

# **Cu/Ag-catalyzed double decarboxylative cross-coupling reaction between cinnamic acids and aliphatic acids in aqueous solution**

Wen-Peng Mai,\* Ge Song, Gang-Chun Sun,\* Liang-Ru Yang, Jin-Wei Yuan,  
Yong-Mei Xiao, Pu Mao and Ling-Bo Qu

*Chemistry and Chemical Engineering School, Henan University of Technology,  
Zhengzhou Henan 450001, China*

Email: [maiwp@yahoo.com](mailto:maiwp@yahoo.com) sungangchun@163.com

## **Supporting Information**

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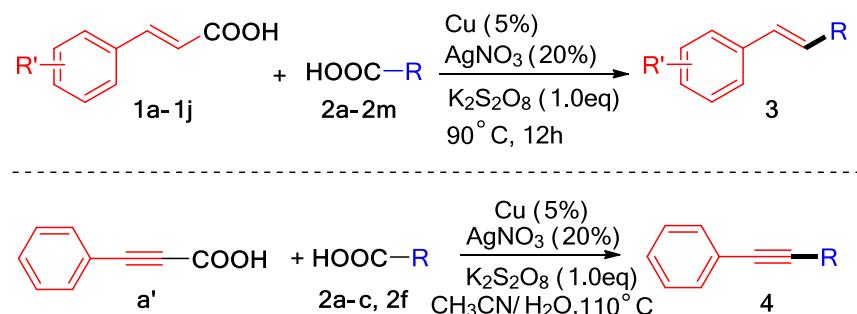
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## I. General Information

All experiments were carried out using common flask in air. All substrates were purchased from commercial suppliers and used as received directly unless otherwise noted. All solvents and other commercially available reagents were purchased from Acros or TCI companies and used directly. Reactions were monitored by thin layer chromatography (TLC) (*Qingdao Haiyang Chemical Co. Ltd. Silica gel 60 F254*). Products were detected using a UV/Vis lamp (254 nm). Column chromatography was performed on *Qingdao Haiyang Chemical Co. Ltd. Gel 60* (200–300 mesh). The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were obtained on a Bruker 400 MHz NMR Fourier transform spectrometer.  $^1\text{H}$  NMR data was reported as: chemical shift ( $\delta$  ppm), multiplicity, coupling constant (Hz), and integration.  $^{13}\text{C}$  NMR data was reported in terms of chemical shift ( $\delta$  ppm) multiplicity, and coupling constant (Hz). Hertz (Hz). The spectra are referenced against the internal solvent ( $\text{CDCl}_3$ ,  $\delta$   $^1\text{H}$ = 7.26 ppm,  $^{13}\text{C}$ = 77.0 ppm;  $\text{DMSO-d}_6$ ,  $\delta$   $^1\text{H}$ = 2.50 ppm,  $^{13}\text{C}$ = 40.0 ppm). Data is reported as follows: s= singlet, d= doublet, t= triplet, q=quartet and m= multiplet. ESI-MS spectra were recorded on a Bruker Esquire 3000. High resolution mass spectra (HR MS) were obtained on a Waters Micromass Q-Tof MicroTM instrument using the ESI technique.

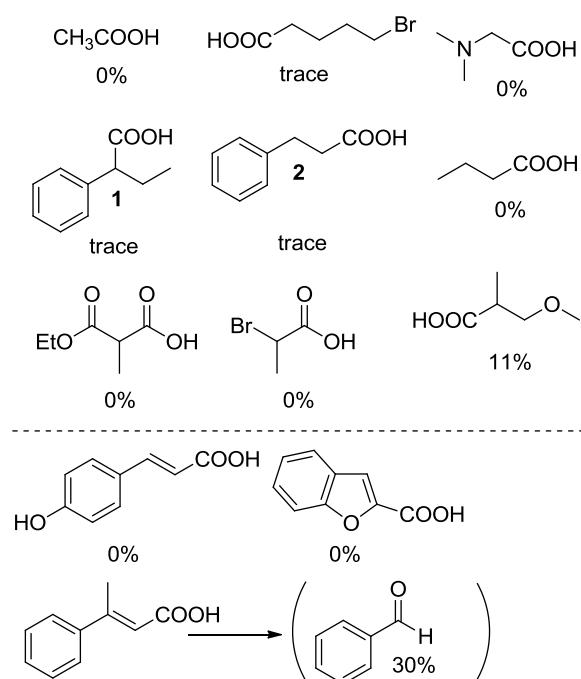
## II. Experimental Section

### (a) General Procedure:



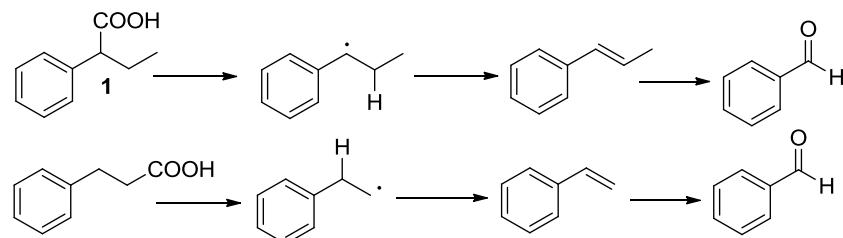
An 50 mL vial was charged with magnetic stir bar, cinnamic acid (**1**) or phenylpropiolic acid (**a'**) (1.0 mmol), aliphatic acid (**2**, 1.5 mmol), copper powder (0.05 mmol),  $\text{AgNO}_3$  (0.2 mmol) and  $\text{K}_2\text{S}_2\text{O}_8$  (1.0 mmol) were added in turn, followed by  $\text{CH}_3\text{CN}/\text{H}_2\text{O}$  (3 mL/3 mL). After stirring at  $90^\circ\text{C}$  for 12 h (or at  $110^\circ\text{C}$  for 12 h), The reaction mixture was concentrated in vacuo and extracted with  $\text{EtOAc}$  ( $3 \times 10$  mL). The combined organic phase was washed by saturated  $\text{NaHCO}_3$  solution and dried by anhydrous  $\text{Na}_2\text{SO}_4$  and then evaporated under reduced pressure. The isolated yield was obtained by flash chromatography column on silica gel (gradient eluent of Ethyl acetate in Petroleum: 0 ~ 2%, v/v).

**(b) Under the optimized conditions, the following substrates were not effective in this transformation.**

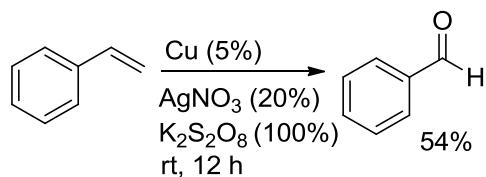


**Scheme S1**

In a word, primary acids failed to proceed under current conditions. The reactions of secondary acids containing ether, halide or ketone groups on adjacent position also do not proceed or produce low yields. Future studies will be aimed at overcoming these limitations. According to our test results, for **1** and **2**, in their reactions, about 40% benzaldehyde was isolated. They maybe proceed via the following routes:



We did the controlled experiment:

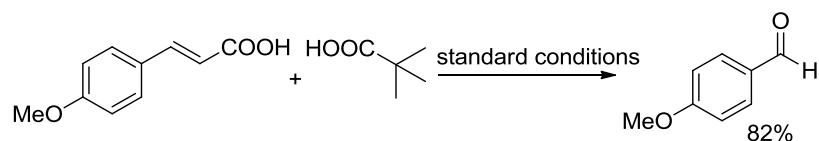


**Scheme S2**

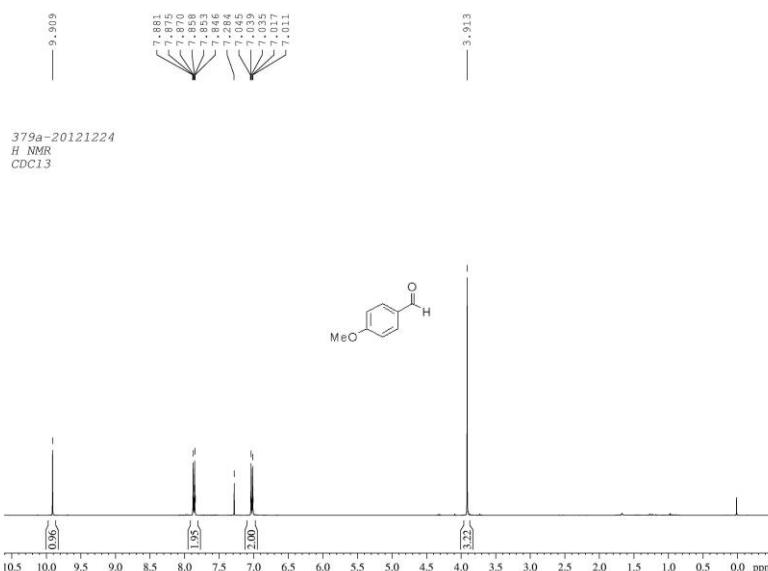
**S4**

**(c) Other experiments**

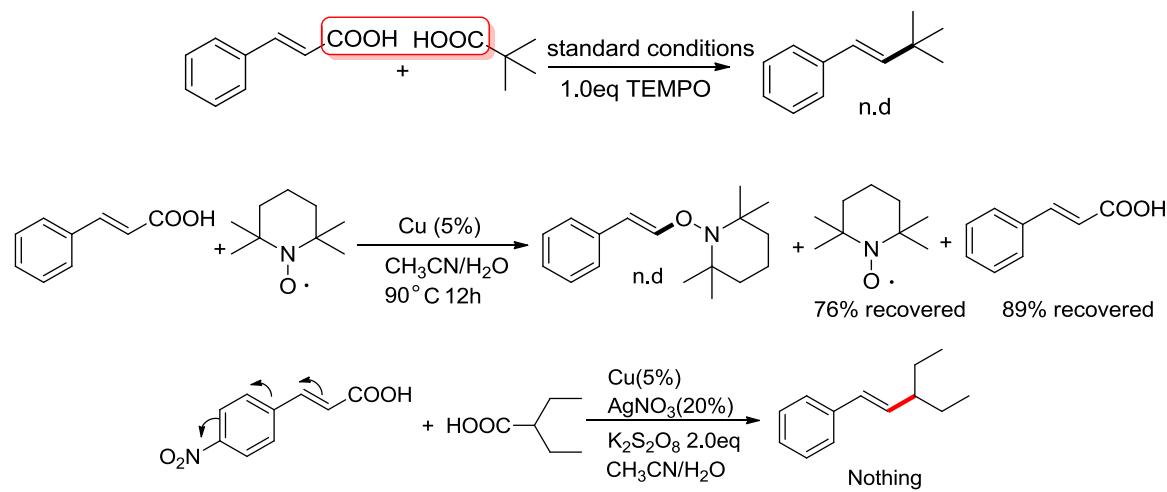
For (*E*)-4-Methoxycinnamic acid, we obtained an interesting result and we do not know the mechanism of this reaction until now.



**Scheme S3**



TEMPO could shut off the reaction:



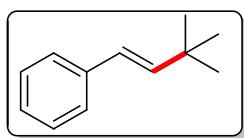
**Scheme S4**

**S5**

### III References and Notes

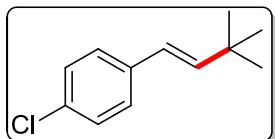
- (1) J. H. Delcamp and M. C. White, *J. Am. Chem. Soc.*, **2006**, *128*, 15076.
- (2) Z. Cui, X. Shang, X. F. Shao and Z. Q. Liu, *Chem. Sci.*, **2012**, *3*, 2853.
- (3) Y. J. Jang, Y. K. Shih, J. Y. Liu, W. Y. Kuo and C. F. Yao, *Chem. Eur. J.*, **2003**, *9*, 2123.
- (4) R. Trivedi and J. A. Tunge, *Org. Lett.*, **2009**, *11*, 5650.

## Characterizations of Products



### **1-((E)-3,3-dimethylbut-1-enyl)benzene (3aa)<sup>1</sup>**

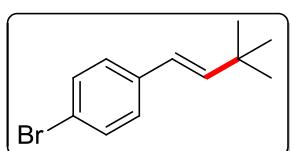
The mixture of *trans*-Cinnamic acid (1.0 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and Pivalic acid (1.5 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 90°C for 12 h. After the usual work-up, compound **3aa** was isolated as a colorless oil. Yield: 78%. R<sub>f</sub> = 0.9 (petroleum ether). **<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)** 7.38-7.40 (m, 2H), 7.30-7.34 (m, 2H), 7.19-7.23 (m, 1H), 6.35 (d, *J* = 16.0 Hz, 1H), 6.30 (d, *J* = 16.0 Hz, 1H), 1.15 (s, 9H). **<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>)** 141.8, 138.0, 128.5, 126.7, 126.0, 124.5, 33.4, 29.6



### **1- chloro-4-((E)-3,3-dimethylbut-1-enyl)benzene (3ba)**

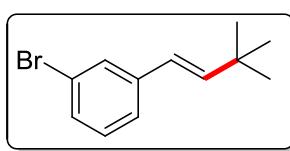
The mixture of 4-Chlorocinnamic acid (1.5 mmol), Cu (0.075 mmol), AgNO<sub>3</sub> (0.3 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.5 mmol) and Pivalic acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at 90°C for 12 h. After the usual work-up, compound **3ba** was isolated as a white solid, mp: 57-59°C. Yield: 85%. R<sub>f</sub> = 0.9 (petroleum ether). **<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)** 7.26-7.31 (m, 4H), 6.21-6.30 (t, *J* = 16.8 Hz, 2H), 1.13 (s, 9H)

**<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>)** 142.5, 136.5, 132.2, 128.5, 127.2, 123.4, 33.4, 29.5. **HRMS ESI (m/z):** [M+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>16</sub>Cl, 195.0941; found, 195.0930



### **1- bromo-4-((E)-3,3-dimethylbut-1-enyl)benzene (3ca)**

The mixture of 4-Bromocinnamic acid (1.5 mmol), Cu (0.075 mmol), AgNO<sub>3</sub> (0.3 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.5 mmol) and Pivalic acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at 90°C for 12 h. After the usual work-up, compound **3ca** was isolated as a colorless oil. Yield: 62%. R<sub>f</sub> = 0.9 (petroleum ether). **<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)** 7.41-7.43 (d, *J* = 8.4 Hz, 2H), 7.23-7.25 (d, *J* = 8.4 Hz, 2H), 6.21-6.30 (t, *J* = 16.8 Hz, 2H), 1.13 (s, 9H) **<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>)** 142.6, 137.0, 131.5, 127.5, 123.5, 120.3, 33.4, 29.7. **HRMS ESI (m/z):** [M+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>16</sub>Br, 239.0435; found, 239.0422

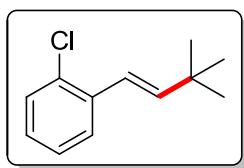


### **1- bromo-3-((E)-3,3-dimethylbut-1-enyl)benzene (3da)**

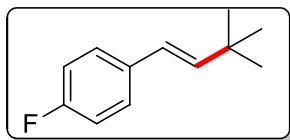
The mixture of trans-3-Bromocinnamic acid (1.5 mmol), Cu (0.075 mmol), AgNO<sub>3</sub> (0.3 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.5 mmol) and Pivalic acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at 90°C for 12

h. After the usual work-up, compound **3da** was isolated as a colorless oil.

Yield: 47%.  $R_f = 0.9$  (petroleum ether).  **$^1\text{H NMR}$  (400MHz,  $\text{CDCl}_3$ )** 7.54 (s, 1H), 7.31-7.34 (m, 1H), 7.27-7.29 (m, 1H), 7.15-7.19 (t,  $J = 7.8$  Hz, 1H), 6.31 (d,  $J = 16.4$  Hz, 1H), 6.25 (d,  $J = 16.4$  Hz, 1H), 1.13 (s, 9H)  **$^{13}\text{C NMR}$  (100MHz,  $\text{CDCl}_3$ )** 143.4, 140.2, 129.9, 129.5, 128.8, 124.8, 123.3, 122.7, 33.5, 29.4. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{12}\text{H}_{16}\text{Br}$ , 239.0435; found, 239.0425

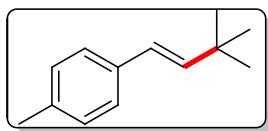


**1-chloro-2-((E)-3,3-dimethylbut-1-enyl)benzene (3ea)** The mixture of 2-Chlorocinnamic acid (1.5 mmol), Cu (0.075 mmol),  $\text{AgNO}_3$  (0.3 mmol),  $\text{K}_2\text{S}_2\text{O}_8$  (1.5 mmol) and Pivalic acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at room temperature for 2 h and then 90°C for 10 h. After the usual work-up, compound **3ea** was isolated as a colorless oil. Yield: 82%.  $R_f = 0.95$  (petroleum ether).  **$^1\text{H NMR}$  (400MHz,  $\text{CDCl}_3$ )** 7.52-7.55 (dd,  $J = 1.6$  Hz, 6.0Hz, 1H), 7.34-7.36 (dd,  $J = 1.2$  Hz, 6.8Hz, 1H), 7.20-7.24 (m, 1H), 7.13-7.17 (m, 1H), 6.73 (d,  $J = 16.0$  Hz, 3H), 6.27 (d,  $J = 16.0$  Hz, 1H), 1.16 (s, 9H)  **$^{13}\text{C NMR}$  (100MHz,  $\text{CDCl}_3$ )** 144.6, 136.1, 132.8, 129.5, 127.7, 126.7, 126.5, 121.0, 33.8, 29.5. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{12}\text{H}_{16}\text{Cl}$ , 195.0941; found, 195.0938



**1- fluoro-4-((E)-3,3-dimethylbut-1-enyl)benzene (3fa)**

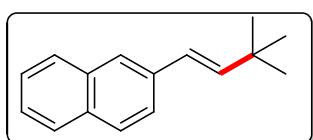
The mixture of 4-Fluorocinnamic acid (1.5 mmol), Cu (0.05 mmol),  $\text{AgNO}_3$  (0.2 mmol),  $\text{K}_2\text{S}_2\text{O}_8$  (1.0 mmol) and Pivalic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at  $90^\circ\text{C}$  for 12 h. After the usual work-up, compound **3fa** was isolated as a colorless oil. Yield: 29%.  $R_f = 0.95$  (petroleum ether).  **$^1\text{H NMR}$  (400MHz,  $\text{CDCl}_3$ )** 7.35 (d,  $J = 5.2$  Hz, 1H), 7.33 (d,  $J = 5.2$  Hz, 1H), 6.98-7.02 (t,  $J = 8.0$  Hz, 2H), 6.30 (d,  $J = 16.0$  Hz, 1H), 6.20 (d,  $J = 16.0$  Hz, 1H), 1.13 (s, 9H)  **$^{13}\text{C NMR}$  (100MHz,  $\text{CDCl}_3$ )** 163.0, 160.6, 141.6, 134.1, 127.4, 123.4, 115.4, 33.3, 29.5. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{12}\text{H}_{16}\text{F}$ , 179.1236; found, 179.1233



**1- methyl-4-((E)-3,3-dimethylbut-1-enyl)benzene(3ga)**

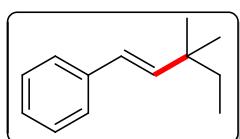
The mixture of 4-Methylcinnamic acid (trans) (1.5 mmol), Cu (0.075 mmol),  $\text{AgNO}_3$  (0.3 mmol),  $\text{K}_2\text{S}_2\text{O}_8$  (1.5 mmol) and Pivalic acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at room temperature for 2 h and then  $90^\circ\text{C}$  for 10 h. After the usual work-up, compound **3ga** was isolated as a colorless oil. Yield: 36%.  $R_f = 0.85$

(petroleum ether).  **$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ )** 7.27-7.29 (m, 2H), 7.11-7.13 (d,  $J$  = 8.0 Hz, 2H), 6.31 (d,  $J$  = 16.4 Hz, 1H), 6.24 (d,  $J$  = 16.4 Hz, 1H), 2.34 (s, 3H), 1.13 (s, 9H).  **$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ )** 140.8, 136.3, 135.2, 129.1, 125.8, 124.3, 33.2, 29.6, 21.1. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{13}\text{H}_{19}$ , 175.1487; found, 175.1459



