Supporting Information

Electrochemical Radical-Polar Crossover Diesterification of Alkenes with Carboxylic Acids

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Content

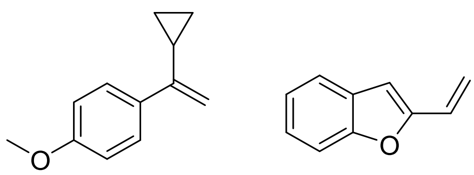
1. General methods

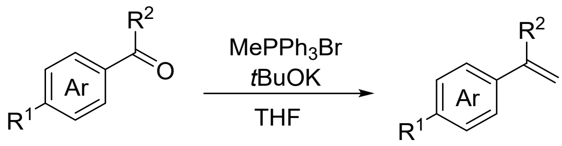
Unless otherwise noted, all reagents were purchased from commercial suppliers and used without further purification. Reactions were monitored by thin-layer chromatography (TLC) with Haiyang GF 254 silica gel plates (Qingdao Haiyang chemical industry Co Ltd, Qingdao, China) using UV light and vanillic aldehyde or phosphomolybdic acid as visualizing agents. Flash column chromatography was performed using 200-300 mesh silica gel at increased pressure. ^{1}H NMR spectra, ^{19}F NMR spectra and ^{13}C NMR spectra were respectively recorded on 600 MHz, 565 MHz, 400 MHz, 151 MHz and 101 MHz NMR spectrometers. Chemical shifts (δ) were expressed in ppm with TMS as the internal standard, and coupling constants (*J*) were reported in Hz. High-resolution mass spectra were obtained by using ESI ionization sources (quadrupole time-of-flight mass spectrometer, Bruker Impact II, Bremen, Germany). Electron paramagnetic resonance (EPR) spectra were recorded on a Bruker EMXplus-9.5/12 spectrometer. Cyclic voltammograms were obtained on a CHI 700E potentiostat (CH Instruments, Inc.).

Abbreviations: DMF = *N*, *N*-dimethylformamide, DMSO = dimethyl sulfoxide, DCE = dichloroethane, DCM = dichloromethane, MeCN = acetonitrile, THF = tetrahydrofuran, TFE = 2,2,2-Trifluoroethanol, DMPO = 3,4-dihydro-2,2-dimethyl-2*H*-pyrrole 1-oxide, dr = diastereomeric ratio.

2. Experimental procedures

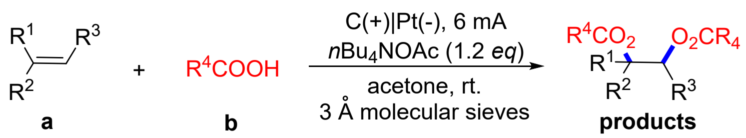
2.1. General procedure for the preparation of the following substrates





General Wittig reaction procedure for alkene synthesis: To a 50 mL flask equipped with a stirring bar was added *t*BuOK (1.12 g, 10 mmol, 2.0 *eq.*), $MePPh_3Br$ (3.57 g, 10 mmol, 2.0 eq.) and THF (25 mL) at room temperature. After stirring for 0.5 h, the mixture was added drop by drop to a 50 mL flask containing a stirring bar, aldehyde or ketone (5 mmol, 1.0 *eq.*) and THF (5 mL). The reaction mixture was stirred at room temperature. Upon completion (monitored by TLC), concentrated in vacuo, diluted with water, and extracted with ethyl acetate. The combined organic layers were dried over anhydrous Na_2SO_4 and evaporated in vacuo. The residue was purified by flash chromatography on silica gel using petroleum ether/ethyl acetate as the eluent to give desired product.

2.2. General procedure for the electrochemical synthesis of 1,2-diester derivatives



To an undivided beaker-type electrolysis cell (10 mL) equipped with a magnetic stirring bar was added **a** (0.2 mmol, 1 *equiv*), **b** (0.7 mmol, 3.5 *equiv*), $n\mathrm{Bu_4NOAc}$ (0.24 mmol, 1.2 *equiv*), 150 mg 3Å molecular sieve powder and acetone (6 mL, dried over 3Å molecular sieve pellets, Φ = 3−5 mm). A carbon rod electrode (Φ = 0.5 cm) was used as the anode and a platinum plate (1 cm x 1 cm x 0.2 mm) was used as the cathode (the electrodes were immersed 1 cm in the reaction mixture). The reaction mixture was stirred and electrolyzed at a constant current of 6 mA at room temperature open to air. After reaction completion (monitored by TLC), the reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate as the eluent to obtain the target product.

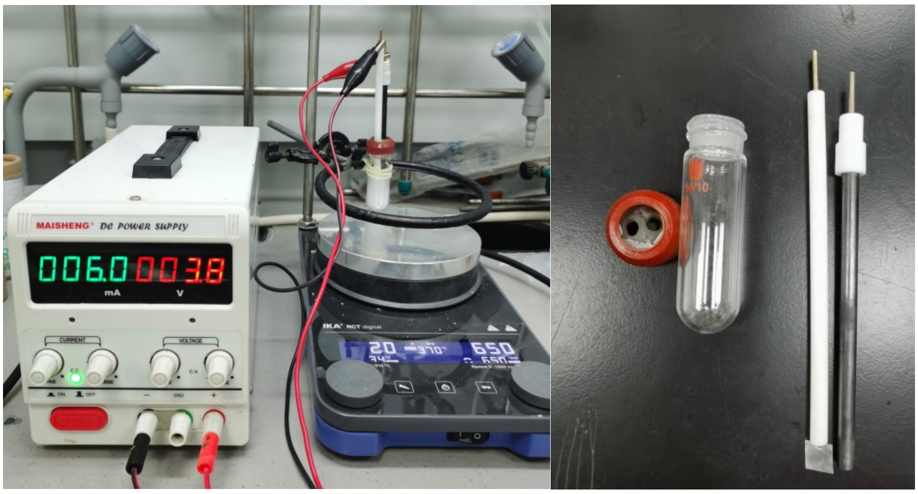
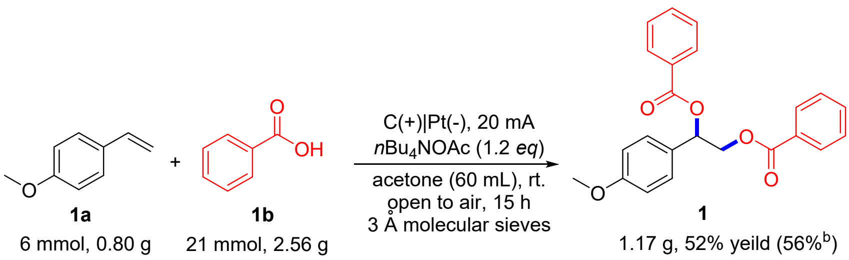


Figure S1 Electrochemical setup used.

The experimental setup consisted of a carbon rod electrode (Φ = 0.5 cm) and a platinum plate (1 cm x 1 cm x 0.2 mm), a tube (10 mL) with perforated rubber plugs, an adjustable DC regulated power supply (MS-150V 100 mA), and a magnetic stirrer.

2.3. Gram-scale experiment for the synthesis of 1



[a] Reaction conditions: A mixture of **1a** (6 mmol, 1 *equiv*), **1b** (21 mmol, 3.5 *equiv*), nBu$_4$NOAc (7.2 mmol, 1.2 *equiv*) and 1.5 g 3Å molecular sieve powder in acetone (60 mL, dried over 3 Å molecular sieve pellets, Φ = 3−5 mm) under a constant current of 20 mA (anode: three carbon rod electrodes, Φ = 0.5 cm each; cathode: Pt plate, 3 cm x 3 cm x 0.2 mm) in an undivided cell at RT open to air. [b] Current efficiency.

Substrate **1a** (6 mmol, 1 *equiv*), **1b** (21 mmol, 3.5 *equiv*), nBu$_4$NOAc (7.2 mmol, 1.2 *equiv*), 1.5 g 3Å molecular sieve powder and acetone (60 mL, dried over 3Å molecular sieve pellets, Φ = 3−5 mm) were added to an undivided beaker-type electrolysis cell (100 mL) equipped with a magnetic stir bar. Three carbon rod electrodes (Φ = 0.5 cm each, 0.5 cm apart from each other) were used as the anode and a platinum plate (3 cm x 3 cm x 0.2 mm) was used as cathode (the electrodes were immersed 3 cm in the reaction solution). The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature open to air. After reaction completion (monitored by TLC), the reaction mixture was filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (20:1, v/v) as the eluent to obtain the target product **1** (1.17 g, 52% yield, 56% current efficiency).

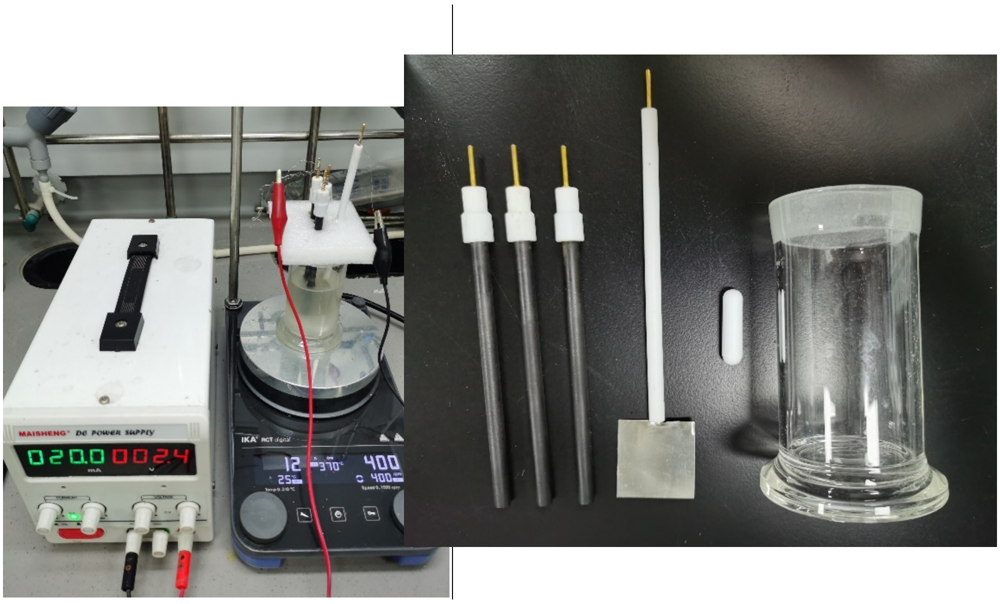
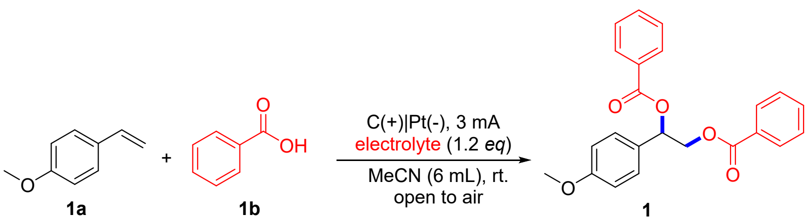


Figure S2 Electrochemical setup used for gram-scale experiment.

The experimental setup for gram-scale experiment consisted of three carbon rod electrodes (Φ = 0.5 cm each) and a platinum plate (3 cm x 3cm x 0.2 mm), a beaker-type electrolysis cell (100 mL), an adjustable DC regulated power supply (MS-150V 100 mA), and a magnetic stirrer.

3. Optimization of reaction conditions

Table S1. Electrolyte screening [a]

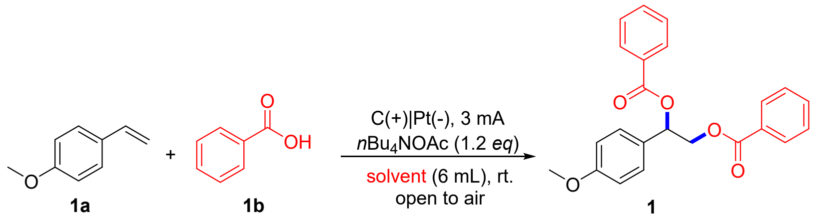


Entry	Electrolyte	Yield (%) [b]	Time (h)
1	--	Voltage overload	0.5
2	*n*Bu_4NF	15	5
3	*n*Bu_4NBr	N.D.	5

4	*n*Bu$_4$NI	N.D.	5
5	*n*Bu$_4$NOAc	35	5
6	*n*Bu$_4$NPF$_6$	Trace	5
7	*n*Bu$_4$NBF$_4$	N.D.	5
8	*n*Bu$_4$NClO$_4$	N.D.	5
9	KClO$_4$	N.D.	5
10	NaBF$_4$	Trace	5

[a] Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (0.7 mmol, 3.5 *equiv*) and electrolyte (0.24 mmol, 1.2 *equiv*) in MeCN (6 mL) under a constant current of 3 mA (C anode: Φ = 0.5 cm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT open to air. [b] Isolated yield.

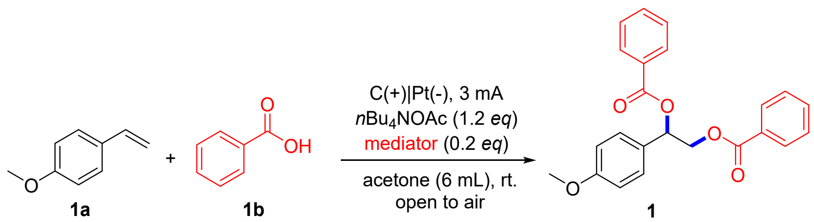
Table S2. Solvent screening [a]



Entry	Solvent	Yield (%) [b]	Time (h)
1	MeCN	35	5
2	DCM	38	4
3	DMF	N.D.	5
4	THF	N.D.	5
5	DMSO	35	5
6	MeOH	Trace	5
7	DCE	Trace	5
8	TFE	Trace	5
9	Acetone	42	5
10	Cyclohexane	Voltage overload	0.5
11	EA	Voltage overload	0.5
12	CH_3NO_2	10	5

[a] Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (0.7 mmol, 3.5 *equiv*) and *n*Bu$_4$NOAc (0.24 mmol, 1.2 *equiv*) in a solvent (6 mL) under a constant current of 3 mA (C anode: Φ = 0.5 cm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT open to air. [b] Isolated yield.

Table S3. Mediator screening [a]

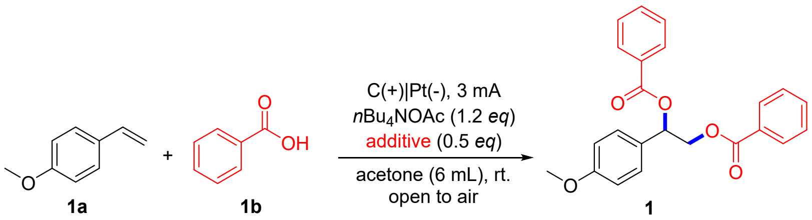


Entry	Mediator	Yield (%)[b]	Time (h)
1	--	42	5

2	2,2,6,6-Tetramethylpiperidinooxy	41	10
3	Ferrocene	N.D.	10
4	N,N,N-Triphenylamine	Trace	10
5	9-Azabicyclo[3.3.1]nonane N-oxyl	35	10
6	1,4-Diaza[2.2.2]bicyclooctane	40	10

[a] Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (0.7 mmol, 3.5 *equiv*), *n*Bu_4NOAc (0.24 mmol, 1.2 *equiv*) and mediator (0.04 mmol, 0.2 *equiv*) in acetone (6 mL) under a constant current of 3 mA (C anode: Φ = 0.5 cm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT open to air. [b] Isolated yield.

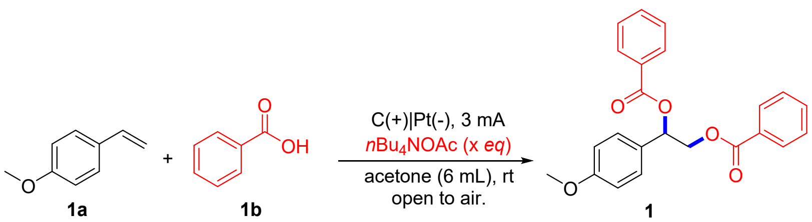
Table S4. Additive screening [a]



Entry	Additive	Yield (%) [b]	Time (h)
1	--	42	5
2	K_2CO_3	41	5
3	KOH	27	5
4	NaOH	42	5
5	$NaHCO_3$	40	5
6	NaH_2PO_4	42	5
7	CsF	27	5
8	1,4-Diaza[2.2.2]bicyclooctane	N.D.	5
9	1,4-Diaza[2.2.2]bicyclooctane	39	5
10	N,N,N-Triethylamine	Trace	5
11	2,6-Lutidine	Trace	5

[a] Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (0.7 mmol, 3.5 *equiv*), *n*Bu_4NOAc (0.24 mmol, 1.2 *equiv*) and additive (0.1 mmol, 0.5 *equiv*) in acetone (6 mL) under a constant current of 3 mA (C anode: Φ = 0.5 cm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT open to air. [b] Isolated yield.

Table S5. Screening of the amount of *n*Bu_4NOAc [a]

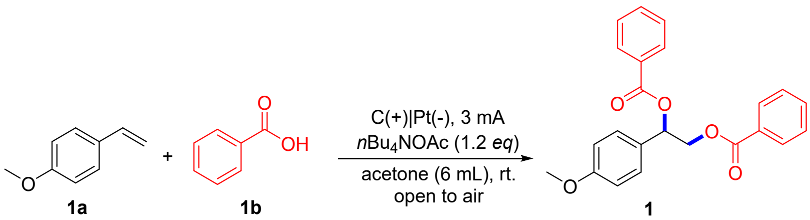


Entry	Amount of *n*Bu_4NOAc (*equiv*)	Yield (%) [b]	Time (h)
1	0.2	20	5
2	0.5	26	5
3	0.8	27.	5

4	1.0	38	5
5	1.2	42	5
6	1.5	27	5
7	2.0	27	5
8	3.0	12.	5

[a] Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (0.7 mmol, 3.5 *equiv*) and *n*Bu_4NOAc (x *equiv*) in acetone (6 mL) under a constant current of 3 mA (C anode: Φ = 0.5 cm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT open to air. [b] Isolated yield.

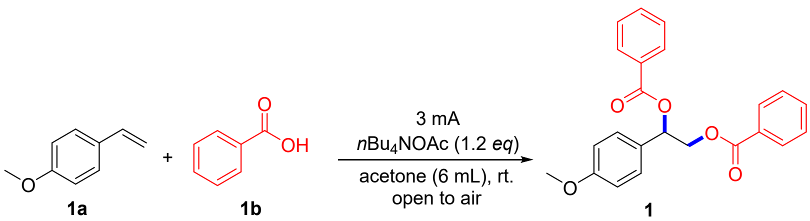
Table S6. Screening of the amount of benzoic acid (**1b**) [a]



Entry	Amount of **1b** (*equiv*)	Yield (%) [b]	Time (h)
1	2.5	37	5
2	3.0	37	5
3	3.5	42	5
4	4.0	42	5
5	4.5	39	5
6	5.0	35	5
7	7.0	32	5

[a] Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (x *equiv*) and *n*Bu_4NOAc (0.24 mmol, 1.2 *equiv*) in acetone (6 mL) under a constant current of 3 mA (C anode: Φ = 0.5 cm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT open to air. [b] Isolated yield.

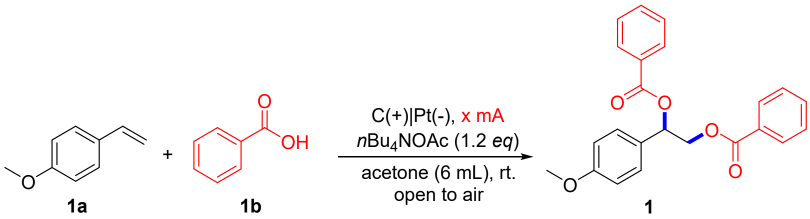
Table S7. Electrode material screening [a]



Entry	Electrode material	Yield (%) [b]	Time (h)
1	C(+) | Pt(-)	42	5
2	C(+) | C(-)	32	7
3	Pt(+) | Pt(-)	Trace	7
4	Pt(+) | C(-)	Trace	7

[a] Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (0.7 mmol, 3.5 *equiv*) and *n*Bu_4NOAc (0.24 mmol, 1.2 *equiv*) in acetone (6 mL) under a constant current of 3 mA in an undivided cell at RT open to air. [b] Isolated yield.

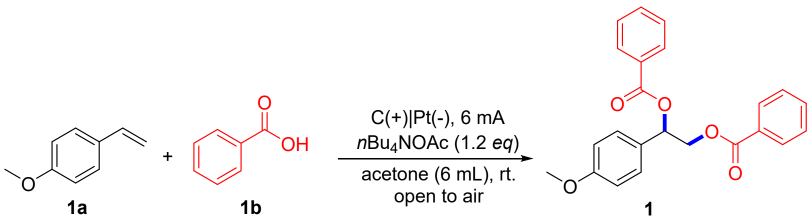
Table S8. Current screening [a]



Entry	Current (mA)	Yield (%) [b]	Time (h)
1	--	N.D.	5
2	3	42	5
3	5	45	3.5
4	6	50	3
5	7	39	2.5
6	9	34	2.5

[a] Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (0.7 mmol, 3.5 *equiv*) and *n*Bu_4NOAc (0.24 mmol, 1.2 *equiv*) in acetone (6 mL) under a constant current of x mA (C anode: Φ = 0.5 cm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT open to air. [b] Isolated yield.

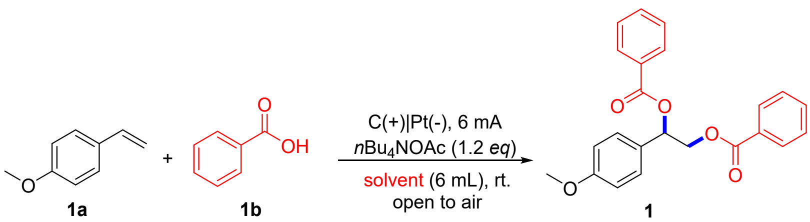
Table S9. Effect of water on reaction [a]



Entry	**Drying of solvent**	**Yield (%)[b]**
1	Analytically pure acetone without drying	50
2	Acetone was dried over 3Å molecular sieve pellets before use	61
3	Acetone was dried over 3Å molecular sieve pellets before use, and 150 mg 3 Å molecular sieve powder was added to the reaction mixture	72

[a] Reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (0.7 mmol, 3.5 *equiv*), *n*Bu_4NOAc (0.24 mmol, 1.2 *equiv*) in acetone (6 mL) under a constant current of 6 mA (C anode: Φ = 0.5 cm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT open to air for 3h. [b] Isolated yield.

Table S10. Green solvent screening [a]



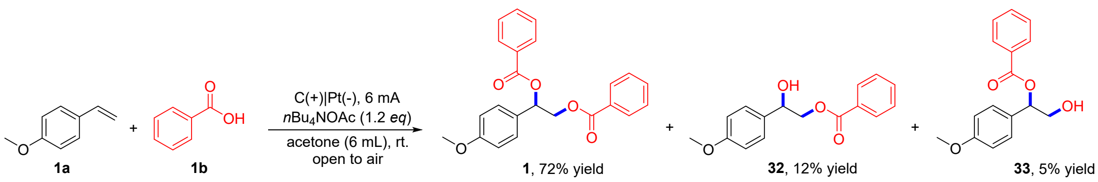
Entry	Solvent	Yield (%) [b]	Time (h)

1	Acetone	72	3
2	Methyl isobutyl ketone [c]	N.R.	3
3	Cyclopentanone [c]	N.R.	3
4	Diethyl carbonate [c]	Voltage overload	0.5
5	2,4,6-Collidine [c]	Voltage overload	0.5
6	Glycerol triacetate [c]	Voltage overload	0.5
7	Acetone/Methyl isobutyl ketone [c] (10:1, v/v)	53	3
8	Acetone/Methyl isobutyl ketone [c] (5:1, v/v)	50	3
9	Acetone/Water (10:1, v/v) [d]	12	3
10	Acetone/Water (5:1, v/v) [d]	Trace	3
11	Acetone/Cyclopentanone [c] (10:1, v/v)	49	3
12	Acetone/Cyclopentanone [c] (5:1, v/v)	48	3
13	Acetone/Diethyl carbonate [c] (10:1, v/v)	50	3
14	Acetone/ Diethyl carbonate [c] (5:1, v/v)	52	3

[a] Unless otherwise noted, reaction conditions: A mixture of **1a** (0.2 mmol, 1 *equiv*), **1b** (0.7 mmol, 3.5 *equiv*), *n*Bu_4NOAc (0.24 mmol, 1.2 *equiv*) and 150 mg 3Å molecular sieve powder in solvent (6 mL, dried over 3 Å molecular sieve pellets, Φ = 3−5 mm) under a constant current of 6 mA (C anode: Φ = 0.5 cm; Pt cathode: 1 cm x 1 cm x 0.2 mm) in an undivided cell at RT open to air. [b] Isolated yield. [c] Dried over 4 Å molecular sieve pellets, Φ = 3−5 mm. [d] Without 150 mg 3Å molecular sieve powder.

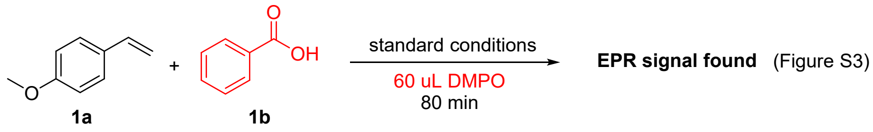
4. Main by-products

In general, there were different degrees of hydroxy ester by-products formation for all reactions examined. For example, diesterification product (**1**), and hydroxy ester secondary products (**32** and **33**) were gave in the model reaction. For the ^{1}H NMR, ^{13}C NMR and HRMS of the secondary products (**32** and **33**), see SI, §7 and §9.



5. Mechanistic investigation

5.1. EPR experiments



The model reaction was carried out under standard conditions in the presence of 60 uL 3,4-dihydro-2,2-dimethyl-2*H*-pyrrole 1-oxide (DMPO). After 80 minutes, the reaction solution was taken out with capillary and analyzed by EPR at room temperature. Radical signals were detected (Figure S3), indicating the possible generation of radicals.

EPR spectra were recorded with a modified Bruker EMXplus-9.5/12 spectrometer operated at 9.8293 GHz. The scan range was 150.0 G, with a resolution of 1500 points and a center field of 3511.15 G. The time constant was 81.92 ms, the conversion time was 66.68 ms, and the scan time was 100.02 s. A modulation amplitude of 10. 0 G, the modulation frequency of 100.00 kHz, and microwave power of 6.325 mW were used for the experiments.

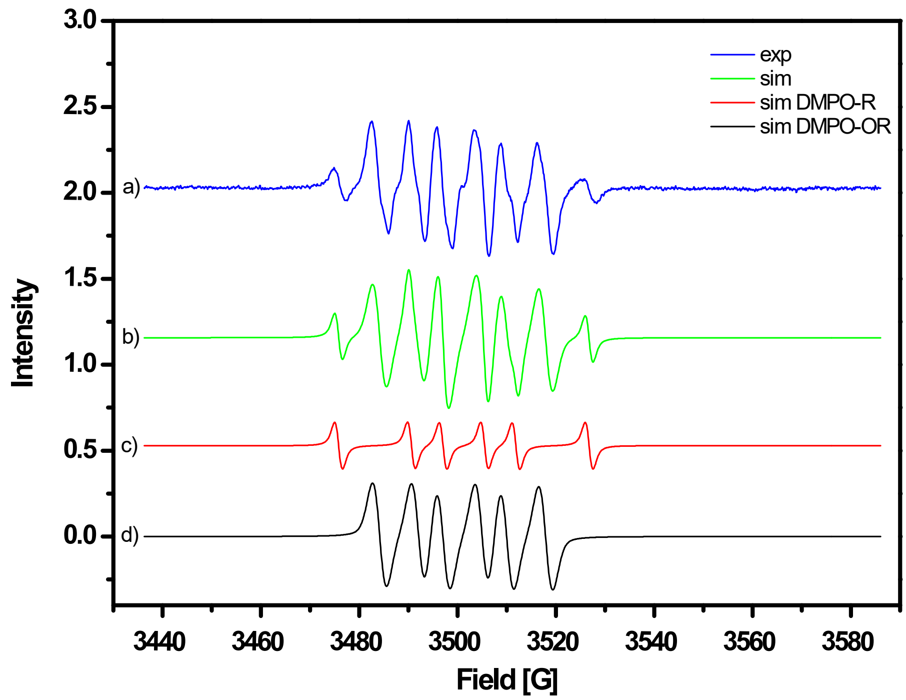
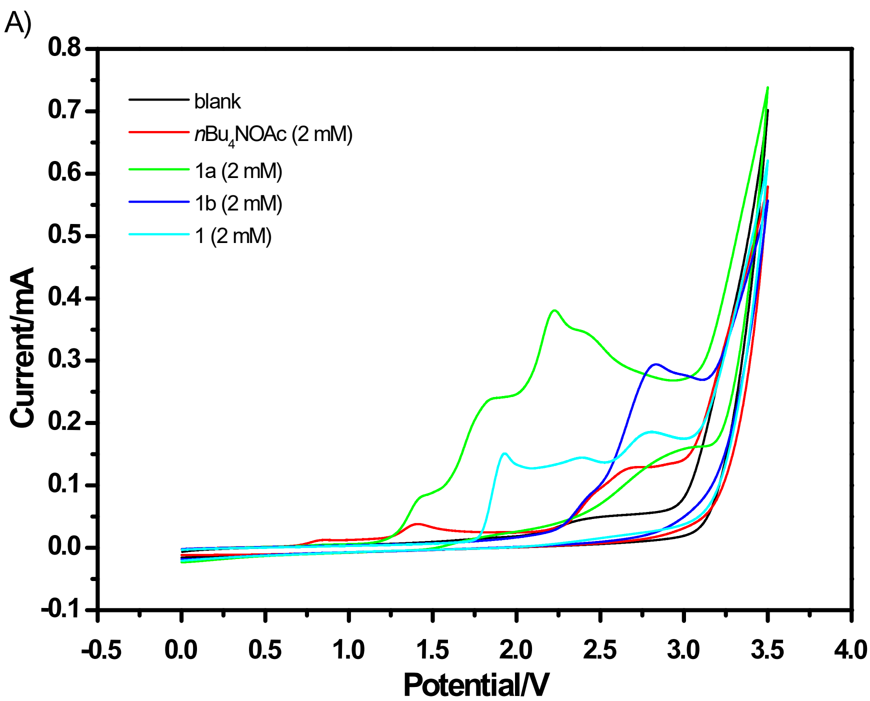


Figure S3 EPR experiments. c) DMPO-R (g-factor=2.00593, A_N = 14.8 G, A_H= 21.3 G); d) DMPO-OR (g-factor=2.00609, A_N = 12.9 G, A_H =7.9 G).

5.2. Cyclic voltammetry experiments

The electrochemical measurement was performed by a computer-controlled electrochemical analyzer. Cyclic voltammetry experiments were performed in a three-electrode cell with MeCN

(15 mL) as solvent, $n\mathrm{Bu_4NClO_4}$ (0.05 M) as supporting electrolyte, and the concentration of the tested compound was 2.0 mM. Glassy carbon (diameter 3 mm) was used as working electrode, platinum wire as auxiliary electrode, and Ag/AgCl (3 M KCl) as reference electrode. And the scanning speed was 100 mV·s^{-1}. The results showed that the onset potential for the oxidation of $n\mathrm{Bu_4NOAc}$ was around +0.715 V, the onset potential for the oxidation of 1-methoxy-4-vinylbenzene (**1a**) was around +1.155 V, the onset potential for the oxidation of benzoic acid (**1b**) was around +1.998 V, the onset potential for the oxidation of product (**1**) was around +1.766 V (Figure S4A). Additionally, cyclic voltammograms with a gradual increase of concentration of $n\mathrm{Bu_4NOAc}$ from 2 to 6 mM showed that oxidation peak of product **1** decreased gradually (Figure S4B). This measurement was from 0.0 V to +3.5 V *vs.* Ag/AgCl (3 M KCl).



B)

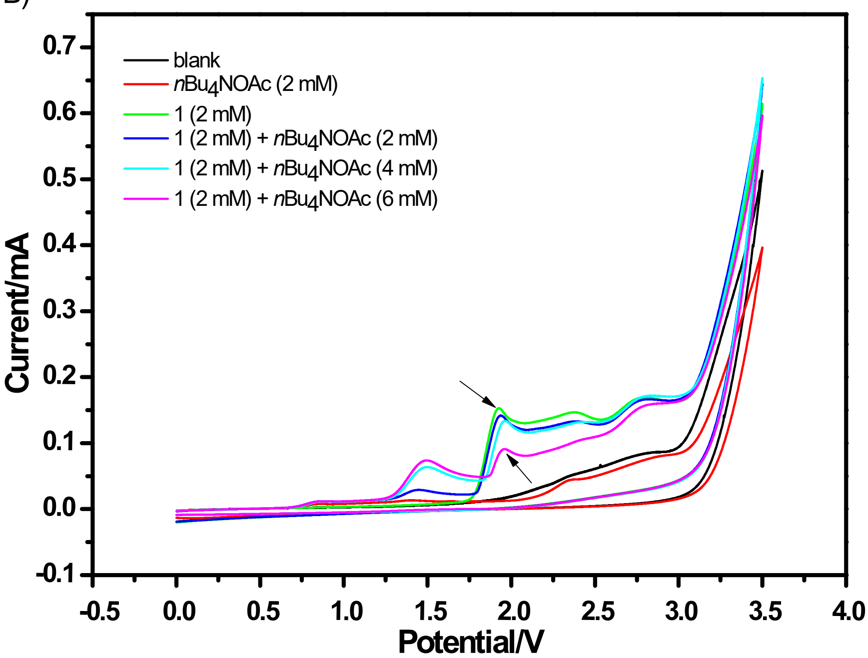


Figure S4 Cyclic voltammetry experiments.

