



Supporting Information

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## Supporting information

### Gold(I)-Catalyzed Cycloisomerization of 3-Methoxy-1,6-enynes Featuring Tandem Cyclization and [3,3]-Sigmatropic Rearrangement\*\*

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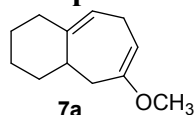
#### Representative Experimental Procedure for the cycloisomerization and the conversion to the cyclohept-4-en-1-ones, and copies of $^1\text{H}$ and $^{13}\text{C}$ spectra for all new compounds.

##### I. General information

All solvents were dried and distilled according to the standard methods before use.  $\text{Au}(\text{PPh}_3)\text{Cl}$  and  $\text{AgSbF}_6$  were purchased from Aldrich Chemicals and stored in a dry-keeper.  $\text{Au}(\text{P}(\text{C}_6\text{F}_5)_3)\text{Cl}$  was prepared according to the literature procedures.<sup>[1]</sup> Experiments were performed in flame-dried glasswares with rubber septa under a positive pressure of nitrogen. Reactions were monitored by thin-layer chromatography carried out on 0.25 mm E. Merck silica gel plates (60F-254) using UV light as a visualizing agent and acidic *p*-anisaldehyde, and heat as developing agent. Flash chromatography was carried out on Merck 60 silica gel (230-400 mesh).  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded with Bruker (300 MHz) spectrometer.  $^1\text{H}$  NMR spectra were referenced to residual TMS (0 ppm) and reported as follows; chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet). Chemical shifts of the  $^{13}\text{C}$  NMR spectra were measured relative to  $\text{CDCl}_3$  (77.23 ppm). Mass spectral data were obtained from the Korea Basic Science Institute (Daegu) on a Jeol JMS 700 high resolution mass spectrometer. Infrared spectra were recorded on a Shimadzu IR-470 spectrometer.

##### II. General procedure for the cycloisomerization

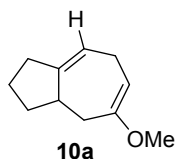
###### Compound 7a



To a stirred solution of gold complex  $\text{Au}(\text{P}(\text{C}_6\text{F}_5)_3)\text{Cl}$  (5.1 mg, 0.0067 mmol) and  $\text{AgSbF}_6$  (2.3 mg, 0.0067 mmol) was added methylene chloride (10 mL) and the solution was stirred for 10 min. The resulting solution was filtered through a pad of Celite and concentrated. The residue was dried over high vacuum for 2 hours, and then cooled to  $-15^\circ\text{C}$ . To this residue was added a solution of **6** (120 mg, 0.67 mmol) in  $\text{CH}_2\text{Cl}_2$  (13.4 mL, 0.05M, pre-cooled to  $-15^\circ\text{C}$ ). The resulting colorless solution was stirred for 2 min. Triethylamine (1 mL) was added and the solution was stirred for 5 min. The resulting solution was passed through a pad of Celite and concentrated. The residual oil was purified by flash chromatography on silica gel (deactivated by triethylamine before use, eluted with pentane: ether = 95:5) to give the compound **7a** as a colorless oil (117 mg, 0.65 mmol, 97% yield).  $R_f$  = 0.45 (pentane: ether = 95:5);

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 1.14-1.47 (m, 3H), 1.66-1.77 (m, 3H), 1.87-1.95 (m, 1H), 2.04-2.14 (m, 2H), 2.38-2.40 (m, 2H), 2.45-2.57 (m, 1H), 2.64-2.74 (m, 1H), 3.47 (s, 3H), 4.79 (app t,  $J$  = 6.4 Hz, 1H), 5.45 (m, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 23.6, 27.0, 28.9, 35.7, 37.5, 38.8, 39.2, 54.6, 95.7, 120.3, 143.8, 159.4; IR: ( $\text{cm}^{-1}$ )  $\nu$  3070, 2925, 2852, 1666, 1155; HRMS calcd for  $\text{C}_{12}\text{H}_{18}\text{O}$ : 178.1358. found: 178.1358.

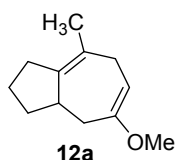
## Compound 10a



Using the representative procedure, a mixture of **9** (105 mg, 0.64 mmol), gold complex  $\text{Au}(\text{P}(\text{C}_6\text{F}_5)_3)\text{Cl}$  (4.9 mg, 0.0064 mmol) and  $\text{AgSbF}_6$  (2.2 mg, 0.0064 mmol) were reacted to give **10a** as a colorless oil. (101 mg, 0.61 mmol, 96 % yield).  $R_f = 0.63$  (hexane : ethyl acetate = 95:5);

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 1.25\text{--}1.35$  (m, 1H), 1.51–1.71 (m, 2H), 1.91–1.96 (m, 1H), 2.18 – 2.35 (m, 4H), 2.65 – 2.82 (m, 3H), 3.46 (s, 3H), 4.60 (app t,  $J = 5.4$  Hz, 1H), 5.51 – 5.54 (m, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 24.9, 25.2, 34.3, 35.0, 37.6, 39.8, 54.3, 94.1, 118.4, 148.5, 157.8$ ; IR: ( $\text{cm}^{-1}$ )  $\nu$  3071, 2945, 2856, 2829, 1668, 1144; HRMS calcd for  $\text{C}_{11}\text{H}_{16}\text{O}$ : 164.1201. found: 164.1202.

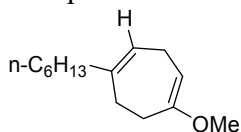
## Compound 12a



Using the representative procedure, a mixture of **11** (30 mg, 0.17 mmol), gold complex  $\text{Au}(\text{P}(\text{C}_6\text{F}_5)_3)\text{Cl}$  (6.4 mg, 0.0084 mmol) and  $\text{AgSbF}_6$  (2.9 mg, 0.0084 mmol) were reacted to give **12a** as a colorless oil. (29 mg, 0.16 mmol, 95 % yield).  $R_f = 0.45$  (pentane: ether = 95:5);

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 1.25\text{--}1.38$  (m, 1H), 1.52–1.76 (m, 2H), 1.68 (s, 3H), 1.85–1.96 (m, 1H), 2.15 – 2.22 (m, 2H), 2.25 – 2.32 (m, 2H), 2.45 (dd,  $J = 18.0, 7.2$  Hz, 1H), 2.67–2.83 (m, 1H), 2.89 – 3.04 (m, 1H), 3.45 (s, 3H), 4.63 (dd,  $J = 6.8, 4.3$  Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 22.2, 25.3, 31.3, 31.8, 35.0, 37.4, 39.8, 54.1, 93.9, 126.8, 141.0, 157.5$ ; IR: ( $\text{cm}^{-1}$ )  $\nu$  2938, 2856, 2830, 1667, 1158; HRMS calcd for  $\text{C}_{12}\text{H}_{18}\text{O}$ : 178.1358. found: 178.1358.

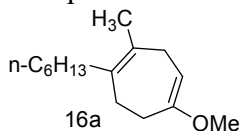
## Compound 14a



Using the representative procedure, a mixture of **13** (73 mg, 0.35 mmol), gold complex  $\text{Au}(\text{P}(\text{C}_6\text{F}_5)_3)\text{Cl}$  (2.7 mg, 0.0035 mmol) and  $\text{AgSbF}_6$  (1.2 mg, 0.0035 mmol) were reacted to give **14a** as a colorless oil. (66 mg, 0.32 mmol, 90 % yield).  $R_f = 0.45$  (pentane: ether = 95:5);

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 0.88$  (t,  $J = 6.7$  Hz, 3 H), 1.26–1.43 (m, 8H), 1.97 (app t,  $J = 7.3$  Hz, 2H), 2.21 – 2.27 (m, 2H), 2.28 – 2.31 (m, 2H), 2.69 (app t,  $J = 5.7$  Hz, 1H), 3.46 (s, 3H), 4.66 (t,  $J = 5.8$  Hz, 1H), 5.54 (t,  $J = 5.7$  Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 14.4, 23.0, 23.7, 28.4, 28.5, 29.5, 30.5, 32.1, 39.5, 54.3, 94.6, 123.5, 142.8, 158.9$ ; IR: ( $\text{cm}^{-1}$ )  $\nu$  2927, 2856, 2829, 1668, 1160; HRMS calcd for  $\text{C}_{14}\text{H}_{24}\text{O}$ : 208.1827. found: 208.1829.

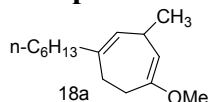
## Compound 16a



Using the representative procedure, a mixture of **15** (80 mg, 0.36 mmol), gold complex Au(P(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>)Cl (2.7 mg, 0.0036 mmol) and AgSbF<sub>6</sub> (1.2 mg, 0.0036 mmol) were reacted to give **16a** as a colorless oil. (68 mg, 0.31 mmol, 85 % yield). R<sub>f</sub> = 0.43 (pentane: ether = 95:5);

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.88 (t, *J* = 6.7 Hz, 3 H), 1.22-1.38 (m, 8H), 1.71 (s, 3H), 2.05 (app t, *J* = 7.4 Hz, 2H), 2.12 - 2.19 (m, 2H), 2.26 - 2.30 (m, 2H), 2.66 (d, *J* = 6.0 Hz, 2H), 3.45 (s, 3H), 4.68 (t, *J* = 6.0 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 14.5, 20.8, 23.0, 28.9, 29.5, 29.7, 30.4, 30.6, 32.2, 34.2, 54.0, 94.3, 132.6, 134.7, 157.7; IR: (cm<sup>-1</sup>) ν 2927, 2856, 1666, 1162; HRMS calcd for C<sub>15</sub>H<sub>26</sub>O: 222.1984. found: 222.1981.

### Compound 18a



(a) From the reaction of **17**

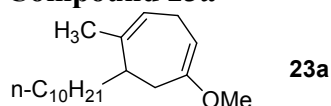
Using the representative procedure, a mixture of **17** (67 mg, 0.30 mmol), gold complex Au(P(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>)Cl (2.3 mg, 0.0030 mmol) and AgSbF<sub>6</sub> (1.0 mg, 0.0030 mmol) were reacted to give **18a** as a colorless oil. (60 mg, 0.27 mmol, 89 % yield). R<sub>f</sub> = 0.45 (pentane: ether = 95:5);

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.88 (t, *J* = 6.7 Hz, 3 H), 1.13 (d, *J* = 7.4 Hz, 3H), 1.21-1.42 (m, 8H), 1.96 (app t, *J* = 7.5 Hz, 2H), 2.01 - 2.18 (m, 2H), 2.31 - 2.43 (m, 2H), 3.11 - 3.22 (m, 1H), 3.46 (s, 3H), 4.42 (d, *J* = 3.3 Hz, 1H), 5.29 (d, *J* = 3.6 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 14.5, 23.0, 23.9, 28.4, 28.4, 28.6, 29.5, 30.2, 32.1, 39.4, 54.2, 102.6, 131.5, 141.0, 157.4; IR: (cm<sup>-1</sup>) ν 2956, 2928, 2856, 1659, 1202; HRMS calcd for C<sub>15</sub>H<sub>26</sub>O: 222.1984. found: 222.1988.

(b) From the reaction of **19**

Using the representative procedure, a mixture of **19** (120 mg, 0.54 mmol), gold complex Au(P(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>)Cl (4.1 mg, 0.0054 mmol) and AgSbF<sub>6</sub> (1.8 mg, 0.0054 mmol) were reacted to give **18a** as a colorless oil. (114 mg, 0.51 mmol, 95 % yield). R<sub>f</sub> = 0.77 (hexane : ethyl acetate = 95:5);

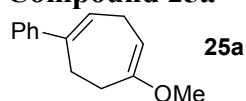
### Compound 23a



Using the representative procedure, a mixture of **22** (38 mg, 0.14 mmol), gold complex Au(P(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>)Cl (5.2 mg, 0.0068 mmol) and AgSbF<sub>6</sub> (2.3 mg, 0.0068 mmol) were reacted to give **23a** as a colorless oil. (36 mg, 0.13 mmol, 94 % yield). R<sub>f</sub> = 0.45 (pentane: ether = 95:5);

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 0.88 (t, *J* = 6.6 Hz, 3 H), 1.19 -1.57 (m, 18H), 1.69 (d, *J* = 1.2 Hz, 3H), 2.12 - 2.23 (m, 2H), 2.51 - 2.62 (m, 2H), 2.74-2.90 (m, 1H), 3.46 (s, 3H), 4.62 - 4.65 (m, 1H), 5.43 (app t, *J* = 4.9 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 14.5, 23.0, 24.7, 24.9, 28.1, 29.7, 29.9, 30.0, 30.2, 32.3, 32.5, 35.0, 40.1, 54.3, 94.3, 123.0, 141.2, 157.9; IR: (cm<sup>-1</sup>) ν 2924, 2854, 1672, 1145; Anal calcd for C<sub>19</sub>H<sub>34</sub>O: C, 81.95; H, 12.31; found: C, 81.94; H, 12.09.

### Compound 25a

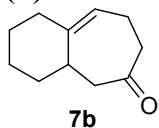


Using the representative procedure, a mixture of **24** (60 mg, 0.30 mmol), gold complex Au(P(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>)Cl (4.5 mg, 0.0060 mmol) and AgSbF<sub>6</sub> (2.1 mg, 0.0060 mmol) were reacted to give **25a** as a colorless oil. (58 mg, 0.29 mmol, 97 % yield). R<sub>f</sub> = 0.45 (pentane: ether = 95:5);

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.40 - 2.45 (m, 2H), 2.72 - 2.77 (m, 2H), 2.90 (app t,  $J$  = 6.0 Hz, 2H), 3.49 (s, 3H), 4.70 (t,  $J$  = 5.8 Hz, 1H), 6.15 (t,  $J$  = 6.2 Hz, 1H), 7.17 - 7.37 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 24.2, 28.2, 30.6, 54.3, 93.9, 125.9, 126.9, 128.2, 128.6, 142.3, 143.7, 158.6; IR: ( $\text{cm}^{-1}$ )  $\nu$  2950, 2850, 1734, 1704, 1491, 1446, 1105, 758, 699; HRMS calcd for  $\text{C}_{14}\text{H}_{16}\text{O}$ : 200.1201. found: 200.1204.

## Representative procedure for the conversion into the cyclohept-4-en-1-one

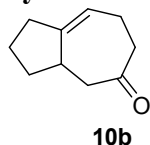
### (b) Conversion of **7a** to **7b**:



To a solution of **7a** (22mg, 0.12 mmol) in THF (1 mL) at  $0^\circ\text{C}$  was added water (0.1 mL) and p-toluenesulfonic acid (2.3 mg, 0.012 mmol). The reaction mixture was stirred for 7 h at  $0^\circ\text{C}$ , and then diluted with ether (20 mL). This solution was washed with saturated aq.  $\text{NaHCO}_3$  solution (2 x 10 mL), water (10 mL), dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated. The residual oil was purified by flash chromatography on silica gel (eluted with pentane : ether = 85:15) to give the compound **7b** as a colorless oil (18 mg, 0.11 mmol, 90% yield). The spectral data are in complete agreement with those in the literature.<sup>[2]</sup>

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 1.16-1.44 (m, 3H), 1.75-1.80 (m, 3H), 1.93 (m, 1H), 2.14 (m, 1H), 2.32 (m, 2H), 2.46 (m, 1H), 2.54 (m, 2H), 2.74 (dd,  $J$  = 4.5, 4.5 Hz, 1H), 2.82 (dd,  $J$  = 15, 9 Hz, 1H), 5.57 (m, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 22.1, 26.9, 28.8, 36.5, 38.6, 41.2, 44.4, 47.5, 120.7, 144.1, 213.6; IR: ( $\text{cm}^{-1}$ )  $\nu$  3053, 1700, 1419, 1265, 739, 704.

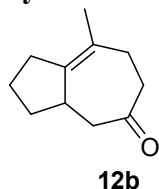
### Synthesis of **10b**



Using the representative procedure, **10a** (33 mg, 0.20 mmol) was converted to **10b** (27 mg, 0.18 mmol, 90% yield). The spectral data are in complete agreement with the literature value.<sup>[2]</sup>

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 1.27-1.40 (m, 1H), 1.50-1.74 (m, 2H), 2.04 (m, 1H), 2.26-2.48 (m, 6H), 2.63 (dd,  $J$  = 2.7, 2.7 Hz, 1H), 2.76-2.90 (m, 2H), 5.61 (m, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.3, 25.9, 35.1, 35.6, 38.4, 42.7, 49.9, 119.6, 148.5, 213.5; IR: ( $\text{cm}^{-1}$ )  $\nu$  3053, 2986, 2932, 2856, 2685, 2410, 1305, 1702, 1422, 1261, 895, 828.

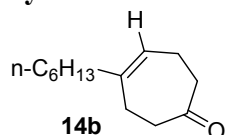
### Synthesis of **12b**



Using the representative procedure, **12a** (32 mg, 0.18 mmol) was converted to **12b** (26 mg, 0.16 mmol, 88% yield). The spectral data are in complete agreement with the literature value.<sup>[3]</sup>

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 1.24-1.35 (m, 1H), 1.50-1.56 (m, 1H), 1.66 (s, 3H), 1.69-1.77 (m, 1H), 1.92-1.98 (m, 1H), 2.30-2.43 (m, 6H), 2.58 (dd,  $J$  = 3, 3 Hz, 1H), 2.79-2.92 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 22.1, 25.3, 32.2, 32.9, 36.1, 39.0, 42.4, 49.7, 126.4, 140.6, 213.6; IR: ( $\text{cm}^{-1}$ )  $\nu$  3053.8, 2986, 2685, 2410, 2305, 1702, 1551, 1421, 1271, 895; HRMS calcd for  $\text{C}_{11}\text{H}_{16}\text{O}$ :  $m/z$  164.1201. found 164.1202.

