Cerium(III)-Catalyzed Cascade Cyclization: An Efficient Approach to Functionalized pyrrolo[1,2-a]quinolines

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Supporting Information

1. General Information

Commercial reagents were used as received, unless otherwise stated. ^{1}H and ^{13}C NMR spectra were recorded on Broker Avance 400, and tetramethylsilane (TMS) was used as a reference. Data for ^{1}H are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). Data for ^{13}C NMR are reported as ppm. High resolution electrospray ionization (ESI) mass spectra (MS, m/z) were recorded on a Thermo Scientific LTQ Orbitrap XL instrument.

2. General Procedure for the Synthesis of pyrrolo[1,2-*a*]quinoline derivatives **3**.

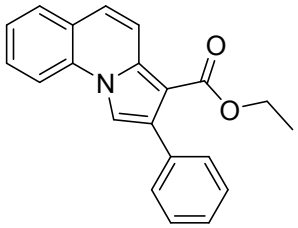
2.1 A tube was charged with **1** (0.25 mmol), **2** (0.20 mmol), $CeCl_3 \cdot 7H_2O$ (0.02 mmol). Then 2 mL of ethanol was added to the reaction system. The reaction mixture was stirred at room temperature. The reaction was monitored by TLC until the **2** was completely consumed (about 24h). The solvent was removed under reduced pressure. The residue was purified through column chromatography using silica gel to give **3**.

2.2 Typical Procedure for the Synthesis of pyrrolo[1,2-*a*]quinoline derivatives **(3r. 3s .3t)** under Ultrasound.

2-Alkylazaarenes **1** (0.25 mmol), nitroolefins **2** (0.2 mmol), and $CeCl_3 \cdot 7H_2O$ (0.02 mmol) were placed in a 10 mL glass flask with 2mL of ethanol. After the mixture was sonicated at 40°C in running water bath for 3.0 h (The ultrasonic power is 100 W), the solvent was removed under reduced pressure; the mixture was then purified by column chromatography over silica gel to afford target molecules.

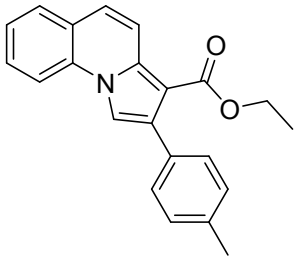
3. Characterization Data for the Products

ethyl 2-phenylpyrrolo[1,2-a]quinoline-3-carboxylate (3a).



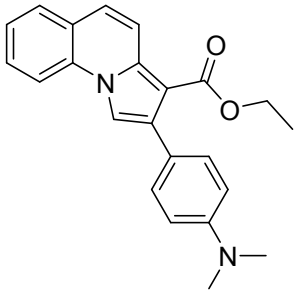
White solid (88% yield). m.p. 129-130 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ8.22 (d, *J*=9.52 Hz, 1H), 7.91 (m, 1H), 7.79 (m, 1H), 7.75 (d, *J*=7.88 Hz, 1H), 7.58 (m, 3H), 7.42 (t, *J*=7.40 Hz, 3H), 7.36 (m, 2H), 4.29 (q, *J*=7.10 Hz, 2H), 1.25 (t, *J*=7.12 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.2, 59.6, 105.2, 112.4, 114.5, 118.9, 123.6, 123.9, 124.6, 127.0, 127.6, 128.7, 128.9, 129.9, 131.3, 132.3, 134.9, 135.1, 165.1; **HRMS** calcd for $C_{21}H_{18}NO_2$ ([M+ H]$^+$）: 316.1336 found: 316.1338.

ethyl 2-p-tolylpyrrolo[1,2-a]quinoline-3-carboxylate (3b).



White solid (86% yield). m.p. 146-147 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ8.21 (d, *J*=9.52 Hz, 1H), 7.93 (d, *J*=8.40 Hz, 1H), 7.78(s, 1H), 7.76 (dd, J_1=7.88 Hz, J_2=1.08 Hz, 1H), 7.62-7.58 (m, 1H), 7.46-7.41 (m, 3H), 7.37 (d, *J*=9.52 Hz, 1H), 7.22 (d, *J*=7.84 Hz, 2H), 4.29 (d, *J*=7.12 Hz, 2H), 2.41 (s, 3H), 1.27 (t, *J*=7.14 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.3, 21.2, 59.5, 105.2, 112.2, 114.5, 119.0, 123.4, 123.9, 124.5, 128.3, 128.7, 128.9, 129.7, 131.4, 132.1, 132.3, 134.8, 136.6, 165.0; **HRMS** calcd for $C_{22}H_{20}NO_2$ ([M+ H]$^+$）: 330.1494 found: 330.1495.

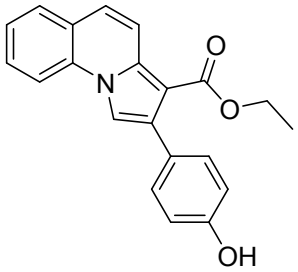
ethyl 2-(4-(dimethylamino)phenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3c).



White solid (82% yield). m.p. 183-184 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ8.21 (d, *J*=9.48 Hz, 1H), 7.93 (d, *J*=8.36 Hz, 1H), 7.77 (s, 1H), 7.75 (d, *J*=8.04 Hz, 1H), 7.59 (t, *J*=7.76 Hz, 1H), 7.48 (d, *J* = 8.36 Hz, 2H), 7.41 (t, *J* = 7.48 Hz, 1H), 7.35 (d, *J*=9.48 Hz, 1H), 6.82 (s, 2H), 4.31 (q, *J*=7.02 Hz, 2H), 3.00 (s, 6H), 1.31 (t, *J*=7.04 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.4, 40.8, 59.5,

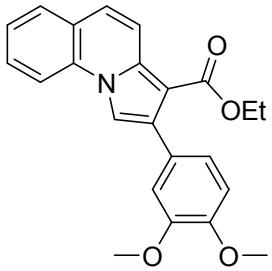
105.1, 111.9, 112.1, 114.5, 119.0, 123.1, 123.9, 124.4, 128.6, 128.8, 130.6, 131.6, 132.3, 134.8, 165.2; **HRMS** calcd for $C_{23}H_{23}N_2O_2$ ([M+ H]$^+$) : 359.1760 found: 359.1761.

ethyl 2-(4-hydroxyphenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3d).



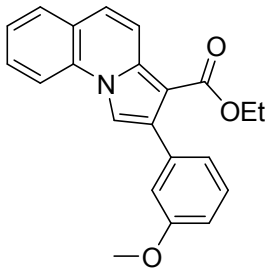
White solid (90% yield). m.p. 151-153 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.18 (d, *J*=9.52 Hz, 1H), 7.92 (d, *J*=8.40 Hz, 1H), 7.75-7.73 (m, 2H), 7.60-7.56 (m, 1H), 7.43-7.42 (m, 3H), 7.35 (d, *J*=9.52 Hz, 1H), 6.85 (d, *J*=8.56 Hz, 2H), 5.81 (s, 1H), 4.33 (q, *J*=7.12 Hz, 2H), 1.32 (t, *J*=7.12 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.3, 59.8, 105.0, 112.3, 114.5, 114.8, 118.9, 123.6, 123.9, 124.5, 127.1, 128.7, 128.8, 131.0, 131.3, 132.3, 134.8, 155.2, 165.5; **HRMS** calcd for $C_{21}H_{18}NO_3$ ([M+ H]$^+$) : 332.1287 found: 332.1290.

ethyl 2-(3,4-dimethoxyphenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3e).



White solid (98% yield). m.p. 192-193 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.20 (d, *J*=9.48 Hz, 1H), 7.92 (d, *J*=8.40 Hz, 1H), 7.78 (s, 1H), 7.74 (dd, *J*=7.86 Hz, *J*=1.06 Hz, 1H), 7.58-7.56 (m, 1H), 7.43-7.39 (m, 1H), 7.36 (d, *J*=9.52 Hz, 1H), 7.11-7.08 (m, 2H), 6.92(d, *J*=8.32 Hz, 1H), 4.29 (q, *J*=7.12 Hz, 2H), 3.93 (s, 3H), 3.92 (s, 3H), 1.26 (t, *J*=7.12 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.3, 55.9, 55.9, 59.5, 105.2, 110.6, 112.2, 113.6, 114.5, 118.9, 122.1, 123.5, 123.9, 124.6, 127.8, 128.7, 128.9, 131.1, 132.3, 134.8, 148.1, 148.3, 165.0; **HRMS** calcd for $C_{23}H_{22}NO_4$ ([M+ H]$^+$) : 376.1543 found: 376.1541.

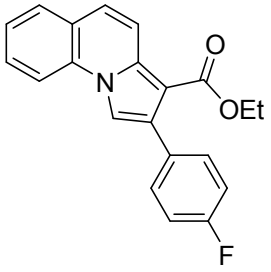
ethyl 2-(3-methoxyphenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3f).



White solid (92% yield). m.p. 132-133 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.22 (d, *J*=9.48 Hz, 1H), 7.93 (d, *J*=8.40 Hz, 1H), 7.81 (s, 1H), 7.75 (d, *J*=7.84 Hz, 1H), 7.62-7.58 (m, 1H), 7.44-7.41 (m,

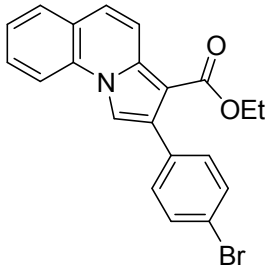
1H), 7.39-7.36 (m, 1H), 7.34-7.30 (m, 1H), 7.15-7.11 (m, 2H), 6.93-6.90 (m, 1H), 4.29 (q, *J*=7.11 Hz, 2H), 3.86 (s, 3H), 1.25 (t, *J*=7.11 Hz, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δPeak 14.2, 55.2, 59.5, 105.3, 112.4, 112.5, 114.5, 115.6, 118.9, 122.5, 123.6, 123.9, 124.6, 128.5, 128.7, 128.9, 131.1, 132.3, 134.9, 136.5, 158.9, 165.0; **HRMS** calcd for $C_{22}H_{20}NO_3$ ([M+ H]$^+$) : 346.1437 found: 346.1435.

ethyl 2-(4-fluorophenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3g).



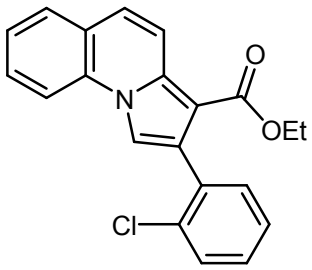
White solid (93% yield). m.p. 161-162 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.21 (d, *J*=9.52 Hz, 1H), 7.92 (d, *J*=8.36 Hz, 1H), 7.76-7.74 (m, 2H), 7.60 (t, *J*=7.78Hz, 1H), 7.52-7.49 (m, 2H), 7.45-7.36 (m, 2H), 7.12-7.08 (m, 2H), 4.27 (q, *J*=7.13 Hz, 2H), 1.23 (d, *J*=7.13 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δPeak 14.2, 59.6, 105.2, 112.3, 114.3, 114.5, 114.5, 118.9, 123.7, 123.9, 124.7, 128.8, 128.9, 130.3, 131.1, 131.1, 131.4, 131.5, 132.3, 134.9, 161.0, 163.4, 164.9; **HRMS** calcd for $C_{21}H_{17}O_2NF$ ([M+ H]$^+$) : 334.1237 found: 334.1239.

ethyl 2-(4-bromophenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3h).



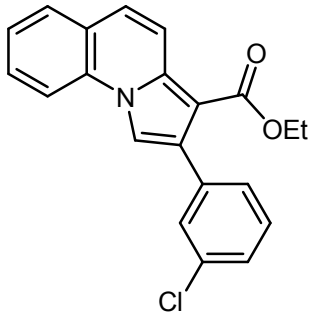
White solid (96% yield). m.p. 162-163 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.19 (d, *J*=9.52 Hz, 1H), 7.91 (d, *J*=8.40 Hz, 1H), 7.743-7.75 (m, 2H), 7.60-7.56 (m, 1H), 7.53-7.50 (m, 2H), 7.44-7.35 (m, 4H), 4.28 (q, *J*=7.12 Hz, 2H), 1.26 (t, *J*=7.12 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δPeak 14.2, 59.6, 76.7, 77.0, 77.2, 77.3, 105.0, 112.3, 114.5, 118.8, 121.2, 123.8, 123.9, 124.7, 128.8, 128.9 130.1, 130.7, 131.5, 132.2, 134.1, 135.0, 164.8; **HRMS** calcd for $C_{21}H_{16}O_2NBrNa$ ([M+ Na]$^+$) : 416.0256 found: 416.0254.

ethyl 2-(2-chlorophenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3i).



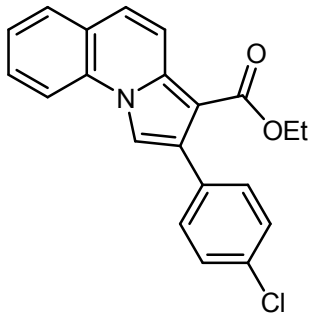
White solid (83% yield). m.p. 181-182 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.24 (d, *J*=9.48 Hz, 1H), 7.91 (d, *J*=8.36 Hz, 1H), 7.77 (s, 1H), 7.76 (d, *J*=9.12 Hz, 1H), 7.61-7.57 (m, 1H), 7.48-7.38 (m, 4H), 7.33-7.29 (m, 2H), 4.19 (q, *J*=7.12 Hz, 2H), 1.10 (d, *J*=7.12Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.9, 59.4, 106.6, 112.4, 114.5, 118.8, 123.7, 124.0, 124.6, 125.9, 127.8, 128.5, 128.7, 128.9, 131.7, 132.4, 134.3, 134.6, 134.9, 164.7; $C_{21}H_{16}O_2NClNa$ ([M+ Na]$^+$) : 372.0761 found: 372.0759.

ethyl 2-(3-chlorophenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3j).



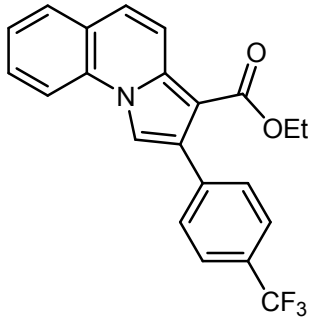
White solid (85% yield). m.p. 160-161 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.23 (d, *J*=9.52 Hz, 1H), 7.92 (d, *J*=8.40 Hz, 1H), 7.78 (s, 1H), 7.74 (dd, J_1=7.86 Hz, J_2=1.10 Hz, 1H), 7.61-7.55 (m, 2H), 7.45-7.41 (m, 2H), 7.38 (d, *J*=9.52 Hz, 1H), 7.33-7.32 (m, 2H), 4.28 (q, *J*=7.13 Hz, 2H), 1.25 (t, *J*=7.13 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.1, 59.6, 105.0, 112.4, 114.5, 118.8, 123.9, 123.9, 124.8, 127.0, 128.1, 128.7, 128.8, 128.9, 129.7, 130.0, 132.2, 133.3, 135.1, 137.0, 164.8; $C_{21}H_{16}O_2NClNa$ ([M+ Na]$^+$) : 372.0761 found: 372.0758.

ethyl 2-(4-chlorophenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3k).



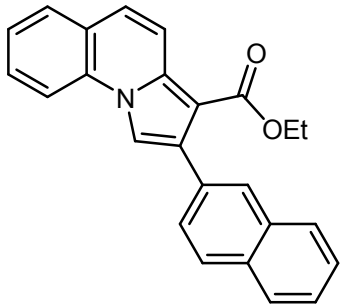
White solid (91% yield). m.p. 171-172 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.20 (d, *J*=9.48 Hz, 1H), 7.91 (d, *J*=8.40 Hz, 1H), 7.76-7.73 (m, 2H), 7.61-7.57 (m, 1H), 7.48-7.35 (m, 6H), 4.28 (q, *J*=7.13 Hz, 2H), 1.26 (t, *J*=7.13 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.2, 59.6, 105.1, 112.3, 114.5, 118.9, 123.8, 123.9, 124.7, 127.7, 128.8, 128.9, 130.1, 131.2, 132.2, 133.0, 133.6, 134.9, 164.8; $C_{21}H_{16}O_2NClNa$ ([M+ Na]$^+$) : 372.0761 found: 372.0757.

ethyl 2-(4-(trifluoromethyl)phenyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3l).



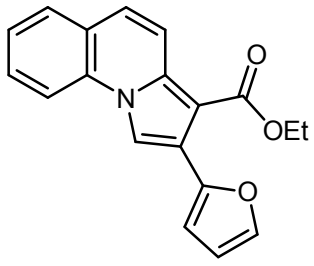
White solid (80% yield). m.p. 152-153 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.22 (d, *J*=9.52 Hz, 1H), 7.94 (d, *J*=8.40 Hz, 1H), 7.81 (s, 1H), 7.78-7.76 (m, 1H), 7.66 (s, 4H), 7.61-7.59 (m, 1H), 7.47-7.45 (m, 1H), 7.41-7.39 (m, 1H), 4.29 (q, *J*=7.12 Hz, 2H), 1.24 (t, *J*=7.12 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.2, 59.7, 105.1, 112.5, 114.5, 118.8, 124.0, 124.1, 124.4, 124.4, 124.5, 124.5, 124.9, 128.9, 128.9, 129.0, 129.9, 130.2, 132.3, 135.0, 139.0, 164.7; $C_{22}H_{16}O_2NF_3Na$ ([M+ Na]$^{+}$) : 406.1025 found: 406.1020.

ethyl 2-(naphthalen-2-yl)pyrrolo[1,2-a]quinoline-3-carboxylate (3m).



White solid (89% yield). m.p. 146-147 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.26 (d, *J*=9.48 Hz, 1H), 8.00 (s, 1H), 7.97-7.94 (m, 1H), 7.87 (m, 4H), 7.88-7.86 (m, 1H), 7.71 (d, *J*=8.44 Hz, 1H), 7.59-7.57 (m, 1H), 7.52-7.38 (m, 4H), 4.28 (q, *J*=7.06 Hz, 2H), 1.20 (t, *J*=7.06 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.2, 59.6, 105.3, 112.6, 114.5, 118.9, 123.7, 124.0, 124.6, 125.6, 125.9, 126.7, 127.6, 127.9, 127.9, 128.7, 128.8, 128.9, 131.2, 132.3, 132.5, 132.8, 133.1, 135.0, 165.0; $C_{25}H_{19}O_2NNa$ ([M+ Na]$^{+}$) : 388.1308 found: 388.1303.

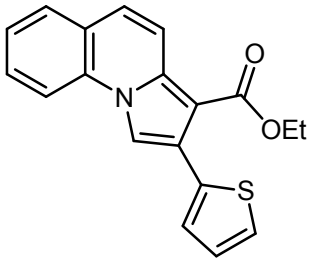
ethyl 2-(furan-2-yl)pyrrolo[1,2-a]quinoline-3-carboxylate (3n).



White solid (85% yield). m.p. 90-91 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.16 (d, *J*=9.48 Hz, 1H), 8.15 (s, 1H), 7.94 (d, *J*=8.44 Hz, 1H), 7.72-7.70 (m, 1H), 7.60-7.56 (m, 1H), 7.48-7.47 (m, 1H), 7.43-7.39 (m, 1H), 7.32 (d, *J*=9.60 Hz, 1H), 7.22-7.21 (m, 1H), 6.52-6.51 (m, 1H), 4.44 (q, *J*=7.13 Hz, 2H), 1.45 (t, *J*=7.13 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.5, 59.8, 103.5, 110.0, 111.5, 111.7, 114.6, 118.9, 120.5, 123.6, 123.9, 124.7, 128.8, 128.8, 132.1, 134.9, 141.2, 148.7, 164.7;

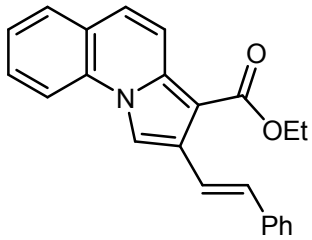
$C_{19}H_{15}O_3NNa$ ([M+ Na]$^+$) : 328.0944 found: 328.0940.

ethyl 2-(thiophen-2-yl)pyrrolo[1,2-a]quinoline-3-carboxylate (3o).



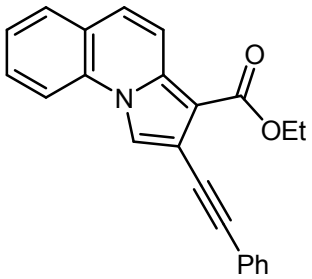
White solid (83% yield). m.p. 142-143 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.18 (d, *J*=9.56 Hz, 1H), 7.92-7.90 (m, 2H), 7.73 (d, *J*=7.80 Hz, 1H), 7.58 (t, *J*=7.26 Hz, 1H), 7.44-7.32 (m, 4H), 7.12-7.09 (m, 1H), 4.36 (q, *J*=7.13 Hz, 2H), 1.36 (t, *J*=7.13 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.3, 59.7, 105.2, 112.9, 114.6, 118.8, 123.5, 123.8, 123.9, 124.8, 125.0, 126.9, 127.5, 128.8, 128.9, 132.1, 135.0, 135.8, 164.8; $C_{19}H_{15}O_2NNaS$ ([M+ Na]$^+$) : 344.0715 found: 344.0712.

(*E*)-ethyl 2-styrylpyrrolo[1,2-a]quinoline-3-carboxylate (3p).



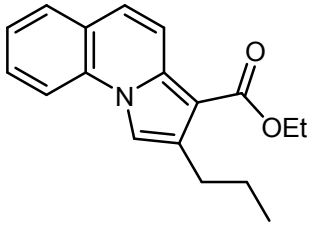
White solid (74% yield). m.p. 135-137 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.12 (d, *J*=9.48 Hz, 1H), 8.04 (s, 1H), 7.95-7.91 (m, 2H), 7.70-7.68 (m, 1H), 7.59-7.54 (m, 3H), 7.41-7.33 (m, 3H), 7.29-7.22 (m, 2H), 7.06 (d, *J*=16.5 Hz, 1H), 4.42 (q, *J*=7.12 Hz, 2H), 1.47 (t, *J*=9.56 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.6, 59.7, 105.1, 109.3, 114.7, 118.8, 121.8, 123.3, 124.0, 124.6, 126.4, 127.3, 128.0, 128.6, 128.6, 128.8, 129.2, 132.1, 134.9, 137.7, 165.3; $C_{23}H_{19}O_2NNa$ ([M+ Na]$^+$) : 364.1308 found: 364.1304.

ethyl 2-(phenylethynyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3q).



