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Supplementary Information

Deoxygenative Cross-Electrophile Coupling of Benzyl Chloroformates with Aryl Iodides

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Part I. Experimental Section

1. General Information

(1) Chemicals and Reagents

All reactions were carried out under an atmosphere of N_2. All glassware was oven- or flame-dried prior to use. Ni(acac)$_2$ was purchased from Meryer Chemical Co. (China) and used as received. $MgBr_2$ was purchased from Strem and used as received. Nickel (II) chloride ethylene glycol dimethyl ether complex was synthesized, stored in a glovebox under an atmosphere of N_2 atmosphere. Zinc powder was activated using HCl before use. 4,4'-Dimethoxy-2,2'bipyridine **L4** was purchased from Nine-Ding Chemical Co. (China), and used as received. The anhydrous solvents were purchased from Acros, THF (99.5%, stabilized), CH_3CN (acetonitrile, 99.9%), DMA (*N*, *N*-dimethylacetamide, 99.5%, extra pure). Unless otherwise noted, all other reagents were purchased commercially and used as received.

(2) Physical methods

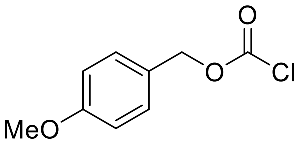
Column chromatography was performed using silica gel 300-400 mesh (purchased from shaoyuan Co., China) as the solid support. All NMR spectra were recorded on Bruker Avance 600MHz spectrometer at STP unless otherwise indicated. ^{1}H NMR and ^{13}C NMR chemical shifts are reported in δ units, parts per million (ppm) relative to the chemical shift of residual solvent. Reference peaks for chloroform in ^{1}H NMR and ^{13}C NMR spectra were set at 7.26 ppm and 77.16 ppm, respectively. The real-time EESI-MS breath experiments were carried out using an Orbitrap Fusion^{TM} Tribrid^{TM} mass spectrometer (Thermo Scientific). Melting point was recorded on a micro melting point apparatus (X-4, YUHUA Co., Ltd, Gongyi, China).

2. Preparation of chloroformate substrates

A general procedure for the preparation of chloroformate substrates (method 1): To a stirred solution of the corresponding alcohol (1 equiv., 20 mmol) in dichloromethane (100 mL) at 0 °C was added triethylamine (2 equiv., 40 mmol) followed by triphosgene (1.2 equiv., 24 mmol). The resulting mixture was stirred at room temperature overnight, and then it was partially evaporated at reduced pressure and diluted with hexane (100 mL). The solid was removed by filtration and the filtrate was evaporated to afford the crude product. Then the crude oily liquid was purified by flash column chromatography, using a mixture of ethyl acetate in petroleum ether (1:99) as eluent. The filtrate was evaporated to afford the corresponding chloroformate in quantitative yield.[1]

A general procedure for the preparation of alcohol substrates (method 2): (Aldehyde/ ketone reduction reaction). To a stirred solution of the corresponding aldehyde or ketone (1 equiv., 30 mmol) in methanol (100 ml) at 0 °C was added sodium borohydride (1.1 equiv., 22 mmol). The resulting mixture was stirred at room temperature for 3h. The reaction solvent was evaporated under reduced pressure. Then the residue was added saturated aqueous ammonium chloride solution (100 mL) and washed with dichloromethane (50 mL × 3). The organic phase was collected, dried over anhydrous Na_2SO_4. After filtration and concentration, the crude mixture was purified and afforded the alcohol in quantitative yield.[1]

4-Methoxybenzyl chloroformate

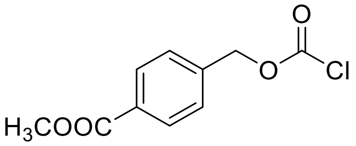


This compound was prepared from 4-methoxybenzyl alcohol (2.8 g, 20.0 mmol) according to the *General Procedure*. The crude residue was purified by silica gel chromatography (2% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 95% yield.

^{1}H NMR (600MHz, $CDCl_3$) δ 7.32-7.31 (m, 2H), 6.89-6.88 (m, 2H), 4.57 (s, 2H), 3.81 (s, 3H).

^{13}C NMR (151 MHz, CDCl3) δ 159.70, 130.07, 114.15, 55.33, 46.30, 29.71.

4-(Methoxycarbonyl) benzyl chloroformate

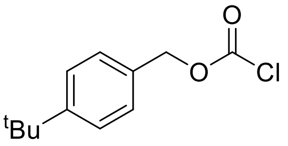


This compound was prepared from methyl (4-hydroxymethyl) benzoate (3.3 g, 20.0 mmol) according to the *General Procedure*. The crude residue was purified by silica gel chromatography (2% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 94% yield.

^{1}H NMR (600MHz, $CDCl_3$) δ 8.04- 8.02 (m, 2H), 7.47-7.45 (m, 2H), 4.61 (s, 2H), 3.92 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.68, 142.31, 130.10, 128.57, 52.32, 45.46.

4-*Tert*-butyl benzyl chloroformate

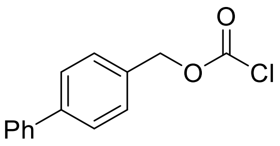


This compound was prepared from 4-*tert*-butylbenzyl alcohol (3.3 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 95% yield.

^{1}H NMR (600MHz, $CDCl_3$) δ 7.38-7.37 (m, 2H), 7.32-7.30 (m, 2H,) 4.56 (s, 2H), 1.31 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 151.63, 134.58, 128.40, 125.75, 46.18, 34.67, 31.33.

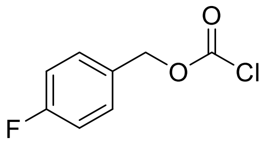
4-Phenyl benzyl chloroformate[2]



This compound was prepared from 4-phenylbenzyl alcohol (3.7 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 96% yield.

^{1}H NMR (600MHz, $CDCl_3$) δ7.60-7.58 (m, 4H), 7.47-7.43 (m, 4H), 7.37-7.35 (m, 1H), 4.64 (s, 2H).

4-Fluorobenzyl chloroformate (6)[3]

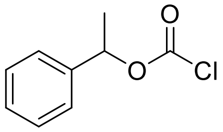


This compound was prepared from 4-fluorobenzyl alcohol (2.5 g, 20.0 mmol) according to the *General Procedure*. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 95% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38-7.35 (m, 2H), 7.06-7.03 (m, 2H), 4.57 (s, 2H).

1-Phenylethyl chloroformate[1]

This compound was prepared from 1-phenethylalcohol (2.5 g, 20.0 mmol) according to the *General Procedure*. The crude residue was purified by silica gel chromatography (1% ethyl acetate/

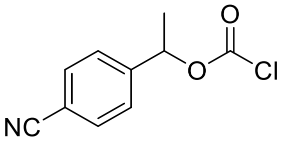


petroleum ether) to yield the product as a colorless oil in 92% yield.

^{1}H NMR (600MHz, $CDCl_3$) δ 7.47-7.45 (m, 2H), 7.40-7.38 (m, 2H), 7.35-7.32 (m, 1H), 5.13 (q, *J* = 6.8 Hz, 1H), 1.89 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 142.83, 128.65, 128.27, 126.52, 58.80, 26.53.

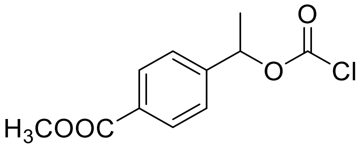
1-(4-Cyanophenyl) ethyl chloroformate



This compound was prepared from methyl 4-(1-hydroxyethyl) benzonitrile (3.0 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (5% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 98% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65-7.64 (m, 2H), 7.53-7.52 (m, 2H), δ 5.08 (q, *J* = 6.8 Hz, 1H), 1.83 (d, *J* = 6.8 Hz, 3H).

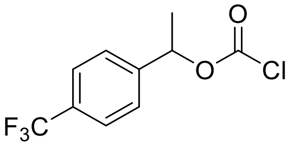
1-(4-(Methyl formate) phenyl) ethyl) chloroformate



This compound was prepared from methyl 4-(1-hydroxyethyl) benzoate (3.6 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (3% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 95% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03-8.01 (m, 2H), 7.49-7.47 (m, 2H), δ 5.10 (q, *J* = 6.8 Hz, 1H), 3.91 (s, 3H), 1.84 (d, *J* = 6.8 Hz, 3H).

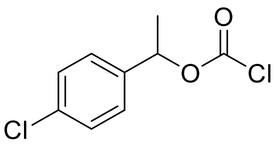
1-(4-(Trifluoromethyl) phenyl) ethyl chloroformate



This compound was prepared from methyl 1-(4-(trifluoromethyl) phenyl) ethan-1-ol (3.8 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (2% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 97% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63-7.62 (m, 2H), 7.55-7.54 (m, 2H), δ 5.11 (q, *J* = 6.8 Hz, 1H), 1.86 (d, *J* = 6.9 Hz, 3H).

1-(4-Chlorophenyl) ethyl chloroformate

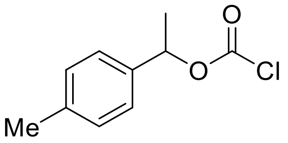


This compound was prepared from methyl 1-(4-chlorophenyl) ethan-1-ol (3.2 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 90% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32-7.28 (m, 4H), δ 5.02 (q, *J* = 6.8 Hz, 1H), 1.79 (d, *J* = 6.8 Hz, 3H).

1-(*P*-tolyl) ethyl chloroformate

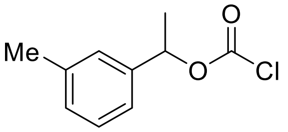
This compound was prepared from methyl 1-(p-tolyl) ethan-1-ol (2.8 g, 20.0 mmol) which was



obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 97% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30-7.28 (m, 2H), 7.15-7.13 (m, 2H), δ 5.06 (q, *J* = 6.8 Hz, 1H), 2.33 (s, 3H), 1.82 (d, *J* = 6.8 Hz, 3H).

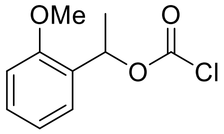
1-(*M*-tolyl) ethyl chloroformate



This compound was prepared from methyl 1-(m-tolyl) ethan-1-ol (2.8 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 98% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25-7.21 (m, 3H), 7.12-7.11 (m, 1H), δ 5.07 (q, *J* = 6.8 Hz, 1H), 2.37 (s, 3H), 1.85 (d, *J* = 6.8 Hz, 3H).

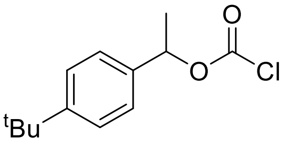
1-(2-Methoxyphenyl) ethyl chloroformate



This compound was prepared from methyl 1-(2-methoxyphenyl) ethan-1-ol (3.1 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (2% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 95% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53-7.51 (m, 1H), 7.27-7.25 (m, 1H), δ 6.97 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 8.3 Hz, 1H), 5.60 (q, *J* = 6.8 Hz, 1H), 3.84 (s, 3H) 1.80 (d, *J* = 6.9 Hz, 3H).

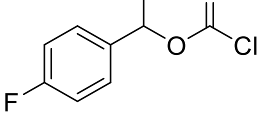
1-(4-(*Tert*-butyl) phenyl) ethyl chloroformate



This compound was prepared from 1-(*tert*-butyl) methyl-benzenemethanol (3.6 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 90% yield.

^{1}H NMR (600MHz, $CDCl_3$) δ 7.39-7.37 (m, 2H), 7.36-7.35 (m, 2H), 5.10 (q, *J* = 6.8 Hz, 1H), 1.85 (d, *J* = 6.8 Hz, 3H), 1.32 (s, 9H).

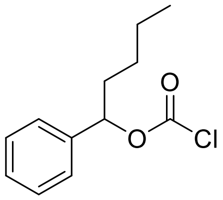
1-(4-Fluorophenyl) ethyl chloroformate



This compound was prepared from 1-(4-fluorophenyl) ethanol (2.8 g, 20.0 mmol) according to the *General Procedure*. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 92% yield.

^{1}H NMR (600MHz, $CDCl_3$) δ 7.28-2.26 (m, 2H), 7.19-7.18 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 1H), 1.61 (d, *J* = 7.2 Hz, 3H).

1-Phenylpentyl chloroformate

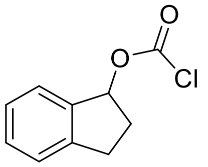


This compound was prepared from fenipentol (3.3 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (2% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 96% yield.

^{1}H NMR (600MHz, $CDCl_3$) δ 7.41-7.36 (m, 4H), 7.34-7.31 (m, 1H), 4.87 (t, 1H), 2.19-2,13 (m, 1H), 2.09-2.03 (m, 1H), 1.53-1.46 (m, 1H), 1.38-1.28 (m. 3H), 0.92 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 142.04, 128.60, 128.18, 126.95, 63.89, 39.75, 29.23, 22.14, 13.91.

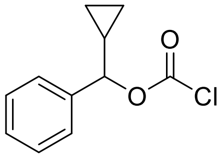
2,3-Dihydro-1H-inden-1-yl chloroformate



This compound was prepared from methyl 1-indanol (2.7 g, 20.0 mmol) according to the *General Procedure*. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 82% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.40 (d, *J* = 7.1 Hz, 1H), 7.24-7.21 (m, 3H), 5.40-5.38 (m, 1H), 3.15 (dt, *J* = 15.4, 7.5 Hz, 1H), 2.88-2.83 (m, 1H), 2.58-2.52 (m, 1H), 2.37-2.32 (m, 1H).

Cyclopropyl(phenyl)methyl chloroformate (20)

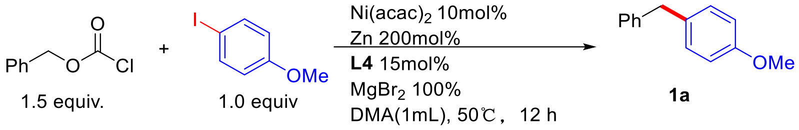


This compound was prepared from cyclopropyl(phenyl)methanol (3.0 g, 20.0 mmol) which was obtained by reduction of the corresponding aldehyde precursor using method 2. The crude residue was purified by silica gel chromatography (1% ethyl acetate/ petroleum ether) to yield the product as a colorless oil in 92% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ7.31-7.29 (m, 3H), 7.23-7.22 (m, 1H), 4.29 (d, *J* = 9.2 Hz, 1H), 1.59-1.53 (m, 1H), 0.84-0.79 (m, 1 H), 0.58-0.55 (m, 1H), 0.44-0.40 (m, 1H).

3. Control experiments and optimization

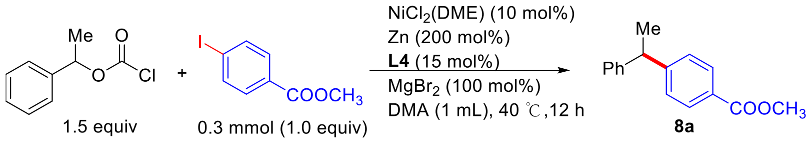
Table S1. Variations from the conditions for the formation of **1a.**[a]



entry	variation from the condition1	yield for **1a**[a]
1	r.t	87%
2	30℃	88%
3	40℃	96%
4	$MgCl_2$ (1.0 equiv)	64%
5	$MgBr_2$ (2.0 quiv)	89%
6	MgI_2 (1.0 equiv)	56%

[a] NMR yield using 2,5-dimethylfuran as the internal reference

Table S2. Variation of reaction conditions for the coupling of secondary chloroformate derivatives and aryl iodide.[a]



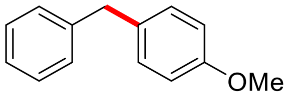
Entry	deviation from the standard conditions	Yield (%)[a]
1	none	91
2	$Ni(acac)_2$ instead of $NiCl_2$(DME)	63
3	NiI_2 instead of $NiCl_2$(DME)	69
4	$NiCl_2$ instead of $NiCl_2$(DME)	73
5	**L2** instead of **L4**	79
6	**L6** instead of **L4**	44
7	w/o nickel catalyst	ND
8	w/o Zn	ND
9	w/o ligand	34
10	w/o $MgBr_2$	58
11	chloroformate (1.2 equiv)	88
12	chloroformate (1.0 equiv)	79
13	DMA 0.5ml	84
14	DMA 2.0ml	59
15	4-chloroanisole instead of 4-iodoanisole	34
16	4-bromoanisole instead of 4-iodoanisole	66

[a] NMR yield using 2,5-dimethylfuran as the internal reference

4. Characterization Data of Compounds

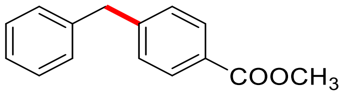
To a flame-dried Schlenk tube equipped with a stir bar was loaded aryl iodide (0.3 mmol, 100 mol%), followed by addition of ligand 4,4'-dimethoxy-2,2'-bipyridine **L4** (0.045 mmol, 15 mol%), zinc powder (0.6 mmol, 200 mol%), and $MgBr_2$ (0.3 mmol, 100 mol%). The tube was moved into a dry glovebox, at which point Ni (II) catalyst (0.03 mmol, 10 mol%) was added. The tube was capped with a rubber septum and moved out of the glovebox. *N*, *N*-Dimethyl acetamide (1 mL) and benzyl chloroformate (0.45 mmol, 150 mol%) were added by syringes. The mixture was stirred for 12 h under N_2 atmosphere at 40 °C, then directly loaded onto a silica column without work-up. The residue in the reaction vessel was rinsed with small amount of petroleum ether. Flash column chromatography provided the product as a solid or oil.

1-Benzyl-4-methoxybenzene (1a)[6]



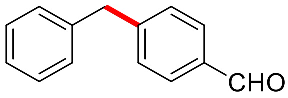
Yield 98%. Light yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.30-7.27 (m, 2H), 7.21 – 7.18 (m, 2H), 7.11 (d, *J* = 8.6 Hz, 1H), 6.84 (d, *J* = 8.6 Hz, 1H), 3.94 (s, 2H), 3.79(s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$, ppm) δ 157.98, 141.60, 133.27, 129.88, 128.83, 128.44, 125.99, 113.89, 55.25, 41.04. HRMS (ESI) $C_{14}H_{14}O$ for [+H] calcd. 199.1123; found 199.1118.

Methyl 4-benzylbenzoate (1b)[7]



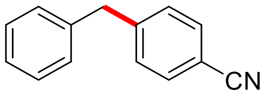
Yield 93%. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$, ppm): δ 7.97 (d, *J*=8.31, 2H), 7.30-7.26 (m, 2H), 7.24- 7.16 (m, 3H), 4.03 (s, 2H), 3.89 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$, ppm): δ 167.20, 146.63, 140.22, 129.88, 129.04, 128.71, 128.17, 126.48, 52.12, 42.01. HRMS (ESI) $C_{15}H_{14}O_2$ for [+H] calcd. 227.1075; found 227.1068.

4-Benzylbenzaldehyde (1c)[8]



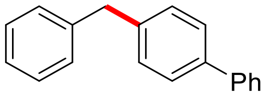
Yield 83%. Light yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.00 (s, 1H), δ 7.83 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 7.9 Hz, 2H), 7.35-7.31 (m, 3H), 7.21 (d, *J* = 7.5 Hz, 1H), 4.08 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.02, 148.47, 139.78, 134.70, 130.49, 130.08, 129.59, 129.00, 128.72, 126.55, 42.12.

4-Benzylbenzonitrile (1d)[9]



Yield 89%. Light yellow solid. m. p. 45-46 °C. ^{1}H NMR (600 MHz, $CDCl_3$, ppm): δ 7.57 (d, *J*=8.36Hz, 2H), 7.55-7.29 (m, 3H), 7.29-7.23 (m,2H), 7.16-7.14 (m, 2H), 4.02 (s, 2H). ^{13}C NMR (151 MHz, CDCl3) δ 146.80, 139.40, 132.33, 129.69, 129.01, 128.82, 126.72, 119.04, 110.07, 42.00. HMRS (ESI) $C_{14}H_{11}N$ for [+H] calcd. 194.0969; found 194.0967.

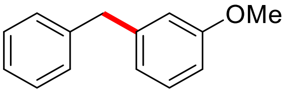
4-Benzyl-1,1'-biphenyl (1e)[10]



Yield 92%. Pale yellow solid. m.p. 84-85 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.3 Hz, 2H), 7.50 (d, *J* = 8.1 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.31-7.27 (m, 3H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.22-7.18 (m, 3H), 4.00

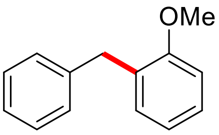
(s, 2H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 141.10, 140.35, 139.14, 129.43, 129.07, 128.82, 128.62, 127.31, 127.18, 127.11, 126.25, 41.69.

1-Benzyl-3-methoxybenzene (1f)[11]



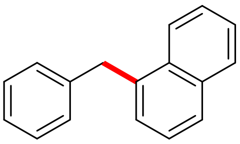
Yield 94%. Light yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.29-7.26 (m, 2H), 7.23-7.19 (m, 4H), 7.08-7.07 (m, 1H), 6.90-6.87 (m, 2H), 4.00 (s, 2H), 3.80 (s,3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 159.80, 142.77, 140.99, 129.48, 128.99, 128.53, 126.17, 121.45, 114.87, 111.38, 55.17, 42.03. HMRS (ESI) $C_{14}H_{14}O$ for [+H] calcd. 194.1123; found 194.1119.

1-Benzyl-2-methoxybenzene (1g)[12]



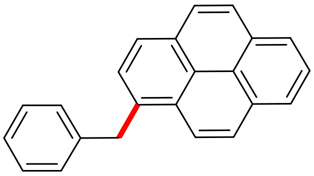
Yield 88%. Light yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$, ppm) δ 7.27 (t, J = 7.6 Hz, 2H), 7.20-7.17 (m, 4H), 6.78 (d, J = 7.5 Hz, 1H), 6.74-6.73 (m, 2H), 3.94 (s, 2H), 3.74 (s, 3H). ^{13}C NMR (151 MHz, CDCl$_3$, ppm) δ 159.78, 142.76, 140.97, 129.46, 128.97, 128.51, 126.15, 121.44, 114.85, 111.37, 55.16, 42.02. HMRS (ESI) $C_{14}H_{14}O$ for [+H] calcd. 194.1123; found 194.1120.

1-Benzylnaphthalene (1h)[13]



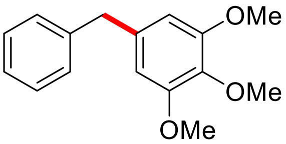
Yield 95%. Yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.97-7.95 (like d, 1H), 7.83-7.81 (m, 1H), δ 7.72 (d, J = 8.2 Hz, 1H), 7.43-7.37 (m, 3H), 7.26-7.22 (m, 3H), 7.17-7.15 (m, 3H), 4.41 (s, 2H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 140.71, 136.70, 134.02, 132.22, 128.82, 128.74, 128.52, 127.41, 127.23, 126.13, 126.04, 125.63, 124.35, 39.11. HMRS (ESI) $C_{17}H_{14}$ for [+H] calcd. 219.1174; found 219.1124.

1-Benzylpyrene (1i)



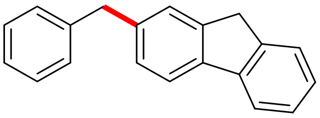
Yield 93%. Pale yellow solid. m.p. 86-87 °C. ^{1}H NMR (600 MHz, CDCl$_3$) δ 8.20 (d, J = 9.2 Hz, 1H), 8.13-8.08 (m, 3H), 8.01-7.99 (m, 3H), 7.96-7.93 (m, 1H), 7.83 (d, J = 7.7 Hz, 1H), 7.26-7.23 (m, 2H), 7.20-7.18 (m, 3H), 4.69 (s, 2H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 141.27, 134.55, 131.47, 130.95, 130.30, 129.26, 128.78, 128.60, 128.32, 127.58, 126.95, 126.16, 125.95, 125.25, 125.10, 124.96, 123.82, 39.36. HMRS (ESI) $C_{23}H_{16}$ for [+H] calcd. 293.1330; found 293.1324.

5-Benzyl-1,2,3-trimethoxybenzene (1j)[14]



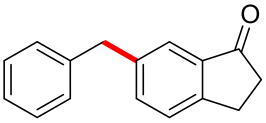
Yield 93%. Yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.32-7.30 (m, 2H), 7.2-7.21 (m, 3H), 6.42 (s, 2H), 3.94 (s, 2H), 3.84 (s, 3H), 3.82 (s, 6H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 153.23, 140.92, 136.75, 136.36, 128.82, 128.51, 126.21, 105.99, 60.87, 56.07, 42.23. HMRS (ESI) $C_{16}H_{18}O_3$ for [+H] calcd. 259.1344; found 259.1330.

2-Benzyl-9H-fluorene (1k)[15]



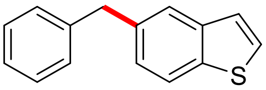
Yield 96%. White solid. m.p. 104-105 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.5 Hz, 1H), 7.67 (d, *J* = 7.7 Hz, 1H), 7.48 (d, *J* = 7.4 Hz, 1H), 7.33-7.32 (m. 2H), 7.29-7.27 (m, 2H), 7.26-7.23 (m, 1H), 7.22-7.18 (m, 4H), 4.03 (s, 2H), 3.81 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 143.74, 143.27, 141.69, 141.49, 139.90, 129.02, 128.57, 127.70, 126.76, 126.46, 126.15, 125.69, 125.04, 119.86, 119.75, 42.16, 36.90. HMRS (ESI) $C_{20}H_{16}$ for [+H] calcd. 257.1330; found 293.1325.

6-Benzyl-2,3-dihydro-1H-inden-1-one (1l)



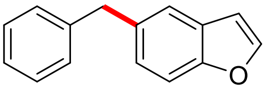
Yield 60%. Brick-red solid. m.p. 93-94 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.9 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.28 (s, 1H), 7.25-7.19 (m, 4H), 4.06 (s, 2H), 3.09-3.07 (t, 2H), 2.68-2.66 (t, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 206.61, 155.85, 148.73, 140.02, 135.42, 128.99, 128.70, 128.47, 126.91, 126.50, 123.81, 42.30, 36.43, 25.74. HMRS (ESI) $C_{16}H_{14}O$ for [+H] calcd. 223.1123; found 223.1118.

5-Benzylbenzothiophene (1m)[16]



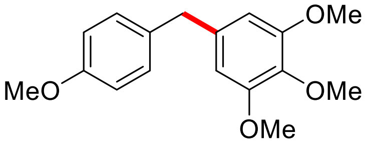
Yield 93%. Yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 1H), 7.61 (s, 1H), 7.38 (d, *J* = 5.4 Hz, 1H), 7.28-7.26 (m, 2H), 7.23 (d, *J* = 5.4 Hz, 1H), 7.21-7.16 (m, 4H), 4.08 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.37, 140.05, 137.68, 137.35, 129.02, 128.56, 126.66, 126.18, 125.83, 123.76, 123.67, 122.47, 41.90.

5-Benzylbenzofuran (1n)[17]



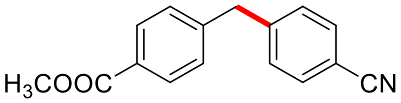
Yield 92%. Yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 (d, *J*=2.0, 1H), 7.41-7.38 (m, 2H), 7.2-7.26 (m, 2H), 7.21-7.17 (m, 3H), 7.12-7.11 (m, 1H), 6.67 (d, *J* = 1.2 Hz, 1H), 4.06 (s, 2H). ^{13}C NMR (151 MHz, CDCl3) δ 153.75, 145.22, 141.70, 135.69, 128.94, 128.51, 127.66, 126.09, 125.49, 121.17, 111.24, 106.52, 41.84. HMRS (ESI) $C_{15}H_{12}O$ for [+H] calcd. 209.0966; found 209.0963.

1, 2, 3-Trimethoxy-5-(4-methoxybenzyl) benzene (2a)[18]



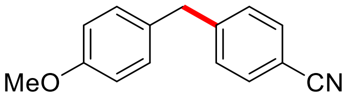
Yield 87%. Yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.13-7.11 (m, 2H), 6.86-6.84 (m, 2H), 6.39 (s, 2H), 3.87 (s, 2H), 3.82 (s, 3H), 3.81 (s, 6H), 3.79 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 158.10, 153.25, 144.98, 137.34, 136.26, 133.06, 129.84, 113.96, 105.82, 60.94, 56.12, 55.33, 41.39. HMRS (ESI) $C_{17}H_{20}O_4$ for [+H] calcd. 289.1440; found 289.1433.

Methyl 4-(4-cyanobenzyl) benzoate (3a)[19]



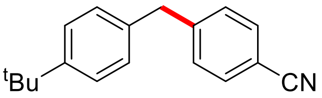
Yield 95%. White solid. m.p. 128-129 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.58 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.3 Hz, 2H), 4.08 (s, 2H), 3.90 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.84, 145.68, 144.58, 132.45, 130.10, 129.69, 129.00, 128.72, 118.83, 110.44, 52.12, 41.88. HMRS (ESI) $C_{16}H_{13}NO_2$ for [+H] calcd. 252.1042; found 252.1020.

4-(4-Methoxybenzyl) benzonitrile (4a)[19]



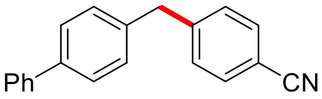
Yield 94.5%. Light yellow solid. m. p. 48-49 °C. NMR (600 MHz) ^{1}H δ 7.57-7.55 (m, 2H), 7.28- 7.26 (m, 2H), 7.09-7.07 (m, 2H), 6.86-6.84 (m, 2H), 3.97 (s, 2H), 3.79 (s, 3H). NMR (151 MHz) ^{13}C δ 158.43, 147.34, 132.37, 131.48, 130.04, 129.61, 119.15, 114.23, 109.98, 55.36, 41.17. HMRS (ESI) $C_{15}H_{13}NO$ for [+H] calcd. 224.1075; found 224.1072.

4-(4-(Tert-butyl) benzyl) benzonitrile (5a)[15]



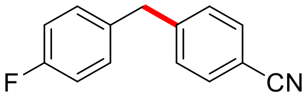
Yield 86%. Yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.2 Hz, 2H), 7.34 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 7.10 (d, *J* = 8.2 Hz, 2H), 4.01 (s, 2H), 1.32 (s, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 149.59, 146.98, 136.30, 132.29, 129.69, 128.60, 125.68, 119.07, 109.97, 41.50, 34.45, 31.38. HMRS (ESI) $C_{18}H_{19}N$ for [+H] calcd. 250.1595; found 250.1590.

4-([1,1'-Biphenyl]-4-ylmethyl) benzonitrile (6a)[20]



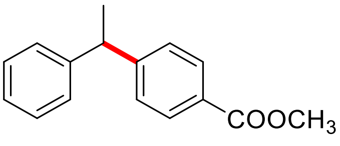
Yield 96%. White solid. m.p. 126-127 °C. ^{1}H NMR (600 MHz, CDCl3) δ 7.60-7.56 (m, 4H), δ 7.54 (d, *J* = 8.1 Hz, 2H), 7.43 (t, *J* = 7.7 Hz, 1H), 7.35-7.32 (m, 3H), 7.23 (d, *J* = 8.1 Hz, 2H), 4.07 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 146.64, 140.69, 139.69, 138.40, 132.38, 129.69, 129.38, 128.80, 127.50, 127.32, 127.02, 118.99, 110.15, 41.63. HMRS (ESI) $C_{20}H_{15}N$ for [+H] calcd. 270.1282; found 270.1277.

4-(4-Fluorobenzyl) benzonitrile (7a)[20]



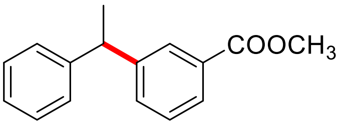
Yield 95%. Yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.9 Hz, 2H), 7.26 (d, *J* = 7.8 Hz, 2H), 7.12-7.10 (m, 2H), 7.00-6.97 (m, 2H), 4.00 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.52, 160.89, 146.53, 135.04, 132.38, 130.46, 130.41, 129.57, 118.93, 115.68, 115.54, 110.22, 41.10. HMRS (ESI) $C_{14}H_{10}FN$ for [+H] calcd. 212.0875; found 212.0871.

Methyl 4-(1-phenylethyl) benzoate (8a)[6]



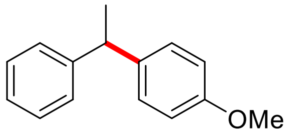
Yield 86%. Light yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.0 Hz, 2H), 7.31-7.30 (m, 2H), 7.22-7.20 (m, 2H), 4.22 (q, *J* = 7.2 Hz, 1H), 3.90 (s, 3H), 1.67 (d, *J* = 7.3 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.07, 151.75, 145.46, 129.78, 128.54, 128.05, 127.70, 127.62, 126.36, 52.00, 44.85, 21.61. HMRS (ESI) $C_{16}H_{16}O_2$ for [+H] calcd. 241.1228; found 241.1224.

Methyl 3-(1-phenylethyl) benzoate (8b)[21]



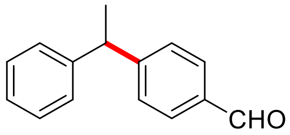
Yield 65%. Yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (s, 1H), δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.41-7.40 (m, 1H), δ 7.35 (t, *J* = 7.6 Hz, 1H), 7.30 (t, *J* = 7.4 Hz, 2H), 7.23-7.19 (m, 3H), 4.22 (q, *J* = 7.1 Hz, 1H), 3.91 (s, 3H), 1.68 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.25, 146.74, 145.76, 132.39, 130.26, 128.66, 128.51, 128.47, 127.58, 127.42, 126.26, 52.09, 44.67, 21.76. HMRS (ESI) $C_{16}H_{16}O_2$ for [+H] calcd. 241.1228; found 241.1224.

1-Methoxy-4-(1-phenylethyl) benzene (8d)[22]



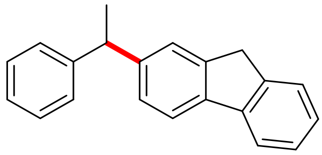
Yield 82%. Yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31-7.28 (m, 2H), 7.23-7.18 (m, 3H), 7.17-7.15 (m, 2H), 6.86-6.84 (m, 2H), δ 4.13 (q, *J* = 7.2 Hz, 1H), 3.79 (s, 3H), 1.64 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 157.86, 146.80, 138.58, 128.54, 128.36, 127.55, 125.96, 113.75, 55.25, 43.96, 22.08.