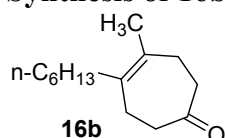
### Synthesis of 14b



Using the representative procedure, **14a** (31 mg, 0.15 mmol) was converted to **14b** (24 mg, 0.013 mmol, 84 % yield).

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 0.9 (t,  $J$  = 6.6 Hz, 3H), 1.27-1.41(m, 8H), 2.00 (t,  $J$  = 7.5 Hz, 2H), 2.27-2.34 (m, 4H), 2.56-2.62 (m, 4H), 5.56 (t,  $J$  = 4.8 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 14.5, 23.0, 24.1, 28.0, 28.4, 29.3, 32.1, 40.4, 42.7, 43.1, 123.7, 142.3, 214.0; IR: ( $\text{cm}^{-1}$ )  $\nu$  3053, 2928, 2856, 2685, 2305, 1702, 1422, 1378, 1263, 895, 738; Anal. calcd for  $\text{C}_{13}\text{H}_{22}\text{O}$ : C, 80.35; H, 11.41. found: C, 80.39; H, 11.32.

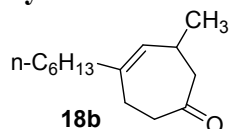
### Synthesis of 16b



Using the representative procedure, **16a** (26 mg, 0.12 mmol) was converted to **16b** (21 mg, 0.010 mmol, 85 % yield).

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 0.88 (t,  $J$  = 6.6 Hz, 3 H), 1.28 -1.38 (m, 8H), 1.74 (s, 3H), 2.06 (t,  $J$  = 7.3 Hz, 2H), 2.24 - 2.30 (m, 4H), 2.43 - 2.50 (m, 4H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 14.4, 20.9, 23.0, 28.6, 29.0, 29.6, 31.1, 32.2, 35.5, 42.7, 43.6, 130.6, 135.2, 213.4; IR: ( $\text{cm}^{-1}$ )  $\nu$  2954, 2926, 2856, 1738, 1710, 1464, 1377; HRMS calcd for  $\text{C}_{14}\text{H}_{24}\text{O}$ : 208.1827. found: 208.1829.

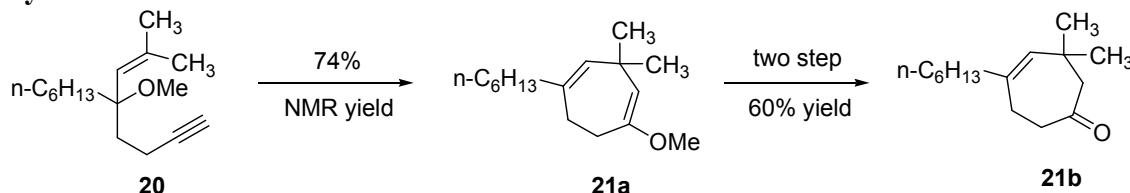
### Synthesis of 18b



Using the representative procedure, **18a** (44 mg, 0.20 mmol) was converted to **18b** (33 mg, 0.16 mmol, 80 % yield).

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 0.89 (t,  $J$  = 6.5 Hz, 3H), 1.09 (d,  $J$  = 6.8 Hz, 3H), 1.28-1.40 (m, 8H), 1.97-2.02 (m, 2H), 2.26-2.53 (m, 5H), 2.58-2.74 (m, 2H), 5.31 (d,  $J$  = 3.4 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 14.5, 23.0, 23.3, 27.7, 28.4, 29.3, 30.5, 32.1, 40.6, 42.9, 50.9, 130.7, 140.6, 213.1; IR: ( $\text{cm}^{-1}$ )  $\nu$  2957, 2927, 2856, 1708, 1457, 1405, 1277, 1120, 962, 830; HRMS calcd for  $\text{C}_{14}\text{H}_{24}\text{O}$ : m/z 208.1827. found 208.1824.

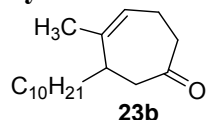
### Synthesis of 21b



Using the representative procedure, a mixture of **20** (68 mg, 0.29 mmol), gold complex  $\text{Au}(\text{P}(\text{C}_6\text{F}_5)_3)\text{Cl}$  (4.4 mg, 0.0057 mmol) and  $\text{AgSbF}_6$  (2.0 mg, 0.0057 mmol) were reacted to give **21a** as a colorless oil. (51 mg, 0.22 mmol, 74 % yield). The yield of this reaction was approximated as 74%, which was determined by  $^1\text{H}$  NMR using 1,3,5-trimethoxybenzene as an internal standard. Crude reaction mixture was treated with catalytic p-TsOH (10 %) to give **21b** as a colorless oil (38 mg, 0.17 mmol, 60 % yield for two step conversion)

$R_f = 0.45$  (pentane: ether = 95:5);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 0.89$  (t,  $J = 6.6$  Hz, 3H), 1.05 (s, 6H), 1.27-1.37 (m, 8H), 1.95-2.00 (m, 2H), 2.31-2.35 (m, 2H), 2.46-2.66 (m, 2H), 2.66 (s, 2H), 5.13 (s, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 14.1, 22.6, 26.3, 28.0, 28.8, 31.2, 31.7, 35.6, 41.4, 43.3, 53.8, 133.9, 137.0, 212.7$ ; IR: ( $\text{cm}^{-1}$ )  $\nu$  2957, 2928, 2857, 1707, 1466, 1389, 1363, 1232, 918; HRMS calcd for  $\text{C}_{15}\text{H}_{26}\text{O}$ :  $m/z$  222.1984. found 222.1987.

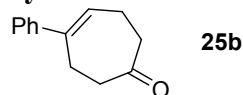
### Synthesis of **23b**



Using the representative procedure, **23a** (28 mg, 0.10 mmol) was converted to **23b** (24 mg, 0.090 mmol, 90 % yield).

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 0.88$  (t,  $J = 6.7$  Hz, 3 H), 1.19 -1.41 (m, 18H), 1.74 (s, 3H), 2.27 - 2.33 (m, 3H), 2.45 - 2.49 (m, 2H), 2.63 - 2.75 (m, 2H), 5.59 (app t,  $J = 6.3$  Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 14.5, 22.9, 23.0, 25.4, 27.4, 29.7, 29.9, 30.0, 32.3, 33.5, 41.5, 44.3, 46.0, 124.1, 141.6, 213.7$ ; IR: ( $\text{cm}^{-1}$ )  $\nu$  2925, 2854, 1745, 1709, 1465, 1378; HRMS calcd for  $\text{C}_{18}\text{H}_{32}\text{O}$ :  $m/z$  264.2453. found 264.2455.

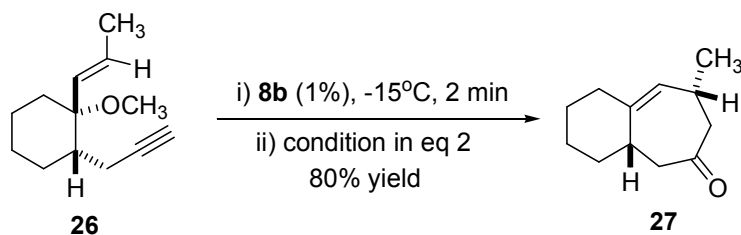
### Synthesis of **25b**



Using the representative procedure, compound **25a** (40 mg, 0.20 mmol) was converted into **25b** (28 mg, 0.15 mmol, 74 % yield). The spectral data are in full accord with those in the literature.<sup>[4]</sup>

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 2.50$ -2.56 (m, 2H), 2.67-2.71 (m, 2H), 2.74-2.82 (m, 4H), 6.11 (t,  $J = 5.9$  Hz, 1H), 7.26-7.27 (m, 1H), 7.29-7.34 (m, 4H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 24.7, 28.1, 42.8, 43.1, 126.2, 127.4, 128.0, 128.8, 142.5, 144.2, 213.1$ ; IR: ( $\text{cm}^{-1}$ )  $\nu$  3079, 3054, 3028, 1706, 1597, 1356, 1324, 1242, 763, 698.

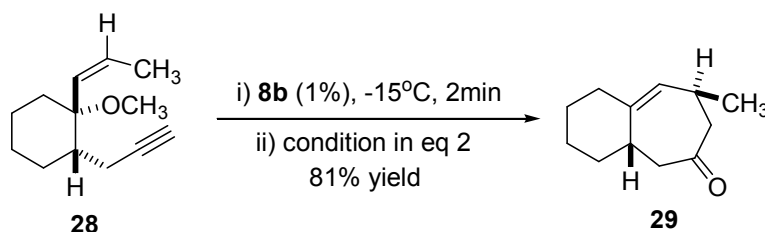
### Synthesis of **27**



Using the representative procedure, a mixture of **26** (125 mg, 0.65 mmol), gold complex  $\text{Au}(\text{P}(\text{C}_6\text{F}_5)_3)\text{Cl}$  (5.0 mg, 0.0065 mmol) and  $\text{AgSbF}_6$  (2.2 mg, 0.0065 mmol) were reacted in  $\text{CH}_2\text{Cl}_2$  (13 mL) to give the crude cycloheptadiene as a colorless oil. The yield of this reaction was approximated as 90%, which was determined by  $^1\text{H}$  NMR using 1,3,5-trimethoxybenzene as an internal standard. The crude reaction mixture was treated with catalytic  $p\text{-TsOH}$  (10 %) for 7 hours at  $0^\circ\text{C}$  to give **27** as a white solid (93 mg, 0.52 mmol, 80 % yield for two step conversion)

$R_f = 0.45$  (pentane: ether = 95:5); m. p  $30$ - $31^\circ\text{C}$  (from ether/pentane);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 1.03$  (d,  $J = 7.1$  Hz, 3H), 1.08-1.48 (m, 3H), 1.62-1.82 (m, 3H), 1.88-2.03 (m, 1H), 2.07-2.22 (m, 2H), 2.25-2.52 (m, 3H), 2.85-3.04 (m, 1H), 3.47 (dd,  $J = 12.2, 4.9$  Hz, 1H), 5.22 (s, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 23.3, 26.8, 27.7, 28.7, 35.3, 38.6, 40.3, 47.0, 53.4, 128.4, 142.7, 212.7$ ; IR: ( $\text{cm}^{-1}$ )  $\nu$  2928, 2855, 1704, 1444; HRMS calcd for  $\text{C}_{12}\text{H}_{18}\text{O}$ :  $m/z$  178.1358. found 178.1358.

## Synthesis of 29

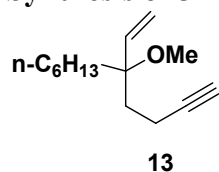


Using the representative procedure, a mixture of **28** (150 mg, 0.78 mmol), gold complex  $\text{Au}(\text{P}(\text{C}_6\text{F}_5)_3)\text{Cl}$  (6.0 mg, 0.0078 mmol) and  $\text{AgSbF}_6$  (2.7 mg, 0.0078 mmol) were reacted in  $\text{CH}_2\text{Cl}_2$  (16 mL) to give the crude cycloheptadiene as a colorless oil. The yield of this reaction was approximated as ~90%, which was determined by  $^1\text{H}$  NMR using 1,3,5-trimethoxybenzene as an internal standard. The crude reaction mixture was treated with catalytic *p*-TsOH (10 %) for 7 hours at  $0^\circ\text{C}$  to give **29** as a colorless oil (113 mg, 0.63 mmol, 81 % yield for two step conversion)

$R_f = 0.45$  (pentane: ether = 95:5);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 1.03$  (d,  $J = 7.0$  Hz, 3H), 1.18-1.46 (m, 3H), 1.70-1.82 (m, 3H), 1.83-1.95 (m, 1H), 2.10-2.16 (m, 1H), 2.18-2.34 (m, 3H), 2.43-2.51 (m, 1H), 2.99-3.19 (m, 2H), 5.19 (s, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 23.0, 26.6, 27.3, 28.6, 36.8, 38.4, 41.5, 47.9, 52.5, 128.3, 142.2, 212.8$ ; IR: ( $\text{cm}^{-1}$ )  $\nu$  2928, 2854, 1709, 1446; HRMS calcd for  $\text{C}_{12}\text{H}_{18}\text{O}$ :  $m/z$  178.1358. found 178.1358.

## Representative procedure for the preparation of substrates

### Synthesis of 5-methoxy-5-vinylundec-1-yne (**13**).

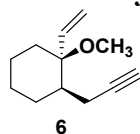


To a solution of sodium hydride (185 mg, 4.64 mmol, 60% in oil) in 15 mL of DMF was added a solution of 5-vinylundec-1-yn-5-ol (600 mg, 3.09 mmol) in DMF dropwise at  $0^\circ\text{C}$  under nitrogen. The reaction was maintained at  $0^\circ\text{C}$  for 1 h prior to the slow addition of methyl iodide (578  $\mu\text{l}$ , 9.27 mmol). The resulting reaction mixture was stirred for 6 h before being quenched with ice-water.

The reaction mixture was diluted with water and extracted with ether (3 x 10 ml). The combined organic layers were washed with brine (10 ml), dried ( $\text{Na}_2\text{SO}_4$ ) and concentrated to give a crude compound. Crude thus obtained was purified by silica gel column chromatography (eluted with EA : Hexane = 5 : 95) to afford 400 mg (62% yield) of **13** as colorless oil.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 0.88$  (t,  $J = 6.4$  Hz, 3H), 1.46-1.51 (m, 2H), 1.26 (m, 8H), 1.80-1.86 (m, 2H), 1.93 (t,  $J = 2.6$  Hz, 1H), 2.11-2.17 (m, 2H), 3.12 (s, 3H), 5.11-5.17 (dd,  $J = 1.3, 17.6$  Hz, 1H), 5.20-5.24 (dd,  $J = 1.3, 11.0$  Hz, 1H), 5.59-5.68 (dd,  $J = 11.1, 17.6$  Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 12.5, 14.1, 22.6, 23.0, 29.7, 31.8, 33.7, 35.2, 49.4, 69.9, 78.6, 84.9, 115.7, 141.3$ ; IR: ( $\text{cm}^{-1}$ )  $\nu$  3312, 2934, 2858, 2826, 2119, 1462, 1414, 1136, 1000, 925, 629; HRMS calcd for  $\text{C}_{14}\text{H}_{24}\text{O}$ :  $m/z$  208.1827. found 208.1825.

### 1-methoxy-2-(prop-2-ynyl)-1-vinylcyclohexane (**6**)

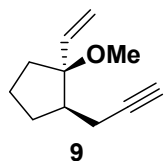


Yield: 91%.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 1.20$ -1.34 (m, 3H), 1.42-1.56 (m, 3H), 1.69-1.76 (m, 1H), 1.88-1.20 (m, 3H), 2.02-2.11 (m, 1H), 2.44-2.51 (m, 1H), 3.10 (s, 3H), 5.06-5.12 (dd,  $J = 1.4, 17.7$  Hz, 1H), 5.20-5.24 (dd,  $J = 1.4, 11.0$  Hz, 1H), 5.65-5.75 (dd,  $J = 11.1, 17.7$  Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz,



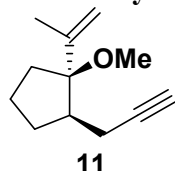
CDCl<sub>3</sub>):  $\delta$  = 19.1, 21.6, 25.7, 27.0, 31.3, 45.6, 49.7, 68.7, 77.9, 85.4, 115.9, 141.7; IR: (cm<sup>-1</sup>)  $\nu$  3309, 2941, 2857, 2826, 1638, 1449, 1148, 620; HRMS calcd for C<sub>12</sub>H<sub>18</sub>O: m/z 178.1358. found 178.1362.

### 1-methoxy-2-(prop-2-ynyl)-1-vinylcyclopentane (9)



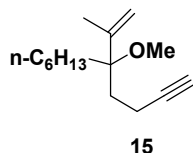
Yield: 42 %. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 1.55-1.71 (m, 4H), 1.83-1.93 (m, 2H), 2.00-2.06 (m, 2H), 2.07-2.19 (m, 1H), 2.31-2.40 (m, 1H), 3.10 (s, 3H), 5.11-5.21 (m, 2H), 5.71-5.80 (dd,  $J$  = 10.98, 17.43 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 17.5, 21.0, 29.7, 32.0, 49.3, 50.8, 68.2, 85.3, 86.5, 115.5, 139.8; IR: (cm<sup>-1</sup>)  $\nu$  3310, 3086, 2964, 2875, 2826, 2118, 1640, 1449, 1181, 1164, 1071, 627; HRMS calcd for C<sub>11</sub>H<sub>16</sub>O: m/z 164.1201. found 164.1205.

### 1-methoxy-1-(prop-1-en-2-yl)-2-(prop-2-ynyl)cyclopentane (11)



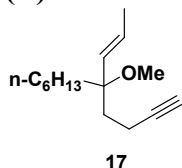
Yield: 61%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 1.56-1.68 (m, 3H), 1.70 (s, 3H), 1.80-1.93 (m, 3H), 1.96-2.10 (m, 2H), 2.10-2.16 (m, 1H), 2.32-2.40 (m, 1H), 3.06 (s, 3H), 4.91 (d,  $J$  = 1.1 Hz, 1H), 4.99 (d,  $J$  = 1.5 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 18.1, 19.7, 21.2, 29.3, 31.2, 46.5, 50.6, 67.8, 85.1, 88.4, 112.8, 144.2; IR: (cm<sup>-1</sup>)  $\nu$  3309, 2964, 2874, 2117, 1640, 1448, 1216, 1168, 1105, 1077, 906, 626; HRMS calcd for C<sub>12</sub>H<sub>18</sub>O: m/z 178.1358. found 178.1357.

