

## Supporting Information for

### Reactions of [60]Fullerene with Alkynes promoted by OH<sup>-</sup>

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## Experimental Section

**Chemicals.** All reagents were obtained commercially and used without further purification, unless otherwise specified. CS<sub>2</sub> was distilled over P<sub>2</sub>O<sub>5</sub> at 305 K prior to use.

**Preparation of Compound 2a with C<sub>60</sub>.** Typically, 36 mg of C<sub>60</sub> (50 μmol) and 20 equiv of **1a** (110 μL) was put into mixture of *o*-DCB (20 mL), which was degassed with argon for 15 min under vigorous stirring at 100°C. Then 3 equiv of TBAOH (1.0 M in methanol, 150 μL) was added into the solution. The color of the solution changed instantly from purple to dark-green. Then 5 equiv of CF<sub>3</sub>COOH (19 μL) was added after 1h and the reaction was allowed to proceed for 80 min. The mixture was dried with a rotary evaporator under reduced pressure. The residue was washed with methanol to remove excessive TBAOH and CF<sub>3</sub>COOH. The crude product was put into toluene, and the soluble part was purified using a semi-preparative Buckyprep column (10 mm × 250 mm) eluted with toluene at a flow rate of 3.7 mL/min with the detector wavelength set at 380 nm. Compound **2a** was obtained with an isolated yield of 35.2% (11.11 mg), along with 28.9% of C<sub>60</sub>.

**Spectral Characterizations of Compound 2a.** Positive ESI-ICR HRMS: *m/z* calculated for (C<sub>68</sub>H<sub>6</sub><sup>+</sup>) [M]<sup>+</sup> 822.0464, found: 822.0471; <sup>1</sup>H NMR (600 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>): δ 7.49 (m, 2H), 7.20 (m, 3H), 6.87 (s, 1H); <sup>13</sup>C NMR (125 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>): δ 151.26 (2C), 151.05 (2C), 147.45 (2C), 147.19 (2C), 146.52 (2C), 146.26 (2C), 146.09 (2C), 145.65 (2C), 145.59 (2C), 145.48 (2C), 145.41 (2C), 145.30 (2C), 145.23 (2C), 144.57 (2C), 144.40 (2C), 143.11 (2C), 142.50 (2C), 142.47 (2C), 142.00 (2C), 141.94 (2C), 141.92 (2C), 141.77 (2C), 141.60 (2C), 141.52 (2C), 140.29 (2C), 140.27 (2C), 136.43 (2C), 136.01 (2C), 135.03 (2C), 132.07 (2C), 128.82, 128.49 (2C), 122.52, 92.35, 84.03, 61.76, 54.98; UV-vis (toluene) λ<sub>max</sub> /nm: 326 and 434nm.

**Preparation of Compound 2b.** The procedures were similar to those for preparation of **2a**,

except **1b** (80mg) was used instead of **1a**, and the reaction was allowed to proceed for 1 h after the addition of CF<sub>3</sub>COOH. Compound **2b** was obtained with an isolated yield of 29.0% (8.84 mg), along with 22.3% of C<sub>60</sub>.

**Spectral Characterizations of Compound 2b.** Negative ESI-ICR HRMS: *m/z* calculated for (C<sub>69</sub>H<sub>7</sub>O<sup>+</sup>) [M]<sup>+</sup> 851.0491, found:851.0474; <sup>1</sup>H NMR (500 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>) δ 7.39 (d, *J* = 8.5 Hz, 2H), 6.85 (s, 1H), 6.67 (d, *J* = 8.5 Hz, 2H), 3.64 (s, 3H); <sup>13</sup>C NMR (150 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>) δ 159.70 (2C), 151.18 (2C), 151.14 (2C), 147.23 (2C), 146.97 (2C), 146.32 (2C), 146.04 (2C), 145.86 (2C), 145.44 (2C), 145.39 (2C), 145.33 (2C), 145.30 (2C), 145.24 (2C), 145.18 (2C), 145.06 (2C), 145.00 (2C), 144.35 (2C), 144.20 (2C), 142.88 (2C), 142.26 (2C), 141.79 (2C), 141.72 (2C), 141.69 (2C), 141.60 (2C), 141.38 (2C), 141.29 (2C), 140.07 (2C), 140.01 (2C), 135.84 (2C), 134.72, 133.26 (2C), 114.29, 113.90 (2C), 90.84, 83.97, 61.77, 54.84, 54.55; UV-vis (toluene) λ<sub>max</sub> /nm: 327 and 434 nm.

**Preparation of Compound 2c.** The procedures were similar to those for preparation of **2a**, except **1c** (12.6mg) was used instead of **1a**, and the reaction was allowed to proceed for 1 h after the addition of CF<sub>3</sub>COOH. Compound **2c** was obtained with an isolated yield of 31.7% (11.38 mg), along with 21.1% of C<sub>60</sub>.

**Spectral Characterizations of Compound 2c.** Positive ESI-ICR HRMS: *m/z* calculated for (C<sub>70</sub>H<sub>6</sub><sup>+</sup>) [M]<sup>+</sup> 846.0464, found:846.0456; <sup>1</sup>H NMR (500 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>) δ 7.46 (d, *J* = 6.7 Hz, 2H), 7.29 (d, *J* = 8.2 Hz, 2H), 6.87 (s, 1H), 2.94 (s, 1H); <sup>13</sup>C NMR (125 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>) δ 151.11 (2C), 150.74 (2C), 147.47 (2C), 147.20 (2C), 146.49 (2C), 146.30 (2C), 146.27 (2C), 146.12 (2C), 146.10 (2C), 145.63 (2C), 145.62 (2C), 145.42 (2C), 145.41 (2C), 145.32 (2C), 145.24 (2C), 144.57 (2C), 144.39 (2C), 143.12 (2C), 142.53 (2C), 142.49 (2C), 141.99 (2C), 141.95 (2C), 141.92 (2C), 141.74 (2C), 141.61 (2C), 141.54 (2C), 140.31 (2C), 135.96 (2C), 135.08 (2C), 132.24 (2C), 131.85 (2C), 123.04, 122.72, 94.21, 83.43, 83.33, 80.01, 61.79, 54.98; UV-vis

(toluene)  $\lambda_{\text{max}}$ : 327 and 434 nm.

**Preparation of Compound 2d.** The procedures were similar to those for preparation of **2a**, except 20 equiv of **1d** (100 $\mu$ L) was used instead of **1a**, and the reaction was allowed to proceed for 1 h after the addition of PhCH<sub>2</sub>Br. Compound **2d** was obtained with an isolated yield of 31.5% (8.95 mg), along with 16.4% of C<sub>60</sub>.

**Spectral Characterizations of Compound 2d:** Positive ESI-ICR MS:  $m/z$  calculated for (C<sub>75</sub>H<sub>12</sub><sup>+</sup>) [M]<sup>+</sup> 912.0933, found: 912.0950; <sup>1</sup>H NMR (600 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>)  $\delta$  7.47(m, 2H), 7.21-7.16 (m, 3H), 4.97 (s, 1H); <sup>13</sup>C NMR (150 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>)  $\delta$  153.18 (2C), 152.84 (2C), 147.09 (2C), 146.96 (2C), 145.83 (2C), 145.79 (2C), 145.68 (2C), 145.63 (2C), 145.52 (2C), 145.22 (2C), 144.88 (2C), 144.74 (2C), 144.71 (2C), 144.38 (2C), 143.98 (2C), 143.95 (2C), 142.46 (2C), 142.00 (2C), 141.95 (2C), 141.58 (2C), 141.49 (2C), 141.34 (2C), 141.16 (2C), 141.11 (2C), 140.62 (2C), 139.66 (2C), 138.20 (2C), 136.14 (2C), 134.02 (2C), 133.58, 131.47 (2C), 131.25 (2C), 128.28, 128.00 (2C), 127.60 (2C), 126.65, 122.10, 87.79, 86.47, 65.79, 60.03, 49.92; UV-vis (toluene)  $\lambda_{\text{max}}$  /nm: 327 and 434nm

X-ray Crystal data for **2d**: Empirical formula: C<sub>75</sub> H<sub>12</sub>; Formula weight: 912.85; Crystal system, space group: Monoclinic, P2(1)/c; Unit cell dimensions: a= 20.5241(16) Å, b= 10.0720(8)Å, c= 19.8797(16)Å,  $\alpha$ =90.00°,  $\beta$ = 115.3050°,  $\gamma$ =90.00°; Volume: 3715.2(5) Å<sup>3</sup>; Calculated density: 1.632 Mg m<sup>-3</sup>; Crystal size: 0.32 x 0.18 x 0.13 mm; Reflections collected: 23317; Max. and min. transmission: 0.9879 and 0.9706; Final R indices [ $I > 2\sigma(I)$ ]: R<sub>1</sub> = 0.0516, wR<sub>2</sub> = 0.0843; R indices (all data): R<sub>1</sub> = 0.1342, wR<sub>2</sub> = 0.1083.

**Preparation of Compound 7a and 7b.** Typically, 36 mg of C<sub>60</sub> (50 $\mu$ mol) and 10 equiv of **7** (50 $\mu$ L) was put into mixture of *o*-DCB (20mL), which was degassed with argon for 15 min under vigorous stirring at 25°C. Then 3 equiv of TBAOH (1.0 M in methanol, 150 $\mu$ L) was added into the

solution. The color of the solution changed instantly from purple to dark-green. Then 5 equiv of CF<sub>3</sub>COOH (19 μL) was added after 1h and the reaction was allowed to proceed for 80 min. The mixture was dried with a rotary evaporator under reduced pressure. The residue was washed with methanol to remove excessive TBAOH and CF<sub>3</sub>COOH. The crude product was put into toluene, and the soluble part was purified using a semi-preparative Buckyprep column (10 mm × 250 mm) eluted with toluene at a flow rate of 3.7 mL/min with the detector wavelength set at 380 nm. Compound **7a** and **7b** was obtained with an isolated yield of 35.2% and 11.8% respectively, along with 28.9% of C<sub>60</sub>.

**Spectral Characterizations of Compound 7a:** Positive ESI-ICR MS: *m/z* calculated for (C<sub>66</sub>H<sub>13</sub>O<sub>4</sub><sup>+</sup>) [M+H]<sup>+</sup> 869.0814, found: 869.6473; <sup>1</sup>H NMR (500 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>) δ 6.97(s, 1H), 5.48 (d, *J* = 7.4 Hz, 1H), 4.33 (d, *J* = 7.4 Hz, 1H), 3.65 (s, 3H), 3.51(s, 3H), 3.48(s, 3H); <sup>13</sup>C NMR (125 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>) δ 169.37, 154.34 (2C), 153.47, 153.22, 152.91, 147.40, 147.06, 146.87, 146.84, 146.48, 146.28, 146.26, 146.22, 146.18, 146.07, 146.03, 145.99 (2C), 145.70, 145.67, 145.54, 145.50, 145.33, 145.26, 145.21, 145.18, 145.17(2C), 144.71, 144.67, 144.39, 144.30, 143.14, 143.08, 142.47, 142.45, 142.44 (2C), 142.38, 142.17, 142.02, 141.92, 141.90, 141.88 (2C), 141.45, 141.40, 141.39, 141.31, 141.25, 140.26, 140.22, 139.49, 139.36, 136.49, 136.43, 135.98, 135.88, 105.31, 63.48, 61.13, 58.16, 55.29, 54.79, 51.74; UV-vis (toluene) λ<sub>max</sub>: 329 and 435 nm.

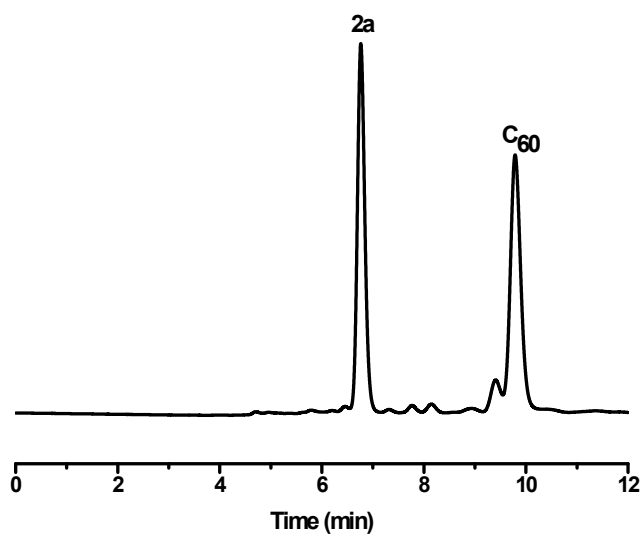
**Spectral Characterizations of Compound 7b:** Negative ESI-ICR MS: *m/z* calculated for (C<sub>67</sub>H<sub>13</sub>O<sub>4</sub><sup>-</sup>) [M-H]<sup>-</sup> 881.0814, found: 881.0799; <sup>1</sup>H NMR (500 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>) δ 6.96(s, 1H), 5.47 (d, *J* = 7.6 Hz, 1H), 4.29 (d, *J* = 7.6 Hz, 1H), 4.10 (q, *J* = 7.4 Hz, 2H), 3.65(s, 3H), 3.49(s, 3H), 1.15 (t, *J* = 7.4 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>) δ 168.83, 154.39 (2C), 153.50, 153.28, 152.92, 147.39, 147.05, 147.00, 146.88, 146.84, 146.63, 146.26, 146.22, 146.18, 146.06, 146.02, 145.98, 145.70, 145.68, 145.65, 145.53, 145.32, 145.23, 145.20 (2C), 145.16 (2C),

144.70, 144.66, 144.39, 144.30, 143.14, 143.07, 142.44 (2C), 142.38, 142.14, 142.01, 141.90 (2C), 141.45, 141.39, 141.36, 141.30, 141.24, 140.25, 140.21, 139.41, 139.27, 136.49, 136.43, 136.00, 135.93, 129.01, 128.21, 105.33, 63.56, 61.13 (2C), 60.94 (2C), 58.24, 55.34, 54.72, 14.57; UV-vis (toluene)  $\lambda_{\text{max}}$ : 329 and 435 nm.

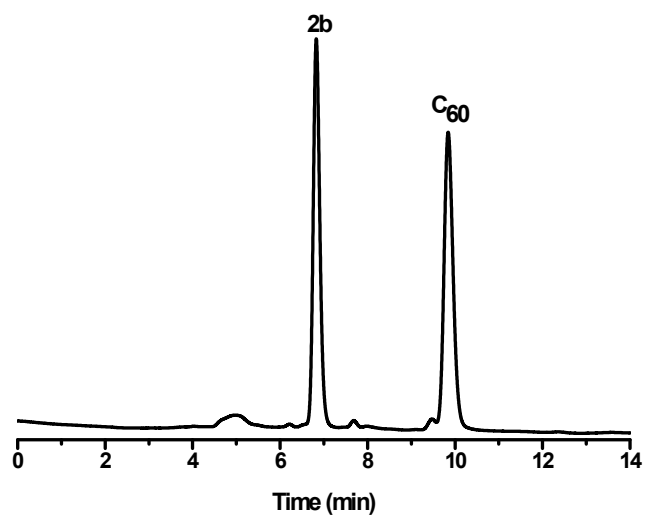
**Preparation of Compound 7c.** The procedures were similar to those for preparation of 7, except TBAOH (1.0 M in methanol) was used instead of TBAOH (1.0 M in isopropanol, 389 $\mu$ L), and the reaction was allowed to proceed for 1 h after the addition of CF<sub>3</sub>COOH. Compound 7c was obtained with an isolated yield of 27.46% (8.72 mg), along with 6.9% of C<sub>60</sub>.

