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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Table of Contents

1. Experimental Procedures.....	S2
1.1 Synthesis of γ -aryl- β , γ -unsaturated α -ketoesters 2	S2
1.2 Procedures for the discovery of the reaction.....	S3
1.3 Synthesis of 2-carboxy-4-aryl-quinolines 3	S5
1.4 Synthesis of Schiff's base 5t	S10
1.5 Procedures for reaction mechanism.....	S10
2. ^1H and ^{13}C NMR spectra of synthesized compounds.....	S12
2.1 γ -Aryl- β , γ -unsaturated α -ketoesters 2	S12
2.2 2-Aryl-4-carboxy-quinoline 4a	S17
2.3 2-Carboxy-4-aryl-quinolines 3	S18
2.4 Schiff's base 5t	S37
2.5 Schiff's base 5a	S38
3. Single-crystal X-ray analyses.....	S39
3.1 The single-crystal X-ray analysis of 4a	S39
3.2 The single-crystal X-ray analysis of 3a	S44
3.3 The single-crystal X-ray analysis of 3o	S50
3.4 The single-crystal X-ray analysis of 3s	S56

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

(3E)-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); ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) δ 182.4, 162.5, 148.6, 134.0, 131.7, 129.1, 129.1, 120.5, 53.0.

(3E)-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); ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) δ 182.2, 162.8, 162.7, 148.5, 131.1, 126.8, 118.0, 114.6, 55.5, 53.0.

(3E)-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); ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) δ 182.4, 162.6, 148.7, 142.5, 131.2, 129.8, 129.1, 119.4, 53.0, 21.6.

(3E)-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); ¹H NMR (300 MHz,

CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) δ 182.6, 162.0, 146.7, 137.6, 132.5, 130.1, 129.4, 121.0, 62.6, 14.1.

(3E)-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); ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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), (3E)-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 CH₂Cl₂, washed with 5 mL of saturated aqueous NaHCO₃ and then 5 mL of water, dried over anhydrous Na₂SO₄, 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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)⁺ C₁₉H₁₈NO₂: 292.1332, Found: 292.1335; Anal. Calcd. For C₁₉H₁₇NO₂: 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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)⁺ C₁₉H₁₈NO₂: 292.1332, Found: 292.1332; Anal. Calcd. For C₁₉H₁₇NO₂: 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 NaHCO₃ until pH = 7, and then was extracted with 20 mL of CH₂Cl₂. 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 CH₂Cl₂, washed with 5 mL of saturated aqueous NaHCO₃ and then 5 mL of water, dried over anhydrous Na₂SO₄, 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 CH₂Cl₂, washed with 5 mL of saturated aqueous NaHCO₃, dried over anhydrous Na₂SO₄, 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 CH₂Cl₂, washed with 5 mL of saturated aqueous NaHCO₃, dried over anhydrous Na₂SO₄, 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 CH₂Cl₂, washed with 5 mL of saturated aqueous NaHCO₃, dried over anhydrous Na₂SO₄, 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 CH₂Cl₂, washed with 5 mL of saturated aqueous NaHCO₃, dried over anhydrous Na₂SO₄, 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 CH₂Cl₂, and the solution was washed with 5 mL of saturated aqueous NaHCO₃, dried over anhydrous Na₂SO₄, 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; ^1H 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 $(\text{M} + \text{H})^+$ $\text{C}_{19}\text{H}_{18}\text{NO}_2$: 292.1332, Found: 292.1332; Anal. Calcd. For $\text{C}_{19}\text{H}_{17}\text{NO}_2$: 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; ^1H 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 $\text{C}_{18}\text{H}_{15}\text{NO}_3$: 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; ^1H 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 $\text{C}_{18}\text{H}_{15}\text{NO}_2$: 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; ^1H 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, $^1J_{\text{CF}}$ = 249.8 Hz, 1C), 149.4(d, $^3J_{\text{CF}}$ = 5.9 Hz, 1C), 146.9(d, $^4J_{\text{CF}}$ = 2.9 Hz, 1C), 145.2, 137.0, 133.8(d, $^3J_{\text{CF}}$ = 9.3 Hz, 1C), 129.4, 129.0, 128.9, 121.8, 120.7(d, $^2J_{\text{CF}}$ = 26.0 Hz, 1C), 109.3(d, $^2J_{\text{CF}}$ = 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^+ $\text{C}_{17}\text{H}_{12}\text{NO}_2\text{F}$: 281.0852, Found: 281.0851; Anal. Calcd. For $\text{C}_{17}\text{H}_{12}\text{NO}_2\text{F}$: 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; ^1H 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, CDCl₃) 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)⁺ C₁₇H₁₃N₂O₄: 309.0870, Found: 309.0869; Anal. Calcd. For C₁₇H₁₂N₂O₄: 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, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₂₁H₁₅NO₂: 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, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₂₁H₁₅NO₂: 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, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₇H₁₃NO₂: 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, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₈H₁₅NO₃: 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;

¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₈H₁₅NO₂: 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₈H₁₄ClNO₂: 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₇H₁₂N₂O₄: 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₉H₁₆ClNO₃: 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₉H₁₆ClNO₃: 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₈H₁₃Cl₂NO₂: 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₈H₁₃Cl₂NO₂: 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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 C₁₈H₁₃ClN₂O₄: 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; ¹H NMR (300 MHz, CDCl₃) 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); ¹³C NMR (75 MHz, CDCl₃) 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⁺ C₁₈H₁₄ClN₂O₄: 357.0637, Found: 357.0641; Anal. Calcd. For C₁₈H₁₄ClN₂O₄: 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; ¹H 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); ¹³C 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 $(\text{M} + \text{H})^+$ $\text{C}_{18}\text{H}_{15}\text{ClNO}_3$: 328.0735, Found: 328.0743; Anal. Calcd. For $\text{C}_{18}\text{H}_{14}\text{ClNO}_3$: 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); ^1H 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; ^1H 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^+ $\text{C}_{19}\text{H}_{19}\text{NO}_2$: 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 CH₂Cl₂, washed with 5 ml of saturated aqueous NaHCO₃, dried over anhydrous Na₂SO₄, 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. ^1H and ^{13}C NMR spectra of synthesized compounds

