

## Supplementary Information

### **Decarbonylative C(sp<sup>2</sup>)-C(sp<sup>2</sup>) Reductive Cross-Coupling of Aryl Fluorides with Aryl Bromides by Palladium/Cobalt Co-catalysis**

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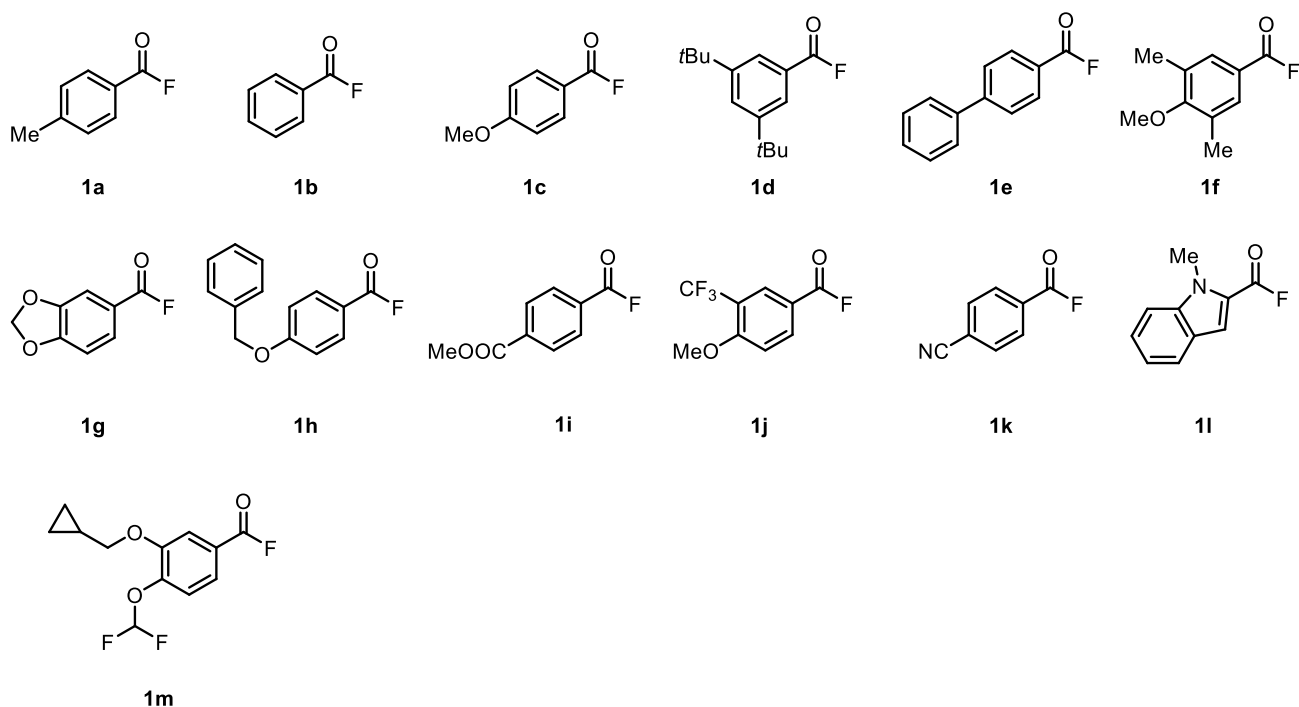
## 1. General Information

Unless otherwise stated, all manipulations were carried out under an atmosphere of nitrogen using standard Schlenk or glove box techniques. The heat source of all reactions is oil bath. Anhydrous DMAc (CaH<sub>2</sub>), DMPA (CaH<sub>2</sub>), 1,4-Dioxane (Na), and 2-Me-THF(Na) were distilled and stored over activated 3Å molecular sieves and transferred under nitrogen. Deuterated solvents were used as received (CDCl<sub>3</sub> from Leyan, China). PdCl<sub>2</sub> (Leyan, China), Pd(OAc)<sub>2</sub> (Leyan, China), Pd(acac)<sub>2</sub> (Leyan, China), Pd(PPh<sub>3</sub>)<sub>4</sub> (Leyan, China), CoBr<sub>2</sub> (Leyan, China), Co(acac)<sub>3</sub> (Leyan, China), CoCl<sub>2</sub> (Leyan, China) were used as received. Zn powder was purchased from Sigma-Aldrich and activated by acid according to M's method<sup>[1]</sup>. Dimethyl [2,2'-bipyridine]-4,4'-dicarboxylate (Leyan, China), 4,4'-Di-tert-butyl-2,2'-dipyridyl (dtbpy, Leyan, China), 1,2-Bis(dicyclohexylphosphanyl)ethane (dCype, Sigma-Aldrich), 1,2-bis(diphenylphosphanyl)ethane (dppe, Leyan, China), and tricyclohexylphosphane (PCy<sub>3</sub>, Leyan, China), triphenylphosphine (PPh<sub>3</sub>, Leyan, China) were used directly. XtalFluor-E (Bidepharm, China) were used as received. Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification.

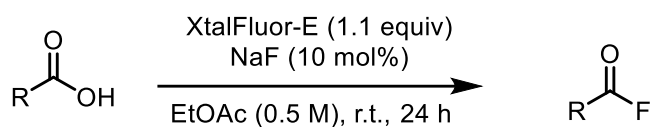
For chromatographic purification, 200-300 mesh silica gel (Leyan, China) was employed. For thin layer chromatography (TLC) analysis, High efficiency thin layer chromatography silica gel plates (HPTLC Silica Gel 60 GF254, 2.5\*5.0 cm) were used. <sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectra were recorded at room temperature using a Bruker Avance-500 instruments. The <sup>1</sup>H NMR (500 MHz) chemical shifts were measured relative to tetramethylsilane or solvent residual of CDCl<sub>3</sub> as an internal standard (TMS: δ = 0 ppm or CDCl<sub>3</sub>: δ = 7.26 ppm). The <sup>13</sup>C NMR (126 MHz) chemical shifts were given using CDCl<sub>3</sub> as the internal standard (CDCl<sub>3</sub>: δ = 77.16 ppm). Melting points were determined with a YRT-3 (Tian Jin Optical Instrument Factory). Gas chromatograph (GC) was performed using SHIMADZU Nexis GC-2030 coupled to a DM-5MS gas chromatography column. High-resolution mass spectra (HRMS) were performed using an Agilent quadrupole time of flight (QTQF, 6540) mass spectrometer, Vanquish HPLC; Thermo Q Exactive MS spectrometer and a high-resolution quadrupole-orbitrap tandem mass spectrometer (Q-Exactive plus; Thermo Fisher Scientific, Waltham, MA, USA) with electrospray ionization (ESI) probe operated in the positive-ion mode.

## 2. Synthesis of Substrates

### 2.1. The Scope of Aroyl Fluorides

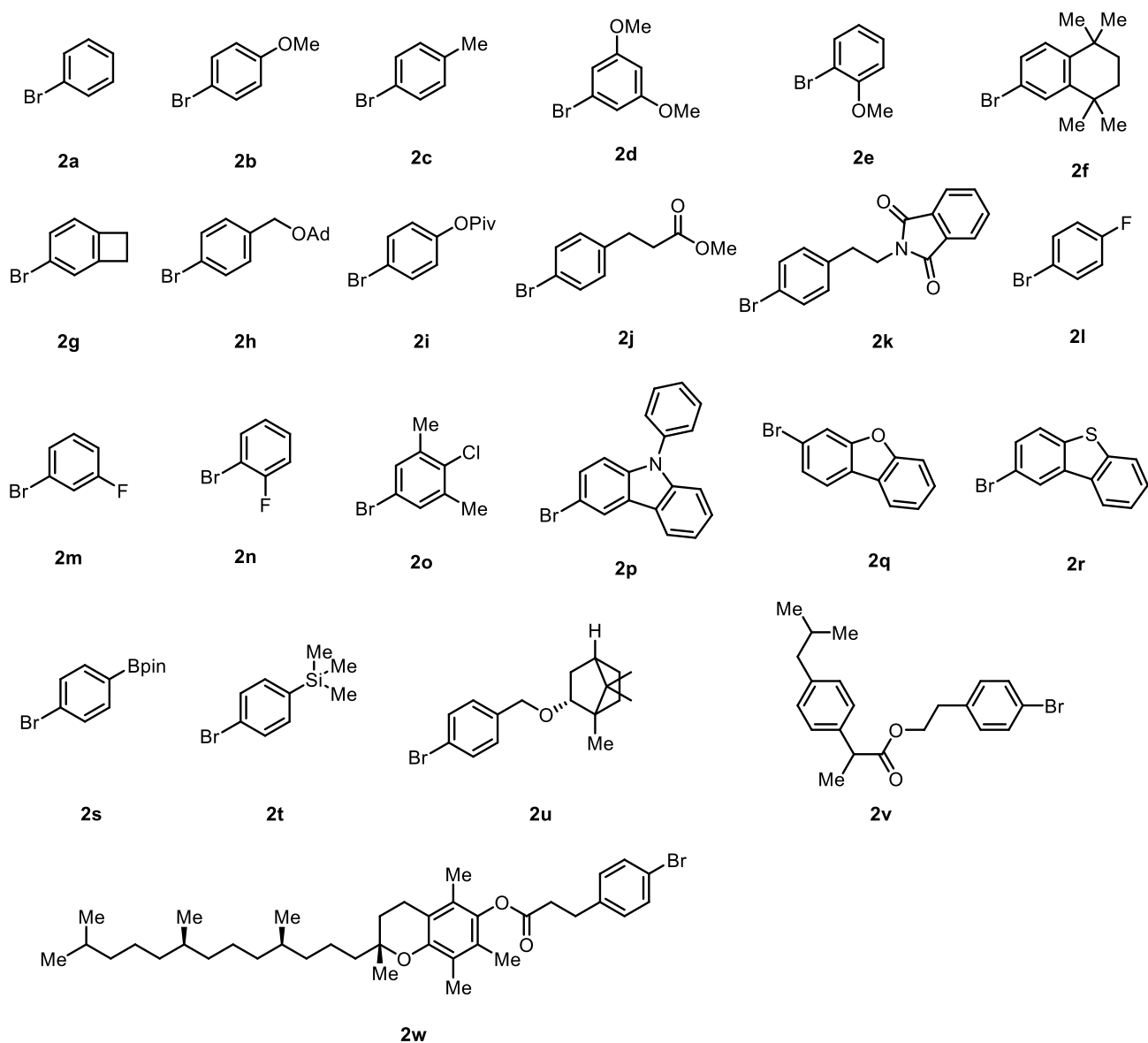


### 2.2. Synthesis of Aroyl Fluorides



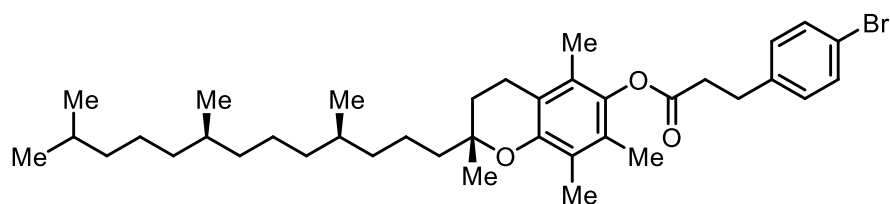
To a solution of carboxylic acid (1.0 equiv., 5.0 mmol) in dry EtOAc (0.5 M) was added NaF (10 mol%, 0.50 mmol, 21.0 mg), followed by XtalFluor-E (1.1 equiv., 5.5 mmol, 1.26 g). After 24 h of stirring at room temperature under nitrogen, the reaction mixture was vacuumed to remove the volatiles. The crude mixture was then purified by column chromatography on silica gel to afford the desired product.

### 2.3. The Scope of Aryl Bromides



**2h**<sup>[2]</sup>, **2i**<sup>[3]</sup>, **2k**<sup>[4]</sup>, **2u**<sup>[2]</sup>, **2v**<sup>[2]</sup>, **2w**<sup>[2]</sup> were synthesized following literature procedures.

### Characterization of New Compounds



**(R)-2,5,7,8-Tetramethyl-2-((4R,8R)-4,8,12-trimethyltridecyl)chroman-6-yl 3-(4-bromophenyl)propanoate, 2u**

A 100 mL round bottom flask equipped with a stir bar was charged with 3-(4-bromophenyl)propionic acid (1.15 g, 5 mmol), Vitamin E (2.58 g, 6 mmol), DCC (1.24 g, 6 mmol) and 50 mL of dry CH<sub>2</sub>Cl<sub>2</sub>.

Then 4-DMAP (0.05 g, 0.4 mmol) was added in one portion. The reaction was stirred for 4 h at room temperature. After filtration, the solution was concentrated under reduced pressure. Purification by column chromatography on silica gel (PE:EtOAc = 15:1, v/v) afforded **2u** as yellow oil (1.9 g, 3.0 mmol, 59% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.43 (d, *J* = 8.5 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 3.06 (t, *J* = 7.5 Hz, 2H), 2.91 (t, *J* = 7.5 Hz, 2H), 2.57 (t, *J* = 7.0 Hz, 2H), 2.07 (s, 3H), 1.91 (s, 3H), 1.86 (s, 3H), 1.83 – 1.72 (m, 2H), 1.55 – 1.50 (m, 3H), 1.43 – 1.34 (m, 4H), 1.31 – 1.23 (m, 10H), 1.16 – 1.11 (m, 3H), 1.09 – 1.04 (m, 3H), 0.88 – 0.84 (m, 13H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 171.4, 149.6, 140.5, 139.4, 131.7, 130.4, 126.7, 124.9, 123.2, 120.3, 117.5, 75.2, 39.5, 37.59, 37.57, 37.4, 35.5, 32.94, 32.85, 30.5, 28.1, 25.0, 24.6, 22.9, 22.8, 21.2, 20.7, 19.9, 19.8, 13.0, 12.2, 12.0 ppm.

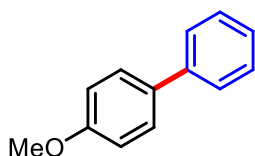
**HRMS (APCI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>38</sub>H<sub>58</sub>BrO<sub>3</sub><sup>+</sup> 641.3564, found 641.3552.

### 3. Reductive Cross-Coupling of Aryl Fluorides with Aryl Bromides

#### 3.1 General Procedure

An oven-dried Schlenk tube (20 mL) was charged with CoBr<sub>2</sub> (0.015 mmol, 3.3 mg, 5.0 mol%), dimethyl [2,2'-bipyridine]-4,4'-dicarboxylate (0.018 mmol, 4.9 mg, 6.0 mol%), Pd(OAc)<sub>2</sub> (0.015 mmol, 3.4 mg, 5.0 mol%), dCype (0.018 mmol, 7.6 mg, 6.0 mol%), Zn dust (0.9 mmol, 58.9 mg, 3.0 equiv.) aroyl fluorides (0.3 mmol, 1.0 equiv.), aryl bromides (0.45 mmol, 1.5 equiv.) and 2-Me-THF (0.8 mL). The reaction mixture was allowed to stir under N<sub>2</sub> atmosphere at 130 °C (oil bath) for 24 h. After this time, the tube was cooled to room temperature, the residue was diluted with DCM (~10 mL) and filtered through a triangular suction filter funnel with a thin layer of celite, and the filtrate was concentrated under vacuum. The crude mixture was then purified by column chromatography on silica gel (Petroleum ether/Dichloromethane) to afford the desired products.

#### 3.2 Characterization Data



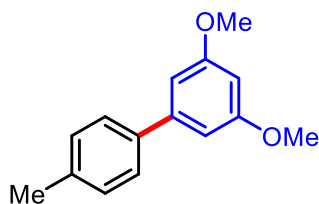
#### 4-Methoxy-1,1'-biphenyl<sup>[5]</sup>, **3a**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3a** as a white solid (32.2 mg, 0.18 mmol, 58% yield). **m.p.** 83.5 – 84.6 °C.

When using phenyl trifluoromethanesulfonate as substrate, the product **3a** can be obtained at the (17.7 mg, 0.10 mmol, 32% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.53 (dd, *J* = 10.5, 8.0 Hz, 4H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 6.97 (d, *J* = 8.5 Hz, 2H), 3.83 (s, 3H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 159.2, 140.9, 133.9, 128.8, 128.3, 126.9, 126.8, 114.3, 55.5 ppm.

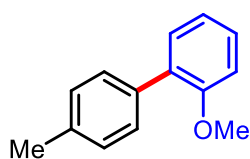


### 3,5-Dimethoxy-4'-methyl-1,1'-biphenyl<sup>[6]</sup>, **3b**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3b** as a colorless oil (32.0 mg, 0.14 mmol, 47% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.47 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 6.72 (d, *J* = 2.5 Hz, 2H), 6.45 (t, *J* = 2.0 Hz, 1H), 3.83 (s, 6H), 2.39 (s, 3H) ppm.

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 161.1, 143.5, 138.4, 137.5, 129.5, 127.1, 105.4, 99.1, 55.5, 21.3 ppm.



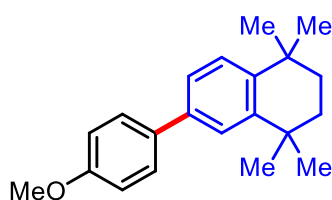
### 2-Methoxy-4'-methyl-1,1'-biphenyl<sup>[7]</sup>, **3c**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3c** as a white solid (30.0 mg, 0.15 mmol, 51% yield). **m.p.** 81.5 – 82.6 °C.

When using 2-methoxyphenyl trifluoromethanesulfonate as substrate, the product **3c** can be obtained at the (14.9 mg, 0.08 mmol, 25% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.21 (d, *J* = 7.5 Hz, 2H), 7.01 (t, *J* = 7.5 Hz, 1H), 6.96 (d, *J* = 8.0 Hz, 1H), 3.79 (s, 3H), 2.38 (s, 3H) ppm.

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 156.6, 136.7, 135.7, 130.9, 130.8, 129.5, 128.9, 128.5, 120.9, 111.3, 55.6, 21.3 ppm.



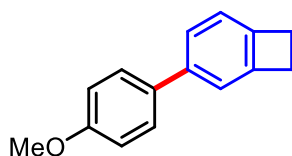
### 6-(4-Methoxyphenyl)-1,1,4,4-tetramethyl-1,2,3,4-tetrahydronaphthalene, **3d**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3d** as a white solid (46.0 mg, 0.16 mmol, 52% yield). **m.p.** 119.0 – 119.6 °C.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.52 – 7.49 (m, 2H), 7.47 (d, *J* = 2.0 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.31 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.97 – 6.94 (m, 2H), 3.83 (s, 3H), 1.71 (s, 4H), 1.33 (s, 6H), 1.31 (s, 6H) ppm.

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 159.0, 145.3, 143.5, 138.1, 134.3, 128.2, 127.1, 125.0, 124.3, 114.2, 55.4, 35.3, 35.2, 34.5, 34.2, 32.1, 32.0 ppm.

HRMS (ESI<sup>+</sup>): *m/z*: [M+H]<sup>+</sup> calcd for C<sub>21</sub>H<sub>27</sub>O<sup>+</sup> 295.2056, found 295.2053.



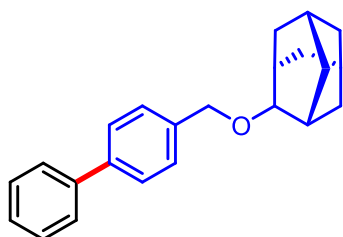
### 3-(4-Methoxyphenyl)bicyclo[4.2.0]octa-1(6),2,4-triene, **3e**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3e** as a white solid. (24.2 mg, 0.12 mmol, 38% yield). **m.p.** 43.6 – 44.9 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.48 – 7.45 (m, 2H), 7.36 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.23 (s, 1H), 7.09 (d, *J* = 7.5 Hz, 1H), 6.97 – 6.94 (m, 2H), 3.83 (s, 3H), 3.20 (s, 4H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 158.9, 146.3, 144.4, 140.1, 135.1, 128.4, 125.9, 122.8, 121.4, 114.2, 55.5, 29.6, 29.5 ppm.

**HRMS (ESI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>15</sub>H<sub>15</sub>O<sup>+</sup> 211.1117, found 211.1116.



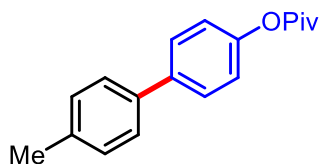
### 2-([1,1'-biphenyl]-4-ylmethoxy)adamantane, **3f**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3f** as a white solid (43.9 mg, 0.14 mmol, 46% yield). **m.p.** 94.2 – 96.0 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.60 – 7.56 (m, 4H), 7.46 – 7.42 (m, 4H), 7.33 (t, *J* = 7.5 Hz, 1H), 4.58 (s, 2H), 3.58 (s, 1H), 2.17 – 2.11 (m, 4H), 1.88 – 1.81 (m, 4H), 1.73 (s, 2H), 1.66 (d, *J* = 11.5 Hz, 2H), 1.52 (d, *J* = 11.5 Hz, 2H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 141.2, 140.3, 138.7, 128.9, 127.9, 127.3, 127.22, 127.20, 81.4, 69.1, 37.7, 36.7, 31.9, 31.8, 27.61, 27.59 ppm.

**HRMS (ESI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>23</sub>H<sub>27</sub>O<sup>+</sup> 319.2056, found 319.2080.



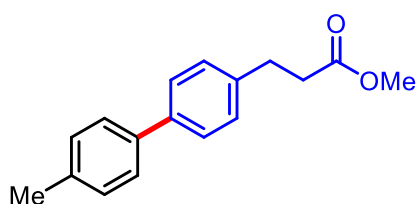
### 4'-Methyl-[1,1'-biphenyl]-4-yl pivalate<sup>[8]</sup>, **3g**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3g** as a white solid (34.8 mg, 0.13 mmol, 43% yield). **m.p.** 91.9 – 93.0 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.57 – 7.54 (m, 2H), 7.46 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.12 – 7.09 (m, 2H), 2.38 (s, 3H), 1.37 (s, 9H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 177.3, 150.4, 138.8, 137.7, 137.2, 129.6, 128.0, 127.1, 121.8, 39.2, 27.3, 21.2 ppm.





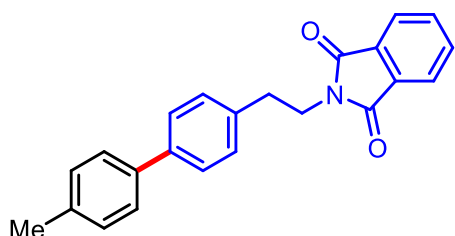
### Methyl 3-(4'-methyl-[1,1'-biphenyl]-4-yl)propanoate, **3h**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3h** as a white solid (35.2 mg, 0.14 mmol, 46% yield). **m.p.** 67.2 – 69.0 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.50 (d, *J* = 8.5 Hz, 2H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.24 (dd, *J* = 11.0, 8.5 Hz, 4H), 3.68 (s, 3H), 2.98 (t, *J* = 7.5 Hz, 2H), 2.66 (t, *J* = 8.0 Hz, 2H), 2.38 (s, 3H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 173.5, 139.4, 139.3, 138.1, 137.0, 129.6, 128.8, 127.2, 126.9, 51.8, 35.8, 30.7, 21.2 ppm.

**HRMS (ESI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>17</sub>H<sub>19</sub>O<sub>2</sub><sup>+</sup> 255.1380, found 255.1375.



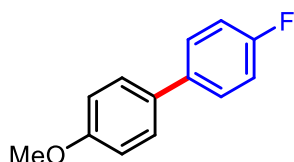
### 2-(2-(4'-Methyl-[1,1'-biphenyl]-4-yl)ethyl)isoindoline-1,3-dione, **3i**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 3/1, v/v) afforded **3i** as a white solid (30.8 mg, 0.09 mmol, 30% yield). **m.p.** 180.9 – 182.2 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.83 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.70 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.50 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 3.95 (t, *J* = 8.0 Hz, 2H), 3.03 (t, *J* = 8.0 Hz, 2H), 2.38 (s, 3H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 168.3, 139.6, 138.1, 137.0, 136.9, 134.0, 132.2, 129.6, 129.4, 127.2, 127.0, 123.4, 39.4, 34.4, 21.2 ppm.

**HRMS (ESI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>23</sub>H<sub>20</sub>NO<sub>2</sub><sup>+</sup> 342.1489, found 342.1482.



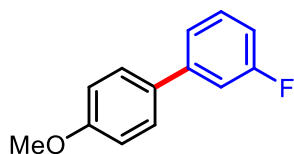
### 4-Fluoro-4'-methoxy-1,1'-biphenyl<sup>[5]</sup>, **3j**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3j** as a white solid (27.0 mg, 0.13 mmol, 45% yield). **m.p.** 92.1 – 93.4 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.50 – 7.45 (m, 4H), 7.11 – 7.07 (m, 2H), 6.98 – 6.95 (m, 2H), 3.84 (s, 3H) ppm.

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  162.2 (d,  $J = 246.0$  Hz), 159.2, 137.1 (d,  $J = 3.3$  Hz), 133.0, 128.3 (d,  $J = 7.9$  Hz), 128.2, 115.7 (d,  $J = 21.3$  Hz), 114.4, 55.5 ppm.

$^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ):  $\delta$  -116.7 (m) ppm.



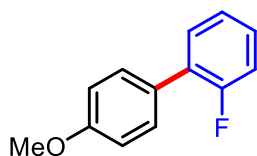
### 3-Fluoro-4'-methoxy-1,1'-biphenyl<sup>[9]</sup>, **3k**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3k** as a white solid (25.0 mg, 0.12 mmol, 41% yield). **m.p.** 65.2 – 66.8 °C.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.52 – 7.50 (m, 2H), 7.38 – 7.34 (m, 1H),  $\delta$  7.32 (dt,  $J = 7.5, 1.5$  Hz, 1H), 7.26 – 7.23 (m, 1H), 7.00 – 6.96 (m, 3H), 3.85 (s, 3H) ppm.

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  163.4 (d,  $J = 245.6$  Hz), 159.7, 143.2 (d,  $J = 7.6$  Hz), 132.6 (d,  $J = 2.1$  Hz), 130.3 (d,  $J = 8.7$  Hz), 128.3, 122.4 (d,  $J = 2.8$  Hz), 114.4, 113.6 (d,  $J = 21.9$  Hz), 113.5 (d,  $J = 21.3$  Hz), 55.5 ppm.

$^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ):  $\delta$  -113.3 (m) ppm.



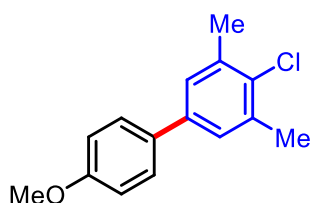
### 2-Fluoro-4'-methoxy-1,1'-biphenyl<sup>[10]</sup>, **3l**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3l** as a white solid (30.4 mg, 0.15 mmol, 50% yield). **m.p.** 42.0 – 43.0 °C.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.49 (dd,  $J = 8.5, 1.0$  Hz, 2H), 7.41 (td,  $J = 7.5, 1.5$  Hz, 1H), 7.29 – 7.25 (m, 1H), 7.18 (td,  $J = 7.5, 1.0$  Hz, 1H), 7.15 – 7.11 (m, 1H), 6.98 (d,  $J = 9.0$  Hz, 2H), 3.85 (s, 3H) ppm.

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  159.9 (d,  $J = 247.2$  Hz), 159.3, 130.6 (d,  $J = 3.5$  Hz), 130.3 (d,  $J = 3.0$  Hz), 128.8 (d,  $J = 13.4$  Hz), 128.5 (d,  $J = 8.2$  Hz), 128.3, 124.4 (d,  $J = 3.8$  Hz), 116.2 (d,  $J = 22.9$  Hz), 114.0, 55.4 ppm.

$^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ ):  $\delta$  -118.2 (s) ppm.

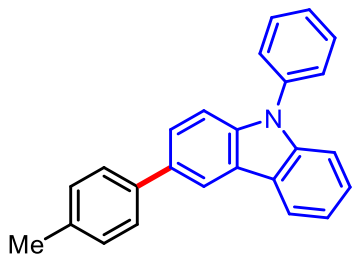


### 4-Chloro-4'-methoxy-3,5-dimethyl-1,1'-biphenyl<sup>[11]</sup>, **3m**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3m** as a white solid (35.0 mg, 0.14 mmol, 47% yield). **m.p.** 121.4 – 122.9 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.46 (d, *J* = 8.5 Hz, 2H), 7.24 (s, 2H), 6.94 (d, *J* = 9.0 Hz, 2H), 3.83 (s, 3H), 2.42 (s, 6H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 159.3, 138.7, 136.5, 133.4, 133.0, 128.1, 126.8, 114.3, 55.4, 21.0 ppm.

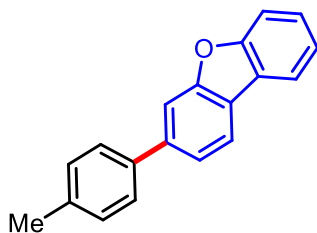


### 9-Phenyl-3-(*p*-tolyl)-9*H*-carbazole<sup>[12]</sup>, **3n**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3n** as a white solid (36.2 mg, 0.11 mmol, 36% yield). **m.p.** 117.2 – 118.2 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 8.33 (d, *J* = 1.5 Hz, 1H), 8.18 (d, *J* = 7.5 Hz, 1H), 7.64 – 7.57 (m, 7H), 7.48 – 7.41 (m, 4H), 7.31 – 7.28 (m, 3H), 2.42 (s, 3H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 141.4, 140.3, 139.2, 137.8, 136.4, 133.6, 130.0, 129.6, 127.6, 127.3, 127.2, 126.2, 125.5, 124.0, 123.6, 120.5, 120.1, 118.7, 110.1, 110.0, 21.3 ppm.



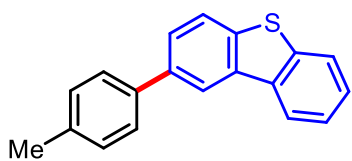
### 3-(*p*-Tolyl)dibenzo[*b,d*]furan, **3o**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3o** as a white solid (31.0 mg, 0.12 mmol, 40% yield). **m.p.** 154.0 – 156.0 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.96 (t, *J* = 8.5 Hz, 2H), 7.76 (d, *J* = 1.0 Hz, 1H), 7.59 – 7.56 (m, 4H), 7.47 – 7.43 (m, 1H), δ 7.34 (td, *J* = 7.5, 1.0 Hz, 1H), 7.28 (d, *J* = 7.5 Hz, 2H), 2.41 (s, 3H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 157.0, 156.7, 140.9, 138.3, 137.5, 129.8, 127.4, 127.1, 124.2, 123.2, 122.9, 122.1, 120.8, 120.7, 111.8, 110.0, 21.3 ppm.

**HRMS (ESI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>19</sub>H<sub>15</sub>O<sup>+</sup> 259.1117, found 259.1114.



### 2-(*p*-Tolyl)dibenzo[*b,d*]thiophene, **3p**

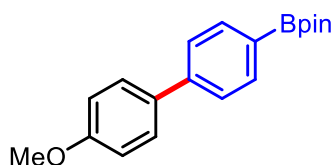
This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3p** as a white

solid (30.4 mg, 0.11 mmol, 37% yield). **m.p.** 150.0 – 152.0 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 8.32 (d, *J* = 1.5 Hz, 1H), 8.22 – 8.19 (m, 1H), 7.89 – 7.85 (m, 2H), 7.67 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.48 – 7.44 (m, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 2.42 (s, 3H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 140.0, 138.4, 138.3, 138.0, 137.2, 136.2, 135.7, 129.7, 127.3, 126.9, 126.2, 124.5, 123.1, 123.0, 121.8, 119.9, 21.3 ppm.

**HRMS (ESI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>19</sub>H<sub>15</sub>S<sup>+</sup> 275.0889, found 275.0873.

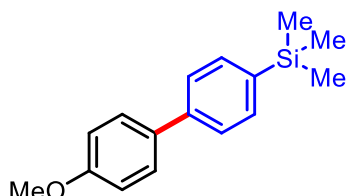


### 2-(4'-Methoxy-[1,1'-biphenyl]-4-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane<sup>[13]</sup>, **3q**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3q** as a white solid (39.1 mg, 0.13 mmol, 42% yield). **m.p.** 144.0 – 144.7 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.86 (d, *J* = 8.0 Hz, 2H), 7.57 (dd, *J* = 8.5, 4.5 Hz, 4H), 6.98 (d, *J* = 9.0 Hz, 2H), 3.85 (s, 3H), 1.36 (s, 12H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 159.5, 143.6, 135.4, 133.6, 128.4, 126.1, 114.3, 83.9, 55.5, 25.0 ppm. (the signal for the carbon that is attached to the boron atom was not observed.)

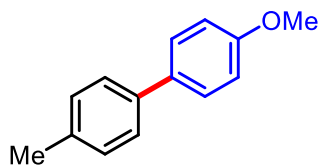


### (4'-Methoxy-[1,1'-biphenyl]-4-yl)trimethylsilane<sup>[14]</sup>, **3r**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3r** as a white solid (39.2 mg, 0.15 mmol, 51% yield). **m.p.** 91.9 – 93.0 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.59 – 7.52 (m, 6H), 6.99 – 6.96 (m, 2H), 3.84 (s, 3H), 0.29 (s, 9H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 159.3, 141.3, 138.6, 133.9, 133.8, 128.3, 126.2, 114.3, 55.5, 0.9 ppm.



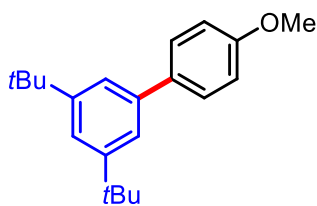
### 4-Methoxy-4'-methyl-1,1'-biphenyl<sup>[15]</sup>, **3s**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3s** as a white solid (25.0 mg, 0.13 mmol, 42% yield). **m.p.** 104.8 – 105.7 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.50 (d, *J* = 9.0 Hz, 2H), 7.44 (d, *J* = 8.5 Hz, 2H), 7.22 (d, *J* = 8.0 Hz,

2H), 6.95 (d,  $J = 9.0$  Hz, 2H), 3.83 (s, 3H), 2.37 (s, 3H) ppm.

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  159.0, 138.1, 136.5, 133.9, 129.6, 128.1, 126.7, 114.3, 55.5, 21.2 ppm.

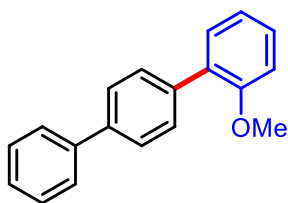


### 3,5-Di-*tert*-butyl-4'-methoxy-1,1'-biphenyl<sup>[16]</sup>, **3t**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3t** as a white solid (59.6 mg, 0.20 mmol, 67% yield). **m.p.** 88.0 – 89.9 °C.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.53 (d,  $J = 9.0$  Hz, 2H), 7.40 – 7.38 (m, 3H), 6.98 (d,  $J = 8.5$  Hz, 2H), 3.84 (s, 3H), 1.37 (s, 18H) ppm.

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  159.0, 151.2, 140.4, 135.2, 128.6, 121.5, 121.0, 114.2, 55.5, 35.1, 31.7 ppm.

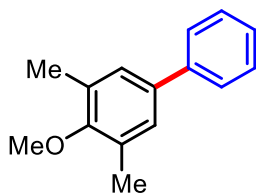


### 2-Methoxy-1,1':4',1''-terphenyl<sup>[17]</sup>, **3u**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3u** as a white solid (40.7 mg, 0.16 mmol, 52% yield). **m.p.** 116.5 – 118.4 °C.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.65 – 7.60 (m, 6H), 7.44 (t,  $J = 7.5$  Hz, 2H), 7.38 – 7.31 (m, 3H), 7.06 – 7.03 (m, 1H), 6.99 (d,  $J = 8.0$  Hz, 1H), 3.82 (s, 3H) ppm.

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  156.6, 141.1, 139.9, 137.6, 130.9, 130.3, 130.0, 128.9, 128.8, 127.3, 127.2, 126.9, 121.0, 111.3, 55.7 ppm.



