

## Supporting information

### Hydroxyl Proton Transfer for the Deoxygenative Reduction of Alcohols Mediated by $\text{Ph}_3\text{P}/\text{ICH}_2\text{CH}_2\text{I}$

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### Contents

1	General information .....	S2
2	Optimization of reaction conditions .....	S2
3	Procedure for the Synthesis of alcohols .....	S4
4	General Procedures for Reductive Deoxygenation of Alcohols .....	S12
5	Mechanistic Investigations .....	S23
	5.1 The synthesis of deuterated substrate .....	S23
	5.2 Reductive deoxygenation of deuterated substrate .....	S23
6	Gram-Scale Reaction.....	S24
7	Copies of <sup>1</sup> H NMR, <sup>19</sup> F NMR and <sup>13</sup> C NMR Spectra of Substrates and Products .....	S25
8	References .....	S77

## 1 General information

$^1\text{H}$ ,  $^{13}\text{C}$  and  $^{19}\text{F}$  NMR spectra were detected on a 400 MHz spectrometers (400 MHz for  $^1\text{H}$ , 101 MHz for  $^{13}\text{C}$ , 376 MHz for  $^{19}\text{F}$  respectively). Data for  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR and  $^{19}\text{F}$  NMR were recorded as follows: chemical shift ( $\delta$ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, br = broad, coupling constant (s) in Hz). All reactions were monitored by TLC or  $^{19}\text{F}$  NMR. Flash column chromatography was carried out using 300-400 mesh silica gel at medium pressure. Mass spectra were obtained on GC-MS (EI). High resolution mass spectrometry (HRMS) was performed on JEOL-AccuTOF-MS instrument with electrospray ionization (ESI) positive ion mode. Oil baths serve as the heat sources for reactions that require controlled heating. Unless otherwise noted, all reagents and solvents were purchased from commercial sources and used without further purification.

## 2 Optimization of reaction conditions

We chose triphenylmethanol (**1-1**) as the model substrate to screen the reaction conditions (Table 1). During the solvent screening process, we observed that the nature of the solvent exerted a significant influence on the reaction yield (entries **1-4**, Table 1). Acetonitrile demonstrated a distinct advantage as the solvent (entry **4**, Table 1), affording the target product in good yield (77%) after stirring at room temperature overnight. With acetonitrile selected as the solvent, we proceeded to screen several representative organophosphines. The reaction proved feasible with triarylphosphines serving as the phosphorus source (entries **5-9**), and triphenylphosphine (entry **4**, Table 1) provided the best performance (77%). However, the introduction of methyl or methoxy substituents on the phenyl rings (entries **5-6**, Table 1) led to a sharp decline in yield, which may be attributed to increased nucleophilicity of the phosphorus center, potentially promoting competitive side reactions. A comparison between  $^n\text{Bu}_3\text{P}$  and  $^t\text{Bu}_3\text{P}$  as phosphorus sources (entries **7-8**, Table 1) revealed a pronounced influence of steric bulk on the reaction yield, where increased steric hindrance led to a marked decrease in efficiency. Notably,  $^n\text{Bu}_3\text{P}$  proved less effective than triphenylphosphine.

Replacing one phenyl group in triphenylphosphine with a methyl group still afforded the product in relatively high yield (entry **9**, Table 1). Based on our group's prior work, the R<sub>3</sub>P/ICH<sub>2</sub>CH<sub>2</sub>I system typically requires equimolar amounts of phosphine and ICH<sub>2</sub>CH<sub>2</sub>I for dehydroxylative functionalization. However, since no external hydrogen donor was used in the current transformation—suggesting a distinct mechanistic pathway—we hypothesized that equimolar quantities may not be necessary. The loadings of both Ph<sub>3</sub>P and ICH<sub>2</sub>CH<sub>2</sub>I were then screened. When the amount of R<sub>3</sub>P was fixed at 1.2 equiv, gradually reducing the equivalents of ICH<sub>2</sub>CH<sub>2</sub>I (entries **10-12**, Table 1) still maintained a relatively high product yield. Conversely, with ICH<sub>2</sub>CH<sub>2</sub>I held constant at 0.3 equiv, varying the loading of R<sub>3</sub>P consistently led to a decrease in yield (entry **13-15**, Table 1). Subsequently, a slight increase in temperature to 50 °C afforded the product in excellent yield, and further elevation of the temperature showed no additional benefit (entries **16-17**, Table 1). The transformation at 50 °C reached completion within 30 minutes (entries **18-19**, Table 1). Control experiments indicated that both Ph<sub>3</sub>P and ICH<sub>2</sub>CH<sub>2</sub>I are indispensable for the reaction to proceed (entries **21-22**, Table 1).

**Table 1** Optimization of Reaction Conditions<sup>a</sup>.

$$\text{Ph}_3\text{C-OH} \xrightarrow[\text{Solv., T } ^\circ\text{C, 10 h}]{[\text{P}], \text{ICH}_2\text{CH}_2\text{I}} \text{Ph}_3\text{C-H}$$

Entry	Solv.	R <sub>3</sub> P	Molar ratio <sup>b</sup>	T/°C	Yield (%) <sup>c</sup>
1	NMP	Ph <sub>3</sub> P	1:1.2:1.2	rt	32
2	DMF	Ph <sub>3</sub> P	1:1.2:1.2	rt	27
3	DMSO	Ph <sub>3</sub> P	1:1.2:1.2	rt	12
4	MeCN	Ph <sub>3</sub> P	1:1.2:1.2	rt	77
5	MeCN	( <i>p</i> -MePh) <sub>3</sub> P	1:1.2:1.2	rt	12
6	MeCN	( <i>p</i> -MeOPh) <sub>3</sub> P	1:1.2:1.2	rt	35
7	MeCN	<sup>n</sup> Bu <sub>3</sub> P	1:1.2:1.2	rt	61
8	MeCN	<sup>t</sup> Bu <sub>3</sub> P	1:1.2:1.2	rt	1
9	MeCN	Ph <sub>2</sub> PCH <sub>3</sub>	1:1.2:1.2	rt	68
10	MeCN	Ph <sub>3</sub> P	1:1.2:0.9	rt	95
11	MeCN	Ph <sub>3</sub> P	1:1.2:0.6	rt	86
12	MeCN	Ph <sub>3</sub> P	1:1.2:0.3	rt	88
13	MeCN	Ph <sub>3</sub> P	1:1.0:0.3	rt	62

14	MeCN	Ph <sub>3</sub> P	1:1.4:0.3	rt	85
15	MeCN	Ph <sub>3</sub> P	1:1.6:0.3	rt	42
16	MeCN	Ph <sub>3</sub> P	1:1.2:0.3	50	95
17	MeCN	Ph <sub>3</sub> P	1:1.2:0.3	70	94
18 <sup>d</sup>	MeCN	Ph <sub>3</sub> P	1:1.2:0.3	50	94
19 <sup>e</sup>	MeCN	Ph <sub>3</sub> P	1:1.2:0.3	50	95
20 <sup>f</sup>	MeCN	Ph <sub>3</sub> P	1:1.2:0.3	50	60
21	MeCN	Ph <sub>3</sub> P	1:1.2:0	rt	0
22	MeCN	Ph <sub>3</sub> P	1:0:0.3	rt	0

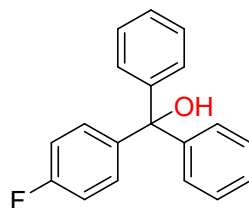
<sup>a</sup>Reaction conditions: substrate **1-1** (0.2 mmol), R<sub>3</sub>P and ICH<sub>2</sub>CH<sub>2</sub>I in MeCN (2 mL) at the specified temperature for 10 h without inert atmosphere protection. <sup>b</sup>Molar ratio of **1-1** : R<sub>3</sub>P : ICH<sub>2</sub>CH<sub>2</sub>I. <sup>c</sup>Yields were determined by GC-MS. <sup>d</sup>The reaction was performed at 50 °C for 1 h. <sup>e</sup>The reaction was performed at 50 °C for 0.5 h. <sup>f</sup>I<sub>2</sub> was used instead of ICH<sub>2</sub>CH<sub>2</sub>I under the conditions described in entry 19, using 0.5 mmol of substrate. The yield shown is the isolated yield.

### 3 Procedure for the Synthesis of alcohols



A 15 mL Schlenk tube was charged with ketone (1.0 equiv., 2.0 mmol,) and THF (4.0 mL). The solution was cooled by an ice bath and stirred under an argon atmosphere. Phenyl magnesium bromide solution (1.0 M in THF, 1.2 equiv., 2.4 mmol, 2.4 mL) was added dropwise and the reaction was stirred under an Ar atmosphere at rt overnight until the conversion was complete (monitored by TLC). The reaction was quenched with saturated aq. NH<sub>4</sub>Cl (5.0 mL) and extracted with EtOAc (3×10 mL). The combined extracts were dried with Na<sub>2</sub>SO<sub>4</sub>, and the solvent was removed under vacuum. The residue was purified by flash chromatography on silica gel to give the target product, **1**. The spectral data obtained were in accordance with those described in the literature.

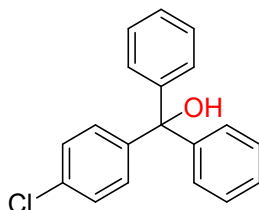
#### (4-Fluorophenyl)diphenylmethanol (**1-2**)<sup>1</sup>



Ph-MgBr was used as the Grignard reagent. **1-2** was obtained in 80% yield (445.6 mg). White solid (eluent: 40:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.28 – 7.13

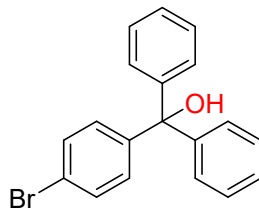
(m, 12H), 6.89 (t,  $J = 8.6$  Hz, 2H), 3.03 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  161.9 (d,  $J = 246.3$  Hz), 146.8, 142.8 (d,  $J = 3.2$  Hz), 129.8 (d,  $J = 8.0$  Hz), 128.1, 127.9, 127.4, 114.7 (d,  $J = 21.2$  Hz), 81.8.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -115.18 – -115.32 (m, 1F). GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{15}\text{FO}$   $[\text{M}]^+$ : 278.1, found: 278.1.

#### (4-Chlorophenyl)diphenylmethanol (**1-3**)<sup>1</sup>



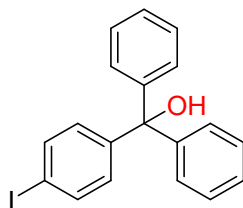
Ph-MgBr was used as the Grignard reagent. **1-3** was obtained in 55% yield (322.7 mg). Yellowish oil (eluent: 20:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.28 – 7.14 (m, 14H), 2.99 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  146.5, 145.4, 133.2, 129.5, 128.11, 128.06, 127.9, 127.5, 81.8. GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{15}\text{ClO}$   $[\text{M}]^+$ : 294.1, found: 294.1.

#### (4-Bromophenyl)diphenylmethanol (**1-4**)<sup>1</sup>



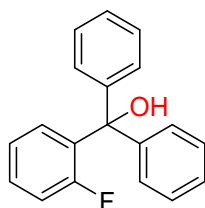
Ph-MgBr was used as the Grignard reagent. **1-4** was obtained in 74% yield (504.6 mg). Colorless oil (eluent: 50:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.35 (d,  $J = 8.5$  Hz, 2H), 7.27 – 7.16 (m, 10H), 7.10 (d,  $J = 8.5$  Hz, 2H), 3.00 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  146.3, 145.9, 131.0, 129.8, 128.1, 127.9, 127.5, 121.4, 81.8. GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{15}\text{BrO}$   $[\text{M}]^+$ : 338.0, found: 338.0.

#### (4-Iodophenyl)diphenylmethanol (**1-5**)<sup>2</sup>



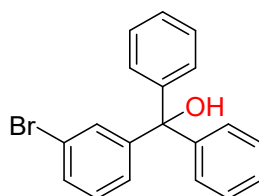
Ph-MgBr was used as the Grignard reagent. **1-5** was obtained in 67% yield (519.5 mg). Colorless oil (eluent: 40:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.56 (d,  $J$  = 8.0 Hz, 2H), 7.31 – 7.13 (m, 10H), 6.99 (d,  $J$  = 8.1 Hz, 2H), 2.92 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  146.6, 146.3, 137.0, 130.0, 128.1, 127.9, 127.5, 93.3, 81.8. GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{15}\text{IO}$   $[\text{M}]^+$ : 386.0, found: 386.0.

#### (2-Fluorophenyl)diphenylmethanol (**1-6**)<sup>3</sup>



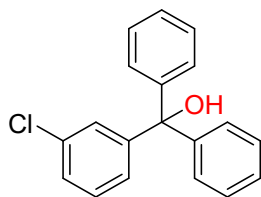
Ph-MgBr was used as the Grignard reagent. **1-6** was obtained in 75% yield (416.7 mg). White solid (eluent: 40:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  7.38 – 7.22 (m, 12H), 7.20 – 7.14 (m, 1H), 7.12 – 7.06 (m, 1H), 6.45 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  159.6 (d,  $J$  = 248.4 Hz), 146.3, 134.5 (d,  $J$  = 11.3 Hz), 129.5 (d,  $J$  = 8.4 Hz), 129.0 (d,  $J$  = 3.7 Hz), 127.5, 127.3, 126.7, 123.7 (d,  $J$  = 3.3 Hz), 116.0 (d,  $J$  = 22.7 Hz), 78.8.  $^{19}\text{F}$  NMR (376 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  -105.30 – -105.46 (m, 1F). GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{15}\text{FO}$   $[\text{M}]^+$ : 278.1, found: 278.1.

#### (3-Bromophenyl)diphenylmethanol (**1-8**)<sup>4</sup>



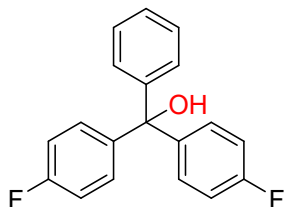
Ph-MgBr was used as the Grignard reagent. **1-8** was obtained in 61% yield (411.5 mg). Colorless oil (eluent: 50:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.51 (t,  $J$  = 1.9 Hz, 1H), 7.39 – 7.32 (m, 1H), 7.31 – 7.17 (m, 10H), 7.18 – 7.11 (m, 1H), 7.09 (t,  $J$  = 7.8 Hz, 1H), 2.91 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  149.2, 146.3, 130.9, 130.4, 129.5, 128.2, 127.9, 127.6, 126.8, 122.4, 81.7. GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{15}\text{BrO}$   $[\text{M}]^+$ : 338.0, found: 338.1.

#### (3-Chlorophenyl)diphenylmethanol (**1-9**)<sup>4</sup>



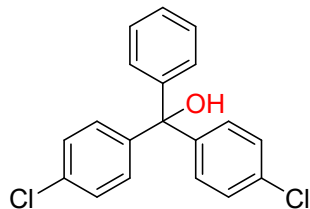
Ph-MgBr was used as the Grignard reagent. **1-9** was obtained in 98% yield (576.9 mg). Colorless oil (eluent: 70:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.36 – 7.31 (m, 1H), 7.27 – 7.16 (m, 11H), 7.15 – 7.08 (m, 2H), 2.94 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  148.9, 146.4, 134.0, 129.2, 128.14, 128.06, 127.9, 127.6, 127.4, 126.3, 81.8. GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{15}\text{ClO}$   $[\text{M}]^+$ : 294.1, found: 294.1.

### Bis(4-fluorophenyl)(phenyl)methanol (**1-10**)<sup>3</sup>



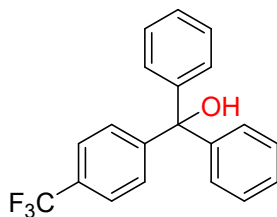
Ph-MgBr was used as the Grignard reagent. **1-10** was obtained in 88% yield (523.0 mg). White solid (eluent: 50:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.34 – 7.27 (m, 3H), 7.25 – 7.18 (m, 6H), 7.03 – 6.93 (m, 4H), 2.80 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.1 (d,  $J = 246.7$  Hz), 146.7, 142.7 (d,  $J = 3.2$  Hz), 129.8 (d,  $J = 7.7$  Hz), 128.3, 127.8, 127.7, 114.9 (d,  $J = 21.3$  Hz), 81.4.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -115.17 – -115.27 (m, 2F). GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{14}\text{F}_2\text{O}$   $[\text{M}]^+$ : 296.1, found: 296.1.

### Bis(4-chlorophenyl)(phenyl)methanol (**1-11**)<sup>5</sup>



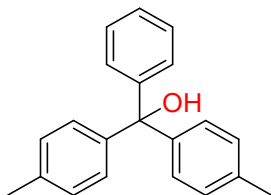
Ph-MgBr was used as the Grignard reagent. **1-11** was obtained in 91% yield (600.5 mg). Colorless oil (eluent: 50:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.33 – 7.22 (m, 7H), 7.21 – 7.13 (m, 6H), 2.87 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  146.2, 145.0, 133.6, 129.4, 128.4, 128.3, 127.9, 127.8, 81.4. GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{14}\text{Cl}_2\text{O}$   $[\text{M}]^+$ : 328.0, found: 328.0.

### Diphenyl(4-(trifluoromethyl)phenyl)methanol (**1-14**)<sup>6</sup>



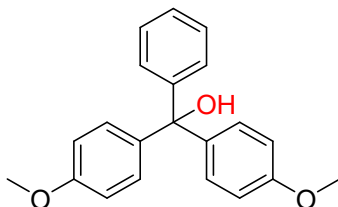
Ph-MgBr was used as the Grignard reagent. **1-14** was obtained in 62% yield (403.9 mg). Colorless oil (eluent: 20:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.49 (d, *J* = 8.2 Hz, 2H), 7.39 (d, *J* = 8.2 Hz, 2H), 7.28 – 7.22 (m, 6H), 7.22 – 7.20 (m, 4H), 3.10 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 150.7 (q, *J* = 1.5 Hz), 146.3, 129.4 (q, *J* = 32.3 Hz), 128.4, 128.3, 128.0, 127.8, 125.0 (q, *J* = 3.6 Hz), 124.3 (d, *J* = 272.1 Hz), 82.0. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -62.28 (s, 3F). GC-MS (EI) calcd. for C<sub>20</sub>H<sub>15</sub>F<sub>3</sub>O [M]<sup>+</sup>: 328.1, found: 328.1.

### Bis(4-methylphenyl)(phenyl)methanol (**1-16**)<sup>3</sup>



Ph-MgBr was used as the Grignard reagent. **1-16** was obtained in 73% yield (421.4 mg). White solid (eluent: 50:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.24 (m, 5H), 7.18 – 7.07 (m, 8H), 2.72 (s, 1H), 2.33 (s, 6H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 147.3, 143.7, 137.0, 128.7, 127.99, 127.98, 127.97, 127.2, 81.9, 21.2. GC-MS (EI) calcd. for C<sub>21</sub>H<sub>20</sub>O [M]<sup>+</sup>: 288.2, found: 288.1.

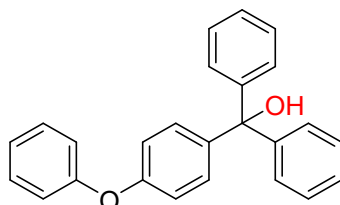
### Bis(4-methoxyphenyl)(phenyl)methanol (**1-19**)<sup>7</sup>



Ph-MgBr was used as the Grignard reagent. **1-19** was obtained in 62% yield (395.9 mg). Colorless oil (eluent: 5:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.20 (m, 5H), 7.16 – 7.11 (m, 4H), 6.81 – 6.76 (m, 4H), 3.73 (s, 6H), 2.94 (s, 1H). <sup>13</sup>C

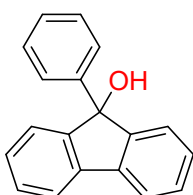
NMR (101 MHz, Chloroform-*d*)  $\delta$  158.6, 147.4, 139.6, 129.2, 127.89, 127.86, 127.1, 113.2, 81.5, 55.3. GC-MS (EI) calcd. for C<sub>21</sub>H<sub>20</sub>O<sub>3</sub> [M]<sup>+</sup>: 320.1, found: 320.2.

**(4-Phenoxyphenyl)diphenylmethanol (1-21)<sup>8</sup>**



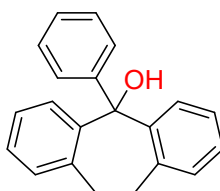
Ph-MgBr was used as the Grignard reagent. **1-21** was obtained in 83% yield (588.9 mg). Colorless oil (eluent: 30:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.29 – 7.14 (m, 14H), 7.03 (t, *J* = 7.4 Hz, 1H), 6.97 (d, *J* = 8.0 Hz, 2H), 6.86 (d, *J* = 8.5 Hz, 2H), 2.95 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  156.9, 156.5, 146.9, 141.7, 129.8, 129.6, 128.0, 127.9, 127.3, 123.5, 119.2, 117.9, 81.8. HRMS (ESI) calcd. for C<sub>25</sub>H<sub>20</sub>O<sub>2</sub>Na [M + Na]<sup>+</sup>: 375.1356, found: 375.1350.

**9-Phenyl-9H-fluoren-9-ol (1-22)<sup>9</sup>**



Ph-MgBr was used as the Grignard reagent. **1-22** was obtained in 98% yield (506.0 mg). White solid (eluent: 50:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.61 (d, *J* = 7.5 Hz, 2H), 7.35 – 7.15 (m, 11H), 2.50 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  150.5, 143.2, 139.6, 129.1, 128.5, 128.3, 127.2, 125.5, 124.9, 120.1, 83.6. GC-MS (EI) calcd. for C<sub>19</sub>H<sub>14</sub>O [M]<sup>+</sup>: 258.1, found: 258.1.

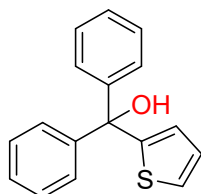
**5-Phenyl-10,11-dihydro-5H-dibenzo[a,d][7]annulen-5-ol (1-23)<sup>10</sup>**



Ph-MgBr was used as the Grignard reagent. **1-23** was obtained in 76% yield (435.4 mg). White solid (eluent: 60:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.06 (d, *J* = 7.8 Hz, 2H), 7.31 – 7.16 (m, 7H), 7.07 (d, *J* = 7.3 Hz, 2H), 7.04 – 6.97 (m, 2H),

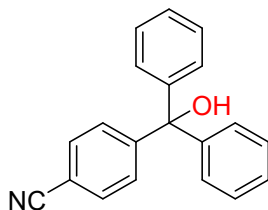
2.94 – 2.82 (m, 2H), 2.75 – 2.64 (m, 2H), 2.32 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 148.6, 143.6, 137.9, 130.7, 128.8, 127.8, 127.7, 126.7, 126.0, 125.7, 79.5, 32.6. GC-MS (EI) calcd. for C<sub>21</sub>H<sub>18</sub>O [M]<sup>+</sup>: 286.1, found: 286.1.

#### Diphenyl(thiophen-2-yl)methanol (**1-27**)<sup>11</sup>



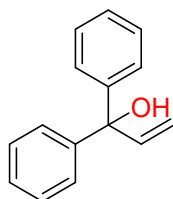
Ph-MgBr was used as the Grignard reagent. **1-27** was obtained in 86% yield (460.5 mg). White solid (eluent: 40:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.39 – 7.25 (m, 11H), 6.94 (dd, *J* = 5.1, 3.6 Hz, 1H), 6.72 (dd, *J* = 3.6, 1.2 Hz, 1H), 2.96 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 152.4, 146.7, 128.1, 127.7, 127.4, 127.0, 126.6, 125.8, 80.2. GC-MS (EI) calcd. for C<sub>17</sub>H<sub>14</sub>OS [M]<sup>+</sup>: 266.1, found: 266.1.

#### 4-(Hydroxydiphenylmethyl)benzonitrile (**1-28**)<sup>4</sup>



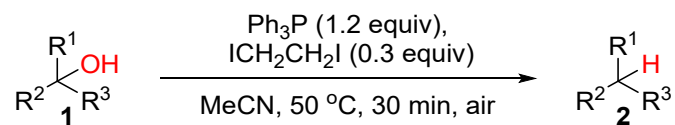
Ph-MgBr was used as the Grignard reagent. **1-28** was obtained in 83% yield (475.6 mg). Pale yellow solid (eluent: 20:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.63 – 7.56 (m, 2H), 7.51 – 7.43 (m, 2H), 7.37 – 7.28 (m, 6H), 7.24 – 7.20 (m, 4H), 2.88 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 152.0, 145.9, 131.9, 128.7, 128.4, 128.0, 127.9, 118.9, 111.1, 81.9. GC-MS (EI) calcd. for C<sub>20</sub>H<sub>15</sub>NO [M]<sup>+</sup>: 285.1, found: 285.1.

#### 1,1-Diphenylprop-2-en-1-ol (**1-29**)<sup>12</sup>



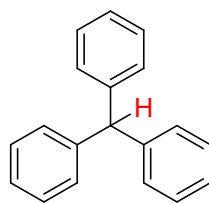
CH<sub>2</sub>=CH-MgBr was used as the Grignard reagent. **1-29** was obtained in 81% yield (340.8 mg). Colorless oil (eluent: 50:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.31 (m, 4H), 7.29 – 7.23 (m, 4H), 7.22 – 7.17 (m, 2H), 6.49 – 6.38 (m, 1H), 5.28 – 5.22 (m, 2H), 2.44 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 145.8, 143.5, 128.2, 127.3, 127.0, 114.1, 79.5. GC-MS (EI) calcd. for C<sub>15</sub>H<sub>14</sub>O [M]<sup>+</sup>: 210.1, found: 210.1.

## 4 General Procedures for Reductive Deoxygenation of Alcohols



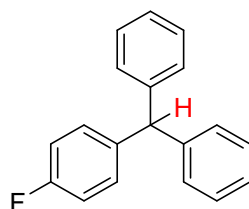
Into a 15 mL sealed tube were added Ph<sub>3</sub>P (0.6 mmol, 157.4 mg, 1.2 equiv.) and alcohol **1** (0.5 mmol, 1.0 equiv.), and MeCN (5 mL) under air atmosphere, followed by the addition of ICH<sub>2</sub>CH<sub>2</sub>I (0.15 mmol, 42.3 mg, 0.3 equiv.). After complete dissolution of all reagents, the reaction tube was sealed and the mixture was stirred at 50°C for 30 min. Upon completion of the reaction, the mixture was concentrated under reduced pressure. The resulting crude material was purified by flash column chromatography to afford target product **2**.

### Triphenylmethane (2-1)<sup>6</sup>



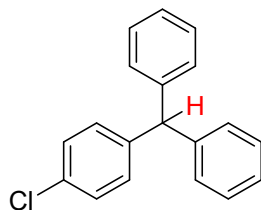
**2-1** was obtained in 81% yield (98.9 mg). White solid (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.28 (t, *J* = 7.2 Hz, 6H), 7.21 (t, *J* = 7.2 Hz, 3H), 7.12 (d, *J* = 7.1 Hz, 6H), 5.55 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 144.0, 129.6, 128.4, 126.4, 57.0. GC-MS (EI) calcd. for C<sub>19</sub>H<sub>16</sub> [M]<sup>+</sup>: 244.1, found: 244.1.

### ((4-Fluorophenyl)methylene)dibenzene (2-2)<sup>6</sup>



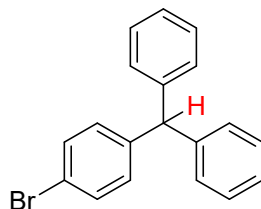
**2-2** was obtained in 78% yield (102.8 mg). White solid (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.18 (m, 6H), 7.17 – 7.01 (m, 6H), 7.01 – 6.91 (m, 2H), 5.52 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 161.6 (d, *J* = 244.8 Hz), 143.9, 139.8 (d, *J* = 3.2 Hz), 131.0 (d, *J* = 7.8 Hz), 129.5, 128.5, 126.6, 115.2 (d, *J* = 21.3 Hz), 56.2. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -116.81 – -116.95 (m, 1F). GC-MS (EI) calcd. for C<sub>19</sub>H<sub>15</sub>F [M]<sup>+</sup>: 262.1, found: 262.1.

**((4-Chlorophenyl)methylene)dibenzene (2-3)<sup>6</sup>**



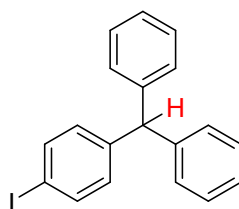
**2-3** was obtained in 79% yield (109.9 mg). Colorless oil (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.15 (m, 8H), 7.10 – 7.04 (m, 4H), 7.03 – 6.98 (m, 2H), 5.48 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 143.5, 142.6, 132.3, 130.9, 129.5, 128.6, 126.6, 56.3. GC-MS (EI) calcd. for C<sub>19</sub>H<sub>15</sub>Cl [M]<sup>+</sup>: 278.1, found: 278.1.

**((4-Bromophenyl)methylene)dibenzene (2-4)<sup>6</sup>**



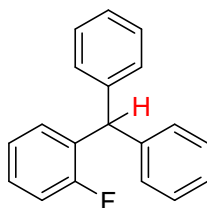
**2-4** was obtained in 71% yield (115.0 mg). White solid (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.38 (d, *J* = 8.0 Hz, 2H), 7.31 – 7.17 (m, 6H), 7.08 (d, *J* = 7.3 Hz, 4H), 6.97 (d, *J* = 8.0 Hz, 2H), 5.48 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 143.4, 143.1, 131.5, 131.3, 129.5, 128.6, 126.7, 120.4, 56.4. GC-MS (EI) calcd. for C<sub>19</sub>H<sub>15</sub>Br [M]<sup>+</sup>: 322.0, found: 322.0.

**((4-Iodophenyl)methylene)dibenzene (2-5)<sup>13</sup>**



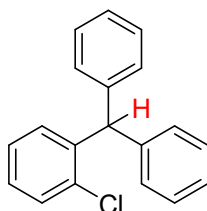
**2-5** was obtained in 83% yield (154.3 mg). White solid (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.56 (d, *J* = 8.3 Hz, 2H), 7.28 – 7.14 (m, 6H), 7.06 (d, *J* = 7.0 Hz, 4H), 6.83 (d, *J* = 8.3 Hz, 2H), 5.46 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 143.8, 143.3, 137.5, 131.6, 129.4, 128.5, 126.6, 92.0, 56.4. GC-MS (EI) calcd. for C<sub>19</sub>H<sub>15</sub>I [M]<sup>+</sup>: 370.0, found: 370.1.

**((2-Fluorophenyl)methylene)dibenzene (2-6)<sup>14</sup>**



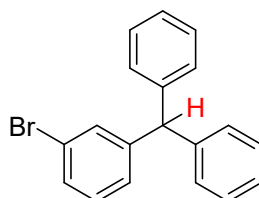
**2-6** was obtained in 73% yield (95.3 mg). White solid (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.06 (m, 11H), 6.98 (t, *J* = 8.6 Hz, 2H), 6.92 (t, *J* = 7.7 Hz, 1H), 5.83 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 160.9 (d, *J* = 246.9 Hz), 142.7, 131.2 (d, *J* = 14.1 Hz), 131.0 (d, *J* = 3.9 Hz), 129.4, 128.5, 128.3 (d, *J* = 8.2 Hz), 126.6, 124.0 (d, *J* = 3.7 Hz), 115.5 (d, *J* = 22.3 Hz), 49.5 (d, *J* = 3.3 Hz). <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -116.00 – -116.11 (m). GC-MS (EI) calcd. for C<sub>19</sub>H<sub>15</sub>F [M]<sup>+</sup>: 262.1, found: 262.1.

**((2-Chlorophenyl)methylene)dibenzene (2-7)<sup>15</sup>**



**2-7** was obtained in 62% yield (86.8 mg). White solid (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.40 – 7.33 (m, 1H), 7.30 – 7.18 (m, 6H), 7.17 – 7.11 (m, 2H), 7.10 – 7.04 (m, 4H), 6.97 – 6.91 (m, 1H), 5.96 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 142.7, 141.8, 134.8, 131.3, 129.8, 129.7, 128.5, 127.9, 126.7, 126.6, 53.5. GC-MS (EI) calcd. for C<sub>19</sub>H<sub>15</sub>Cl [M]<sup>+</sup>: 278.1, found: 278.0.

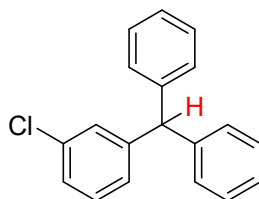
**((3-Bromophenyl)methylene)dibenzene (2-8)<sup>16</sup>**



**2-8** was obtained in 64% yield (103.7 mg). Colorless oil (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.31 (m, 1H), 7.31 – 7.24 (m, 5H), 7.24 – 7.18 (m, 2H), 7.15 – 7.06 (m, 5H), 7.02 (d, *J* = 7.8 Hz, 1H), 5.49 (s, 1H). <sup>13</sup>C NMR (101 MHz,

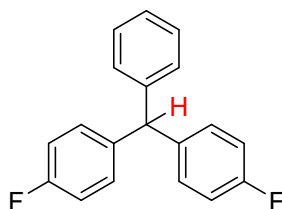
Chloroform-*d*)  $\delta$  146.4, 143.2, 132.5, 130.0, 129.6, 129.5, 128.6, 128.3, 126.7, 122.7, 56.6. GC-MS (EI) calcd. for  $C_{19}H_{15}Br$   $[M]^+$ : 322.0, found: 322.1.

**((3-Chlorophenyl)methylene)dibenzene (2-9)<sup>17</sup>**



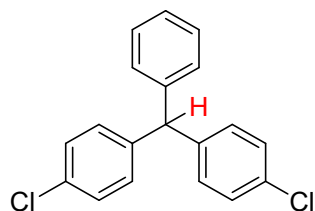
**2-9** was obtained in 77% yield (107.4 mg). Colorless oil (eluent: PE).  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.30 – 7.25 (m, 4H), 7.23 – 7.16 (m, 4H), 7.13 – 7.05 (m, 5H), 7.01 – 6.96 (m, 1H), 5.50 (s, 1H).  $^{13}C$  NMR (101 MHz, Chloroform-*d*)  $\delta$  146.2, 143.2, 134.4, 129.66, 129.65, 129.5, 128.6, 127.8, 126.71, 126.70, 56.6. GC-MS (EI) calcd. for  $C_{19}H_{15}Cl$   $[M]^+$ : 278.1, found: 278.1.

**4,4'-(Phenylmethylene)bis(fluorobenzene) (2-10)<sup>18</sup>**



**2-10** was obtained in 73% yield (101.7 mg). Colorless oil (eluent: PE).  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.27 (t,  $J = 7.3$  Hz, 2H), 7.24 – 7.18 (m, 1H), 7.09 – 7.00 (m, 6H), 6.95 (t,  $J = 8.7$  Hz, 4H), 5.49 (s, 1H).  $^{13}C$  NMR (101 MHz, Chloroform-*d*)  $\delta$  161.6 (d,  $J = 245.2$  Hz), 143.7, 139.6 (d,  $J = 3.2$  Hz), 130.9 (d,  $J = 7.8$  Hz), 129.4, 128.6, 126.7, 115.3 (d,  $J = 21.0$  Hz), 55.4.  $^{19}F$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -116.40 – -116.56 (m, 2H). GC-MS (EI) calcd. for  $C_{19}H_{14}F_2$   $[M]^+$ : 280.1, found: 280.1.

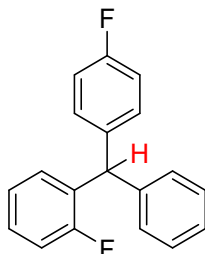
**4,4'-(Phenylmethylene)bis(chlorobenzene) (2-11)<sup>19</sup>**



**2-11** was obtained in 58% yield (89.4 mg). Colorless oil (eluent: PE).  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.32 – 7.18 (m, 7H), 7.05 (d,  $J = 7.0$  Hz, 2H), 7.01 (d,  $J = 8.4$

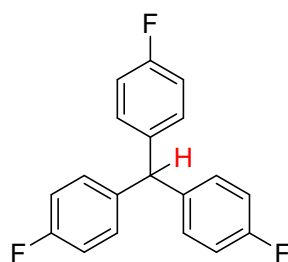
Hz, 4H), 5.47 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  143.0, 142.1, 132.5, 130.8, 129.4, 128.70, 128.69, 126.9, 55.6. GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{14}\text{Cl}_2$   $[\text{M}]^+$ : 312.0, found: 312.0.

### 1-Fluoro-2-((4-fluorophenyl)(phenyl)methyl)benzene (2-12)<sup>20</sup>



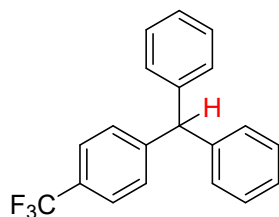
**2-12** was obtained in 72% yield (100.8 mg). White solid (eluent: PE).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.32 – 7.17 (m, 4H), 7.13 – 6.86 (m, 9H), 5.80 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  161.7 (d,  $J = 245.0$  Hz), 160.8 (d,  $J = 246.9$  Hz), 142.5, 138.5 (d,  $J = 3.3$  Hz), 131.1 (d,  $J = 14.3$  Hz), 130.84 (d,  $J = 4.3$  Hz), 130.82 (d,  $J = 7.5$  Hz), 129.3, 128.6, 128.5 (d,  $J = 8.2$  Hz), 126.8, 124.1 (d,  $J = 3.7$  Hz), 115.6 (d,  $J = 22.2$  Hz), 115.3 (d,  $J = 21.3$  Hz), 48.8 (d,  $J = 3.2$  Hz).  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -116.27 – -116.39 (m, 1H), -116.45 – -116.63 (m, 1H). GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{14}\text{F}_2$   $[\text{M}]^+$ : 280.1, found: 280.1.

### Tris(4-fluorophenyl)methane (2-13)<sup>21</sup>



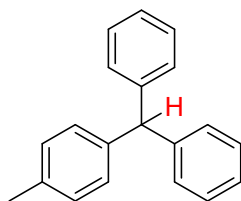
**2-13** was obtained in 75% yield (111.7 mg). Colorless oil (eluent: PE).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.05 – 6.99 (m, 6H), 6.99 – 6.92 (m, 6H), 5.48 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  161.7 (d,  $J = 245.6$  Hz), 139.4 (d,  $J = 3.3$  Hz), 130.8 (d,  $J = 8.2$  Hz), 115.4 (d,  $J = 21.3$  Hz), 54.6.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -116.15 – -116.27 (m, 3H). GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{13}\text{F}_3$   $[\text{M}]^+$ : 298.1, found: 298.0.

### ((4-(Trifluoromethyl)phenyl)methylene)dibenzene (2-14)<sup>6</sup>



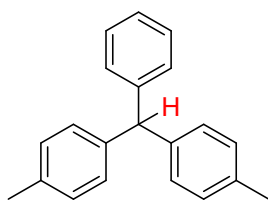
**2-14** was obtained in 85% yield (132.1 mg). Colorless oil (eluent: PE).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.51 (d,  $J = 8.1$  Hz, 2H), 7.30 – 7.24 (m, 4H), 7.23 – 7.16 (m, 4H), 7.08 (d,  $J = 7.2$  Hz, 4H), 5.57 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  148.2, 143.1, 129.9, 129.5, 128.8 (q,  $J = 32.6$  Hz), 128.7, 126.8, 125.4 (q,  $J = 3.8$  Hz), 124.4 (q,  $J = 272.0$  Hz), 56.8.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -62.2 (s, 3F). GC-MS (EI) calcd. for  $\text{C}_{20}\text{H}_{15}\text{F}_3$   $[\text{M}]^+$ : 312.1, found: 312.1.

**(*p*-Tolylmethylene)dibenzene (2-15)<sup>6</sup>**



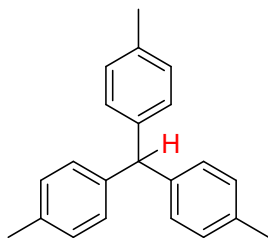
**2-15** was obtained in 83% yield (107.2 mg). White solid (eluent: PE).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.28 – 7.21 (m, 4H), 7.20 – 7.14 (m, 2H), 7.12 – 7.05 (m, 6H), 7.02 – 6.97 (m, 2H), 5.50 (s, 1H), 2.29 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  144.2, 141.1, 135.9, 129.5, 129.4, 129.1, 128.4, 126.3, 56.6, 21.1. GC-MS (EI) calcd. for  $\text{C}_{20}\text{H}_{18}$   $[\text{M}]^+$ : 258.1, found: 258.1.

**4,4'-(Phenylmethylene)bis(methylbenzene) (2-16)<sup>22</sup>**



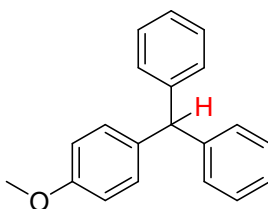
**2-16** was obtained in 74% yield (100.9 mg). Colorless oil (eluent: PE).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.25 (t,  $J = 7.3$  Hz, 2H), 7.20 – 7.14 (m, 1H), 7.13 – 7.04 (m, 6H), 6.99 (d,  $J = 7.9$  Hz, 4H), 5.46 (s, 1H), 2.30 (s, 6H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  144.5, 141.3, 135.8, 129.5, 129.4, 129.1, 128.4, 126.3, 56.2, 21.1. GC-MS (EI) calcd. for  $\text{C}_{21}\text{H}_{20}$   $[\text{M}]^+$ : 272.2, found: 272.1.

### Tri-*p*-tolylmethane (2-17)<sup>22</sup>



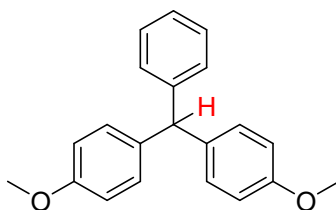
**2-17** was obtained in 79% yield (113.0 mg). Colorless oil (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.06 (d, *J* = 8.0 Hz, 6H), 6.99 (d, *J* = 8.1 Hz, 6H), 5.42 (s, 1H), 2.29 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 141.5, 135.7, 129.4, 129.1, 55.8, 21.1. GC-MS (EI) calcd. for C<sub>22</sub>H<sub>22</sub> [M]<sup>+</sup>: 286.2, found: 286.1.

### ((4-Methoxyphenyl)methylene)dibenzene (2-18)<sup>6</sup>



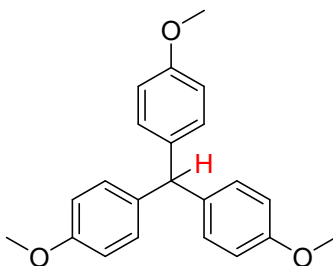
**2-18** was obtained in 91% yield (125.4 mg). White solid (eluent: 100:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.25 (t, *J* = 7.5 Hz, 4H), 7.20 – 7.14 (m, 2H), 7.10 (d, *J* = 7.5 Hz, 4H), 7.01 (d, *J* = 8.7 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 5.48 (s, 1H), 3.73 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 158.1, 144.4, 136.2, 130.5, 129.5, 128.4, 126.3, 113.8, 56.1, 55.3. GC-MS (EI) calcd. for C<sub>20</sub>H<sub>18</sub>O [M]<sup>+</sup>: 274.1, found: 274.1.

### 4,4'-(Phenylmethylene)bis(methoxybenzene) (2-19)<sup>23</sup>



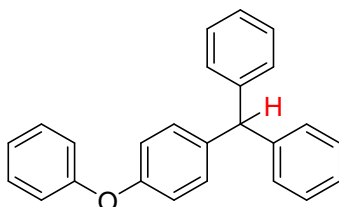
**2-19** was obtained in 57% yield (87.3 mg). Colorless oil (eluent: 20:1 PE / EA). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.24 (t, *J* = 7.4 Hz, 2H), 7.19 – 7.15 (m, 1H), 7.09 (d, *J* = 7.6 Hz, 2H), 7.00 (d, *J* = 8.7 Hz, 4H), 6.80 (d, *J* = 8.7 Hz, 4H), 5.43 (s, 1H), 3.73 (s, 6H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 158.1, 144.7, 136.5, 130.4, 129.4, 128.4, 126.2, 113.7, 55.29, 55.28. GC-MS (EI) calcd. for C<sub>21</sub>H<sub>20</sub>O<sub>2</sub> [M]<sup>+</sup>: 304.1, found: 304.1.

