*Supporting Information***One-Step Synthesis of α -chloro Acetophenones from Acid Chlorides and Aryl Precursors**

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General Procedures

All reactions were run under an atmosphere of nitrogen using commercially available anhydrous solvents.

All NMR spectra were recorded on either a 400 or 500 MHz instrument in CDCl₃ as denoted on the spectra.

The following general procedure was used for the synthesis of all substrates unless otherwise noted:

To a solution of aryl substrate (8.8 mmol) in dry THF (10 mL) at -60 °C is added *n*-hexyllithium (1.0 equiv. of 2.3M solution in hexane) maintaining the internal temperature below -55 °C. Upon complete addition

the reaction is aged for 1 hour at -60 °C followed by the addition of ZnCl₂ (1.0 equiv. of 0.5M solution in THF) dropwise maintaining the internal temperature below -55 °C. The resulting solution is warmed to -10 °C followed by the addition of CuCl (0.10 equiv.) and acyl chloride (1.1 equiv.). The reaction is allowed to warm to room temperature and aged overnight.

The reaction is quenched with 1N HCl (15 mL) and diluted with isopropyl acetate (15 mL). The aqueous layer is cut and the organic washed again with 1N HCl (15 mL) followed by 1N NH₄OH (2 x 15 mL) and water (15 mL). The product is either isolated directly from the crude mixture by crystallization from -30 °C heptane or chromatographed. Differences between assay and isolated yields are due to mother liquor losses in unoptimized crystallizations.

Compound 2a: The above procedure was followed exactly except for the substitution of *sec*-butyllithium for *n*-hexyllithium. Isolated as a solid in 68% yield from aryl precursor after crystallization: ¹H NMR δ 7.96 (m, 1H), 7.59 (m, 1H), 7.29 (t, 1H, *J* = 7.6 Hz) 7.17 (m, 1H), 4.73 (m, 2H), ppm; ¹³C NMR δ 50.0 (d, *J* = 11.2 Hz), 116.6 (d, *J* = 23.2 Hz), 122.8 (d, *J* = 13.6 Hz), 124.8 (d, *J* = 3.2 Hz), 131.1 (d, *J* = 2.4 Hz), 135.6 (d, *J* = 9.6 Hz), 161.8 (d, *J* = 253.9 Hz), 189.1 (d, *J* = 4.0 Hz); ¹⁹F NMR δ -108.6. Anal. Calcd for C₈H₆ClFO: C, 55.67; H, 3.5. Found: C, 55.30; H, 3.44.

Current Data Parameters
NAME 224662-41d
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 2006077
Time 18.35
INSTRUM spect
PROBHD 5 mm QNP
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 161.3
DM 76.200 usec
DE 6.00 usec
TE 300.2 K
T1 0.10000000 sec
MREST 0.00000000 sec
MCWK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SFO1 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 9.750 ppm
F1 3901.27 Hz
F2P -0.250 ppm
F2 -100.03 Hz
PPHCH 0.42553 ppm/cm
HZCM 170.26608 Hz/cm

nmr400c h1

2a

Current Data Parameters
NAME 254662-41d
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 2006077
Time 18.41
INSTRUM spect
PROBHD 5 mm QNP
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 25246.719 Hz
FIDRES 0.400463 Hz
AQ 1.2468118 sec
RG 3981
DM 19.000 usec
DE 8.00 usec
TE 300.2 K
T1 0.10000000 sec
T11 0.03000000 sec
MREST 0.00000000 sec
MCWK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 8.00 usec
PL1 0.00 dB
SFO1 100.6297884 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 190.00 dB
PL12 18.00 dB
SFO2 400.132007 MHz

F2 - Processing parameters
SI 367808
SF 100.6197780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 266.000 ppm
F1 26234.81 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHCH 9.38170 ppm/cm

c-13 nmr400c

2a

```

Current Data Parameters
NAME 204682-416
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060707
Time 16.38
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4
DS 4
SWH 119944.860 Hz
FIDRES 0.00070 Hz
AQ 0.0003486 sec
RG 3200.0
SN 4.40 usec
DE 0.30 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

===== CHANNEL f1 =====
NUC1 19F
P1 0.30 usec
PL1 -1.00 dB
SFO1 376.480133 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 19.00 dB
PL12 19.00 dB
SFO2 400.130007 MHz

F2 - Processing parameters
SI 32768
SF 376.480133 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

SD NMR slit parameters
CX 23.50 cm
CY 10.00 cm
F1P -44.300 gpm
F1 -10001.70 Hz
F2P -10001.00 Hz
F2 -10001.00 Hz
FPMCH 9.04347 gpm/cm