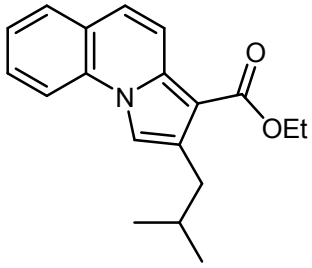
White solid (70% yield). m.p. 157-158 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.18 (dd, J_1=9.46 Hz, J_2=2.42 Hz, 1H), 8.05 (d, *J*=9.48 Hz, 1H), 7.90-7.88 (m, 1H), 7.74-7.72 (m, 1H), 7.61-7.59 (m, 3H), 7.45-7.31 (m, 5H), 4.44 (q, *J*=7.11 Hz, 2H), 1.46 (t, *J*=7.11 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.6, 59.9, 83.4, 92.5, 107.7, 110.2, 114.6, 116.9, 118.5, 123.8, 124.1, 124.2, 125.0, 128.0, 128.3, 128.9, 129.0, 131.5, 131.9, 134.2, 164.3; $C_{23}H_{17}O_2NNa$ ([M+ Na]$^+$) : 362.1151 found: 362.1147.

ethyl 2-propylpyrrolo[1,2-a]quinoline-3-carboxylate (3r).



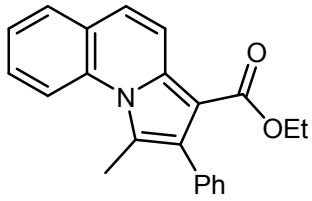
White solid (76% yield). m.p. 106-107 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.15 (d, *J*=9.48 Hz, 1H), 7.89 (d, *J*=8.44 Hz, 1H), 7.71 (d, *J*=7.88 Hz, 1H), 7.60-7.53 (m, 2H), 7.39-7.35 (m, 1H), 7.30 (d, *J*=9.48 Hz, 1H), 4.39 (q, *J*=7.13 Hz, 2H), 2.94 (t, *J*=7.32 Hz, 2H), 1.79-1.69 (m, 2H), 1.43 (t, *J*=7.13 Hz, 3H), 1.03 (t, *J*=7.36 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.2, 14.5, 23.6, 29.3, 59.3, 105.6, 111.3, 114.5, 118.8, 122.9, 123.8, 124.1, 128.4, 128.7, 131.6, 132.3, 134.8, 165.5; $C_{18}H_{19}O_2NNa$ ([M+ Na]$^+$) : 304.1308 found: 304.1305.

ethyl 2-isobutylpyrrolo[1,2-a]quinoline-3-carboxylate (3s).



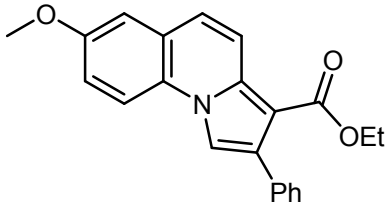
White solid (80% yield). m.p. 97-98 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.16 (d, *J*=9.48 Hz, 1H), 7.89 (d, *J*=8.44 Hz, 1H), 7.71 (m, 1H), 7.58-7.53 (m, 2H), 7.39-7.35 (m, 1H), 7.30 (d, *J*=9.48 Hz, 1H), 4.38 (q, *J*=7.13 Hz, 2H), 2.81 (d, *J*=6.96 Hz, 2H), 2.03-2.00 (m, 1H), 1.43 (t, *J*=7.13 Hz, 3H), 0.97 (d, *J*=6.64 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.5, 22.6, 29.0, 36.3, 59.3, 105.8, 112.1, 114.5, 118.9, 122.8, 123.8, 124.1, 128.4, 128.7, 130.4, 132.3, 134.8, 165.6; $C_{19}H_{21}O_2NNa$ ([M+ Na]$^+$) : 318.1464 found: 318.1462.

ethyl 1-methyl-2-phenylpyrrolo[1,2-a]quinoline-3-carboxylate (3t).



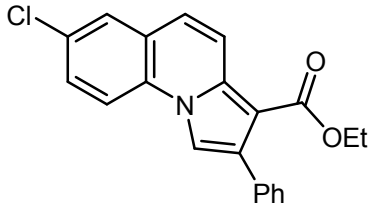
White solid (56% yield). m.p. 162-163 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.48 (d, *J*=8.68 Hz, 1H), 8.30 (d, *J*=9.40 Hz, 1H), 7.75 (dd, J_1=1.40 Hz, J_2=7.80 Hz, 1H), 7.55-7.50 (m, 1H), 7.44-7.30 (m, 7H), 4.14 (q, *J*=7.12 Hz, 2H), 2.79 (s, 3H), 1.036 (t, *J*=7.12 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.9, 16.5, 59.2, , 105.4, 116.8, 118.8, 123.0, 124.0, 124.9, 125.8, 126.6, 127.5, 128.8, 129.7, 130.8, 134.7, 135.1, 136.1, 165.1; $C_{22}H_{19}O_2NNa$ ([M+ Na]$^+$) : 352.1308 found: 352.1305.

ethyl 7-methoxy-2-phenylpyrrolo[1,2-a]quinoline-3-carboxylate (3u).



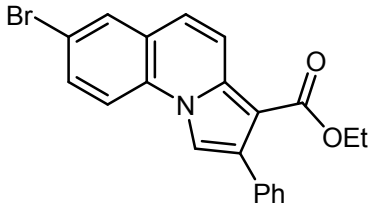
White solid (86% yield). m.p. 151-152 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.21 (d, *J*=9.52 Hz, 1H), 7.84 (d, *J*=9.08 Hz, 1H), 7.71 (s, 1H), 7.55-7.53 (m, 2H), 7.42-7.38 (m, 2H), 7.36-7.33 (m, 1H), 7.30 (d, *J*=9.56 Hz, 1H), 7.20-7.14 (m, 2H), 4.26 (q, *J*=7.12 Hz, 2H), 3.91(s, 3H), 1.22 (t, *J*=7.12 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.1, 55.5, 59.3, 104.7, 109.8, 112.0, 115.7, 117.7, 119.3, 123.1, 124.9, 126.8, 126.9, 127.4, 129.8, 131.0, 134.2, 135.1, 156.3, 164.9; $C_{22}H_{19}O_3NNa$ ([M+ Na]$^+$) : 368.1257 found: 368.1252.

ethyl 7-chloro-2-phenylpyrrolo[1,2-a]quinoline-3-carboxylate (3v).



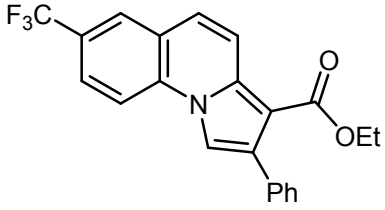
White solid (93% yield). m.p. 219-220 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.23 (d, *J*=9.52 Hz, 1H), 7.85 (d, *J*=8.92 Hz, 1H), 7.75 (s, 1H), 7.71 (d, *J*=9.52 Hz, 1H), 7.54-7.51 (m, 3H), 7.42-7.33 (m, 3H), 7.28-7.27 (m, 1H), 4.27 (q, *J*=7.13 Hz, 2H), 1.21 (t, *J*=9.52 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.1, 59.6, 105.9, 112.5, 116.0, 120.2, 122.3, 125.1, 127.1, 127.6, 127.9, 128.8, 129.8, 130.0, 130.8, 131.7, 134.5, 134.8, 164.8; $C_{21}H_{16}O_2NClNa$ ([M+ Na]$^+$) : 372.0761 found: 372.0757

ethyl 7-bromo-2-phenylpyrrolo[1,2-a]quinoline-3-carboxylate (3w).



White solid (91% yield). m.p. 207-208 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.22 (d, *J*=9.52 Hz, 1H), 7.86 (m, 1H), 7.77 (d, *J*=8.92 Hz, 1H), 7.73 (s, 1H), 7.64 (dd, *J*=8.88 Hz, *J*=2.12 Hz, 1H), 7.52 (m, 2H), 7.38 (m, 3H), 7.22 (m, 1H), 4.26 (q, *J*=7.12 Hz, 2H), 1.22 (t, *J*=7.12 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.1, 59.7, 105.9, 112.5, 116.2, 117.6, 120.2, 122.2, 125.5, 127.1, 127.6, 129.8, 131.0, 131.1, 131.5, 131.7, 134.5, 134.8, 164.8; $C_{21}H_{16}O_2NBrNa$ ([M+ Na]$^+$) : 416.0256 found: 416.0250.

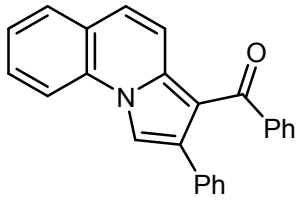
ethyl 2-phenyl-7-(trifluoromethyl)pyrrolo[1,2-a]quinoline-3-carboxylate (3x).



White solid (95% yield). m.p. 129-130 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.32 (d, *J*=9.52 Hz, 1H),

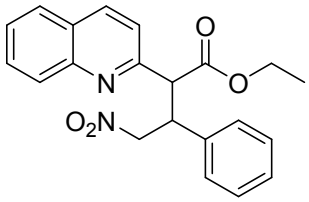
8.03-8.01 (m, 2H), 7.82-7.79 (m, 2H), 7.55-7.53 (m, 2H), 7.44-7.35 (m, 4H), 4.28 (q, *J*=7.12 Hz, 2H), 1.23 (t, *J*=7.12 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.1, 59.8, 106.4, 112.7, 115.3, 120.5, 122.5, 122.8, 123.6, 125.0, 125.0, 125.2, 126.2, 126.2, 126.3, 126.6, 126.9, 127.2, 127.7, 129.8, 132.1, 134.0, 134.6, 134.9, 164.7; $C_{22}H_{16}O_2NF_3Na$ ([M+ Na]$^+$) : 406.1025 found: 406.1019.

phenyl(2-phenylpyrrolo[1,2-a]quinolin-3-yl)methanone (3y).



White solid (82% yield). m.p. 149-150 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00 (d, *J*=8.40 Hz, 1H), 7.91 (s, 1H), 7.89 (d, *J*=9.52 Hz, 1H), 7.76 (d, *J*=7.84 Hz, 1H), 7.65-7.61 (m, 3H), 7.46-7.43 (m, 1H), 7.35 (d, *J*=9.44 Hz, 1H), 7.30-7.24 (m, 3H), 7.17-7.07 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 110.5, 113.4, 113.6, 117.5, 122.8, 123.2, 123.7, 125.5, 126.6, 126.9, 127.8, 127.9, 128.3, 128.7, 130.1, 130.4, 131.2, 133.5, 134.0, 138.7, 191.5; $C_{25}H_{17}ONNa$ ([M+ Na]$^+$) : 370.1202 found: 370.1196.

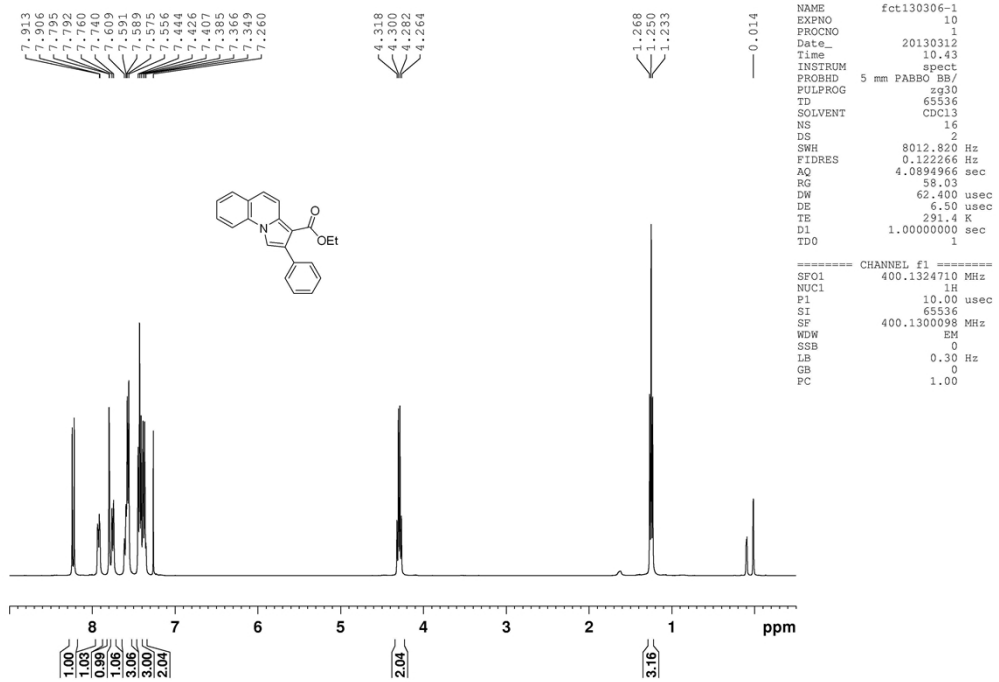
ethyl 4-nitro-3-phenyl-2-(quinolin-2-yl)butanoate (4a).



White solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.20 (d, *J*=8.48 Hz, 1H), 8.13 (d, *J*=8.48 Hz, 1H), 7.83 (d, *J*=8.04 Hz, 1H), 7.77-7.73 (m, 1H), 7.64 (d, *J*=8.48 Hz, 1H), 7.59-7.56 (m, 1H), 7.37-7.27 (m, 5H), 4.70-4.64 (m, 1H), 4.60-4.52 (m, 2H), 4.46 (d, *J*=10.76 Hz, 1H), 3.95-3.85 (m, 2H), 0.91 (t, *J*=7.10 Hz, 3H) ; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.7, 45.7, 58.0, 61.2, 78.4, 120.4, 127.0, 127.4, 127.6, 128.1, 128.2, 128.8, 129.4, 130.0, 137.3, 137.5, 147.8, 155.3, 170.2.

4. Copies of ^{1}H NMR and ^{13}C NMR Spectra

3a

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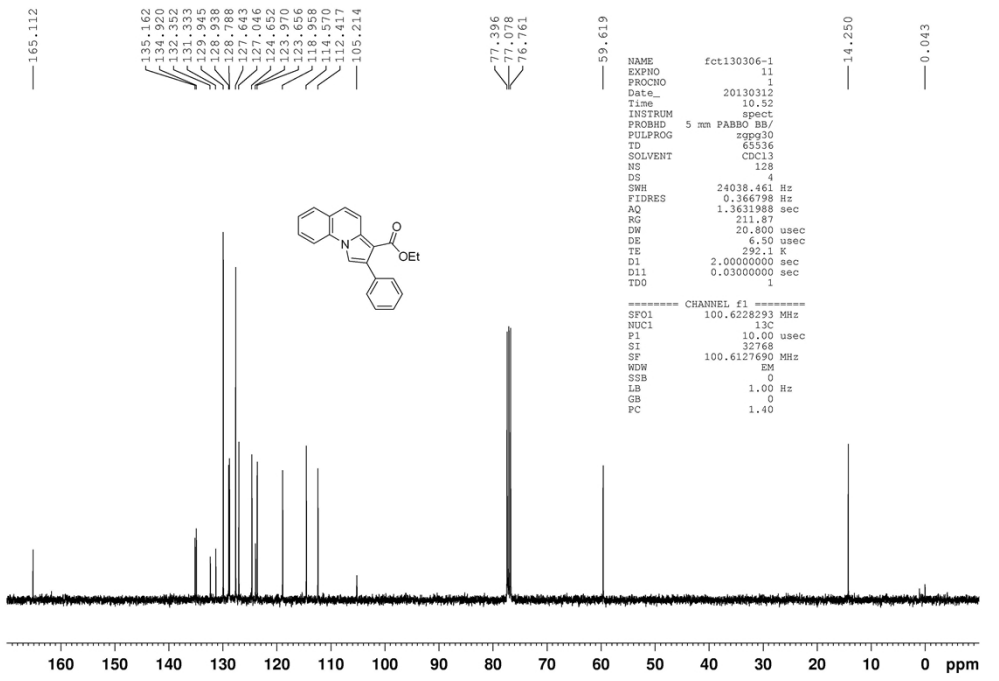