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

Current Data Parameters
NAME wpc-12-21
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041110
Time 3.50
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 262144
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.833 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 104.6
DM 66.000 usec
DE 6.00 usec
TE 287.4 K
D1 2.00000000 sec

CHANNEL f1 -
NUC1 ^1H
P1 9.30 usec
PL1 -1.00 dB
GB1 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

3D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.200 ppm
F1 300.133 Hz
F2P -0.800 ppm
F2 -60.00 Hz
PPM0 0.00000000 ppm/cm
HZCM 195.06761 Hz/cm

Current Data Parameters
NAME wpc-12-21
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041110
Time 4.01
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 262144
SOLVENT CDCl3
NS 16
DS 4
SWH 17908.514 Hz
FIDRES 0.274430 Hz
AQ 1.8248660 sec
RG 362
DM 27.900 usec
DE 6.00 usec
TE 287.5 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

CHANNEL f1 -
NUC1 ^{13}C
P1 9.40 usec
PL1 -1.00 dB
GB1 75.4752853 MHz

CHANNEL f2 -
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL18 18.00 dB
RFDR2 300.1360000 MHz

F2 - Processing parameters
SI 32768
SF 75.4767740 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3D NMR plot parameters
CX 80.00 cm
CY 8.00 cm
F1P 219.455 ppm
F1 18528.11 Hz
F2P -14.167 ppm
F2 -14.167 Hz
PPM0 11.31900 ppm/cm
HZCM 839.09356 Hz/cm

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

Current Data Parameters
NAME wpc-2-DME
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090226
Time 3.12
INSTRUM spect
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 101.6
DM 68.000 umsec
DE 6.00 umsec
TE 284.5 K
D1 2.00000000 sec

CHANNEL f1
NUC1 13C
P1 9.00 umsec
PL1 -1.00 dB
SFO1 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360534 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 300.133 Hz
F2P -0.800 ppm
F2 -60.03 Hz
PPHMM 0.00000 ppm/cm
HSCN 196.06761 Hz/cm

Current Data Parameters
NAME wpc-2-DME
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090226
Time 3.14
INSTRUM spect
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10
DS 4
SWH 17988.511 Hz
FIDRES 0.274430 Hz
AQ 1.0280660 sec
RG 256
DM 27.000 umsec
DE 6.00 umsec
TE 294.7 K
D1 2.00000000 sec
d11 0.00000000 sec
d12 0.00000000 sec

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

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P2 80.00 umsec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 75.4752953 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 182.203 Hz
F2P -16.187 ppm
F2 -14.46 Hz
PPHMM 11.51869 ppm/cm
HSCN 889.08868 Hz/cm

(3E)-4-(4-Methylphenyl)-2-oxobut-3-enoic acid methyl ester (2c)

Current Data Parameters
NAME: mpc-11-36
EXPNO: 10
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20041027
Time: 3.17
INSTRUM: mv300
PROBHD: 5 mm QNP 13C-1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 16
DS: 2
SWH: 5172.839 Hz
FIDRES: 0.094190 Hz
AQ: 0.3084660 sec
RG: 39.9
RW: 04.000 usec
DE: 6.00 usec
TE: 283.5 K
D1: 2.00000000 sec

CHANNEL f1
NUC1: 1H
P1: 0.30 usec
PL1: -1.00 dB
SFO1: 300.1360534 MHz

F2 - Processing parameters
SI: 32768
SF: 300.1360000 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: 300.133 Hz
F2P: -0.200 ppm
F2: -60.03 Hz
PFGH1: 0.52000 ppm/sec
H2OH: 195.06761 Hz/cm

Current Data Parameters
NAME: mpc-11-36
EXPNO: 11
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20041027
Time: 3.21
INSTRUM: mv300
PROBHD: 5 mm QNP 13C-1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 27
DS: 4
SWH: 17980.511 Hz
FIDRES: 0.271408 Hz
AQ: 1.8218698 sec
RG: 512
RW: 27.000 usec
DE: 6.00 usec
TE: 284.2 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: 75.4762693 MHz

CHANNEL f2
CPDPRG2: mltz15
NUC2: 1H
P2P2G2: 90.00 usec
PL2: -1.00 dB
PL12: 18.00 dB
PL13: 18.00 dB
SFO2: 300.1360000 MHz

F2 - Processing parameters
SI: 32768
SF: 75.4767460 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: 18238.11 Hz
F2P: -10.180 ppm
F2: -1446.03 Hz
PFGH1: 11.91808 ppm/sec
H2OH: 1881.08008 Hz/cm

(3E)-4-(4-Chlorophenyl)-2-oxobut-3-enoic acid ethyl ester (2d)

Current Data Parameters
NAME myc-10-48-2
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040721
Time 20.15
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 85536
SOLVENT CDCl3
NS 180
DS 2
SWH 5172.839 Hz
FIDRES 0.054190 Hz
AQ 5.3084590 sec
RG 128
RW 88.000 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec

CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360534 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 0.200 ppm
F1 300.133 Hz
F2P -0.800 ppm
F2 -60.03 Hz
PPHON 0.52000 ppm/cm
H2ON 195.05764 Hz/cm

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

F2 - Acquisition Parameters
Date_ 20040721
Time 20.18
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 85536
SOLVENT CDCl3
NS 70
DS 4
SWH 17389.511 Hz
FIDRES 0.3274438 Hz
AQ 1.5846590 sec
RG 2386.8
RW 27.880 usec
DE 6.00 usec
TE 300.2 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 75.4752853 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1360534 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 6.00 cm
F1P 210.155 ppm
F1 185208.11 Hz
F2P -15.167 ppm
F2 -14.460 Hz
PPHON 11.91809 ppm/cm
H2ON 833.08308 Hz/cm

