Supporting Information

The Skraup and Doebner-von Miller Quinoline Synthesis Revisited — Reversal of the Regiochemistry for γ-Aryl-β, γ-Unsaturated α-Ketoesters

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1. Experimental Procedures

1.1 Synthesis of γ-aryl-β, γ-unsaturated α-ketoesters **2.**[1-2]

To a solution of pyruvic acid (20 mL, 0.286 mol), substituted or unsubstituted benzaldehyde (0.286 mol) in 15 mL of methanol stirring in an ice bath, a solution of potassium hydrate (24 g, 0.429 mol) in 75 mL of methanol was added. The first 50 mL of the base solution was added slowly and the reaction temperature was kept below 25 °C. The ice bath was then removed and the rest of the base solution was added quickly. Yellow precipitate was formed at once. The reaction temperature was kept at 30 °C for 1 h and then at zero overnight. The yellow crystals were filtered off and washed twice with cold methanol and once with ether. The yellow crystals were air dried to afford the potassium salt.[1]

24 mL of acetyl chloride was added to 140 mL of methanol in 250 mL 3-necked round bottom at zero to generate hydrochloric acid. The potassium salt (0.1 mol) was added and the mixture stirred for 30 min before the ice bath was removed. After 2 h the mixture was refluxed overnight. The reaction mixture was evaporated and the yellow solid was extracted with 50 mL of water and two times with 50 mL of dichloromethane. The combined organic phases were washed with 50 mL of saturated sodium carbonate and then 50 mL of water. The organic phase was dried with anhydrous potassium sulfate and evaporated. The yellow crystals of pure γ-aryl-β, γ-unsaturated α-ketoesters **2** were obtained via recrystallization from methanol or ethanol.[2]

(3*E*)-2-Oxo-4-phenyl-but-3-enoic acid methyl ester (2a): 37.0% yield in two steps; yellow crystal; mp = 68-70 °C (literature.[2] 70-71 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (d, *J* = 16.1 Hz, 1H), 7.60 (dd, *J* = 9.5, 1.9 Hz, 2H), 7.43-7.31 (m, 4H), 3.91 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.4, 162.5, 148.6, 134.0, 131.7, 129.1, 129.1, 120.5, 53.0.

(3*E*)-4-(4-Methoxyphenyl)-2-oxobut-3-enoic acid methyl ester (2b): 44.0% yield in two steps; yellow crystal; mp = 99-100 °C (literature.[3] 109 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 (d, *J* = 16.0 Hz, 1H), 7.56 (d, *J* = 8.7 Hz, 2H), 7.21 (d, *J* = 16.0 Hz, 1H), 6.90 (d, *J* = 8.7 Hz, 2H), 3.89 (s, 3H), 3.82 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.2, 162.8, 162.7, 148.5, 131.1, 126.8, 118.0, 114.6, 55.5, 53.0.

(3*E*)-4-(4-Methylphenyl)-2-oxobut-3-enoic acid methyl ester (2c): 41.0% yield in two steps; yellow crystal; mp = 81-83 °C (literature.[4] 81 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 16.1 Hz, 1H), 7.44 (d, *J* = 8.1 Hz, 2H), 7.24 (d, *J* = 16.1 Hz, 1H), 7.14 (d, *J* = 8.1 Hz, 2H), 3.86 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.4, 162.6, 148.7, 142.5, 131.2, 129.8, 129.1, 119.4, 53.0, 21.6.

(3*E*)-4-(4-Chlorophenyl)-2-oxobut-3-enoic acid ethyl ester (2d): 31.0% yield in two steps; yellow crystal; mp = 76-77 °C (literature.[5] 77.5-78.5 °C); ^{1}H NMR (300 MHz,

$CDCl_3$) δ 7.77 (d, *J* = 16.1 Hz, 1H), 7.54 (d, *J* = 8.5 Hz, 2H), 7.39 -7.33 (m, 3H), 4.37 (q, *J* = 7.1 Hz, 2H), 1.39 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.6, 162.0, 146.7, 137.6, 132.5, 130.1, 129.4, 121.0, 62.6, 14.1.

(3*E*)-4-(4-Nitrophenyl)-2-oxobut-3-enoic acid methyl ester (2e): 23.0% yield in two steps; yellow crystal; mp = 185-187 °C (literature.[6] 182.5-183.5 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.30 (d, *J* = 8.8 Hz, 2H), 7.90 (d, *J* = 16.2 Hz, 1H), 7.81 (d, *J* = 8.8 Hz, 2H), 7.52 (d, *J* = 16.2 Hz, 1H), 3.97 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.7, 161.9, 149.2, 145.0, 139.9, 129.6, 124.3, 123.8, 53.4.

1.2 Procedures for the discovery of the reaction

A solution of 2,3-dimethylaniline (**1a**, 0.2 mmol)**,** (3*E*)-2-oxo-4-phenylbut-3-enoate methyl ester (**2a**, 0.2 mmol) and $Hf(OTf)_4$ (0.02 mmol) in 2mL of dichloromethane was stirred at room temperature for 48 h. Then, the solution was diluted with 20 mL of CH_2Cl_2, washed with 5 mL of saturated aqueous $NaHCO_3$ and then 5 mL of water, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give methyl 7,8-dimethyl-2-phenylquinoline-4-carboxylate (**4a**, 44%) and methyl 7,8-dimethyl-4-phenylquinoline-2-carboxylate (**3a**, 18%).

Methyl 7,8-dimethyl-2-phenylquinoline-4-carboxylate (4a): 44.0% yield; colorless crystal; mp = 133-134 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.46 (d, *J* = 8.7 Hz, 1H), 8.34 (s, 1H), 8.31-8.27 (m, 2H), 7.57-7.42 (m, 4H), 4.06 (s, 3H), 2.88 (s, 3H), 2.53 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.3, 154.6, 148.0, 139.2, 137.7, 135.8, 135.3, 130.7, 129.5, 128.8, 127.4, 122.3, 122.1, 118.5, 52.6, 20.7, 13.7; FTIR (KBr) 2949, 1724, 1432, 1254, 1201, 761, 695 cm^{-1}; HRMS (FAB) Calcd. For $(M + H)^+$ $C_{19}H_{18}NO_2$: 292.1332, Found: 292.1335; Anal. Calcd. For $C_{19}H_{17}NO_2$: C, 78.33; H, 5.88; N, 4.81. Found: C, 78.11; H, 5.89; N, 4.79.

Methyl 7,8-dimethyl-4-phenylquinoline-2-carboxylate (3a): 18.0% yield; colorless crystal; mp = 119-120 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.70 (d, *J* = 8.6 Hz, 1H), 7.51 (s, br, 5H), 7.39 (d, *J* = 8.6 Hz, 1H), 4.07 (s, 3H), 2.90 (s, 3H), 2.52 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.5, 149.7, 147.2, 146.1, 138.1, 137.9, 136.2, 131.5, 129.6, 128.6, 128.5, 126.2, 122.6, 120.2, 52.9, 20.7, 13.8; FTIR (KBr) 2951, 1718, 1444, 1248, 1131, 765, 706 cm^{-1}; HRMS (FAB) Calcd. For $(M + H)^+$ $C_{19}H_{18}NO_2$: 292.1332, Found: 292.1332; Anal. Calcd. For $C_{19}H_{17}NO_2$: C, 78.33; H, 5.88; N, 4.81. Found: C, 78.19; H, 5.92; N, 4.79.

A mixture of **1a** (0.2 mmol) and **2a** (0.2 mmol) in 2 mL of 37% aqueous hydrochloric acid was stirred at room temperature for 24 h. The solution was added saturated aqueous $NaHCO_3$ until pH = 7, and then was extracted with 20 mL of CH_2Cl_2. The organic part

was dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (trace, <2%) and **3a** (trace, <2%).

Hydrogen chloride passed through the mixture of **1a** (0.2 mmol) and **2a** (0.2 mmol) in 2 mL of CH_2Cl_2 for 10 min. Then the solution was stirred at room temperature for 24 h. The solution was added saturated aqueous $NaHCO_3$ until pH = 7, and then was extracted with 20 mL of CH_2Cl_2. The organic part was dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (trace, <2%) and **3a** (trace, <2%).

Hydrogen chloride passed through the mixture of **1a** (0.2 mmol) and **2a** (0.2 mmol) in 2 mL of CH_2Cl_2 for 10 min. Then the solution was refluxed for 24 h. The solution was added saturated aqueous $NaHCO_3$ until pH = 7, and then was extracted with 20 mL of CH_2Cl_2. The organic part was dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (trace, <2%) and **3a** (trace, <2%).

Hydrogen chloride passed through the mixture of **1a** (0.2 mmol) and **2a** (0.2 mmol) in 2 mL of PhMe for 10 min. Then the solution was refluxed for 24 h. The solution was added saturated aqueous $NaHCO_3$ until pH = 7, and then was extracted with 20 mL of CH_2Cl_2. The organic part was dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (trace, <2%) and **3a** (trace, <2%).

A mixture of **1a** (0.2 mmol), **2a** (0.2 mmol) and H_2SO_4 (0.2 mmol) in 2 mL of CH_2Cl_2 was refluxed for 24 h. The solution was added saturated aqueous $NaHCO_3$ until pH = 7, and then was extracted with 20 mL of CH_2Cl_2. The organic part was dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (trace, <2%) and **3a** (trace, <2%).

A mixture of **1a** (0.2 mmol), **2a** (0.2 mmol) and TFA (0.2 mmol) in 2 mL of boiling CH_2Cl_2 was stirred for 24 h. Then, the solution was diluted with 20 mL of CH_2Cl_2, washed with 5 mL of saturated aqueous $NaHCO_3$ and then 5 mL of water, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (46%) and **3a** (18%).

A mixture of **1a** (0.2 mmol), **2a** (0.2 mmol) and TFA (0.2 mmol) in 2 mL of boiling PhMe was stirred for 24 h. Then, the solution was diluted with 20 mL of CH_2Cl_2, washed with 5 mL of saturated aqueous $NaHCO_3$ and then 5 mL of water, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (33%) and **3a** (29%).

A mixture of **1a** (0.2 mmol) and **2a** (0.2 mmol) in 2 mL of boiling TFA was stirred for 12 h. TFA was distilled out for reuse, the residue was redissolved in 20 mL of CH_2Cl_2, washed with 5 mL of saturated aqueous $NaHCO_3$, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (trace, <2%) and **3a** (61%).

A mixture of **1a** (0.2 mmol) and **2a** (0.4 mmol) in 2 mL of boiling TFA was stirred for 12 h. TFA was distilled out for reuse, the residue was redissolved in 20 mL of CH_2Cl_2, washed with 5 mL of saturated aqueous $NaHCO_3$, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (trace, <2%) and **3a** (80%).

A mixture of **1a** (0.2 mmol) and **2a** (0.4 mmol) in 2 mL of boiling formic acid was stirred for 12 h. Formic acid was distilled out for reuse, the residue was redissolved in 20 mL of CH_2Cl_2, washed with 5 mL of saturated aqueous $NaHCO_3$, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (trace, <2%) and **3a** (76%).

A mixture of **1a** (0.2 mmol) and **2a** (0.4 mmol) in 2 mL of boiling acetic acid was stirred for 24 h. Acetic acid was distilled out for reuse, the residue was redissolved in 20 mL of CH_2Cl_2, washed with 5 mL of saturated aqueous $NaHCO_3$, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give **4a** (trace, <2%) and **3a** (trace, <2%).

1.3 Synthesis of 2-carboxy-4-aryl-quinolines

A mixture of an aniline (**1**, 0.2 mmol) and an γ-aryl-β, γ-unsaturated α-ketoesters **2** (0.4 mmol) in 2 mL of TFA was stirred at reflux for 8-18 h, after which TFA was distilled out for reuse. The residue was redissolved in 20 mL of CH_2Cl_2, and the solution was washed with 5 mL of saturated aqueous $NaHCO_3$, dried over anhydrous Na_2SO_4, filtered, and

evaporated under reduced pressure. The products **3** were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v).

Methyl 7,8-dimethyl-4-phenylquinoline-2-carboxylate (3a): 80% yield; white solid; mp = 119-120 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.70 (d, *J* = 8.6 Hz, 1H), 7.51 (s, br, 5H), 7.39 (d, *J* = 8.6 Hz, 1H), 4.07 (s, 3H), 2.90 (s, 3H), 2.52 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.5, 149.7, 147.2, 146.1, 138.1, 137. 9, 136.2, 131.5, 129.6, 128.6, 128.5, 126.2, 122.6, 120.2, 52.9, 20.7, 13.8; FTIR (KBr) 2951, 1718, 1444, 1248, 1131, 765, 706 cm^{-1}; HRMS (FAB) Calcd. For $(M + H)^+$ $C_{19}H_{18}NO_2$: 292.1332, Found: 292.1332; Anal. Calcd. For $C_{19}H_{17}NO_2$: C, 78.33; H, 5.88; N, 4.81. Found: C, 78.19; H, 5.92; N, 4.79.

Methyl 6-methoxy-4-phenylquinoline-2-carboxylate (3b): 81% yield; white solid; mp = 141-142 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.22 (dd, *J* = 9.2, 2.4 Hz, 1H), 8.08 (d, *J* = 2.4 Hz, 1H), 7.51-7.50 (s, br, 5H), 7.40 (dd, *J* = 9.3, 2.7 Hz, 1H), 7.19 (d, *J* = 2.4 Hz, 1H), 4.04 (s, 3H), 3.77 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 159.6, 148.1, 145.0, 144.3, 137.79, 132.6, 129.3, 129.2, 128.8, 128.7, 122.9, 121.8, 103.3, 55.5, 53.1; FTIR (KBr) 3055, 2952, 1740, 1621, 1472, 1226, 1108, 836, 707 cm^{-1}; Anal. Calcd. For $C_{18}H_{15}NO_3$: C, 73.71; H, 5.15; N, 4.78. Found: C, 73.77; H, 5.14; N, 4.75.

Methyl 6-methyl-4-phenylquinoline-2-carboxylate (3c): 82% yield; white solid; mp = 129-130 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 (d, *J* = 8.7 Hz, 1H), 8.11 (s, 1H), 7.71 (s, 1H), 7.58 (d, *J* = 8.8 Hz, 1H), 7.52 (s, br, 5H), 4.07 (s, 3H), 2.48 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.2, 149.0, 146.8, 146.5, 139.1, 137.7, 132.5, 130.8, 129.5, 128.7, 128.6, 127.9, 124.4, 121.5, 53.2, 22.1; FTIR (KBr) 3058, 2945, 1720, 1448, 1361, 1253, 1119, 824, 759, 703 cm^{-1}; MS (TOF EI): m/z 277; Anal. Calcd. For $C_{18}H_{15}NO_2$: C, 77.96; H, 5.45; N, 5.05. Found: C, 77.92; H, 5.47; N, 4.91.

Methyl 6-fluoro-4-phenylquinoline-2-carboxylate (3d): 80% yield; white solid; mp = 134-135 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.40-8.35 (m 1H), 8.17 (s, 1H), 7.61-7.49 (m, 7H), 4.09 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.8, 162.0(d, $^{1}J_{CF}$ = 249.8 Hz, 1C), 149.4(d, $^{3}J_{CF}$ = 5.9 Hz, 1C), 146.9(d, $^{4}J_{CF}$ = 2.9 Hz, 1C), 145.2, 137.0, 133.8(d, $^{3}J_{CF}$ = 9.3 Hz, 1C), 129.4, 129.0, 128.9, 121.8, 120.7(d, $^{2}J_{CF}$ = 26.0 Hz, 1C), 109.3(d, $^{2}J_{CF}$ = 23.2 Hz, 1C), 53.3; FTIR (KBr) 3056, 1714, 1465, 1366, 1258, 1231, 1195, 1140, 1108, 836, 785, 710 cm^{-1}; GCT-MS (TOF EI$^+$): m/z 281; HRMS (TOF EI$^+$) Calcd. For M^+ $C_{17}H_{12}NO_2F$: 281.0852, Found: 281.0851; Anal. Calcd. For $C_{17}H_{12}NO_2F$: C, 72.59; H, 4.30; N, 4.98. Found: C, 72.47; H, 4.22; N, 4.97.

Methyl 6-nitro-4-phenylquinoline-2-carboxylate (3e): 69% yield; yellow solid; mp = 250-251 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.92 (d, *J* = 2.1 Hz, 1H), 8.55 (dd, *J* = 9.3, 2.3 Hz, 1H), 8.50 (d, *J* = 9.2 Hz, 1H), 8.29 (s, 1H), 7.63-7.53 (m, 5H), 4.12 (s, 3H); ^{13}C

NMR (75 MHz, $CDCl_3$) δ 165.3, 152.5, 150.4, 150.1, 147.0, 136.0, 133.0, 129.8, 129.5, 129.3, 127.0, 123.6, 122.8, 122.7, 53.6; FTIR (KBr) 3074, 2955, 1724, 1487, 1367, 1344, 1258, 1140, 797, 704 cm^{-1}; HRMS (FAB) calcd. For $(M + H)^+$ $C_{17}H_{13}N_2O_4$: 309.0870, Found: 309.0869; Anal. Calcd. For $C_{17}H_{12}N_2O_4$: C, 66.23; H, 3.92; N, 9.09. Found: C, 65.95; H, 3.92; N, 8.93.

Methyl 4-phenylbenzo[*h*]quinoline-2-carboxylate (3f): 81% yield; white solid; mp = 177-178 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 9.42 (dd, *J* = 8.4, 1.3 Hz, 1H), 8.19 (s, 1H), 7.74-7.64 (m, 5H), 7.47-7.45 (m, 5H), 4.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.3, 149.4, 146.7, 145.7, 137.9, 133.4, 131.8, 129.9, 129.7, 128.9, 128.7, 128.6, 127.7, 127.6, 126.2, 125.5, 122.7, 122.6, 53.0; FTIR (KBr) 3047, 2947, 1739, 1434, 1244, 1131, 839, 756, 707 cm^{-1}; Anal. Calcd. For $C_{21}H_{15}NO_2$: C, 80.49; H, 4.82; N, 4.47. Found: C, 80.24; H, 4.97; N, 4.31.

Methyl 1-phenylbenzo[*f*]quinoline-3-carboxylate (3g): 82% yield; white solid; mp = 152-153 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 8.10 (d, *J* = 9.1 Hz, 1H), 8.07 (s, 1H), 7.92 (d, *J* = 9.1 Hz, 1H), 7.77 (d, *J* = 7.9 Hz, 1H), 7.65 (d, *J* = 8.5 Hz, 1H), 7.44-7.31 (m, 6H), 7.08 (dt, *J* = 7.8, 1.3 Hz, 1H), 4.00 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.8, 149.7, 149.5, 145.8, 142.1, 133.7, 132.3, 129.4, 129.2, 128.9, 128.7, 128.6, 128.5, 128.2, 127.7, 126.0, 125.9, 124.8, 53.2; FTIR (KBr) 3027, 2950, 1743, 1240, 1131, 777, 756, 710 cm^{-1}; Anal. Calcd. For $C_{21}H_{15}NO_2$: C, 80.49; H, 4.82; N, 4.47. Found: C, 80.57; H, 4.89; N, 4.62.

Methyl 4-phenylquinoline-2-carboxylate (3h): 80% yield; white solid; mp = 101-102 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 8.39 (t, *J* = 8.1 Hz, 1H), 8.17 (s, 1H), 7.98 (t, *J* = 7.7 Hz, 1H), 7.79 (dd, *J* = 7.0, 7.9 Hz, 1H), 7.61-7.53 (m, 6H), 4.11 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 150.0, 148.1, 147.4, 137.4, 131.1, 130.1, 129.6, 128.8, 128.7, 127.9, 125.8, 121.3, 53.2; FTIR (KBr) 2953, 1714, 1560, 1457, 1365, 1257, 1131, 1111, 792, 766, 699 cm^{-1}; Anal. Calcd. For $C_{17}H_{13}NO_2$: C, 77.55; H, 4.98; N, 5.32. Found: C, 77.34; H, 5.06; N, 5.47.

Methyl 4-(4-methoxyphenyl)quinoline-2-carboxylate (3i): 79% yield; white solid; mp = 131-132 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 8.29 (d, *J* = 8.5 Hz, 1H), 8.05 (s, 1H), 7.94 (d, *J* = 8.5 Hz, 1H), 7.70 (dt, *J* = 7.1, 1.3 Hz, 1H), 7.51 (dt, *J* = 8.2, 1.1 Hz, 1H), 7.40 (dd, *J* = 8.8, 2.1 Hz, 2H), 6.99 (d, *J* = 8.8 Hz, 2H), 4.01 (s, 3H), 3.82 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 160.2, 149.8, 148.2, 147.4, 131.1, 130.9, 130.0, 129.7, 128.5, 128.0, 125.8, 121.2, 114.2, 55.4, 53.2; FTIR (KBr) 2955, 1718, 1455, 1368, 1259, 1135, 839, 821, 787, 762 cm^{-1}; Anal. Calcd. For $C_{18}H_{15}NO_3$: C, 73.71; H, 5.15; N, 4.78. Found: C, 73.85; H, 5.33; N, 4.62.

Methyl 4-*p*-tolylquinoline-2-carboxylate (3j): 82% yield; white solid; mp = 114-115 °C;

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.50 (d, *J* = 8.5 Hz, 1H), 8.07 (s, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.71 (dt, *J* = 7.7, 1.2 Hz, 1H), 7.52 (dt, *J* = 8.2, 1.1 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 4.02 (s, 3H), 2.39 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 150.2, 148.1, 147.4, 138.8, 134.5, 131.0, 130.1, 129.5, 129.4, 128.6, 128.0, 125.9, 121.3, 53.2, 21.3; FTIR (KBr) 2948, 1723, 1254, 1131, 1111, 825, 780, 728 cm^{-1}; Anal. Calcd. For $C_{18}H_{15}NO_2$: C, 77.96; H, 5.45; N, 5.05. Found: C, 77.75; H, 5.61; N, 5.00.

Ethyl 4-(4-chlorophenyl)quinoline-2-carboxylate (3k): 83% yield; white solid; mp = 126-127 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.36 (d, *J* = 8.4 Hz, 1H), 8.09 (s, 1H), 7.89 (d, *J* = 8.3 Hz, 1H), 7.78 (t, *J* = 7.6 Hz, 1H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.51 (d, *J* = 8.3 Hz, 2H), 7.45 (q, *J* = 8.3 Hz, 2H), 4.55 (q, *J* = 7.0 Hz, 2H), 1.47 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.4, 148.5, 148.2, 147.8, 135.9, 135.0, 131.3, 130.9, 130.2, 129.0, 128.8, 127.5, 125.3, 121.2, 62.4, 14.4; FTIR (KBr) 3074, 2984, 1718, 1490, 1377, 1253, 1108, 1018, 831, 763 cm^{-1}; Anal. Calcd. For $C_{18}H_{14}ClNO_2$: C, 69.35; H, 4.53; N, 4.49. Found: C, 69.18; H, 4.57; N, 4.35.

Methyl 4-(4-nitrophenyl)quinoline-2-carboxylate (3l): 72% yield; yellow solid; mp = 205-206 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.33-8.31 (m, 3H), 8.06 (s, 1H), 7.75 (t, *J* = 7.1 Hz, 2H), 7.65-7.56 (m, 3H), 4.01 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7, 148.1, 148.1, 147.5, 147.3, 143.9, 131.4, 130.6, 129.5, 127.0, 124.9, 124.0, 121.1, 53.4; FTIR (KBr) 3066, 2951, 1723, 1514, 1347, 1254, 1133, 1110, 832, 768; Anal. Calcd. For $C_{17}H_{12}N_2O_4$: C, 66.23; H, 3.92; N, 9.09. Found: C, 66.09; H, 4.13; N, 9.05.

Ethyl 4-(4-chlorophenyl)-6-methoxyquinoline-2-carboxylate (3m): 82% yield; white solid; mp = 171-172 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 (d, *J* = 9.3 Hz, 1H), 8.04 (s, 1H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.6 Hz, 2H), 7.42 (dd, *J* = 9.3, 2.7 Hz, 1H), 7.11 (d, *J* = 2.6 Hz, 1H), 4.53 (q, *J* = 7.1 Hz, 2H), 3.80 (s, 3H), 1.47 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.5, 159.7, 146.7, 145.3, 144.3, 136.3, 134.8, 132.9, 130.7, 129.1, 128.9, 123.0, 121.7, 102.9, 62.2, 55.6, 14.4; FTIR (KBr) 3053, 2975, 1729, 1618, 1489, 1471, 1280, 1228, 1110, 1022, 840 cm^{-1}; Anal. Calcd. For $C_{19}H_{16}ClNO_3$: C, 66.77; H, 4.72; N, 4.10. Found: C, 66.50; H, 4.73; N, 3.82.

Ethyl 4-(4-chlorophenyl)-8-methoxyquinoline-2-carboxylate (3n): 66% yield; white solid; mp = 161-162 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.14 (d, *J* = 4.6 Hz, 1H), 7.57-7.43 (m, 6H), 7.11 (t, *J* = 5.9 Hz, 1H), 4.58-4.50 (m, 2H), 4.11 (d, *J* = 5.3 Hz, 3H), 1.54-1.48 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.5, 156.2, 148.4, 146.4, 140.1, 136.2, 134.9, 130.9, 129.2, 128.9, 128.8, 121.9, 116.9, 107.9, 62.4, 56.2, 14.3; FTIR (KBr) 3423, 3046, 2983, 1721, 1466, 1261, 1081, 836, 756 cm^{-1}; Anal. Calcd. For $C_{19}H_{16}ClNO_3$: C, 66.77; H, 4.72; N, 4.10. Found: C, 66.63; H, 4.80; N, 3.99.

Ethyl 6-chloro-4-(4-chlorophenyl)quinoline-2-carboxylate (3o): 83% yield; white

solid; mp = 220-221 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.31 (d, *J* = 9.0 Hz, 1H), 8.12 (s, 1H), 7.87 (d, *J* = 2.3 Hz, 1H), 7.73 (dd, *J* = 9.0, 2.3 Hz, 1H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 4.58 (q, *J* = 7.1 Hz, 2H), 1.50 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.0, 148.0, 147.7, 146.5, 135.4, 135.2, 135.1, 132.8, 131.2, 130.8, 129.2, 128.2, 124.2, 122.0, 62.5, 14.4; FTIR (KBr) 3031, 2985, 1716, 1597, 1485, 1450, 1377, 1273, 1253, 1141, 1087, 1020, 847, 829, 789 cm^{-1}; Anal. Calcd. For $C_{18}H_{13}Cl_2NO_2$: C, 62.45; H, 3.78; N, 4.05. Found: C, 62.18; H, 3.89, N, 3.93.

Ethyl 8-chloro-4-(4-chlorophenyl)quinoline-2-carboxylate (3p): 71% yield; white solid; mp = 179-180 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.91 (dd, *J* = 7.4, 0.8 Hz, 1H), 7.83 (dd, *J* = 8.5, 1.0 Hz, 1H), 7.51 (t, *J* = 8.2 Hz, 1H), 7.53 (d, *J* = 8.5 Hz, 2H), 7.46 (d, *J* = 8.5 Hz, 2H), 4.54 (q, *J* = 7.1, 2H), 1.50 (t, *J* = 7.1, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.1, 149.2, 148.2, 144.6, 135.6, 135.5, 135.3, 130.9, 129.4, 129.1, 129.0, 128.5, 124.5, 122.0, 62.5, 14.3; FTIR (KBr) 3052, 2985, 1721, 1447, 1250, 1130, 785, 764 cm^{-1}; Anal. Calcd. For $C_{18}H_{13}Cl_2NO_2$: C, 62.45; H, 3.78; N, 4.05. Found: C, 62.18; H, 3.84; N, 3.89.

Ethyl 4-(4-chlorophenyl)-6-nitroquinoline-2-carboxylate (3q): 68% yield; yellow solid; mp = 240-241 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.86 (d, *J* = 1.9 Hz, 1H), 8.56 (dd, *J* = 9.3, 2.2 Hz, 1H), 8.52 (d, *J* = 9.2 Hz, 1H), 8.25 (s, 1H), 7.61 (dd, *J* = 8.5, 2.1 Hz, 2H), 7.50 (dd, *J* = 8.4, 2.1 Hz, 1H), 4.60 (q, *J* = 7.1 Hz, 2H), 1.46 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.6, 151.0, 150.9, 150.2, 147.0, 136.2, 134.4, 133.2, 130.8, 129.6, 126.7, 123.6, 122.6, 122.4, 62.9, 14.4; FTIR (KBr) 3070, 2981, 1721, 1487, 1345, 1250, 1112, 837, 745 cm^{-1}; Anal. Calcd. For $C_{18}H_{13}ClN_2O_4$: C, 60.60; H, 3.67; N, 7.85. Found: C, 60.60; H, 3.65; N, 7.65.

