Supplementary Information for the Manuscript:

Synthesis and Cross-coupling Reactions of 7-Azaindoles

via a New Donor-Acceptor Cyclopropane

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Experimental

General

Melting points were determined using a Gallenkamp melting point apparatus and are uncorrected. Infrared spectra were obtained as thin films on NaCl plates using a Bruker Vector 33 FT-IR instrument. ^1H and ^{13}C NMR experiments were performed on a Varian Inova 600 or 400 or Mercury 400 instrument as solutions in CDCl_3 (referenced to 7.26ppm for ^1H and 77.0ppm for ^{13}C) and acetone- d_6 (referenced to 2.05ppm for ^1H and 29.9ppm for ^{13}C). Mass spectra were obtained on a Finnigan MAT 8200 spectrometer at 70eV. Analytical TLC was performed on silica gel (EM Science, silica gel 60 F₂₅₄) coated on aluminum or glass plates visualizing by UV and by staining with acidic p-anisaldehyde stain. Flash chromatography was performed using silica gel purchased from Silicycle Chemical Division Inc. (230-400 mesh).

Dichloromethane, tetrahydrofuran, N,N-dimethylformamide, pyridine, and toluene were distilled prior to use according to the standard procedures. Dichloroethane and benzene were dried by activated 4 angstrom molecular sieves before use. All other reagents and solvents were used as purchased from Aldrich or Caledon.

Experimental Section

Compound 6

Compound **6** was prepared in two steps. Monoamide cyclopropane ester was prepared according to the procedure of *Synthetic Communications* **1994**, 24, 2477-2483. 10.2g (600mmol) of NH_3 was dissolved in 80mL of methanol by bubbling NH_3 into the methanol for about 15 minutes. Then 12.64g (80mmol) of **5** was added to the above solution, and the mixture stirred for 18 hours. The reaction mixture was concentrated on a rotary evaporator, 60mL of Et_2O was added and the mixture stirred for 10 minutes. The precipitated solid was collected on a Buchner funnel, and was washed with 100mL of Et_2O . The filter cake was added to 100mL of CH_2Cl_2 , and after 10 minutes of stirring, the remaining solid was removed by filtration. The mother liquor was concentrated on a rotary evaporator to yield 9.9g (69.2mmol) of monoamide cyclopropane intermediate (86%). Fine, white crystalline solid, m.p. 158-160°C; ^1H NMR (400 MHz, CDCl_3): δ = 8.57 (s, broad, 1H), 5.62 (s, broad, 1H), 3.69 (s, 3H), 1.73-1.72 (m, 2H), 1.60-1.59 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 173.4, 171.3, 52.2, 25.9, 20.2; IR (thin film): ν_{max} (cm^{-1}) = 3420, 3187, 1711, 1671, 1581, 1443, 1409, 1326, 1193, 1149, 1106, 971; HRMS calc'd for $\text{C}_6\text{H}_9\text{NO}_3$ = 143.0582, found 143.0579.

A solution of diisobutylaluminum hydride (39.4mL, 1.5M in toluene) was added dropwise over 1.5 hours to 3.4g (23.6mmol) of monoamide cyclopropane ester intermediate in dry CH₂Cl₂ (70mL) at -78°C. After stirring for an additional 5.5 hours, 15mL of methanol was added at -78°C dropwise to destroy the excess DIBAL. 30 minutes later, 40mL of 15% HCl was added to the vigorously stirred mixture and the solution was warmed to room temperature gradually. The pH was adjusted to 3.5 and the mixture was stirred until the two layers separated. The solution was extracted with CH₂Cl₂ and the combined organic extracts were dried with MgSO₄. The solvent was removed under reduced pressure to yield 1.67 g (14.8mmol) of crude **6** (63%), which was used directly in the next step without further purification. White solid; ¹H NMR (600 MHz, CDCl₃): δ= 8.56 (s, 1H), 8.27 (s, broad, 1H), 5.73 (s, broad, 1H), 1.98-1.96 (m, 2H), 1.50-1.48 (m, 2H).

Compound 1

2.632 g (14.45mmol) of trimethyl phosphonoacetate and 0.613 g (14.45mmol) of lithium chloride in 25mL of THF were cooled to 0°C, and then 2.16mL (14.45mol) of DBU was added. The mixture was stirred for 15 minutes at 0°C. Then 1.42g (12.6mmol) of **6** was added to the above mixture and the reaction mixture was slowly warmed to room temperature. After one hour, H₂O was added to quench the reaction and the product was extracted using CH₂Cl₂. The extracts were washed with H₂O and brine, dried over MgSO₄ and the solvent was removed under reduced pressure. The crude mixture was purified by column chromatography using EtOAc/Hexanes as the eluent yielding 1.25g (7.39mmol) of Horner-Emmons olefination intermediate (59%): White crystalline solid, m.p. 139-140°C; R_f = 0.17 (EtOAc : hexanes = 2: 1); ¹H NMR (600 MHz, CDCl₃): δ = 7.26 (d, J = 15.6 Hz, 1H), 5.90 (d, J = 15.6 Hz, 1H), 5.69 (s, broad, 2H), 3.75 (s, 3H), 1.58-1.56 (m, 2H), 1.08-1.06 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ =173.5, 166.6, 146.5, 121.2, 51.8, 26.4, 18.1; IR (thin film): ν_{max} (cm⁻¹) = 3431, 3150, 1707, 1680, 1642, 1621, 1414, 1327, 1214, 1175, 1006; HRMS calc'd for C₈H₁₁NO₃ = 169.0739, found 169.0736 .

To 140mL of dry THF was added 1.217g (7.2mmol) of Horner-Emmons olefination intermediate. The reaction mixture was heated to 70°C and 1.697g (15.12mmol) of 95% potassium tert-butoxide was added. After 20 minutes the mixture was quenched with 5% HCl solution, and adjusted to acidic (pH about 3). The solution was extracted with CH₂Cl₂, and the combined organic extracts were washed with H₂O, and brine, dried over MgSO₄ and the solvent removed under reduced pressure to yield 0.73g (5.33mmol) of **1** (74%). In most cases the crude product is of sufficient purity to be used in the next step. If necessary, the crude product can be purified by column chromatography using EtOAc/Hexanes as eluent. White crystalline solid, m.p. 161.5-163.5°C; R_f = 0.31 (EtOAc : hexanes =

2: **1**); ^1H NMR (400 MHz, CDCl_3): δ = 8.53 (b, 1H), 6.31 (d, J = 10.0Hz, 1H), 6.19 (d, J = 10.0Hz, 1H), 2.00-1.97 (m, 2H), 1.51-1.48 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 172.8, 166.1, 150.4, 120.4, 27.0, 23.3; IR (thin film): ν_{max} (cm^{-1}) = 3164, 3066, 2844, 1683, 1616, 1421, 1389, 1316, 1256, 1222, 973, 870, 842, 811; HRMS calc'd for $\text{C}_7\text{H}_7\text{NO}_2$ = 137.0477, found 137.0481.

Compound 11

To a solution of 0.399 g (2.91mmol) of **1** in dry CH_2Cl_2 was added 0.47mL (0.461g, 5.83mmol) of dry pyridine at 0°C . After 5 minutes, 1.96mL (3.29g, 11.65mmol) of triflic anhydride was added dropwise to the above solution, after which the white pyridinium salt precipitate was observed. The resulting solution was stirred for 1.5 hours and the solution was warmed to room temperature. The white precipitate was removed by filtration, and the resulting solution was concentrated under reduced pressure. The residue was purified by flash column chromatography using EtOAc/Hexanes as eluent yielding 1.2 g (2.18mmol) of **11** (75%). Light brown oil; R_f = 0.51 (EtOAc : hexanes = 2: 3); ^1H NMR (400 MHz, CDCl_3): δ = 8.01 (d, J = 8.0Hz, 1H), 7.32 (d, J = 8.0Hz, 1H), 4.76 (t, J = 6.0Hz, 2H), 3.28 (t, J = 6.0Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 152.2, 151.8, 146.3, 123.3, 118.5 (quartet, J = 318.9 Hz), 116.2, 73.6, 29.4; IR (thin film): ν_{max} (cm^{-1}) = 3112, 2984, 2926, 1743, 1605, 1592, 1428, 1214, 1138, 1106, 965, 917, 875, 844, 817; HRMS calc'd for $\text{C}_{10}\text{H}_6\text{F}_9\text{NO}_3\text{S}_3$ = 550.9061, found 550.9071.

Compound 12

If the procedure for compound **11** was performed without the addition of pyridine, substantial quantities of **12** resulted. Similar to the above procedure, 0.11g (0.8mmol) of **1** and 0.45g (0.27mL, 1.6mmol) of triflic anhydride were used to yield 0.08g (0.30mmol) of **12** (37%) (and 0.13g of **11** (30%)). Colorless oil; R_f = 0.34 (EtOAc: Hexanes= 1:1); ^1H NMR (600 MHz, CDCl_3): δ = 7.60 (d, J = 7.8Hz, 1H), 6.67 (d, J = 7.8Hz, 1H), 4.74 (t, J = 8.4Hz, 2H), 3.30 (t, J = 8.4Hz, 2H); ^{13}C NMR (150 MHz, CDCl_3): δ = 167.7, 154.0, 136.6, 120.9, 118.5 (quartet, J = 319.4Hz), 106.5, 70.7, 27.2; IR (thin film): ν_{max} (cm^{-1}) = 3106, 2989, 2933, 1618, 1594, 1429, 1246, 1213, 1138, 1000, 962, 867, 818, 609; HRMS calc'd for $\text{C}_8\text{H}_6\text{F}_3\text{NO}_4\text{S}$ = 268.9970, found 268.9966.

General procedure for preparation of compounds 13a-f

To a solution of **11** in dry CH₂Cl₂ was added the corresponding primary amine. Several hours later, the white aminium salt precipitate was observed. Once the reaction is complete by TLC, the aminium salt was filtered out and the resulting solution was concentrated under reduced pressure. The residue was purified by flash column chromatography using EtOAc/Hexanes as the eluent to give the corresponding azaindoline.

Compound 13a

13a was prepared according to the general procedure using 0.595g (1.08mmol) of **11** and 0.358g (3.35mmol) of benzylamine in 15mL of CH₂Cl₂. The reaction mixture was stirred for one day at room temperature. The crude product was purified by column chromatography yielding 0.34g (0.95mmol) of **13a** (88%). Pale brown solid, m.p. 44 – 45.5°C; R_f = 0.57 (EtOAc: hexanes = 1: 2); ¹H NMR (600 MHz, CDCl₃): δ = 7.34-7.29 (m, 5H), 7.20 (d, J = 7.2Hz, 1H), 6.21 (d, J = 7.8Hz, 1H), 4.51(s, 2H), 3.51 (t, 9.0Hz, 2H), 2.97 (t, J = 9.0Hz, 2H); ¹³C NMR (150 MHz, CDCl₃): δ = 162.4, 155.4, 137.0, 133.3, 128.6, 128.4, 127.5, 122.9, 118.7 (quartet, J= 318.8Hz), 100.3, 49.0, 48.8, 25.0; IR (thin film): ν_{max} (cm⁻¹) = 3064, 2922, 2855, 1628, 1577, 1521, 1421, 1385, 1211, 1138, 937, 869; HRMS calc'd for C₁₅H₁₃F₃N₂O₃S = 358.0599, found 358.0592.

