

## Supporting information

for

### Intramolecular radical non-reductive alkylation of ketones via transient enamines

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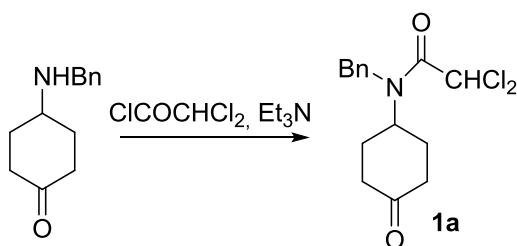
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## EXPERIMENTAL SECTION

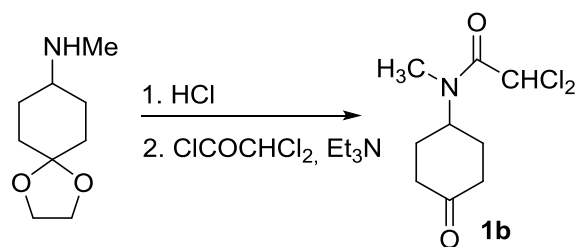
**1. General information.**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded in  $\text{CDCl}_3$  solution. Chemical shifts are reported as  $\delta$  values (ppm) relative to internal  $\text{Me}_4\text{Si}$  and in benzene- $\text{D}_6$  (7.16 ppm),  $^{13}\text{C}$  NMR spectra are referenced to the deuterated solvent signal ( $\text{CDCl}_3$ : 77.00 ppm) and benzene- $\text{D}_6$  (128.4 ppm). All NMR data assignments are supported by COSY and HSQC experiments. Infrared spectra were recorded on a Nicolet 320 FT-IR spectrophotometer. TLC was performed on  $\text{SiO}_2$  (silica gel 60 F<sub>254</sub>, Merck) or on  $\text{Al}_2\text{O}_3$  (aluminium oxide 60 F254 neutral, Merck). The spots were located by UV light or a 1%  $\text{KMnO}_4$  aqueous solution. Chromatography refers to flash chromatography and was carried out on  $\text{SiO}_2$  (Silica Flash P60, Wet & Dry, 200-500 mesh) and when indicated on  $\text{Al}_2\text{O}_3$  (aluminium oxide 90 standardized, Merck). Drying of the organic extracts during reaction work-up was performed over anhydrous  $\text{Na}_2\text{SO}_4$ .

### 1. Synthesis of dichloroacetamides **1a-1g**, **7** and **10**

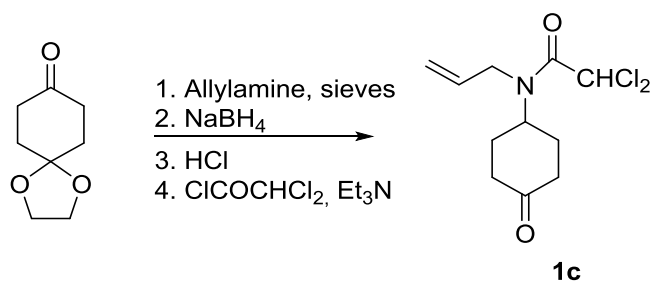


**1a:** To a solution of 4-(benzylamino)cyclohexan-1-one<sup>1</sup> (11 g, 54.11 mmol) and triethylamine (11.31 mL, 81.16 mmol) in  $\text{CH}_2\text{Cl}_2$  (110 mL) was added dichloroacetyl chloride (6.24 mL, 64.93 mmol) dropwise at 0 °C. The mixture was stirred at rt for 1 h then poured into water and extracted with  $\text{CH}_2\text{Cl}_2$ . The organic extracts were dried, concentrated and purified by chromatography ( $\text{CH}_2\text{Cl}_2/\text{EtOAc}$  1:1) to yield **1a** as a white solid (10.5 g, 62%): mp 86-88 °C; IR (NaCl) 3088, 3062, 3029, 2956, 2873, 1716, 1670  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 2 rotamers)  $\delta$  1.82-2.02 (m, 2H), 2.02-2.20 (m, 2H), 2.36-2.56 (m, 4H), 4.59 and 4.62 (2 s, 2H), 4.80 (m, 1H), 6.08 and 6.39 (2 s, 1H), 7.14-7.46 (m, 5H, ArH);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz, 2 rotamers)  $\delta$  28.6 and 30.0 ( $\text{CH}_2$ ), 39.5 and 39.6 ( $\text{CH}_2$ ), 45.8 and 47.4 ( $\text{CH}_2$ ), 54.0 and 55.9 (CH), 64.9 and 66.5 (CH), 125.5 and 126.4 (CH), 127.2 and 128.2 (CH), 128.6 and 129.3 (CH), 136.1 and 137.3 (C), 163.8 and 164.8 (CO), 207.9 and 208.7 (CO); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{15}\text{H}_{18}\text{Cl}_2\text{NO}_2$  314.0709; found 314.0710.

<sup>1</sup> Diaba, F.; Pujol-Grau, C.; Martínez-Laporta, A.; Fernández, I.; Bonjoch, J. *Org. Lett.* **2015**, *17*, 568-571.



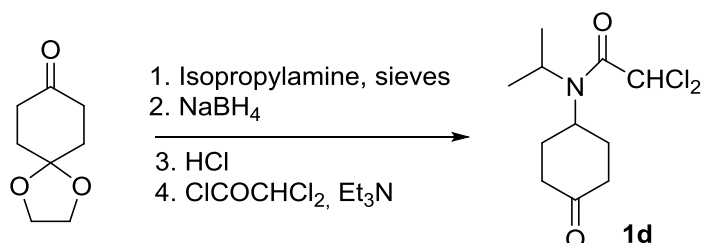
**1b:** A mixture of 4-(methylamino)cyclohexanone ethylene acetal<sup>2</sup> (3.82 g, 22.31 mmol) and 10% HCl solution (100 mL) was stirred at rt overnight. The mixture was basified with 10% NaOH solution and extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic extracts were dried, concentrated and the residue (1.3 g, 10.22 mmol) was treated with Et<sub>3</sub>N (2.14 mL, 15.33 mmol) and dichloroacetyl chloride (1.18 mL, 12.26 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (15 mL) at 0 °C then at rt for 1 h. The mixture was poured into water and extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic extracts were dried, concentrated and after chromatography (Al<sub>2</sub>O<sub>3</sub>, CH<sub>2</sub>Cl<sub>2</sub>) **1b** was isolated as a white solid (1.05 g, 43% over the 2 steps): mp 126-127 °C; IR (NaCl) 3004, 2953, 2873, 1716, 1664 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 2 rotamers) δ 1.84-2.24 (m, 4H), 2.42-2.62 (m, 4H), 2.88 and 3.07 (2 s, 3H), 4.67 and 4.85 (2 m, 1H), 6.27 and 6.29 (2 s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 2 rotamers) δ 28.2 and 29.0 (CH<sub>2</sub>), 28.6 and 30.0 (CH<sub>3</sub>), 39.5 (CH<sub>2</sub>), 52.4 and 54.9 (CH), 65.6 and 66.4 (CH), 163.2 and 163.5 (CO), 208.0 and 208.7 (CO); HRMS (ESI-TOF) m/z: [M+H]<sup>+</sup> calcd for C<sub>9</sub>H<sub>14</sub>Cl<sub>2</sub>NO<sub>2</sub> 238.0396; found 238.0395.



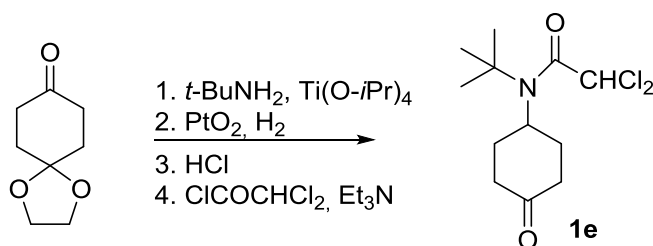
**1c:** A mixture of allylamine (1.6 mL, 20.81 mmol), 1,4-cyclohexanedione monoethylene acetal (2.5 g, 16.0 mmol) and 4 Å molecular sieves (2 g) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was stirred at rt for 4 h then filtered on a short celite pad and concentrated. The residue was treated with NaBH<sub>4</sub> (1.21 g, 19.2 mmol) in MeOH (20 ml) at 0 °C then at rt for 1 h. The mixture was concentrated, quenched with water extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic extracts were dried, concentrated and the resulting viscous oil was treated with 10% HCl solution (30 mL) overnight. The mixture was basified with 10% NaOH solution and extracted with

<sup>2</sup> W. J. Greenlee, Y. Huang, J. M. Kelly, S. W. McCombie, A. Stamford and Y. Wu, in US 2005/0038100 A1, Schering-Plough Corp., USA, **2005**.

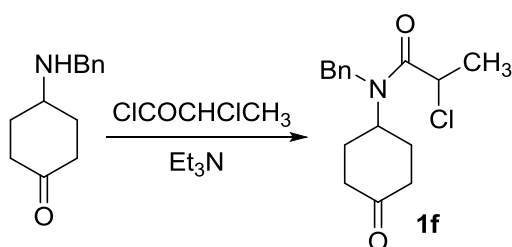
CH<sub>2</sub>Cl<sub>2</sub>. The organic extracts were dried, concentrated and the residue was treated with Et<sub>3</sub>N (4.46 mL, 32.01 mmol) and dichloroacetyl chloride (2.67 mL, 24.01 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (25 mL) at 0 °C then at rt for 1 h. The mixture was poured into water and extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic extracts were dried, concentrated and purified by chromatography (CH<sub>2</sub>Cl<sub>2</sub>) to yield **1c** as a white solid (1.3 g, 43% over the 4 steps): mp 84-86 °C; IR (NaCl) 3007, 2985, 2959, 2923, 1717, 1665 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 2 rotamers) δ 1.84-2.26 (m, 4H), 2.40-2.60 (m, 4H), 3.88-4.04 (m, 2H), 4.66-4.86 (M, 1H), 5.12-5.38 (m, 2H), 5.76-5.96 (m, 1H), 6.20 and 6.31 (2 s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 2 rotamers) δ 28.7 and 29.8 (2 CH<sub>2</sub>), 39.6 (2 CH<sub>2</sub>), 45.1 and 45.9 (CH<sub>2</sub>), 53.3 and 55.6 (CH), 64.7 and 66.5 (CH), 116.9 and 117.5 (CH<sub>2</sub>), 133.0 and 133.7 (CH), 162.9 and 164.5 (CO), 208.1 and 208.8 (CO); HRMS (ESI-TOF) m/z: [M+H]<sup>+</sup> calcd for C<sub>11</sub>H<sub>16</sub>Cl<sub>2</sub>NO<sub>2</sub> 264.0553; found 264.0556.



**1d**: Operating as above from isopropylamine (2.15 mL, 24.97 mmol) and 1,4-cyclohexanedione monoethylene acetal (3 g, 18.6 mmol), **1d** was obtained as a white solid (1.72 g, 34% for the 4 steps): mp 133-135 °C; IR (NaCl) 3044, 3004, 2967, 2891, 1715, 1655 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 2 rotamers) δ 1.34 and 1.41 (2 d, *J* = 6.8 Hz, 6H), 1.85 and 2.23 (2 m, 2H), 2.06 and 2.83 (2 qd, *J* = 12.4, 5.2 Hz, 2H), 2.30-2.241 (m, 2H), 2.47-2.58 (m, 2H), 3.36-3.60 (m, 1H), 4.45-4.60 (m, 1H), 6.17 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 2 rotamers) δ 19.6 and 20.3 (CH<sub>3</sub>), 27.0 and 29.1 (CH<sub>2</sub>), 39.5 (CH<sub>2</sub>), 48.5 and 49.6 (CH), 52.6 and 55.9 (CH), 67.0 and 67.8 (CH), 162.0 and 162.4 (CO), 208.2 and 209.7 (CO); HRMS (ESI-TOF) m/z: [M+H]<sup>+</sup> calcd for C<sub>11</sub>H<sub>18</sub>Cl<sub>2</sub>NO<sub>2</sub> 266.0709; found 266.0707.



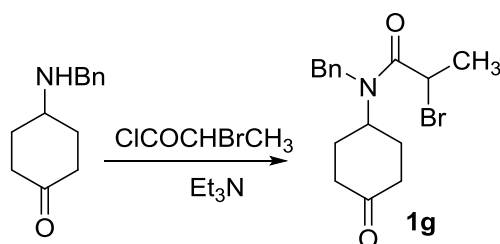
**1e:** To a mixture of *t*-butylamine (4.04 mL, 38.42 mmol) and 1,4-cyclohexanedione monoethylene acetal (3 g, 19.2 mmol) was added titanium(IV) isopropoxide<sup>3</sup> (7.10 mL, 24.01 mmol) and the mixture was stirred at rt under argon atmosphere for 2.5 h. Ethanol (60 mL) and PtO<sub>2</sub> (0.3 g, 10%) were then added and the mixture was stirred under a hydrogen atmosphere (50 psi) and at rt overnight. The mixture was then filtered on a short celite pad and concentrated to yield a viscous oil which was treated with 10% HCl (38 ml) overnight. The mixture was basified with a saturated Na<sub>2</sub>CO<sub>3</sub> solution and extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic extracts were dried, concentrated and the residue was treated with Et<sub>3</sub>N (2.74 mL, 19.67 mmol) and dichloroacetyl chloride (1.51 mL, 15.75 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (40 mL) at 0 °C then at rt for 1 h. The mixture was poured into water and extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic extracts were dried, concentrated and purified by chromatography (CH<sub>2</sub>Cl<sub>2</sub>) to yield **1e** as a white solid (2.06 g, 38%). mp 183-184 °C; IR (NaCl) 3071, 2979, 2960, 2923, 2882, 1708, 1670 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 1 rotamer) δ 1.53 (s, 9H), 2.04-2.17 (m, 2H), 2.33-2.58 (m, 6H), 4.11 (br s, 1H), 6.28 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 1 rotamer) δ 29.6 (CH<sub>3</sub>), 30.5 (CH<sub>2</sub>), 40.3 (CH<sub>2</sub>), 55.1 (CH), 59.3 (C), 67.4 (CH), 166.5 (CO), 208.8 (CO); HRMS (ESI-TOF) m/z: [M+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>20</sub>Cl<sub>2</sub>NO<sub>2</sub> 280.0866; found 280.0862.



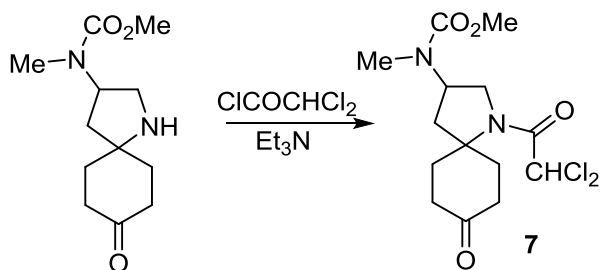
**1f:** To a solution of 4-(benzylamino)cyclohexanone (1g, 4.92 mmol), and triethylamine (1.03 mL, 7.38 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was added 2-chloropropionyl chloride (0.62 mL, 6.40 mmol) dropwise at 0 °C. The mixture was stirred at rt for 1 h then poured into water and extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic extracts were dried, concentrated and purified by chromatography (CH<sub>2</sub>Cl<sub>2</sub>) to yield **1f** as a yellowish oil (1.1 g, 76%): IR (NaCl) 3087, 3061, 3030, 2954, 2871, 1716, 1653 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 2 rotamers) δ 1.63 (d, *J* = 6.4 Hz, 3H), 1.68-2.18 (m, 4H), 2.30-2.58 (m, 4H), 4.32-4.52 (m, 2H), 4.68-4.98 (m, 2H), 7.14-7.41 (m, 5H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 2 rotamers) δ 20.8 and 21.1 (CH<sub>3</sub>), 28.8 and 30.2 (CH<sub>2</sub>), 289.0 and 30.9 (CH<sub>2</sub>), 39.6 and 39.7 (CH<sub>2</sub>), 45.1 and 46.7 (CH<sub>2</sub>), 50.0 and 50.1 (CH), 52.4 and 55.4 (CH), 125.3 and 126.4 (CH), 126.9 and 127.6 (CH), 128.4 and 129.0 (CH), 137.4 and 138.2 (C), 169.0 and 170.1 (CO), 208.1 and

<sup>3</sup> Palmer, J. T. et al. *J. Med. Chem.* **2005**, *48*, 7520-7534.

209.1 (CO); HRMS (ESI-TOF)  $m/z$ :  $[M+H]^+$  calcd for  $C_{16}H_{21}ClNO_2$  294.1255; found 294.1254.



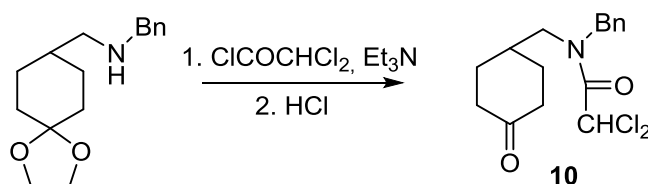
**1g**: To a solution of 4-(benzylamino)cyclohexanone (**1g**, 4.92 mmol) and triethylamine (1.03 mL, 7.38 mmol) in  $CH_2Cl_2$  (10 mL) was added 2-bromopropionyl chloride (0.67 mL, 6.40 mmol) dropwise at 0 °C. The mixture was stirred at rt for 1 h then poured into water and extracted with  $CH_2Cl_2$ . The organic extracts were dried, concentrated and purified by chromatography ( $CH_2Cl_2$ ) to yield **1f** as a yellowish oil (1.01 g, 60%): IR (NaCl) 3087, 3060, 3029, 2956, 2871, 1716, 1652  $cm^{-1}$ ;  $^1H$  NMR ( $CDCl_3$ , 400 MHz, 2 rotamers)  $\delta$  1.65-1.77 (m, 0.9 H), 1.78 and 1.94 (2 d,  $J = 6.8$  Hz, 3H,  $CH_3$ ), 1.86-2.04 (m, 2H), 2.07-2.18 (m, 1.1 H), 2.31-2.59 (m, 4H), 4.32 (q,  $J = 6.8$  Hz, 1H), 4.42 (d,  $J = 18.4$  Hz, 1H), 4.80 (d,  $J = 18.4$  Hz, 1H), 4.97 (tt,  $J = 12, 4$  Hz, 1H), 7.12-7.41 (m, 5H);  $^{13}C$  NMR ( $CDCl_3$ , 100 MHz, 2 rotamers)  $\delta$  21.4 and 21.9 ( $CH_3$ ), 28.6 and 30.0 ( $CH_2$ ), 29.1 and 31.1 ( $CH_2$ ), 38.8 and 39.3 (CH), 39.7 ( $CH_2$ ), 39.8 and 39.9 ( $CH_2$ ), 45.2 and 47.0 ( $CH_2$ ), 52.6 and 55.8 (CH), 125.2 and 126.5 (CH), 127.0 and 127.7 (CH), 128.6 and 129.1 (CH), 137.5 and 138.4 (C), 169.3 and 170.5 (CO), 208.2 and 209.3 (CO); HRMS (ESI-TOF)  $m/z$ :  $[M+H]^+$  calcd for  $C_{16}H_{21}BrNO_2$  338.0750; found 338.0745.



**7**: To a solution of 3-[(Methoxycarbonyl)(methyl)amino]-1-azaspiro[4.5]deca-8-one<sup>4</sup> (0.3 g, 1.25 mmol) and triethylamine (0.26 mL, 1.87 mmol) in  $CH_2Cl_2$  (10 mL) was added dichloroacetyl chloride (0.14 mL, 1.5 mmol) at 0 °C. The mixture was stirred at rt for 1 h then poured into water and extracted with  $CH_2Cl_2$ . The organic extracts were dried, concentrated and purified by chromatography ( $CH_2Cl_2$  to  $CH_2Cl_2/MeOH$  99.5:0.5) to yield **7** as a white solid (0.31 g, 70%): mp 76-77 °C; IR (NaCl): 3010, 2955, 2913, 2886, 1713,

<sup>4</sup> Diaba, F.; Martínez-Laporta, A.; Bonjoch, J. *J. Org. Chem.* **2014**, *79*, 9365-9372.