4-(1-Phenylethyl) benzaldehyde (8e)[6]



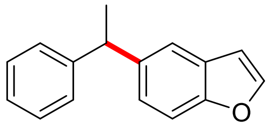
Yield 62%. White solid. m.p. 125-126 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.97 (s, 1H), δ 7.81 (d, *J* = 8.2 Hz, 2H), 7.39 (d, *J* = 8.1 Hz, 2H), 7.33-7.30 (m, 2H), 7.23-7.21 (m, 2H), 4.24 (q, *J* = 7.2 Hz, 1H), 1.68 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.96, 153.65, 145.09, 134.65, 130.00, 128.61, 128.33, 127.62, 126.50, 45.04, 21.54. HMRS (ESI) $C_{15}H_{14}O$ for [+H] calcd. 211.1123; found 211.1119.

2-(1-Phenylethyl)-9H-fluorene (8f)



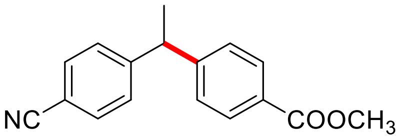
Yield 46%. Pale yellow solid. m.p. 104-105 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.6 Hz, 1H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 7.4 Hz, 1H), 7.38 (s, 1H), 7.33 (t, *J* = 7.4 Hz, 1H), 7.30-7.22 (m, 6H), 7.19-7.17 (m, 1H), 4.22 (q, *J* = 7.2 Hz, 1H), 3.83 (s, 2H), 1.68 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 146.62, 145.26, 143.56, 143.29, 141.66, 139.80, 128.43, 127.67, 126.70, 126.38, 126.05, 124.98, 124.30, 119.72, 44.93, 36.92, 22.10. HMRS (ESI) $C_{21}H_{18}$ for [+H] calcd. 271.1487; found 271.1485.

5-(1-Phenylethyl) benzofuran (8g)



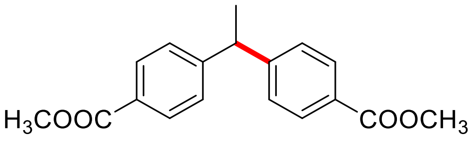
Yield 60%. Yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 (d, *J* = 2.0 Hz, 1H), 7.44 (1H), 7.40-7.38 (m, 1H), 7.29-7.23 (m, 4H), 7.18-7.13 (m, 2H), 6.69 (d, *J* = 1.2 Hz, 1H), 4.25 (q, *J* = 7.2 Hz, 1H), 4.25 (q, *J* = 7.2 Hz, 1H), 1.68 (d, *J* = 7.2 Hz, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 153.61, 146.80, 145.19, 141.11, 128.39, 127.69, 127.46, 126.01, 124.45, 119.63, 111.13, 106.62, 44.67, 22.36.

Methyl 4-(1-(4-cyanophenyl) ethyl) benzoate (9a)



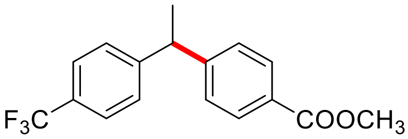
Yield 76%. Light yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.58 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 7.25 (d, *J* = 8.2 Hz, 2H), 3.90 (s, 3H), 4.25 (q, *J* = 7.1 Hz, 1H), 1.66 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.81, 150.92, 149.88, 132.40, 130.02, 128.64, 128.43, 127.64, 118.85, 110.32, 52.10, 44.89, 21.22. HMRS (ESI) $C_{17}H_{15}NO_2$ for [+H] calcd. 266.1181; found 266.1176.

Dimethyl 4,4'-(ethane-1,1-diyl) dibenzoate (10a)



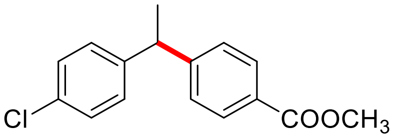
Yield 68%. Light yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.96 (d, *J* = 8.4 Hz, 4H), 7.27 –7.25 (m, 4H), 4.24 (q, *J* = 7.2 Hz, 1H), 1.66 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 166.93, 150.71, 129.89, 128.35, 127.66, 52.04, 44.84, 21.36.

Methyl 4-(1-(4-(trifluoromethyl) phenyl) ethyl) benzoate (11a)



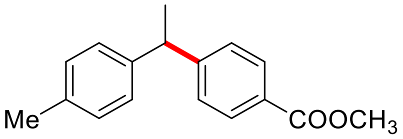
Yield 54%. Light yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.97 (d, *J* = 8.2 Hz, 2H), 7.54 (d, *J* = 8.1 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 8.2 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 1H), 3.90 (s, 3H), 1.67 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 166.91, 150.49, 149.49, 129.93, 128.45, 127.95, 127.64, 125.50, 125.47, 52.06, 44.68, 21.40. HMRS (ESI) $C_{17}H_{15}F_3O_2$ for [+H] calcd. 309.1102; found.

Methyl 4-(1-(4-chlorophenyl) ethyl) benzoate (12a)[6]



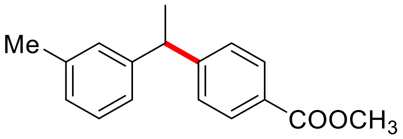
Yield 57%. Light yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 4H), 7.12 (d, *J* = 8.4 Hz, 2H), 4.17 (q, *J* = 7.2 Hz, 1H), 3.89 (s, 3h), 1.62 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 166.96, 151.10, 143.94, 132.12, 129.86, 128.97, 128.63, 128.26, 127.59, 52.04, 44.22, 21.55. HMRS (ESI) $C_{16}H_{15}ClO_2$ for [+H] calcd. 275.0839; found 275.0829.

Methyl 4-(1-(p-tolyl) ethyl) benzoate (13a)



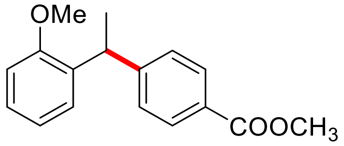
Yield 72%. Colorless oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.2 Hz, 2H), 7.12-7.09 (m, 4H), 4.17 (q, *J* = 7.2 Hz, 1H), 3.90 (s, 3H), 2.32 (s, 3H), 1.64 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 167.10, 152.01, 142.49, 135.88, 129.76, 129.21, 127.95, 127.63, 127.47, 51.99, 44.45, 21.67, 20.99. HMRS (ESI) $C_{17}H_{18}O_2$ for [+H] calcd. 255.1385; found. 255.1378.

Methyl 4-(1-(m-tolyl) ethyl) benzoate (14a)[6]



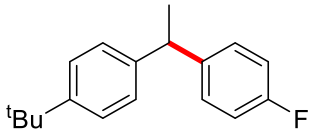
Yield 60%. Colorless oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 7.20-7.17 (m, 1H), 7.03-7.01 (m, 3H), 4.17 (q, *J* = 7.2 Hz, 1H), 3.90 (s, 3H), 2.32 (s, 3H), 1.65 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 167.11, 151.86, 145.41, 138.09, 129.75, 128.41, 127.97, 127.67, 127.10, 124.60, 51.99, 44.79, 21.61, 21.49. HMRS (ESI) $C_{17}H_{18}O_2$ for [+H] calcd. 255.1385; found. 255.1378.

Methyl 4-(1-(2-methoxyphenyl) ethyl) benzoate (15a)



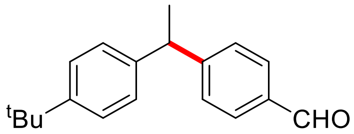
Yield 40%. Colorless oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.92 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.2 Hz, 2H), 7.22-7.16 (m, 2H), 6.93 (t, *J* = 7.4 Hz, 1H), 6.84 (d, *J* = 8.1 Hz, 1H), 4.59 (q, *J* = 7.2 Hz, 1H), 3.89 (s, 3H), 3.74 (s, 3H), 1.59 (d, *J* = 7.3 Hz, 3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 167.23, 156.84, 152.12, 133.88, 129.49, 127.69, 127.54, 127.44, 120.57, 110.65, 55.37, 51.94, 37.76, 20.60. HMRS (ESI) $C_{17}H_{18}O_3$ for [+H] calcd. 271.1334; found. 271.1332

1-(*Tert*-butyl)-4-(1-(4-fluorophenyl) ethyl) benzene (16a)



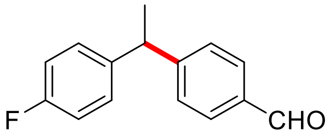
Yield 68%. Yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.32-7.31 (m, 2H), 7.20-7.18 (m, 2H), 7.14-7.13 (m, 2H), 6.99-6.96 (m, 2H), δ 4.12 (q, *J* = 7.2 Hz, 1H), 1.62 (d, *J* = 7.3 Hz, 3H), 1.31 (s, 9H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 160.45, 148.90, 143.06, 129.02, 128.97, 127.09, 125.31, 115.10, 114.97, 43.60, 34.37, 31.40, 22.08.

4-(1-(4-(*Tert*-butyl) phenyl) ethyl) benzaldehyde (16b)



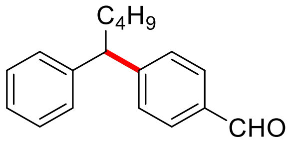
Yield 51%. Yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 9.98, 7.82-7.81 (m, 2H), 7.42-7.40 (m, 2H), 7.34-7.33 (m, 2H), 7.16-7.15 (m, 2H), 4.22 (q, *J* = 7.2 Hz, 1H), 1.68 (d, *J* = 7.2 Hz, 3H), 1.32 (s, 9H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 191.99, 153.94, 149.29, 141.97, 134.61, 130.00, 128.35, 127.20, 125.48, 44.61, 34.41, 31.39, 21.58. HMRS (ESI) $C_{19}H_{22}O$ for [+H] calcd. 267.1749; found 267.1742.

4-(1-(4-Fluorophenyl) ethyl) benzaldehyde (17a)



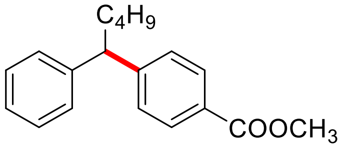
Yield 70%. Yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 9.97 (s, 1H), δ 7.81 (d, *J* = 7.9 Hz, 2H), 7.36 (d, *J* = 7.8 Hz, 2H), 7.18-7.15 (m, 2H), 7.00-6.97 (m, 2H), 4.21 (q, *J* = 7.1 Hz, 1H), 1.69 – 1.61 (m, 3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 191.89, 160.67, 153.37, 140.77, 134.74, 130.05, 129.00, 128.22, 115.30, 44.28, 21.69. HMRS (ESI) $C_{15}H_{13}FO$ for [+H] calcd. 229.1028; found 229.1022.

4-(1-Phenylpentyl) benzaldehyde (18a)



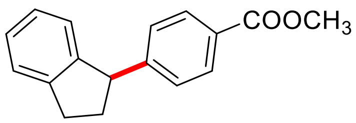
Yield 54%. Yellow oil. ^{1}H NMR (600 MHz, CDCl$_3$) δ 9.96 (s, 1H), δ 7.80 (d, *J* = 8.2 Hz, 2H), 7.42 (d, *J* = 8.1 Hz, 2H), 7.32-7.29 (m, 2H), 7.25-7.19 (m, 3H), 3.98 (t, *J* = 7.8 Hz, 1H), 2.11-2.06 (m, 2H), 1.38-1.35 (m, 2H), 1.29-1.24 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 191.95, 152.72, 144.08, 134.65, 130.02, 128.62, 128.57, 127.87, 126.48, 51.60, 35.14, 30.15, 22.67, 13.98. HMRS (ESI) $C_{18}H_{20}O$ for [+H] calcd. 253.1592; found 253.1586.

Methyl 4-(1-phenylpentyl) benzoate (18b)



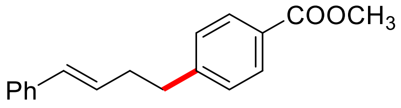
Yield 52%. Yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.33-7.31 (m, 2H), 7.29-7.28 (m, 1H), 7.24-7.23 (m, 2H), 7.20-7.18 (m, 1H), 3.95 (t, *J* = 7.8 Hz, 1H), 3.89 (s, 3H), 2.09-2.05 (m, 2H), 1.37-1.34 (m, 2H), 1.26-1.23 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.08, 150.80, 144.44, 129.79, 128.54, 128.02, 127.94, 127.86, 126.33, 51.98, 51.40, 35.20, 30.16, 22.67, 13.98. HMRS (ESI) $C_{19}H_{22}O_2$ for [+H] calcd. 283.1698; found 283.1690.

Methyl 4-(2,3-dihydro-1H-inden-1-yl) benzoate (19a)[6]



Yield 78%. Colorless solid. m.p. 66-67 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 8.1 Hz, 2H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.13 (t, *J* = 7.4 Hz, 1H), 6.92 (d, *J* = 7.5 Hz, 1H), 4.39 (t, *J* = 8.3 Hz, 1H), 3.90 (s, 3H), 3.08-3.04 (m, 1H), 3.00-2.94 (m, 1H), 2.62, -2.57 (m, 1H), 2.08-2.02 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.12, 150.98, 146.06, 144.35, 129.88, 128.32, 128.15, 126.86, 126.53, 124.87, 124.51, 52.03, 51.64, 36.39, 31.87. HMRS (ESI) $C_{17}H_{16}O_2$ for [+H] calcd. 252.1228; found 253.1222.

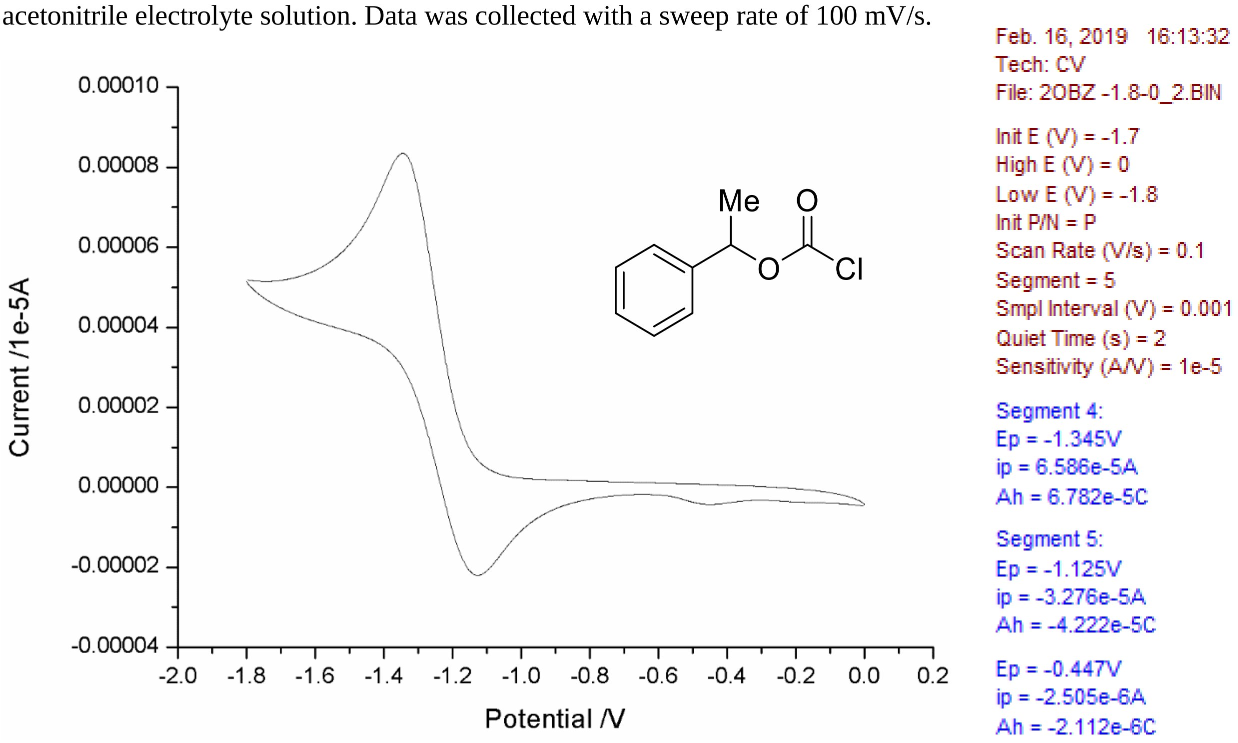
Methyl 4-(4-phenylbut-3-en-1-yl) benzoate (21)



Yield 70%. Pale yellow solid. m.p. 57-58 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.1 Hz, 2H) 7.33-7.29 (m, 6H), 7.22-7.20 (m, 1H), 6.41 (d, *J* = 15.8 Hz, 0H), 6.25-6.20 (m, 1H), 3.91 (s, 3H), 2.85 (t, *J* = 7.7 Hz, 1H), 2.56 (dd, *J* = 14.7, 7.3 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.16, 147.23, 137.53, 130.82, 129.75, 129.28, 128.56, 128.53, 127.96, 127.09, 126.02, 52.00, 35.90, 34.44. HMRS (ESI) $C_{18}H_{18}O_2$ for [+H] calcd. 267.1385; found. 267.1379.

5. Cyclic Voltammetry Measurements

Cyclic voltammetric studies were conducted according to a literature procedure using a Pine Research Instruments Wavedriver potentiostat,[23] a glassy carbon disk working electrode, a glassy carbon rod counter electrode, and a Ag/Ag+ reference electrode (Vycor tip). Samples were prepared with a substrate concentration of 0.01 M in a 0.1 M of tetrabutylammonium tetrafluoroborate in acetonitrile electrolyte solution. Data was collected with a sweep rate of 100 mV/s.



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Feb. 16, 2019   16:13:32
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Low E (V) = -1.8
Init P/N = P
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Smpl Interval (V) = 0.001
Quiet Time (s) = 2
Sensitivity (A/V) = 1e-5

Segment 4:
Ep = -1.345V
ip = 6.586e-5A
Ah = 6.782e-5C

Segment 5:
Ep = -1.125V
ip = -3.276e-5A
Ah = -4.222e-5C

Ep = -0.447V
ip = -2.505e-6A
Ah = -2.112e-6C
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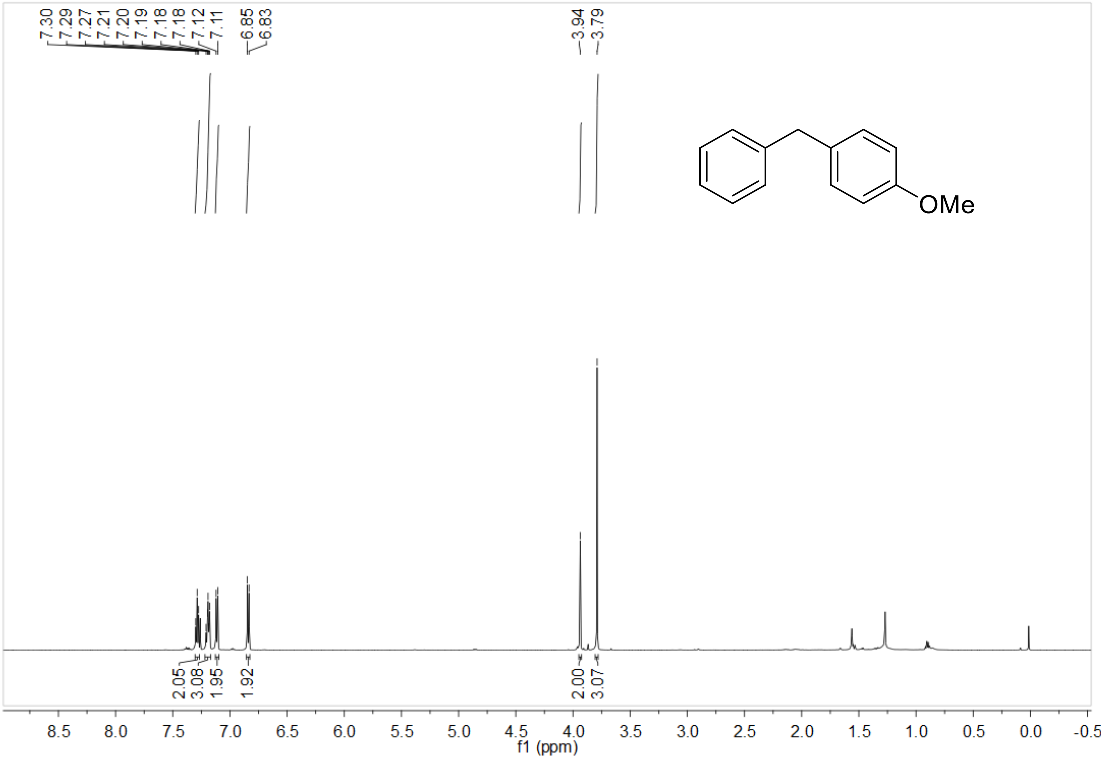
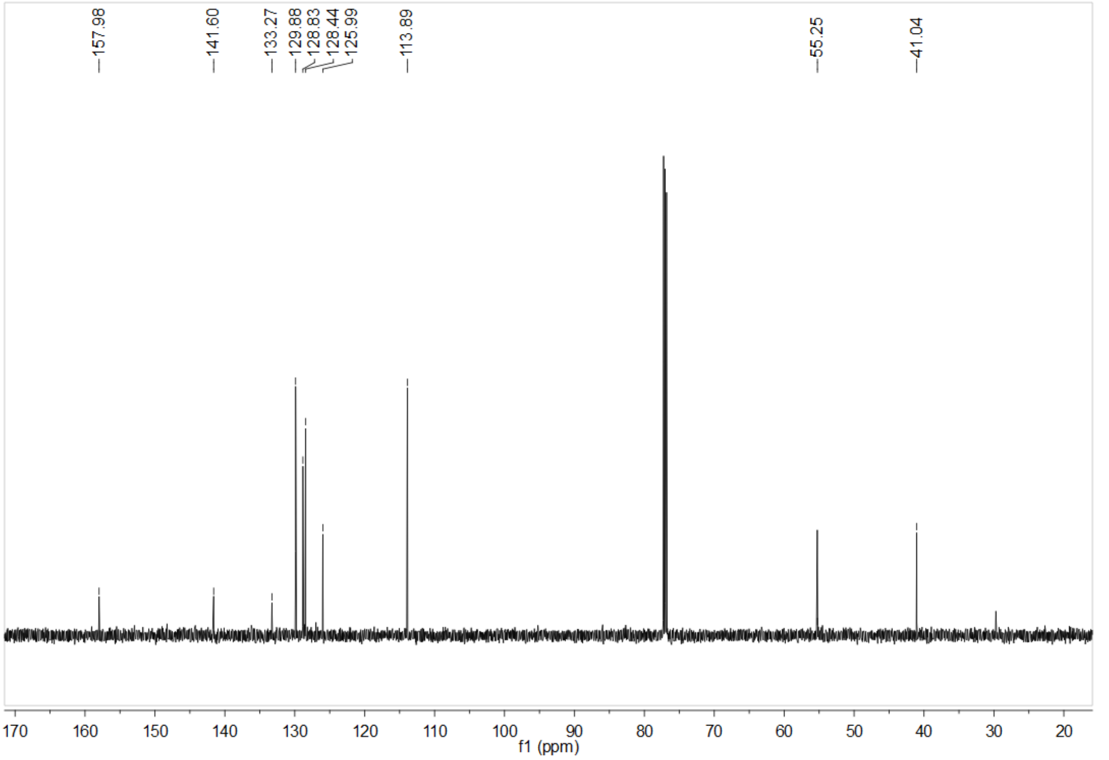
Part II. NMR Data for New Compounds

