

## Supporting Information

### Synthesis of the Pyridine Core of Cyclothiazomycin

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**General methods.** Unless otherwise stated, reactions were performed under nitrogen using flame-dried glassware. Some solvents used in reactions were dried by different ways. CH<sub>2</sub>Cl<sub>2</sub>, THF and diethyl ether were dried by MB SPS Compact solvent purification system. Toluene and xylenes were distilled from sodium metal/benzophenoneketyl. 1,2-Dichloroethane, CH<sub>3</sub>CN and pyridine were distilled from CaH<sub>2</sub>. DMF and acetone were distilled from CaCO<sub>3</sub>. EtOH was distilled over Mg/I<sub>2</sub>. All other reagent quality solvents were used without further purification. The microwave used is a CEM Discover microwave synthesizer. All spectra were recorded in CDCl<sub>3</sub> or CD<sub>3</sub>OD, and chemical shifts are reported relative to CDCl<sub>3</sub> (7.24 ppm for <sup>1</sup>H NMR) or CDCl<sub>3</sub> (77.23 ppm for <sup>13</sup>C NMR), CD<sub>3</sub>OD (3.30 ppm for <sup>1</sup>H NMR) or CD<sub>3</sub>OD (49.15 ppm for <sup>13</sup>C NMR). Chiral compounds were analyzed using a Jasco P-1010 polarimeter, using the sodium D line at the indicated temperature, and are given in deg cm<sup>3</sup> g<sup>-1</sup> dm<sup>-1</sup> for and 10<sup>-2</sup>g cm<sup>-3</sup> for c.

**Ethyl 2-cyanothiazole-4-carboxylate (8).** Ethyl 2-bromothiazole-4-carboxylate (**16**) (900.0 mg, 3.81 mmol) was dissolved in 2.5 mL of dry pyridine in a pressure tube. CuCN(684 mg, 7.62 mmol) was added. The tube was sealed and heated at 150 °C for 2 h. Cooled to rt, 10 mL of saturated NH<sub>4</sub>Cl was added and the mixture was extracted with EtOAc (4 x 20 mL). The combined organic layers were washed with brine (5 mL), dried over anhydrous MgSO<sub>4</sub>, filtered and the filtrate was concentrated. The product was purified by column chromatography SiO<sub>2</sub>(eluted with hexanes/ethyl acetate = 2:1) delivering 412.0 mg (59% yield) of **8** as white solid. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 8.45 (s, 1H), 4.47 (q, *J* = 7.2 Hz, 2H), 1.42 (t, *J* = 7.2 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 160.0, 149.5, 137.9, 112.0, 62.6, 14.5; MS calcd for [M + Na] C<sub>7</sub>H<sub>6</sub>N<sub>2</sub>NaO<sub>2</sub>S 205.0048, found 205.0113.

**(R)-4-[N-(tert-butoxycarbonyl)amino]-2-pentyn-1-ol (23).** The dibromoalkene **9** (200.0 mg, 0.61 mmol) was dissolved in THF (1.2 mL) under argon and cooled to -78 °C. *n*-BuLi (0.96 mL, 1.82 mmol, 2.45 M in hexanes) was added dropwise and stirred for 20 min. The solution of DMF (94 µl, 1.22 mmol) in 0.24 mL of THF was added dropwise. The temperature was warmed to 0 °C and stirred for 1 h. Saturated citric acid solution (0.35 mL) was added and warmed to room temperature and stirred for 10 min. Saturated NH<sub>4</sub>Cl solution (2 mL) was then added and extracted with ether (3 x 2 ml). The combined organic layers were washed with brine (2 mL), dried over anhydrous MgSO<sub>4</sub>, filtered and the filtrate was concentrated under high vacuum. The resulting solid was dissolved in EtOH (0.7 mL) and cooled to 0 °C. NaBH<sub>4</sub> (23.0 mg, 0.61 mmol) was added and the reaction mixture was stirred at 0 °C for 50 min. Saturated citric acid solution (1 mL) was added, stirred at 0 °C for 5 min. The reaction mixture was extracted with ether (3 x 2 mL). The combined organic layers were washed with brine (2 mL), dried over anhydrous MgSO<sub>4</sub>, filtered and the filtrate was concentrated under vacuum. The product was purified by column chromatography on SiO<sub>2</sub> (eluted with hexanes/EtOAc = 3:1, 2:1), delivering 60.6 mg (50% yield) of **23** as colorless liquid. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 4.75 (br, 1H), 4.49 (br, 1H), 4.23 (s, 2H), 2.23 (br, 1H), 1.42 (s, 9H), 1.35 (d, *J* = 6.9 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 155.0, 86.4, 80.5, 51.2, 38.6, 28.6, 22.8; MS calcd for [M + Na] C<sub>10</sub>H<sub>17</sub>NNaO<sub>3</sub> 222.1106, found 222.1171. [α]<sub>D</sub><sup>20</sup> +76.9 (c 1.00, CHCl<sub>3</sub>).

**4-(N-Benzyl-N-tosylamino)but-2-yn-1-ol (13).** TsBnNH (50.0 mg, 0.18 mmol) and 4-hydroxybut-2-ynyl methanesulfonate (**12**) (36.3 mg, 0.22 mmol) were dissolved in dry CH<sub>3</sub>CN (3 mL). To this stirring solution was added K<sub>2</sub>CO<sub>3</sub> (31.0 mg, 0.22 mmol). The reaction mixture was reflux overnight, cooled to rt, filtered and the filtrate was concentrated under vacuum. The product was purified by column chromatography on SiO<sub>2</sub> (eluted with hexanes/EtOAc = 1:1), delivering 52.0 mg (86% yield) of **13** as colorless liquid. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 8.4 Hz, 2H), 7.33-7.24 (m, 7H), 4.31 (s, 2H), 3.94 (br, 2H), 3.91 (s, 2H), 2.42 (s, 2H), 1.66 (br, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 143.9, 136.1, 135.1, 129.6, 128.9, 128.8, 128.3, 128.1, 84.4, 78.3, 50.8, 50.3, 36.1, 21.7; MS calcd for [M + Na] C<sub>18</sub>H<sub>19</sub>NNaO<sub>3</sub>S 352.0983, found 352.1.

**General procedure for diyne synthesis.** To the solution of the alcohol (1 equiv) in DCM (0.23 M) were added TEA (1 equiv) and DMAP (0.1 equiv), followed by the solution of freshly prepared alkynylsilyl bromide (**15**) (1 equiv) in DCM. The resulting mixture was stirred under N<sub>2</sub> at room temperature overnight, concentrated under vacuum. The product was purified by column chromatography on SiO<sub>2</sub> (eluted with hexanes/EtOAc mixture), delivering **7**, **10** and **11**.

**{{(R)-4-[(N-tert-Butoxycarbonyl)amino]pent-2-ynyloxy}(ethynyl)diisopropylsilane (7).** Eluted with hexane/EtOAc = 10:1. 284.2 mg (84% yield) of **7** as colorless liquid was obtained from 200.0 mg of alcohol **23**. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 4.69 (br, 1H), 4.49 (br, 1H), 4.41 (s, 2H), 2.45 (d, *J* = 0.6 Hz, 1H), 1.42 (s, 9H), 1.35 (d, *J* = 6.9 Hz, 3H), 1.20-1.00 (m, 14H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 154.8, 95.8, 85.8, 84.0, 80.3, 53.1, 38.7, 28.6, 22.9, 17.1, 17.0, 13.0; MS calcd for [M + Na] C<sub>18</sub>H<sub>31</sub>NNaO<sub>3</sub>Si 360.1971, found 360.2060. [α]<sub>D</sub><sup>20</sup> +53.54 (c 0.54, CHCl<sub>3</sub>).

**(4-*N,N*-Diethylamino-but-2-ynyl)oxy)(ethynyl)diisopropylsilane (10).** Eluted with hexanes/EtOAc = 1:1, 1:2. 65.5 mg (70% yield) of **10** was obtained as colorless liquid from 54.4 mg of **14**. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 4.44 (t, *J* = 1.6 Hz, 2H), 3.42 (t, *J* = 1.6 Hz, 2H), 2.52 (q, *J* = 6.8 Hz, 4H), 2.43 (s, 1H), 1.04-1.00 (m, 20H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 95.6, 84.0, 83.0, 80.0, 53.2, 47.4, 40.9, 18.0, 17.0, 16.9, 13.3, 13.0, 12.8; MS calcd for [M + H] C<sub>16</sub>H<sub>30</sub>NOSi 280.2097, found 280.2.

**[4-(*N*-Benzyl-*N*-tosylamino)-but-2-ynyl)oxy](ethynyl)diisopropylsilane (11).** Eluted with hexanes/EtOAc = 3:1. 105.0 mg (81% yield) of **11** as white solid was obtained from 90.7 mg of **13**. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 7.6 Hz, 1H), 7.35-7.24 (m, 7H), 4.31 (s, 2H), 4.14 (s, 2H), 3.95 (s, 2H), 2.44 (s, 1H), 2.43 (s, 3H), 1.09-0.94 (m, 14H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 143.6, 136.3, 135.2, 129.6, 129.0, 128.8, 128.3, 128.1, 95.8, 84.3, 83.9, 77.6, 52.8, 50.1, 36.2, 21.7, 17.3, 17.1, 13.0; MS calcd for [M + H] C<sub>26</sub>H<sub>34</sub>NO<sub>3</sub>SSi 468.2029, found 468.0.

**General procedure for cyclotrimerization reactions using CpCo(CO)<sub>2</sub>.** To the solution of diyne (1equiv) and nitrile (10 equiv) in dry xylenes (0.11 M for diyne) in a vial was added CpCo(CO)<sub>2</sub> (0.2 equiv). The vial was sealed and the reaction was heated to 150 °C overnight. After cooling to room temperature, the reaction mixture was concentrated under vacuum. The product was purified by column chromatography on SiO<sub>2</sub> (eluted with EtOAc, EtOAc:MeOH = 95:5), delivering **18**.

**4-(*N,N*-Diethylamino)-1,3-dihydro-1,1-diisopropyl-6-[4-(ethoxycarbonyl)thiazol-2-yl]-[1,2]oxasilolo[4,3-*c*]pyridine (18).** 17.9 mg (39% yield) of **18** as yellow solid was obtained from 33.2 mg of **10**. <sup>1</sup>H NMR (400 MHz, CD<sub>3</sub>OD) δ 8.01 (d, *J* = 8.0 Hz, 2H), 7.89 (s, 1H), 7.50-7.40 (m, 3H), 5.33 (s, 2H), 3.78 (s, 2H), 2.64 (q, *J* = 6.8 Hz, 4H), 1.34-1.24 (m, 2H), 1.08-1.01 (m, 18H); <sup>13</sup>C NMR (100 MHz, CD<sub>3</sub>OD) δ 159.1, 145.5, 145.1, 140.9, 130.0, 129.9, 129.7, 128.4, 123.4, 72.3, 59.0, 48.6, 17.4, 17.2, 14.1, 12.1; MS calcd for [M + Na] C<sub>23</sub>H<sub>35</sub>N<sub>2</sub>OSi 383.2519, found 383.2.