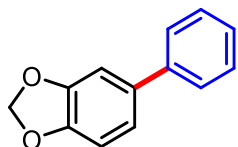
### 4-Methoxy-3,5-dimethyl-1,1'-biphenyl<sup>[18]</sup>, **3v**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3v** as a colorless oil (35.5 mg, 0.17 mmol, 56% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.53 (d,  $J = 7.0$  Hz, 2H), 7.39 (t,  $J = 7.5$  Hz, 2H), 7.29 (t,  $J = 7.5$  Hz, 1H), 7.23 (s, 2H), 3.75 (s, 3H), 2.34 (s, 6H) ppm.

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  156.7, 141.1, 136.9, 131.2, 128.7, 127.7, 127.1, 127.0, 59.9, 16.4

ppm.

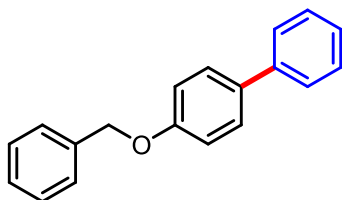


#### 5-Phenylbenzo[d][1,3]dioxole<sup>[5]</sup>, **3w**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3w** as a colorless oil (28.5 mg, 0.14 mmol, 48% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.52 – 7.50 (m, 2H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.07 – 7.04 (m, 2H), 6.88 (d, *J* = 8.0 Hz, 1H), 5.99 (s, 2H) ppm.

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 148.2, 147.2, 141.0, 135.7, 128.9, 127.1, 127.0, 120.8, 108.7, 107.8, 101.3 ppm.

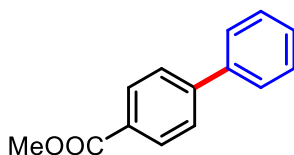


#### 4-(Benzyloxy)-1,1'-biphenyl<sup>[19]</sup>, **3x**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3x** as a white solid (32.0 mg, 0.12 mmol, 41% yield). **m.p.** 131.7 – 132.8 °C.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.55 – 7.50 (m, 4H), 7.45 (d, *J* = 7.5 Hz, 2H), 7.42 – 7.37 (m, 4H), 7.34 – 7.27 (m, 2H), 7.06 – 7.03 (m, 2H), 5.09 (s, 2H) ppm.

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 158.5, 140.9, 137.1, 134.1, 128.9, 128.7, 128.3, 128.1, 127.6, 126.9, 126.8, 115.3, 70.2 ppm.

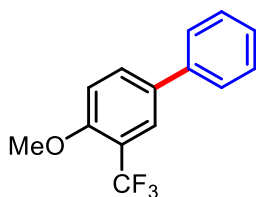


#### Methyl [1,1'-biphenyl]-4-carboxylate<sup>[20]</sup>, **3y**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3y** as a white solid (18.2 mg, 0.09 mmol, 29% yield). **m.p.** 111.6 – 113.1 °C.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.11 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.63 (d, *J* = 7.5 Hz, 2H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.40 (t, *J* = 7.0 Hz, 1H), 3.94 (s, 3H) ppm.

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 167.1, 145.8, 140.1, 130.2, 129.1, 129.0, 128.3, 127.4, 127.2, 52.3 ppm.



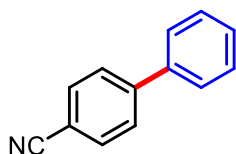
#### 4-Methoxy-3-(trifluoromethyl)-1,1'-biphenyl<sup>[21]</sup>, **3z**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3z** as a white solid (31.0 mg, 0.12 mmol, 41% yield). **m.p.** 54.4 – 56.1 °C.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.79 (d, *J* = 2.5 Hz, 1H), 7.70 (dd, *J* = 9.0, 2.5 Hz, 1H), 7.54 (d, *J* = 7.5 Hz, 2H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.34 (t, *J* = 7.0 Hz, 1H), 7.07 (d, *J* = 9.0 Hz, 1H), 3.94 (s, 3H) ppm.

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 157.0, 139.7, 133.5, 131.8, 129.1, 127.5, 126.9, 125.9 (q, *J* = 5.3 Hz), 123.8 (q, *J* = 272.9 Hz), 119.2 (q, *J* = 30.6 Hz), 112.5, 56.2 ppm.

<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>): δ -62.4 (s) ppm.

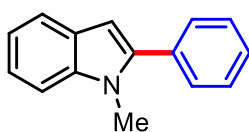


#### [1,1'-Biphenyl]-4-carbonitrile<sup>[22]</sup>, **3aa**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3aa** as a white solid (21.4 mg, 0.12 mmol, 40% yield). **m.p.** 86.6 – 88 °C.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.72 (d, *J* = 8.5 Hz, 2H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.58 (d, *J* = 7.0 Hz, 2H), 7.48 (t, *J* = 7.0 Hz, 2H), 7.42 (t, *J* = 7.0 Hz, 1H) ppm.

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 145.8, 139.3, 132.7, 129.2, 128.8, 127.8, 127.3, 119.1, 111.0 ppm.

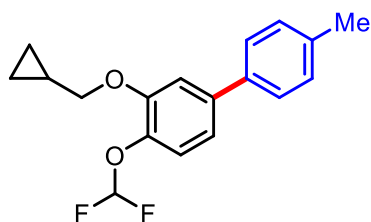


#### 1-Methyl-2-phenyl-1H-indole<sup>[23]</sup>, **3ab**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3ab** as a white solid (31.7 mg, 0.15 mmol, 51% yield). **m.p.** 97.5 – 99.3 °C.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.63 (d, *J* = 7.5 Hz, 1H), 7.52 – 7.49 (m, 2H), 7.48 – 7.44 (m, 2H), 7.41 – 7.37 (m, 1H), 7.36 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.26 – 7.23 (m, 1H), 7.16 – 7.12 (m, 1H), 6.56 (d, *J* = 0.5 Hz, 1H), 3.74 (s, 3H) ppm.

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 141.7, 138.5, 133.0, 129.5, 128.6, 128.1, 128.0, 121.8, 120.6, 120.0, 109.7, 101.8, 31.3 ppm.



### 3-(Cyclopropylmethoxy)-4-(difluoromethoxy)-4'-methyl-1,1'-biphenyl, **3ac**

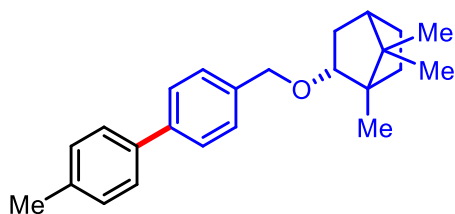
This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3ac** as a colorless oil (48.0 mg, 0.16 mmol, 53% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.43 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.5 Hz, 1H), 7.12 – 7.09 (m, 2H), 6.65 (t, *J* = 75.5 Hz, 1H), 3.93 (d, *J* = 7.0 Hz, 2H), 2.39 (s, 3H), 1.34 – 1.30 (m, 1H), 0.65 (q, *J* = 6.0 Hz, 2H), 0.37 (q, *J* = 5.0 Hz, 2H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 150.7, 140.2, 139.7, 137.6, 137.5, 129.7, 127.0, 123.0, 119.8, 116.5 (t, *J* = 259.8 Hz), 113.4, 74.1, 21.2, 10.3, 3.4 ppm.

**<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>):** δ -81.4 (d, *J* = 75.8 Hz) ppm.

**HRMS (ESI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>18</sub>H<sub>19</sub>F<sub>2</sub>O<sub>2</sub><sup>+</sup> 305.1348, found 305.1342.



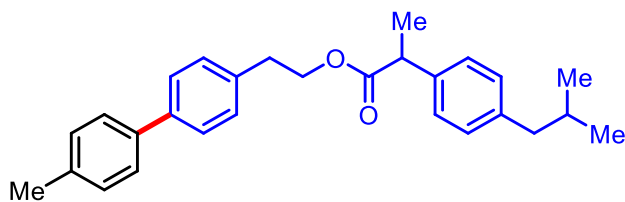
### (2R)-1,7,7-Trimethyl-2-((4'-methyl-[1,1'-biphenyl]-4-yl)methoxy)bicyclo[2.2.1]heptane, **3ad**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3ad** as a white solid (48.0 mg, 0.14 mmol, 48% yield). **m.p.** 56.0 – 57.7 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.54 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.5 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 4.60 (d, *J* = 12.0 Hz, 1H), 4.48 (d, *J* = 12.0 Hz, 1H), 3.74 – 3.71 (m, 1H), 2.39 (s, 3H), 2.18 – 2.09 (m, 2H), 1.76 – 1.68 (m, 1H), 1.66 (t, *J* = 4.5 Hz, 1H), 1.28 – 1.25 (m, 2H), 1.12 (dd, *J* = 13.0, 3.5 Hz, 1H), 0.92 (s, 3H), 0.86 (s, 3H), 0.84 (s, 3H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 140.2, 138.5, 138.3, 137.0, 129.6, 127.7, 127.0, 126.9, 84.5, 71.5, 49.5, 48.0, 45.2, 36.3, 28.4, 26.9, 21.2, 20.0, 19.1, 14.2 ppm.

**HRMS (APCI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>24</sub>H<sub>31</sub>O<sup>+</sup> 335.2369, found 335.2357.



### 2-(4'-Methyl-[1,1'-biphenyl]-4-yl)ethyl 2-(4-isobutylphenyl)propanoate, **3ae**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3ae** as white solid

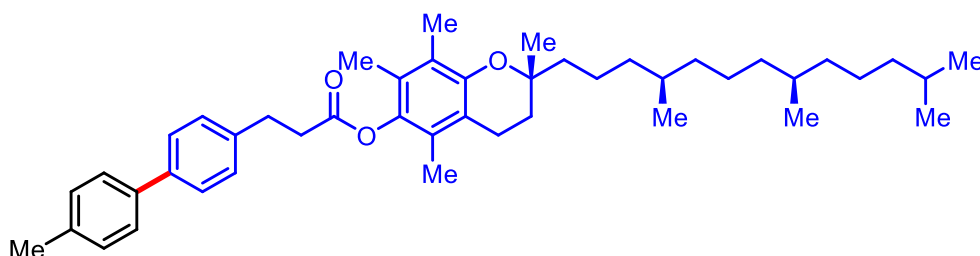


(66.0 mg, 0.17 mmol, 55% yield). **m.p.** 39.7 – 41.8 °C.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.46 (t, *J* = 8.0 Hz, 4H), 7.24 (d, *J* = 7.5 Hz, 2H), 7.15 (t, *J* = 8.0 Hz, 4H), 7.07 (d, *J* = 7.5 Hz, 2H), 4.35 – 4.25 (m, 2H), 3.68 (q, *J* = 7.0 Hz, 1H), 2.93 – 2.87 (m, 2H), 2.43 (d, *J* = 7.0 Hz, 2H), 2.39 (s, 3H), 1.86 – 1.78 (m, 1H), 1.47 (d, *J* = 7.0 Hz, 3H), 0.88 (d, *J* = 6.5 Hz, 6H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 174.8, 140.6, 139.4, 138.1, 137.8, 137.0, 136.7, 129.6, 129.4, 127.3, 127.0, 126.9, 65.3, 45.3, 45.1, 34.8, 30.3, 22.5, 21.2, 18.6 ppm.

**HRMS (ESI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>28</sub>H<sub>33</sub>O<sub>2</sub><sup>+</sup> 401.2475, found 401.2477.



**(*R*)-2,5,7,8-Tetramethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl 3-(4'-methyl-[1,1'-biphenyl]-4-yl)propanoate, 3af**

This compound was prepared according to the general procedure. Purification by column chromatography on silica gel (Petroleum ether/Dichloromethane = 6/1, v/v) afforded **3af** as a colorless oil (105.5 mg, 0.16 mmol, 54% yield).

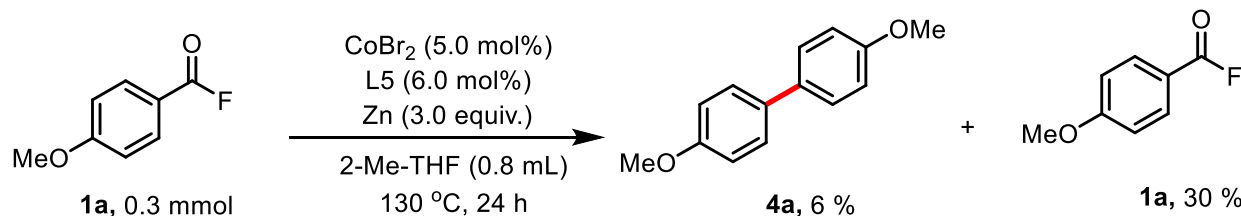
**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):** δ 7.52 (dd, *J* = 8.0, 2.0 Hz, 2H), 7.48 (dd, *J* = 8.0, 2.5 Hz, 2H), 7.33 (dd, *J* = 8.0, 2.0 Hz, 2H), 7.24 (dd, *J* = 8.0, 2.0 Hz, 2H), 3.13 (td, *J* = 7.5, 2.5 Hz, 2H), 2.96 (td, *J* = 8.0, 2.5 Hz, 2H), 2.55 (t, *J* = 7.0 Hz, 2H), 2.39 (s, 3H), 2.07 (s, 3H), 1.92 (s, 3H), 1.86 (s, 3H), 1.82 – 1.71 (m, 2H), 1.57 – 1.50 (m, 3H), 1.39 – 1.34 (m, 4H), 1.30 – 1.22 (m, 11H), 1.16 – 1.11 (m, 3H), 1.10 – 1.04 (m, 3H), 0.88 – 0.84 (m, 12H) ppm.

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):** δ 171.7, 149.5, 140.5, 139.4, 139.2, 138.2, 137.0, 129.6, 129.0, 127.2, 126.9, 126.7, 125.0, 123.1, 117.4, 75.1, 39.5, 37.6, 37.5, 37.4, 35.7, 32.9, 32.8, 30.8, 28.1, 24.9, 24.6, 22.9, 22.8, 21.2, 21.1, 20.7, 19.9, 19.8, 13.0, 12.2, 12.0 ppm.

**HRMS (APCI<sup>+</sup>):** *m/z*: [M+H]<sup>+</sup> calcd for C<sub>45</sub>H<sub>65</sub>O<sub>3</sub><sup>+</sup> 653.4928, found 653.4916.

## 4. Mechanistic Studies

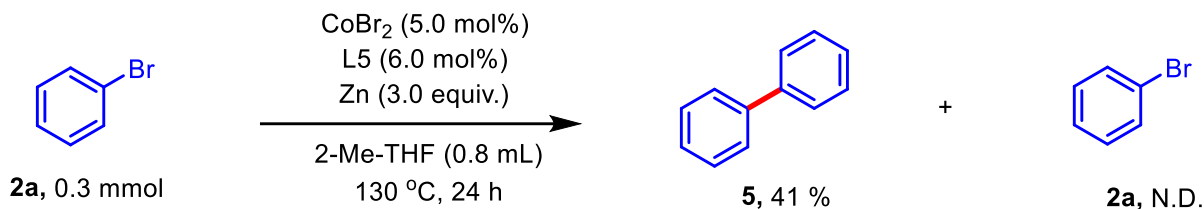
### 4.1 The reaction of aroyl fluoride **1a** under cobalt catalysis.



An oven-dried Schlenk tube (20 mL) was charged with CoBr<sub>2</sub> (0.015 mmol, 3.3 mg, 5.0 mol%), dimethyl [2,2'-bipyridine]-4,4'-dicarboxylate (0.018 mmol, 4.9 mg, 6.0 mol%), Zn dust (0.9 mmol, 58.9 mg, 3.0 equiv.), 4-methoxybenzoyl fluoride (0.3 mmol, 46.2 mg, 1.0 equiv.) and 2-Me-THF (0.8 mL). The reaction mixture was allowed to stir under N<sub>2</sub> atmosphere at 130 °C (oil bath) for 24 h. After

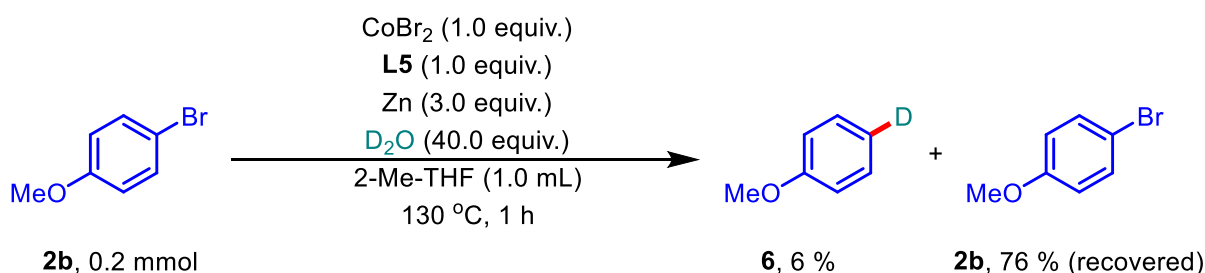
this time, the tube was cooled to room temperature. The residue was diluted with ethyl acetate (10 mL). The mixture was detected by GC-MS analysis. The yields of products were confirmed by GC using 1,3,5-trimethoxybenzene as an internal standard.

#### 4.2 The reaction of bromobenzene under cobalt catalysis.



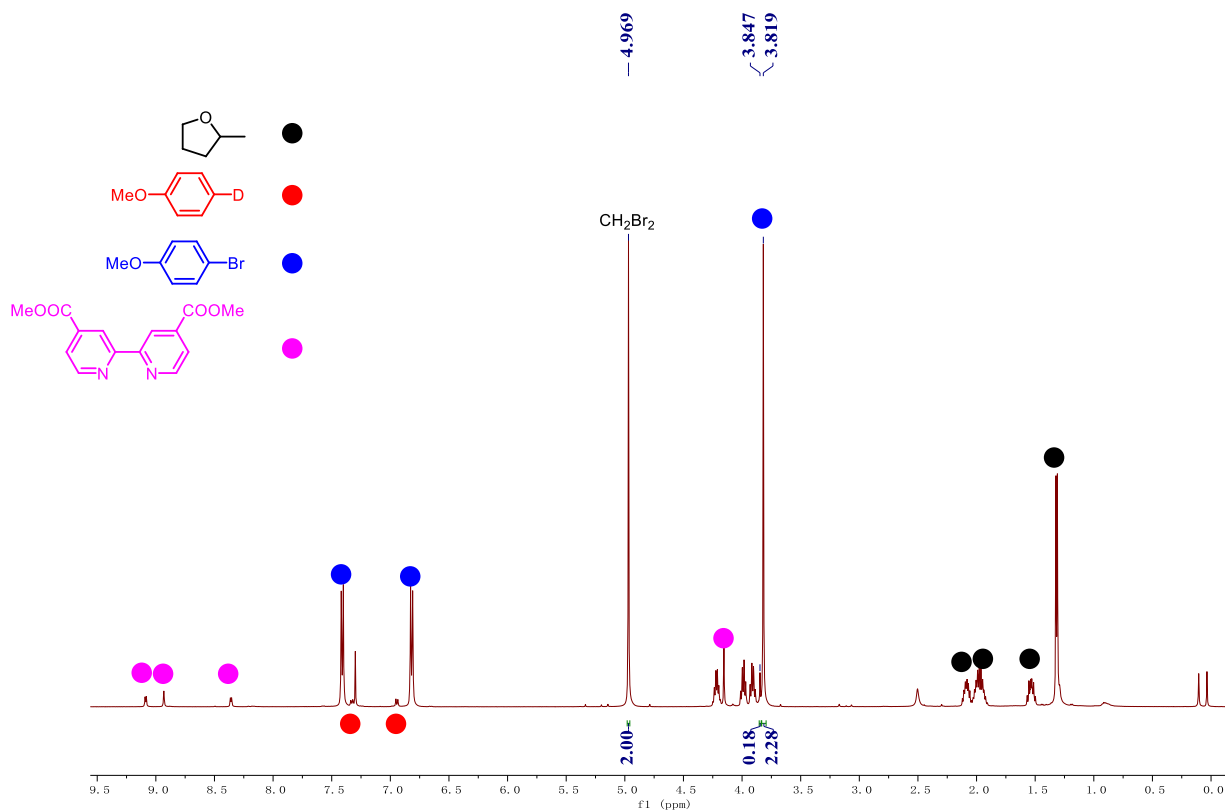
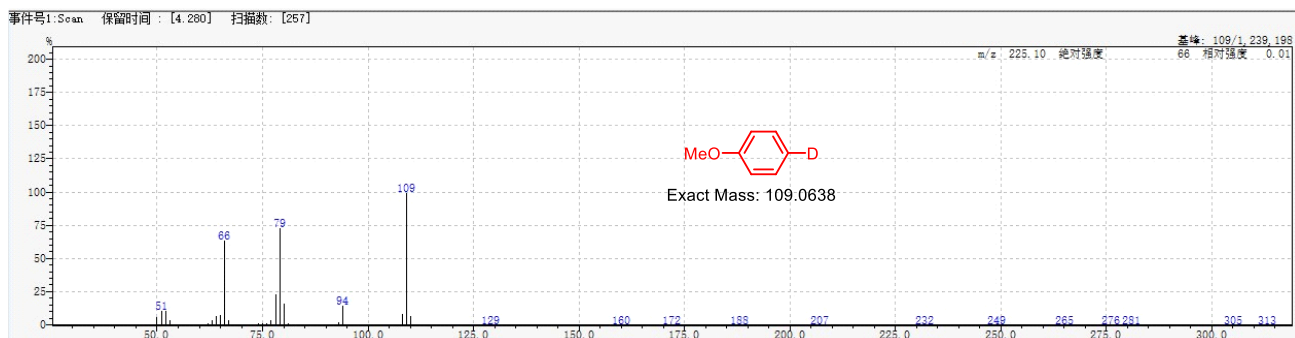
An oven-dried Schlenk tube (20 mL) was charged with CoBr<sub>2</sub> (0.015 mmol, 3.3 mg, 5.0 mol%), dimethyl [2,2'-bipyridine]-4,4'-dicarboxylate (0.018 mmol, 4.9 mg, 6.0 mol%), Zn dust (0.9 mmol, 58.9 mg, 3.0 equiv.), bromobenzene (0.3 mmol, 46.8 mg 1.0 equiv.) and 2-Me-THF (0.8 mL). The reaction mixture was allowed to stir under N<sub>2</sub> atmosphere at 130 °C (oil bath) for 24 h. After this time, the tube was cooled to room temperature. The residue was diluted with ethyl acetate (10 mL). The mixture was detected by GC-MS analysis. The yields of product **5** was confirmed by GC using 1,3,5-trimethoxybenzene as an internal standard.

#### 4.3 Deuterium-labelling experiment.



An oven-dried Schlenk tube (20 mL) was charged with CoBr<sub>2</sub> (0.2 mmol, 43.7 mg), L5 (0.2 mmol, 54.4 mg), Zn dust (0.6 mmol, 39.0 mg), 1-bromo-4-methoxybenzene (0.2 mmol, 37.2 mg), D<sub>2</sub>O (8 mmol, 144 μL) and 2-Me-THF (1.0 mL). The reaction mixture was allowed to stir under N<sub>2</sub> atmosphere at 130 °C (oil bath) for 1 h. After this time, the tube was cooled to room temperature. The residue was diluted with ethyl acetate (10 mL). The mixture was detected by GC-MS analysis. The yields of product **5** and recovered **2b** was confirmed by NMR using dibromomethane as an internal standard.





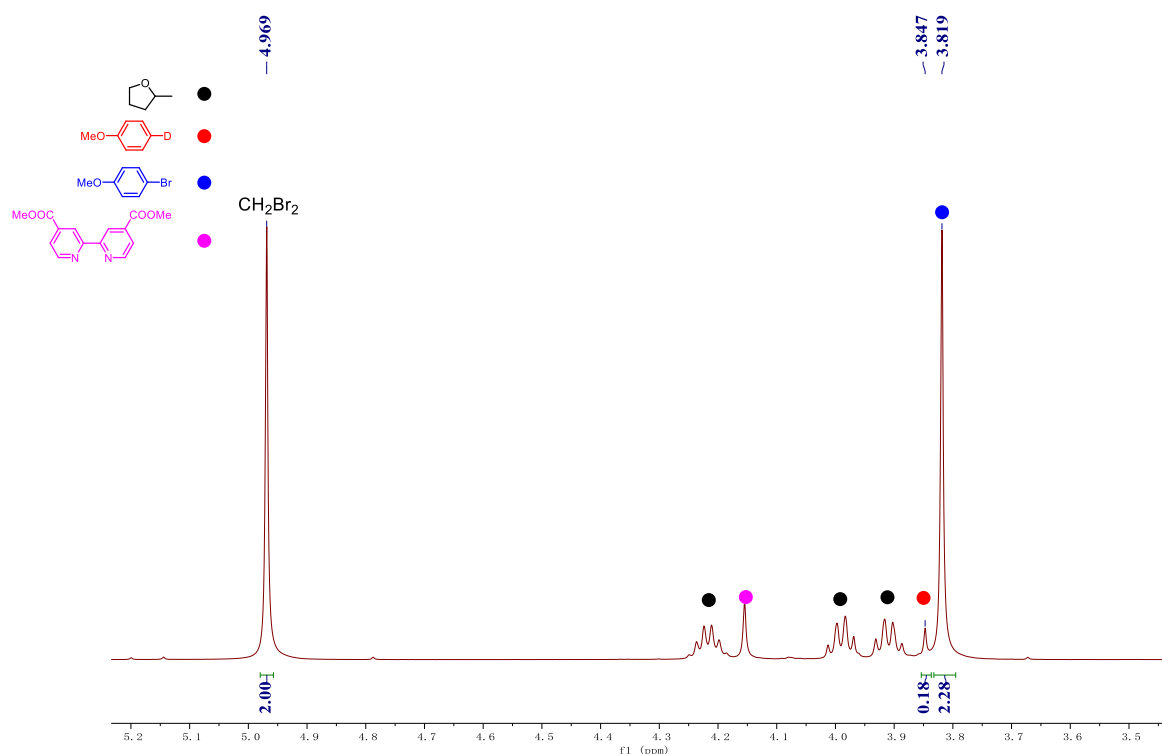
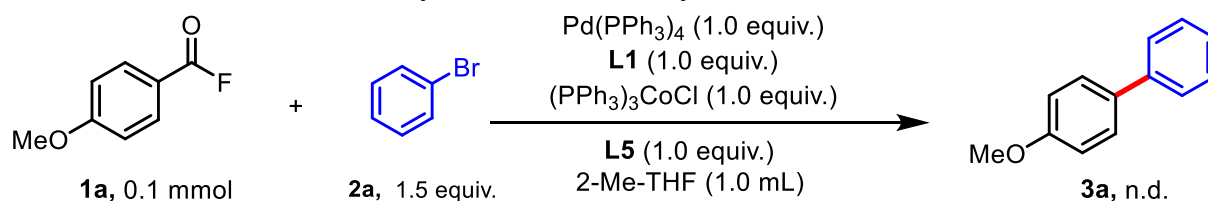


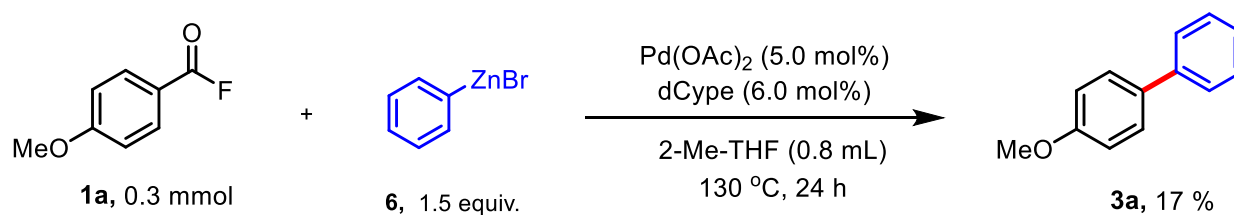
Figure S1. <sup>1</sup>H NMR and GC-MS spectra for anisole-4-*d* (**6**)

#### 4.4 Stoichiometric reaction of aroyl fluorides with aryl bromides.



An oven-dried Schlenk tube (20 mL) was charged with Pd(PPh<sub>3</sub>)<sub>4</sub> (0.1 mmol, 115.6 mg), **L1** (0.1 mmol, 42.3 mg), (PPh<sub>3</sub>)<sub>3</sub>CoCl (0.1 mmol, 88.1 mg), **L5** (0.1 mmol, 27.3 mg), 4-methoxybenzoyl fluoride (0.1 mmol, 15.4 mg), bromobenzene (0.15 mmol, 23.4 mg) and 2-Me-THF (1.0 mL). The reaction mixture was allowed to stir under N<sub>2</sub> atmosphere at 130 °C (oil bath) for 24 h. After this time, the tube was cooled to room temperature. The residue was diluted with ethyl acetate (10 mL). The mixture was detected by GC-MS analysis.

#### 4.5 The reaction of aroyl fluoride **1a** with arylzinc reagent under palladium catalysis.

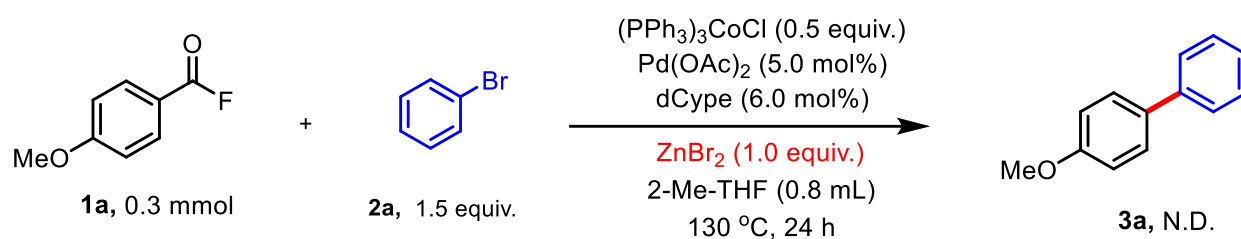


A dried Schlenk tube (20 mL) equipped with a stir bar was charged with a solution of bromobenzene (0.5 mmol, 93.5 mg) in THF (0.2 mL). The reaction mixture was cooled to -78 °C and *n*-BuLi (0.2 mL, 0.5 mmol, 2.5 M in hexane) was added dropwise over 5 min. A precipitate formed immediately, the

reaction mixture was stirred for 60 min. After the indicated time, ZnBr<sub>2</sub> (0.5 mmol, 112.6 mg) was added in portions, the reaction mixture was allowed to warm to room temperature, and stirred for an additional 30 min at room temperature. The phenylzinc(II) bromide solution was used without further titration.

A dried Schlenk tube (20 mL) equipped with a stir bar was charged with 4-methoxybenzoyl fluoride (0.3 mmol, 46.2 mg), Pd(OAc)<sub>2</sub> (3.4 mg, 5.0 mol%), dCype (7.6 mg, 6.0 mol%) and 2-Me-THF (0.8 mL). Then the solution of phenylzinc(II) bromide in THF was added. The reaction was sealed and taken out of the glovebox. The resulting mixture was stirred at 130 °C (oil bath) for 24 h under N<sub>2</sub> atmosphere. After this time, the tube was cooled to room temperature. The reaction mixture was vacuumed to remove the volatiles. The residue was diluted with ethyl acetate (10 mL). The yield of product was confirmed by GC using 1,3,5-trimethoxybenzene as an internal standard.

#### 4.6 Palladium-catalyzed cross-coupling of aroyl fluoride **1a** with **2a** in the presence of (PPh<sub>3</sub>)<sub>3</sub>CoCl and ZnBr<sub>2</sub>.



A oven-dried Schlenk tube (20 mL) was charged with chlorotris(triphenylphosphine)cobalt (0.15 mmol, 132.2 mg, 0.5 equiv.), Pd(OAc)<sub>2</sub> (3.4 mg, 5.0 mol%), dCype (7.6 mg, 6.0 mol%), ZnBr<sub>2</sub> (0.3 mmol, 67.6 mg, 1.0 equiv.), 4-methoxybenzoyl fluoride (0.3 mmol, 46.2 mg, 1.0 equiv.), bromobenzene (0.45 mmol, 70.7 mg, 1.5 equiv.) and 2-Me-THF (0.8 mL). The reaction mixture was allowed to stir under N<sub>2</sub> atmosphere at 130 °C (oil bath) for 24 h. After this time, the tube was cooled to room temperature, The residue was diluted with ethyl acetate (10 mL). The mixture was detected by GC-MS analysis.