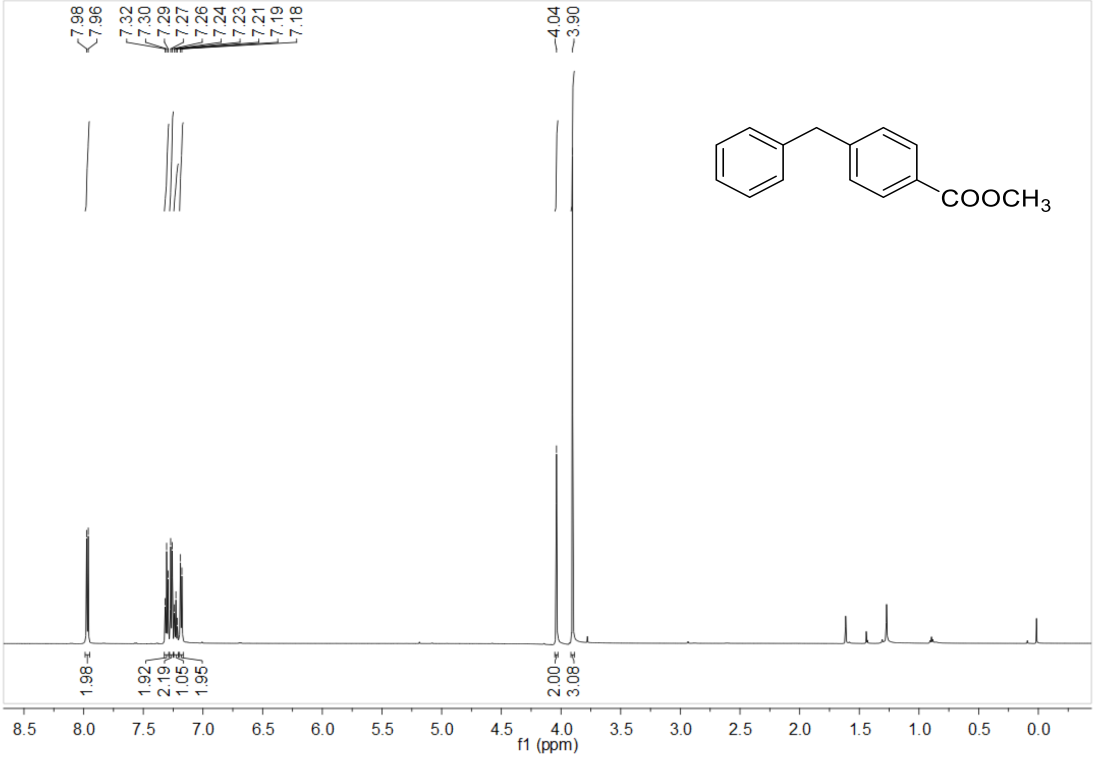
^{1}H NMR Spectrum of Compound 1a



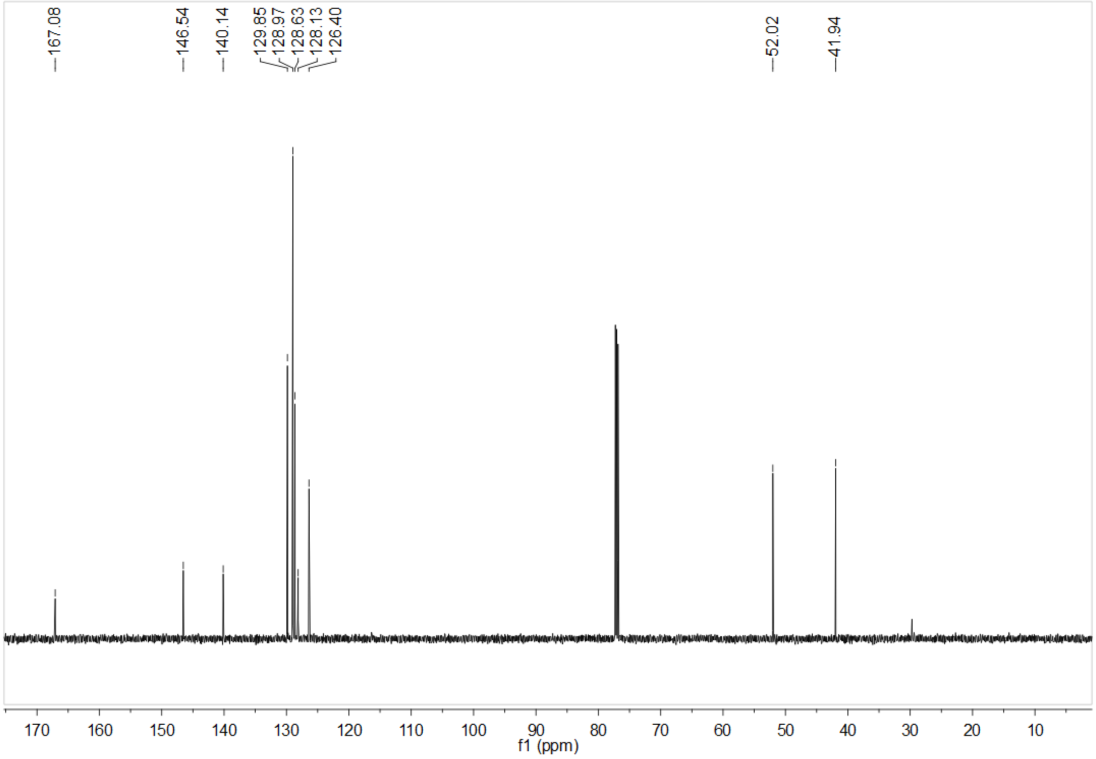
^{13}C NMR Spectrum of Compound 1a



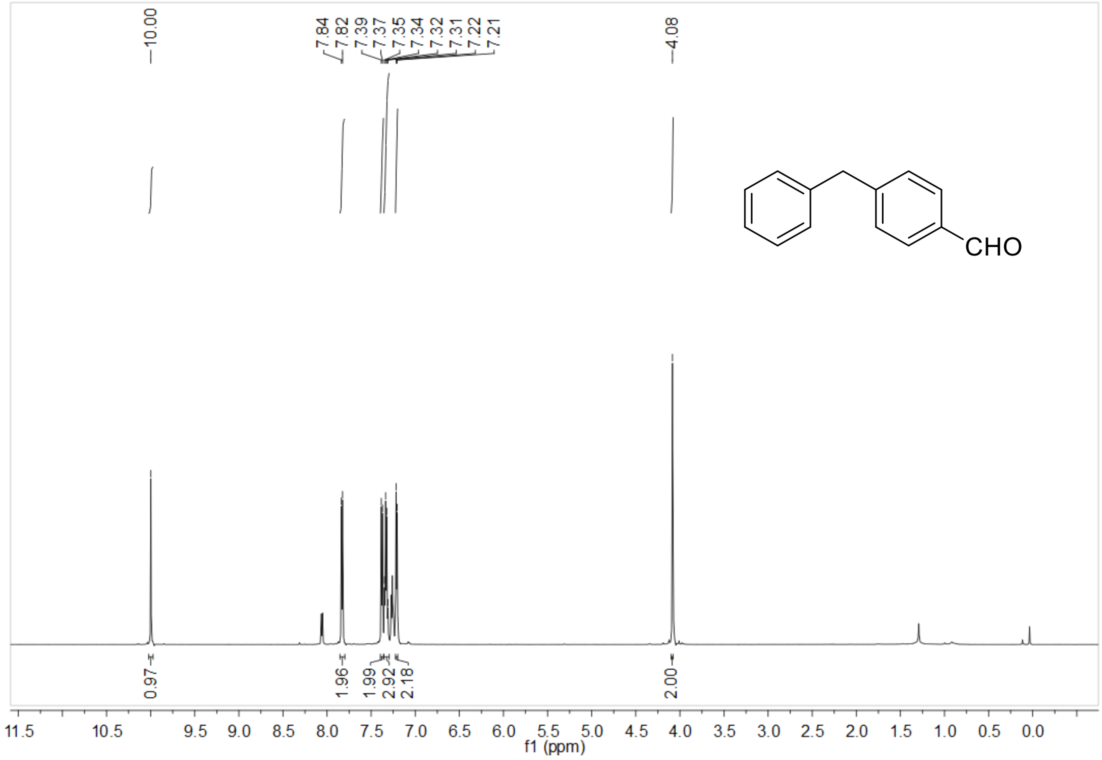
^{1}H NMR Spectrum of Compound 1b



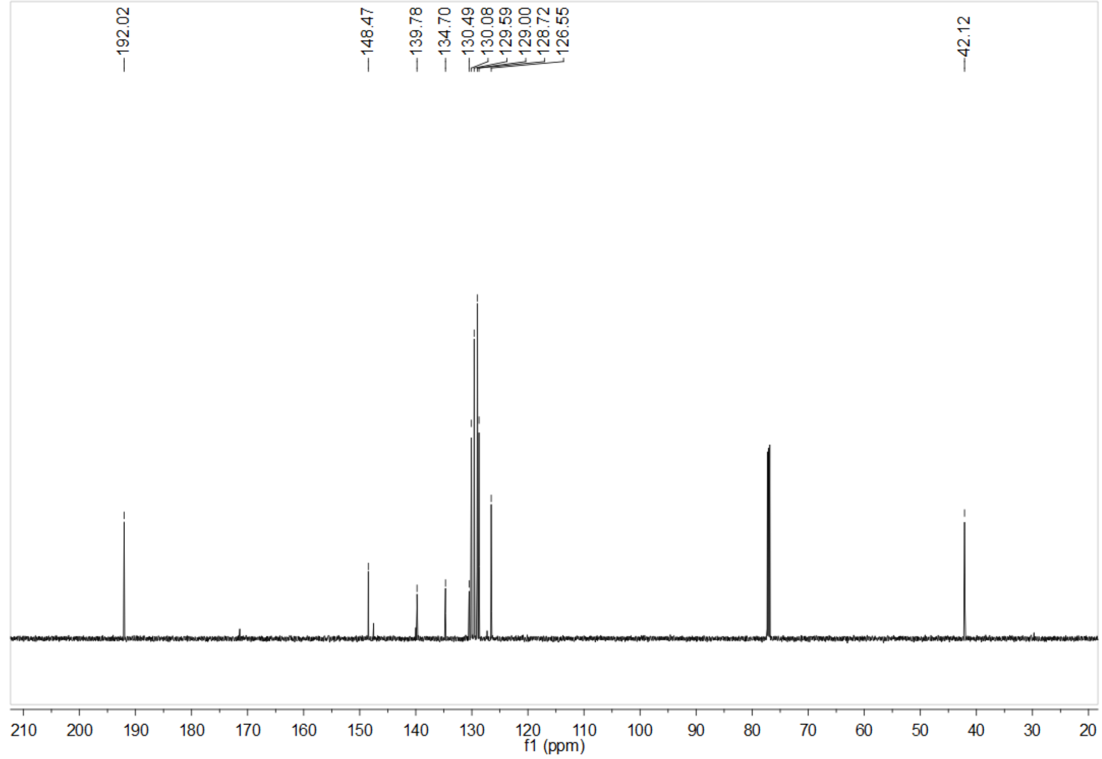
^{13}C NMR Spectrum of Compound 1b



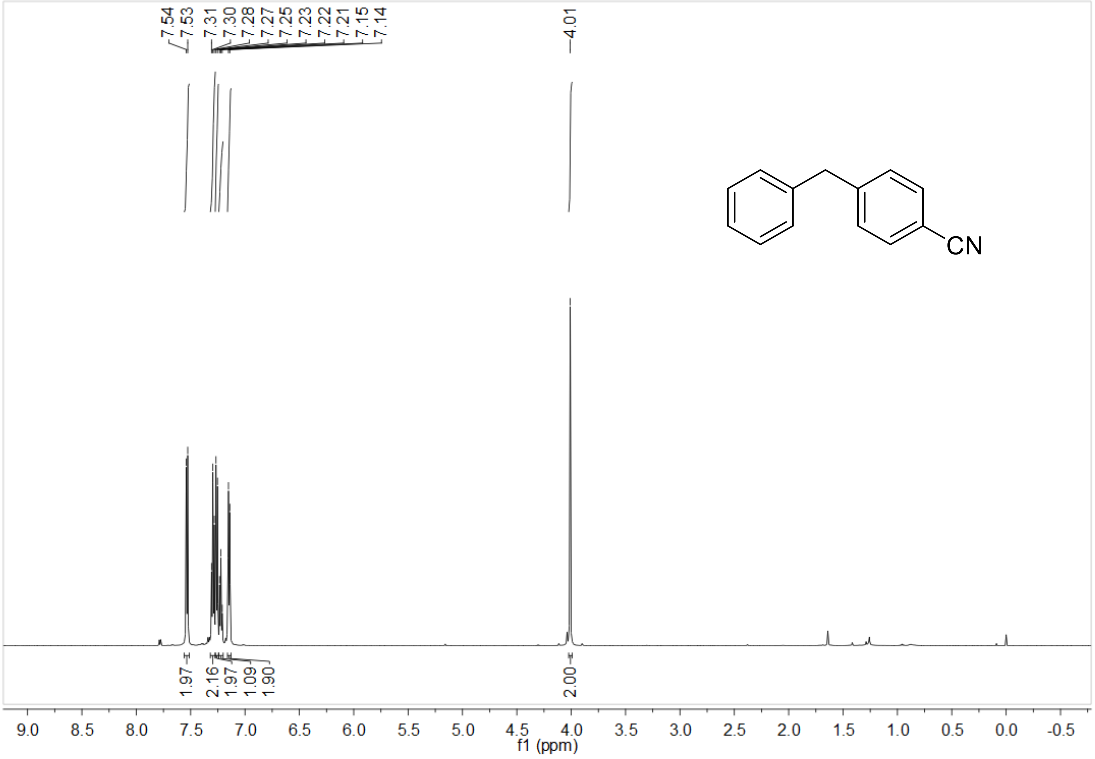
^{1}H NMR Spectrum of Compound 1c



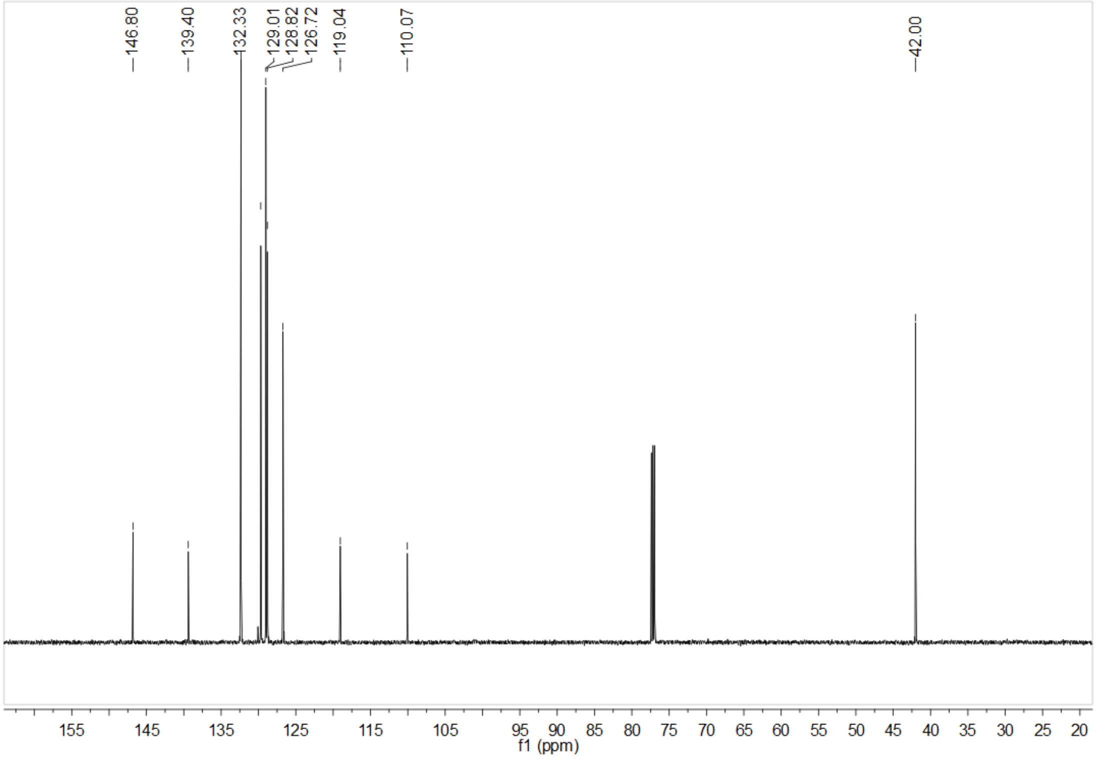
^{13}C NMR Spectrum of Compound 1c



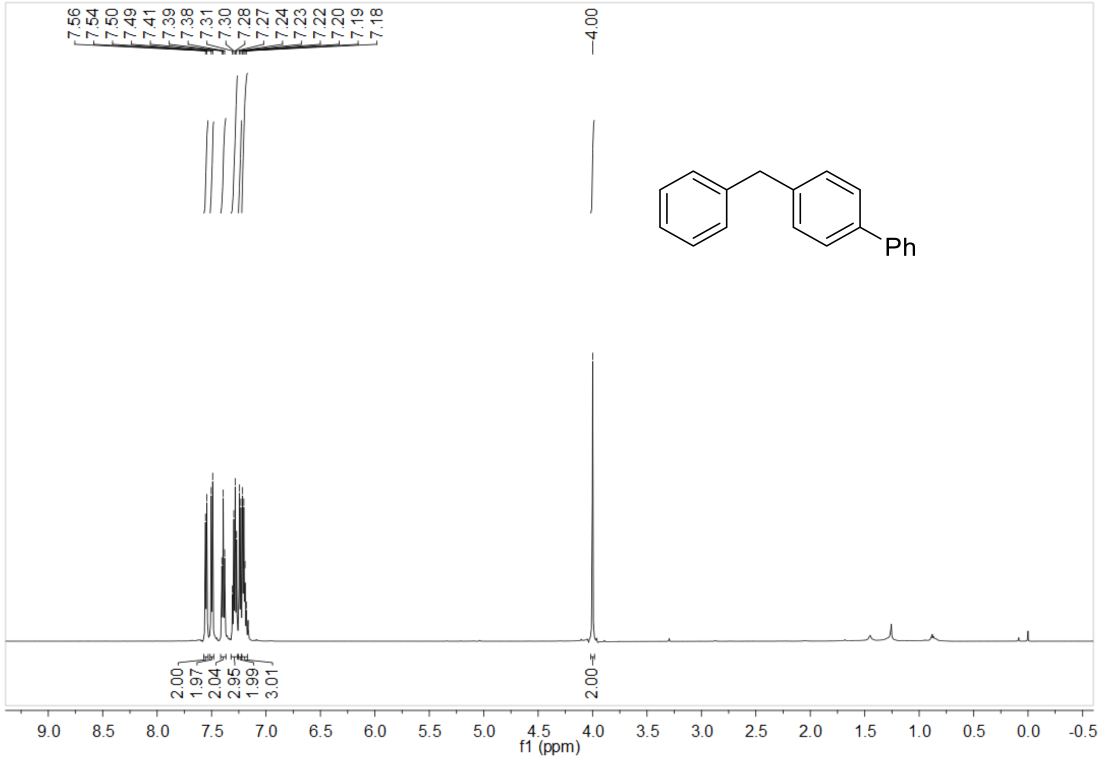
^{1}H NMR Spectrum of Compound 1d



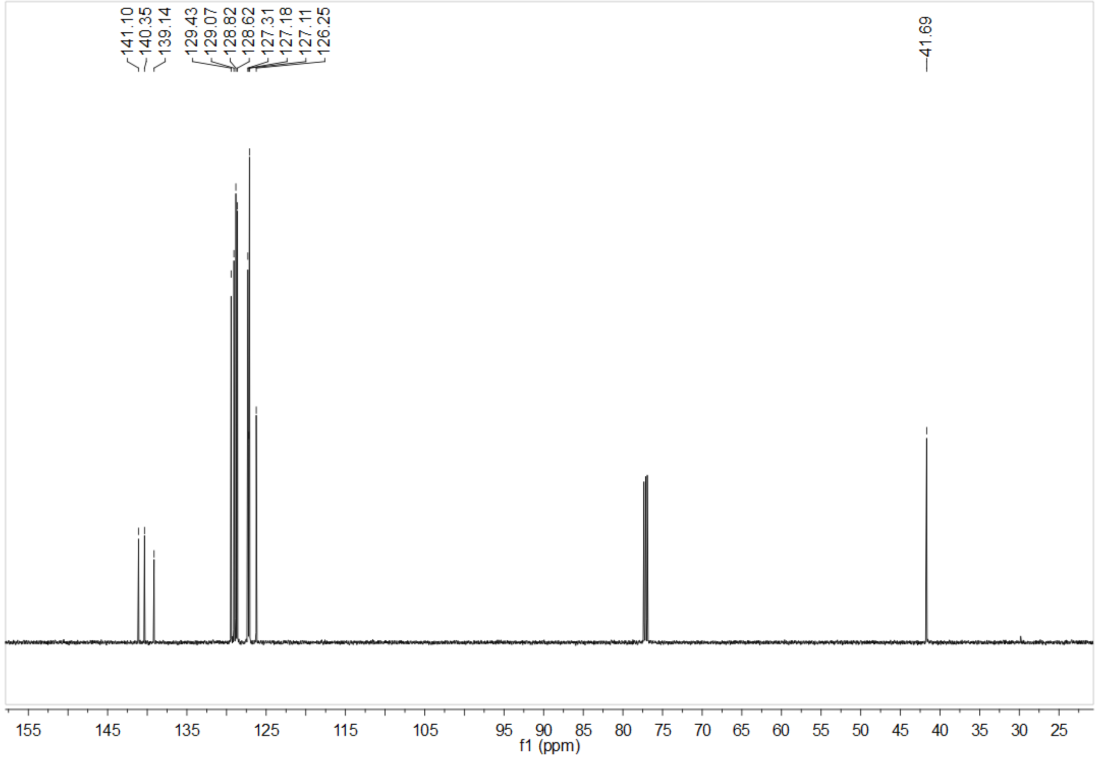
^{13}C NMR Spectrum of Compound 1d



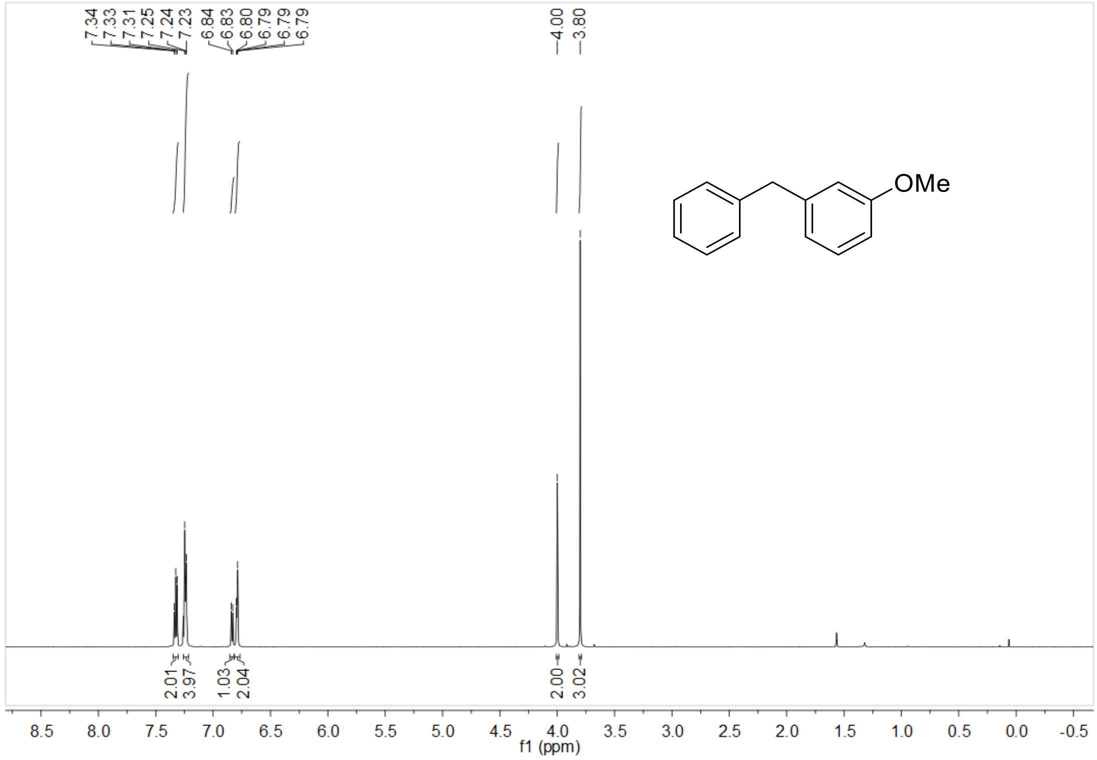
^{1}H NMR Spectrum of Compound 1e



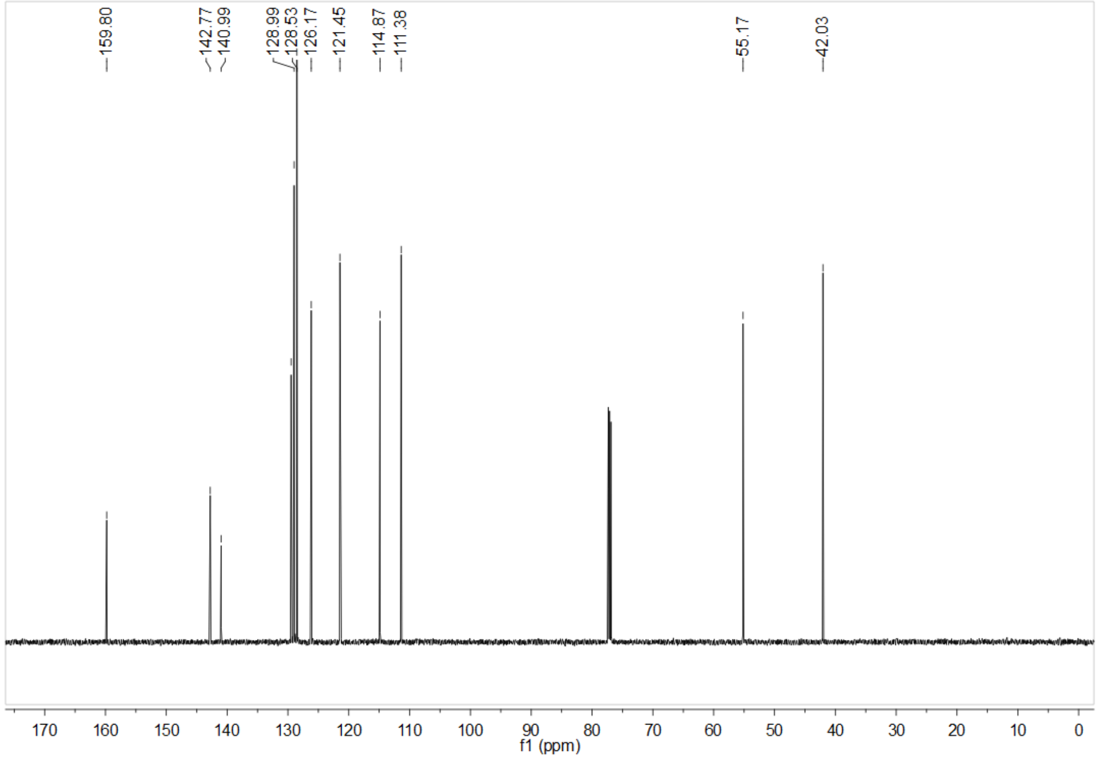
^{13}C NMR Spectrum of Compound 1e



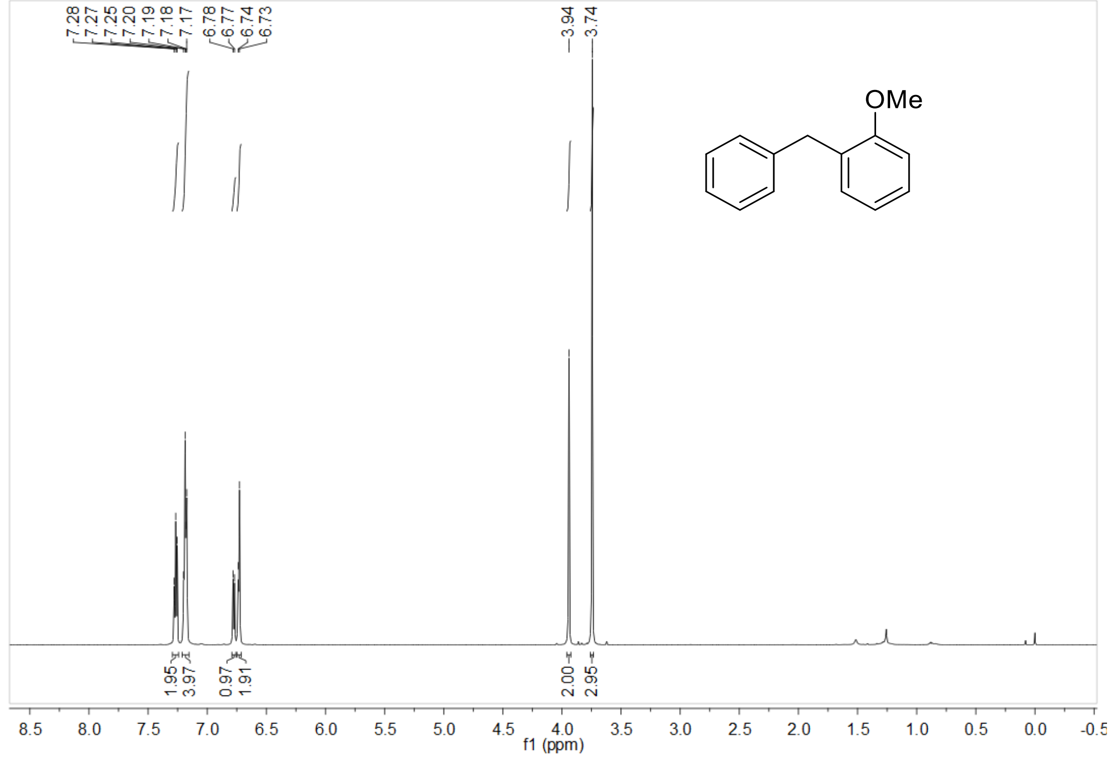
^{1}H NMR Spectrum of Compound 1f



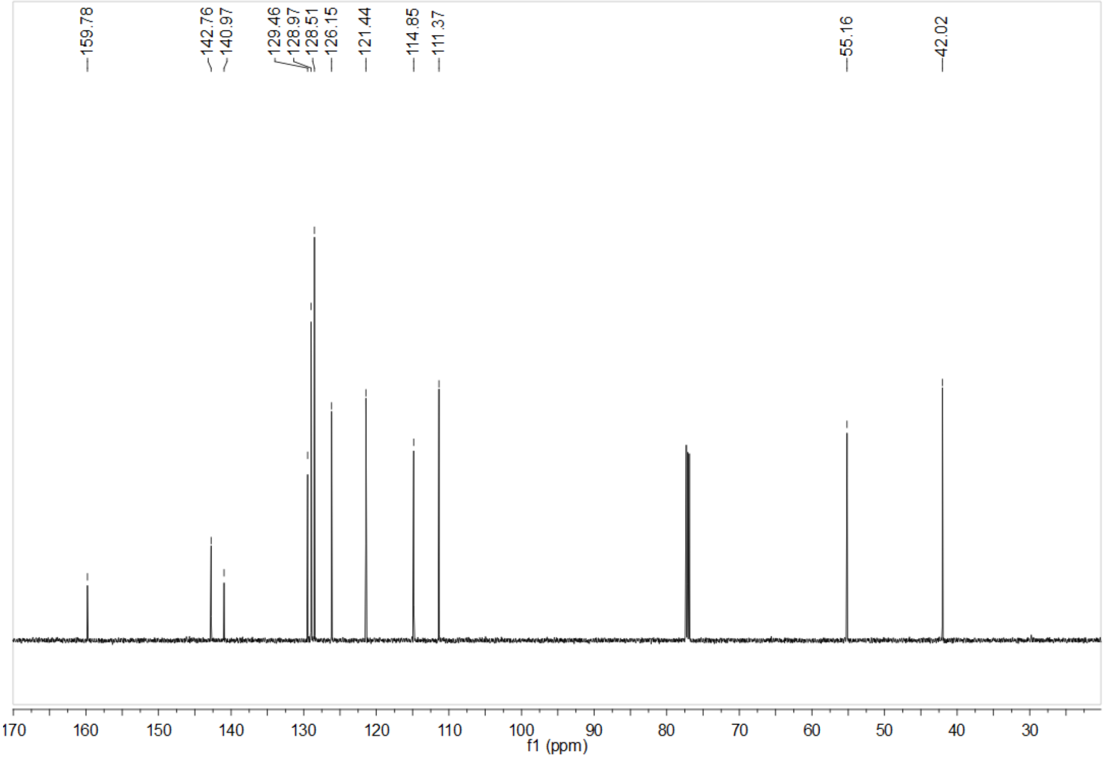
^{13}C NMR Spectrum of Compound 1f