### Tris(4-methoxyphenyl)methane (2-20)<sup>21</sup>



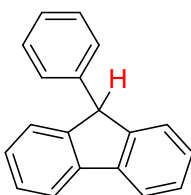
**2-20** was obtained in 53% yield (88.9 mg). Colorless oil (eluent: 30:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.03 – 6.97 (m, 6H), 6.83 – 6.77 (m, 6H), 5.39 (s, 1H), 3.75 (s, 9H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  158.1, 136.9, 130.3, 113.8, 55.3, 54.5. GC-MS (EI) calcd. for  $\text{C}_{22}\text{H}_{22}\text{O}_3$   $[\text{M}]^+$ : 334.2, found: 334.1.

**((4-Phenoxyphenyl)methylene)dibenzene (2-21)<sup>24</sup>**



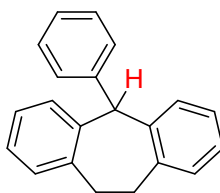
**2-21** was obtained in 89% yield (149.6 mg). White solid (eluent: 100:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.36 – 7.25 (m, 6H), 7.24 – 7.18 (m, 2H), 7.15 – 7.04 (m, 7H), 7.03 – 6.97 (m, 2H), 6.95 – 6.89 (m, 2H), 5.53 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  157.4, 155.8, 144.1, 138.9, 130.8, 129.8, 129.5, 128.5, 126.5, 123.3, 119.0, 118.8, 56.3. HRMS (ESI) calcd. for  $\text{C}_{25}\text{H}_{20}\text{ONa}$   $[\text{M} + \text{Na}]^+$ : 359.1406, found: 359.1399.

**9-Phenyl-9H-fluorene (2-22)<sup>25</sup>**



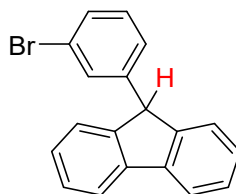
**2-22** was obtained in 85% yield (103.3 mg). White solid (eluent: PE).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.79 (d,  $J = 7.6$  Hz, 2H), 7.37 (t,  $J = 7.4$  Hz, 2H), 7.30 (d,  $J = 7.4$  Hz, 2H), 7.28 – 7.20 (m, 5H), 7.08 (d,  $J = 6.8$  Hz, 2H), 5.04 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  148.0, 141.7, 141.1, 128.8, 128.5, 127.4, 127.0, 125.5, 120.0, 54.6. GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{14}$   $[\text{M}]^+$ : 242.1, found: 242.0.

### 5-Phenyl-10,11-dihydro-5H-dibenzo[a,d][7]annulene (2-23)<sup>26</sup>



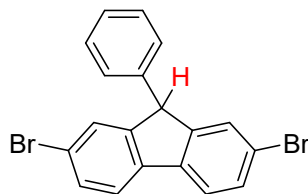
**2-23** was obtained in 80% yield (108.3 mg). White solid (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.27 (m, 2H), 7.24 – 7.07 (m, 9H), 6.88 (d, *J* = 7.5 Hz, 2H), 5.21 (s, 1H), 3.16 – 2.97 (m, 2H), 2.81 – 2.62 (m, 2H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 145.6, 140.4, 140.0, 131.6, 130.9, 128.2, 127.4, 126.2, 125.8, 58.4, 32.4. GC-MS (EI) calcd. for C<sub>21</sub>H<sub>18</sub> [M]<sup>+</sup>: 270.1, found: 270.1.

### 9-(3-Bromophenyl)-9H-fluorene (2-24)<sup>27</sup>



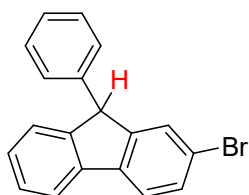
**2-24** was obtained in 80% yield (127.7 mg). White solid (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.77 (d, *J* = 7.5 Hz, 2H), 7.40 – 7.31 (m, 3H), 7.29 – 7.20 (m, 5H), 7.09 (t, *J* = 7.8 Hz, 1H), 6.98 (d, *J* = 7.7 Hz, 1H), 4.95 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 147.2, 144.2, 141.1, 131.4, 130.4, 130.1, 127.7, 127.6, 127.2, 125.4, 122.8, 120.1, 54.0. GC-MS (EI) calcd. for C<sub>19</sub>H<sub>13</sub>Br [M]<sup>+</sup>: 320.0, found: 320.0.

### 2,7-Dibromo-9-phenyl-9H-fluorene (2-25)<sup>28</sup>



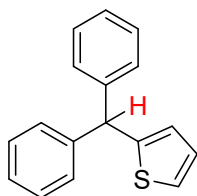
**2-25** was obtained in 65% yield (130.9 mg). White solid (eluent: PE). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 8.1 Hz, 2H), 7.50 (dd, *J* = 8.1, 1.8 Hz, 2H), 7.41 (s, 2H), 7.32 – 7.25 (m, 3H), 7.04 (dd, *J* = 7.6, 1.9 Hz, 2H), 4.98 (s, 1H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 149.7, 139.8, 139.1, 130.9, 129.2, 128.8, 128.4, 127.6, 121.6, 121.4, 54.3. GC-MS (EI) calcd. for C<sub>19</sub>H<sub>12</sub>Br<sub>2</sub> [M]<sup>+</sup>: 397.9, found: 399.8.

### 2-Bromo-9-phenyl-9H-fluorene (2-26)<sup>29</sup>



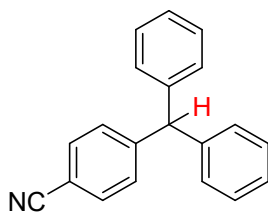
**2-26** was obtained in 69% yield (111.5 mg). White solid (eluent: PE).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.76 (d,  $J = 7.6$  Hz, 1H), 7.64 (d,  $J = 8.1$  Hz, 1H), 7.49 (dd,  $J = 8.1, 1.8$  Hz, 1H), 7.42 (s, 1H), 7.42 – 7.33 (m, 1H), 7.32 – 7.19 (m, 5H), 7.12 – 7.00 (m, 2H), 5.01 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  150.0, 147.7, 140.8, 140.1, 130.6, 129.0, 128.7, 128.4, 127.9, 127.7, 127.3, 125.5, 121.3, 121.2, 120.1, 54.5. GC-MS (EI) calcd. for  $\text{C}_{19}\text{H}_{13}\text{Br}$   $[\text{M}]^+$ : 320.0, found: 319.9.

#### 2-Benzhydrylthiophene (2-27)<sup>30</sup>



**2-27** was obtained in 7% yield (9.2 mg). Colorless oil (eluent: PE).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.33 – 7.27 (m, 4H), 7.26 – 7.18 (m, 6H), 6.93 (dd,  $J = 5.1, 3.5$  Hz, 1H), 6.69 (dt,  $J = 3.5, 1.2$  Hz, 1H), 5.68 (s, 1H). GC-MS (EI) calcd. for  $\text{C}_{17}\text{H}_{14}\text{S}$   $[\text{M}]^+$ : 250.1, found: 250.1.

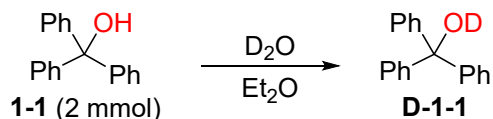
#### 4-Benzhydrylbenzonitrile (2-28)<sup>31</sup>



**2-28** was obtained in 42% yield (56.6 mg). Pale yellow oil (eluent: 50:1 PE / EA).  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.56 (d,  $J = 8.1$  Hz, 2H), 7.33 – 7.19 (m, 8H), 7.07 (d,  $J = 6.9$  Hz, 4H), 5.58 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  149.6, 142.5, 132.2, 130.3, 129.4, 128.7, 127.0, 119.0, 110.4, 56.9. GC-MS (EI) calcd. for  $\text{C}_{20}\text{H}_{15}\text{N}$   $[\text{M}]^+$ : 269.1, found: 269.1.

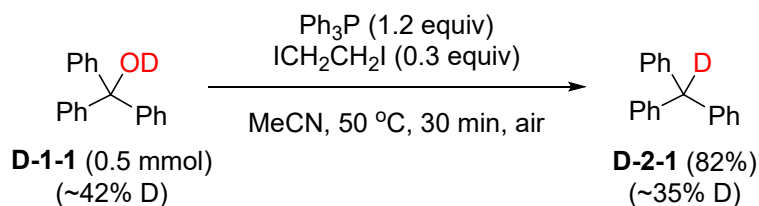
## 5 Mechanistic Investigations

### 5.1 The synthesis of deuterated substrate



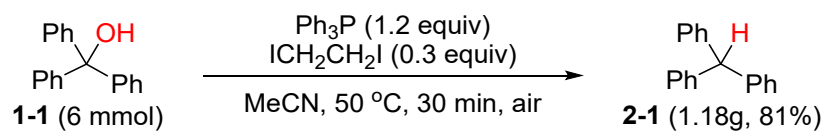
Deuterium labeling was performed following a literature method: In a separatory funnel, the substrate (2 mmol, 520.6 mg) in anhydrous  $Et_2O$  (8 mL) was shaken with ten successive portions of  $D_2O$  (0.8 mL each) to facilitate H/D exchange. The ethereal phase was separated, dried over anhydrous  $Na_2SO_4$  to remove residual  $D_2O$ , filtered, and concentrated. The solid residue was dried at  $60^\circ C$  to afford *d*-triphenylmethanol (**D-1-1**). Quantitative D NMR analysis of the product (**D-1-1**) indicated 42% deuterium incorporation.

### 5.2 Reductive deoxygenation of deuterated substrate



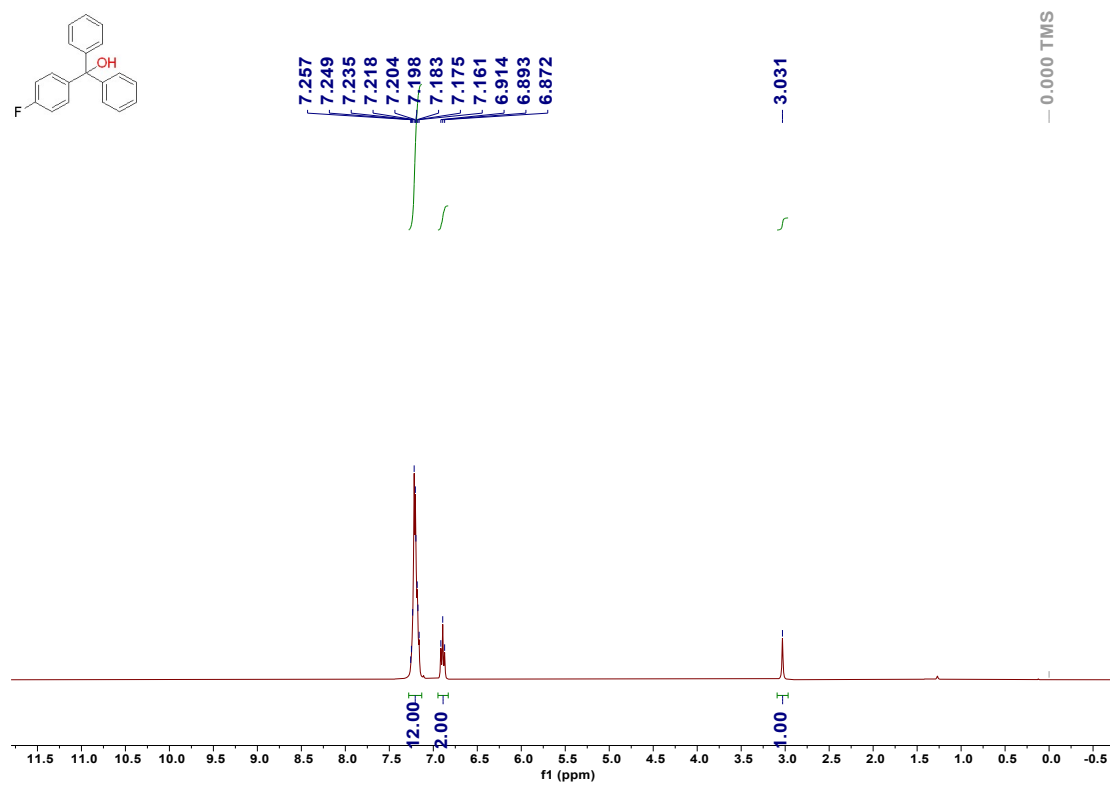
When **D-1-1** (42% D) was subjected to the reductive deoxygenation under optimal conditions, it yielded D-triphenylmethane (**D-2-1**) with 82% yield and 35% deuterium incorporation (determined by Quantitative D NMR analysis). This significant retention of the deuterium label confirms that the hydrogen incorporated during the reaction originates directly from the substrate.

## 6 Gram-Scale Reaction

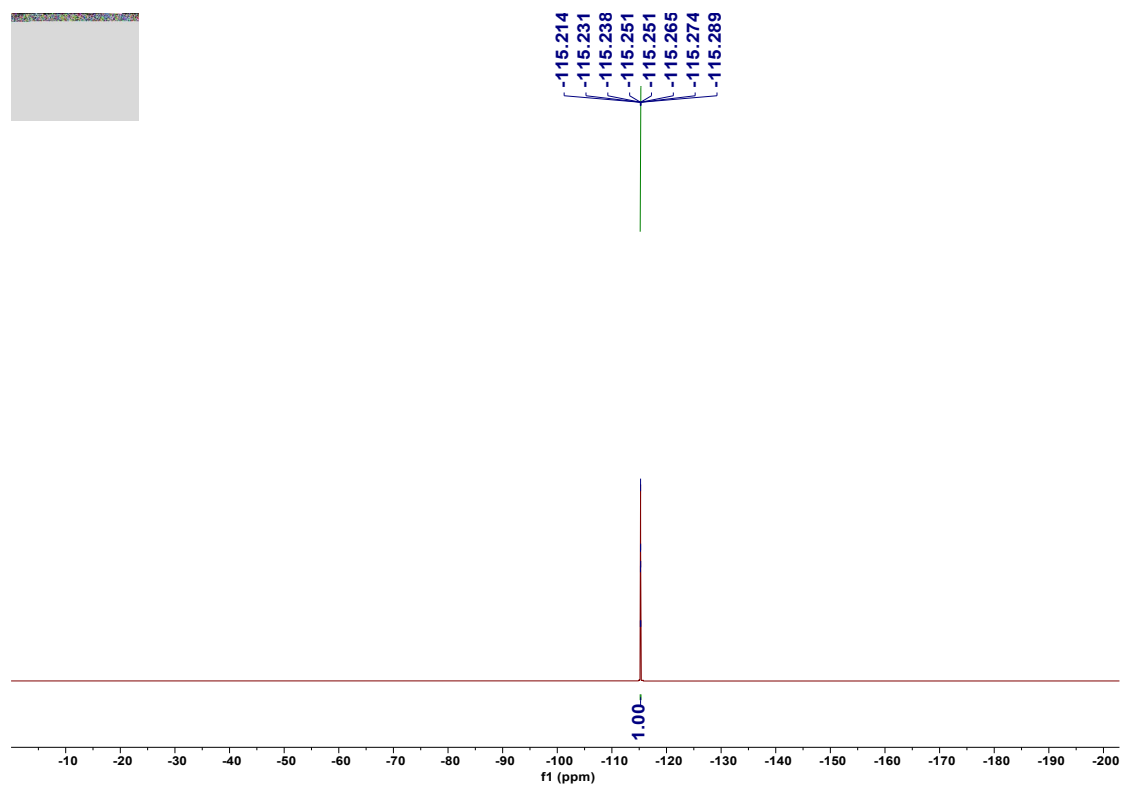


Into a 15 mL sealed tube were added  $\text{Ph}_3\text{P}$  (7.2 mmol, 1.889g, 1.2 equiv.) and **1-1** (6 mmol, 1.0 equiv.), and MeCN (60 mL) under air atmosphere, followed by addition of  $\text{ICH}_2\text{CH}_2\text{I}$  (1.8 mmol, 507.3 mg, 0.3 equiv.). After complete dissolution of all reagents, the reaction tube was sealed and the mixture was stirred at 50 °C in an oil bath for 30 min. Upon completion of the reaction, the mixture was concentrated under reduced pressure. The resulting crude material was purified by flash column chromatography (eluent: PE) to afford the target product **2-1** as white solid (1.18g, 81%).

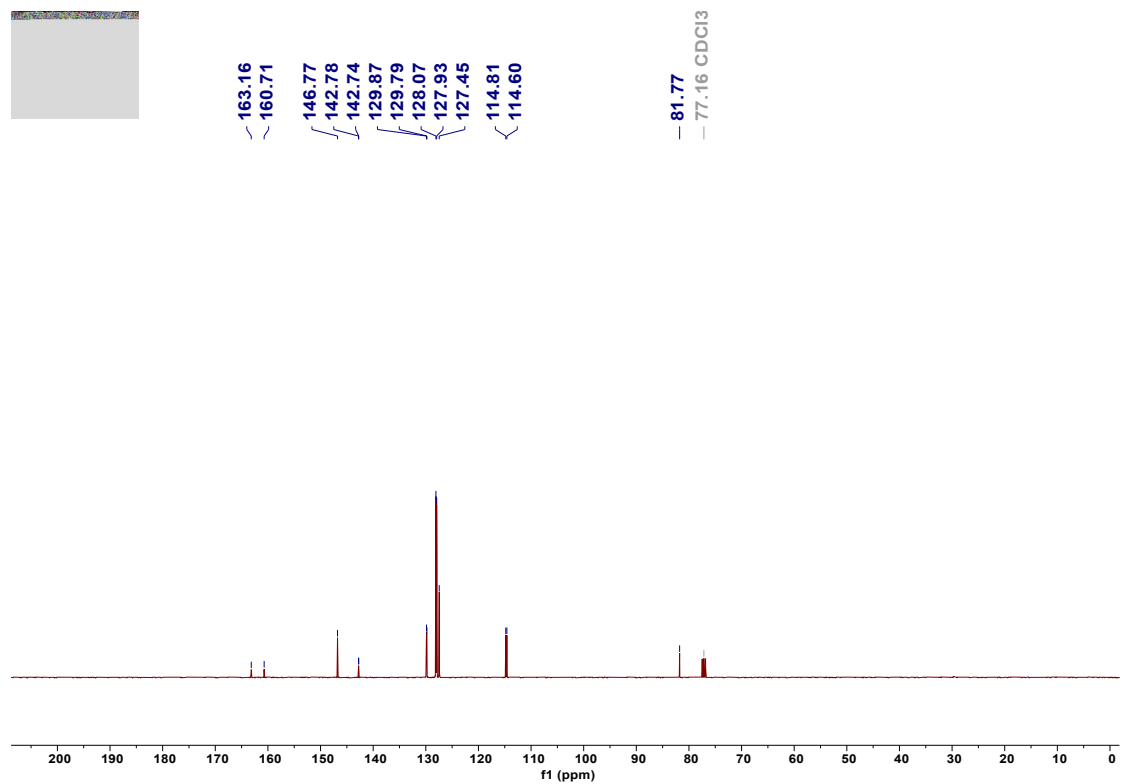
## 7 Copies of $^1\text{H}$ NMR, $^{19}\text{F}$ NMR and $^{13}\text{C}$ NMR Spectra of Substrates and Products



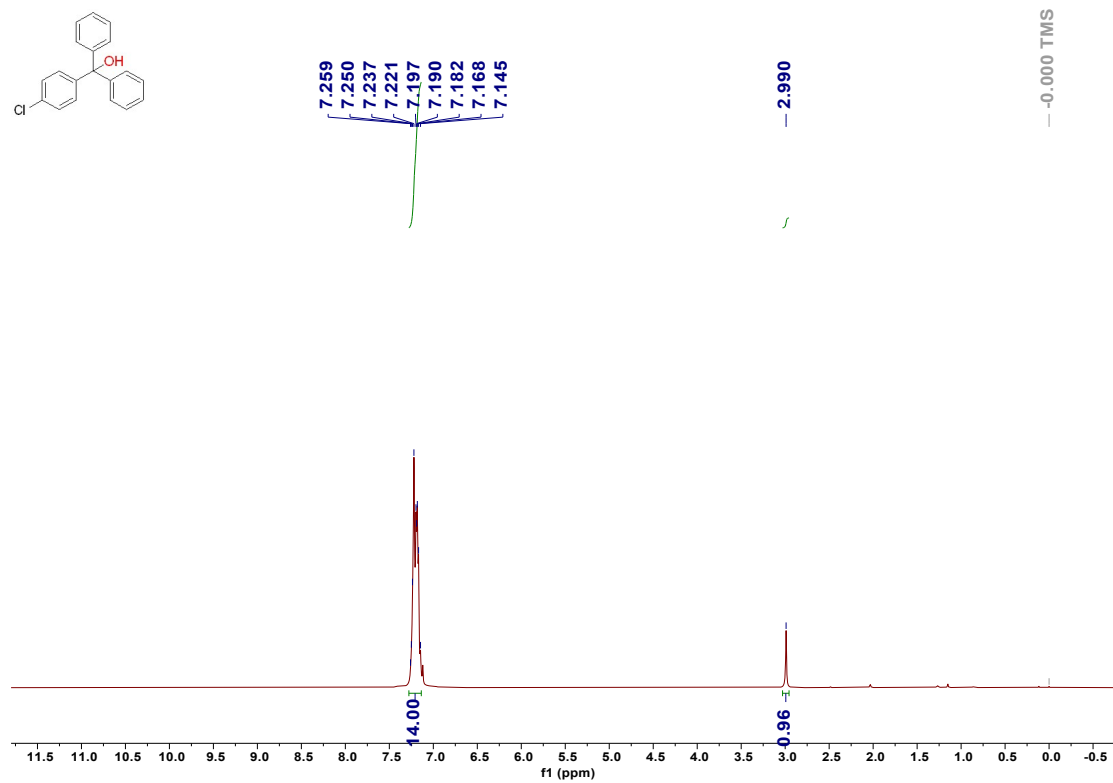
$^1\text{H}$  NMR spectra (400 MHz,  $\text{CDCl}_3$ ) of **1-2**



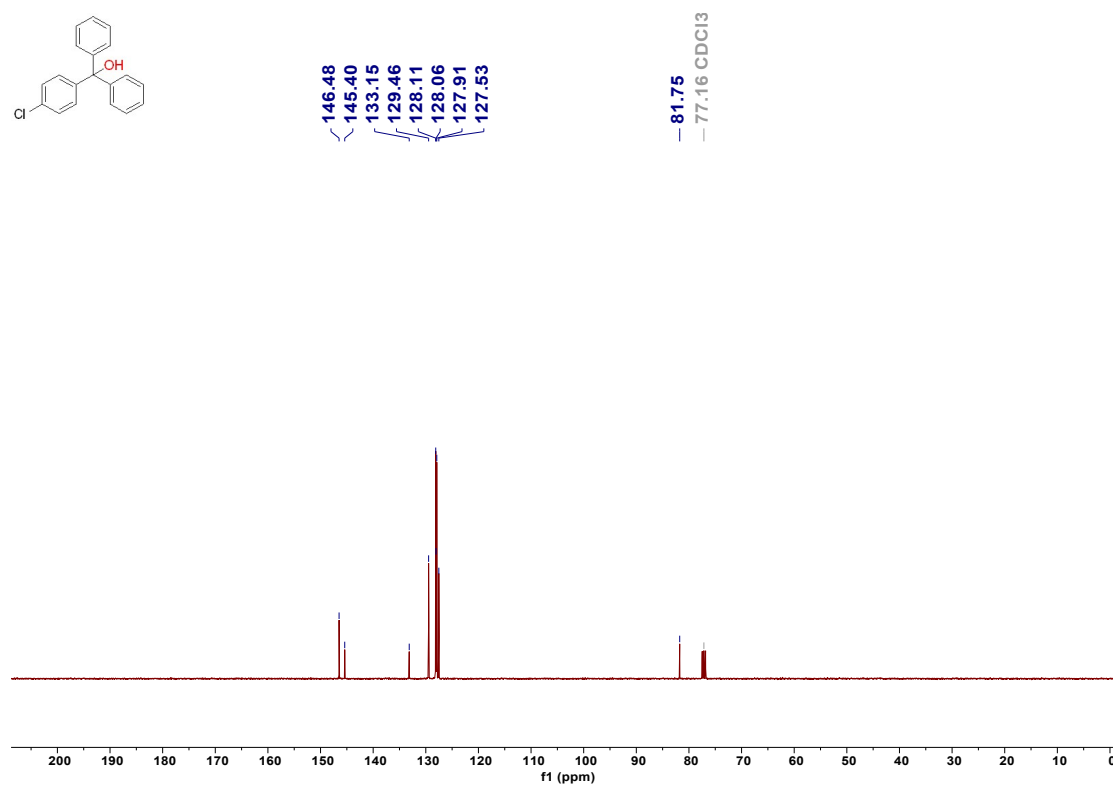
$^{19}\text{F}$  NMR spectra (376 MHz,  $\text{CDCl}_3$ ) of **1-2**



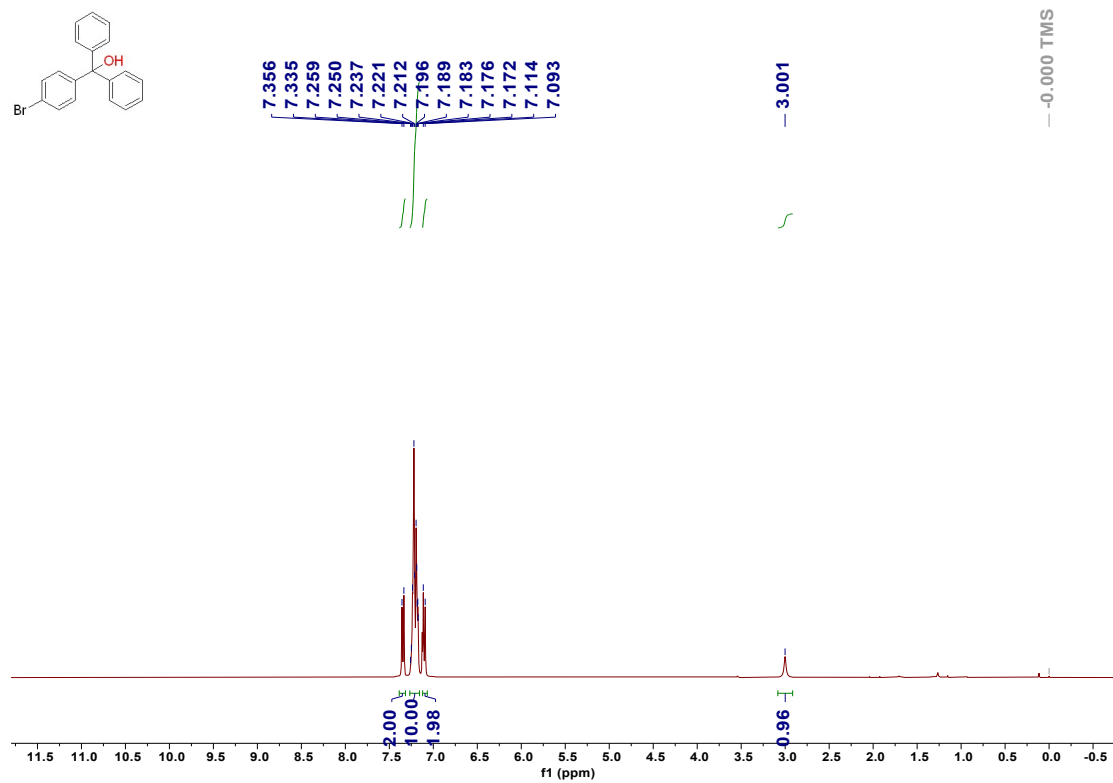
$^{13}\text{C}$  NMR spectra (101 MHz,  $\text{CDCl}_3$ ) of **1-2**



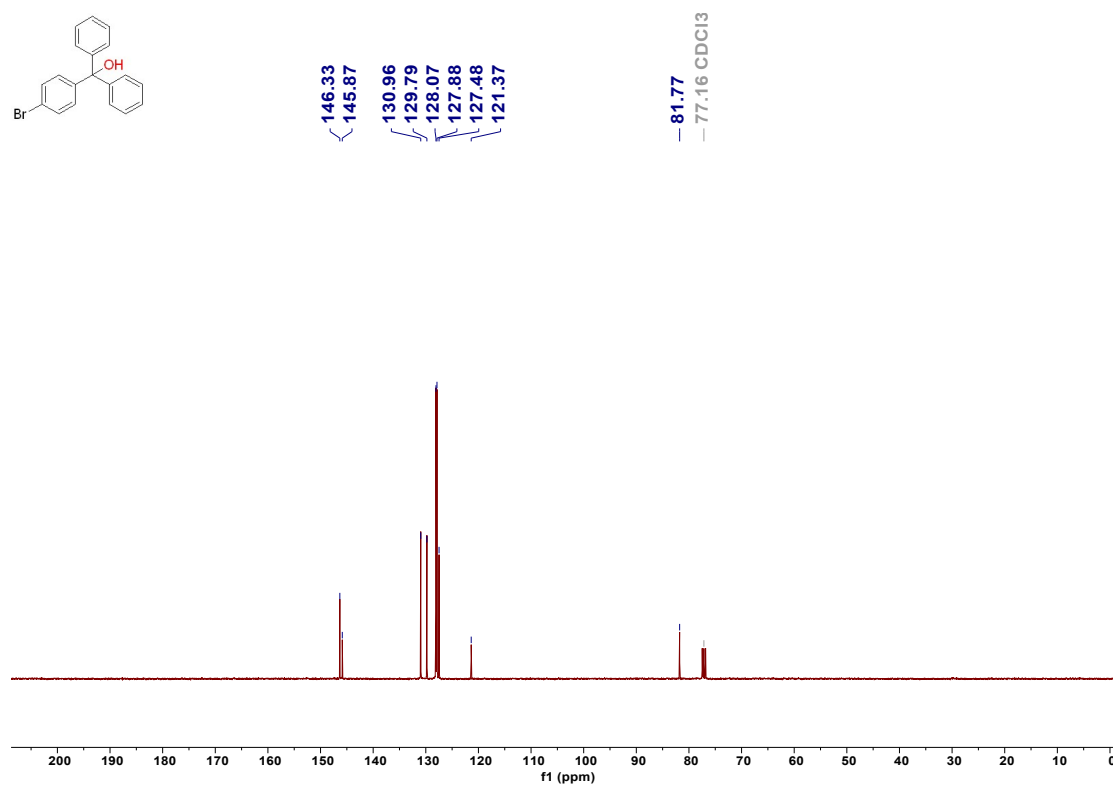
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-3**



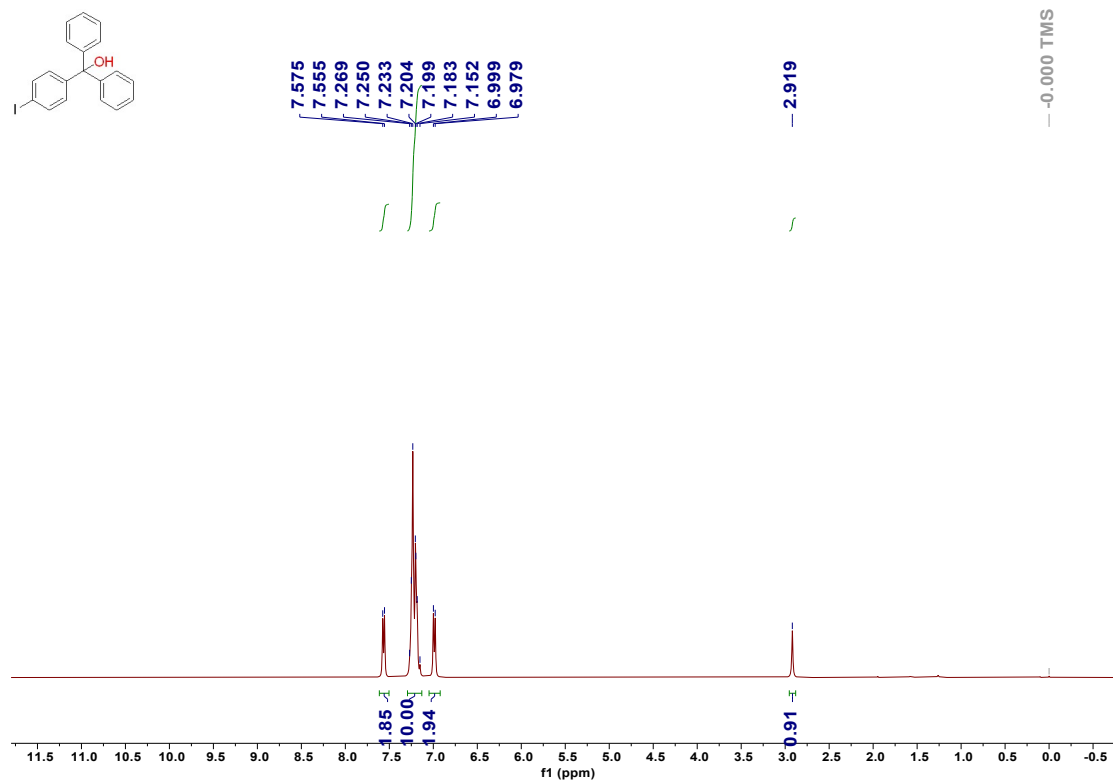
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-3**



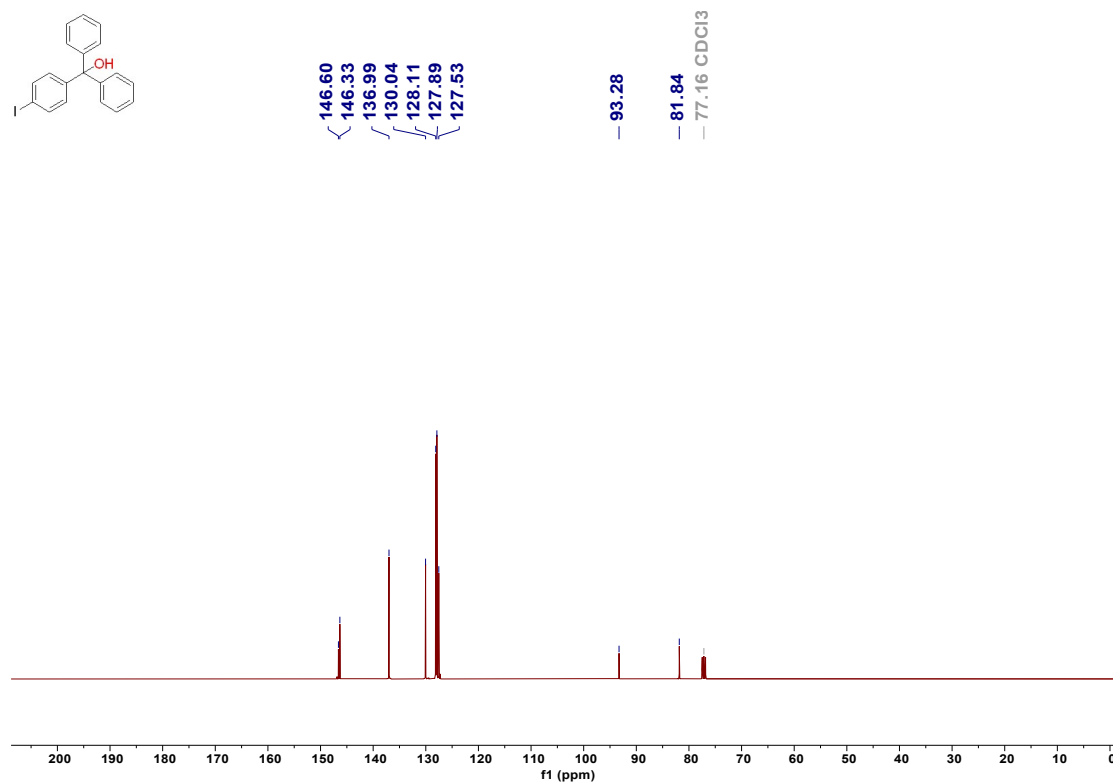
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of 1-4



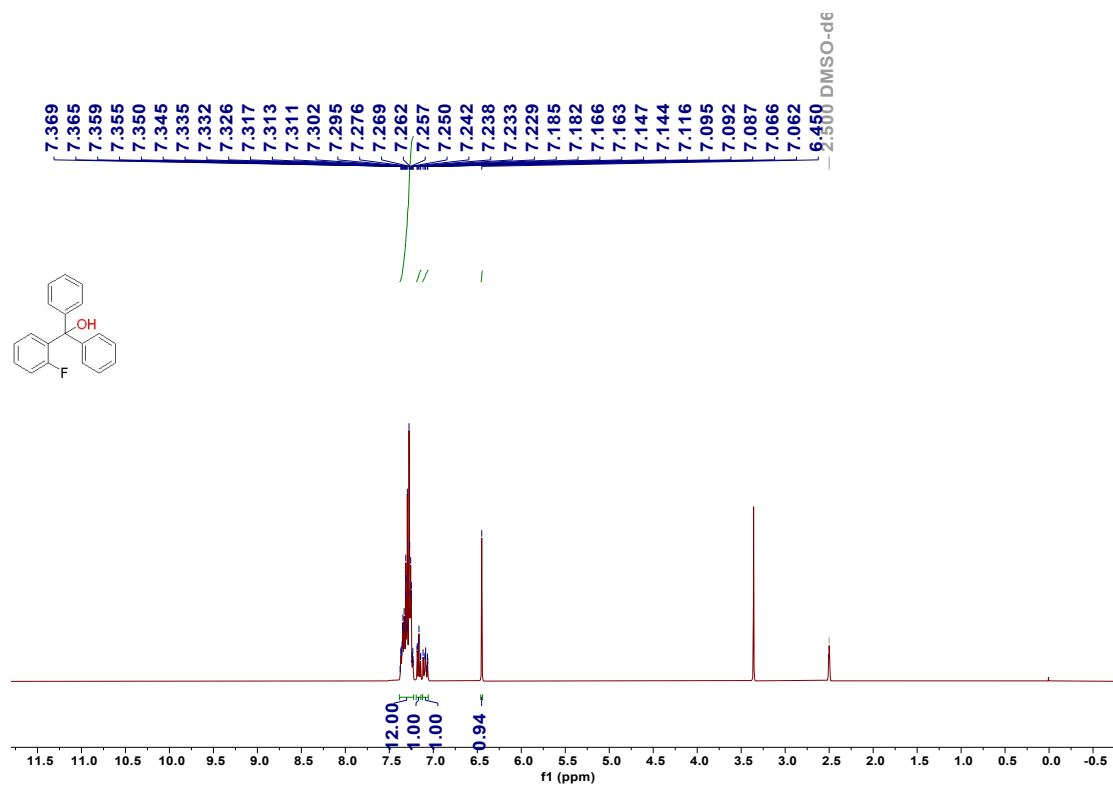
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of 1-4



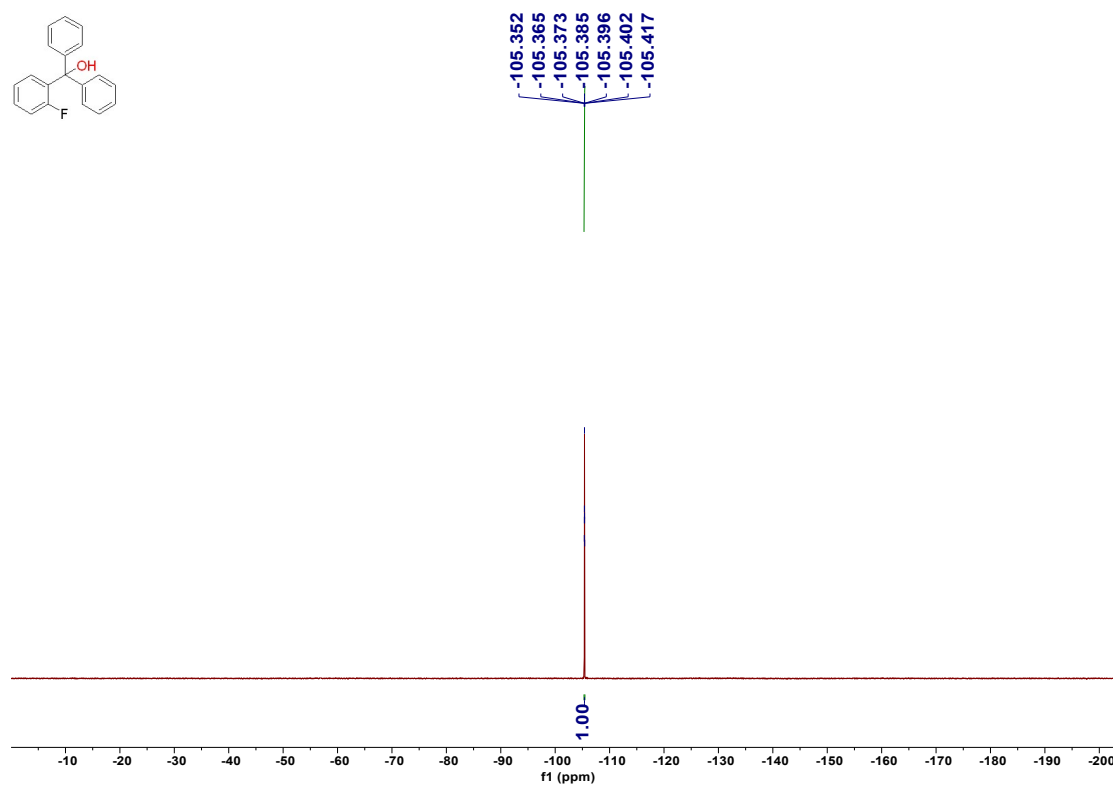
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-5**



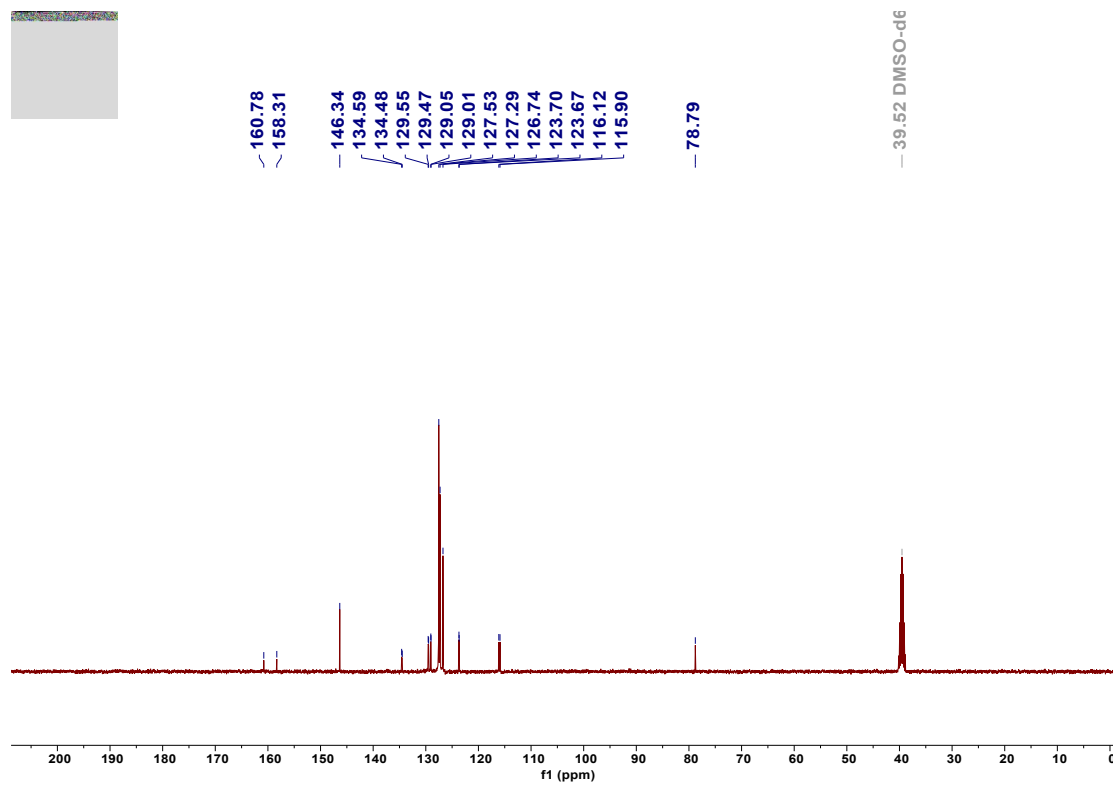
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-5**