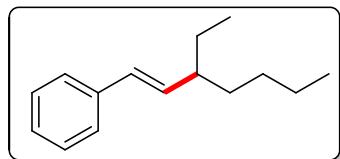
### **2-((E)-3,3-dimethylbut-1-enyl)naphthalene (3ha)**

The mixture of 3-(Naphtha-2-yl)acrylic acid (1.5 mmol), Cu (0.075 mmol),  $\text{AgNO}_3$  (0.3 mmol),  $\text{K}_2\text{S}_2\text{O}_8$  (1.5 mmol) and Pivalic acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at 90°C for 12 h. After the usual work-up, compound **3ha** was isolated as a white solid, mp: 47-51 °C. Yield: 40%.  $R_f$  = 0.90 (petroleum ether).  **$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ )** 7.80 (t,  $J$  = 7.6 Hz, 3H), 7.73 (s, 1H), 7.61-7.64 (dd,  $J$  = 1.6 Hz, 6.8 Hz, 1H), 7.41-7.48 (m, 2H), 6.52 (d,  $J$  = 17.2 Hz, 1H), 6.43 (d,  $J$  = 17.2 Hz, 1H), 1.19 (s, 9H)  **$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ )** 142.3, 135.5, 133.7, 132.6, 128.0, 127.7, 127.6, 126.0, 125.4, 125.3, 124.7, 123.6, 33.5, 22.7. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{16}\text{H}_{19}$ , 211.1487; found, 211.1482



### 1-((E)-3,3-dimethylpent-1-enyl)benzene (3ab)

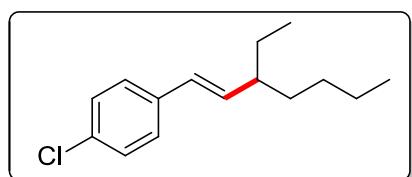
The mixture of trans-Cinnamic acid (1.5 mmol), Cu (0.075 mmol), AgNO<sub>3</sub> (0.3 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.5 mmol) and 2,2-Dimethylbutyric acid (2.0 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at 90 °C for 12 h. After the usual work-up, compound **3ab** was isolated as a colorless oil. Yield: 89%.  $R_f$  = 0.95 (petroleum ether). **1H NMR** (400MHz, CDCl<sub>3</sub>) 7.40 (dd,  $J$  = 1.6 Hz, 8.8Hz, 2H), 7.30-7.34 (m, 2H), 7.23 (ddd,  $J$  = 6.4 Hz, 2.4 Hz, 1.2 Hz, 1H), 6.33 (d,  $J$  = 16.0 Hz, 1H), 6.21 (d,  $J$  = 16.0 Hz, 1H), 1.47 (q,  $J$  = 7.6 Hz, 2H), 1.10 (s, 6H), 0.88 (t,  $J$  = 7.2 Hz, 3H) **13C NMR** (100MHz, CDCl<sub>3</sub>) 140.6, 138.1, 128.4, 126.7, 126.0, 125.8, 36.4, 35.5, 26.7, 9.06. **HRMS ESI (m/z):** [M+H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>19</sub>, 175.1487; found, 175.1488



### 1-((S,E)-3-ethylhept-1-enyl)benzene (3ac)

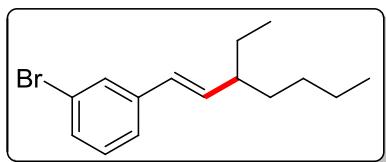
The mixture of trans-Cinnamic acid (1.5 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and 2-Ethylhexanoic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 90 °C for 12 h. After the usual work-up, compound **3ac** was isolated as a colorless oil. Yield: 90%.  $R_f$  = 0.95 (petroleum ether). **1H NMR** (400MHz, CDCl<sub>3</sub>) 7.38-7.40 (d,  $J$  = 7.8 Hz, 2H), 7.30-7.34 (m, 2H), 7.21 (t,  $J$  = 7.4 Hz, 1H), 6.36 (d,  $J$

= 15.6 Hz, 1H), 5.94-6.01 (dd,  $J$  = 8.8 Hz, 15.6 Hz, 1H), 1.99-2.08 (m, 1H), 1.45-1.57 (m, 2H), 1.22-1.40 (m, 6H), 0.88-0.92 (m, 6H).  **$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ )** 137.9, 135.6, 129.6, 128.4, 126.7, 125.9, 45.2, 34.9, 29.6, 28.2, 22.9, 14.1, 11.9. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{15}\text{H}_{23}$ , 203.1800; found, 203.1794



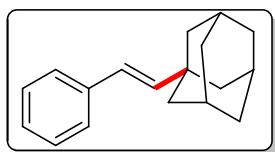
### 1- chloro-4-((S,E)-3-ethylhept-1-enyl)benzene (3bc)

The mixture of 4-Chlorocinnamic acid (1.5 mmol), Cu (0.05 mmol),  $\text{AgNO}_3$  (0.2 mmol),  $\text{K}_2\text{S}_2\text{O}_8$  (1.0 mmol) and 2-Ethylhexanoic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 90 °C for 12 h. After the usual work-up, compound **3bc** was isolated as a colorless oil. Yield: 92%.  $R_f$  = 0.94 (petroleum ether).  **$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ )** 7.26-7.31 (m, 4H), 6.31 (d,  $J$  = 15.6 Hz, 1H), 5.92-5.98 (dd,  $J$  = 8.8 Hz, 15.6 Hz, 1H), 1.98-2.07 (m, 1H), 1.44-1.57 (m, 2H), 1.21-1.39 (m, 6H), 0.87-0.91 (m, 6H).  **$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ )** 136.4, 136.3, 132.2, 128.5, 128.4, 127.1, 45.2, 34.8, 29.6, 28.1, 22.9, 14.1, 11.9. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{15}\text{H}_{22}\text{Cl}$ , 237.1410; found, 237.1402



**1- bromo-3-((S,E)-3-ethylhept-1-enyl)benzene (3dc)**

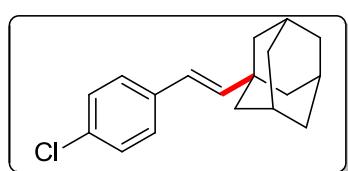
The mixture of trans-3-Bromocinnamic acid (1.5 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and 2-Ethylhexanoic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 90 °C for 12 h. After the usual work-up, compound **3dc** was isolated as a colorless oil. Yield: 71%. R<sub>f</sub> = 0.92 (petroleum ether). **<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)** 7.53 (d, *J* = 1.6 Hz, 1H), 7.32-7.34 (m, 1H), 7.26 (m, 1H), 7.17 (t, *J* = 7.8 Hz, 1H), 6.29 (d, *J* = 16.0 Hz, 1H), 5.95-6.01 (dd, *J* = 8.8 Hz, 16.0 Hz, 1H), 1.98-2.07 (m, 1H), 1.43-1.57 (m, 2H), 1.21-1.39 (m, 6H), 0.87-0.91 (m, 6H) **<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>)** 140.1, 137.3, 129.9, 129.5, 128.7, 128.3, 124.7, 122.7, 45.2, 34.7, 29.6, 28.1, 22.8, 14.1, 11.8. **HRMS ESI (m/z):** [M+H]<sup>+</sup> calcd for C<sub>15</sub>H<sub>22</sub>Br, 281.0905; found, 281.0877



**(1-phenylethenyl)adanantine (3ad)<sup>2</sup>**

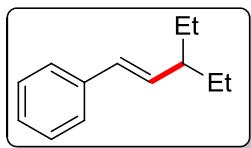
The mixture of trans-Cinnamic acid (1.5 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and 1-Adamantanecarboxylic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 90 °C for 12 h. After the usual work-up, compound **3ad** was isolated as a white solid.

Yield: 65%.  $R_f$  = 0.90 (petroleum ether). **1H NMR (400MHz, CDCl<sub>3</sub>)** 7.39 (d,  $J$  = 7.2 Hz, 2H), 7.29-7.33 (m, 2H), 7.22 (t,  $J$  = 7.2 Hz, 1H), 6.28 (d,  $J$  = 16.4 Hz, 1H), 6.15 (d,  $J$  = 16.4 Hz, 1H), 2.05 (s, 3H), 1.70-1.79 (m, 12H) **13C NMR (100MHz, CDCl<sub>3</sub>)** 142.1, 138.2, 128.4, 126.7, 126.0, 124.4, 42.2, 36.9, 35.1, 28.4.



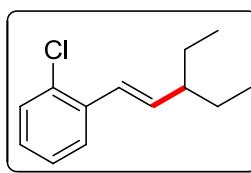
### 1-((4-chlorophenyl)ethenyl)adamantine (3bd)

The mixture of 4-Chlorocinnamic acid (1.5 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and 1-Adamantanecarboxylic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 90°C for 12 h. After the usual work-up, compound **3bd** was isolated as a white solid. Yield: 70%.  $R_f$  = 0.9 (petroleum ether). **1H NMR (400MHz, CDCl<sub>3</sub>)** 7.29-7.31 (dd,  $J$  = 2.0 Hz, 4.4 Hz, 2H), 7.25-7.27 (dd,  $J$  = 2.0 Hz, 4.4 Hz, 2H), 6.22 (d,  $J$  = 16.4 Hz, 1H), 6.11 (d,  $J$  = 16.4 Hz, 1H), 2.05 (s, 3H), 1.72-1.79 (m, 3H), 1.69 (s, 9H) **13C NMR (100MHz, CDCl<sub>3</sub>)** 142.7, 136.7, 132.1, 128.5, 127.2, 123.4, 42.1, 36.8, 35.2, 28.4. **HRMS ESI (m/z):** [M+H]<sup>+</sup> calcd for C<sub>18</sub>H<sub>22</sub>Cl, 273.1410; found, 273.1412



### 1-((E)-3-ethylpent-1-enyl)benzene (3ae)

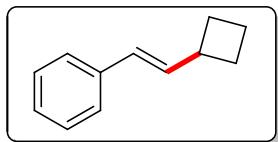
The mixture of trans-Cinnamic acid (1.5 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and 2-Ethylbutyric acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 90°C for 12 h. After the usual work-up, compound **3ae** was isolated as a colorless oil. Yield: 82%. R<sub>f</sub> = 0.95 (petroleum ether). **1H NMR** (400MHz, CDCl<sub>3</sub>) 7.39 (d, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.23 (t, *J* = 7.2 Hz, 1H), 6.38 (d, *J* = 16.0 Hz, 1H), 5.94-6.00 (dd, *J* = 8.8 Hz, 16.0 Hz, 1H), 1.59-2.00 (m, 1H), 1.48-1.59 (m, 2H), 1.27-1.41 (m, 2H), 0.92 (t, *J* = 7.4 Hz, 6H). **13C NMR** (100MHz, CDCl<sub>3</sub>) 137.9, 135.3, 129.8, 128.4, 126.7, 125.9, 46.9, 27.8, 11.9. **HRMS ESI (m/z):** [M+H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>19</sub>, 175.1487; found, 175.1475



### 1- chloro-2-((E)-3-ethylpent-1-enyl)benzene (3be)

The mixture of 2-Chlorocinnamic acid (1.5 mmol), Cu (0.075 mmol), AgNO<sub>3</sub> (0.3 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.5 mmol) and 2-Ethylbutyric acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at room temperature for 2 h and then 90°C for 10 h. After the usual work-up,

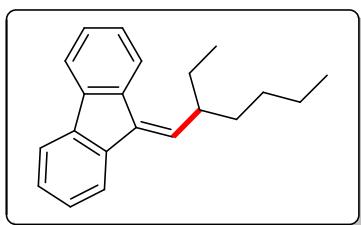
compound **3be** was isolated as a colorless oil. Yield: 89%.  $R_f = 0.90$  (petroleum ether).  **$^1\text{H NMR}$  (400MHz,  $\text{CDCl}_3$ )** 7.55 (dd,  $J = 2.0$  Hz, 8.0 Hz, 1H), 7.36 (dd,  $J = 1.2$  Hz, 7.6 Hz, 1H), 7.24 (td,  $J = 7.2$  Hz, 0.8 Hz, 1H), 7.17 (td,  $J = 7.6$  Hz, 1.6 Hz, 1H), 6.75 (d,  $J = 16.0$  Hz, 1H), 5.98 (dd,  $J = 9.2$  Hz, 16.0 Hz, 1H), 1.60-2.08 (m, 1H), 1.51-1.60 (m, 2H), 1.32-1.49 (m, 2H), 0.94 (t,  $J = 7.4$  Hz, 6H)  **$^{13}\text{C NMR}$  (100MHz,  $\text{CDCl}_3$ )** 138.2, 136.1, 132.5, 129.5, 127.7, 126.7, 126.4, 46.9, 27.7, 11.8. **HRMS ESI (m/z):  $[\text{M}+\text{H}]^+$**  calcd for  $\text{C}_{13}\text{H}_{18}\text{Cl}$ , 209.1097; found, 209.1096



### 1-((E)-2-cyclobutylvinyl)benzene (3af)

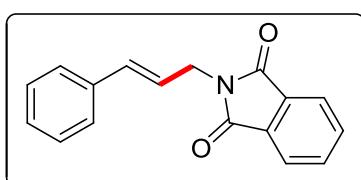
The mixture of trans-Cinnamic acid (1.5 mmol), Cu (0.05 mmol),  $\text{AgNO}_3$  (0.2 mmol),  $\text{K}_2\text{S}_2\text{O}_8$  (1.0 mmol) and Cyclobutanecarboxylic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 90 °C for 12 h. After the usual work-up, compound **3af** was isolated as a colorless oil. Yield: 63%.  $R_f = 0.90$  (petroleum ether).  **$^1\text{H NMR}$  (400MHz,  $\text{CDCl}_3$ )** 7.36-7.38 (dd,  $J = 2.0$  Hz, 8.4 Hz, 2H), 7.29-7.33 (m, 2H), 7.23 (ddd,  $J = 6.8$  Hz, 2.8Hz, 1.2Hz, 1H), 6.39 (d,  $J = 16.0$  Hz, 1H), 6.33 (d,  $J = 16.0$  Hz, 1H), 3.13 (dd,  $J = 5.6$  Hz, 8.0 Hz, 1H), 2.17-2.23 (m, 2H), 1.91-2.01 (m, 3H), 1.82-1.90 (m, 1H).  **$^{13}\text{C NMR}$  (100MHz,  $\text{CDCl}_3$ )** 137.7, 135.3, 128.4, 127.5, 126.8, 125.9, 38.7, 28.7, 18.6. **HRMS ESI (m/z):  $[\text{M}+\text{H}]^+$**  calcd

for  $C_{12}H_{15}$ , 159.1174; found, 159.1158



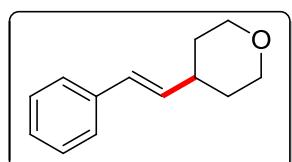
### 9-(heptan-3-ylidene)-9H-fluorene (3ic)

The mixture of 2-(9H-fluoren-9-ylidene)acetic acid (1.0 mmol), Cu (0.05 mmol),  $AgNO_3$  (0.2 mmol),  $K_2S_2O_8$  (1.0 mmol) and 2-Ethylhexanoic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at room temperature for 2 h and then 90 °C for 12 h. After the usual work-up, compound **3ic** was isolated as a colorless oil. Yield: 96%.  $R_f = 0.95$  (petroleum ether). **1H NMR** (400 MHz,  $CDCl_3$ ) 7.98 (d,  $J = 7.6$  Hz, 1H), 7.80 (d,  $J = 7.6$  Hz, 1H), 7.71-7.75 (m, 2H), 7.30-7.41 (m, 4H), 6.55 (d,  $J = 10.4$  Hz, 1H), 3.20-3.34 (m, 1H), 1.69-1.75 (m, 2H), 1.50-1.57 (m, 2H), 1.32-1.38 (m, 4H), 1.00 (t,  $J = 7.4$  Hz, 3H), 0.89 (t,  $J = 7.0$  Hz, 3H). **13C NMR** (100 MHz,  $CDCl_3$ ) 140.9, 139.6, 138.3, 137.6, 136.8, 135.1, 127.5, 127.3, 126.9, 126.8, 124.8, 119.8, 119.7, 119.4, 40.4, 35.3, 29.8, 28.7, 22.9, 14.1, 12.0. **HRMS ESI (m/z):**  $[M+H]^+$  calcd for  $C_{21}H_{25}$ , 277.1956; found, 277.1948



## 2- cinnamylisoindoline-1,3-dione (3ag)

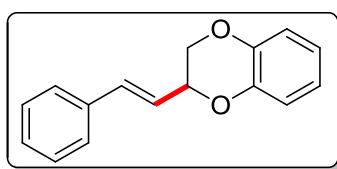
The mixture of trans-Cinnamic acid (1.5 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (2.0 mmol) and N-phthaloylglycine (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 90°C for 12 h. After the usual work-up, compound **3ag** was isolated as a yellow solid, mp: 126-130°C. Yield: 35%. R<sub>f</sub> = 0.6 (petroleum ether/ethyl acetate = 10 : 1).  
**<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)** 7.90 (q, *J* = 2.8 Hz, 2H), 7.73-7.76 (m, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 7.2 Hz, 2H), 7.22-7.26 (m, 1H), 6.70 (d, *J* = 15.6 Hz, 1H), 6.31 (dt, *J* = 15.6 Hz, 6.4 Hz, 1H), 4.48 (dd, *J* = 0.8 Hz, 6.4 Hz, 2H) **<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>)** 167.9, 136.2, 134.0, 133.7, 132.1, 128.5, 127.9, 126.5, 123.3, 122.7, 39.6. **HRMS ESI (m/z):** [M+H]<sup>+</sup> calcd for C<sub>17</sub>H<sub>14</sub>NO<sub>2</sub>, 264.1025; found, 264.1015



## tetrahydro-4-styryl-2H-pyran (3ah)<sup>3</sup>

The mixture of trans-Cinnamic acid (1.5 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and Tetrahydro-2H-pyran-4-carboxylic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at room temperature for 2 h and then 70°C for 10 h. After the usual work-up, compound **3ah** was isolated as a colorless oil. Yield: 28%. R<sub>f</sub> = 0.70 (petroleum ether/ethyl acetate = 20 : 1). **<sup>1</sup>H NMR (400MHz,**

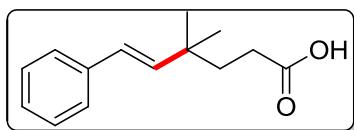
**CDCl<sub>3</sub>**) 7.39 (d, *J* = 8.0 Hz, 2H), 7.30-7.34 (m, 2H), 7.21-7.25 (m, 1H), 6.43 (d, *J* = 16.0 Hz, 1H), 6.21 (dd, *J* = 6.8 Hz, 16.0 Hz, 1H), 4.01-4.05 (m, 2H), 3.52 (td, *J* = 11.6 Hz, 2.0 Hz, 2H), 2.19-2.42 (m, 1H), 1.43-1.76 (m, 4H) **<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>)** 137.5, 134.6, 128.5, 128.2, 127.1, 126.0, 38.3, 32.6.



**(S)-2,3-dihydro-2-styrylbenzo[b][1,4]dioxine (3ai)**

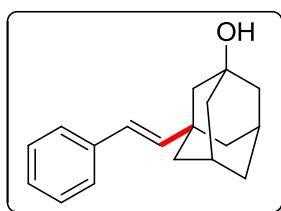
The mixture of trans-Cinnamic acid (1.5 mmol), Cu (0.075 mmol), AgNO<sub>3</sub>(0.3mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.5 mmol) and 1,4-Benzodioxan-2-carboxylic acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at room temperature for 2 h and then 90°C for 12 h. After the usual work-up, compound **3ai** was isolated as a yellow solid, mp: 65-68°C. Yield: 70%.

