Supporting Information

Electricity-Driven 1,4-Alkoxydimerization of Alkenes via Radical-Polar Crossover

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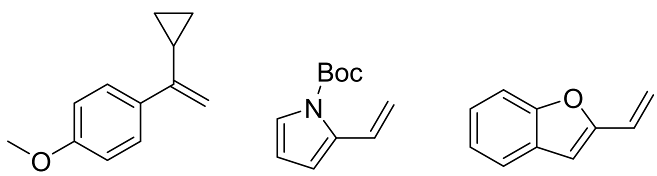
Content

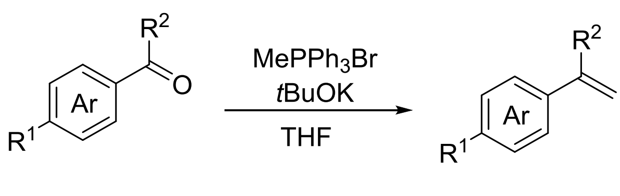
1. General methods

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 NMR 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). **Abbreviations:** BHT = 2,6-di-*tert*-butyl-4-methylphenol, RT = room temperature, dr = diastereomeric ratio, MeOH = methanol, HRMS = High-resolution mass spectrometry.

2. Experimental procedures

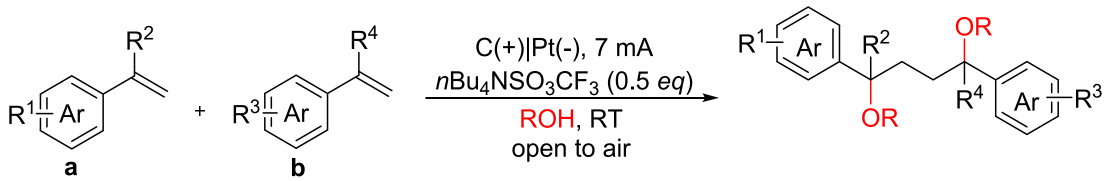
2.1. General procedure for the preparation of substrates





General Wittig reaction procedure for alkene synthesis: To a 50 mL dried flask equipped with a stirring bar was added *t*BuOK (1.12 g, 10 mmol, 2.0 *equiv*) and THF (5 mL). To another 50 mL dried flask equipped with a stirring bar was added $MePPh_3Br$ (3.57 g, 10 mmol, 2.0 *equiv*) and THF (20 mL,) at RT. After stirring for 0.5 h, the $MePPh_3Br$ mixture was added dropwise to the flask containing the *t*BuOK mixture. Finally, aldehyde or ketone (5 mmol, 1.0 *equiv*) was added. The reaction mixture was stirred at RT. Upon completion (monitored by TLC), it was concentrated in vacuo, diluted with water, and then 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 1,4-dialkoxybutane derivatives



To an undivided beaker-type electrolysis cell (10 mL) equipped with a magnetic stirring bar was added olefin **a** (0.5 mmol, 1 *equiv*) [or olefin **a** (0.25 mmol, 1 *equiv*) and olefin **b** (1.25 mmol, 5 *equiv*)], $n\text{Bu}_4\text{NSO}_3\text{CF}_3$ (0.25 mmol, 0.5 *equiv*), and ROH (5 mL). 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 7 mA at RT 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.

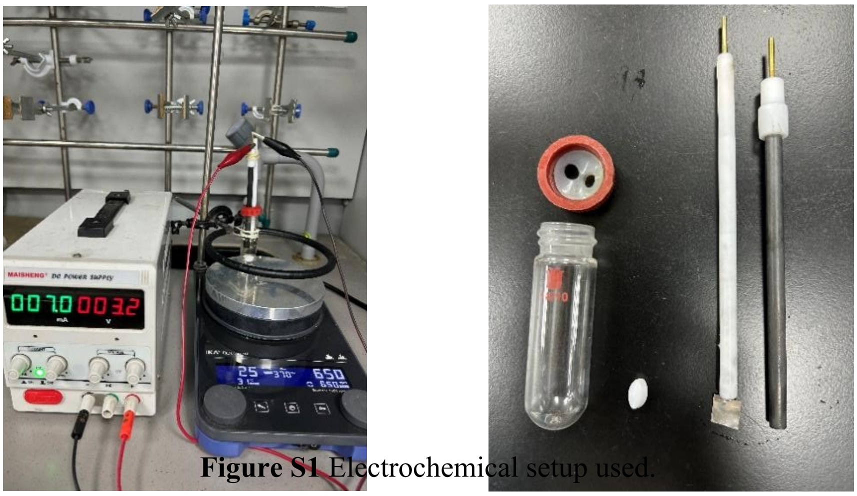
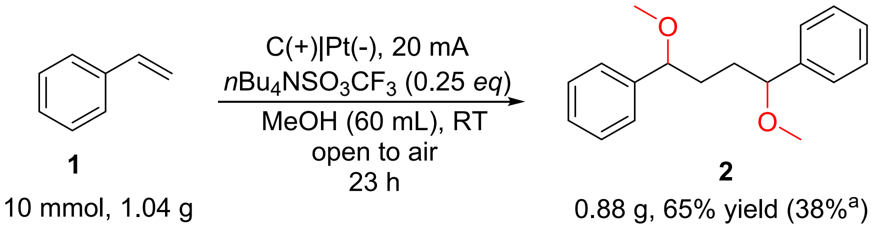


Figure S1 Electrochemical setup used.

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 2



[a] Reaction conditions: A mixture of **1** (10 mmol, 1 *equiv*) and *n*$Bu_4NSO_3CF_3$ (2.5 mmol, 0.25 *equiv*) in MeOH (60 mL) under a constant current of 20 mA (C anode: three carbon rods, Φ = 0.5 cm each; Pt cathode: 3 cm x 3 cm x 0.2 mm) in an undivided cell at RT open to air. [a] Current efficiency.

Substrate **1** (10 mmol, 1 *equiv*), *n*$Bu_4NSO_3CF_3$ (2.5 mmol, 0.25 *equiv*), and MeOH (60 mL,) were added to an undivided beaker-type electrolysis cell (100 mL) equipped with a magnetic stirring bar. Three carbon rod electrodes (Φ = 0.5 cm each) were used as the anode and a platinum plate (3 cm x 3 cm x 0.2 mm) was used 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 RT 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 (150:1, v/v) as the eluent to obtain the target product **2** (0.88 g, 65% yield).

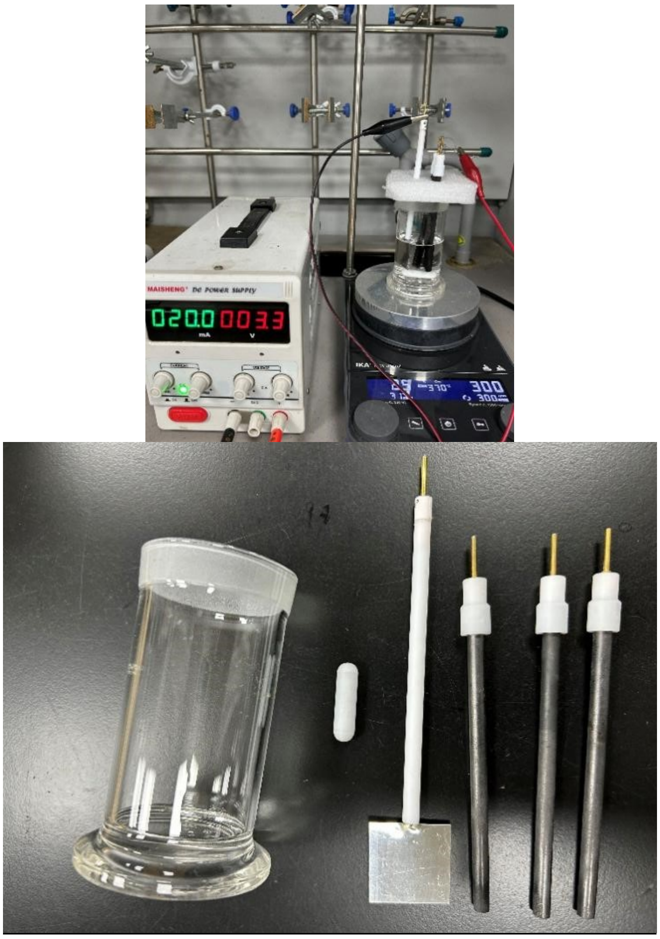


Figure S2 Electrochemical setup for gram-scale experiment.

The experimental setup 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.

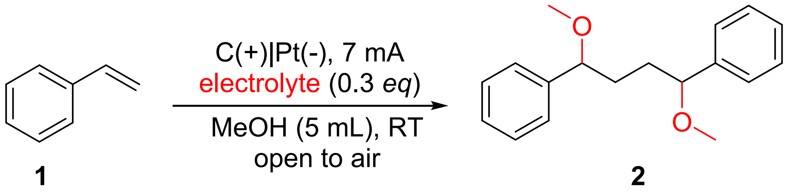
2.4. Further transformations of 1,4-dialkoxybutane 1

2.4.1. Synthesis of 33 and 34

The 1,4-dialkoxybutane **2** (47.3 mg, 0.175 mmol, 1.0 *equiv*) was treated with 4-toluenesulfonamide or 4-nitroaniline (0.455 mmol, 2.6 *equiv*) and $FeCl_3$ (20 mol%) in CH_2Cl_2 (4 mL) at RT under argon atmosphere for 10 h. 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 (10:1, v/v) as the eluent to afford the target product **33** and **34** in 48% and 73% yields, respectively (33.2 and 48.1 mg).

3. Optimization of reaction conditions

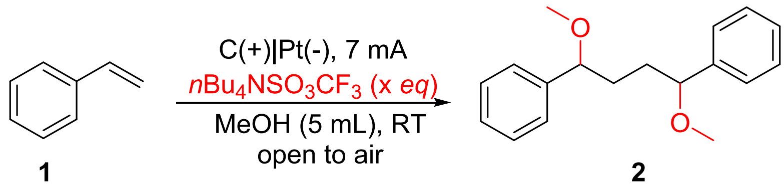
Table S1. Electrolyte screening [a]



Entry	Electrolyte	Yield (%) [b]	Time (h)	Current efficiency (%) [c]
1	--	Voltage overload	0.5	--
2	*n*Bu_4NClO_4	52	4	25
3	*n*Et_4NClO_4	22	4	11
4	*n*Bu_4NI	Trace	4	--
5	*n*Bu_4NOAc	15	4	7
6	*n*Bu_4NPF_6	20	4.5	9
7	*n*Bu_4NBF_4	28	5	11
8	*n*$Bu_4NSO_3CF_3$	65	3.5	36
9	$NaClO_4$	27	4	13
10	$LiClO_4$	44	4	21
11	$KClO_4$	33	4	16

[a] Reaction conditions: A mixture of **1** (0.5 mmol, 1 *equiv*) and electrolyte (0.15 mmol, 0.3 *equiv*) in MeOH (5 mL) under a constant current of 7 mA (C anode: carbon rod 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] Current efficiency.

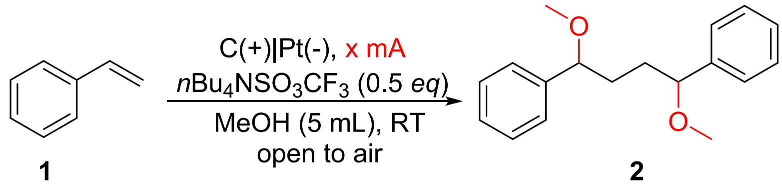
Table S2. Screening of the amount of *n*$Bu_4NSO_3CF_3$ [a]



Entry	*n*$Bu_4NSO_3CF_3$ (*equiv*)	Yield (%) [b]	Time (h)	Current efficiency (%) [c]
1	0.1	50	3.5	28
2	0.3	65	3.5	36
3	0.5	81	3	52
4	0.7	59	3.5	33
5	0.9	58	4	28

[a] Reaction conditions: A mixture of **1** (0.5 mmol, 1 *equiv*) and *n*$Bu_4NSO_3CF_3$ (x *equiv*) in MeOH (5 mL) under a constant current of 7 mA (C anode: carbon rod 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] Current efficiency.

Table S3. Current screening [a]

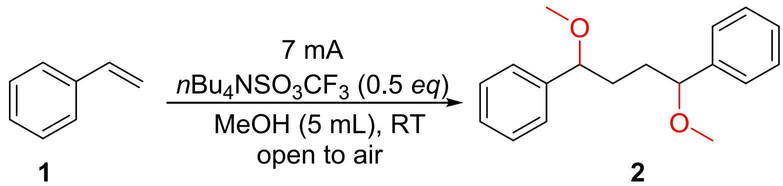


1 **2**

Entry	Current (mA)	Yield (%) [b]	Time (h)	Current efficiency (%) [c]
1	--	N.R.	3	--
2	5	70	4.5	42
3	7	81	3	52
4	9	53	2.5	32
5	11	52	2	32

[a] Reaction conditions: A mixture of **1** (0.5 mmol, 1 *equiv*) and nBu$_4$NSO$_3$CF$_3$ (0.25 mmol, 0.5 *equiv*) in MeOH (5 mL) under a constant current of x mA (C anode: carbon rod 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] Current efficiency.

Table S4. Electrode material screening [a]

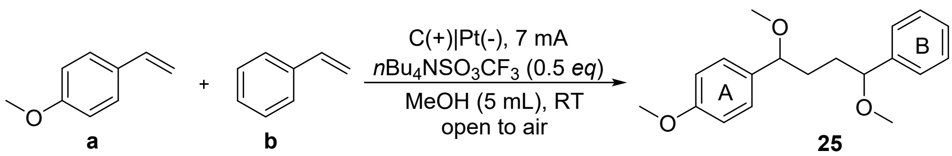


1 **2**

Entry	Electrode material	Yield (%) [b]	Current efficiency (%) [c]
1	C(+) | Pt(-)	81	52
2	C(+) | C(-)	32	21
3	Pt(+) | Pt(-)	10	6
4	Pt(+) | C(-)	15	10
5	C felt(+) | Pt(-) instead of C(+) | Pt(-)	34	22
6	graphite felt(+) | Pt(-) instead of C(+) | Pt(-)	15	10
7	RVC(+) | Pt(-) instead of C(+) | Pt(-)	5	3
8	glassy C(+) | Pt(-) instead of C(+) | Pt(-)	8	5
9	Ni foam(+) | Pt(-) instead of C(+) | Pt(-)	N.D.	--

[a] Reaction conditions: A mixture of **1** (0.5 mmol, 1 *equiv*) and nBu$_4$NSO$_3$CF$_3$ (0.25 mmol, 0.5 *equiv*) in MeOH (5 mL) under a constant current of 7 mA in an undivided cell at RT open to air for 3 h. [b] Isolated yield. [c] Current efficiency. C electrode: carbon rod Φ = 0.5 cm; Pt electrode: 1 cm x 1 cm x 0.2 mm

Table S5. The ratio of alkene **a** and **b** screening [a]



a **b** **25**

Entry	**a** : **b**	Yield (%) [b]	Current efficiency (%) [c]
1	1:1	N.D.	--
2	1:2	15	8

3	1:3	31	17
4	1:4	34	19
5	1:5	40	22
6	1:6	38	21
7	2:1	Trace	--
8	3:1	Trace	--
9	4:1	Trace	--
10	5:1	Trace	--

[a] Reaction conditions: A mixture of alkene **a** and alkene **b** and *n*$Bu_4NSO_3CF_3$ (0.25 mmol, 0.5 *equiv*) in MeOH (5 mL) under a constant current of 7 mA (C anode: carbon rod 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 3.5 h. b Isolated yield. [c] Current efficiency.

4. Mechanistic investigation

4.1. 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 MeOH (15 mL) as solvent, *n*$Bu_4NSO_3CF_3$ (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. The studied range was from 0.0 V to +3.0 V *vs.* Ag/AgCl, and the scanning speed was 100 $mV{\cdot}s^{-1}$. The results showed that the onset potential for the oxidation of styrene (**1**) was around +1.713 V *vs.* Ag/AgCl (3 M KCl) (Figure S3).

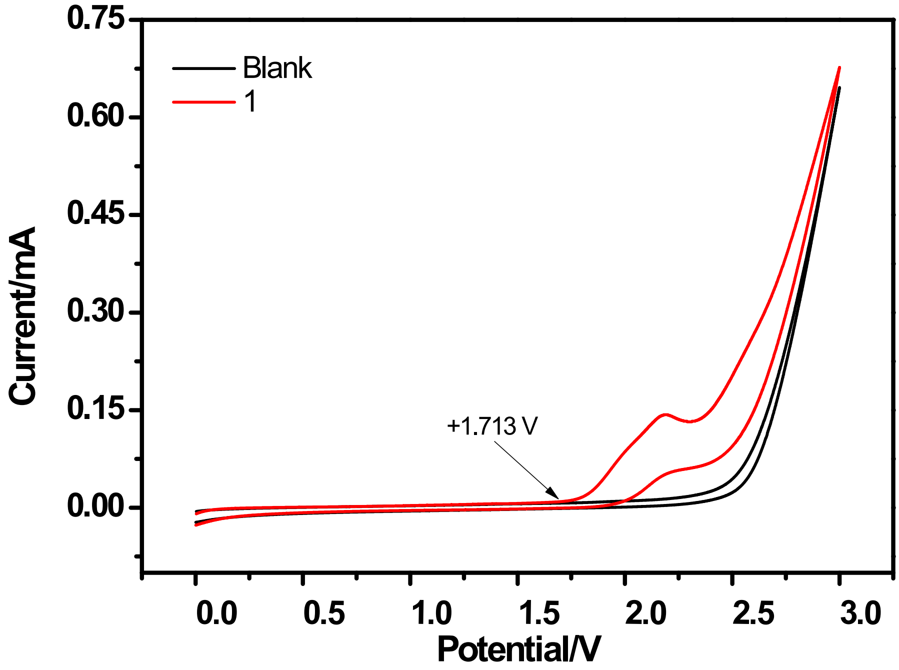
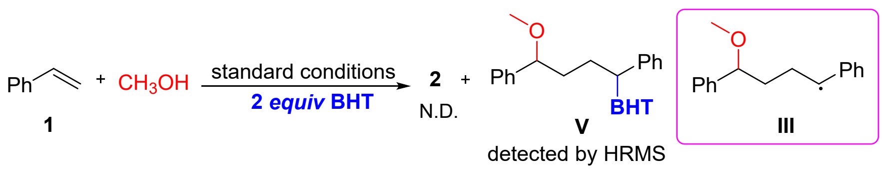


Figure S3 Cyclic voltammetry experiments.

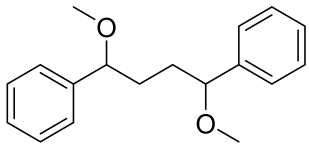
4.2. Radical trapping experiments

In order to confirm whether the reaction undergoes a radical mechanism, commonly used radical scavengers, 2,6-di-*tert*-butyl-4-methylphenol (BHT) was used in radical trapping experiments. Under standard conditions, BHT (2.0 *equiv* to **1a**) was added to the model reaction system at the beginning of the reaction. After 3 h, a small amount of reaction mixture was taken out for high-resolution mass spectrometry (HRMS) measurement.

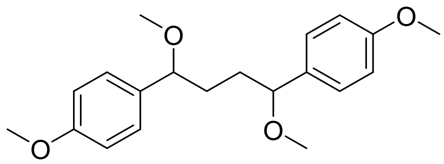


Scheme S1. Radical trapping experiments.

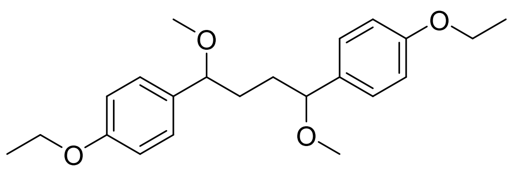
5. Characterization data of the products



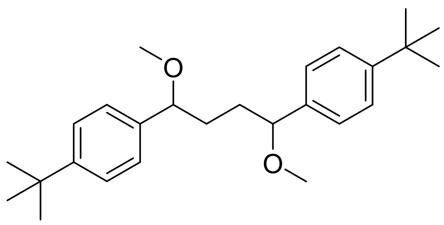
1,4-dimethoxy-1,4-diphenylbutane (2)[1]: R_f = 0.25 (Petroleum ether/EtOAc, 150:1). 54.7 mg, 81% yield (dr = 9.5:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.6 Hz, 4H), 7.26 – 7.20 (m, 6H), 4.04 (t, *J* = 5.6 Hz, 2H), 3.17 (s, 6H), 1.95 – 1.87 (m, 1.81H), 1.82 – 1.78 (m, 0.29H), 1.76 – 1.71 (m, 0.22H), 1.62 – 1.55 (m, 1.78H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.3, 142.2, 128.4, 127.5, 126.7, 126.6, 84.1, 83.8, 56.7, 56.6, 34.8, 34.2.



1,4-dimethoxy-1,4-bis(4-methoxyphenyl)butane (3)[2]: R_f = 0.25 (Petroleum ether/EtOAc, 50:1). 71.0 mg, 86% yield (dr = 12.3:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.16 – 7.10 (m, 4H), 6.82 – 6.78 (d, *J* = 8.4 Hz, 3.69H), 6.82 – 6.78 (d, *J* = 8.6 Hz, 0.3H), 4.36 (t, *J* = 5.3 Hz, 0.8H), 3.99 (t, *J* = 5.7 Hz, 1.92H), 3.79 (s, 5.61H), 3.76 (s, 0.40H), 3.25 (s, 0.42H), 3.14 (s, 5.57H), 1.92 – 1.84 (m, 2H), 1.55 – 1.48 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.0, 157.9, 134.2, 128.6, 128.6, 127.9, 127.8, 114.0, 113.8, 113.8, 113.7, 83.6, 83.4, 56.3, 55.2, 34.6, 34.1.

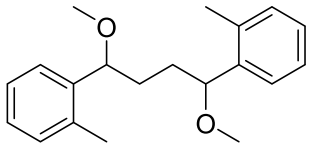


1,4-bis(4-ethoxyphenyl)-1,4-dimethoxybutane (4): R_f = 0.25 (Petroleum ether/EtOAc, 50:1). 70.7 mg, 79% yield (dr = 1.4:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.17 – 7.10 (m, 4H), 6.84 (t, *J* = 9.2 Hz, 4H), 4.05 – 3.94 (m, 6H), 3.16 – 3.10 (m, 6H), 1.91 – 1.83 (m, 1.16H), 1.76 – 1.66 (m, 1.68H), 1.66 – 1.56 (m, 1.16H), 1.42 – 1.38 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 158.4, 158.4, 134.1, 134.0, 127.9, 127.8, 114.3, 114.3, 83.6, 83.4, 63.4, 56.4, 56.3, 34.6, 34.1, 14.9. HRMS (ESI): m/z: calcd for $C_{22}H_{20}O_2$ $(M+Na)^+$ 381.2036; found 381.2030.

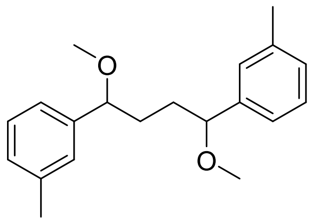


1,4-bis(4-(tert-butyl)phenyl)-1,4-dimethoxybutane (5): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 55.4 mg, 58% yield (dr = 4.0:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 – 7.29 (m, 4H), 7.19

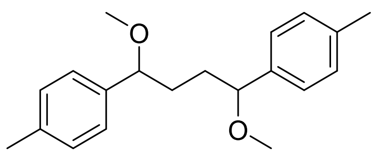
– 7.12 (m, 4H), 4.04 – 3.98 (m, 2H), 3.19 – 3.13 (m, 6H), 1.97 – 1.87 (m, 1.6H), 1.84 – 1.77 (m, 0.4H), 1.75– 1.69 (m, 0.39H), 1.63 – 1.55 (m, 1.61H), 1.33 – 1.28 (m, 18H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 150.3, 139.2, 139.1, 126.4, 126.3, 125.2, 125.2, 83.9, 83.7, 56.6, 56.5, 34.8, 34.5, 34.1, 31.4. HRMS (ESI): m/z: calcd for $C_{26}H_{38}O_2$ $(M+Na)^+$ 405.2764; found 405.2757.



1,4-dimethoxy-1,4-di-o-tolylbutane (6): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 50.7 mg, 68% yield (dr = 4.1:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 2H), 7.21 – 7.16 (m, 2H), 7.16 – 7.08 (m, 4H), 4.44 – 4.38 (m, 1.61H), 4.38 – 4.33 (m, 0.39H), 3.22 – 3.14 (m, 6H), 3.32 – 2.26 (m, 6H), 1.89 – 1.80 (m, 2H), 1.74 – 1.68 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.4, 140.3, 135.4, 135.3, 130.4, 127.0, 126.2, 126.2, 125.8, 125.8, 80.7, 79.7, 56.6, 33.9, 33.1, 19.2, 19.0. HRMS (ESI): m/z: calcd for $C_{20}H_{26}O_2$ $(M+H)^+$ 299.2006; found 299.2005.



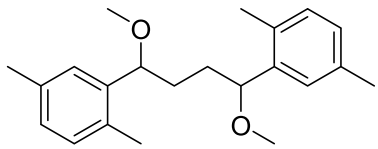
1,4-dimethoxy-1,4-di-m-tolylbutane (7): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 46.2 mg, 62% yield (dr > 20:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.19 (t, *J* = 7.5 Hz, 2H), 7.08 – 7.00 (m, 6H), 4.00 (t, *J* = 5.4 Hz, 2H), 3.17 (s, 6H), 2.33 (s, 6H), 1.95 – 1.86 (m, 2H), 1.62 – 1.51 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.2, 137.9, 128.2, 128.2, 127.4, 123.9, 83.9, 56.6, 34.1, 21.4. HRMS (ESI): m/z: calcd for $C_{20}H_{26}O_2$ $(M+H)^+$ 299.2006; found 299.2003.



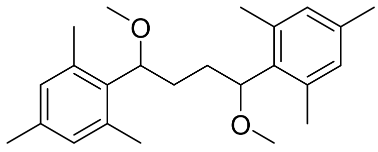
1,4-dimethoxy-1,4-di-p-tolylbutane (8):

Major diastereoisomer: R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 52.9 mg, 71% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.19 – 7.07 (m, 8H), 4.01 (t, *J* = 5.6 Hz, 2H), 3.16 (s, 6H), 2.32 (s, 6H), 1.94 – 1.85 (m, 2H), 1.60 – 1.50 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.1, 137.1, 129.0, 126.7, 83.7, 56.5, 34.2, 21.1. HRMS (ESI): m/z: calcd for $C_{20}H_{26}O_2$ $(M+H)^+$ 299.2006; found 299.2003.

Minor diastereoisomer: R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 5.9 mg, 8% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.19 – 7.09 (m, 8H), 4.04 – 3.97 (m, 2H), 3.15 (s, 6H), 2.33 (s, 6H), 1.80 – 1.68 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.2, 137.1, 129.1, 126.6, 83.9, 56.5, 34.8, 21.1. HRMS (ESI): m/z: calcd for $C_{20}H_{26}O_2$ $(M+H)^+$ 299.2006; found 299.2002.



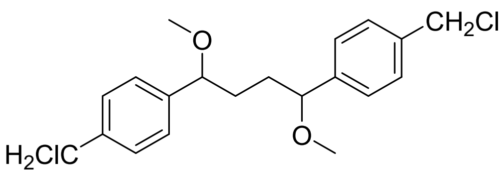
1,4-bis(2,5-dimethylphenyl)-1,4-dimethoxybutane (9): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 56.3 mg, 69% yield (dr = 9:1). Yellow oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.16 – 7.10 (m, 2H), 7.03 – 6.92 (m, 4H), 4.38 – 4.33 (m, 1.8H), 4.33 – 4.29 (m, 0.2H), 3.25 – 3.14 (m, 6H), 2.36 – 2.22 (m, 12H), 1.90 – 1.81 (m, 2H), 1.73 – 1.67 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.2, 140.1, 135.6, 132.2, 132.2, 130.3, 127.7, 126.5, 80.8, 79.8, 56.6, 34.0, 33.1, 21.1, 18.7, 18.6. HRMS (ESI): m/z: calcd for $C_{22}H_{30}O_2$ $(M+H)^+$ 327.2319; found 327.2314.



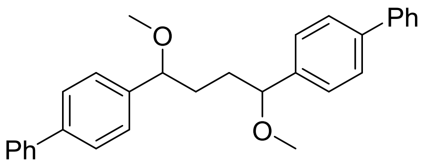
1,4-bis(2,5-dimethylphenyl)-1,4-dimethoxybutane (10):

Major diastereoisomer: R_f = 0.25 (Petroleum ether/EtOAc, 150:1). 30.1 mg, 34% yield. White solid. ^1H NMR (600 MHz, $CDCl_3$) δ 6.78 (s, 4H), 4.65 (dd, J = 7.8, 3.0 Hz, 2H), 3.16 (s, 6H), 2.31 (s, 12H), 2.24 (s, 6H), 2.14 – 2.06 (m, 2H), 1.73 – 1.62 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 136.6, 136.3, 134.1, 79.7, 56.2, 31.0, 20.8, 20.5. (ESI): m/z: calcd for $C_{24}H_{34}O_2$ $(M+Na)^+$ 377.2451; found 377.2445.

Minor diastereoisomer: R_f = 0.25 (Petroleum ether/EtOAc, 150:1). 15.2 mg, 17% yield. White solid. ^1H NMR (600 MHz, $CDCl_3$) δ 6.79 (s, 4H), 4.60 (s, 2H), 3.12 (s, 6H), 2.34 (s, 12H), 2.24 (s, 6H), 1.91 – 1.85 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 136.6, 136.2, 134.3, 80.9, 56.2, 32.2, 20.8, 20.5. (ESI): m/z: calcd for $C_{24}H_{34}O_2$ $(M+Na)^+$ 377.2451; found 377.2447.

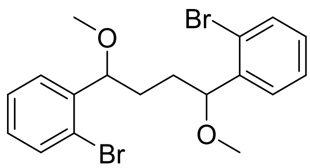


1,4-bis(4-(chloromethyl)phenyl)-1,4-dimethoxybutane (11): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 76.9 mg, 84% yield (dr = 4.0:1). Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.36 – 7.31 (m, 4H), 7.25 – 7.20 (m, 4H), 4.59 – 4.54 (m, 4H), 4.08 – 4.02 (m, 2H), 3.19 – 3.04 (m, 6H), 1.94 – 1.83 (m, 1.61H), 1.83 – 1.76 (m, 0.39H), 1.72 – 1.69 (m, 0.42H), 1.62 – 1.51 (m, 1.58H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.7, 142.6, 136.7, 136.7, 128.7, 128.6, 127.0, 127.0, 83.7, 83.4, 56.7, 56.7, 46.0, 34.7, 34.0. (ESI): m/z: calcd for $C_{20}H_{24}O_2Cl_2$ $(M+Na)^+$ 389.1046; found 389.1044.

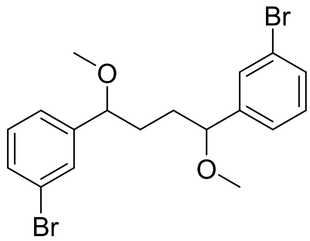


1,4-di([1,1'-biphenyl]-4-yl)-1,4-dimethoxybutane (12): R_f = 0.25 (Petroleum ether/EtOAc, 50:1). 65.5 mg, 62% yield (dr > 20:1). White solid. ^1H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.53 (m, 8H), 7.43 (t, *J* = 7.3 Hz, 4H), 7.36 – 7.28 (m, 6H), 4.12 (t, *J* = 5.0 Hz, 2H), 3.22 (s, 6H), 2.01 – 1.94 (m, 2H), 1.70 – 1.62

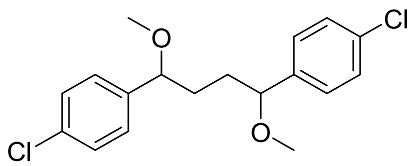
(m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.2, 140.2, 140.4, 128.7, 127.2, 127.2, 127.1, 127.1, 83.6, 56.7, 34.1. (ESI): m/z: calcd for $C_{30}H_{30}O_2$ $(M+H)^+$ 423.2319; found 423.2313.



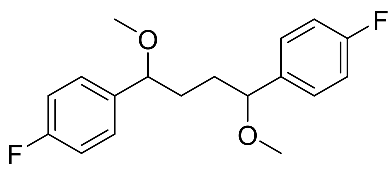
tetrahydro-2H-pyran-2,3-diyl dibenzoate (13): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 60.7 mg, 57% yield (dr = 5.6:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 – 7.49 (m, 2H), 7.45 – 7.41 (m, 2H), 7.34 – 7.29 (m, 2H), 7.15 – 7.02 (m, 2H), 4.64 – 4.61 (m, 1.7H), 4.59 – 4.56 (m, 0.3H), 3.25 – 3.19 (m, 6H), 1.95 – 1.71 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.5, 141.5, 132.7, 128.7, 127.8, 127.7, 127.5, 127.5, 123.3, 123.2, 82.2, 81.6, 57.1, 57.0, 33.3, 33.0. (ESI): m/z: calcd for $C_{18}H_{20}O_2Br_2$ $(M+Na)^+$ 448.9722; found 448.9709.



1,4-bis(3-bromophenyl)-1,4-dimethoxybutane (14): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 76.6 mg, 72% yield (dr = 4.7:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.45 – 7.34 (m, 4H), 7.23 – 7.12 (m, 4H), 4.01 (t, J = 5.8 Hz, 2H), 3.31 – 3.13 (m, 6H), 1.90 – 1.82 (m, 1.65H), 1.79 – 1.75 (m, 0.35H), 1.71 – 1.68 (m, 0.36H), 1.60 – 1.55 (m, 1.65H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 144.8, 144.7, 130.7, 130.1, 130.0, 129.7, 129.6, 125.3, 125.2, 122.7, 122.7, 83.3, 83.1, 56.9, 56.8, 34.6, 34.0. (ESI): m/z: calcd for $C_{18}H_{20}O_2Br_2$ $(M+Na)^+$ 448.9722; found 448.9712.

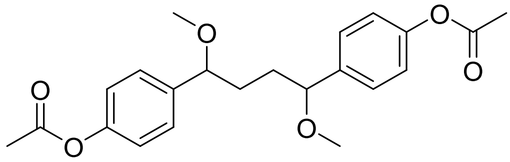


1,4-bis(4-chlorophenyl)-1,4-dimethoxybutane (15): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 76.9 mg, 91% yield (dr = 4.0:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.26 – 7.31 (m, 4H), 7.19 – 7.13 (m, 4H), 4.03 (t, J = 5.7 Hz, 2H), 3.18 – 3.12 (m, 6H), 1.88 – 1.82 (m, 1.6H), 1.76 – 1.72 (m, 0.4H), 1.68 – 1.65 (m, 0.4H), 1.55 – 1.49 (m, 1.6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.7, 140.6, 133.2, 128.6, 128.6, 128.0, 128.0, 83.3, 83.1, 56.7, 56.7, 34.5, 34.0. (ESI): m/z: calcd for $C_{18}H_{20}O_2Cl_2$ $(M+Na)^+$ 361.0733; found 361.0727.

