

# Palladium-Catalyzed Multicomponent Reaction of Alkynes, Carboxylic Acids, and Isocyanides: A Direct Approach to Captodative Olefins

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## Supporting Information

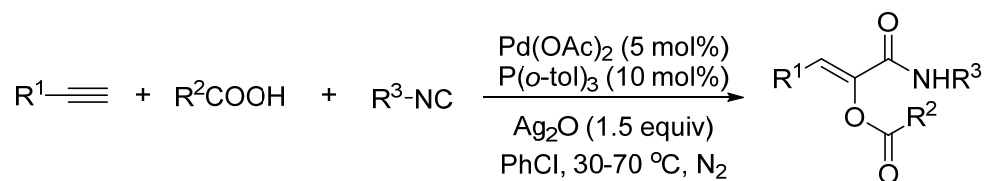
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## 1. General Information

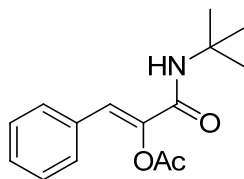
All reagents and metal catalysts were obtained from commercial sources without further purification, and commercially available solvents were purified before use. All new compounds were fully characterized. All melting points were taken on a WRS-1A or a WRS-1B Digital Melting Point Apparatus without correction. Infrared spectra were obtained using an AVATAR 370 FT-IR spectrometer.  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{19}\text{F}$  NMR spectra were recorded with a Bruker AV-500 spectrometer operating at 500 MHz, 125 MHz and 470 MHz, respectively, with chemical shift values being reported in ppm relative to chloroform ( $\delta = 7.26$  ppm), acetone ( $\delta = 2.05$  ppm) or TMS ( $\delta = 0.00$  ppm) for  $^1\text{H}$  NMR; chloroform ( $\delta = 77.16$  ppm) or acetone ( $\delta = 29.84, 206.26$  ppm) for  $^{13}\text{C}$  NMR; and  $\text{C}_6\text{F}_6$  ( $\delta = -164.9$  ppm) for  $^{19}\text{F}$  NMR. Mass spectra and high resolution mass spectra were recorded with an Agilent 5975N using an Electron impact (EI) or Electrospray ionization (ESI) techniques. Silica gel plate GF254 was used for thin layer chromatography (TLC) and silica gel H or 300-400 mesh was used for flash column chromatography. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise indicated. Isocyanides are purchased commercially or prepared according to the literature reported procedures, for example, 1-isocyanoadamantane.<sup>1</sup>

## 2. Synthesis and Characterization of Compounds 4 and 5



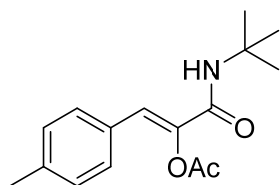
*General Method A:* To a test tube were added Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), isocyanide (0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL). The mixture was stirred at 30 °C under N<sub>2</sub> atmosphere. Alkyne (0.3 mmol) in PhCl (0.5 mL) was then added *via* syringe pump for 3 h. The reaction was kept stirring at 30 °C for another 2 h. Upon completion monitored by TLC, the reaction was cooled down to room temperature and filtered. The filtrate was washed with H<sub>2</sub>O and brine. The aqueous phase was then extracted with ethyl acetate (3 × 10 mL) and the combined organic phase was dried over Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent under reduced pressure, the crude product was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate) to give the pure product.

*General Method B:* To a test tube were added Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), carboxylic acid (0.9 mmol) and PhCl (1.0 mL). The mixture was stirred at 70 °C under N<sub>2</sub> atmosphere for 30 min. Then isocyanide (0.6 mmol) was added in one portion and alkyne (0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump for 3 h. The mixture was kept stirring at 70 °C for another 12 h. Upon completion monitored by TLC, the reaction was cooled down to room temperature and filtered. The filtrate was washed with H<sub>2</sub>O and brine. The aqueous phase was then extracted with ethyl acetate (3 × 10 mL) and the combined organic phase was dried over Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent under reduced pressure, the crude product was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate) to give the pure product.

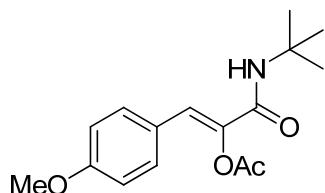


**(Z)-3-(tert-Butylamino)-3-oxo-1-phenylprop-1-en-2-yl acetate (4a):** Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), *t*BuNC (68  $\mu$ L, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1a** (33  $\mu$ L, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **4a** was afforded as a white solid (65.4 mg, 83%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 130-131 °C. IR (KBr, cm<sup>-1</sup>): 3351, 3060, 2970, 2927, 1766, 1636, 1546, 1449, 1363, 1294, 1200, 1113, 1041, 1008, 935, 876, 766, 687, 614; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  7.48 (d, *J* = 7.0 Hz, 2H), 7.39-7.28 (m, 3H), 7.11 (s, 1H), 5.83 (br, 1H), 2.29 (s, 3H), 1.41 (s, 9H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta$  167.9, 161.9, 140.7, 132.6, 129.4, 129.0, 128.7, 122.4, 51.6, 28.7, 20.9; LC-MS (ESI) *m/z*: 262 [M<sup>+</sup>H]; HRMS (DART Positive) *m/z*: calcd for C<sub>15</sub>H<sub>20</sub>NO<sub>3</sub> [M<sup>+</sup>H] 262.1438, found 262.1432.

**1.0 mmol scale reaction for preparation of 4a:** To a test tube were added Pd(OAc)<sub>2</sub> (11.2 mg, 0.05 mmol), tri(2-methylphenyl)phosphine (30.4 mg, 0.1 mmol), Ag<sub>2</sub>O (347.6 mg, 1.5 mmol), *t*BuNC (227  $\mu$ L, 2.0 mmol), AcOH (172  $\mu$ L, 3.0 mmol) and PhCl (3.0 mL). The mixture was stirred at 30 °C under N<sub>2</sub> atmosphere. Phenylacetylene (110  $\mu$ L, 1.0 mmol) in PhCl (2.0 mL) was then added *via* syringe pump for 12 h. The reaction was kept stirring at 30 °C for another 2.5 h. Upon completion monitored by TLC, the reaction was cooled down to room temperature and filtered. The filtrate was washed with saturated NaHCO<sub>3</sub> aqueous and brine. The aqueous phase was then extracted with ethyl acetate (3  $\times$  20 mL) and the combined organic phase was dried over Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent under reduced pressure, the crude product was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) to give the pure product **4a** (180.4 mg, 69%).

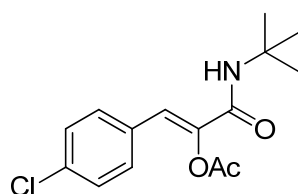


**(Z)-3-(tert-Butylamino)-3-oxo-1-(p-tolyl)prop-1-en-2-yl acetate (4b):** Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1b** (38 μL, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **4b** was afforded as a white solid (63.6 mg, 77%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 153-154 °C. IR (KBr, cm<sup>-1</sup>): 3324, 2966, 2924, 1769, 1633, 1540, 1447, 1366, 1310, 1193, 1009, 937, 812; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.37 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 3H), 7.09 (s, 1H), 5.82 (br, 1H), 2.34 (s, 3H), 2.29 (s, 3H), 1.41 (s, 9H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 168.0, 162.1, 140.1, 139.4, 129.8, 129.6, 129.5, 122.6, 51.7, 28.8, 21.5, 21.0; LC-MS (ESI) *m/z*: 276 [M<sup>+</sup>H]; HRMS (DART Positive) *m/z*: calcd for C<sub>16</sub>H<sub>22</sub>NO<sub>3</sub> [M<sup>+</sup>H] 276.1594, found 276.1588.



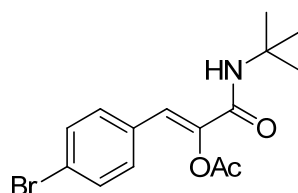
**(Z)-3-(tert-Butylamino)-1-(4-methoxyphenyl)-3-oxoprop-1-en-2-yl acetate (4c):** Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1c** (39 μL, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **4c** was afforded as a white solid (64.6 mg, 74%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 99-100 °C. IR (KBr, cm<sup>-1</sup>): 3397, 2972, 2931, 1752, 1675, 1643, 1603, 1518, 1453, 1367, 1296, 1252, 1183,

1105, 1024, 831, 537;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.43 (d,  $J = 9.0$  Hz, 2H), 7.07 (s, 1H), 6.87 (d,  $J = 9.0$  Hz, 2H), 5.80 (br, 1H), 3.81 (s, 3H), 2.29 (s, 3H), 1.40 (s, 9H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  168.1, 162.2, 160.3, 139.2, 131.1, 125.3, 122.3, 114.3, 55.4, 51.6, 28.8, 21.0; LC-MS (ESI)  $m/z$ : 292 [ $\text{M}^+\text{H}$ ]; HRMS (DART Positive)  $m/z$ : calcd for  $\text{C}_{16}\text{H}_{22}\text{NO}_4$  [ $\text{M}^+\text{H}$ ] 292.1543, found 292.1537.



**(Z)-3-(tert-Butylamino)-1-(4-chlorophenyl)-3-oxoprop-1-en-2-yl acetate (4d):**

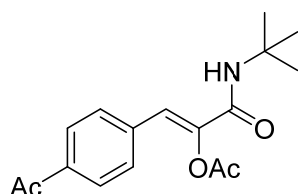
Following the *General Method A*, to the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol),  $\text{Ag}_2\text{O}$  (104.3 mg, 0.45 mmol),  $t\text{BuNC}$  (68  $\mu\text{L}$ , 0.6 mmol),  $\text{AcOH}$  (54.0 mg, 0.9 mmol) and  $\text{PhCl}$  (1.0 mL), **1d** (42 mg, 0.3 mmol) in  $\text{PhCl}$  (0.5 mL) was added *via* syringe pump at 50  $^\circ\text{C}$  for 3 h. The reaction was kept stirring at 50  $^\circ\text{C}$  for another 2 h, and the desired product **4d** was afforded as a white solid (58.3 mg, 74%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 155-156  $^\circ\text{C}$ . IR (KBr,  $\text{cm}^{-1}$ ): 3352, 3060, 2981, 1767, 1629, 1538, 1456, 1364, 1360, 1196, 1105, 1010, 939, 830, 753, 668, 584, 491;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.41 (d,  $J = 8.5$  Hz, 2H), 7.33 (d,  $J = 8.5$  Hz, 2H), 7.06 (s, 1H), 5.81 (br, 1H), 2.34 (s, 3H), 2.29 (s, 3H), 1.41 (s, 9H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  167.8, 161.7, 141.2, 135.0, 131.2, 130.7, 129.1, 121.3, 51.8, 28.8, 21.0; LC-MS (ESI)  $m/z$ : 318 [ $\text{M}^+\text{Na}$ ]; HRMS (DART Positive)  $m/z$ : calcd for  $\text{C}_{15}\text{H}_{19}\text{NO}_3\text{Cl}$  [ $\text{M}^+\text{H}$ ] 296.1048, found 296.1046.



**(Z)-1-(4-Bromophenyl)-3-(tert-butylamino)-3-oxoprop-1-en-2-yl acetate (4e):**

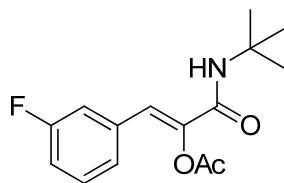
Following the *General Method A*, to the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol),  $\text{Ag}_2\text{O}$  (104.3 mg, 0.45 mmol),

<sup>t</sup>BuNC (68  $\mu$ L, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1e** (56 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 50 °C for 3 h. The reaction was kept stirring at 50 °C for another 2 h, and the desired product **4e** was afforded as a white solid (60.2 mg, 59%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 166-168 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3361, 2969, 1764, 1632, 1534, 1455, 1364, 1305, 1198, 1110, 1008, 939, 827, 580, 492; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.49 (d,  $J$  = 8.5 Hz, 2H), 7.34 (d,  $J$  = 8.5 Hz, 2H), 7.04 (s, 1H), 5.80 (br, 1H), 2.29 (s, 3H), 1.41 (s, 9H); <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  167.6, 161.6, 141.2, 132.0, 131.5, 130.8, 123.2, 121.3, 51.7, 28.6, 20.9; LC-MS (ESI)  $m/z$ : 340 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{15}\text{H}_{19}\text{NO}_3\text{Br}$  [ $\text{M}^+\text{H}$ ] 340.0543, found 340.0542.



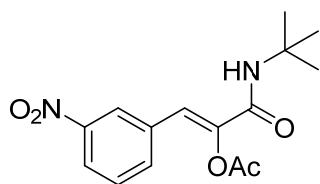
**(Z)-1-(4-Acetylphenyl)-3-(tert-butylamino)-3-oxoprop-1-en-2-yl acetate (4f):**

Following the *General Method A*, to the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol),  $\text{Ag}_2\text{O}$  (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68  $\mu$ L, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1f** (43 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 50 °C for 3 h. The reaction was kept stirring at 50 °C for another 2 h, and the desired product **4f** was afforded as a yellow solid (44.7 mg, 50%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 130-131 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3394, 2974, 2931, 1769, 1677, 1637, 1514, 1459, 1408, 1365, 1269, 1177, 1106, 1010, 913, 829, 579; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.94 (d,  $J$  = 8.5 Hz, 2H), 7.56 (d,  $J$  = 8.5 Hz, 2H), 7.14 (s, 1H), 5.85 (br, 1H), 2.60 (s, 3H), 2.30 (s, 3H), 1.42 (s, 9H); <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  197.5, 167.6, 161.4, 142.2, 137.3, 136.9, 129.4, 128.6, 121.2, 51.8, 28.6, 26.6, 20.9; LC-MS (ESI)  $m/z$ : 304 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{17}\text{H}_{22}\text{NO}_4$  [ $\text{M}^+\text{H}$ ] 304.1543, found 304.1542.



**(Z)-3-(tert-Butylamino)-1-(3-fluorophenyl)-3-oxoprop-1-en-2-yl acetate (4g):**

Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1g** (38 μL, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 50 °C for 3 h. The reaction was kept stirring at 50 °C for another 2 h, and the desired product **4g** was afforded as a white solid (50.3 mg, 60%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 123-125 °C. IR (KBr, cm<sup>-1</sup>): 3329, 3073, 2988, 1772, 1630, 1544, 1456, 1420, 1365, 1314, 1228, 1194, 1109, 938, 839; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.37-7.29 (m, 1H), 7.25-7.17 (m, 2H), 7.06 (s, 1H), 7.04-6.99 (m, 1H), 5.82 (br, 1 H), 2.30 (s, 3H), 1.41 (s, 9H); <sup>19</sup>F (CDCl<sub>3</sub>, 470 MHz): δ -112.5 (m, Ar-F); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 167.8, 162.9 (d, <sup>1</sup>J<sub>C-F</sub> = 244.5 Hz), 161.7, 141.7, 134.8 (d, <sup>3</sup>J<sub>C-F</sub> = 8.1 Hz), 130.3 (d, <sup>3</sup>J<sub>C-F</sub> = 8.4 Hz), 125.5 (d, <sup>4</sup>J<sub>C-F</sub> = 2.7 Hz), 121.3 (d, <sup>4</sup>J<sub>C-F</sub> = 2.5 Hz), 116.1 (d, <sup>2</sup>J<sub>C-F</sub> = 21.1 Hz), 115.8 (d, <sup>2</sup>J<sub>C-F</sub> = 22.2 Hz), 51.9, 28.8, 21.0; LC-MS (ESI) m/z: 280 [M<sup>+</sup>H]; HRMS (ESI) m/z: calcd for C<sub>15</sub>H<sub>19</sub>NO<sub>3</sub>F [M<sup>+</sup>H] 280.1343, found 280.1346.

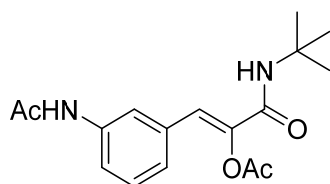


**(Z)-3-(tert-Butylamino)-1-(3-nitrophenyl)-3-oxoprop-1-en-2-yl acetate (4h):**

Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1h** (44 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 50 °C for 3 h. The reaction was kept stirring at 50 °C for another 2 h, and the desired product **4h** was afforded as a white solid (55.2 mg, 60%) after flash column chromatography

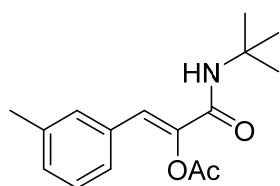


purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 84-85 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3739, 3412, 3281, 3064, 2972, 2928, 1768, 1632, 1533, 1447, 1355, 1206, 1118, 1007, 911, 675;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  8.43 (s, 1H), 8.16 (d,  $J$  = 8.0 Hz, 1H), 7.73 (d,  $J$  = 8.0 Hz, 1H), 7.54 (t,  $J$  = 8.0 Hz, 1H), 7.16 (s, 1H), 5.90 (br, 1H), 2.35 (s, 3H), 1.42 (s, 9H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  167.5, 161.1, 148.4, 142.7, 135.5, 134.3, 129.8, 123.5, 123.3, 119.8, 51.9, 28.6, 20.9; LC-MS (ESI)  $m/z$ : 307 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{15}\text{H}_{19}\text{N}_2\text{O}_5$  [ $\text{M}^+\text{H}$ ] 307.1288, found 307.1290.

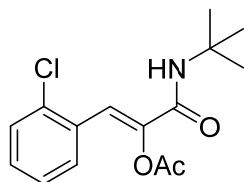


**(Z)-1-(3-Acetamidophenyl)-3-(tert-butylamino)-3-oxoprop-1-en-2-yl acetate (4i):**

Following the *General Method A*, to the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol),  $\text{Ag}_2\text{O}$  (104.3 mg, 0.45 mmol),  $t\text{BuNC}$  (68  $\mu\text{L}$ , 0.6 mmol),  $\text{AcOH}$  (54.0 mg, 0.9 mmol) and  $\text{PhCl}$  (1.0 mL), **1i** (48 mg, 0.3 mmol) in  $\text{PhCl}$  (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **4i** was afforded as a white solid (45.7 mg, 48%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 103-104 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3306, 2973, 1768, 1678, 1533, 1439, 1368, 1309, 1194, 1109, 1011, 904, 789, 689, 533;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.92 (s, 1H), 7.80-7.65 (m, 1H), 7.31-7.20 (m, 2H), 7.18-7.11 (d,  $J$  = 7.0 Hz, 1H), 7.07 (s, 1H), 5.91 (br, 1H), 2.35 (s, 3H), 2.16 (s, 3H), 1.40 (s, 9H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  168.7, 168.3, 162.0, 140.7, 138.6, 133.2, 129.2, 125.5, 122.4, 120.2, 120.1 51.7, 28.7, 24.6, 20.9; LC-MS (ESI)  $m/z$ : 319 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{17}\text{H}_{23}\text{N}_2\text{O}_4$  [ $\text{M}^+\text{H}$ ] 319.1652, found 319.1651.

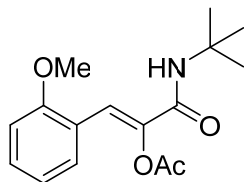


**(Z)-3-(tert-Butylamino)-3-oxo-1-(m-tolyl)prop-1-en-2-yl acetate (4j):** Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1j** (36 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **4j** was afforded as a white solid (69.1 mg, 84%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 116-117 °C. IR (KBr, cm<sup>-1</sup>): 3316, 3059, 2967, 2920, 1766, 1634, 1541, 1479, 1442, 1364, 1312, 1193, 1113, 1008, 907, 784, 689; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.29 (d, *J* = 7.5 Hz, 1H), 7.27 (s, 1H), 7.24 (t, *J* = 7.5 Hz, 1H), 7.12 (d, *J* = 7.5 Hz, 1 H), 7.09 (s, 1H), 5.82 (br, 1H), 2.34 (s, 3H), 2.28 (s, 3H), 1.41 (s, 9H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 167.9, 161.9, 140.5, 138.3, 132.5, 130.2, 129.9, 128.6, 126.3, 122.6, 51.6, 28.7, 21.4, 20.9; LC-MS (ESI) *m/z*: 276 [M<sup>+</sup>H]; HRMS (DART Positive) *m/z*: calcd for C<sub>16</sub>H<sub>22</sub>NO<sub>3</sub> [M<sup>+</sup>H] 276.1594, found 276.1589.



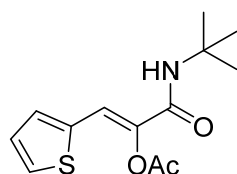
**(Z)-3-(tert-Butylamino)-1-(2-chlorophenyl)-3-oxoprop-1-en-2-yl acetate (4k):** Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1k** (43 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 50 °C for 3 h. The reaction was kept stirring at 50 °C for another 2 h, and the desired product **4k** was afforded as a white solid (49.8 mg, 56%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 123-124 °C. IR (KBr, cm<sup>-1</sup>): 3851, 3740, 3352, 3061, 2982, 1767, 1630, 1538, 1456, 1364, 1307, 1197, 1105, 939, 830, 585; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.56-7.50 (m, 1H), 7.43-7.38 (m, 1H), 7.32 (s, 1H), 7.26-7.21 (m, 2H), 5.86 (br, 1H), 2.21 (s, 3H), 1.42 (s, 9H); <sup>13</sup>C NMR

(CDCl<sub>3</sub>, 125 MHz):  $\delta$  167.8, 161.6, 142.2, 134.3, 131.1, 129.9, 129.8, 129.7, 126.7, 118.7, 51.8, 28.6, 20.8; LC-MS (ESI)  $m/z$ : 296 [M<sup>+</sup>H]; HRMS (ESI)  $m/z$ : calcd for C<sub>15</sub>H<sub>19</sub>NO<sub>3</sub>Cl [M<sup>+</sup>H] 296.1048, found 296.1048.



**(Z)-3-(tert-Butylamino)-1-(2-methoxyphenyl)-3-oxoprop-1-en-2-yl acetate (4l):**

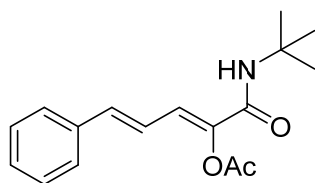
Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68  $\mu$ L, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1l** (41 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **4l** was afforded as a white solid (79.0 mg, 90%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 109-111 °C. IR (KBr, cm<sup>-1</sup>): 3326, 2967, 2929, 1774, 1664, 1631, 1524, 1491, 1452, 1399, 1365, 1295, 1254, 1180, 1106, 1018, 926, 751, 668; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  7.52 (dd,  $J$  = 8.0, 1.5 Hz, 1H), 7.39 (s, 1H), 7.32-7.26 (m, 1H), 6.92 (t,  $J$  = 7.5 Hz, 1H), 6.88 (d,  $J$  = 8.5 Hz, 1H), 5.84 (br, 1H), 3.84 (s, 3H), 2.24 (s, 3H), 1.41 (s, 9H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta$  168.1, 162.1, 157.5, 140.8, 130.4, 129.2, 121.5, 120.4, 117.0, 110.8, 55.5, 51.6, 28.7, 20.9; LC-MS (ESI)  $m/z$ : 314 [M<sup>+</sup>Na]; HRMS (DART Positive)  $m/z$ : calcd for C<sub>16</sub>H<sub>22</sub>NO<sub>4</sub> [M<sup>+</sup>H] 292.1543, found 292.1537.



**(Z)-3-(tert-Butylamino)-3-oxo-1-(thiophen-2-yl)prop-1-en-2-yl acetate (4m):**

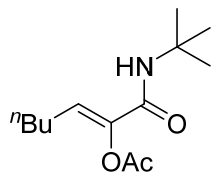
Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68  $\mu$ L, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1m** (34

mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 50 °C for 3 h. The reaction was kept stirring at 50 °C for another 2 h, and the desired product **4m** was afforded as a yellow solid (35.3 mg, 44%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 156-157 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3320, 2972, 2925, 1769, 1624, 1533, 1446, 1365, 1301, 1177, 1099, 1042, 1007, 934, 858, 703;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.43-7.40 (m, 2H), 7.23 (d,  $J$  = 3.5 Hz, 1H), 7.05 (dd,  $J$  = 5.0, 4.0 Hz, 1H), 5.73 (br, 1H), 2.41 (s, 3H), 1.40 (s, 9H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  168.1, 161.4, 138.4, 134.8, 131.5, 129.2, 127.2, 117.0, 51.7, 28.7, 21.3; LC-MS (ESI)  $m/z$ : 268 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{13}\text{H}_{18}\text{NO}_3\text{S}$  [ $\text{M}^+\text{H}$ ] 268.1002, found 268.1002.

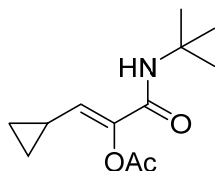


**(2Z,4E)-1-(tert-Butylamino)-1-oxo-5-phenylpenta-2,4-dien-2-yl acetate (4n):**

Following the *General Method A*, to the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol),  $\text{Ag}_2\text{O}$  (104.3 mg, 0.45 mmol),  $t\text{BuNC}$  (68  $\mu\text{L}$ , 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1n** (38.4 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 50 °C for 3 h. The reaction was kept stirring at 50 °C for another 2 h, and the desired product **4n** was afforded as a white solid (48.8 mg, 57%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 114-115 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3276, 3058, 2968, 2926, 2766, 1767, 1645, 1543, 1454, 1363, 1327, 1300, 1201, 1103, 975, 746, 686;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.43 (d,  $J$  = 7.5 Hz, 2H), 7.37-7.31 (m, 2H), 7.31-7.27 (m, 1H), 6.96 (d,  $J$  = 11.0 Hz, 1 H), 6.85 (d,  $J$  = 15.5 Hz, 1H), 6.70 (dd,  $J$  = 16.0, 11.5 Hz, 1H), 5.73 (br, 1H), 2.35 (s, 3H), 1.40 (s, 9H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  168.2, 161.4, 140.2, 138.9, 136.3, 128.9, 128.7, 127.1, 123.5, 120.2, 51.6, 28.7, 20.6; LC-MS (ESI)  $m/z$ : 288 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{17}\text{H}_{22}\text{NO}_3$  [ $\text{M}^+\text{H}$ ] 288.1594, found 288.1591.