6. Green chemistry metrics

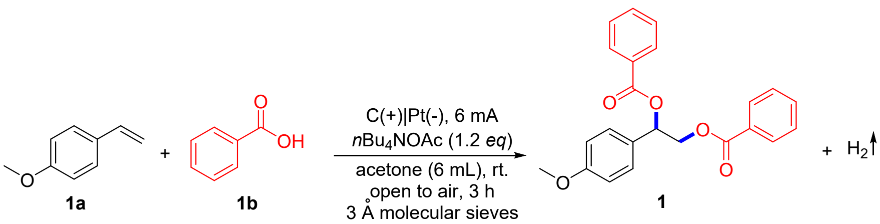


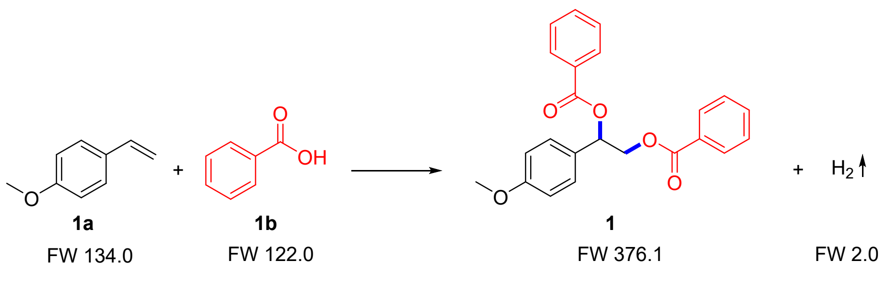
Table S11. The penalty points to calculate the EcoScale

Parameter	Penalty points
1 Yield: 72 %	(100 – 72%yield)/2 = 14
2 Price of reaction components (to obtain 10 mmol of end product)	
*n*Bu$_4$NOAc (1.2 *equiv*), Inexpensive (< $10)	0
Acetone, Inexpensive (< $10)	0
3Å molecular sieve, Inexpensive (< $10)	0
3 Safety	
Acetone (T, toxic)	5
Acetone (F, highly flammable)	5
4 Technical setup	

Common setup	0
5 Temperature/time	
Room temperature, < 1 h	1
6 Workup and purification	
Classcal chromatography	10
Removal of Acetone	0
Penalty points total:	**35**

EcoScale = 100 - sum of individual penalties=100-35=65

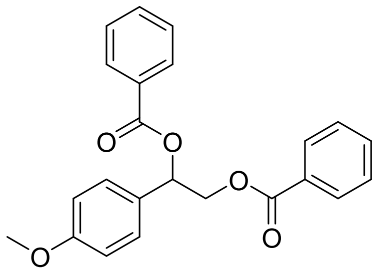
Substrate **1a** (26.8 mg 0.2 mmol, FW 134.0), **1b** (85.4 mg 0.7 mmol, FW 122.0), nBu$_4$NOAc (72.3 mg 0.24 mmol, FW 301.5), and acetone (6 mL, 4739.4 mg) were added to an undivided beaker-type electrolysis cell (10 mL) equipped with a magnetic stir bar. A carbon rod electrode (Φ = 0.5 cm) was used as the anode and a platinum plate (1 cm x 1 cm x 0.2 mm) was used cathode (the electrodes were immersed 1 cm in the reaction solution). The reaction mixture was stirred and electrolyzed at a constant current of 6 mA at room temperature open to air. After reaction completion (monitored by TLC), the reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with 500 mL of petroleum ether/ethyl acetate (20:1, v/v) as the eluent to obtain the target product 1 in 72% yield (54.1 mg, 0.14 mmol, FW 376.1). In addition, recycling 500 mL of petroleum ether/ethyl acetate (20:1, v/v) gave 450 mL of petroleum ether/ethyl acetate (20:1, v/v), which means that 50 mL of petroleum ether/ethyl acetate (20:1, v/v) was lost. That is, petroleum ether (47.6 mL, 30940.0 mg), ethyl acetate (2.4 mL, 2164.8 mg) were lost.



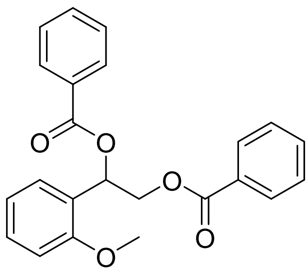
Atom efficiency = 376.1 / (134.0 + 122.0*2) = 99%

Mass intensity = (26.8 + 85.4 + 72.3 + 4739.4 + 30940.0 + 2164.8) / 54.1 = 702.9 mg/mg

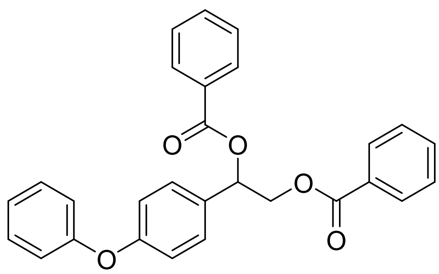
7. Characterization data of the products and by-products



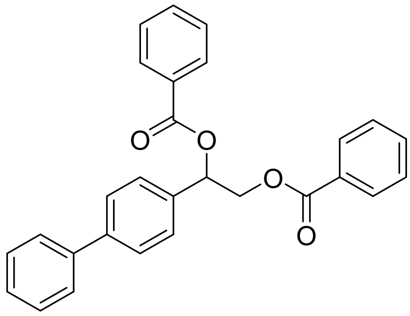
1-(4-methoxyphenyl)ethane-1,2-diyl dibenzoate (1): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 54.1 mg, 72% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.9 Hz, 2H), 7.99 (d, *J* = 7.9 Hz, 2H), 7.58 – 7.49 (m, 2H), 7.48 – 7.37 (m, 6H), 6.92 (d, *J* = 8.2 Hz, 2H), 6.37 (dd, *J* = 8.1, 3.5 Hz, 1H), 4.78 – 4.72 (m, 1H), 4.63 (dd, *J* = 11.8, 3.5 Hz, 1H), 3.80 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.2, 165.6, 160.0, 133.1, 133.0, 130.1, 129.9, 129.7, 129. 7, 128.8, 128.4, 128.4, 128.2, 114.2, 73.7, 66.6, 55.3. HRMS (ESI): m/z: calcd for $C_{23}H_{20}O_5$ $(M+Na)^+$ 399.1203; found 399.1202. Calcd for $C_{23}H_{20}O_5$ $(M+K)^+$ 415.0942; found 415.0941.



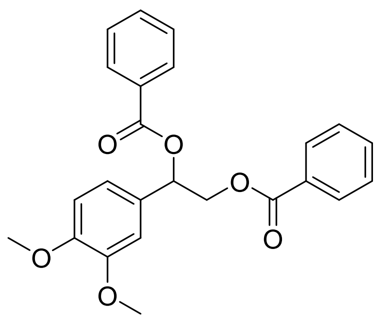
1-(2-methoxyphenyl)ethane-1,2-diyl dibenzoate (2): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 47.4 mg, 63% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12 (d, *J* = 7.8 Hz, 2H), 7.99 (d, *J* = 7.9 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.53 – 7.48 (m, 2H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.30 (t, *J* = 7.8 Hz, 1H), 6.97 (t, *J* = 7.5 Hz, 1H), 6.92 (d, *J* = 8.2 Hz, 1H), 6.80 (dd, *J* = 7.3, 3.1 Hz, 1H), 4.71 (dd, *J* = 11.8, 7.5 Hz, 1H), 4.67 (dd, *J* = 11.8, 3.2 Hz, 1H), 3.90 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.3, 165.5, 156.4, 133.0, 132.9, 130.2, 130.1, 129.8, 129.7, 129.5, 128.4, 128.3, 127.0, 125.0, 120.7, 110.7, 69.4, 65.8, 55.5. HRMS (ESI): m/z: calcd for $C_{23}H_{20}O_5$ $(M+Na)^+$ 399.1203; found 399.1202. Calcd for $C_{23}H_{20}O_5$ $(M+K)^+$ 415.0942; found 415.0942.



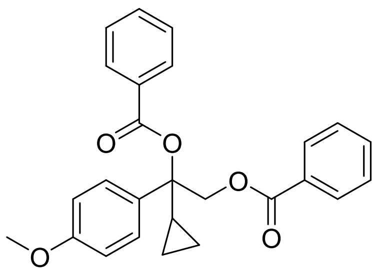
1-(4-phenoxyphenyl)ethane-1,2-diyl dibenzoate (3): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 39.4 mg, 45% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12 – 8.06 (m, 2H), 8.01 – 7.96 (m, 2H), 7.58 – 7.52 (m, 2H), 7.50 – 7.46 (m, 2H), 7.45 – 7.38 (m, 4H), 7.36 – 7.30 (m, 2H), 7.11 (tt, *J* = 7.3, 1.1 Hz, 1H), 7.05 – 6.98 (m, 4H), 6.40 (dd, *J* = 8.1, 3.8 Hz, 1H), 4.75 (dd, *J* = 11.8, 8.2 Hz, 1H), 4.66 (dd, *J* = 11.8, 3.8 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.2, 165.6, 157.8, 156.8, 133.2, 133.1, 131.3, 130.0, 129.8, 129.8, 129.7, 128.4, 128.4, 128.4, 123.6, 119.2, 118.8, 73.6, 66.6. HRMS (ESI): m/z: calcd for $C_{28}H_{22}O_5$ $(M+Na)^+$ 461.1359; found 461.1359. Calcd for $C_{28}H_{22}O_5$ $(M+K)^+$ 477.1099; found 477.1098.



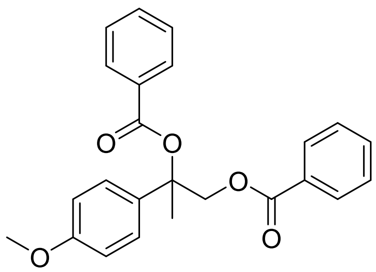
1-([1,1'-biphenyl]-4-yl)ethane-1,2-diyl dibenzoate (4): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 44.8 mg, 53% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12 (d, *J* = 7.4 Hz, 2H), 8.01 (d, *J* = 7.4 Hz, 2H), 7.63 – 7.51 (m, 8H), 7.43 (dq, *J* = 15.6, 7.9 Hz, 6H), 7.35 (t, *J* = 7.4 Hz, 1H), 6.46 (dd, *J* = 8.0, 3.6 Hz, 1H), 4.79 (dd, *J* = 11.8, 8.2 Hz, 1H), 4.71 (dd, *J* = 11.9, 3.7 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.2, 165.6, 141.7, 140.6, 135.6, 133.2, 133.1, 130.0, 129.8, 129.8, 129.7, 128.8, 128.4, 128.4, 127.5, 127.5, 127.2, 127.1, 73.9, 66.6. HRMS (ESI): m/z: calcd for C28H22O5 (M+Na)+ 445.1410; found 445.1410.



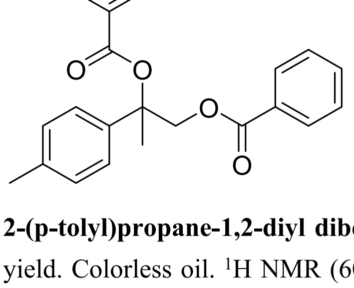
1-(3,4-dimethoxyphenyl)ethane-1,2-diyl dibenzoate (5): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 54.1 mg, 67% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.8 Hz, 2H), 8.00 (d, *J* = 7.9 Hz, 2H), 7.58 – 7.51 (m, 2H), 7.42 (m, 4H), 7.46 – 7.37 (m, 1H), 7.03 (s, 1H), 6.88 (d, *J* = 8.3 Hz, 1H), 6.36 (dd, *J* = 8.3, 3.7 Hz, 1H), 4.77 (dd, *J* = 11.8, 8.4 Hz, 1H), 4.65 (dd, *J* = 11.9, 3.7 Hz, 1H), 3.88 (d, *J* = 7.1 Hz, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.2, 165.7, 149.5, 149.3, 133.1, 133.1, 130.1, 129.8, 129.7, 129.7, 129.2, 128.4, 128.4, 119.4, 111.5, 110.3, 73.9, 66.6, 56.0, 56.0. HRMS (ESI): m/z: calcd for $C_{24}H_{22}O_6$ $(M+Na)^+$ 429.1309; found 429.1307. Calcd for $C_{24}H_{22}O_6$ $(M+K)^+$ 455.1048; found 455.1045.



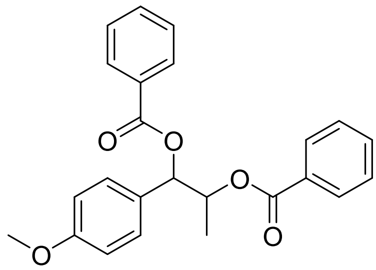
1-cyclopropyl-1-(4-methoxyphenyl)ethane-1,2-diyl dibenzoate (6): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 54.1 mg, 65% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05 (d, J = 7.8 Hz, 2H), 7.95 (d, J = 7.8 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.3 Hz, 1H), 7.47 – 7.39 (m, 4H), 7.37 (t, J = 7.6 Hz, 2H), 6.89 (d, J = 8.6 Hz, 2H), 5.18 (d, J = 11.4 Hz, 1H), 5.03 (d, J = 11.4 Hz, 1H), 3.78 (s, 3H), 1.91 – 1.83 (m, 1H), 0.76 – 0.46 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 164.5, 163.3, 157.5, 131.4, 131.4, 130.6, 129.6, 128.5, 128.1, 126.9, 126.8, 125.3, 112.2, 82.6, 65.7, 53.7, 17.5, 1.10, 0.0. HRMS (ESI): m/z: calcd for $C_{26}H_{24}O_5$ $(M+Na)^+$ 439.1516; found 439.1514. Calcd for $C_{26}H_{24}O_5$ $(M+K)^+$ 445.1255; found 445.1252.



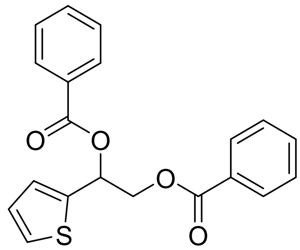
2-(4-methoxyphenyl)propane-1,2-diyl dibenzoate (7): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 50.7 mg, 65% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (t, J = 7.7 Hz, 4H), 7.59 – 7.50 (m, 2H), 7.45 – 7.38 (m, 6H), 6.89 (d, J = 8.8 Hz, 2H), 4.81 (d, J = 11.5 Hz, 1H), 4.58 (d, J = 11.5 Hz, 1H), 3.78 (s, 3H), 2.13 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.1, 164.8, 159.2, 133.2, 133.1, 132.9, 131.1, 130.0, 129.7, 128.4, 128.4, 126.4, 113.4, 82.3, 70.8, 55.2, 21.9. HRMS (ESI): m/z: calcd for $C_{24}H_{22}O_5$ $(M+Na)^+$ 413.1359; found 413.1358. Calcd for $C_{24}H_{22}O_5$ $(M+K)^+$ 429.1099; found 429.1096.



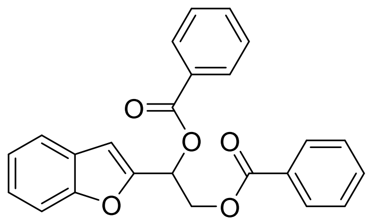
2-(p-tolyl)propane-1,2-diyl dibenzoate (8): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 30.6 mg, 41% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (t, J = 8.9 Hz, 4H), 7.55 (t, J = 6.7 Hz, 2H), 7.42 (t, J = 7.7 Hz, 4H), 7.36 (d, J = 8.1 Hz, 2H), 7.17 (d, J = 8.0 Hz, 2H), 4.81 (d, J = 11.5 Hz, 1H), 4.59 (d, J = 11.5 Hz, 1H), 2.33 (s, 3H), 2.13 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.1, 164.8, 138.2, 137.6, 133.1, 132.9, 131.1, 130.0, 129.7, 129.7, 129.3, 128.4, 128.4, 125.0, 82.5, 70.8, 22.0, 21.0. HRMS (ESI): m/z: calcd for $C_{24}H_{22}O_4$ $(M+Na)^+$ 397.1410; found 397.1410. Calcd for $C_{24}H_{22}O_4$ $(M+K)^+$ 413.1150; found 413.1149.



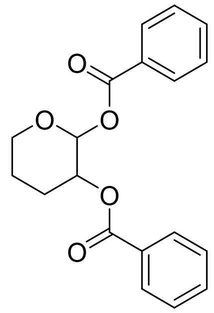
1-(4-methoxyphenyl)propane-1,2-diyl dibenzoate (9): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 46.8 mg, 60% yield (dr = 1.9:1). Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 8.11 – 7.93 (m, 4H), 7.58 – 7.47 (m, 2H), 7.46 – 7.34 (m, 6H), 6.90 (d, *J* = 8.4 Hz, 2H), 6.15 (d *J* = 4.3 Hz, 1H), 5.70 – 5.57 (m, 1H), 3.79 (s, 3H), 1.42 (d, *J* = 6.4 Hz, 2H), 1.28 (d, *J* = 6.4 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.0, 165.7, 165.6, 165.3, 159.9, 159.6, 133.1, 132.9, 132.9, 130.3, 130.3, 130.2, 130.1, 129.7, 129.6, 129.6, 129.6, 129.0, 128.9, 128.8, 128.4, 128.4, 128.3, 128.3, 114.1, 113.9, 77.9, 76.9, 72.5, 72.3, 55.2, 16.7, 15.4. HRMS (ESI): m/z: calcd for $C_{24}H_{22}O_5$ $(M+Na)^+$ 413.1359; found 413.1359. Calcd for $C_{24}H_{22}O_5$ $(M+K)^+$ 429.1099; found 429.1097.



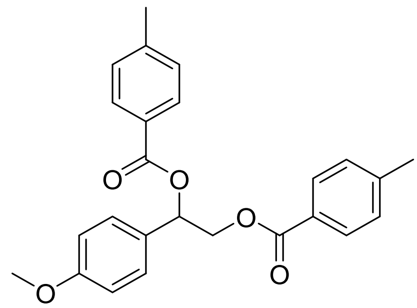
(thiophen-2-yl)ethane-1,2-diyl dibenzoate (10): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 43.0 mg, 61% yield. Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.0 Hz, 2H), 8.02 (d, *J* = 8.0 Hz, 2H), 7.58 – 7.52 (m, 2H), 7.45 – 7.39 (m, 4H), 7.33 (d, *J* = 5.0 Hz, 1H), 7.24 (d, *J* = 3.4 Hz, 1H), 7.02 (t, *J* = 4.1 Hz, 1H), 6.72 (dd, *J* = 6.9, 4.5 Hz, 1H), 4.82 – 4.74 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.1, 165.5, 138.8, 133.2, 133.1, 129.8, 129.8, 129.8, 129.7, 128.4, 126.8, 126.7, 126.2, 69.5, 66.3. HRMS (ESI): m/z: calcd for C20H16O4S (M+Na)+ 375.0662; found 375.0664. Calcd for C20H16O4S (M+K)+ 391.0401; found 391.0400.



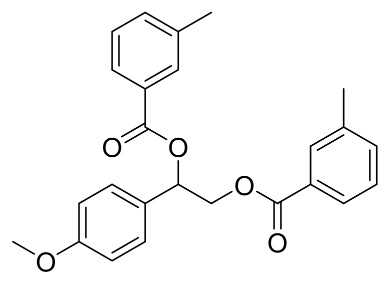
(benzofuran-2-yl)ethane-1,2-diyl dibenzoate (11): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 47.9 mg, 62% yield. Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.3 Hz, 2H), 8.00 (d, *J* = 8.3 Hz, 2H), 7.59 – 7.47 (m, 4H), 7.45 – 7.38 (m, 4H), 7.30 (d, *J* = 8.3 Hz, 1H), 7.24 – 7.20 (m, 1H), 6.90 (s, 1H), 6.64 (dd, *J* = 7.3, 4.4 Hz, 1H), 4.97 (dd, *J* = 11.8, 7.5 Hz, 1H), 4.92 (dd, *J* = 11.8, 4.4 Hz, 1H). HRMS (ESI): m/z: calcd for $C_{24}H_{18}O_5$ $(M+Na)^+$ 409.1046; found 409.1044. Calcd for $C_{27}H_{20}O_5$ $(M+K)^+$ 425.0786; found 425.0786.



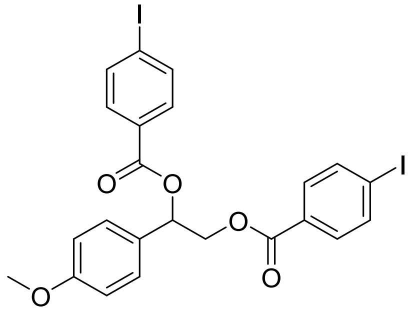
tetrahydro-2H-pyran-2,3-diyl dibenzoate (12): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 37.8 mg, 58% yield (dr > 20:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.09 (dd, *J* = 7.8, 4.2 Hz, 4H), 7.61 – 7.54 (m, 2H), 7.51 – 7.42 (m, 4H), 6.25 (d, *J* = 3.4 Hz, 1H), 5.20 – 5.15 (m, 1H), 4.08 – 4.01 (m, 1H), 3.88 – 3.81 (m, 1H), 2.34 – 2.26 (m, 1H), 2.15 – 2.02 (m, 2H), 1.70 – 1.60 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 165.5, 164.6, 133.4, 133.2, 130.0, 129.9, 129.8, 129.7, 128.5, 128.4, 92.2, 68.2, 63.0, 24.7, 21.1. HRMS (ESI): m/z: calcd for $C_{19}H_{18}O_5$ $(M+Na)^+$ 349.1046; found 349.1047. Calcd for $C_{19}H_{18}O_5$ $(M+K)^+$ 365.0786; found 365.0786.



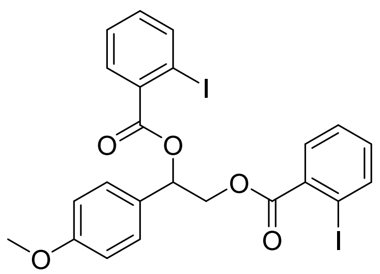
1-(4-methoxyphenyl)ethane-1,2-diyl bis(4-methylbenzoate) (13): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 57.4 mg, 71% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.1 Hz, 2H), 7.88 (d, *J* = 8.1 Hz, 2H), 7.44 (d, *J* = 8.7 Hz, 2H), 7.20 (dd, *J* = 18.4, 8.0 Hz, 4H), 6.91 (d, *J* = 8.7 Hz, 2H), 6.34 (dd, *J* = 8.2, 3.6 Hz, 1H), 4.71 (dd, *J* = 11.8, 8.4 Hz, 1H), 4.60 (dd, *J* = 11.8, 3.7 Hz, 1H), 3.79 (s, 3H), 2.38 (d, *J* = 9.1 Hz, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.3, 165.7, 159.9, 143.8, 129. 8, 129.7, 129.0, 129.1, 128.2, 127.4, 127.1, 114.2, 73.5, 66.5, 55.3, 21.6. HRMS (ESI): m/z: calcd for $C_{25}H_{24}O_5$ $(M+Na)^+$ 427.1516; found 427.1515. Calcd for $C_{25}H_{24}O_5$ $(M+K)^+$ 443.1255; found 443.1255.



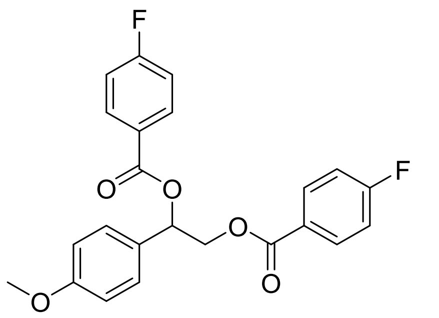
1-(4-methoxyphenyl)ethane-1,2-diyl bis(3-methylbenzoate) (14): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 51.7 mg, 64% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.7 Hz, 1H), 7.87 (d, *J* = 7.7 Hz, 1H), 7.45 (d, *J* = 8.7 Hz, 2H), 7.41 – 7.34 (m, 2H), 7.25 – 7.19 (m, 4H), 6.92 (d, *J* = 8.7 Hz, 2H), 6.36 (dd, *J* = 8.3, 3.7 Hz, 1H), 4.70 (dd, *J* = 11.8, 8.5 Hz, 1H), 4.61 (dd, *J* = 11.9, 3.8 Hz, 1H), 3.80 (s, 3H), 2.55 (d, *J* = 8.7 Hz, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.9, 166.5, 159.9, 140.4, 132.1, 132.1, 131.7, 131.7, 130.7, 130.7, 129.5, 129.2, 128.9, 128.3, 125.7, 125.7, 114.2, 73.5, 66.4, 55.3, 21.7. HRMS (ESI): m/z: calcd for $C_{25}H_{24}O_5$ $(M+Na)^+$ 427.1516; found 427.1516. Calcd for $C_{25}H_{24}O_5$ $(M+K)^+$ 443.1255; found 443.1254.



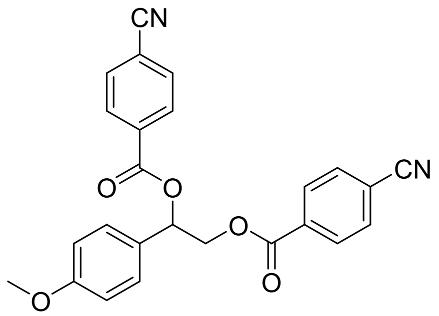
1-(4-methoxyphenyl)ethane-1,2-diyl bis(4-iodobenzoate) (15): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 41.4 mg, 33% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 – 7.73 (m, 6H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.42 (d, *J* = 8.6 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 6.33 (dd, *J* = 8.5, 3.6 Hz, 1H), 4.72 (dd, *J* = 11.9, 8.5 Hz, 1H), 4.60 (dd, *J* = 11.9, 3.7 Hz, 1H), 3.80 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 165.7, 165.2, 160.1, 137.8, 137.8, 131.1, 131.0, 129.5, 129.2, 128.2, 128.2, 114.3, 101.0, 73.9, 66.6, 55.3. HRMS (ESI): m/z: calcd for $C_{23}H_{18}I_2O_5$ $(M+Na)^+$ 650.9136; found 650.9128. Calcd for $C_{23}H_{18}I_2O_5$ $(M+K)^+$ 666.8875; found 666.8866.



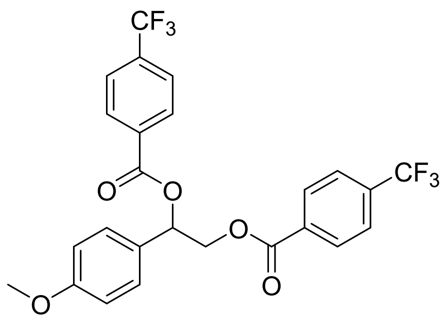
1-(4-methoxyphenyl)ethane-1,2-diyl bis(2-iodobenzoate) (16): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 94.1 mg, 75% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.9 Hz, 2H), 7.85 (d, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.8 Hz, 1H), 7.46 (d, *J* = 8.6 Hz, 2H), 7.41 – 7.33 (m, 2H), 7.12 (t, *J* = 7.6 Hz, 2H), 6.93 (d, *J* = 8.6 Hz, 2H), 6.41 (dd, *J* = 8.6, 3.3 Hz, 1H), 4.79 (dd, *J* = 11.8, 8.9 Hz, 1H), 4.64 (dd, *J* = 12.0, 3.4 Hz, 1H), 3.80 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 165.9, 165.4, 160.1, 141.5, 141.4, 134.8, 134.5, 132.8, 132.8, 131.4, 131.3, 128.5, 128.0, 127.9, 127.9, 114.3, 94.3, 94.2, 74.4, 66.9, 55.3. HRMS (ESI): m/z: calcd for $C_{23}H_{18}I_2O_5$ $(M+Na)^+$ 650.9136; found 650.9129. Calcd for $C_{23}H_{18}I_2O_5$ $(M+K)^+$ 666.8875; found 666.8864.



1-(4-methoxyphenyl)ethane-1,2-diyl bis(4-fluorobenzoate) (17): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 50.3 mg, 61% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11 – 8.05 (m, 2H), 8.03 – 7.96 (m, 2H), 7.43 (d, *J* = 8.6 Hz, 2H), 7.13 – 7.04 (m, 4H), 6.93 (d, *J* = 8.7 Hz, 2H), 6.34 (dd, *J* = 8.4, 3.5 Hz, 1H), 4.74 (dd, *J* = 11.8, 8.5 Hz, 1H), 4.60 (dd, *J* = 11.9, 3.7 Hz, 1H), 3.81 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.7 (d, *J* = 234.5 Hz), 165.2, 165.1 (d, *J* = 254.5 Hz) , 164.7, 160.0, 132.2 (d, *J* = 6.6 Hz), 132.2 (d, *J* = 6.6 Hz), 128.4, 128.2, 126.3 (d, *J* = 3.0 Hz), 126.0 (d, *J* = 3.0 Hz), 115.6 (d, *J* = 22.0 Hz), 115.6 (d, *J* = 22.0 Hz), 114.26 , 73.84 , 66.60 , 55.30 . ^{19}F NMR (565 MHz, $CDCl_3$) δ -105.2, -105.3. HRMS (ESI): m/z: calcd for $C_{23}H_{18}F_2O_5$ $(M+Na)^+$ 435.1015; found 435.1013. Calcd for $C_{23}H_{18}F_2O_5$ $(M+K)^+$ 451.0754; found 451.0748.

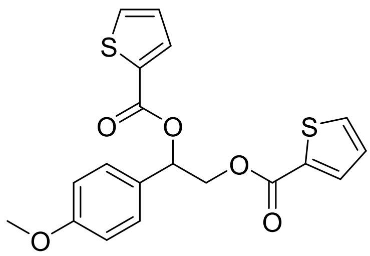


1-(4-methoxyphenyl)ethane-1,2-diyl bis(4-cyanobenzoate) (18): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 51.2 mg, 62% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.3 Hz, 2H), 8.07 (d, *J* = 8.3 Hz, 2H), 7.73 (dd, *J* = 13.1, 8.4 Hz, 4H), 7.44 (d, *J* = 8.6 Hz, 2H), 6.95 (d, *J* = 8.6 Hz, 2H), 6.39 (dd, *J* = 8.4, 3.3 Hz, 1H), 4.81 (dd, *J* = 11.9, 8.7 Hz, 1H), 4.66 (dd, *J* = 12.0, 3.5 Hz, 1H), 3.82 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 164.5, 164.0, 160.3, 133.7, 133.4, 132.3, 132.3, 130.2, 130.1, 128.2, 127.5, 117.7, 116.8, 114.4, 74.5, 66.9, 55.3. HRMS (ESI): m/z: calcd for $C_{25}H_{18}N_2O_5$ $(M+Na)^+$ 449.1108; found 449.1107. Calcd for $C_{25}H_{18}N_2O_5$ $(M+K)^+$ 465.0847; found 465.0842.

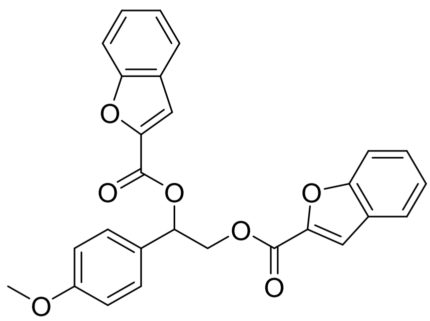


1-(4-methoxyphenyl)ethane-1,2-diyl bis(4-(trifluoromethyl)benzoate) (19): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 65.5 mg, 64% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.1

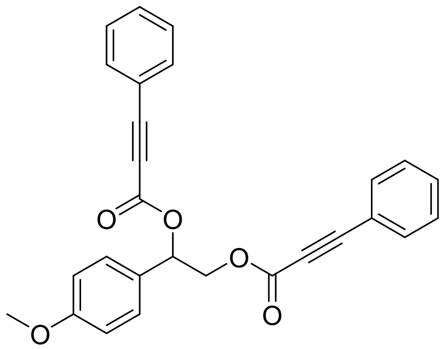
Hz, 2H), 8.09 (d, J = 8.1 Hz, 2H), 7.69 (dd, J = 13.8, 8.3 Hz, 4H), 7.45 (d, J = 8.6 Hz, 2H), 6.94 (d, J = 8.7 Hz, 2H), 6.41 (dd, J = 8.5, 3.5 Hz, 1H), 4.81 (dd, J = 11.9, 8.6 Hz, 1H), 4.67 (dd, J = 12.0, 3.6 Hz, 1H), 3.81 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 165.0, 164.5, 160.2, 134.7 (q, J = 32.6 Hz), 134.7 (q, J = 32.6 Hz), 133.2, 132.9, 130.1, 130.0, 128.2, 127.9, 126.3, 125.5, 125.5, 123.4 (q, J = 280.0 Hz), 123.4 (q, J = 280.0 Hz), 122.6, 120.8, 114.4, 74.2, 66.8, 55.3. ^{19}F NMR (565 MHz, $CDCl_3$) δ -63.2, -63.2. HRMS (ESI): m/z: calcd for $C_{25}H_{18}F_6O_5$ $(M+Na)^+$ 535.0951; found 535.0949. Calcd for $C_{25}H_{18}F_6O_5$ $(M+K)^+$ 551.0690; found 551.0689.