1694, 1680, 1674  $\text{cm}^{-1}$ ;  $^1\text{H}$  RMN (400 MHz):  $\delta$  1.72-1.85 (m, 2H), 2.04 (td,  $J = 12.4$  Hz, 1.2 Hz, 1H), 2.28-2.40 (m, 2H), 2.44-2.54 (m, 2H), 2.68 (dtd,  $J = 15.6, 5.2, 1.6$  Hz, 1H), 2.91 (s, 3H,  $\text{CH}_3\text{N}$ ), 2.94 (m, 1H), 3.20 (td,  $J = 12.8, 5.2$  Hz, 1H), 3.60 (t,  $J = 10$  Hz, 1H, H-2), 3.76 (s, 3H,  $\text{CH}_3\text{O}$ ), 4.01 (dd,  $J = 10, 8$  Hz, 1H, H-2), 4.84 (br s, 1H, H-3), 6.05 (s, 1H,  $\text{CHCl}_2$ ).  $^{13}\text{C}$  RMN (100 MHz):  $\delta$  29.4 ( $\text{CH}_2$  and  $\text{CH}_3$ ), 33.0 ( $\text{CH}_2$ ), 37.7 ( $\text{CH}_2$ ), 38.2 ( $\text{CH}_2$ ), 48.1 ( $\text{CH}_2$ ), 52.0 (CH), 53.1 ( $\text{CH}_3$ ), 65.2 (C), 66.8 (CH), 156.9 (CO), 161.8 (CO), 209.5 (CO); HRMS (ESI-TOF): Calcd for  $\text{C}_{14}\text{H}_{21}\text{Cl}_2\text{N}_2\text{O}_4$  351.0873 (M+1). Found 351.0877.



**10:** To a solution of 4-[(benzylamino)methyl]cyclohexanone ethylene acetal<sup>5</sup> (7.18 g, 27.47 mmol), and triethylamine (5.74 mL, 41.21 mmol) in  $\text{CH}_2\text{Cl}_2$  (70 mL) was added dichloroacetyl chloride (3.17 mL, 32.96 mmol) dropwise at  $0^\circ\text{C}$ . The mixture was stirred at rt for 1 h then poured into water and extracted with  $\text{CH}_2\text{Cl}_2$ . The organic extracts were dried, concentrated and treated with 10% HCl (100 mL) and THF (10 mL) overnight. The mixture was extracted with  $\text{CH}_2\text{Cl}_2$ , the organic extracts were dried, concentrated and purified by chromatography ( $\text{CH}_2\text{Cl}_2$ ) to yield **10** as a white solid (5.13 g, 58%): mp  $99-101^\circ\text{C}$ ; IR (NaCl) 3030, 2932, 2861, 1711, 1670  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 2 rotamers)  $\delta$  1.36-1.56 (m, 2H), 1.94-2.08 (m, 2H), 2.08-2.50 (m, 5H), 3.31 and 3.38 (2 d,  $J = 6.8$  Hz, 2H), 4.71 and 4.77 (2 s, 2H), 6.21 and 6.35 (2 s, 1H), 7.15-7.45 (m, 5H, ArH);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz, 2 rotamers)  $\delta$  30.1 ( $\text{CH}_2$ ), 34.4 and 35.5 (CH), 40.2 ( $\text{CH}_2$ ), 49.9 and 52.2 ( $\text{CH}_2$ ), 51.7 and 51.9 ( $\text{CH}_2$ ), 64.5 and 65.2 (CH), 126.3 and 127.8 (CH), 127.9 and 128.3 (CH), 128.9 and 129.3 (CH), 135.1 and 135.8 (C), 164.5 and 164.6 (CO), 209.8 and 210.9 (CO). HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{16}\text{H}_{20}\text{Cl}_2\text{NO}_2$  328.0866; found 328.0889.

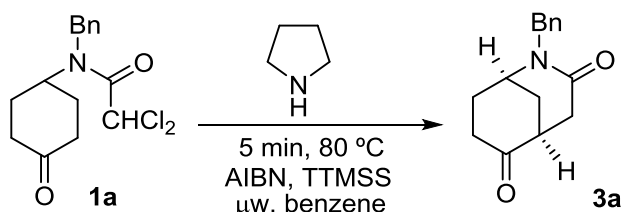
## 2. Synthesis of enamine 2

A mixture of **1a** (100 mg, 0.32 mmol) and pyrrolidine (0.027 mL, 0.32 mmol) in benzene (1 mL) was heated to reflux for 5 min then concentrated to yield enamine **2** as a yellowish oil.  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 400 MHz, 2 rotamers)  $\delta$  1.38-1.64 (m, 6H), 1.88-2.10 (m, 2H), 2.10-2.30 (m, 2H), 2.66-2.86 (m, 4H), 4.02 (m, 1H,  $\text{CH}=\text{C}$ ), 4.19 and 4.76 (2 m, 1H), 4.40 (d,  $J = 15.2$  Hz, 1H), 4.49 (d,  $J = 15.2$  Hz, 1H), 5.94 and 6.16 (2 s, 1H), 6.85-7.25 (m, 5H,

<sup>5</sup> Diaba, F.; Montiel, J. A.; Serban, G.; Bonjoch, J. *Org. Lett.* **2015**, *17*, 3860–3863.

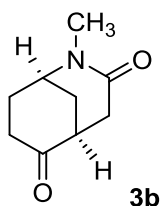
ArH);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz, 2 rotamers, some of the signals corresponding to the minor rotamer are not listed)  $\delta$  25.4 ( $\text{CH}_2$ ), 28.1 ( $\text{CH}_2$ ), 28.8 ( $\text{CH}_2$ ), 29.7 ( $\text{CH}_2$ ), 46.4 ( $\text{CH}_2$ ), 47.84 ( $\text{CH}_2$ ), 53.8 and 55.9 (CH), 66.5 and 67.3 (CH), 90.0 and 90.3 ( $\text{CH}=\text{}$ ), 126.3 (CH), 127.4 and 127.5 (CH), 128.9 and 129.0 (CH), 129.5 (CH), 138.3 and 139.5 (C), 142.3 and 142.5 (C), 164.4 and 164.7 (CO).

### 3. Synthesis of morphans 3a-3f



#### Typical procedure for the radical cyclization using microwave activation from 1a.

In a 10 mL vessel were placed **1a** (200 mg, 0.64 mmol), pyrrolidine (0.266 mL, 3.18 mmol), AIBN (105 mg, 0.64 mmol) and TTMSS (0.39 mL, 1.27 mmol) in benzene (1 mL) and the mixture was heated with stirring to 80 °C using microwave irradiation for 5 min. After chromatography ( $\text{CH}_2\text{Cl}_2$ ) **3a**<sup>6</sup> was obtained as a white solid (120 mg, 77%)<sup>7</sup>.

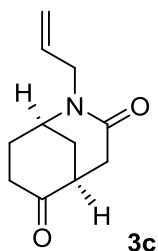


**3b**: IR (NaCl) 2942, 2880, 1708, 1624  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.81-1.91 (m, 1H, H-8ax), 2.11 (ddt,  $J = 13.6, 3.2, 2.4$  Hz, 1H, H-9), 2.26 (dq,  $J = 13.6, 3.2$  Hz, 1H, H-9), 2.29-2.38 (m, 1H, H-8eq), 2.38-2.44 (m, 2H,  $\text{CH}_2$ -7), 2.44 (d,  $J = 18.4$  Hz, 1H, H-4), 2.70 (dd,  $J = 18.4, 6.8$  Hz, 1H, H-4), 2.83 (m, 1H, H-5), 3.05 (s, 3H,  $\text{CH}_3$ ), 3.67 (m, 1H, H-1);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  29.7 (C-8), 31.9 (C-9), 33.8 ( $\text{CH}_3$ ), 33.9 (C-7), 35.0 (C-4), 44.4 (C-5), 53.3 (C-1), 168.4 (C-3), 210.8 (C-6); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_9\text{H}_{14}\text{NO}_2$  168.1019; found 168.1017.

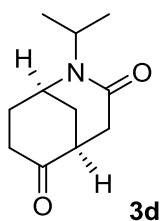
<sup>6</sup> For NMR data of **3a** see: Quirante, J.; Escolano, C.; Diaba, F.; Bonjoch, J. *J. Chem. Soc., Perkin Trans. 1* **1999**, 1157–1162.

<sup>7</sup> For the yields of **3b-3f** see the article.

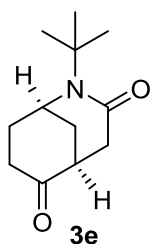




**3c:** IR (NaCl) 2933, 2885, 2853, 1712, 1637  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.85 (tdd,  $J = 14, 5.6, 2.4$  Hz, 1H, H-8ax), 2.13 (ddt,  $J = 13.6, 2.8, 2.4$  Hz, 1H, H-9), 2.20 (dq,  $J = 13.6, 3.2$  Hz, 1H, H-9), 2.27-2.35 (m, 1H, H-8eq), 2.35-2.53 (m, 3H,  $\text{CH}_2$ -7 and H-4), 2.74 (dd,  $J = 18.4, 7.2$  Hz, 1H, H-4), 2.84 (m, 1H, H-5), 3.60 (dd,  $J = 15.2, 6.4$  Hz, 1H), 3.74 (m, 1H, H-1), 4.61 (ddt,  $J = 15.2, 5.6, 1.2$  Hz, 1H), 5.19-5.26 (m, 2H), 5.80-5.92 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  30.2 (C-8), 32.2 (C-9), 34.0 (C-7), 34.9 (C-4), 44.2 (C-5), 48.1 ( $\text{CH}_2$ ), 50.3 (C-1), 117.7 ( $\text{CH}_2$ ), 132.9 (CH), 167.9 (C-3), 210.9 (C-6); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{11}\text{H}_{16}\text{NO}_2$  194.1176; found 194.1168.

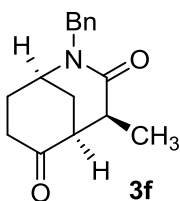


**3d:** mp 114-115  $^\circ\text{C}$ ; IR (NaCl) 2966, 2942, 2873, 1714, 1620  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.27 (d,  $J = 6.8$  Hz, 3H,  $\text{CH}_3$ ), 1.30 (d,  $J = 6.8$  Hz, 3H,  $\text{CH}_3$ ), 1.93 (tdd,  $J = 13.6, 4.8, 2.4$  Hz, 1H, H-8ax), 2.08 (dq,  $J = 13.2, 3.2$  Hz, 1H, H-9), 2.14 (dq,  $J = 13.2, 2$  Hz, 1H, H-9), 2.19-2.29 (m, 1H, H-8eq), 2.36 (dd,  $J = 15.6, 4.8$  Hz, 1H, H-7), 2.45 (dd,  $J = 18.8, 1.2$  Hz, 1H, H-4), 2.56 (ddd,  $J = 15.6, 14, 6.8$  Hz, 1H, H-7), 2.71 (dd,  $J = 18.8, 7.6$  Hz, 1H, H-4), 2.79 (m, 1H, H-5), 3.82 (br s, 1H, H-1), 4.64 (sept,  $J = 6.8$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  20.0 ( $\text{CH}_3$ ), 20.7 ( $\text{CH}_3$ ), 33.0 (C-8), 33.4 (C-9), 33.8 (C-7), 35.4 (C-4), 43.7 (C-5), 47.0 (C-1), 47.4 (CH), 167.9 (C-3), 211.2 (C-6); HRMS (ESI-TOF)  $m/z$ :  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{11}\text{H}_{18}\text{NO}_2$  196.1332; found 196.1332.



**3e:** mp 133-135  $^\circ\text{C}$ ; IR (NaCl) 2990, 2959, 2943, 2913, 2870, 1716, 1622  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.54 (s, 9H,  $\text{CH}_3$ ), 1.93 (tdd,  $J = 14, 5.2, 2.8$  Hz, 1H, H-8ax), 2.08-

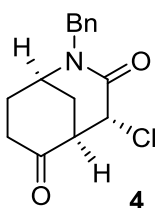
2.19 (m, 2H, CH<sub>2</sub>-9), 2.19-2.27 (m, 1H, H-8eq), 2.34 (dd, *J* = 15.2, 5.2 Hz, 1H, H-7), 2.37-2.45 (m, 1H, H-4), 2.53 (ddd, *J* = 15.2, 14, 6.8 Hz, 1H, H-7), 2.68-2.76 (m, 2H, H5 and H-4), 4.09 (m, 1H, H-1); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 28.7 (CH<sub>3</sub>), 33.2 (C-7), 33.7 (C-8 and C-9), 37.1 (C-4), 43.8 (C-5), 47.4 (C-1), 58.0 (C), 168.5 (C-3), 211.3 (C-6); HRMS (ESI-TOF) *m/z*: [M+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>20</sub>NO<sub>2</sub> 210.1489; found 210.1487.



**3f**: mp 189-191 °C; IR (NaCl) 3062, 3029, 2961, 2936, 2870, 1710, 1635 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.22 (d, *J* = 6.8 Hz, 3H, CH<sub>3</sub>), 1.74 (ddd, *J* = 13.2, 5.2, 2.4 Hz, 1H, H-8ax), 2.07 (dt, *J* = 13.2, 2.4 Hz, 1H, H-9), 2.14-2.26 (m, 2H, H-8eq and H-9), 2.27-2.34 (m, 1H, H-7), 2.40 (dd, *J* = 15.2, 4.8 Hz, 1H, H-7), 2.78 (m, 2H, H-4 and H-5), 3.68 (br s, 1H, H-1), 4.04 (d, *J* = 15 Hz, 1H), 5.35 (d, *J* = 15 Hz, 1H), 7.25-7.38 (m, 5H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 14.5 (CH<sub>3</sub>), 29.3 (C-8), 32.9 (C-9), 35.1 (C-7), 39.4 (C-4), 48.5 (CH<sub>2</sub>), 50.6 (C-1 and C-5), 127.6 (CH), 127.9 (CH), 128.8 (CH), 137.4 (C), 171.9 (C-3), 210.5 (C-6); HRMS (ESI-TOF) *m/z*: [M+H]<sup>+</sup> calcd for C<sub>16</sub>H<sub>20</sub>NO<sub>2</sub> 258.1489; found 258.1485.

#### 4. Obtention of 4

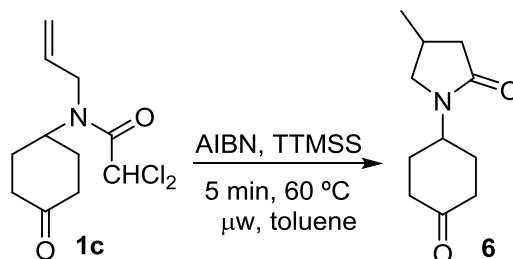
In a 10 mL vessel were placed **1a** (100 mg, 0.32 mmol), pyrrolidine (0.13 mL, 1.59 mmol) and benzene (1 mL) and the mixture was heated with stirring to 80 °C using microwave irradiation for 5 min. The mixture was then purified by chromatography (CH<sub>2</sub>Cl<sub>2</sub>) to provide recovered **1a** (31 mg, 31%) and **4** (6 mmg, 7%) as a white solid.



**4**: mp 85-87 °C; IR (NaCl) 3065, 3055, 3031, 2944, 2932, 2875, 2864, 2852, 1714, 1652 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.81 (tdd, *J* = 12.8, 5.6, 2.4 Hz, 1H, H-8ax), 1.99 (dm, *J* = 14.4 Hz, 1H, H-9), 2.17-2.26 (m, 1H, H-8eq), 2.35 (ddd, *J* = 16.4, 12.8, 7.2 Hz, 1H, H-7ax), 2.49 (dd, *J* = 16.4, 6 Hz, 1H, H-7eq), 2.70 (dq, *J* = 14.4, 3.2 Hz, 1H, H-9), 3.08 (br s, 1H, H5), 3.73 (br s, 1H, H-1), 4.03 (d, *J* = 15 Hz, 1H), 4.37 (t, *J* = 1.6 Hz, 1H, H-4), 5.39 (d, *J* = 15 Hz, 1H), 7.28-7.40 (m, 5H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 27.5 (C-9),

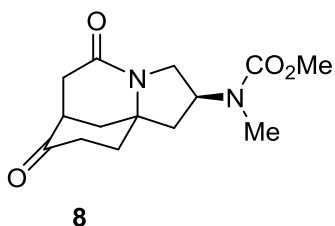
28.9 (C-8), 35.0 (C-7), 48.5 (CH<sub>2</sub>), 50.3 (C-1), 53.4 (C-5), 54.1 (C-4), 127.8 (CH), 128.0 (CH), 129.0 (CH), 136.3 (C), 165.7 (C-3), 206.7 (C-6); HRMS (ESI-TOF) m/z: [M+H]<sup>+</sup> calcd for C<sub>15</sub>H<sub>17</sub>ClNO<sub>2</sub> 178.0942; found 178.0937.

## 5. Synthesis of 6



In a 10 mL vessel were placed **1c** (100 mg, 0.38 mmol), AIBN (63 mg, 0.38 mmol) and TTMSS (0.23 mL, 0.76 mmol) in toluene (1 mL) and the mixture was heated with stirring to 60 °C using microwave irradiation for 5 min. After chromatography (CH<sub>2</sub>Cl<sub>2</sub> to AcOEt) **6** was obtained as a white solid (42 mg, 57%). mp 70-71 °C; IR (NaCl) 2957, 2872, 1715, 1668 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.13 (d, *J* = 7.2 Hz, 3H, CH<sub>3</sub>), 1.78-1.93 (m, 2H), 1.98-2.09 (m, 3H), 2.38-2.62 (m, 6H), 2.89 (dd, *J* = 9.6, 6 Hz, 1H), 3.46 (dd, *J* = 9.6, 7.6 Hz, 1H), 4.48 (tt, *J* = 12, 4 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 19.7 (CH<sub>3</sub>), 26.6 (CH), 29.2 (CH<sub>2</sub>), 29.3 (CH<sub>2</sub>), 39.6 (CH<sub>2</sub>), 48.2 (CH), 50.0 (CH<sub>2</sub>), 174.2 (CO), 209.3 (CO); HRMS (ESI-TOF) m/z: [M+H]<sup>+</sup> calcd for C<sub>11</sub>H<sub>18</sub>NO<sub>2</sub> 196.1332; found 196.1334.

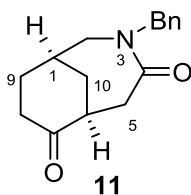
## 6. Synthesis of 8



In a 10 mL vessel were placed **7** (100 mg, 0.28 mmol), pyrrolidine (0.12 mL, 1.43 mmol), AIBN (47 mg, 0.29 mmol) and TTMSS (0.18 mL, 0.57 mmol) in toluene (0.5 mL) and the mixture was heated with stirring to 60 °C using microwave irradiation for 5 min. After chromatography (CH<sub>2</sub>Cl<sub>2</sub> to CH<sub>2</sub>Cl<sub>2</sub>/AcOEt/MeOH 49.5:49.5:1) **8** (27 mg, 34%) then **9** (11 mg, 14%) were isolated.<sup>8</sup>

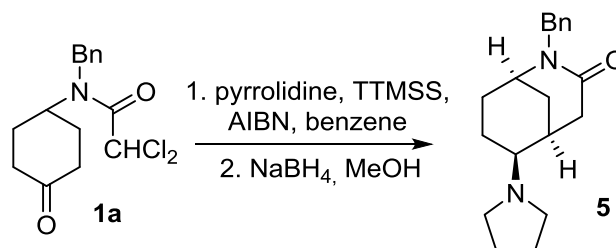
<sup>8</sup> For NMR data of **8** and **9** see ref. 4.