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TE                292.1 K
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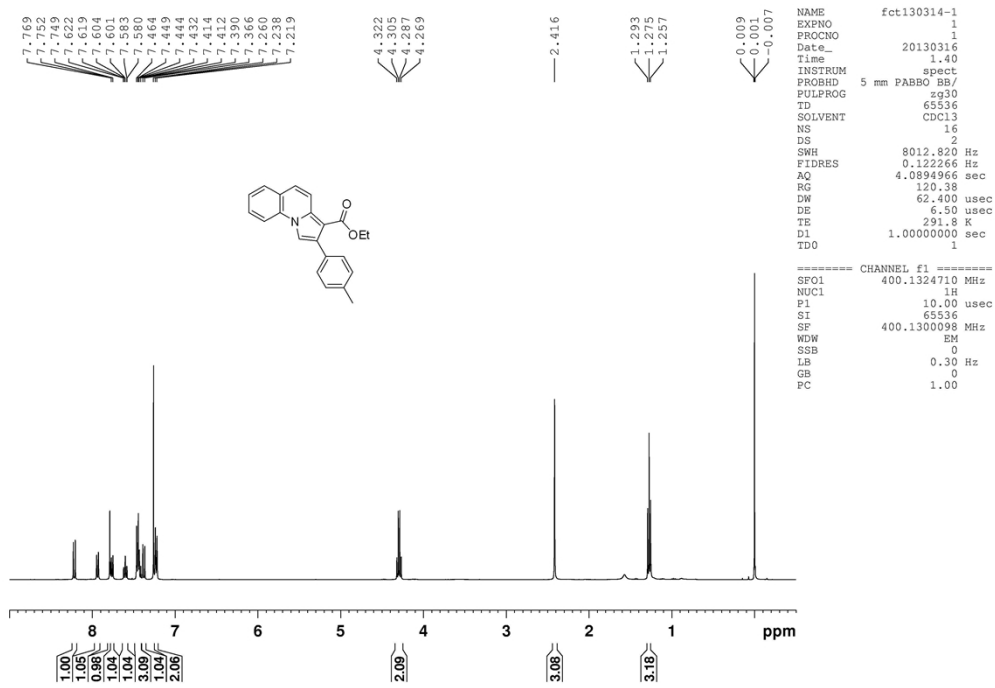
3b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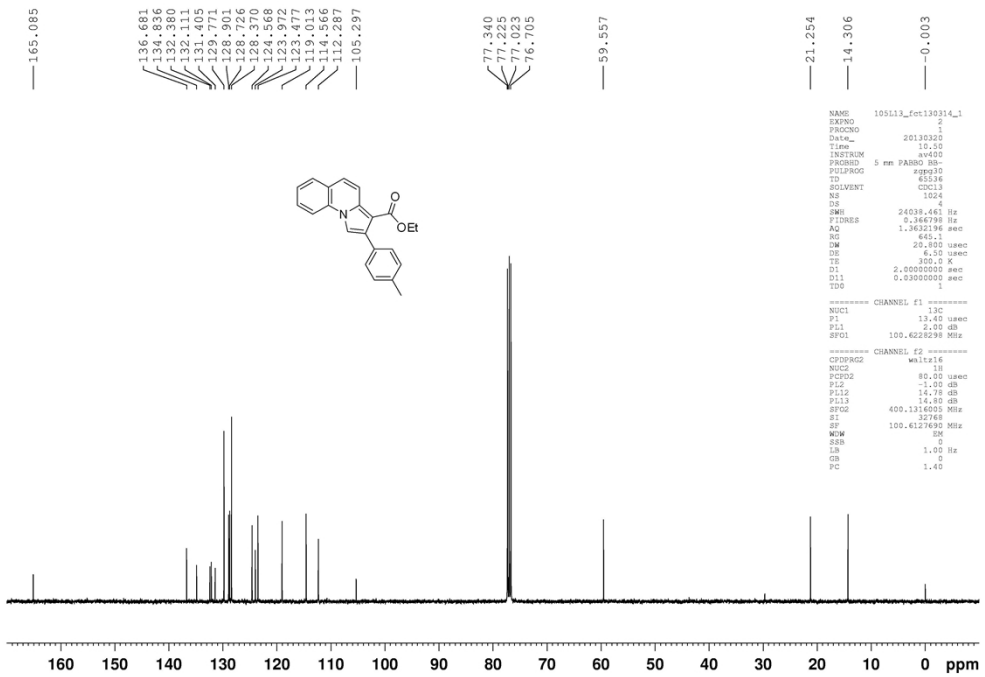
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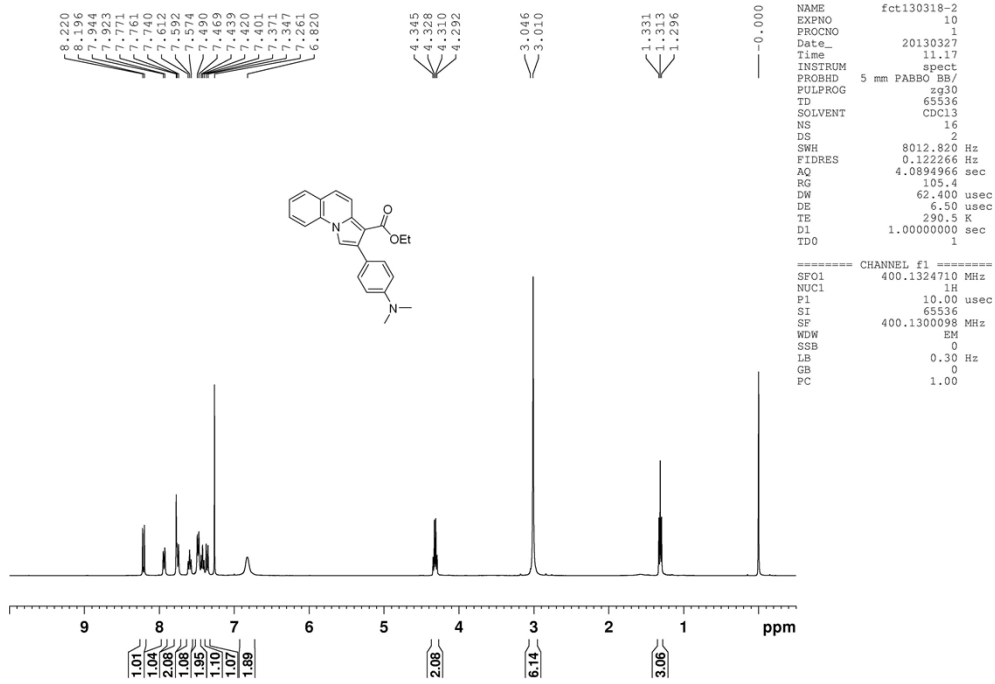
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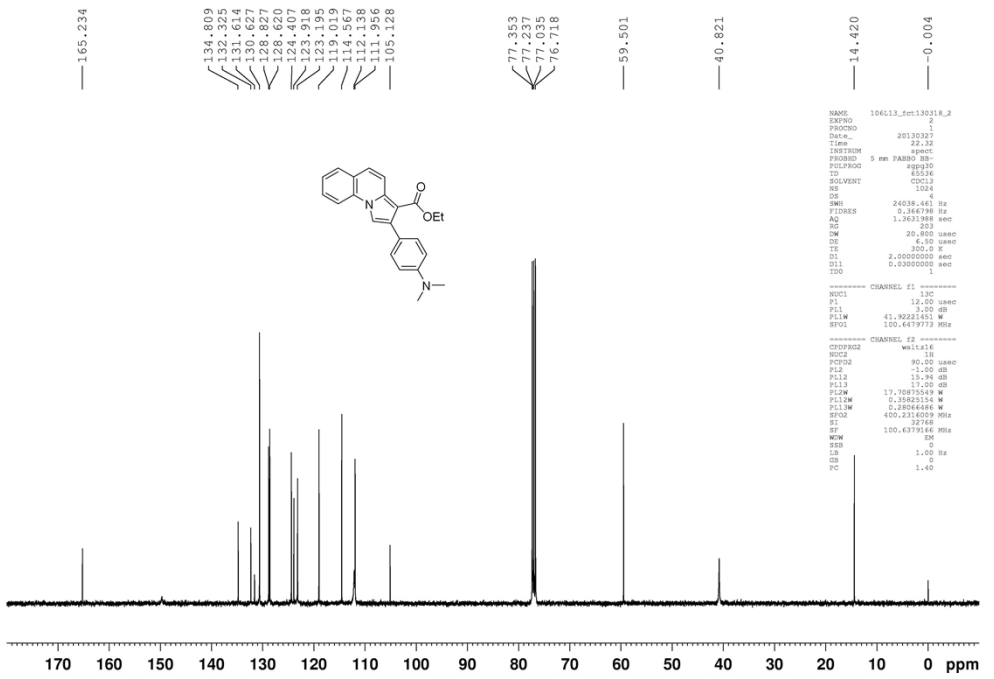
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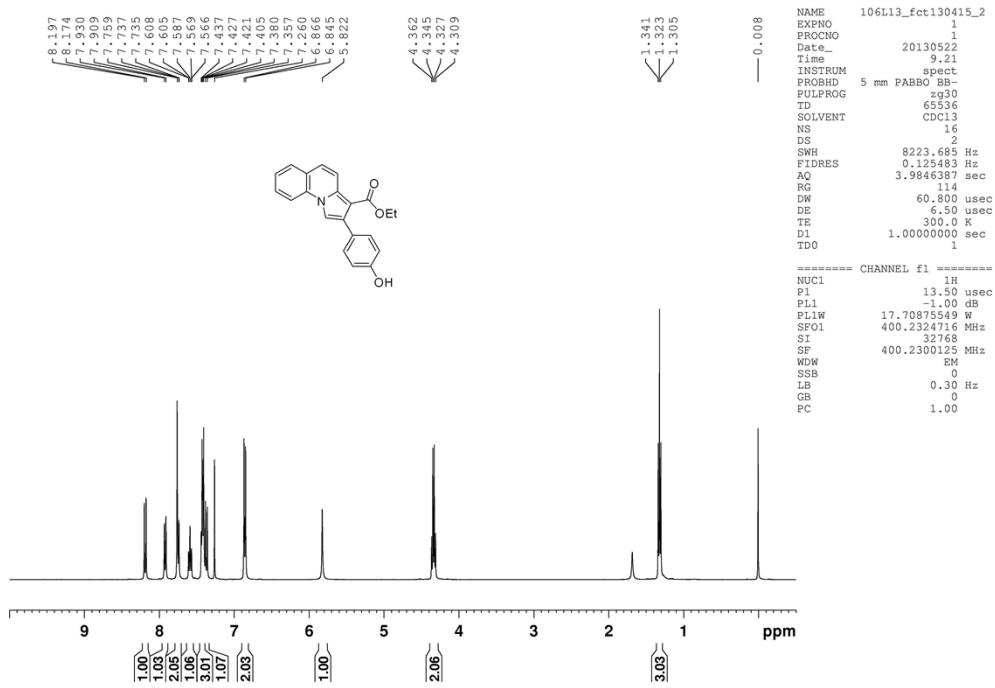
3d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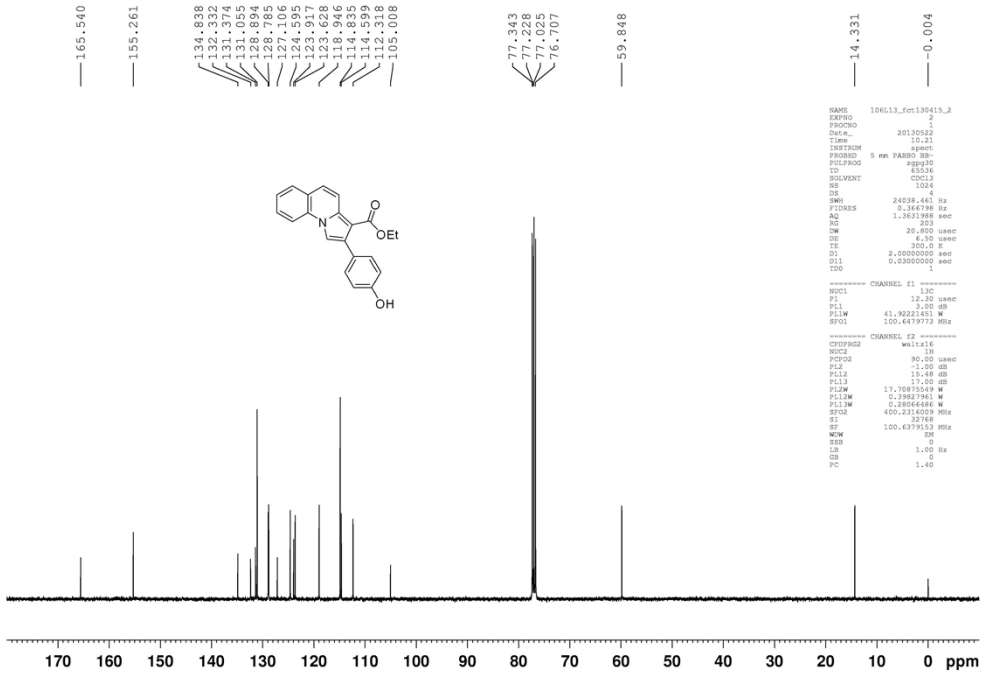
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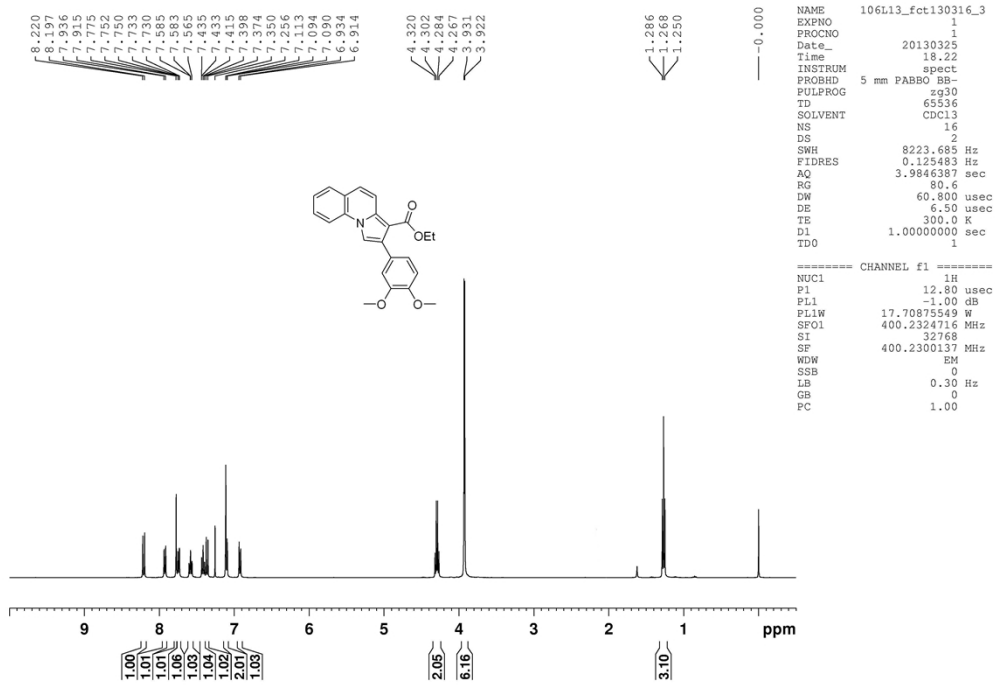
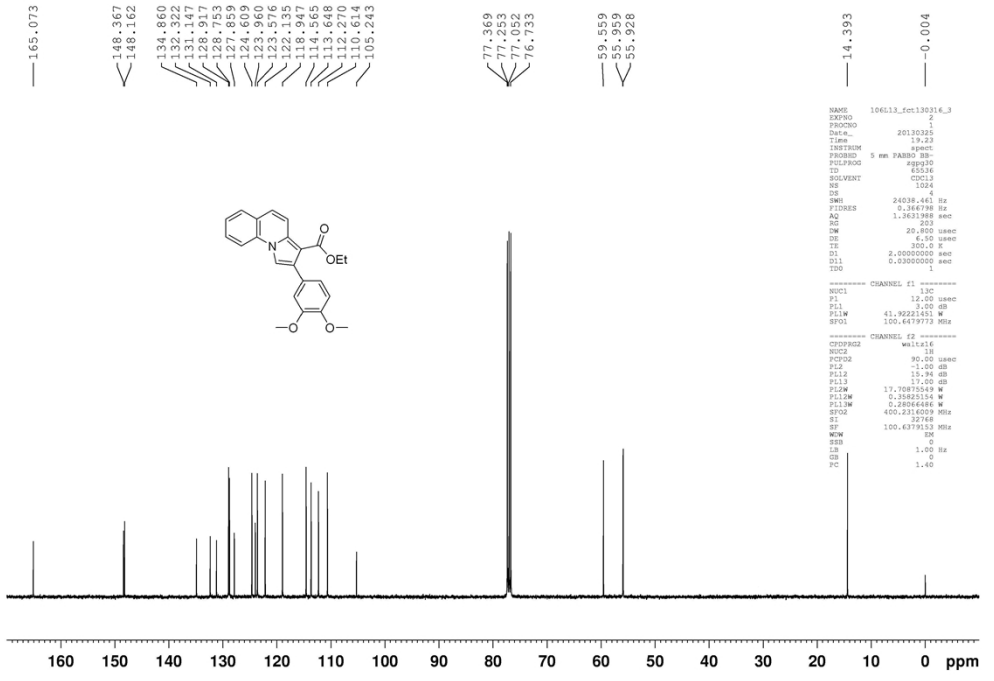
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.48 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.39827961 W
PL13W 0.28064486 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379153 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3e



NAME 106L13_fct130316_3
EXPNO 1
PROCNO 1
Date_ 20130325
Time 18.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 -1.00 dB
PL1W 17.70875549 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300137 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

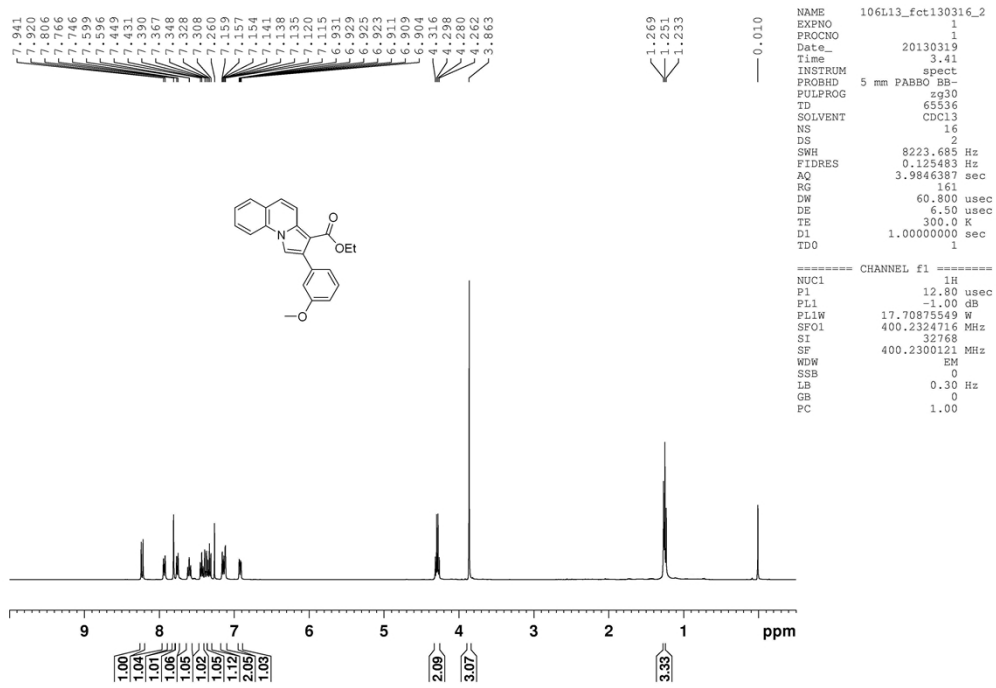


NAME 106L13_fct130316_3
EXPNO 2
PROCNO 1
Date_ 20130325
Time 19.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

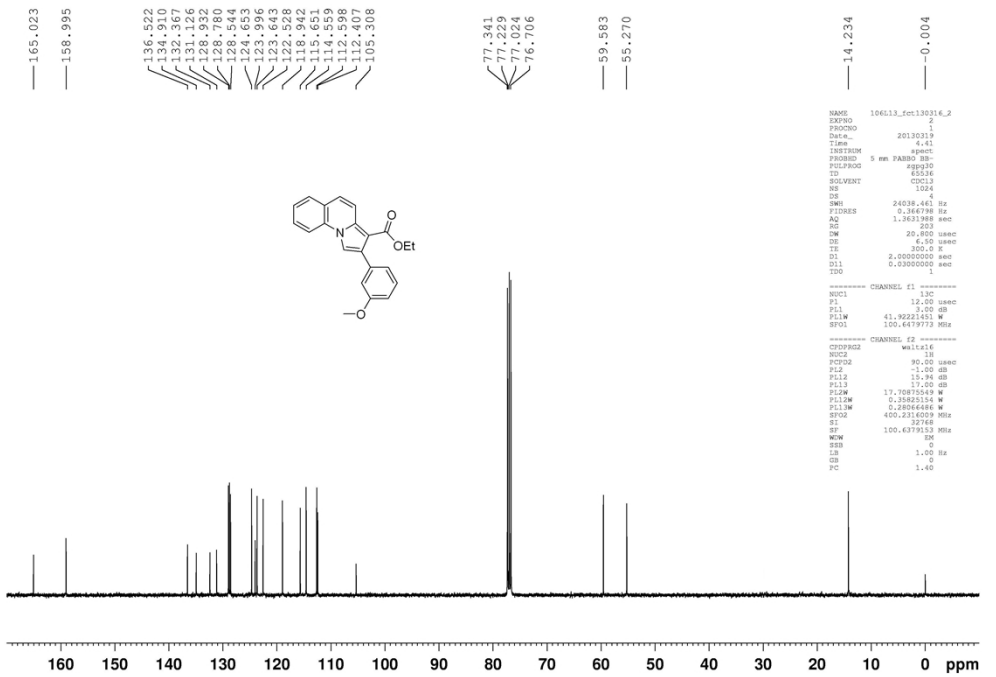
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.94 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.35825154 W
PL13W 0.28064486 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379153 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3f

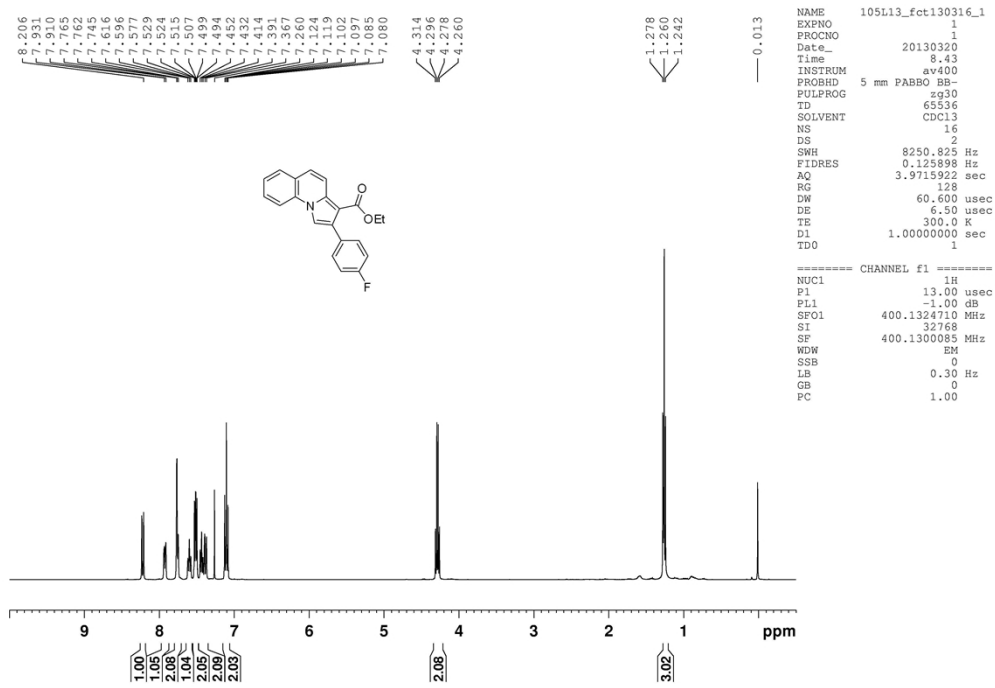



```
NAME          106L13_fct130316_2
EXPNO                          1
PROCNO                         1
Date_                   20130319
Time                        3.41
INSTRUM                    spect
PROBHD   5 mm PABBO BB-
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    8223.685 Hz
FIDRES                 0.125483 Hz
AQ                     3.9846387 sec
RG                          161
DW                       60.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                       12.80 usec
PL1                      -1.00 dB
PL1W                17.70875549 W
SFO1                400.2324716 MHz
SI                          32768
SF                  400.2300121 MHz
WDW                           EM
SSB                            0
LB                        0.30 Hz
GB                             0
PC                          1.00
```

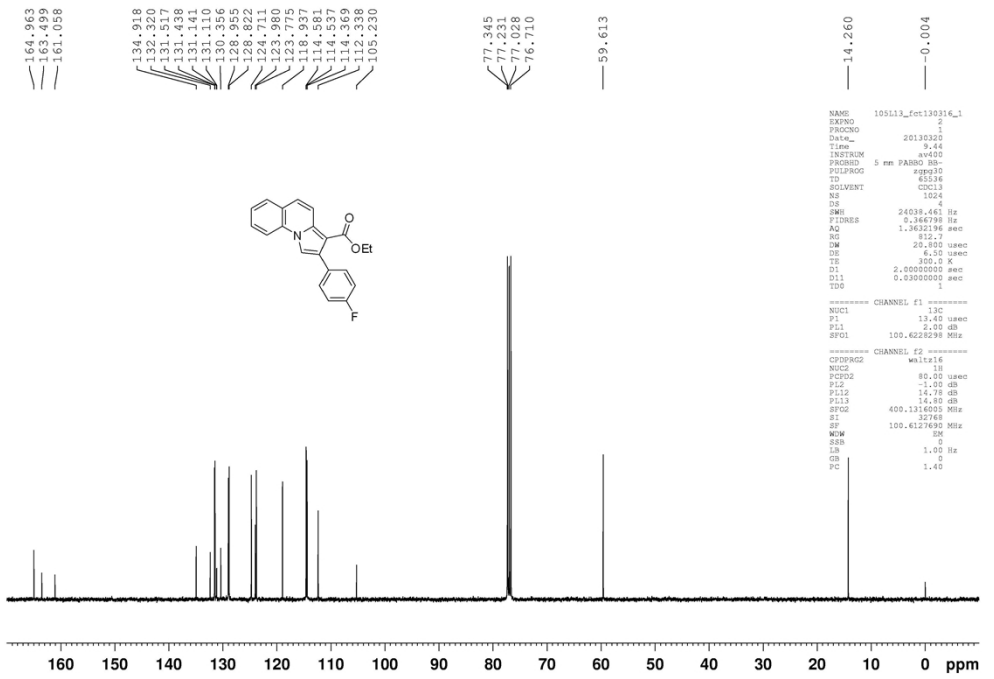



```
NAME          106L13_fct130316_2
EXPNO                          2
PROCNO                         1
Date_                   20130319
Time                        3.41
INSTRUM                    spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                   24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631988 sec
RG                          203
DW                       20.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                       12.00 usec
PL1                       3.00 dB
PL1W                41.92221451 W
SFO1                100.6479773 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                    90.00 usec
PL2                      -1.00 dB
PL12                     15.94 dB
PL13                     17.00 dB
PL2W                17.70875549 W
PL12W                0.35825154 W
PL13W                0.28064496 W
SFO2                400.2316009 MHz
SI                          32768
SF                  100.6379153 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                          1.40
```