Ethyl 4-(4-chlorophenyl)-8-nitroquinoline-2-carboxylate (3r): 42% yield; yellow solid; mp = 206-207 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.22 (s, 1H), 8.13 (d, *J* = 8.5 Hz, 1H), 8.17 (d, *J* = 7.5 Hz, 1H), 7.68 (t, *J* = 8.0 Hz, 1H), 7.57 (d, *J* = 8.5 Hz, 2H), 7.46 (d, *J* = 8.5 Hz, 2H), 4.52 (q, *J* = 7.1, 2H), 1.48 (t, *J* = 7.1, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.7, 149.8, 149.2, 149.0, 139.6, 135.8, 134.8, 130.9, 129.5, 129.3, 128.3, 127.2, 124.4, 122.7, 62.6, 14.2; FTIR (KBr) 2992, 1724, 1531, 1374, 1351, 1255, 1130, 1091, 1016, 842, 767 cm^{-1}; HRMS (FAB) Calcd. For M^+ $C_{18}H_{14}ClN_2O_4$: 357.0637, Found: 357.0641; Anal. Calcd. For $C_{18}H_{14}ClN_2O_4$: C, 60.43; H, 3.94; N, 7.83. Found: C, 60.40; H, 3.98; N, 7.57.

Ethyl 4-(4-chlorophenyl)-6-hydroxyquinoline-2-carboxylate (3s): 79% yield; white solid; mp = 250-253 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 10.42 (s, 1H), 8.08 (d, *J* = 8.8 Hz, 1H), 7.84 (s, 1H), 7.65-7.56 (m, 4H), 7.41 (d, *J* = 8.3 Hz, 1H), 7.07 (s, 1H), 4.38 (q, *J* = 6.5 Hz, 2H), 1.34 (t, *J* = 6.5 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 165.3, 158.6, 158.4, 145.7, 143.2, 136.6, 134.0, 132.8, 131.6, 129.3, 129.1, 121.3, 106.2, 106.1, 61.8,

14.7; FTIR (KBr) 3035, 1731, 1616, 1468, 1279, 1228, 836 cm^{-1}; HRMS (FAB) calcd. For $(M + H)^+$ $C_{18}H_{15}ClNO_3$: 328.0735, Found: 328.0743; Anal. Calcd. For $C_{18}H_{14}ClNO_3$: C, 65.96; H, 4.31; N, 4.27. Found: C, 65.86; H, 4.38; N, 4.11.

1.4 Synthesis of Schiff's base **5t**

According to the reported paper,[7] a mixture of *trans*-cinnamaldehyde (0.10 mol), aniline (0.11 mol) and *p*-toluenesulfonic acid (0.05 mol) in CH_2Cl_2 (40 mL) was stirred at room temperature. When the mixture became turbid due to the formation of water, 25 g of 3 Å molecular sieves was added and the mixture then agitated for 45 min. The mixture was filtered, evaporated under reduced pressure, and recrystallized from ethanol to give pale yellow solid in 83% yield; mp 106-108 °C (literature.[7] 105-106 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 (d, *J* = 7.4 Hz, 1H), 7.54 (d, *J* = 7.3 Hz, 2H), 7.43-7.32 (m, 5H), 7.25 (t, *J* = 7.6 Hz, 3H), 7.16-7.14(m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.6, 151.8, 144.0, 135.7, 129.7, 129.3, 129.0, 128.7, 127.6, 126.2, 121.1.

1.5 Procedures for reaction mechanism

A mixture of 2,3-dimethylaniline (**1a,** 10 mmol) and (3*E*)-2-oxo-4-phenylbut-3-enoate methyl ester (**2a**, 20 mmol) in 10 mL of TFA was stirred at reflux for 6 h, after which TFA was distilled out for reuse. The residue was redissolved in 40 mL of CH_2Cl_2, washed with 10 mL of saturated aqueous $NaHCO_3$, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give (3*E*)-2-(2,3-dimethylphenylimino)-4-phenylbut-3-enoate methyl ester (**5a**, 2%) and methyl 7,8-dimethyl-4-phenylquinoline-2-carboxylate (**3a**, 51%).

(3*E*)-2-(2,3-Dimethylphenylimino)-4-phenylbut-3-enoate methyl ester (5a, syn and anti): 2% yield; yellow foam; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65-6.61 (m, 10H), 4,03 (3.60) (s, 3H), 2.34 (2.31) (s, 3H), 2.17 (2.11) (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.4 (165.0), 158.2 (159.9), 147.5 (149.0), 142.2 (141.0), 137.8 (137.3), 135.4 (135.3), 130.0 (129.8), 128.9 (128.9), 127.9 (127.7), 126.6 (127.6), 126.5 (126.7), 125.7 (125.8), 117.3 (125.5), 116.7 (115.4), 51.88 (51.85), 20.2 (20.1), 14.1 (14.0); FTIR (neat) 3061, 3027, 2951, 1735, 1619, 1579, 1464, 1322, 1193, 1148, 969, 757, 730, 695 cm^{-1}; GCT-MS (TOF MS EI^+) m/z 293 (M^+), 234, 218; HRMS (TOF MS EI^+) Calcd. For M^+ $C_{19}H_{19}NO_2$: 293.1416, Found: 293.1415.

A solution of Schiff's base **5a** (0.1 mmol) in 2 mL of TFA was stirred at reflux for 2 h, after that TFA was distilled out for reuse. The residue was redissolved in 20 mL of CH_2Cl_2, washed with 5 ml of saturated aqueous $NaHCO_3$, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The products were isolated by flash chromatography on silica gel with ethyl acetate-petroleum ether mixture (1:5, v/v) to give methyl 7,8-dimethyl-4-phenylquinoline-2-carboxylate (**3a**, 91%).

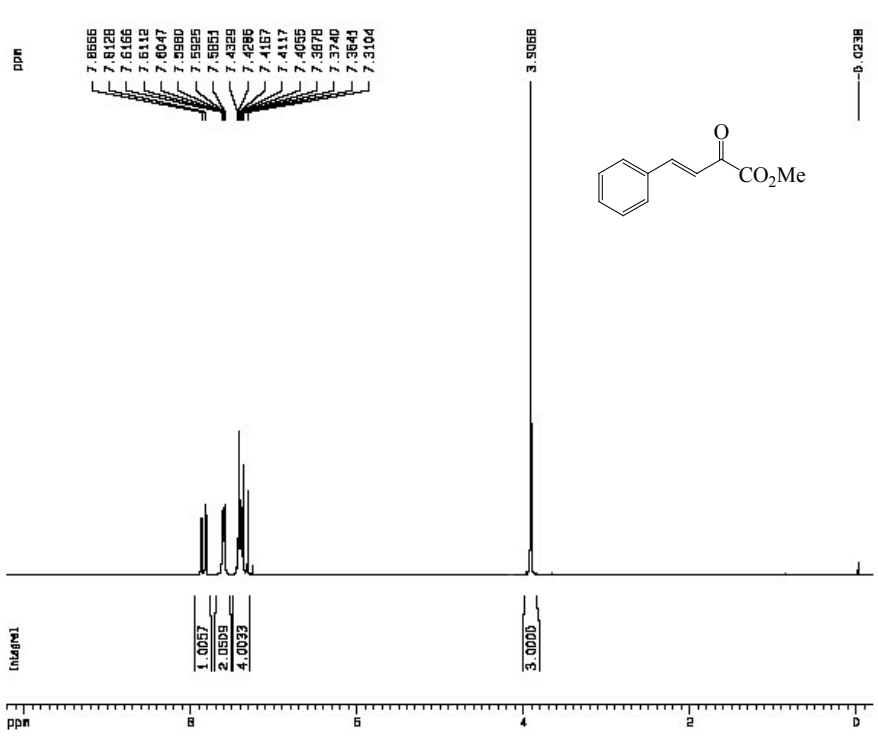
A solution of Schiff's base **5t** (0.1 mmol) in 2 mL of TFA was stirred at reflux for 2 h, after that TFA was distilled out for reuse. The residue was redissolved in 20 mL of CH_2Cl_2, washed with 5 ml of saturated aqueous $NaHCO_3$, dried over anhydrous Na_2SO_4, but Schiff's base **5t** was not consumed.

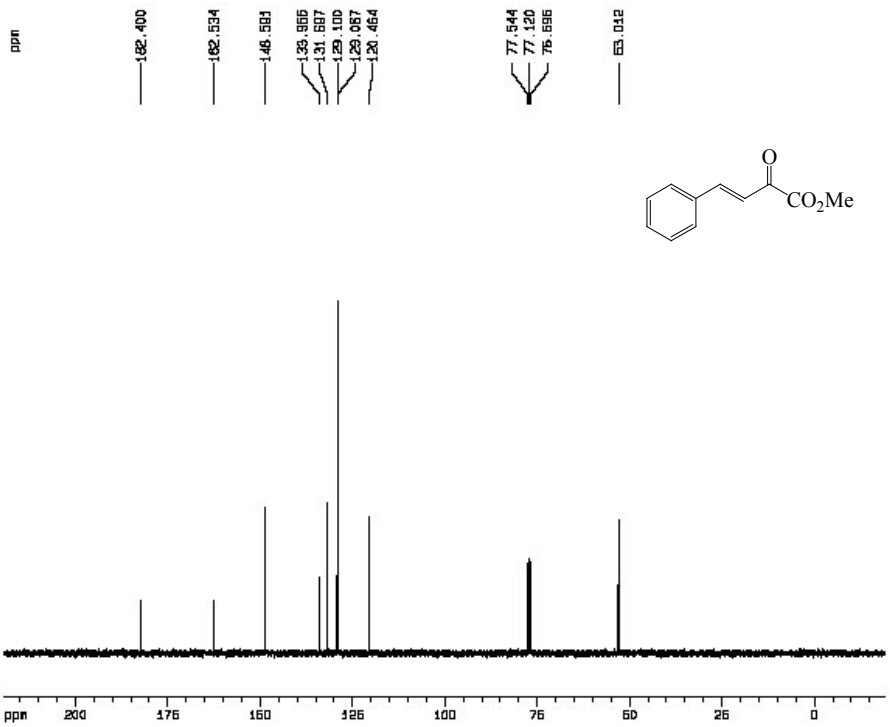
References

1. Stecher, E. D.; Ryder, H. F. *J. Am. Chem. Soc*, **1952**, *74*, 4392.
2. Audrain, H.; Thorhauge, J.; Hazell, R. G.; Joergensen, K. A. *J. Org. Chem,* **2000**, *65*, 4487.
3. Dujardin, G.; Maudet, M.; Brown, E. *Tetrahedron Lett,* **1994**, *35*, 8619.
4. Vaughan, W. R.; Tripp, R. C. M. *J. Am. Chem. Soc*. **1960**, *82*, 4370.
5. Sugimura, H.; Yoshida, K. *Bull. Chem. Soc. Jpn*, **1992**, *65*, 3209.
6. Dujardin, G.; Leconte, S.; Benard, A.; Brown, E. *Synlett,* **2001**, *1*, 147.
7. Eisch, J. J.; Dluzniewski, T. *J. Org. Chem*. **1989**, *54*, 1269.

2. ^{1}H and ^{13}C NMR spectra of synthesized compounds

(3*E*)-2-Oxo-4-phenylbut-3-enoic acid methyl ester (2a)





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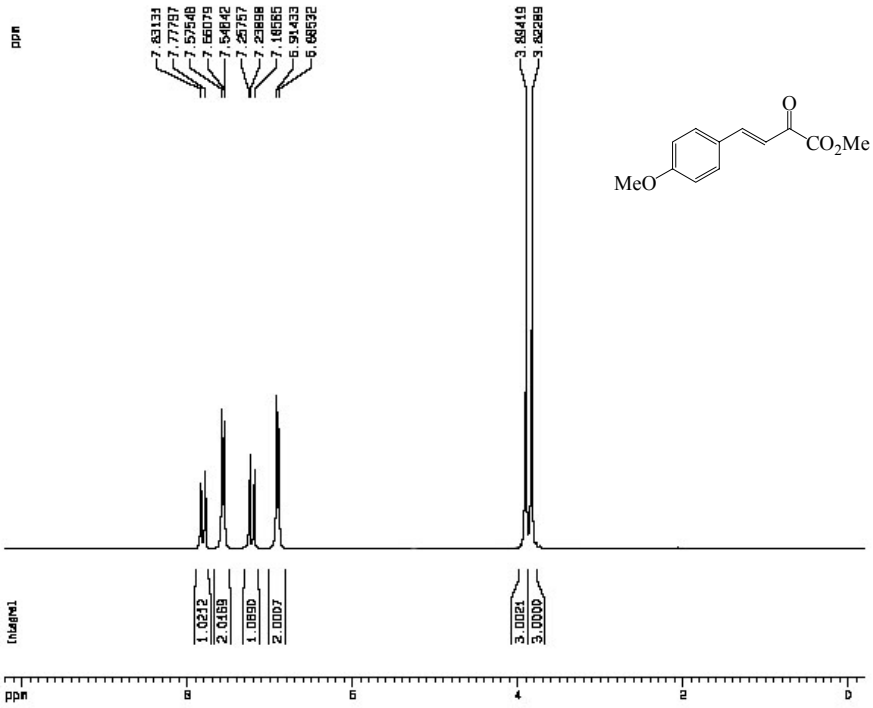
(3*E*)-4-(4-Methoxyphenyl)-2-oxobut-3-enoic acid methyl ester (2b)

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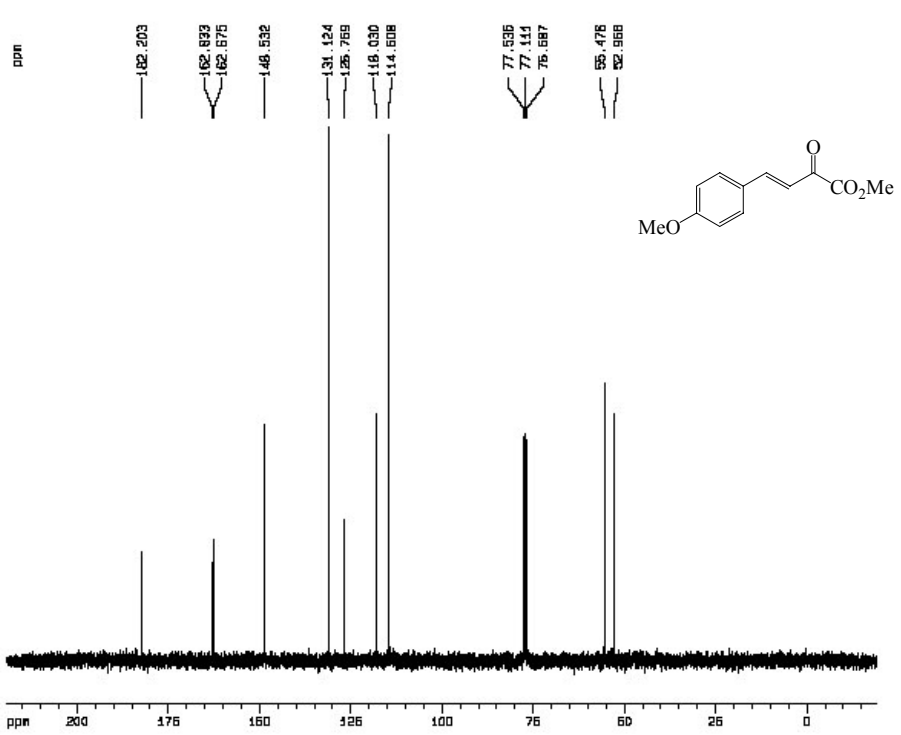


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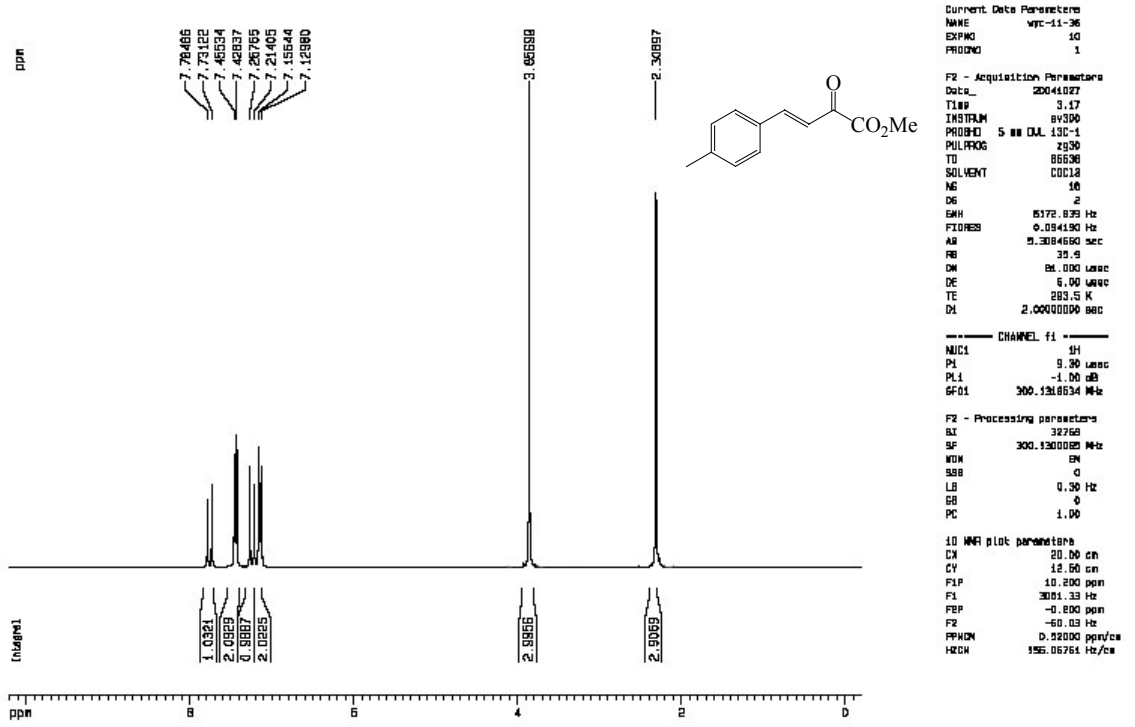

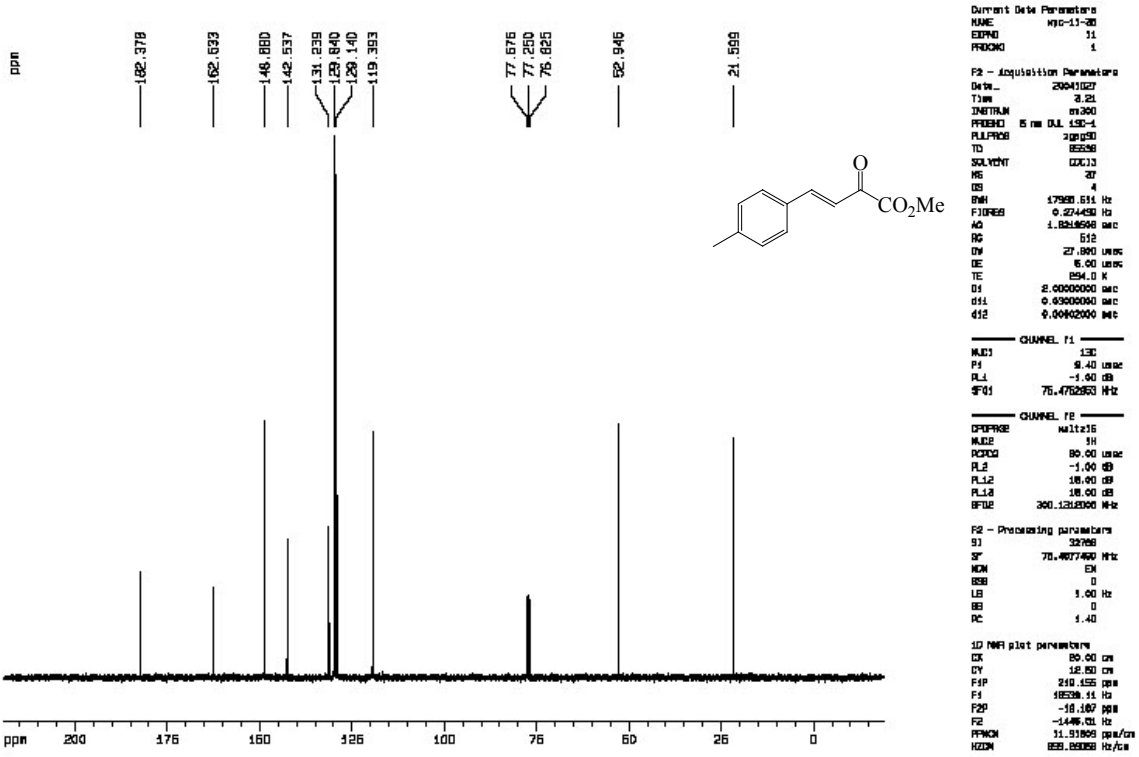

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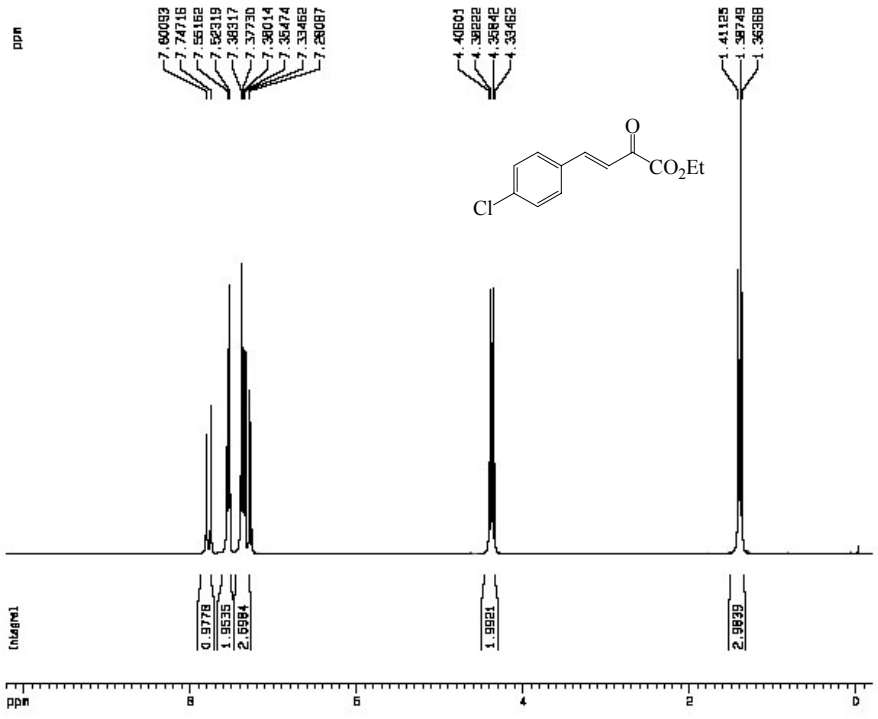


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HZCM         156.06761 Hz/cm
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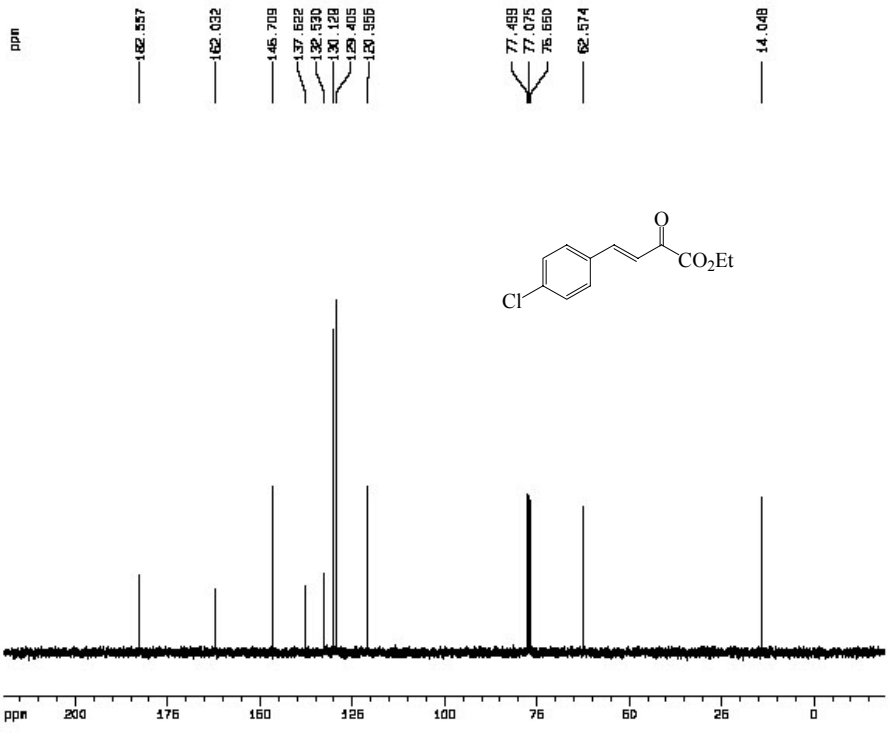


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Current Data Parameters
NAME          wyc-10-48-2
EXPNO              11
PROCNO              1

F2 - Acquisition Parameters
Date_          20040721
Time              80.18
INSTRUM           av300
PROBHD   5 mm DUL 13C-1
PULPROG        zgpg30
TD               65536
SOLVENT          CDCl3
NS                  70
DS                   4
SWH           17985.611 Hz
FIDRES        0.274450 Hz
AQ            1.8249598 sec
RG                2286.0
DW               27.800 usec
DE                6.00 usec
TE              300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

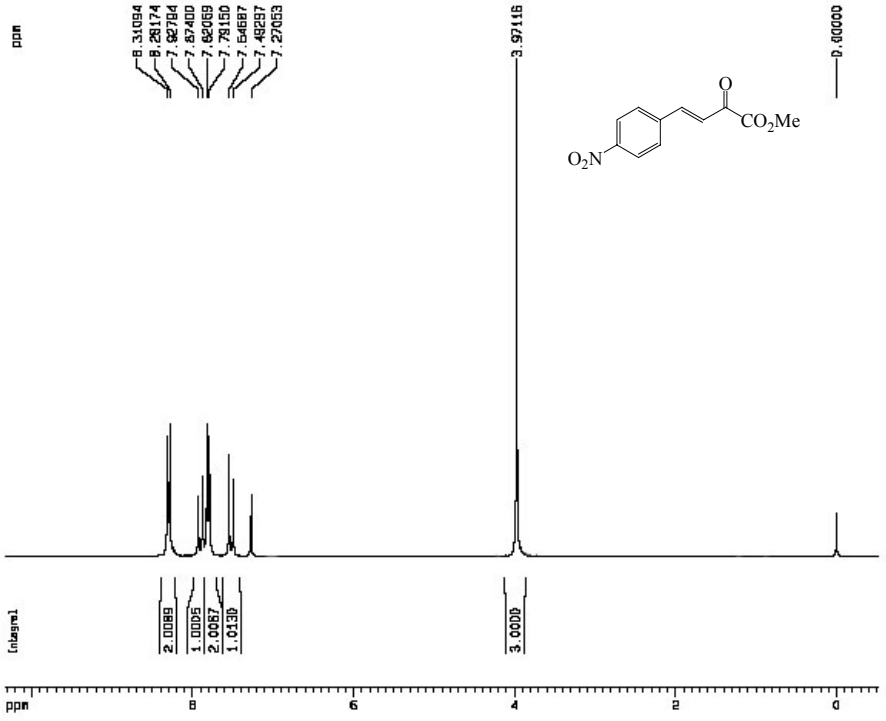
======== CHANNEL f1 ========
NUC1               13C
P1                9.40 usec
PL1              -1.00 dB
SFO1        75.4752853 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             16.00 dB
PL13             16.00 dB
SFO2        300.1318000 MHz

F2 - Processing parameters
SI                32768
SF          75.4677480 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
CY               12.00 cm
F1P             240.155 ppm
F1           18038.11 Hz
F2P             -18.187 ppm
F2           -1446.01 Hz
PPMCM         11.91858 ppm/cm
HZCM        895.89358 Hz/cm
```

(3*E*)-4-(4-Nitrophenyl)-2-oxobut-3-enoic acid methyl ester (2e)



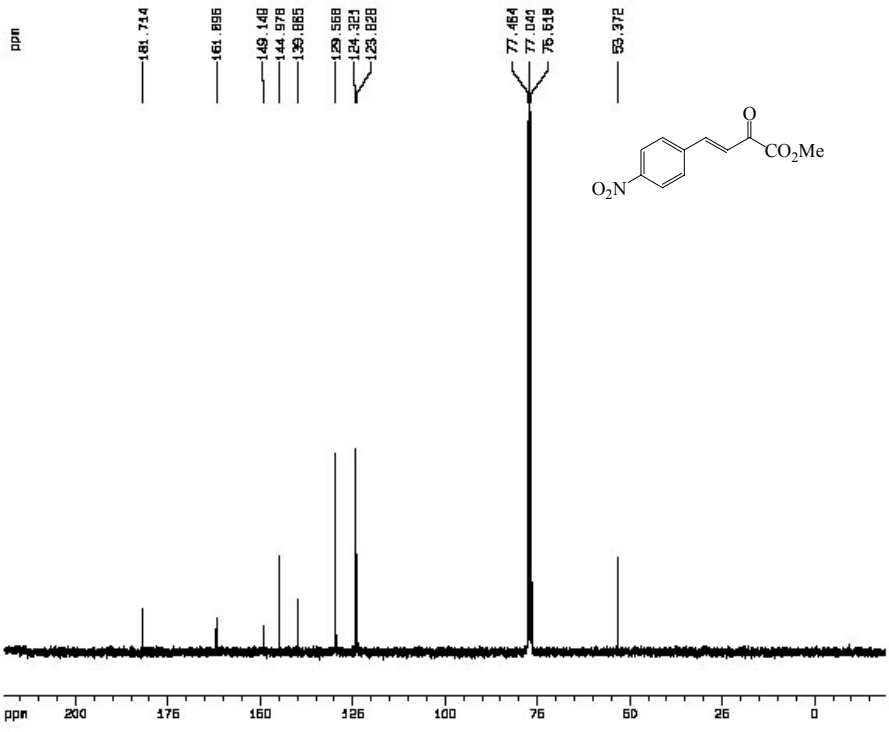