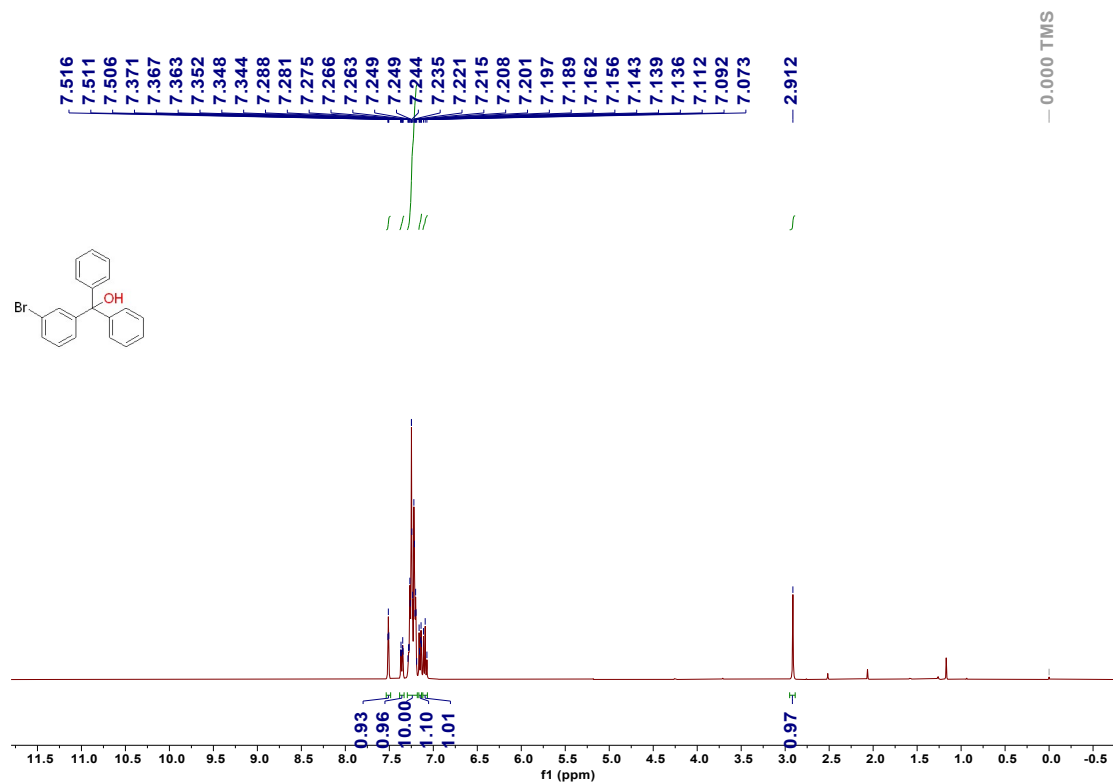
<sup>1</sup>H NMR spectra (400 MHz, DMSO-*d*<sub>6</sub>) of 1-6



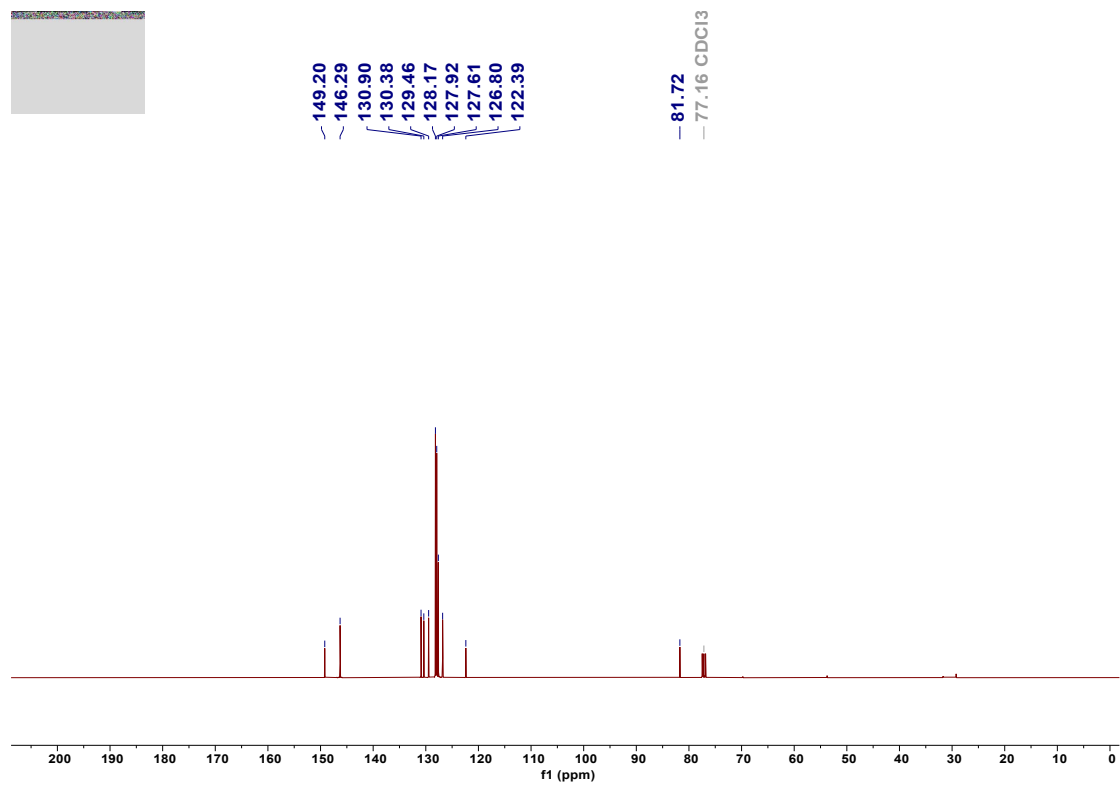
<sup>19</sup>F NMR spectra (376 MHz, DMSO-*d*<sub>6</sub>) of 1-6



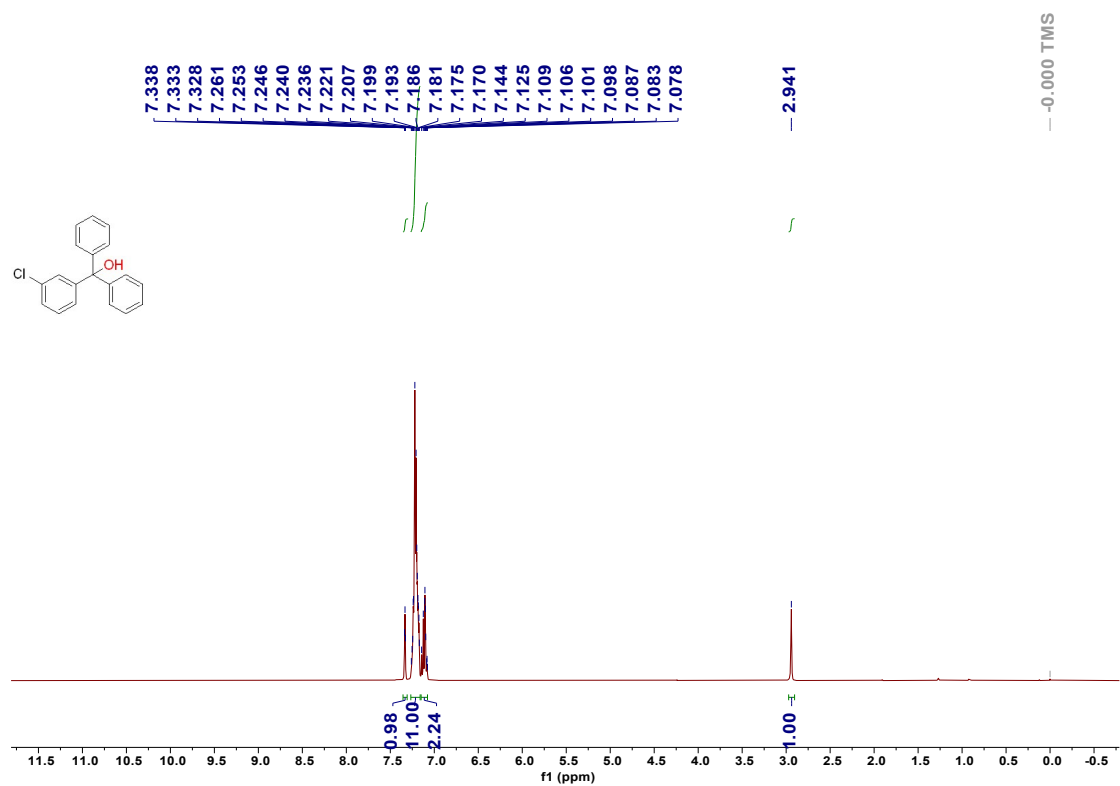
$^{13}\text{C}$  NMR spectra (101 MHz,  $\text{DMSO-}d_6$ ) of **1-6**



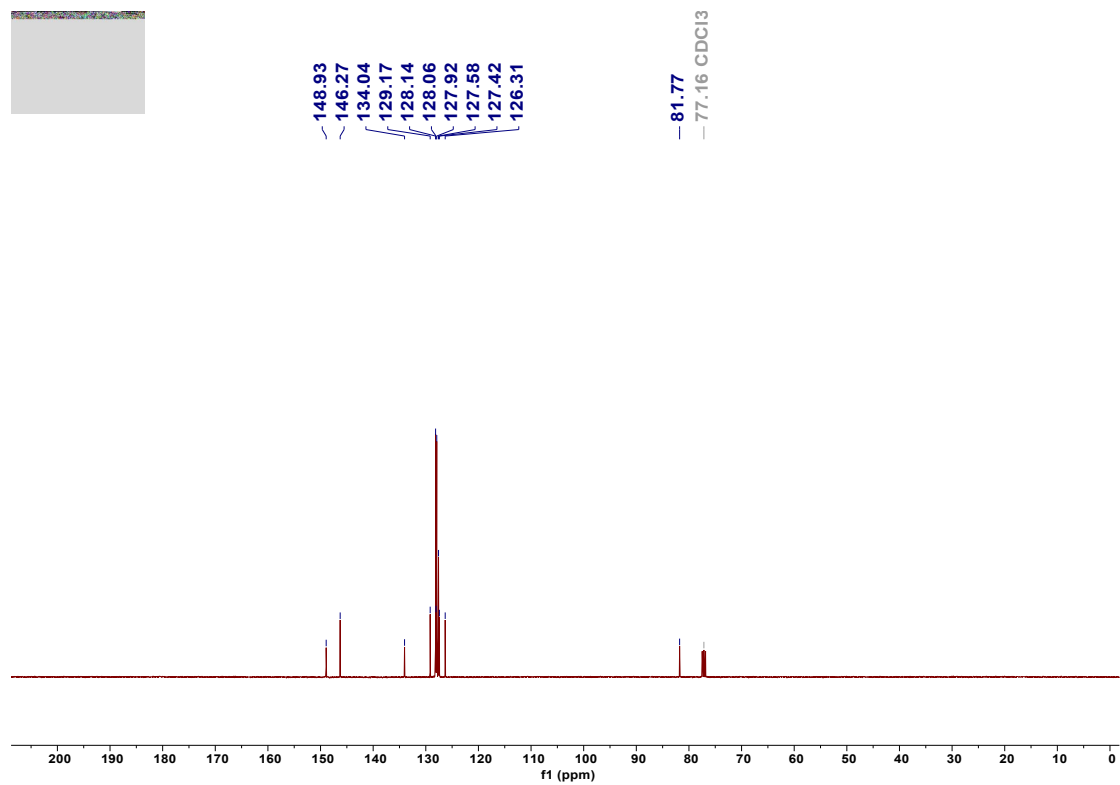
$^1\text{H}$  NMR spectra (400 MHz,  $\text{CDCl}_3$ ) of **1-8**



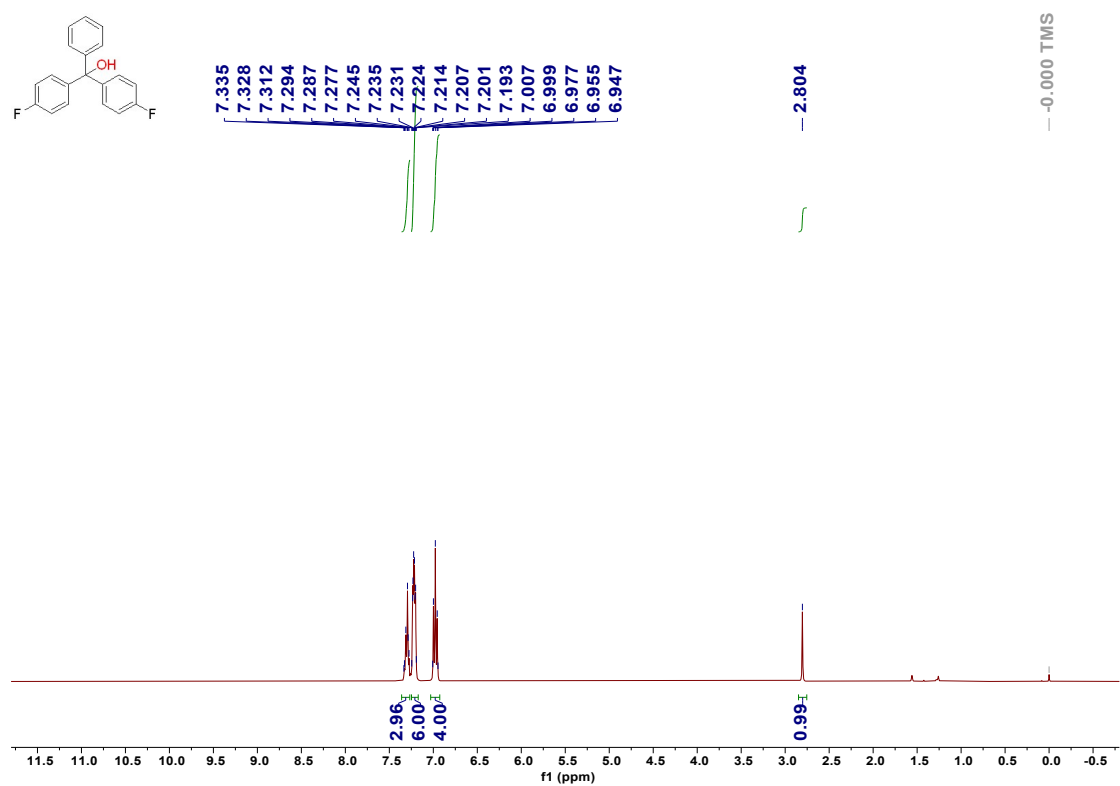
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-8**



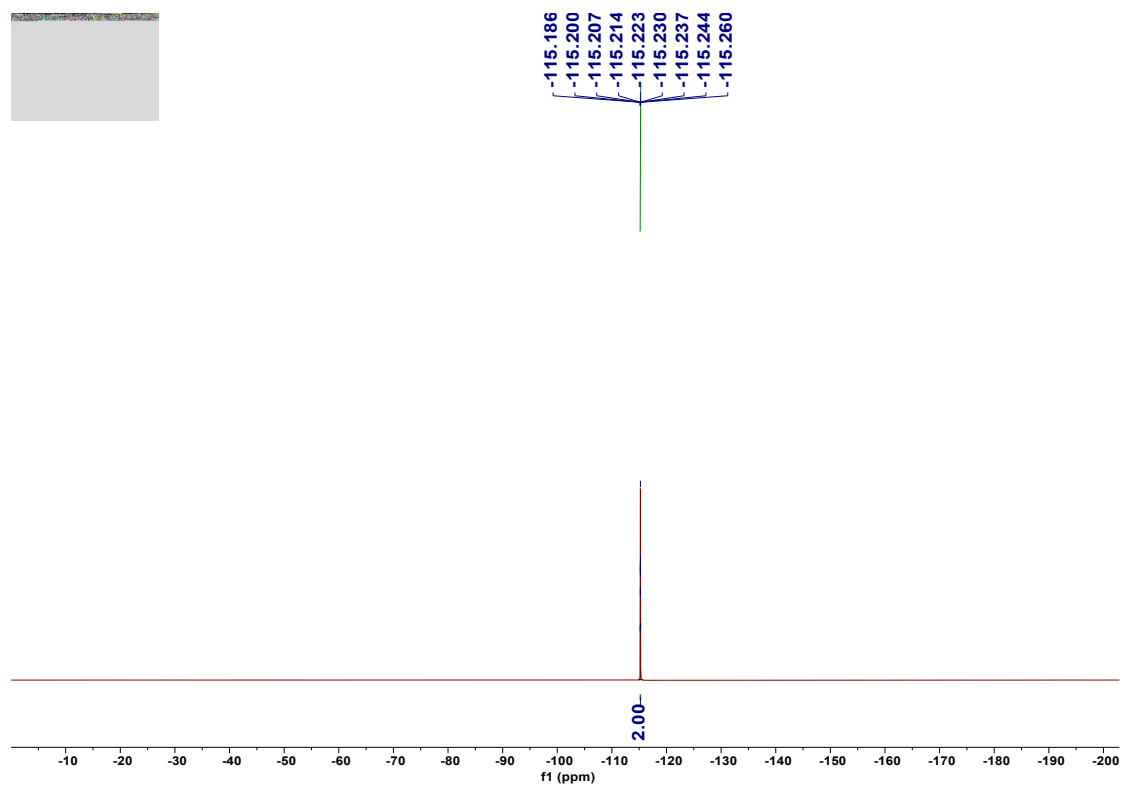
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-9**



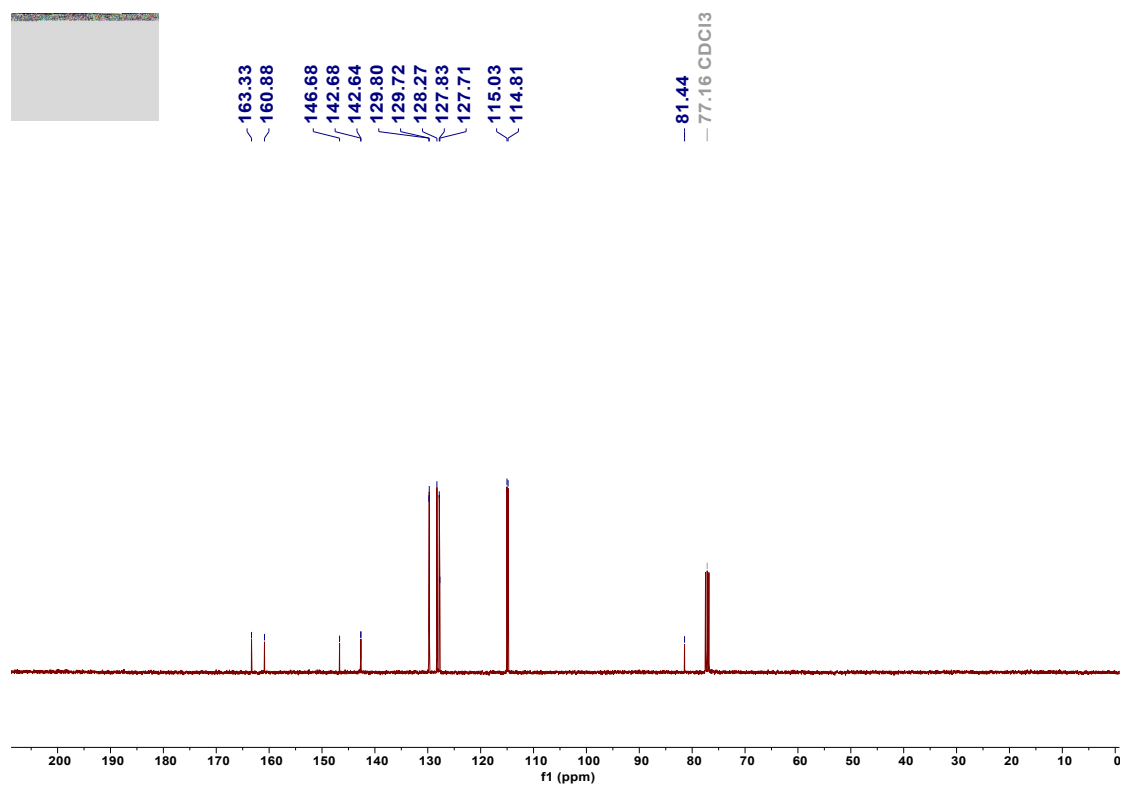
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-9**



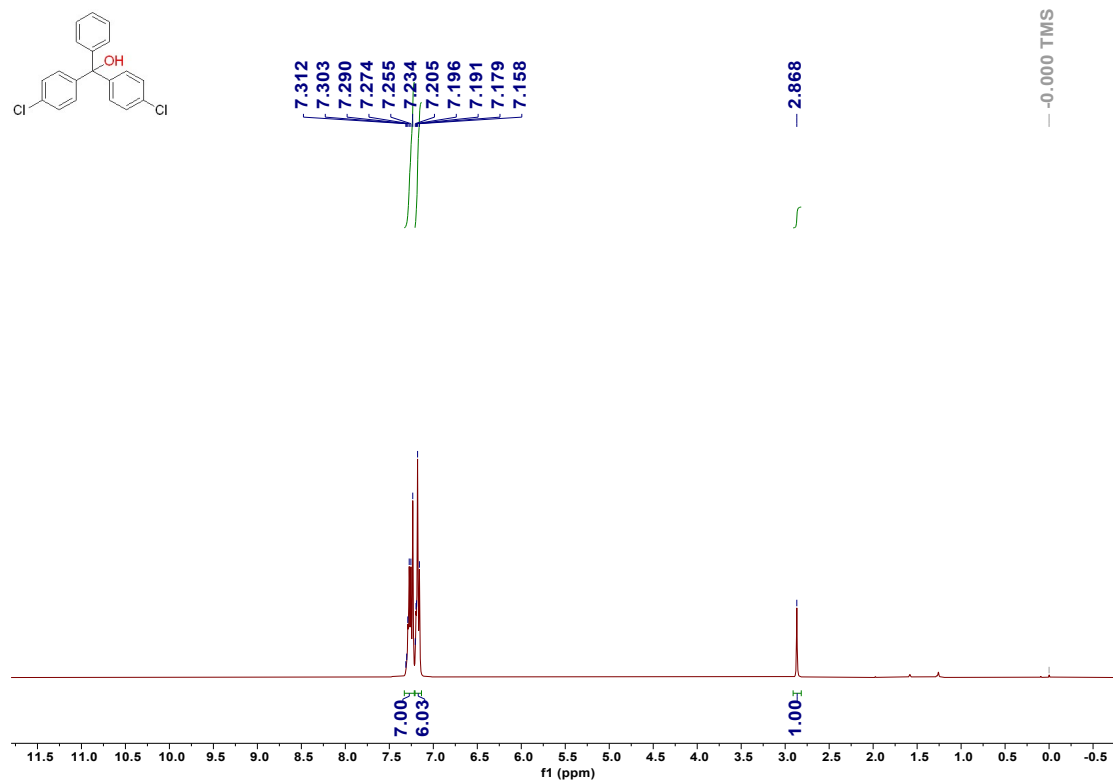
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-10**



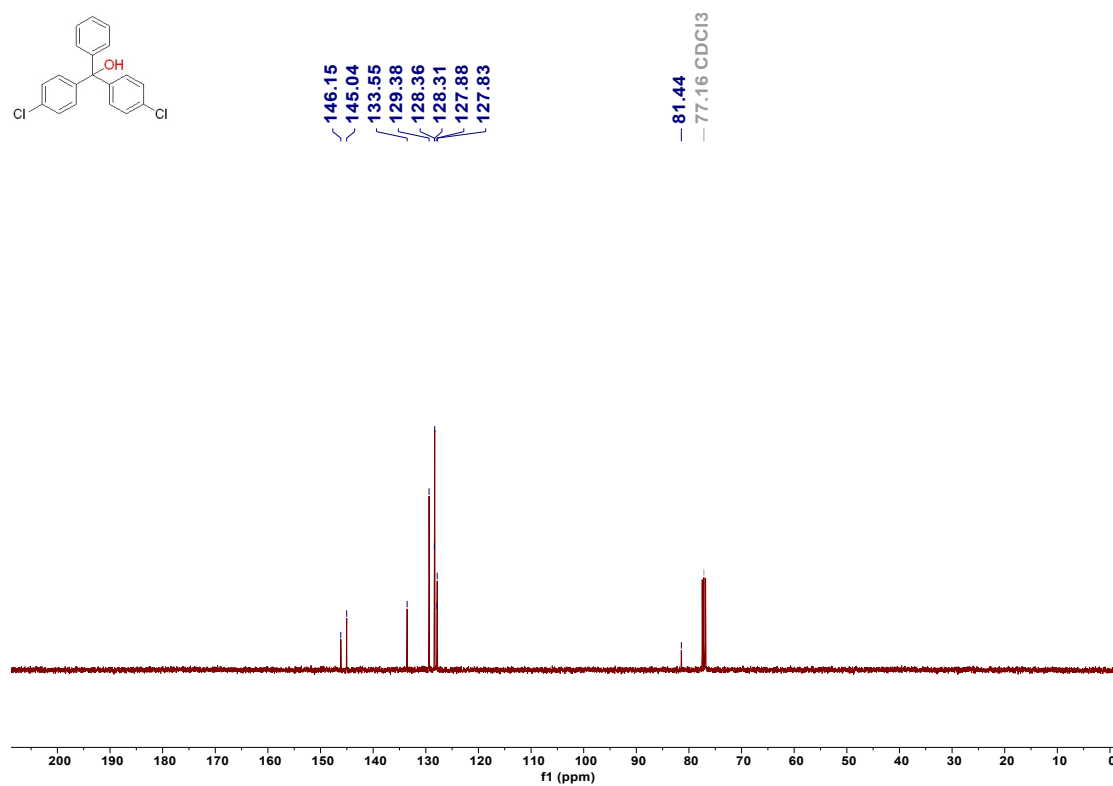
$^{19}\text{F}$  NMR spectra (376 MHz,  $\text{CDCl}_3$ ) of **1-10**



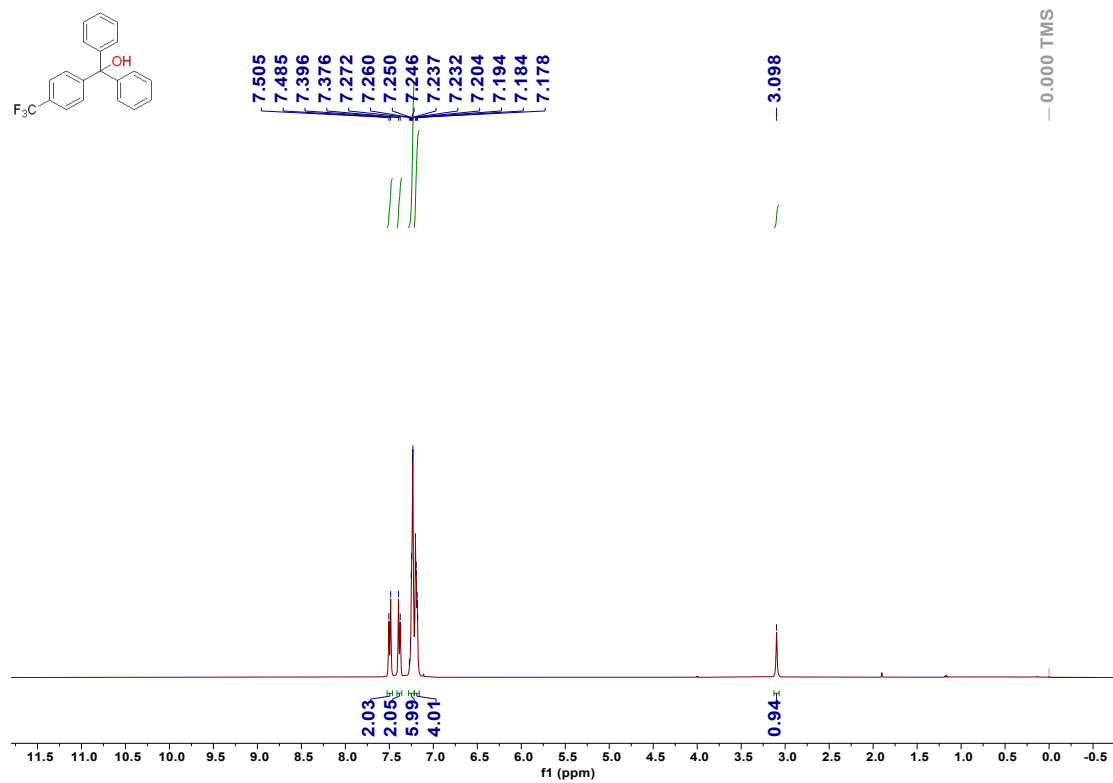
$^{13}\text{C}$  NMR spectra (101 MHz,  $\text{CDCl}_3$ ) of **1-10**



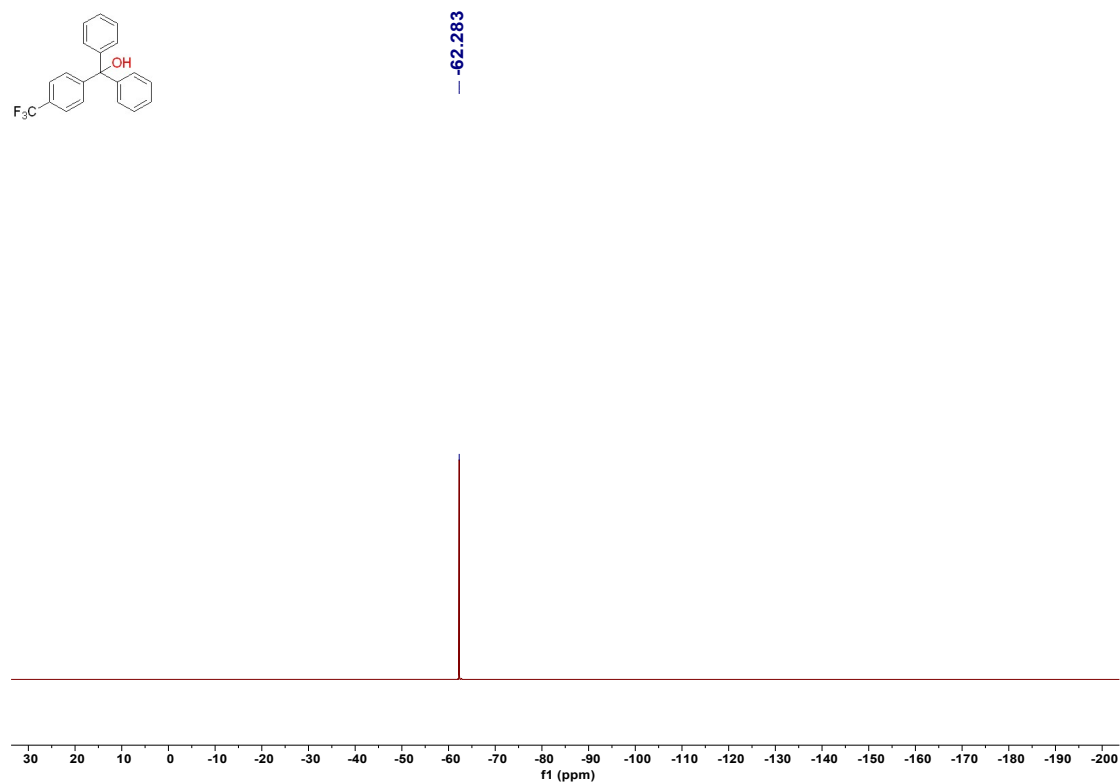
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-11**



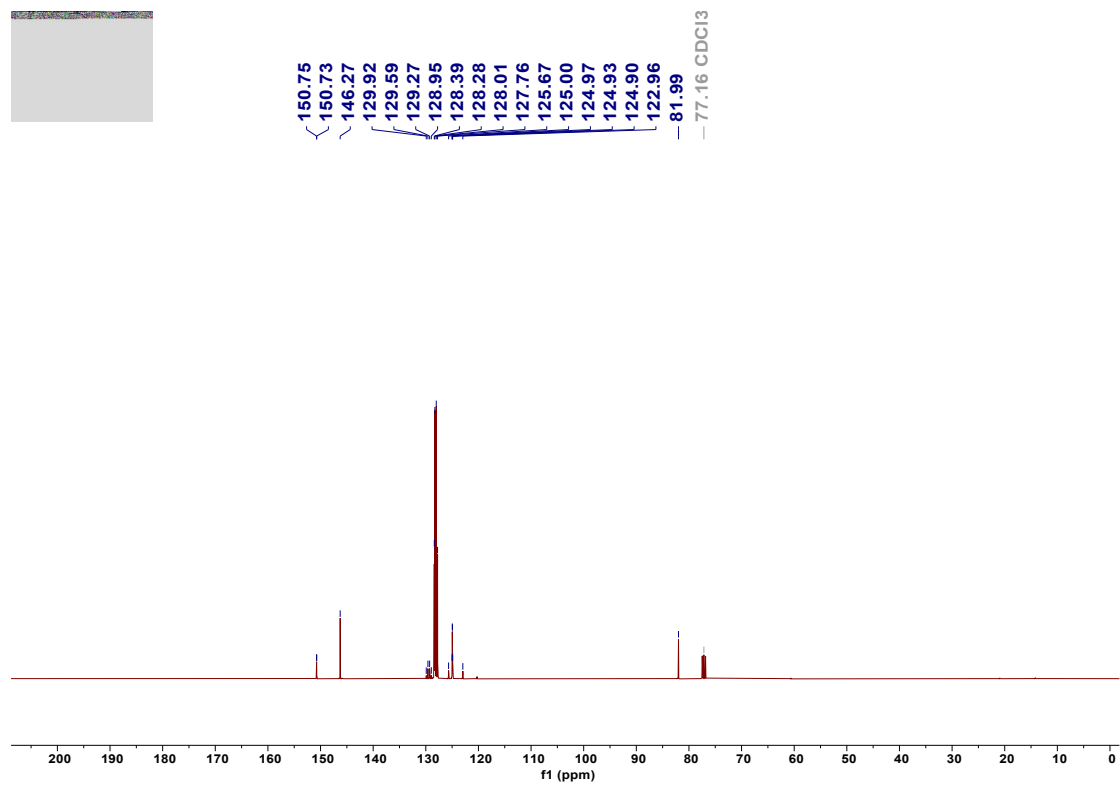
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-11**



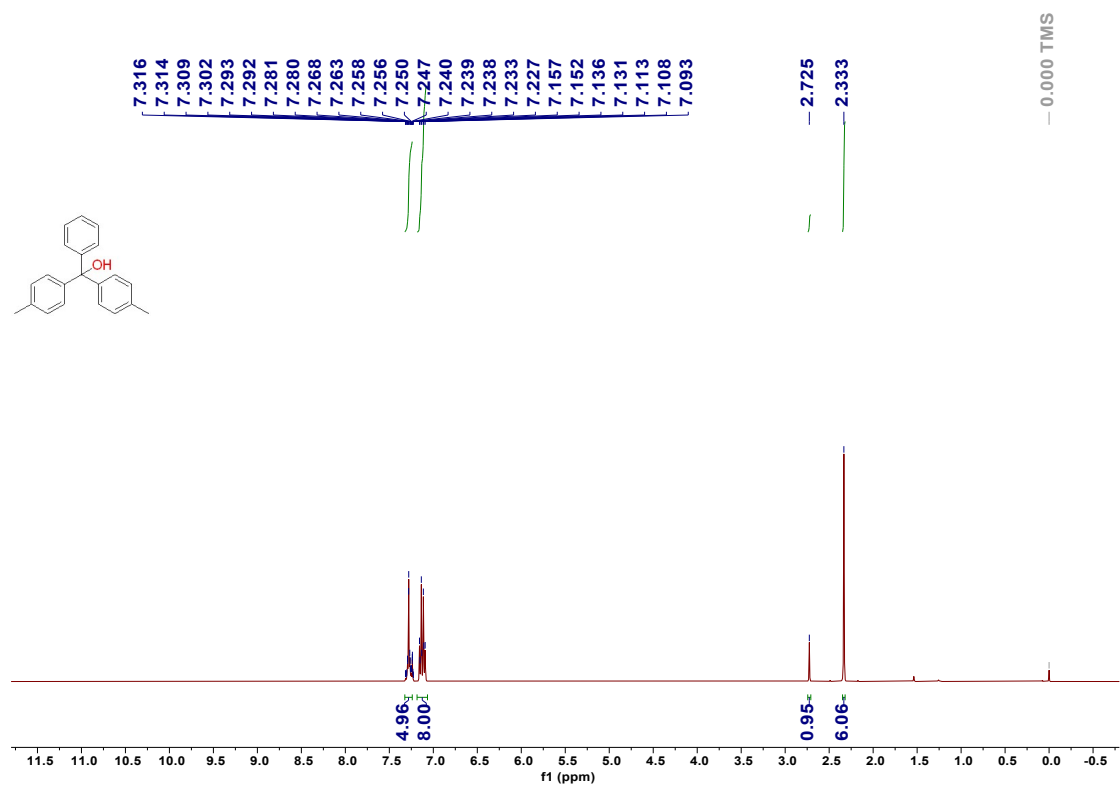
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of 1-14



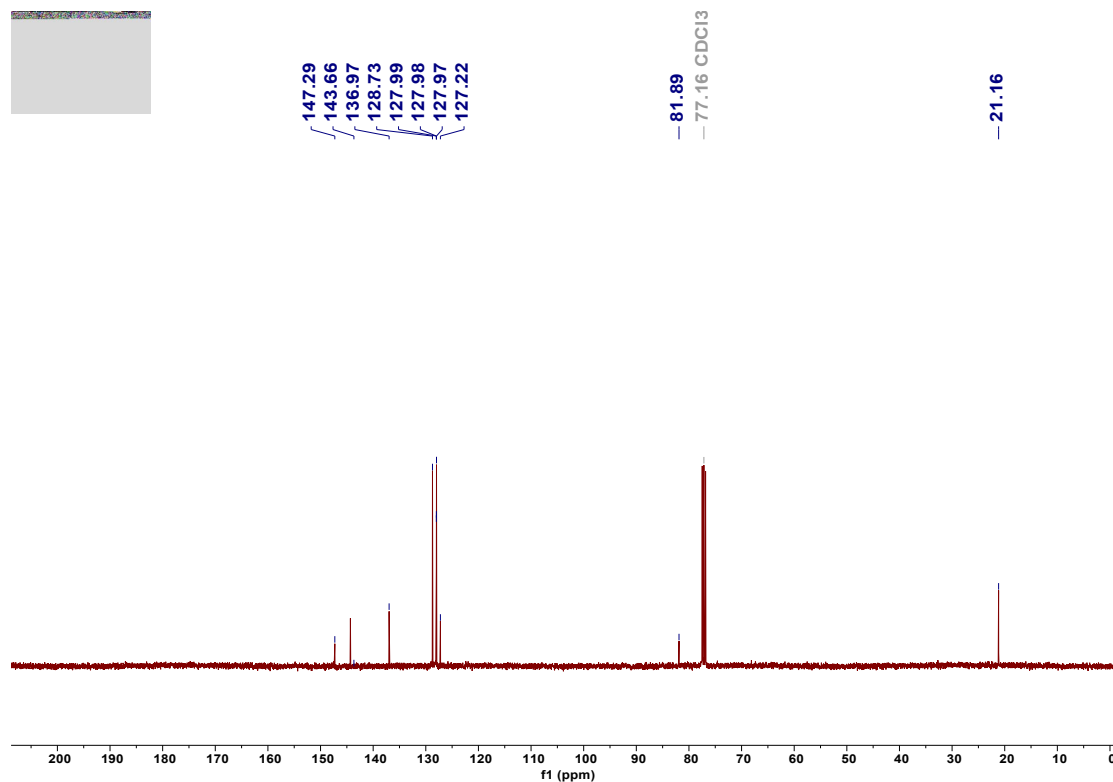
<sup>19</sup>F NMR spectra (376 MHz, CDCl<sub>3</sub>) of 1-14



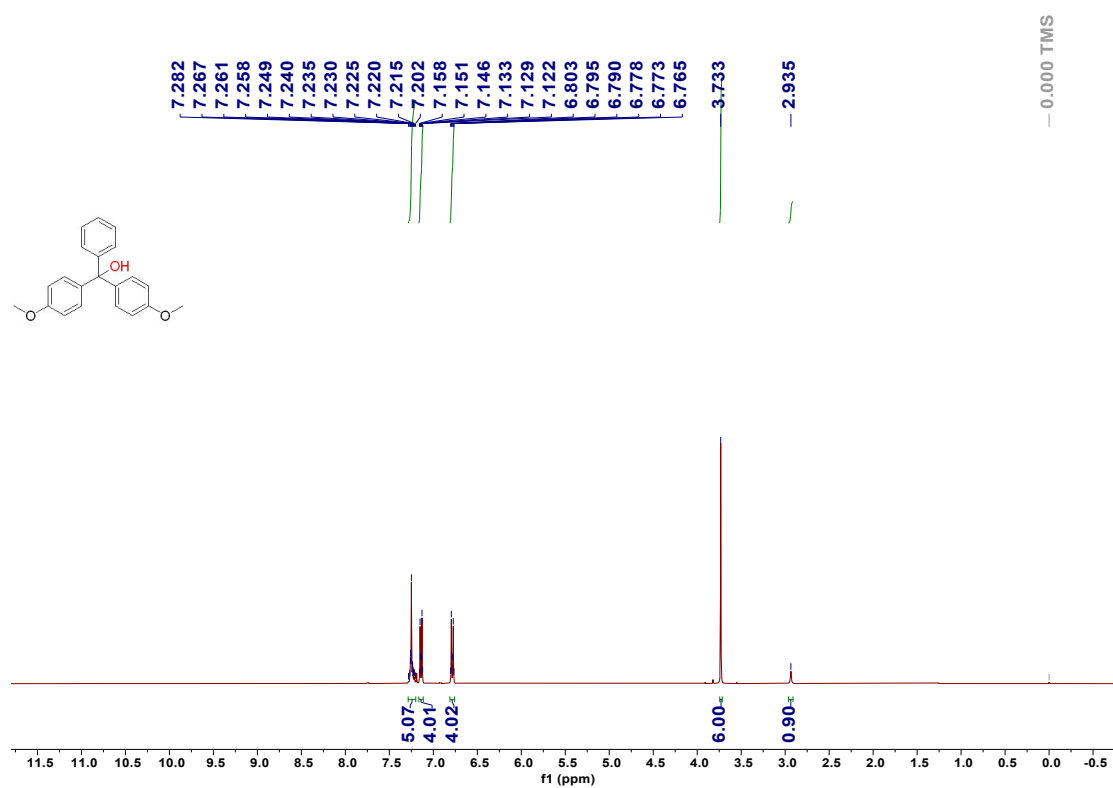
$^{13}\text{C}$  NMR spectra (101 MHz,  $\text{CDCl}_3$ ) of **1-14**