3g

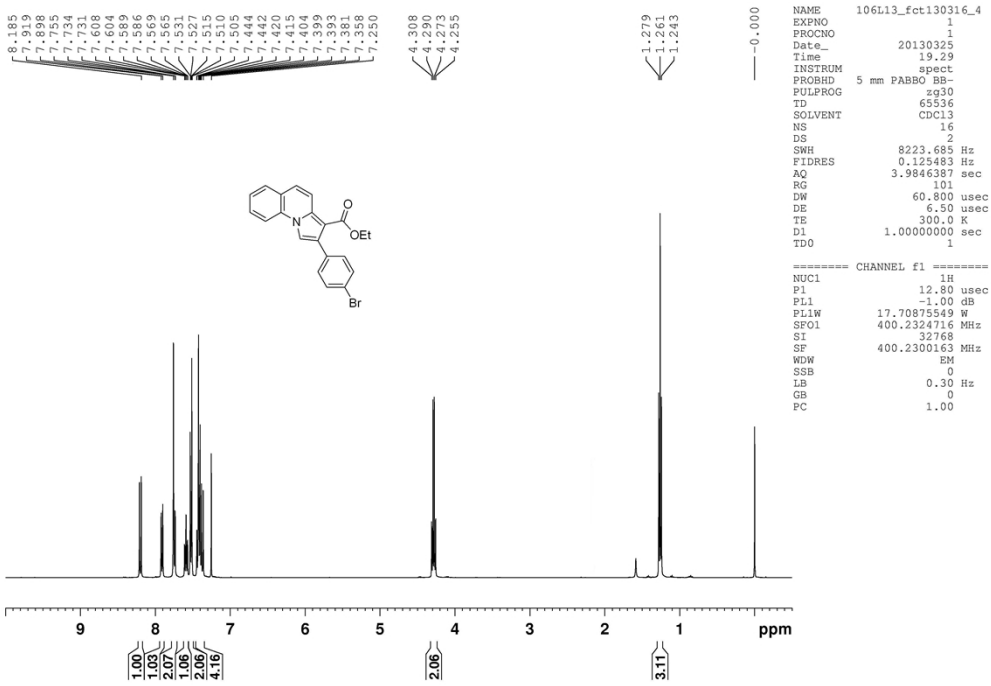


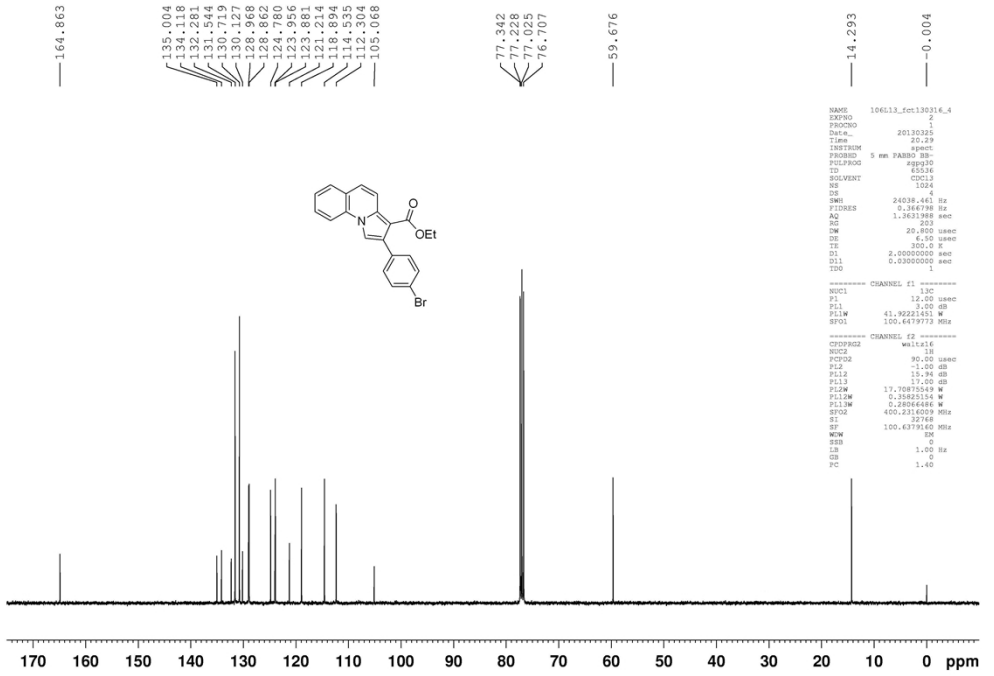

```
NAME          105L13_fct130316_1
EXPNO                          1
PROCNO                         1
Date_                   20130320
Time                        8.43
INSTRUM                   av400
PROBHD     5 mm PABBO BB-
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    8250.825 Hz
FIDRES                 0.125898 Hz
AQ                     3.9715922 sec
RG                          128
DW                       60.600 usec
DE                        6.50 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                       13.00 usec
PL1                      -1.00 dB
SFO1                400.1324710 MHz
SI                        32768
SF                  400.1300085 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                        1.00
```



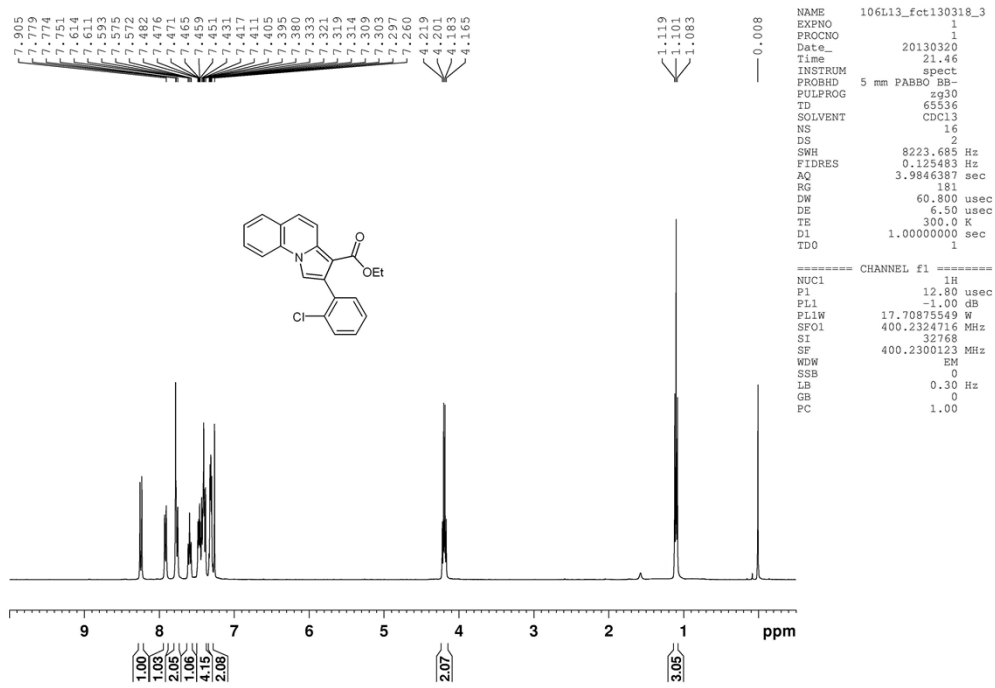

```
NAME          105L13_fct130316_1
EXPNO                          2
PROCNO                         1
Date_                   20130320
Time                        9.44
INSTRUM                   av400
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                   24038.461 Hz
FIDRES                 0.366798 Hz
AQ                     1.3632196 sec
RG                         812.7
DW                       20.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                       13.40 usec
PL1                       2.00 dB
SFO1                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                      -1.00 dB
PL12                     14.78 dB
PL13                     14.80 dB
SFO2                400.1316005 MHz
SI                        32768
SF                  100.6127690 MHz
WDW                          EM
SSB                           0
LB                        1.00 Hz
GB                            0
PC                        1.40
```

3h

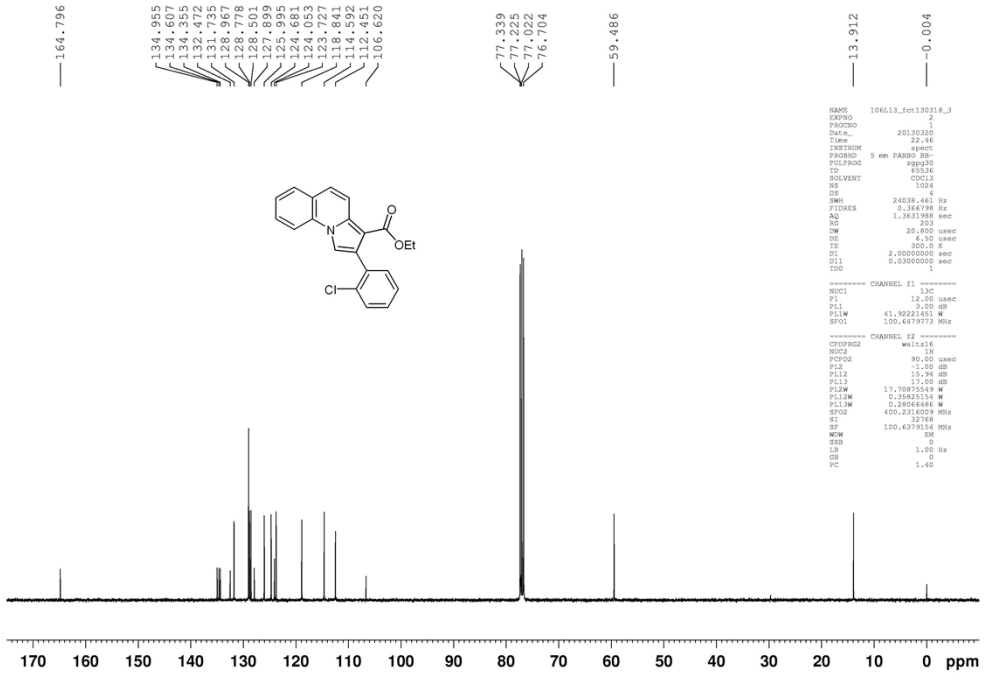

8.185
7.919
7.898
7.755
7.734
7.731
7.608
7.604
7.589
7.586
7.569
7.565
7.531
7.527
7.515
7.510
7.505
7.444
7.442
7.420
7.415
7.404
7.399
7.393
7.381
7.358
7.250
4.308
4.290
4.273
4.255
1.279
1.261
1.243
-0.000
NAME 106L13_fct130316_4
EXPNO 1
PROCNO 1
Date_ 20130325
Time 19.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 -1.00 dB
PL1W 17.70875549 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300163 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OEt
N
Br
9
8
7
6
5
4
3
2
1
ppm
1.00
1.03
2.07
1.06
2.06
4.16
2.06
3.11


164.863
135.004
134.118
132.281
131.544
130.719
130.127
128.968
128.862
124.780
123.956
123.881
121.214
118.894
114.535
112.304
105.068
77.342
77.228
77.025
76.707
59.676
14.293
-0.004
NAME 106L13_fct130316_4
EXPNO 2
PROCNO 1
Date_ 20130325
Time 20.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.94 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.35825154 W
PL13W 0.28064686 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379160 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OEt
N
Br
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3i

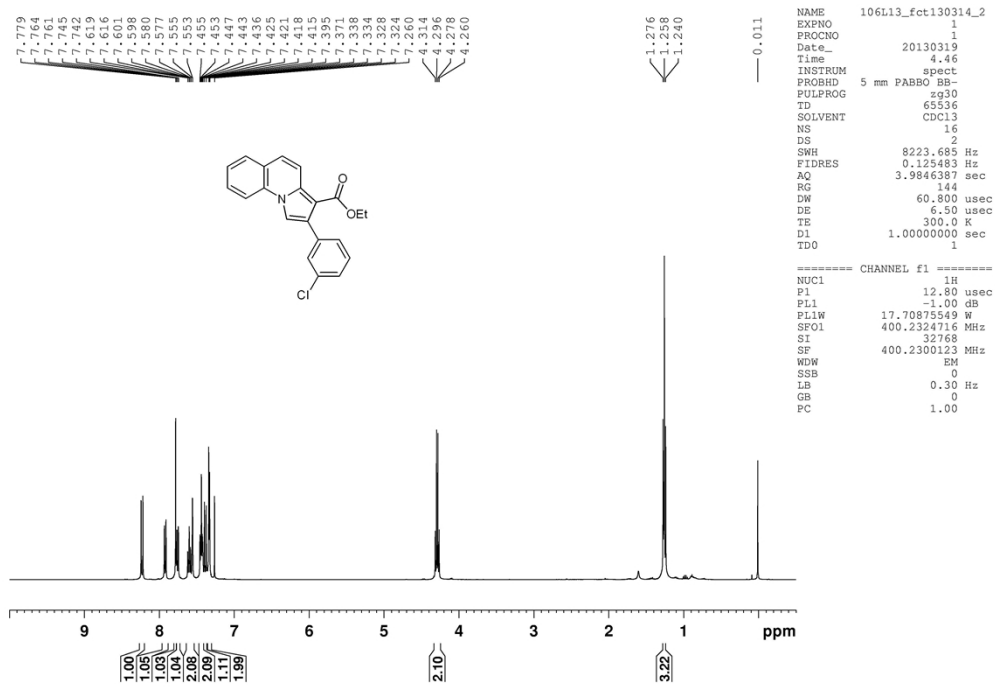


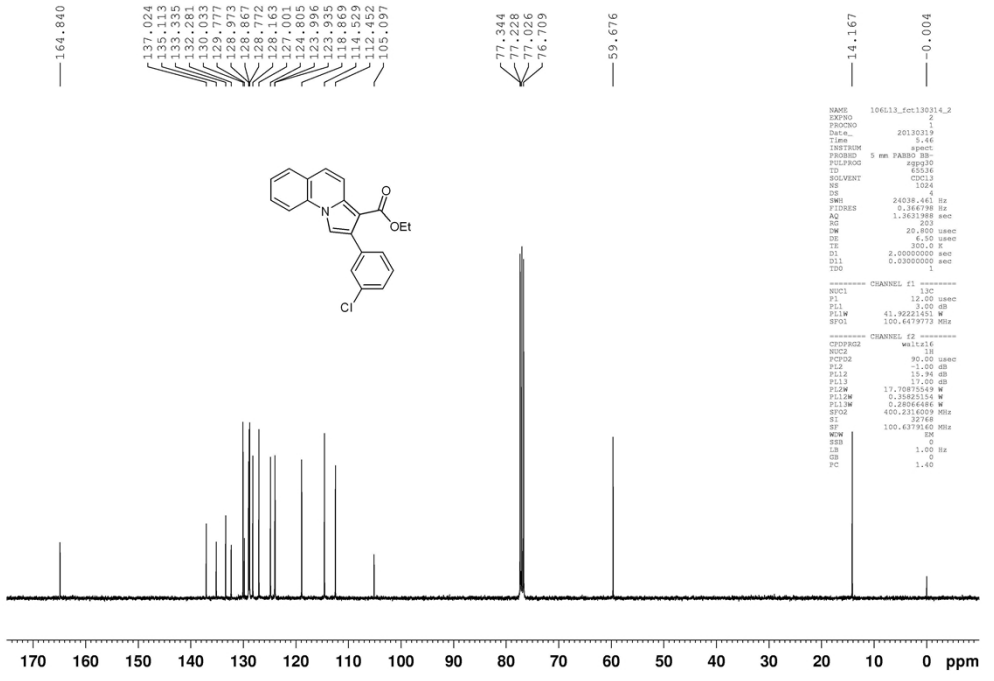

```
NAME          106L13_fct130318_3
EXPNO                          1
PROCNO                         1
Date_                   20130320
Time                       21.46
INSTRUM                    spect
PROBHD  5 mm PABBO BB-
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     8223.685 Hz
FIDRES                  0.125483 Hz
AQ                      3.9846387 sec
RG                           181
DW                        60.800 usec
DE                         6.50 usec
TE                        300.0 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         12.80 usec
PL1                        -1.00 dB
PL1W                17.70875549 W
SFO1                400.2324716 MHz
SI                         32768
SF                  400.2300123 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



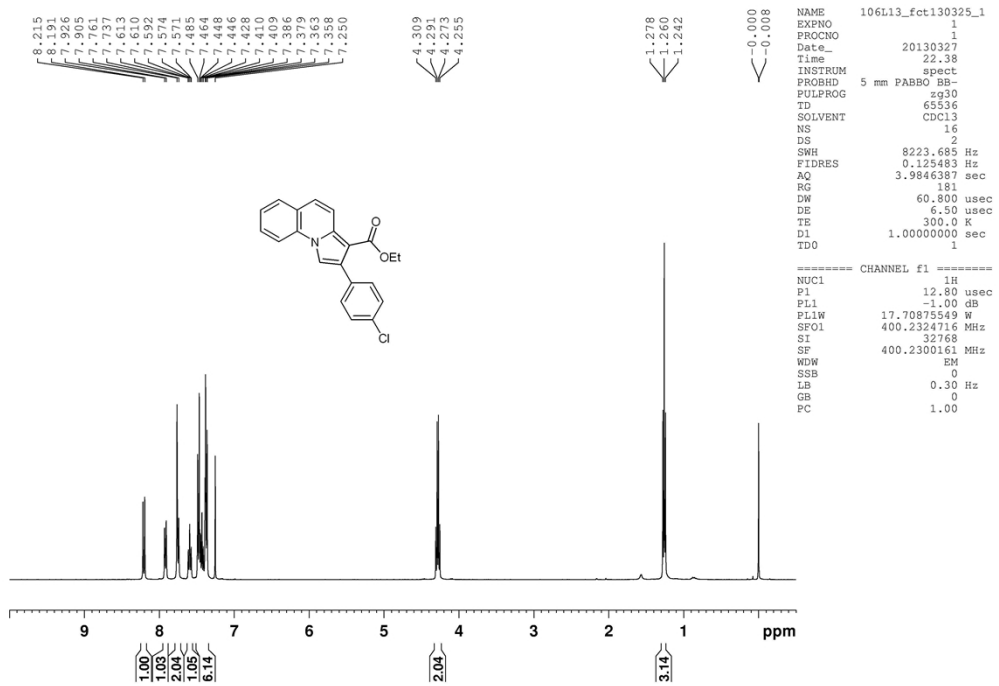

```
NAME          106L13_fct130318_3
EXPNO                          2
PROCNO                         1
Date_                   20130320
Time                       22.46
INSTRUM                    spect
PROBHD  5 mm PABBO BB-
PULPROG                 zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          1024
DS                             4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631988 sec
RG                           203
DW                        20.800 usec
DE                         6.50 usec
TE                        300.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          13C
P1                         12.00 usec
PL1                         3.00 dB
PL1W                41.92221451 W
SFO1                100.6479773 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                          1H
PCPD2                      90.00 usec
PL2                        -1.00 dB
PL12                       15.94 dB
PL13                       17.00 dB
PL2W                17.70875549 W
PL12W                0.35825154 W
PL13W                0.28064486 W
SFO2                400.2316009 MHz
SI                         32768
SF                  100.6379154 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

3j

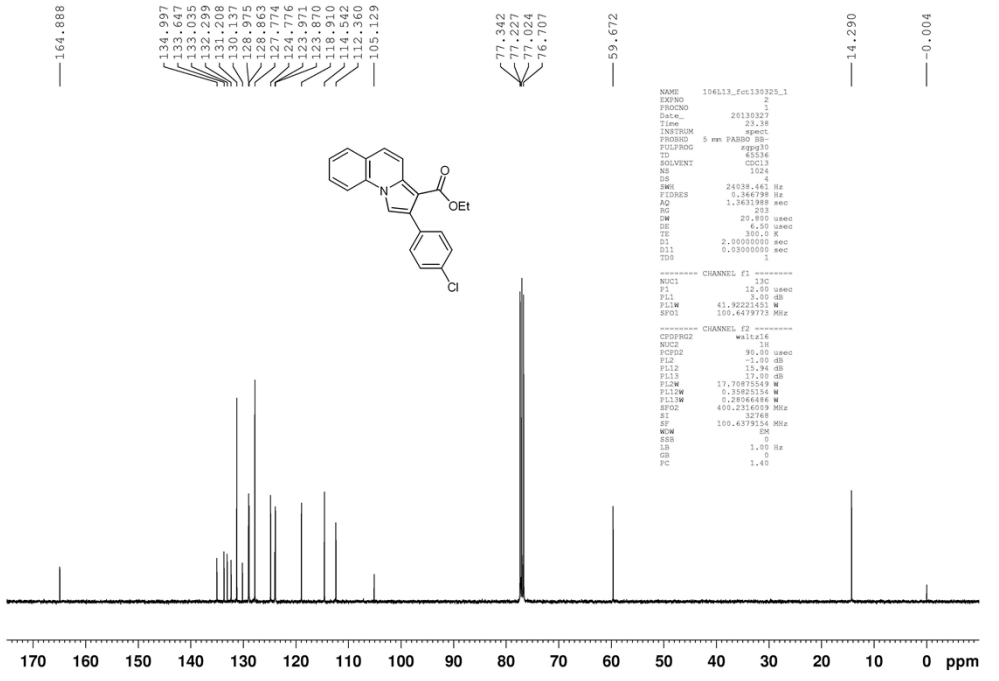

7.779
7.764
7.761
7.745
7.742
7.619
7.616
7.601
7.598
7.580
7.577
7.555
7.553
7.455
7.453
7.447
7.443
7.436
7.425
7.421
7.418
7.415
7.395
7.371
7.338
7.334
7.328
7.324
7.260
4.314
4.296
4.278
4.260
1.276
1.258
1.240
0.011
OEt
Cl
NAME 106L13_fct130314_2
EXPNO 1
PROCNO 1
Date_ 20130319
Time 4.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 -1.00 dB
PL1W 17.70875549 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 ppm
1.00 1.05 1.03 1.04 2.08 2.09 1.11 1.99
2.10
3.22


164.840
137.024
136.113
133.335
132.281
130.033
129.777
128.973
128.867
128.772
128.163
127.001
124.805
123.996
123.935
118.869
114.529
112.452
105.097
77.344
77.228
77.026
76.709
59.676
14.167
-0.004
OEt
Cl
NAME 106L13_fct130314_2
EXPNO 2
PROCNO 1
Date_ 20130319
Time 5.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.94 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.35825154 W
PL13W 0.28064486 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379160 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3k

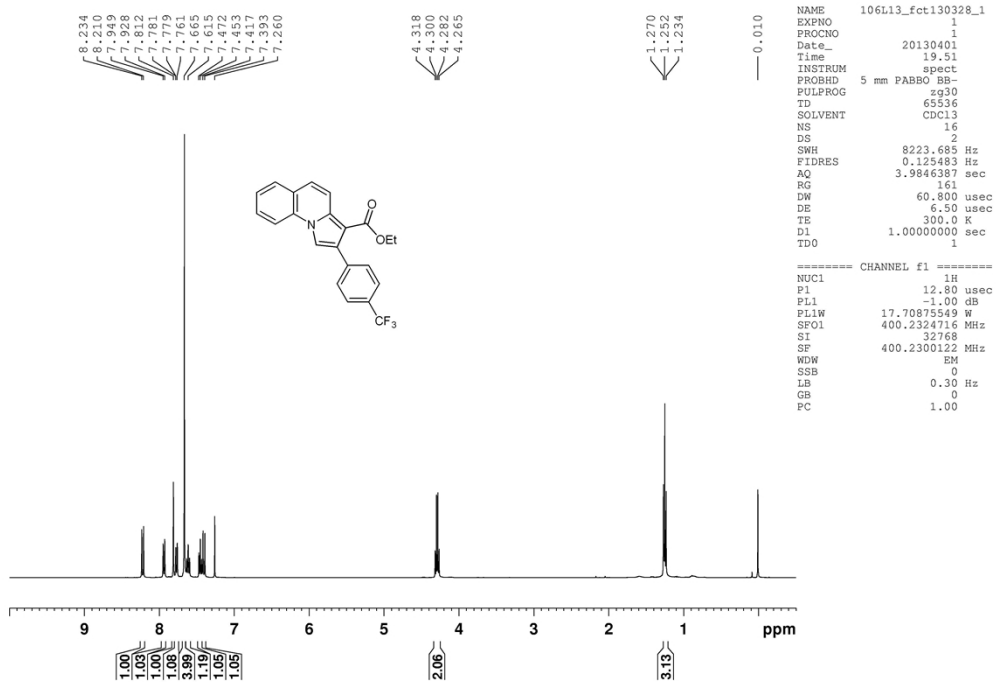



```
NAME        106L13_fct130325_1
EXPNO                        1
PROCNO                       1
Date_                 20130327
Time                     22.38
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                3.9846387 sec
RG                         181
DW                    60.800 usec
DE                     6.50 usec
TE                     300.0 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                     12.80 usec
PL1                    -1.00 dB
PL1W              17.70875549 W
SFO1              400.2324716 MHz
SI                       32768
SF                400.2300161 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                       1.00
```



