

## Supplementary Information for

### **Pd catalyzed cross coupling reactions of less activated alkenyl electrophiles (for tosylates and mesylates) with tosylhydrazones: synthesis of various 1,3-dienes**

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## 1. Reagents

All reactions were carried out under a nitrogen atmosphere. 1,4-Dioxane and toluene were dried over Na with benzophenone-ketyl intermediate as indicator. Air- and moisture-sensitive solvents and solutions were transferred via syringe or stainless-steel cannula. All other chemicals were used as received from the appropriate suppliers. Solvents used were of analytical grade. All reactions were routinely checked by TLC. TLC was performed on aluminium-backed silica gel plates (silica gel 60 F254 grade, Merck DC) with spots visualized by UV light. Column chromatography was performed on silica gel LC 60A (70-200 micron).

## Instrumental

All compounds were characterized by  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR as well as elemental analysis. Melting points were determined in open capillaries on a Veego electronic apparatus VMP-D (Veego Instrument Corporation, Mumbai, India) and are uncorrected.  $^1\text{H}$ NMR and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker400 MHz model spectrometer using  $\text{DMSO-d}_6$  as a solvent and TMS as internal standard with  $^1\text{H}$  resonant frequency of 400 MHz and  $^{13}\text{C}$  resonant frequency of 100 MHz. The  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR chemical shifts were reported as parts per million (ppm) downfield from TMS ( $\text{Me}_4\text{Si}$ ). The splitting patterns are designated as follows; s, singlet; d, doublet; t, triplet; m, multiplet. Elemental analyses (C, H, N) were performed using a Heraeus CarloErba 1180 CHN analyzer (Hanau, Germany).

## 2. Preparation of alkenyl tosylates and mesylates substrates

Pyronyl tosylates and mesylates were prepared from their corresponding precursors with TsCl or MsCl in the presence of triethylamine in CH<sub>2</sub>Cl<sub>2</sub> according to the literature method without modifications.<sup>1</sup> Other alkenyl tosylates and mesylates were prepared from their corresponding species according to the literature method without modifications.<sup>2</sup>

## 3. General Procedure for Preparation of Hydrazones.<sup>3</sup>

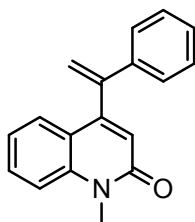
To an oven dried flat-bottomed flask previously equipped with a magnetic stir bar, was charged with p-toluenesulphonylhydrazide (5 mmol) in dry methanol (10 mL) at 60 °C, the ketone (5 mmol) was added drop wise. After the completion of reaction the product was began to precipitate. The crude product was filtered, washed with petroleum ether: ethyl acetate (10:1) and dried to afford the corresponding pure N-tosylhydrazones. The reaction provides the *N*-tosylhydrazone derivatives in about 85–98% yields.

## 4. Typical procedure for Pd-catalyzed N-tosylhydrazones coupling with heteroaryl pseudohalides

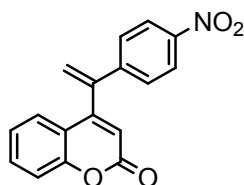
To an oven dried flat-bottomed flask which was equipped with a condenser under nitrogen atmosphere was charged with *N*-tosylhydrazone **1** (1.0 mmol), base (3 mmol), ligand (3 mol %), Pd (1.5 mol %), stir it for two minutes thereafter alkenyl tosylate **2** (1.0 mmol) was added in the above reaction mixture (5.0 mL). The reaction was stirred and heated to 90 °C for 1-2 hours. After completion of reaction, the crude reaction mixture was cooled to room temperature and filtered

through a pad of Celite eluting with ethyl acetate. The filtrate was concentrated and purified by column chromatography on silica gel.

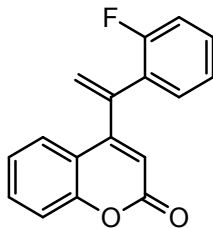
### 5. Characterization of coupling yield



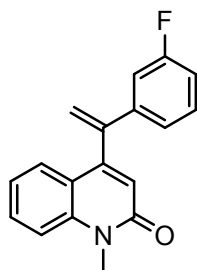
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 7.82 (dd, *J* = 7.4, 2.5 Hz, 1H), 7.44-7.30 (m, 5H), 7.17 (dd, *J* = 7.5, 2.0 Hz, 2H), 7.07 (dd, *J* = 7.1, 1.5 Hz, 1H), 6.20 (s, 1H), 5.52 (s, 1H), 5.21 (s, 1H), 3.49 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 165.52, 144.30, 142.82, 139.03, 129.45, 128.75, 128.37, 128.05, 126.85, 124.75, 124.58, 121.75, 120.18, 117.70, 115.11, 29.56. **Anal. Calcd.** For C<sub>18</sub>H<sub>15</sub>NO: C: 82.77; H: 5.79; N: 5.36. **Found:** C: 82.68; H: 5.57; N: 5.38. **mp** 202-204 °C.



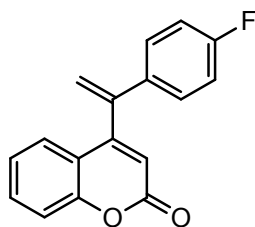
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 7.94 (d, *J* = 7.4 Hz, 2H), 7.64-7.42 (m, 4H), 7.24 (d, *J* = 8.1 Hz, 2H), 6.64 (s, 1H), 5.71 (s, 1H), 5.28 (s, 1H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 161.37, 153.81, 151.78, 147.43, 146.12, 141.82, 131.87, 128.93, 126.55, 125.48, 123.27, 120.60, 119.42, 118.29, 109.17. **Anal. Calcd.** For C<sub>17</sub>H<sub>11</sub>NO<sub>4</sub>: C: 69.62; H: 3.78; N: 4.78. **Found:** C: 69.68; H: 3.55; N: 4.79. **mp** 159-162 °C.



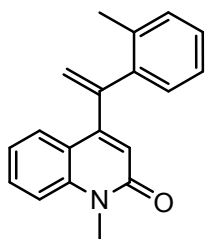
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 7.82 (dd, *J* = 7.4, 1.5 Hz, 1H), 7.59 (t, *J* = 7.1 Hz, 1H), 7.36-7.17 (m, 6H), 6.26 (s, 1H), 5.52 (s, 1H), 5.38 (s, 1H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 165.26, 158.25 (d, *J* = 240.2 Hz), 152.76, 151.24, 140.35, 132.48, 131.19 (d, *J* = 7.1 Hz), 128.93, 128.81 (d, *J* = 15.2 Hz), 126.57 (d, *J* = 7.6 Hz), 125.48, 124.54 (d, *J* = 3.3 Hz), 120.69, 118.54, 117.24, 115.72 (d, *J* = 13.4 Hz), 105.32. **Anal. Calcd.** For C<sub>17</sub>H<sub>11</sub>FO<sub>2</sub>: C, 76.68; H, 4.16. **Found:** C, 76.79; H, 4.16. **mp** 219-221°C.



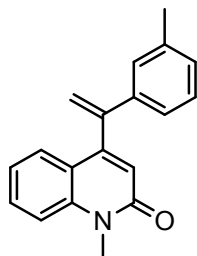
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 7.48-7.44 (m, 2H), 7.32 (dd, *J* = 7.8, 2.1 Hz, 1H), 7.28 – 7.12 (m, 3H), 7.03 (d, *J* = 7.8 Hz, 2H), 6.89 (s, 1H), 5.52 (s, 1H), 5.14 (s, 1H), 3.39 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 164.8, 159.81 (d, *J* = 241.6 Hz), 146.32, 142.41 (d, *J* = 7.9 Hz), 140.86, 138.54, 130.03, 129.39 (d, *J* = 7.5 Hz), 124.67 (d, *J* = 16.4 Hz), 121.86, 121.64 (d, *J* = 3.2 Hz), 120.36, 115.69 (d, *J* = 13.2 Hz), 115.31 (d, *J* = 12.7 Hz), 114.66, 113.57, 113.01, 31.24. **Anal. Calcd.** For C<sub>18</sub>H<sub>14</sub>FNO: C, 77.44; H, 5.05; N, 5.01. **Found:** C, 77.40; H, 5.05; N, 5.13. **mp** 231-233 °C.



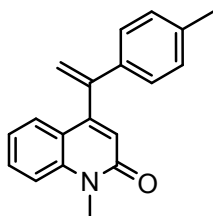
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 7.74 (dd, *J* = 7.9, 2.1 Hz, 1H), 7.61 (dd, *J* = 7.5, 2.0 Hz, 1H), 7.44 – 7.25 (m, 5H), 6.43 (s, 1H), 5.64 (s, 1H), 5.29 (s, 1H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 164.54, 162.23 (d, *J* = 236.7 Hz), 153.45, 152.67, 143.89, 139.37 (d, *J* = 3.1 Hz), 131.71, 129.19 (d, *J* = 8.4 Hz), 125.71, 123.19, 120.73, 117.18, 116.30, 115.27 (d, *J* = 23.6 Hz), 110.78. **Anal. Calcd.** For C<sub>17</sub>H<sub>11</sub>FO<sub>2</sub>: C, 76.68; H, 4.16. **Found:** C, 76.71; H, 4.14. **mp** 219-221°C.



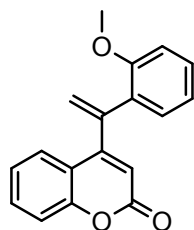
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 8.19 (dd, *J* = 7.7, 2.1 Hz, 1H), 7.76 (dd, *J* = 7.4, 2.0 Hz, 1H), 7.62 (dd, *J* = 7.8, 2.6 Hz, 1H), 7.37-7.23 (m, 3H), 6.98 (dd, *J* = 7.7, 2.0 Hz, 2H), 6.28 (s, 1H), 5.62 (s, 1H), 5.34 (s, 1H), 3.68 (s, 3H), 2.28 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 162.28, 144.58, 142.41, 140.11, 139.23, 137.37, 130.48, 130.79, 129.98, 129.15, 125.35, 124.39, 123.17, 121.49, 119.13, 115.68, 113.58, 29.78, 19.84. **Anal. Calcd.** For C<sub>19</sub>H<sub>17</sub>NO: C, 82.88; H, 6.22; N, 5.09. **Found:** C, 82.79; H, 6.25; N, 5.19. **mp** 183-185°C.



**<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>) δ ppm: 8.05 (dd, *J* = 7.4, 2.1 Hz, 1H), 7.41-7.29 (m, 2H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.16 – 7.01 (m, 4H), 6.27 (s, 1H), 5.71 (s, 1H), 5.24 (s, 1H), 3.61 (s, 3H), 2.31 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-d<sub>6</sub>) δ ppm: 163.38, 146.67, 143.72, 139.11, 138.58, 137.81, 131.19, 129.24, 128.88, 128.45, 127.73, 124.49, 123.71, 121.39 117.58, 115.77, 113.87, 29.67, 21.87. **Anal. Calcd.** For C<sub>19</sub>H<sub>17</sub>NO: C, 82.88; H, 6.22; N, 5.09. **Found:** C, 82.81; H, 6.27; N, 5.11. **mp** 212-215°C.

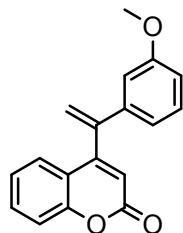


**<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>) δ ppm : 7.84 (dd, *J* = 7.4, 1.9 Hz, 1H), 7.43-7.32 (m, 3H), 7.19-6.98 (m, 4H), 6.19 (s, 1H), 5.67 (s, 1H), 5.21 (s, 1H), 3.64 (s, 3H), 2.32 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-d<sub>6</sub>) δ ppm: 164.51, 144.84, 143.69, 140.58, 139.93, 137.51, 131.29, 129.47, 127.78, 124.13, 123.25, 121.81, 117.45, 115.71, 113.86, 28.96, 21.42. **Anal. Calcd.** For C<sub>19</sub>H<sub>17</sub>NO: C, 82.88; H, 6.22; N, 5.09. **Found:** C, 82.76; H, 6.20; N, 5.09. **mp** 198-200°C.

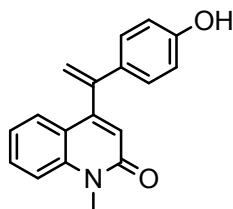


**<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>) δ ppm : 7.78-7.69 (m, 2H), 7.40-7.31 (m, 2H), 7.28 – 7.18 (m, 2H), 7.04-6.98 (m, 2H), 6.57 (s, 1H), 5.54 (s, 1H), 5.29 (s, 1H), 3.67 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-d<sub>6</sub>) δ ppm: 162.85, 157.71, 153.19, 151.32, 141.59, 132.05, 131.43, 130.89, 128.51,

125.74, 124.09, 123.37, 121.47, 120.53, 116.48, 111.96, 109.44, 56.27. **Anal. Calcd.** For  $C_{18}H_{14}O_3$ : C, 77.68; H, 5.07. **Found:** C, 77.64; H, 5.07. **mp** 225-228°C.

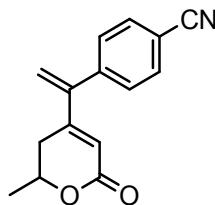


**$^1H$  NMR** (400 MHz,  $DMSO-d_6$ )  $\delta$  ppm : 7.83 (dd,  $J = 7.3, 2.0$  Hz, 1H), 7.66 (dd,  $J = 7.7, 4.7$  Hz, 1H), 7.41-7.25 (m, 3H), 7.01-6.85 (m, 3H), 6.36 (s, 1H), 5.69 (s, 1H), 5.45 (s, 1H), 3.73 (s, 3H).  **$^{13}C$  NMR** (100 MHz,  $DMSO-d_6$ )  $\delta$  ppm: 164.85, 158.97, 153.52, 152.42, 143.71, 139.27, 131.33, 129.38, 125.74, 123.82, 123.27, 120.45, 117.63, 117.54, 117.38, 113.04, 110.59, 55.79. **Anal. Calcd.** For  $C_{18}H_{14}O_3$ : C, 77.68; H, 5.07. **Found:** C, 77.86; H, 5.12. **mp** 205-208°C.