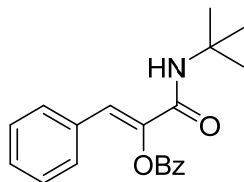


**(Z)-1-(tert-Butylamino)-1-oxohept-2-en-2-yl acetate (4o):** Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)-phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1o** (35 μL, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **4o** was afforded as a white solid (50.8 mg, 70%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 10:1). M.p. 78-80 °C. IR (KBr, cm<sup>-1</sup>): 3063, 2962, 2870, 1764, 1534, 1540, 1458, 1368, 1315, 1211, 1150, 1089, 942; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 6.31 (t, *J* = 7.5 Hz, 1H), 5.67 (br, 1H), 2.25 (s, 3H), 2.01 (q, *J* = 7.0 Hz, 2H), 1.43-1.30 (m, 13H), 0.89 (t, *J* = 7.5 Hz, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 168.0, 161.3, 141.1, 126.2, 51.4, 30.3, 28.7, 25.8, 22.3, 20.5, 13.8; LC-MS (ESI) *m/z*: 242 [M<sup>+</sup>H]; HRMS (ESI) *m/z*: calcd for C<sub>13</sub>H<sub>24</sub>NO<sub>3</sub> [M<sup>+</sup>H] 242.1751, found 242.1750.

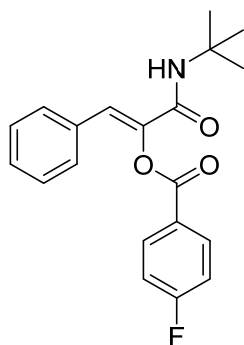


**(Z)-3-(tert-Butylamino)-1-cyclopropyl-3-oxoprop-1-en-2-yl acetate (4p):** Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), <sup>t</sup>BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), **1p** (26 μL, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **4p** was afforded as a white solid (45.6 mg, 67%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 131-132 °C. IR (KBr, cm<sup>-1</sup>): 3298, 3065, 2981, 1765, 1629, 1543, 1449, 1370, 1317, 1217, 1099, 967, 879; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 5.82 (d, *J* = 11.0 Hz, 1H), 5.63 (br, 1H), 2.28 (s, 3H),

1.43-1.30 (m, 10H), 0.92-0.87 (m, 2H), 0.63-0.57 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  168.1, 161.3, 140.5, 131.5, 51.4, 28.8, 20.7, 9.5, 8.0; LC-MS (ESI)  $m/z$ : 226  $[\text{M}^+\text{H}]$ ; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{12}\text{H}_{20}\text{NO}_3$   $[\text{M}^+\text{H}]$  226.1438, found 226.1436.

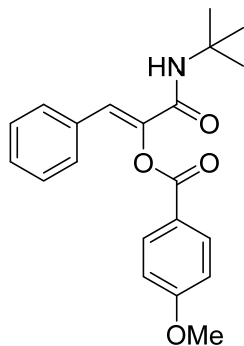


**(Z)-3-(tert-Butylamino)-3-oxo-1-phenylprop-1-en-2-yl benzoate (5a):** Following the *General Method B*, the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol),  $\text{Ag}_2\text{O}$  (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), benzoic acid (110 mg, 0.9 mmol) and  $\text{PhCl}$  (1.0 mL) was stirred at 70  $^\circ\text{C}$  under  $\text{N}_2$  atmosphere for 30 min. Then  $t\text{-BuNC}$  (68  $\mu\text{L}$ , 0.6 mmol) was added in one portion and phenylacetylene (33  $\mu\text{L}$ , 0.3 mmol) in  $\text{PhCl}$  (0.5 mL) was added *via* syringe pump for 3 h. The mixture was kept stirring at 70  $^\circ\text{C}$  for another 12 h and afforded the desired product **5a** as a white solid (80.0 mg, 82%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 145-146  $^\circ\text{C}$ . IR (KBr,  $\text{cm}^{-1}$ ): 3324, 3067, 2970, 1743, 1637, 1546, 1452, 1314, 1251, 1119, 1056, 1022, 910, 756, 700;  $^1\text{H}$  NMR (Acetone, 500 MHz):  $\delta$  8.18 (d,  $J = 7.5$  Hz, 2H), 7.75 (t,  $J = 7.5$  Hz, 1H), 7.66-7.57 (m, 4H), 7.36-7.26 (m, 3H), 7.17 (s, 1H), 7.08 (br, 1H), 1.38 (s, 9H);  $^{13}\text{C}$  NMR (Acetone, 125 MHz):  $\delta$  164.6, 162.6, 142.8, 134.9, 133.9, 131.0, 130.3, 130.0, 129.8, 129.7, 129.6, 122.1, 52.1, 28.9; LC-MS (ESI)  $m/z$ : 324  $[\text{M}^+\text{H}]$ ; HRMS (DART Positive)  $m/z$ : calcd for  $\text{C}_{20}\text{H}_{22}\text{NO}_3$   $[\text{M}^+\text{H}]$  324.1594, found 324.1587.



**(Z)-3-(tert-Butylamino)-3-oxo-1-phenylprop-1-en-2-yl 4-fluorobenzoate (5b):** Following the *General Method B*, the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol),

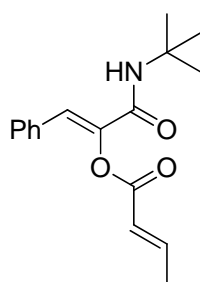
Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), 4-fluorobenzoic acid (126 mg, 0.9 mmol) and PhCl (1.0 mL) was stirred at 70 °C under N<sub>2</sub> atmosphere for 30 min. Then <sup>t</sup>BuNC (68 μL, 0.6 mmol) was added in one portion and phenylacetylene (33 μL, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump for 3 h. The mixture was kept stirring at 70 °C for another 12 h and afforded the desired product **5b** as a white solid (87.9 mg, 86%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 128-130 °C. IR (KBr, cm<sup>-1</sup>): 3326, 3063, 2971, 1744, 1633, 1604, 1537, 1511, 1453, 1393, 1363, 1300, 1251, 1117, 1059, 761; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 8.24-8.19 (m, 2H), 7.53-7.45 (m, 2H), 7.32-7.26 (m, 3H), 7.25-7.17 (m, 3H), 5.85 (br, 1H), 1.40 (s, 9H); <sup>19</sup>F (CDCl<sub>3</sub>, 470 MHz): δ -103.0 (m, Ar-F); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 167.5, 165.5, 162.7 (d, <sup>1</sup>J<sub>C-F</sub> = 119.0 Hz), 140.6, 133.0 (d, <sup>3</sup>J<sub>C-F</sub> = 9.0 Hz), 132.4, 129.5, 129.1, 128.7, 124.7 (d, <sup>4</sup>J<sub>C-F</sub> = 3.0 Hz), 122.8, 116.3 (d, <sup>2</sup>J<sub>C-F</sub> = 22.0 Hz), 51.7, 28.7; LC-MS (ESI) m/z: 342 [M<sup>+</sup>H]; HRMS (ESI) m/z: calcd for C<sub>20</sub>H<sub>21</sub>NO<sub>3</sub>F [M<sup>+</sup>H] 342.1500, found 342.1500.



**(Z)-3-(tert-Butylamino)-3-oxo-1-phenylprop-1-en-2-yl 4-methoxybenzoate (5c):**

Following the *General Method B*, the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), 4-methoxybenzoic acid (137 mg, 0.9 mmol) and PhCl (1.0 mL) was stirred at 70 °C under N<sub>2</sub> atmosphere for 30 min. Then <sup>t</sup>BuNC (68 μL, 0.6 mmol) was added in one portion and phenylacetylene (33 μL, 0.3mmol) in PhCl (0.5 mL) was added *via* syringe pump for 3 h. The mixture was kept stirring at 70 °C for another 12 h and afforded the desired product **5c** as a white solid (72.8 mg, 68%) after flash column

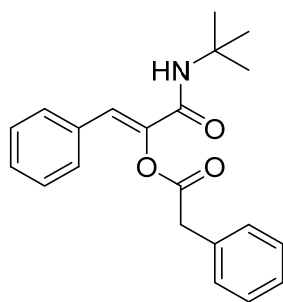
chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 145-146 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3740, 3422, 3059, 2972, 2923, 2835, 1740, 1673, 1637, 1602, 1510, 1452, 1363, 1249, 1169, 1104, 1042, 1006, 844, 760, 688, 558, 452;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  8.15 (d,  $J$  = 9.0 Hz, 2H), 7.53-7.48 (m, 2H), 7.29-7.25 (m, 3H), 7.01 (d,  $J$  = 9.0 Hz, 2H), 5.89 (br, 1H), 3.91 (s, 3H), 1.39 (s, 9H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  164.4, 163.3, 162.0, 140.5, 132.6, 132.5, 129.5, 129.0, 128.7, 122.9, 120.6, 114.3, 55.6, 51.6, 28.7; LC-MS (ESI)  $m/z$ : 354 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{21}\text{H}_{24}\text{NO}_4$  [ $\text{M}^+\text{H}$ ] 354.1700, found 354.1700.



**(Z)-3-(tert-Butylamino)-3-oxo-1-phenylprop-1-en-2-yl (E)-but-2-enoate (5d):**

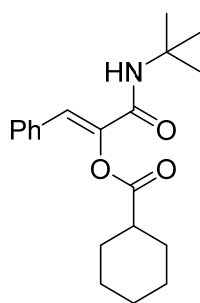
Following the *General Method B*, the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol),  $\text{Ag}_2\text{O}$  (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), (*E*)-but-2-enoic acid (77.5 mg, 0.9 mmol) and  $\text{PhCl}$  (1.0 mL) was stirred at 70 °C under  $\text{N}_2$  atmosphere for 30 min. Then  $t\text{BuNC}$  (68  $\mu\text{L}$ , 0.6 mmol) was added in one portion and phenylacetylene (33  $\mu\text{L}$ , 0.3mmol) in  $\text{PhCl}$  (0.5 mL) was added *via* syringe pump for 3 h. The mixture was kept stirring at 70 °C for another 12 h and afforded the desired product **5d** as a white solid (62.6 mg, 73%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 120-121 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3472, 3314, 3065, 2966, 2872, 2771, 1747, 1640, 1546, 1448, 1389, 1365, 1316, 1225, 1151, 980, 930, 755, 685;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.47 (d,  $J$  = 7.0 Hz, 2H), 7.35-7.26 (m, 3H), 7.25-7.17 (m, 2H), 6.07 (dd,  $J$  = 15.5, 1.5 Hz, 1H), 5.83 (br, 1H), 1.99 (dd,  $J$  = 7.0, 1.5 Hz, 3H), 1.40 (s, 9H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  163.1, 161.9, 149.0, 140.3, 132.6, 129.5, 129.0, 128.6, 122.7, 120.9, 51.5, 28.7, 18.5; LC-MS (ESI)  $m/z$ : 288 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{17}\text{H}_{22}\text{NO}_3$  [ $\text{M}^+\text{H}$ ] 288.1594, found 288.1593.





**(Z)-3-(tert-Butylamino)-3-oxo-1-phenylprop-1-en-2-yl 2-phenylacetate (5e):**

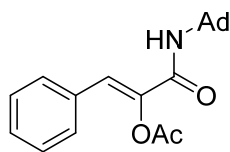
Following the *General Method B*, the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), 2-phenylacetic acid (122 mg, 0.9 mmol) and PhCl (1.0 mL) was stirred at 70 °C under N<sub>2</sub> atmosphere for 30 min. Then <sup>t</sup>BuNC (68 μL, 0.6 mmol) was added in one portion and phenylacetylene (33 μL, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump for 3 h. The mixture was kept stirring at 70 °C for another 12 h and afforded the desired product **5e** as a white solid (48.1 mg, 47%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 132-134 °C. IR (KBr, cm<sup>-1</sup>): 3853, 3742, 3412, 3298, 3064, 2972, 1762, 1700, 1624, 1535, 1448, 1358, 1307, 1216, 1104, 933, 763, 690, 517; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.43-7.32 (m, 7H), 7.31-7.27 (m, 3H), 7.21 (s, 1H), 5.44 (br, 1H), 3.82 (s, 2H), 1.21 (s, 9H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 167.9, 161.4, 140.1, 132.7, 132.5, 129.4, 129.3, 129.2, 129.0, 128.7, 127.9, 123.1, 51.4, 41.7, 28.4; LC-MS (ESI) m/z: 338 [M<sup>+</sup>H]; HRMS (ESI) m/z: calcd for C<sub>21</sub>H<sub>23</sub>NO<sub>3</sub> [M<sup>+</sup>H] 338.1751, found 338.1738.



**(Z)-3-(tert-Butylamino)-3-oxo-1-phenylprop-1-en-2-yl cyclohexanecarboxylate (5f):**

Following the *General Method B*, the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), cyclohexanecarboxylic acid (115 mg, 0.9 mmol) and PhCl (1.0 mL) was

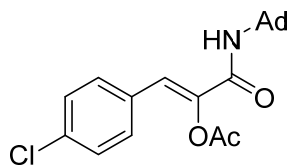
stirred at 70 °C under N<sub>2</sub> atmosphere for 30 min. Then <sup>t</sup>BuNC (68 μL, 0.6 mmol) was added in one portion and phenylacetylene (33 μL, 0.3mmol) in PhCl (0.5 mL) was added *via* syringe pump for 3 h. The mixture was kept stirring at 70 °C for another 12 h and afforded the desired product **5f** as a white solid (64.7 mg, 65%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 79-80 °C. IR (KBr, cm<sup>-1</sup>): 3317, 3063, 2935, 2859, 1751, 1631, 1541, 1450, 1360, 1311, 1224, 1164, 1116, 1018, 929, 754, 692; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.46 (d, *J* = 7.0 Hz, 2H), 7.34 (t, *J* = 7.0 Hz, 2H), 7.29 (t, *J* = 7.0 Hz, 1H), 7.17 (s, 1H), 5.80 (br, 1H), 2.64-2.55 (m, 1H), 2.07-1.97 (m, 2H), 1.87-1.77 (m, 2H), 1.74-1.65 (m, 1H), 1.60-1.50 (m, 2H), 1.49-1.19 (m, 13H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 172.6, 161.9, 140.5, 132.7, 129.4, 128.9, 128.6, 122.6, 51.5, 43.2, 28.9, 28.7, 25.6, 25.3; LC-MS (ESI) *m/z* 330 [M<sup>+</sup>H]; HRMS (ESI) *m/z* calcd for C<sub>20</sub>H<sub>28</sub>NO<sub>3</sub> [M<sup>+</sup>H] 330.2064, found 330.2065.



**(Z)-3-(((3R)-Adamantan-1-yl)amino)-3-oxo-1-phenylprop-1-en-2-ylmacetate (**5g**):**

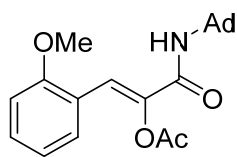
Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), AdNC (96.7 mg, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), phenylacetylene (33 μL, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **5g** was afforded as a yellow solid (79.4 mg, 79%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 169-171 °C. IR (KBr, cm<sup>-1</sup>): 3333, 3064, 2907, 2850, 2658, 1773, 1627, 1535, 1447, 1364, 1305, 1250, 1186, 1130, 1101, 1003, 918, 768, 691, 596, 475; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.47 (d, *J* = 7.5 Hz, 2H), 7.35(t, *J* = 7.0 Hz, 2H), 7.31 (t, *J* = 7.0 Hz, 1H), 7.09 (s, 1H), 5.69 (br, 1H), 2.29 (s, 3H), 2.11 (s, 3H), 2.06 (s, 6H), 1.70 (s, 6H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 167.9, 161.5, 140.8, 132.6, 129.4, 129.0, 128.7, 122.3, 52.3, 41.5, 36.3, 29.4, 20.9; LC-MS (ESI) *m/z*: 362 [M<sup>+</sup>Na]; HRMS (ESI) *m/z*: calcd

for C<sub>21</sub>H<sub>26</sub>NO<sub>3</sub> [M<sup>+</sup>H] 340.1907, found 340.1909.



**(Z)-3-(((3r)-Adamantan-1-yl)amino)-1-(4-chlorophenyl)-3-oxoprop-1-en-2-yl**

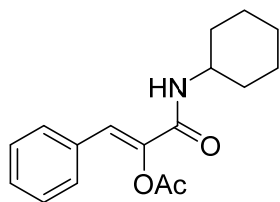
**acetate (5h):** Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), AdNC (96.7 mg, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), 1-chloro-4-ethynylbenzene (42 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **5h** was afforded as a white solid (67.1 mg, 60%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 159-160 °C. IR (KBr, cm<sup>-1</sup>): 3395, 2910, 2854, 1764, 1640, 1448, 1363, 1302, 1246, 1196, 1134, 1091, 1008, 824, 620; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.43 (d, *J* = 8.5 Hz, 2H), 7.35 (d, *J* = 8.5 Hz, 2H), 7.06 (s, 1H), 5.70 (br, 1H), 2.31 (s, 3H), 2.13 (s, 3H), 2.07 (s, 6H), 1.72 (s, 6H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 167.7, 161.2, 141.1, 134.8, 131.1, 130.5, 129.0, 121.1, 52.4, 41.4, 36.3, 29.4, 20.9; LC-MS (ESI) *m/z*: 374 [M<sup>+</sup>H]; HRMS (ESI) *m/z*: calcd for C<sub>21</sub>H<sub>25</sub>NO<sub>3</sub>Cl [M<sup>+</sup>H] 374.1517, found 374.1507.



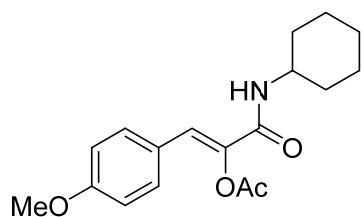
**(Z)-3-(((3r)-Adamantan-1-yl)amino)-1-(2-methoxyphenyl)-3-oxoprop-1-en-2-yl**

**acetate (5i):** Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), AdNC (96.7 mg, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), 1-ethynyl-2-methoxybenzene (41.0 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **5i** was afforded as a white solid (68.0 mg, 61%) after flash column chromatography purification (eluent: petroleum ether/ether acetate =

5:1). M.p. 165-167 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3439, 3327, 3064, 2909, 2852, 1774, 1668, 1634, 1531, 1485, 1443, 1368, 1300, 1249, 1167, 1085, 1016, 758;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.52 (d,  $J$  = 7.5 Hz, 2H), 7.38 (s, 1H), 7.28 (t,  $J$  = 7.5 Hz, 1H), 6.92 (t,  $J$  = 7.0 Hz, 1H), 6.88 (d,  $J$  = 8.5 Hz, 1H), 5.70 (br, 1H), 3.83 (s, 3H), 2.24 (s, 3H), 2.10 (s, 3H), 2.07 (s, 6H), 1.70 (s, 6H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  168.1, 161.7, 157.5, 140.8, 130.3, 129.2, 121.6, 120.4, 116.8, 110.8, 55.6, 52.2, 41.5, 36.3, 29.5, 20.9; LC-MS (ESI)  $m/z$ : 370 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{22}\text{H}_{28}\text{NO}_4$  [ $\text{M}^+\text{H}$ ] 370.2013, found 370.1997.

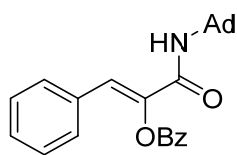


**(Z)-3-(Cyclohexylamino)-3-oxo-1-phenylprop-1-en-2-yl acetate (5j):** Following the *General Method A*, to the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol),  $\text{Ag}_2\text{O}$  (104.3 mg, 0.45 mmol), isocyanocyclohexane (65 mg, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), phenylacetylene (33  $\mu\text{L}$ , 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **5j** was afforded as a yellow solid (38.3 mg, 44%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 146-147 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3908, 3328, 3092, 3032, 2935, 2854, 2665, 1765, 1667, 1633, 1530, 1445, 1371, 1325, 1289, 1255, 1190, 1121, 1076, 1011, 894, 759, 685;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.49 (d,  $J$  = 7.0 Hz, 2H), 7.36 (t,  $J$  = 7.0 Hz, 2H), 7.34-7.30 (m, 1H), 7.16 (s, 1H), 5.84 (d,  $J$  = 8.0 Hz, 1H), 3.92-3.82 (m, 1H), 2.30 (s, 3H), 2.01-1.94 (m, 2H), 1.76-1.69 (m, 2H), 1.67-1.60 (m, 1H), 1.46-1.35 (m, 2H), 1.25-1.14 (m, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  167.9, 161.7, 140.2, 132.5, 129.4, 129.1, 128.7, 123.1, 48.6, 33.0, 25.5, 24.8, 20.9; LC-MS (ESI)  $m/z$ : 288 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{17}\text{H}_{22}\text{NO}_3$  [ $\text{M}^+\text{H}$ ] 288.1594, found 288.1595.



**(Z)-3-(Cyclohexylamino)-1-(2-methoxyphenyl)-3-oxoprop-1-en-2-yl acetate (5k):**

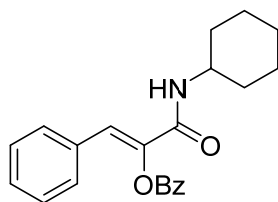
Following the *General Method A*, to the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), isocyanocyclohexane (68.0 mg, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol) and PhCl (1.0 mL), 1-ethynyl-4-methoxybenzene (40.0 mg, 0.3 mmol) in PhCl (0.5 mL) was added *via* syringe pump at 30 °C for 3 h. The reaction was kept stirring at 30 °C for another 2 h, and the desired product **5k** was afforded as a white solid (50.4 mg, 53%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 185-186 °C. IR (KBr, cm<sup>-1</sup>): 3305, 3041, 2931, 2850, 1754, 1619, 1524, 1452, 1377, 1337, 1252, 1213, 1175, 1126, 1082, 1025, 951, 879, 832, 679, 531; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 7.45 (d, *J* = 8.8 Hz, 2H), 7.12 (s, 1H), 6.88 (d, *J* = 8.8 Hz, 2H), 5.83 (d, *J* = 7.7 Hz, 1H), 3.90-3.83 (m, 1H), 3.81 (s, 3H), 2.30 (s, 3H), 1.97-1.94 (m, 2H), 1.73-1.68 (m, 4H), 1.41-1.38 (m, 2H), 1.19-1.17 (m, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 168.1, 162.0, 160.3, 138.7, 131.2, 125.2, 123.0, 114.3, 55.4, 48.6, 33.2, 25.6, 24.9, 21.0; LC-MS (ESI) *m/z*: 318 [M<sup>+</sup>H]; HRMS (ESI) *m/z*: calcd for C<sub>18</sub>H<sub>24</sub>NO<sub>4</sub> [M<sup>+</sup>H] 318.1700, found 318.1689



**(Z)-3-(((3r)-Adamantan-1-yl)amino)-3-oxo-1-phenylprop-1-en-2-yl benzoate (5l):**

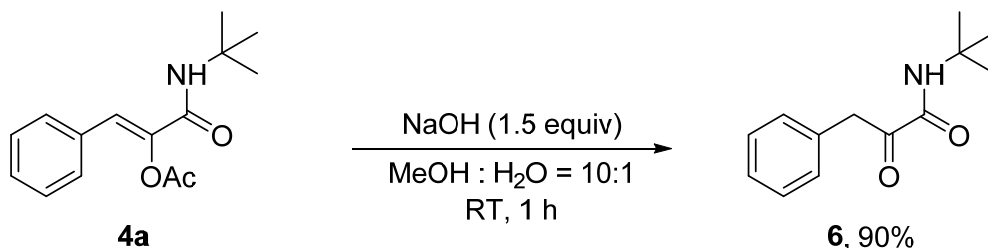
Following the *General Method B*, the mixture of Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), benzoic acid (110.0 mg, 0.9 mmol) and PhCl (1.0 mL) was stirred at 70 °C under N<sub>2</sub> atmosphere for 30 min. Then AdNC (96.7 mg, 0.6 mmol) was added in one portion and phenylacetylene (33 μL, 0.3mmol) in PhCl (0.5 mL) was added *via* syringe pump for 3 h. The mixture was kept stirring at 70 °C for another 12 h and afforded the

desired product **5l** as a white solid (88.1 mg, 73%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 164-166 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3268, 3057, 2907, 2852, 1732, 1630, 1538, 1448, 1354, 1308, 1248, 1099, 1055, 700;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  8.19 (d,  $J = 7.5$  Hz, 2H), 7.68 (t,  $J = 7.5$  Hz, 1H), 7.54 (t,  $J = 8.0$  Hz, 2H), 7.52-7.49 (m, 2H), 7.29-7.25 (m, 4H), 5.75 (br, 1H), 2.08 (s, 3H), 2.04 (s, 6H), 1.68 (s, 6H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  163.6, 161.4, 140.5, 134.2, 132.5, 130.3, 129.5, 129.0, 128.9, 128.7, 128.4, 122.9, 52.3, 41.4, 36.3, 29.4; LC-MS (ESI)  $m/z$ : 402 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{26}\text{H}_{28}\text{NO}_3$  [ $\text{M}^+\text{H}$ ] 402.2064, found 402.2046.