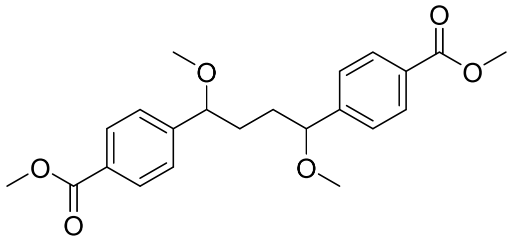


1,4-bis(4-fluorophenyl)-1,4-dimethoxybutane (16):

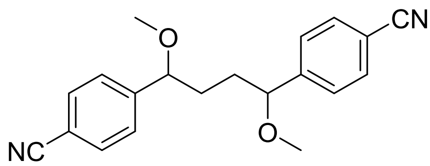
R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 53.7 mg, 58.8% yield. Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.19 (dd, *J* = 8.4, 5.6 Hz, 4H), 7.00 (t, *J* = 8.7 Hz, 4H), 4.03 (t, *J* = 5.5 Hz, 2H), 3.15 (s, 6H), 1.93 – 1.81 (m, 2H), 1.59 – 1.46 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.2 ((d, *J* = 245.4 Hz), 137.8 (d, *J* = 3.0 Hz), 128.3 (d, *J* = 8.1 Hz), 115.3 (d, *J* = 21.3 Hz), 83.1, 56.6, 34.1. ^{19}F NMR (565 MHz, $CDCl_3$) δ -115.2. (ESI): m/z: calcd for $C_{18}H_{20}O_2F_2$ $(M+Na)^+$ 329.1324; found 329.1321.



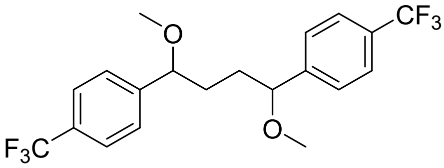
(1,4-dimethoxybutane-1,4-diyl)bis(4,1-phenylene) diacetate (17): R_f = 0.25 (Petroleum ether/EtOAc, 30:1). 40.6 mg, 42% yield (dr = 6.1:1). White solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25 – 7.21 (m, 4H), 7.06 – 7.01 (m, 4H), 4.05 (t, *J* = 5.4 Hz, 2H), 3.19 – 3.13 (m, 6H), 2.30 – 2.25 (m, 6H), 1.94 – 1.85 (m, 1.72H), 1.81 – 1.77 (m, 0.28H), 1.71 – 1.69 (m, 0.28H), 1.61 – 1.54 (m, 1.73H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.5, 150.0, 139.8, 139.7, 127.6, 127.6, 121.5, 121.4, 83.5, 83.2, 56.7, 56.69, 34.8, 34.2, 21.2. (ESI): m/z: calcd for $C_{22}H_{26}O_6$ $(M+Na)^+$ 409.1622; found 409.1611.



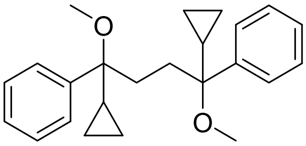
dimethyl 4,4'-(1,4-dimethoxybutane-1,4-diyl)dibenzoate (18): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 50.2 mg, 52% yield (dr = 3.4:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08 – 7.95 (m, 4H), 7.35 – 7.27 (m, 4H), 4.17 – 4.07 (m, 2H), 3.93 – 3.83 (m, 6H), 3.26 – 3.11 (m, 6H), 1.90 – 1.79 (m, 2H), 1.74 – 1.68 (m, 0.46H), 1.65 – 1.55 (m, 1.54H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.9, 147.6, 147.5, 129.8, 129.8, 129.5, 126.6, 126.5, 83.5, 83.2, 56.9, 56.9, 52.1, 34.4, 33.8. (ESI): m/z: calcd for $C_{22}H_{26}O_6$ $(M+H)^+$ 387.1802; found 387.1795.



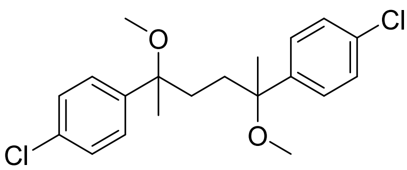
4,4'-(1,4-dimethoxybutane-1,4-diyl)dibenzonitrile (19): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 42.4 mg, 53% yield (dr = 2.6:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.66 – 7.61 (m, 4H), 7.41 – 7.32 (m, 4H), 4.17 – 4.10 (m, 2H), 3.26 – 3.16 (m, 6H), 1.86 – 1.77 (m, 2H), 1.69 – 1.67 (m, 0.53H), 1.63 – 1.58 (m, 1.37H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 147.8, 147.7, 132.4, 132.4, 127.2, 127.2, 118.8, 111.5, 83.2, 82.9, 57.1, 57.1, 34.4, 33.8. (ESI): m/z: calcd for $C_{20}H_{20}O_2N_2$ $(M+Na)^+$ 343.1417; found 343.1410.



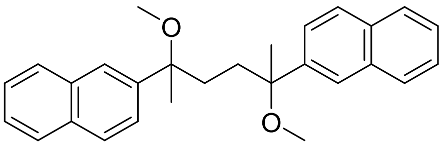
1,4-dimethoxy-1,4-bis(4-(trifluoromethyl)phenyl)butane (20): R_f = 0.25 (Petroleum ether/EtOAc, 20:1). 66.0 mg, 65% yield (dr > 20:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.1 Hz, 4H), 7.37 (d, *J* = 8.0 Hz, 4H), 4.16 – 4.10 (m, 2H), 3.18 (s, 6H), 1.95 – 1.80 (m, 2H), 1.65 – 1.55 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 146.4, 129.8 (q, *J* = 32.1 Hz), 126.8, 125.5 (q, *J* = 272.1 Hz), 124.2 (q, *J* = 3.5 Hz), 83.4, 57.0, 34.6. ^{19}F NMR (565 MHz, $CDCl_3$) δ -62.46. (ESI): m/z: calcd for $C_{20}H_{20}O_2F_2$ $(M+Na)^+$ 429.1260; found 429.1252.



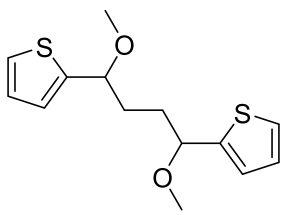
1,4-dicyclopropyl-1,4-dimethoxy-1,4-diphenylbutane (21): R_f = 0.25 (Petroleum ether/EtOAc, 150:1). 18.4 mg, 21% yield (dr = 1.4:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.9 Hz, 2H), 7.35 – 7.26 (m, 6H), 7.24 – 7.17 (m, 2H), 3.17 (s, 4H), 3.14 (s, 2H), 2.09 – 1.99 (m, 1H), 1.92 – 1.81 (m, 1H), 1.81 – 1.76 (m, 1H), 1.57 – 1.50 (m, 1H), 1.15 – 1.06 (m, 2H), 0.51 – 0.29 (m, 7H), 0.23 – 0.17 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.4, 141.4, 125.4, 125.4, 124.7, 124.6, 124.3, 124.3, 77.8, 74.5, 47.8, 47.7, 25.9, 25.9, 17.4, 17.4, -1.0, -1.2. (ESI): m/z: calcd for $C_{24}H_{30}O_2$ $(M+Na)^+$ 373.2138; found 373.2132.



4,4'-(2,5-dimethoxyhexane-2,5-diyl)bis(chlorobenzene) (22): R_f = 0.25 (Petroleum ether/EtOAc, 150:1). 51.3 mg, 56% yield (dr = 2.3:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.28 – 7.23 (m, 4H), 7.23 – 7.14 (m, 4H), 3.03 – 2.94 (m, 6H), 1.62 – 1.54 (m, 4H), 1.43 (s, 4.2H), 1.41 (s, 1.8H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 143.7, 143.5, 132.5, 132.5, 128.2, 128.2, 127.6, 127.6, 78.4, 78.4, 50.2, 36.2, 36.2, 23.2, 23.0. (ESI): m/z: calcd for $C_{20}H_{24}O_2Cl_2$ $(M+Na)^+$ 389.1046; found 389.1040.



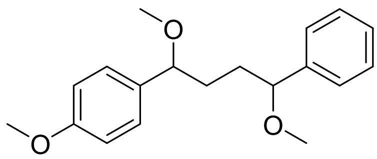
2,2'-(2,5-dimethoxyhexane-2,5-diyl)dinaphthalene (23): R_f = 0.25 (Petroleum ether/EtOAc, 30:1). 40.8 mg, 41% yield (dr = 5.5:1). White solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 – 7.64 (m, 8H), 7.47 – 7.42 (m, 4.59H), 7.37 – 7.33 (m, 1.41H), 3.05 – 2.99 (m, 6H), 1.86 – 1.77 (m, 2H), 1.76 – 1.69 (m, 2H), 1.56 (s, 4.23H), 1.54 (s, 1.77H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.5, 142.3, 133.1, 133.1, 132.4, 128.2, 127.9, 127.8, 127.5, 127.4, 125.9, 125.7, 125.7, 125.1, 125.1, 124.5, 124.5, 79.0, 79.0, 50.3, 36.3, 36.1, 23.1, 22.9. (ESI): m/z: calcd for $C_{28}H_{30}O_2$ $(M+Na)^+$ 421.2138; found 421.2128.



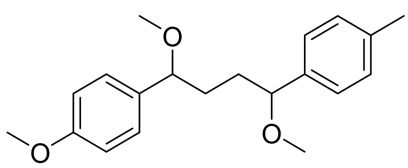
1,4-dimethoxy-1,4-di(thiophen-2-yl)butane (24):

Major diastereoisomer: R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 19.7 mg, 28% yield. Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.26 – 7.23 (m, 2H), 7.03 – 6.90 (m, 4H), 4.35 (t, J = 5.4 Hz, 2H), 3.24 (s, 6H), 2.09 – 1.96 (m, 2H), 1.75 – 1.66 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 145.9, 126.3, 125.3, 124.9, 79.1, 56.4, 34.4. (ESI): m/z: calcd for $C_{14}H_{18}O_2S_2$ $(M+Na)^+$ 305.0640; found 305.0639.

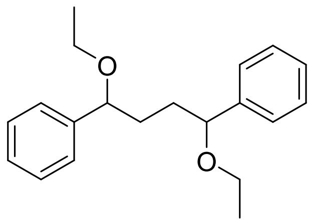
Minor diastereoisomer: R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 7.1 mg, 10% yield. Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 2H), 7.00 – 6.90 (m, 4H), 4.39 – 4.29 (m, 2H), 3.23 (s, 6H), 1.94 – 1.84 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 146.0, 126.4, 125.2, 124.9, 79.2, 56.5, 34.8. (ESI): m/z: calcd for $C_{14}H_{18}O_2S_2$ $(M+Na)^+$ 305.0640; found 305.0635.



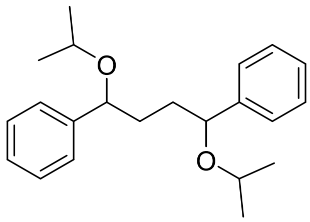
1-(1,4-dimethoxy-4-phenylbutyl)-4-methoxybenzene (25): R_f = 0.25 (Petroleum ether/EtOAc, 50:1). 30.0 mg, 40% yield (dr = 1.1:1). Colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.26 (m, 3H), 7.25 – 7.20 (m, 2H), 7.19 – 7.07 (m, 2H), 6.91 – 6.80 (m, 2H), 4.01 – 3.94 (m, 2H), 3.83 – 3.75 (m, 3H), 3.21 – 3.09 (m, 6H), 1.95 – 1.84 (m, 1.06H), 1.80 – 1.70 (m, 1.96H), 1.59 – 1.51 (m, 1.04H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.1, 159.0, 142.3, 142.2, 134.3, 134.2, 128.4, 128.3, 127.9, 127.8, 127.5, 127.5, 126.7, 126.6, 113.8, 113.7, 84.1, 83.9, 83.6, 83.4, 56.6, 56.6, 56.4, 56.3, 55.3, 55.2, 34.7, 34.6, 34.2, 34.0. (ESI): m/z: calcd for $C_{19}H_{24}O_3$ $(M+Na)^+$ 323.1618; found 323.1615.



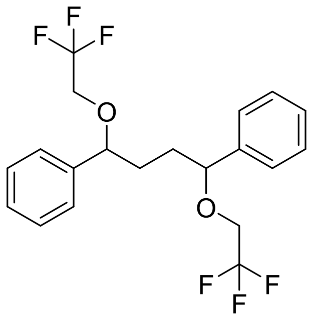
1-(1,4-dimethoxy-4-(4-methoxyphenyl)butyl)-4-methylbenzene (26): R_f = 0.25 (Petroleum ether/EtOAc, 50:1). 36.9 mg, 47% yield (dr = 2.3:1). Colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.18 – 7.09 (m, 6H), 6.89 – 6.80 (m, 2H), 4.05 – 3.96 (m, 2H), 3.75 – 3.83 (m, 3H), 3.20 – 3.09 (m, 6H), 2.37 – 2.28 (m, 3H), 1.96 – 1.83 (m, 1.2H), 1.77 – 1.72 (m, 1.4H), 1.58 – 1.49 (m, 1.2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.1, 159.0, 139.2, 139.1, 137.1, 137.1, 134.3, 134.2, 129.1, 129.0, 127.9, 127.8, 126.7, 126.6, 113.8, 113.7, 83.9, 83.7, 83.6, 83.4, 56.5, 56.5, 56.4, 56.3, 55.2, 34.7, 34.6, 34.2, 34.1, 21.1. (ESI): m/z: calcd for $C_{20}H_{26}O_3$ $(M+Na)^+$ 377.1774; found 377.1776.



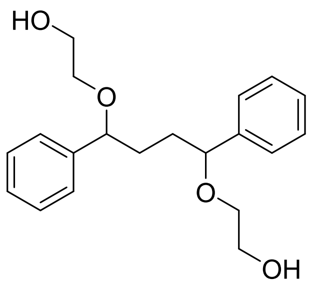
1,4-diethoxy-1,4-diphenylbutane (27): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 37.3 mg, 50% yield (dr = 2.1:1). Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.38 – 7.18 (m, 10H), 4.15 (t, *J* = 5.5 Hz, 2H), 3.36 – 3.29 (m, 2H), 3.29 – 3.31 (m, 2H), 1.99 – 1.90 (m, 1.35H), 1.86 – 1.80 (m, 0.65H), 1.76 – 1.71 (m, 0.64H), 1.59 – 1.51 (m, 1.36H), 1.18 – 1.10 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 143.1, 143.0 128.3, 128.3, 127.3, 126.7, 126.6, 82.3, 82.0, 64.1, 64.1, 35.1, 34.5, 15.4, 15.3. (ESI): m/z: calcd for $C_{20}H_{26}O_2$ $(M+H)^+$ 299.2006; found 299.2003.



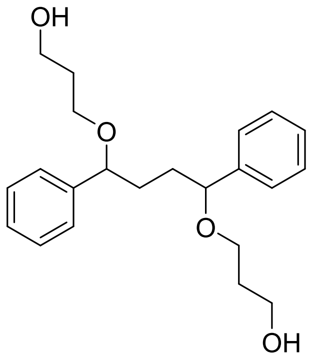
1,4-diisopropoxy-1,4-diphenylbutane (28): R_f = 0.25 (Petroleum ether/EtOAc, 10:1). 33.4 mg, 41% yield (dr = 1.7:1). Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.35 – 7.20 (m, 10H), 4.33 – 4.26 (m, 2H), 3.52 – 3.33 (m, 2H), 1.93 – 1.85 (m, 1.27H), 1.85 – 1.77 (m, 0.73H), 1.66 – 1.47 (m, 2H), 1.14 – 1.02 (m, 12H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 144.0, 143.8, 128.2, 128.2, 127.2, 127.2, 126.7, 126.6, 79.3, 79.0, 68.8, 68.6, 35.7, 35.0, 23.5, 21.3, 21.2. (ESI): m/z: calcd for $C_{22}H_{30}O_2$ $(M+H)^+$ 327.2319; found 327.2314.



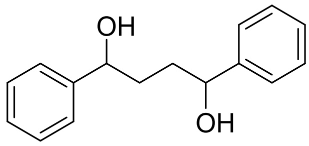
1,4-diphenyl-1,4-bis(2,2,2-trifluoroethoxy)butane (29): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 35.5 mg, 35% yield (dr = 1.2:1). Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.37 – 7.24 (m, 10H), 4.46 – 4.38 (m, 2H), 3.71 – 3.61 (m, 2H), 3.61 – 3.51 (m, 2H), 2.03 – 1.96 (m, 1.1H), 1.89 – 1.86 (m, 1.81H), 1.76 – 1.67 (m, 1.1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.5, 140.4, 128.7, 128.7, 128.2, 128.2, 126.7, 126.6, 124.1 (q, *J* = 8.3 Hz), 83.8, 83.1, 65.9 (q, *J* = 34.1 Hz), 65.8 (q, *J* = 34.1 Hz), 34.3, 33.8. ^{19}F NMR (565 MHz, $CDCl_3$) δ -73.92, -73.97. (ESI): m/z: calcd for $C_{20}H_{20}O_2F_6$ $(M+H)^+$ 429.1260; found 429.1250.



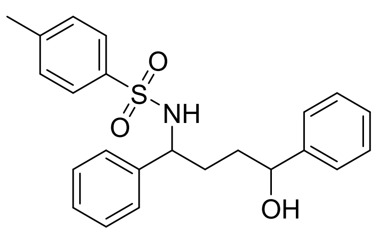
2,2'-((1,4-diphenylbutane-1,4-diyl)bis(oxy))bis(ethan-1-ol) (30): R_f = 0.25 (Petroleum ether/EtOAc, 3:1). 27.2 mg, 33% yield (dr = 1.1:1). Blue oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41 – 7.19 (m, 10H), 4.37 – 4.27 (m, 0.96H), 4.27 – 4.20 (m, 1.04H), 3.75 – 3.60 (m, 4H), 3.47 – 3.31 (m, 4H), 2.31 (s, 2H), 2.01 – 1.93 (m, 1H), 1.92 – 1.84 (m, 1H), 1.83 – 1.75 (m, 1H), 1.71 – 1.63 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.3, 128.5, 128.5, 127.7, 126.6, 126.5, 82.8, 82.6, 70.2, 70.1, 62.0, 62.0, 34.8, 34.3. (ESI): m/z: calcd for $C_{20}H_{26}O_4$ $(M+K)^+$ 369.1463; found 369.1464.



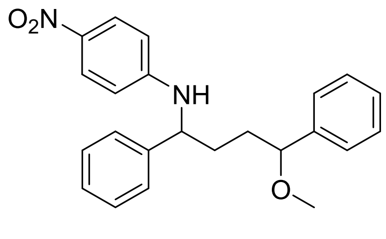
3,3'-((1,4-diphenylbutane-1,4-diyl)bis(oxy))bis(propan-1-ol) (31): R_f = 0.25 (Petroleum ether/EtOAc, 3:1). 26.7 mg, 30% yield (dr = 1:1). Blue soild. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.36 – 7.22 (m, 10H), 4.22 – 4.12 (m, 2H), 3.80 – 3.67 (m, 4H), 3.49 – 3.38 (m, 4H), 2.50 (s, 2H), 1.94 – 1.87 (m, 1.1H), 1.83 – 1.72 (m, 6.03H), 1.60 – 1.56 (m, 1.03H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.2, 142.1, 128.5, 128.5, 127.7, 126.6, 126.5, 82.7, 82.7, 67.9, 67.8, 62.0, 61.9, 34.5, 34.2, 32.2, 32.1. (ESI): m/z: calcd for $C_{22}H_{30}O_4$ $(M+Na)^+$ 381.2036; found 381.2035.



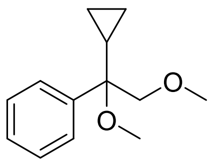
1,4-diphenylbutane-1,4-diol (32): R_f = 0.25 (Petroleum ether/EtOAc, 3:1). 7.3 mg, 12% yield (dr = 2:1). Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 8H), 7.27 – 7.24 (m, 2H), 4.75 – 4.70 (m, 0.67H), 4.70 – 4.66 (m, 1.33H), 2.45 (s, 2H), 1.94 – 1.77 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 144.6, 144.6, 128.5, 127.5, 127.5, 125.8, 74.6, 74.3, 35.7, 35.2. (ESI): m/z: calcd for $C_{16}H_{18}O_2$ $(M+Na)^+$ 265.1199; found 265.1203.



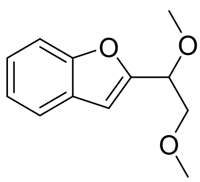
***N*-(4-hydroxy-1,4-diphenylbutyl)-4-methylbenzenesulfonamide (33)**: R_f = 0.25 (Petroleum ether/EtOAc, 10:1). 29.7 mg, 48% yield (dr = 1.8:1). Yellow oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.90 – 7.79 (m, 2H), 7.42 – 6.91 (m, 11H), 6.85 – 6.72 (m, 1H), 4.98 – 4.92 (m, 0.65H), 4.86 – 4.77 (m, 0.35H), 4.61 – 4.56 (m, 0.36H), 4.53 – 4.48 (m, 0.64H), 4.14 – 4.05 (m, 0.36H), 3.98 – 3.93 (m, 0.64H), 2.45 (s, 3H), 2.02 – 1.91 (m, 2H), 1.82 – 1.65 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 146.3, 146.2, 146.2, 143.5, 143.5, 143.5, 140.1, 139.8, 138.2, 138.1, 136.4, 136.0, 130.4, 130.2, 129.8, 129.0, 128.8, 128.7, 128.5, 128.4, 128.0, 127.8, 127.2, 127.2, 126.9, 126.8, 126.4, 126.3, 52.3, 52.1, 45.4, 44.9, 28.7, 28.5, 21.6. (ESI): m/z: calcd for $C_{23}H_{25}O_3NS$ $(M+Na)^+$ 418.1447; found 418.1500.



N-(4-methoxy-1,4-diphenylbutyl)-4-nitroaniline (34): R_f = 0.25 (Petroleum ether/EtOAc, 10:1). 48.1 mg, 73% yield (dr = 1.2:1). Yellow oil. ^1H NMR (600 MHz, $CDCl_3$) δ 8.04 – 7.91 (m, 2H), 7.38 – 7.21 (m, 10H), 6.49 – 6.36 (m, 2H), 5.32 (s, 1H), 4.44 – 4.37 (m, 0.54H), 4.36 – 4.29 (m, 0.47H), 4.17 – 4.04 (m, 1H), 3.40 – 3.39 (m, 3H), 2.08 – 1.76 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 152.8, 152.6, 142.3, 142.0, 141.5, 141.5, 138.0, 128.9, 128.9, 128.6, 127.9, 127.9, 127.6, 127.6, 126.6, 126.5, 126.2, 126.2, 111.8, 83.7, 83.5, 58.2, 57.9, 56.8, 56.7, 35.0, 34.7, 34.5, 34.2. (ESI): m/z: calcd for $C_{23}H_{25}O_3N_2$ $(M+Na)^+$ 399.1679; found 399.1682.



(1-cyclopropyl-1,2-dimethoxyethyl)benzene (A)[3]: R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 53.7 mg, 53% yield. Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.46 – 7.38 (m, 2H), 7.36 – 7.28 (m, 2H), 7.25 (t, J = 6.8 Hz, 1H), 3.70 – 3.61 (m, 2H), 3.36 – 3.30 (m, 3H), 3.27 – 3.20 (m, 3H), 1.29 – 1.23 (m, 1H), 0.59 – 0.46 (m, 2H), 0.46 – 0.35 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.1, 125.8, 125.1, 125.0, 78.1, 57.3, 49.1, 16.0, -0.0, -0.9.



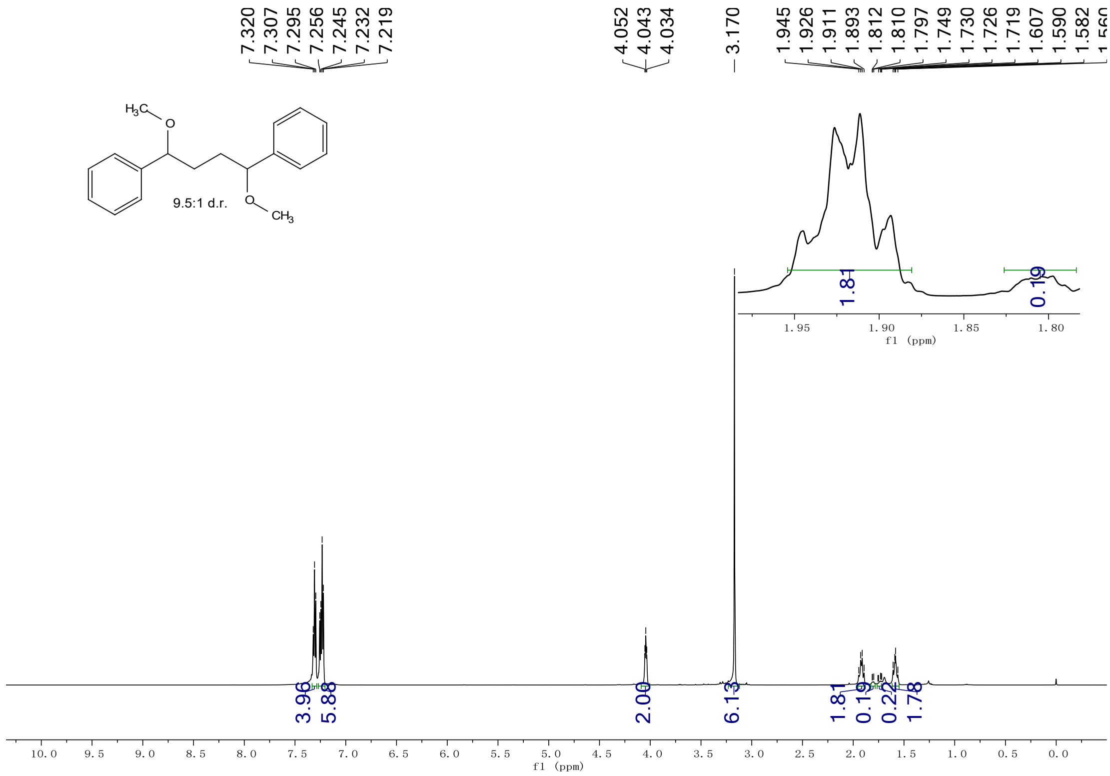
2-(1,2-dimethoxyethyl)benzofuran (B): R_f = 0.25 (Petroleum ether/EtOAc, 100:1). 46.4 mg, 45% yield (dr = 1.2:1). Colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.6 Hz, 1H), 7.48 (d, *J* = 8.2 Hz, 1H), 7.30 – 7.25 (m, 1H), 7.22 (t, *J* = 7.4 Hz, 1H), 6.73 (s, 1H), 4.56 (dd, *J* = 7.0, 4.2 Hz, 1H), 3.83 (dd, *J* = 10.0, 7.7 Hz, 1H), 3.75 – 3.70 (m, 1H), 3.47 – 3.34 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 155.0, 128.0, 124.4, 122.9, 121.1, 111.4, 105.4, 76.6, 74.1, 59.4, 57.4. (ESI): m/z: calcd for $C_{16}H_{18}O_2$ $(M+Na)^+$ 229.0835; found 229.0836.

6. References

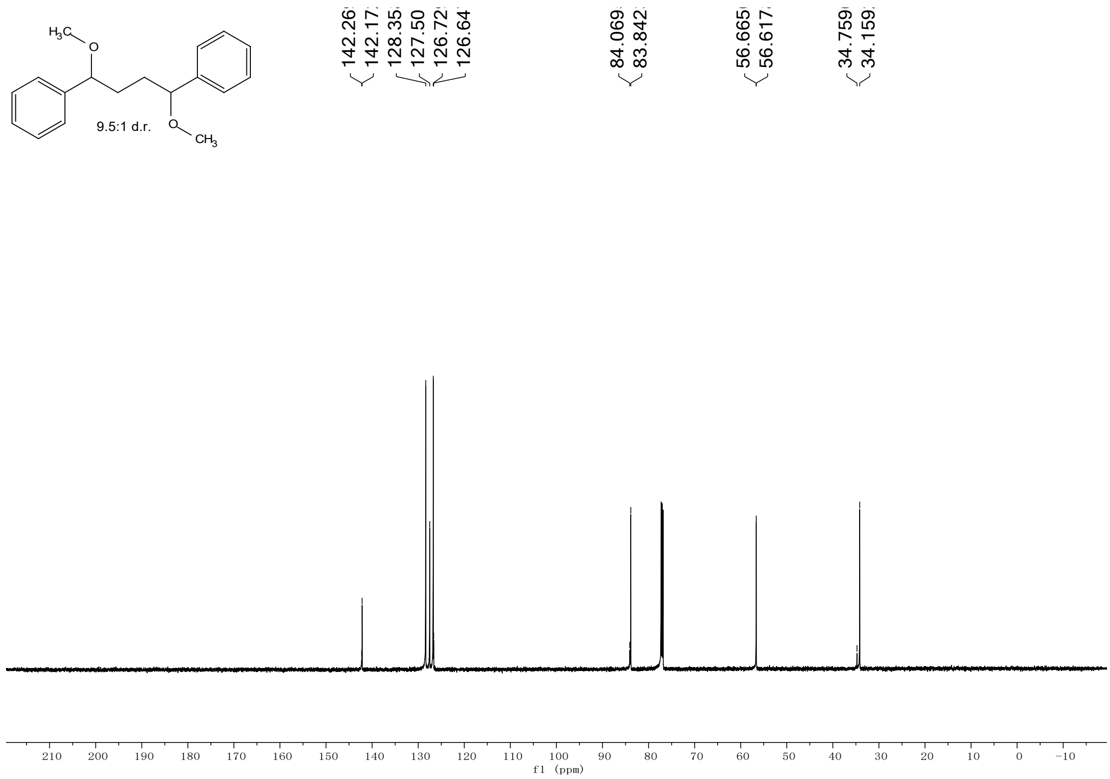
1 R. Engels, Schäfer J. Hans, E. Steckhan, *Liebigs Ann. Chem.*, **1977**, *1977*, 204–224.
2 V. Nair, V. Sheeba, S. B. Panicker, T. G. George, R. Rajan, L. Balagopal, M. Vairamani, S. Prabhakar, *Tetrahedron*, **2000**, *56*, 2461–2467.
3 S. Zhang, L. Li, P. Gong, R. Liu, K. Xu, *Adv. Synth. Catal.*, **2019**, *361*, 485–489.