**General procedure for cyclotrimerization reactions using Cp\*RuCl(COD) in DCM.** The diyne (1 equiv) and nitrile (3 equiv) were dissolved in DCM and Cp\*RuCl(COD) (0.05 equiv) was added. The resulting mixture was stirred at room temperature overnight, and concentrated under vacuum. The product was purified by column chromatography on SiO<sub>2</sub> (eluted with hexanes/ EtOAc mixture), delivering **21** and **22**.

**Diethyl 5-(4-(ethoxycarbonyl)thiazol-2-yl)-1H-indene-2,2(3H)-dicarboxylate (21).** Eluted with hexanes/EtOAc = 2:1, 1:1. 22.9 mg (93% yield) of **21** as a white solid was obtained from 15.6 mg of **17**. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.40 (s, 1H), 8.19 (s, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 4.20 (q, *J* = 7.2 Hz, 4H), 3.62 (d, *J* = 3.2 Hz, 4H), 1.42 (t, *J* = 7.2 Hz, 3H), 1.23 (t, *J* = 7.2 Hz, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 171.0, 170.2, 151.43, 149.4, 148.2, 145.3, 138.5, 129.4, 116.5, 62.3, 61.7, 60.4, 40.2, 38.4, 14.6, 14.2; MS calcd for [M + H] C<sub>20</sub>H<sub>23</sub>N<sub>2</sub>O<sub>6</sub>S 419.1277, found 419.

**4-(*N*-Benzyl-*N*-tosylethanamine)-1,3-dihydro-1,1-diisopropyl-6-[4-(ethoxycarbonyl)thiazol-2-yl]-[1,2]oxasilolo[4,3-*c*]pyridine (22).** Eluted with hexanes/EtOAc = 4:1, 2:1. 10.9 mg (78% yield) of **22** as a white solid was obtained from 10.0 mg of **11**. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.22 (s, 1H), 8.18 (s, 1H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.21-7.20 (m, 2H), 7.19-7.13 (m, 3H), 7.07 (d, *J* = 8.4 Hz, 2H), 5.10 (s, 2H), 4.65 (s, 2H), 4.44 (q, *J* = 6.7 Hz, 2H), 4.33 (s, 2H), 2.25 (s, 3H), 1.43 (t, *J* = 6.7 Hz, 3H), 1.23-1.17 (m, 2H), 0.97-0.94 (m, 12H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.5, 161.7, 149.2, 148.4, 147.5, 146.6, 145.4, 143.5, 136.0, 129.7, 129.3, 128.9, 128.7, 128.0, 127.3, 122.0, 70.6, 61.9, 53.1, 49.7, 21.7, 17.05, 17.02, 14.6, 12.9; MS calcd for [M + H] C<sub>33</sub>H<sub>40</sub>N<sub>3</sub>O<sub>5</sub>S<sub>2</sub>Si 650.2179, found 650.0.

**General procedure for cyclotrimerization reactions using Cp\*RuCl(COD) in DCE.** The solution of diyne (1equiv) in dry degassed DCE (0.25 M for diyne) was added dropwise to the solution of nitrile (3 equiv) and Cp\*RuCl(COD) (5 mol%) in dry degassed DCE (0.25M for nitrile). The reaction mixture was stirred at room temperature overnight and concentrated under vacuum. The product was purified by column chromatography on SiO<sub>2</sub> (eluted with hexanes/EtOAc mixture), delivering **21** and **6**.

**(*R*)-2-[1-(*N*-*tert*-Butoxycarbonyl)aminoethyl]-1,3-dihydro-1,1-diisopropyl-6-[4-(ethoxycarbonyl)thiazol-2-yl]-[1,2]oxasilolo[4,3-*c*]pyridine (6).** Eluted with hexanes/EtOAc = 5:1, 2:1. 24.4 mg (82% yield) of **6** as white solid was obtained from 19.2 mg of **7**. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 8.34 (s, 1H), 8.21 (s, 1H), 5.74 (d, *J* = 8.4 Hz, 1H), 5.26 (d, *J* = 15.3 Hz, 1H), 5.12 (d, *J* = 15.3 Hz, 1H), 4.76 (pentet, *J* = 7.5 Hz, 1H), 4.42 (q, *J* = 7.2 Hz, 2H), 1.53-1.21 (m, 15H), 1.19-1.17 (m, 2H), 0.99-0.93 (m, 12H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.5, 161.7, 155.3, 154.7, 148.3, 147.7, 145.7, 144.1, 129.4, 121.7, 79.7, 70.5 61.8, 48.0, 28.6, 21.9, 16.94, 16.92, 12.9, 12.8; MS calcd for [M + Na] C<sub>25</sub>H<sub>37</sub>N<sub>3</sub>NaO<sub>5</sub>SSi 542.2121, found 542.2231. [α]<sub>D</sub><sup>20</sup> +106.1 (c 0.98, CHCl<sub>3</sub>).

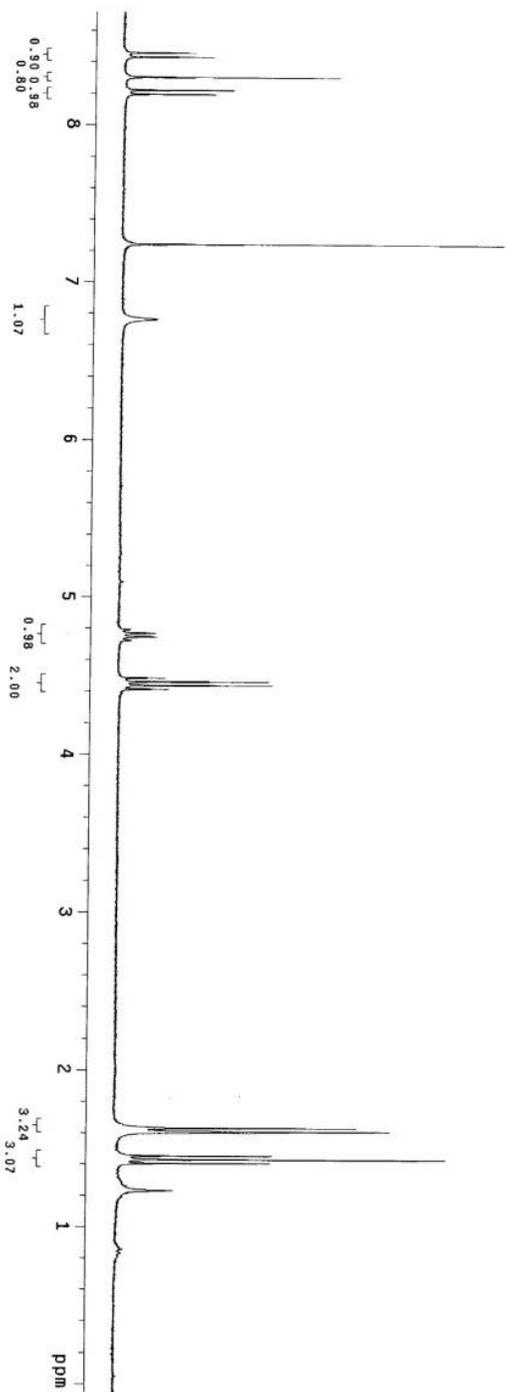
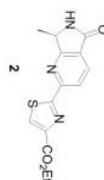
**(*R*)-2-[1-(*N*-*tert*-Butoxycarbonyl)aminoethyl]-3-(hydromethyl)-6-[4-(ethoxycarbonyl)thiazol-2-yl] pyridine (5).** To a solution of **6** (110 mg, 0.21 mmol) in 5.1 mL of dry THF was added TBAF (1M solution in THF, 0.64 mL, 0.64 mmol) dropwise. The reaction mixture was stirred at room temperature until TLC showed completion of the reaction. The mixture was concentrated under vacuum and the product was purified by column chromatography on SiO<sub>2</sub> (eluted with hexanes/EtOAc= 1:2), delivering 83.5 mg (97% yield) of **5** as light yellow solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.21 (s, 1H), 8.15 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 5.54 (d, *J* = 8.8 Hz, 1H), 5.11 (m, 1H), 4.92 (d, *J* = 12.8 Hz, 1H), 4.62-4.57 (m, 1H), 4.44 (t, *J* = 7.2 Hz, 2H), 4.1 (br s, 1H), 1.47 (d, *J* = 6.0 Hz, 3H), 1.46 (t, *J* = 7.2 Hz, 3H); 1.36 (s, 9H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.1, 161.7, 159.5, 156.0, 149.4, 148.4, 138.5, 134.8, 129.5, 119.1, 80.4, 61.7, 61.4, 46.7, 28.5, 21.7, 14.5; MS calcd for [M + Na] C<sub>19</sub>H<sub>25</sub>N<sub>3</sub>NaO<sub>5</sub>S 430.1413, found 430.1513. [α]<sub>D</sub><sup>20</sup> +165.7 (c 0.62, CHCl<sub>3</sub>).

**(*R*)-2-[4-(Ethoxycarbonyl)thiazol-2-yl]-7-methyl-5-oxo-6,7-dihydro-5*H*-pyrrolo[3,4-*b*]pyridine (2).** To a solution of the alcohol **5** (51.7 mg, 0.13 mmol) in 1.1 mL of acetone was

added Jones' reagent (2.7 M aqueous solution, 104  $\mu$ M, 0.28 mmol) dropwise at 0 °C. The reaction mixture was stirred at room temperature for 1.5 h. 2 mL of H<sub>2</sub>O was added and the mixture was extracted with DCM (3 x 2 mL). The combined organic layers were washed with brine (2 mL), dried over anhydrous MgSO<sub>4</sub>, filtered and the filtrate was concentrated and dried under high vacuum to yield crude solid. The solid was dissolved in 2 mL of dry DCM. TFA (211  $\mu$ L, 27.6 mmol) was added and the reaction mixture was stirred at room temperature for 1 h until TLC show completion of reaction. Saturated NaHCO<sub>3</sub> (3 mL) was added and the mixture was extracted with DCM (3 x 3 mL). The combined organic layers were washed with brine (2 mL), dried over anhydrous MgSO<sub>4</sub>, filtered and the filtrate was concentrated under vacuum. The product was purified by column chromatography on SiO<sub>2</sub> (eluted with EtOAc), delivering 30.8 mg (80% yield) of **2** as white solid. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.45 (d, *J* = 8.1 Hz, 1H), 8.30 (s, 1H), 8.21 (d, *J* = 8.1 Hz, 1H), 6.76 (br s, 1H), 4.76 (q, *J* = 6.9 Hz, 1H), 4.47 (q, *J* = 7.2 Hz, 2H), 1.61 (d, *J* = 6.9 Hz, 3H), 1.43 (t, *J* = 7.2 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  168.9, 168.5, 168.2, 161.5, 153.5, 148.8, 133.3, 130.5, 120.2, 61.9, 54.4, 18.9, 14.5; MS calcd for [M + Na] C<sub>14</sub>H<sub>13</sub>N<sub>3</sub>NaO<sub>3</sub>S 326.0575, found 326.0658. [ $\alpha$ ]<sub>D</sub><sup>20</sup> +51.9 (*c* 0.54, CHCl<sub>3</sub>).