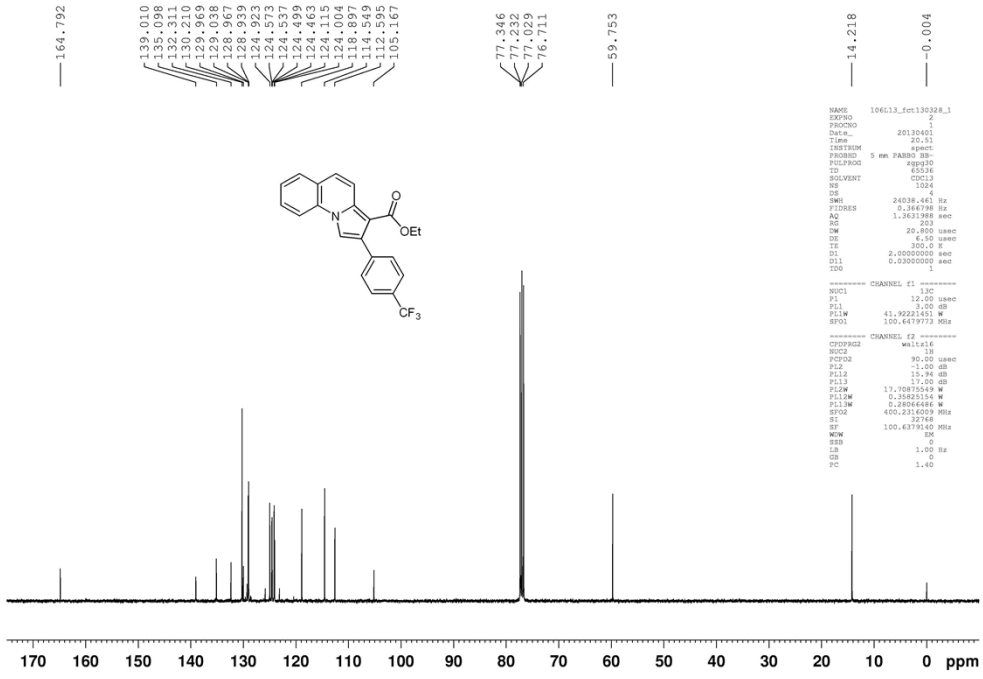

```
NAME        106L13_fct130325_1
EXPNO                        2
PROCNO                       1
Date_                 20130327
Time                     23.38
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                1.3631988 sec
RG                         203
DW                    20.800 usec
DE                     6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                     12.00 usec
PL1                     3.00 dB
PL1W              41.92221451 W
SFO1              100.6479773 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  90.00 usec
PL2                    -1.00 dB
PL12                   15.96 dB
PL13                   17.00 dB
PL2W              17.70875549 W
PL12W              0.35925154 W
PL13W              0.28066496 W
SFO2              400.2316009 MHz
SI                       32768
SF                100.6379154 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                       1.40
```

3l



NAME 106L13_fct130328_1
EXPNO 1
PROCNO 1
Date_ 20130401
Time 19.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 -1.00 dB
PL1W 17.70875549 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

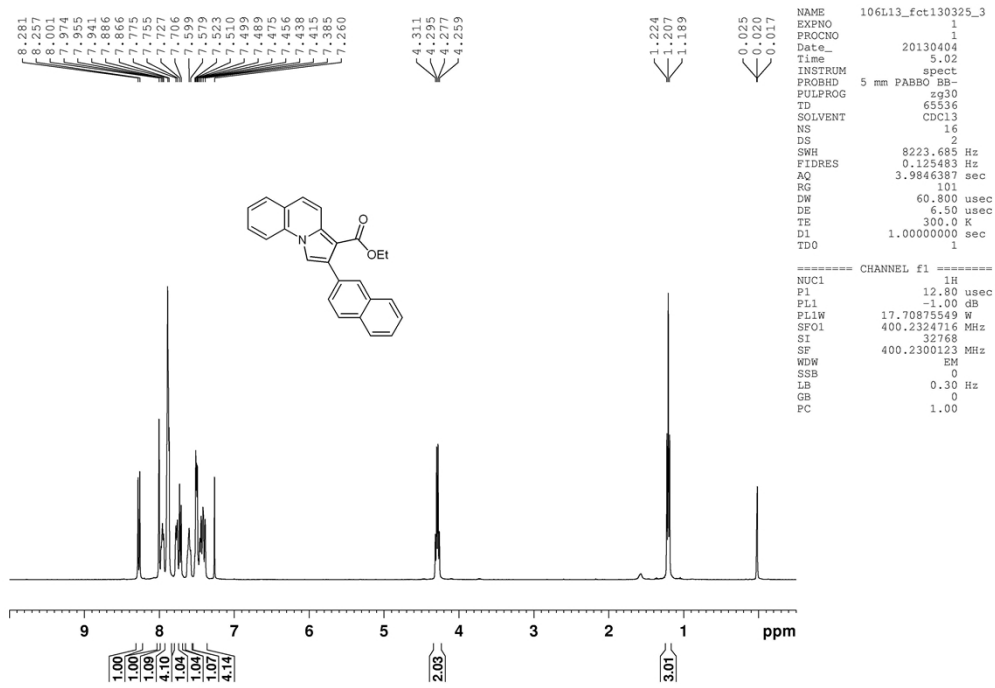


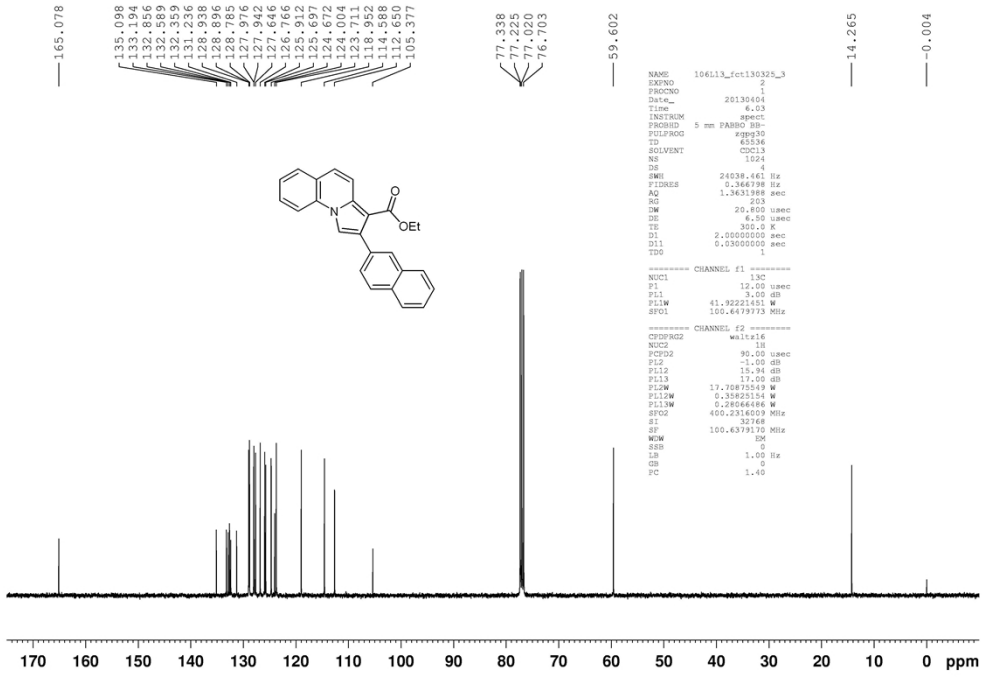
NAME 106L13_fct130328_1
EXPNO 2
PROCNO 1
Date_ 20130401
Time 20.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz

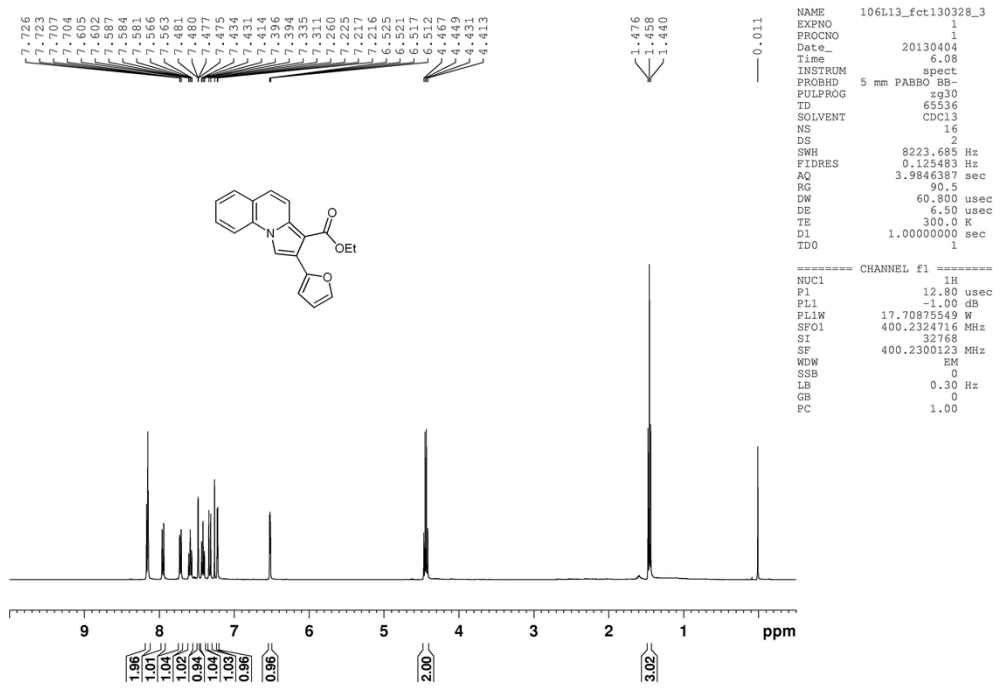
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.94 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.35825154 W
PL13W 0.28064486 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

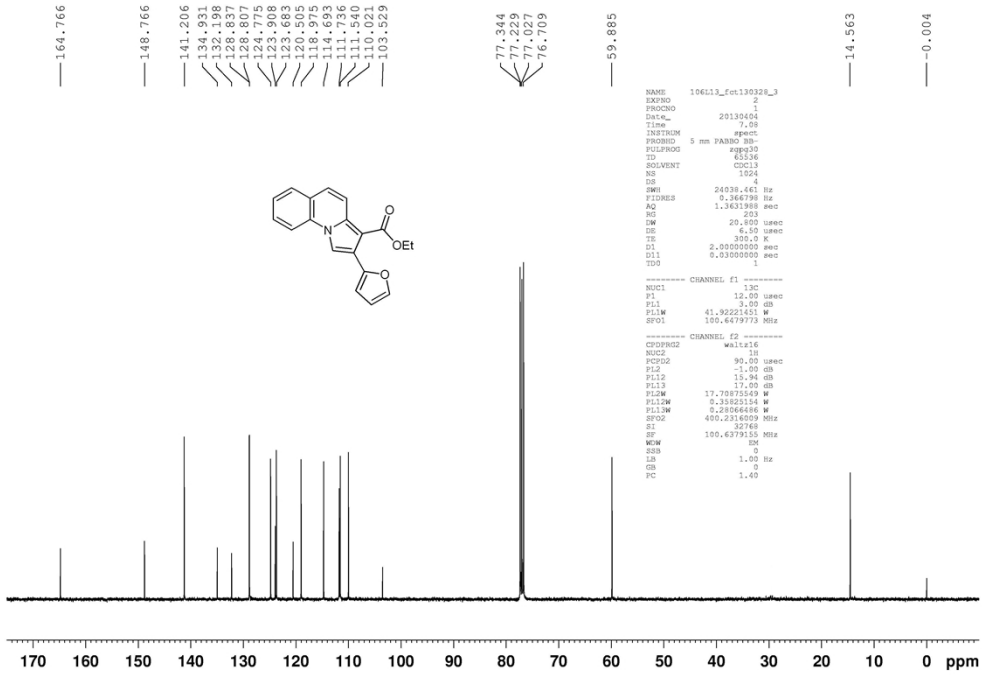
3m


8.281
8.257
8.001
7.974
7.955
7.941
7.866
7.796
7.775
7.755
7.727
7.706
7.599
7.579
7.523
7.510
7.499
7.489
7.475
7.456
7.438
7.415
7.385
7.260
4.311
4.295
4.277
4.259
1.224
1.207
1.189
0.025
0.020
0.017
OEt
9
8
7
6
5
4
3
2
1
ppm
1.00
1.00
1.09
4.10
1.04
1.04
1.07
4.14
2.03
3.01

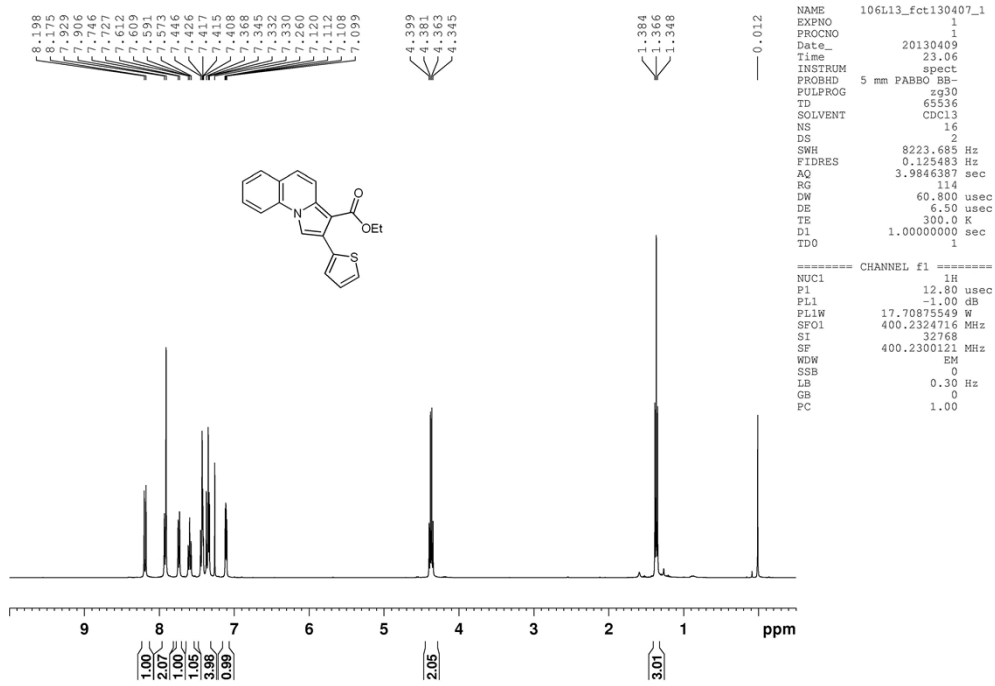

165.078
135.098
133.194
132.855
132.589
132.359
131.236
128.938
128.896
128.785
127.976
127.942
127.646
126.766
125.912
125.697
124.672
124.004
123.711
118.952
114.588
112.650
105.377
77.338
77.225
77.020
76.703
59.602
14.265
-0.004
OEt
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3n

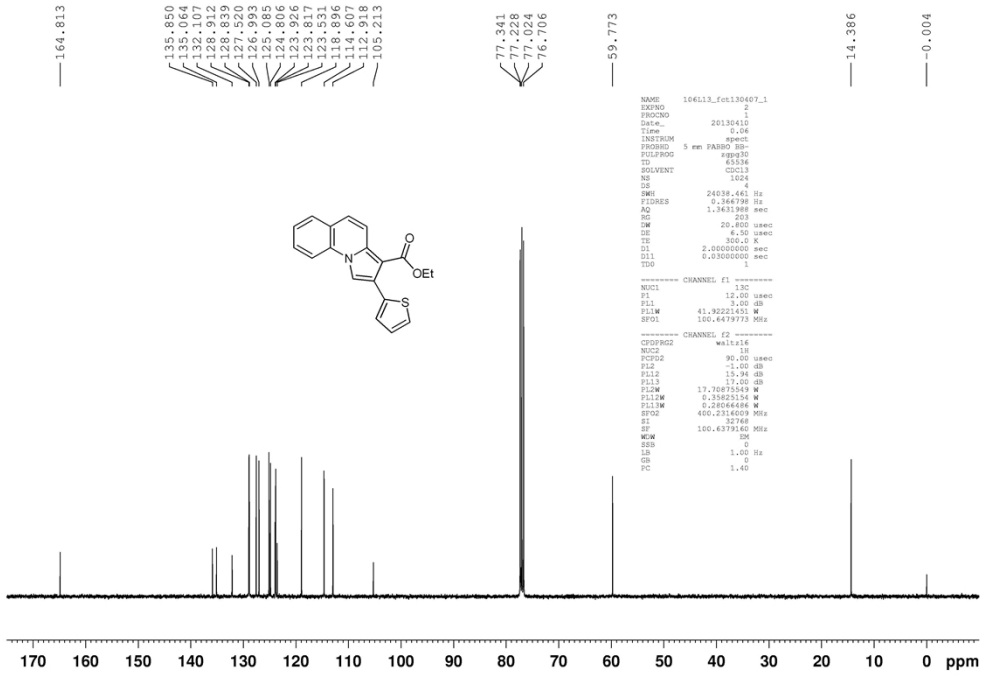

7.726
7.723
7.707
7.704
7.605
7.602
7.587
7.584
7.581
7.566
7.563
7.481
7.480
7.477
7.475
7.434
7.431
7.414
7.396
7.394
7.335
7.311
7.260
7.225
7.217
7.216
6.525
6.521
6.517
6.512
4.467
4.449
4.431
4.413
1.476
1.458
1.440
0.011
OEt
9
8
7
6
5
4
3
2
1
ppm
1.96
1.01
1.04
1.02
0.94
1.04
1.03
0.96
0.96
2.00
3.02


164.766
148.766
141.206
134.931
132.198
128.837
128.807
124.775
123.908
123.683
120.505
118.975
114.693
111.736
111.540
110.021
103.529
77.344
77.229
77.027
76.709
59.885
14.563
-0.004
OEt
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3o

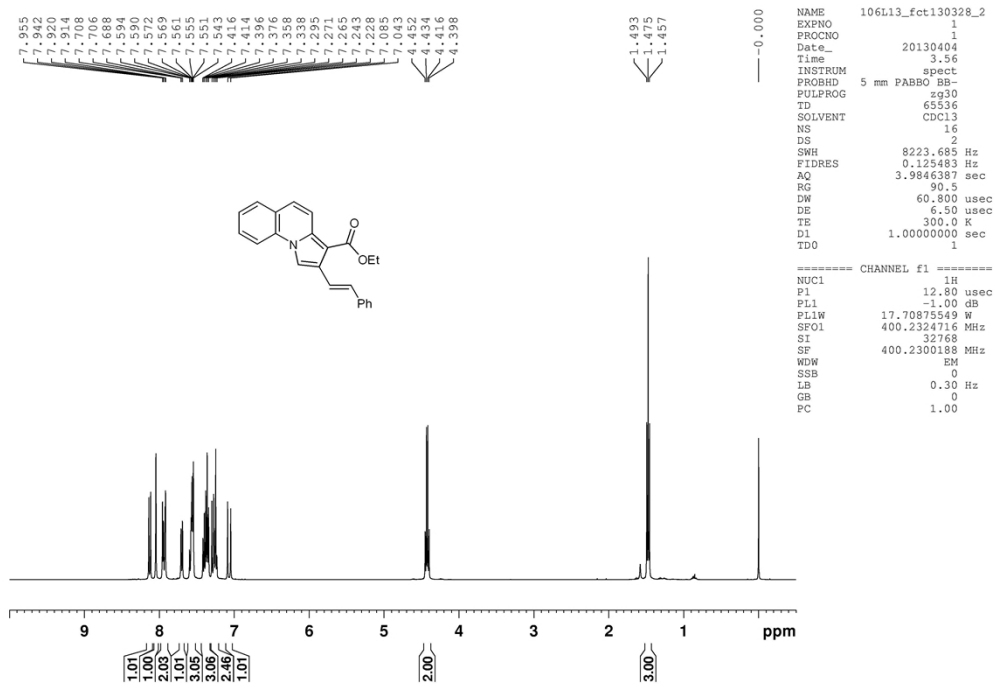


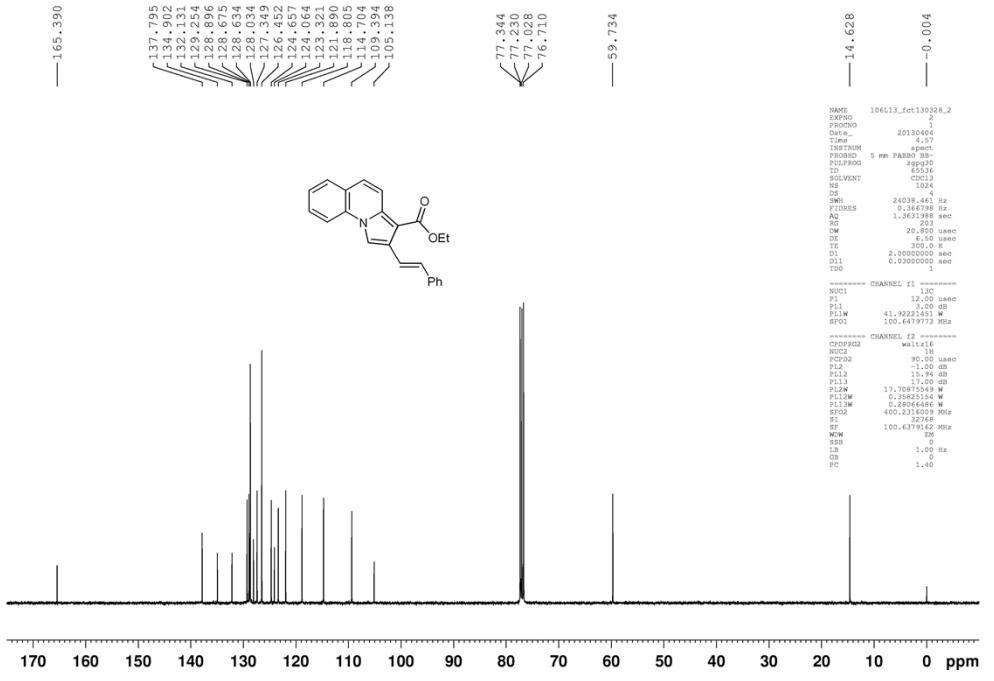

```
NAME          106L13_fct130407_1
EXPNO                          1
PROCNO                         1
Date_                   20130409
Time                       23.06
INSTRUM                    spect
PROBHD    5 mm PABBO BB-
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     8223.685 Hz
FIDRES                  0.125483 Hz
AQ                    3.9846387 sec
RG                            114
DW                       60.800 usec
DE                        6.50 usec
TE                        300.0 K
D1                   1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                       12.80 usec
PL1                      -1.00 dB
PL1W                17.70875549 W
SFO1                400.2324716 MHz
SI                         32768
SF                  400.2300121 MHz
WDW                           EM
SSB                            0
LB                        0.30 Hz
GB                             0
PC                          1.00
```