```

nmr400c h-1 decoupled f-19

2a

Compound 2b: Isolated as a solid in 72% yield from aryl precursor after crystallization: ^1H NMR δ 7.72 (m, 1H), 7.44 (m, 1H), 7.26 (m, 1H), 4.74 (d, 2H, $J = 2.8$ Hz), ppm; ^{13}C NMR δ 49.7 (d, $J = 10.5$ Hz), 122.5 (dd, $J = 17.2, 1.2$ Hz), 124.8 (m, 2C), 125.7 (m), 150.3 (dd, $J = 256.6, 14.2$ Hz), 150.8 (dd, $J = 251.0, 14.2$ Hz), 188.3 (dd, $J = 4.3, 3.1$ Hz); ^{19}F NMR δ -135.2 (d, $J = 21.4$ Hz), -137.1 (d, $J = 21.4$ Hz). Anal. Calcd for $\text{C}_8\text{H}_5\text{ClF}_2\text{O}$: C, 50.42; H, 2.64. Found: C, 50.14; H, 2.59.

Current Data Parameters
 NAME 224662-41b
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060706
 Time 18.50
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 13020.833 Hz
 FIDRES 0.186682 Hz
 AQ 2.5166323 sec
 RG 256
 DN 38.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 DCREST 0.0000000 sec
 MCWPR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.00 usec
 PL1 -4.00 dB
 SFO1 500.1337700 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300000 MHz
 WDW nc
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 0.20

1D NMR plot parameters
 CX 23.50 cm
 CY 15.00 cm
 F1P 9.700 ppm
 F1 4876.27 Hz
 F2P -0.250 ppm
 F2 -126.03 Hz
 PPMCM 0.42653 ppm/cm
 HZCM 212.82126 Hz/cm

Current Data Parameters
 NAME 254662-42b
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060706
 Time 18.50
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 40389.880 Hz
 FIDRES 0.307687 Hz
 AQ 1.6862458 sec
 RG 8192
 DN 12.400 usec
 DE 6.00 usec
 TE 300.1 K
 D1 0.10000000 sec
 D11 0.03000000 sec
 DCREST 0.0000000 sec
 MCWPR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 126.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 11.00 dB
 PL12 11.00 dB
 SFO2 500.1305000 MHz

F2 - Processing parameters
 SI 65536
 SF 126.7677880 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 0.20

1D NMR plot parameters
 CX 23.50 cm
 CY 15.00 cm
 F1P 200.000 ppm
 F1 27586.71 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 9.58170 mm/cm

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Current Data Parameters
NAME      2504682-41b
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20160710
Time      16.53
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         27
DS         4
SWH        100000.000 Hz
FIDRES     0.7081000 Hz
AQ         0.40254100 sec
RG         64
SWH        8.000 usec
DE         8.00 usec
TE         300.2 K
D1         2.00000000 sec
d11        0.03000000 sec
d12        0.00000000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         8.00 usec
PL1        -4.00 dB
SFO1       470.3217731 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        19.00 dB
PL12       19.00 dB
SFO2       500.1365000 MHz

F2 - Processing parameters
SI         32768
SF         470.3282000 MHz
RG         64
SWH        1.00 Hz
DS         0
SC         1.00

