Supporting Information

A Regioselective One-step Synthesis of 4-
Fluoroalkylated Isoquinolines *via* Carbopalladation
Reaction of Fluorine-containing Alkynes

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Experimental Section

General. ^1H NMR and ^{13}C NMR spectra were measured with 500.13 MHz and 125.77 MHz spectrometer in a chloroform-*d* (CDCl_3) solution with tetramethylsilane (Me_4Si) as an internal reference. A 54.21 MHz spectrometer was used for determining ^{19}F NMR yield with internal C_6F_6 . CFCl_3 was used ($\delta_{\text{F}} = 0$) as an internal standard for ^{19}F NMR.

Materials. Dimethylformamide (DMF) was freshly distilled from calcium hydride (CaH_2). All chemicals were of reagent grade and, if necessary, were purified in the usual manner prior to use.

Representative procedure for the synthesis of isoquinoline derivatives

To a solution of $\text{Pd}(\text{PPh}_3)_4$ (0.026 g, 0.024 mmol), Na_2CO_3 (0.05 g, 0.488 mmol), the 2-iodobenzylidenamine (0.206 g, 0.72 mmol) in DMF (6 mL) was added 3,3,3-trifluoro-1-phenyl-1-propyne (83 mg, 0.488 mmol) at room temperature. After being heated for 8 h at 100 °C, the mixture was quenched with aq. NaHCO_3 , and extracted with Et_2O three times. The organic layer was dried over anhydrous Na_2SO_4 and concentrated in *vacuo*. The residue was chromatographed on silica gel using hexane-EtOAc (3 : 2) to afford 3-phenyl-4-trifluoromethylisoquinoline (116 mg, 0.42 mmol).

3-Phenyl-4-trifluoromethylisoquinoline (3b): Yield 87%; ^1H NMR (CDCl_3) δ 7.46 ~ 7.57 (5H, m), 7.72 (1H, t, $J = 7.46$ Hz), 7.86 (1H, t, $J = 7.41$ Hz), 8.08 (1H, d, $J = 8.05$ Hz), 8.28 (1H, d, $J = 7.65$ Hz), 9.39 (1H, s) ppm; ^{13}C NMR (CDCl_3) δ 117.6 (q, $J = 29.8$ Hz), 124.3 (q, $J = 3.9$ Hz), 124.7 (q, $J = 276.0$ Hz), 127.3, 127.8, 127.9, 128.3, 128.4, 128.9 (d, $J = 1.3$ Hz), 132.0, 133.2, 140.9, 153.1, 154.9 ppm; ^{19}F NMR (CDCl_3) δ -51.63 (3F, s) ppm; IR (KBr) ν 3032, 1695, 1622, 1576 cm^{-1} ; HRMS (FAB) calcd for $\text{C}_{16}\text{H}_{12}\text{F}_3\text{N}$ 274.0844, found (M+H) 274.0838.

3-(4-Chlorophenyl)-4-trifluoromethylisoquinoline (3a): Yield 87%; mp 103 ~ 105 °C; ^1H NMR (CDCl_3) δ 7.44 (2H, d, $J = 8.54$ Hz), 7.49 (2H, d, $J = 8.47$ Hz), 7.73 (1H, t, $J = 7.53$ Hz), 7.87 (1H, tt, $J = 7.84, 1.27$ Hz), 8.28 (1H, d, $J = 8.26$ Hz), 8.26 (1H, dd, $J = 8.58, 1.54$ Hz), 9.36 (1H, s) ppm; ^{13}C NMR (CDCl_3) δ 117.8 (q, $J = 30.1$ Hz), 124.3 (q, $J = 3.9$ Hz), 124.6 (q, $J = 276.0$ Hz), 127.4, 128.0, 128.2, 128.3, 130.4 (q, $J = 1.5$ Hz), 132.2, 133.2, 134.7, 139.3, 151.9 (q, $J = 2.1$ Hz), 155.0 ppm; ^{19}F NMR (CDCl_3) δ -51.58 (3F, s) ppm; IR (KBr) ν 3063, 3044, 1929, 1692 cm^{-1} ; HRMS (FAB) calcd for $\text{C}_{16}\text{H}_9^{35}\text{ClF}_3\text{N}$ 308.0454, found 308.0451.

3-(4-Methoxyphenyl)-4-trifluoromethylisoquinoline (3c): Yield 83%; mp 91 ~ 93 °C; ^1H NMR (CDCl_3) δ 2.43 (3H, s), 7.28 (2H, d, $J = 7.91$ Hz), 7.45 (2H, d, $J = 7.93$ Hz), 7.71 (1H, t, $J = 7.54$ Hz), 7.85 (1H, t, $J = 7.81$ Hz), 8.07 (1H, d, $J = 8.11$ Hz), 8.26 (1H, d, $J = 8.73$ Hz), 9.37 (1H, s) ppm; ^{13}C NMR (CDCl_3) δ 21.3, 117.4 (q, $J = 30.1$ Hz), 124.3 (q, $J = 3.6$ Hz), 124.8 (q, $J = 275.6$ Hz), 127.3, 127.6, 128.3, 128.7, 128.9 (q, $J = 1.5$ Hz), 132.0, 133.3, 138.0, 138.4, 153.3 (q, $J = 2.0$ Hz), 154.8 ppm; ^{19}F NMR (CDCl_3) δ -51.68 (3F, s) ppm; IR (KBr) ν 3031, 2360, 1608, 1444 cm^{-1} ; HRMS (FAB) calcd for $\text{C}_{17}\text{H}_{13}\text{F}_3\text{N}$ 288.1000, found (M+H) 288.0999.

3-(4-Methoxyphenyl)-4-trifluoromethylisoquinoline (3d): Yield 92%; mp 92 ~ 94 °C; ^1H NMR (CDCl_3) δ 3.88 (3H, s), 7.00 (2H, d, $J = 8.61$ Hz), 7.52 (2H, d, $J = 8.59$ Hz), 7.70 (1H, d, $J = 7.52$ Hz), 7.85 (1H, t, $J = 8.22$ Hz), 8.06 (1H, d, $J = 8.11$ Hz), 8.25 (1H, d, $J = 7.29$ Hz), 9.35 (1H, s) ppm; ^{13}C

NMR (CDCl₃) δ 55.2, 113.4, 117.0 (q, J = 30.1 Hz), 124.2 (q, J = 3.8 Hz), 124.8 (q, J = 275.8 Hz), 127.1, 127.5, 128.2, 130.5 (q, J = 1.5 Hz), 131.8, 133.3, 133.4, 152.8 (q, J = 2.0 Hz), 154.7, 160.0 ppm; ¹⁹F NMR (CDCl₃) δ -51.61 (3F, s) ppm; IR (KBr) ν 3004, 2839, 1608, 1444 cm⁻¹; HRMS (FAB) calcd for C₁₇H₁₂F₃NO 303.0871, found 303.0867; Anal. Calcd: C, 67.32; H, 3.99; N, 4.62. Found: C, 66.98; H, 4.09; N, 4.56; Crystal data C₁₇H₁₂F₃NO, M = 303.08, monoclinic, $P1$ (no. 2), a = 30.895(3) Å, b = 5.587(7) Å, c = 7.818(1) Å, β = 93.066(9)°, V = 1348.0(3) Å³, Z = 4, D_c = 1.494 g/cm⁻³, μ (CuK α) = 10.42 cm⁻¹, T = 198K, colorless; 12880 independent measured reflections, F^2 refinement, R_1 = 0.035, wR_2 = 0.105, 4662 independent observed absorption corrected reflections, $2\theta_{\max}$ = 133.5°.