```
Current Data Parameters
NAME            wyc-8-15
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20040208
Time               21.59
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8172.838 Hz
FIDRES        0.094150 Hz
AQ            5.3084660 sec
RG                  256
DW                81.000 usec
DE                 6.00 usec
TE                281.8 K
D1             2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.30 usec
PL1              -1.00 dB
SFO1        300.1318634 MHz

F2 - Processing parameters
SI                32768
SF          300.1300032 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.60 cm
F1P               10.310 ppm
F1              3098.84 Hz
F2P               -0.022 ppm
F2              -156.67 Hz
PPMCM             0.54685 ppm/cm
HZCM            162.62924 Hz/cm
```




```
Current Data Parameters
NAME            wyc-8-10
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_          20040208
Time               03.22
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH          17960.611 Hz
FIDRES        0.274150 Hz
AQ            1.8249598 sec
RG                 6160.6
DW                27.800 usec
DE                 6.00 usec
TE                293.4 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00402000 sec

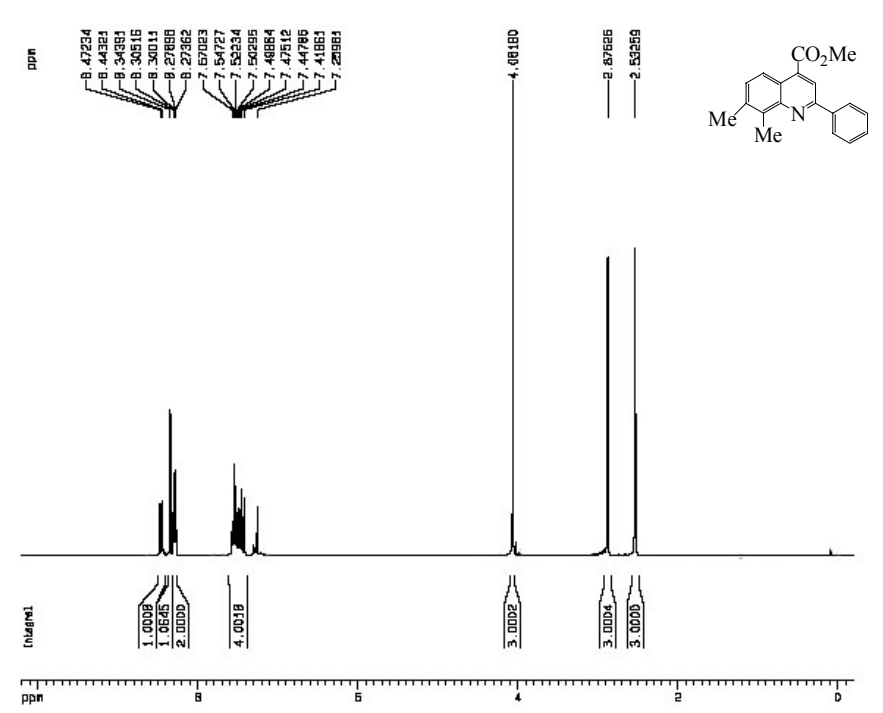
======== CHANNEL f1 ========
NUC1                13C
P1                9.40 usec
PL1              -1.00 dB
SFO1        75.4762953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        300.1312006 MHz

F2 - Processing parameters
SI                32768
SF          75.4677480 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
CY                12.60 cm
F1P              210.155 ppm
F1             15839.11 Hz
F2P              -18.187 ppm
F2             -1371.01 Hz
PPMCM            11.51809 ppm/cm
HZCM           868.86058 Hz/cm
```

Methyl 7,8-dimethyl-2-phenylquinoline-4-carboxylate (4a)



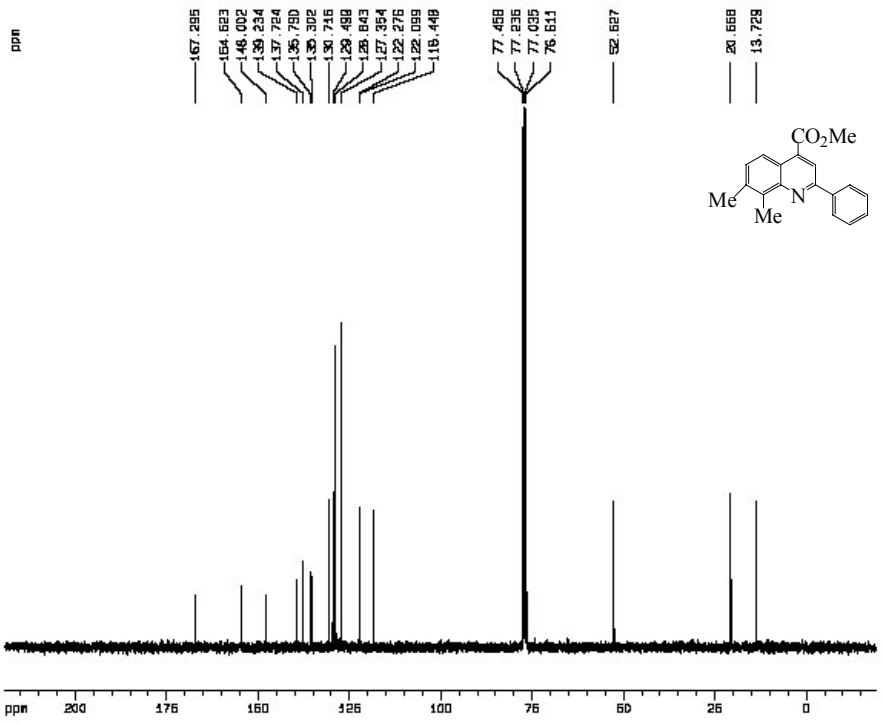

```
Current Data Parameters
NAME          kyc-11-20e1
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20041004
Time                4.19
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG           zg30
TD               86638
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                 256
DW              81.000 usec
DE                6.00 usec
TE              287.8 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.30 usec
PL1              -1.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                32768
SF          300.1300085 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               20.00 cm
CY               12.60 cm
F1P              10.200 ppm
F1              3061.33 Hz
F2P              -0.800 ppm
F2               -60.03 Hz
PPMCM         0.52000 ppm/cm
HZCM          156.06764 Hz/cm
```




```
Current Data Parameters
NAME          kyc-11-20c1
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_           20041004
Time                4.21
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                 908
DS                   4
SWH          17985.611 Hz
FIDRES        0.274450 Hz
AQ            1.8249568 sec
RG                646.1
DW              27.800 usec
DE                6.00 usec
TE              298.2 K
D1            2.00000000 sec
d11           0.03000040 sec
d12           0.00402000 sec

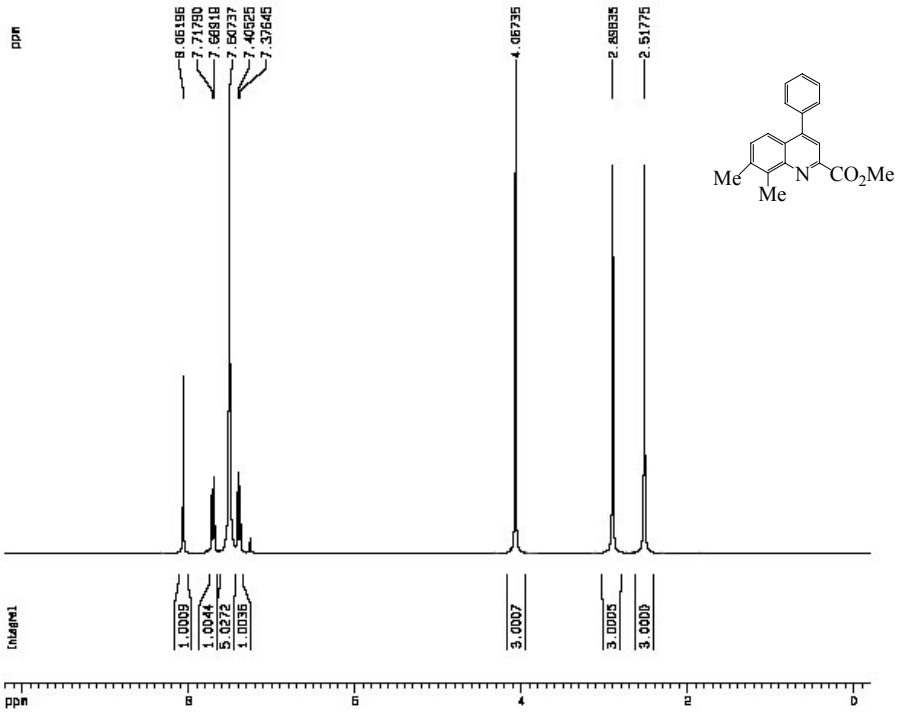
======== CHANNEL f1 ========
NUC1               13C
P1               9.40 usec
PL1              -1.00 dB
SFO1        75.4752853 MHz

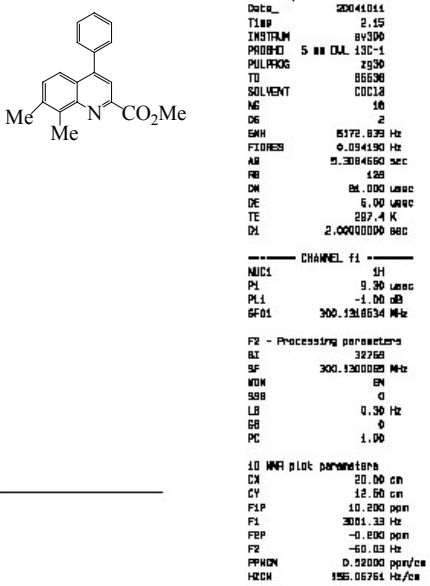
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        300.1318060 MHz

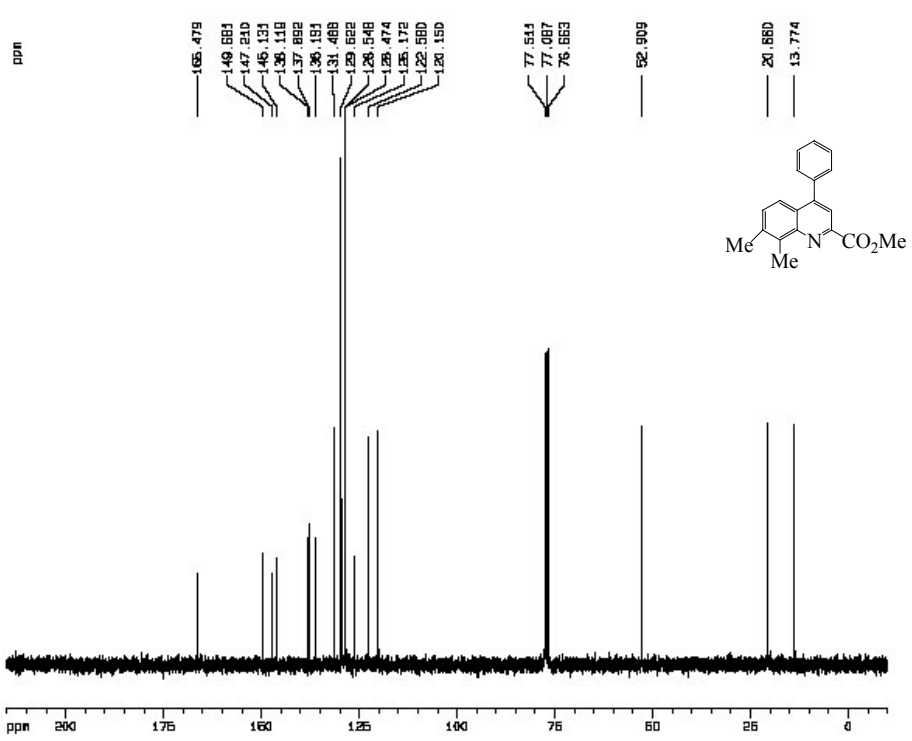
F2 - Processing parameters
SI                32768
SF          75.4677490 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40

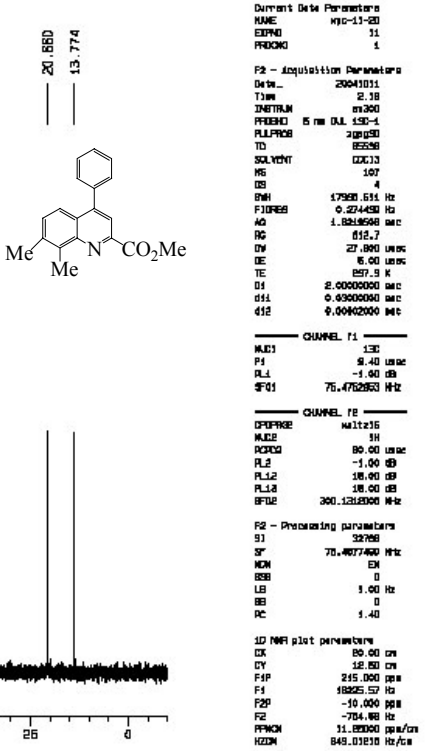
1D NMR plot parameters
CX               20.00 cm
CY               12.60 cm
F1P             219.155 ppm
F1            16538.11 Hz
F2P             -18.187 ppm
F2            -1446.01 Hz
PPMCM         11.91809 ppm/cm
HZCM          899.89058 Hz/cm
```

Methyl 7,8-dimethyl-4-phenylquinoline-2-carboxylate (3a)

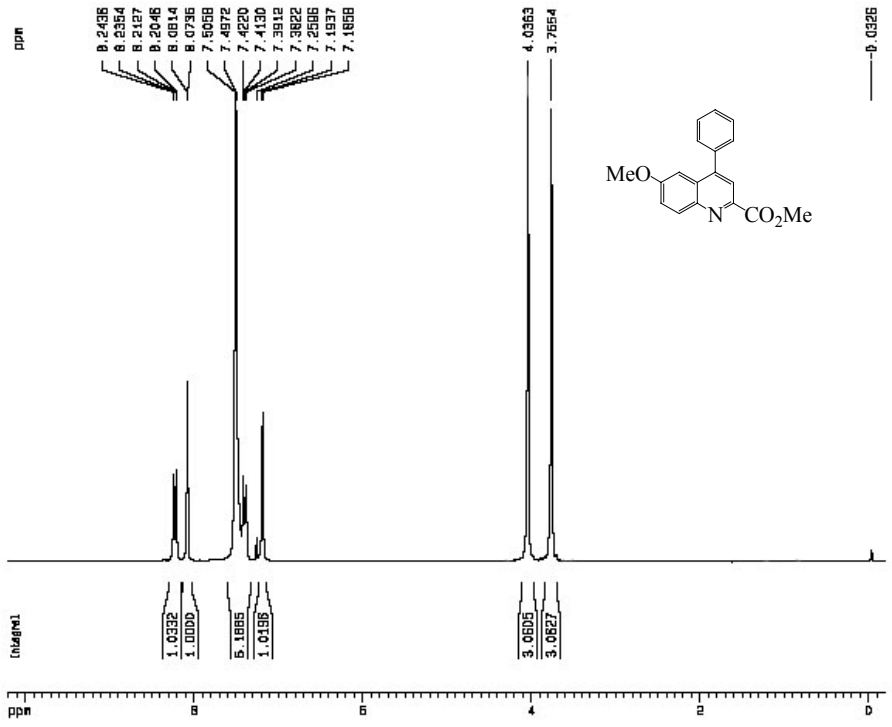








Methyl 6-methoxy-4-phenylquinoline-2-carboxylate (3b)



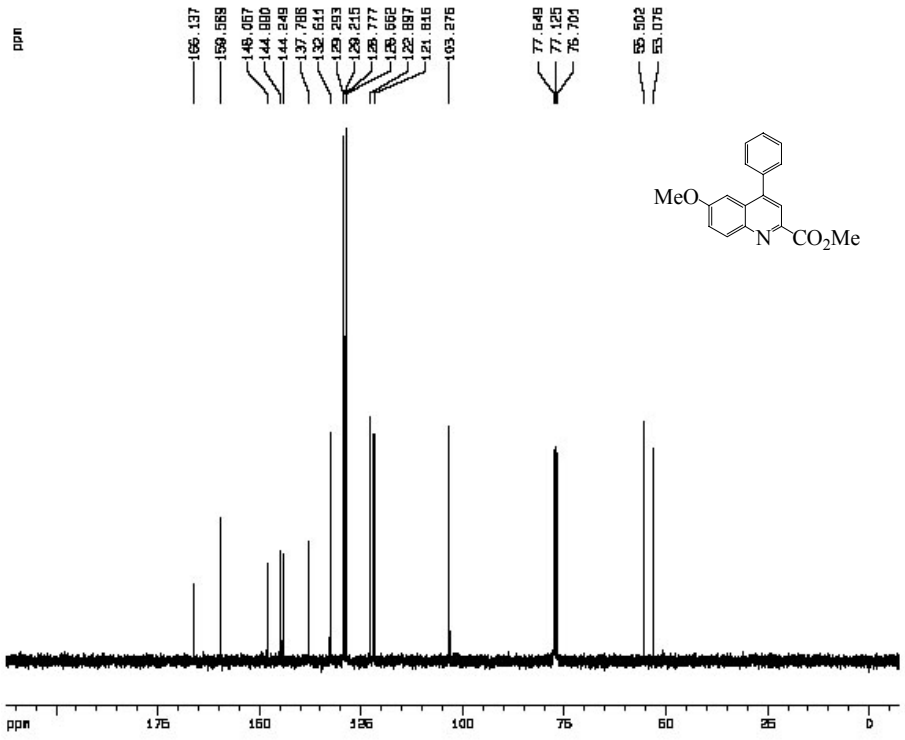

```
Current Data Parameters
NAME            wyc-0-002
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_          20031118
Time               3.09
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                90.5
DW               81.000 usec
DE                6.00 usec
TE               294.8 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1               9.30 usec
PL1              -1.00 dB
SFO1        300.1318634 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300025 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.60 cm
F1P               10.200 ppm
F1                3061.33 Hz
F2P               -0.800 ppm
F2                -60.03 Hz
PPMCM          0.52000 ppm/cm
HZCM          156.06761 Hz/cm
```




```
Current Data Parameters
NAME            wyc-0-002E
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_          20031118
Time               3.08
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  127
DS                    4
SWH           17985.611 Hz
FIDRES        0.274450 Hz
AQ            1.8219598 sec
RG                 256
DW               27.800 usec
DE                6.00 usec
TE               298.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

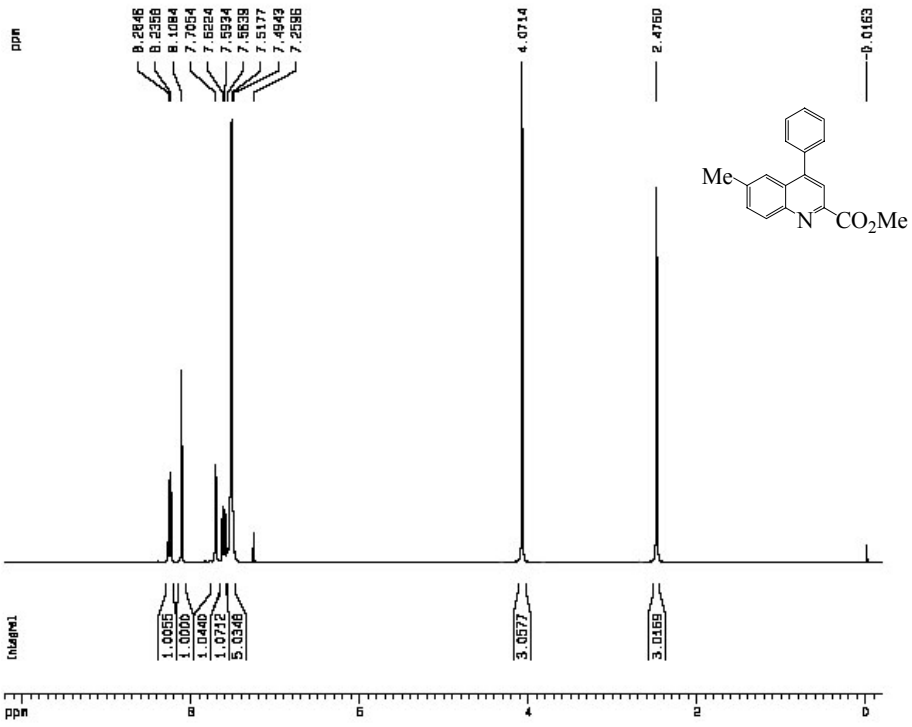
======== CHANNEL f1 ========
NUC1                13C
P1               9.40 usec
PL1              -1.00 dB
SFO1        75.4762953 MHz

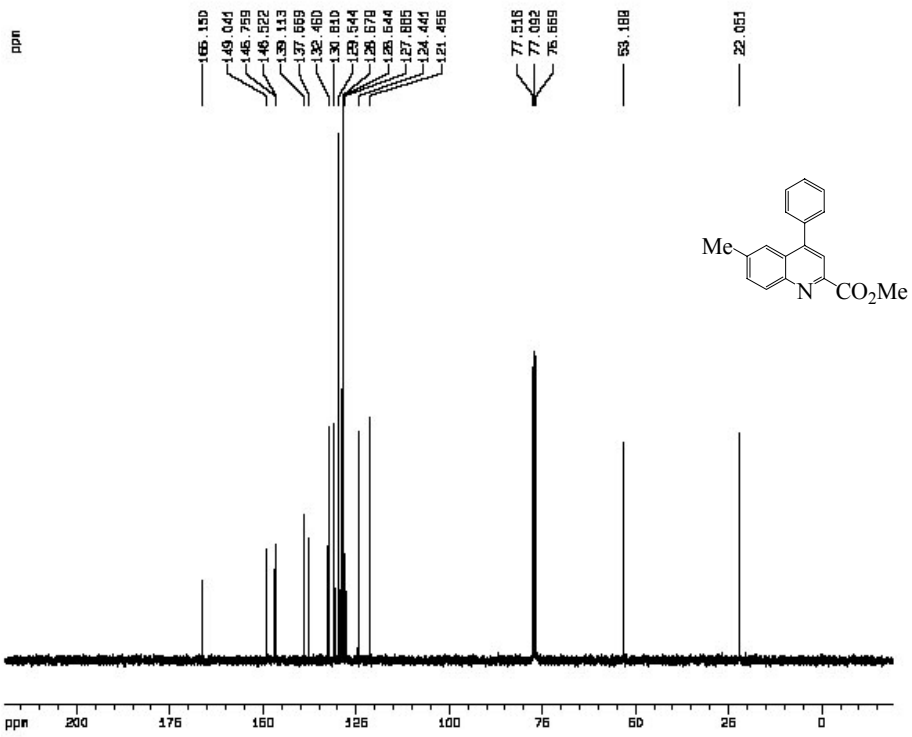
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             16.00 dB
PL13             16.00 dB
SFO2        300.1318000 MHz

F2 - Processing parameters
SI                 32768
SF          75.4677460 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P              212.068 ppm
F1             16005.63 Hz
F2P               -7.386 ppm
F2              -557.28 Hz
PPMCM         10.57207 ppm/cm
HZCM          839.18100 Hz/cm
```

Methyl 6-methyl-4-phenylquinoline-2-carboxylate (3c)





Current Data Parameters
NAME wyc-6-012
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031114
Time 5.26
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.835 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 143.7
DW 96.000 usec
DE 6.00 usec
TE 283.0 K
D1 2.00000000 sec

CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1316834 MHz

F2 - Processing parameters
SI 32768
SF 300.1300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.60 cm
F1P 10.200 ppm
F1 3061.33 Hz
F2P -0.800 ppm
F2 -60.03 Hz
PPMCM 0.52000 ppm/cm
HZCM 156.06751 Hz/cm

Current Data Parameters
NAME wyc-6-012
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031114
Time 10.42
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 260
DS 4
SWH 17980.611 Hz
FIDRES 0.274450 Hz
AQ 1.8218598 sec
RG 645.1
DW 27.800 usec
DE 6.00 usec
TE 293.6 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

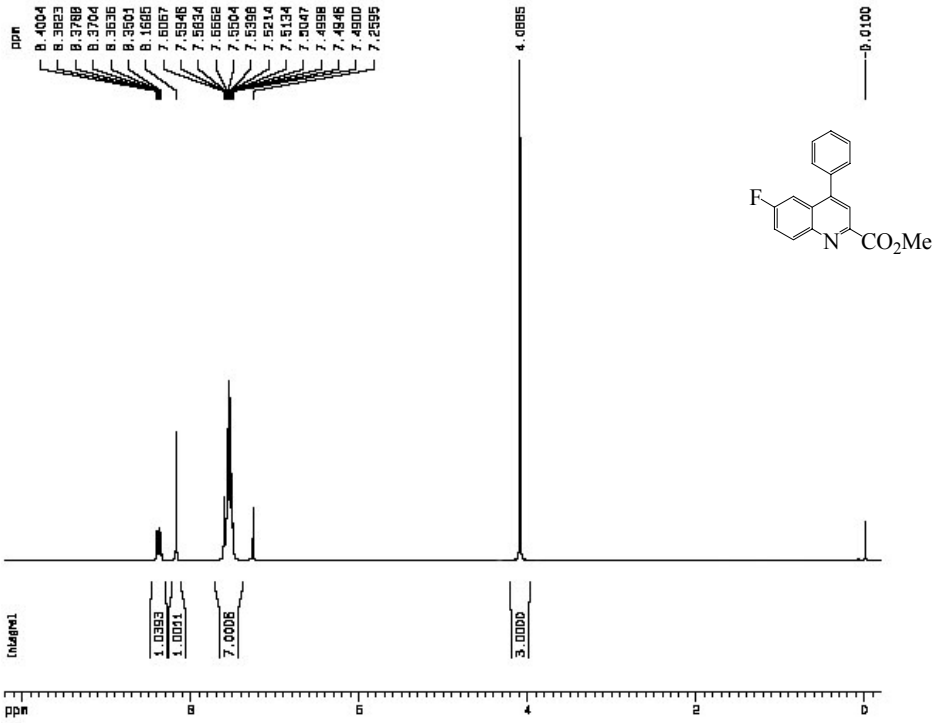
CHANNEL f1
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677460 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.60 cm
F1P 210.455 ppm
F1 15838.11 Hz
F2P -16.167 ppm
F2 -1446.01 Hz
PPMCM 11.91809 ppm/cm
HZCM 896.86058 Hz/cm

Methyl 6-fluoro-4-phenylquinoline-2-carboxylate (3d)



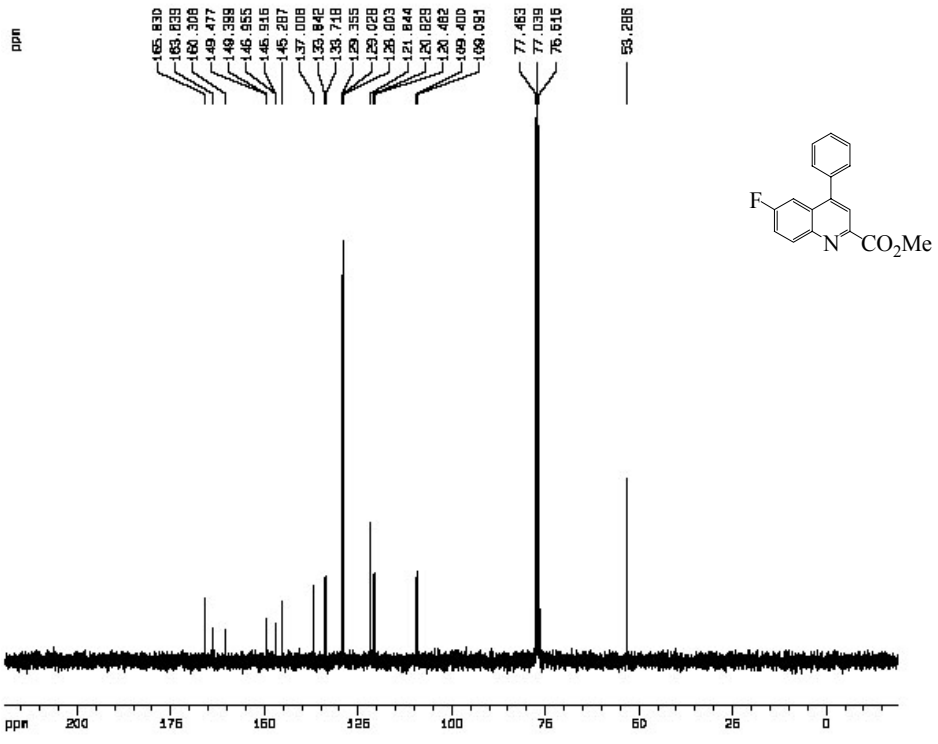