## 7. Synthesis of 11



In a 10 mL vessel were placed **10** (100 mg, 0.30 mmol), pyrrolidine (0.13 mL, 1.52 mmol), AIBN (50 mg, 0.30 mmol) and TTMSS (0.19 mL, 0.61 mmol) in benzene (1 mL) and the mixture was heated with stirring to 80 °C using microwave irradiation for 5 min. After chromatography (CH<sub>2</sub>Cl<sub>2</sub> to CH<sub>2</sub>Cl<sub>2</sub>/AcOEt 1:1) **11** was obtained (40 mg, 51%) as a white solid. mp 200-202 °C; IR (NaCl) 3060, 3029, 2928, 2868, 1708, 1641 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.64-1.72 (m, 1H, H-9), 1.85-2.02 (m, 3H, H-9 and CH<sub>2</sub>-10), 2.05 (br s, 1H, H-1), 2.33 (ddd, *J* = 17, 7.6, 2.8 Hz, 1H, H-8), 2.54 (ddd, *J* = 17, 10.8, 8.4 Hz, 1H, H-8), 2.77 (br s, 1H, H-6), 2.76-2.90 (m, 2H, CH<sub>2</sub>-5), 3.44 (m, 2H, CH<sub>2</sub>-2), 4.49 (d, *J* = 14.8 Hz, 1H, CH<sub>2</sub>Ar), 4.79 (d, *J* = 14.8 Hz, 1H, CH<sub>2</sub>Ar), 7.27-7.36 (m, 5H, ArH); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) 27.3 (C-9), 27.8 (C-1), 33.6 (C-10), 34.7 (C-8), 38.5 (C-5), 42.2 (C-6), 50.8 (C-2), 51.7 (CH<sub>2</sub>Ar), 127.7 (CH), 128.5 (CH), 128.7 (CH), 137.2 (C), 171.7 (C-4), 211.6 (C-7); HRMS (ESI-TOF) *m/z*: [M+H]<sup>+</sup> calcd for C<sub>16</sub>H<sub>20</sub>NO<sub>2</sub> 258.1489; found 258.1487.

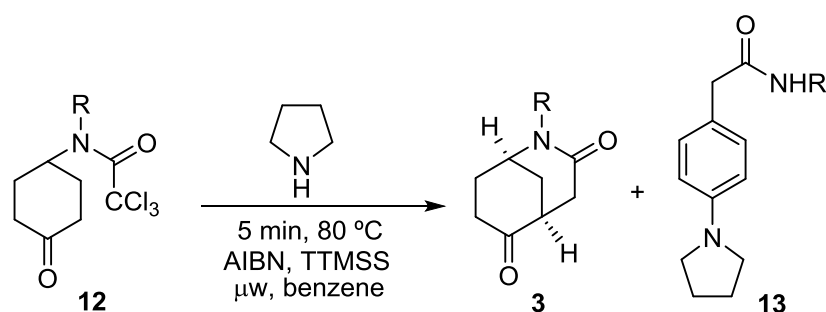
## 8. Synthesis of 5



In a 10 mL vessel were placed **1a** (200 mg, 0.64 mmol), pyrrolidine (0.266 mL, 3.18 mmol), AIBN (105, 0.64 mmol) and TTMSS (0.39 mL, 1.27 mmol) in benzene (1 mL) and the mixture was heated with stirring to 80 °C using microwave irradiation for 5 min. MeOH (0.5 mL) was added and the mixture was treated with NaBH<sub>4</sub> (25 mg, 0.64 mmol) at 0 °C then at rt for 1 h. The reaction mixture was concentrated, water was added and the mixture extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic extracts were dried, concentrated and purified by chromatography (CH<sub>2</sub>Cl<sub>2</sub> to (CH<sub>2</sub>Cl<sub>2</sub>,NH<sub>3</sub>)/MeOH 9.5:0.5) to yield **5** as a white

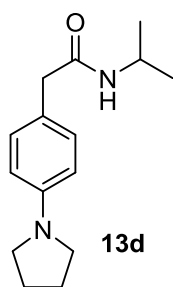
solid (115 mg, 61%).<sup>9</sup> mp 89-91 °C; IR (NaCl) 3029, 2932, 2872, 2777, 1634 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.32-1.48 (m, 2H, H-7 and H-8), 1.64 (dd, *J* = 13.2, 2 Hz, 1H, H-9), 1.77 (br s, 6H), 1.91 (dq, *J* = 13.2, 2.8 Hz, 1H, H-9), 2.16 (br s, 1H, H-6), 2.35 (br s, 1H, H-5), 2.44 (dd, *J* = 18, 7.2 Hz, 1H, H-4), 2.56 (br s, 4H), 2.98 (d, *J* = 18 Hz, 1H, H-4), 3.41 (br s, 1H, H-1), 3.95 (d, *J* = 15.2 Hz, 1H), 5.23 (d, *J* = 15.2 Hz, 1H), 7.22-7.34 (m, 5H, ArH); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 22.9 (C-7), 23.3 (CH<sub>2</sub>), 27.9 (C-8), 30.8 (C-5), 31.3 (C-4), 31.5 (C-9), 48.2 (CH<sub>2</sub>), 50.6 (C-1), 51.5 (CH<sub>2</sub>), 66.0 (C-6), 127.2 (CH), 127.8 (CH), 128.5 (CH), 137.9 (C), 171.0 (C-3); HRMS (ESI-TOF) *m/z*: [M+H]<sup>+</sup> calcd for C<sub>19</sub>H<sub>27</sub>N<sub>2</sub>O 299.2118; found 299.2125.

### 9. Radical cyclization from trichloroacetamides **12a-12d**



#### Typical procedure for the radical cyclization using microwave activation from **12d**.

In a 10 mL vessel were placed **12d** (100 mg, 0.33 mmol), pyrrolidine (0.14 mL, 1.66 mmol), AIBN (54.6 mg, 0.33 mmol) and TTMSS (0.20 mL, 0.66 mmol) in benzene (1 mL) and the mixture was heated with stirring to 80 °C using microwave irradiation for 5 min. After chromatography (CH<sub>2</sub>Cl<sub>2</sub>) **13d** (14 mg, 16%) was isolated then **2d** (37 mg, 57%).<sup>10</sup>

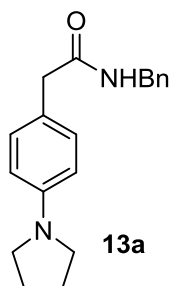


**13d**: IR (NaCl) 3249, 3069, 2960, 2929, 2870, 2818, 1643, 1617 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.04 (d, *J* = 6.8 Hz, 6H, CH<sub>3</sub>), 2.01 (m, 4H), 3.29 (m, 4H), 3.44 (s, 2H), 4.05 (m, 1H), 5.21 (br s, 1H, NH), 6.54 (d, *J* = 8.4 Hz, 2H), 7.06 (d, *J* = 8.4 Hz, 2H); <sup>13</sup>C NMR

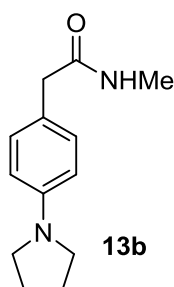
<sup>9</sup> The other epimer at C-6 was observed as traces in some fractions but was not isolated.

<sup>10</sup> For the products obtained with the other substrates and their yields see the article.

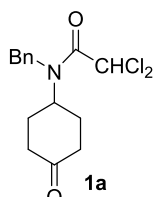
(CDCl<sub>3</sub>, 100 MHz)  $\delta$  22.6 (CH<sub>3</sub>), 25.4 (CH<sub>2</sub>), 41.2 (CH), 43.1 (CH<sub>2</sub>), 47.6 (CH<sub>2</sub>), 112.1 (CH), 121.1 (C), 130.3 (CH), 147.1 (C), 171.4 (CO); HRMS (ESI-TOF) m/z: [M+H]<sup>+</sup> calcd for C<sub>15</sub>H<sub>23</sub>N<sub>2</sub>O 247.1804; found 247.1799.



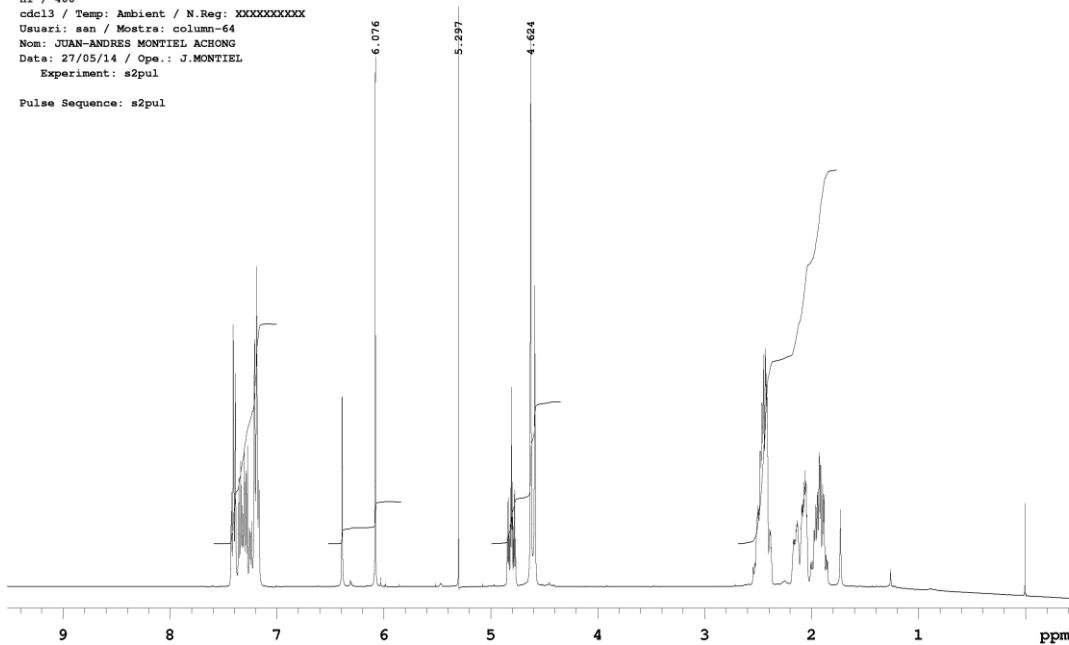
**13a:** <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  1.98-2.02 (m, 4H), 3.24-3.30 (m, 4H), 3.55 (s, 2H), 4.40 (d, *J* = 6 Hz, 2H), 5.75 (br s, 1H, NH), 6.53 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 7.15-7.32 (m, 5H, ArH); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  25.4 (CH<sub>2</sub>), 42.9 (CH<sub>2</sub>), 43.4 (CH<sub>2</sub>), 47.6 (CH<sub>2</sub>), 112.1 (CH), 119.9 (C), 127.3 (CH), 127.4 (CH), 128.6 (CH), 130.4 (CH), 138.4 (C), 147.2 (C), 172.2 (CO); HRMS (ESI-TOF) m/z: [M+H]<sup>+</sup> calcd for C<sub>19</sub>H<sub>23</sub>N<sub>2</sub>O 295.1805; found 295.1796.



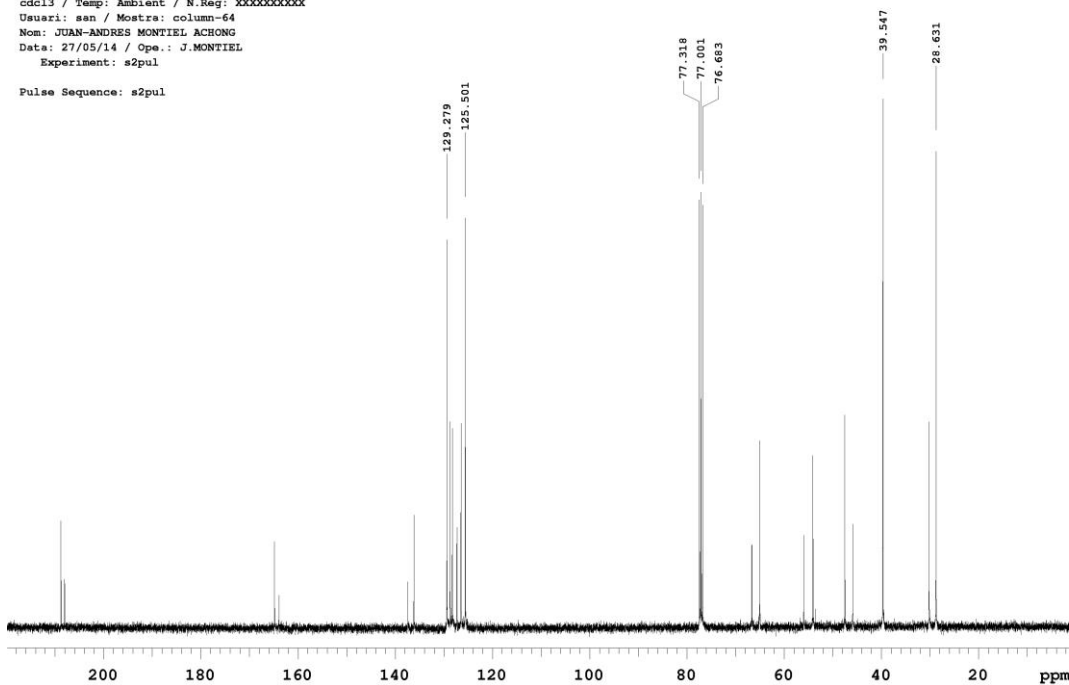
**13b:** IR (NaCl) 3292, 3094, 2964, 2926, 2850, 2822, 1646, 1617 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  2.01 (m, 4H), 2.73 (d, *J* = 4.8 Hz, 3H, CH<sub>3</sub>), 3.28 (m, 4H), 3.48 (s, 2H), 5.38 (br s, 1H, NH), 6.54 (d, *J* = 8.4 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  25.5 (CH<sub>2</sub>), 26.4 (CH<sub>3</sub>), 42.8 (CH<sub>2</sub>), 47.6 (CH<sub>2</sub>), 112.1 (CH), 121.0 (C), 130.5 (CH), 147.3 (C), 172.9 (CO); HRMS (ESI-TOF) m/z: [M+H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>19</sub>N<sub>2</sub>O 219.1492; found 219.1489.

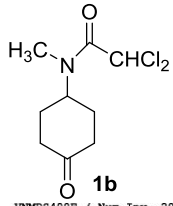


H1 / 400  
 cdcl3 / Temp: Ambient / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: column-64  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 27/05/14 / Ope.: J.MONTEIEL  
 Experiment: s2pul  
 Pulse Sequence: s2pul

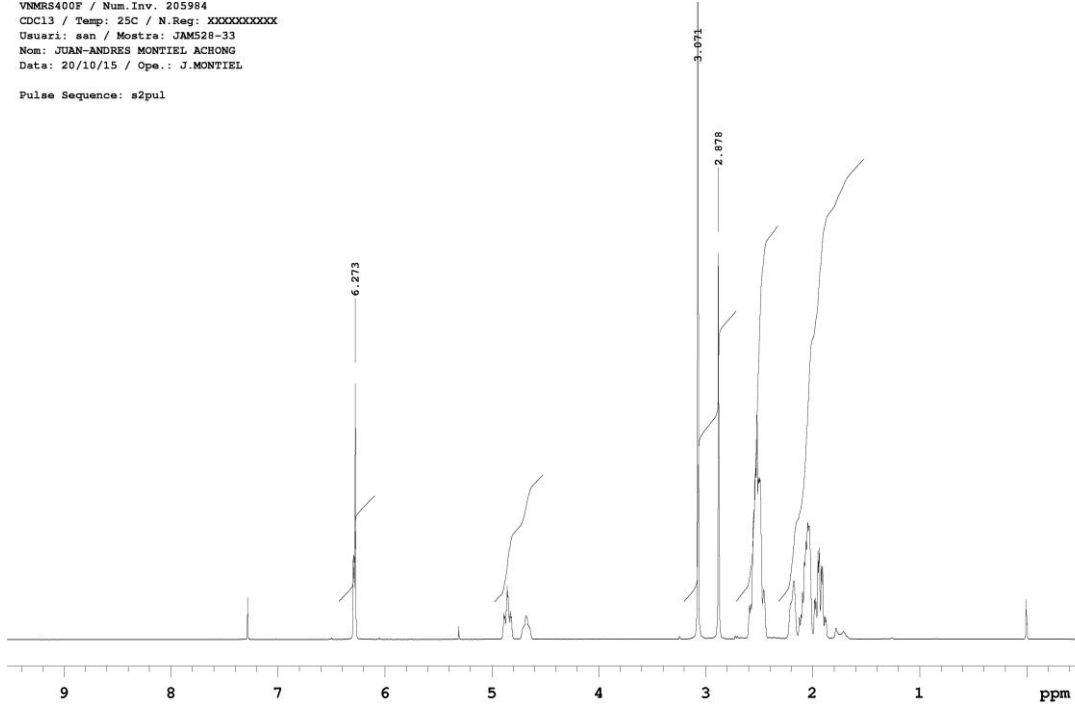


H1 / 400  
 cdcl3 / Temp: Ambient / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: column-64  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 27/05/14 / Ope.: J.MONTEIEL  
 Experiment: s2pul  
 Pulse Sequence: s2pul

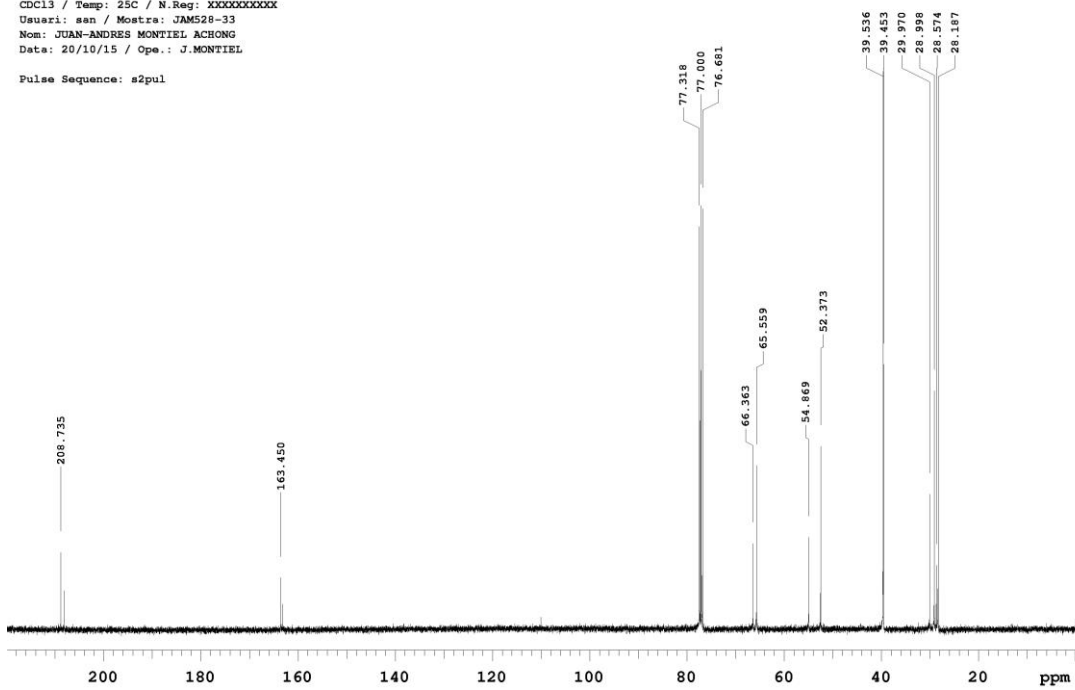




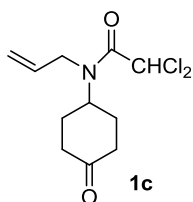
VNMRS400F / Num.Inv. 205984  
 CDCl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: JAM528-33  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 20/10/15 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul



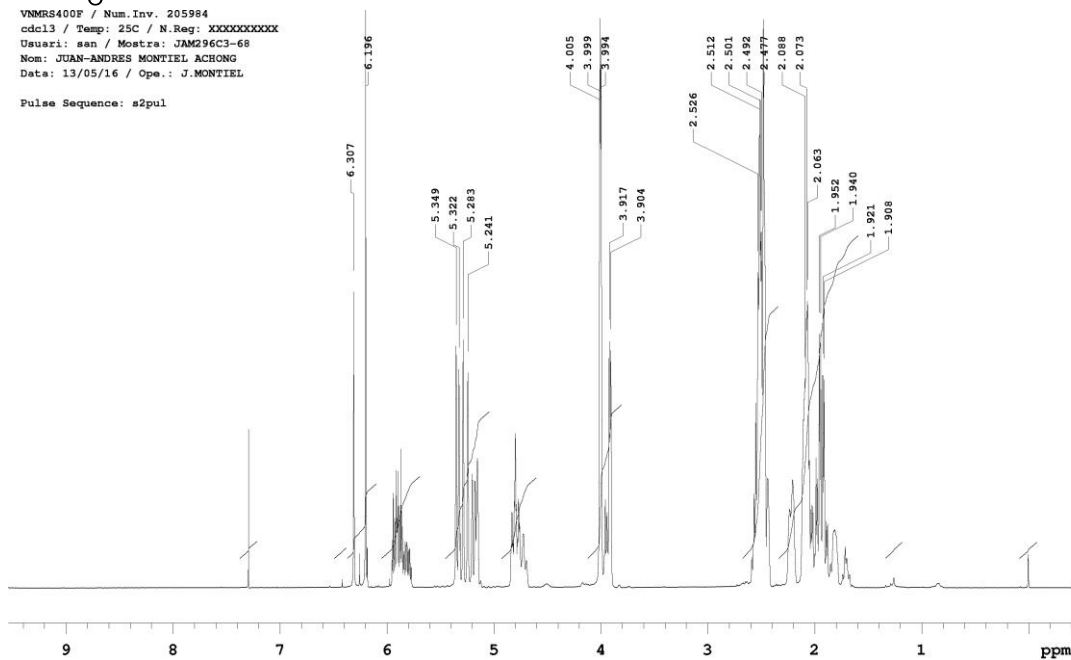
VNMRS400F / Num.Inv. 205984  
 CDCl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: JAM528-33  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 20/10/15 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul



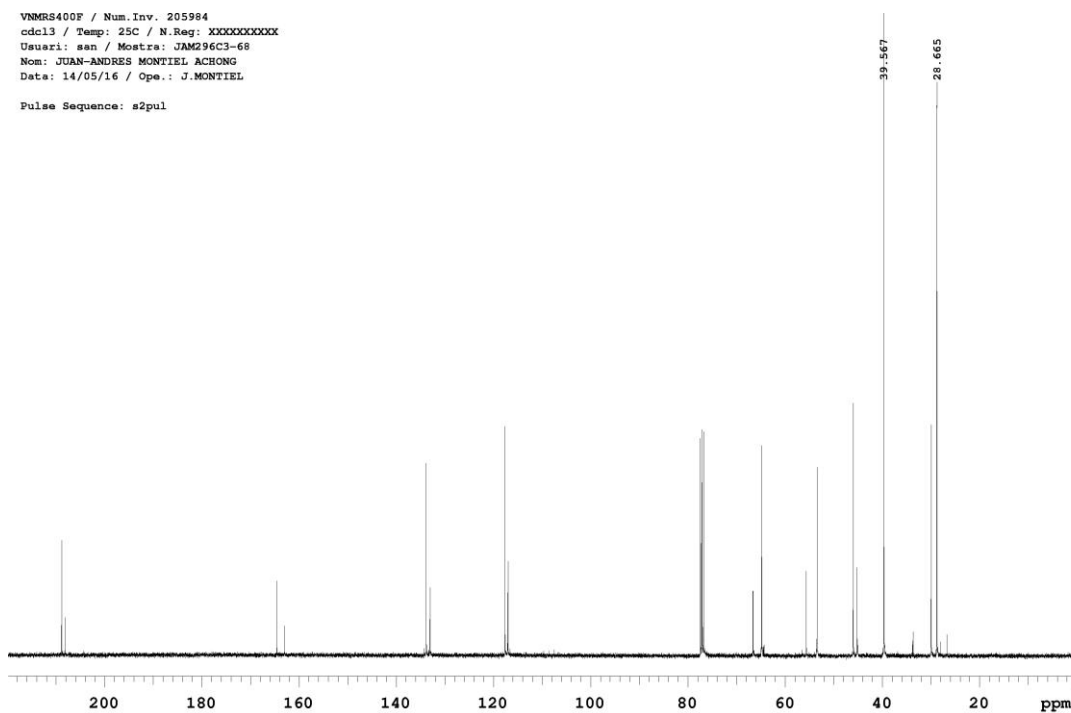


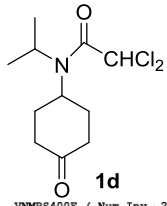


VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM296C3-68  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 13/05/16 / Ope.: J.MONTIEL  
 Pulse Sequence: s2pul

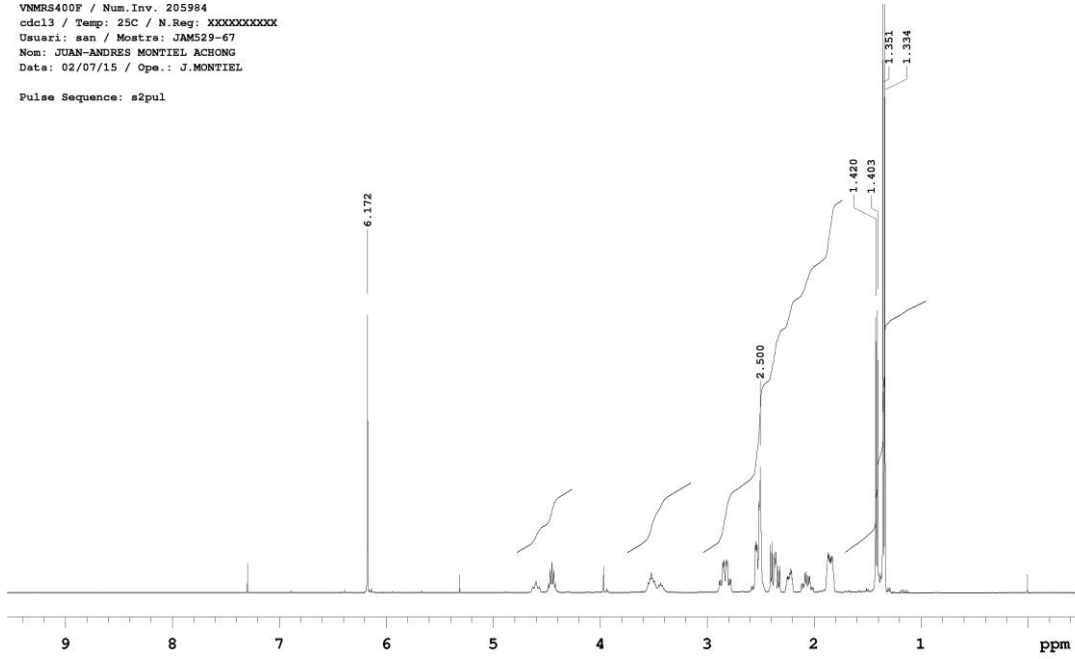


VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM296C3-68  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 14/05/16 / Ope.: J.MONTIEL  
 Pulse Sequence: s2pul

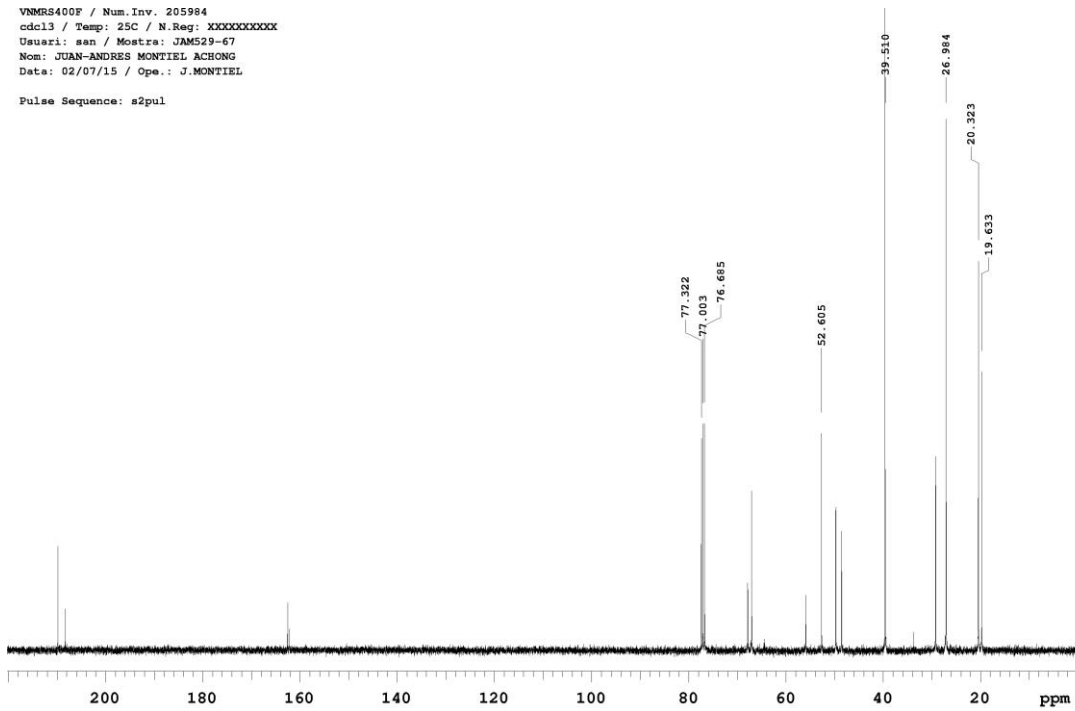


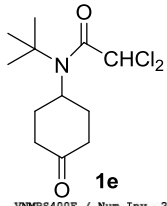


VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: JAM529-67  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 02/07/15 / Ope.: J.MONTIEL  
 Pulse Sequence: s2pul

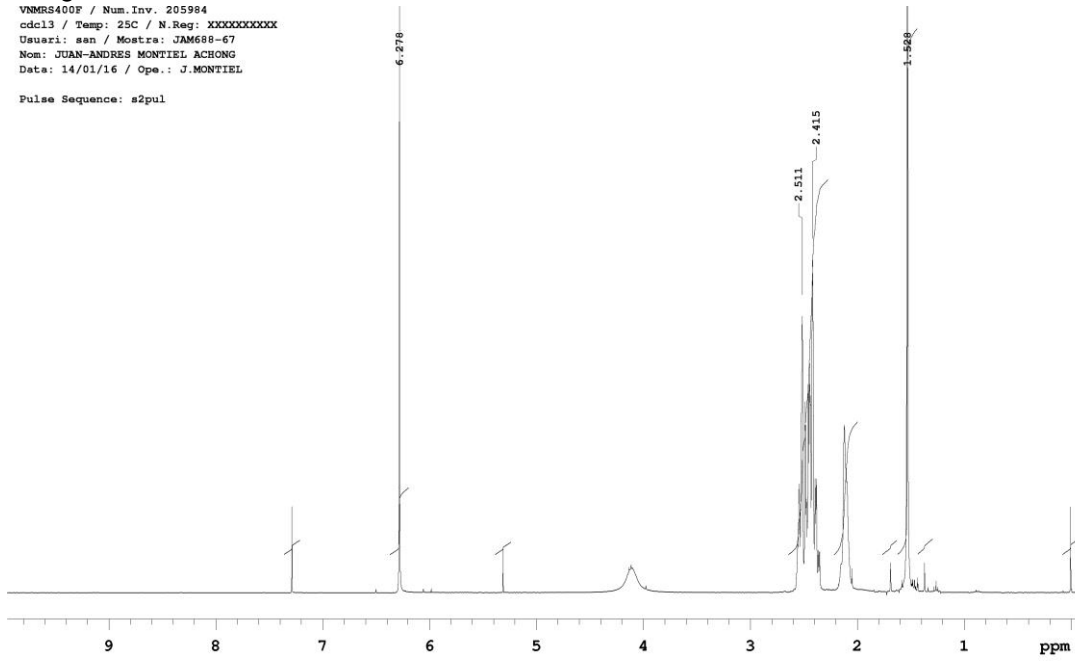


VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: JAM529-67  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 02/07/15 / Ope.: J.MONTIEL  
 Pulse Sequence: s2pul

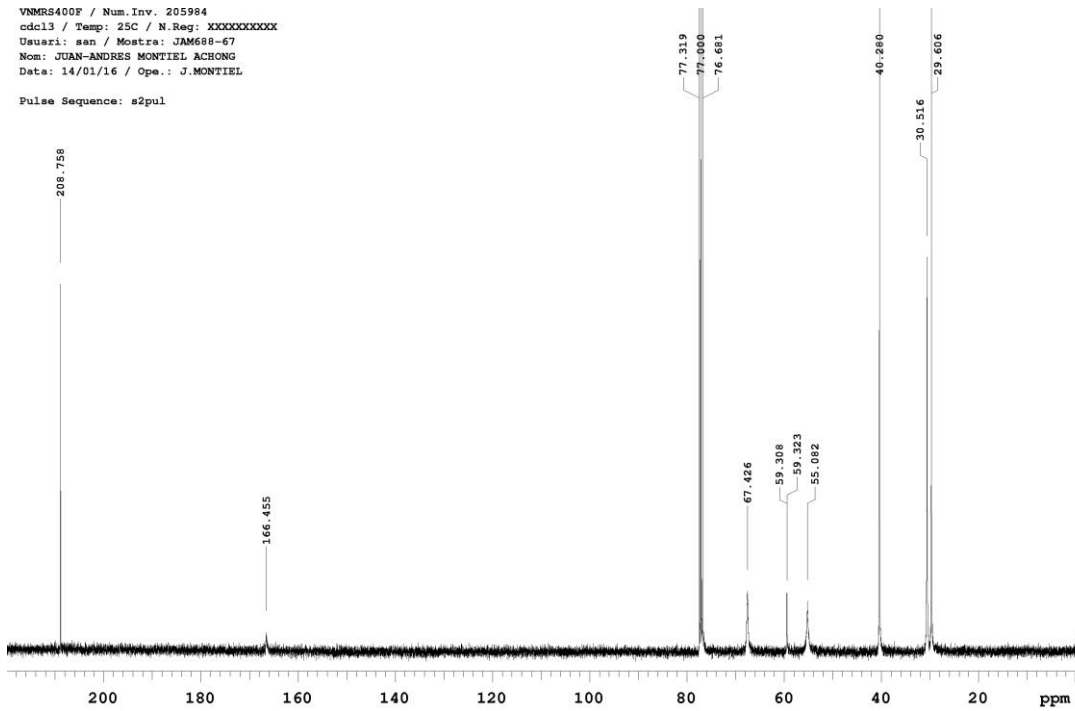


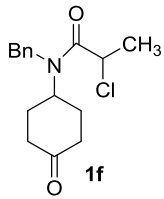


VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM688-67  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 14/01/16 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul



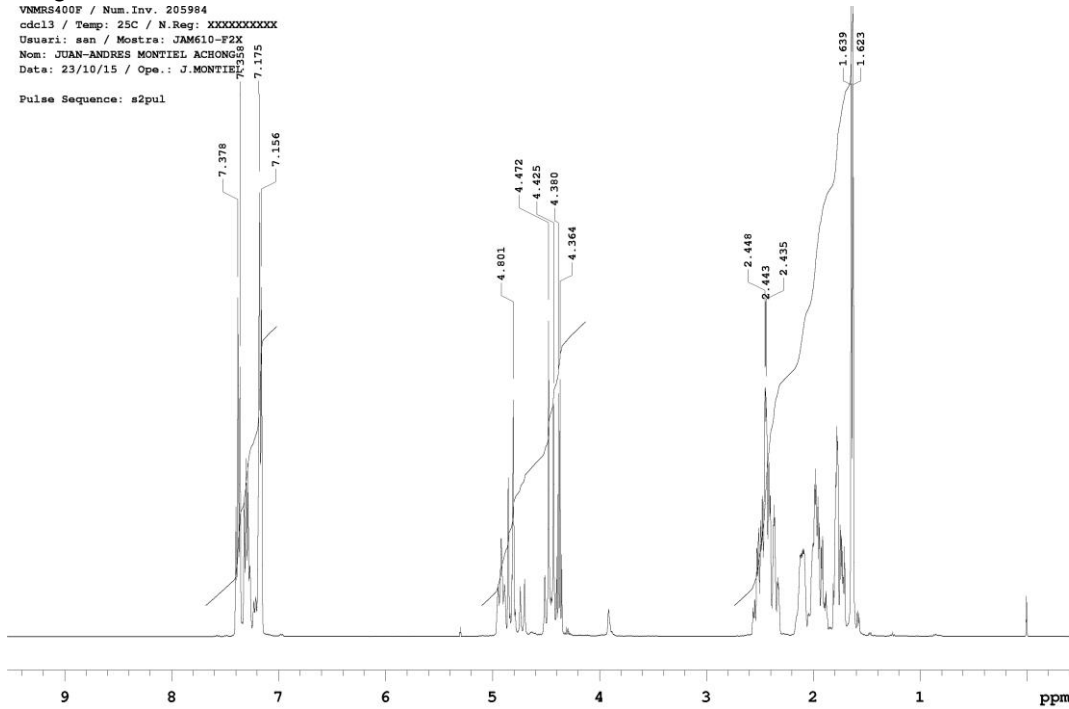
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM688-67  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 14/01/16 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul





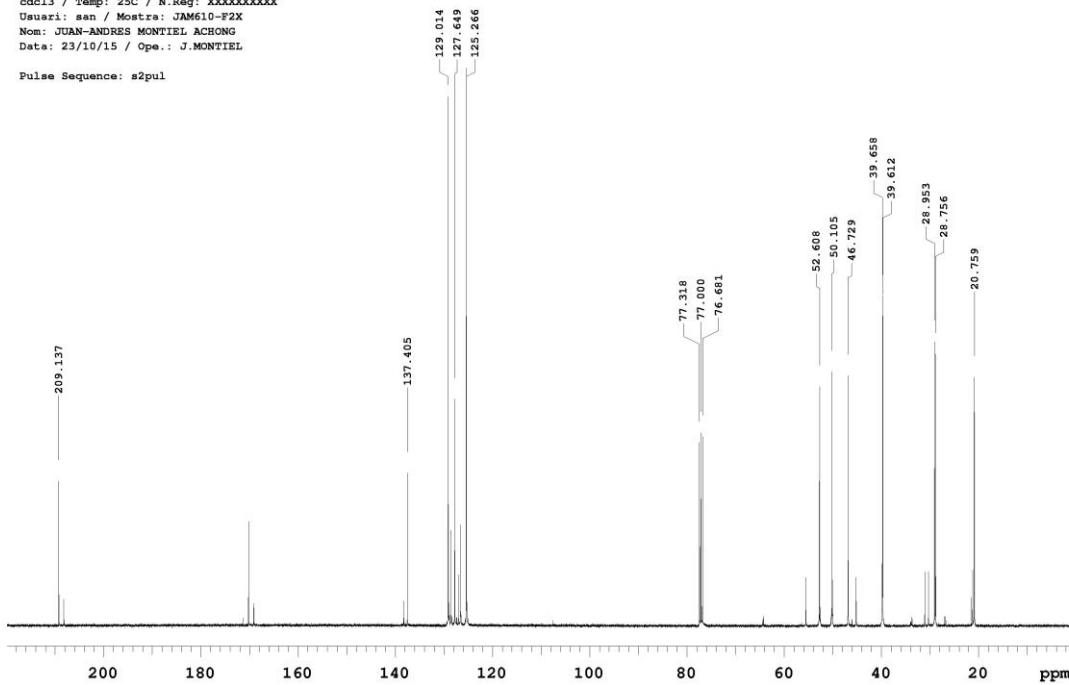
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM610-F2X  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 23/10/15 / Ope.: J.MONTIEL

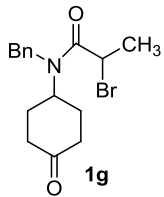
Pulse Sequence: s2pul



VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM610-F2X  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 23/10/15 / Ope.: J.MONTIEL