(3E)-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 21.53
INSTRUM spect
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8172.833 Hz
FIDRES 0.094150 Hz
AQ 5.3084620 sec
RG 256
DM 81.000 usec
DE 9.00 usec
TE 291.2 K
D1 2.00000000 sec

--- CHANNEL f1 ---
NUC1 13C
P1 9.00 usec
PL1 -1.00 dB
SFO1 300.1318634 MHz

F2 - Processing parameters
SI 32768
SF 300.1300632 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CK 20.00 cm
CY 12.50 cm
F1P 10.310 ppm
F1 3099.84 Hz
F2P -0.002 ppm
F2 -126.57 Hz
PPM0 0.54165 ppm/cm
HZCM 152.62524 Hz/cm

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

F2 - Acquisition Parameters
Date_ 20040208
Time 22.28
INSTRUM spect
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1624
DS 4
SWH 17929.511 Hz
FIDRES 0.224450 Hz
AQ 1.8218650 sec
RG 6180.0
DM 27.000 usec
DE 6.00 usec
TE 291.2 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 75.4762853 MHz

--- CHANNEL f2 ---
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1318634 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
CK 80.00 cm
CY 12.50 cm
F1P 210.455 ppm
F1 18238.11 Hz
F2P -10.187 ppm
F2 -114.85 Hz
PPM0 11.91905 ppm/cm
HZCM 888.88008 Hz/cm

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

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

F2 - Acquisition Parameters
Date_ 20041004
Time 4.13
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 5172.835 Hz
FIDRES 0.034190 Hz
AQ 5.3084660 sec
RG 296
OW 84.000 usec
DE 6.00 usec
TE 297.6 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 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.200 ppm
F1 3001.33 Hz
F2P -0.800 ppm
F2 -60.63 Hz
PPHOM 0.02000 ppm/cm
H2OM 196.06761 Hz/cm

Current Data Parameters
NAME myc-11-20e1
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041004
Time 4.25
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10
DS 4
SWH 51728.511 Hz
FIDRES 0.224450 Hz
AQ 1.0240590 sec
RG 645.1
OW 27.000 usec
DE 6.00 usec
TE 297.6 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 75.4762603 MHz

--- CHANNEL f2 ---
DPHPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1318500 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 12.00 cm
F1P 210.455 ppm
F1 16530.14 Hz
F2P -18.107 ppm
F2 -1446.01 Hz
PPHOM 11.51809 ppm/cm
H2OM 899.09908 Hz/cm

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

Current Data Parameters
NAME: WYC-11-20
EXPNO: 10
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20041011
Time: 2.15
INSTRUM: BVS300
PROBHD: 5 mm QNP 13C-1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 16
DS: 2
SWH: 5372.839 Hz
FIDRES: 0.094190 Hz
AQ: 5.3084650 sec
RG: 1.00
OW: 0.000 usec
DE: 6.00 usec
TE: 297.4 K
D1: 2.00000000 sec

CHANNEL f1
NUC1: 1H
P1: 9.30 usec
PL1: -1.00 dB
SFO1: 300.1360534 MHz

F2 - Processing parameters
SI: 32768
SF: 300.1360534 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
FAP: 10.000 ppm
F1: 300.136 Hz
F2: -0.000 ppm
F3: -60.03 Hz
PPM0: 0.00000000 ppm/cm
H2O: 155.05761 Hz/cm

Current Data Parameters
NAME: WYC-11-20
EXPNO: 11
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20041011
Time: 2.16
INSTRUM: BVS300
PROBHD: 5 mm QNP 13C-1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 167
DS: 4
SWH: 17980.531 Hz
FIDRES: 0.224450 Hz
AQ: 1.0214550 sec
RG: 0.127
OW: 27.000 usec
DE: 6.00 usec
TE: 297.5 K
D1: 2.00000000 sec
d11: 0.00000000 sec
d12: 0.00000000 sec

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

CHANNEL f2
OPFPRG: waltz16
NUC2: 1H
PCPD: 80.00 usec
PL12: -1.00 dB
PL13: 18.00 dB
PL14: 18.00 dB
SFO2: 300.1360534 MHz

F2 - Processing parameters
SI: 32768
SF: 75.4752953 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
FAP: 215.000 ppm
F1: 100.625 Hz
F2: -10.000 ppm
F3: -704.00 Hz
PPM0: 11.00000 ppm/cm
H2O: 849.01210 Hz/cm

COC(=O)c1nc2cc(OC)ccc2c(c1)c3ccccc3

```

Current Data Parameters
NAME          wpc-8-002
EXPNO         1
PROCNO        10

Data - Acquisition Parameters
Date_         20030414
Time          9.09
INSTRUM       spect
PROBHD        5 mm QNP 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            66
DS            2
AQ            0.572839 Hz
FIDRES        0.094130 Hz
AQRESB        9.3084660 sec
RB            50.5
GB            0x.000 usec
DE            6.00 usec
TE            284.6 K
D1            2.00000000 sec

----- CHANNEL f1 -----
NUC1          13C          SH
P1            9.30 usec
PL1           -1.00 dB
SFO1          300.1316534 MHz

Data - 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 ch
CY            12.00 cm
P1            3001.33 Hz
F2            -0.000 ppm
F3            -60.13 Hz
PP4MNM        0.52000 ppm/cx
MAGN          156.05764 Hz/cm