SD NMR slit parameters
CX         23.50 cm
CY         10.00 cm
13CP       -44.300 gpm
13F1        -20180.000 Hz
13F2        -250.000 gpm
13F3        -190100.000 Hz
FPMCH      0.04347 gpm/cm

```

Compound 2c: Isolated as a solid in 49% yield from aryl precursor after crystallization: ^1H NMR δ 7.50 (quin, 1H, $J = 7.6$ Hz), 7.02 (t, 2H, $J = 8.3$ Hz), 4.56 (s, 2H), ppm; ^{13}C NMR δ 49.6 (t, $J = 3.1$ Hz), 112.3 (dd, $J = 21.5, 4.3$ Hz), 114.9 (t, $J = 18.5$ Hz), 133.8 (t, $J = 11.1$ Hz), 160.3 (dd, $J = 255.3, 6.8$ Hz), 188.6; ^{19}F NMR δ -111.5. Anal. Calcd for $\text{C}_8\text{H}_5\text{ClF}_2\text{O}$: C, 50.42; H, 2.64. Found: C, 49.71; H, 2.59.

Current Data Parameters
NAME 224682-41c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 200807
Time 18.14
INSTRUM spect
PROBHD 5 mm QNP 1H/19
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 13020.833 Hz
FIDRES 0.188682 Hz
AQ 2.8186323 sec
RG 528.1
DM 38.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -4.00 dB
SFO1 500.1333758 MHz

===== CHANNEL f2 =====
CPDPRG2 off
NUC2 off
PCPD2 100.00 usec
PL2 120.00 dB
PL12 120.00 dB
SFO2 500.1300000 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.20

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 8.750 ppm
F1 4876.27 Hz
F2P -0.250 ppm
F2 -125.03 Hz
PPMCM 0.42503 ppm/cm
HZCM 212.65126 Hz/cm

nmr500c h-1

2c

Current Data Parameters
NAME 254682-41c
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 200807
Time 18.20
INSTRUM spect
PROBHD 5 mm QNP 1H/19
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 40389.888 Hz
FIDRES 0.307887 Hz
AQ 1.8882458 sec
RG 8192
DM 32.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00000000 sec
RGRES 0.01000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 11.00 dB
PL12 11.00 dB
SFO2 500.1300000 MHz

F2 - Processing parameters
SI 65536
SF 125.77077880 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20

1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 27588.11 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.35110 ppm/cm

c-13 nmr500c

2c


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Current Data Parameters
NAME 204682-41c
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060707
Time 08:27
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4
DS 4
SWH 100000.000 Hz
FIDRES 0.700000 Hz
AQ 0.0004100 sec
RG 64
AQ 0.0000000 sec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -4.00 dB
SFO1 470.321731 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 19.00 dB
PL12 19.00 dB
SFO2 500.1365000 MHz

F2 - Processing parameters
SI 32768
SF 470.321731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

SD NMR plot parameters
CX 23.50 cm
CY 10.00 cm
FID -44.300 gpm
F1 -20000.000 Hz
F2 -20000.000 Hz
F3 -10000.000 Hz
FPMCH 0.04047 gpm/cm

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nmr500c f-19

Compound 2d: Isolated as a solid in 70% yield from aryl precursor after crystallization: ^1H NMR δ 7.64 (m, 1H, $J = 1.9$ Hz), 7.30 (m, 1H), 7.19 (td, 1H, $J = 9.5, 4.0$ Hz), 4.73 (d, 2H, $J = 3.0$ Hz), ppm; ^{13}C NMR δ 49.8 (d, $J = 12.3$ Hz), 117.0 (dd, $J = 25.8, 3.7$ Hz), 118.3 (dd, $J = 27.1, 8.0$ Hz), 122.5 (dd, $J = 24.6, 9.2$ Hz), 123.9 (dd, $J = 16.6, 6.8$ Hz), 157.9 (dd, $J = 250.4, 1.8$ Hz), 158.8 (dd, $J = 246.1, 1.8$ Hz), 188.1 (dd, $J = 4.9, 1.2$ Hz); ^{19}F NMR δ -114.5 (d, $J = 18.3$ Hz), -117.1 (d, $J = 18.3$ Hz). Anal. Calcd for $\text{C}_8\text{H}_5\text{ClF}_2\text{O}$: C, 50.42; H, 2.64. Found: C, 50.33; H, 2.55.

Current Data Parameters
 NAME 224662-411
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060726
 Time 18.08
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 13020.833 Hz
 FIDRES 0.186682 Hz
 AQ 2.5166323 sec
 RG 161.3
 DN 39.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 DCREST 0.0000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.00 usec
 PL1 -4.00 dB
 SFO1 500.1352700 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1350000 MHz
 WDW nc
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 0.20

1D NMR plot parameters
 CX 23.50 cm
 CY 10.00 cm
 F1P 9.700 ppm
 F1 4876.27 Hz
 F2P -0.250 ppm
 F2 -126.03 Hz
 PPMCM 0.42653 ppm/cm
 HZCM 212.82126 Hz/cm

nmr500c h-1

2d

Current Data Parameters
 NAME 254662-411
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060726
 Time 18.18
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 40389.880 Hz
 FIDRES 0.307687 Hz
 AQ 1.8892408 sec
 RG 8180
 DN 12.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 D11 0.03000000 sec
 DCREST 0.0000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 126.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2P2 80.00 usec
 PL2 11.00 dB
 PL12 11.00 dB
 SFO2 500.1350000 MHz

F2 - Processing parameters
 SI 65536
 SF 126.7677880 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 0.20

1D NMR plot parameters
 CX 23.50 cm
 CY 10.00 cm
 F1P 200.000 ppm
 F1 27086.71 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 9.58170 mm/cm

c-13 nmr500c

2d

