

## *Electronic Supplementary Information*

### **A Mild and Efficient Method for the Selective Deprotection of Silyl Ethers Using KF in the Presence of Tetraethylene Glycol**

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## 1. General

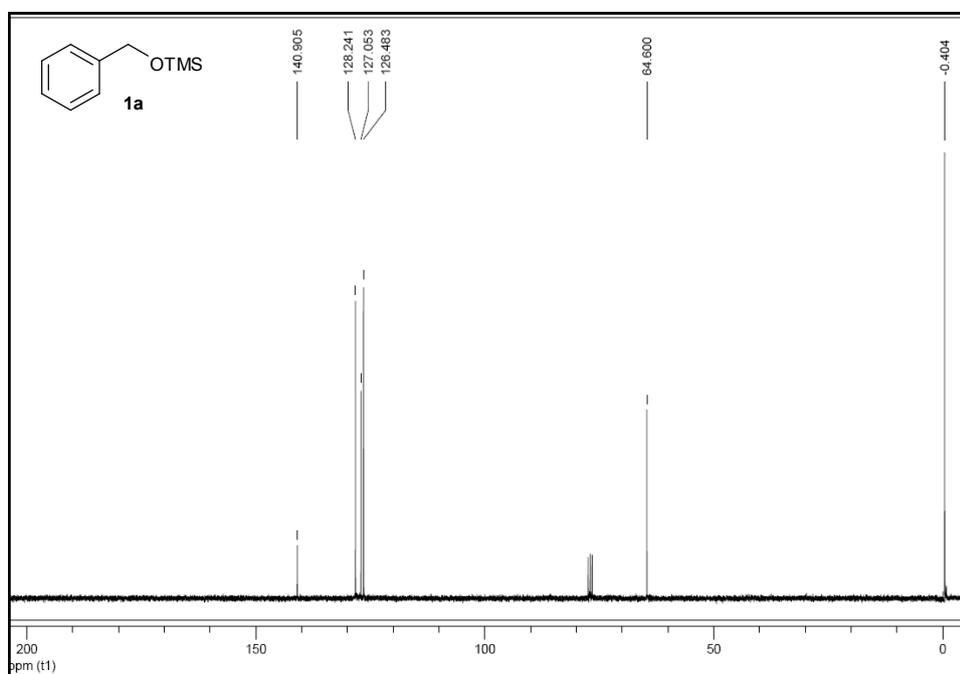
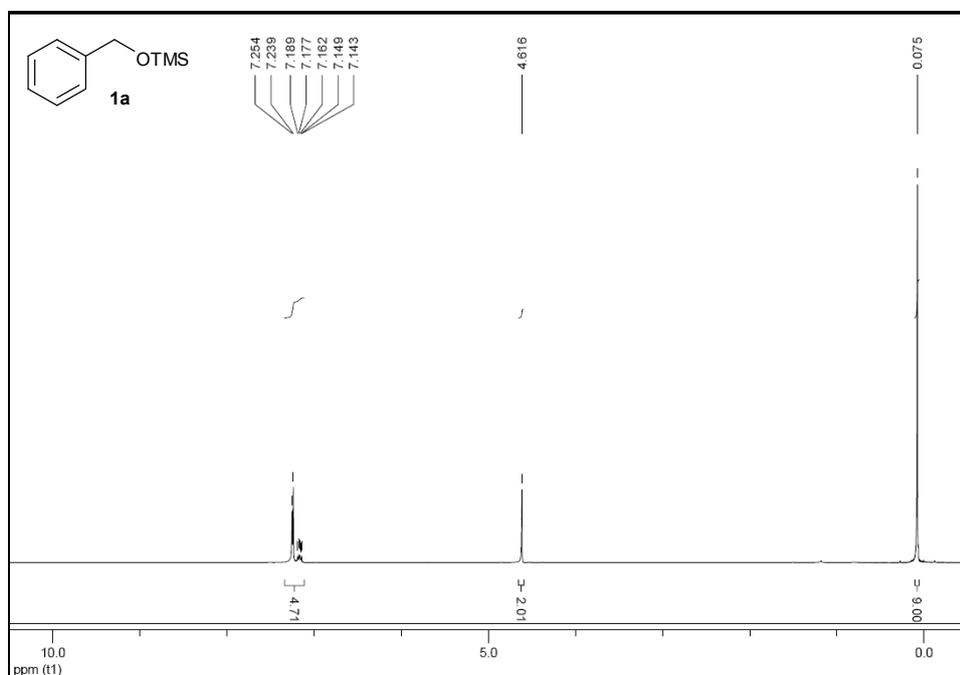
All reagents and solvents were purchased from *Aldrich* or TCI and used without further purification unless otherwise indicated. Thin-layer chromatography was carried out on Merck silica gel 60F plates. The chromatographic purification of the products was carried out by using Merck silica gel 60 (230–400 mesh). The  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded on Varian 300 or Varian 500 spectrometers using TMS as an internal standard. The HPLC analyses were performed on a Varian Pro Star Series instrument equipped with an isostatic pump using a CHIRALCEL Column (250 × 4.6 mm).

## 2. General procedure for the deprotection of silyl ethers

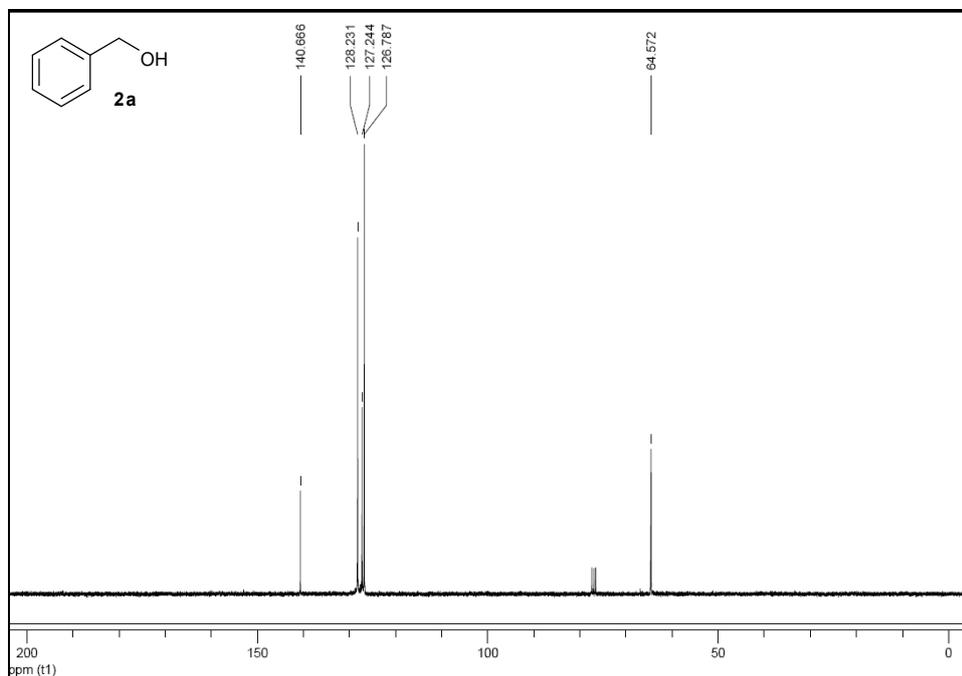
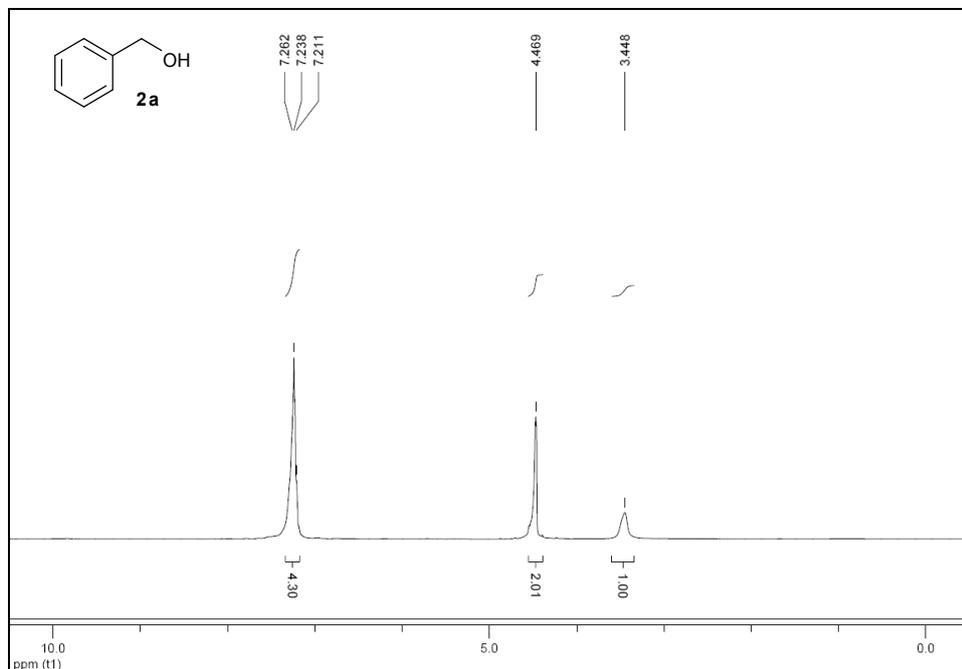
Spray dried potassium fluoride (8.7 mg, 0.15 mmol) and dried tetraethylene glycol (1.0 mL) were added to a vial and stirred with a magnetic stirring bar. The silyl protected substrate (0.1 mmol) was then added and the reaction mixture stirred at room temperature or 80 °C. The reaction was followed by TLC (EtOAc : hexanes = 1: 4) until the starting materials were no longer detected. After the reaction was completely finished, the reaction mixture was quenched with water (10 mL) and extracted with diethyl ether (10 mL X 3). The combined organic layer was dried over anhydrous  $\text{MgSO}_4$ , filtered and purified by short silica column chromatography (EtOAc : hexane = 1: 4) to afford the desilylated alcohol product as a colorless oil or solid.

### 3. NMR Data for Table 1

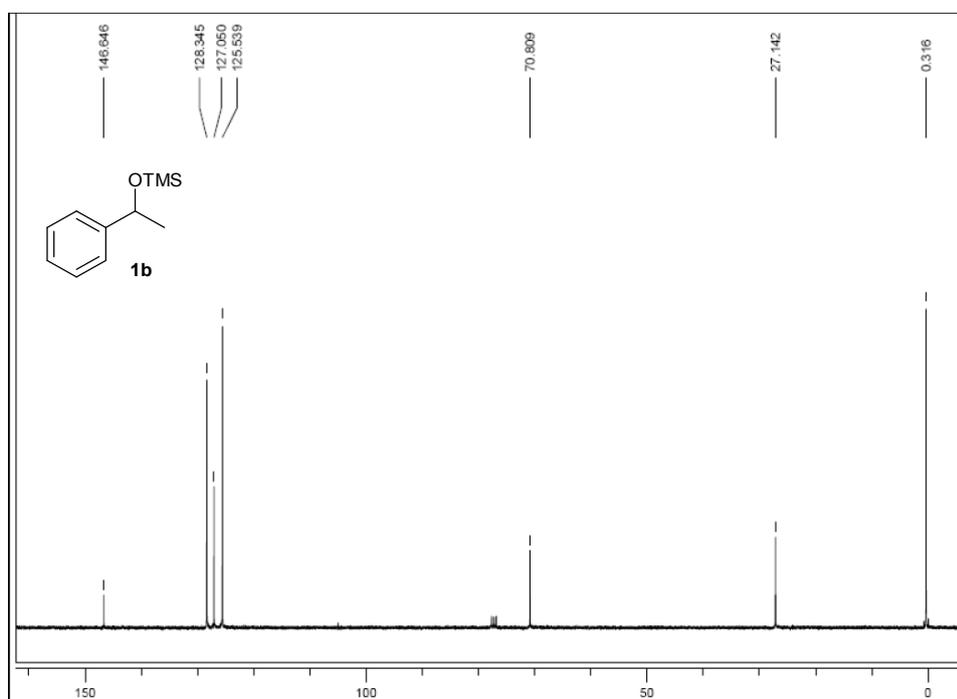
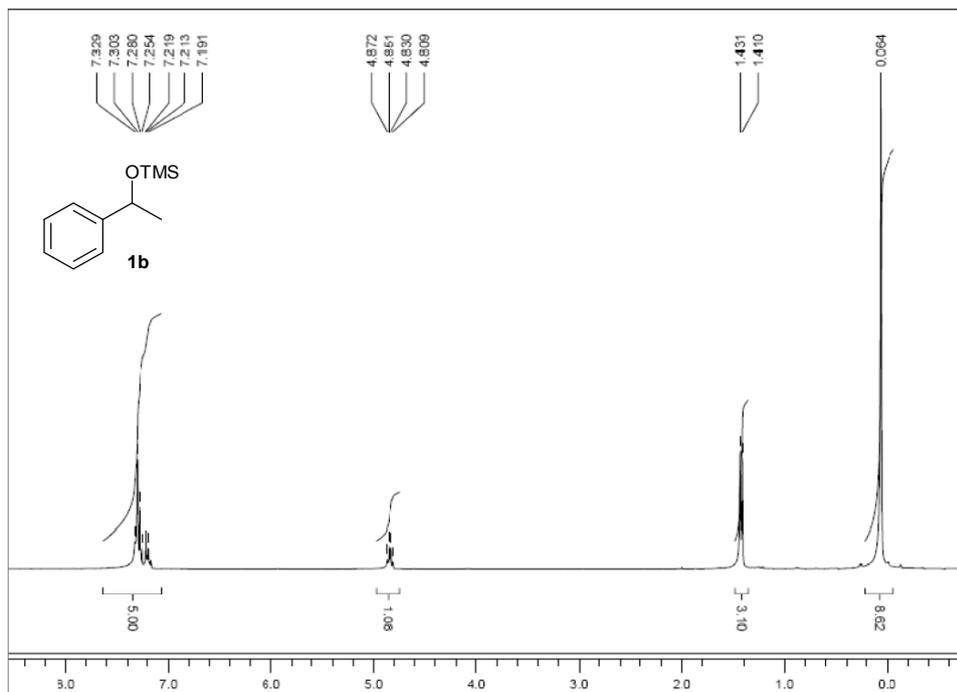
**Entry 1 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.08 (s, 9H), 4.62 (s, 2H), 7.14-7.25 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  -0.40, 64.60, 126.48, 127.05, 128.24, 140.91



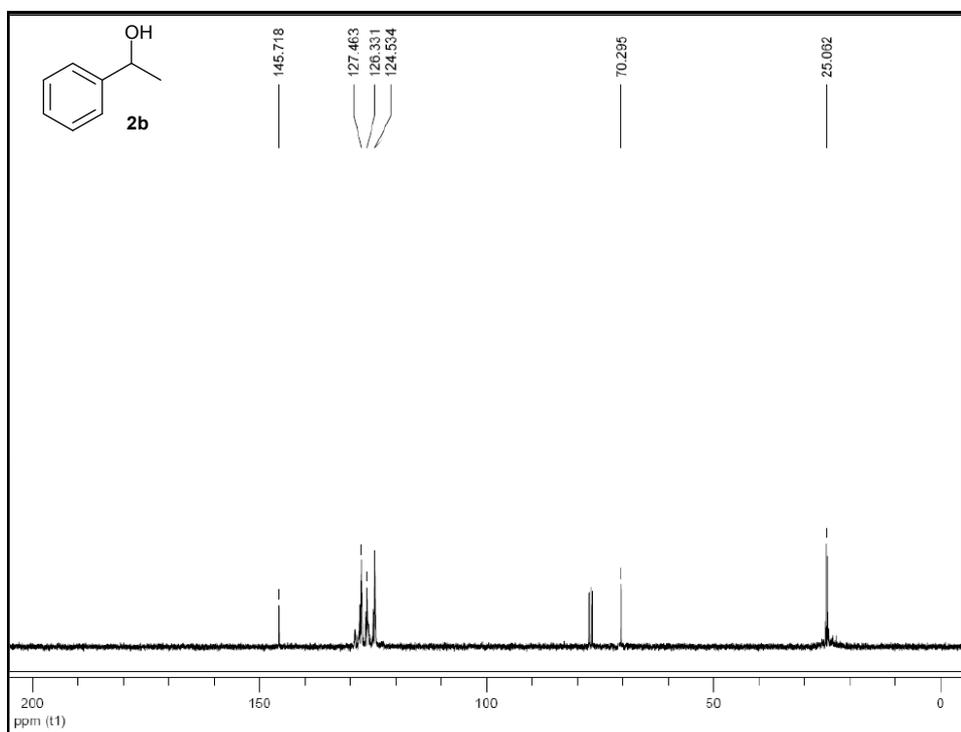
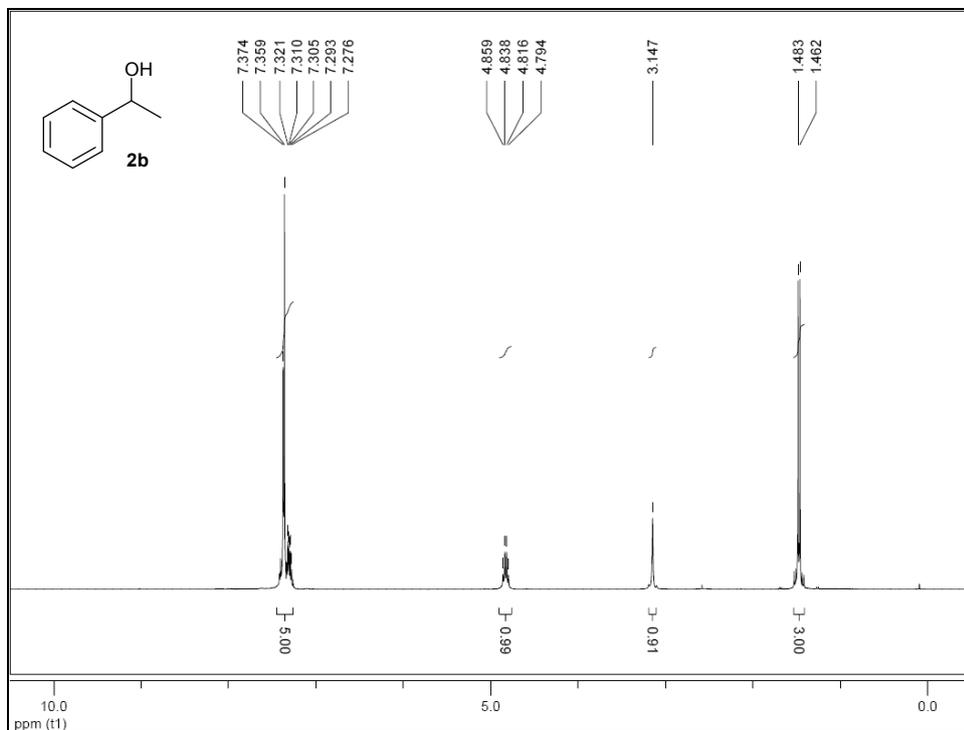
**Entry 1 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.45 (s, 1H), 4.48 (s, 2H), 7.21-7.26 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  64.57, 126.79, 127.24, 128.23, 140.67



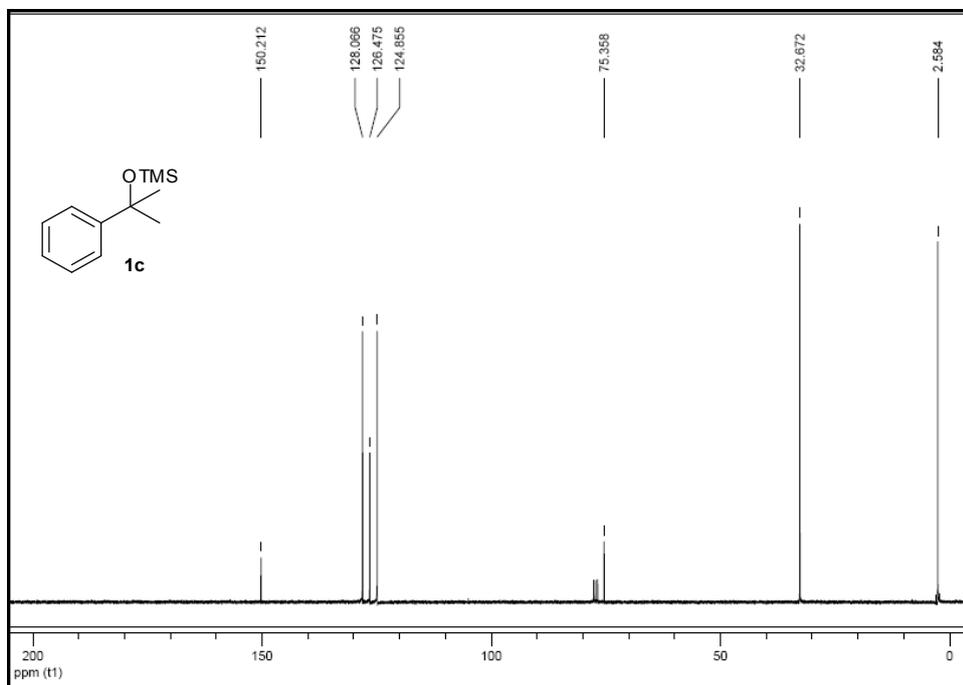
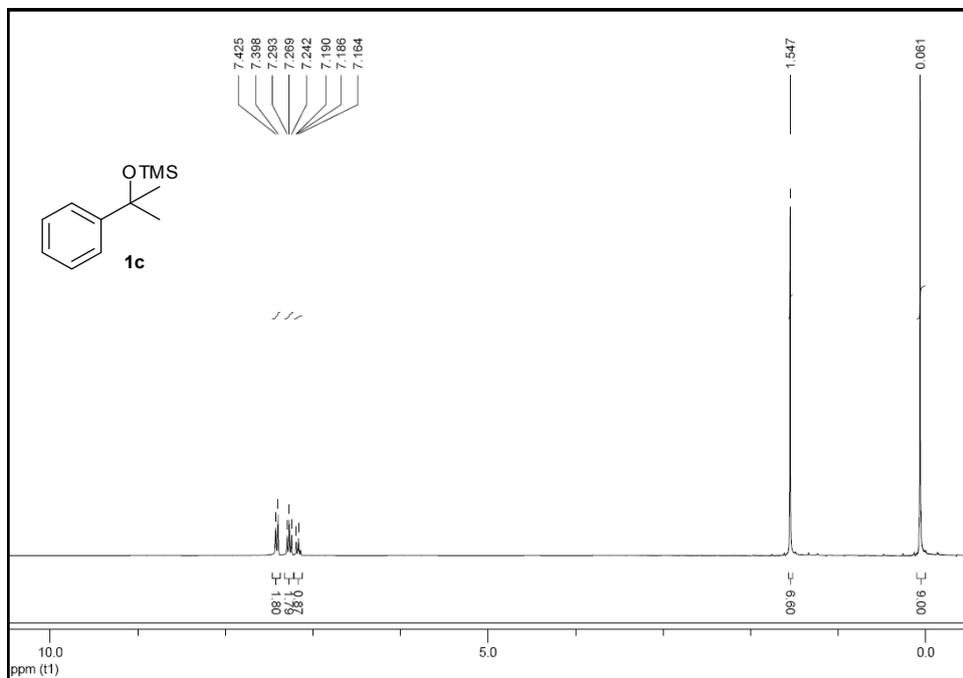
**Entry 2 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.06 (s, 9 H), 1.42 (d,  $J = 6.3$  Hz, 3H), 4.84 (q,  $J = 6.3$  Hz, 1H), 7.19-7.33 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  0.32, 27.14, 70.80, 125.53, 127.05, 128.34, 146.46.



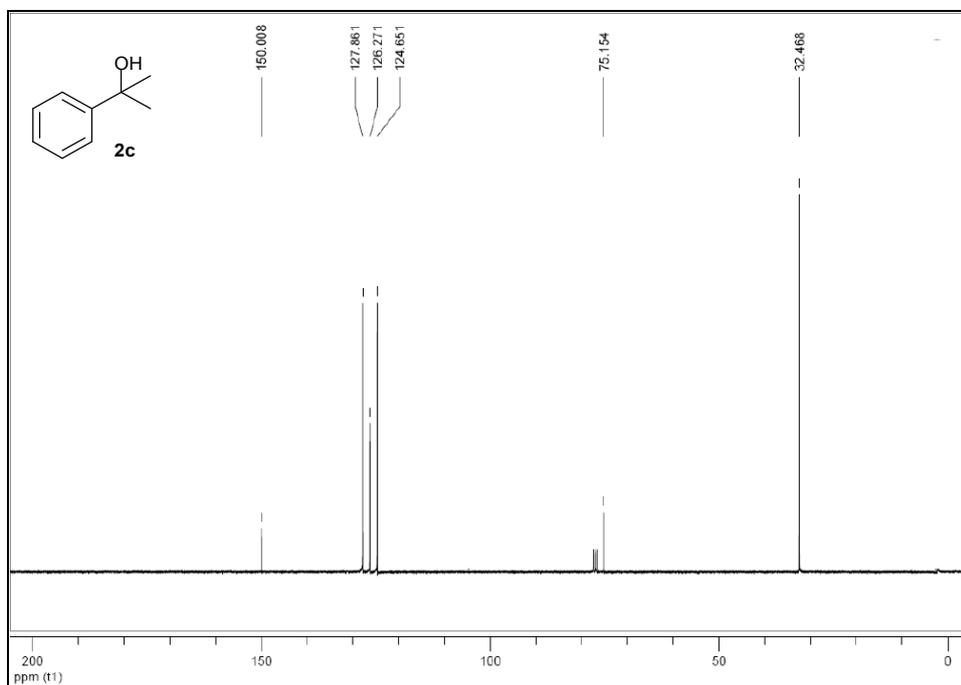
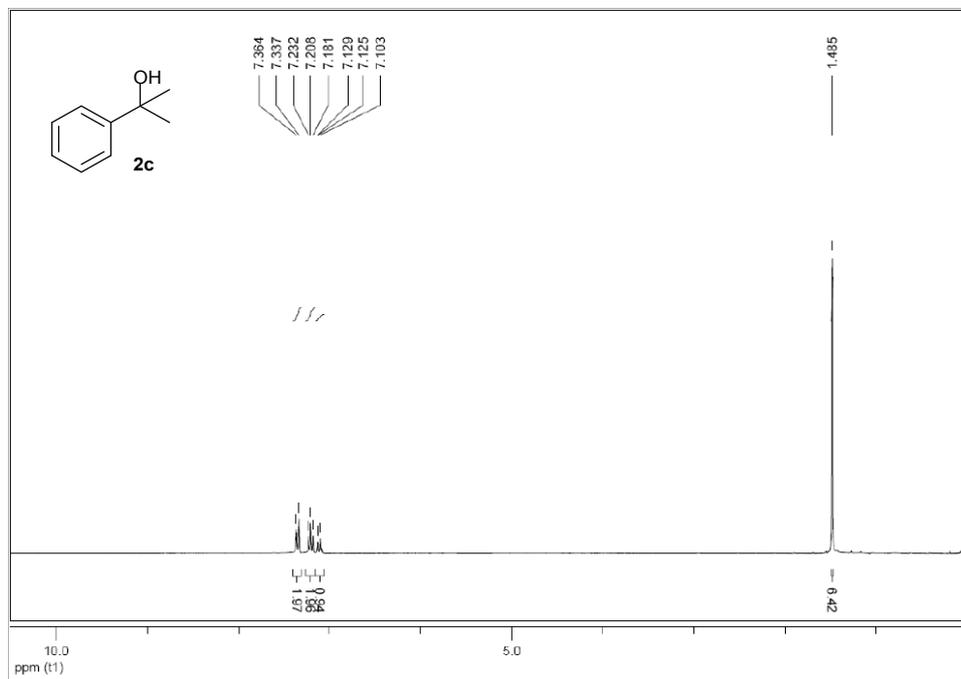
**Entry 2 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.47 (d,  $J = 6.3$  Hz, 3H), 3.15 (s, 1H), 4.83 (q,  $J = 6.5$  Hz, 1H), 7.28-7.37 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  25.06, 70.30, 124.51, 126.53, 127.46, 145.72



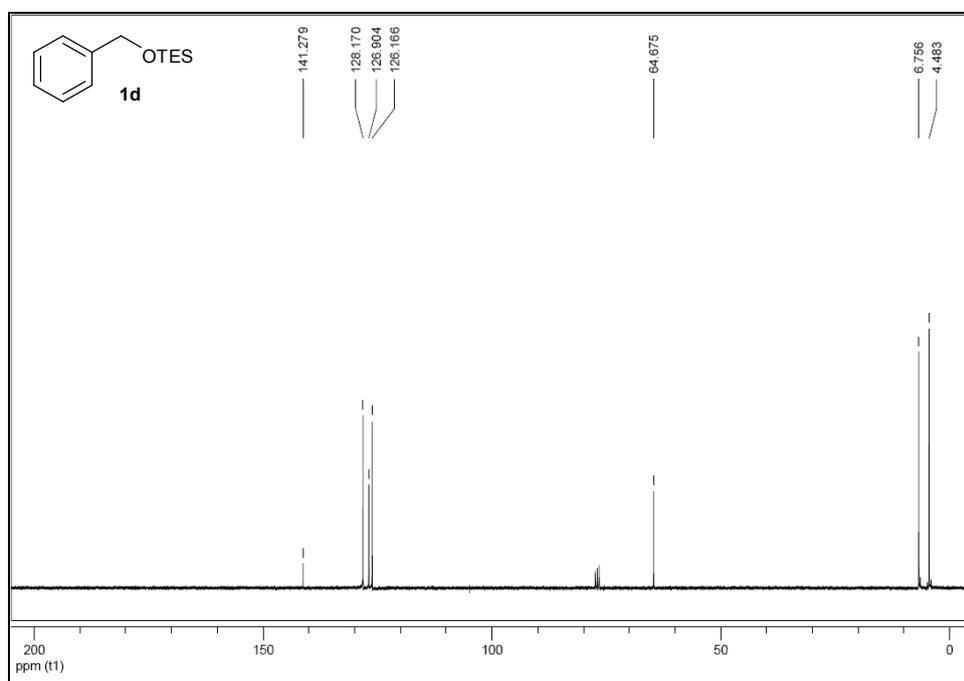
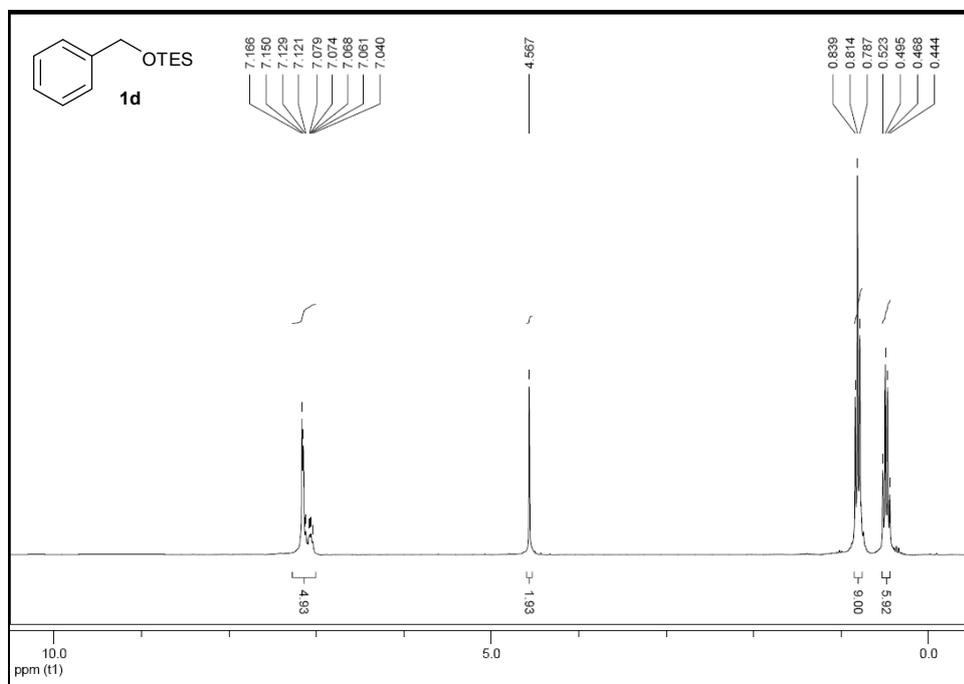
**Entry 3 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.06 (s, 9H), 1.55 (s, 6H), 7.16-7.43 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  2.58, 32.67, 75.36, 124.86, 126.48, 128.07, 150.21.