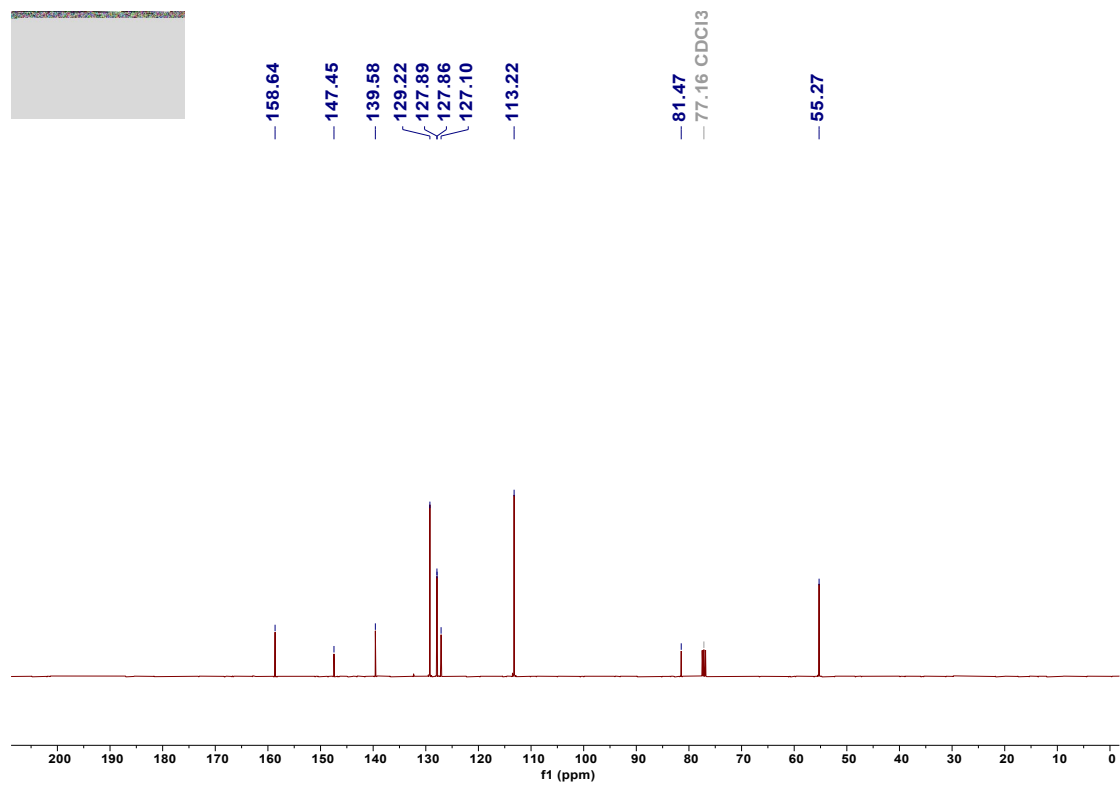
$^1\text{H}$  NMR spectra (400 MHz,  $\text{CDCl}_3$ ) of **1-16**



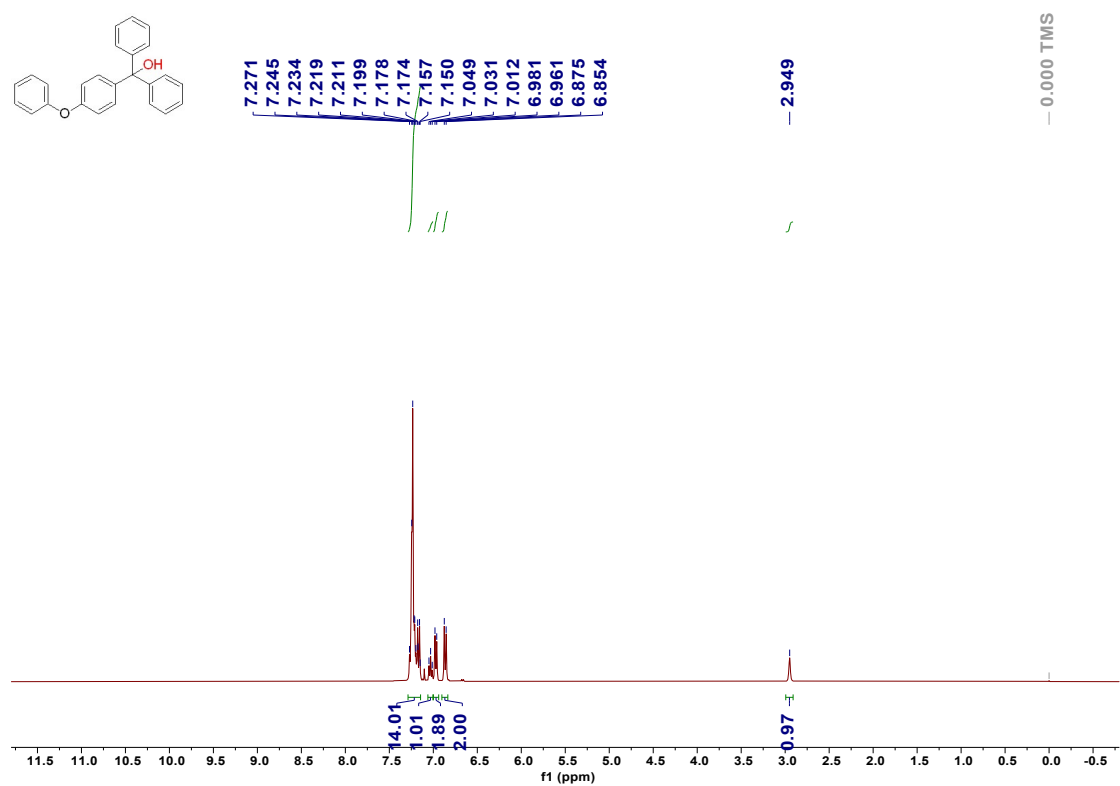
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-16**



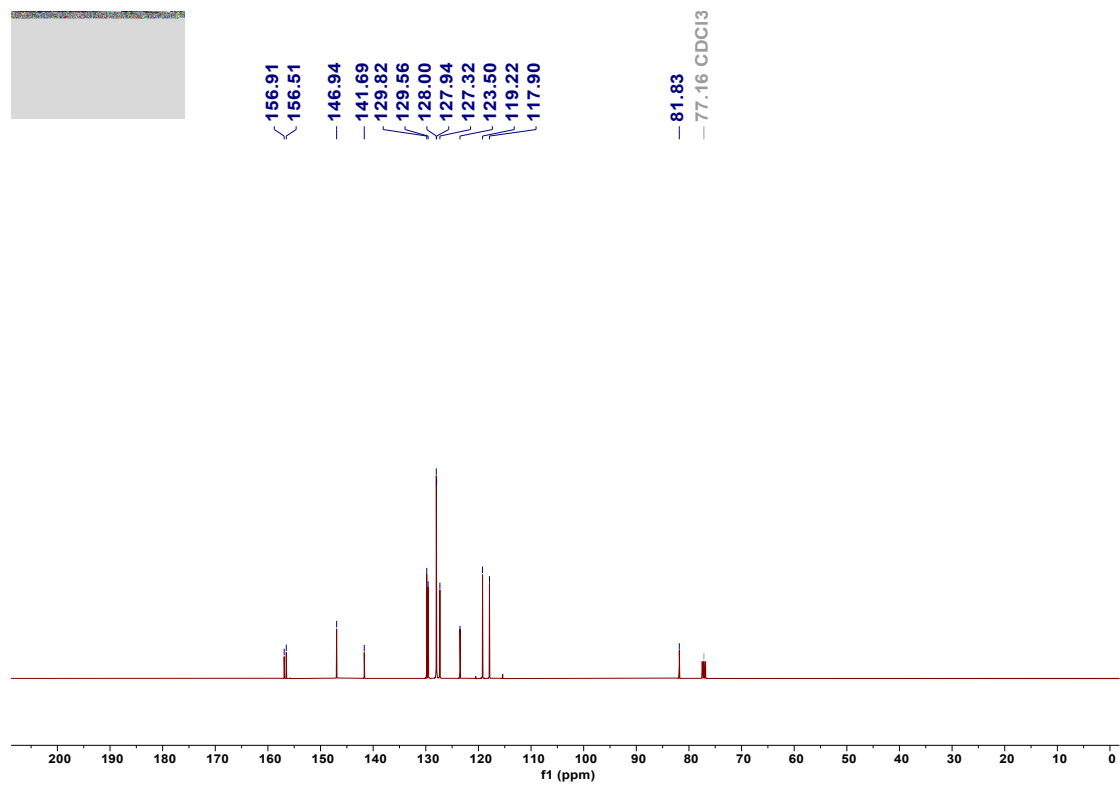
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-19**



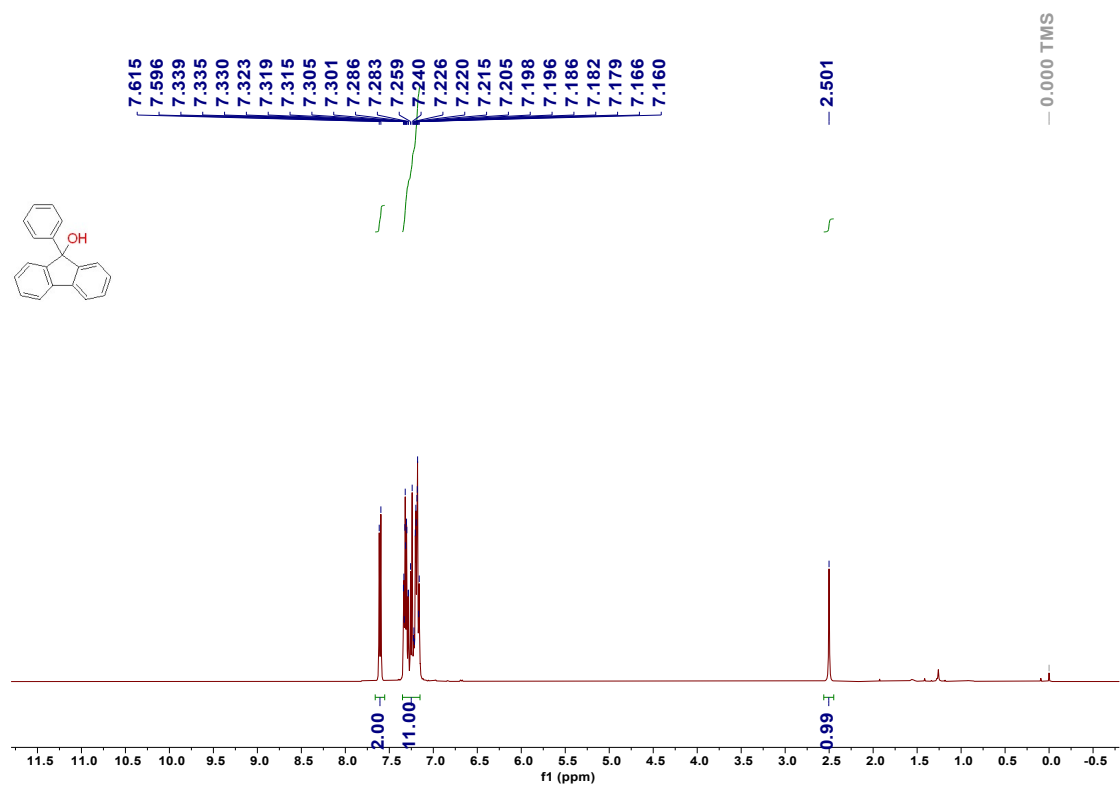
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-19**



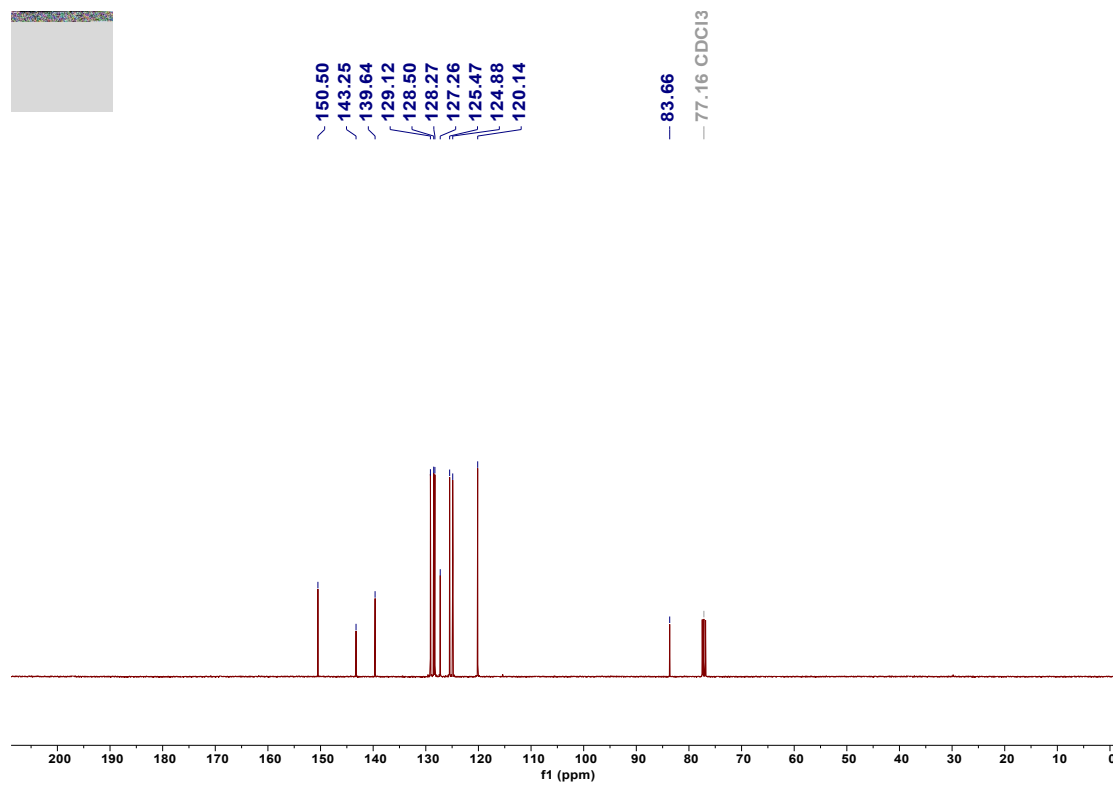
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-21**



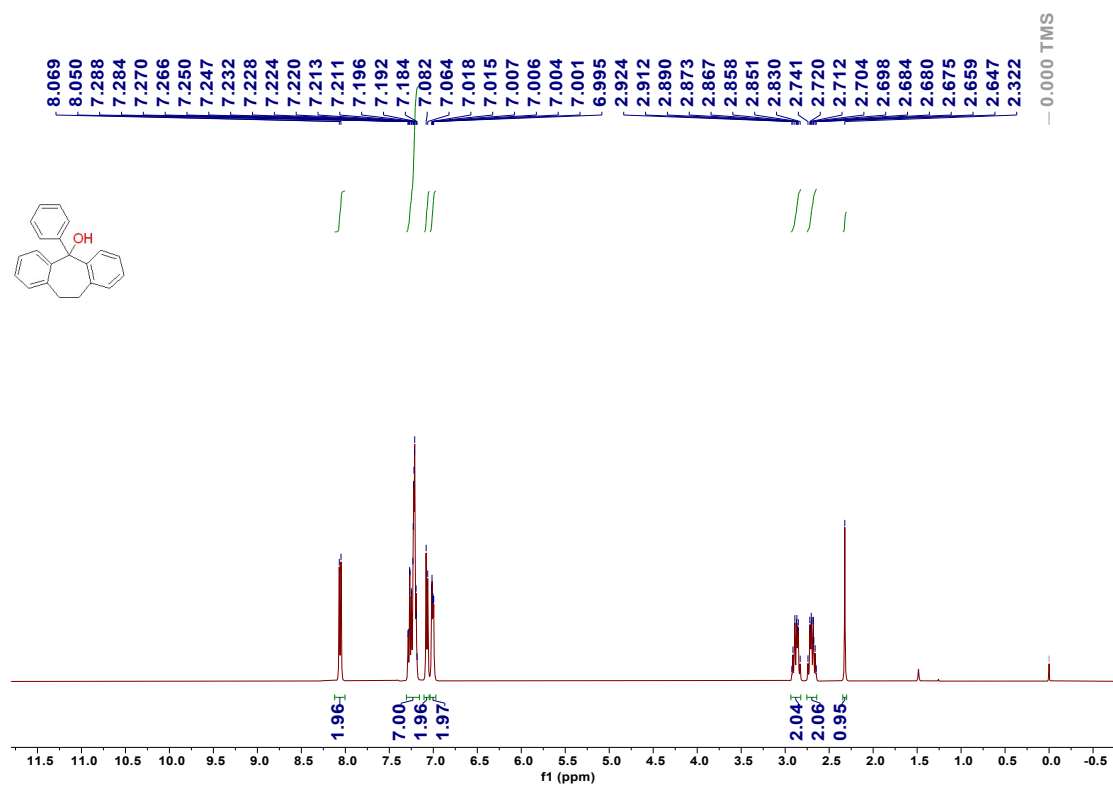
$^{13}\text{C}$  NMR spectra (101 MHz,  $\text{CDCl}_3$ ) of **1-21**



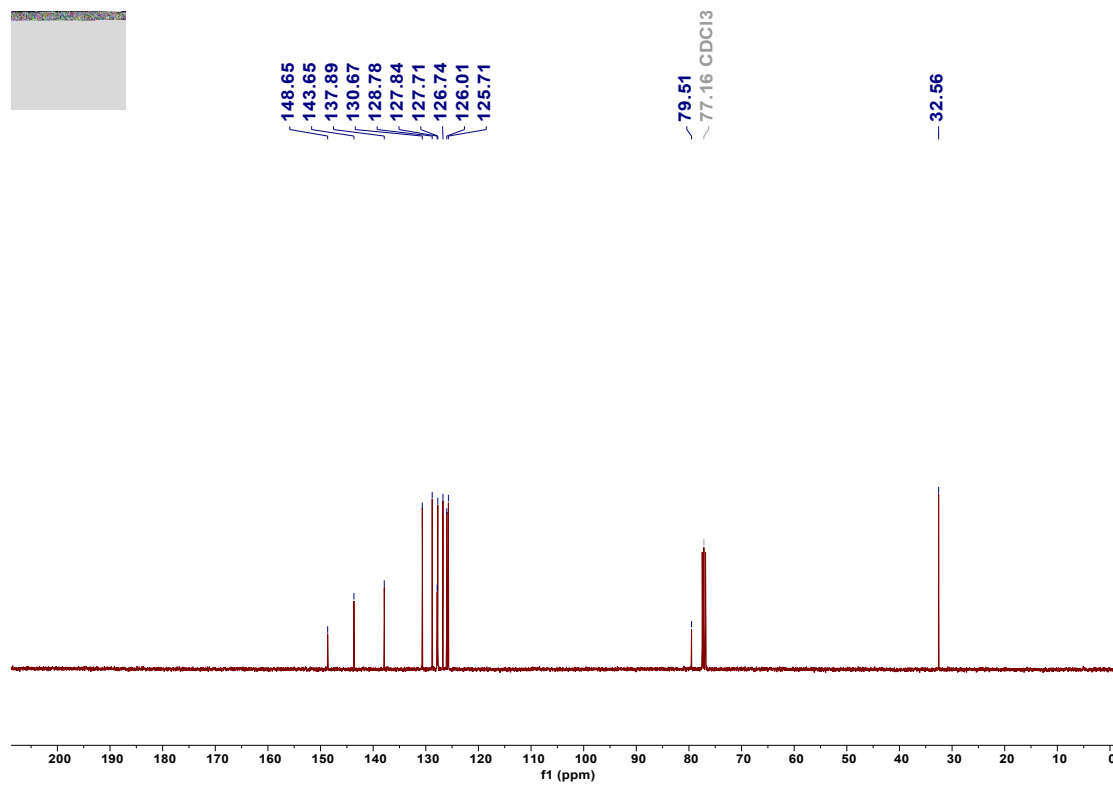
$^1\text{H}$  NMR spectra (400 MHz,  $\text{CDCl}_3$ ) of **1-22**



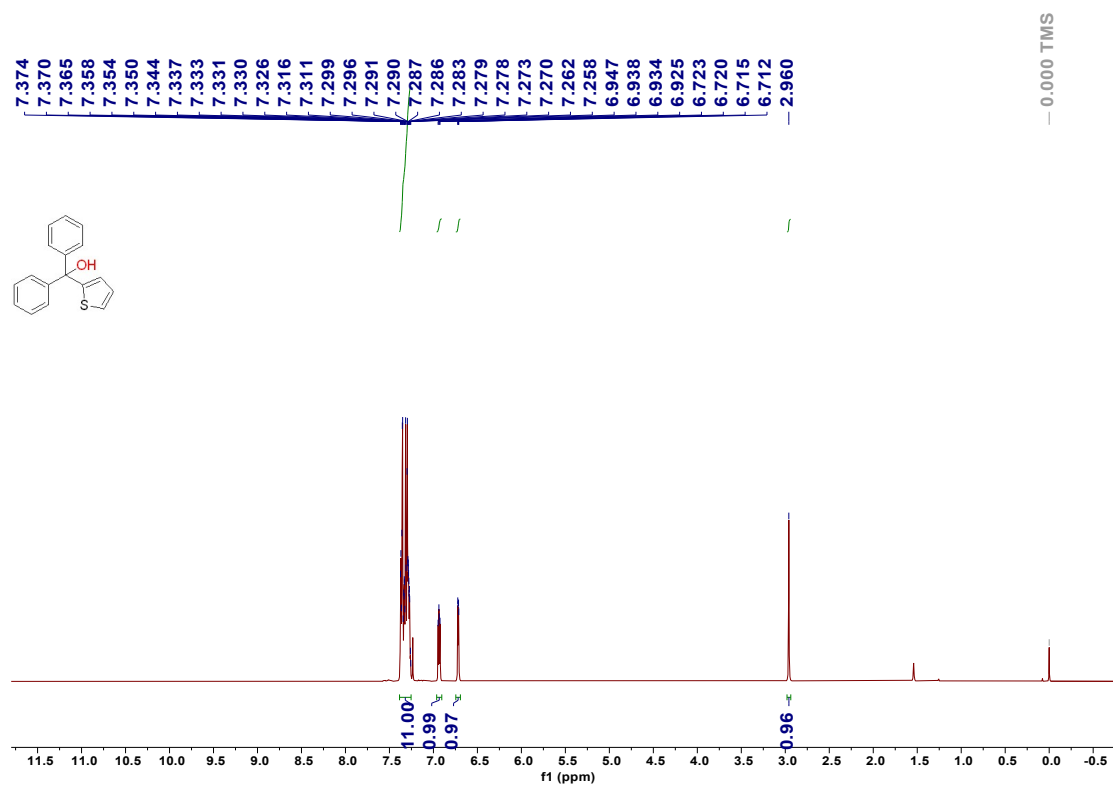
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-22**



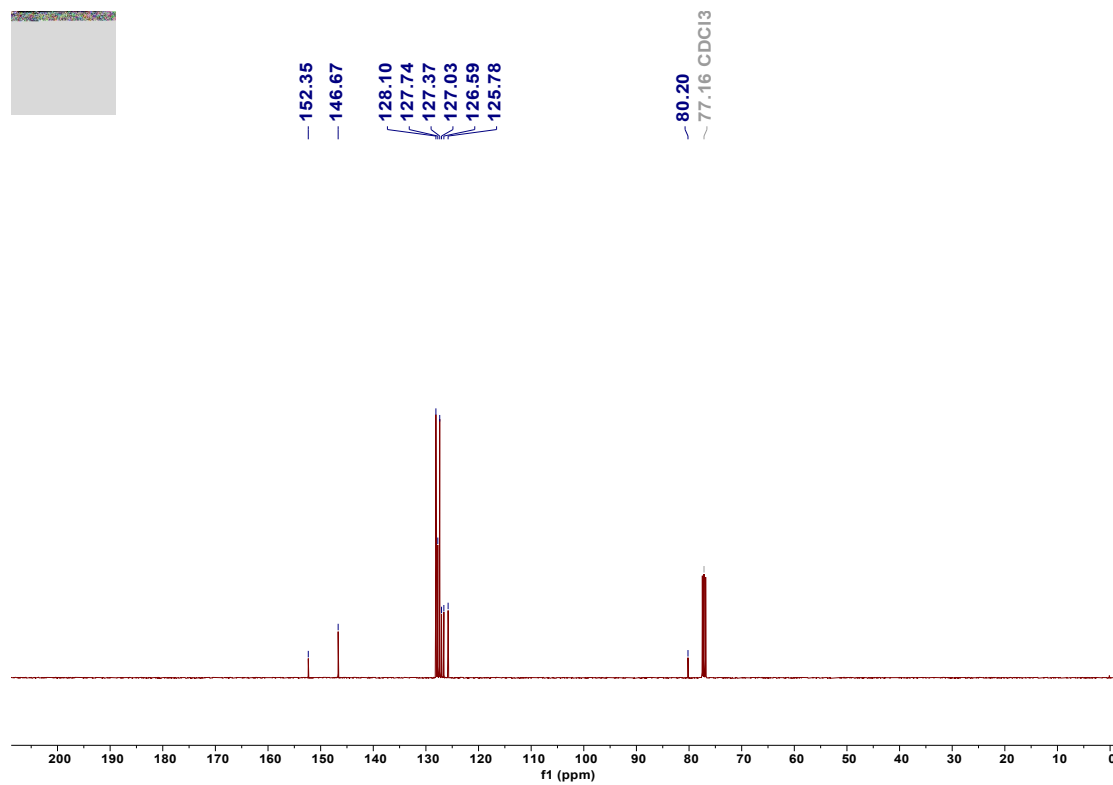
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-23**



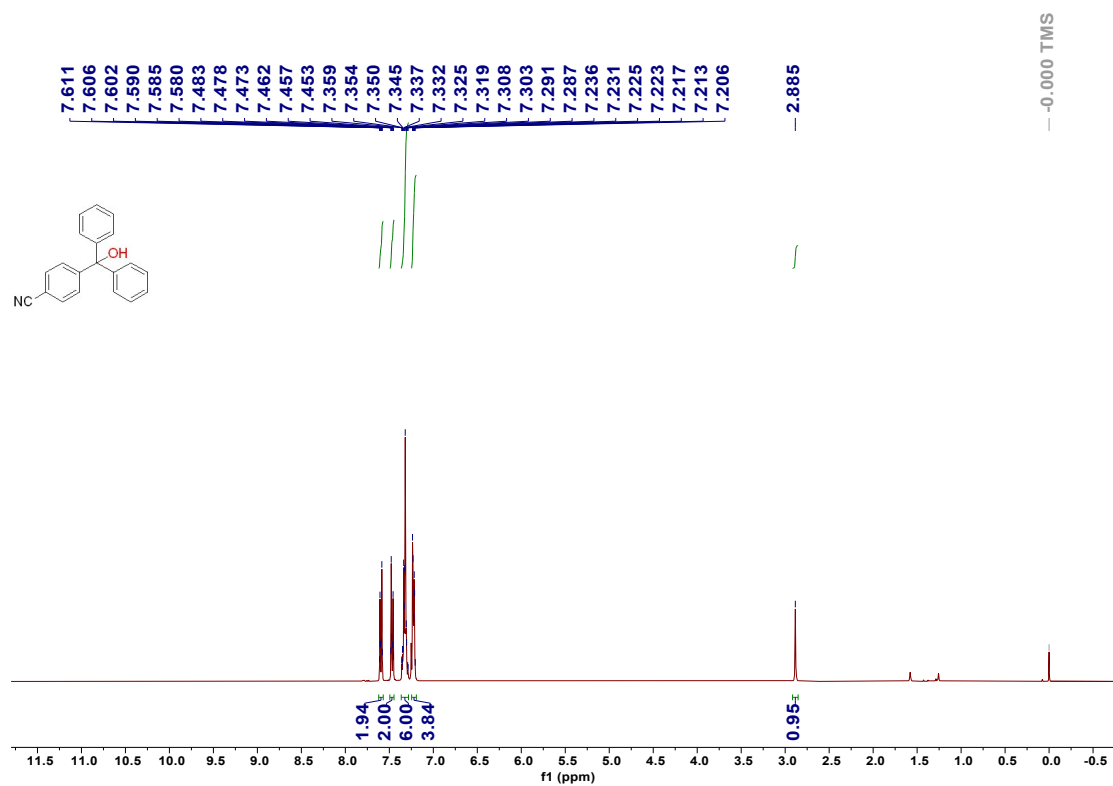
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-23**



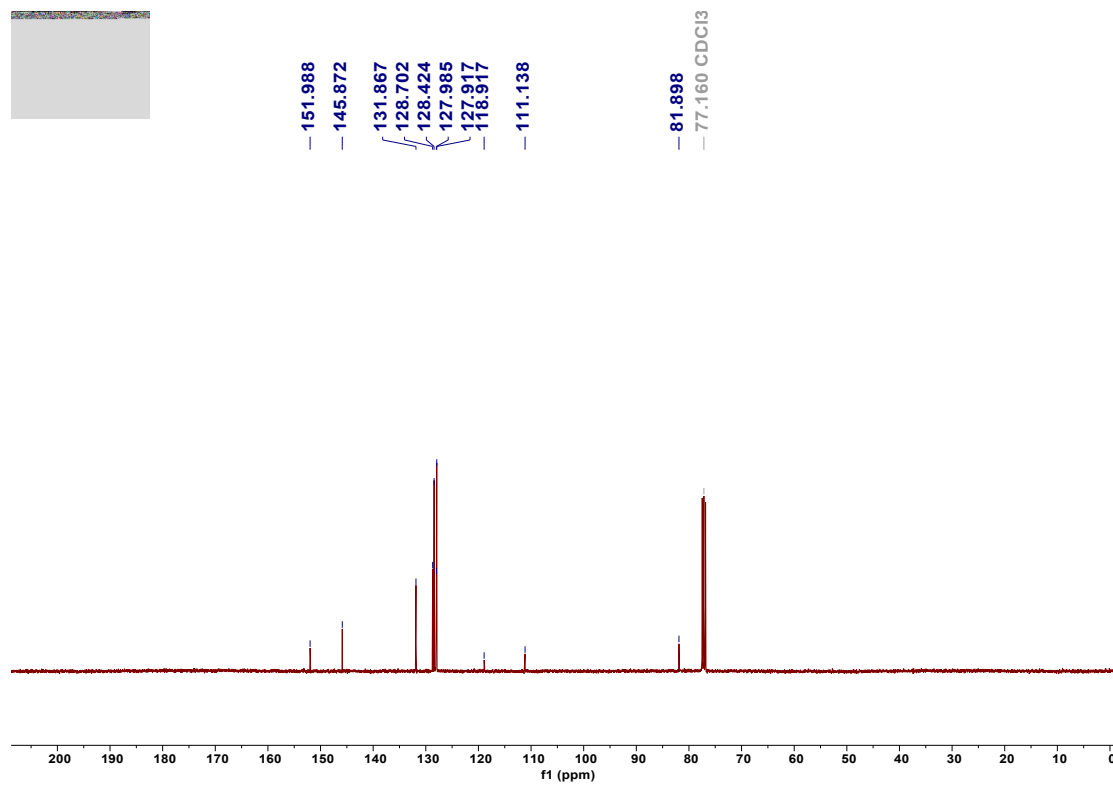
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-27**



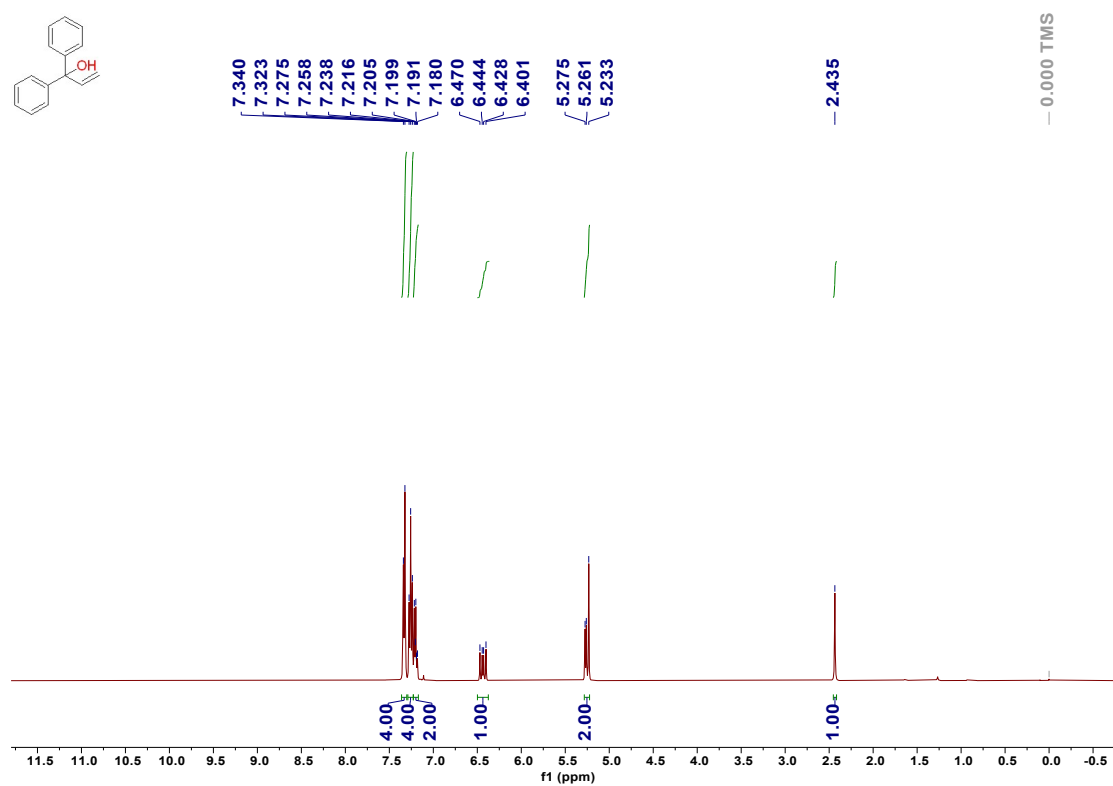
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-27**



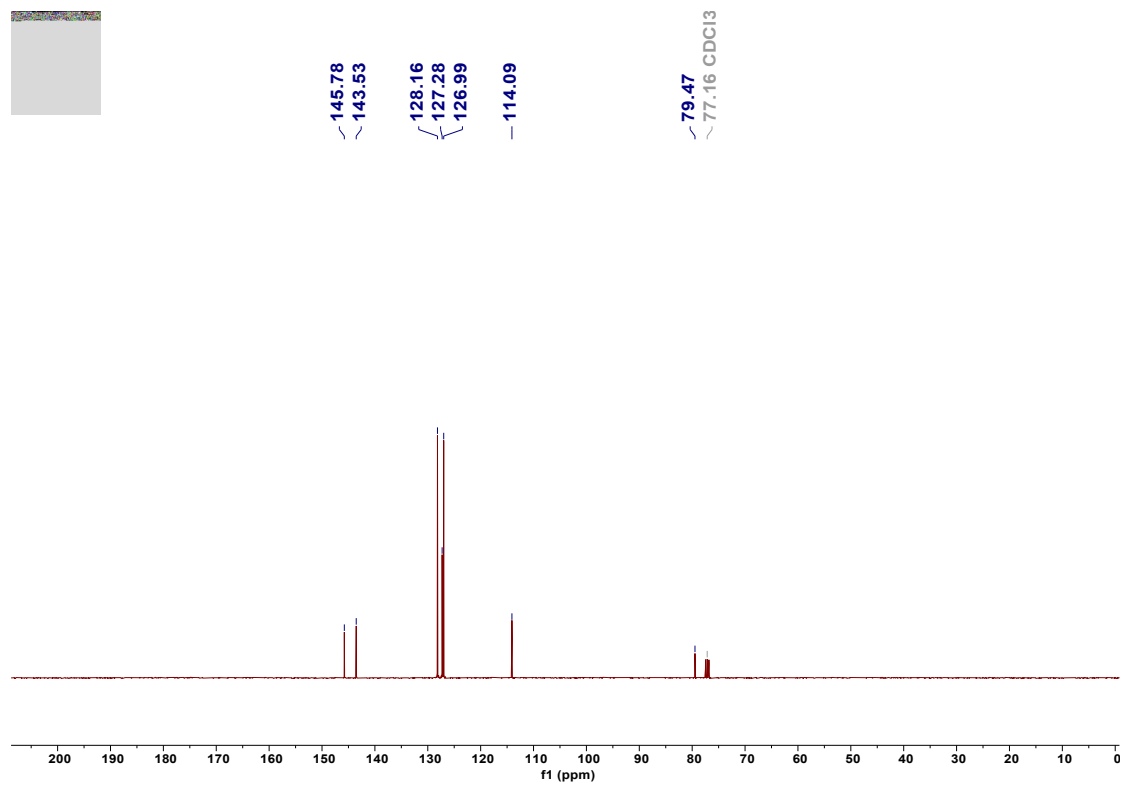
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-28**



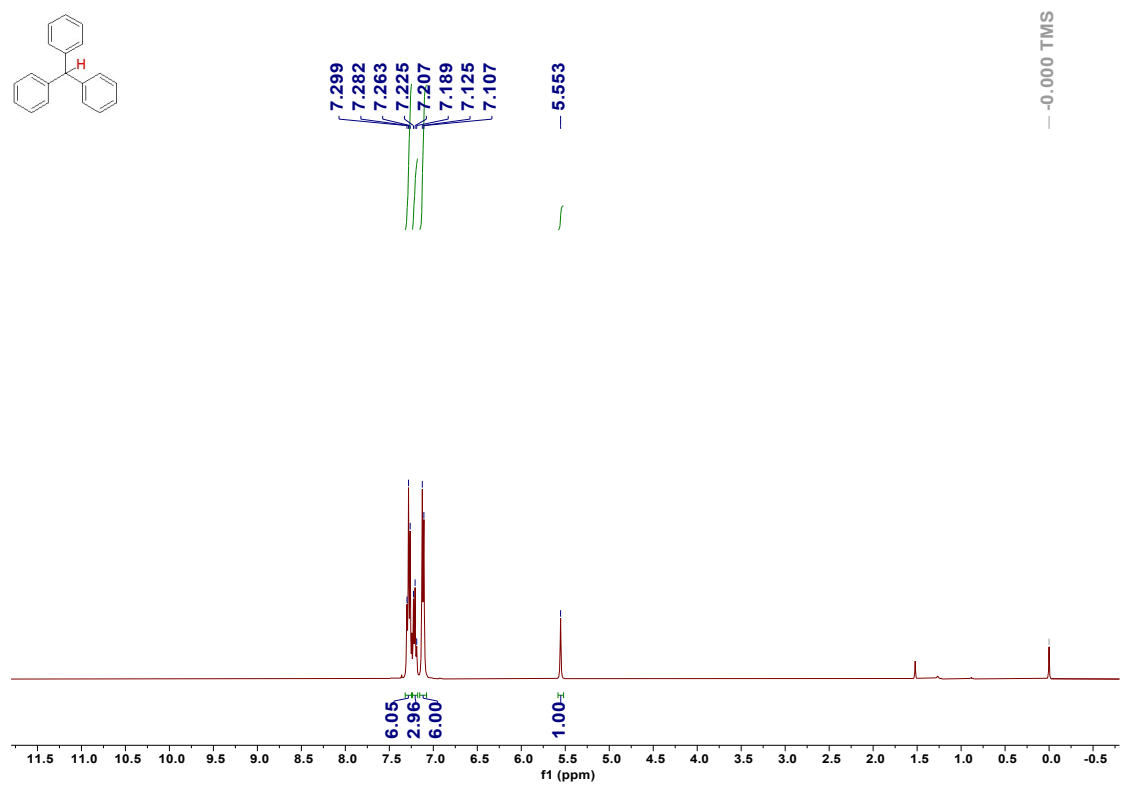
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-28**



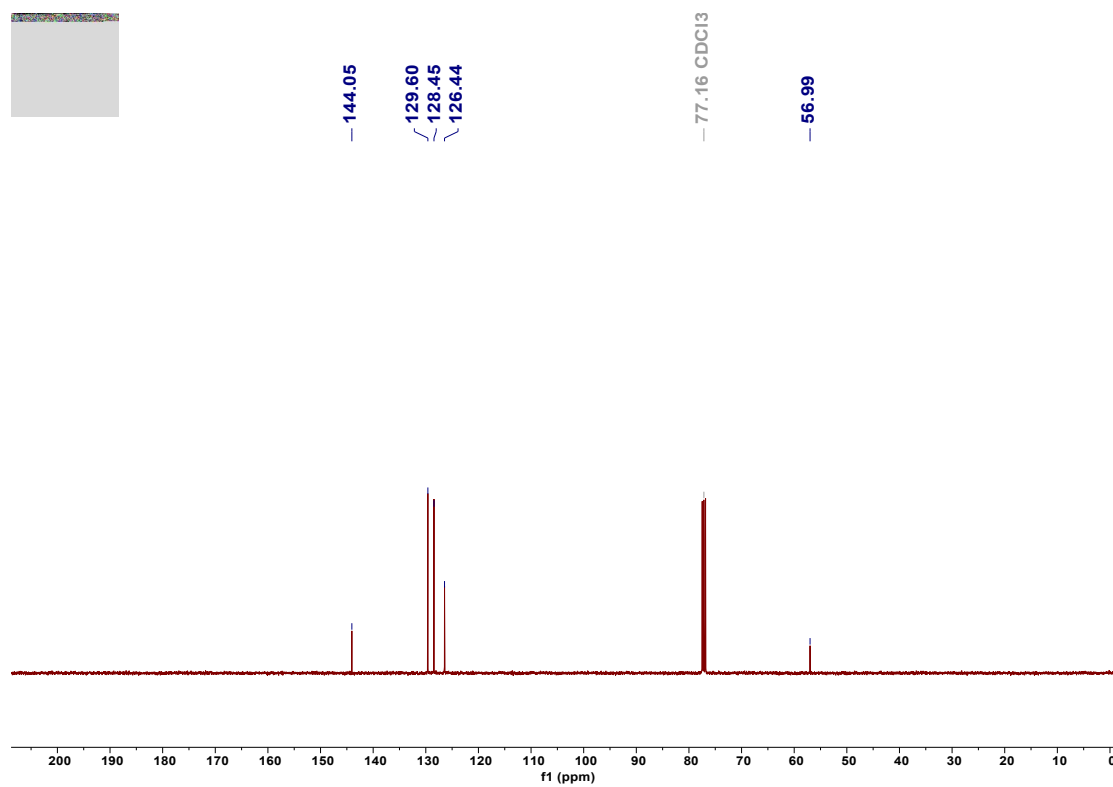
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **1-29**



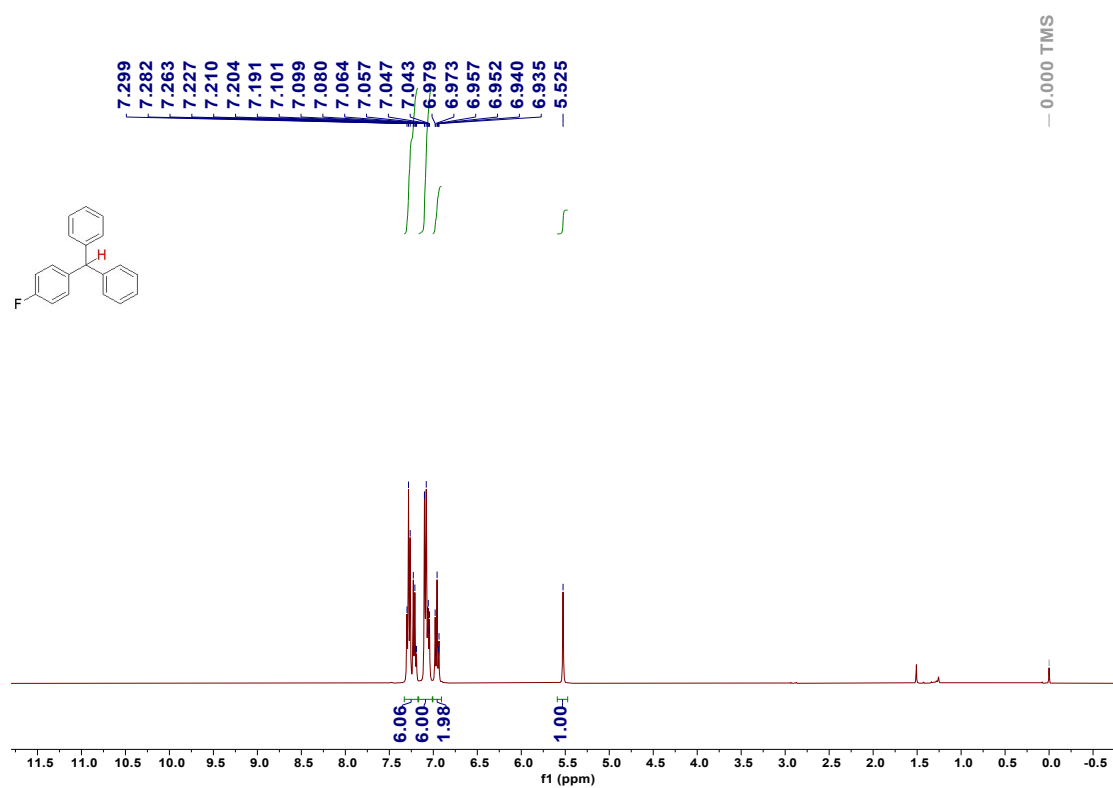
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **1-29**