```

```

Current Data Parameters
NAME          wxy-0-00E
EXPNO         11
PROCNO        1

F2 - Acquisition Parameters
Date_         20031118
Time          2.00
INSTRUM       spect
PROBHD        5 mm BIL 125-4
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
AQ            1.271
RG            627
DS            4
SWH            17959.841 Hz
FIDRES        0.274404 Hz
AQ            1.8265690 sec
RG            350
DE            27.800 MHz
DE            0.640000 sec
TE            300.2
D1            0.30000000 sec
d11            0.03000000 sec
d12            0.04000000 sec

===== CHANNEL f1 =====
NUC1           13C
P1            13.0
PL1           -1.80 dB
PL12          19.00 dB
PL13          19.00 dB
PL14          19.00 dB
PL15          19.00 dB
PL16          19.00 dB
PL17          19.00 dB
PL18          19.00 dB
PL19          19.00 dB
PL20          19.00 dB
PL21          19.00 dB
PL22          19.00 dB
PL23          19.00 dB
PL24          19.00 dB
PL25          19.00 dB
PL26          19.00 dB
PL27          19.00 dB
PL28          19.00 dB
PL29          19.00 dB
PL30          19.00 dB
PL31          19.00 dB
PL32          19.00 dB
PL33          19.00 dB
PL34          19.00 dB
PL35          19.00 dB
PL36          19.00 dB
PL37          19.00 dB
PL38          19.00 dB
PL39          19.00 dB
PL40          19.00 dB
PL41          19.00 dB
PL42          19.00 dB
PL43          19.00 dB
PL44          19.00 dB
PL45          19.00 dB
PL46          19.00 dB
PL47          19.00 dB
PL48          19.00 dB
PL49          19.00 dB
PL50          19.00 dB
PL51          19.00 dB
PL52          19.00 dB
PL53          19.00 dB
PL54          19.00 dB
PL55          19.00 dB
PL56          19.00 dB
PL57          19.00 dB
PL58          19.00 dB
PL59          19.00 dB
PL60          19.00 dB
PL61          19.00 dB
PL62          19.00 dB
PL63          19.00 dB
PL64          19.00 dB
PL65          19.00 dB
PL66          19.00 dB
PL67          19.00 dB
PL68          19.00 dB
PL69          19.00 dB
PL70          19.00 dB
PL71          19.00 dB
PL72          19.00 dB
PL73          19.00 dB
PL74          19.00 dB
PL75          19.00 dB
PL76          19.00 dB
PL77          19.00 dB
PL78          19.00 dB
PL79          19.00 dB
PL80          19.00 dB
PL81          19.00 dB
PL82          19.00 dB
PL83          19.00 dB
PL84          19.00 dB
PL85          19.00 dB
PL86          19.00 dB
PL87          19.00 dB
PL88          19.00 dB
PL89          19.00 dB
PL90          19.00 dB
PL91          19.00 dB
PL92          19.00 dB
PL93          19.00 dB
PL94          19.00 dB
PL95          19.00 dB
PL96          19.00 dB
PL97          19.00 dB
PL98          19.00 dB
PL99          19.00 dB
PL100         19.00 dB
PL101         19.00 dB
PL102         19.00 dB
PL103         19.00 dB
PL104         19.00 dB
PL105         19.00 dB
PL106         19.00 dB
PL107         19.00 dB
PL108         19.00 dB
PL109         19.00 dB
PL110         19.00 dB
PL111         19.00 dB
PL112         19.00 dB
PL113         19.00 dB
PL114         19.00 dB
PL115         19.00 dB
PL116         19.00 dB
PL117         19.00 dB
PL118         19.00 dB
PL119         19.00 dB
PL120         19.00 dB
PL121         19.00 dB
PL122         19.00 dB
PL123         19.00 dB
PL124         19.00 dB
PL125         19.00 dB
PL126         19.00 dB
PL127         19.00 dB
PL128         19.00 dB
PL129         19.00 dB
PL130         19.00 dB
PL131         19.00 dB
PL132         19.00 dB
PL133         19.00 dB
PL134         19.00 dB
PL135         19.00 dB
PL136         19.00 dB
PL137         19.00 dB
PL138         19.00 dB
PL139         19.00 dB
PL140         19.00 dB
PL141         19.00 dB
PL142         19.00 dB
PL143         19.00 dB
PL144         19.00 dB
PL145         19.00 dB
PL146         19.00 dB
PL147         19.00 dB
PL148         19.00 dB
PL149         19.00 dB
PL150         19.00 dB
PL151         19.00 dB
PL152         19.00 dB
PL153         19.00 dB
PL154         19.00 dB
PL155         19.00 dB
PL156         19.00 dB
PL157         19.00 dB
PL158         19.00 dB
PL159         19.00 dB
PL160         19.00 dB
PL161         19.00 dB
PL162         19.00 dB
PL163         19.00 dB
PL164         19.00 dB
PL165         19.00 dB
PL166         19.00 dB
PL167         19.00 dB
PL168         19.00 dB
PL169         19.00 dB
PL170         19.00 dB
PL171         19.00 dB
PL172         19.00 dB
PL173         19.00 dB
PL174         19.00 dB
PL175         19.00 dB
PL176         19.00 dB
PL177         19.00 dB
PL178         19.00 dB
PL179         19.00 dB
PL180         19.00 dB
PL181         19.00 dB
PL182         19.00 dB
PL183         19.00 dB
PL184         19.00 dB
PL185         19.00 dB
PL186         19.00 dB
PL187         19.00 dB
PL188         19.00 dB
PL189         19.00 dB
PL190         19.00 dB
PL191         19.00 dB
PL192         19.00 dB
PL193         19.00 dB
PL194         19.00 dB
PL195         19.00 dB
PL196         19.00 dB
PL197         19.00 dB
PL198         19.00 dB
PL199         19.00 dB
PL200         19.00 dB
PL201         19.00 dB
PL202         19.00 dB
PL203         19.00 dB
PL204         19.00 dB
PL205         19.00 dB
PL206         19.00 dB
PL207         19.00 dB
PL208         19.00 dB
PL209         19.00 dB
PL210         19.00 dB
PL211         19.00 dB
PL212         19.00 dB
PL213         19.00 dB
PL214         19.00 dB
PL215         19.00 dB
PL216         19.00 dB
PL217         19.00 dB
PL218         19.00 dB
PL219         19.00 dB
PL220         19.00 dB
PL221         19.00 dB
PL222         19.00 dB
PL223         19.00 dB
PL224         19.00 dB
PL225         19.00 dB
PL226         19.00 dB
PL227         19.00 dB
PL228         19.00 dB
PL229         19.00 dB
PL230         19.00 dB
PL231         19.00 dB
PL232         19.00 dB
PL233         19.00 dB
PL234         19.00 dB
PL235         19.00 dB
PL236         19.00 dB
PL237         19.00 dB
PL238         19.00 dB
PL239         19.00 dB
PL240         19.00 dB
PL241         19.00 dB
PL242         19.00 dB
PL243         19.00 dB
PL244         19.00 dB
PL245         19.00 dB
PL246         19.00 dB
PL247         19.00 dB
PL248         19.00 dB
PL249         19.00 dB
PL250         19.00 dB
PL251         19.00 dB
PL252         19.00 dB
PL253         19.00 dB
PL254         19.00 dB
PL255         19.00 dB
PL256         19.00 dB
PL257         19.00 dB
PL258         19.00 dB
PL259         19.00 dB
PL260         19.00 dB
PL261         19.00 dB
PL262         19.00 dB
PL263         19.00 dB
PL264         19.00 dB
PL265         19.00 dB
PL266         19.00 dB
PL267         19.00 dB
PL268         19.00 dB
PL269         19.00 dB
PL270         19.00 dB
PL271         19.00 dB
PL272         19.00 dB
PL273         19.00 dB
PL274         19.00 dB
PL275         19.00 dB
PL276         19.00 dB
PL277         19.00 dB
PL278         19.00 dB
PL279         19.00 dB
PL280         19.00 dB
PL281         19.00 dB
PL282         19.00 dB
PL283         19.00 dB
PL284         19.00 dB
PL285         19.00 dB
PL286         19.00 dB
PL287         19.00 dB
PL288         19.00 dB
PL289         19.00 dB
PL290         19.00 dB
PL291         19.00 dB
PL292         19.00 dB
PL293         19.00 dB
PL294         19.00 dB
PL295         19.00 dB
PL296         19.00 dB
PL297         19.00 dB
PL298         19.00 dB
PL299         19.00 dB
PL300         19.00 dB
PL301         19.00 dB
PL302         19.00 dB
PL303         19.00 dB
PL304         19.00 dB
PL305         19.00 dB
PL306         19.00 dB
PL307         19.00 dB
PL308         19.00 dB
PL309         19.00 dB
PL310         19.00 dB
PL
```