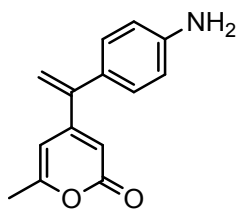


**$^1H$  NMR** (400 MHz,  $DMSO-d_6$ )  $\delta$  ppm: 9.50 (s, 1H), 8.08 (dd,  $J = 7.4, 2.1$  Hz, 1H), 7.41-7.32 (m, 3H), 7.08-6.98 (m, 2H), 6.82 (d,  $J = 7.2$  Hz, 2H), 6.21 (s, 1H), 5.71 (s, 1H), 5.25 (s, 1H), 3.68 (s, 3H).  **$^{13}C$  NMR** (100 MHz,  $DMSO-d_6$ )  $\delta$  ppm: 164.12, 160.26, 145.70, 143.69, 139.43, 135.68, 129.21, 128.72, 124.47, 123.61, 121.87, 116.68, 115.51, 113.86, 29.88. **Anal. Calcd.** For  $C_{18}H_{15}NO_2$ : C, 77.96; H, 5.45; N, 5.05. **Found:** C, 77.91; H, 5.47; N, 5.05 **mp** 247-248°C.

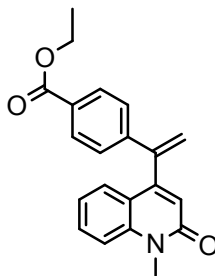




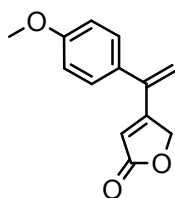
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 7.74 (d, *J* = 8.1 Hz, 2H), 7.40 (d, *J* = 7.4 Hz, 2H), 5.85 (s, 1H), 5.73 (s, 1H), 5.40 (s, 1H), 4.62 (m, 1H), 2.68 (dd, *J* = 16.3, 7.0 Hz, 1H), 2.29 (d, *J* = 7.0 Hz, 1H), 1.30 (d, *J* = 6.8 Hz, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 163.76, 156.73, 144.12, 141.47, 133.29, 128.90, 118.58, 115.45, 112.82, 110.37, 72.87, 31.07, 21.07. **Anal. Calcd.** For C<sub>15</sub>H<sub>13</sub>NO<sub>2</sub>: C, 75.30; H, 5.48; N, 5.85. **Found:** C, 75.33; H, 5.54; N, 5.81. **mp** 238-240°C.



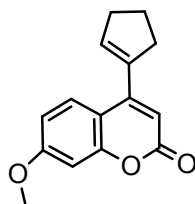
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 7.31 (d, *J* = 7.3 Hz, 2H), 6.47 (d, *J* = 7.3 Hz, 2H), 6.20-6.16 (m, 2H), 5.81 (s, 1H), 5.57 (s, 1H), 5.21 (s, 2H), 2.23 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 166.28, 162.82, 151.27, 147.45, 144.19, 134.55, 128.20, 114.61, 113.30, 109.90, 104.14, 21.87. **Anal. Calcd.** For C<sub>14</sub>H<sub>13</sub>NO<sub>2</sub>: C, 74.00; H, 5.77; N, 6.16. **Found:** C, 74.09; H, 5.72; N, 6.11. **mp** 220-222°C



**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 8.18 (dd, *J* = 8.1, 2.7 Hz, 1H), 7.71 (d, *J* = 7.3 Hz, 2H), 7.52-7.44 (m, 3H), 7.08-7.02 (m, 2H), 6.41 (s, 1H), 5.69 (s, 1H), 5.24 (s, 1H), 4.08 (q, *J* = 8.1 Hz, 2H), 3.68 (s, 3H), 1.34 (t, *J* = 7.7 Hz, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 162.47, 159.81, 145.66, 143.96, 142.53, 139.61, 131.72, 129.37, 128.78, 125.74, 124.47, 123.19, 122.36, 117.31, 115.67, 113.87, 61.27, 29.72, 14.36. **Anal. Calcd.** For C<sub>21</sub>H<sub>19</sub>NO<sub>3</sub>: C, 75.66; H, 5.74; N, 4.20. **Found:** C, 75.60; H, 5.79; N, 4.29. **mp** 215-217°C.

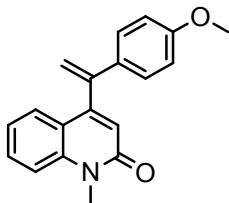


**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 7.37 (d, *J* = 7.8 Hz, 2H), 6.93 (d, *J* = 7.6 Hz, 2H), 6.07 (s, 1H), 5.71 (s, 1H), 5.40 (s, 1H), 4.87 (s, 2H), 3.74 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 176.72, 157.18, 149.89, 136.27, 135.74, 128.24, 119.05, 113.92, 110.54, 72.37, 55.61. **Anal. Calcd.** For C<sub>13</sub>H<sub>12</sub>O<sub>3</sub>: C, 72.21; H, 5.59. **Found:** C, 72.19; H, 5.60. **mp** 212-214°C.

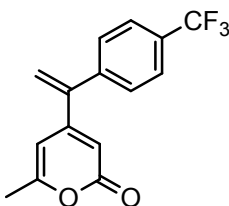


**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 7.71 (d, *J* = 7.6 Hz, 1H), 7.09 (d, *J* = 7.7 Hz, 1H), 6.94 (s, 1H), 6.48 (s, 1H), 6.07 (t, *J* = 6.3 Hz, 1H), 3.82 (s, 3H), 2.30-2.26 (m, 4H), 1.90-1.83 (m, 2H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 163.87, 159.56, 154.23, 137.51, 136.27, 129.71, 126.86,

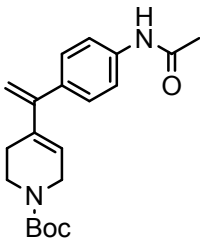
116.41, 110.33, 108.53, 105.81, 54.22, 34.67, 32.63, 22.83. **Anal. Calcd.** For C<sub>15</sub>H<sub>14</sub>O<sub>3</sub>: C, 74.36; H, 5.82. **Found:** C, 74.19; H, 5.97. **mp** 158-160 °C.



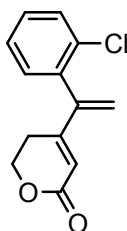
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 7.79 (dd, *J* = 7.6, 2.1 Hz, 1H), 7.48 – 7.36 (m, 3H), 7.06-6.93 (m, 4H), 6.18 (s, 1H), 5.68 (s, 1H), 5.16 (s, 1H), 3.79 (s, 3H), 3.57 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 163.37, 161.65, 144.60, 143.69, 138.62, 136.08, 129.29, 128.53, 124.65, 123.53, 122.72, 117.38, 115.68, 113.95, 111.86, 58.35, 30.59. **Anal. Calcd.** For C<sub>19</sub>H<sub>17</sub>NO<sub>2</sub>: C, 78.33; H, 5.05; N, 5.45. **Found:** C, 78.58; H, 5.09; N, 5.52. **mp** 252-254 °C.



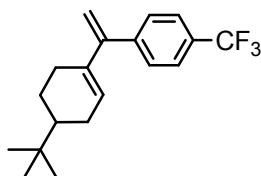
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 7.49 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 7.9 Hz, 2H), 6.31 (s, 1H), 6.19 (s, 1H), 5.59 (s, 1H), 5.45 (s, 1H), 2.31 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 164.70, 161.78, 161.63, 144.17, 141.24, 131.91 (q, *J* = 30.7 Hz), 131.73, 126.90 (q, *J* = 3.9 Hz), 125.16 (d, *J* = 271.14 Hz), 114.46, 109.92, 104.45, 20.19. **Anal. Calcd.** For C<sub>15</sub>H<sub>11</sub>F<sub>3</sub>O<sub>2</sub>: C, 64.29; H, 3.96. **Found:** C, 64.37; H, 3.87. **mp** 198-200 °C.



**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 9.24 (s, 1H), 7.75 (d, *J* = 7.9 Hz, 2H), 7.64 (d, *J* = 7.6 Hz, 2H), 5.79 (t, *J* = 6.2 Hz, 1H), 5.42 (s, 1H), 5.04 (s, 1H), 3.95 (t, *J* = 5.8 Hz, 2H), 3.45 (d, *J* = 6.2 Hz, 2H), 2.65 (t, *J* = 6.0 Hz, 2H), 2.07 (s, 3H), 1.42 (s, 9H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 167.19, 158.79, 144.97, 141.53, 138.13, 136.54, 127.68, 124.52, 118.66, 118.19, 79.30, 49.28, 45.63, 31.27, 27.51, 24.05. **Anal. Calcd.** For C<sub>20</sub>H<sub>26</sub>N<sub>2</sub>O<sub>3</sub>: C, 70.15; H, 7.65; N, 8.18. **Found:** C, 70.24; H, 7.71; N, 8.12. **mp** 237-239 °C.



**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 7.51 (dd, *J* = 7.7, 2.4 Hz, 1H), 7.17-7.07 (m, 3H), 5.97 (s, 1H), 5.59 (s, 1H), 5.38 (s, 1H), 4.27 (t, *J* = 5.1 Hz, 2H), 2.24 (t, *J* = 5.3 Hz, 2H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 165.67, 154.72, 144.67, 138.91, 133.67, 130.27, 129.52, 129.37, 125.49, 116.32, 112.32, 64.81, 29.37. **Anal. Calcd.** For C<sub>13</sub>H<sub>11</sub>ClO<sub>2</sub>: C, 66.53; H, 4.72. **Found:** C, 66.57; H, 4.69. **mp** 208-211 °C.



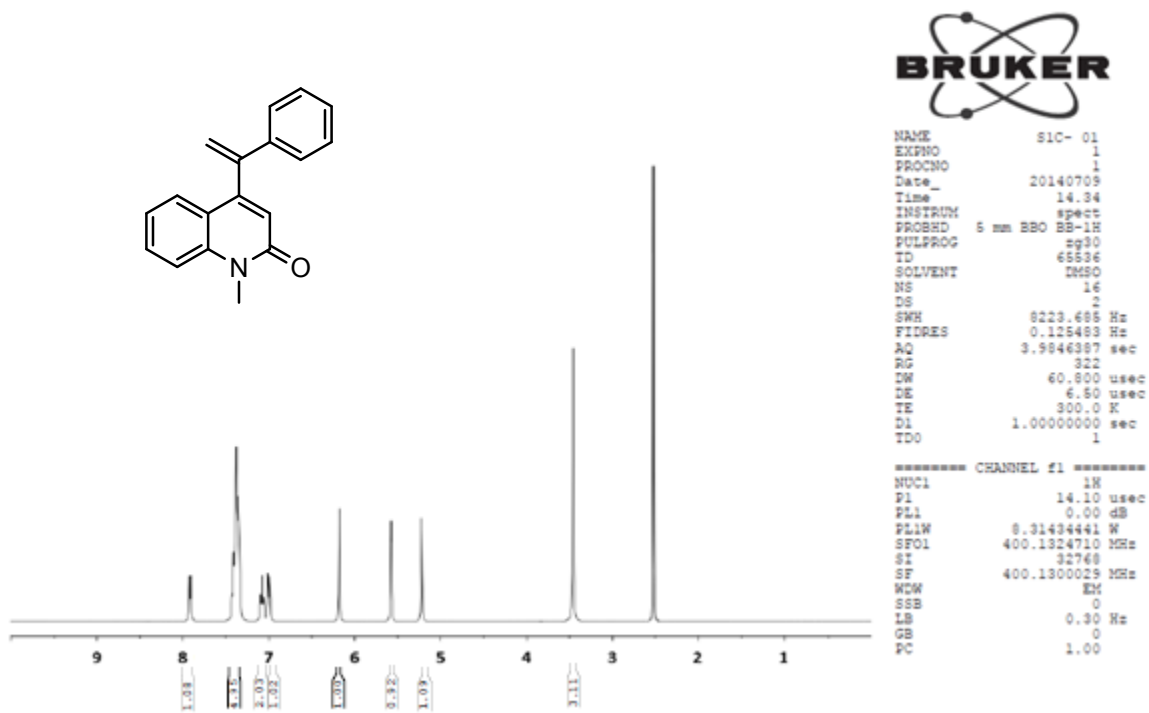
**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ ppm : 7.48 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 5.63 (t, *J* = 6.2 Hz, 1H), 5.43 (s, 1H), 5.11 (s, 1H), 2.31-2.23 (m, 1H), 2.20–2.02 (m, 2H), 1.81–1.66 (m, 2H), 1.39-1.35 (m, 2H), 0.84 (s, 9H). **<sup>13</sup>C NMR** (100 MHz, DMSO-*d*<sub>6</sub>) δ ppm: 145.07, 141.18, 139.17, 134.21, 131.39 (q, *J* = 32.2 Hz), 128.31 (d, *J* = 3.4 Hz), 123.17 (d, *J* = 269.7 Hz), 119.90, 118.19, 44.27, 32.36, 28.89, 27.10, 25.13, 24.95. **Anal. Calcd.** For C<sub>19</sub>H<sub>23</sub>F<sub>3</sub>: C, 74.01; H, 7.52. **Found:** C, 74.07; H, 7.58. **mp** 217-220 °C.