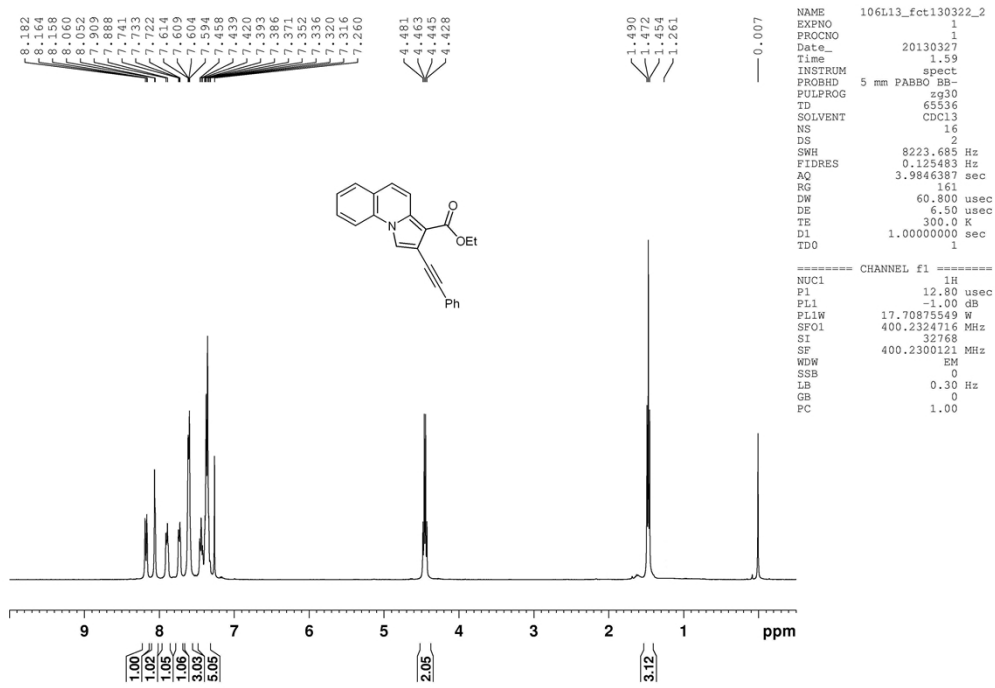

```
NAME          106L13_fct130407_1
EXPNO                          2
PROCNO                         1
Date_                   20130410
Time                       0.06
INSTRUM                    spect
PROBHD    5 mm PABBO BB-
PULPROG                  zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          1024
DS                             4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                    1.3631988 sec
RG                            203
DW                       20.800 usec
DE                        6.50 usec
TE                        300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                       12.00 usec
PL1                       3.00 dB
PL1W                41.92221451 W
SFO1                100.6479773 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                    90.00 usec
PL2                      -1.00 dB
PL12                     15.94 dB
PL13                     17.00 dB
PL2W                17.70875549 W
PL12W                0.35825154 W
PL13W                0.28066486 W
SFO2                400.2314009 MHz
SI                         32768
SF                  100.6379160 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                          1.40
```

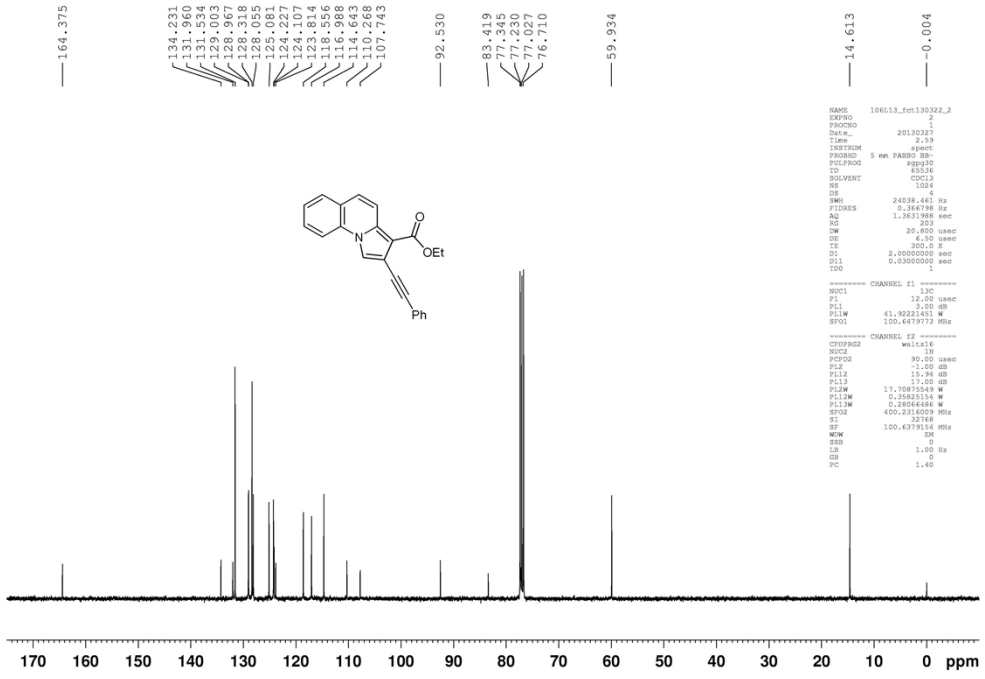
3p


7.955
7.942
7.920
7.914
7.708
7.706
7.688
7.594
7.590
7.572
7.569
7.561
7.555
7.551
7.543
7.416
7.414
7.396
7.376
7.358
7.338
7.295
7.271
7.265
7.243
7.228
7.085
7.043
4.452
4.434
4.416
4.398
1.493
1.475
1.457
-0.000
NAME 106L13_fct130328_2
EXPNO 1
PROCNO 1
Date_ 20130404
Time 3.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 -1.00 dB
PL1W 17.70875549 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300188 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OEt
N
Ph
9
8
7
6
5
4
3
2
1
ppm
1.01
1.00
2.03
1.01
3.05
3.06
2.46
1.01
2.00
3.00

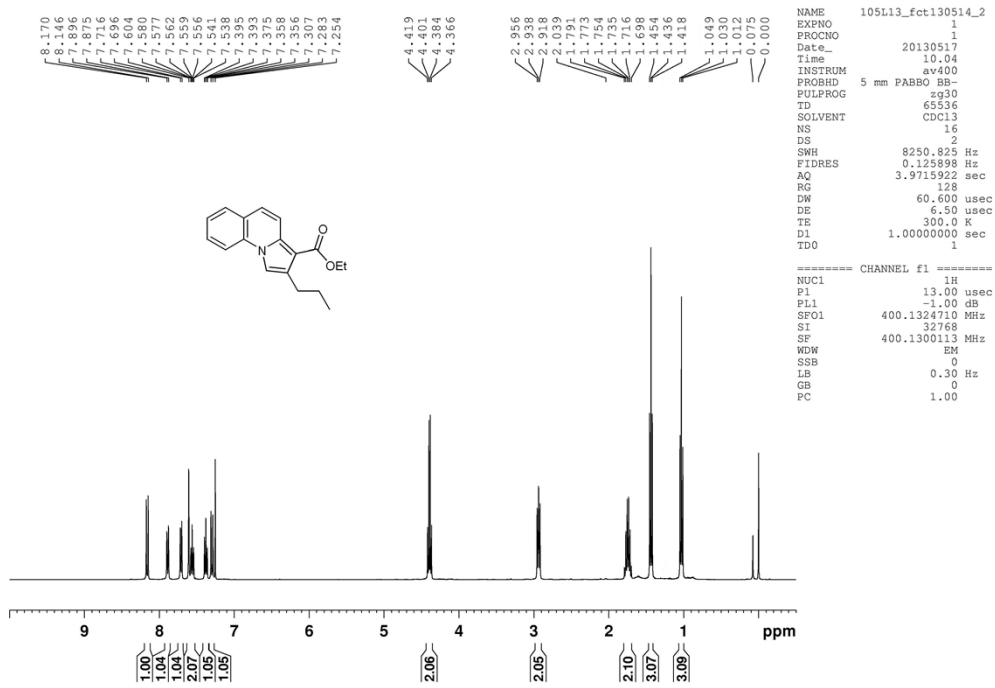

165.390
137.795
134.902
132.131
129.254
128.896
128.675
128.634
128.034
127.349
126.452
124.657
124.064
123.321
121.890
118.805
114.704
109.394
105.138
77.344
77.230
77.028
76.710
59.734
14.628
-0.004
NAME 106L13_fct130328_2
EXPNO 2
PROCNO 1
Date_ 20130404
Time 4.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.94 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.35825154 W
PL13W 0.28064486 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379162 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OEt
N
Ph
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

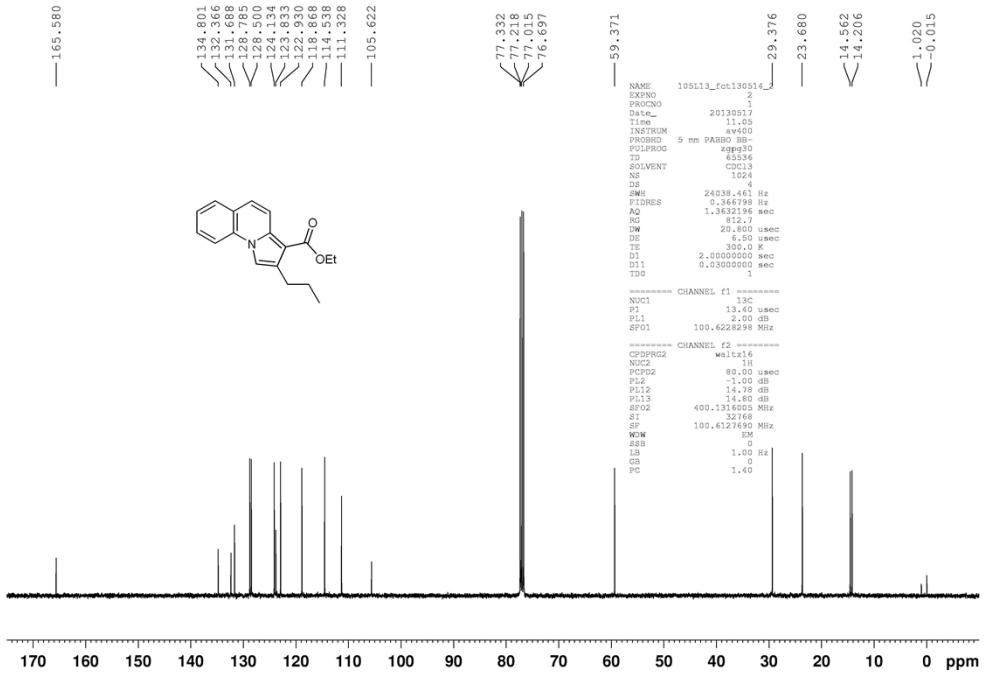
3q


8.182
8.164
8.158
8.060
8.052
7.909
7.888
7.741
7.733
7.722
7.614
7.609
7.604
7.594
7.458
7.439
7.420
7.393
7.386
7.371
7.352
7.336
7.320
7.316
7.260
4.481
4.463
4.445
4.428
1.490
1.472
1.454
1.261
0.007
OEt
Ph
ppm
1.00
1.02
1.05
1.06
3.03
5.05
2.05
3.12

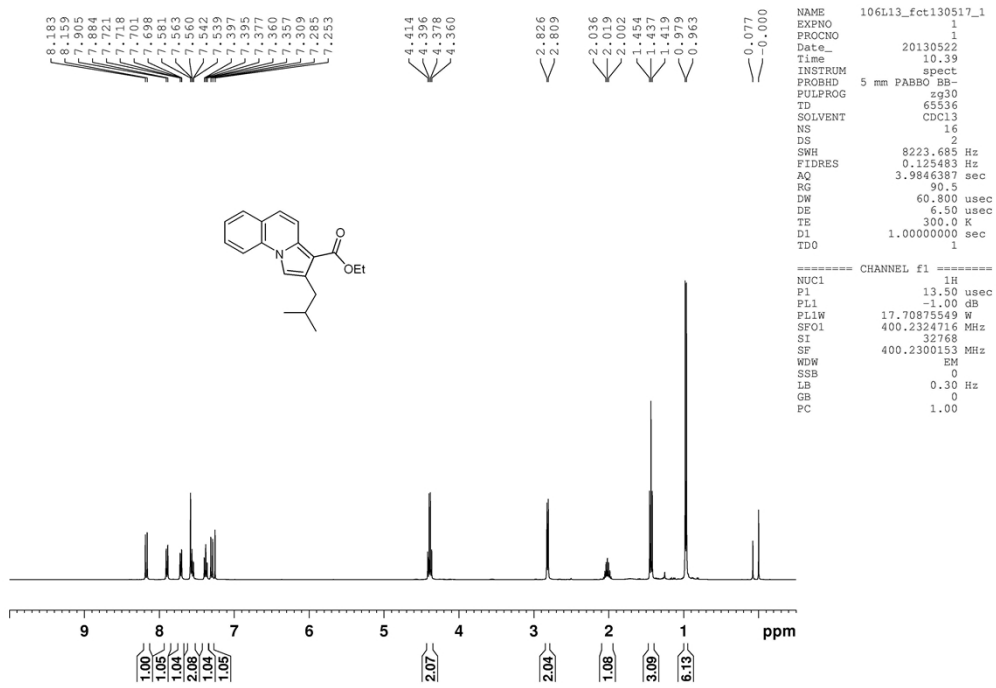

164.375
134.231
131.960
131.534
129.003
128.967
128.318
128.055
125.081
124.227
124.107
123.814
118.556
116.988
114.643
110.268
107.743
92.530
83.419
77.345
77.230
77.027
76.710
59.934
14.613
-0.004
OEt
Ph
ppm

3r

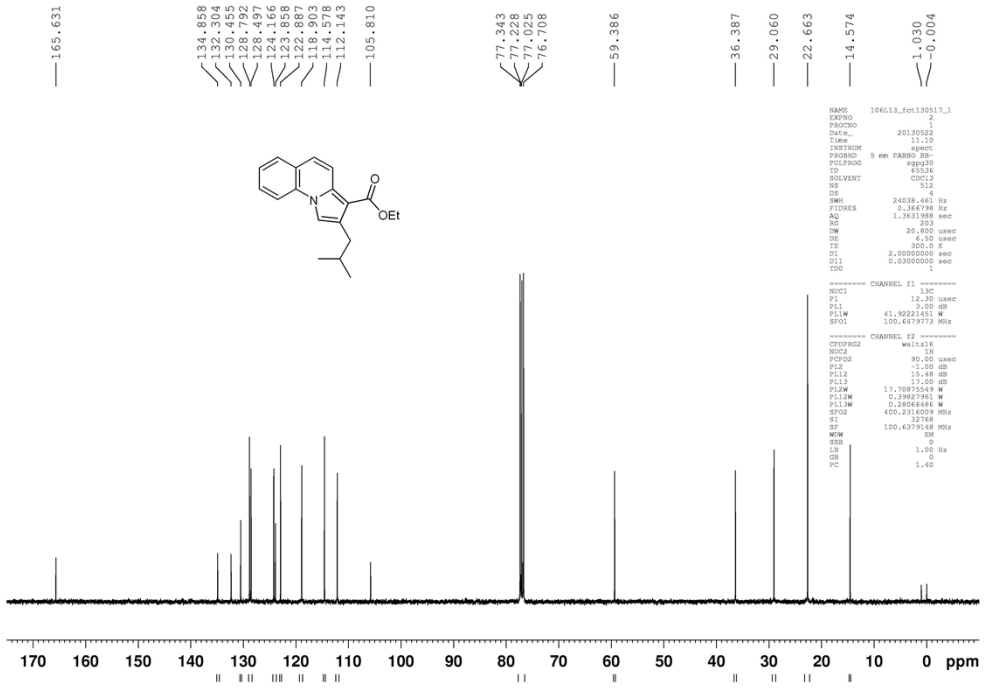

8.170
8.146
7.896
7.875
7.716
7.696
7.604
7.580
7.577
7.562
7.559
7.556
7.541
7.538
7.395
7.393
7.375
7.358
7.356
7.307
7.283
7.254
4.419
4.401
4.384
4.366
2.956
2.938
2.918
2.039
1.791
1.773
1.754
1.735
1.716
1.698
1.454
1.436
1.418
1.049
1.030
1.012
0.075
0.000
O
OEt
NAME 105L13_fct130514_2
EXPNO 1
PROCNO 1
Date_ 20130517
Time 10.04
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715922 sec
RG 128
DW 60.600 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
1.04
1.04
2.07
1.05
1.05
2.06
2.05
2.10
3.07
3.09


165.580
134.801
132.366
131.688
128.785
128.500
124.134
123.833
122.930
118.868
114.538
111.328
105.622
77.332
77.218
77.015
76.697
59.371
29.376
23.680
14.562
14.206
1.020
-0.015
O
OEt
NAME 105L13_fct130514_3
EXPNO 2
PROCNO 1
Date_ 20130517
Time 11.05
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 812.7
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.40 usec
PL1 2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.78 dB
PL13 14.80 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3s

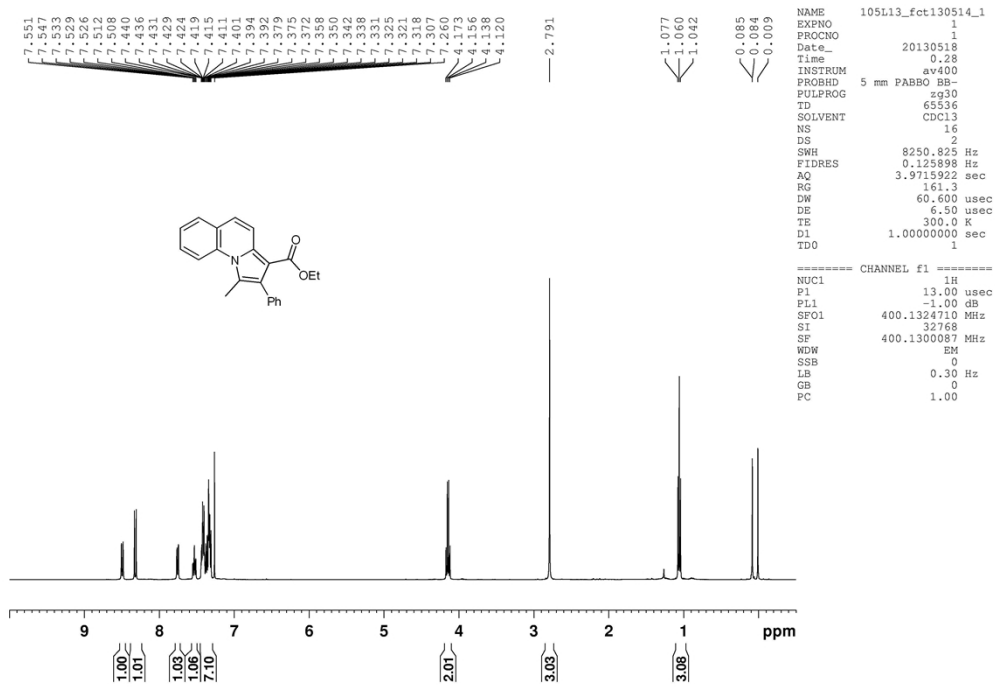


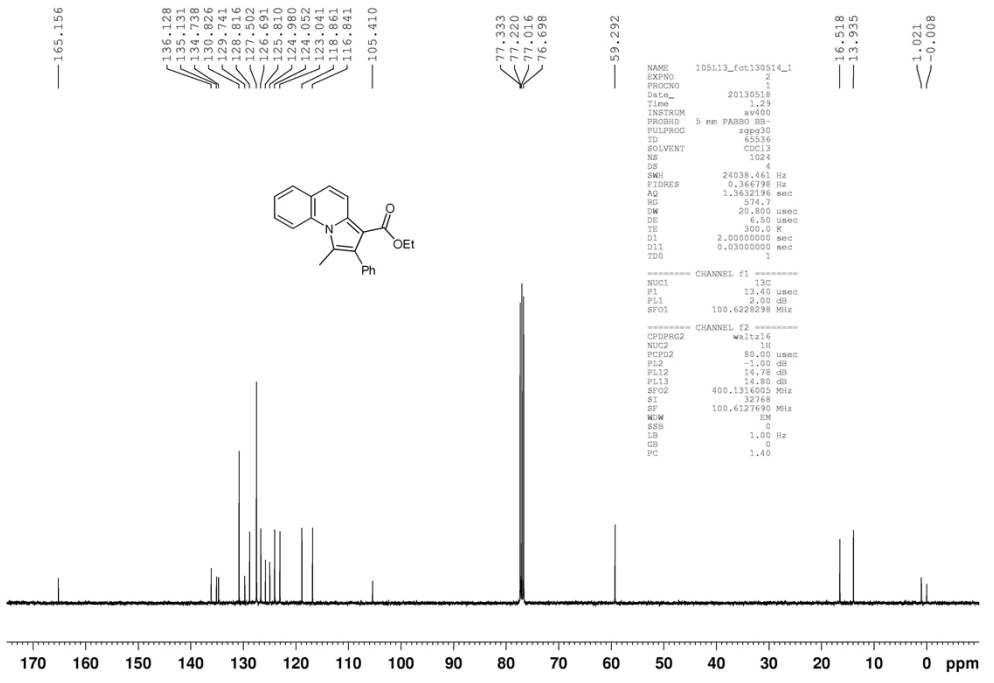

```
NAME        106L13_fct130517_1
EXPNO                        1
PROCNO                       1
Date_                 20130522
Time                     10.39
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                 3.9846387 sec
RG                        90.5
DW                    60.800 usec
DE                     6.50 usec
TE                     300.0 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                      13.50 usec
PL1                     -1.00 dB
PL1W               17.70875549 W
SFO1               400.2324716 MHz
SI                        32768
SF                 400.2300153 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```



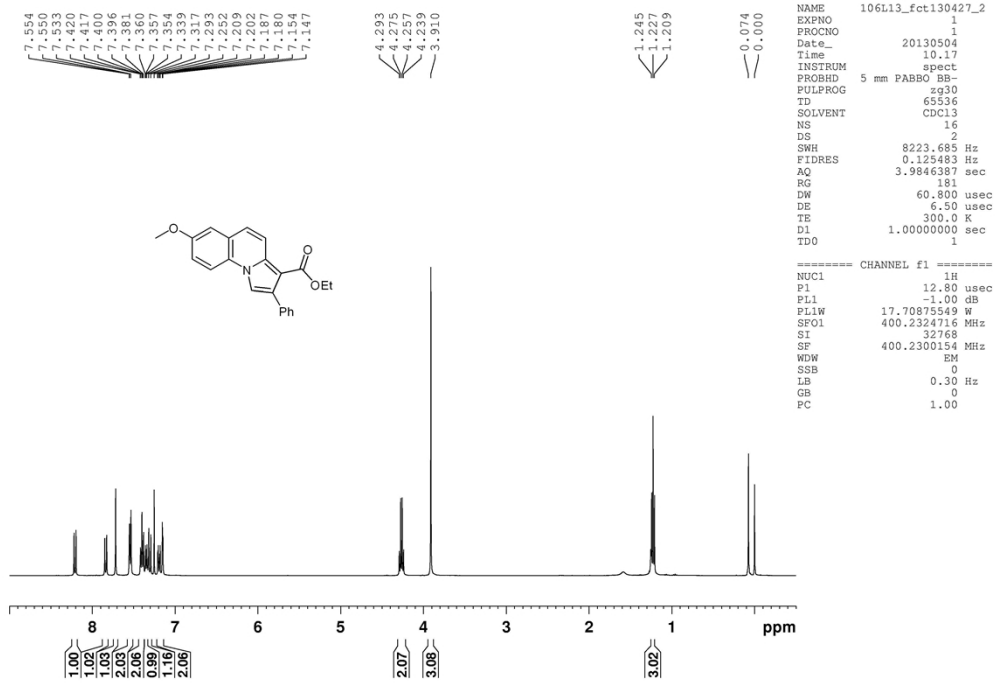