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

Current Data Parameters
NAME wyc-0-012
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031114
Time 5.26
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.839 Hz
FIDRES 0.094190 Hz
AQ 51.3884590 sec
RG 143.77
DM 64.000 usec
DE 6.00 usec
TE 283.0 K
D1 2.00000000 sec

CHANNEL f1
NUC1 13C
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.800 ppm
F1 300.133 Hz
F2P -0.800 ppm
F2 -60.03 Hz
PPHON 0.52000 ppm/cm
H2CH 156.05761 Hz/cm

Current Data Parameters
NAME wyc-0-012
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031114
Time 6.48
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 17900.511 Hz
FIDRES 0.274450 Hz
AQ 1.0218598 sec
RG 645.1
DM 27.000 usec
DE 6.00 usec
TE 283.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

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

CHANNEL f2
OPROBE mltz16
NUC2 1H
PCPD 90.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1360029 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 12.00 cm
F1P 210.155 ppm
F1 155.391 Hz
F2P -10.000 ppm
F2 -144.603 Hz
PPHON 11.91809 ppm/cm
H2CH 899.80099 Hz/cm

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

Current Data Parameters
NAME wpc-13-26
EXPNO 80
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090329
Time 4.07
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.835 Hz
FIDRES 0.094190 Hz
AQ 0.3084680 sec
RG 362
DM 64.000 usec
DE 6.00 uV
TE 285.9 K
D1 2.00000000 sec

CHANNEL f1
NUC1 13C
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360534 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.200 ppm
F1 3001.33 Hz
F2P -0.000 ppm
F2 -60.03 Hz
PPHON 0.02000 ppm/cm
H2H 155.06761 Hz/cm

Current Data Parameters
NAME wpc-13-26
EXPNO 28
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090329
Time 21.07
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 17928.511 Hz
FIDRES 0.274430 Hz
AQ 1.0218690 sec
RG 4000
DM 27.000 usec
DE 6.00 uV
TE 273.2 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 76.4702853 MHz

CHANNEL f2
CPDPRG2 hllt216
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 76.4702853 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 210.455 ppm
F1 18238.11 Hz
F2P -18.167 ppm
F2 -1446.01 Hz
PPHON 11.31005 ppm/cm
H2H 825.88028 Hz/cm

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

Current Data Parameters
NAME wyc-0-04
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031110
Time 3.22
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 5172.839 Hz
FIDRES 0.054190 Hz
AQ 9.3084560 sec
RG 322.5
CW 88.000 usec
DE 6.00 usec
TE 284.7 K
D1 2.0000000 sec

CHANNEL f1
NUC1 13C
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360534 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
FAP 10.000 ppm
F1 3001.33 Hz
FEP -0.000 ppm
F2 -60.03 Hz
PPHON 0.02000 ppm/cm
H2H 156.06761 Hz/cm

Current Data Parameters
NAME wyc-0-04
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031110
Time 3.28
INSTRUM mv300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 4
SWH 17980.514 Hz
FIDRES 0.274450 Hz
AQ 1.0218598 sec
RG 812.3
CW 27.000 usec
DE 6.00 usec
TE 280.0 K
D1 2.0000000 sec
d11 0.0000000 sec
d12 0.0000000 sec

CHANNEL f1
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SFO1 75.4752553 MHz

CHANNEL f2
DPRPGE Hs1216
NUC2 1H
DPRPGE 90.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL12 18.00 dB
SFO2 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 75.4752553 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
FAP 210.000 ppm
F1 16528.11 Hz
F2 -10.000 Hz
F2 -1446.00 Hz
PPHON 11.01905 ppm/cm
H2H 888.00000 Hz/cm