## 5. Reference

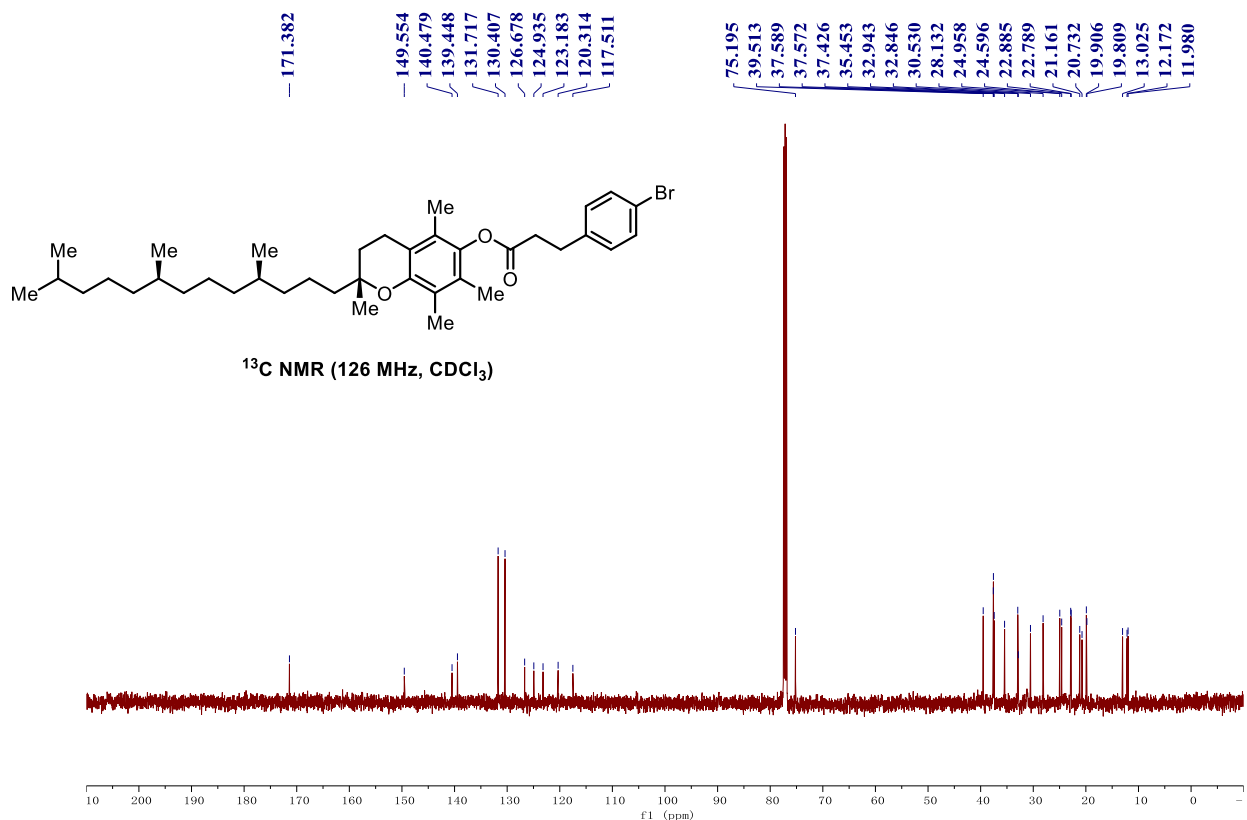
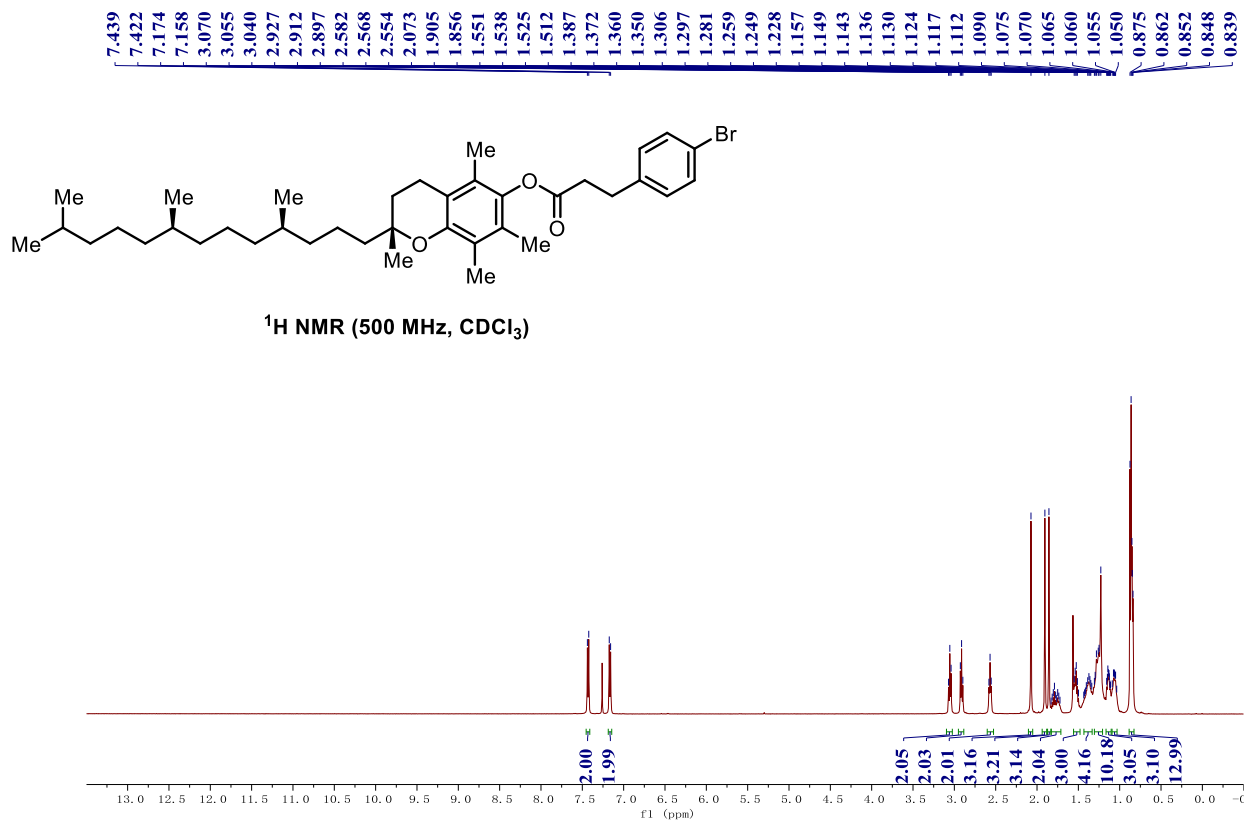
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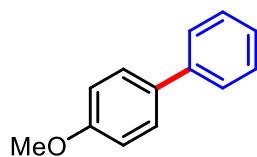
## 6. NMR Spectra

### (*R*)-2,5,7,8-Tetramethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl bromophenyl)propanoate, **2u**

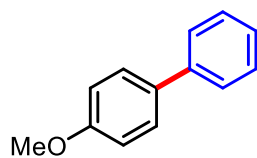
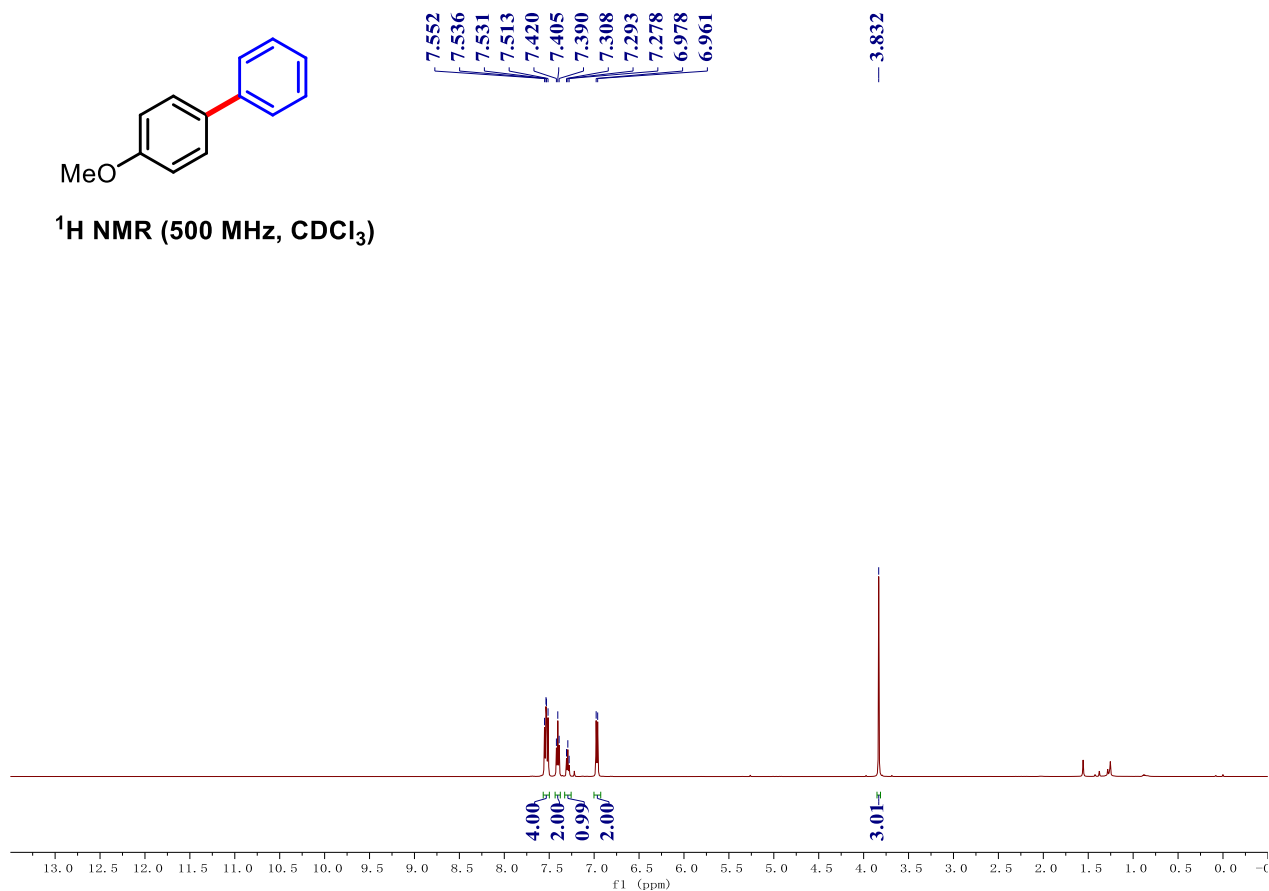
3-(4-



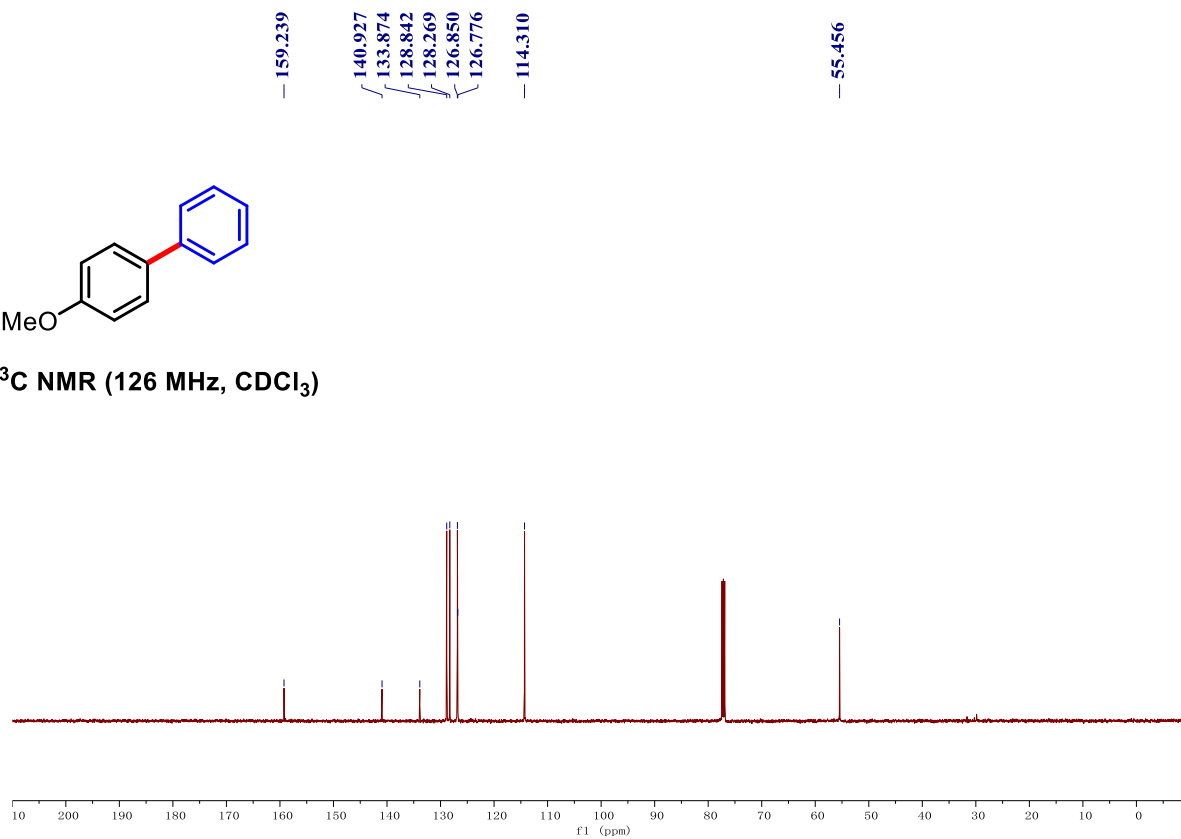
### 4-Methoxy-1,1'-biphenyl, 3a



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )

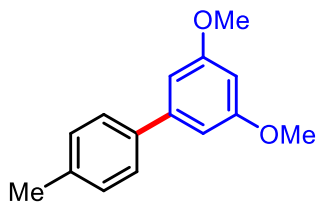


$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )

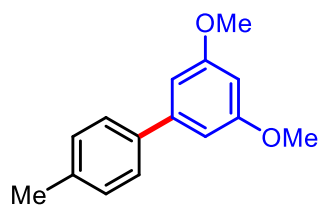
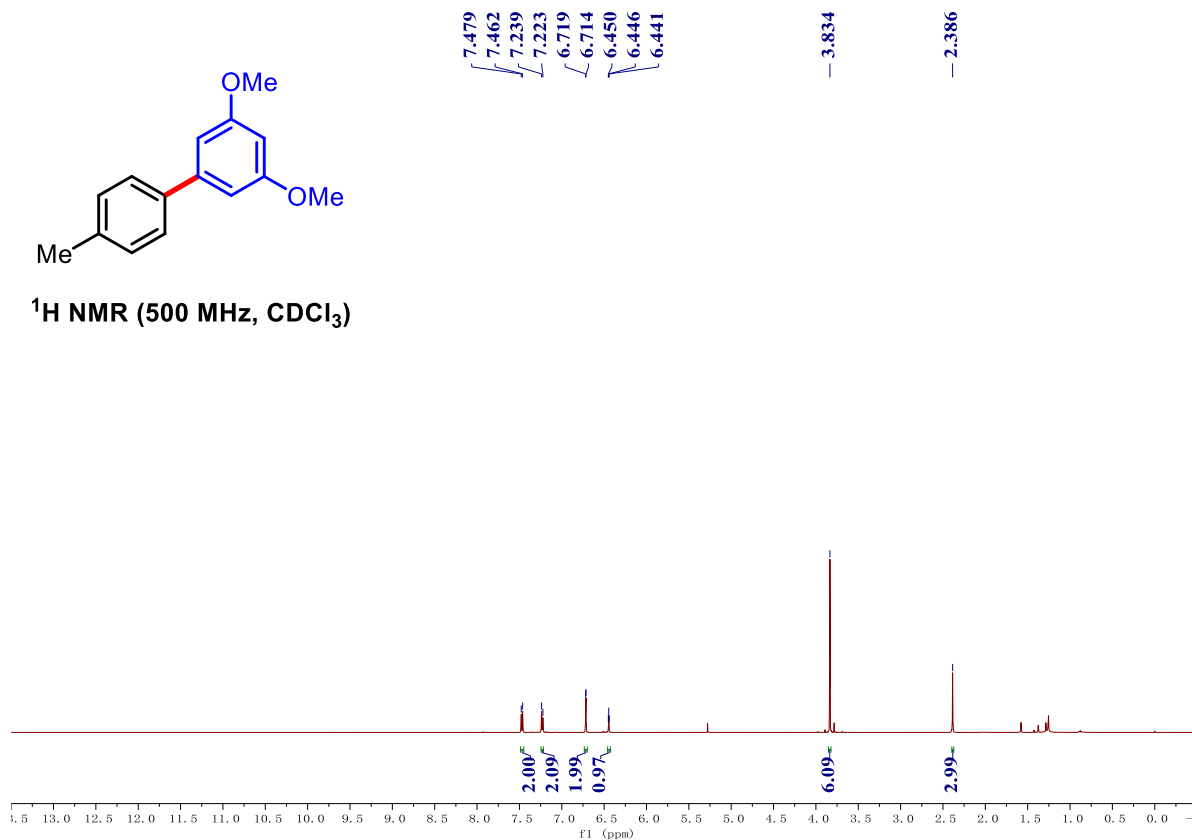




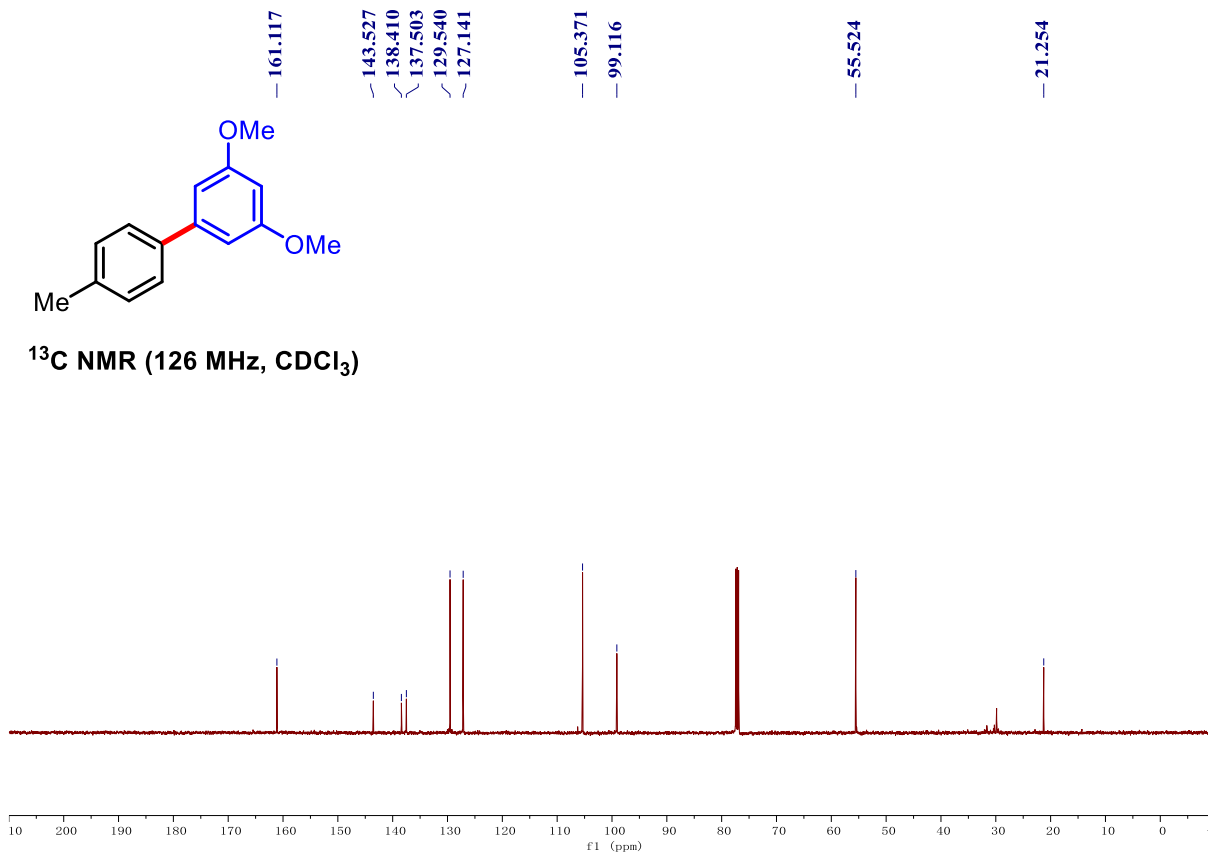
### 3,5-Dimethoxy-4'-methyl-1,1'-biphenyl, 3b



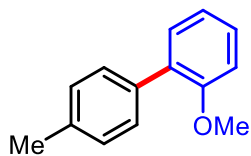
$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )



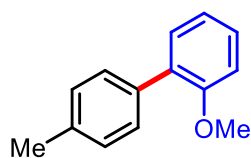
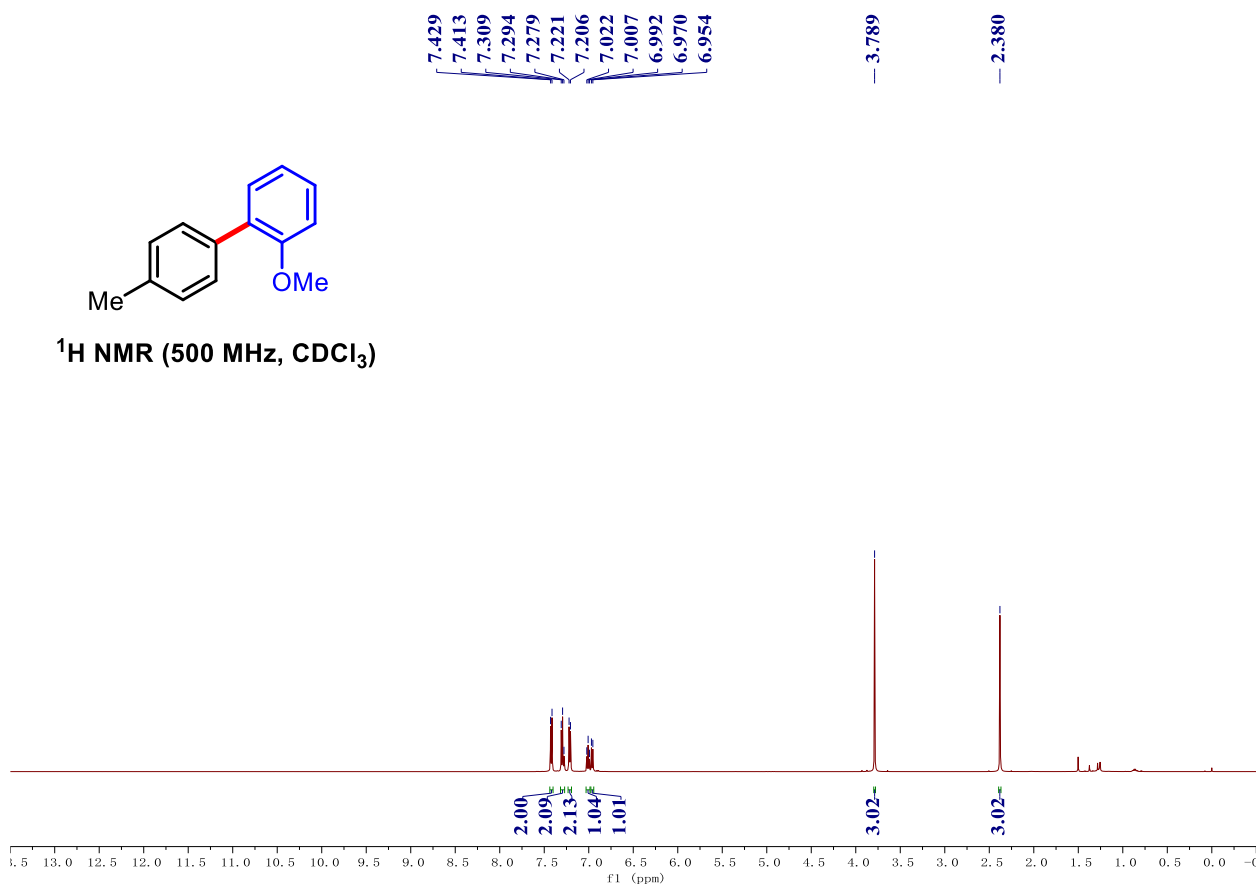
$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )



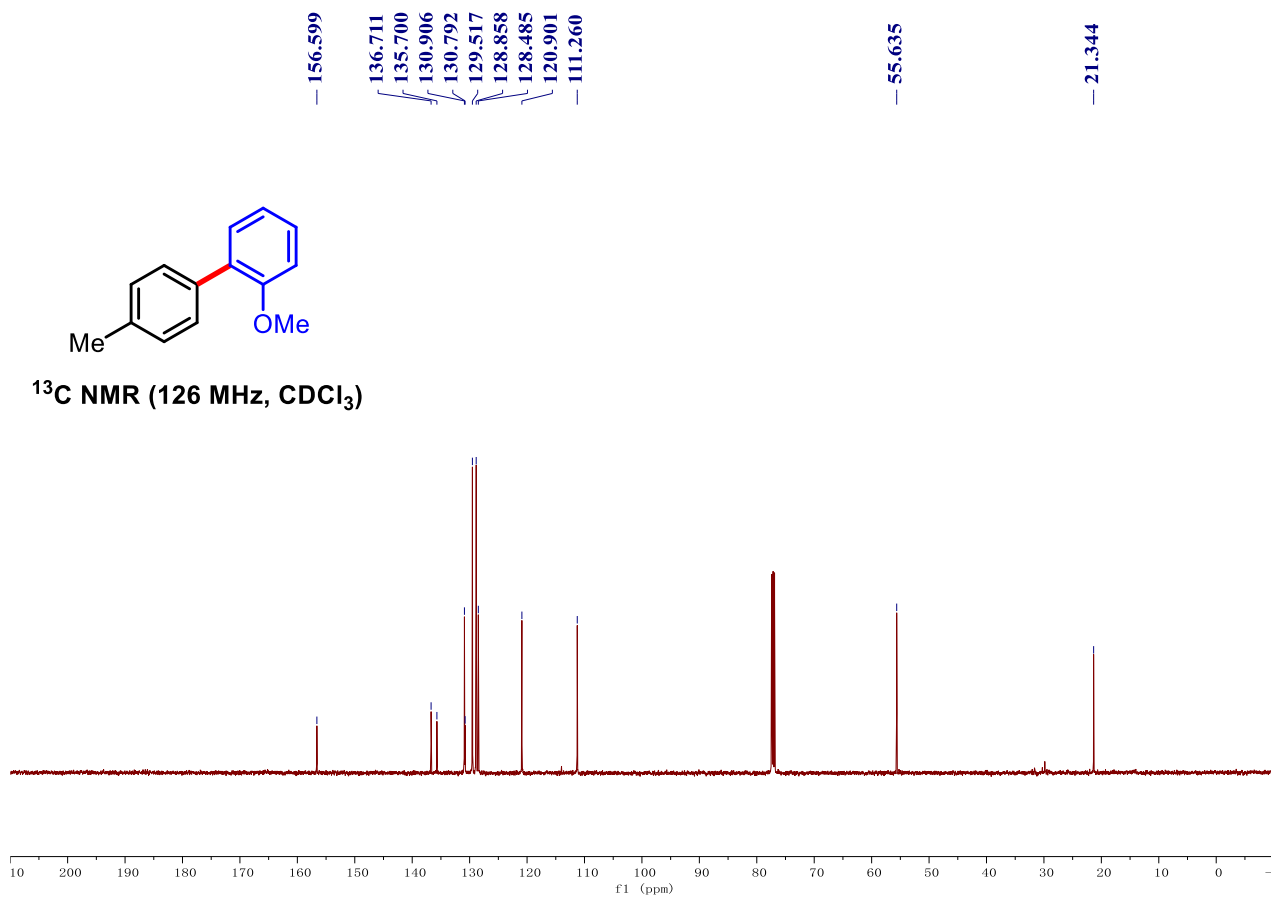
## 2-Methoxy-4'-methyl-1,1'-biphenyl, 3c



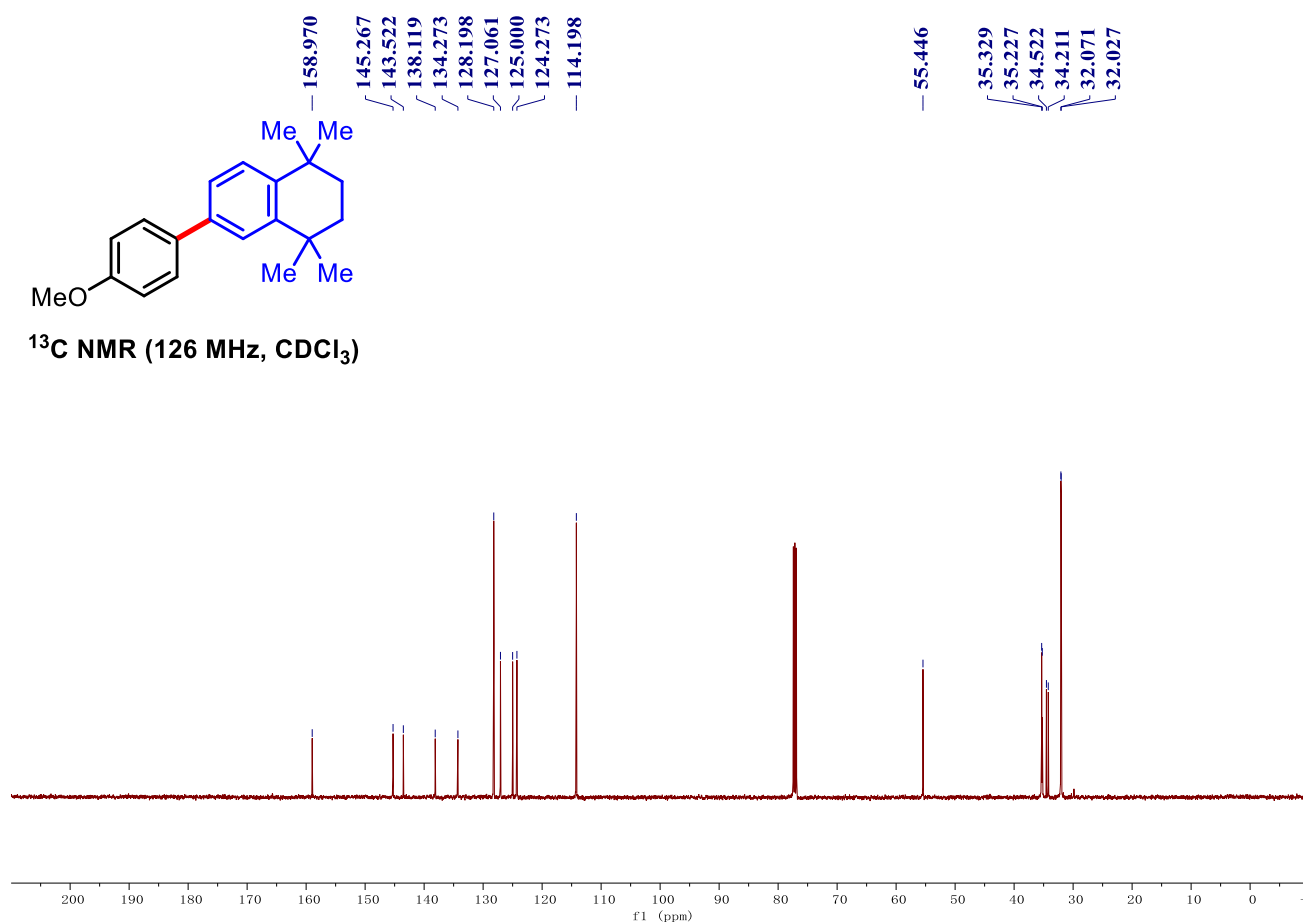
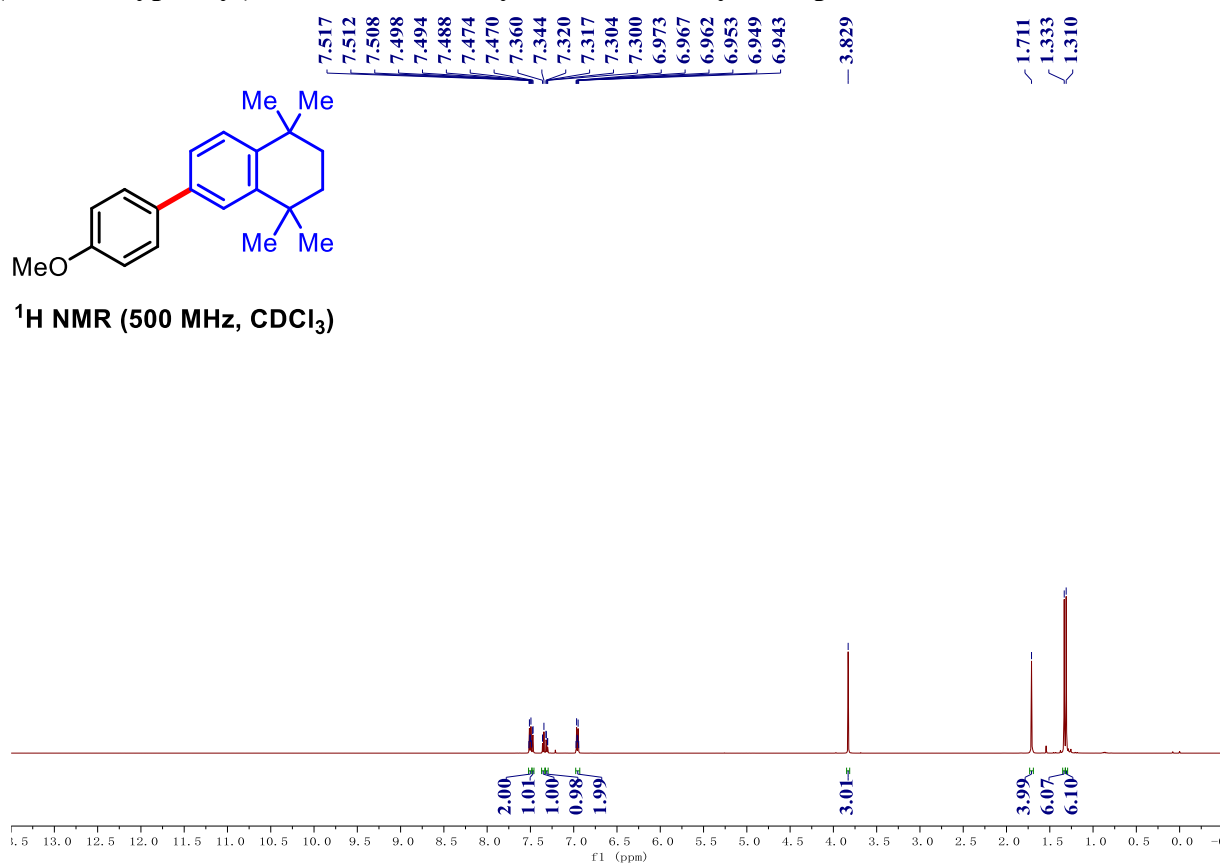
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )



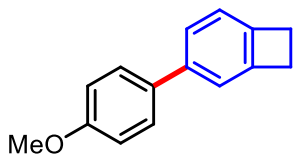
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )



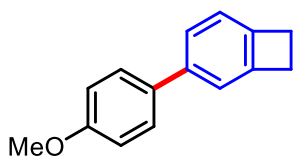
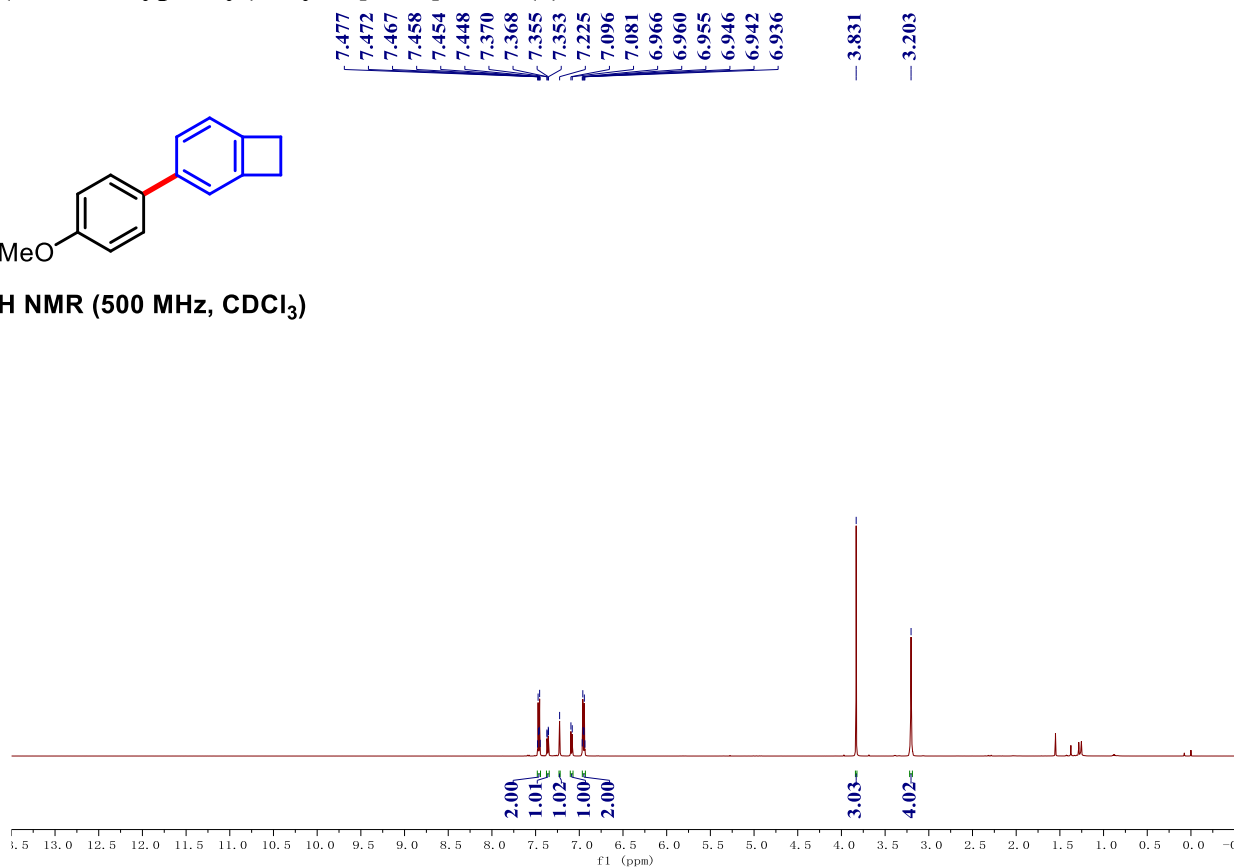
### 6-(4-Methoxyphenyl)-1,1,4,4-tetramethyl-1,2,3,4-tetrahydronaphthalene, 3d



