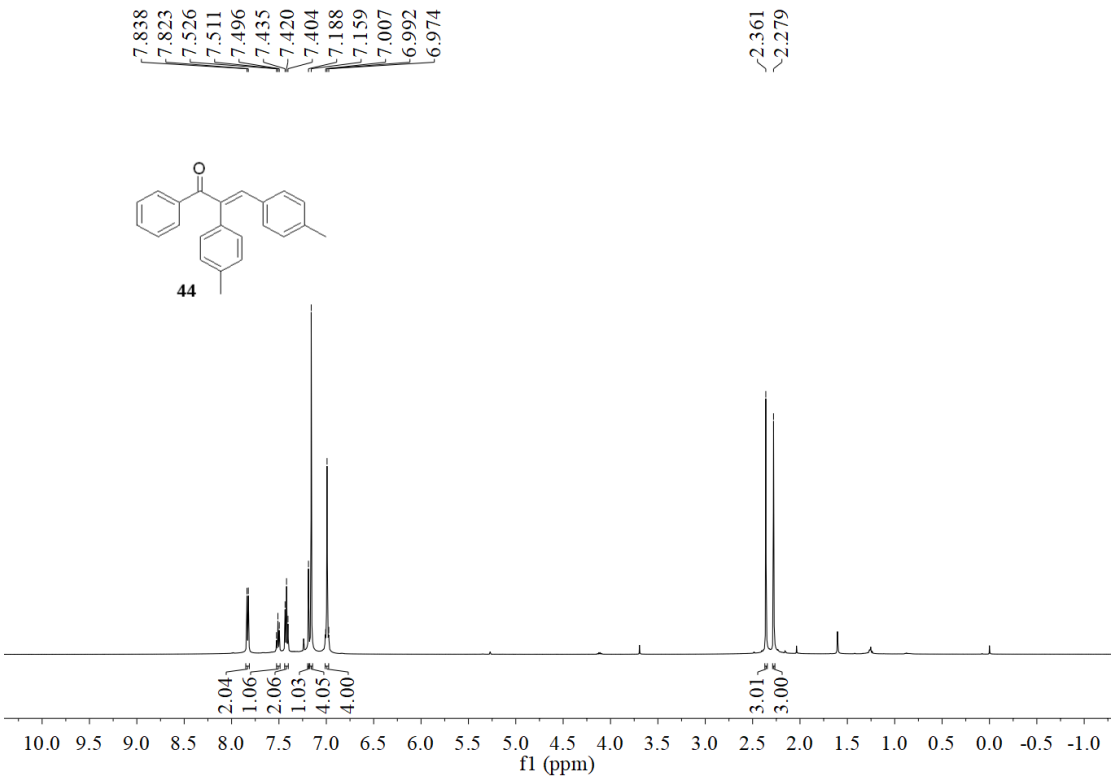

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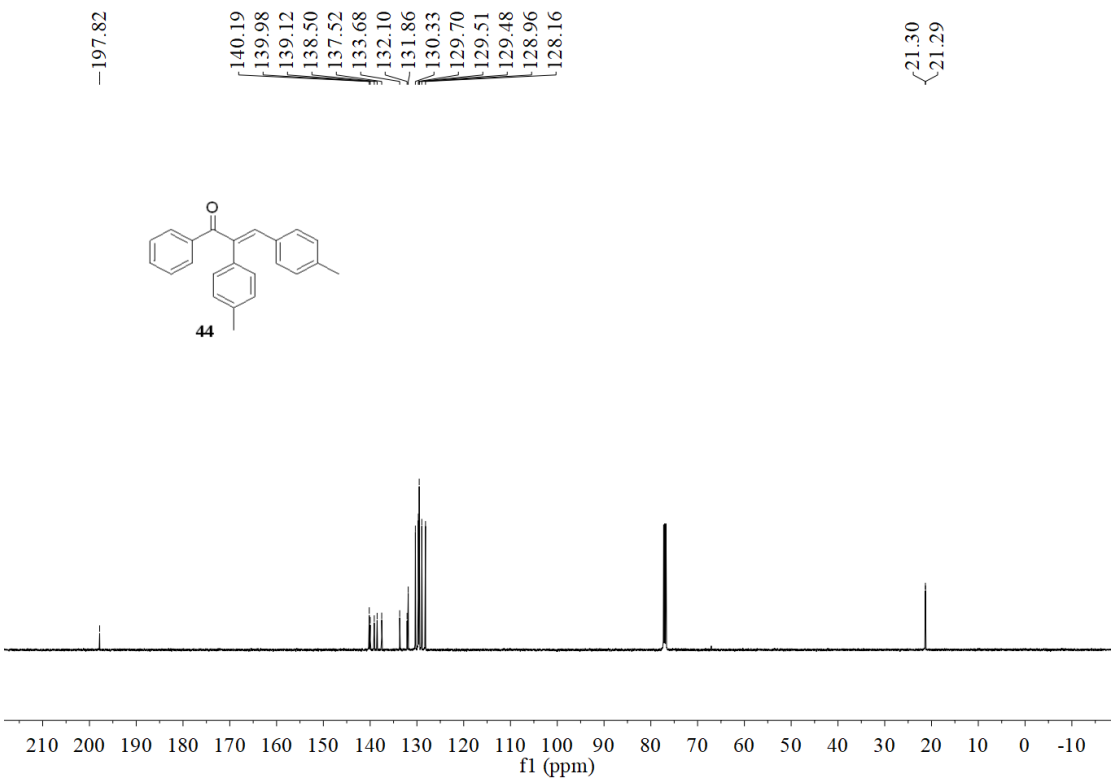