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

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

F2 - Acquisition Parameters
Date_ 20090822
Time 4.53
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 15.3084990 sec
RG 114
DM 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 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360534 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 2201.48 Hz
F2P -1.000 ppm
F2 -300.13 Hz
RG 0.60000 ppm/cm
HZCM 180.07602 Hz/cm

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

F2 - Acquisition Parameters
Date_ 20090822
Time 4.57
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 48
DS 4
SWH 17980.561 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 1024
DM 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 75.4752993 MHz

===== CHANNEL f2 =====
PROBHD2 mltiz18
NUC2 1H
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1360534 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 10.00 cm
F1P 210.000 ppm
F1 18220.87 Hz
F2P -5.000 ppm
F2 -877.34 Hz
RG 11.00000 ppm/cm
HZCM 800.14520 Hz/cm

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

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

F2 - Acquisition Parameters
Date_: 20090622
Time: 9.05
INSTRUM: av300
PROBHD: 5 mm DUL 13C-1
PULPROG: zg30
TD: 65536
SOLVENT: CDCl3
NS: 18
DS: 2
SWH: 6172.820 Hz
FIDRES: 0.034190 Hz
AQ: 0.3084950 sec
RG: 114
DN: 81.000 usec
DE: 6.00 usec
TE: 673.2 K
D1: 2.0000000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 9.30 uS
PL1: -1.00 dB
SFO1: 300.1314634 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: 2201.43 Hz
F2P: -1.000 ppm
F2: -300.13 Hz
PPHM: 0.00000 ppm/cm
HZCM: 180.07802 Hz/cm

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

F2 - Acquisition Parameters
Date_: 20090622
Time: 6.10
INSTRUM: av300
PROBHD: 5 mm DUL 13C-1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 144
DS: 4
SWH: 17980.541 Hz
FIDRES: 0.274439 Hz
AQ: 1.8219008 sec
RG: 4088
DN: 27.800 usec
DE: 6.00 usec
TE: 673.2 K
D1: 2.0000000 sec
d11: 0.0300000 sec
d12: 0.0000200 sec

===== CHANNEL f1 =====
NUC1: 13C
P1: 9.40 usec
PL1: -1.00 dB
SFO1: 75.4762903 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 60.00 usec
PL2: -1.00 dB
PL12: 18.00 dB
PL13: 18.00 dB
SFO2: 300.1312000 MHz

F2 - Processing parameters
SI: 32768
SF: 75.4677480 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: 18220.87 Hz
F2P: -5.000 ppm
F2: -277.34 Hz
PPHM: 11.00000 ppm/cm
HZCM: 830.14520 Hz/cm

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

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

F2 - Acquisition Parameters
Date_ 20050622
Time 3.46
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 6172.839 Hz
FIDRES 0.034150 Hz
AQ 5.3084660 sec
RG 161.3
DM 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 300.1318534 MHz

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

SD 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
FREQM 0.60000 ppm/cm
HZCM 180.07680 Hz/cm

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

F2 - Acquisition Parameters
Date_ 20050622
Time 3.48
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 117
DS 4
SWH 17580.541 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 4587.8
DM 27.500 usec
DE 0.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

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

===== CHANNEL f2 =====
CPDPRG2 h311218
NUC2 1H
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1318000 MHz

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

SD NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 210.000 ppm
F1 16220.87 Hz
F2P -5.000 ppm
F2 -377.34 Hz
FREQM 11.00000 ppm/cm
HZCM 830.14520 Hz/cm

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

Current Data Parameters
NAME vyc-15-15c
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090822
Time 4.20
INSTRUM mv300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 6178.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084980 sec
RG 828.1
DN 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 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300256 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 3201.48 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPM0 0.50000 ppm/cm
HZCM 180.07602 Hz/cm

Current Data Parameters
NAME vyc-15-15c
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090822
Time 4.26
INSTRUM mv300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 18
DS 4
SWH 17980.541 Hz
FIDRES 0.274439 Hz
AQ 1.8219000 sec
RG 3648.1
DN 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 75.4752962 MHz

===== CHANNEL f2 =====
CHOPRG2 Multiz18
NUC2 1H
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1312008 MHz

F2 - Processing parameters
SI 32768
SF 75.4677480 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 18220.87 Hz
F2P -5.000 ppm
F2 -877.34 Hz
PPM0 11.00000 ppm/cm
HZCM 800.14520 Hz/cm

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

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

F2 - Acquisition Parameters
Date_ 20090822
Time 4.01
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 6170.839 Hz
FIDRES 0.094190 Hz
AQ 0.3084990 sec
RG 887.4
DN 81.000 usec
DE 6.00 usec
TE 273.2 K
D1 2.00000000 sec

===== CHANNEL f1 =====
NUC1 13H
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1310534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300894 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 3201.48 Hz
F2P -1.800 ppm
F2 -300.13 Hz
FREQH 0.60000 ppm/cm
HZCM 180.07802 Hz/cm

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

F2 - Acquisition Parameters
Date_ 20090822
Time 4.08
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 17980.511 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 1024
DN 27.900 usec
DE 0.00 usec
TE 273.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

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

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677480 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 10220.67 Hz
F2P -5.000 ppm
F2 -277.34 Hz
FREQH 11.00000 ppm/cm
HZCM 830.14560 Hz/cm

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

Current Data Parameters
NAME vpc-8-28
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040226
Time 3.37
INSTRUM sv300
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.839 Hz
FIDRES 0.054190 Hz
AQ 9.3084650 sec
RG 128
OW 64.000 usec
DE 5.00 uV
TE 282.7 K
D1 2.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.30 usec
PL1 -1.00 dB
GFO1 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.200 ppm
F1 3001.33 Hz
F2P -0.800 ppm
F2 -60.83 Hz
PPHON 0.52000 ppm/c
HZON 156.06761 Hz/c