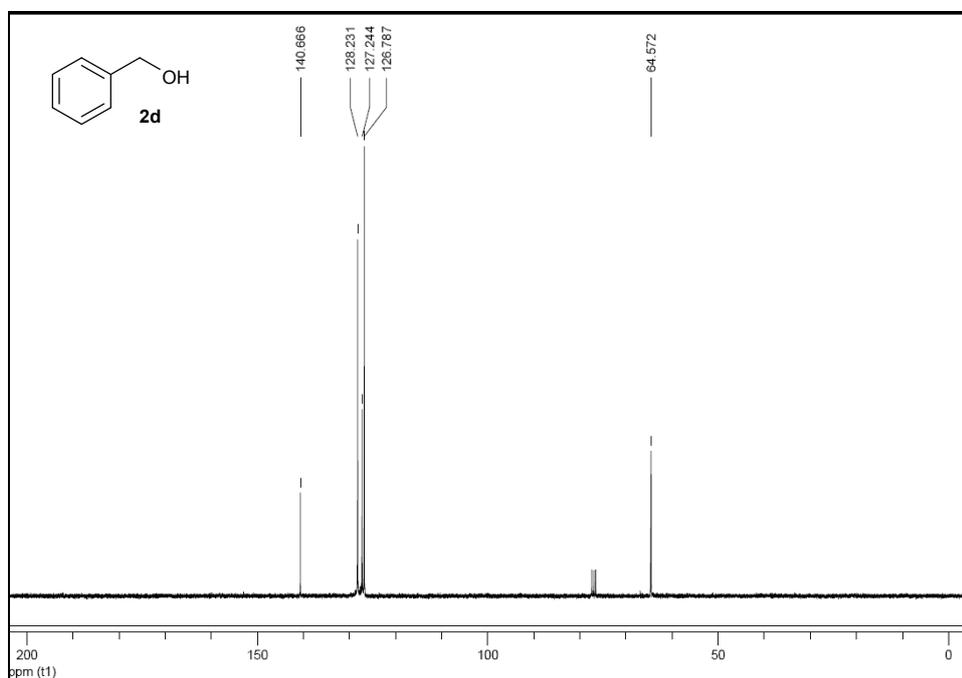
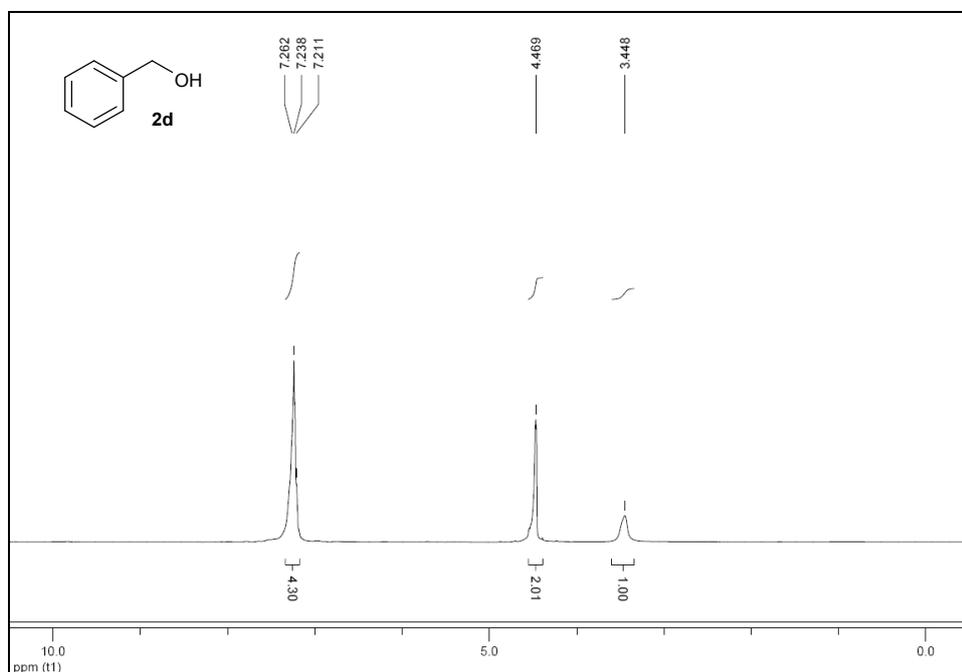
**Entry 3 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.49 (s, 6H), 7.10-7.37 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  32.47, 75.15, 124.65, 126.27, 127.86, 150.01.



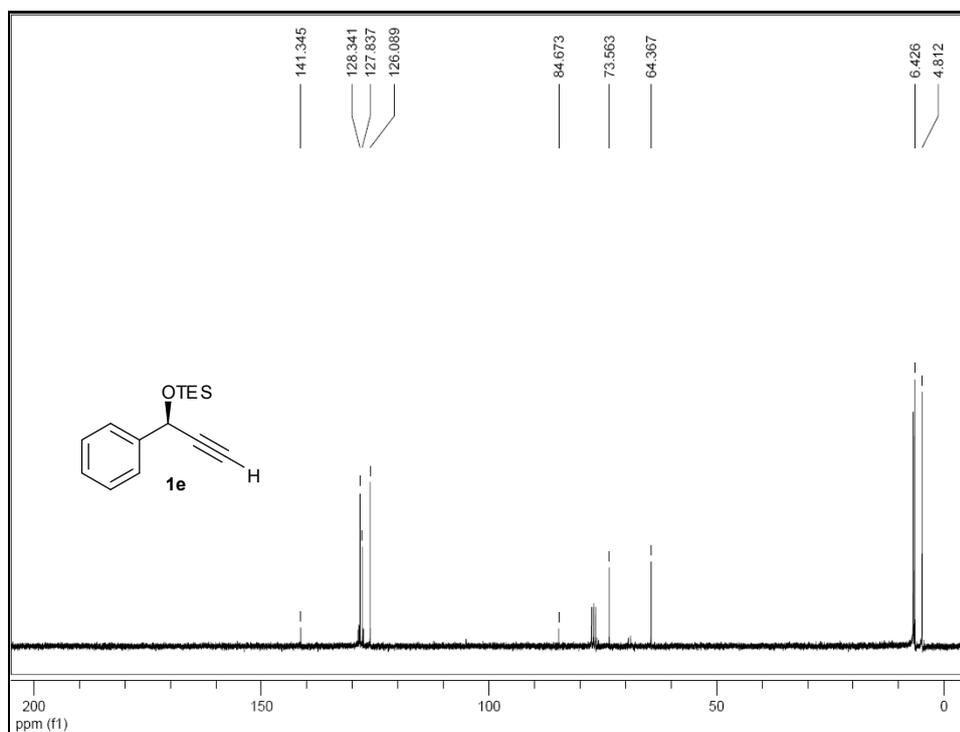
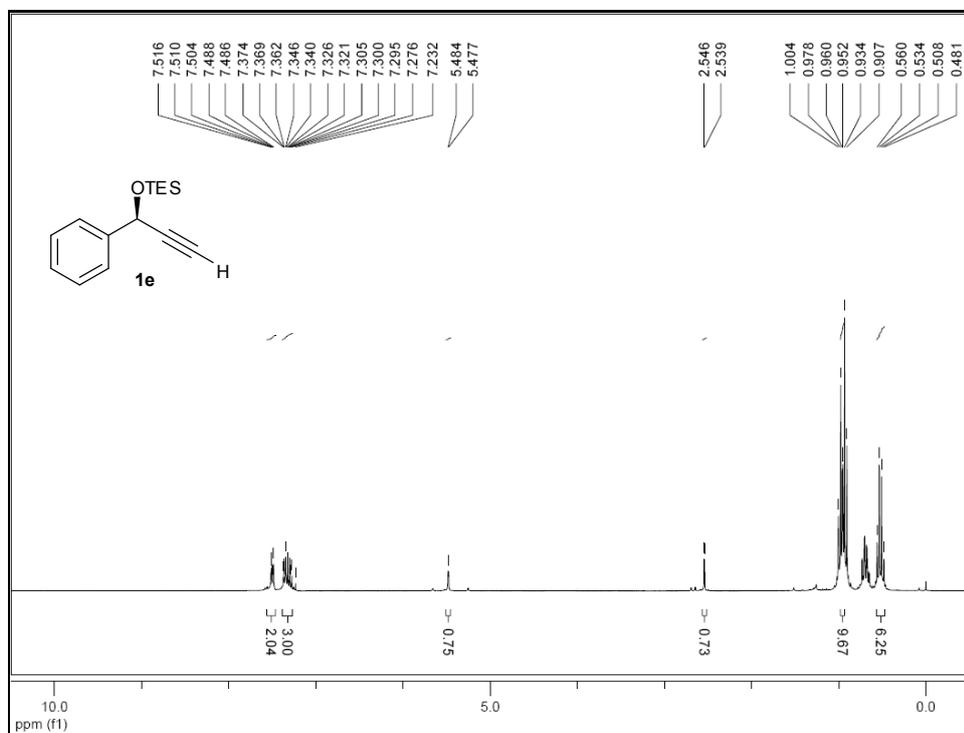
**Entry 4 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.48 (q,  $J = 8.1$  Hz, 6H), 0.81 (t,  $J = 8.1$  Hz, 9H), 4.57 (s, 2H), 7.04-7.17 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  4.48, 6.76, 64.68, 126.17, 126.90, 128.17, 141.35.



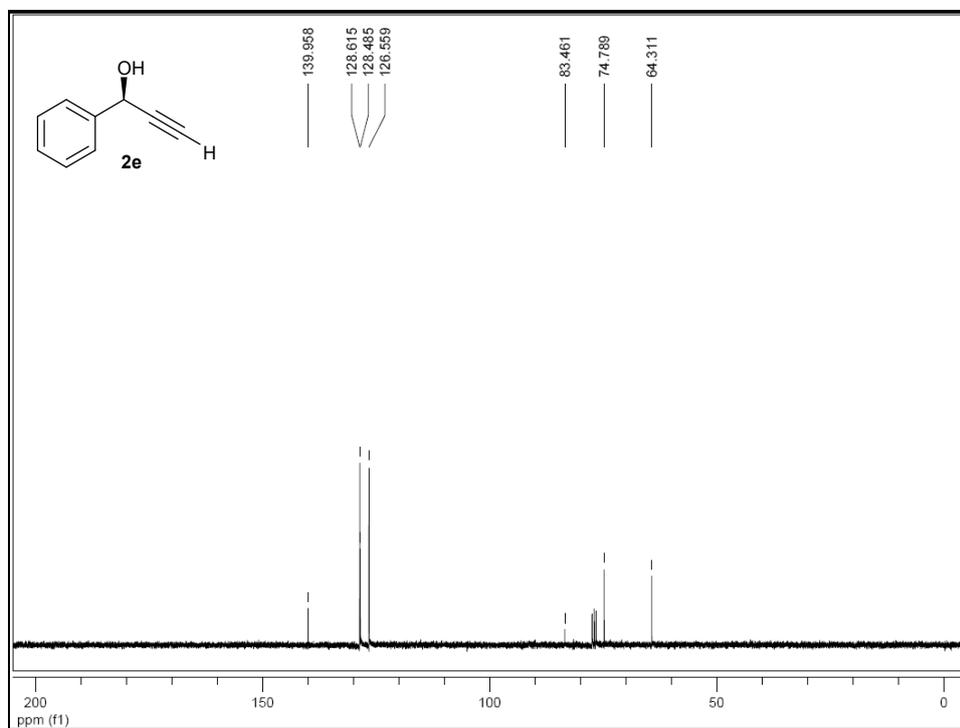
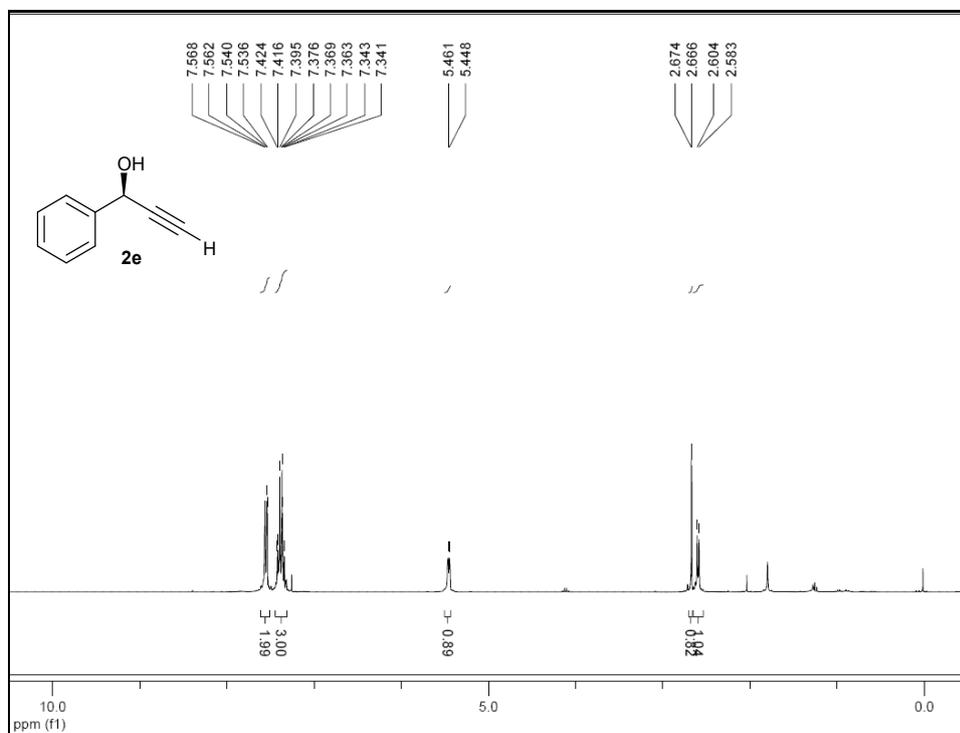
**Entry 4 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.45 (s, 1H), 4.47 (s, 2H), 7.21-7.26 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  64.57, 126.79, 127.24, 128.23, 140.67.



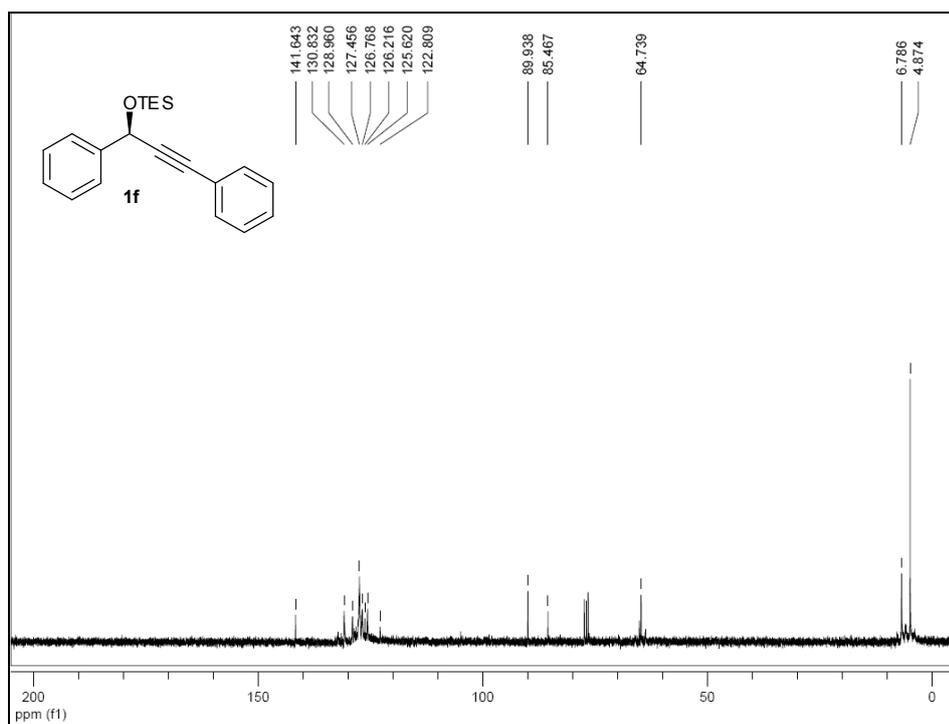
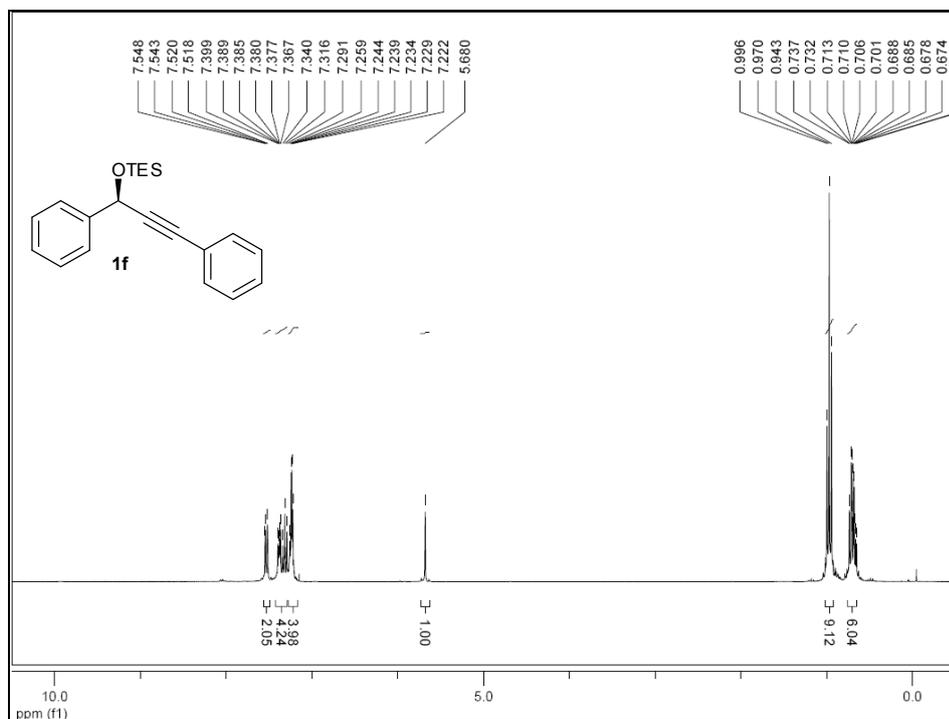
**Entry 5 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.52 (q,  $J = 7.9$  Hz, 6H), 0.91-1.00 (m, 9H), 2.54 (d,  $J = 2.1$  Hz, 1H), 5.48 (d,  $J = 2.1$  Hz, 1H), 7.27-7.37 (m, 3H), 7.49-7.52 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  4.81, 6.43, 64.37, 73.56, 84.67, 126.09, 127.84, 128.34, 141.35.



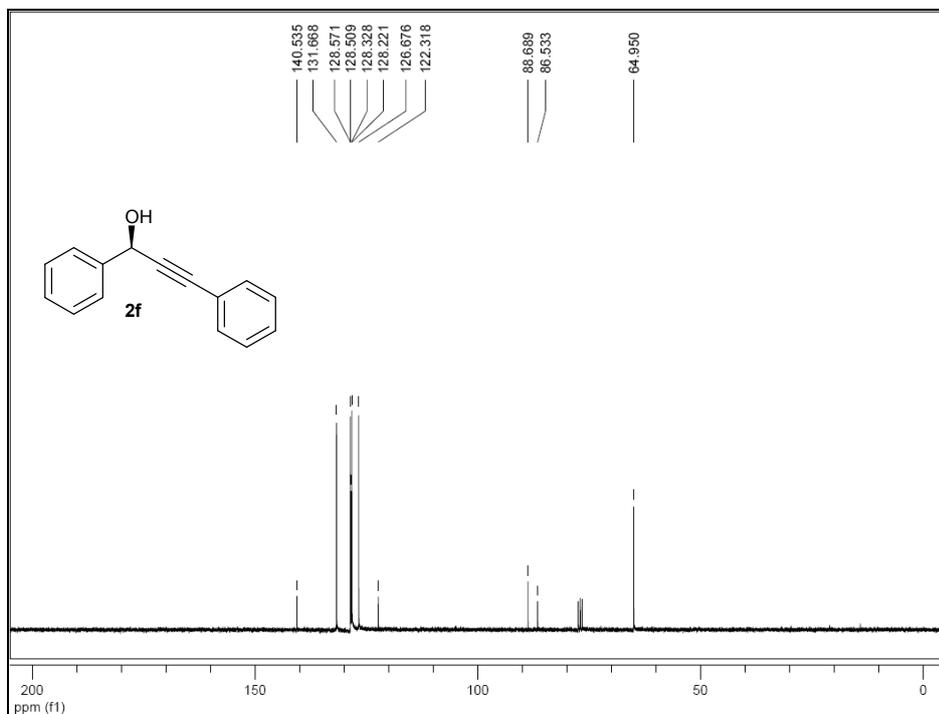
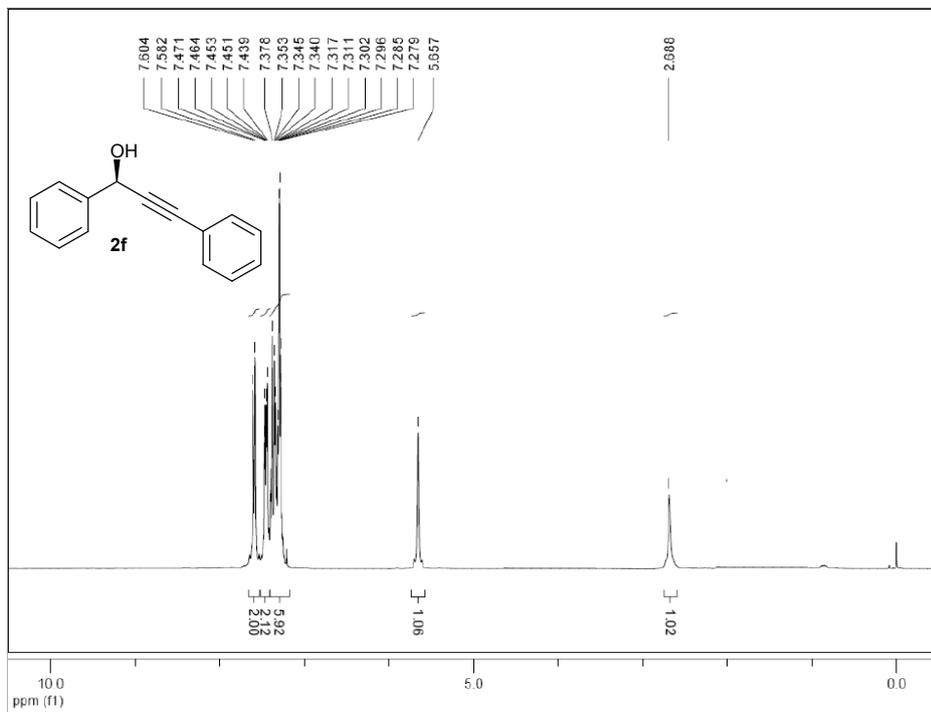
**Entry 5 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  2.59 (d,  $J = 6.3$  Hz, 1H), 2.67 (d,  $J = 2.1$  Hz, 1H), 5.46 (dd,  $J = 6.0$  Hz,  $J = 2.1$  Hz, 1H), 7.34-7.42 (m, 3H), 7.54-7.57 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  64.31, 74.79, 83.46, 126.56, 128.49, 128.62, 139.96.



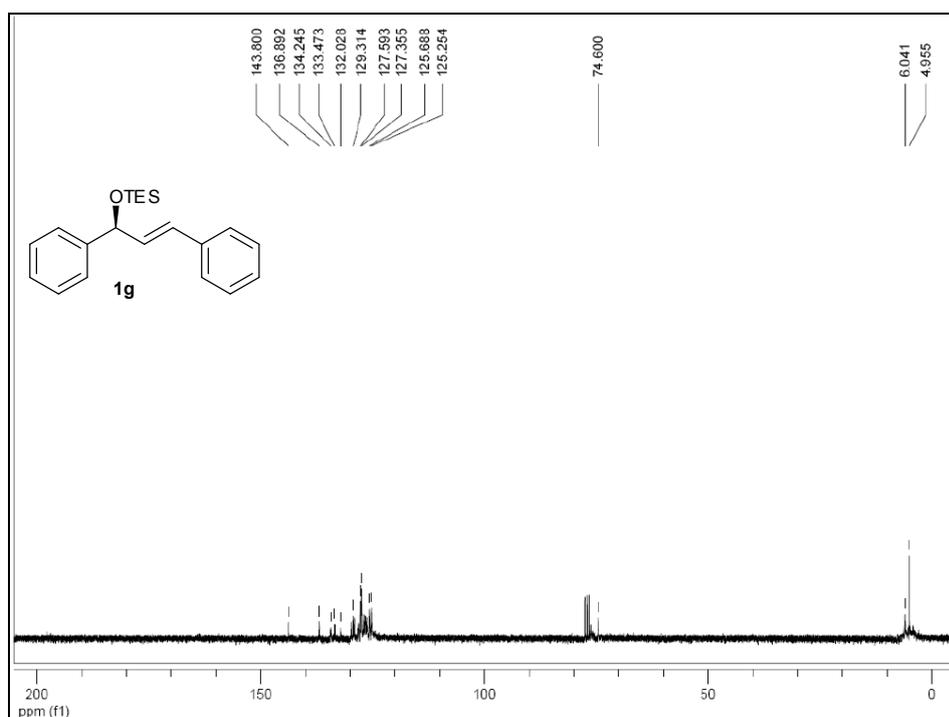
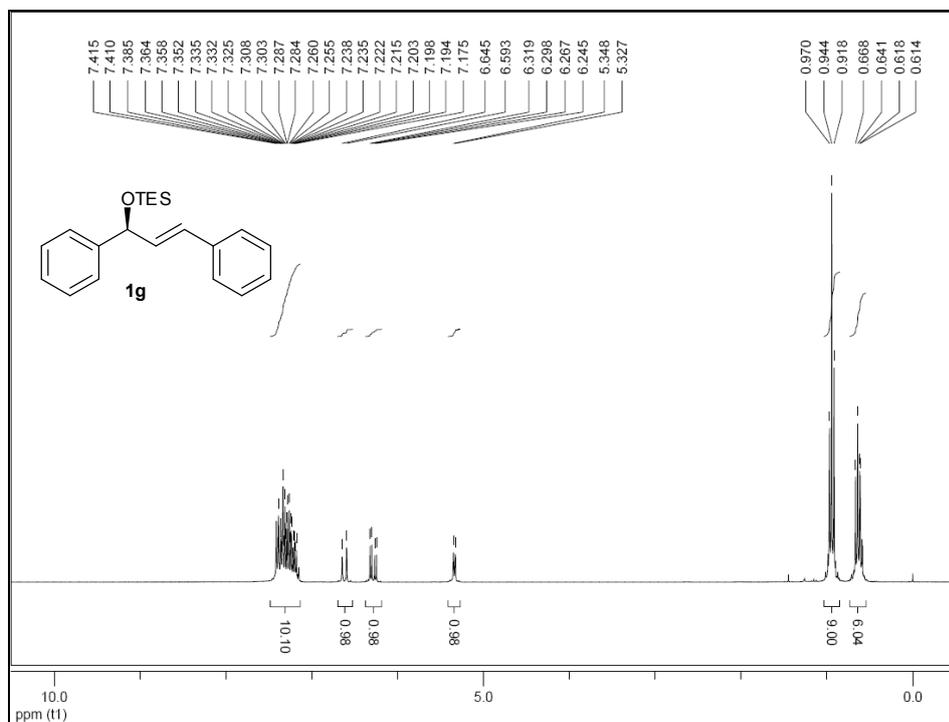
**Entry 6 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.68-0.74 (m, 6H), 0.97 (t,  $J = 7.8$  Hz, 9H), 5.68 (s, 1H), 7.22-7.26 (m, 4H), 7.29-7.40 (m, 4H), 7.52-7.55 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  4.87, 6.79, 64.74, 85.47, 89.94, 122.81, 125.62, 126.22, 126.77, 127.46, 128.96, 130.83, 141.64.