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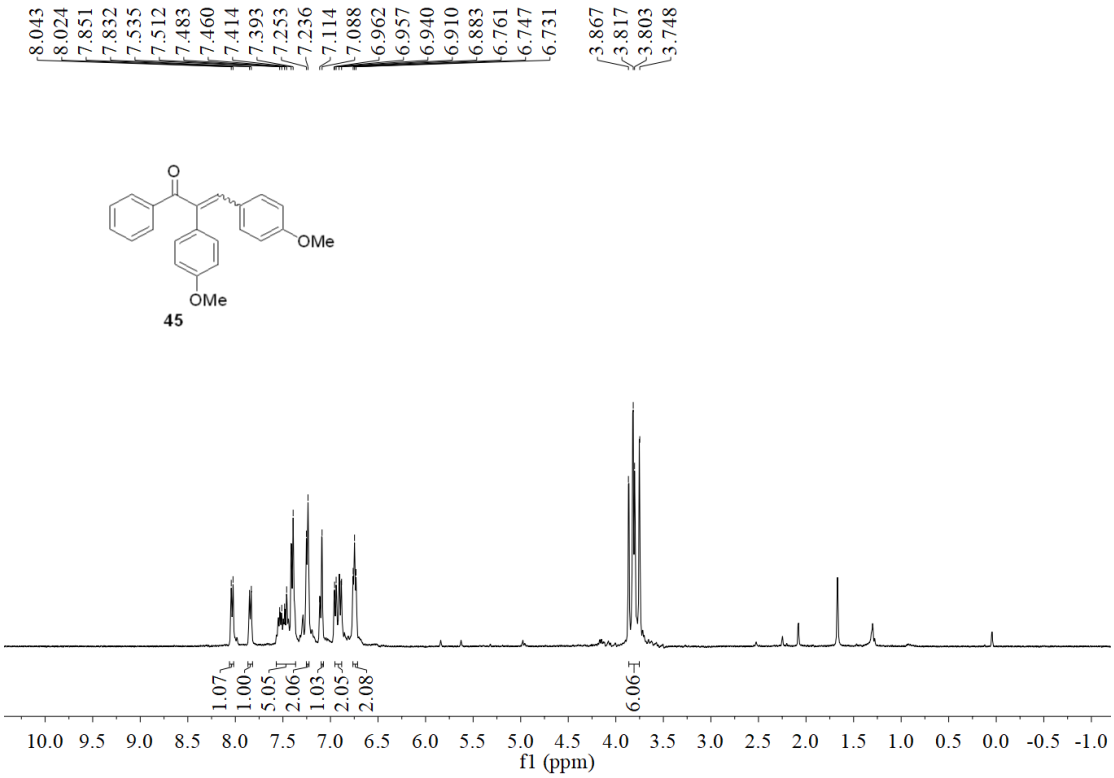

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