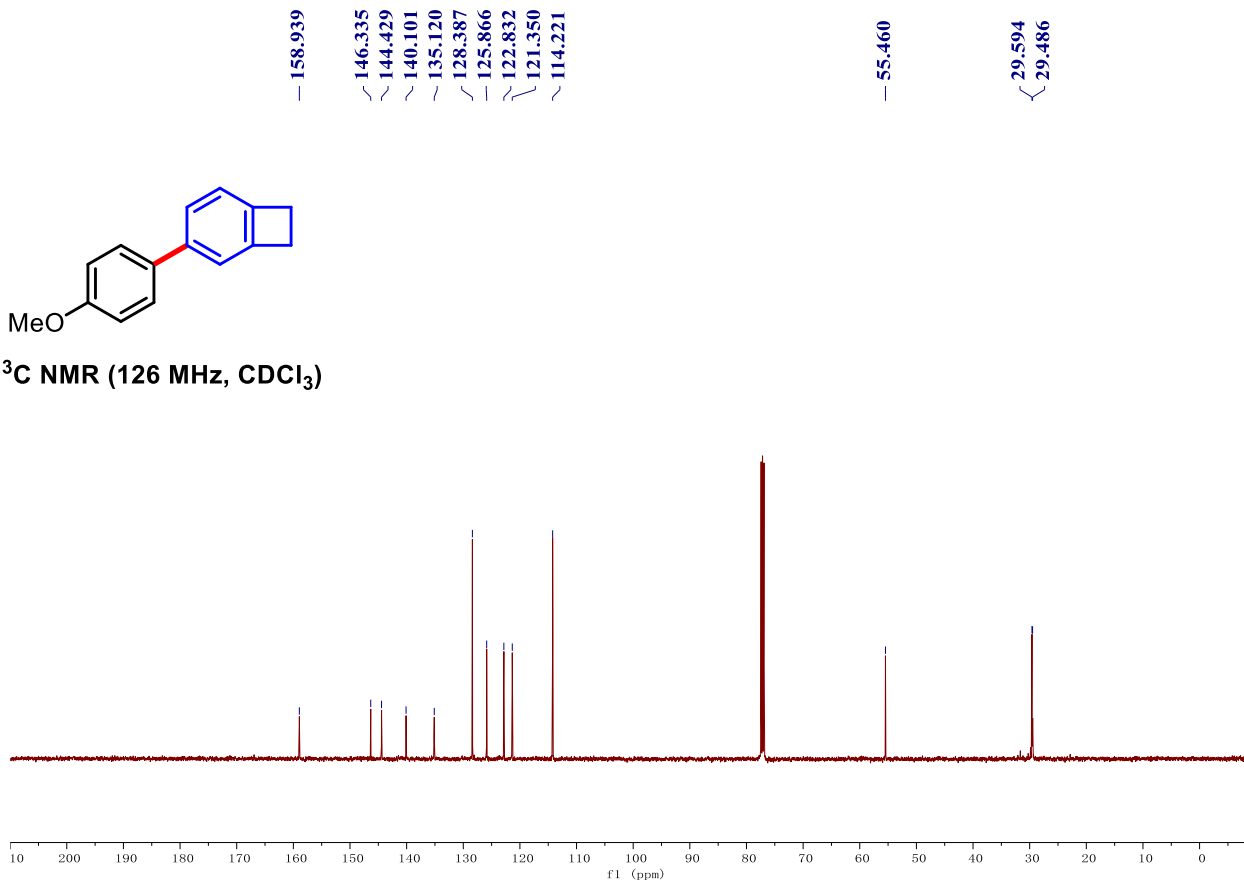
### 3-(4-Methoxyphenyl)bicyclo[4.2.0]octa-1(6),2,4-triene, 3e



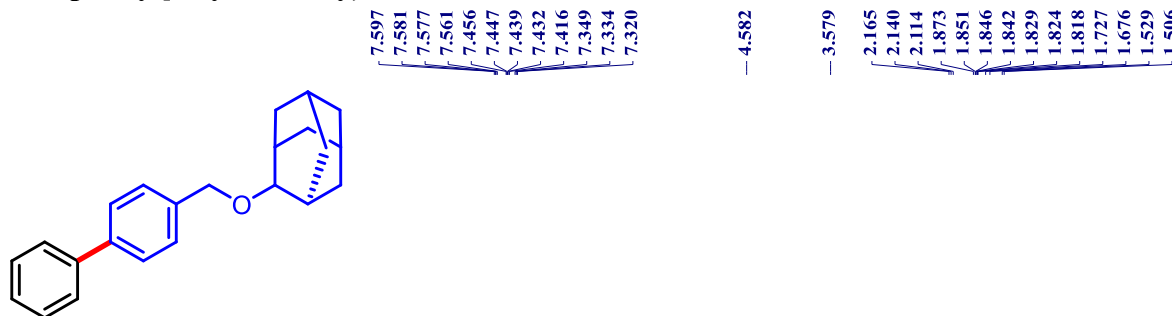
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



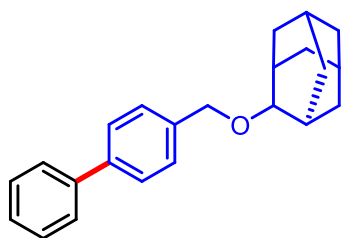
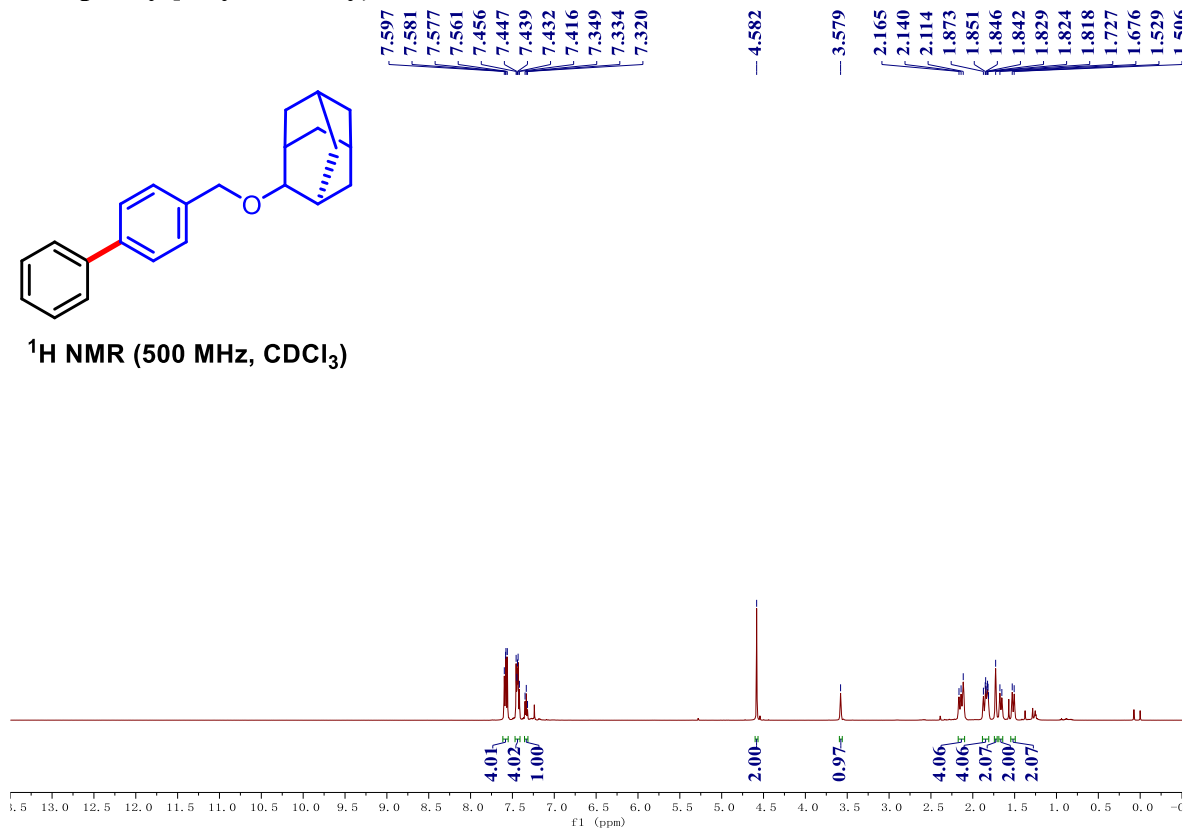
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)



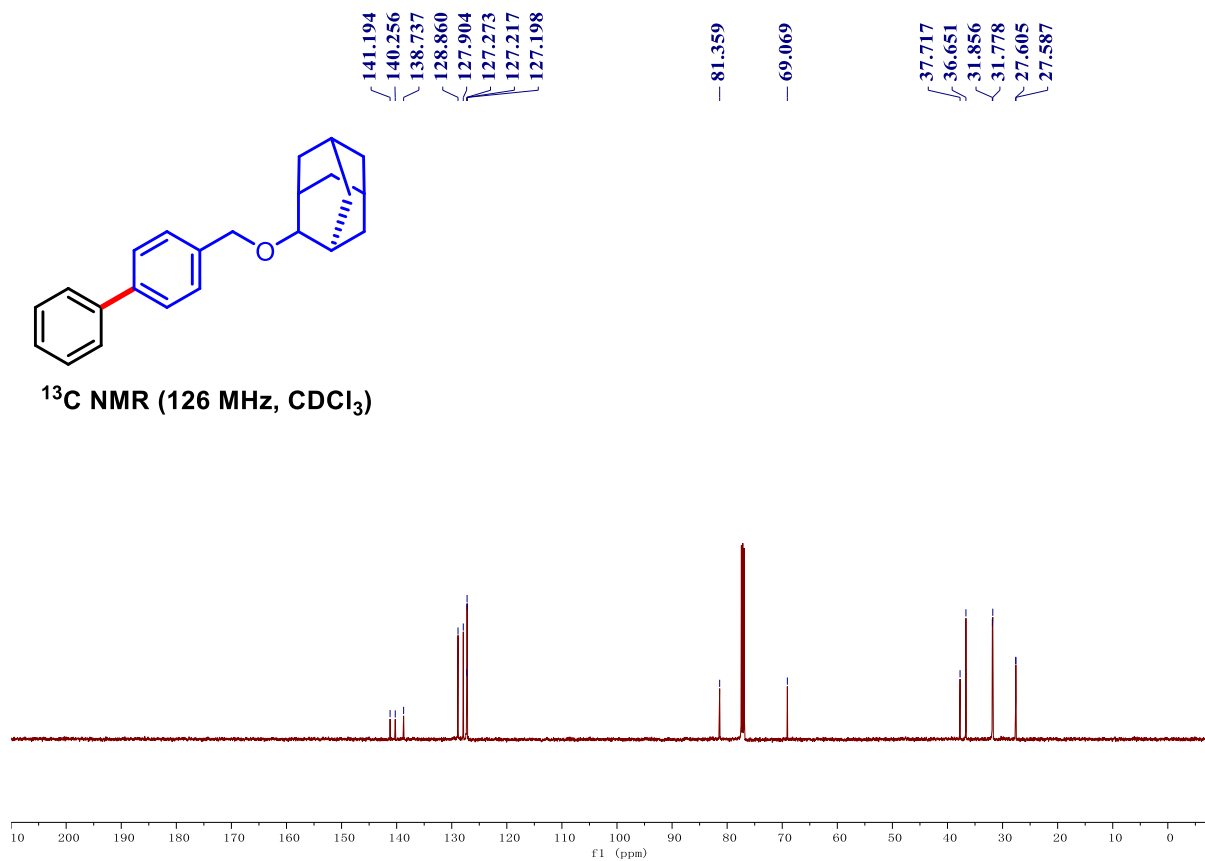
## 2-([1,1'-biphenyl]-4-ylmethoxy)adamantane, 3f



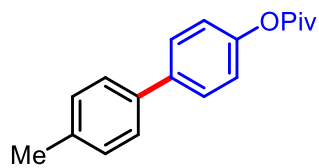
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



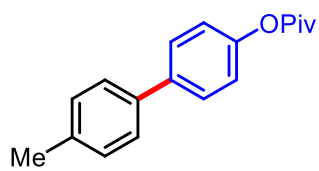
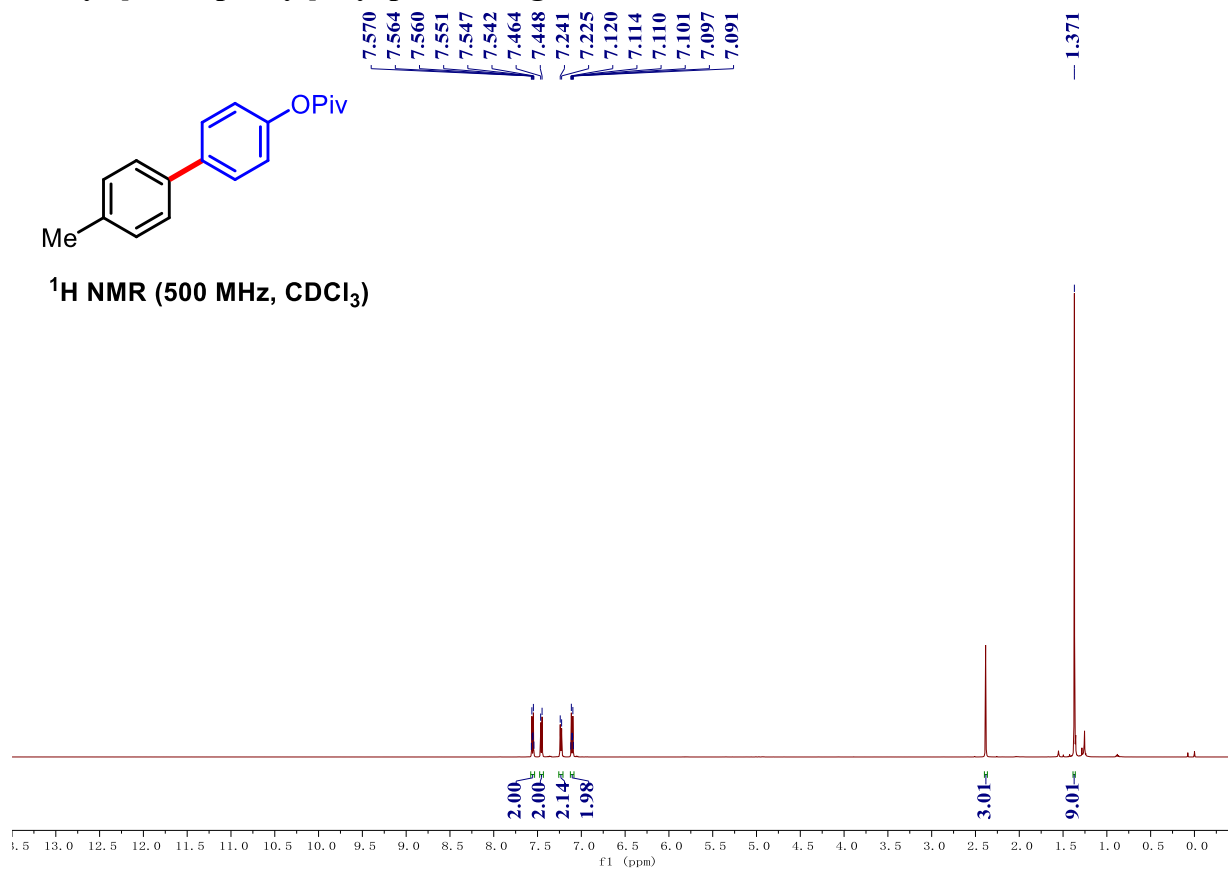
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)



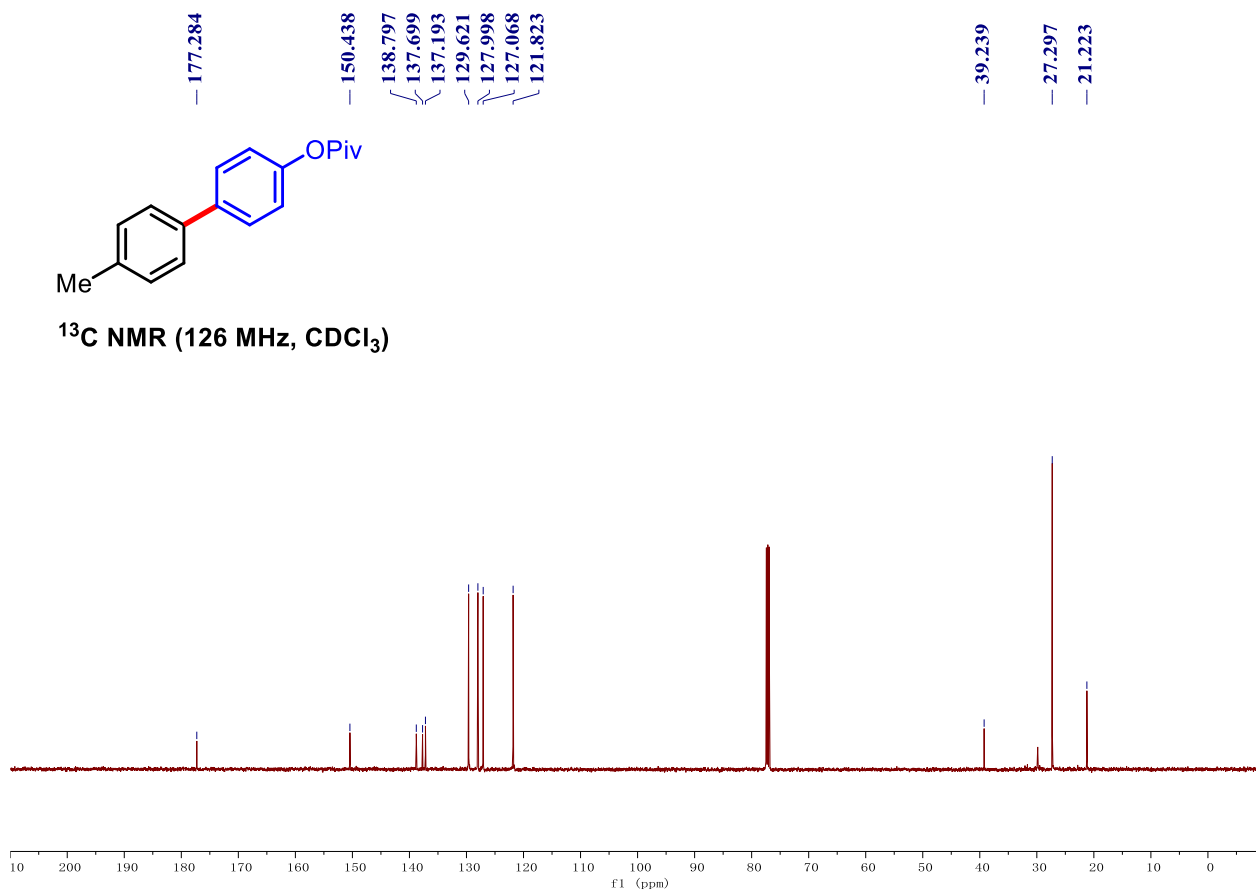
### 4'-Methyl-[1,1'-biphenyl]-4-yl pivalate, 3g



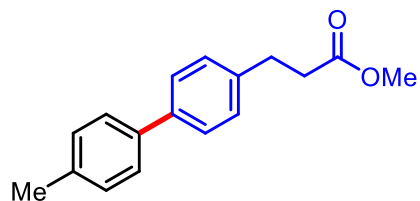
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



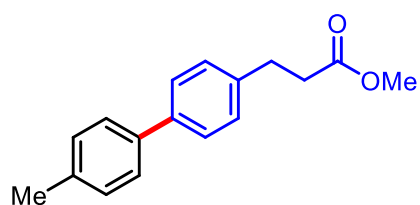
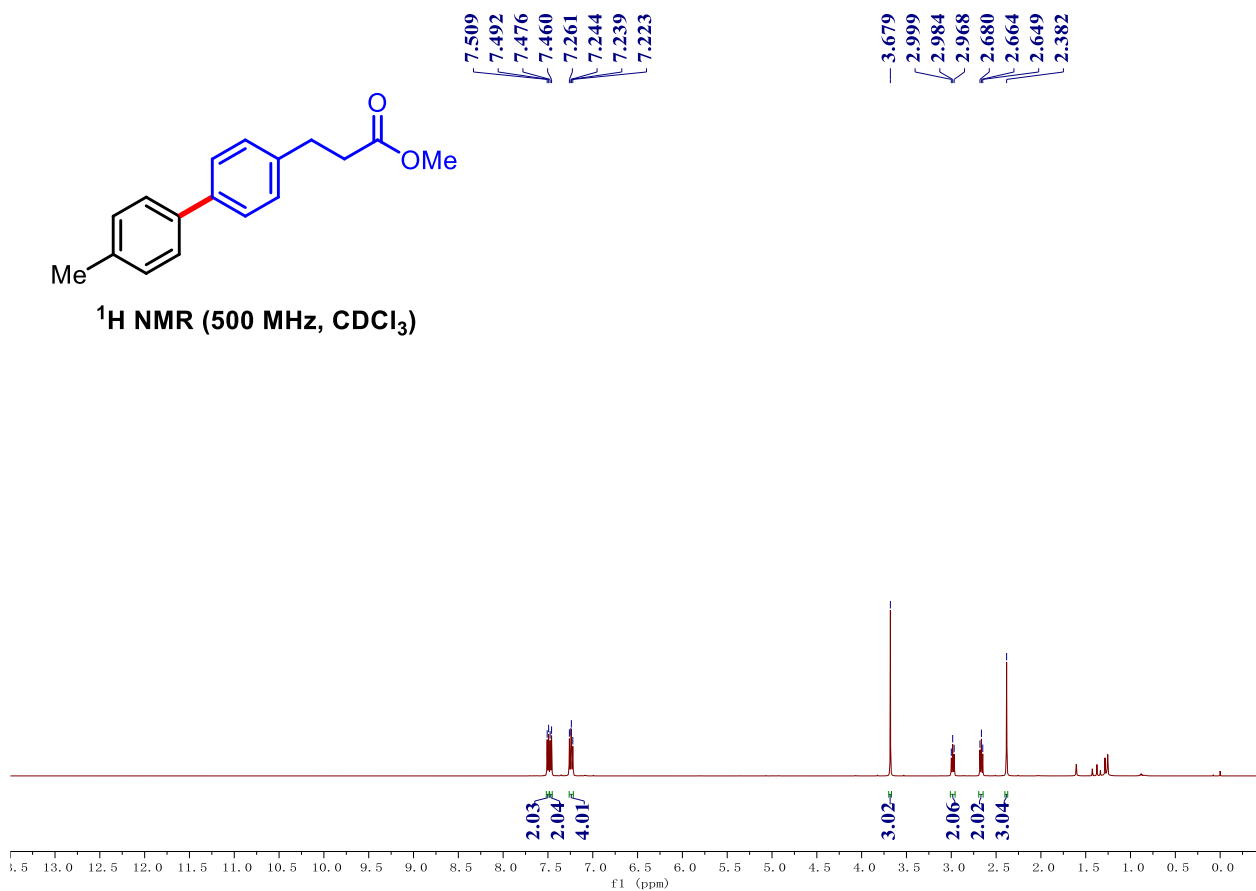
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)



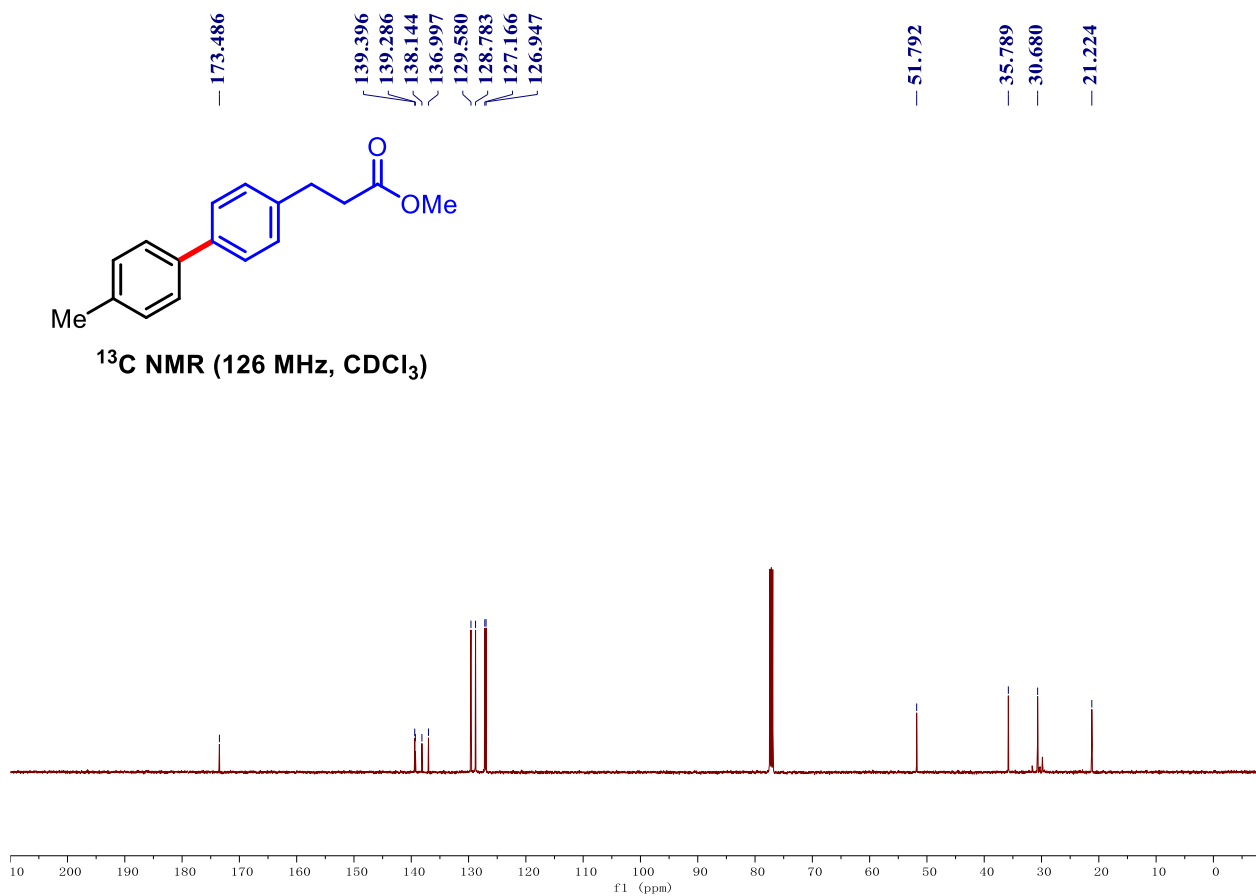
# Methyl 3-(4'-methyl-[1,1'-biphenyl]-4-yl)propanoate, 3h



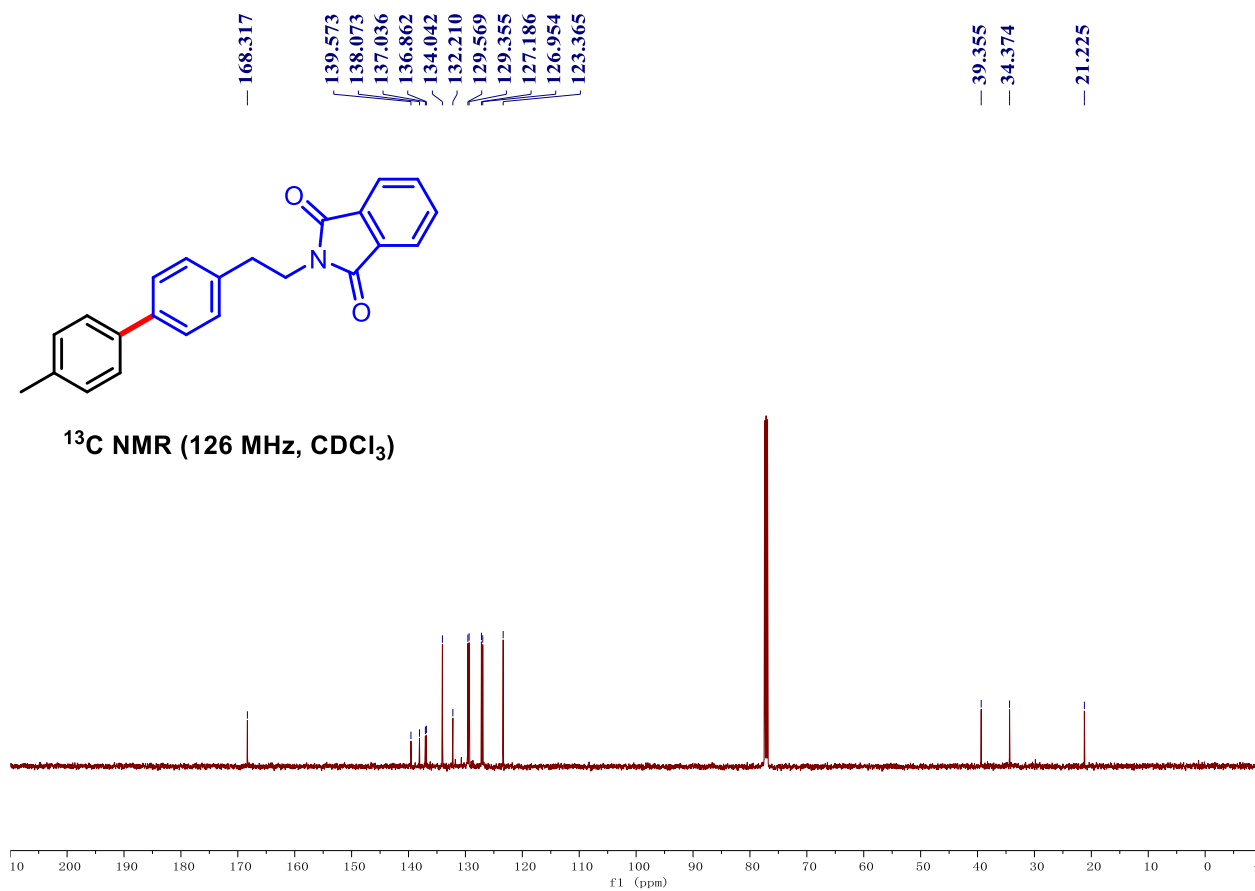
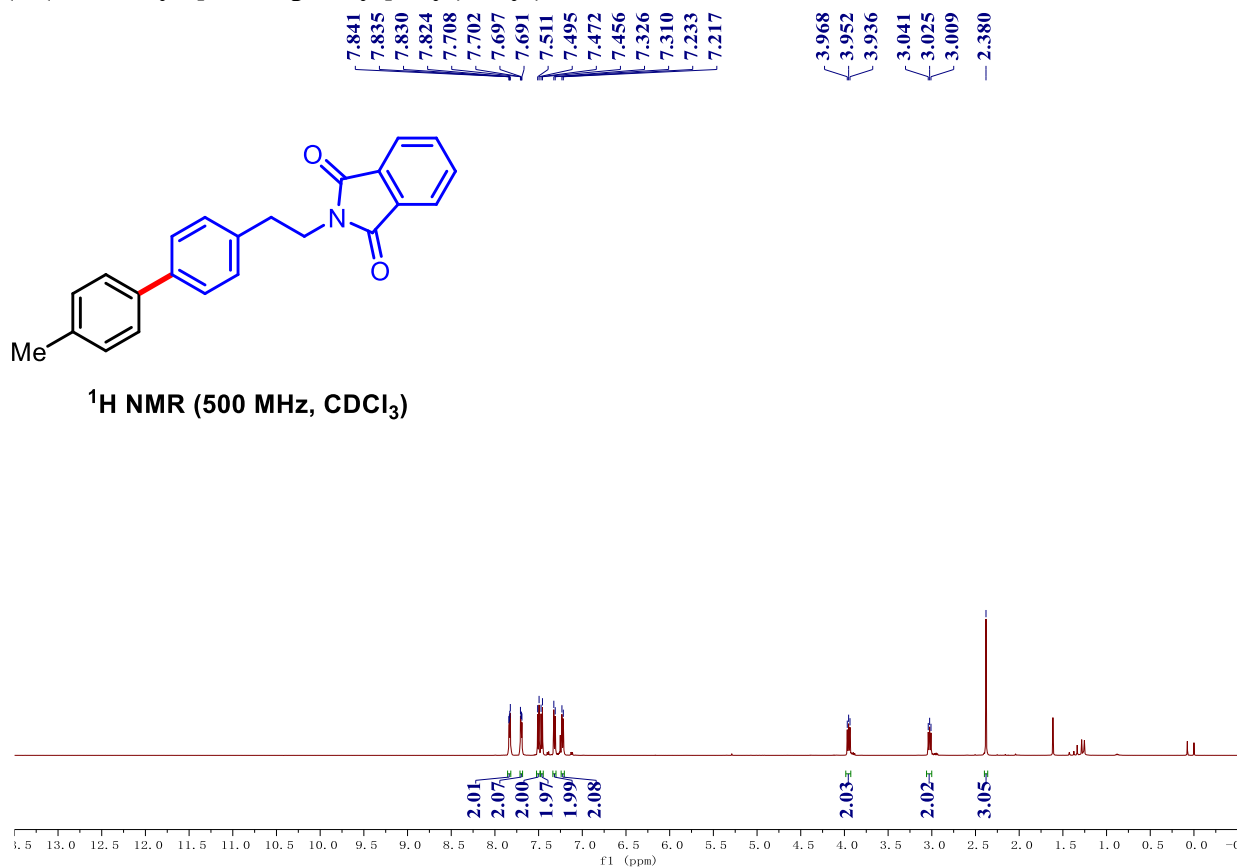
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)

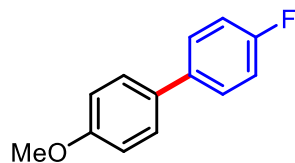


2-(2-(4'-Methyl-[1,1'-biphenyl]-4-yl)ethyl)isoindoline-1,3-dione, **3i**

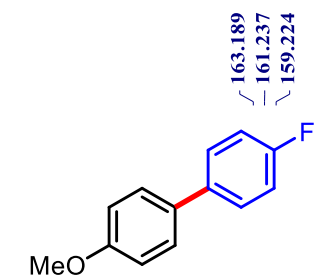
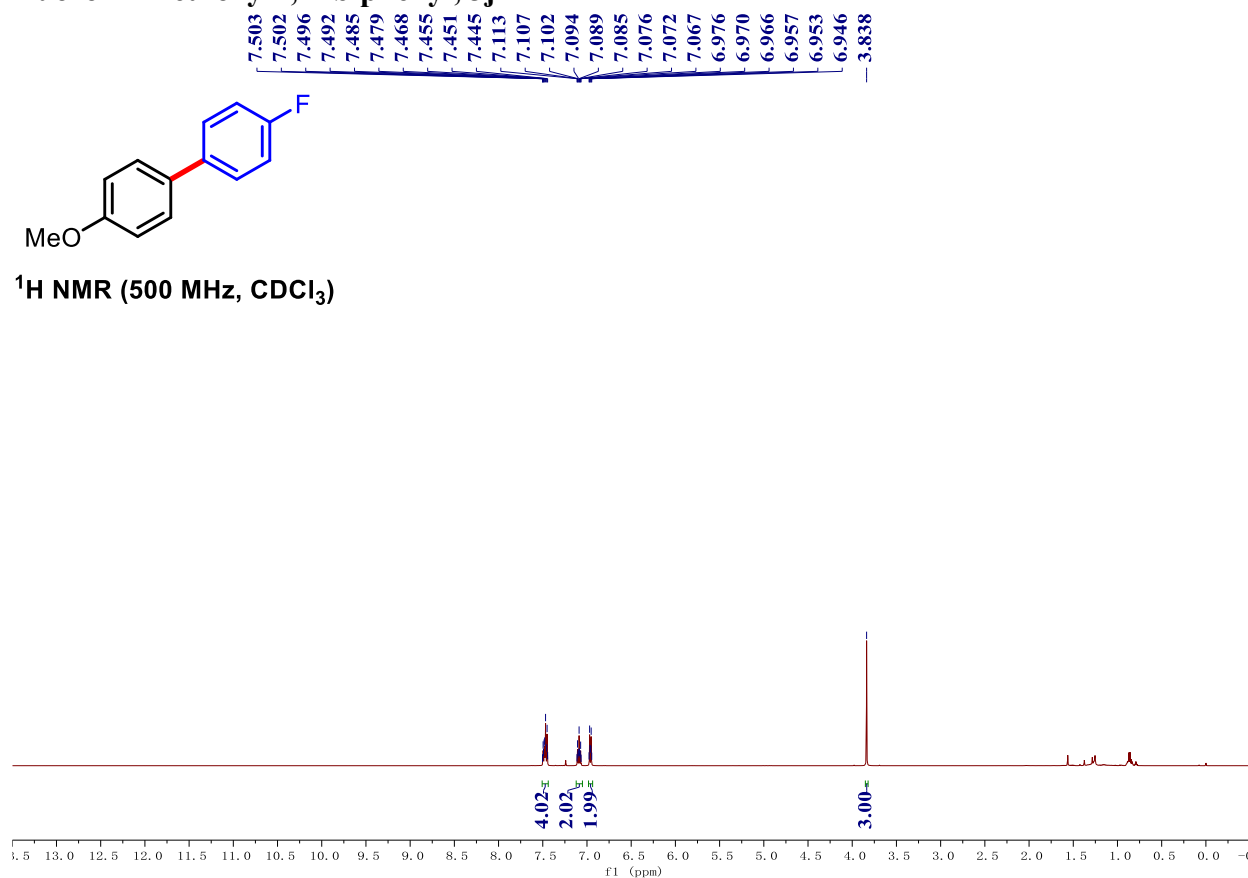