**Spectral Characterizations of Compound 7c:** Negative ESI-ICR HRMS:  $m/z$  calculated for (C<sub>65</sub>H<sub>6</sub>O<sub>2</sub><sup>-</sup>) [M - H]<sup>-</sup> 817.0290, found:817.0260; <sup>1</sup>H NMR (600 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>)  $\delta$  7.09 (s, 1H), 4.44 (q,  $J$  = 7.2 Hz, 2H), 1.49 (t,  $J$  = 7.2 Hz, 3H); <sup>13</sup>C NMR (150 MHz, CS<sub>2</sub>/ DMSO-*d*<sub>6</sub>)  $\delta$  153.17, 150.63 (2C), 149.12 (2C), 147.75, 147.47, 146.62 (2C), 146.63 (2C), 146.53 (2C), 146.40 (4C), 146.39 (4C), 146.01 (2C), 145.86, 145.65 (4C), 145.50 (4C), 144.82 (2C), 144.52 (2C), 143.35 (2C), 142.81 (2C), 142.75 (2C), 142.21 (2C), 142.20 (2C), 142.19 (2C), 142.17 (2C), 141.82 (2C), 141.80 (2C), 141.71 (2C), 140.65 (2C), 140.52 (2C), 135.85, 135.76, 129.10, 128.32, 88.50, 75.26, 62.46, 61.00, 54.32, 14.51; UV-vis (toluene)  $\lambda_{\text{max}}$ : 327 and 433 nm.

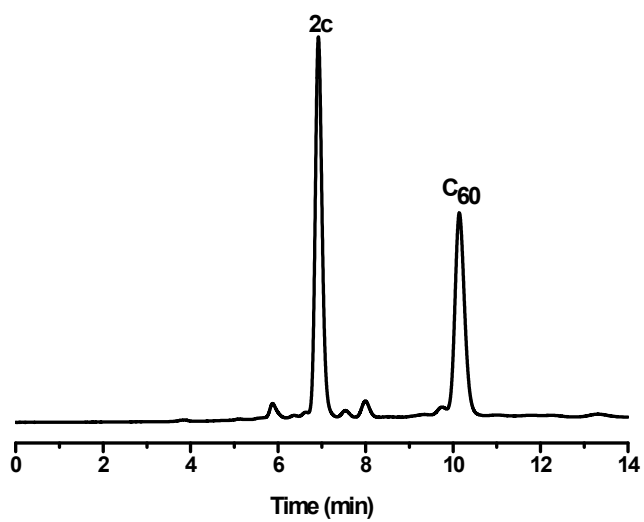




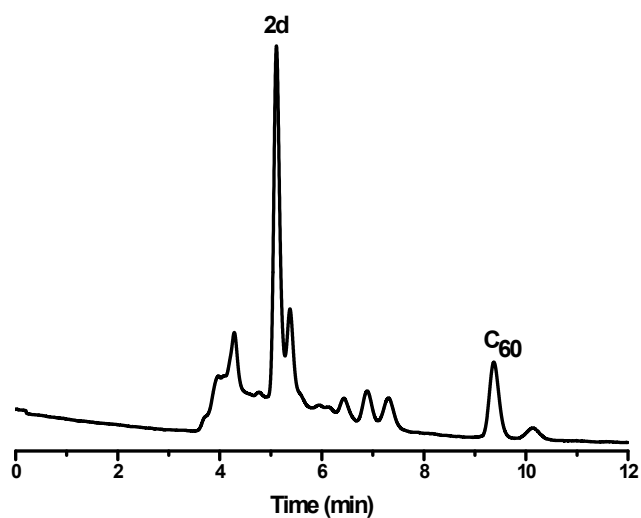
**Figure S1.** HPLC trace of the crude mixture formed from the reaction of **1a** with C<sub>60</sub> in the presence of TBAOH/CH<sub>3</sub>OH at 100 °C. The product was eluted with toluene over a semi-preparative Buckyprep column at a flow rate of 3.7 mL/min with the detector wavelength set at 380 nm.



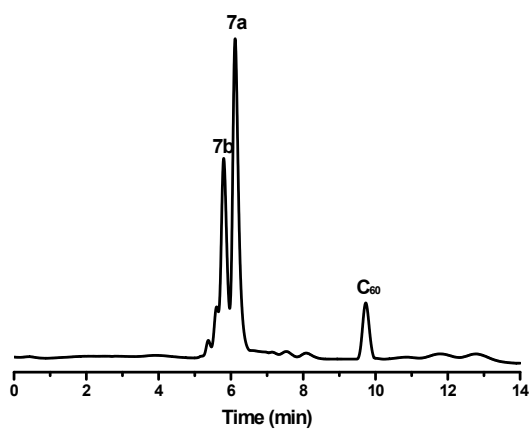
**Figure S2.** HPLC trace of the crude mixture formed from the reaction of **1b** with C<sub>60</sub> in the presence of TBAOH/CH<sub>3</sub>OH at 100 °C. The product was eluted with toluene over a semi-preparative Buckyprep column at a flow rate of 3.7 mL/min with the detector wavelength set at 380 nm.



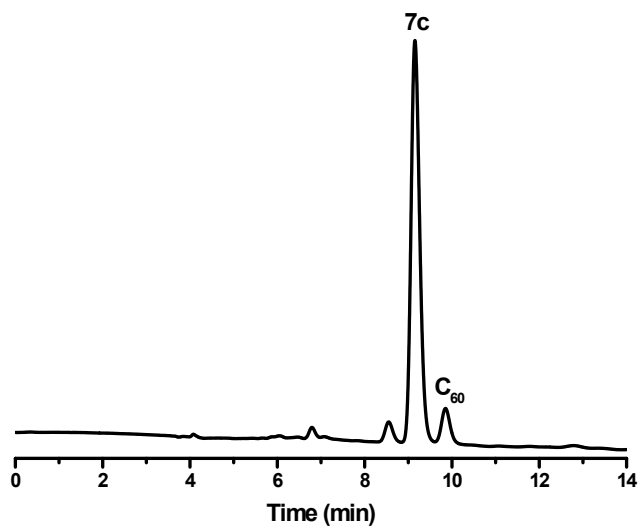
**Figure S3.** HPLC trace of the crude mixture formed from the reaction of **1c** with C<sub>60</sub> in the presence of TBAOH/CH<sub>3</sub>OH at 100 °C. The product was eluted with toluene over a semi-preparative Buckyprep column at a flow rate of 3.7 mL/min with the detector wavelength set at 380 nm.



**Figure S4.** HPLC trace of the crude mixture formed from the reaction of **1d** with C<sub>60</sub> in the presence of TBAOH/CH<sub>3</sub>OH at 100 °C. The product was eluted with toluene over a semi-preparative Buckyprep column at a flow rate of 3.7 mL/min with the detector wavelength set at 380 nm.



**Figure S5.** HPLC trace of the crude mixture formed from the reaction of **7** with C<sub>60</sub> in the presence of TBAOH/CH<sub>3</sub>OH at 100 °C. The product was eluted with toluene over a semi-preparative Buckyprep column at a flow rate of 3.7 mL/min with the detector wavelength set at 380 nm.

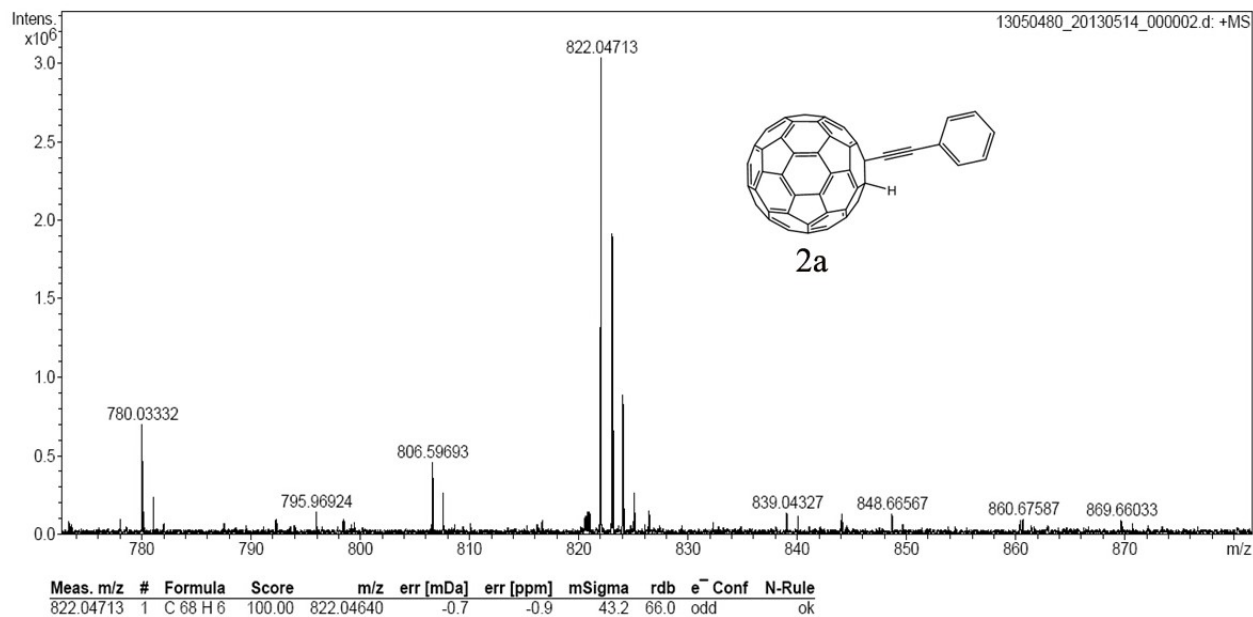


**Figure S6.** HPLC trace of the crude mixture formed from the reaction of **7** with  $C_{60}$  in the presence of 1.0 M TBAOH in isopropanol at 25 °C. The product was eluted with toluene over a semi-preparative Buckyprep column at a flow rate of 3.7 mL/min with the detector wavelength set at 380 nm.

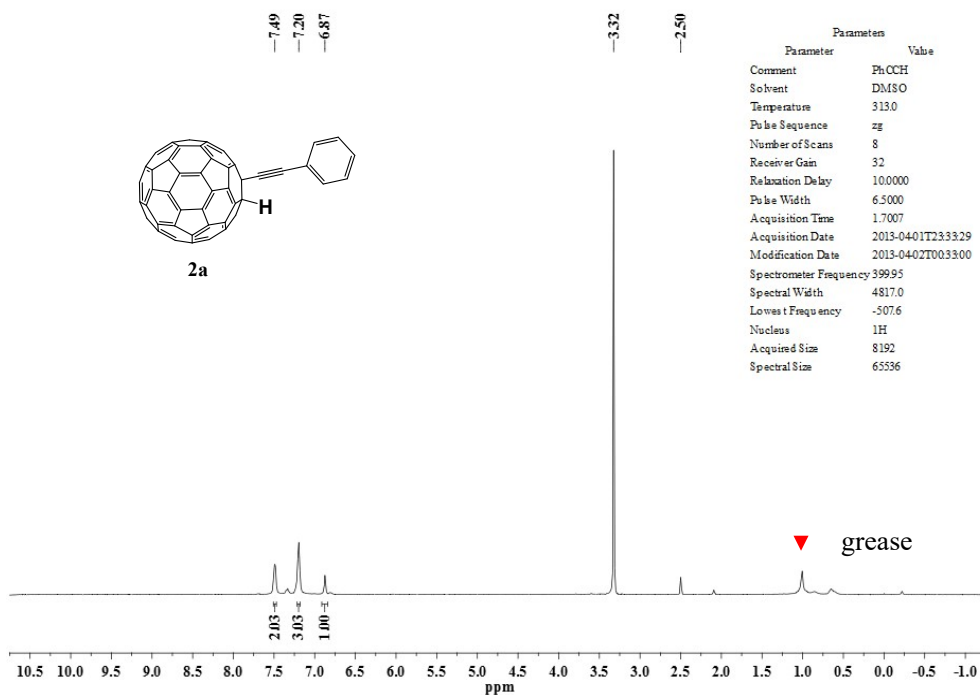
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Sample PhCCH  
Comment ESI Positive

Acquisition Date 5/14/2013 6:37:25 PM  
Instrument Bruker Apex IV FTMS  
Operator Peking University