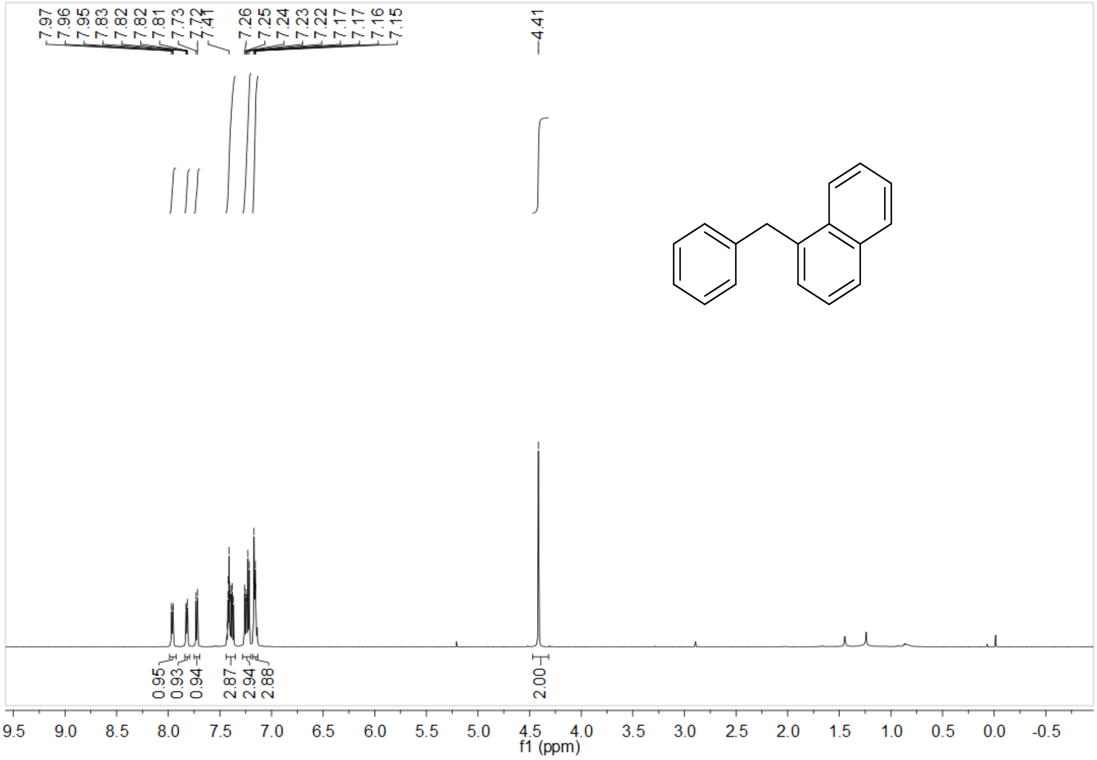
^{1}H NMR Spectrum of Compound 1g



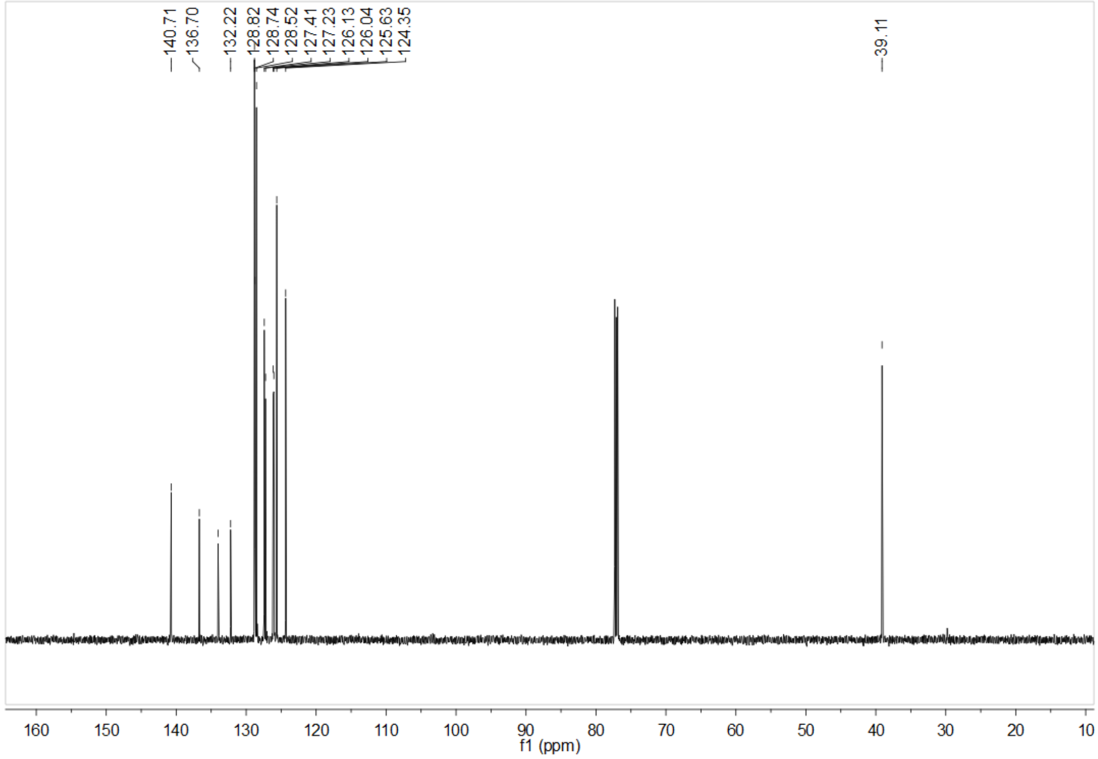
^{13}C NMR Spectrum of Compound 1g



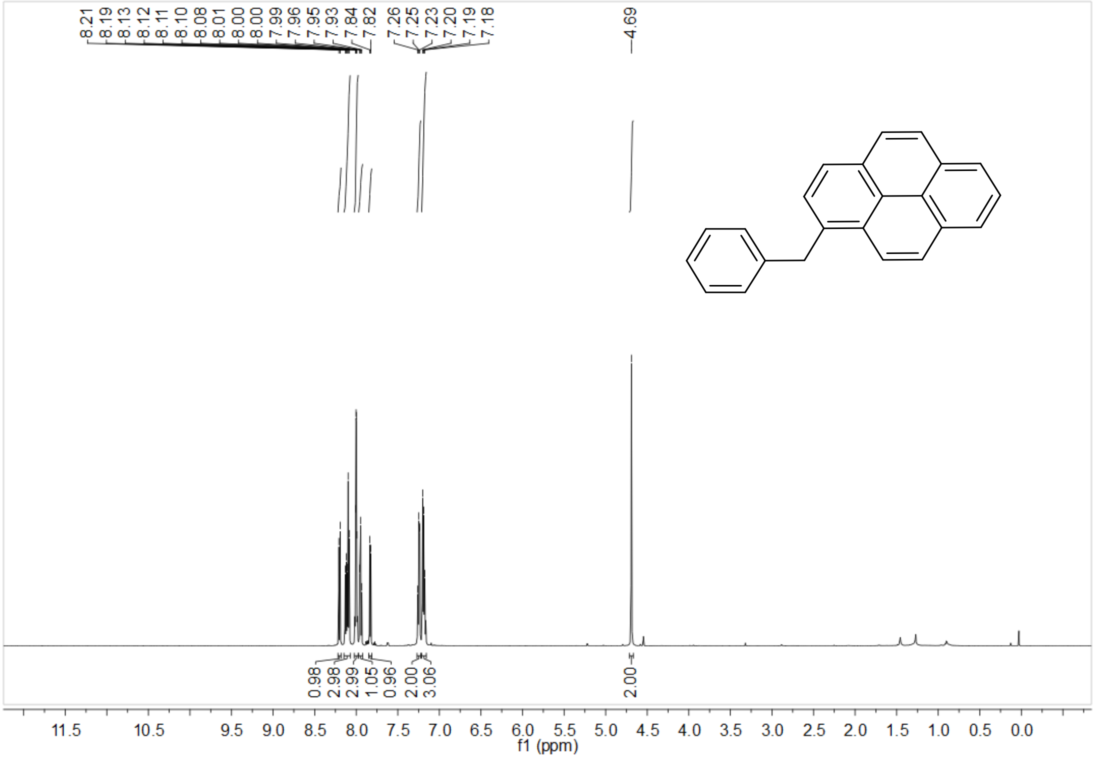
^{1}H NMR Spectrum of Compound 1h



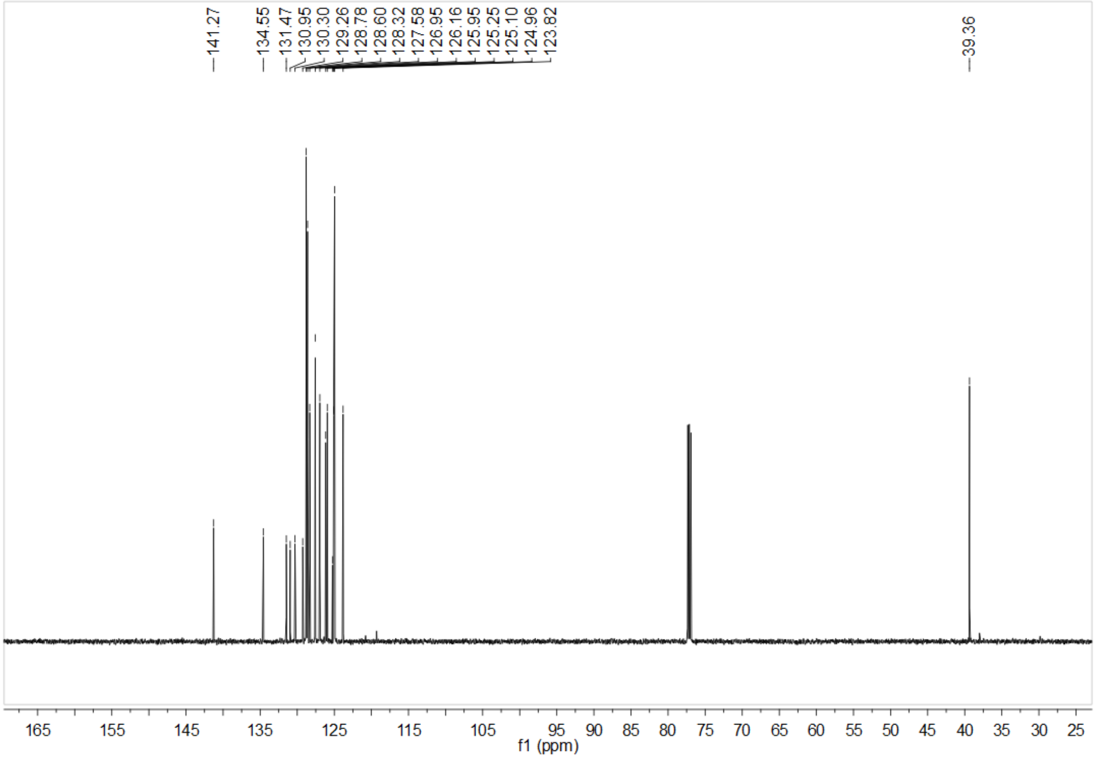
^{13}C NMR Spectrum of Compound 1h



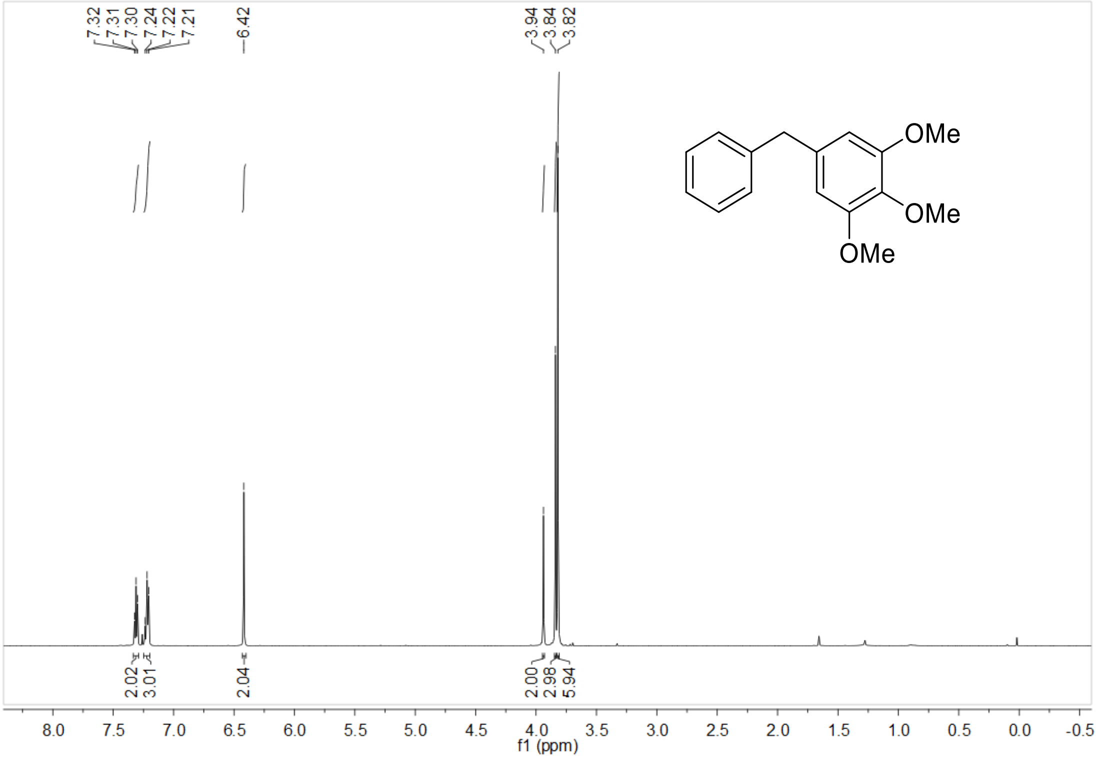
^{1}H NMR Spectrum of Compound 1i



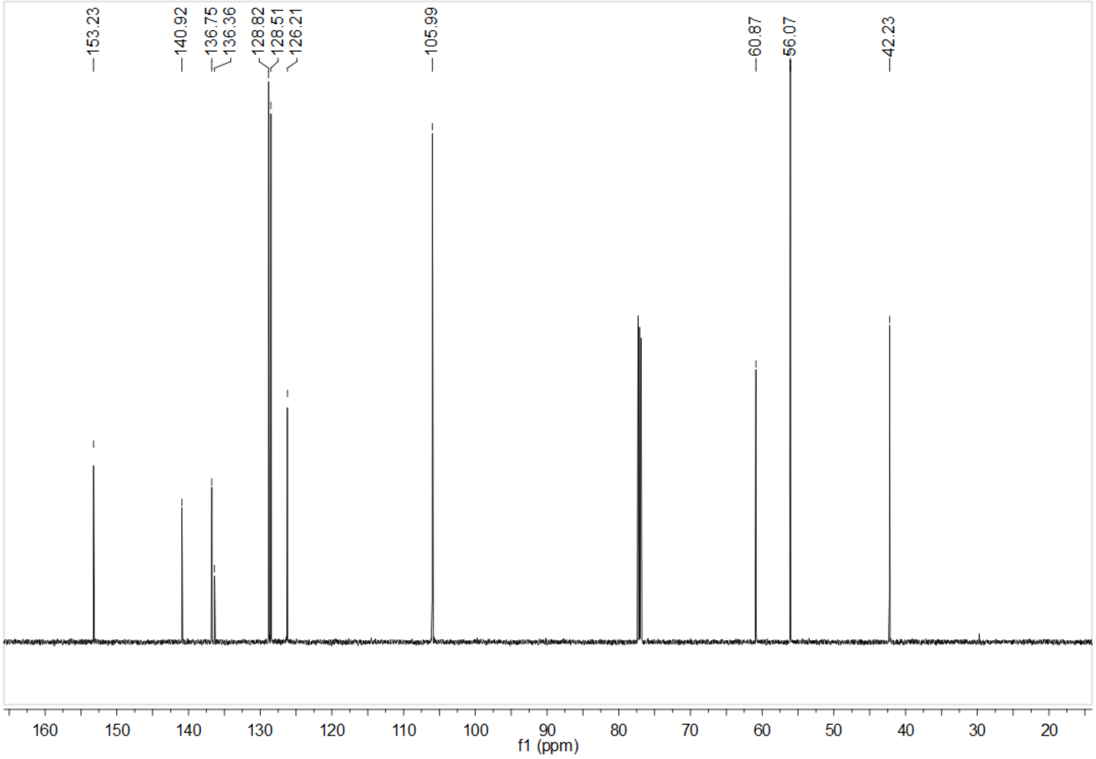
^{13}C NMR Spectrum of Compound 1i



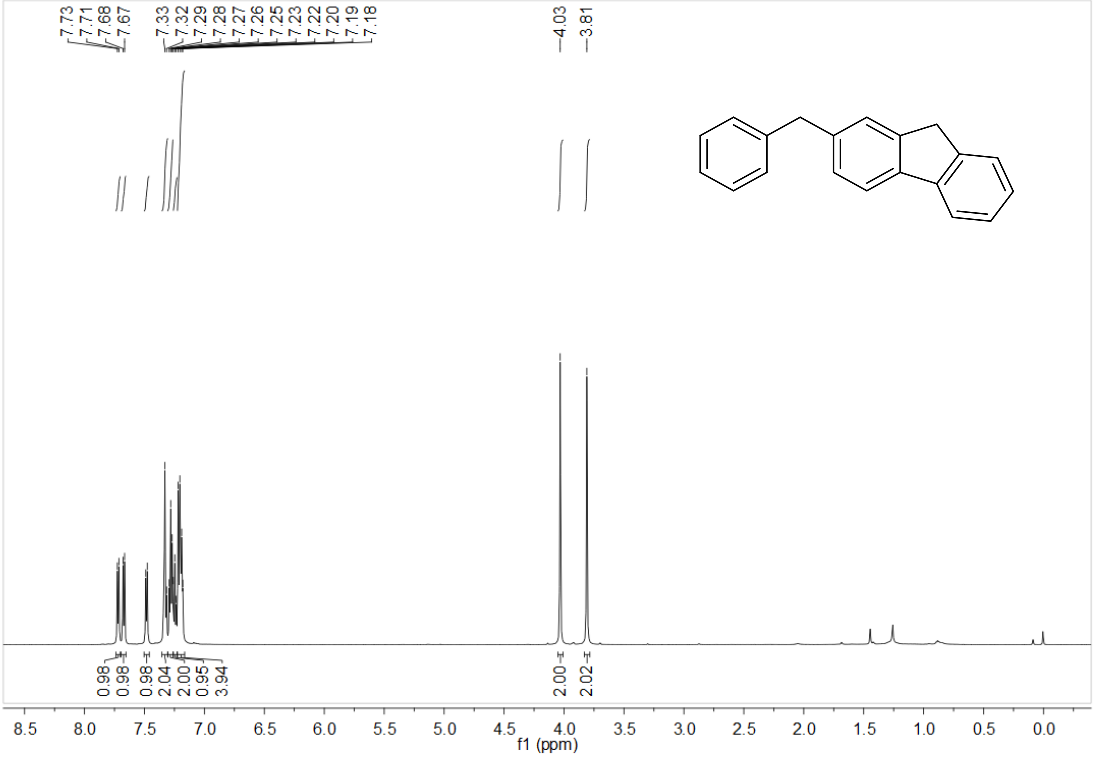
^{1}H NMR Spectrum of Compound 1j



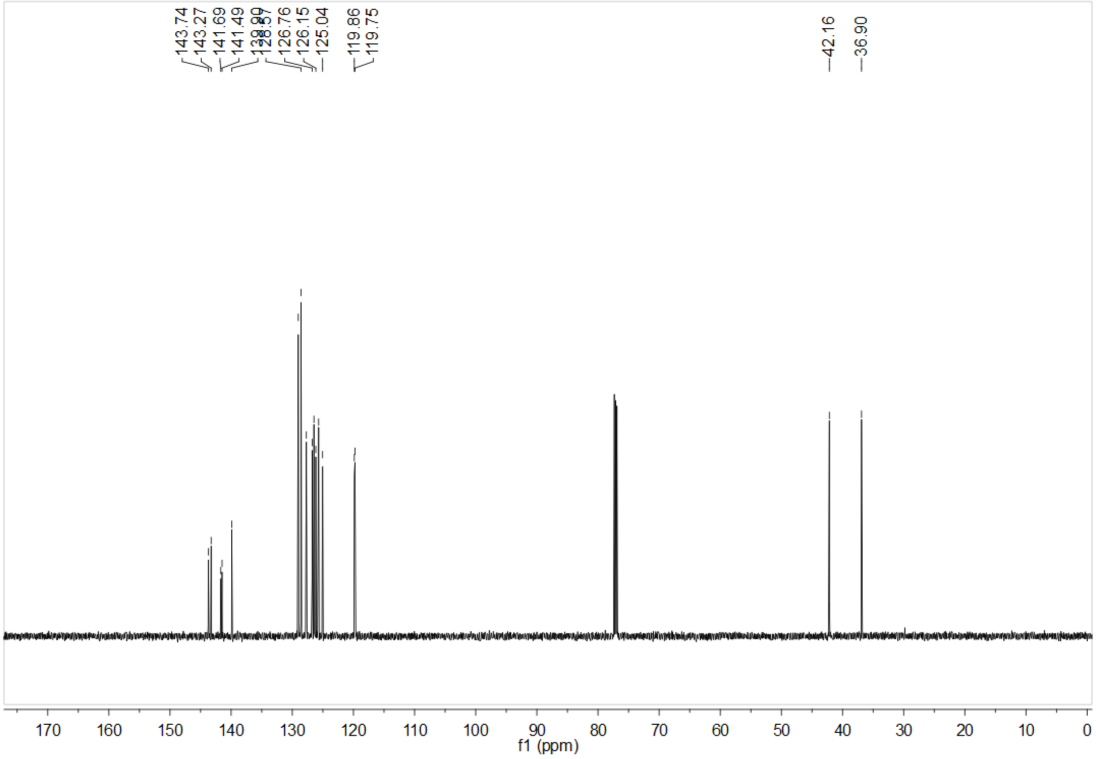
^{13}C NMR Spectrum of Compound 1j



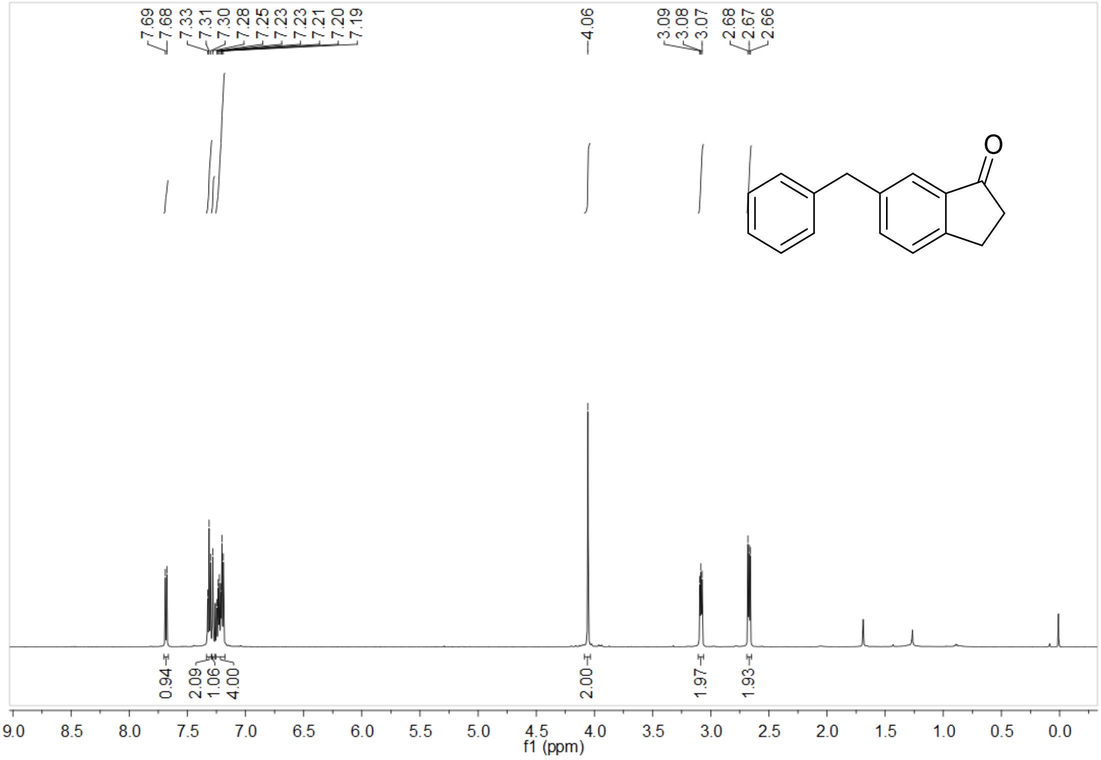
^{1}H NMR Spectrum of Compound 1k



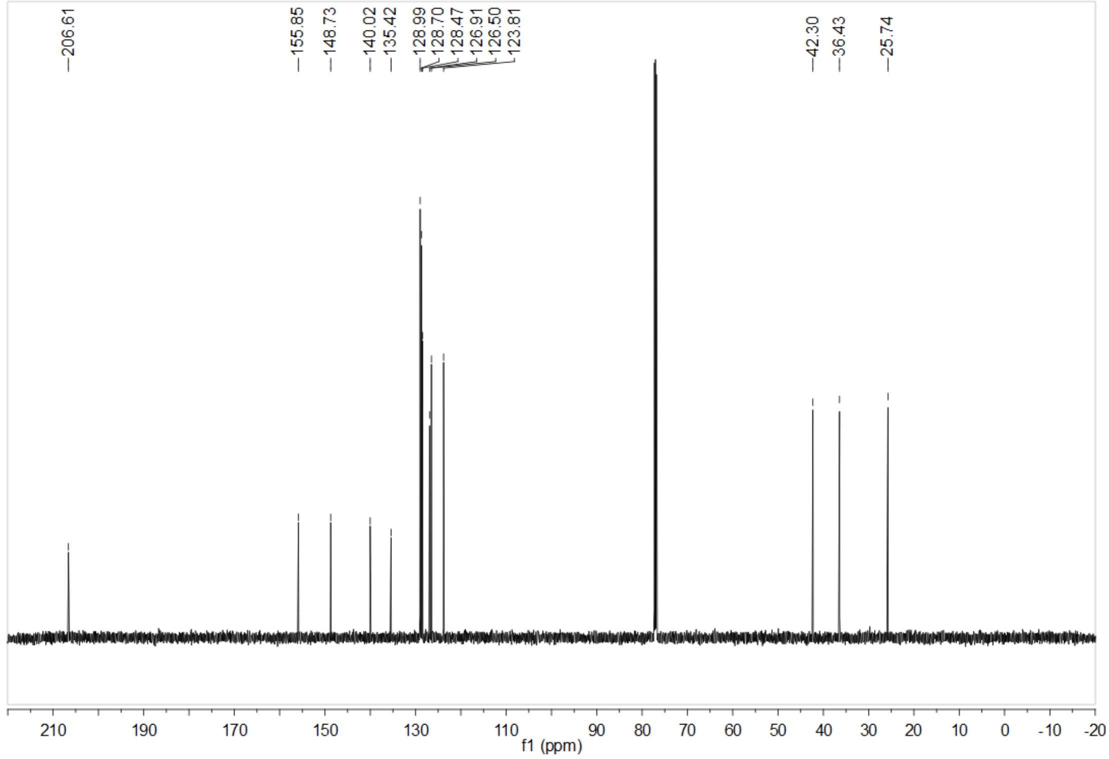
^{13}C NMR Spectrum of Compound 1k



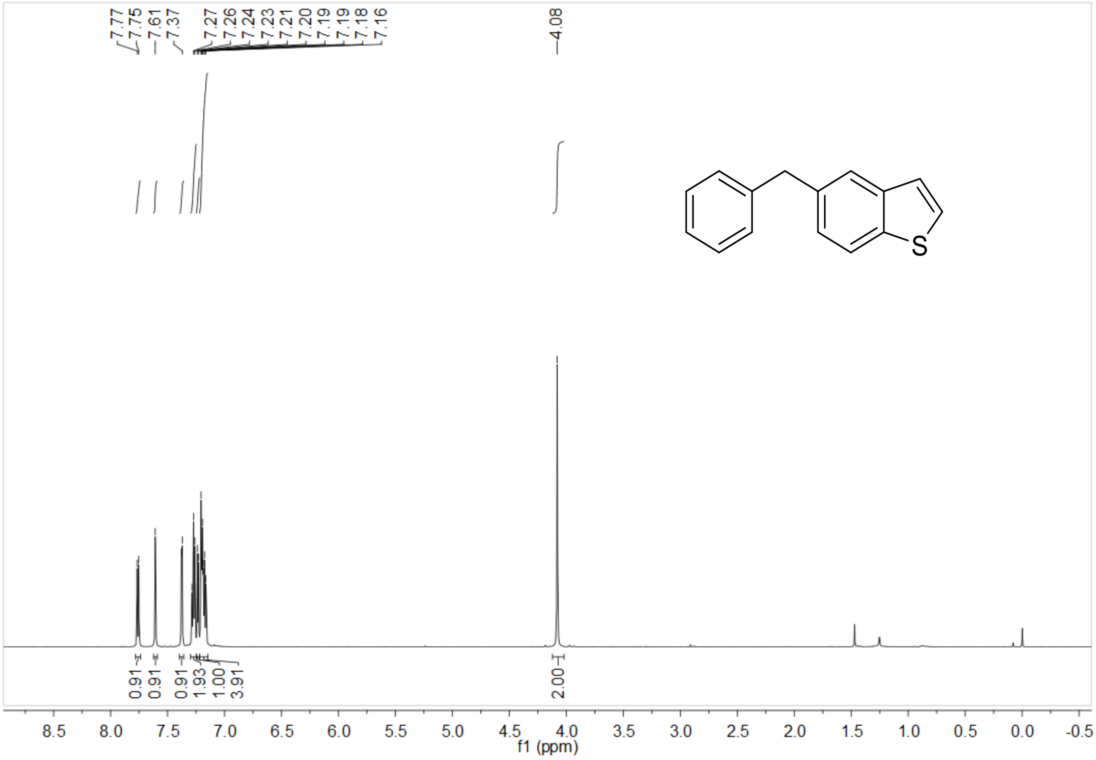
^{1}H NMR Spectrum of Compound 1l



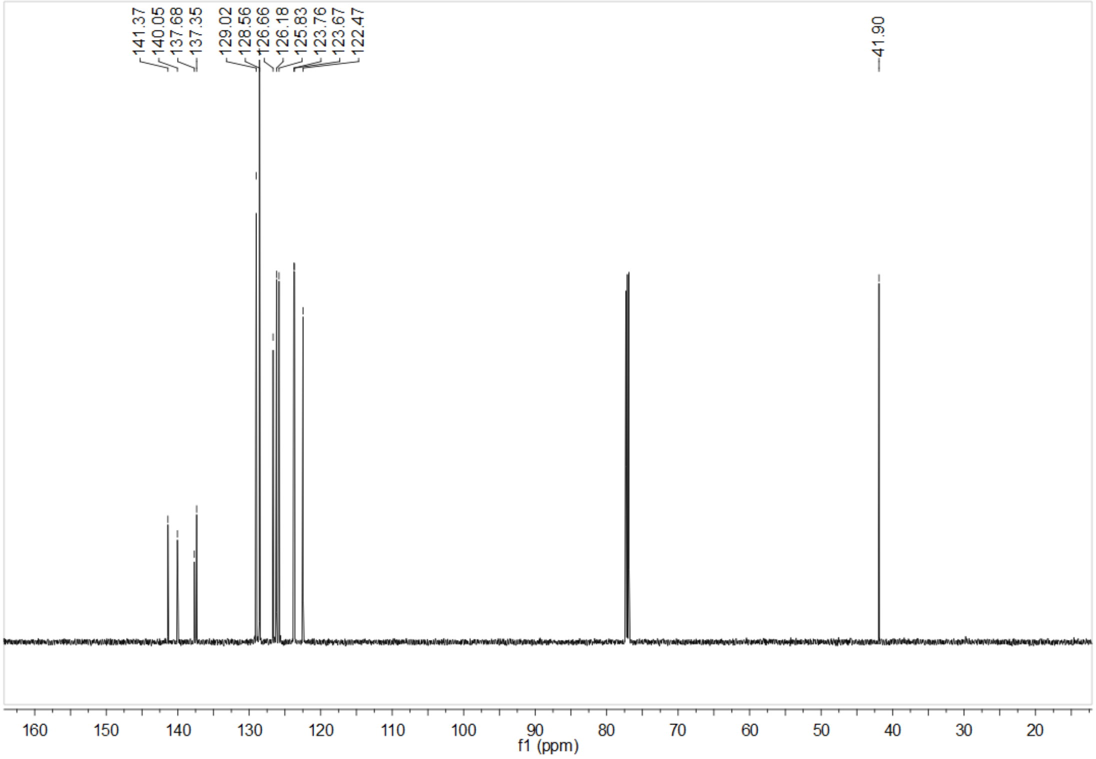
^{13}C NMR Spectrum of Compound 1l