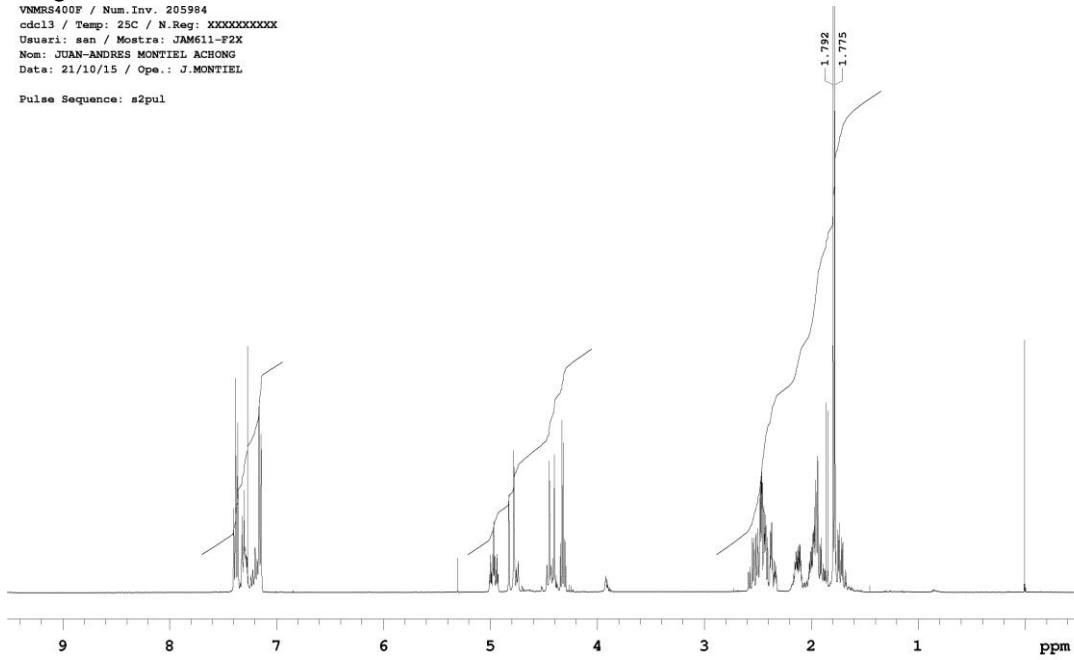
Pulse Sequence: s2pul





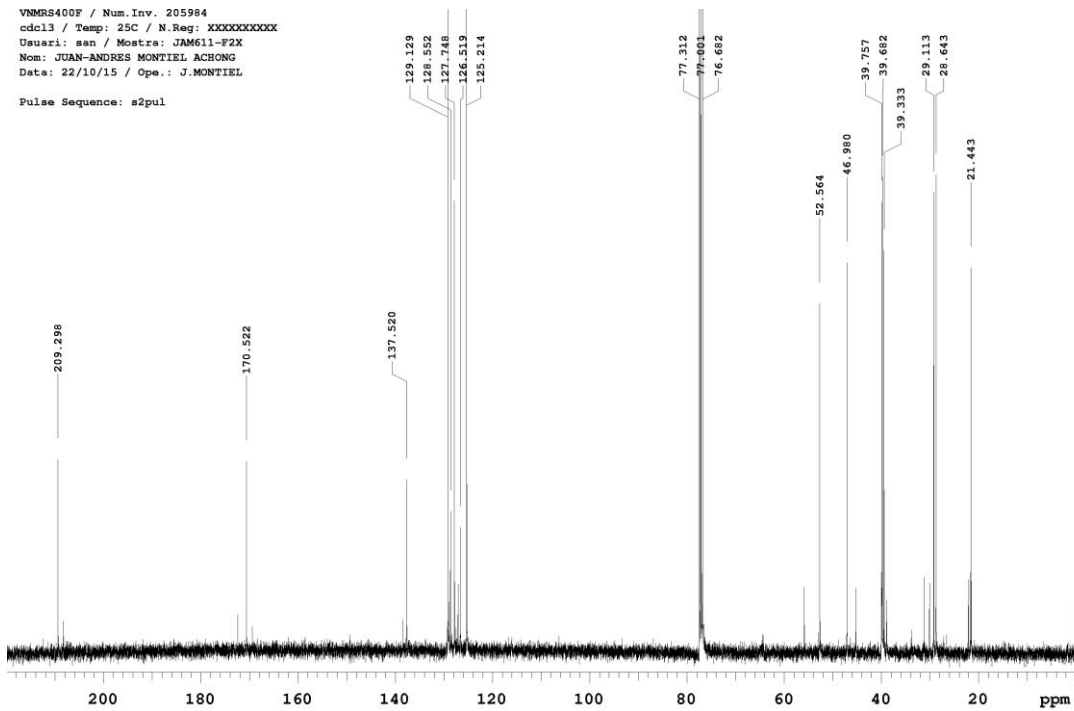
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM611-F2X  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 21/10/15 / Ope.: J.MONTIEL

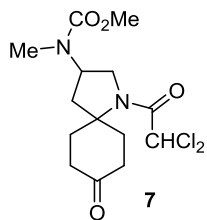
Pulse Sequence: s2pul



VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM611-F2X  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 22/10/15 / Ope.: J.MONTIEL

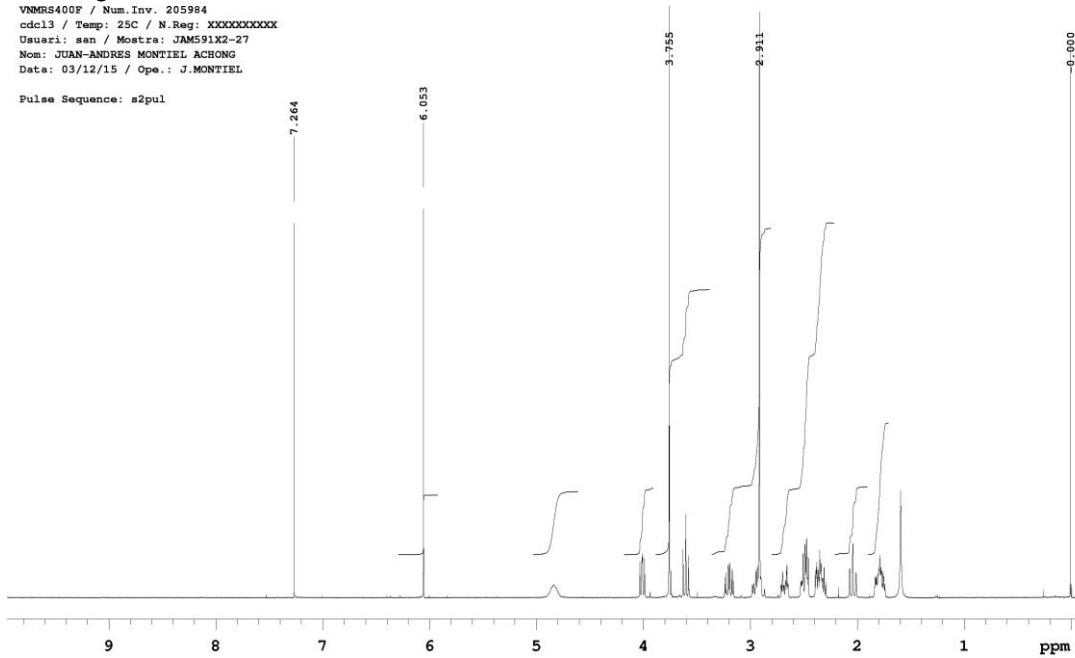
Pulse Sequence: s2pul





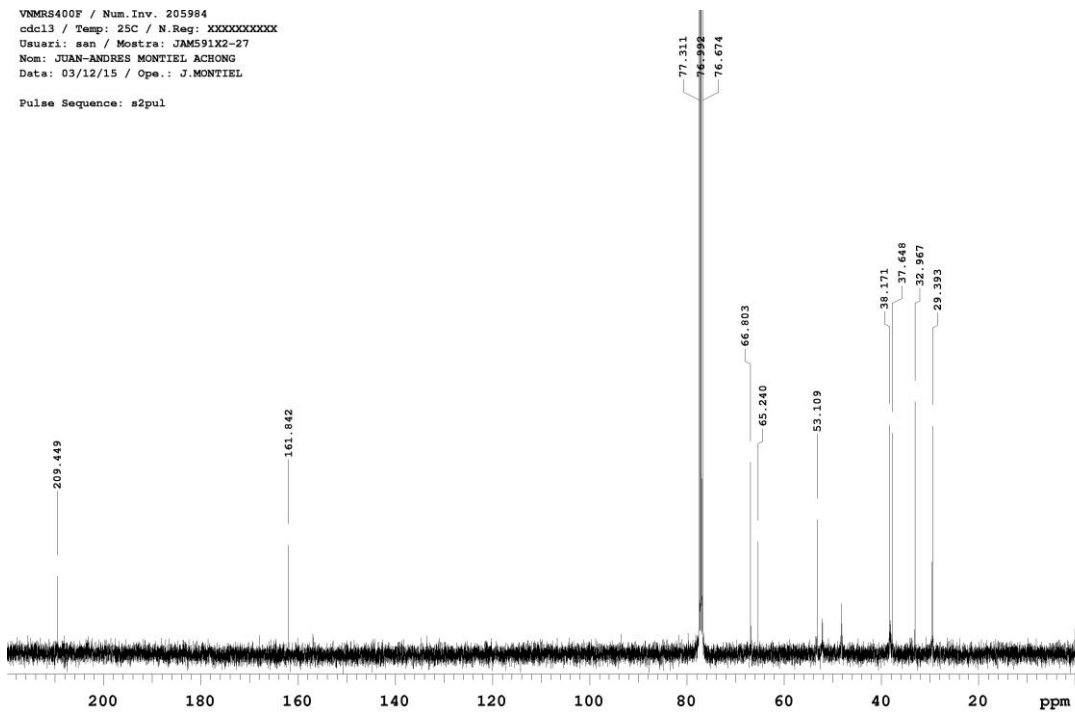
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: JAM591X2-27  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 03/12/15 / Ope.: J.MONTIEL

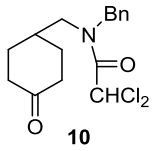
Pulse Sequence: s2pul



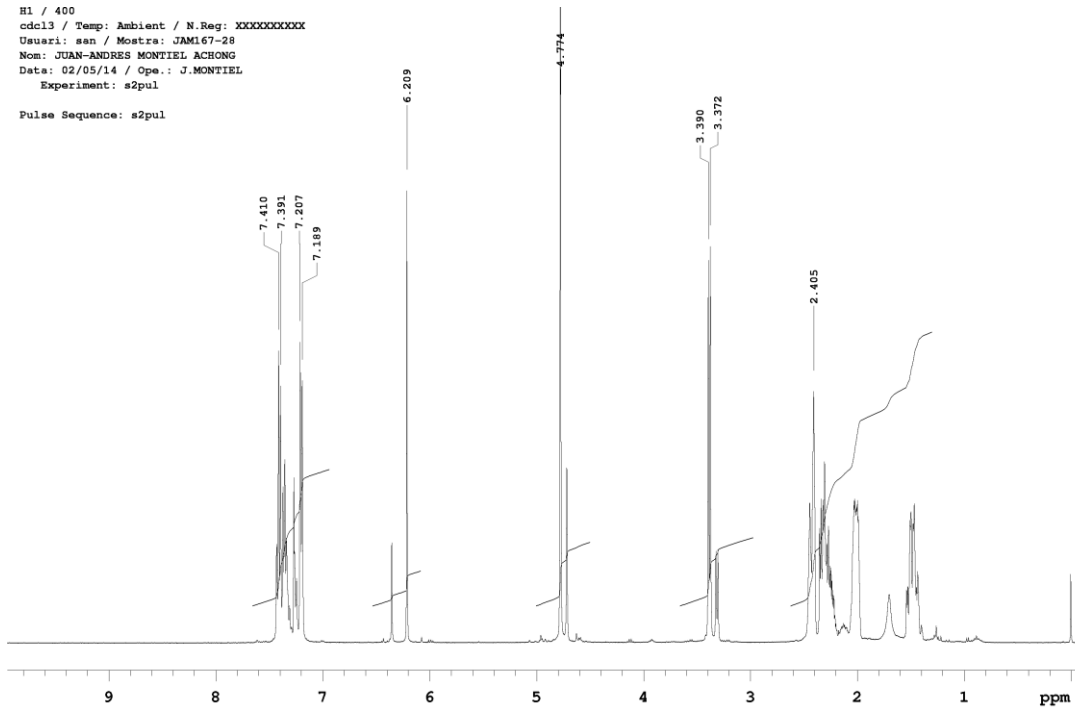
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: JAM591X2-27  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 03/12/15 / Ope.: J.MONTIEL

Pulse Sequence: s2pul

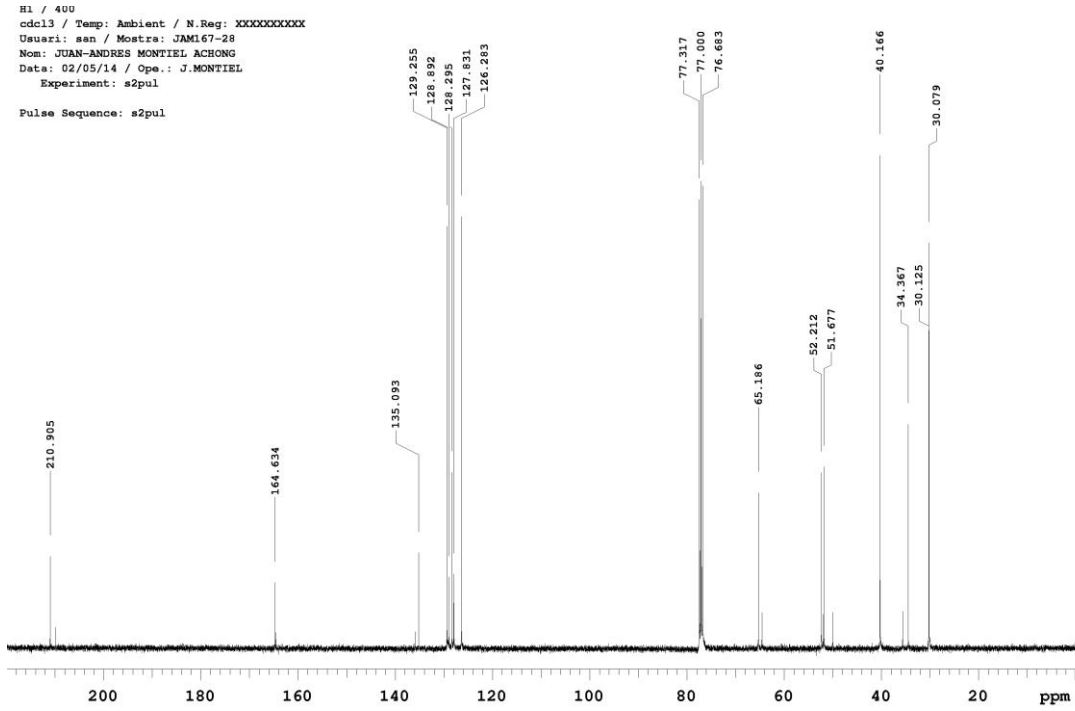


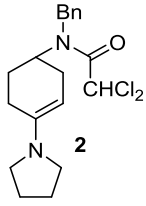


H1 / 400  
 cdcl3 / Temp: Ambient / N.Reg: XXXXXXXXXXXX  
 Usuario: sen / Mostra: JAM167-28  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 02/05/14 / Ope.: J.MONTEIEL  
 Experiment: s2pul  
 Pulse Sequence: s2pul

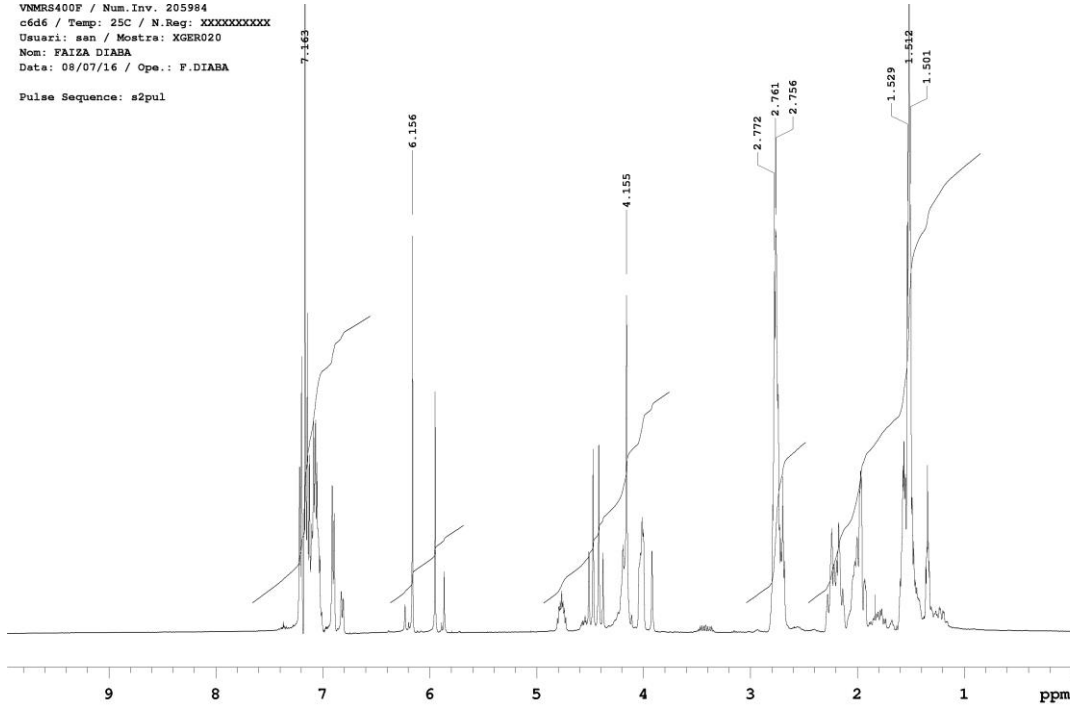


H1 / 400  
 cdcl3 / Temp: Ambient / N.Reg: XXXXXXXXXXXX  
 Usuario: sen / Mostra: JAM167-28  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 02/05/14 / Ope.: J.MONTEIEL  
 Experiment: s2pul  
 Pulse Sequence: s2pul

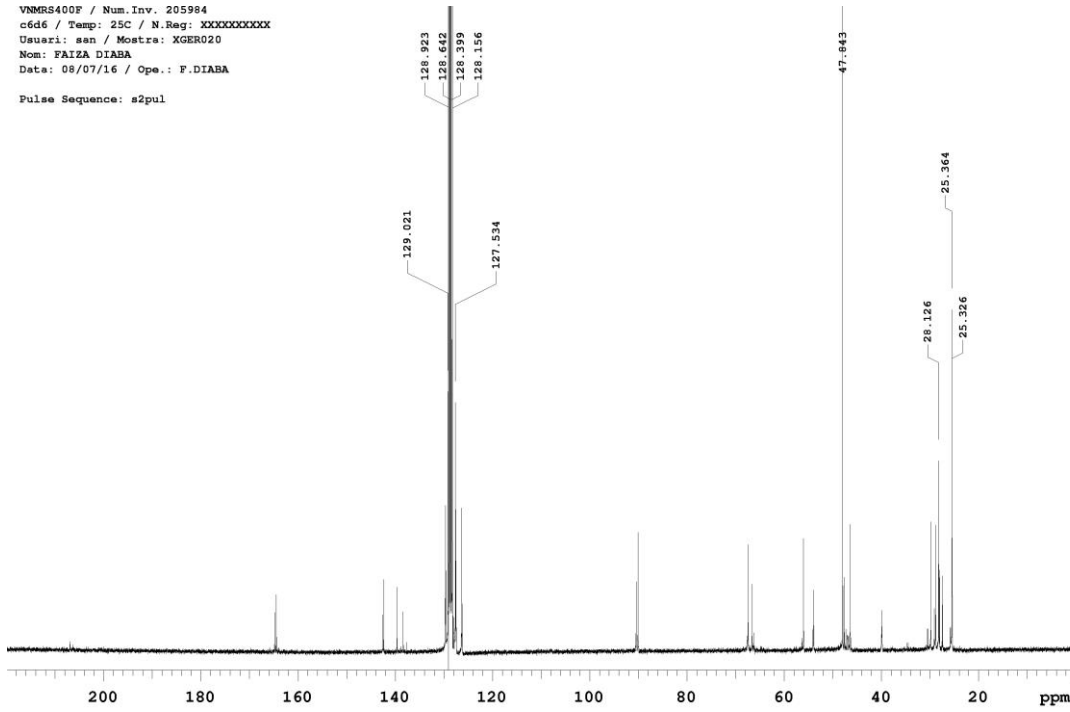




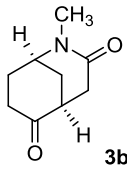
VNMRS400F / Num.Inv. 205984  
 c6d6 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: XGER020  
 Nom: FAIZA DIABA  
 Data: 08/07/16 / Ope.: F.DIABA  
 Pulse Sequence: s2pul