3-(2-Methoxyphenyl)-4-trifluoromethylisoquinoline (3e): Yield 79%; mp 105 ~ 107 °C; ¹H NMR (CDCl₃) δ 3.74 (3H, s), 6.96 (1H, d, J = 8.29 Hz), 7.07 (1H, t, J = 7.44 Hz), 7.37 (1H, dd, J = 8.81, 1.50 Hz), 7.40 (1H, dt, J = 7.89, 1.67 Hz), 7.69 (1H, t, J = 7.54 Hz), 7.83 (1H, dt, J = 7.82, 1.14 Hz), 8.05 (1H, d, J = 8.13 Hz), 8.25 (1H, dd, J = 8.64, 1.48 Hz), 9.40 (1H, s) ppm; ¹³C NMR (CDCl₃) δ 55.3, 110.5, 119.3 (q, J = 29.7 Hz), 120.4, 124.3 (q, J = 3.5 Hz), 124.6 (q, J = 275.5 Hz), 127.5, 127.7, 128.2, 129.8, 130.0, 130.2, 131.8, 132.9, 149.3 (q, J = 2.5 Hz), 155.3, 156.5 ppm; ¹⁹F NMR (CDCl₃) δ -51.76 (3F, s) ppm; IR (KBr) ν 3080, 2999, 2968, 2841, 1624, 1663 cm⁻¹; HRMS (FAB) calcd for C₁₇H₁₃F₃NO 304.0949, found (M+H) 304.0952; Anal. Calcd: C, 67.32; H, 3.99; N, 4.62. Found: C, 66.97; H, 4.31; N, 4.62.

3-(3-Methoxyphenyl)-4-trifluoromethylisoquinoline (3f): Yield 70%; mp 103 ~ 105 °C; ¹H NMR (CDCl₃) δ 3.86 (3H, s), 7.00 (1H, d, J = 8.29, 2.49 Hz), 7.08 ~ 7.11 (2H, m), 7.37 (1H, t, J = 7.89 Hz), 7.73 (1H, t, J = 7.53 Hz), 7.87 (1H, t, J = 7.82 Hz), 7.73 (1H, t, J = 7.53 Hz), 7.87 (1H, t, J = 7.82 Hz), 8.09 (1H, d, J = 8.10 Hz), 8.27 (1H, d, J = 7.31 Hz), 9.38 (1H, s) ppm; ¹³C NMR (CDCl₃) δ 55.3, 114.3, 114.4, 117.6 (q, J = 29.8 Hz), 121.5, 124.3 (q, J = 3.7 Hz), 124.7 (q, J = 275.8 Hz), 127.4, 127.8, 128.3, 129.0, 132.1, 133.2, 142.2, 153.0 (q, J = 1.8 Hz), 154.9, 159.2 ppm; ¹⁹F NMR (CDCl₃) δ -55.30 (3F, s) ppm; IR (KBr) ν 2999, 2968, 1622, 1605 cm⁻¹; HRMS (FAB) calcd for C₁₇H₁₃F₃NO 304.0949, found (M+H) 304.0944.

3-(4-Ethoxycarbonylphenyl)-4-trifluoromethylisoquinoline (3g): Yield 48%; mp 70 ~ 72 °C; ¹H NMR (CDCl₃) δ 1.43 (3H, t, J = 7.13 Hz), 4.42 (2H, q, J = 7.12 Hz), 7.61 (2H, d, J = 8.17 Hz), 7.76 (1H, t, J = 7.53 Hz), 7.90 (1H, t, J = 7.83 Hz), 8.11 (1H, d, J = 8.14 Hz), 8.16 (2H, d, J = 8.19 Hz), 8.28 (1H, d, J = 8.34 Hz), 9.40 (1H, s) ppm; ¹³C NMR (CDCl₃) δ 14.3, 61.1, 118.0 (q, J = 30.3 Hz), 124.3 (q, J = 3.7 Hz), 124.5 (q, J = 275.8 Hz), 127.6, 128.1, 128.4, 129.0, 129.2, 130.4, 132.3, 133.1, 145.2, 152.1 (q, J = 2.26 Hz), 155.1, 166.3 ppm; ¹⁹F NMR (CDCl₃) δ -51.61 (3F, s) ppm; IR (KBr) ν 2968, 1717, 1618, 1574 cm⁻¹; HRMS (FAB) calcd for C₁₉H₁₅F₃NO₂ 346.1055, found (M+H) 346.1059; Anal. Calcd: C, 66.09; H, 4.09; N, 4.06. Found: C, 65.74; H, 4.13; N, 3.99.

3-(4-Nitrophenyl)-4-trifluoromethylisoquinoline (3h): Yield 40%; mp 116 ~ 118 °C; ¹H NMR (CDCl₃) δ 7.71 (2H, d, J = 8.58 Hz), 7.80 (1H, t, J = 7.55 Hz), 7.93 (1H, t, J = 7.84 Hz), 8.14 (1H, d, J = 8.10 Hz), 8.30 (1H, d, J = 8.45 Hz), 8.34 (2H, d, J = 8.58 Hz), 9.41 (1H, s) ppm; ¹³C NMR (CDCl₃) δ 118.4 (q, J = 30.2 Hz), 123.2, 124.4 (q, J = 3.7 Hz), 124.4 (q, J = 276.0 Hz), 127.8, 128.5, 128.6, 130.0, 132.6, 132.9, 147.2, 147.8, 150.6 (q, J = 2.0 Hz), 155.3 ppm; ¹⁹F NMR (CDCl₃) δ -51.55 (3F, s) ppm; IR (KBr) ν 3421,

3057, 1622, 1601 cm^{-1} ; HRMS (FAB) calcd for $\text{C}_{16}\text{H}_{10}\text{F}_3\text{N}_2\text{O}_2$ 319.0694, found (M+H) 319.0699.

3-(3-Phenylpropyl)-4-trifluoromethylisoquinoline (3i): The product could not be purified completely, so that only observed unique signal is shown as follows; ^{19}F NMR (CDCl_3) δ -53.07 (3F, s) ppm.