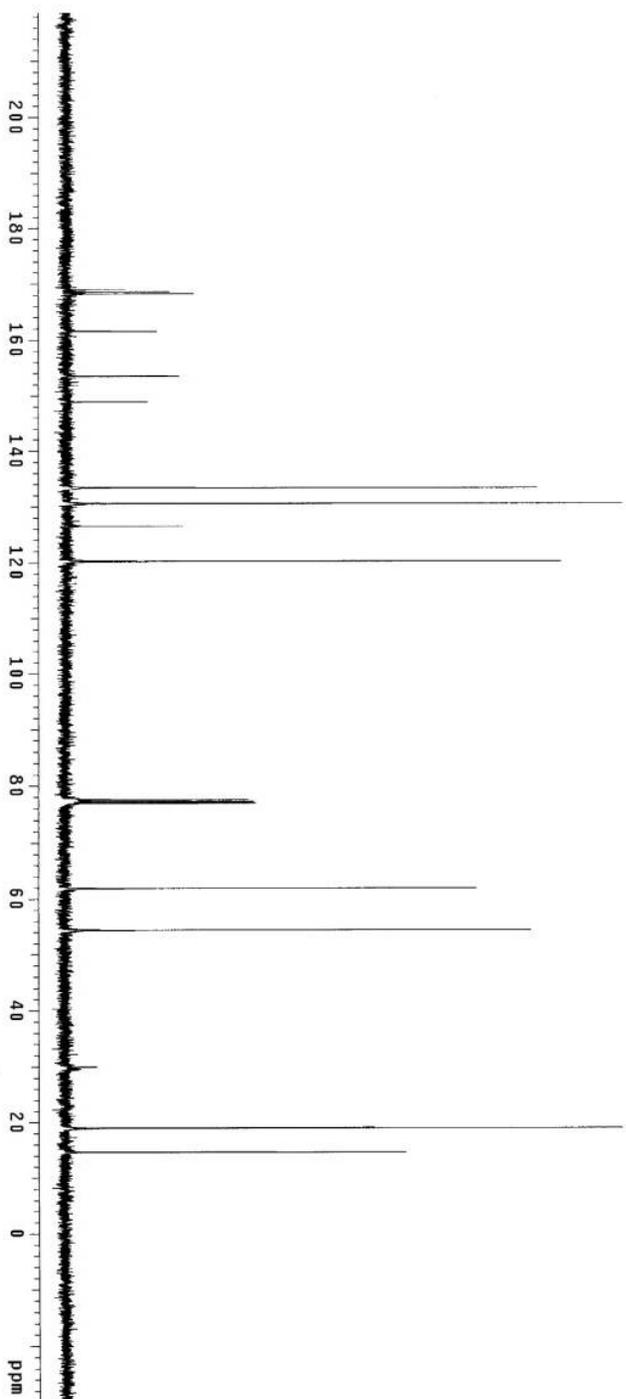
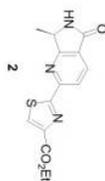
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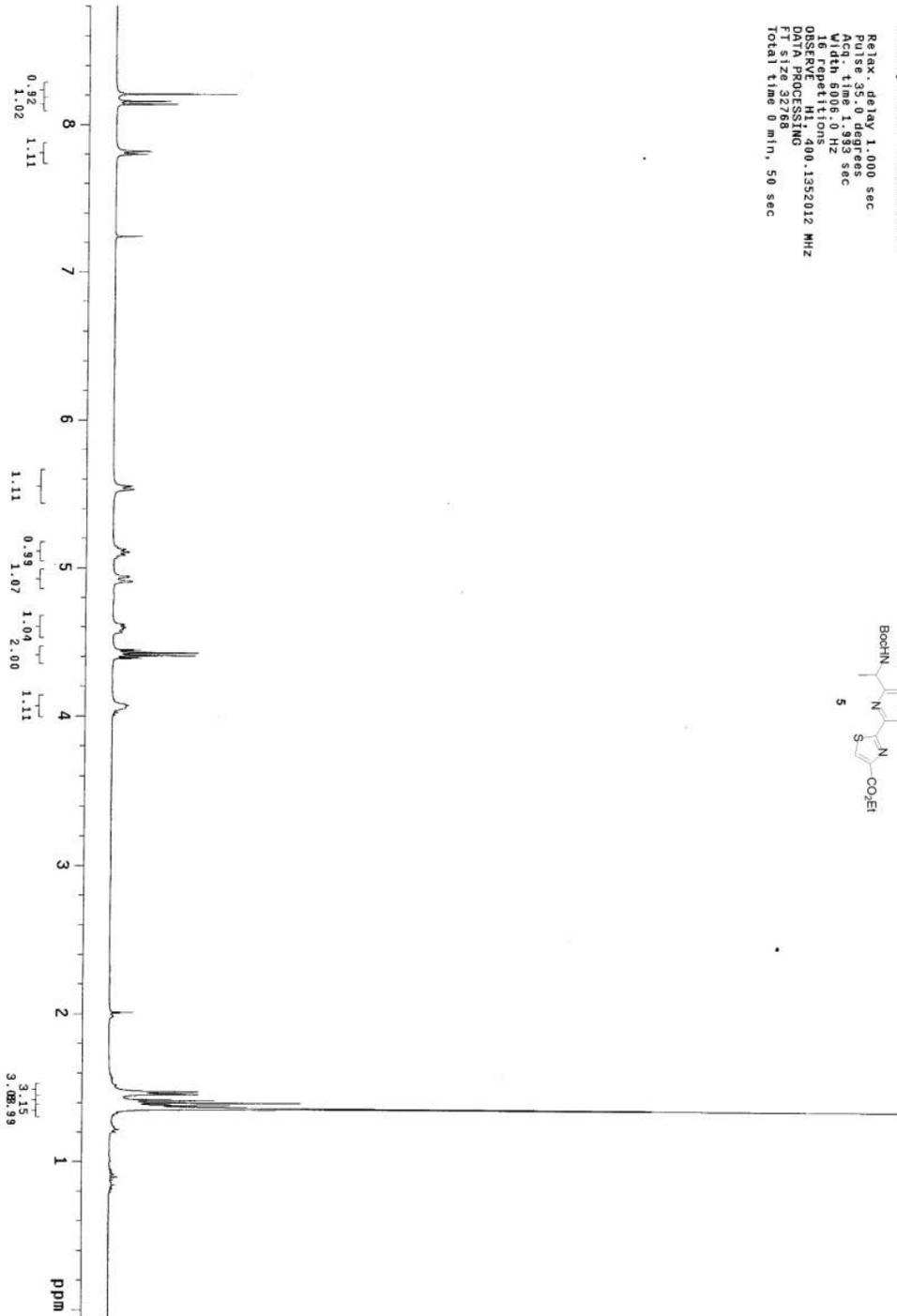
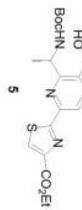


7-178-C 2

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SI size 65536  
Total time 2 hr, 26 min, 38 sec



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Ambient temperature

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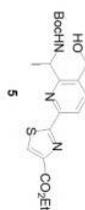
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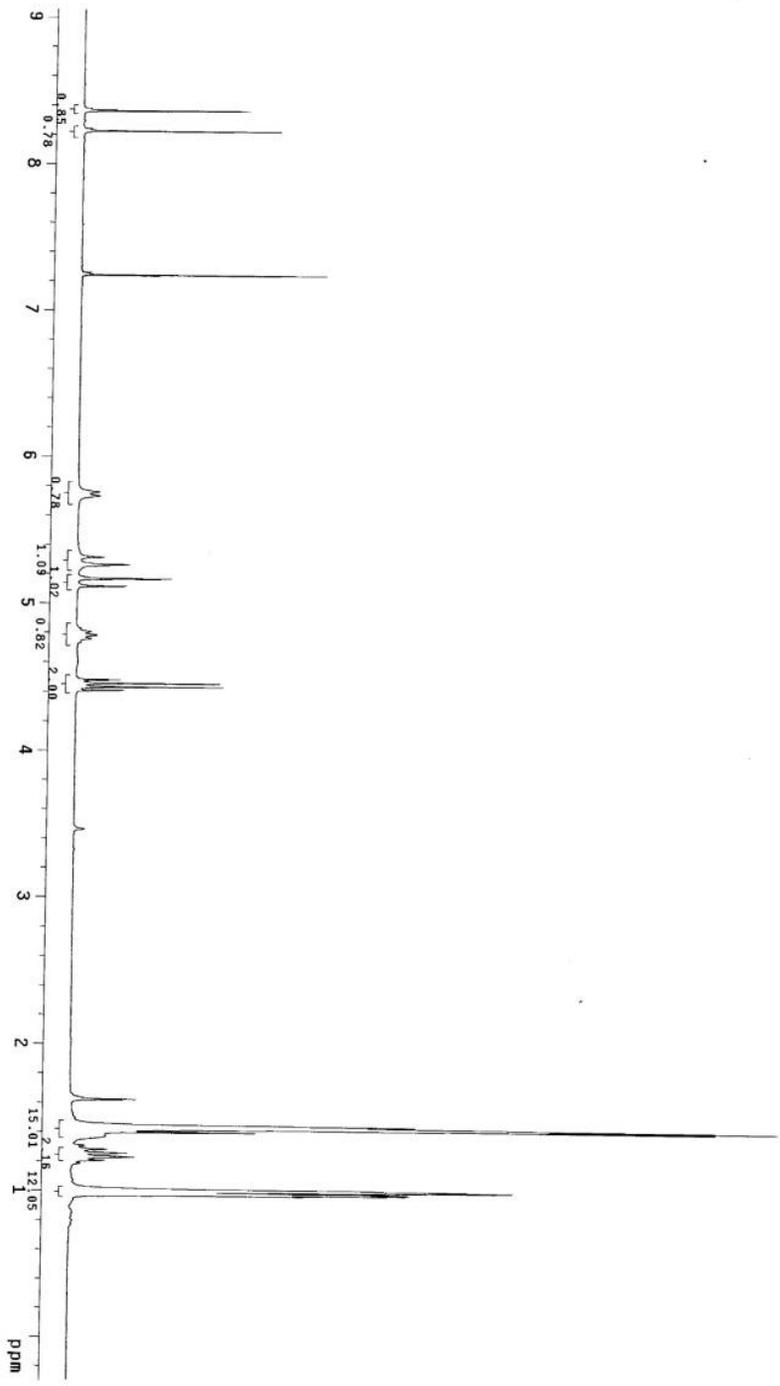
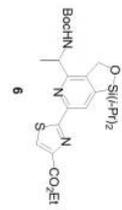
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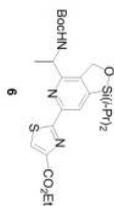
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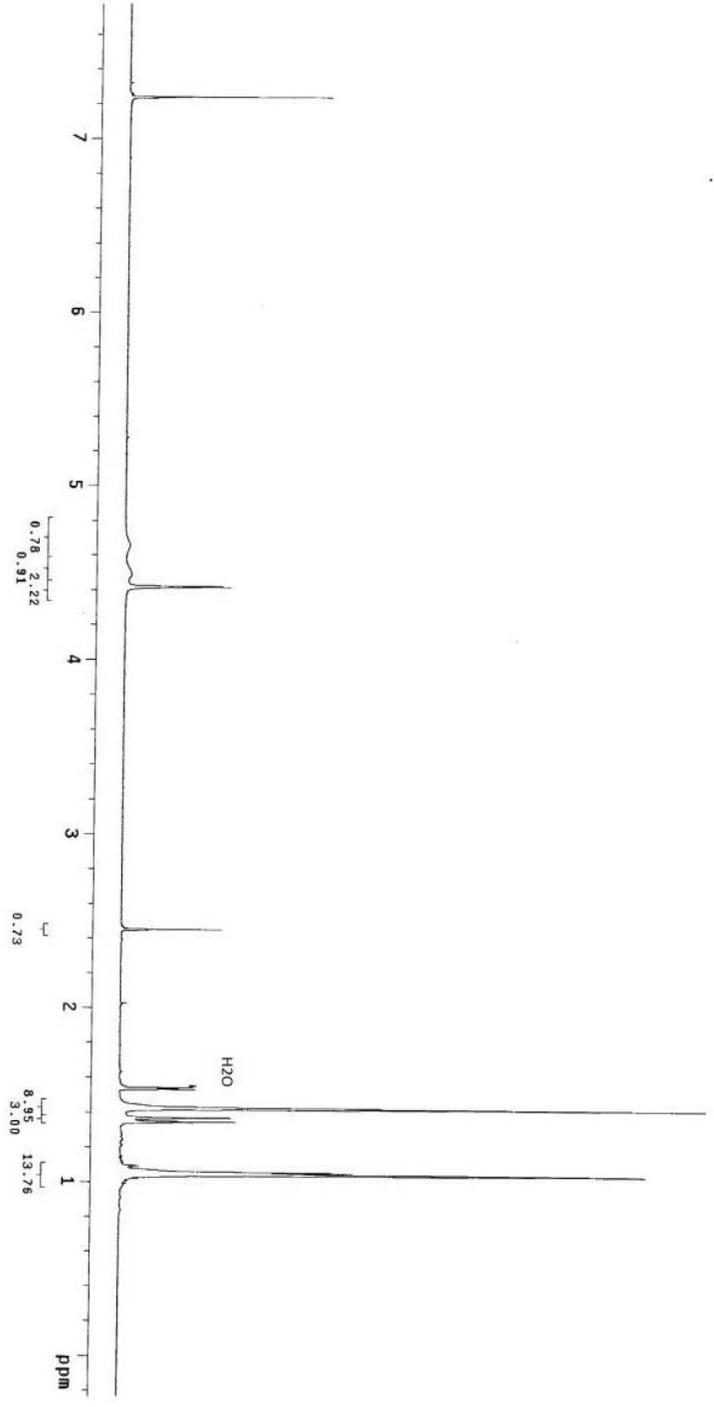
13C OBSERVE