Current Data Parameters
NAME vpc-8-28
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040226
Time 3.44
INSTRUM sv300
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 360
DS 4
SWH 17529.518 Hz
FIDRES 0.274458 Hz
AQ 1.0218698 sec
RG 2560.3
OW 27.000 usec
DE 5.00 uV
TE 283.2 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
GFO1 75.4752653 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
BFO2 300.1360534 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 12.00 cm
F1P 210.455 ppm
F1 16529.11 Hz
F2P -1.000 ppm
F2 -14.400 Hz
PPHON 11.91808 ppm/c
HZON 899.88008 Hz/c

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

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

F2 - Acquisition Parameters
Date_ 20090822
Time 4.37
INSTRUM zvg300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084990 sec
RG 183
DN 81.000 usec
DE 6.00 usec
TE 273.2 K
D1 2.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1318534 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 3201.43 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPHM 0.60000 ppm/cm
HZCM 180.07802 Hz/cm

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

F2 - Acquisition Parameters
Date_ 20090822
Time 4.41
INSTRUM zvg300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 60
DS 4
SWH 17980.541 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 1825.6
DN 27.800 usec
DE 6.00 usec
TE 273.2 K
D1 2.0000000 sec
d11 0.0300000 sec
d12 0.0000200 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SFO1 75.4762903 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 75.4677480 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 18220.87 Hz
F2P -5.000 ppm
F2 -277.34 Hz
PPHM 11.00000 ppm/cm
HZCM 830.14520 Hz/cm

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

Current Data Parameters
NAME: vpc-8-07
EXPNO: 10
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20031125
Time: 21.24
INSTRUM: sv300
PROBHD: 5 mm QNP 13C-1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 16
DS: 2
SWH: 8172.833 Hz
FIDRES: 0.094190 Hz
AQ: 9.3084590 sec
RG: 143.7
DM: 64.000 umsec
DE: 6.00 umsec
TE: 281.2 K
D1: 2.00000000 sec

CHANNEL f1
NUC1: 1H
P1: 9.30 umsec
PL1: -1.00 dB
SFO1: 300.1360534 MHz

F2 - Processing parameters
SI: 32768
SF: 300.1300020 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
CX: 20.00 cm
CY: 12.00 cm
F1P: 10.200 ppm
F1: 300.133 Hz
F2P: -0.800 ppm
F2: -0.03 Hz
PPHON: 0.52000 ppm/cm
HZCN: 196.06761 Hz/cm

Current Data Parameters
NAME: vpc-8-07
EXPNO: 20
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20031125
Time: 22.10
INSTRUM: sv300
PROBHD: 5 mm QNP 13C-1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 400
DS: 4
SWH: 17980.511 Hz
FIDRES: 0.074438 Hz
AQ: 1.8218598 sec
RG: 250
DM: 27.890 umsec
DE: 6.00 umsec
TE: 281.2 K
D1: 2.00000000 sec
d11: 0.03000000 sec
d12: 0.00002000 sec

CHANNEL f1
NUC1: 13C
P1: 9.40 umsec
PL1: -1.00 dB
SFO1: 75.4762953 MHz

CHANNEL f2
PULPROG: zgpg30
NUC2: 1H
PCPD2: 90.00 umsec
PL2: -1.00 dB
PL12: 18.00 dB
PL18: 18.00 dB
SFO2: 300.1360534 MHz

F2 - Processing parameters
SI: 32768
SF: 75.4762953 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: 210.155 ppm
F1: 18539.11 Hz
F2P: -18.000 ppm
F2: -14.000 Hz
PPHON: 11.91809 ppm/cm
HZCN: 899.88898 Hz/cm

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

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

Current Data Parameters
NAME myc-0-61
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031126
Time 4.09
INSTRUM svt300
PROBHD 5 mm DUL 130-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.839 Hz
FIDRES 0.094150 Hz
AQ 5.308460 sec
RG 183
DN 81.000 usec
DE 5.00 uVpp
TE 282.4 K
D1 2.0000000 sec

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

F2 - Processing parameters
SI 32768
SF 300.1289968 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.428 ppm
F1 3129.58 Hz
F2P -0.380 ppm
F2 -119.61 Hz
PPHM 0.54070 ppm/cm
HZCM 162.27804 Hz/cm

F2 - Acquisition Parameters
Date_ 20031126
Time 4.10
INSTRUM svt300
PROBHD 5 mm DUL 130-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 131
DS 4
SWH 17589.611 Hz
FIDRES 0.274438 Hz
AQ 1.8240508 sec
RG 236
DN 27.800 usec
DE 8.00 uVpp
TE 282.4 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0000000 sec

--- CHANNEL f1 ---
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SFO1 75.4752833 MHz

--- CHANNEL f2 ---
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1318006 MHz

F2 - Processing parameters
SI 32768
SF 75.4672484 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 216.800 ppm
F1 16225.67 Hz
F2P -0.000 ppm
F2 -377.34 Hz
PPHM 10.00000 ppm/cm
HZCM 754.67229 Hz/cm

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

Current Data Parameters
NAME wyc-5-72
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031207
Time 17.11
INSTRUM spect
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 1572.839 Hz
FIDRES 0.094190 Hz
AQ 9.304690 sec
RG 203.2
OW 84.000 usec
DE 6.00 usec
TE 280.2 K
D1 2.00000000 sec

CHANNEL f1
NUC1 13C
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360534 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
FAP 10.200 ppm
F1 300.133 Hz
F2 -0.800 ppm
F3 -60.03 Hz
PPM0 0.000000 ppm/cm
HZ00 195.05764 Hz/cm

Current Data Parameters
NAME wyc-6-72
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031207
Time 18.13
INSTRUM spect
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 17980.514 Hz
FIDRES 0.024450 Hz
AQ 1.0218598 sec
RG 19521.3
OW 27.000 usec
DE 6.00 usec
TE 280.2 K
D1 2.00000000 sec
D11 0.00000000 sec
d12 0.00000000 sec