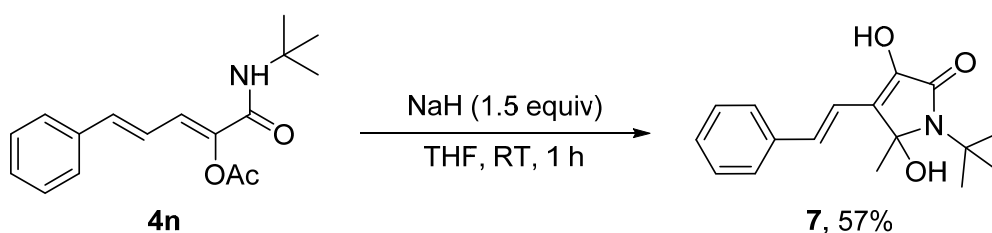


**(Z)-3-(Cyclohexylamino)-3-oxo-1-phenylprop-1-en-2-yl benzoate (5m):** Following the *General Method B*, the mixture of  $\text{Pd}(\text{OAc})_2$  (3.4 mg, 0.015 mmol),  $\text{Ag}_2\text{O}$  (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), benzoic acid (110.0 mg, 0.9 mmol) and  $\text{PhCl}$  (1.0 mL) was stirred at 70 °C under  $\text{N}_2$  atmosphere for 30 min. Then isocyanocyclohexane (65.0 mg, 0.6 mmol) was added in one portion and phenylacetylene (33.0  $\mu\text{L}$ , 0.3 mmol) in  $\text{PhCl}$  (0.5 mL) was added *via* syringe pump for 3 h. The mixture was kept stirring at 70 °C for another 12 h and afforded the desired product **5m** as a white solid (77.0 mg, 74%) after flash column chromatography purification (eluent: petroleum ether/ether acetate = 5:1). M.p. 158-159 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3739, 3306, 3062, 2927, 2853, 1743, 1631, 1533, 1449, 1329, 1248, 1133, 1084, 1050, 923, 754, 695;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  8.20 (d,  $J = 7.5$  Hz, 2H), 7.70 (t,  $J = 7.5$  Hz, 1H), 7.60-7.48 (m, 4H), 7.34 (s, 1H), 7.31-7.25 (m, 3H), 5.89 (d,  $J = 7.0$  Hz, 1H), 3.96-3.83 (m, 1H), 1.99-1.92 (m, 2H), 1.73-1.64 (m, 2H), 1.64-1.55 (m, 2H), 1.44-1.31 (m, 2H), 1.20-1.08 (m, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  163.6, 161.7, 140.0, 134.3, 132.4, 130.3, 129.6, 129.1, 129.0, 128.7, 128.4, 123.7, 48.6, 32.9, 25.5, 24.7; LC-MS (ESI)  $m/z$ : 350 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{22}\text{H}_{24}\text{NO}_3$  [ $\text{M}^+\text{H}$ ] 350.1751, found 350.1742.

### 3. Further Transformation of Compounds 4a and 4n

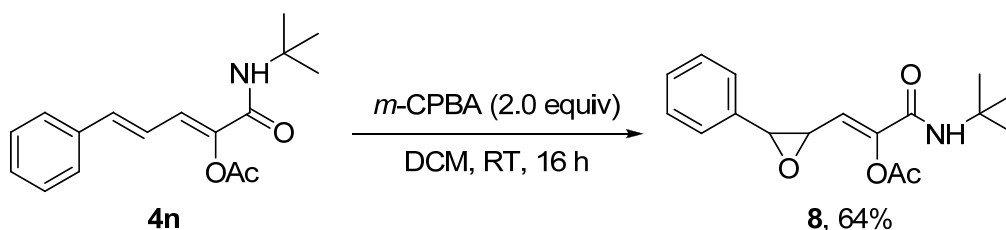


***N*-(*tert*-Butyl)-2-oxo-3-phenylpropanamide (6):**<sup>2</sup> To a test tube were added **4a** (26.1 mg, 0.1 mmol), NaOH (6.0 mg, 0.15 mmol), MeOH/H<sub>2</sub>O (10:1, 1.10 mL). The mixture was stirred at room temperature for 1 h. Upon the completion of the reaction, the solvent was removed under reduced pressure. The residue was diluted with ethyl acetate and washed with brine. The organic phase was then dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent, the crude product was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) to give the pure compound **6** as a white solid (21.7 mg, 90%). M.p. 47-48 °C. IR (KBr, cm<sup>-1</sup>): 3387, 2925, 2859, 1685, 1604, 1520, 1459, 1402, 1364, 1224, 1084, 825, 705, 603. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  7.33 (t, *J* = 7.5 Hz, 2H), 7.29-7.22 (m, 3H), 6.82 (br, 1H), 4.20 (s, 2H), 1.38 (s, 9H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta$  197.1, 159.2, 133.0, 129.8, 128.6, 127.1, 51.4, 42.5, 28.3; LC-MS (ESI) *m/z*: 220 [M<sup>+</sup>H].



**(*E*)-1-(*tert*-Butyl)-3,5-dihydroxy-5-methyl-4-styryl-1,5-dihydro-2*H*-pyrrol-2-one (7):** To a test tube were added **4n** (86.0 mg, 0.3 mmol), NaH (60% dispersion in mineral oil, 18 mg, 0.45 mmol) and THF (3.0 mL). The mixture was stirred at room temperature for 1 h under N<sub>2</sub> atmosphere. Upon the completion of the reaction, the solvent was removed under reduced pressure. The residue was diluted with ethyl acetate and washed with 5% NH<sub>4</sub>Cl aqueous and brine. The organic phase was then dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent, the crude product was

purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 2:1) to give the pure compound **7** as a white solid (49.0 mg, 57%). M.p. 69-70 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3661, 3620, 3172, 2923, 2858, 1665, 1525, 1365, 1079, 976, 926, 784, 688;  $^1\text{H}$  NMR (Acetone- $\text{d}_6$ , 500 MHz):  $\delta$  8.68 (br, 1H), 7.54 (d,  $J = 7.5$  Hz, 2H), 7.35 (t,  $J = 7.5$  Hz, 2H), 7.25 (t,  $J = 7.5$  Hz, 1H), 7.19 (d,  $J = 16.5$  Hz, 1H), 6.88 (d,  $J = 16.5$  Hz, 1H), 5.03 (br, 1H), 1.80 (s, 3H), 1.62 (s, 9H);  $^{13}\text{C}$  NMR (Acetone- $\text{d}_6$ , 125 MHz):  $\delta$  166.2, 143.3, 138.8, 133.0, 129.6, 128.4, 127.0, 123.7, 117.7, 91.0, 56.6, 29.3, 27.3; LC-MS (ESI)  $m/z$ : 288 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{17}\text{H}_{22}\text{NO}_3$  [ $\text{M}^+\text{H}$ ] 288.1594, found 288.1593.

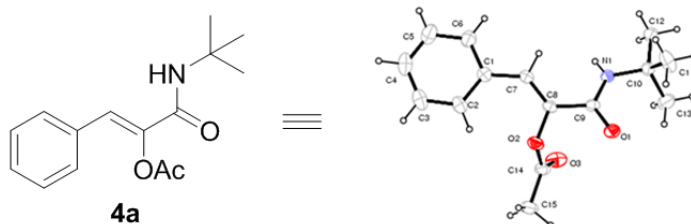


**(Z)-3-(tert-Butylamino)-3-oxo-1-(3-phenyloxiran-2-yl)prop-1-en-2-yl acetate (**8**):**

To a test tube were added **4n** (57.4 mg, 0.2 mmol), *m*-CPBA (70% purity, 98.6 mg, 0.4 mmol) and DCM (2.0 ml). The mixture was stirred at room temperature for 16 h. Upon the completion of the reaction, the mixture was filtered. The filtrate was concentrated and purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) to give the pure compound **8** as a white solid (38.9 mg, 64%). M.p. 149-151 °C. IR (KBr,  $\text{cm}^{-1}$ ): 3291, 3068, 2970, 2924, 2859, 1766, 1637, 1547, 1458, 1366, 1320, 1213, 1165, 1088, 1012, 947, 895, 853, 756, 696;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.39-7.32 (m, 3H), 7.27 (d,  $J = 6.5$  Hz, 2H), 6.08 (d,  $J = 7.5$  Hz, 1H), 5.75 (br, 1H), 3.89 (d,  $J = 1.5$  Hz, 1H), 3.45 (dd,  $J = 8.0, 2.0$  Hz, 1H), 2.23 (s, 3H), 1.38 (s, 9H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  167.9, 160.4, 145.2, 136.1, 128.6, 128.5, 125.5, 121.1, 59.6, 57.2, 51.7, 28.6, 20.5; LC-MS (ESI)  $m/z$ : 304 [ $\text{M}^+\text{H}$ ]; HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{17}\text{H}_{22}\text{NO}_4$  [ $\text{M}^+\text{H}$ ] 304.1543, found 304.1539.

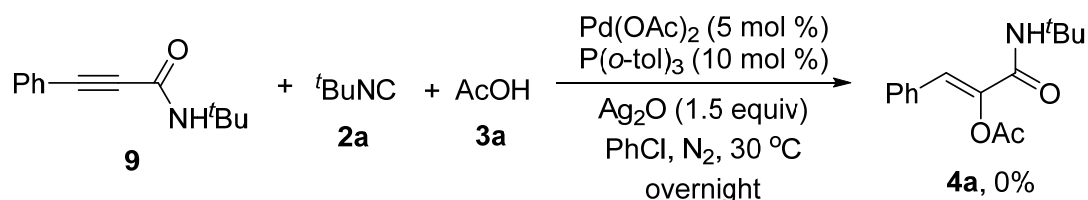


## 4. X-Ray Crystallographic Analysis for Compound 4a

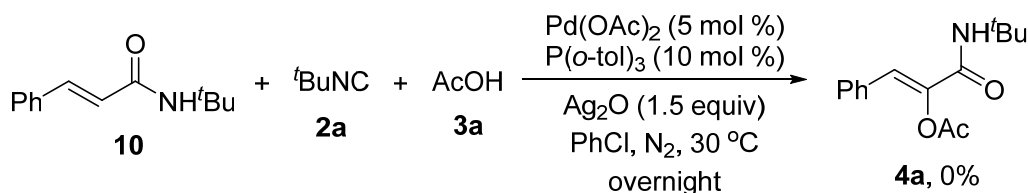


Crystallographic data for compound **4a**: C<sub>15</sub>H<sub>19</sub>NO<sub>3</sub>, M = 261.31, Orthorhombic F 2dd (No. 43), a = 32.25 (3) Å, b = 17.433 (18) Å, c = 10.608 (11) Å, V = 5963 (11) Å<sup>3</sup>, Z = 16, Crystal size: 0.29 × 0.21 × 0.14 mm, T = 295 K, ρ<sub>calcd</sub> = 1.164 g·cm<sup>-3</sup>, R<sub>1</sub> = 0.0374 (I > 4σ(I)), wR<sub>2</sub> = 0.1118 (all data), GOF = 1.044, reflections collected/unique: 9066 / 3273 (R<sub>int</sub> = 0.0175), Data: 2623, restraints: 0, parameters: 177. CCDC 1836502 contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

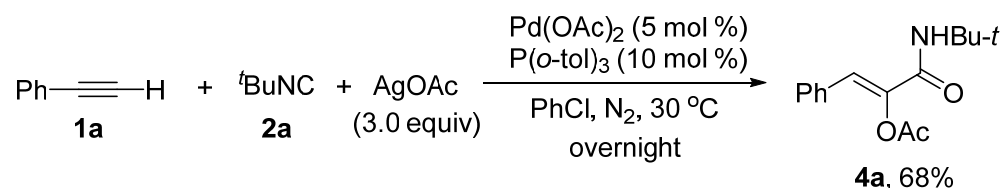
## 5. Mechanistic Studies



To a test tube were added Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), *t*BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol), *N*-(*tert*-butyl)-3-phenylpropiolamide **9** (38.7 mg, 0.3 mmol) and PhCl (1.5 mL). The reaction was degassed and refilled with N<sub>2</sub> atmosphere for three times. After the mixture was stirred at 30 °C overnight, no desired product could be observed from the reaction.



To a test tube were added Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), Ag<sub>2</sub>O (104.3 mg, 0.45 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), *t*BuNC (68 μL, 0.6 mmol), AcOH (54.0 mg, 0.9 mmol), *N*-(*tert*-butyl)cinnamamide **10** (61 mg, 0.3 mmol) and PhCl (1.5 mL). The reaction was degassed and refilled with N<sub>2</sub> atmosphere for three times. After the mixture was stirred at 30 °C overnight, no desired product could be observed from the reaction.

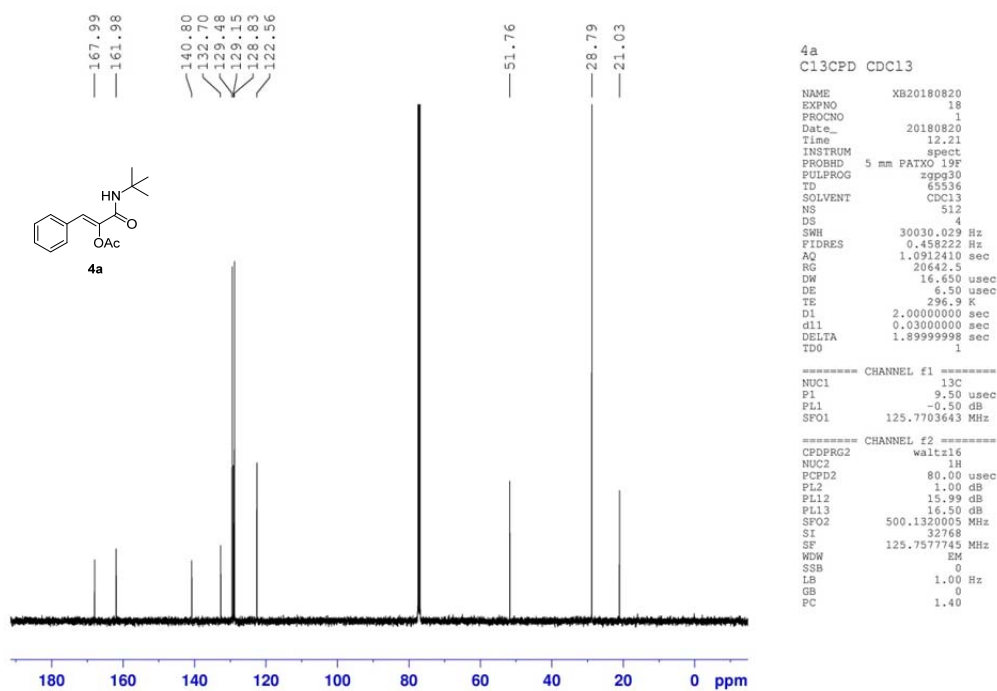
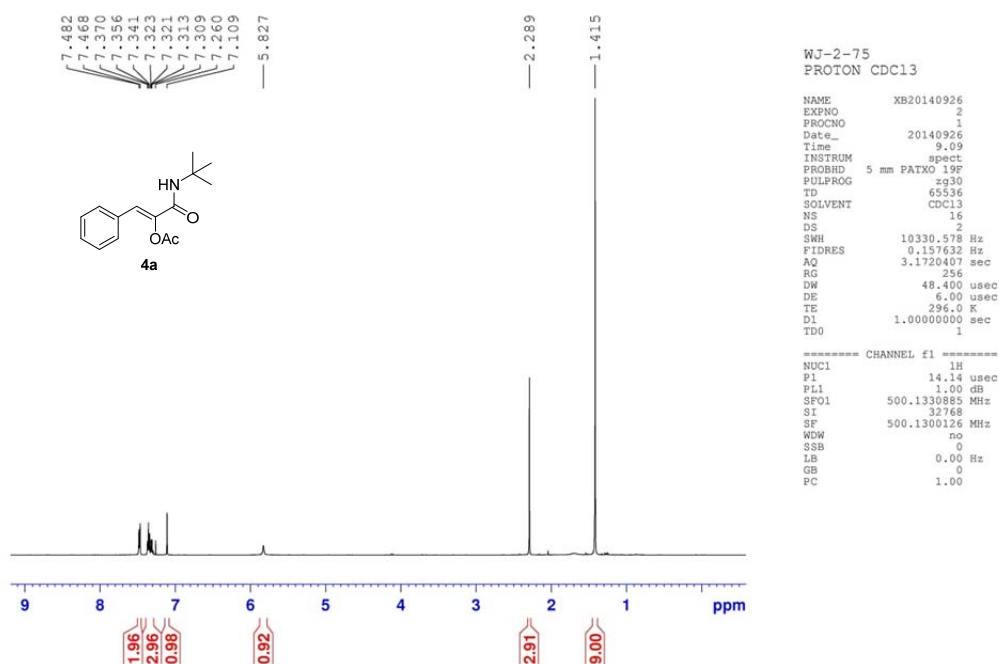


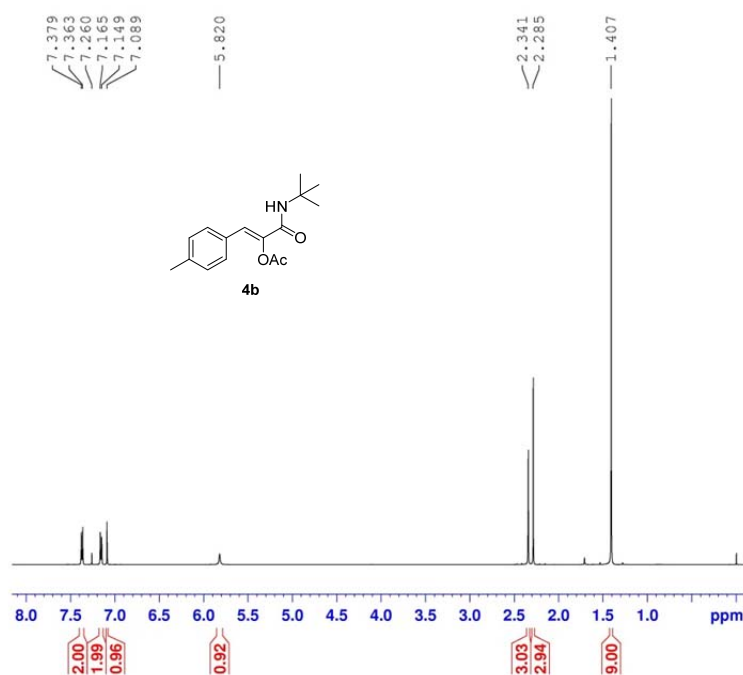
To a test tube were added Pd(OAc)<sub>2</sub> (3.4 mg, 0.015 mmol), AgOAc (150.2 mg, 0.9 mmol), tri(2-methylphenyl)phosphine (9.1 mg, 0.03 mmol), *t*BuNC (68 μL, 0.6 mmol) and PhCl (1.0 mL). The mixture was stirred at 30 °C under N<sub>2</sub> atmosphere. Phenylacetylene **1a** (33 μL, 0.3 mmol) in PhCl (0.5 mL) was then added *via* syringe pump for 3 h. The reaction was kept stirring at 30 °C for another 2 h. Upon completion monitored by TLC, the reaction was cooled down to room temperature and filtered. The filtrate was washed with H<sub>2</sub>O and brine. The aqueous phase was then extracted with ethyl acetate (3 × 10 mL) and the combined organic phase was dried over Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent under reduced pressure, the crude product was purified by flash column chromatography on silica gel to give the pure product **4a** (52.4 mg, 68%).

## 6. Reference

1. Peng, J.; Zhao, J.; Hu, Z.; Liang, D.; Huang, J.; Zhu, Q. *Org. Lett.* **2012**, *14*, 4966.
2. Steuer, C.; Gege, C.; Fischl, W.; Heinonen, K. H.; Bartenschlager, R.; Klein, C. D. *Bioorg. Med. Chem.* **2011**, *19*, 4067.

## 7. Copies of $^1\text{H}$ , $^{13}\text{C}$ and $^{19}\text{F}$ NMR Spectra for All Compounds





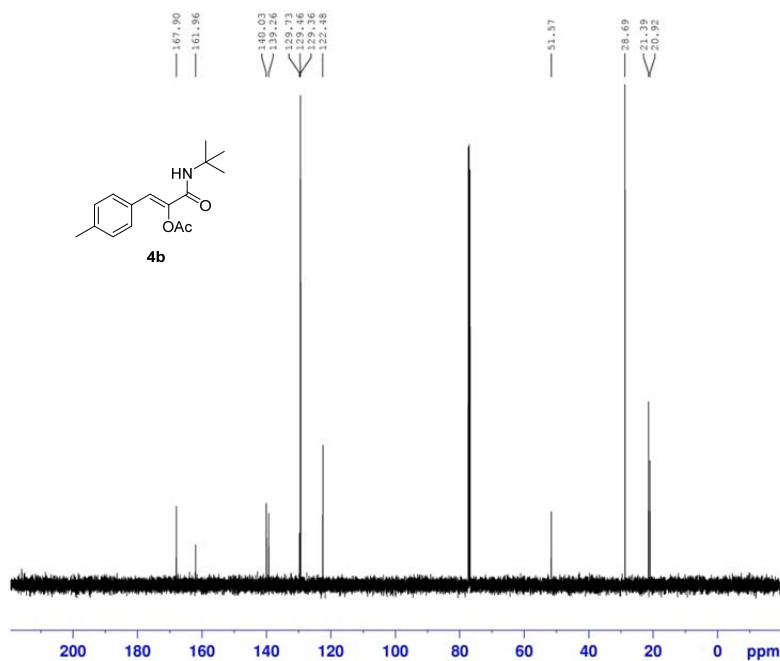
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PROTON CDC13

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TD         65536
SOLVENT   CDC13
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         143.7
DW         48.400 usec
DE         6.00 usec
TE         296.3 K
D1         1.00000000 sec
TD0        1

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NUC1       1H
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PL1        0.00 dB
SFO1       500.1330885 MHz
SI         32768
SF         500.1300126 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

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WJ-3-77-1  
C13CPD CDC13

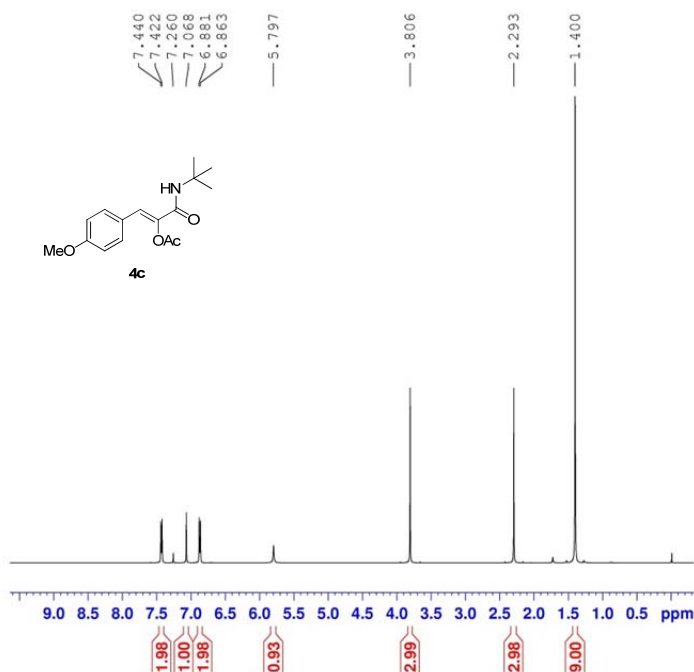
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SOLVENT   CDC13
NS         128
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FIDRES     0.458222 Hz
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RG         287.4
DW         16.650 usec
DE         6.00 usec
TE         297.5 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999998 sec
TD0        1

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PL1        -0.50 dB
SFO1       125.7703643 MHz

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NUC2       1H
PCPD2     80.00 usec
PL2        1.00 dB
PL12       16.05 dB
PL13       16.50 dB
SFO2       500.1320005 MHz
SI         32768
SF         125.7577890 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

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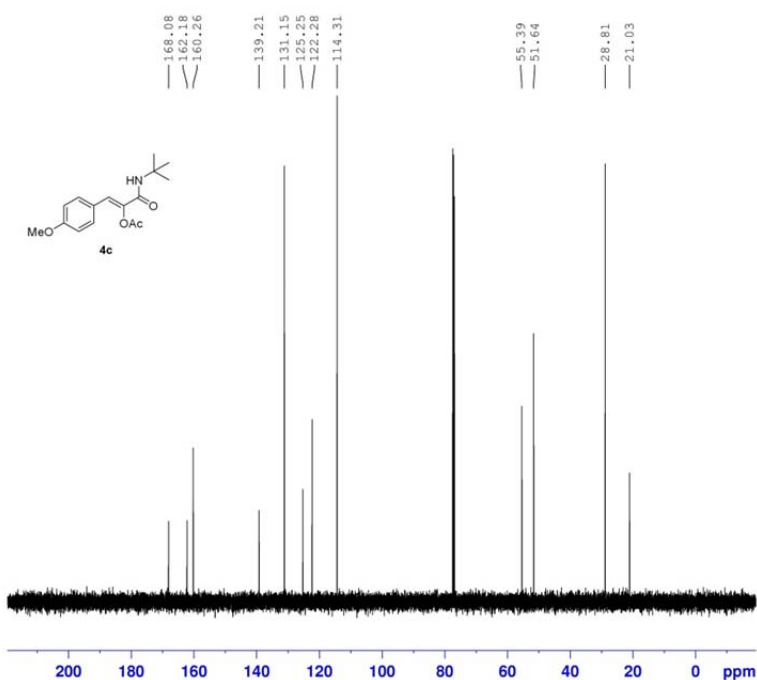
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PROTON CDC13