### 5-methoxy-5-(prop-1-en-2-yl)undec-1-yne (15)



Yield: 47%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.88 (t,  $J$  = 6.4 Hz, 3H), 1.13-1.16 (m, 2H), 1.26 (m, 6H), 1.46-1.52 (m, 2H), 1.67 (s, 3H), 1.82-1.88 (m, 2H), 1.93 (t,  $J$  = 2.61 Hz, 1H), 2.04-2.08 (m, 2H), 3.05 (s, 3H), 4.93 (s, 1H), 5.03 (d,  $J$  = 1.3 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 12.6, 14.1, 18.9, 22.7, 23.1, 29.7, 31.4, 31.8, 32.8, 49.2, 67.9, 80.7, 84.9, 114.3, 145.6; IR (cm<sup>-1</sup>):  $\nu$  3312, 2935, 2872, 2858, 2824, 2119, 1641, 1466, 1451, 1376, 1192, 1128, 1082, 905, 629; Anal. calcd for C<sub>15</sub>H<sub>26</sub>O: C 81.29; H 11.94. found: C 81.12 H 11.86.

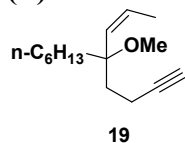
### (E)-5-methoxy-5-(prop-1-enyl)undec-1-yne (17)



Yield: 41%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.88 (t,  $J$  = 6.6 Hz, 3H), 1.19-1.33 (m, 8H), 1.44-1.49 (m, 2H), 1.74-1.71 (dd,  $J$  = 6.4, 1.6 Hz, 3H), 1.82-1.75 (m, 2H), 1.93 (t,  $J$  = 2.64 Hz, 1H), 2.10-2.16 (m, 2H), 3.09 (s, 3H), 5.23-5.28 (dq,  $J$  = 15.8, 1.44 Hz, 1H), 5.50-5.60 (dq,  $J$  = 6.5, 15.8 Hz, 1H); <sup>13</sup>C NMR (75

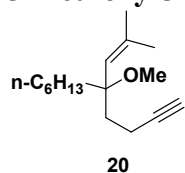
MHz, CDCl<sub>3</sub>):  $\delta$  = 12.6, 14.1, 18.0, 22.7, 23.1, 29.8, 31.8, 34.1, 35.5, 49.3, 67.8, 78.2, 85.0, 126.4, 134.3; IR: (cm<sup>-1</sup>)  $\nu$  3312, 2935, 2857, 2824, 1460, 1377, 1195, 1080, 627; Anal. calcd for C<sub>15</sub>H<sub>26</sub>O: C 81.04; H 11.83. found: C 81.02; H 11.79.

**(Z)-5-methoxy-5-(prop-1-enyl)undec-1-yne (19)**



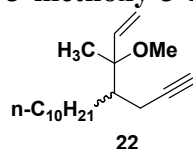
Yield: 63%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.88 (t,  $J$  = 6.5 Hz, 3H), 1.27 (m, 8H), 1.52-1.55 (m, 2H), 1.76-1.79 (dd,  $J$  = 7.3, 1.8 Hz, 3H), 1.83-1.89 (m, 2H), 1.93 (t,  $J$  = 2.6 Hz, 1H), 2.15-2.21 (m, 2H), 3.12 (s, 3H), 5.02-5.07 (dq,  $J$  = 1.7, 11.9 Hz, 1H), 5.23-5.61 (dq,  $J$  = 7.2, 11.9 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 12.8, 14.1, 14.2, 22.7, 23.4, 29.8, 31.8, 35.4, 36.5, 49.1, 67.8, 79.9, 85.0, 127.6, 131.9; IR: (cm<sup>-1</sup>)  $\nu$  3312, 3013, 2933, 2859, 2829, 2119, 1651, 1463, 1377, 1263, 1102, 724, 689; Anal. calcd for C<sub>15</sub>H<sub>26</sub>O: C 81.02; H 11.79. found: C 81.16 H 12.12.

**5-methoxy-5-(2-methylprop-1-enyl)undec-1-yne (20)**



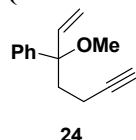
Yield = 60%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.88 (t,  $J$  = 6.3 Hz, 3H), 1.26 (m, 8H), 1.50-1.55 (m, 2H), 1.70 (s, 3H), 1.78 (s, 3H), 1.81-1.86 (m, 2H), 1.93 (t,  $J$  = 2.6 Hz, 1H), 2.18-2.12 (m, 2H), 3.08 (s, 3H), 4.85 (s, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 13.3, 14.5, 19.1, 23.1, 23.8, 27.7, 30.2, 32.2, 36.0, 37.0, 49.4, 68.1, 79.5, 85.5, 126.5, 136.2; IR: (cm<sup>-1</sup>)  $\nu$  3313, 2932, 2859, 2823, 2119, 1664, 1454, 1377, 1268, 1235, 1188, 1088, 830, 628; HRMS calcd for C<sub>16</sub>H<sub>28</sub>O:  $m/z$  236.2140. found 236.2141.

**3-methoxy-3-methyl-4-(prop-2-ynyl)tetradec-1-ene (22)**



Yield: 60 %. (A mixture of diastereomers). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.88 (t,  $J$  = 6.4 Hz, 3H), 1.20-1.26 (m, 19H), 1.32-1.49 (m, 2H), 1.61-1.63 (m, 1H), 1.91 (t,  $J$  = 2.7 Hz, 1H), 2.26-2.48 (m, 2H), 3.13 (s, 3H), 5.10-5.17 (dd,  $J$  = 1.2, 17.6 Hz, 1H), 5.23-5.28 (dd,  $J$  = 1.4, 10.9 Hz, 1H), 5.60-5.70 (dd,  $J$  = 10.9, 17.61 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 14.5, 16.5, 19.1, 23.1, 29.0, 29.5, 29.7, 29.9, 30.0, 30.3, 30.4, 32.3, 46.5, 50.2, 69.4, 80.3, 84.9, 116.7, 142.2; IR: (cm<sup>-1</sup>)  $\nu$  3312, 3084, 2925, 2854, 2825, 2117, 1465, 1412, 1375, 1157, 1107, 1059, 1002, 924, 864, 721, 628; Anal. calcd for C<sub>19</sub>H<sub>34</sub>O: C 81.95; H 12.31. found: C 81.93 H 12.56.

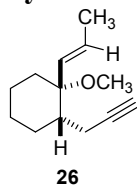
**(3-methoxyhept-1-en-6-yn-3-yl)benzene (24)**



Yield: 67%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 1.90-1.95 (m, 2H), 2.15-2.27 (m, 3H), 3.14 (s, 3H), 5.27-5.36 (m, 2H), 5.82-5.91 (dd,  $J$  = 10.6, 17.6 Hz, 1H), 7.21-7.38 (m, 5H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  =

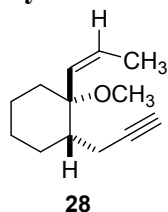
12.9, 34.8, 50.5, 68.0, 80.7, 84.7, 115.6, 126.5, 127.1, 128.1, 140.9, 142.3; IR: (cm<sup>-1</sup>)  $\nu$  3301, 2939, 2827, 1492, 1446, 1092, 1077, 899, 724, 700, 633; HRMS calcd for C<sub>14</sub>H<sub>16</sub>O: m/z 200.1201. found 200.1201.

### Synthesis of compound 26



Yield : 81%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 1.21-1.55 (m, 6H), 1.67-1.76 (m, 4H), 1.82-1.96 (m, 3H), 2.00-2.11 (m, 1H), 2.45-2.53 (m, 1H), 3.07 (s, 3H), 5.30-5.36 (m, 1H), 5.45 (m, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 18.3, 19.0, 21.5, 25.6, 27.0, 31.6, 45.9, 49.4, 68.5, 77.1, 85.5, 126.4, 134.5; IR: (cm<sup>-1</sup>)  $\nu$  3310, 2935, 2856, 2825, 2117, 1448, 1075, 625; HRMS calcd for C<sub>13</sub>H<sub>20</sub>O: m/z 192.1514 found 192.1513.

### Synthesis of compound 28



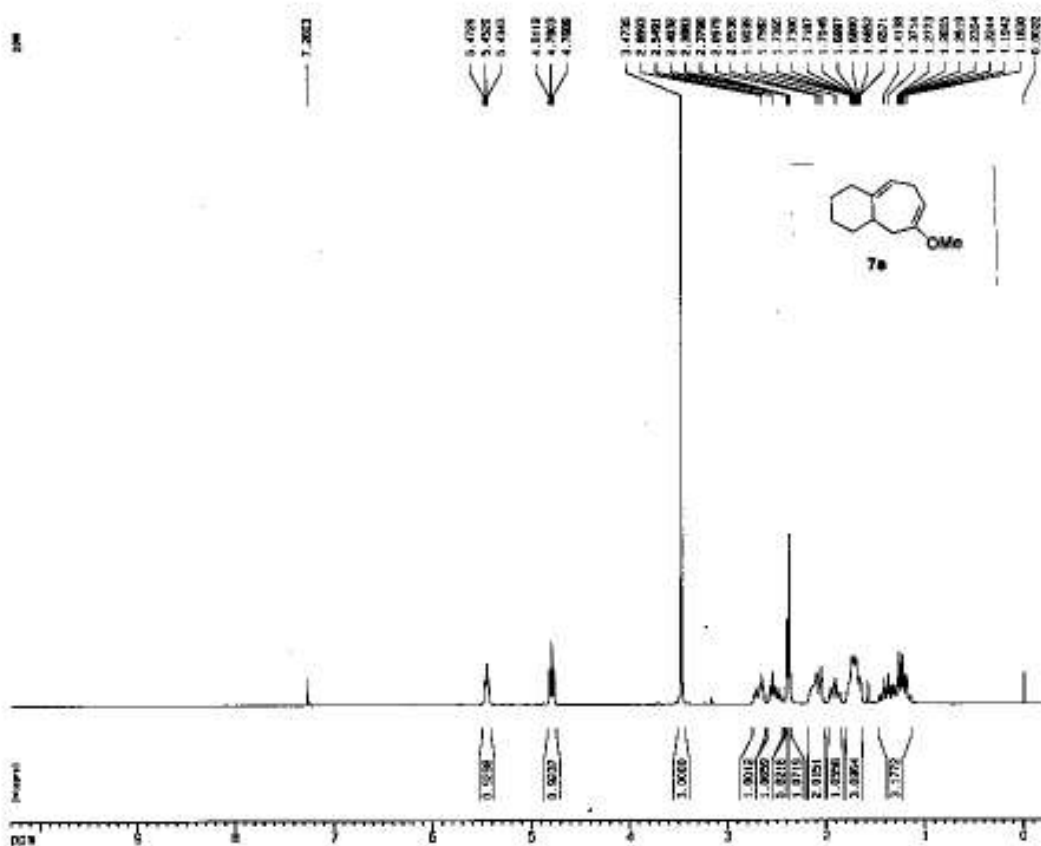
Yield : 84%. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 1.23-1.71 (m, 7H), 1.76 (dd,  $J$  = 7.2, 1.8 Hz, 3H), 1.85-2.02 (m, 3H), 2.07-2.19 (m, 1H), 2.48 (dt,  $J$  = 16.7, 2.9 Hz, 1H), 3.10 (s, 3H), 5.00-5.07 (m, 1H), 5.51-5.63 (m, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 14.6, 19.1, 21.5, 25.1, 26.4, 33.6, 44.8, 49.5, 68.5, 79.3, 85.4, 127.3, 132.2; IR: (cm<sup>-1</sup>)  $\nu$  3309, 2934, 2857, 2826, 2117, 1652, 1448, 1161, 1078, 743; HRMS calcd for C<sub>13</sub>H<sub>20</sub>O: m/z 192.1514 found 192.1513.

### References:

- [1] J.-E. Kang, H.-B. Kim, J.-W. Lee, S. Shin, *Org. Lett.* 2006, 8, 3537.
- [2] X. Li, R. E. Kyne, T. V. Ovaska, *J. Org. Chem.* 2007, 72, 6624 .
- [3] C. E. McIntosh, I. Matinez, T. V. Ovaska, *synlett* 2004, 2579.
- [4] P. A. Wender, H. Rieck, M. Fuji, *J. Am. Chem. Soc.* 1998, 120, 10976.



bnj-2-118-P-A 1 1 v yhr 20070513



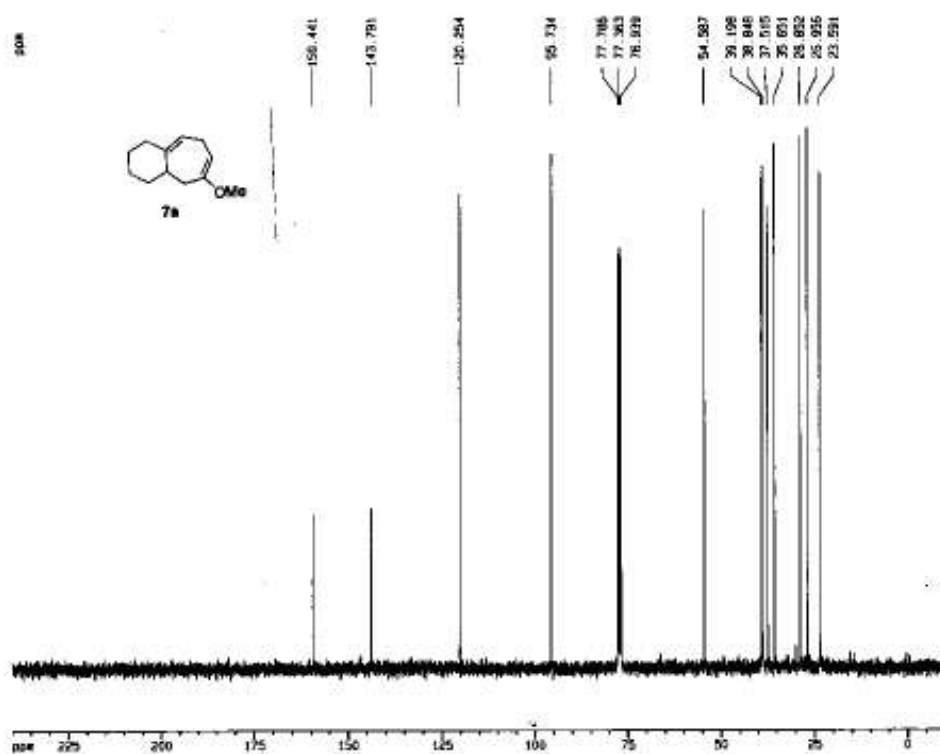
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PROCNO 1

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PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 320  
DS 4  
SWH 4784.800 Hz  
FIDRES 0.250044 Hz  
AQ 1.7023701 sec  
RG 114  
DM 195.000 MHz  
DE 4.50 uV  
IE 200.0 K  
QF 1.0000000 sec  
FI 15.00 MHz  
DC 4.50 uV  
SFO 300.136062 MHz  
NUC1 1H  
PL1 -5.00 dB

F2 - Processing parameters  
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SF 300.136062 MHz  
WDW EM  
SSB 0  
LB 0.20 Hz  
GB 0  
RC 1.00