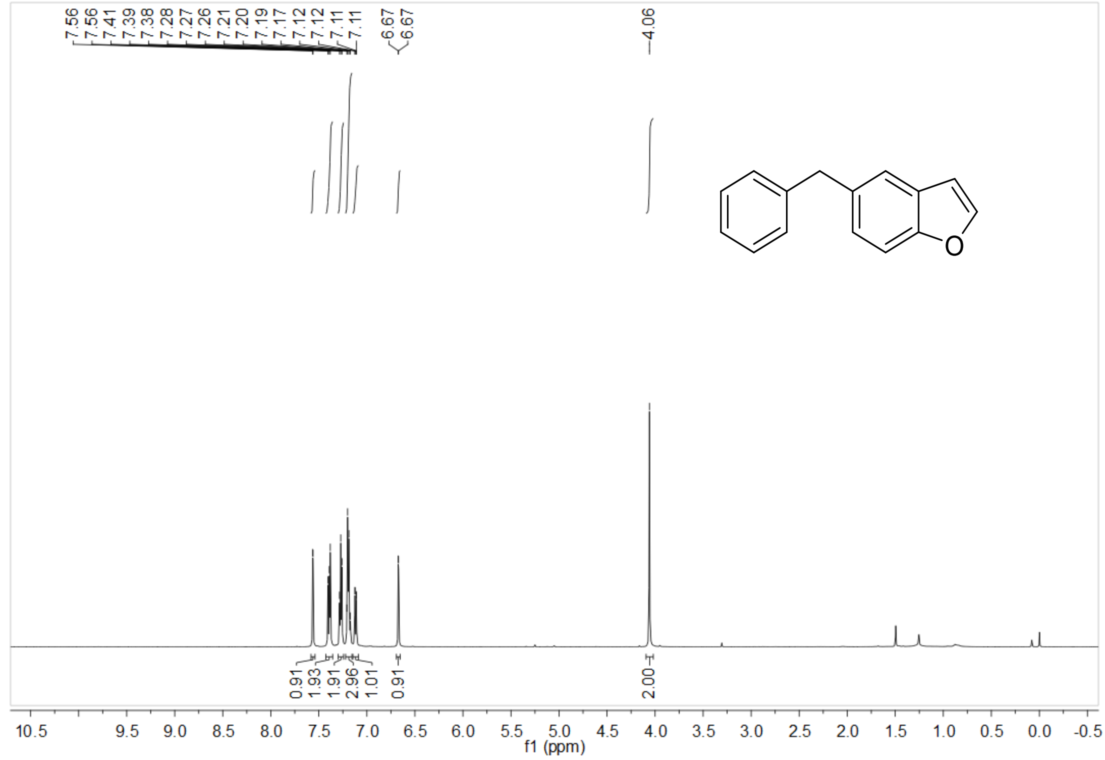
^{1}H NMR Spectrum of Compound 1m



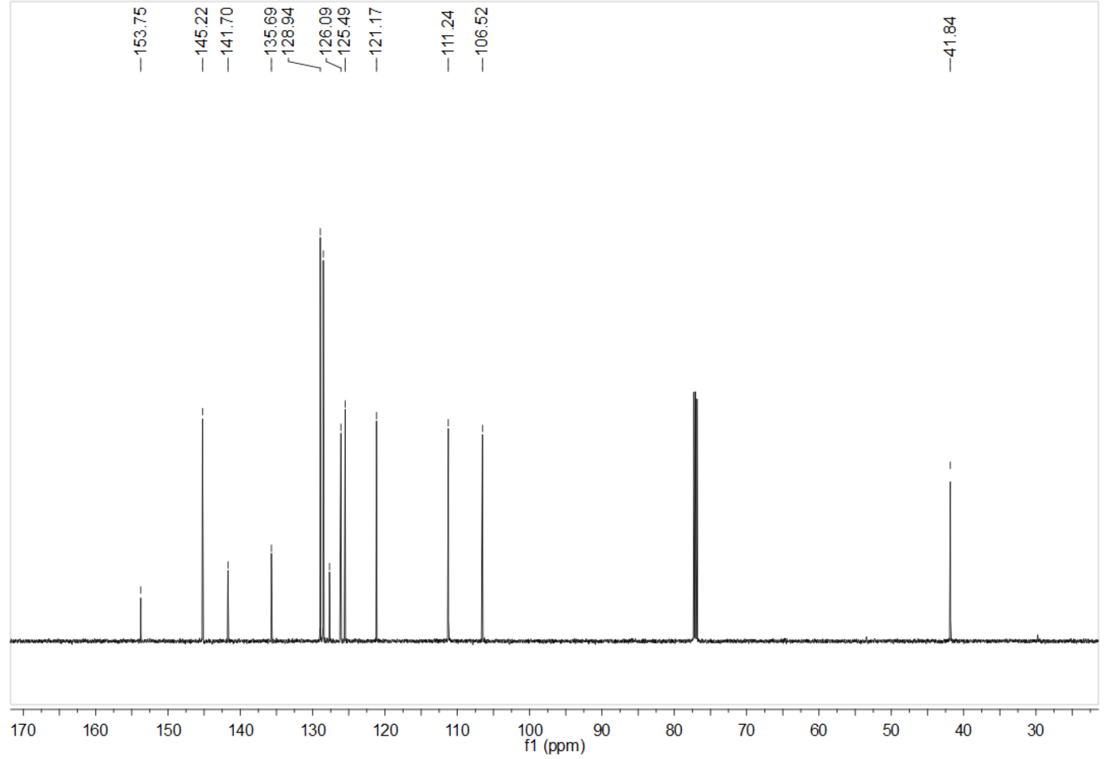
^{13}C NMR Spectrum of Compound 1m



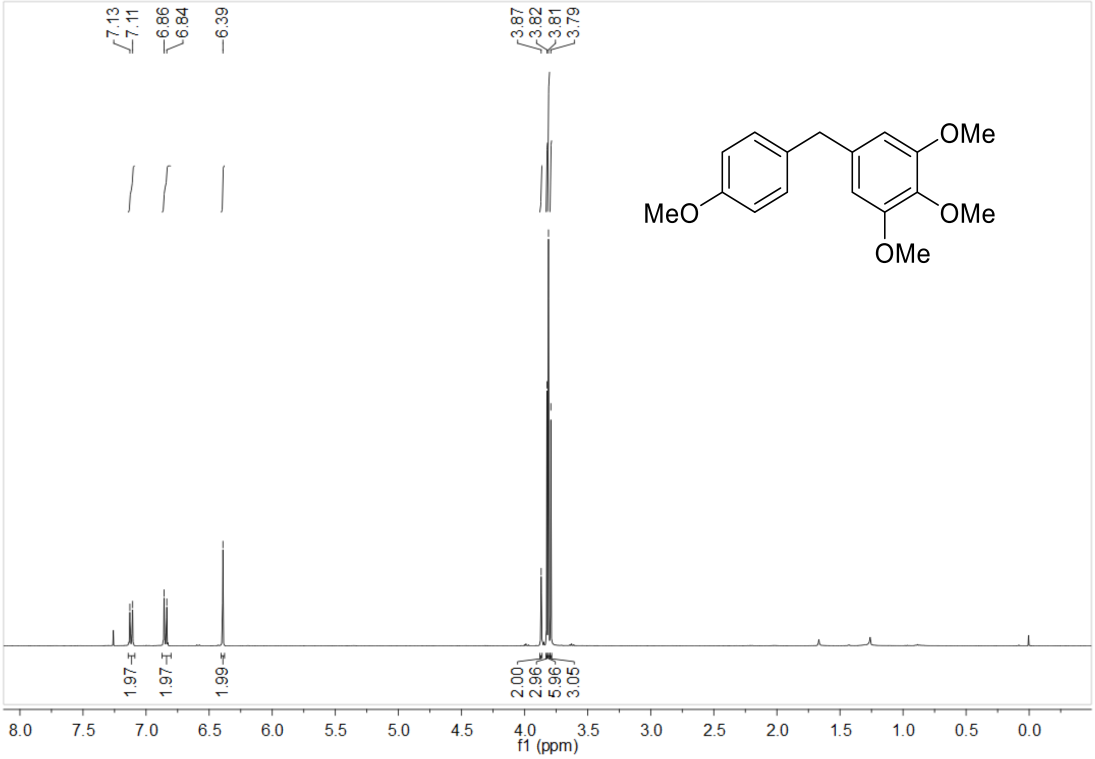
^{1}H NMR Spectrum of Compound 1n



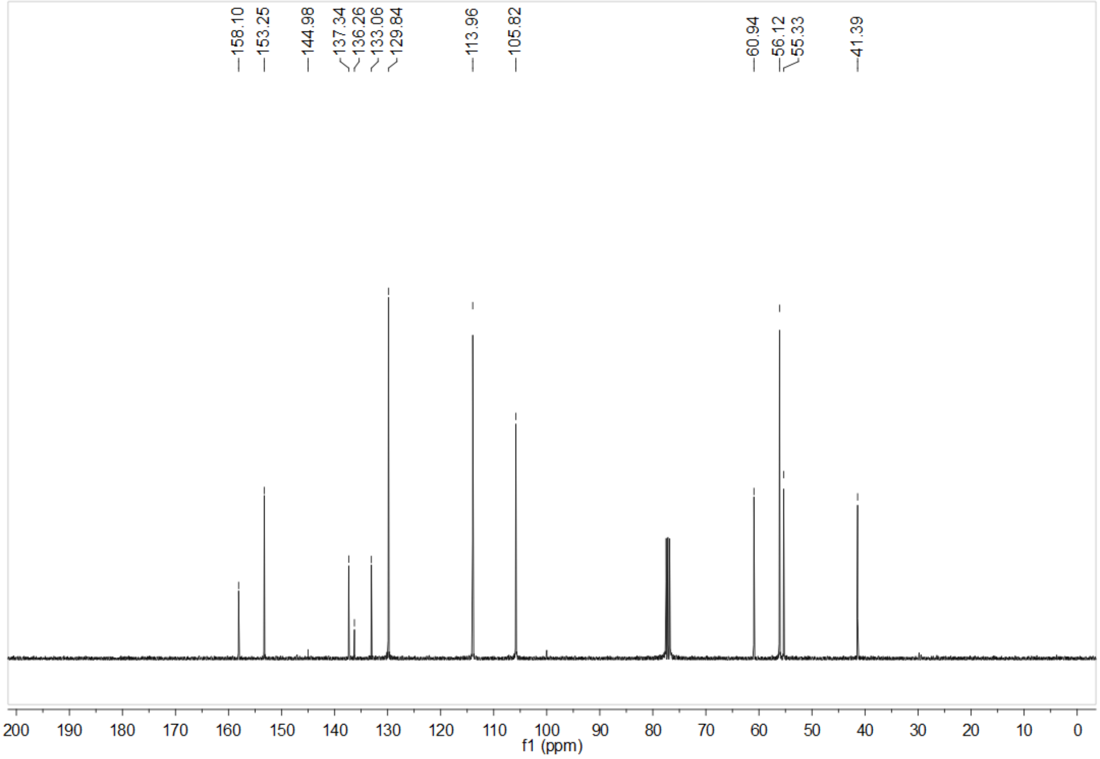
^{13}C NMR Spectrum of Compound 1n



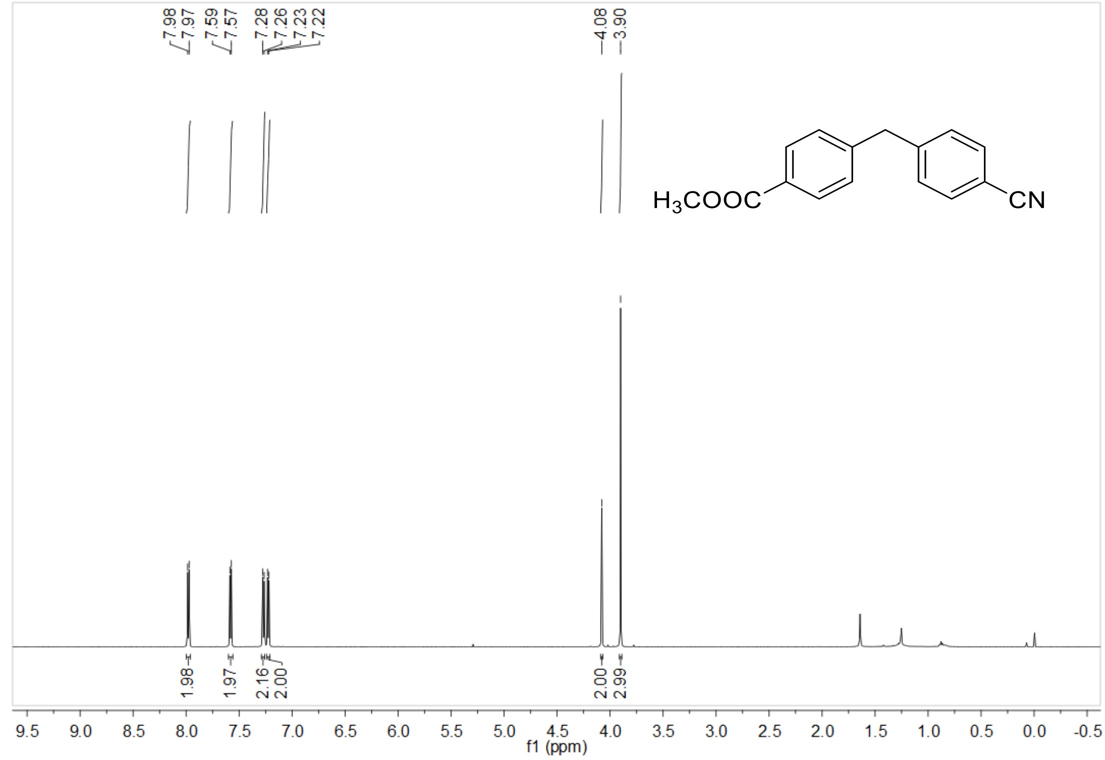
^{1}H NMR Spectrum of Compound 2a



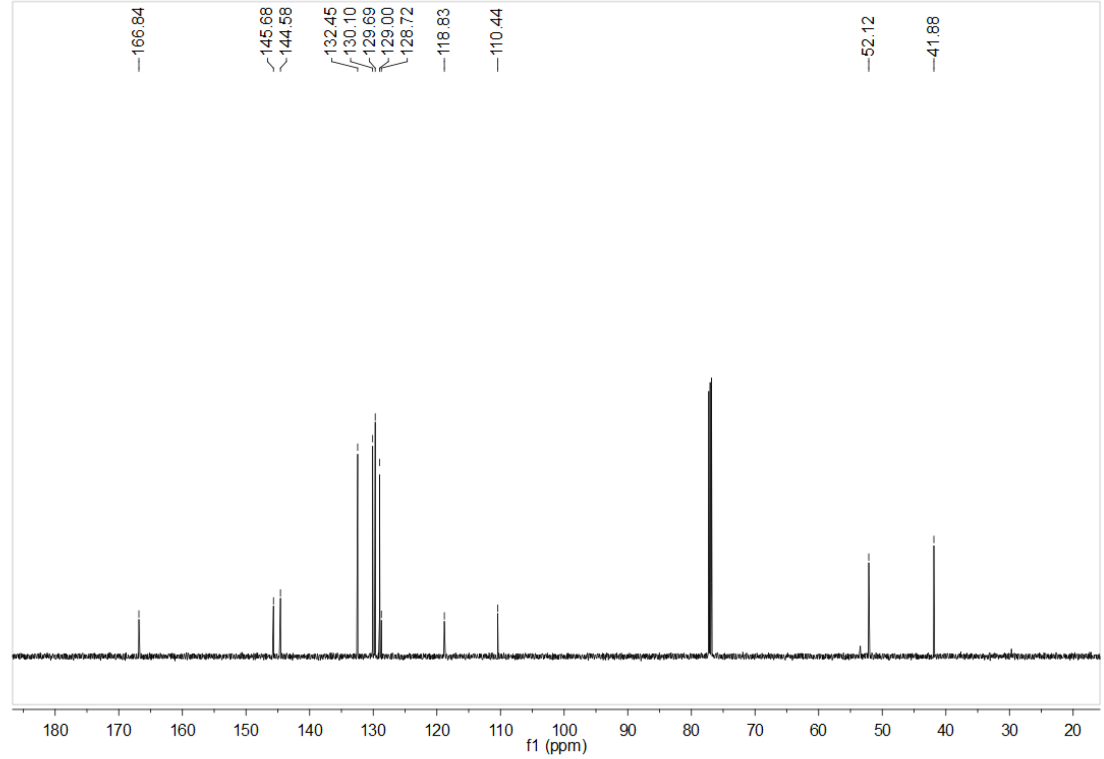
^{13}C NMR Spectrum of Compound 2a



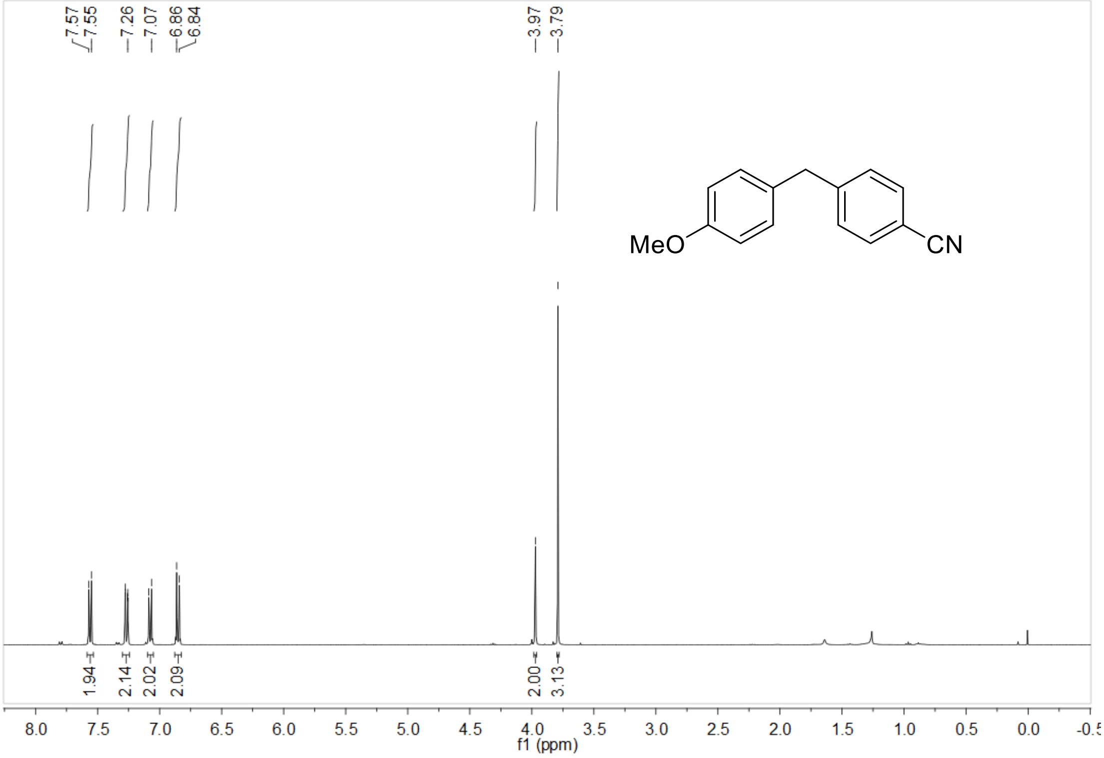
^{1}H NMR Spectrum of Compound 3a



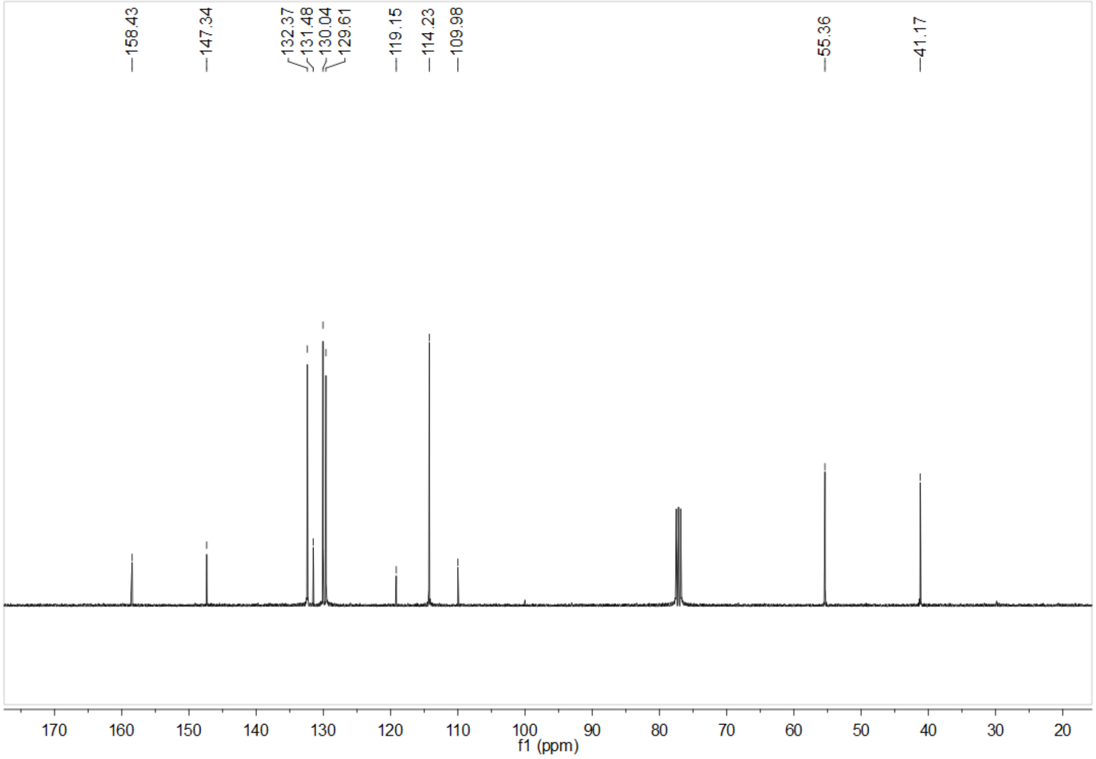
^{13}C NMR Spectrum of Compound 3a



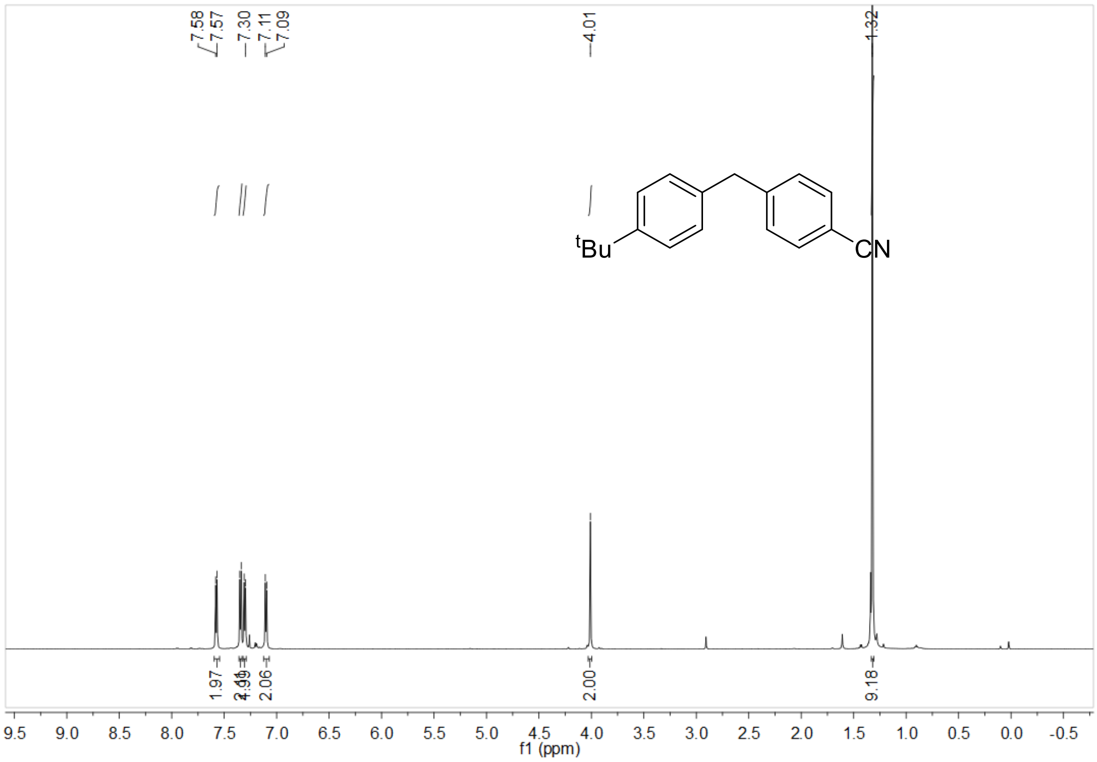
^{1}H NMR Spectrum of Compound 4a



^{13}C NMR Spectrum of Compound 4a



^{1}H NMR Spectrum of Compound 5a



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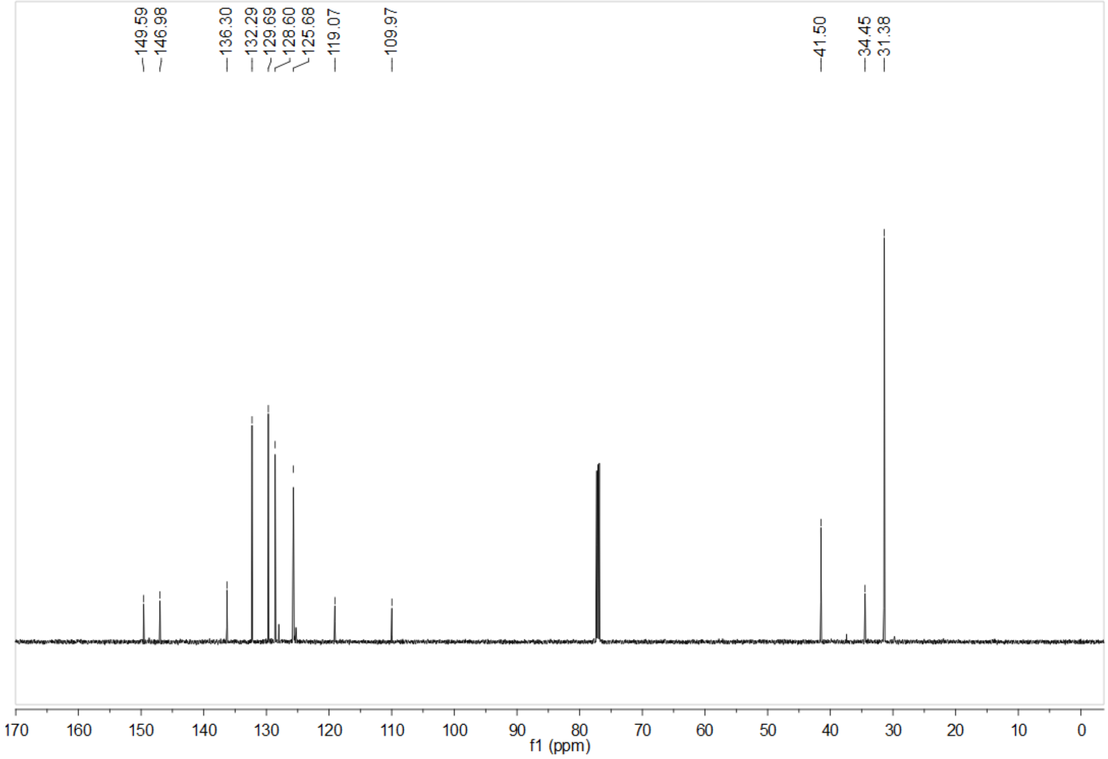
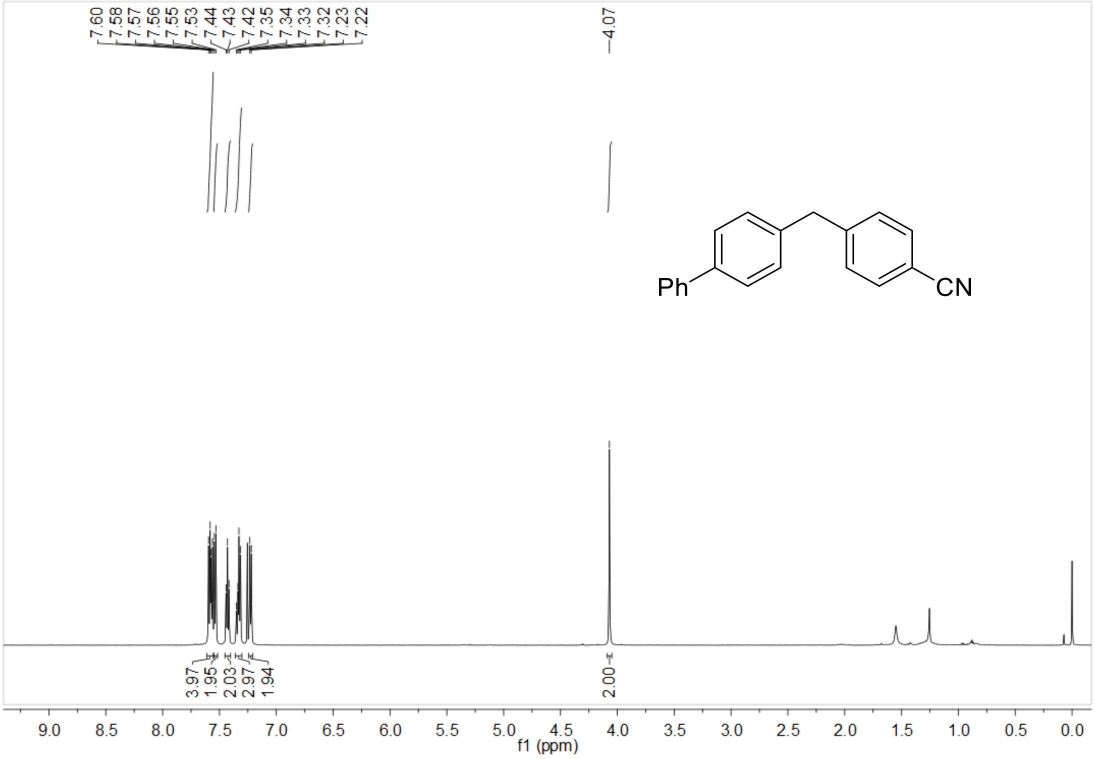
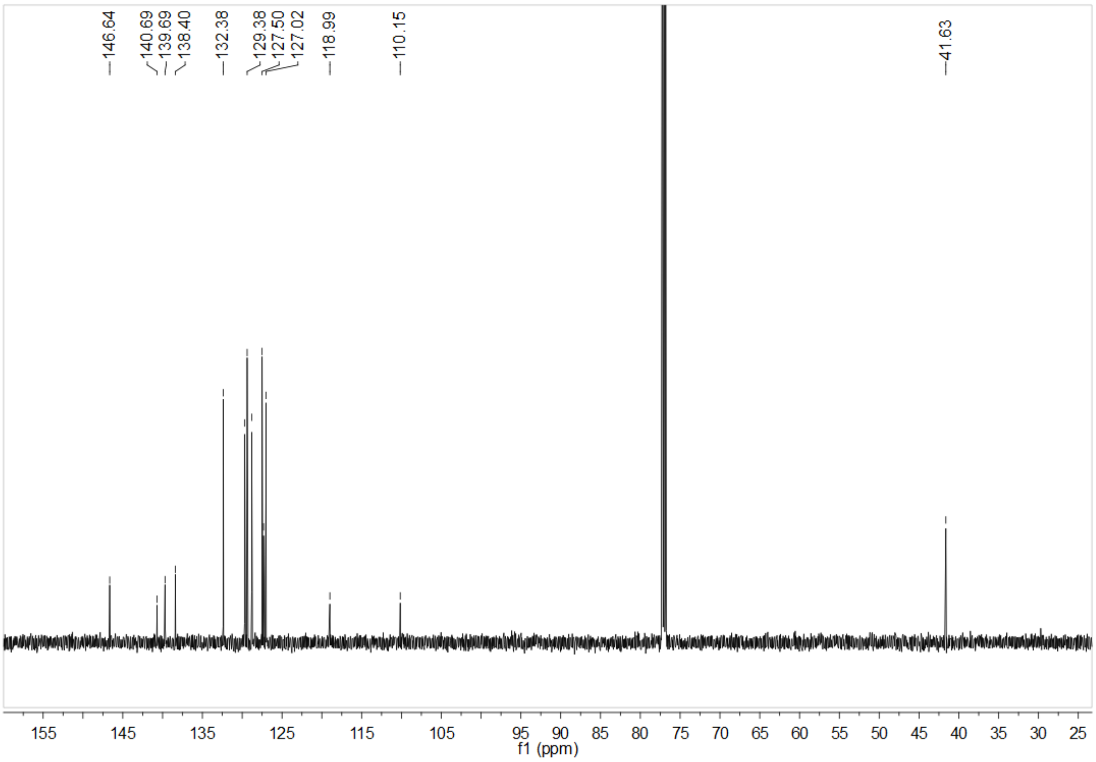