7. NMR spectra of products

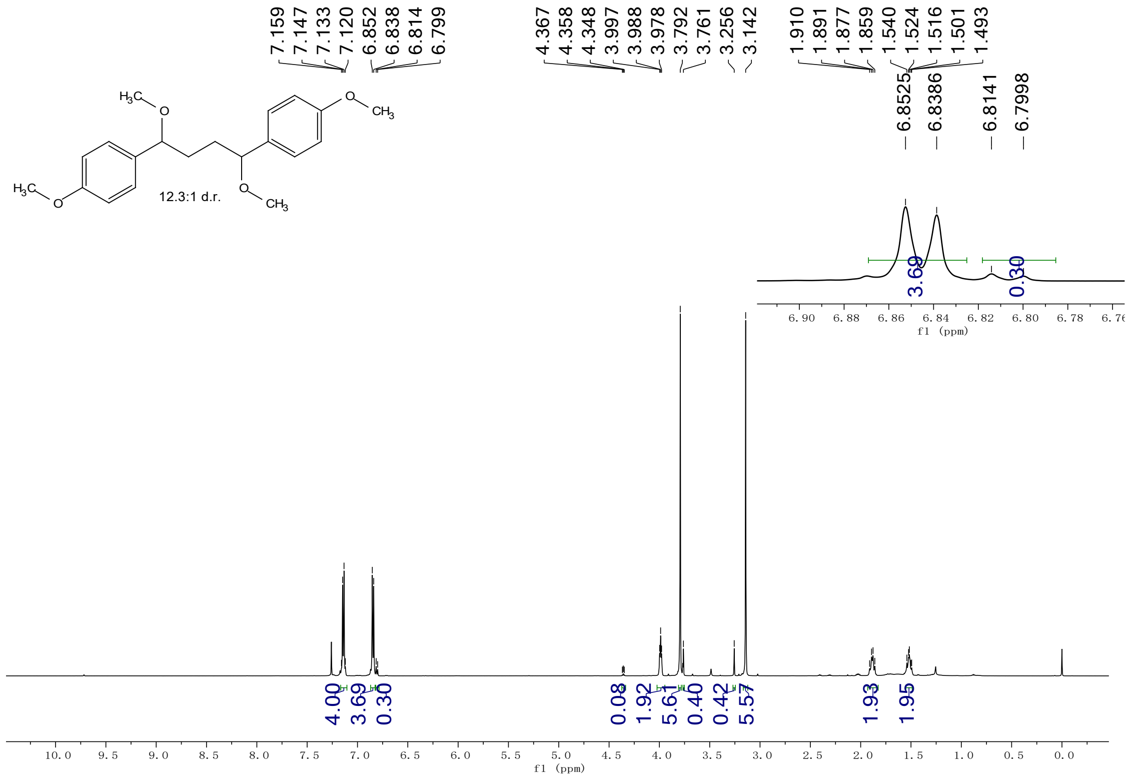
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 2



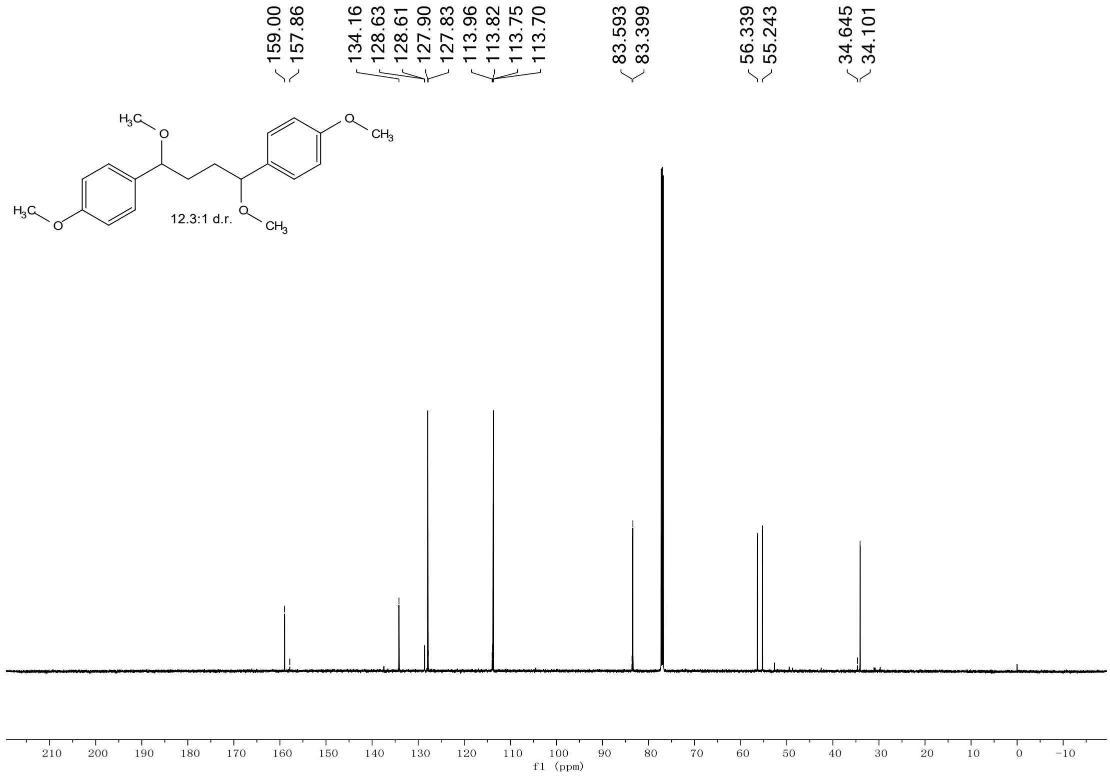
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 2



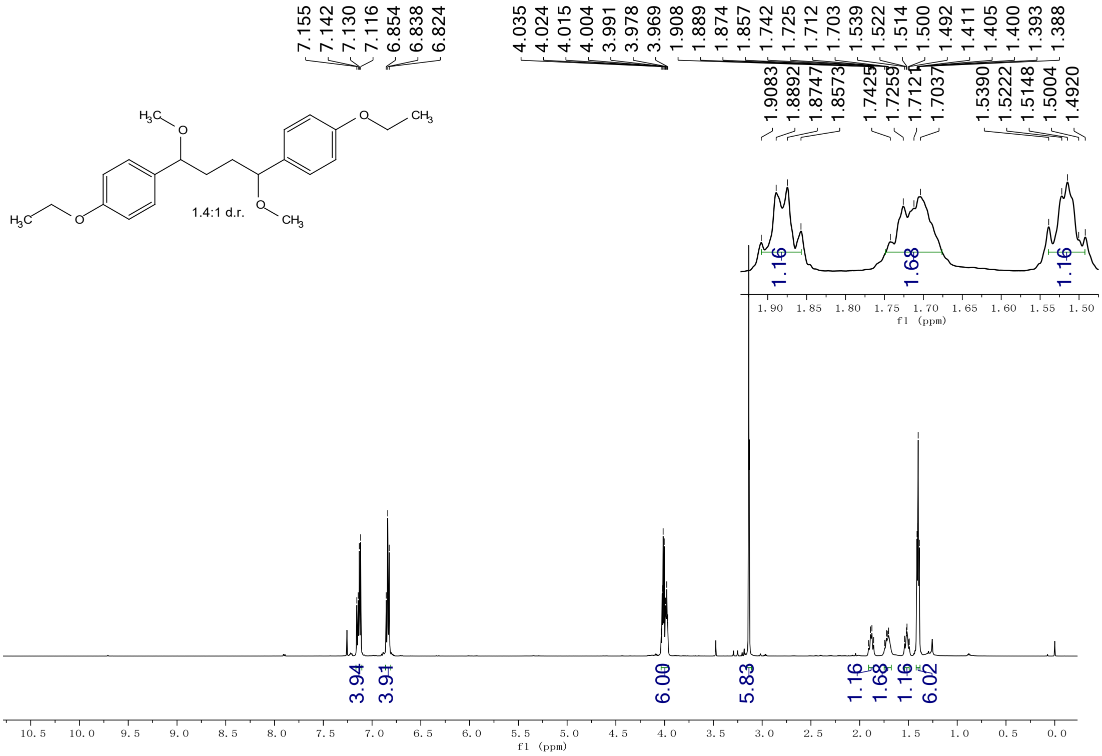
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 3



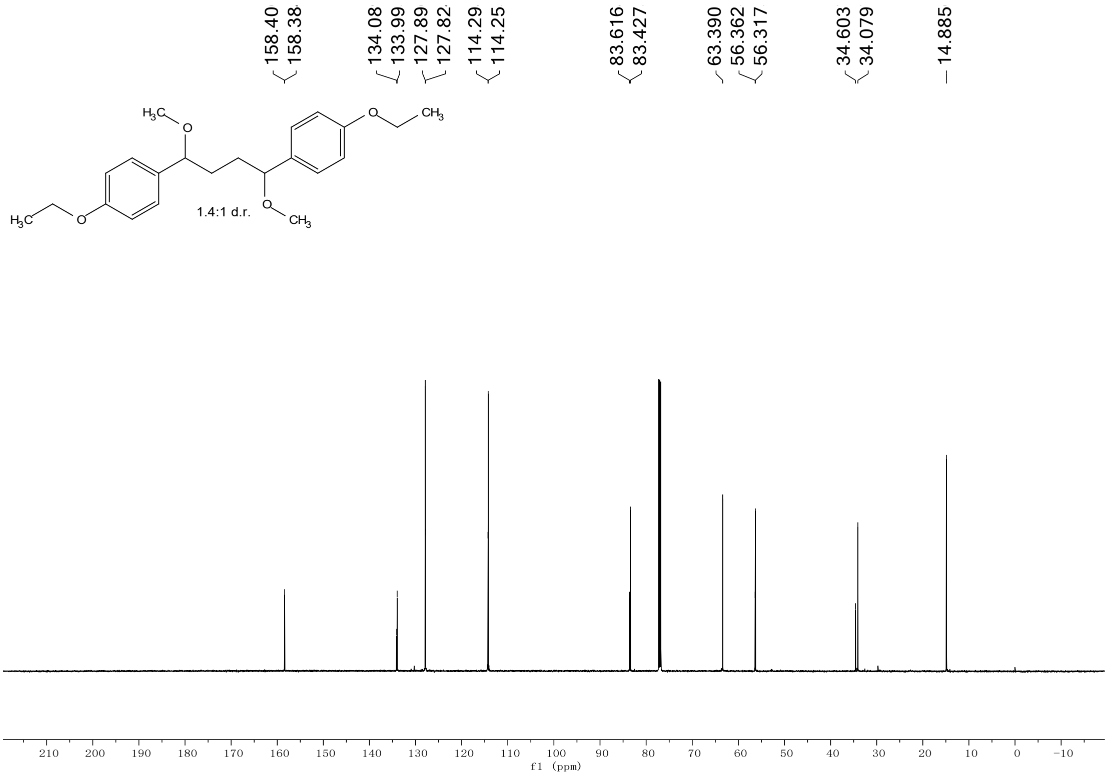
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 3



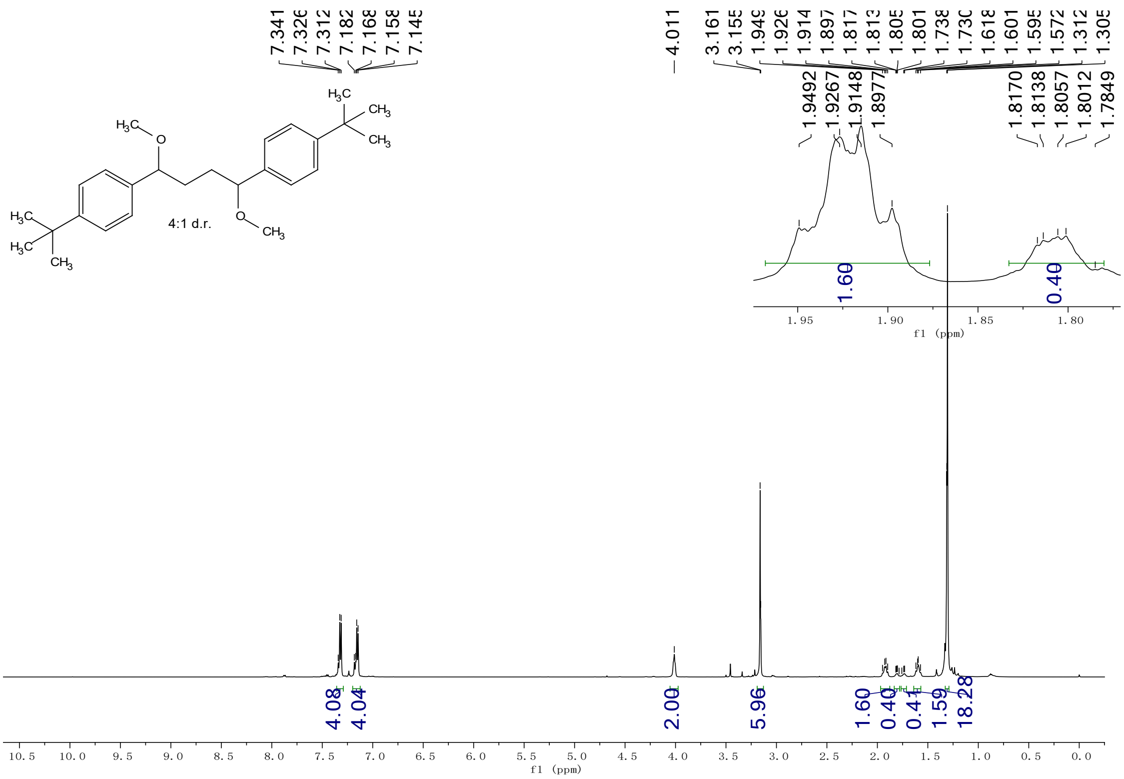
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 4



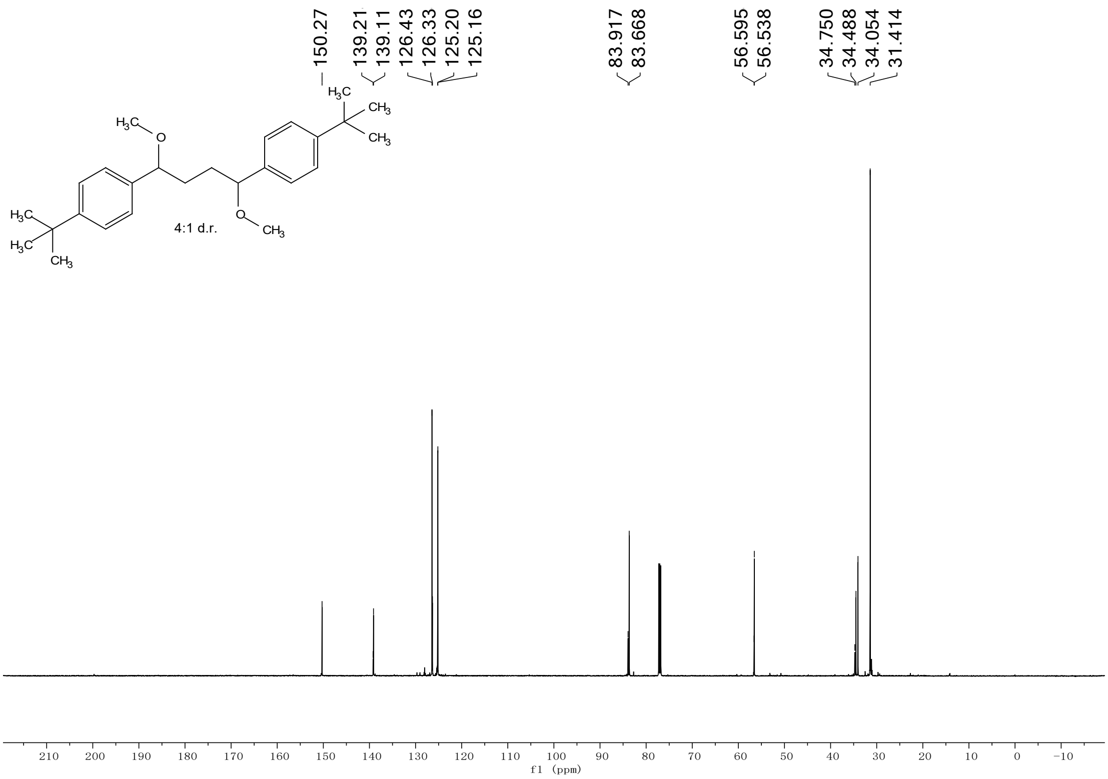
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 4



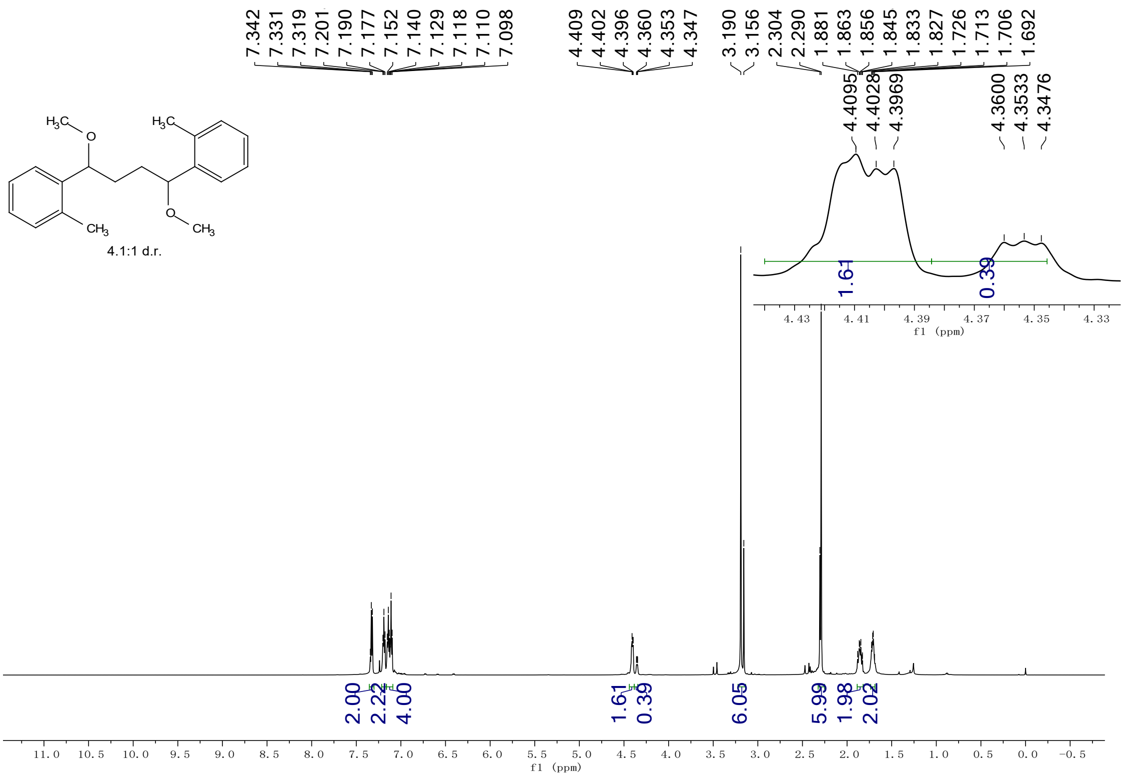
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 5



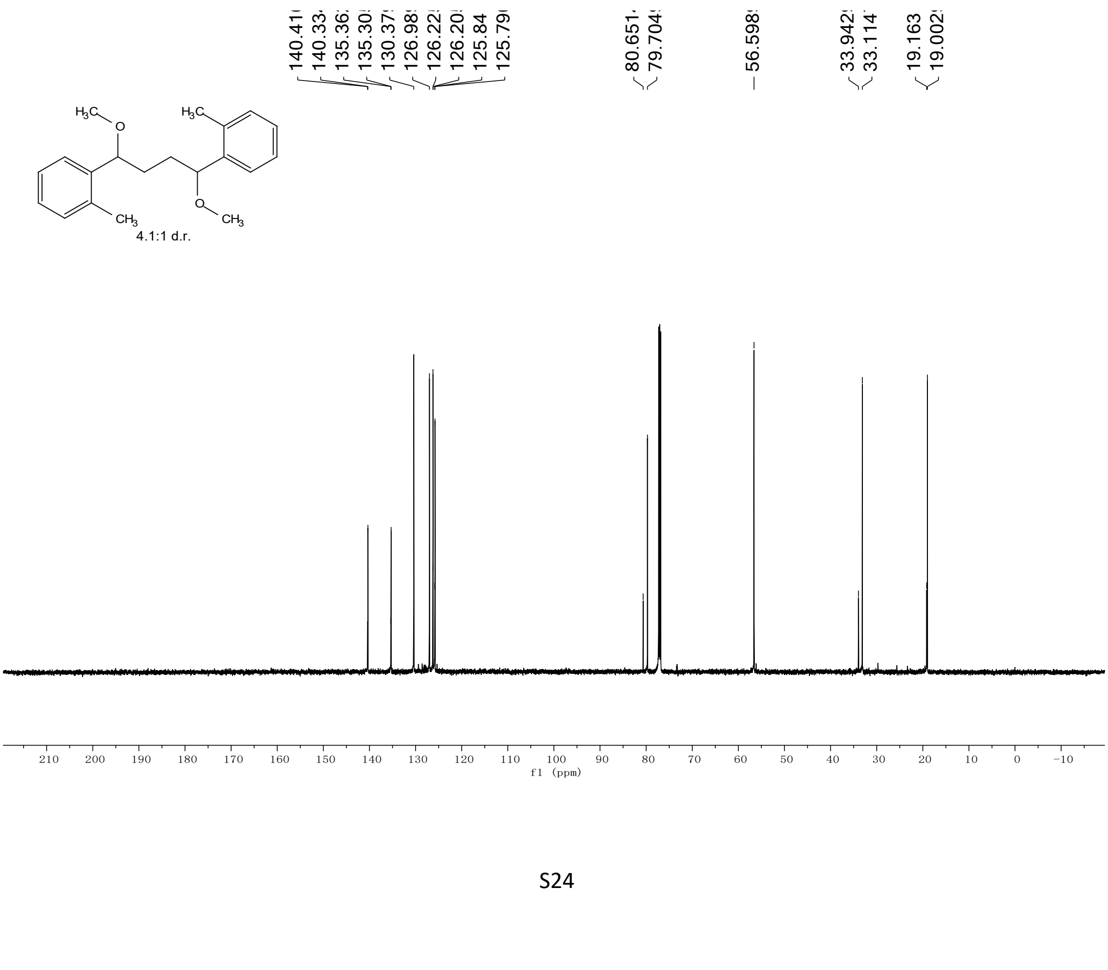
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 5



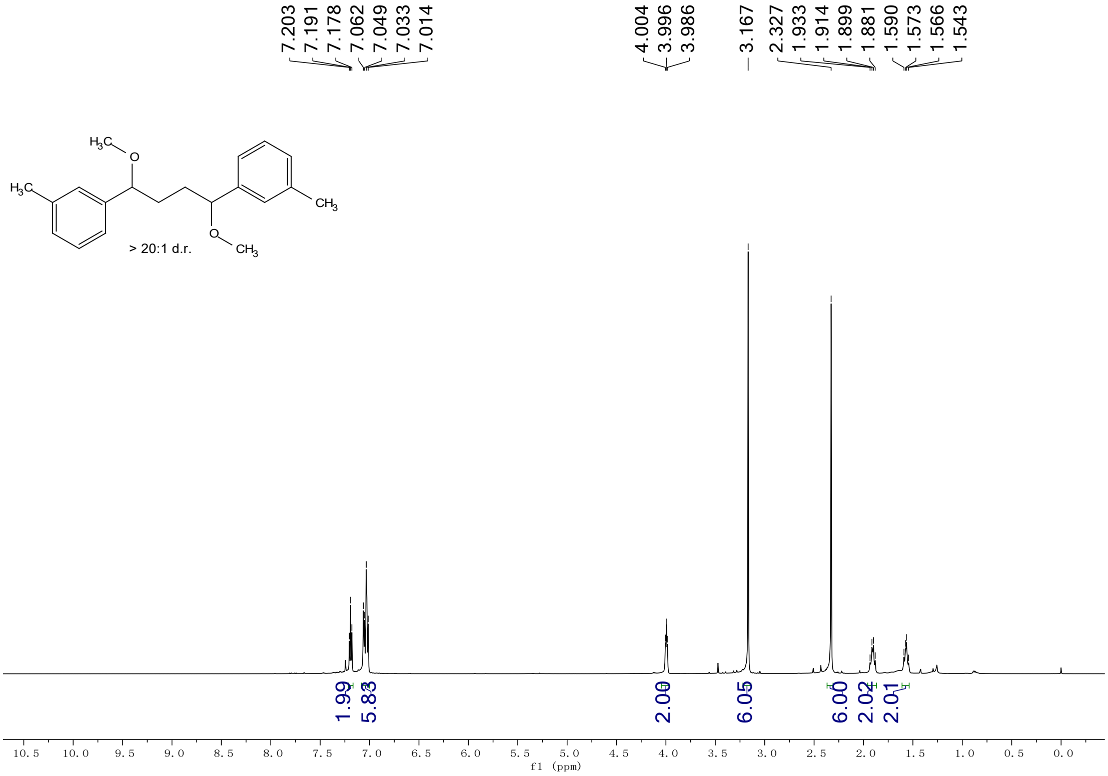
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 6



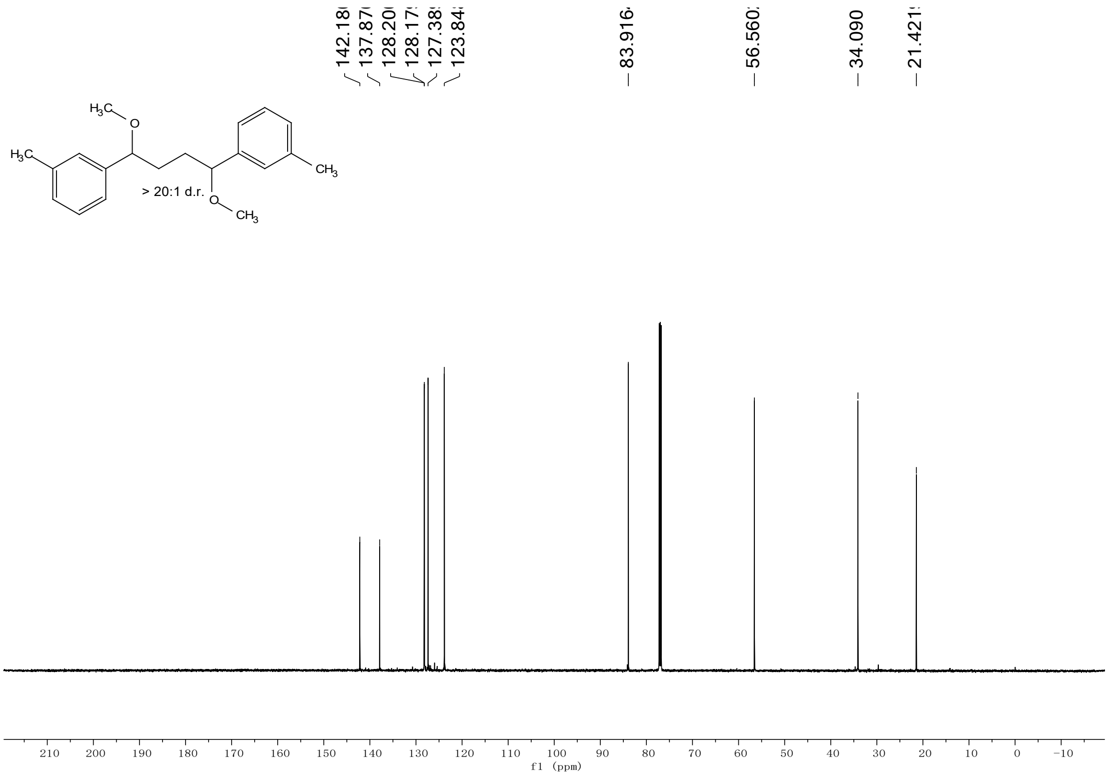
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 6



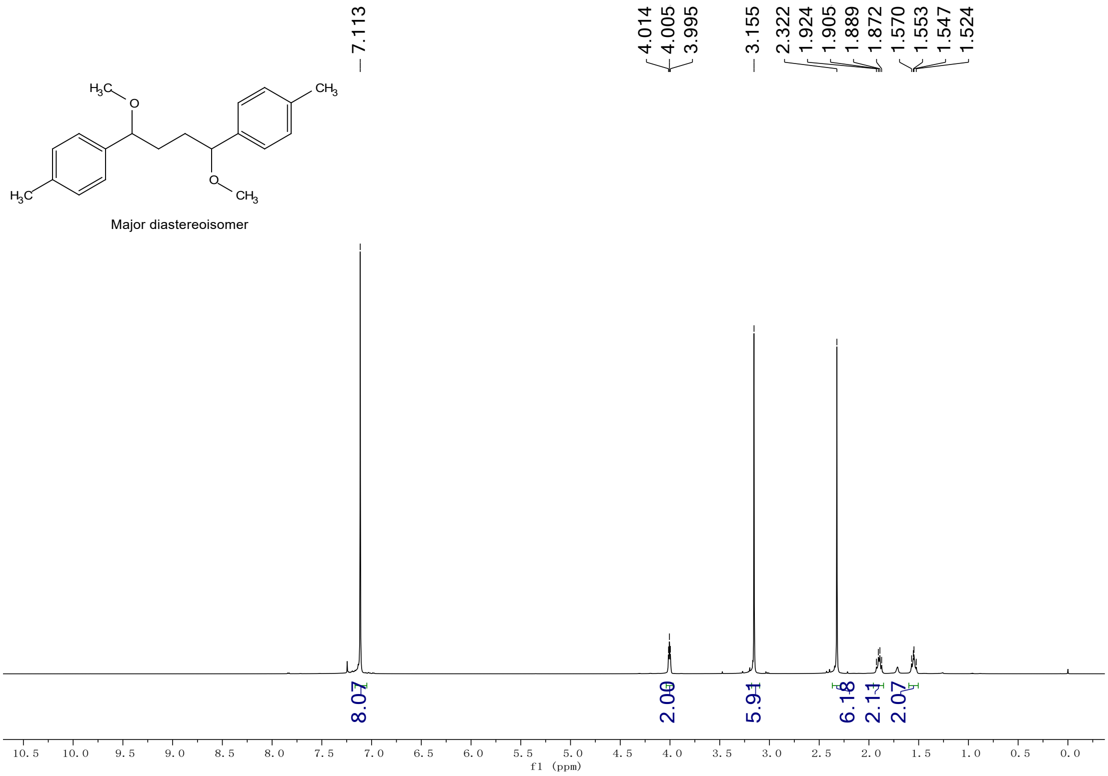
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 7



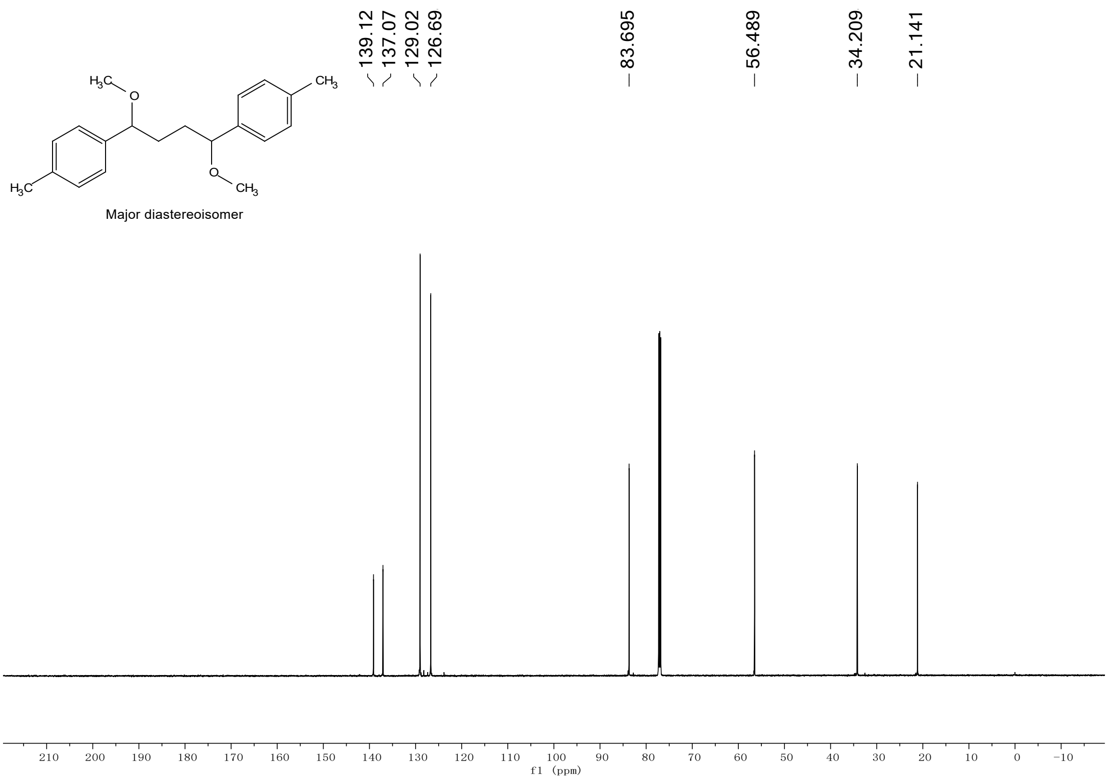
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 7



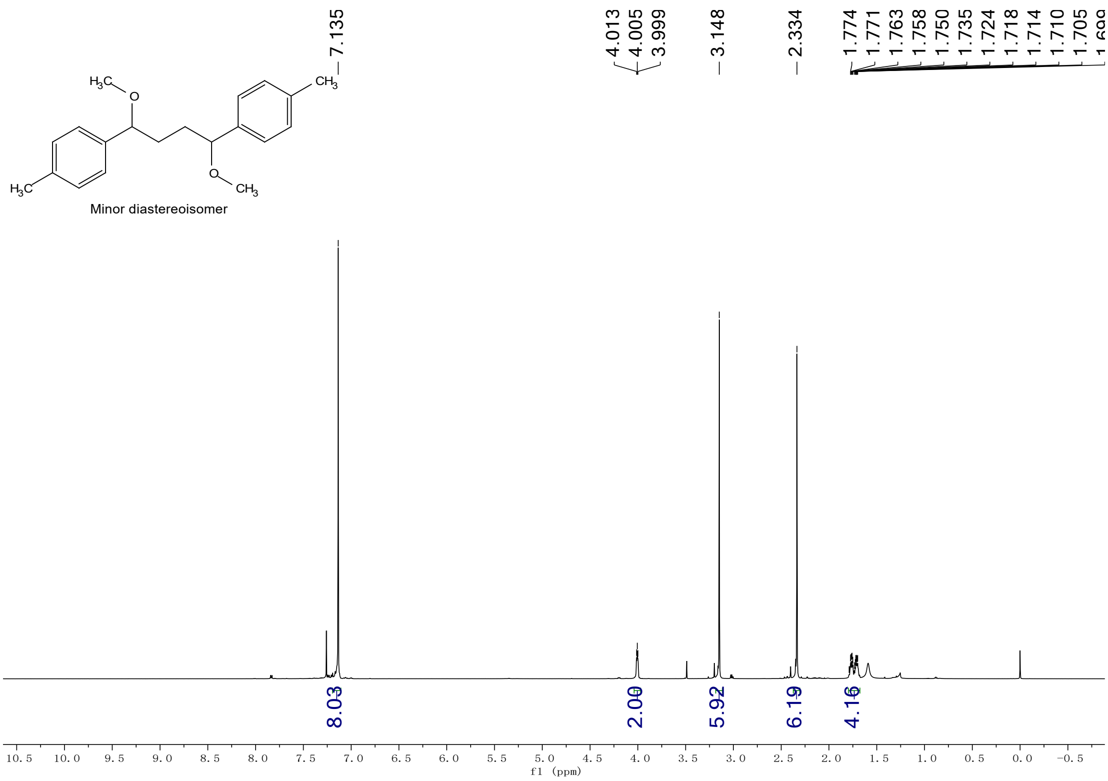
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 8 (major diastereoisomer)



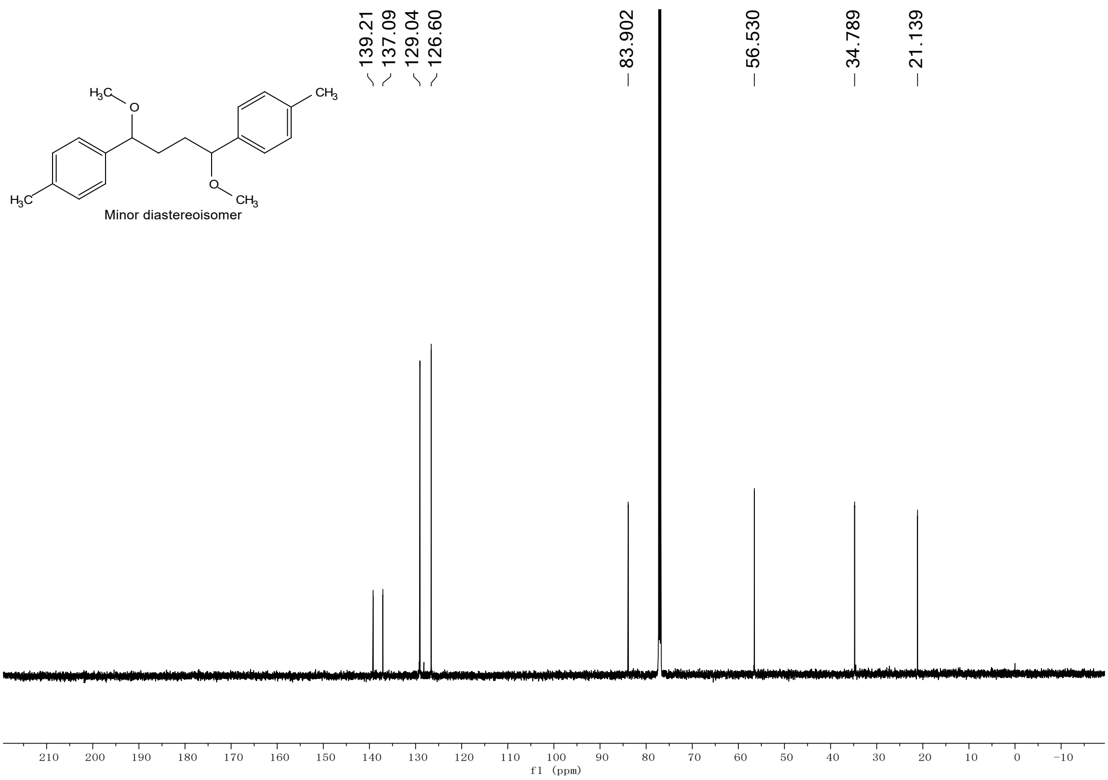
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 8 (major diastereoisomer)