GD NMR post parameters  
CH 27.00 cm  
F1 29.300 cm  
F2 3001.24 Hz  
F3 -15.000 cm  
F4 -80.00 Hz  
F5 0.47727 cm/cm  
XDC 143.04305 Hz/cm

bnj-2-115-P 1 3 v yhr 20070510

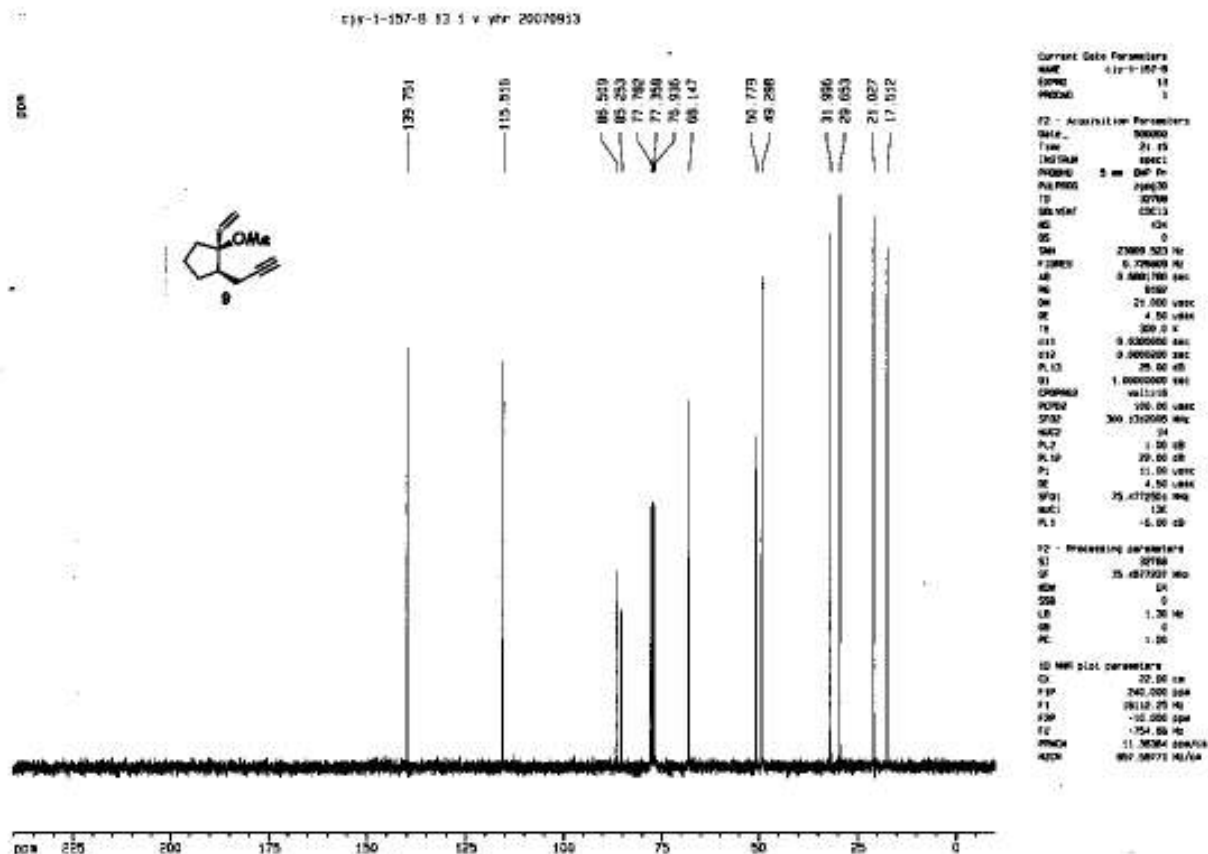


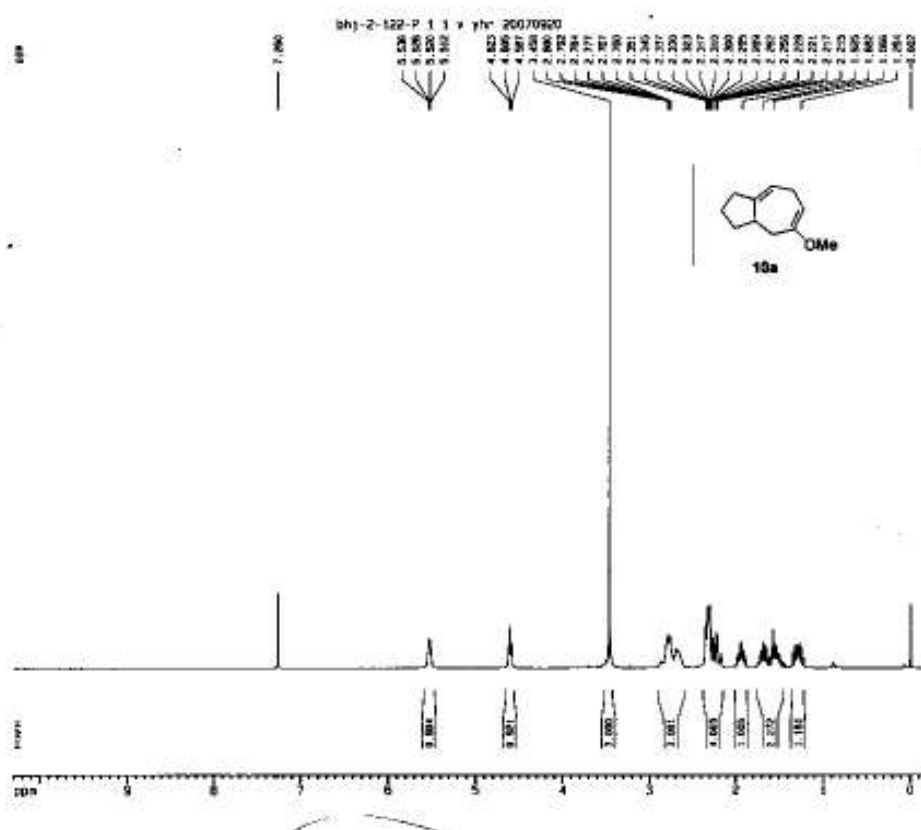
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PROCNO 1

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PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 1280  
DS 4  
SWH 23800.521 Hz  
FIDRES 0.230000 Hz  
AQ 0.001780 sec  
RG 8182  
DM 21.000 MHz  
DE 4.50 uV  
IE 200.0 K  
QF 0.0000000 sec  
FI 0.0000000 sec  
PL1 0.0000000 sec  
SFO 100.626120 MHz  
LPROG2 waltz16  
P2PRG2 100.00 MHz  
SFO2 300.136062 MHz  
NUC1 13C  
PL1 -5.00 dB

F2 - Processing parameters  
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WDW EM  
SSB 0  
LB 1.30 Hz  
GB 0  
RC 1.00

GD NMR post parameters  
CH 27.00 cm  
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F3 -15.000 cm  
F4 -794.36 Hz  
F5 11.36364 cm/cm  
XDC 857.22711 Hz/cm



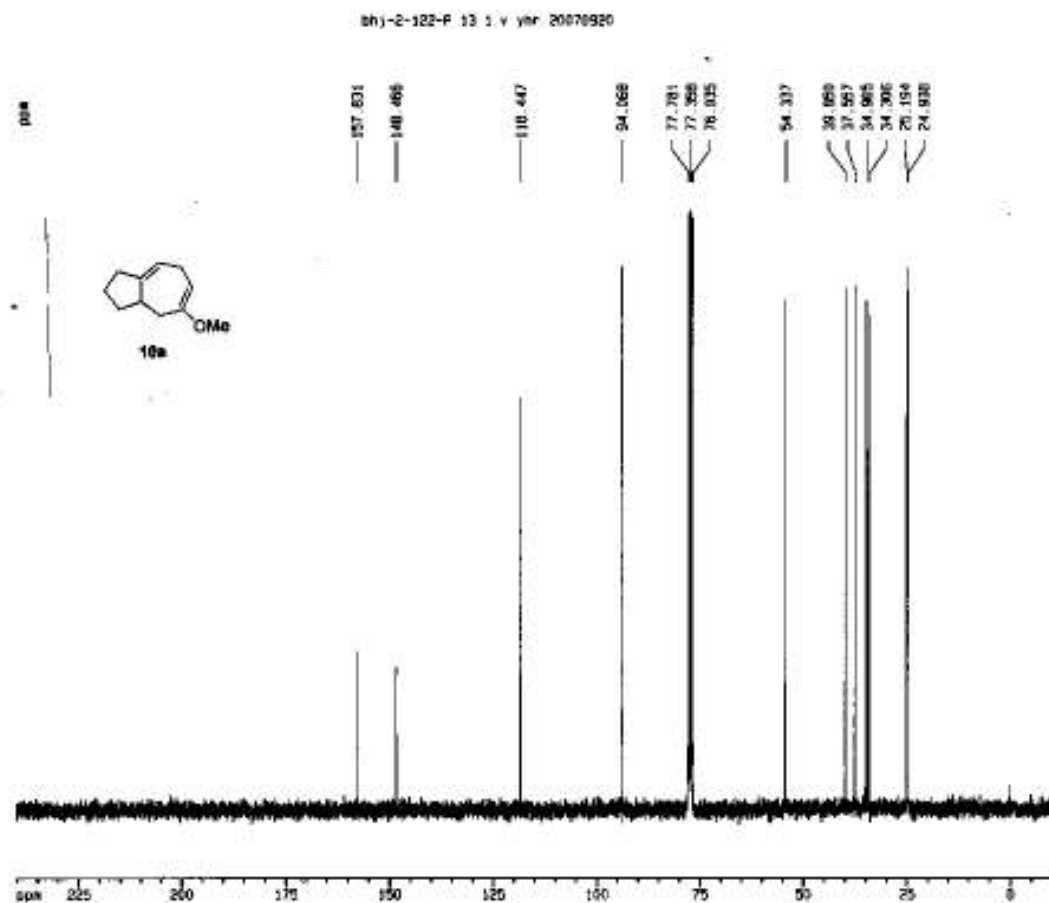


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EXPNO 1  
PROCNO 1

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PULPROG zgpg30  
TD 32768  
SOLVENT CDCl<sub>3</sub>  
NS 36  
DS 4  
SWH 6172.499 Hz  
FIDRES 0.000300 Hz  
AQ 2.6542960 sec  
RG 256  
DM 61.000 usec  
DE 4.50 usec  
TE 300.2 K  
D1 1.0000000 sec  
P1 8.00 usec  
DE 4.50 usec  
SFO1 300.1318534 MHz  
NUC1 1H  
NUC2  
PL1 1.00 dB

F2 - Processing parameters  
SI 16384  
SF 300.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR list parameters  
CX 20.00 cm  
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F1 9361.34 Hz  
FAP 0.300 ppm  
F2 -60.00 Hz  
PRGCM 0.00000 ppm/cm  
KICK 150.00000 Hz/cm



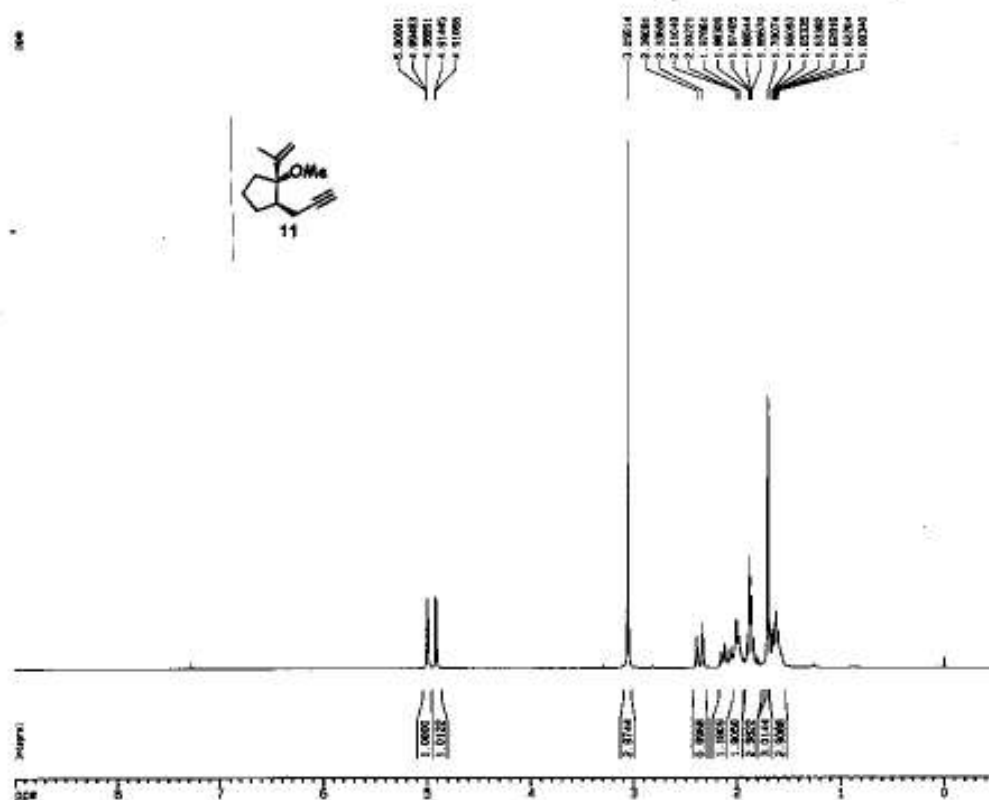
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EXPNO 1  
PROCNO 1

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PULPROG zgpg30  
TD 32768  
SOLVENT CDCl<sub>3</sub>  
NS 36  
DS 4  
SWH 23600.522 Hz  
FIDRES 0.725800 Hz  
AQ 0.0000000 sec  
RG 256  
DM 61.000 usec  
DE 4.50 usec  
TE 300.2 K  
D1 1.0000000 sec  
P1 8.00 usec  
DE 4.50 usec  
SFO1 100.6260550 MHz  
NUC1 13C  
NUC2  
PL1 1.00 dB

F2 - Processing parameters  
SI 16384  
SF 100.6260550 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR list parameters  
CX 20.00 cm  
FAP 240.000 ppm  
F1 10132.25 Hz  
FAP 15.000 ppm  
F2 -75.00 Hz  
PRGCM 15.30364 ppm/cm  
KICK 857.56771 Hz/cm

88-3-38 C0C13 21 09 07



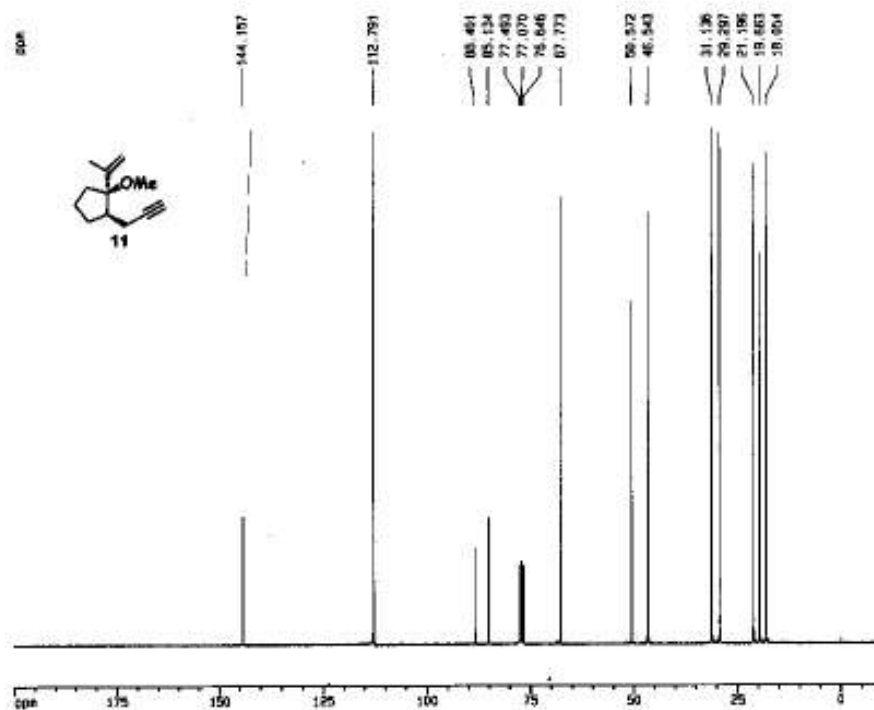
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PROCNO 1

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PULPROG zgpg30  
TD 65536  
FIDRES 0.16304  
SOLVENT CDCl3  
NS 30  
DS 2  
SWH 4781.900 Hz  
F2RES 0.200000 Hz  
AQ 1.720701 sec  
RG 40.2  
CW 255.000 kHz  
DE 4.50 uV  
TE 300.2 K  
D1 1.0000000 sec  
P1 15.00 uV  
DE 4.50 uV  
SFO1 300.131835 MHz  
NAC1 1H  
PL1 -6.00 dB

F2 - Processing parameters  
SI 18384  
SF 300.130017 MHz  
WDW EM  
SSB 0  
LB 0.20 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CH 20.00 cm  
FOP 0.000 ppm  
F1 2701.17 Hz  
F2 -0.500 ppm  
F3 -150.00 Hz  
PRNCH 0.43180 ppm/Hz  
HCHN 129.50125 Hz/Hz

88-3-38c13 C0C13 21 09 07



Current Data Parameters  
NAME 88-3-38c13  
EXPNO 4  
PROCNO 1

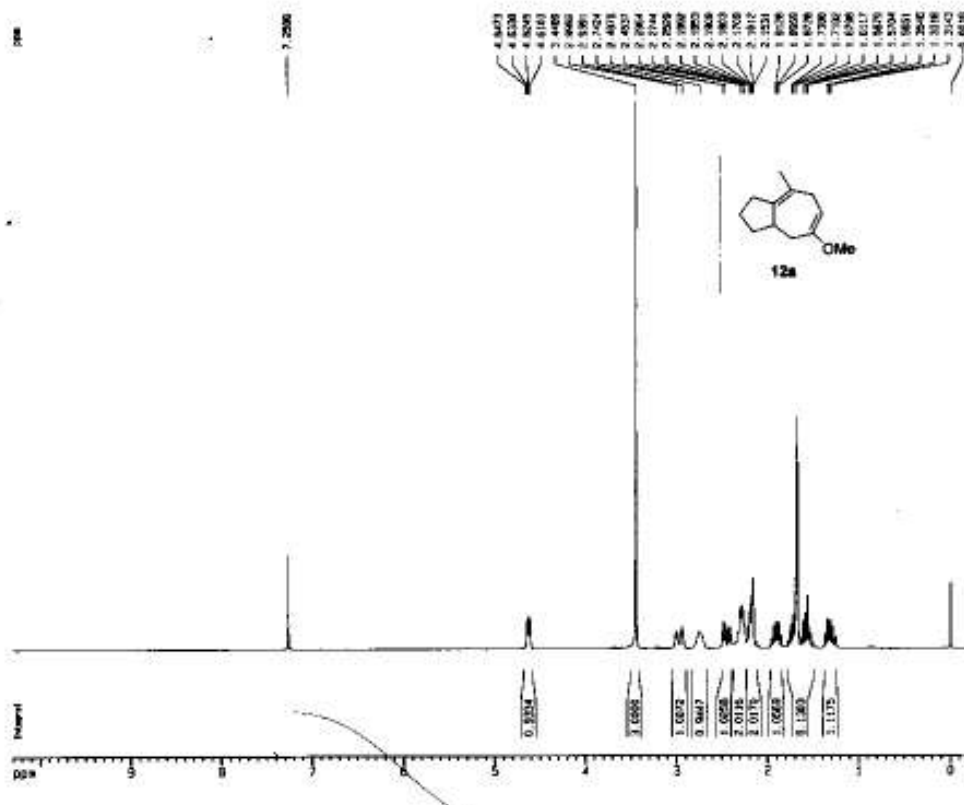
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Date\_ 20090201  
Time 21.27  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 65536  
FIDRES 0.16304  
SOLVENT CDCl3  
NS 30  
DS 2  
SWH 2907.844 Hz  
F2RES 0.200000 Hz  
AQ 0.0380340 sec  
RG 1024  
CW 25.500 kHz  
DE 4.50 uV  
TE 300.2 K  
D12 0.0000000 sec  
PL12 0.00 dB  
D1 1.0000000 sec  
PRNCH 0.43180 ppm/Hz  
HCHN 129.50125 Hz/Hz  
SFO1 300.130017 MHz  
NAC1 1H  
PL1 -6.00 dB  
PL12 0.00 dB  
P1 7.50 uV  
DE 4.50 uV  
SFO1 25.476098 MHz  
NAC1 13C  
PL1 0.0000000 sec