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TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         143.7
DW         48.400 usec
DE         6.00 usec
TE         296.3 K
D1         1.0000000 sec
D10        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.14 usec
PL1       1.00 dB
SFO1      500.130885 MHz
SI        32768
SF        500.1300126 MHz
WDW        no
SSB        0
LB         0.00 Hz
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PC         1.00
  
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WJ-3-77-2  
C13CPD CDC13

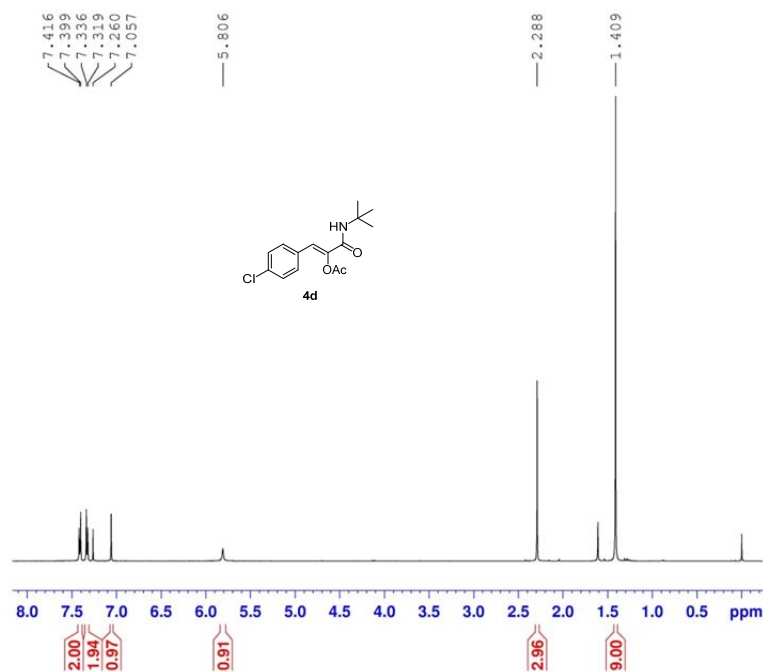
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PROCNO    1
Date_     20150313
Time      2.30
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TD         65536
SOLVENT   CDCl3
NS         128
DS         4
SWH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912410 sec
RG         203.2
DW         16.650 usec
DE         6.00 usec
TE         297.6 K
D1         2.0000000 sec
d11        0.0300000 sec
DELTA     1.89999998 sec
D10        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         9.50 usec
PL1       -0.50 dB
SFO1      125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        1.00 dB
PL12       16.05 dB
PL13       16.50 dB
SFO2      500.1320005 MHz
SI        32768
SF        125.7577754 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.40
  
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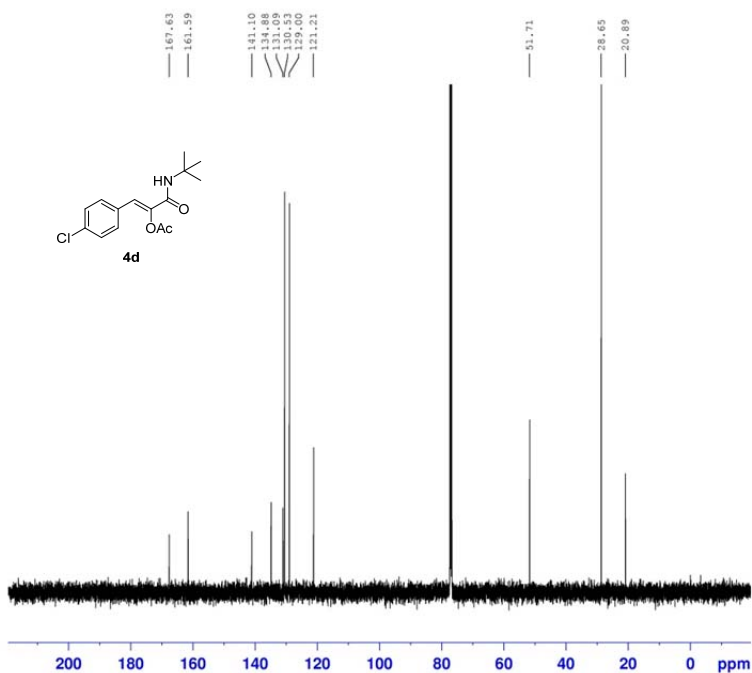
WJ-3-102-1  
PROTON CDC13

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SOLVENT   CDC13
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         287.4
DW         48.400 usec
DE         6.00 usec
TE         296.5 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.14 usec
PL1        1.00 dB
SFO1       500.1330885 MHz
SI         32768
SF         500.1300126 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

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WJ-3-102-1  
C13CPD CDC13

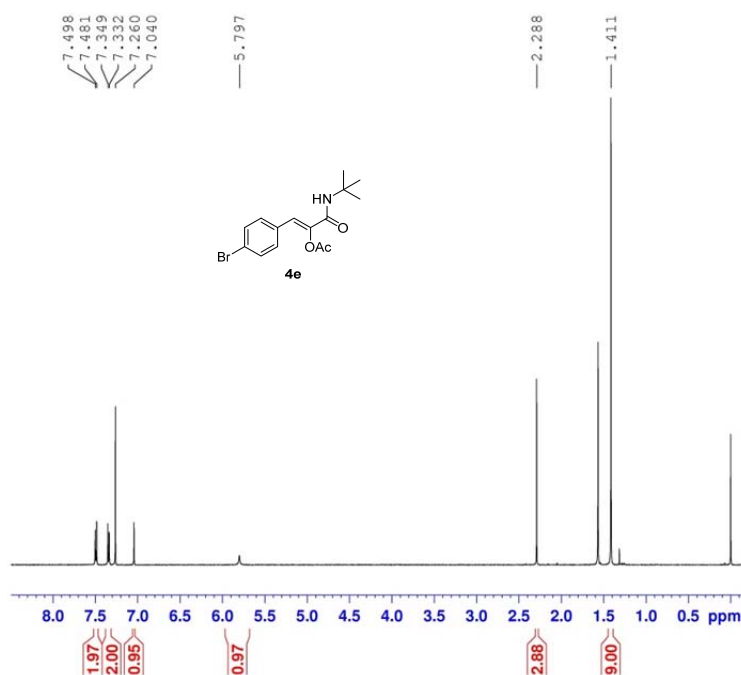
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SOLVENT   CDC13
NS         512
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FIDRES     0.458222 Hz
AQ         1.0912410 sec
RG         374.1
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DE         6.00 usec
TE         298.2 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999998 sec
TD0        1

===== CHANNEL f1 =====
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P1         9.50 usec
PL1        -0.50 dB
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
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NUC2       1H
PCPD2      80.00 usec
PL2        1.00 dB
PL12       16.05 dB
PL13       16.50 dB
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GB         0
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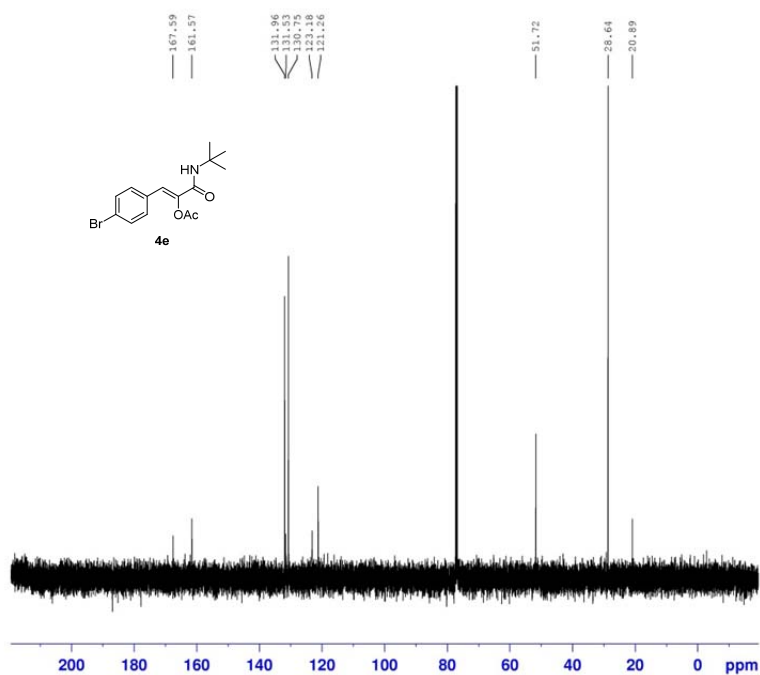
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WJ-3-102-2  
PROTON CDC13

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EXPNO 2  
PROCNO 1  
Date\_ 20150414  
Time 9.45  
INSTRUM spect  
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PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 2  
SWH 10330.578 Hz  
FIDRES 0.157632 Hz  
AQ 3.1720407 sec  
RG 406.4  
DW 48.400 usec  
DE 6.00 usec  
TE 296.5 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.14 usec  
PL1 1.00 dB  
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WDW no  
SSB 0  
LB 0.00 Hz  
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PC 1.00

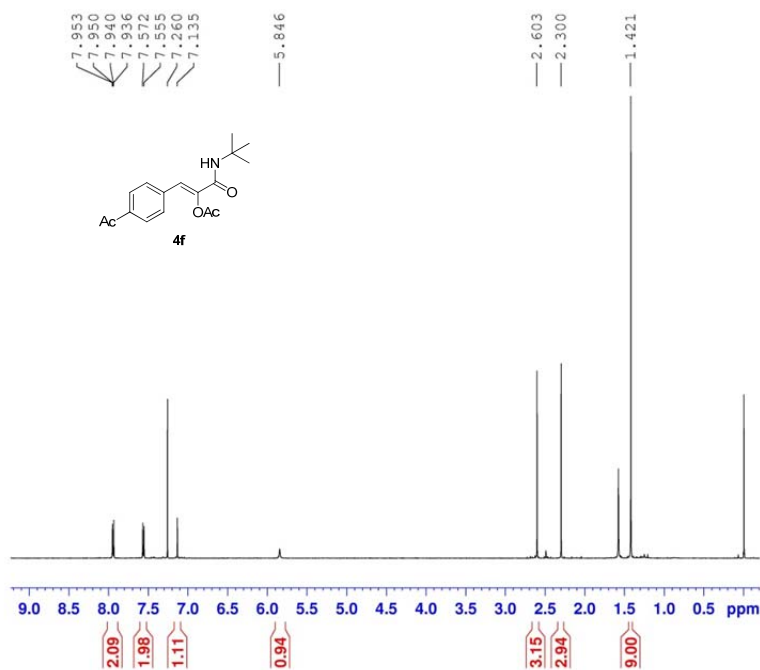


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Date\_ 20150423  
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INSTRUM spect  
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PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 256  
DS 4  
SWH 30030.029 Hz  
FIDRES 0.458222 Hz  
AQ 1.0912410 sec  
RG 181  
DW 16.650 usec  
DE 6.00 usec  
TE 297.9 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.50 dB  
SFO1 125.7703643 MHz

===== CHANNEL f2 =====  
CPOPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 1.00 dB  
PL12 16.05 dB  
PL13 16.50 dB  
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WDW no  
SSB 0  
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GB 0  
PC 1.40

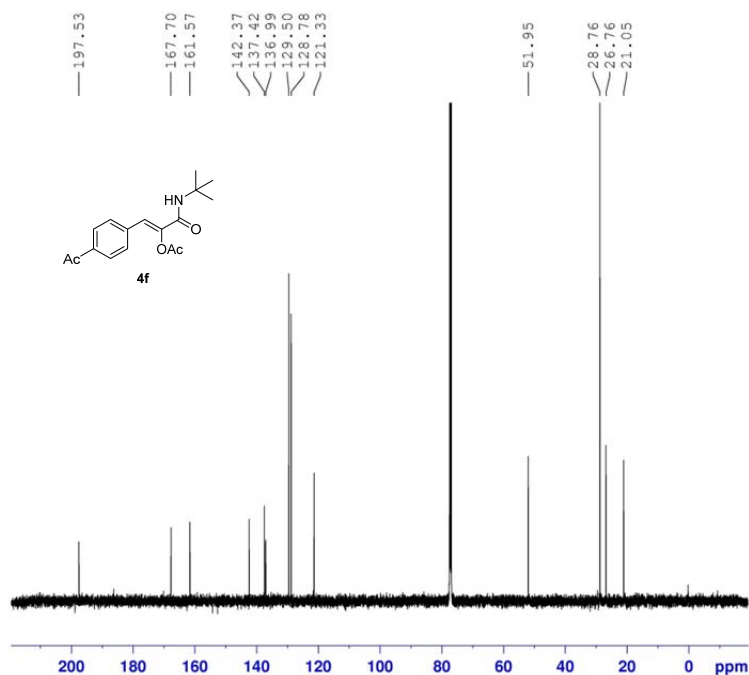


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SOLVENT	CDCl <sub>3</sub>
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DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	362
DW	48.400 usec
DE	6.00 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



ZMF-4F  
C13CPD CDCl<sub>3</sub>

NAME	XB20180820
EXPNO	53
PROCNO	1
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PULPROG	zgpg30
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FIDRES	0.458222 Hz
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RG	23170.5
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DELTA	1.89999998 sec
TD0	1