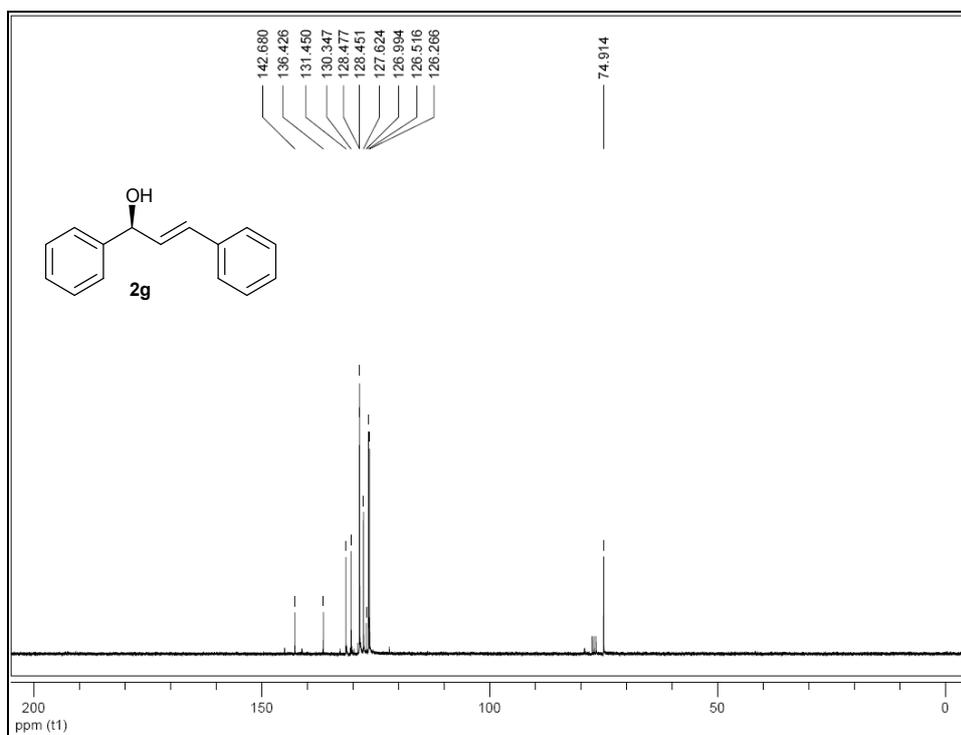
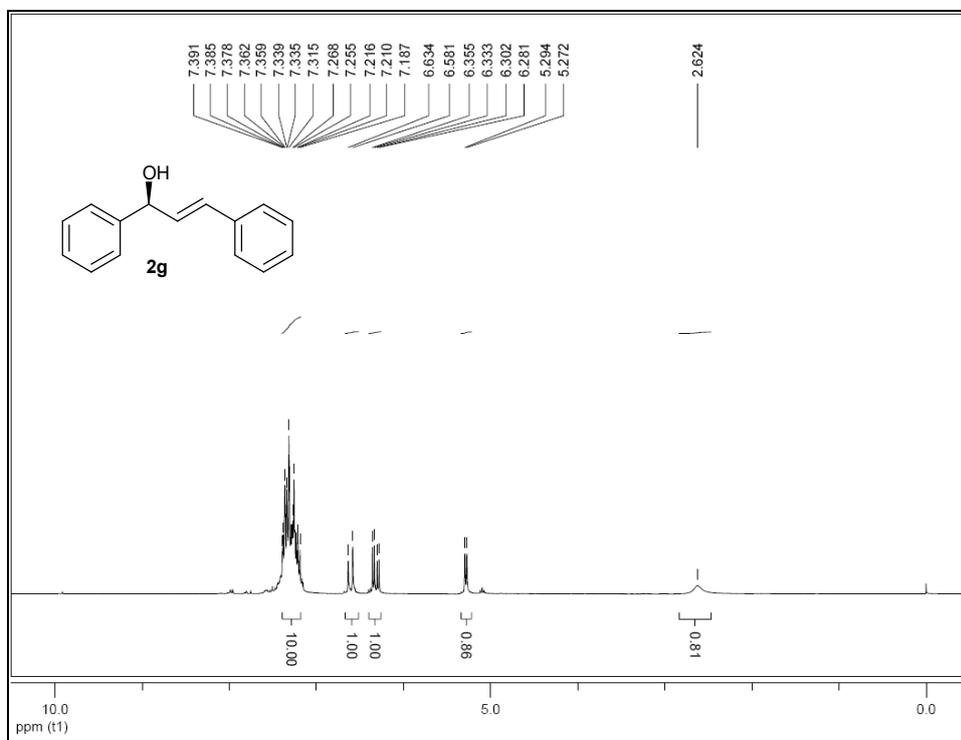
**Entry 6 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  2.69 (s, 1H), 5.66 (s, 1H), 7.28-7.32 (m, 6H), 7.44-7.47 (m, 2H), 7.58-7.60 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  64.95, 86.53, 88.69, 122.32, 126.68, 128.22, 128.32, 128.51, 128.57, 131.67, 140.54.



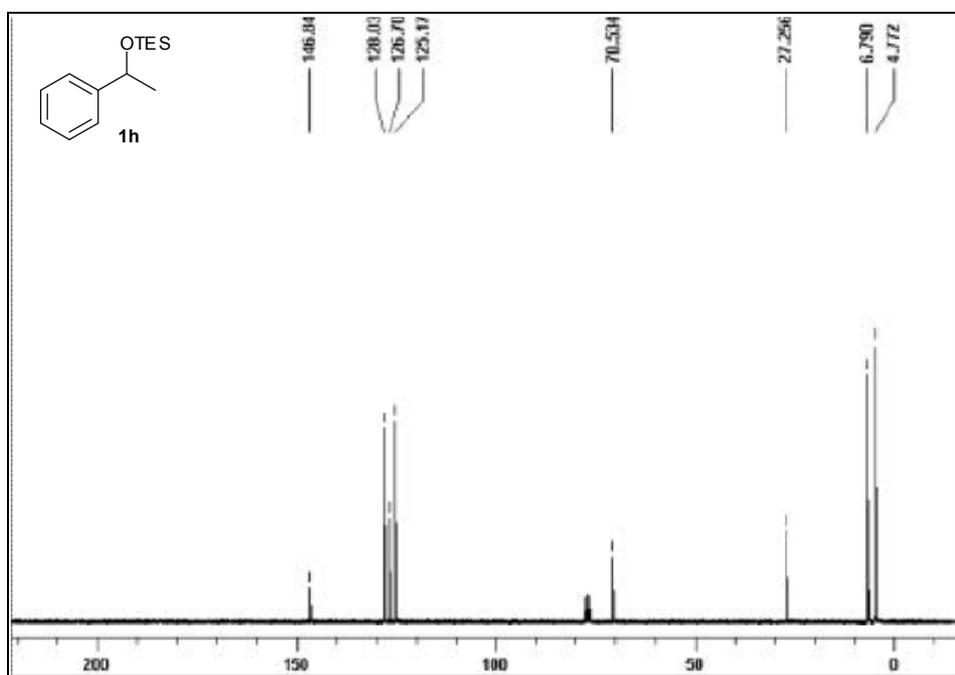
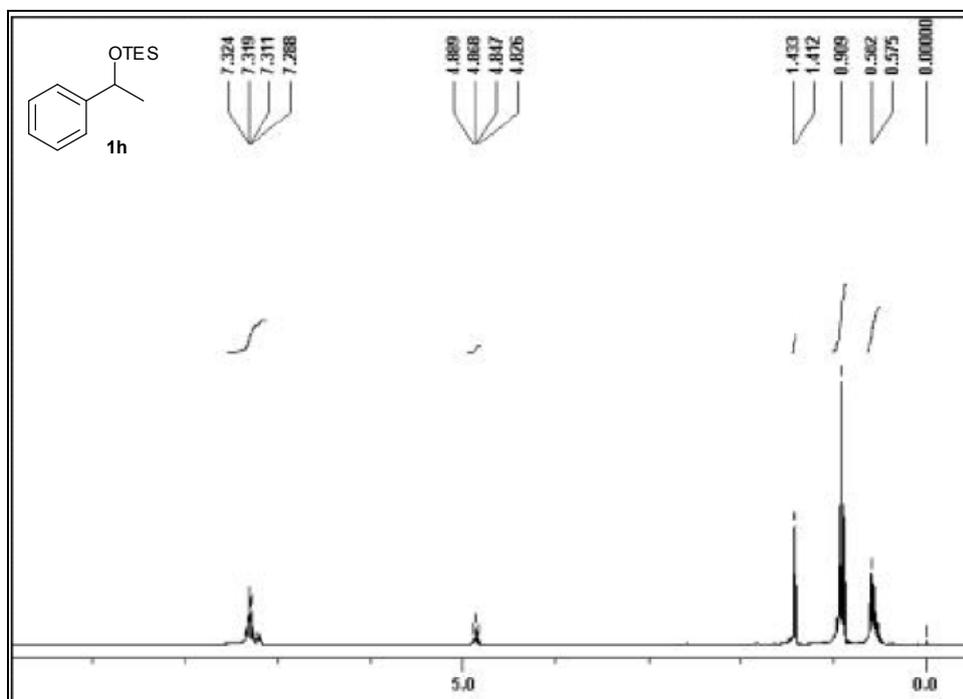
**Entry 7 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.61-0.67 (m, 6H), 0.94 (t,  $J = 7.8$  Hz, 9H), 5.33 (t,  $J = 6.3$  Hz, 1H), 6.28 (dd,  $J = 15.6$  Hz,  $J = 6.6$  Hz, 1H), 6.62 (d,  $J = 15.6$  Hz, 1H), 7.18-7.42 (m, 10H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  4.96, 6.04, 74.60, 125.25, 125.69, 127.36, 127.59, 129.31, 132.03, 133.47, 134.25, 136.89, 143.80.



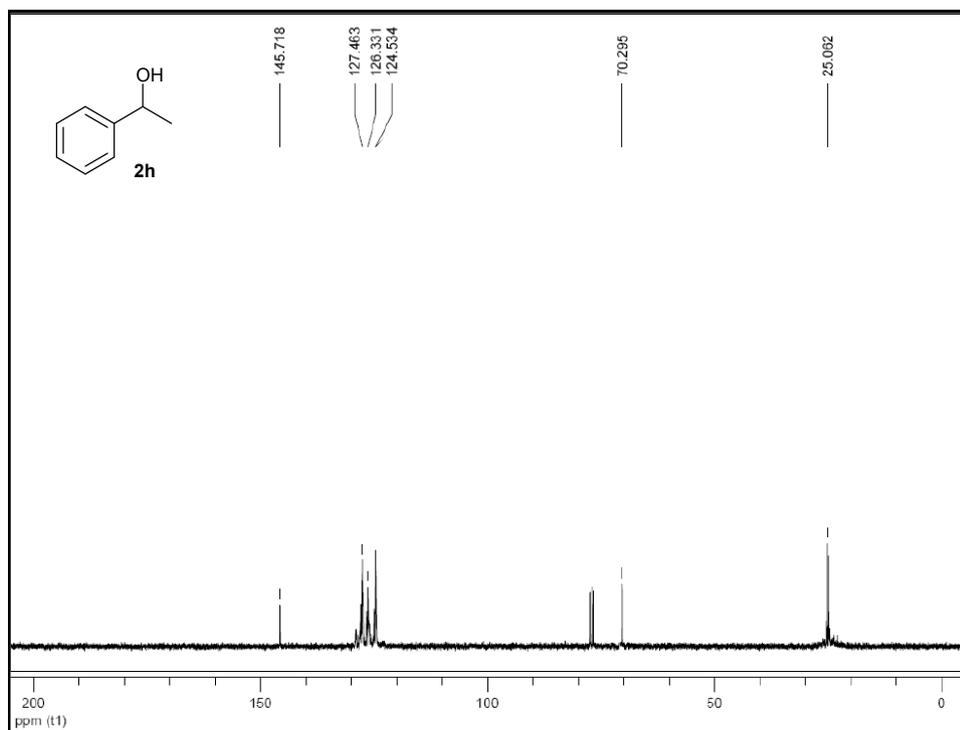
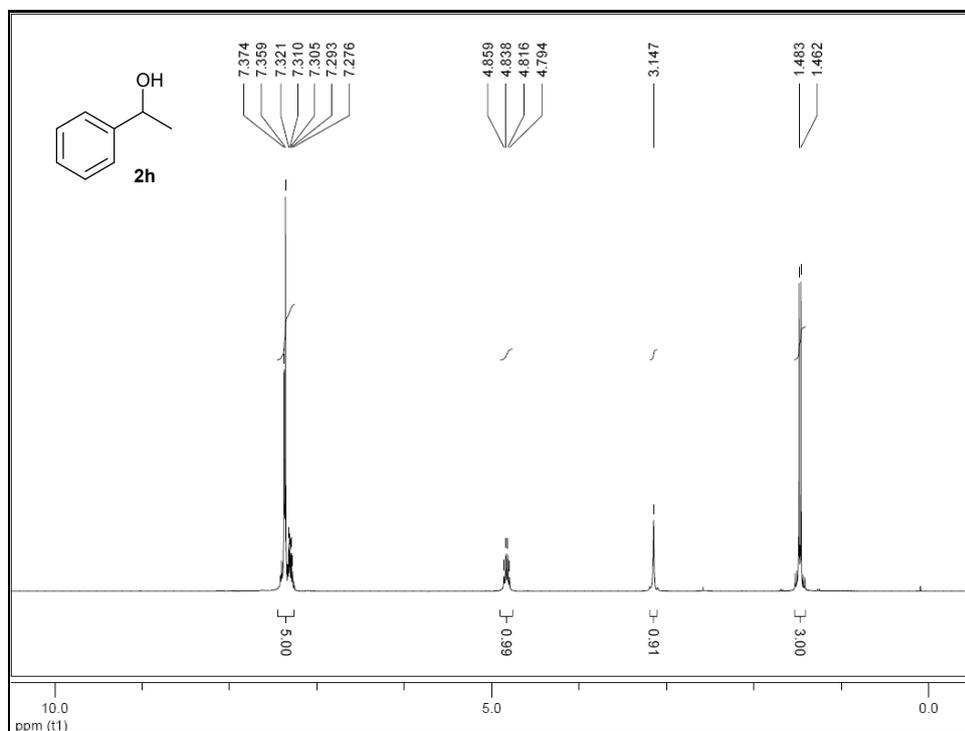
**Entry 7 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  2.62 (br s, 1H), 5.28 (d,  $J = 6.6$  Hz, 1H), 6.32 (dd,  $J = 15.6$  Hz,  $J = 6.3$  Hz, 2H), 6.61 (d,  $J = 15.6$  Hz, 1H), 7.19-7.39 (m, 10H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  74.91, 126.26, 126.52, 126.99, 127.62, 128.45, 128.48, 130.35, 131.45, 136.43, 142.68.



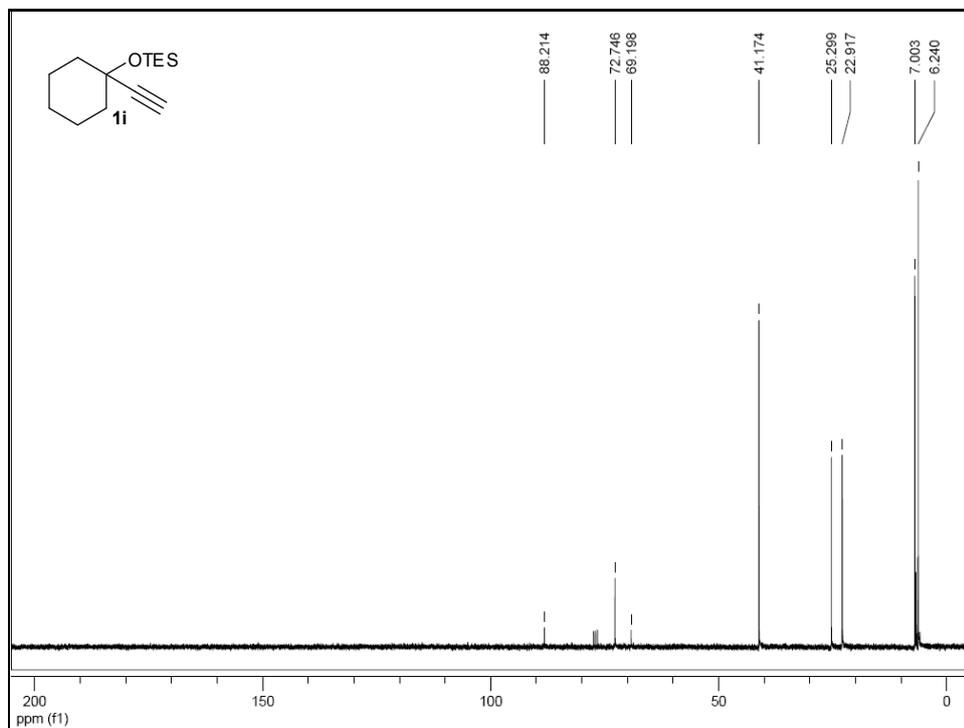
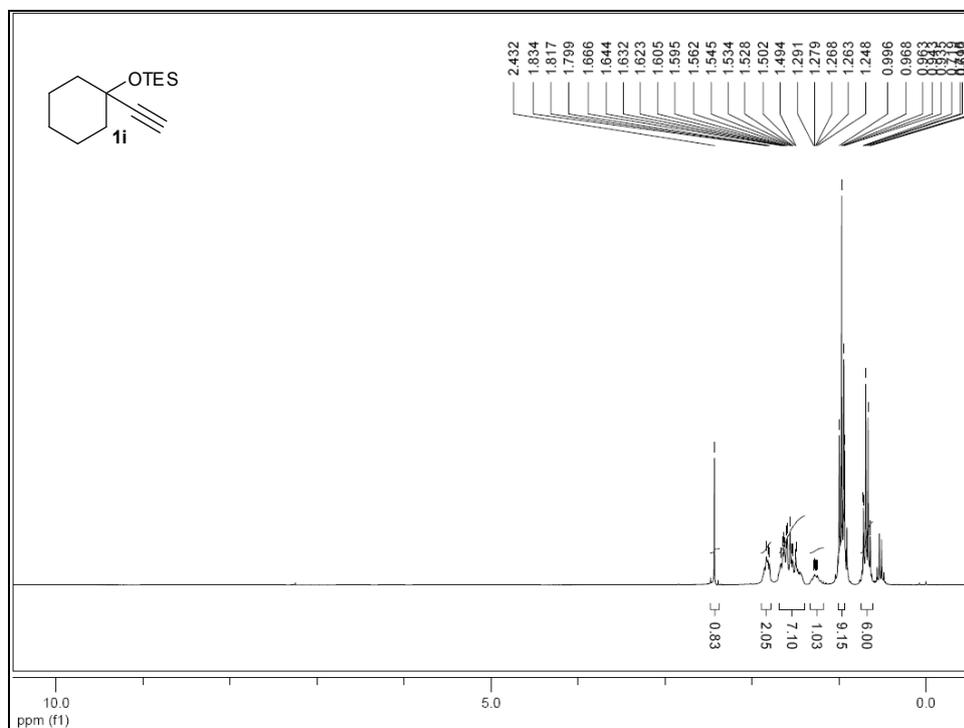
**Entry 8 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.58-0.59 (m, 6H), 0.88-0.93 (m, 9H), 1.42 (d,  $J = 6.4$  Hz, 3H), 4.85 (q,  $J = 6.3$  Hz, 1H), 7.55-7.35 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  4.77, 6.79, 27.26, 70.53, 125.18, 126.71, 128.04, 146.84.



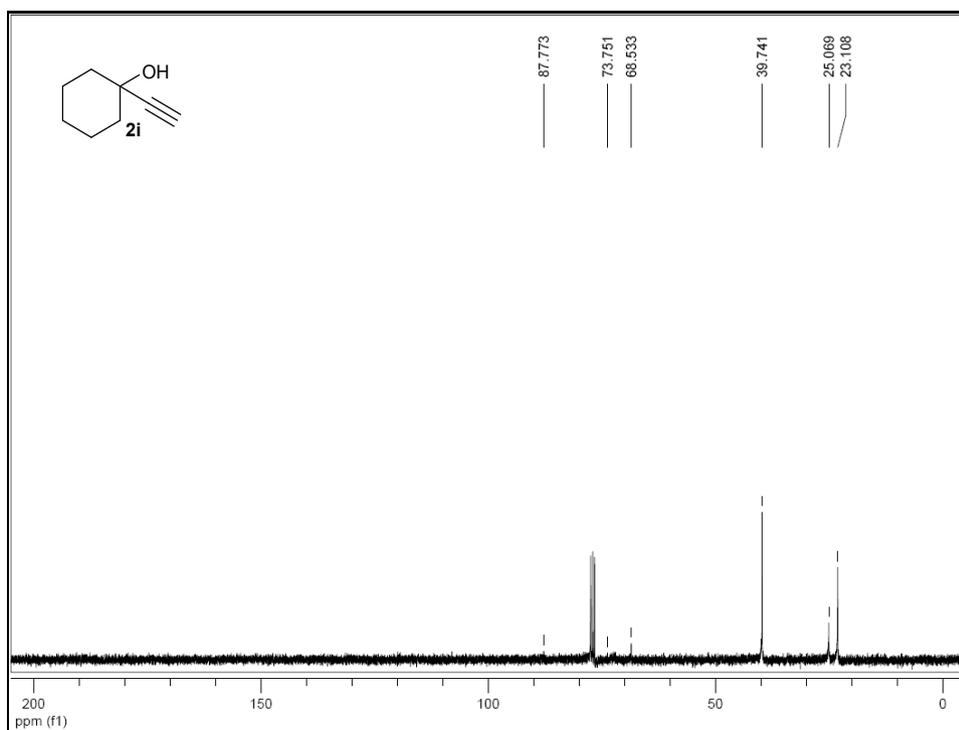
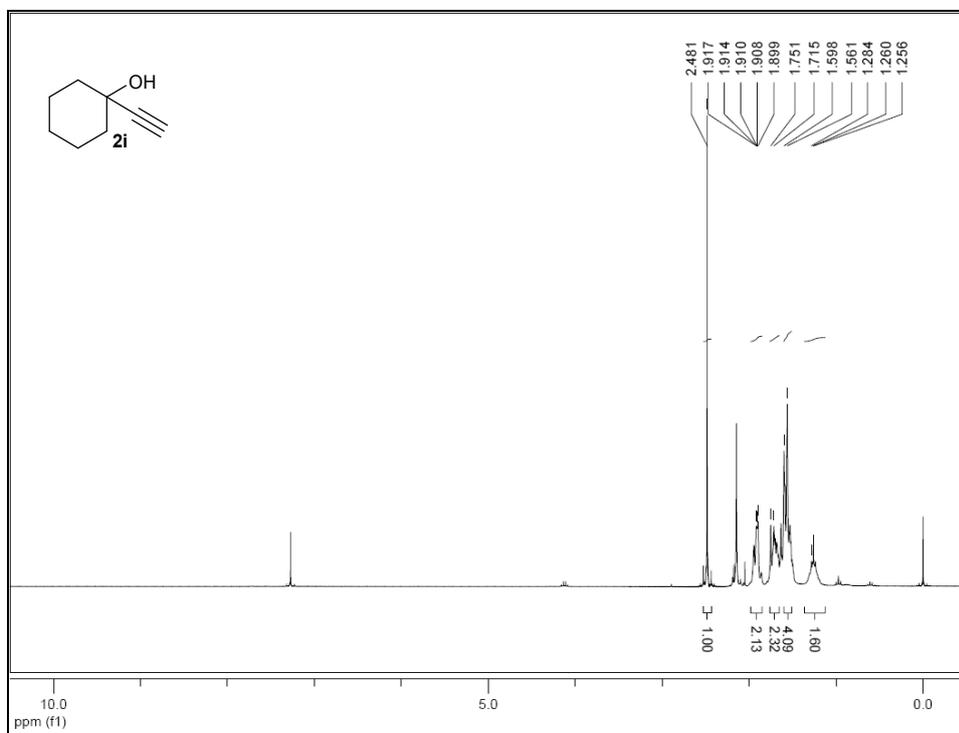
**Entry 8 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.47 (d,  $J = 6.3$  Hz, 3H), 3.15 (s, 1H), 4.83 (q,  $J = 6.6$  Hz, 1H), 7.26-7.42 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  25.05, 70.30, 124.51, 124.53, 126.33, 145.72.



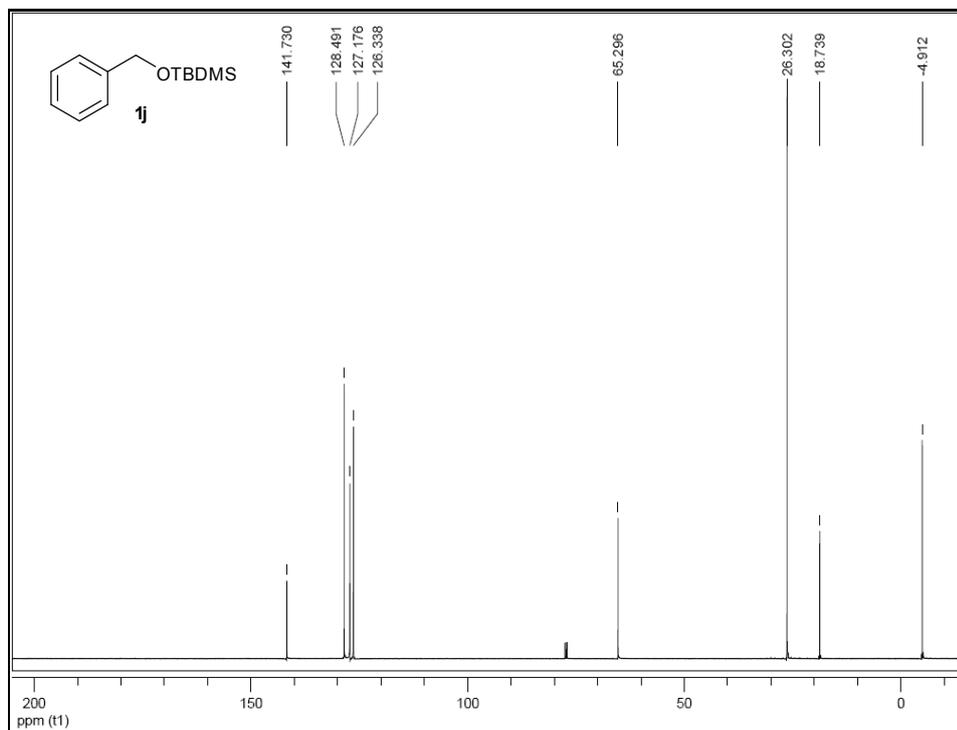
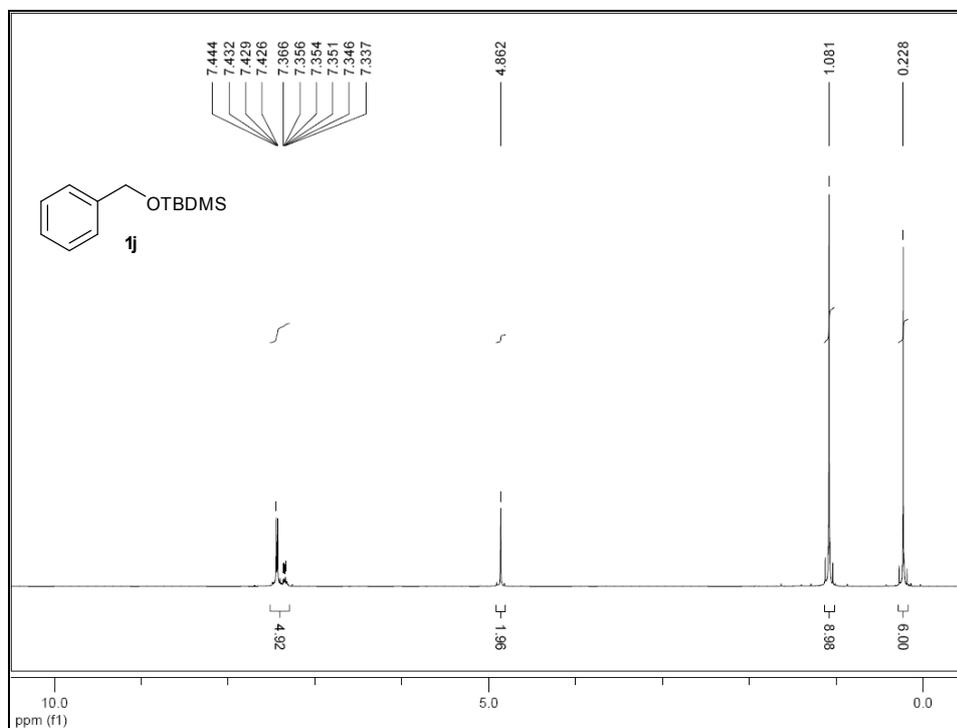
**Entry 9 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.64-0.72 (m, 6H), 0.94-0.10 (m, 9H), 1.25-1.30 (m, 1H), 1.42-1.70 (m, 7H), 1.80-1.86 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  6.24, 7.00, 22.92, 25.30, 41.17, 69.20, 72.75, 88.21.