3-(4-Chlorophenyl)-4-difluoromethylisoquinoline (3j): Yield 83%; mp 77 ~ 79 $^{\circ}\text{C}$; ^1H NMR (CDCl_3) δ 6.96 (1H, t, J = 53.36 Hz), 7.51 (2H, d, J = 8.45 Hz), 7.56 (2H, d, J = 8.41 Hz), 7.72 (1H, t, J = 7.53 Hz), 7.84 (1H, t, J = 7.20 Hz), 8.07 (1H, d, J = 8.13 Hz), 8.50 (1H, d, J = 8.54 Hz), 9.36 (1H, s) ppm; ^{13}C NMR (CDCl_3) δ 115.7 (t, J = 237.4 Hz), 120.4 (t, J = 22.6 Hz), 125.0 (t, J = 4.2 Hz), 127.9, 128.2, 128.3, 128.8, 131.3, 131.7, 133.0, 135.3, 136.9, 152.2 (t, J = 8.0 Hz), 155.0 ppm; ^{19}F NMR (CDCl_3) δ -106.56 (d, J = 53.65 Hz) ppm; IR (KBr) ν 3005, 1618, 1578, 1562 cm^{-1} ; HRMS (FAB) calcd for $\text{C}_{16}\text{H}_{10}\text{ClF}_2\text{N}$ 290.0548, found (M+H) 290.0544.

3-(4-Chlorophenyl)-4-Hexafluoropropylisoquinoline (3k): The product could not be purified completely, so that only observed unique signals are shown as follows; ^{19}F NMR (CDCl_3) δ -97.98 (2F, t, J = 8.75 Hz), -126.65 (2F, s), -137.17 (2F, dt, J = 50.83, 8.47 Hz) ppm.

7-Methoxy-3-(4-methoxyphenyl)-4-trifluoromethylisoquinoline (3l): Yield 82%; mp 120 ~ 122 $^{\circ}\text{C}$; ^1H NMR (CDCl_3) δ 3.87 (3H, s), 3.99 (3H, s), 6.99 (2H, d, J = 8.73 Hz), 7.30 (1H, d, J = 2.6 Hz), 7.47 ~ 7.50 (3H, m), 8.15 (1H, dd, J = 9.41, 2.18 Hz), 9.26 (1H, s) ppm; ^{13}C NMR (CDCl_3) δ 55.2, 55.5, 105.4, 113.4, 117.2 (q, J = 30.1 Hz), 124.8, 124.9 (q, J = 275.8 Hz), 125.8 (q, J = 3.7 Hz), 128.7, 128.8, 130.4, 133.4, 150.8, 153.3, 158.4, 159.8 ppm; ^{19}F NMR (CDCl_3) δ -51.49 (3F, s) ppm; IR (KBr) ν 2962, 1622, 1607 cm^{-1} ; HRMS (FAB) calcd for $\text{C}_{18}\text{H}_{15}\text{F}_3\text{NO}_2$ 334.1055, found (M+H) 334.1063; Anal. Calcd: C, 64.86; H, 4.23, N; 4.20. Found: C, 64.51; H, 4.20, N; 4.17.

3-(4-Methoxyphenyl)-7-dimethylamino-4-trifluoromethylisoquinoline (3m): Yield 83%; mp 153 ~ 155 $^{\circ}\text{C}$; ^1H NMR (CDCl_3) δ 3.14 (6H, s), 3.87 (3H, s), 6.98 ~ 7.03 (3H, m), 7.45 ~ 7.51 (3H, m), 8.11 (1H, dd, J = 9.36, 2.07 Hz), 9.19 (1H, s) ppm; ^{13}C NMR (CDCl_3) δ 40.3, 55.3, 104.5, 113.3, 117.1 (q, J = 28.9 Hz), 121.1, 125.1 (q, J = 275.9 Hz), 125.1 (q, J = 3.8 Hz), 125.5, 129.3, 130.4 (d, J = 1.4 Hz), 133.7, 148.6, 149.0, 153.1, 159.6 ppm; ^{19}F NMR (CDCl_3) δ -51.69 (3F, s) ppm; IR (KBr) 3649, 3566, 2968, 1645 cm^{-1} ; HRMS (FAB) calcd for $\text{C}_{19}\text{H}_{17}\text{F}_3\text{N}_2\text{O}$ 346.1293, found 346.1284.

8-(4-Methoxyphenyl)-7-trifluoromethyl-1,3-dioxolo[4,5-g]isoquinoline (3n): Yield 76%; mp 137 ~ 139 $^{\circ}\text{C}$; ^1H NMR (CDCl_3) δ 3.87 (3H, s), 6.16 (2H, s), 6.98 (2H, d, J = 8.75 Hz), 7.25 (1H, s), 7.46 (2H, d, J = 8.64 Hz), 7.52 (1H, d, J = 2.04 Hz), 9.07 (1H, s) ppm; ^{13}C NMR (CDCl_3) δ 55.3, 101.3 (q, J = 4.28 Hz), 102.2, 103.5, 113.4, 117.1 (q, J = 29.3 Hz), 124.9, 124.9 (q, J = 275.8 Hz), 130.3 (d, J = 1.5 Hz), 132.1, 133.5, 148.4, 152.2 (q, J = 2.26 Hz), 152.2, 152.5, 159.8 ppm; ^{19}F NMR (CDCl_3) δ -52.24 (3F, s) ppm; IR (KBr) ν 2968, 2914, 1605 cm^{-1} ; HRMS (FAB) calcd for $\text{C}_{18}\text{H}_{12}\text{F}_3\text{NO}_3$ 347.0765, found 347.0775.

3-(4-Methoxyphenyl)-4-trifluoromethylbenzo[h]isoquinoline (3o): Yield 87%; mp 141 ~ 143 $^{\circ}\text{C}$; ^1H NMR (CDCl_3) δ 3.89 (3H, s), 7.02 ~ 7.05 (2H, m), 7.57 (2H, d, J = 8.63 Hz), 7.74 (1H, t, J = 7.44 Hz), 8.01 (1H, d, J = 7.94 Hz), 8.10 ~ 8.17 (2H, m), 8.84 (1H, d, J = 8.29 Hz), 10.19 (1H, s) ppm; ^{13}C NMR (CDCl_3) δ 55.3, 113.5, 117.9 (q, J = 29.6 Hz), 121.58 (q, J = 3.8 Hz), 122.0, 123.6, 124.9 (q, J = 276.0 Hz), 128.0, 123.6, 124.9 (q, J = 276.0 Hz), 128.8, 128.9, 130.5, 131.4, 133.2, 133.4, 134.4, 148.5, 154.2,

160.1 ppm; ^{19}F NMR (CDCl_3) δ -50.97 (3F, s) ppm; IR (KBr) ν 2928, 2831, 2341, 1609 cm^{-1} ; HRMS (FAB) calcd for $\text{C}_{21}\text{H}_{15}\text{F}_3\text{NO}$ 353.1027, found 353.1031.