R<sub>f</sub> = 0.5 (petroleum ether/ethyl acetate = 10 : 1). **<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)** 7.44-7.46 (m, 2H), 7.35-7.39 (m, 2H), 7.29-7.33 (m, 1H), 6.88-6.99 (m, 4H), 6.884 (d, *J* = 16.0 Hz, 1H), 6.29 (dd, *J* = 6.4 Hz, 16.0 Hz, 1H), 4.80-4.85 (m, 1H), 4.37 (dd, *J* = 2.4 Hz, 11.2 Hz, 1H), 4.05 (dd, *J* = 8.0 Hz, 11.2 Hz, 1H) **<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>)** 143.2, 143.0, 135.9, 134.3, 128.7, 128.3, 126.7, 123.1, 121.7, 121.5, 117.4, 117.1, 73.7, 67.8. **HRMS ESI (m/z): [M+H]<sup>+</sup>** calcd for C<sub>16</sub>H<sub>15</sub>O<sub>2</sub>, 239.1072; found, 239.1070



**(E)-4,4-dimethyl-6-phenylhex-5-enoic acid (3aj)**

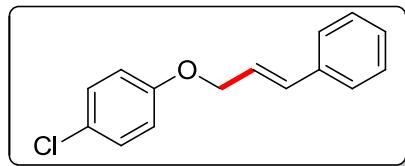
The mixture of trans-Cinnamic acid (1.0 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and Dimethylglutaric acid (1.2 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at room temperature for 2 h and then 70°C for 10 h. After the usual work-up, compound **3aj** was isolated as a sticky yellow liquid. Yield: 49%. R<sub>f</sub> = 0.40 (petroleum ether/ethyl acetate = 2 : 1). <sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>) 7.38 (d, *J* = 8.0 Hz, 2H), 7.33 (q, *J* = 7.6 Hz, 2H), 7.23 (t, *J* = 7.2 Hz, 1H), 6.33 (d, *J* = 16.0 Hz, 1H), 6.16 (d, *J* = 16.0 Hz, 1H), 2.34 (td, *J* = 8.0 Hz, 4.0 Hz, 2H), 1.78 (m, 2H), 1.14 (s, 6H) <sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>) 180.0, 138.8, 137.5, 128.5, 127.0, 126.9, 126.0, 37.2, 35.9, 29.8, 26.9. HRMS ESI (m/z): [M-H] calcd for C<sub>14</sub>H<sub>17</sub>O<sub>2</sub>, 217.1229; found, 217.1171



**3-((E)-styryl)adamantan-1-ol (3ak)**

The mixture of trans-Cinnamic acid (1.0 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and 3-hydroxy-1-adamantanecarboxylic acid (1.2 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at

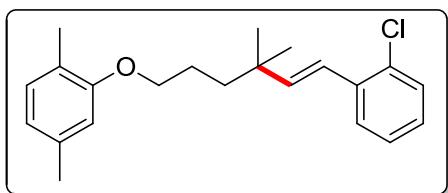
90°C for 12 h. After the usual work-up, compound **3bk** was isolated as a yellow solid, mp: 58-62°C. Yield: 76%.  $R_f$  = 0.3 (petroleum ether/ethyl acetate = 4 : 1). **1H NMR** (400MHz,  $\text{CDCl}_3$ ) 7.38-7.39 (m, 2H), 7.29-7.37 (m, 2H), 7.19-7.24 (m, 1H), 6.32 (d,  $J$  = 16.0 Hz, 1H), 6.17 (d,  $J$  = 16.0 Hz, 1H), 2.29 (s, 2H), 1.58-1.78 (m, 13H). **13C NMR** (100MHz,  $\text{CDCl}_3$ ) 139.9, 137.8, 128.5, 126.9, 126.1, 125.2, 68.9, 49.6, 44.6, 40.9, 38.8, 35.3, 30.6. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{18}\text{H}_{23}\text{O}$ , 255.1749; found, 255.1745



**1-((E)-3-(4-chlorophenoxy)prop-1-enyl)benzene (3al)<sup>4</sup>**

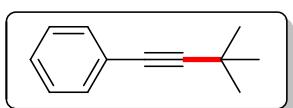
The mixture of trans-Cinnamic acid (1.0 mmol), Cu (0.05 mmol),  $\text{AgNO}_3$  (0.2 mmol),  $\text{K}_2\text{S}_2\text{O}_8$  (1.0 mmol) and 4-Chlorophenoxyacetic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at room temperature for 2 h and then 90°C for 10 h. After the usual work-up, compound **3al** was isolated as a yellow solid, mp: 79-81°C. Yield: 65%.  $R_f$  = 0.6 (petroleum ether/ethyl acetate = 10 : 1). **1H NMR** (400MHz,  $\text{CDCl}_3$ ) 7.44 (d,  $J$  = 8.0 Hz, 2H), 7.33-7.37 (m, 2H), 7.25-7.30 (m, 6H: ArH overlapping  $\text{CHCl}_3$ ), 6.89-6.93 (dt,  $J$  = 8.8 Hz, 3.6 Hz, 2H), 6.76 (d,  $J$  = 16.0 Hz, 1H), 6.45 (dt,  $J$  = 16.0 Hz, 5.6 Hz, 1H), 4.70 (dd,  $J$  = 1.6 Hz, 6.0 Hz, 2H). **13C NMR** (100MHz,  $\text{CDCl}_3$ ) 157.2, 136.2, 133.3, 129.3,

128.6, 128.0, 126.6, 125.7, 123.9, 116.0, 68.9.



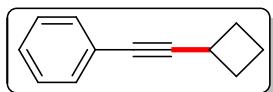
**(E)-2-((6-(2-chlorophenyl)-4,4-dimethylhex-5-en-1-yl)oxy)-1,4-dimethylbenzene (3em)**

The mixture of Gemfibrozil (1.0 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and (E)-3-(2-chlorophenyl)acrylic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at room temperature for 2 h and then 70°C for 10 h. After the usual work-up, compound **3em** was isolated as a colorless oil. Yield: 20%. R<sub>f</sub> = 0.8 (petroleum ether/ethyl acetate = 10:1). **<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)** 7.52 (d, *J* = 7.6Hz, 1H), 7.35 (d, *J* = 7.6Hz, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 6.66-6.74 (m, 3H), 6.21 (d, *J* = 16 Hz, 1H), 3.95 (t, *J* = 6.4 Hz, 2H), 2.3 (s, 3H), 2.2 (s, 3H), 1.77-1.84 (m, 2H), 1.56-1.63 (m, 2H), 1.19 (s, 6H). **<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>)** 157.04, 143.21, 136.46, 136.10, 132.82, 130.28, 129.57, 127.87, 126.74, 126.64, 123.60, 122.71, 120.61, 111.99, 68.33, 39.29, 36.53, 27.21, 24.97, 21.42, 15.84. **HRMS ESI (m/z): [M+H]<sup>+</sup>** calcd for C<sub>22</sub>H<sub>28</sub>ClO, 343.1829; found, 343.1807



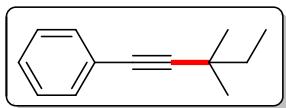
### 1-(3,3-dimethylbut-1-ynyl)benzene (**4a'a**)

The mixture of Phenylpropiolic acid (1.5 mmol), Cu (0.075 mmol), AgNO<sub>3</sub> (0.3 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.5 mmol) and Pivalic acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at 110°C for 15 h. After the usual work-up, compound **4a'a** was isolated as a colorless oil. Yield: 88%. R<sub>f</sub> = 0.9 (petroleum ether). **1H NMR (400MHz, CDCl<sub>3</sub>)** 7.39-7.42 (m, 2H), 7.27-7.32 (m, 3H), 1.35 (s, 9H) **13C NMR (100MHz, CDCl<sub>3</sub>)** 131.5, 128.1, 127.3, 124.0, 98.5, 79.0, 31.0, 27.9. **ESI-HRMS: (m/z):** [M+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>15</sub>, 159.1174; found, 159.1180



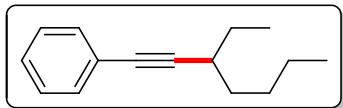
### 1-(2-cyclobutylethynyl)benzene (**4a'f**)

The mixture of Phenylpropiolic acid (1.5 mmol), Cu (0.05 mmol), AgNO<sub>3</sub> (0.2 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.0 mmol) and Cyclobutanecarboxylic acid (1.0 mmol) in acetonitrile (3 mL) and water (3 mL) was stirred at 110°C for 15 h. After the usual work-up, compound **4a'f** was isolated as a colorless oil. Yield: 84%. R<sub>f</sub> = 0.9 (petroleum ether). **1H NMR (400MHz, CDCl<sub>3</sub>)** 7.40-7.43 (m, 2H), 7.28-7.32 (m, 3H), 3.21-3.29 (m, 1H), 2.32-2.39 (m, 2H), 2.19-2.29 (m, 2H), 1.91-2.04 (m, 2H). **13C NMR (100MHz, CDCl<sub>3</sub>)** 131.5, 128.1, 127.5, 123.9, 93.9, 81.1, 30.0, 25.5, 19.2. **HRMS ESI (m/z):** [M+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>13</sub>, 157.1017; found, 157.1013



### 1-(3,3-dimethylpent-1-ynyl)benzene (4a'b)

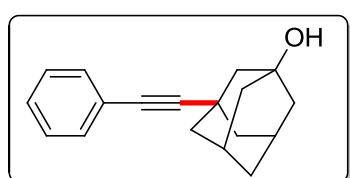
The mixture of Phenylpropiolic acid (1.5 mmol), Cu (0.075 mmol), AgNO<sub>3</sub> (0.3 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.5 mmol) and 2,2-Dimethylbutyric acid (1.5 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at 110°C for 15 h. After the usual work-up, compound **4a'b** was isolated as a colorless oil. Yield: 91%. R<sub>f</sub> = 0.9 (petroleum ether). **<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)** 7.43 (dt, *J* = 7.2 Hz, 2.4 Hz, 2H), 7.27-7.32 (m, 3H), 1.57 (q, *J* = 7.6 Hz, 2H), 1.29 (s, 6H), 1.10 (t, *J* = 7.6 Hz, 3H), **<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>)** 131.6, 128.1, 127.4, 124.2, 97.2, 80.4, 36.1, 32.1, 28.8, 9.8. **HRMS ESI (m/z): [M+H]<sup>+</sup>** calcd for C<sub>13</sub>H<sub>17</sub>, 173.1330; found, 173.1335



### 1-(3-ethylhept-1-ynyl)benzene (4a'c)

The mixture of Phenylpropiolic acid (1.5 mmol), Cu (0.075 mmol), AgNO<sub>3</sub> (0.3 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1.5 mmol) and 2-Ethylhexanoic acid (2.0 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at 110°C for 15 h. After the usual work-up, compound **4a'c** was isolated as a colorless oil. Yield: 95%. R<sub>f</sub> = 0.9 (petroleum ether). **<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)** 7.41-7.44 (m, 2H), 7.27-7.32 (m, 3H), 2.47-2.51 (m, 1H), 1.44-1.63 (m,

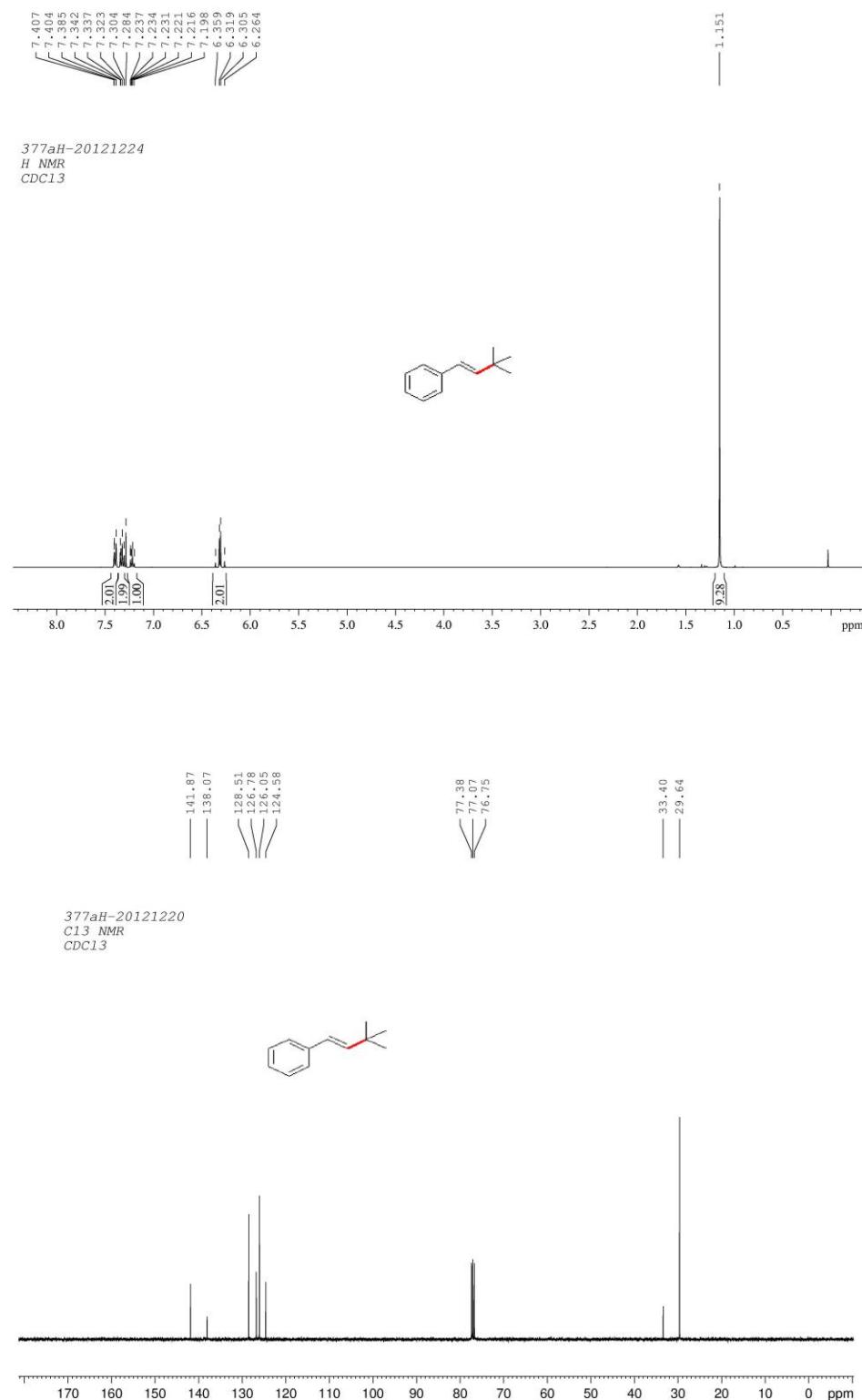
6H), 1.36-1.43 (m, 3H), 1.10 (t,  $J$  = 7.2 Hz, 3H), 0.97 (t,  $J$  = 7.2 Hz, 3H)  
 **$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ )** 131.6, 128.1, 127.3, 124.2, 93.7, 81.7, 34.5,  
34.0, 29.7, 28.2, 22.6, 14.1, 11.9. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  
 $\text{C}_{15}\text{H}_{21}$ , 201.1643; found, 201.1636

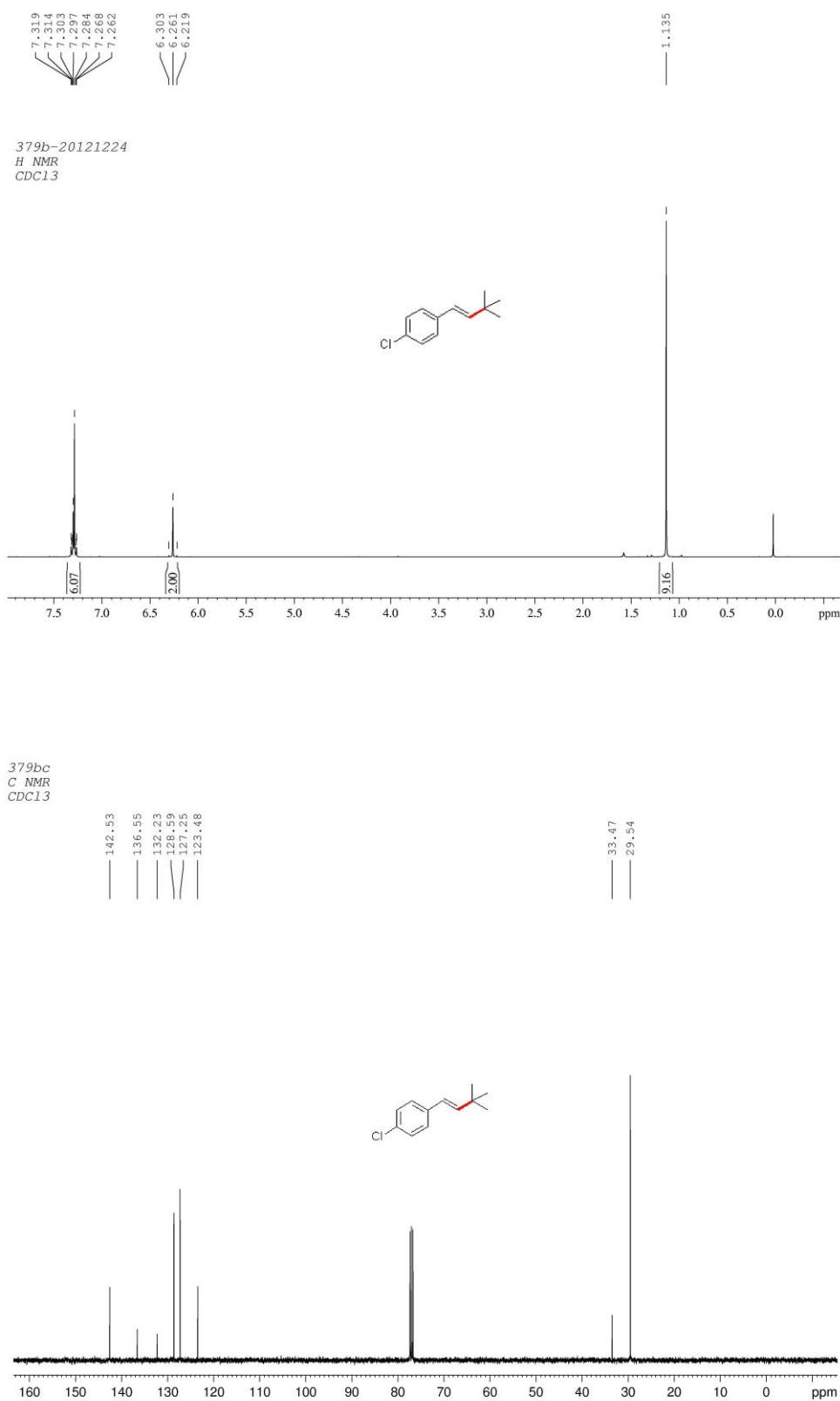


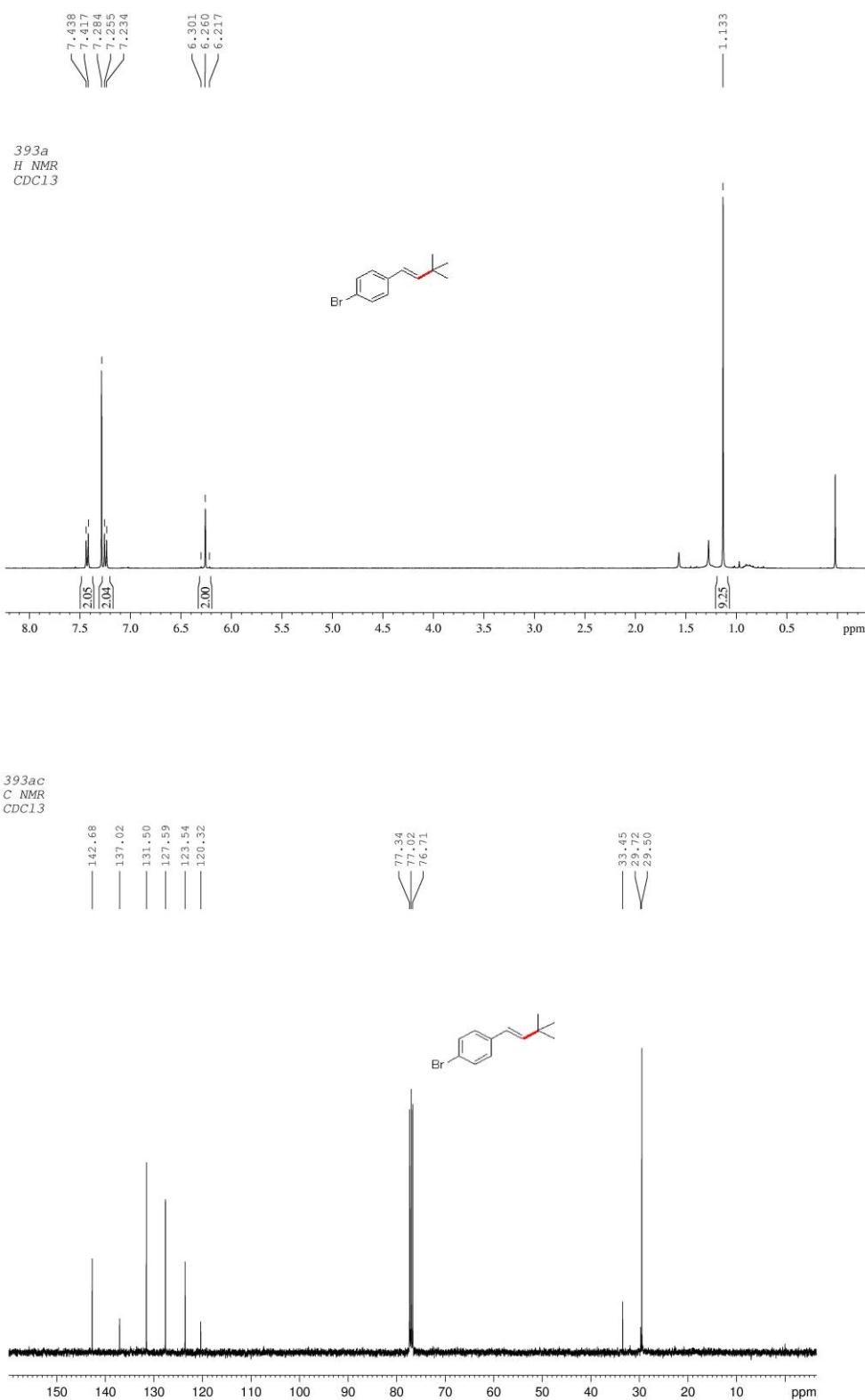
### 3-(phenylethynyl)adamantan-1-ol (4a'k)