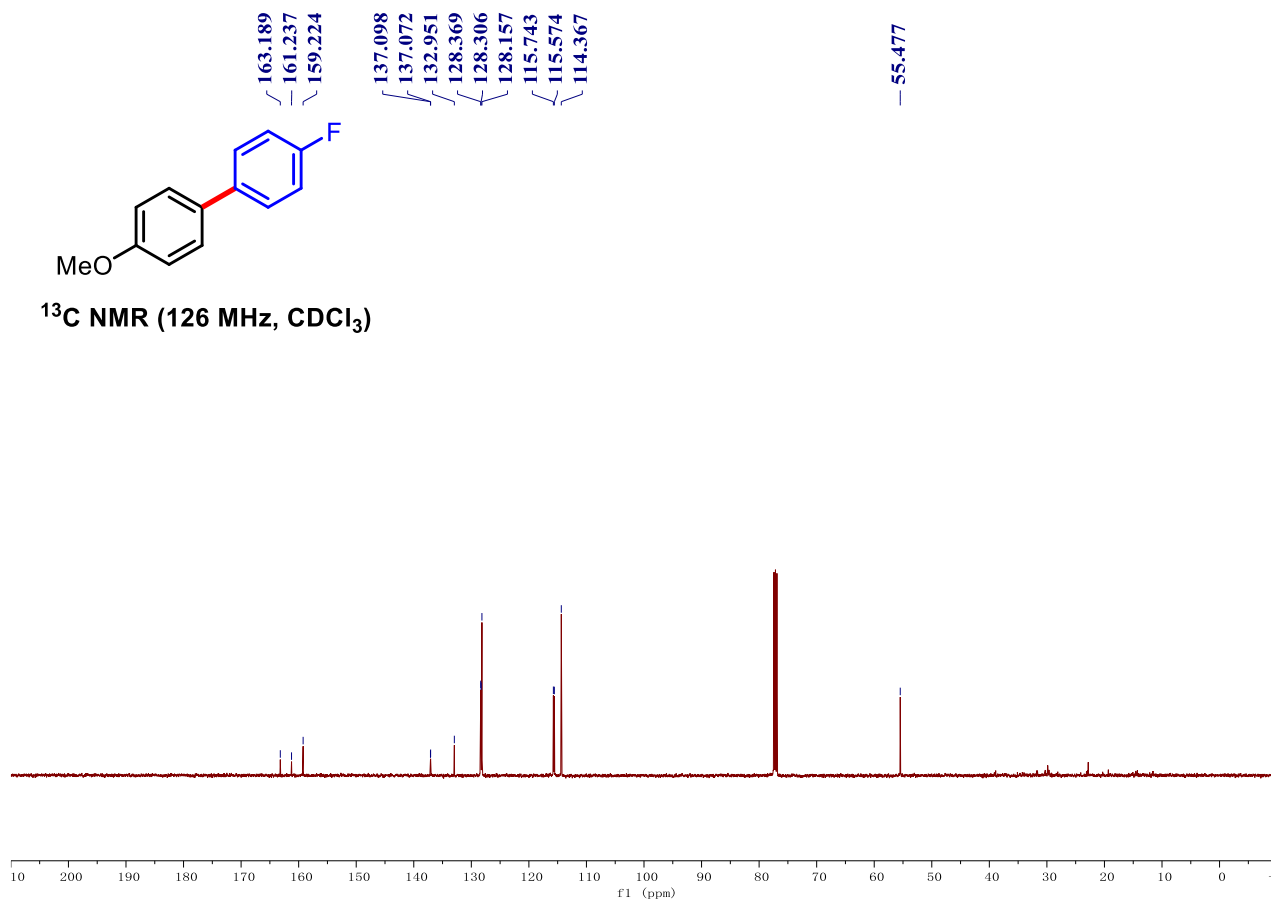
### 4-Fluoro-4'-methoxy-1,1'-biphenyl, 3j

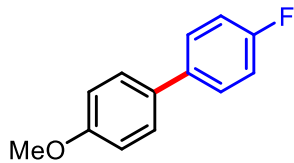


$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )

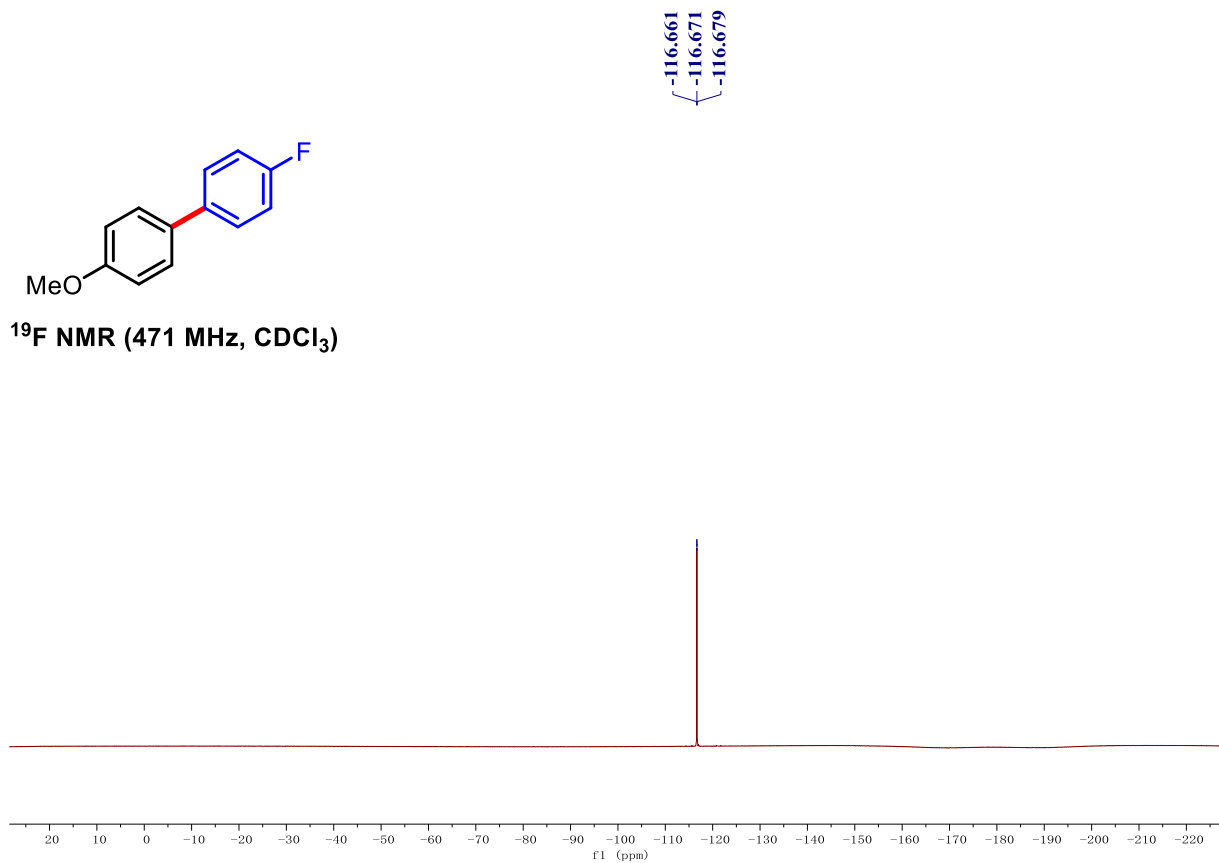


$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )

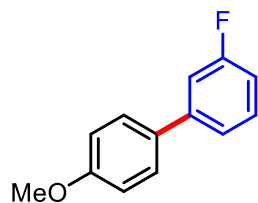




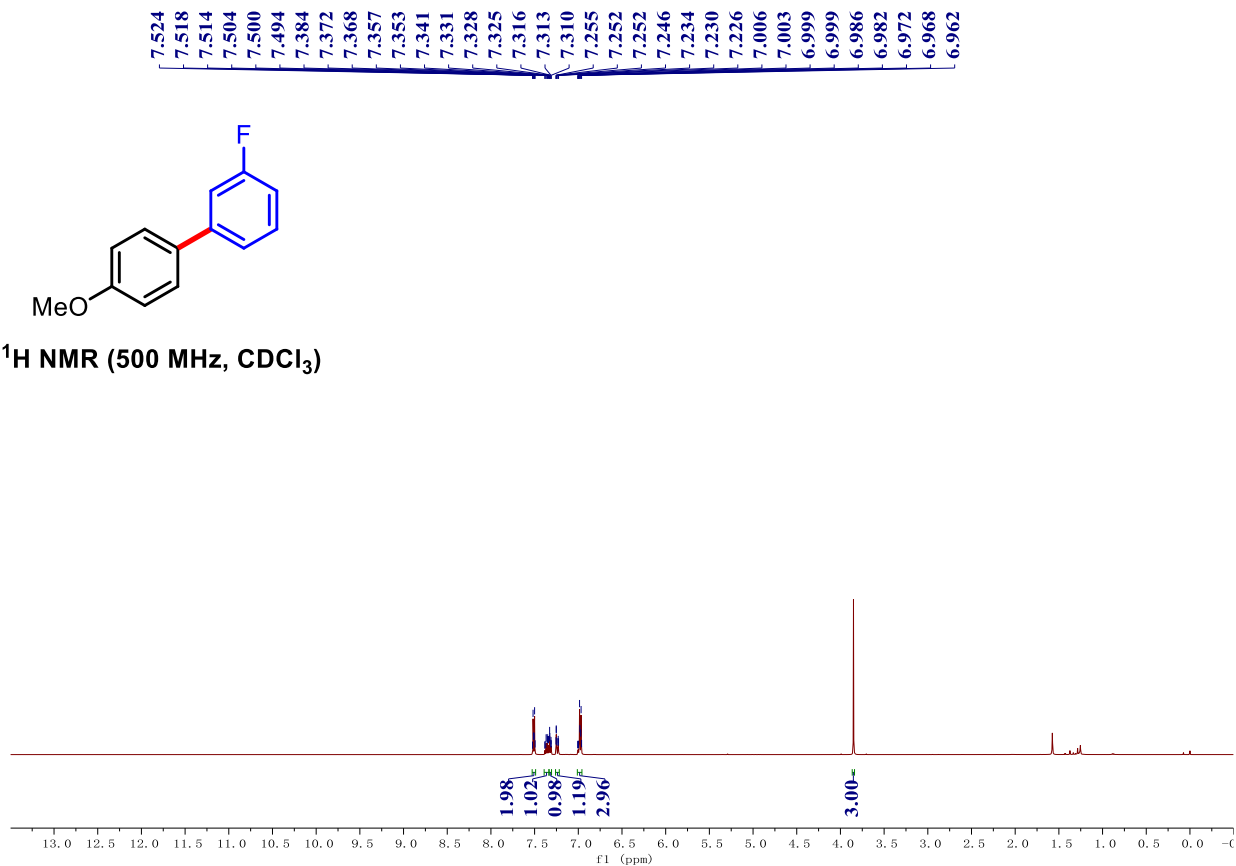
$^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )

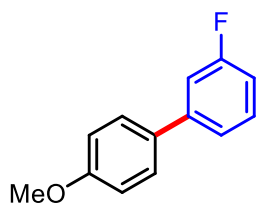


**3-Fluoro-4'-methoxy-1,1'-biphenyl, 3k**

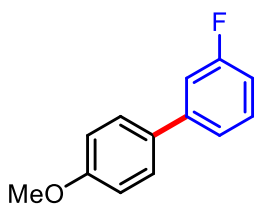
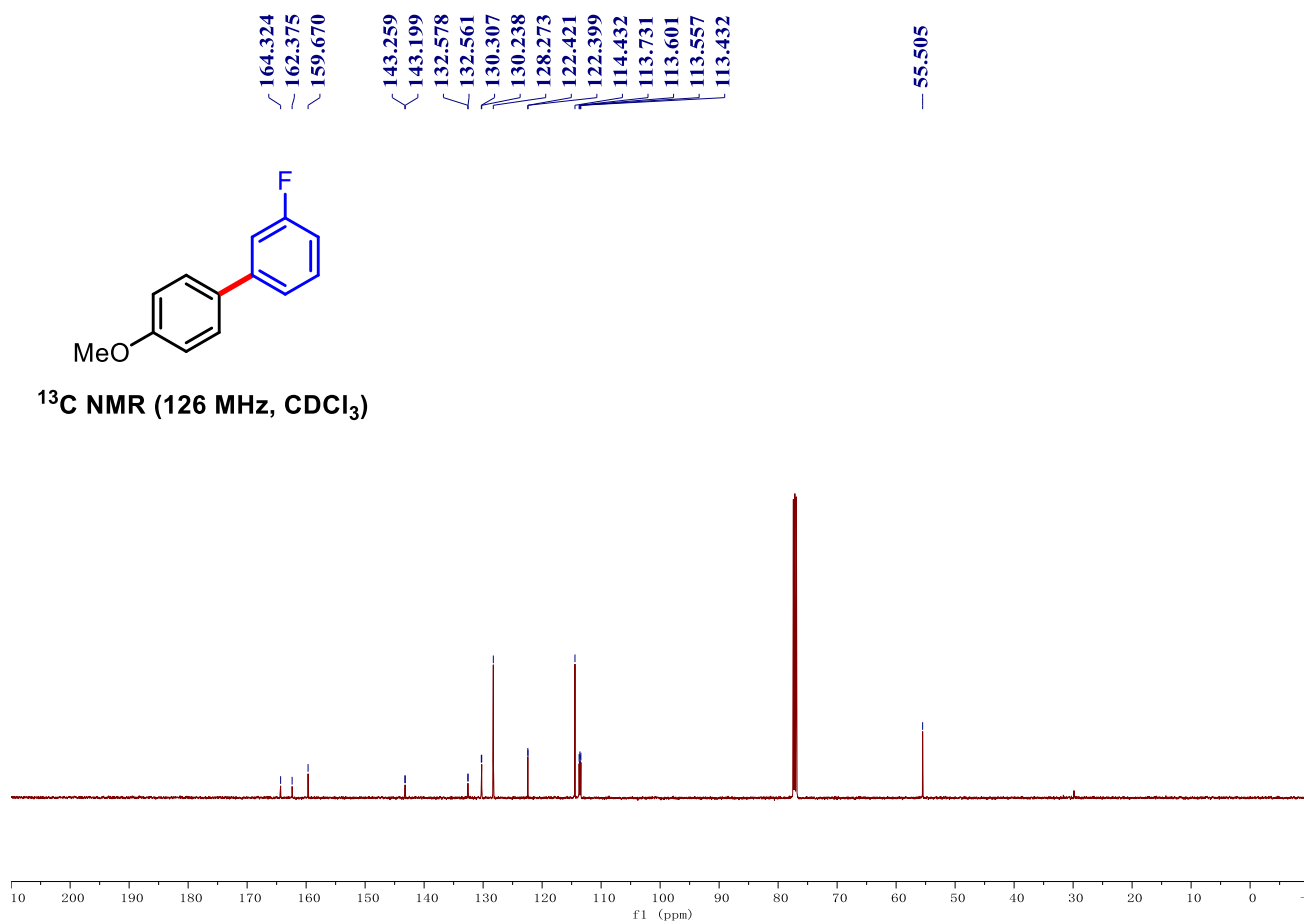


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )

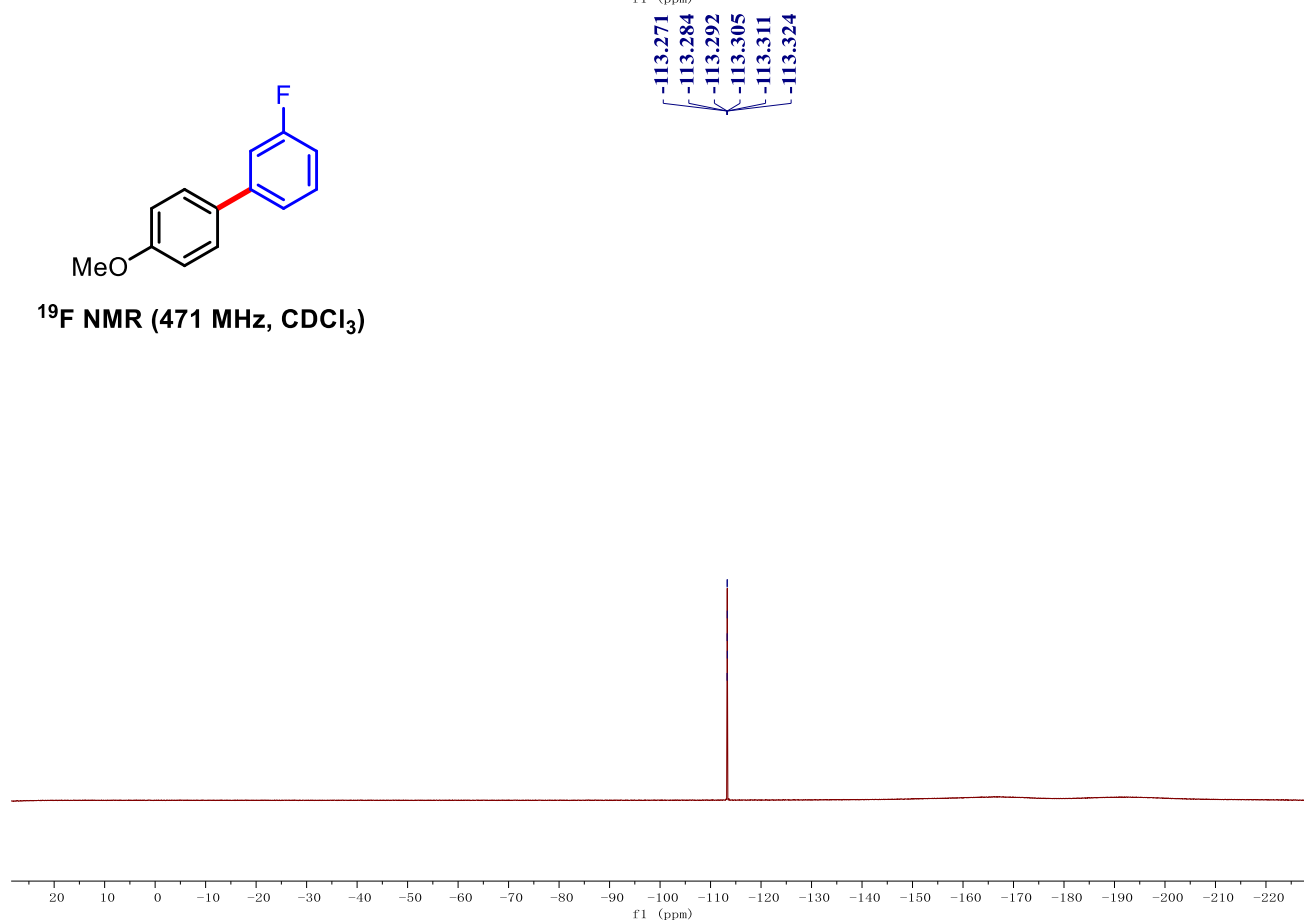




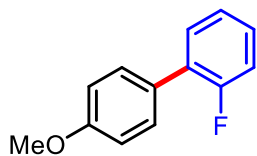
**$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )**



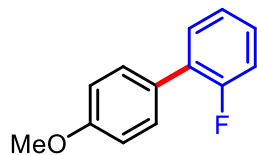
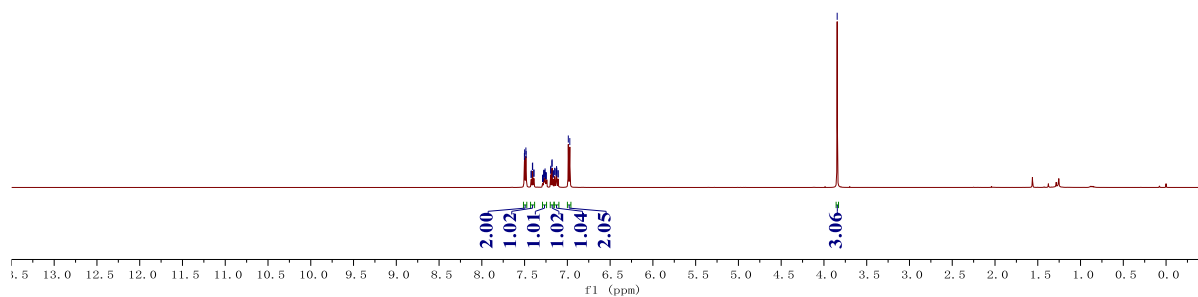
**$^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )**



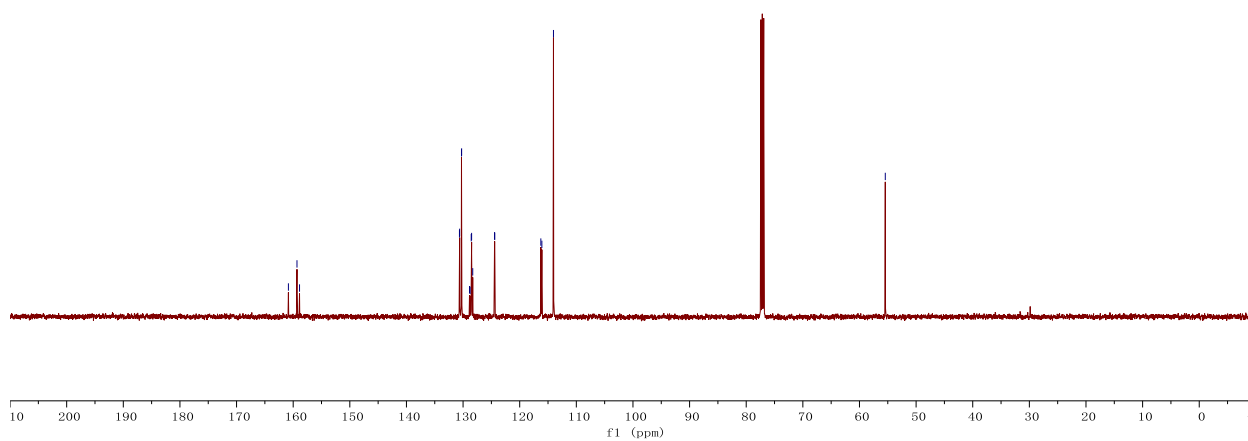
## 2-Fluoro-4'-methoxy-1,1'-biphenyl, 3l

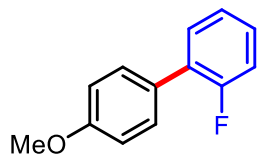


$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )

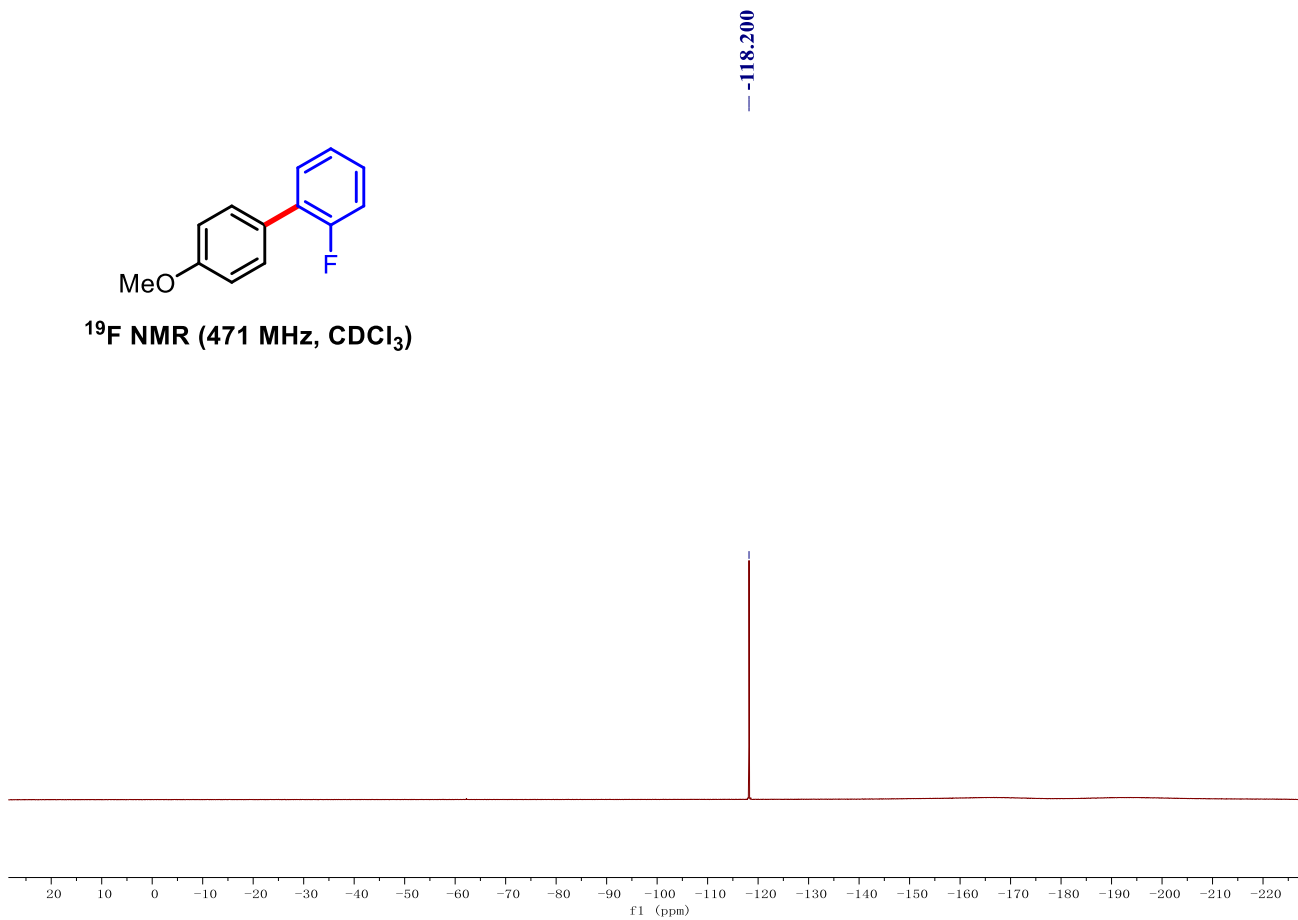


$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )

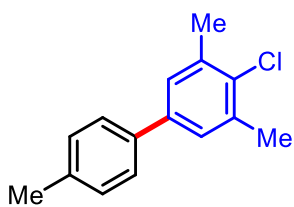




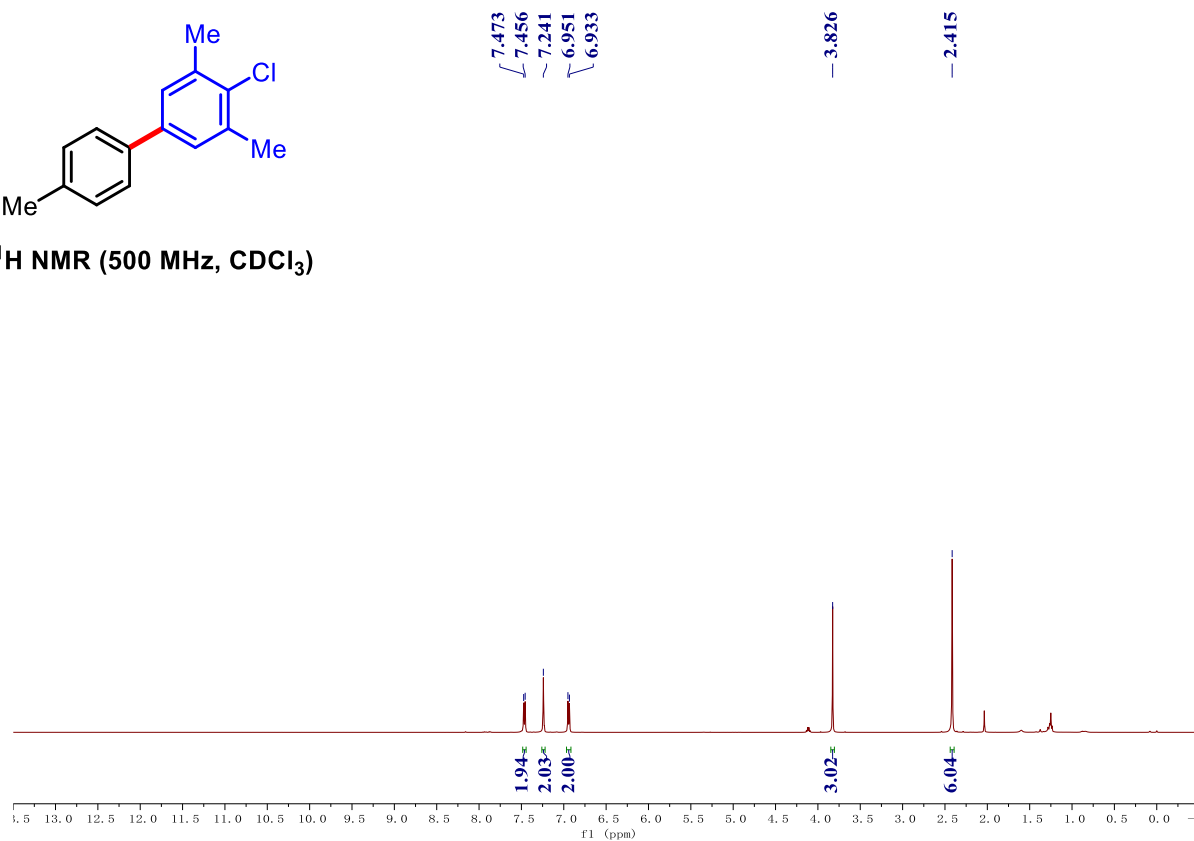
**$^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )**