## References

1. J. Kuroda, K. Inamoto, K. Hiroya, T. Doi, *Eur. J. Org. Chem.* 2009, 2251-2261.
2. A. Klapars, K. R. Campos, C. Y. Chen, R. P. Volante. *Org. Lett.* 2005, **7**, 1185-1188.
3. Creary, X.; Tam, W. W.; Albizati, K. F.; Stevens, R. V. *Org. Synth.* 1986, **64**, 207.s

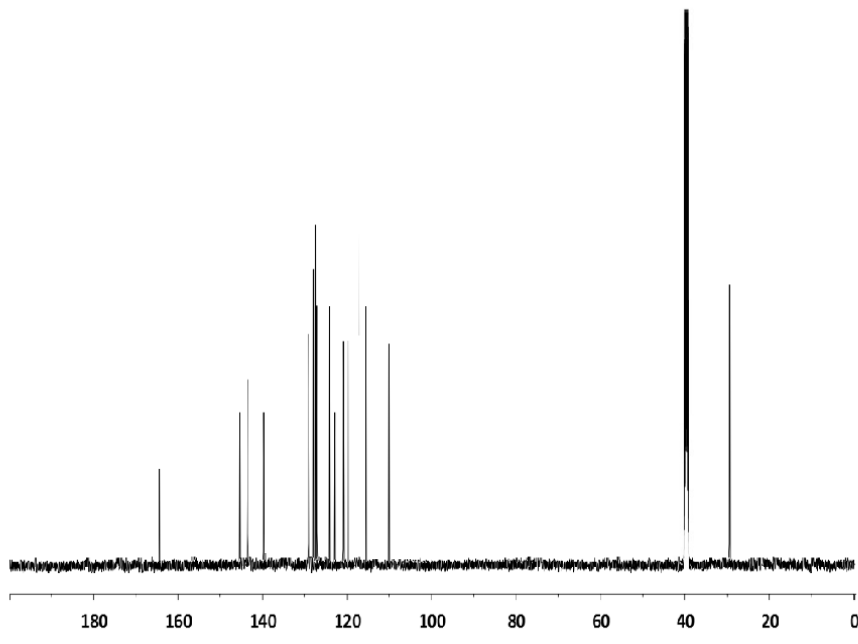
## 7. $^1\text{H}$ and $^{13}\text{C}$ NMR Spectra

Compound 3a





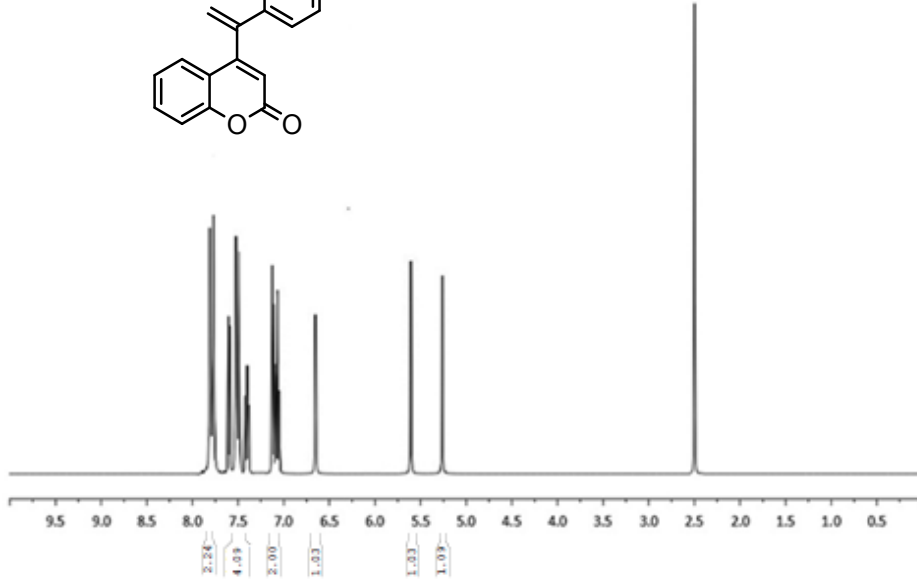
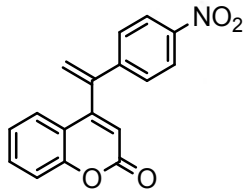
NAME SIC-01  
EXPNO 2  
PROCNO 1  
Date\_ 20140701  
Time 11.41  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT DMSO  
NS 249  
DS 4  
SMH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 2050  
DW 20.800 usec  
DE 6.50 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TD0 1



==== CHANNEL f1 =====  
NUC1 13C  
P1 10.00 usec  
PL1 0.00 dB  
PL1W 35.41759872 W  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 0.00 dB  
PL12 15.08 dB  
PL13 18.08 dB  
PL2W 8.31434441 W  
PL12W 0.25812379 W  
PL13W 0.12936834 W  
SFO2 400.1316005 MHz  
SI 32768  
SF 100.6128193 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

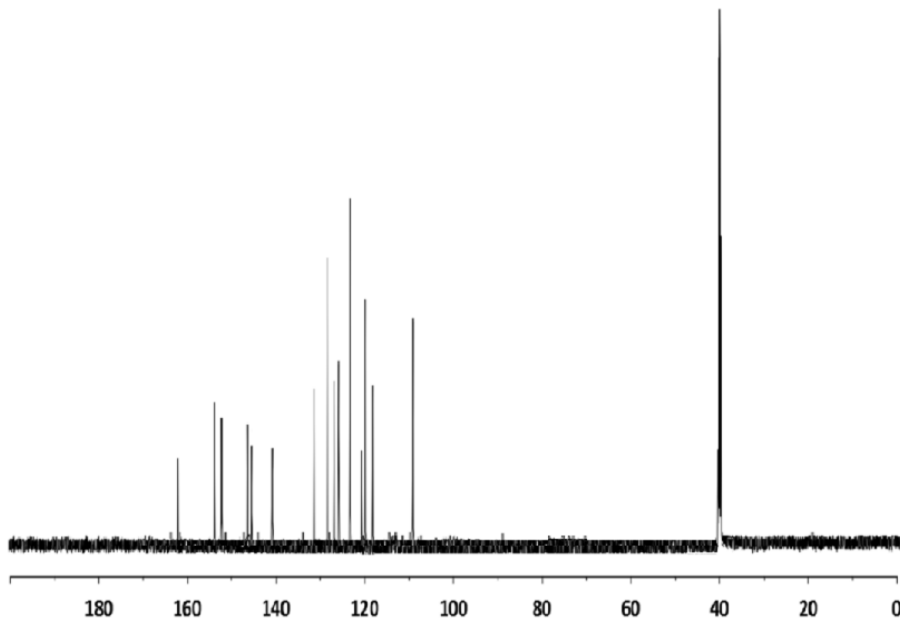
Compound 3b



```

NAME          SIC-02
EXPNO         1
PROCNO        1
Date_         20140709
Time          10.29
INSTRUM       spect
PROBHD        5 mm BBO BB-1K
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8222.688 Hz
FIDRES        0.125482 Hz
AQ            3.9846387 sec
RG            322
DW            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1

CHANNEL f1
NUC1          1H
P1            14.10 usec
PL1           0.00 dB
PL1W          8.31434441 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300029 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



```

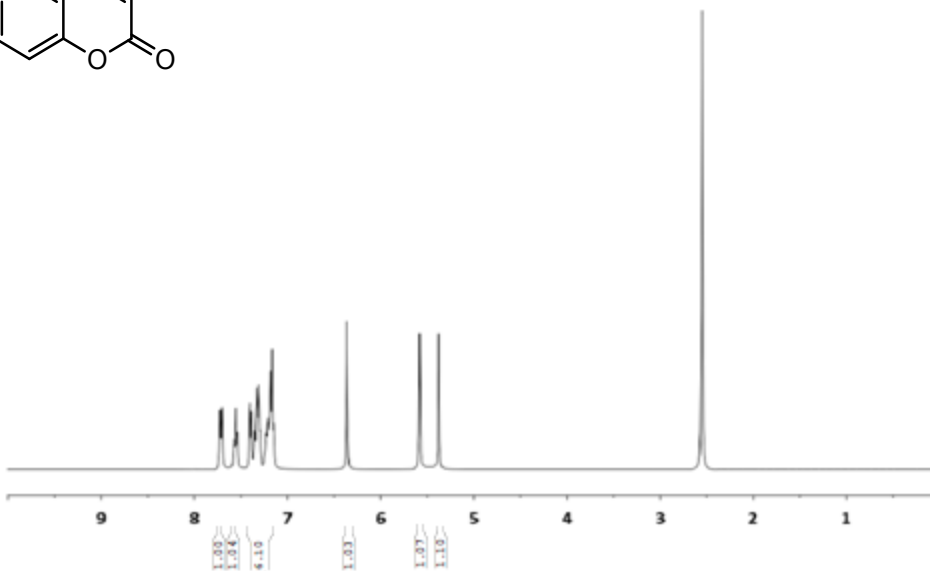
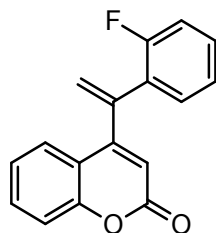
NAME          SIC-02
EXPNO         3
PROCNO        1
Date_         20140702
Time          13.29
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
PL1W          35.41759872 W
SFO1          100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W          8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



Compound 3c

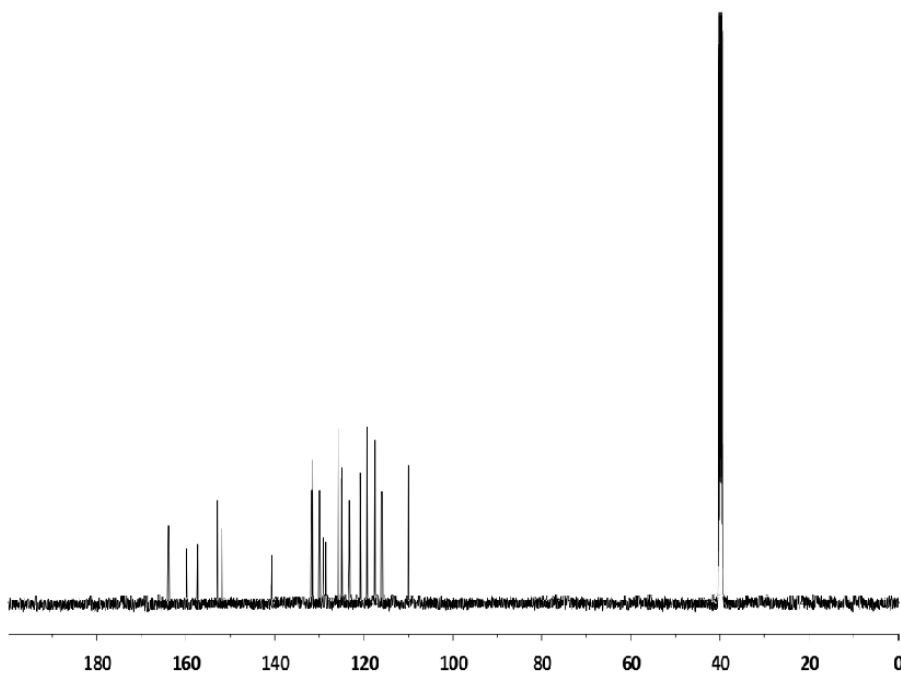


```

NAME          SIC- 03
EXPNO         1
PROCNO        1
Date_         20140708
Time_         09.15
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DM            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TDO           1
    
```

```

===== CHANNEL f1 =====
NUC1           1H
P1             14.10 usec
PL1            0.00 dB
PL1W           8.31434441 W
SFO1           400.1324710 MHz
SI             32768
SF            400.1300029 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
    
```



```

NAME          SIC-03
EXPNO         2
PROCNO        1
Date_         20140702
Time_         08.58
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DM            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
    
```

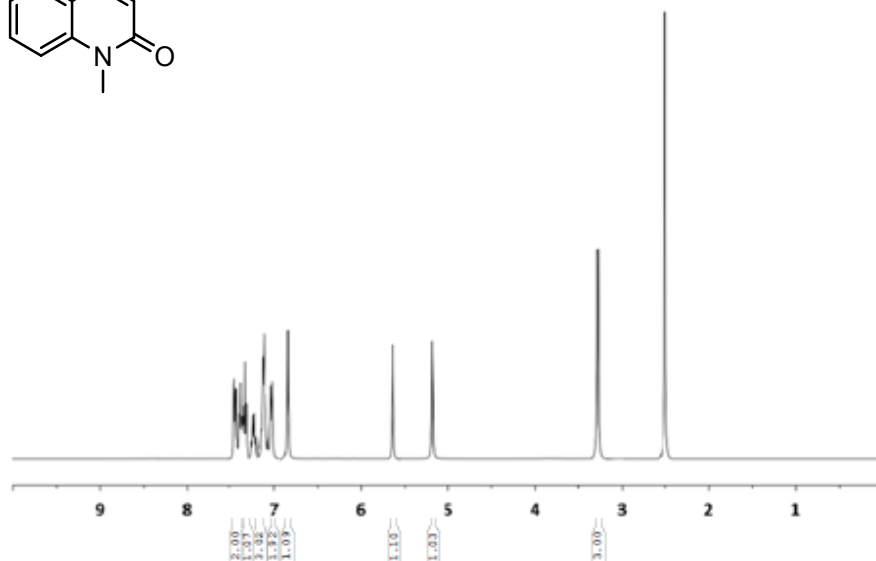
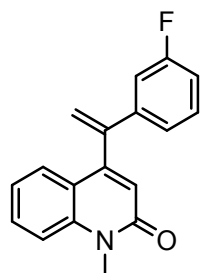
```

===== CHANNEL f1 =====
NUC1           13C
P1             10.00 usec
PL1            0.00 dB
PL1W           35.41759872 W
SFO1           100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2            0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W           8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SFO2           400.1316005 MHz
SI             32768
SF            100.6128193 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
    
```

Compound 3d

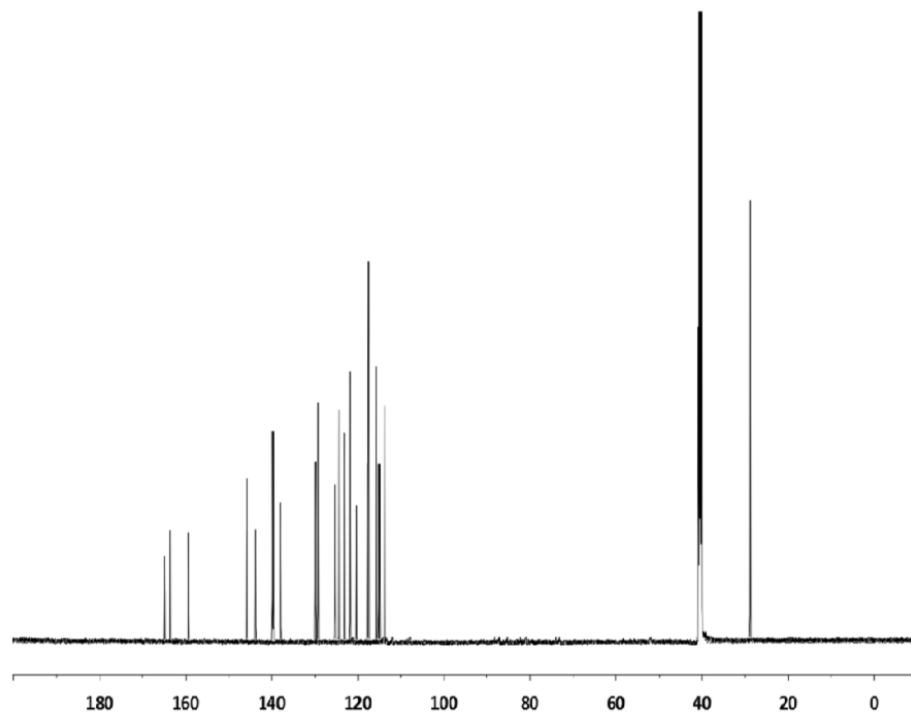


```

NAME          SIC- 04
EXPNO         1
PROCNO        1
Date_         20140708
Time_         19.21
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SMH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DW            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TDO           1
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.10 usec
PL1           0.00 dB
PL1W          8.31434441 W
SF01          400.1324710 MHz
SI            32768
SF            400.1300029 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