Compound 13b

13b was prepared according to the general procedure using 0.414g (0.75mmol) of **11** and 0.170g (2.33mmol) of tert-butylamine in 15mL of CH₂Cl₂. The reaction mixture was stirred for three days at room temperature. The crude product was purified by column chromatography yielding 0.185g (0.57mmol) of **13b** (76%). Pale brown solid, m.p. 50 – 50.5°C; R_f = 0.51 (EtOAc : hexanes = 1:3); ¹H NMR (400 MHz, CDCl₃): δ = 7.14 (d, J = 7.6Hz, 1H), 6.15 (d, J = 7.6Hz, 1H), 3.62 (t, J = 8.8Hz, 2H), 2.87 (t, J = 8.8Hz, 2H), 1.41 (s, 9H); ¹³C NMR (100 MHz, CDCl₃): δ = 162.5, 154.6, 132.4, 124.8, 118.7 (quartet, J = 318.2Hz), 99.7, 54.3, 47.6, 26.7, 24.8; IR (thin film): ν_{max} (cm⁻¹) = 2976, 2924, 2859, 1630, 1570, 1422, 1273, 1249, 1212, 1140, 1114, 1077, 921, 872; HRMS calc'd for C₁₂H₁₅F₃N₂O₃S = 324.0755, found 324.0722.

Compound 13c

13c was prepared according to the general procedure using 0.77g (1.40mmol) of **11** and 0.256g (4.33mmol) of isopropylamine in 10mL of CH₂Cl₂. The reaction mixture was stirred for one day at room temperature. The crude product was purified by column chromatography yielding 0.36g (1.16mmol) of **13c** (83%). Pale brown oil; R_f = 0.37 (EtOAc: hexanes = 1:4); ¹H NMR (400 MHz, CDCl₃): δ = 7.14 (d, J = 7.6Hz, 1H), 6.12 (d, J = 7.6Hz, 1H), 4.22 (septet, J = 7.2Hz, 1H), 3.57 (t, J = 8.8Hz, 2H), 2.95 (t, J = 8.8Hz, 2H), 1.17 (d, J = 7.2Hz, 6H); ¹³C NMR (150 MHz, CDCl₃): δ = 162.0, 155.5, 132.6, 123.5, 118.7 (quartet, J = 318.6Hz), 99.5, 44.3, 43.8, 24.8, 18.8; IR (thin film): ν_{max} (cm⁻¹) = 2975, 2937, 2878, 1630, 1575, 1512, 1473, 1419, 1279, 1250, 1208, 1139, 1037, 929, 872; HRMS calc'd for C₁₁H₁₃F₃N₂O₃S = 310.0599, found 310.0602.

Compound 13d

13d was prepared according to the general procedure using 0.24g (0.436mmol) of **11** and 0.173g (1.742mmol) of cyclohexylamine in 8mL of CH₂Cl₂. The reaction mixture was stirred for one day at room temperature. The crude product was purified by column chromatography yielding 0.13g (0.371mmol) of **13d** (85%). Pale brown oil; R_f = 0.54 (EtOAc: hexanes = 1:3); ¹H NMR (600 MHz, CDCl₃): δ = 7.12 (d, J = 7.2Hz, 1H), 6.11 (d, J = 7.2Hz, 1H), 3.79-3.75 (m, 1H), 3.60 (t, J = 8.4Hz, 2H), 2.95 (t, J = 8.4Hz, 2H), 1.83-1.78 (m, 4H), 1.69 (m, 1H), 1.43-1.34 (m, 4H), 1.14-1.11 (m, 1H); ¹³C NMR (150 MHz, CDCl₃): δ = 162.0, 155.5, 132.5, 123.4, 118.8 (quartet, J = 319.2Hz), 99.3, 52.5, 44.9, 29.4, 25.7, 25.6, 25.0; IR (thin film): ν_{max} (cm⁻¹) = 2934, 2858, 1630, 1574, 1512, 1422, 1209, 1136, 932, 870; HRMS calc'd for C₁₄H₁₇F₃N₂O₃S = 350.0912, found 350.0904.

Compound 13e

13e was prepared according to the general procedure using 0.276g (0.50mmol) of **11** and 0.244g (1.55mmol) of n-decylamine in 10mL of CH₂Cl₂. The reaction mixture was stirred for one day at room temperature. The crude product was purified by column chromatography yielding 0.168g (0.41mmol) of **13e** (82%). Pale yellow oil; R_f = 0.4 (EtOAc: hexanes = 1:5); ¹H NMR (600 MHz, CDCl₃): δ = 7.15 (d, J = 7.2Hz, 1H), 6.13 (d, J = 7.2Hz, 1H), 3.59 (t, J = 8.4Hz, 2H), 3.29 (t, J = 7.8Hz, 2H), 2.98 (t, J = 8.4Hz, 2H), 1.57-1.56 (m, 2H), 1.32-1.26 (m, 14H), 0.88 (t, J = 7.2Hz, 3H); ¹³C NMR (150 MHz, CDCl₃): δ = 162.8, 155.6, 132.7, 123.0, 118.7 (quartet, J = 318.6Hz), 99.6, 49.6, 44.9, 31.9, 29.54, 29.52, 29.31, 29.28, 27.0, 26.9, 25.1, 22.6, 14.1; IR (thin film): ν_{max} (cm⁻¹) = 2958, 2928, 2856, 1635, 1576, 1521, 1419, 1208, 1138, 1117, 932, 869; HRMS calc'd for C₁₈H₂₇F₃N₂O₃S = 408.1694, found 408.1703.

Compound 13f

13f was prepared according to the general procedure using 0.328g (0.60mmol) of **11** and 0.224g (1.85mmol) of (±) α-methylbenzylamine in 6mL of (CH₂Cl)₂. The reaction mixture was stirred for 2 hours at 90°C. The crude product was purified by column yielding 0.186g (0.50mmol) of **13f** (84%). Pale brown oil; R_f = 0.53 (EtOAc : hexanes = 1:3); ¹H NMR (400 MHz, CDCl₃): δ = 7.38-7.25 (m, 5H), 7.15 (d, J = 7.2Hz, 1H), 6.16 (d, J = 7.2Hz, 1H), 5.42 (quartet, J = 7.2Hz, 1H), 3.60 (td, J = 9.6, 7.2Hz, 1H), 3.26 (td, J = 9.6, 7.2Hz, 1H), 2.95- 2.87 (m, 2H), 1.57 (d, J = 6.8Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 161.9, 155.3, 140.4, 133.1, 128.4, 127.4, 127.3, 123.2, 118.7 (quartet, J = 318.6Hz), 100.0, 50.9, 44.2, 24.8, 15.8; IR (thin film): ν_{max} (cm⁻¹) = 3033, 2978, 2937, 2878, 1629, 1574, 1510, 1471, 1423, 1277, 1211, 1138, 920, 868; HRMS calc'd for C₁₆H₁₅F₃N₂O₃S = 372.0756, found 372.0717.

General procedure for preparation of Compound 14a-f

This procedure referred to *Tetrahedron Letters* **2004**, 45, 7783-7786. To a solution of azaindoline in dry benzene was added an excess of activated MnO₂ and activated 4 angstrom molecular sieves at room temperature. The reaction mixture was gradually heated to 70-80°C and stirred for 1 hour. The solid was removed by filtration through a Celite plug and the filter cake washed with CH₂Cl₂. The solvent was removed under reduced pressure to yield the corresponding compounds of very high purity without column chromatography.

Compound 14a

14a was prepared according to the general procedure using 1.06g (2.96mmol) of **13a**, 2.32g (26.65mmol) of activated MnO₂ and 12g of molecular sieves in 30mL of dry benzene. After filtration, 0.843g (2.37mmol) of **14a** was obtained (80%). Pale orange solid, m.p. 40.5 - 41°C; R_f = 0.43 (EtOAc : hexanes = 1:3); ¹H NMR (600 MHz, CDCl₃): δ = 8.03 (d, J = 8.4Hz, 1H), 7.32 - 7.26 (m, 6 H), 6.95 (d, J = 7.8Hz, 1H), 6.54 (d, J = 3.6Hz, 1H), 5.40 (s, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 151.1, 144.5, 136.8, 132.7, 129.5, 128.8, 128.0, 127.9, 120.4, 118.8 (quartet, J = 318.6Hz), 106.6, 100.8, 48.3; IR (thin film): ν_{max} (cm⁻¹) = 3105, 3068, 3034, 2934, 1604, 1509, 1423, 1327, 1214, 1138, 952, 880, 820; HRMS calc'd for C₁₅H₁₁F₃N₂O₃S = 356.0442, found 356.0440.

Compound 14b

14b was prepared according to the general procedure using 0.103g (0.32mmol) of **13b**, 0.249g (2.86mmol) of activated MnO₂ and 1.3g of molecular sieves in 3mL of dry benzene. After filtration, 0.081g (0.25mmol) of **14b** was obtained (79%). Yellow oil; R_f = 0.51 (EtOAc: hexanes = 1:3); ¹H NMR (600 MHz, CDCl₃): δ = 7.99 (d, J = 7.8Hz, 1H), 7.44 (d, J = 3.6Hz, 1H), 6.90 (d, J = 8.4Hz, 1H), 6.47 (d, J = 3.6Hz, 1H), 1.78 (s, 9H); ¹³C NMR (150 MHz, CDCl₃): δ = 149.5, 144.1, 132.2, 127.5, 122.0, 118.8 (quartet, J = 318.6Hz), 105.8, 99.0, 57.1, 29.0; IR (thin film): ν_{max} (cm⁻¹) = 2936, 1567, 1502, 1422, 1322, 1218, 1138, 944, 880; HRMS calc'd for C₁₂H₁₃F₃N₂O₃S = 322.0599, found 322.0608.

Compound 14c

14c was prepared according to the general procedure using 0.234g (0.755mmol) of **13c**, 0.59g (6.79mmol) of activated MnO₂ and 3.02g of molecular sieves in 15mL of dry benzene. After filtration, 0.207g (0.67mmol) of **14c** was obtained (89%). Pale brown oil; R_f = 0.51 (EtOAc : hexanes = 1:2); ¹H NMR (400 MHz, CDCl₃): δ = 8.00 (d, J = 8.4Hz, 1H), 7.38 (d, J = 3.6Hz, 1H), 6.91 (d, J = 8.0Hz, 1H),

6.53 (d, $J = 3.6\text{Hz}$, 1H), 5.01 (septet, $J = 6.8\text{Hz}$, 1H), 1.53 (d, 6.8Hz , 6H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 150.6, 143.8, 132.4, 126.3, 120.7, 118.8$ (quartet, $J = 319.0\text{ Hz}$), 106.2, 100.1, 46.5, 22.3; IR (thin film): $\nu_{\text{max}} (\text{cm}^{-1}) = 2981, 2939, 1604, 1505, 1431, 1419, 1321, 1210, 1136, 950, 874, 818$; HRMS calc'd for $\text{C}_{11}\text{H}_{11}\text{F}_3\text{N}_2\text{O}_3\text{S} = 308.0442$, found 308.0449.