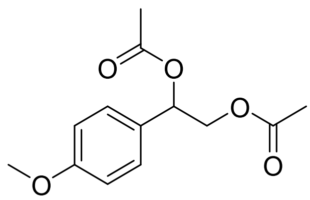
1-(4-methoxyphenyl)ethane-1,2-diyl bis(thiophene-2-carboxylate) (20): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 46.6 mg, 60% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, J = 3.6 Hz, 1H), 7.77 (d, J = 3.5 Hz, 1H), 7.54 (dd, J = 10.8, 4.9 Hz, 2H), 7.43 (d, J = 8.6 Hz, 2H), 7.07 (dt, J = 11.1, 4.3 Hz, 2H), 6.91 (d, J = 8.6 Hz, 2H), 6.28 (dd, J = 8.1, 3.8 Hz, 1H), 4.66 (dd, J = 11.8, 8.2 Hz, 1H), 4.59 (dd, J = 11.8, 3.8 Hz, 1H), 3.80 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 161.7, 161.2, 160.0, 133.8, 133.8, 133.5, 133.3, 132.7, 132.7, 128.4, 128.3, 128.2, 127.8, 114.2, 73.8, 66.5, 55.3. HRMS (ESI): m/z: calcd for $C_{19}H_{16}O_5S_2$ $(M+Na)^+$ 411.0331; found 411.0331. Calcd for $C_{19}H_{16}O_5S_2$ $(M+K)^+$ 427.0071; found 427.0070.



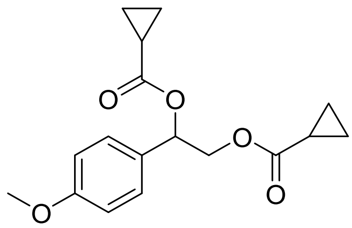
1-(4-methoxyphenyl)ethane-1,2-diyl bis(benzofuran-2-carboxylate) (21): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 52.9 mg, 58% yield. Light yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 – 7.62 (m, 2H), 7.57 (dd, J = 19.3, 10.4 Hz, 3H), 7.52 – 7.46 (m, 3H), 7.45 – 7.40 (m, 2H), 7.31 – 7.25 (m, 2H), 6.94 (d, J = 8.6 Hz, 2H), 6.43 (dd, J = 8.6, 3.3 Hz, 1H), 4.83 (dd, J = 11.9, 8.8 Hz, 1H), 4.68 (dd, J = 11.9, 3.4 Hz, 1H), 3.81 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.1, 165.5, 155.1, 151.9, 133.4, 133.2, 129.9, 129.8, 129.6, 129.6, 128.5, 128.4, 127.7, 125.0, 123.1, 121.4, 111.5, 106.2, 67.6, 64.0. HRMS (ESI): m/z: calcd for $C_{27}H_{20}O_7$ $(M+Na)^+$ 479.1101; found 479.1100. Calcd for $C_{27}H_{20}O_7$ $(M+K)^+$ 495.0841; found 495.0841.



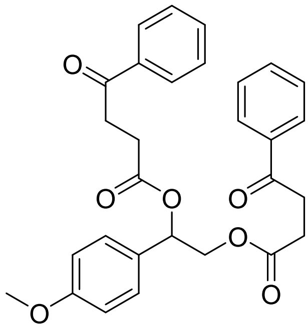
1-(4-methoxyphenyl)ethane-1,2-diyl bis(3-phenylpropiolate) (22): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 36.5 mg, 43% yield. Light yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 – 7.57 (m, 4H), 7.48 – 7.41 (m, 2H), 7.41 – 7.34 (m, 6H), 6.93 (d, *J* = 8.6 Hz, 2H), 6.15 (dd, *J* = 8.5, 3.2 Hz, 1H), 4.57 (dd, *J* = 11.9, 8.7 Hz, 1H), 4.48 (dd, *J* = 12.1, 3.4 Hz, 1H), 3.81 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 160.2, 153.6, 153.0, 133.1, 133.0, 130.7, 130.7, 128.6, 128.4, 127.5, 119.6, 119.5, 114.3, 87.4, 87.2, 80.5, 80.2, 74.5, 66.9, 55.3. HRMS (ESI): m/z: calcd for $C_{27}H_{20}O_5$ $(M+Na)^+$ 447.1203; found 447.1203. Calcd for $C_{27}H_{20}O_5$ $(M+K)^+$ 463.0942; found 463.0944.



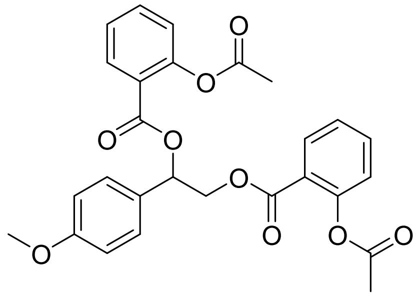
1-(4-methoxyphenyl)ethane-1,2-diyl diacetate (23) [1]: R_f = 0.25 (Petroleum ether/EtOAc, 10:1). 41.3 mg, 82% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.7 Hz, 2H), 6.89 (d, *J* = 8.7 Hz, 2H), 5.98 – 5.95 (m, 1H), 4.32 – 4.27 (m, 2H), 3.80 (s, 3H), 2.09 (s, 3H), 2.05 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 170.5, 170.0, 159.9, 128.7, 128.2, 114.1, 73.0, 66.0, 55.3, 21.0, 20.7.



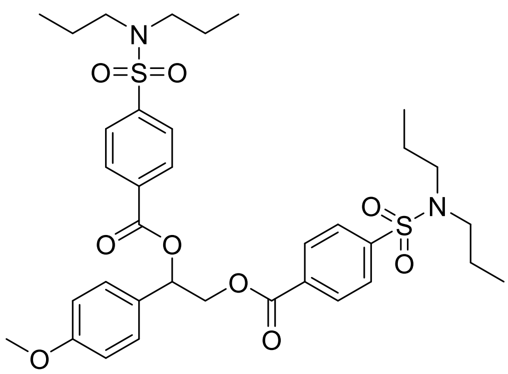
1-(4-methoxyphenyl)ethane-1,2-diyl dicyclopropanecarboxylate (24): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 42.6 mg, 70% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.6 Hz, 2H), 6.89 (d, *J* = 8.6 Hz, 2H), 6.01 – 5.94 (m, 1H), 4.33 (dd, *J* = 11.7, 8.4 Hz, 1H), 4.26 (dd, *J* = 11.8, 3.9 Hz, 1H), 3.80 (s, 3H), 1.67 – 1.59 (m, 2H), 1.05 – 0.95 (m, 4H), 0.91 – 0.82 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.3, 173.8, 159.8, 128.9, 128.1, 114.0, 72.9, 66.0, 55.3, 8.5, 8.4, 8.42. HRMS (ESI): m/z: calcd for $C_{17}H_{20}O_5$ $(M+Na)^+$ 327.1203; found 327.1203. Calcd for $C_{17}H_{20}O_5$ $(M+K)^+$ 343.0942; found 343.0943.



1-(4-methoxyphenyl)ethane-1,2-diyl bis(4-oxo-4-phenylbutanoate) (25): R_f = 0.25 (Petroleum ether/EtOAc, 2:1). 52.7 mg, 58% yield. Light red oil. ^1H NMR (600 MHz, $CDCl_3$) δ 8.00 – 7.92 (m, 4H), 7.55 (t, *J* = 7.3 Hz, 2H), 7.47 – 7.41 (m, 4H), 7.28 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 8.5 Hz, 2H), 6.02 (dd, *J* = 8.1, 3.6 Hz, 1H), 4.40 – 4.30 (m, 2H), 3.79 (s, 3H), 3.36 – 3.21 (m, 4H), 2.89 – 2.82 (m, 1H), 2.81 – 2.71 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.8, 172.5, 171.9, 159.8, 136.6, 136.6, 133.1, 128.6, 128.5, 128.1, 128.0, 128.0, 114.1, 73.2, 66.1, 55.3, 33.4, 33.3, 28.5, 28.2. HRMS (ESI): m/z: calcd for $C_{29}H_{28}O_7$ $(M+Na)^+$ 511.1727; found 511.1726. Calcd for $C_{29}H_{28}O_7$ $(M+K)^+$ 527.1467; found 527.1467.

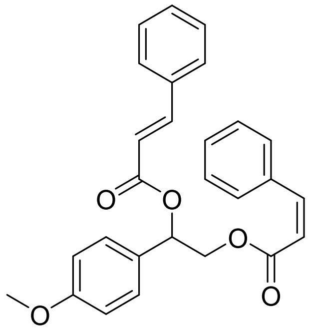


1-(4-methoxyphenyl)ethane-1,2-diyl bis(2-acetoxybenzoate) (26): R_f = 0.25 (Petroleum ether/EtOAc, 10:1). 71.8 mg, 73% yield. Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.94 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.57 – 7.51 (m, 2H), 7.41 – 7.37 (m, 2H), 7.32 – 7.25 (m, 2H), 7.08 (dd, *J* = 8.1, 4.3 Hz, 2H), 6.93 – 6.88 (m, 2H), 6.30 (dd, *J* = 7.8, 4.3 Hz, 1H), 4.63 – 4.56 (m, 2H), 3.80 (s, 3H), 2.29 (s, 3H), 2.22 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.5, 169.4, 163.8, 163.5, 160.0, 160.0, 150.8, 134.0, 133.9, 131.8, 131.8, 128.3, 128.2, 126.0, 126.0, 123. 9, 123.8, 123.3, 122.7, 114.2, 73.6, 66.5, 55.3, 20.9, 20.8. HRMS (ESI): m/z: calcd for $C_{27}H_{24}O_9$ $(M+Na)^+$ 515.1313; found 515.1307. Calcd for $C_{27}H_{24}O_9$ $(M+K)^+$ 531.1052; found 531.1047.

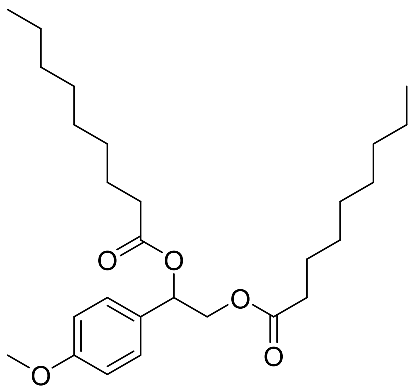


1-(4-methoxyphenyl)ethane-1,2-diyl bis(4-(N,N-dipropylsulfamoyl)benzoate) (27): R_f = 0.25

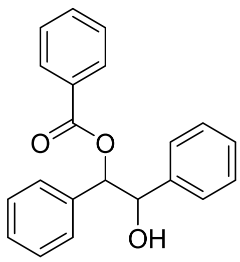
(Petroleum ether/EtOAc, 20:1). 94.1 mg, 67% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.17 (d, *J* = 7.9 Hz, 2H), 8.09 (d, *J* = 7.9 Hz, 2H), 7.86 (dd, *J* = 14.0, 8.2 Hz, 4H), 7.45 (d, *J* = 8.1 Hz, 2H), 6.95 (d, *J* = 8.1 Hz, 2H), 6.45 – 6.35 (m, 1H), 4.81 (dd, *J* = 12.0, 8.5 Hz, 1H), 4.67 (dd, *J* = 12.0, 3.6 Hz, 1H), 3.81 (s, 3H), 3.22 – 3.00 (m, 8H), 1.63 – 1.47 (m, 8H), 0.86 (t, *J* = 7.0 Hz, 12H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 164.8, 164.3, 160.2, 144.7, 133.2, 132.9, 130.3, 130.2, 128.2, 127.8, 127.1, 127.1, 114.4, 74.3, 66.9, 55.3, 50.0, 50.0, 22.0, 22.0, 11.1. HRMS (ESI): m/z: calcd for $C_{35}H_{46}N2O_9S_2$ $(M+Na)^+$ 725.2537; found 725.2535. Calcd for r $C_{35}H_{46}N2O_9S_2$ $(M+K)^+$ 741.2276; found 741.2279.



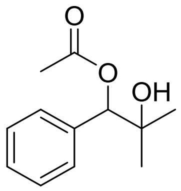
1-(4-methoxyphenyl)ethane-1,2-diyl bis(benzofuran-2-carboxylate) (28): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 48.0 mg, 56% yield. Light yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 (dd, *J* = 18.4, 16.2 Hz, 2H), 7.54 – 7.48 (m, 4H), 7.42 – 7.33 (m, 8H), 6.92 (d, *J* = 8.5 Hz, 2H), 6.50 (d, *J* = 16.0 Hz, 1H), 6.43 (dd, *J* = 16.0, 2.9 Hz, 1H), 6.19 (dd, *J* = 8.3, 3.6 Hz, 1H), 4.57 (dd, *J* = 11.8, 8.5 Hz, 1H), 4.48 (dd, *J* = 12.0, 3.8 Hz, 1H), 3.80 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.5, 166.0, 159.9, 145.4, 134.4, 134.4, 130.4, 128.9, 128.9, 128.2, 128.2, 117.9, 117.6, 114.1, 114.1, 73.3, 66.2, 55.3. HRMS (ESI): m/z: calcd for $C_{27}H_{24}O_5$ $(M+Na)^+$ 451.1516; found 451.1513. Calcd for $C_{27}H_{24}O_5$ $(M+K)^+$ 467.1255; found 467.1252.



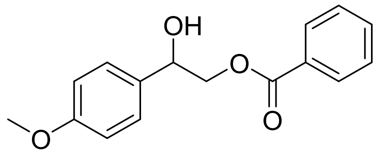
1-(4-methoxyphenyl)ethane-1,2-diyl dinonanoate (29): R_f = 0.25 (Petroleum ether/EtOAc, 50:1). 28.6 mg, 32% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 2H), 6.91 – 6.85 (m, 2H), 5.98 (t, *J* = 6.1 Hz, 1H), 4.29 (d, *J* = 6.1 Hz, 2H), 3.80 (s, 3H), 2.33 (td, *J* = 7.5, 2.7 Hz, 2H), 2.29 (t, *J* = 7.5 Hz, 2H), 1.59 (dd, *J* = 15.4, 7.6 Hz, 4H), 1.36 – 1.17 (m, 20H), 0.90 – 0.86 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.3, 172.4, 159.8, 128.9, 128.1, 114.0, 72.7, 65.8, 55.2, 34.5, 34.2, 31.8, 31.8, 29.2, 29.1, 29.1, 25.0, 24.9, 22.6, 14.0. HRMS (ESI): m/z: calcd for $C_{27}H_{44}O_5$ $(M+Na)^+$ 471.3081; found 471.3079. Calcd for $C_{27}H_{44}O_5$ $(M+K)^+$ 487.2820; found 487.2822.



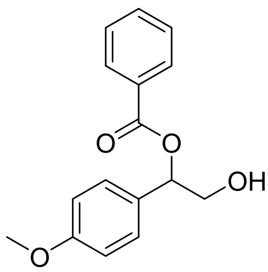
2-hydroxy-1,2-diphenylethyl benzoate (30): [2] R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 26.7 mg, 42% yield (dr = 6.7:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05 (dd, *J* = 60.4, 7.4 Hz, 2H), 7.60 – 7.52 (m, 1H), 7.43 (t, *J* = 7.4 Hz, 2H), 7.30 (t, *J* = 7.7 Hz, 1H), 7.20 (m, 9H), 6.14 (d, *J* = 7.2 Hz, 1H), 5.11 (d, *J* = 7.2 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 165.7, 165.4, 139.5, 139.1, 136.9, 136.5, 133.2, 130.1, 129.7, 129.7, 128.5, 128.4, 128.4, 128.3, 128.2, 128.2, 128.1, 128.1, 127.6, 127.3, 127.1, 127.0, 80.5, 79.5, 77.3, 76.6.



2-hydroxy-2-methyl-1-phenylpropyl acetate (31): [3] R_f = 0.25 (Petroleum ether/EtOAc, 2:1). 15.4 mg, 37% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.25 (m, 5H), 5.60 (s, 1H), 2.13 (s, 3H), 1.24 (s, 3H), 1.18 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 137.2, 128.2, 128.1, 127.9, 81.6, 72.3, 26.3, 25.5, 21.1.



2-hydroxy-2-(4-methoxyphenyl)ethyl benzoate (32): [4] R_f = 0.25 (Petroleum ether/EtOAc, 5:1). 6.5 mg, 12% yield (dr > 20:1). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.0 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.37 (d, *J* = 8.6 Hz, 2H), 6.92 (d, *J* = 8.5 Hz, 2H), 5.06 (dd, *J* = 8.1, 3.5 Hz, 1H), 4.49 (dd, *J* = 11.5, 3.6 Hz, 1H), 4.41 (dd, *J* = 11.5, 8.2 Hz, 1H), 3.81 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.7, 159.6, 133.2, 132.1, 129.9, 129.7, 128.4, 127.5, 114.1, 72.2, 69.8, 55.3.



2-hydroxy-1-(4-methoxyphenyl)ethyl benzoate (33): R_f = 0.25 (Petroleum ether/EtOAc, 5:1). 2.7 mg, 5% yield (dr > 20:1). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 – 8.02 (m, 2H), 7.57 (t, *J* = 7.3

Hz, 1H), 7.45 (t, J = 6.4 Hz, 2H), 7.41 – 7.34 (m, 2H), 6.97 – 6.86 (m, 2H), 6.12 – 5.99 (m, 1H), 4.10 – 4.00 (m, 1H), 3.97 – 3.88 (m, 1H), 3.80 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.2, 159.8, 133.2, 130.1, 129.7, 129.2, 128.4, 128.10, 114.2, 66.1, 55.3. HRMS (ESI): m/z: calcd for $C_{16}H_{16}O_4$ $(M+Na)^+$ 295.0941; found 295.0949. Calcd for $C_{16}H_{16}O_4$ $(M+K)^+$ 311.0680; found 311.0686.

8. References

1 J. Huang, L. Ouyang, J. Li, J. Zheng, W. Yan, W. Wu, H. Jiang, *Org. Lett.*, **2018**, *20*, 5090–5093.

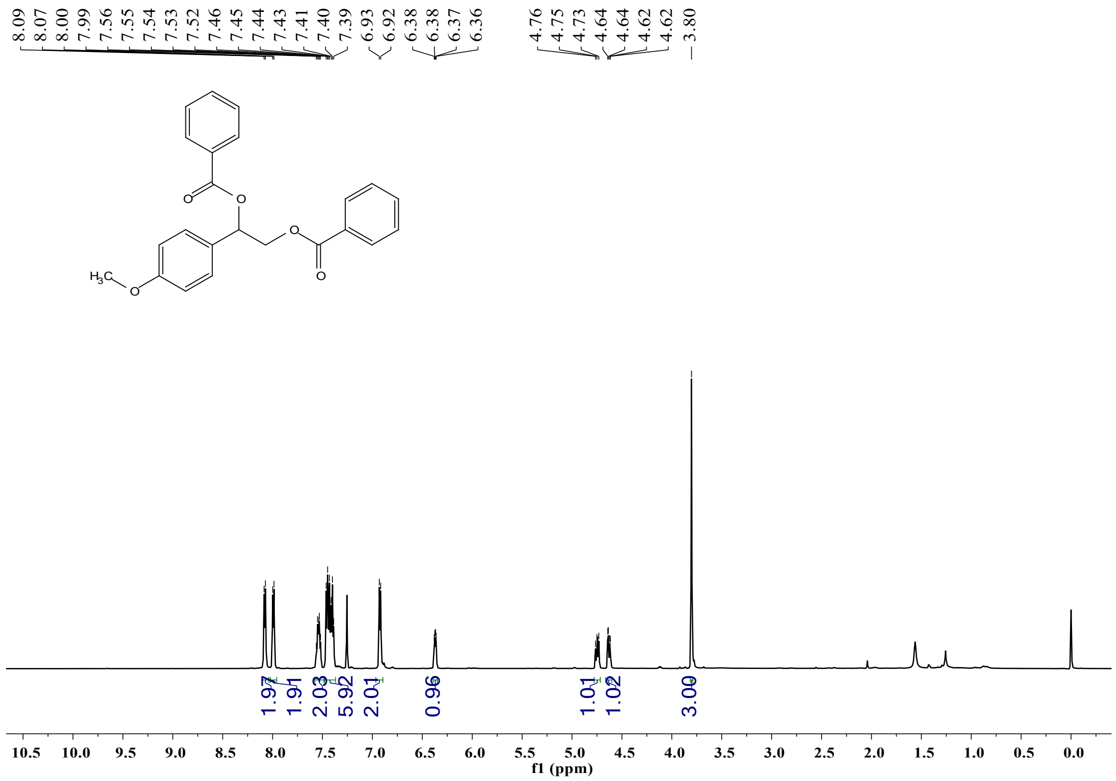
2 T. Arai, K. Sakagami, *Eur. J. Org. Chem.*, **2012**, *6*, 1097–1100.

3 E. Ghera, *J. Org. Chem.*, **1970**, *35*, 660–666.

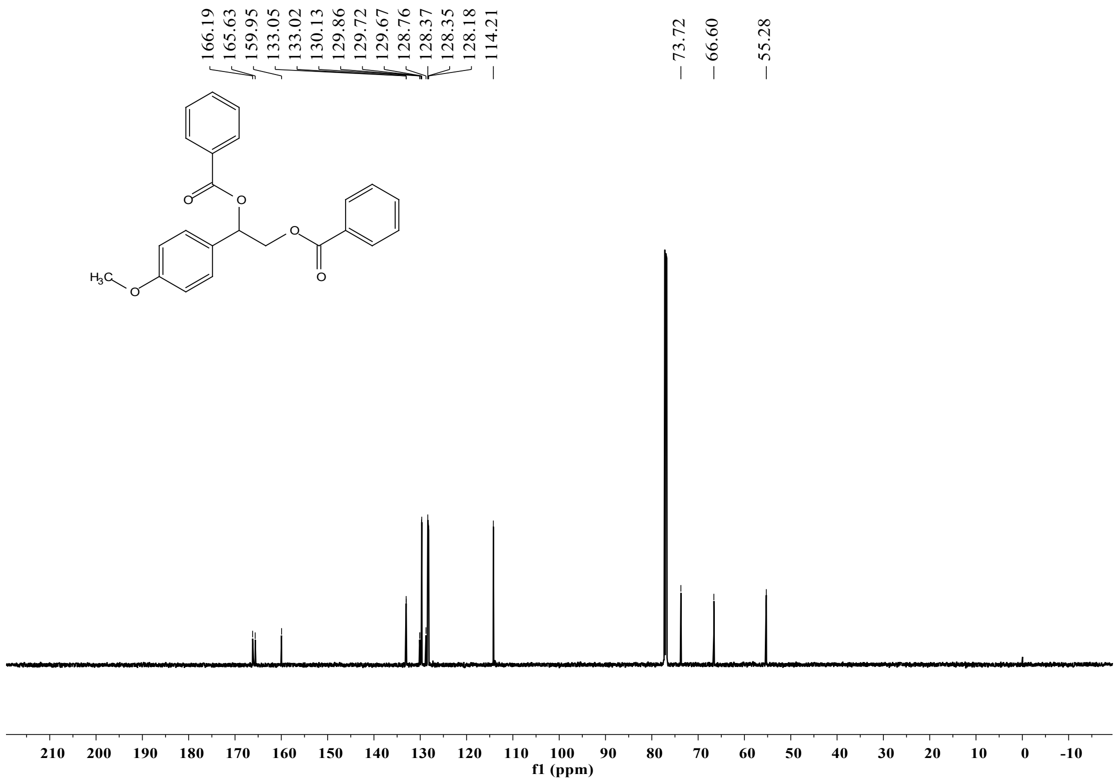
4 B. Liu, J. Yan, R. Huang, W. Wang, Z. Jin, G. Zanoni, P. Zheng, S. Yang, Y. R. Chi, *Org. Lett.*, **2018**, *20*, 3447-3450.

9. NMR spectra of products and by-products

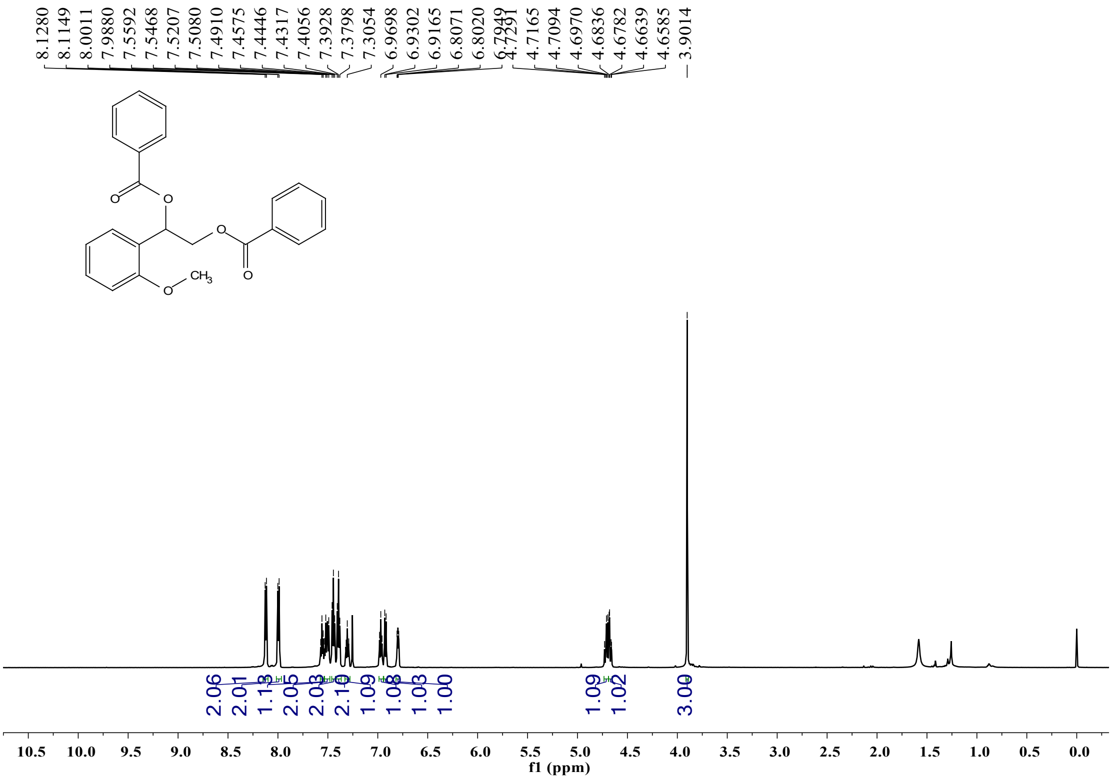
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 1



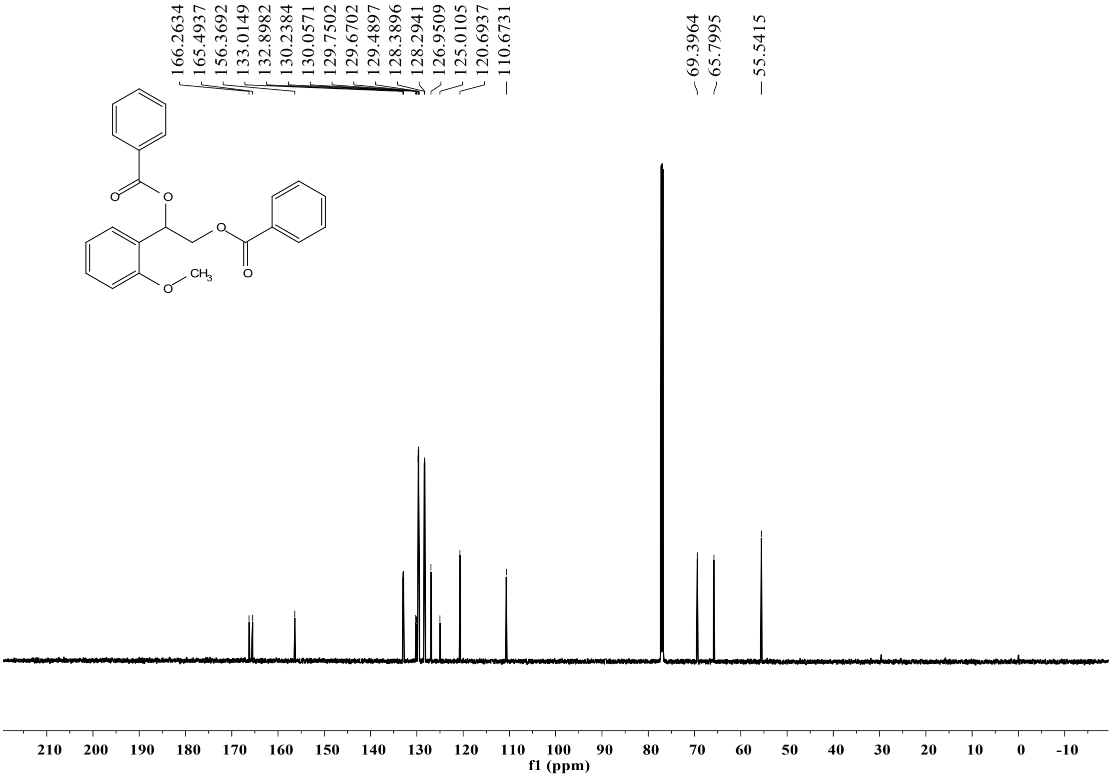
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 1



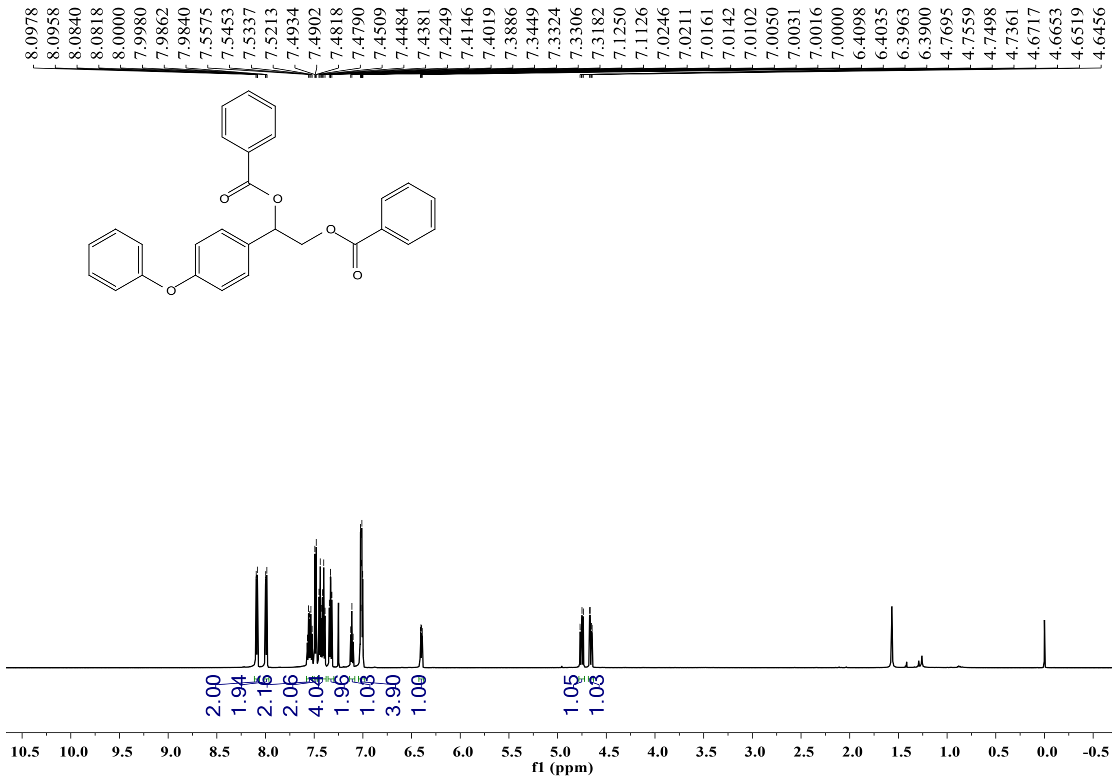
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 2



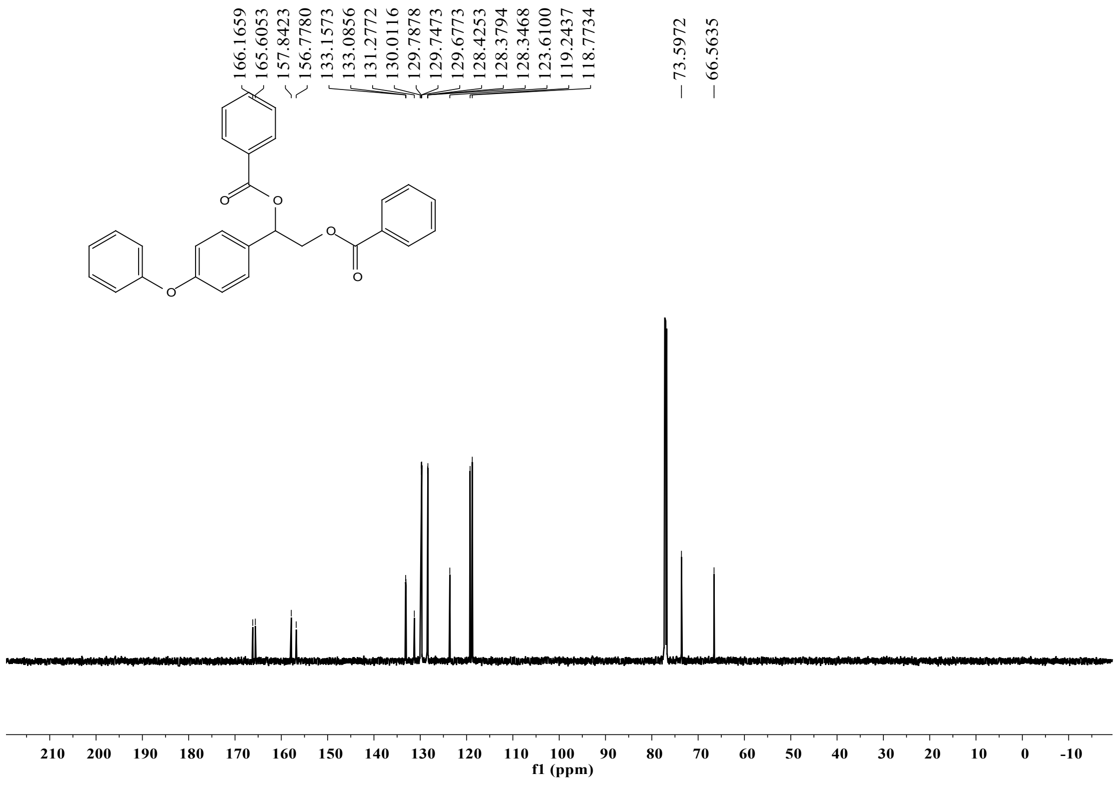
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 2