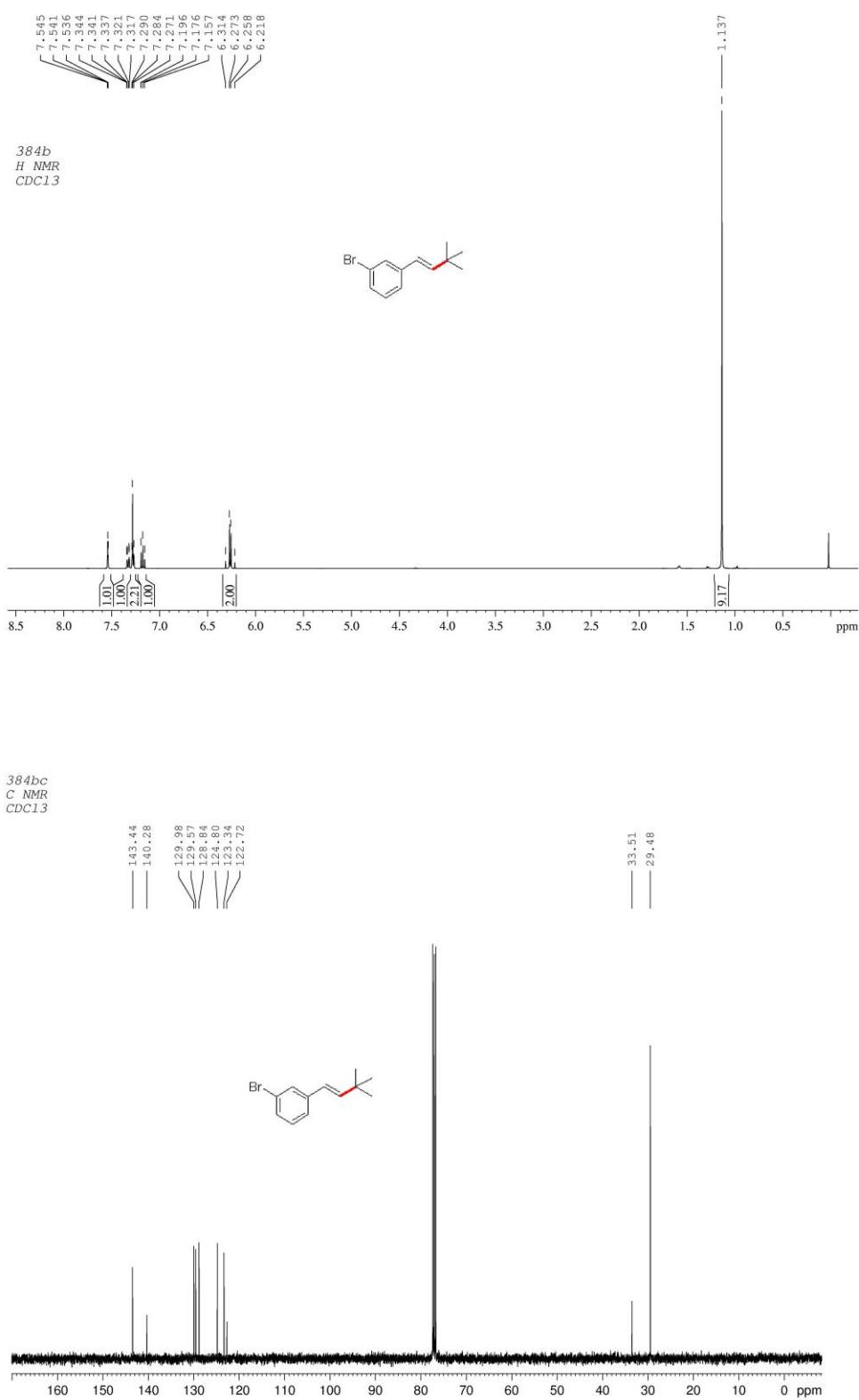
The mixture of Phenylpropiolic acid (1.5 mmol), Cu (0.075 mmol),  $\text{AgNO}_3$  (0.3 mmol),  $\text{K}_2\text{S}_2\text{O}_8$  (1.5 mmol) and 3-hydroxy-1-adamantanecarboxylic acid (2.0 mmol) in acetonitrile (5 mL) and water (5 mL) was stirred at 110°C for 12 h. After the usual work-up, compound **4a'd** was isolated as a colorless oil. Yield: 85%.  $R_f$  = 0.4 (petroleum ether/ethyl acetate = 4:1).  **$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ )** 7.39-7.42 (m, 2H), 7.28-7.31 (m, 3H), 2.26 (s, 2H), 1.93 (s, 2H), 1.88 (s, 4H), 1.73 (s, 4H), 1.56-1.66 (m, 3H).  **$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ )** 131.6, 128.2, 127.6, 123.7, 96.5, 79.9, 68.0, 50.2, 44.2, 41.6, 34.9, 33.2, 30.4. **HRMS ESI (m/z):**  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{18}\text{H}_{21}\text{O}$ , 253.1592; found, 253.1581