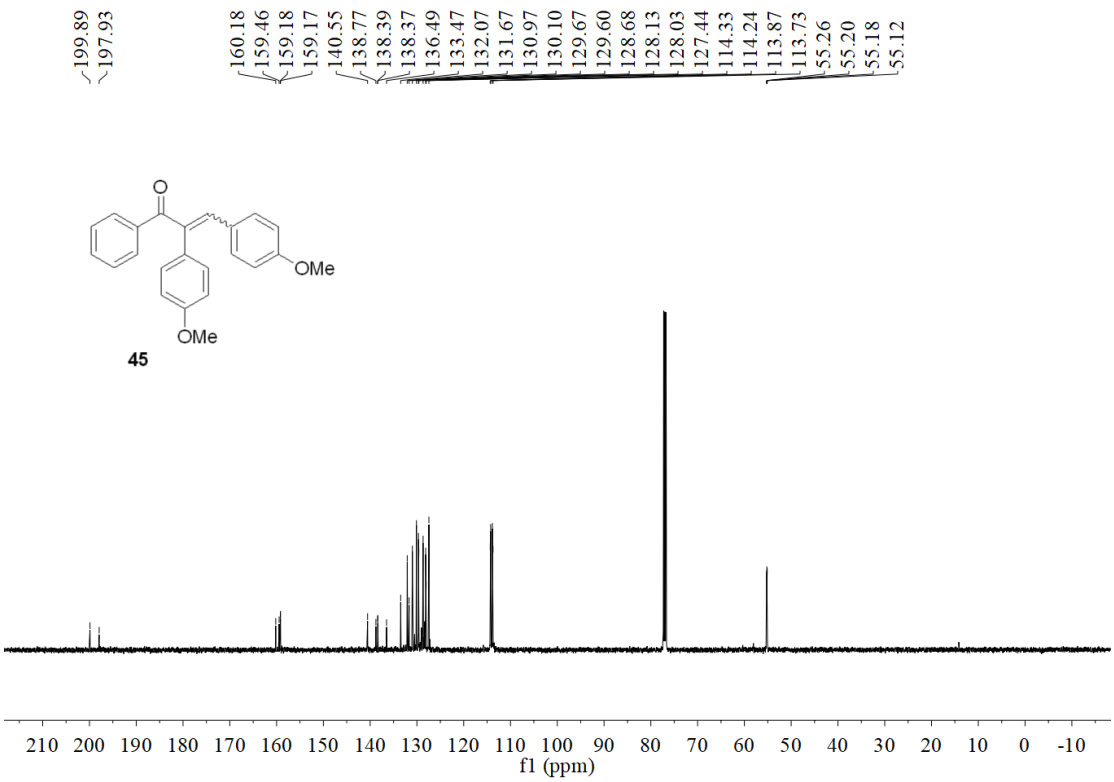

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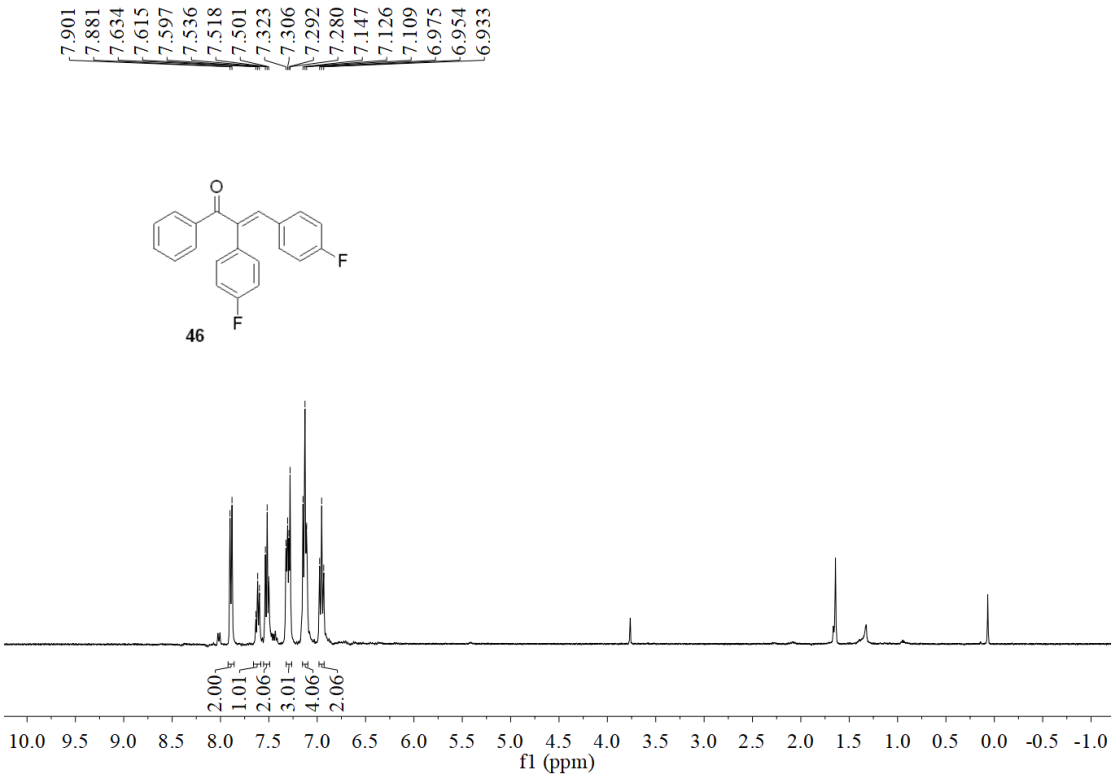