```
Current Data Parameters
NAME            wyc-13-26
EXPNO                  80
PROCNO                  1

F2 - Acquisition Parameters
Date_         20080329
Time               4.07
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                  362
DW              81.000 usec
DE               6.00 usec
TE              286.9 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1               9.30 usec
PL1             -1.00 dB
SFO1        300.1318634 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300029 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.60 cm
F1P              10.200 ppm
F1              3061.33 Hz
F2P              -0.800 ppm
F2               -80.03 Hz
PPMCM          0.52000 ppm/cm
HZCM          156.05761 Hz/cm
```




```
Current Data Parameters
NAME            wyc-13-26
EXPNO                  22
PROCNO                  1

F2 - Acquisition Parameters
Date_         20080324
Time               21.07
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  200
DS                    4
SWH          17985.611 Hz
FIDRES        0.274450 Hz
AQ            1.8249598 sec
RG                 4096
DW              27.800 usec
DE               6.00 usec
TE              293.2 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

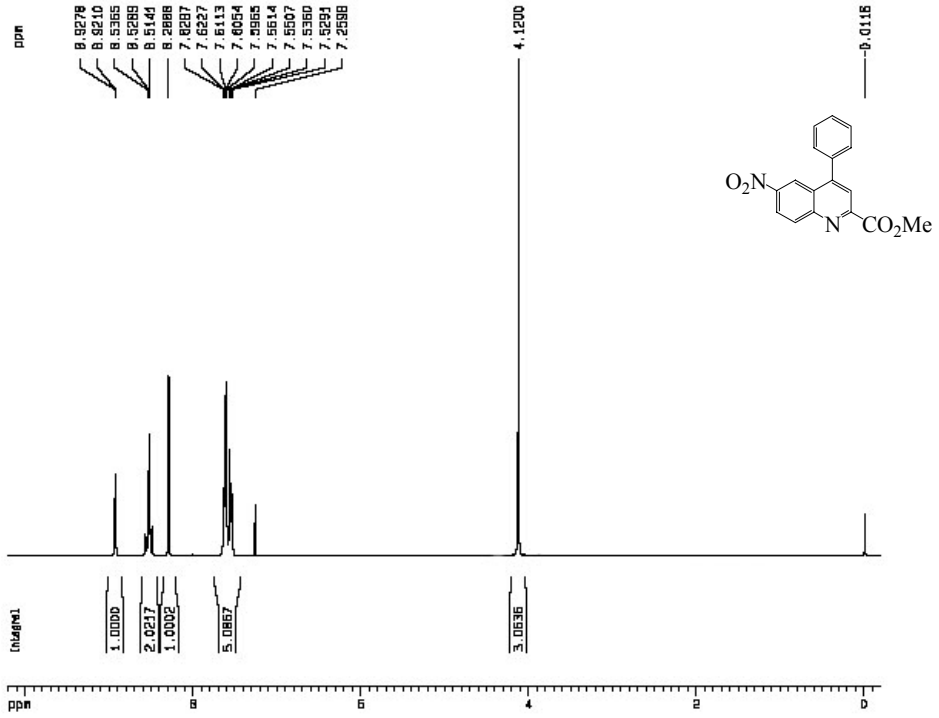
======== CHANNEL f1 ========
NUC1                13C
P1               9.40 usec
PL1             -1.00 dB
SFO1        75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           80.00 usec
PL2             -1.00 dB
PL12            18.00 dB
PL13            18.00 dB
SFO2        300.1318000 MHz

F2 - Processing parameters
SI                 32768
SF          75.4677460 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                12.60 cm
F1P             210.155 ppm
F1            15839.11 Hz
F2P             -10.167 ppm
F2            -1446.01 Hz
PPMCM         11.91809 ppm/cm
HZCM          898.89058 Hz/cm
```

Methyl 6-nitro-4-phenylquinoline-2-carboxylate (3e)



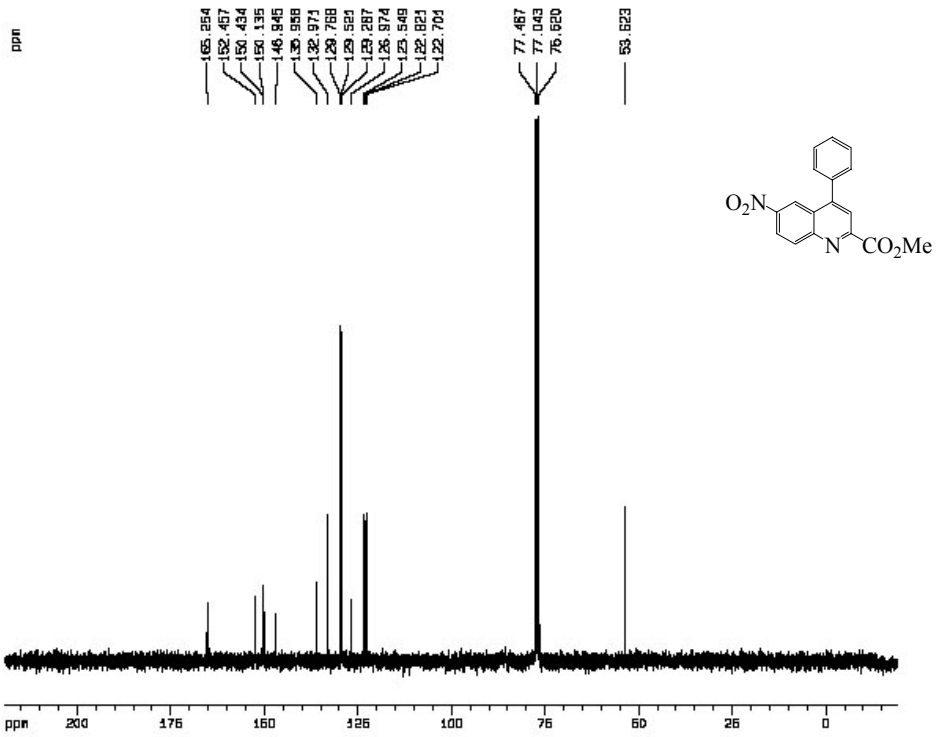

```
Current Data Parameters
NAME            wyc-6-54
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20091119
Time                3.32
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                322.5
DW               81.000 usec
DE                6.00 usec
TE               294.7 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.30 usec
PL1              -1.00 dB
SFO1        300.1318634 MHz

F2 - Processing parameters
SI                32768
SF          300.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P              10.200 ppm
F1               3061.33 Hz
F2P              -0.800 ppm
F2               -60.03 Hz
PPMCM            0.52000 ppm/cm
HZCM           156.06764 Hz/cm
```




```
Current Data Parameters
NAME            wyc-6-54
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_          20091118
Time                2.28
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  248
DS                    4
SWH           17985.611 Hz
FIDRES        0.274450 Hz
AQ            1.8219598 sec
RG                812.3
DW               27.800 usec
DE                6.00 usec
TE               298.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

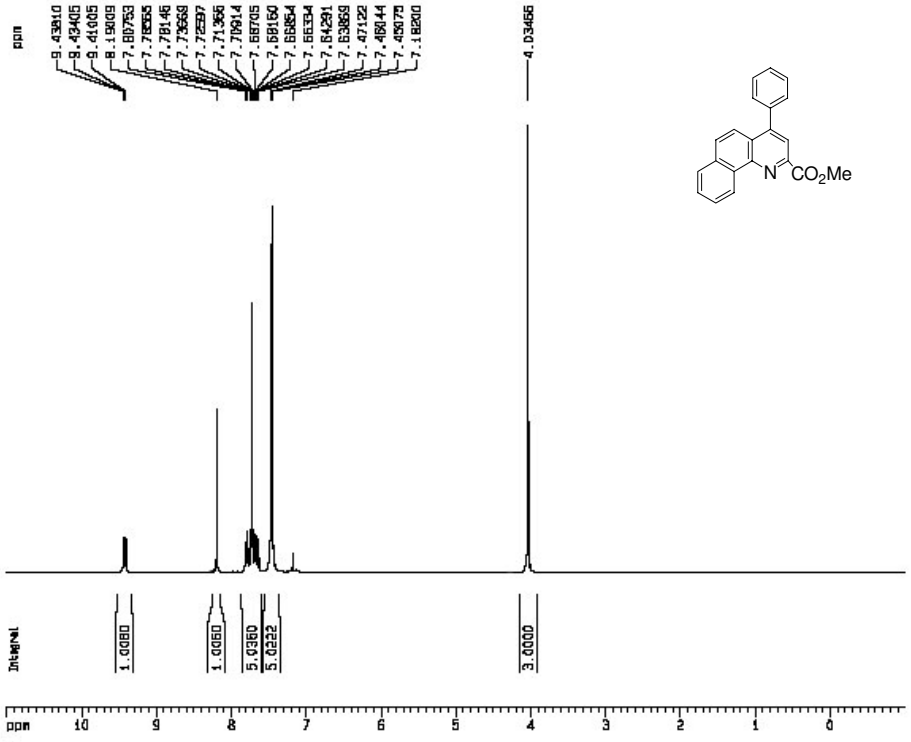
======== CHANNEL f1 ========
NUC1                13C
P1                9.40 usec
PL1              -1.00 dB
SFO1        75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        300.1318000 MHz

F2 - Processing parameters
SI                32768
SF          75.4677400 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P             210.155 ppm
F1            15839.11 Hz
F2P             -18.187 ppm
F2            -1446.01 Hz
PPMCM          11.91809 ppm/cm
HZCM          898.85058 Hz/cm
```

Methyl 4-phenylbenzo[*h*]quinoline-2-carboxylate (3f)



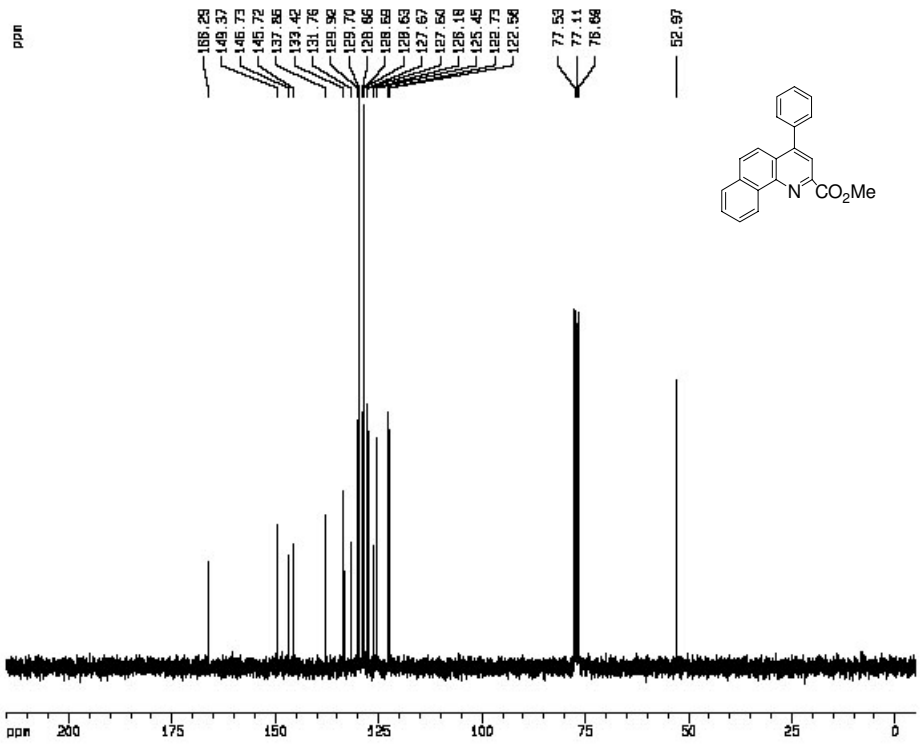

```
Current Data Parameters
NAME          wyc-15-19a
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20050822
Time               4.53
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084950 sec
RG                 114
DW              81.000 usec
DE                6.00 usec
TE               673.2 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.30 usec
PL1              -1.00 dB
SFO1        300.1316534 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300295 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               11.000 ppm
F1              3301.43 Hz
F2P               -1.000 ppm
F2              -300.13 Hz
PPMCM             0.50000 ppm/cm
HZCM            150.07802 Hz/cm
```




```
Current Data Parameters
NAME          wyc-15-19c
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_          20050822
Time               4.57
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG        zgpg30
TD               65536
SOLVENT          CDCl3
NS                  48
DS                   4
SWH          17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219808 sec
RG                1024
DW              27.800 usec
DE                6.00 usec
TE               673.2 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

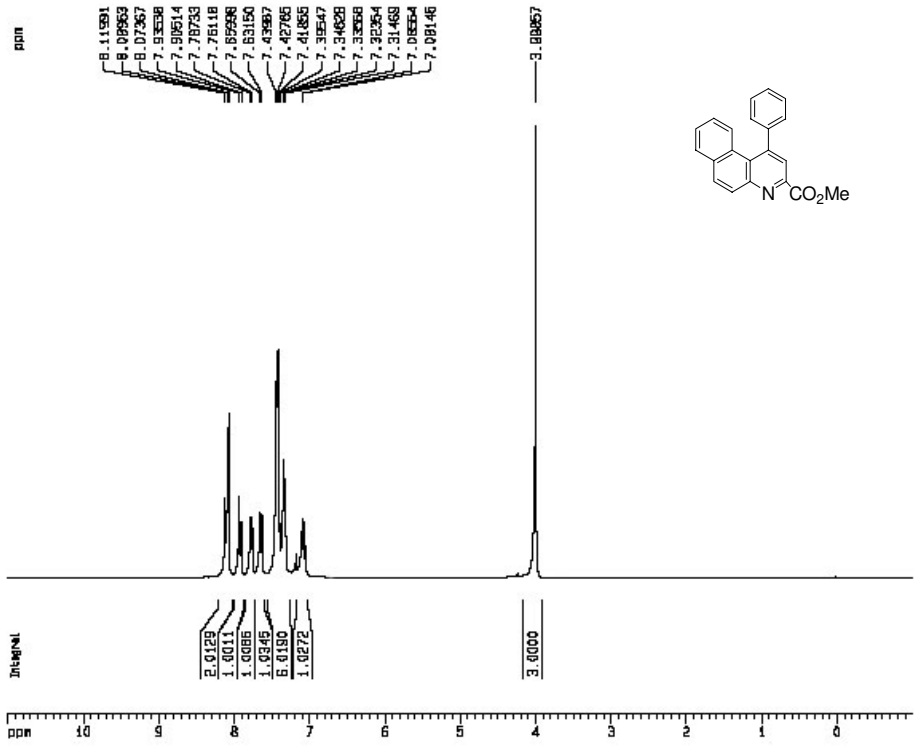
======== CHANNEL f1 ========
NUC1                13C
P1                9.40 usec
PL1              -1.00 dB
SFO1        75.4752903 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -1.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2        300.1312008 MHz

F2 - Processing parameters
SI                 32768
SF          75.4877480 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                 12.00 cm
F1P              218.000 ppm
F1             16325.07 Hz
F2P               -5.000 ppm
F2              -377.34 Hz
PPMCM            11.00000 ppm/cm
HZCM           830.14520 Hz/cm
```

Methyl 1-phenylbenzo[*f*]quinoline-3-carboxylate (3g)



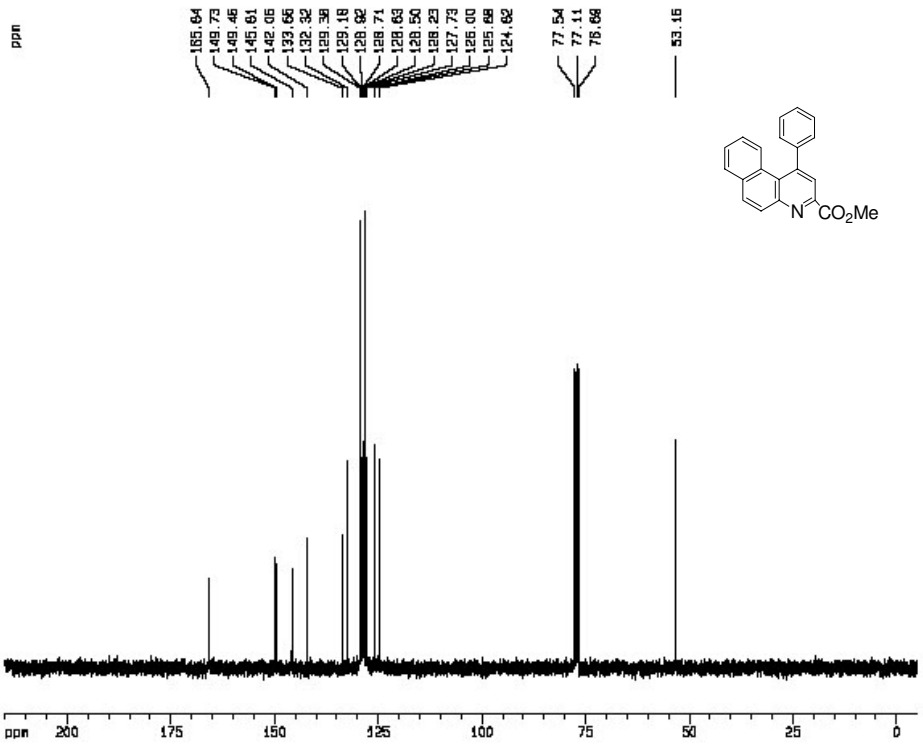

```
Current Data Parameters
NAME          wyc-15-19f
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20090822
Time               9.05
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           6172.839 Hz
FIDRES        0.094159 Hz
AQ            5.3084950 sec
RG                 114
DW              81.000 usec
DE               6.00 usec
TE             673.2 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1               9.30 usec
PL1              -1.00 dB
SFO1        300.1316534 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300235 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               11.000 ppm
F1                3301.43 Hz
F2P               -1.000 ppm
F2                -300.13 Hz
PPMCM              0.60000 ppm/cm
HZCM             180.07802 Hz/cm
```




```
Current Data Parameters
NAME          wyc-15-19f
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_          20090822
Time               5.10
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG        zgpg30
TD               65536
SOLVENT          CDCl3
NS                 141
DS                   4
SWH          17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219008 sec
RG                4088
DW              27.800 usec
DE               6.00 usec
TE             673.2 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

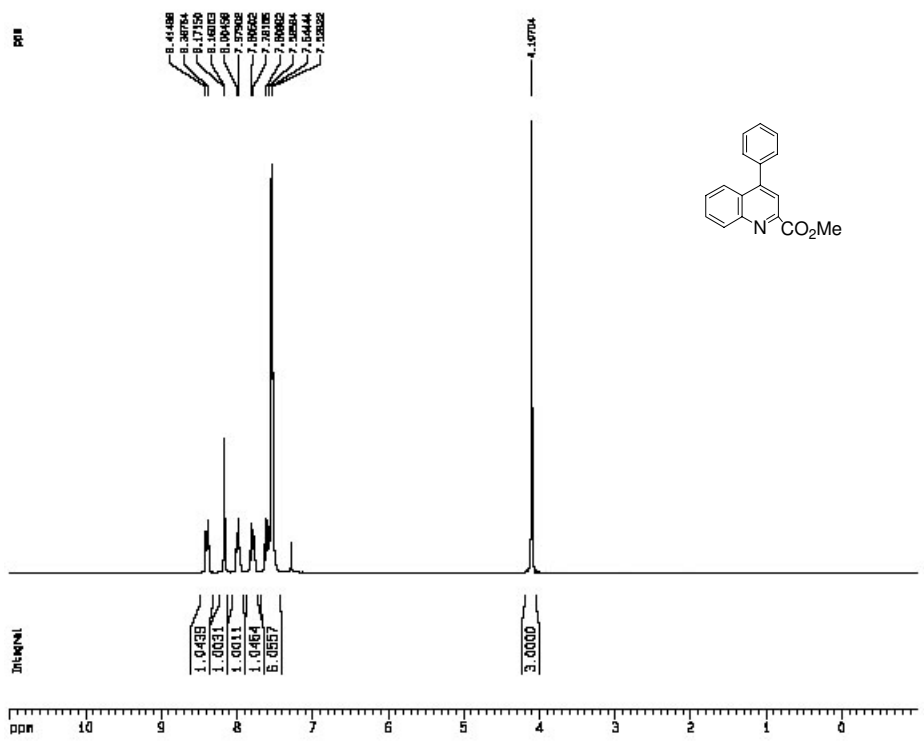
======== CHANNEL f1 ========
NUC1                13C
P1               9.40 usec
PL1              -1.00 dB
SFO1        75.4752903 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        300.1312008 MHz

F2 - Processing parameters
SI                 32768
SF          75.4877480 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P              215.000 ppm
F1             16225.87 Hz
F2P               -5.000 ppm
F2              -377.34 Hz
PPMCM             11.00000 ppm/cm
HZCM           830.14520 Hz/cm
```

Methyl 4-phenylquinoline-2-carboxylate (3h)



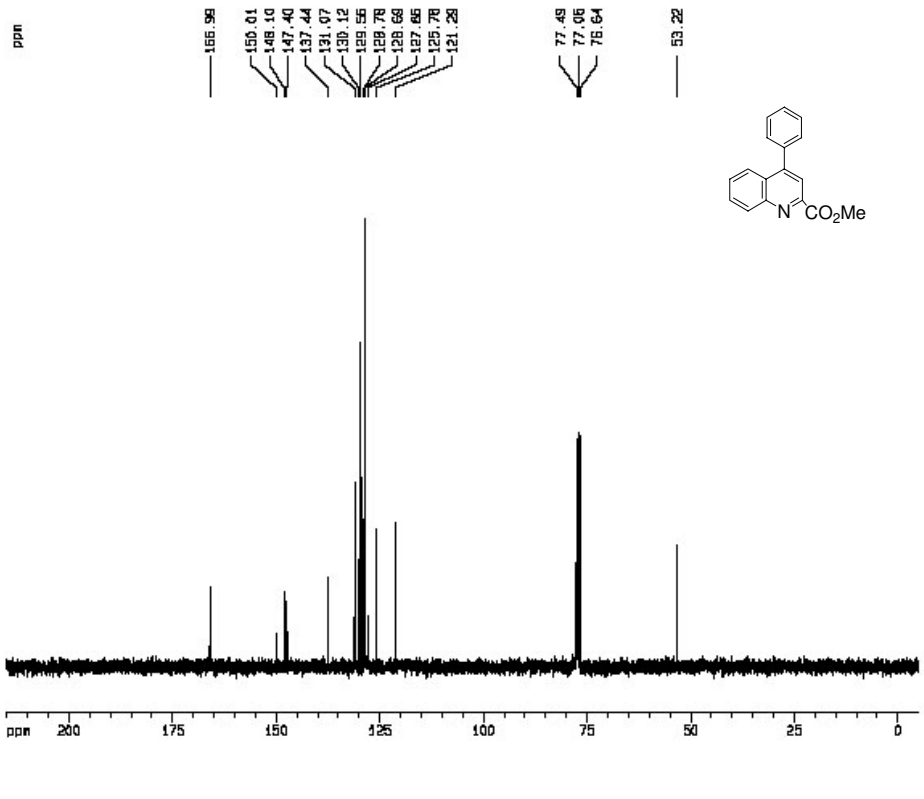

```
Current Data Parameters
NAME          wyc-15-19a
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20050822
Time               3.45
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG           zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084950 sec
RG                161.3
DW               81.000 usec
DE                6.00 usec
TE               673.2 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.30 usec
PL1              -1.00 dB
SFO1        300.1316534 MHz

F2 - Processing parameters
SI                32768
SF          300.1300000 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00

1D NMR plot parameters
CX                20.00 cm
CY                19.00 cm
F1P              11.000 ppm
F1             3301.43 Hz
F2P              -1.000 ppm
F2             -300.13 Hz
PPMCM             0.60000 ppm/cm
HZCM            180.07800 Hz/cm
```




```
Current Data Parameters
NAME          wyc-15-19a
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_          20050822
Time               3.48
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG        zgpg30
TD                65536
SOLVENT           CDCl3
NS                  117
DS                    4
SWH          17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                4567.8
DW               27.800 usec
DE                6.00 usec
TE               673.2 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

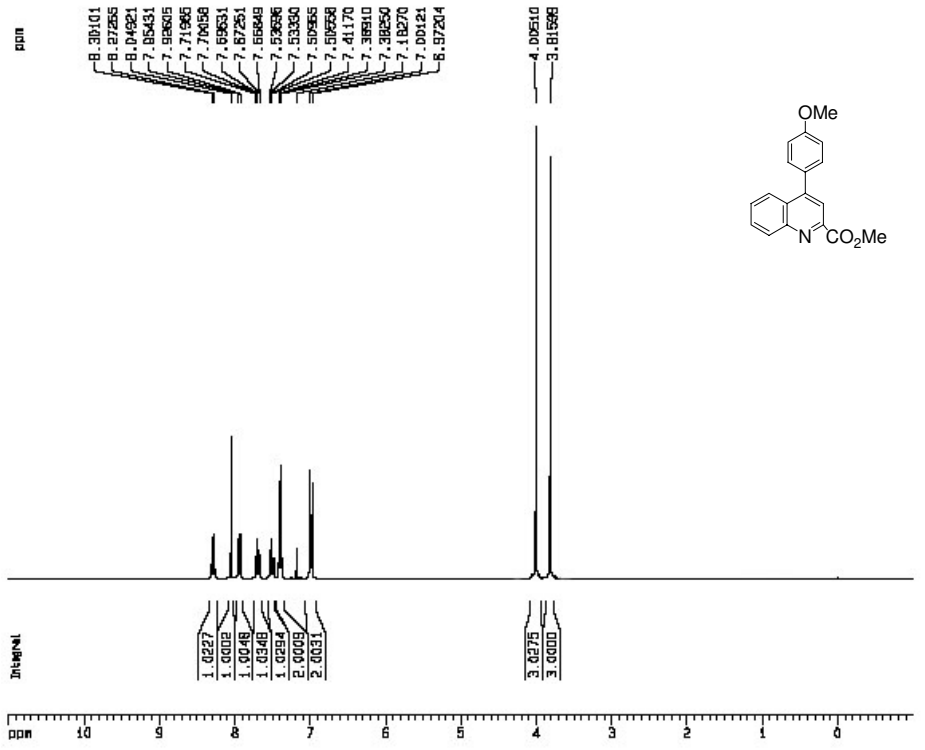
======== CHANNEL f1 ========
NUC1                13C
P1                9.40 usec
PL1              -1.00 dB
SFO1        75.4752903 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        300.1312008 MHz