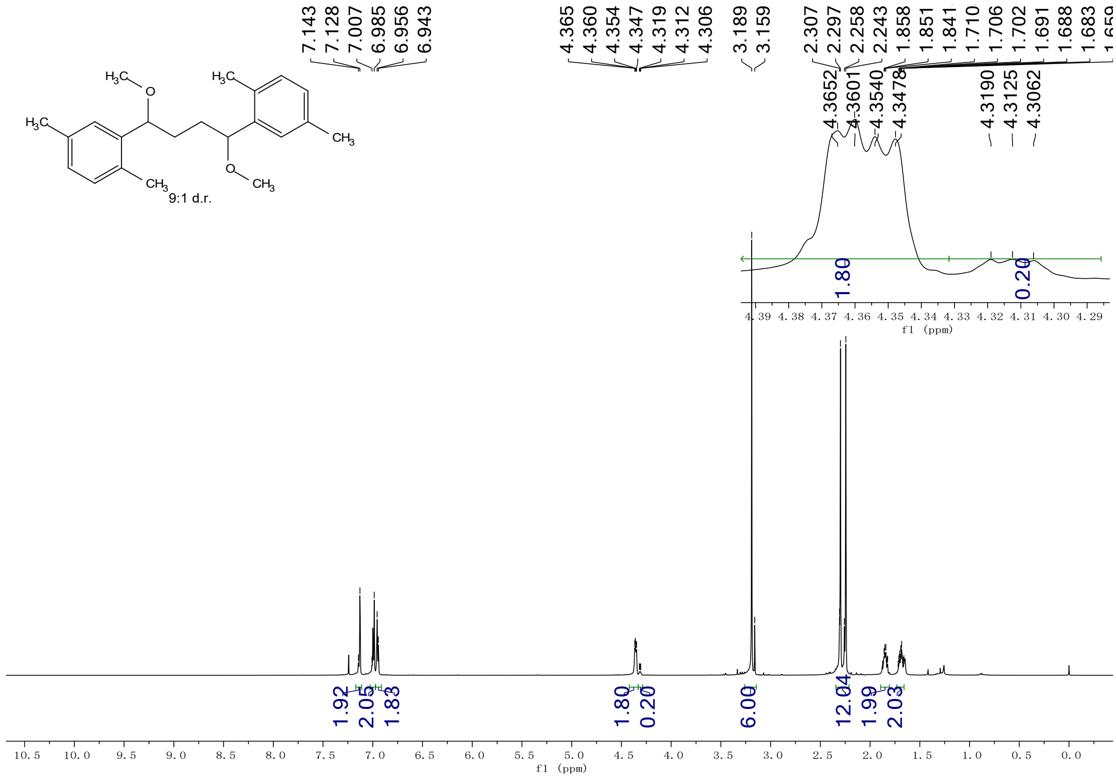
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 8 (minor diastereoisomer)



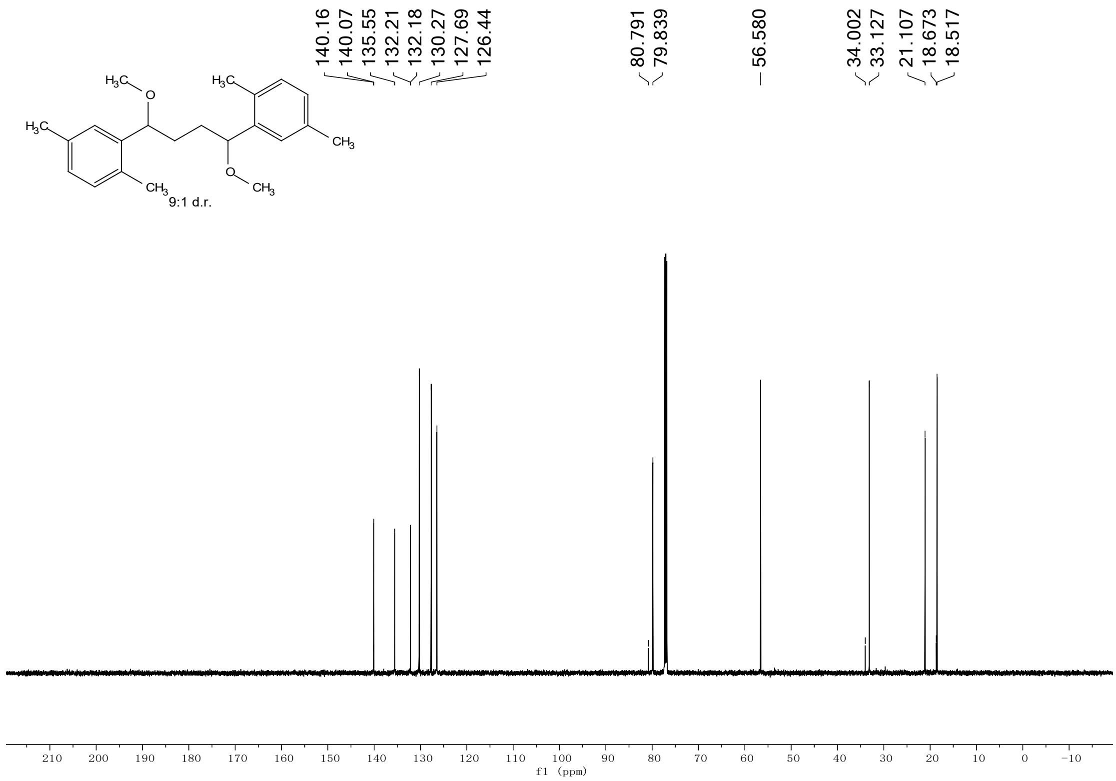
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 8 (minor diastereoisomer)



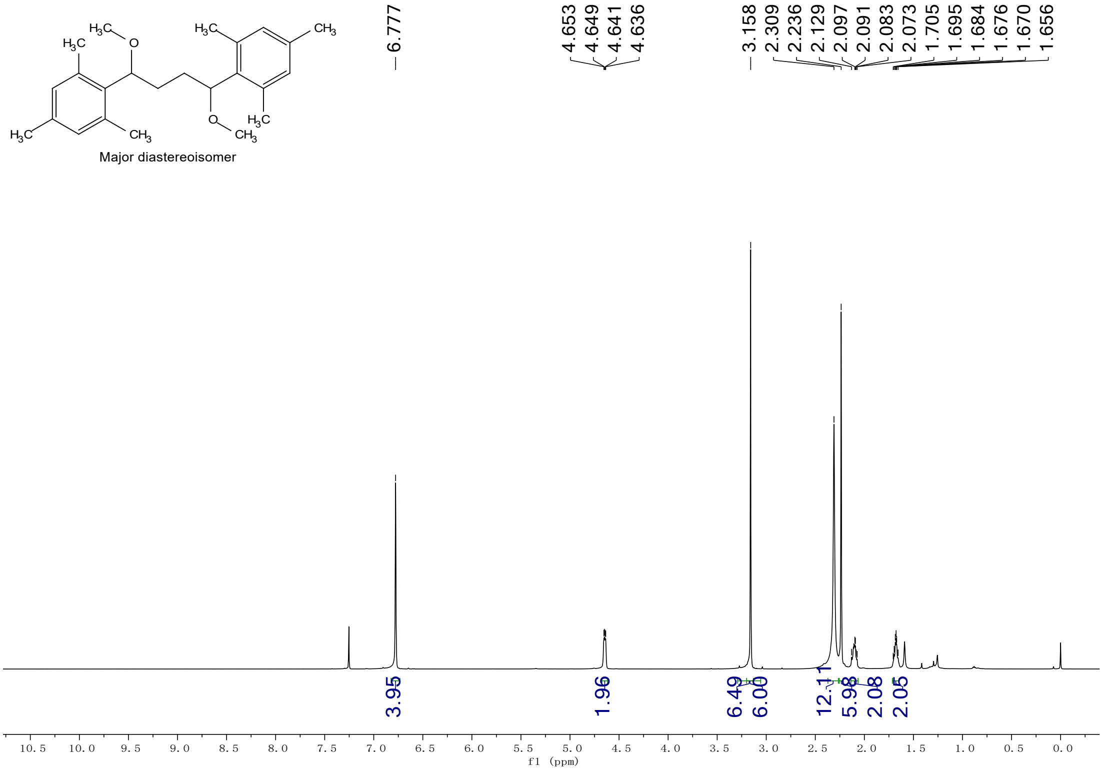
¹H-NMR Spectrum (600 MHz, $CDCl_3$) of 9



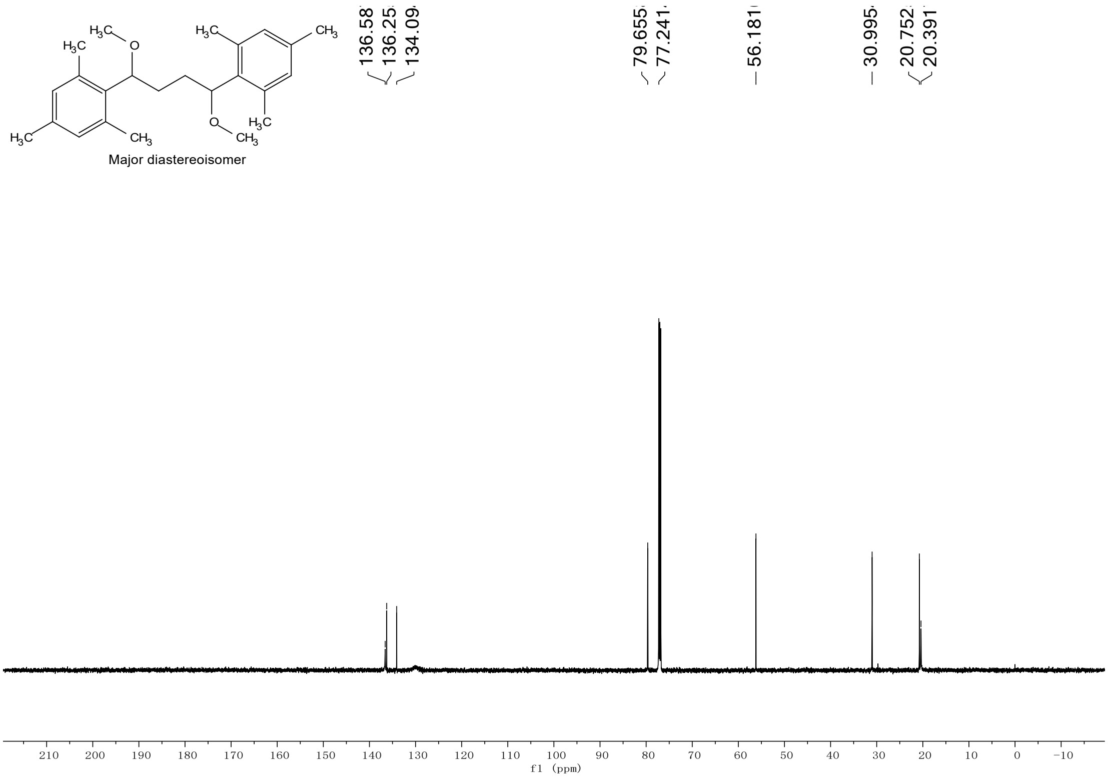
¹³C-NMR Spectrum (151 MHz, $CDCl_3$) of 9



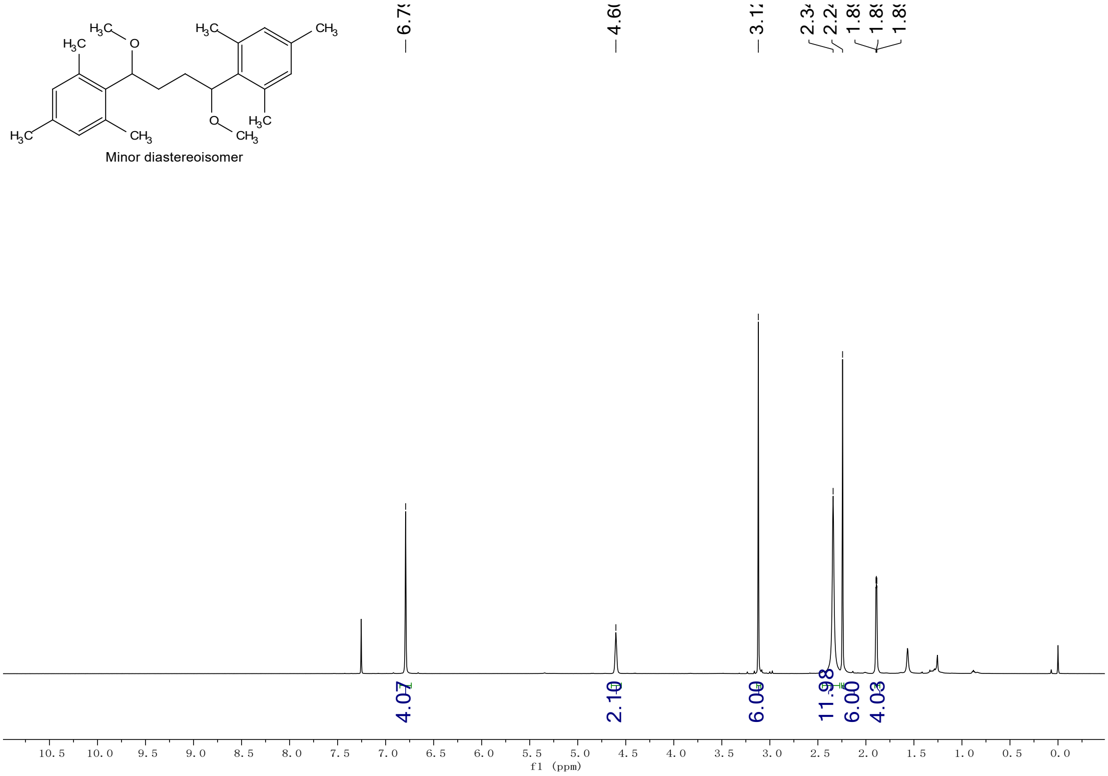
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 10 (major diastereoisomer)



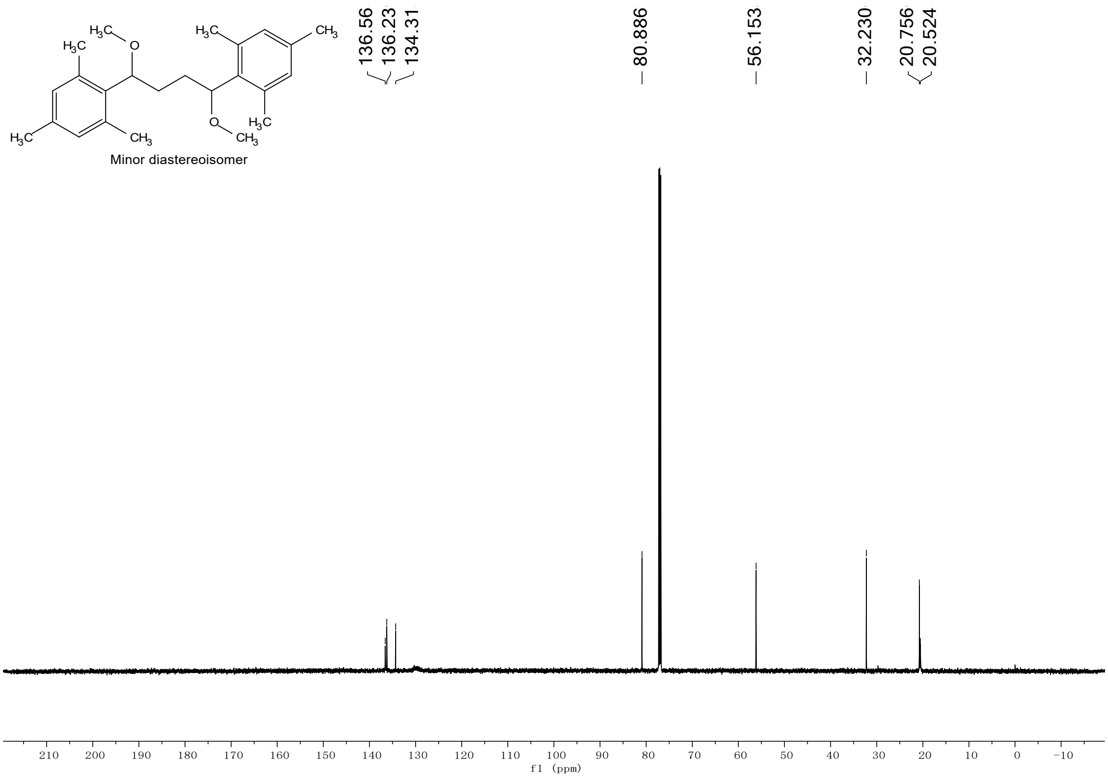
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 10 (major diastereoisomer)



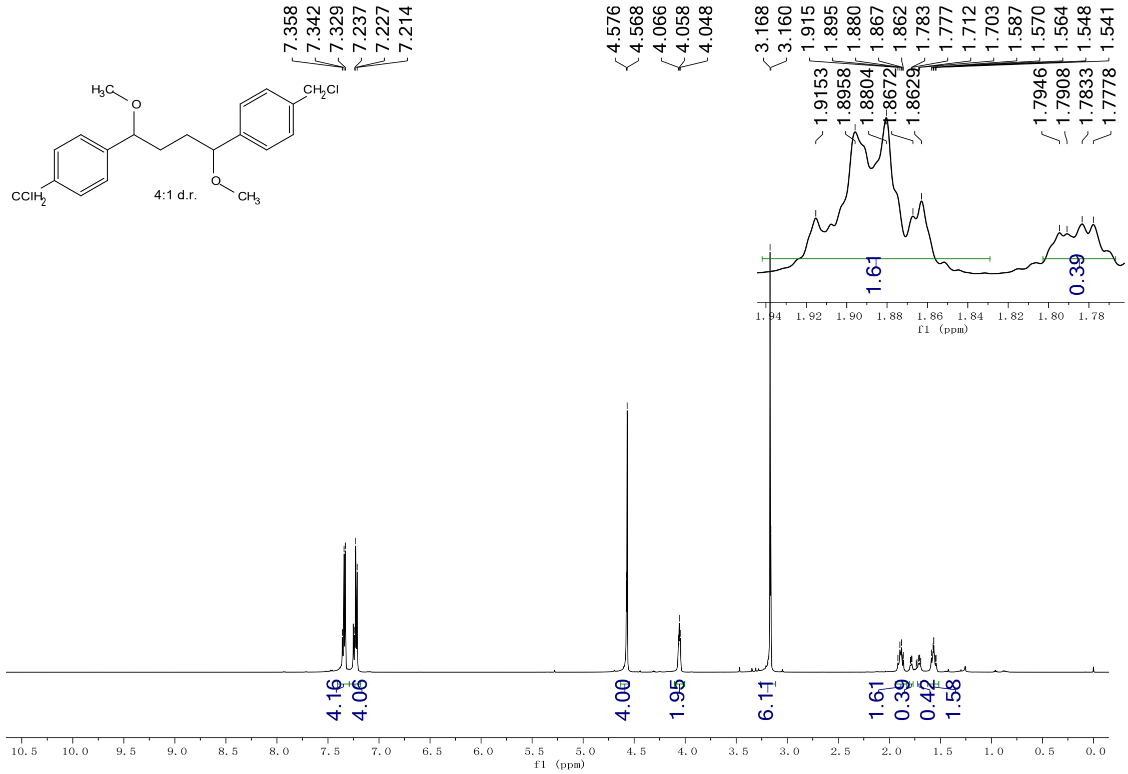
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 10 (minor diastereoisomer)



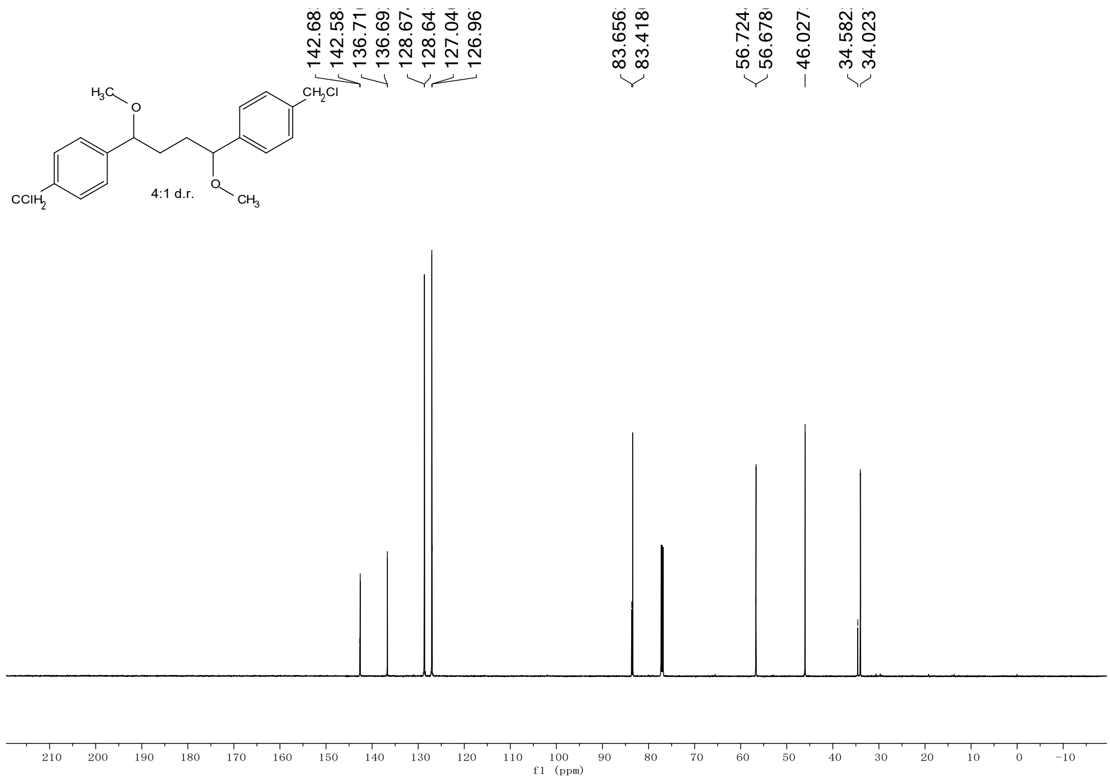
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 10 (minor diastereoisomer)



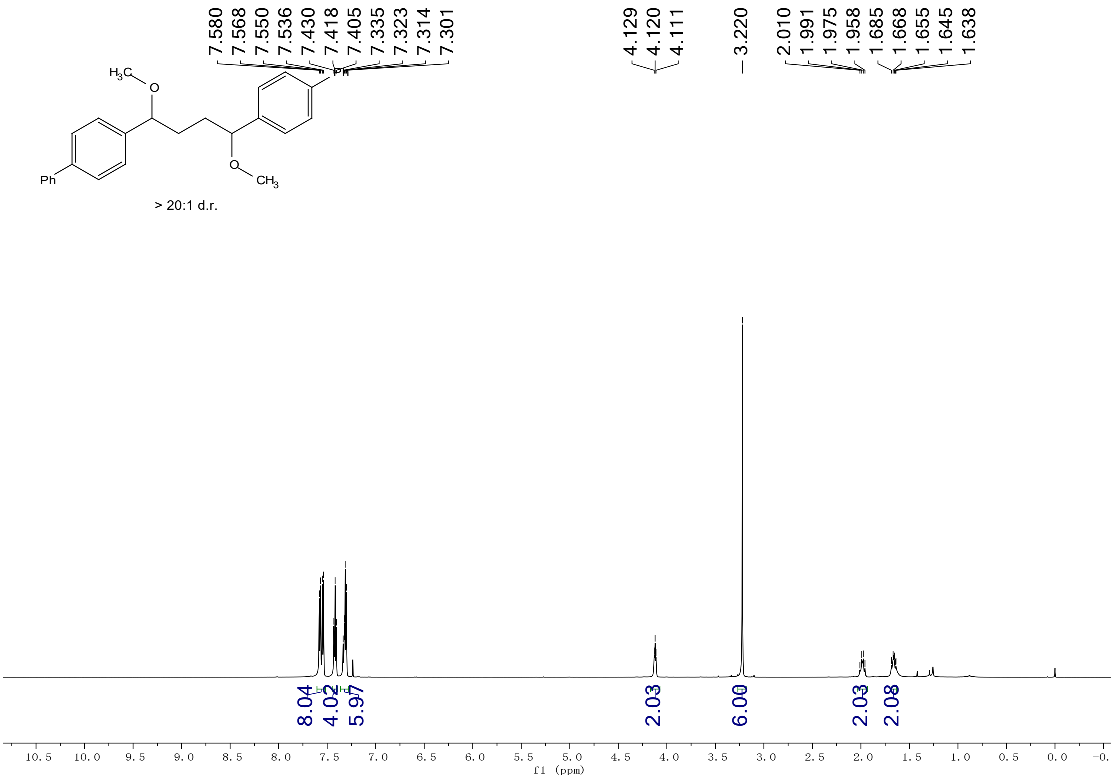
^{1}H-NMR Spectrum (600 MHz, CDCl$_3$) of 11



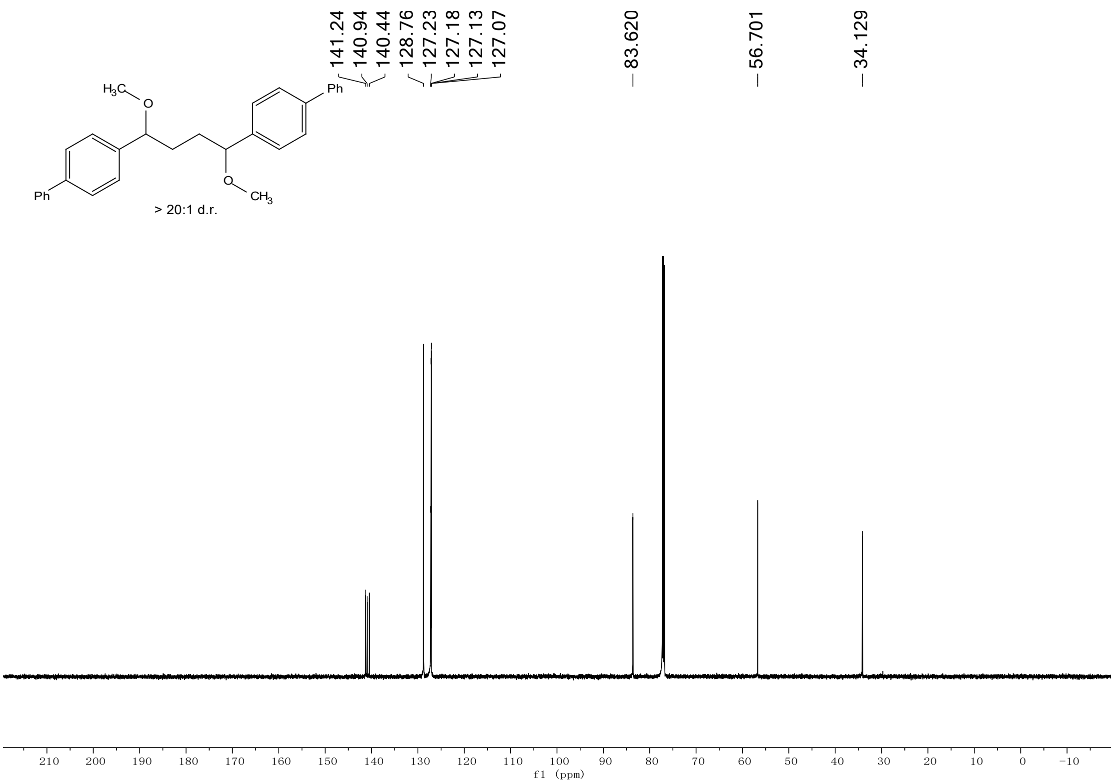
^{13}C-NMR Spectrum (151 MHz, CDCl$_3$) of 11



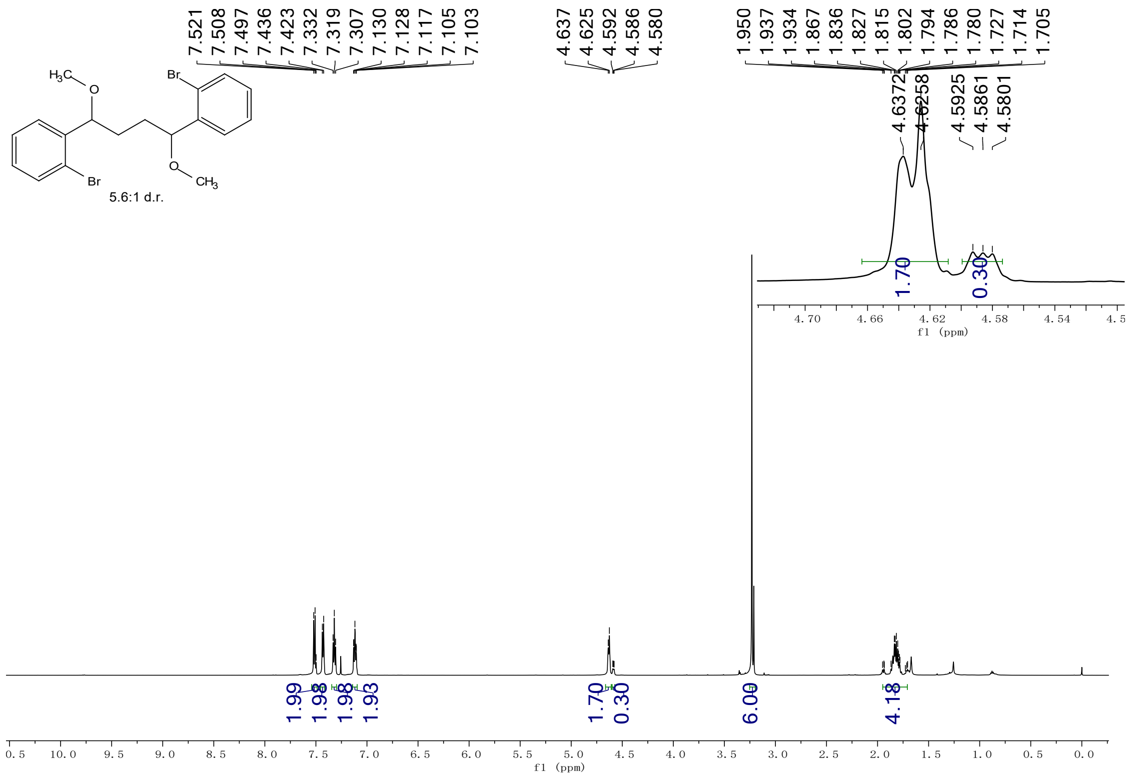
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 12



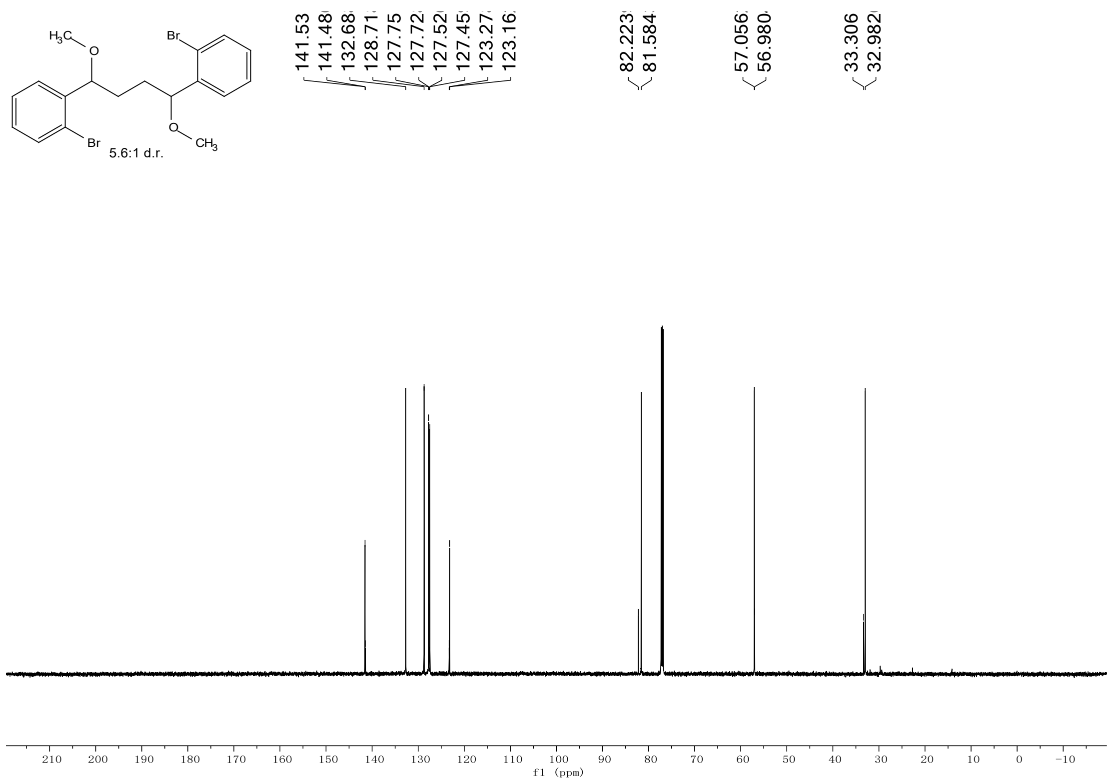
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 12



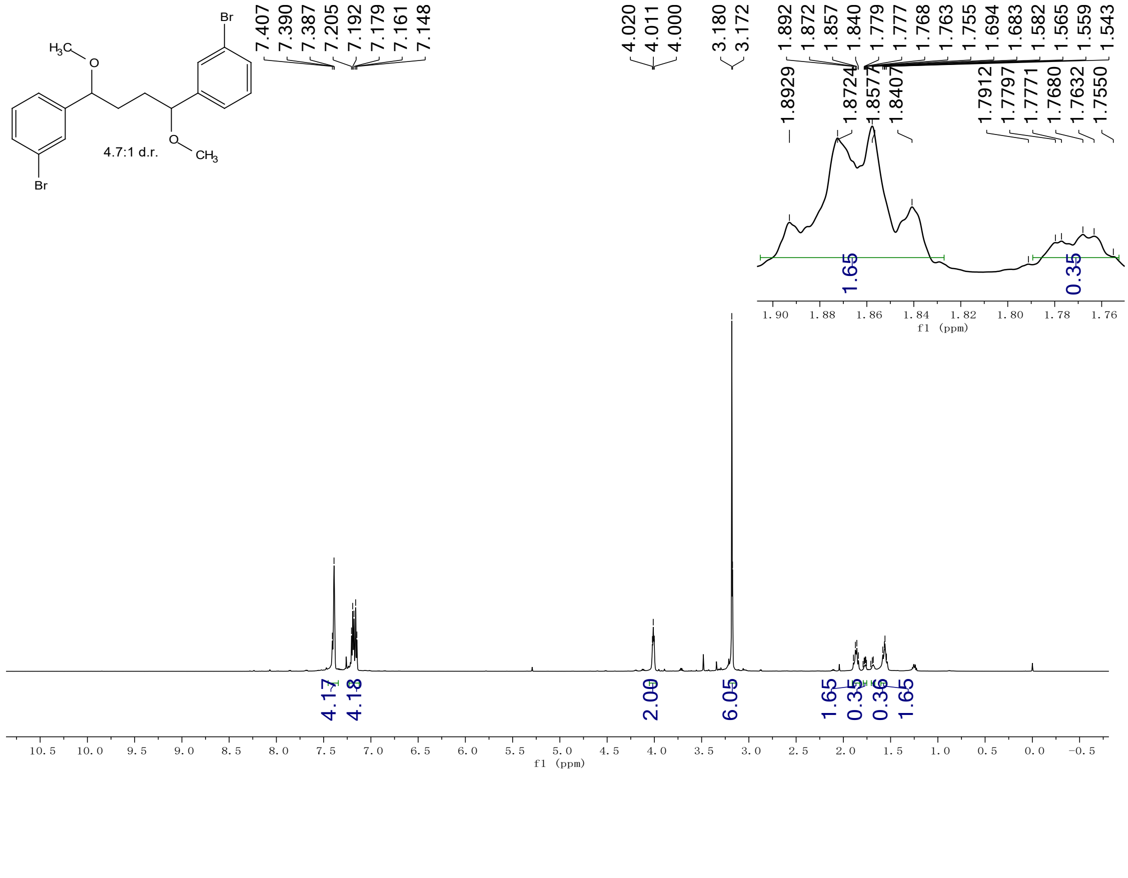
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 13



