Supporting Information

Generation of 3-Pyridyl Biaryl Systems via Palladium-Catalyzed Suzuki Cross-Coupling of Aryl Halides with 3-Pyridylboroxin

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Experimental Procedures and Analytical Data

General. All non-aqueous reactions were performed under a dry atmosphere of nitrogen unless otherwise specified. Commercial grade reagents and anhydrous solvents were used as received from vendors and no attempts were made to purify or dry these components further. Removal of solvents under reduced pressure was accomplished with a Buchi rotary evaporator at approximately 28 mm Hg pressure using a Teflon-linked KNF vacuum pump. Thin layer chromatography was performed using 1" x 3" AnalTech No. 02521 silica gel plates with fluorescent indicator. Visualization of TLC plates was made by observation with either short wave UV light, 10% phosphomolybdic acid in ethanol or in iodine vapors. Flash column chromatography was carried out using Kieselgel silica gel 60. Proton and carbon NMR spectra were obtained on a 300 MHz Nuclear Magnetic Resonance Spectrometer and are reported in ppm δ values, using tetramethylsilane as an internal reference. Melting points are uncorrected. API Mass spectroscopic analyses were performed using atmospheric pressure chemical ionization (APCI).

Preparation of 3-Pyridylboroxin (3).¹ A 1 L, three-necked flask equipped with an internal temperature probe, dropwise addition funnel, stirbar, and septa was charged with toluene (320 mL) and tetrahydrofuran (80 mL) and placed under a nitrogen atmosphere. The flask was then charged with triisopropyl borate (55.4 mL, 240 mmol) and 3-bromopyridine (1, 19.3 mL, 200 mmol). The mixture was cooled to $-40\text{ }^{\circ}\text{C}$ using a dry ice/acetone bath. *n*-Butyllithium (1.6 M in hexanes, 160 mL, 240 mmol) was added dropwise via the addition funnel over a period of 1 h, and the mixture was stirred for an additional 30 min while the temperature was held at $-40\text{ }^{\circ}\text{C}$. The acetone/dry ice bath was removed and the reaction was allowed to warm to $-20\text{ }^{\circ}\text{C}$ after which an aqueous 2 N hydrogen chloride solution (200 mL) was added. The mixture was allow to reach room temperature and the aqueous layer (pH 1) was collected and adjusted to pH 7 with an aqueous 5 N sodium hydroxide solution (30 mL), causing a white solid to precipitate out of solution. The mixture was treated with sodium chloride (50 g), and extracted three times with tetrahydrofuran (250 mL). The combined extracts were concentrated under

reduced pressure to provide a solid, which was taken up in acetonitrile (100 mL) and the resulting slurry was heated at 70 °C for 30 min. The mixture was cooled to 0 °C and stirred for 5 h, after which the solids were collected by vacuum filtration and washed with cold acetonitrile (20 mL). The material was dried under vacuum to afford of 3-pyridylboroxin (**3**) as a white solid (18.8 g, 90%). A satisfactory melting point for this solid could not be obtained: ¹H NMR (300 MHz, CD₃OD) δ 8.61 (br, 1H), 8.56-8.54 (m, 1H), 8.45 (m, 1H), 7.72 (m, 1H); ¹³C NMR (300 MHz, CD₃OD) δ 123.5, 123.3, 147.8; APCI MS: *m/z* 315 [C₁₅H₁₂B₃N₃O₃ + H]⁺; Anal. Calcd for C₁₅H₁₂B₃N₃O₃ • 0.85 H₂O: C, 54.59; H, 4.18; N, 12.73. Found: C, 54.95; H, 3.96; N, 12.39.