7-Fluoro-3-(4-methoxyphenyl)-4-trifluoromethylisoquinoline (3p): Yield 78%; mp 97 ~ 98 °C; ^1H NMR (CDCl_3) δ 3.88 (3 H, s), 7.01 (2H, d, J = 8.60 Hz), 7.32 (1H, t, J = 8.68 Hz), 7.53 (2H, d, J = 8.56 Hz), 7.77 (1H, q, J = 2.27 Hz), 8.02 (1H, d, J = 8.54 Hz) ppm; ^{13}C NMR (CDCl_3) δ 55.3, 111.4 (d, J = 18.7 Hz), 113.5, 117.8 (q, J = 16.1 Hz), 120.3 (q, J = 3.9 Hz), 124.6 (q, J = 275.7 Hz), 130.5 (d, J = 1.38 Hz), 132.3 (d, J = 8.8 Hz), 132.9, 134.8, 148.3 (d, J = 6.5 Hz), 153.9, 158.3, 160.2, 160.4 ppm; ^{19}F NMR (CDCl_3) δ -51.8 (3F, s) ppm; HRMS (FAB) calcd for $\text{C}_{17}\text{H}_{11}\text{F}_4\text{NO}$ 321.0777, found 321.0779.

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 PROCNO 1

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F2 - Processing parameters
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 LB 0.30 Hz
 GB 0
 PC 1.00
 ID NMR plot parameters
 CX 20.00 cm
 CY 5.70 cm
 F1P 11.000 ppm
 F1 5501.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07800 Hz/cm

0.10280
0.101341

1.76725

3a

9.38553
8.29026
8.27497
8.08611
8.07001
7.87886
7.86438
7.84922
7.84779
7.73259
7.71762
7.70278
7.56653
7.55411
7.49512
7.48474
7.47069
7.45009
7.44765
7.26057

1.0653
1.1103
1.1091
1.1863
5.5589

Integral

S7

-0.062

77.251
76.997
76.743

154.886
153.117
140.878
133.192
132.027
128.914
128.903
128.411
128.289
127.918
127.748
127.326
125.788
124.306
124.275
123.593
117.693
117.456

3a

Current Data Parameters
NAME m1-2.25
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20050225
Time 19.35
INSTRUM drx500
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 3649.1
DM 16.650 usec
DE 6.00 usec
TE 296.7 K
D1 0.50000000 sec
d11 0.03000000 sec
HCREST 0.00000000 sec
MCWPK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.70 usec
PL1 -2.00 dB
SF01 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 13.74 dB
PL13 13.74 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
SF 125.7577987 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 6.89 cm
FIP 200.000 ppm
F1 25151.56 Hz
F2P -10.000 ppm
F2 -1257.58 Hz
PPMCM 10.50000 ppm/cm
HZCM 1320.45679 Hz/cm

Current Data Parameters
 NAME miyabe-(p-Cl)
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040514
 Time 20.32
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 256
 DN 48.400 usec
 DE 6.00 usec
 TE 296.5 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300144 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 20.00 cm
 CY 5.27 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCH 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

0.08108

3b

9.36951
 8.09501
 8.07850
 7.87945
 7.87550
 7.86206
 7.85948
 7.75122
 7.73645
 7.49936
 7.49586
 7.48623
 7.48242
 7.45605
 7.45191
 7.44302
 7.43898
 7.25814

ppm

6S

Current Data Parameters
 NAME miyabe-(p-Cl)
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040514
 Time 20.40
 INSTRUM drx500
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 30030.029 Hz
 FIDRES 0.456222 Hz
 AQ 1.0912244 sec
 RG 1625.5
 DW 16.650 usec
 DE 6.00 usec
 TE 297.5 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCHRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577951 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 7.98 cm
 F1P 200.000 ppm
 F1 25151.56 Hz
 F2P -10.000 ppm
 F2 -1257.58 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1320.45679 Hz/cm

3b

Current Data Parameters
 NAME miyabe-04.6.15
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040615
 Time 17.40
 INSTRUM drx500
 PROBHD 5 mm Multinuc1
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 406.4
 DW 48.400 usec
 DE 6.00 usec
 TE 297.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCNWK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300134 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 5.33 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

Current Data Parameters
 NAME miyabe-04.6.15
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040615
 Time 17.48
 INSTRUM drx500
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT COCl3
 NS 256
 DS 2
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 724.1
 DW 16.650 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCPRK 0.01500000 sec

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

NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

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

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
 SF 125.7577932 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 CY 23.89 cm
 F1P 200.000 ppm
 F1 25151.56 Hz
 F2P -10.000 ppm
 F2 -1257.56 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1320.45679 Hz/cm

21.335

77.251
 76.997
 76.743

154.819
 138.386
 138.041
 133.338
 131.964
 128.943
 128.931
 128.665
 128.295
 127.633
 127.268
 124.332
 124.303

S12

ppm

ppm

Current Data Parameters
 NAME chae-R415
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 2004041E
 Time 21.42
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SAH 30030.029 Hz
 FIDRES 0.458222 Hz
 A3 1.0912244 sec
 RG 3649.1
 DW 16.650 usec
 DE 6.00 usec
 TE 297.4 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWK 0.01500000 sec

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

NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

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

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
 SF 125.7578015 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 CY 11.18 cm
 FIP 200.000 ppm
 F1 25151.56 Hz
 F2 -1257.58 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1320.45691 Hz/cm

55.238

3d

Current Data Parameters
 NAME miyabe-7-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040701
 Time 20.17
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 228.1
 DW 48.400 usec
 DE 6.00 usec
 TE 298.9 K
 D1 1.0000000 sec
 MCREST 0.0000000 sec
 MCWPK 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300200 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 20.00 cm
 CY 10.03 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

0.0850
 0.0835
 0.0800

3e

3.7461

3.0767

9.4014
 8.0620
 8.0457
 7.8358
 7.8332
 7.8185
 7.7062
 7.6907
 7.4028
 7.3864
 7.3810
 7.3780
 7.3660
 7.3631
 7.0884
 7.0874
 7.0736
 7.0587
 6.9771
 6.9605

1.0570
 1.0506
 1.0657
 1.0597
 1.0431
 1.0168
 1.0339
 1.0225

1.0000

Integral

S15

Current Data Parameters
 NAME miyabe-7-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040701
 Time 20.26
 INSTRUM drx500
 PROBD 5 mm Multinucl
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 3649.1
 DM 16.650 usec
 DE 6.00 usec
 TE 299.9 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577960 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 10.22 cm
 F1P 200.000 ppm
 F1 25151.56 Hz
 F2P -10.000 ppm
 F2 -1257.58 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1320.45679 Hz/cm

3e

55.328

76.748
 77.002
 77.256

110.498
 120.420
 124.308
 124.335
 127.493
 127.652
 127.652
 128.244
 129.794
 129.970
 131.823

155.253

Current Data Parameters
 NAME miyabe-m-OMe
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040714
 Time 18.44
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 724.1
 DW 48.400 usec
 DE 5.00 usec
 TE 298.4 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters

SI 32768
 SF 500.1300134 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 20.00 cm
 CY 3.74 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

0.00005
 0.07147

3.86486

9.38130
 8.27369
 8.10438
 8.08822
 7.88058
 7.87826
 7.86338
 7.75089
 7.73533
 7.39330
 7.37769
 7.36176
 7.26001
 7.11143
 7.09430
 7.08935
 7.01624
 7.01118
 6.99959
 6.99469