Compound 14d

14d was prepared according to the general procedure using 0.126g (0.36mmol) of **13d**, 0.281g (3.23mmol) of activated MnO_2 and 1.44g of molecular sieves in 5mL of dry benzene. After filtration, 0.106g (0.30mmol) of **14d** was obtained (85%). Pale orange oil; $R_f = 0.66$ (EtOAc: hexanes = 1:3); ^1H NMR (600 MHz, CDCl_3): $\delta = 7.99$ (d, $J = 8.4\text{Hz}$, 1H), 7.38 (d, $J = 3.6\text{Hz}$, 1H), 6.91 (d, $J = 8.4\text{Hz}$, 1H), 6.52 (d, $J = 3.6\text{Hz}$, 1H), 4.60-4.56 (m, 1H), 2.11-2.09 (m, 2H), 1.94-1.92 (m, 2H), 1.81- 1.71 (m, 3H), 1.57-1.50 (m, 2H), 1.32-1.26 (m, 1H); ^{13}C NMR (150 MHz, CDCl_3): $\delta = 150.6, 143.9, 132.3, 126.8, 120.7, 118.9$ (quartet, $J = 319.2\text{Hz}$), 106.2, 99.9, 54.2, 33.1, 25.7, 25.5; IR (thin film): $\nu_{\text{max}} (\text{cm}^{-1}) = 2937, 2860, 1604, 1571, 1504, 1424, 1316, 1209, 1160, 1138, 951, 880, 822$; HRMS calc'd for $\text{C}_{14}\text{H}_{15}\text{F}_3\text{N}_2\text{O}_3\text{S} = 348.0755$, found 348.0758.

Compound 14e

14e was prepared according to the general procedure using 0.164g (0.40mmol) of **13e**, 0.315g (3.62mmol) of activated MnO_2 and 2g of molecular sieves in 10mL of dry benzene. After filtration, 0.133g (0.33mmol) of **14e** was obtained (82%). Yellow oil; $R_f = 0.41$ (EtOAc : hexanes = 1:3); ^1H NMR (400 MHz, CDCl_3): $\delta = 8.00$ (d, $J = 8.4\text{Hz}$, 1H), 7.28 (d, $J = 3.2\text{Hz}$, 1H), 6.91 (d, $J = 8.0\text{Hz}$, 1H), 6.52 (d, $J = 3.6\text{Hz}$, 1H), 4.21 (t, $J = 7.2\text{Hz}$, 2H), 1.86-1.82 (m, 2H), 1.30-1.24 (m, 14H), 0.87 (t, $J = 7.2\text{Hz}$, 3H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 150.9, 144.4, 132.4, 129.7, 120.4, 118.8$ (quartet, $J = 319.3\text{Hz}$), 106.2, 100.0, 44.9, 31.8, 30.0, 29.45, 29.41, 29.2, 29.1, 26.7, 22.6, 14.0; IR (thin film): $\nu_{\text{max}} (\text{cm}^{-1}) = 2929, 2857, 1690, 1604, 1510, 1425, 1329, 1212, 1138, 952, 880, 819$; HRMS calc'd for $\text{C}_{18}\text{H}_{25}\text{F}_3\text{N}_2\text{O}_3\text{S} = 406.1538$, found 406.1544.

Compound 14f

14f was prepared according to the general procedure using 0.153g (0.41mmol) of **13f**, 0.325g (3.74mmol) of activated MnO₂ and 1.66g of molecular sieves in 10mL of dry benzene. After filtration, 0.138g (0.37mmol) of **14f** was obtained (91%). Pale brown oil; R_f = 0.32 (EtOAc: hexanes = 1:5); ¹H NMR (400 MHz, CDCl₃): δ = 8.00 (d, J = 8.4Hz, 1H), 7.34-7.26 (m, 6H), 6.93 (d, J = 8.0Hz, 1H), 6.53 (d, J = 3.6Hz, 1H), 6.10 (quartet, J = 7.6Hz, 1H), 1.91 (d, J = 6.8Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 150.8, 144.2, 141.2, 132.5, 128.6, 127.8, 127.0, 126.5, 120.6, 118.8 (quartet, J = 319.0Hz), 106.6, 100.6, 53.0, 20.3; IR (thin film): ν_{max} (cm⁻¹) = 3034, 2983, 2936, 1603, 1502, 1424, 1318, 1212, 1137, 942, 879, 819; HRMS calc'd for C₁₆H₁₃F₃N₂O₃S = 370.0599, found 370.0588.

General procedure for preparation of compound 19 and 20

In a round bottom flask, **14a** was dissolved in dry DMF, followed by addition of LiCl, Pd(PPh₃)₂Cl₂, CuI, and excess triethylamine. The mixture was stirred at room temperature for several minutes and then the alkyne was added. The reaction mixture was purged with argon and heated for several hours. The reaction mixture was then cooled to room temperature, poured into water and extracted with ether. The combined organic layers were washed with water and brine, and dried over MgSO₄. The solvent was removed under reduced pressure and crude products were purified by column chromatography using EtOAc/Hexanes as the eluent.

Compound 19

Compound **19** was prepared according to the general procedure using 0.159g (0.447mmol) of **14a** in 5mL of dry DMF, 0.057g (1.34mmol) of LiCl, 0.025g (0.036mmol) of Pd(PPh₃)₂Cl₂, 0.004g (0.022mmol) of CuI and 4.5mL of Et₃N. Several minutes later, 0.055g (0.06mL, 0.536mmol) of phenyl acetylene was added and the reaction mixture stirred at 65°C for 1.5 hours. After the column chromatography purification, 0.12g (0.389mmol) of **19**(87%) was obtained. Orange solid, m.p. 118-120°C; R_f = 0.34 (EtOAc: hexanes = 1:7); ¹H NMR (400 MHz, CDCl₃): δ = 7.90 (d, J = 8.0Hz, 1H), 7.62-7.64 (m, 2H), 7.37 - 7.22 (m, 9H), 7.20 (d, J = 3.6Hz, 1H), 6.49 (d, J = 3.6Hz, 1H), 5.55 (s, 2H);

^{13}C NMR (100 MHz, CDCl_3): δ = 147.4, 137.5, 135.8, 131.8, 129.2, 128.6, 128.5, 128.4, 128.2, 127.6, 127.5, 122.8, 120.1, 120.0, 100.4, 90.2, 87.8, 47.5; IR (thin film): ν_{max} (cm^{-1}) = 3062, 3032, 2925, 2855, 2204, 1589, 1504, 1425, 1387, 1284, 1212, 823, 756, 725, 693; RMS calc'd for $\text{C}_{22}\text{H}_{16}\text{N}_2$ = 308.1313, found 308.1303.

Compound 20

Compound **20** was prepared according to the general procedure using 0.194g (0.544mmol) of **14a** in 6mL of dry DMF, 0.069g (1.63mmol) of LiCl, 0.03g (0.043mmol) of $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$, 0.01g (0.054mmol) of CuI and 5.5mL of Et_3N . Several minutes later, 0.374g (0.53mL, 3.81mmol) of trimethylsilyl acetylene was added and the reaction mixture stirred at 75°C for 2 hours. After column chromatography, 0.143g (0.47mmol) of **20** (86%) was obtained. Light brown solid, m.p. $121.5\text{--}122^\circ\text{C}$; R_f = 0.4 (EtOAc : hexanes = 1:5); ^1H NMR (600 MHz, CDCl_3): δ = 7.84 (d, J = 7.8Hz, 1H), 7.32–7.27 (m, 5H), 7.19 (d, J = 6.6Hz, 1H), 7.17 (d, J = 3.6Hz, 1H), 6.46 (d, J = 3.6Hz, 1H), 5.52 (s, 2H), 0.29 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3): δ = 147.4, 137.6, 135.5, 129.3, 128.6, 128.5, 127.7, 127.6, 120.5, 120.2, 105.4, 100.4, 92.9, 47.6, -0.1; IR (thin film): ν_{max} (cm^{-1}) = 3065, 3033, 2960, 2156, 1589, 1506, 1425, 1389, 1327, 1284, 1249, 954, 861, 842, 716; HRMS calc'd for $\text{C}_{19}\text{H}_{20}\text{N}_2\text{Si}$ = 304.1396, found 304.1401.

General procedure for preparation of compound 21 and 22

In a round bottom flask, **14a** was dissolved in dry DMF, followed by addition of excess organotin, LiCl, and $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$. The mixture was purged with argon and heated for several hours. The reaction mixture was then cooled to room temperature, poured into water and extracted with ether. The combined organic layers were washed with H_2O and brine, and dried over MgSO_4 . The solvent was removed under reduced pressure and crude products were purified by column chromatography using EtOAc/Hexanes as the eluent.

Compound 21

Compound **21** was prepared according to the general procedure using 0.195g (0.546mmol) of **14a** in 6mL of DMF, 0.116g (2.73mmol) of LiCl, 0.038g (0.055mmol) of Pd(PPh₃)₂Cl₂, and 0.208g (0.19mL, 0.656mmol) of tributylvinyl tin. The mixture was heated at 80°C for one hour. The byproduct tributyltin chloride was removed using the method reported by J.E. Leibner and John Jacobus (*J. Org. Chem.* **1979**, 44,449-450). The excess KF (0.75g in 7mL of H₂O) aqueous solution was added to the reaction mixture and the mixture was stirred at room temperature for one hour. The yellow tributyltin fluoride precipitate was removed by filtration. After column chromatography, 0.09g (0.384mmol) of **21** (70%) was obtained. Yellow oil; R_f = 0.43 (EtOAc: hexanes = 1:5); ¹H NMR (400 MHz, CDCl₃): δ = 7.85 (d, J = 8.0Hz, 1H), 7.30 - 7.27 (m, 5H), 7.18 (d, 8.0Hz, 1H), 7.15 (d, J = 3.6Hz, 1H), 6.92(dd, J = 17.2, 10.8Hz, 1H), 6.43 (d, J = 3.6Hz, 1H), 6.25 (dd, J = 17.6, 1.6Hz, 1H), 5.51 (s, 2H), 5.40 (dd, J = 10.8, 1.6Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 149.4, 147.6, 137.9, 137.8, 128.7, 128.6, 128.2, 127.7, 127.5, 119.7, 116.0, 114.3, 100.1, 47.5; IR (thin film): ν_{max} (cm⁻¹) = 3090, 3063, 3031, 2925, 1590, 1565, 1507, 1427, 1388, 1319, 1303, 826, 706; HRMS calc'd for C₁₆H₁₄N₂ = 234.1157, found 234.1149.