```

Current Data Parameters
NAME      2016082-411
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20160720
Time      08:21
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30m
TD         32768
SOLVENT   CDCl3
NS         27
DS         4
SWH        100000.000 Hz
FIDRES     0.7694000 Hz
AQ          0.4024430 sec
RG          684
WDW         8.000 usec
SSB         0.000 usec
TE          300.2 K
D1          2.00000000 sec
d11         0.03000000 sec
d12         0.00000000 sec

===== CHANNEL f1 =====
NUC1       13C
P1          5.00 usec
PL1         -4.00 dB
SFO1       470.321731 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
P2          80.00 usec
PL2         18.00 dB
PL12        18.00 dB
SFO2       500.1360000 MHz

F2 - Processing parameters
SI          32768
SF          470.3283400 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.00

1D NMR plot parameters
CX          23.50 cm
CY          10.00 cm
F1          -44.300 ppm
F2          -20180.000 Hz
F3          -250.000 ppm
F4          -130100.00 Hz
RG          9.04347 um/cm

```

nmr500c f-19

2d

Compound 2e: Isolated as a solid in 65% yield from aryl precursor after crystallization: ^1H NMR δ 7.09 (td, 1H, $J = 9.3, 4.8$ Hz), 6.93 (td, 1H, $J = 9.3, 2.0$ Hz), 4.55 (s, 2H), 3.91 (s, 3H), ppm; ^{13}C NMR δ 49.5 (t, $J = 3.7$ Hz), 57.0, 111.2 (dd, $J = 23.4, 4.3$ Hz), 115.6 (dd, $J = 20.3, 16.0$ Hz), 116.8 (dd, $J = 4.2, 3.1$ Hz), 144.7 (dd, $J = 11.1, 3.7$ Hz), 149.6 (dd, $J = 256.6, 7.4$ Hz), 153.2 (dd, $J = 248.0, 5.5$ Hz), 188.6; ^{19}F NMR δ -123.0, -132.5,. Anal. Calcd for $\text{C}_9\text{H}_7\text{ClF}_2\text{O}_2$: C, 49.0; H, 3.2. Found: C, 48.91; H, 3.12.

Current Data Parameters
NAME 224662-41e
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060710
Time 18.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 13020.833 Hz
FIDRES 0.138682 Hz
AQ 2.5166323 sec
RG 161.3
DM 39.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
dPCREST 0.0000000 sec
dCMRFR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -4.00 dB
SFO1 500.1333700 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW nc
SSB 0
LB 0.00 Hz
GB 0
PC 0.20

1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 9.700 ppm
F1 4876.27 Hz
F2P -0.250 ppm
F2 -126.03 Hz
PPMCM 0.42653 ppm/cm
HZCM 212.82126 Hz/cm

Current Data Parameters
NAME 254662-41e
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060710
Time 18.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 40389.880 Hz
FIDRES 0.307807 Hz
AQ 1.6892458 sec
RG 8192
DM 12.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
dPCREST 0.0000000 sec
dCMRFR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 11.00 dB
PL12 11.00 dB
SFO2 500.1300000 MHz

F2 - Processing parameters
SI 65536
SF 125.7677880 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20

1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 200.000 ppm
F1 27586.71 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.36170 ppm/cm

```

Current Data Parameters
NAME 204682-41a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060710
Time 18:23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 4
DS 4
SWH 100000.000 Hz
FIDRES 0.7081000 Hz
AQ 0.4854100 sec
RG 64
AQ 0.0000000 sec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -4.00 dB
SFO1 470.321731 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 19.00 dB
PL12 19.00 dB
SFO2 500.1360000 MHz