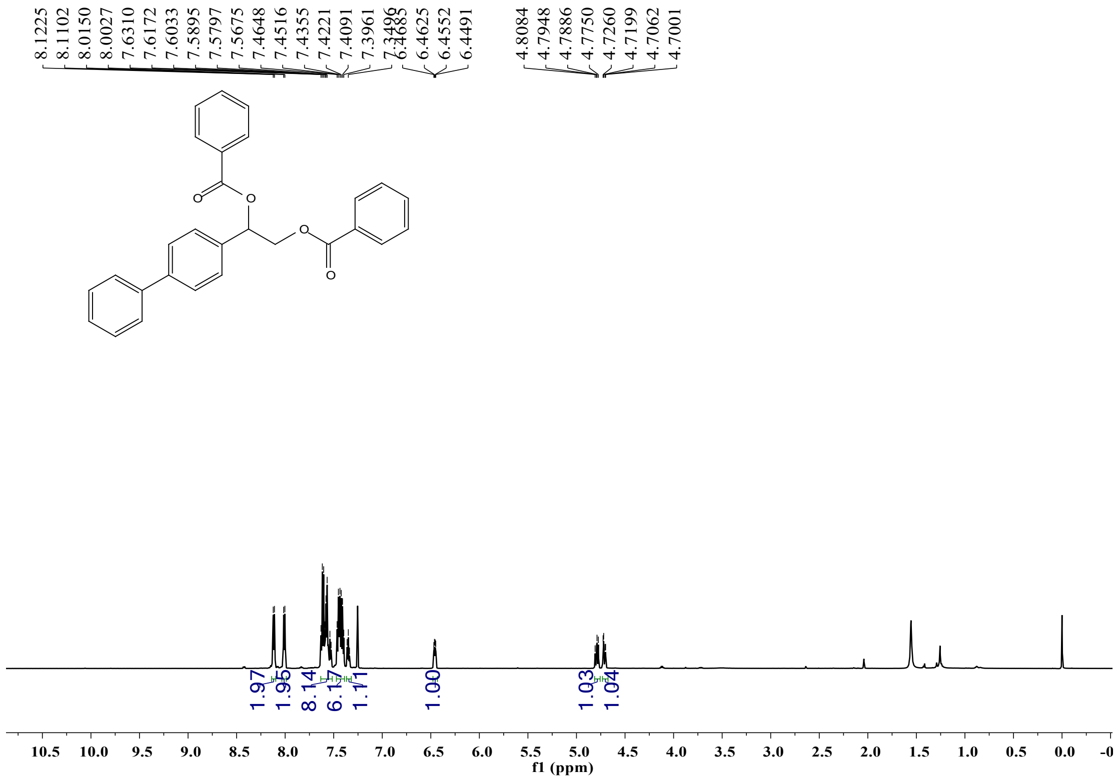
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 3



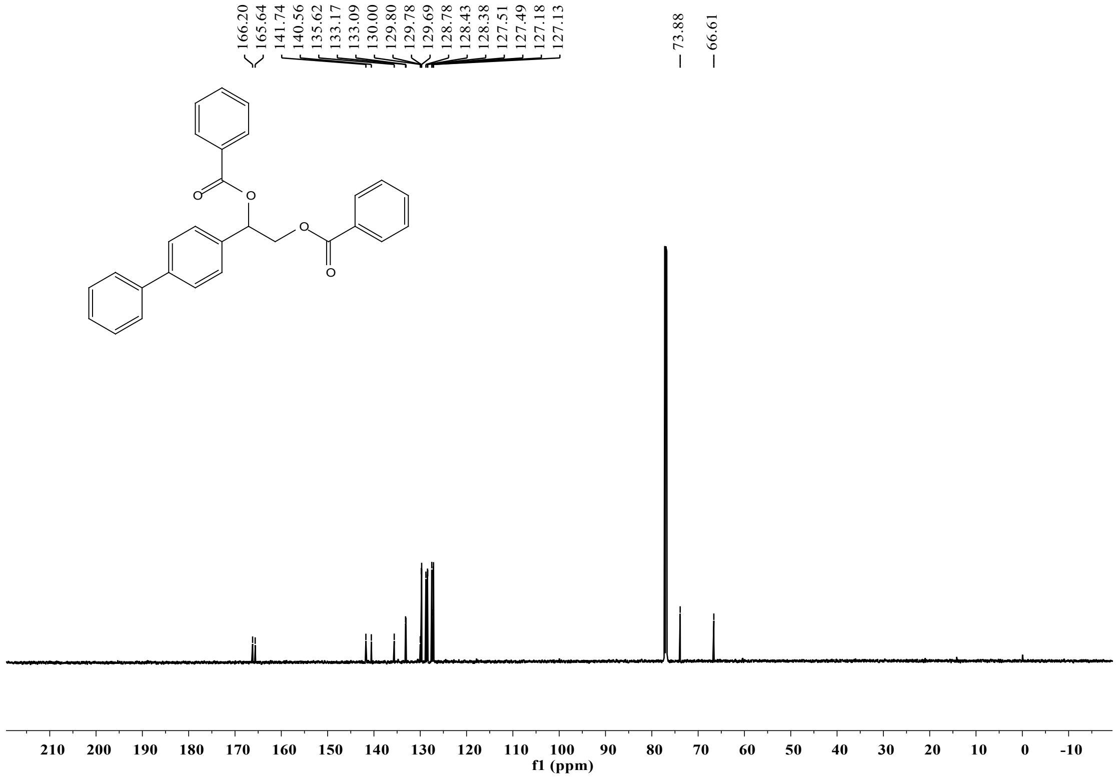
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 3



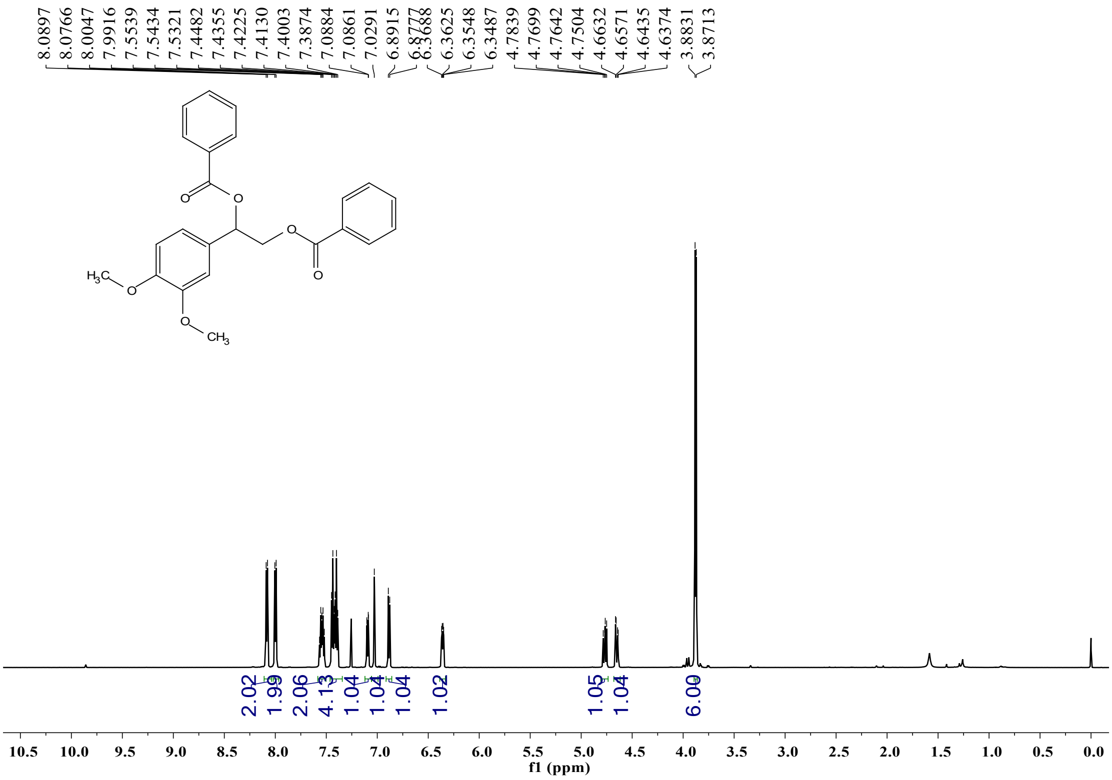
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 4



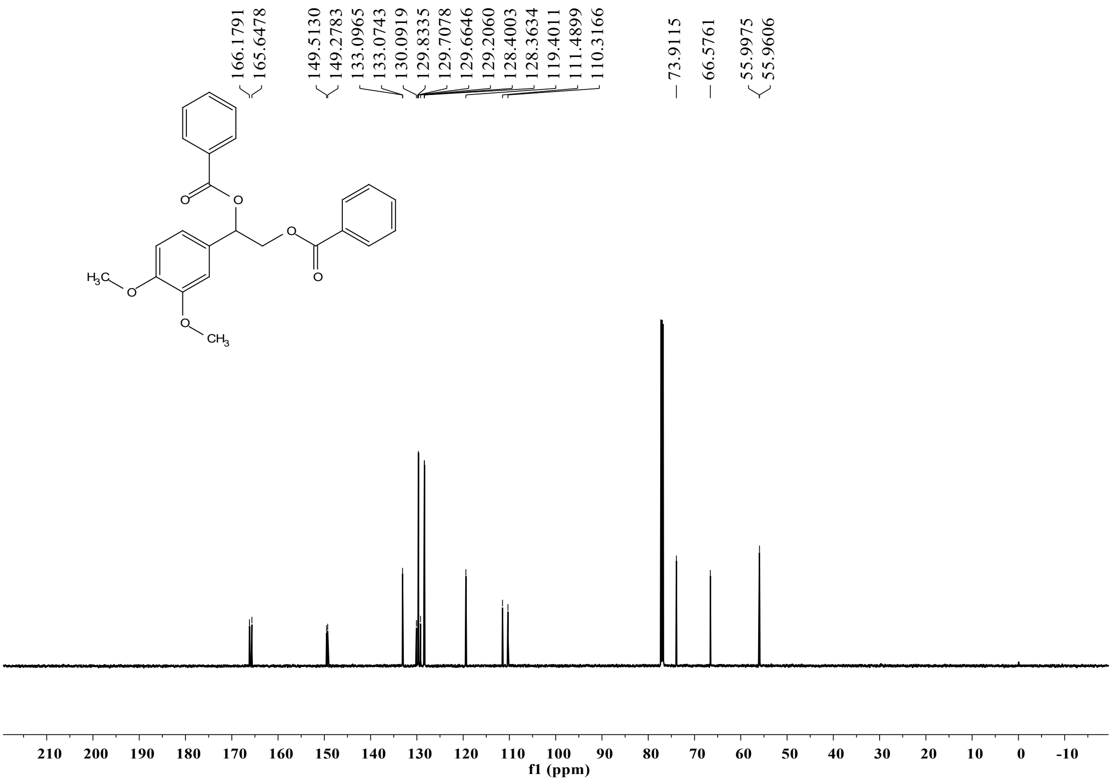
^{13}C-NMR Spectrum (151 MHz, CDCl$_3$) of 4



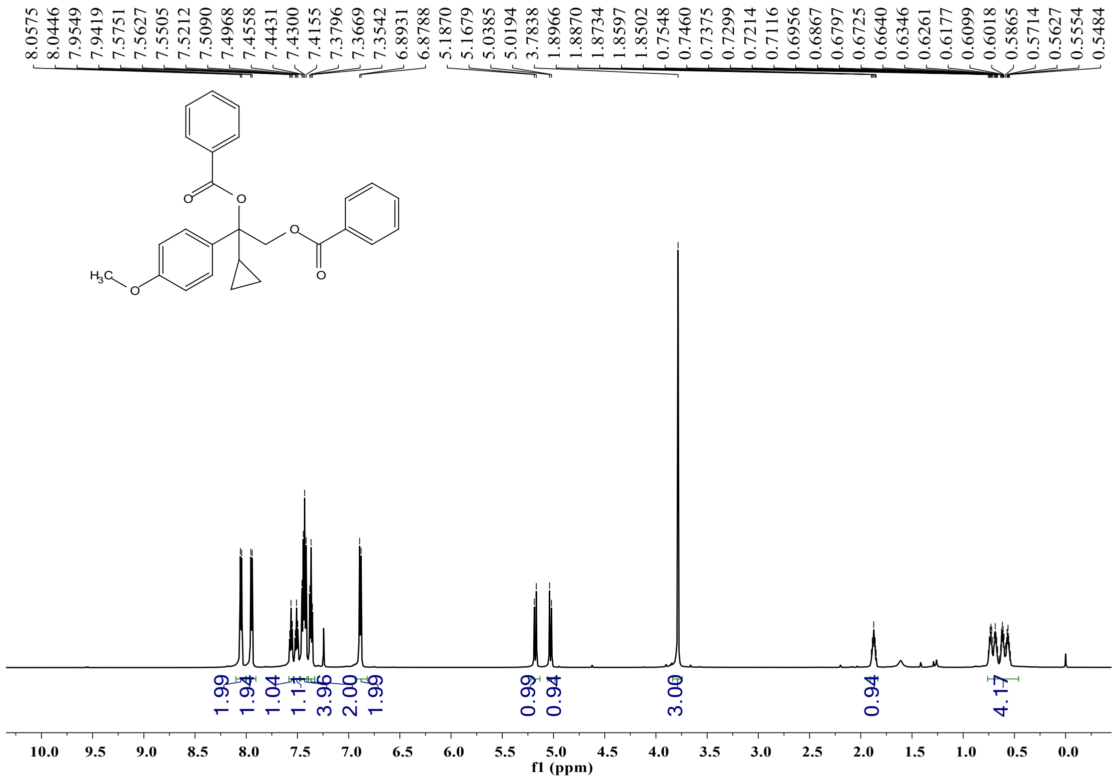
^{1}H-NMR Spectrum (600 MHz, CDCl$_3$) of 5



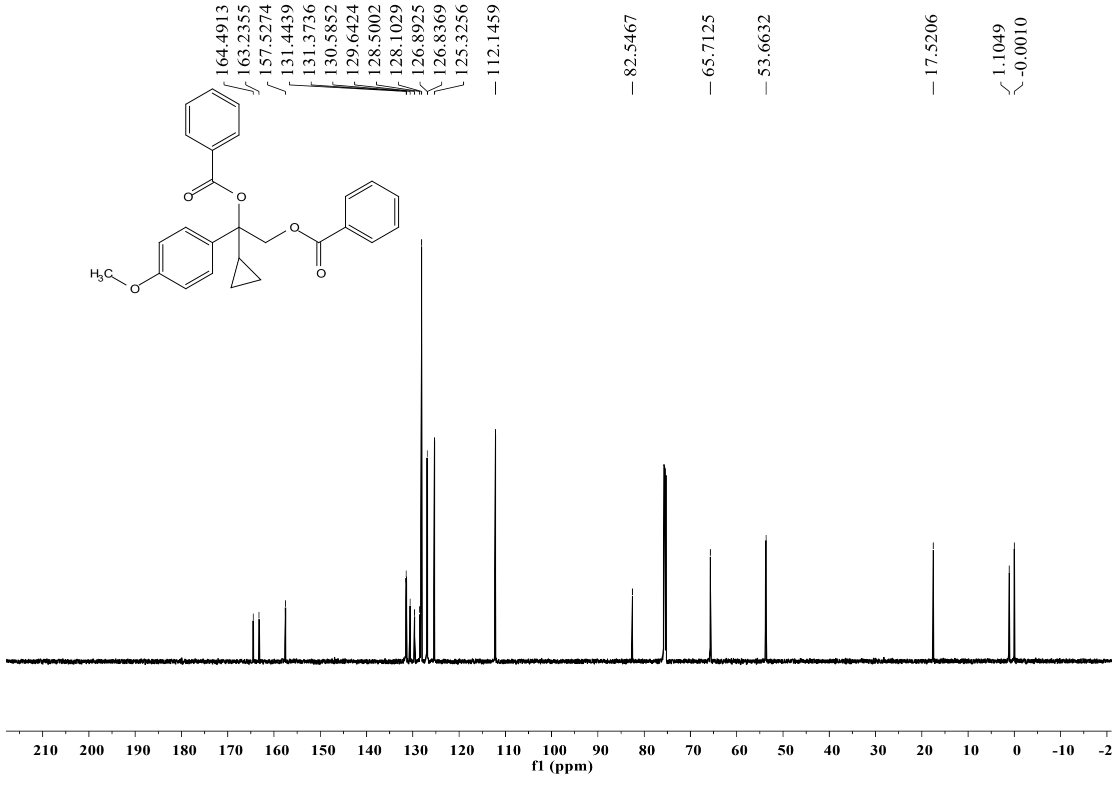
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 5



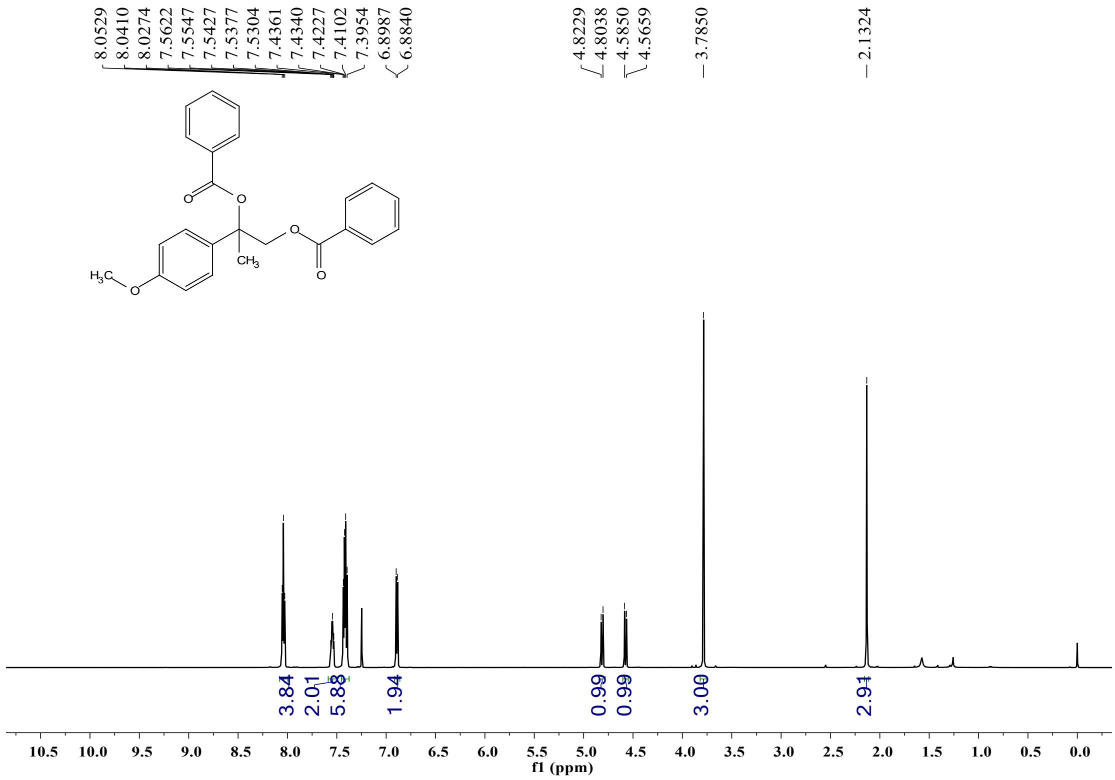
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 6



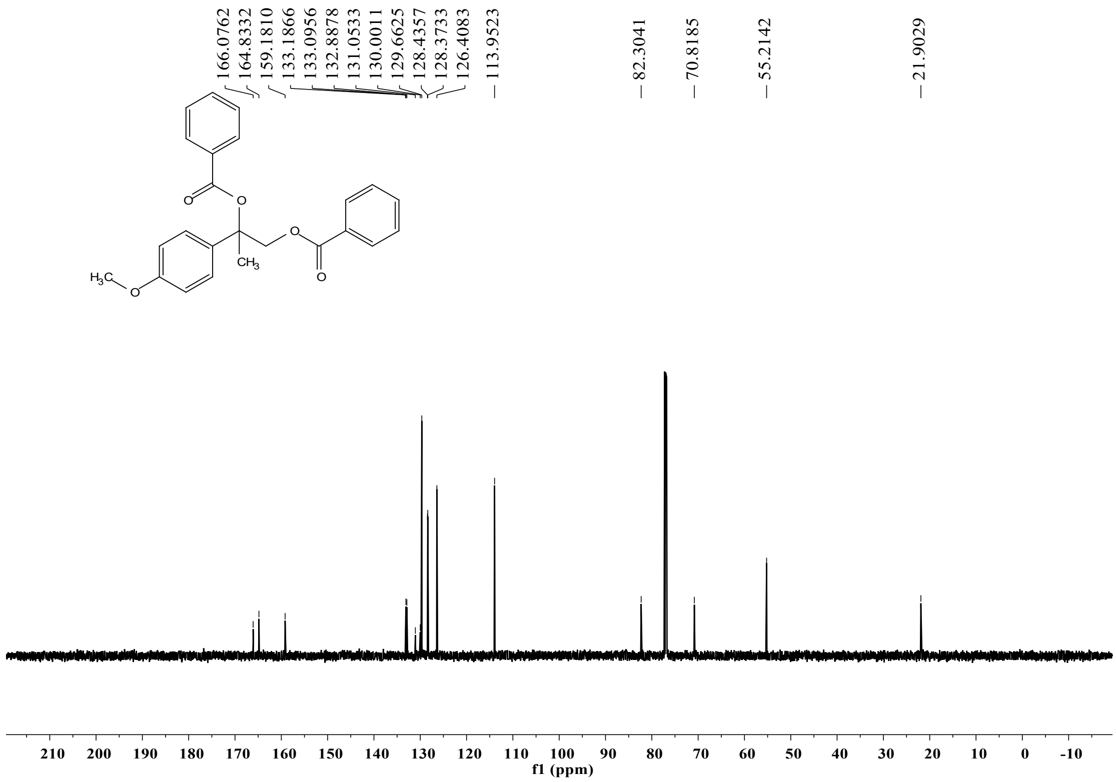
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 6



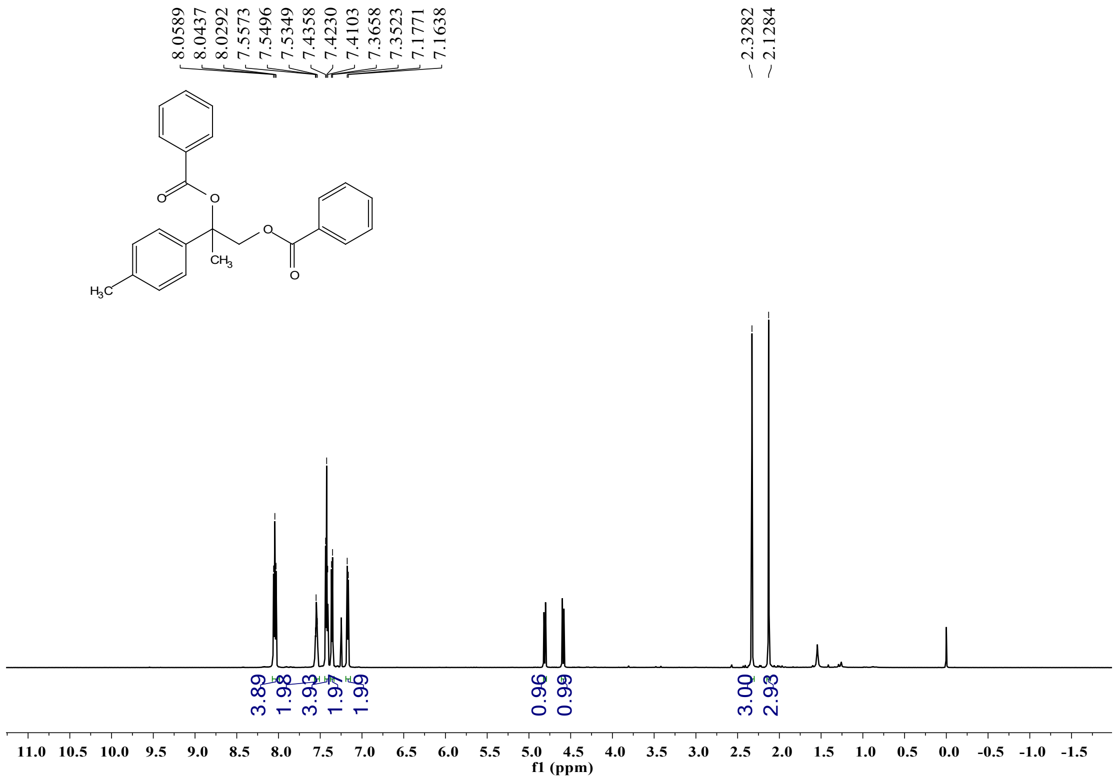
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 7



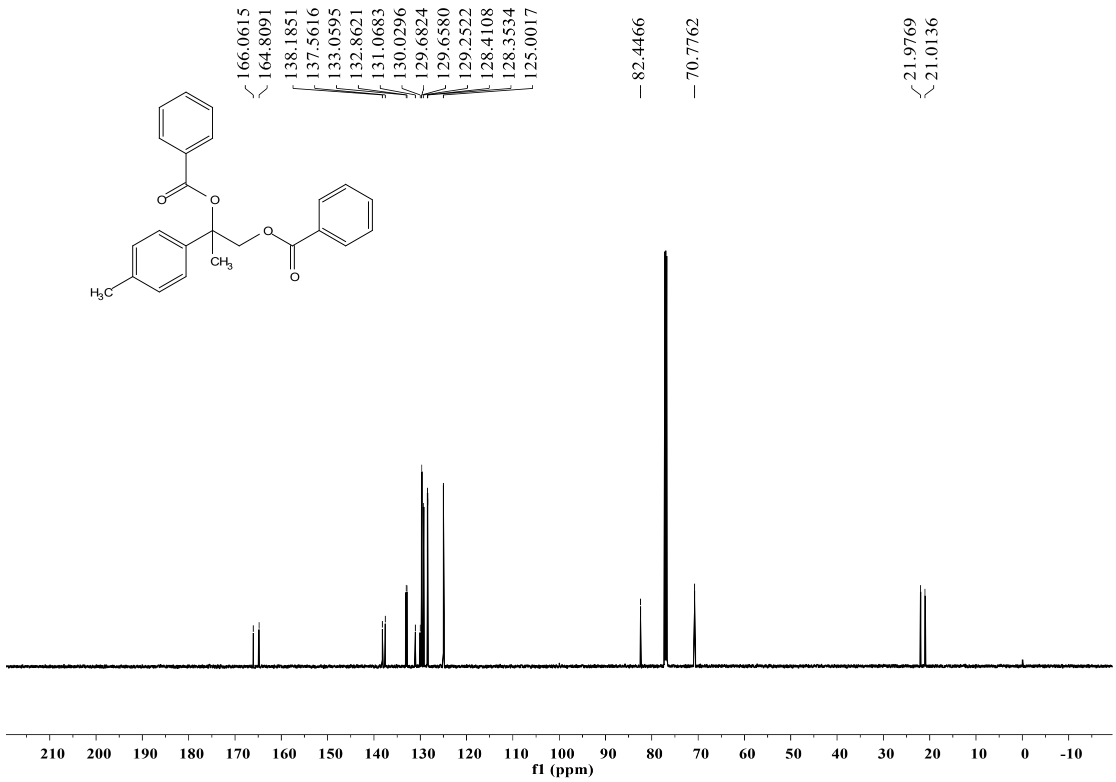
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 7



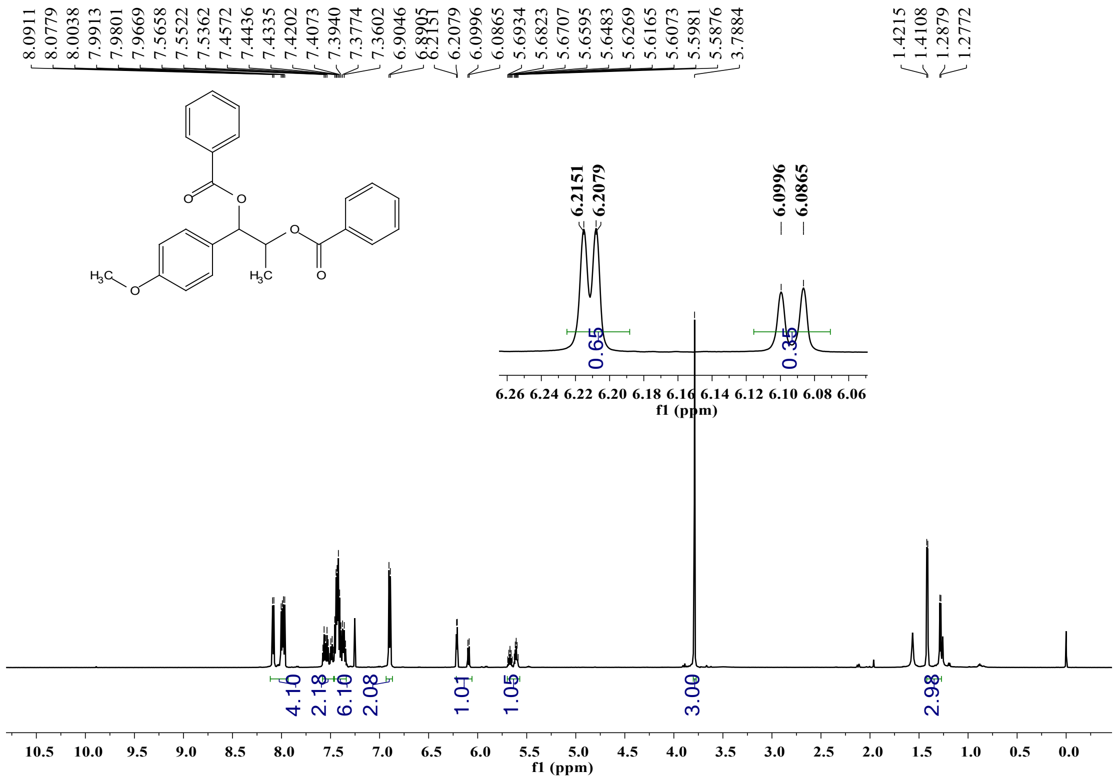
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 8



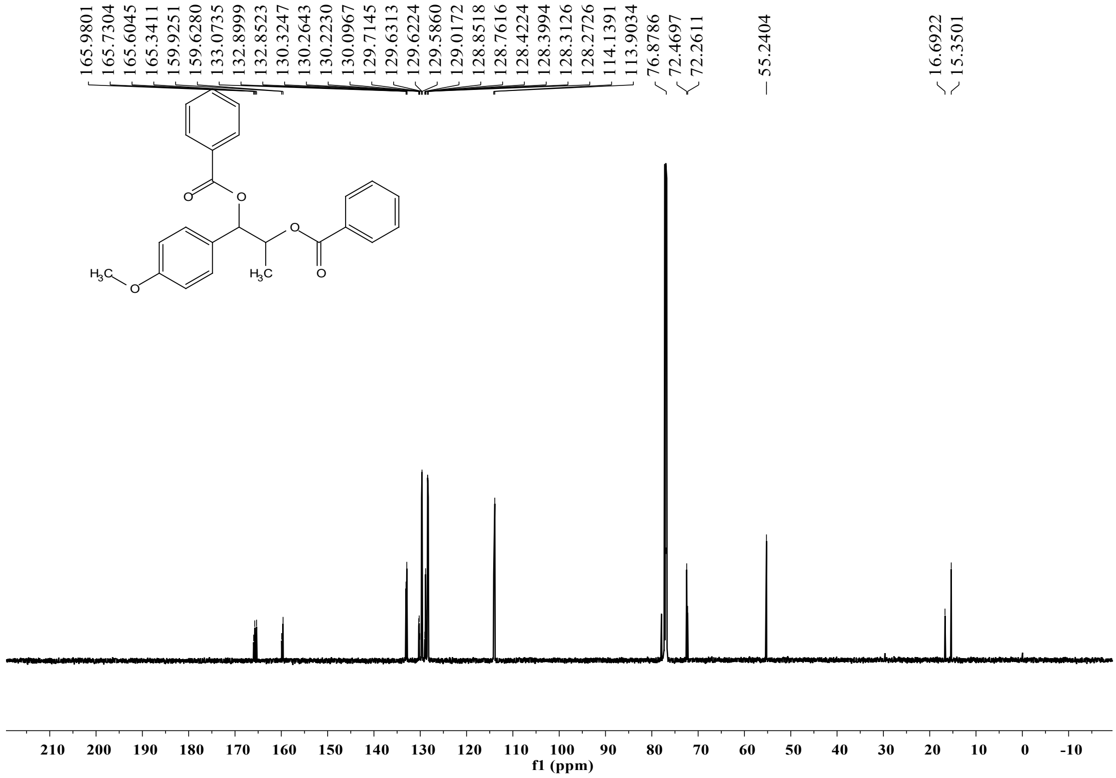
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 8