Compound **22**

Compound **22** was prepared according to the general procedure using 0.205g (0.576mmol) of **14a** in 5mL of DMF, 0.122g (2.88mmol) of LiCl, 0.02g (0.029mmol) of Pd(PPh₃)₂Cl₂, and 0.206g (0.19mL, 1.152mmol) of tetramethyl tin. The mixture was purged with argon and heated at 140°C for one hour. After column chromatography, 0.104g (0.468mmol) of **22** (81%) was obtained. Pale yellow oil, R_f = 0.43 (EtOAc : hexanes = 1:4); ¹H NMR (600 MHz, CDCl₃): δ = 7.79 (d, J = 7.8Hz, 1H), 7.31-7.26 (m, 3H), 7.21 (d, J = 7.8Hz, 2H), 7.05 (d, J = 3.0Hz, 1H), 6.95 (d, J = 7.8Hz, 1H), 6.41 (d, J = 3.6Hz, 1H), 5.49 (s, 2H), 2.65 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 151.8, 147.6, 138.0, 128.8, 128.5, 127.5, 127.3, 126.6, 117.7, 115.8, 99.8, 47.3, 24.5; IR (thin film): ν_{max} (cm⁻¹) = 3064, 3033, 2923, 2856, 1596, 1574, 1511, 1438, 1388, 1292, 814, 708; HRMS calc'd for C₁₅H₁₄N₂ = 222.1157, found 222.1151.

General procedure for preparation the compound **23** and **24**

To a solution of **14a** in dry DMF was added alkene, triethylamine and Pd(PPh₃)₂Cl₂. The mixture was purged with argon and heated. Several hours later, the mixture was cooled to room temperature, poured into water and extracted with ether. The combined organic layers were washed with H₂O and

brine, and dried over MgSO_4 . The solvent was removed under reduced pressure, and the crude products were purified by column chromatography using EtOAc/Hexanes as the eluent.

Compound 23

Compound **23** was prepared according to the general procedure using 0.12g (0.337mmol) of **14a**, 0.145g (0.15mL, 1.685mmol) of methyl acrylate, 0.102g (0.14mL, 1.011mmol) of triethylamine and 0.036g (0.051mmol) of $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ in 4mL of dry DMF. The mixture was stirred between 130-140°C for 3 hours. After column chromatography, 0.077g (0.264mmol) of **23** (78%) was obtained. Pale yellow solid, m.p. 113.5-114.5°C; R_f = 0.43 (EtOAc: hexanes = 1:3); ^1H NMR (400 MHz, CDCl_3): δ = 7.90 (d, J = 8.0Hz, 1H), 7.83 (d, J = 15.6Hz, 1H), 7.32- 7.24 (m, 7H), 7.01 (d, J = 15.6Hz, 1H), 6.47 (d, J = 3.6Hz, 1H), 5.51 (s, 2H), 3.83 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3): δ = 167.6, 147.8, 146.0, 145.1, 137.6, 130.0, 128.7, 128.6, 127.7, 127.6, 121.5, 119.3, 117.4, 100.3, 51.6, 47.7; IR (thin film): ν_{max} (cm^{-1}) = 3065, 3031, 2948, 1715, 1641, 1505, 1433, 1339, 1298, 1271, 1208, 1193, 1162; HRMS calc'd for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_2$ = 292.1212, found 292.1217.

Compound 24

Compound **24** was prepared according to the general procedure using 0.197g (0.552mmol) of **14a**, 0.387g (0.45mL, 5.52mmol) of methyl vinyl ketone, 0.168g (0.23mL, 1.66mmol) of triethylamine and 0.039g (0.055mmol) of $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ in 6mL of dry DMF. The mixture was stirred at 140°C for 2 hours. After column chromatography, 0.142g (0.514mmol) of **24** (93%) was obtained. Yellow solid, m.p. 77-79°C; R_f = 0.26 (EtOAc : hexanes = 1:3); ^1H NMR (400 MHz, CDCl_3): δ = 7.91 (d, J = 8.0Hz, 1H), 7.65 (d, J = 16.0Hz, 1H), 7.32-7.21 (m, 8H), 6.49 (d, J = 3.6Hz, 1H), 5.52 (s, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ = 198.9, 147.8, 146.2, 143.9, 137.5, 130.2, 128.8, 128.7, 128.5, 127.7, 127.6, 121.6, 117.3, 100.4, 47.7, 27.5; IR (thin film): ν_{max} (cm^{-1}) = 3063, 3032, 2925, 1663, 1617, 1504, 1425, 1359, 1316, 1294, 1247, 979, 816, 717; HRMS calc'd for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}$ = 276.1263, found 276.1272.

Compound 25

0.246g (0.691mmol) of **14a** was dissolved in 5mL of dry DMF and 2mL of dry methanol. Several minutes later, 0.0485g (0.069mmol) of $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ was added to the above solution followed by addition of 2mL of triethylamine. The mixture was purged with CO for 10 minutes. The mixture was heated to 110°C under 1atm of CO and stirred for six hours. The reaction mixture was cooled to room temperature, poured into water, and extracted with ether. The combined organic layers were washed with water and brine, and dried over MgSO_4 . The solvent was removed under reduced pressure and purified by column chromatography using EtOAc/Hexanes as eluent yielding 0.132g (0.496mmol) of **25** (72%). Yellow solid, m.p. 70.5-72°C; R_f = 0.43 (EtOAc : hexanes = 1:2); ^1H NMR (600 MHz, CDCl_3): δ = 8.02-7.98 (m, 2H), 7.33-7.25 (m, 6H), 6.54 (d, J = 3.6Hz, 1H), 5.59 (s, 2H), 4.02 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ = 166.6, 147.1, 140.7, 137.1, 131.3, 128.5, 127.7, 127.6, 123.2, 117.4, 100.3, 52.4, 47.7; IR (thin film): ν_{max} (cm^{-1}) = 3088, 3063, 3031, 2948, 2843, 1716, 1504, 1427, 1273, 1208, 1127, 725, 698; HRMS calc'd for $\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_2$ = 266.1055, found 266.1049.

Compound 18

To a solution of 0.177g (0.575mmol) of **14c** in 5mL of DMF was added 0.075g (1.77mmol) of LiCl, 0.066g (0.057mmol) of $\text{Pd}(\text{PPh}_3)_4$, and 0.86mL of 2M Na_2CO_3 solution. The mixture was stirred at room temperature for several minutes. Then 0.14g (1.15mmol) of phenyl boronic acid was added and the mixture was purged with argon, heated to 90°C and stirred for 5 hours. The reaction mixture was cooled to room temperature, poured into water, and extracted with ether. The combined organic layers were washed with water and brine, and dried over MgSO_4 . The solvent was removed by a rotary evaporator and the crude compound was purified by column chromatography using EtOAc/Hexanes as the eluent yielding 0.121g (0.512mmol) of **18** (89%). Pale yellow solid, m.p. 109-110°C; R_f = 0.51 (EtOAc : hexanes = 1:5); ^1H NMR (600MHz, CDCl_3): δ = 8.12 (d, J = 8.4Hz, 2H), 7.94 (d, J = 8.4 Hz, 1H), 7.55 (d, J = 8.4Hz, 1H), 7.47 (t, J = 7.8Hz, 2H), 7.37 (t, J = 7.2Hz, 1H), 7.33 (d, J = 3.6Hz, 1H), 6.47 (d, J = 3.6Hz, 1H), 5.34 (septet, J = 6.6Hz, 1H), 1.57 (d, J = 7.2Hz, 6H); ^{13}C NMR (150MHz, CDCl_3): δ = 150.3, 147.2, 140.7, 128.9, 128.5, 128.0, 126.9, 124.7, 119.5, 113.0, 99.3, 45.3, 22.9; IR (thin film): ν_{max} (cm^{-1}) = 3062, 3019, 2974, 2928, 2869, 1590, 1563, 1499, 1428, 1401, 1291, 1235, 1211, 831, 758, 719, 692; HRMS calc'd for $\text{C}_{16}\text{H}_{16}\text{N}_2$ = 236.1314, found 236.1319.

Compound 16

The preparation of compound **16** followed the procedure of *Helvetica Chimica Acta* **1978**, 61, 104-7. To the solution of 0.733g (4.97mmol) of 2,6-dihydroxypyridin-hydrochloride in 50mL of anhydrous EtOH was added 1.42g (4.97mmol) of 2-azido-3-ethylbenzothiazolium tetrafluoroborate (preparation according to *Ann.* **1961**, 647, 1-10). The mixture was stirred at 50°C for 1 hour, cooled to room temperature, and then put in fridge for overnight. The crystals were collected by filtration and washed with cold EtOH yielding 0.5g (3.65mmol) of **16** (73%). Green crystals, decomp. 199-201°C; ¹H NMR (600 MHz, acetone-d₆): δ = 9.99 (s, broad, 1H), 7.68 (d, J = 10.2Hz, 1H), 5.66 (d, J = 10.2Hz, 1H); IR (thin film): ν_{max} (cm⁻¹) = 3023, 2151, 1660, 1573, 1441, 1370, 1246, 1201, 856, 806; HRMS calc'd for C₅H₃N₃O₂ = 137.0225, found.137.0226.

Compound 17

A solution of 0.11g (0.803mmol) of **16** in 30mL of dry (CH₂Cl)₂ was heated to reflux. Several minutes later, 7.1mg (0.016mmol) of Rh₂(OAc)₄ was added followed by addition of 1.25g (12mmol) of styrene. The reaction was stirred at 90°C under an argon atmosphere for 45 minutes. After concentration, the residue was purified by column chromatography with EtOAc/Hexans yielding 37mg (0.174mmol) of **17** (22%). Major diastereoisomer: yellow solid, m.p. 122-124°C; ¹H NMR (600 MHz, CDCl₃): δ = 8.15 (s, broad, 1H), 7.37-7.25 (m, 5H), 6.09 (d, J = 9.6Hz, 1H), 6.04 (d, J = 9.6Hz, 1H), 3.59 (dd, J = 8.4, 8.4Hz, 1H), 2.41 (dd, J = 9.0, 4.8Hz, 1H), 2.15 (dd, J = 8.4, 4.8Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 172.5, 165.6, 147.8, 134.3, 128.9, 128.7, 128.0, 121.1, 40.9, 33.5, 26.6; IR (thin film): ν_{max} (cm⁻¹) = 3202, 3069, 2850, 1691, 1619, 1426, 1390, 1357, 1311, 1246, 1215, 1186, 986, 837, 816, 701; HRMS calc'd for C₁₃H₁₁NO₂ = 213.0790, found 213.0780.

z-2-137c 1400

expt szpu1

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z-4-169c C13 1400

expt s2pu1

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ct	256	PROCESSING	1.00
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ai	no	ph

z-2-135-1

exp1 PROTON

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2005-07-09/PROTON-~		atfa	0
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bs	32	fn	not used
d1	1.008	display	
nt	0	sp	15.7
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compound 6

Z-4-85-1 1680
expt1 PROTON

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ct	TRANSMITTER	8	sp
tn	H1	sp	sp
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dm	nm	th	54
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dnt	17094

z-3-163-1b c13

expt s2pu1

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np	60286	dp	y
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pw	4.050	lp	-87.2
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dmm	w	th	19
dpwr	39	at	
dnt	9174	no	ph

z-3-73c 1400
expt1 s2pu1

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compound 1

z-3-173c1 c13 1400

Archive directory: /export/home/kerr/vnarsys/data
Sample directory: z-3-173c1c13_07Mar2006
File: CARBON