CHANNEL f1
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SFO1 75.4752553 MHz

CHANNEL f2
PULPROG zgpg30
NUC2 1H
PCPD0 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 75.4752553 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
FAP 10.200 ppm
F1 16529.15 Hz
F2 -10.000 ppm
F3 -1446.03 Hz
PPM0 11.01909 ppm/cm
HZ00 898.18058 Hz/cm

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

CCOC(=O)c1c(ClC2=CC=CC=C2)c3ccccc3n1[N+](=O)[O-]

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

Current Data Parameters
NAME vpc-8-03
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031126
Time 4.22
INSTRUM mv300
PROBHD 5 mm QNP-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 5172.839 Hz
FIDRES 0.094390 Hz
AQ 5.3094590 sec
RG 151.3
DM 0.000 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 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360534 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 11.003 ppm
F1 3300.38 Hz
F2P -0.096 ppm
F2 -175.82 Hz
PPHON 0.58245 ppm/cg
HZCN 174.80002 Hz/cg

Current Data Parameters
NAME vpc-8-03
EXPNO 80
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031126
Time 3.40
INSTRUM mv300
PROBHD 5 mm QNP-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 308
DS 4
SWH 17500.514 Hz
FIDRES 0.024438 Hz
AQ 1.8218698 sec
RG 1445.2
DM 27.000 usec
DE 6.00 usec
TE 282.3 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 75.4752550 MHz

CHANNEL f2
CPROGME waltz16
NUC2 1H
P2 90.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL18 18.00 dB
SFO2 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 75.4752550 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 210.155 ppm
F1 16538.11 Hz
F2P -15.187 ppm
F2 -1446.701 Hz
PPHON 11.91805 ppm/cg
HZCN 899.80002 Hz/cg

Schiff base 5t

Current Data Parameters
NAME wpc-18-Ba
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090705
Time 3.00
INSTRUM m300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.835 Hz
FIDRES 0.354190 Hz
AQ 5.3084660 sec
RG 40.3
RW 84.000 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec

CHANNEL f1
NUC1 13C
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 300.1360534 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 80.00 cm
CY 8.00 cm
F4P 10.000 ppm
F1 38.0136 Hz
F2P -0.000 ppm
F2 -150.06 Hz
PPHON 0.00000 ppm/cm
HZCM 105.07190 Hz/cm

Current Data Parameters
NAME wpc-18-Ba
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090705
Time 3.00
INSTRUM m300
PROBHD 5 mm QNP 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 17928.511 Hz
FIDRES 0.221430 Hz
AQ 1.8218698 sec
RG 6792.6
RW 27.880 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00420000 sec

CHANNEL f1
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SFO1 75.4752833 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL18 18.00 dB
SFO2 300.1360534 MHz

F2 - Processing parameters
SI 32768
SF 75.4752833 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 80.00 cm
CY 10.00 cm
F4P 240.455 ppm
F1 19528.11 Hz
F2P -18.167 ppm
F2 -144.46 Hz
PPHON 11.31908 ppm/cm
HZCM 888.88008 Hz/cm

(3E)-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	C ₁₉ H ₁₇ N O ₂
Formula weight	291.34
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, Aba2
Unit cell dimensions	a = 19.877(4) Å α = 90 deg.
	b = 18.927(4) Å β = 90 deg.
	c = 8.0012(16) Å γ = 90 deg.
Volume	3010.2(10) Å 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 > 2σ(I)]	R ₁ = 0.0474, wR ₂ = 0.1278
R indices (all data)	R ₁ = 0.0614, wR ₂ = 0.1322
Absolute structure parameter	-2(2)
Extinction coefficient	0.013(2)
Largest diff. peak and hole	0.241 and -0.232 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for **4a**

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} 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 [Å] 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 ($\text{\AA}^2 \times 10^3$) for **4a**

The anisotropic displacement factor exponent takes the form:

$$-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$$

	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 ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) 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	C ₁₉ H ₁₇ N O ₂
Formula weight	291.34
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 7.9843(16) Å alpha = 90 deg.
	b = 19.014(4) Å beta = 109.99(3) deg.
	c = 10.550(2) Å gamma = 90 deg.
Volume	1505.1(5) Å 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.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for **3a**

U(eq) is defined as one third of the trace of the orthogonalized
U_{ij} 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 [Å] 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 ($\text{\AA}^2 \times 10^3$) for **3a**

The anisotropic displacement factor exponent takes the form:

$$-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$$

	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 ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) 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 Å
Crystal system, space group	monoclinic, P2(1)/c
Unit cell dimensions	a = 10.3615(4) Å alpha = 90 deg.
	b = 8.4311(4) Å beta = 98.8060(7) deg.
	c = 18.5887(6) Å gamma = 90 deg.
Volume	1604.75(11) Å 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 > 2σ(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.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for **3o** U(eq) is defined as one third of the trace of the orthogonalized U_{ij} 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 [Å] 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 ($\text{\AA}^2 \times 10^3$) for **3o**

The anisotropic displacement factor exponent takes the form:

$$-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
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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 ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) 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	C ₁₈ H ₁₄ Cl N O ₃
Formula weight	327.75
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 10.300(2) Å alpha = 90 deg.
	b = 14.278(3) Å beta = 114.40(3) deg.
	c = 11.790(2) Å gamma = 90 deg.
Volume	1578.9(6) Å 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 > 2σ(I)]	R ₁ = 0.0493, wR ₂ = 0.1266
R indices (all data)	R ₁ = 0.0835, wR ₂ = 0.1374
Extinction coefficient	0.011(2)
Largest diff. peak and hole	0.313 and -0.314 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for **3s**

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} 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 [Å] 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 ($\text{\AA}^2 \times 10^3$) for **3s**

The anisotropic displacement factor exponent takes the form:

$$-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$$

	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 ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) 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** [$\text{\AA}$ and deg.]

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
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 $x, -y+1/2, z-1/2$