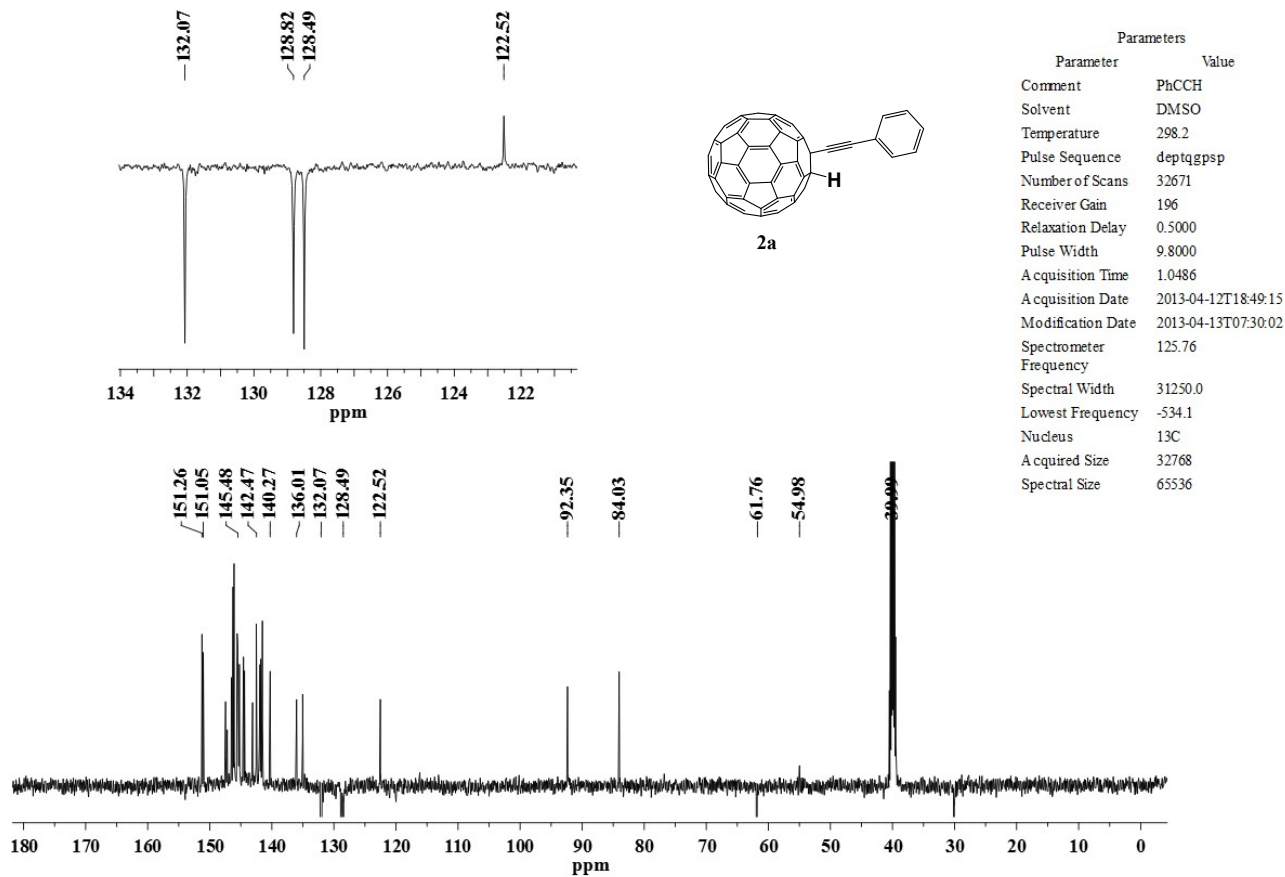


**Figure S7.** Positive ESI FT-ICR MS of **2a**.

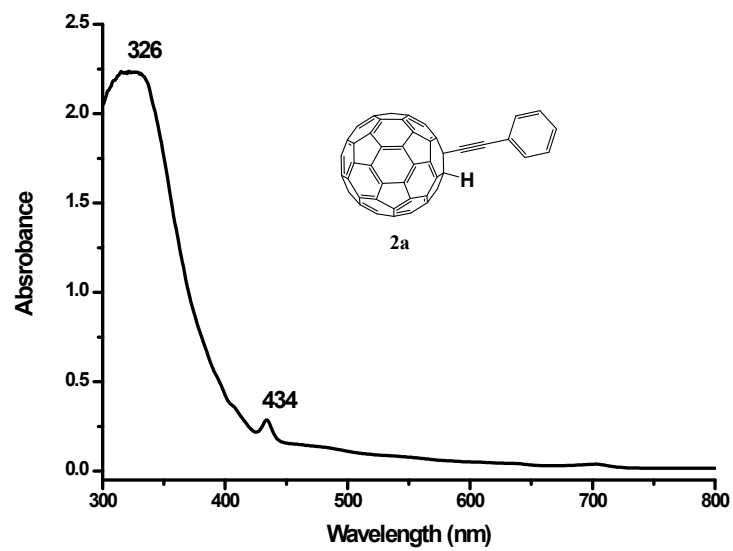


**Figure S8.** <sup>1</sup>H NMR spectrum of **2a** recorded in CS<sub>2</sub> with DMSO-*d*<sub>6</sub> as the external lock. The resonance around 2.5 ppm is due to the DMSO solvent, and the resonance around 3.3 ppm is due to H<sub>2</sub>O in DMSO.





**Figure S9.**  $^{13}\text{C}$  NMR spectrum of **2a** recorded in  $\text{CS}_2$  with  $\text{DMSO}-d_6$  as the external lock.

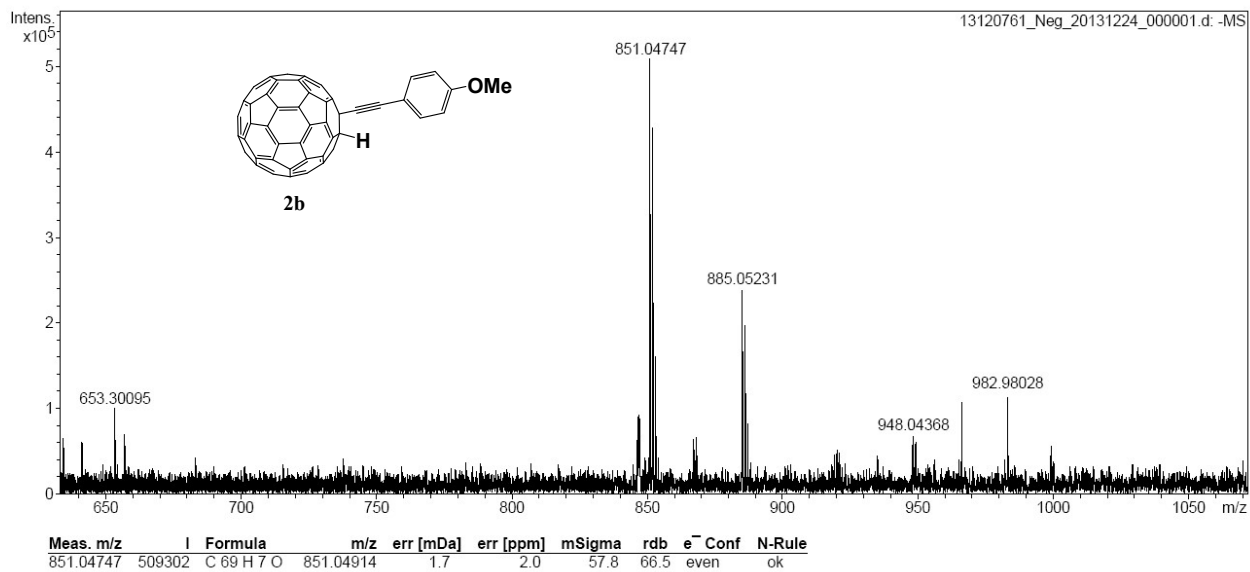


**Figure S10.** UV-visible spectrum of compound **2a** recorded in toluene.

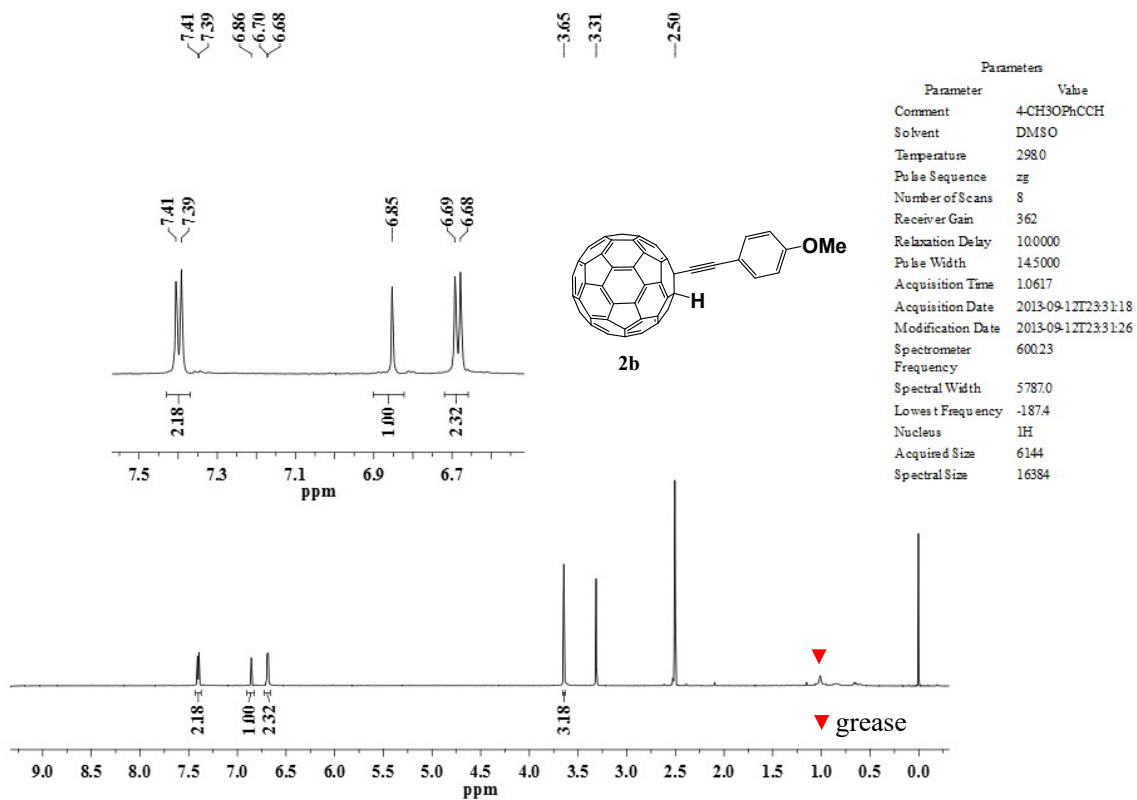
**Analysis Info**

Analysis Name 13120761\_Neg\_20131224\_000001.d  
Sample 4-CH3O  
Comment ESI Negative

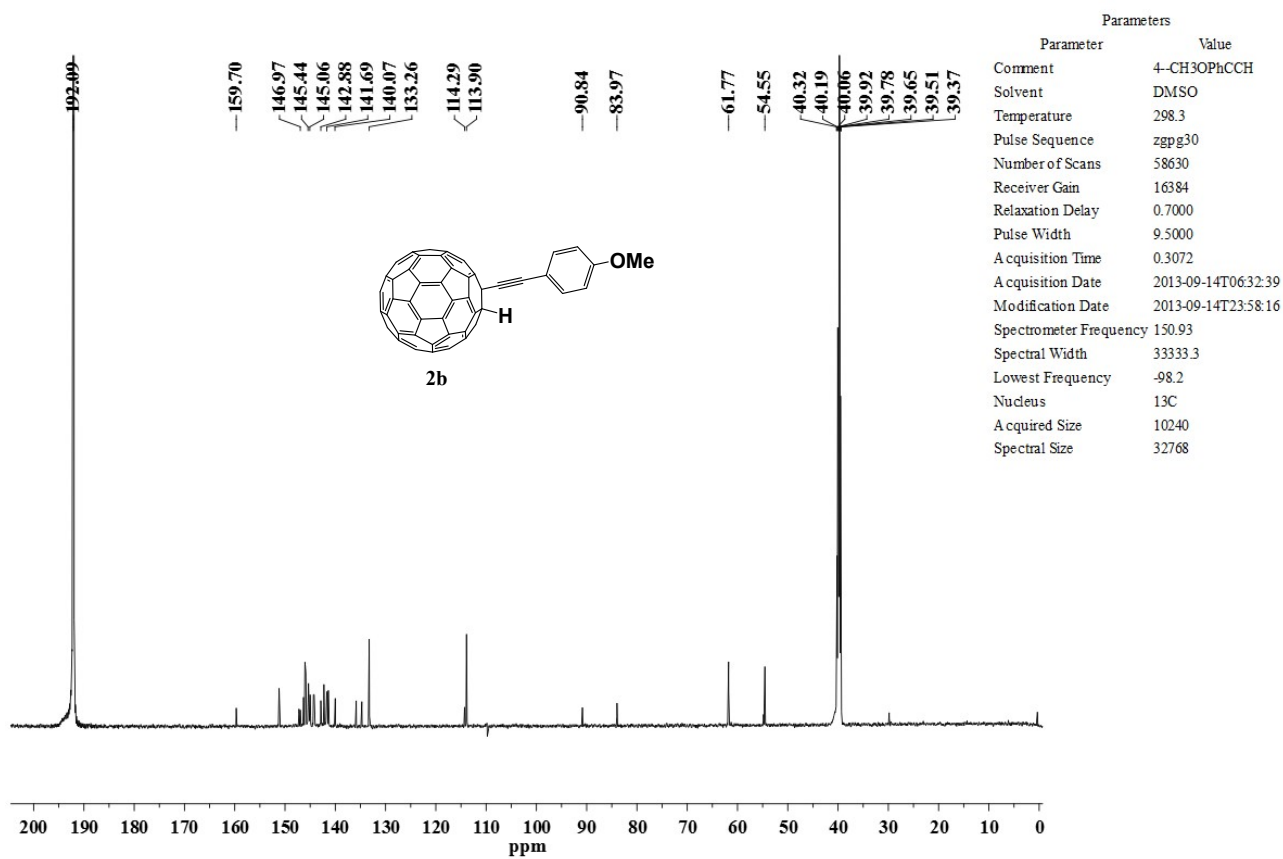
Acquisition Date 12/24/2013 4:57:23 PM  
Instrument Bruker Apex IV FTMS  
Operator Peking University



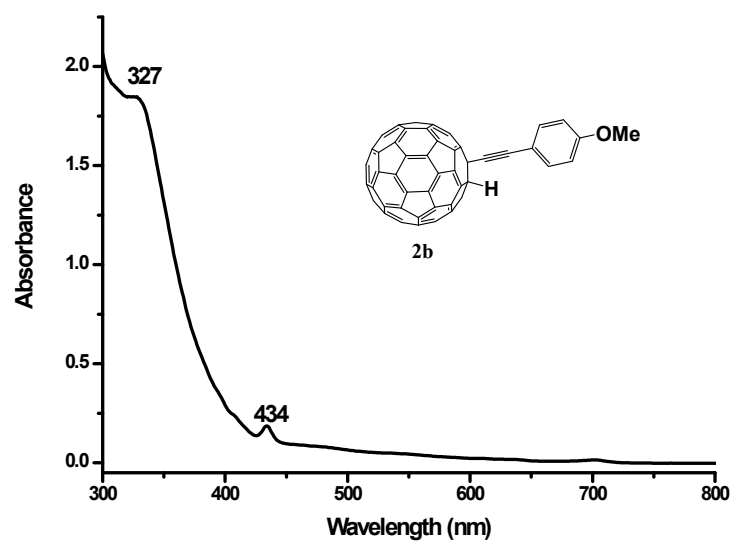
**Figure S11.** Negative ESI FT-ICR MS of **2b**.



**Figure S12.**  $^1\text{H}$  NMR spectrum of **2b** recorded in  $\text{CS}_2$  with  $\text{DMSO}-d_6$  as the external lock. The resonance around 2.5 ppm is due to the DMSO solvent, and the resonance around 3.3 ppm is due to  $\text{H}_2\text{O}$  residue in DMSO.



**Figure S13.**  $^{13}\text{C}$  NMR spectrum of **2b** recorded in  $\text{CS}_2$  with  $\text{DMSO}-d_6$  as the external lock.