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
INDV4-400 "nmr3c400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.193 sec
Width 25133.5 Hz
S2 repetitions
OBSERVED F1 125.7612 MHz
DECOUPLE "H1", 399.7617612 MHz
Power 39 dB,
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 50 sec

compound 1

Z-4-181-1 1400

expi szpul

SAMPLE		SPECIAL	
date	Feb 6 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	hst	20
ACQUISITION		pw90	11.700
sw	6395.9	alpa	6.600
at	3.744	flags	
np	47892	11	n
fb	4000	16	n
dl	1.000	dp	y
nt	8	hs	nn
ct	8	fn	not used
tn	399.762	sp	-3.2
strq	399.762	wp1	3513.1
tot	399.762	ft1	3867.1
tpwr	5.850	ftp	2802.3
pw	DECOUPLER	tp	-33.7
dn	C13	lp	-17.1
dof	0	plot	
dm	nmn	wc	250
dmm	c	sc	0
dpr	37	vs	25
dpr	17094	th	54
at	cdc	ph

compound 11

z-4-181-1 C13 1400

expt s2pul1

SAMPLE		SPECIAL	
date	Feb 6 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	6.008
sw	25133.5	pw90	8.100
at	1.199	alfa	20.000
np	60288	flags	n
fb	14000	11	n
bs	64	in	y
d1	1.000	dp	nm
nt	256	hs	nm
ct	256	PROCESSING	1.00
tn	100.531	fb	not used
stfq	1545.3	sp	208.6
tdpr	52	wp	1828.9
pw	4.050	f1	8278.4
DECOUPLER	H1	f1p	7740.4
dn	0	lp	-314.5
dot	yvy	plot	250
dam	w	sc	0
dpr	39	vs	1629
dpr	9174	th	13
ai	no	ph

compound 11

z-2-99-2a 1600
 exp1 PROTON
 SAMPLE SPECIAL
 date Mar 14 2006 temp 25.0
 solvent CDCl3 gain not used
 file /export/home/~ nst 22
 kern/vmr-sys/data/~ nst 0.008
 z-2-99-2a-2006-03-~ pw90 10.300
 14/PROTON.01.fid a1fa 0
 ACQUISITION
 sw 3591.2 11 flags n
 at 1.892 1n n
 ap 3634 1p y
 bs 32 32 ns PROCESSING nm
 dt 1.006 1n not used
 nt 8 8 DISPLAY 20.5
 ct TRANSMITTER 8 SP 5361.1
 tn H1 r1 5545.8
 stf4 539.479 r1p 4352.2
 tot 539.3 r1p 75.2
 tpwr 57 1p -36.0
 pw 5.150 wc PLOT 250
 dn DECOUPLER C13 SC 0
 dot 8 SC 10
 dm 8 nm 54
 decwawe at cdc ph
 dpwr 45
 dmf 17094

z-2-99-2 C13 1600
expt CARBON

SAMPLE		SPECIAL	
date	Mar 14 2006	temp	25.0
solvent	CDCl3	ns	not used
file	/export/home/~	ns	0.006
kerf	/nmer/sv/data/~	hs	18.000
z-2-99-2C13	2006-0~	pw90	10.000
3-14/CARBON	01.fid	alpha	
ACQUISITION			
sw	37682.5	f1	n
at	1.300	in	y
np	98010	dp	nm
fb	21000	hs	
bs	64	lb	8.50
dl	1.000	fn	not used
cl	128		
ct	TRANSMITTER	SD	DISPLAY
tn	C13	WD	-336.3
strq	150.755	FT1	27531.1
tof	3127.6	FTP	13874.5
tpwr	56	TP	11686.8
pw	9.000	TP	149.3
DECOUPLER			
dn	H1	WC	250
dof	0	SC	0
dm	yyv	VS	334
decave	w	tn	9
cpw	AA	cdc	ph
dmf	14286

z-4-143-1 1690

expt1 PROTON

```
SAMPLE          SPECIAL
date Jan 24 2006 temp 25.0
solvent CDCl3 gain not used
file /export/home/~ hst 22
kerr/vnmrsvs/data/~ hst 0.006
z-4-143-1-2006-01~ pw90 10.300
24/PROTON_01.fid alpha 0
ACQUISITION
sw 3591.2 f1 11 flags n
at 1.892 f2 1n y
np 38294 dp 16
hb 38294 hs 16
hs 38294 tn 10.7
nt 1.008 f1 10.7
ct 8 sd 10.7
TRANSMITTER H1 f1 4785.3
tn 589.479 f2 2424.4
stfq 589.3 f3 1228.9
tof 589.3 f4 -8.5
tpwr 57 lp -34.3
pw 5.150 PLOT 250
DECOUPLER C13 wc 0
dn 0 sc 0
dof 0 vs 0
nm 0 th 16
dncwave 45 at cdc ph 54
dpr 45
dmf 17094
```

compound 13a

Z-4-143-1 C13 1600

expl CARBON

```
SAMPLE SPECIAL
date Jan 24 2006 temp 25.0
solvent CDC13 gain not used
file /export/home/~ sp1n 16
kerf/vnmr/sv/data/~ hst 0.008
Z-4-143-1C13-2006~ PW90 18.000
01-24/CARBON_01.f1~ a1fa 10.000
d 11 FLAGS
ACQUISITION 11 n
sw 37682.5 1n n
at 1.300 dp n
np 96810 hs y
fb 21000 1b not used
bs 54 1b not used
d1 1.004 fn DISPLAY 1105.8
d2 256 SD 24856.8
ct TRANSMITTER C13 rfi 13868.2
tn 150.755 rfp 11606.8
tof 3127.6 rfp 44.3
tpwr 56 1p PLOT -321.1
pw DECOUPLER 9.000 WC 250
dn H1 SC 0
dof 0 VS 453
dm yyy th ai cdc ph 4
decwawe w al
dpr 44
dmf 14286
```

compound 13a

z-3-189-1 1400

expl s2pu1

SAMPLE		SPECIAL	
date	Nov 4 2005	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	20
kerf	/vnm/sys/date/~	hst	0.008
z-3-189-1	NOV2005	ps90	11.700
5-DB01N	fid	alra	8.600
ACQUISITION		flags	
sw	6395.9	11	n
at	3.744	in	n
md	47892	dp	y
td	4000	hs	nn
bs	16	fn	not used
dl	1.000	fn	not used
nt	8	sp	5.0
ct	8	wd	3685.4
tn	TRANSMITTER	hl	rfl
strq	399.762	rfl	3687.2
tor	399.8	fp	2902.3
tpwr	57	tp	-149.6
pw	5.850	vc	-14.6
dn	DECOUPLER	c13	250
dof	0	vs	0
dm	nmn	th	34
dpm	c	at	14
dpr	37	cdc	ph
daf	17094

compound 13b

z-3-189-1 C13 1400
expi s2pu1

SAMPLE		SPECIAL	
date	Nov 4 2005	temp	not used
solvent	CDCl3	spin	not used
file	\\export/home/~	spin	20
kerf	/vmpsvs/data/~	hsr	0.008
z-3-189-1C13	04Nov~	pw90	8.100
2005/CARBON.fid	atfa	atfa	20.000
ACQUISITION			
sw	25133.5	f1	n
at	1.199	in	n
np	60288	dp	y
fb	14000	hs	nn
bs	64	lb	not used
d1	1.000	fn	1.00
nt	256	fn	not used
ct	128	fn	not used
TRANSMITTER			
tn	C13	sp	-1497.4
strq	100.531	wp	19716.7
tof	1345.3	rf1	9270.4
tpwr	32	rfp	7740.0
pw	4.050	tp	-265.5
DECOUPLER			
dn	H1	wc	250
dof	0	sc	0
dca	yyv	vs	1077
dca	y	th	9
dpwr	39	at	no
dnt	9174	ph

z-5-35-1 H1 I400
 exp1 s2pu1
 SAMPLE
 date Feb 28 2006 temp not used
 solvent CDCl3 bath not used
 file C0013 exp hst 0.008
 ACQUISITION
 sw 6395.9 pw90 11.700
 at 3.744 alfa 6.600
 np 47892 il n
 fb 4000 in n
 bs 16 in y
 d1 1.000 dp hs
 nt 8 hs
 ct 8 hs
 TRANSMITTER H1 fn
 tp 399.762 SP DISPLAY -20.4
 st 399.570 WP 3294.5
 tot 57 rfi 3687.4
 pw 5.850 rfp 2902.3
 DECOUPLER C13 lp -18.3
 dn 0 wc PLOT
 dof 0 SC 250
 dm nm 0
 dmm c SC 52
 dpwr 37 VS 54
 dmf 17094 th
 at cdc ph

z-3-159-1 C13 1600

expl CARBON

SAMPLE		SPECIAL	
date	Oct 7 2005	temp	not used
solvent	CDC13	gain	not used
file	/export/home/~	sp1n	16
kerf/vnmr/sys/data/~	hst	0.008	
z-3-159-1C13-2005-~	PW90	20.900	
10-07/CARBON_01.f1~	alpha	10.000	

ACQUISITION		PROCESSING	
sw	37682.5	in	n
at	13300	dp	y
fp	95010	hs	nm
bs	21000	1b	0.50
d1	64	fn	not used
nt	1.000	sp	-30.9
ct	128	wd	27184.4
tn	C13	rf1	2259.6
stfq	150.759	rfp	0
tof	3127.7	fp	169.4
tpwr	57	lp	-324.9
pw	10.450		

DECOUPLER		PLOT	
dn	H1	sc	250
dof	0	vs	0
dm	yyv	th	267
decwave	w	at	cdc ph
dpwr	36		7
dnt	13158

compound 13c

z-4-53-1a.1600

exp1 PROTON

SAMPLE		SPECIAL	
date	Dec 13 2005	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~hsc	spin	22
kerf	vmr/sys/data/~hsc	hsc	0.005
z-4-53-1a.1600-12	pw50	10.300	
137400000.000	alt0	0	
ACQUISITION		FLAGS	
sw	9591.2	fl	n
at	1.892	in	v
np	36294	dp	mn
fb	5000	hs	
bs	32		
dl	1.000	fn	not used
nt	8	display	
ct	8	sp	12.3
TRANSMITTER		hl	5144.2
in	599.479	rf1	5546.1
strq	599.479	rfp	4352.2
tor	399.3	tp	-120.2
tpwr	5.150	plot	-29.5
pw	DECUPPLER	cl3	250
dn	0	vs	66
dor	0	th	54
dm	nm	at	
decwave	g	cdc	ph
dpr	45		
dmf	17094

compound 13d

z-4-53-1a C13 1600
expt1 CARBON

SAMPLE SPECIAL 25.0
date Dec 13 2005 temp 25.0
vent Dec 13 2005 not used
fil/EXPORT/home/~ not used
kerf/vnmrsvs/data/~ hst 0.008
z-4-53-1aC13 2005-~ pw90 18.000
12-13/CARBON_01.f~ af1a 10.000
d
ACQUISITION f1 n
sw 37682.5 in n
at 1.300 dp n
mp 98010 hs y
fb 21000 PROCESSING nm
bs 64 lb 0.50
dl 1.000 fn not used
ct 256 DISPLAY
tr TRANSMITTER C13 SP 2209.0
tf 150.755 WP 2335.3
tof 3127.6 Ff 1365.3
tdw 56 fd 11605.8
pw 9.000 lp -273.6 PLOT
DECOUPLER WC 250
dn H1 SC 0
dof 0 VS 589
dm yvy th 12
decwave w at cdc ph
dwr 44
dmf 14286