S17

Integral

1.0000

1.1349

1.0436

1.0980

1.0592

1.0930

2.0919

1.0244

2.9882

Current Data Parameters
 NAME miyabe-7-6
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040706
 Time 17.23
 INSTRUM drx500
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 3649.1
 DW 16.650 usec
 DE 6.00 usec
 TE 299.8 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577951 MHz
 MDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 9.90 cm
 FIP 200.000 ppm
 F1 25151.56 Hz
 F2 -10.000 ppm
 F2 -1257.58 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1320.45679 Hz/cm

3f

55.268

77.257
77.003
76.749

159.173
154.864
152.978
142.168
133.178
132.055
128.974
128.306
127.788
127.419
124.338
124.309
121.495
114.384
114.277

Current Data Parameters

NAME miyabe-ethoxycarbonyl
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040713
Time 21:54
INSTRUM drx500
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 574.7
DM 48.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

NUC1 1H
P1 10.30 usec
PL1 -6.00 dB
SF01 500.1330885 MHz

F2 - Processing parameters

SI 32768
SF 500.1300137 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 9.51 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P -1.000 ppm
F2 -500.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 275.07150 Hz/cm

615

Current Data Parameters
 NAME miyabe-ethoxy
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040716
 Time 10.01
 INSTRUM drx500
 PROBD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SMH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 3649.1
 DM 15.650 usec
 DE 6.00 usec
 TE 299.7 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWIRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577942 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 10.96 cm
 F1P 219.352 ppm
 F1 27585.16 Hz
 F2P -19.441 ppm
 F2 -2444.87 Hz
 PPMCM 11.93963 ppm/cm
 HZCM 1501.50159 Hz/cm

Current Data Parameters
 NAME miyabe-ND2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040714
 Time 18.39
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 1149.4
 DW 48.400 usec
 DE 6.00 usec
 TE 298.4 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300125 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 3.82 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

0.00029
 0.07191

1.59124

5.29985

7.26188
 7.70615
 7.72330
 7.80464
 7.82021
 7.93609
 8.13572
 8.15191
 8.33870
 8.35586

9.41324

3h

125

Integral

1.0000

1.9438

1.0346

0.9788

0.9741

0.9838

1.9525

ppm

Current Data Parameters
 NAME miyabe-nitro
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040716
 Time 11.56
 INSTRUM drx500
 PROBD 5 mm Multinuc1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDC13
 NS 256
 DS 2
 SMH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 812.7
 DM 16.650 usec
 DE 6.00 usec
 TE 299.8 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPOPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577942 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.33 cm
 FIP 219.352 ppm
 F1 27585.16 Hz
 F2 -19.441 ppm
 F2 -2444.87 Hz
 PPMCM 11.93963 ppm/cm
 HZCM 1501.50159 Hz/cm

Current Data Parameters
 NAME miyabe10.14 B
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20041014
 Time 13.48
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 287.4
 DW 48.400 usec
 DE 6.00 usec
 TE 297.2 K
 D1 1.0000000 sec
 MCREST 0.0000000 sec
 MCWPK 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.130885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300137 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 6.70 cm
 F1P 12.000 ppm
 F1 6001.56 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.65000 ppm/cm
 HZCM 325.08450 Hz/cm

0.0770
 0.0071
 0.0008
 -0.0057

4.0131
 3.9976
 3.9873
 3.8718

9.2621
 8.1697
 8.1653
 8.1509
 8.1465
 7.5059
 7.5006
 7.4972
 7.4918
 7.4868
 7.4834
 7.4783
 7.4730
 7.3059
 7.3006
 7.2599
 7.0035
 6.9994
 6.9901
 6.9860

3.0672
 3.0000

0.9694
 0.9966
 2.9766
 1.0172
 1.9684

Integral

Current Data Parameters
 NAME miyabe 10.19
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20041019
 Time 12.13
 INSTRUM drx500
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 30030.029 Hz
 FIDRES 0.456222 Hz
 AQ 1.0912244 sec
 RG 2048
 DM 16.650 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577987 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 8.75 cm
 F1P 199.900 ppm
 F1 25138.98 Hz
 F2P -0.100 ppm
 F2 -12.58 Hz
 PPMCM 10.00000 ppm/cm
 HZCM 1257.57800 Hz/cm

0.970

55.491
55.244

77.255
77.000
76.746

159.770
158.414
153.320
150.845
133.351
130.423
128.750
128.710
125.957
125.860
125.831
124.751
123.764
117.290
117.051
113.366
105.406

Current Data Parameters
 NAME chae-difluoro-cl
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040713
 Time 22.05
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 456.1
 DW 48.400 usec
 DE 6.00 usec
 TE 298.2 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRRK 0.01500000 sec

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

NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters

SI 32768
 SF 500.1300144 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 CY 4.15 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07150 Hz/cm

0.0001

3j

S25

Current Data Parameters
 NAME miyabe.7.12
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20050712
 Time 16.26
 INSTRUM drx500
 PROBD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SMH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 7298.2
 DM 16.650 usec
 DE 6.00 usec
 TE 300.4 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577942 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 0.00

1D NMR plot parameters
 CX 20.00 cm
 CY 21.63 cm
 F1P 219.352 ppm
 F1 27585.16 Hz
 F2P -19.441 ppm
 F2 -2444.87 Hz
 PPMCM 11.93963 ppm/cm
 HZCM 1501.50159 Hz/cm

3j

Current Data Parameters

NAME miyabe-amine-anis
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20041119
Time 23.02
INSTRUM drx500
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 912.3
DM 48.400 usec
DE 6.00 usec
TE 298.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

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

NUC1 1H
P1 10.30 usec
PL1 -6.00 dB
SF01 500.1330885 MHz

F2 - Processing parameters

SI 32768
SF 500.1300137 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
CY 19.16 cm
F1P 12.000 ppm
F1 6001.56 Hz
F2P 0.100 ppm
F2 50.01 Hz
PPMCM 0.59500 ppm/cm
HZCM 297.57736 Hz/cm

S27

Current Data Parameters
 NAME: miyabe 11.22
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters

Date_: 20041122
 Time: 17.12
 INSTRUM: drx500
 PROBHD: 5 mm Multinucl
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 256
 DS: 2
 SWH: 30030.029 Hz
 FIDRES: 0.458222 Hz
 AQ: 1.0912244 sec
 RG: 3649.1
 DM: 16.650 usec
 DE: 6.00 usec
 TE: 298.8 K
 D1: 0.50000000 sec
 d11: 0.03000000 sec
 MCREST: 0.00000000 sec
 MCNRK: 0.01500000 sec

===== CHANNEL f1 =====
 NUC1: 13C
 P1: 5.70 usec
 PL1: -2.00 dB
 SF01: 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2: waltz16
 NUC2: 1H
 PCPD2: 100.00 usec
 PL2: -6.00 dB
 PL12: 13.74 dB
 PL13: 13.74 dB
 SF02: 500.1320005 MHz