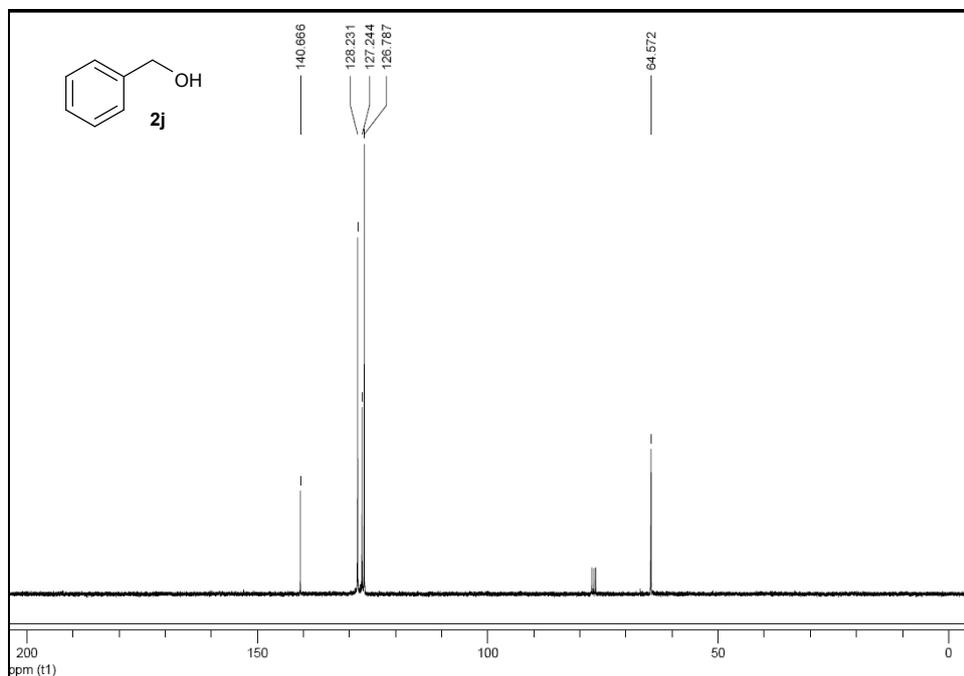
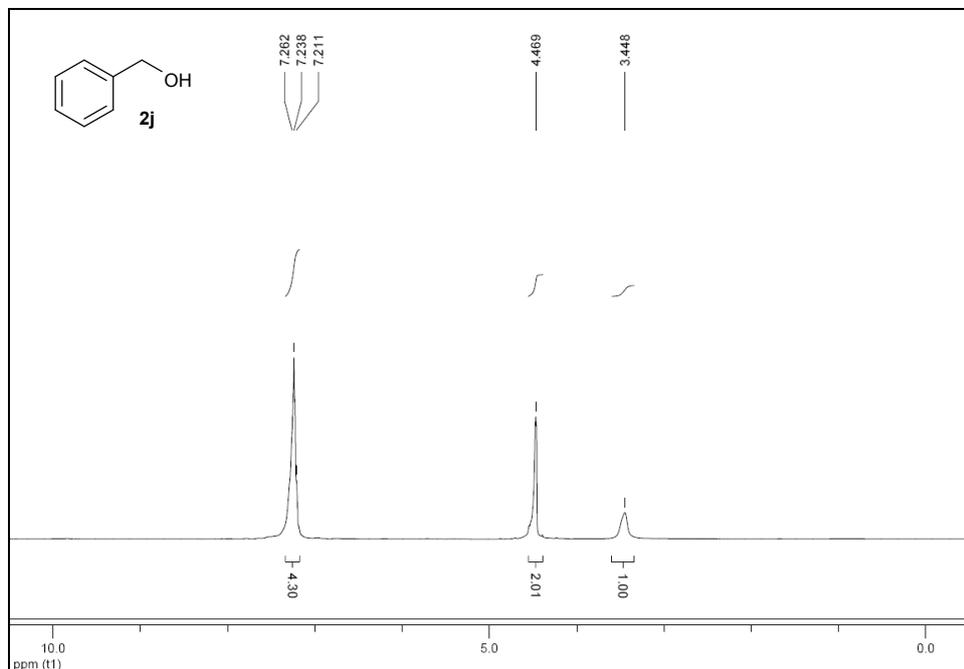
**Entry 9 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.50-1.60 (m, 4H), 1.66-1.75 (m, 2H), 1.90-1.93 (m, 2H), 2.480 (s, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  23.11, 25.15, 39.83, 68.56, 72.01, 87.81.



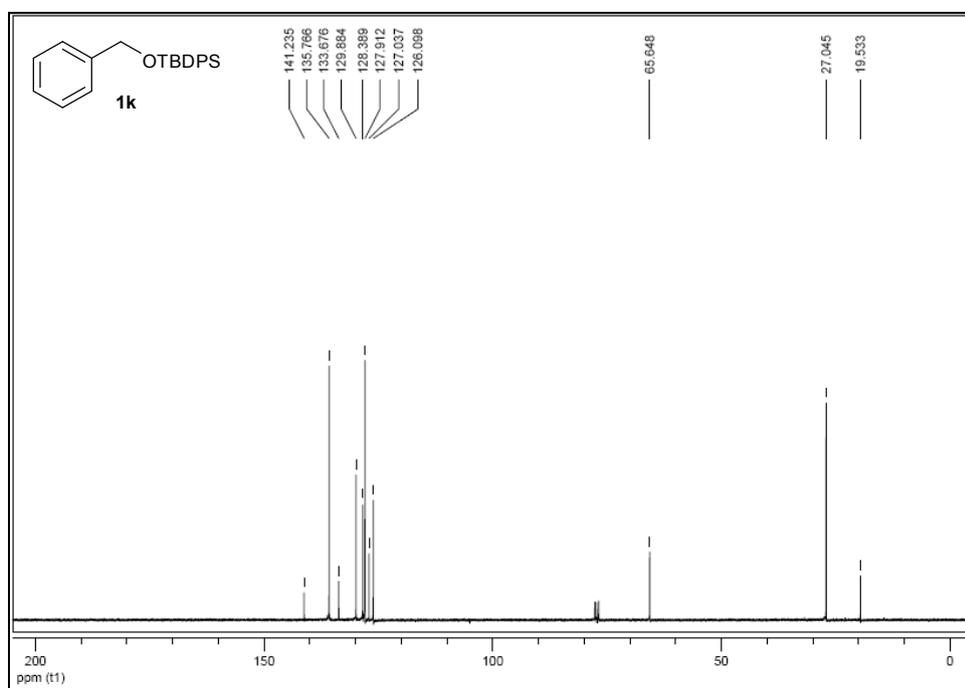
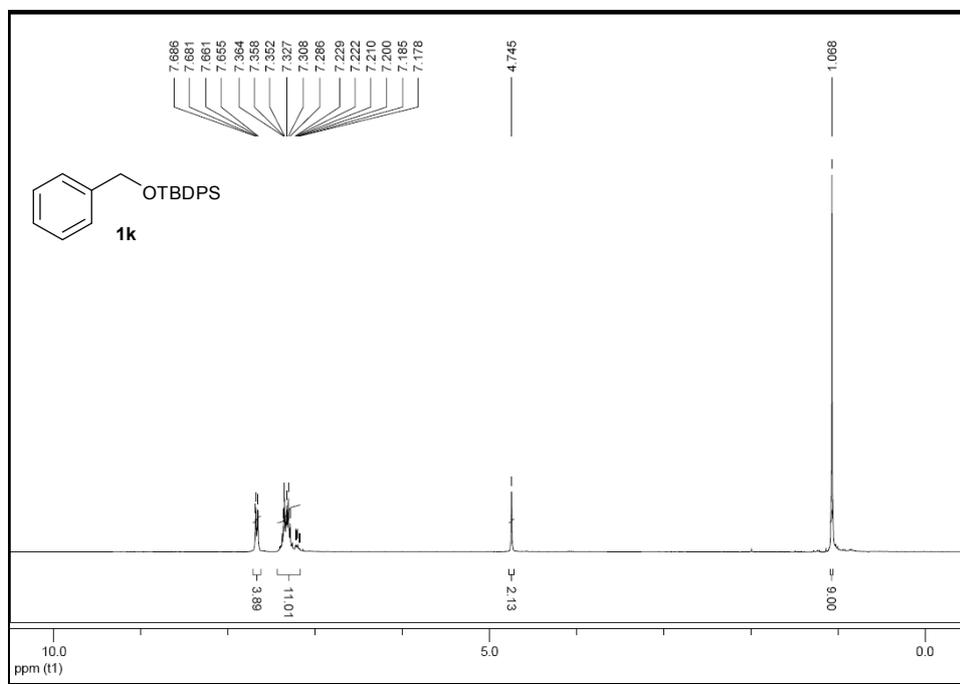
**Entry 10 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.23 (s, 6H), 1.08 (s, 9H), 4.86 (s, 2H), 7.34-7.44 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  -4.91, 18.74, 26.30, 65.30, 126.34, 127.18, 128.49, 141.73



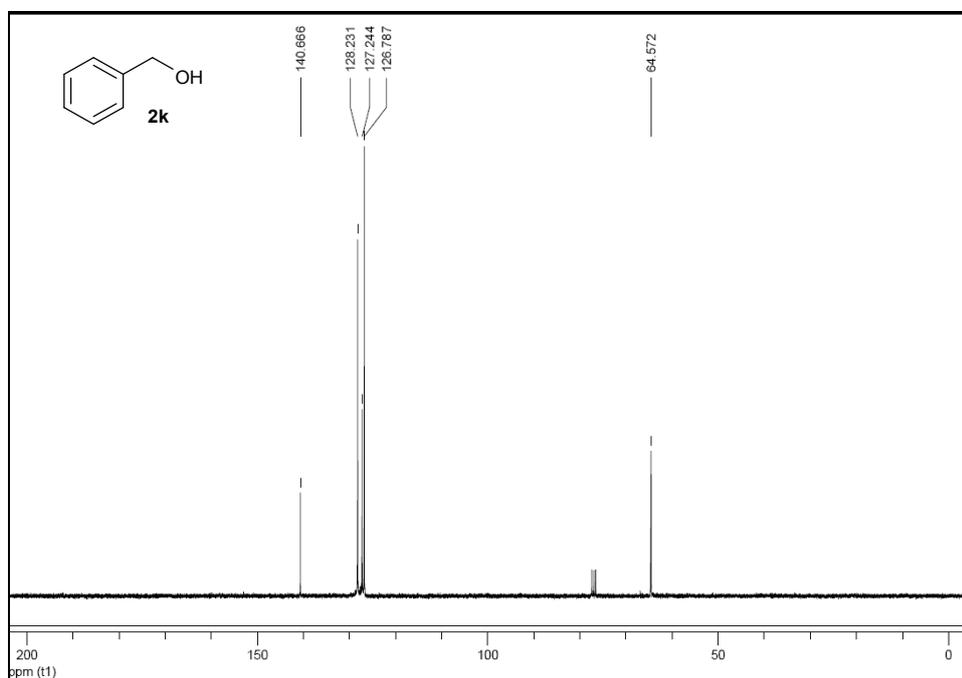
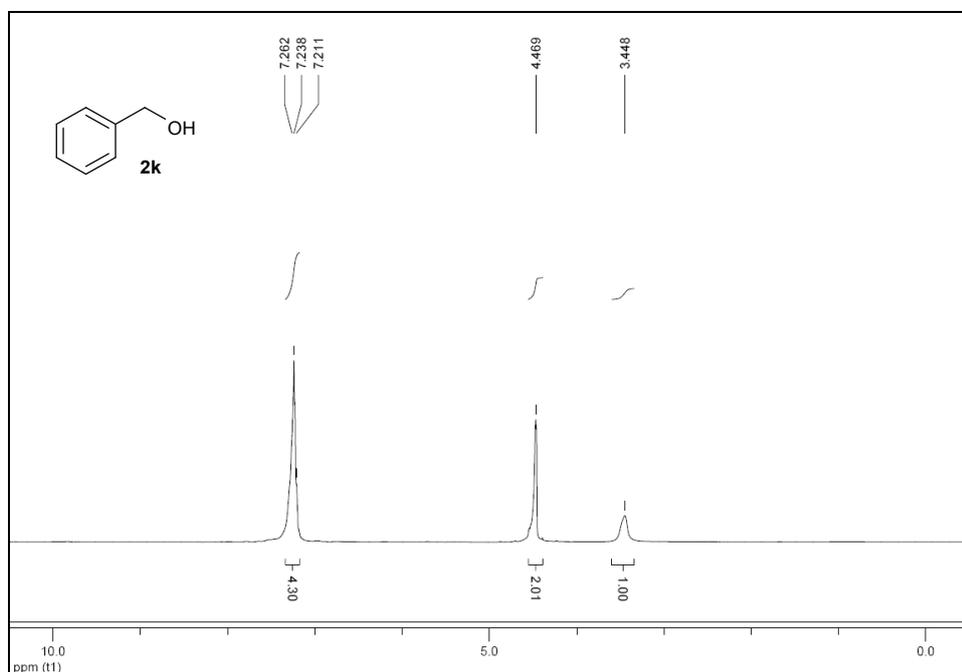
**Entry 10 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.45 (s, 1H), 4.48 (s, 2H), 7.21-7.26 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  64.57, 126.79, 127.24, 128.23, 140.67.



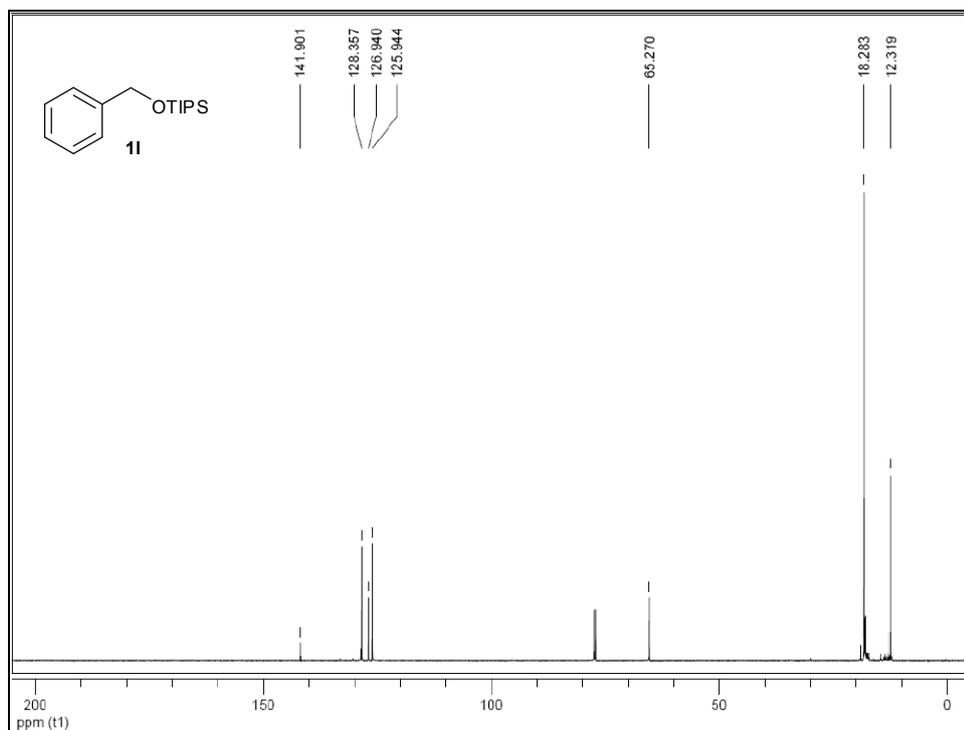
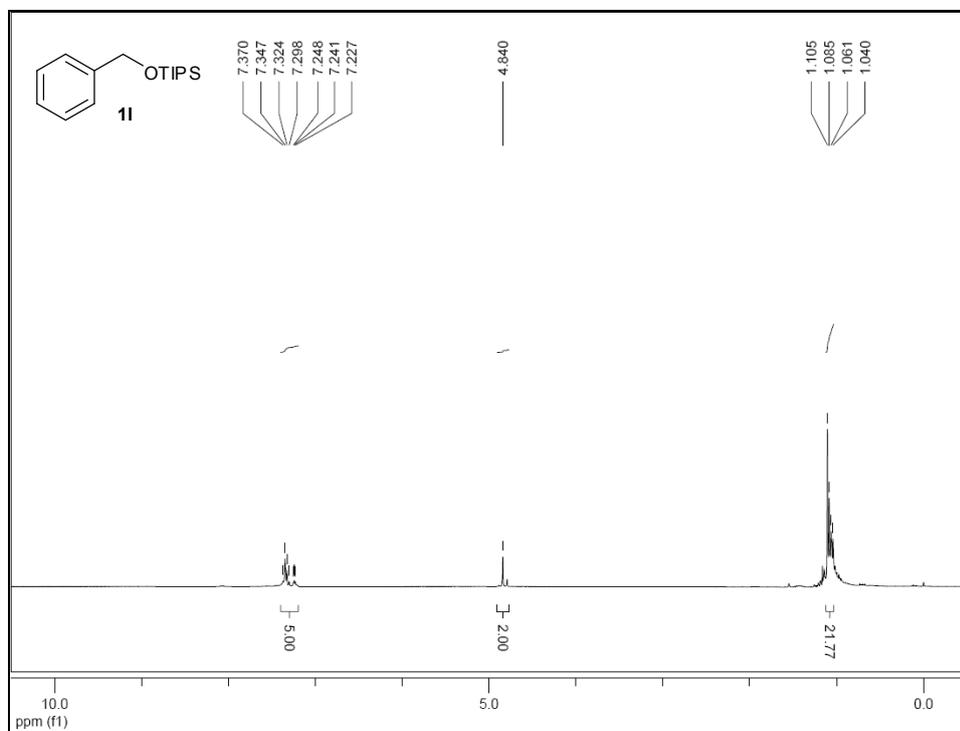
**Entry 11 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.07 (s, 9H), 4.75 (s, 2H), 7.18-7.36 (m, 10H), 7.66-7.69 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  19.53, 27.0, 65.65, 126.09, 127.04, 127.91, 128.39, 129.88, 133.68, 125.77, 141.24.



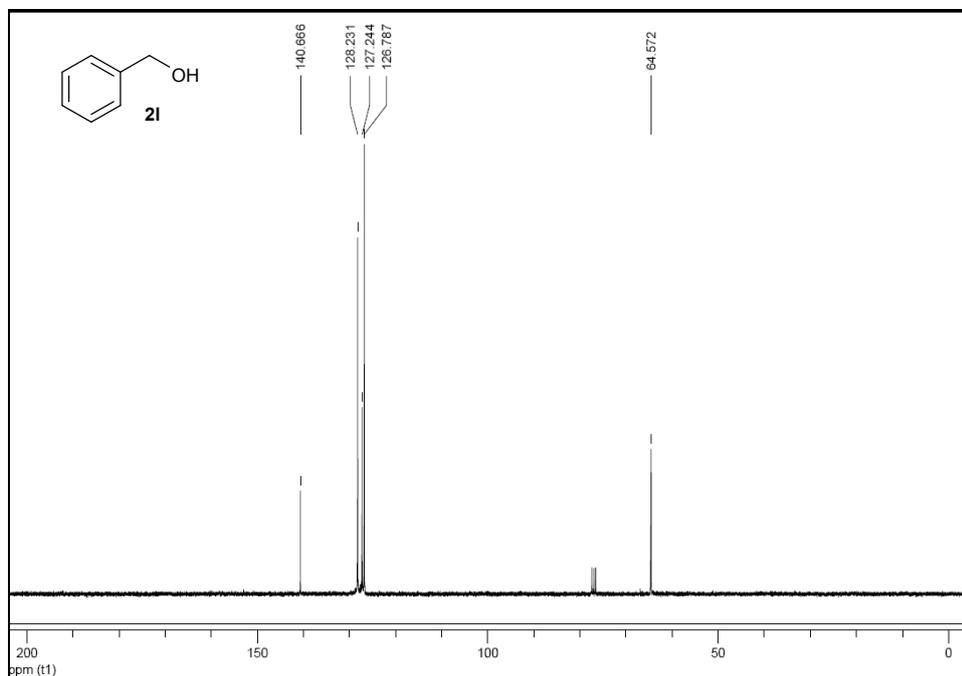
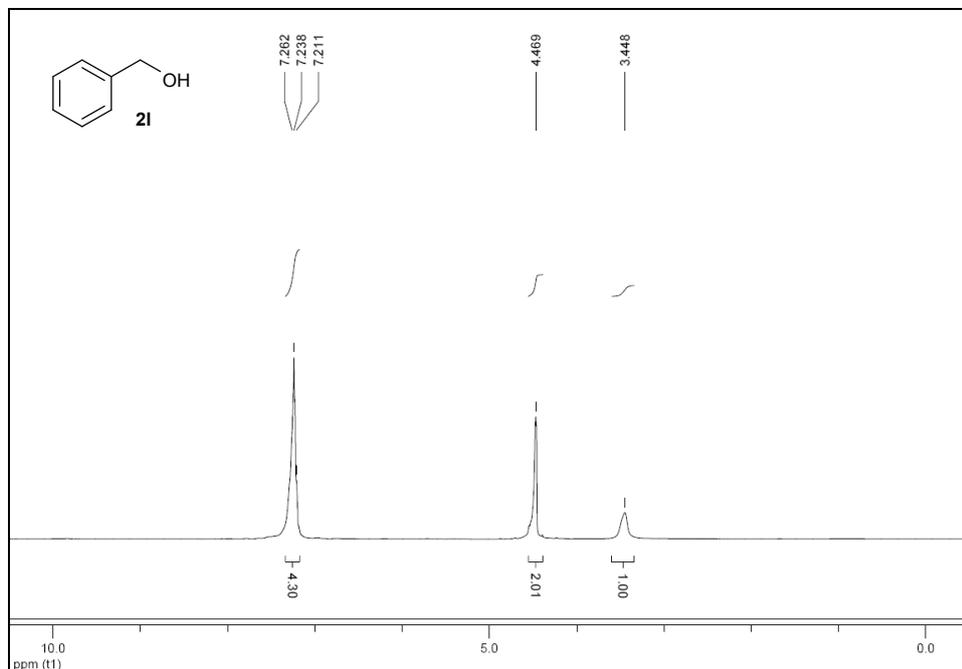
**Entry 11 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.45 (br s, 1H), 4.47 (s, 2H), 7.21-7.26 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  64.57, 126.79, 127.24, 128.23, 140.67.



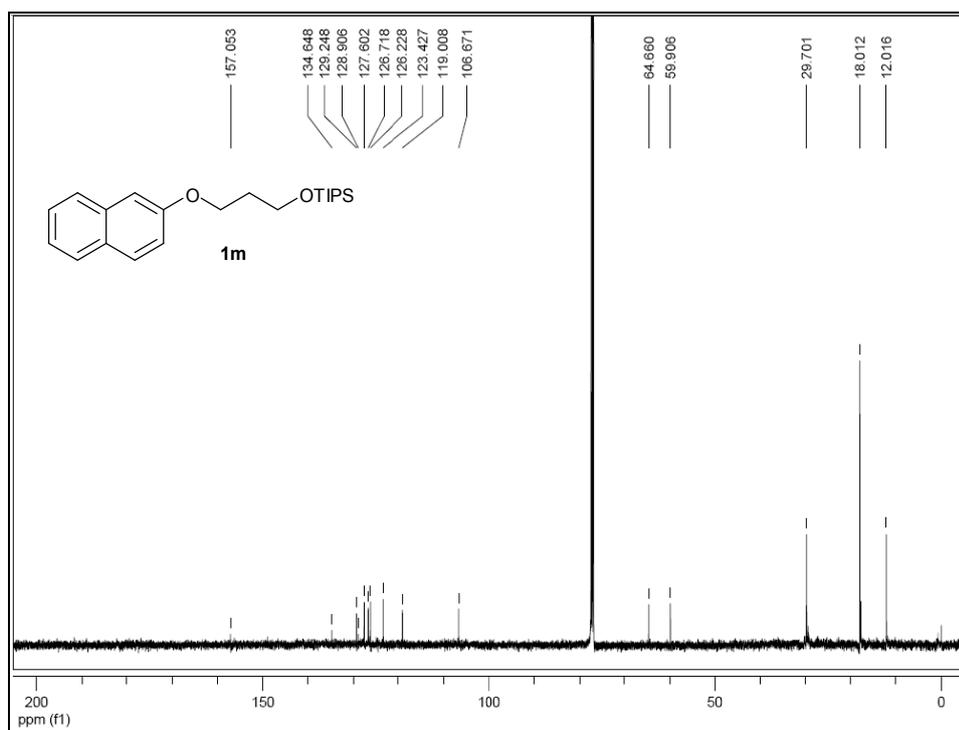
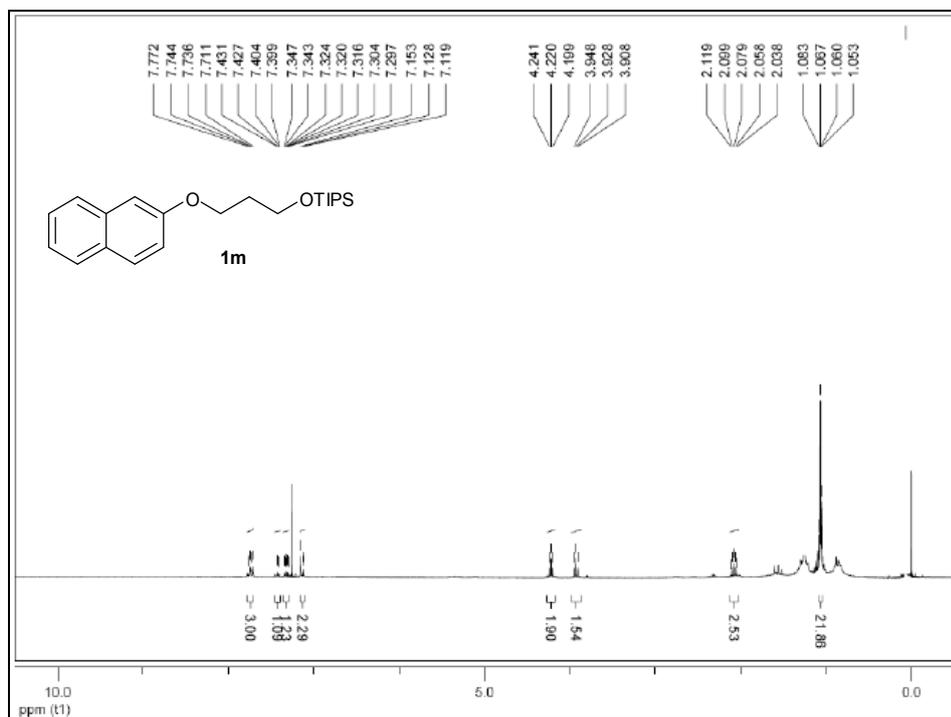
**Entry 12 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.04-1.11 (m, 21H), 4.84 (s, 2H), 7.23-7.34 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  12.32, 18.28, 65.27, 125.94, 126.94, 128.36, 141.90



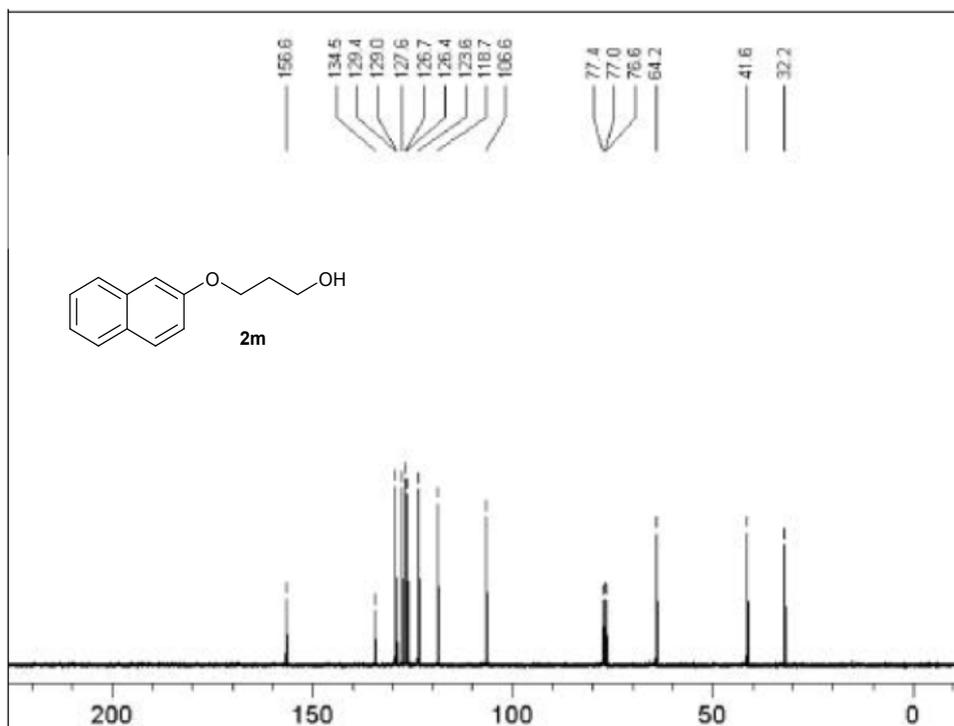
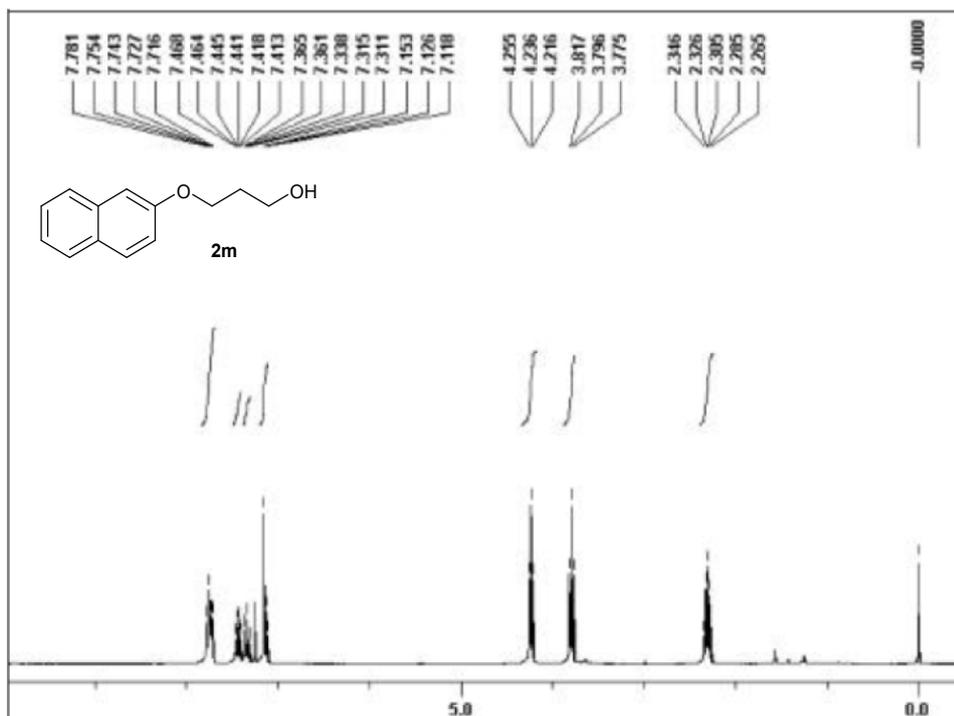
**Entry 12 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.45 (s, 1H), 4.47 (s, 2H), 7.21-7.26 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  64.57, 126.79, 127.24, 128.23, 140.67.