F2 - Processing parameters
SI                32768
SF          75.4877480 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
CY                19.00 cm
F1P             210.000 ppm
F1          15855.87 Hz
F2P              -5.000 ppm
F2            -377.24 Hz
PPMCM            11.00000 ppm/cm
HZCM           830.14520 Hz/cm
```

Methyl 4-(4-methoxyphenyl)quinoline-2-carboxylate (3i)



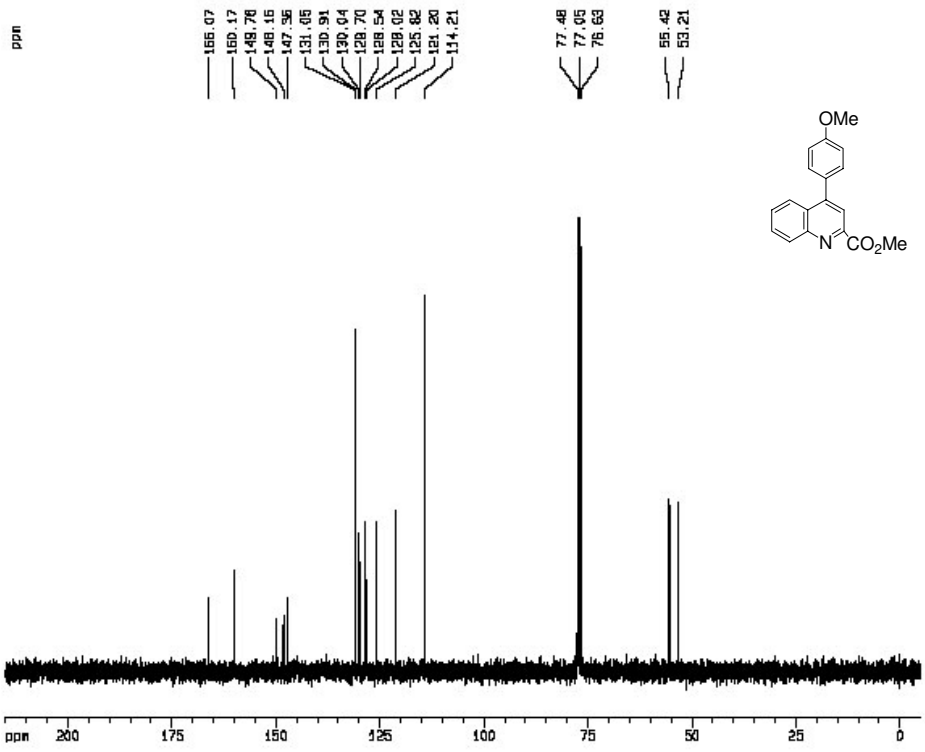
Current Data Parameters
NAME wyc-15-19c
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050822
Time 4.20
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084650 sec
RG 828.1
DW 81.000 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec

CHANNEL f1
NUC1 1H
P1 9.50 usec
PL1 -1.00 dB
SFO1 300.1316534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300295 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.07802 Hz/cm



Current Data Parameters
NAME wyc-15-19c
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050822
Time 4.26
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 89
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 3640.1
DW 27.800 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

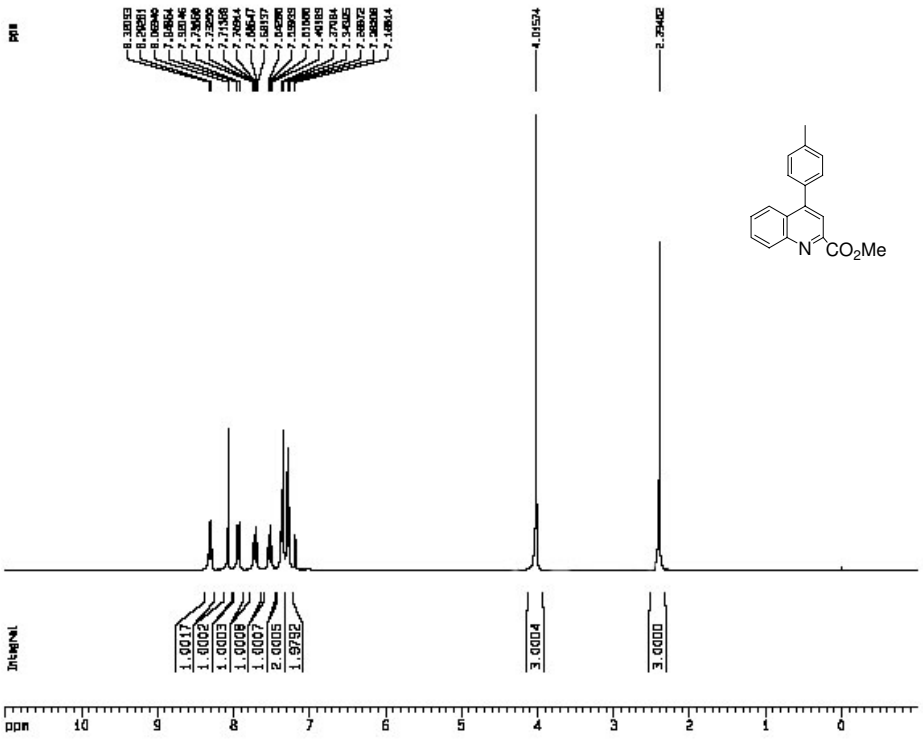
CHANNEL f1
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SFO1 75.4752963 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1312008 MHz

F2 - Processing parameters
SI 32768
SF 75.4877480 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 210.000 ppm
F1 15825.07 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14520 Hz/cm

Methyl 4-*p*-tolylquinoline-2-carboxylate (3j)



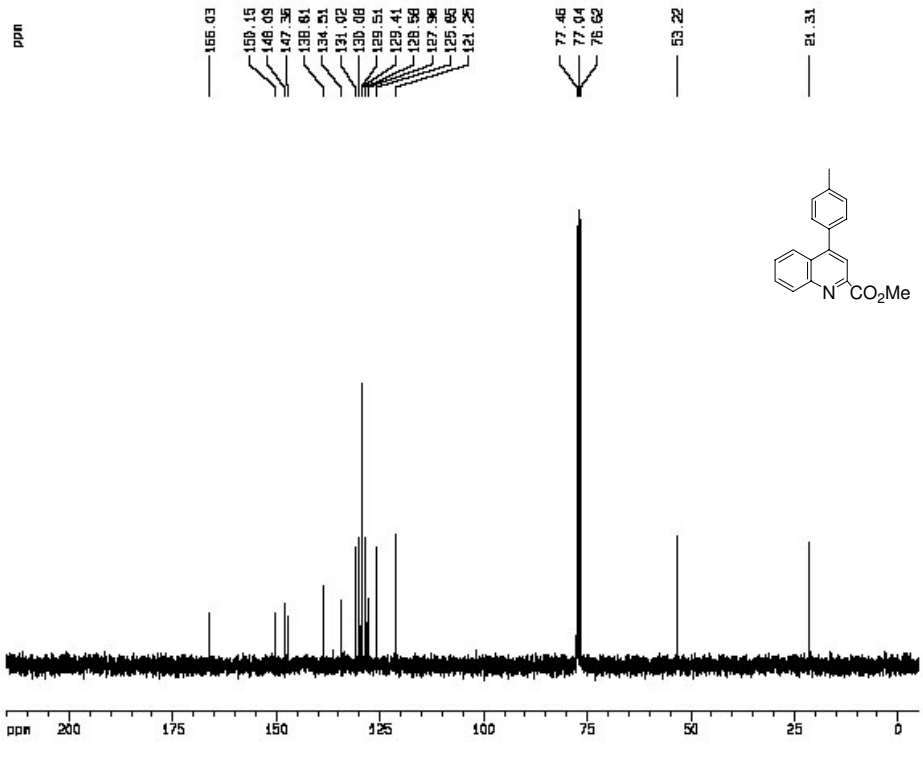

```
Current Data Parameters
NAME          wyc-15-19b
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20090922
Time               4.01
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084960 sec
RG                 057.4
DW               81.000 usec
DE                6.00 usec
TE                673.2 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.30 usec
PL1              -1.00 dB
SFO1        300.1318634 MHz

F2 - Processing parameters
SI                32768
SF          300.1300294 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P               11.000 ppm
F1                3301.43 Hz
F2P               -1.000 ppm
F2                -300.13 Hz
PPMCM          0.60000 ppm/cm
HZCM          180.07802 Hz/cm
```




```
Current Data Parameters
NAME          wyc-15-19b
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_          20090922
Time               4.08
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  137
DS                    4
SWH          17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219008 sec
RG                 1024
DW               27.800 usec
DE                6.00 usec
TE                673.2 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

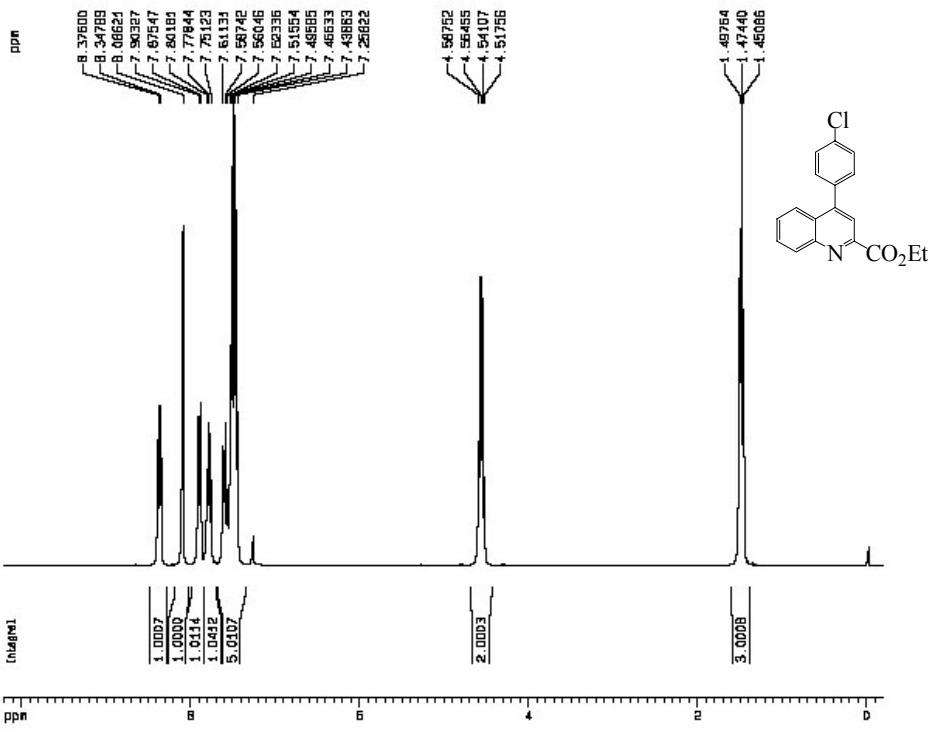
======== CHANNEL f1 ========
NUC1                13C
P1                9.40 usec
PL1              -1.00 dB
SFO1        75.4752963 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2              -1.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2        300.1312000 MHz

F2 - Processing parameters
SI                32768
SF          75.4877480 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P              215.000 ppm
F1             16220.67 Hz
F2P               -5.000 ppm
F2              -377.24 Hz
PPMCM         11.00000 ppm/cm
HZCM          830.14520 Hz/cm
```

Ethyl 4-(4-chlorophenyl)quinoline-2-carboxylate (3k)



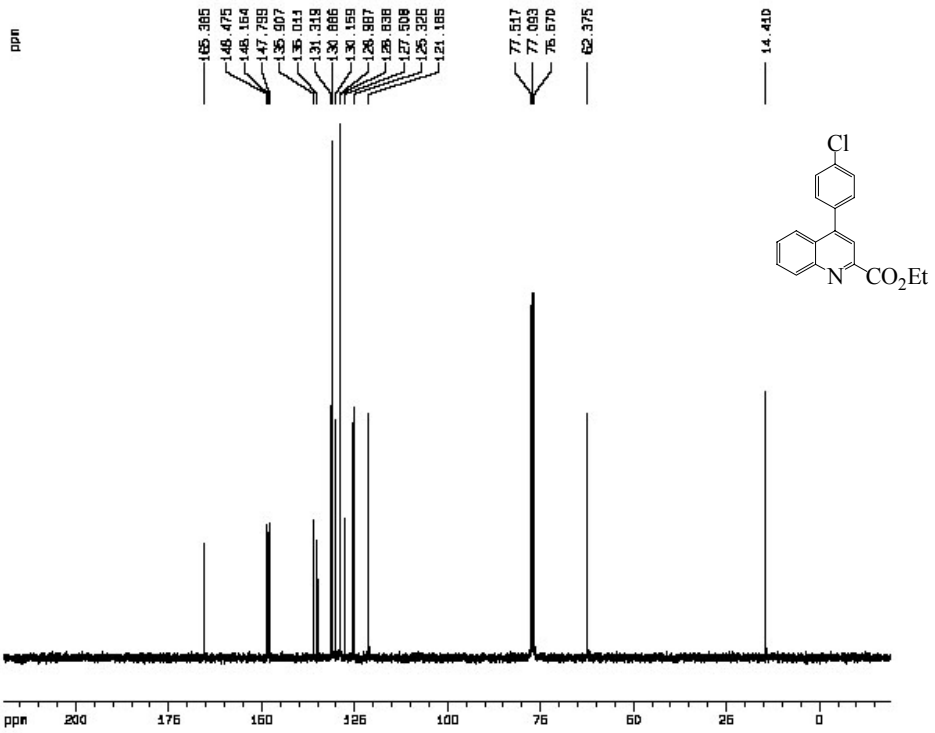

```
Current Data Parameters
NAME                wyc-8-28
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20040226
Time                   3.37
INSTRUM                av300
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    86638
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                5172.839 Hz
FIDRES             0.094190 Hz
AQ                 5.3084660 sec
RG                      128
DW                   84.000 usec
DE                    6.00 usec
TE                  282.7 K
D1                 2.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                      9.30 usec
PL1                    -1.00 dB
SFO1            300.1318634 MHz

F2 - Processing parameters
SI                        32768
SF                 300.1300085 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      20.00 cm
CY                      12.60 cm
F1P                     10.200 ppm
F1                    3061.33 Hz
F2P                    -0.200 ppm
F2                     -60.03 Hz
PPMCM                 0.52000 ppm/cm
HZCM                156.06761 Hz/cm
```




```
Current Data Parameters
NAME                wyc-8-28
EXPNO                     11
PROCNO                     1

F2 - Acquisition Parameters
Date_              20040226
Time                   3.44
INSTRUM                av300
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      300
DS                        4
SWH                17980.611 Hz
FIDRES             0.274450 Hz
AQ                 1.8218598 sec
RG                    2580.3
DW                   27.800 usec
DE                    6.00 usec
TE                  293.2 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

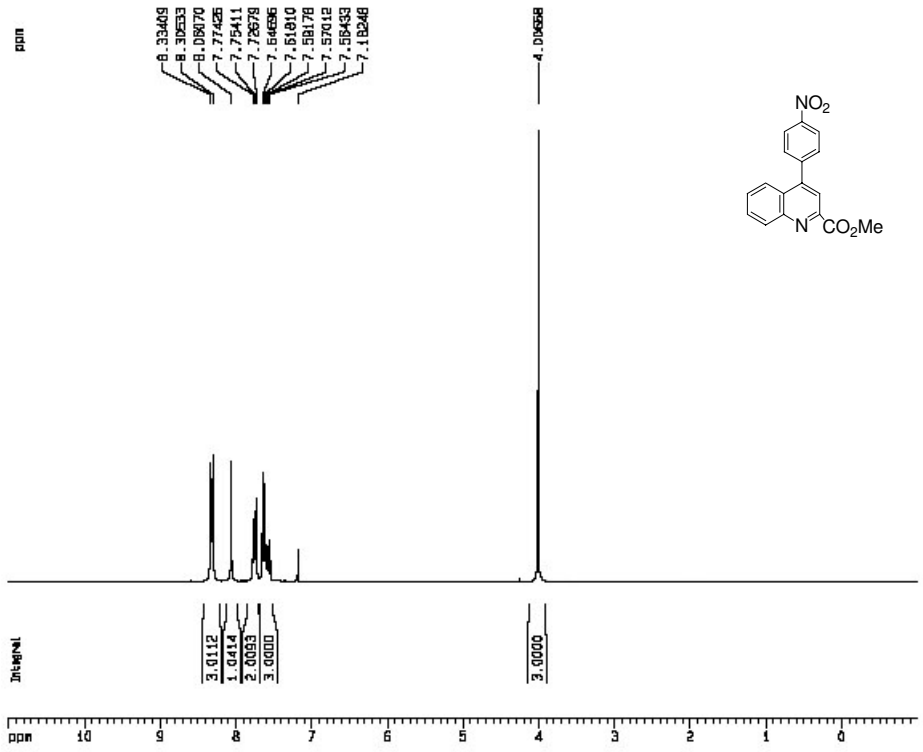
======== CHANNEL f1 ========
NUC1                      13C
P1                      9.40 usec
PL1                    -1.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                    -1.00 dB
PL12                   18.00 dB
PL13                   18.00 dB
SFO2            300.1318000 MHz

F2 - Processing parameters
SI                        32768
SF                 75.4677460 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                      20.00 cm
CY                      12.60 cm
F1P                    210.155 ppm
F1                   15839.11 Hz
F2P                   -18.187 ppm
F2                   -1446.01 Hz
PPMCM                11.51859 ppm/cm
HZCM                 868.86058 Hz/cm
```

Methyl 4-(4-nitrophenyl)quinoline-2-carboxylate (3l)



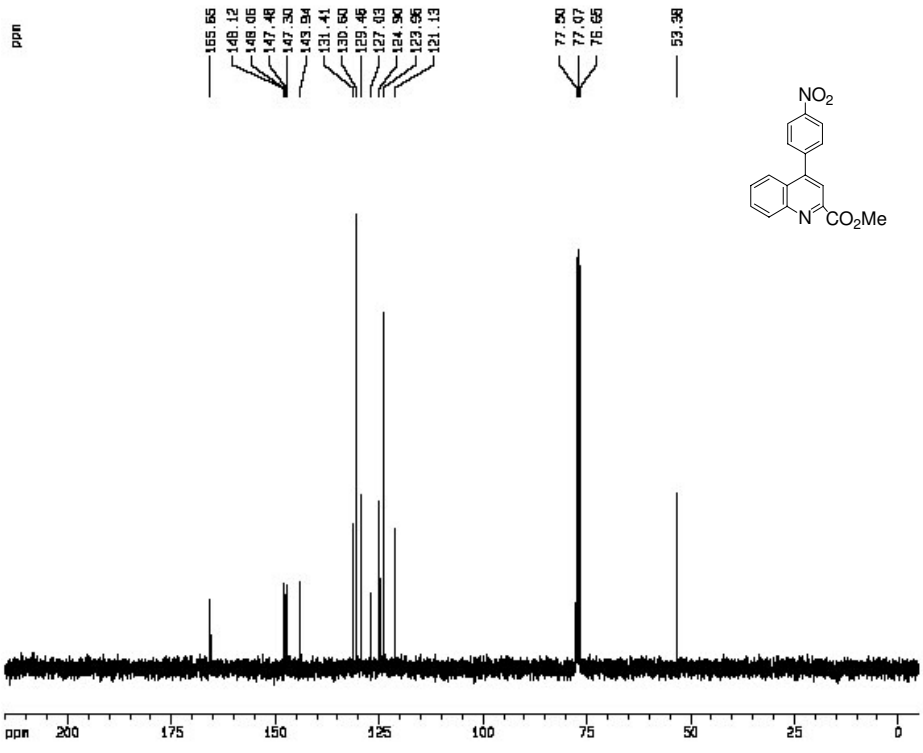

```
Current Data Parameters
NAME          wyc-15-19d
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20050822
Time               4.37
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   16
DS                    2
SWH           6172.836 Hz
FIDRES        0.094190 Hz
AQ            5.3084950 sec
RG                  181
DW             81.000 usec
DE              6.00 usec
TE             573.2 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1               9.30 usec
PL1             -1.00 dB
SFO1       300.1316534 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300295 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P              11.000 ppm
F1                3301.43 Hz
F2P              -1.000 ppm
F2                -300.13 Hz
PPMCM          0.60000 ppm/cm
HZCM          180.07802 Hz/cm
```




```
Current Data Parameters
NAME          wyc-15-19d
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_          20050822
Time               4.41
INSTRUM           av300
PROBHD  5 mm DUL 13C-1
PULPROG        zgpg30
TD               65536
SOLVENT          CDCl3
NS                   60
DS                    4
SWH          17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219008 sec
RG                1625.6
DW             27.800 usec
DE              6.00 usec
TE             573.2 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

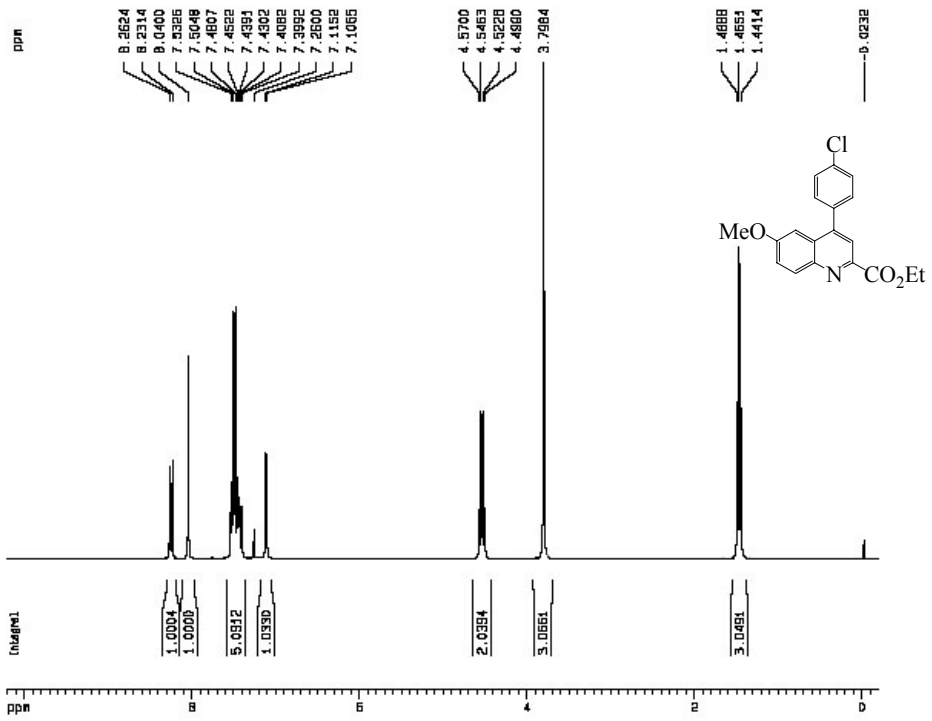
======== CHANNEL f1 ========
NUC1                13C
P1               9.40 usec
PL1             -1.00 dB
SFO1       75.4752903 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2             -1.00 dB
PL12            18.00 dB
PL13            18.00 dB
SFO2       300.1312008 MHz

F2 - Processing parameters
SI                 32768
SF          75.4877480 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P             210.000 ppm
F1             15825.87 Hz
F2P              -5.000 ppm
F2              -377.34 Hz
PPMCM          11.00000 ppm/cm
HZCM          829.14520 Hz/cm
```

Ethyl 4-(4-chlorophenyl)-6-methoxyquinoline-2-carboxylate (3m)



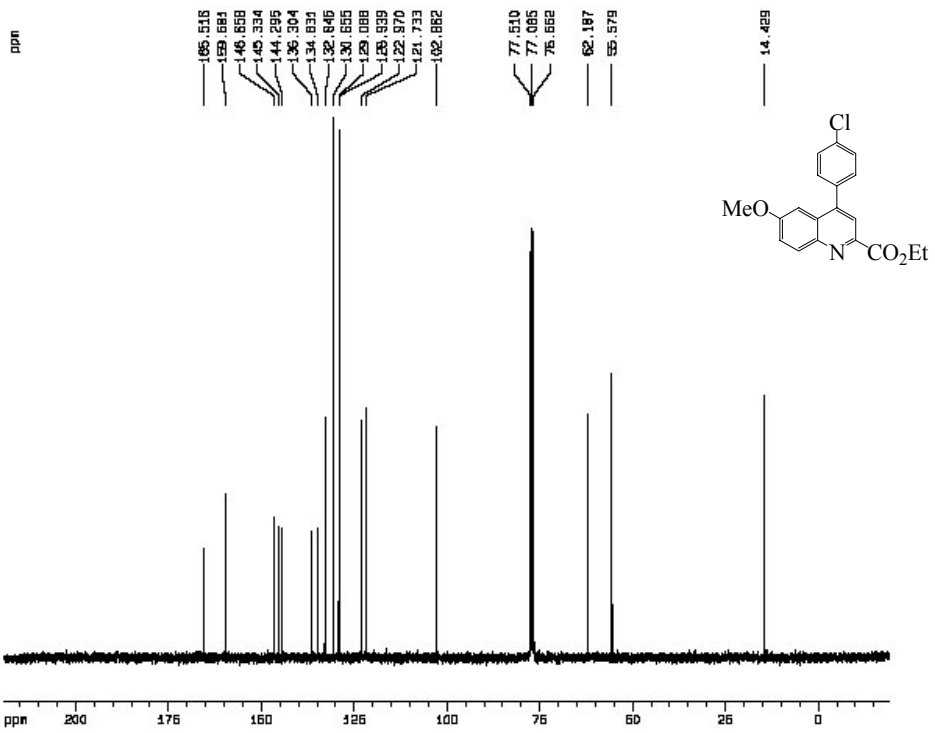

```
Current Data Parameters
NAME            wyc-8-87
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20031125
Time                21.24
INSTRUM           av300
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                143.7
DW                81.000 usec
DE                 6.00 usec
TE                281.5 K
D1              2.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.30 usec
PL1               -1.00 dB
SFO1        300.1318634 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300000 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.60 cm
F1P              10.200 ppm
F1              3061.33 Hz
F2P              -0.800 ppm
F2               -60.03 Hz
PPMCM            0.52000 ppm/cm
HZCM           156.06761 Hz/cm
```




```
Current Data Parameters
NAME            wyc-8-87
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20031128
Time                02.10
INSTRUM           av300
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   400
DS                     4
SWH            17980.631 Hz
FIDRES          0.274459 Hz
AQ              1.8248598 sec
RG                  256
DW                27.800 usec
DE                 6.00 usec
TE                298.2 K
D1              2.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

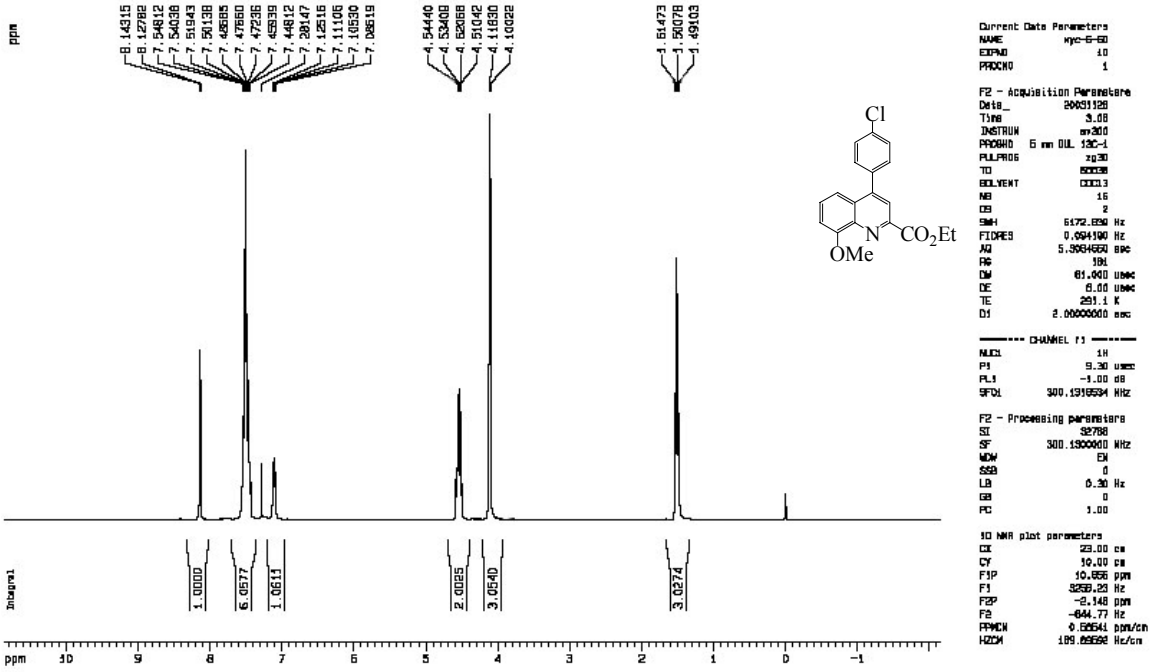
======== CHANNEL f1 ========
NUC1                  13C
P1                 9.40 usec
PL1               -1.00 dB
SFO1        75.4752853 MHz

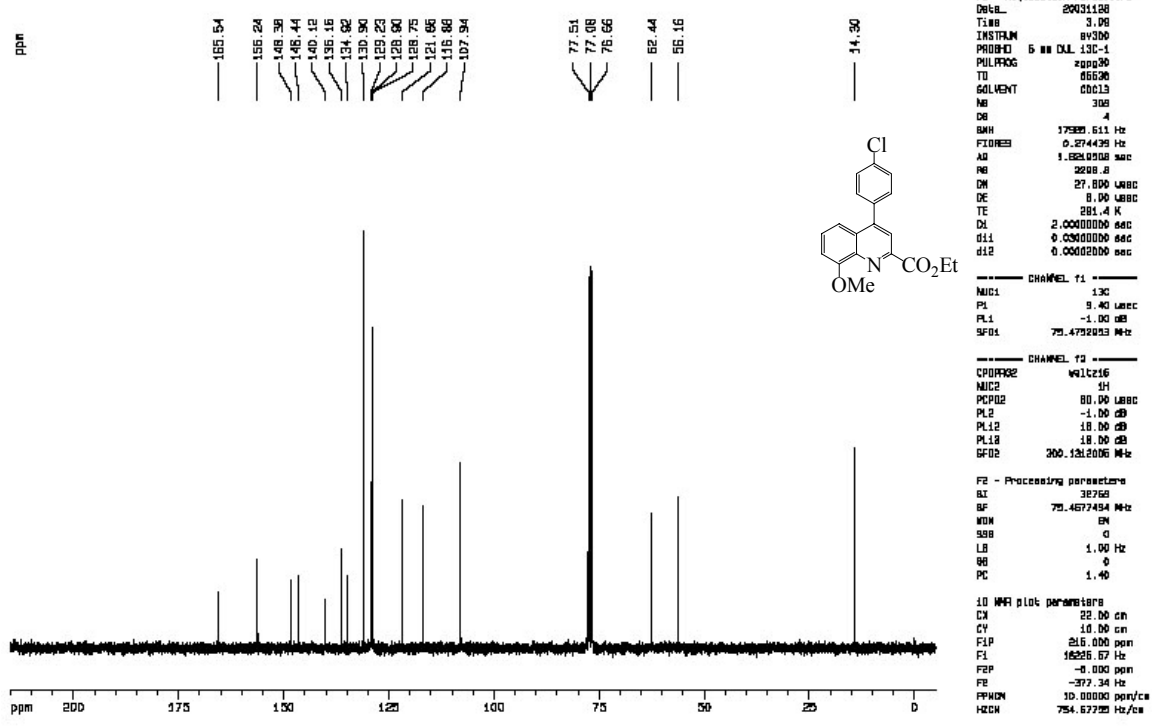
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -1.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2        300.1318000 MHz