compound 13d

Z-3-177-1 1600

expt1 PROTON

SAMPLE		SPECIAL	
date	Oct 21 2005	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~	spin	22
kerf/vmr/sv/date/~	hst		0.008
Z-3-177-1-2005-10-~	pv90		10.900
21/PROTON_01.fid	alpha		0
ACQUISITION		FLAGS	
sw	9592.3	11	n
at	1.892	in	n
np	36298	dp	y
fb	5080	hs	nm
ds	32		
dl	1.000	fm	not used
nt	0		
ct	0	SP	21.6
TRANSMITTER	H1	wp	5392.8
strq	599.485	rfl	5587.4
tof	599.3	rp	4382.3
lpwr	53	lp	115.5
pw	5.450		-30.7
DECOUPLER	WC	PLDT	250
dn	C13	sc	0
dof	0	vs	61
dm	nmn	th	34
decwave	W40_hcx	ai	cdc
dpwr	39		ph
dwt	6578

z-3-177-1C13 1600

expt CARBON

SAMPLE		SPECIAL	
date	Oct 21 2005	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~ spin	22	
kerf	/vnmr/sv/data/~	0.006	
z-3-177-1C13_20051-	pw90	20.000	
10-21/CARBON_01.f	atfa	10.000	
ACQUISITION		0	11
sw	37682.5	in	n
at	1.300	dp	y
mp	98010	hs	nn
fb	21000	bs	0.50
bs	64	lb	not used
d1	1.000	fn	not used
nt	256	ct	256
ct	256	sp	-2207.0
TRANSMITTER		wd	29363.0
tn	C13	rf1	13864.2
strq	150.759	rfp	11607.2
tof	3127.7	fp	123.3
tpwr	57	lp	-293.3
pw	10.450	PLOT	
DECOUPLER		wd	250
dn	H1	sc	0
dof	0	vs	480
dm	yy	th	14
decwave	yy	at	cdc
dpwr	36	ph	
dmf	13158

compound 13e

Z-4-205-1 1400

expl szpul

SAMPLE		SPECIAL	
date	Feb 17 2006	temp	not used
solvent	CDC13	gain	not used
title	exp	sp1n	20
ACQUISITION		hst	0.006
sw	6395.9	pw90	11.700
at	3.744	aiTa	6.600
np	47892	FLAGS	
fb	4000	11	n
bs	16	in	n
dl	1.008	dp	y
hs	8	hs	nm
ct	TRANSMITTER	fn	not used
tn	H1	SD	-3.8
stfr	399.762	WD	3613.8
tof	393.8	rt1	3687.6
tpwr	57	rtP	2902.3
pw	5.850	rp	-37.9
DECOUPLER		1p	-19.3
dn	0	PL0T	
dof	nmn	WC	250
dm	nmn	SC	0
dma	C	VS	39
dpr	37	th	54
dmt	17094	ai	cdc ph

Z-4-113-1 M400
STANDARD 1H OBSERVE

expt s2pu1

SAMPLE		SPECIAL	
date	Jan 7 2006	temp	not used
solvent	CDCl3	gain	not used
sfreq	400.145	not used	
1h/2h/acq/aut	1h	not used	
nmr1/acq/aut	1h	not used	
nmr1/acq/aut	1h	not used	
07.01.06/0402	0402	11.500	
ACQUISITION	25125.6	4174	20.000
sw	1.199	11	
at	60270	1n	n
mp	13800	dp	y
fb	64	hs	nm
bs	1.000		
d1	512		
nt	512		
ct	512		
TRANSMITTER	C13	SP	not used
sfreq	100.613	WP	16820.1
tof	1550.8	rf1	19824.4
tpwr	57	rfp	7746.3
pw	5.800	tp	-137.6
DECOUPLER	H1	1p	-309.7
dn	0		
dof	0		
dma	yyv	SC	250
dpwr	40	VS	0
dat	9300	AI	193
		no	14
		ph

Z-5-11c H1 1600

expt1 PROTON

SAMPLE		SPECIAL	
date	Mar 2 2006	temp	25.0
solvent	CDCl3	gain	not used
title	/export/home/~	spin	22
kerf	/vmar/sys/data/~	hst	0.006
Z-5-11c	2006-03-~	pw90	10.500
02/NOE	0.000	alt1a	0
02/NOE	0.000	alt1b	0
ACQUISITION	9591.2	11	11
at	1.892	in	n
np	36294	dp	v
fb	5000	hs	nm
bs	32	hs	nm
di	1.000	fn	not used
nt	8	fn	not used
ct	8	sp	10.6
TRANSMITTER	H1	wp	5384.8
tn	599.479	rf1	5547.8
strq	599.479	rfp	4352.2
tof	599.37	tp	-24.7
tpwr	5.150	tp	-24.6
PLOT			
PW	DECoupler	C13	250
dn	0	vs	0
dm	0	th	32
decwave	g	at	35
dpr	45		
def	17094

compound 14a

Z-4-119c M400
STANDARD 1H OBSERVE

expi s2pu1

SAMPLE		SPECIAL	
date	Jan 7 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/	spn	not used
nmr1	07.01.06/0202	hst	not used
nmr2	07.01.06/0202	pw90	11.600
ACQUISITION		alfa	20.000
sw	25125.6	flags	
at	1.199	11	n
np	60270	in	y
fb	13800	dp	nn
bs	64	hs	
d1	1.000	1b	not used
nt	512	fn	1.00
ct	256	tn	not used
TRANSMITTER		display	
tn	c13	sp	-2.9
strq	100.613	wp	1611.9
tof	1530.16	ft1	9267.3
tpwr	57	ftp	7.67.3
pw	5.800	tp	-148.3
DECOUPLER		1p	-398.2
dn	h1	plot	
dof	0	wc	250
dmm	yyv	sc	0
w	vs	th	111
dpwr	40	at	12
dmf	9300	no	ph

compound 14a

z-4-109c 1600
exp2 PROTON

SAMPLE		SPECIAL	
date	Jan 5 2006	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~	spin	22
kerf	/vnmr32006-01-06	hst	0.008
z-4-109c	2006-01-06	pw90	10.300
PROTON	30.1	atla	0
ACQUISITION	3591.2	11	FLAGS
at	1.892	in	n
fb	36294	dp	y
bs	5000	hs	mn
di	32	fn	not used
nt	1.000	fn	not used
ct	8	sp	17.3
tn	TRANSMITTER	wp	5129.3
stfq	H1	rt1	5544.9
tof	589.479	rfp	4352.2
tpwr	589.3	rp	-20.4
pw	5.150	ip	-30.5
DECOUPLER	C13	WC	250
dn	0	VS	0
dm	0	mn	24
decwave	45	at	54
opwr	17094	cdc	ph
dmf

z-4-109C13 1600

exp2 CARBON

SAMPLE		SPECIAL	
date	Jan 5 2006	temp	25.0
solvent	CDCl3	gain	not used
file /export/home/~	sp1n	16	
kerf /vnm/sv/date/~	hsc	0.100	
z-4-109C13 200611-	ps90	10.000	
-05/200611-01	at1a	10.000	
ACQUISITION		FLAGS	
sw	37682.5	11	n
at	1.300	in	n
np	96010	dp	v
fb	21000	hs	mn
bs	64		
dt	1.000	1b	PROCESSING
nt	256	fn	0.50
ct	64		not used
TRANSMITTER		SP	DISPLAY
tn	C13	wp	3087.8
strq	150.755	r1	20994.6
tor	3127.6	r1p	13867.0
tpwr	36	tp	11686.8
pw	9.000	1p	-284.1
DECOUPLER		1p	PLOT
dn	H1	wc	250
dof	0	sc	0
de	yvy	vs	221
decave	w	th	8
dpwr	44	at	cdc
def	14286	ph

compound 14b

z-5-41c H1 1400

expt s2pu1

SAMPLE		SPECIAL	
date	Mar 1 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spn	20
ftle	exp	hst	0.008
ACQUISITION		pw90	11.700
sw	6385.9	altf	6.600
at	3.744	flags	
np	47692		
fb	4000		
bs	16		
ol	1.000		
nt	8		
ct	8		
tn	TRANSMITTER	fn	PROCESSING
sfreq	H1	sp	not used
tof	339.762	wp	-37.0
tpwr	57	rf1	3622.2
pw	5.850	rfp	3687.2
DECOUPLER	C13	tp	2982.3
dn	0		2.7
dof	0		-23.3
dm	nmn	WC	250
dma	37	SC	8
dpr	17094	VS	31
dpr	17094	th	54
at	cdc	ph

Z-5-41c C13 1400
 exp1 s2pu1

SAMPLE		SPECIAL	
date	1 2006	temp	not used
solvent	CDCl3	spin	not used
file	exp	hst	20
ACQUISITION		pw90	8.100
sw	25133.5	alpha	20.000
at	1.199	flags	
np	60288	ll	n
fb	14000	in	n
bs	64	dp	y
d1	1.000	hs	nm
nt	256	PROCESSING	
ct	64	lb	1.00
tn	TRANSMITTER C13	fn	not used
sfrq	100.631	sp	DISPLAY-544.8
tot	1345.3	wp	19823.3
tpwr	4.050	rf1	9975.0
pw	DECOUPLER	rfp	7740.0
dn	H1	fp	-2.4
dof	0	lp	-261.3
dm	YVY	WC	250
dmm	39	SC	0
dpwr	9174	VS	509
dmf		th	9
at	no	ph

compound 14c

z-4-125 1600

exp2 PROTON

SAMPLE		SPECIAL	
date	Jan 9 2006	temp	25.0
solvent	CDC13	gain	not used
file	/export/home/~	spin	22
kerf	/vmsr/s/data/~	hst	0.008
z-4-125	2006-01-09	psd	10.300
PROTON	1.710	atla	0
ACQUISITION	9591.2	11	0
at	1.892	in	n
np	36234	dp	v
fb	5000	hs	mn
bs	32	fn	not used
dl	1.000	fn	not used
nt	8	sp	-85.1
ct	8	wp	5645.9
tn	599.479	ft1	5544.6
sfrq	599.3	rfp	4352.2
tot	57	tp	30.9
tpwr	5.158	ip	-36.5
pw	5.158	ip	-36.5
DECOUPLER	C13	WC	258
dn	8	sc	8
dot	8	ss	69
dm	mm	tn	z
discwave	45	at	cdc
dnwt	45	ph	
dat	17094

compound 14d

z-4-125 C13 1600

exp2 CARBON

SAMPLE		SPECIAL	
date	Jan 9 2006	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~	spn	16
kerf	1.000	nsf	1.000
z-4-125	1.000	prf	18.000
-9/CARBON	01 f1d	a1fa	18.000
ACQUISITION		FLAGS	
sw	37562.5	11	n
at	1.300	in	n
np	98010	dp	y
fb	21000	hs	nm
bs	64		
d1	1.000	1b	0.50
nt	256	tn	not used
ct	128		
TRANSMITTER		DISPLAY	-339.7
tn	150.755	C13	2728.7
sfreq	3127.6	rfl	13667.0
tpwr	56	rfp	11606.8
pw	9.000	1p	65.3
DECOUPLER		PLOT	-286.4
dn	H1	wc	250
dot	0	sc	0
dm	yyy	vs	324
decwave	w	th	9
dpr	44	at	
dpr	14266	cdc	ph

compound 14d

z-3-205c H1 1600

exp1 PROTON

SAMPLE		SPECIAL	
date	Mar 2 2006	temp	25.0
solvent	CDCl3	gain	not used
title	/export/home/~	spn	22
key	/nmr38/data/~	hst	0.006
z-3-205c	38.38.38.38	pw90	10.300
2/PROTON	01.71d	atfs	0
ACQUISITION		FLAGS	0
sw	9591.2	11	n
at	1.892	in	n
np	36294	dp	v
fb	5000	hs	nm
bs	32	PROCESSING	nm
d1	1.000	fn	not used
nt	8	DISPLAY	not used
ct	8	sp	-23.7
TRANSMITTER		wp	5419.0
tn	H1	rfl	5548.1
strq	599.479	rtp	4352.2
tot	599.3	tp	-20.4
tpwr	5.7	tp	-35.2
pw	5.150	PL0T	250
DECOUPLER		sc	50
dn	C13	vc	53
dnf	0	th	45
dm	nmr	at	cdc
decave	45	ph	
dpr	45		
dmf	17034