F2 - Processing parameters
SI 32768
SF 470.321731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

SD NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1 44.000 ppm
F2 -2000.000 Hz
F3 -200.000 ppm
F4 -10000.000 Hz
F5 0.04047 ppm/cm

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Compound 2f: The above procedure was followed exactly; lithium halogen exchange occurs under the deprotonation conditions. Isolated as a solid in 60% yield from aryl precursor after crystallization: ^1H NMR δ 7.94 (t, 1H, $J = 1.8$ Hz), 7.85 (m, 1H), 7.60 (m, 1H), 7.47 (t, 1H, $J = 8.0$ Hz), 4.69 (s, 2H), ppm; ^{13}C NMR δ 45.7, 126.6, 128.6, 130.2, 133.9, 135.3, 135.7, 190.0. Anal. Calcd for $\text{C}_8\text{H}_6\text{Cl}_2\text{O}$: C, 50.83; H, 3.2. Found: C, 50.72; H, 3.14.

Current Data Parameters
 NAME 224662-41
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050706
 Time 18.04
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TO 69936
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 13020.833 Hz
 FIDRES 0.186682 Hz
 AQ 2.5166323 sec
 RG 161.3
 DN 39.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 MCPEST 0.0000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.00 usec
 PL1 -4.00 dB
 SFO1 500.135739 MHz

F2 - Processing parameters
 SI 32768
 SF 500.135000 MHz
 WDW nc
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 0.20

1D NMR plot parameters
 CX 23.50 cm
 CY 15.00 cm
 F1P 9.790 ppm
 F1 4876.27 Hz
 F2P -0.250 ppm
 F2 -126.03 Hz
 PPWM 0.42553 ppm/cm
 HZCM 212.82126 Hz/cm

Current Data Parameters
 NAME 224662-41
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060708
 Time 18.03
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TO 131070
 SOLVENT CDCl3
 NS 4
 DS 4
 SWH 40389.880 Hz
 FIDRES 0.307687 Hz
 AQ 1.6982458 sec
 RG 8180
 DN 12.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 D11 0.03000000 sec
 MCPEST 0.0000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 126.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 11.00 dB
 PL12 11.00 dB
 SFO2 500.135000 MHz

F2 - Processing parameters
 SI 65536
 SF 126.7677880 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 0.20

1D NMR plot parameters
 CX 23.50 cm
 CY 10.00 cm
 F1P 200.000 ppm
 F1 27586.71 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPWM 9.58170 mm/cm

c-13 nmr500c

Compound 2g: Isolated as a solid in 65% yield from aryl precursor after crystallization: ^1H NMR δ 7.74 (d, 1H, $J = 8.0$ Hz), 7.66 (d, 1H, $J = 0.8$ Hz), 7.60 (m, 1H), 7.53 (ddd, 1H, $J = 8.4, 7.2, 1.2$ Hz), 7.35 (m, 1H), 4.71 (s, 2H) ppm; ^{13}C NMR δ 45.4, 112.4, 114.3, 123.5, 124.2, 126.7, 128.9, 150.3, 155.7, 182.1. Anal. Calcd for $\text{C}_{10}\text{H}_7\text{ClO}_2$: C, 61.72; H, 3.63. Found: C, 61.35; H, 3.32.

```

Current Data Parameters
NAME      C295-405-19a
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20060907
Time      17.20
INSTRUM   spect
PROBHD    5 mm QNP
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         32
DS         2
SWH        6561.680 Hz
FIDRES     0.200247 Hz
AQ         2.498716 sec
RG          256
DN         76.200 usec
DE         6.00 usec
TE         300.2 K
D1         0.10000000 sec
dCHEST     0.00000000 sec
dCMRPR     0.01500000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         10.00 usec
PL1        -5.00 dB
SFO1       400.132007 MHz

F2 - Processing parameters
SI          16384
SF          400.1300056 MHz
WDW         no
SSB         0
LB          0.00 Hz
GB          0
PC          1.00

ID NMR plot parameters
CX          23.50 cm
CY          10.00 cm
*F1         5.750 ppm
F1          3901.27 Hz
F2P         -0.250 ppm
F2          -100.03 Hz
PPMCM       0.42553 ppm/cm
HZCM        170.26808 Hz/cm