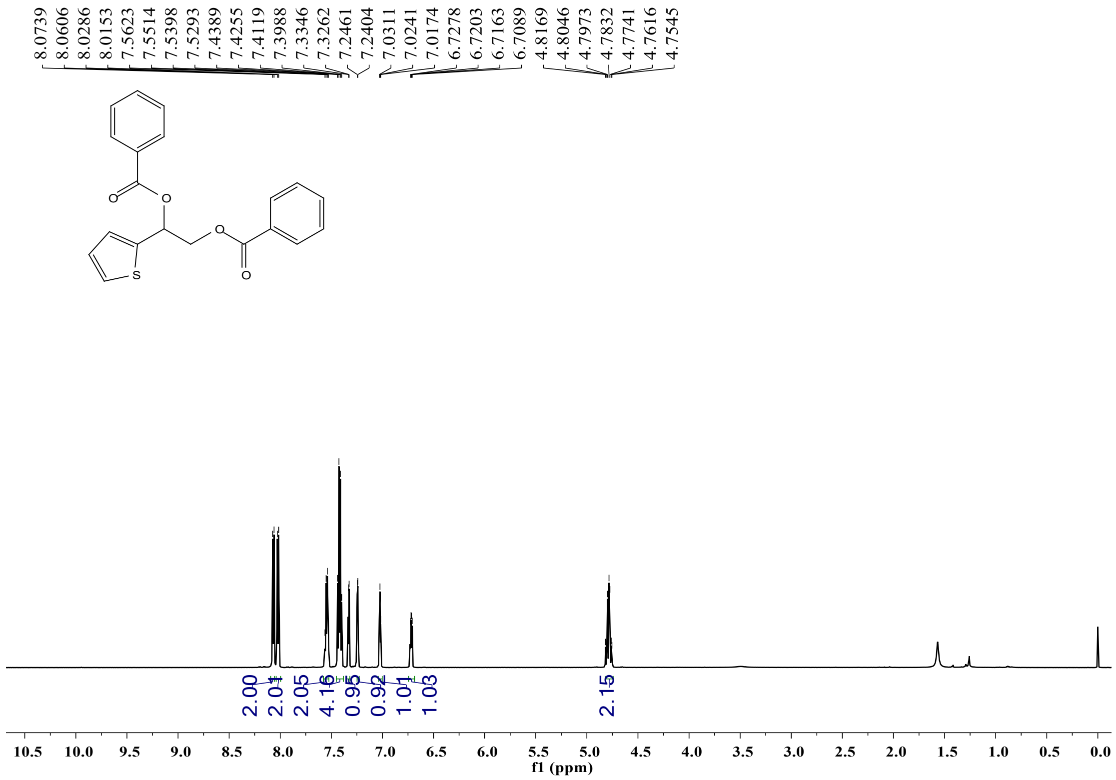
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 9



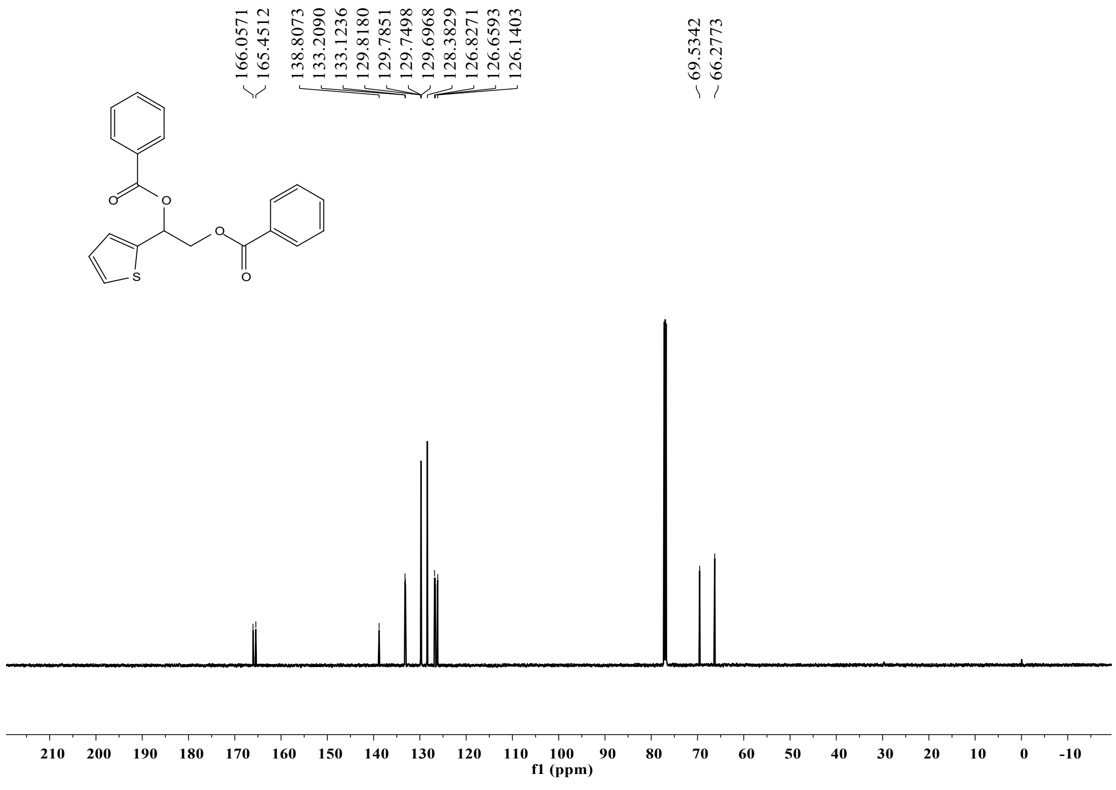
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 9



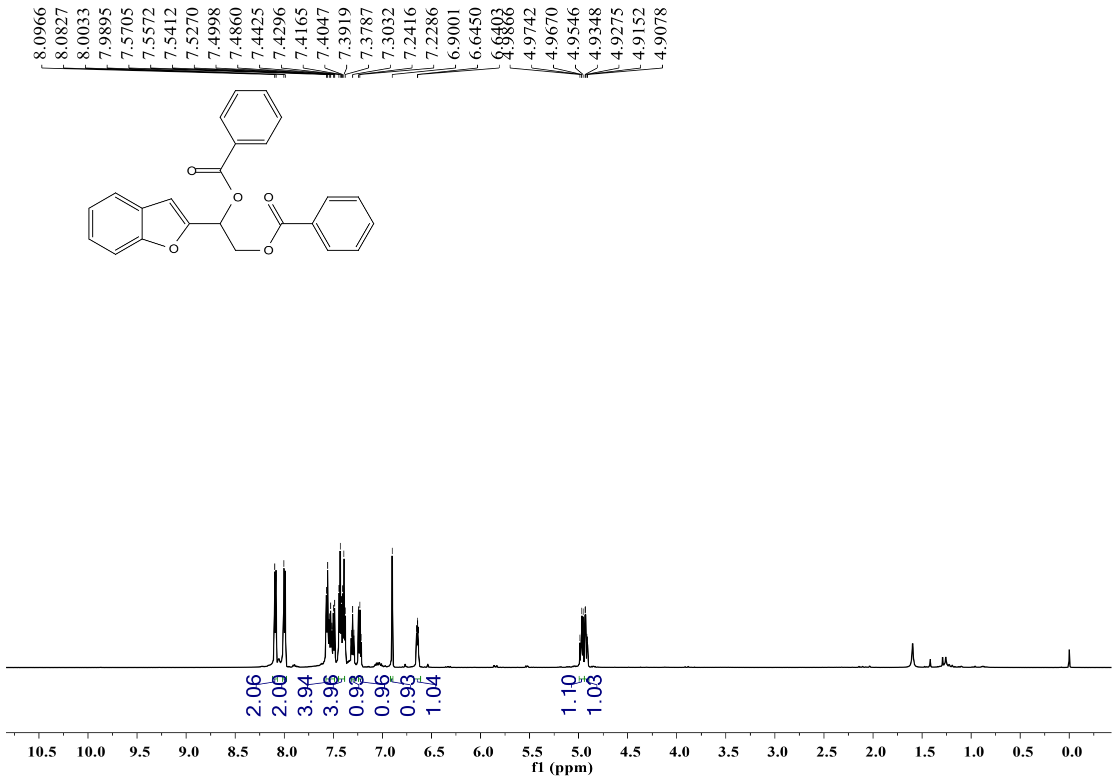
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 10



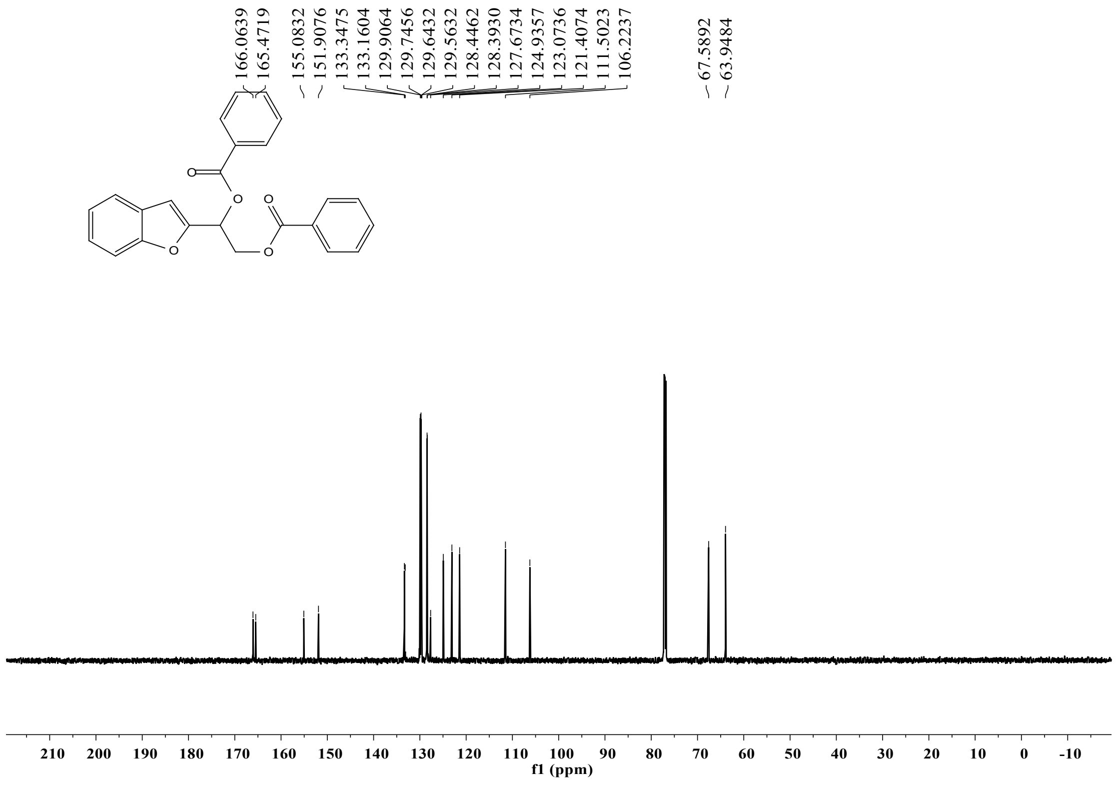
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 10



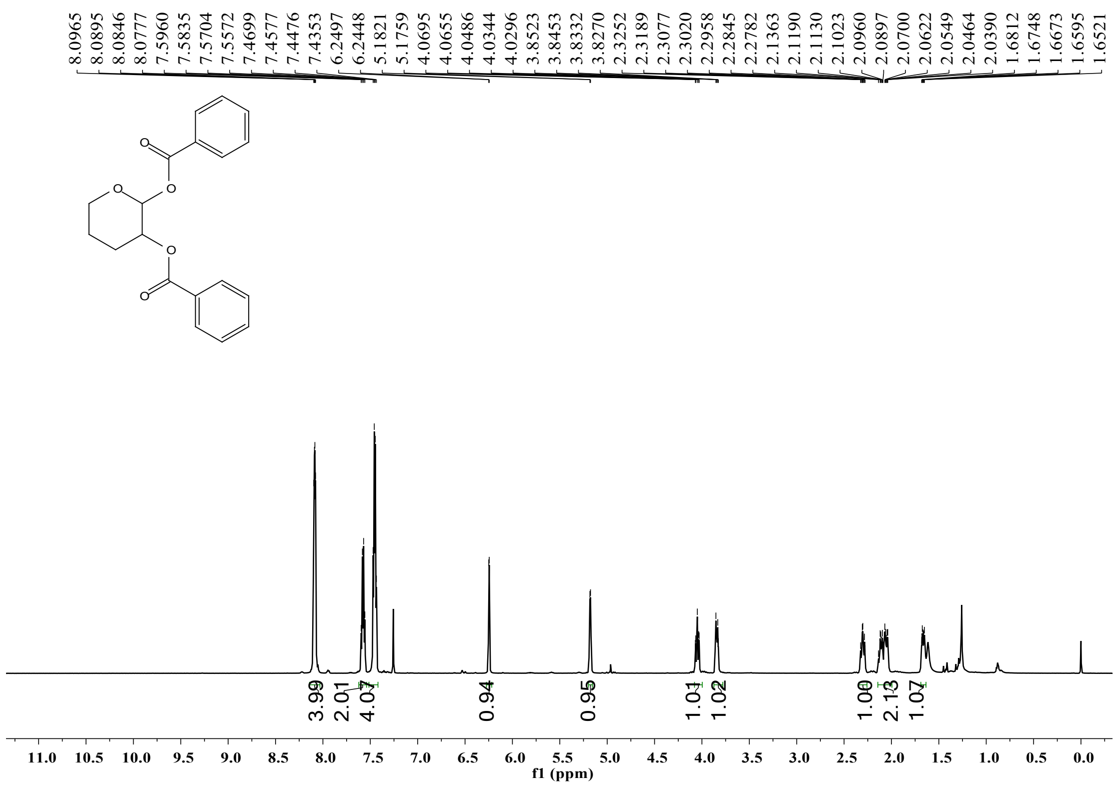
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 11



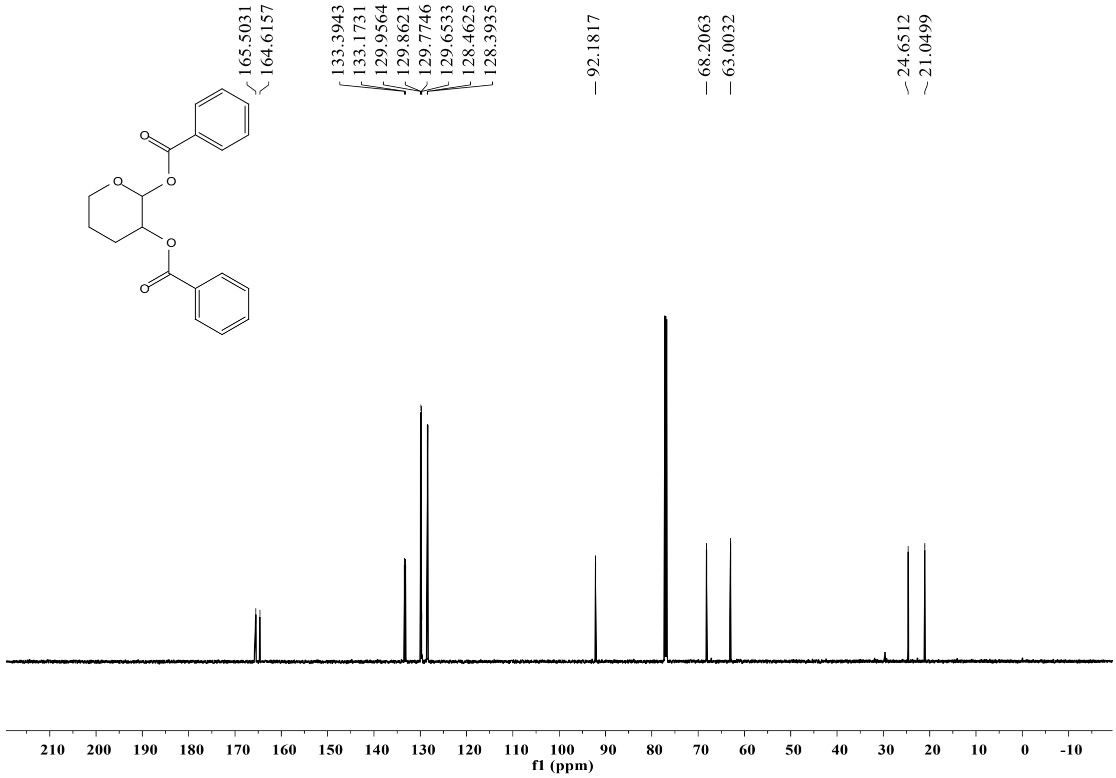
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 11



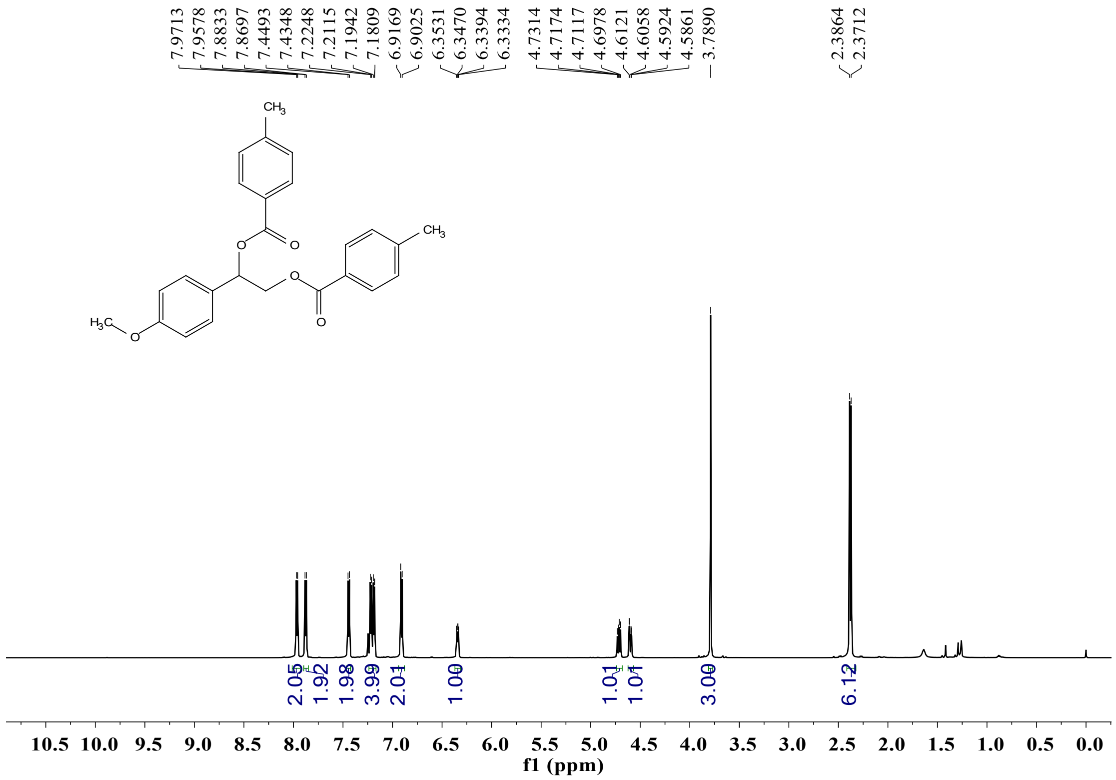
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 12



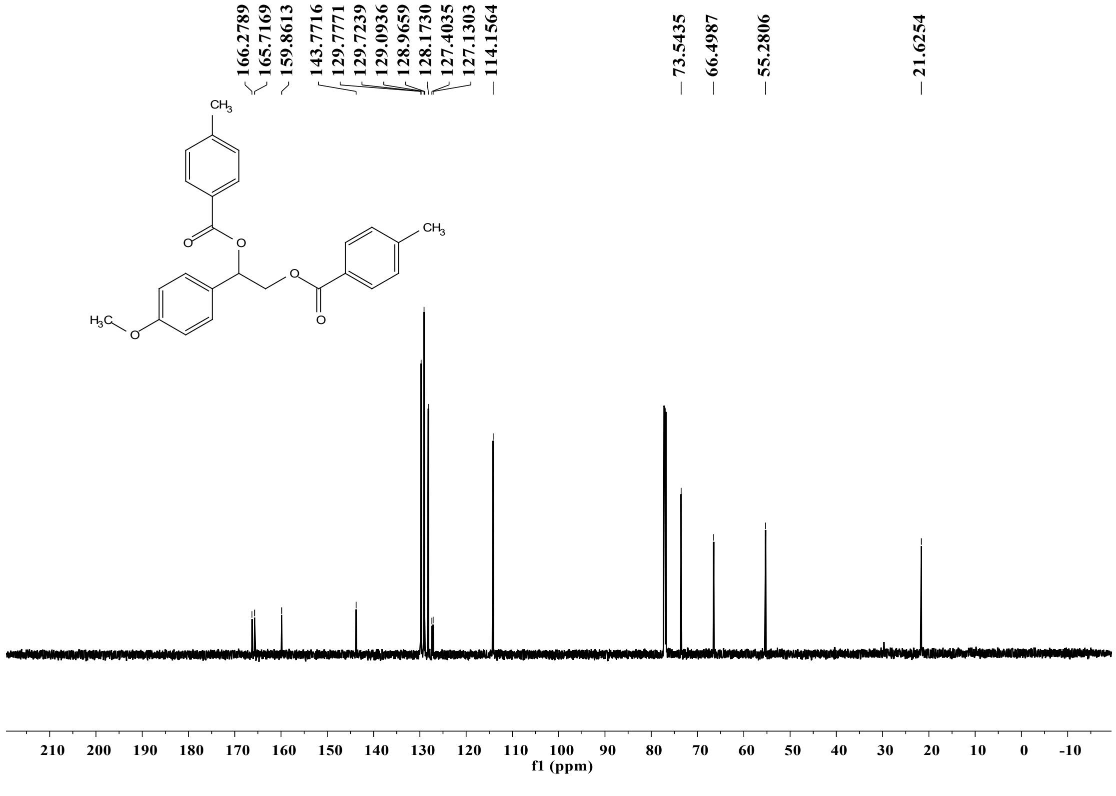
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 12



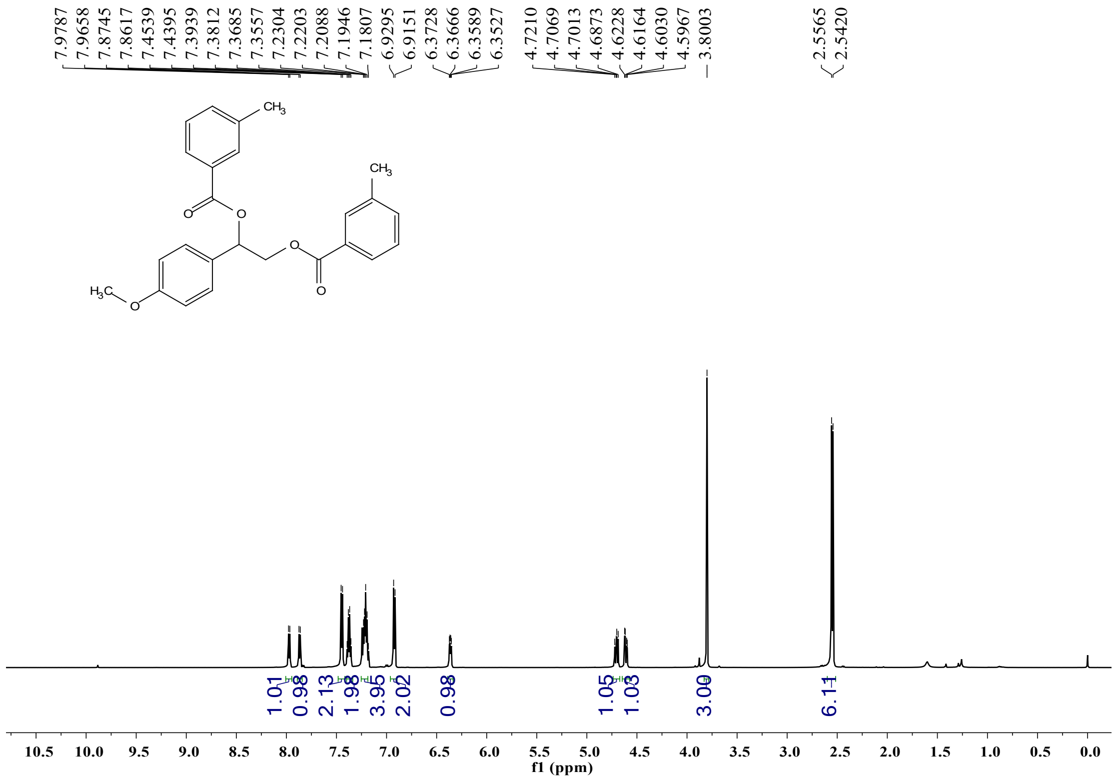
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 13



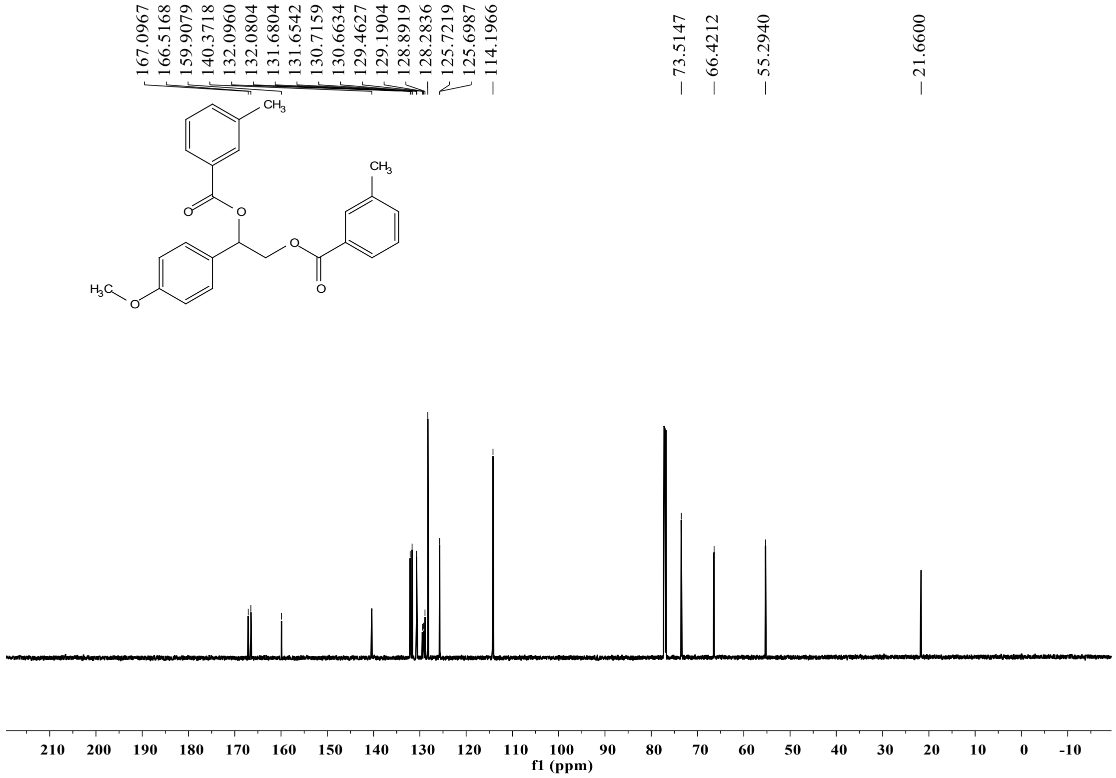
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 13



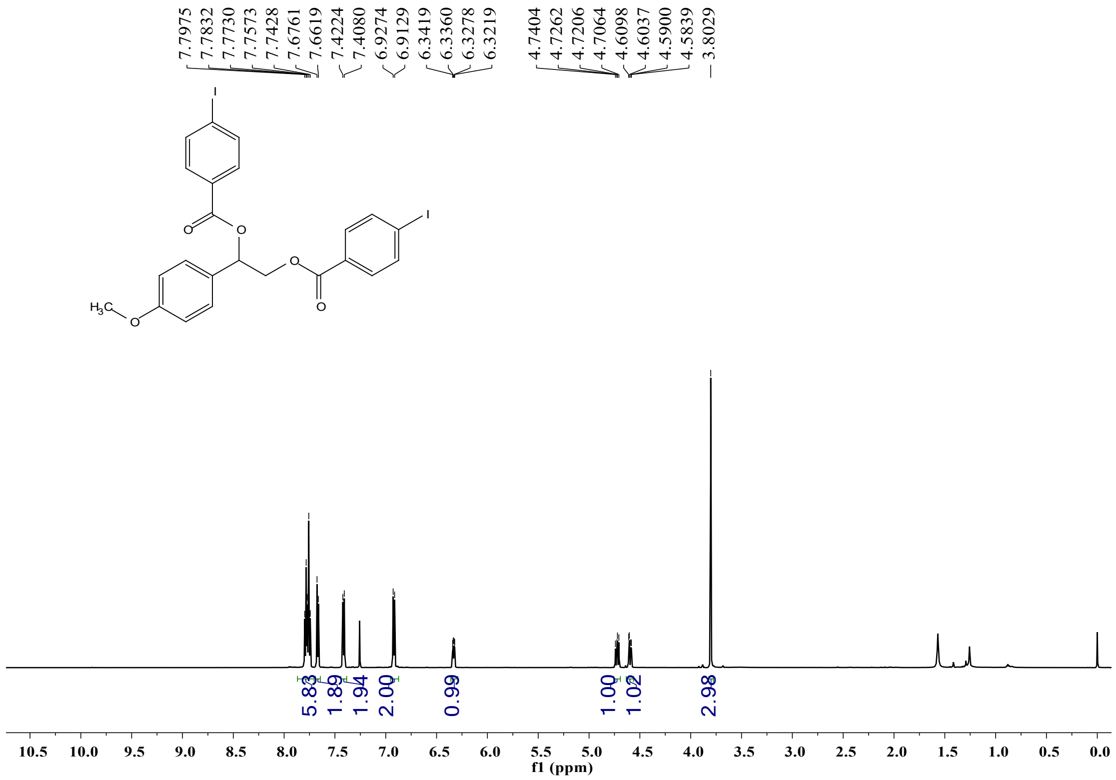
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 14



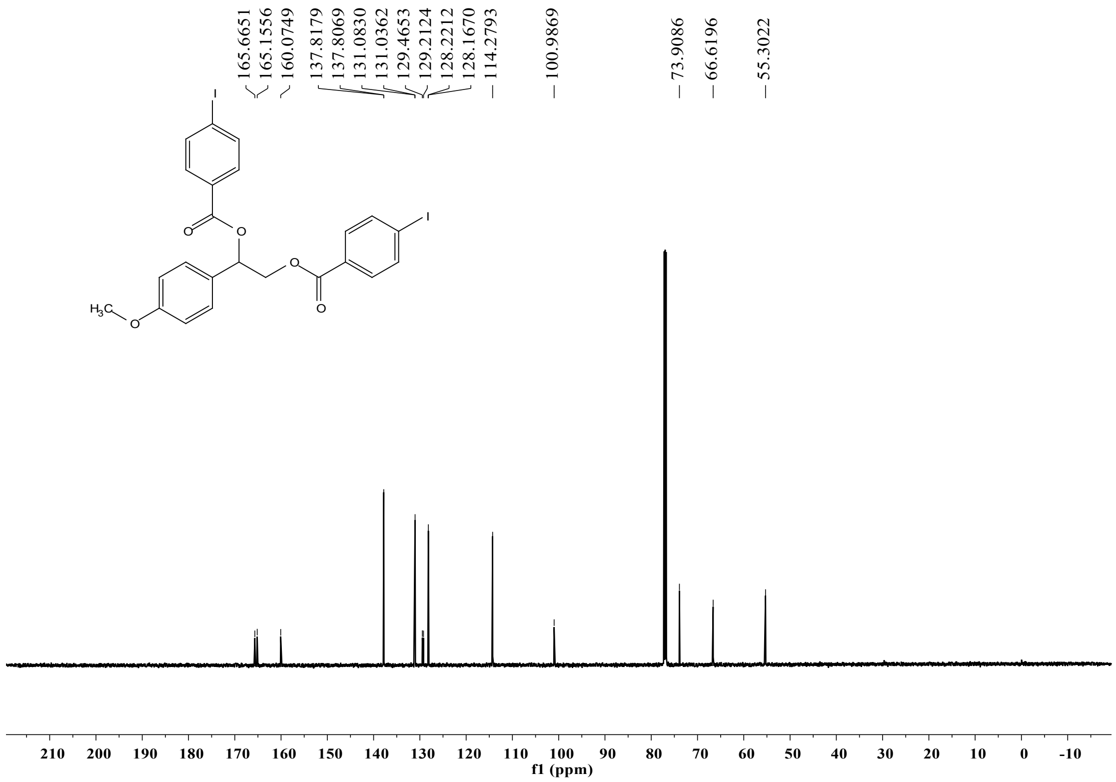
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 14