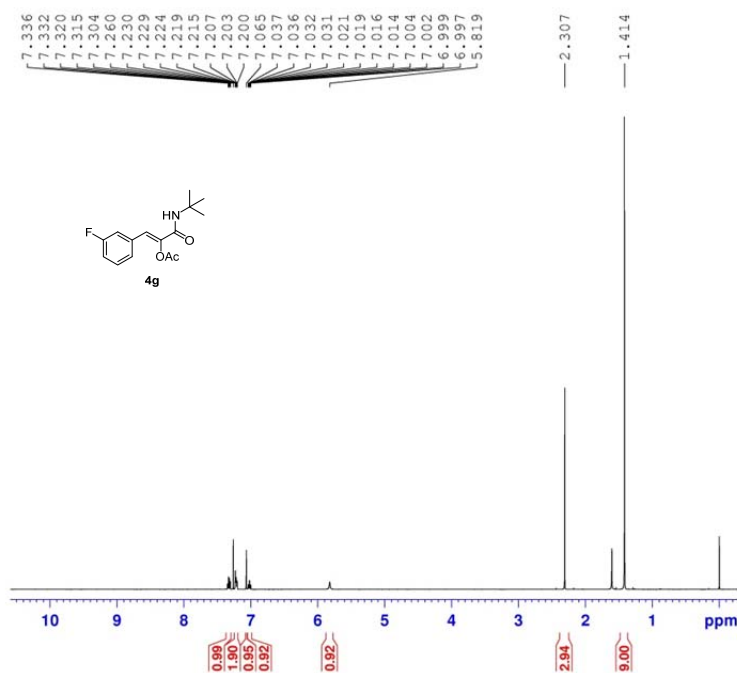
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===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
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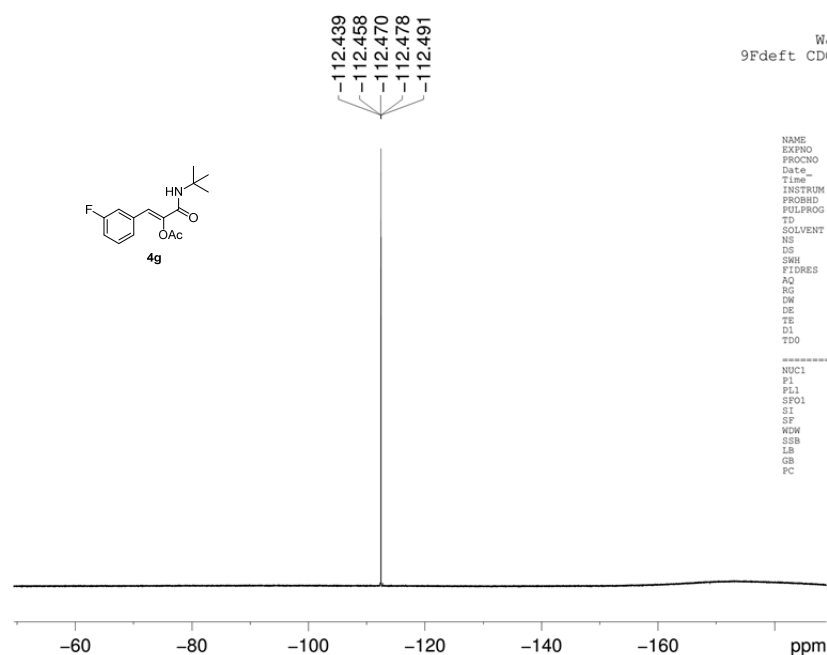
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FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         362
DW         48.400 usec
DE         6.50 usec
TE         295.2 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
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PL1        1.00 dB
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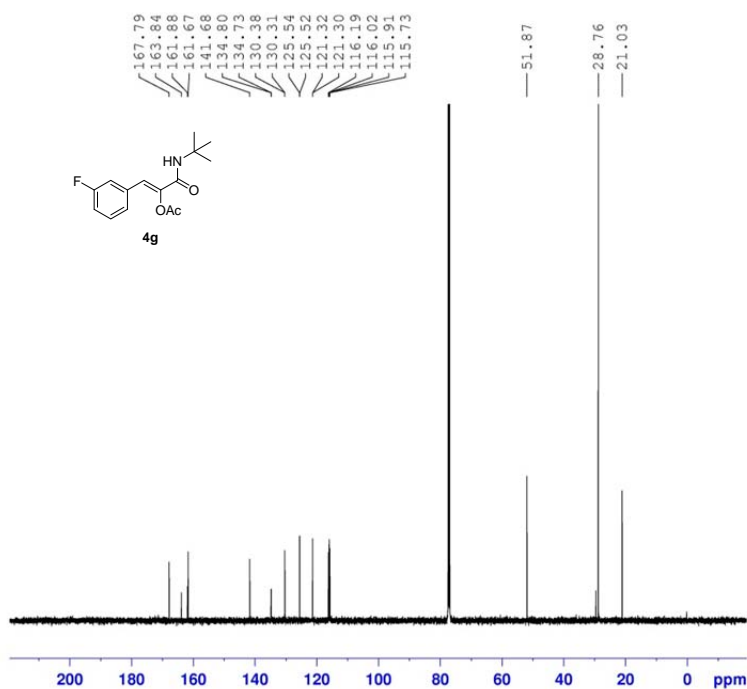
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PULPROG   zg
TD         131072
SOLVENT   CDCl3
NS         16
DS         4
SWH        100000.000 Hz
FIDRES     0.762939 Hz
AQ         0.6554150 sec
RG         287.4
DW         5.000 usec
DE         6.00 usec
TE         295.6 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       19F
P1         19.30 usec
PL1        4.00 dB
SFO1       470.5453180 MHz
SI         65536
SF         470.5923770 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

```



ZMF-2-2  
C13CPD CDC13 C

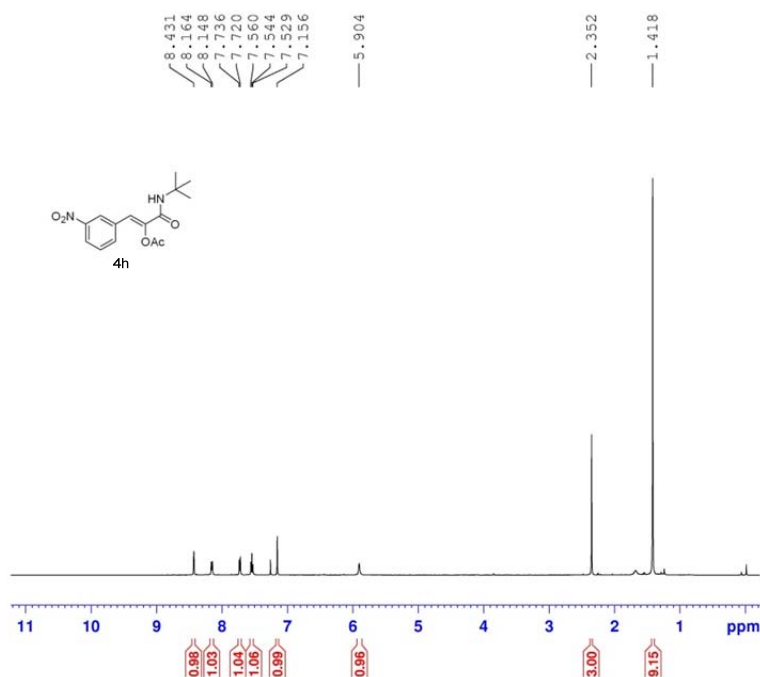
NAME	XB20180822
EXPNO	53
PROCNO	1
Date_	20180823
Time	9.35
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	1636
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	23170.5
DW	16.650 usec
DE	6.50 usec
TE	297.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999999 sec
TD0	1

===== CHANNEL f1 =====

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	15.99 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577727 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

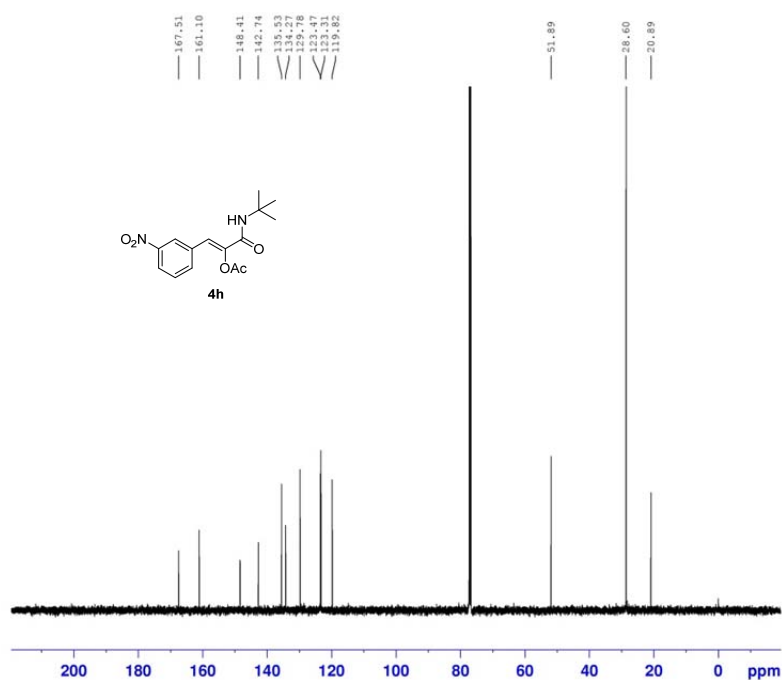


WJ-3-119  
PROTON CDC13

NAME	1h
EXPNO	1
PROCNO	1
Date_	20150707
Time	18.19
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	203.2
DW	48.400 usec
DE	6.00 usec
TE	295.0 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	EM
SSB	0
LB	0.70 Hz
GB	0
PC	1.00



WJ-3-119  
C13CPD CDCl3

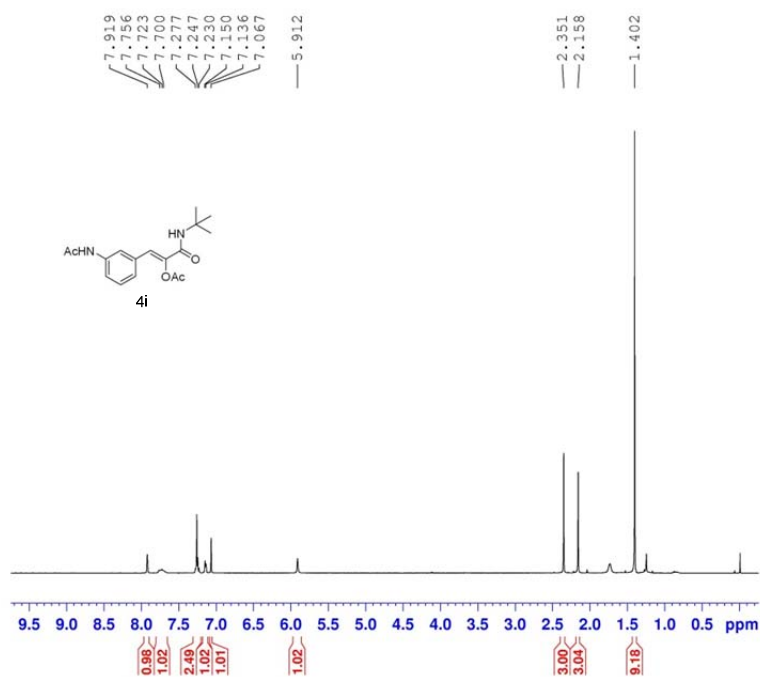
NAME	XB20150710
EXPNO	1
PROCNO	1
Date_	20150710
Time	9.17
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.498222 Hz
AQ	1.0912410 sec
RG	114
DW	16.650 usec
DE	6.00 usec
TE	297.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

===== CHANNEL f1 =====

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

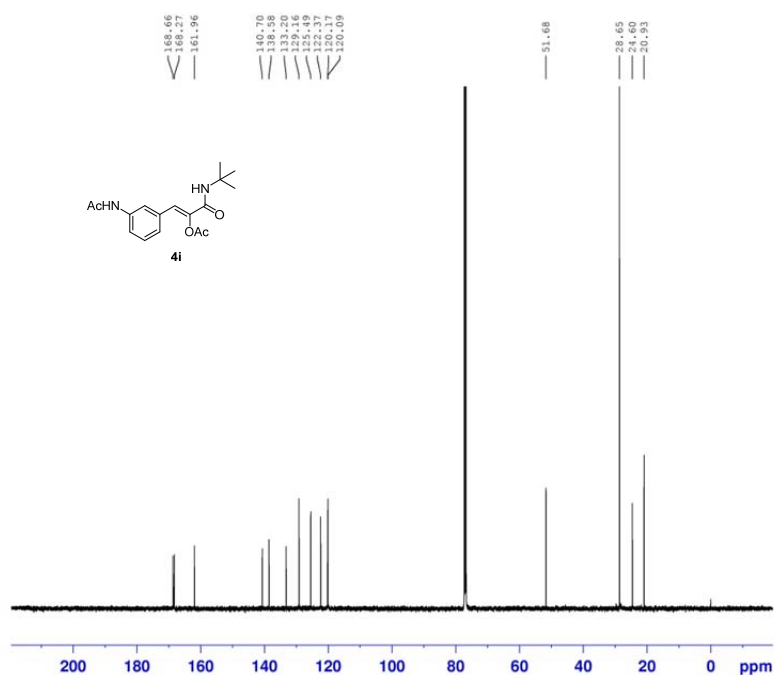


WJ-3-134-P2  
PROTON CDCl3

NAME	1i
EXPNO	1
PROCNO	1
Date_	20150706
Time	22.36
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	256
DW	48.400 usec
DE	6.00 usec
TE	295.4 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.50



WJ-3-134  
C13CPD CDC13

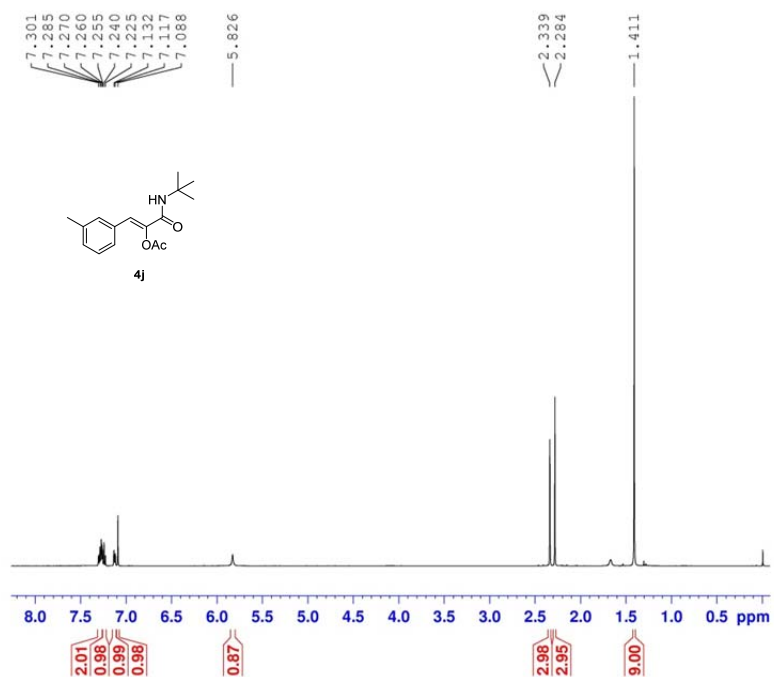
NAME	XB20150917
EXPNO	12
PROCNO	1
Date_	20150918
Time	5.09
INSTRUM	spect
PROBHD	5 mm PATXO 19P
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl <sub>3</sub>
NS	3300
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	228.1
DW	16.650 usec
DE	6.00 usec
TE	297.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

===== CHANNEL f1 =====

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

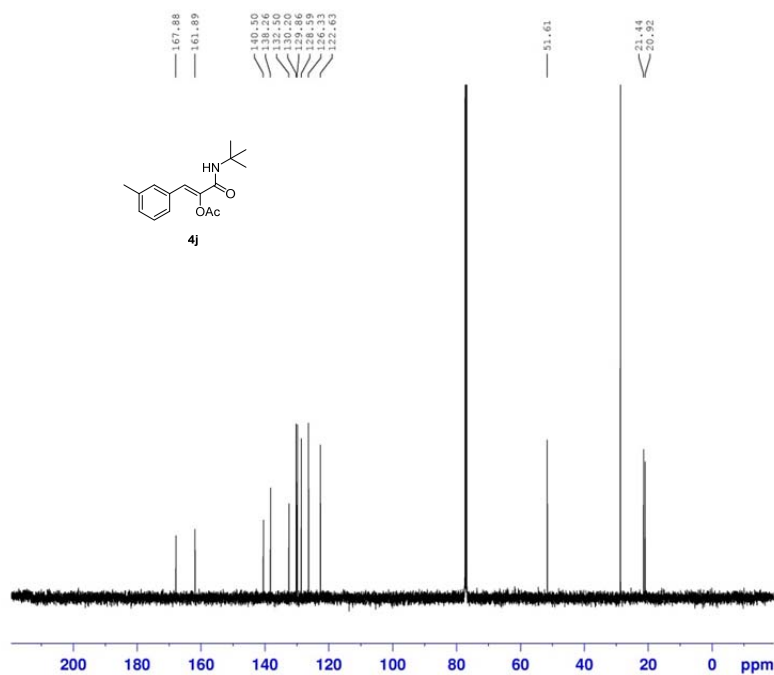


WJ-3-126  
PROTON CDC13

NAME	XB20150602
EXPNO	14
PROCNO	1
Date_	20150602
Time	15.40
INSTRUM	spect
PROBHD	5 mm PATXO 19P
PULPROG	zg30
TD	65536
SOLVENT	CDCl <sub>3</sub>
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	181
DW	48.400 usec
DE	6.00 usec
TE	295.9 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300127 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



WJ-3-126  
C13CPD CDC13

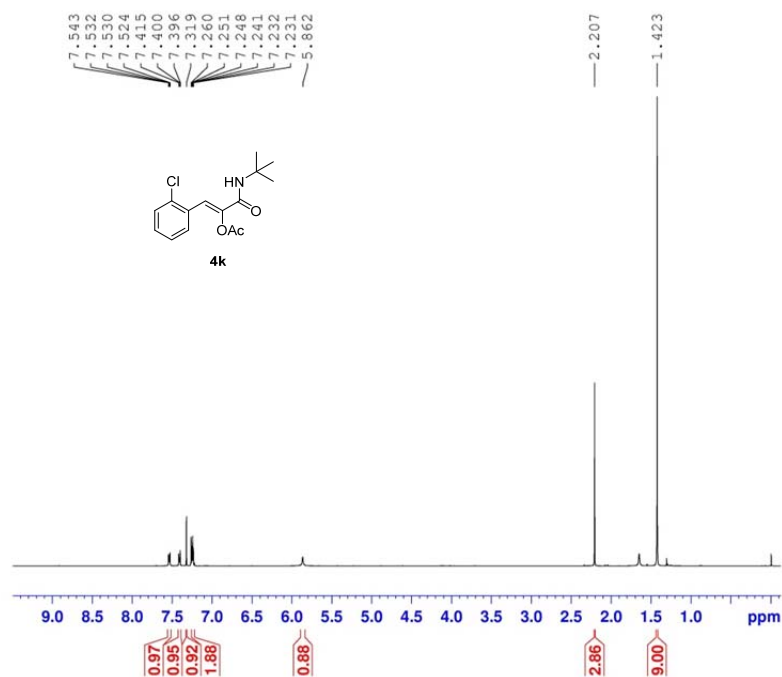
NAME	XB20150613
EXPNO	1
PROCNO	1
Date_	20150614
Time	0.14
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	256
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	101.6
DW	16.650 usec
DE	6.00 usec
TE	295.3 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999999 sec
TD0	1

===== CHANNEL f1 =====

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

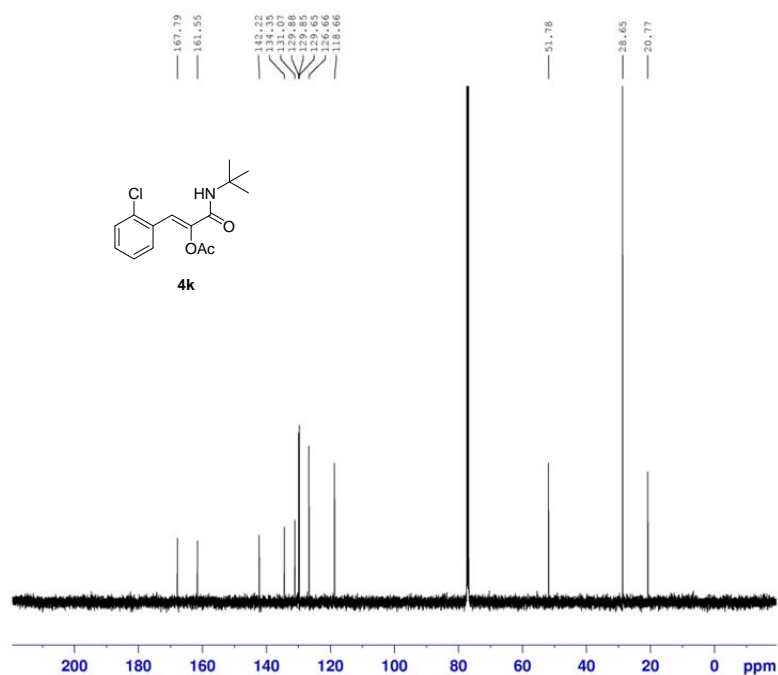


WJ-3-144  
PROTON CDC13

NAME	XB20150907
EXPNO	3
PROCNO	1
Date_	20150907
Time	10.35
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	203.2
DW	48.400 usec
DE	6.00 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



WJ-3-144  
C13CPD CDC13

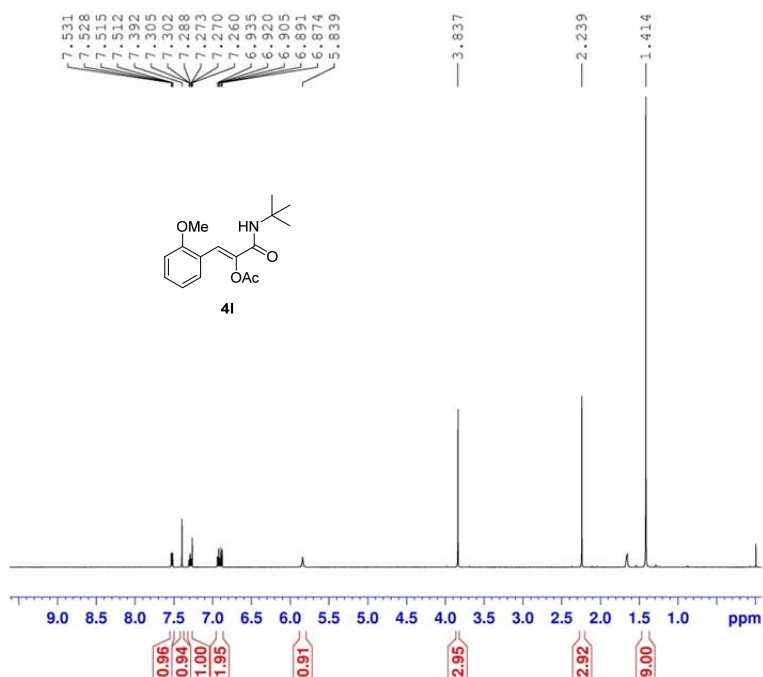
NAME	XB20150908
EXPNO	4
PROCNO	1
Date_	20150908
Time	16.47
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	128
DW	16.650 usec
DE	6.00 usec
TE	297.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

===== CHANNEL f1 =====

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EK
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

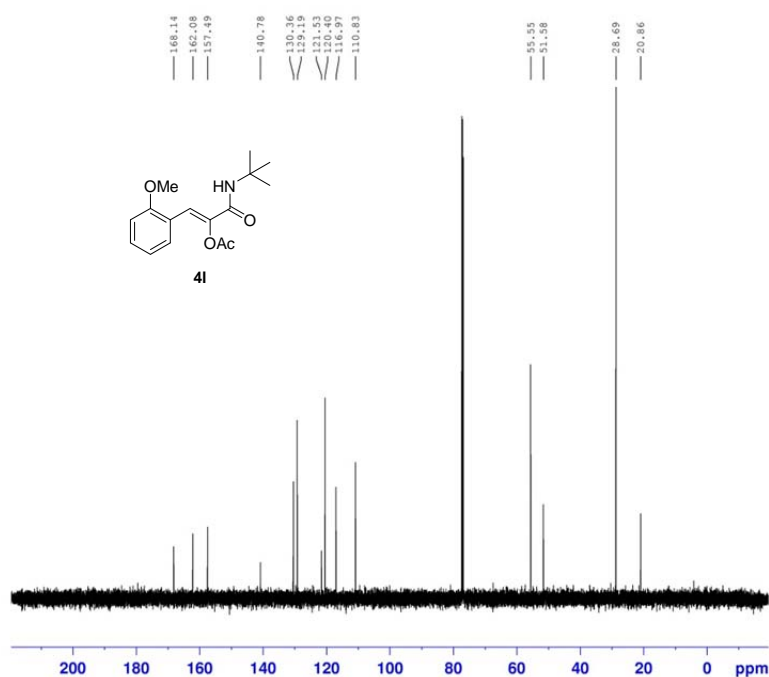


WJ-3-125  
PROTON CDC13

NAME	XB20150602
EXPNO	13
PROCNO	1
Date_	20150602
Time	15.34
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	228.1
DW	48.400 usec
DE	6.00 usec
TE	295.3 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300130 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



WJ-3-125  
C13CPD CDC13

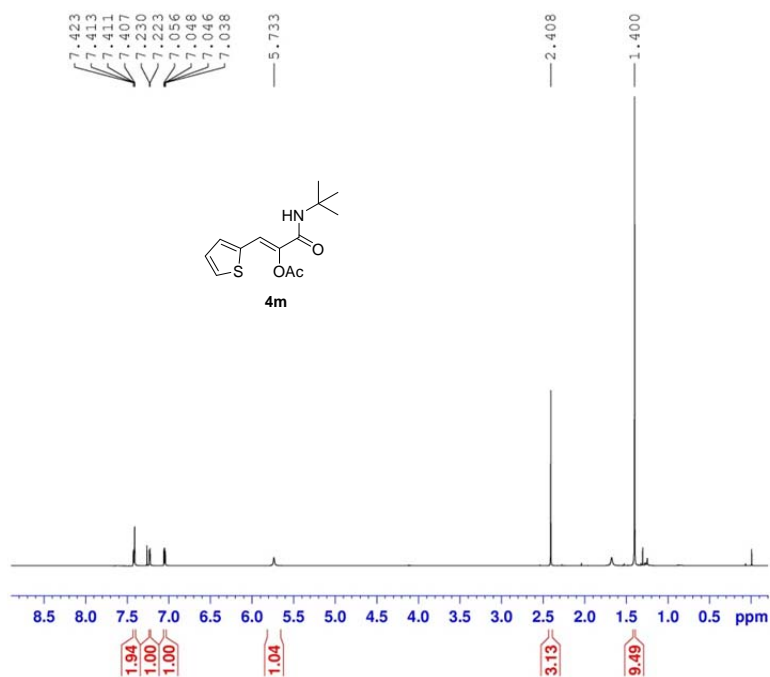
NAME	XB20150612
EXPNO	20
PROCNO	1
Date_	20150612
Time	16.27
INSTRUM	spect
PROBHD	5 mm PATXO 19T
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	256
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	101.6
DW	16.650 usec
DE	6.00 usec
TE	296.9 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

===== CHANNEL f1 =====

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

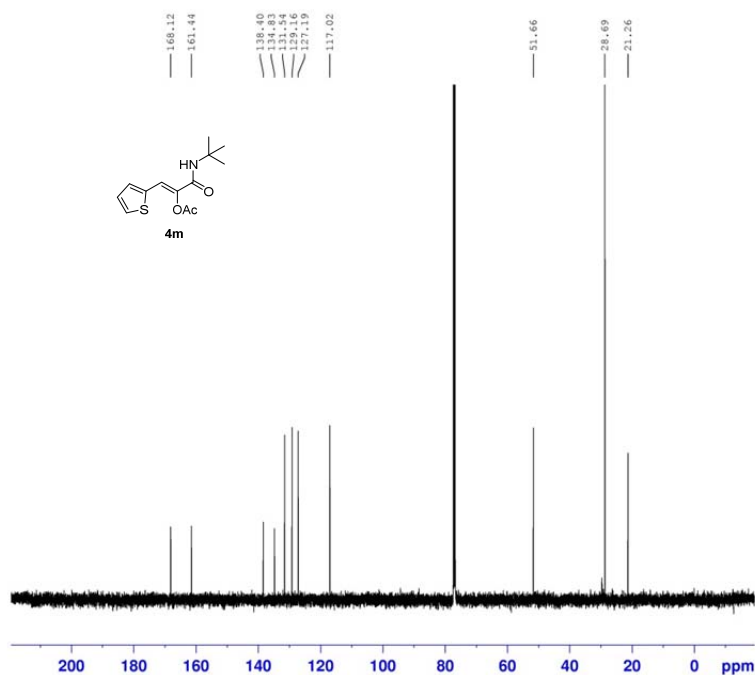


WJ-3-130  
PROTON CDC13

NAME	XB20150824
EXPNO	2
PROCNO	1
Date_	20150824
Time	10.37
INSTRUM	spect
PROBHD	5 mm PATXO 19T
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	203.2
DW	48.400 usec
DE	6.00 usec
TE	295.3 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



WJ-3-130  
C13CPD CDC13

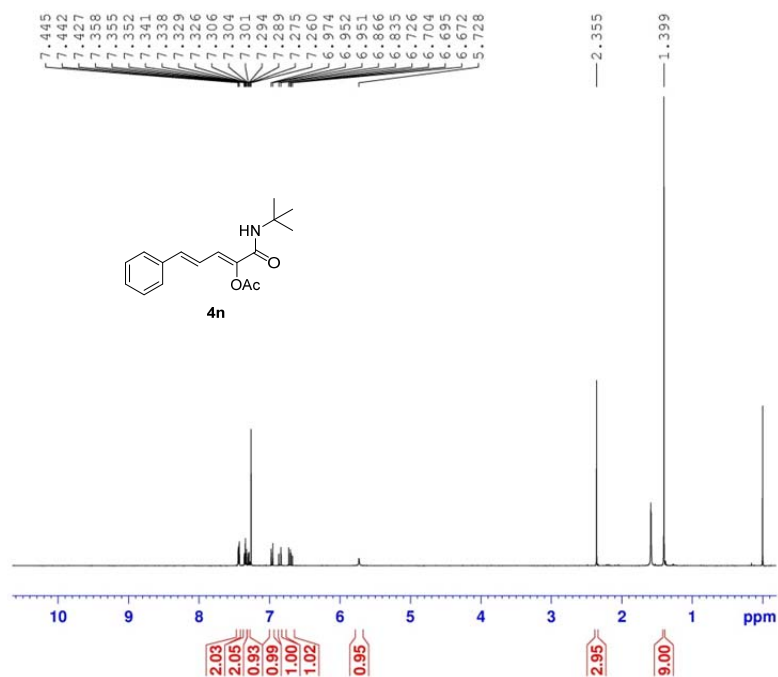
NAME	XB20150824
EXPNO	11
PROCNO	1
Date_	20150824
Time	13.31
INSTRUM	spect
PROBHD	5 mm PATXO 19P
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	512
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	128
DW	16.650 usec
DE	6.00 usec
TE	297.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

===== CHANNEL f1 =====

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



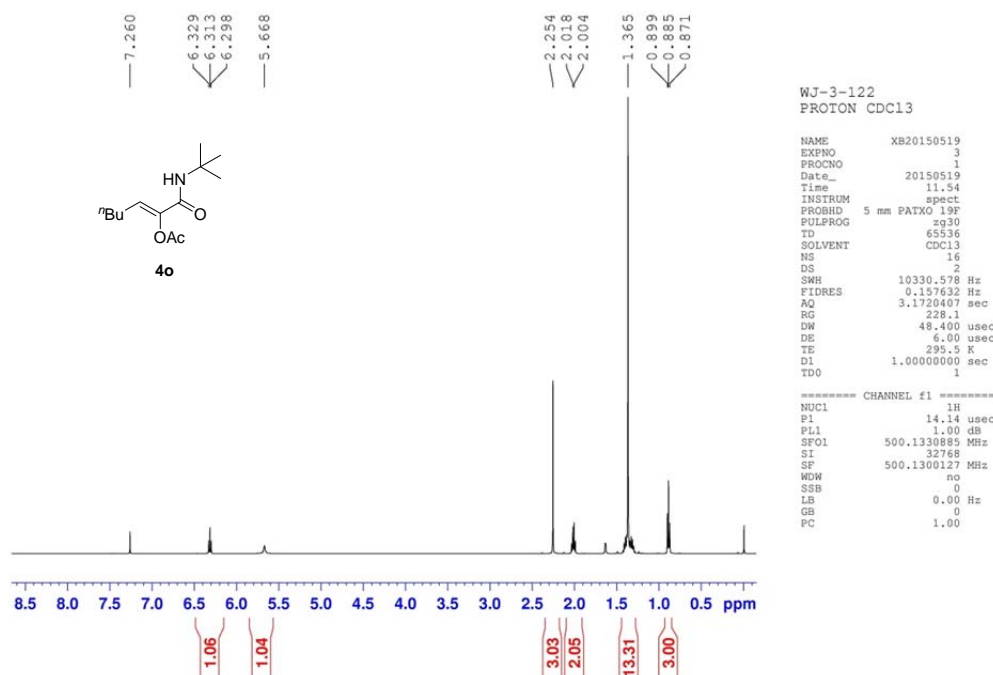
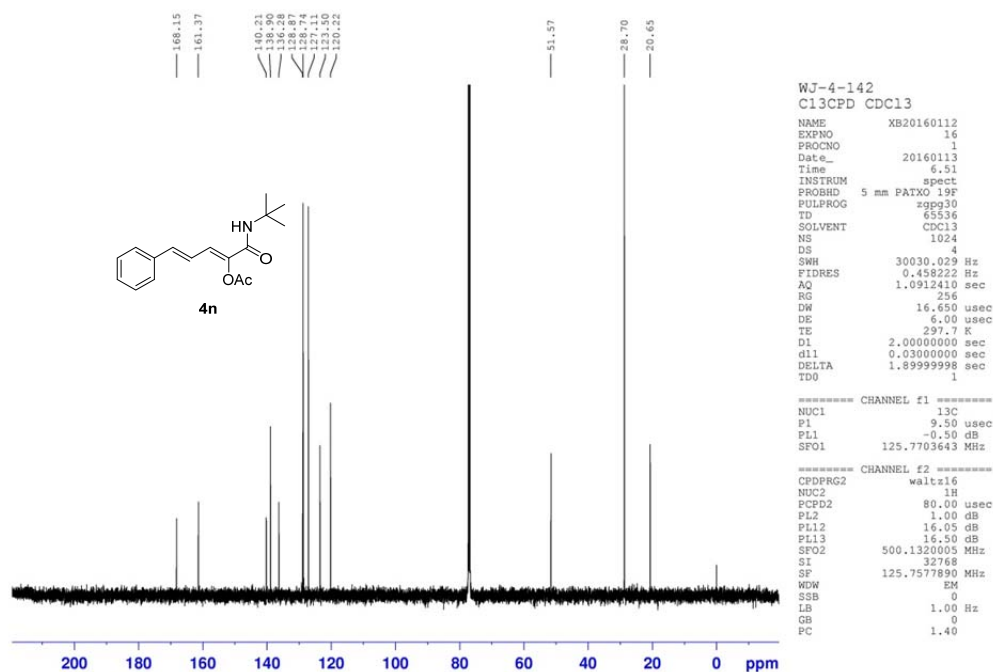
ZMF-2-5  
PROTON CDC13

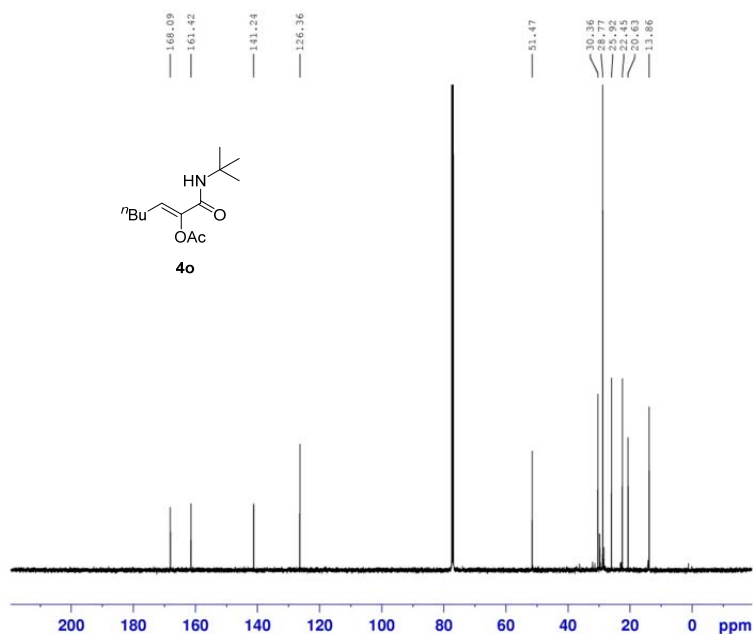
NAME	XB20180827
EXPNO	10
PROCNO	1
Date_	20180827
Time	16.06
INSTRUM	spect
PROBHD	5 mm PATXO 19P
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	362
DW	48.400 usec
DE	6.50 usec
TE	295.6 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.24 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300135 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00







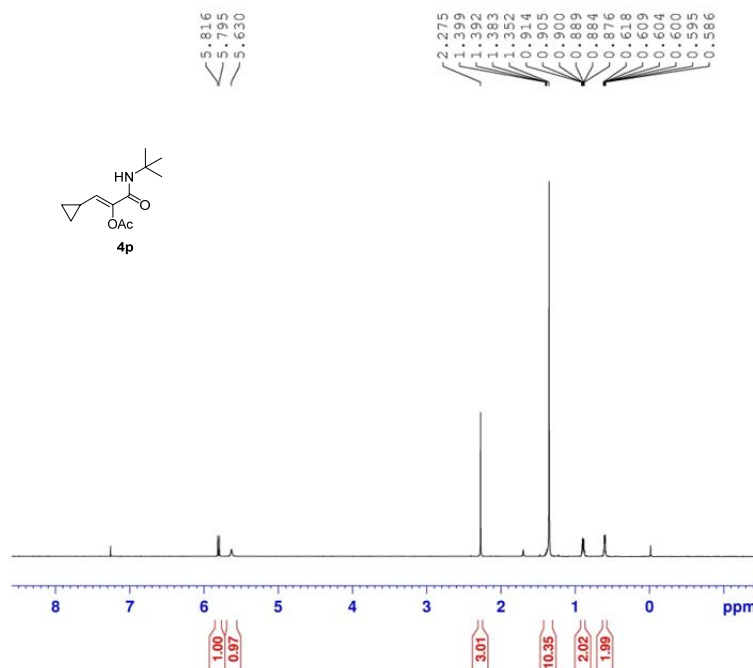
**4o**  
C13CPD CDC13