F2 - Processing parameters
SI                 32768
SF          75.4677460 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40

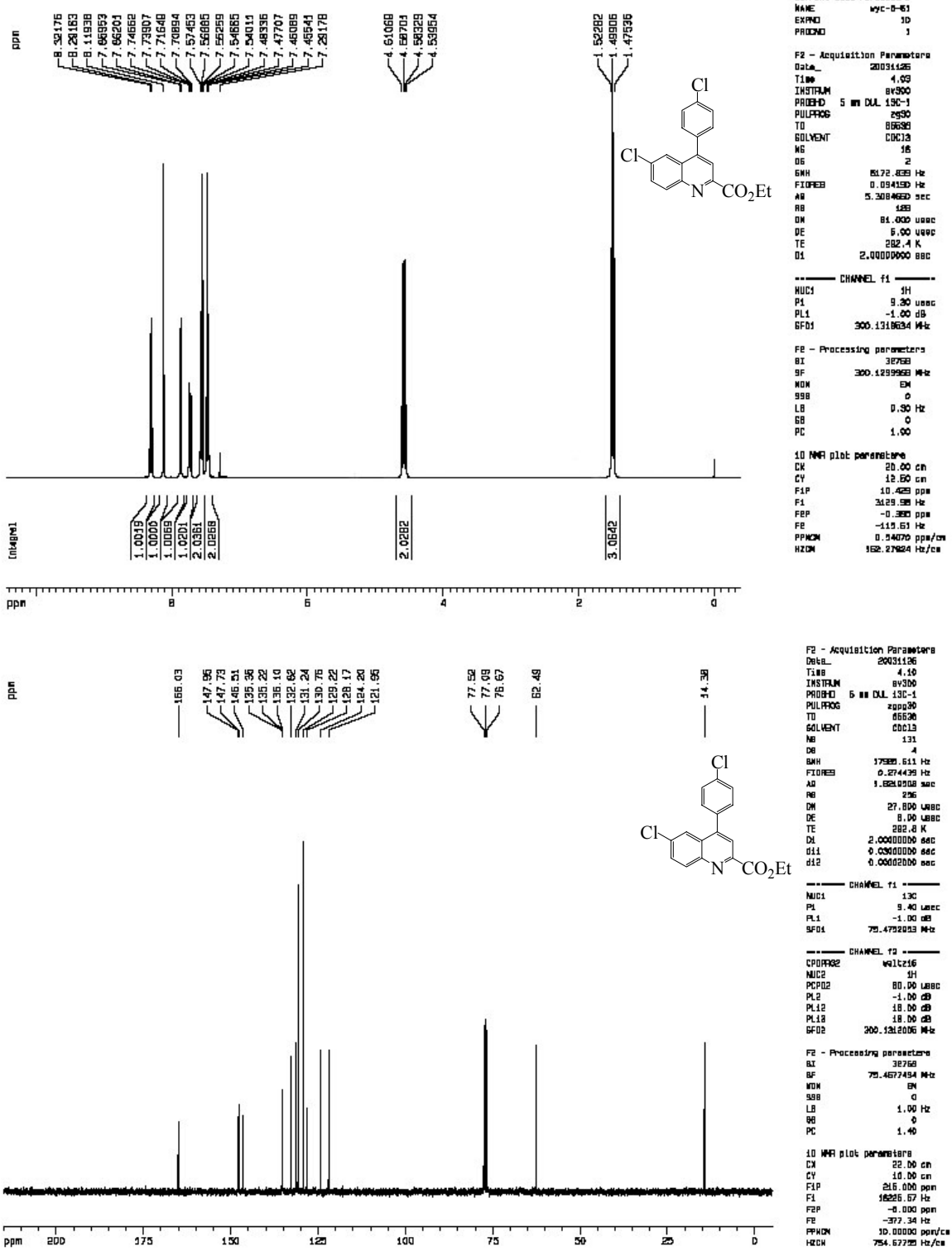
1D NMR plot parameters
CX                20.00 cm
CY                12.60 cm
F1P              240.155 ppm
F1             18039.11 Hz
F2P             -18.167 ppm
F2              -1446.01 Hz
PPMCM           11.91905 ppm/cm
HZCM           898.86068 Hz/cm
```

Ethyl 4-(4-chlorophenyl)-8-methoxyquinoline-2-carboxylate (3n)

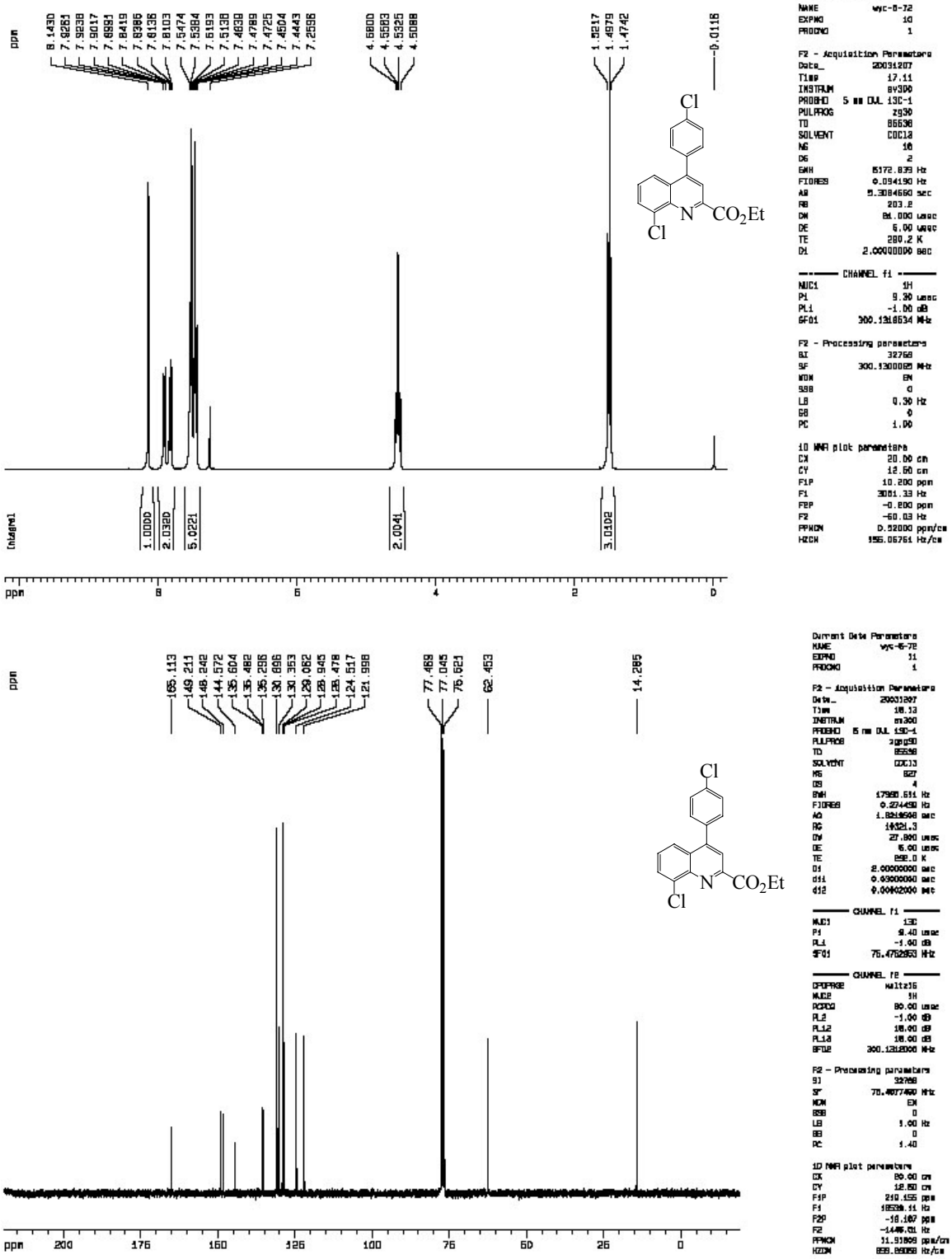




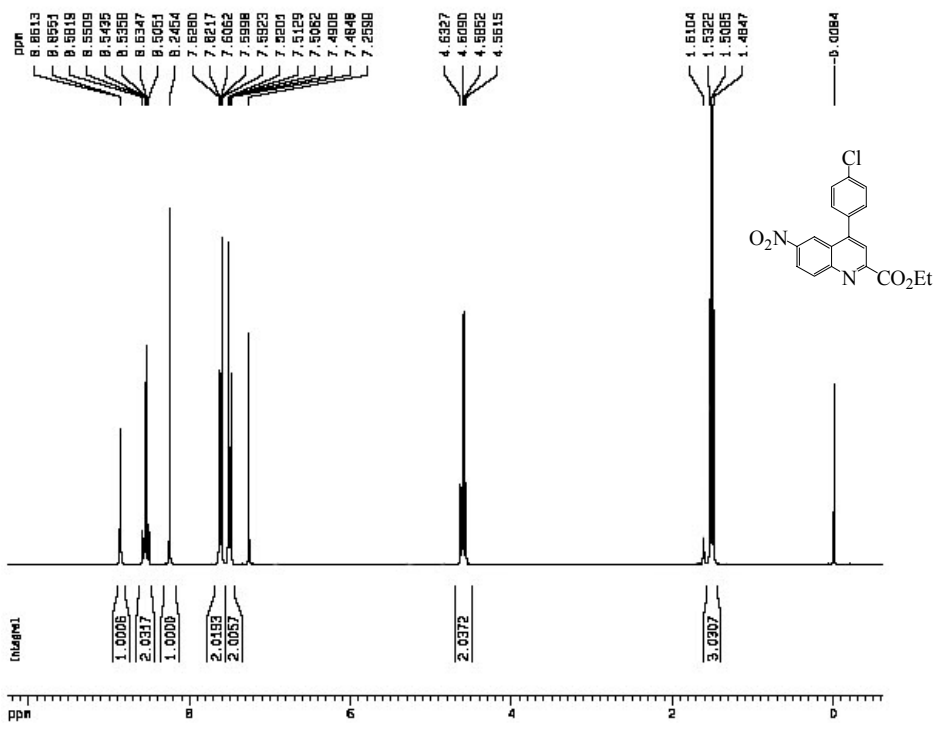
Ethyl 6-chloro-4-(4-chlorophenyl)quinoline-2-carboxylate (3o)



Ethyl 8-chloro-4-(4-chlorophenyl)quinoline-2-carboxylate (3p)



Ethyl 4-(4-chlorophenyl)-6-nitroquinoline-2-carboxylate (3q)



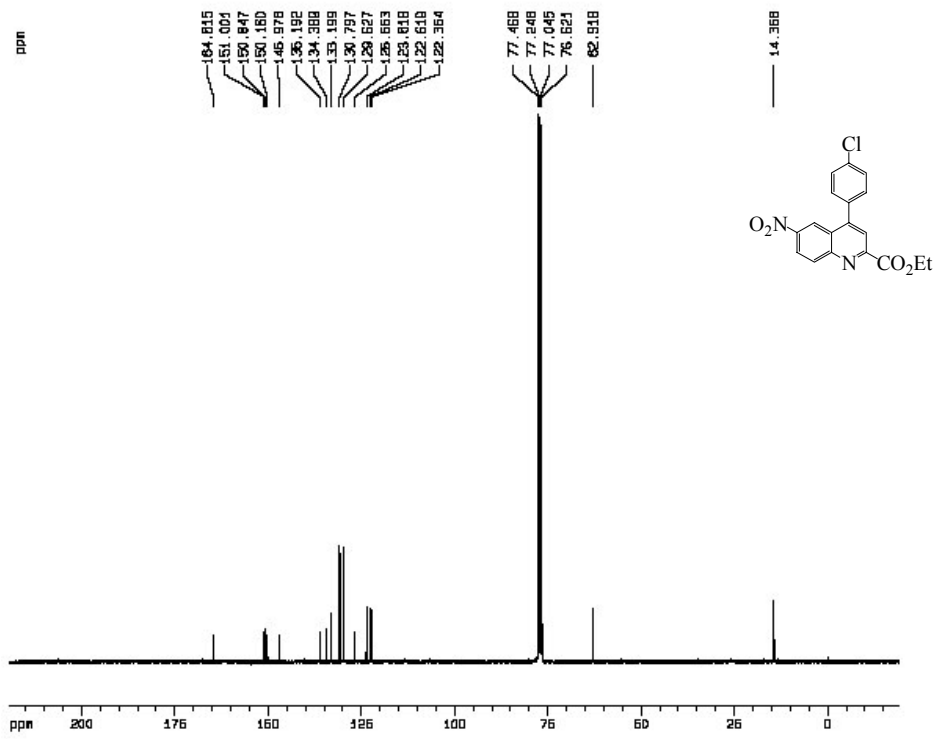

```
Current Data Parameters
NAME                wyc-6-58
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20091126
Time                   2.59
INSTRUM                av300
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    86638
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                    456.1
DW                   81.000 usec
DE                    6.00 usec
TE                   281.0 K
D1                2.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    9.30 usec
PL1                  -1.00 dB
SFO1           300.1318634 MHz

F2 - Processing parameters
SI                    32768
SF           300.1300085 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   12.66 cm
F1P                  10.250 ppm
F1                 3078.25 Hz
F2P                 -0.015 ppm
F2                  -184.54 Hz
PPMCM              0.54323 ppm/cm
HZCM             163.03896 Hz/cm
```




```
Current Data Parameters
NAME                wyc-6-58
EXPNO                     40
PROCNO                     1

F2 - Acquisition Parameters
Date_              20091128
Time                   0.43
INSTRUM                av300
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     4096
DS                        4
SWH              17985.611 Hz
FIDRES            0.274450 Hz
AQ                1.8219598 sec
RG                    2296.6
DW                   27.800 usec
DE                    6.00 usec
TE                   298.9 K
D1                2.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

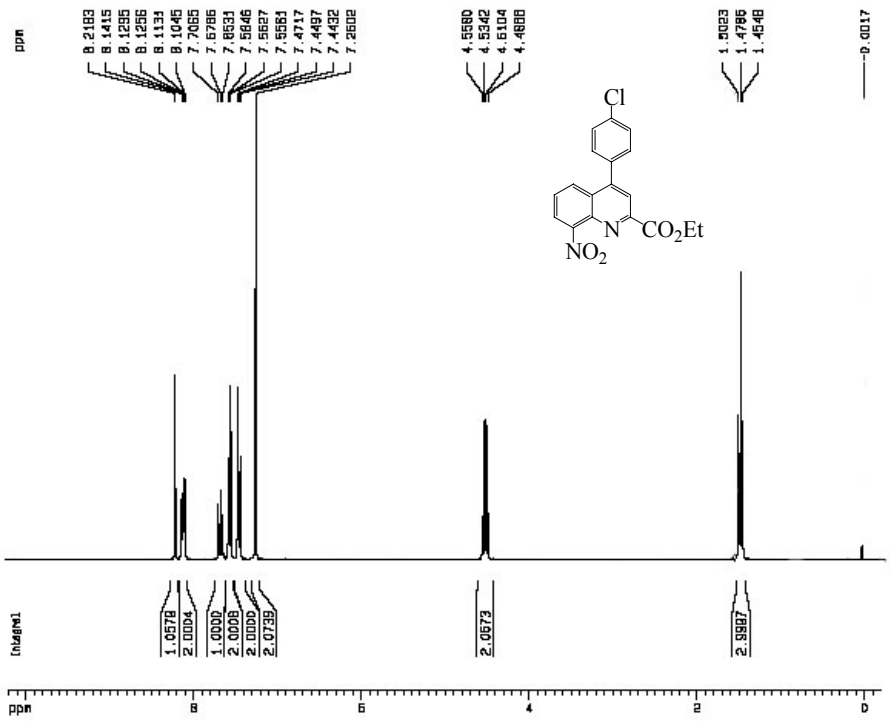
======== CHANNEL f1 ========
NUC1                    13C
P1                    9.40 usec
PL1                  -1.00 dB
SFO1           75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 18.00 dB
PL13                 18.00 dB
SFO2           300.1312006 MHz

F2 - Processing parameters
SI                    32768
SF            75.4677480 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   12.50 cm
F1P                 210.155 ppm
F1               15838.16 Hz
F2P                -16.187 ppm
F2               -1446.01 Hz
PPMCM             11.91806 ppm/cm
HZCM             898.89058 Hz/cm
```

Ethyl 4-(4-chlorophenyl)-8-nitroquinoline-2-carboxylate (3r)



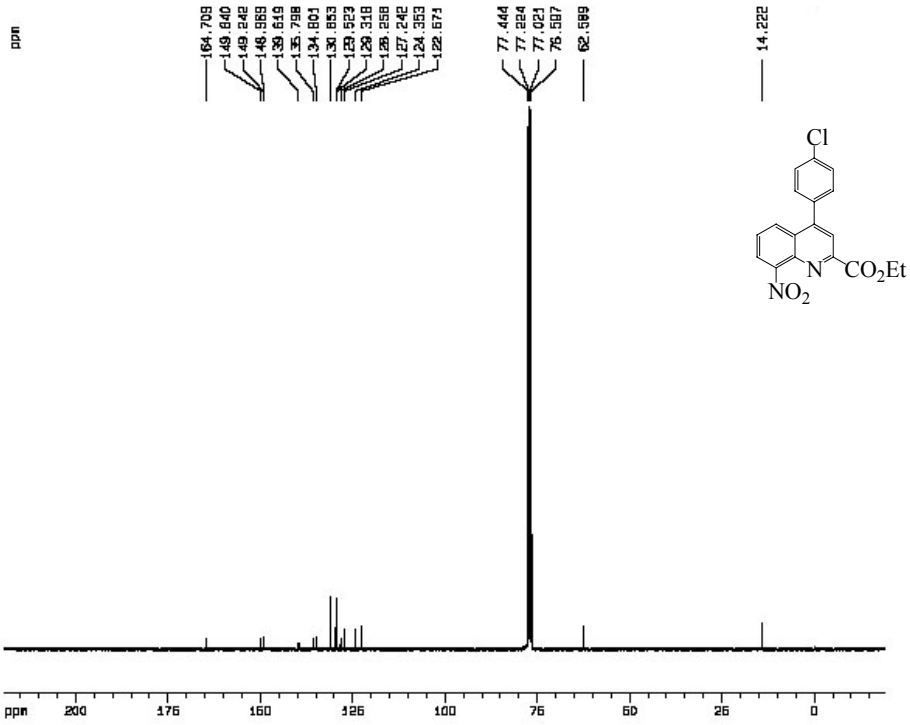
Current Data Parameters
NAME wpc-8-07a
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040311
Time 21.32
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 645.1
DW 84.000 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec

CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1318634 MHz

F2 - Processing parameters
SI 32768
SF 300.1300025 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.60 cm
F1P 10.200 ppm
F1 3061.33 Hz
F2P -0.800 ppm
F2 -60.03 Hz
PPMCM 0.52000 ppm/cm
HZCM 156.06761 Hz/cm



Current Data Parameters
NAME wpc-8-07a
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040312
Time 16.40
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6960
DS 4
SWH 17960.591 Hz
FIDRES 0.274459 Hz
AQ 1.8219698 sec
RG 3251
DW 27.880 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

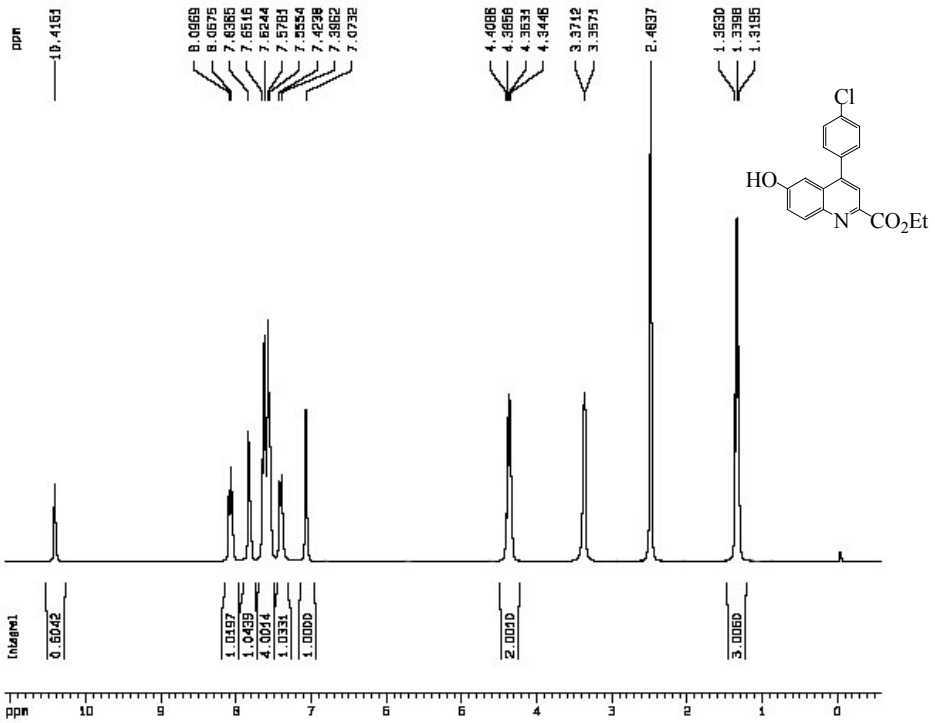
CHANNEL f1
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz

CHANNEL f2
DPOPRGB waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677459 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.60 cm
F1P 210.155 ppm
F1 15838.11 Hz
F2P -18.187 ppm
F2 -1446.01 Hz
PPMCM 11.91809 ppm/cm
HZCM 898.89058 Hz/cm

Ethyl 4-(4-chlorophenyl)-6-hydroxyquinoline-2-carboxylate (3s)



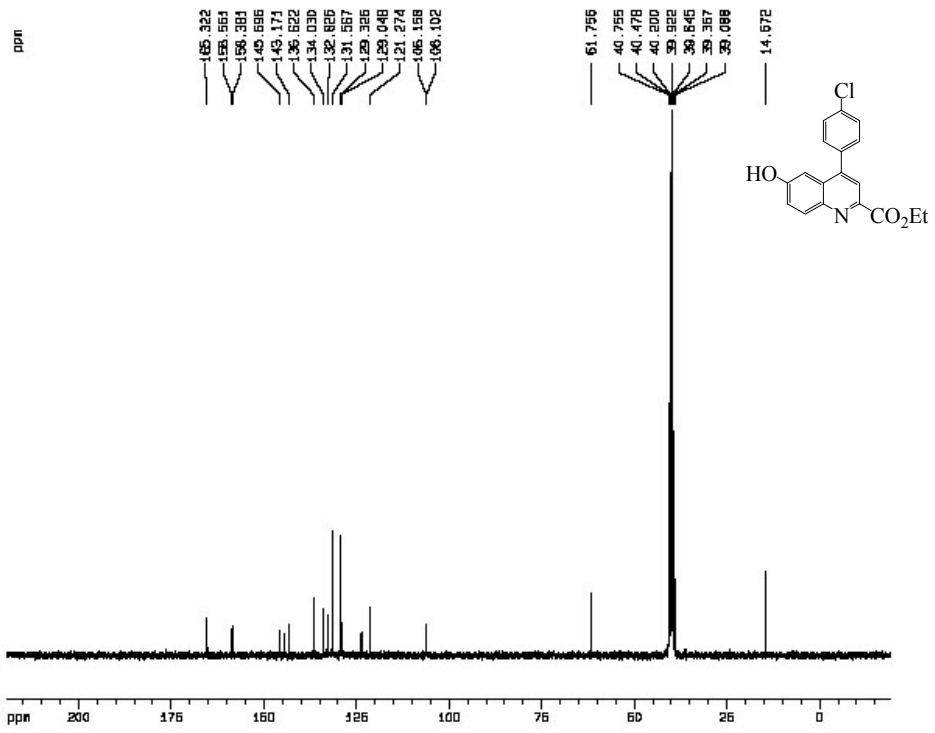

```
Current Data Parameters
NAME            wyc-6-09
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_         20091126
Time               4.22
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG            zg30
TD                 65536
SOLVENT            DMSO
NS                    16
DS                     2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084650 sec
RG                 161.3
DW                81.000 usec
DE                 6.00 usec
TE                282.3 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.30 usec
PL1              -1.00 dB
SFO1        300.1318634 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300025 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.60 cm
F1P               11.003 ppm
F1                3320.38 Hz
F2P               -0.096 ppm
F2                -175.82 Hz
PPMCM             0.58245 ppm/cm
HZCM            174.80902 Hz/cm
```




```
Current Data Parameters
NAME            wyc-6-09
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_         20091127
Time               3.40
INSTRUM            av300
PROBHD  5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            DMSO
NS                   308
DS                     4
SWH          17980.631 Hz
FIDRES        0.274459 Hz
AQ            1.8248598 sec
RG                1448.2
DW               27.800 usec
DE                 6.00 usec
TE                298.2 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

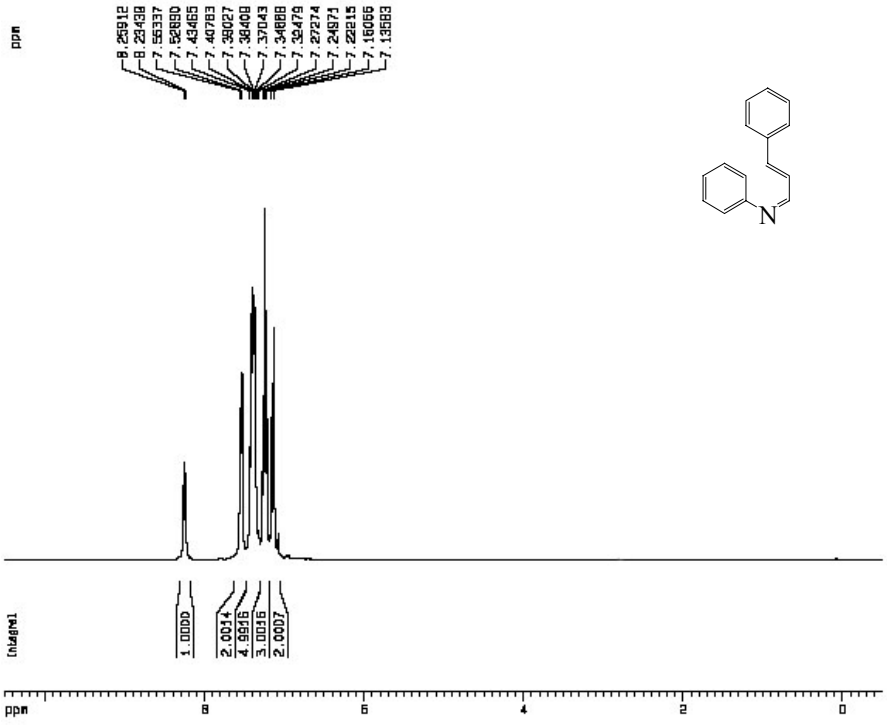
======== CHANNEL f1 ========
NUC1                13C
P1                9.40 usec
PL1              -1.00 dB
SFO1        75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             18.00 dB
PL13             18.00 dB
SFO2        300.1318066 MHz

F2 - Processing parameters
SI                 32768
SF          75.4677460 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                12.60 cm
F1P              210.455 ppm
F1             15836.14 Hz
F2P              -16.167 ppm
F2             -1446.01 Hz
PPMCM          11.91809 ppm/cm
HZCM          898.86058 Hz/cm
```

Schiff base **5t**



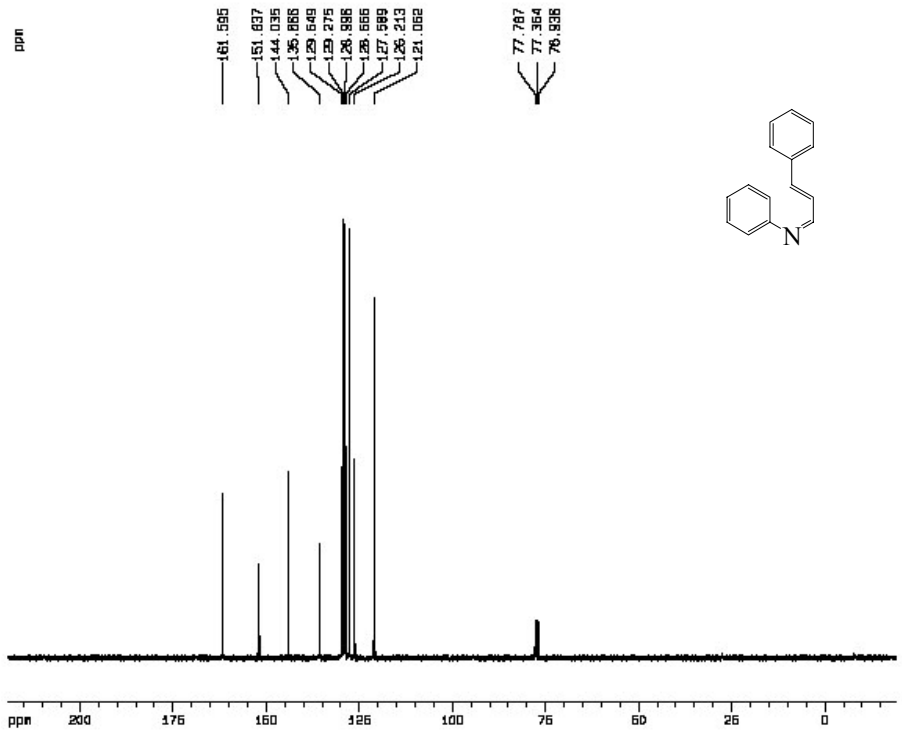