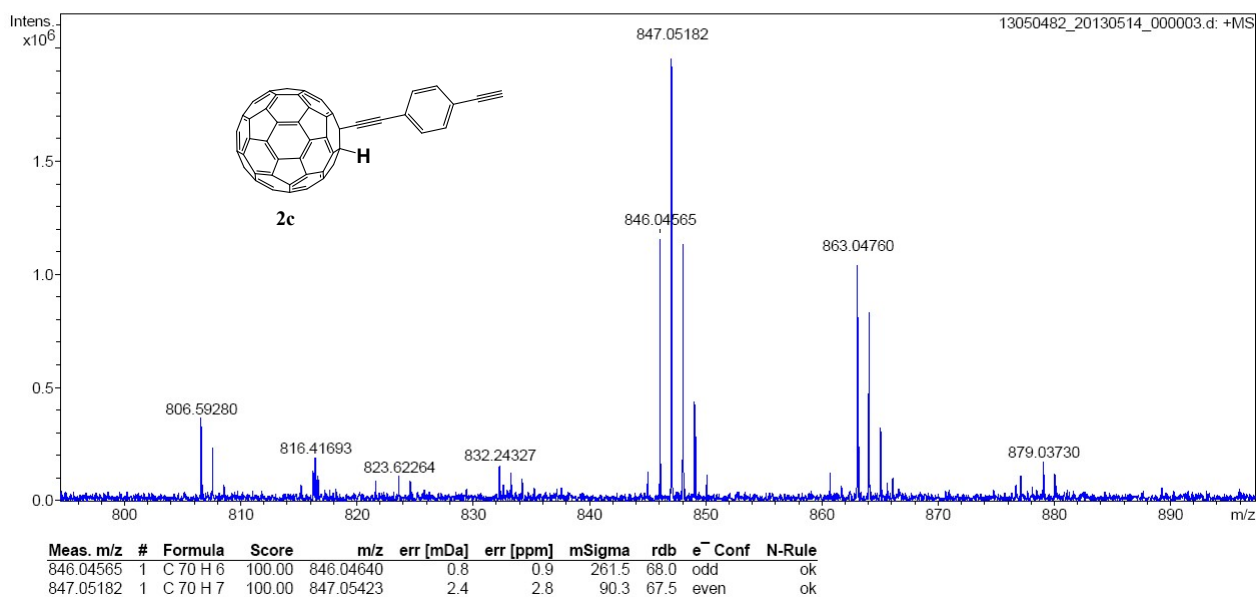


**Figure S14** UV-visible spectrum of compound **2b** recorded in toluene.

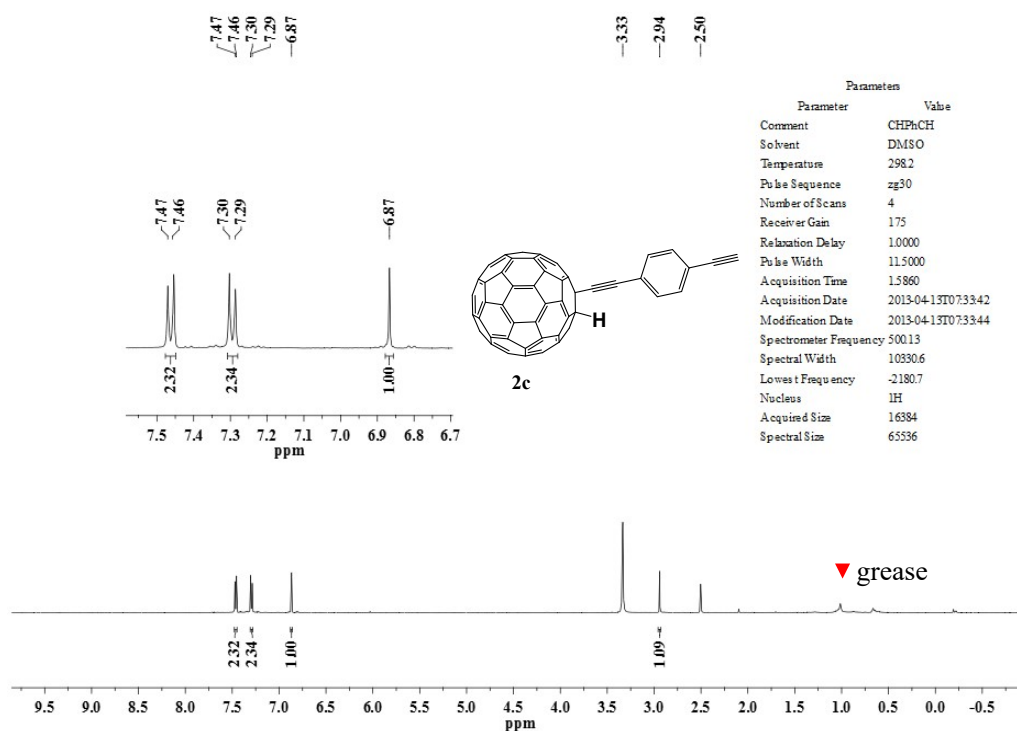
**Analysis Info**

Analysis Name 13050482\_20130514\_000003.d  
 Sample HCCPhCCH  
 Comment ESI Positive

Acquisition Date 5/14/2013 6:46:41 PM  
 Instrument Bruker Apex IV FTMS  
 Operator Peking University

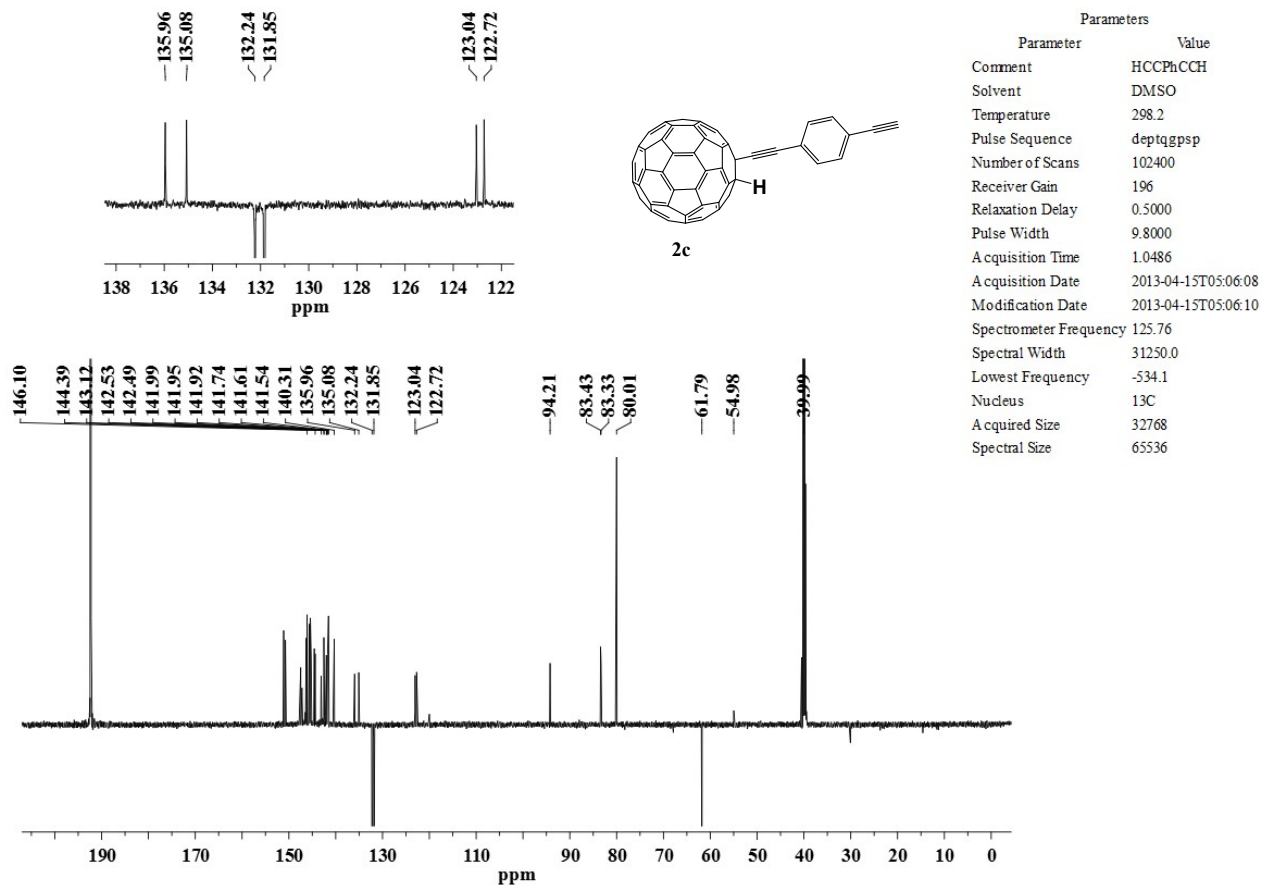


**Figure S15.** Positive ESI FT-ICR MS of **2c**.

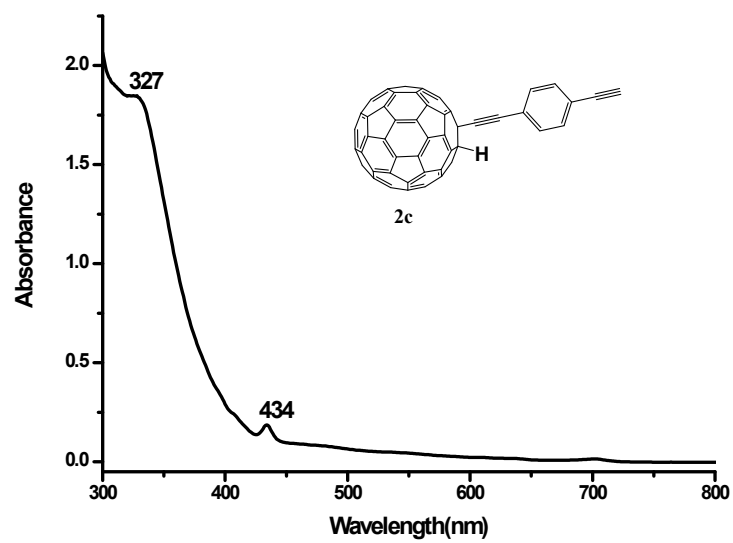


**Figure S16.** <sup>1</sup>H NMR spectrum of **2c** recorded in CS<sub>2</sub> with DMSO-*d*<sub>6</sub> as the external lock. The resonance around 2.5 ppm is due to the DMSO solvent, and the resonance around 3.3 ppm is due to H<sub>2</sub>O in DMSO.





**Figure S17.** <sup>13</sup>C NMR spectrum of **2c** recorded in CS<sub>2</sub> with DMSO-*d*<sub>6</sub> as the external lock.

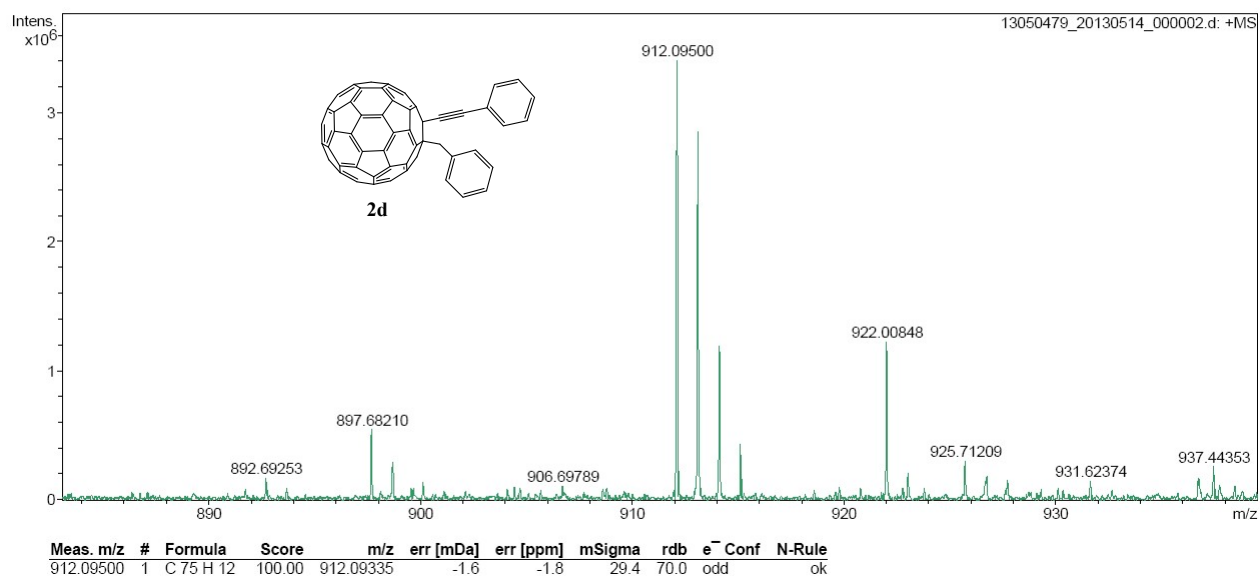


**Figure S18.** UV-visible spectrum of compound **2c** recorded in toluene.

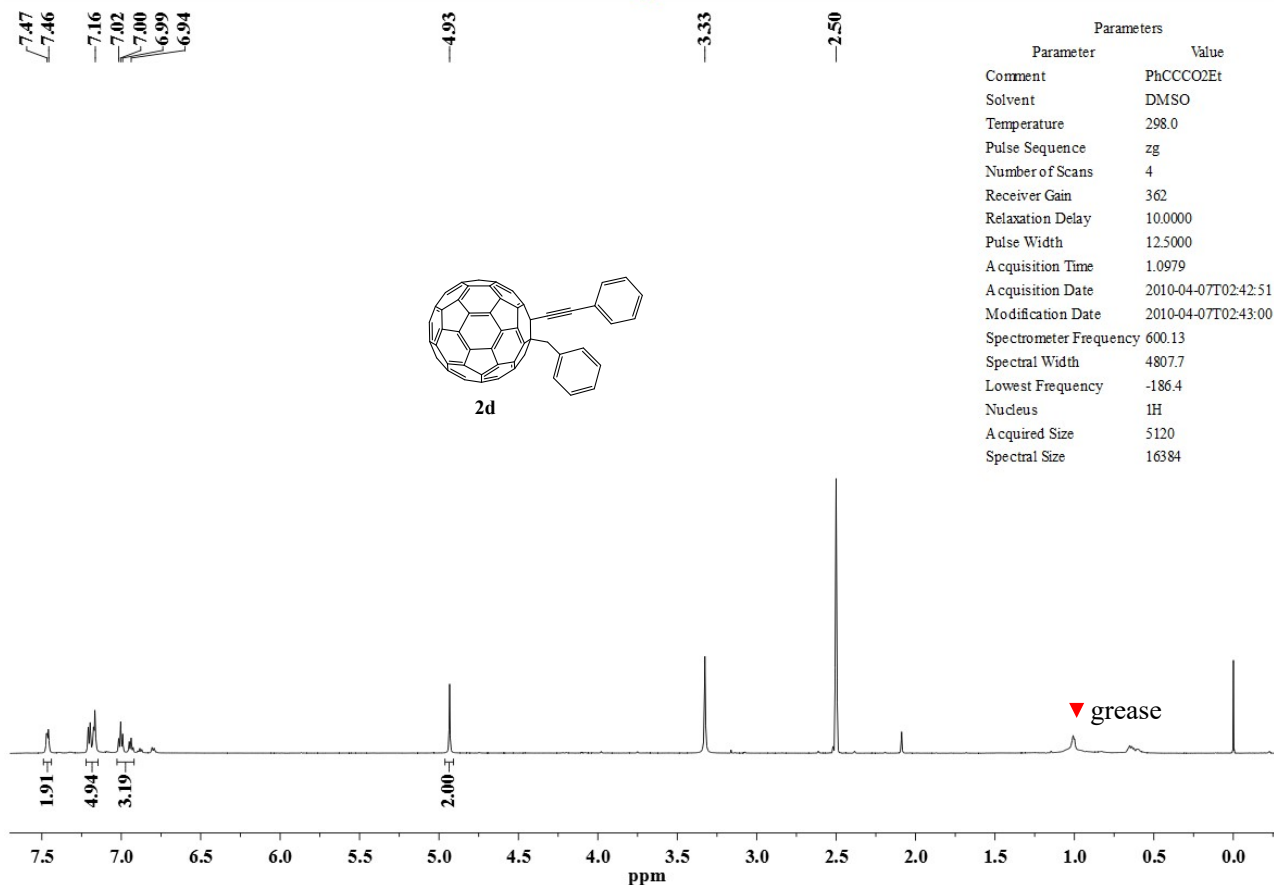
**Analysis Info**

Analysis Name 13050479\_20130514\_000002.d  
Sample di-adduct  
Comment ESI Positive