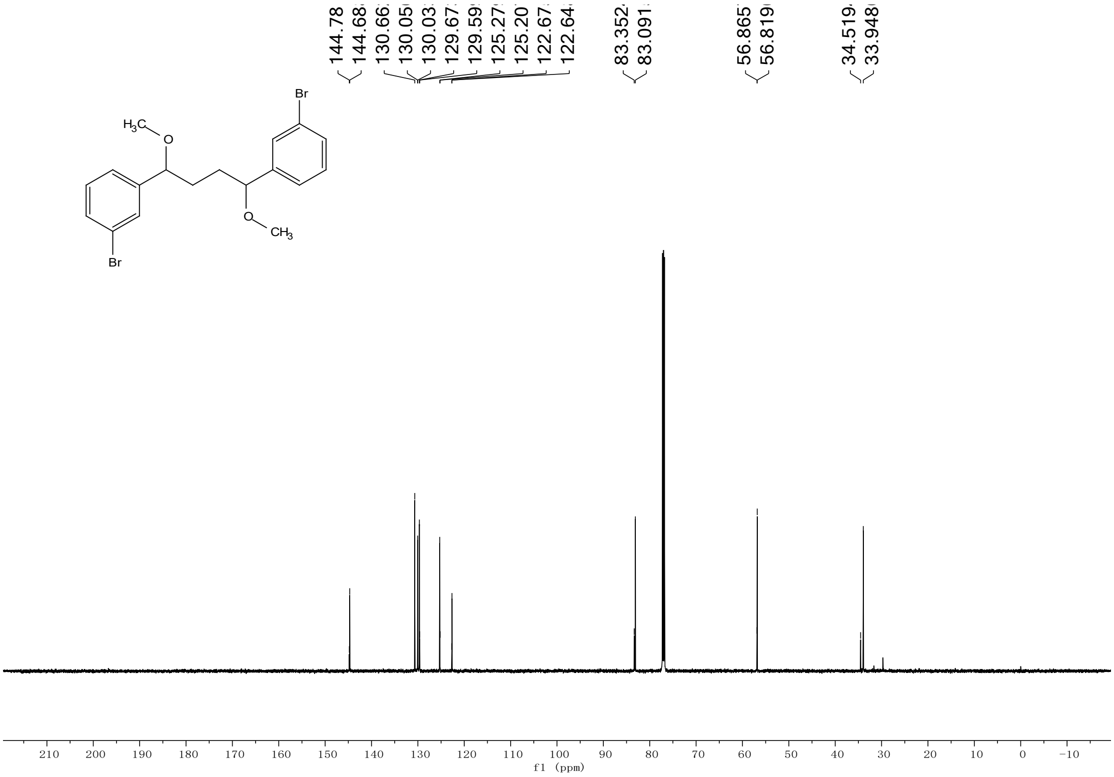
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 13



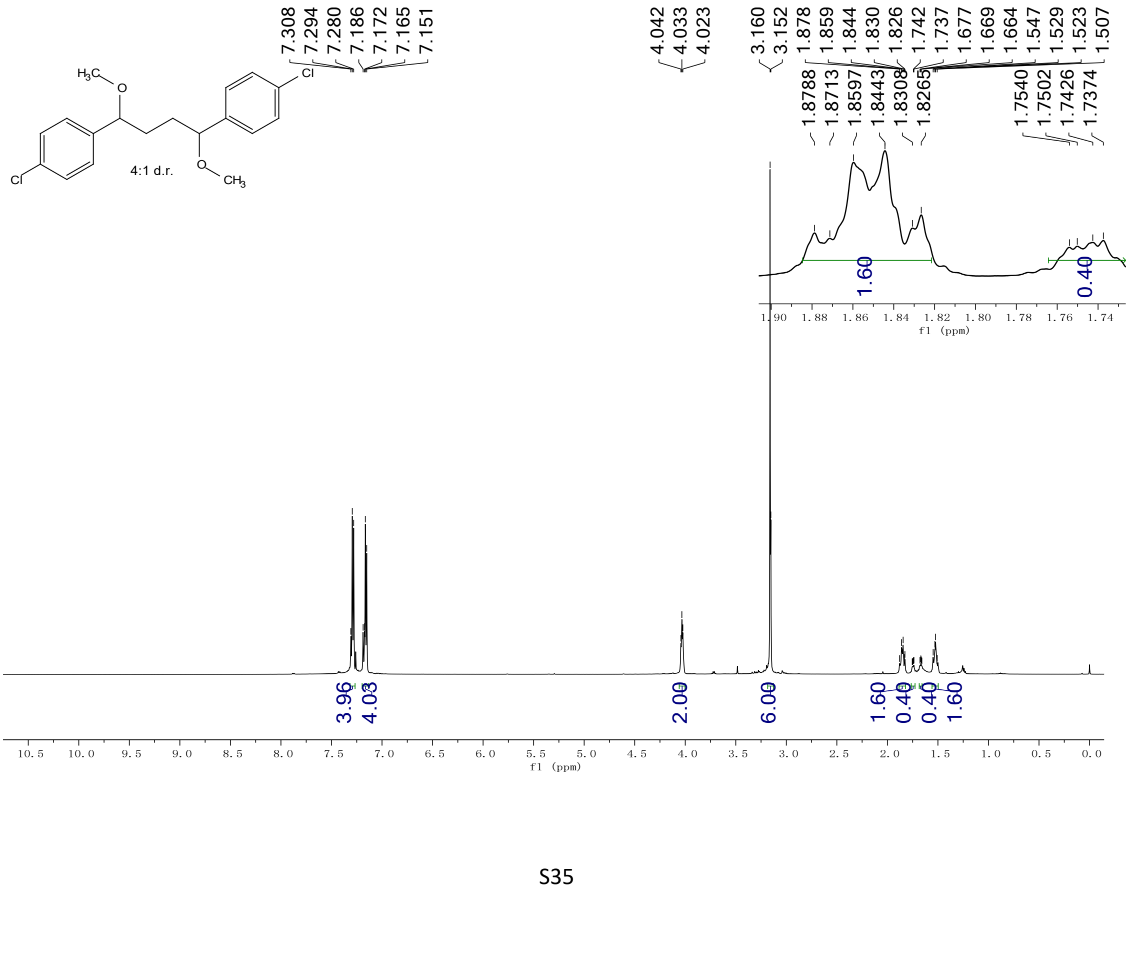
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 14



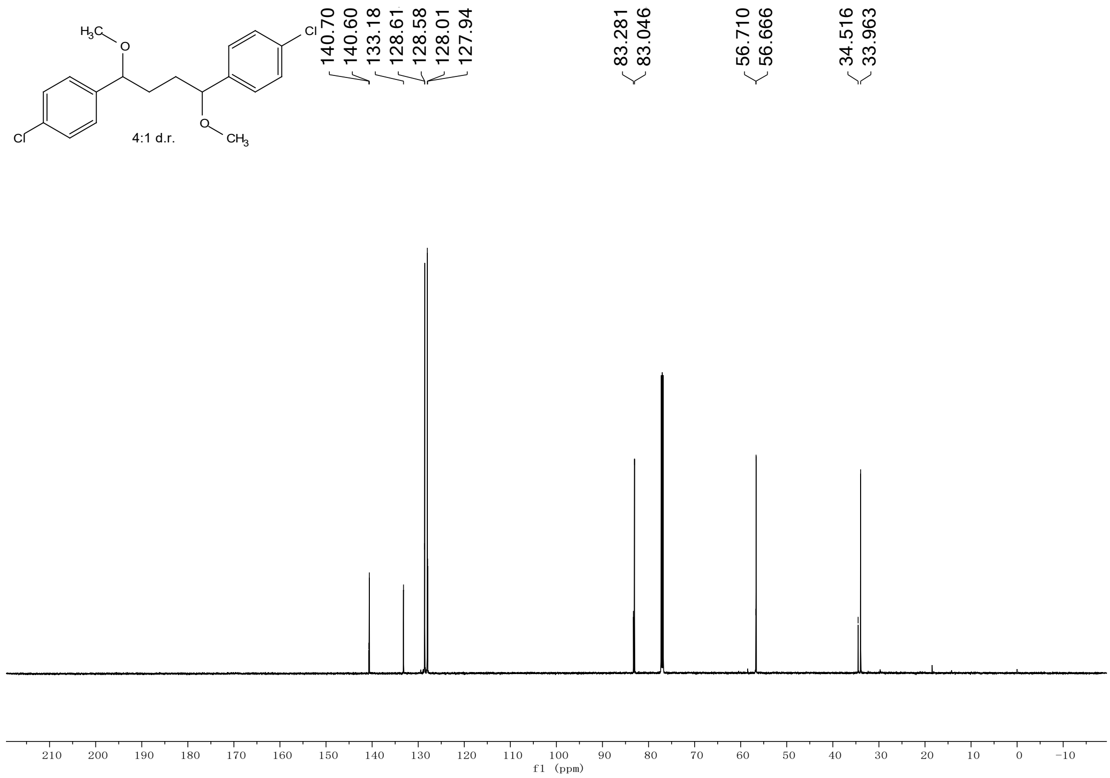
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 14



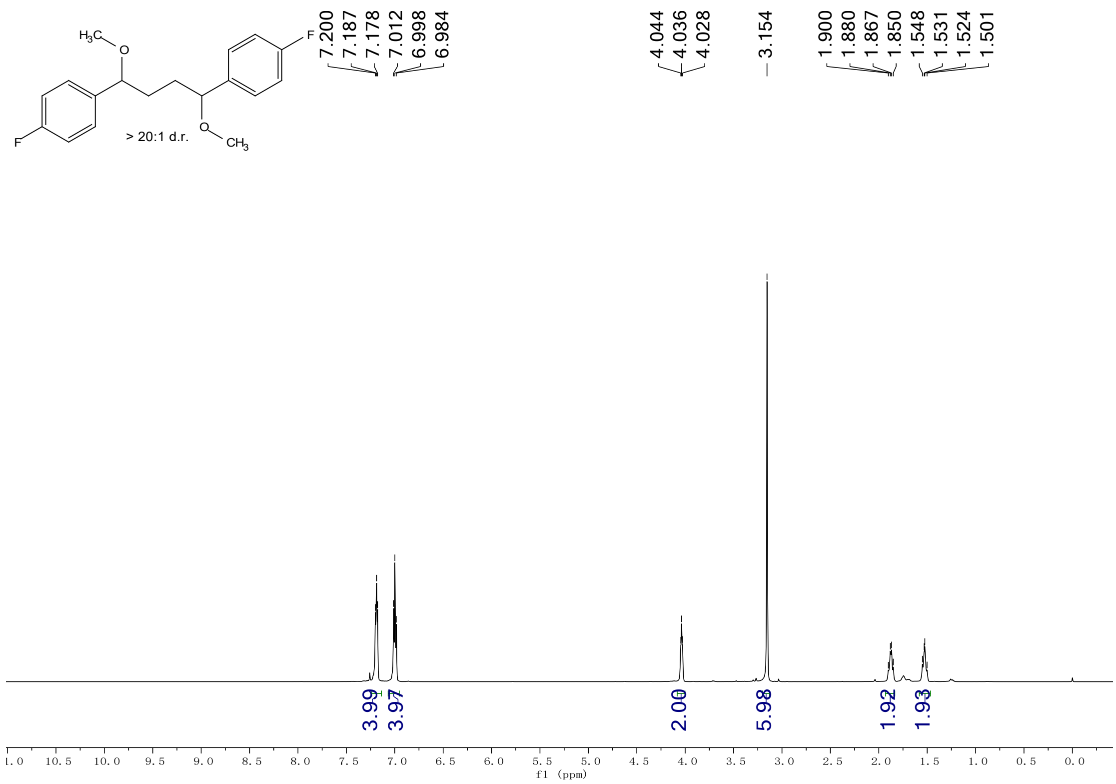
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 15



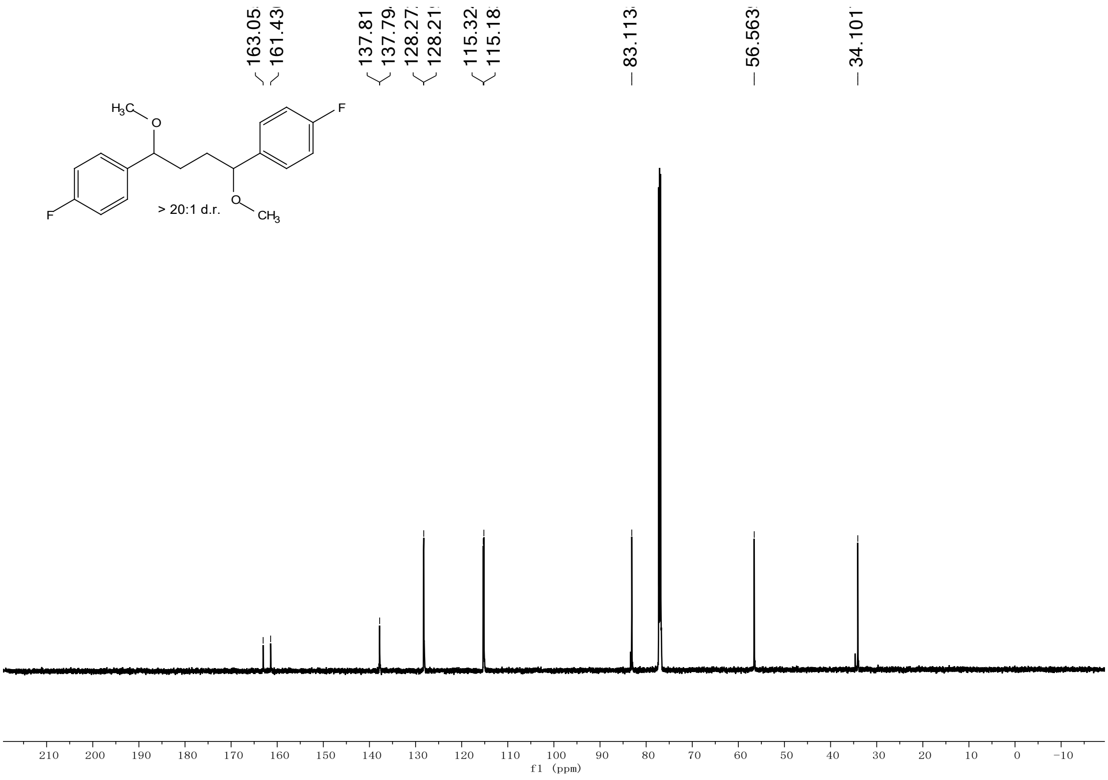
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 15



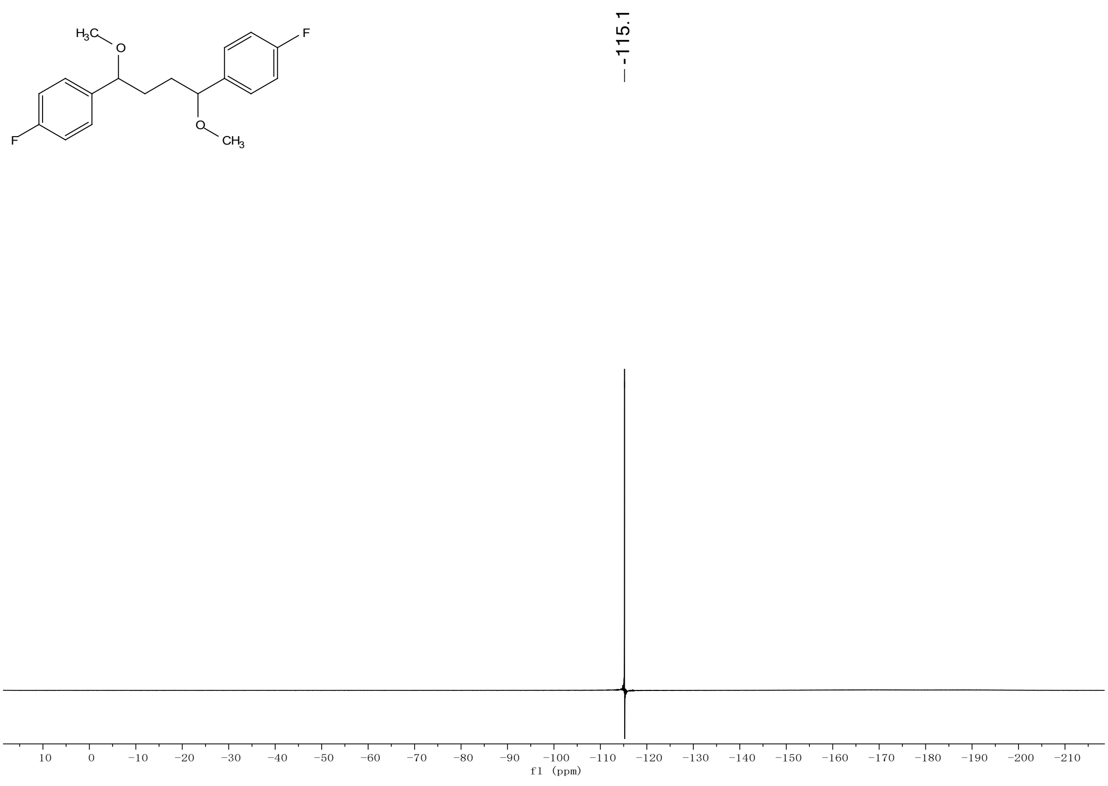
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 16



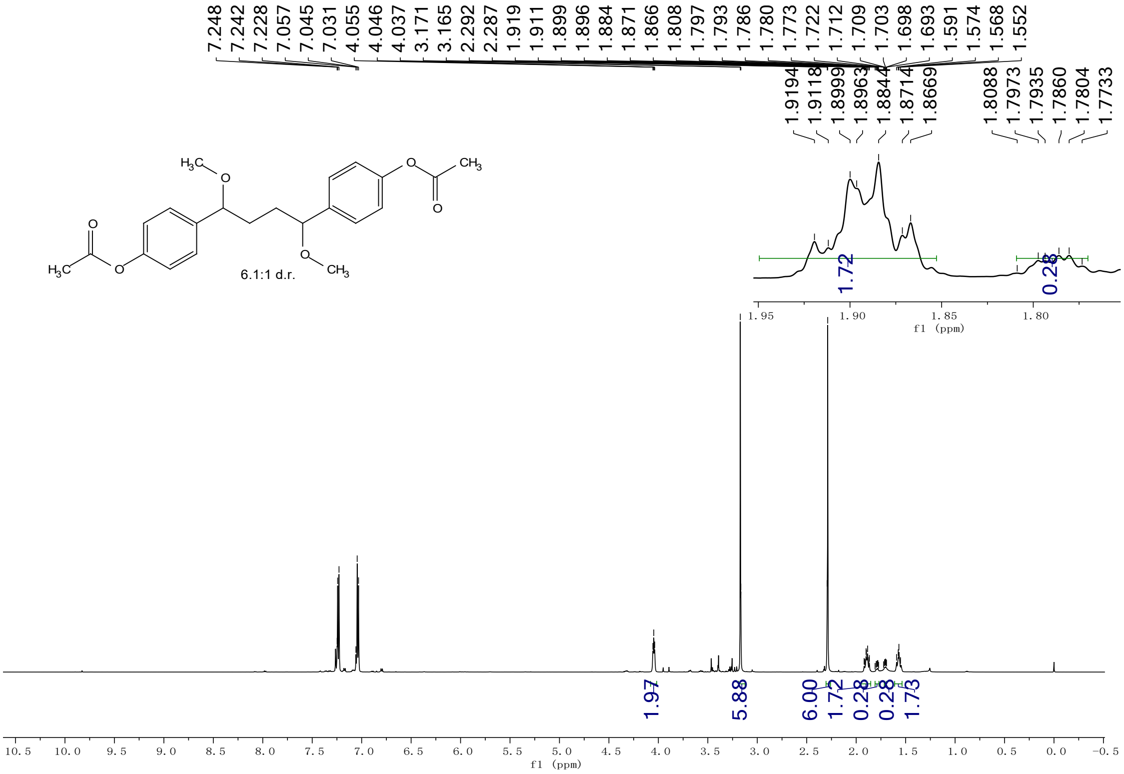
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 16



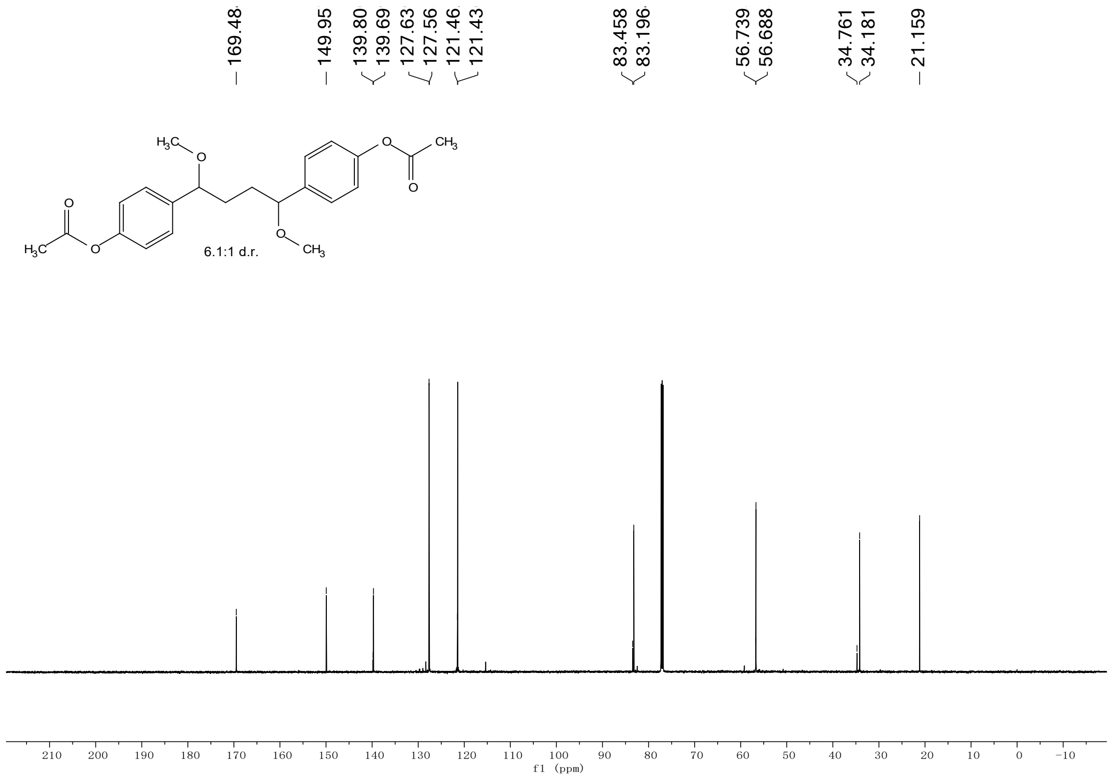
^{19}F-NMR Spectrum (151 MHz, $CDCl_3$) of 16 (major diastereoisomer)



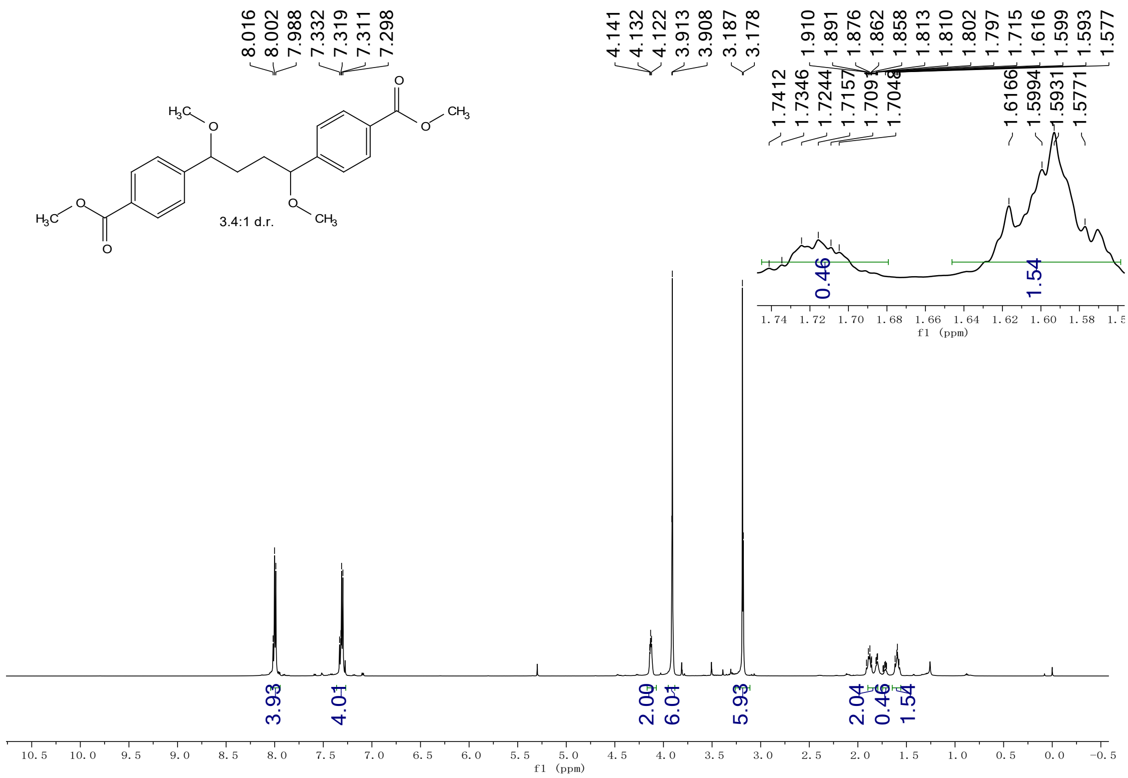
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 17



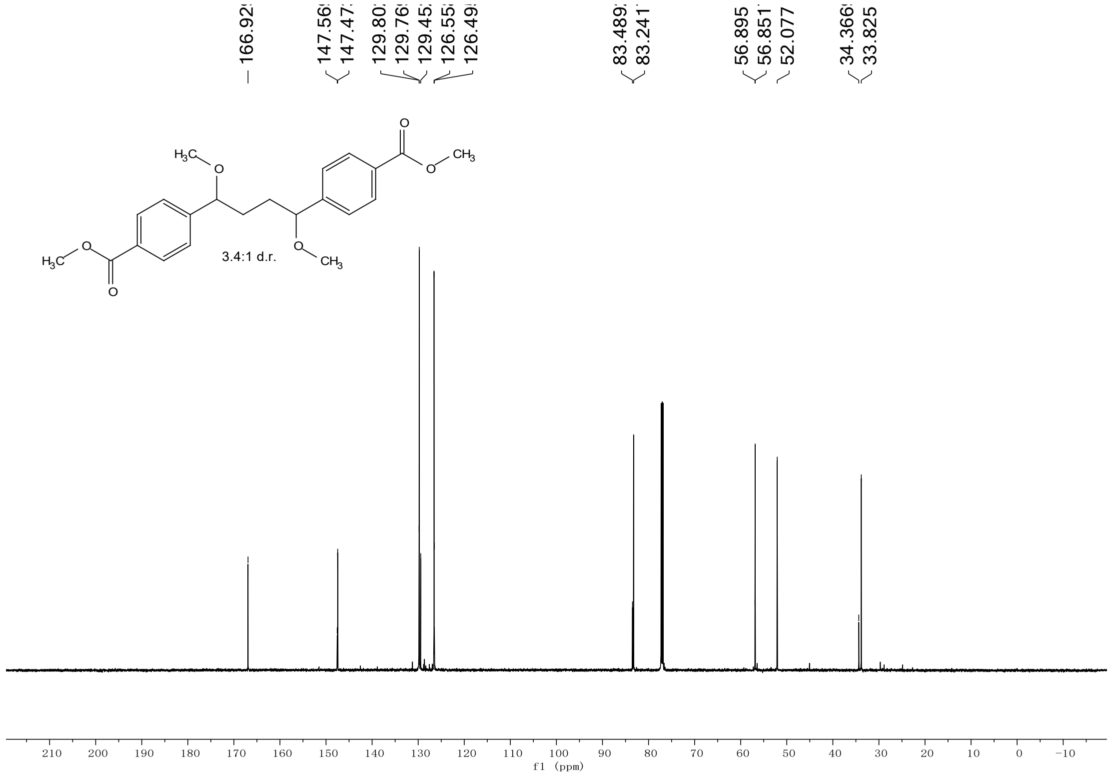
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 17



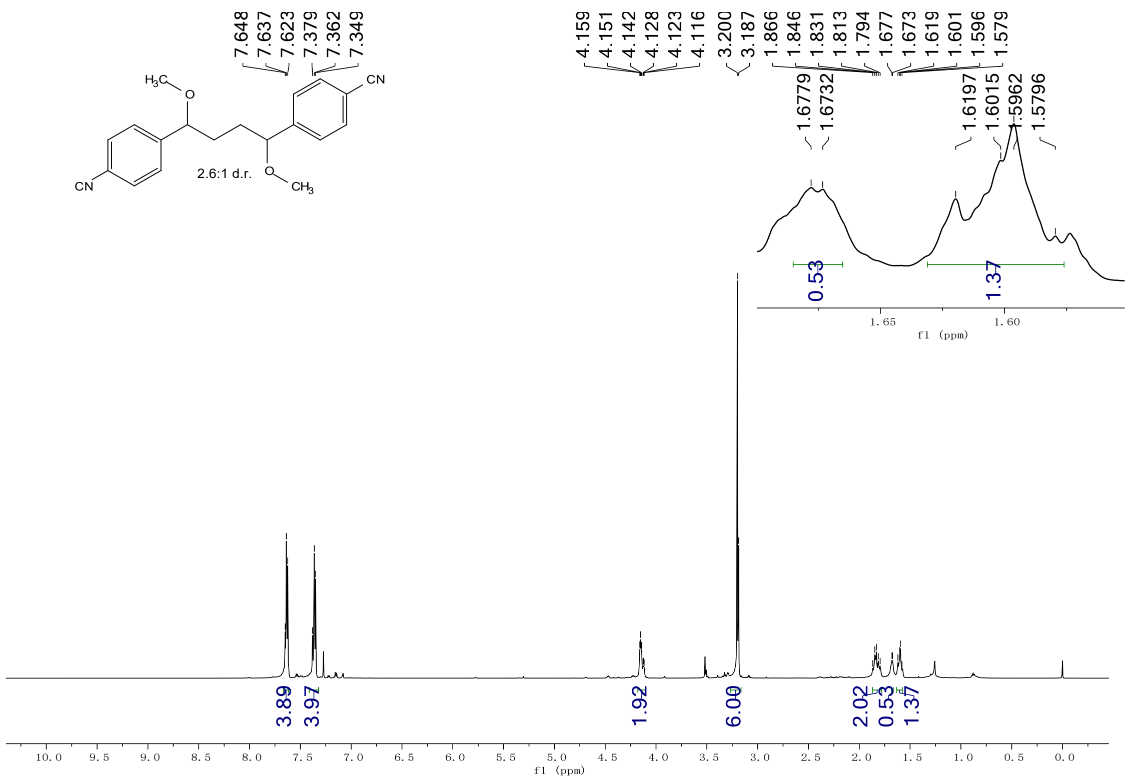
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 18



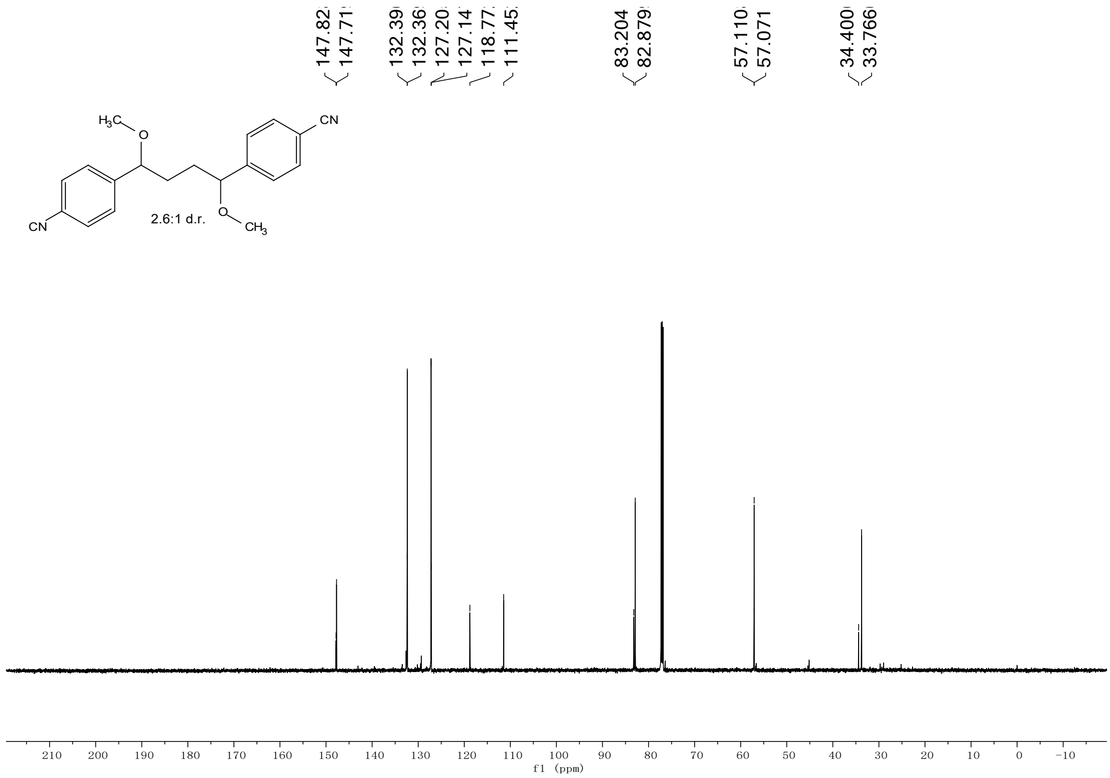
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 18