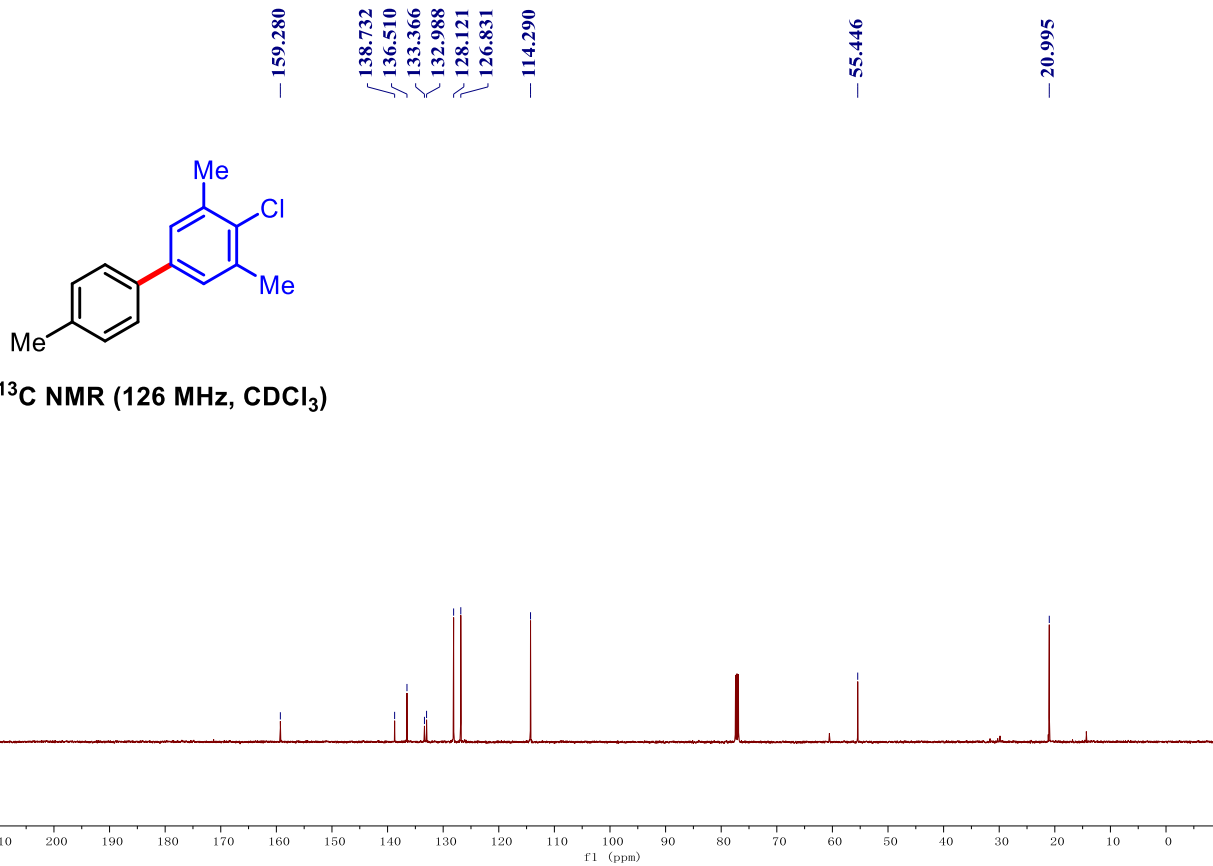


**4-Chloro-3,4,5-trimethyl-1,1'-biphenyl, 3m**

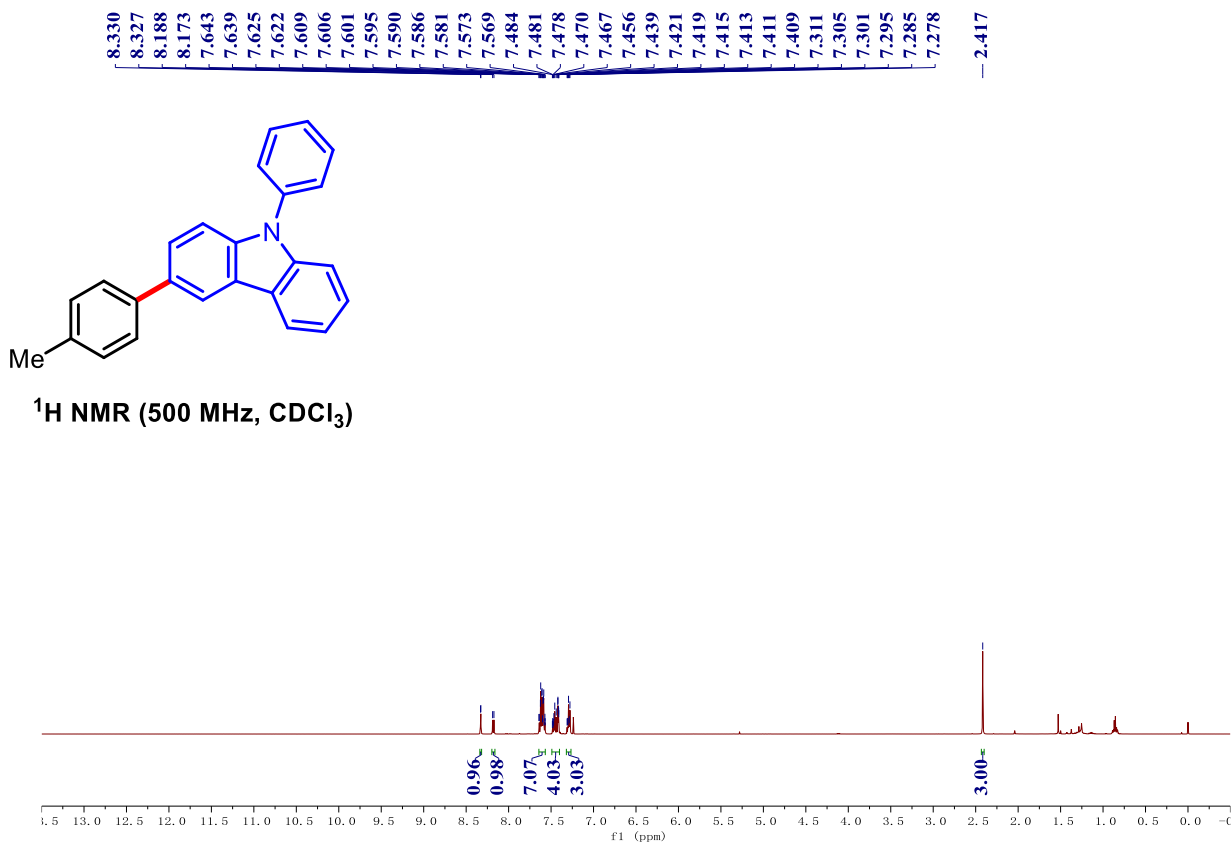


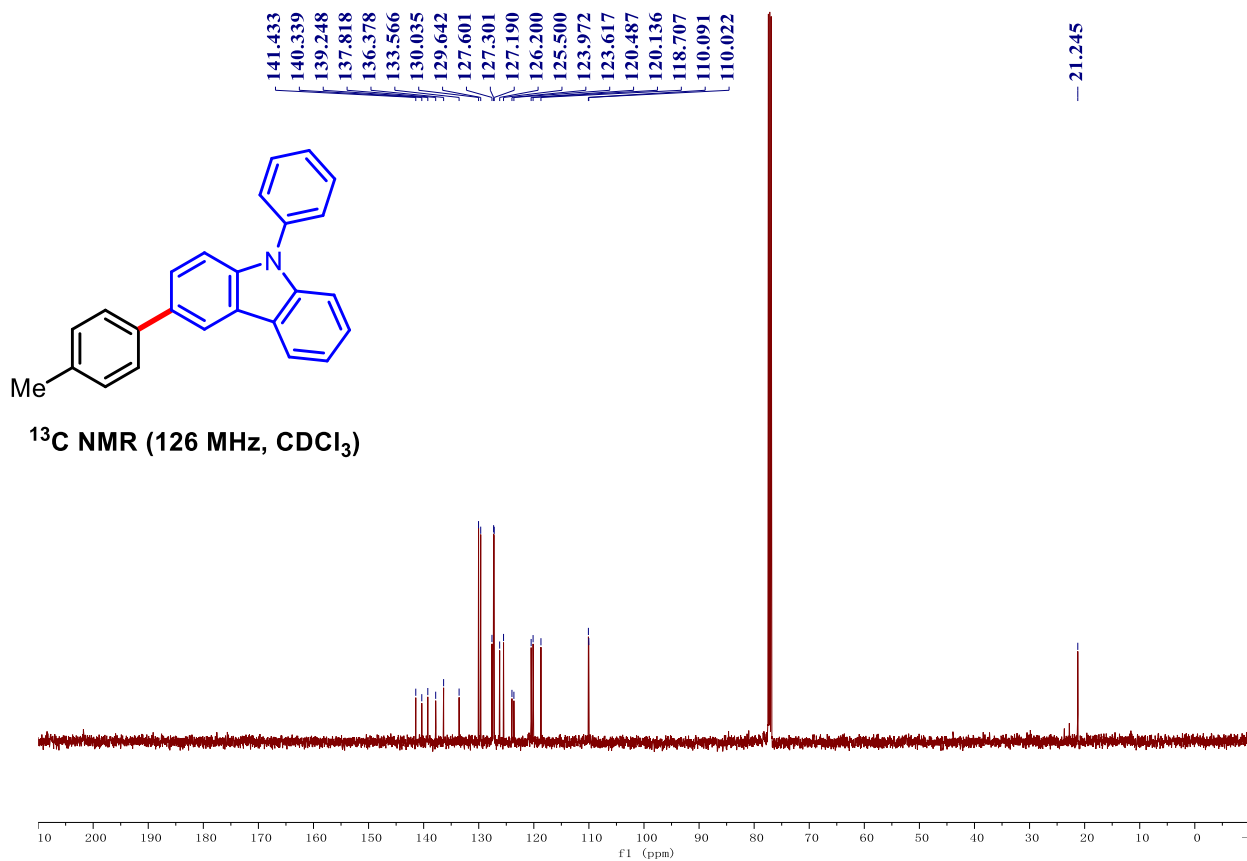
**$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )**



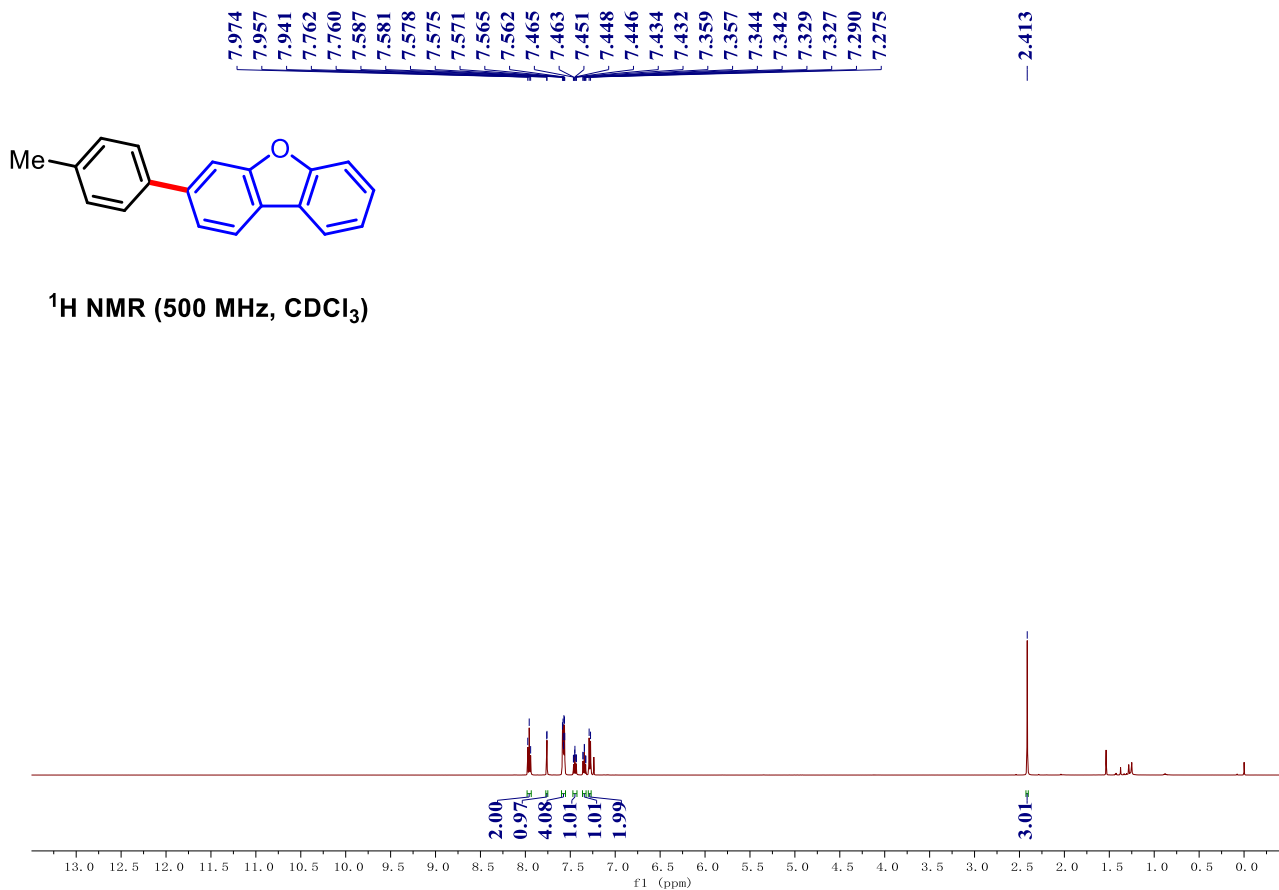


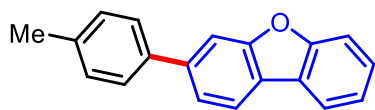
9-Phenyl-3-(*p*-tolyl)-9*H*-carbazole, 3n



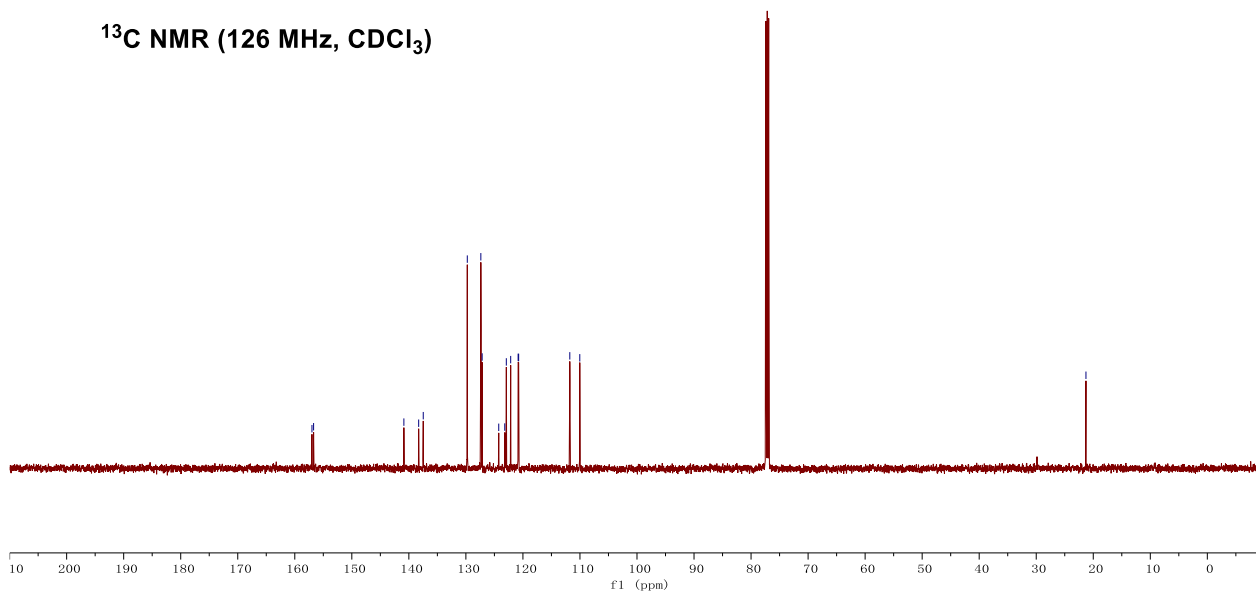


2-(*p*-Tolyl)dibenzo[*b,d*]furan, 3o

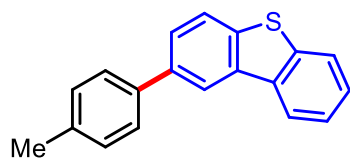




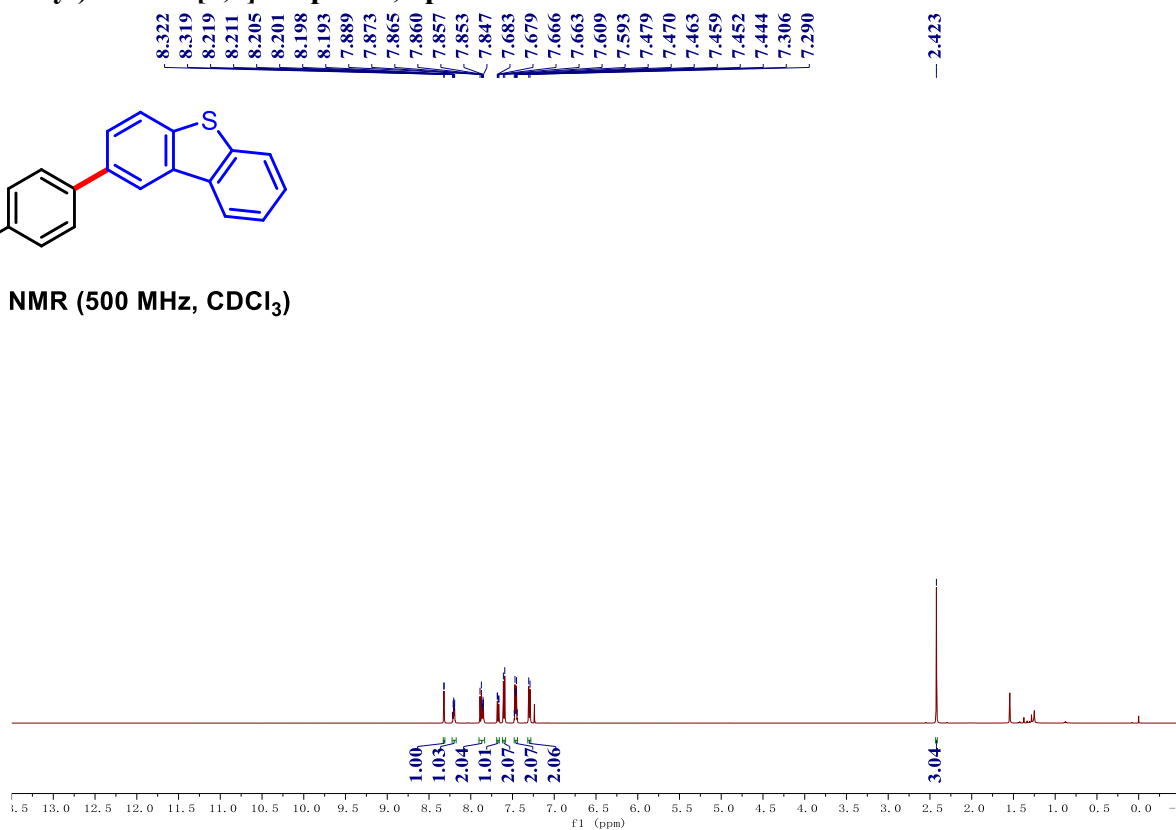
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)



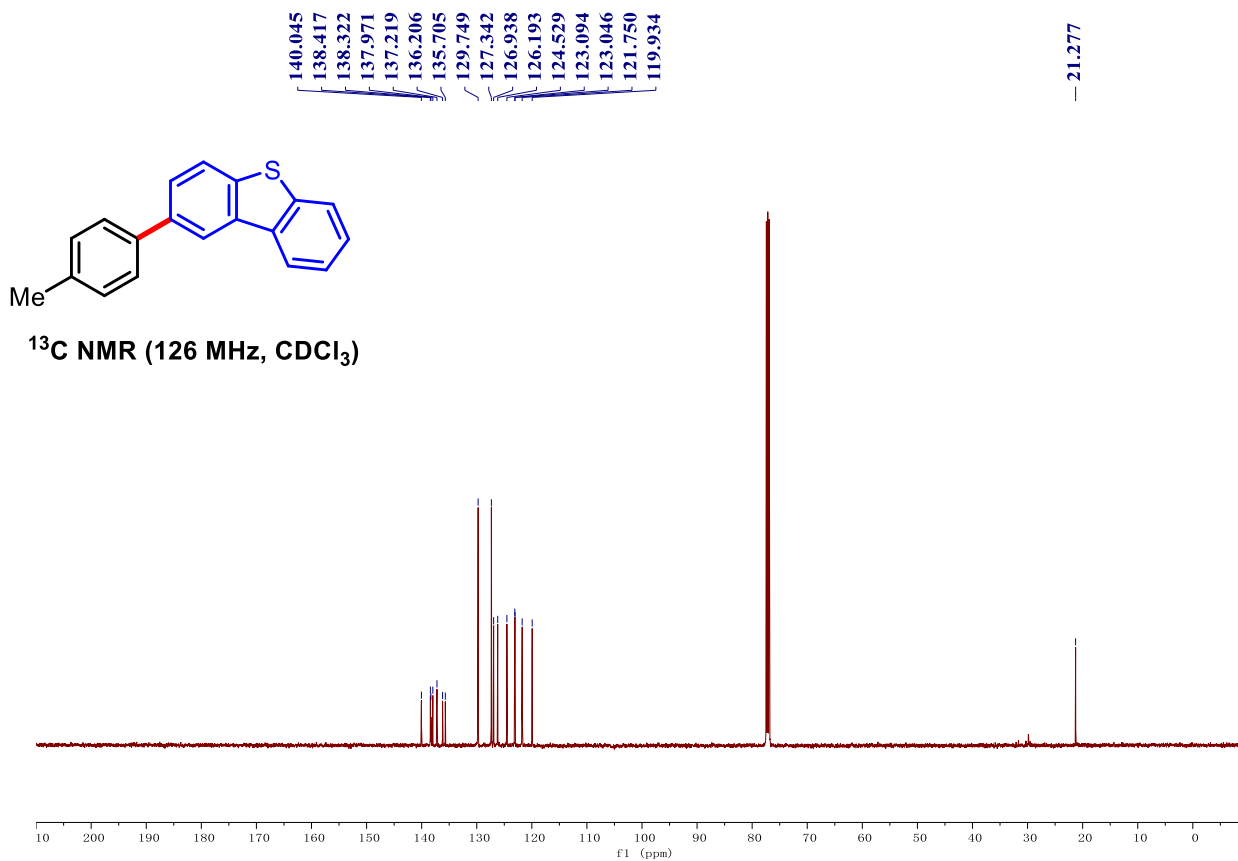
2-(*p*-Tolyl)dibenzo[*b,d*]thiophene, 3p



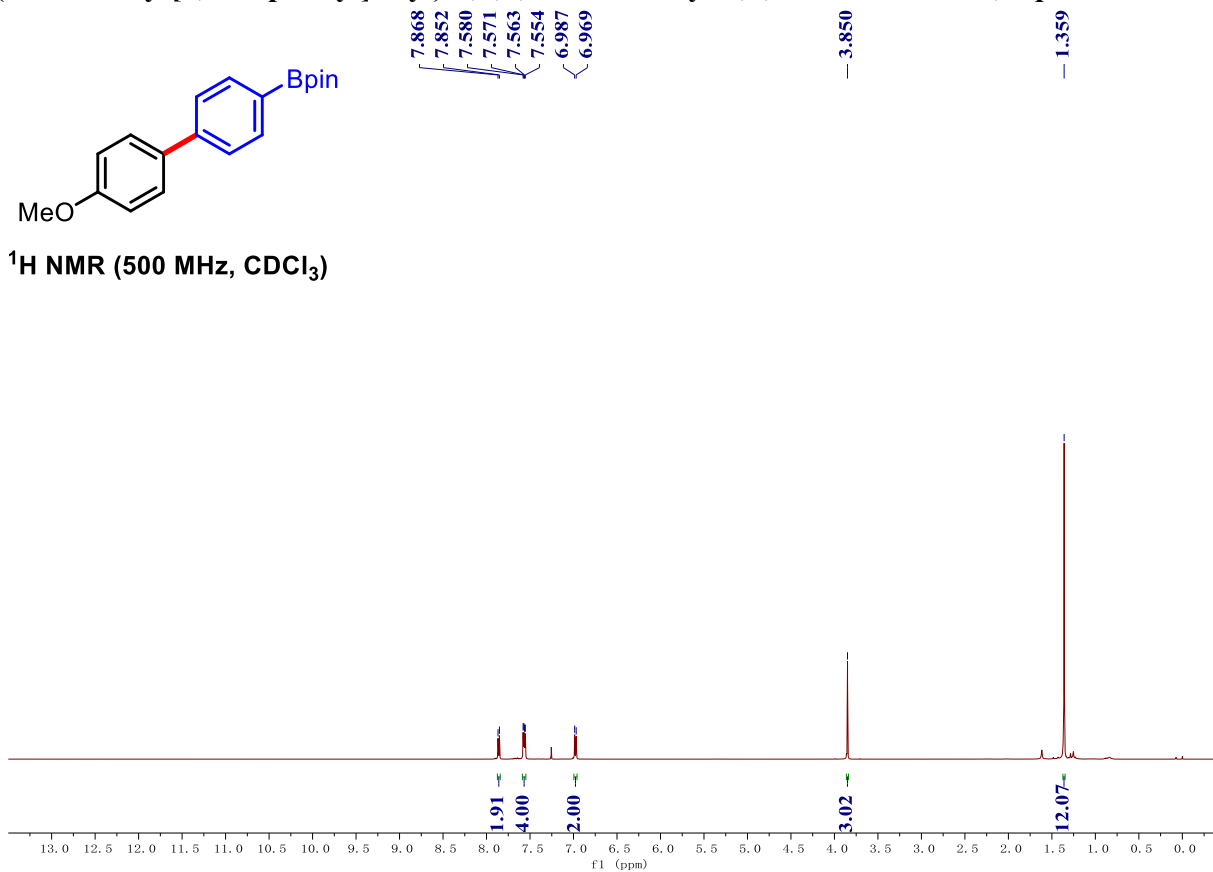
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)

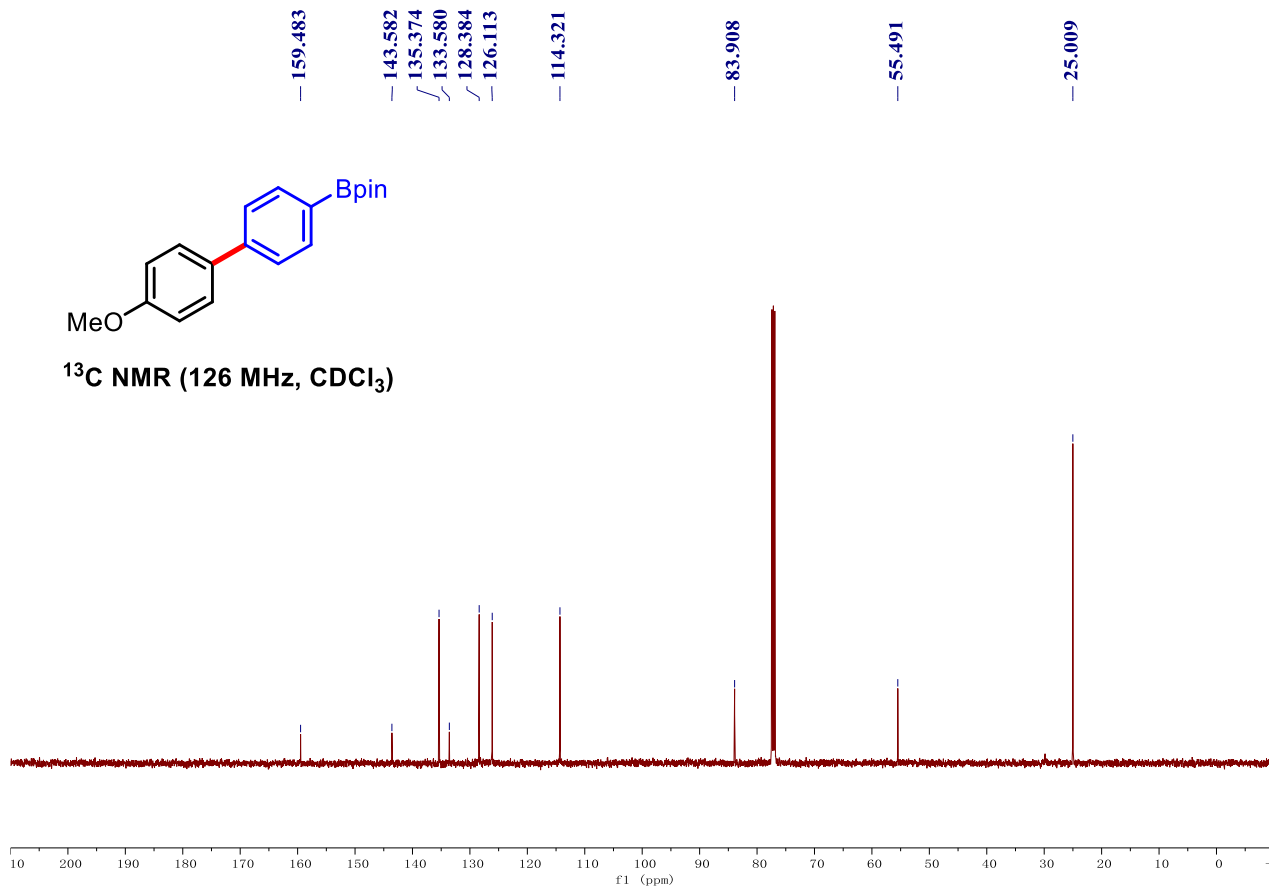




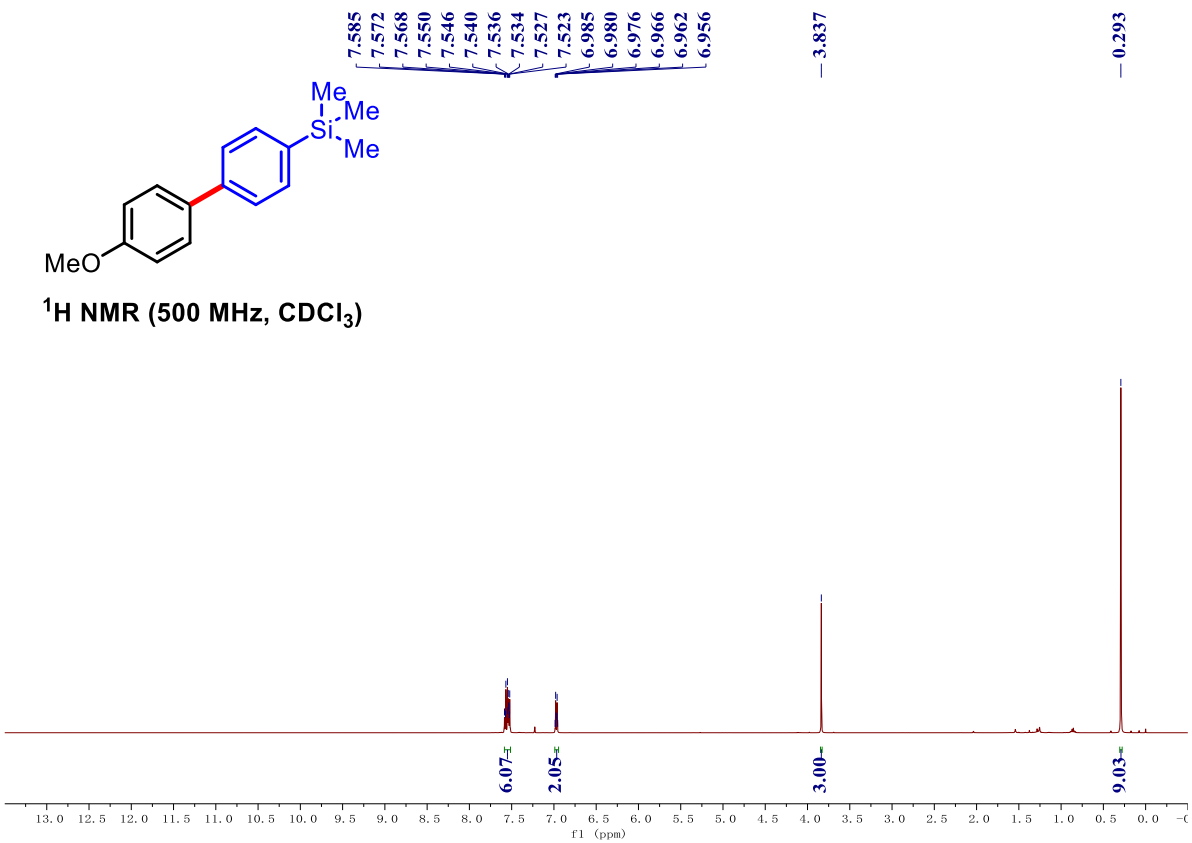


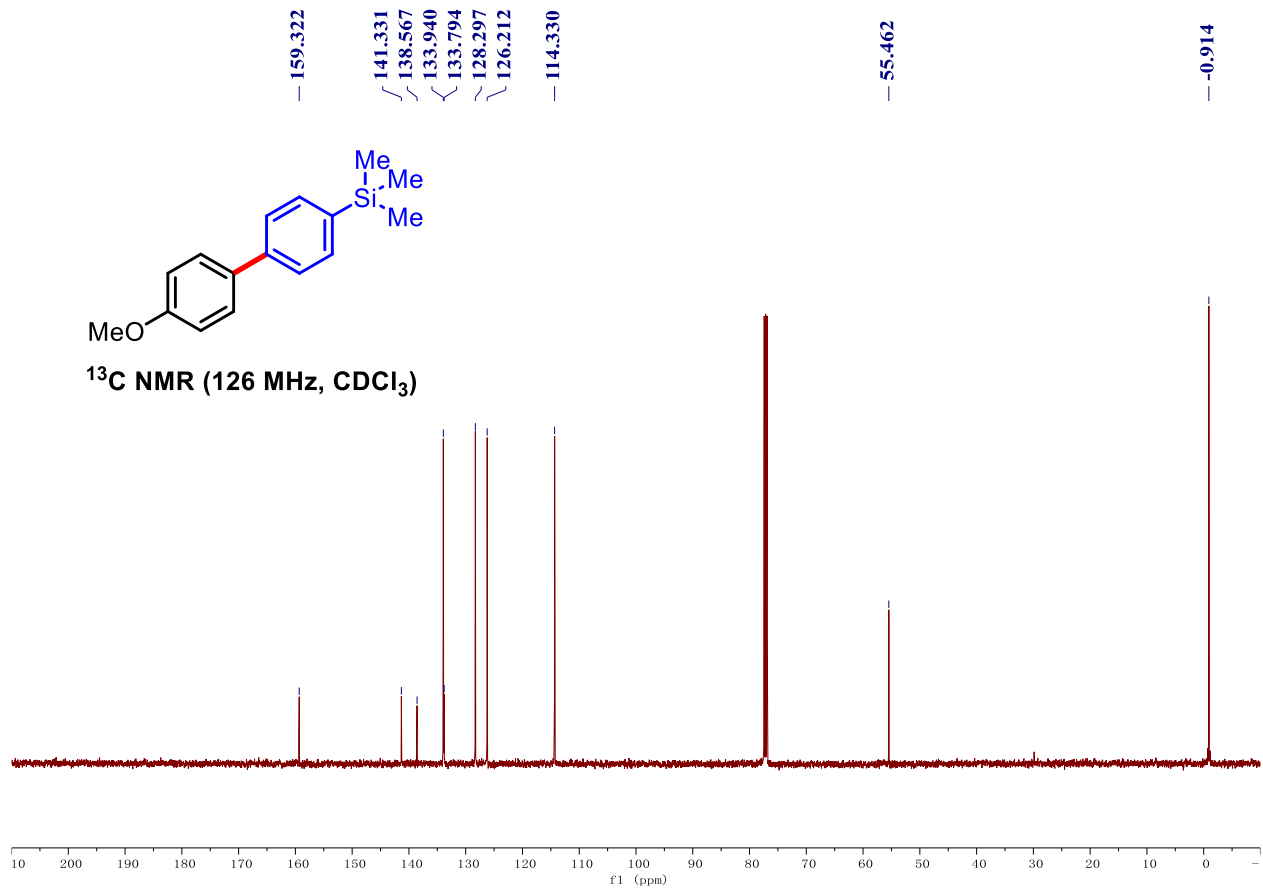
**2-(4'-Methoxy-[1,1'-biphenyl]-4-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane, 3q**



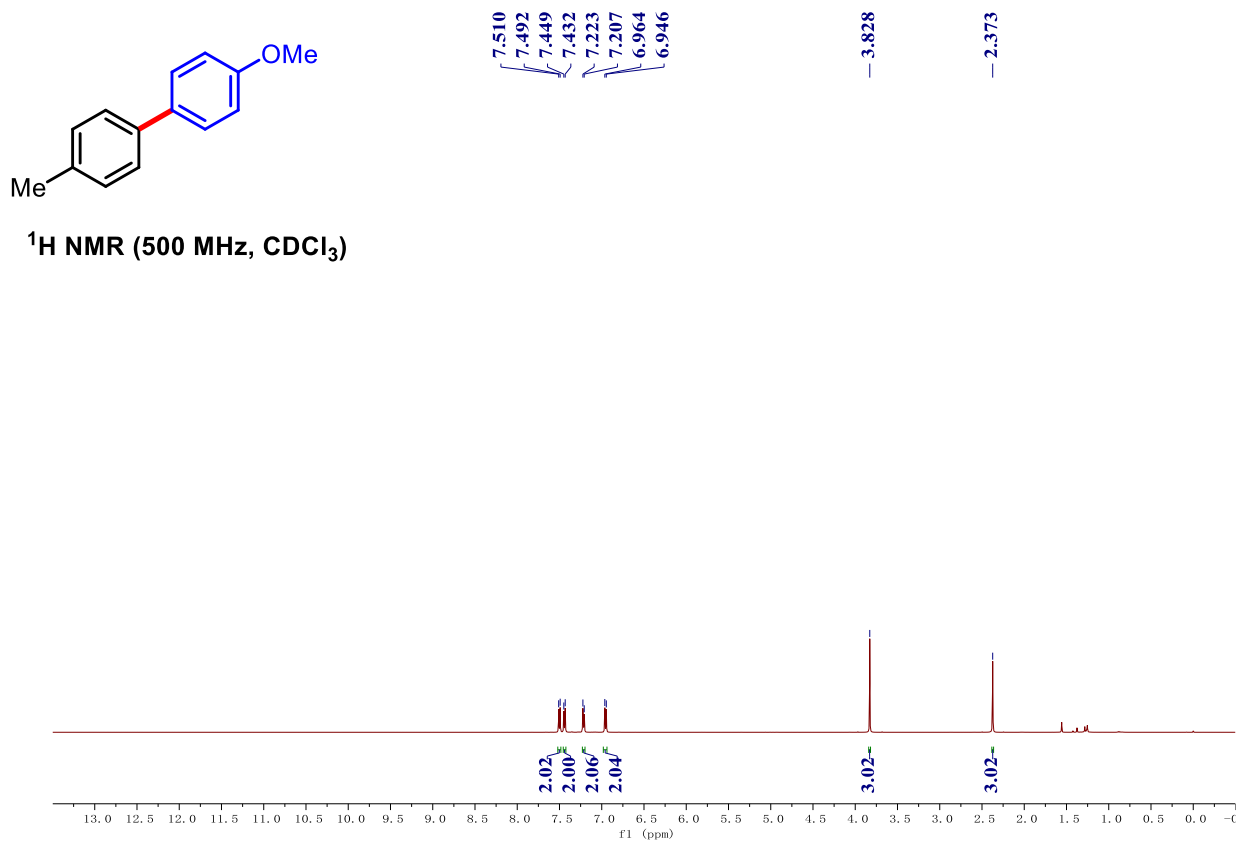


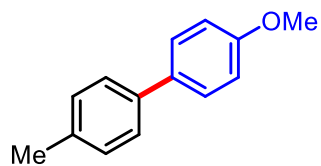
**(4'-Methoxy-[1,1'-biphenyl]-4-yl)trimethylsilane, 3r**