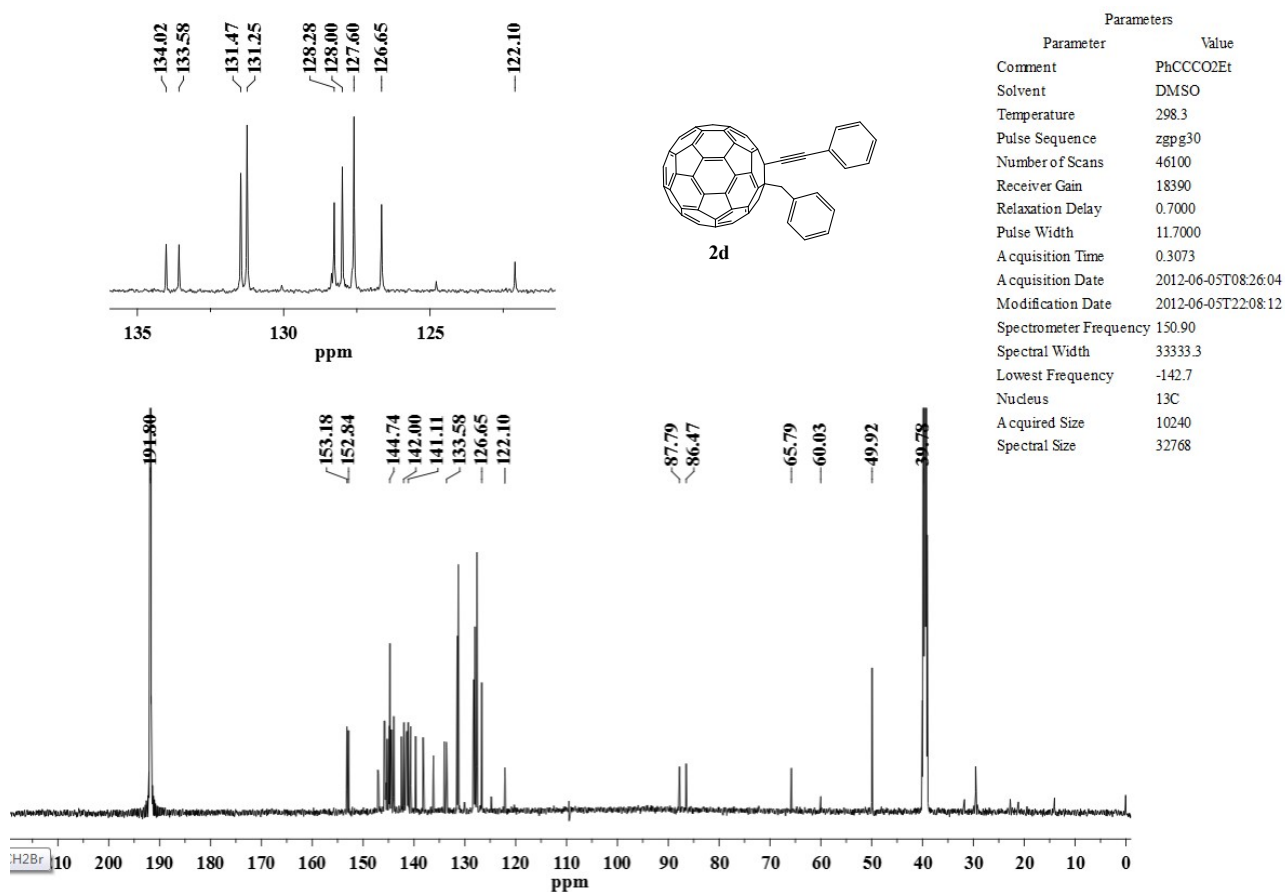
Acquisition Date 5/14/2013 6:34:14 PM  
Instrument Bruker Apex IV FTMS  
Operator Peking University



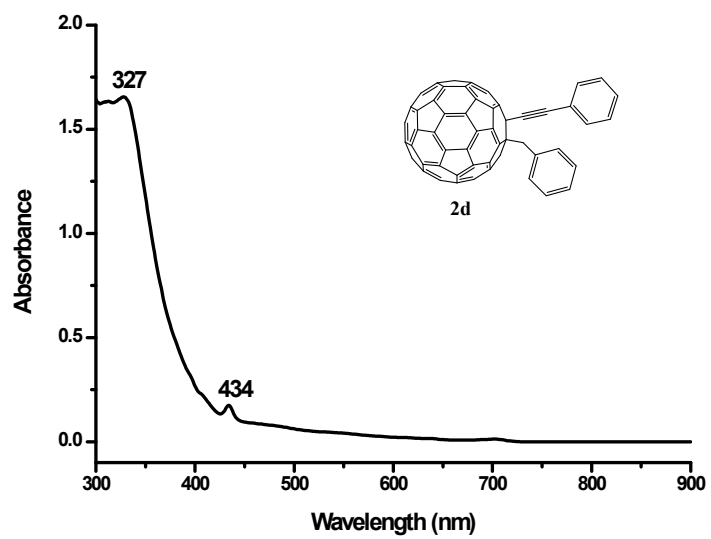
**Figure S19.** Positive ESI FT-ICR MS of **2d**.



**Figure S20.**  $^1\text{H}$  NMR spectrum of **2d** recorded in  $\text{CS}_2$  with  $\text{DMSO-}d_6$  as the external lock. The resonance around 2.5 ppm is due to the DMSO solvent, and the resonance around 3.33 ppm is due to  $\text{H}_2\text{O}$  in DMSO.



**Figure S21.** <sup>13</sup>C NMR spectrum of **2d** recorded in CS<sub>2</sub> with DMSO-*d*<sub>6</sub> as the external lock.



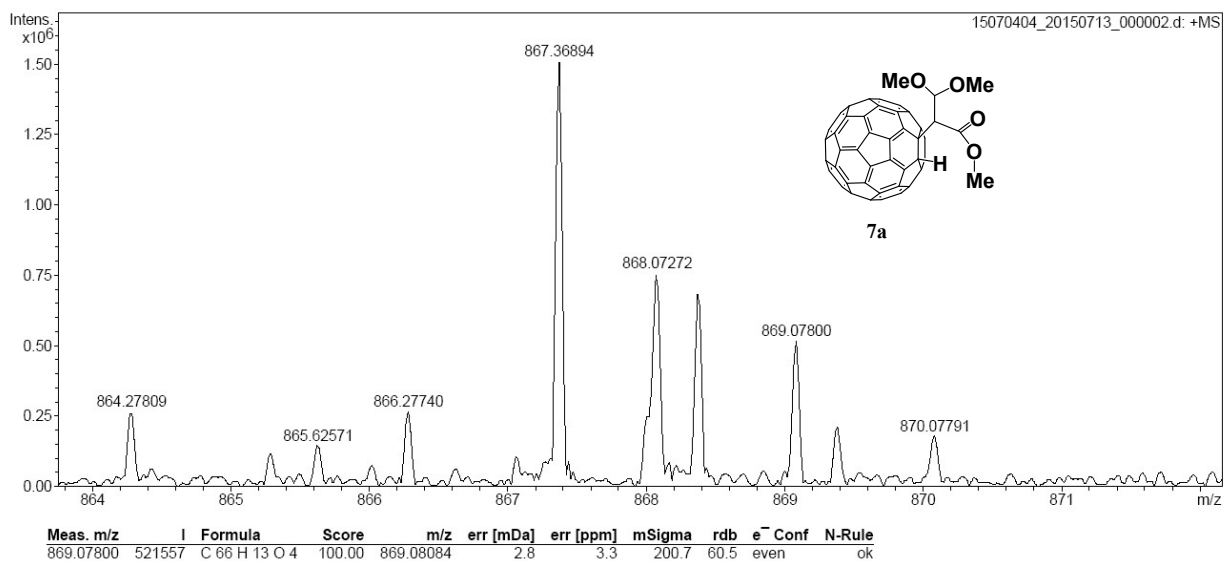
**Figure S22.** UV-visible spectrum of compound **2d** recorded in toluene.

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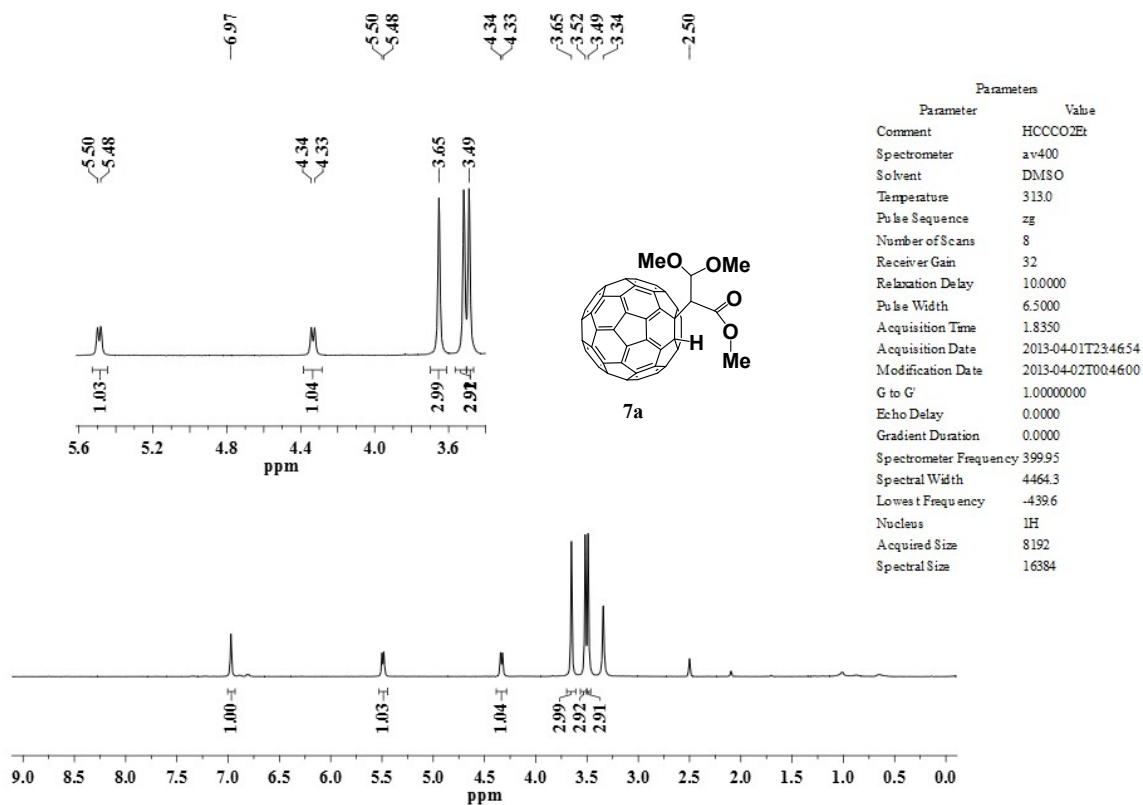
### Analysis Info

Analysis Name 15070404\_20150713\_000002.d  
 Sample Etpro-5.7  
 Comment ESI Positive

Acquisition Date 7/13/2015 1:11:56 PM  
 Instrument Bruker Apex IV FTMS  
 Operator Peking University



**Figure S23.** Positive ESI FT-ICR MS of **7a**.



**Figure S24.** <sup>1</sup>H NMR spectrum of **7a** recorded in CS<sub>2</sub> with DMSO-*d*<sub>6</sub> as the external lock. The resonance around 2.5 ppm is due to the DMSO solvent, and the resonance around 3.3 ppm is due to H<sub>2</sub>O in DMSO.