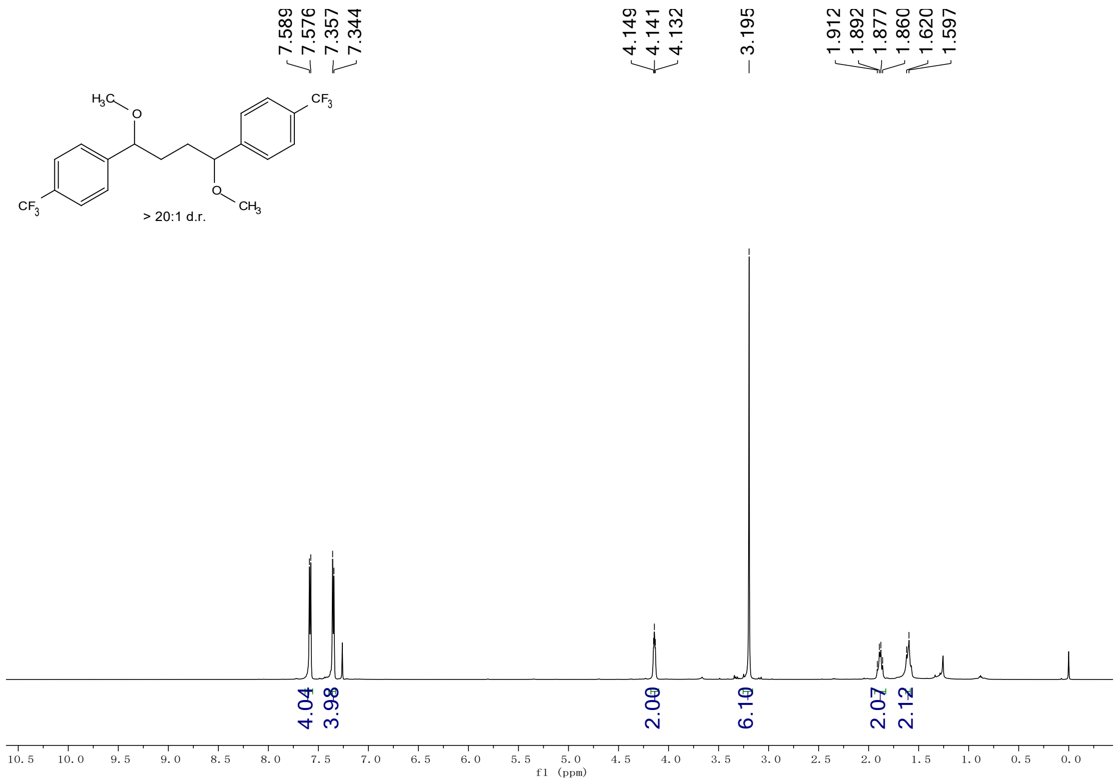
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 19



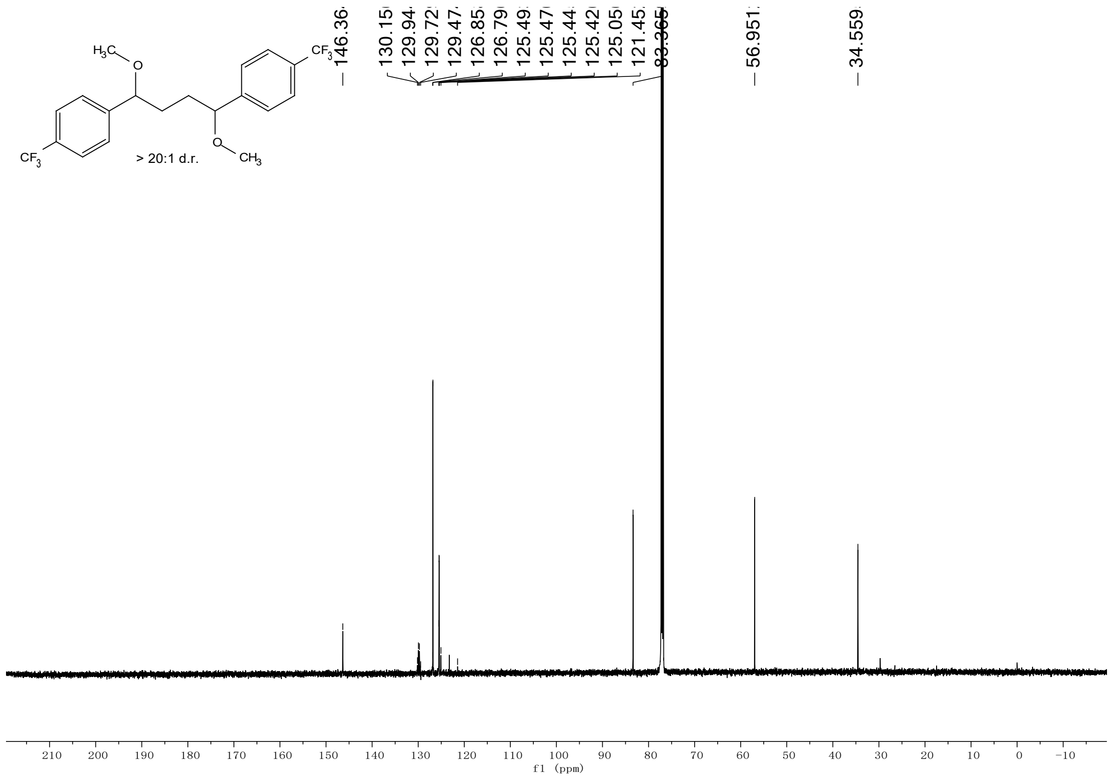
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 19



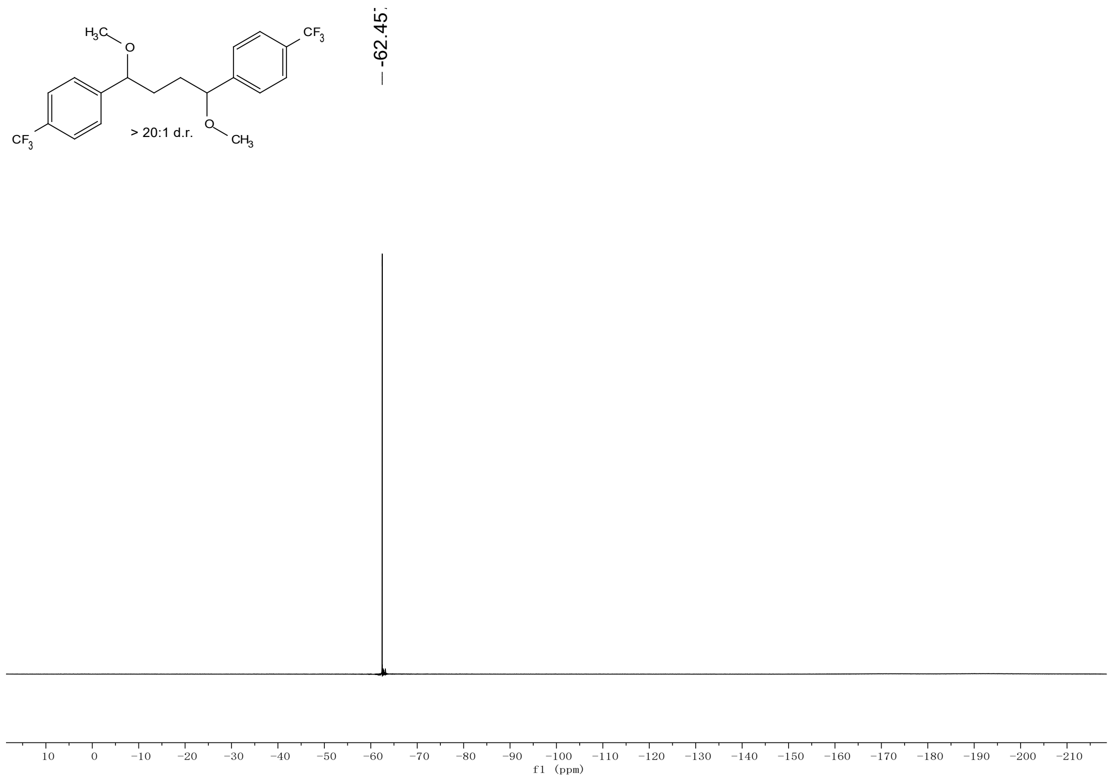
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 20



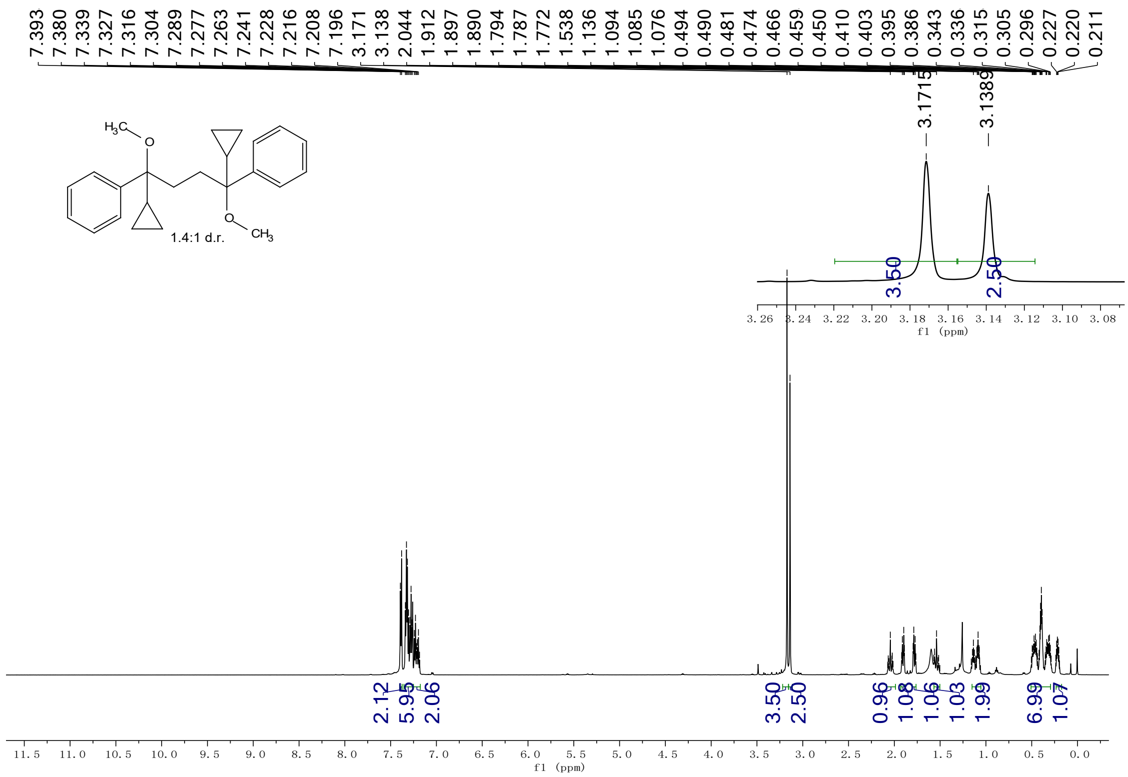
^{13}C-NMR Spectrum (151 MHz, CDCl$_3$) of 20



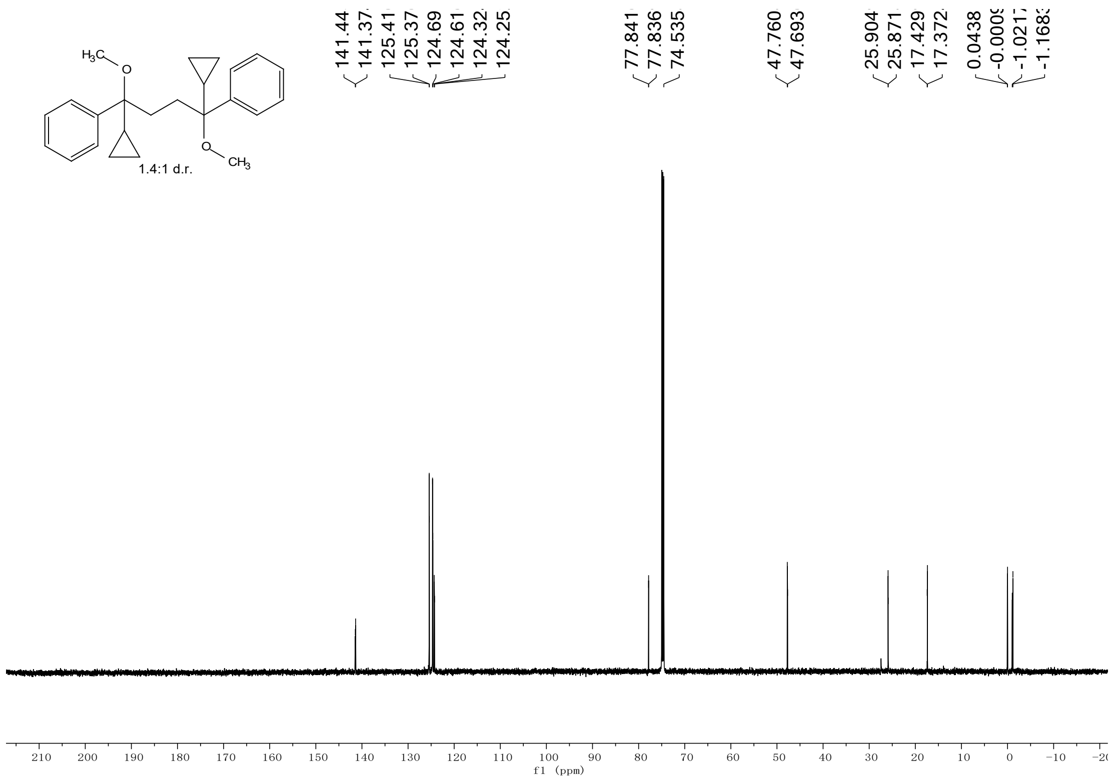
^{19}F-NMR Spectrum (151 MHz, CDCl$_3$) of 20



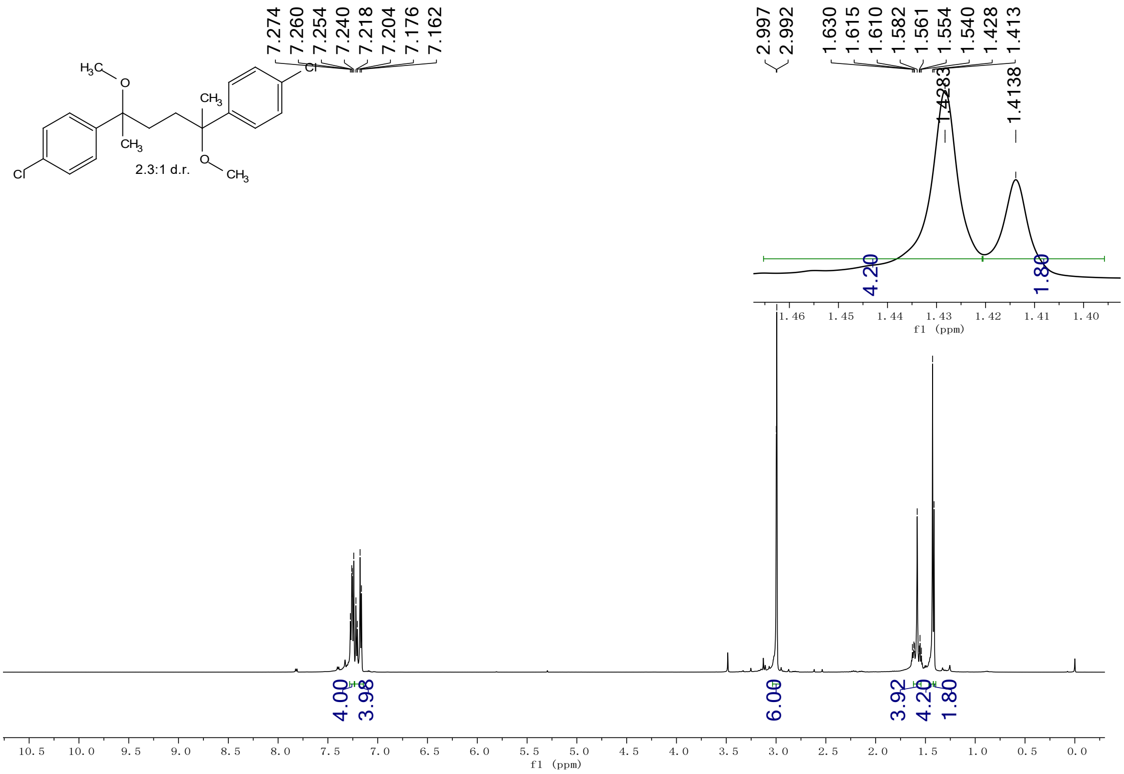
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 21



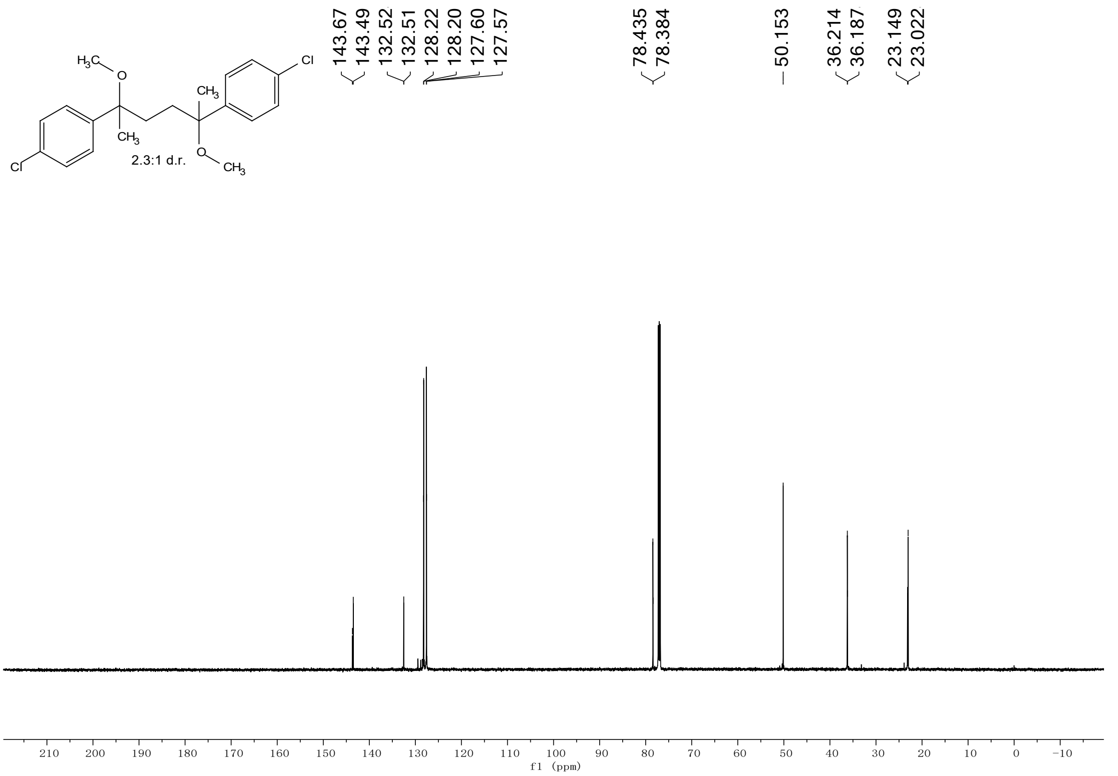
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 21



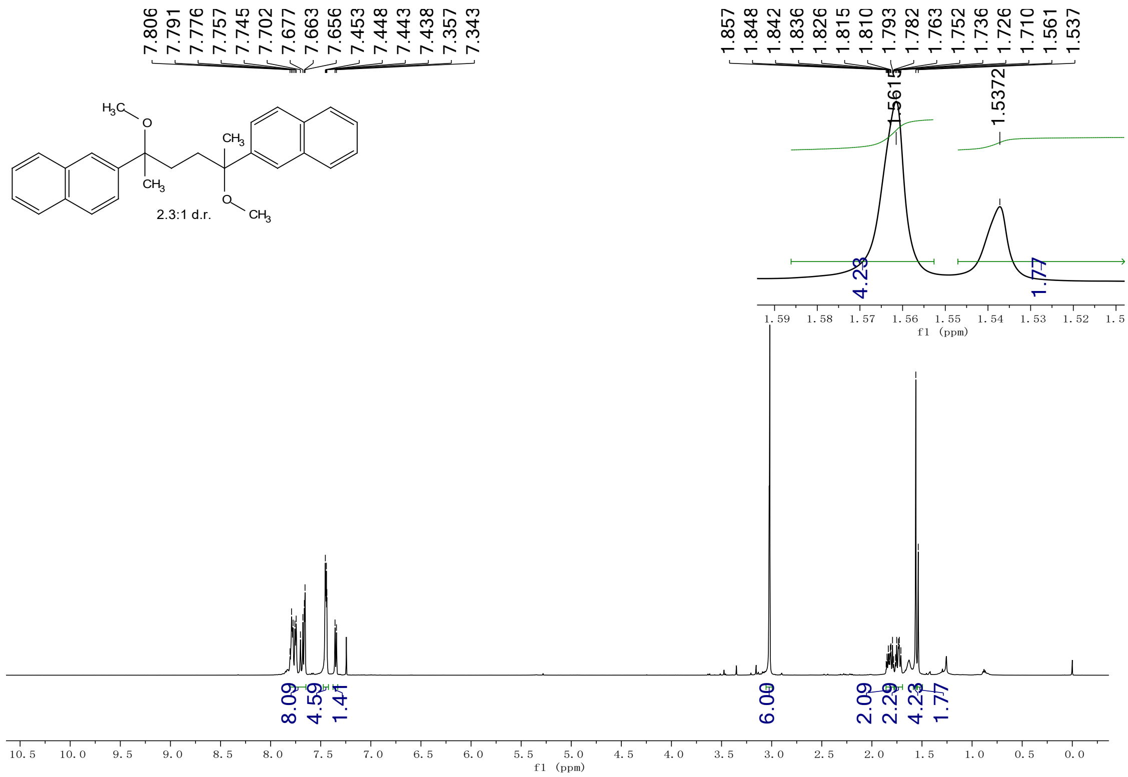
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 22



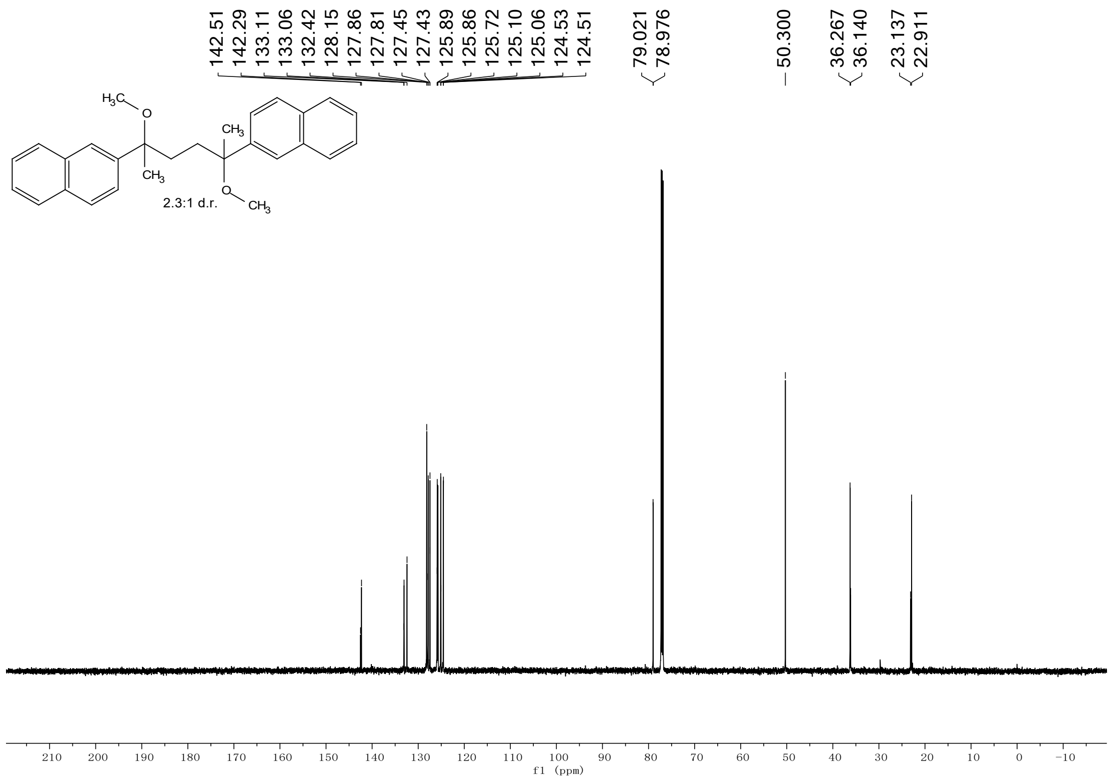
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 22



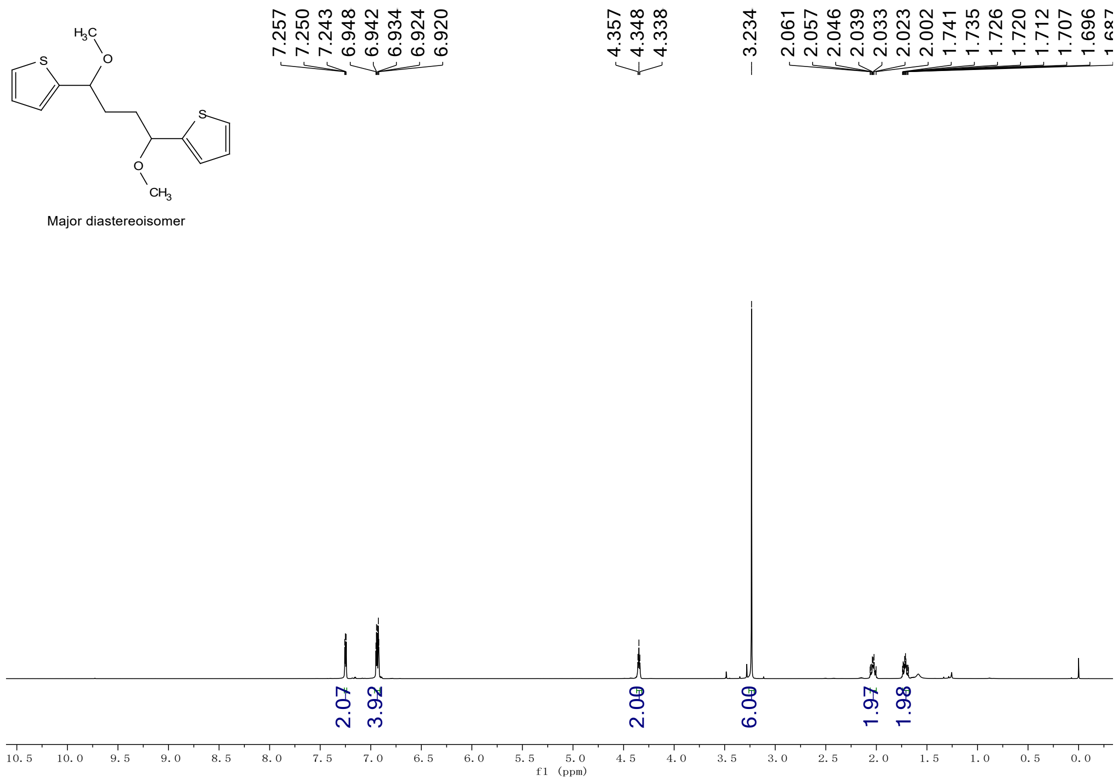
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 23



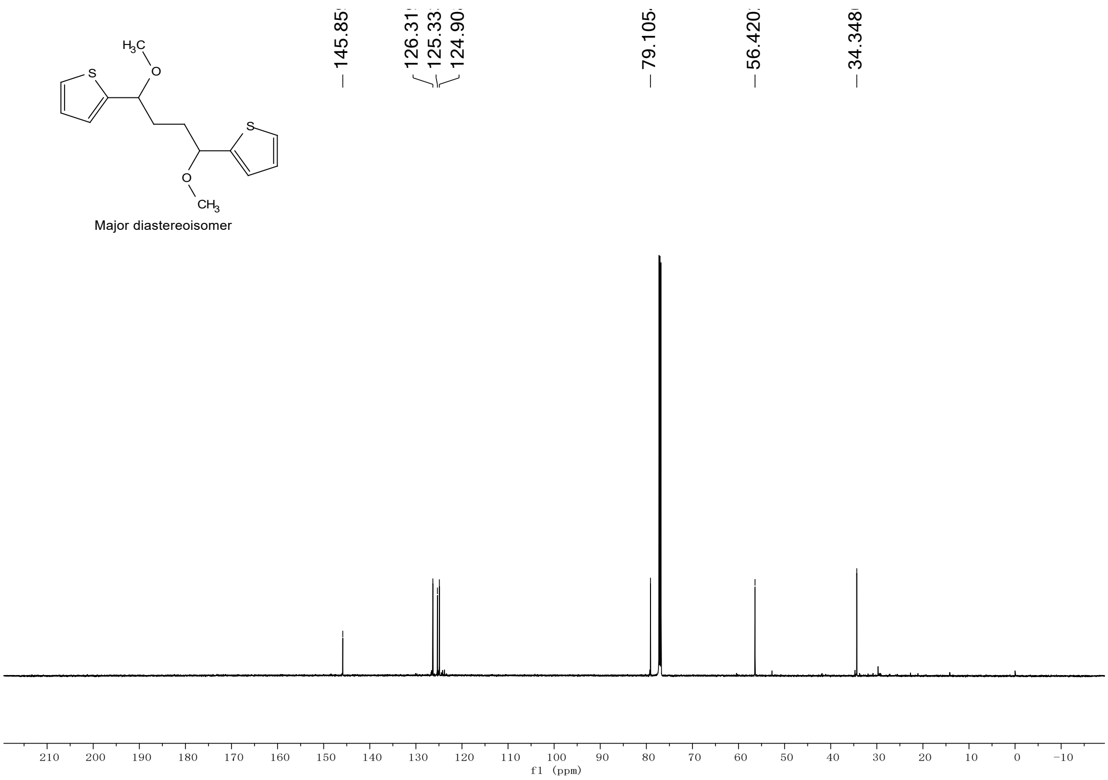
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 23



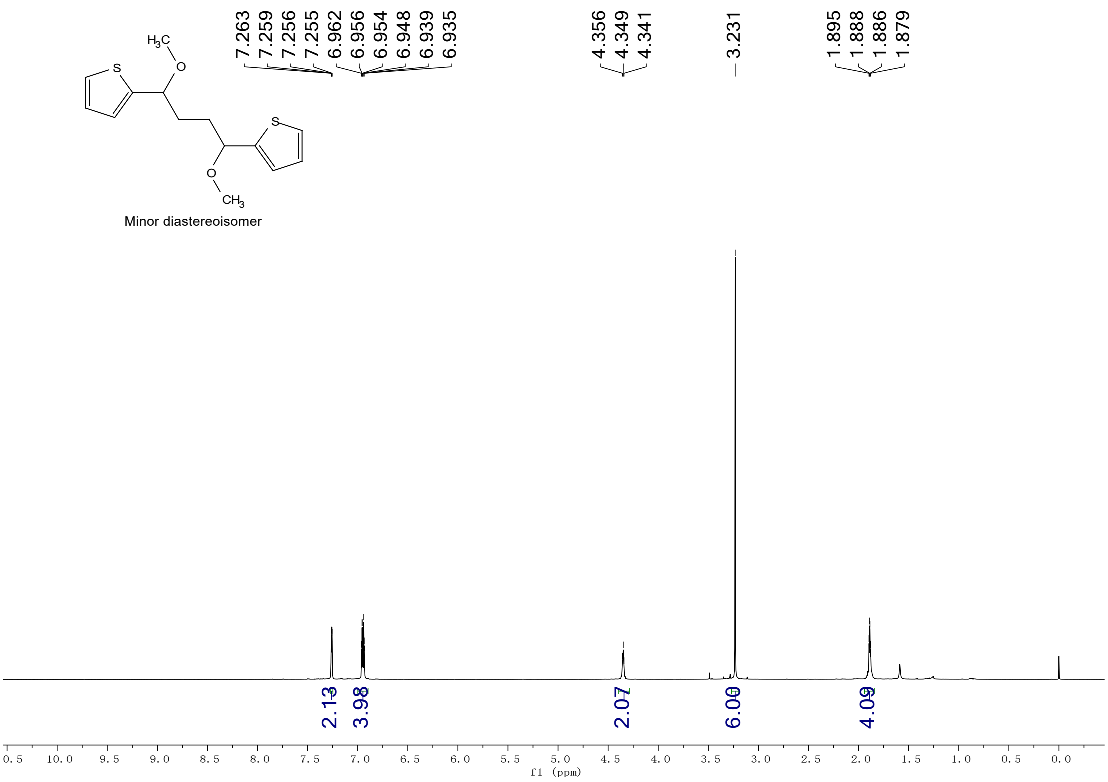
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 24 (major diastereoisomer)



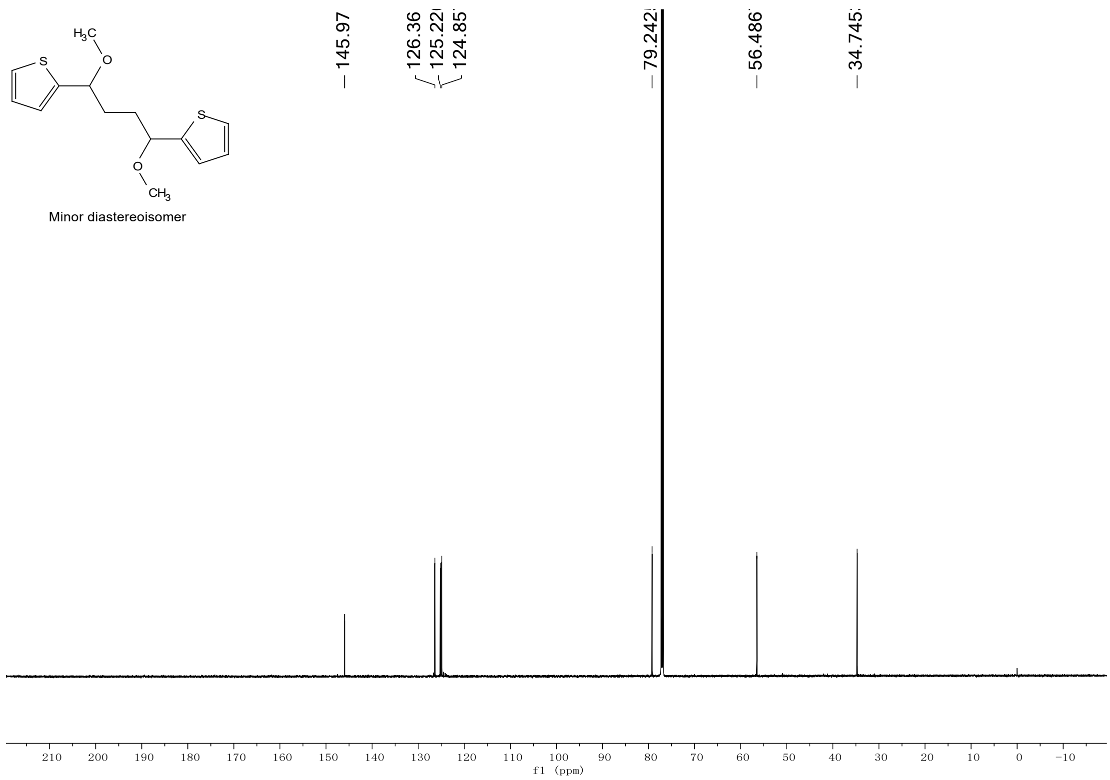
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 24 (major diastereoisomer)