```

NAME          SIC-04
EXPNO         2
PROCNO        1
Date_         20140702
Time_         13.31
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SMH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
    
```

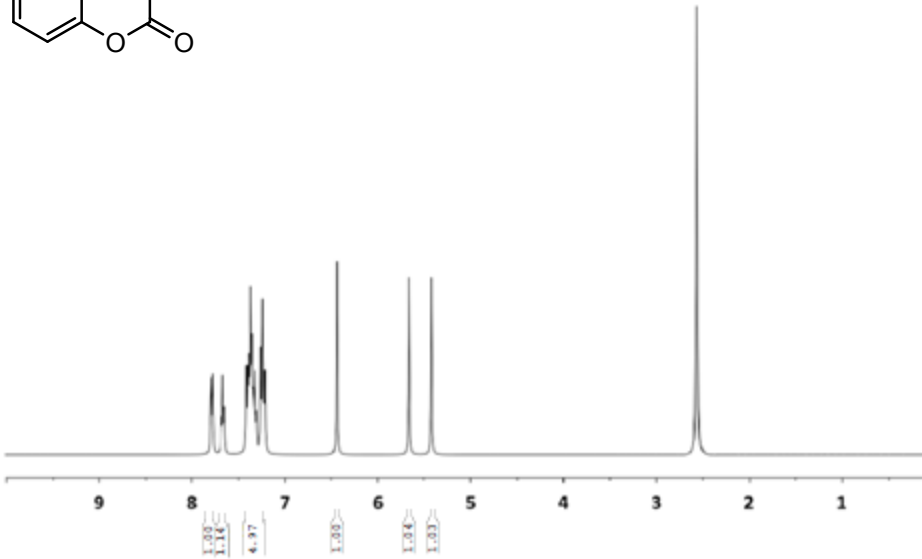
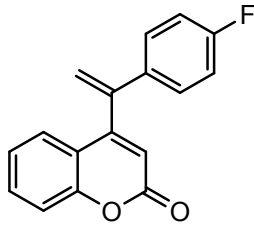
```

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
PL1W          35.41759872 W
SF01          100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W          8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SF02          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

Compound 3e

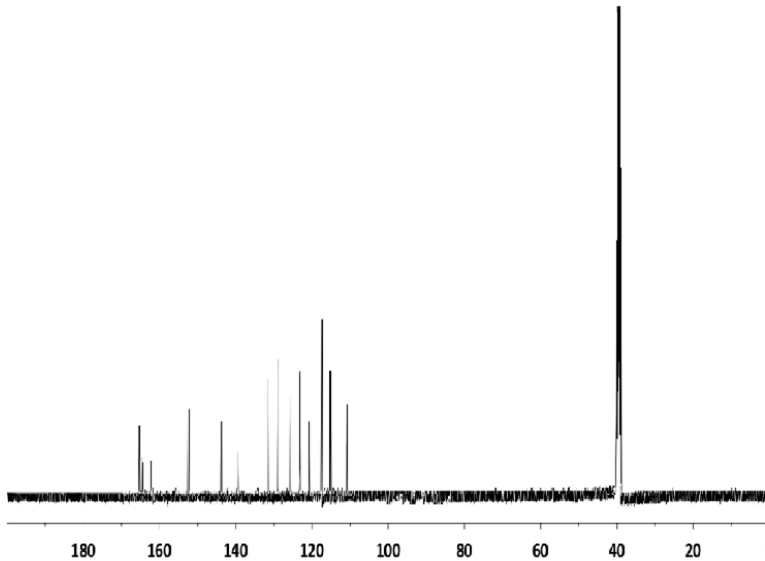


```

NAME      SIC- 05
EXPNO     1
PROCNO    1
Date_     20140708
Time      11.12
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         16
DS         2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ         3.9846387 sec
RG         322
DW         60.800 usec
DE         6.50 usec
TE         300.0 K
D1         1.00000000 sec
TDO        1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.10 usec
PL1       0.00 dB
PL1W      8.31434441 W
SFO1      400.1324710 MHz
SI         32768
SF         400.1300029 MHz
WDW       EM
SSB       0
LB         0.30 Hz
GB         0
PC         1.00
    
```



```

NAME      SIC-05
EXPNO     2
PROCNO    1
Date_     20140701
Time      12.36
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         249
DS         4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631908 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO        1
    
```

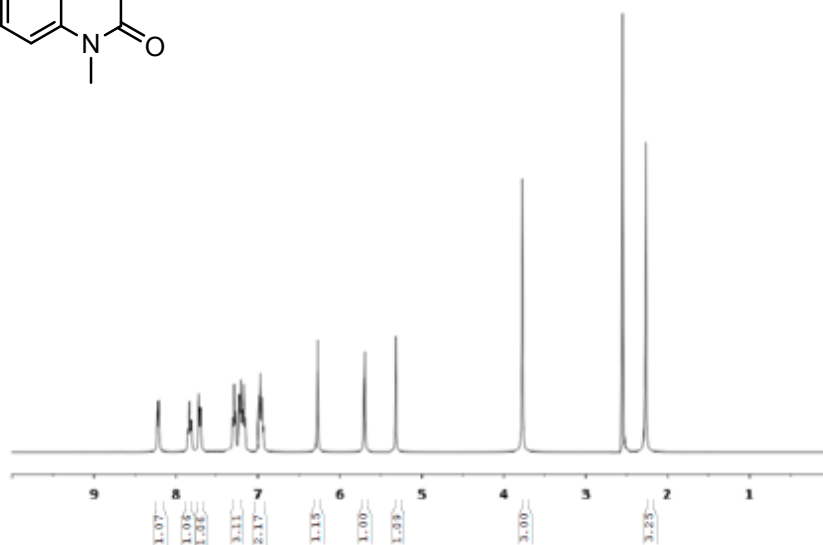
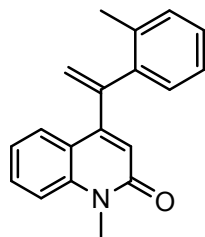
```

===== CHANNEL f1 =====
NUC1      13C
P1        10.00 usec
PL1       0.00 dB
PL1W      35.41759872 W
SFO1      100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       0.00 dB
PL12      15.08 dB
PL13      18.08 dB
PL2W      8.31434441 W
PL12W     0.25812379 W
PL13W     0.12936834 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6128193 MHz
WDW       EM
SSB       0
LB         1.00 Hz
GB         0
PC         1.40
    
```

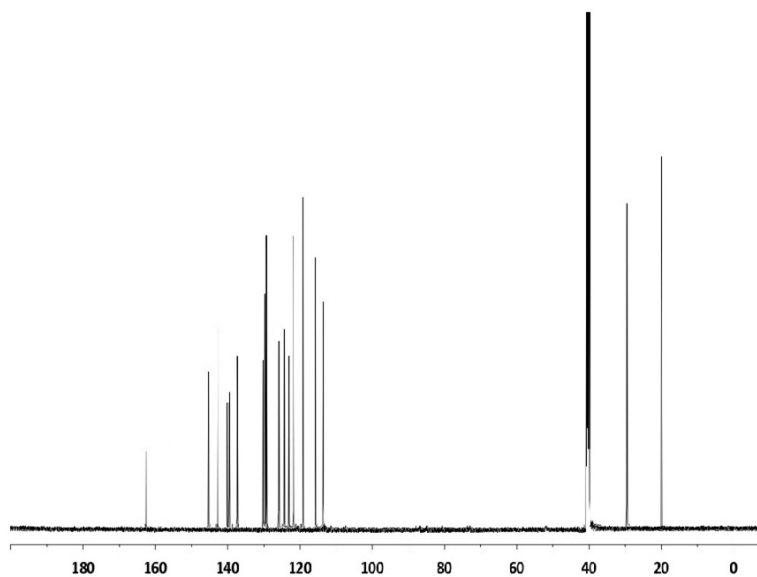
Compound 3f



```

NAME          SIC- 06
EXPNO         1
PROCNO        1
Date_         20140708
Time          11.07
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 MHz
FIDRES        0.123483 MHz
AQ            3.9846387 sec
RG            322
CW            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TDO           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.10 usec
PL1           0.00 dB
PL1W          8.31434441 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300029 MHz
WDW           EM
SSB           0
LB            0.30 MHz
GB            0
PC            1.00
    
```



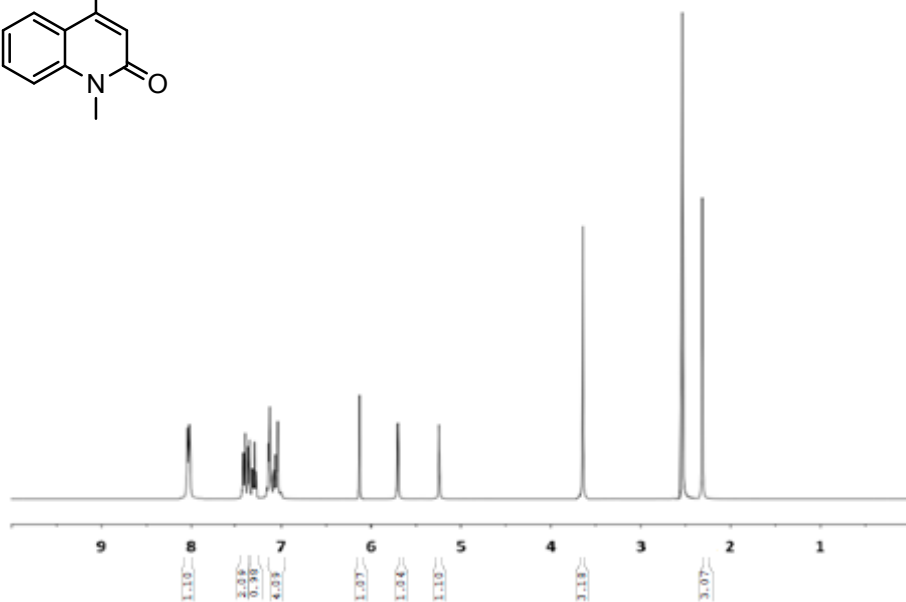
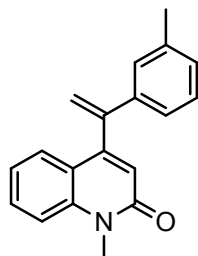
```

NAME          SIC-06
EXPNO         2
PROCNO        1
Date_         20140701
Time          07.14
INSTRUM       spect
PROBHD        5 mm BBO BS-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
CW            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
PL1W          35.41758872 W
SFO1          100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W          8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 MHz
GB            0
PC            1.40
    
```

Compound 3g

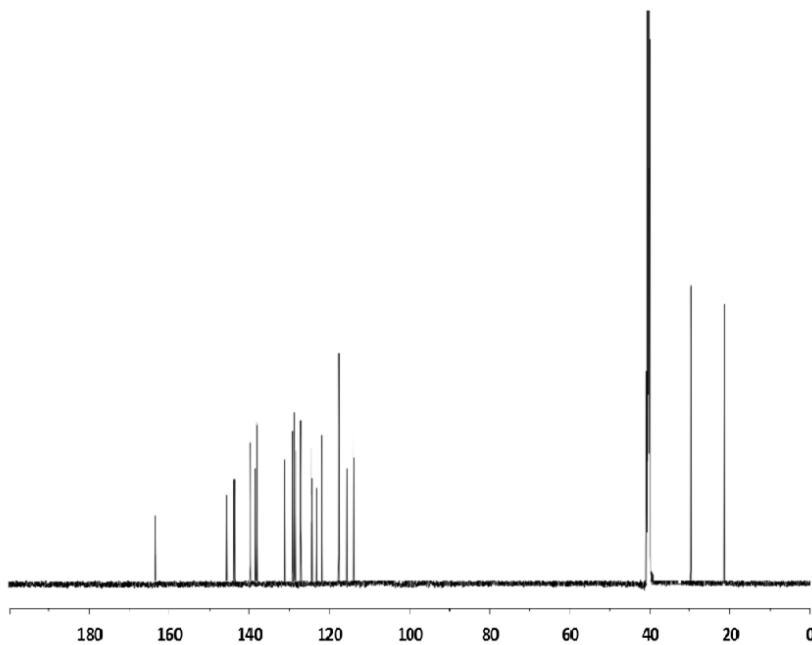


```

NAME          sic- 07
EXPNO         1
PROCNO        1
Date_         20140708
Time_         14.19
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DW            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TDO           1
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.10 usec
PL1           0.00 dB
PL1W          8.31434441 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300029 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



```

NAME          sic-07
EXPNO         2
PROCNO        1
Date_         20140702
Time_         12.31
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
    