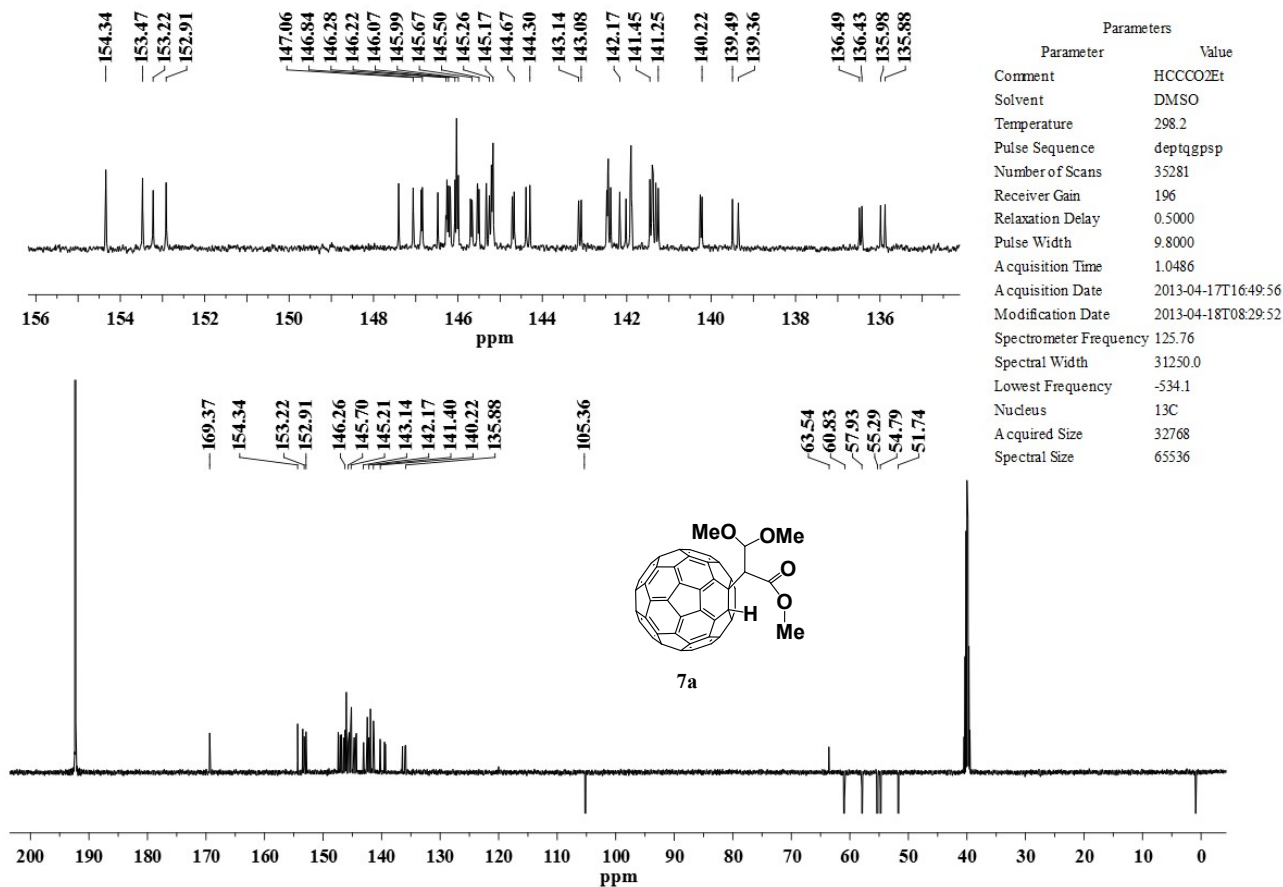
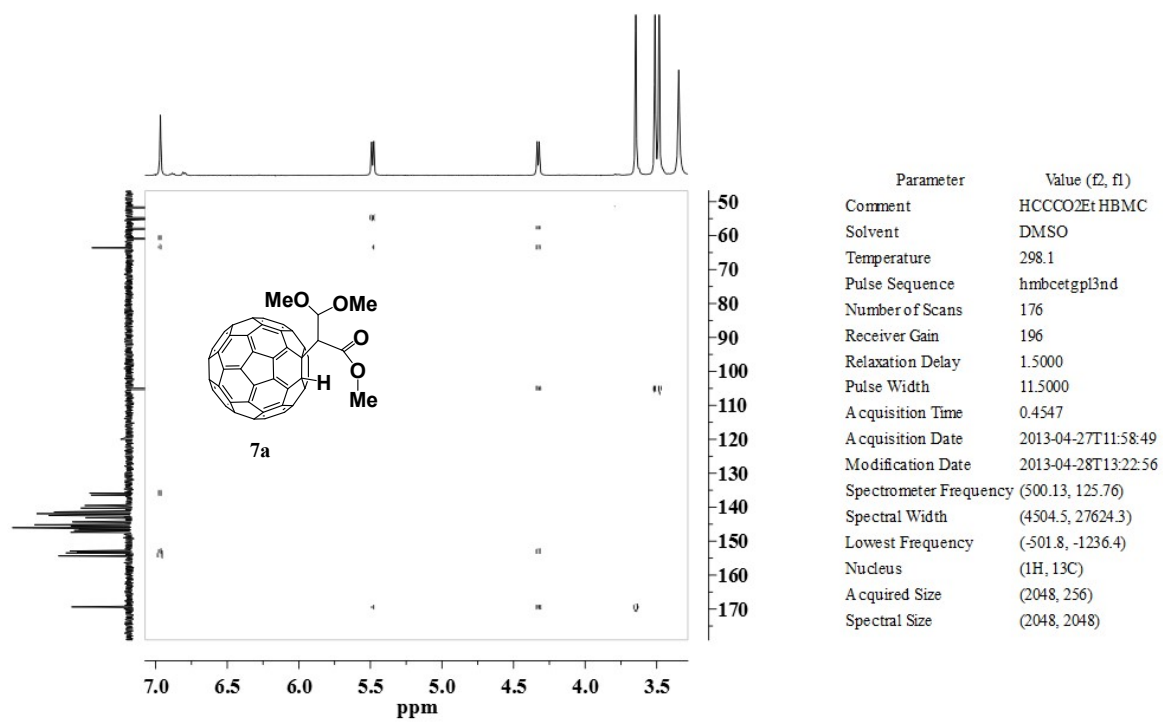
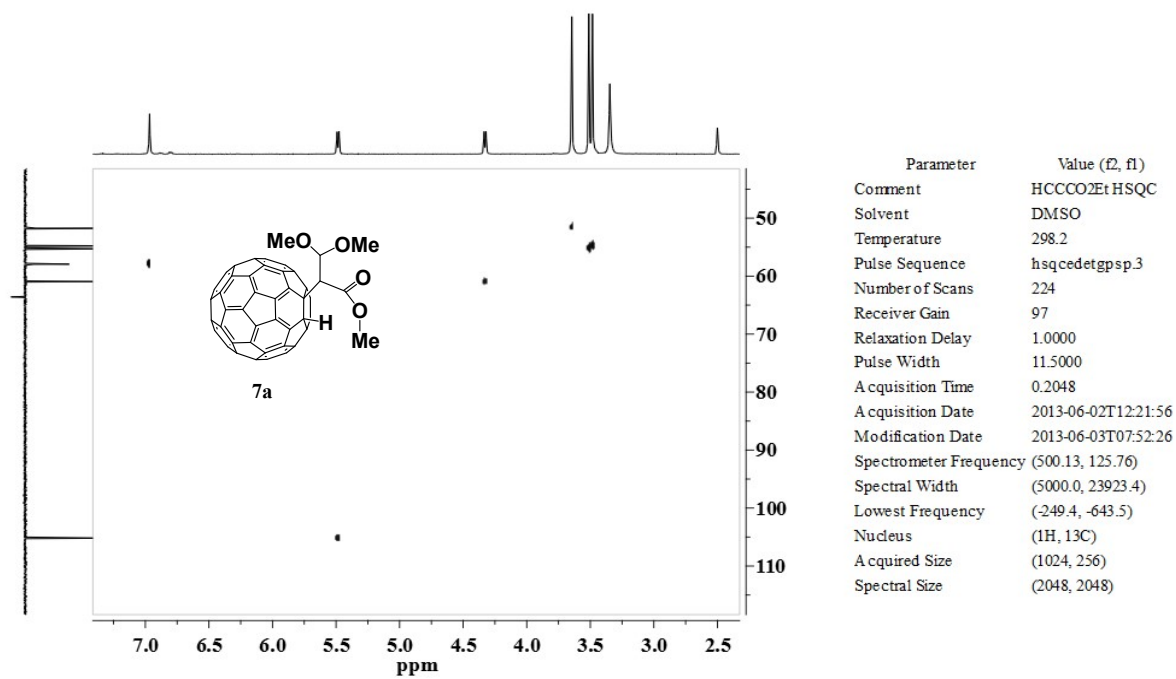


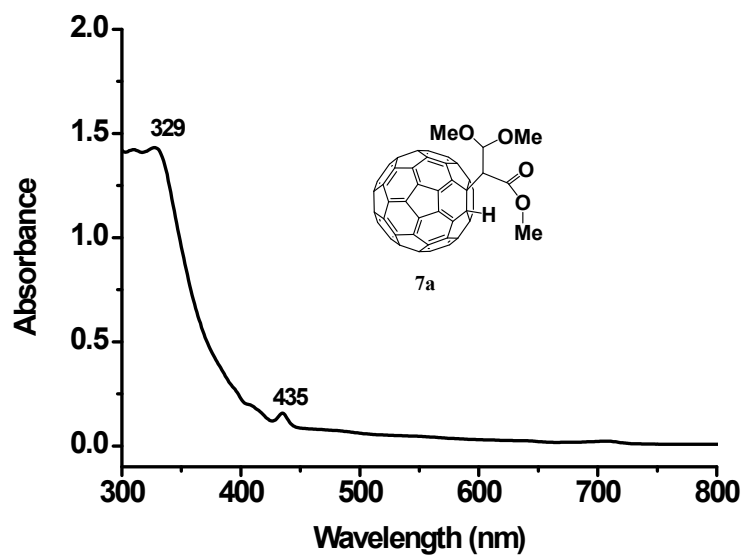
Figure S25. <sup>13</sup>C NMR spectrum of **7a** recorded in CS<sub>2</sub> with DMSO-*d*<sub>6</sub> as the external lock.



**Figure S26.** HMBC NMR spectrum of **7a** recorded in  $\text{CS}_2$  with  $\text{DMSO}-d_6$  as the external lock.



**Figure S27.** HSQC NMR spectrum of **7a** recorded in CS<sub>2</sub> with DMSO-*d*<sub>6</sub> as the external lock.



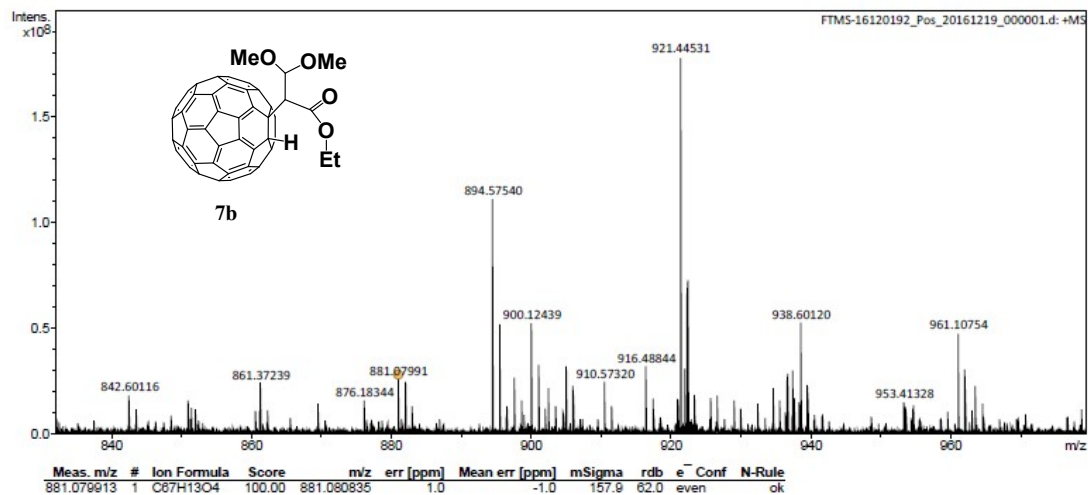
**Figure S28.** UV-visible spectrum of compound **7a** recorded in toluene.

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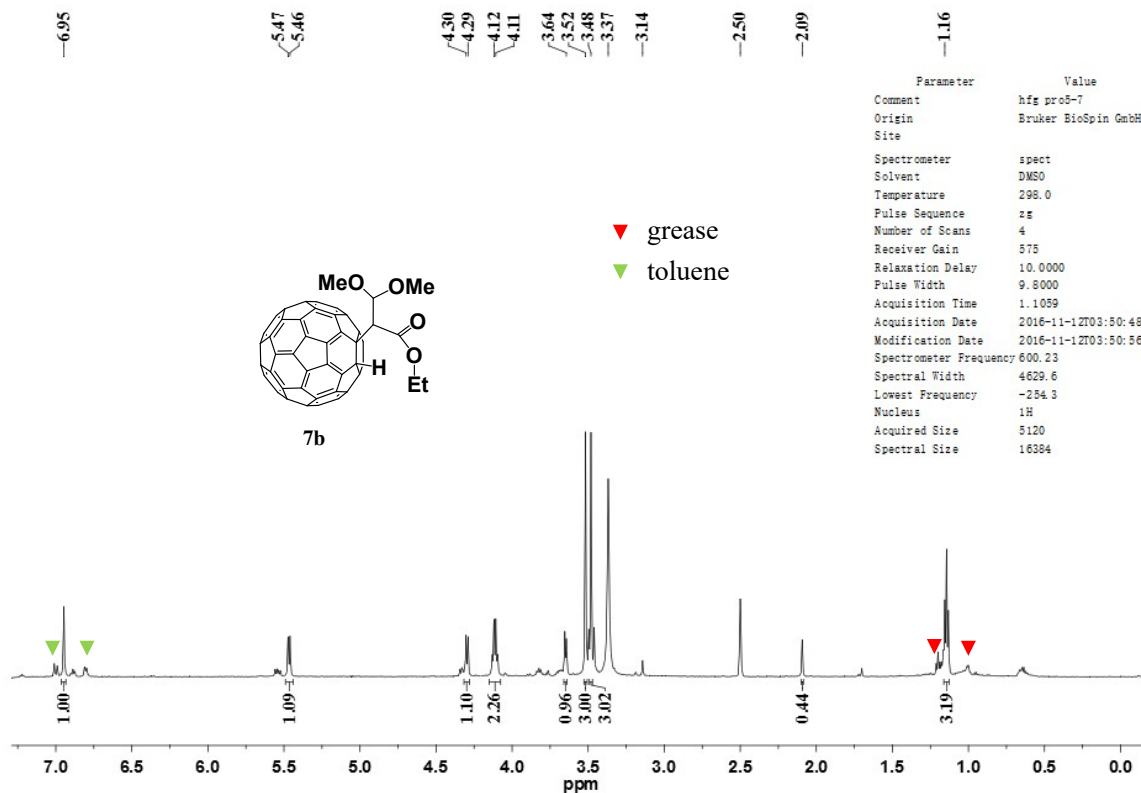
### Analysis Info

Analysis Name FTMS-16120192\_Pos\_20161219\_000001.d  
 Sample C60C3TH  
 Comment

Acquisition Date 12/19/2016 2:56:33 PM  
 Instrument Bruker Solarix XR FTMS  
 Operator Peking University



**Figure S29.** Positive ESI FT-ICR MS of **7b**.



**Figure S30.**  $^1\text{H}$  NMR spectrum of **7b** recorded in  $\text{CS}_2$  with  $\text{DMSO-}d_6$  as the external lock. The resonance around 2.5 ppm is due to the DMSO solvent, and the resonance around 3.3 ppm is due to  $\text{H}_2\text{O}$  in DMSO.