```

NAME       XB20180913
EXPNO      6
PROCNO     1
Date_      20180913
Time       13.28
INSTRUM    spect
PROBHD     5 mm PATXO 19F
PULPROG    zgpg30
TD         65536
SOLVENT    CDC13
NS         1024
DS         4
SWH         30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912410 sec
RG         20642.5
DW         16.650 usec
DE         6.50 usec
TE         297.8 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.50 usec
PL1        -0.50 dB
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2         1.00 dB
PL12       15.99 dB
PL13       16.50 dB
SFO2       500.1320005 MHz
SI         32768
SF         125.7577745 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

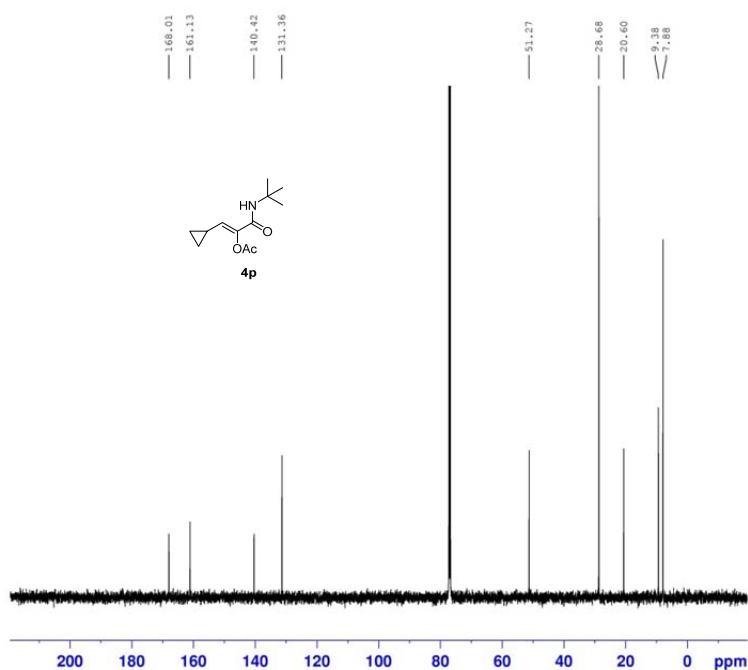


**WJ-3-123**  
PROTON CDC13

```

NAME       1n
EXPNO      1
PROCNO     1
Date_      20150518
Time       21.00
INSTRUM    spect
PROBHD     5 mm PATXO 19F
PULPROG    zg30
TD         65536
SOLVENT    CDC13
NS         16
DS         2
SWH         10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         161.3
DW         48.400 usec
DE         6.00 usec
TE         295.4 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.14 usec
PL1         1.00 dB
SFO1       500.1330883 MHz
SI         32768
SF         500.1300129 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
  
```



WJ-3-123  
C13CPD CDC13

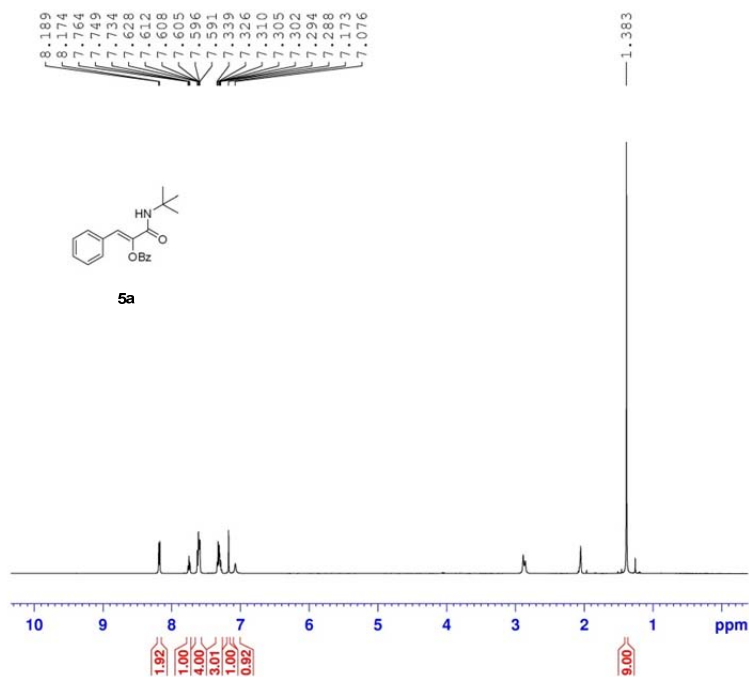
NAME	XB20150519
EXPNO	12
PROCNO	1
Date_	20150519
Time	15.26
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	256
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	287.4
DW	16.650 usec
DE	6.00 usec
TE	296.8 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

===== CHANNEL f1 =====

NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

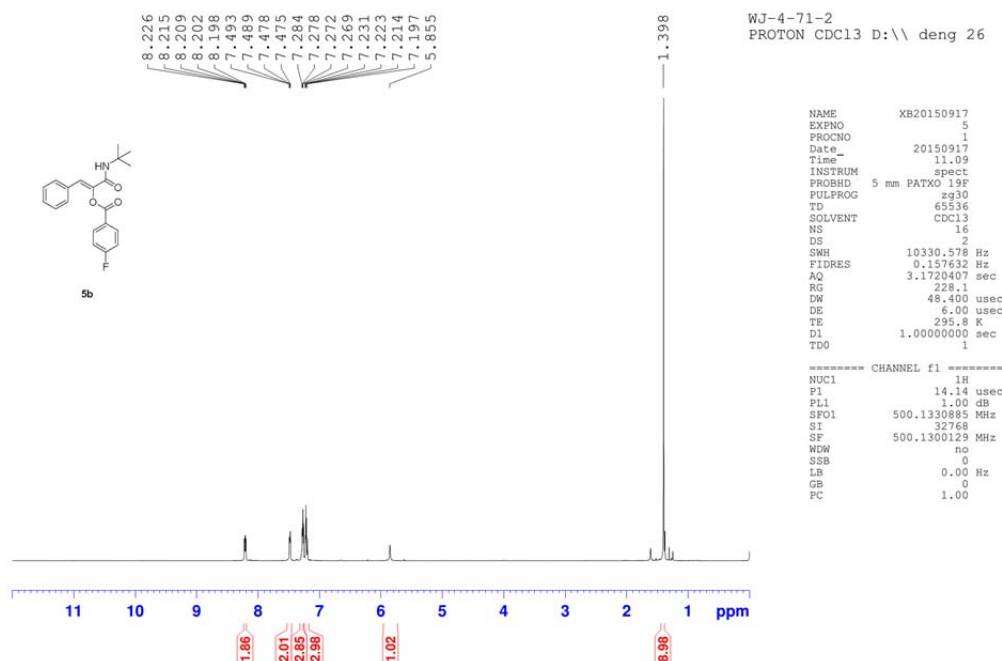
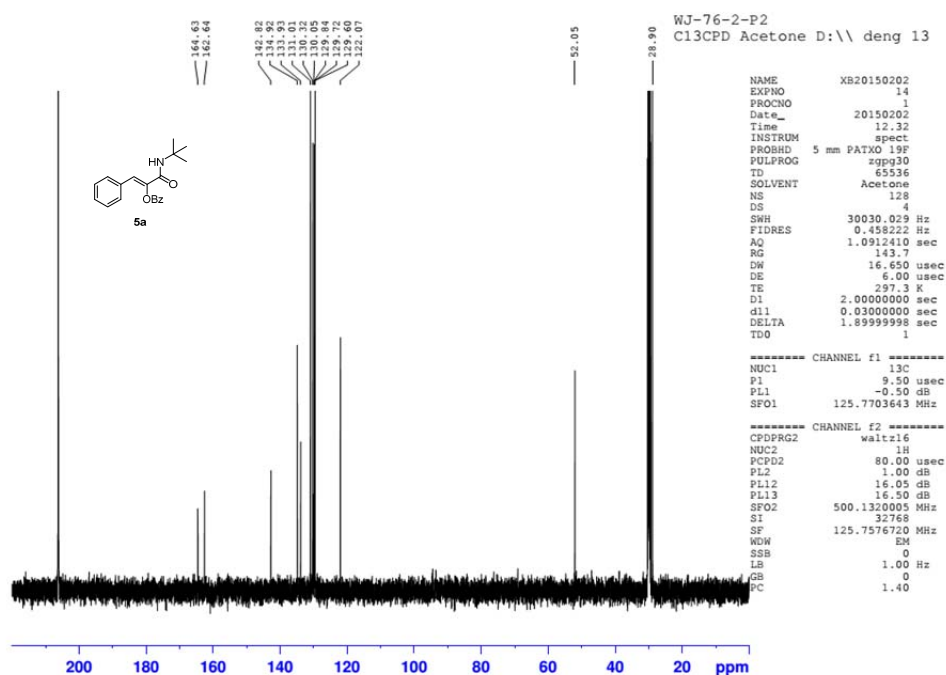


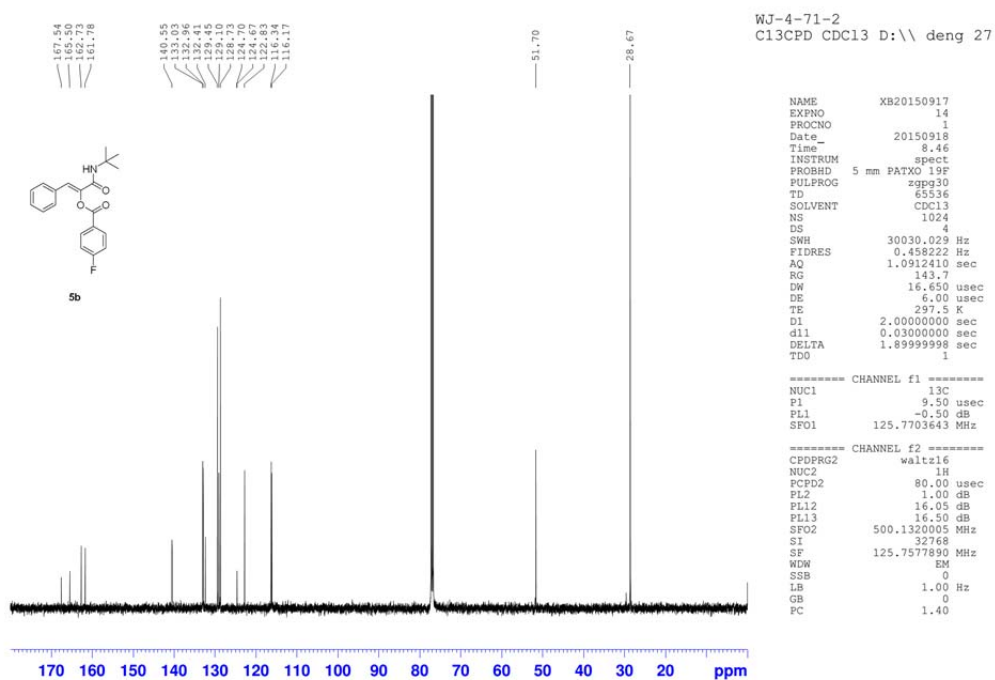
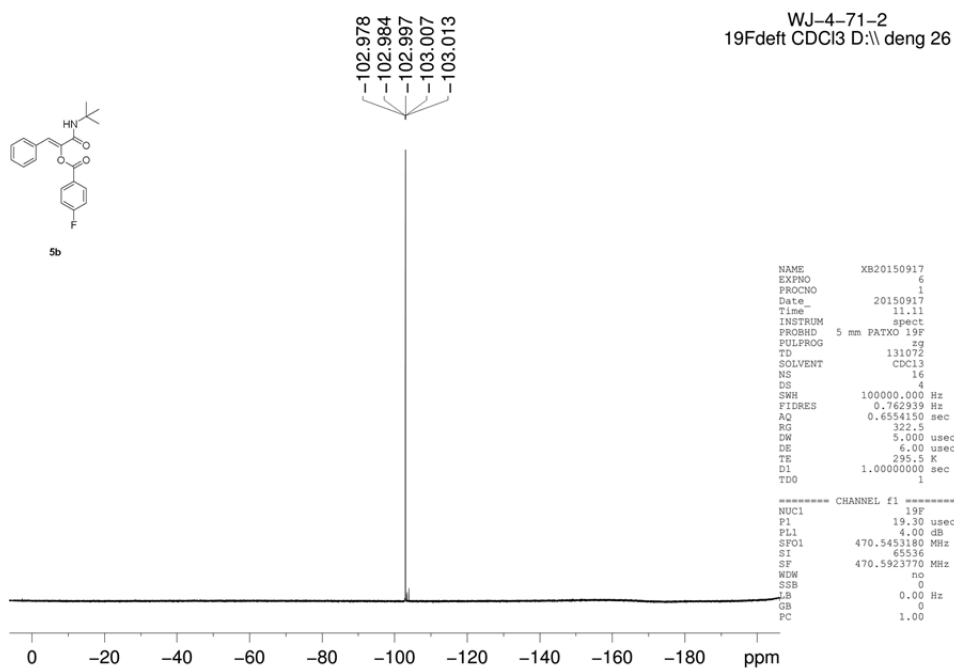
WJ-76-2-P2  
PROTON Acetone

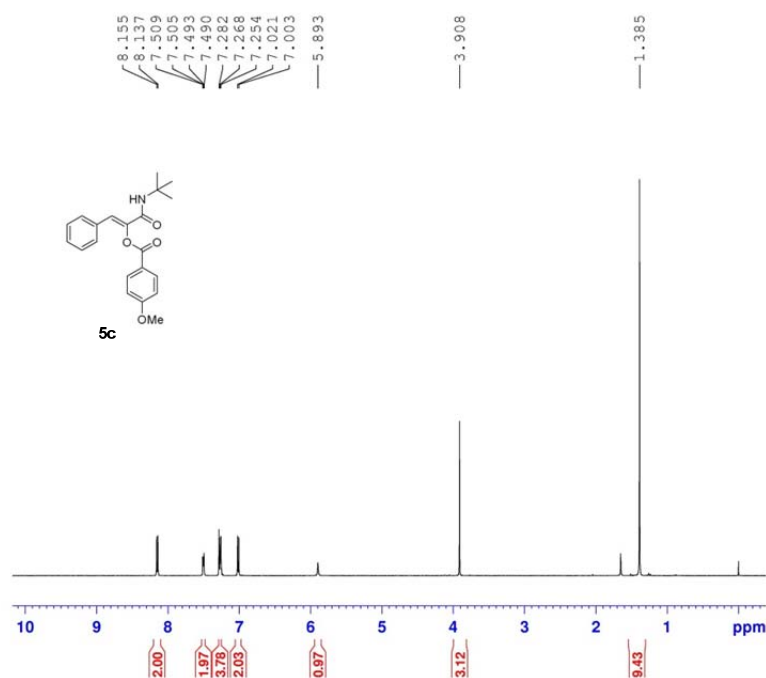
NAME	5a
EXPNO	1
PROCNO	1
Date_	20150201
Time	20.23
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	Acetone
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	128
DW	48.400 usec
DE	6.00 usec
TE	296.1 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300095 MHz
WDW	EM
SSB	0
LB	0.70 Hz
GB	0
PC	1.00





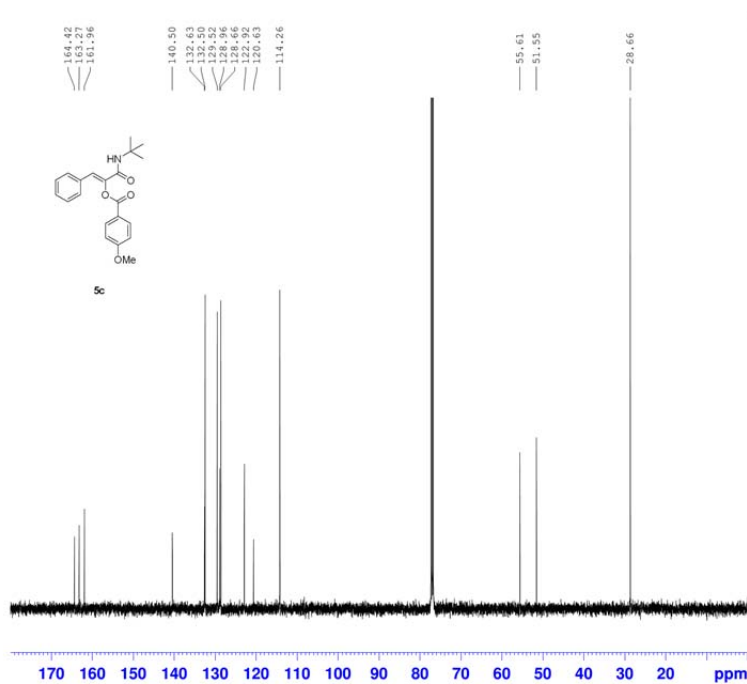


WJ-4-15  
PROTON CDC13

```

NAME      5c
EXPNO     1
PROCNO    1
Date_     20150827
Time      1.33
INSTRUM   spect
PROBHD    5 mm PATXO 19F
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         203.2
DW         48.400 usec
DE         6.00 usec
TE         295.6 K
D1         1.00000000 sec
D11
DELTA
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.14 usec
PL1        1.00 dB
SFO1       500.1330885 MHz
SI         32768
SF         500.1300126 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
  
```



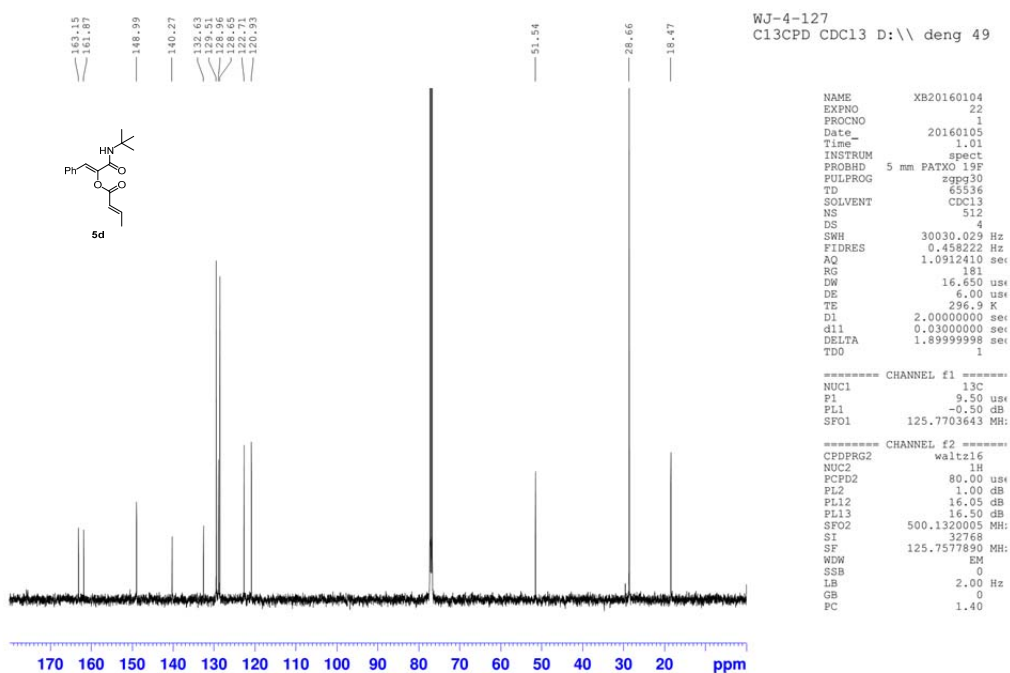
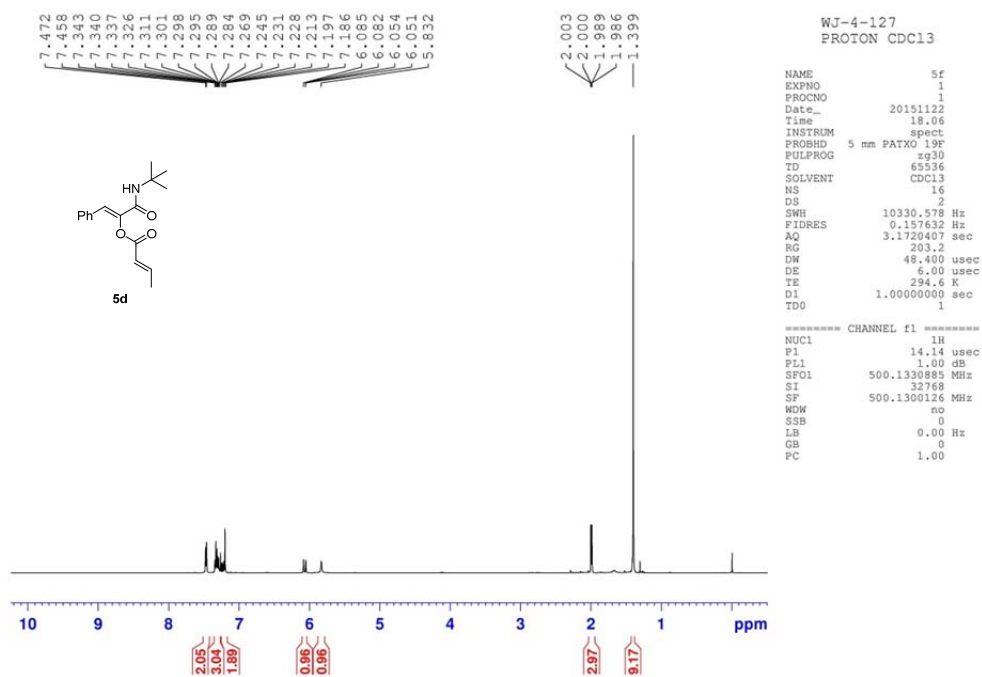
WJ-4-15  
C13CPD CDC13 D:\ deng 60