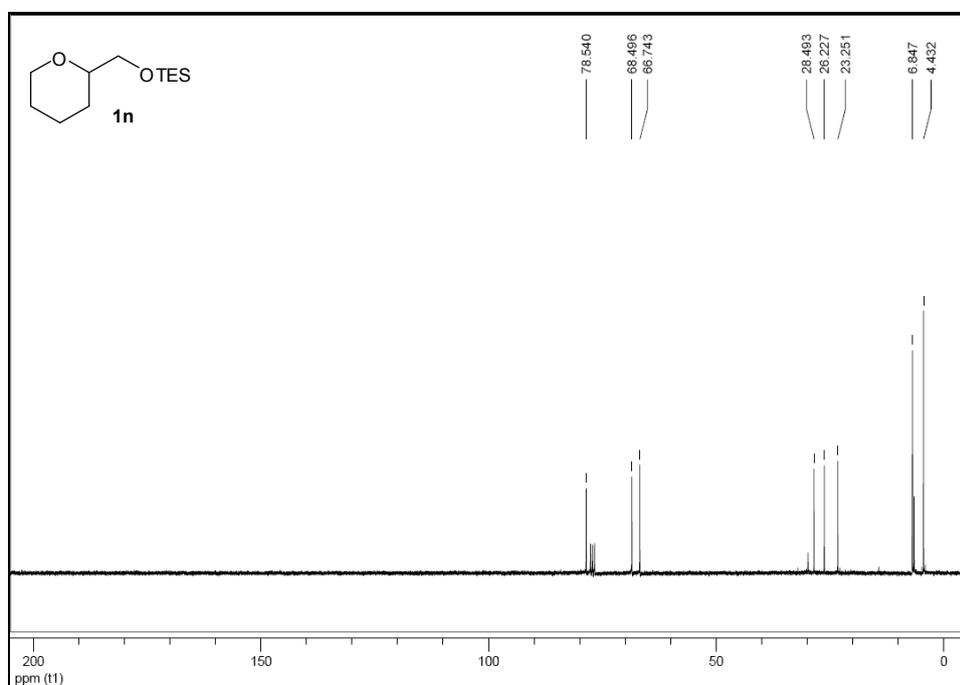
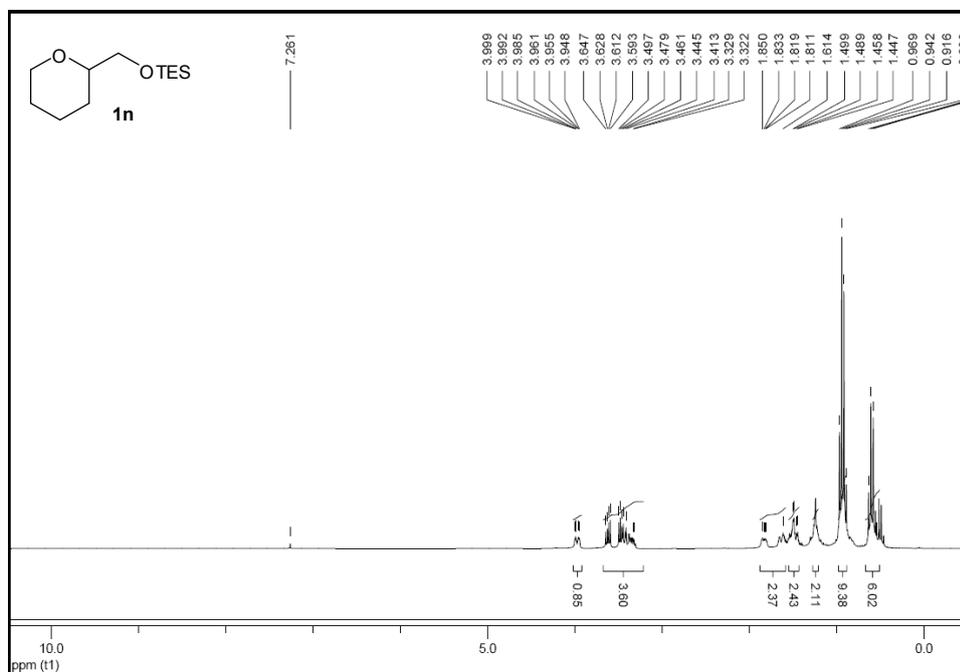
**Entry 13 (Substrate 1m):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.05-1.08 (m, 21H), 2.08 (p,  $J = 6.3$  Hz, 2H), 3.93 (t,  $J = 6.0$  Hz, 2H), 4.22 (d,  $J = 6.3$  Hz, 2H), 7.12-7.77 (m, 7H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  12.02, 18.01, 29.70, 59.91, 64.66, 105.67, 119.01, 123.43, 126.22, 126.72, 127.60, 128.91, 129.24, 134.65, 157.05.



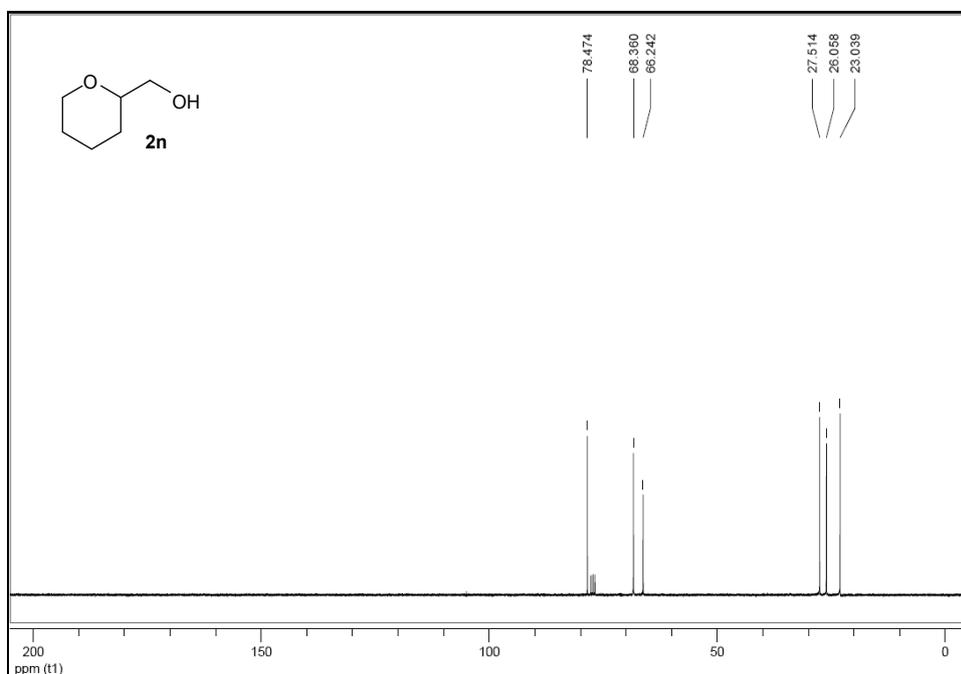
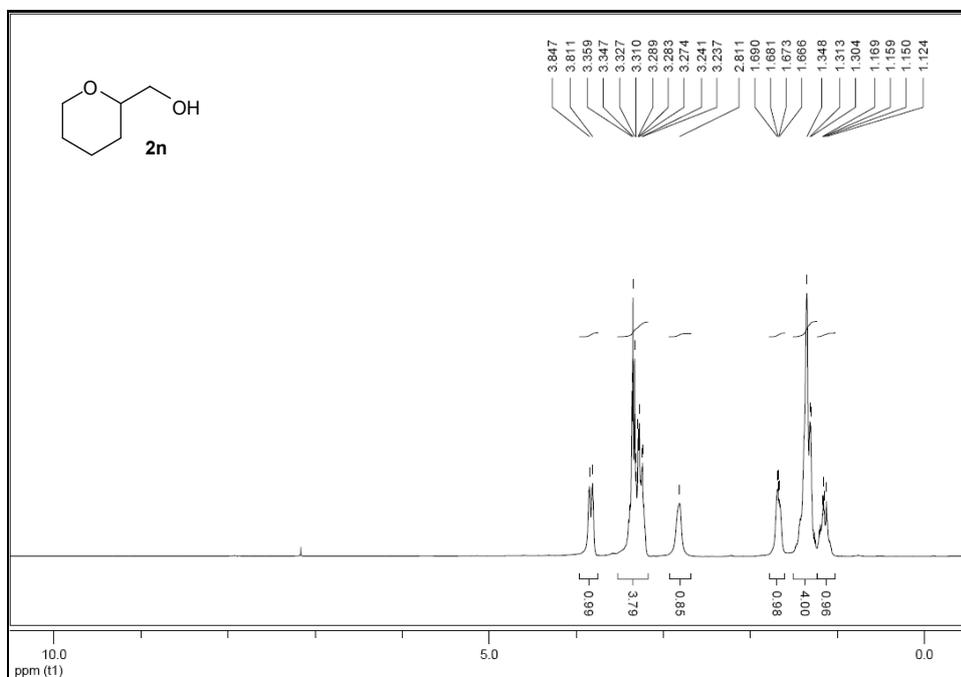
**Entry 13 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  2.27-2.35 (m, 2H), 3.80 (t,  $J = 6.3$  Hz, 2H), 4.24 (t,  $J = 6.0$  Hz, 2H), 7.12-7.78 (m, 7H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  32.25, 41.63, 64.28, 106.66, 118.72, 123.66, 126.41, 126.76, 127.63, 129.02, 129.43, 134.53, 156.80.



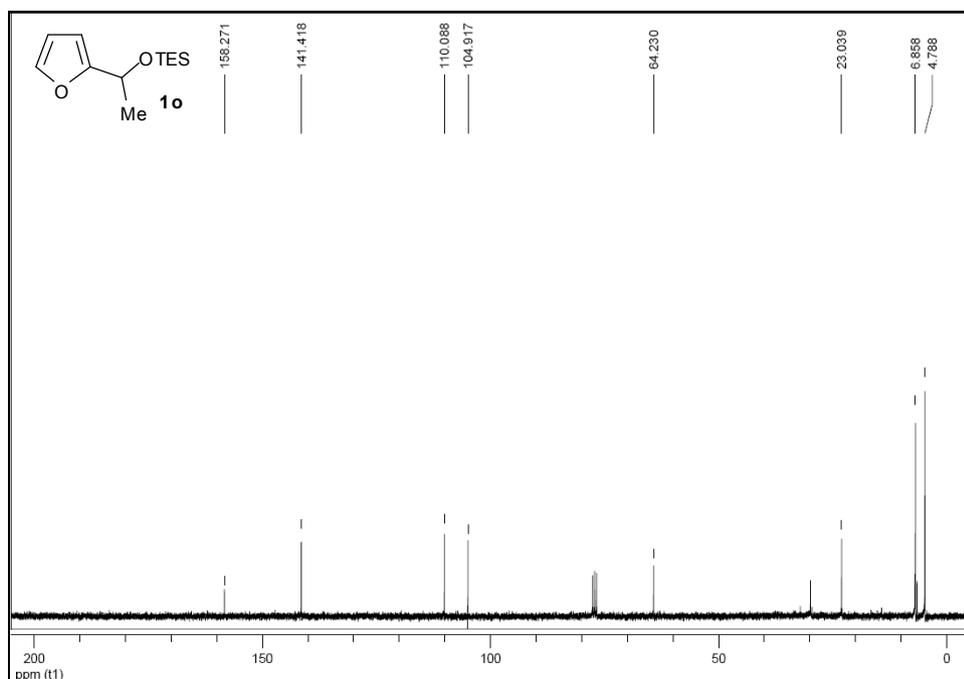
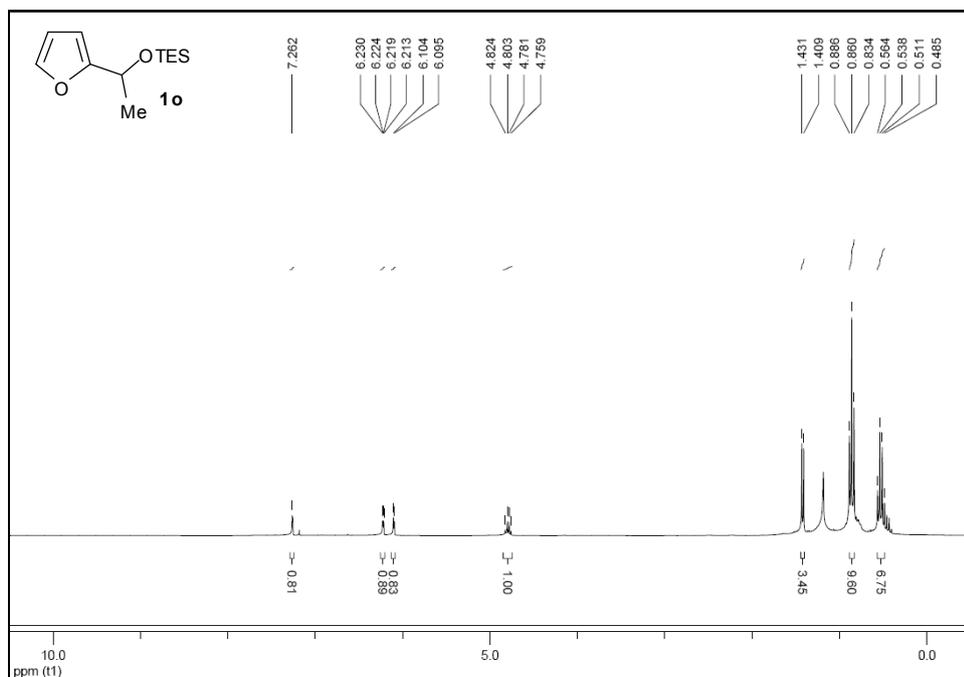
**Entry 14 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.59 (q,  $J = 7.8$  Hz, 6H), 0.94 (t,  $J = 7.8$  Hz, 9H), 1.45-1.61 (m, 6 H), 1.83 (q,  $J = 4.2$  Hz, 1H), 3.32-3.65 (m, 3H), 3.95-3.99 (m, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  4.43, 6.85, 23.25, 26.23, 28.49, 66.74, 68.49, 78.54.



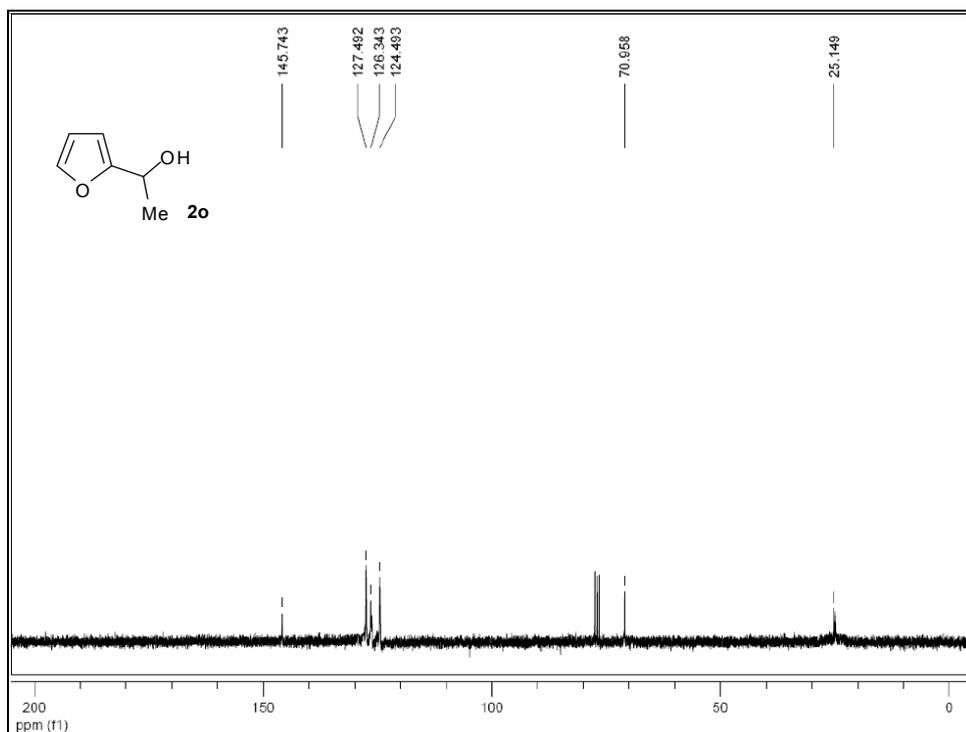
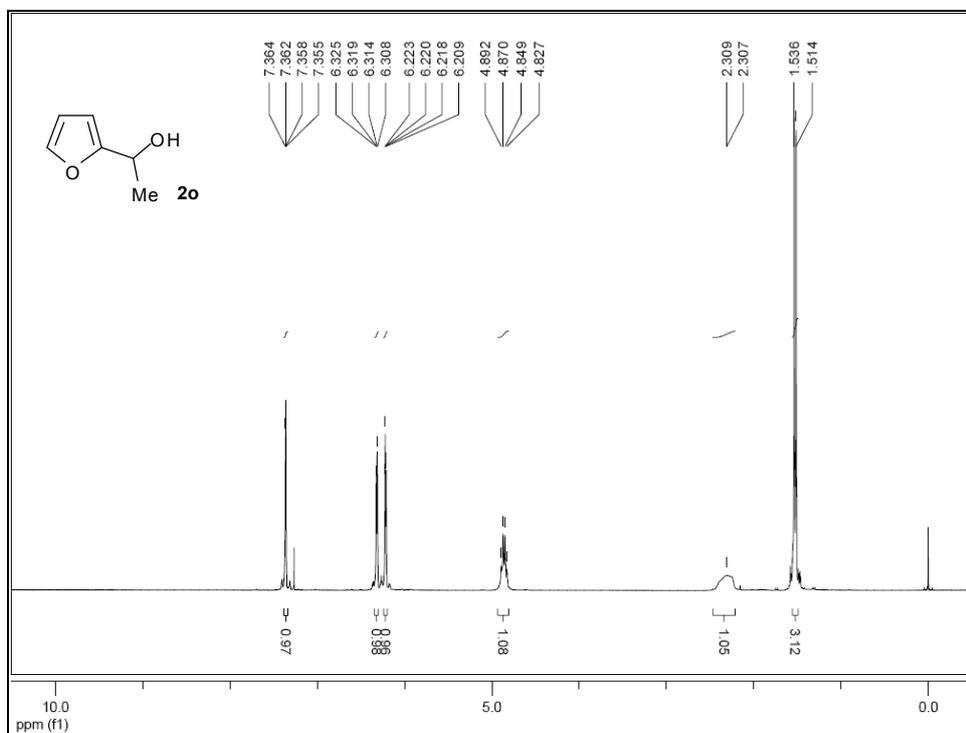
**Entry 14 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.12-1.17 (m, 1H), 1.30-1.43 (m, 4H), 1.67-1.69 (m, 1H), 2.8 (br s, 1H), 3.23-3.36 (m, 4H), 3.81-3.85 (m, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  23.04, 25.06, 27.51, 66.24, 68.36, 78.47.



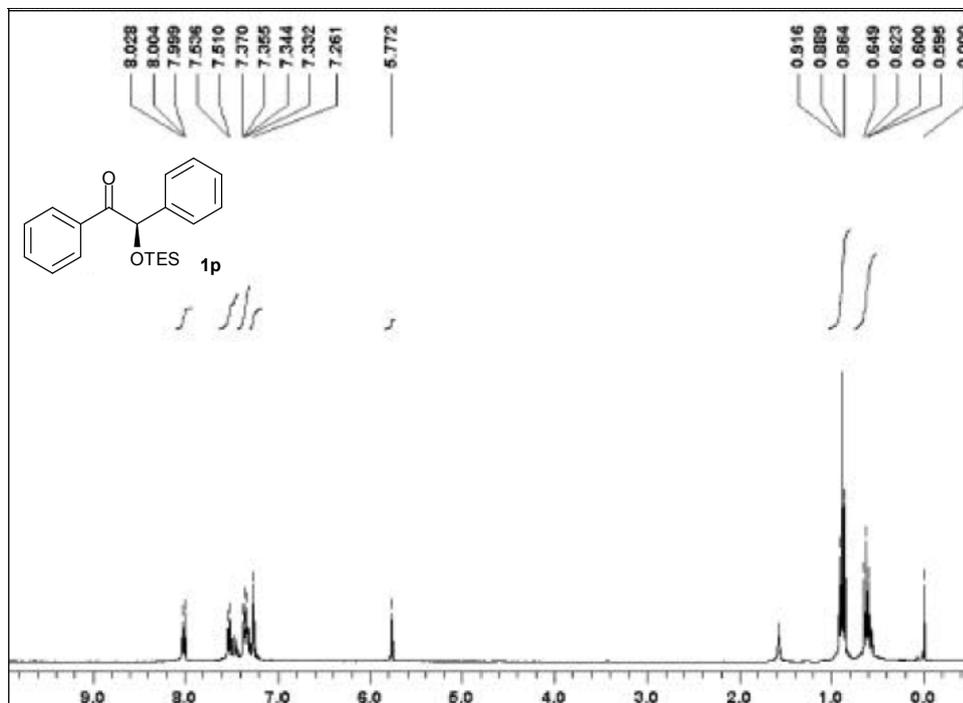
**Entry 15 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  = 0.52 (q,  $J$  = 7.8 Hz, 6H), 0.86 (t,  $J$  = 7.8 Hz, 9H), 1.42 (d,  $J$  = 6.3 Hz, 3H), 4.79 (q,  $J$  = 6.6 Hz, 1H), 6.10 (d,  $J$  = 3.0 Hz, 1H), 6.22 (q,  $J$  = 1.2 Hz, 1H), 7.26 (d,  $J$  = 1.5 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  4.79, 6.86, 23.04, 64.23, 104.92, 110.09, 141.42, 158.27.



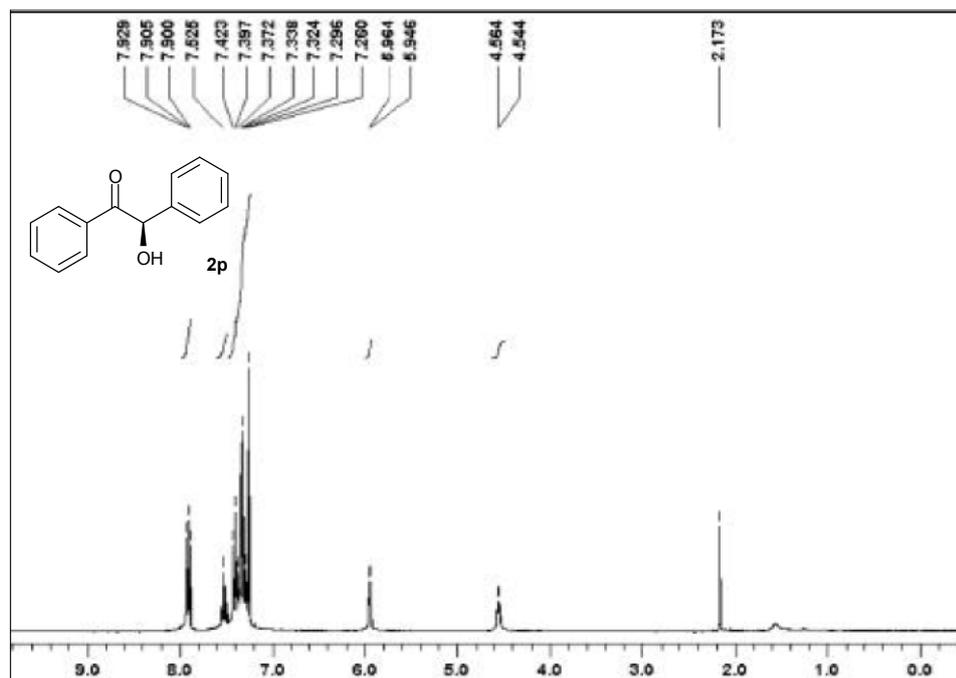
**Entry 15 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.52 (d,  $J = 6.6$  Hz, 3H), 2.31 (br s, 1H), 4.86 (q,  $J = 6.6$  Hz, 1H), 7.35-7.36 (m, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  25.15, 70.96, 124.49, 126.34, 127.49, 145.74.



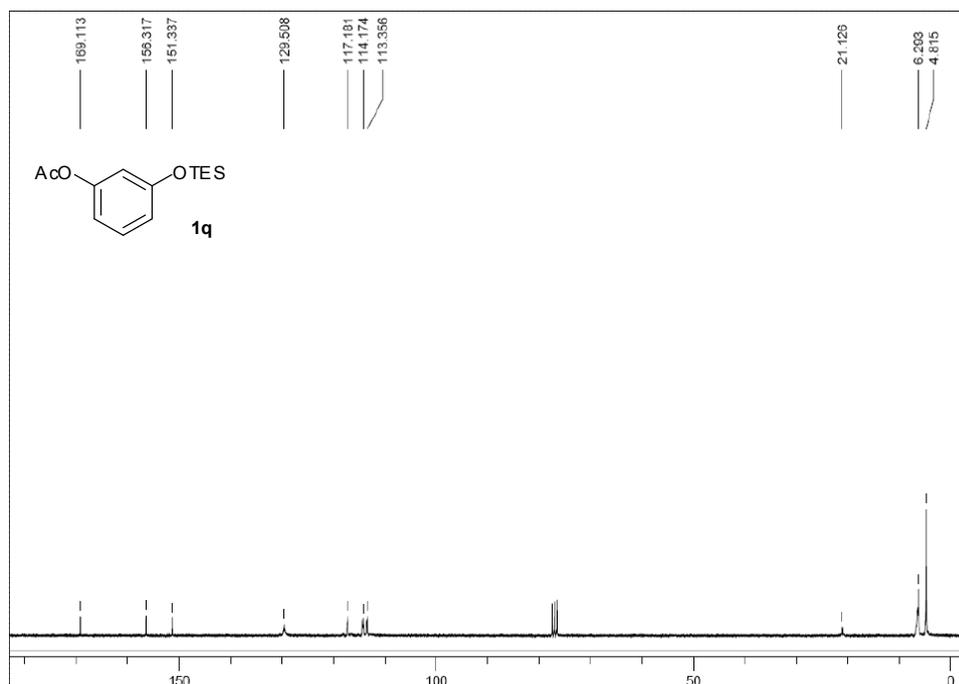
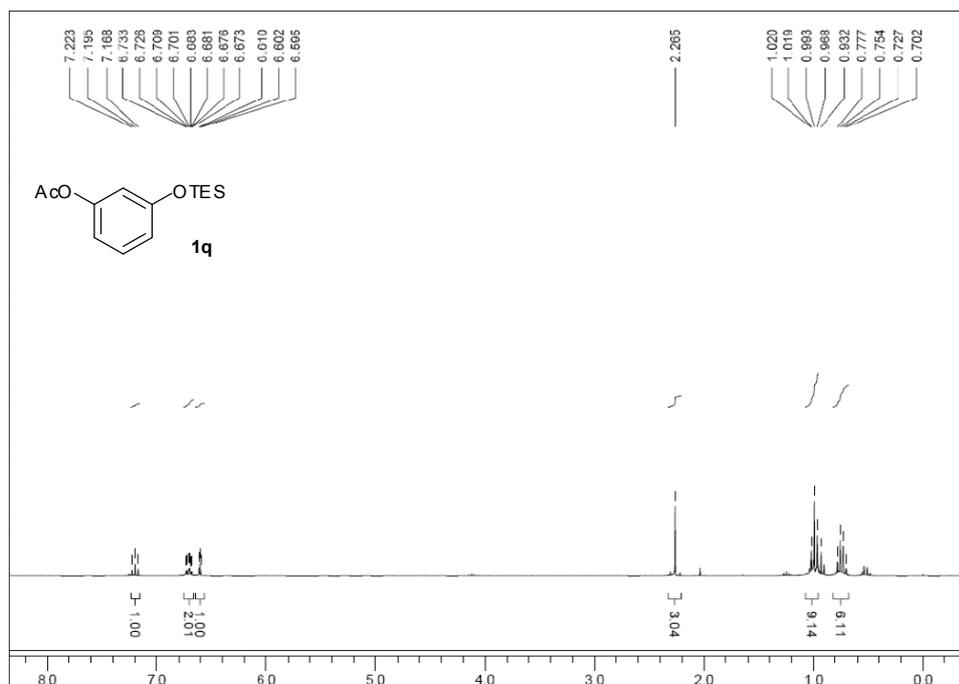
**Entry 16 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.57-0.65 (m, 6H), 0.89 (t,  $J = 7.8$  Hz, 9H), 5.77 (s, 1H), 7.22-7.27 (m, 1H), 7.31-7.37 (m, 4H), 7.43-7.54 (m, 3H), 8.00-8.03 (m, 2H).