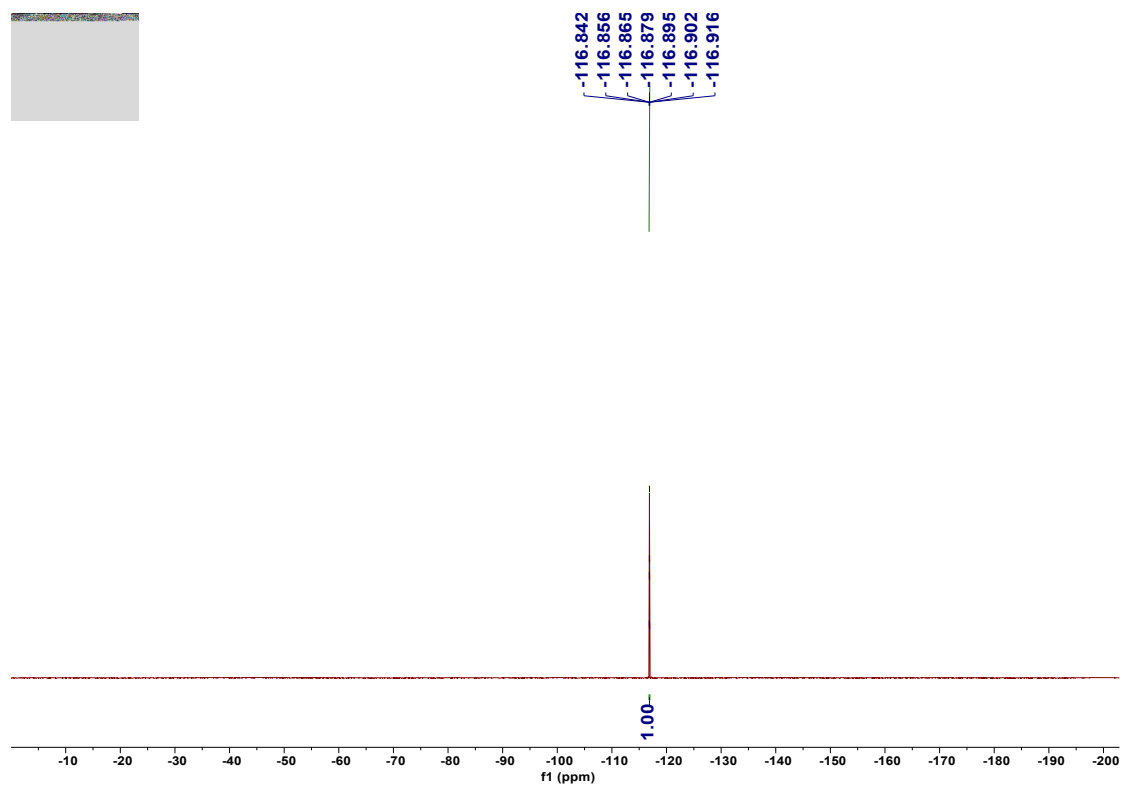
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-1**



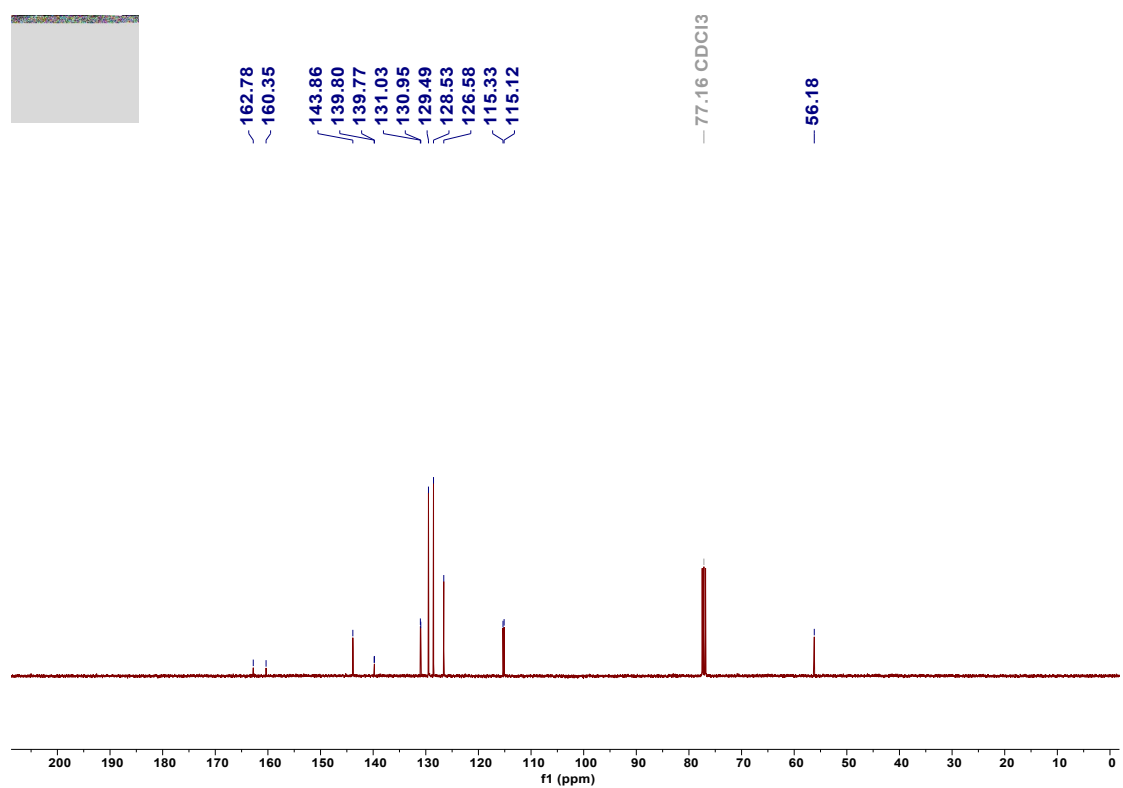
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-1**



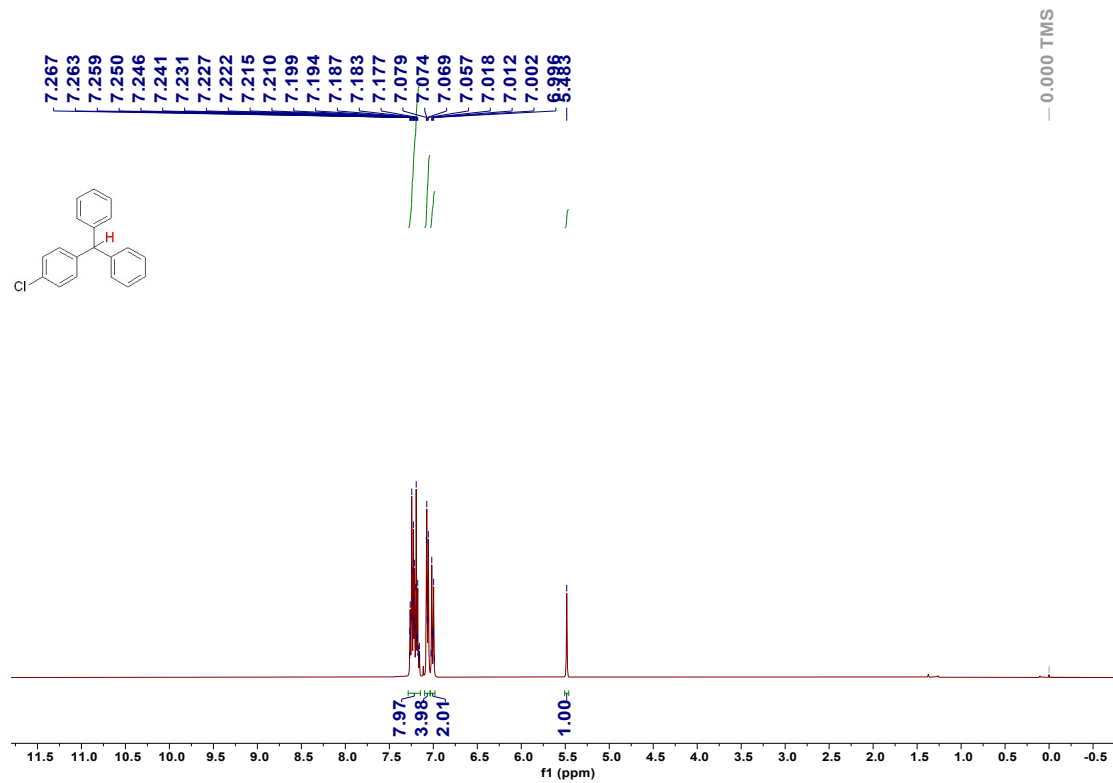
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-2**



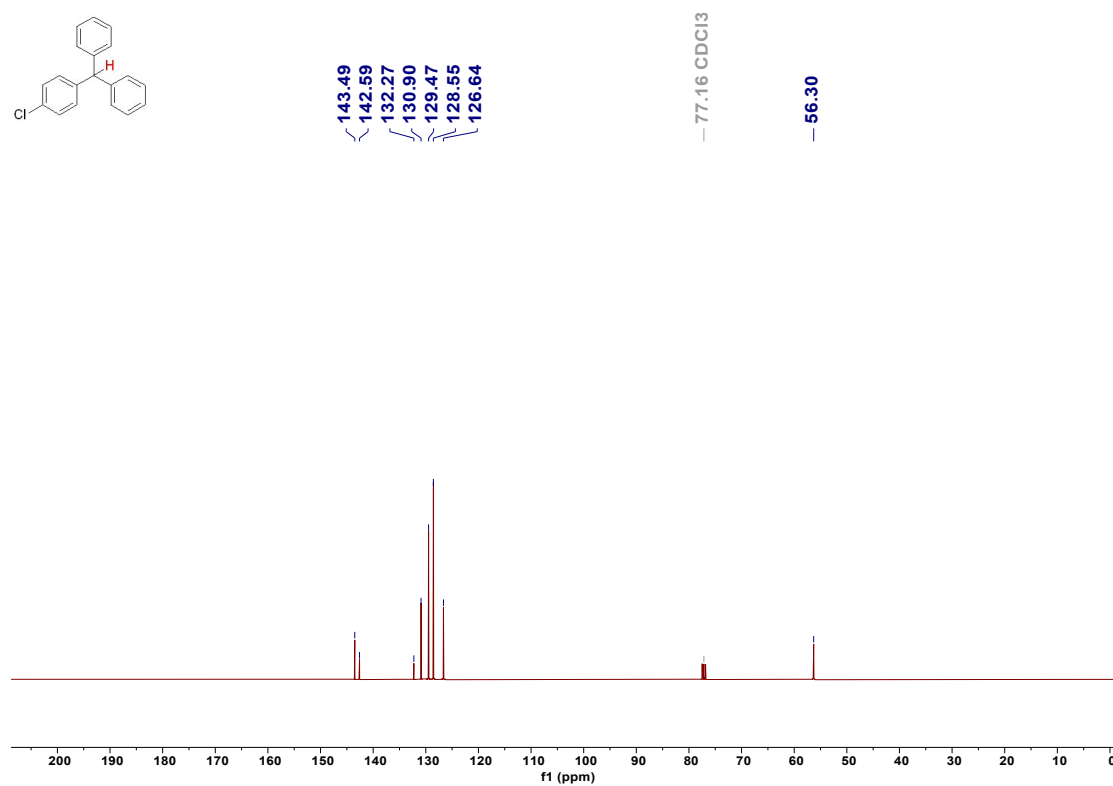
$^{19}\text{F}$  NMR spectra (376 MHz,  $\text{CDCl}_3$ ) of **2-2**



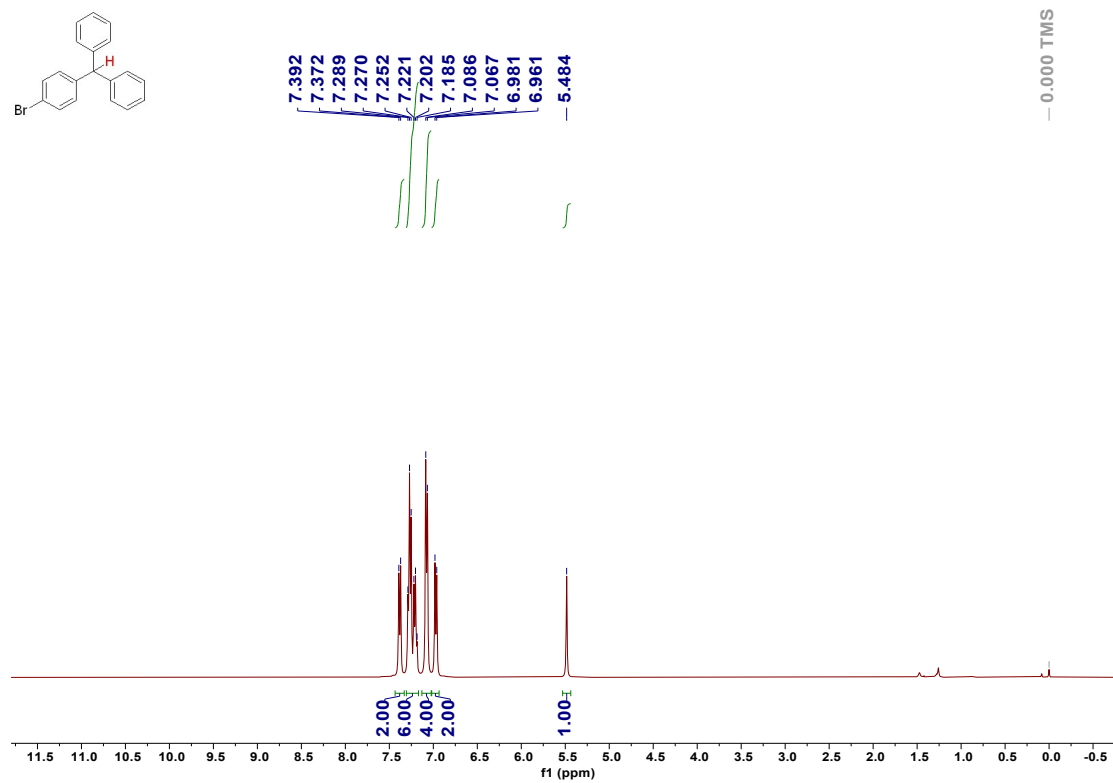
$^{13}\text{C}$  NMR spectra (101 MHz,  $\text{CDCl}_3$ ) of **2-2**



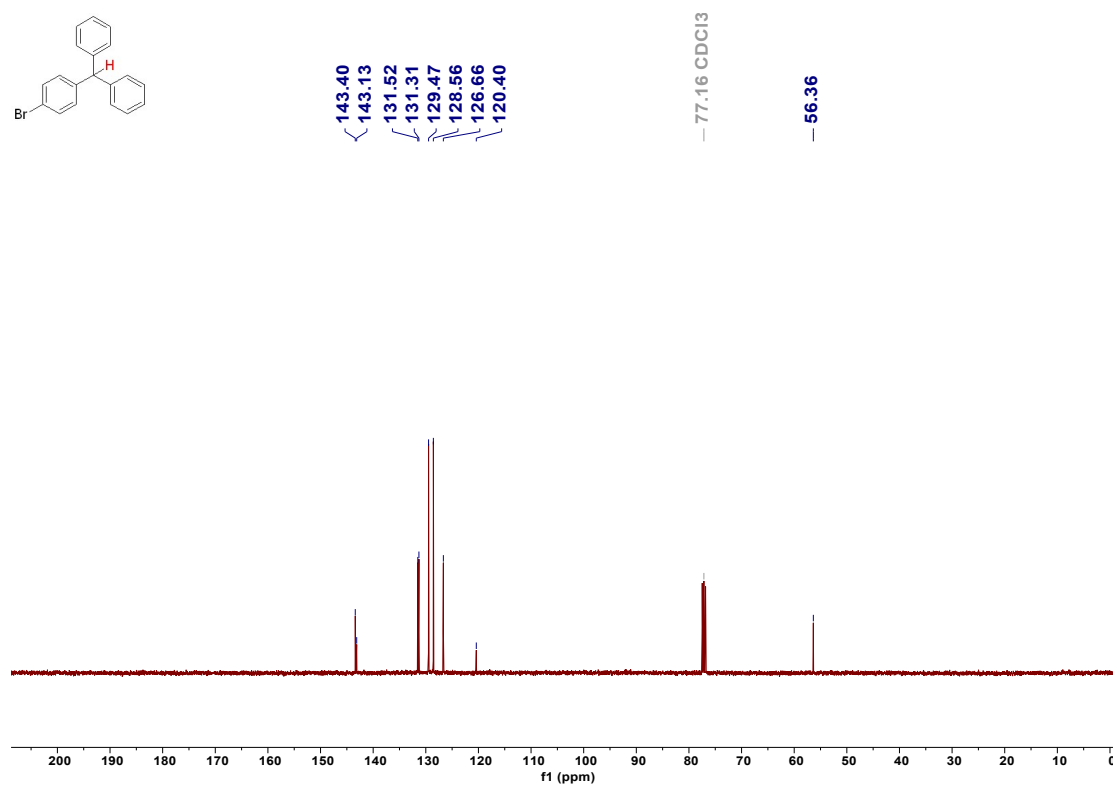
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-3**



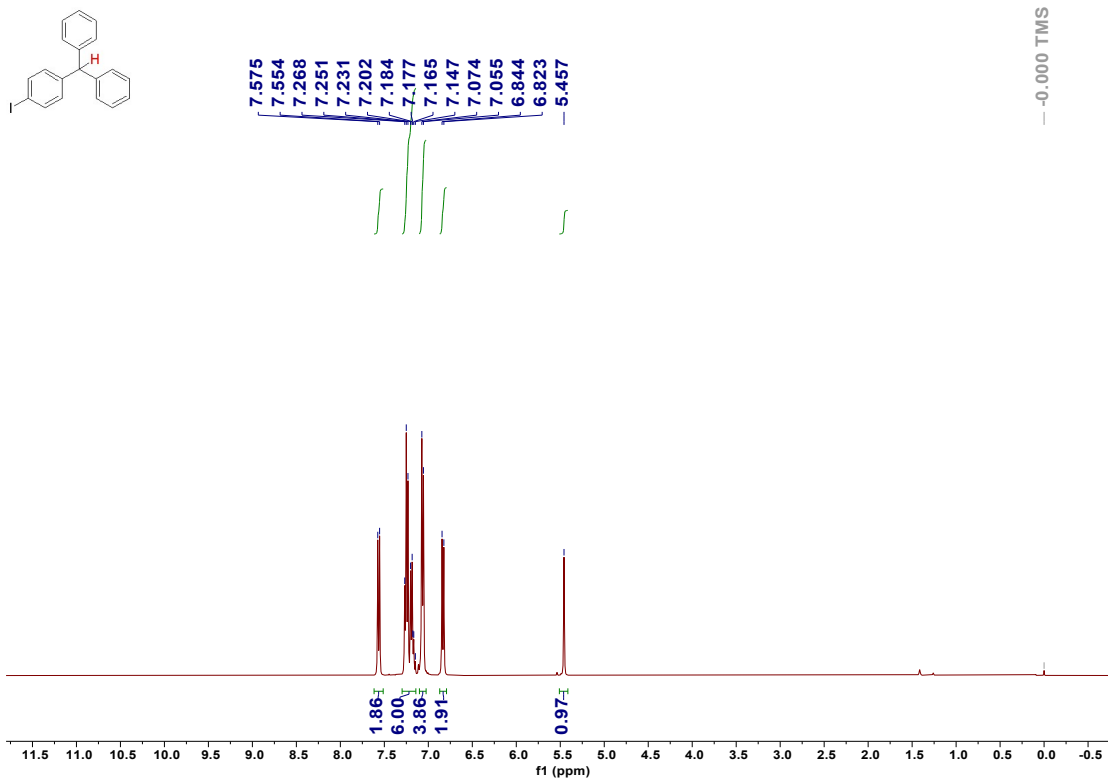
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-3**



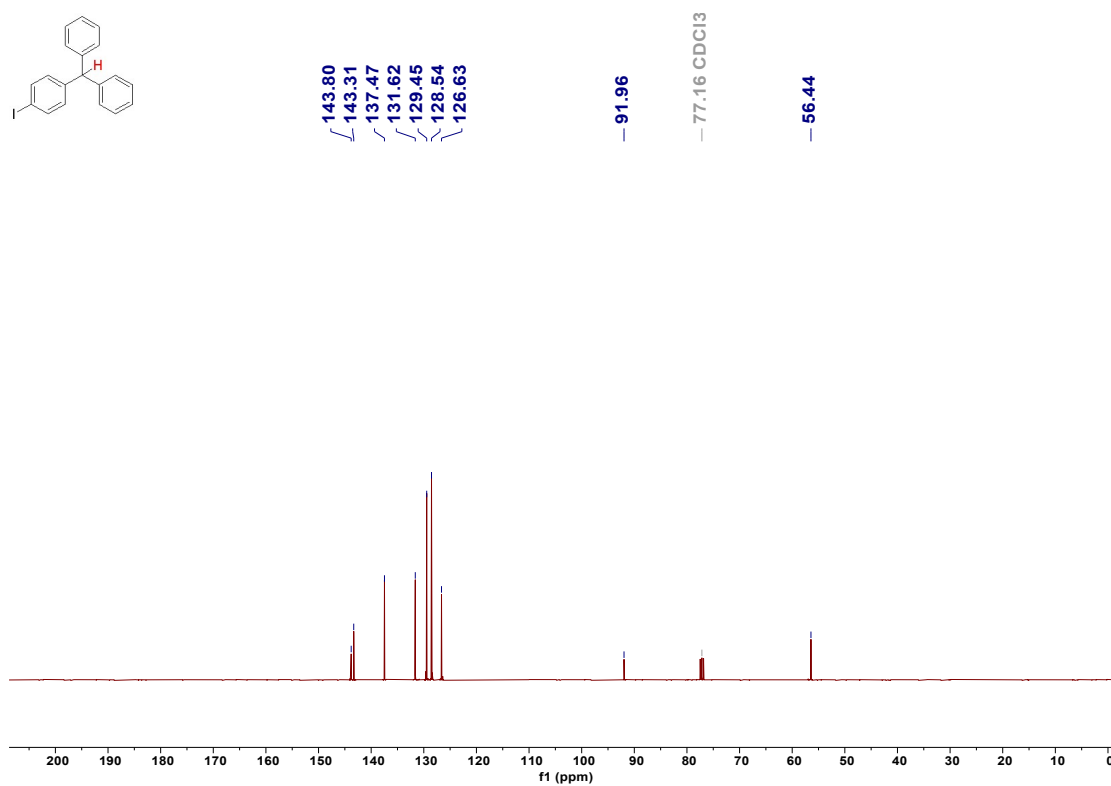
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of 2-4



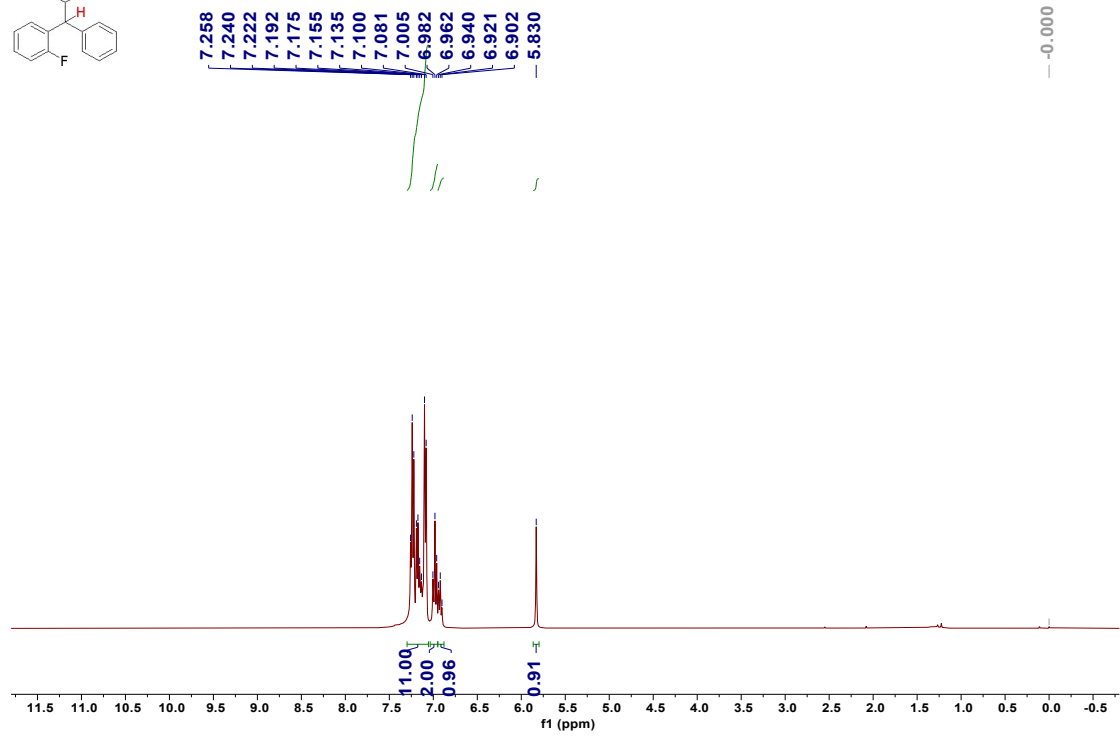
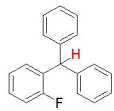
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of 2-4



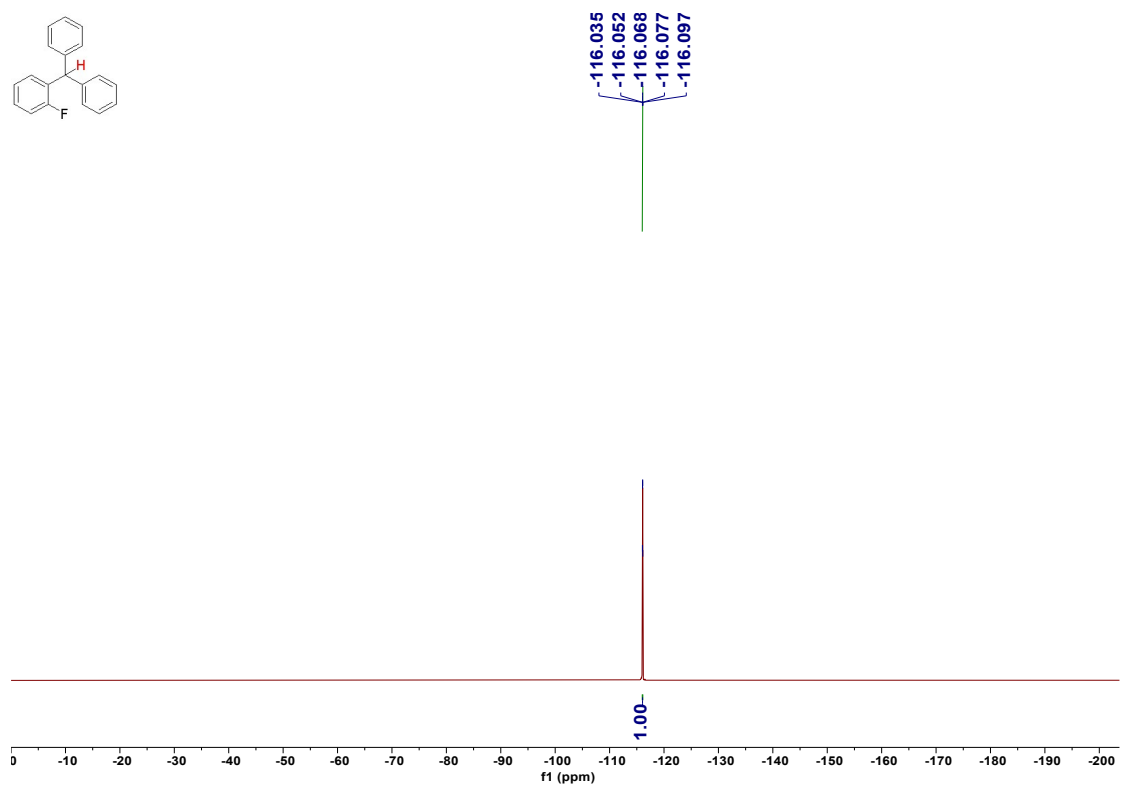
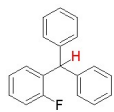
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-5**



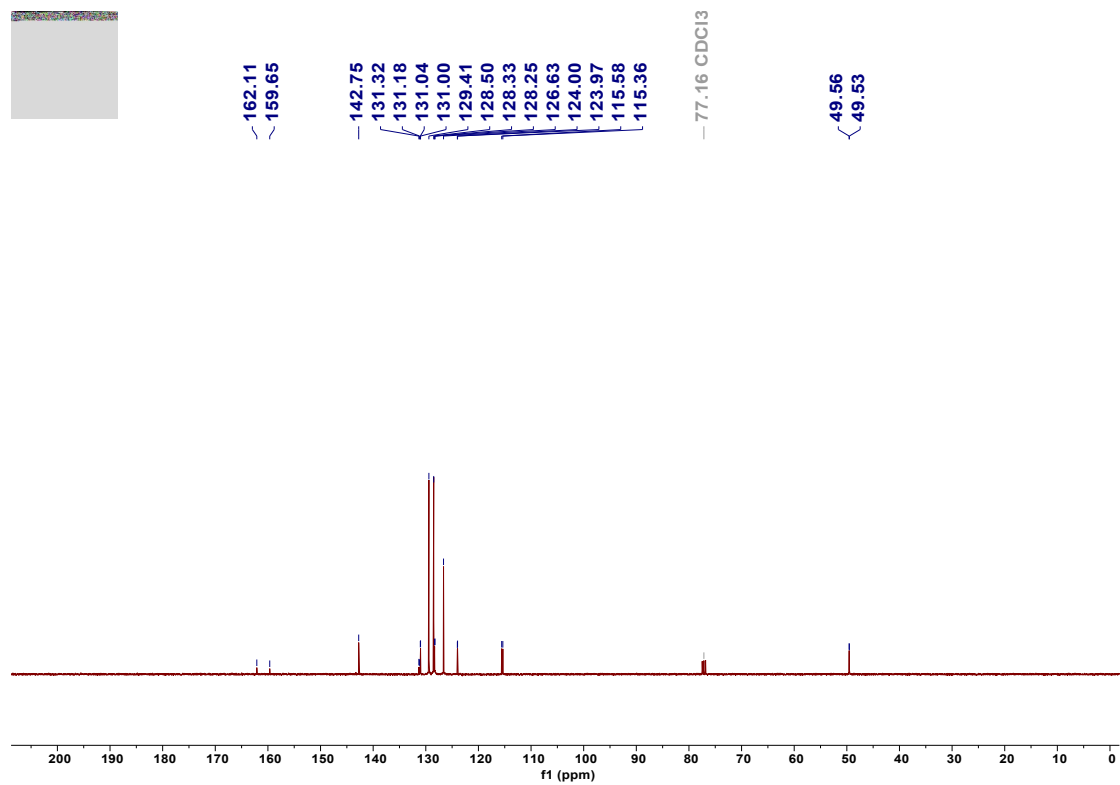
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-5**



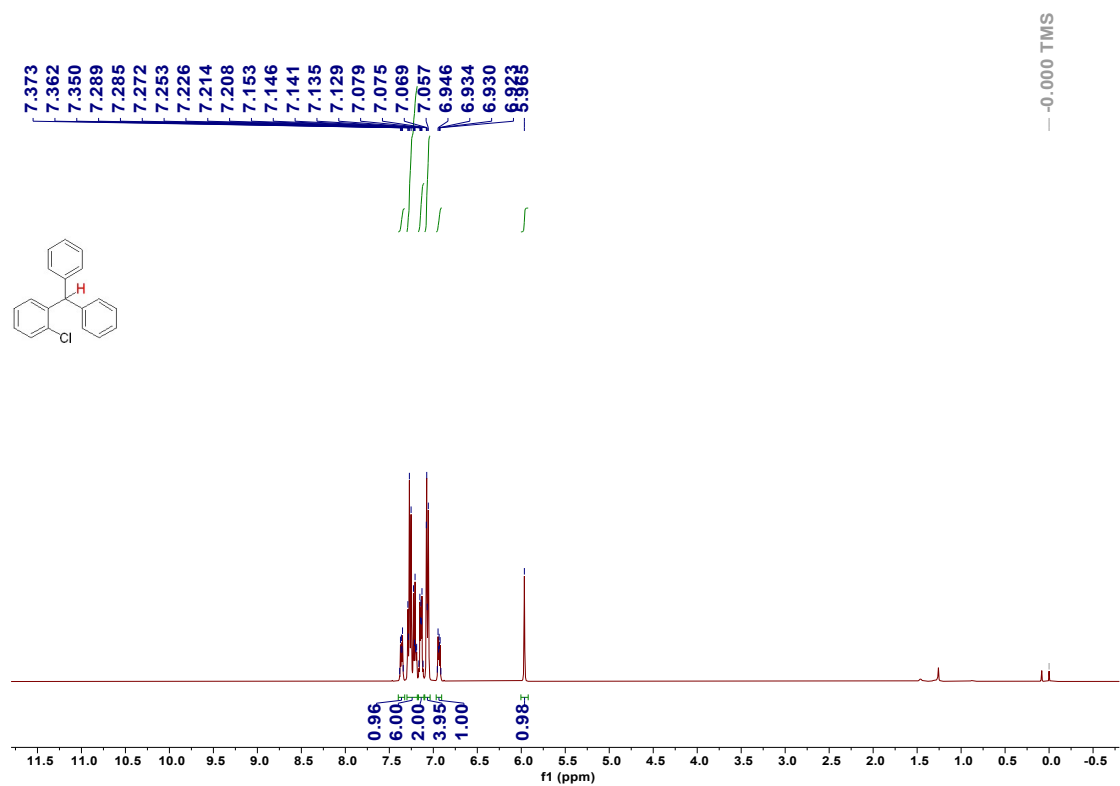
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of 2-6



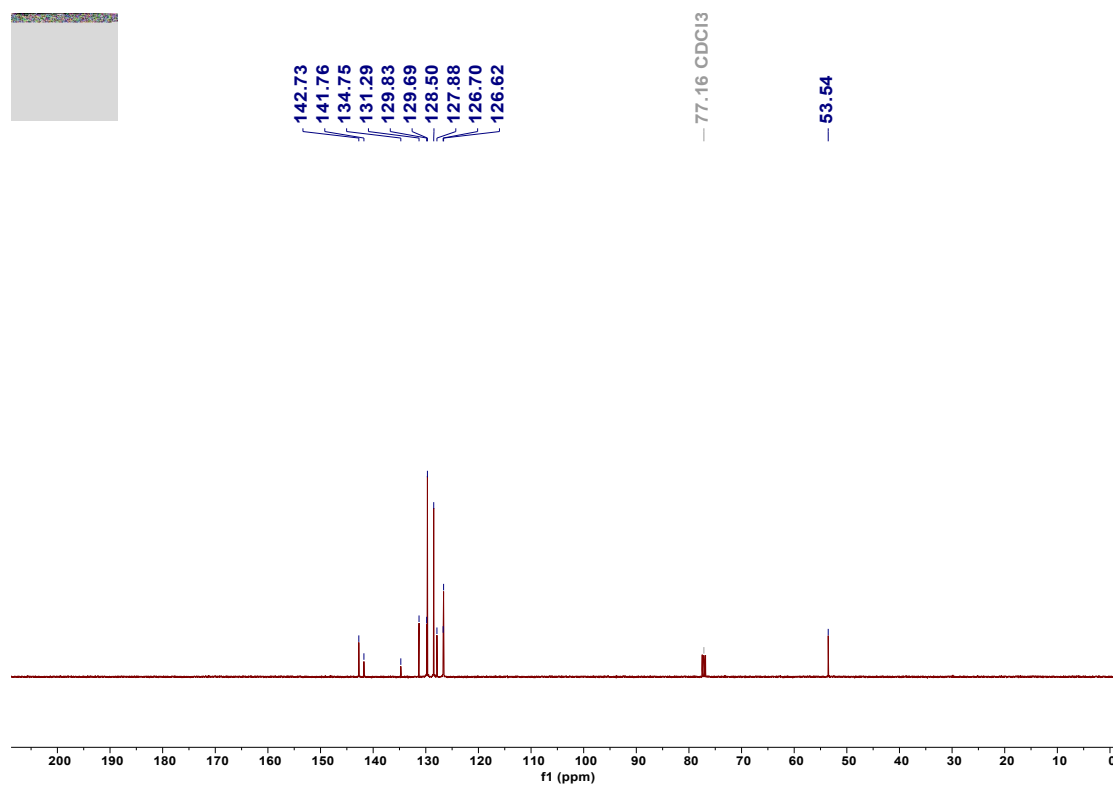
<sup>19</sup>F NMR spectra (376 MHz, CDCl<sub>3</sub>) of 2-6



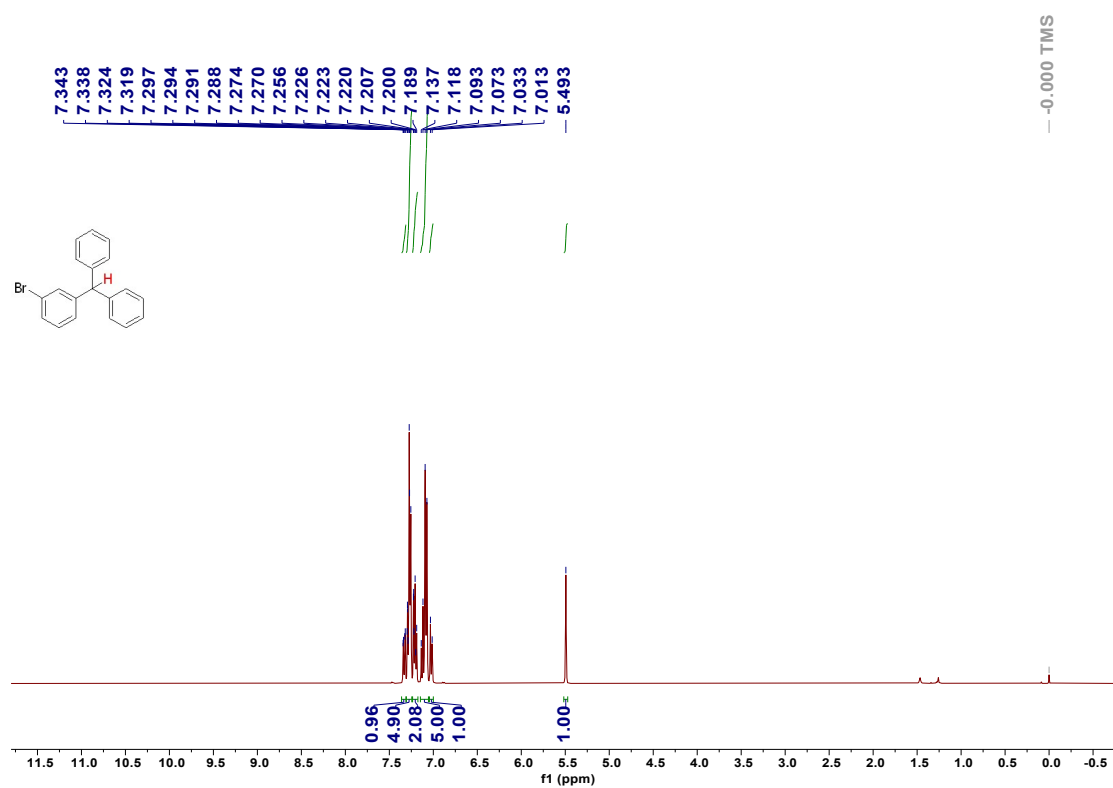
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-6**



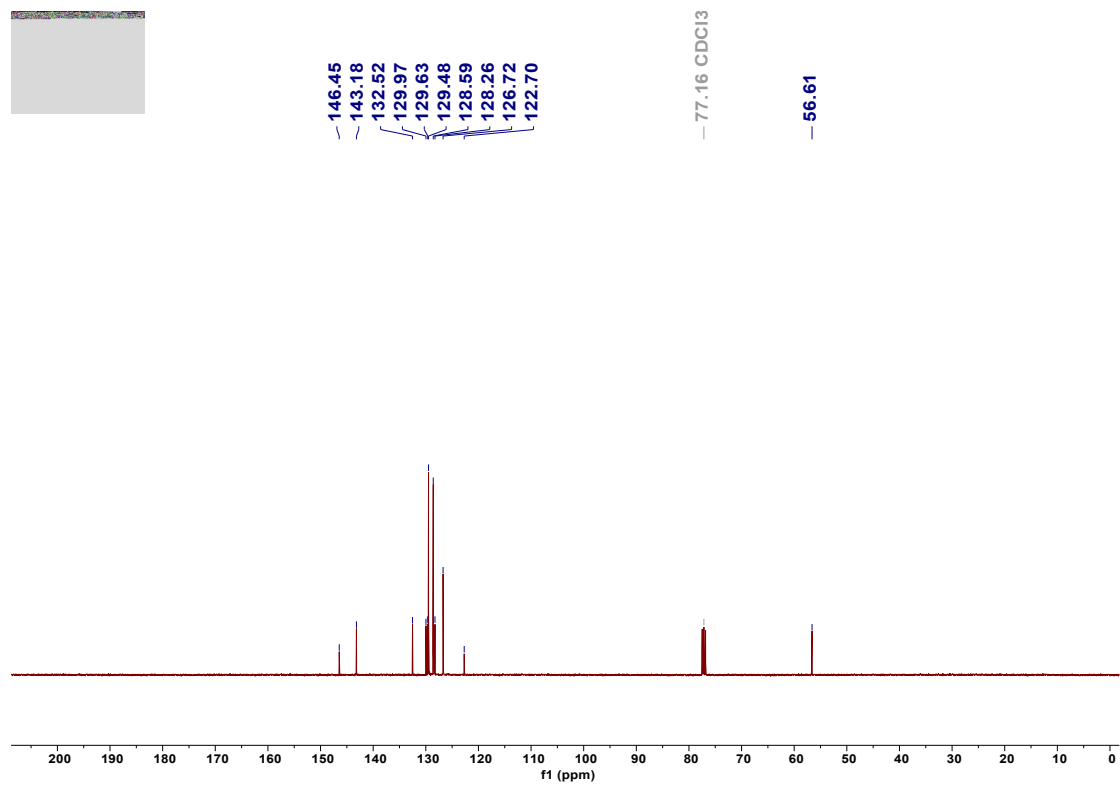
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-7**