```
Current Data Parameters
NAME          wyc-16-8a
EXPNO              10
PROCNO              1

F2 - Acquisition Parameters
Date_         20080705
Time               3.09
INSTRUM          av300
PROBHD   5 mm DUL 13C-1
PULPROG          zg30
TD              86536
SOLVENT          CDCl3
NS                 16
DS                  2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                40.3
DW               81.000 usec
DE                6.00 usec
TE              673.2 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1               1H
P1                9.30 usec
PL1              -1.00 dB
SF01        300.1318634 MHz

F2 - Processing parameters
SI                32768
SF          300.1300085 MHz
WDW                EM
SSB                 0
LB                0.30 Hz
GB                  0
PC                1.00

1D NMR plot parameters
CX                20.00 cm
CY                 8.00 cm
F1P               10.000 ppm
F1              3101.36 Hz
F2P               -0.000 ppm
F2              -150.06 Hz
PPMCM             0.95000 ppm/cm
HZCM            285.07150 Hz/cm
```




```
Current Data Parameters
NAME          wyc-16-8a
EXPNO              11
PROCNO              1

F2 - Acquisition Parameters
Date_         20080708
Time               3.08
INSTRUM          av300
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD              65536
SOLVENT          CDCl3
NS                 80
DS                  4
SWH          17985.611 Hz
FIDRES        0.274450 Hz
AQ            1.8219598 sec
RG              5792.6
DW               27.800 usec
DE                6.00 usec
TE              673.2 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

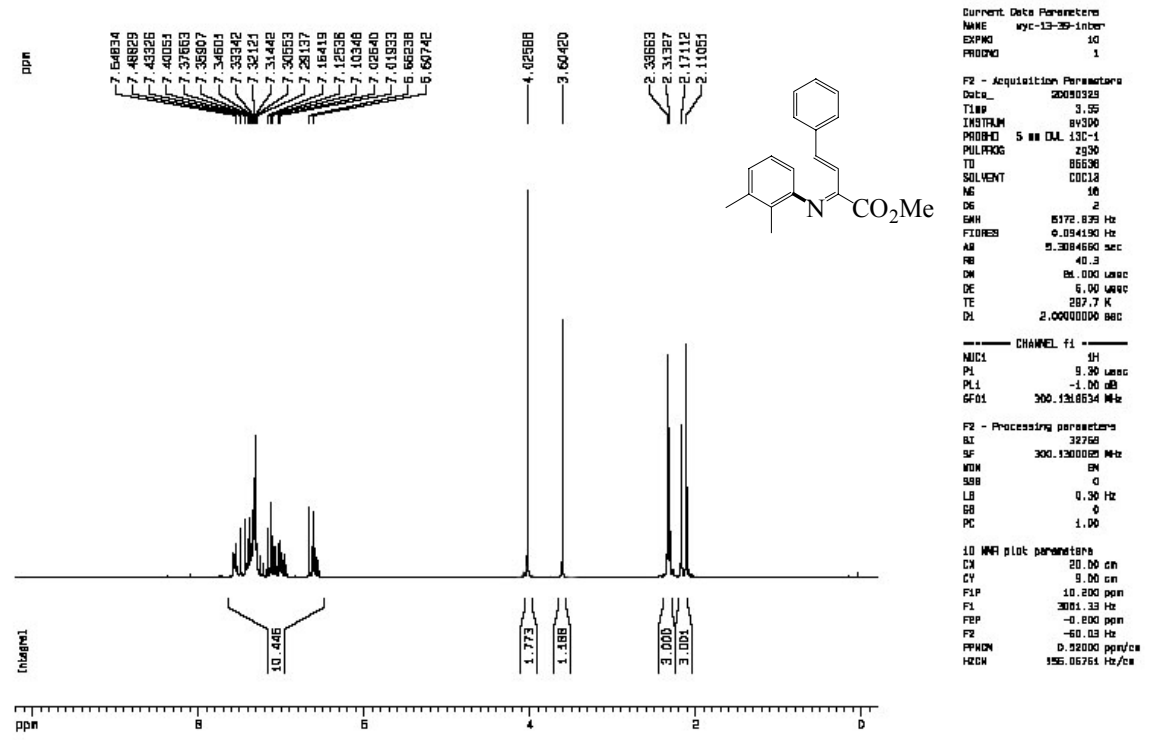
======== CHANNEL f1 ========
NUC1               13C
P1                9.40 usec
PL1              -1.00 dB
SF01        75.4752953 MHz

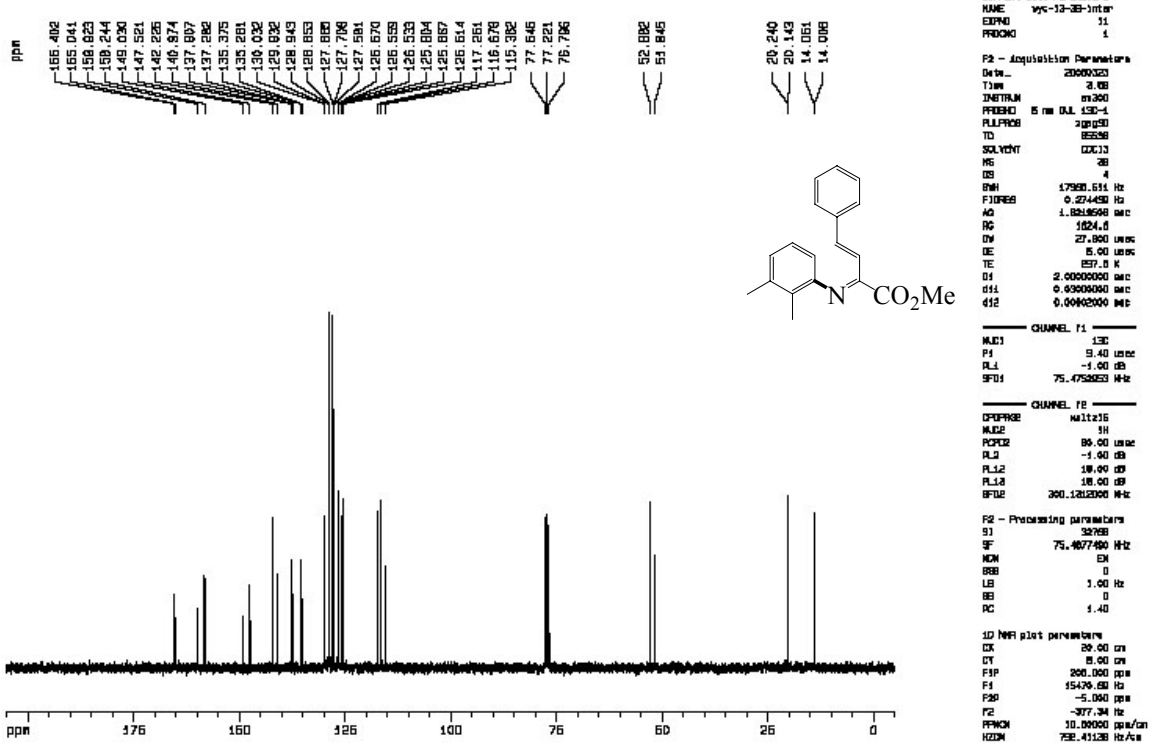
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2             80.00 usec
PL2              -1.00 dB
PL12             16.00 dB
PL13             16.00 dB
SF02        300.1318000 MHz