2-3-205C C13 M400
STANDARD 1H OBSERVE

expt szpu1

SAMPLE		SPECIAL	
date	Mar 2 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
nmr1/walkup/autoc	hst	pw90	0.008
02.03.06/0203	pw90	alt90	11.000
ACQUISITION	alt90	flags	20.000
sv	29125.6	in	n
sz	29125.6	in	n
np	60270	in	v
fb	13800	dp	nm
bs	64	hs	nm
d1	1.000	nt	1.00
nt	256	lb	not used
ct	128	fn	not used
TRANSMITTER			
tn	C13	sp	-1491.3
strq	100.613	wp	19640.7
tof	1550.8	rf1	9269.0
tpwr	58	rfp	7749.3
pw	5.500	tp	159.2
DECOUPLER			
dn	H1	plot	-291.0
dof	0	wc	250
dm	yy	sc	0
dma	41	vs	87
dpwr	9900	ai	10
dmf	no	ph

exp1 szput

SAMPLE				SPECIAL			
date	Feb 28 2006	temp	not used	date	Feb 28 2006	temp	not used
solvent	CDCl3	spin	not used	solvent	CDCl3	spin	not used
file	exp	gfn	20	file	exp	gfn	20
ACQUISITION				ACQUISITION			
SW	6395.9	hst	0.008	SW	6395.9	hst	0.008
at	3.744	pw90	11.700	at	3.744	pw90	11.700
np	47692	altA	6.600	np	47692	altA	6.600
fb	400	FLAGS		fb	400	FLAGS	
DS	16	h1	n	DS	16	h1	n
h1	1.008	h2	n	h1	1.008	h2	n
h2	1.008	h3	n	h2	1.008	h3	n
ct	8	PROCESSING	not used	ct	8	PROCESSING	not used
TRANSMITTER				TRANSMITTER			
tn	H1	fn	DISPLAY	tn	H1	fn	DISPLAY
stfq	399.762	sp	-12.0	stfq	399.762	sp	-12.0
tof	399.8	wd	3605.4	tof	399.8	wd	3605.4
tpwr	57	rT1	3607.4	tpwr	57	rT1	3607.4
pw	5.650	rTP	2902.3	pw	5.650	rTP	2902.3
DECOUPLER				DECOUPLER			
C13	1p	PL0T	-17.8	C13	1p	PL0T	-17.8
dn	0	WC	250	dn	0	WC	250
dof	mn	SC	0	dof	mn	SC	0
dsm	37	VS	85	dsm	37	VS	85
dpwr	17094	th	43	dpwr	17094	th	43
dmf		cdc	ph	dmf		cdc	ph

compound 14f

z-5-39c c13 1400

expt s2pu1

SAMPLE		SPECIAL	
date	Feb 28 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	sp1n	20
ACQUISITION		nst	0.008
sw	25133.5	pw90	8.100
at	1.189	alfa	20.000
ap	60208	flacs	
ap	1400		
bs	1.000		
dl	1.256		
nt	128		
ct	TRANSMITTER	ib	PROCESSING
tn	100.531	fn	not used
sfrq	1545.3	sp	13.6
tof	52	wd	18019.2
tpwr	4.050	rf1	9279.6
pw	DECOUPLER	rfp	7740.0
dn	H1	fp	16.1
dof	0	tp	-298.0
dm	y/y		PLOT
dmm	w	wc	250
dpwr	39	sc	0
dmt	9174	vs	527
	at	no	8
		ph

compound 14f

z-5-17-1 H1 I400
expi s2pu1

SAMPLE		SPECIAL	
date	Feb 21 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	hsr	0.008
ACQUISITION		pw90	11.700
sw	6395.9	at7a	6.600
at	3.744	flags	
np	47892	11	n
fb	4000	16	y
bs	1.000	dp	nm
dl	8	hs	
nt	8	fn	not used
ct	8	PROCESSING	
TRANSMITTER		DISPLAY	
tn	399.762	sp	14.0
strq	399.762	wp	3437.4
tof	399.762	ft1	3685.6
tpwr	5.850	ft2	2941.8
pw	5.850	ft3	-21.1
DECOUPLER		PL0T	
dn	C13	1p	250
dof	0	wc	0
dm	nmn	sc	130
dmm	C	vs	54
dpr	37	th	
dpr	17094	at	
dpr		cds	ph

compound 19

z-5-17-1 C13 1400

expt szpu1

SAMPLE		SPECIAL	
date	Feb 21 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	sp1n	20
ACQUISITION			
sw	29133.5	hs	8.066
nt	29133.5	pw	8.100
nt	60288	at1a	20.000
nt	14000	fl	n
bs	64	in	n
dl	1.000	dp	y
nt	256	hs	m
ct	256	PROCESSING	1.00
TRANSMITTER			
tn	C13	fb	not used
sfrq	100.531	sp	DISPLAY
tof	1545.3	wp	2989.8
tpwr	32	ft1	13084.1
pw	4.050	ft2	9268.6
DECOUPLER			
dn	H1	ft3	7258.0
dm	0	ft4	-2711.4
dm	yy	IPLOT	
dpm	39	WC	250
dpm	39	SC	0
dpm	3174	VS	493
dpm		th	10
dpm		ai	no
dpm		ph

z-5-25-1 H1 1600
expt1 PROTON

SAMPLE		SPECIAL	
date	Feb 24 2006	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~	spin	22
kerf	vmarsys/data/~	hst	0.005
z-5-25-1H1	2006-02~	pw90	10.300
-24/PROTON_01.tid	alra	0	0
ACQUISITION		FLAGS	
sw	9591.2	11	n
at	1.692	in	n
mp	36284	dp	y
nb	5032	ns	n
di	1.006	fn	not used
nt	8	td	not used
ct	8	sp	-173.2
tn	TRANSMITTER	wd	5255.4
stfrq	599.479	rf1	5546.4
tot	599.3	rfp	4352.2
tpwr	57	lp	-11.1
pw	5.150	lp	-38.4
DECOUPLER		PLOT	
dn	C13	sc	250
dot	0	vs	0
dm	nmh	th	25
decwave	at	cdc	ph
dpr	45		3
dnt	17034

compound 20

Z-5-25-1 C13 1400

expt s2pu1

SAMPLE		SPECIAL	
date	Feb 24 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	sp1n	20
ACQUISITION		hst	0.008
sw	25133.5	pw90	8.100
at	1.199	atfa	20.000
np	60288	flags	n
fb	14000	in	n
bs	64	dp	y
dl	1.000	hs	nm
nt	512	PROCESSING	1.00
ct	512	lb	not used
tn	100.531	fr	DISPLAY
strq	13453	sp	1501.3
tot	13453	wp	19749.7
tpwr	4.050	rfl	19724.3
pw	DECouPLER	rtp	7740.0
dn	H1	fp	14.1
dof	0	lp	-246.9
dm	yyv	plot	
dma	w	wc	250
dpr	39	sc	0
dpr	VS	vs	1145
daf	9174	th	6
ai	no	ph

compound 20

z-5-13-1 H1 1400

expt s2pu1

SAMPLE		SPECIAL	
date	Feb 18, 2006	temp	not used
solvent	CDC13	gain	not used
f11e	exp	sp1n	28
ACQUISITION		hst	0.008
sw	6395.9	pw90	11.700
at	3.744	af1e	6.600
np	47692	11	n
fb	4000	16	n
bs	1.000	dp	y
d1	8	hs	nn
nt	8	fn	not used
ct	8	DISP	not used
tn	399.762	sp	13.0
strq	399.8	wp	3622.2
tpwr	37	ft1	3622.5
pw	5.850	ftp	2962.5
DECOUPLER	C13	tp	-11.8
dn	0	PLOT	250
dof	0	nmn	0
dm	37	sc	115
dma	37	vs	2
dpr	17094	th	
dpr	17094	ai	cdc ph

compound 21

z-5-13-1 C13 1400
expt s2pu1

SAMPLE		SPECIAL	
date	ed 16 2006	temp	not used
solvent	cdcl3	sat	not used
file	/export/home/~	spin	48
kerf	/nmrsvs/data/~	hst	0.008
z-5-13-1C13	18Feb2~	pw90	8.100
006/CARBON	fid	alfa	20.000
ACQUISITION			
sw	25133.5	11	n
at	1.199	tn	n
np	60288	dp	y
fb	14000	hs	nm
bs	64	1b	1.00
dl	1.000	fn	not used
nt	255	ct	not used
TRANSMITTER			
tn	C13	sd	800.6
sfreq	100.531	rf1	17536.8
tof	1545.3	rfp	9284.2
tpwr	52	tp	7740.0
pw	4.050	tp	-132.8
DECOUPLER			
dn	H1	wc	250
dof	0	sc	0
dm	yvy	vs	836
dma	38	tn	8
dpr	38	at	no ph
dnt	9174

compound 21

Z-4-193-1 H1 1600

expt1 PROTON

SAMPLE		SPECIAL	
date	Feb 25 2006	temp	25.0
solvent	CDC13	gain	not used
file	/export/home/~	sp1n	22
kerf/vnmr/sv/data/~	hst	0.006	
Z-4-193-1H1-2006-0~	pw90	10.300	
2-25/PROTON_01.fid	atfa	0	
ACQUISITION		FLAGS	
sw	939.2	11	n
ac	1.032	1n	n
tp	3.024	dp	y
hs	5000	hs	nm
di	32	tn	not used
nt	1.000	tn	not used
ct	8	sp	-11.4
tn	8	wd	5368.7
stfq	599.479	rft1	5546.4
tof	599.3	rtp	4352.2
tpwr	57	ip	-12.1
pw	5.150	ip	-29.8
DECOUPLER		PL0T	250
dn	C13	vc	25
dm	vs	th	25
decave	nmn	at	54
dpwr	45	cdc	ph
def	17094