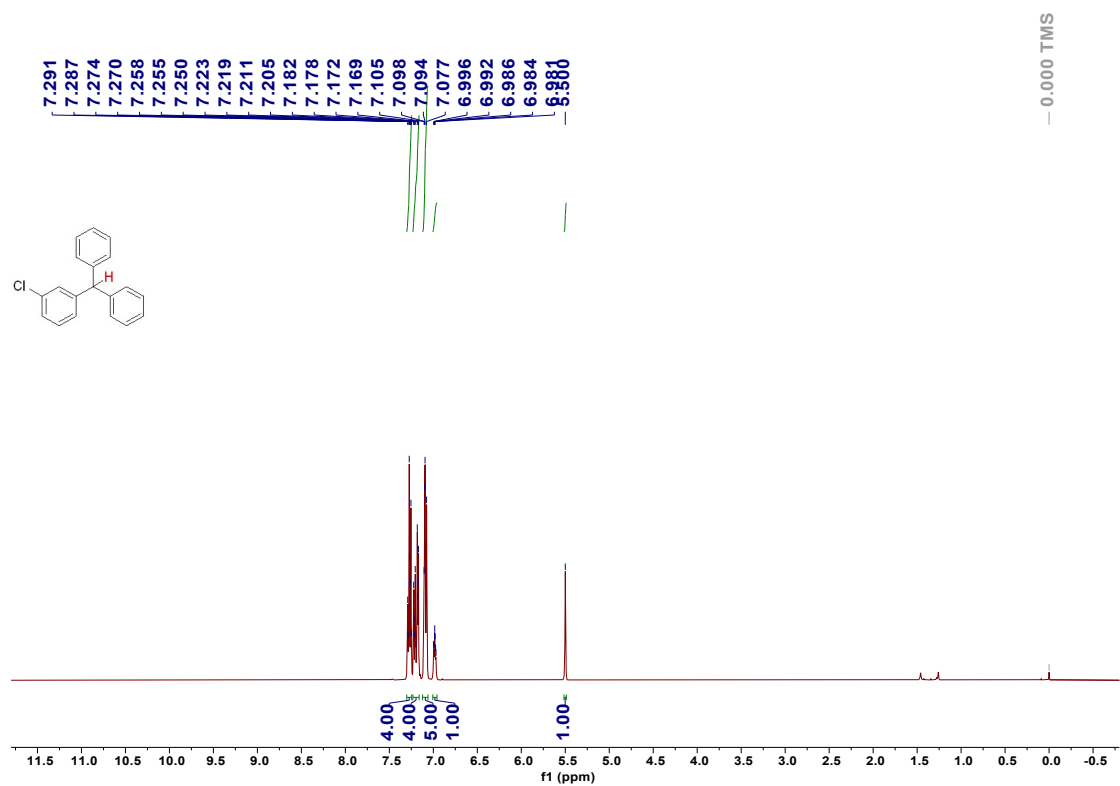
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-7**



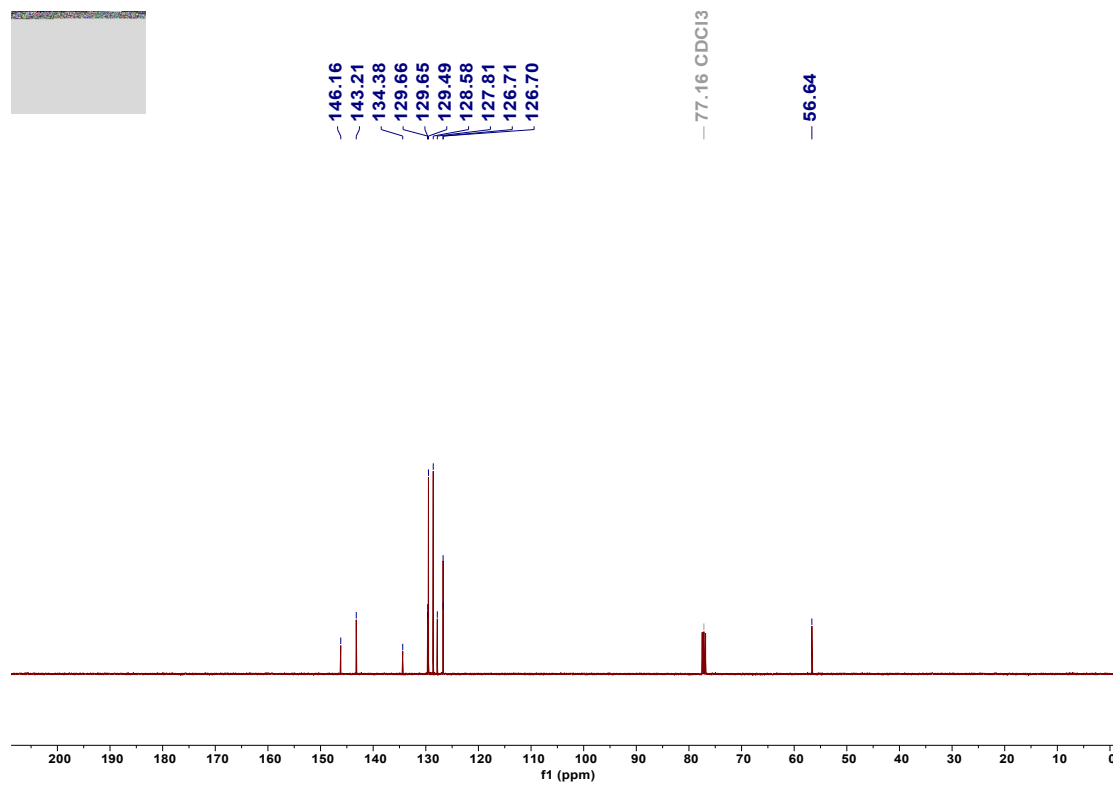
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-8**



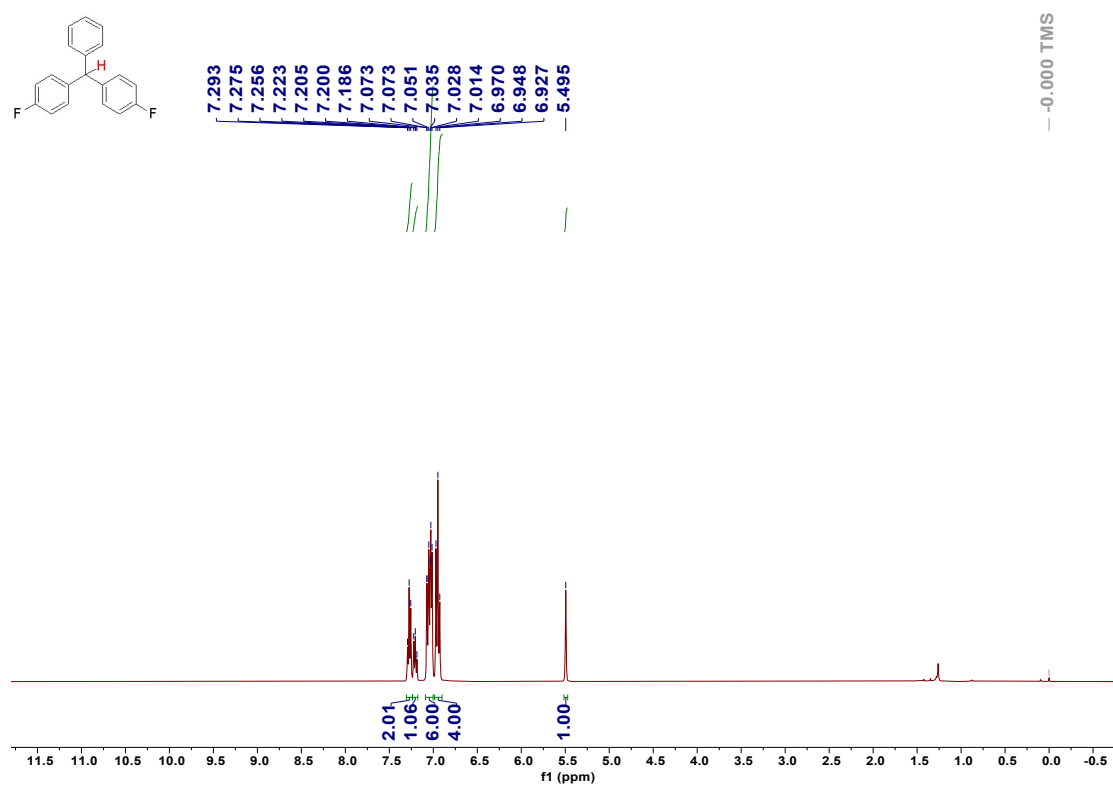
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-8**



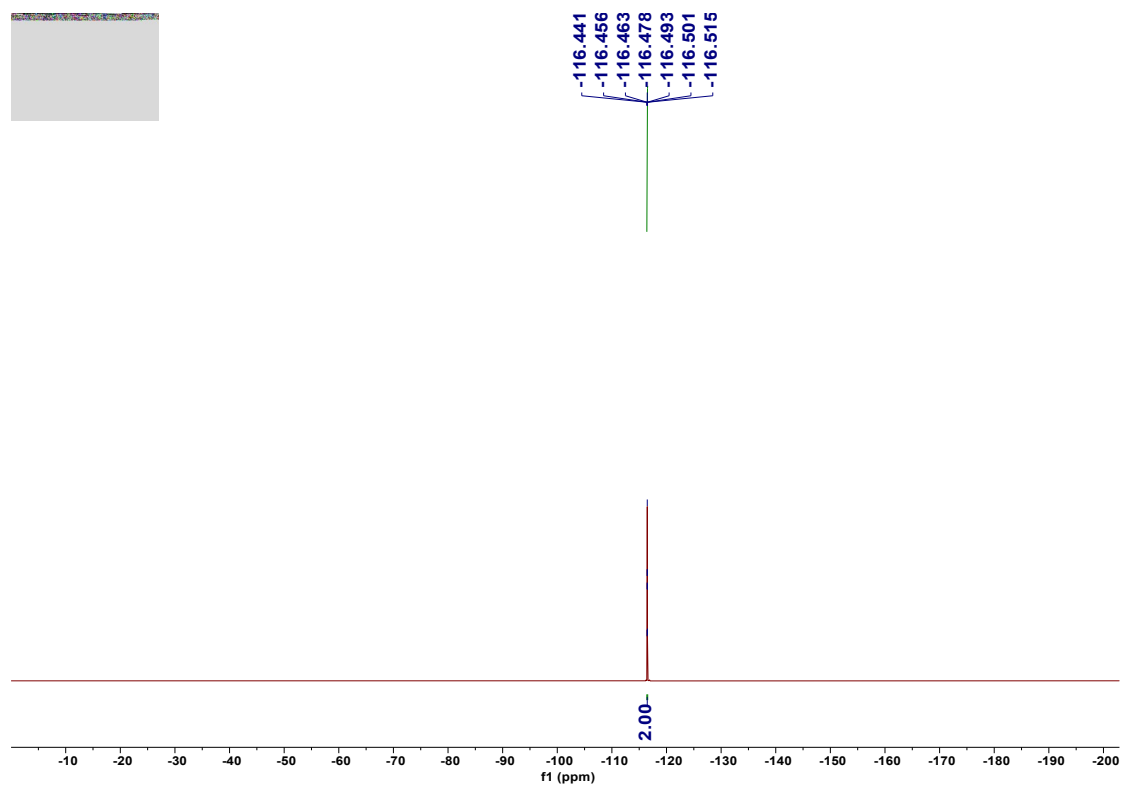
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-9**



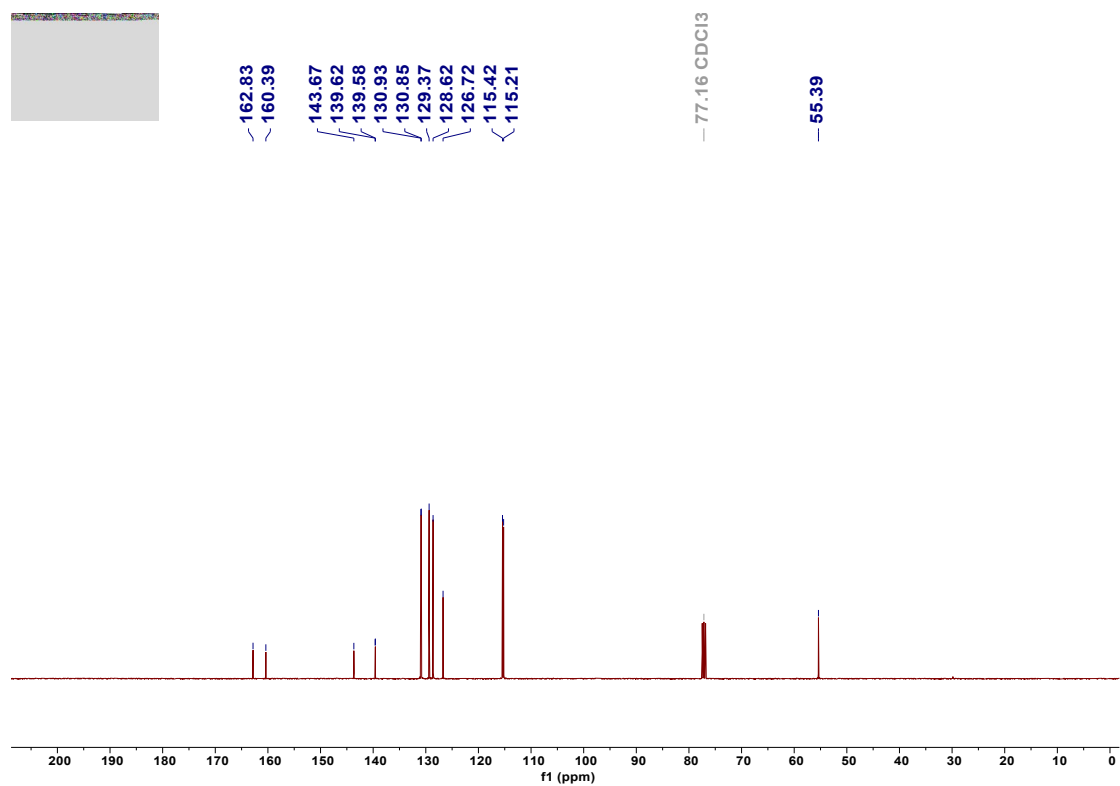
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-9**



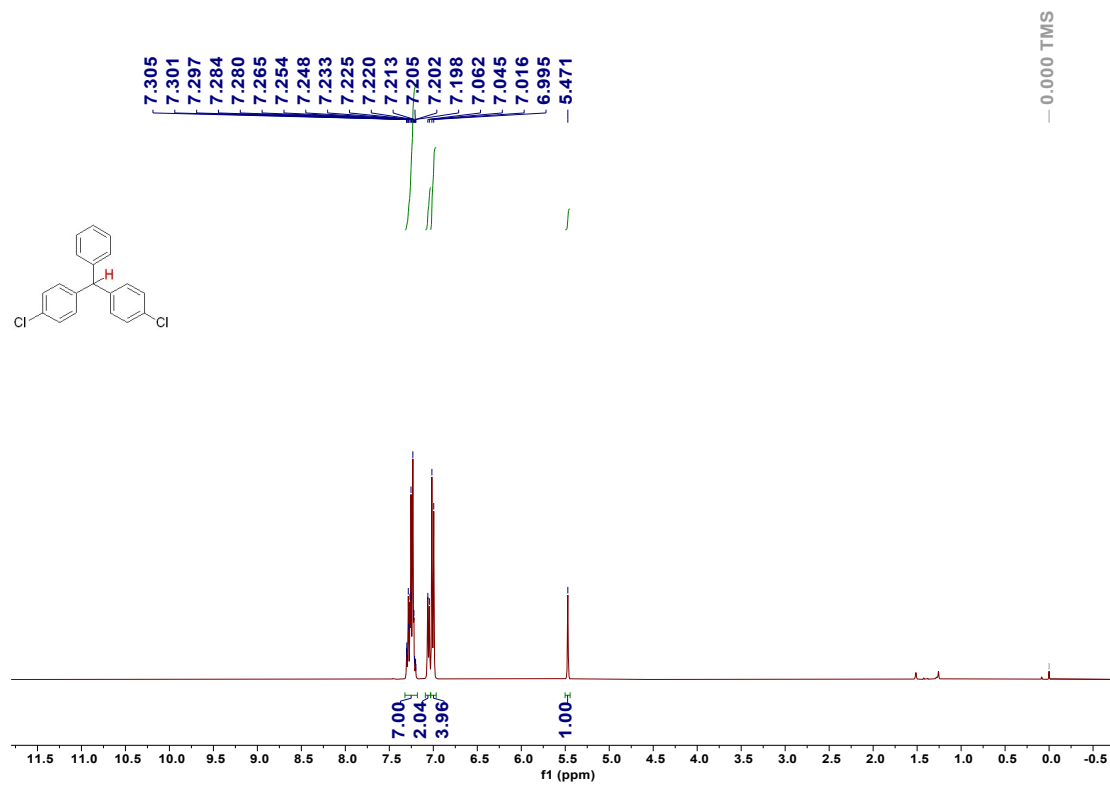
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-10**



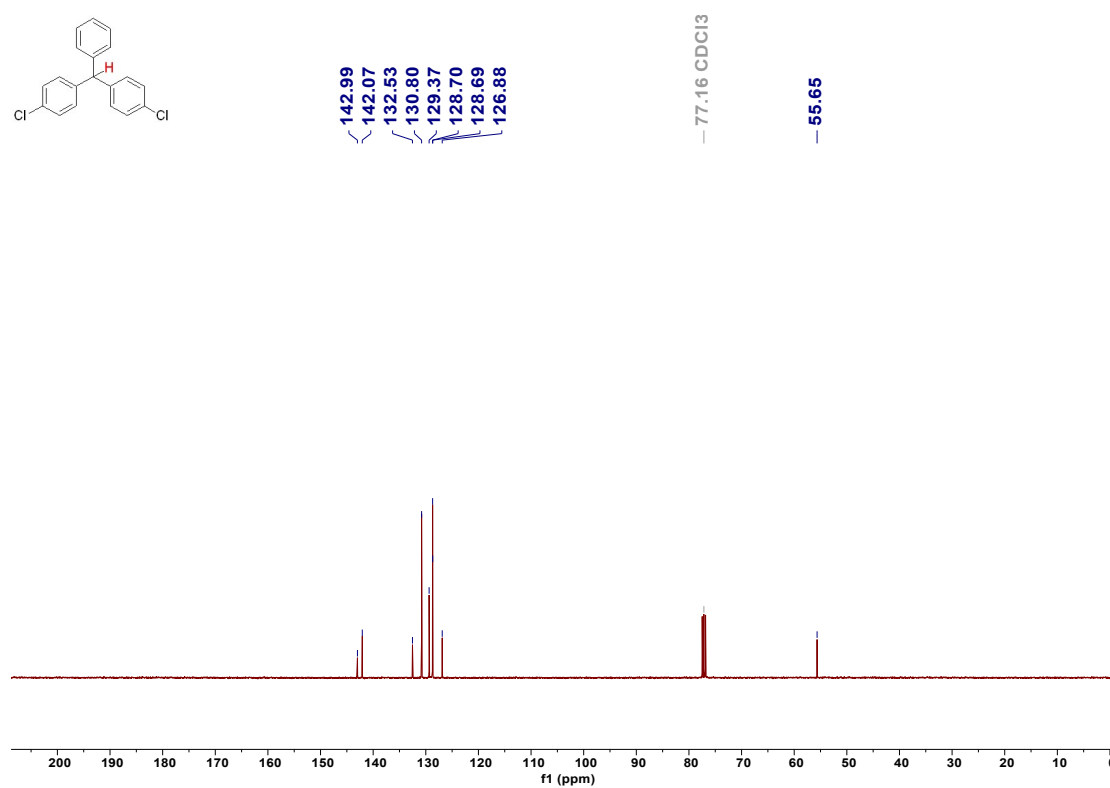
$^{19}\text{F}$  NMR spectra (376 MHz,  $\text{CDCl}_3$ ) of **2-10**



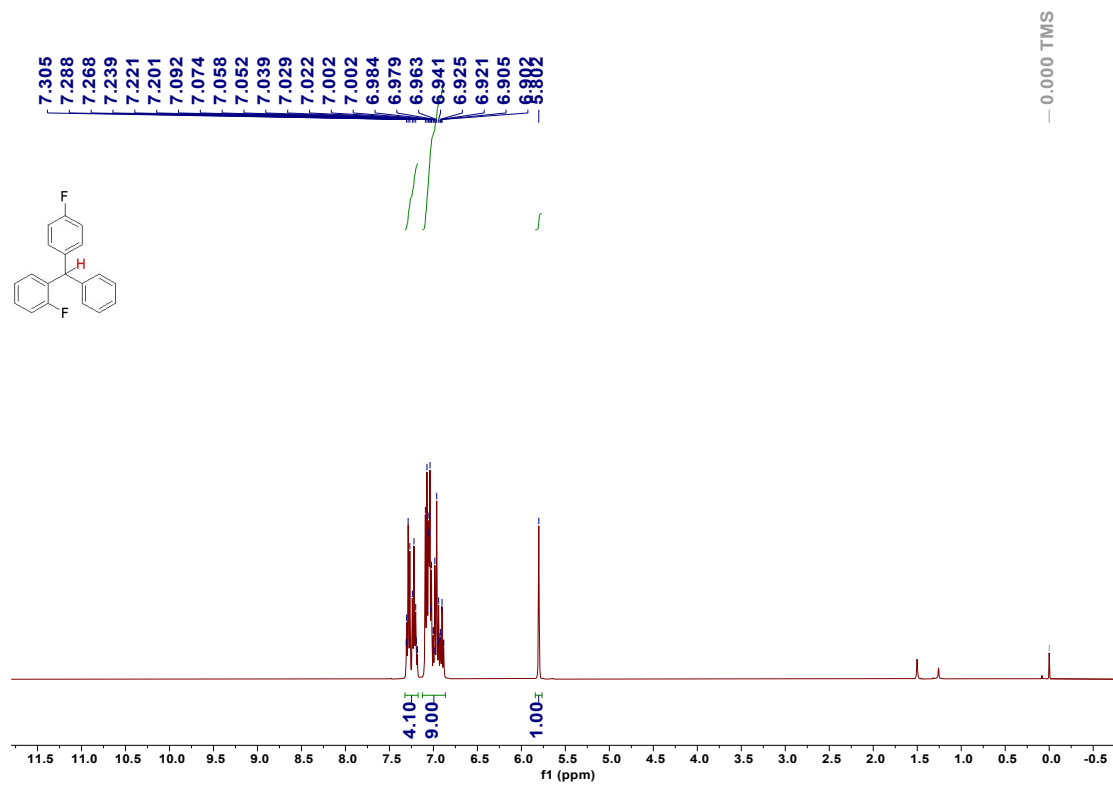
$^{13}\text{C}$  NMR spectra (101 MHz,  $\text{CDCl}_3$ ) of **2-10**



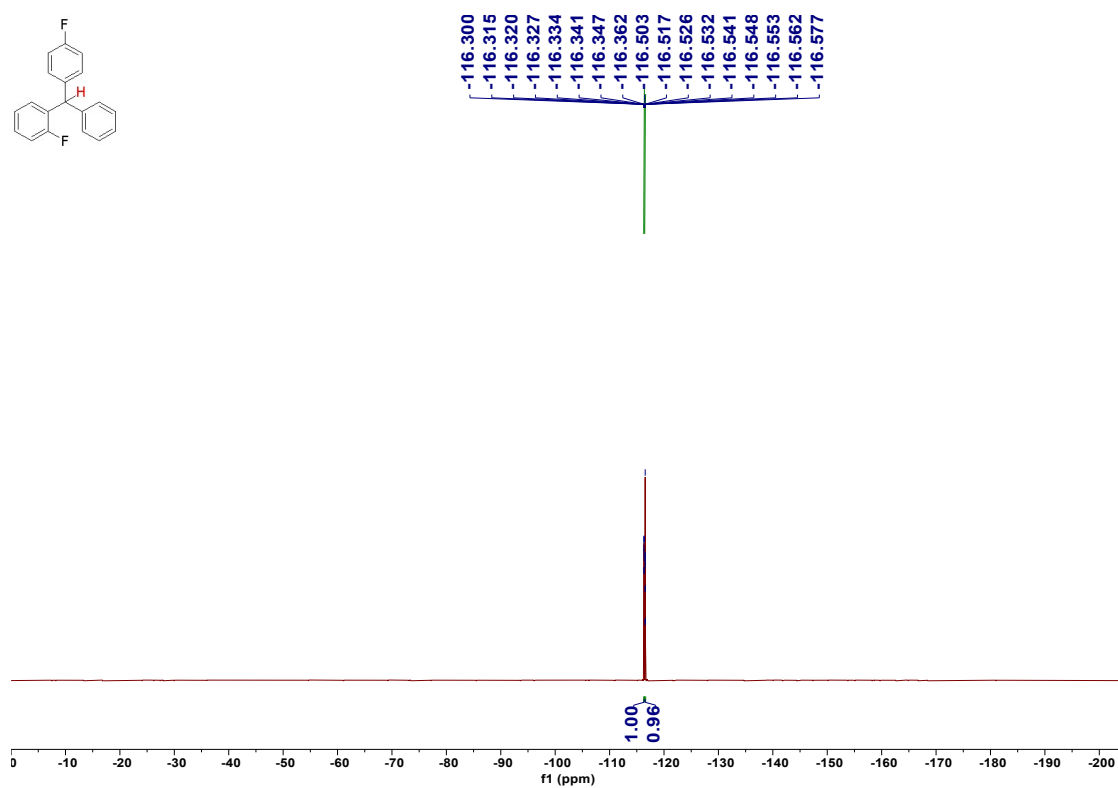
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-11**



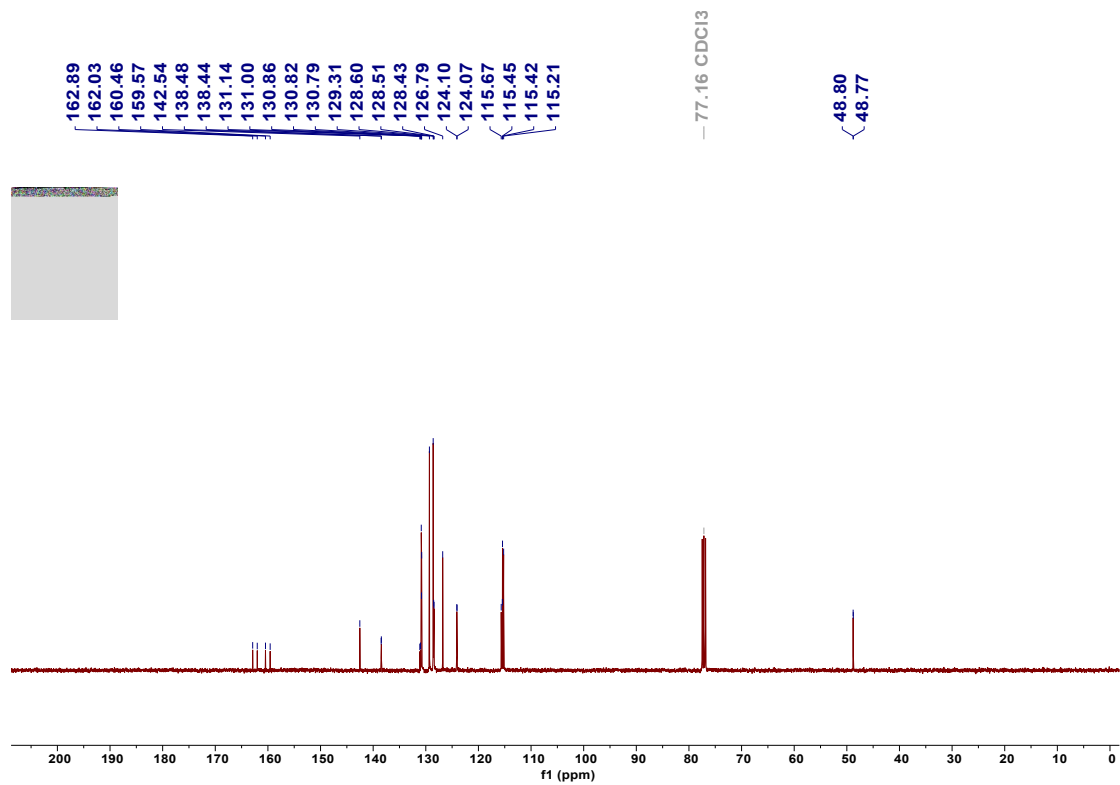
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-11**



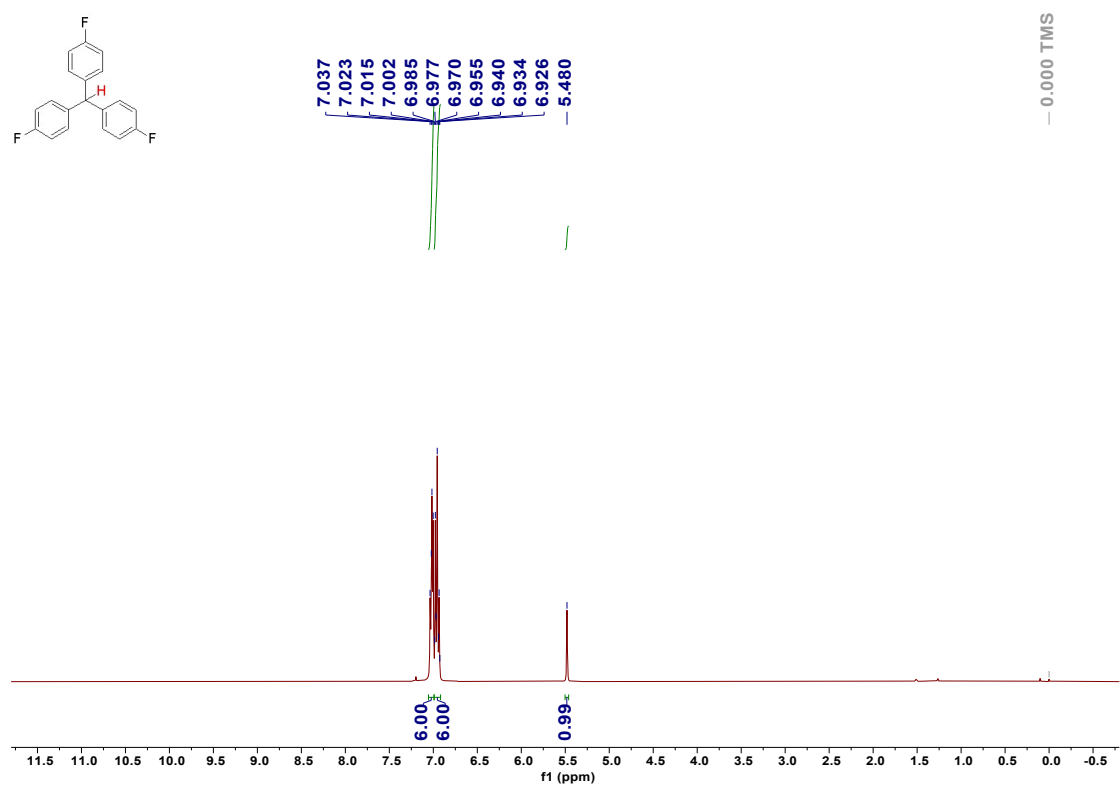
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of 2-12



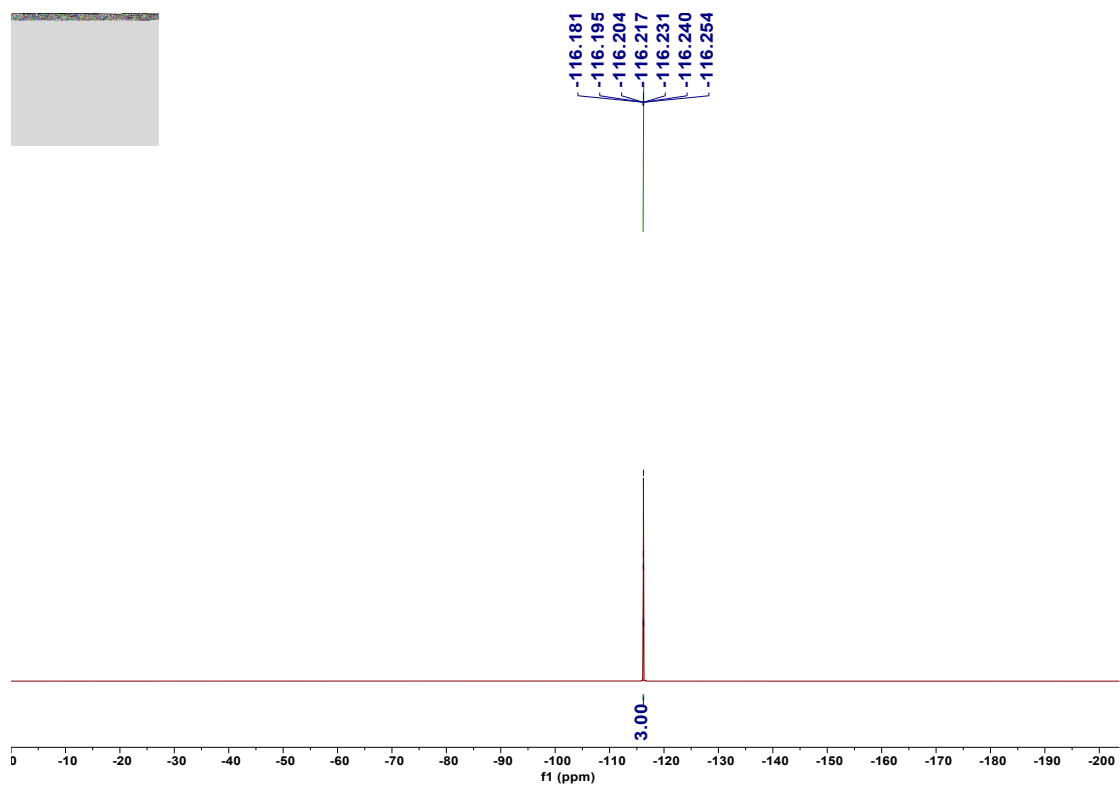
<sup>19</sup>F NMR spectra (376 MHz, CDCl<sub>3</sub>) of 2-12



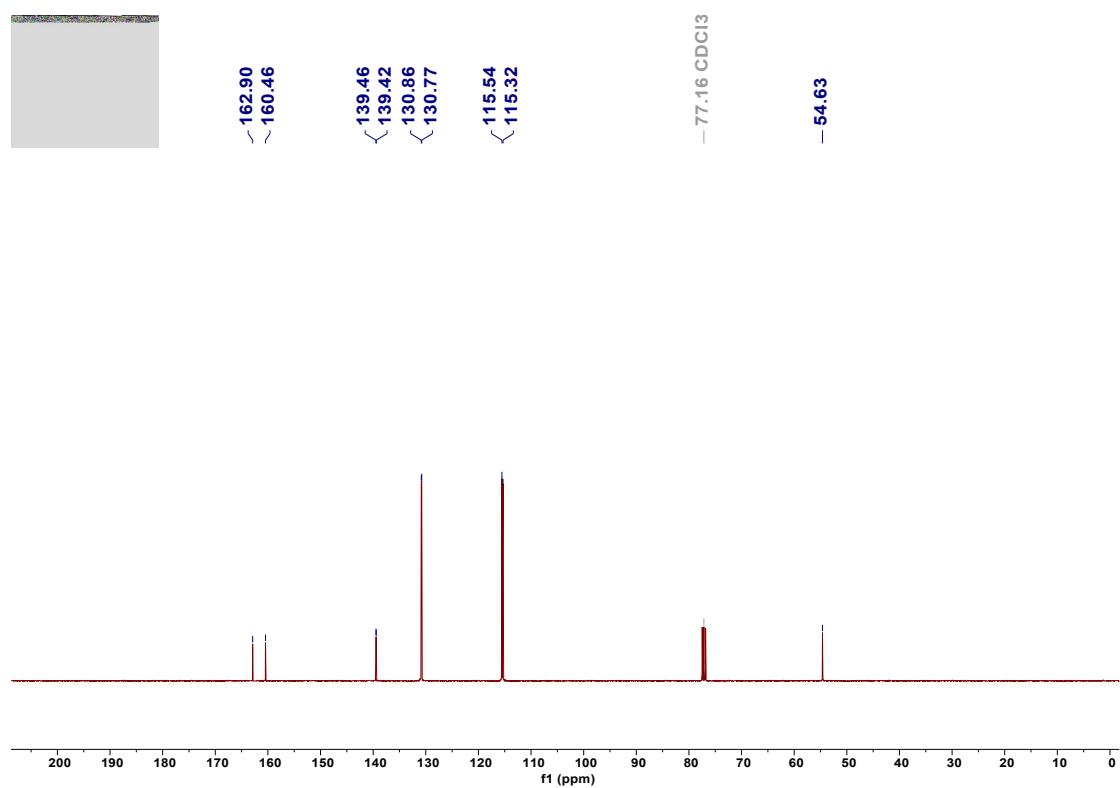
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-12**



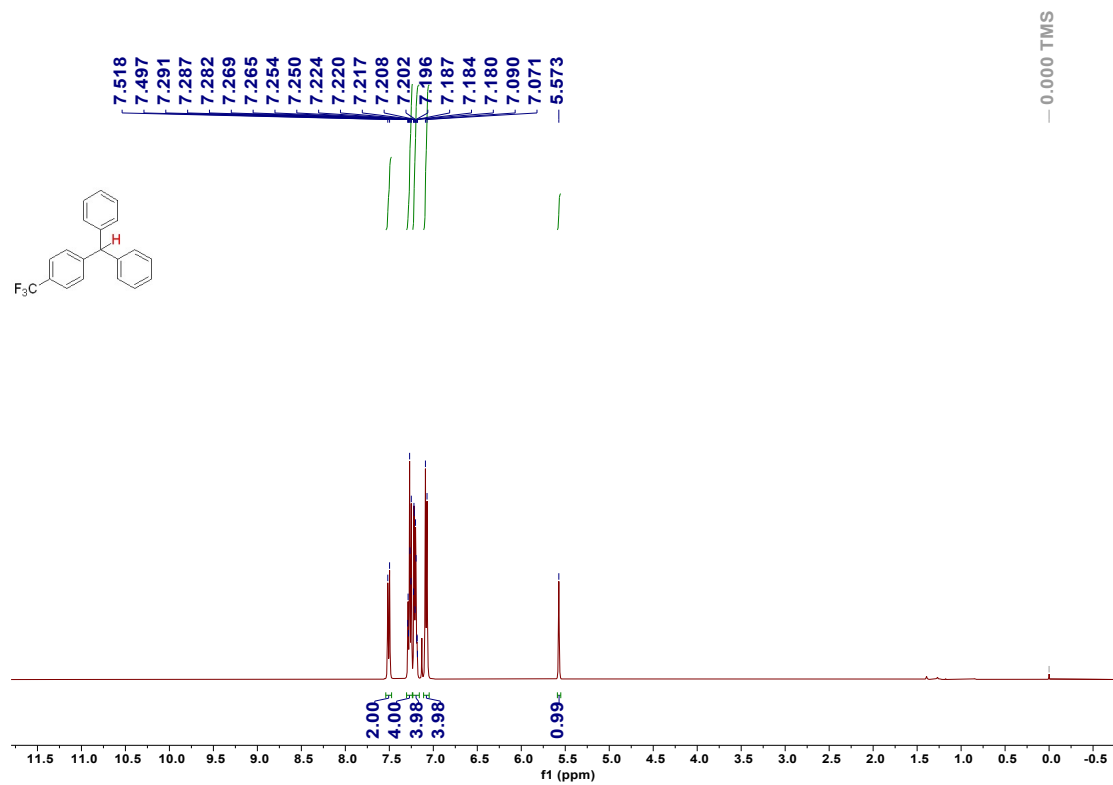
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-13**



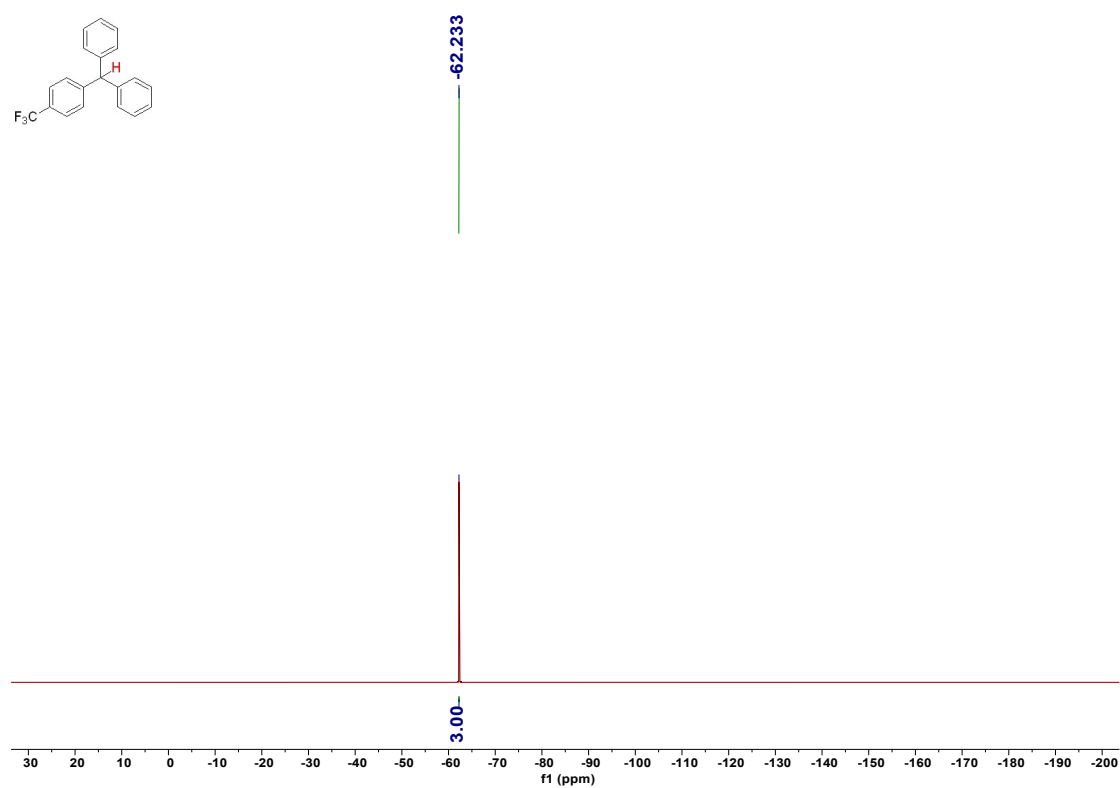
<sup>19</sup>F NMR spectra (376 MHz, CDCl<sub>3</sub>) of **2-13**



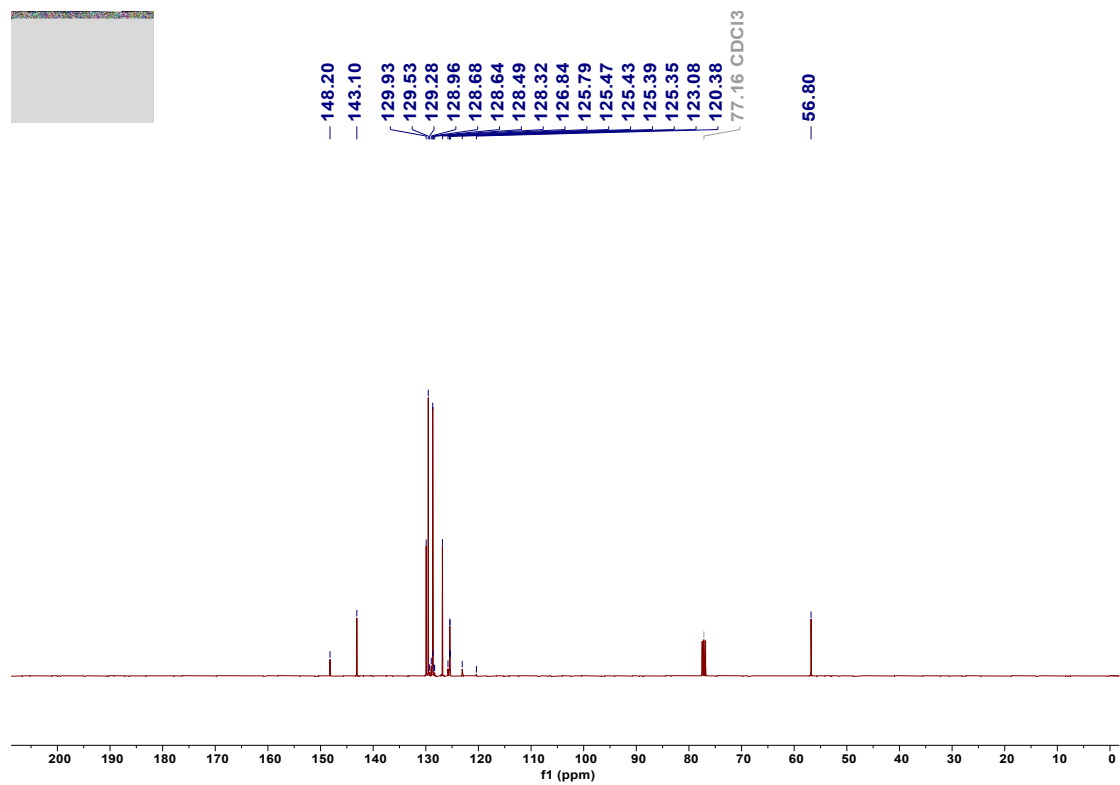
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-13**