F2 - Processing parameters
 SI: 32768
 SF: 125.7577951 MHz
 WDW: EM
 SSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 1.40

1D NMR plot parameters
 CX: 20.00 cm
 CY: 12.99 cm
 F1P: 200.000 ppm
 F1: 25151.56 Hz
 F2P: -10.000 ppm
 F2: -1257.56 Hz
 PPMCM: 10.50000 ppm/cm
 HZCM: 1320.45679 Hz/cm

Current Data Parameters
 NAME tricyclo-anis
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20041028
 Time 19.12
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 256
 DM 48.400 usec
 DE 6.00 usec
 TE 296.5 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300134 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 24.84 cm
 F1P 12.000 ppm
 F1 6001.56 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.65000 ppm/cm
 HZCM 325.08450 Hz/cm

Current Data Parameters
 NAME miyabe 11.2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20041102
 Time 21.41
 INSTRUM drx500
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SMH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 3649.1
 DW 16.650 usec
 DE 6.00 usec
 TE 301.9 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
 SF 125.7577942 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 CY 7.14 cm
 F1P 200.000 ppm
 F1 25151.56 Hz
 F2P -10.000 ppm
 F2 -1257.58 Hz
 PPMCH 10.50000 ppm/cm
 HZCM 1320.45679 Hz/cm

Current Data Parameters
 NAME miyabe 1.5
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20050105
 Time 13 59
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 812.7
 DN 48.400 usec
 DE 6.00 usec
 TE 295.5 K
 D1 1.0000000 sec
 MCREST 0.0000000 sec
 MCWK 0.0150000 sec

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

NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.130085 MHz

F2 - Processing parameters

SI 32768
 SF 500.1300137 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 CY 57.27 cm
 F1P 12.000 ppm
 F1 6001.56 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.65000 ppm/cm
 HZCM 325.08450 Hz/cm

0.1163
 0.0402
 0.0062
 0.0002
 -0.0068
 -0.1201

1.5973

3.8945

10.1868
 8.8472
 8.8306
 8.1544
 8.1500
 8.1222
 8.1035
 8.0134
 7.9975
 7.8162
 7.8021
 7.7996
 7.7584
 7.7441
 7.7287
 7.5780
 7.5607
 7.2603
 7.0384
 7.0346
 7.0250
 7.0211

2.9983

2.0119
 1.9959
 1.0027
 0.9980
 1.0349
 2.0080

1.0091

1.0000

S31

PDF

Integral

ppm

Current Data Parameters
 NAME miyabe 1.5 carbon
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050105
 Time 20.46
 INSTRUM drx500
 PROBD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 4597.6
 DM 16.650 usec
 DE 6.00 usec
 TE 296.3 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCMRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577951 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 13.00 cm
 F1P 200.000 ppm
 F1 25151.56 Hz
 F2P -10.000 ppm
 F2 -1257.58 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 1320.45679 Hz/cm

Current Data Parameters
 NAME miyabe.5.20-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050520
 Time 20.54
 INSTRUM drx500
 PROBD 5 mm Multinuc1
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 128
 DW 48.400 usec
 DE 6.00 usec
 TE 298.4 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300127 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.38 cm
 F1P 11.000 ppm
 F1 5501.43 Hz
 F2P -1.000 ppm
 F2 -500.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07800 Hz/cm

Current Data Parameters
 NAME miyabe_5.20-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20050520
 Time 21.03
 INSTRUM drx500
 PROBD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 1824.6
 DM 16.650 usec
 DE 6.00 usec
 TE 299.3 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCMRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.70 usec
 PL1 -2.00 dB
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -6.00 dB
 PL12 13.74 dB
 PL13 13.74 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
 SF 125.7577951 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 CY 10.47 cm
 F1P 219.344 ppm
 F1 27584.25 Hz
 F2P -19.448 ppm
 F2 -2445.78 Hz
 PPMCM 11.93963 ppm/cm
 HZCM 1501.50146 Hz/cm

3p

160.484
 160.430
 158.412
 154.857
 148.340
 148.288
 134.815
 132.923
 132.330
 132.260
 130.604
 130.593
 120.279
 120.248
 117.889
 117.761
 113.517
 111.468
 111.319
 77.256
 77.001
 76.747
 55.302

X-ray Structure Report

April 26, 2004

3d

Experimental

Data Collection

A colorless plate crystal of $C_{17}H_{12}F_3NO$ having approximate dimensions of 0.30 x 0.04 x 0.22 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with filtered $Cu-K\alpha$ radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $50.02 < 2\theta < 54.19^\circ$ corresponded to a primitive monoclinic cell with dimensions:

$$\begin{aligned}a &= 30.895(3) \approx \\b &= 5.5887(7) \approx \quad \beta = 93.066(9)^\circ \\c &= 7.818(1) \approx \\V &= 1348.0(3) \approx^3\end{aligned}$$

For $Z = 4$ and F.W. = 303.28, the calculated density is 1.49 g/cm^3 . The systematic absences of:

$$\begin{aligned}h0l: & h \pm 2n \\0k0: & k \pm 2n\end{aligned}$$

uniquely determine the space group to be:

$$P2_1/a \text{ (\#14)}$$

The data were collected at a temperature of $-75 \pm 1^\circ\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 133.5° . Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.24° with a take-off angle of 6.0° . Scans of $(1.84 + 0.30 \tan \theta)^\circ$ were made at a speed of $16.0^\circ/\text{min}$ (in ω). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 7 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 0.5 mm and the crystal to detector distance was 235 mm. The computer-controlled slits were set to 3.0 mm (horizontal) and 3.0 mm (vertical).

Data Reduction

Of the 3648 reflections that were collected, 2401 were unique ($R_{\text{int}} = 0.025$). The intensities of three representative reflections were measured after every 0 reflections. No decay correction was applied.

The linear absorption coefficient, μ , for Cu-K α radiation is 10.4 cm⁻¹. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction¹ was applied (coefficient = 0.008710).

Structure Solution and Refinement

The structure was solved by direct methods² and expanded using Fourier techniques³. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement⁴ on F^2 was based on 2401 observed reflections and 310 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.035$$

$$wR2 = [\sum (w (F_o^2 - F_c^2)^2) / \sum w(F_o^2)^2]^{1/2} = 0.105$$

The standard deviation of an observation of unit weight⁵ was 1.01. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.22 and -0.18 e⁻/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁶. Anomalous dispersion effects were included in F_{calc} ⁷; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁸. The values for the mass attenuation coefficients are those of Creagh and Hubbell⁹. All calculations were performed using the CrystalStructure^{10,11} crystallographic software package except for refinement, which was performed using SHELXL-97¹².