```

nmr400c h1

2g

```

Current Data Parameters
NAME: 080405-10a
EXPNO: 2
PROCNO: 1

F2 - Acquisition Parameters
Date_ : 20060811
Time : 22:47
INSTRUM : spect
PROBHD : 5 mm QNP
PULPROG : zgpg
TD : 65536
SOLVENT : CDCl3
NS : 4317
DS : 0
SWH : 20040.710 Hz
FIDRES : 0.400400 Hz
AQ : 1.2486310 sec
RG : 8192
AQ : 19.000 usec
DE : 8.00 usec
TE : 300.2 K
SI : 0.1000000 sec
SFO : 0.0300000 sec
RG : 0.0000000 sec
RG : 0.0000000 sec
RG : 0.0100000 sec

===== CHANNEL f1 =====
NUC1 : 13C
P1 : 2.00 usec
PL1 : 0.00 dB
SFO1 : 100.627764 MHz

===== CHANNEL f2 =====
CPDPRG2 : waltz16
NUC2 : 1H
PCPD2 : 88.00 usec
PL2 : 120.00 dB
PL12 : 18.00 dB
SFO2 : 400.136077 MHz

F2 - Processing parameters
SI : 32768
SF : 100.627766 MHz
WDW : EM
SSB : 0
LB : 1.00 Hz
GB : 0
PC : 1.40

1D NMR plot parameters
CX : 20.00 cm
CY : 10.00 cm
P1P : 200.000 ppm
F1 : 200.34.01 Hz
F2P : 1.000 ppm
F2 : 0.00 Hz
PPMCH : 9.36170 nm/cm

```

c-13 nmr400c

2g

Compound 2h: Isolated as a solid in 71% yield from aryl precursor after crystallization: ^1H NMR δ 7.60 (dd, 1H, J = 8.8, 6.8 Hz), 7.28 (dd, 1H, J = 8.8, 1.6 Hz), 4.69 (d, 2H, J = 2.8 Hz), 4.00 (d, 3H, J = 1.2 Hz) ppm; ^{13}C NMR δ 49.6 (d, J = 11.2 Hz), 61.7 (d, J = 4.0 Hz), 122.9 (d, J = 12.8 Hz), 124.9 (d, J = 4.0 Hz), 125.9 (d, J = 3.2 Hz), 134.9 (d, J = 4.0 Hz), 145.0 (d, J = 15.2 Hz), 155.8 (d, J = 257.9 Hz), 188.1 (d, J = 4.8 Hz); ^{19}F NMR δ -124.8. Anal. Calcd for $\text{C}_9\text{H}_7\text{Cl}_2\text{FO}_2$: C, 45.60; H, 2.98. Found: C, 45.57; H, 2.80.

Current Data Parameters
 NAME 025645-18a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060606
 Time 15.17
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 6561.680 Hz
 FIDRES 0.200247 Hz
 AQ 2.4989716 sec
 RG 481
 DM 75.200 usec
 DE 6.00 usec
 TE 300.2 K
 D1 0.10000000 sec
 DCREST 0.0000000 sec
 MCNMR 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 -5.00 dB
 SFO1 400.1362007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300566 MHz
 WDW nc
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 23.50 cm
 CY 15.00 cm
 F1P 9.700 ppm
 F1 3001.27 Hz
 F2P -0.250 ppm
 F2 -100.03 Hz
 PPMCM 0.42553 ppm/cm
 HZCM 170.25508 Hz/cm

Current Data Parameters
 NAME 025645-18a
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060606
 Time 17.48
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 6
 DS 0
 SWH 25246.719 Hz
 FIDRES 0.400463 Hz
 AQ 1.2498018 sec
 RG 8192
 DM 15.000 usec
 DE 5.00 usec
 TE 300.2 K
 D1 0.10000000 sec
 D11 0.03000000 sec
 DCREST 0.0000000 sec
 MCNMR 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 100.6257864 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 190.00 dB
 PL12 18.00 dB
 SFO2 400.1300007 MHz