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Solvent: CDCl3  
Ambient temperature  
Mercury-400DB "ncsumarc100"

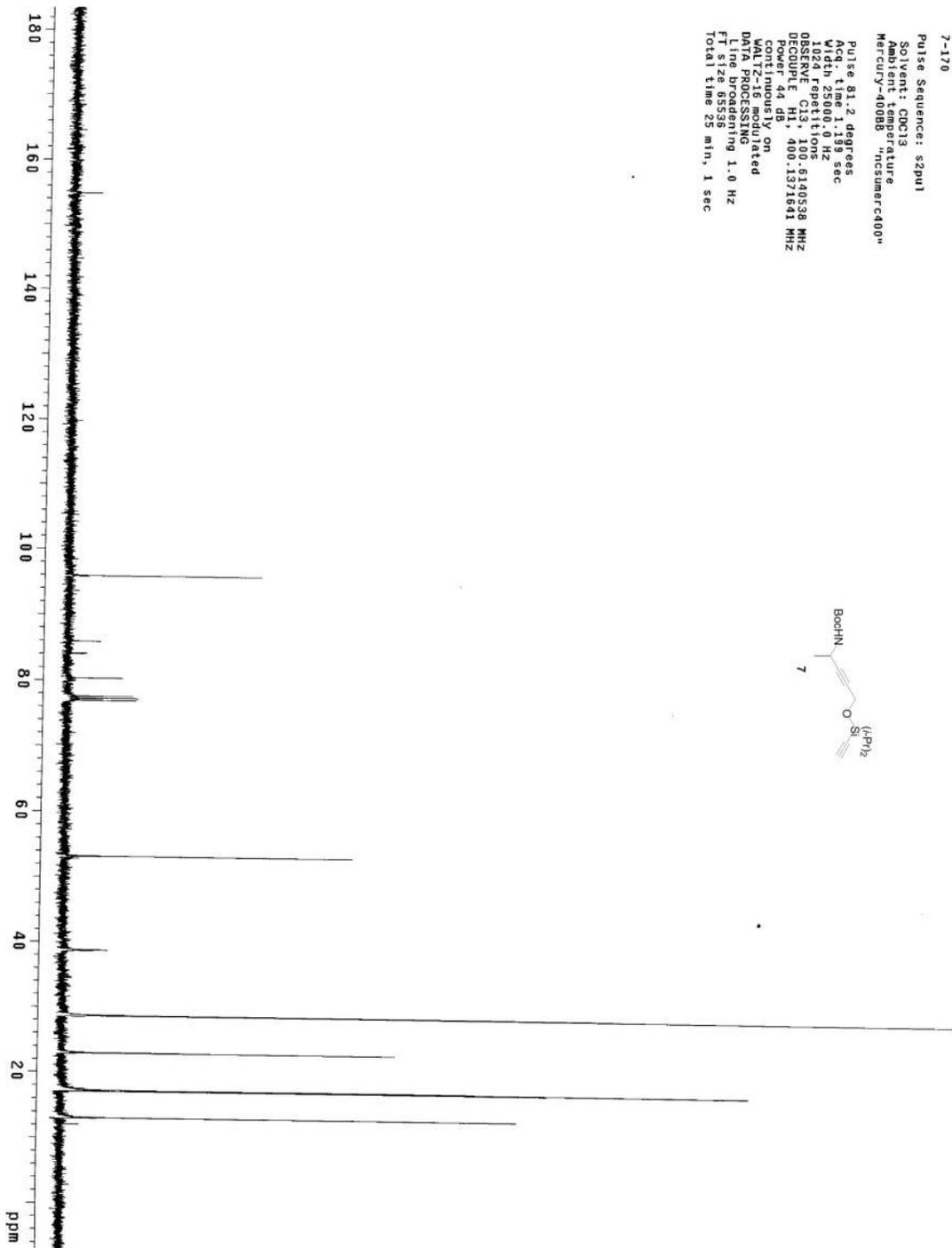
Pulse 81.2 deg/sec  
Acq time 1.59 sec  
Width 25000.0 Hz  
1744 repetitions  
OBSERVE C13, 100.6140568 MHz  
DECUPLE H1, 400.1571641 MHz  
COUPLE 4 H1  
CONTINUOUSLY ON  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
F1 size 65538  
Total time 2 hr, 26 min, 38 sec



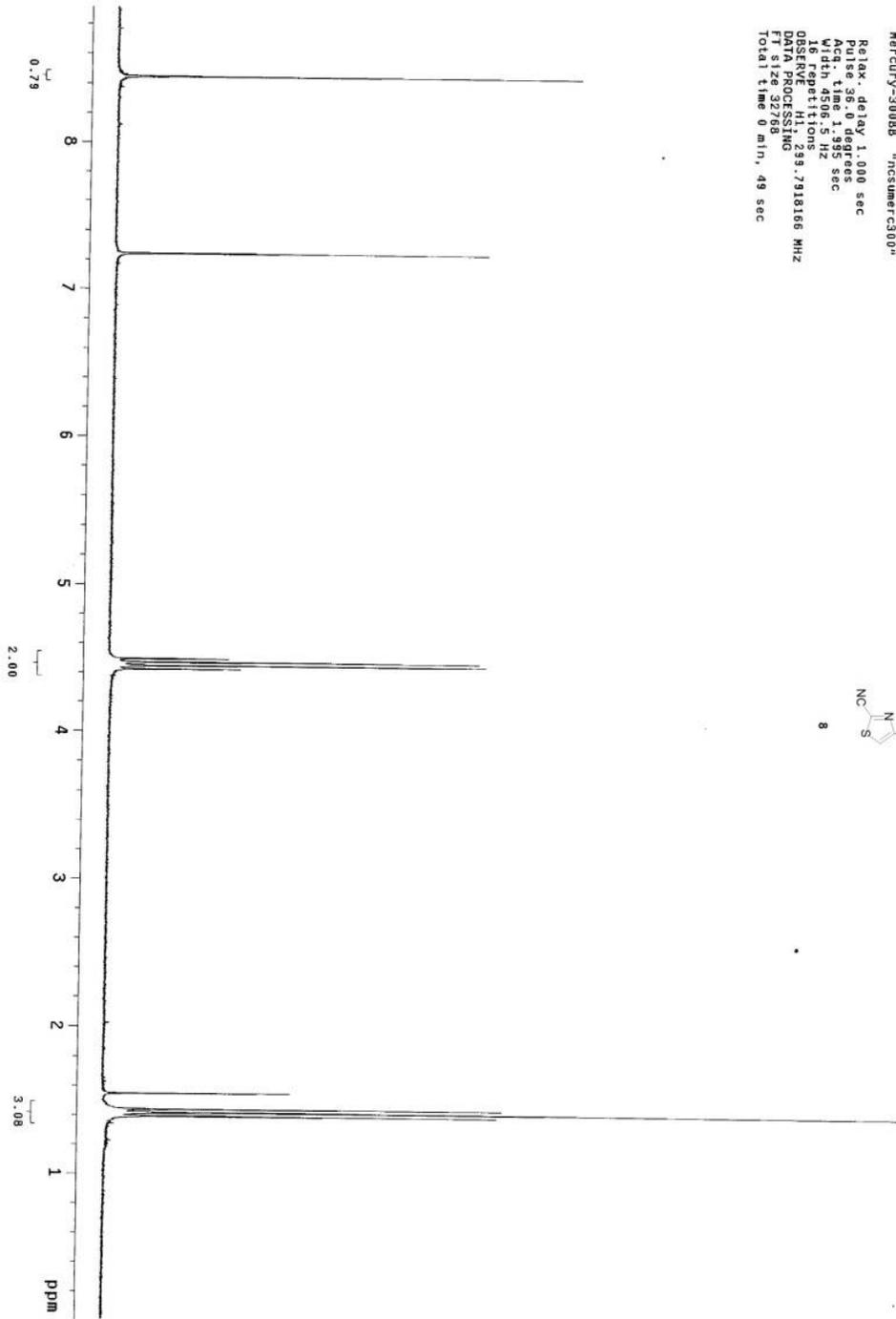
7-170  
 Pulse Sequence: s2pu1  
 Solvent: CDCl3  
 Ambient temperature  
 File: YZ-7-170  
 Mercury-300DB "ncsmrcs38"  
 Relax. delay 1.000 sec  
 Pulse 45.7 degrees  
 Pulse time 1.935 sec  
 Width 6.000  
 16 repetitions  
 OBSERVE H1, 300.0953838 MHz  
 DATA PROCESSING  
 FT size 32768  
 Total time 0 min, 49 sec