#### IV. NMR Spectrum Copies

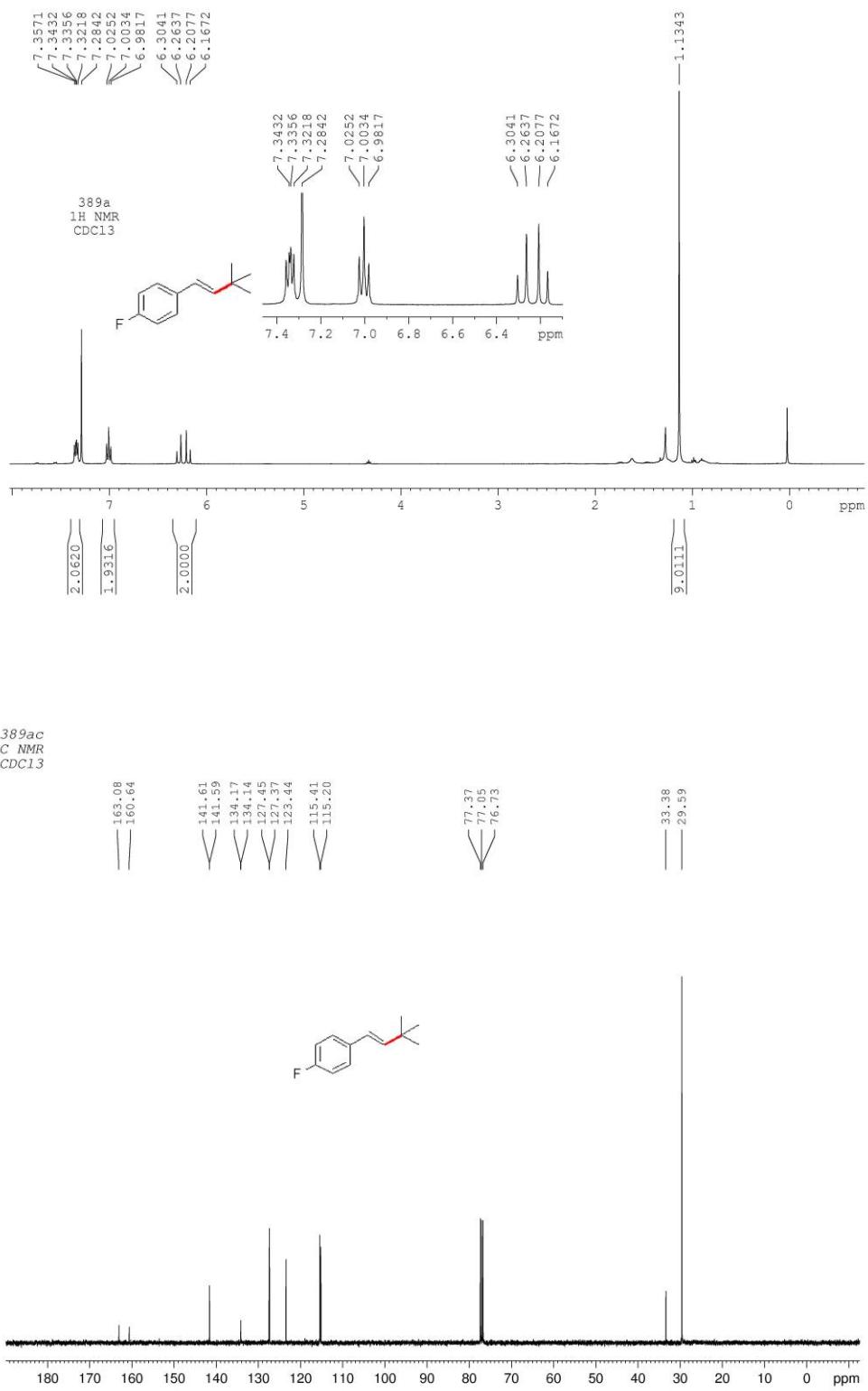


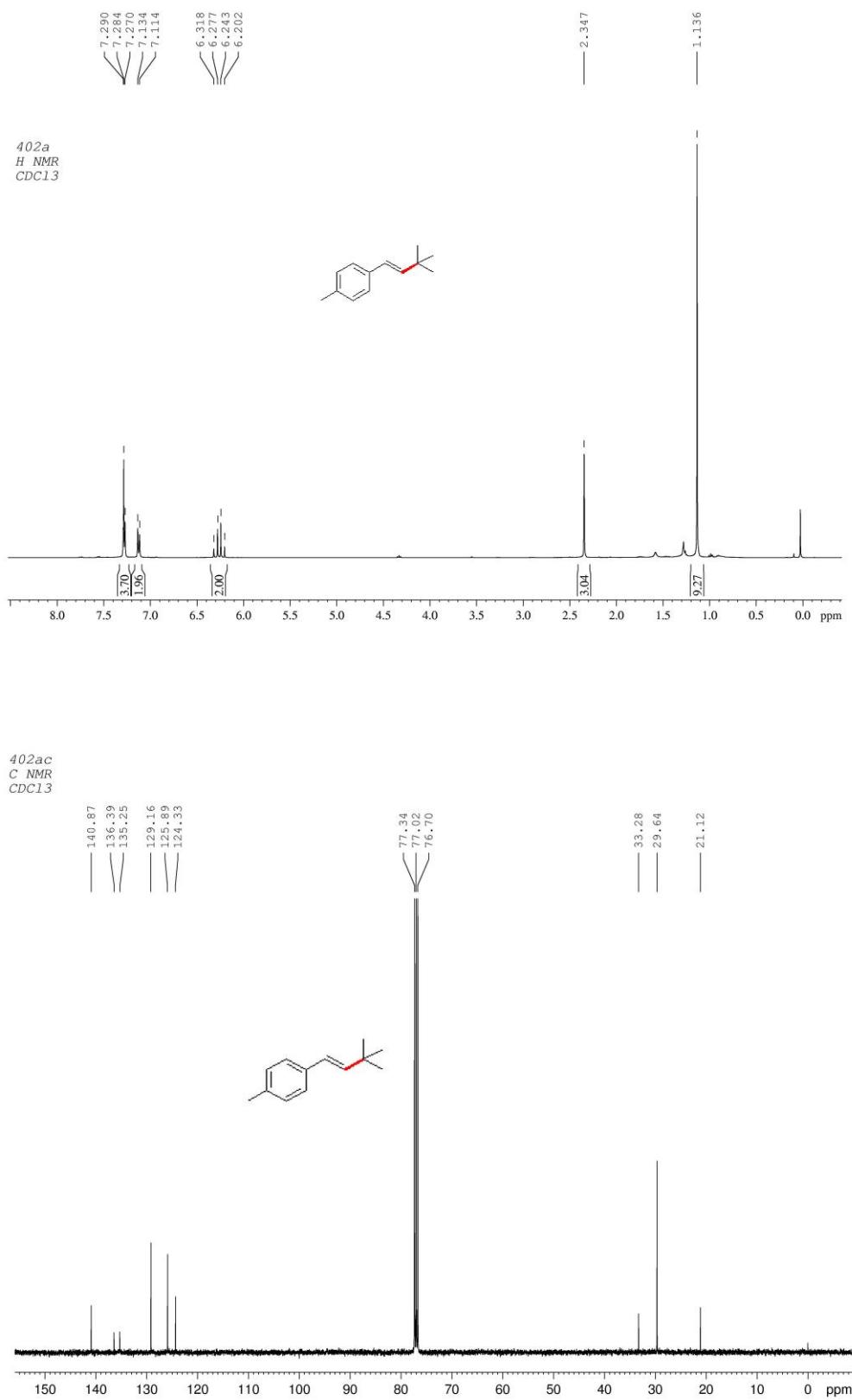


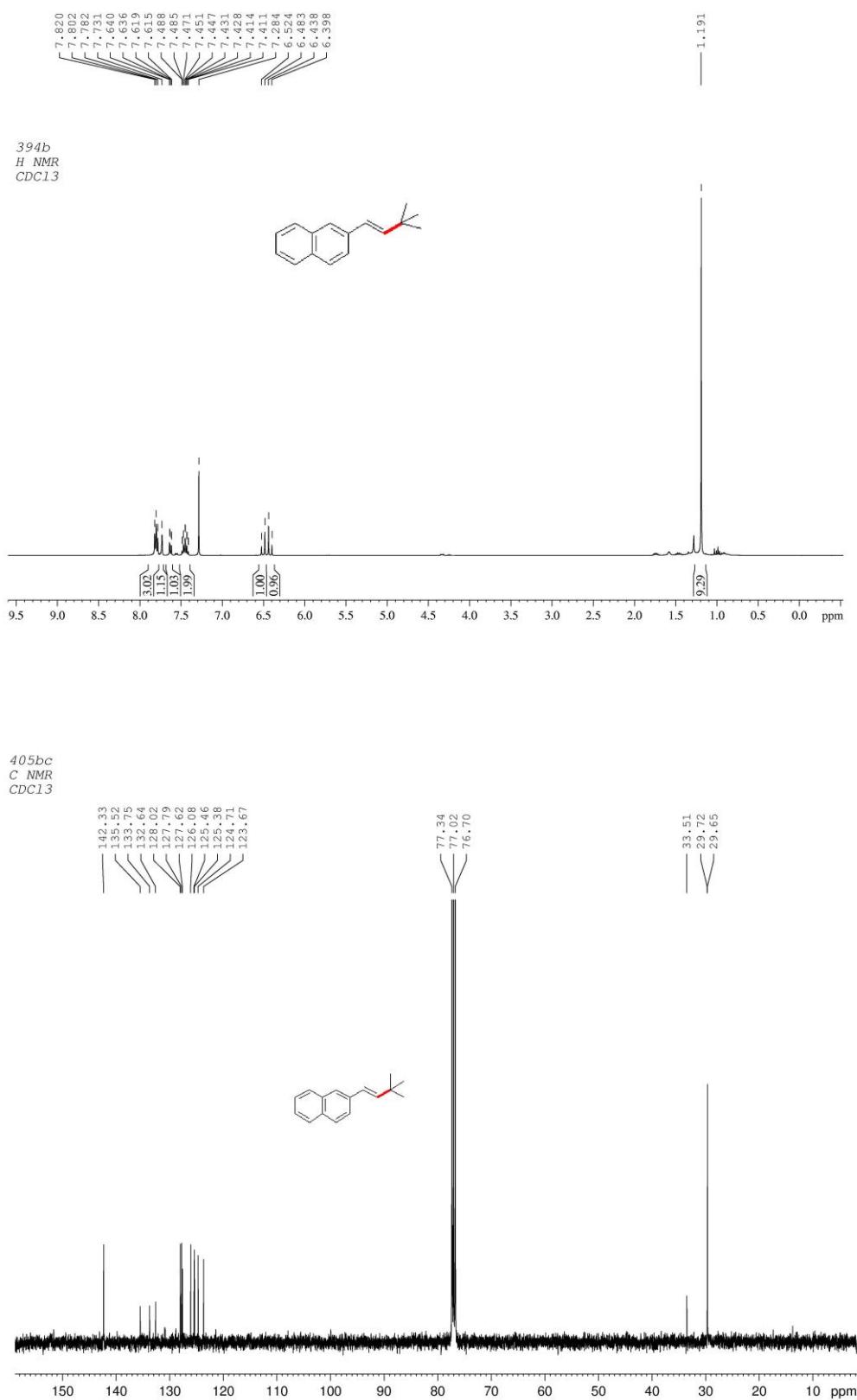


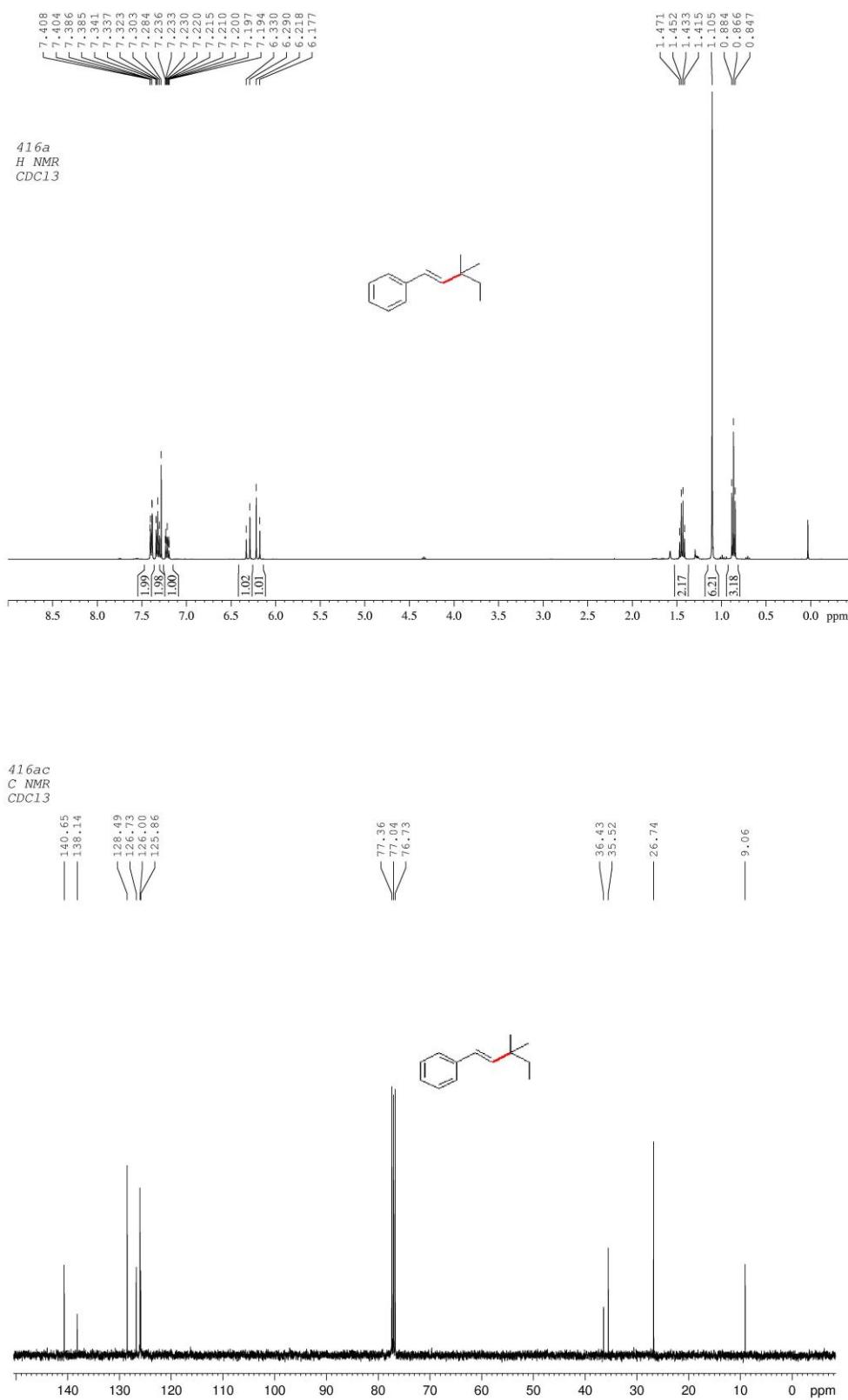


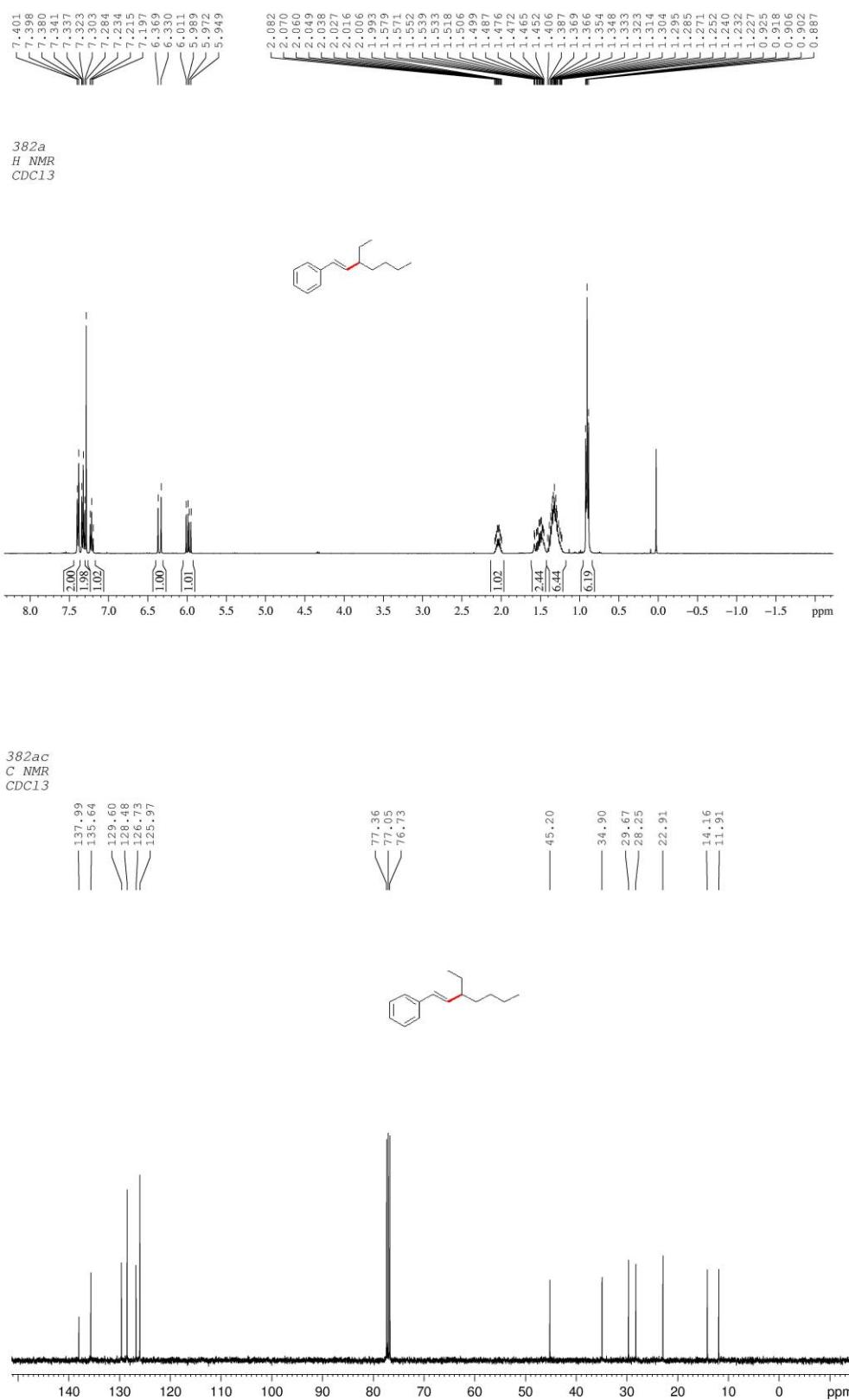


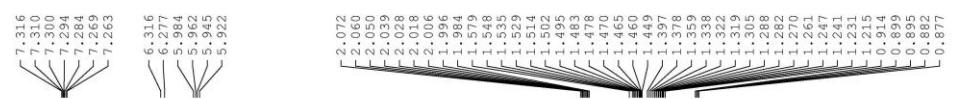




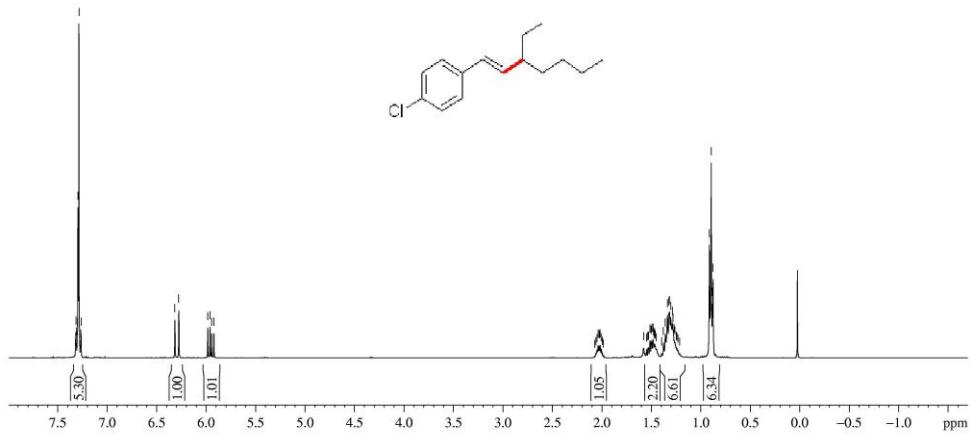




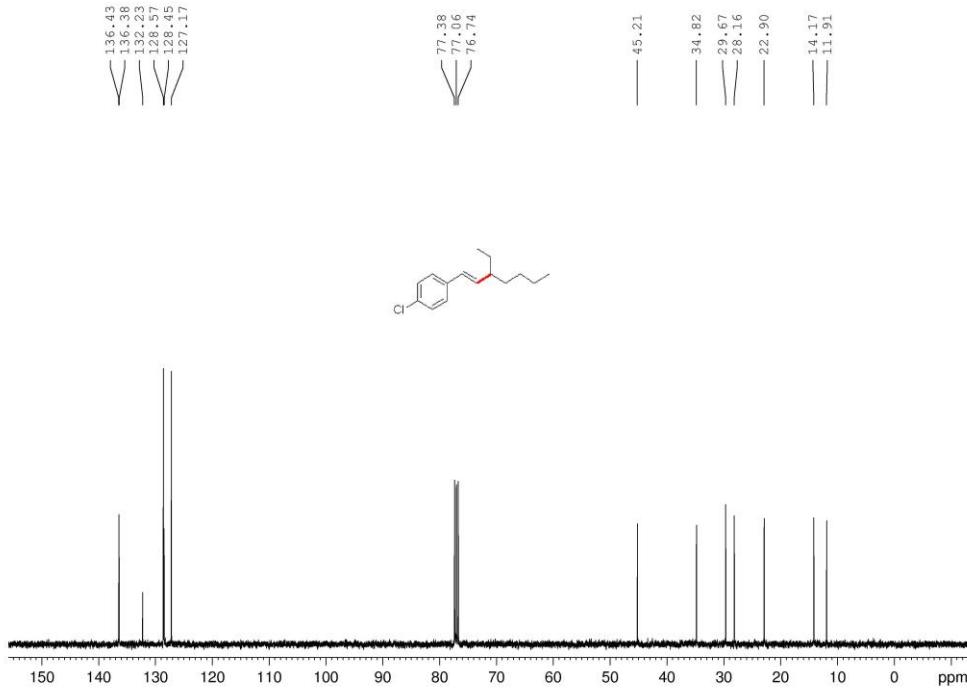


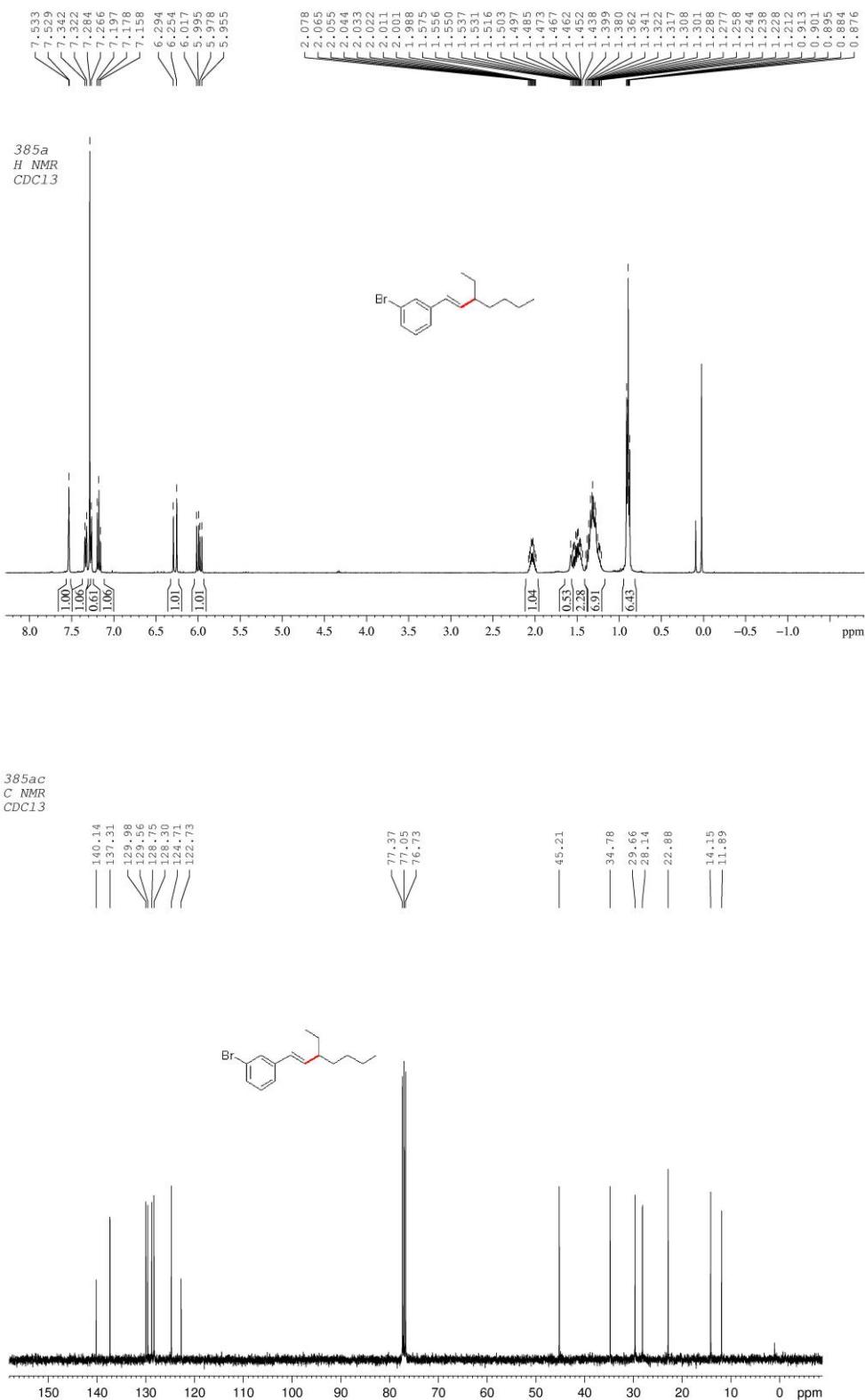


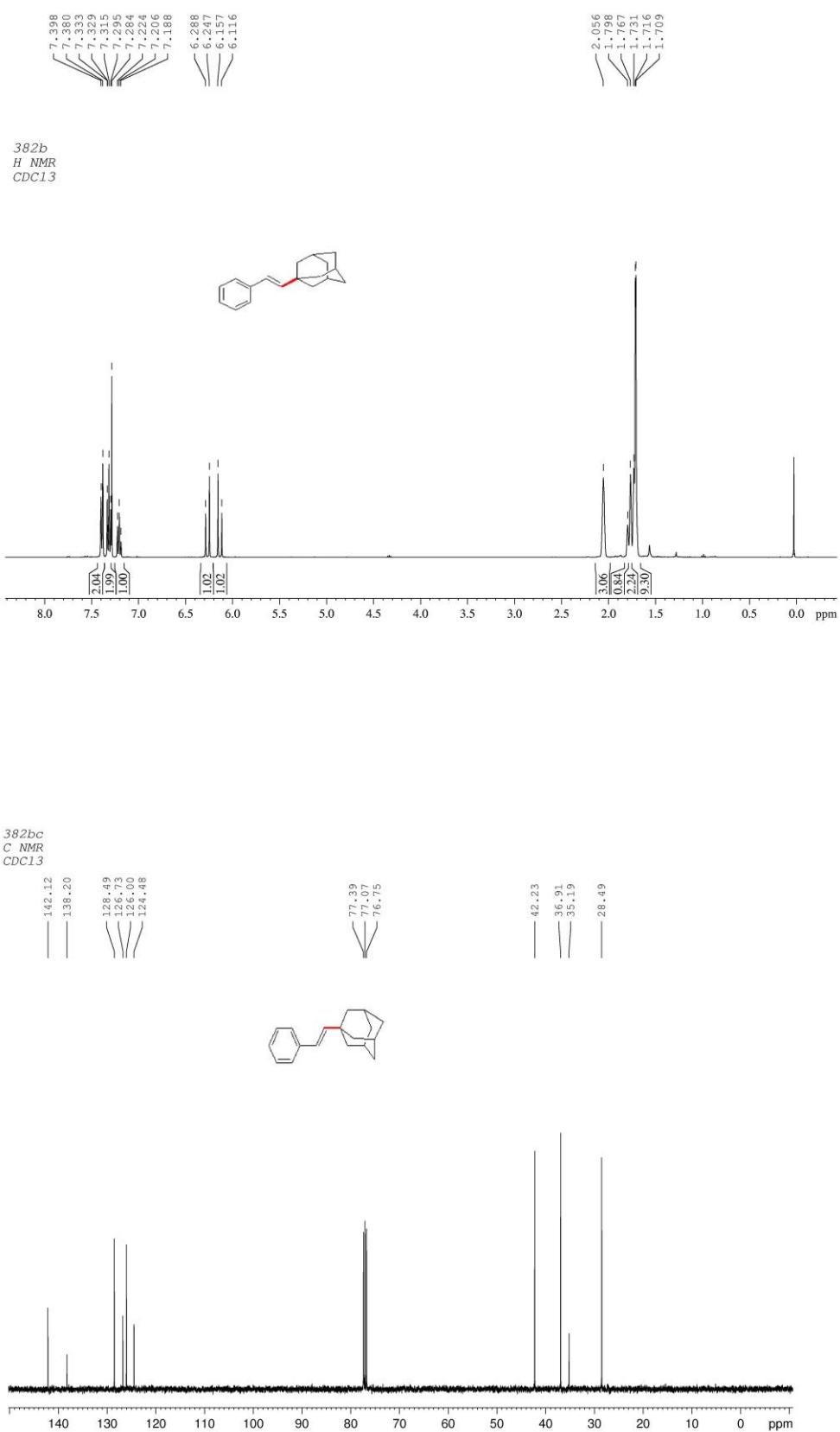
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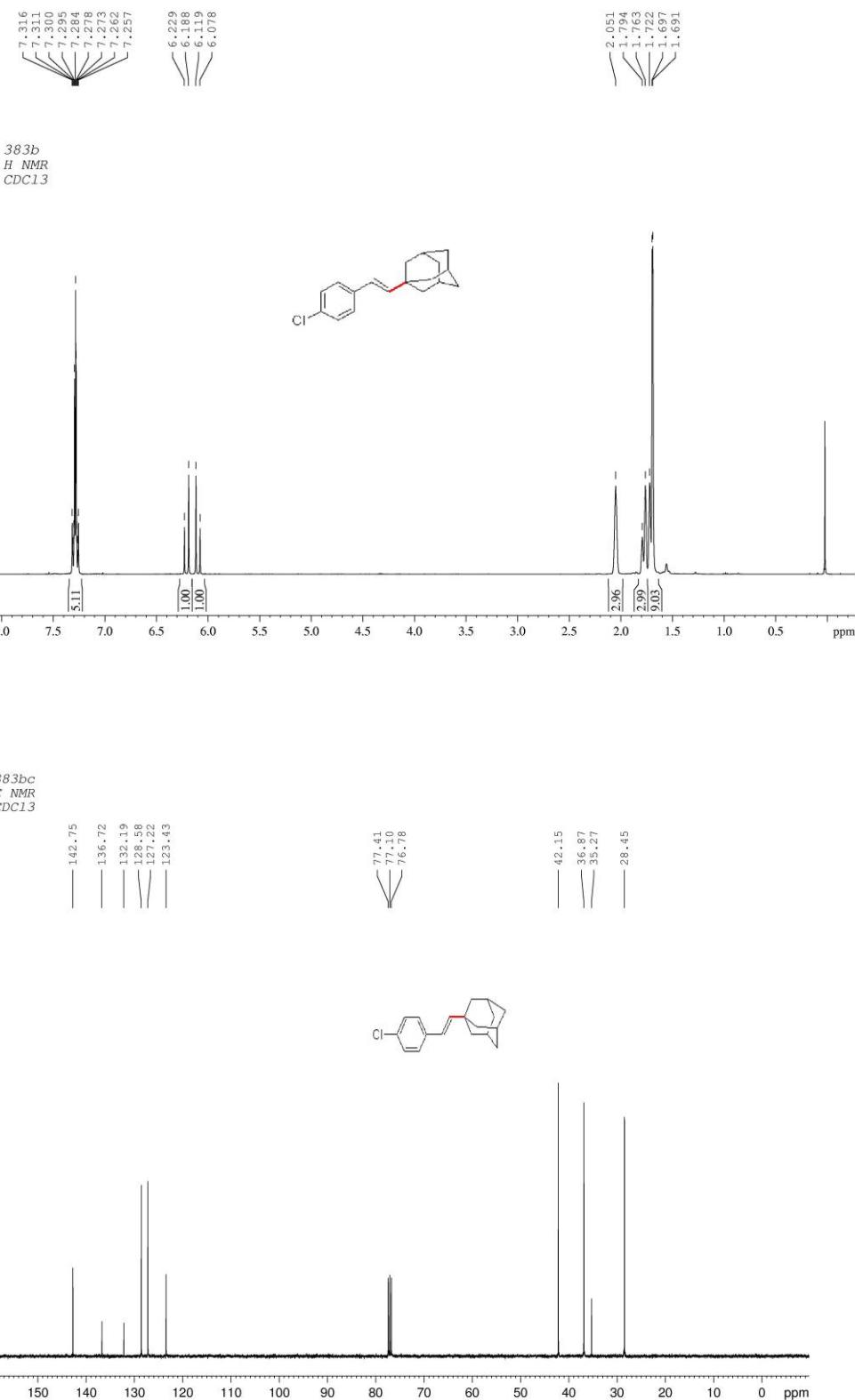