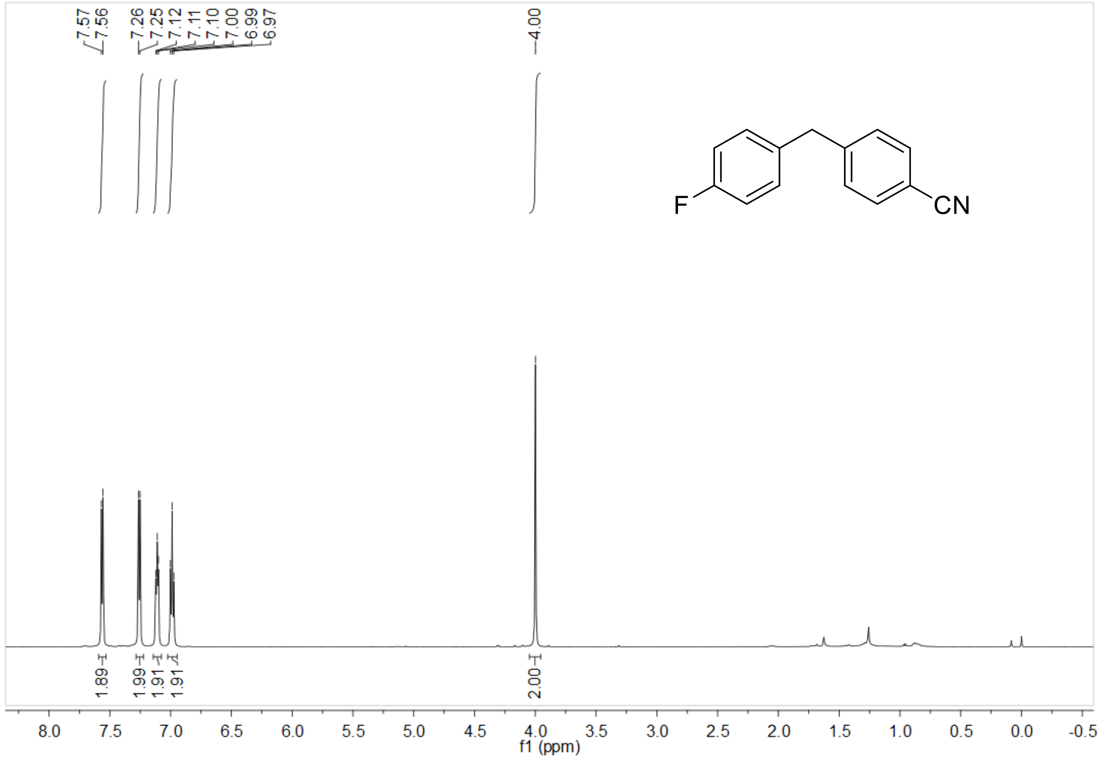
^{1}H NMR Spectrum of Compound 6a



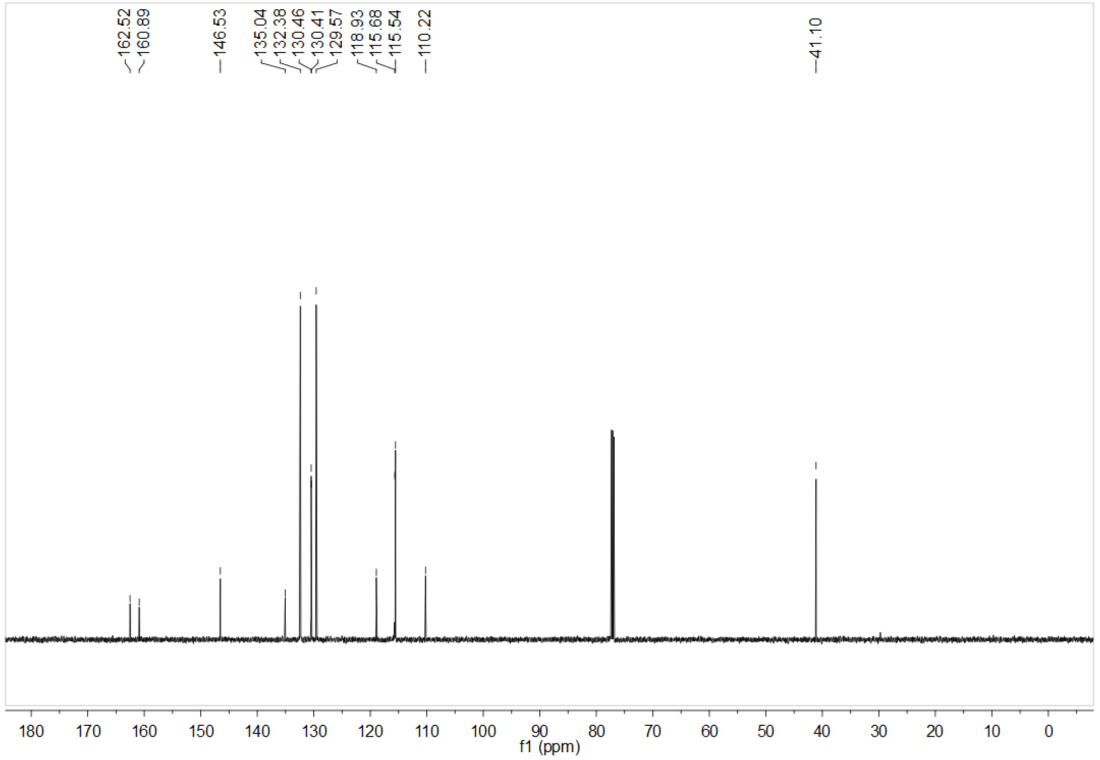
^{13}C NMR Spectrum of Compound 6a



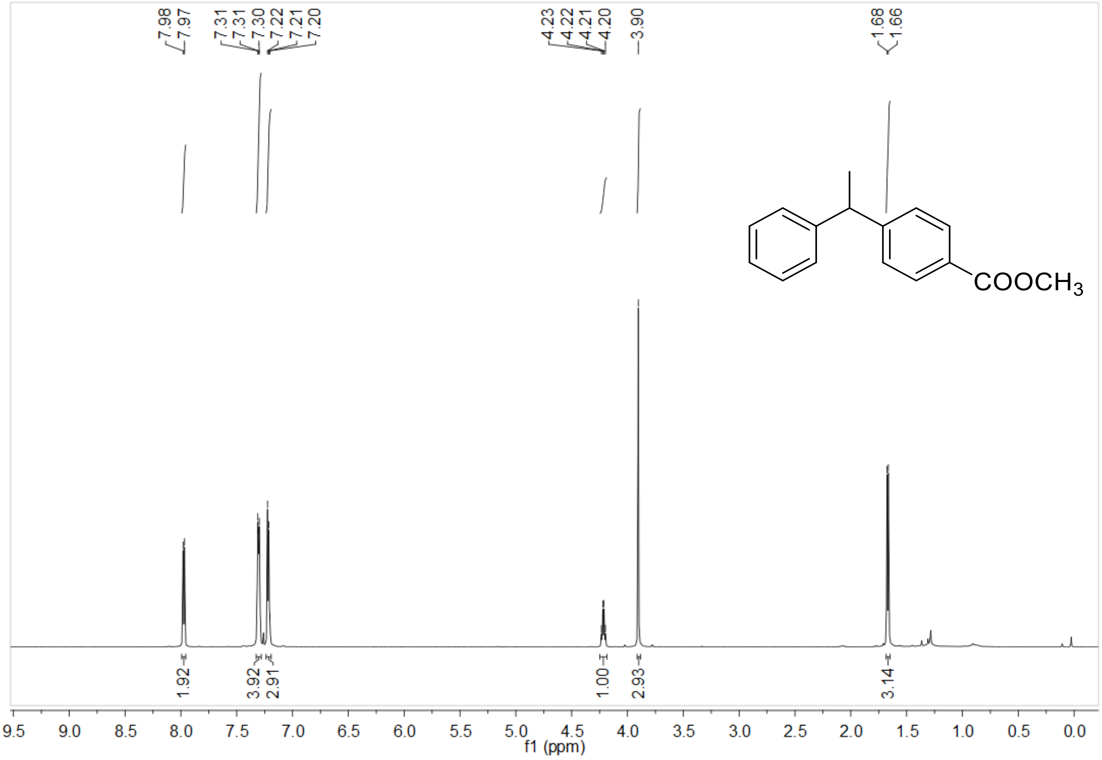
^{1}H NMR Spectrum of Compound 7a



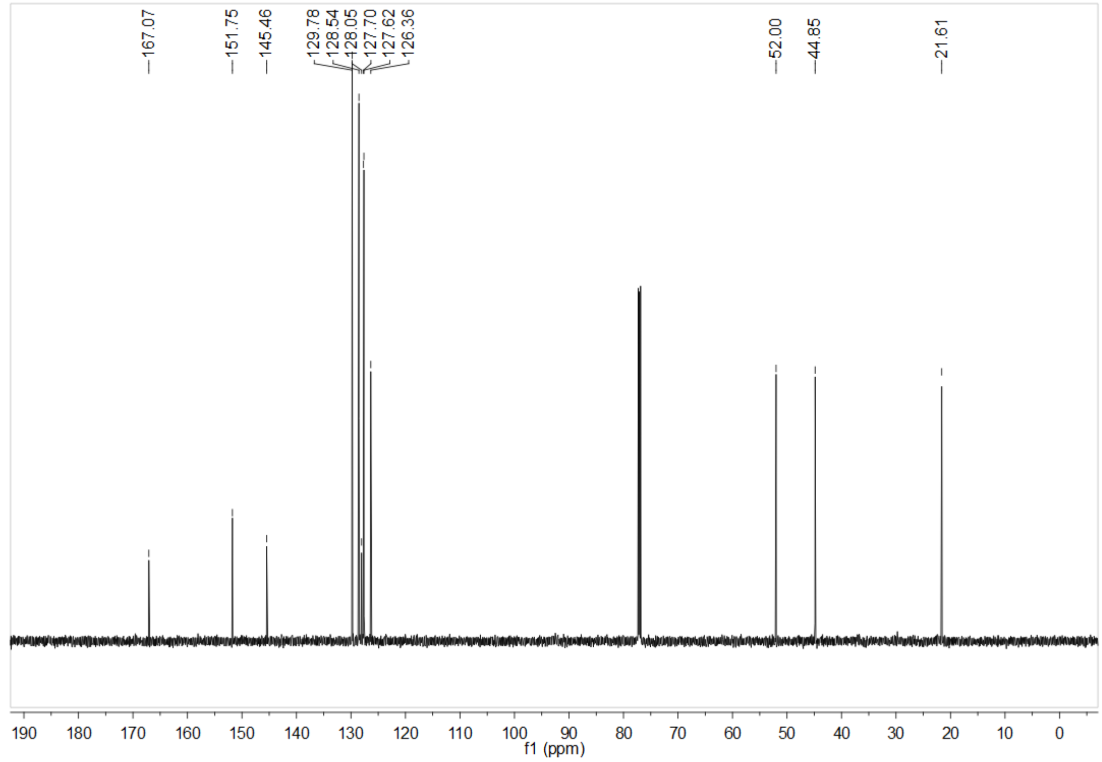
^{13}C NMR Spectrum of Compound 12b



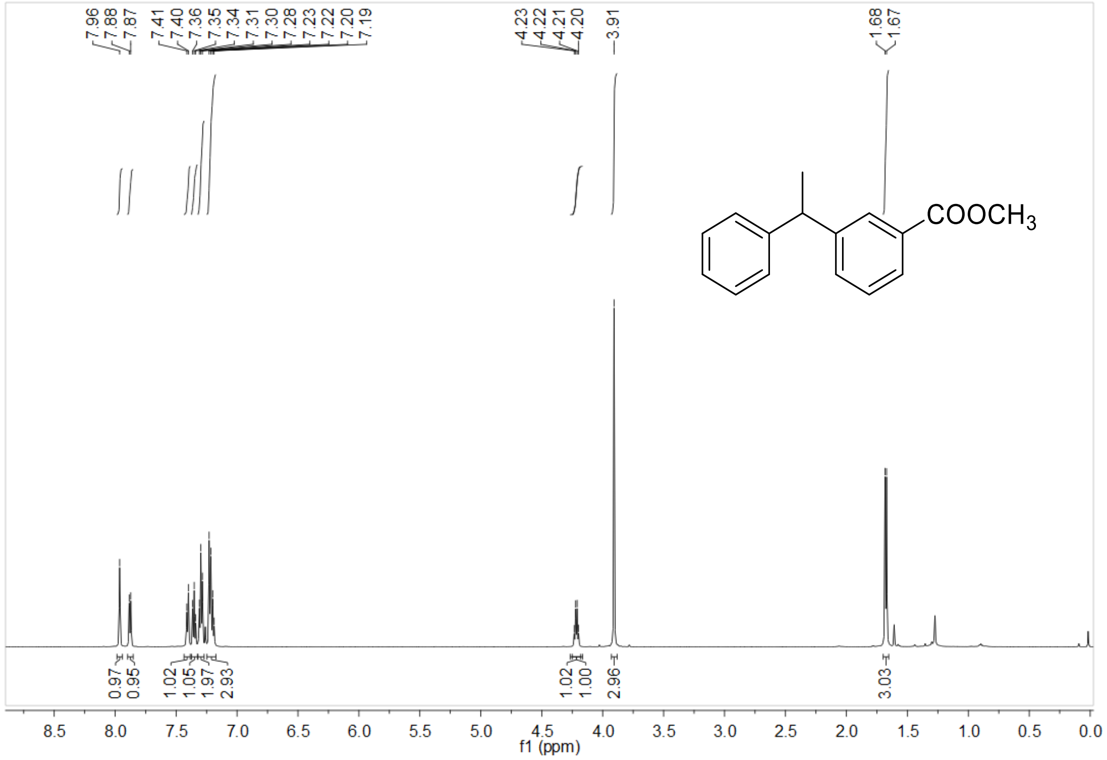
^{1}H NMR Spectrum of Compound 8a



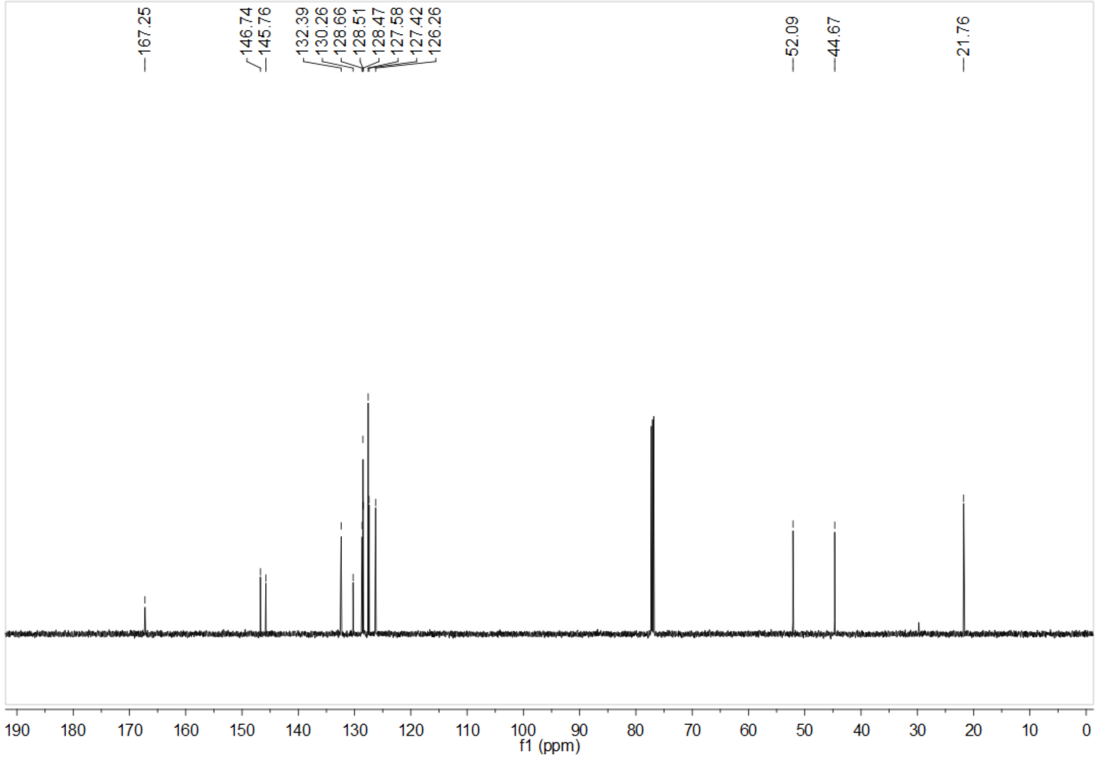
^{13}C NMR Spectrum of Compound 8a



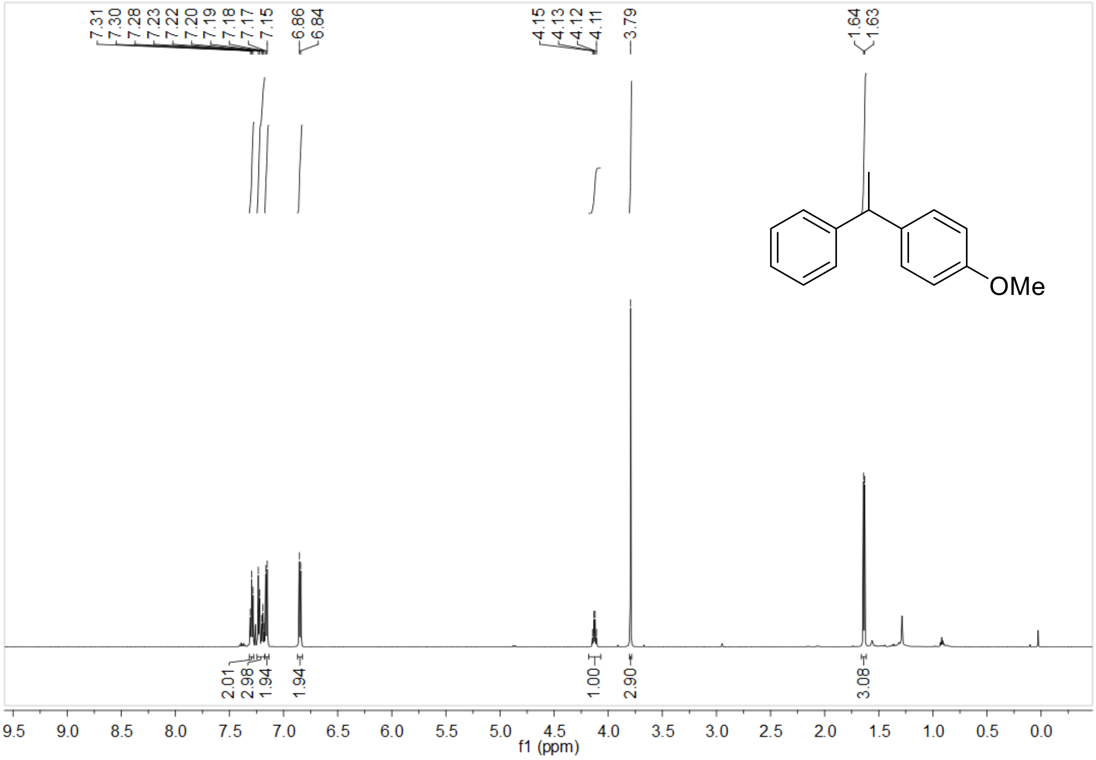
^{1}H NMR Spectrum of Compound 8b



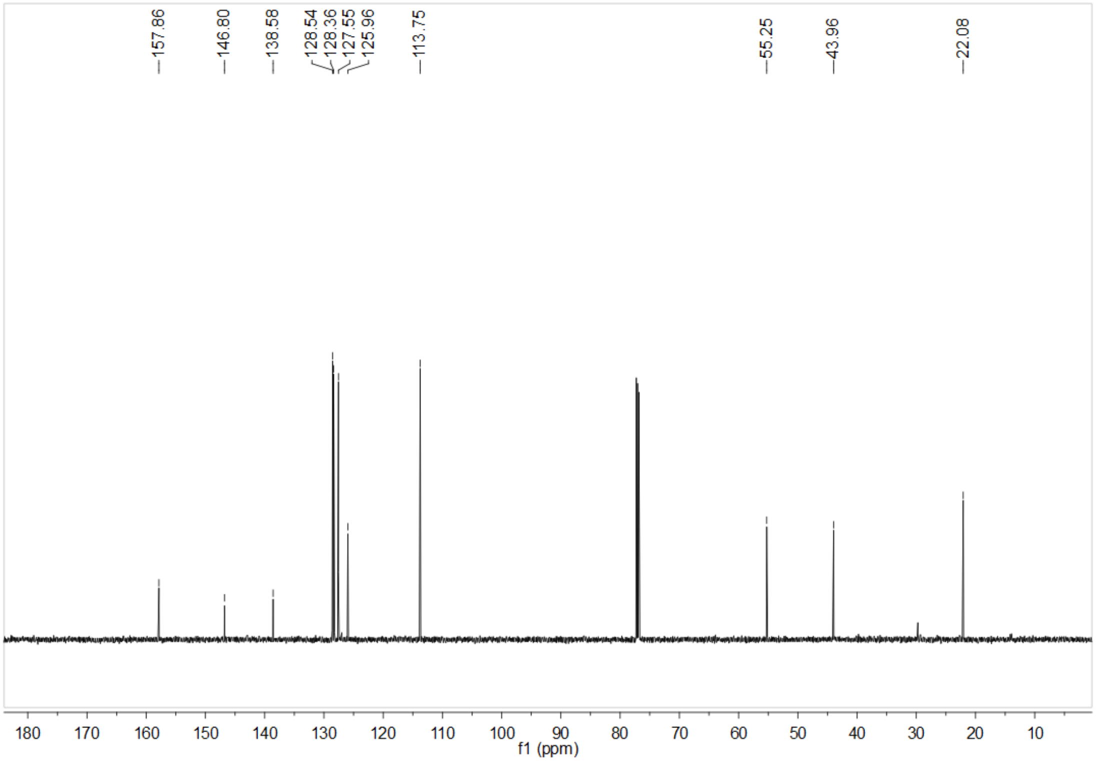
^{13}C NMR Spectrum of Compound 8b



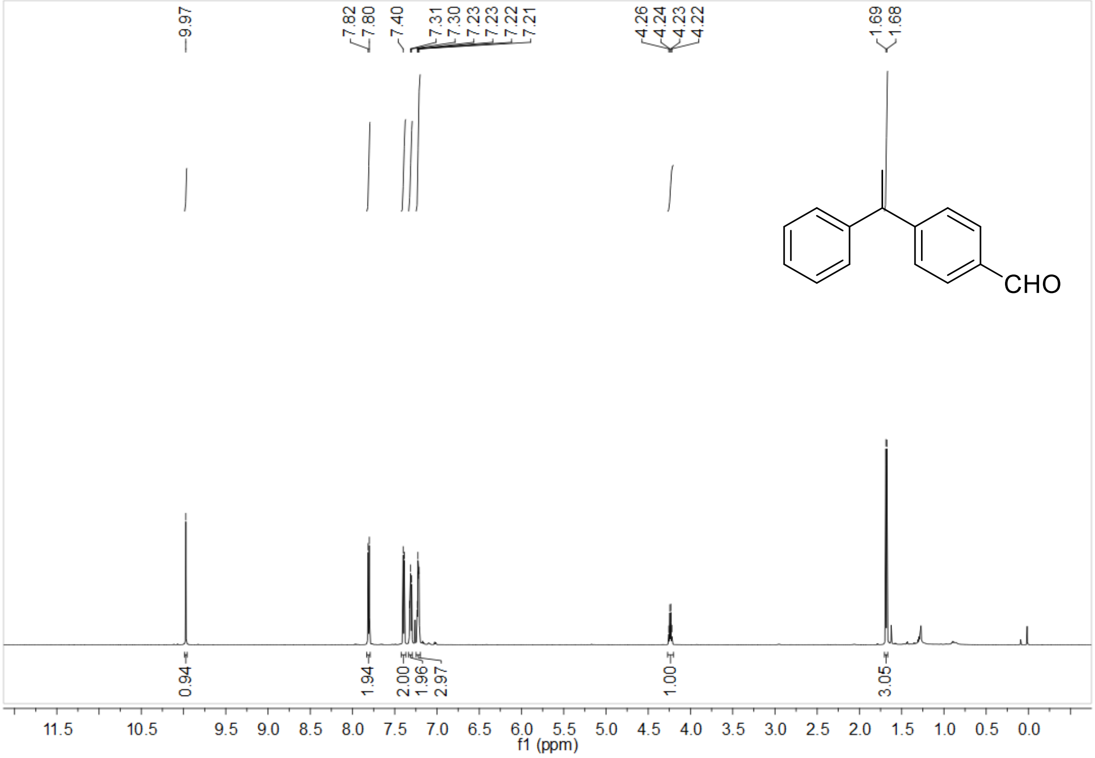
^{1}H NMR Spectrum of Compound 8d



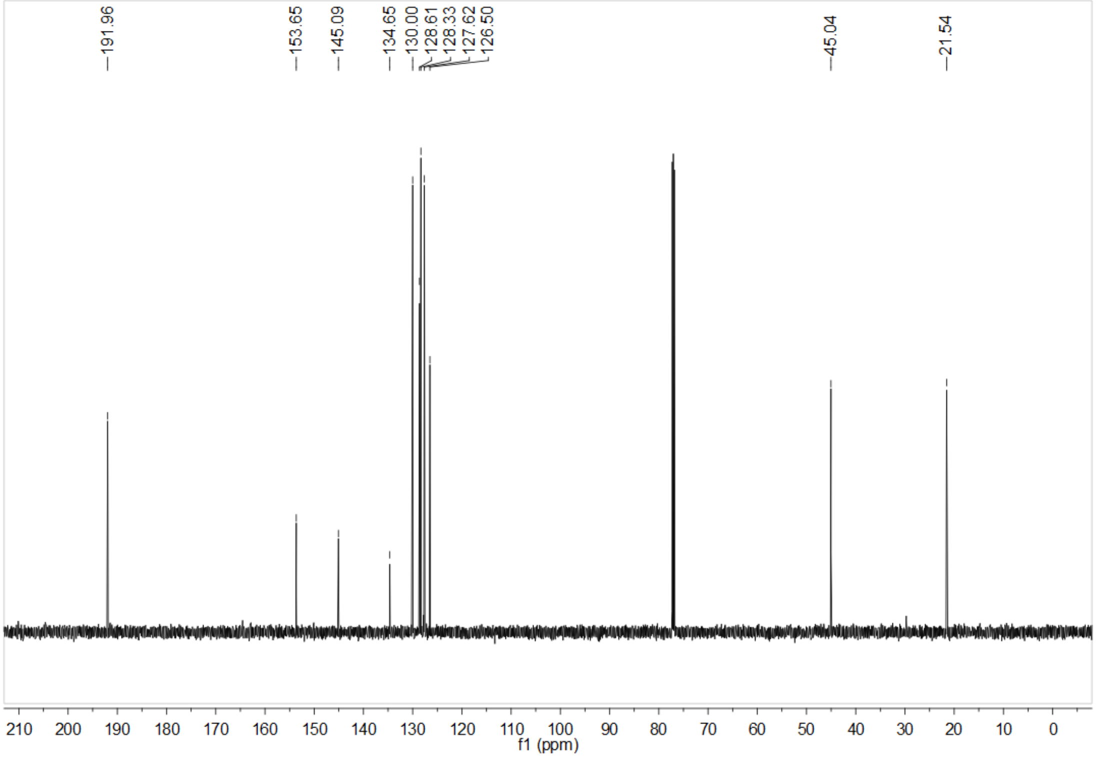
^{13}C NMR Spectrum of Compound 8d



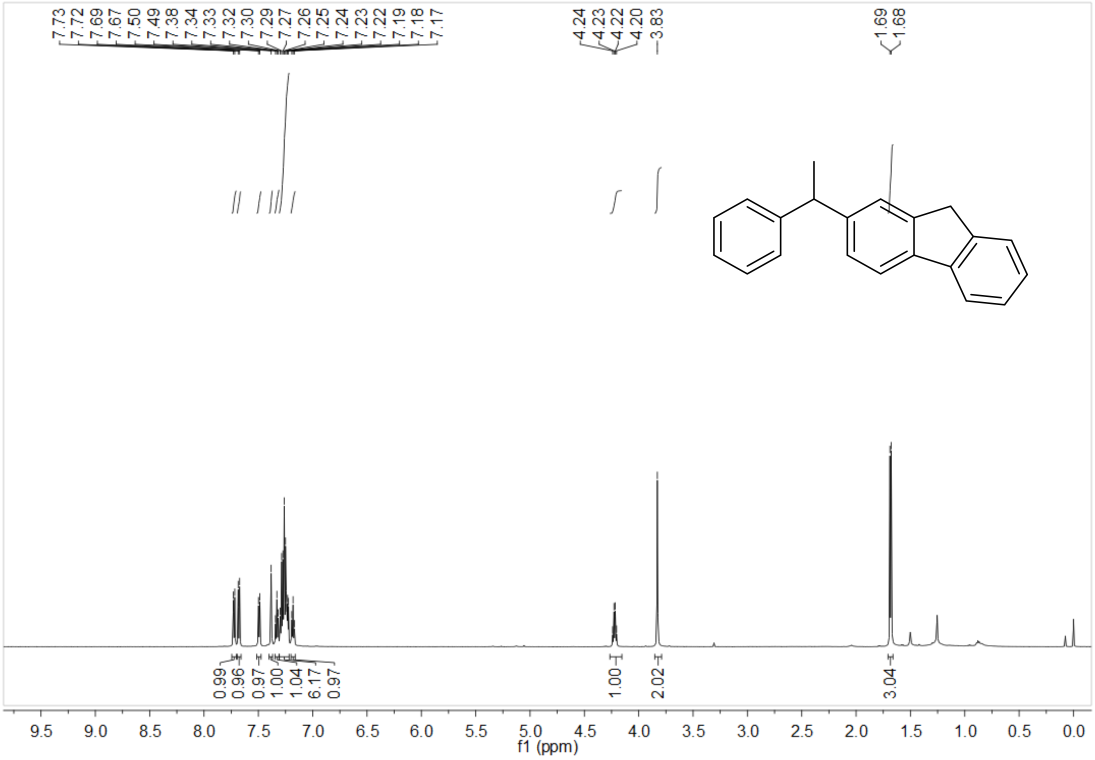
^{1}H NMR Spectrum of Compound 8e



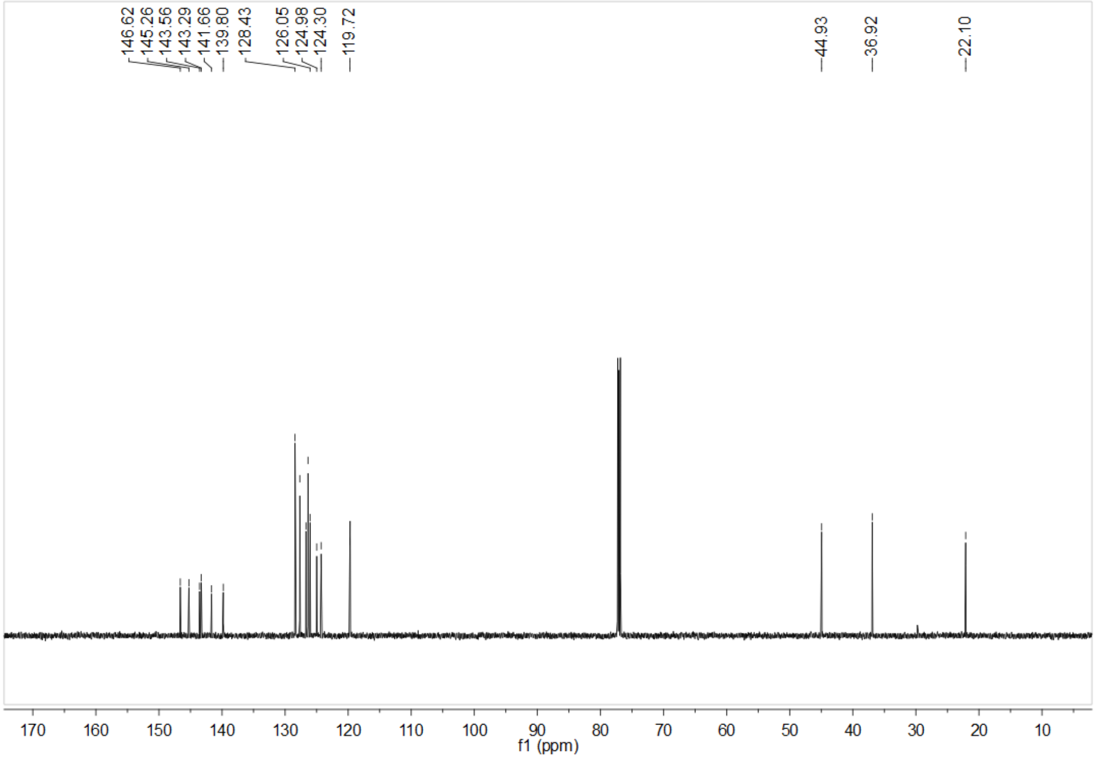
^{13}C NMR Spectrum of Compound 8e



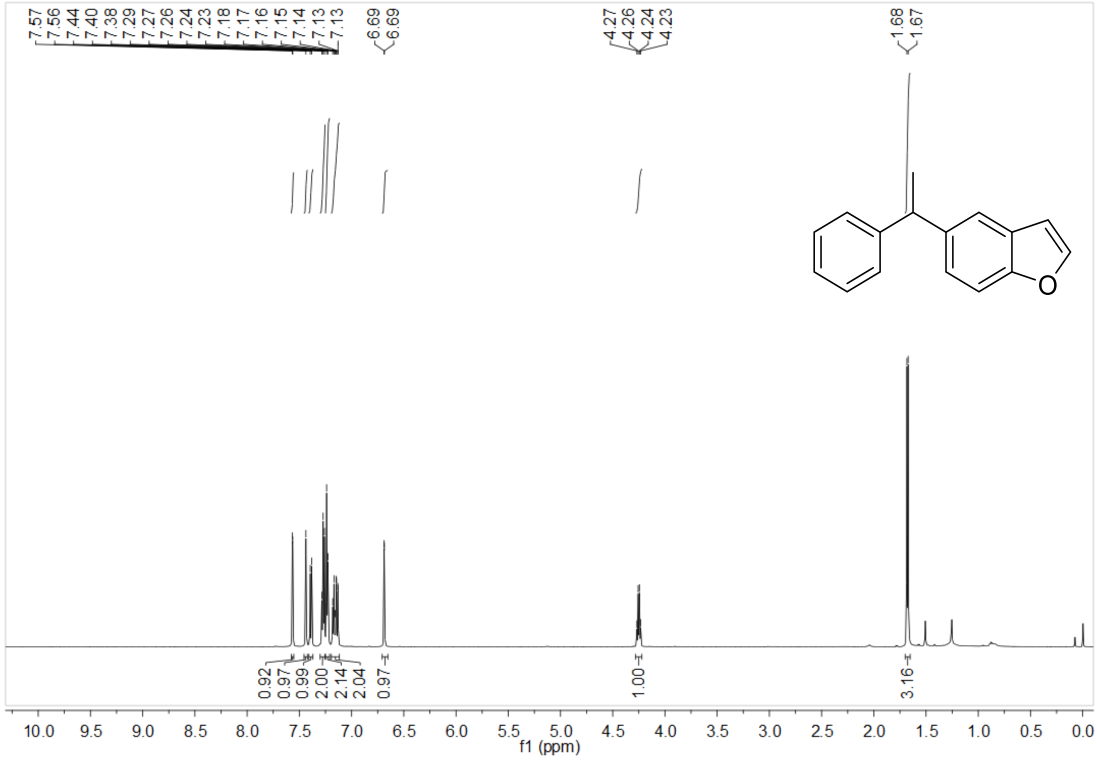
^{1}H NMR Spectrum of Compound 8f



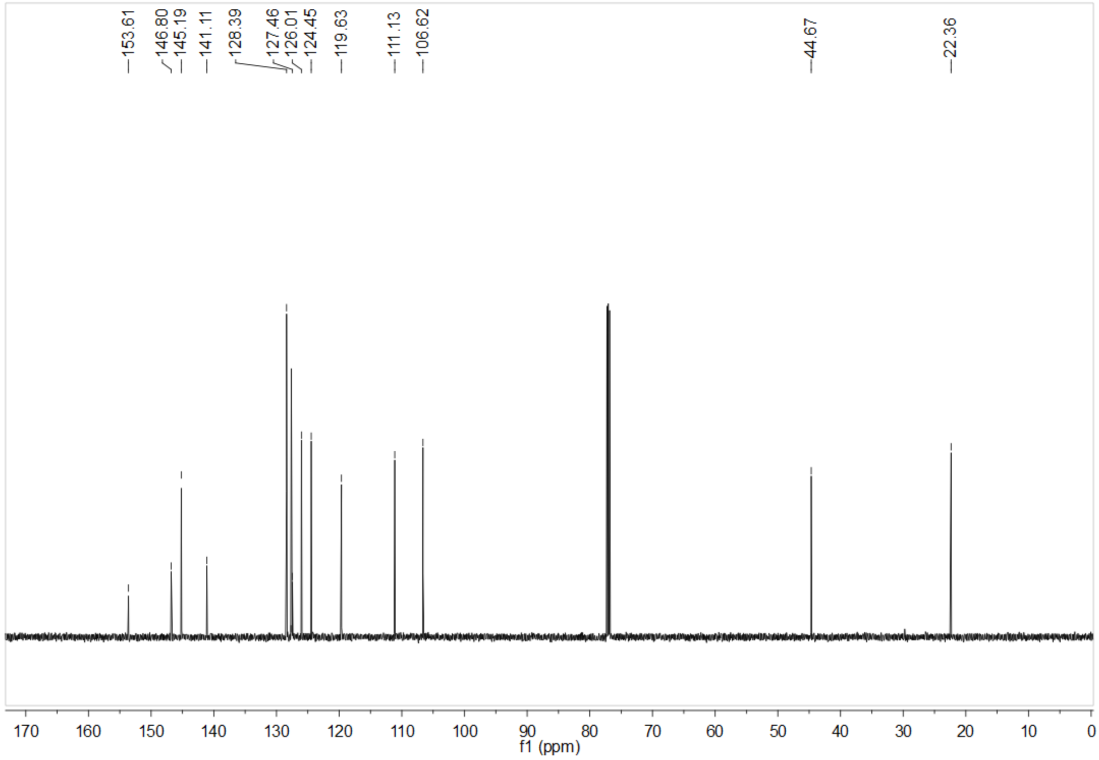
^{13}C NMR Spectrum of Compound 8f



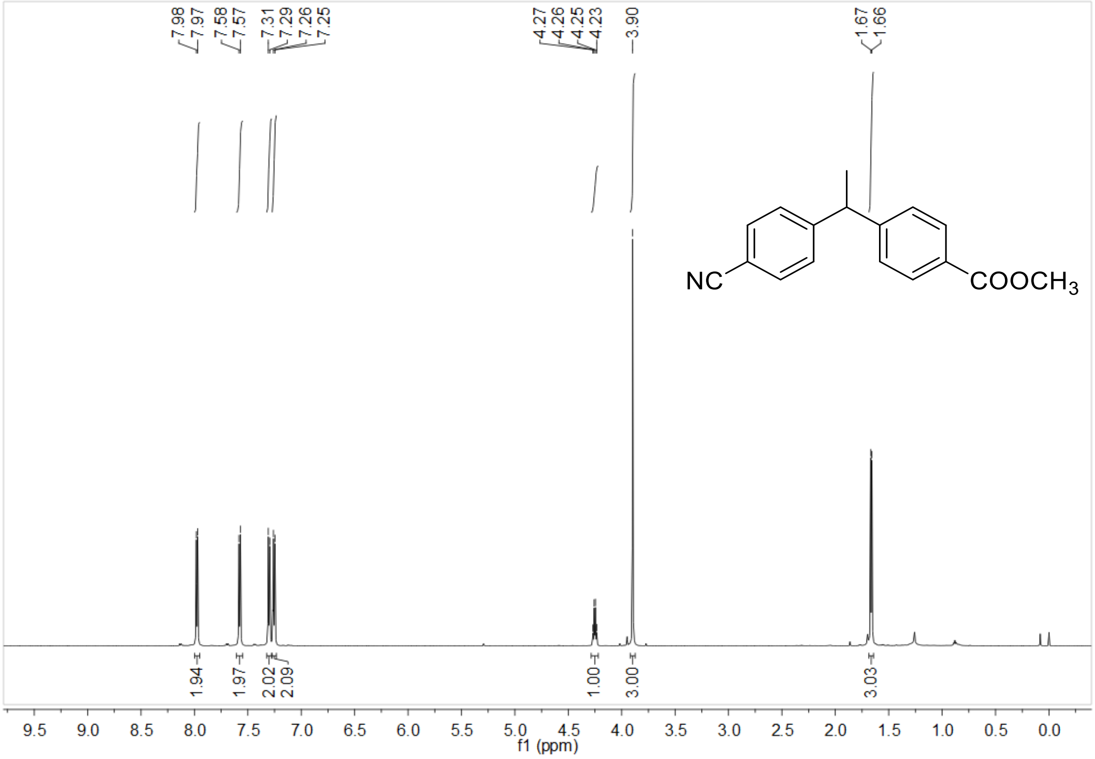
^{1}H NMR Spectrum of Compound 8g