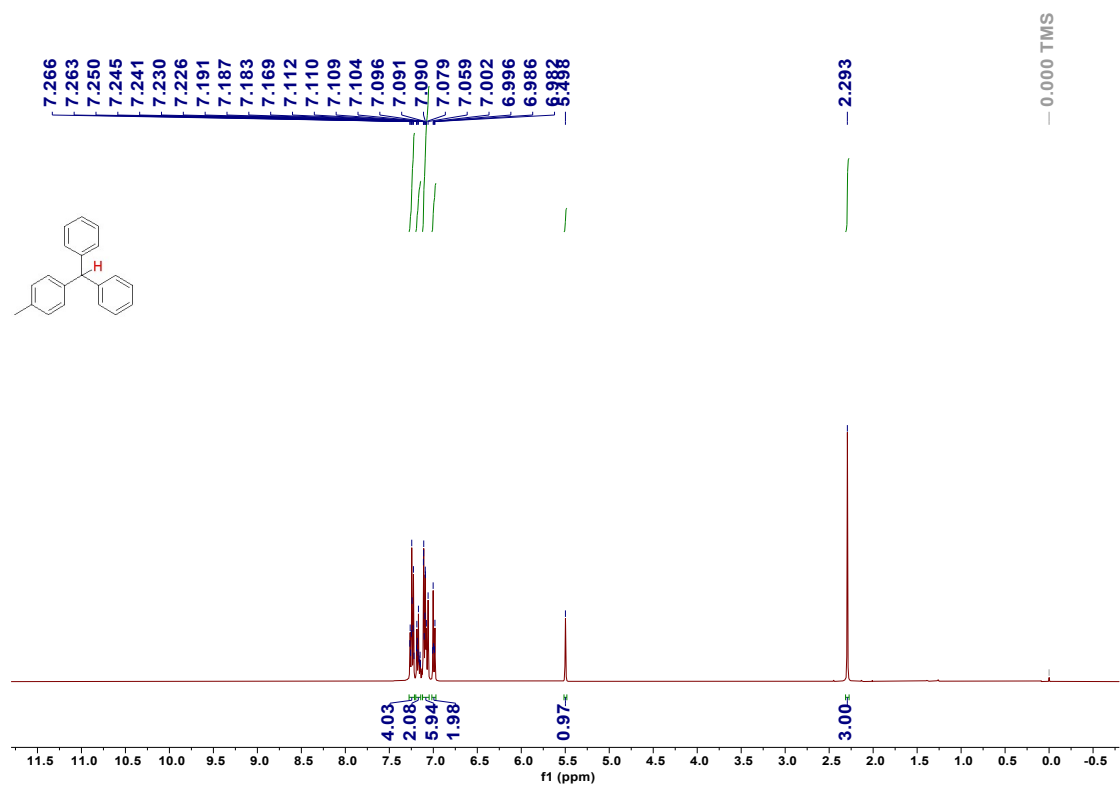
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-14**



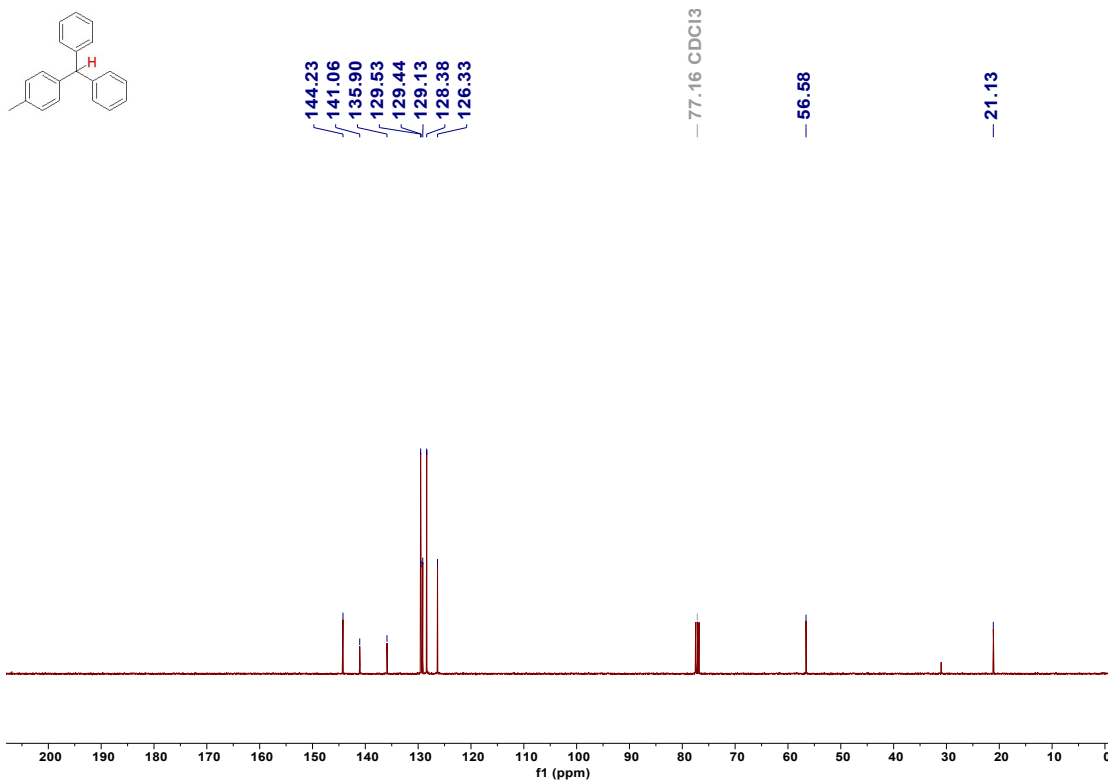
<sup>19</sup>F NMR spectra (376 MHz, CDCl<sub>3</sub>) of **2-14**



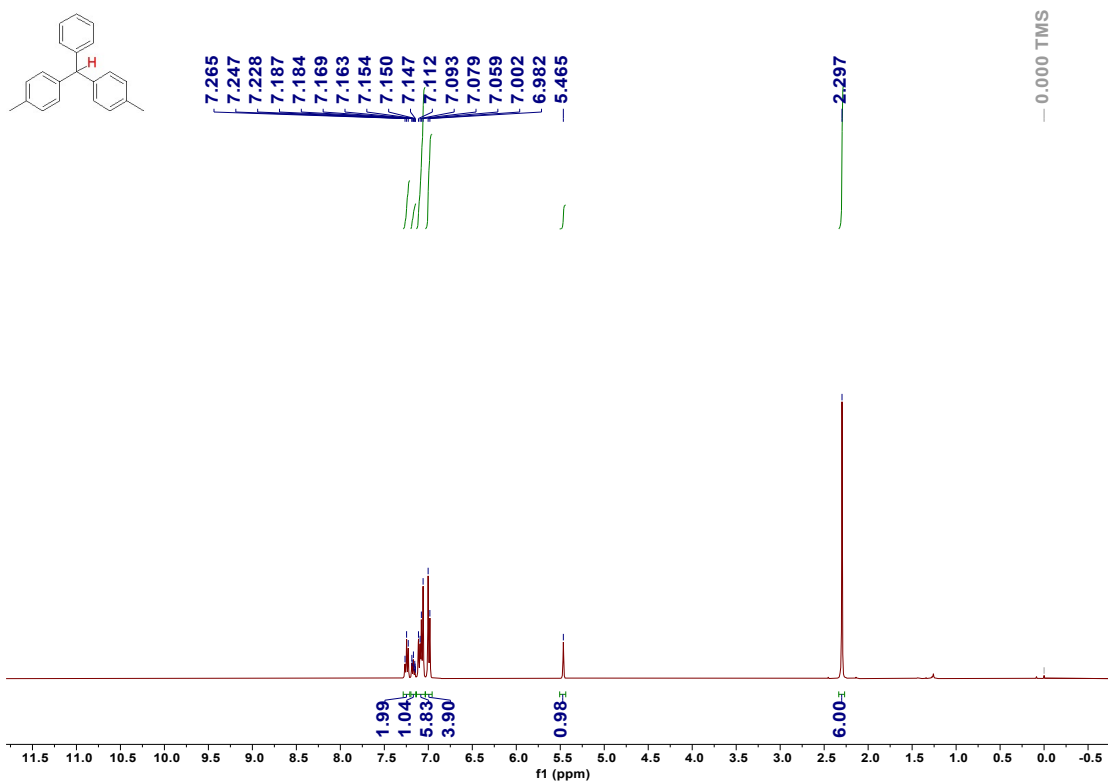
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-14**



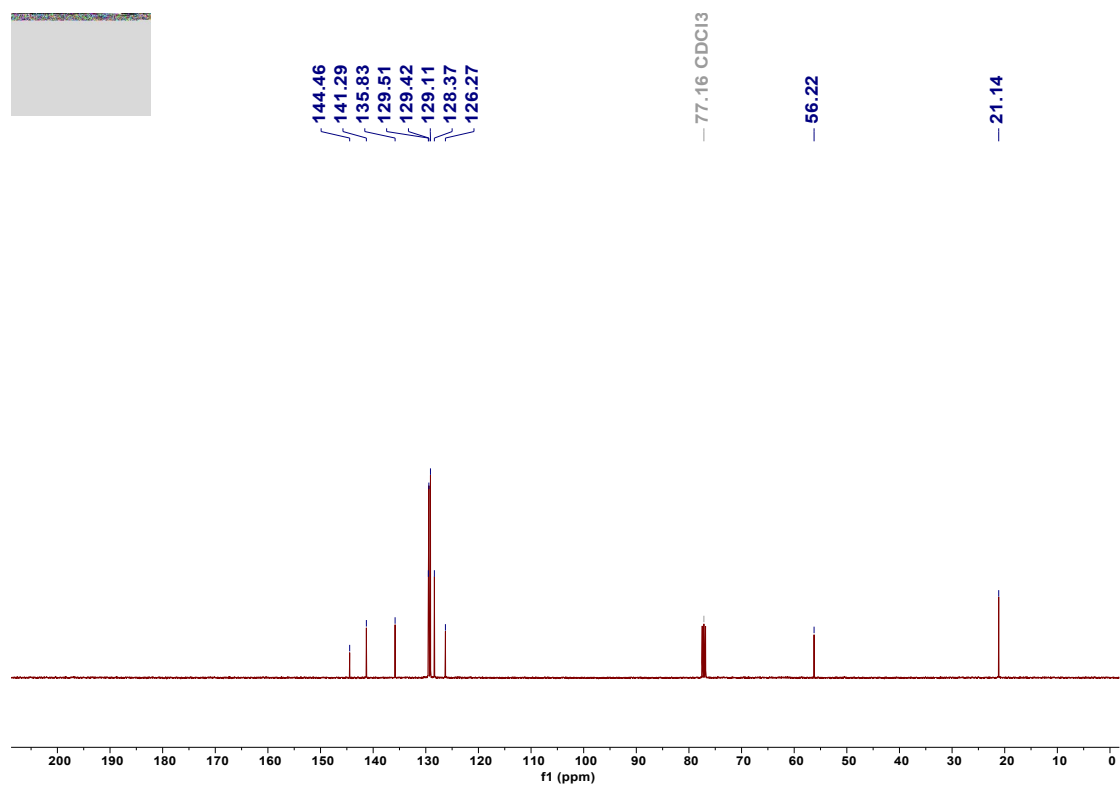
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-15**



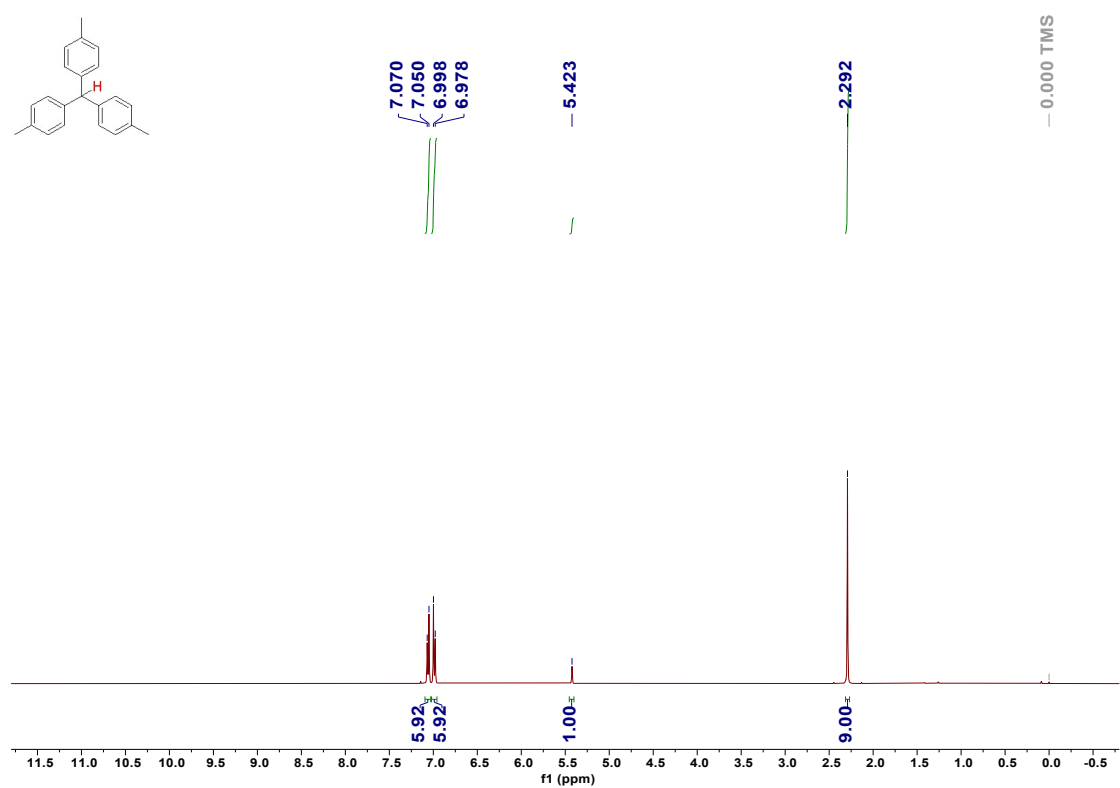
$^{13}\text{C}$  NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-15**



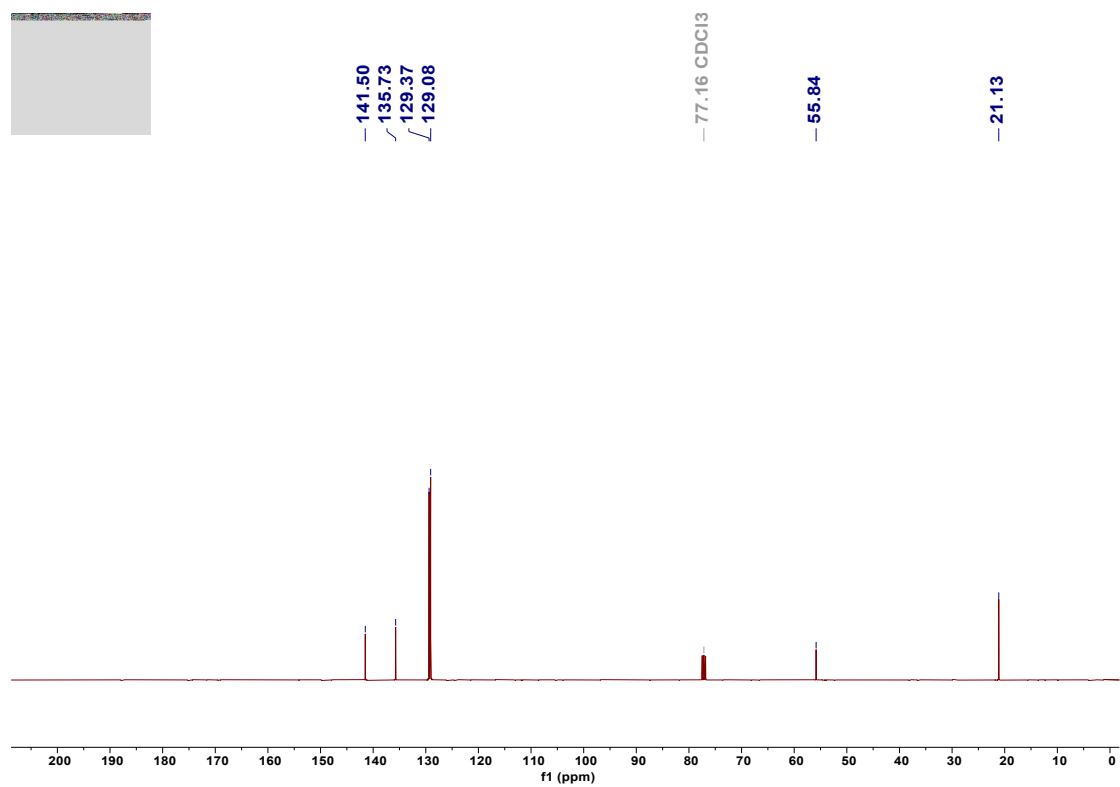
$^1\text{H}$  NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-16**



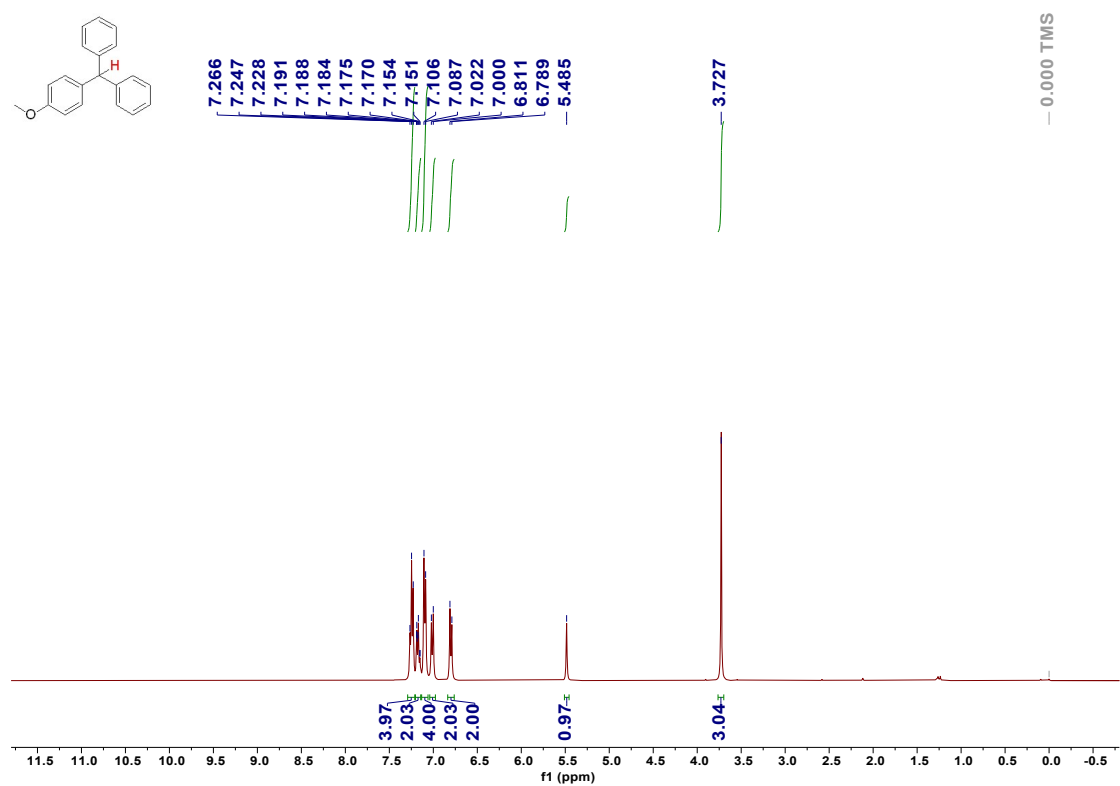
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-16**



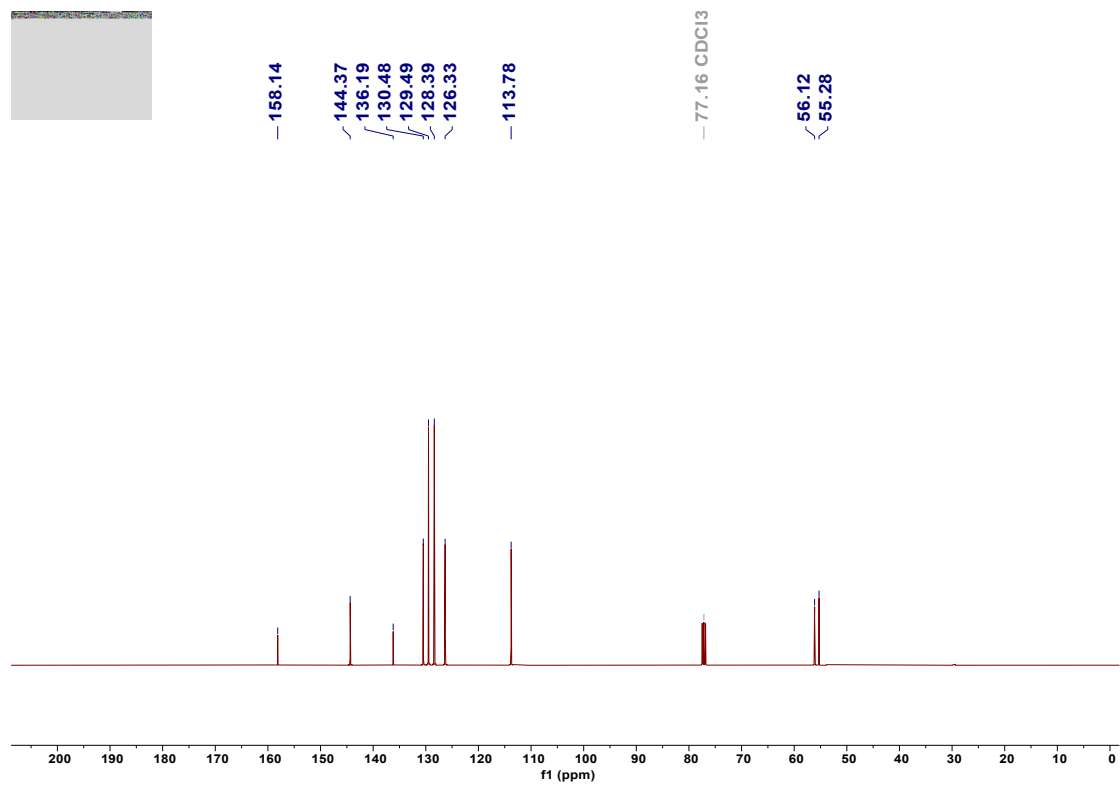
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-17**



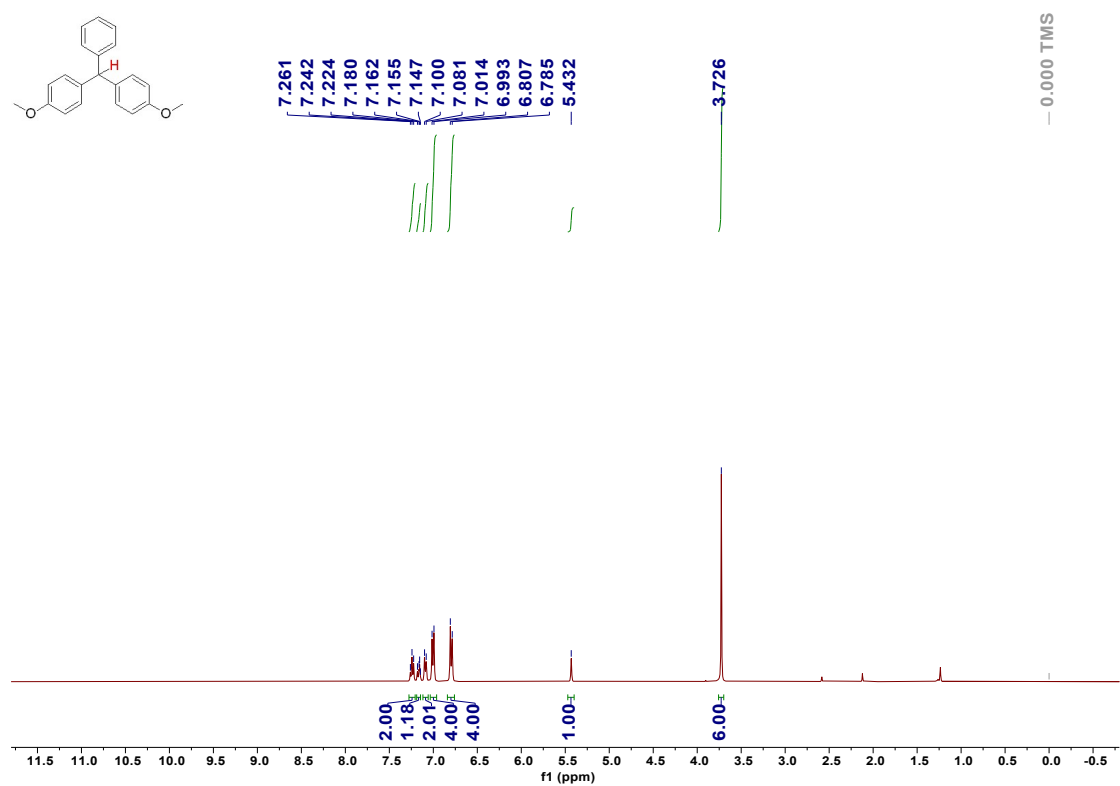
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-17**



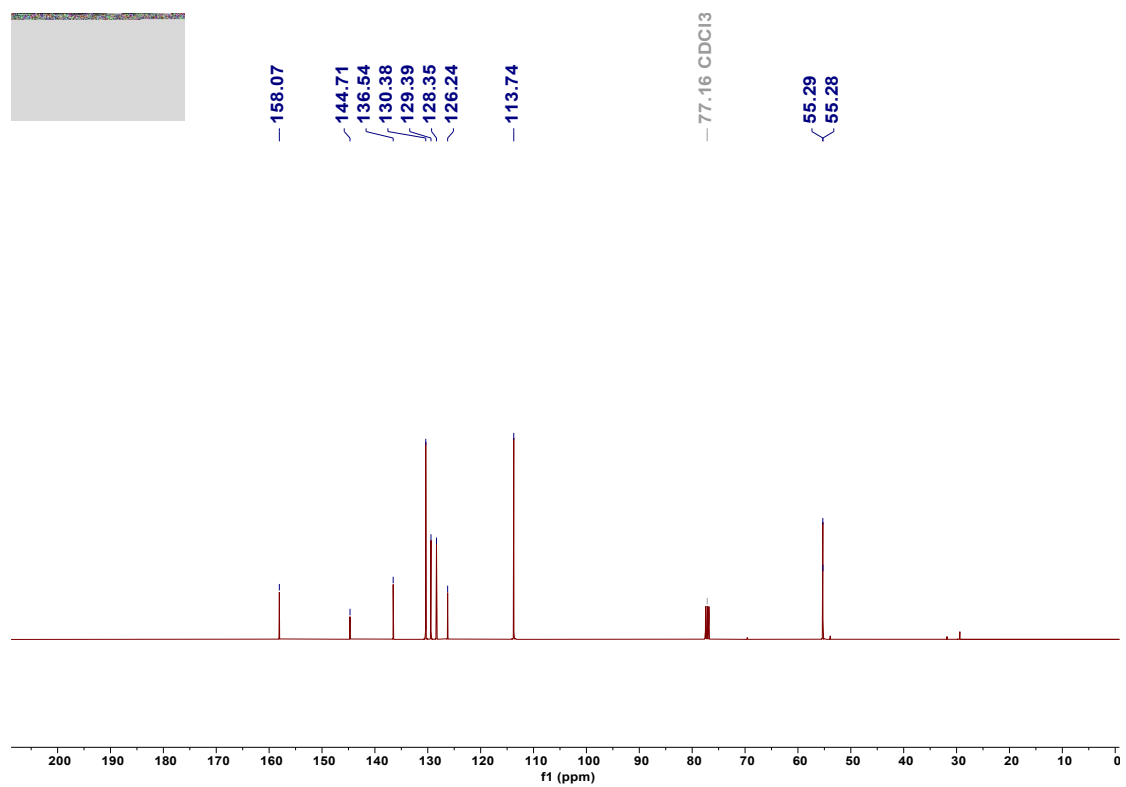
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-18**



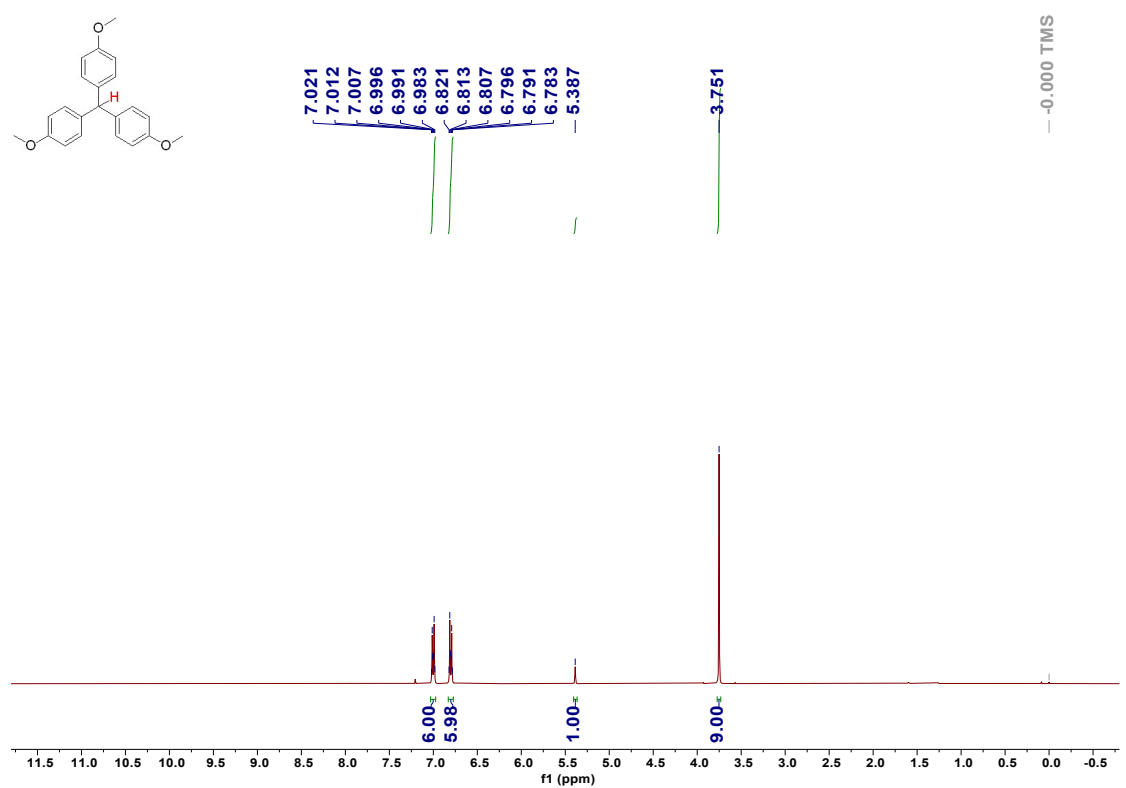
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-18**



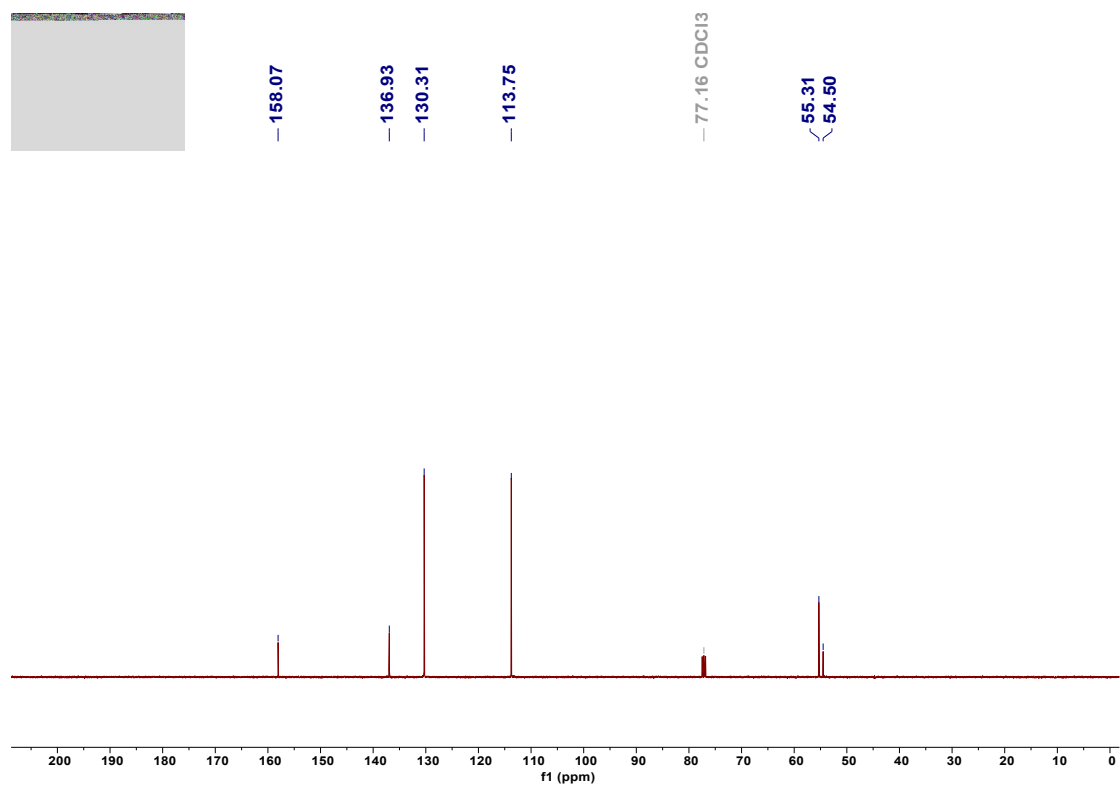
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-19**



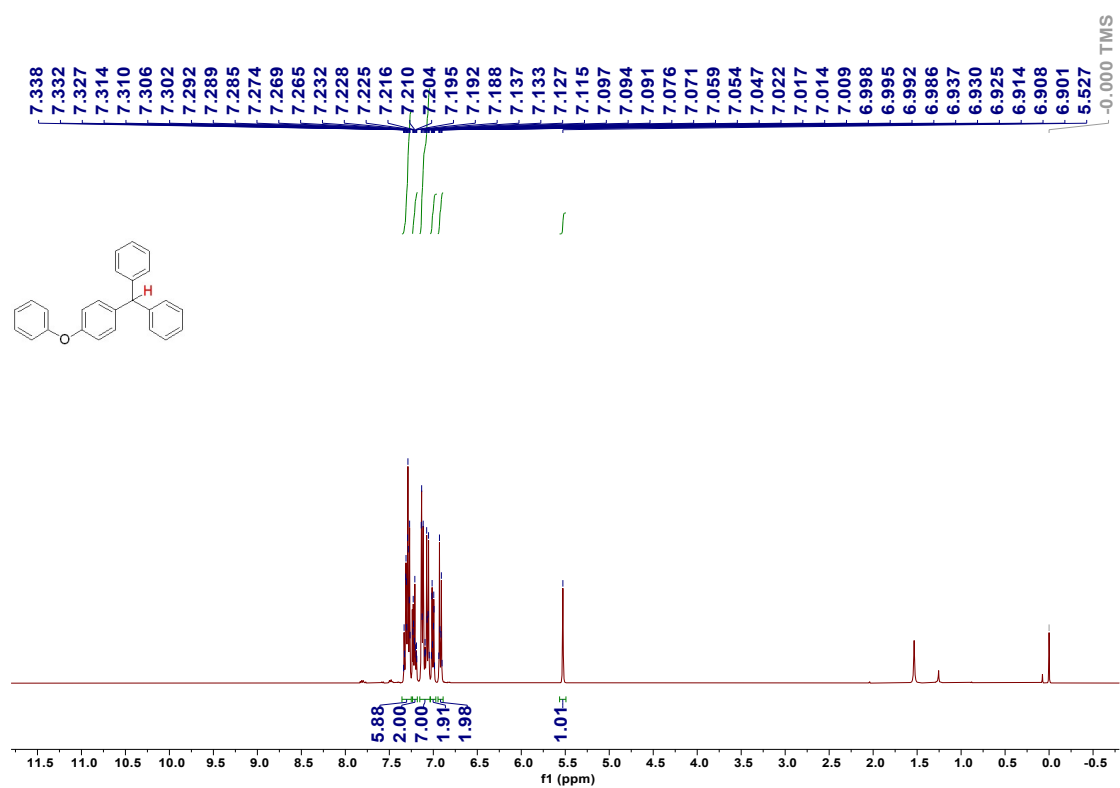
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-19**



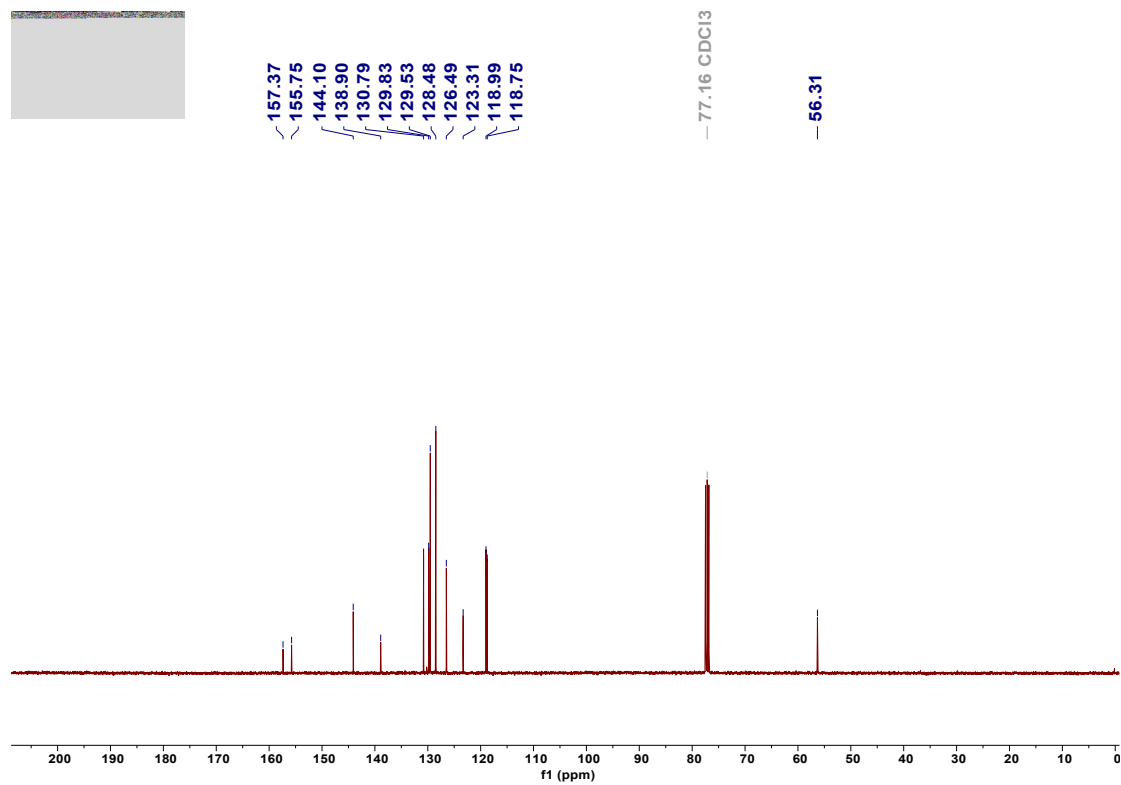
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-20**



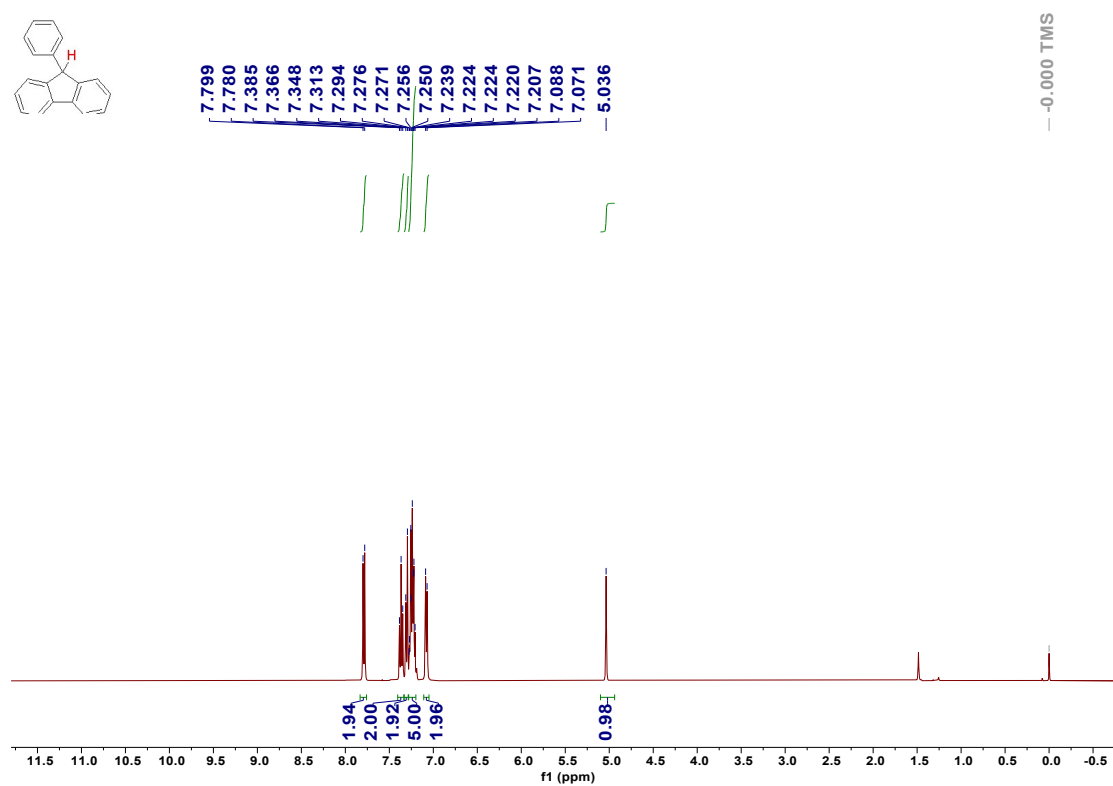
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-20**