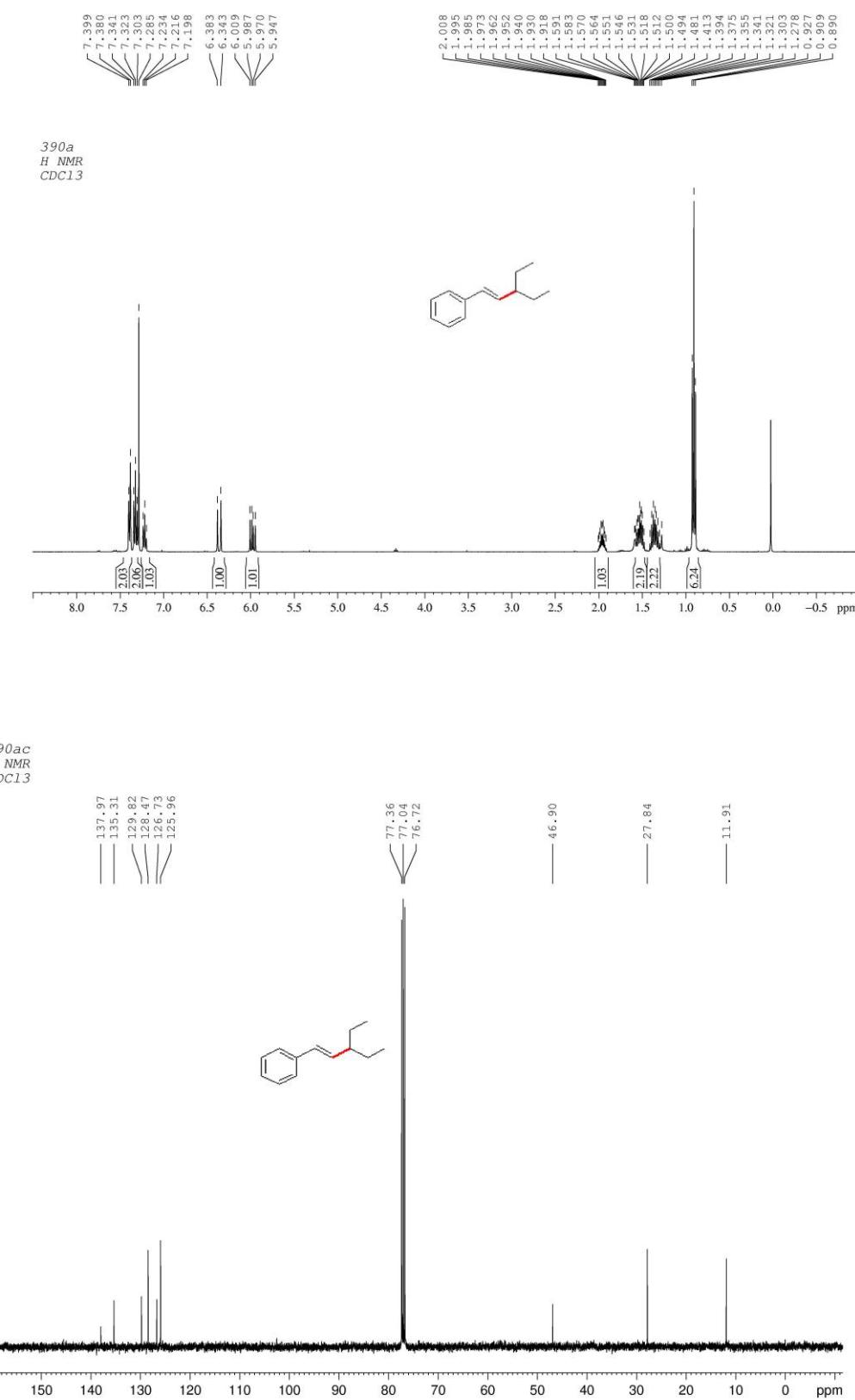
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 $C$  NMR  
*CDCl*<sub>3</sub>

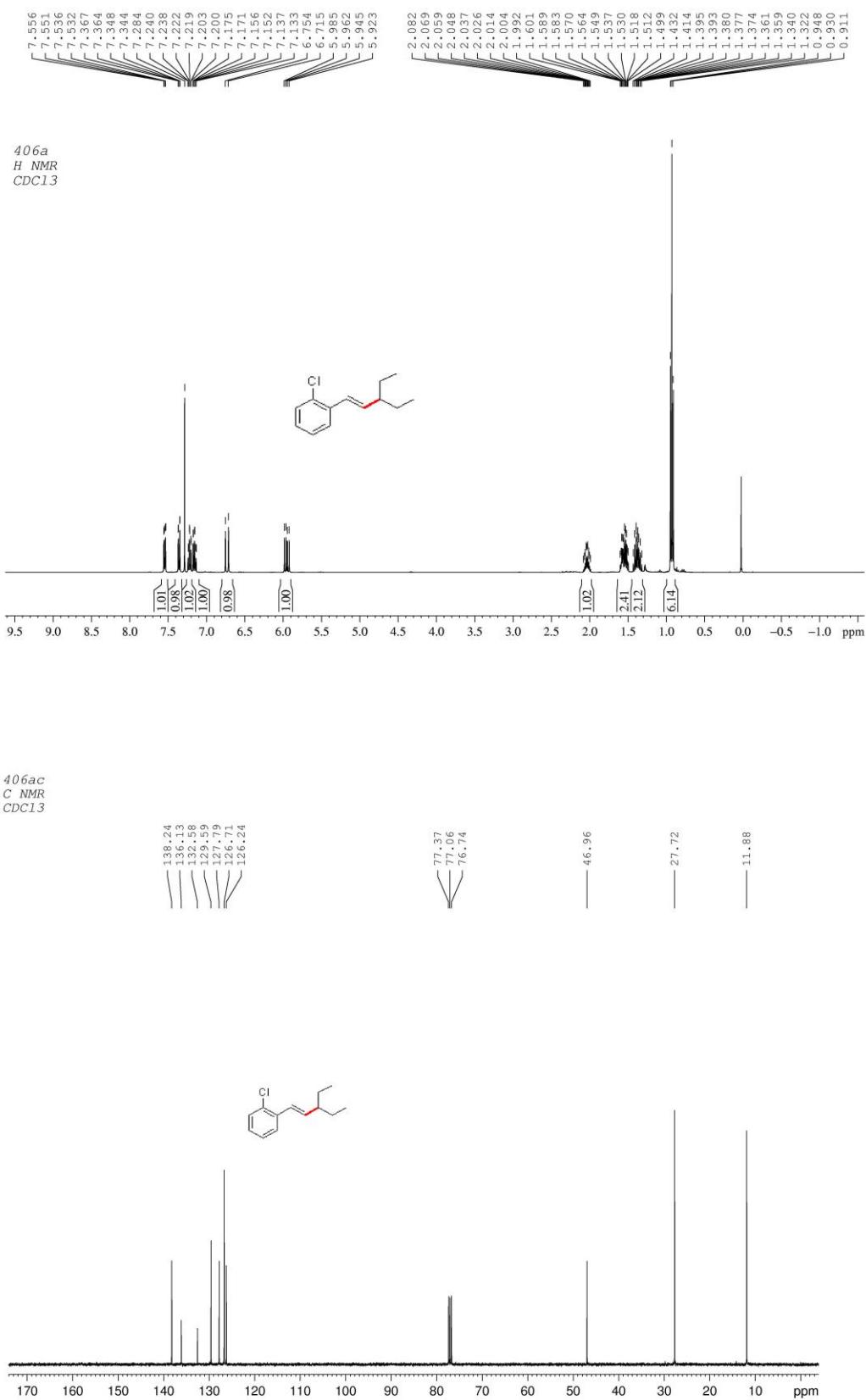


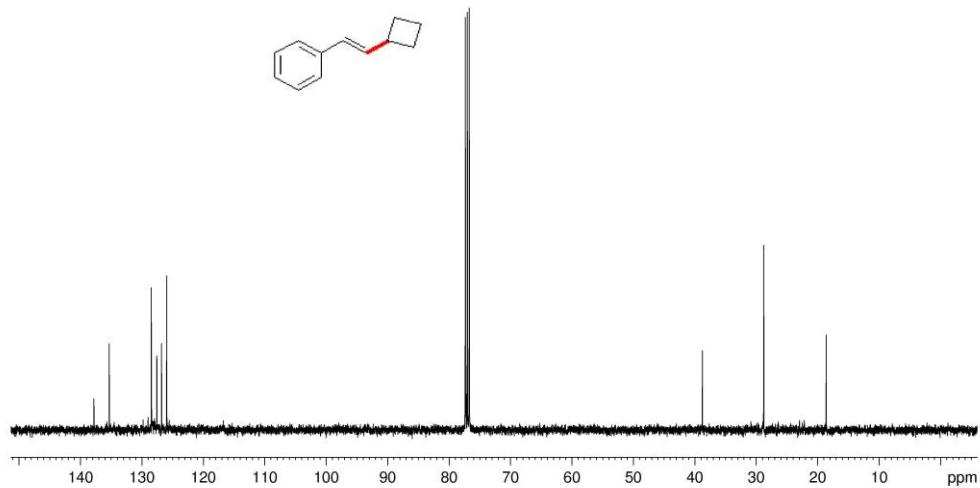
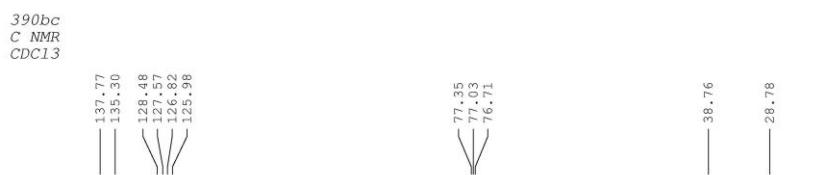
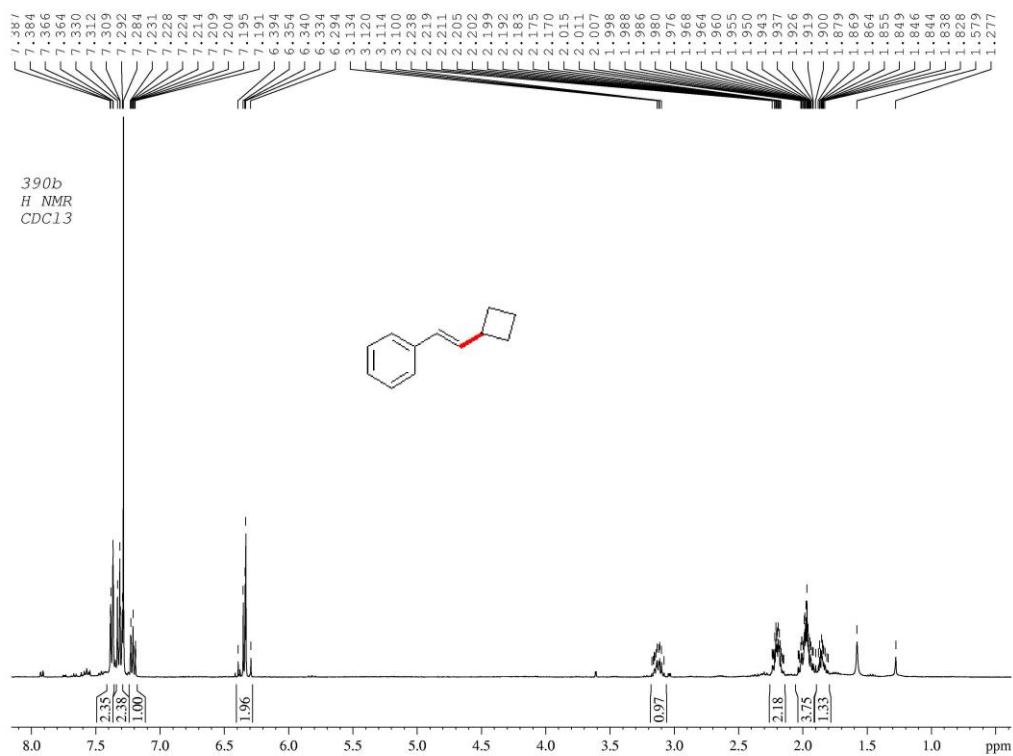


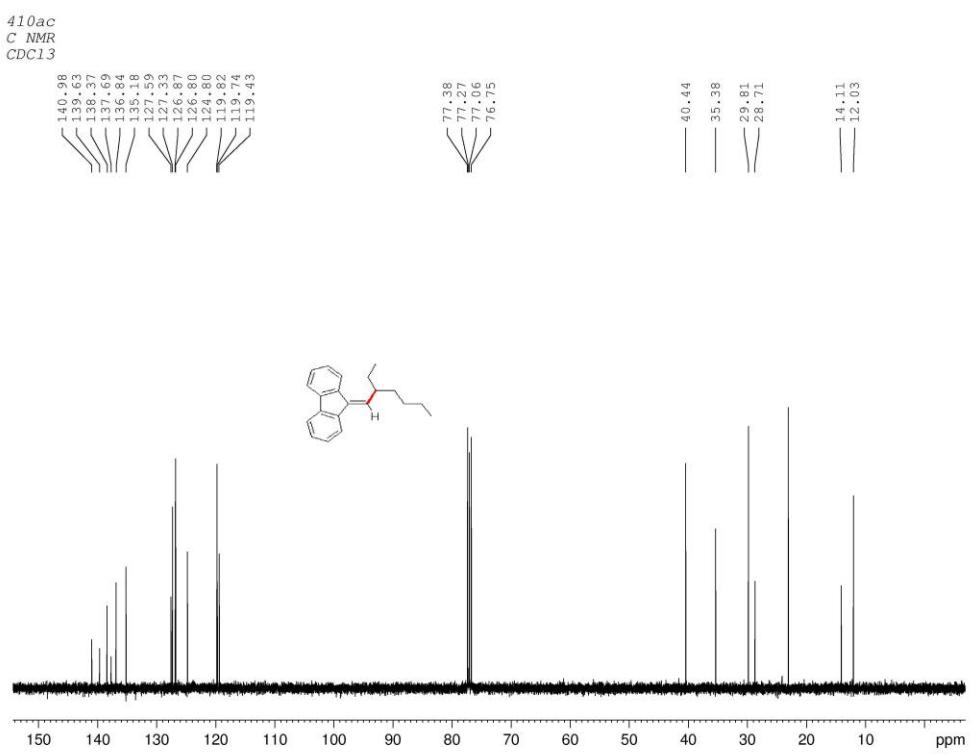
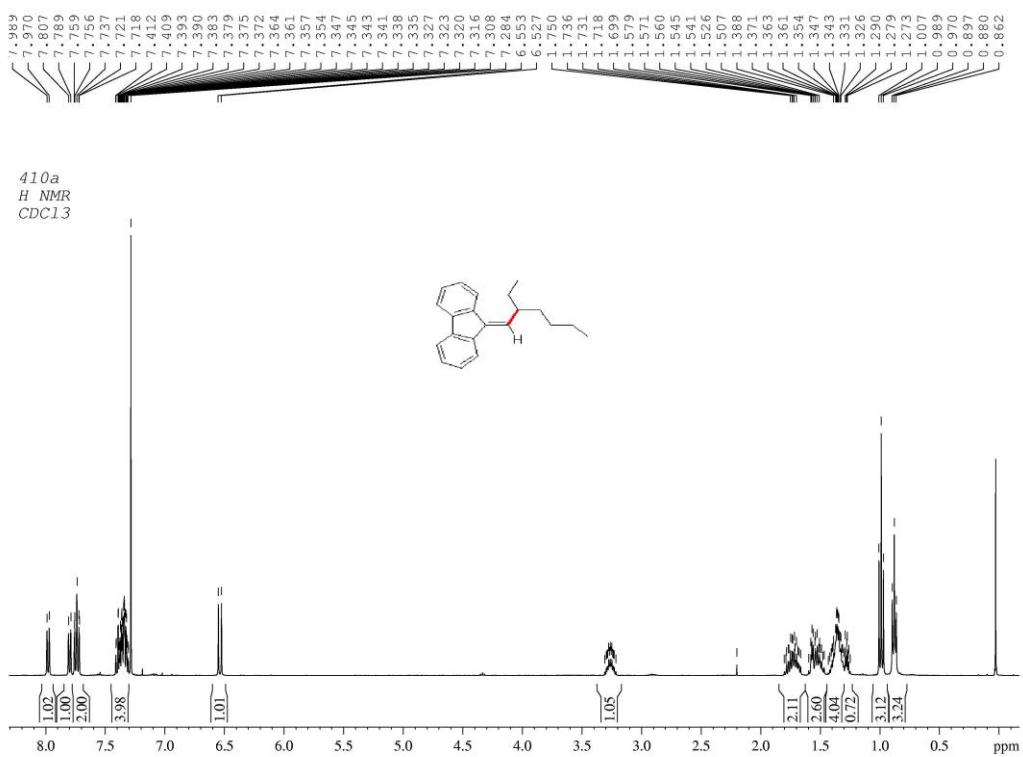