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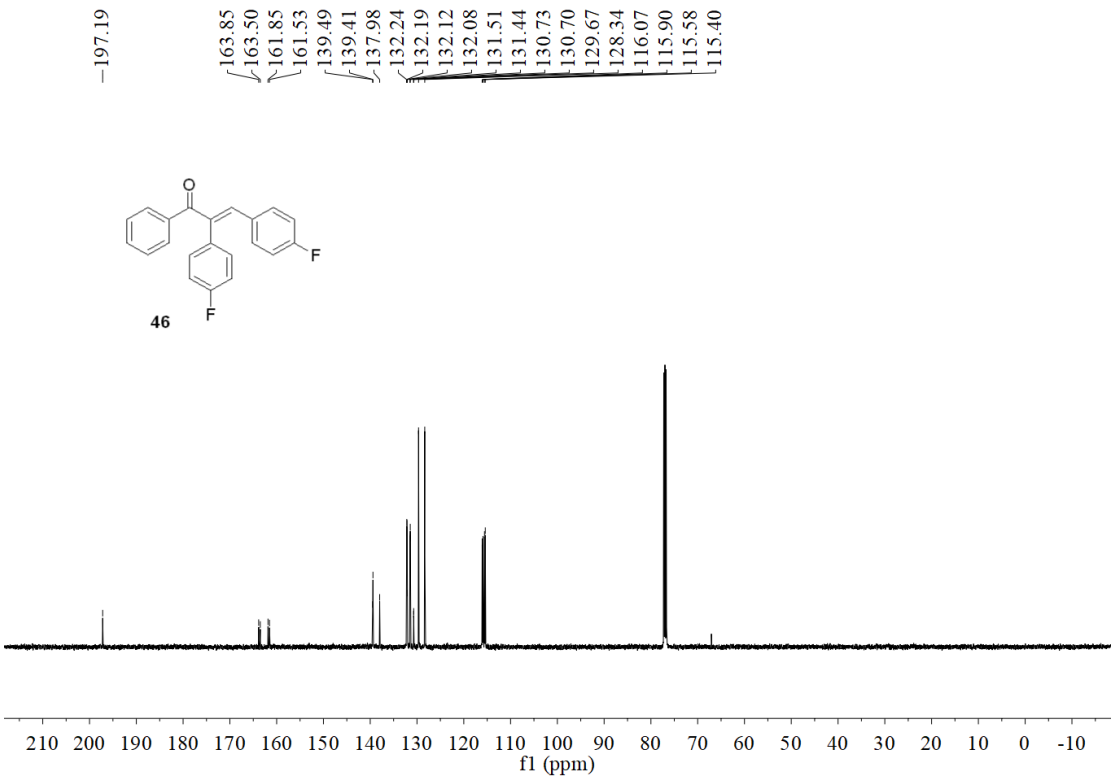

(125 MHz for ^{13}C NMR with $CDCl_3$ as solvent)