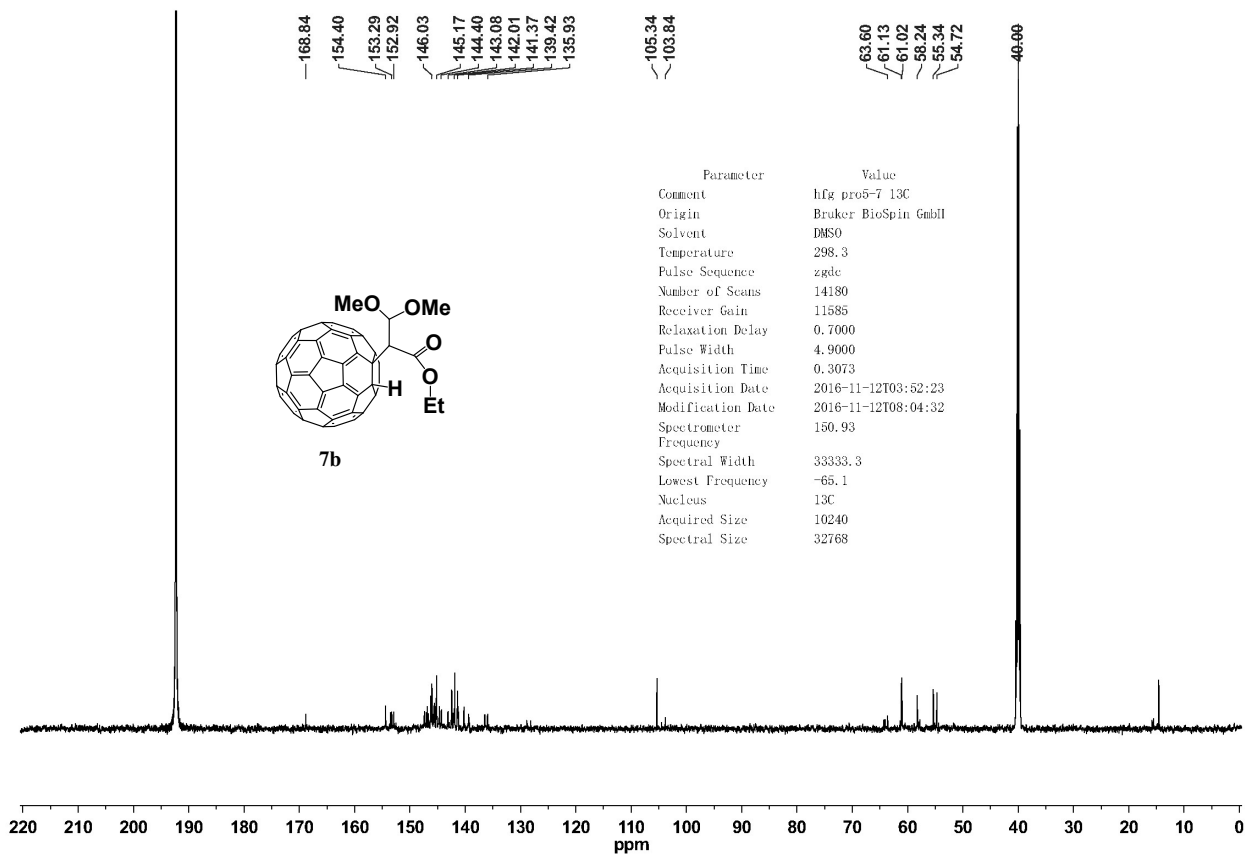
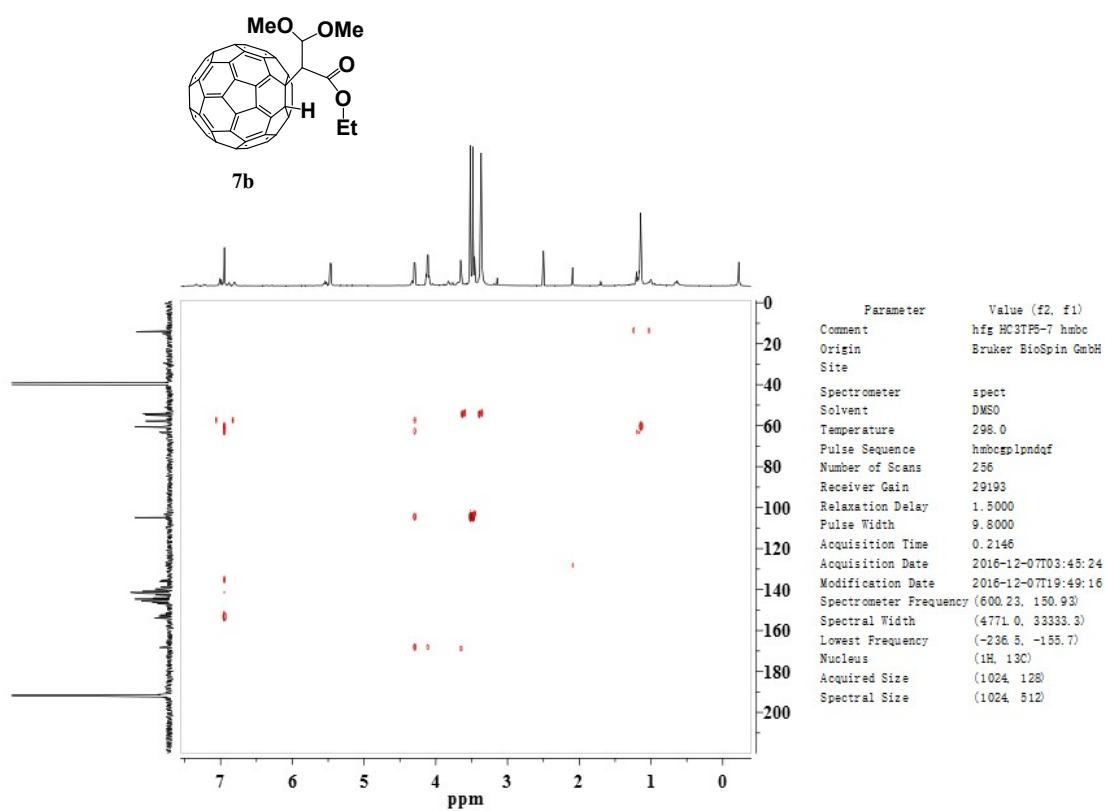
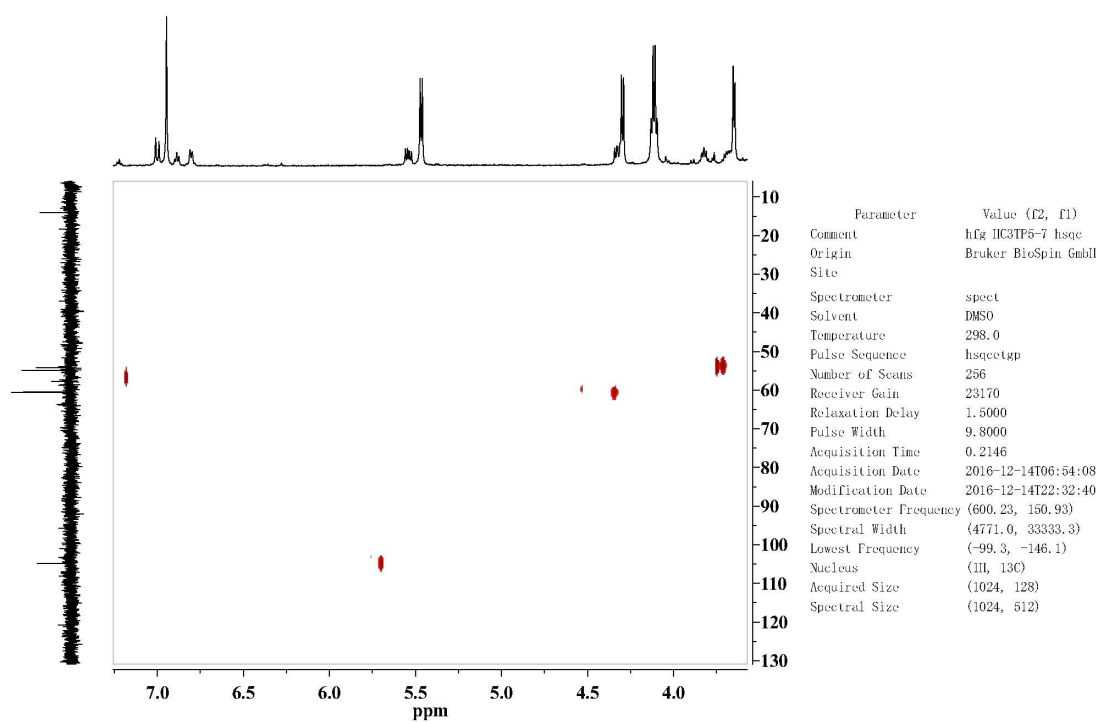


Figure S31.  $^{13}\text{C}$  NMR spectrum of **7b** recorded in  $\text{CS}_2$  with  $\text{DMSO}-d_6$  as the external lock

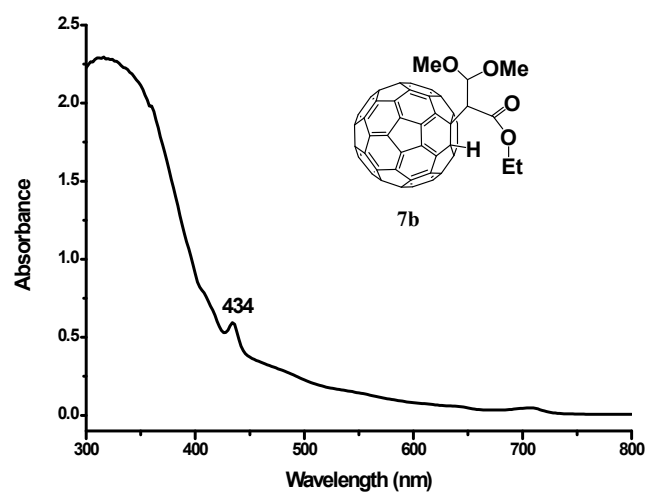


**Figure S32.** HMBC NMR spectrum of **7b** recorded in CS<sub>2</sub> with DMSO-*d*<sub>6</sub> as the external lock.





**Figure S33.** HSQC NMR spectrum of **7b** recorded in CS<sub>2</sub> with DMSO-*d*<sub>6</sub> as the external lock.

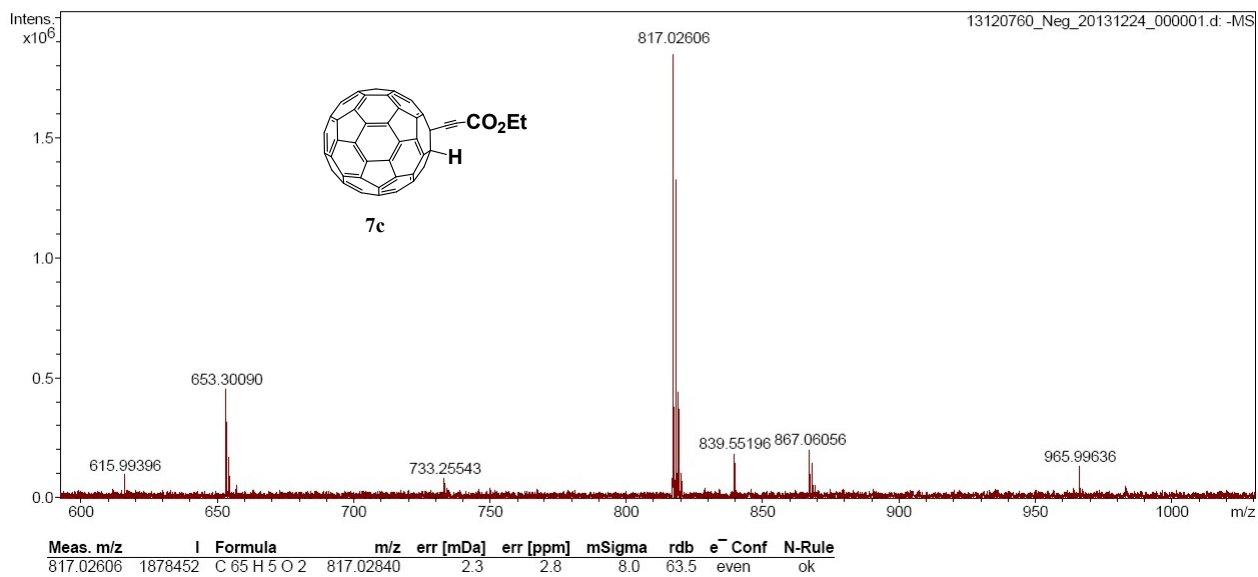


**Figure S34.** UV-visible spectrum of compound **7b** recorded in toluene.

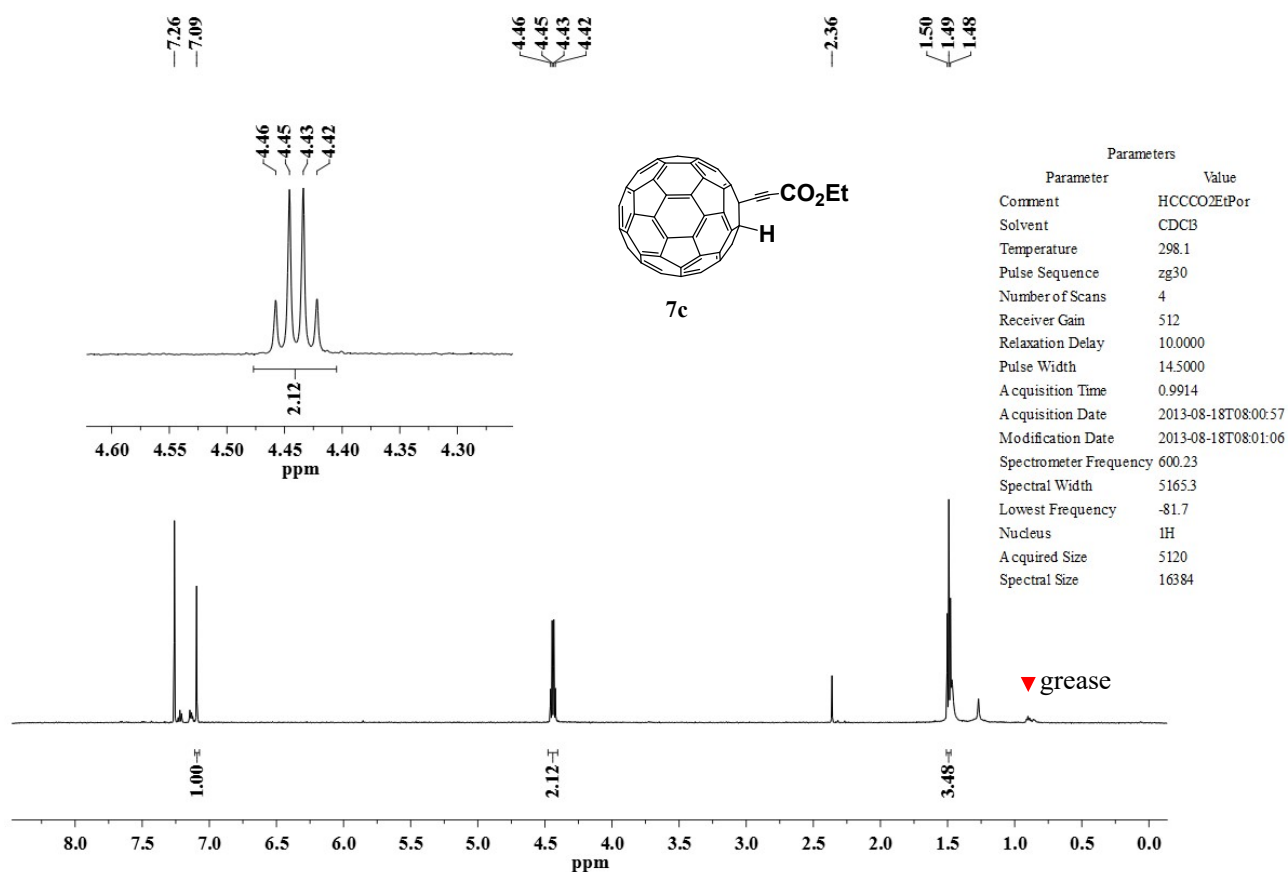
**Analysis Info**

Analysis Name 13120760\_Neg\_20131224\_000001.d  
Sample 3-c3por  
Comment ESI Negative

Acquisition Date 12/24/2013 4:55:17 PM  
Instrument Bruker Apex IV FTMS  
Operator Peking University



**Figure S35.** Negative ESI FT-ICR MS of **7c**.



**Figure S36.** <sup>1</sup>H NMR spectrum of **7c** recorded in CS<sub>2</sub> with CDCl<sub>3</sub>- as the external lock. The resonance around 7.26 ppm is due to the CDCl<sub>3</sub> solvent, and the resonance around 2.36 ppm is due to toluene in solvent.