```
NAME        106L13_fct130517_1
EXPNO                        2
PROCNO                       1
Date_                 20130522
Time                     11.10
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        512
DS                          4
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                 1.3631988 sec
RG                        203
DW                    20.800 usec
DE                     6.50 usec
TE                     300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                      12.30 usec
PL1                      3.00 dB
PL1W              41.92221451 W
SFO1              100.6479773 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                  90.00 usec
PL2                     -1.00 dB
PL12                    15.48 dB
PL13                    17.00 dB
PL2W               17.70875549 W
PL12W               0.39827961 W
PL13W               0.28064486 W
SFO2               400.2316009 MHz
SI                        32768
SF                 100.6379148 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

3t


7.551
7.547
7.533
7.529
7.526
7.512
7.508
7.440
7.436
7.431
7.429
7.424
7.419
7.415
7.411
7.401
7.394
7.392
7.379
7.375
7.372
7.358
7.350
7.342
7.338
7.331
7.325
7.321
7.318
7.307
7.260
4.173
4.156
4.138
4.120
2.791
1.077
1.060
1.042
0.085
0.084
0.009
NAME 105L13_fct130514_1
EXPNO 1
PROCNO 1
Date_ 20130518
Time 0.28
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715922 sec
RG 161.3
DW 60.600 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OEt
Ph
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.03
1.06
7.10
2.01
3.03
3.08


165.156
136.128
135.131
134.738
130.826
129.741
128.816
127.502
126.691
125.810
124.980
124.052
123.041
118.861
116.841
105.410
77.333
77.220
77.016
76.698
59.292
16.518
13.935
1.021
0.008
NAME 105L13_fct130514_1
EXPNO 2
PROCNO 1
Date_ 20130518
Time 1.29
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 574.7
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.40 usec
PL1 2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.78 dB
PL13 14.80 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OEt
Ph
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3u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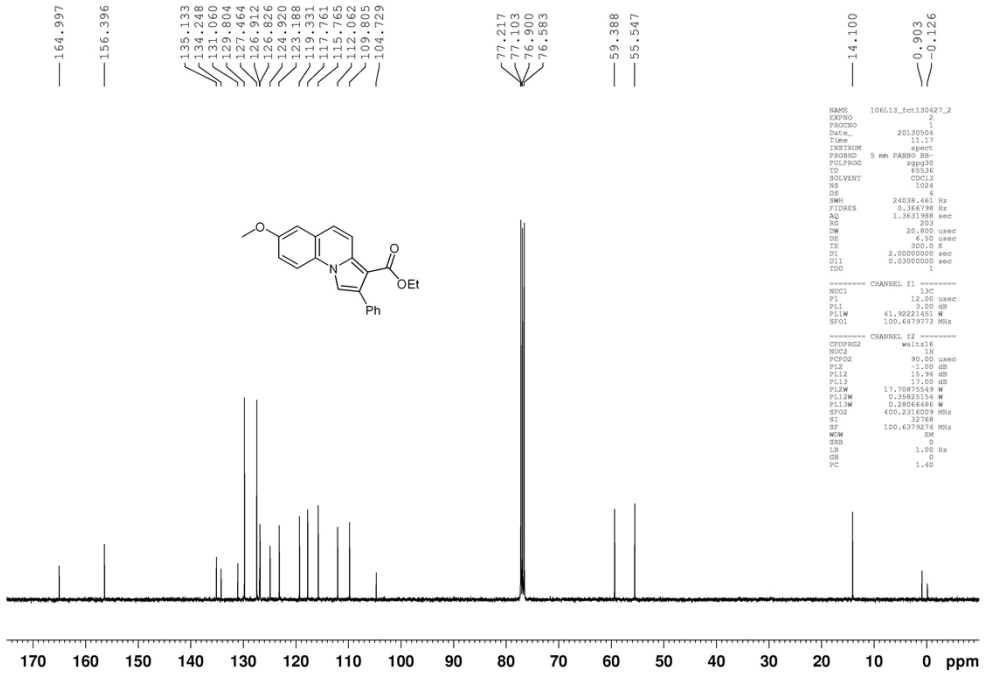
7.420
7.417
7.400
7.396
7.381
7.360
7.357
7.354
7.339
7.317
7.293
7.252
7.209
7.202
7.187
7.180
7.154
7.147
4.293
4.275
4.257
4.239
3.910
1.245
1.227
1.209
0.074
0.000
NAME 106L13_fct130427_2
EXPNO 1
PROCNO 1
Date_ 20130504
Time 10.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 -1.00 dB
PL1W 17.70875549 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
OEt
Ph
8
7
6
5
4
3
2
1
ppm
1.00
1.02
1.03
2.03
2.06
0.99
1.16
2.06
2.07
3.08
3.02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156.396
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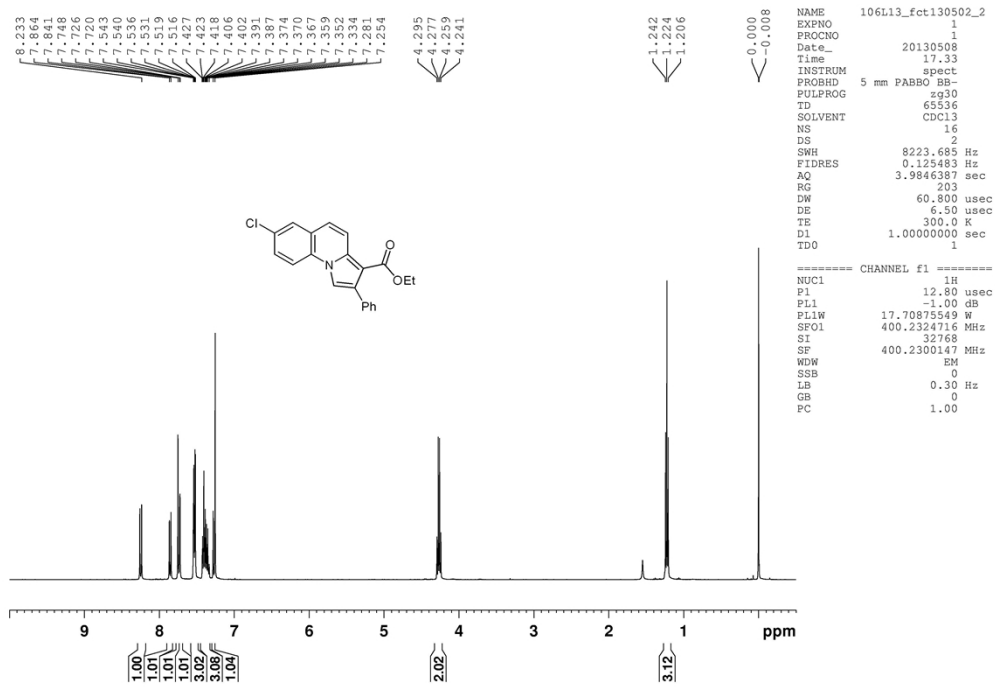
134.248
131.060
129.804
127.464
126.912
126.826
124.920
123.188
119.331
117.761
115.765
112.062
109.805
104.729
77.217
77.103
76.900
76.583
59.388
55.547
14.100
0.903
-0.126
NAME 106L13_fct130427_2
EXPNO 2
PROCNO 1
Date_ 20130504
Time 11.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.94 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.35825154 W
PL13W 0.28064486 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379276 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
N
OEt
Ph
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3v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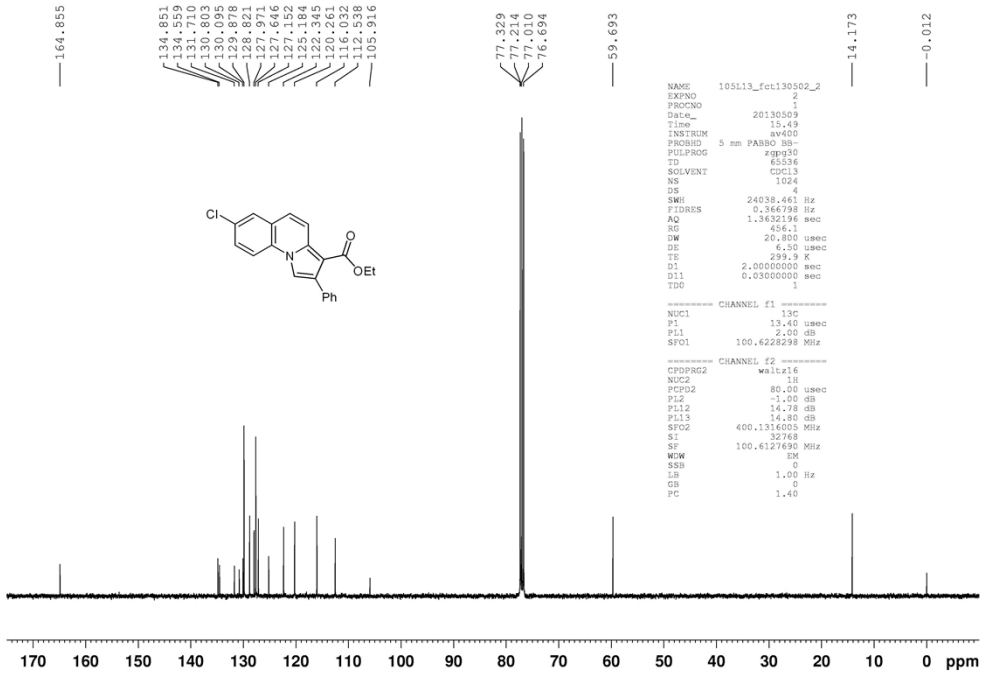
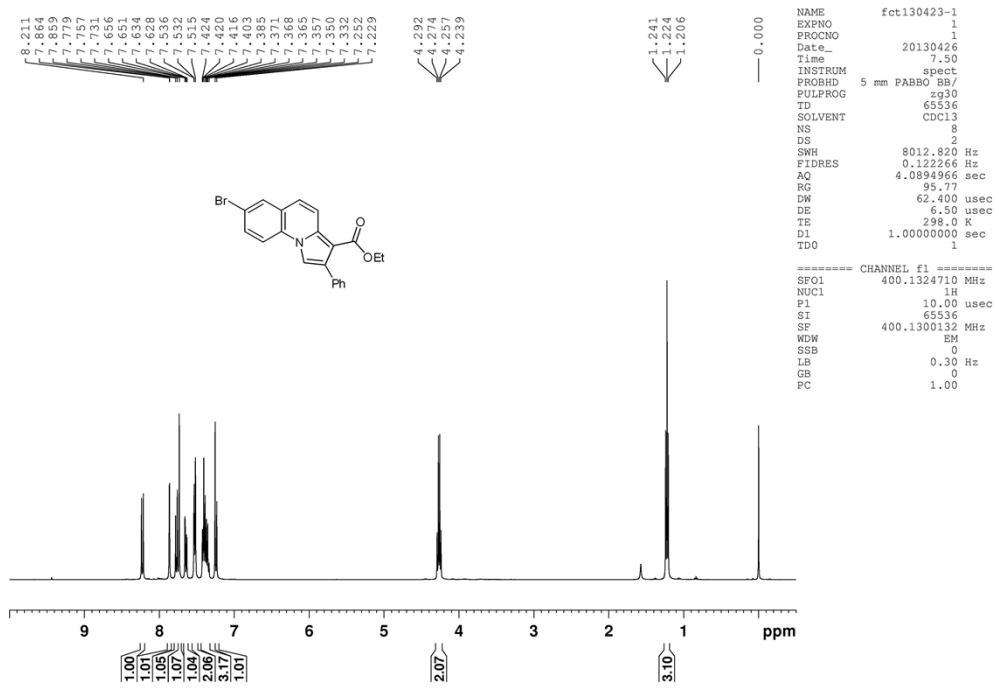
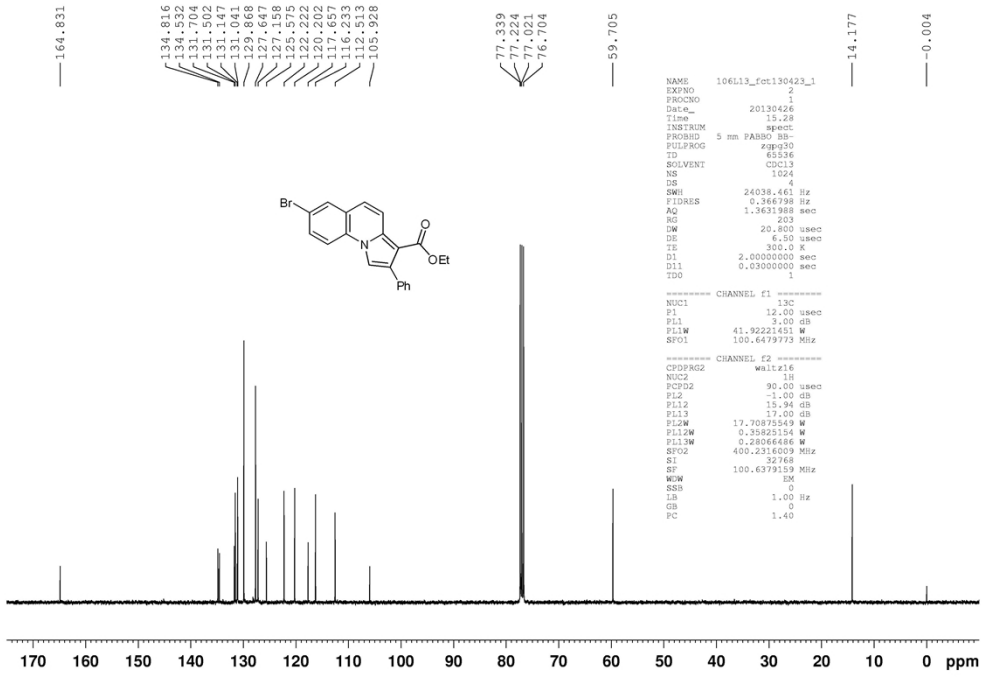
7.864
7.841
7.748
7.726
7.720
7.543
7.540
7.536
7.531
7.519
7.516
7.427
7.423
7.418
7.406
7.402
7.391
7.387
7.374
7.370
7.367
7.359
7.352
7.334
7.281
7.254
4.295
4.277
4.259
4.241
1.242
1.224
1.206
0.000
-0.008
NAME 106L13_fct130502_2
EXPNO 1
PROCNO 1
Date_ 20130508
Time 17.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 -1.00 dB
PL1W 17.70875549 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300147 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cl
N
O
OEt
Ph
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.01
1.01
3.02
3.08
1.04
2.02
3.12

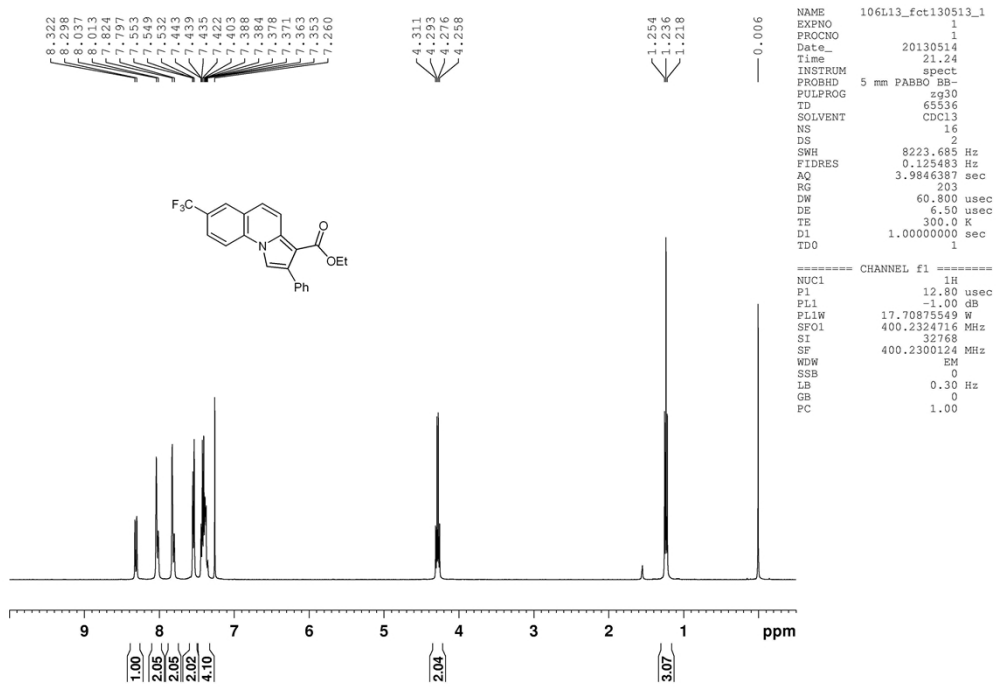

164.855
134.851
134.559
131.710
130.803
130.095
129.878
128.821
127.971
127.646
127.152
125.184
122.345
120.261
116.632
112.338
105.916
77.329
77.214
77.010
76.694
59.693
14.173
-0.012
NAME 105L13_fct130502_2
EXPNO 2
PROCNO 1
Date_ 20130509
Time 15.49
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 456.1
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.40 usec
PL1 2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.78 dB
PL13 14.80 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cl
N
O
OEt
Ph
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3w


8.211
7.864
7.859
7.779
7.757
7.731
7.656
7.651
7.634
7.628
7.536
7.532
7.515
7.424
7.420
7.416
7.403
7.385
7.371
7.368
7.365
7.357
7.350
7.332
7.252
7.229
4.292
4.274
4.257
4.239
1.241
1.224
1.206
0.000
Br
O
OEt
N
Ph
NAME fct130423-1
EXPNO 1
PROCNO 1
Date_ 20130426
Time 7.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 95.77
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300132 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.05
1.07
1.04
2.06
3.17
1.01
2.07
3.10

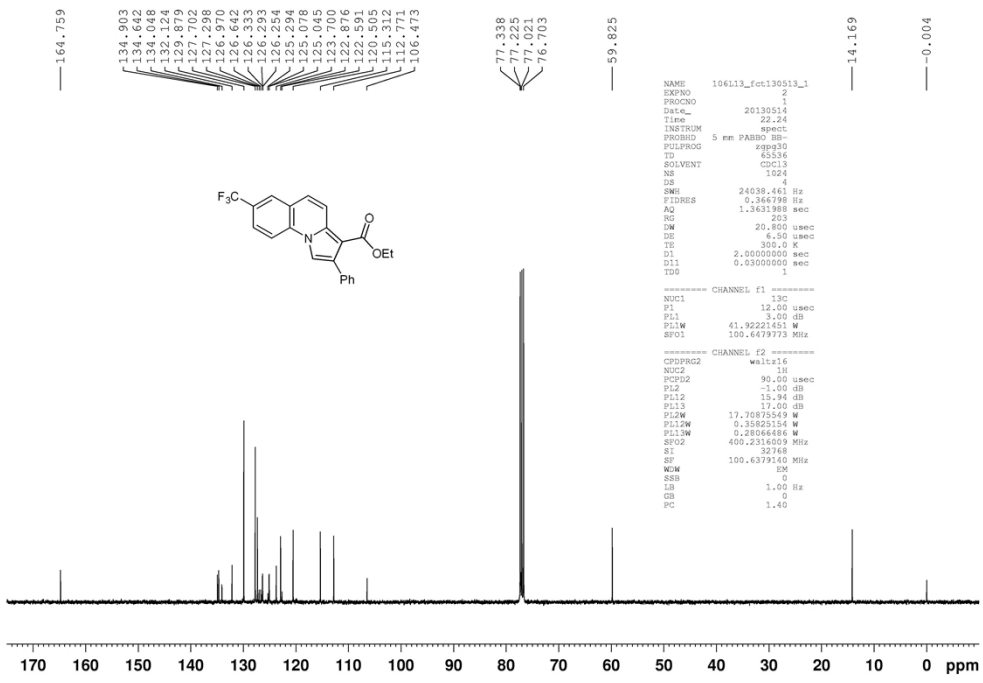

164.831
134.816
134.532
131.704
131.502
131.147
131.041
129.868
127.647
127.158
125.575
122.222
120.202
117.657
116.233
112.513
105.928
77.339
77.224
77.021
76.704
59.705
14.177
-0.004
Br
O
OEt
N
Ph
NAME 106L13_fct130423_1
EXPNO 2
PROCNO 1
Date_ 20130426
Time 15.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.94 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.35825154 W
PL13W 0.28066486 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379159 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3x



NAME 106L13_fct130513_1
EXPNO 1
PROCNO 1
Date_ 20130514
Time 21.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 -1.00 dB
PL1W 17.70875549 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