Suzuki Coupling Reaction: General Procedure. To a degassed mixture of the aryl halide or triflate (1.0 mmol), 3-pyridylboroxin (**3**, 0.5 mmol), and 2 M aqueous sodium carbonate solution (4.0 mmol) in 1,4-dioxane (5 mL) at room temperature under nitrogen was added tetrakis(triphenylphosphine)palladium(0) (0.10 mmol). The mixture was heated at reflux for 16 h and the solvent was removed under reduced pressure. The residue was dissolved in a 1:1 solution of methylene chloride/methanol (20 mL), filtered through diatomaceous earth and the solvents were removed under reduced pressure. The crude product was purified by medium-pressure liquid chromatography on silica gel, eluting with hexanes/ethyl acetate (20:1 to 10:1). Final products were characterized by APCI MS, ¹H NMR, ¹³C NMR, and CHN Analysis. The reaction conditions were not optimized.

[2,3']Bipyridinyl (4a).² ¹H NMR (CDCl₃) δ 9.21 (s, 1H), 8.68 (d, *J* = 8.3 Hz, 1H), 8.64 (d, *J* = 8.3 Hz, 1H), 8.35 (dd, *J* = 1.0, 8.4 Hz, 1H), 7.81 (m, 2H), 7.38 (m, 1H), 7.20 (m, 1H); ¹³C NMR (CDCl₃) δ 120.9, 123.1, 123.9, 134.7, 135.2, 137.3 148.6, 150.2, 150.4, 155.1; APCI MS: *m/z* 157 [C₁₀H₈N₂ + H]⁺. Anal. Calcd for C₁₀H₈N₂ • 0.1 CH₃OH: C, 76.11; H, 5.31; N, 17.58. Found: C, 76.18; H, 5.31; N, 17.15.

4-Pyridin-3-ylbenzonitrile (4b).³ mp 95-96 °C; ¹H NMR (CDCl₃) δ 8.92 (s, 1H), 8.66 (d, *J* = 8.2 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.38 (m, 1H); ¹³C NMR (CDCl₃) δ 110.1, 116.6, 121.9, 125.9, 131.0, 132.6, 132.9, 140.5, 146.4, 147.9;

APCI MS: m/z 181 [$C_{12}H_8N_2 + H$]⁺. Anal. Calcd for $C_{12}H_8N_2 \cdot 0.2 H_2O$: C, 78.41; H, 4.61; N, 15.24. Found: C, 78.80; H, 4.34; N, 14.80.

3-(4-Methoxyphenyl)pyridine (4c).⁴ mp 63-64 °C; ¹H NMR (CDCl₃) δ 8.78 (s, 1H), 8.50 (m, 1H), 7.79 (d, J = 8.2 Hz, 1H), 7.50 (d, J = 8.4 Hz, 2H), 7.36 (m, 1H), 6.95 (d, J = 8.4 Hz, 2H) 3.83 (s, 3H); ¹³C NMR (CDCl₃) δ 55.7, 114.9, 123.8, 128.5, 134.2, 136.6, 148.3, 148.4, 160.1; APCI MS: m/z 186 [$C_{12}H_{11}NO + H$]⁺. Anal. Calcd for $C_{12}H_{11}NO$: C, 77.81; H, 5.99; N, 7.56. Found: C, 77.85; H, 5.89; N, 7.46.

3-*p*-Tolylpyridine (4d,e).⁵ ¹H NMR (CDCl₃) δ 8.81 (s, 1H), 8.52 (m, 1H), 7.83 (m, 1H), 7.44 (d, J = 8.2 Hz, 2H), 7.21 (m, 3H), 2.34 (s, 3H); ¹³C NMR (CDCl₃) δ 21.4, 123.8, 127.3, 127.5, 129.4, 130.1, 134.4, 135.3, 136.9, 138.3, 148.5; APCI MS: m/z 170 [$C_{12}H_{11}N + H$]⁺. Anal. Calcd for $C_{12}H_{11}N \cdot 0.1 H_2O$: C, 84.27; H, 6.60; N, 8.19. Found: C, 84.33; H, 6.49; N, 8.27.

3-Thiophen-2-ylpyridine (4f).⁶ ¹H NMR (CDCl₃) δ 8.87 (s, 1H), 8.56 (m, 1H), 7.87 (m, 1H), 7.29 (m, 3H), 7.05 (m, 1H); ¹³C NMR (CDCl₃) δ 122.5, 123.2, 124.9, 127.2, 129.4, 131.9, 139.4, 146.0, 147.4; APCI MS: m/z 162 [$C_9H_7NS + H$]⁺. Anal. Calcd for $C_9H_7NS \cdot 0.1 H_2O$: C, 66.31; H, 4.45; N, 8.59. Found: C, 66.12; H, 4.27; N, 8.55.