F2 - Processing parameters  
SI 32768  
SF 125.761450 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CH 20.00 cm  
FOP 0.000 ppm  
F1 125.761450 MHz  
F2 -15.500 ppm  
F3 -754.88 Hz  
PRNCH 0.43180 ppm/Hz  
HCHN 129.50125 Hz/Hz



hhj-2-124-P-A 1 1 v yhr 20070928



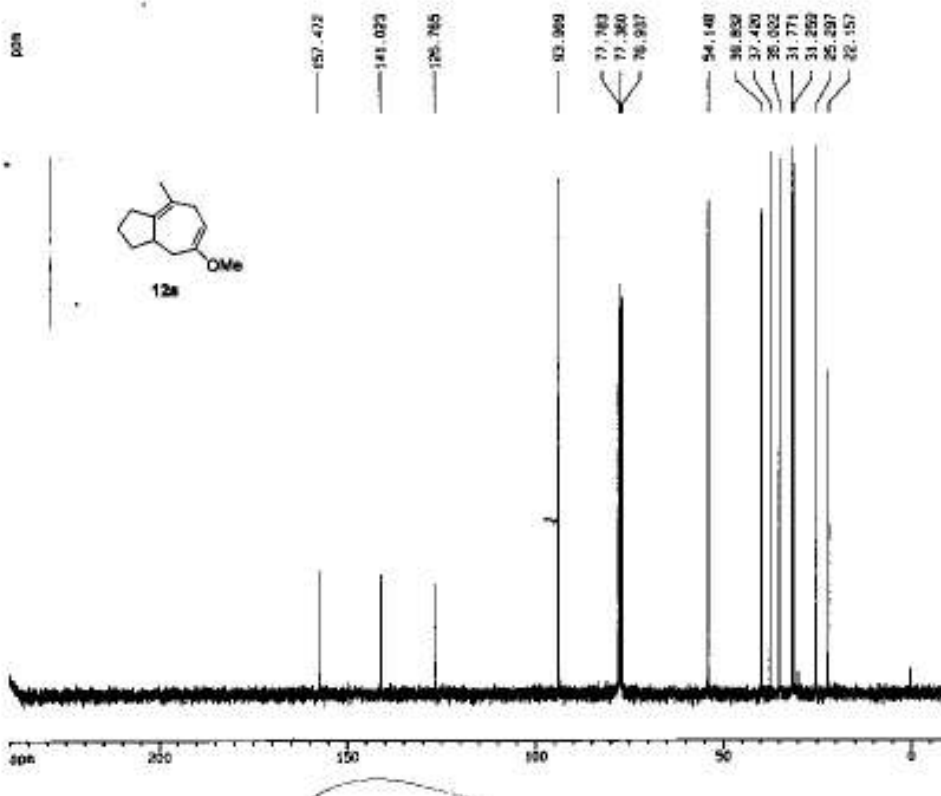
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PROCNO 1

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AQ 1.00000000  
RG 327.68  
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RG3 327.68  
RG4 327.68  
RG5 327.68  
RG6 327.68  
RG7 327.68  
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RG98 327.68  
RG99 327.68  
RG100 327.68

F2 - Processing parameters  
SI 32768  
SF 300.1360500 MHz  
WDW EM  
SSB 0  
LB 0.20 Hz  
GB 0  
RC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1 243.200 MHz  
F2 300.136050 MHz  
F3 300.136050 MHz  
WDW EM  
SSB 0  
LB 0.20 Hz  
GB 0  
RC 1.40

hhj-2-124-P-A 13 1 v yhr 20070928



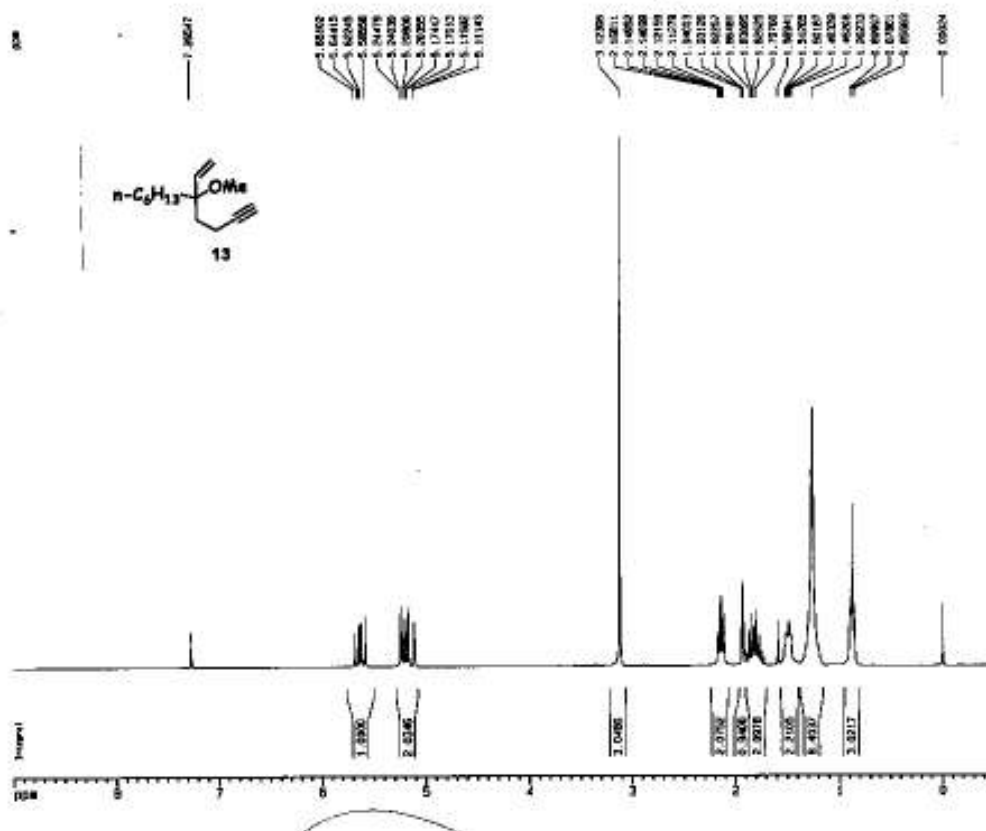
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RG98 327.68  
RG99 327.68  
RG100 327.68

F2 - Processing parameters  
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SSB 0  
LB 1.20 Hz  
GB 0  
RC 1.40

1D NMR plot parameters  
CX 20.00 cm  
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F2 300.136050 MHz  
F3 300.136050 MHz  
WDW EM  
SSB 0  
LB 1.20 Hz  
GB 0  
RC 1.40

88-3-40 CDCl3 26 09 07



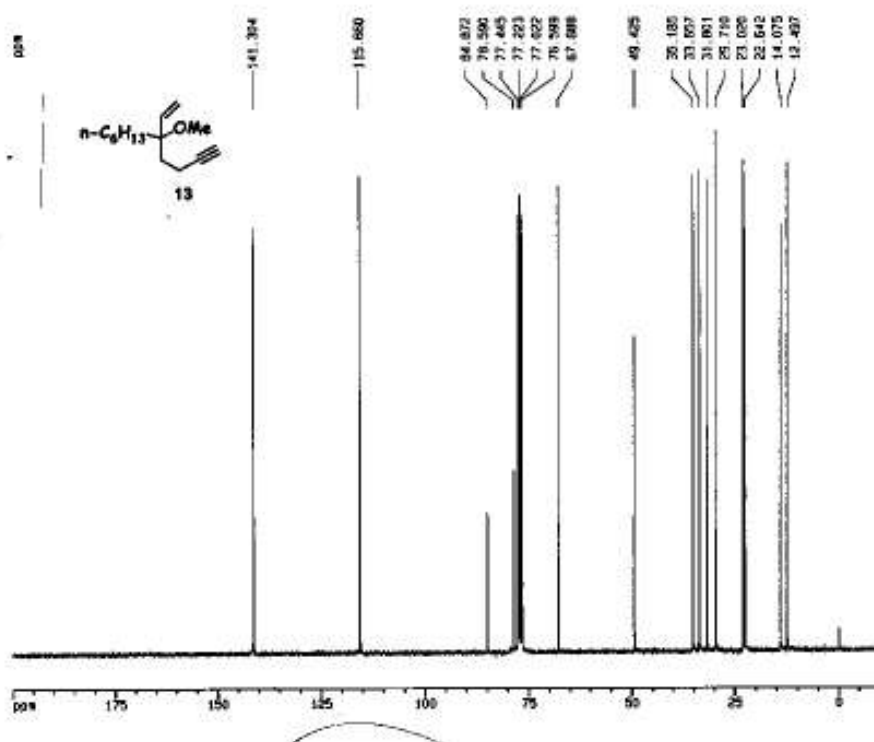
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PROCNO 1

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PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 0  
SWH 4701.025 Hz  
FIDRES 0.200644 Hz  
AQ 1.7003701 sec  
RG 184.3  
DE 105.000 uS  
TE 300.2 K  
D1 1.00000000 sec  
P1 15.00 uS  
DE 4.50 uS  
SFO1 300.131995 MHz  
HLC1 1H  
PL1 -6.00 dB

F2 - Processing parameters  
SI 1024  
SF 300.1300646 MHz  
WDW EM  
SSB 0  
LB 0.20 Hz  
GB 0  
PC 1.00

1D 1H 13C parameters  
CX 22.00 CH  
FAP 0.000 ppm  
P1 270.17 Hz  
FAP 0.000 ppm  
F2 100.07 Hz  
PACB 4.4000 ppm/Hz  
H2O 129.6055 Hz/Hz

88-3-40c13 CDCl3 26 09 07

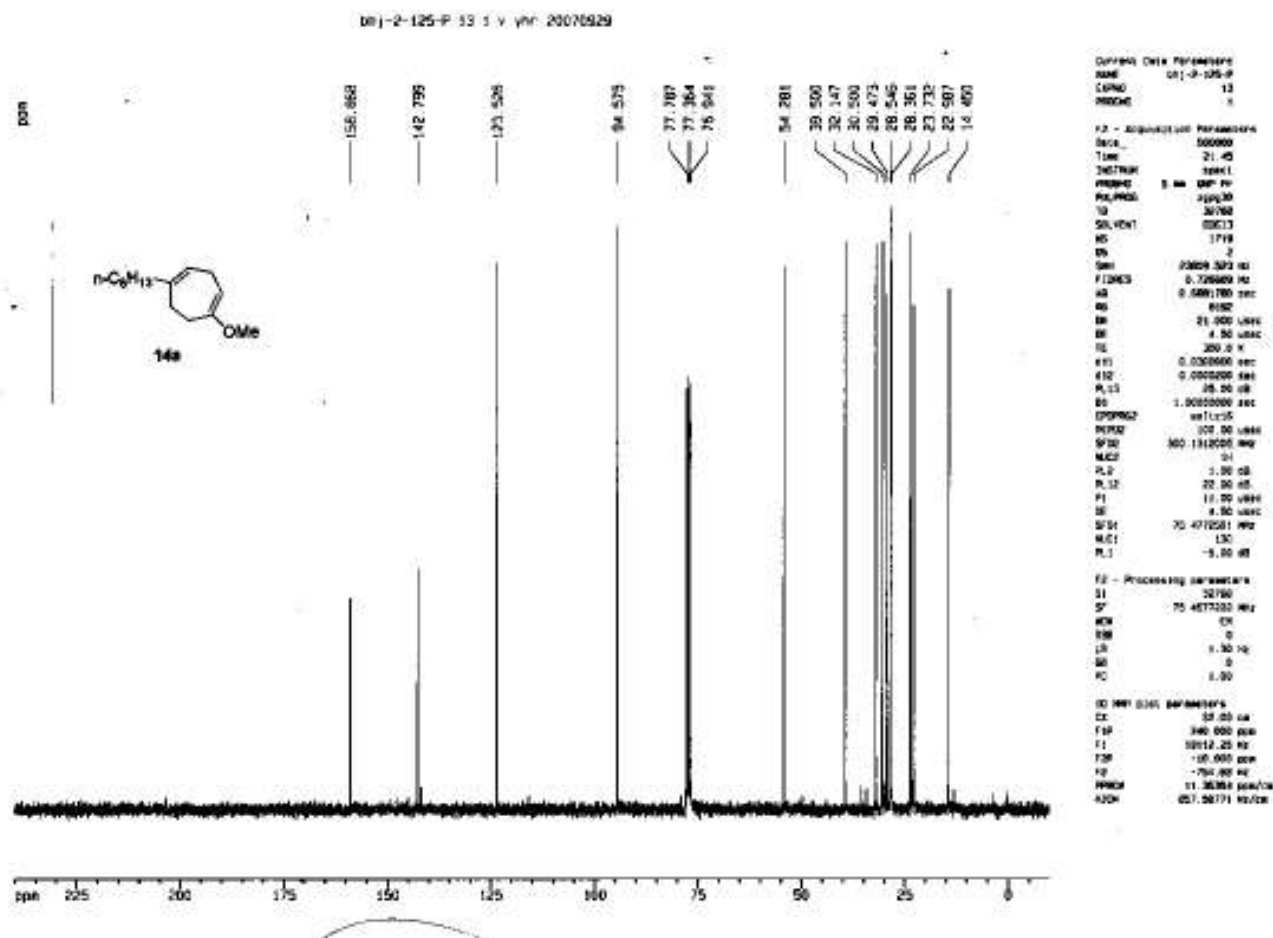
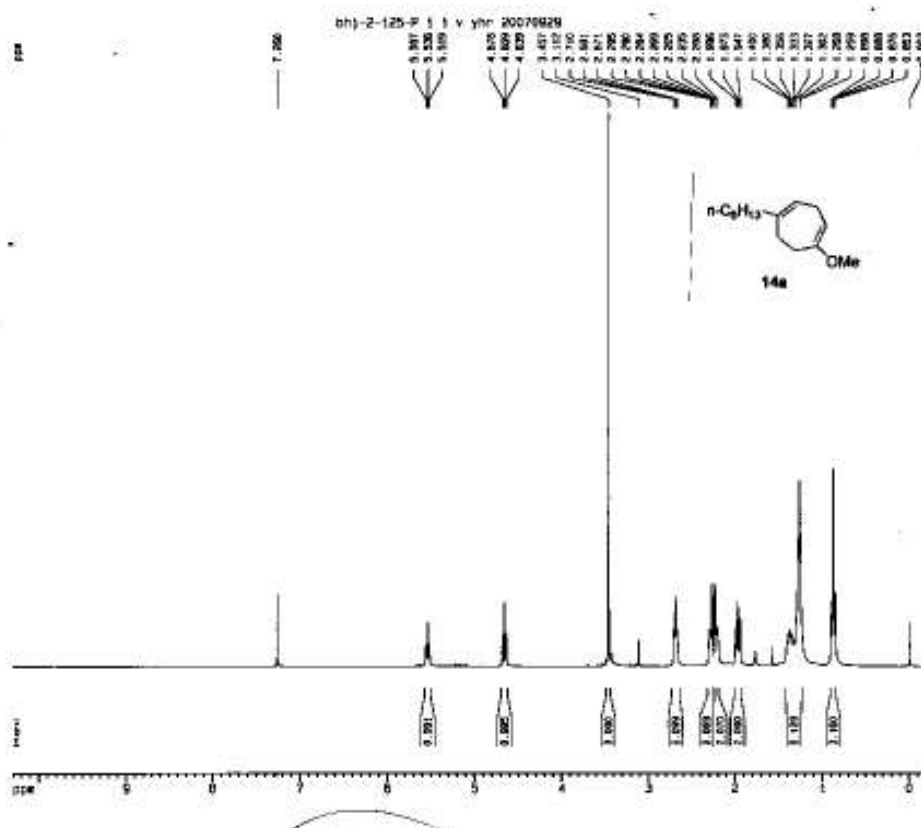