7-170  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-40088 "hcsuserc400"  
Pulse 81.2 degrees  
Acq. time 1.189 sec  
SFO 132811.0 MHz  
1024 F2S411.0 MHz  
OBSERVE C13, 100.6140538 MHz  
DECUPLE H1, 400.1371641 MHz  
Power 44 dB  
Continuously on  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 25 min, 1 sec



7-69 8  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient Temperature  
File: YZ-7-69  
Mercury-300BB "ncsummerc300"  
Relax. delay 1.000 sec  
Pulse 35.0 degrees  
Acq. time 1.995 sec  
Acq. date 11/11/95  
16 Repetitions  
OBSERVE H1 299.7918166 MHZ  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 49 sec



13C OBSERVE

Pulse Sequence: szpu1

Solvent: CDCl3

Acq. Temp: 300.2

File: YZ-7-68-C "ncsmcr300"

Mercury-300BB "ncsmcr300"

Pulse 39.0 degrees

Acq. time 1.815 sec

Width 18781.7 Hz

OBSERVE C13 1075.3826965 MHz

DECOUPLE H1 299.7932667 MHz

Power 40 db

continuously on

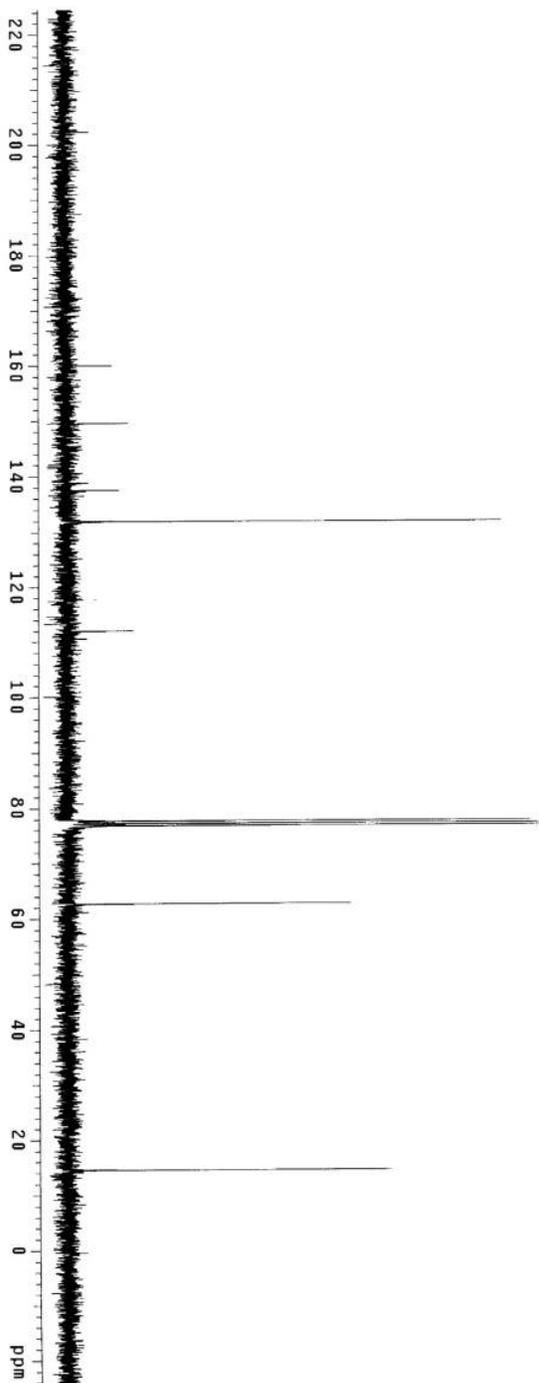
MULTI216 modulated

DM 1000000

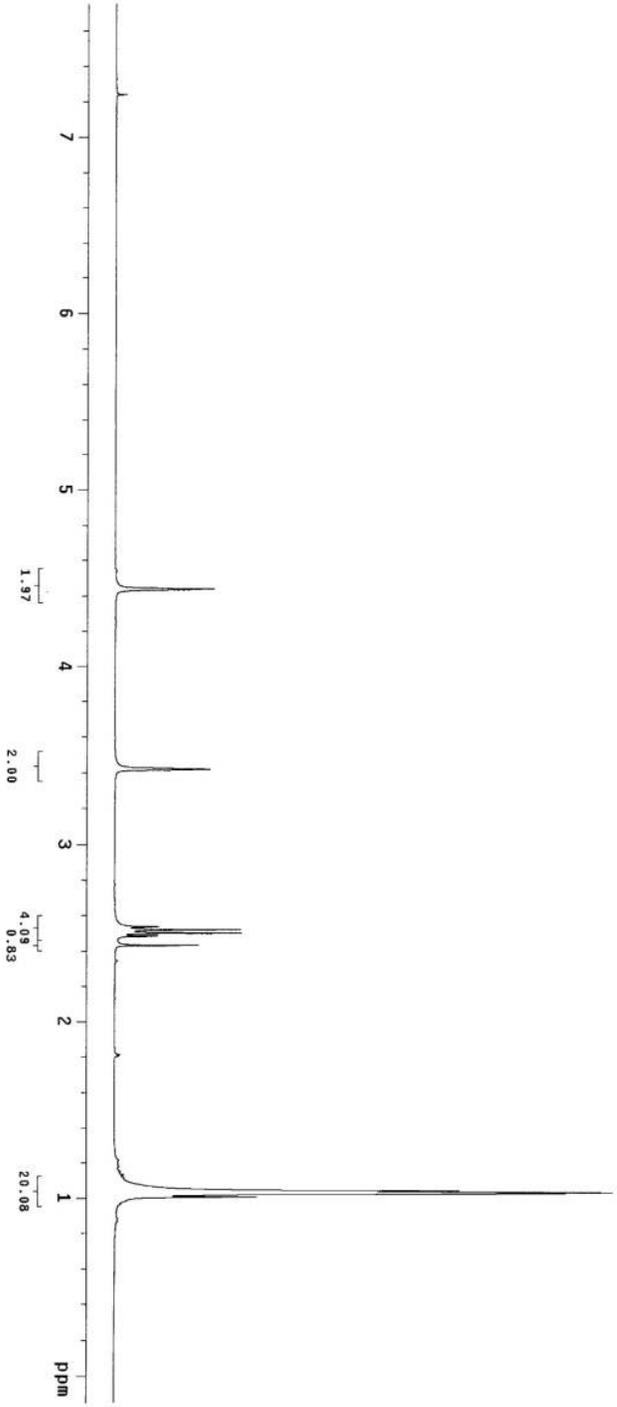
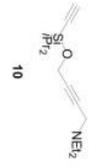
Line broadening 1.0 Hz

FT size 131072

Total time 1 hr. 12 min. 1 sec



8-25-H  
 Pulse Sequence: szpu1  
 Solvent: GDC13  
 Ambient temperature  
 Mercury-400BB "ncsummerc400"  
 Relax. delay 1.000 sec  
 Pulse 35.0 degrees  
 Acq. time 1.933 sec  
 16 Repetitions  
 OBSERVE H1 400.1352016 MHZ  
 DATA PROCESSING  
 FT size 32768  
 Total time 0 min, 50 sec



13C OBSERVE

Pulse Sequence: zgpg30

Solvent: CDCl3

Acq: 400 MHz

File: YZ-8-25-10

Mercury-40088 "ncsumarc400"

Pulse 81.2 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

ASB

DECOUPLE M1, 400.6140538 MHz

Power 44 dB

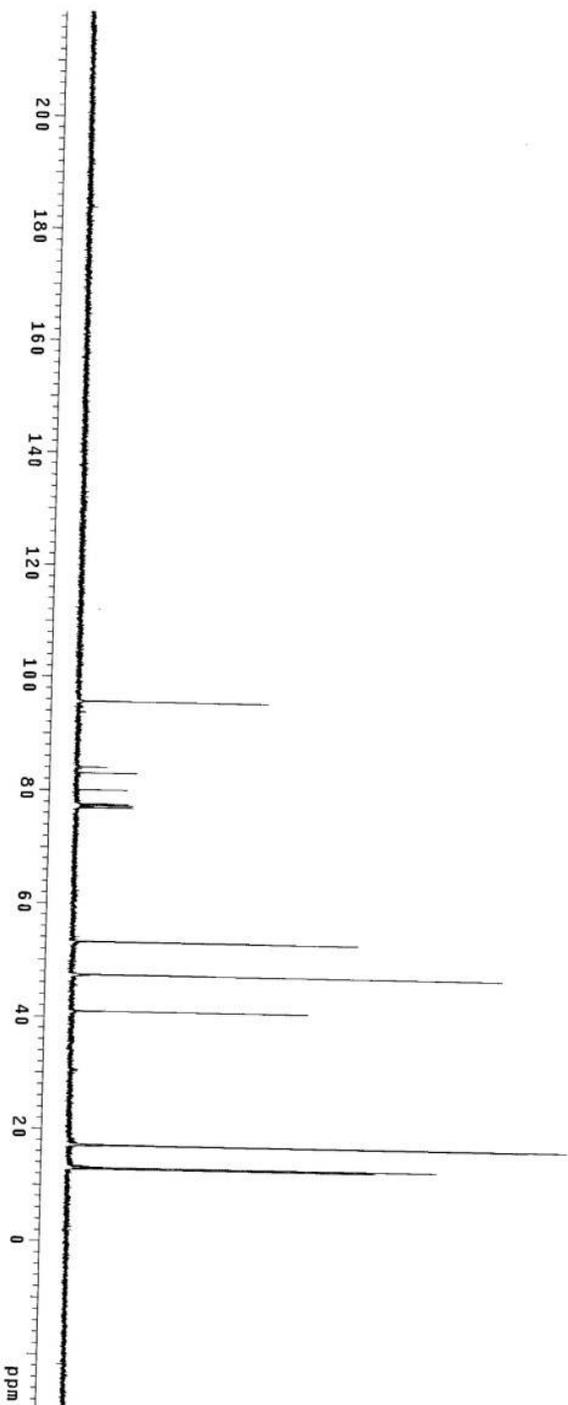
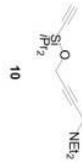
continuously on