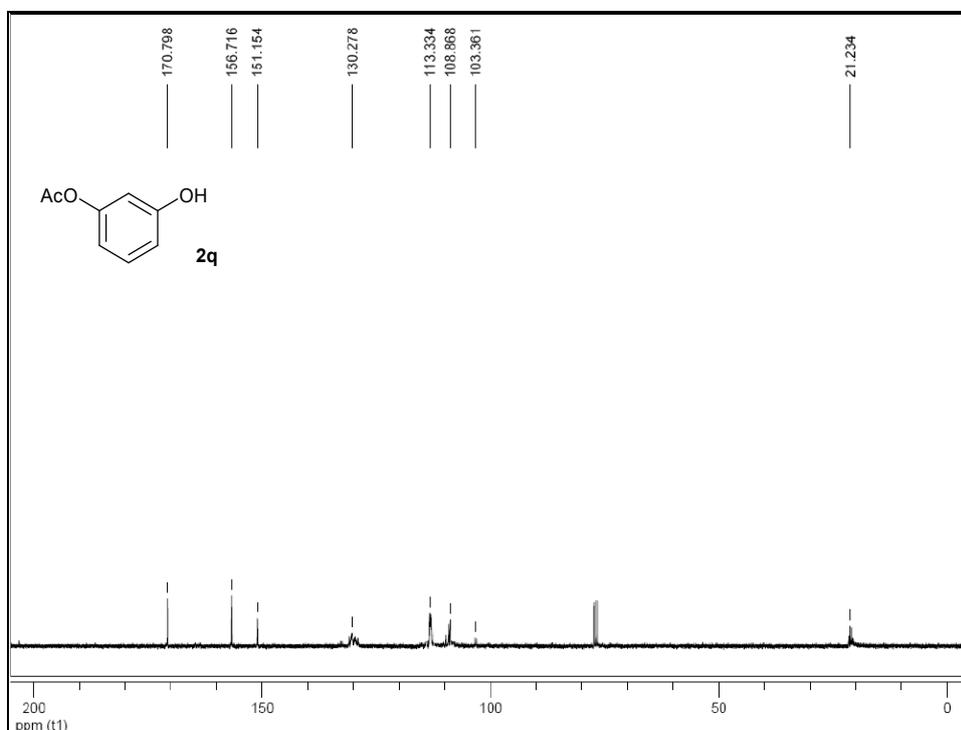
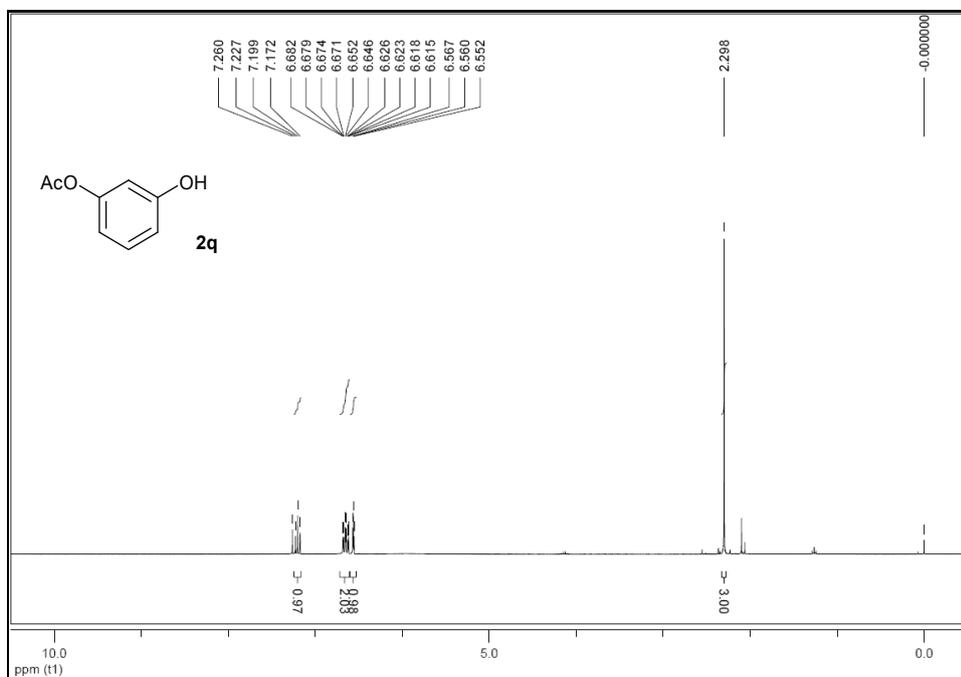
**Entry 16 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  4.55 (br s, 1H), 5.95 (s, 1H), 7.26-7.42 (m, 8H), 7.90-7.93 (m, 2H)



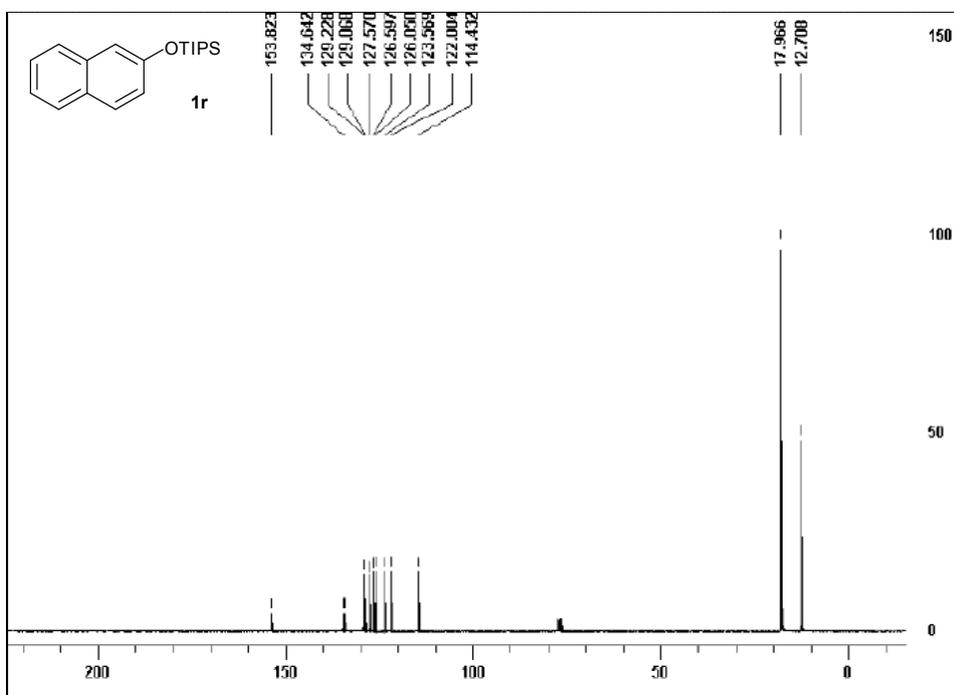
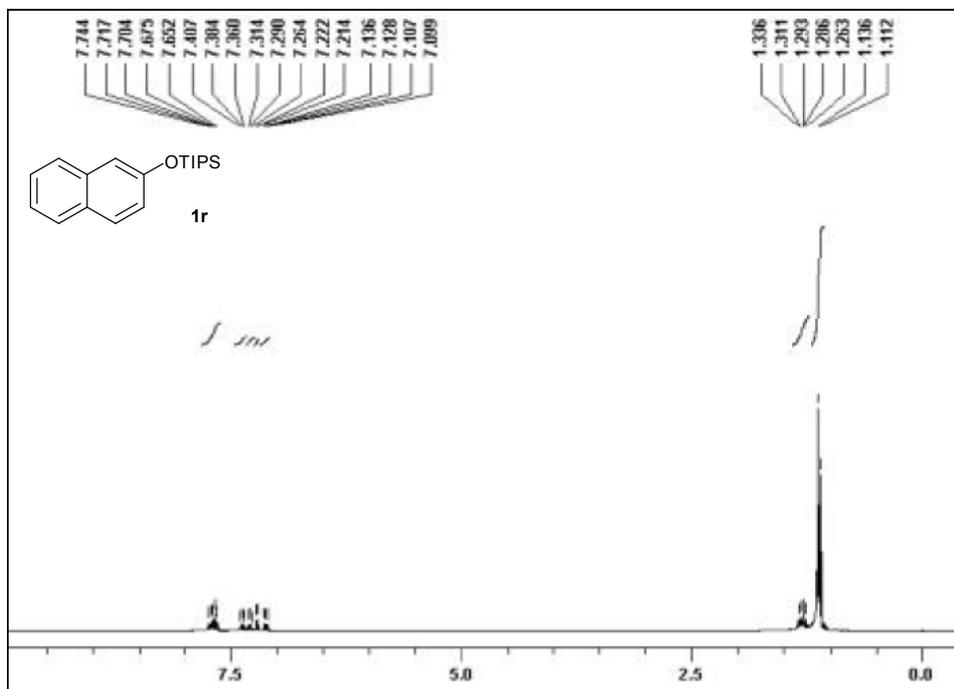
**Entry 17 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.74 (q,  $J = 8.1$  Hz, 6H), 1.02 (t,  $J = 7.8$  Hz, 9H), 2.27 (s, 3H), 6.60 (t,  $J = 2.1$  Hz, 1H), 6.66-6.73 (m, 2H), 7.20 (t,  $J = 8.10$  Hz, 1H) ;  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  4.82, 6.29, 21.13, 113.36, 114.17, 117.18, 129.51, 151.34, 156.32, 169.11.



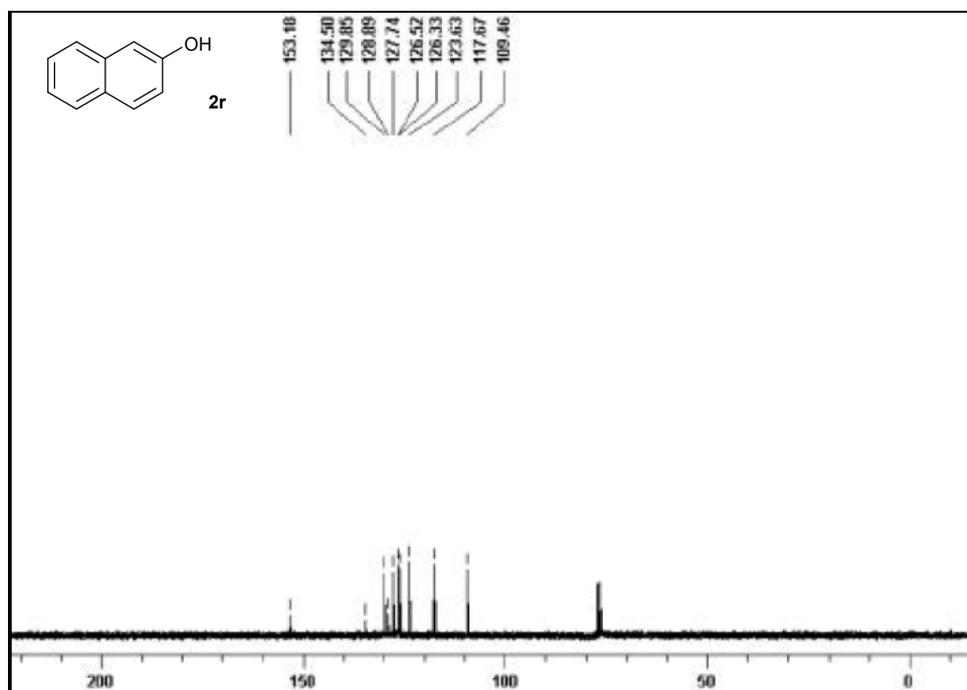
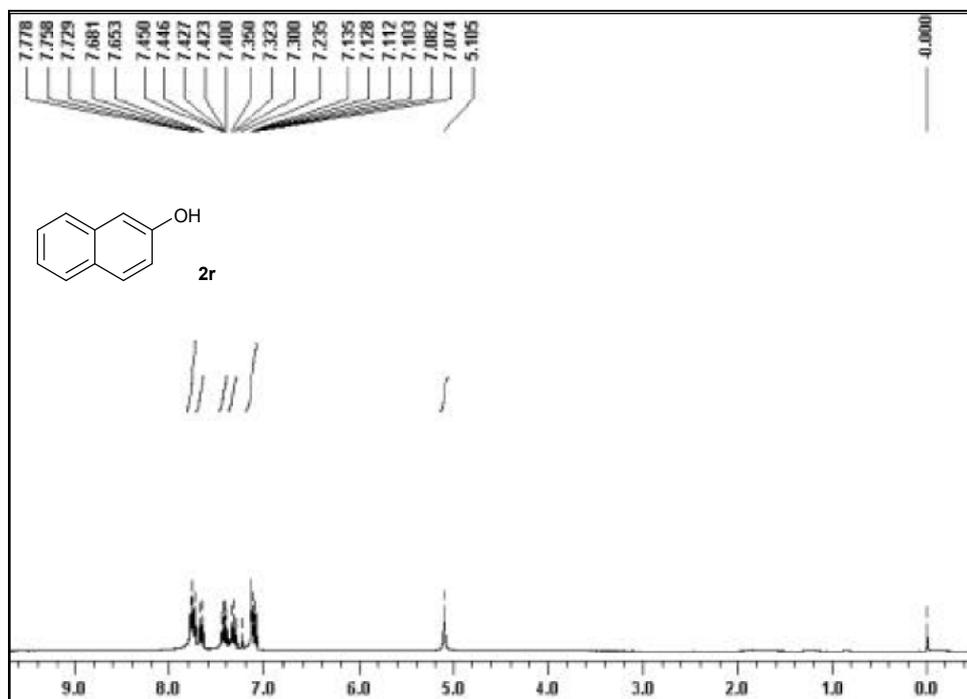
**Entry 17 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  2.30 (s, 1H), 6.56 (t,  $J = 2.4$  Hz, 1H), 6.62-6.68 (m, 2H), 7.20 (d,  $J = 8.4$  Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  21.23, 103.36, 108.87, 113.33, 130.28, 151.15, 156.72, 170.80.



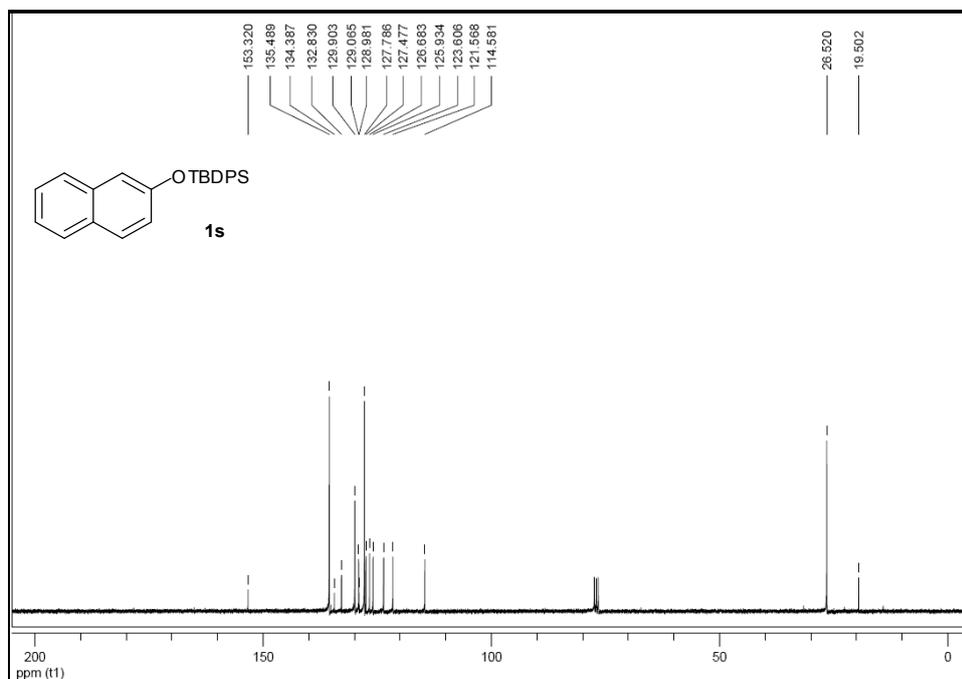
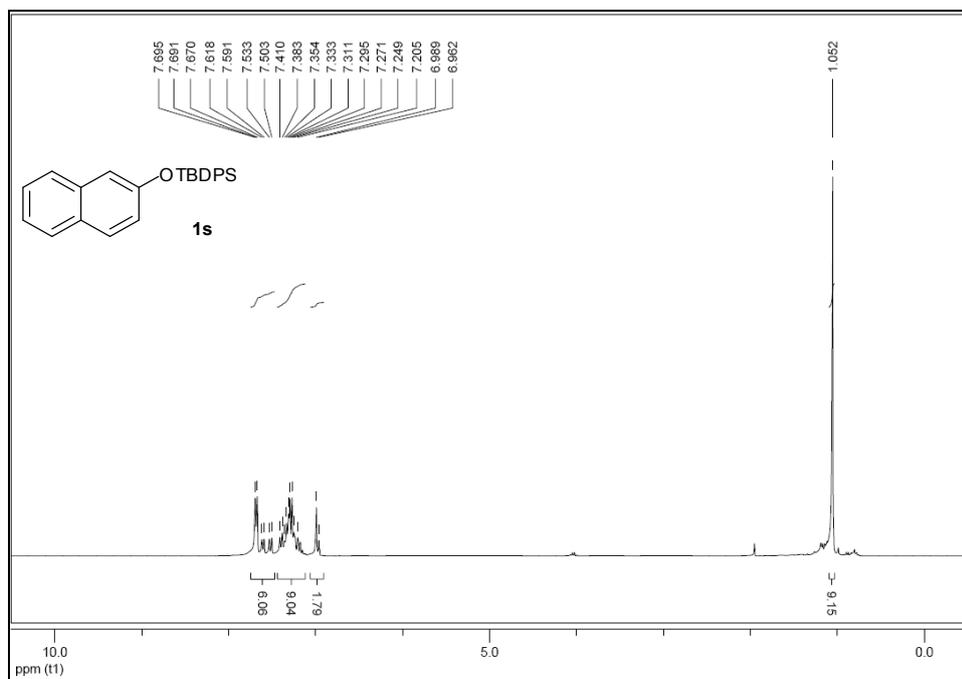
**Entry 18 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.11-1.36 (m, 18H), 1.24-1.36 (m, 3H), 7.12-7.74 (m, 7H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  12.71, 17.97, 114.43, 122.00, 123.57, 126.05, 126.60, 127.57, 129.07, 129.23, 134.64, 153.82.



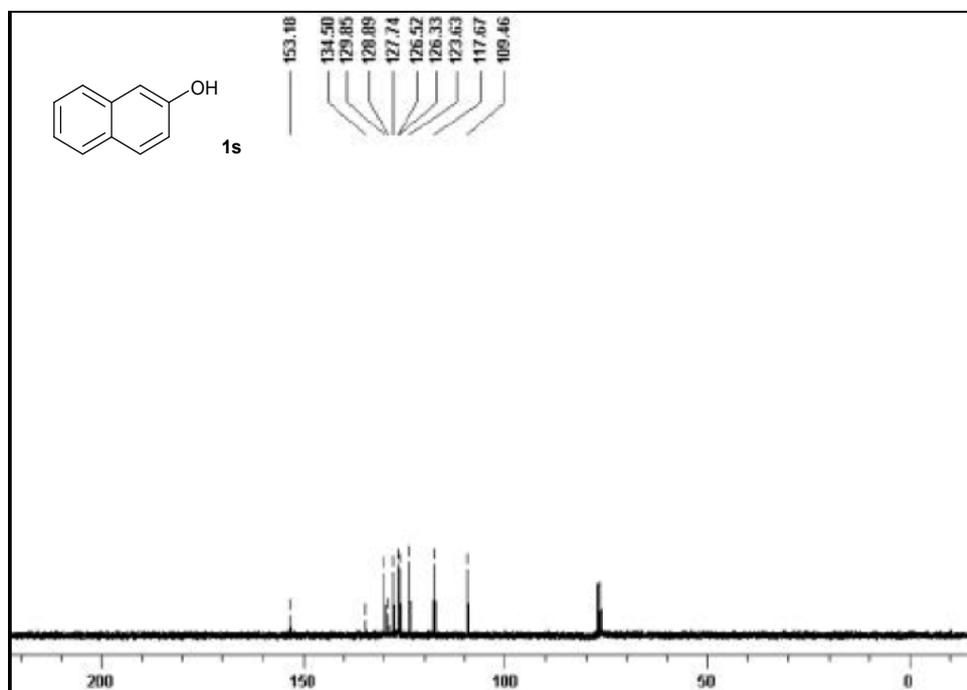
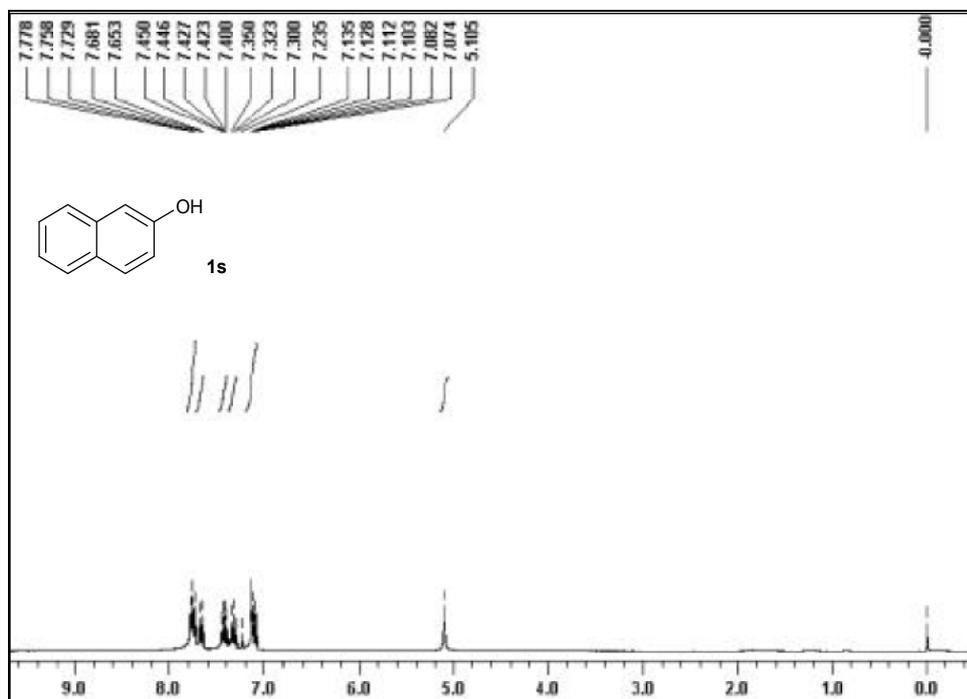
**Entry 18 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  5.11 (br s, 1H), 7.07-7.78 (m, 7H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  109.46, 117.67, 123.63, 126.33, 126.52, 127.74, 128.90, 129.85, 134.50, 153.18.



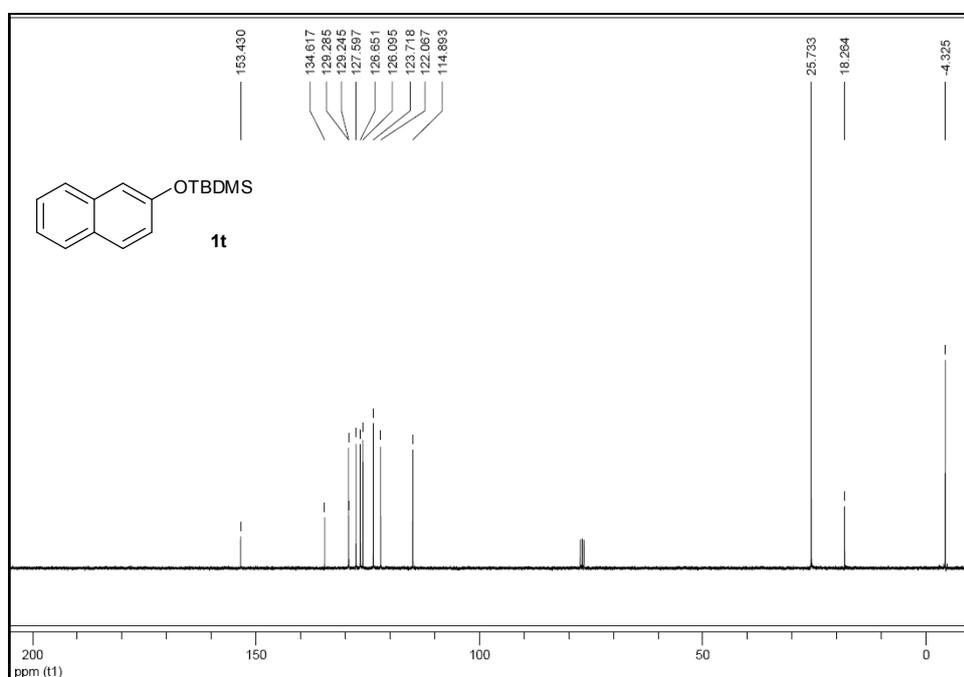
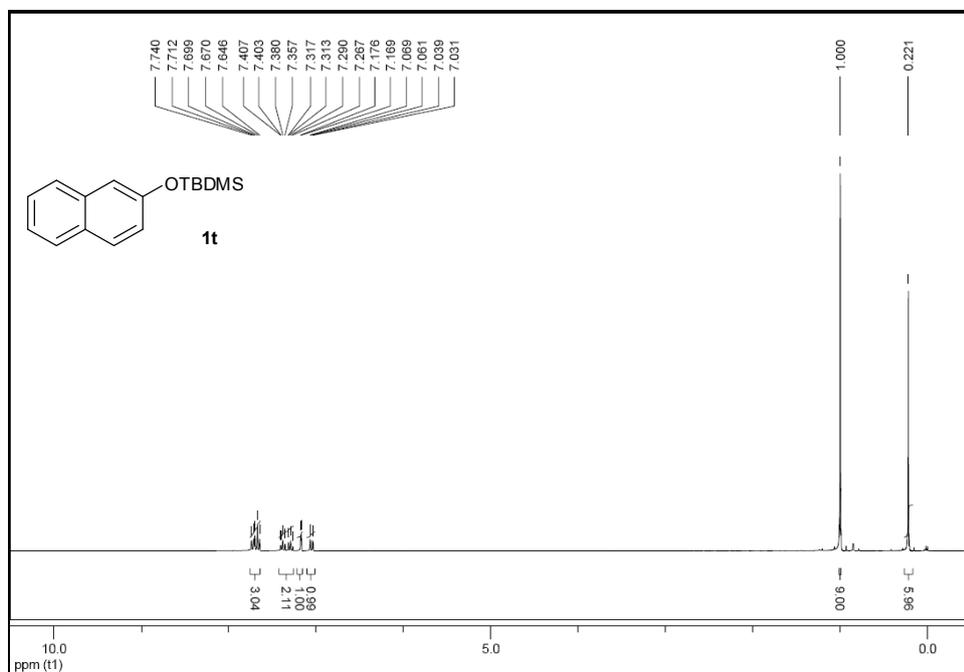
**Entry 19 S(substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  = 1.05 (s, 9H), 6.96-6.99 (m, 2H), 7.21-7.41 (m, 9H), 7.50-7.77 (m, 6H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  = 19.50, 26.52, 114.58, 121.57, 123.61, 125.93, 126.68, 127.48, 127.79, 128.98, 129.07, 129.90, 132.83, 134.39, 135.49, 153.32.