```

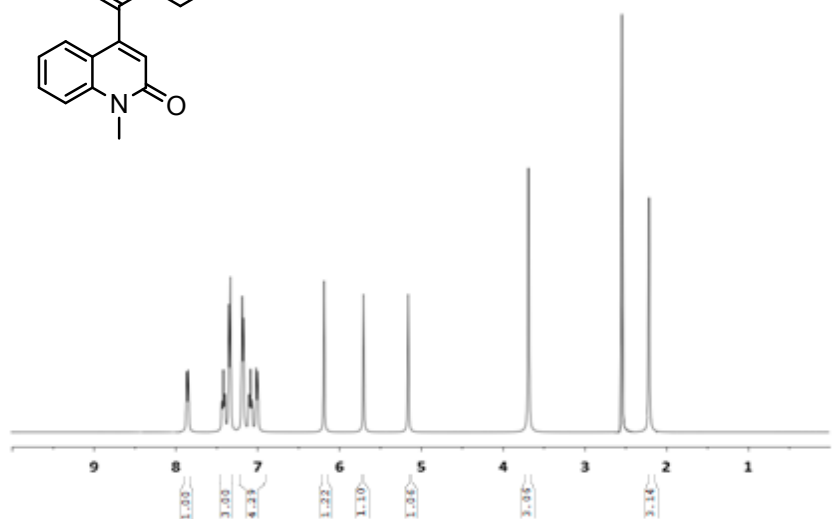
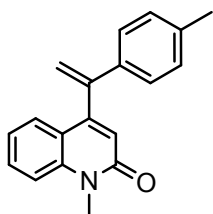
```

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
PL1W          35.41759872 W
SFO1          100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W          8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

Compound 3h



```

NAME      SIC- 08
EXPNO     1
PROCNO    1
Date_     20140709
Time      14.27
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         322
DW         60.800 usec
DE         6.50 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.10 usec
PL1       0.00 dB
PL1W      8.31434441 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300029 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



```

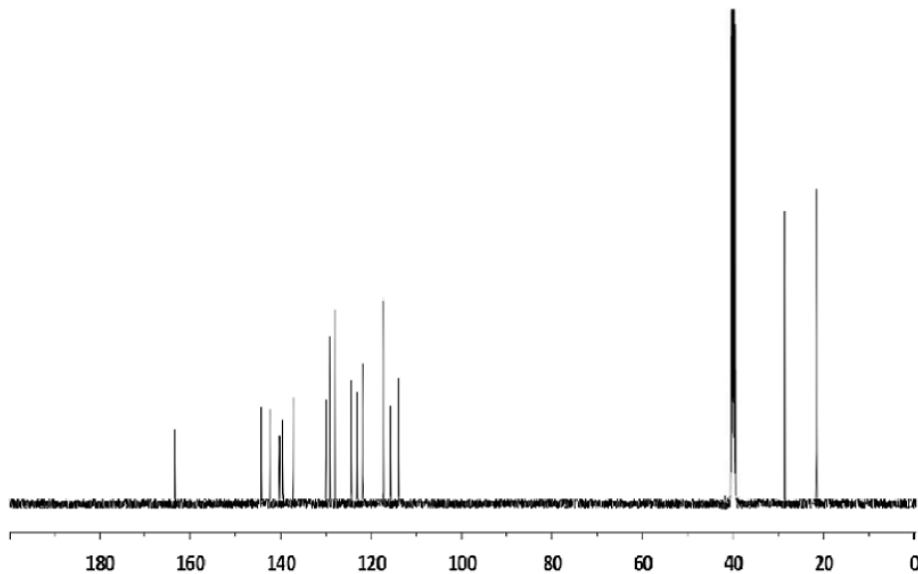
NAME      SIC-08
EXPNO     2
PROCNO    1
Date_     20140702
Time      13.27
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         249
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.300 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

```

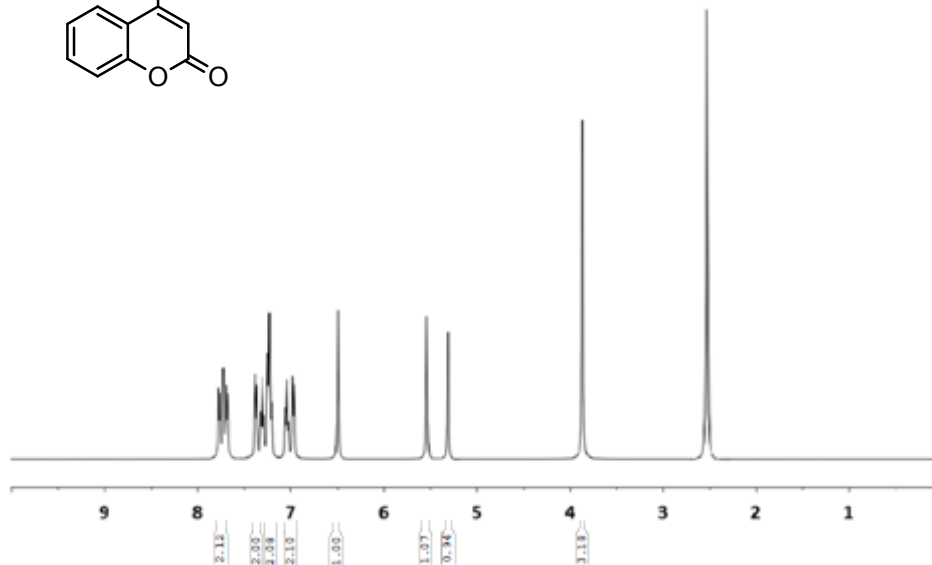
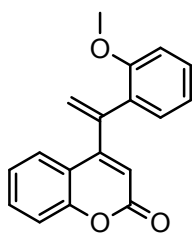
===== CHANNEL f1 =====
NUC1      13C
P1        10.00 usec
PL1       0.00 dB
PL1W      35.41759872 W
SFO1      100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CEDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       0.00 dB
PL12      15.08 dB
PL13      18.08 dB
PL2W      8.31434441 W
PL12W     0.25812379 W
PL13W     0.12936834 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6128193 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```



Compound 3i

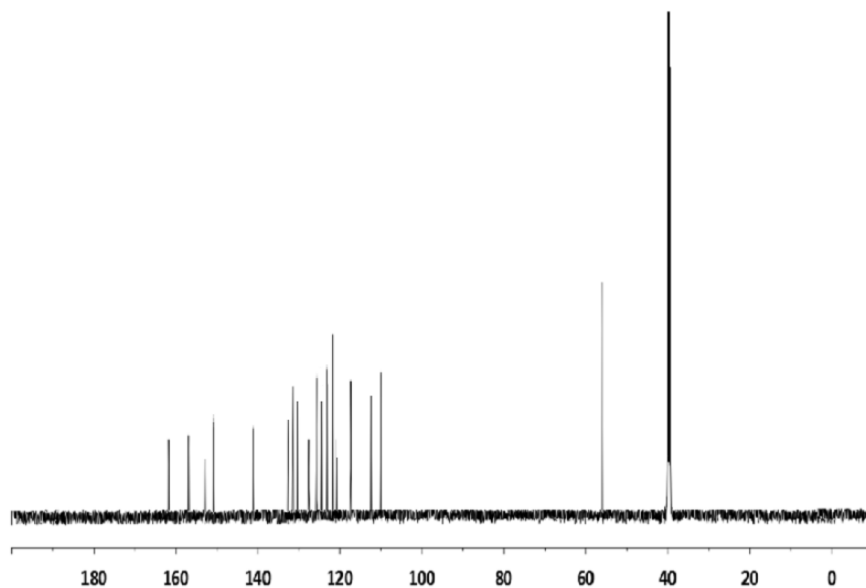


```

NAME          SIC- 09
EXPNO         1
PROCNO        1
Date_         20140702
Time          15.41
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DW            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.10 usec
PL1           0.00 dB
PL1W          8.31434441 W
SF01          400.1324710 MHz
SI            32768
SF            400.1300029 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



```

NAME          SIC-09
EXPNO         2
PROCNO        1
Date_         20140702
Time          09.37
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
    
```

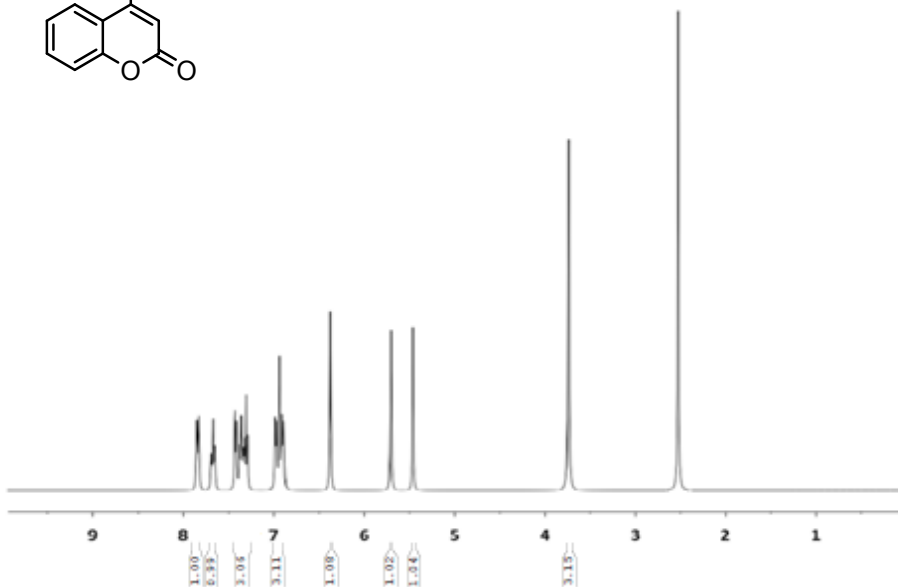
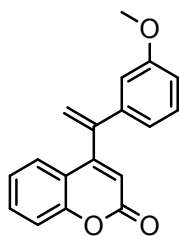
```

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
PL1W          35.41759872 W
SF01          100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W          8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SF02          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

Compound 3j

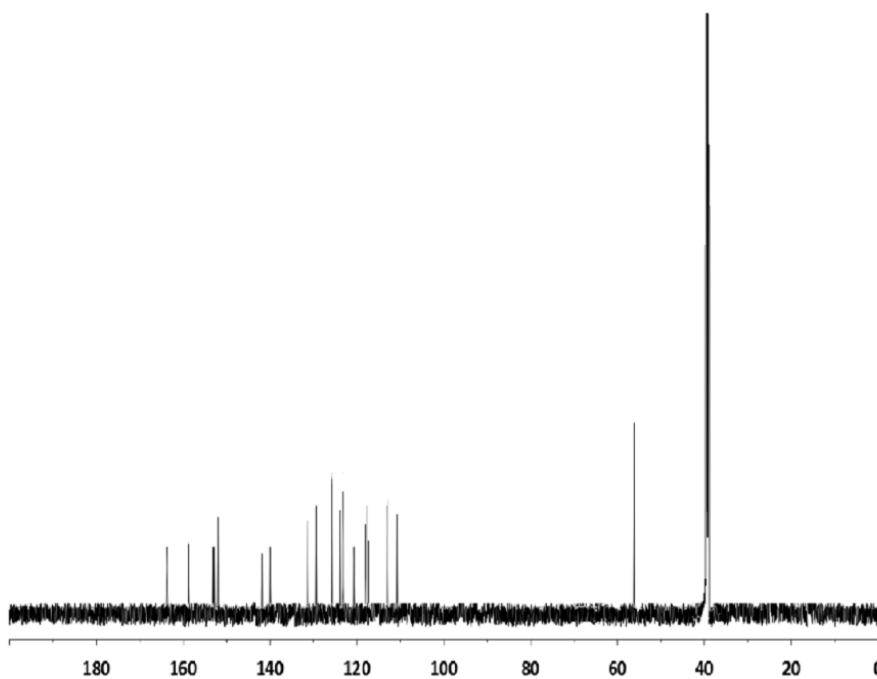


```

NAME          SIC- 10
EXPNO         1
PROCNO        1
Date_         20140708
Time_         19.49
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DM            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1           1H
P1             14.10 usec
PL1            0.00 dB
PL1W           8.31434441 W
SFO1           400.1324710 MHz
SI             32768
SF             400.1300029 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
    
```



```

NAME          SIC-10
EXPNO         2
PROCNO        1
Date_         20140702
Time_         20.38
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DM            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
    
```

```

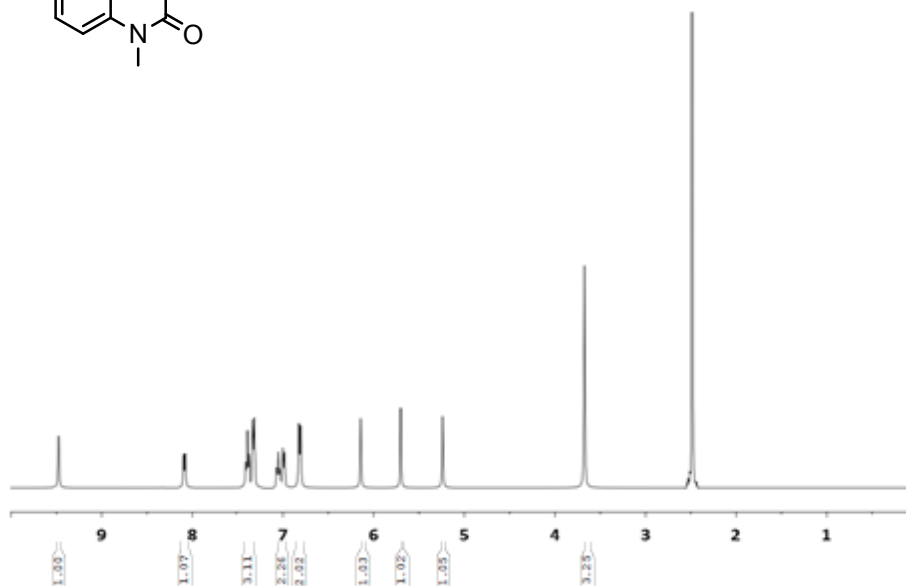
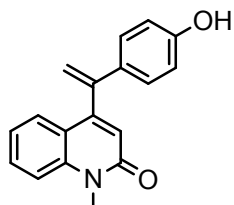
===== CHANNEL f1 =====
NUC1           13C
P1             10.00 usec
PL1            0.00 dB
PL1W           35.41759872 W
SFO1           100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2            0.00 dB
PL12           15.08 dB
PL13           18.08 dB
PL2W           8.31434441 W
PL12W          0.25812379 W
PL13W          0.12936834 W
SFO2           400.1316005 MHz
SI             32768
SF             100.6128193 MHz
WDW            BM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
    