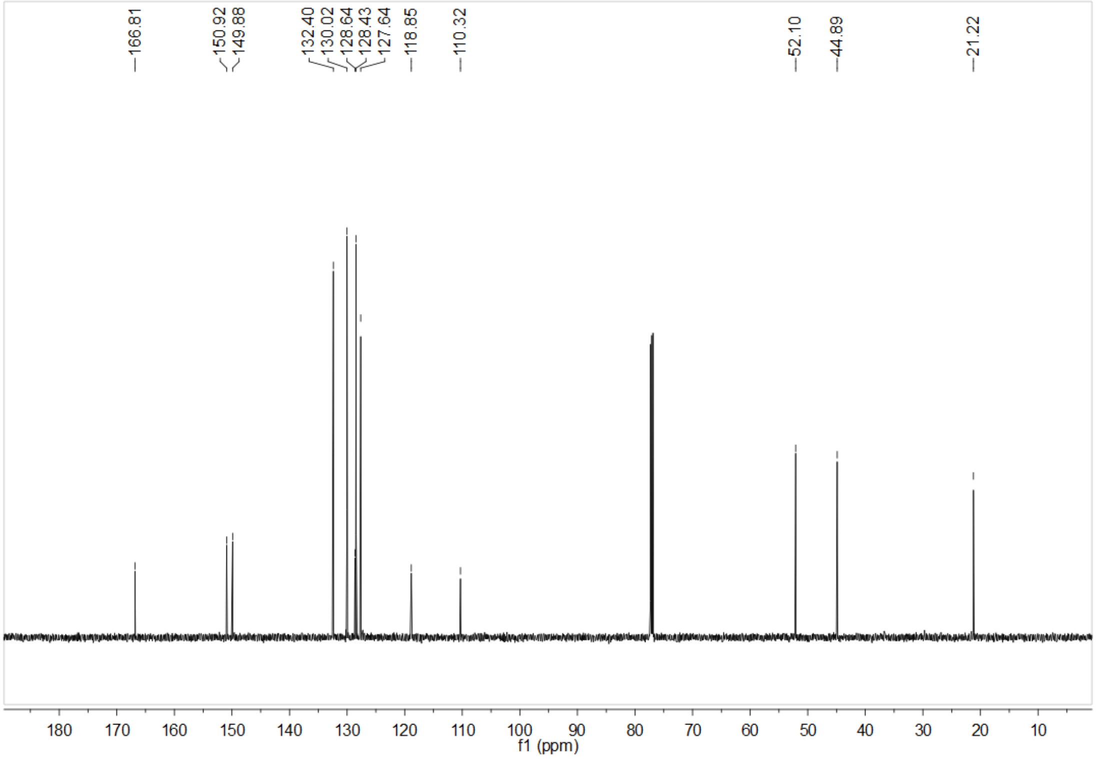
^{13}C NMR Spectrum of Compound 8g



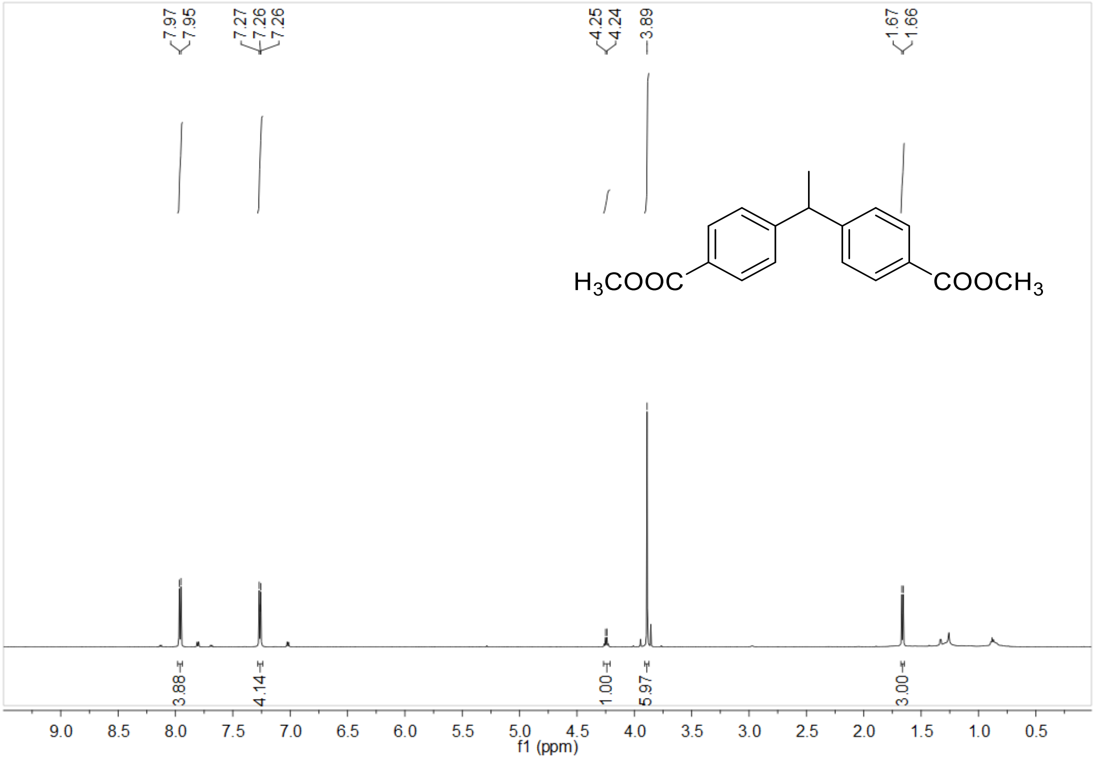
^{1}H NMR Spectrum of Compound 9a



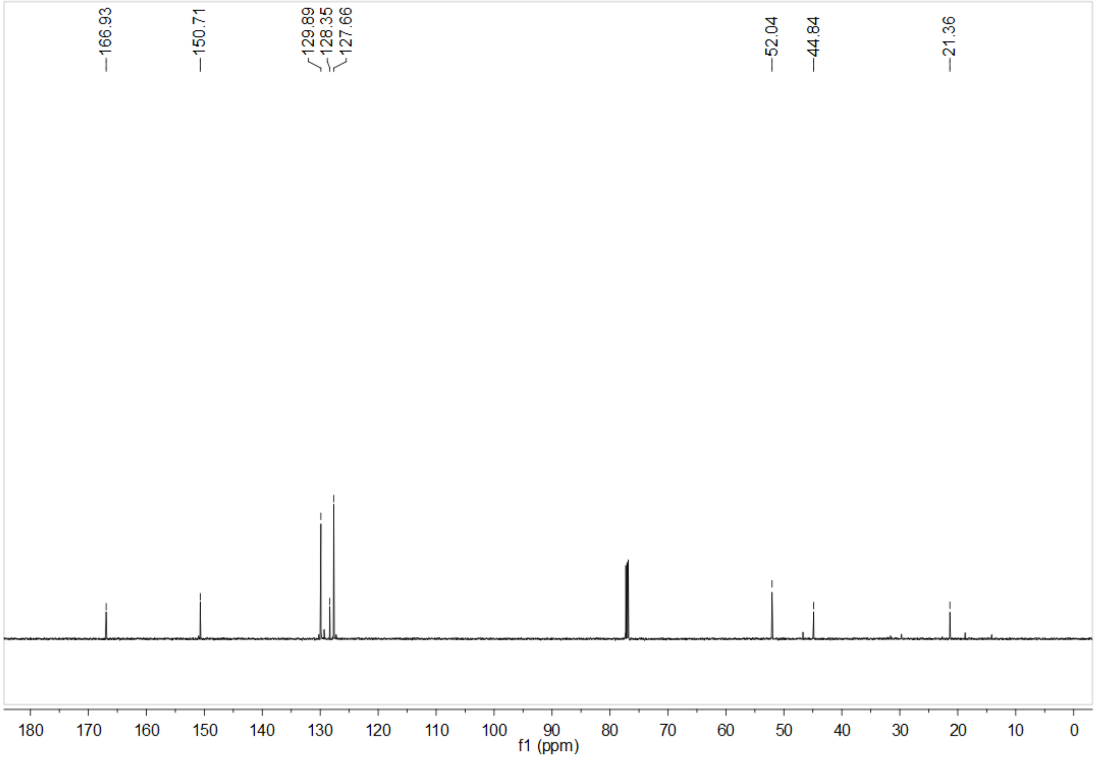
^{13}C NMR Spectrum of Compound 9a



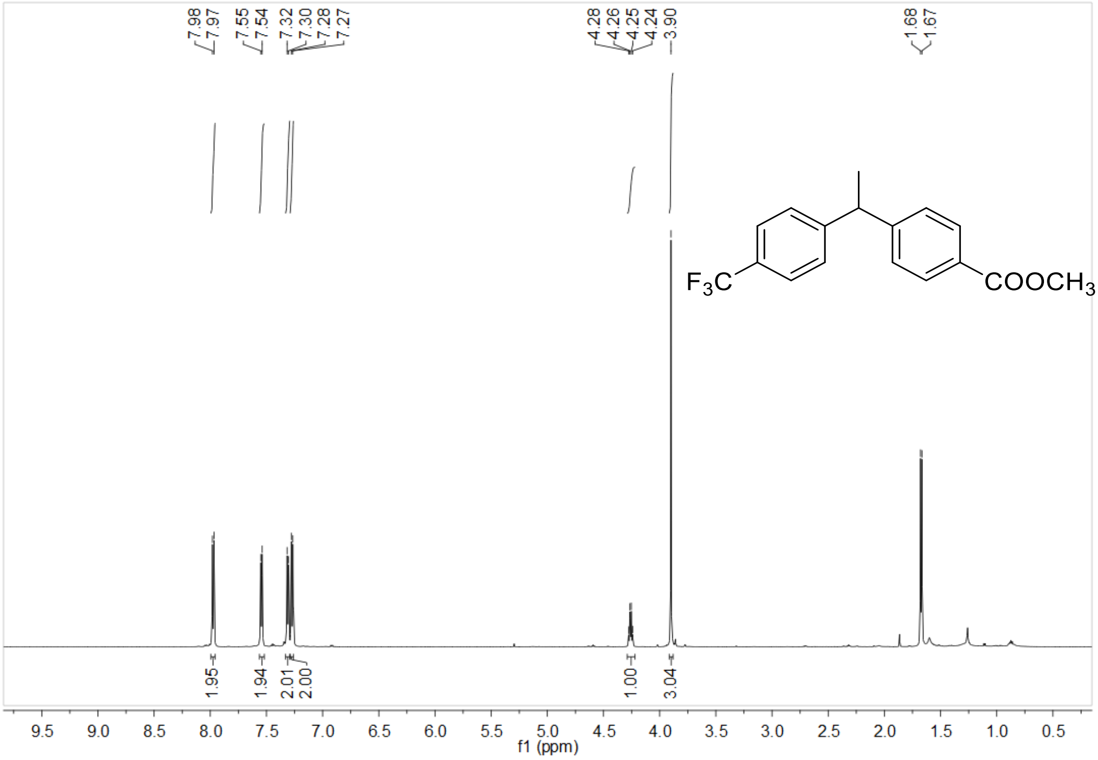
^{1}H NMR Spectrum of Compound 10a



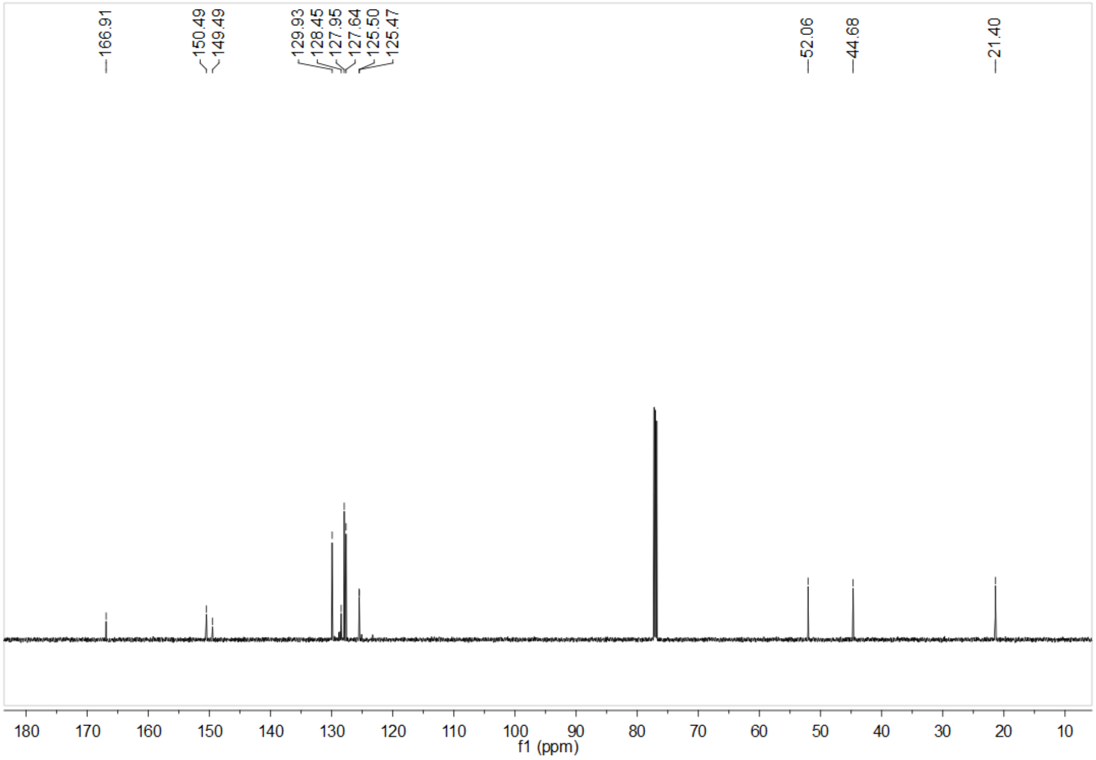
^{13}C NMR Spectrum of Compound 10a



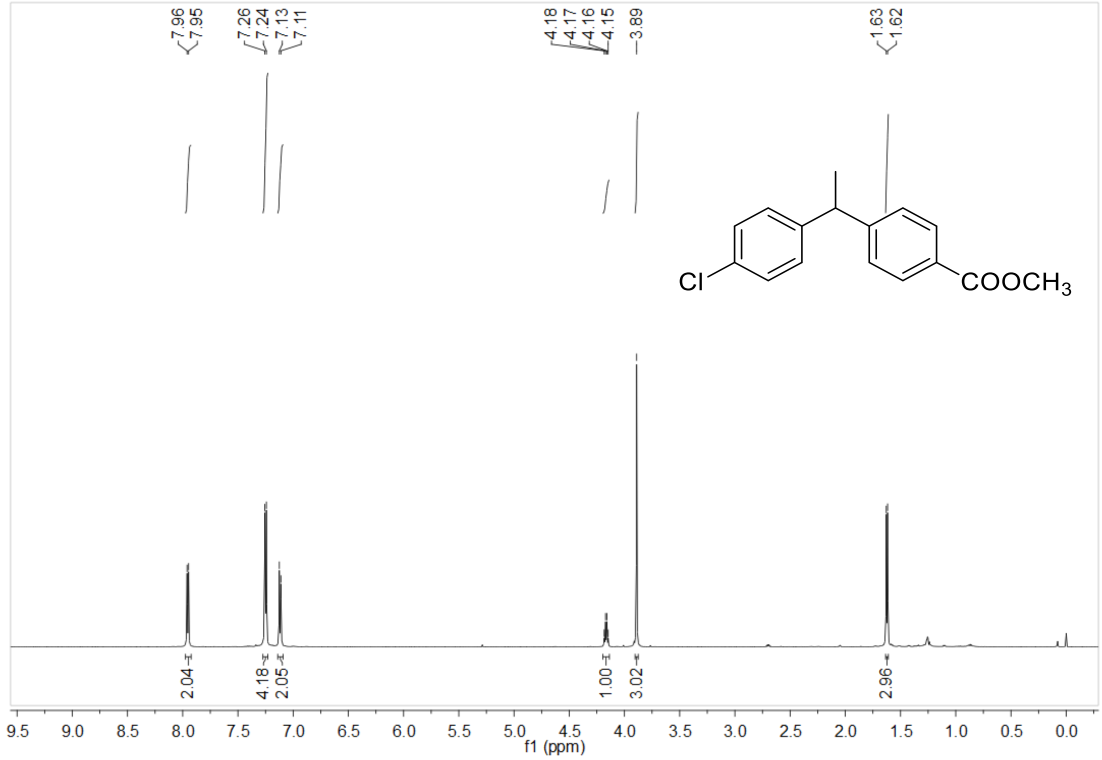
^{1}H NMR Spectrum of Compound 11a



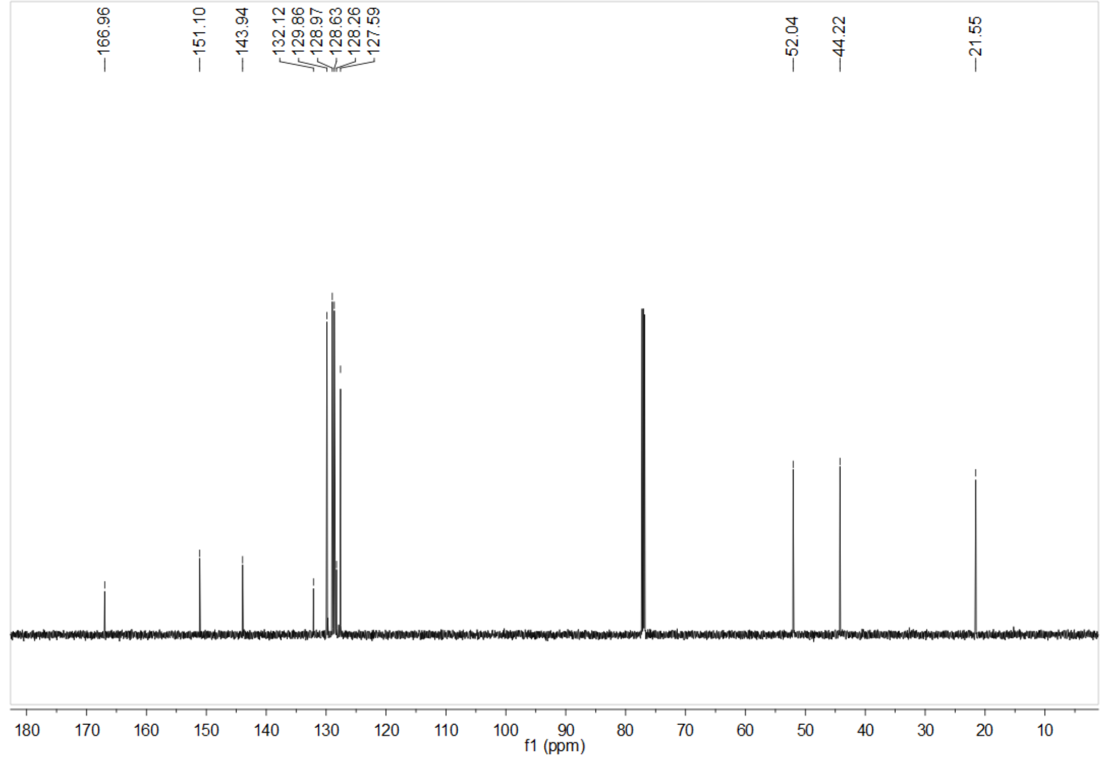
^{13}C NMR Spectrum of Compound 11a



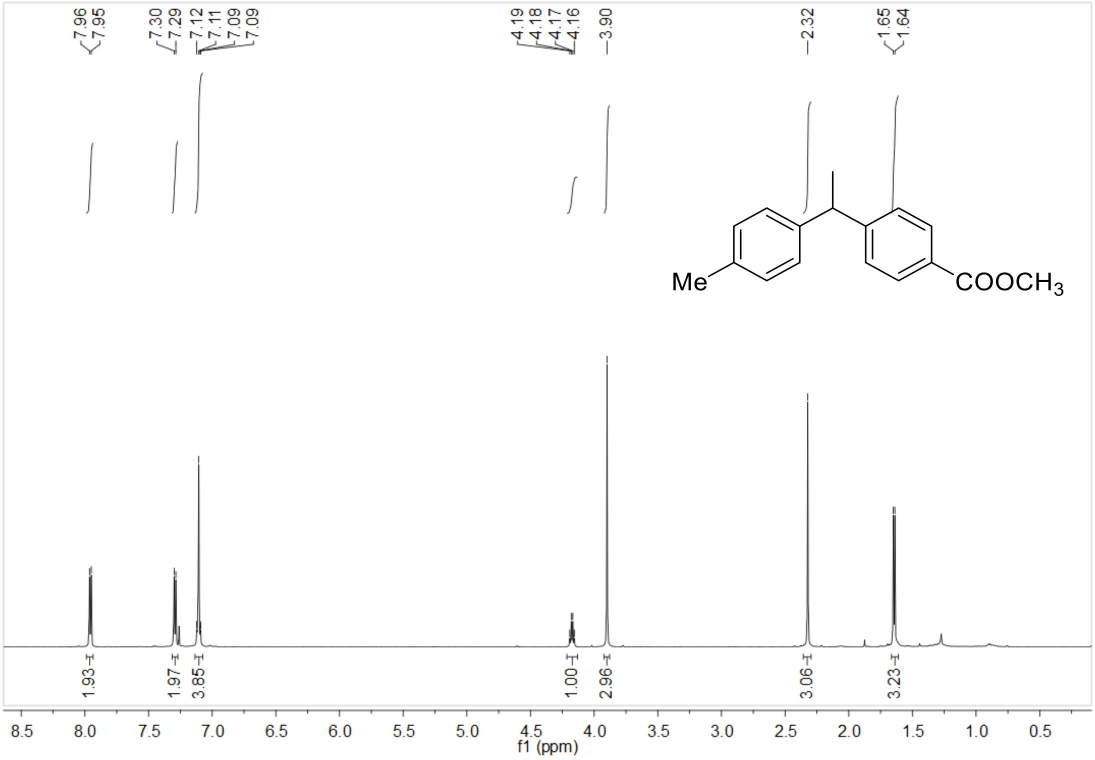
^{1}H NMR Spectrum of Compound 12a



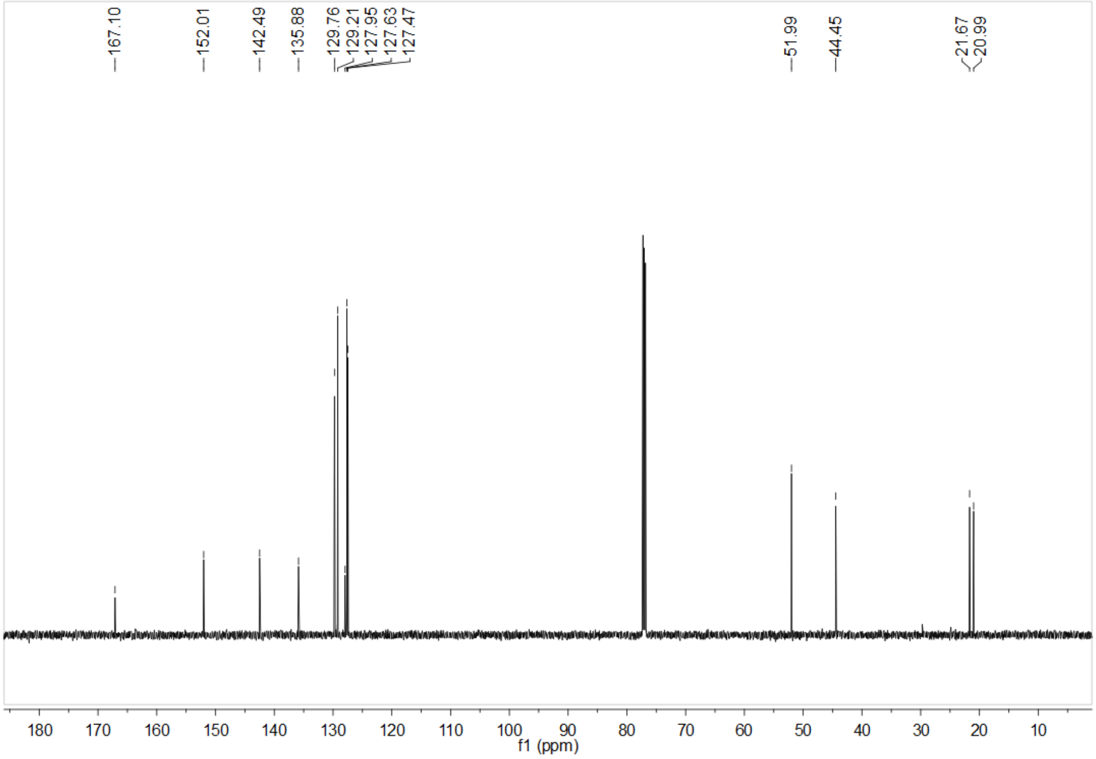
^{13}C NMR Spectrum of Compound 12a



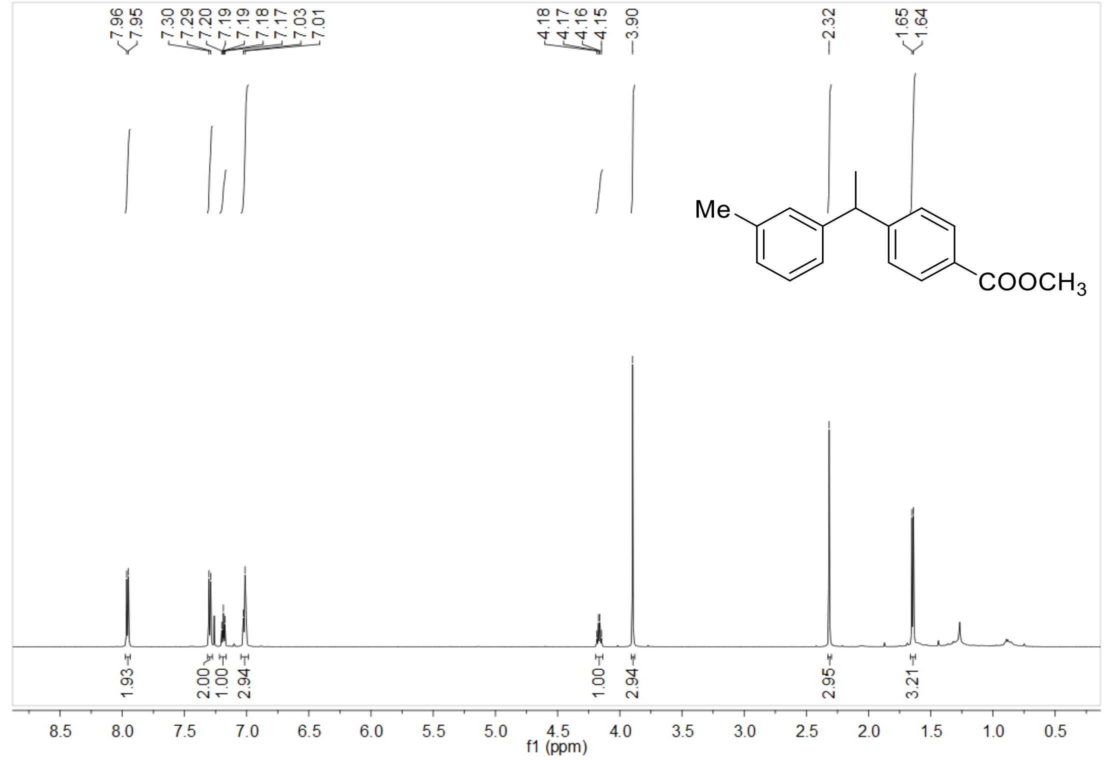
^{1}H NMR Spectrum of Compound 13a



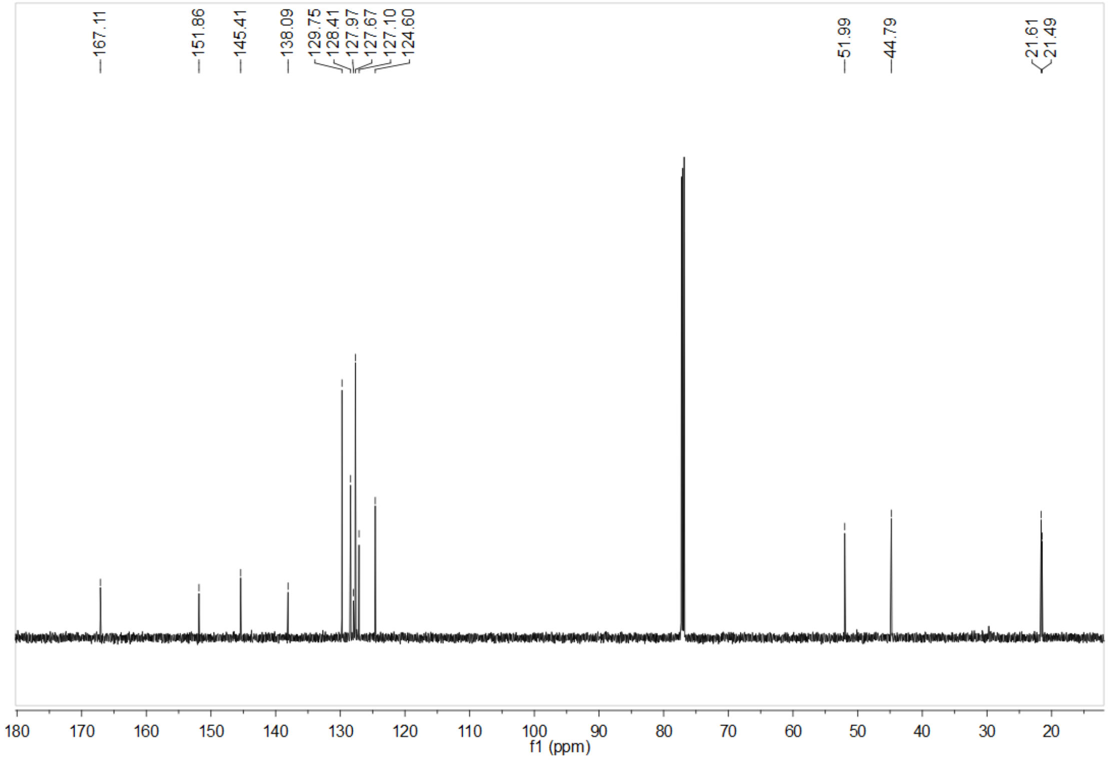
^{13}C NMR Spectrum of Compound 13a



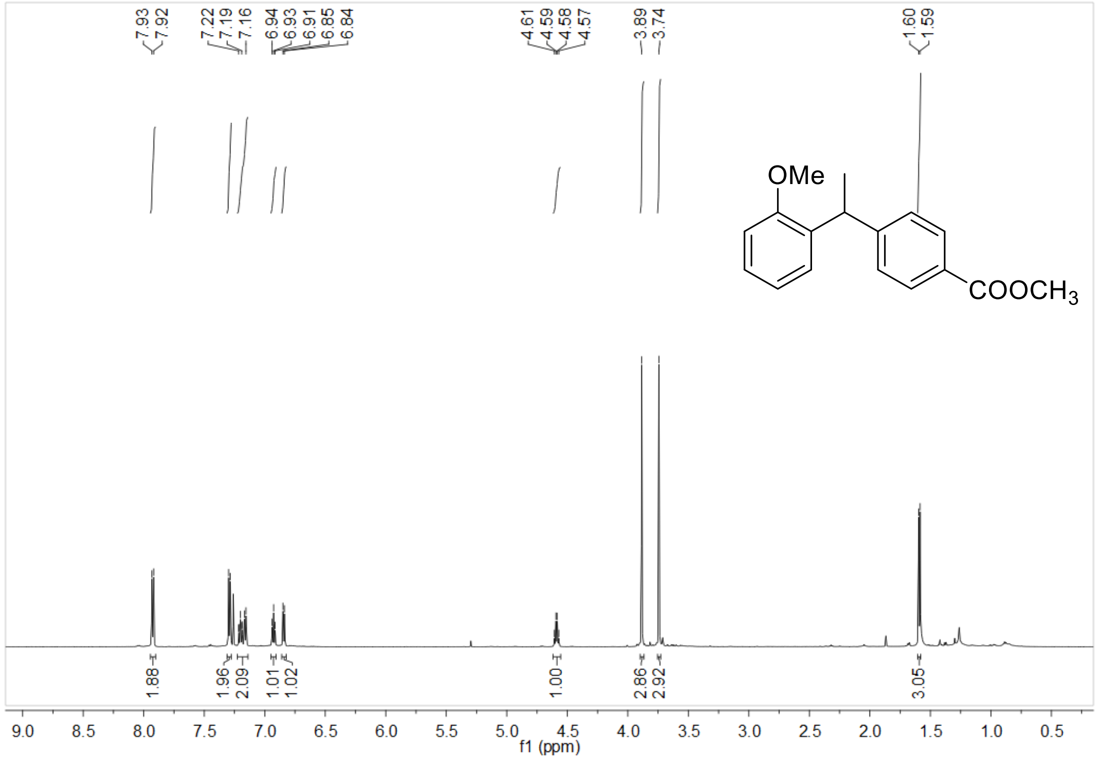
^{1}H NMR Spectrum of Compound 14a



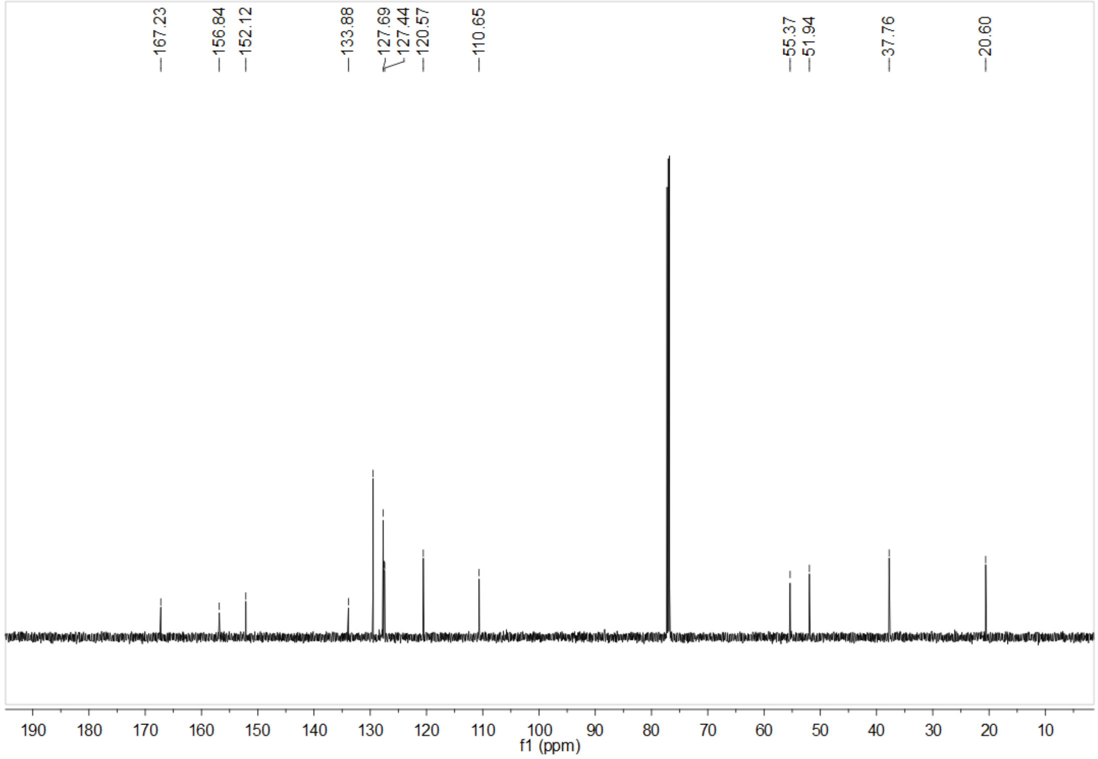
^{13}C NMR Spectrum of Compound 14a



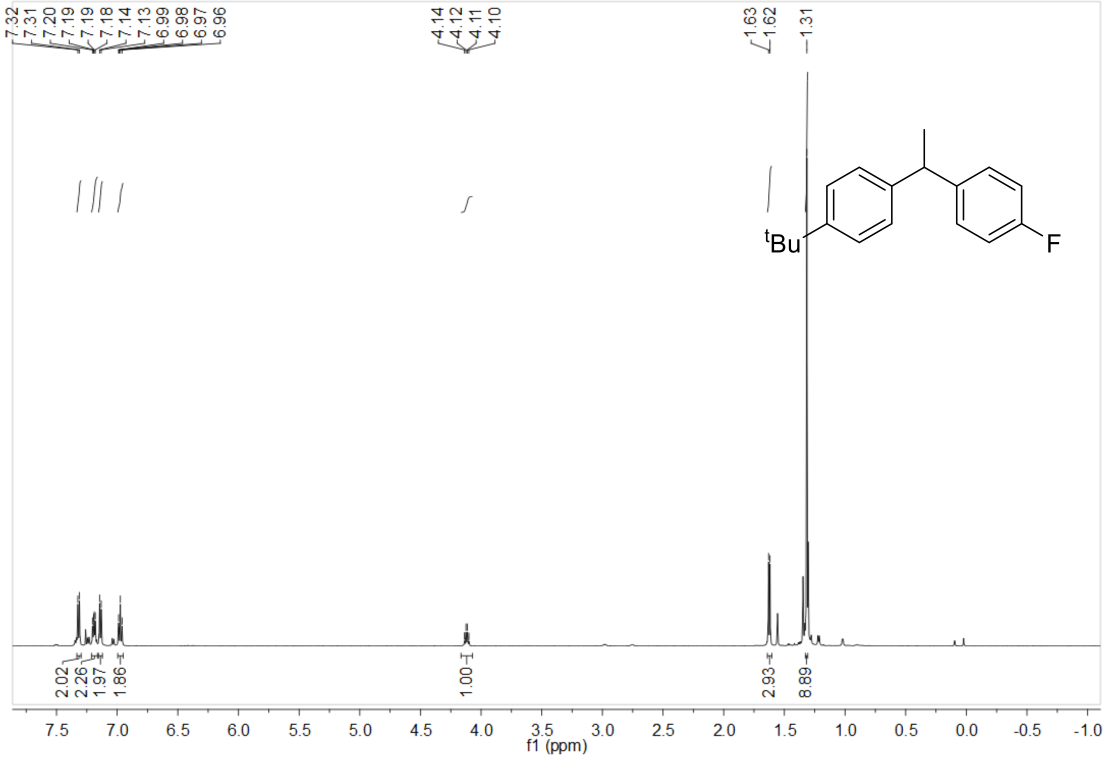
^{1}H NMR Spectrum of Compound 15a



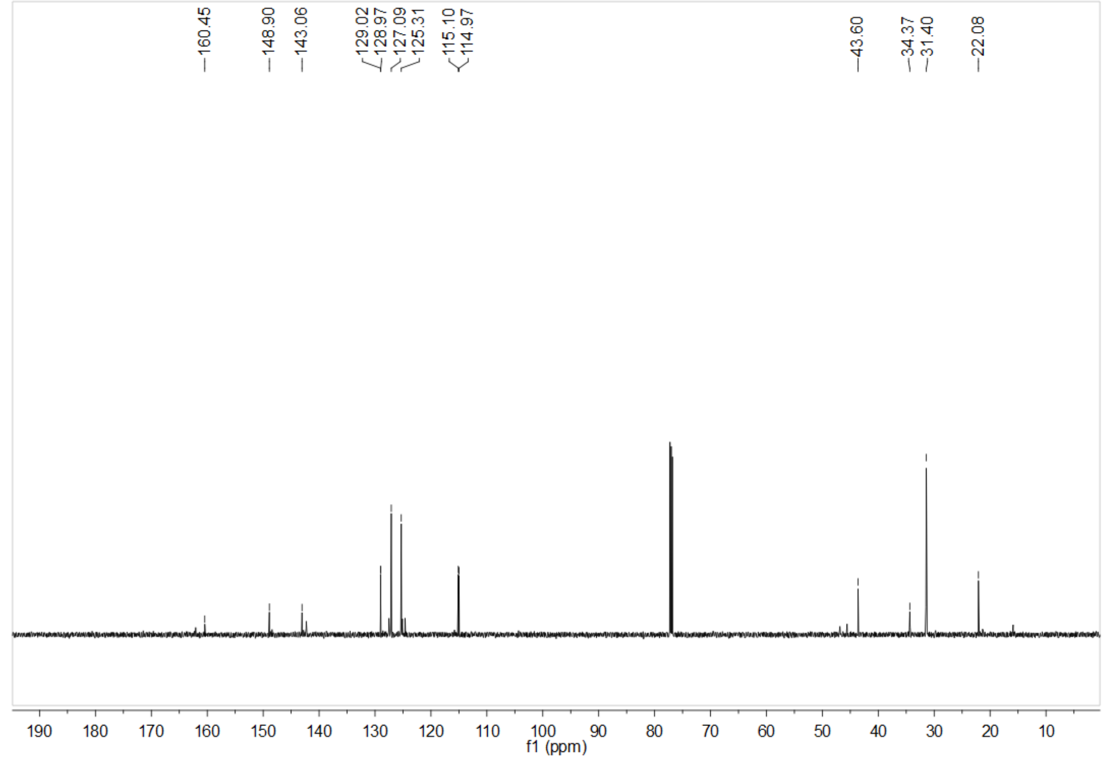