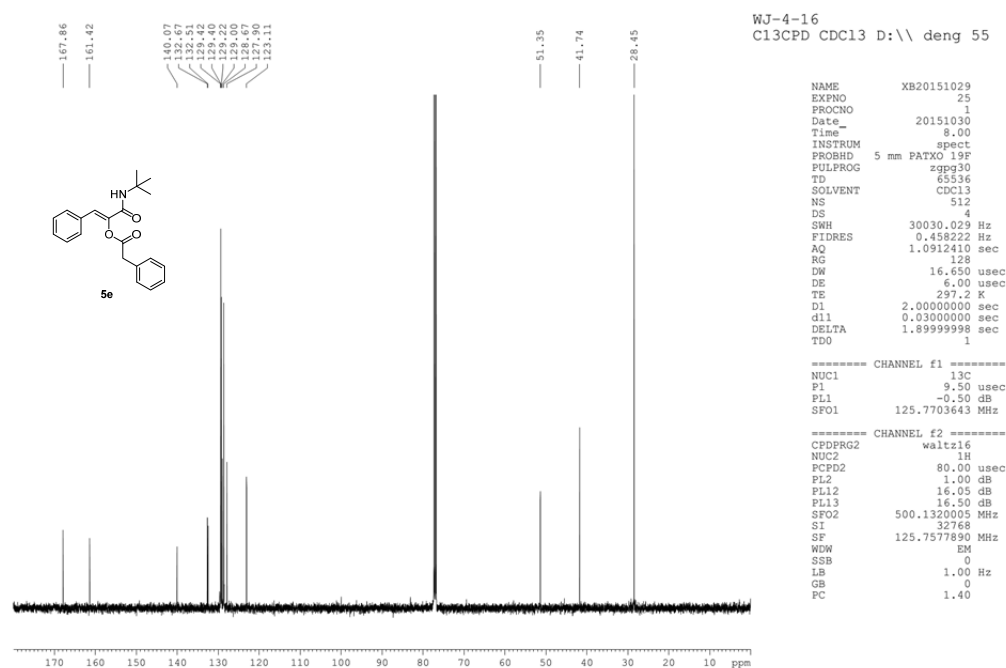
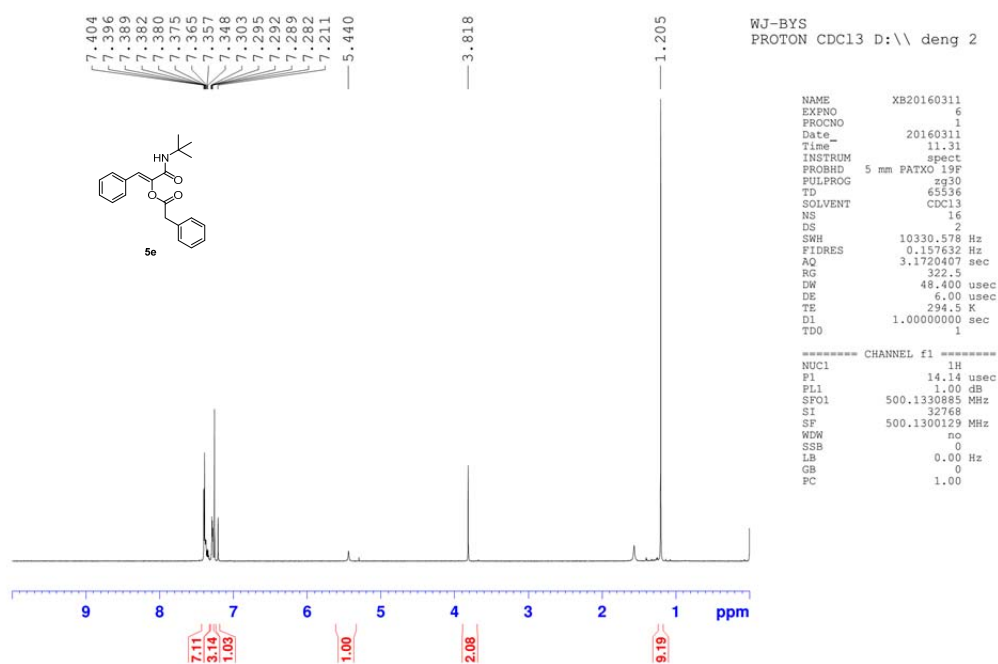
```

NAME      XB20150827
EXPNO     18
PROCNO    1
Date_     20150828
Time      1.21
INSTRUM   spect
PROBHD    5 mm PATXO 19F
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
NS         512
DS         4
SWH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912410 sec
RG         128
DW         16.650 usec
DE         6.00 usec
TE         296.9 K
D1         2.00000000 sec
d11
DELTA      1.89999999 sec
TD0        1

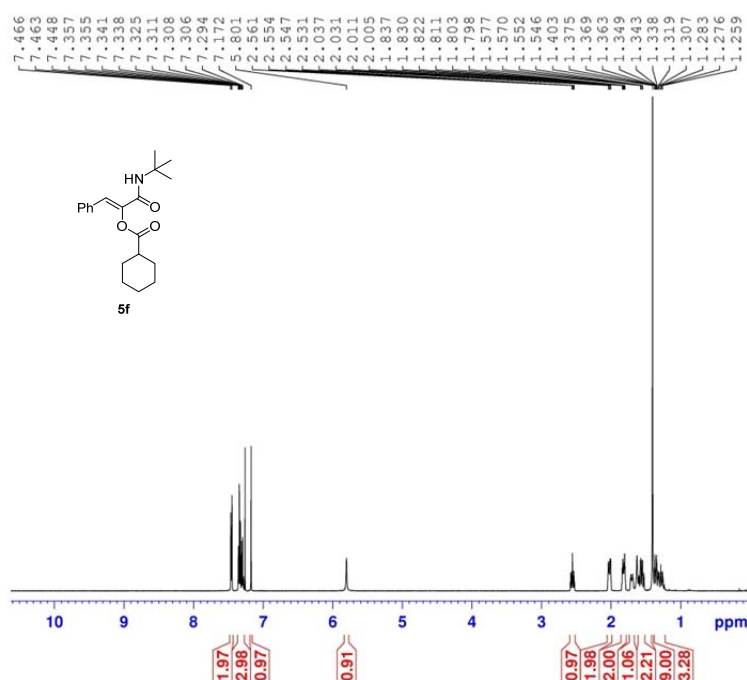
===== CHANNEL f1 =====
NUC1       13C
P1         9.50 usec
PL1        -0.50 dB
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        1.00 dB
PL12       16.05 dB
PL13       16.50 dB
SFO2       500.1320005 MHz
SI         32768
SF         125.7577890 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```





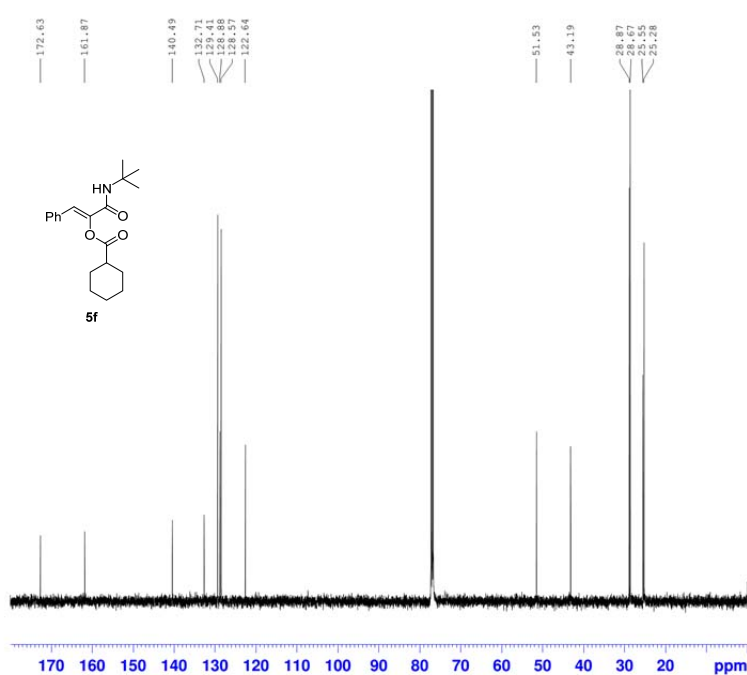




5f  
PROTON CDC13

NAME XB20180822  
EXPNO 16  
PROCNO 1  
Date\_ 20180822  
Time 14.14  
INSTRUM spect  
PROBHD 5 mm PATXO 19F  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 2  
SWH 10330.578 Hz  
FIDRES 0.157632 Hz  
AQ 3.1720407 sec  
RG 287.4  
DW 48.400 usec  
DE 6.50 usec  
TE 296.0 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.24 usec  
PL1 1.00 dB  
SFO1 500.1330885 MHz  
SI 32768  
SF 500.1300139 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

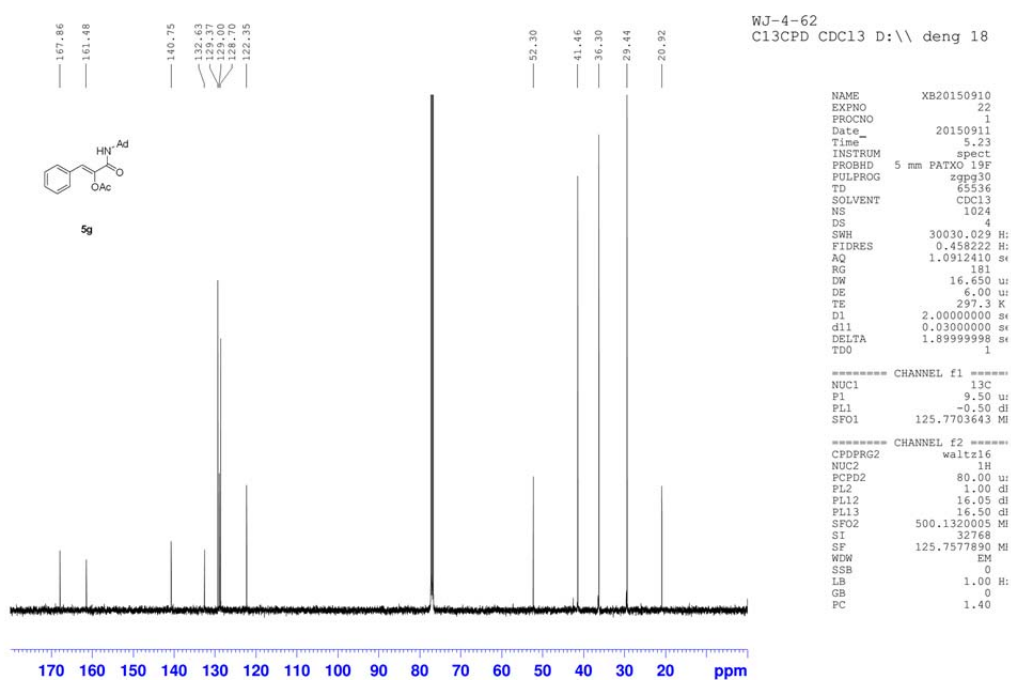
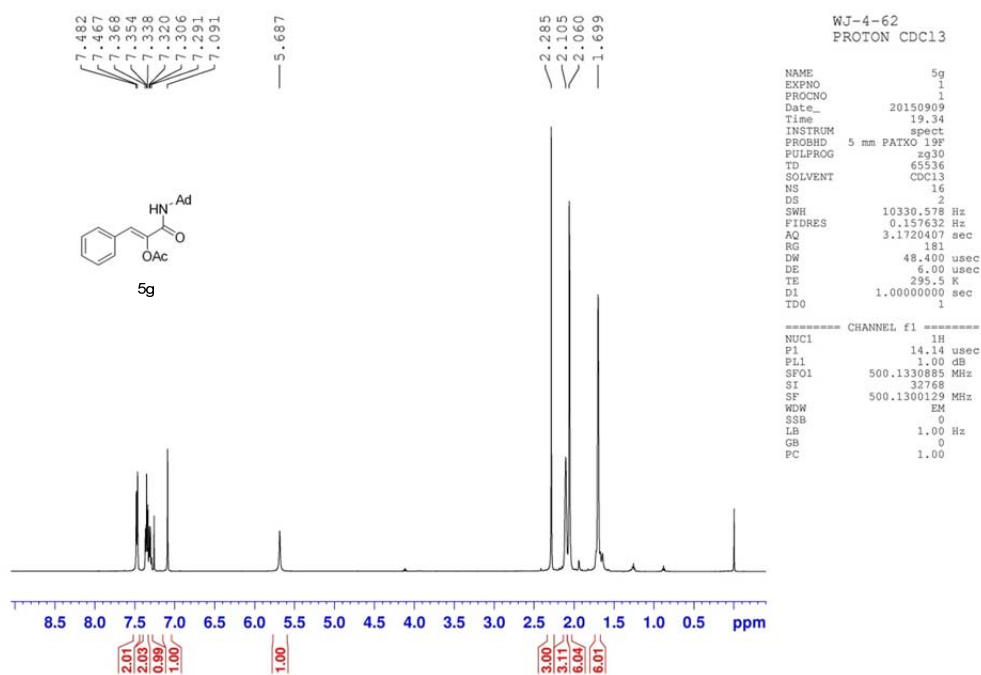


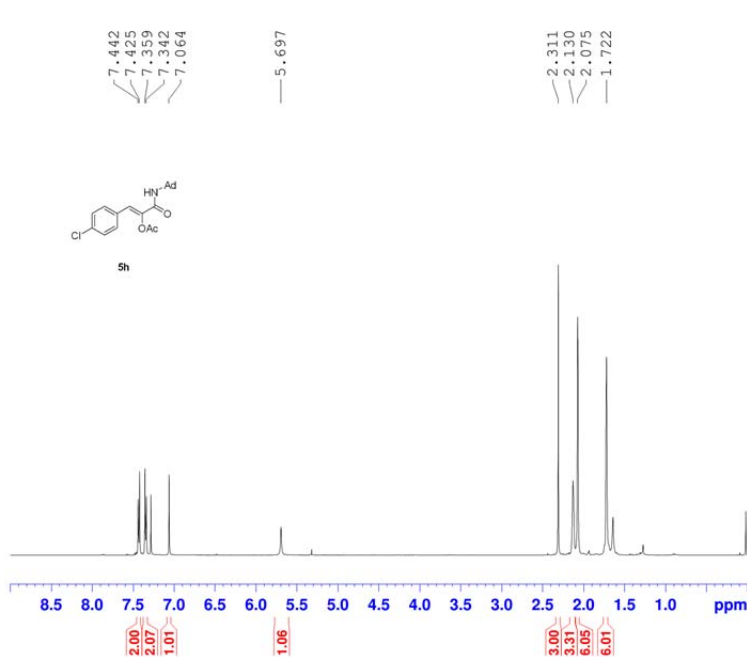
WJ-4-71-1  
C13CPD CDC13 D:\\ deng 2

NAME XB20151013  
EXPNO 19  
PROCNO 1  
Date\_ 20151013  
Time 22.02  
INSTRUM spect  
PROBHD 5 mm PATXO 19F  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 1024  
DS 4  
SWH 30030.029 Hz  
FIDRES 0.458222 Hz  
AQ 1.0912410 sec  
RG 228.1  
DW 16.650 usec  
DE 6.00 usec  
TE 297.3 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.50 dB  
SFO1 125.7703643 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 1.00 dB  
PL12 16.05 dB  
PL13 16.50 dB  
SFO2 500.1320005 MHz  
SI 32768  
SF 125.7577890 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40





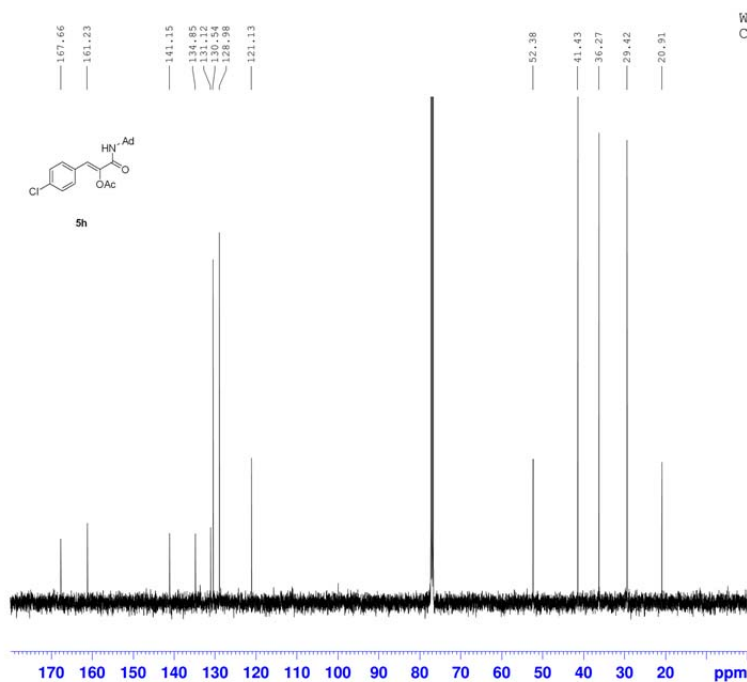
WJ-4-95  
PROTON CDC13 D:\ deng 59

```

NAME      XB20151028
EXPNO     12
PROCNO    1
Date_     20151028
Time      15.41
INSTRUM   spect
PROBHD    5 mm PATXO 19F
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         32
DS         16
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         228.1
DW         48.400 usec
DE         6.00 usec
TE         294.8 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.14 usec
PL1        1.00 dB
SFO1       500.1330885 MHz
SI         32768
SF         500.1299999 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00

```



WJ-4-95  
C13CPD CDC13 D:\ deng 49

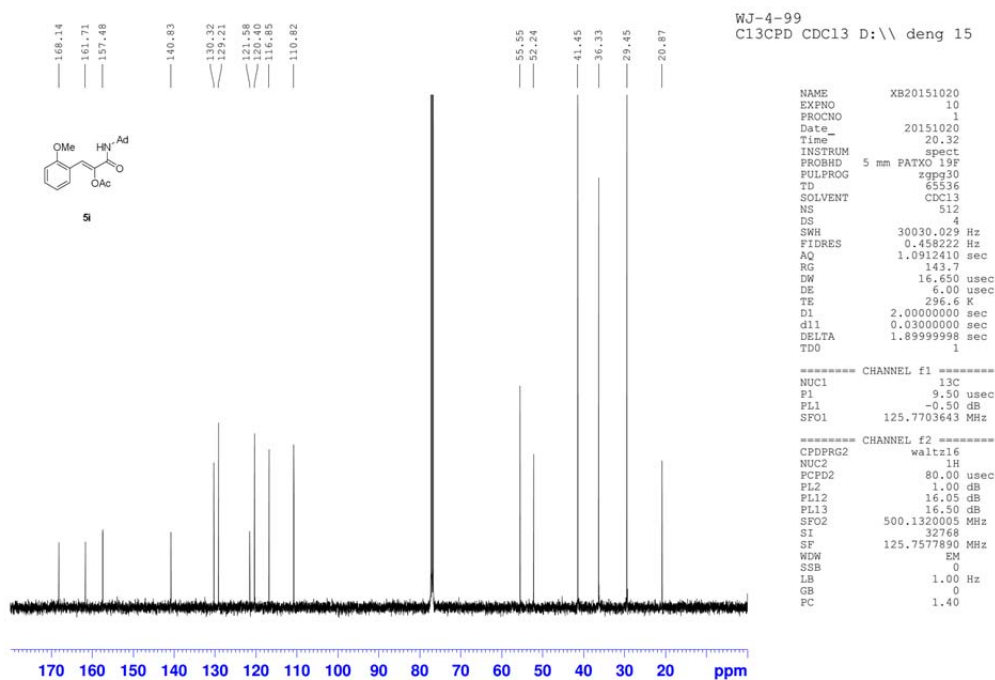
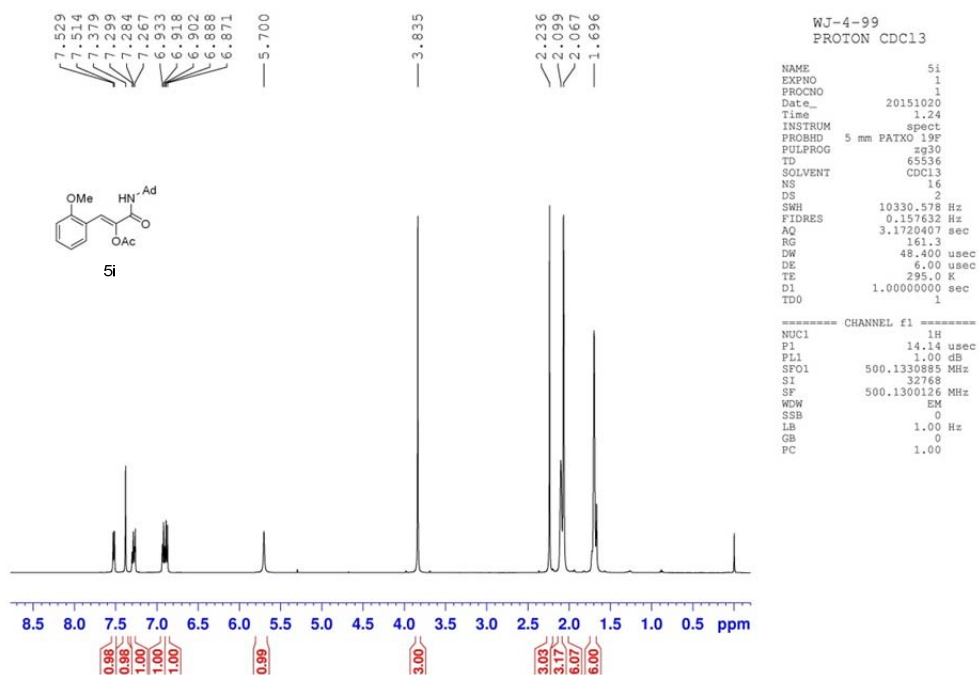
```

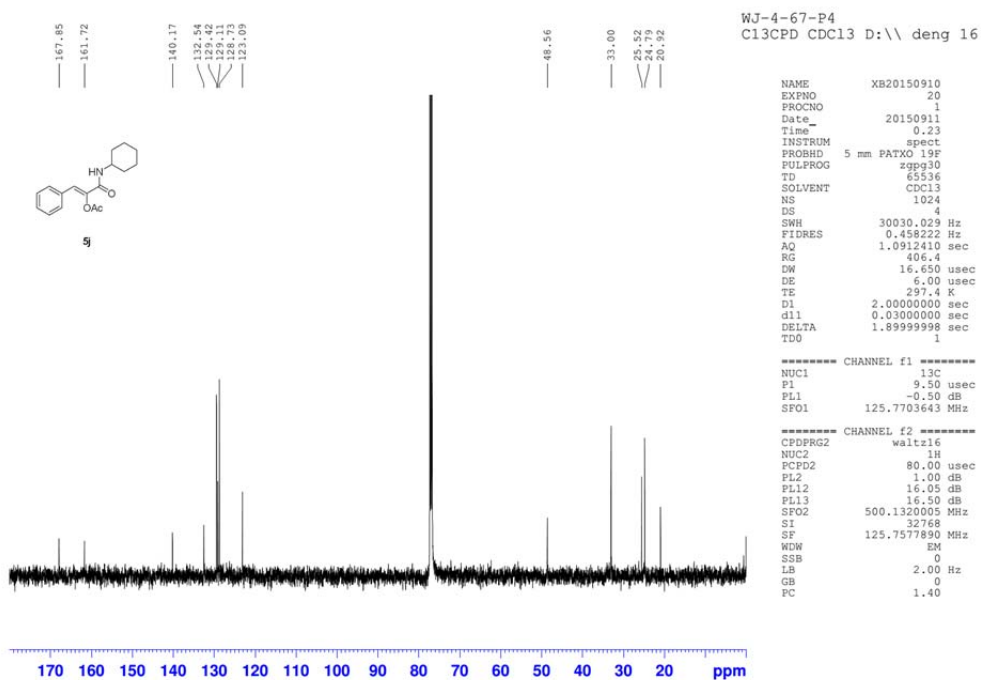
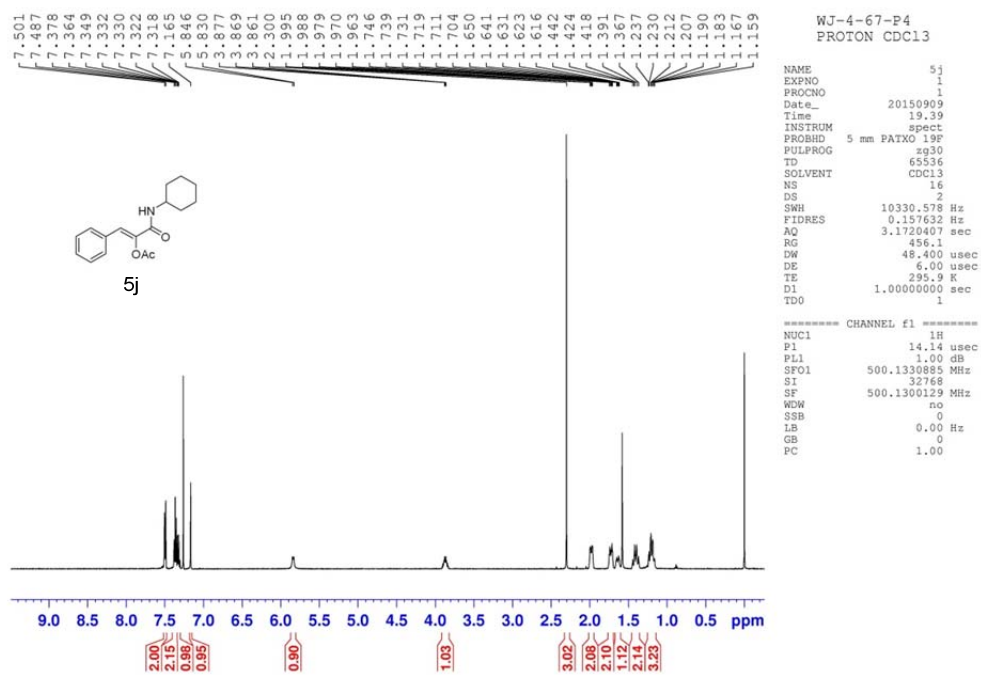
NAME      XB20151029
EXPNO     21
PROCNO    1
Date_     20151029
Time      17.51
INSTRUM   spect
PROBHD    5 mm PATXO 19F
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
NS         512
DS         4
SWH        30030.029 MHz
FIDRES     0.458222 MHz
AQ         1.0912410 sec
RG         143.7
DW         16.650 usec
DE         6.00 usec
TE         296.8 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999999 sec
TD0        1

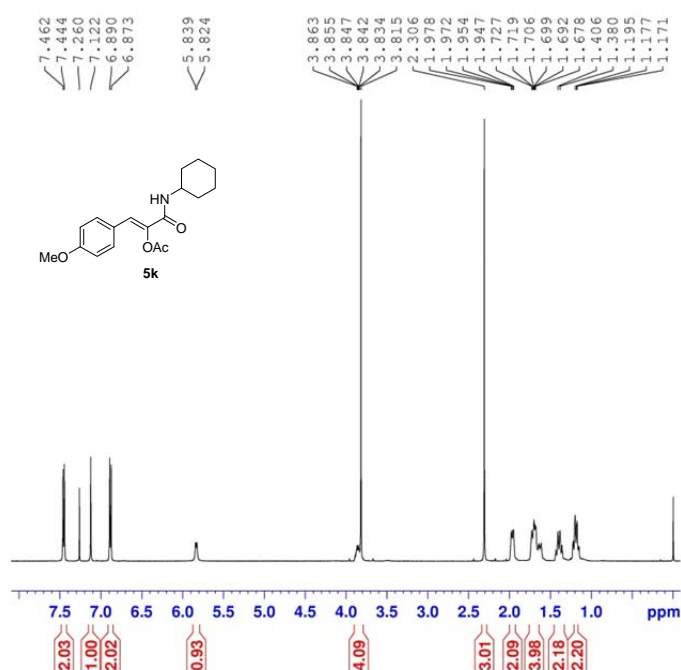
===== CHANNEL f1 =====
NUC1       13C
P1         9.50 usec
PL1        -0.50 dB
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2       1.00 dB
PL12      16.05 dB
PL13      16.50 dB
SFO2       500.1320005 MHz
SI         32768
SF         125.7577890 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```