VNMRS400F / Num.Inv. 205984  
 c6d6 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: XGER020  
 Nom: FAIZA DIABA  
 Data: 08/07/16 / Ope.: F.DIABA  
 Pulse Sequence: s2pul

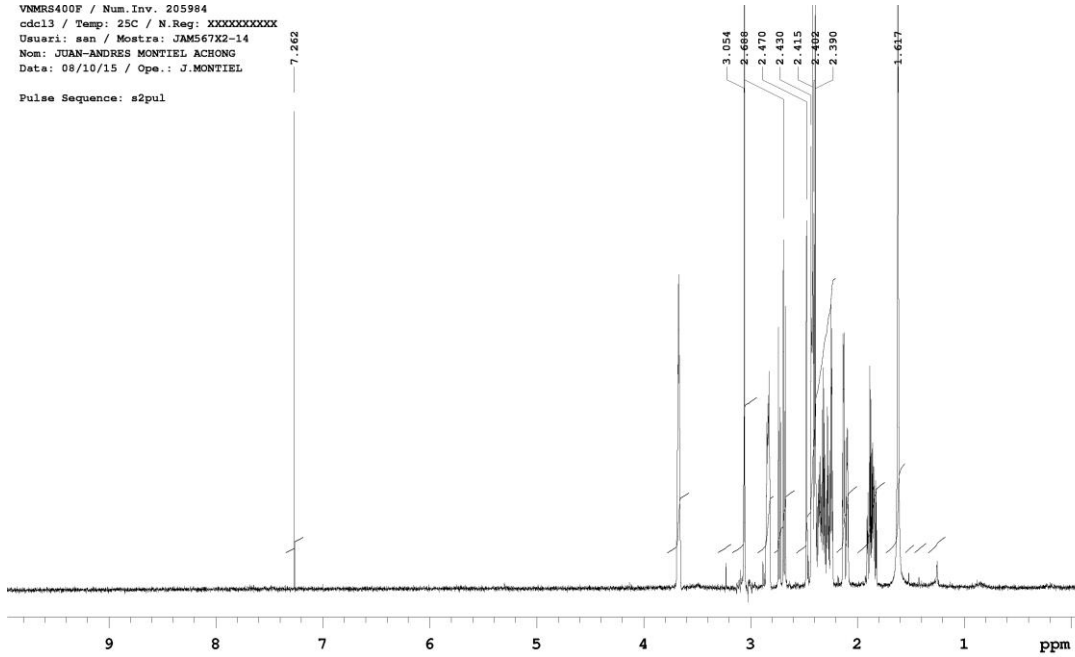






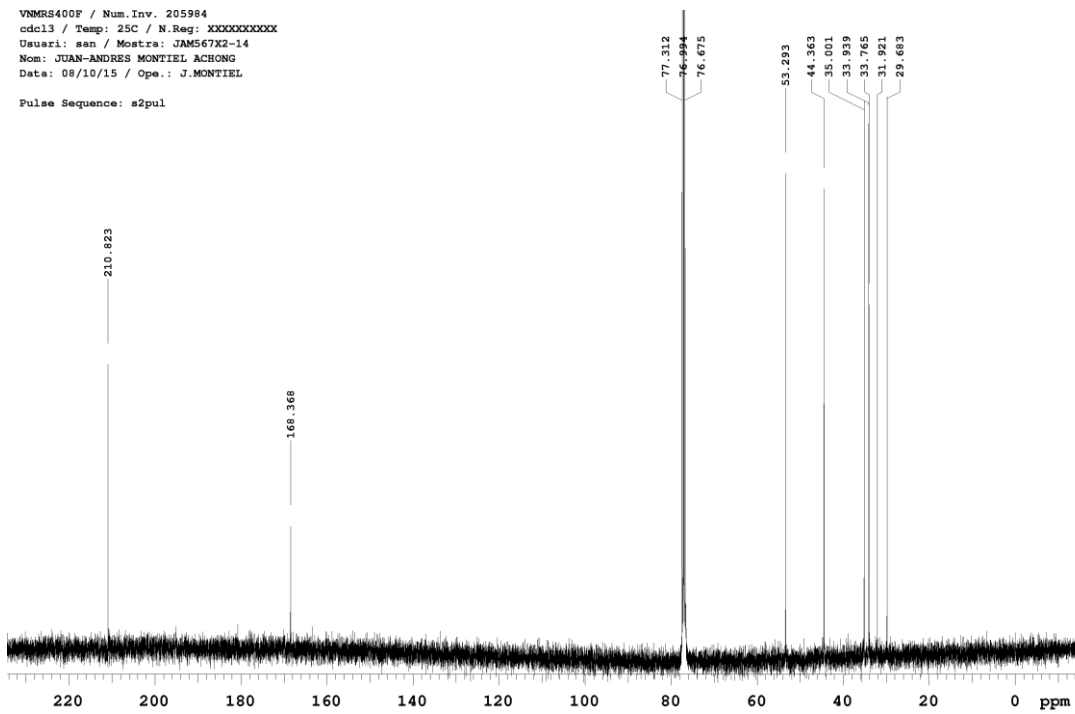
VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM567X2-14  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 08/10/15 / Ope.: J.MONTIEL

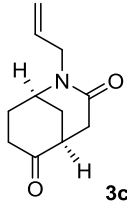
Pulse Sequence: s2pul



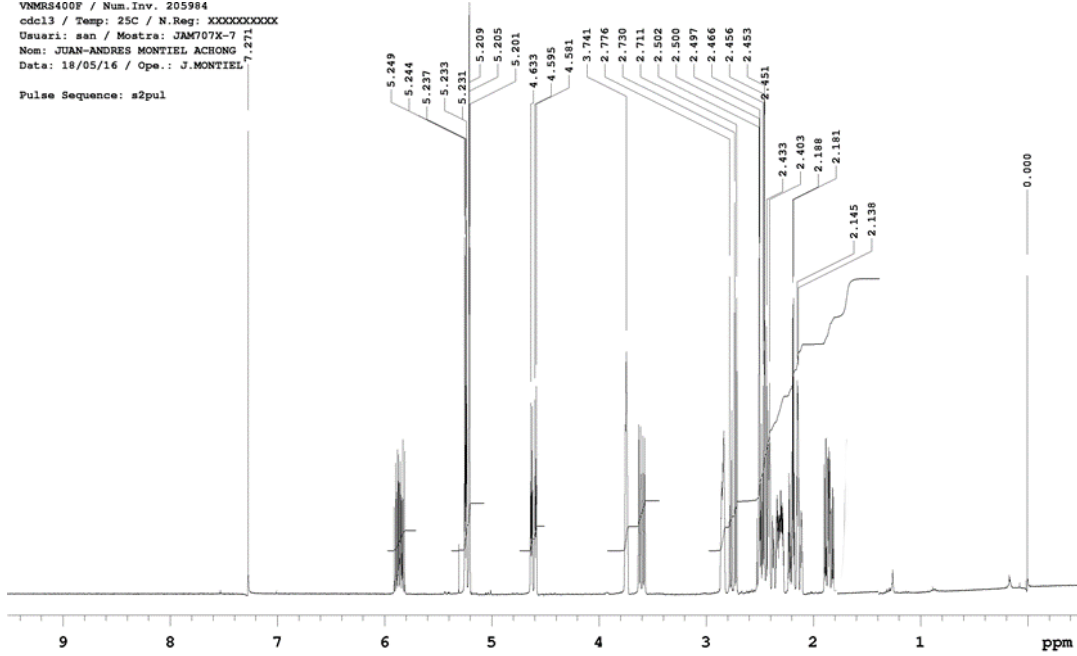
VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM567X2-14  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 08/10/15 / Ope.: J.MONTIEL

Pulse Sequence: s2pul

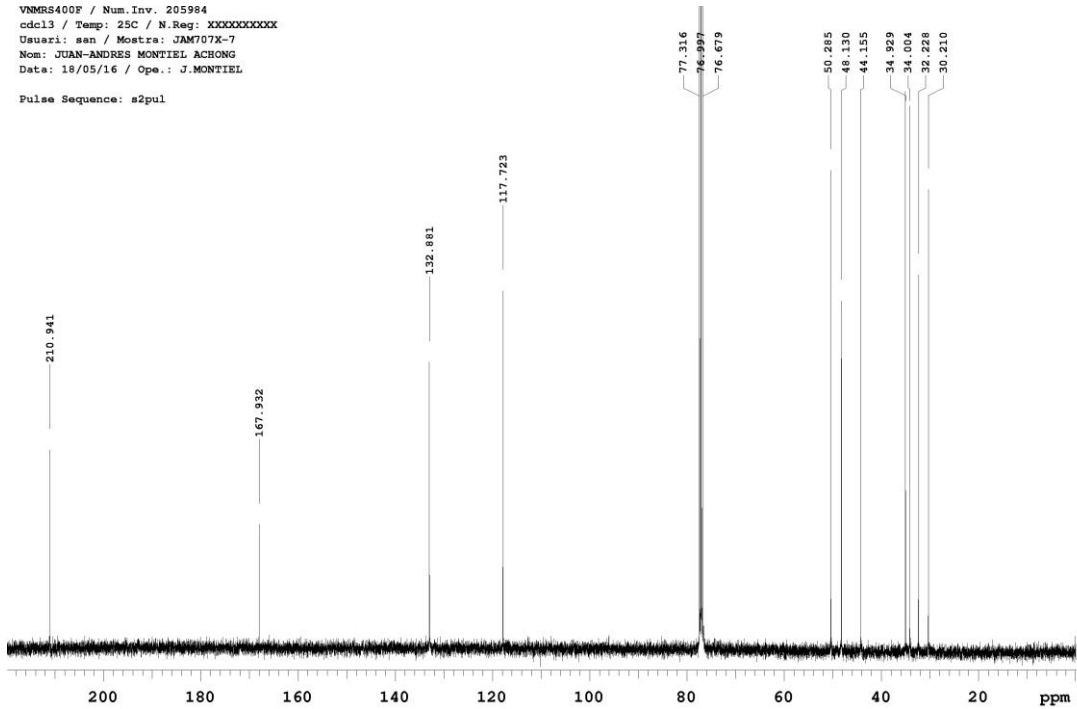


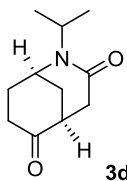


VNMRS400F / Num.Inv. 205984  
 dc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM707X-7  
 Nom: JUAN-ANDRES MONTEIL ACHONG  
 Data: 18/05/16 / Ope.: J.MONTEIL  
 Pulse Sequence: s2pul



VNMRS400F / Num.Inv. 205984  
 dc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM707X-7  
 Nom: JUAN-ANDRES MONTEIL ACHONG  
 Data: 18/05/16 / Ope.: J.MONTEIL  
 Pulse Sequence: s2pul

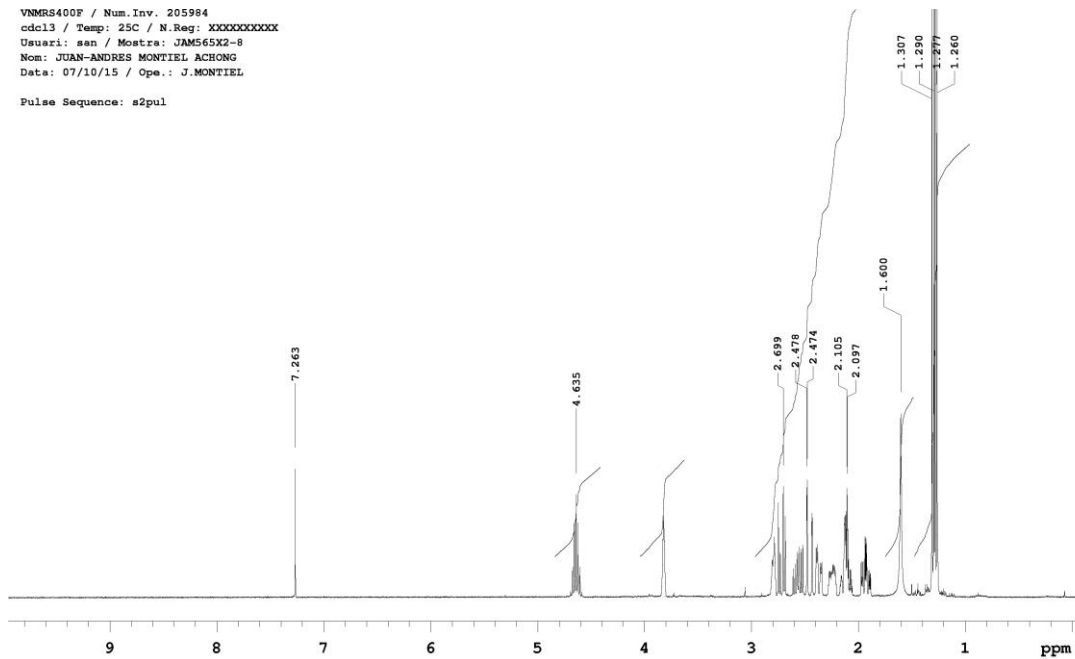




3d

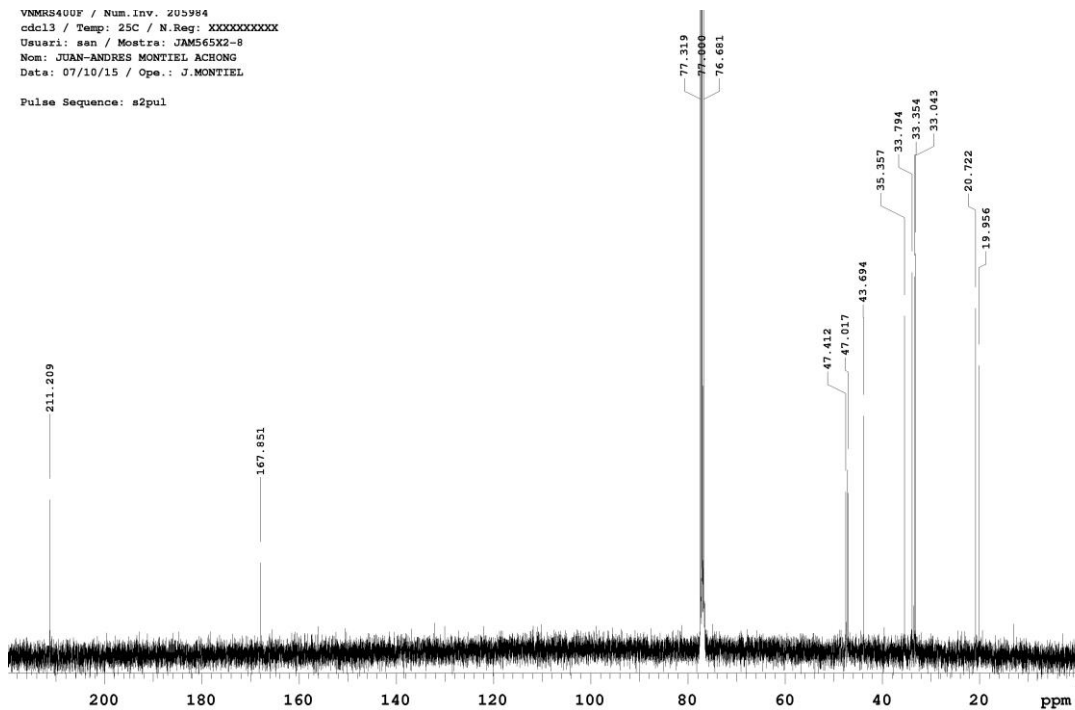
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: sen / Mostra: JAM565X2-8  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 07/10/15 / Ope.: J.MONTEIEL

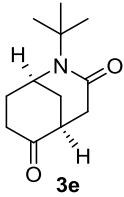
Pulse Sequence: s2pul



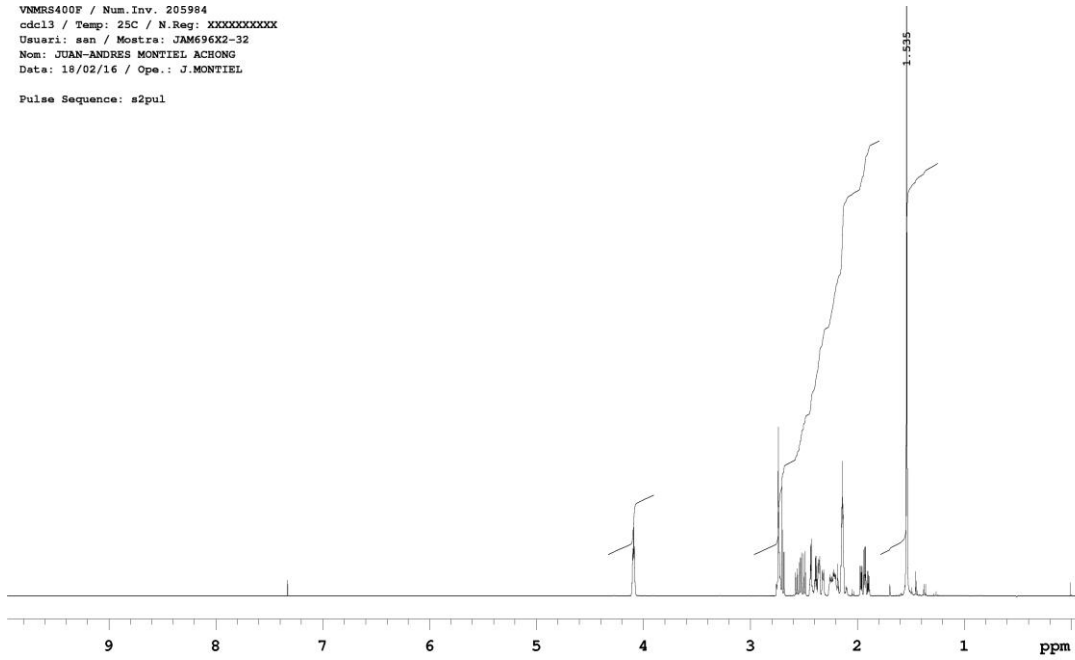
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: sen / Mostra: JAM565X2-8  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 07/10/15 / Ope.: J.MONTEIEL

Pulse Sequence: s2pul

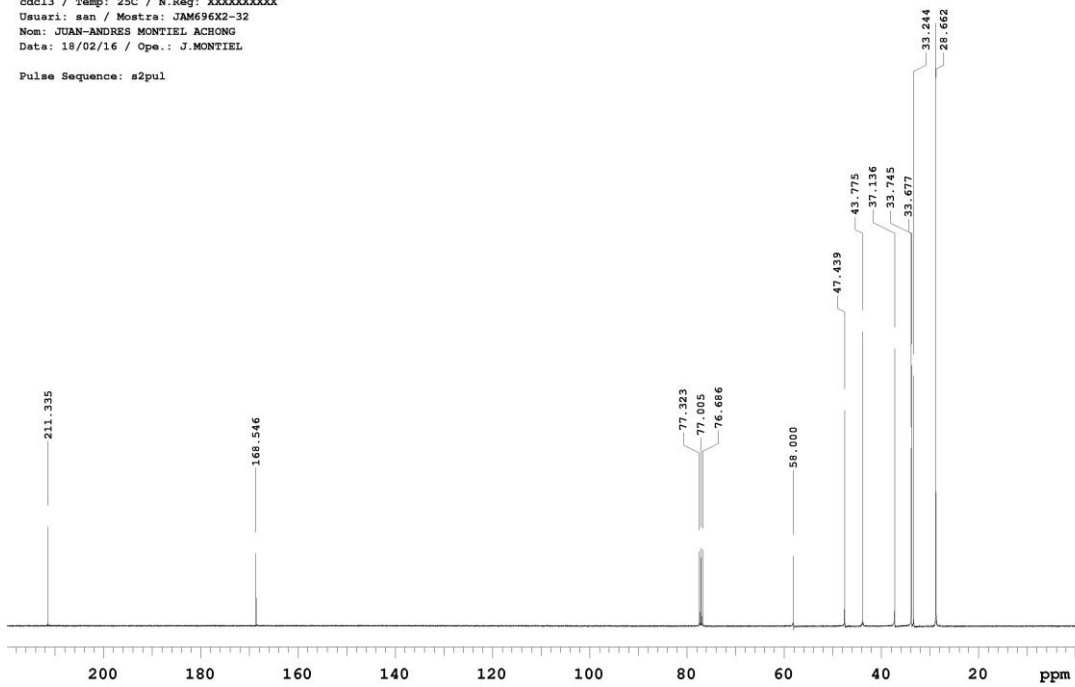


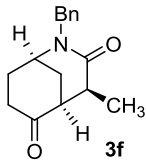


3e  
 VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM696X2-32  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 18/02/16 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul