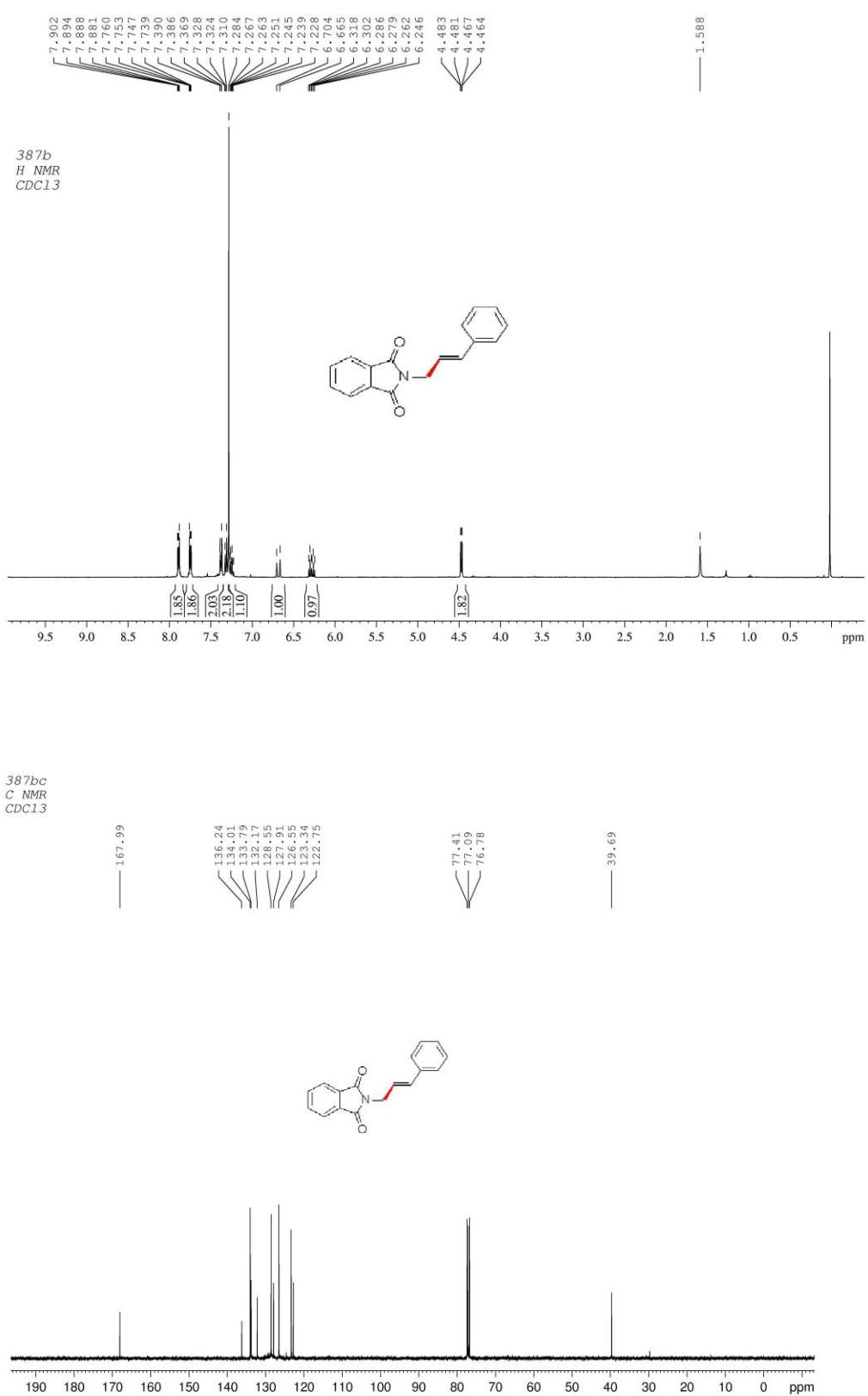


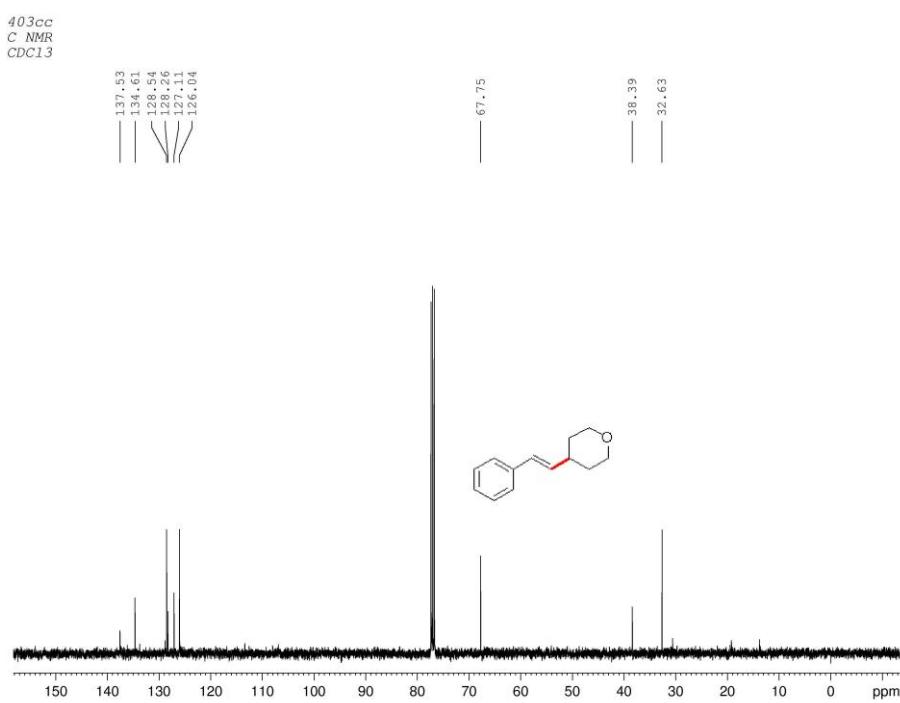
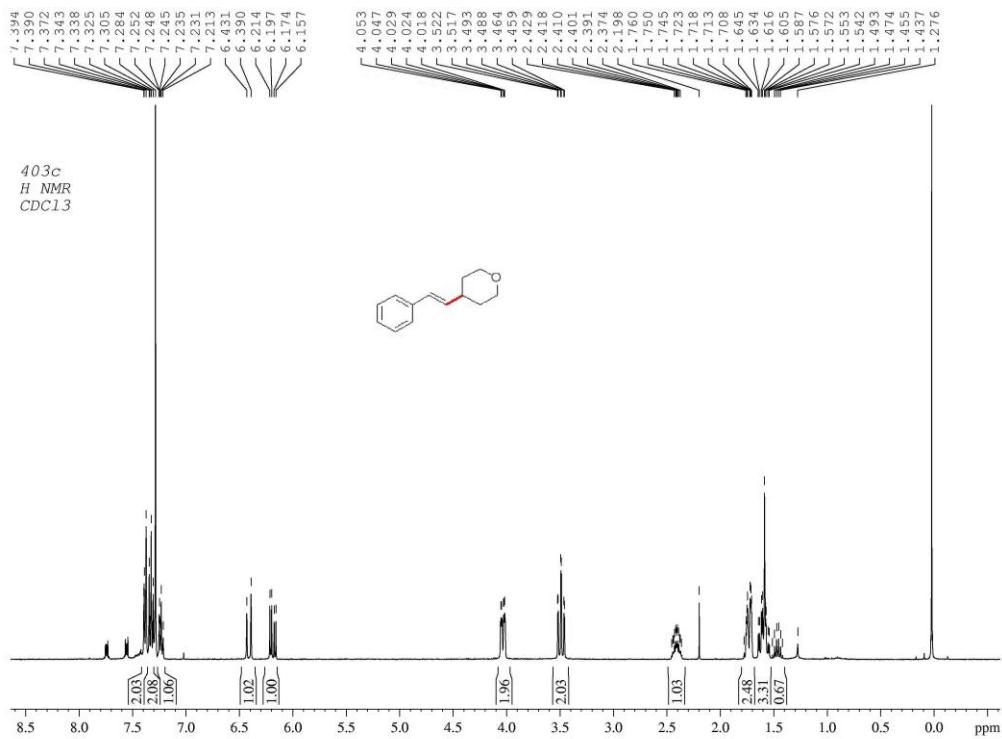


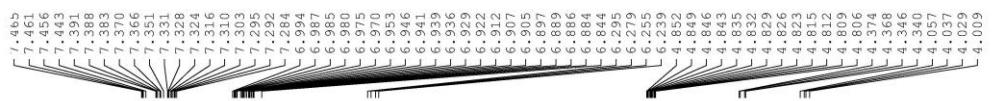




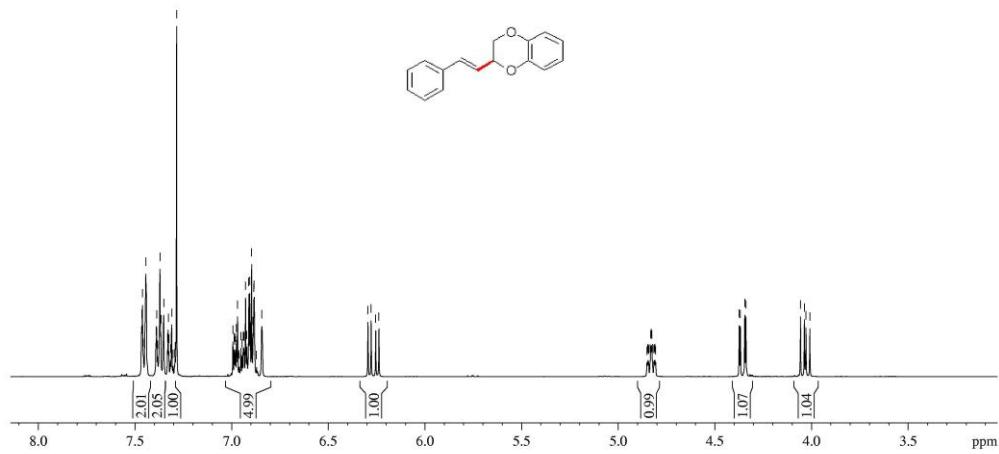
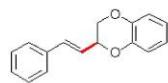




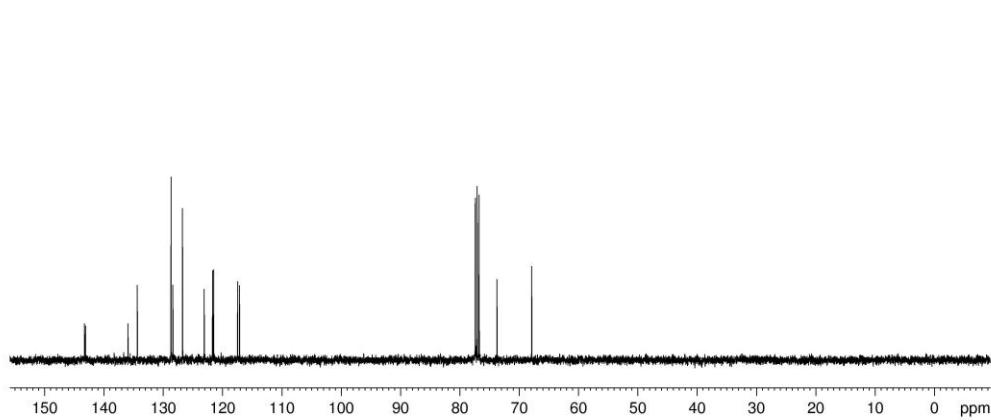
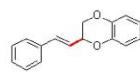
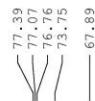


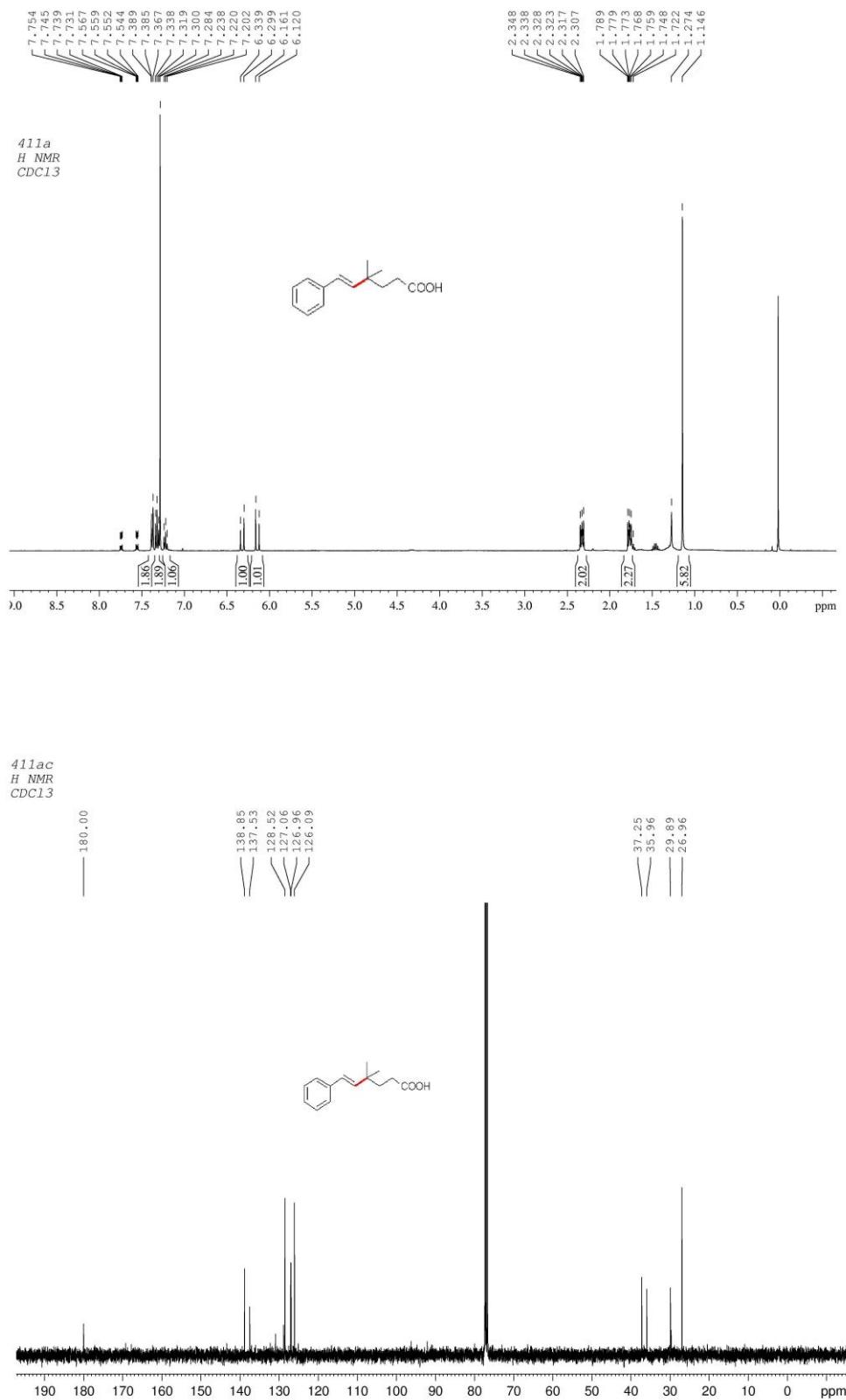


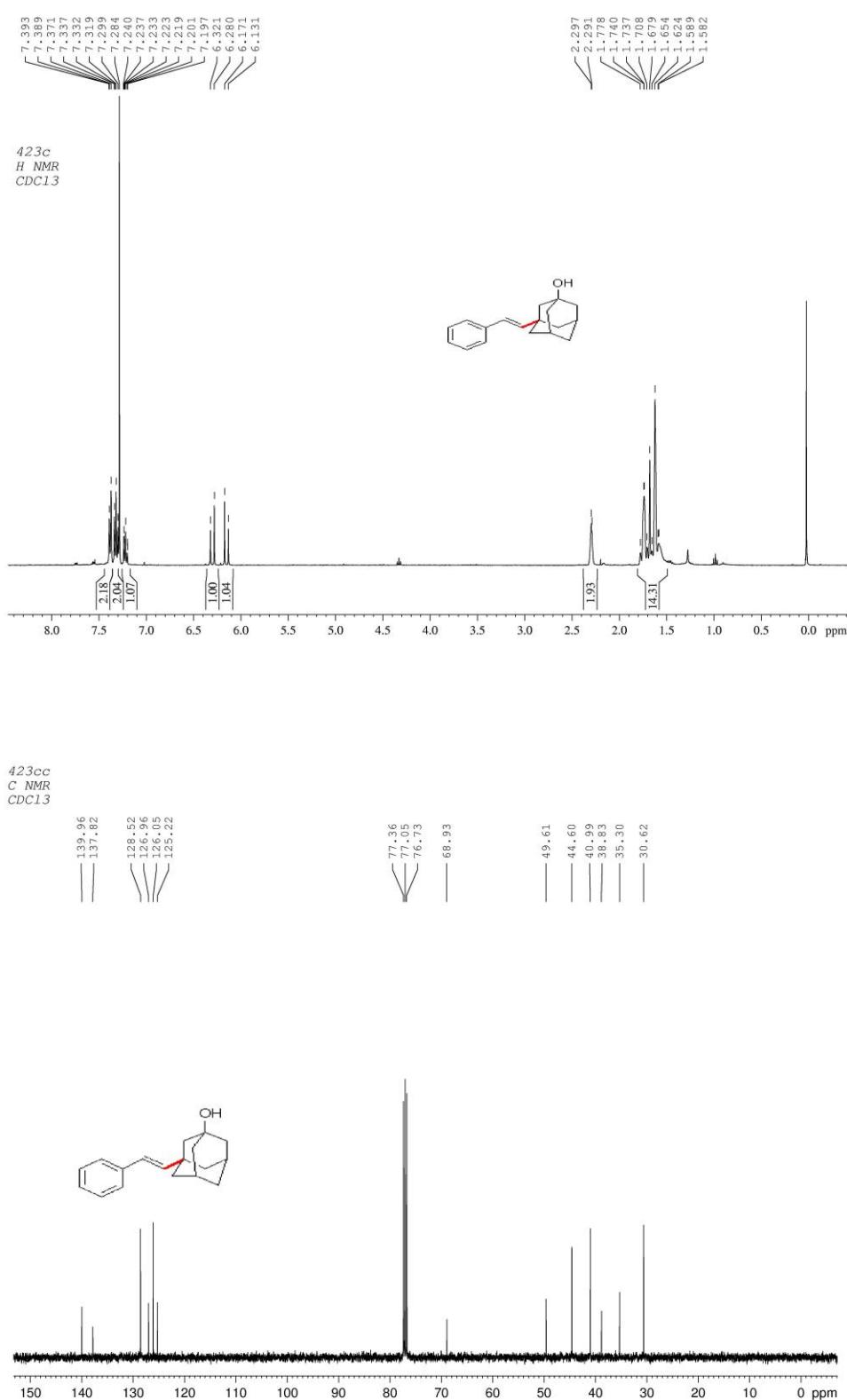
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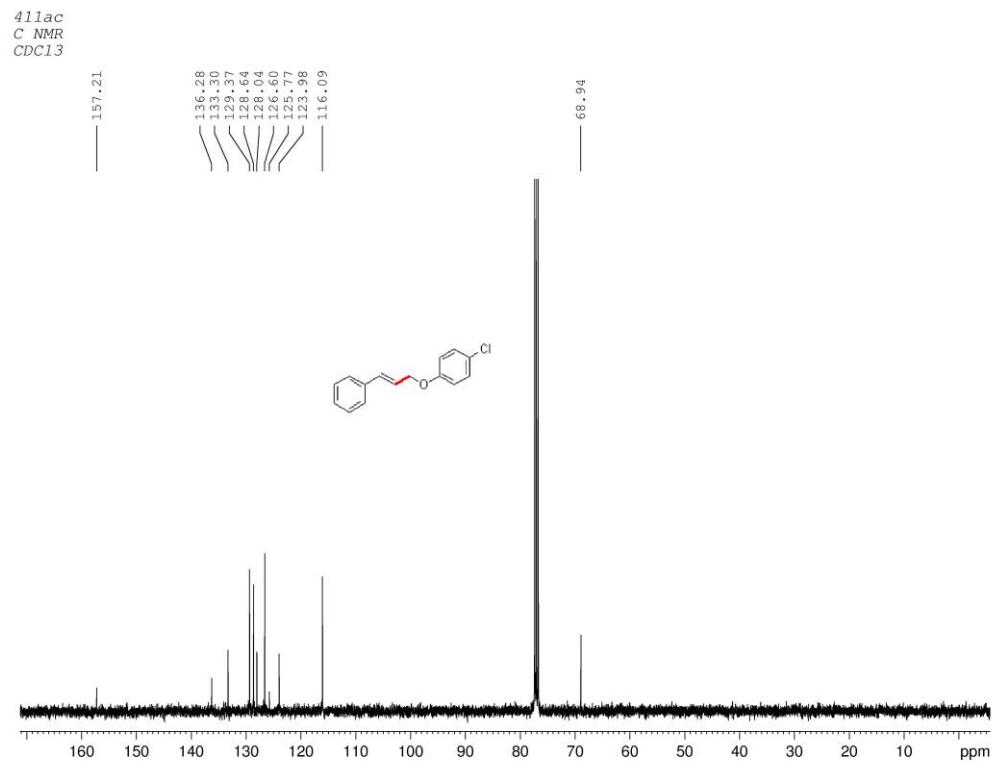
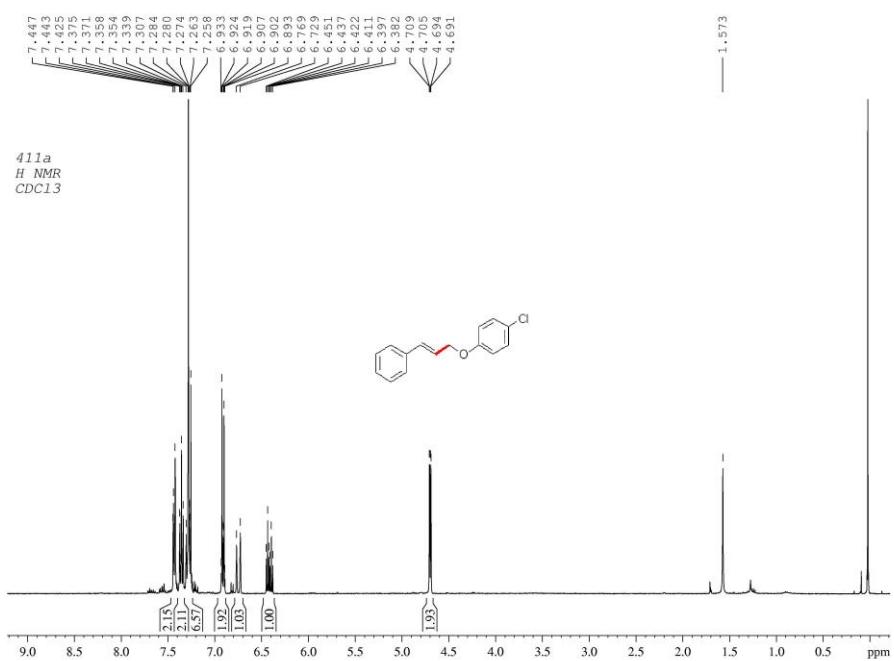