References

- (1) Larson, A.C. (1970), Crystallographic Computing, 291-294. F.R. Ahmed, ed. Munksgaard, Copenhagen (equation 22, with V replaced by the cell volume).
- (2) SIR2002: Burla, M.C., Camalli, M., Carrozzini, B., Cascarano, G.L., Giacovazzo, C., Polidori, G., Spagna, R. (2003).
- (3) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel,

R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least Squares function minimized: (SHELXL97)

$$\sum w(F_o^2 - F_c^2)^2 \quad \text{where } w = \text{Least Squares weights.}$$

(5) Standard deviation of an observation of unit weight:

$$[\sum w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$$

where: N_o = number of observations
 N_v = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J. ; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 3.6.0: Crystal Structure Analysis Package, Rigaku and Rigaku/MSD (2000-2004). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(11) CRYSTALS Issue 10: Watkin, D.J., Prout, C.K. Carruthers, J.R. & Betteridge, P.W. Chemical Crystallography Laboratory, Oxford, UK. (1996)

(12) SHELX97: Sheldrick, G.M. (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₁₇ H ₁₂ F ₃ NO
Formula Weight	303.28
Crystal Color, Habit	colorless, plate
Crystal Dimensions	0.30 X 0.04 X 0.22 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2 θ range)	25 (50.0 - 54.2 $^{\circ}$)
Omega Scan Peak Width at Half-height	0.24 $^{\circ}$
Lattice Parameters	a = 30.895(3) $\approx$ b = 5.5887(7) $\approx$ c = 7.818(1) $\approx$ β = 93.066(9) $^{\circ}$ V = 1348.0(3) $\approx^3$
Space Group	P2 ₁ /a (#14)
Z value	4
D _{calc}	1.494 g/cm ³
F ₀₀₀	624.00
μ (CuK α)	10.42 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku AFC7R
Radiation	CuK α ($\lambda = 1.54178 \text{ \AA}$)
Attenuator	Ni foil (factor = 9.33)
Take-off Angle	6.0 $^\circ$
Detector Aperture	3.0 mm horizontal 3.0 mm vertical
Crystal to Detector Distance	235 mm
Voltage, Current	50kV, 100mA
Temperature	-75.0 $^\circ$ C
Scan Type	ω -2 θ
Scan Rate	16.0 $^\circ$ /min (in ω) (up to 7 scans)
Scan Width	(1.84 + 0.30 tan θ) $^\circ$
2 θ_{max}	133.5 $^\circ$
No. of Reflections Measured	Total: 3648
Corrections	Unique: 3244 ($R_{\text{int}} = 0.025$) Lorentz-polarization Secondary Extinction (coefficient: 8.71000e-003)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1 / [\sigma^2(F_o^2) + (0.0659 \cdot P)^2 + 0.2895 \cdot P]$ where $P = (\text{Max}(F_o^2, 0) + 2F_c^2)/3$
$2\theta_{\text{max}}$ cutoff	133.5°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2401
No. Variables	310
Reflection/Parameter Ratio	7.75
Residuals: R (All reflections)	0.042
Residuals: R1 ($ I > 2.00\sigma(I)$)	0.035
Residuals: wR2 (All reflections)	0.105
Goodness of Fit Indicator	1.013
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.22 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.18 e ⁻ /Å ³

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$

atom	x	y	z	B_{eq}
F1	0.7695	0.1865	0.1007	3.530
F2	0.8192	0.3918	-0.0065	3.728
F3	0.8141	0.0156	-0.0615	4.106
O1	0.9749	0.8141	-0.1785	3.699
N1	0.9001	0.2199	0.4173	2.701
C1	0.8403	0.1214	0.2202	2.315
C2	0.8786	0.2428	0.2590	2.331
C3	0.8853	0.0645	0.5252	2.710
C4	0.8501	-0.0908	0.4910	2.428
C5	0.8390	-0.2711	0.6074	2.815
C6	0.8055	-0.4233	0.5677	3.124
C7	0.7818	-0.3994	0.4094	3.089
C8	0.7915	-0.2246	0.2946	2.740
C9	0.8260	-0.0626	0.3318	2.292
C10	0.8114	0.1790	0.0638	2.774
C11	0.9019	0.3958	0.1393	2.337
C12	0.9095	0.3165	-0.0250	2.692
C13	0.9339	0.4500	-0.1346	2.816
C14	0.9510	0.6673	-0.0800	2.684

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$ (continued)

atom	x	y	z	B_{eq}
C15	0.9443	0.7492	0.0852	2.889
C16	0.9201	0.6137	0.1927	2.655
C17	0.9860	0.7267	-0.3411	3.690

$$B_{\text{eq}} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos \gamma + 2U_{13}(aa^*cc^*)\cos \beta + 2U_{23}(bb^*cc^*)\cos \alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
F1	0.0295	0.0630	0.0407	0.0036	-0.0063	0.0058
F2	0.0445	0.0554	0.0407	0.0003	-0.0076	0.0153
F3	0.0575	0.0685	0.0290	0.0024	-0.0079	-0.0158
O1	0.0543	0.0466	0.0398	-0.0157	0.0035	0.0051
N1	0.0319	0.0447	0.0257	-0.0017	-0.0017	-0.0030
C1	0.0291	0.0360	0.0228	0.0034	0.0005	-0.0034
C2	0.0294	0.0347	0.0244	0.0040	0.0011	-0.0051
C3	0.0336	0.0451	0.0237	0.0020	-0.0029	-0.0030
C4	0.0294	0.0384	0.0245	0.0050	0.0025	-0.0040
C5	0.0369	0.0424	0.0278	0.0064	0.0031	0.0008
C6	0.0429	0.0396	0.0369	0.0024	0.0091	0.0020
C7	0.0382	0.0392	0.0403	-0.0040	0.0055	-0.0045
C8	0.0325	0.0410	0.0305	0.0004	0.0012	-0.0060
C9	0.0278	0.0346	0.0248	0.0050	0.0023	-0.0049
C10	0.0338	0.0446	0.0264	-0.0001	-0.0032	-0.0002
C11	0.0275	0.0338	0.0273	0.0027	-0.0007	-0.0019
C12	0.0390	0.0328	0.0305	-0.0045	0.0025	-0.0054
C13	0.0399	0.0390	0.0284	-0.0021	0.0045	-0.0040
C14	0.0322	0.0351	0.0343	-0.0012	-0.0025	0.0045

Table 2. Anisotropic Displacement Parameters (continued)

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C15	0.0398	0.0320	0.0374	-0.0014	-0.0044	-0.0036
C16	0.0355	0.0352	0.0297	0.0040	-0.0031	-0.0057
C17	0.0431	0.0585	0.0390	-0.0017	0.0062	0.0104

The general temperature factor expression: $\exp(-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a*b*U_{12}hk + 2a*c*U_{13}hl + 2b*c*U_{23}kl))$

Table 3. Bond lengths ($\approx$)

atom	atom	distance	atom	atom	distance
F1	C10	1.3423	F2	C10	1.3379
F3	C10	1.3455	O1	C14	1.3677
O1	C17	1.4211	N1	C2	1.3795
N1	C3	1.3096	C1	C2	1.3839
C1	C9	1.4335	C1	C10	1.5092
C2	C11	1.4835	C3	C4	1.4069
C4	C5	1.4118	C4	C9	1.4244
C5	C6	1.3634	C6	C7	1.4097
C7	C8	1.3707	C8	C9	1.4157
C11	C12	1.3902	C11	C16	1.3947
C12	C13	1.3891	C13	C14	1.3833
C14	C15	1.3963	C15	C16	1.3813