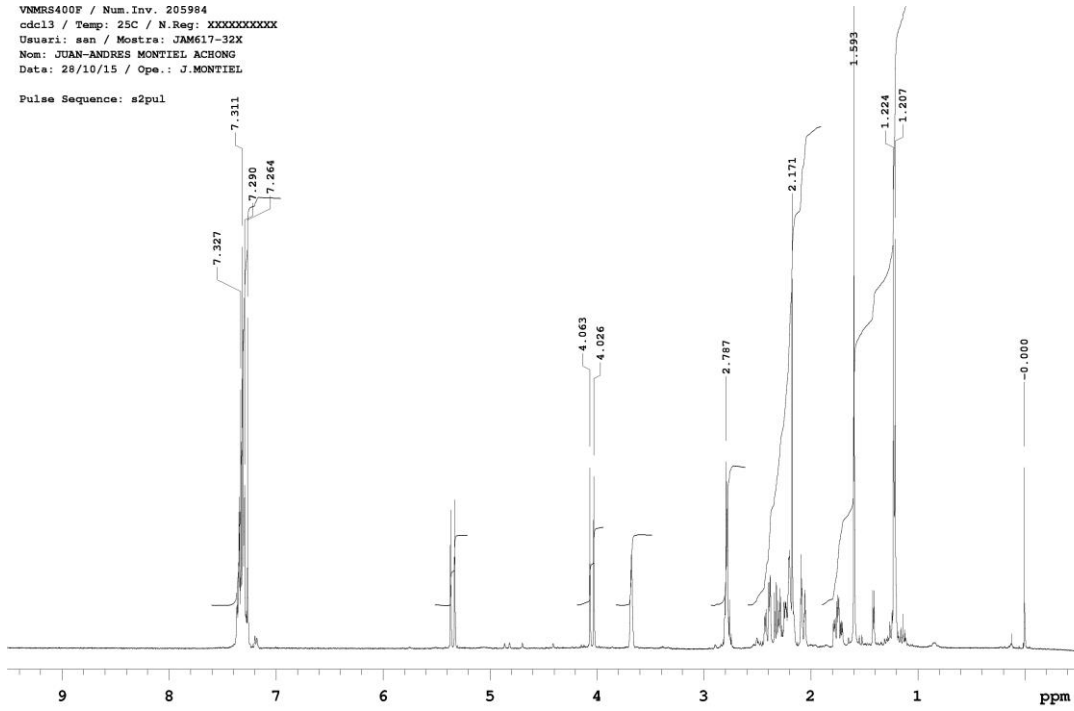
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM696X2-32  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 18/02/16 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul





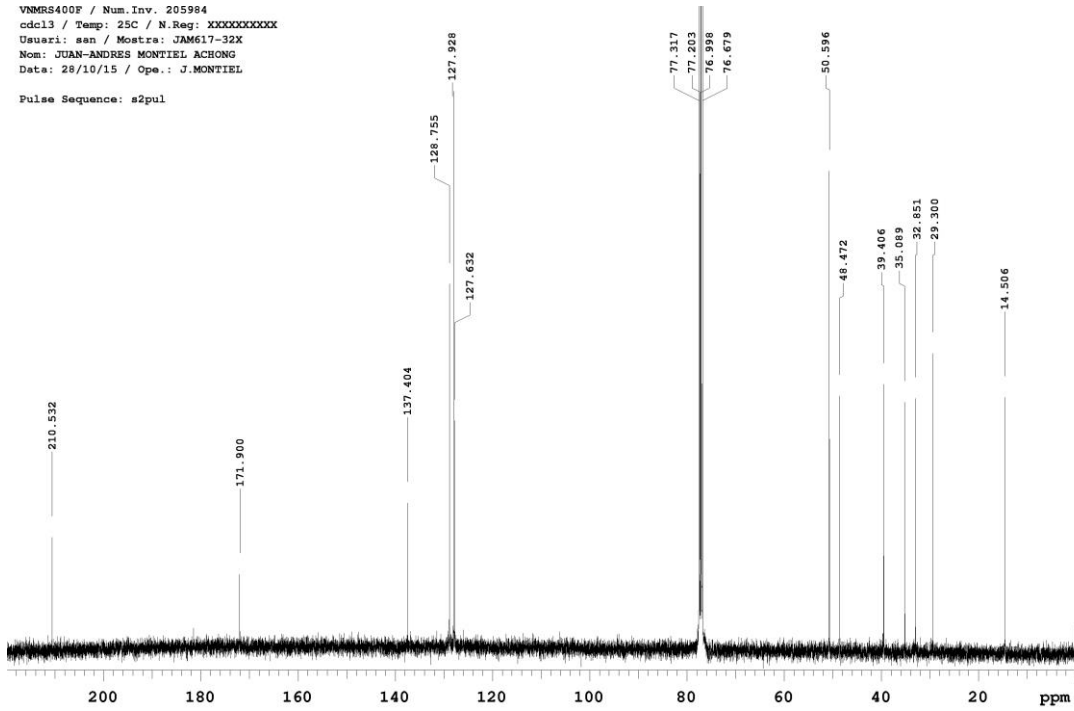
VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: JAM617-32X  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 28/10/15 / Ope.: J.MONTIEL

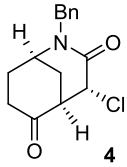
Pulse Sequence: s2pul



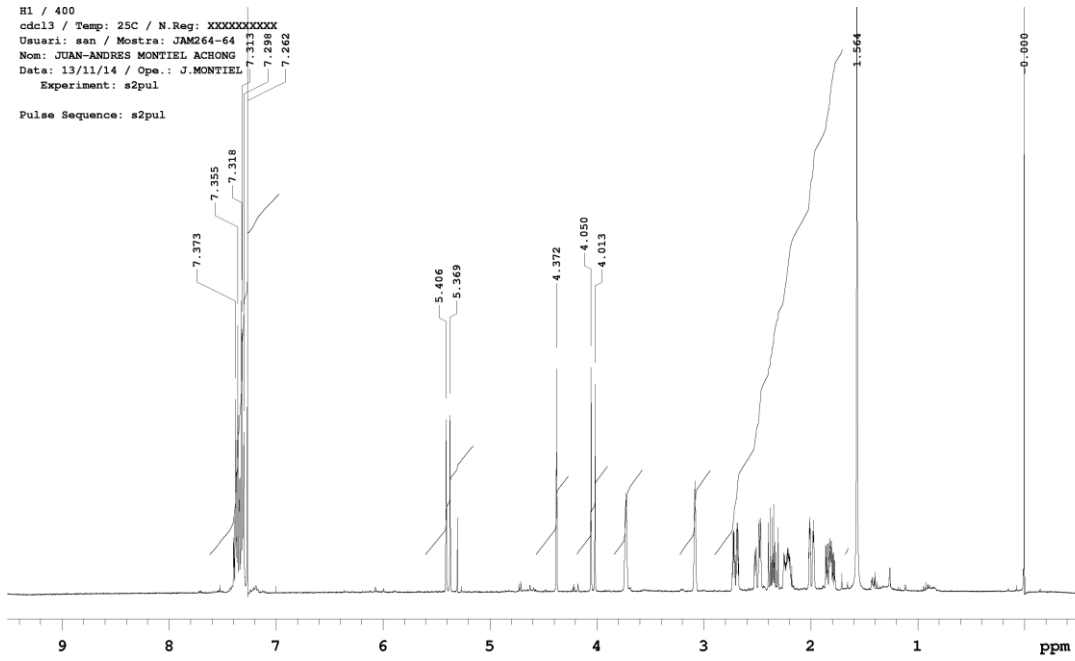
VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: sen / Mostra: JAM617-32X  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 28/10/15 / Ope.: J.MONTIEL

Pulse Sequence: s2pul

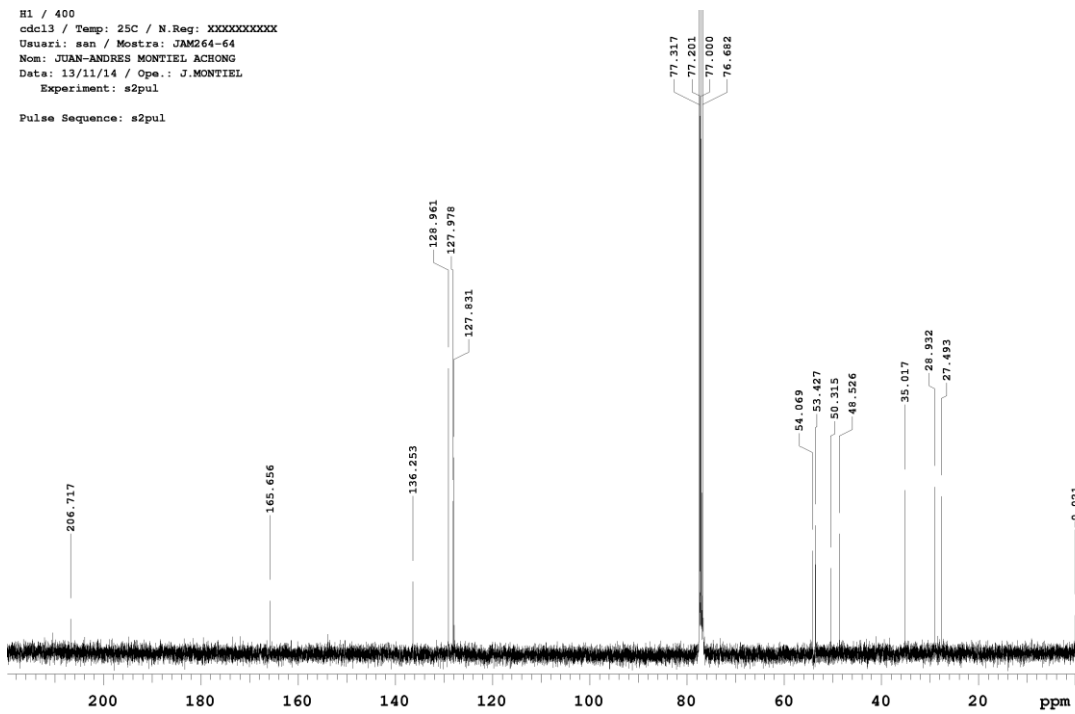


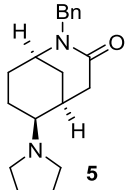


H1 / 400  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM264-64  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 13/11/14 / Ope.: J.MONTIEL  
 Experiment: s2pul  
 Pulse Sequence: s2pul

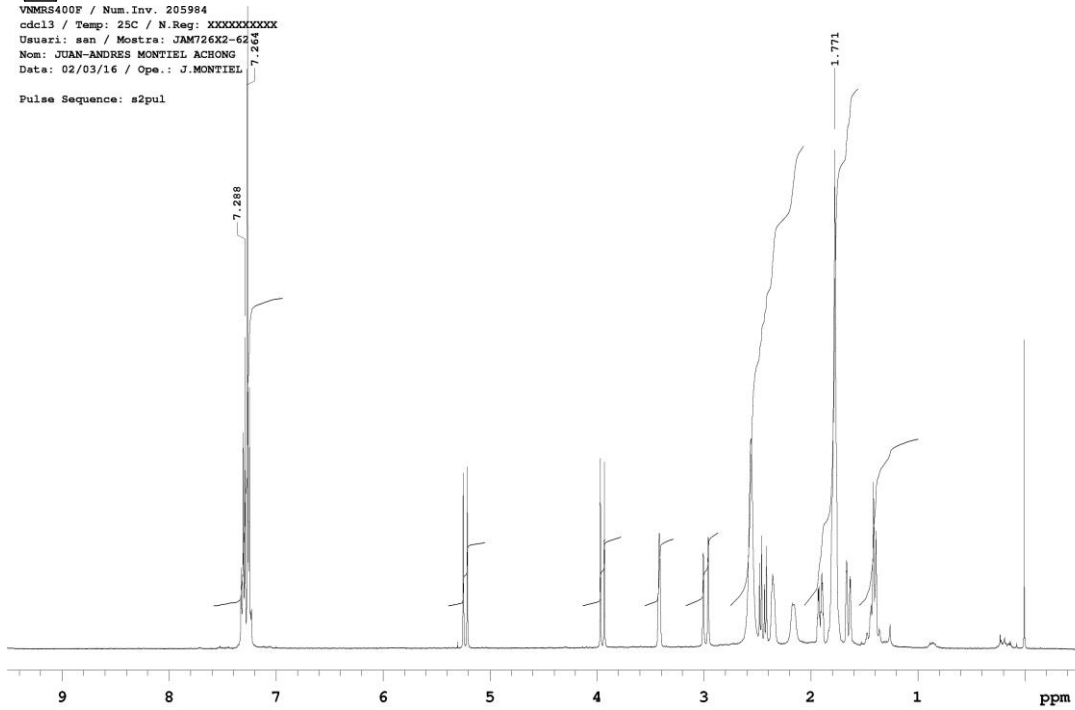


H1 / 400  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM264-64  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 13/11/14 / Ope.: J.MONTIEL  
 Experiment: s2pul  
 Pulse Sequence: s2pul

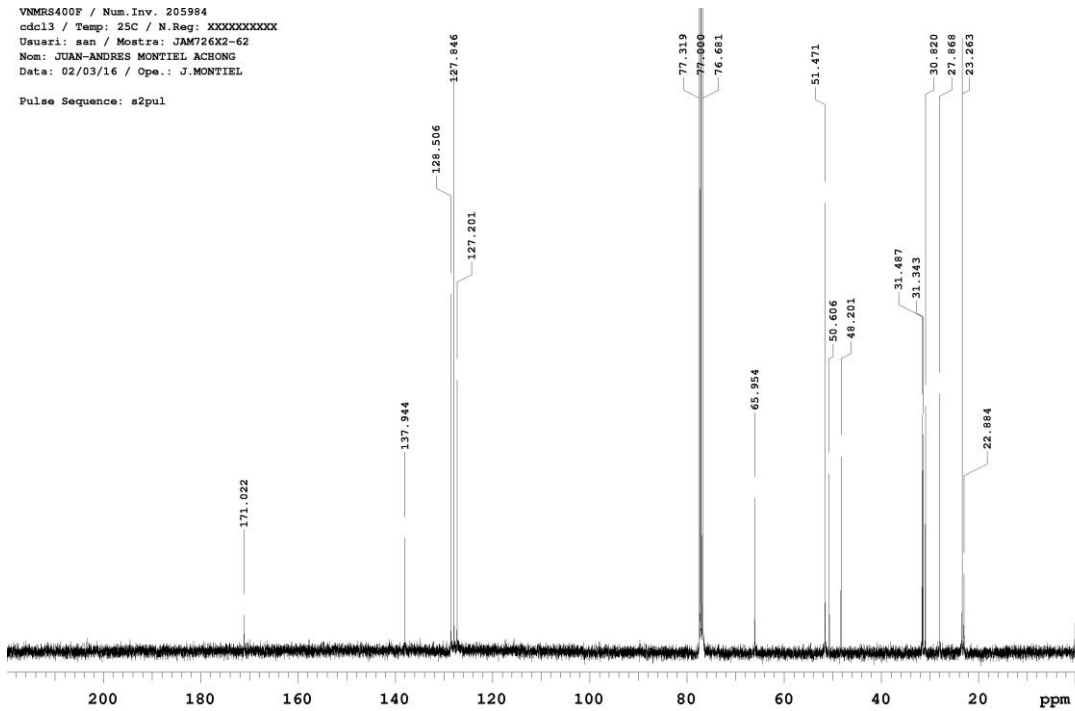


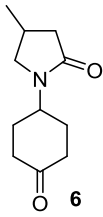


VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM726X2-62  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 02/03/16 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul

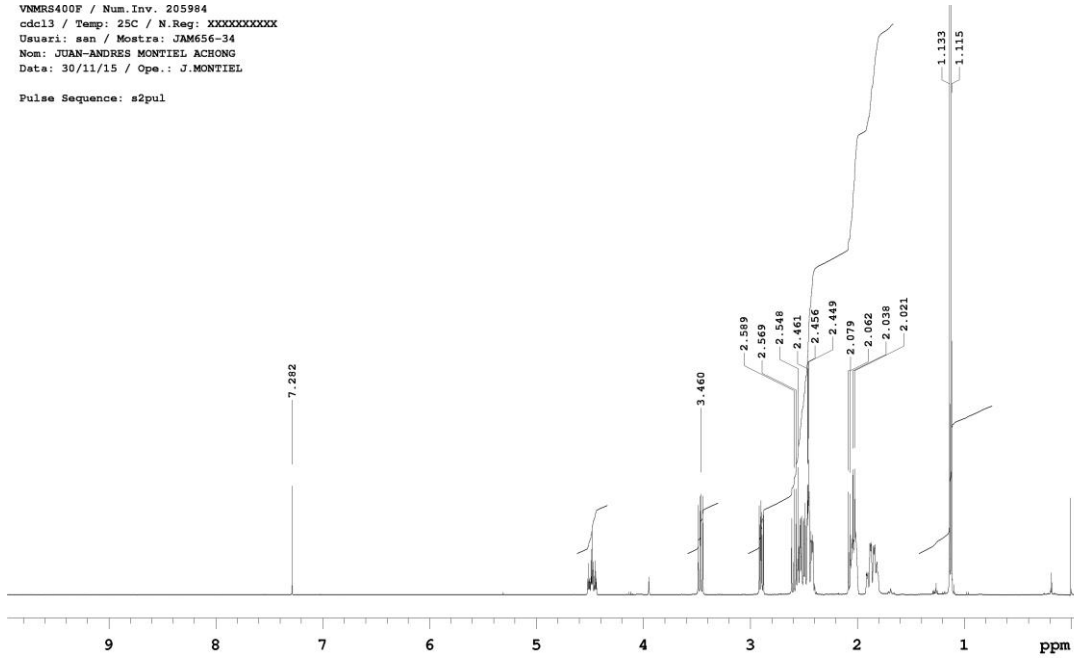


VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuario: san / Mostra: JAM726X2-62  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 02/03/16 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul