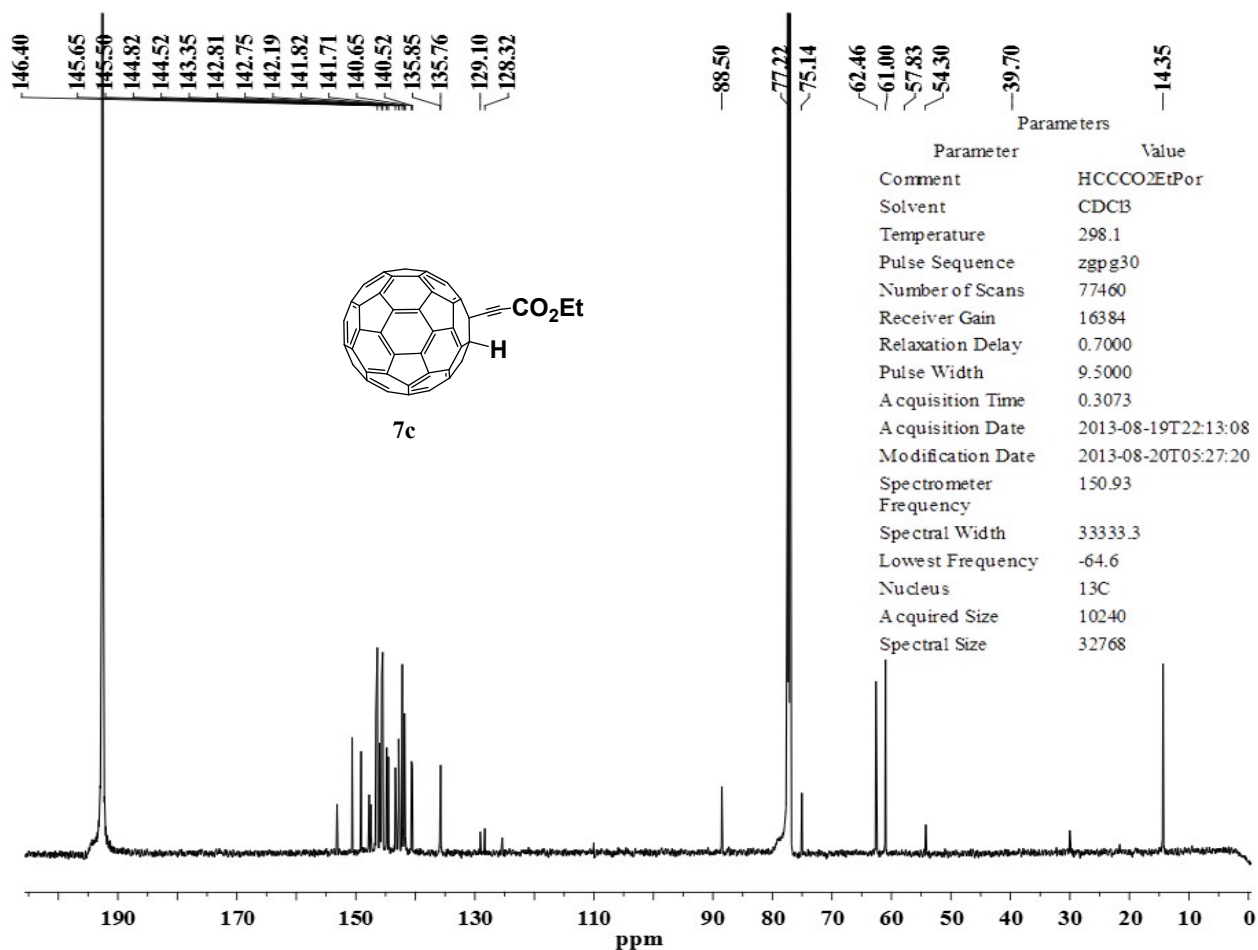
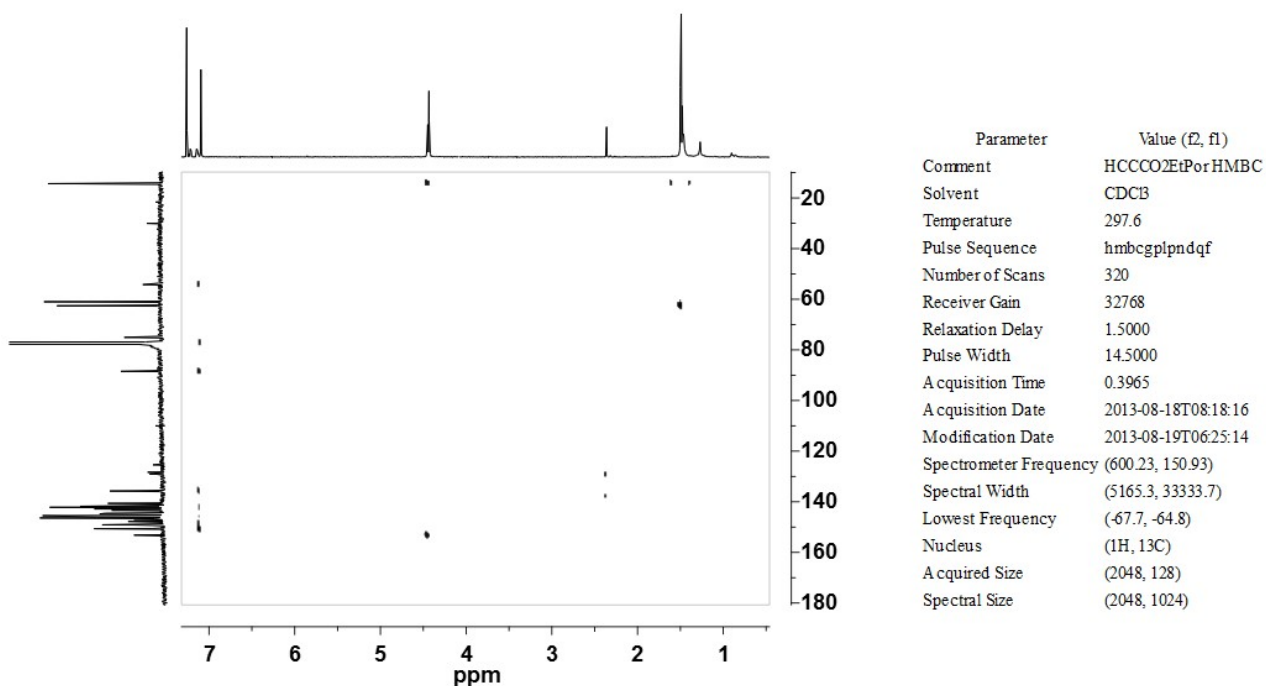
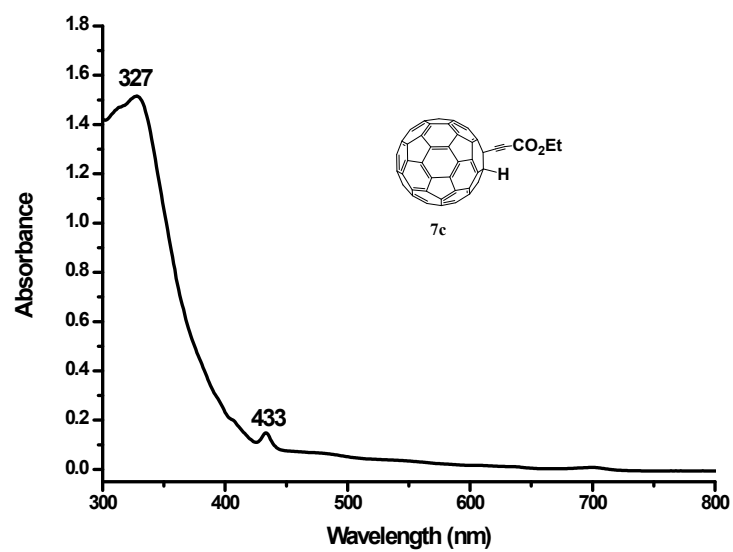


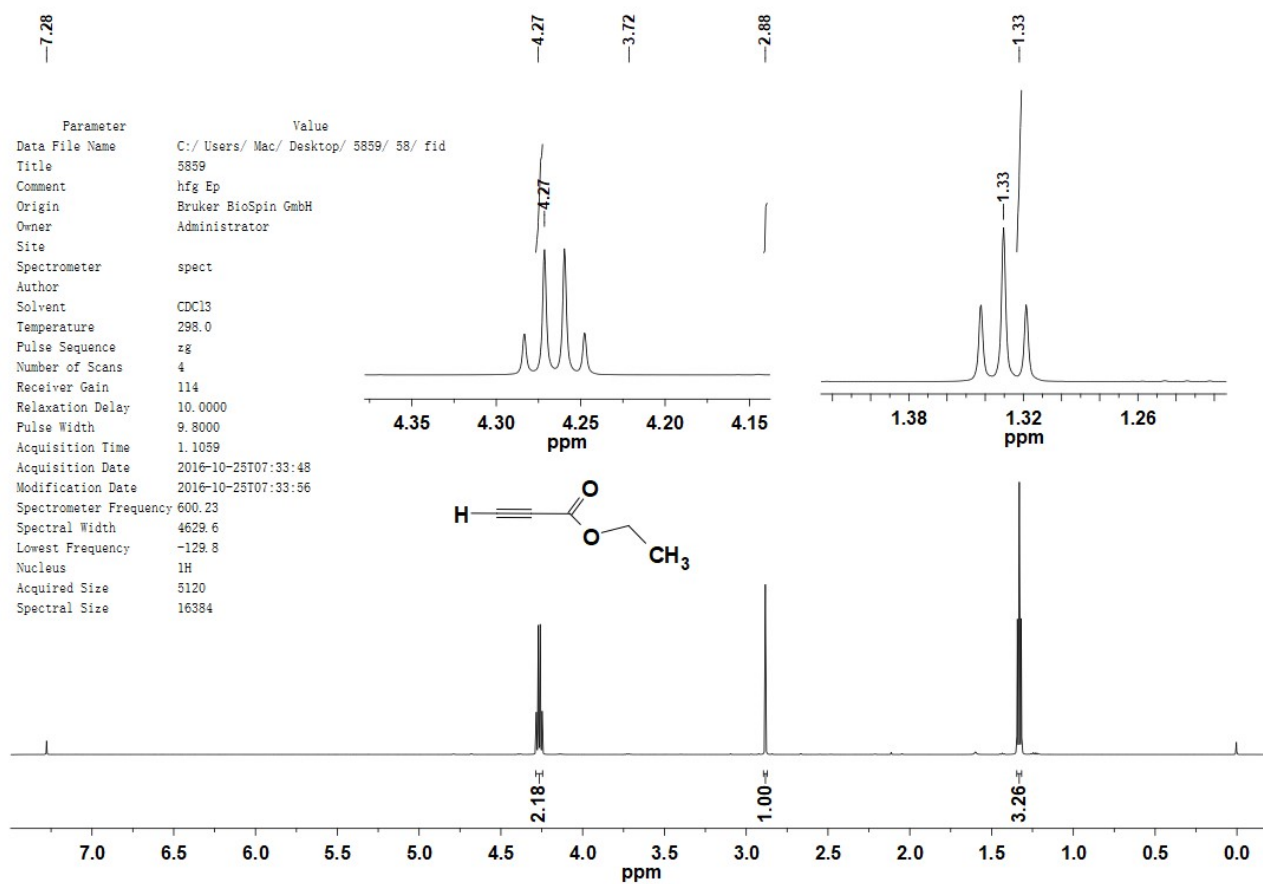
Figure S37.  $^{13}\text{C}$  NMR spectrum of **7c** recorded in  $\text{CS}_2$  with  $\text{CDCl}_3$  as the external lock.



**Figure S38.** HMBC NMR spectrum of **7c** recorded in CS<sub>2</sub> with CDCl<sub>3</sub>- as the external lock.



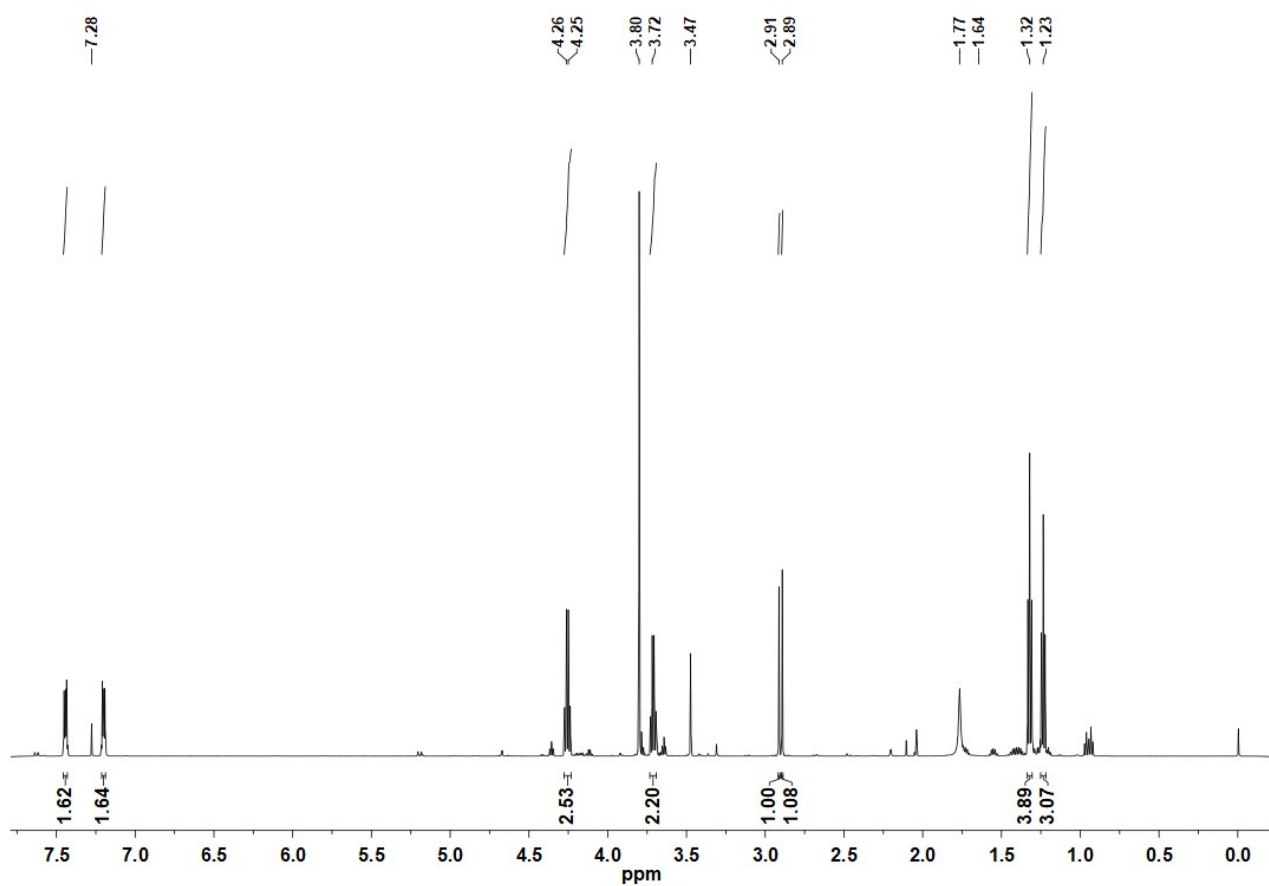
**Figure S39.** UV-visible spectrum of compound **7c** recorded in toluene.



**Figure S40.** <sup>1</sup>H NMR spectrum of **7** recorded in CDCl<sub>3</sub>. The resonance around 7.26 ppm is due to the CDCl<sub>3</sub> solvent.







**Figure S42.**  $^1\text{H}$  NMR spectrum of the crude mixture formed from the reaction of **7** TBAOH/ $\text{CH}_3\text{OH}$  at  $100\text{ }^\circ\text{C}$ , recorded in  $\text{CDCl}_3$ .