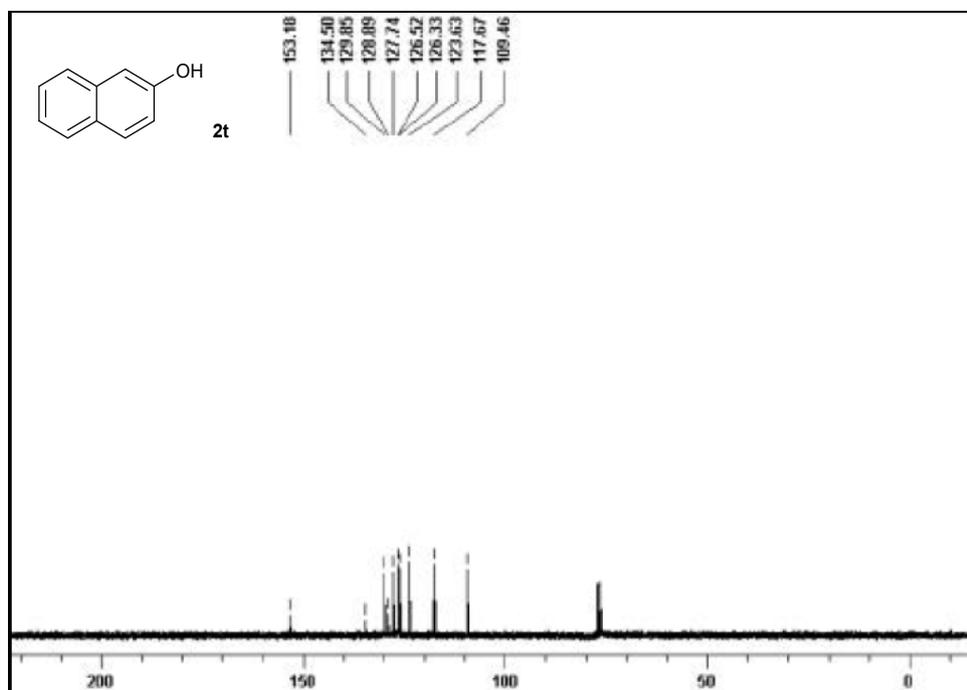
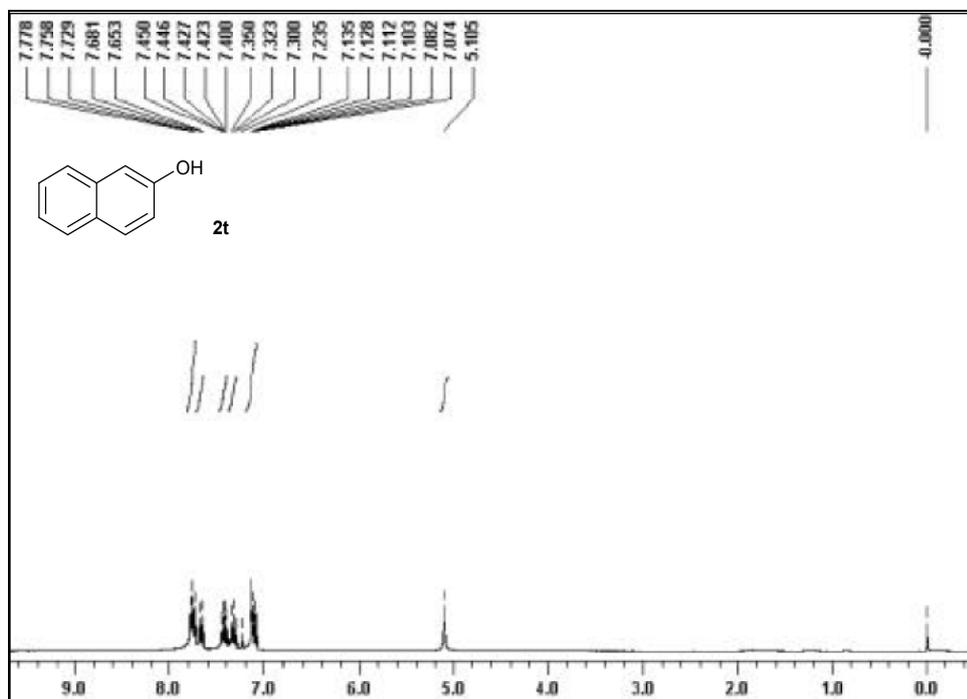
**Entry 19 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  5.11 (br s, 1H), 7.07-7.78 (m, 7H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  109.46, 117.67, 123.63, 126.33, 126.52, 127.74, 128.90, 129.85, 134.50, 153.18.



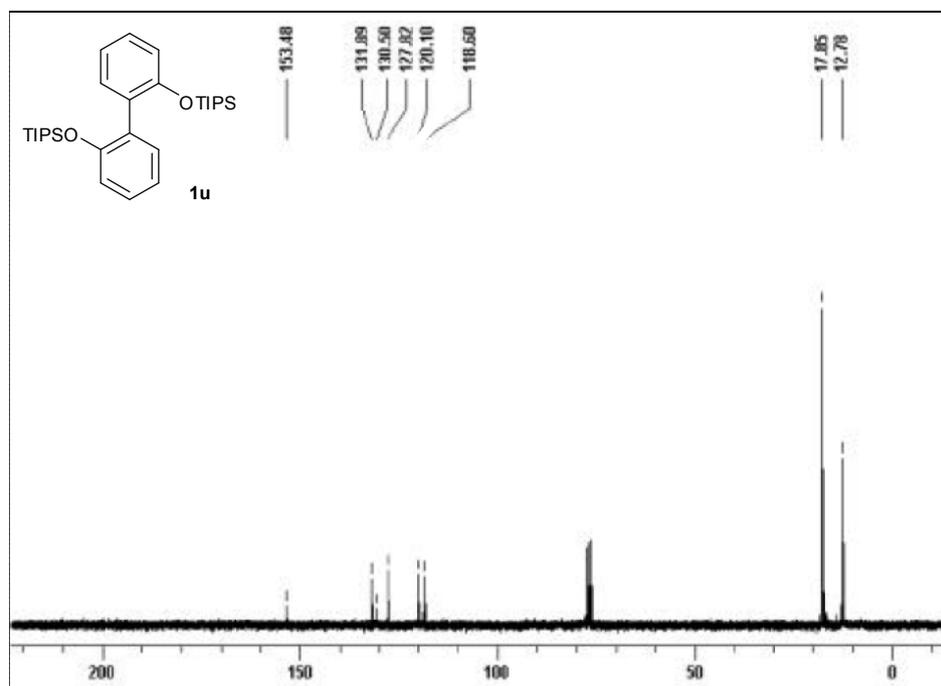
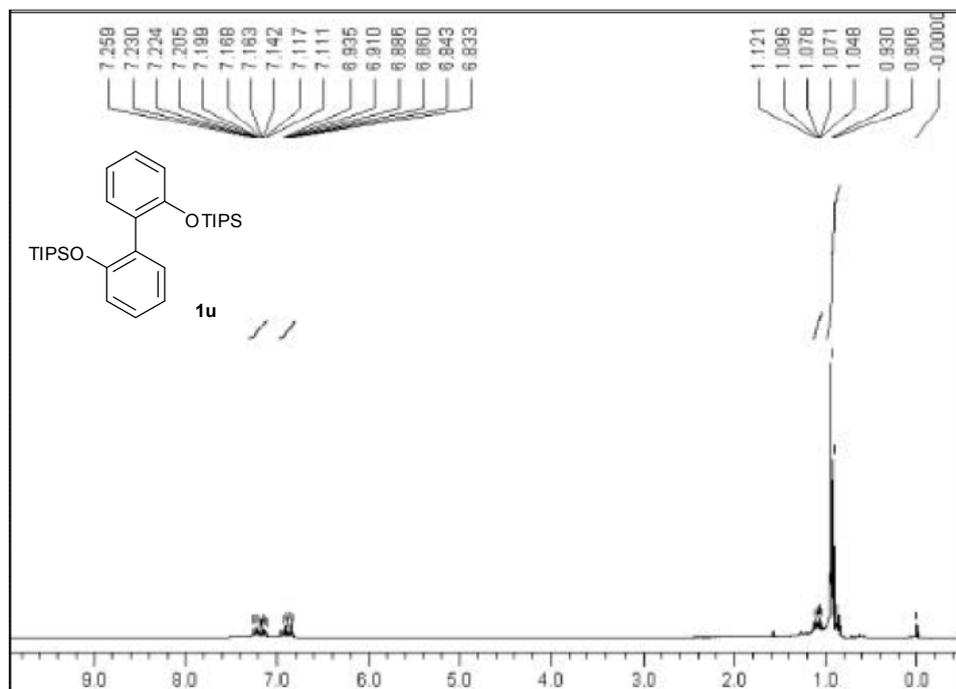
**Entry 20 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.22 (s, 6H), 1.00 (s, 9H), 7.05-7.74 (m, 7H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  -4.33, 18.26, 25.73, 114.89, 122.07, 123.72, 126.10, 126.65, 127.60, 129.25, 129.29, 134.62, 153.43.



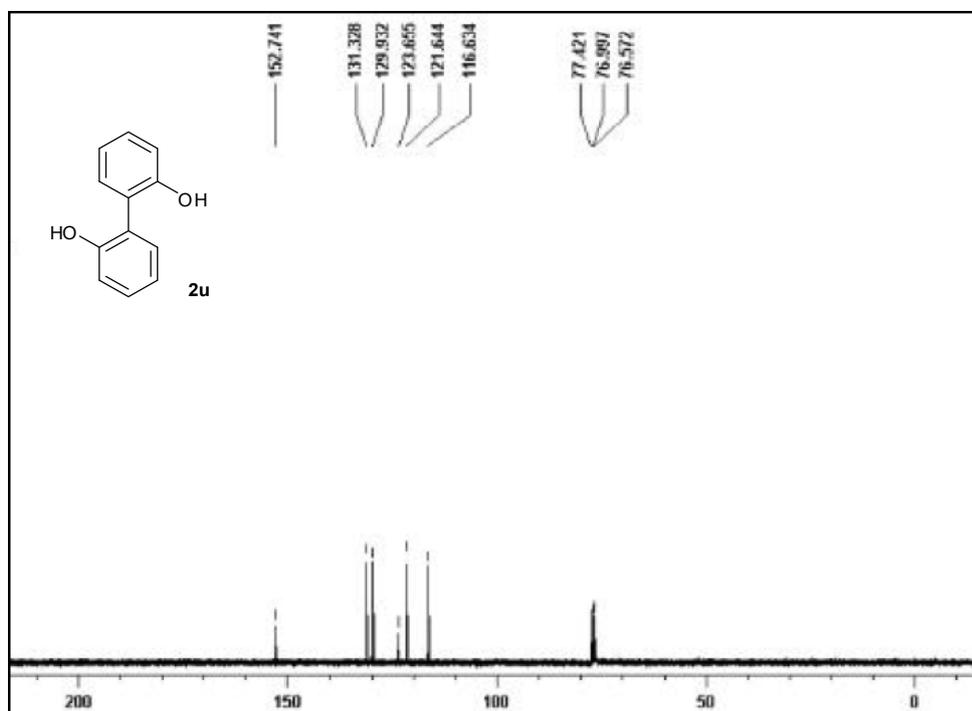
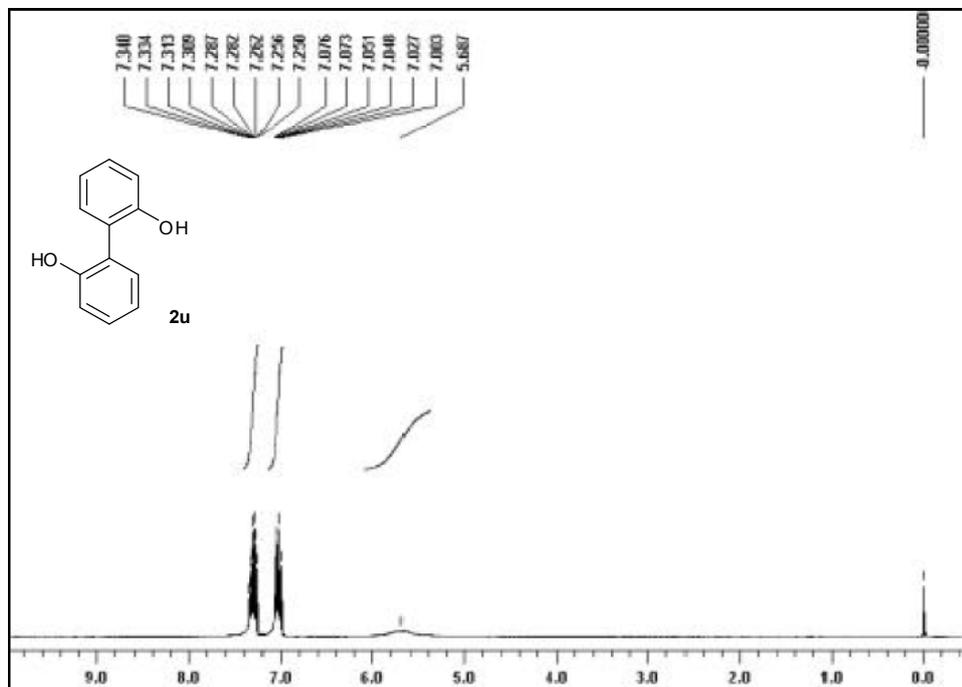
**Entry 20 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  5.11 (br s, 1H), 7.07-7.78 (m, 7H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  109.46, 117.67, 123.63, 126.33, 126.52, 127.74, 128.90, 129.85, 134.50, 153.18.



**Entry 21 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.86-0.93 (m, 18H), 1.05-1.12 (m, 3H), 6.83-8.94 (m, 2H), 7.11-7.26 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  12.78, 17.85, 118.60, 120.10, 127.82, 130.50, 131.89, 153.48.

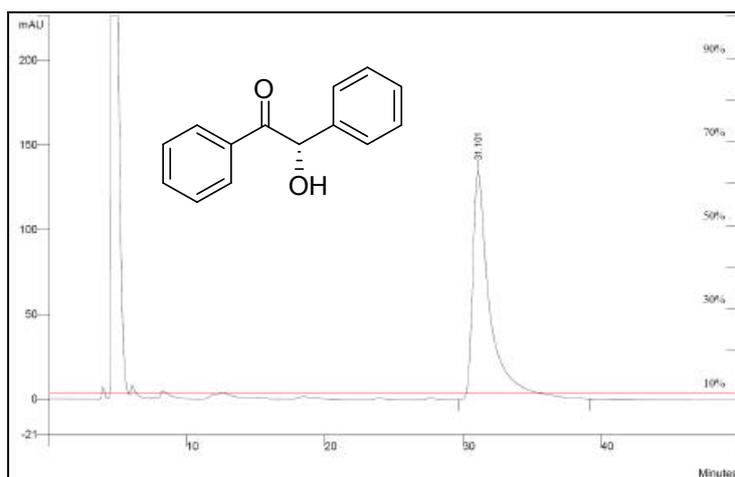
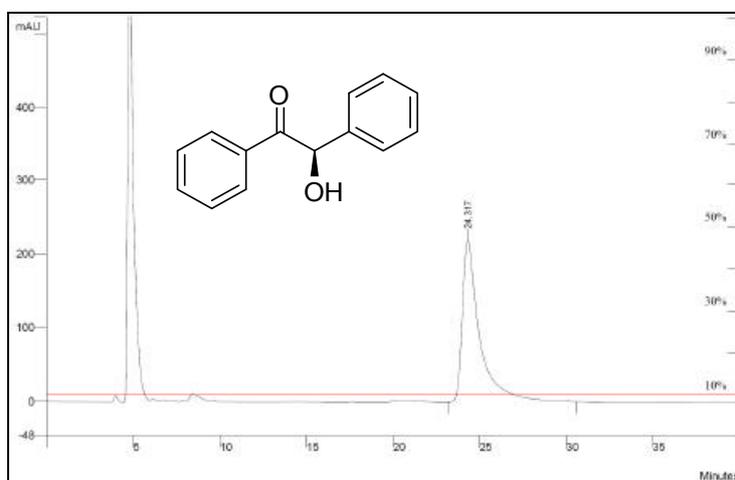


**Entry 21 (Product):**  $^1\text{H}$  NMR (500 MHz;  $\text{CDCl}_3$ )  $\delta$  5.85 (br s, 2H), 7.03-7.35 (m, 8H);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  116.7, 121.7, 123.7, 130.0, 131.4, 152.8.

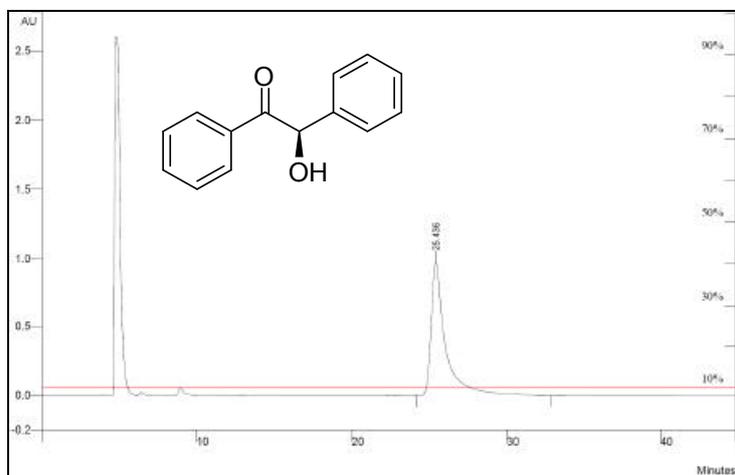


#### 4. HPLC Spectrum for Table 1 (Entry 16)

The ee value of benzoin was determined by HPLC analysis: CHIRALPAK AD-H, Hexane: IPA = 90:10, 0.75 mL/min, UV detection at 210 nm,  $t_R = 24.32$  min,  $t_S = 31.01$  min.

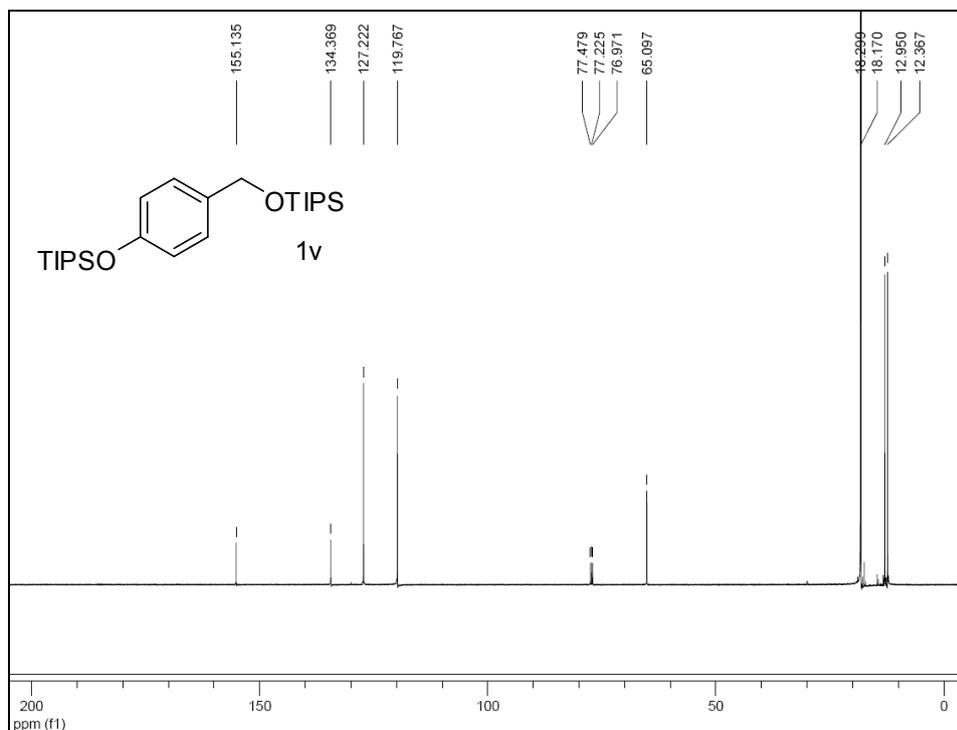
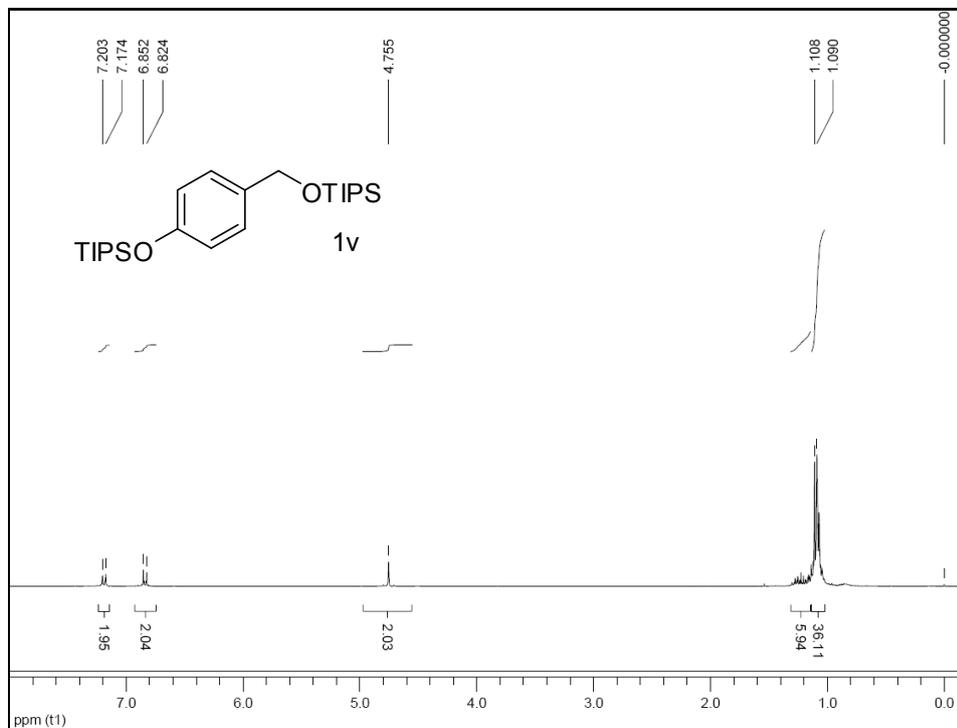


HPLC Spectrum for Table 1, Entry 16

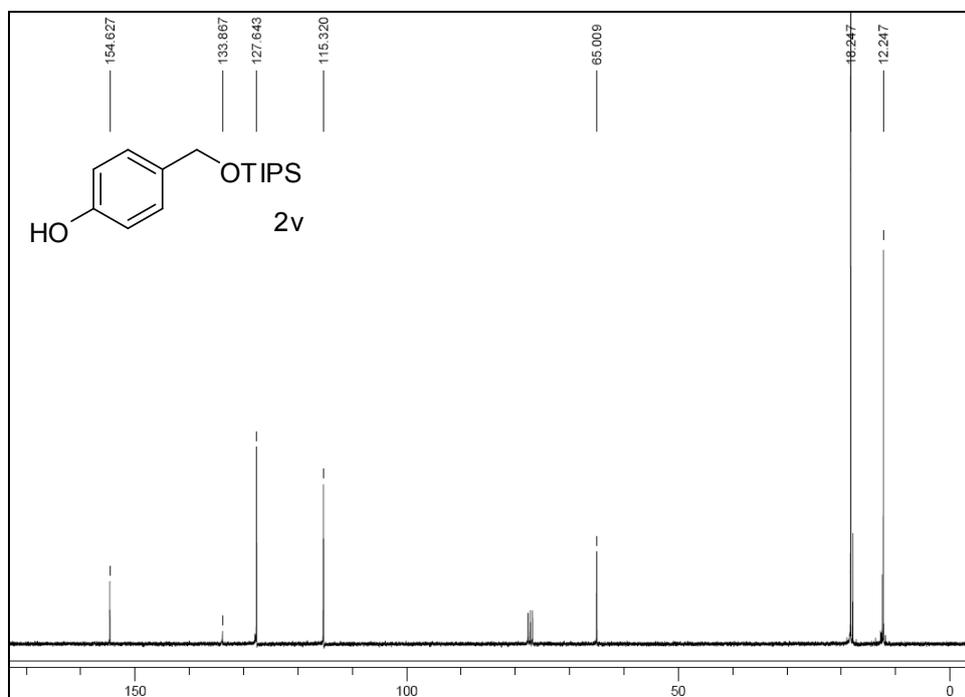
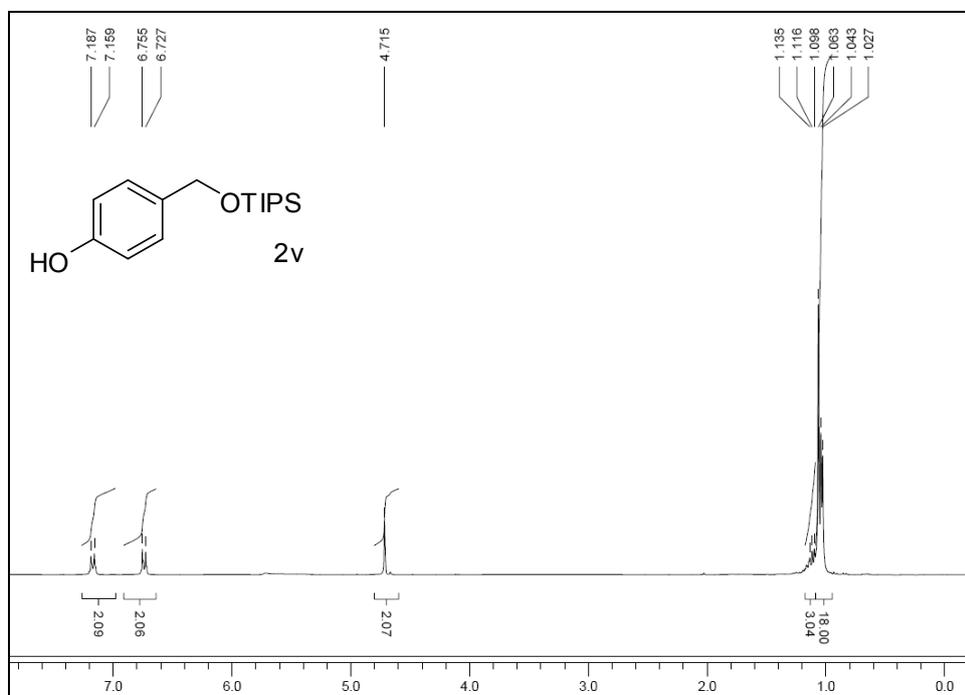


## 5. NMR Data for Table 2.

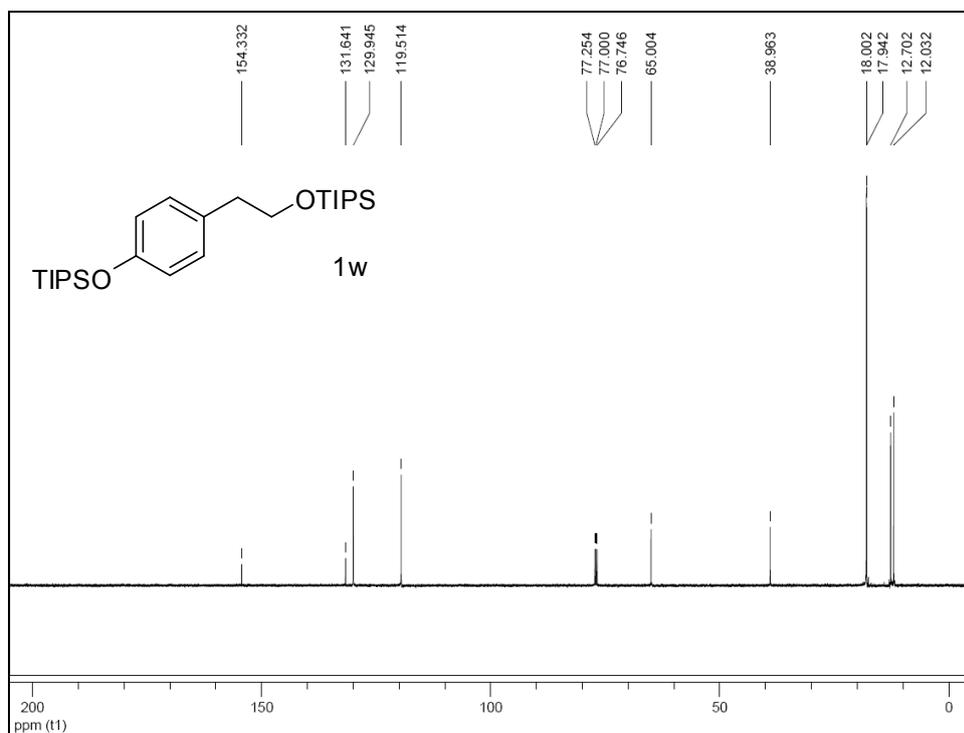
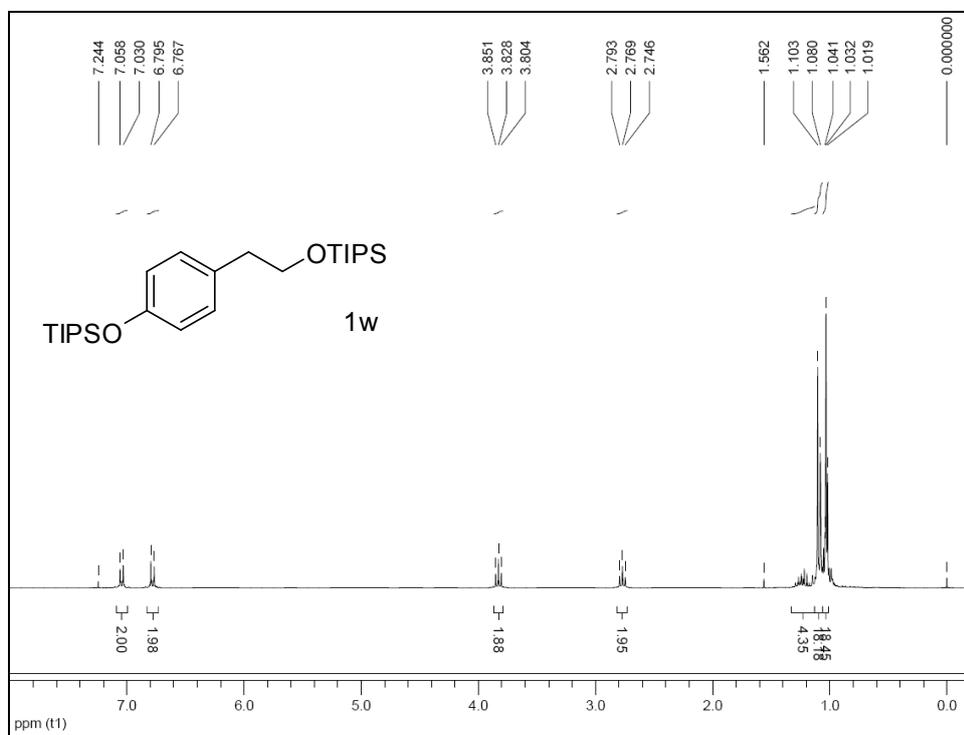
**Entry 1 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  = 1.06-1.21 (m, 42H), 4.75 (s, 2H), 6.84 (d,  $J$  = 8.4 Hz, 2H), 7.19 (d,  $J$  = 8.4 Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  = 12.37, 12.95, 18.17, 18.30, 65.10, 119.77, 127.22, 134.37, 155.13.