^{13}C NMR Spectrum of Compound 15a



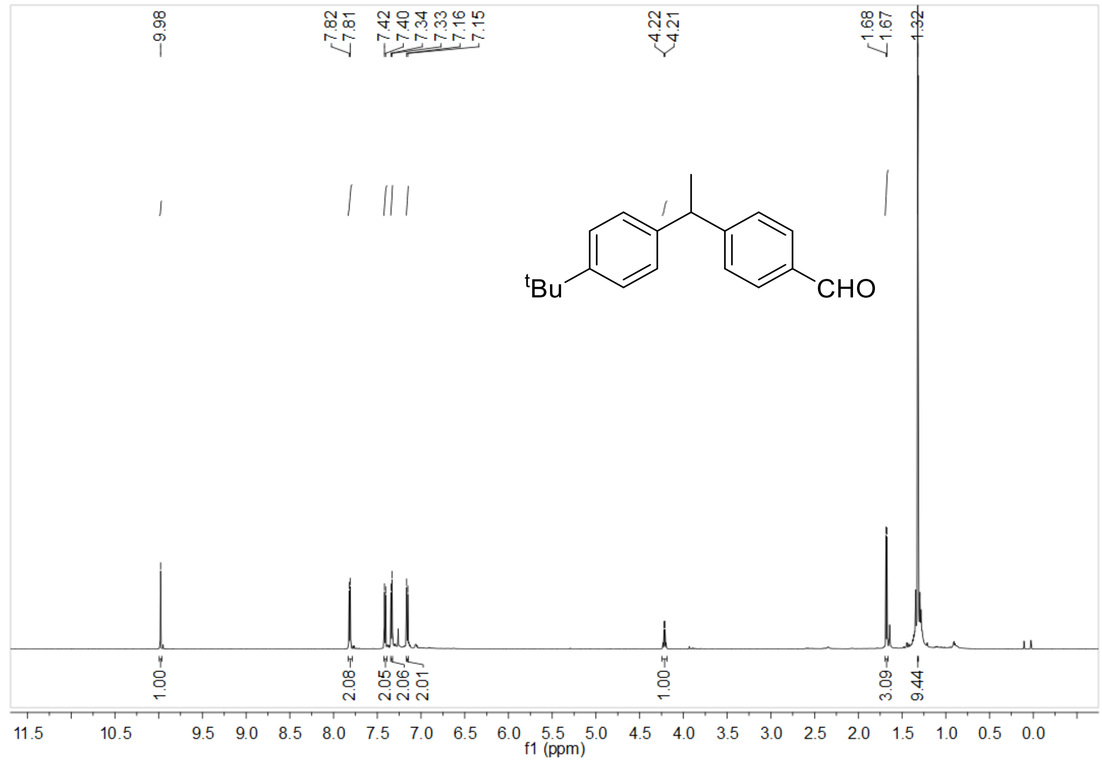
^{1}H NMR Spectrum of Compound 16a



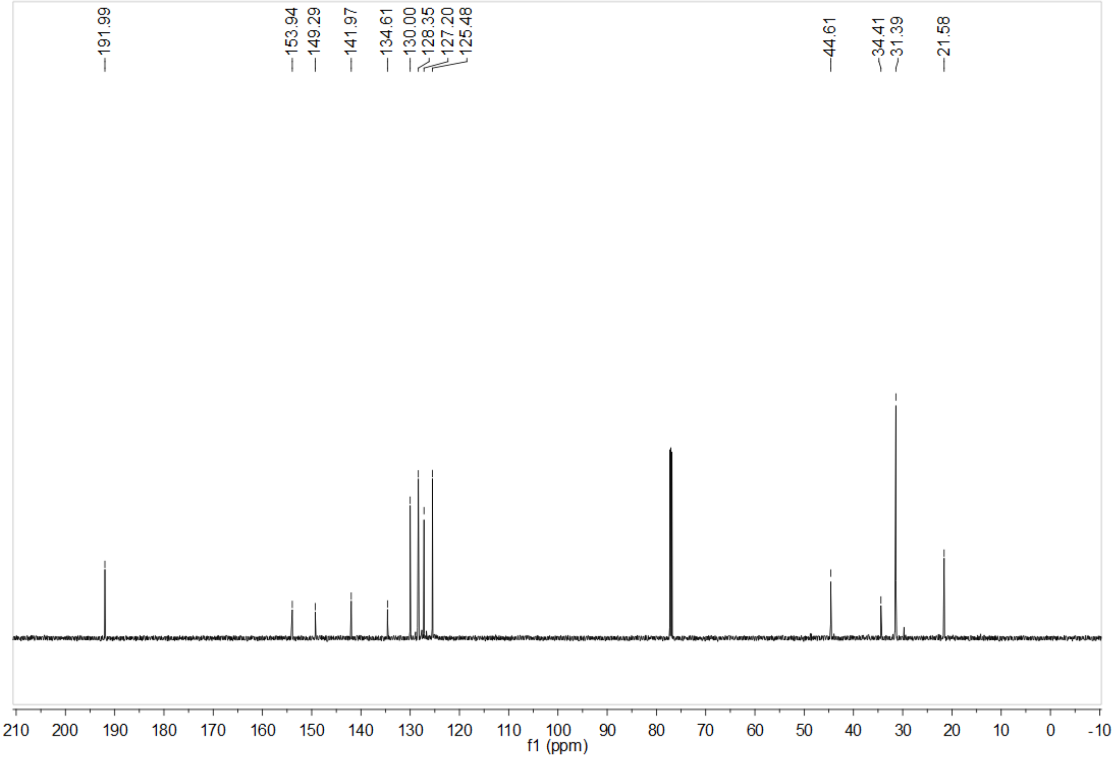
^{13}C NMR Spectrum of Compound 16a



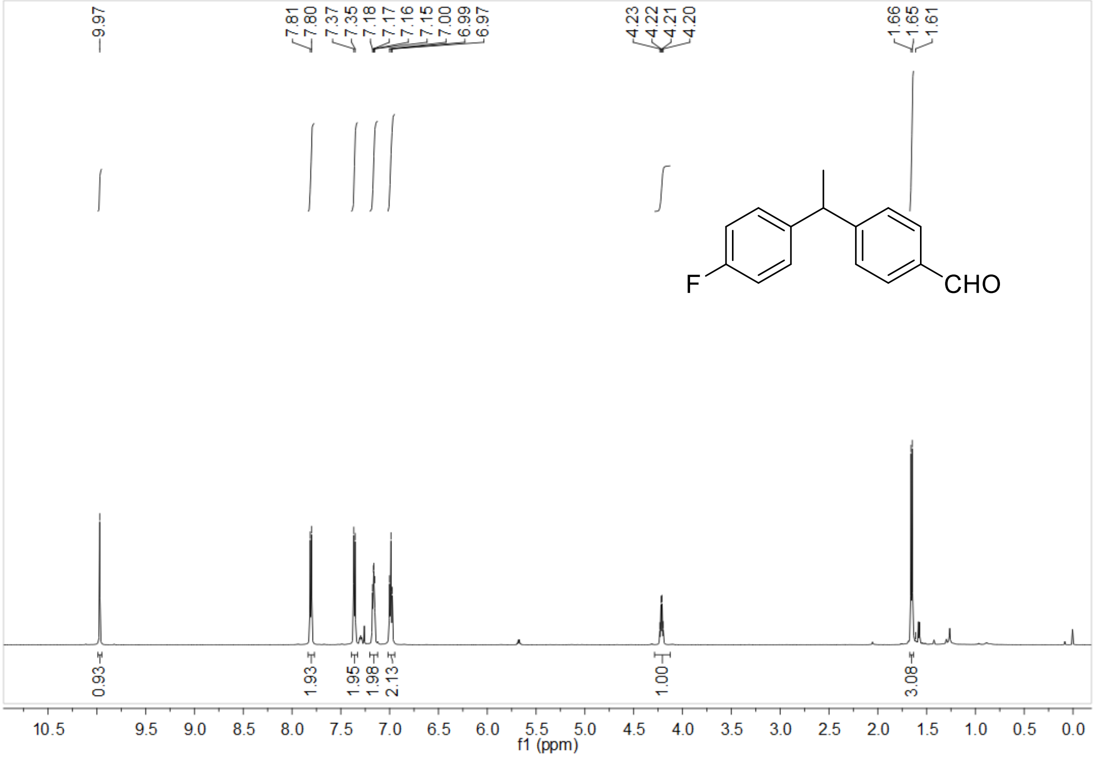
^{1}H NMR Spectrum of Compound 16b



^{13}C NMR Spectrum of Compound 16b



^{1}H NMR Spectrum of Compound 17a



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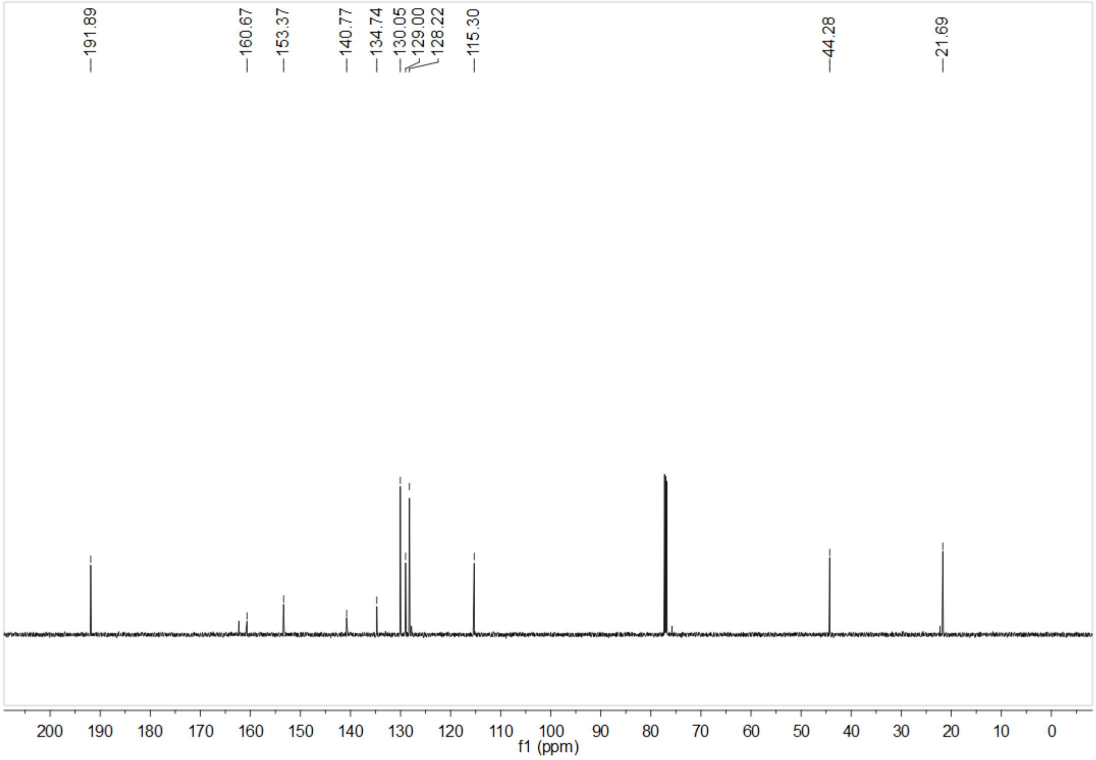
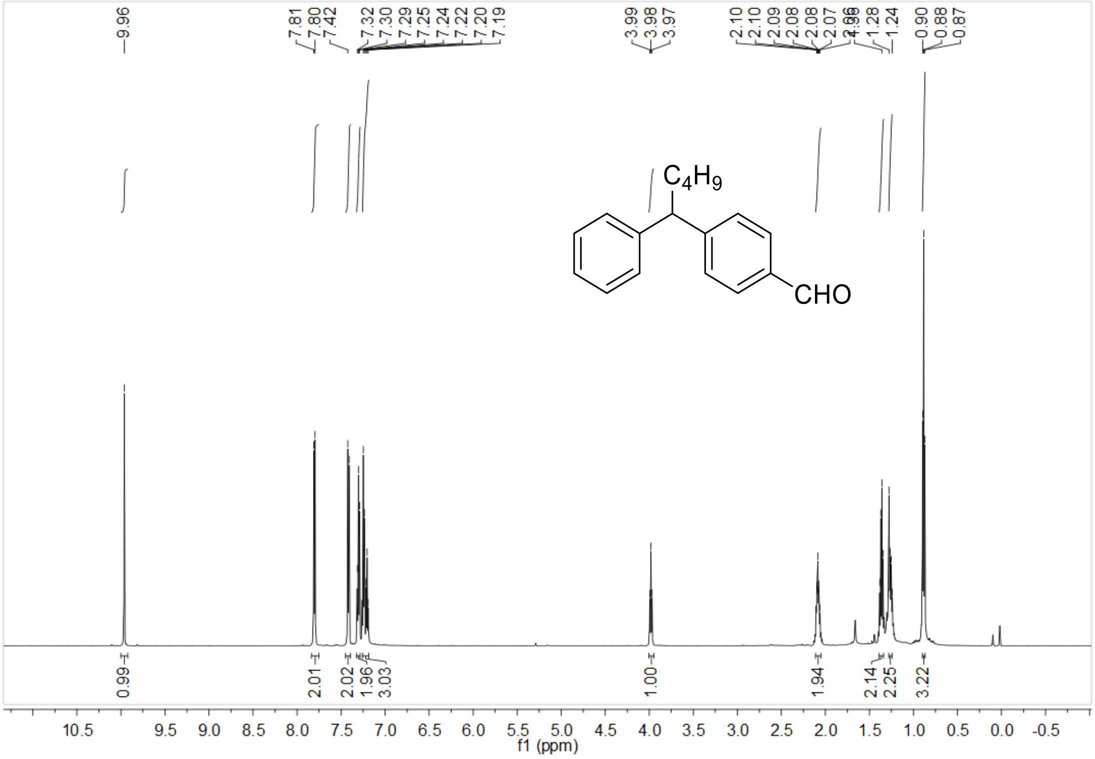


^{1}H NMR Spectrum of Compound 18a



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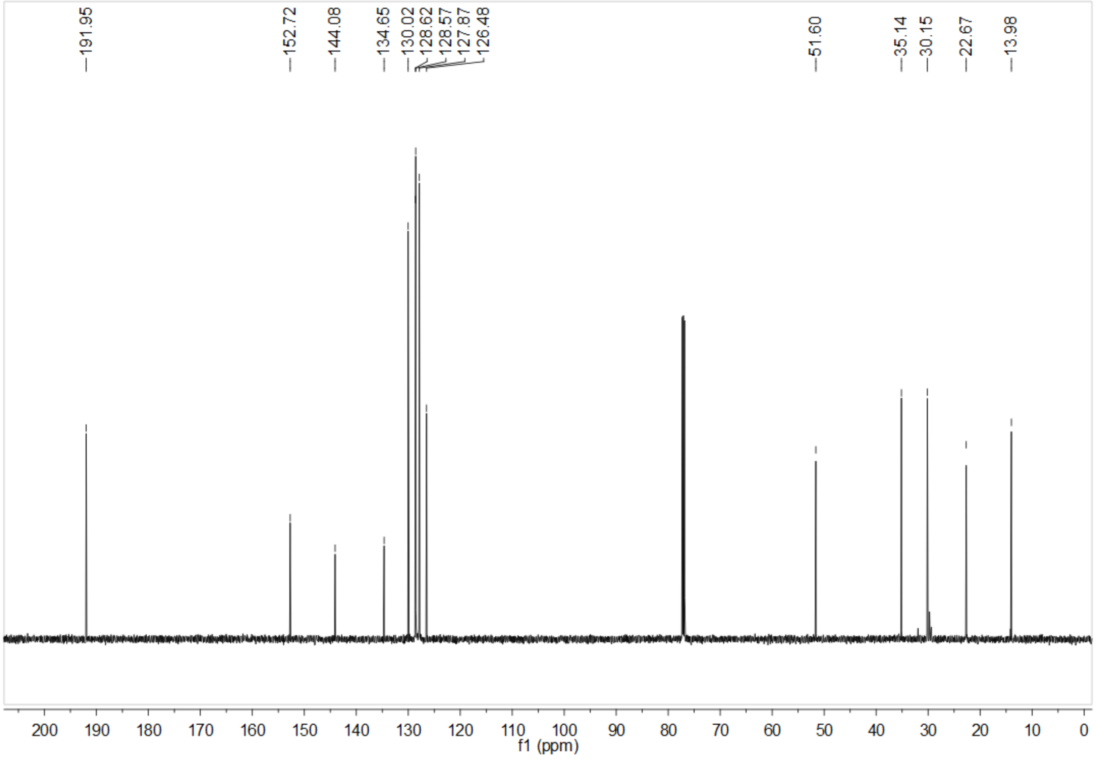

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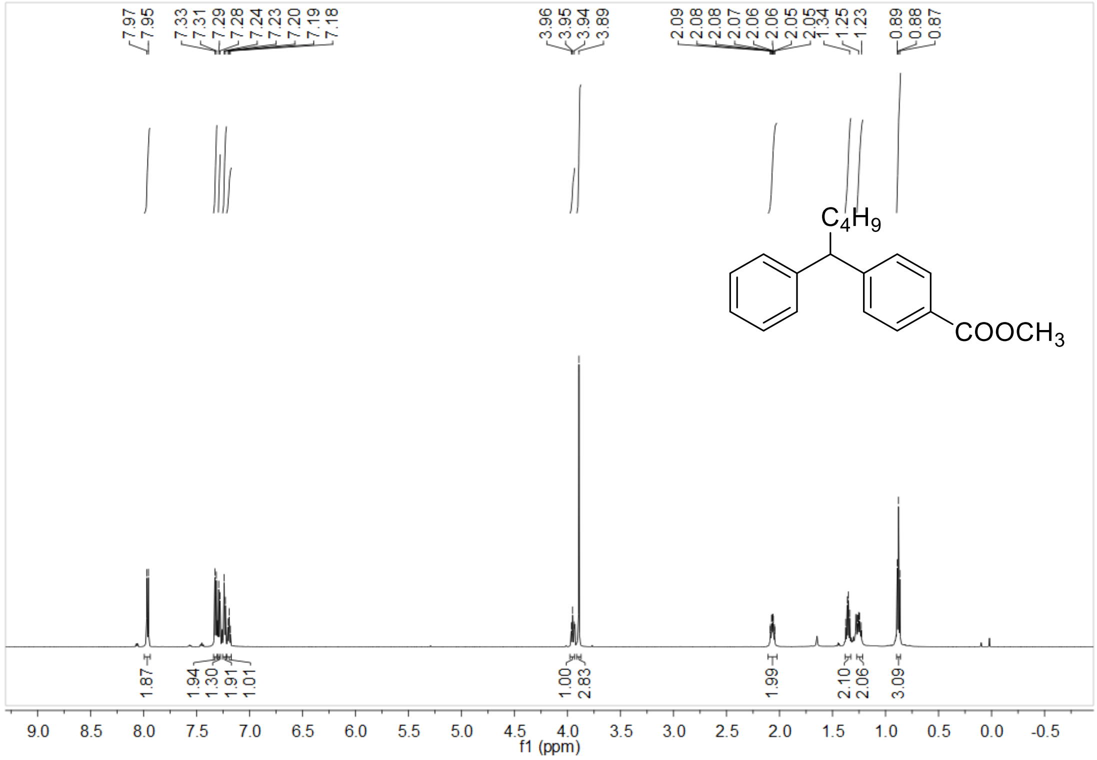
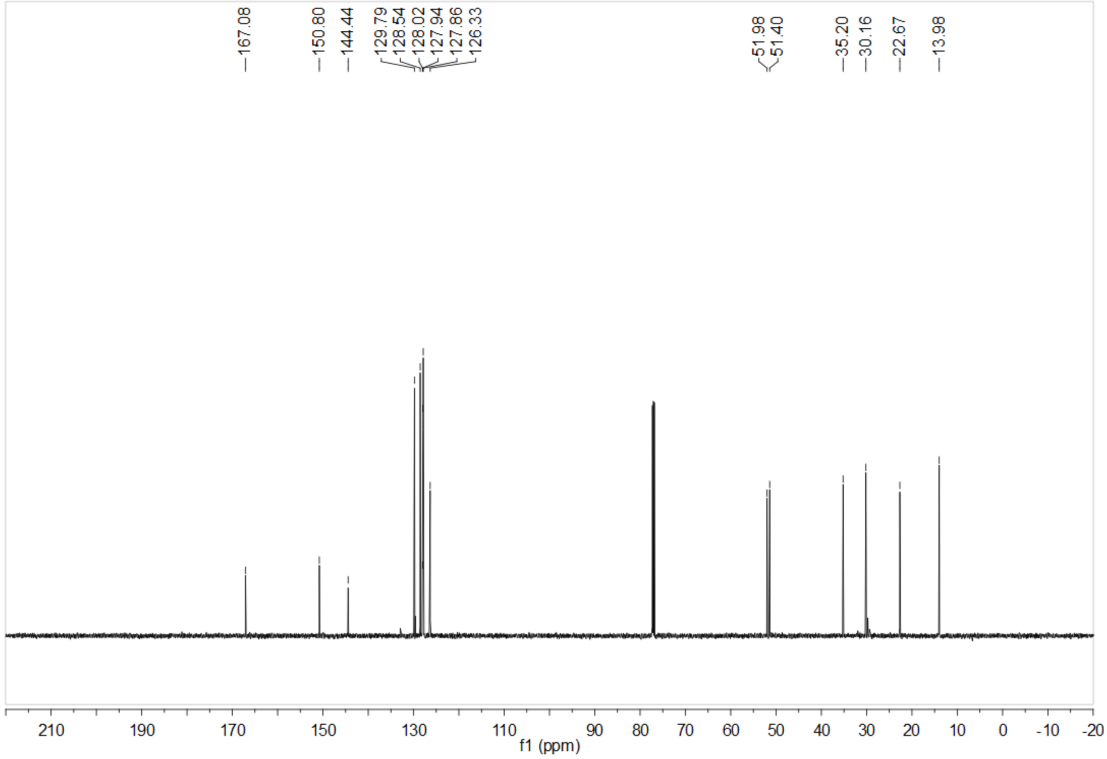
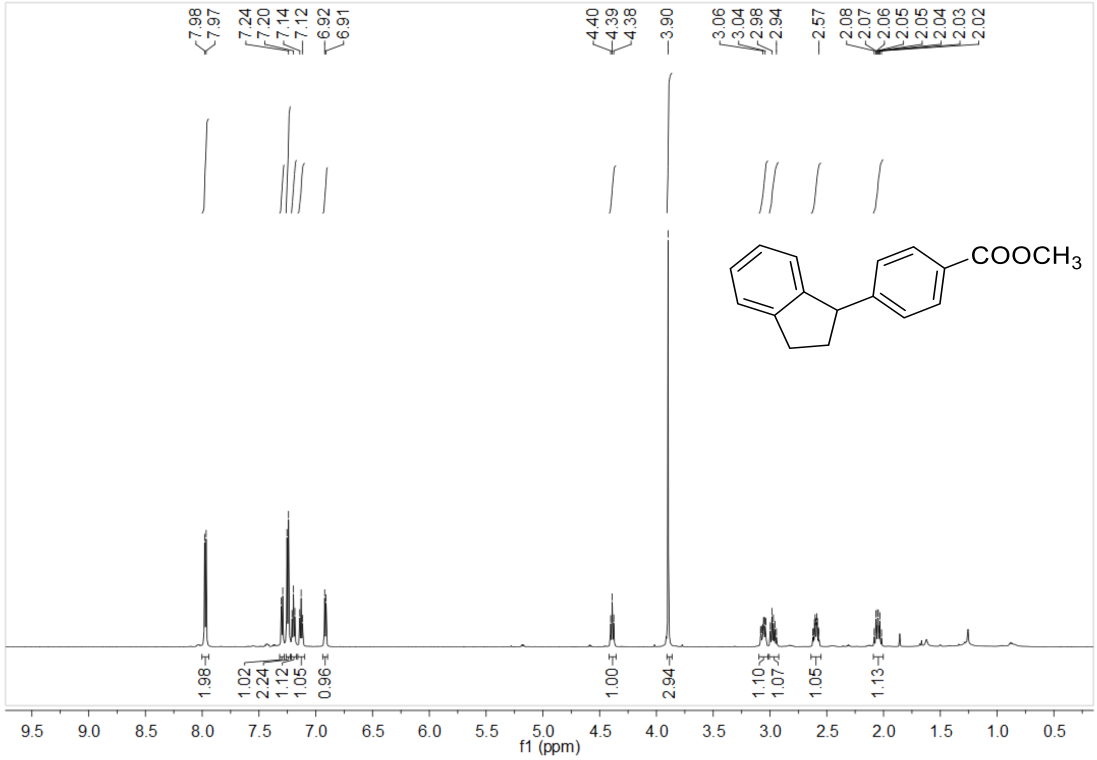


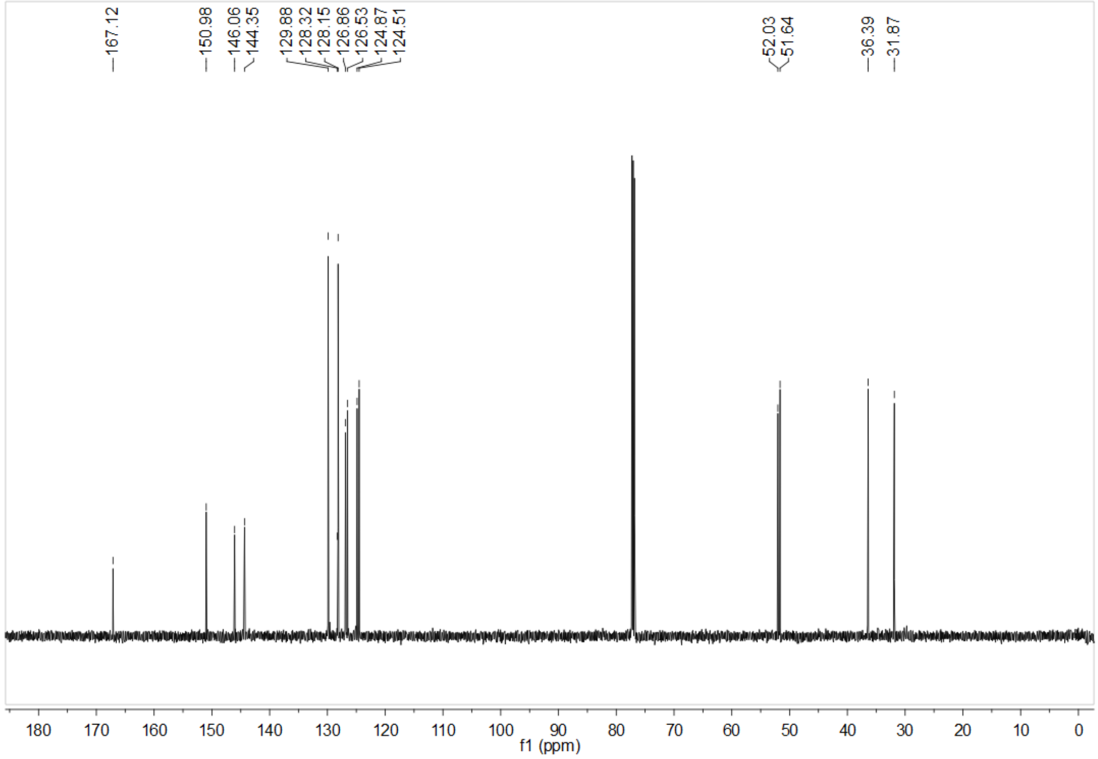
^{13}C NMR Spectrum of Compound 18b



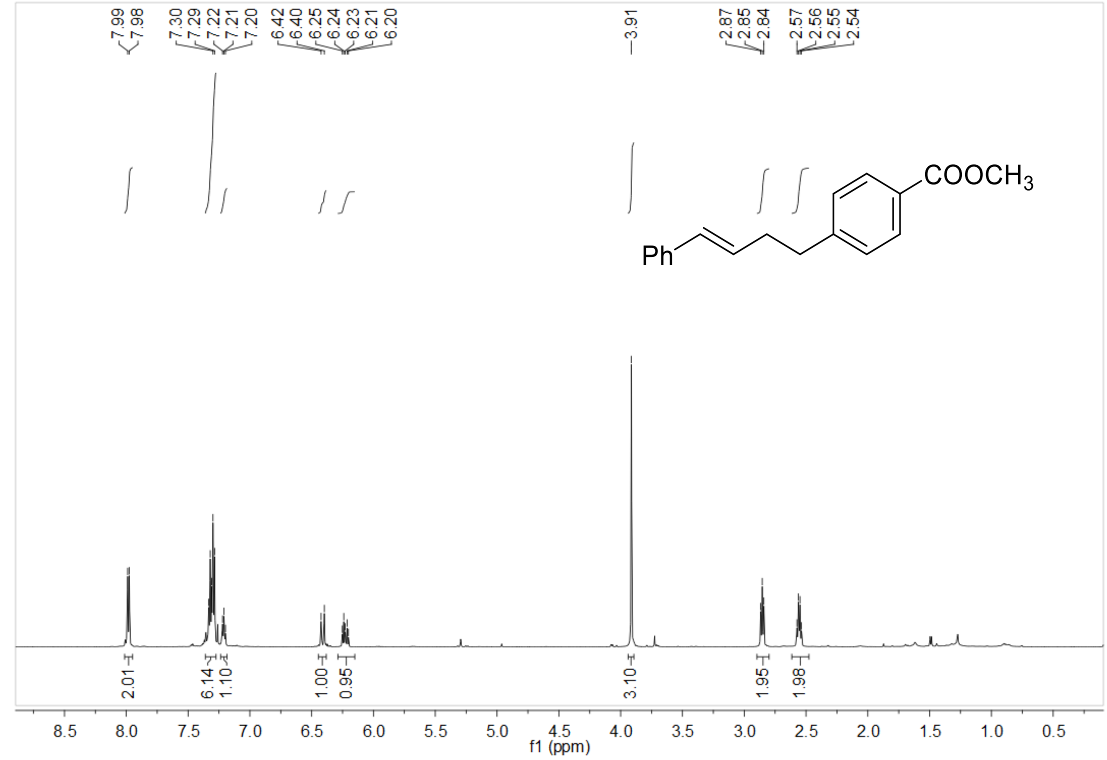
^{1}H NMR Spectrum of Compound 19a



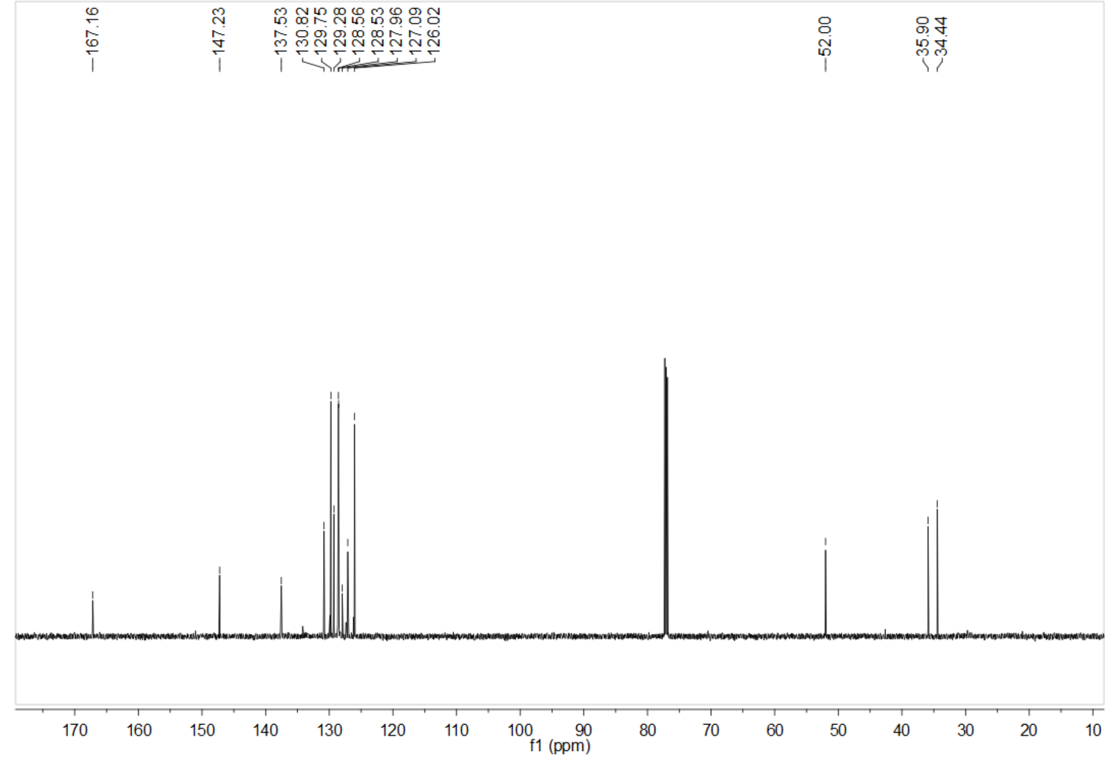
^{13}C NMR Spectrum of Compound 19a



^{1}H NMR Spectrum of Compound 21



^{13}C NMR Spectrum of Compound 21



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