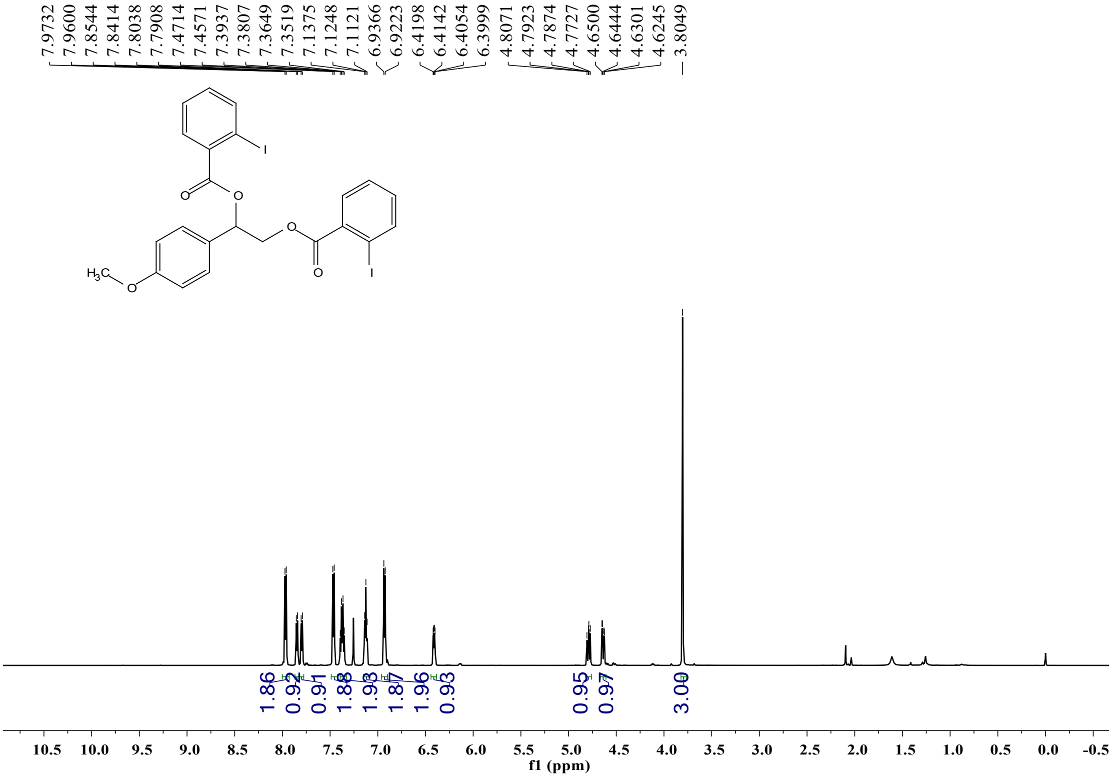
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 15



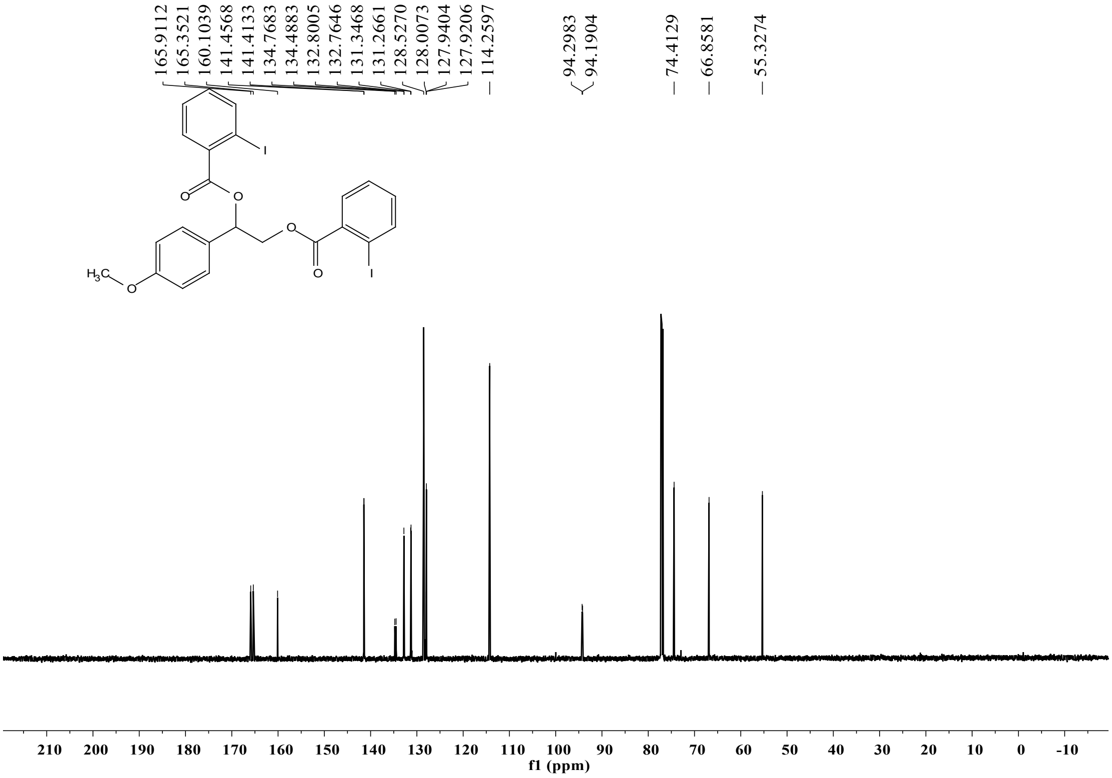
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 15



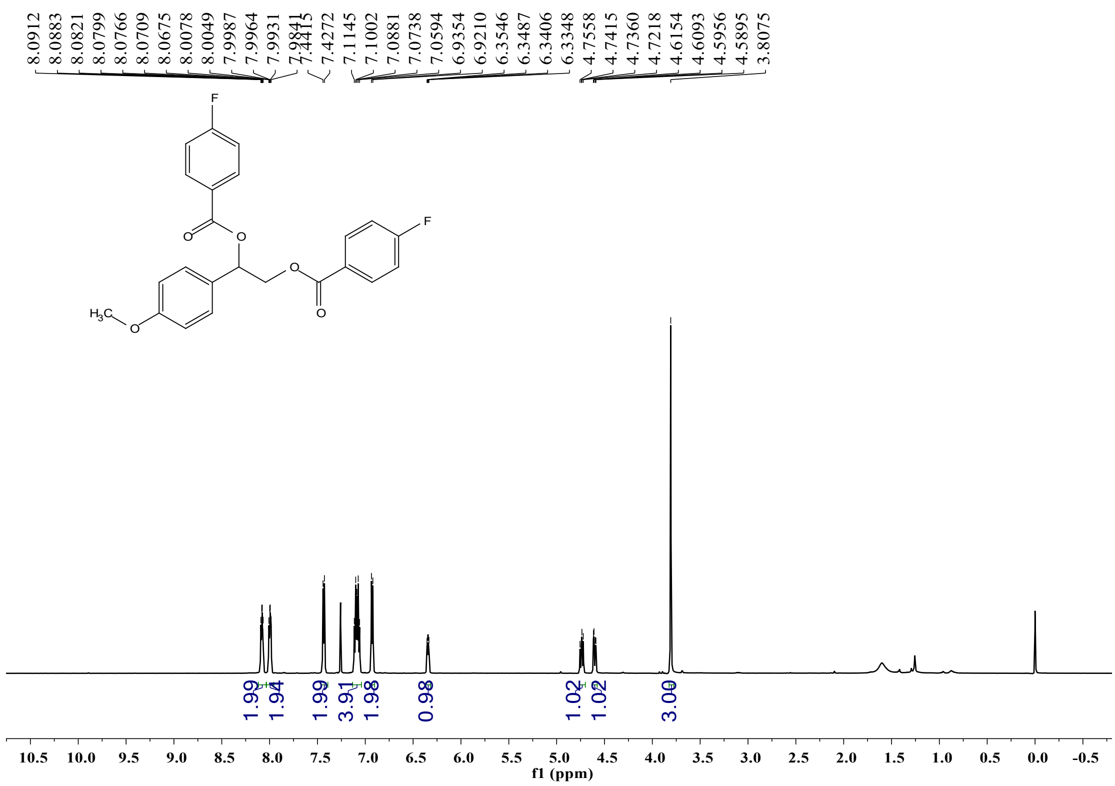
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 16



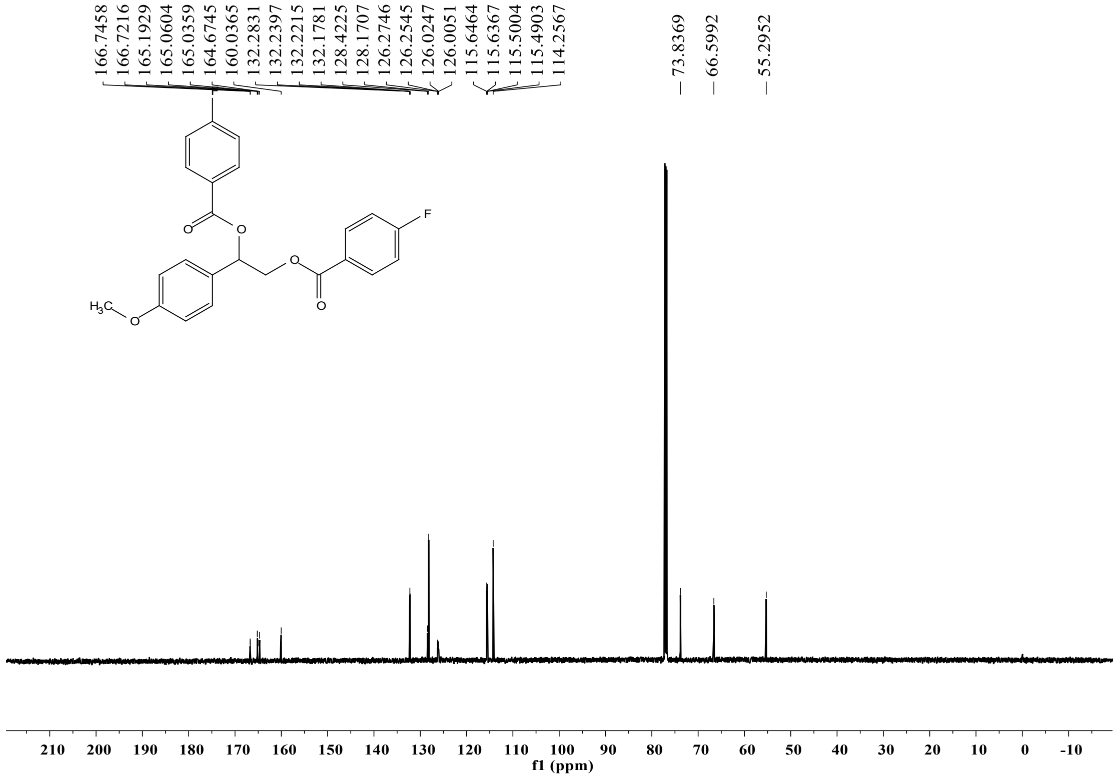
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 16



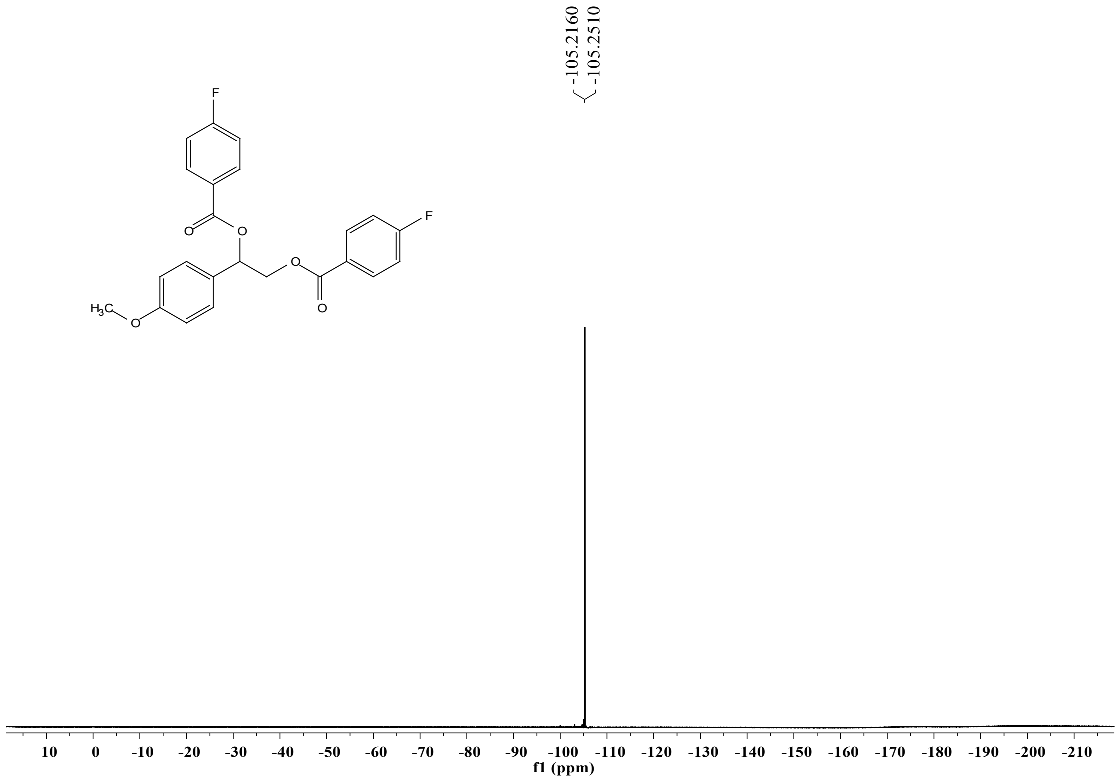
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 17



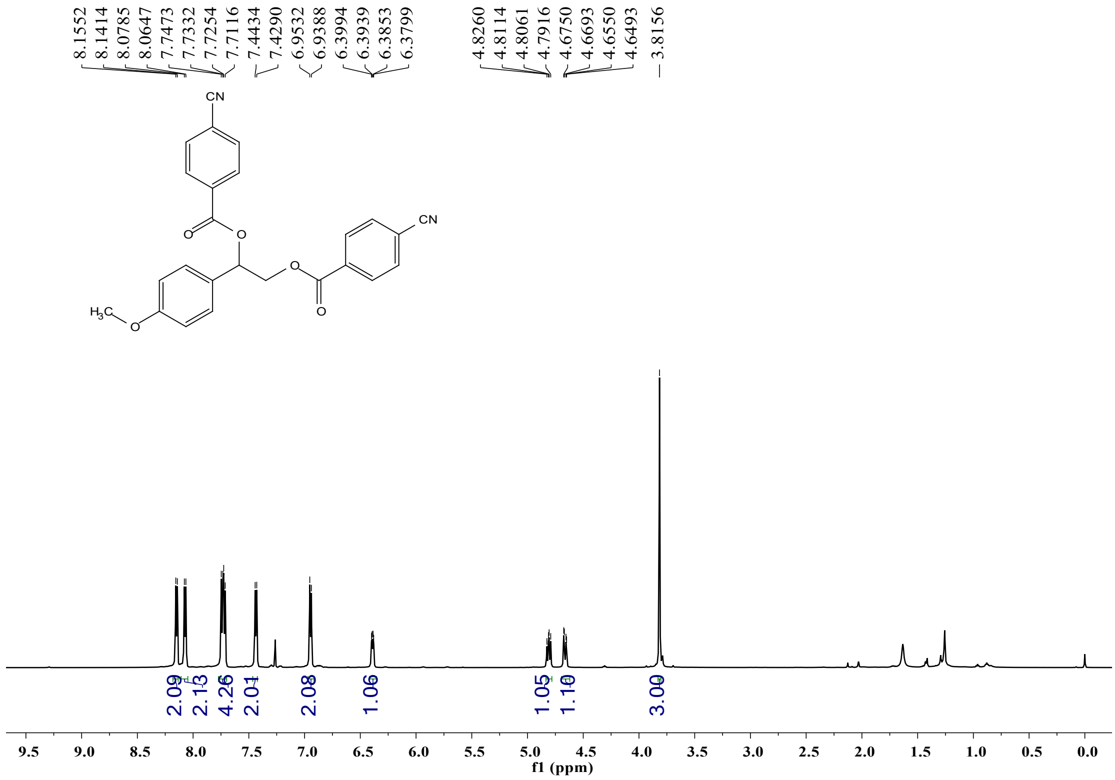
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 17



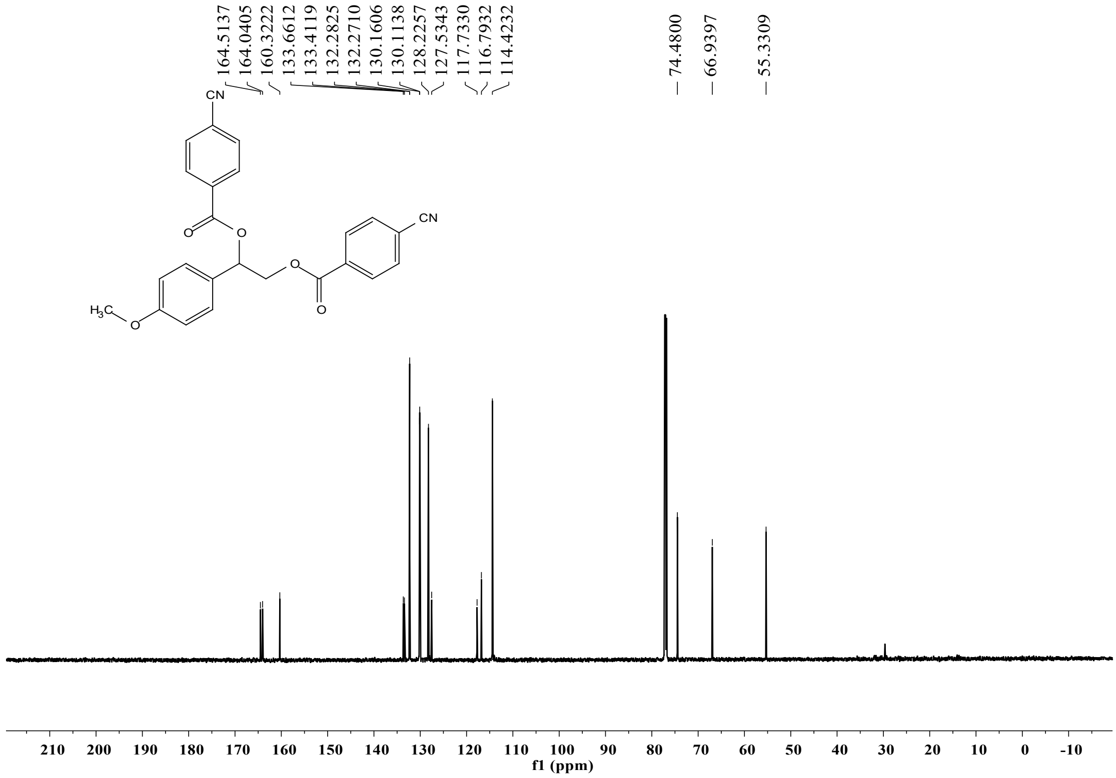
^{19}F-NMR Spectrum (565 MHz, $CDCl_3$) of 17



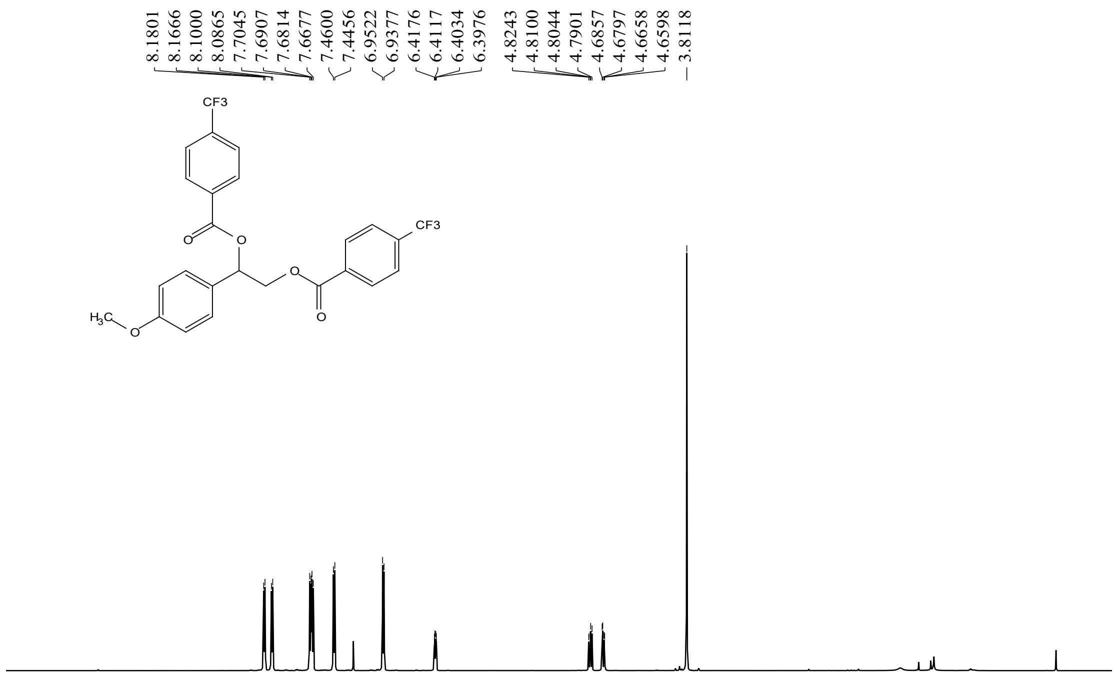
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 18



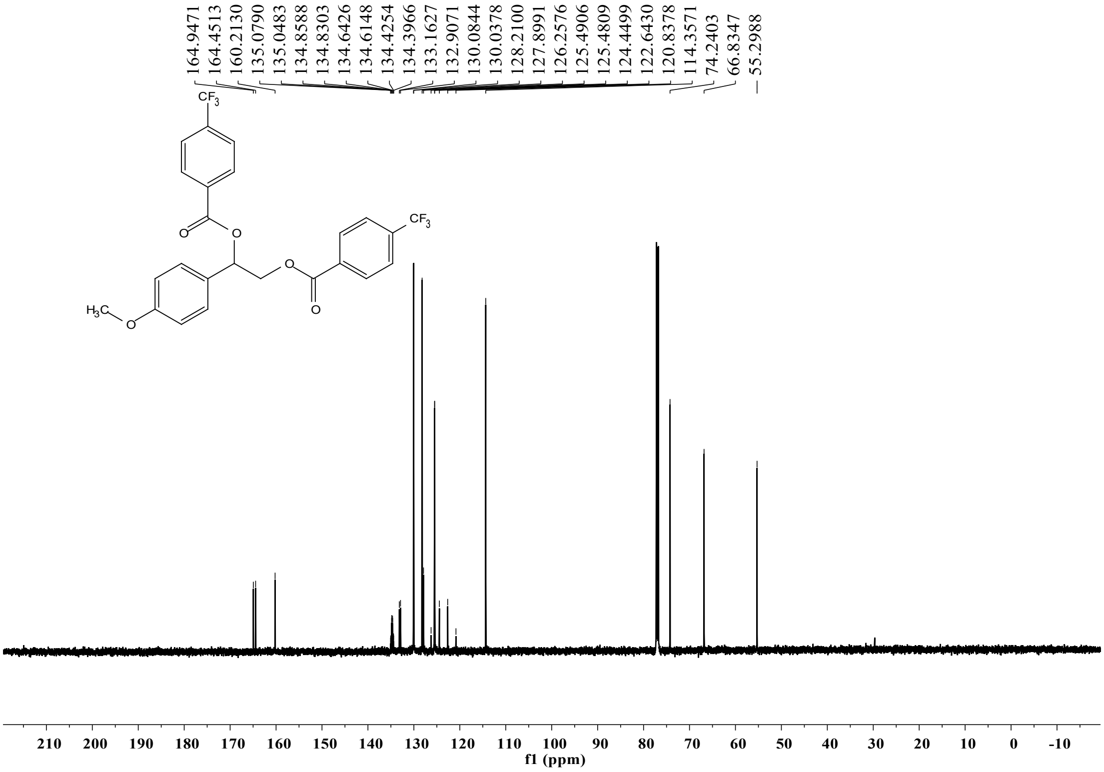
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 18



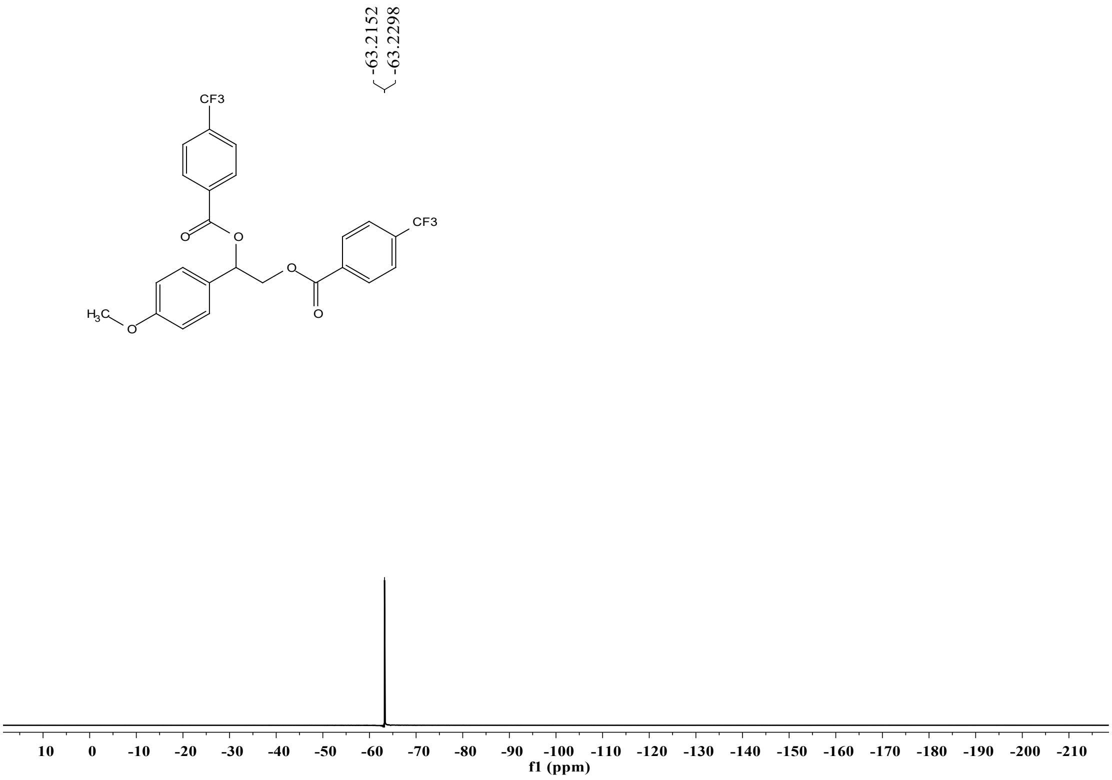
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 19



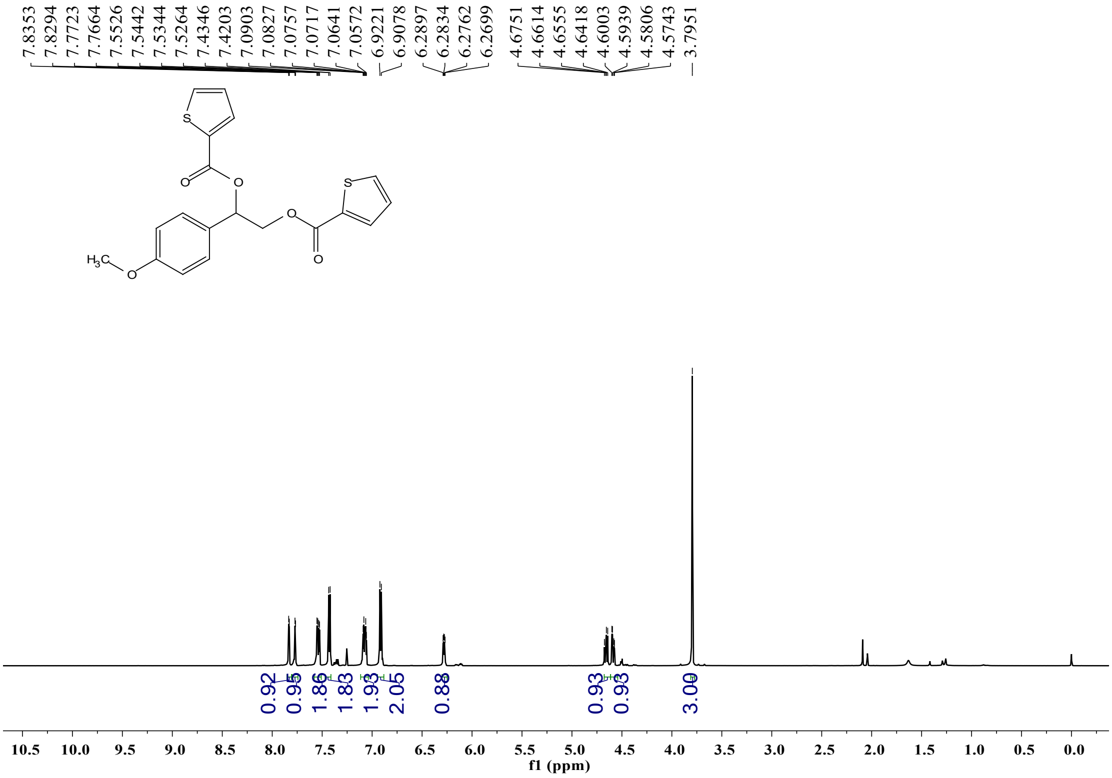
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 19



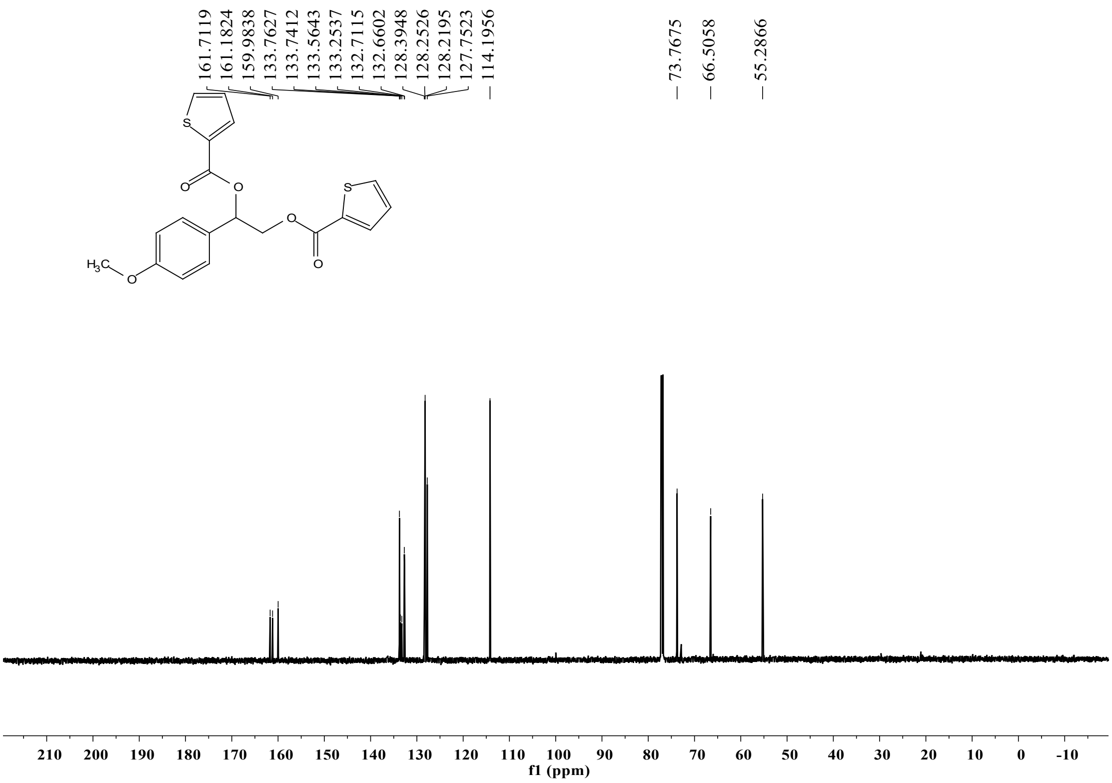
^{19}F-NMR Spectrum (565 MHz, $CDCl_3$) of 19



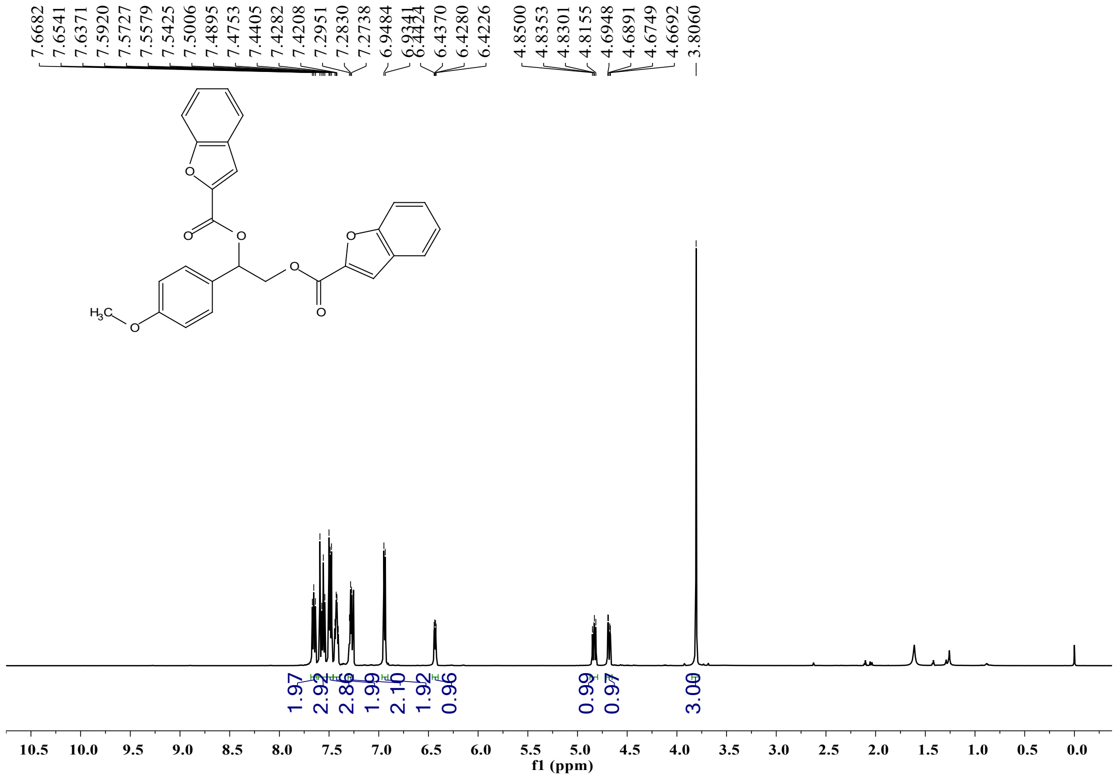
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 20