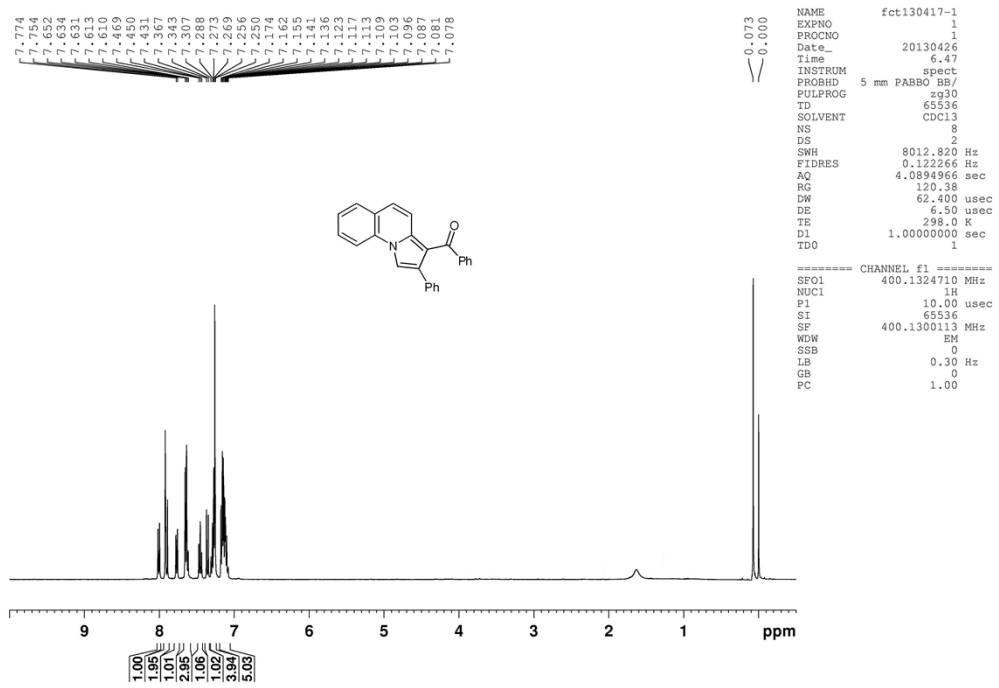


NAME 106L13_fct130513_1
EXPNO 2
PROCNO 1
Date_ 20130514
Time 22.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
PL1W 41.92221451 W
SFO1 100.6479773 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.94 dB
PL13 17.00 dB
PL2W 17.70875549 W
PL12W 0.35825154 W
PL13W 0.28066486 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3y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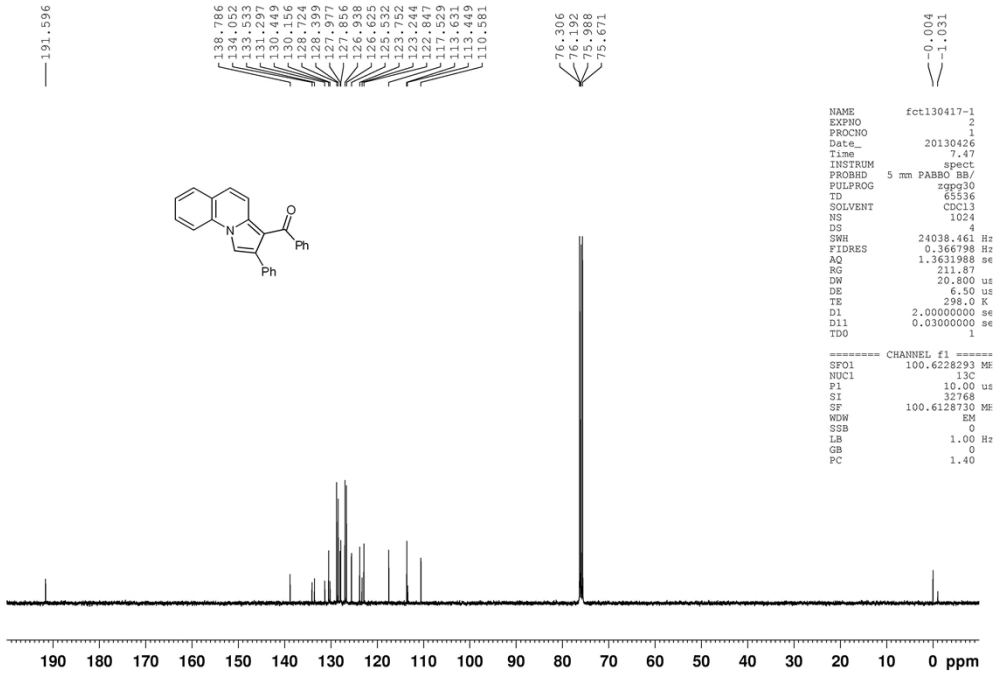
7.634
7.631
7.613
7.610
7.469
7.450
7.431
7.367
7.343
7.307
7.288
7.273
7.269
7.256
7.250
7.174
7.162
7.155
7.141
7.136
7.123
7.117
7.113
7.109
7.103
7.096
7.087
7.081
7.078
0.073
0.000
NAME fct130417-1
EXPNO 1
PROCNO 1
Date_ 20130426
Time 6.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 120.38
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
Ph
Ph
9
8
7
6
5
4
3
2
1
ppm
1.00
1.95
1.01
2.95
1.06
1.02
3.94
5.03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133.533
131.297
130.449
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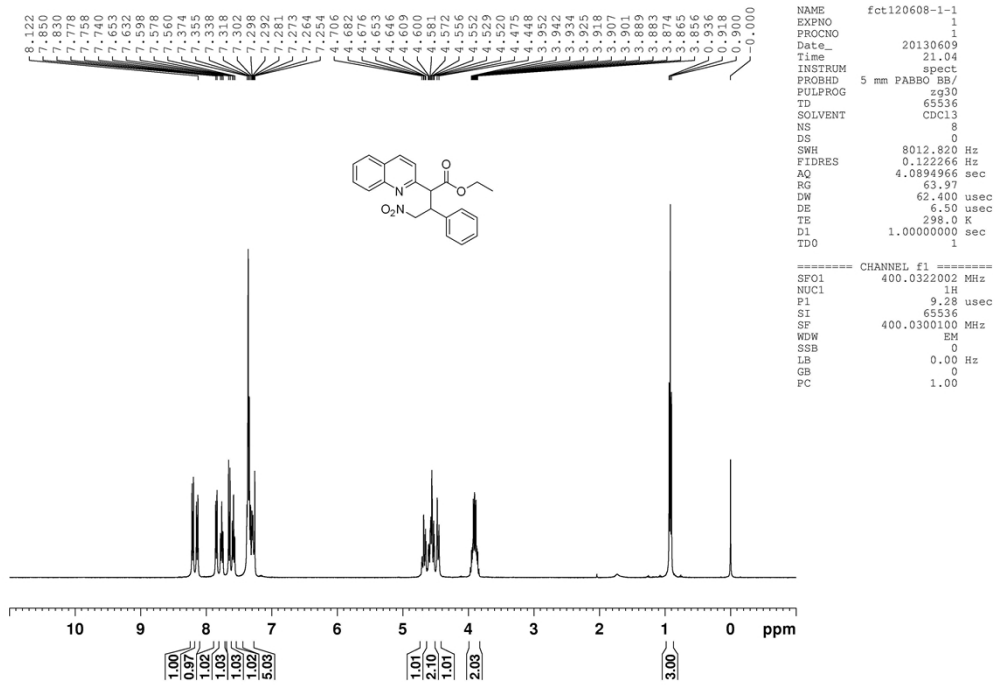
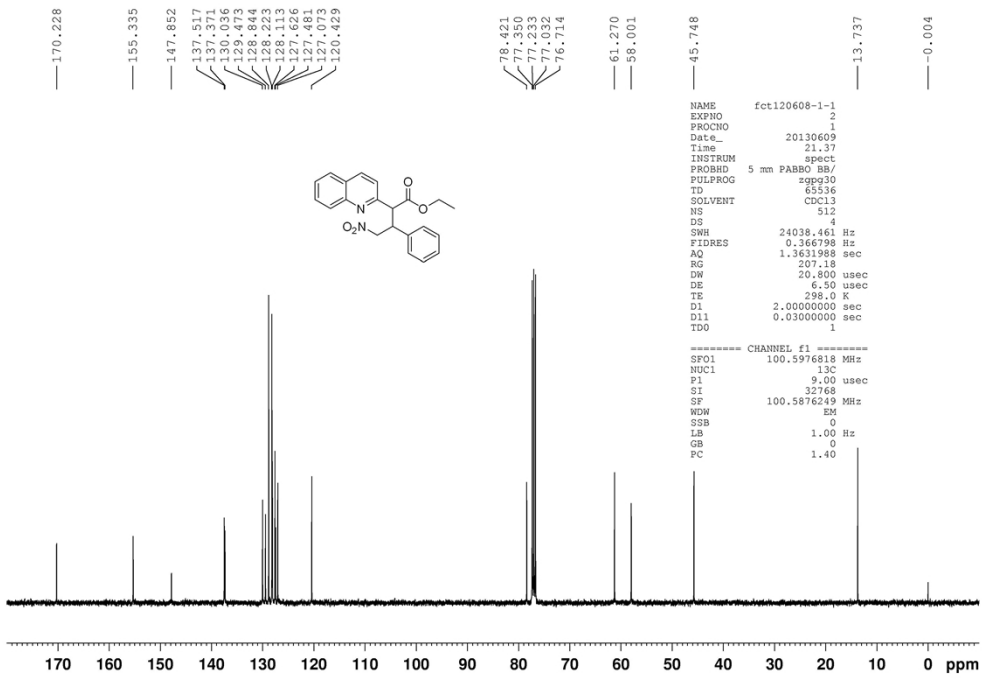
128.724
128.349
127.977
127.856
127.938
126.625
126.532
123.752
123.244
122.847
117.529
113.631
113.449
110.581
76.306
76.192
75.988
75.671
-0.004
-1.031
NAME fct130417-1
EXPNO 2
PROCNO 1
Date_ 20130426
Time 7.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 211.87
DW 20.800 us
DE 6.50 us
TE 298.0 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228293 ME
NUC1 13C
P1 10.00 us
SI 32768
SF 100.6128730 ME
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
Ph
Ph
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

4a


8.122
7.850
7.830
7.778
7.758
7.740
7.653
7.632
7.598
7.578
7.560
7.374
7.355
7.338
7.318
7.302
7.298
7.292
7.281
7.273
7.264
7.254
4.706
4.682
4.676
4.653
4.646
4.609
4.600
4.581
4.572
4.556
4.552
4.529
4.520
4.475
4.448
3.952
3.942
3.934
3.925
3.918
3.907
3.901
3.889
3.883
3.874
3.865
3.856
0.936
0.918
0.900
-0.000
NAME fct120608-1-1
EXPNO 1
PROCNO 1
Date_ 20130609
Time 21.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 63.97
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.0322002 MHz
NUC1 1H
P1 9.28 usec
SI 65536
SF 400.0300100 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O2N
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00 0.97 1.02 1.03 1.03 1.02 5.03
1.01 2.10 1.01 2.03
3.00


170.228
155.335
147.852
137.517
137.371
130.036
129.473
128.844
128.223
128.113
127.626
127.481
127.073
120.429
78.421
77.350
77.233
77.032
76.714
61.270
58.001
45.748
13.737
-0.004
NAME fct120608-1-1
EXPNO 2
PROCNO 1
Date_ 20130609
Time 21.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 207.18
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.5976818 MHz
NUC1 13C
P1 9.00 usec
SI 32768
SF 100.5876249 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O2N
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

5. Determination of nitrous oxide (N_2O)

Preparation of the sample gas

A two-neck tube was charged with **1a** (640 mg, 3 mmol), **2a** (298 mg, 2 mmol), $CeCl_3 \cdot 7H_2O$ (74 mg, 0.2 mmol).The tube was sealed with a rubber stopper. The tube was evacuated and flushed with nitrogen three times via a three-way valve. Then 5 mL of ethanol was added to the reaction system by an injection syringe. The reaction mixture was stirred at room temperature for 5h.

The analysis of the sample gas

Then 100 μL gas sample was collected from the tube by a gas tight syringe. The sample was injected to GC-MS system without undue delay.

The analysis was performed on a GC-MS system (Trace GC/ISQ MS). The mass spectrometer (MS) was used in selected ion monitoring (SIM) mode (8 scans s^{-1}) with electron ionization (70 eV). The selected ions were m/z 22 and m/z 44 for CO_2 and the selected ions were m/z 30 and m/z 44 for N_2O.

Results and discussion

The result was shown in **Fig 1**, **Fig 2** and **Fig 3**.

CO_2 and N_2O have the same base peak at m/z 44, but they have different fragments. CO_2 was analyzed at m/z 22, (CO_2^{2+}), an ion present in 1.4% of the base ion. As showed in **Fig 2**, the relative abundance of ion m/z 22 present in 10^{-4} of the base ion m/z 44. Most of m/z 44 ions may be derived from N_2O. To confirm the result, ion m/z 30 which was the next largest peak of the N_2O, was monitored (in **Fig 1**). Ion m/z 30 was presented in 15% of the base ion m/z 44, it was consistent with the mass spectrogram of N_2O.

Based on the above results, we verify that N_2O gas exist in the reaction system.

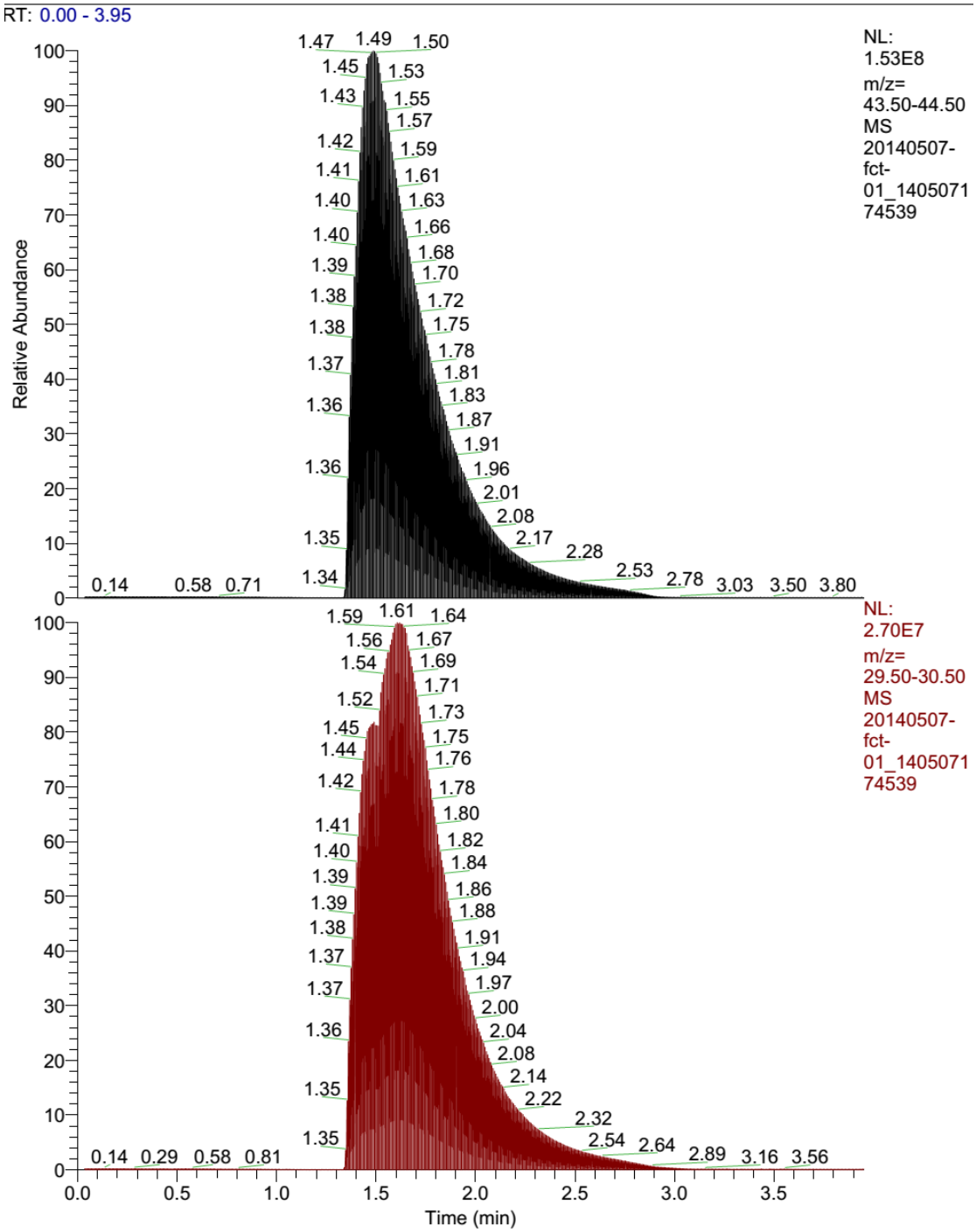


Fig. 1 The SIM mass spectrogram of the sample gas was recorded by m/z 30 and m/z 44.

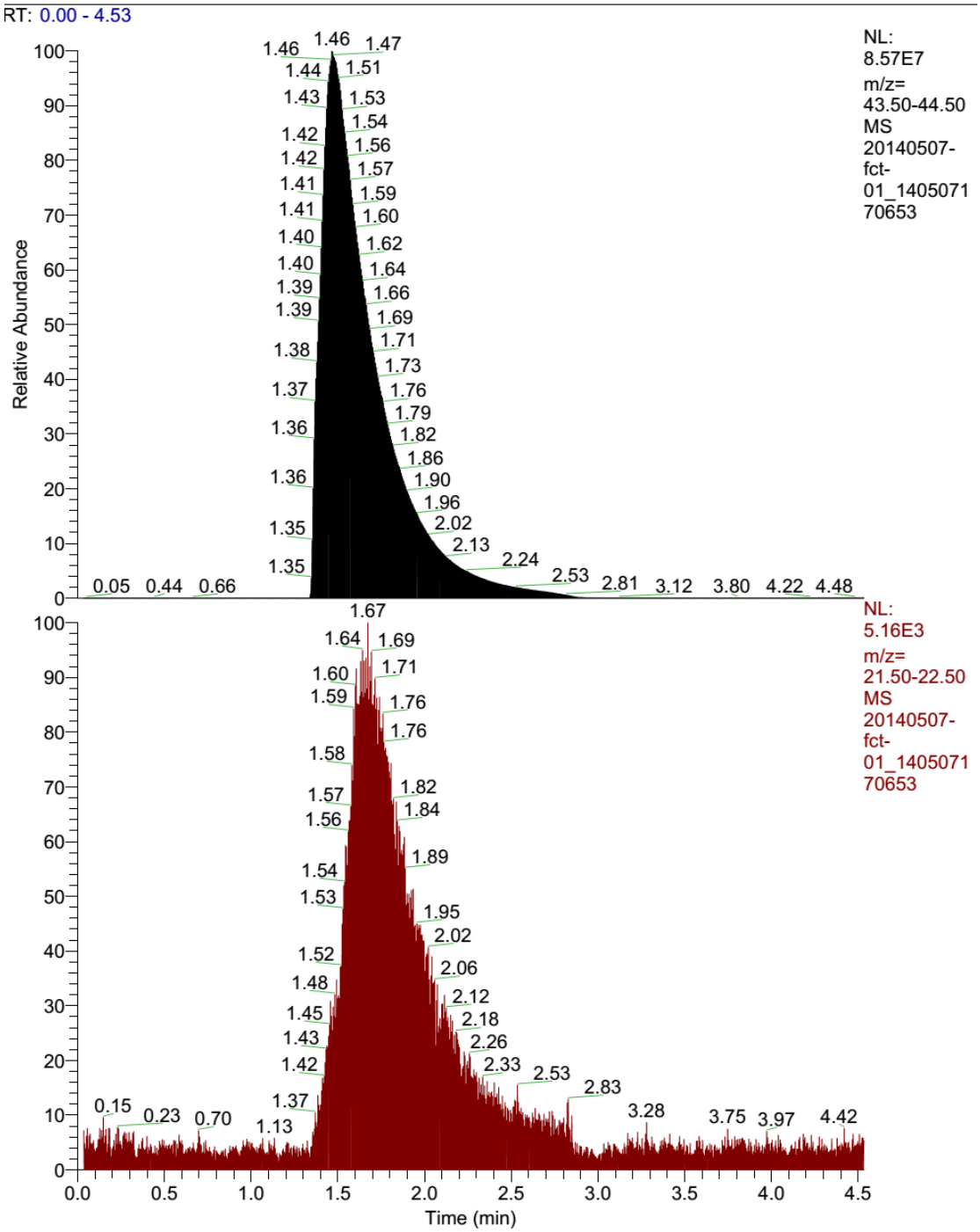


Fig. 2 The SIM mass spectrogram of the sample gas was recorded by m/z 22 and m/z 44.

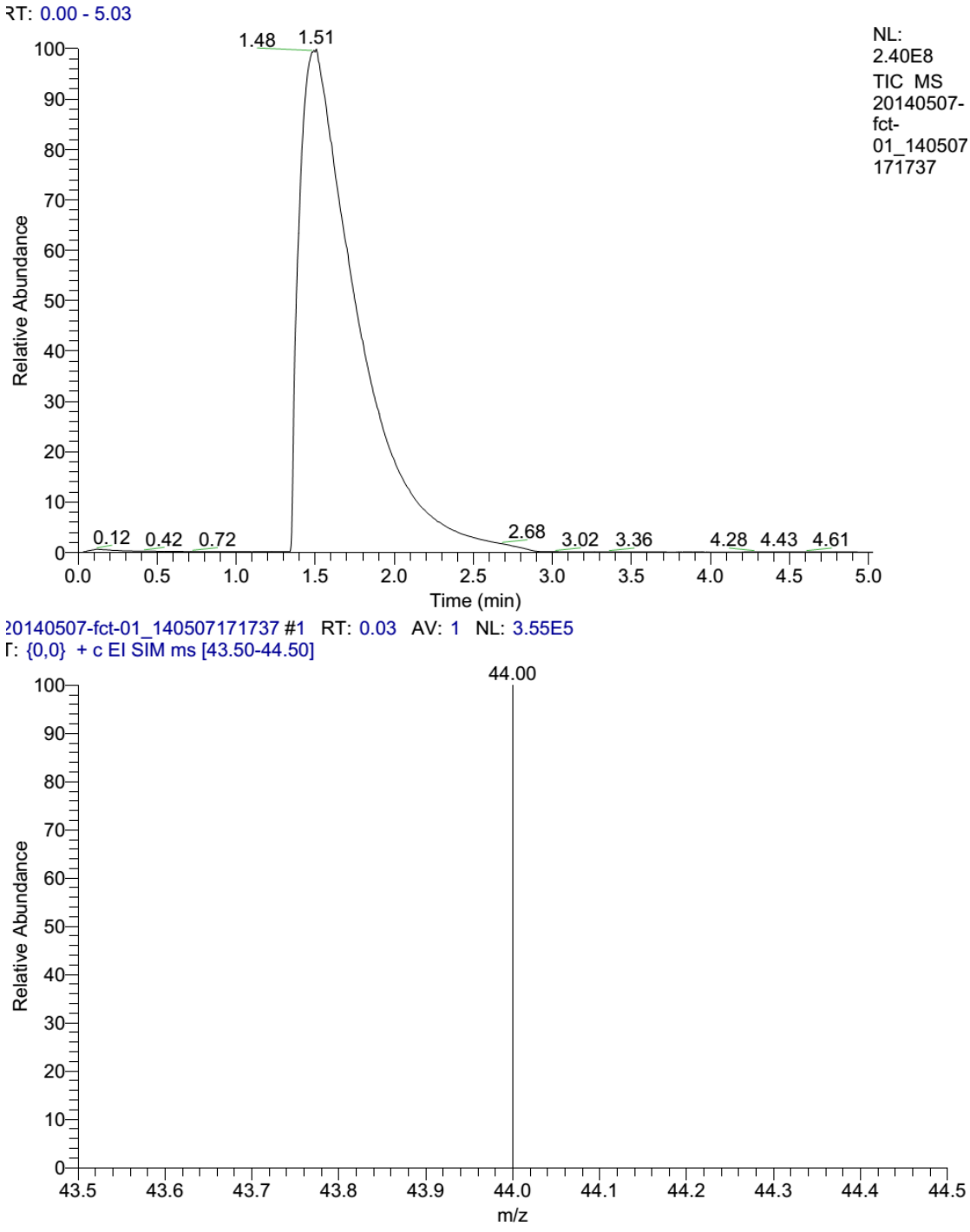


Fig. 3 The SIM chromatograms of the sample gas was recorded by m/z 44.