compound 22

7-4-193-1 M400
STANDARD 1H OBSERVE

expt1 szpu1

SAMPLE		SPECIAL	
date	Feb 10 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
nmr1	/walkup/autoc~	hst	0.008
10-02-06/0103		pw90	1.000
ACQUISITION	25125.6	attda	20.000
sw	25125.6	flags	n
at	1.199	in	n
np	60270	in	y
fb	13600	dp	nm
bs	64	hs	nm
d1	1.000	PROCESSING	1.00
nt	256	fb	not used
ct	64	fn	not used
TRANSMITTER			
tn	C13	sp	-246.0
strq	100.613	wp	18361.7
tof	1530.0	f1	9381.4
tpwr	17	f2	7446.4
pw	5.800	f3	-137.5
DECOUPLER			
dn	H1	ip	-233.2
dof	0	wc	250
dca	yyv	sc	0
dca	w	vs	37
dpwr	40	th	7
dnt	9300	at	no
		ph

compound 22

2-4-121-1 M408
STANDARD 1H OBSERVE

expt s2pu1

SAMPLE		SPECIAL	
date	Jan 7 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	20
nmr1	/walkup/autoc~	hst	0.008
	07.01.06/0203	pw90	14.000
		altA	20.000
ACQUISITION		FLAGS	
sw	6402.0		
at	4.000	n	
fp	312.6	dp	
bs	not used	hs	
di	1.000	fn	not used
nt	8	fr	not used
ct	8	sp	406.7
tn	H1	wp	3205.2
strq	400.088	rf1	3683.9
tof	417.3	rfp	2904.6
tpwr	57	rfp	-128.0
pw	7.300	ip	-102.8
DECOUPLER		C13	250
dn	0	sc	60
dof	0	mc	54
dsm	43	at	cdc ph
dpwr	15900

z-4-127-1bcl3 1600

expt CARBON

SAMPLE		SPECIAL	
date	Jan 11 2006	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~	spin	16
kerf/vmr/sys/data/~	hst	0.008	
z-4-127-1bcl3 2006~	pw90	18.000	
-01-11/CARBON_01.Y~	alra	10.000	
ACQUISITION		FLAGS	
id	10	n	
sw	37082.5	in	
at	3200	dp	
nt	98010	hs	
fb	21000	ib	
bs	21000	fn	
dl	1.000	not used	
nt	256	DISPLAY	
ct	256	SP	-243.1
tn	TRANSMITTER	WD	27778.3
sfreq	150.755	rfl	13880.3
tof	3127.6	rfd	11606.8
tpwr	56	lp	65.4
pw	9.000	PLDT	-291.5
DECOUPLER		WC	250
dn	H1	SC	0
dof	0	VS	372
dm	yyv	th	10
decwave	v	at	cdc ph
dpr	44		
dmt	14286

compound 23

z-4-171-1 1A00

expl szpu1

SAMPLE		SPECIAL	
date	Jan 31 2006	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	20
kerf/vmr/sys/data/~	hst	0.008	
z-4-171-1.31Jan200	ps90	11.700	
6/PROTON.FID	alra	6.600	
ACQUISITION		FLAS	
sw	6395.9	n	
st	6374.4	n	
md	4768.2	dp	
fb	4080	hs	
bs	1.6	fn	
dl	1.000	DISP	not used
nt	8	SP	360.3
ct	8	WD	3345.0
tn	H1	rf1	799.4
sfreq	399.762	rfp	5.0
tof	399.8	ip	-45.7
tdwr	57	WC	254
pw	5.850	SC	0
dn	0	VS	47
dot	0	mm	54
dm	mm	tn	
dmr	37	at	
dwr		cdc	ph
dwt	17084

compound 24

z-4-171-1 C13 1400
 expl s2pu1

SAMPLE		SPECIAL	
date	31 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	25133.5	pw90	8.100
at	1.199	alpha	20.000
np	60288	flags	
fb	14000	i1	n
bs	64	in	n
d1	1.000	dp	y
nt	256	hs	nh
ct	TRANSMITTER C13	fb	1.00
tr	100.531	fn	not used
sfcr	1545.3	sd	DISPLAY 807.5
tpwr	52	wp	20476.1
pw	4.050	ft1	9277.3
DECOUPLER		rfp	7740.0
dn	H1	fp	170.2
dof	0	lp	-275.3
dm	yyv	PLOT	
dma	w	sc	250
dwr	39	vs	0
dmf	9174	lh	830
	at	no	ph

compound 24

z-4-165-1 1600

expt1 PROTON

SAMPLE		SPECIAL	
date	Jan 28 2006	temp	25.0
solvent	CDC13	gain	not used
file	/export/home/~	spin	22
kerf	/vmrjvs/data/~	hst	0.006
z-4-165-1	2006-01-~	pw90	10.300
28/PROTON	01.fid	atfa	0
ACQUISITION		FLAGS	
sw	9591.2	11	n
at	1.092	in	n
np	36294	dp	y
fb	5000	hs	nm
bs	32		
d1	1.000	fn	not used
nt	8		
ct	8	sp	371.9
transmitter	H1	wd	4875.6
trf	599.479	rfp	2428.6
tofr	589.3	rfp	1228.9
tpwr	57	lp	-14.7
pw	5.150	ip	-37.2
DECOUPLER		WC	250
dn	C13	SC	0
dof	0	VS	25
dm	nmn	th	54
decwave	g	at	cdc ph
dpwr	45		
dnt	17094

compound 25

z-4-195-1 C13 1400

expi s2pu1

SAMPLE		SPECIAL	
date	Feb 13 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ft	exp	hst	0.008
ACQUISITION		pw90	8.100
sw	25133.5	at	20.000
at	1.199	al	20.000
np	60288	fb	1.000
bs	14000	in	1.000
dl	1.000	dp	1.000
nt	256	hs	1.000
ct	64	ns	1.000
TRANSMITTER C13		fn	not used
tn	100.531	sp	not used
sf	1545.3	wp	18494.8
tpwr	4.050	rf1	9288.8
pw	4.050	rfp	7740.0
DECOUPLER		fp	63.4
dn	0	tp	-265.9
dof	0	pl	250
dm	yyv	wc	392
dmm	w	sc	392
dpr	39	vs	392
dpr	9174	vs	392
dpr	9174	vs	392
dmf	9174	vs	392
at	no	ph	6

compound 25

Z-5-43-1 1600

exp1 PROTON

SAMPLE		SPECIAL	
date	Mar 2 2006	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~	soin	0.006
kerf/vmrsvs/data/~	hst	pw90	10.300
z-5-43-1 2006-03-0	pw90	alpha	0
2/PROTON.01.fid	alpha	flags	0
ACQUISITION			
sw	9591.2	11	n
at	1.692	in	n
np	36294	dp	y
fb	5000	hs	nm
bs	32	fn	not used
dl	1.000	fn	not used
nt	8	sp	-10.5
ct	8	wp	5419.0
TRANSMITTER			
tn	H1	rfl	5547.5
strq	599.479	rtp	4352.2
tof	599.3	tp	-20.3
tpwr	57	tp	-35.9
pw	5.150	plot	250
DECOUPLER			
dn	C13	sc	0
bof	0	vs	41
dm	mm	th	38
decwave	at	cdc	ph
dpwr	43		
dwt	17094

compound 18

z-3-185-1 C13 1600

exp2 CARBON

SAMPLE		SPECIAL	
date	Jan 9 2006	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~	spin	16
kerf	marcys/date/~	hsc	0.00
z-3-185-1	2006	psd	18.000
01-03/CARBON_01.1	~	atla	10.000
ACQUISITION		FLAGS	
sw	37682.5	in	n
at	1.300	dp	y
np	98010	hs	nm
fb	21000	PROCESSING	
bs	64	1b	0.50
dl	1.000	fn	not used
nt	256	DISPLAY	
ct	256	sp	-2214.8
TRANSMITTER		wp	28759.2
tn	C13	ft1	13871.6
strq	150.735	ftp	11686.8
tot	3127.6	tp	88.5
tpwr	9.000	lp	-286.1
PLOT		PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	424
dm	yvy	th	12
decwave	y	ai	cdc ph
dpr	44		
dmf	14286

compound 18

z-4-2l in acetone 1600
expi PROTON

SAMPLE SPECIAL
date Nov 21 2005 temp 25.0
solvent Acetone gain not used
f1e /export/home/~ hst 22
kerr/vnmrsvs/date/~ hst 0.008
z-4-2lacetone-2005~ pw90 10.300
-11-21/PROTON_01.f~ a1fa 0

ACQUISITION
id 11
sw 9592.3 1n
at 1.892 dp
np 36298 hs
fb 5000
bs 32 fn
dl 1.008
nt 8
ct 8
tn TRANSMITTER 8
stf9 599.498 h1
tof 599.3 f1p
tpwr 57 1p
pw 5.150 wc
DECOUPLER C13 vs
dn 0 th
dm 0 cdc
deccave g ph
dpr 45
dmf 17094

PROCESSING
not used
nm
nm
not used
5.8
5604.7
2414.6
1223.8
33.3
-43.6
250
0
27
2

FLAGS
n
n
y
nm
nm
not used
5.8
5604.7
2414.6
1223.8
33.3
-43.6
250
0
27
2

compound 16

z-5-45-1 H1 1600

expt1 PROTON

SAMPLE		SPECIAL	
date	Mar 6 2006	temp	25.0
solvent	CDCl3	gain	not used
title	/export/home/~hsc	spin	16
kerf	/var/sys/2006-03-~hsc	0.006	
z-5-45-1 H1 2006-03-~hsc		10.500	
-06/PROTON 0.11d	at1a	0	
ACQUISITION		FLAGS	
sw	9591.2	f1	n
at	1.892	in	n
np	36294	dp	y
fb	5000	hs	nm
bs	32		
d1	1.000	fn	not used
nt	8	DISPLAY	
ct	8	sp	15.3
in	TRANSMITTER	wp	5381.3
tn	H1	rfl	5548.9
strq	599.479	rtp	4352.2
tor	599.3	rp	-84.3
cpwr	37	tp	-31.7
pw	5.150	WC	250
dn	DECOUPLER	C13	0
dof	0	vs	70
dm	0	th	5
decwave	g	at	cdc
dpwr	45	ph	
dmf	17094

z-5-45-1 C13 1600
expt1 CARBON

SAMPLE		SPECIAL	
date	Mar 7 2006	temp	25.0
solvent	CDC13	gain	not used
ft18	/export/home/-	spin	22
kerf	/vms35/kerf/-	hst	9.086
z-07	C13-1600	pw9	18.000
3-07	CARBON-61.718	delta	10.000
ACQUISITION		FLAGS	
sw	37682.5	11	n
at	1.300	1n	n
mp	98010	dp	y
td	21000	hs	nm
bs	64		
d1	1.000	1b	0.50
nt	512	fn	not used
ct	192		
TRANSMITTER		DISPLAY	
tr	C13	sp	-2222.2
srq	158.753	wp	2390.5
tdr	3127.8	ftd	1380.1
tdwr	56	ftd	1380.1
pw	9.000	1p	-178.9
DECOUPLER		PLOT	-336.6
dn	H1	WC	250
dof	0	SC	0
dm	YVY	VS	444
decwave	44	th	10
dpwr	44	at	cdc ph
dat	14286