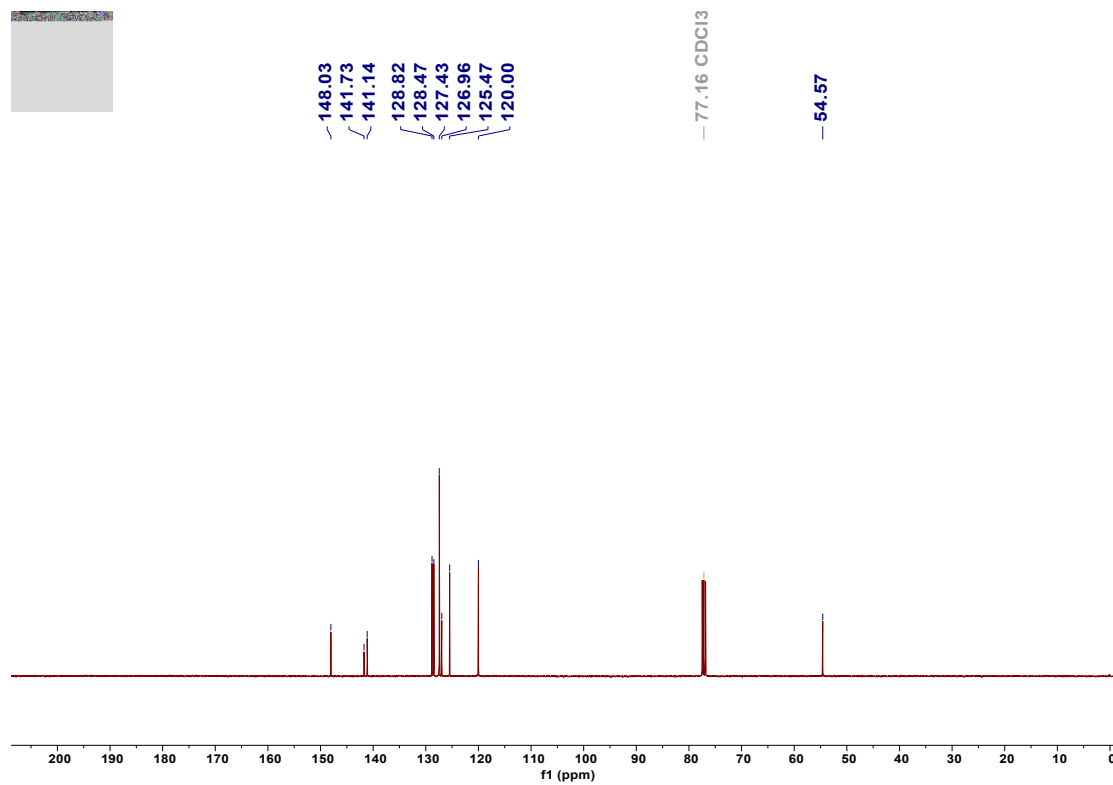
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-21**



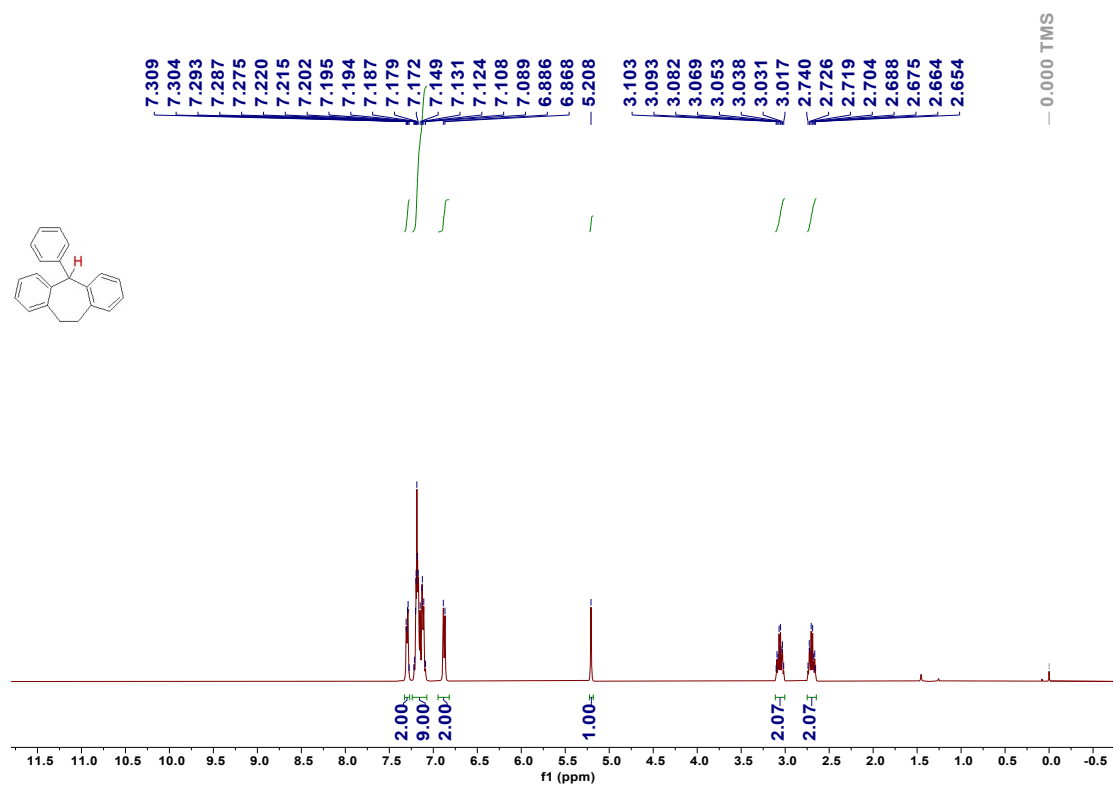
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-21**



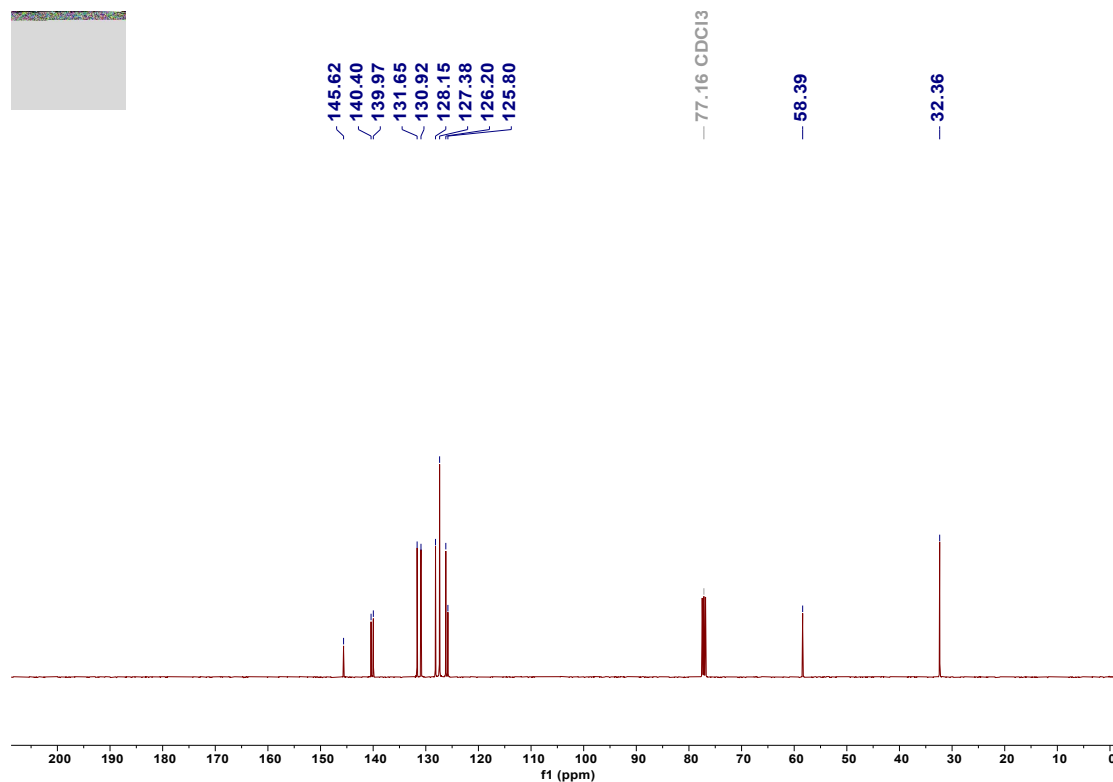
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-22**



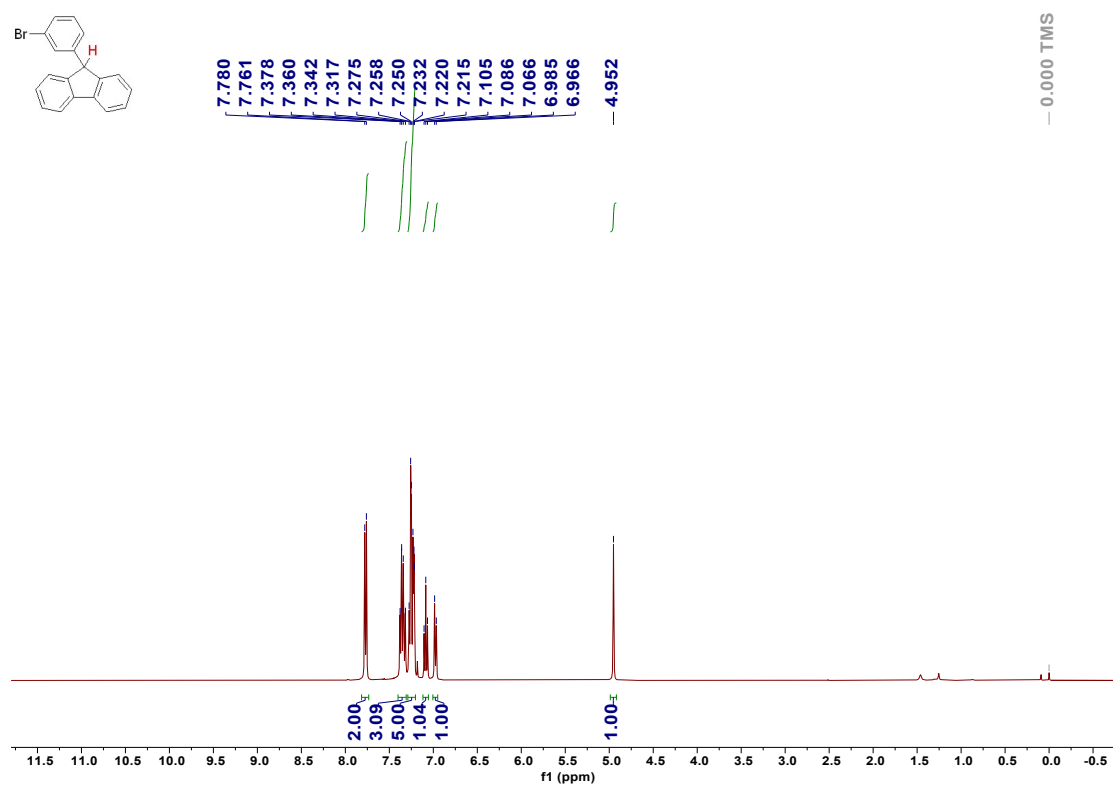
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-22**



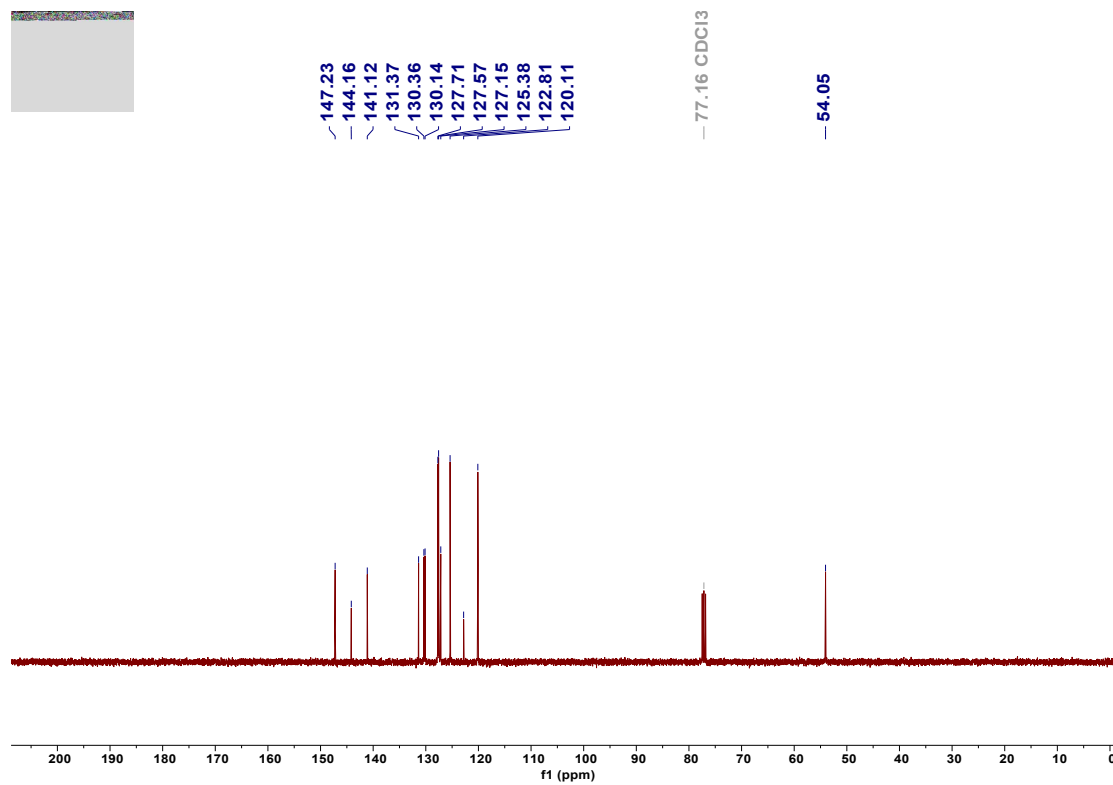
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-23**



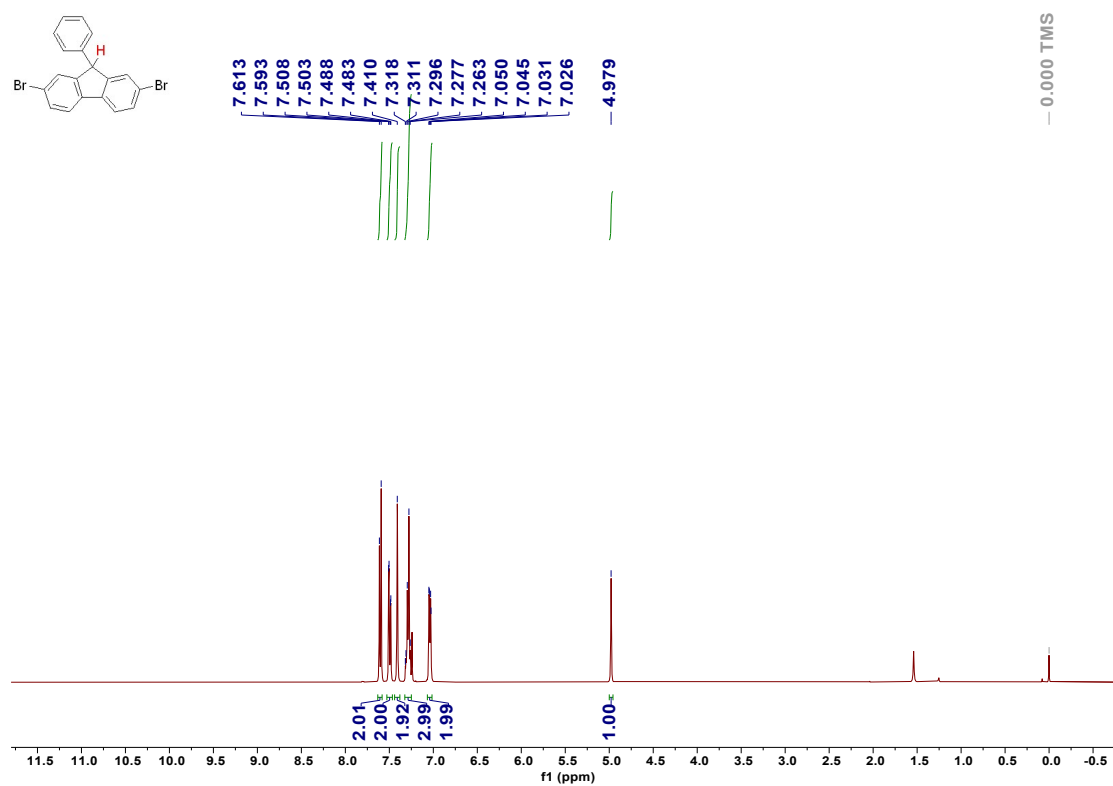
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-23**



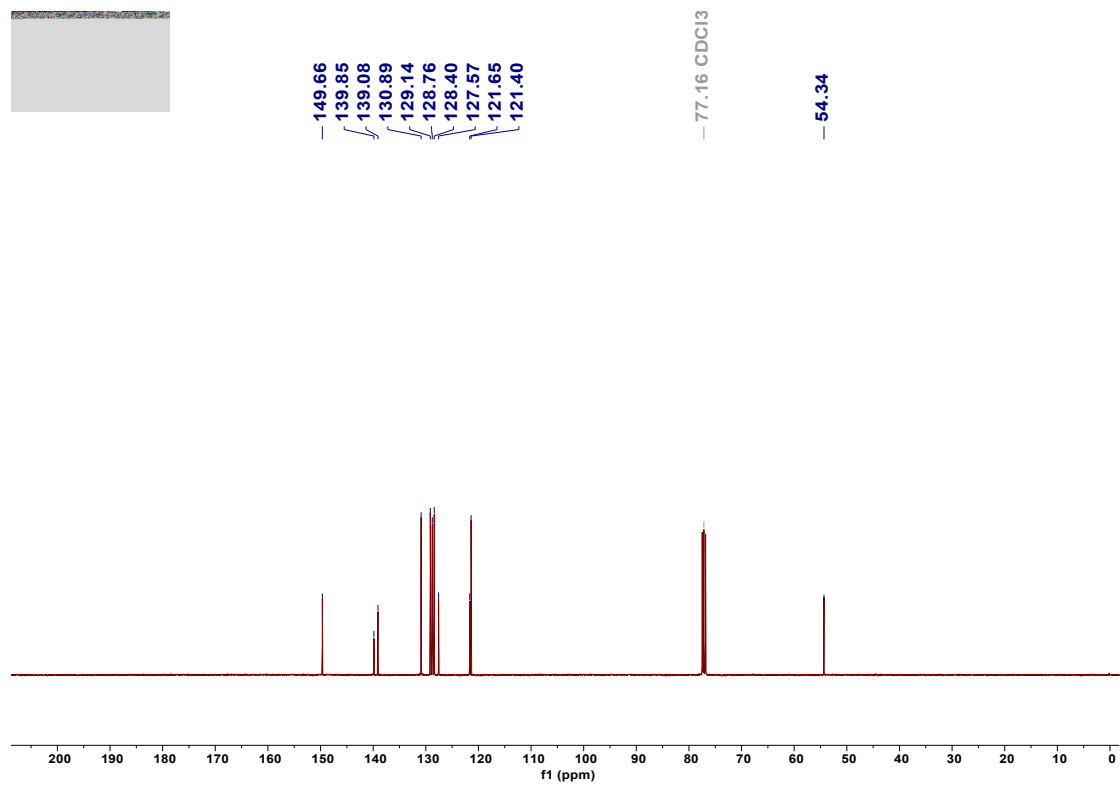
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-24**



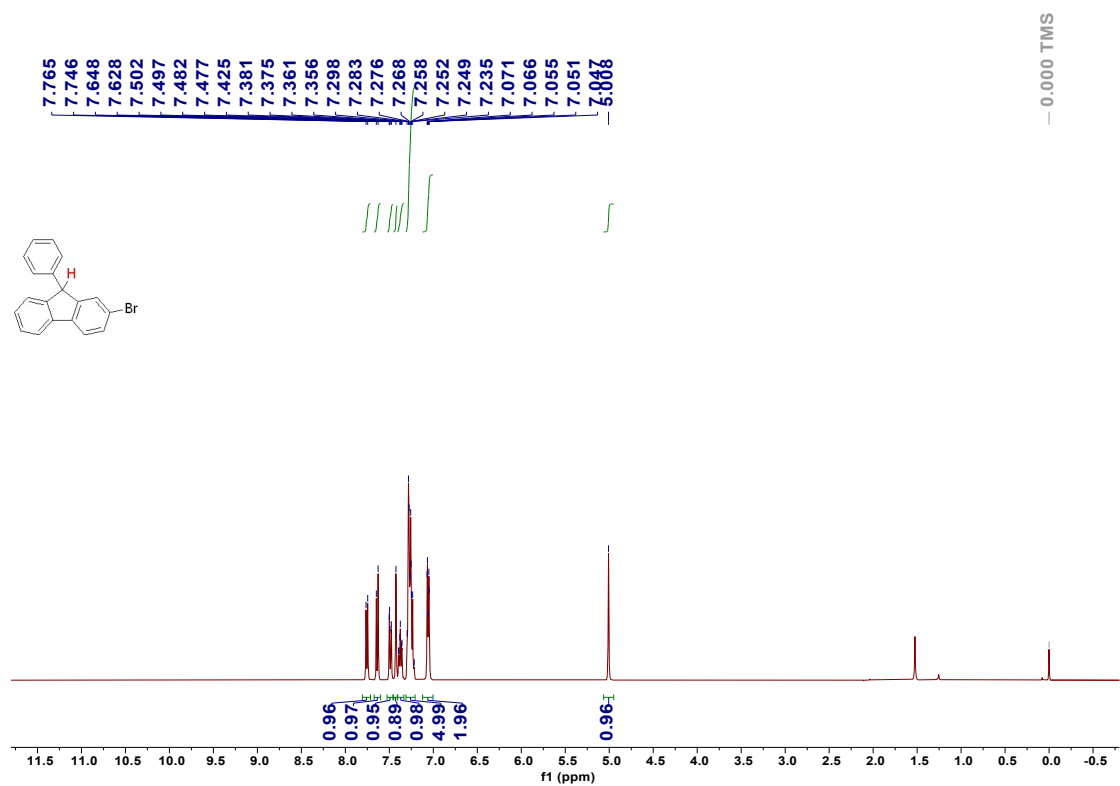
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-24**



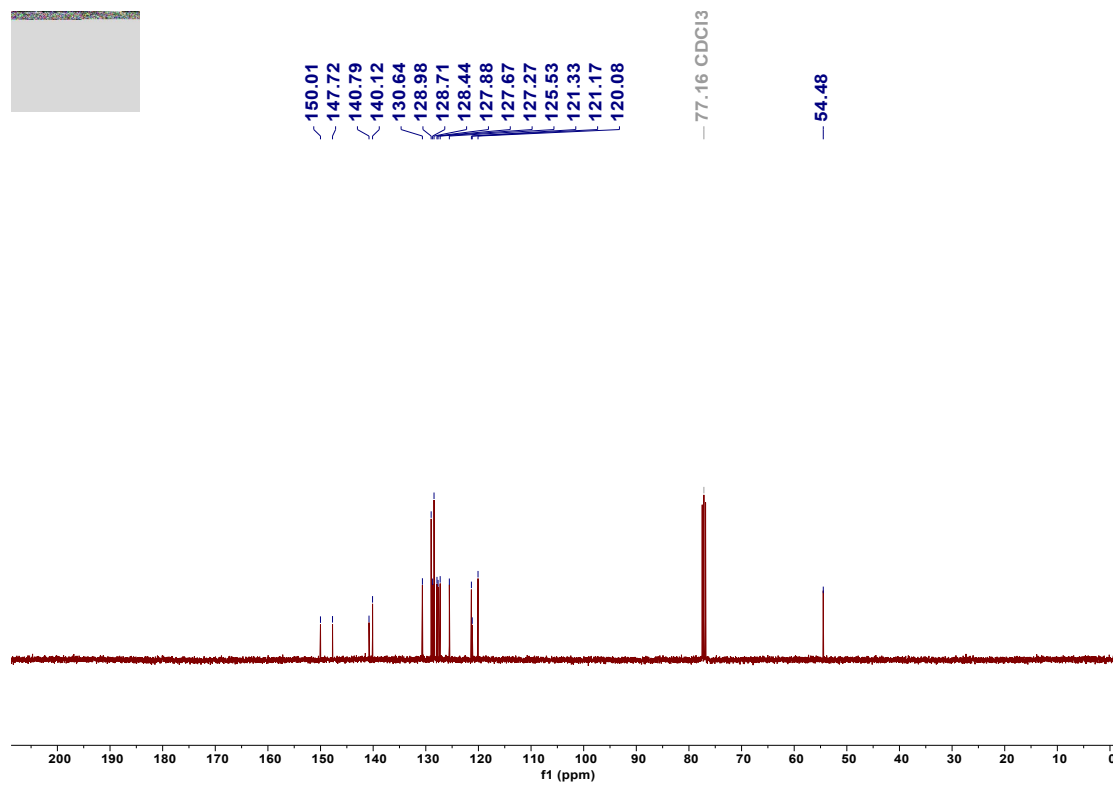
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-25**



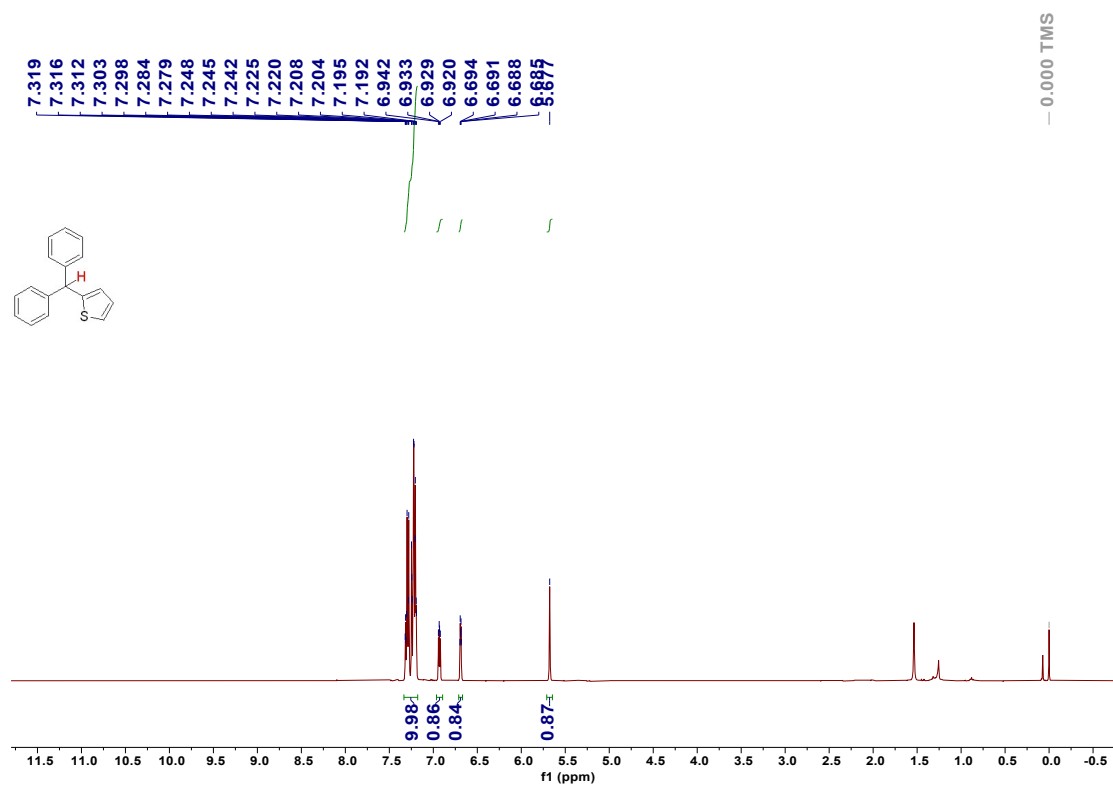
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-25**



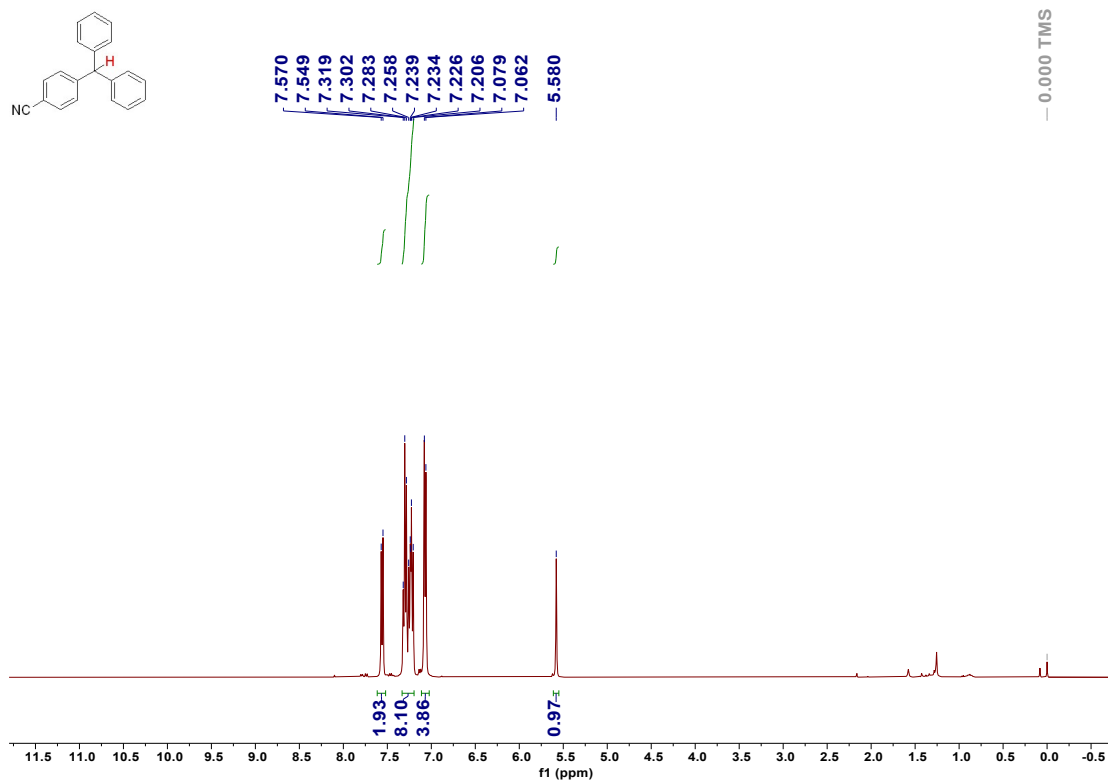
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-26**



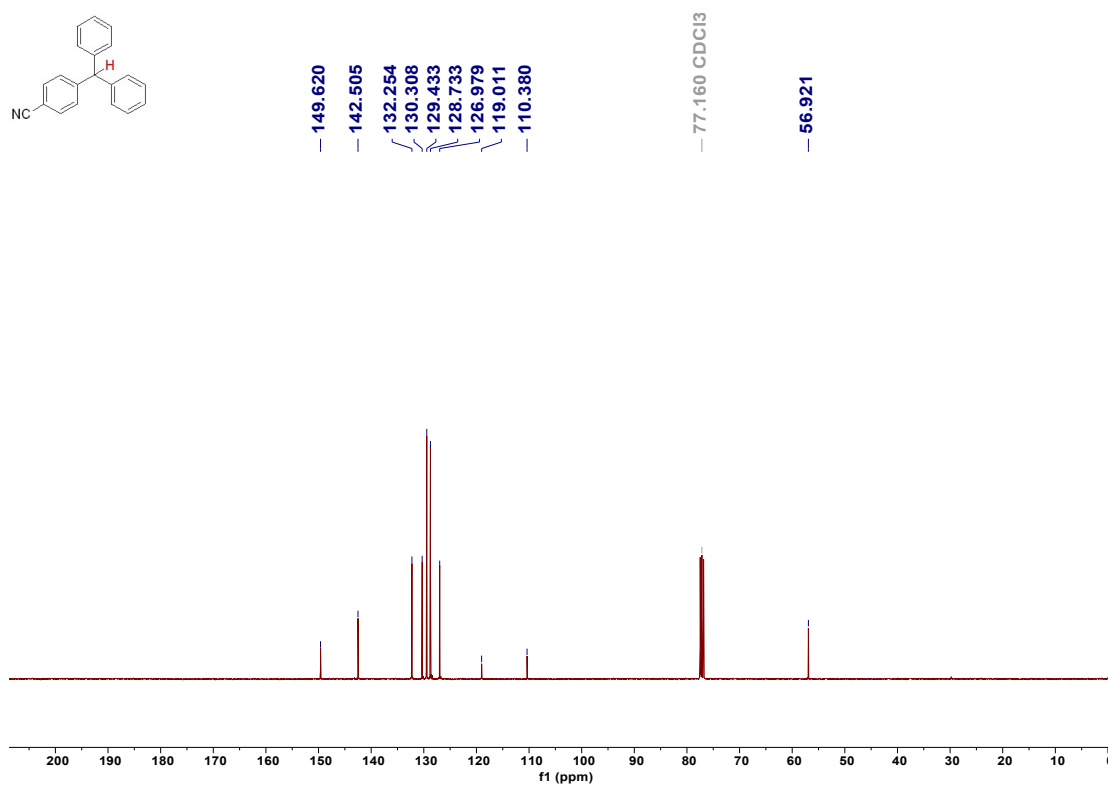
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-26**



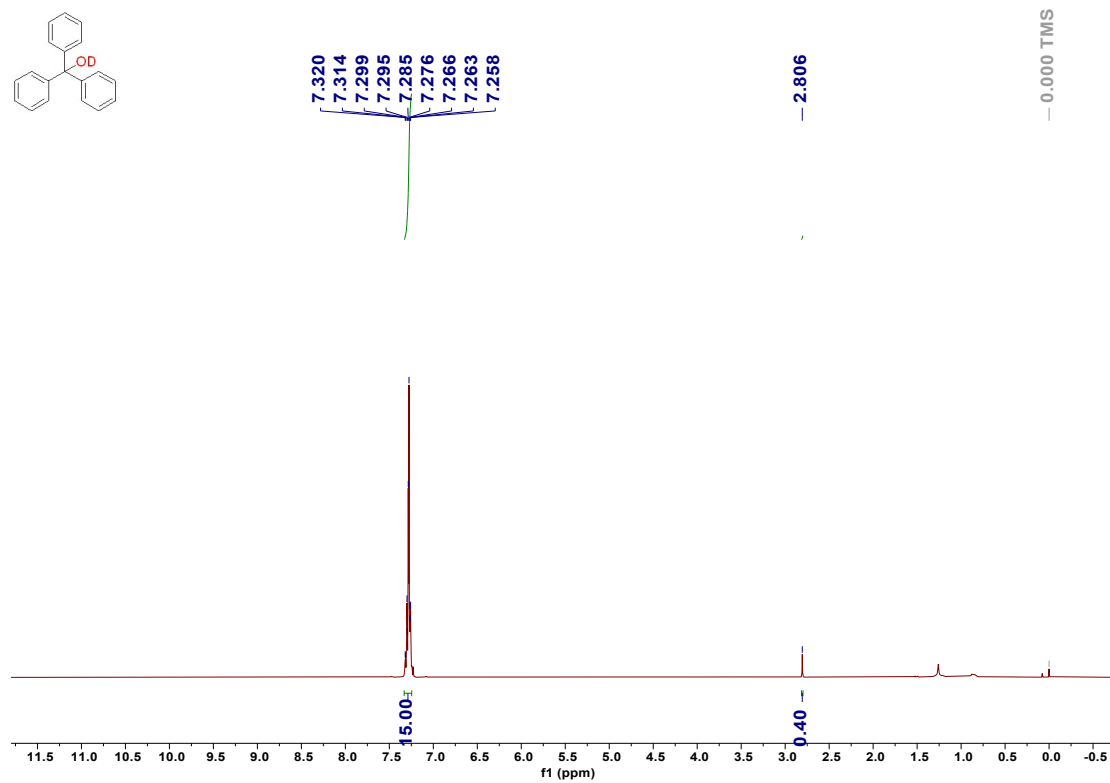
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-27**



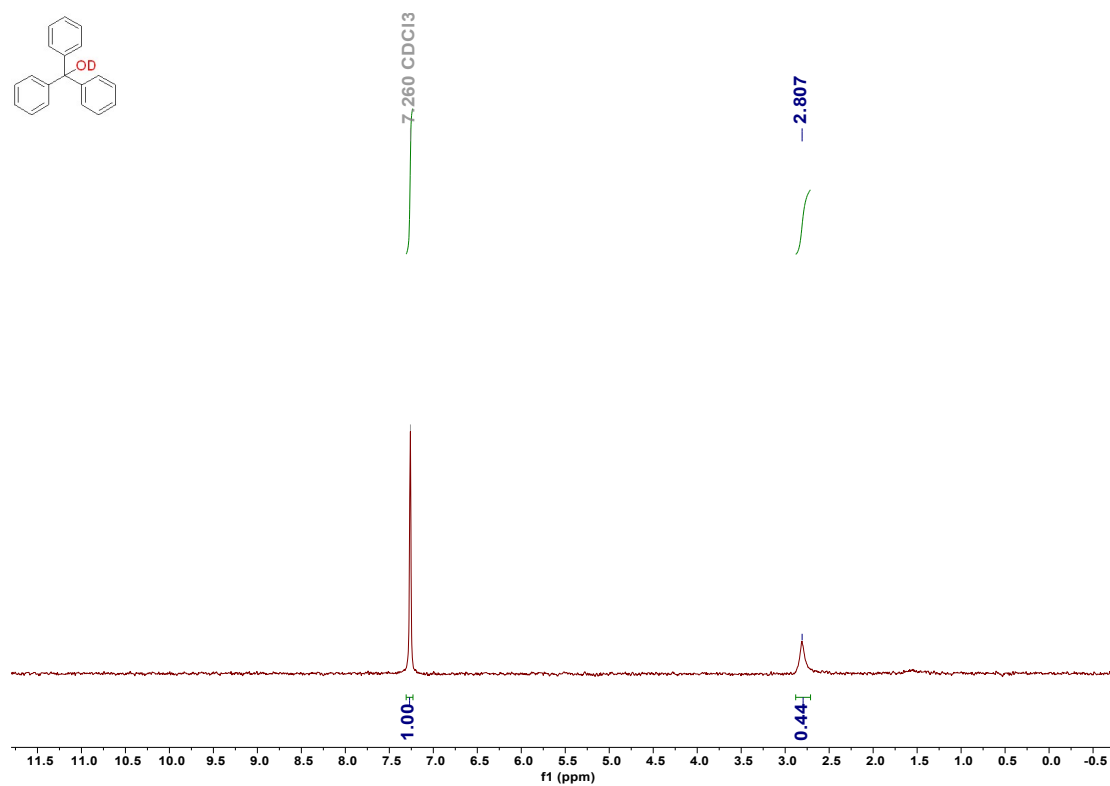
<sup>1</sup>H NMR spectra (400 MHz, CDCl<sub>3</sub>) of **2-28**



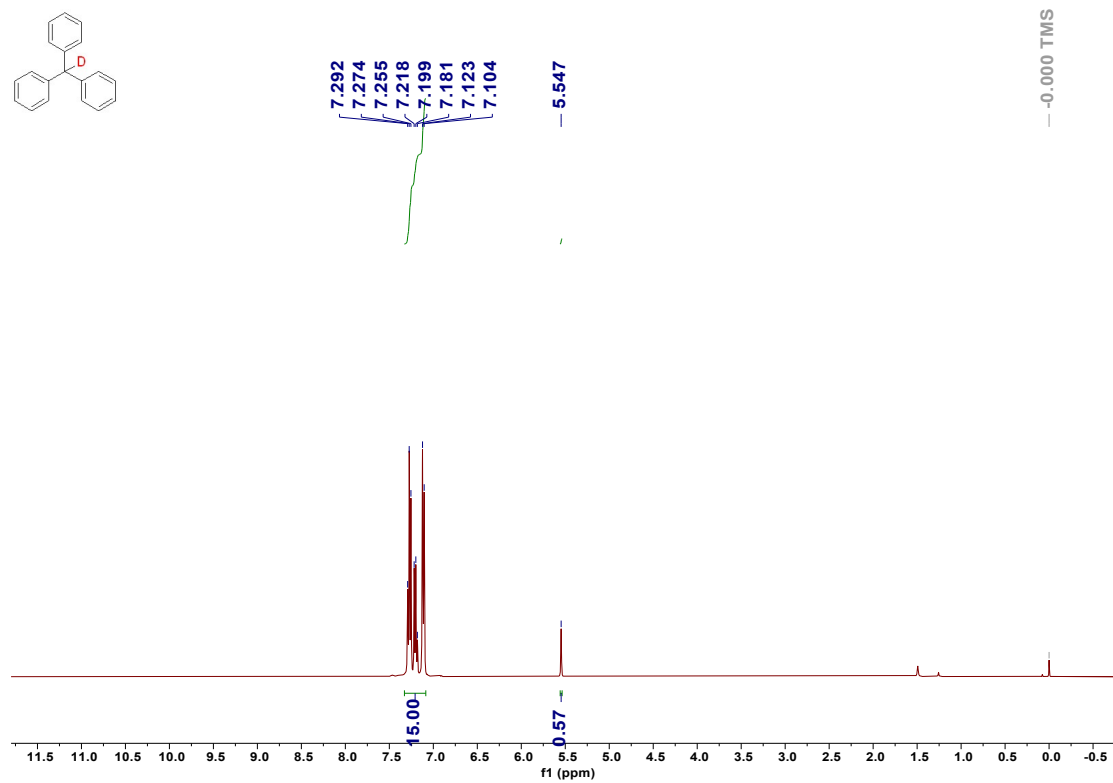
<sup>13</sup>C NMR spectra (101 MHz, CDCl<sub>3</sub>) of **2-28**



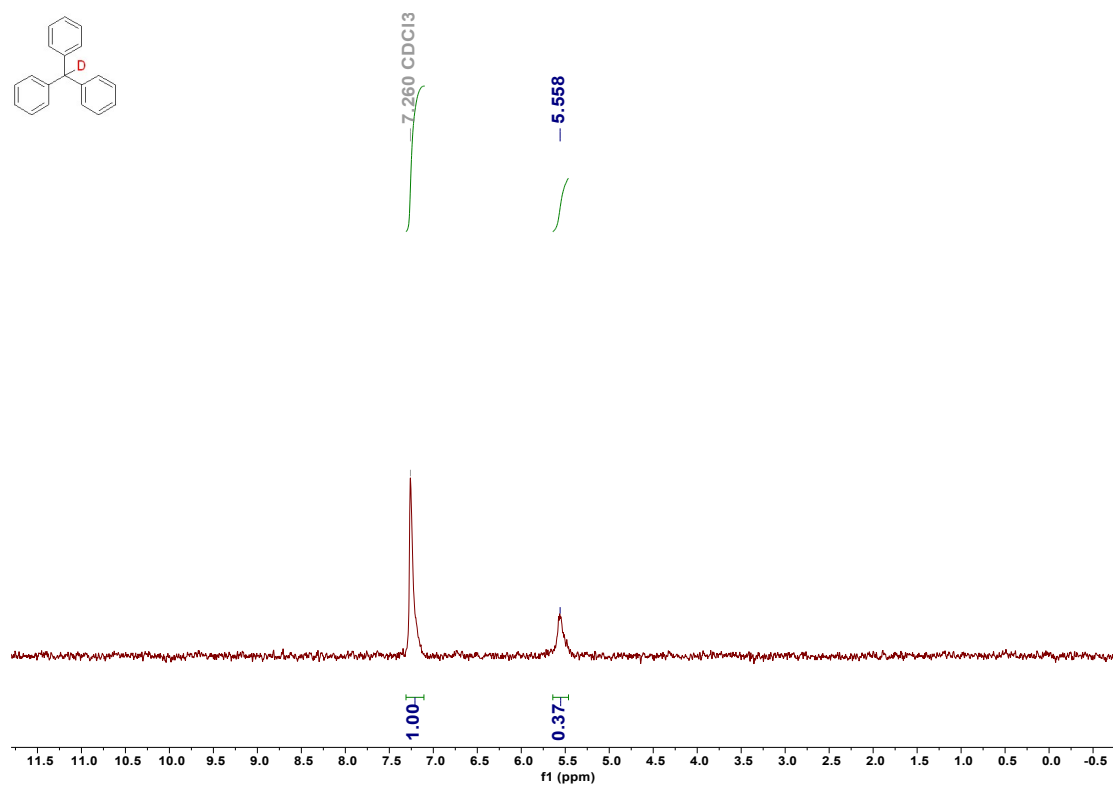
$^1\text{H}$  NMR spectra (400 MHz, Chloroform-*d*) of **D-1-1**



$^2\text{H}$  NMR spectra (61 MHz, Chloroform-*d*) of **D-1-1**



$^1\text{H}$  NMR spectra (400 MHz, Chloroform-*d*) of **D-2-1**



$^2\text{H}$  NMR spectra (61 MHz, Chloroform-*d*) of **D-2-1**

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