WALTZ-16 modulated

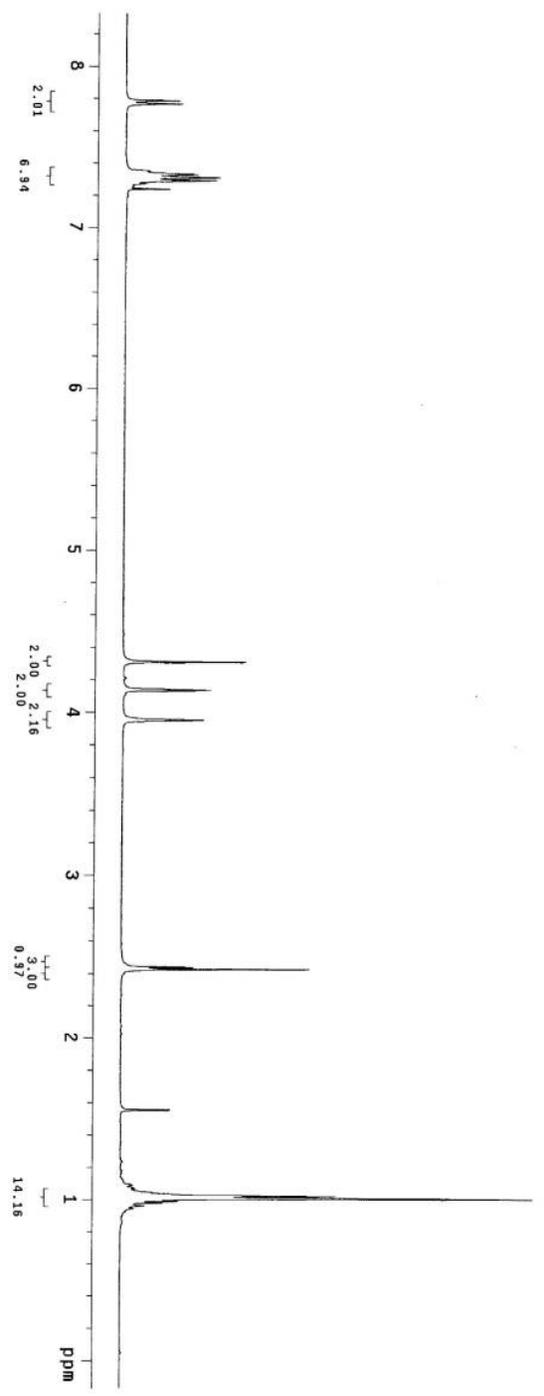
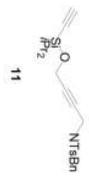
DATA PROCESSING

FT size 65536

Total time 25 min, 1 sec

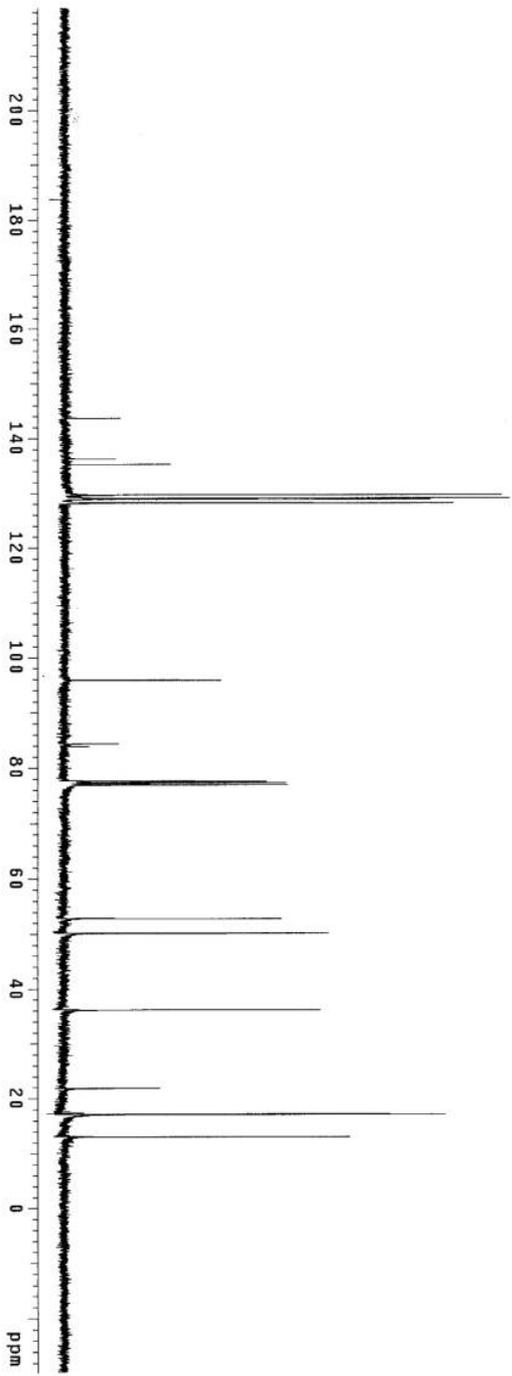
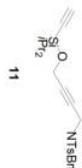


7-74-H  
 Pulse Sequence: szpu1  
 Solvent: GDCl3  
 Ambient Temperature  
 Mercury-40088 "ncsummer400"  
 Relax. delay 1.000 sec  
 Pulse 35.0 degrees  
 Acq. time 1.993 sec  
 V.D.N. delay 0.0 Hz  
 T.D.N. delay 0.0 Hz  
 OBSERVE H1 400.1352023 MHZ  
 DATA PROCESSING  
 FT size 32768  
 Total time 0 min, 50 sec

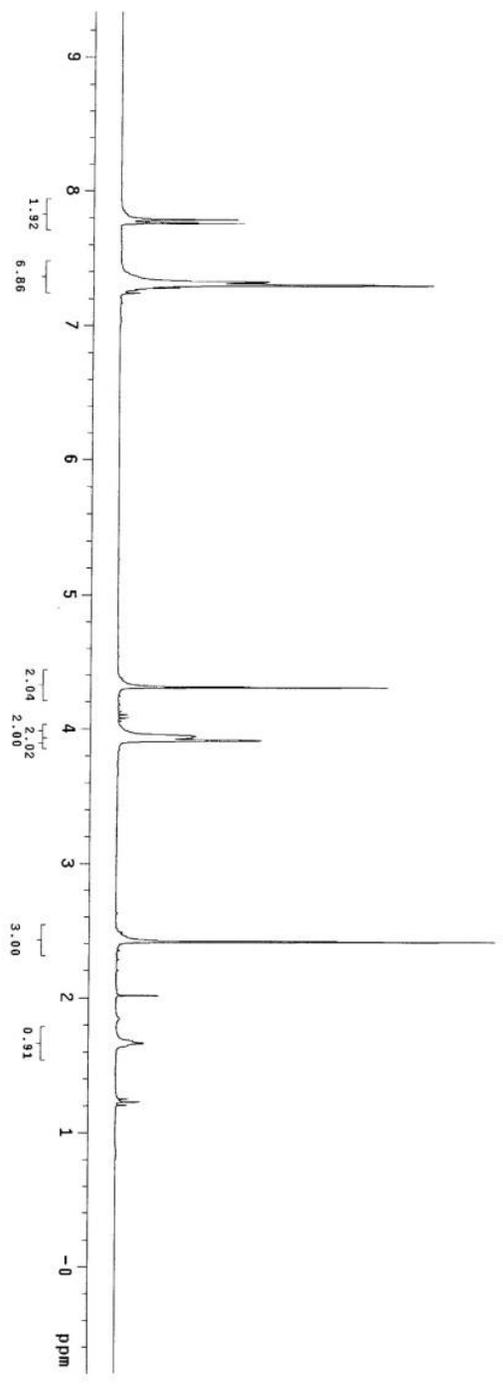
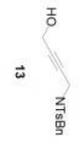


13C OBSERVE

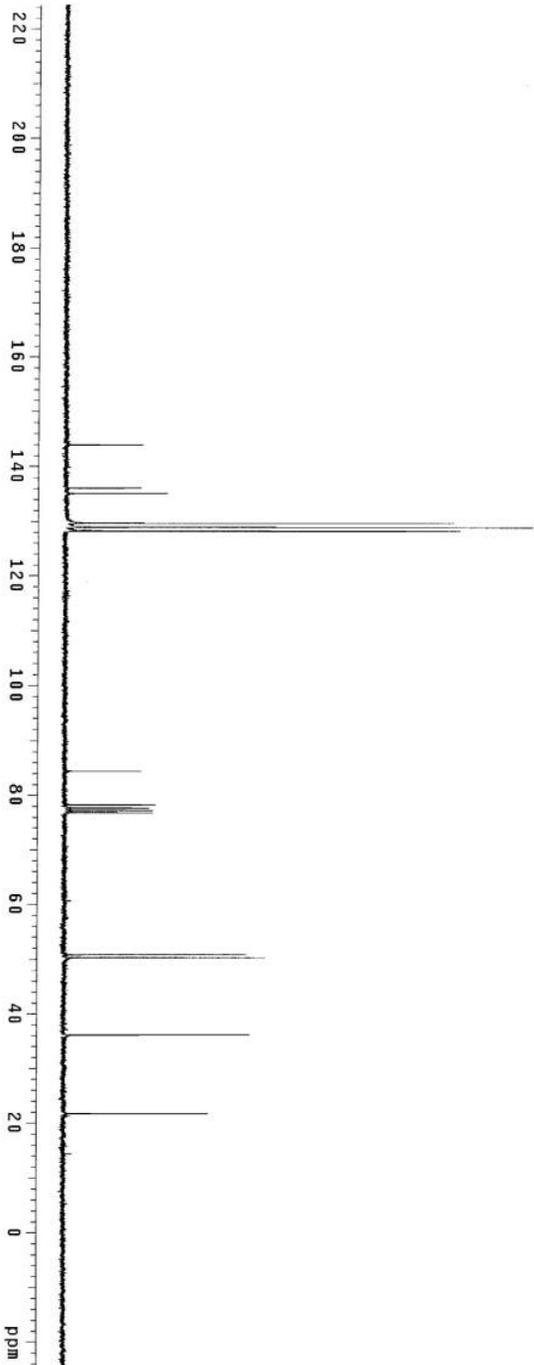
Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Mercury-4000B "ncsumerc400"  
T-1L  
Pulse 81.2 degrees  
Width 2500.0 Hz  
3998 repetitions  
OBSERVE C13, 100.6140530 MHz  
DECUPLE H1, 400.1371641 MHz  
Power 14.8  
Power pulses 09  
VOLTZ-18 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 85536  
Total time 2 hr, 26 min, 38 sec



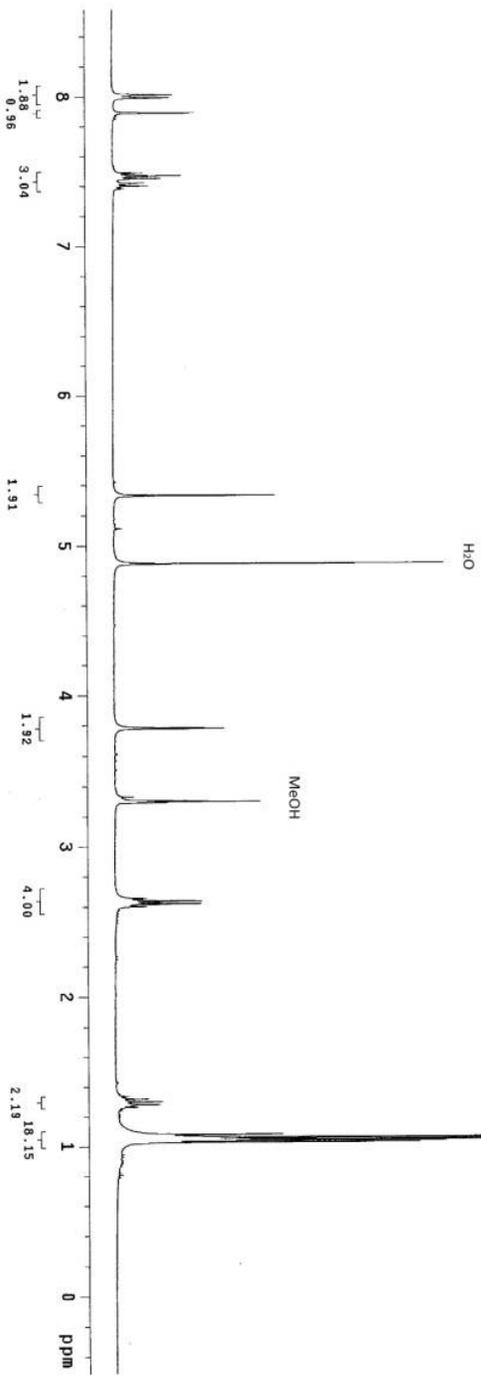
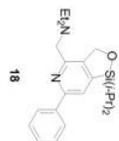
8-34  
 Pulse Sequence: s2pul1  
 Solvent: CDCl3  
 Acquisition Temperature: 300.2 K  
 Mercury-300MR "hncsummerc300"  
 Relax. delay 1.000 sec  
 Pulse 35.0 degrees  
 Acq. time 1.995 sec  
 Width 4508.5 Hz  
 N1 Spectral Width 299.7918155 MHz  
 OBSERVE M1  
 DATA PROCESSING  
 FT size 32768  
 Total time 0 min, 49 sec