3-Naphthalen-1-ylpyridine (4g).⁷ ¹H NMR (CDCl₃) δ 8.87 (s, 1H), 8.75 (d, J = 8.2 Hz, 1H), 7.90 (m, 2H), 7.74 (m, 2H), 7.35 (m, 5H); ¹³C NMR (CDCl₃) δ 122.0, 124.2, 124.3, 124.9, 125.4, 126.2, 127.3, 127.4, 130.4, 132.7, 135.2, 136.1, 147.5, 149.5; APCI MS: m/z 206 [$C_{15}H_{11}N + H$]⁺. Anal. Calcd for $C_{15}H_{11}N$: C, 87.77; H, 5.40; N, 6.82. Found: C, 87.47; H, 5.20; N, 6.81.

4-Pyridin-3-ylbenzaldehyde (4h).⁸ mp 54-56 °C; ¹H NMR (CDCl₃) δ 10.10 (s, 1H), 9.03 (s, 1H), 8.75 (d, J = 8.2 Hz, 1H), 7.86 (m, 3H), 7.64 (d, J = 8.3 Hz, 2H), 7.33 (m, 1H); ¹³C NMR (CDCl₃) δ 124.0, 128.0, 130.7, 134.8, 135.6, 136.2, 144.0, 148.7, 149.9,

191.9; APCI MS: m/z 184 [$C_{12}H_9NO + H$]⁺. Anal. Calcd for $C_{12}H_9NO \cdot 0.1 H_2O$: C, 77.9; H, 5.01; N, 7.57. Found: C, 77.68; H, 4.81; N, 7.43.

1-(4-Pyridin-3-ylphenyl)ethanone (4i).⁴ ¹H NMR (CD_3OD) δ 8.89 (s, 1H), 8.65 (m, 1H), 8.12 (m, 3H), 7.74 (d, $J = 8.3$ Hz, 2H), 7.53 (m, 1H), 2.58 (s, 3H). ¹³C NMR (CD_3OD) δ 125.1, 128.7, 131.7, 136.2, 136.9, 137.2, 144.0, 148.7, 151.2, 200.9; APCI MS: m/z 198 [$C_{13}H_{11}NO + H$]⁺. Anal. Calcd for $C_{13}H_{11}NO \cdot 0.15 H_2O$: C, 78.10; H, 5.70; N, 7.01. Found: C, 78.01; H, 5.85; N, 6.90.

3-(2-Nitrophenyl)pyridine (4j).⁹ ¹H MNR ($CDCl_3$) δ 8.86 (m, 1H), 8.62 (m, 1H), 7.82 (m, 1H), 7.44 (m, 2H), 7.25 (m, 2H); ¹³C NMR ($CDCl_3$) δ 123.2, 124.6, 129.1, 132.1, 132.8, 135.4, 148.6, 149.4; APCI MS: m/z 201 [$C_{11}H_8N_2O_2 + H$]⁺. Anal. Calcd for $C_{11}H_8N_2O_2$: C, 66.00; H, 4.03; N, 13.99. Found: C, 65.93; H, 3.69; N, 13.77.

References

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¹H NMR Spectrum of Compound 3.

¹³C NMR Spectrum of Compound 3.

Current Data Parameters
NAME JJA-A-67
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030428
Time 21.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT MeOD
NS 10000
DS 4
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 14596.5
DM 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SF01 75.4756431 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.06 dB
PL13 26.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 16384
SF 75.4677952 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 155.000 ppm
F1 11697.51 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPNCH 8.00000 Hz
HZCM 603.74243 Hz

location: 4

¹H NMR Spectrum of Compound 4a.

¹³C NMR Spectrum of Compound 4a.