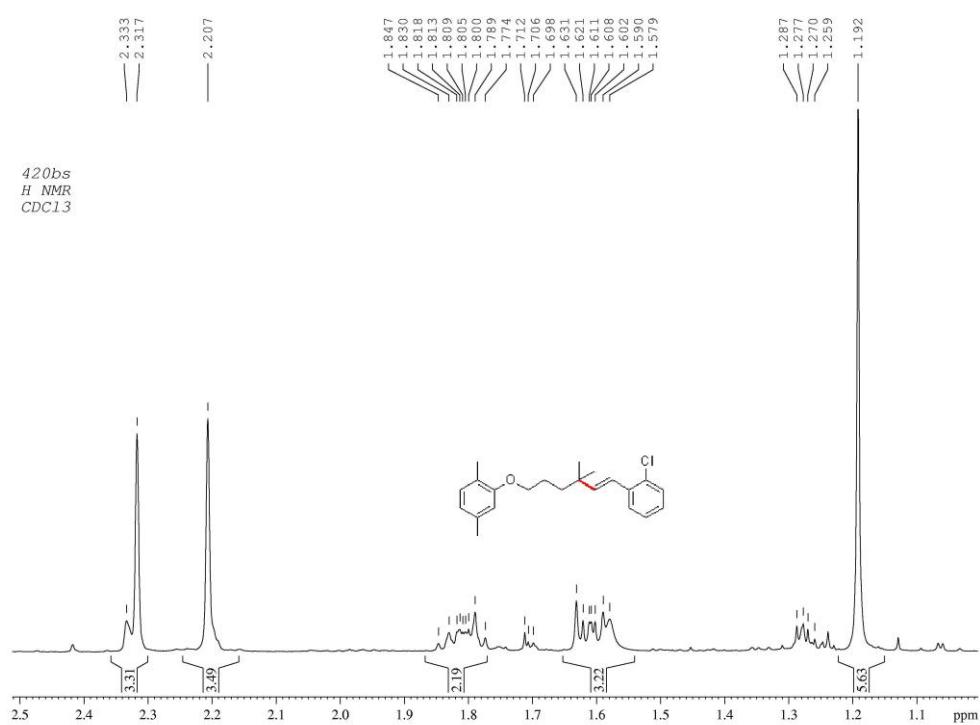
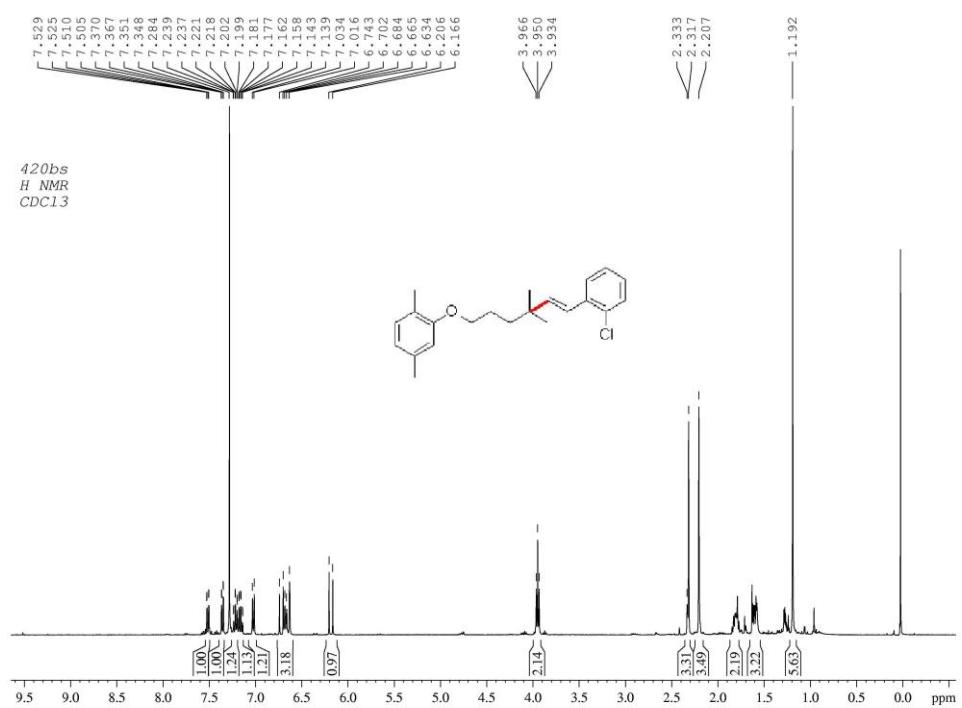
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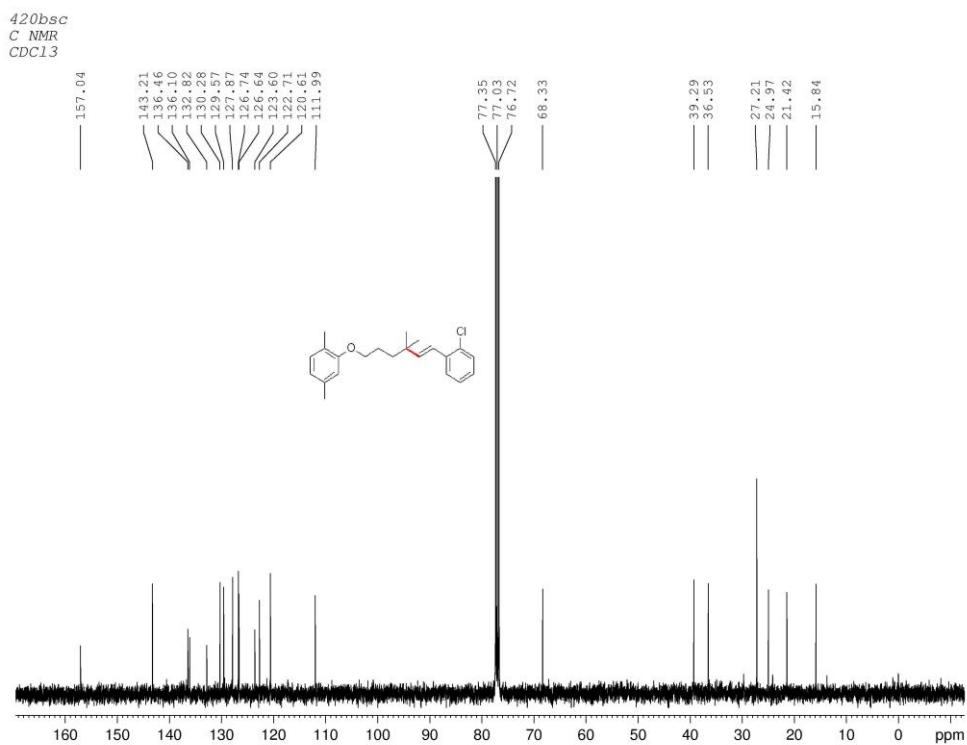


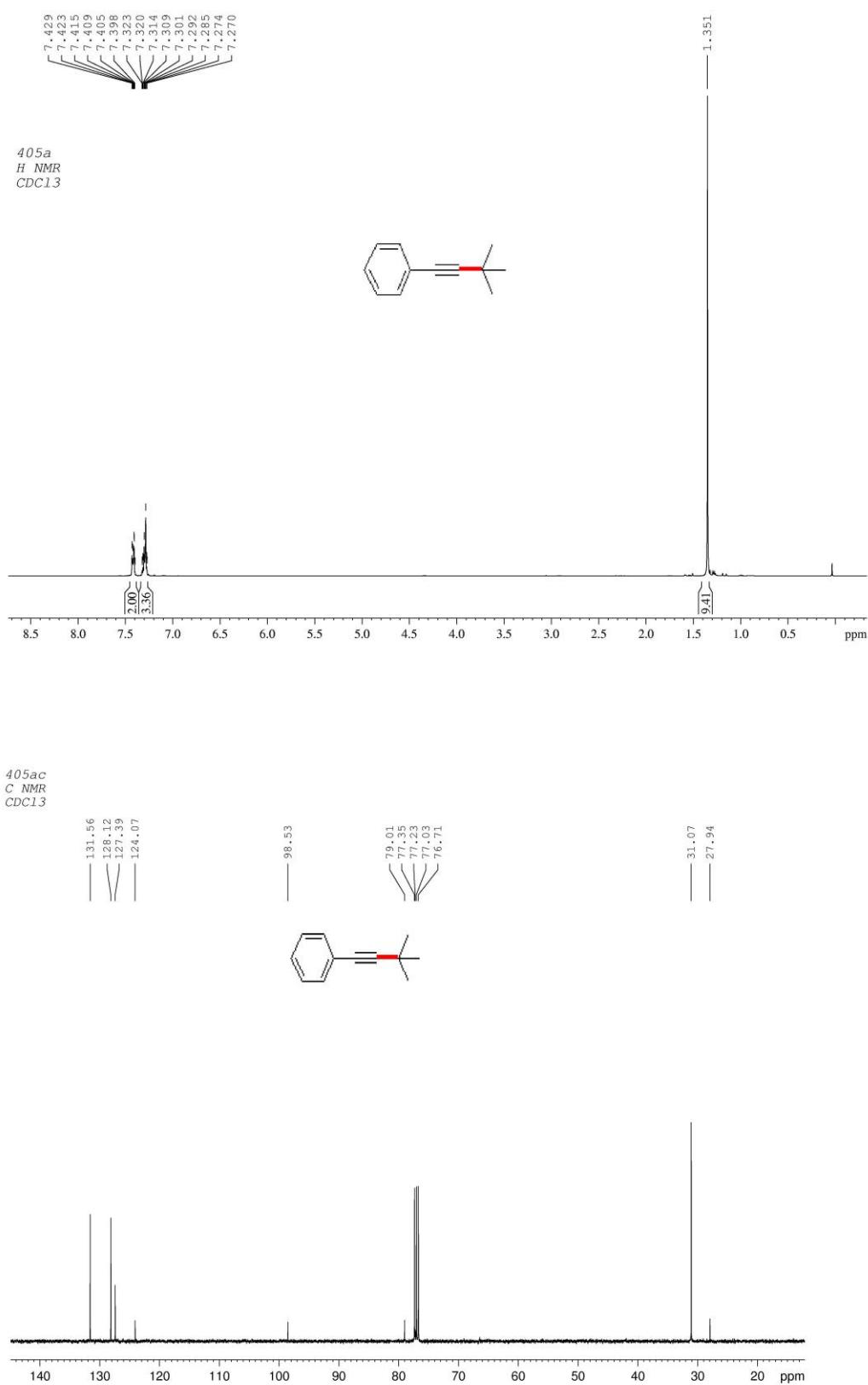


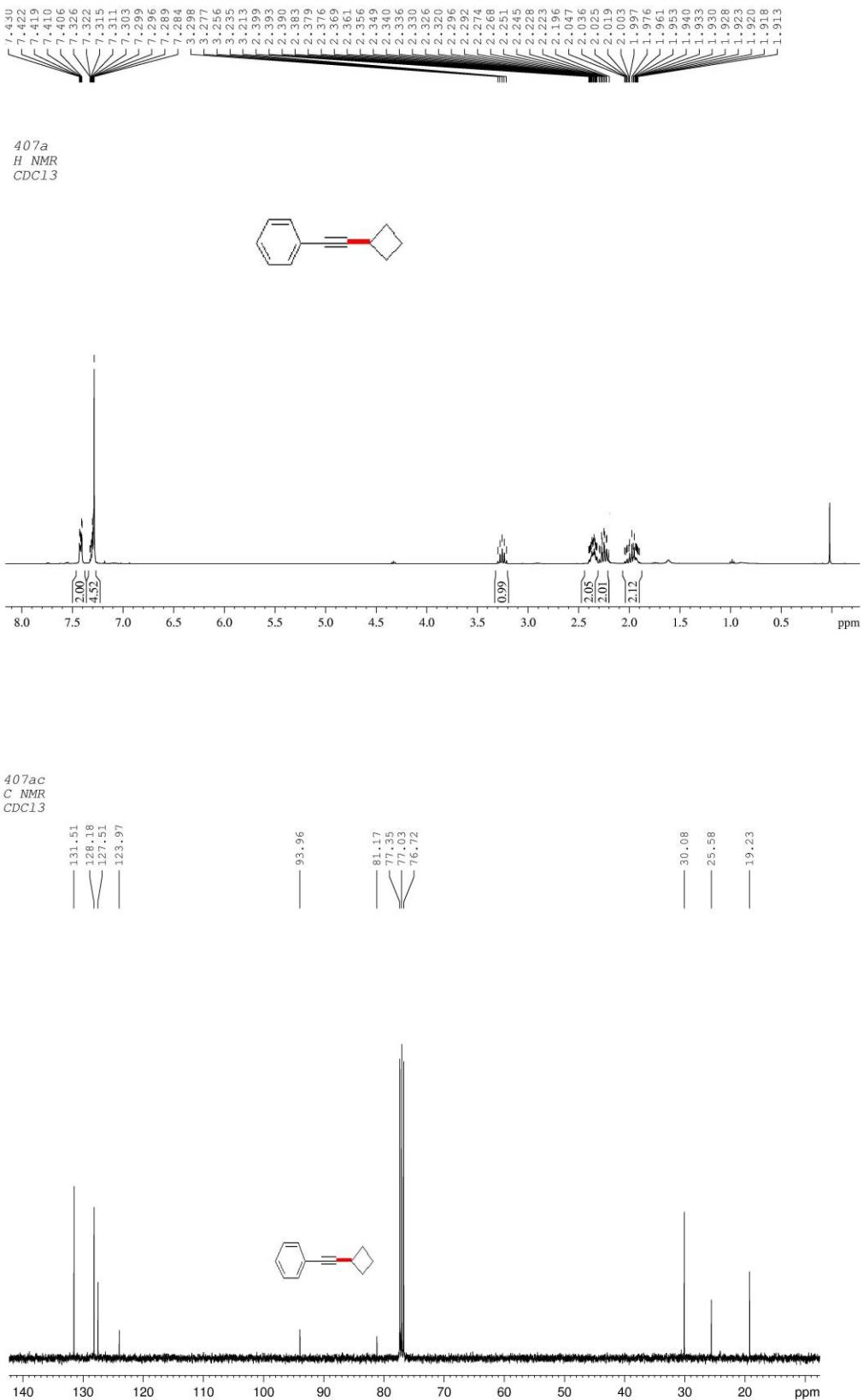


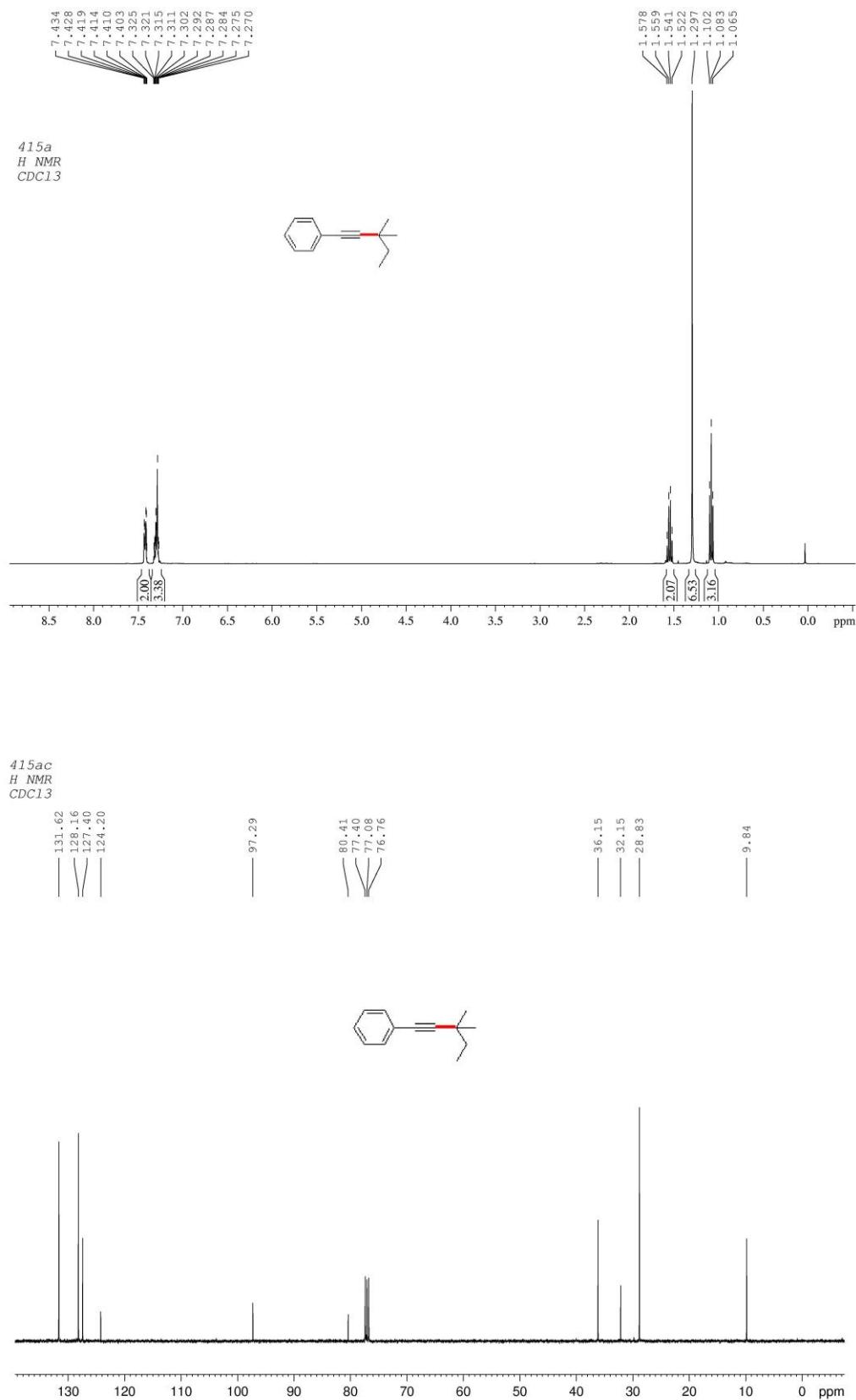


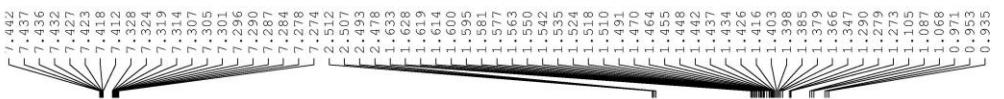




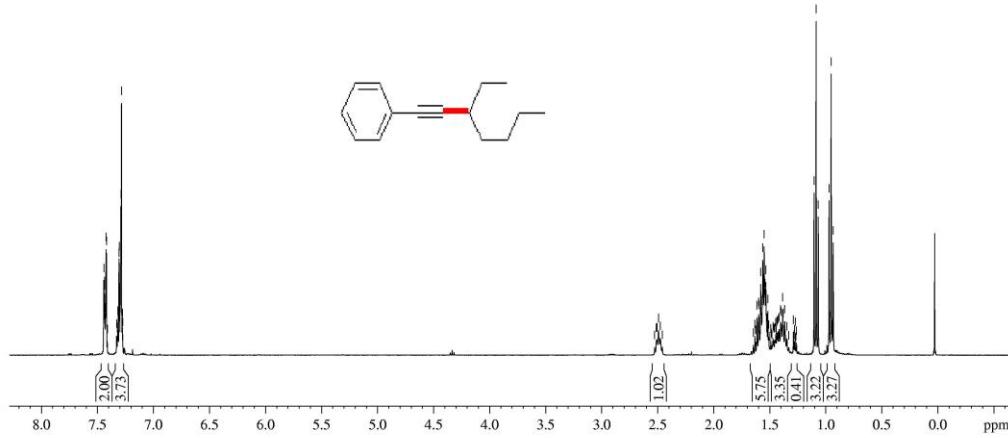
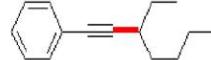








416b  
H NMR  
CDCl<sub>3</sub>



416bc  
C NMR  
CDCl<sub>3</sub>