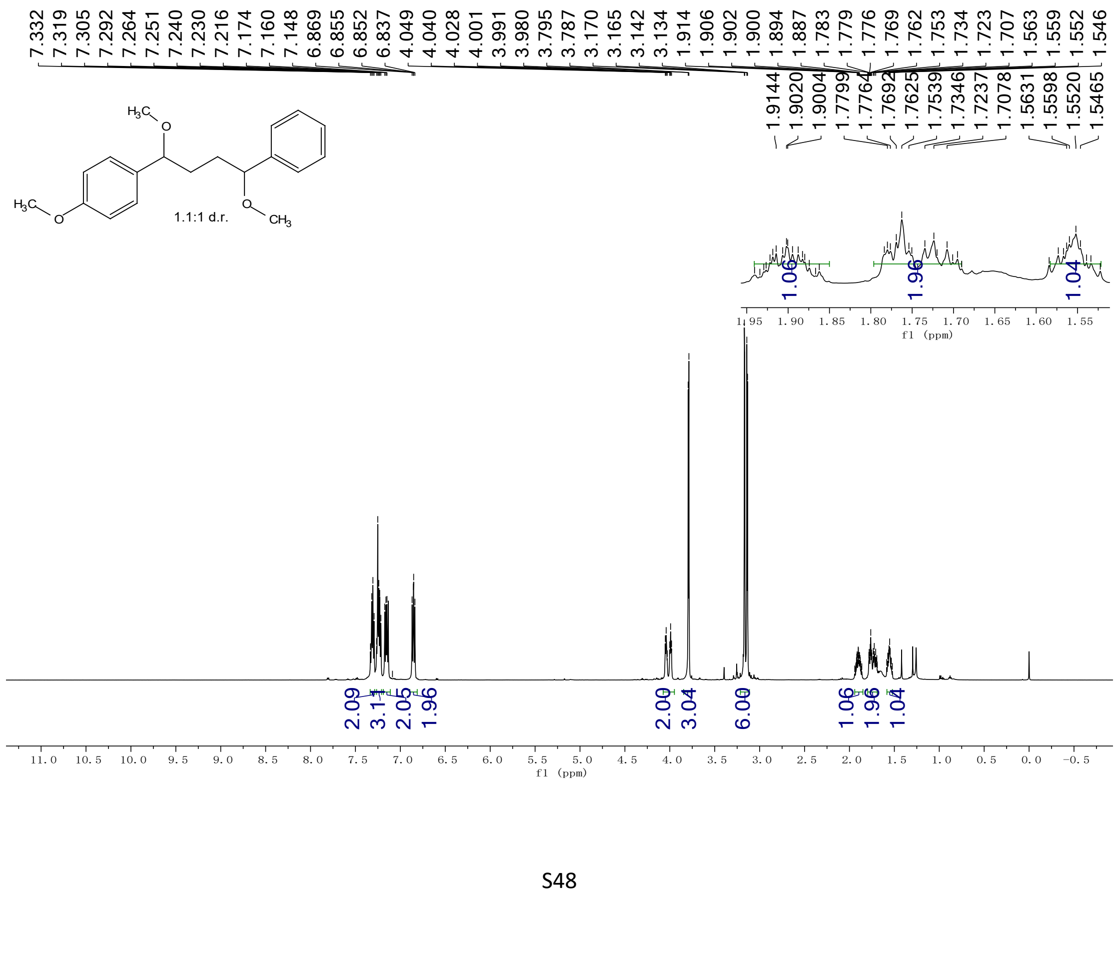
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 24 (minor diastereoisomer)



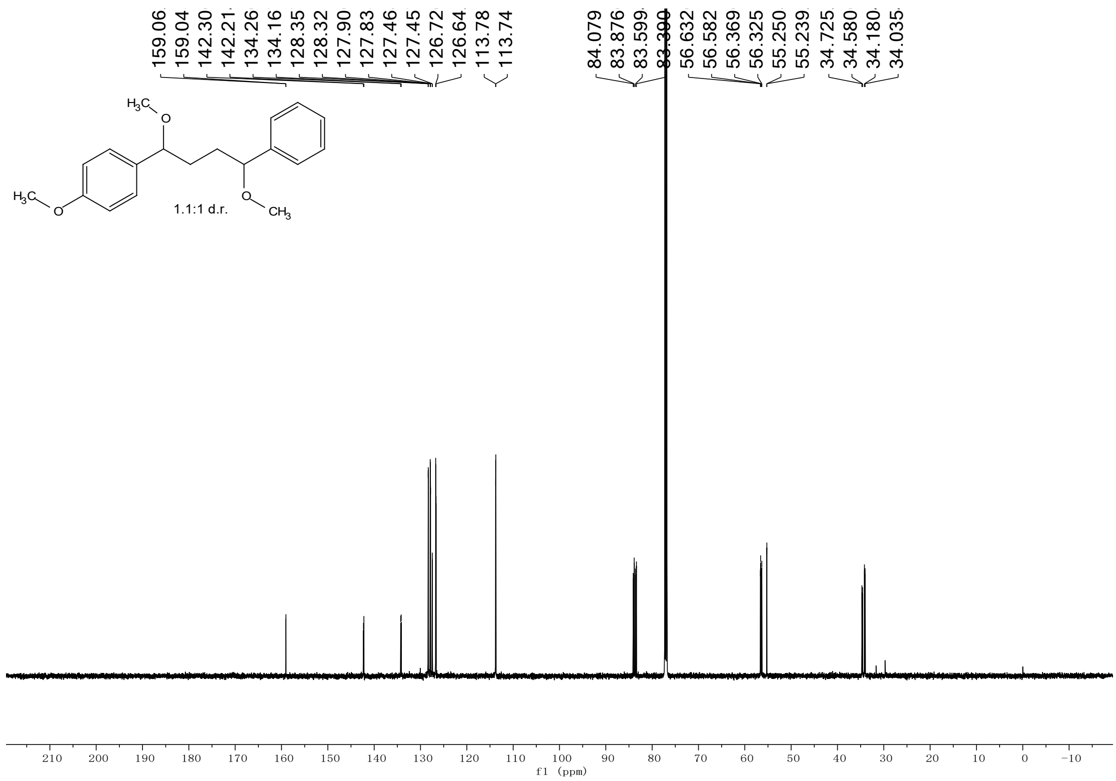
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 24 (minor diastereoisomer)



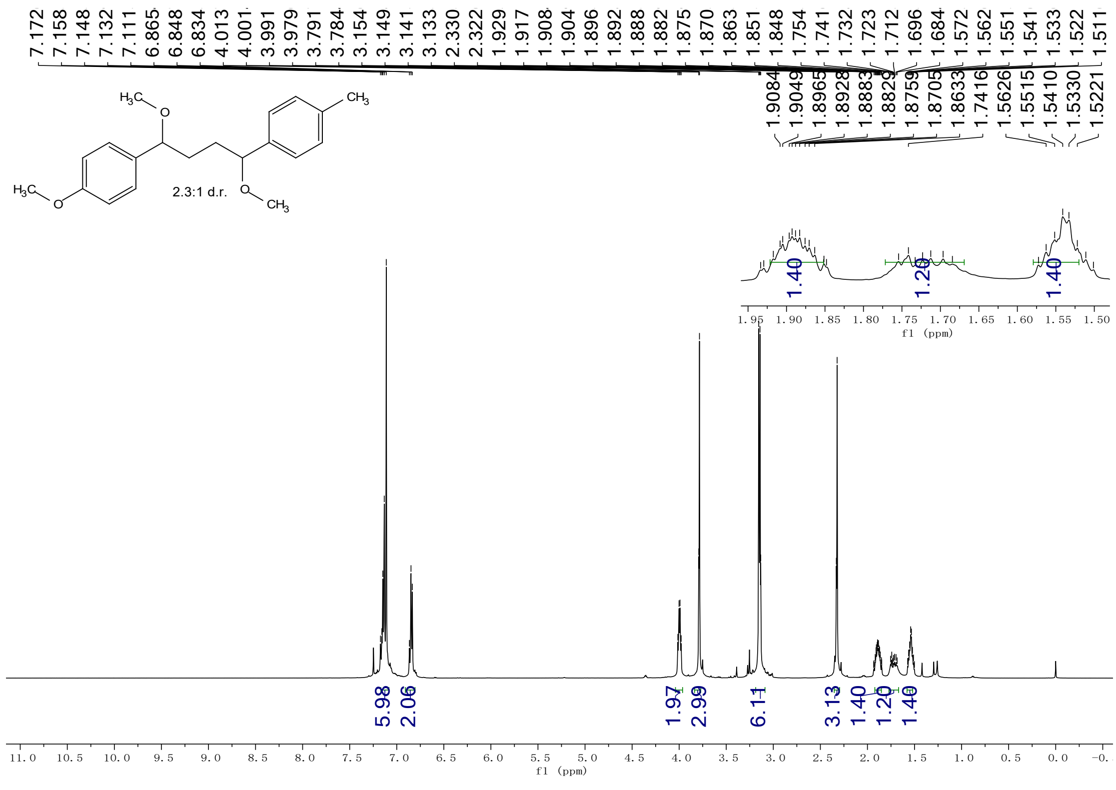
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 25



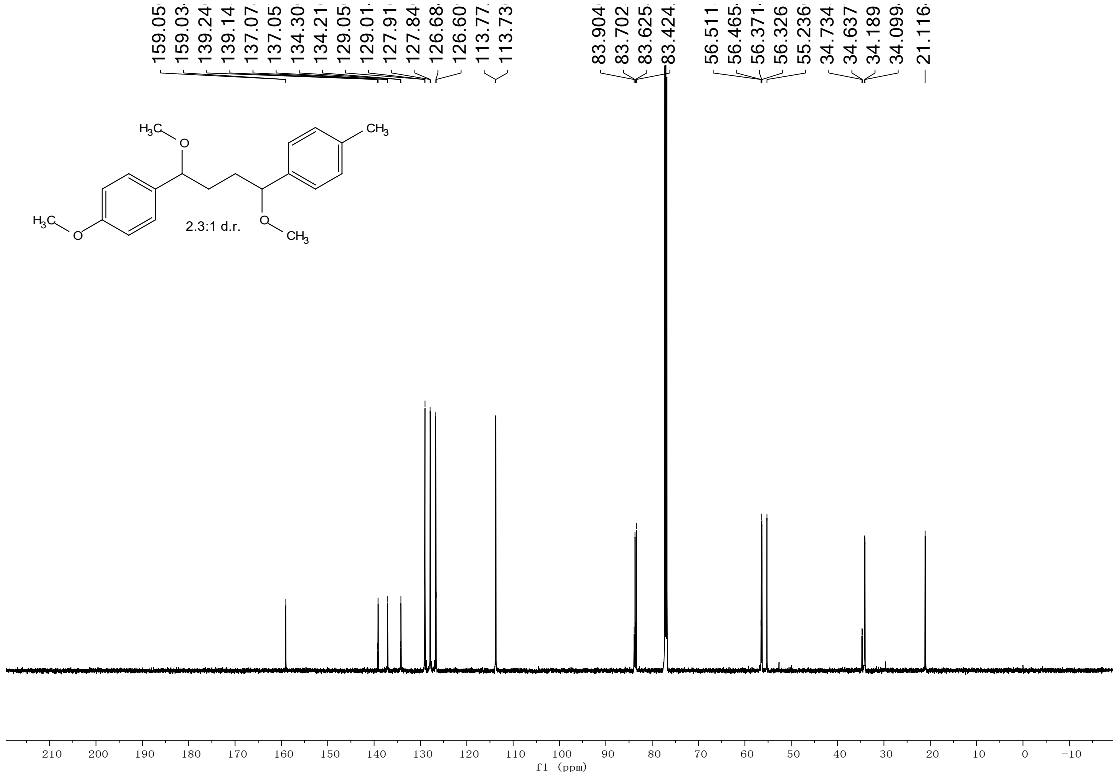
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 25



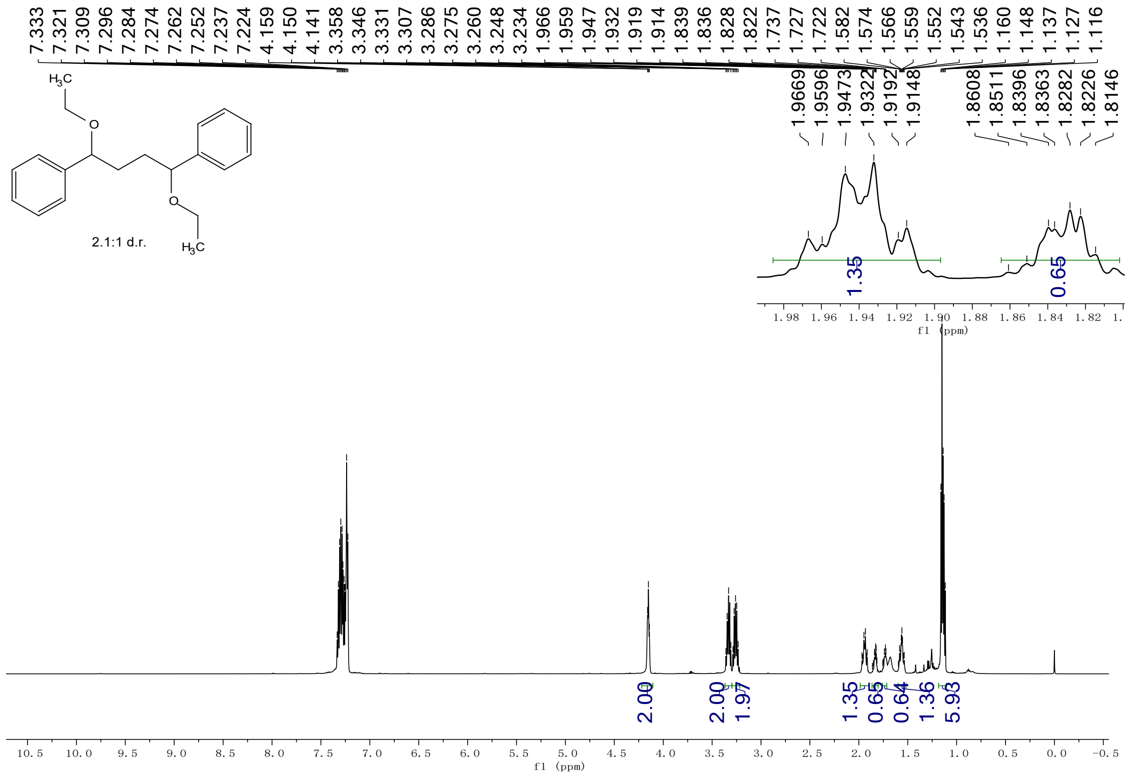
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 26



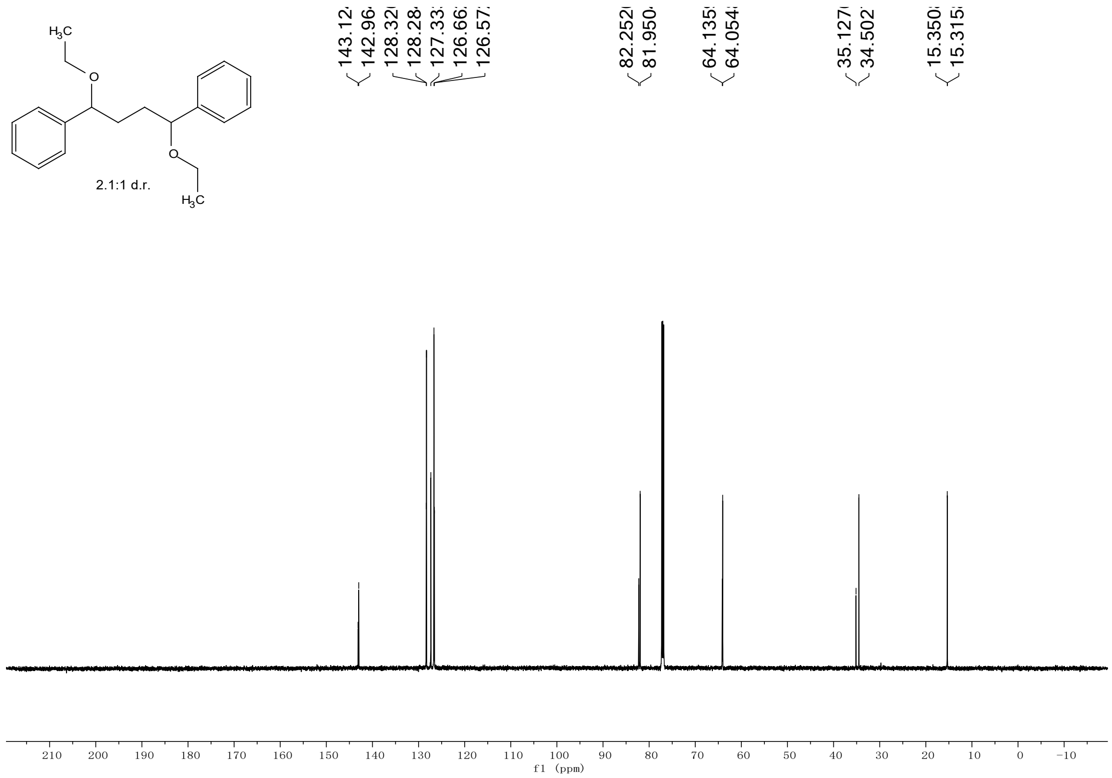
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 26



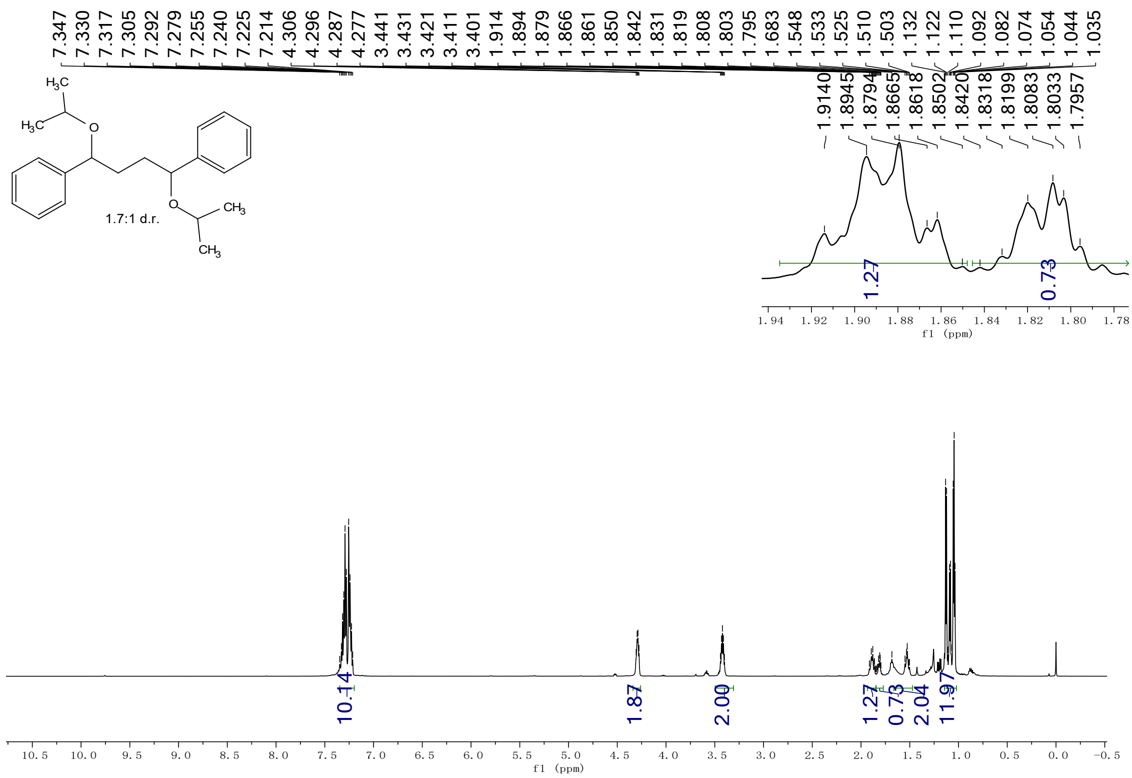
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 27



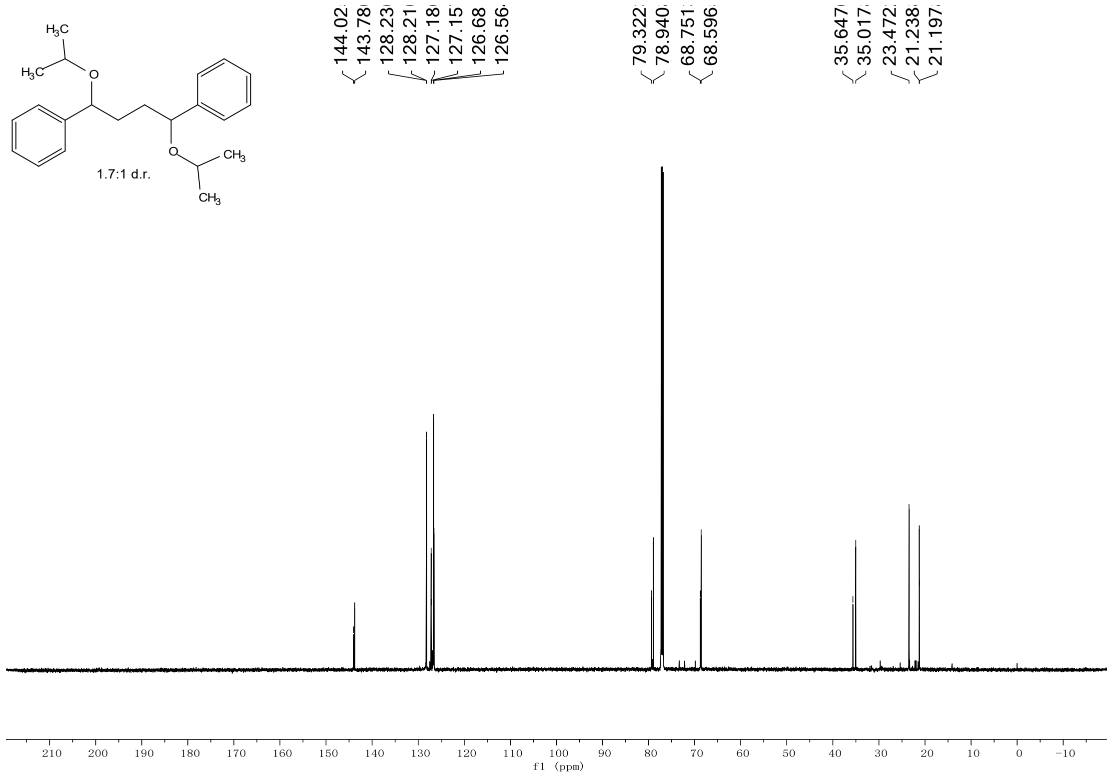
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 27



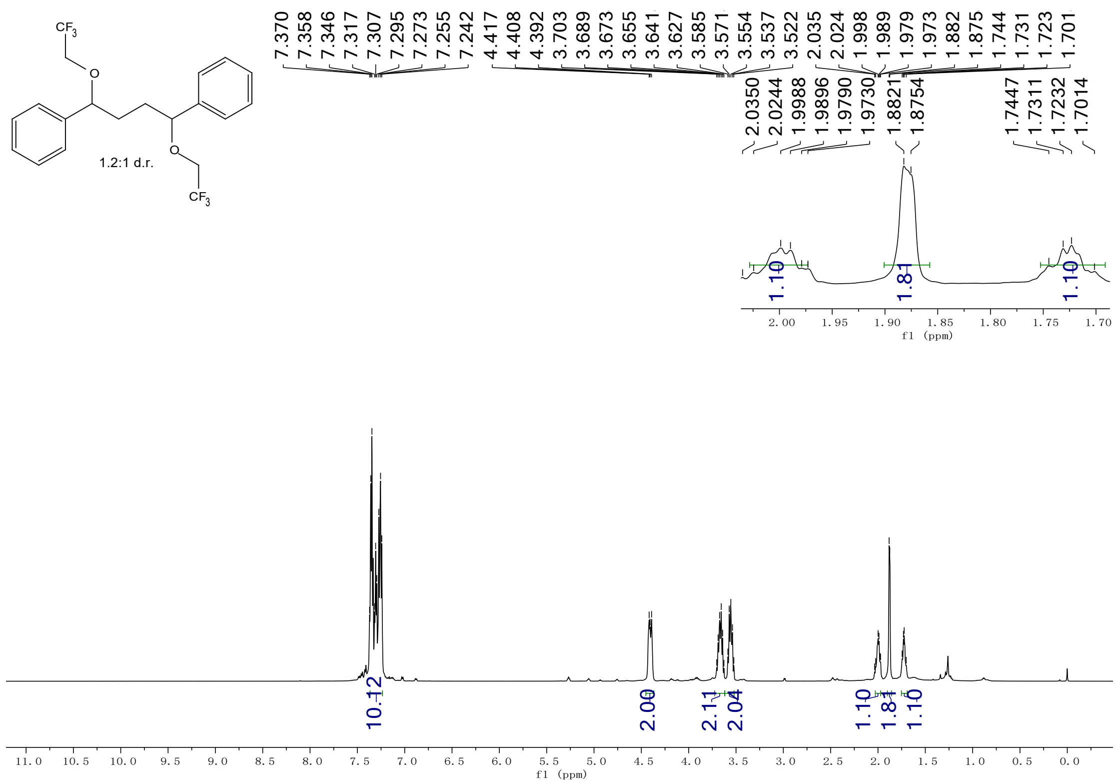
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 28



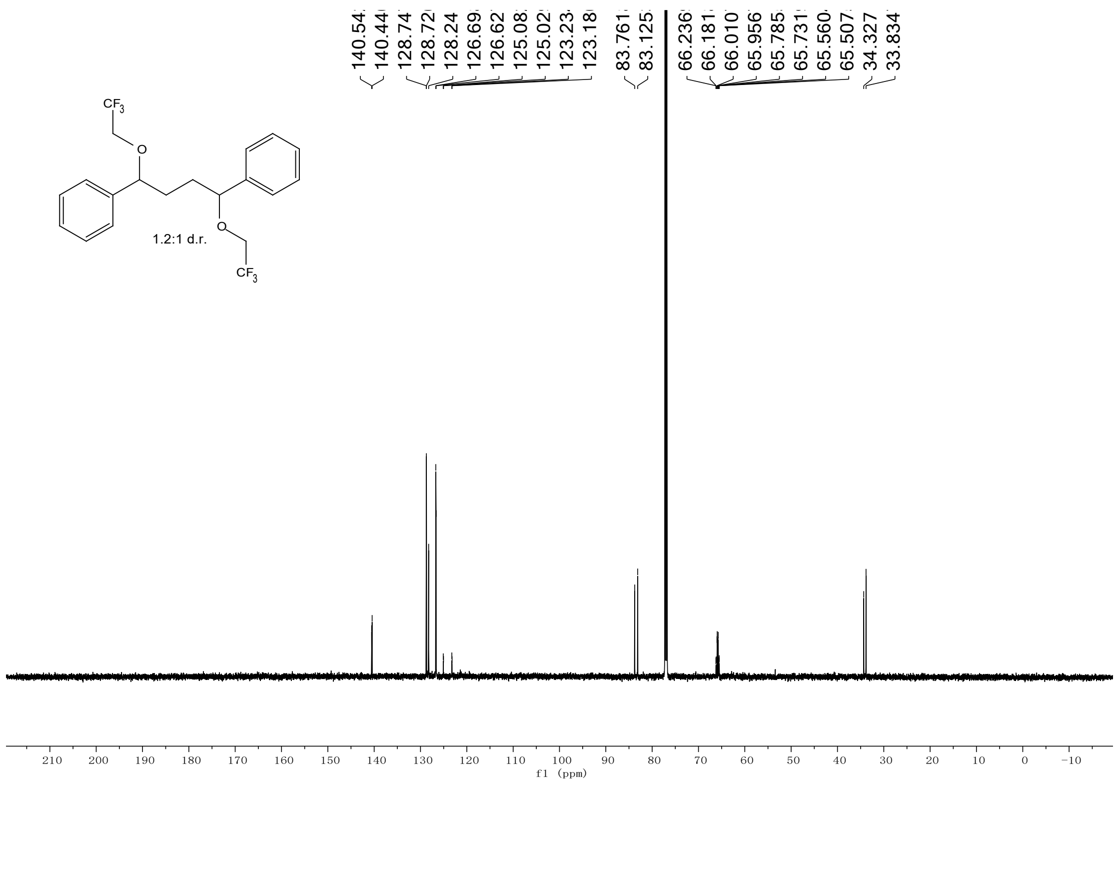
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 28



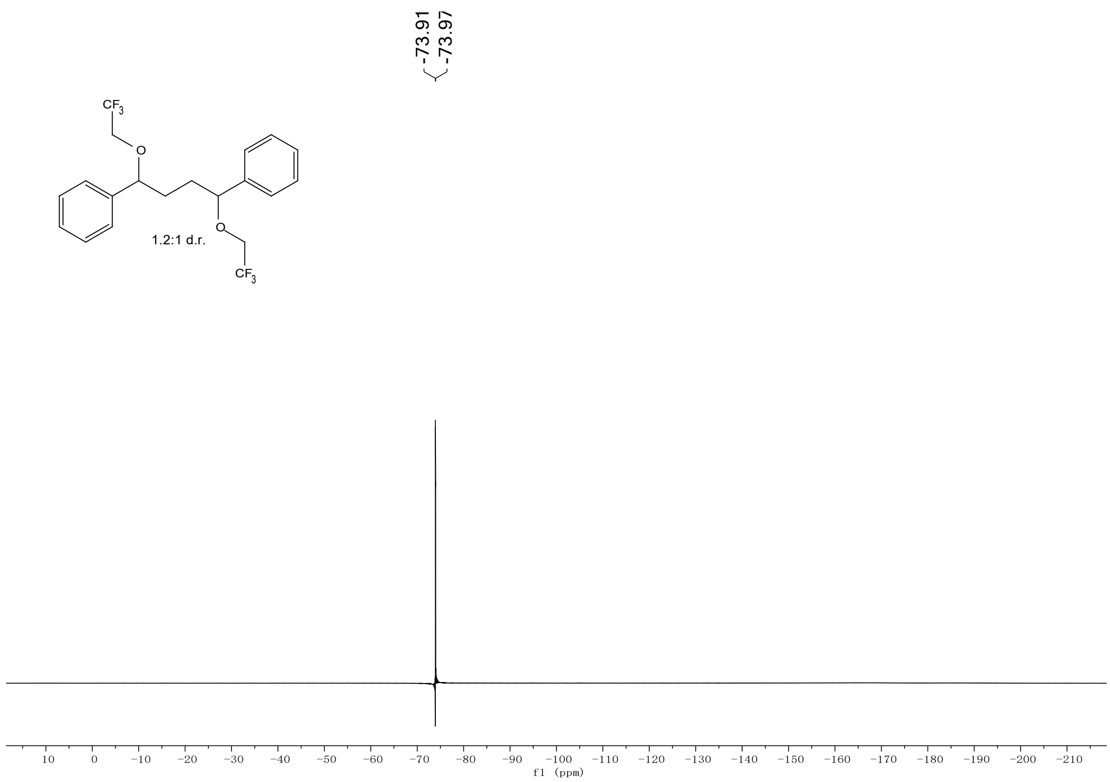
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 29



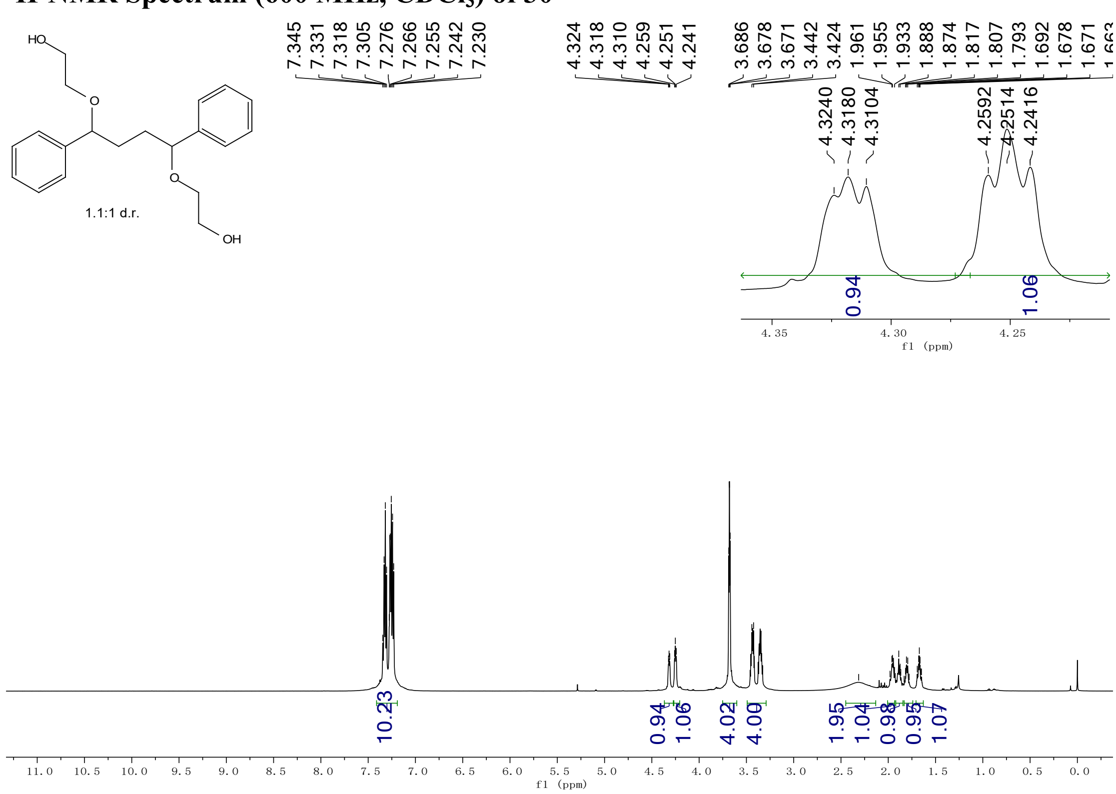
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 29



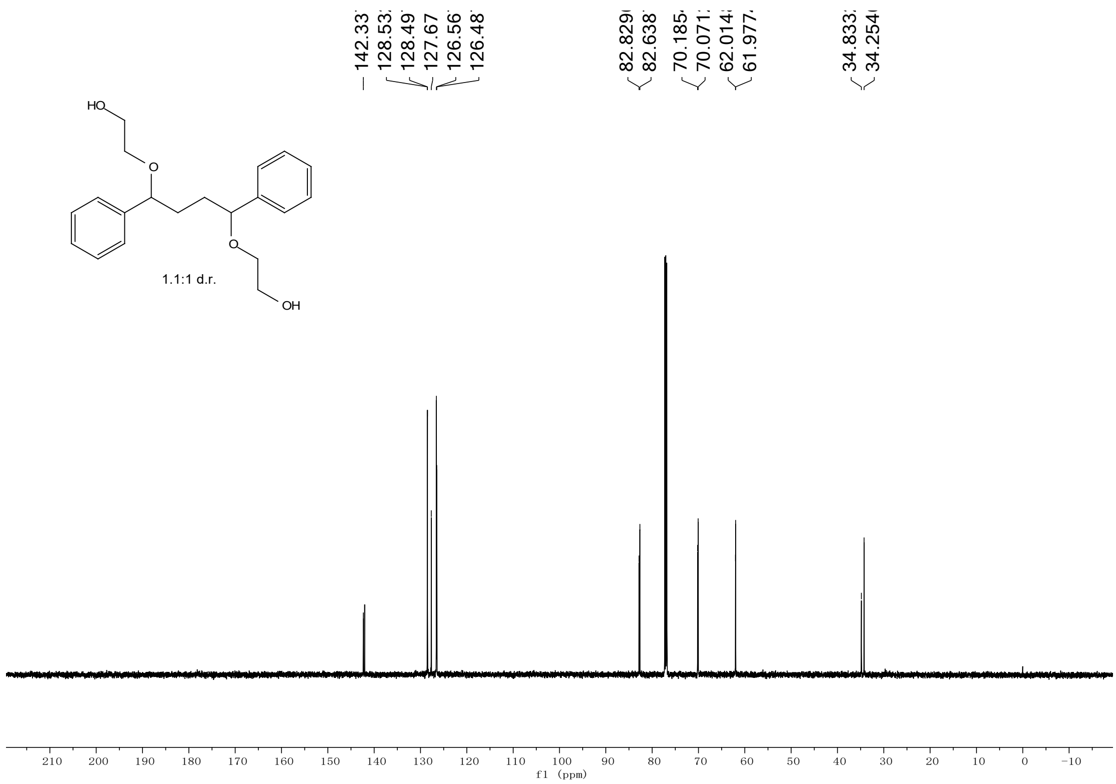
^{19}F-NMR Spectrum (151 MHz, $CDCl_3$) of 29