F2 - Processing parameters
SI                32768
SF          75.4677400 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P              210.155 ppm
F1             15839.11 Hz
F2P              -18.167 ppm
F2             -1446.01 Hz
PPMCM            11.91809 ppm/cm
HZCM           898.86058 Hz/cm
```

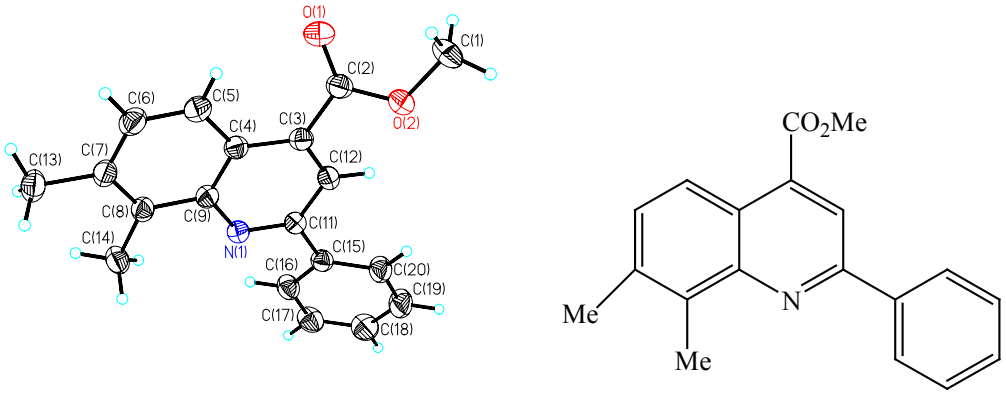
(3*E*)-2-(2,3-dimethylphenylimino)-4-phenylbut-3-enoate methyl ester (5a, syn and anti)





3. Single-crystal X-ray analyses

3.1 The single-crystal X-ray analysis of **4a**



Methyl 7,8-dimethyl-2-phenylquinoline-4-carboxylate (**4a**)

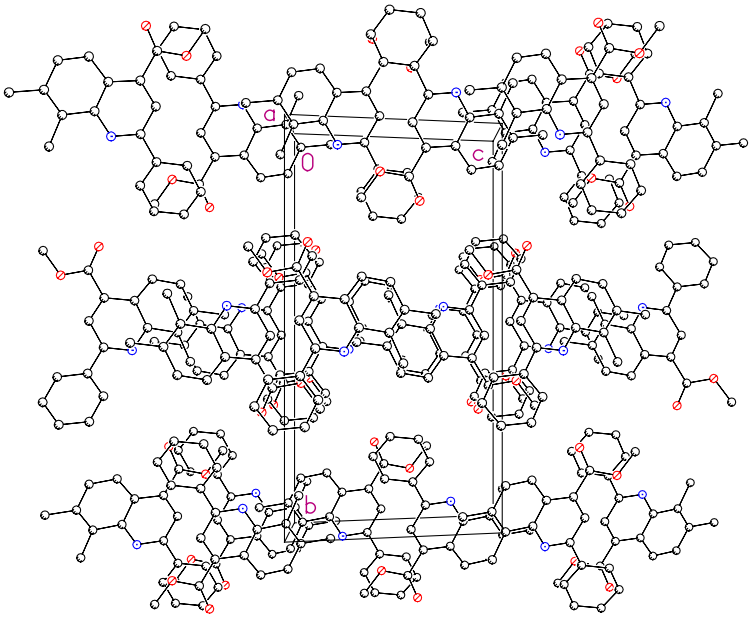


Table 1. Crystal data and structure refinement for **4a**

Identification code	**4a**
Empirical formula	C19 H17 N O2
Formula weight	291.34
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Aba2
Unit cell dimensions	a = 19.877(4) A alpha = 90 deg.
	b = 18.927(4) A beta = 90 deg.
	c = 8.0012(16) A gamma = 90 deg.
Volume	3010.2(10) A^3
Z, Calculated density	8, 1.286 Mg/m^3
Absorption coefficient	0.083 mm^-1
F(000)	1232
Crystal size	0.74 x 0.30 x 0.10 mm
Theta range for data collection	2.05 to 27.38 deg.
Limiting indices	0<=h<=25, -23<=k<=0, 0<=l<=10
Reflections collected / unique	12712 / 1803 [R(int) = 0.0493]
Completeness to theta = 27.38	97.90%
Absorption correction	Empirical
Max. and min. transmission	0.9919 and 0.9407
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1803 / 1 / 200
Goodness-of-fit on F^2	0.988
Final R indices [I>2sigma(I)]	R1 = 0.0474, wR2 = 0.1278
R indices (all data)	R1 = 0.0614, wR2 = 0.1322
Absolute structure parameter	-2(2)
Extinction coefficient	0.013(2)
Largest diff. peak and hole	0.241 and -0.232 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **4a**
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	6230(1)	11704(1)	4927(5)	68(1)

O(2)	5720(1)	10652(1)	4850(4)	55(1)
N(1)	6826(1)	10016(1)	5963(3)	40(1)
C(1)	5094(1)	10987(2)	4385(6)	65(1)
C(2)	6249(1)	11074(2)	5047(5)	46(1)
C(3)	6885(1)	10671(1)	5422(4)	40(1)
C(4)	7497(1)	11015(1)	5170(4)	43(1)
C(5)	8088(1)	10666(1)	5469(4)	39(1)
C(6)	8054(1)	9957(1)	6081(3)	36(1)
C(7)	7401(1)	9655(1)	6309(4)	35(1)
C(8)	7330(1)	8947(1)	6918(4)	37(1)
C(9)	7896(1)	8560(1)	7277(4)	41(1)
C(10)	8537(1)	8874(1)	7078(4)	46(1)
C(11)	8618(1)	9545(1)	6512(4)	44(1)
C(12)	7850(2)	7809(1)	7917(5)	55(1)
C(13)	6634(1)	8651(1)	7150(5)	53(1)
C(14)	8745(1)	11029(1)	5171(4)	42(1)
C(15)	9239(2)	10739(2)	4153(5)	55(1)
C(16)	9839(2)	11094(2)	3884(6)	67(1)
C(17)	9945(2)	11749(2)	4610(6)	66(1)
C(18)	9457(2)	12045(2)	5595(6)	62(1)
C(19)	8857(1)	11690(1)	5884(5)	50(1)

Table 3. Bond lengths [A] and angles [deg] for **4a**

O(1)-C(2)	1.198(3)	N(1)-C(3)-C(4)	124.4(2)
O(2)-C(2)	1.330(3)	N(1)-C(3)-C(2)	117.8(2)
O(2)-C(1)	1.446(3)	C(4)-C(3)-C(2)	117.8(2)
N(1)-C(3)	1.318(3)	C(5)-C(4)-C(3)	119.9(2)
N(1)-C(7)	1.360(3)	C(4)-C(5)-C(6)	118.0(2)
C(2)-C(3)	1.506(3)	C(4)-C(5)-C(14)	120.1(2)
C(3)-C(4)	1.394(4)	C(6)-C(5)-C(14)	121.9(2)
C(4)-C(5)	1.369(4)	C(11)-C(6)-C(7)	118.1(2)
C(5)-C(6)	1.431(3)	C(11)-C(6)-C(5)	124.4(2)
C(5)-C(14)	1.494(3)	C(7)-C(6)-C(5)	117.6(2)
C(6)-C(11)	1.410(3)	N(1)-C(7)-C(6)	122.46(19)
C(6)-C(7)	1.429(3)	N(1)-C(7)-C(8)	117.13(19)
C(7)-C(8)	1.434(3)	C(6)-C(7)-C(8)	120.4(2)
C(8)-C(9)	1.373(3)	C(9)-C(8)-C(7)	119.3(2)

C(8)-C(13)	1.503(3)	C(9)-C(8)-C(13)	122.0(2)
C(9)-C(10)	1.415(4)	C(7)-C(8)-C(13)	118.7(2)
C(9)-C(12)	1.514(3)	C(8)-C(9)-C(10)	119.3(2)
C(10)-C(11)	1.357(4)	C(8)-C(9)-C(12)	121.5(2)
C(14)-C(15)	1.388(5)	C(10)-C(9)-C(12)	119.2(2)
C(14)-C(19)	1.394(4)	C(11)-C(10)-C(9)	122.5(2)
C(15)-C(16)	1.386(4)	C(10)-C(11)-C(6)	120.3(2)
C(16)-C(17)	1.386(6)	C(15)-C(14)-C(19)	118.9(2)
C(17)-C(18)	1.369(5)	C(15)-C(14)-C(5)	122.0(3)
C(18)-C(19)	1.388(4)	C(19)-C(14)-C(5)	119.1(3)
C(2)-O(2)-C(1)	116.6(2)	C(16)-C(15)-C(14)	120.5(3)
C(3)-N(1)-C(7)	117.61(19)	C(17)-C(16)-C(15)	120.0(3)
O(1)-C(2)-O(2)	124.3(2)	C(18)-C(17)-C(16)	120.0(3)
O(1)-C(2)-C(3)	123.2(2)	C(17)-C(18)-C(19)	120.5(3)
O(2)-C(2)-C(3)	112.5(2)	C(18)-C(19)-C(14)	120.2(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Aˆ2 x 10ˆ3) for **4a**
The anisotropic displacement factor exponent takes the form:
-2 piˆ2 [hˆ2 a*ˆ2 U11 + ... + 2 h k a* b* U12]

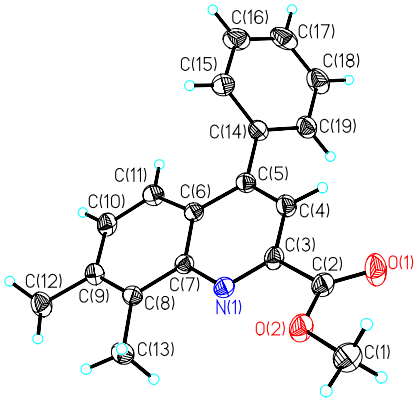
	U11	U22	U33	U23	U13	U12
O(1)	51(1)	43(1)	112(2)	7(1)	-17(2)	9(1)
O(2)	32(1)	49(1)	84(2)	1(1)	-10(1)	8(1)
N(1)	26(1)	39(1)	54(2)	1(1)	-1(1)	3(1)
C(1)	31(1)	68(2)	96(3)	13(2)	-12(2)	8(1)
C(2)	31(1)	45(1)	60(2)	2(1)	-4(1)	5(1)
C(3)	33(1)	37(1)	50(2)	-1(1)	-6(1)	7(1)
C(4)	39(1)	36(1)	53(2)	4(1)	-2(1)	1(1)
C(5)	33(1)	37(1)	46(2)	1(1)	0(1)	-2(1)
C(6)	30(1)	37(1)	41(2)	2(1)	0(1)	0(1)
C(7)	32(1)	35(1)	39(2)	-3(1)	0(1)	1(1)
C(8)	34(1)	36(1)	41(2)	-1(1)	0(1)	-2(1)
C(9)	43(1)	36(1)	46(2)	-1(1)	-4(1)	1(1)
C(10)	36(1)	43(1)	59(2)	3(1)	-9(1)	11(1)
C(11)	29(1)	46(1)	59(2)	1(1)	-5(1)	-1(1)
C(12)	56(2)	41(1)	66(2)	9(2)	-7(2)	4(1)
C(13)	41(2)	44(1)	73(2)	4(2)	4(2)	-4(1)
C(14)	32(1)	39(1)	54(2)	10(1)	-2(1)	0(1)

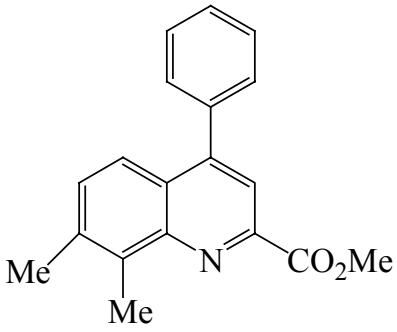
C(15)	43(2)	52(2)	69(2)	2(2)	6(2)	0(1)
C(16)	39(2)	76(2)	85(3)	16(2)	10(2)	-3(2)
C(17)	40(1)	71(2)	88(3)	28(2)	-3(2)	-16(2)
C(18)	49(2)	48(1)	89(3)	8(2)	-9(2)	-13(1)
C(19)	42(1)	40(1)	67(2)	4(1)	-1(1)	-3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **4a**

	x	y	z	U(eq)
H(1B)	4749	10634	4278	98
H(1C)	5149	11227	3337	98
H(1D)	4967	11321	5231	98
H(4A)	7503	11480	4799	51
H(10A)	8917	8611	7345	55
H(11A)	9048	9733	6408	53
H(12A)	7386	7670	7985	82
H(12B)	8051	7781	9006	82
H(12C)	8083	7498	7166	82
H(13A)	6665	8176	7562	79
H(13B)	6402	8653	6099	79
H(13C)	6392	8937	7939	79
H(15A)	9166	10303	3648	66
H(16A)	10171	10892	3217	80
H(17A)	10347	11988	4428	79
H(18A)	9528	12487	6073	74
H(19A)	8528	11895	6556	60

3.2 The single-crystal X-ray analysis of **3a**





Methyl 7,8-dimethyl-4-phenylquinoline-2-carboxylate (**3a**)

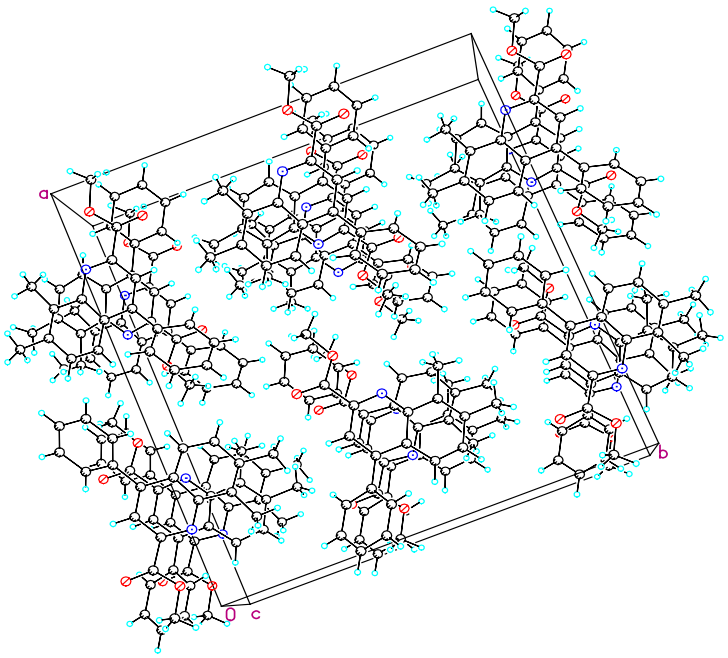


Table 1. Crystal data and structure refinement for **3a**

Identification code	**3a**
Empirical formula	C19 H17 N O2
Formula weight	291.34
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 7.9843(16) A alpha = 90 deg.
	b = 19.014(4) A beta = 109.99(3) deg.
	c = 10.550(2) A gamma = 90 deg.
Volume	1505.1(5) A^3
Z, Calculated density	4, 1.286 Mg/m^3
Absorption coefficient	0.083 mm^-1
F(000)	616
Crystal size	0.79 x 0.32 x 0.26 mm
Theta range for data collection	2.32 to 27.47 deg.
Limiting indices	-9<=h<=10, -22<=k<=24, -13<=l<=12
Reflections collected / unique	9208 / 3352 [R(int) = 0.0543]
Completeness to theta = 27.47	97.20%
Absorption correction	Empirical
Max. and min. transmission	0.9784 and 0.9367
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3352 / 0 / 208
Goodness-of-fit on F^2	1.03
Final R indices [I>2sigma(I)]	R1 = 0.0671, wR2 = 0.1710
R indices (all data)	R1 = 0.0921, wR2 = 0.1904
Largest diff. peak and hole	0.214 and -0.260 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **3a**
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
N(1)	3032(2)	5537(1)	2726(2)	45(1)
O(1)	1641(3)	3008(1)	1090(2)	76(1)
O(2)	749(2)	3708(1)	-710(2)	59(1)

C(1)	136(4)	3104(1)	-1569(3)	74(1)
C(2)	1451(3)	3586(1)	605(2)	52(1)
C(3)	1988(3)	4260(1)	1370(2)	46(1)
C(4)	2030(3)	4325(1)	2726(2)	45(1)
C(5)	1568(3)	3791(1)	3472(2)	55(1)
C(6)	1655(3)	3910(1)	4762(2)	59(1)
C(7)	2161(3)	4566(1)	5402(2)	52(1)
C(8)	2619(3)	5103(1)	4717(2)	48(1)
C(9)	2570(3)	4987(1)	3364(2)	44(1)
C(11)	2971(3)	5461(1)	1465(2)	44(1)
C(12)	2447(3)	4822(1)	759(2)	49(1)
C(13)	2137(4)	4663(2)	6818(2)	67(1)
C(14)	3154(4)	5816(1)	5337(2)	61(1)
C(15)	3489(3)	6083(1)	836(2)	47(1)
C(16)	4491(3)	6615(1)	1645(2)	58(1)
C(17)	4962(4)	7206(1)	1095(3)	69(1)
C(18)	4453(4)	7276(1)	-271(3)	77(1)
C(19)	3452(5)	6757(1)	-1099(3)	78(1)
C(20)	2984(4)	6164(1)	-546(2)	63(1)

Table 3. Bond lengths [A] and angles [deg] for **3a**

N(1)-C(11)	1.322(2)	C(5)-C(4)-C(9)	117.93(18)
N(1)-C(9)	1.361(2)	C(5)-C(4)-C(3)	125.35(19)
O(1)-C(2)	1.200(2)	C(9)-C(4)-C(3)	116.72(17)
O(2)-C(2)	1.326(3)	C(6)-C(5)-C(4)	120.4(2)
O(2)-C(1)	1.441(3)	C(6)-C(5)-H(5A)	119.8
C(1)-H(1A)	0.96	C(4)-C(5)-H(5A)	119.8
C(1)-H(1B)	0.96	C(5)-C(6)-C(7)	122.5(2)
C(1)-H(1C)	0.96	C(5)-C(6)-H(6A)	118.8
C(2)-C(3)	1.497(3)	C(7)-C(6)-H(6A)	118.8
C(3)-C(12)	1.362(3)	C(8)-C(7)-C(6)	119.38(19)
C(3)-C(4)	1.424(3)	C(8)-C(7)-C(13)	121.7(2)
C(4)-C(5)	1.409(3)	C(6)-C(7)-C(13)	118.9(2)
C(4)-C(9)	1.424(3)	C(7)-C(8)-C(9)	119.32(19)
C(5)-C(6)	1.357(3)	C(7)-C(8)-C(14)	122.00(18)
C(5)-H(5A)	0.93	C(9)-C(8)-C(14)	118.68(18)
C(6)-C(7)	1.409(3)	N(1)-C(9)-C(4)	122.38(17)
C(6)-H(6A)	0.93	N(1)-C(9)-C(8)	117.09(17)

C(7)-C(8)	1.371(3)	C(4)-C(9)-C(8)	120.51(17)
C(7)-C(13)	1.513(3)	N(1)-C(11)-C(12)	121.78(17)
C(8)-C(9)	1.432(3)	N(1)-C(11)-C(15)	116.36(17)
C(8)-C(14)	1.503(3)	C(12)-C(11)-C(15)	121.86(17)
C(11)-C(12)	1.411(3)	C(3)-C(12)-C(11)	120.33(18)
C(11)-C(15)	1.482(3)	C(3)-C(12)-H(12A)	119.8
C(12)-H(12A)	0.93	C(11)-C(12)-H(12A)	119.8
C(13)-H(13A)	0.96	C(7)-C(13)-H(13A)	109.5
C(13)-H(13B)	0.96	C(7)-C(13)-H(13B)	109.5
C(13)-H(13C)	0.96	H(13A)-C(13)-H(13B)	109.5
C(14)-H(14A)	0.96	C(7)-C(13)-H(13C)	109.5
C(14)-H(14B)	0.96	H(13A)-C(13)-H(13C)	109.5
C(14)-H(14C)	0.96	H(13B)-C(13)-H(13C)	109.5
C(15)-C(20)	1.382(3)	C(8)-C(14)-H(14A)	109.5
C(15)-C(16)	1.387(3)	C(8)-C(14)-H(14B)	109.5
C(16)-C(17)	1.376(3)	H(14A)-C(14)-H(14B)	109.5
C(16)-H(16A)	0.93	C(8)-C(14)-H(14C)	109.5
C(17)-C(18)	1.364(4)	H(14A)-C(14)-H(14C)	109.5
C(17)-H(17A)	0.93	H(14B)-C(14)-H(14C)	109.5
C(18)-C(19)	1.377(4)	C(20)-C(15)-C(16)	117.82(19)
C(18)-H(18A)	0.93	C(20)-C(15)-C(11)	122.44(19)
C(19)-C(20)	1.378(3)	C(16)-C(15)-C(11)	119.73(18)
C(19)-H(19A)	0.93	C(17)-C(16)-C(15)	121.3(2)
C(20)-H(20A)	0.93	C(17)-C(16)-H(16A)	119.4
C(11)-N(1)-C(9)	119.42(16)	C(15)-C(16)-H(16A)	119.4
C(2)-O(2)-C(1)	116.78(18)	C(18)-C(17)-C(16)	120.0(2)
O(2)-C(1)-H(1A)	109.5	C(18)-C(17)-H(17A)	120
O(2)-C(1)-H(1B)	109.5	C(16)-C(17)-H(17A)	120
H(1A)-C(1)-H(1B)	109.5	C(17)-C(18)-C(19)	119.9(2)
O(2)-C(1)-H(1C)	109.5	C(17)-C(18)-H(18A)	120
H(1A)-C(1)-H(1C)	109.5	C(19)-C(18)-H(18A)	120
H(1B)-C(1)-H(1C)	109.5	C(20)-C(19)-C(18)	120.0(2)
O(1)-C(2)-O(2)	123.50(19)	C(20)-C(19)-H(19A)	120
O(1)-C(2)-C(3)	125.6(2)	C(18)-C(19)-H(19A)	120
O(2)-C(2)-C(3)	110.86(17)	C(19)-C(20)-C(15)	121.0(2)
C(12)-C(3)-C(4)	119.37(18)	C(19)-C(20)-H(20A)	119.5
C(12)-C(3)-C(2)	119.34(18)	C(15)-C(20)-H(20A)	119.5
C(4)-C(3)-C(2)	121.29(17)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Aˆ2 x 10ˆ3) for **3a**
The anisotropic displacement factor exponent takes the form:
-2 piˆ2 [hˆ2 a*ˆ2 U11 + ... + 2 h k a* b* U12]

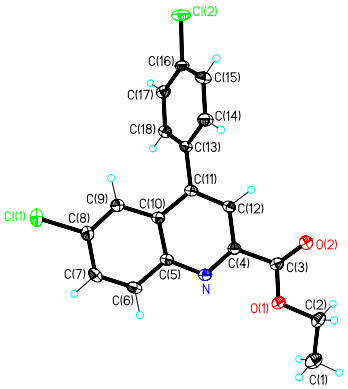
	U11	U22	U33	U23	U13	U12
N(1)	49(1)	43(1)	43(1)	0(1)	15(1)	1(1)
O(1)	115(2)	40(1)	71(1)	2(1)	28(1)	1(1)
O(2)	78(1)	44(1)	52(1)	-9(1)	18(1)	-2(1)
C(1)	91(2)	53(1)	71(2)	-25(1)	21(2)	-5(1)
C(2)	59(1)	43(1)	56(1)	-3(1)	22(1)	2(1)
C(3)	49(1)	39(1)	47(1)	1(1)	15(1)	2(1)
C(4)	46(1)	43(1)	46(1)	3(1)	16(1)	4(1)
C(5)	64(1)	46(1)	56(1)	5(1)	21(1)	-2(1)
C(6)	66(1)	57(1)	58(1)	14(1)	26(1)	4(1)
C(7)	50(1)	63(1)	45(1)	8(1)	18(1)	10(1)
C(8)	45(1)	55(1)	42(1)	1(1)	13(1)	5(1)
C(9)	44(1)	46(1)	43(1)	1(1)	13(1)	3(1)
C(11)	47(1)	43(1)	42(1)	0(1)	16(1)	2(1)
C(12)	61(1)	45(1)	41(1)	-2(1)	20(1)	0(1)
C(13)	69(2)	87(2)	48(1)	10(1)	25(1)	9(1)
C(14)	72(2)	62(1)	51(1)	-11(1)	24(1)	-4(1)
C(15)	53(1)	43(1)	49(1)	0(1)	21(1)	0(1)
C(16)	72(2)	52(1)	53(1)	-6(1)	26(1)	-11(1)
C(17)	87(2)	50(1)	77(2)	-8(1)	38(1)	-16(1)
C(18)	109(2)	53(1)	81(2)	8(1)	50(2)	-11(1)
C(19)	119(2)	66(2)	55(1)	10(1)	37(2)	-8(2)
C(20)	86(2)	53(1)	49(1)	1(1)	24(1)	-9(1)

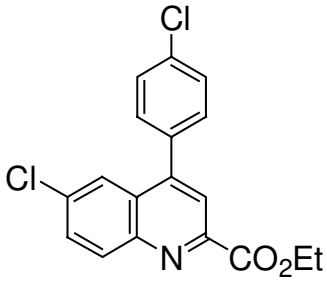
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **3a**

	x	y	z	U(eq)
H(1A)	-527	2800	-1188	101(11)
H(1B)	1142	2853	-1644	122(13)
H(1C)	-615	3255	-2447	113(11)
H(5A)	1201	3354	3079	66
H(6A)	1371	3546	5241	71
H(12A)	2416	4785	-127	58

H(13A)	2044	4212	7199	113(11)
H(13B)	1134	4948	6792	107(11)
H(13C)	3217	4890	7362	93(10)
H(14A)	2136	6121	5074	190(20)
H(14B)	4061	6009	5032	130(13)
H(14C)	3608	5773	6302	125(12)
H(16A)	4852	6570	2578	69
H(17A)	5626	7559	1654	82
H(18A)	4782	7673	-644	92
H(19A)	3092	6807	-2031	93
H(20A)	2319	5814	-1111	75

3.3 The single-crystal X-ray analysis of **3o**





Ethyl 6-chloro-4-(4-chlorophenyl)quinoline-2-carboxylate (**3o**)

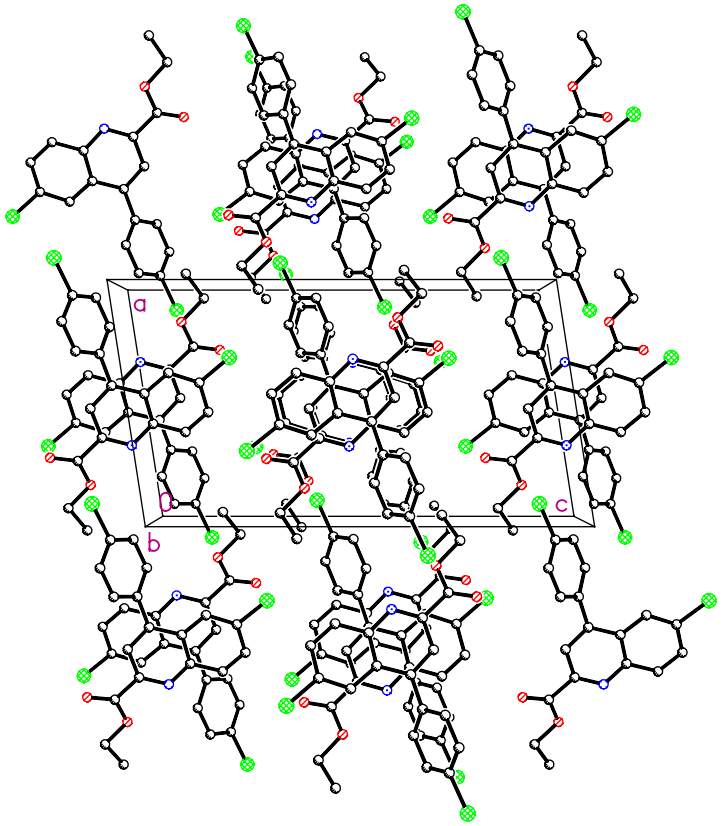


Table 1. Crystal data and structure refinement for **3o**

Identification code	**3o**
Empirical formula	C18 H13 Cl2 N O2
Formula weight	346.19
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	monoclinic, P2(1)/c
Unit cell dimensions	a = 10.3615(4) A alpha = 90 deg.
	b = 8.4311(4) A beta = 98.8060(7) deg.
	c = 18.5887(6) A gamma = 90 deg.
Volume	1604.75(11) A^3
Z, Calculated density	4, 1.433 Mg/m^3
Absorption coefficient	0.413 mm^-1
F(000)	712
Crystal size	0.47 x 0.40 x 0.31 mm
Theta range for data collection	2.22 to 27.44 deg.
Limiting indices	0<=h<=13, 0<=k<=10, -24<=l<=23
Reflections collected / unique	3660 / 3660 [R(int) = 0.0000]
Completeness to theta = 27.44	99.80%
Max. and min. transmission	0.8820 and 0.8297
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3660 / 0 / 208
Goodness-of-fit on F^2	0.824
Final R indices [I>2sigma(I)]	R1 = 0.0382, wR2 = 0.0987
R indices (all data)	R1 = 0.0633, wR2 = 0.1073
Largest diff. peak and hole	0.277 and -0.330 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **3o** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Cl(1)	13123(1)	1777(1)	-2407(1)	67(1)
Cl(2)	9092(1)	507(1)	1134(1)	52(1)
O(1)	18448(1)	4594(2)	1332(1)	42(1)
O(2)	17280(1)	3445(2)	2103(1)	57(1)
N	16764(1)	3660(2)	192(1)	32(1)
C(1)	20424(2)	5990(3)	1657(1)	65(1)

C(2)	19448(2)	4995(3)	1940(1)	55(1)
C(3)	17423(2)	3814(2)	1496(1)	36(1)
C(4)	16441(2)	3413(2)	839(1)	31(1)
C(5)	15856(2)	3260(2)	-392(1)	30(1)
C(6)	16195(2)	3472(2)	-1096(1)	39(1)
C(7)	15367(2)	3049(3)	-1700(1)	42(1)
C(8)	14152(2)	2383(2)	-1625(1)	39(1)
C(9)	13761(2)	2198(2)	-965(1)	36(1)
C(10)	14602(2)	2644(2)	-327(1)	30(1)
C(11)	14279(2)	2468(2)	387(1)	29(1)
C(12)	15226(2)	2828(2)	963(1)	33(1)
C(13)	12971(2)	1937(2)	524(1)	30(1)
C(14)	12877(2)	659(2)	981(1)	37(1)
C(15)	11685(2)	191(2)	1160(1)	39(1)
C(16)	10588(2)	1039(2)	881(1)	35(1)
C(17)	10639(2)	2309(2)	423(1)	39(1)
C(18)	11839(2)	2756(2)	241(1)	37(1)

Table 3. Bond lengths [A] and angles [deg] for **3o**

Cl(1)-C(8)	1.7418(18)	C(1)-C(2)-H(2B)	110.2
Cl(2)-C(16)	1.7465(16)	H(2A)-C(2)-H(2B)	108.5
O(1)-C(3)	1.325(2)	O(2)-C(3)-O(1)	124.34(16)
O(1)-C(2)	1.452(2)	O(2)-C(3)-C(4)	122.68(16)
O(2)-C(3)	1.201(2)	O(1)-C(3)-C(4)	112.98(14)
N-C(4)	1.315(2)	N-C(4)-C(12)	124.45(15)
N-C(5)	1.365(2)	N-C(4)-C(3)	118.23(14)
C(1)-C(2)	1.472(3)	C(12)-C(4)-C(3)	117.30(14)
C(1)-H(1A)	0.96	N-C(5)-C(10)	123.42(14)
C(1)-H(1B)	0.96	N-C(5)-C(6)	117.69(15)
C(1)-H(1C)	0.96	C(10)-C(5)-C(6)	118.90(15)
C(2)-H(2A)	0.97	C(7)-C(6)-C(5)	121.20(16)
C(2)-H(2B)	0.97	C(7)-C(6)-H(6A)	119.4
C(3)-C(4)	1.503(2)	C(5)-C(6)-H(6A)	119.4
C(4)-C(12)	1.403(2)	C(6)-C(7)-C(8)	119.13(16)
C(5)-C(10)	1.422(2)	C(6)-C(7)-H(7A)	120.4
C(5)-C(6)	1.418(2)	C(8)-C(7)-H(7A)	120.4
C(6)-C(7)	1.353(3)	C(9)-C(8)-C(7)	122.10(17)

C(6)-H(6A)	0.93	C(9)-C(8)-Cl(1)	119.42(15)
C(7)-C(8)	1.406(3)	C(7)-C(8)-Cl(1)	118.48(13)
C(7)-H(7A)	0.93	C(8)-C(9)-C(10)	119.84(16)
C(8)-C(9)	1.358(2)	C(8)-C(9)-H(9A)	120.1
C(9)-C(10)	1.411(2)	C(10)-C(9)-H(9A)	120.1
C(9)-H(9A)	0.93	C(9)-C(10)-C(5)	118.75(14)
C(10)-C(11)	1.424(2)	C(9)-C(10)-C(11)	123.52(15)
C(11)-C(12)	1.371(2)	C(5)-C(10)-C(11)	117.71(14)
C(11)-C(13)	1.486(2)	C(12)-C(11)-C(10)	117.53(14)
C(12)-H(12A)	0.93	C(12)-C(11)-C(13)	119.69(14)
C(13)-C(14)	1.385(2)	C(10)-C(11)-C(13)	122.78(14)
C(13)-C(18)	1.392(2)	C(11)-C(12)-C(4)	120.20(15)
C(14)-C(15)	1.385(2)	C(11)-C(12)-H(12A)	119.9
C(14)-H(14A)	0.93	C(4)-C(12)-H(12A)	119.9
C(15)-C(16)	1.375(3)	C(14)-C(13)-C(18)	118.89(15)
C(15)-H(15A)	0.93	C(14)-C(13)-C(11)	119.41(15)
C(16)-C(17)	1.374(3)	C(18)-C(13)-C(11)	121.57(16)
C(17)-C(18)	1.389(2)	C(13)-C(14)-C(15)	121.17(17)
C(17)-H(17A)	0.93	C(13)-C(14)-H(14A)	119.4
C(18)-H(18A)	0.93	C(15)-C(14)-H(14A)	119.4
C(3)-O(1)-C(2)	115.93(14)	C(16)-C(15)-C(14)	118.52(17)
C(4)-N-C(5)	116.58(14)	C(16)-C(15)-H(15A)	120.7
C(2)-C(1)-H(1A)	109.5	C(14)-C(15)-H(15A)	120.7
C(2)-C(1)-H(1B)	109.5	C(15)-C(16)-C(17)	122.06(16)
H(1A)-C(1)-H(1B)	109.5	C(15)-C(16)-Cl(2)	118.77(14)
C(2)-C(1)-H(1C)	109.5	C(17)-C(16)-Cl(2)	119.16(14)
H(1A)-C(1)-H(1C)	109.5	C(16)-C(17)-C(18)	118.86(17)
H(1B)-C(1)-H(1C)	109.5	C(16)-C(17)-H(17A)	120.6
O(1)-C(2)-C(1)	107.59(17)	C(18)-C(17)-H(17A)	120.6
O(1)-C(2)-H(2A)	110.2	C(17)-C(18)-C(13)	120.49(17)
C(1)-C(2)-H(2A)	110.2	C(17)-C(18)-H(18A)	119.8
O(1)-C(2)-H(2B)	110.2	C(13)-C(18)-H(18A)	119.8

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **3o**
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12

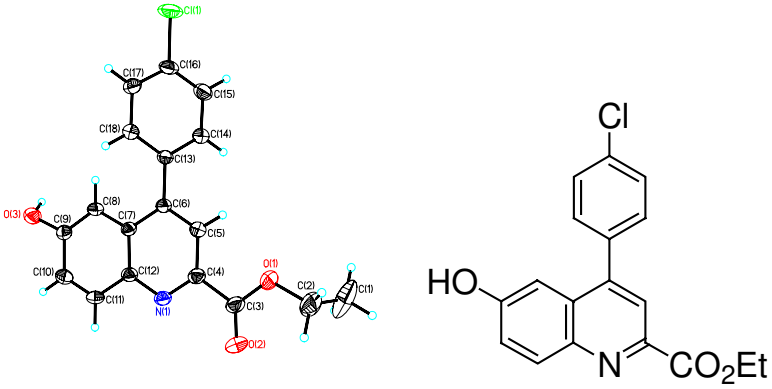
Cl(1)	61(1)	104(1)	34(1)	-16(1)	4(1)	-18(1)
Cl(2)	35(1)	56(1)	70(1)	-1(1)	24(1)	-13(1)
O(1)	32(1)	56(1)	37(1)	3(1)	1(1)	-15(1)
O(2)	44(1)	93(1)	33(1)	10(1)	3(1)	-20(1)
N	28(1)	36(1)	33(1)	3(1)	7(1)	-4(1)
C(1)	51(1)	79(2)	62(1)	-4(1)	1(1)	-29(1)
C(2)	40(1)	76(2)	45(1)	4(1)	-7(1)	-19(1)
C(3)	28(1)	44(1)	37(1)	3(1)	4(1)	-3(1)
C(4)	27(1)	33(1)	32(1)	3(1)	6(1)	-2(1)
C(5)	29(1)	31(1)	32(1)	1(1)	9(1)	-1(1)
C(6)	35(1)	48(1)	38(1)	2(1)	17(1)	-6(1)
C(7)	47(1)	53(1)	29(1)	-1(1)	16(1)	-3(1)
C(8)	41(1)	47(1)	30(1)	-5(1)	5(1)	-2(1)
C(9)	33(1)	42(1)	35(1)	-3(1)	8(1)	-7(1)
C(10)	28(1)	30(1)	32(1)	0(1)	8(1)	-2(1)
C(11)	28(1)	30(1)	32(1)	2(1)	9(1)	-3(1)
C(12)	30(1)	41(1)	30(1)	5(1)	9(1)	-3(1)
C(13)	29(1)	36(1)	28(1)	-2(1)	10(1)	-7(1)
C(14)	31(1)	40(1)	43(1)	7(1)	9(1)	-1(1)
C(15)	39(1)	38(1)	44(1)	7(1)	14(1)	-6(1)
C(16)	29(1)	41(1)	37(1)	-6(1)	13(1)	-11(1)
C(17)	30(1)	44(1)	42(1)	2(1)	7(1)	-2(1)
C(18)	34(1)	42(1)	34(1)	6(1)	7(1)	-5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **3o**

	x	y	z	U(eq)
H(1A)	21097	6280	2049	97
H(1B)	20010	6930	1441	97
H(1C)	20802	5408	1297	97
H(2A)	19854	4038	2158	66
H(2B)	19065	5567	2308	66
H(6A)	17001	3910	-1144	47
H(7A)	15599	3196	-2160	50
H(9A)	12941	1779	-934	43
H(12A)	15063	2684	1436	40
H(14A)	13629	106	1172	45
H(15A)	11627	-678	1461	47

H(17A)	9884	2860	237	46
H(18A)	11887	3608	-71	44

3.4 The single-crystal X-ray analysis of **3s**



Ethyl 4-(4-chlorophenyl)-6-hydroxyquinoline-2-carboxylate (**3s**)

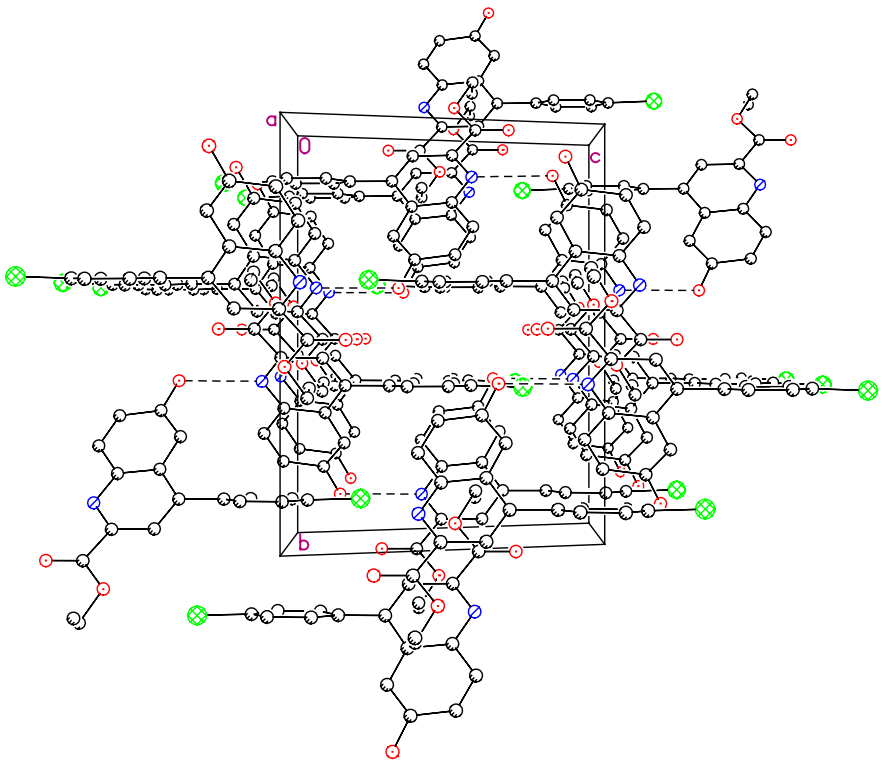


Table 1. Crystal data and structure refinement for **3s**

Identification code	**3s**
Empirical formula	C18 H14 Cl N O3
Formula weight	327.75
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 10.300(2) A alpha = 90 deg.
	b = 14.278(3) A beta = 114.40(3) deg.
	c = 11.790(2) A gamma = 90 deg.
Volume	1578.9(6) A^3
Z, Calculated density	4, 1.379 Mg/m^3
Absorption coefficient	0.256 mm^-1
F(000)	680
Crystal size	0.678 x 0.358 x 0.226 mm
Theta range for data collection	2.17 to 25.00 deg.
Limiting indices	0<=h<=12, 0<=k<=16, -14<=l<=12
Reflections collected / unique	2773 / 2773 [R(int) = 0.0000]
Completeness to theta = 25.00	100.00%
Absorption correction	Empirical
Max. and min. transmission	1.1907 and 0.7310
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2773 / 0 / 209
Goodness-of-fit on F^2	0.98
Final R indices [I>2sigma(I)]	R1 = 0.0493, wR2 = 0.1266
R indices (all data)	R1 = 0.0835, wR2 = 0.1374
Extinction coefficient	0.011(2)
Largest diff. peak and hole	0.313 and -0.314 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **3s**
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Cl(1)	112(1)	1245(1)	-2375(1)	84(1)
O(1)	2132(2)	-718(2)	5253(2)	70(1)
O(2)	3679(2)	-122(2)	7064(2)	86(1)

O(3)	6269(2)	3896(1)	3461(2)	59(1)
N(1)	4626(2)	1115(2)	5817(2)	43(1)
C(1)	348(5)	-1221(3)	5868(5)	136(2)
C(2)	1655(4)	-1421(3)	5877(4)	96(1)
C(3)	3149(3)	-123(2)	5953(3)	53(1)
C(4)	3555(3)	546(2)	5178(2)	44(1)
C(5)	2837(2)	560(2)	3871(2)	44(1)
C(6)	3198(2)	1212(2)	3192(2)	39(1)
C(7)	4294(2)	1868(2)	3853(2)	39(1)
C(8)	4699(2)	2607(2)	3277(2)	43(1)
C(9)	5789(3)	3201(2)	3962(2)	45(1)
C(10)	6513(3)	3079(2)	5266(2)	57(1)
C(11)	6131(3)	2387(2)	5845(2)	53(1)
C(12)	4999(2)	1766(2)	5165(2)	41(1)
C(13)	2438(2)	1215(2)	1806(2)	40(1)
C(14)	954(3)	1218(2)	1228(2)	48(1)
C(15)	236(3)	1212(2)	-50(3)	57(1)
C(16)	1013(3)	1205(2)	-757(2)	52(1)
C(17)	2480(3)	1179(2)	-223(2)	54(1)
C(18)	3188(3)	1181(2)	1063(2)	48(1)

Table 3. Bond lengths [A] and angles [deg] for **3s**

Cl(1)-C(16)	1.744(3)	O(1)-C(3)-C(4)	112.0(2)
O(1)-C(3)	1.336(3)	N(1)-C(4)-C(5)	123.4(2)
O(1)-C(2)	1.445(4)	N(1)-C(4)-C(3)	115.0(2)
O(2)-C(3)	1.193(3)	C(5)-C(4)-C(3)	121.6(2)
O(3)-C(9)	1.348(3)	C(6)-C(5)-C(4)	120.0(2)
N(1)-C(4)	1.325(3)	C(5)-C(6)-C(7)	118.1(2)
N(1)-C(12)	1.359(3)	C(5)-C(6)-C(13)	119.6(2)
C(1)-C(2)	1.372(5)	C(7)-C(6)-C(13)	122.3(2)
C(3)-C(4)	1.496(4)	C(8)-C(7)-C(12)	118.9(2)
C(4)-C(5)	1.408(3)	C(8)-C(7)-C(6)	123.7(2)
C(5)-C(6)	1.375(3)	C(12)-C(7)-C(6)	117.5(2)
C(6)-C(7)	1.425(3)	C(9)-C(8)-C(7)	120.8(2)
C(6)-C(13)	1.492(3)	O(3)-C(9)-C(8)	123.8(2)
C(7)-C(8)	1.408(3)	O(3)-C(9)-C(10)	116.3(2)
C(7)-C(12)	1.419(3)	C(8)-C(9)-C(10)	119.9(2)
C(8)-C(9)	1.373(3)	C(11)-C(10)-C(9)	120.5(2)

C(9)-C(10)	1.415(3)	C(10)-C(11)-C(12)	121.0(2)
C(10)-C(11)	1.349(4)	N(1)-C(12)-C(7)	123.3(2)
C(11)-C(12)	1.420(3)	N(1)-C(12)-C(11)	117.8(2)
C(13)-C(18)	1.387(3)	C(7)-C(12)-C(11)	118.9(2)
C(13)-C(14)	1.393(3)	C(18)-C(13)-C(14)	118.4(2)
C(14)-C(15)	1.378(4)	C(18)-C(13)-C(6)	121.0(2)
C(15)-C(16)	1.374(4)	C(14)-C(13)-C(6)	120.5(2)
C(16)-C(17)	1.376(4)	C(15)-C(14)-C(13)	121.3(3)
C(17)-C(18)	1.385(3)	C(16)-C(15)-C(14)	118.7(2)
C(3)-O(1)-C(2)	118.1(3)	C(15)-C(16)-C(17)	121.8(2)
C(4)-N(1)-C(12)	117.5(2)	C(15)-C(16)-Cl(1)	118.9(2)
C(1)-C(2)-O(1)	113.0(3)	C(17)-C(16)-Cl(1)	119.3(2)
O(2)-C(3)-O(1)	124.4(3)	C(16)-C(17)-C(18)	118.9(3)
O(2)-C(3)-C(4)	123.6(3)	C(17)-C(18)-C(13)	120.9(2)

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **3s**
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Cl(1)	100(1)	99(1)	29(1)	-2(1)	3(1)	-20(1)
O(1)	64(1)	80(2)	60(1)	27(1)	20(1)	-15(1)
O(2)	87(2)	120(2)	45(1)	28(1)	20(1)	-13(1)
O(3)	60(1)	62(1)	48(1)	0(1)	14(1)	-17(1)
N(1)	43(1)	56(1)	31(1)	1(1)	15(1)	5(1)
C(1)	149(4)	107(3)	226(6)	78(4)	152(4)	45(3)
C(2)	90(3)	96(3)	98(3)	52(2)	35(2)	-5(2)
C(3)	48(2)	69(2)	44(2)	19(2)	19(1)	13(1)
C(4)	41(1)	53(2)	38(1)	6(1)	17(1)	9(1)
C(5)	40(1)	51(2)	37(1)	1(1)	13(1)	-3(1)
C(6)	37(1)	48(1)	30(1)	0(1)	13(1)	1(1)
C(7)	38(1)	48(2)	31(1)	-2(1)	16(1)	2(1)
C(8)	45(1)	54(2)	28(1)	-4(1)	12(1)	-2(1)
C(9)	47(1)	51(2)	38(1)	-5(1)	19(1)	-7(1)
C(10)	54(2)	74(2)	37(1)	-16(1)	13(1)	-16(2)
C(11)	55(2)	74(2)	26(1)	-8(1)	12(1)	-10(2)
C(12)	40(1)	52(2)	31(1)	-4(1)	15(1)	3(1)
C(13)	43(1)	41(1)	30(1)	1(1)	10(1)	-6(1)
C(14)	45(1)	60(2)	38(1)	-1(1)	15(1)	-4(1)

C(15)	47(2)	65(2)	46(2)	-3(1)	7(1)	-4(1)
C(16)	65(2)	47(2)	31(1)	-3(1)	7(1)	-13(1)
C(17)	65(2)	61(2)	38(1)	-6(1)	24(1)	-15(2)
C(18)	44(1)	61(2)	37(1)	-2(1)	14(1)	-10(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **3s**

	x	y	z	U(eq)
H(3A)	5610	4251	3070	89
H(1A)	80	-1713	6284	204
H(1B)	-349	-1173	5023	204
H(1C)	398	-639	6290	204
H(2A)	1605	-2021	5474	115
H(2B)	2351	-1475	6733	115
H(5A)	2119	127	3465	52
H(8A)	4221	2694	2421	52
H(10A)	7258	3479	5727	68
H(11A)	6617	2315	6702	64
H(14A)	437	1224	1715	58
H(15A)	-756	1213	-427	68
H(17A)	2987	1160	-717	65
H(18A)	4179	1159	1434	58

Table 6. Hydrogen bonds for **3s** [A and deg.]

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(3)-H(3A)...O(2)#1	0.82	2.23	3.044(3)	174.1
O(3)-H(3A)...N(1)#1	0.82	2.48	2.868(3)	110.4

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+1/2,z-1/2