F2 - Processing parameters
 SI 36768
 SF 100.6197786 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 23.50 cm
 CY 15.00 cm
 F1P 200.000 ppm
 F1 20254.51 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 9.55170 ppm/cm

```

Current Data Parameters
NAME 080545-19a
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060808
Time 16.20
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4
DS 4
SWH 11994.850 Hz
FIDRES 0.00070 Hz
AQ 0.000400 sec
RG 256
AQ 4.400 usec
DE 0.30 usec
TE 300.2 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

===== CHANNEL f1 =====
NUC1 19F
P1 0.30 usec
PL1 -1.00 dB
SFO1 376.480133 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 0.00 usec
PL2 0.00 dB
PL12 18.00 dB
SFO2 400.130007 MHz

F2 - Processing parameters
SI 32768
SF 376.480133 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

DQ NMR plot parameters
CX 25.00 cm
CY 10.00 cm
FID 0.000 gsm
F1 0.00 Hz
F2 -300.000 gsm
F3 -11994.850 Hz
RG 12.78586 gsm/cm

```

nmr400c h-1 decoupled f-19

2h

Compound 3b: Isolated as a solid in 83% yield from aryl precursor after SiO₂ chromatography from 3:1 (Hex:EtOAc): ¹H NMR δ 7.73 (m, 1H), 7.44 (m, 1H), 7.25 (m, 3H), 6.87 (m, 2H), 5.20 (d, 2H, *J* = 3.2 Hz) ppm; ¹³C NMR δ 73.6 (d, *J* = 11.2 Hz), 116.0, 122.3 (dd, *J* = 17.6, 1.6 Hz), 124.6 (d, *J* = 11.2 Hz), 124.9 (dd, *J* = 6.4, 4.0 Hz), 125.2 (dd, *J* = 3.6, 1.2 Hz), 126.6, 129.4, 150.4 (dd, *J* = 255.9, 14.0 Hz), 150.7 (dd, *J* = 251.1, 14.0 Hz), 156.4, 191.4 (dd, *J* = 4.8, 2.4 Hz); ¹⁹F NMR δ -134.8 (d, *J* = 22.4 Hz), -137.2 (d, *J* = 22.4 Hz). Anal. Calcd for C₁₄H₉ClF₂O₂: C, 59.49; H, 3.21. Found: C, 59.10; H, 3.04.

Current Data Parameters
NAME 025645-1Bf
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060113
Time 11.46
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4989716 sec
RG 256
DM 75.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
dPCREST 0.0000000 sec
dCMR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SFO1 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW nc
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 9.700 ppm
F1 3001.27 Hz
F2P -0.250 ppm
F2 -100.03 Hz
PPMCM 0.42553 ppm/cm
HZCM 170.25508 Hz/cm

Current Data Parameters
NAME 025645-1Bf
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060114
Time 1.49
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 25246.719 Hz
FIDRES 0.400468 Hz
AQ 1.24989716 sec
RG 512
DM 15.000 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
dPCREST 0.0000000 sec
dCMR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6207854 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2P2 95.00 usec
PL2 190.00 dB
PL12 18.00 dB
SFO2 400.130007 MHz

F2 - Processing parameters
SI 32768
SF 100.6197786 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 200.000 ppm
F1 20034.51 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.55170 mm/cm

```

Current Data Parameters
NAME  CDE445-1H
EXPNO  3
PROCNO  1

F2 - Acquisition Parameters
Date_  20090313
Time  11:51
INSTRUM  spect
PROBHD  5 mm QNP 1H/1
PULPROG  zgpg30
TD  32768
SOLVENT  CDCl3
NS  4
DS  4
SWH  119994.760 Hz
FIDRES  0.00070 Hz
AQ  0.00000000 sec
RG  256
SN  4.40 uSBC
DE  0.30 uSBC
TE  300.2 K
D1  0.10000000 sec
d11  0.03000000 sec
s12  0.00000000 sec

===== CHANNEL f1 =====
NUC1  1H
P1  0.30 uSBC
PL1  -1.00 dB
SFO1  376.4460000 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2  13C
P2  80.00 uSBC
PL2  190.00 dB
PL12  18.00 dB
SFO2  400.1300007 MHz

F2 - Processing parameters
SI  8192
SF  376.4460000 MHz
WDW  EM
SSB  0
LB  0.50 Hz
GB  0
PC  1.00

SD NMR plot parameters
CX  25.00 cm
CY  10.00 cm
FID  0.000 gsm
F1  0.00 Hz
F2  -100.000 gsm
F3  -1000.00 Hz
RG  12.78586 gsm/cm