Current Data Parameters
NAME CLC-XYZ-50
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021219
Time 21.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 10000
DS 4
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 18390.4
DM 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SF01 75.4756431 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.98 dB
PL13 26.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 16384
SF 75.4677190 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 215.000 ppm
F1 16225.56 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPHMC 11.00000 ppm/cm
HZCM 830.14490 Hz/cm

4a

¹H NMR Spectrum of Compound 4b.

Current Data Parameters
 NAME CLC-XYZ-52
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021120
 Time 18 09
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 143.7
 DW 104.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SF01 300.1319508 MHz

F2 - Processing parameters
 SI 16384
 SF 300.1299979 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 11.000 ppm
 F1 3301.43 Hz
 F2P -0.500 ppm
 F2 -150.07 Hz
 PPMCM 0.57500 ppm/cm
 HZCM 172.57475 Hz/cm

¹³C NMR Spectrum of Compound 4b.

Current Data Parameters
NAME CLC-XY7-52
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021205
Time 21.28
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TO 32768
SOLVENT CDCl3
NS 10000
DS 4
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 16390.4
DM 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SF01 75.4756431 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.98 dB
PL13 26.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 16384
SF 75.4678846 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FIP 215.000 ppm
F1 16225.60 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14673 Hz/cm

¹H NMR Spectrum of Compound 4c.

Current Data Parameters
 NAME CLC-XYZ-54
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021120
 Time 18.16
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 143.7
 DW 104.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SF01 300.1319508 MHz

F2 - Processing parameters
 SI 16384
 SF 300.1300055 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 FIP 11.000 ppm
 F1 3301.43 Hz
 F2 -0.500 ppm
 F2 -150.07 Hz
 PPNCH 0.57500 ppm/cm
 HZCH 172.57475 Hz/cm

¹³C NMR Spectrum of Compound 4c.

Current Data Parameters
NAME CLC-XY2-54_
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021122
Time 21.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TO 32768
SOLVENT CDCl3
NS 10000
DS 4
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.87700404 sec
RG 18390.4
DM 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

===== CHANNEL f1 =====
NUC1 ¹³C
P1 9.00 usec
PL1 0.00 dB
SF01 75.4756431 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.98 dB
PL13 26.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
S1 16384
SF 75.4677190 MHz
WDW EK
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FIP 215.000 ppm
F1 16225.56 Hz
F2 -5.000 ppm
F2 -377.34 Hz
PPMCH 11.00000 ppm/cm
HZCH 830.14490 Hz/cm

¹H NMR Spectrum of Compound 4d.

Current Data Parameters
NAME CLC-XYZ-56
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021120
Time 18.28
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 143.7
DW 104.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SF01 300.1319508 MHz

F2 - Processing parameters
SI 16384
SF 300.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.57500 ppm/cm
HZCM 172.57475 Hz/cm

¹³C NMR Spectrum of Compound 4d.

Current Data Parameters
 NAME CLC-XYZ-56-
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021123
 Time 1.19
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TO 32768
 SOLVENT CDCl3
 NS 10000
 DS 4
 SWH 18832.393 Hz
 FIDRES 0.574719 Hz
 AQ 0.8700404 sec
 RG 16390.4
 DW 26.550 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 0.00 dB
 SF01 75.4756431 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 18.98 dB
 PL13 26.00 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 S1 16384
 SF 75.4677190 MHz
 XDH EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 215.000 ppm
 F1 16225.56 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PTHCN 11.00000 ppm/cm
 HZCN 830.14490 Hz/cm

¹H NMR Spectrum of Compound 4f.

Current Data Parameters
NAME CLC-XYZ-58
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021120
Time 18.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT COC13
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 143.7
DW 104.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SF01 300.1319508 MHz

F2 - Processing parameters
SI 16384
SF 300.1300049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPHMC 0.57500 ppm/cm
HZCM 172.57475 Hz/cm

4f

¹³C NMR Spectrum of Compound 4f.