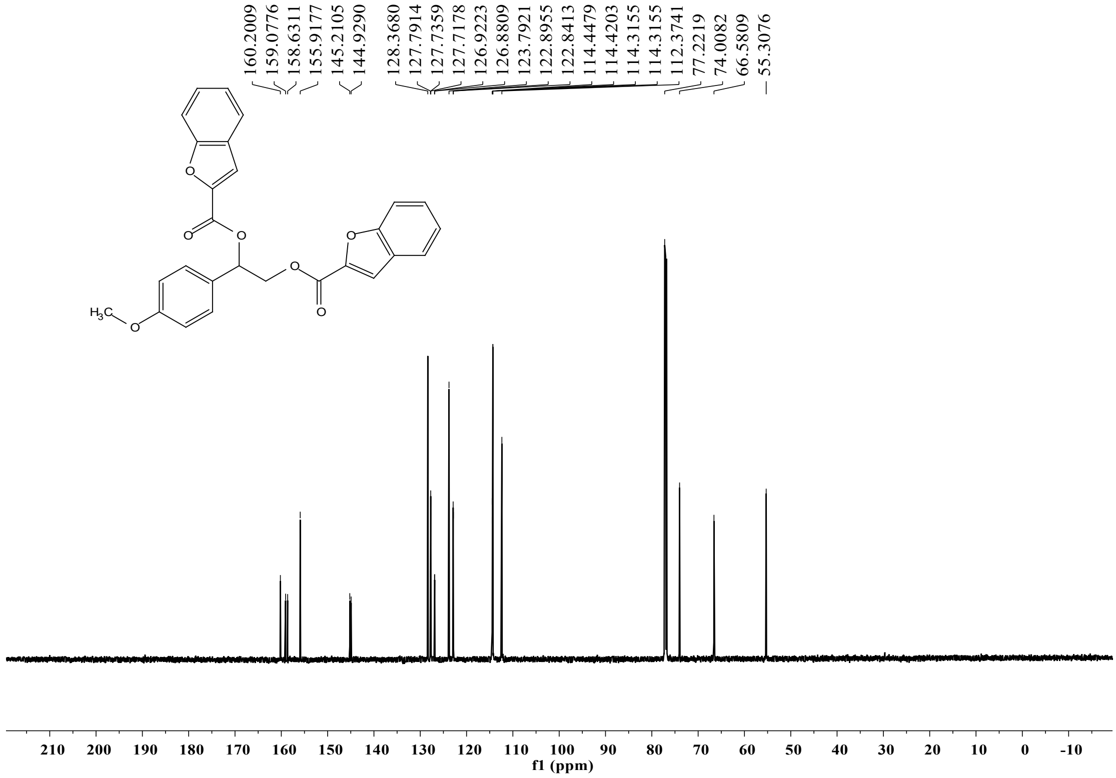
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 20



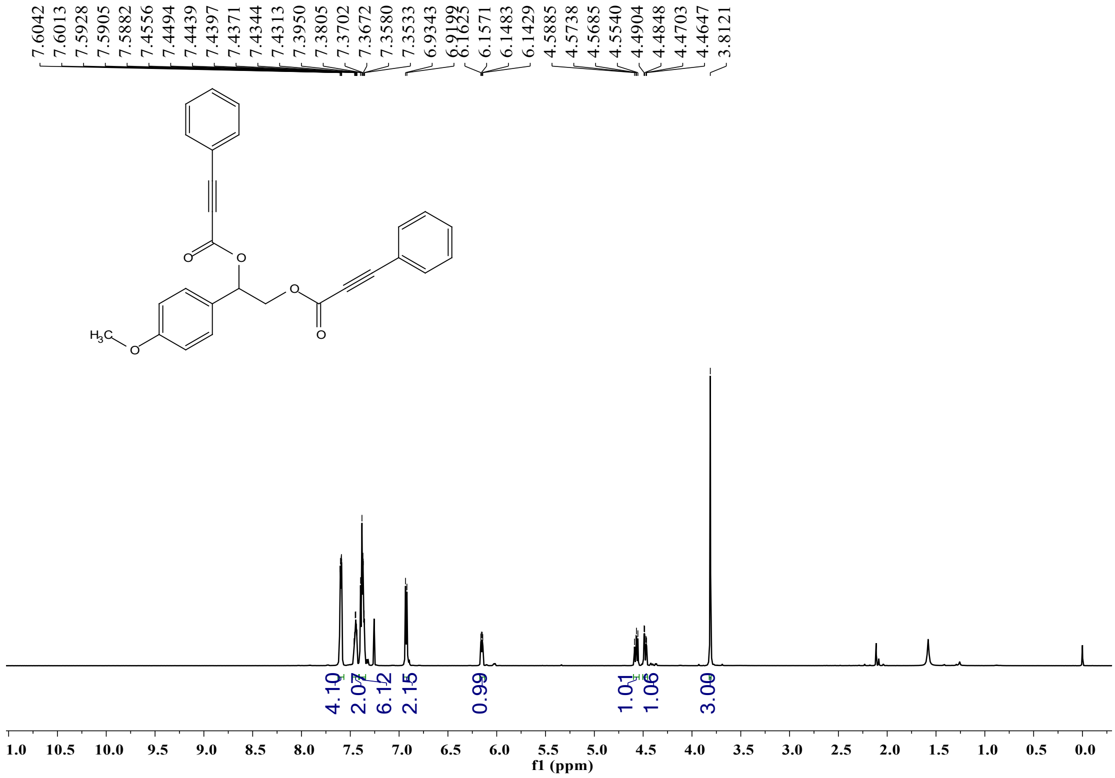
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 21



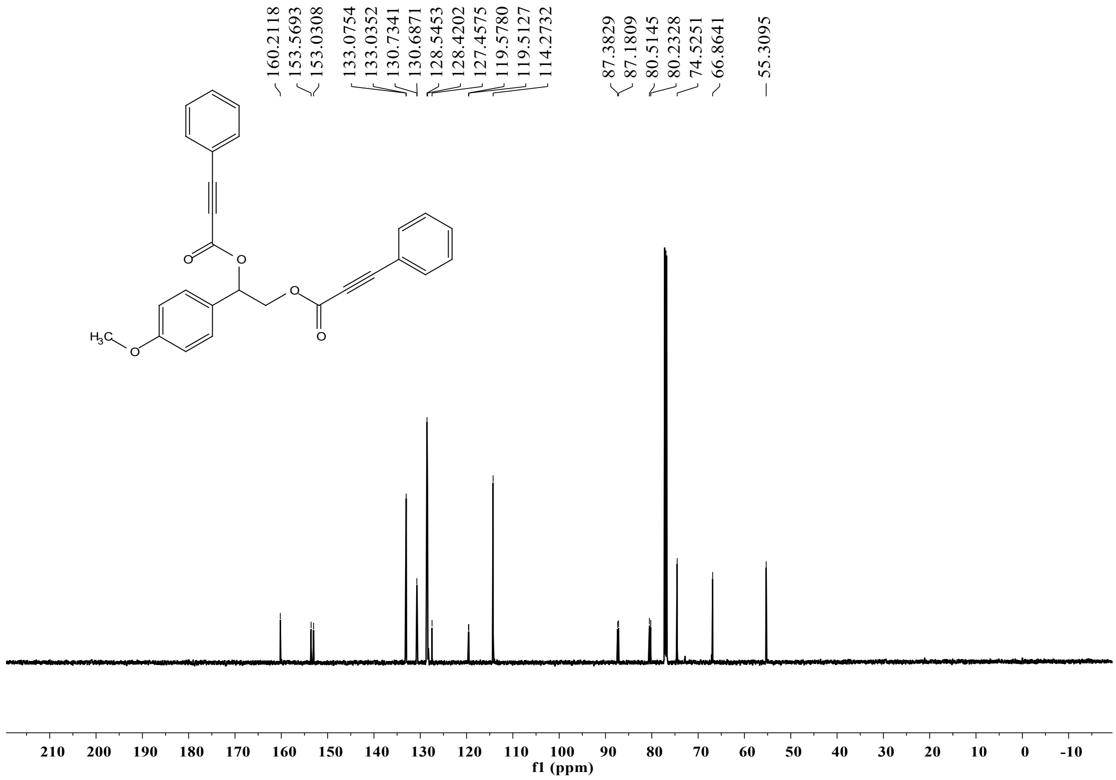
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 21



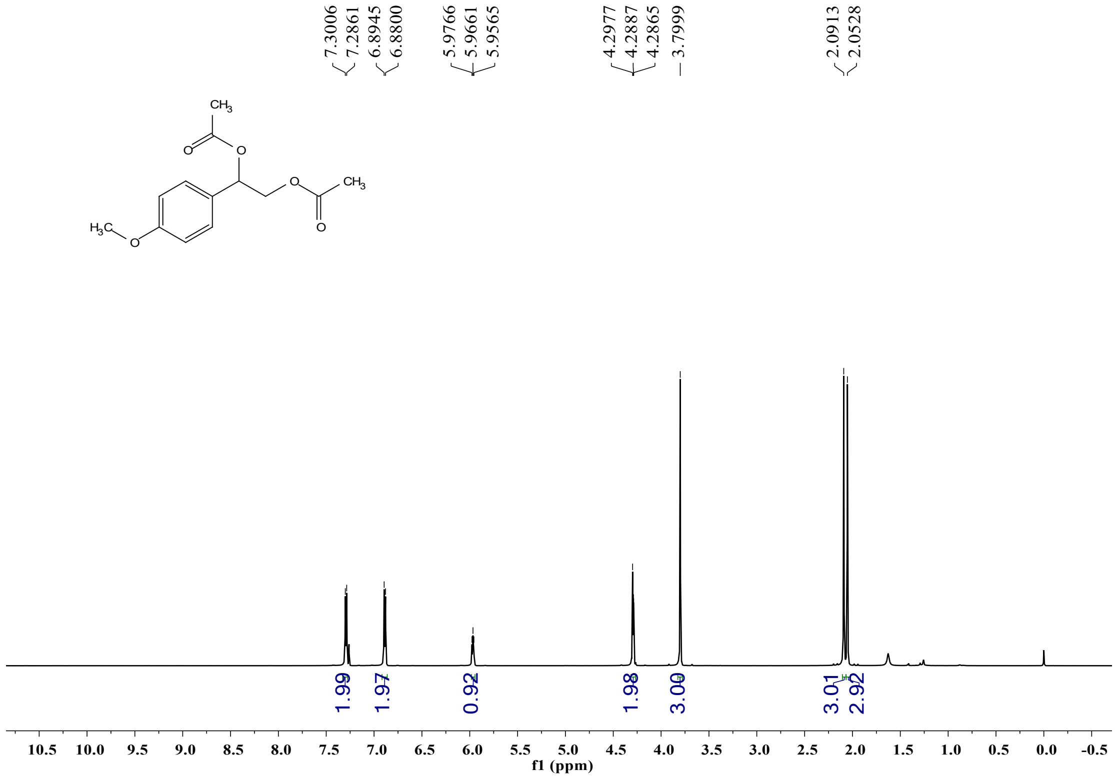
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 22



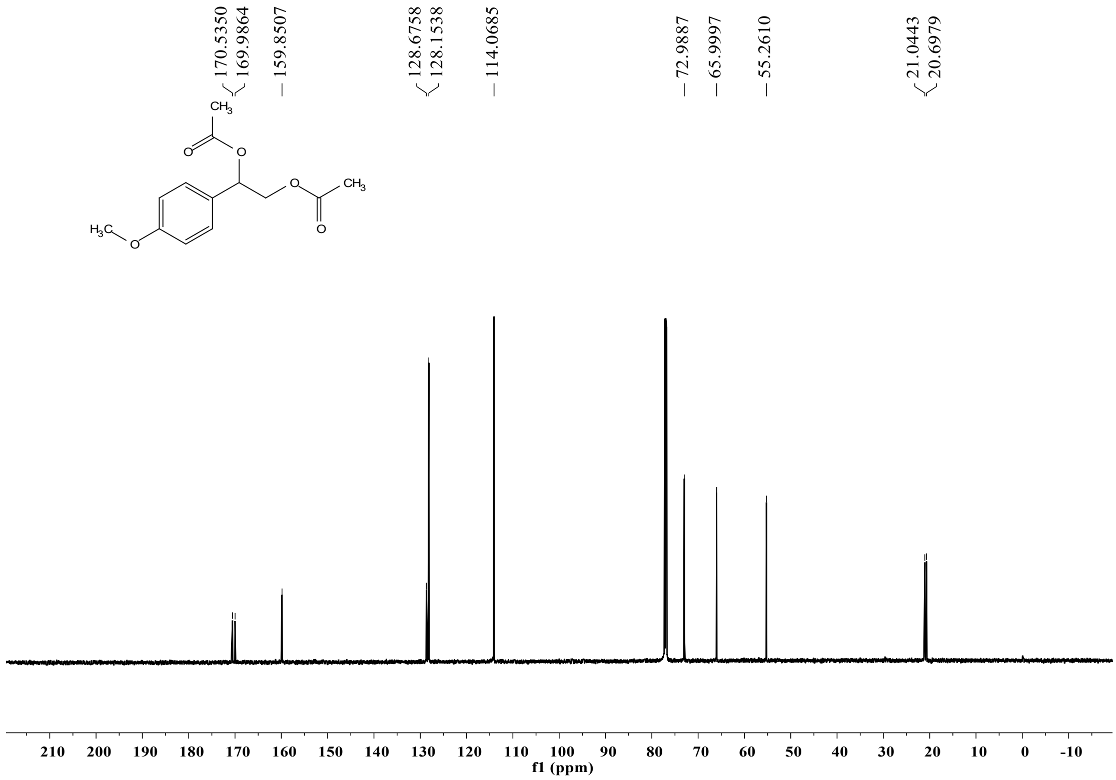
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 22



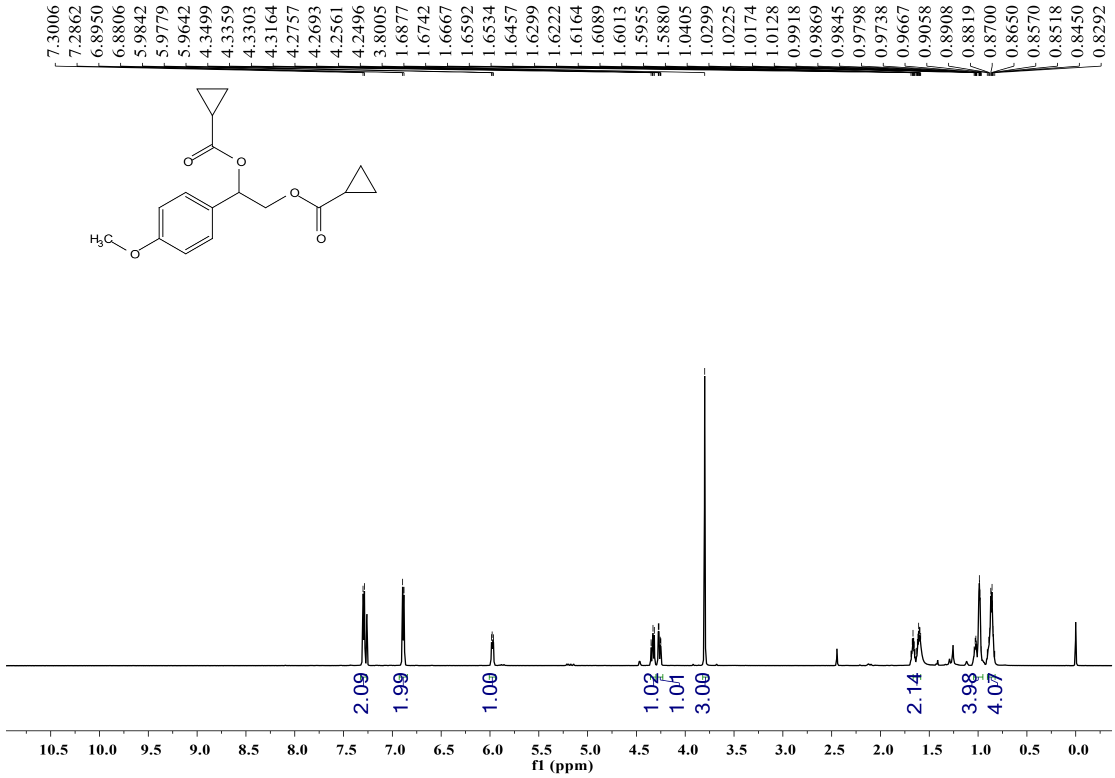
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 23



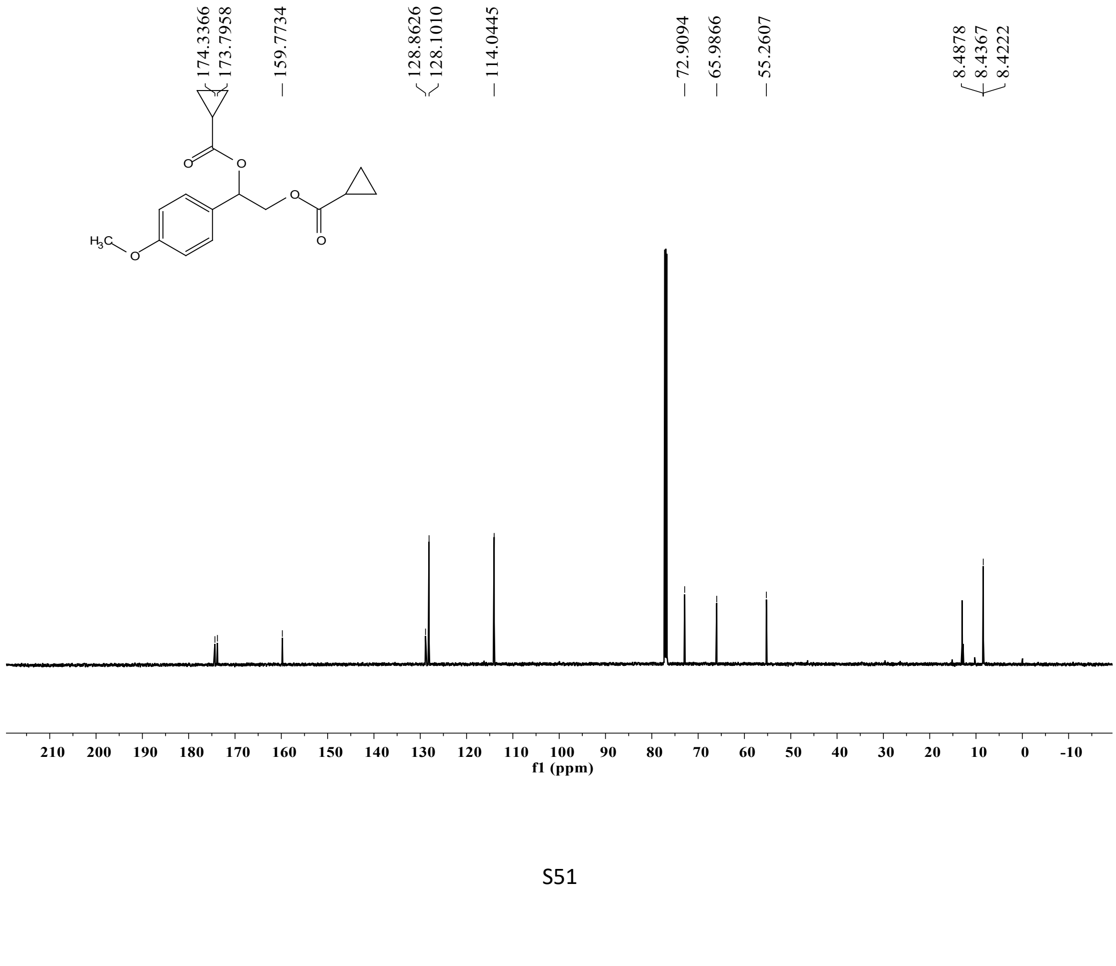
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 23



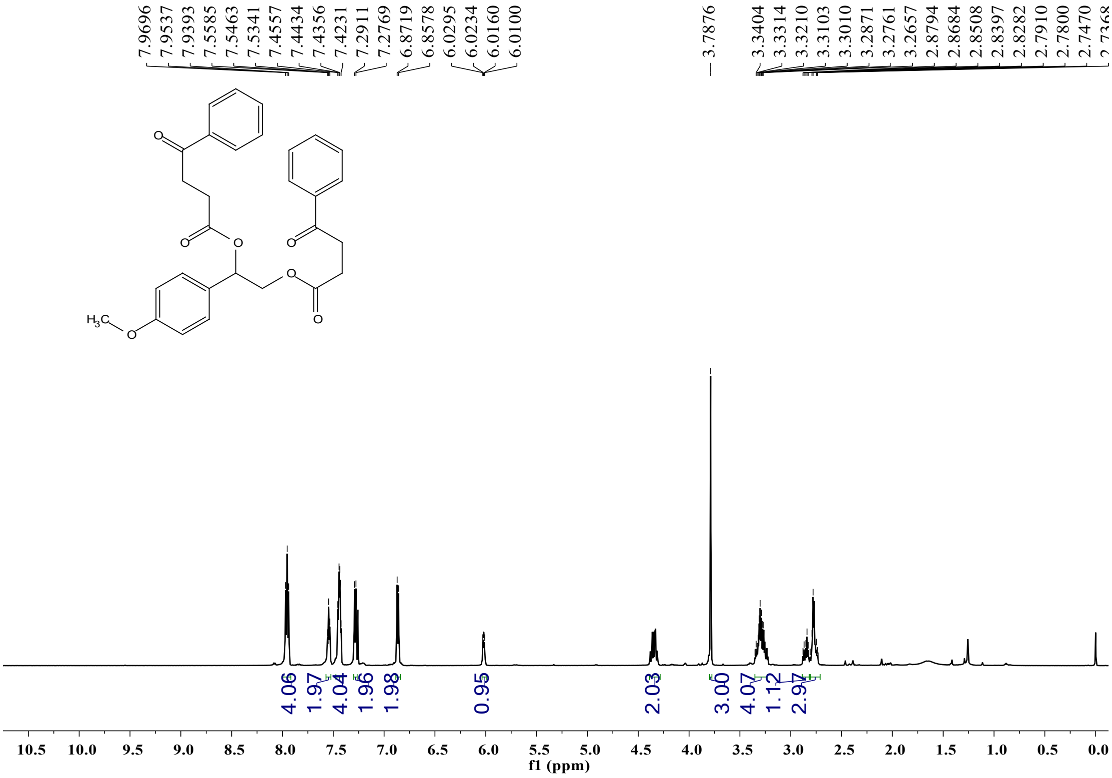
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 24



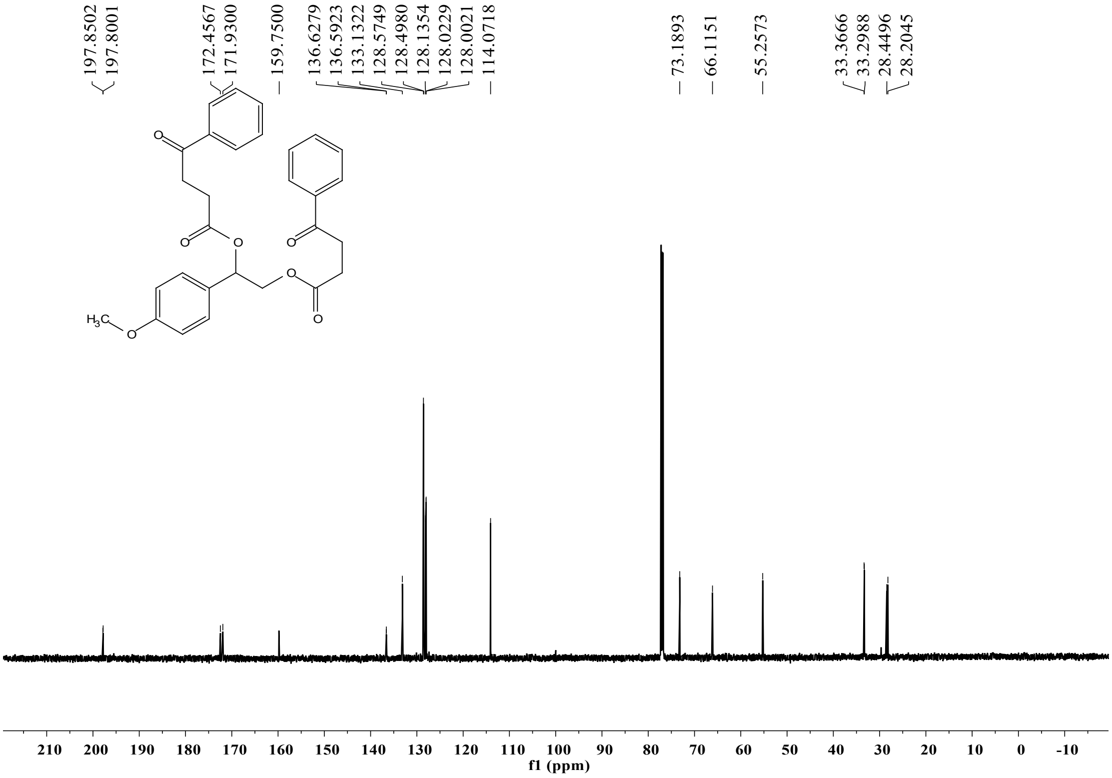
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 24



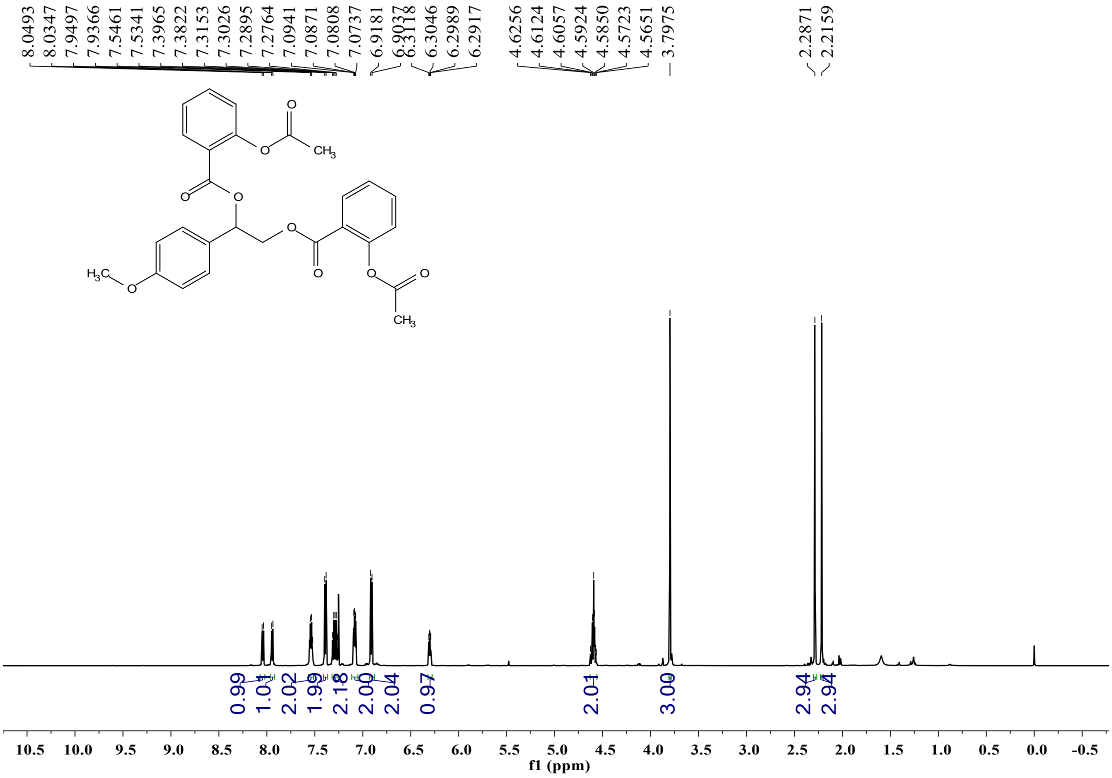
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 25



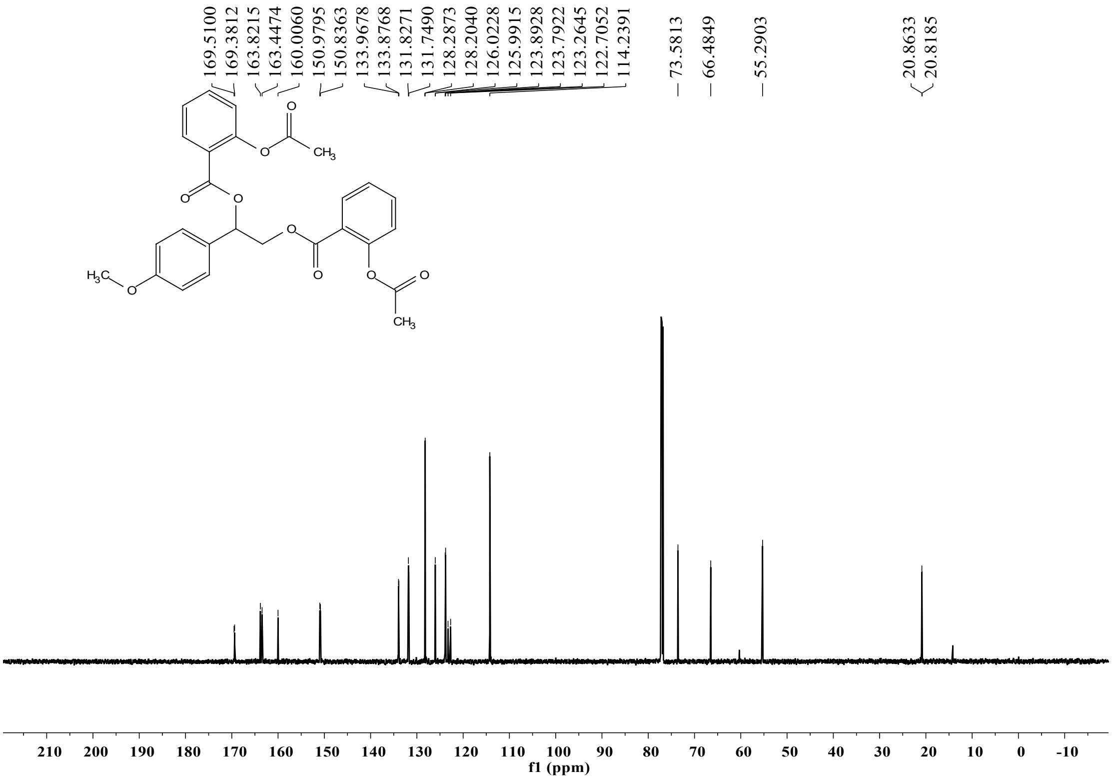
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 25



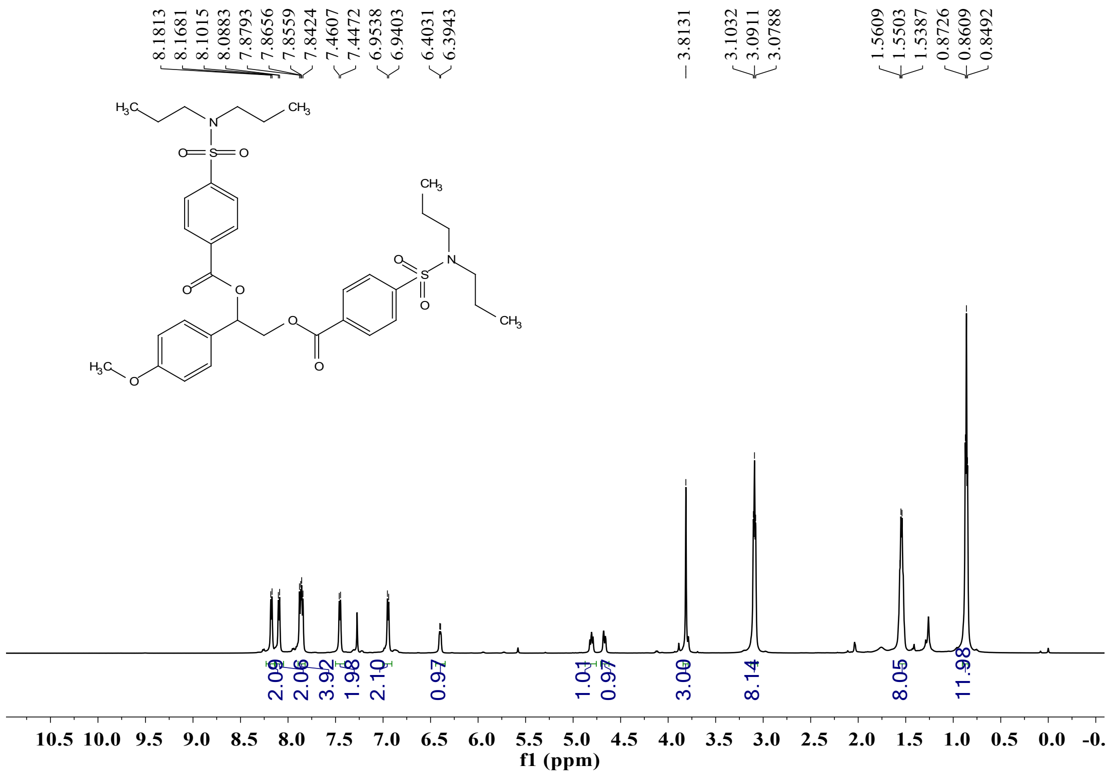
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 26