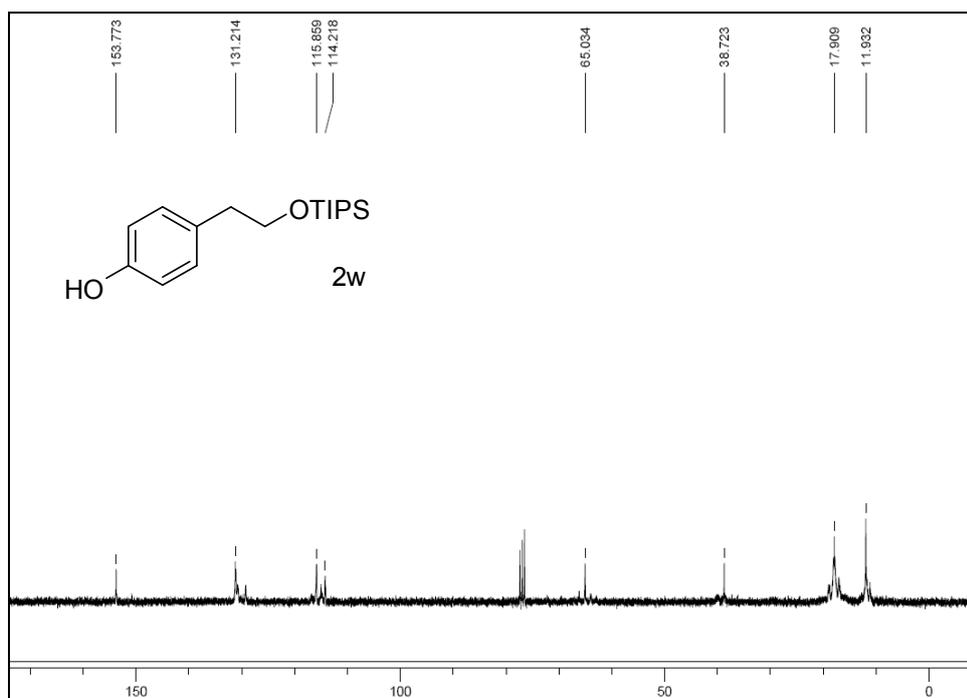
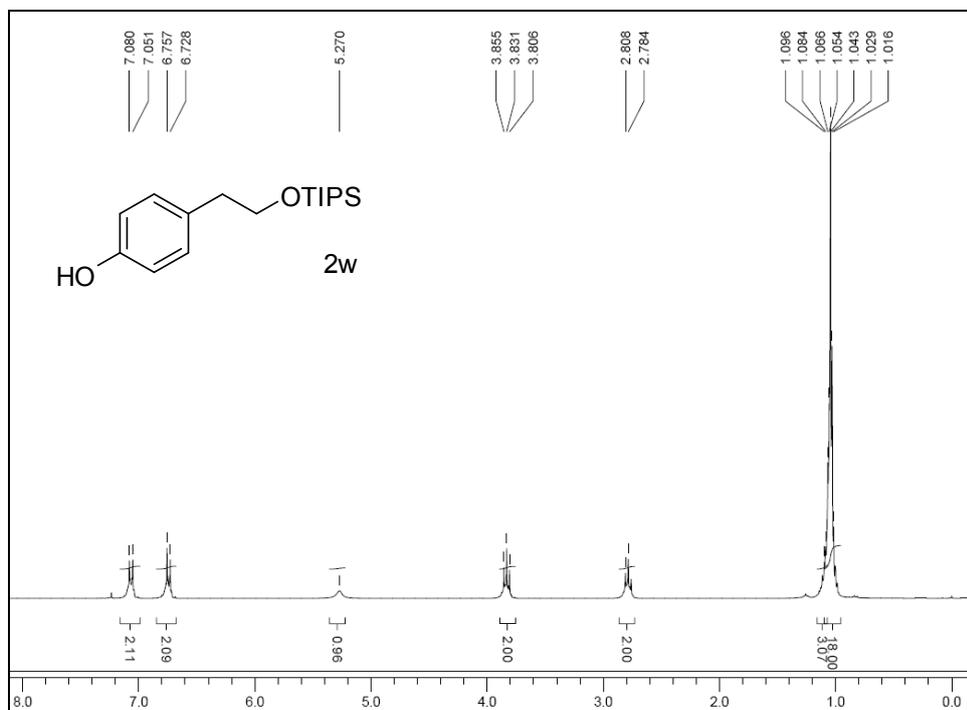
**Entry 1 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.04 (d,  $J = 4.8$  Hz, 18H), 1.08-1.14 (m, 3H), 4.75 (s, 2H), 6.74 (d,  $J = 8.4$  Hz, 2H), 7.17(d,  $J = 8.4$  Hz, 2H)  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  12.25, 18.25, 65.01, 115.32, 127.64, 133.87, 154.63.



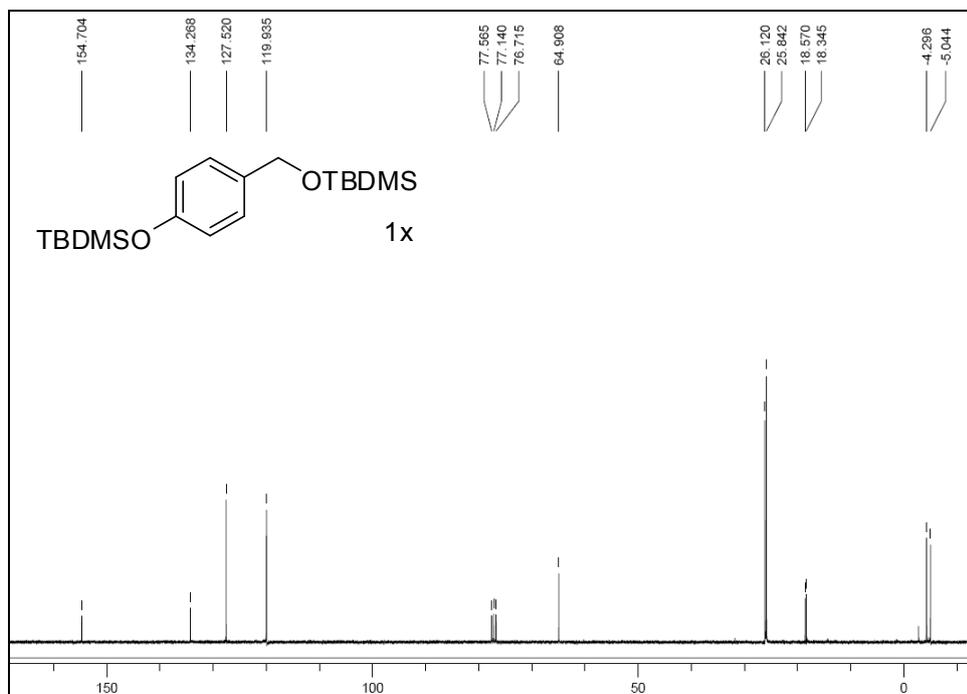
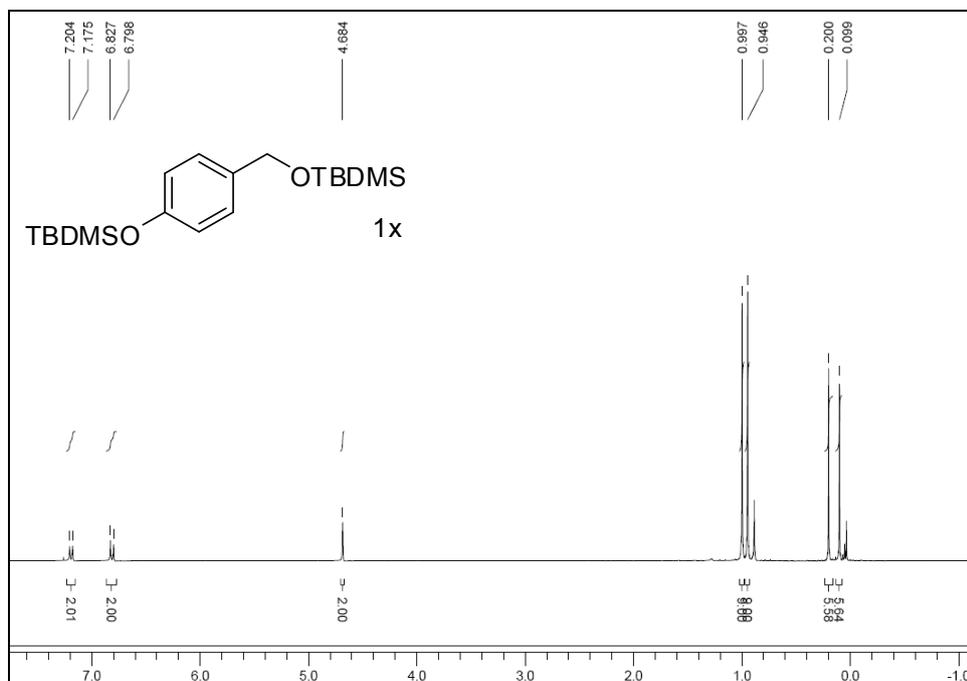
**Entry 2 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.99-1.32 (m, 42H), 2.77 (t,  $J = 7.2$  Hz, 2H), 3.83 (t,  $J = 7.2$  Hz, 2H), 6.78 (d,  $J = 8.4$  Hz, 2H), 7.04 (d,  $J = 8.4$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  11.89, 12.56, 17.80, 17.86, 38.82, 64.86, 119.37, 129.80, 131.50.



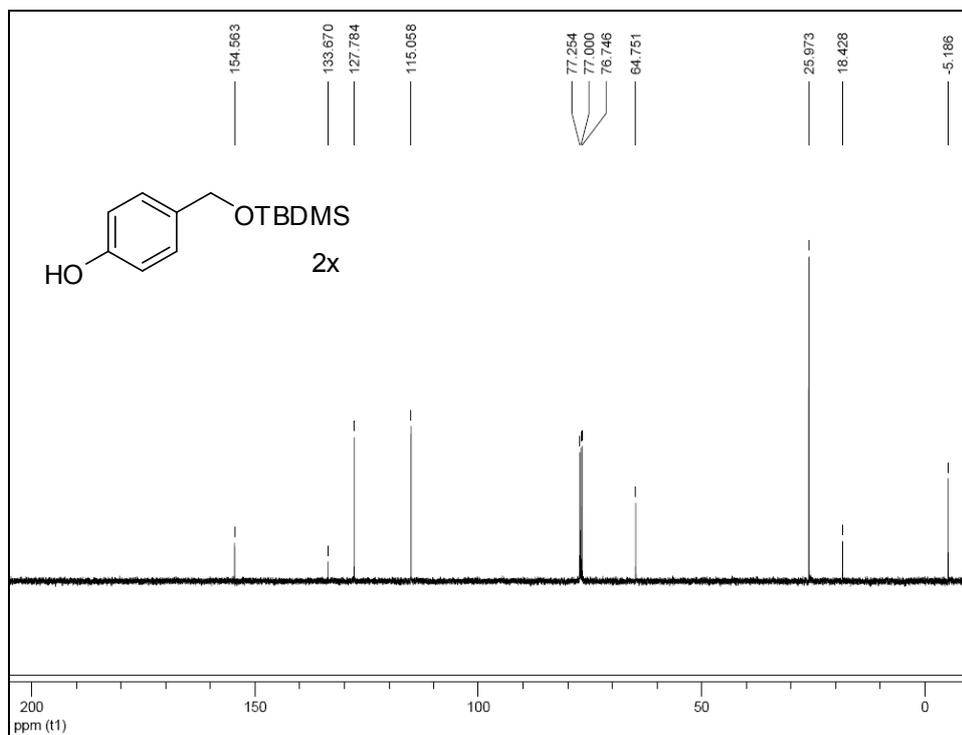
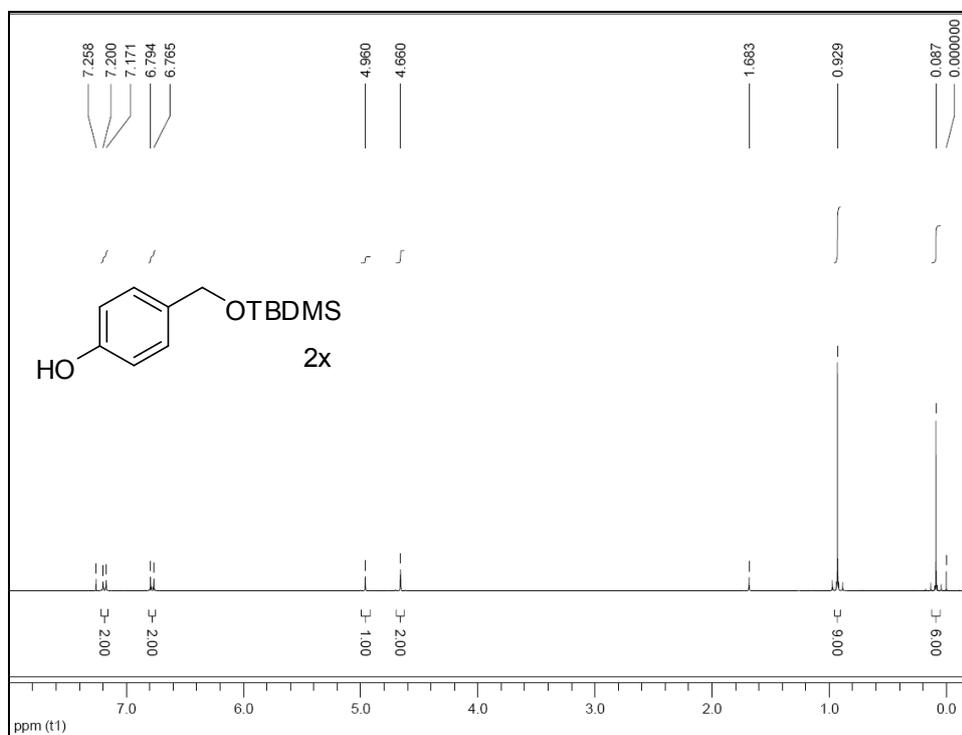
**Entry 2 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.03 (d,  $J = 3.9$  Hz, 18H), 1.05-1.09 (m, 3H), 2.81 (t,  $J = 7.2$  Hz, 2H), 3.83 (t,  $J = 7.2$  Hz, 2H), 5.27 (s, 1H), 6.74 (d,  $J = 8.7$  Hz, 2H), 7.07 (d,  $J = 8.7$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  11.93, 17.91, 38.72, 65.03, 114.21, 115.86, 131.21, 153.77.



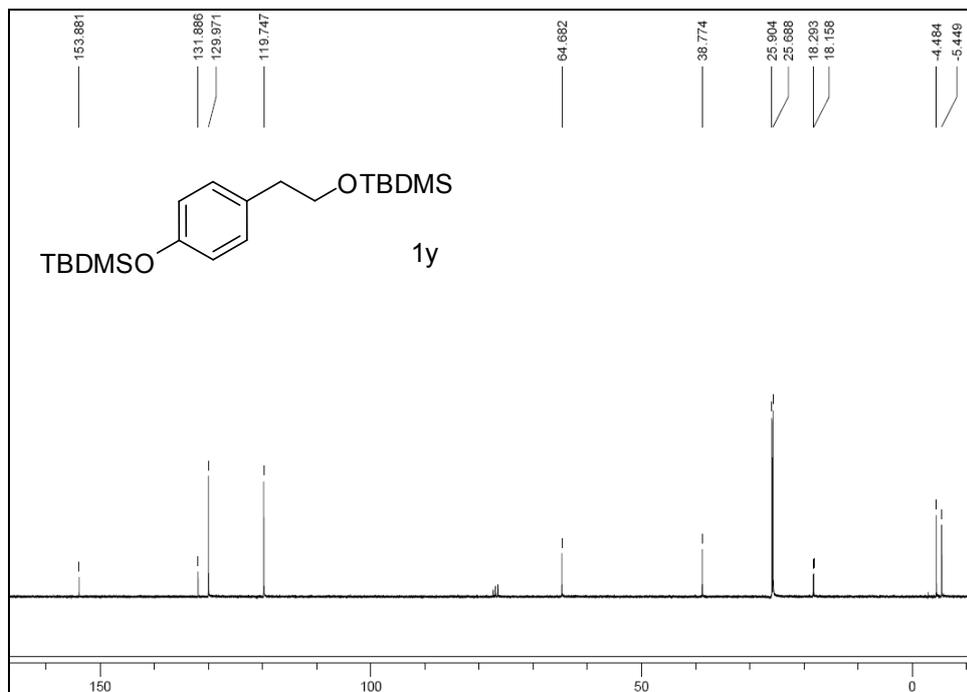
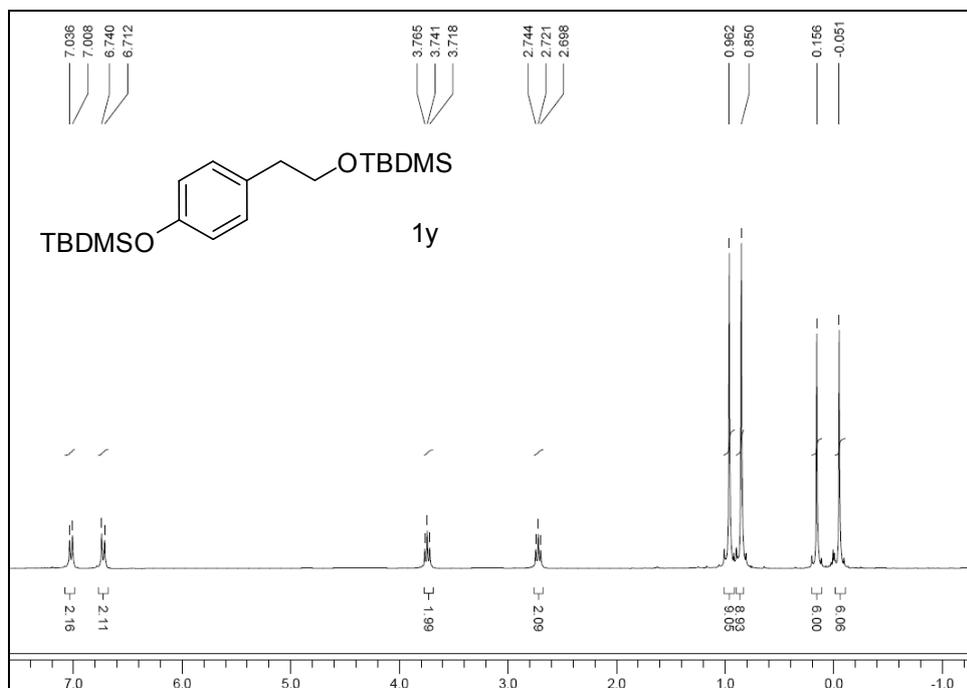
**Entry 3 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.09 (s, 6H), 0.20 (s, 6H), 0.95 (s, 9H), 0.99 (s, 9H), 4.68 (s, 2H), 6.81 (d,  $J = 8.7$  Hz, 2H), 7.19(d,  $J = 8.7$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  -5.04, -4.29, 18.34, 18.57, 25.84, 26.12, 64.91, 119.94, 127.52, 134.27, 154.70.



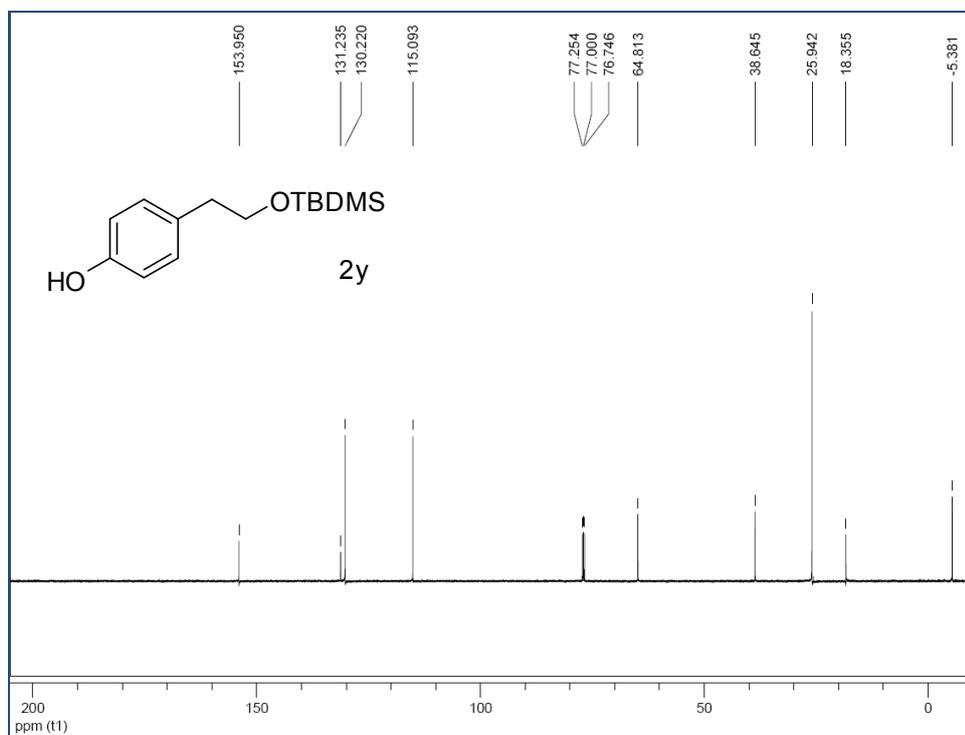
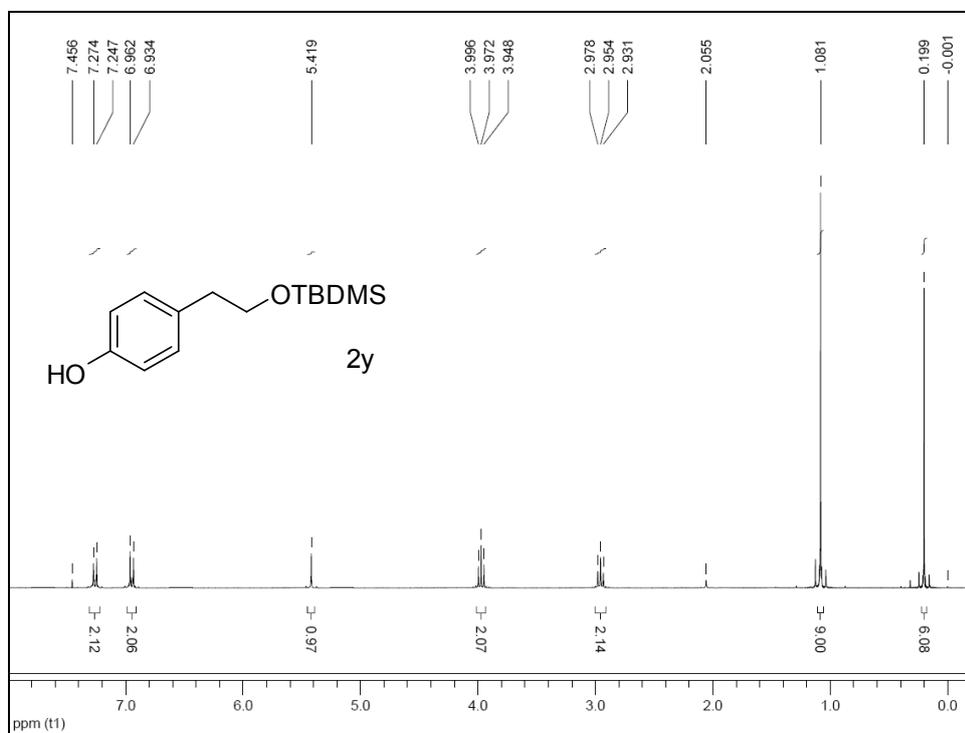
**Entry 3 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.09 (s, 6H), 0.93 (s, 9H), 4.66 (s, 2H), 4.96 (s, 1H), 6.78 (d,  $J = 8.7$  Hz, 2H), 7.19 (d,  $J = 8.7$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  -5.19, 18.43, 25.97, 64.75, 115.06, 127.78, 133.67, 154.56.



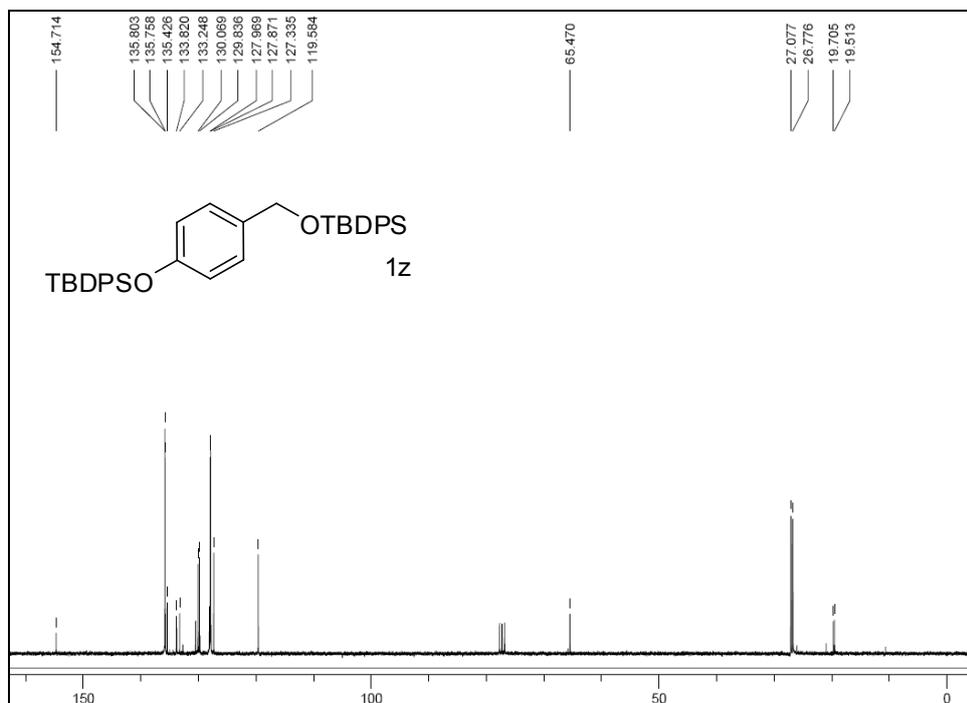
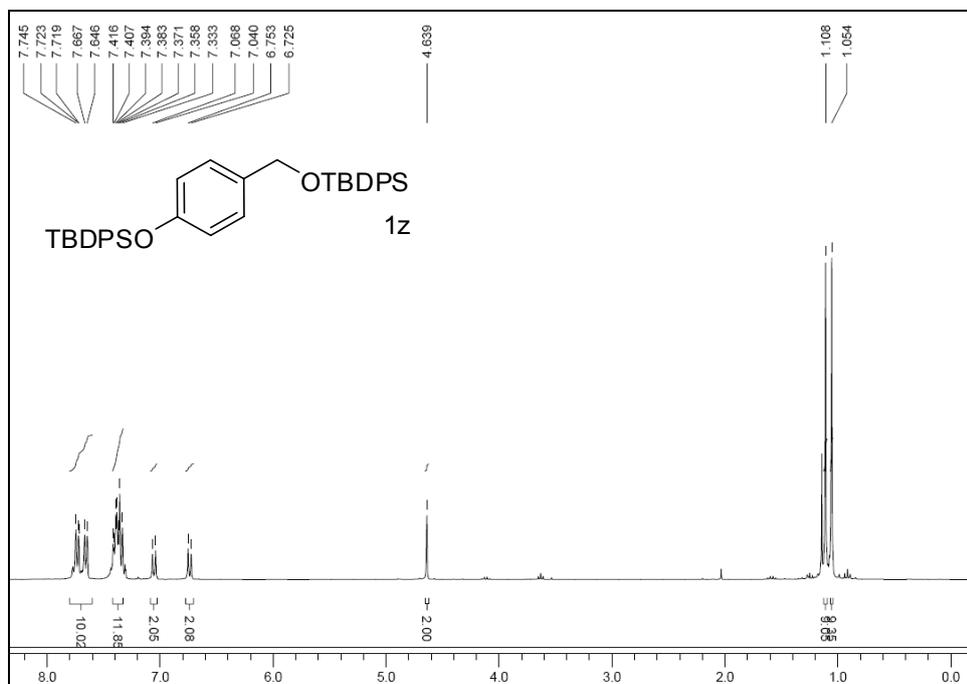
**Entry 4 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  -0.05 (s, 6H), 0.16 (s, 6H), 0.85 (s, 9H), 0.96 (s, 9H), 2.72 (t,  $J = 6.9$  Hz, 2H), 3.74 (t,  $J = 6.9$  Hz, 2H), 6.73 (d,  $J = 8.4$  Hz, 2H), 7.02 (d,  $J = 8.4$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  -5.45, -4.48, 18.16, 18.29, 25.68, 25.90, 36.77, 64.68, 119.75, 129.97, 131.89, 153.88.



**Entry 4 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.20 (s, 6H), 1.08 (s, 9H), 2.95 (t,  $J = 7.2$  Hz, 2H), 3.97 (t,  $J = 7.2$  Hz, 2H), 5.42 (s, 1H), 6.95 (d,  $J = 8.7$  Hz, 2H), 7.26 (d,  $J = 8.4$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  -5.38, 18.35, 25.94, 38.64, 64.81, 115.09, 130.22, 131.23, 153.95.



**Entry 5 (Substrate):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.05 (s, 9H), 1.11 (s, 9H), 4.64 (s, 2H), 6.73 (d,  $J = 8.4$  Hz, 2H), 7.05(d,  $J = 8.4$  Hz, 2H), 7.33-7.42 (m, 10H), 7.65-7.75 (m, 10H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  19.51, 19.71, 26.78, 27.08, 65.47, 119.58, 127.34, 127.81, 127.97, 129.84, 130.07, 133.25, 133.82, 135.43, 135.76, 135.80, 154.71.



**Entry 5 (Product):**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.09 (s, 6H), 0.93 (s, 9H), 4.66 (s, 2H), 6.76 (d,  $J = 8.7$  Hz, 2H), 7.18 (d,  $J = 8.7$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  -5.18, 18.43, 25.98, 64.81, 115.10, 127.84, 133.51, 154.62.