```



Compound 3k

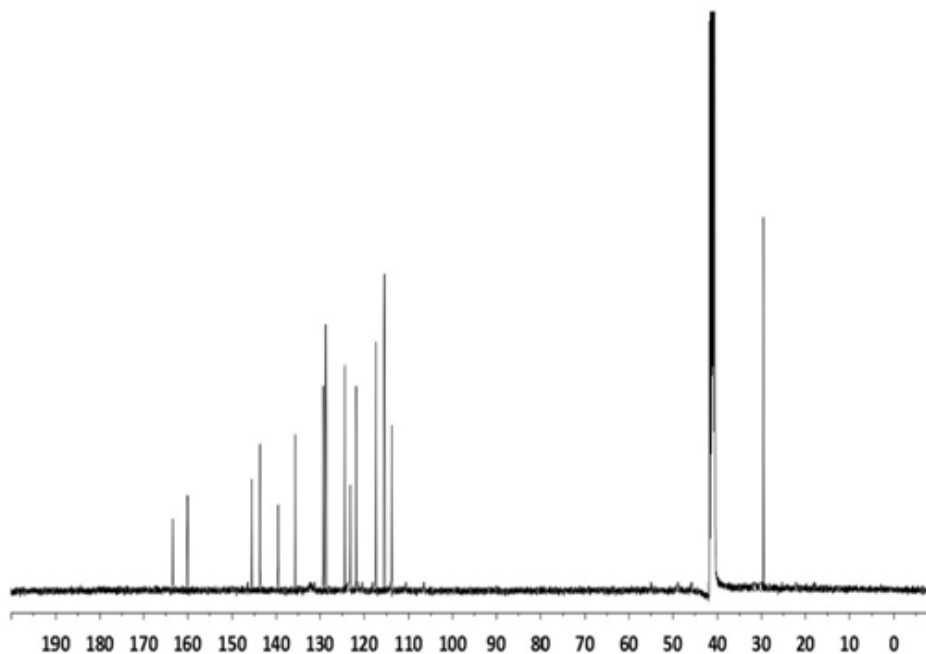


```

NAME      SIC- 12
EXPNO     1
PROCNO    1
Date_     20140708
Time      15.09
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         322
DM         60.800 usec
DE         6.50 usec
TE         300.0 K
D1         1.00000000 sec
TDO        1
    
```

```

----- CHANNEL f1 -----
NUC1      1H
P1         14.10 usec
PL1        0.00 dB
PL1W       8.31434441 W
SFO1      400.1324710 MHz
SI         32768
SF         400.1300029 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



```

NAME      SIC-12
EXPNO     2
PROCNO    1
Date_     20140701
Time      15.27
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         249
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         2050
DM         20.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO        1
    
```

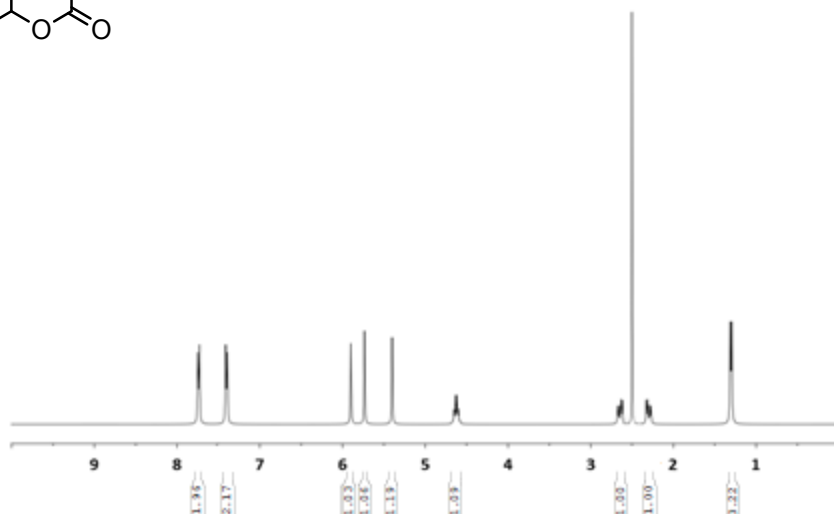
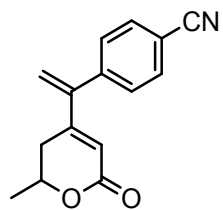
```

----- CHANNEL f1 -----
NUC1      13C
P1         10.00 usec
PL1        0.00 dB
PL1W       35.41759872 W
SFO1      100.6228298 MHz
    
```

```

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        0.00 dB
PL12       15.08 dB
PL13       18.08 dB
PL1W       8.31434441 W
PL12W      0.25812379 W
PL13W      0.12936834 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6128193 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

Compound 3I

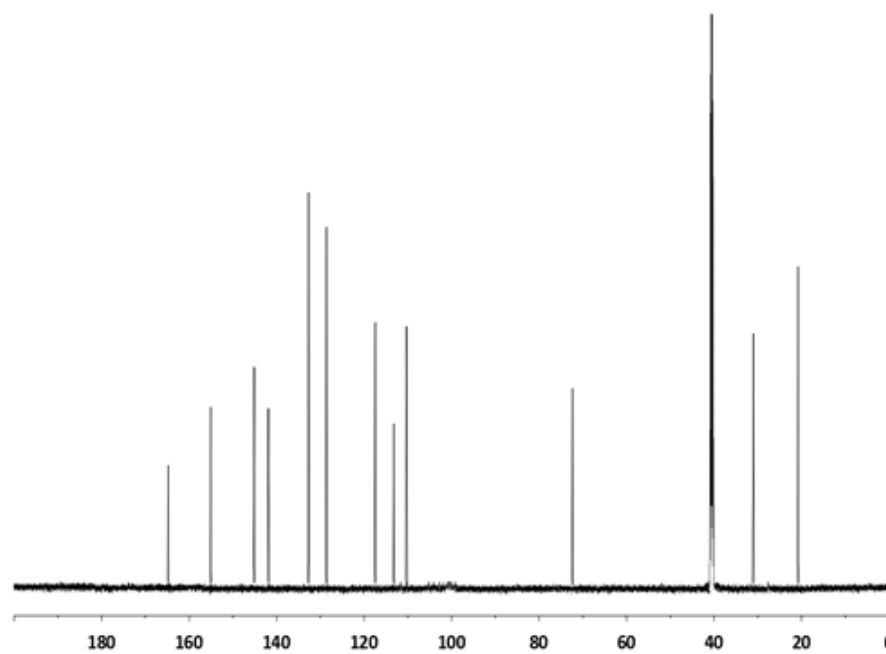


```

NAME      SIC- 13
EXPNO    1
PROCNO   1
Date_    20140708
Time     09.23
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       16
DS       2
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       322
DS       60.800 usec
DE       6.50 usec
TE       300.0 K
D1       1.00000000 sec
TDO      1
    
```

```

***** CHANNEL f1 *****
NUC1     1H
P1       14.10 usec
PL1      0.00 dB
PL1W     8.31434441 W
SFO1     400.1324710 MHz
SI       32768
SF       400.1300029 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



```

NAME      SIC-13
EXPNO    2
PROCNO   1
Date_    20140701
Time     12.08
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zppq30
TD       65536
SOLVENT  DMSO
NS       249
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       2050
DS       20.800 usec
DE       6.50 usec
TE       300.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1
    
```

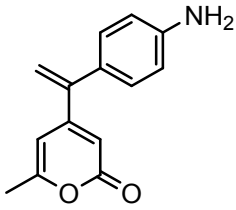
```

----- CHANNEL f1 -----
NUC1     13C
P1       10.00 usec
PL1      0.00 dB
PL1W     35.41759872 W
SFO1     100.6228298 MHz
    
```

```

----- CHANNEL f2 -----
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     15.08 dB
PL13     18.08 dB
PL2W     8.31434441 W
PL12W    0.25812379 W
PL13W    0.12936824 W
SFO2     400.1316005 MHz
SI       32768
SF       100.6128192 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

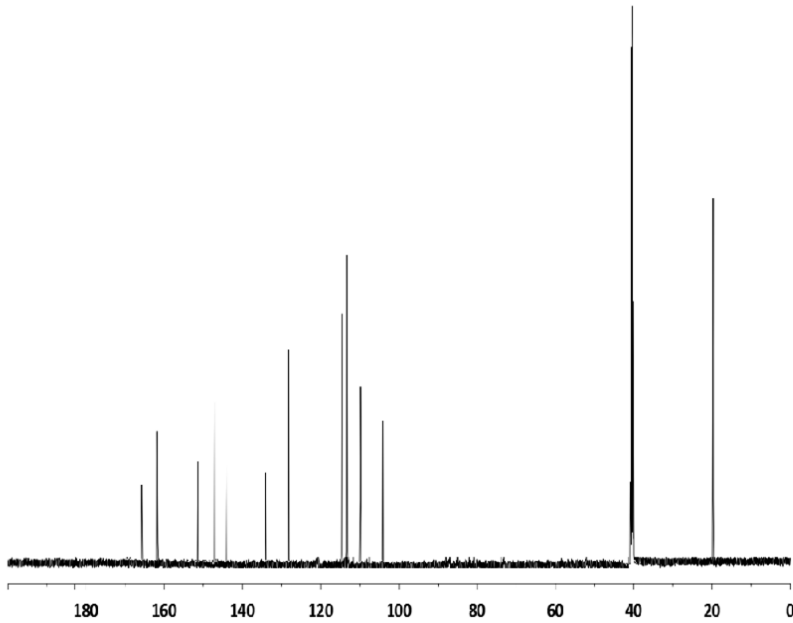
Compound 3m



```

NAME          S1C- 14
EXPNO         1
PROCNO        1
Date_         20140708
Time          15.39
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DM            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.10 usec
PL1           0.00 dB
PL1W          8.31434441 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300029 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



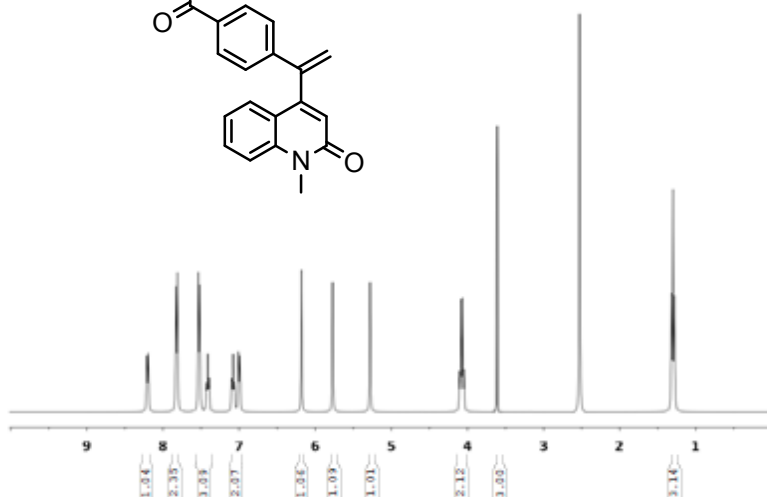
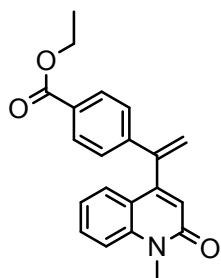
```

NAME          S1C-14
EXPNO         3
PROCNO        1
Date_         20140702
Time          13.10
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            959
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DM            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
PL1W          35.41759872 W
SFO1          100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W          8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

Compound 3n



```

NAME          Slc- 15
EXPNO         1
PROCNO        1
Date_         20140709
Time_         11.19
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DW            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1

***** CHANNEL f1 *****
NUC1           1H
P1             14.10 usec
PL1            0.00 dB
PL1W           8.31434441 W
SFO1           400.1324710 MHz
SI             32768
SF             400.1300029 MHz
WCFW           EM
SSB            0
LB             0.30 Hz
GB            0
PC             1.00
    
```

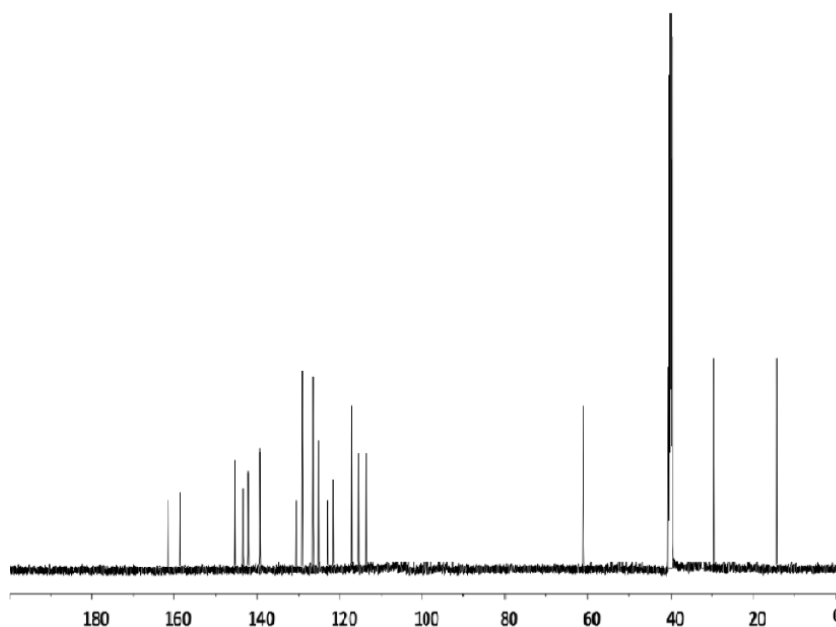


```

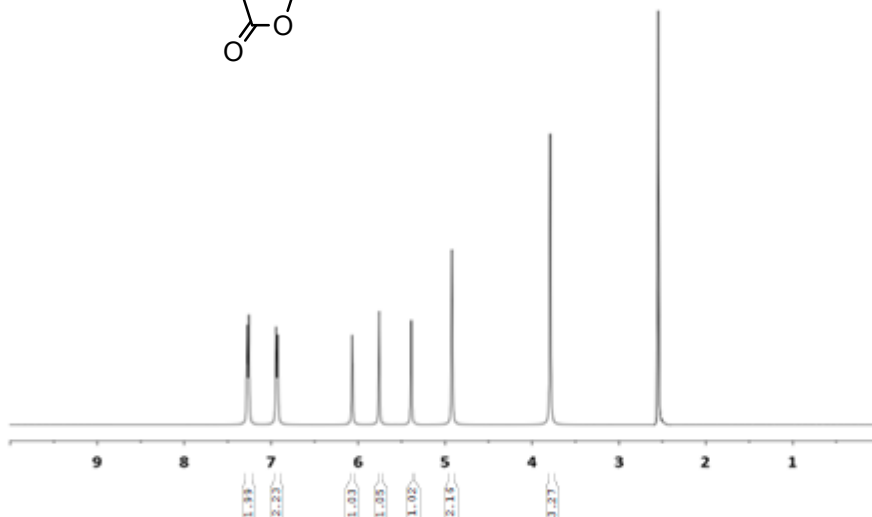
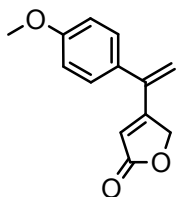
NAME          Slc-15
EXPNO         3
PROCNO        1
Date_         20140702
Time_         14.12
INSTRUM       spelct
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            959
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