Current Data Parameters
NAME CLC-XYZ-SB
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021125
Time 21.42
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TO 32768
SOLVENT COC13
NS 10000
DS 4
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 18390.4
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SF01 75.4756431 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.90 dB
PL13 26.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 16384
SF 75.4678238 MHz
WDW EN
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FIP 215.000 ppm
F1 16225.56 Hz
F2 -5.000 ppm
F2P -377.34 Hz
PPMCH 11.00000 ppm/cm
HZCH 830.14606 Hz/cm

4f

¹H NMR Spectrum of Compound 4g.

Current Data Parameters
NAME CLC-XYZ-64
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030114
Time 12.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
OW 104.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SF01 300.1319508 MHz

F2 - Processing parameters
SI 16384
SF 300.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 11.000 ppm
F1 3301.43 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.07800 Hz/cm

4g

¹³C NMR Spectrum of Compound 4g.

Current Data Parameters
 NAME CLC-XYZ-B4_
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030114
 Time 22.01
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TO 32768
 SOLVENT CDCl3
 NS 10000
 DS 4
 SWH 18632.393 Hz
 FIDRES 0.574719 Hz
 AQ 0.8700404 sec
 RG 18390.4
 DW 26.550 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.50000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 0.00 dB
 SF01 75.4756431 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 18.98 dB
 PL13 26.00 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 16384
 SF 75.4678306 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 215.000 ppm
 F1 16225.58 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PRNCH 11.00000 ppm/cm
 HZCM 830.14606 Hz/cm

4g

AMRI: 3rd Floor C-Wing
 location: 4
 Employee Number: 502
 Project Number: 1537-1246

¹H NMR Spectrum of Compound 4h.

Project Number: 1537-1246

```

Current Data Parameters
NAME          CLC-A-2-68-
EXPNO        10
PROCNO       1

F2 - Acquisition Parameters
Date_        20030106
Time         18.13
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           269630
FIDRES       0.32766
AQ           0.0113
SOLVENT      DMSO
VS           1500
DS           4
SWH          18632.303 Hz
FIDRES5      0.574719 Hz
AQ5          0.8700404 sec
RG           16350.4
RG5          26.550 usec
JE           6.00 usec
TE           300.0 K
D1           0.50000000 sec
d11          0.03000000 sec
d12          0.00502000 sec

***** CHANNEL f1 *****
NUC1         13C
P1           9.00 usec
PL1          0.00 dB
SF01         75.4756431 MHz

***** CHANNEL f2 *****
CPDPRG2      waltz16
NUC2         1H
P2           80.00 usec
PL2          0.00 dB
PL12         18.96 dB
PL13         26.00 dB
SF02         500.132005 MHz

F2 - Processing parameters
SI           16384
SF           75.4677190 MHz
WDW          EP
SSB          0
GB           0
PC           1.40

1D NMR plot parameters
CY           20.00 cm
CX           12.50 cm
YUP          215.000 ppm
F1           16225.56 Hz
F2           -5.006 ppm
F3           -3177.34 Hz
DCPH1       11.00000 ppm/cm
DCPH2       830.14490 Hz/cm

```

¹H NMR Spectrum of Compound 4i.

Current Data Parameters
 NAME WTS-XYZ-7-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021105
 Time 9.28
 INSTRUM spect
 PROBHD 5 mm BBI 1H-BB
 PULPROG zg30
 TO 32768
 SOLVENT MeOD
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 1024
 DW 104.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.75 usec
 PL1 1.00 dB
 SFO1 300.1319508 MHz

F2 - Processing parameters
 SI 16384
 SF 300.1300060 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 0.00 cm
 FIP 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPKCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm

¹³C NMR Spectrum of Compound 4i.