8-34-C  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Sample Temperature  
Methyl 300000 "NCSMERC300"  
Pulse 39.0 degrees  
Acq. time 1.815 sec  
Width 18761.7 Hz  
38000 repetitions  
OBSERVE CH: 75.3827005 MHz  
PULSEPROG: zgpg30  
Power 40 dB  
Continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
F1 size 1314072  
Total time 1 hr, 45 min, 38 sec



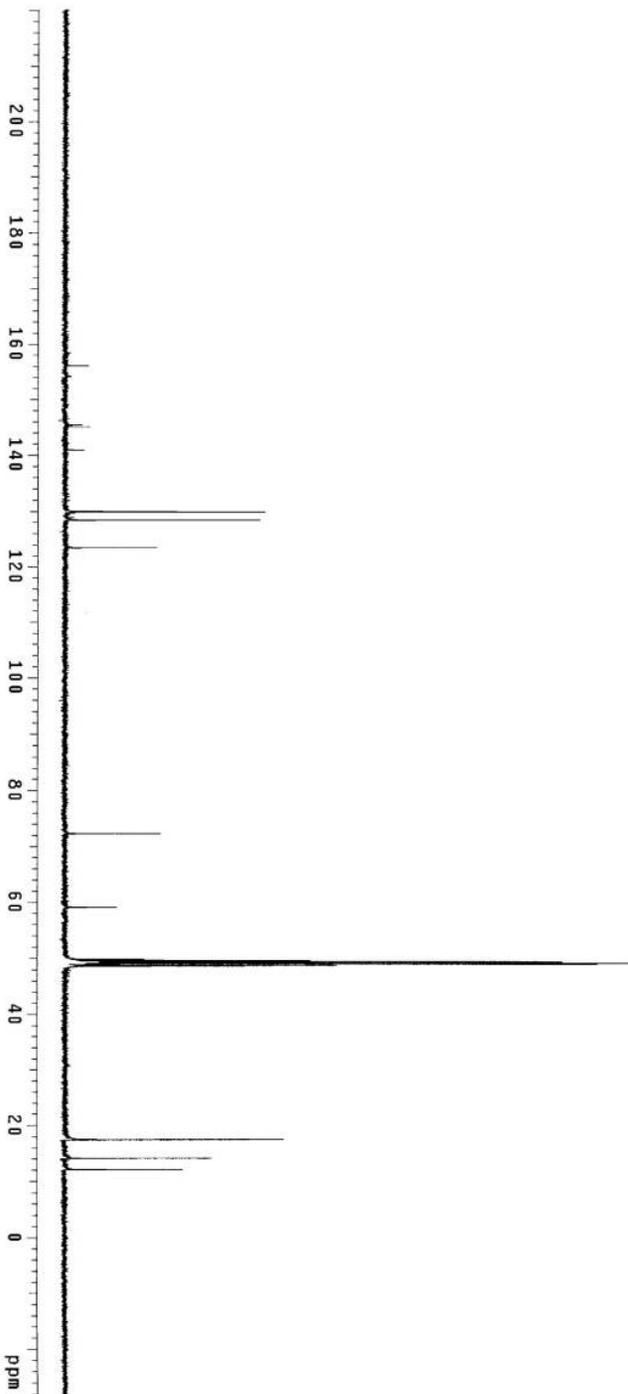
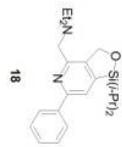
8-38-H 18  
Pulse Sequence: s2pu1  
Solvent: CD3OD  
Acq. Temp: 300.000000  
Mercury-400BB "ncsuser-c400"  
Relax. delay 1.000 sec  
Pulse 35.0 degrees  
Acq. time 1.993 sec  
Width 6000.0 Hz  
F2 400.146400 MHz  
Observer: H1 400.136725 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 50 sec



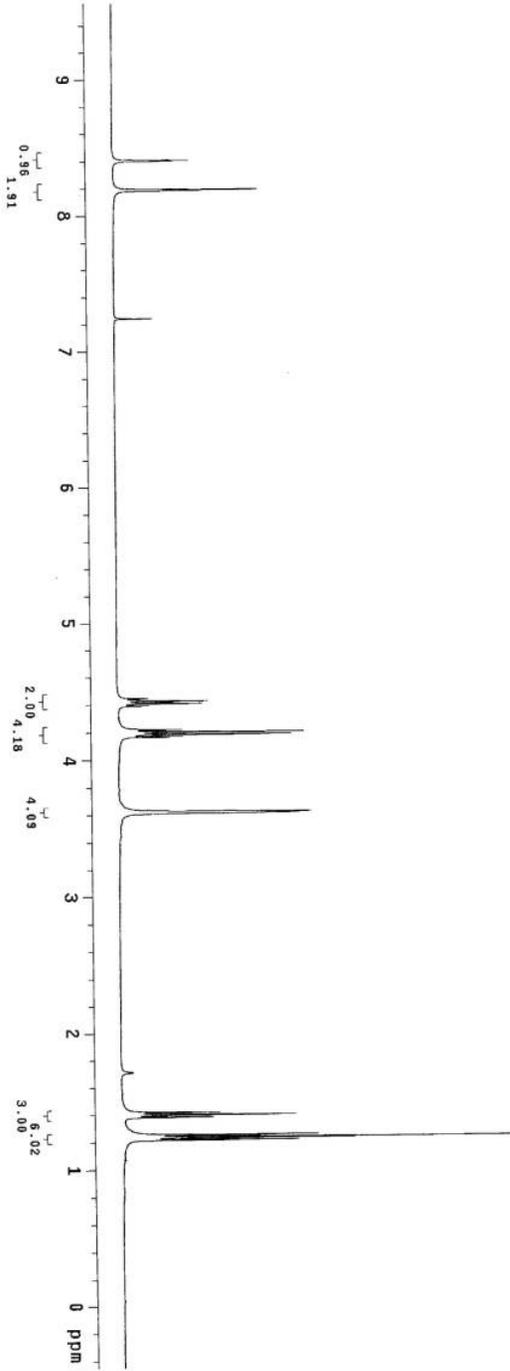
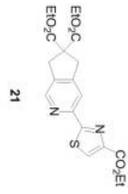
13C OBSERVE

Pulse Sequence: s2pu1  
Solvent: CD3OD  
Ambient Temperature  
Mercury-40055 "ncsmrcc400"

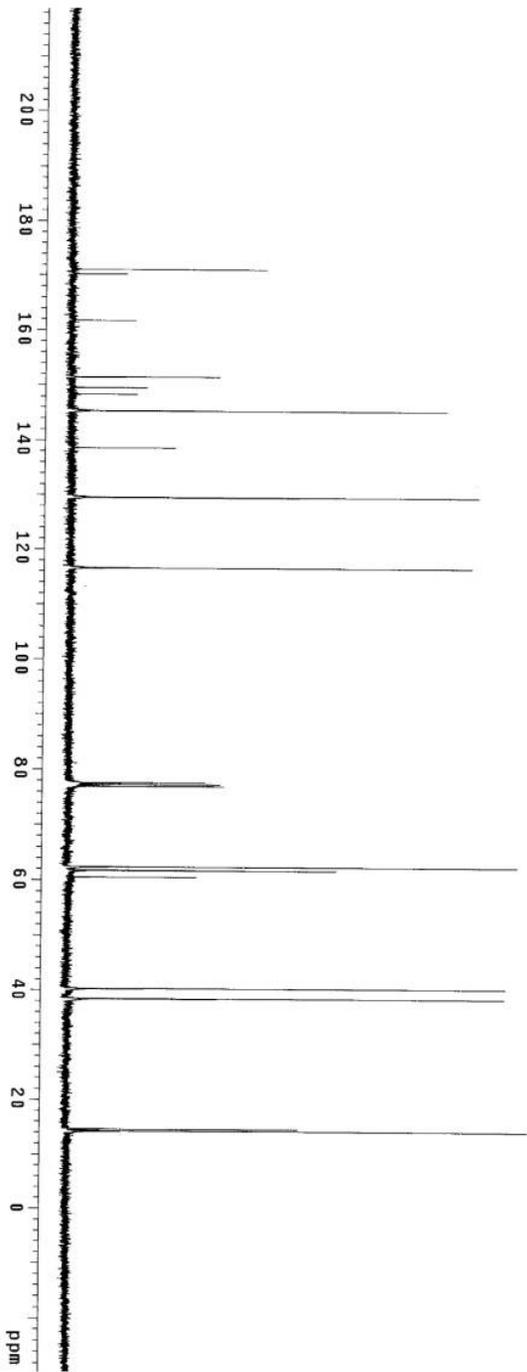
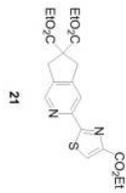
Pulse 81.2 degrees  
Acq. 1.00000000 sec  
Width 2560.139 Hz  
4960 repetitions  
OBSERVE C13, 100.5143130 MHz  
DECUPLE H1, 406.1387407 MHz  
Power 44 dB  
CONTINUOUSLY ON  
VERTICALLY SKIPPED  
DATA PROCESSING  
Line Broadening 1.0 Hz  
FT size 65536  
Total time 2 hr, 26 min, 38 sec