***** CHANNEL f1 *****
NUC1           13C
P1             10.00 usec
PL1            0.00 dB
PL1W           35.41759872 W
SFO1           100.6228290 MHz

***** CHANNEL f2 *****
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2            0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W           8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SFO2           400.1316005 MHz
SI             32768
SF             100.6128193 MHz
WCFW           BM
SSB            0
LB             1.00 Hz
GB            0
PC             1.40
    
```



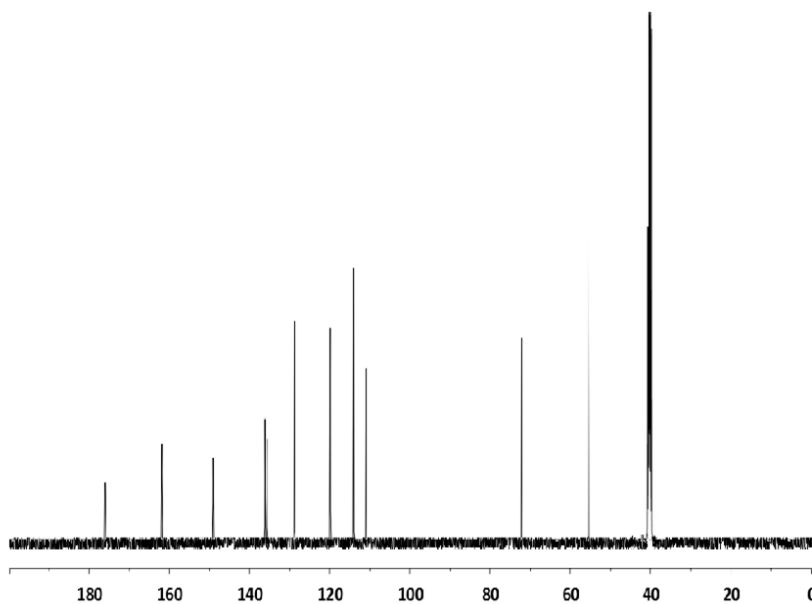
Compound 3o



```

NAME          SIC- 16
EXPNO         1
PROCNO        1
Date_         20140709
Time          10.47
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8229.655 Hz
FIDRES        0.125482 Hz
AQ            2.9846387 sec
RG            322
DW            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.0000000 sec
TD0           1

CHANNEL f1
NUC1          1H
P1            14.10 usec
PL1           0.00 dB
PL1W          8.31434441 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300029 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



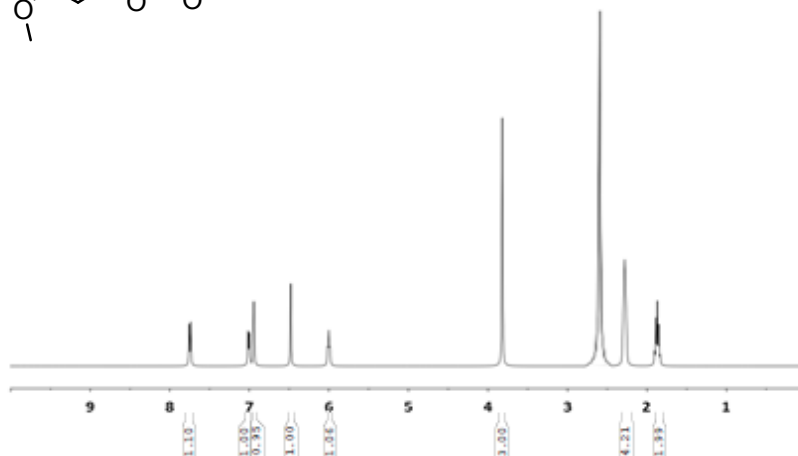
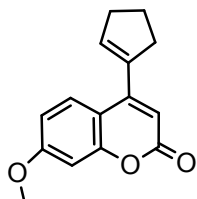
```

NAME          SIC-16
EXPNO         2
PROCNO        1
Date_         20140702
Time          14.02
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
PL1W          35.41759672 W
SFO1          100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W          8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

Compound 3p

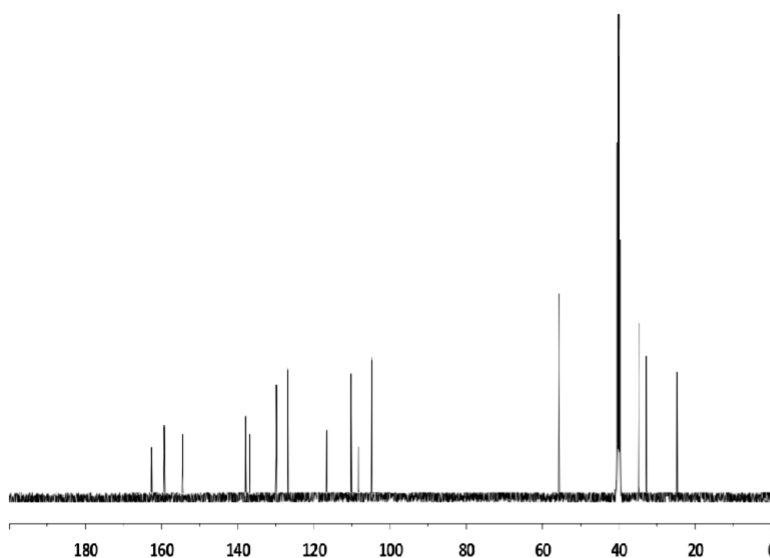


```

NAME          sic-17
EXPNO         1
PROCNO        1
Date_         20140709
Time_         10.07
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DW            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.10 usec
PL1           0.00 dB
PL1W          8.31434441 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300029 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



```

NAME          sic-17
EXPNO         2
PROCNO        1
Date_         20140702
Time_         17.09
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            248
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631888 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
    
```

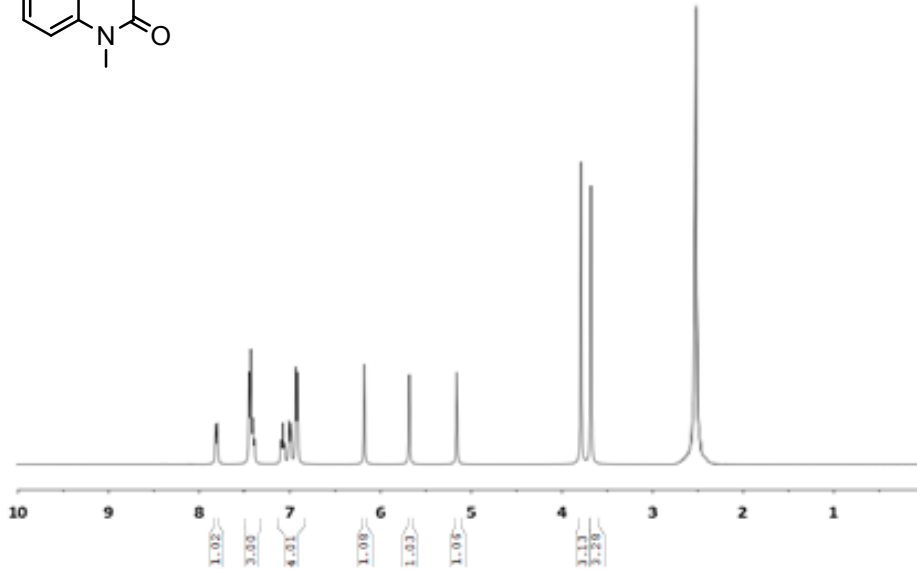
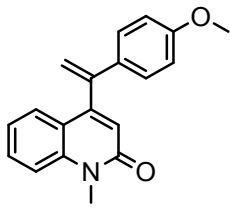
```

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
PL1W          35.41759872 W
SFO1          100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPD PRG2      waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.08 dB
PL13          16.08 dB
PL2W          8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

Compound 3q

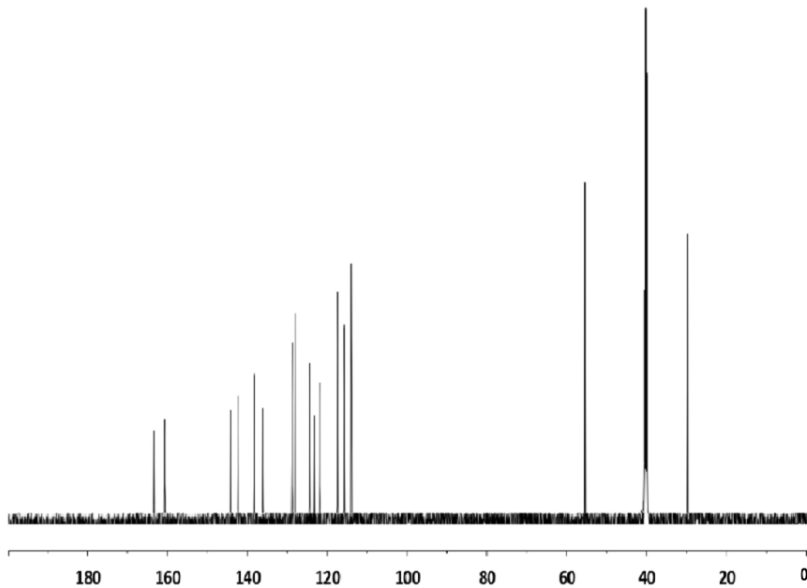


```

NAME      s1c- 18
EXFNO    1
PROCNO   1
Date_    20140708
Time     17.53
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       16
DS       2
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       322
DW       60.800 usec
DE       6.50 usec
TE       300.0 K
D1       1.0000000 sec
TD0      1
    
```

```

===== CHANNEL f1 =====
NUC1     1H
F1       14.10 usec
PL1      0.00 dB
PL1W     8.31434441 W
SFO1     400.1324710 MHz
SI       32768
SF       400.130029 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
FC       1.00
    
```



```

NAME      s1c-18
EXFNO    2
PROCNO   1
Date_    20140702
Time     09.50
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       65536
SOLVENT  DMSO
NS       249
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       2050
DW       20.800 usec
DE       6.50 usec
TE       300.0 K
D1       2.0000000 sec
D11      0.0300000 sec
TD0      1
    
```

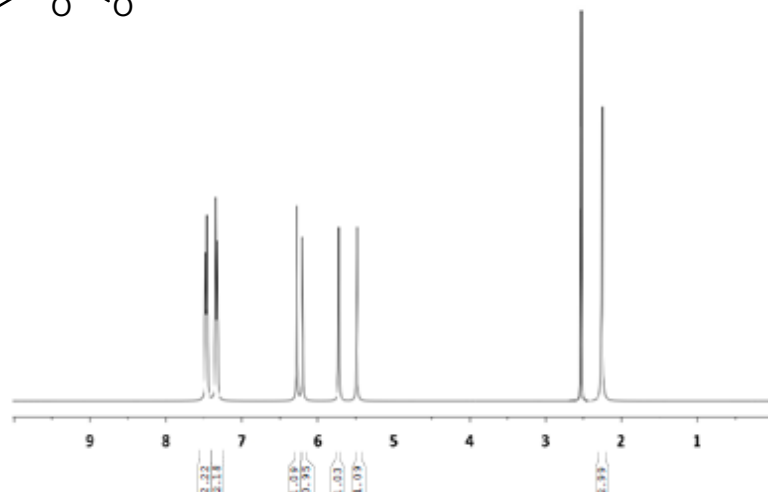
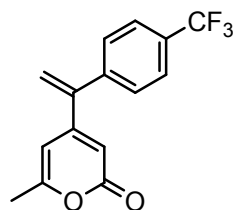
```

===== CHANNEL f1 =====
NUC1     13C
F1       10.00 usec
PL1      0.00 dB
PL1W     35.41759872 W
SFO1     100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     15.08 dB
PL13     18.08 dB
PL2W     8.31434441 W
PL12W    0.25812379 W
PL13W    0.12936834 W
SFO2     400.1316005 MHz
SI       32768
SF       100.6128193 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
FC       1.40
    
```

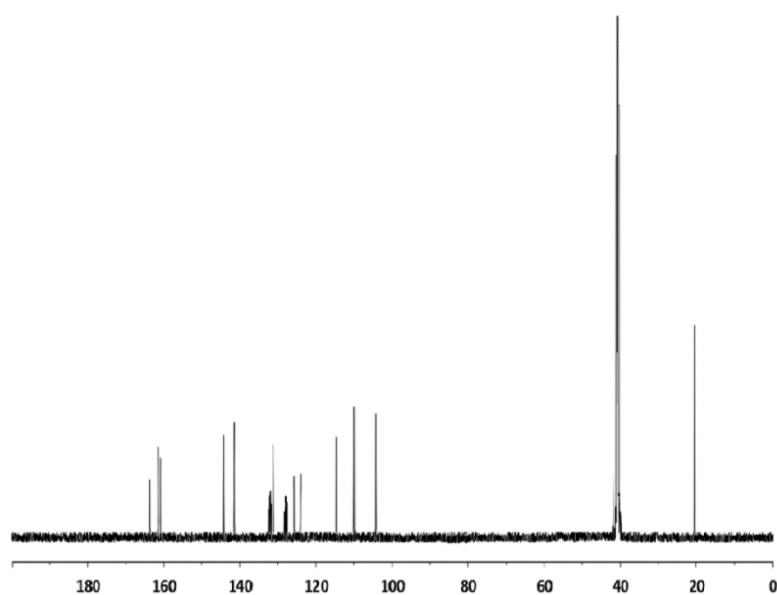
Compound 3r



```

NAME          SIC-19
EXPNO         1
PROCNO        1
Date_         20140709
Time          10.29
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            322
DW            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1