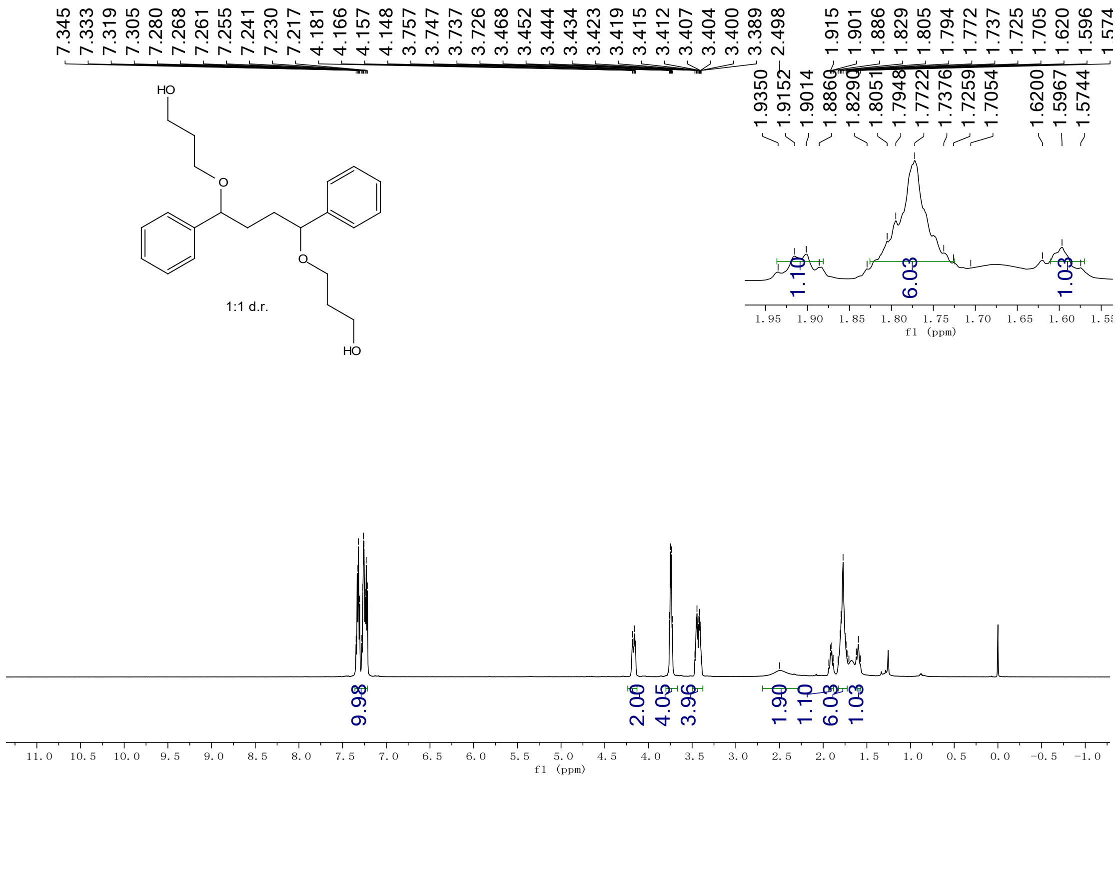
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 30



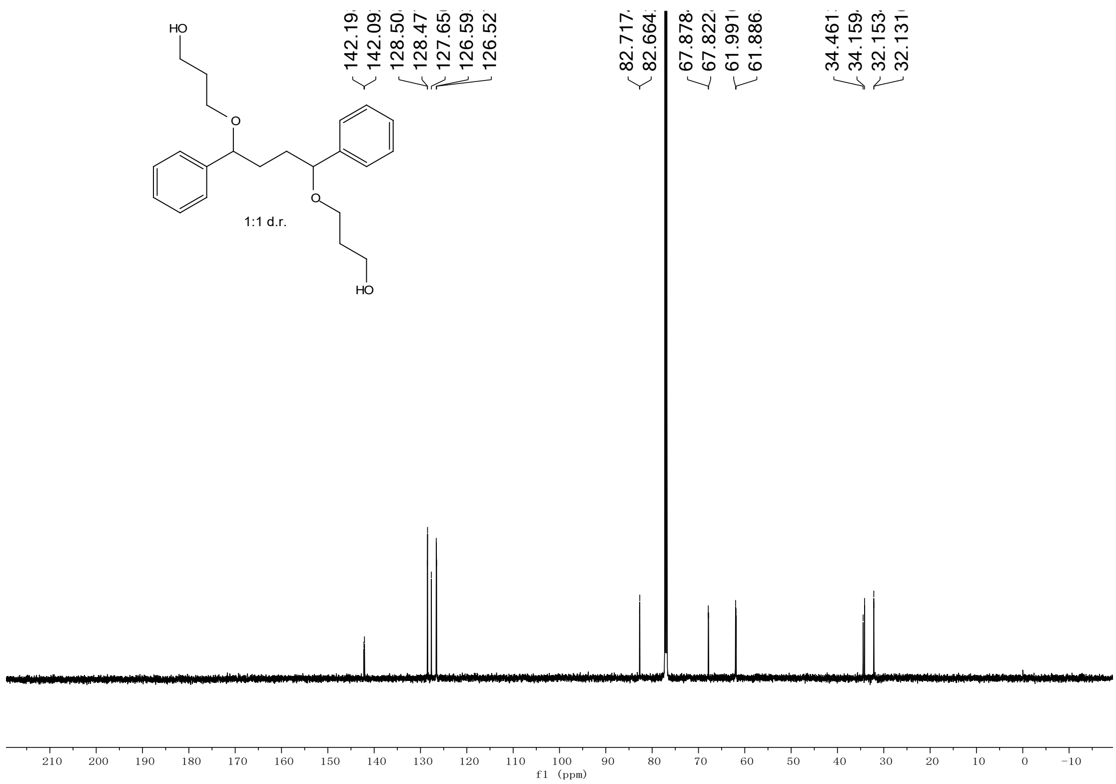
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 30



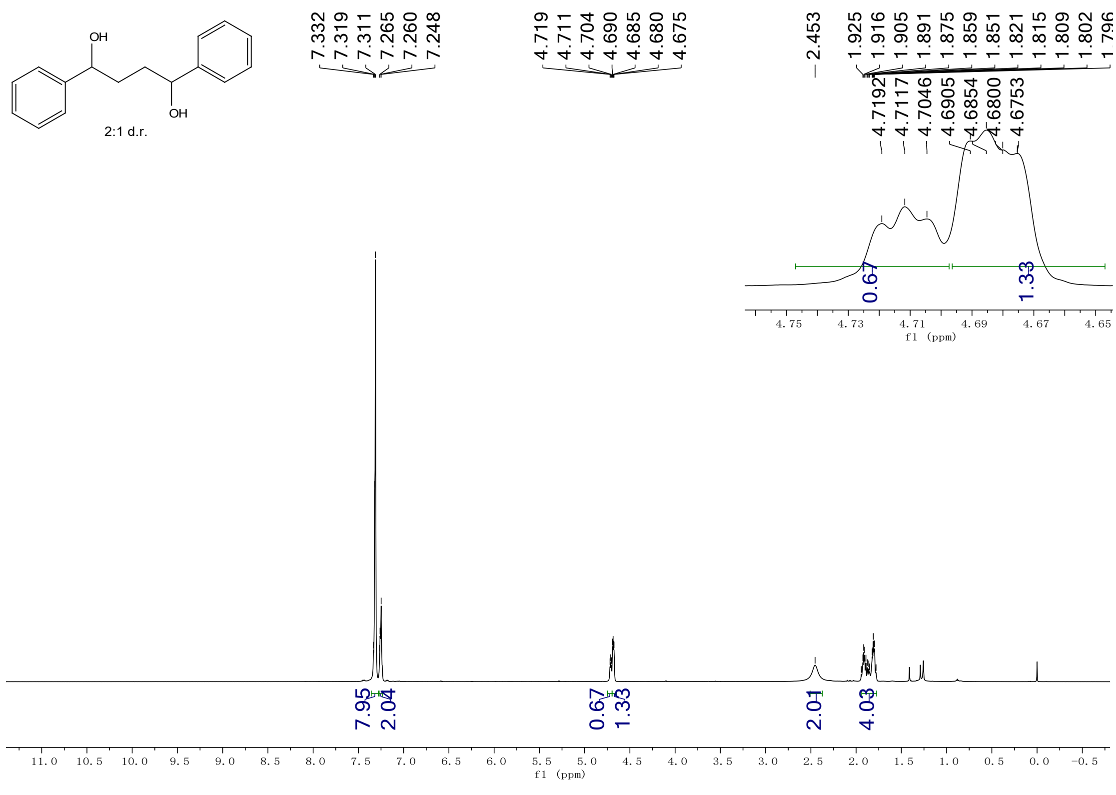
[1]H-NMR Spectrum (600 MHz, $CDCl_3$) of 31



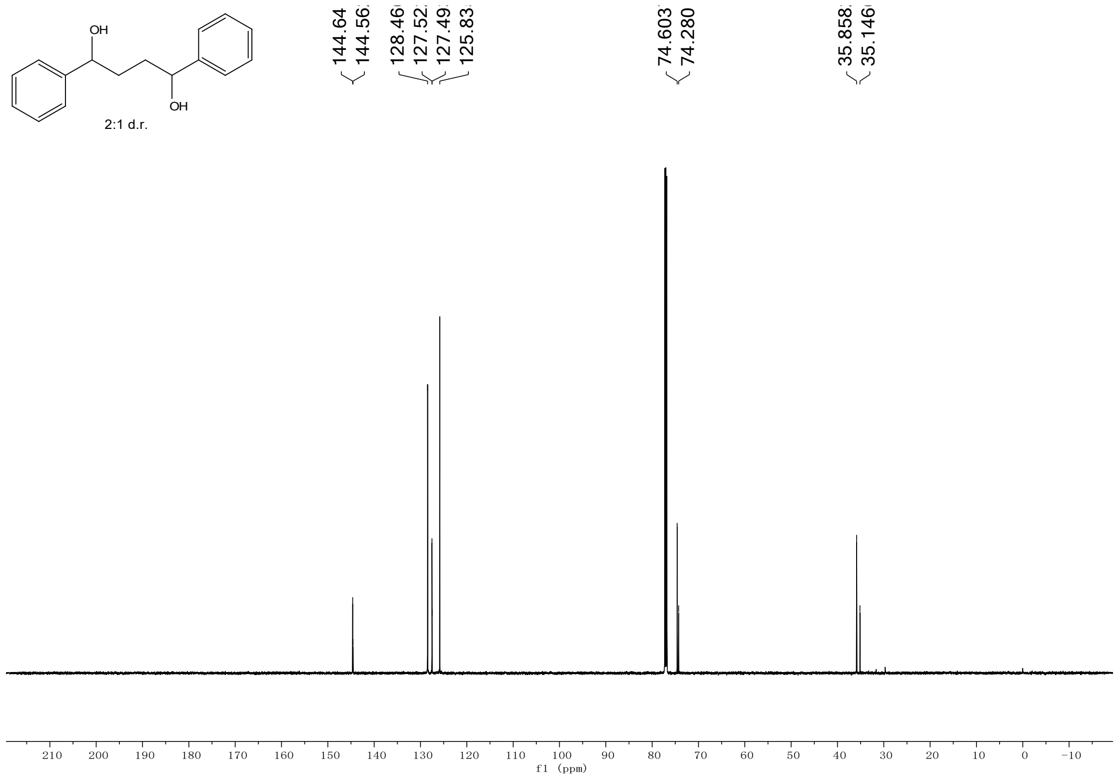
[13]C-NMR Spectrum (151 MHz, $CDCl_3$) of 31



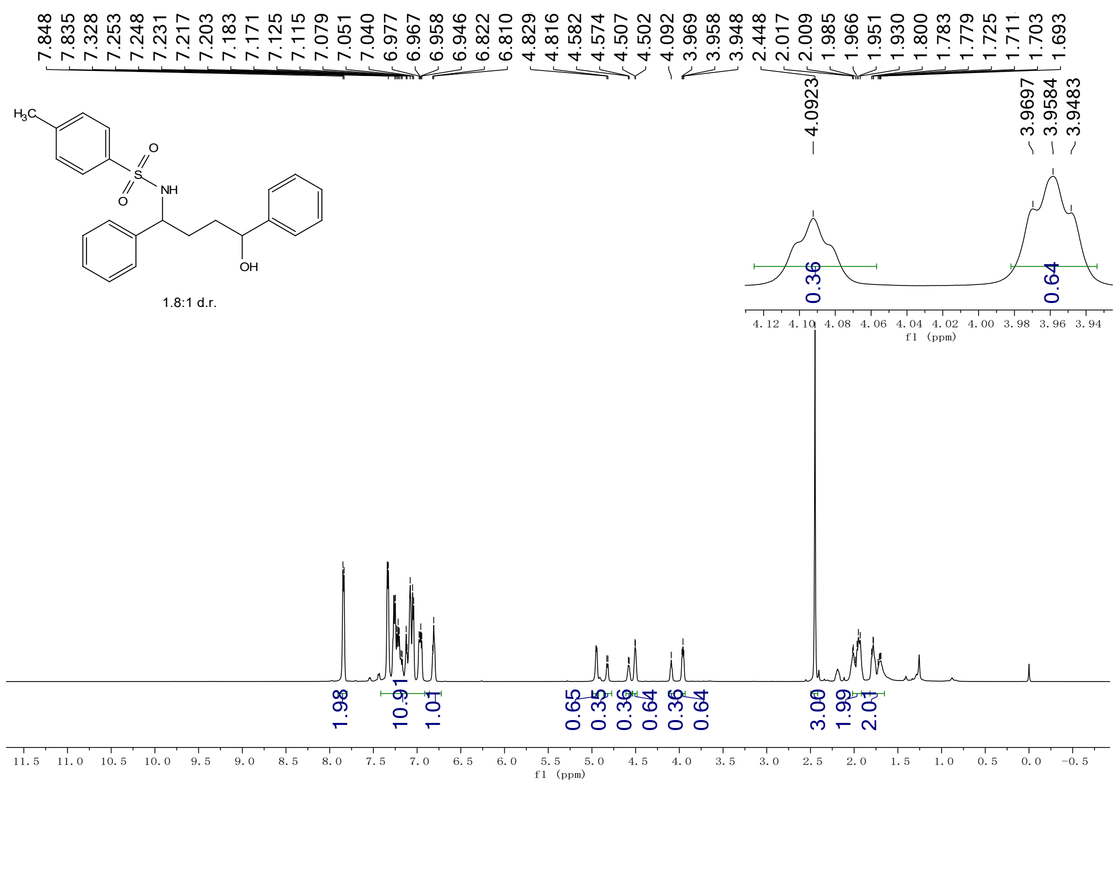
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 32



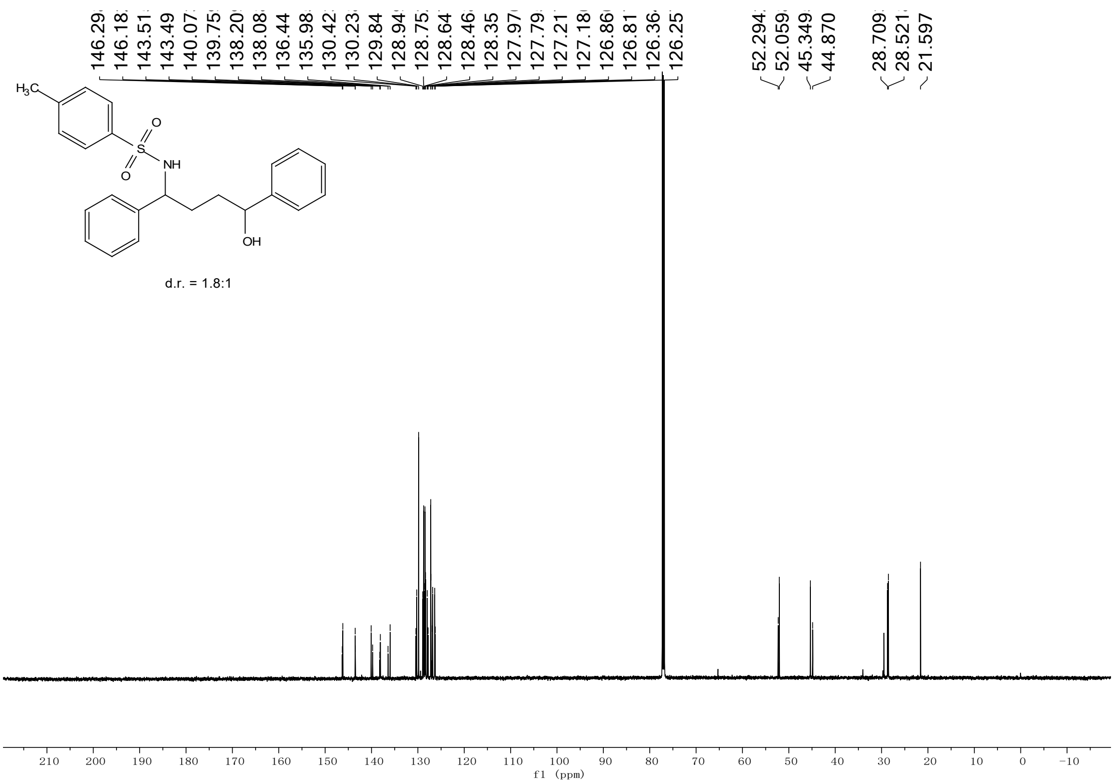
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 32



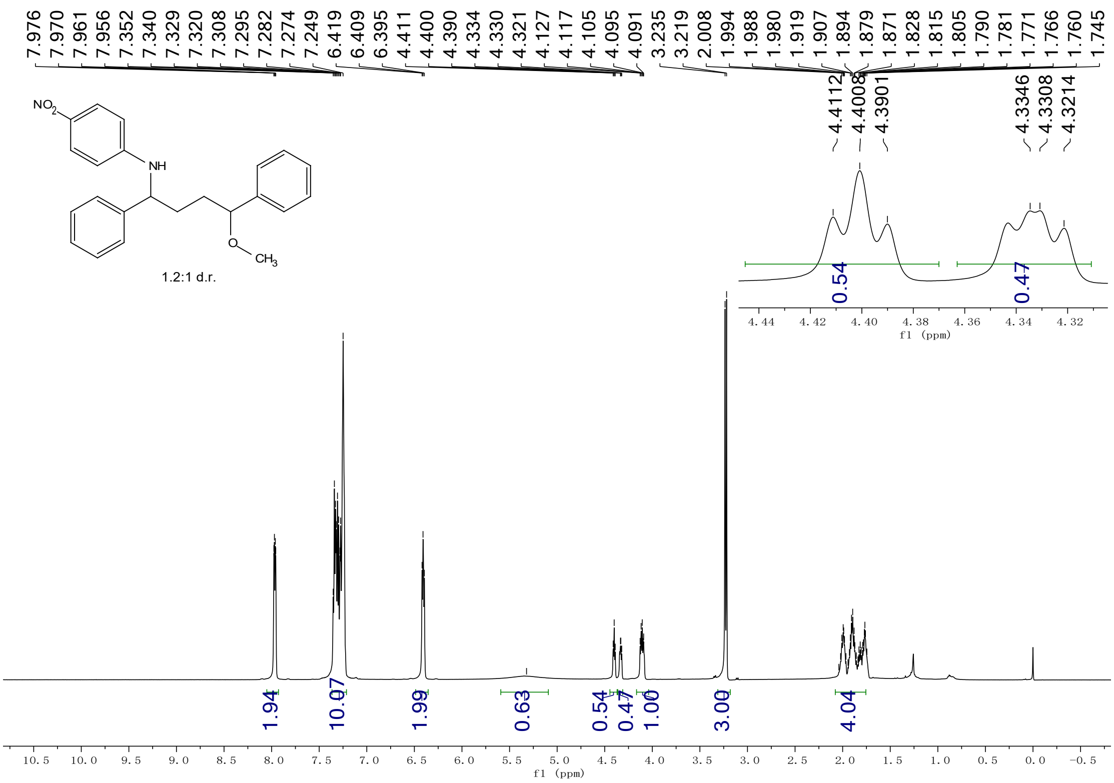
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 33



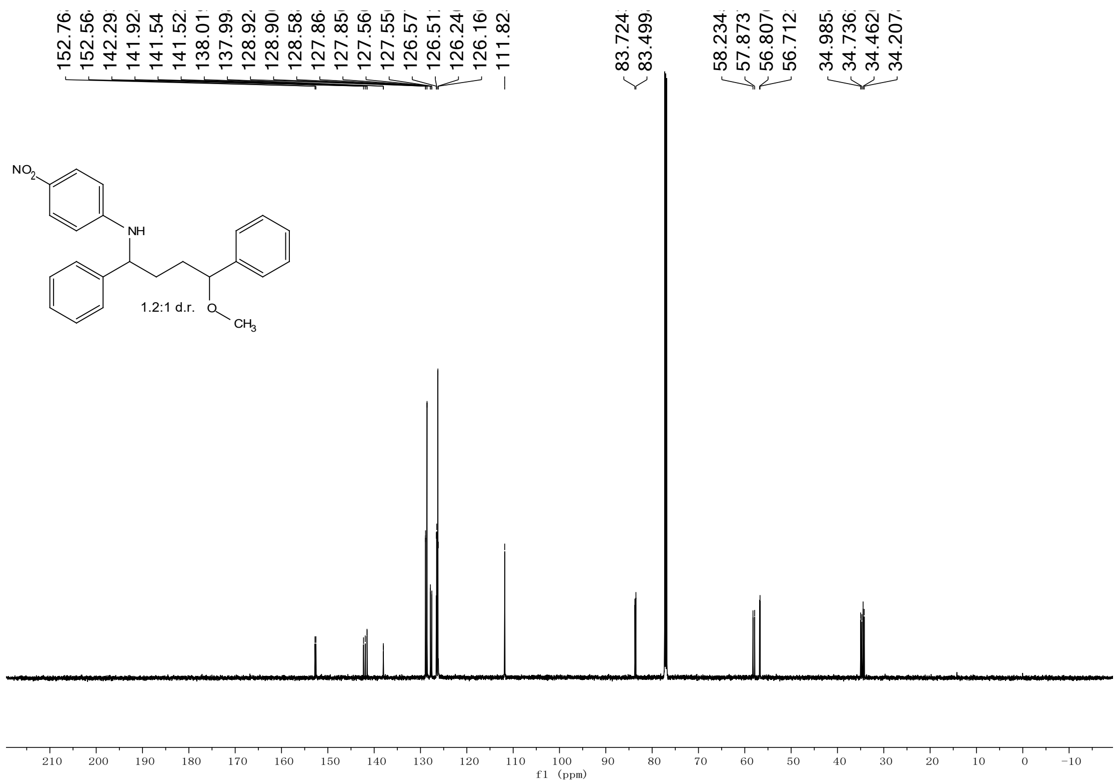
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 33



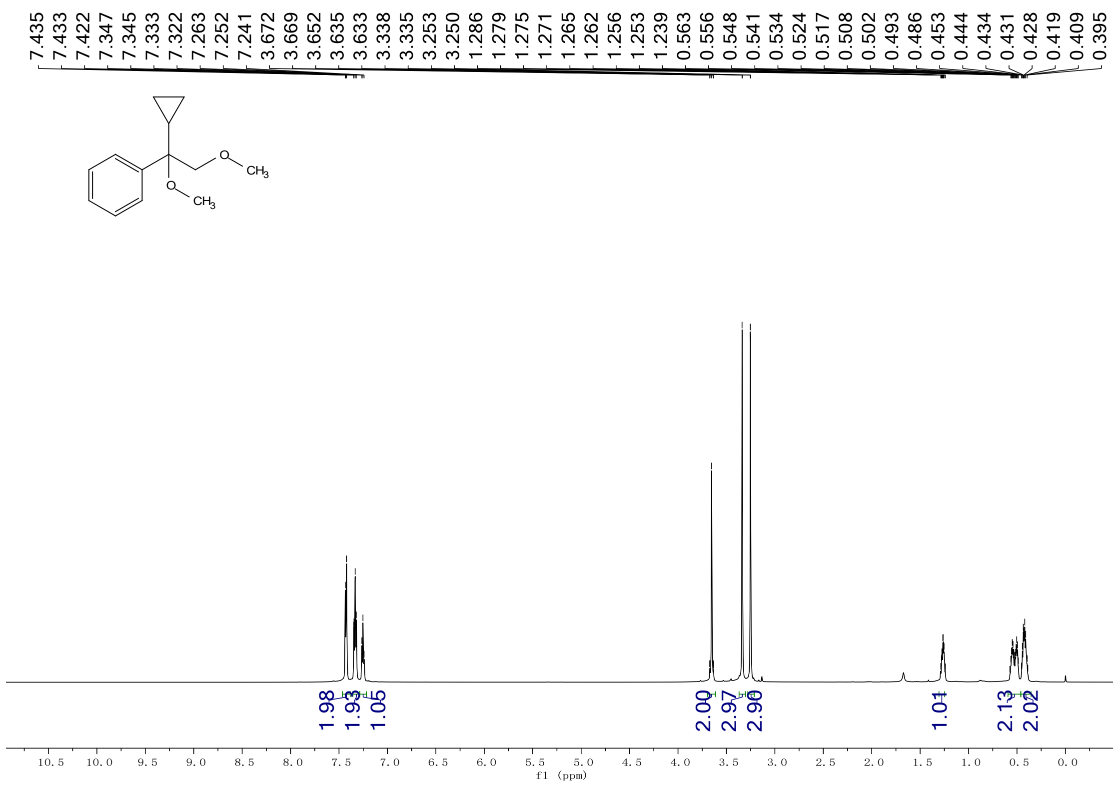
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of 34



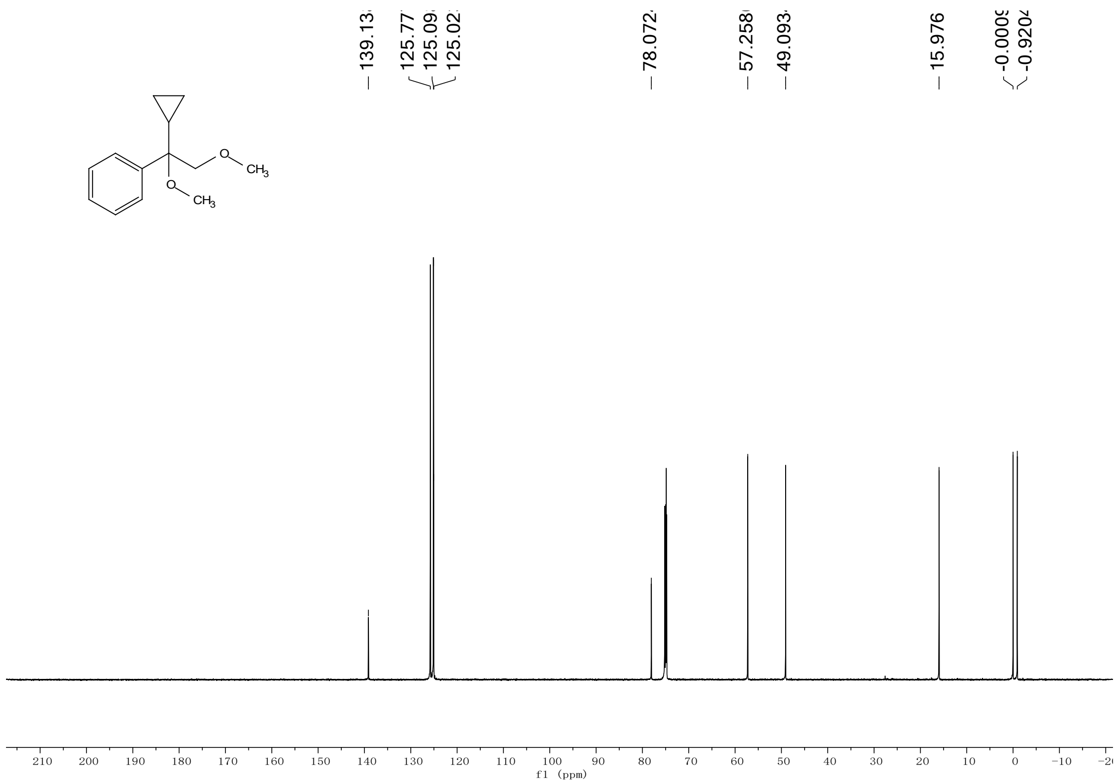
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of 34



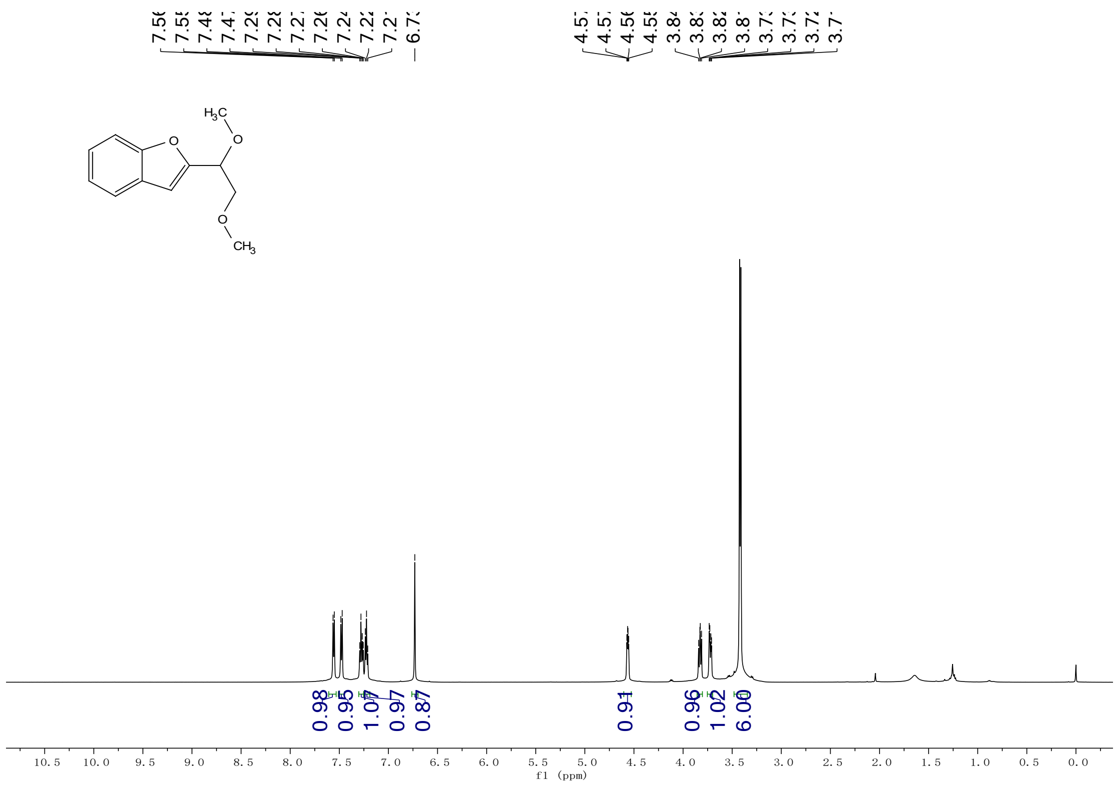
^{1}H-NMR Spectrum (600 MHz, $CDCl_3$) of A



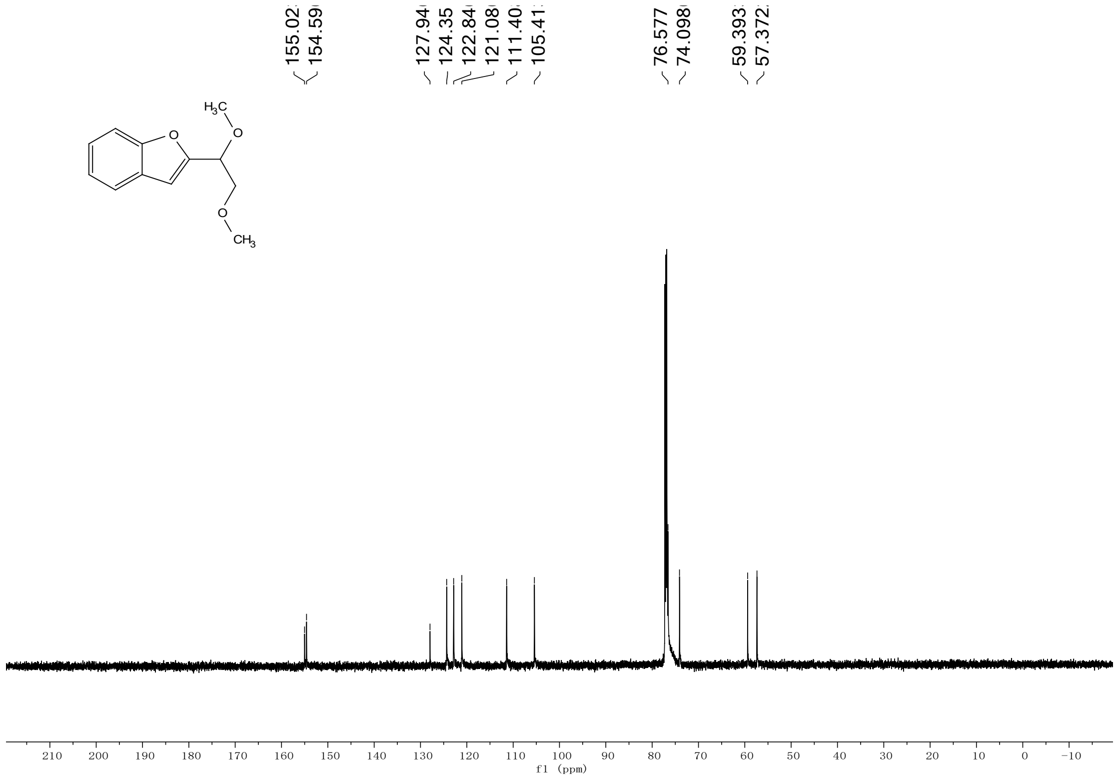
^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of A



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



^{13}C-NMR Spectrum (151 MHz, $CDCl_3$) of B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f1 (ppm)