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EXPNO 6  
PROCNO 1

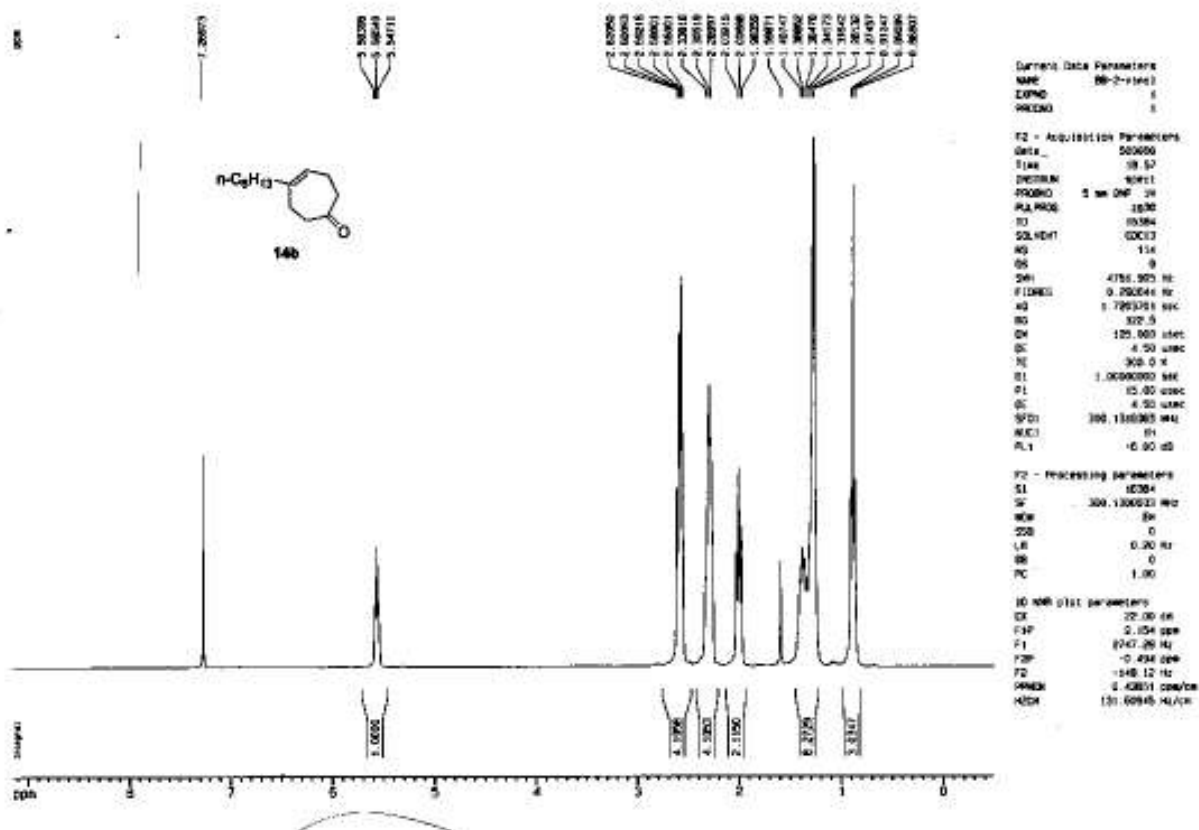
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Time 0.00  
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PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 0  
SWH 18607.864 Hz  
FIDRES 0.580064 Hz  
AQ 0.0000000 sec  
RG 1024  
DE 25.500 uS  
TE 300.2 K  
D12 0.0000000 sec  
PL12 10.00 dB  
D1 1.00000000 sec  
CPDPRG2 waltz16  
PCPD2 100.00 uS  
SFO1 300.1301000 MHz  
HLC2 1H  
PL2 -6.00 dB  
PL12 13.00 dB  
P1 7.50 uS  
DE 4.50 uS  
SFO1 75.4760000 MHz  
HLC1 13C  
PL1 -6.00 dB  
D11 0.0000000 sec

F2 - Processing parameters  
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WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00

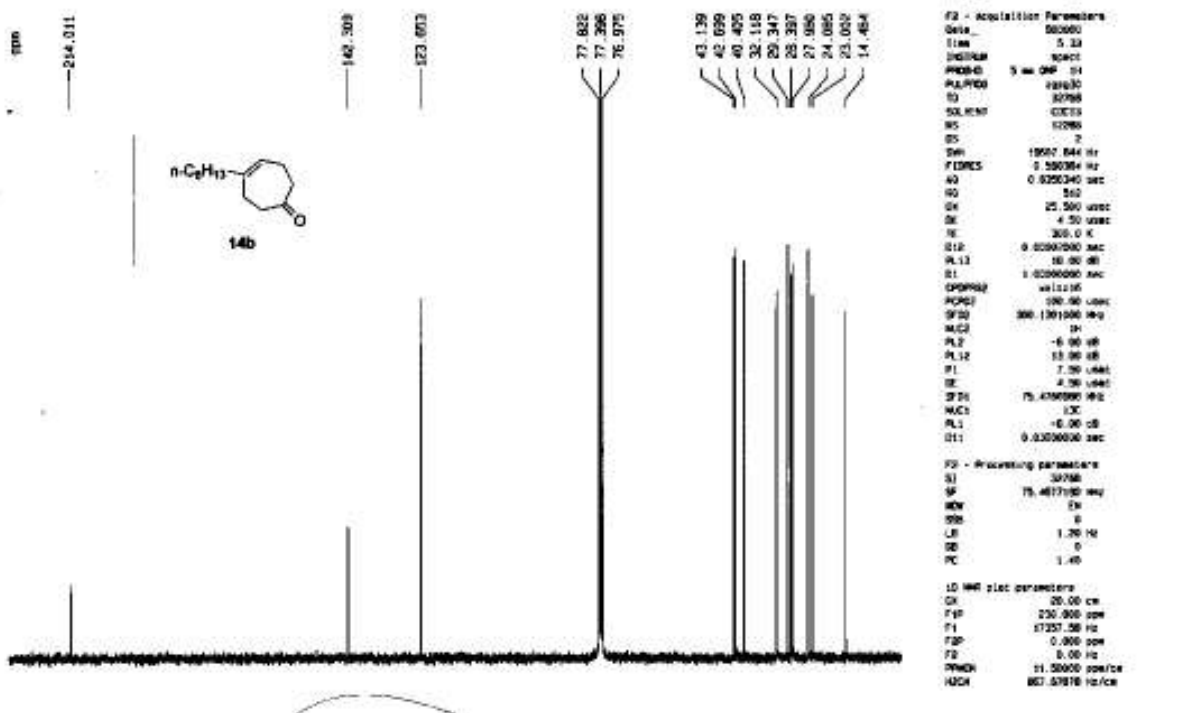
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FAP 0.000 ppm  
P1 1000.00 Hz  
FAP -10.000 ppm  
F2 75.46 Hz  
PACB 10.0000 ppm/Hz  
H2O 76.4118 Hz/Hz



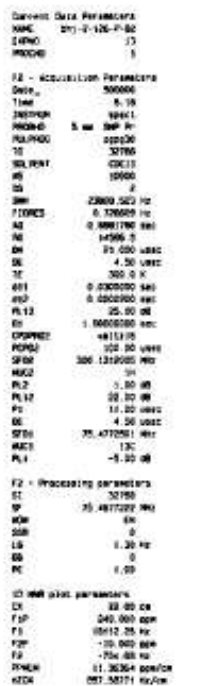
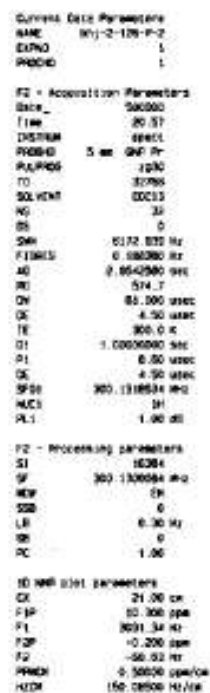
BB-2-vinyl C0C13 30 00 07

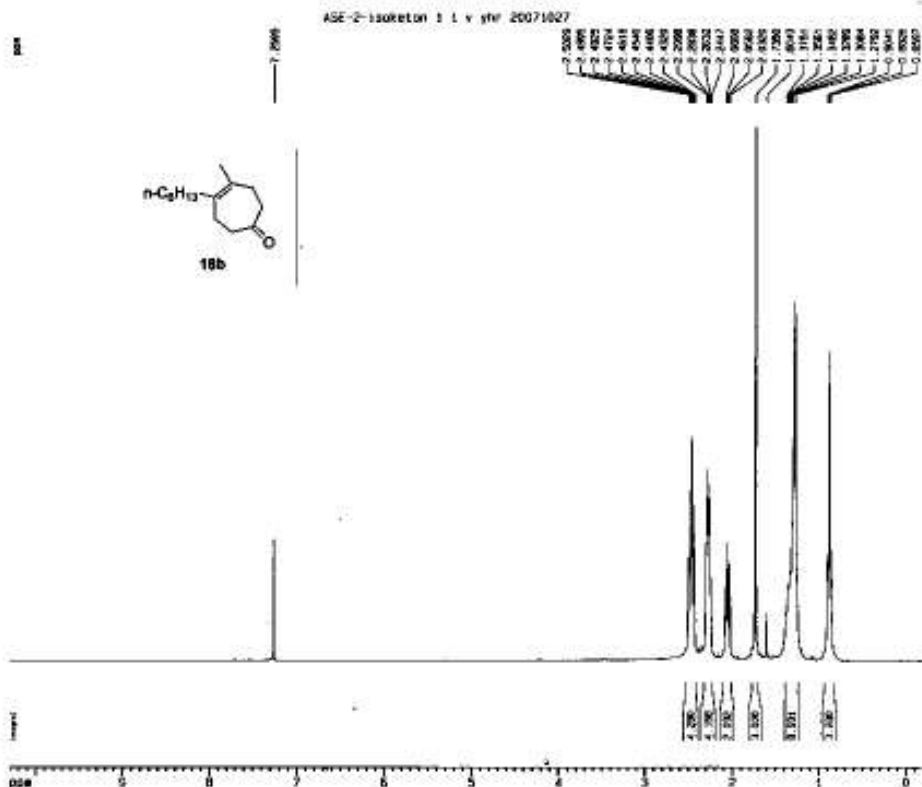


BB-2-216c13 DOC19 12 09 07







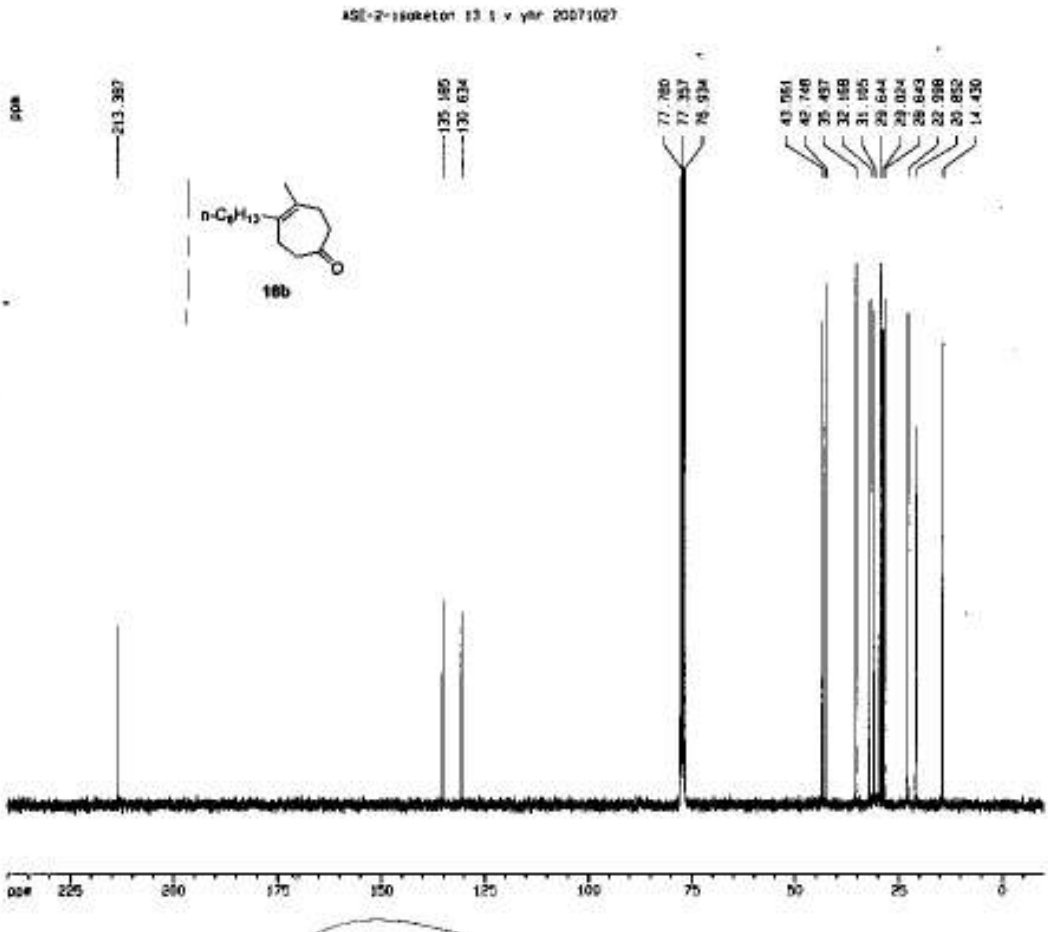


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EXPNO 1  
PROCNO 1

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PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 32  
DS 0  
SWH 5172.836 Hz  
FIDRES 0.186360 Hz  
AQ 2.8242500 sec  
RG 256  
DM 61.800 usec  
DE 4.50 usec  
TE 300.2 K  
D1 1.0000000 sec  
P1 8.00 usec  
DE 4.50 usec  
SFO1 300.1318534 MHz  
NAC1 131  
PC1 1.00 dB

F2 - Processing parameters  
SI 32768  
SF 300.1318534 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
FID 10.000 ppm  
F1 16012.25 Hz  
F2 -60.000 ppm  
F3 -60.00 Hz  
RGWD 0.0000000 Hz/cm  
KDCN 150.00000 Hz/cm



Current Data Parameters  
NAME ASE-2-Isoketon  
EXPNO 13  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 200706  
Time 0.18  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 8192  
DS 2  
SWH 73800.523 Hz  
FIDRES 0.144048 Hz  
AQ 0.8707000 sec  
RG 8192  
DM 20.000 usec  
DE 4.50 usec  
TE 300.2 K  
D1 0.0300000 sec  
P1 0.0300000 sec  
PL12 0.0000000 sec  
D1 1.0000000 sec  
CPDPRG2 waltz16  
PULPROG 100.00 usec  
SFO1 300.1318534 MHz  
NAC1 13C  
PC1 -1.00 dB

F2 - Processing parameters  
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SF 300.1318534 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

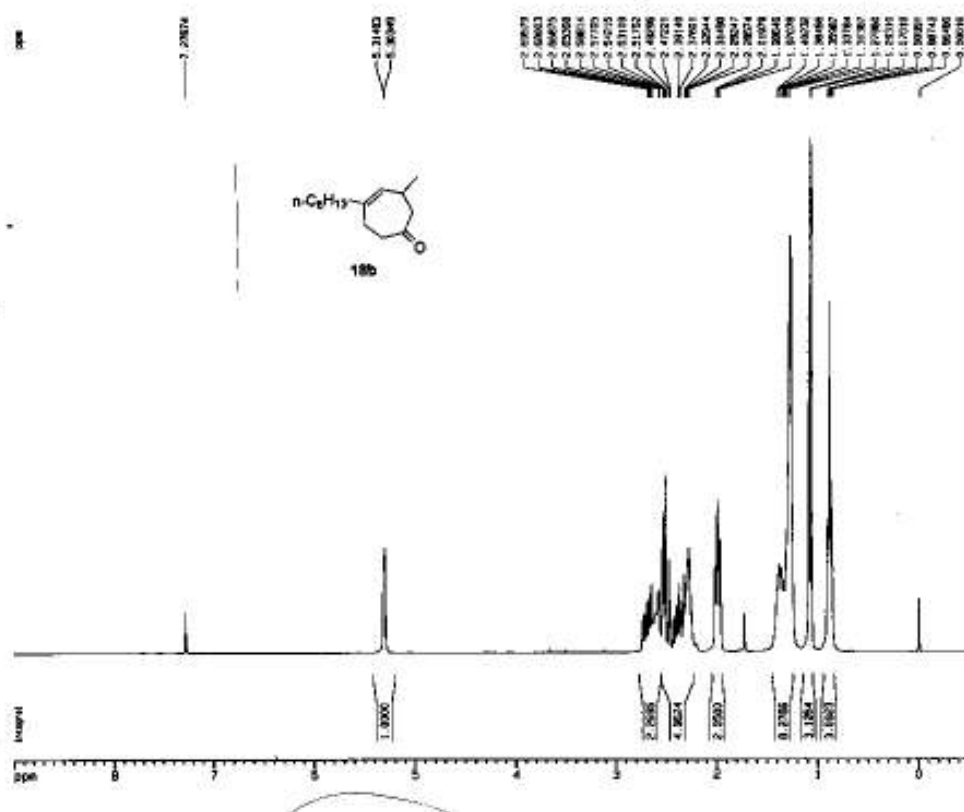
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F2 -100.000 ppm  
F3 -100.00 Hz  
RGWD 11.3000000 Hz/cm  
KDCN 807.50771 Hz/cm







88-bis-cis methyl CDC13 30 10 07



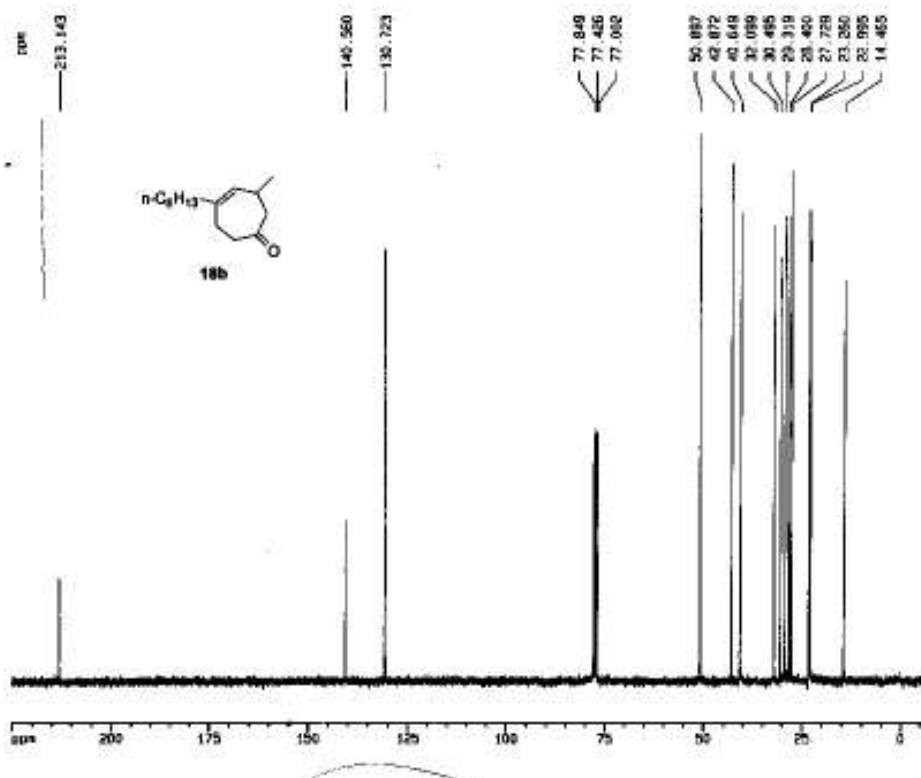
Current Data Parameters  
NAME: 88-bis-cis methyl  
EXPNO: 1  
PROCNO: 1