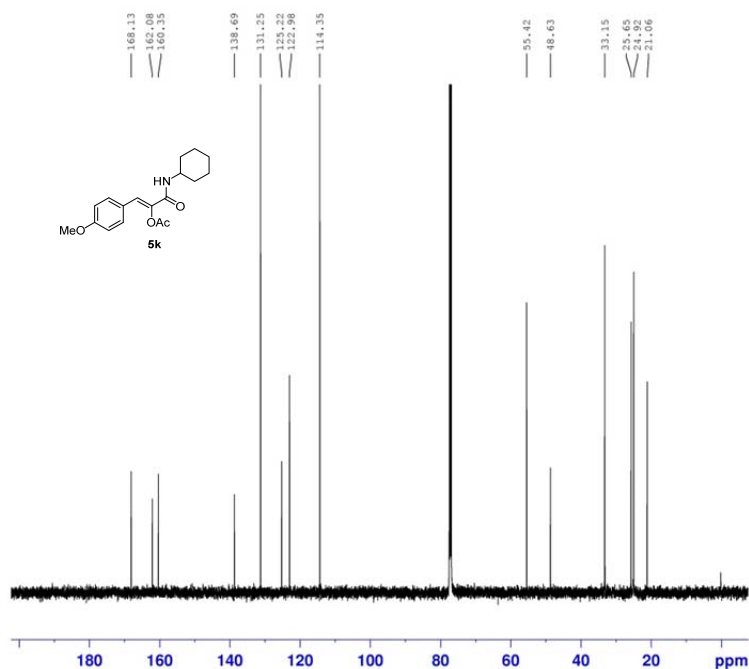
# 5K PROTON CDCl<sub>3</sub>

```

NAME      XB20180830
EXPNO     9
PROCNO    1
Date_     20180830
Time      16.37
INSTRUM   spect
PROBHD    5 mm PATXO 19F
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         228.1
DW         48.400 usec
DE         6.50 usec
TE         296.4 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.24 usec
PL1       1.00 dB
SFO1      500.1330885 MHz
SI        32768
SF        500.1300137 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
  
```



# 5K C13CPD CDCl<sub>3</sub>

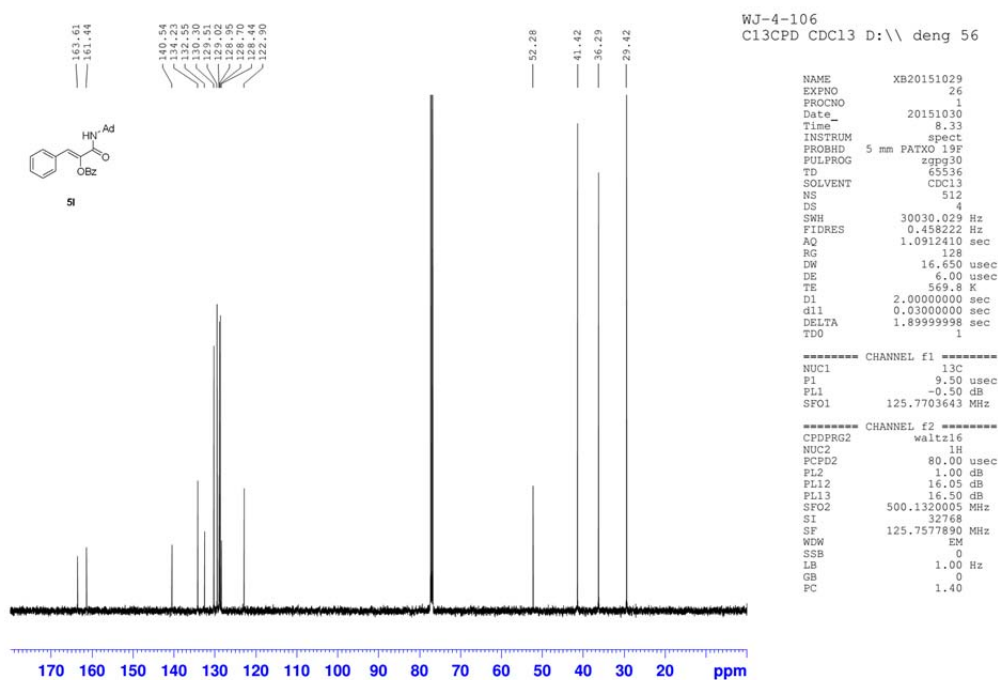
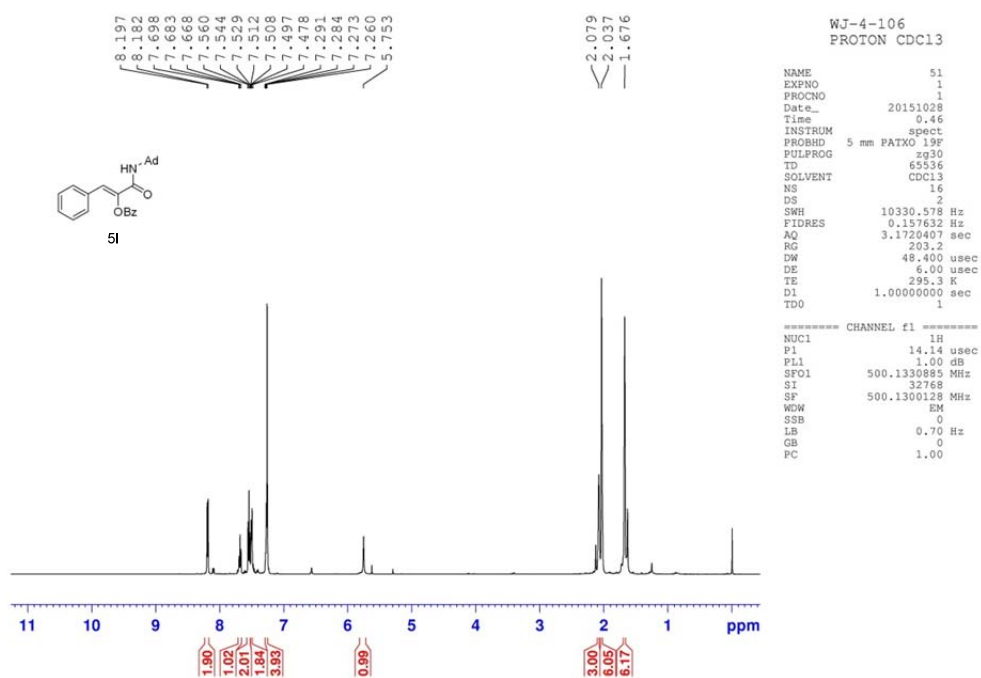
```

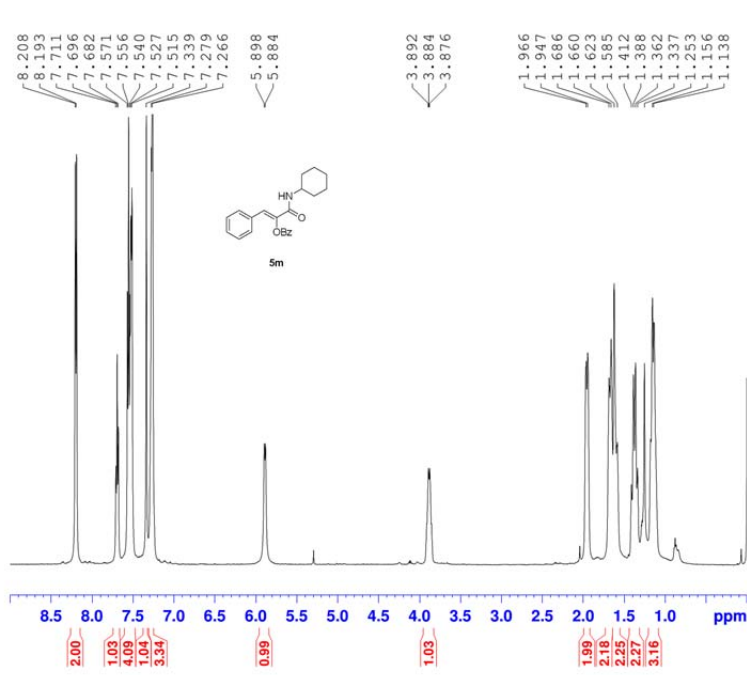
NAME      XB20180830
EXPNO     17
PROCNO    1
Date_     20180830
Time      23.09
INSTRUM   spect
PROBHD    5 mm PATXO 19F
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1837
DS         4
SWH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912410 sec
RG         14596.5
DW         16.650 usec
DE         6.50 usec
TE         297.9 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        9.50 usec
PL1       -0.50 dB
SFO1      125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       1.00 dB
PL12      15.99 dB
PL13      16.50 dB
SFO2      500.1320005 MHz
SI        32768
SF        125.7577736 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

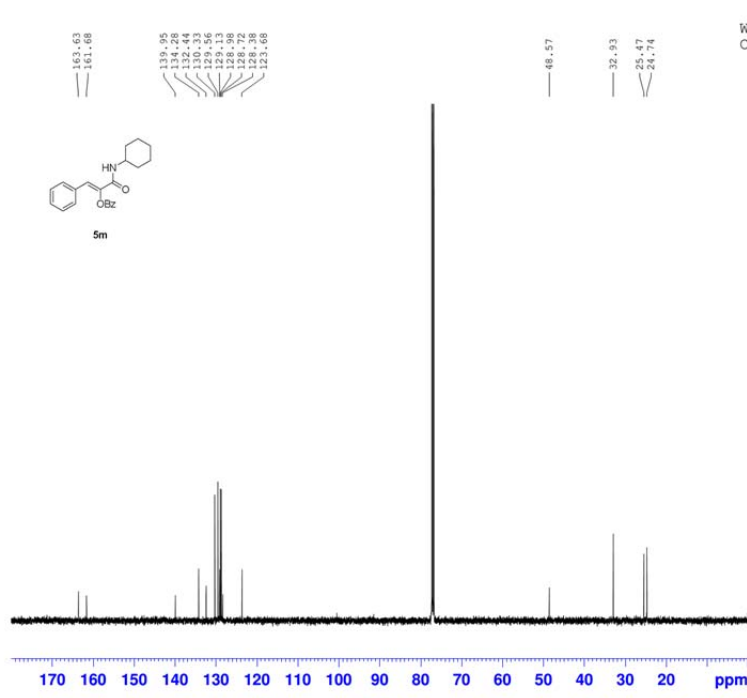




WJ-4-97  
PROTON CDCl3

```

NAME      XB20151021
EXPNO     11
PROCNO    1
Date_     20151021
Time      15.25
INSTRUM    spect
PROBHD     5 mm PATXO 19F
PULPROG    zg30
TD         65536
SOLVENT    CDCl3
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         256
DW         48.400 usec
DE         6.00 usec
TE         295.3 K
D1         1.00000000 sec
TDO        1
===== CHANNEL f1 =====
NUC1       1H
P1         14.14 usec
PL1        1.00 dB
SFO1       500.130885 MHz
SI         32768
SF         500.1300126 MHz
WDW        EM
SSB        0
LB         1.50 Hz
GB         0
PC         1.00
  
```

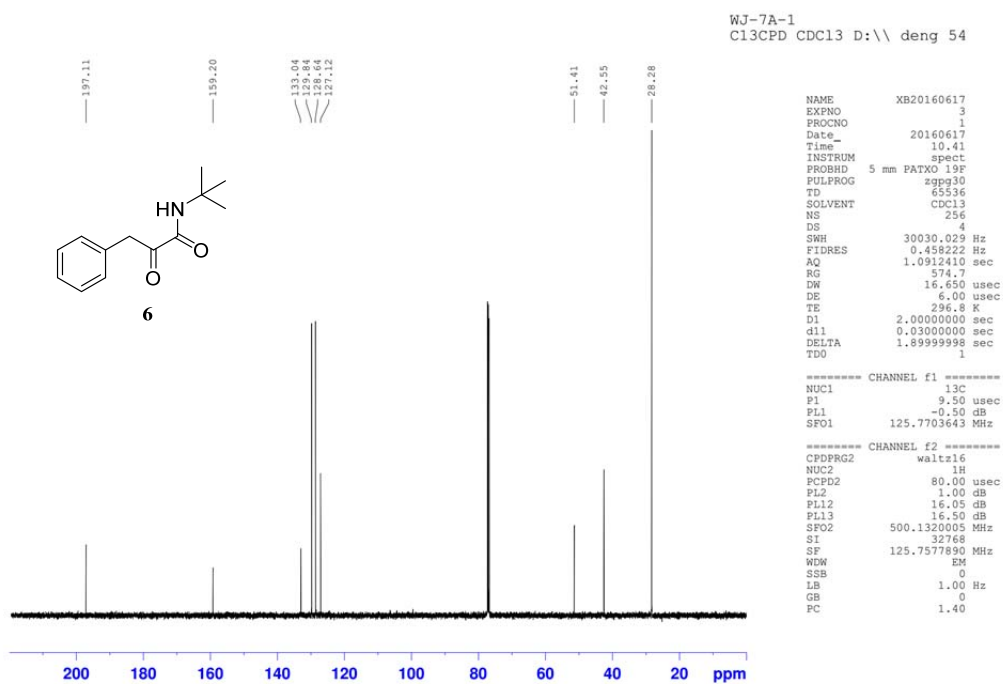
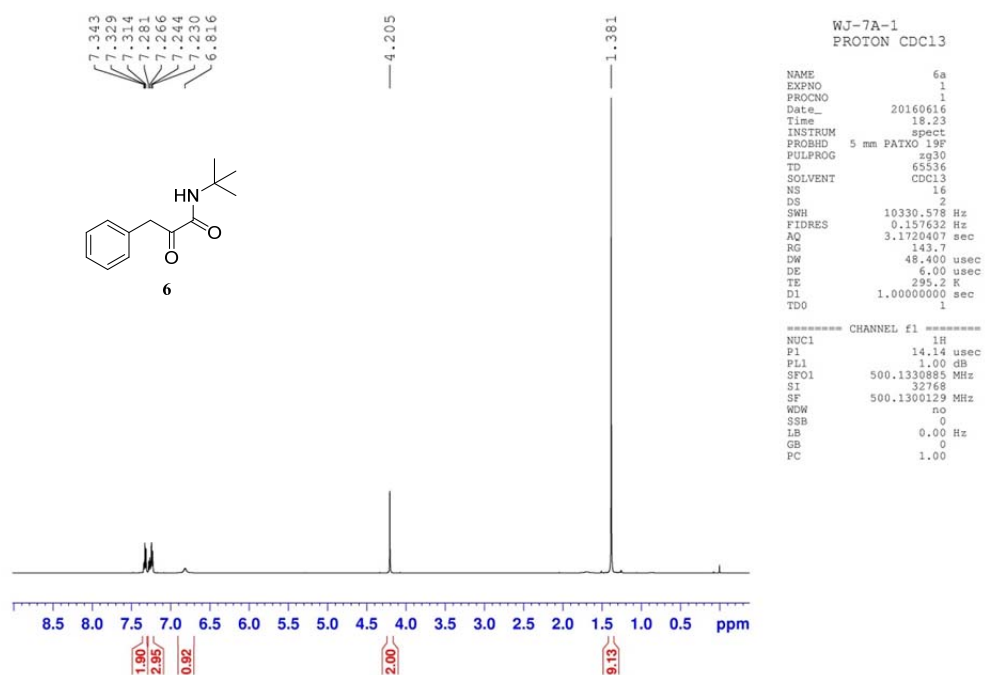


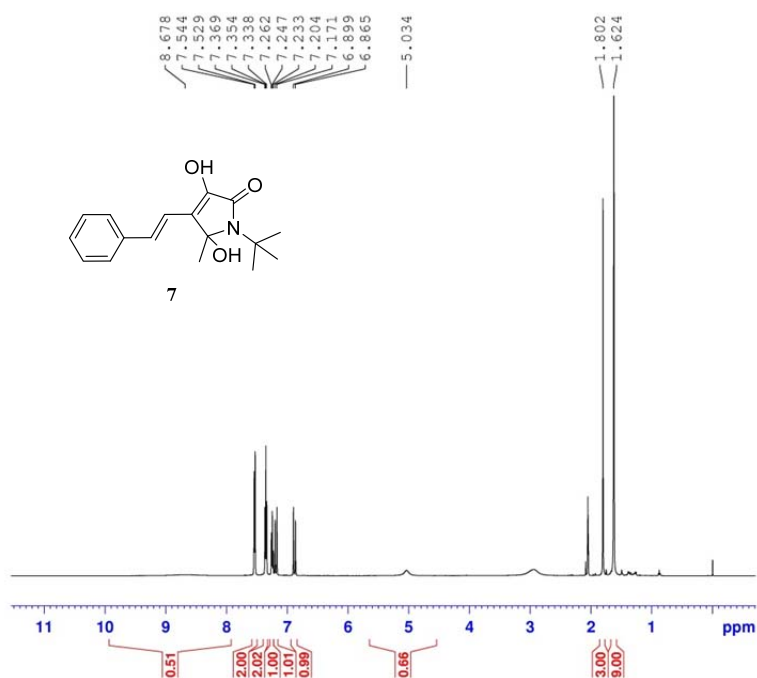
WJ-4-97  
C13CPD CDCl3 D:\ deng 16

```

NAME      XB20151022
EXPNO     14
PROCNO    1
Date_     20151023
Time      5.36
INSTRUM    spect
PROBHD     5 mm PATXO 19F
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS         512
DS         4
SWH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912410 sec
RG         128
DW         16.650 usec
DE         6.00 usec
TE         296.7 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
TDO        1
===== CHANNEL f1 =====
NUC1       13C
P1         9.50 usec
PL1        -0.50 dB
SFO1       125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        1.00 dB
PL12       16.05 dB
PL13       16.50 dB
SFO2       500.1320005 MHz
SI         32768
SF         125.7577890 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```







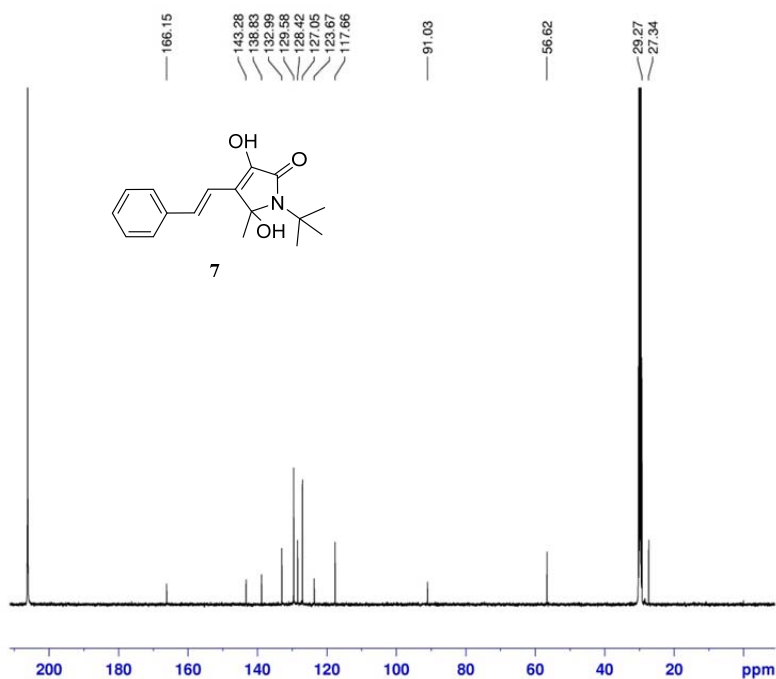
ZMF-7  
PROTON Acetone

```

NAME      XB20181119
EXPNO     22
PROCNO    1
Date_     20181119
Time      0.29
INSTRUM   spect
PROBHD    5 mm PATXO 19P
PULPROG   zg30
TD         65536
SOLVENT   Acetone
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         143.7
DW         48.400 usec
DE         6.50 usec
TE         295.9 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.63 usec
PL1       1.00 dB
SFO1      500.130889 MHz
SI        32768
SF        500.1300100 MHz
WDW        EM
SSB        0
LB         0.70 Hz
GB         0
PC         1.00
  
```



ZMF-2-61  
C13CPD Acetone

```

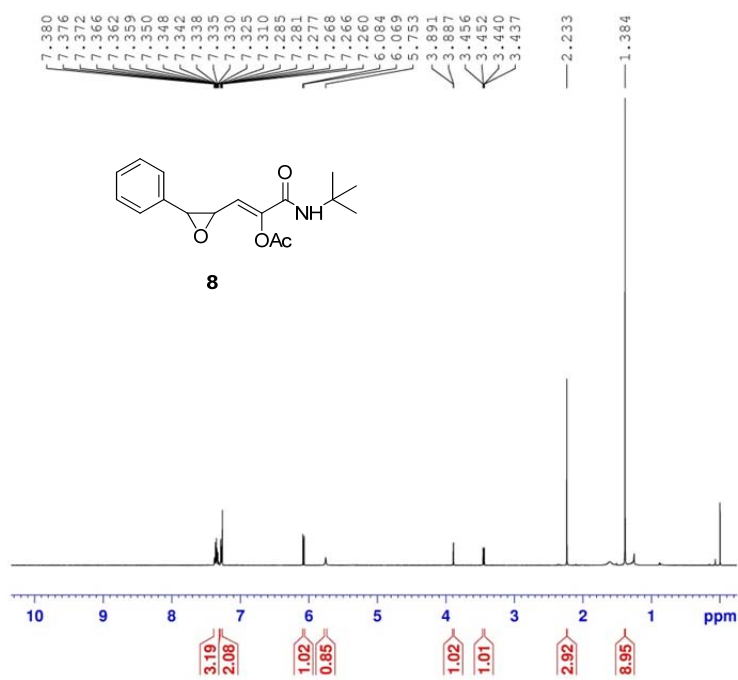
NAME      XB20181025
EXPNO     28
PROCNO    1
Date_     20181026
Time      1.10
INSTRUM   spect
PROBHD    5 mm PATXO 19P
PULPROG   zgpg30
TD         65536
SOLVENT   Acetone
NS         2200
DS         4
SWH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912410 sec
RG         18390.4
DW         16.650 usec
DE         6.50 usec
TE         298.4 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999998 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        9.50 usec
PL1       -0.50 dB
SFO1      125.7703643 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       1.00 dB
PL12      15.99 dB
PL13      16.50 dB
SFO2      500.1320005 MHz
SI        32768
SF        125.7576768 MHz
WDW        EM
SSB        0
LB         2.00 Hz
GB         0
PC         1.40
  
```



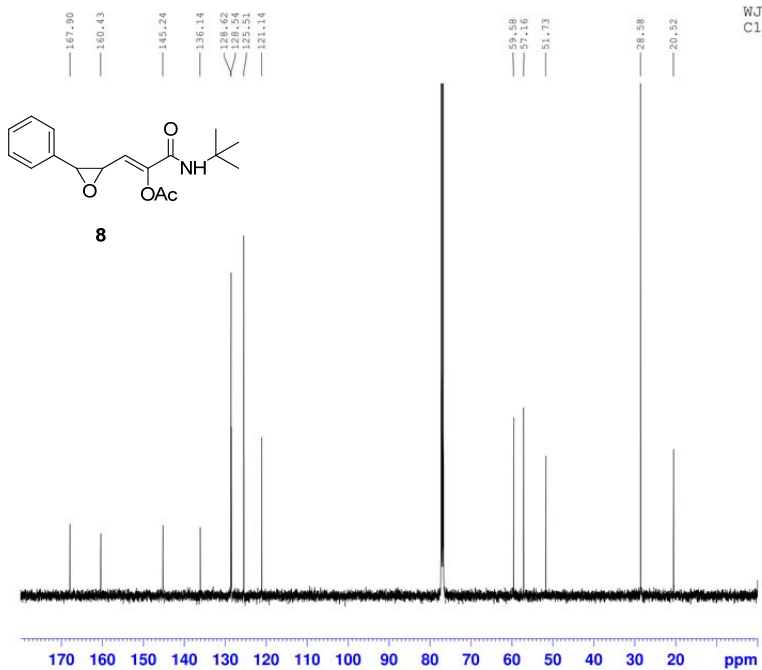
8  
PROTON CDC13

```

NAME      XB20180822
EXPNO     29
PROCNO    1
Date_     20180822
Time      16.01
INSTRUM   spect
PROBHD    5 mm PATXO 19F
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1720407 sec
RG         406.4
DW         48.400 usec
DE         6.50 usec
TE         295.7 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.24 usec
PL1        1.00 dB
SFO1       500.130085 MHz
SI         32768
SF         500.1300137 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

```



WJ-5-78  
C13CPD CDC13 D:\\ deng 13

```

NAME      XB20160620
EXPNO     25
PROCNO    1
Date_     20160621
Time      1.47
INSTRUM   spect
PROBHD    5 mm PATXO 19F
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         4400
DS         4
SWH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912410 sec
RG         143.7
DW         16.650 usec
DE         6.00 usec
TE         298.7 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999999 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.50 usec
PL1        0.50 dB
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        1.00 dB
PL12       15.99 dB
PL13       16.50 dB
SFO2       500.1320005 MHz
SI         32768
SF         125.7577890 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```