----- CHANNEL f1 -----
NUC1           1H
P1             14.10 usec
PL1            0.00 dB
PL1W           8.31434441 W
SFO1           400.1324710 MHz
SI             32768
SF             400.1300029 MHz
WDW            KM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
    
```



```

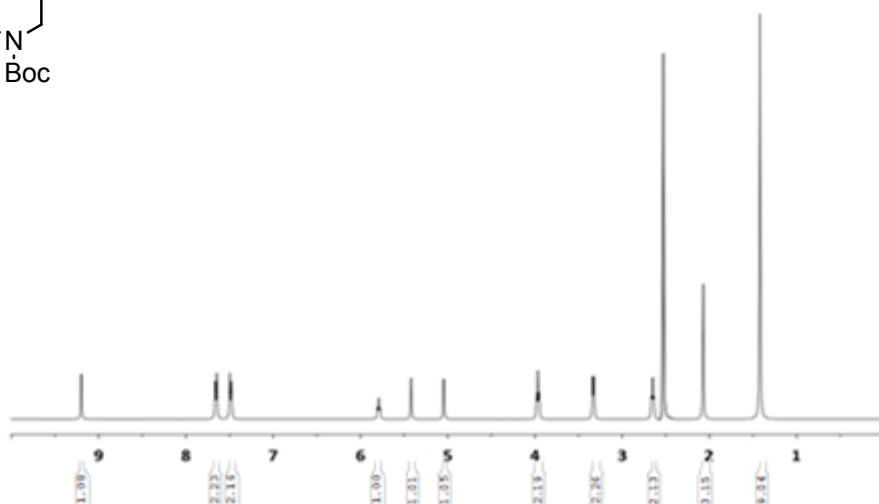
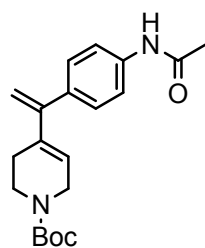
NAME          SIC-19
EXPNO         2
PROCNO        1
Date_         20140702
Time          12.36
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SWH           24038.461 Hz
FIDRES        0.386798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

----- CHANNEL f1 -----
NUC1           13C
P1             10.00 usec
PL1            0.00 dB
PL1W           35.41759872 W
SFO1           100.6228298 MHz

----- CHANNEL f2 -----
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2            0.00 dB
PL12           15.08 dB
PL13           18.08 dB
PL2W           8.31434441 W
PL12W          0.25812379 W
PL13W          0.12936834 W
SFO2           400.1316005 MHz
SI             32768
SF             100.6128193 MHz
WDW            HB
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
    
```



Compound 3s

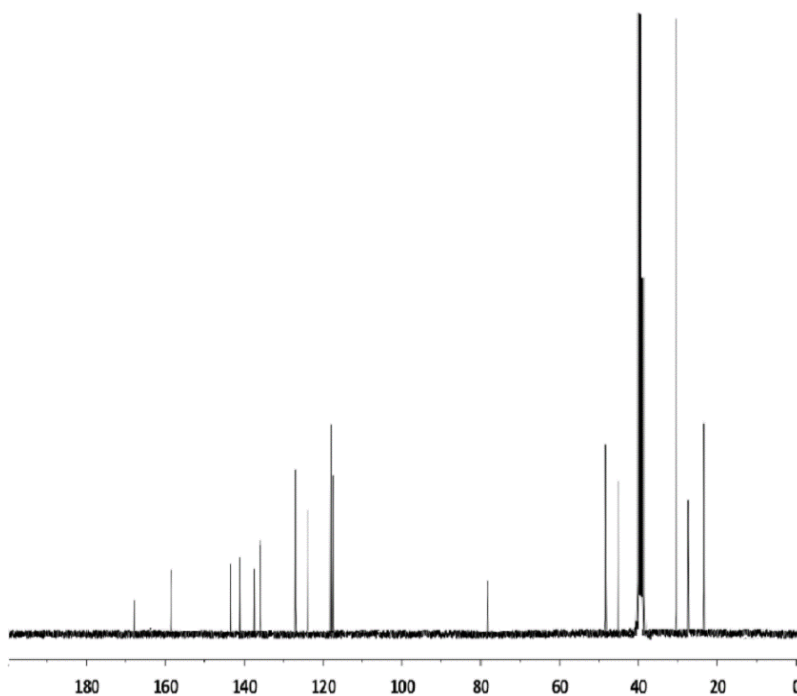


```

NAME          SIC- 20
EXPNO         1
PROCNO        1
Date_         20140709
Time          12.51
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65526
SOLVENT       DMSO
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125482 Hz
AQ            3.9846287 sec
RG            322
DM            60.800 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.10 usec
PL1           0.00 dB
PL1W          8.31424441 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300029 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

V



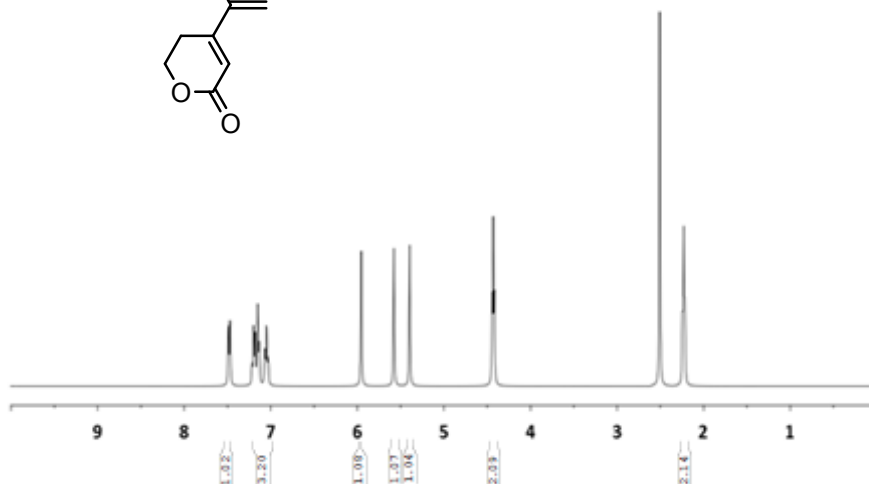
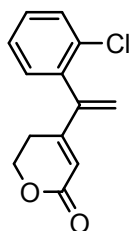
```

NAME          SIC-20
EXPNO         2
PROCNO        1
Date_         20140702
Time          13.51
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            249
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DM            20.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
PL1W          35.41759872 W
SFO1          100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          15.08 dB
PL13          18.08 dB
PL2W          8.31434441 W
PL12W         0.25812379 W
PL13W         0.12936834 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

Compound 3t

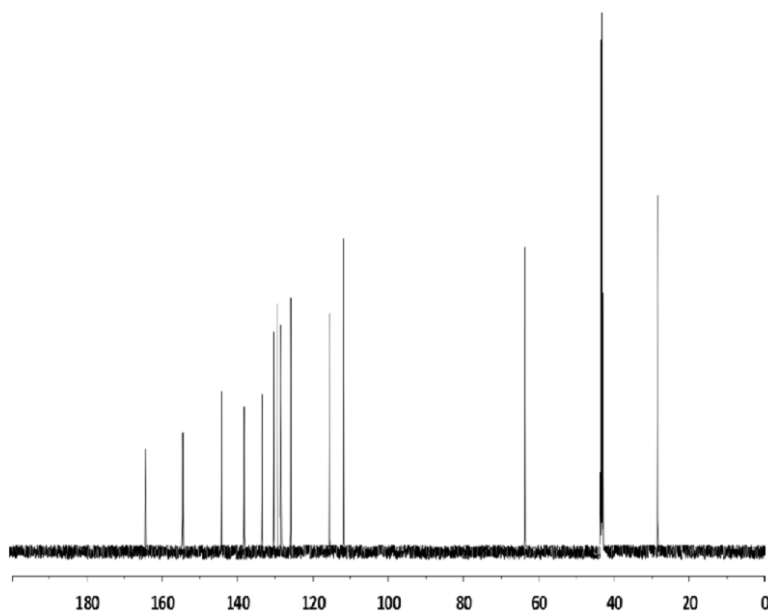


```

NAME      SIC- 21
EXPNO     1
PROCNO    1
Date_     20140709
Time      13.41
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         16
DS         2
SWH        8222.655 Hz
FIDRES     0.125483 Hz
AQ         2.9546287 sec
RG         322
DW         60.800 usec
DE         6.50 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1
    
```

```

CHANNEL f1
NUC1      1H
P1         14.10 usec
PL1        0.00 dB
PL1W      8.31434441 W
SFO1      400.1324710 MHz
SI         32768
SF         400.1300029 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



```

NAME      SIC-21
EXPNO     2
PROCNO    1
Date_     20140701
Time      21.47
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         249
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

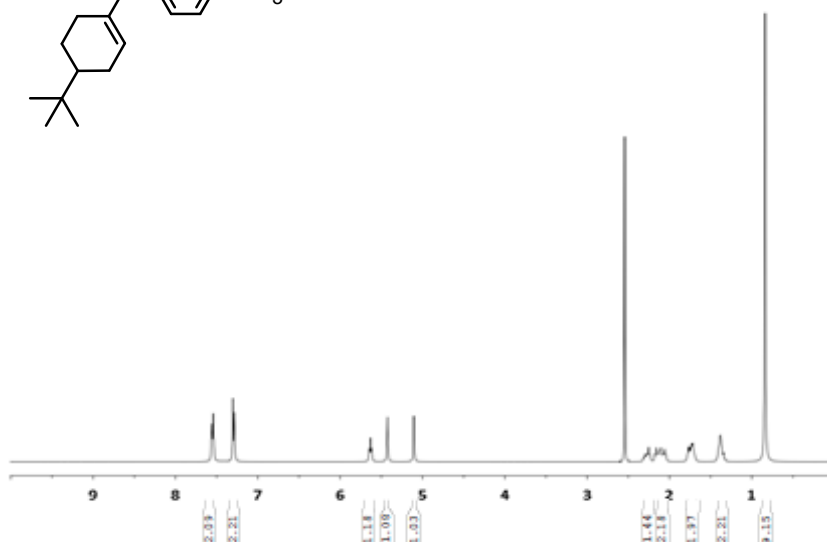
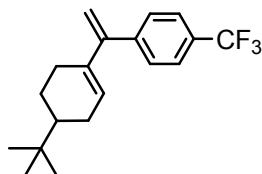
```

===== CHANNEL f1 =====
NUC1      13C
P1         10.00 usec
PL1        0.00 dB
PL1W      35.41759872 W
SFO1      100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2        0.00 dB
PL12       15.08 dB
PL13       18.08 dB
PL2W      8.31434441 W
PL12W     0.25512379 W
PL13W     0.12936834 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6128193 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

Compound 3u

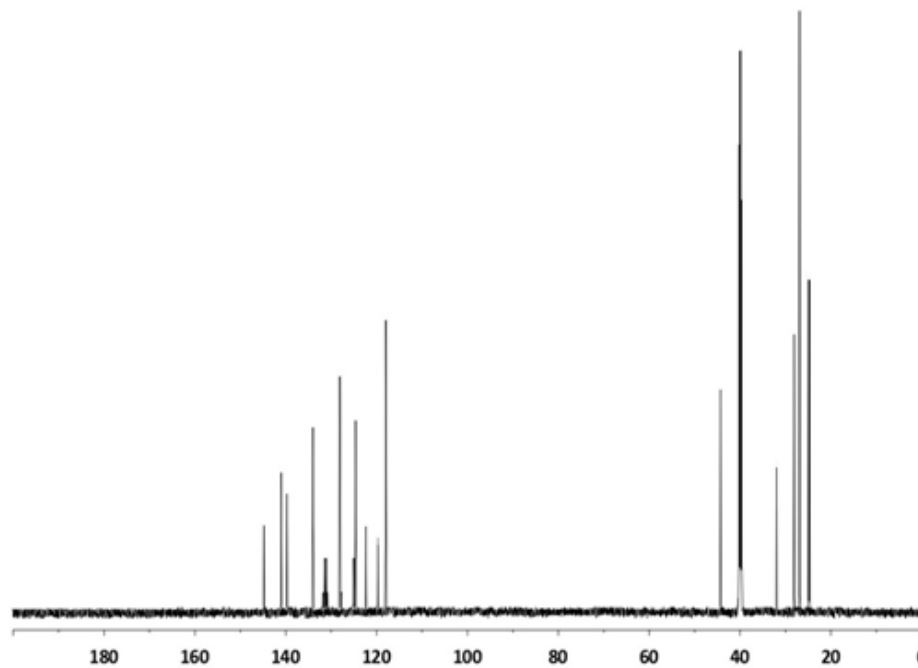


```

NAME      SIC- 22
EXPNO    1
PROCNO   1
Date_    20140709
Time     10.35
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD        65536
SOLVENT  DMSO
NS        16
DS        2
SWH       8223.685 Hz
FIDRES   0.125483 Hz
AQ        3.9846387 sec
RG        322
DW        60.800 usec
DE        6.50 usec
TE        300.0 K
D1        1.00000000 sec
TD0       1
    
```

```

***** CHANNEL f1 *****
NUC1      1H
P1        14.10 usec
PL1       0.00 dB
PL1W      8.31434441 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300029 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



```

NAME      SIC-22
EXPNO    3
PROCNO   1
Date     20140702
Time     10.53
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD        65536
SOLVENT  DMSO
NS        4
DS        4
SWH       24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        300.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
    
```

```

----- CHANNEL #1 -----
NUC1      13C
P1        10.00 usec
PL1       0.00 dB
PL1W      35.41759872 W
SFO1      100.6228298 MHz
    
```

```

----- CHANNEL #2 -----
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2       0.00 dB
PL12     15.08 dB
PL13     18.08 dB
PL2W     8.31434441 W
PL12W    0.25812379 W
PL13W    0.12936834 W
SFO2     400.1316005 MHz
SI        32768
SF        100.6128193 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```