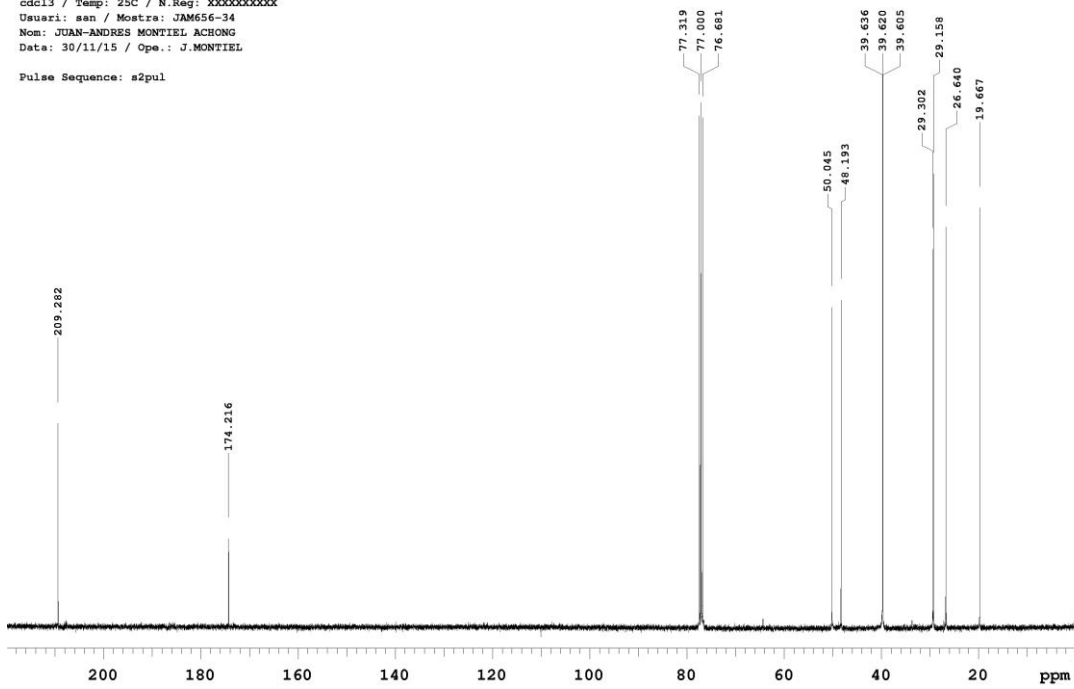




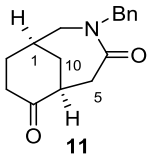
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM656-34  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 30/11/15 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul



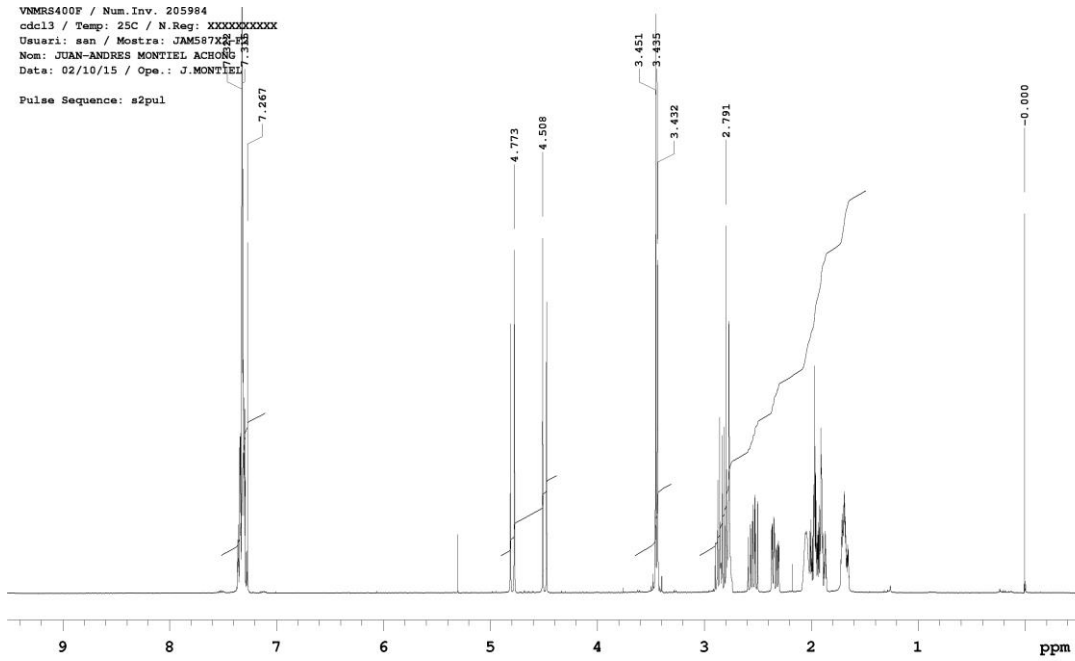
VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM656-34  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 30/11/15 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul



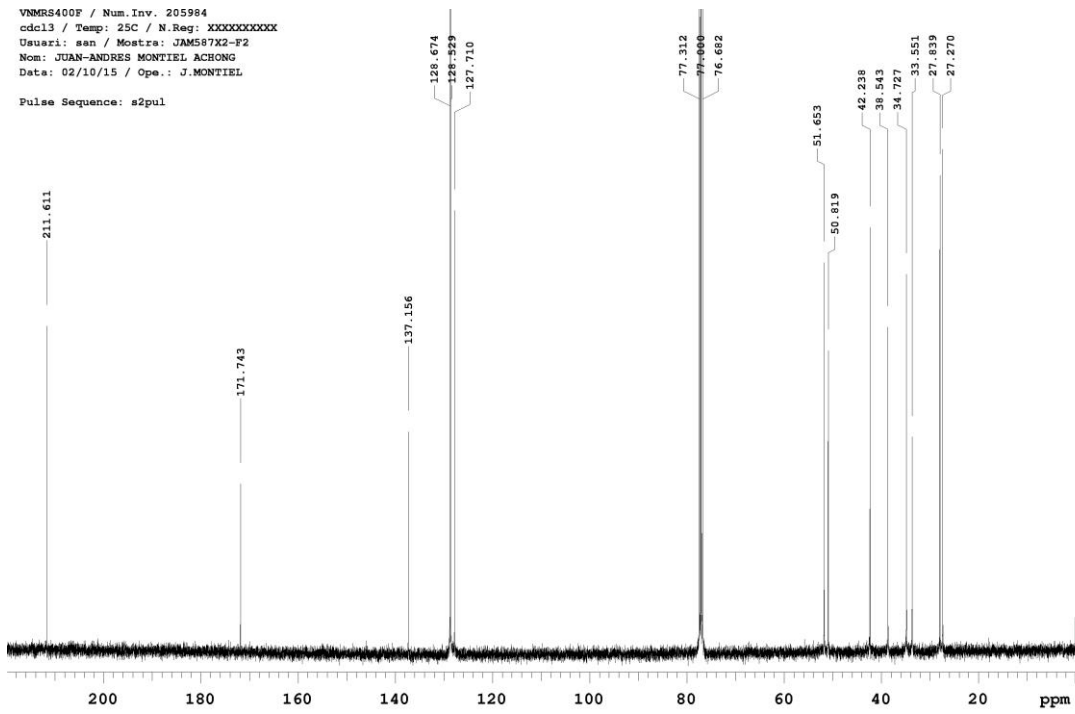


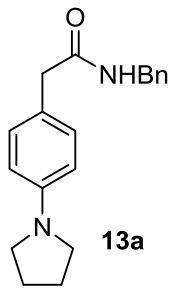


VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM587X2-F2  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 02/10/15 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul



VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM587X2-F2  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 02/10/15 / Ope.: J.MONTEIEL  
 Pulse Sequence: s2pul





VNMRS400F / Num.Inv. 205984

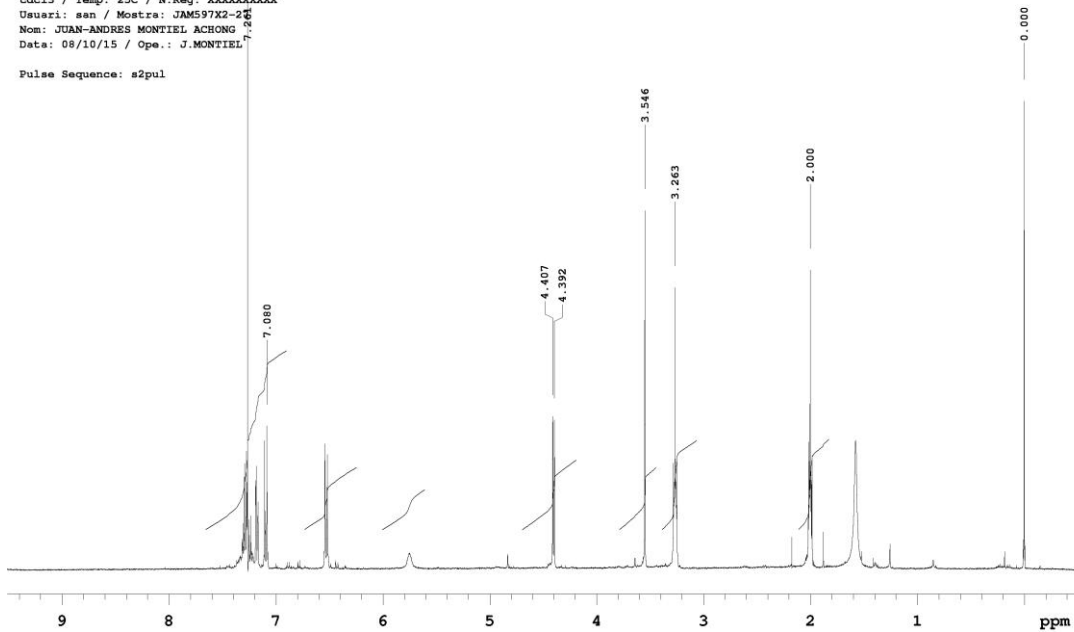
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Usuari: san / Mostra: JAM597X2-27

Nom: JUAN-ANDRES MONTEIEL ACHONG

Data: 08/10/15 / Ope.: J.MONTEIEL

Pulse Sequence: s2pul



VNMRS400F / Num.Inv. 205984

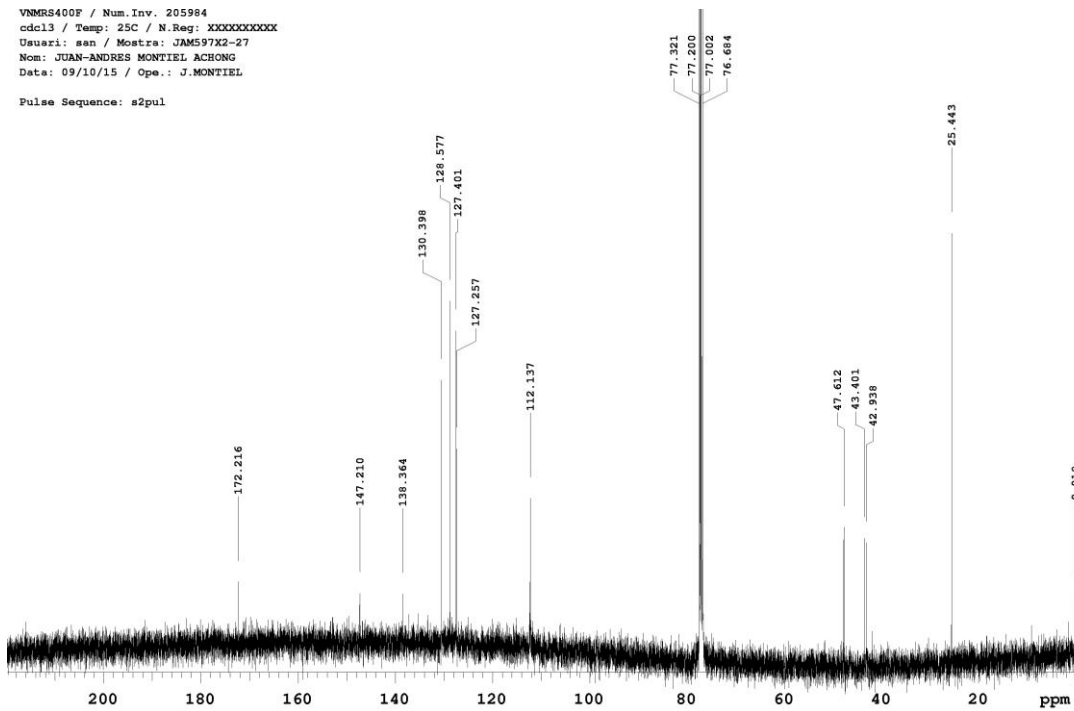
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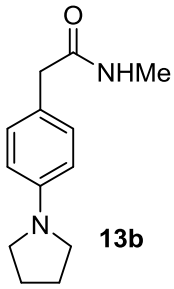
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Nom: JUAN-ANDRES MONTEIEL ACHONG

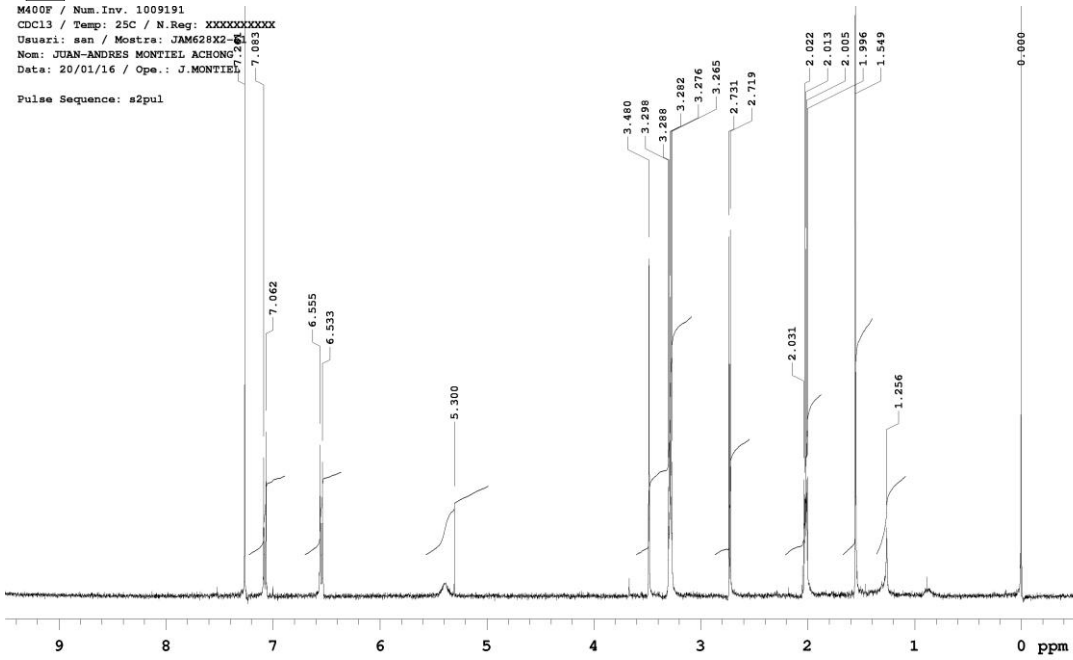
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Pulse Sequence: s2pul

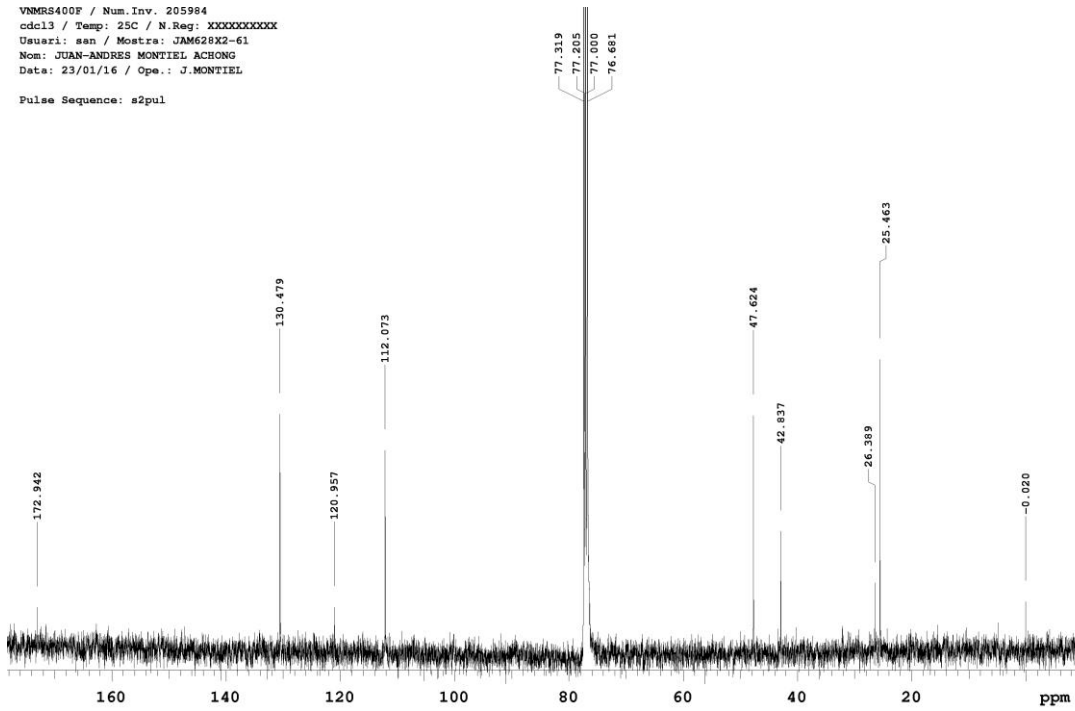


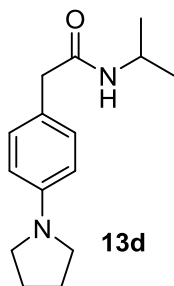


M400F / Num.Inv. 1009191  
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 Usuario: san / Mostra: JAM628X2-61  
 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 20/01/16 / Ope.: J.MONTEIEL

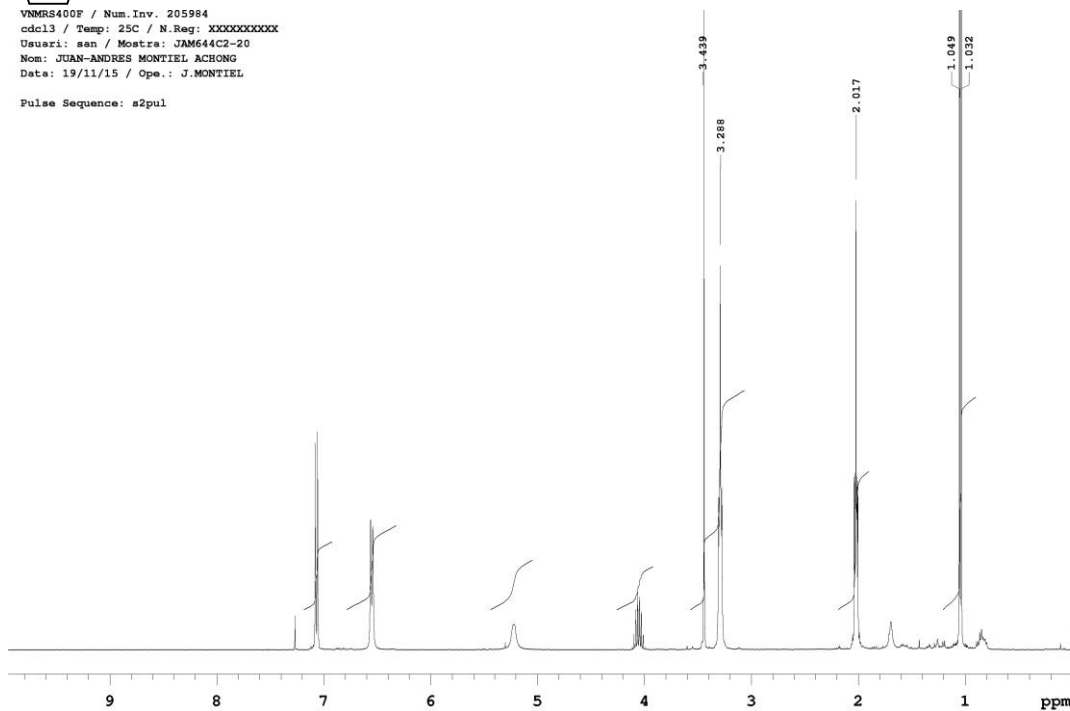


VNMRS400F / Num.Inv. 205984  
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 Nom: JUAN-ANDRES MONTEIEL ACHONG  
 Data: 23/01/16 / Ope.: J.MONTEIEL





VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM644C2-20  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 19/11/15 / Ope.: J.MONTIEL  
 Pulse Sequence: s2pul



VNMRS400F / Num.Inv. 205984  
 cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX  
 Usuari: san / Mostra: JAM644C2-20  
 Nom: JUAN-ANDRES MONTIEL ACHONG  
 Data: 19/11/15 / Ope.: J.MONTIEL  
 Pulse Sequence: s2pul