Table 4. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C10	F2	105.0	F1	C10	F3	106.0
F1	C10	C1	111.6	F2	C10	F3	106.5
F2	C10	C1	114.2	F3	C10	C1	112.8
O1	C14	C13	124.2	O1	C14	C15	115.8
C17	O1	C14	117.4	N1	C2	C1	121.1
N1	C2	C11	113.1	C3	N1	C2	118.1
N1	C3	C4	125.4	C9	C1	C2	120.4
C10	C1	C2	122.3	C1	C2	C11	125.7
C1	C9	C8	125.9	C10	C1	C9	117.3
C1	C9	C4	116.6	C2	C11	C12	120.7
C2	C11	C16	121.1	C3	C4	C9	117.4
C3	C4	C5	122.0	C9	C4	C5	120.6
C4	C5	C6	120.4	C4	C9	C8	117.5
C5	C6	C7	119.5	C6	C7	C8	121.4
C7	C8	C9	120.6	C11	C12	C13	121.5
C16	C11	C12	118.0	C11	C16	C15	121.3
C12	C13	C14	119.5	C13	C14	C15	120.0
C14	C15	C16	119.7				

Table 5. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(17)	O(1)	C(14)	C(13)	-6.1(2)	C(17)	O(1)	C(14)	C(15)	174.0(1)
C(3)	N(1)	C(2)	C(1)	5.6(2)	C(3)	N(1)	C(2)	C(11)	-171.3(1)
C(2)	N(1)	C(3)	C(4)	3.0(2)	C(9)	C(1)	C(2)	N(1)	-10.9(2)
C(9)	C(1)	C(2)	C(11)	165.6(1)	C(10)	C(1)	C(2)	N(1)	167.5(1)
C(10)	C(1)	C(2)	C(11)	-16.0(2)	C(2)	C(1)	C(9)	C(4)	7.5(2)
C(2)	C(1)	C(9)	C(8)	-169.4(1)	C(10)	C(1)	C(9)	C(4)	-171.0(1)
C(10)	C(1)	C(9)	C(8)	12.1(2)	C(2)	C(1)	C(10)	F(1)	-135.9(1)
C(2)	C(1)	C(10)	F(2)	-17.0(2)	C(2)	C(1)	C(10)	F(3)	104.9(2)
C(9)	C(1)	C(10)	F(1)	42.5(2)	C(9)	C(1)	C(10)	F(2)	161.4(1)
C(9)	C(1)	C(10)	F(3)	-76.7(2)	N(1)	C(2)	C(11)	C(12)	129.4(1)
N(1)	C(2)	C(11)	C(16)	-45.1(2)	C(1)	C(2)	C(11)	C(12)	-47.3(2)
C(1)	C(2)	C(11)	C(16)	138.1(2)	N(1)	C(3)	C(4)	C(5)	173.0(2)
N(1)	C(3)	C(4)	C(9)	-6.0(2)	C(3)	C(4)	C(5)	C(6)	-177.9(1)
C(9)	C(4)	C(5)	C(6)	1.1(2)	C(3)	C(4)	C(9)	C(1)	0.5(2)
C(3)	C(4)	C(9)	C(8)	177.7(1)	C(5)	C(4)	C(9)	C(1)	-178.5(1)
C(5)	C(4)	C(9)	C(8)	-1.4(2)	C(4)	C(5)	C(6)	C(7)	-0.1(2)
C(5)	C(6)	C(7)	C(8)	-0.6(3)	C(6)	C(7)	C(8)	C(9)	0.2(3)
C(7)	C(8)	C(9)	C(1)	177.6(1)	C(7)	C(8)	C(9)	C(4)	0.7(2)
C(2)	C(11)	C(12)	C(13)	-175.4(1)	C(16)	C(11)	C(12)	C(13)	-0.7(2)
C(2)	C(11)	C(16)	C(15)	175.6(1)	C(12)	C(11)	C(16)	C(15)	0.9(2)
C(11)	C(12)	C(13)	C(14)	-0.2(2)	C(12)	C(13)	C(14)	O(1)	-178.9(1)
C(12)	C(13)	C(14)	C(15)	1.0(2)	O(1)	C(14)	C(15)	C(16)	179.1(1)
C(13)	C(14)	C(15)	C(16)	-0.9(2)	C(14)	C(15)	C(16)	C(11)	-0.1(2)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 6. Distances beyond the asymmetric unit out to 3.60 $\approx$

atom	atom	distance	atom	atom	distance
F1	F2	2.1263	F1	F3	2.1470
F1	C1	2.3597	F1	C2	3.5416
F1	C7 ¹⁾	3.3507	F1	C8	2.8161
F1	C9	2.8132	F2	F3	2.1506
F2	F3 ¹⁾	3.5148	F2	C1	2.3927
F2	C2	2.8207	F2	C8 ¹⁾	3.3293
F2	C11	2.7417	F2	C12	2.8312
F3	F2 ²⁾	3.5148	F3	C1	2.3797
F3	C2	3.3654	F3	C8	3.2010
F3	C9	3.1086	F3	C12	3.3921
O1	C13	2.4309	O1	C15	2.3418
N1	C1	2.4069	N1	C4	2.4148
N1	C9	2.8320	N1	C11	2.3888
N1	C12	3.5268	N1	C16	2.9025
C1	C3	2.7150	C1	C4	2.4317
C1	C7 ¹⁾	3.5930	C1	C8	2.5370
C1	C11	2.5521	C1	C12	3.1427
C2	C3	2.3066	C2	C4	2.7770
C2	C9	2.4445	C2	C10	2.5348

Table 6. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C2	C12	2.4977	C2	C16	2.5062
C3	C5	2.4654	C3	C9	2.4196
C3	C11	3.5998	C4	C6	2.4087
C4	C7	2.7723	C4	C8	2.4275
C5	F3 ³⁾	3.1734	C5	C7	2.3955
C5	C8	2.7956	C5	C9	2.4632
C6	F2 ⁴⁾	3.4893	C6	C8	2.4250
C6	C9	2.8270	C7	F1 ²⁾	3.3507
C7	C1 ²⁾	3.5930	C7	C9	2.4208
C8	F2 ²⁾	3.3293	C8	C10	2.9731
C9	C10	2.5136	C10	C11	3.0768
C10	C12	3.2378	C11	C13	2.4250
C11	C14	2.7967	C11	C15	2.4193
C12	C14	2.3948	C12	C15 ²⁾	3.4434
C12	C15	2.7655	C12	C16	2.3876
C13	C15	2.4072	C13	C16	2.7721
C13	C17	2.8049	C14	C16	2.4016
C14	C17	2.3836	C15	C12 ¹⁾	3.4434

Symmetry Operators:

(1) X,Y+1,Z

(2) X,Y-1,Z

(3) $X, Y, Z+1$

(4) $X, Y-1, Z+1$