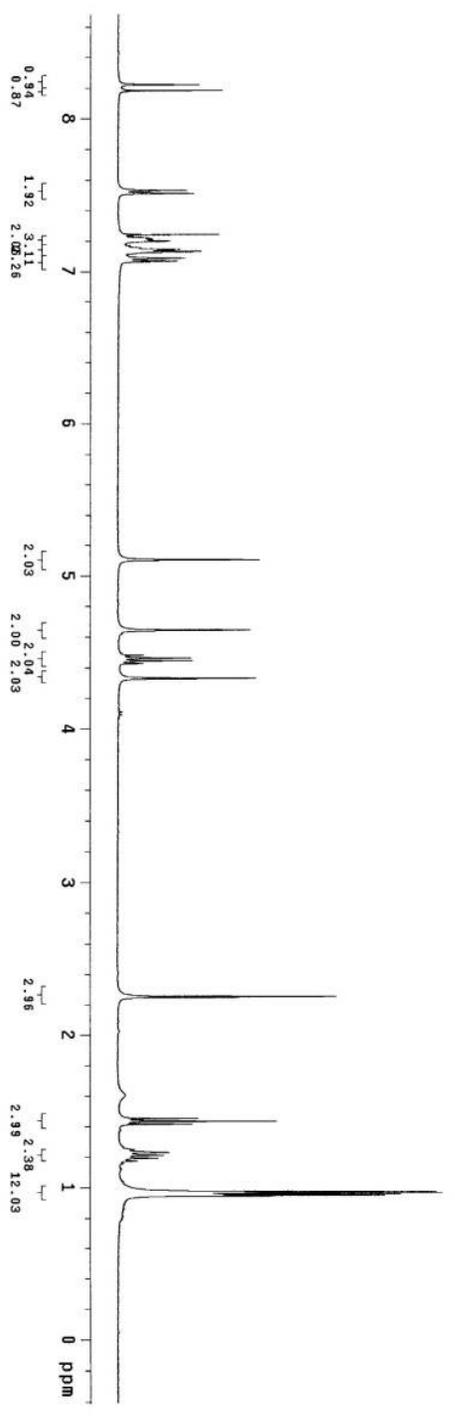
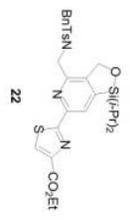
7-95-H  
 Pulse Sequence: s2pu1  
 Solvent: CDCl3  
 Solvent Temperature: 25.00  
 Mercury-40088 "mcsumarc400"  
 Relax. delay 1.000 sec  
 Pulse 35.0 degrees  
 Acq. time 1.993 sec  
 Width 6008.0 Hz  
 Frequency 101.625 MHz  
 OBSERVE 1H 400.1352030 MHz  
 DATA PROCESSING  
 FT size 32768  
 Total time 0 min, 50 sec



7-95-C  
Pulse Sequence: szpul  
Solvent: CDCl3  
Acq. time: 1.139 sec  
MERCURY-400BB InverseGamma  
Pulse 81.2 degrees  
Acq. time 1.139 sec  
Width 25000.0 Hz  
3860. repetitions  
0.3860. repetition  
DECUPLE C13, 400.9140553 MHz  
Power 44 db, 400.1371491 MHz  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Solving for 1.0 Hz  
FT size 65536  
Total time 2 hr, 26 min, 38 sec

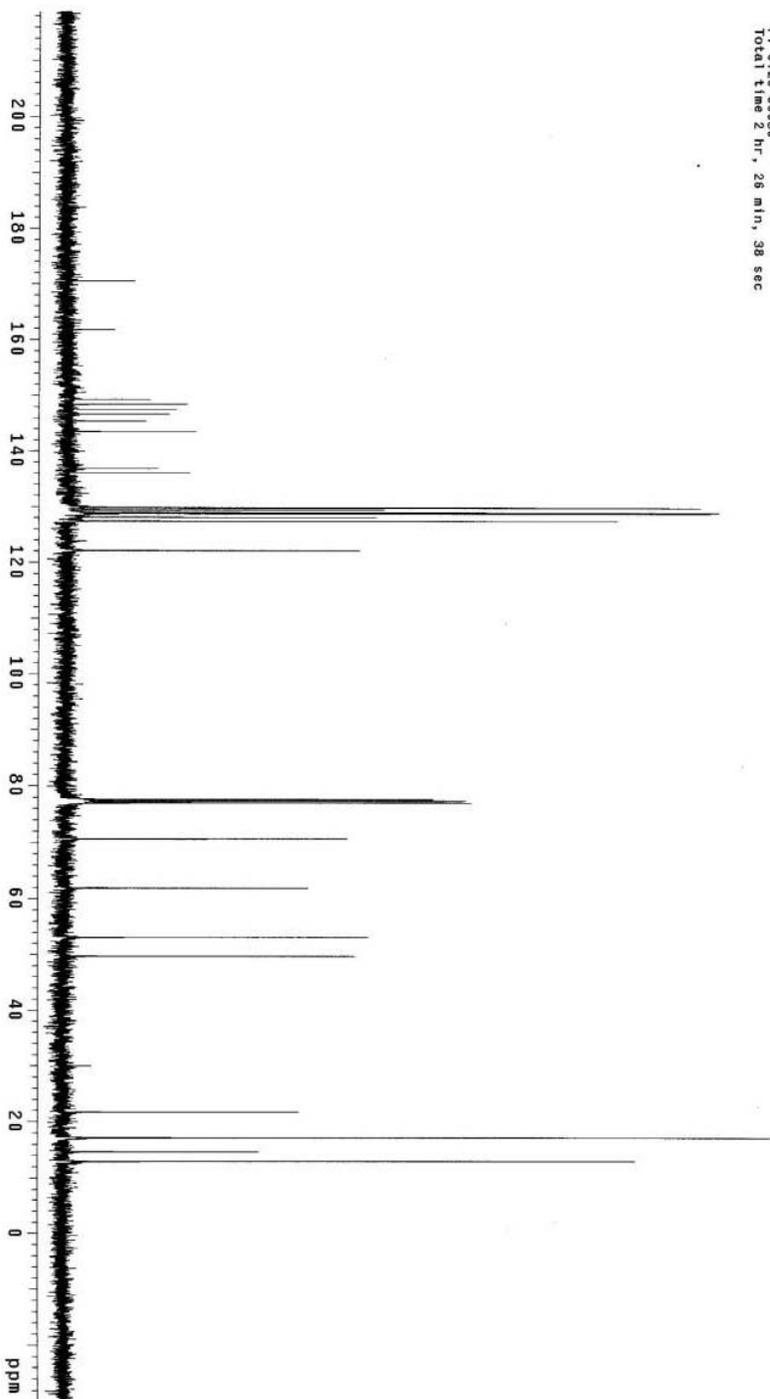
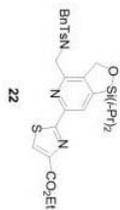


8-24-H  
 Pulse Sequence: szpu1  
 Solvent: CDCl3  
 Ambient temperature  
 Filter: YZ-8-24-H  
 Mercury-40008 "mcuserc400"  
 Relax delay 1.000 sec  
 Relax delay 3.000 sec  
 Acq. time 1.989 sec  
 Width 6005.0 Hz  
 16 Repetitions  
 OBSERVE: H1; 400.1352012 MHz  
 F1 A: PULPROG: zgpg30  
 F2 A: PC: zgpg30  
 Total time 0 min, 50 sec

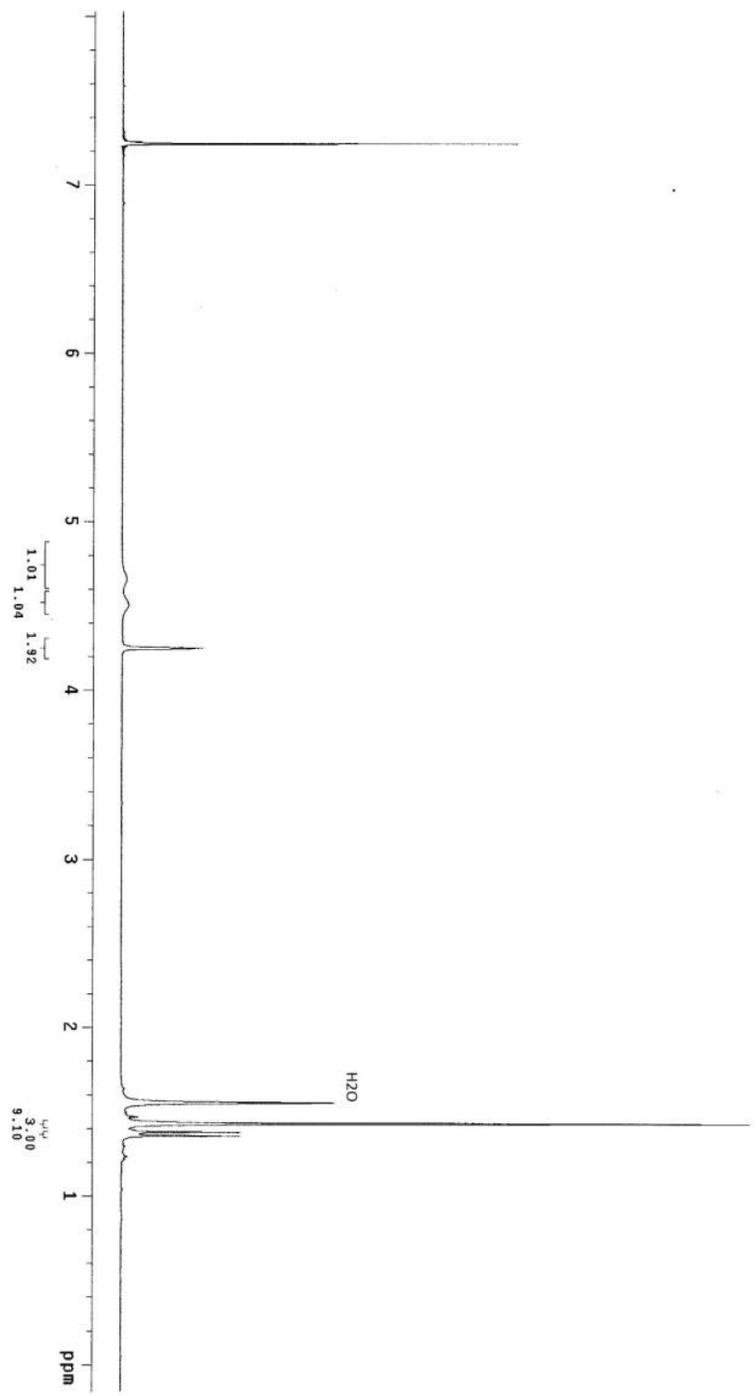
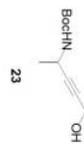


13C OBSERVE

Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient Temperature  
Mercury-100DB "ncsmarc400"  
Pulse B1, 2, dephase  
Acq time 1.18 sec  
Width 25000.0 Hz  
3584 repetitions  
OBSERVE C13, 100.6140530 MHz  
DECOUPLE H1, 400.1371641 MHz  
SFO 400.1371641  
Continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 2 hr, 26 min, 38 sec



7-153  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient Temperature  
Mercury-300DB "hcnsmrcs39"  
Relax: d63w 1.000 sec  
Pulse: 45 degrees  
Acq. time: 1.995 sec  
Width: 4506.5 Hz  
64 repetitions  
OBSERVED: H1: 300.0953843 MHz  
F1: 300.0953843 MHz  
F2: size 32768  
Total time 3 min, 18 sec



13C OBSERVE  
7.19C  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient Temperature  
Mercury-400DB "hcsuwar-c400"  
Pulse 81.2 degrees  
Acq. time 1.199 sec  
Width 25000.0 Hz  
Observed 13C  
OBSERVE P1 1.000  
OBSERVE P2 1.000  
DECUPLE H1 400.1371641 MHz  
Power 44 dB  
continuously on  
MULTI 2.06 accumulated  
Data 1.000 sec  
Line broadening 1.0 Hz  
FT size 65536  
Total time 1 hr, 13 min, 21 sec