Current Data Parameters
 NAME WTZ-XYZ-7-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021106
 Time 22 18
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TO 32768
 SOLVENT MeOD
 NS 10000
 DS 4
 SWH 18632.193 Hz
 FIDRES 0.574719 Hz
 AQ 0.8700404 sec
 RG 4597.6
 DH 26.350 usec
 DE 6.00 usec
 TE 300.2 K
 D1 0.5000000 sec
 d11 0.0300000 sec
 d12 0.0000200 sec
 d13 0.0000000 sec
 d14 0.0000000 sec
 d15 0.0000000 sec
 d16 0.0000000 sec
 d17 0.0000000 sec
 d18 0.0000000 sec
 d19 0.0000000 sec
 d20 0.0000000 sec
 d21 0.0000000 sec
 d22 0.0000000 sec
 d23 0.0000000 sec
 d24 0.0000000 sec
 d25 0.0000000 sec
 d26 0.0000000 sec
 d27 0.0000000 sec
 d28 0.0000000 sec
 d29 0.0000000 sec
 d30 0.0000000 sec
 d31 0.0000000 sec
 d32 0.0000000 sec
 d33 0.0000000 sec
 d34 0.0000000 sec
 d35 0.0000000 sec
 d36 0.0000000 sec
 d37 0.0000000 sec
 d38 0.0000000 sec
 d39 0.0000000 sec
 d40 0.0000000 sec
 d41 0.0000000 sec
 d42 0.0000000 sec
 d43 0.0000000 sec
 d44 0.0000000 sec
 d45 0.0000000 sec
 d46 0.0000000 sec
 d47 0.0000000 sec
 d48 0.0000000 sec
 d49 0.0000000 sec
 d50 0.0000000 sec
 d51 0.0000000 sec
 d52 0.0000000 sec
 d53 0.0000000 sec
 d54 0.0000000 sec
 d55 0.0000000 sec
 d56 0.0000000 sec
 d57 0.0000000 sec
 d58 0.0000000 sec
 d59 0.0000000 sec
 d60 0.0000000 sec
 d61 0.0000000 sec
 d62 0.0000000 sec
 d63 0.0000000 sec
 d64 0.0000000 sec
 d65 0.0000000 sec
 d66 0.0000000 sec
 d67 0.0000000 sec
 d68 0.0000000 sec
 d69 0.0000000 sec
 d70 0.0000000 sec
 d71 0.0000000 sec
 d72 0.0000000 sec
 d73 0.0000000 sec
 d74 0.0000000 sec
 d75 0.0000000 sec
 d76 0.0000000 sec
 d77 0.0000000 sec
 d78 0.0000000 sec
 d79 0.0000000 sec
 d80 0.0000000 sec
 d81 0.0000000 sec
 d82 0.0000000 sec
 d83 0.0000000 sec
 d84 0.0000000 sec
 d85 0.0000000 sec
 d86 0.0000000 sec
 d87 0.0000000 sec
 d88 0.0000000 sec
 d89 0.0000000 sec
 d90 0.0000000 sec
 d91 0.0000000 sec
 d92 0.0000000 sec
 d93 0.0000000 sec
 d94 0.0000000 sec
 d95 0.0000000 sec
 d96 0.0000000 sec
 d97 0.0000000 sec
 d98 0.0000000 sec
 d99 0.0000000 sec
 d100 0.0000000 sec

F2 - Processing parameters
 SI 16384
 SF 75 4676118 MHz
 WDW G
 SSB G
 LB 2.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 ZP 215.000 ppm
 C1 16225.54 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 830.14374 Hz/cm

¹H NMR Spectrum of Compound 4j.

Current Data Parameters
 NAME wts-xyz-19-2
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030207
 Time 17:31
 INSTRUM spect
 PROBHD 5 mm BBI 1H-BB
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 456 1
 DW 104.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 ¹H
 P1 9.07 usec
 PL1 1.00 dB
 SF01 300.1319508 MHz

F2 - Processing parameters
 SI 16384
 SF 300.1300074 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 0.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56827 Hz/cm

4j

¹³C NMR Spectrum of Compound 4j.

Current Data Parameters
NAME wts-xyz-19-2_
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030211
Time 19:52
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3200
DS 4
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 18390.4
DM 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 16.50 usec
PL1 -3.50 dB
SFO1 75.4756431 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 19.90 dB
PL13 20.92 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 16384
SF 75.4677443 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

ID NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 215.000 ppm
F1 16225.57 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPNCH 11.00000 ppm/cm
HZCH 830.14514 Hz/cm