F2 - Acquisition Parameters  
Date\_: 20060205  
Time: 9.21  
INSTRUM: spect  
PROBHD: 5 mm QNP 1H  
PULPROG: zgpg30  
TD: 16384  
SOLVENT: CDCl3  
NS: 32  
DS: 4  
SWH: 4781.955 Hz  
FIDRES: 0.25864 Hz  
AQ: 1.7202701 sec  
RG: 66.5  
DM: 105.000 usec  
DE: 4.50 usec  
TE: 300.2 K  
D1: 1.0000000 sec  
P1: 15.00 usec  
DE: 4.50 usec  
SFO1: 300.131365 MHz  
NUC1: 1H  
PL1: -6.00 dB

F2 - Processing parameters  
SI: 32768  
SF: 300.130011 MHz  
WDW: EM  
SSB: 0  
LB: 0.20 Hz  
GB: 0  
PC: 1.50

3D NMR plot parameters  
CX: 20.00 cm  
FID: 0.000 dB  
F1: 2754.17 Hz  
F2: -6.500 dB  
F3: 150.37 Hz  
PRGMR: 0.42182 sec/cx  
RGCM: 128.00156 Hz/cx

88-bis-cis methyl C13 30 10 07

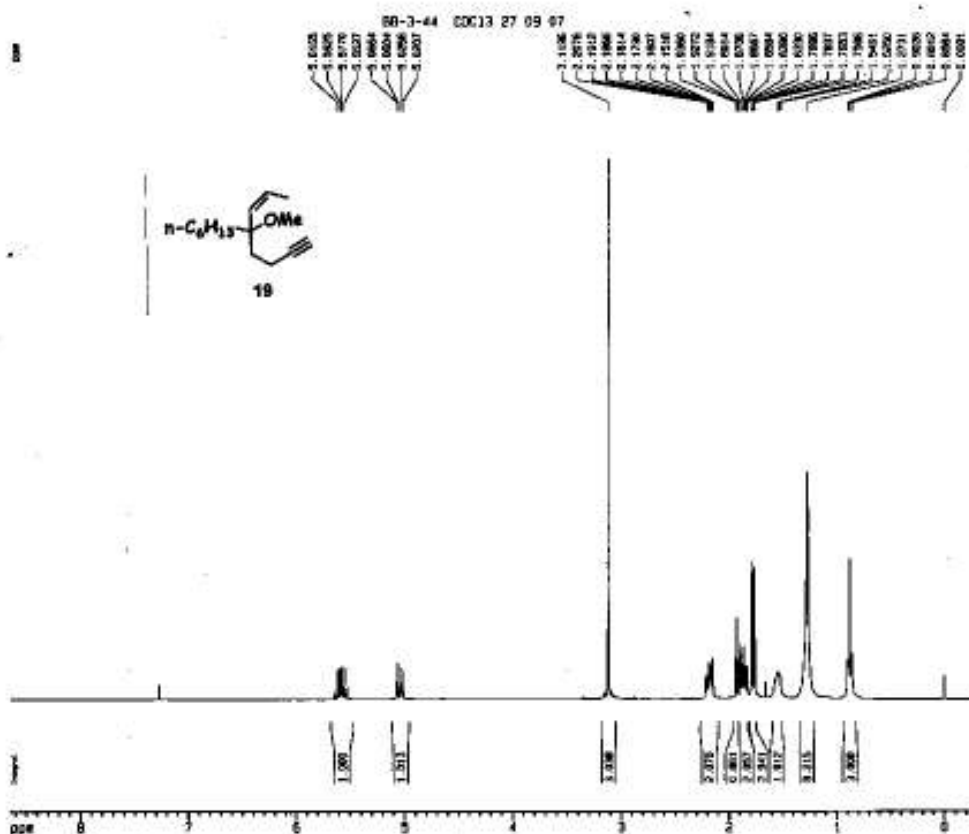


Current Data Parameters  
NAME: 88-bis-cis methyl  
EXPNO: 2  
PROCNO: 1

F2 - Acquisition Parameters  
Date\_: 20060205  
Time: 9.41  
INSTRUM: spect  
PROBHD: 5 mm QNP 1H  
PULPROG: zgpg30  
TD: 32768  
SOLVENT: CDCl3  
NS: 1000  
DS: 2  
SWH: 10607.844 Hz  
FIDRES: 0.500364 Hz  
AQ: 0.8250240 sec  
RG: 2040  
DM: 105.000 usec  
DE: 4.50 usec  
TE: 300.2 K  
SFO1: 101.6261200 MHz  
NUC1: 13C  
PL1: 0.00 dB  
PL12: 19.00 dB  
D1: 1.0000000 sec  
CPDPRG2: waltz16  
PULPROG: zgpg30  
SFO2: 100.6261200 MHz  
SFO3: 300.1300110 MHz  
NUC2: 1H  
PL2: -6.00 dB  
PL12: 19.00 dB  
P1: 7.50 usec  
DE: 4.50 usec  
SFO1: 75.4702900 MHz  
NUC1: 13C  
PL1: -6.00 dB  
D15: 0.0000000 sec

F2 - Processing parameters  
SI: 32768  
SF: 75.467180 MHz  
WDW: EM  
SSB: 0  
LB: 1.20 Hz  
GB: 0  
PC: 1.40

3D NMR plot parameters  
CX: 20.00 cm  
FID: 205.172 dB  
F1: 10062.37 Hz  
F2: -7.500 dB  
F3: -531.84 Hz  
PRGMR: 11.51060 sec/cx  
RGCM: 675.30548 Hz/cx

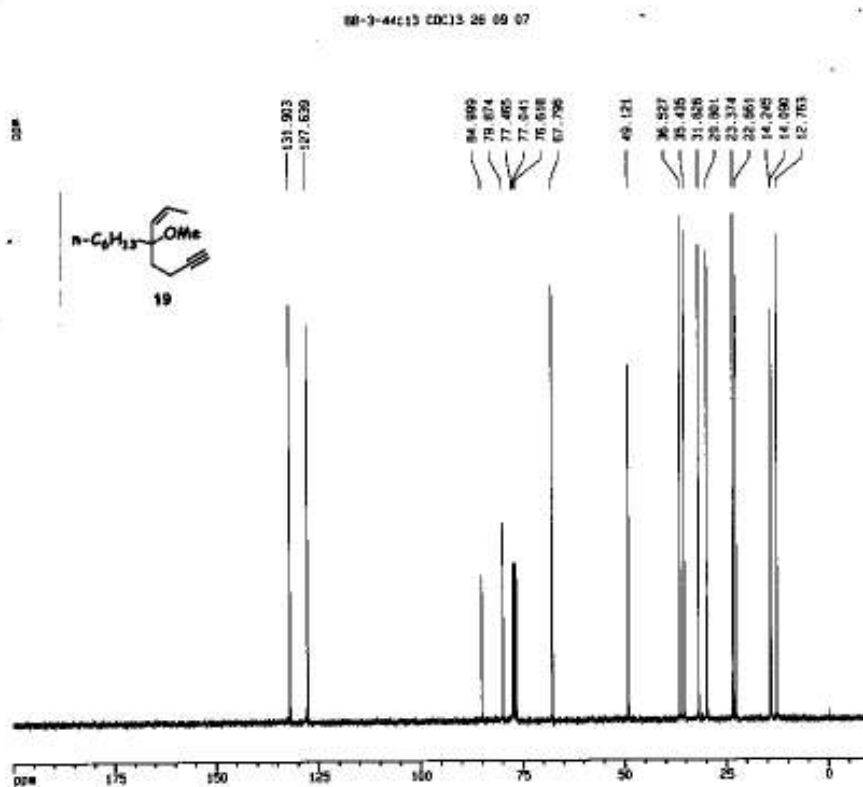


Current Data Parameters  
NAME 50-3-44  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 500000  
Time 17.02  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 32768  
SOLVENT C0C13  
NS 63  
DS 2  
SWH 5172.858 Hz  
FIDRES 0.283300 Hz  
AQ 2.6542000 sec  
RG 64  
DM 80.200 uHz  
DE 4.50 uHz  
TE 300.2 K  
D0 1.00000000 sec  
P1 5.80 uHz  
DE 4.50 uHz  
SFO1 300.136024 MHz  
MAG1 51  
PL1 1.00 dB

F2 - Processing parameters  
SI 16384  
SF 300.130024 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 21.00 cm  
FID 8.548 uHz  
F1 2555.06 Hz  
FAP -0.273 uHz  
F2 -87.05 Hz  
FREQH 0.42475 uHz/sec  
AQCH 127.4555 Hz/sec



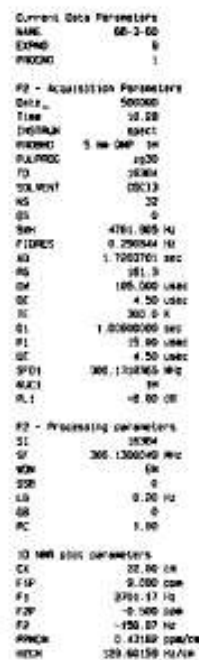
Current Data Parameters  
NAME 50-3-44:13  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 500000  
Time 20.23  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 32768  
SOLVENT C0C13  
NS 1081  
DS 2  
SWH 19857.844 Hz  
FIDRES 0.186384 Hz  
AQ 0.0060348 sec  
RG 16384  
DM 25.500 uHz  
DE 4.50 uHz  
TE 300.2 K  
D12 0.00000000 sec  
PL12 10.00 dB  
D1 1.00000000 sec  
SFO1 101.626115 MHz  
SFO2 100.626115 MHz  
SFO3 300.136024 MHz  
MAG1 51  
PL1 -6.00 dB  
PL2 13.00 dB  
PL12 1.50 uHz  
DE 4.50 uHz  
SFO1 101.626115 MHz  
MAG1 13C  
PL1 -6.00 dB  
PL12 0.00000000 sec

F2 - Processing parameters  
SI 32768  
SF 25.4627460 MHz  
WDW EM  
SSB 0  
LB 1.30 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
FID 795.000 uHz  
F1 19693.00 Hz  
F2 -10.000 uHz  
F3 754.88 Hz  
FREQH 19.50000 uHz/sec  
AQCH 752.41132 Hz/sec