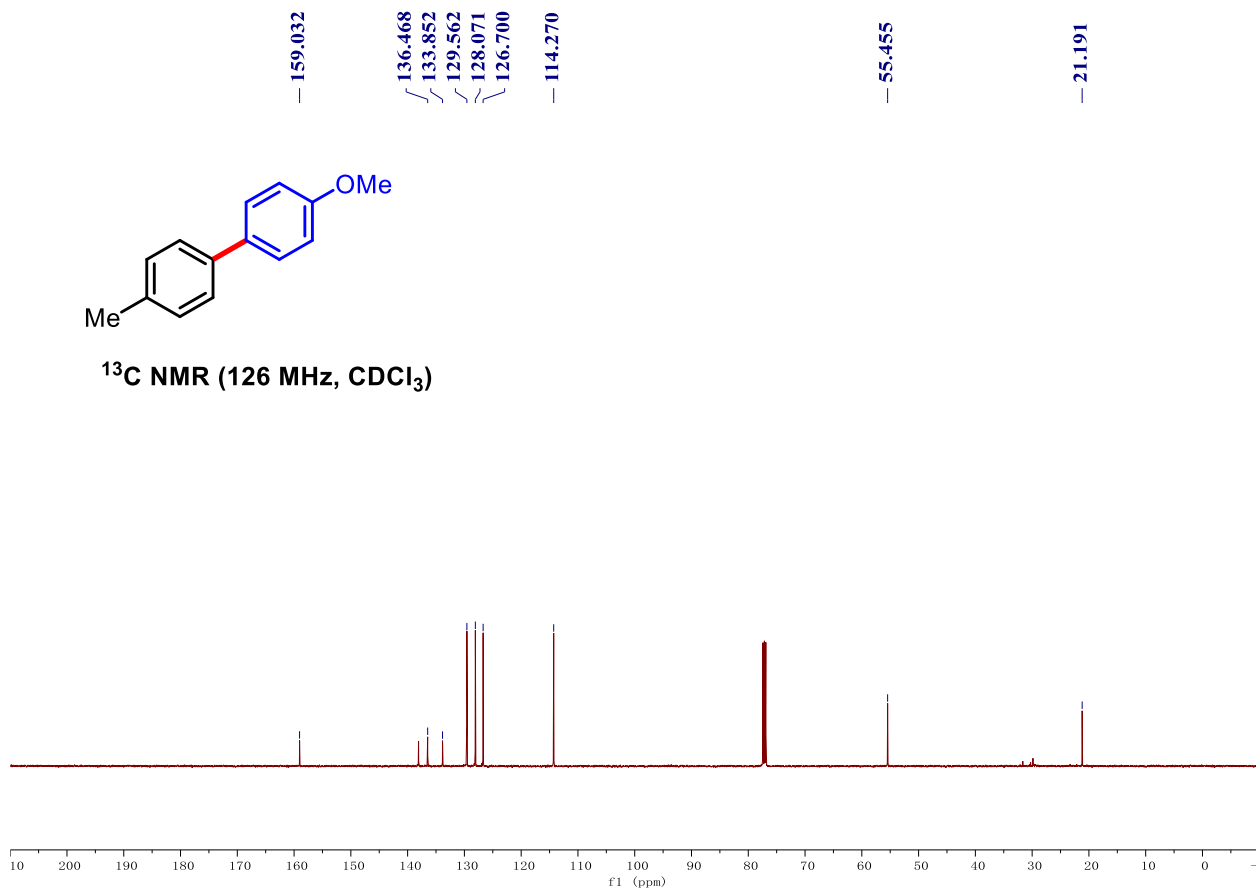


**4-Methoxy-4'-methyl-1,1'-biphenyl, 3s**

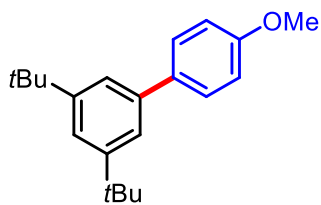




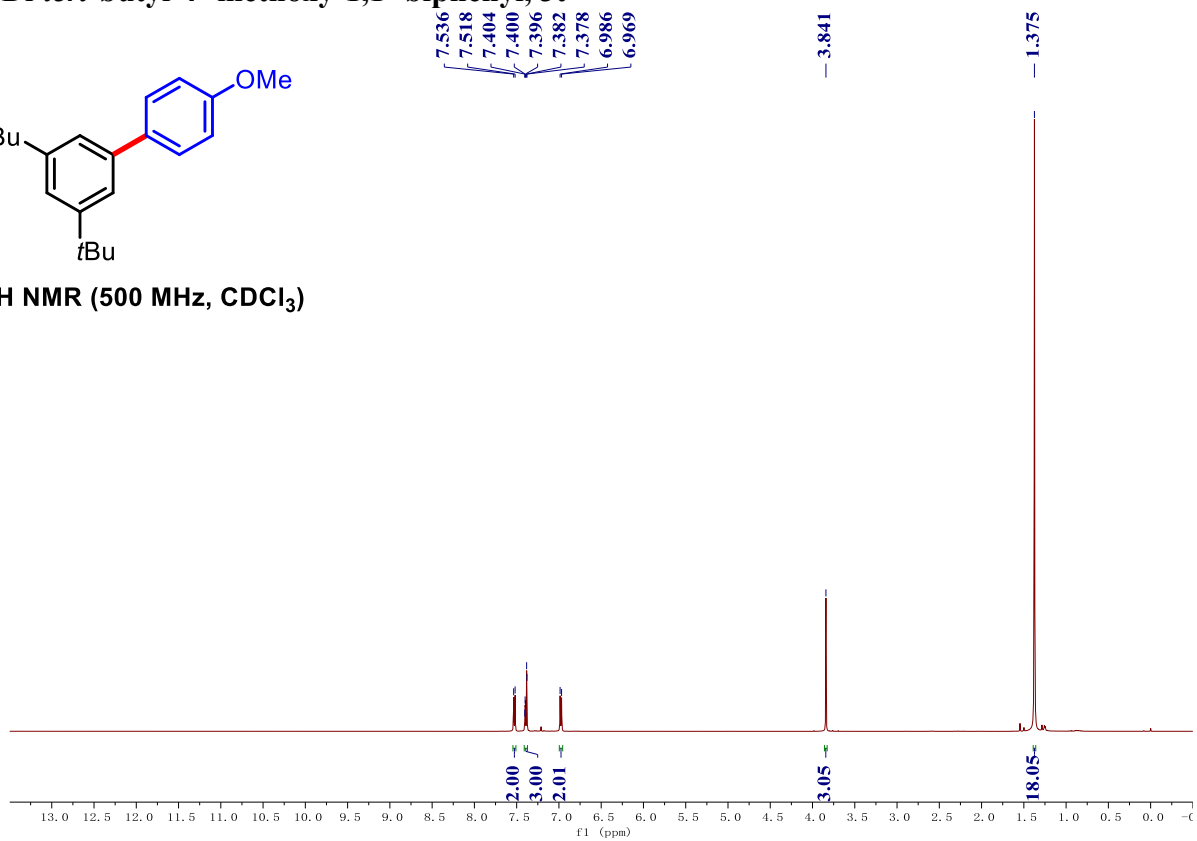
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )

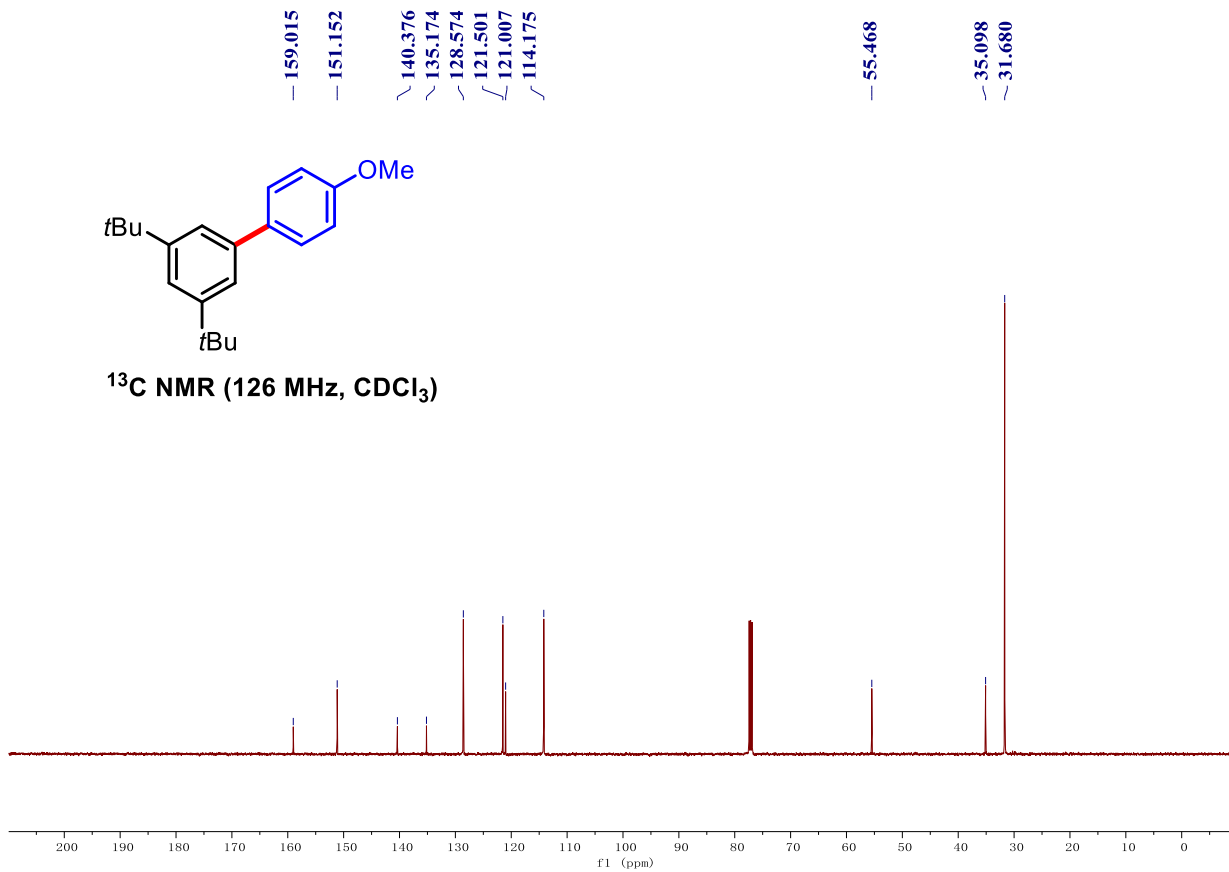


**3,5-Di-*tert*-butyl-4'-methoxy-1,1'-biphenyl, 3t**

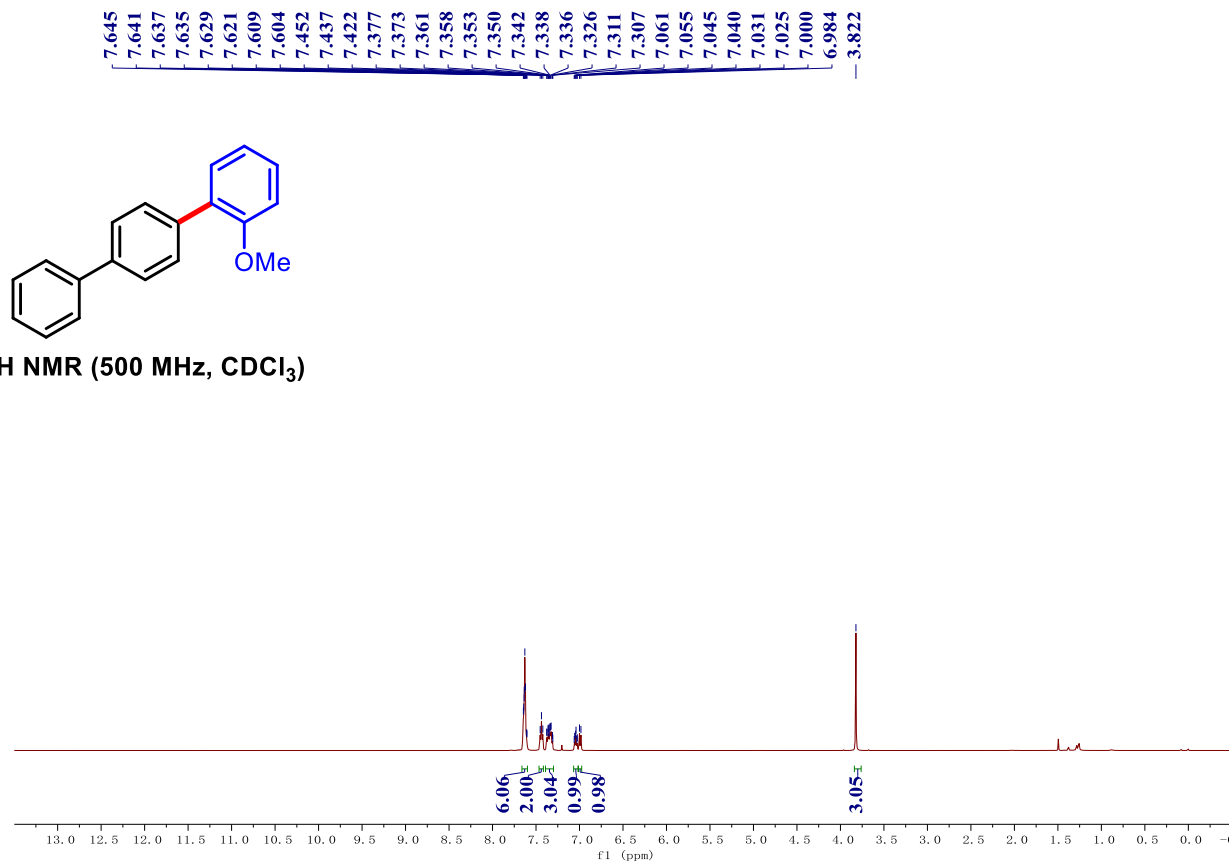


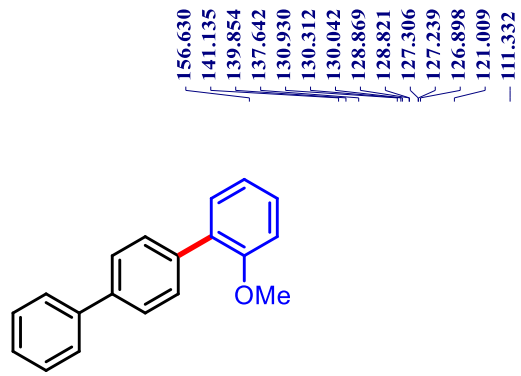
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )





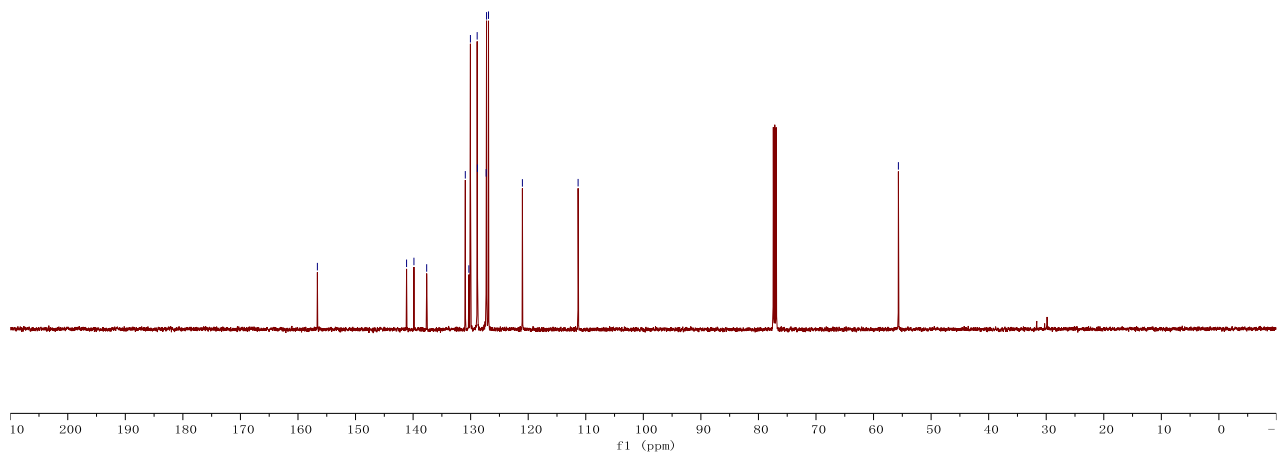
2-Methoxy-1,1':4',1''-terphenyl, 3u



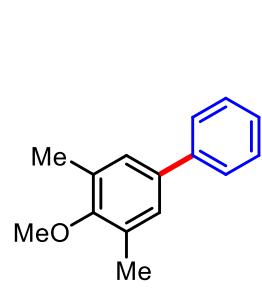


156.630  
 141.135  
 139.854  
 137.642  
 130.930  
 130.312  
 130.042  
 128.869  
 128.821  
 127.306  
 127.239  
 126.898  
 121.009  
 -111.332

-55.683



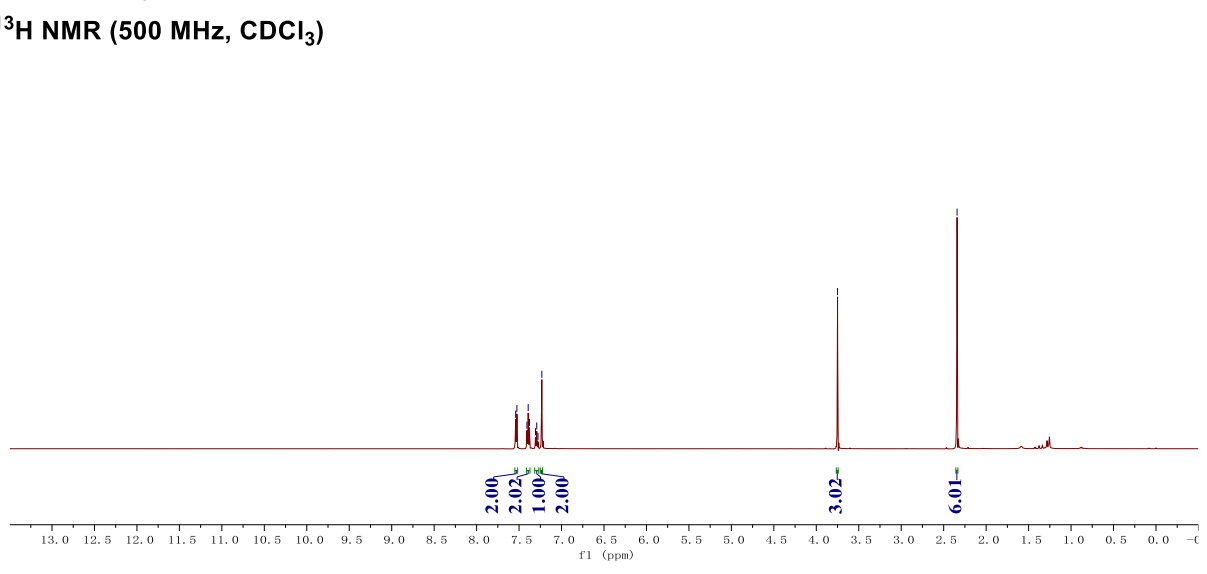
**4-Methoxy-3,5-dimethyl-1,1'-biphenyl, 3v**

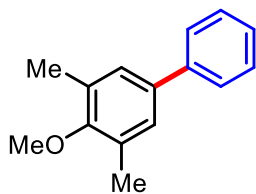


7.540  
 7.526  
 7.408  
 7.393  
 7.377  
 7.307  
 7.292  
 7.277  
 7.232

-3.750

-2.342



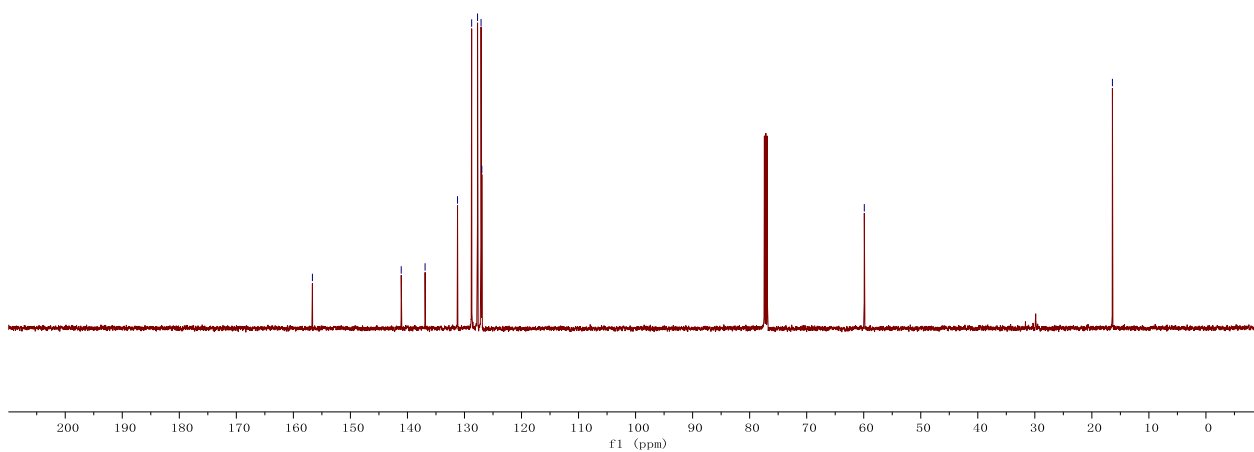


$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )

156.661  
 141.091  
 136.908  
 131.218  
 128.742  
 127.708  
 127.092  
 126.958

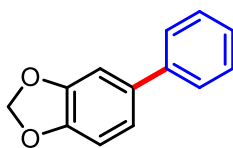
59.897

16.394

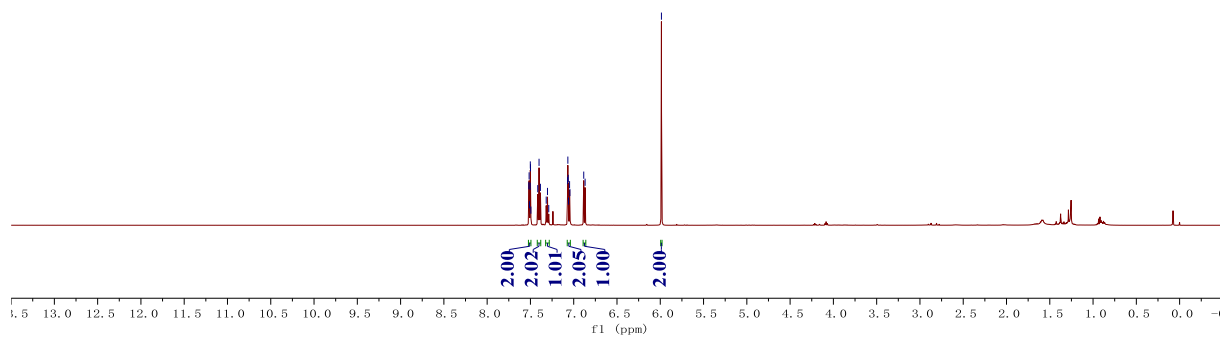


5-Phenylbenzo[d][1,3]dioxole, 3w

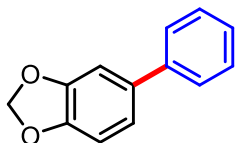
7.518  
 7.515  
 7.511  
 7.503  
 7.501  
 7.499  
 7.495  
 7.415  
 7.400  
 7.385  
 7.318  
 7.303  
 7.288  
 7.071  
 7.068  
 7.064  
 7.060  
 7.048  
 7.044  
 6.884  
 6.868  
 5.987



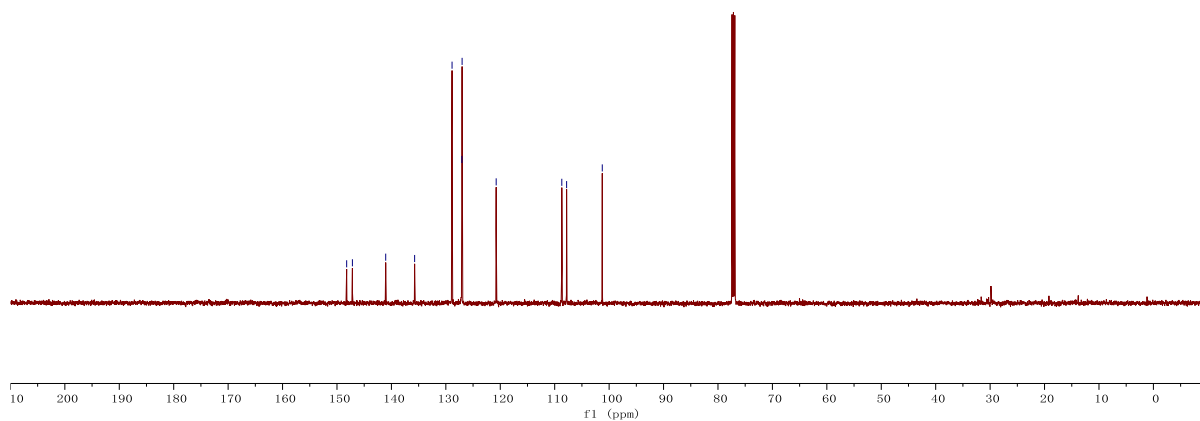
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )



148.222  
 147.168  
 141.047  
 135.735  
 128.859  
 127.052  
 127.013  
 120.752  
 108.695  
 107.810  
 101.259

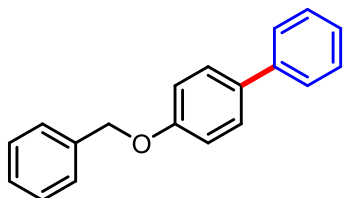


<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)

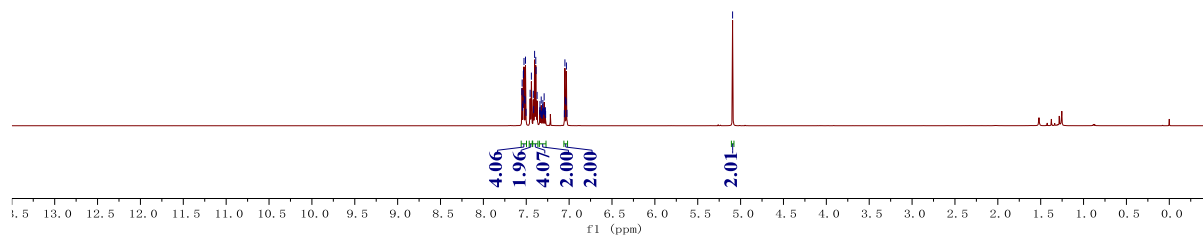


4-(Benzyloxy)-1,1'-biphenyl, 3x

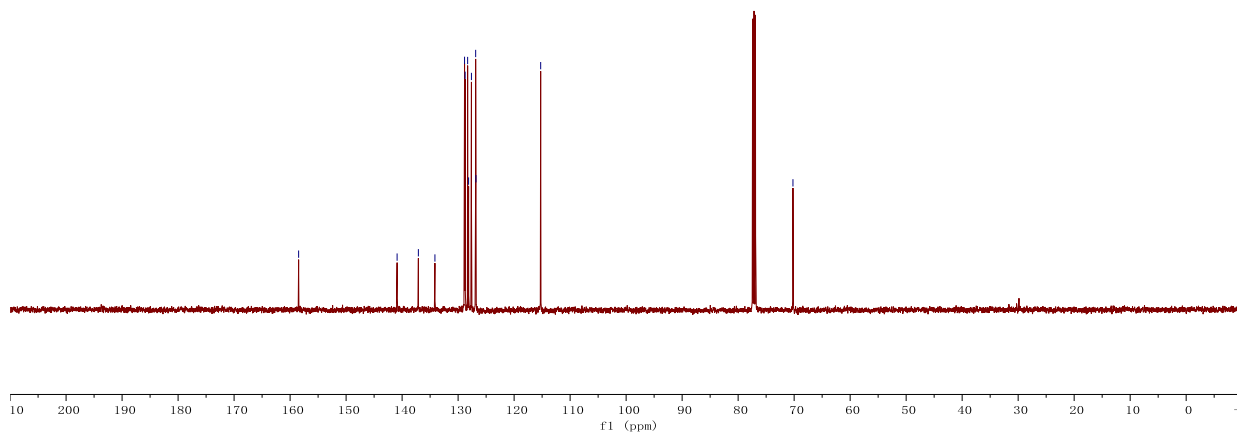
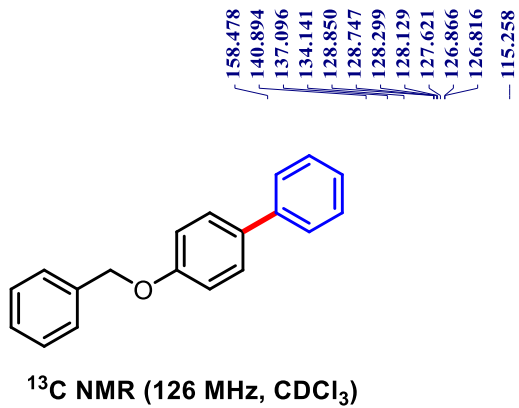
7.551  
 7.548  
 7.544  
 7.538  
 7.536  
 7.534  
 7.532  
 7.526  
 7.522  
 7.513  
 7.509  
 7.455  
 7.440  
 7.417  
 7.413  
 7.403  
 7.389  
 7.386  
 7.374  
 7.340  
 7.326  
 7.309  
 7.306  
 7.304  
 7.291  
 7.049  
 7.045  
 7.036  
 5.093



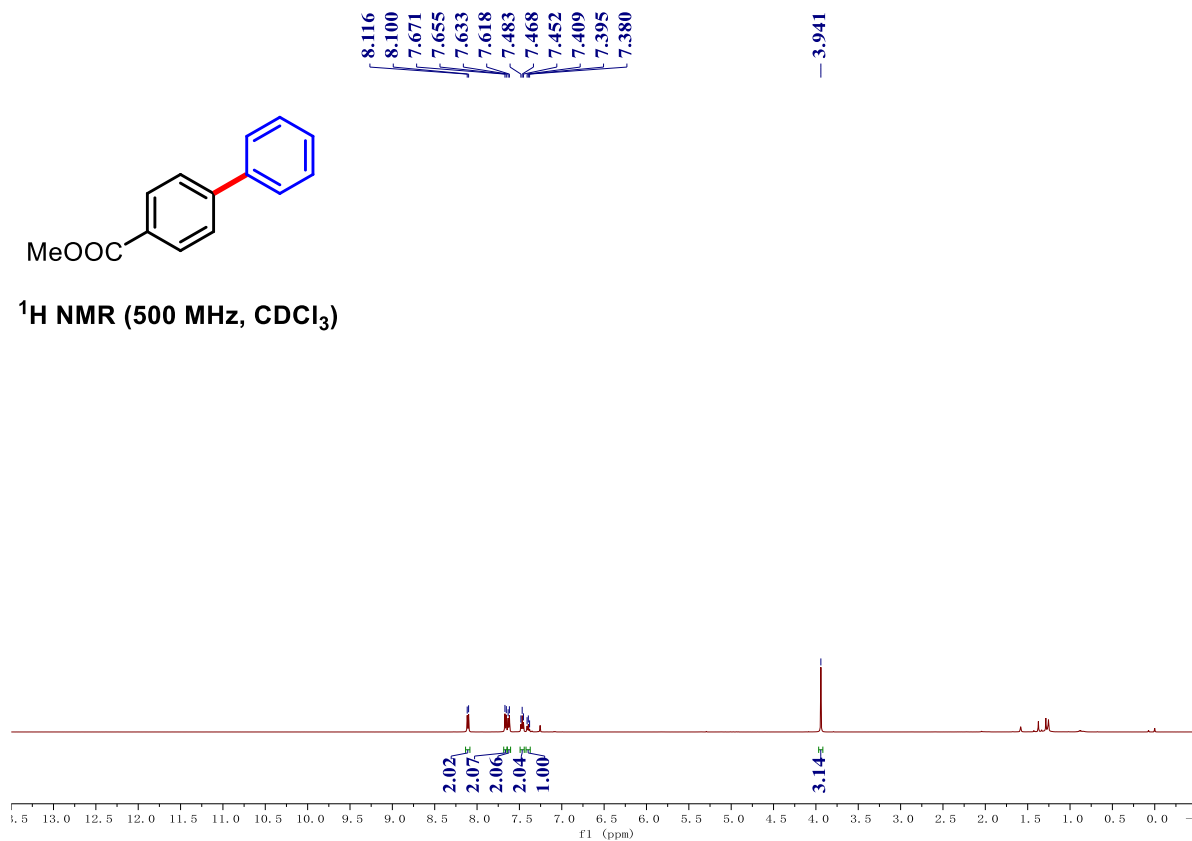
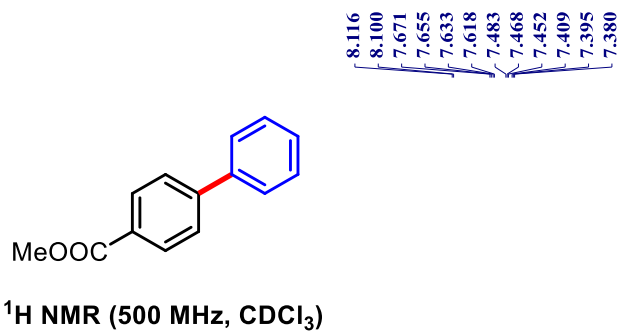
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)