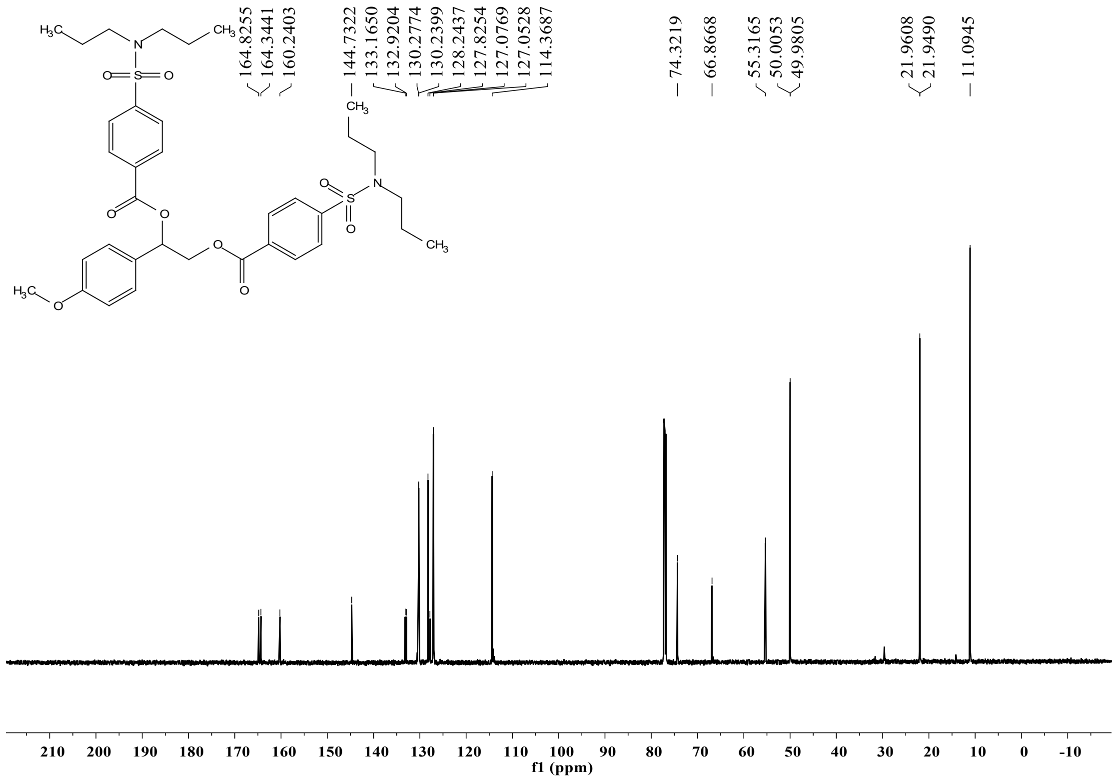
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 26



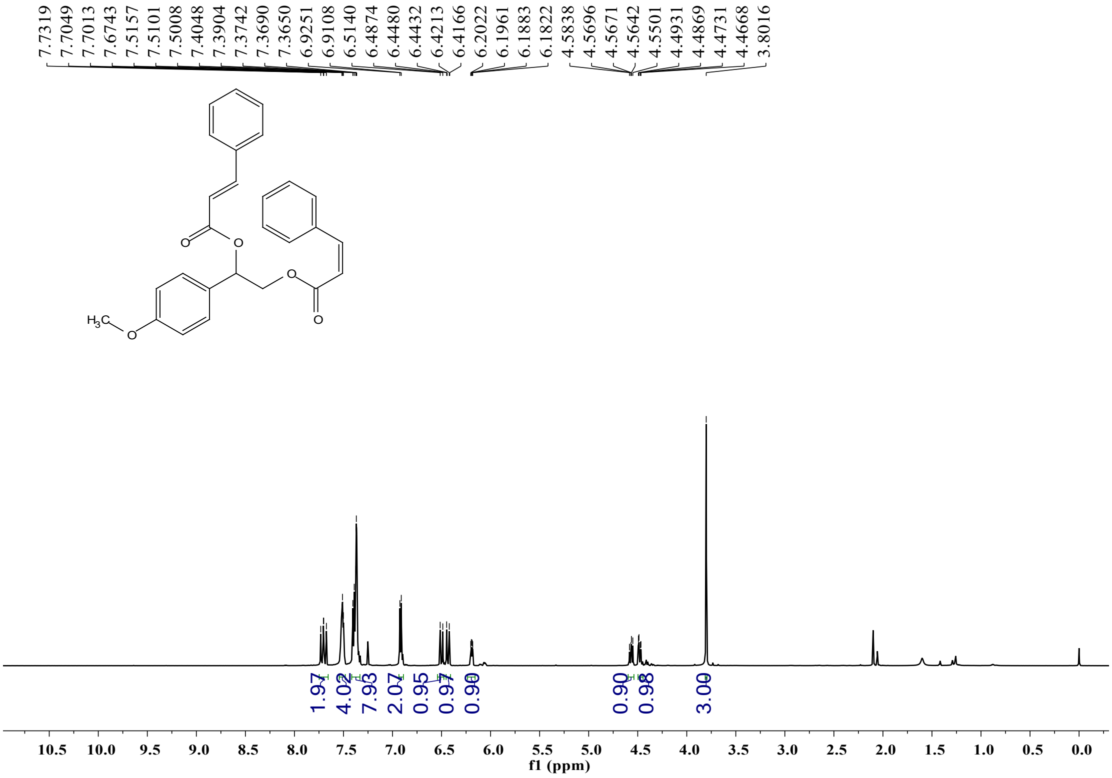
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 27



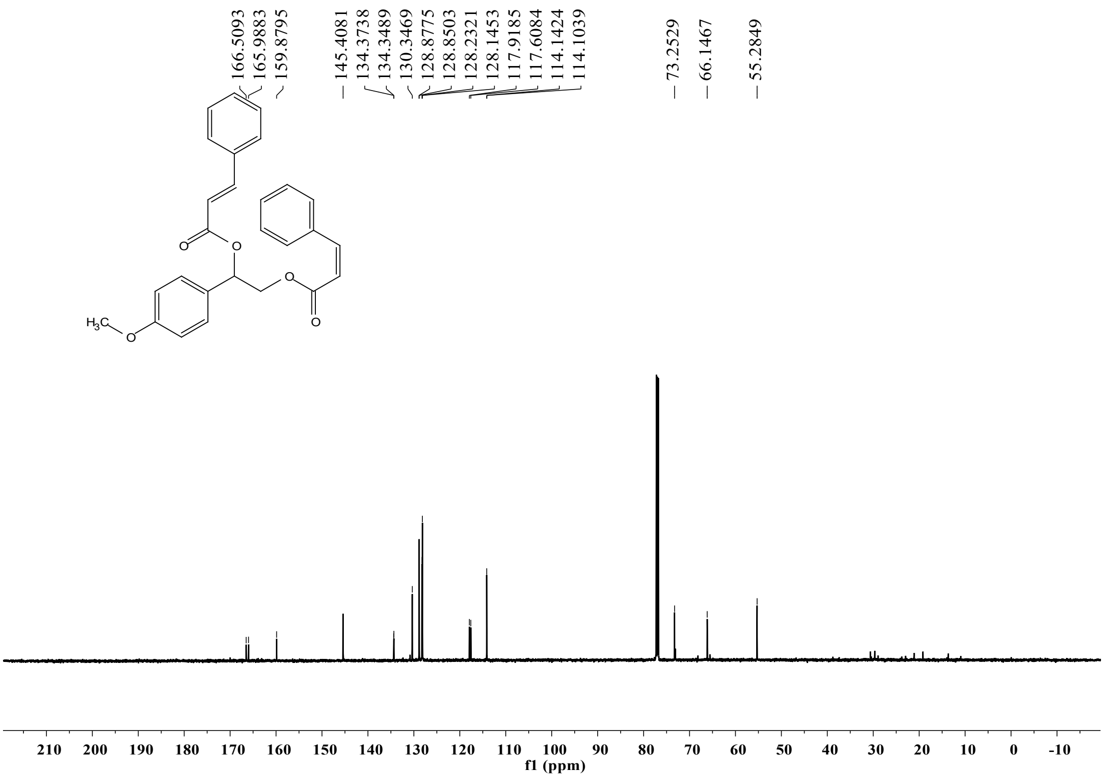
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 27



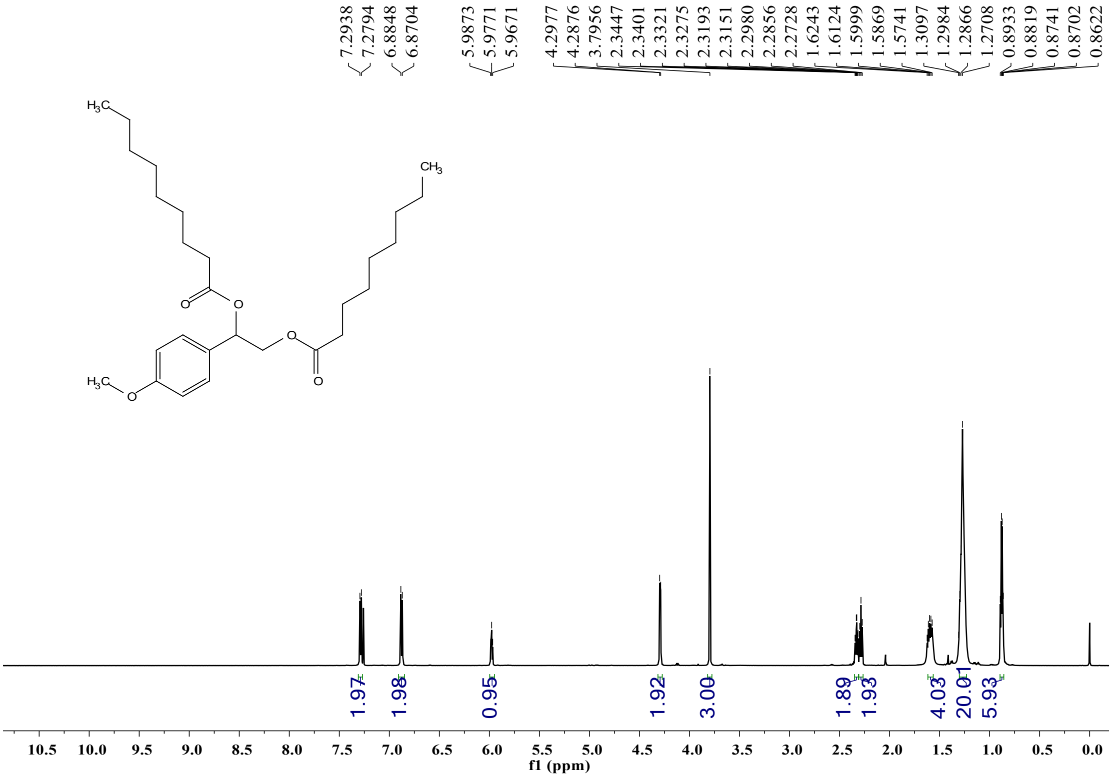
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 28



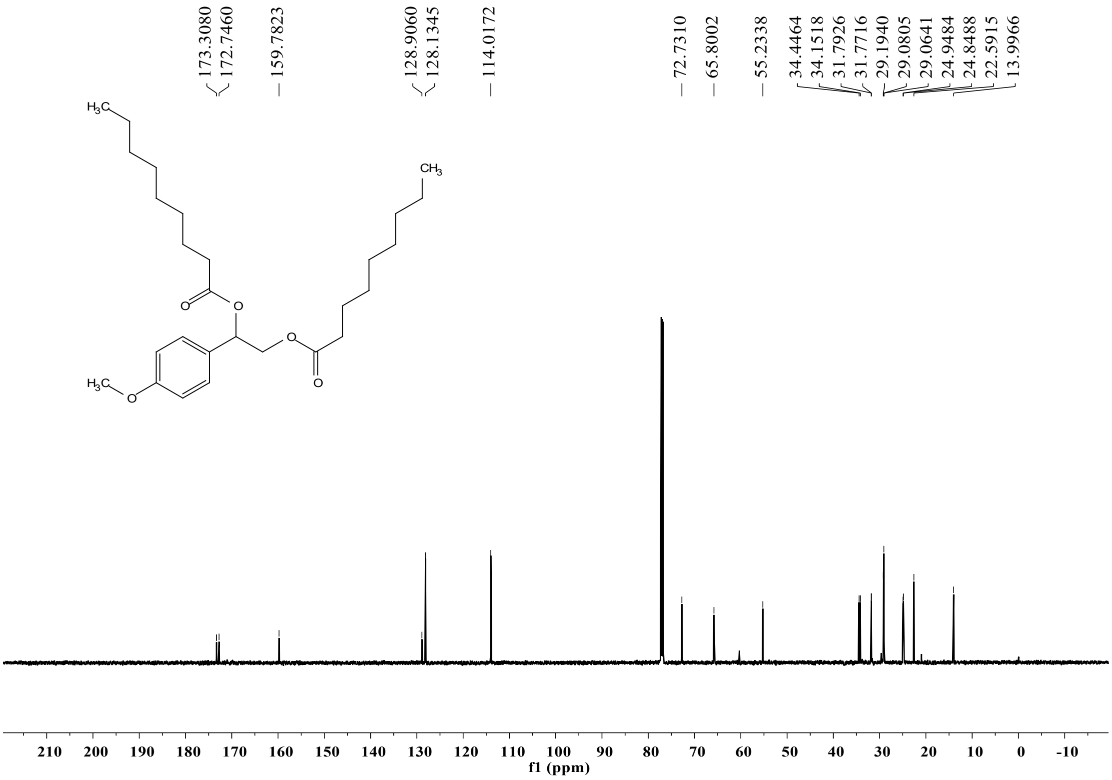
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 28



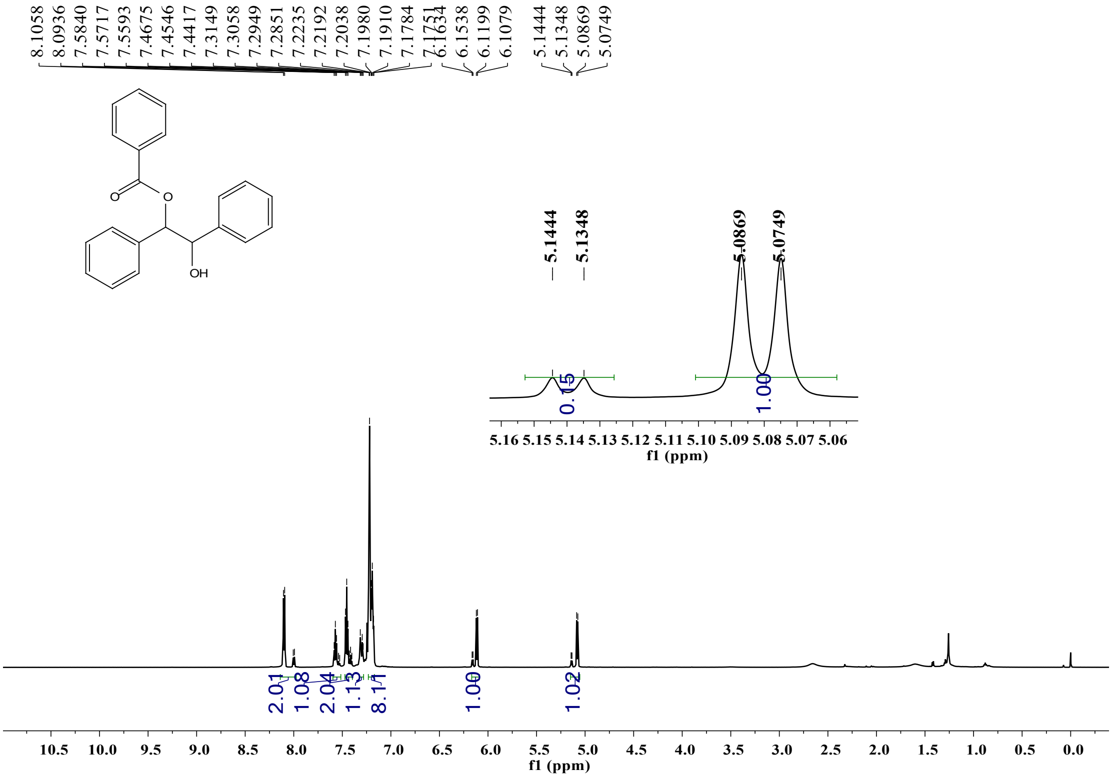
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 29



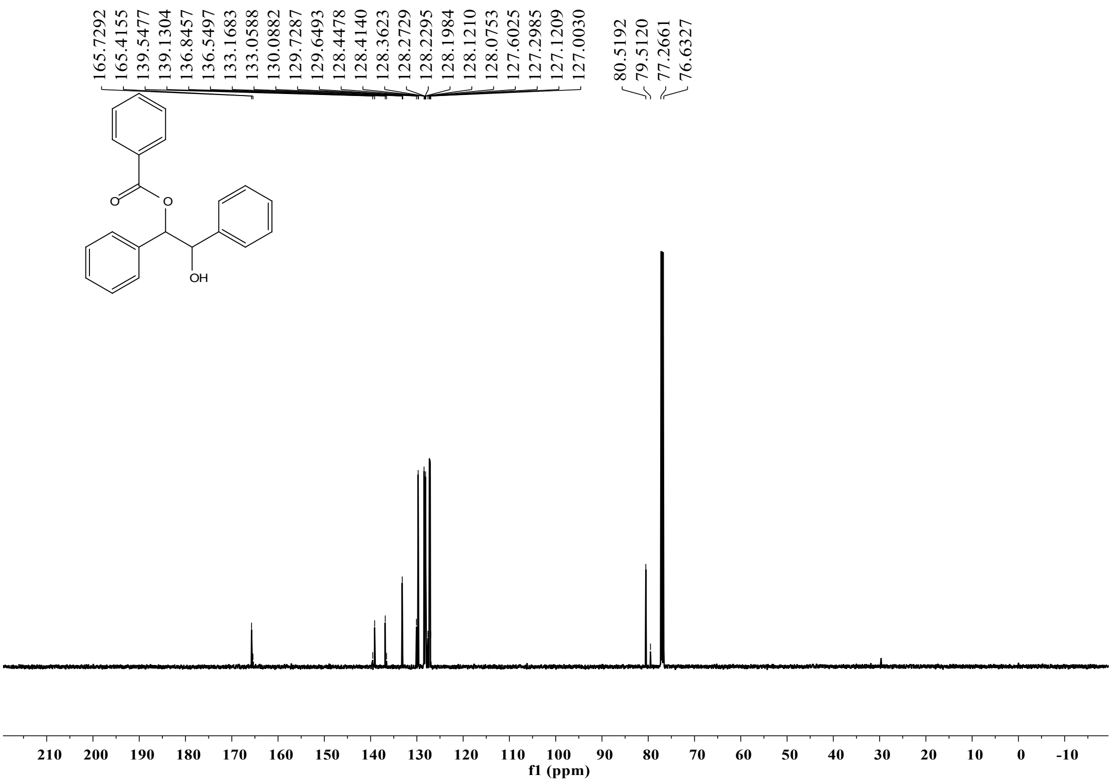
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 29



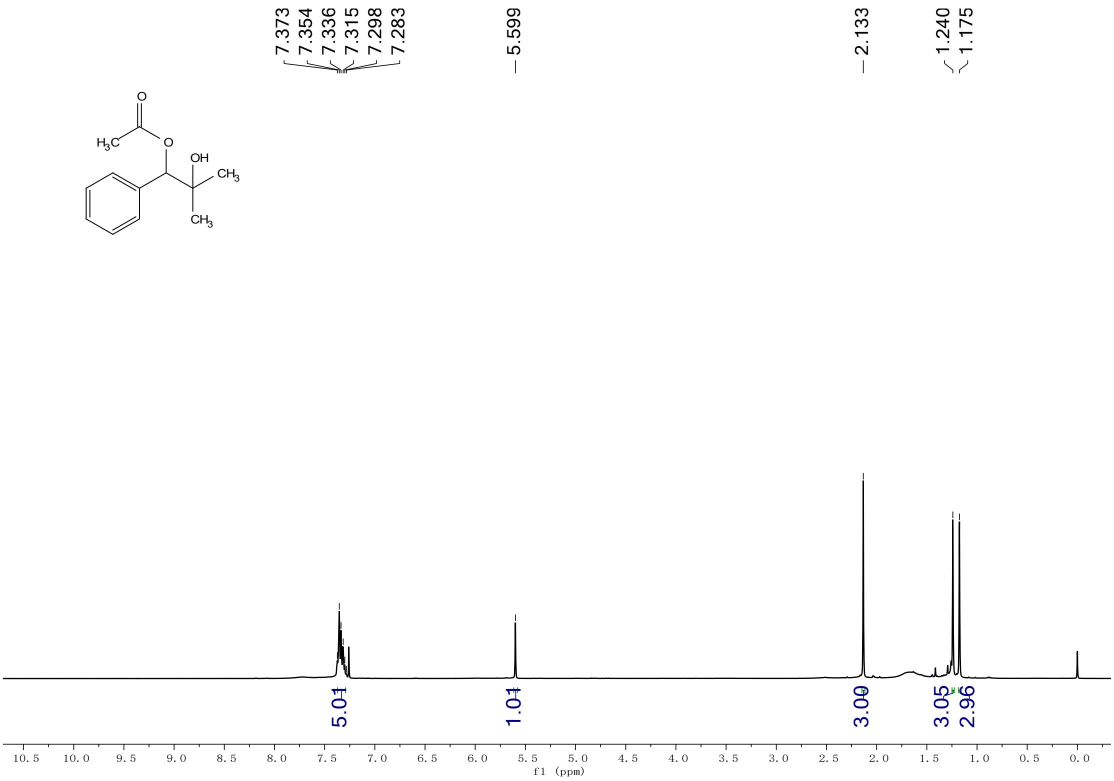
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 30



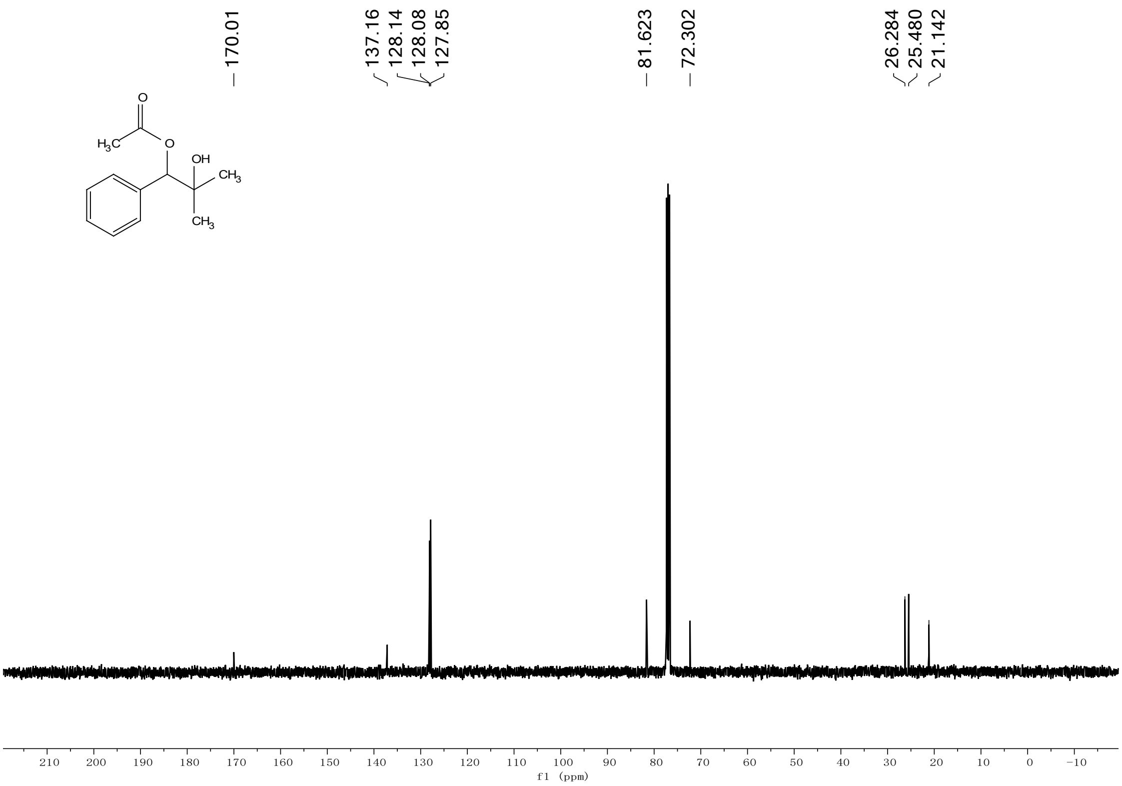
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 30



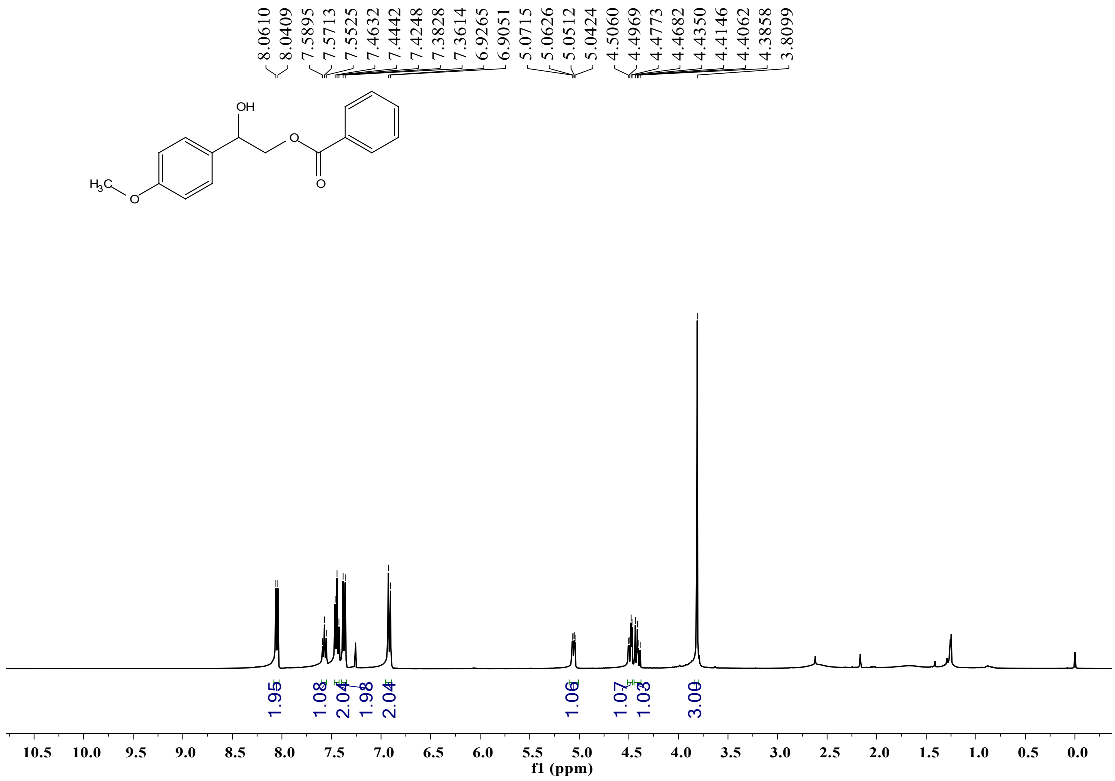
[1]H-NMR Spectrum (400 MHz, $CDCl_3$) of 31



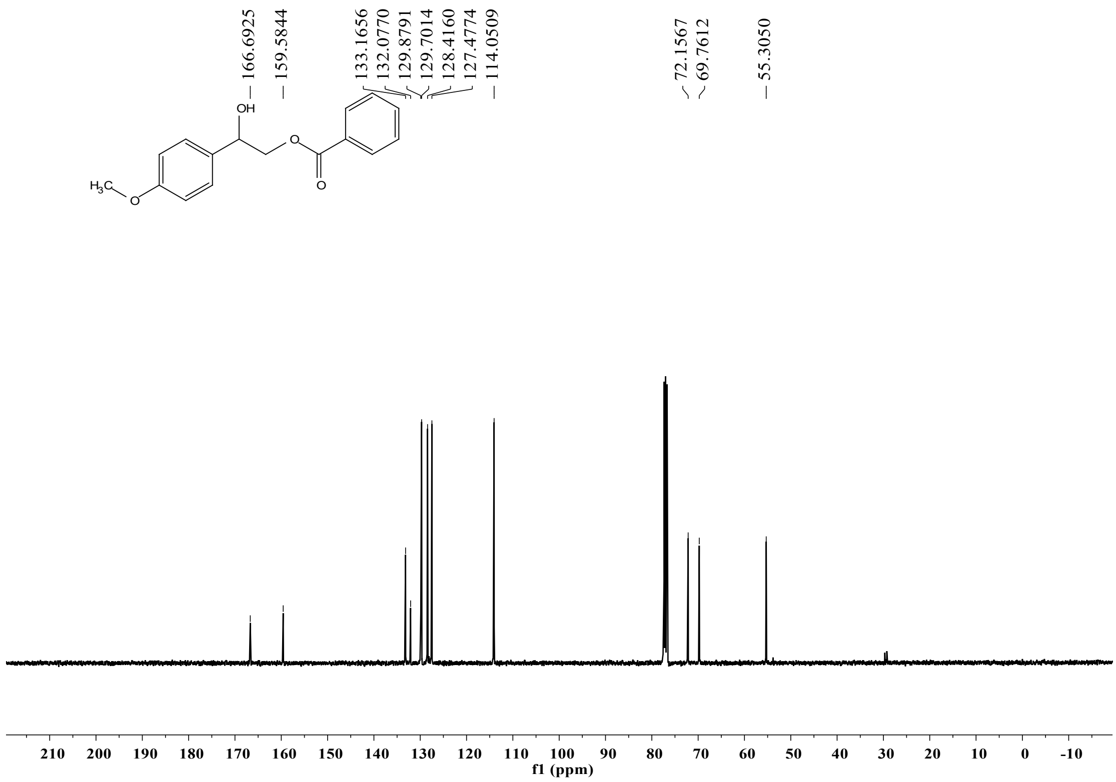
[13]C-NMR Spectrum (101 MHz, $CDCl_3$) of 31



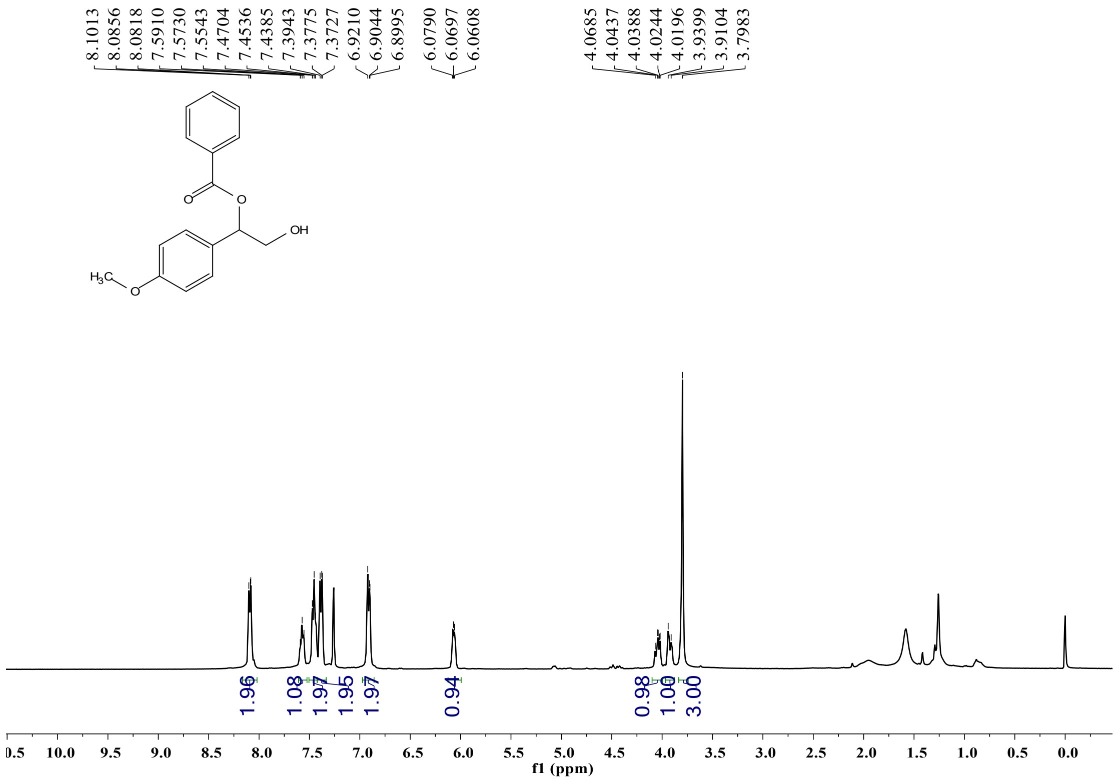
[1]H-NMR Spectrum (400 MHz, $CDCl_3$) of 32



[13]C-NMR Spectrum (101 MHz, $CDCl_3$) of 32



^{1}H-NMR Spectrum (400 MHz, $CDCl_3$) of 33



^{13}C-NMR Spectrum (101 MHz, $CDCl_3$) of 33