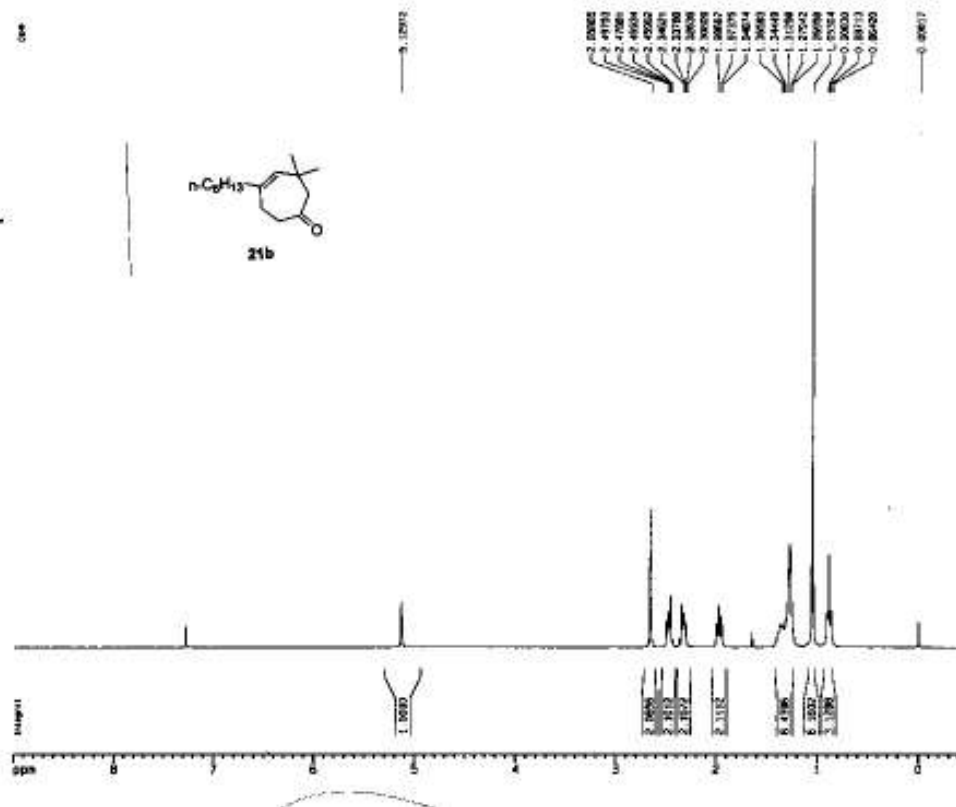
35



605

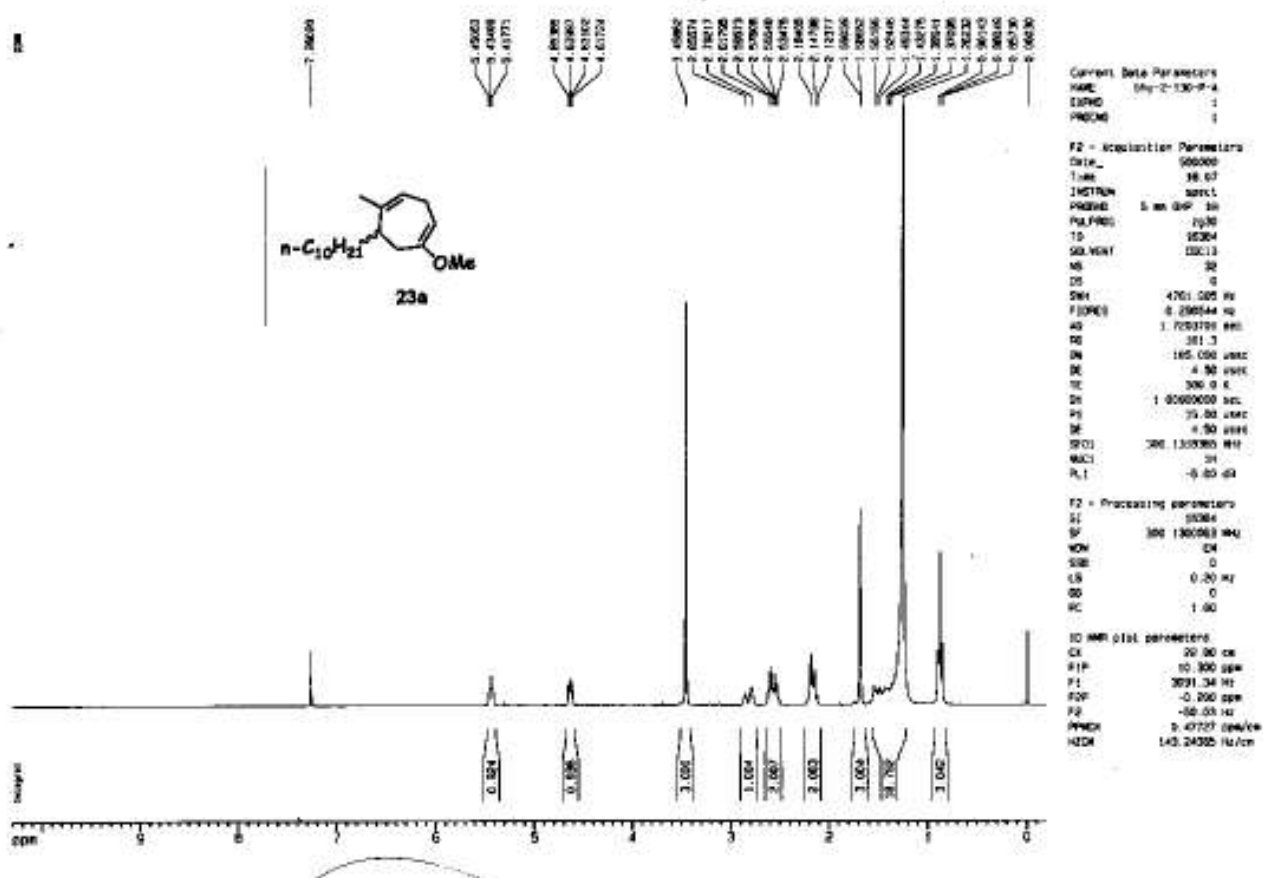


BS-3-BHJ01eet CDC13 29 10 07

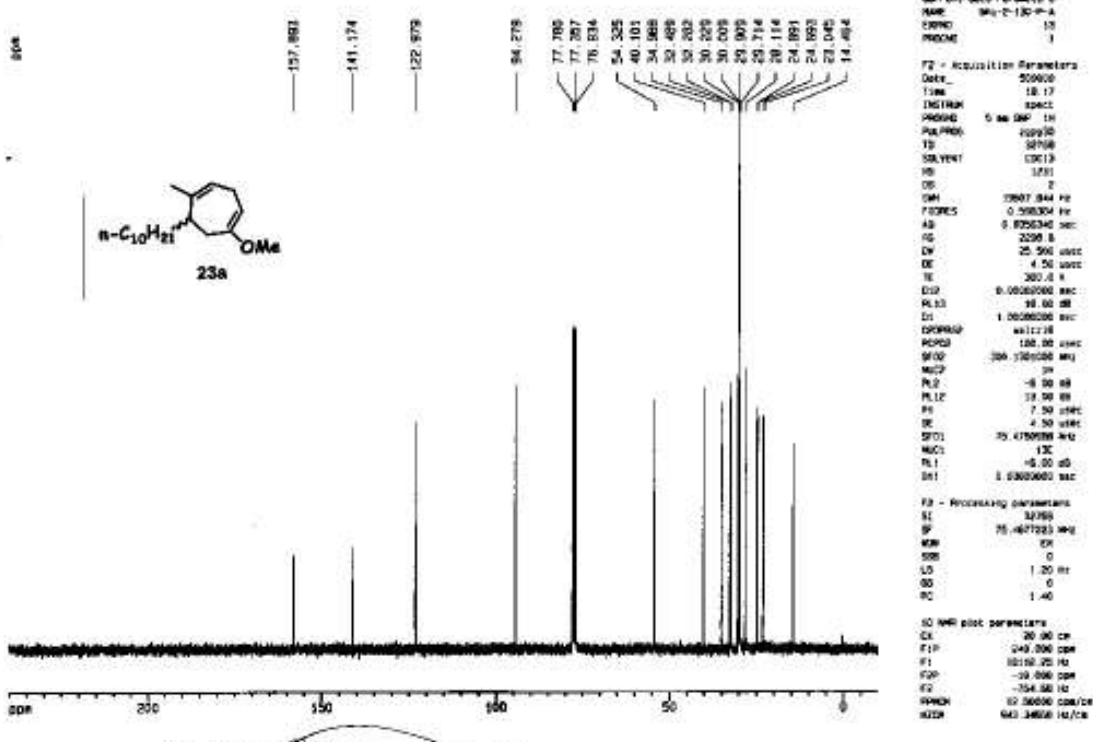




bnj-2-130-P-A 1 1 v yhr 20071007

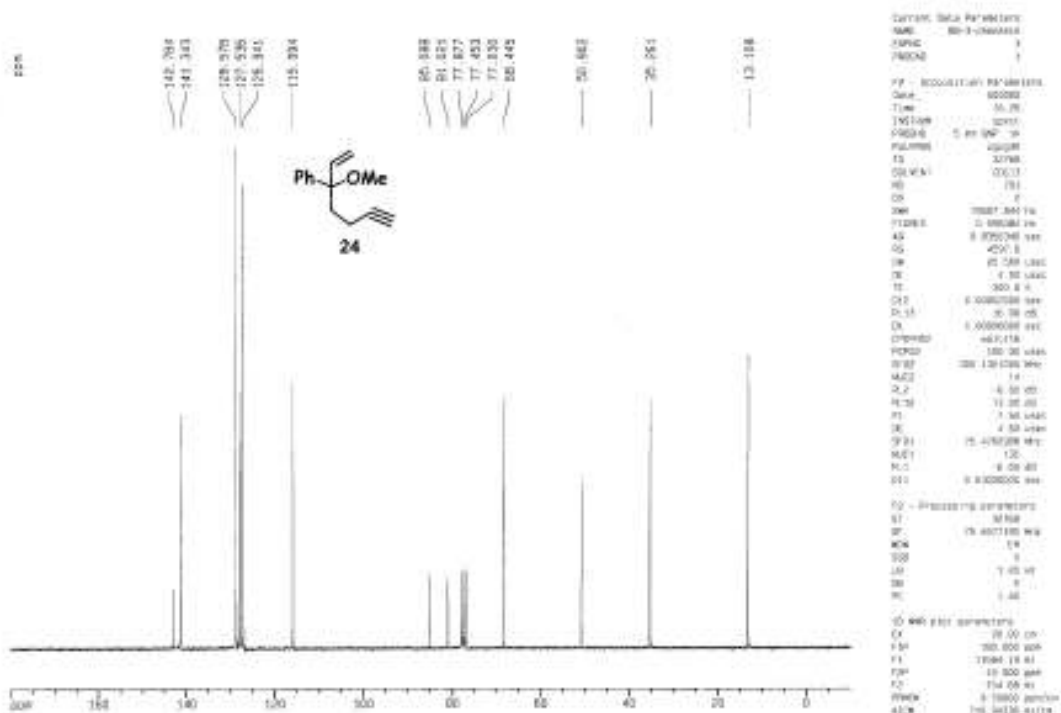


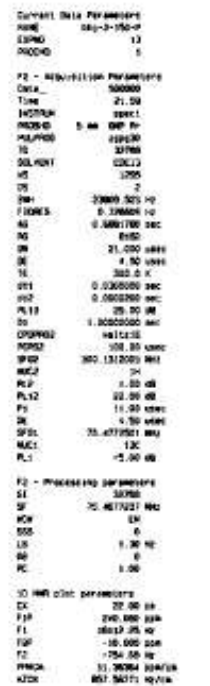
bnj-2-130-P-A 13 1 v yhr 20071007



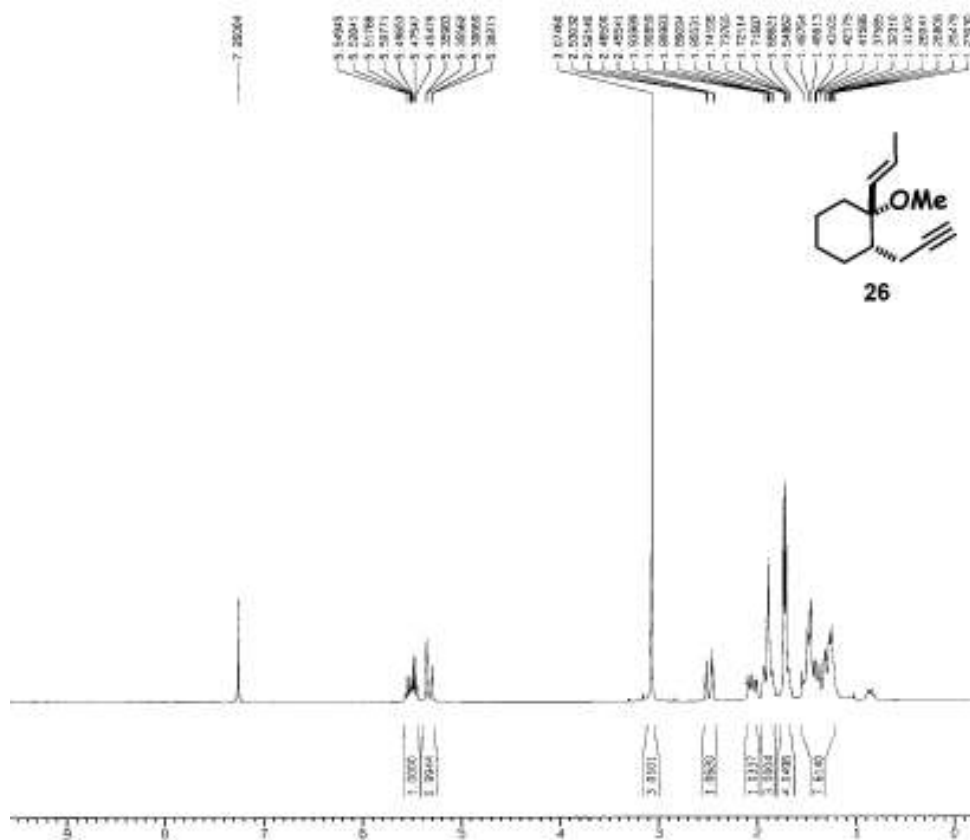








01j-2-162-P 2 1 v1 ynr 20071127



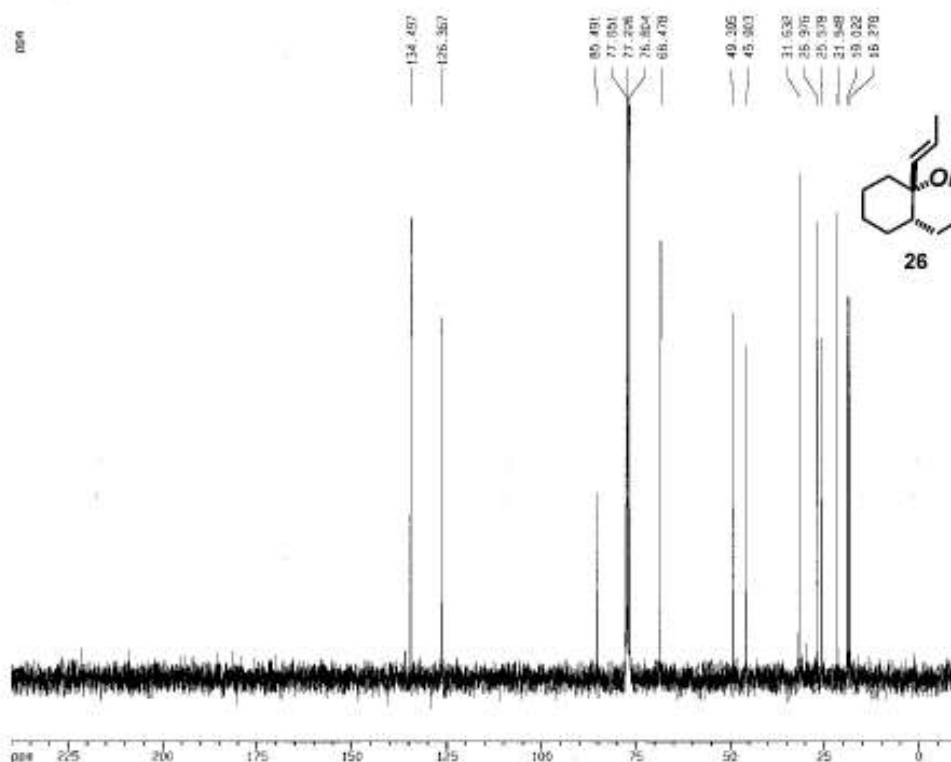
Current Data Parameters  
NAME 01j-2-162-P  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 200603  
Time 1.49  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 32  
DS 0  
SWH 4181.300 Hz  
FIDRES 0.290044 Hz  
AQ 1.7693791 sec  
RG 327.5  
DM 185.000 sec  
DE 8.50 sec  
TE 303.2 K  
D1 1.0000000 sec  
D11 15.00 sec  
DE 4.50 sec  
SFO1 300.1318000 MHz  
NUC1 1H  
PL1 -6.00 dB

F2 - Processing parameters  
SI 10394  
SF 300.1308852 MHz  
WDW EM  
SSB 0  
LB 0.20 Hz  
GB 0  
PC 1.00

3D NMR data parameters  
CX 22.00 cm  
F1F 10.300 cm  
F1 3001.34 Hz  
F2F -0.200 cm  
F2 18.03 Hz  
PRGM 0.47777 sec/ch  
AQDN 143.24385 sec/ch

01j-2-162-P 13 1 v ynr 20071126

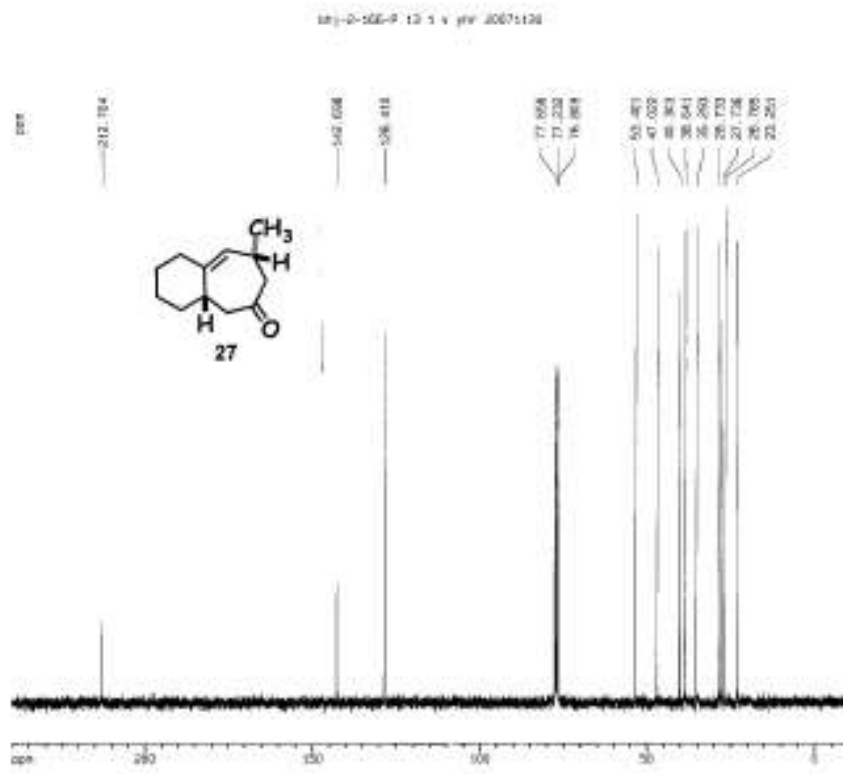
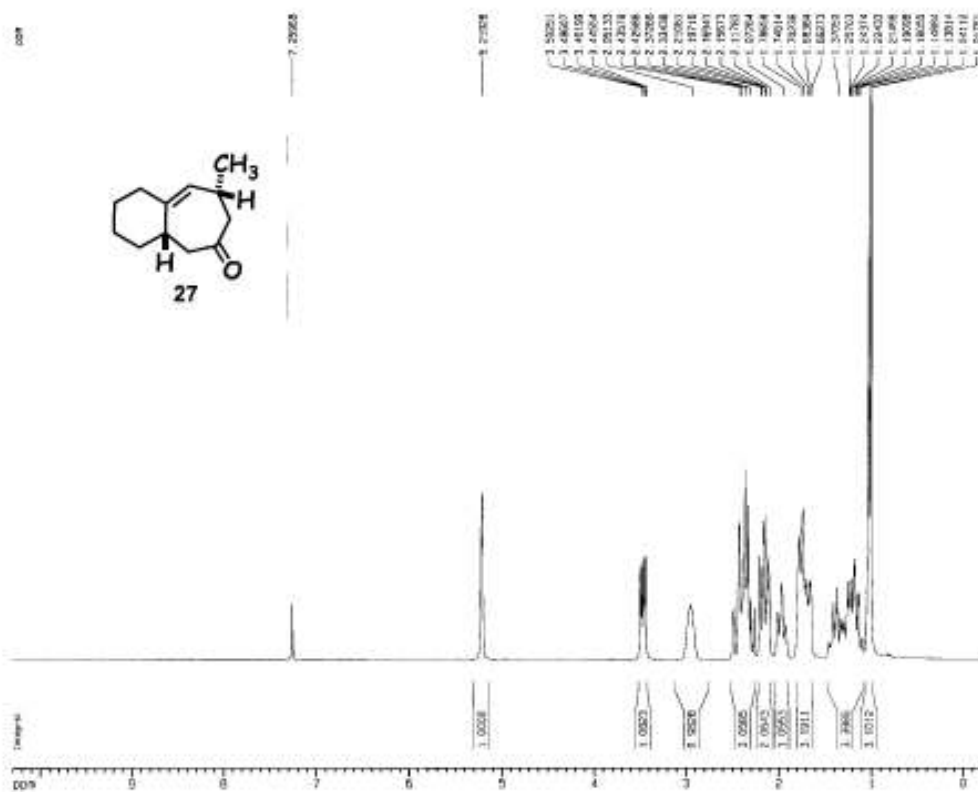


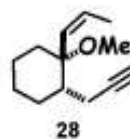
Current Data Parameters  
NAME 01j-2-162-P  
EXPNO 13  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 200603  
Time 21.53  
INSTRUM spect  
PROBHD 5 mm QNP 1H  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 20808.300 Hz  
FIDRES 0.196603 Hz  
AQ 0.8881708 sec  
RG 6592  
DM 21.300 sec  
DE 8.50 sec  
TE 303.2 K  
D1 0.3300000 sec  
D11 0.3300000 sec  
D12 0.3300000 sec  
D13 0.3300000 sec  
SFO1 100.626115 MHz  
SFO2 75.4772501 MHz  
NUC1 13C  
PL1 -6.00 dB

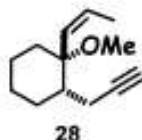
F2 - Processing parameters  
SI 32768  
SF 75.4671227 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00

3D NMR data parameters  
CX 22.00 cm  
F1F 10.300 cm  
F1 3001.34 Hz  
F2F -0.200 cm  
F2 18.03 Hz  
PRGM 0.47777 sec/ch  
AQDN 143.24385 sec/ch





TS	MSR	BCR	off-diagonal
T8		21.30	
T10		30.300	
F5		1091.38	
T20		-0.200	
F2		60.03	
PRAC		0.50000	
HTM		150.0000	



10 MHz plot parameters	
EX	20.00 cm
F1F	240.000 ppm
F1	18110.20 Hz
F2F	-10.000 ppm
F2	-150.00 Hz
PRCH	10.00000 ppm/Hz
WID	857.00000 Hz/cm