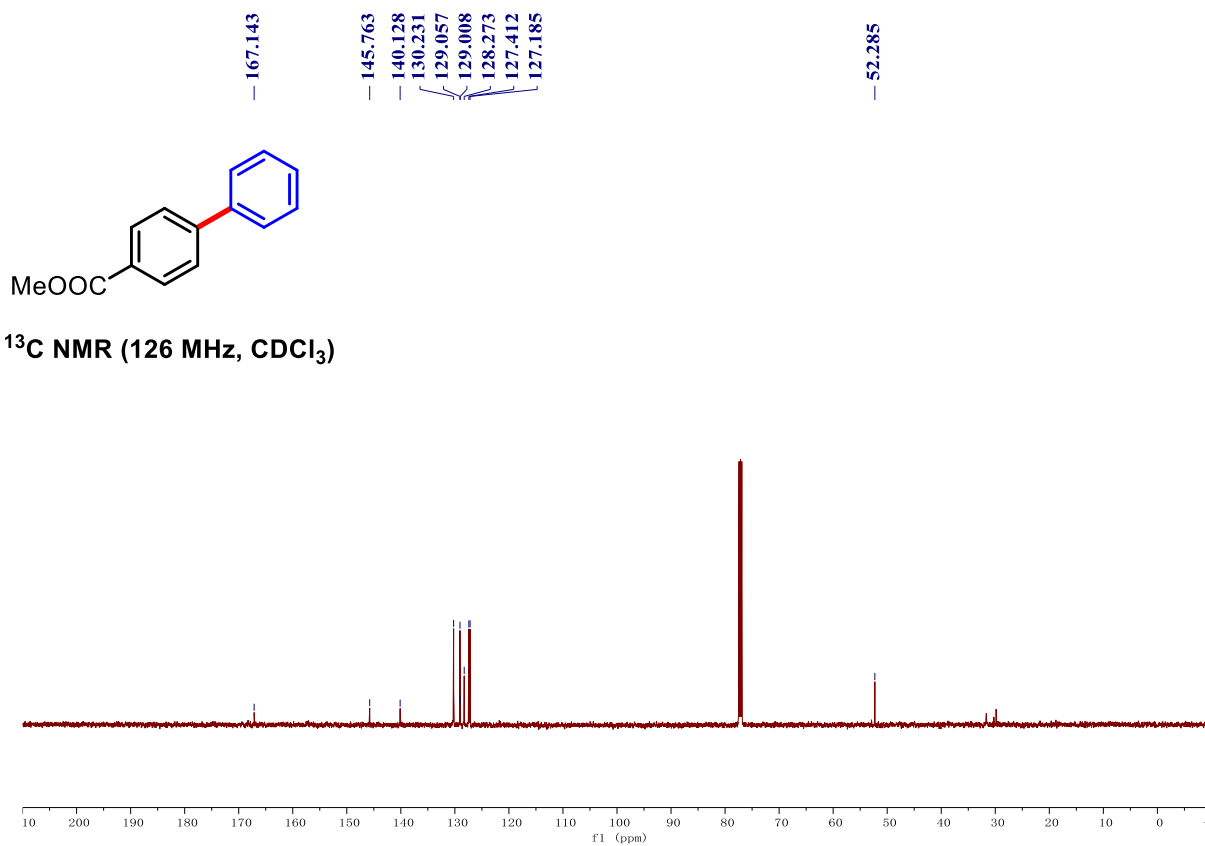




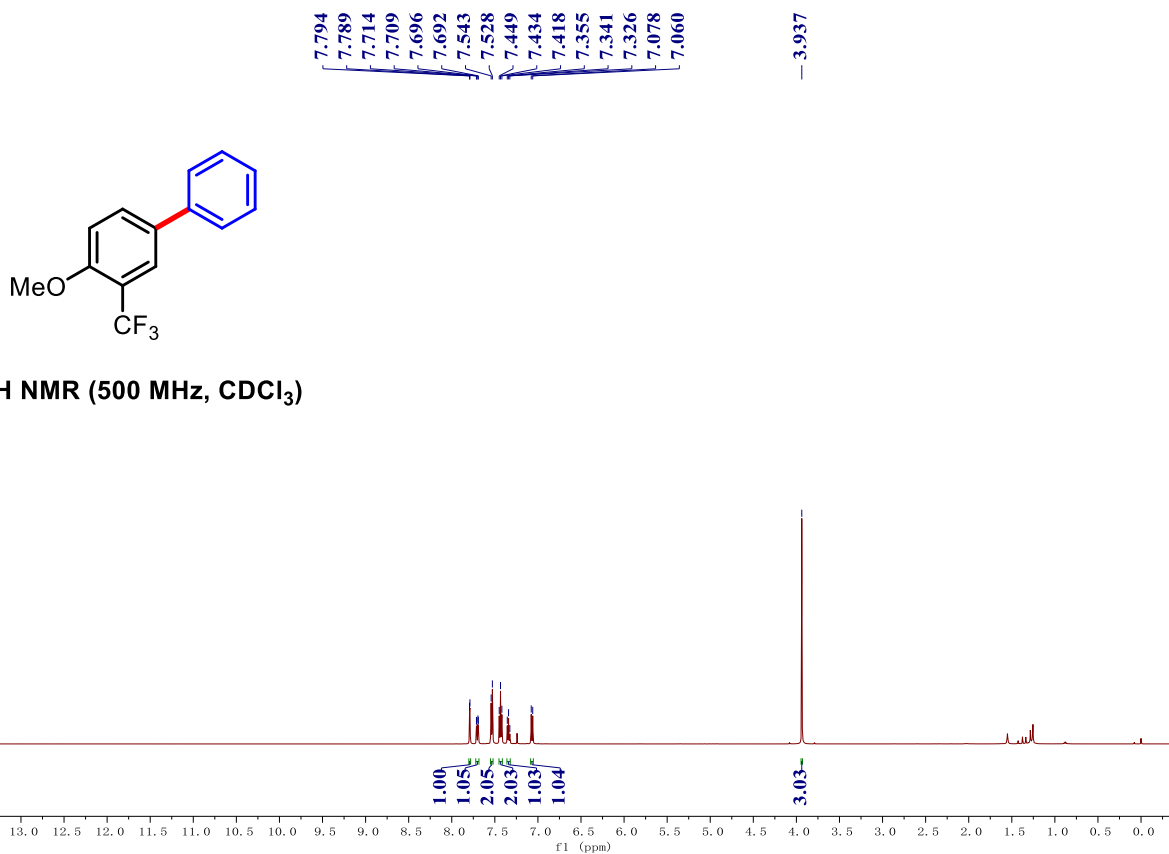


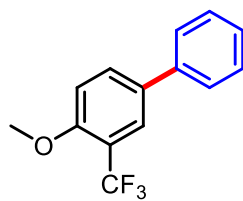
**Methyl [1,1'-biphenyl]-4-carboxylate, 3y**



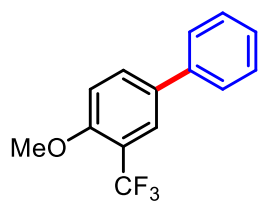
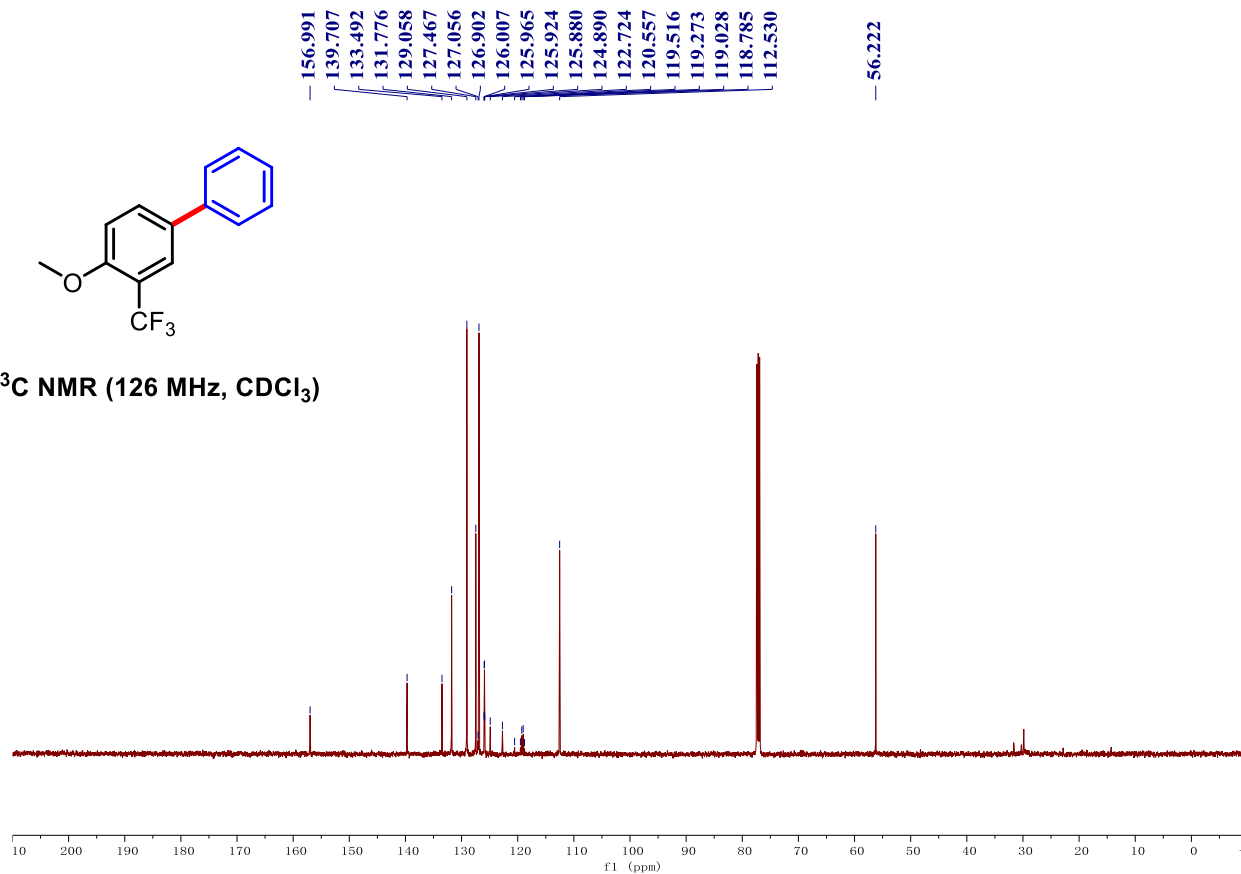


4-Methoxy-3-(trifluoromethyl)-1,1'-biphenyl, 3z

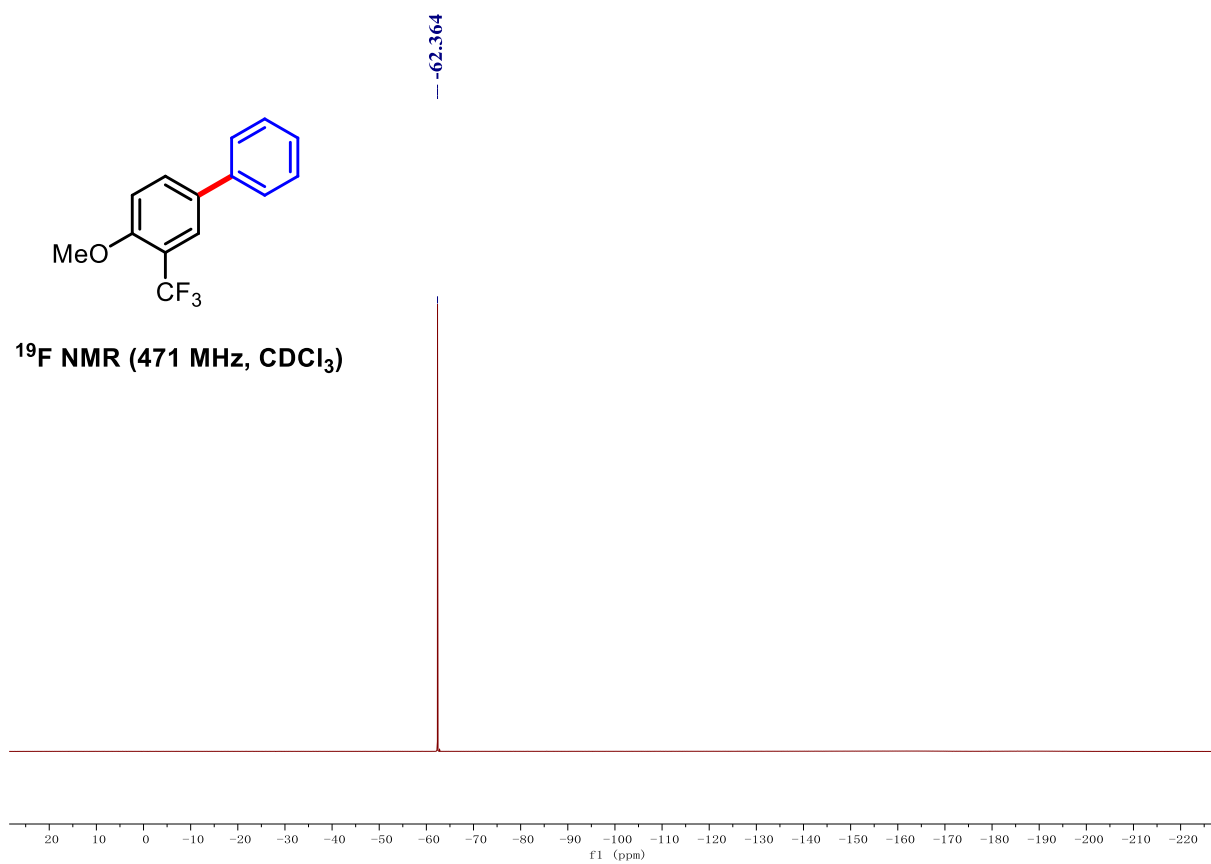




<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)

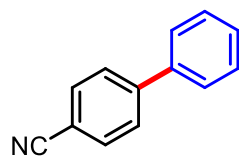


<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

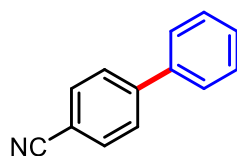
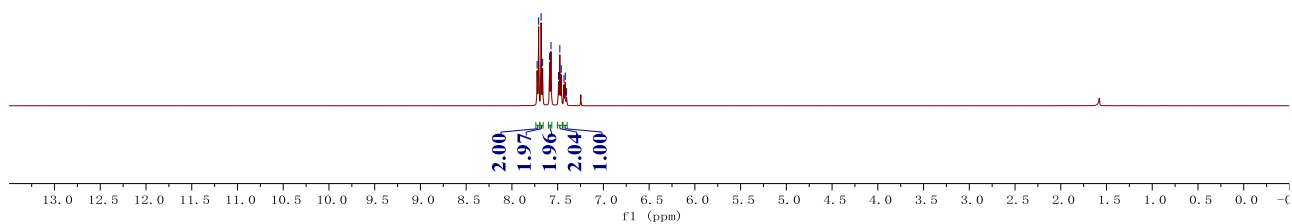


# [1,1'-Biphenyl]-4-carbonitrile, 3aa

7.725  
7.708  
7.681  
7.664  
7.587  
7.573  
7.490  
7.476  
7.461  
7.431  
7.417  
7.402

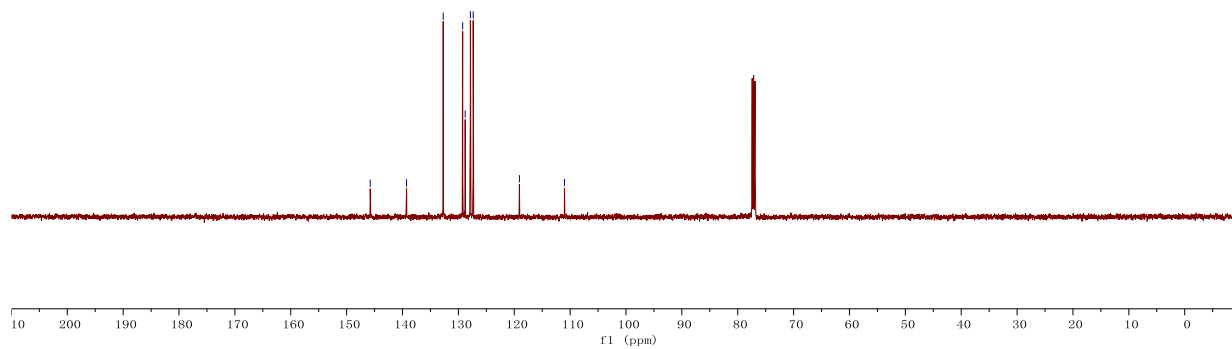


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



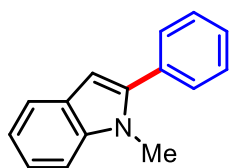
<sup>13</sup>C NMR (124 MHz, CDCl<sub>3</sub>)

145.780  
139.279  
132.713  
129.231  
128.780  
127.849  
127.346  
119.076  
111.010

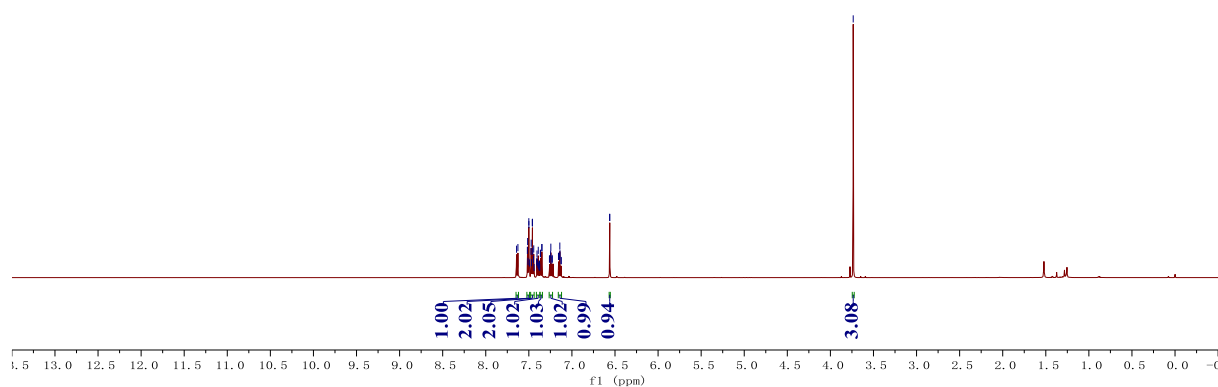


# 1-Methyl-2-phenyl-1H-indole, 3ab

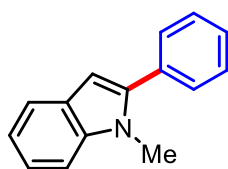
7.642  
7.627  
7.517  
7.514  
7.510  
7.500  
7.494  
7.475  
7.473  
7.469  
7.459  
7.459  
7.458  
7.455  
7.446  
7.443  
7.439  
7.408  
7.405  
7.402  
7.395  
7.391  
7.386  
7.379  
7.376  
7.373  
7.366  
7.364  
7.350  
7.348  
7.261  
7.259  
7.247  
7.245  
7.242  
7.231  
7.228  
7.156  
7.154  
7.142  
7.140  
7.138  
7.126  
7.124  
6.562  
6.561  
3.735



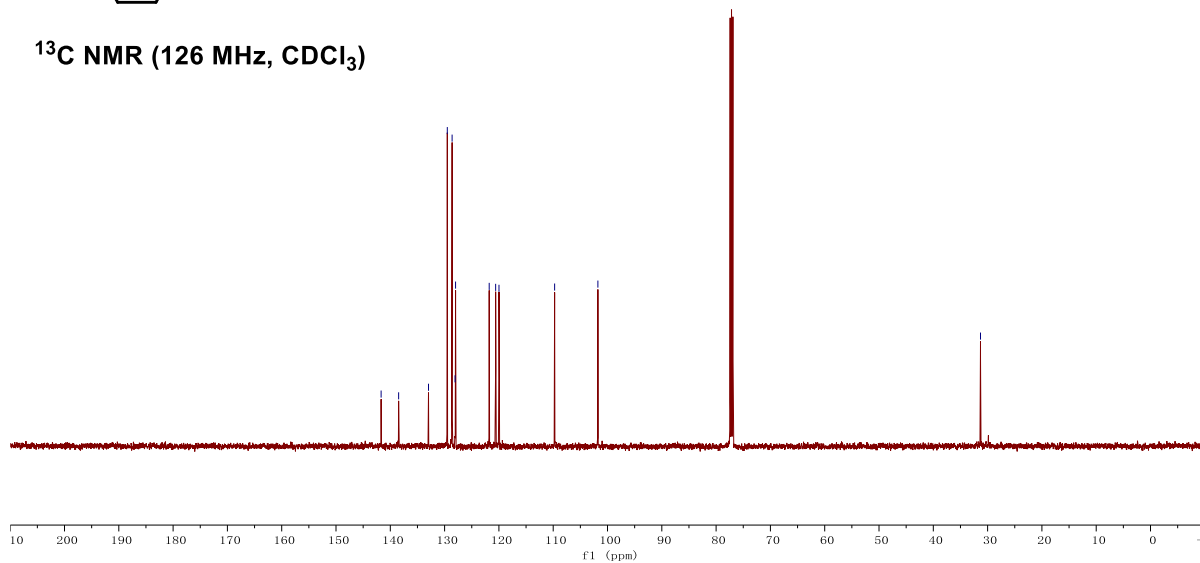
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



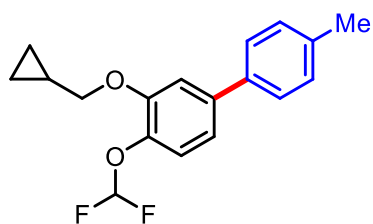
141.693  
138.466  
132.966  
129.504  
128.618  
128.077  
127.981  
121.788  
120.599  
119.984  
109.739  
101.771  
31.309



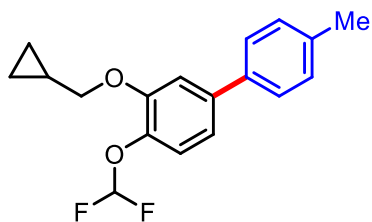
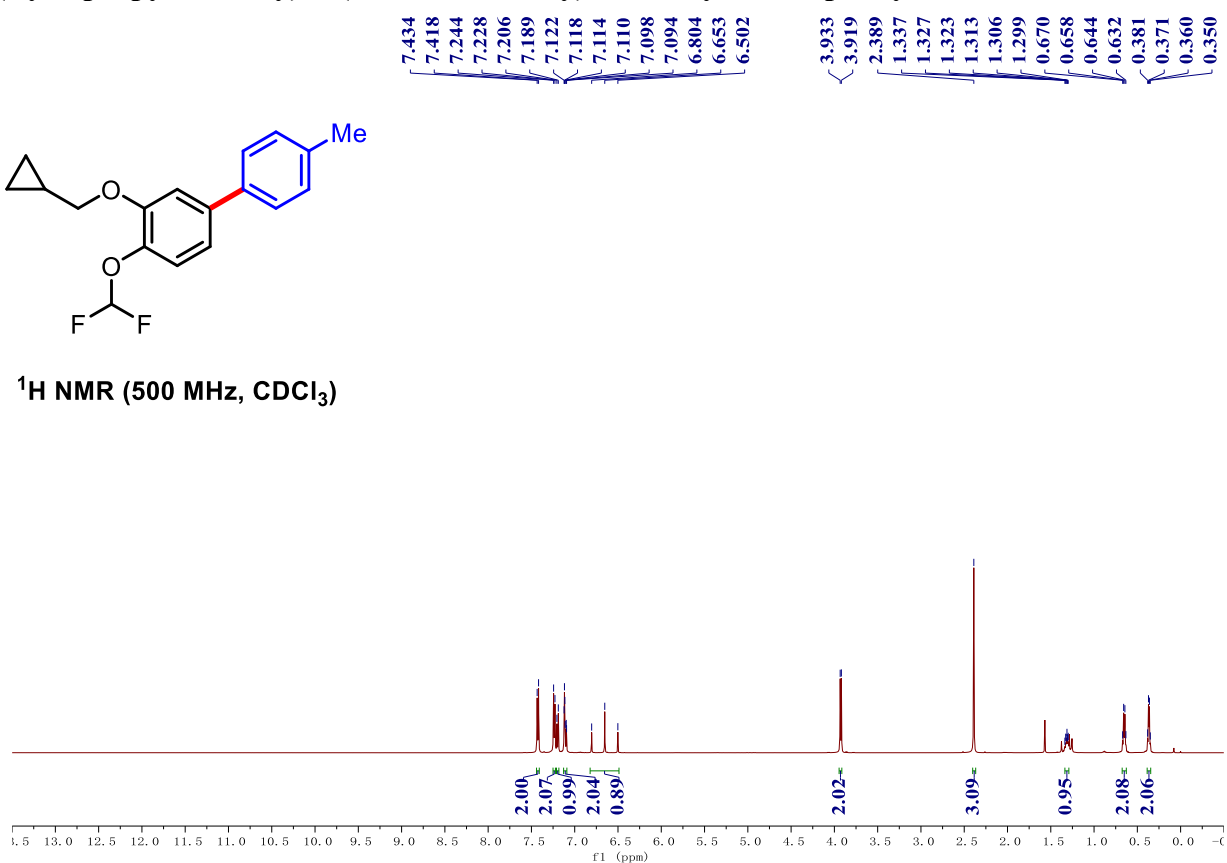
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)



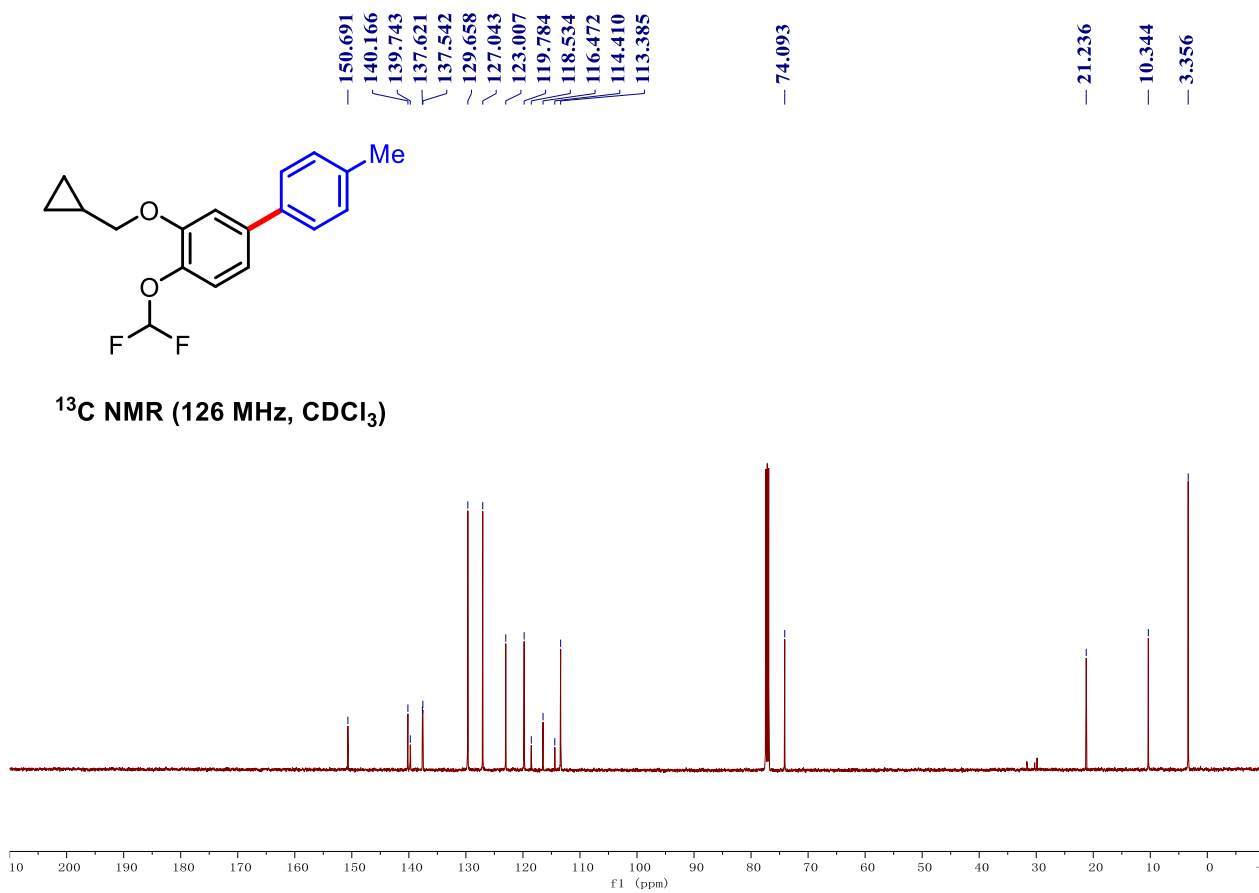
### 3-(Cyclopropylmethoxy)-4-(difluoromethoxy)-4'-methyl-1,1'-biphenyl, 3ac

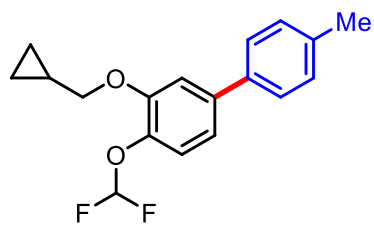


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



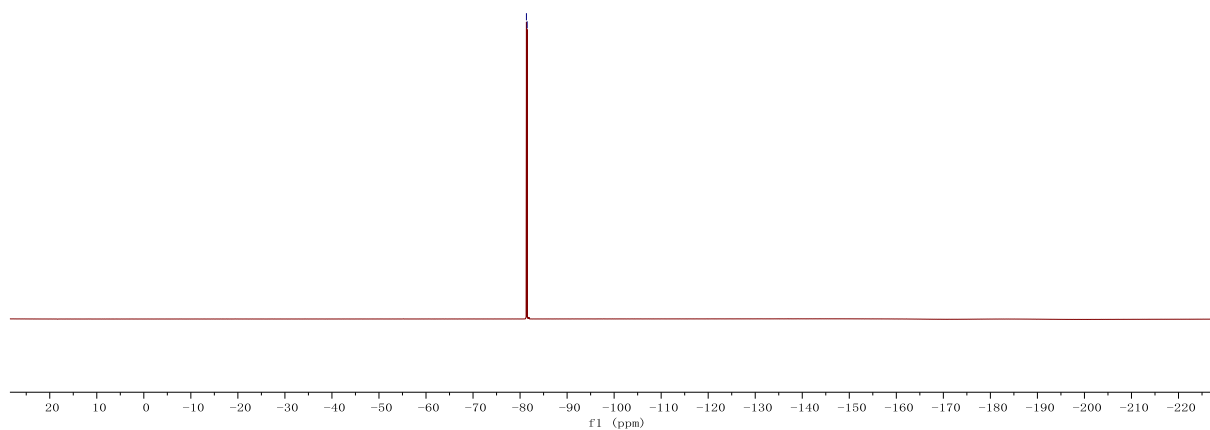
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)





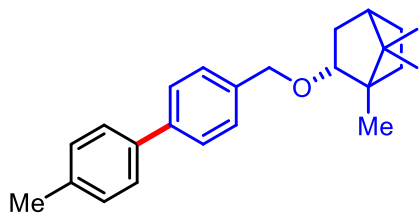
-81.354  
-81.515

<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

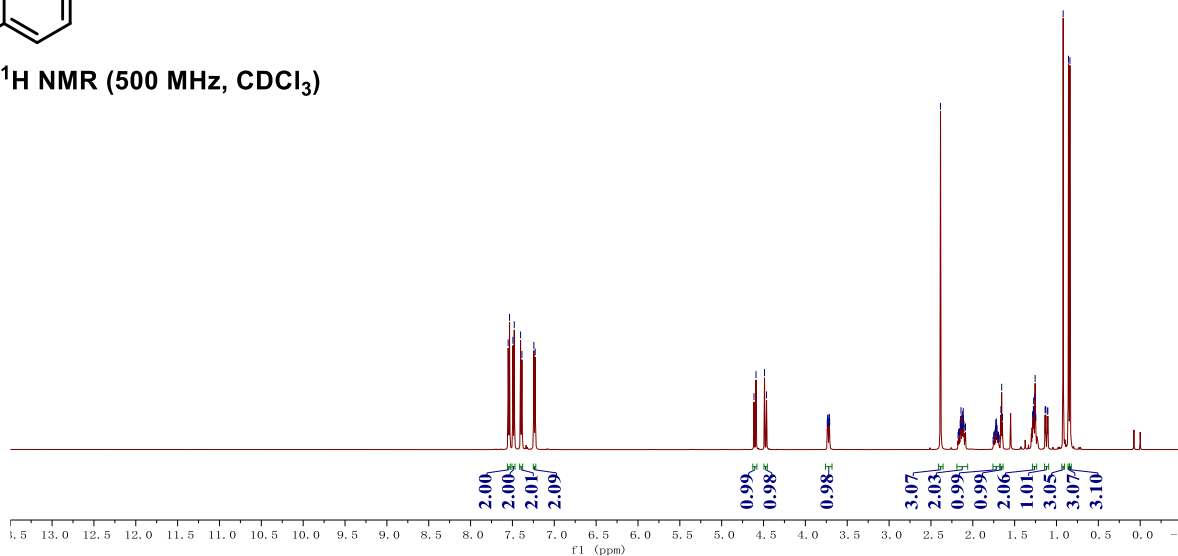


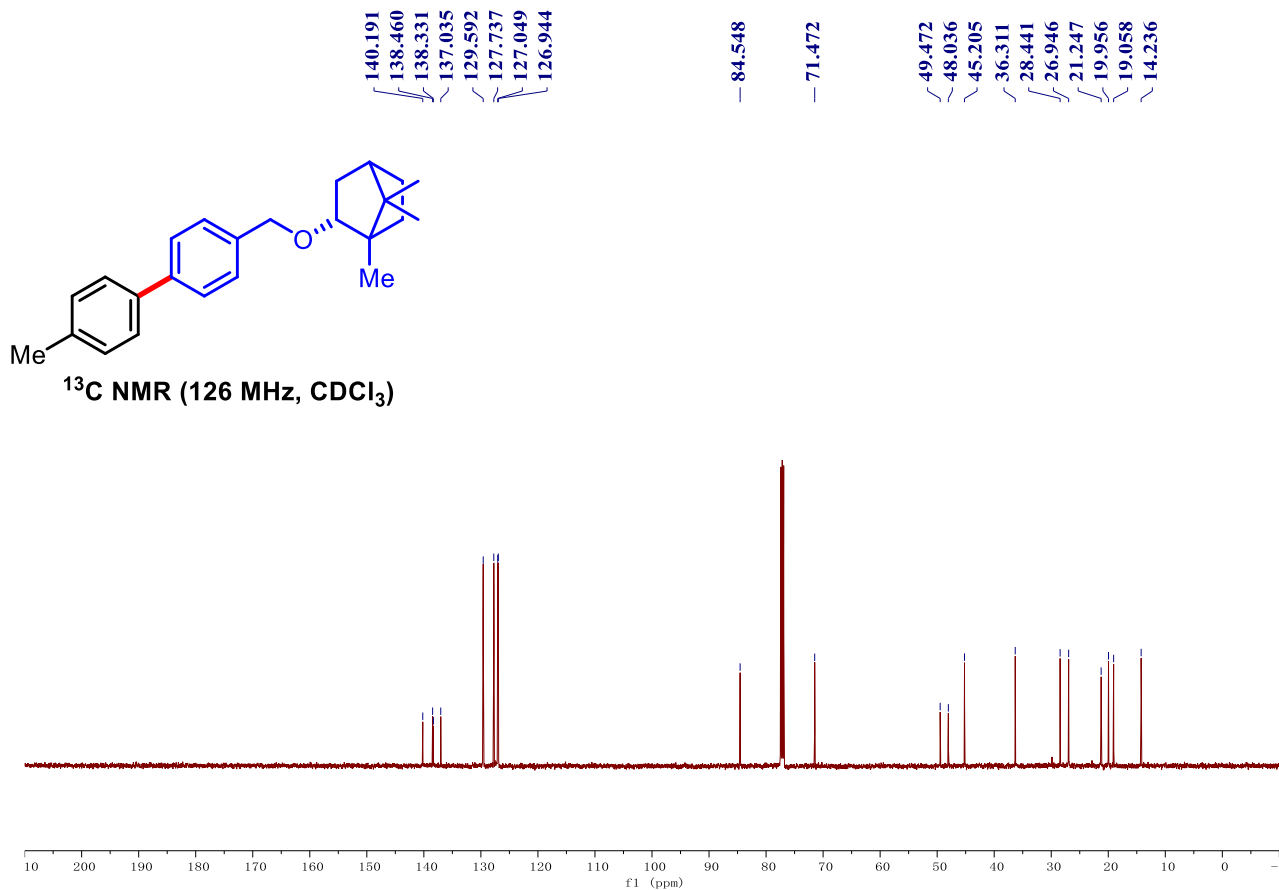
(2*R*)-1,7,7-Trimethyl-2-((4'-methyl-[1,1'-biphenyl]-4-yl)methoxy)bicyclo[2.2.1]heptane, 3ad

7.551  
7.535  
7.494  
7.478  
7.403  
7.386  
7.244  
7.228  
4.614  
4.590  
4.488  
4.464  
3.738  
3.735  
3.732  
3.729  
3.720  
3.716  
3.714  
3.710  
2.386  
2.150  
2.141  
2.134  
2.130  
2.125  
2.121  
2.115  
2.112  
2.105  
1.725  
1.719  
1.664  
1.655  
1.645  
1.284  
1.282  
1.276  
1.273  
1.267  
1.259  
1.255  
1.247  
1.136  
1.129  
1.110  
1.103  
0.920  
0.856  
0.839



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)





**2-(4'-Methyl-[1,1'-biphenyl]-4-yl)ethyl 2-(4-isobutylphenyl)propanoate, 3ae**