```

nmr400c h-1 decoupled f-19

3b

Compound 3c: Isolated as a solid in 72% yield from aryl precursor after SiO₂ chromatography from 3:1 (Hex:EtOAc): ¹H NMR δ 7.47 (m, 1H), 7.24 (m, 2H), 6.99 (m, 2H), 6.82 (m, 2H), 5.00 (s, 2H) ppm; ¹³C NMR δ 73.8 (t, *J* = 3.2 Hz), 112.1 (m), 114.9 (t, *J* = 19.2 Hz), 115.9, 126.7, 129.4, 133.5 (t, *J* = 10.4 Hz), 156.3 160.4 (dd, *J* = 255.5, 7.2 Hz), 192.7; ¹⁹F NMR δ -111.8. Anal. Calcd for C₁₄H₉ClF₂O₂: C, 59.49; H, 3.21. Found: C, 59.38; H, 3.06.

Current Data Parameters
NAME 025645-10e
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060112
Time 14.21
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4989716 sec
RG 512
DM 75.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
DCREST 0.0000000 sec
DCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SFO1 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW nc
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 9.700 ppm
F1 3001.27 Hz
F2P -0.250 ppm
F2 -100.03 Hz
PPMCM 0.42553 ppm/cm
HZCM 170.25508 Hz/cm

Current Data Parameters
NAME 025645-10e
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060112
Time 1.30
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 25246.719 Hz
FIDRES 0.400468 Hz
AQ 1.24989716 sec
RG 512
DM 15.000 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
D11 0.03000000 sec
DCREST 0.0000000 sec
DCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6257854 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.130007 MHz

F2 - Processing parameters
SI 32768
SF 100.6197786 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 20.00 cm
F1P 200.000 ppm
F1 20034.51 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.55170 ppm/cm

```

Current Data Parameters
NAME 082545-19
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090910
Time 14:24
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 200
DS 4
SWH 11994.750 Hz
FIDRES 0.00070 Hz
AQ 0.000400 sec
RG 256
AQ 4.40 usec
DE 0.30 usec
TE 300.2 K
D1 0.1000000 sec
d11 0.0300000 sec
d12 0.0000000 sec

===== CHANNEL f1 =====
NUC1 19F
P1 0.30 usec
PL1 -1.00 dB
SFO1 376.4401942 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 0.00 usec
PL2 150.00 dB
PL12 18.00 dB
SFO2 400.1300077 MHz

F2 - Processing parameters
SI 32768
SF 376.4401942 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

D0 NMR plot parameters
CX 25.00 cm
CY 10.00 cm
FID 0.000 gsm
FI 0.00 Hz
RG 380.000 gsm
F2 -11994.750 Hz
RG 12.76186 gsm/cm

```

nmr400c h-1 decoupled f-19

Compound 3g: Isolated as a solid in 60% yield from aryl precursor after SiO₂ chromatography from 3:1 (Hex:EtOAc): ¹H NMR δ 7.71 (d, 1H, *J* = 8.0 Hz), 7.65 (s, 1H), 7.57 (d, 1H, *J* = 8.0 Hz), 7.49 (m, 1H), 7.44-7.30 (m, 6H), 4.74 (s, 2H), 4.73 (s, 2H) ppm; ¹³C NMR δ 72.6, 73.5, 112.3, 113.8, 123.4, 123.9, 126.7, 128.0 (3C), 128.5 (3C), 137.0, 150.7, 155.4, 187.5. Anal. Calcd for C₁₇H₁₄O₃: C, 76.68; H, 5.30. Found: C, 76.47; H, 5.06.

Current Data Parameters
NAME 0255455-18b
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060907
Time 17.39
INSTRUM spect
PROBHD 5 mm QNP
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 114
DM 75.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
MCPRST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SFO1 400.1362007 MHz

F2 - Processing parameters
SI 16384
SF 400.130056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 9.750 ppm
F1 3901.27 Hz
F2P -0.250 ppm
F2 -100.03 Hz
PPHCH 0.42553 ppm/cm
HZCN 170.26908 Hz/cm

Current Data Parameters
NAME 0255455-18b
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060911
Time 19.31
INSTRUM spect
PROBHD 5 mm QNP
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 640
DS 4
SWH 28246.719 Hz
FIDRES 0.400463 Hz
AQ 1.2496916 sec
RG 8192
DM 15.000 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
D11 0.03000000 sec
MCPRST 0.00000000 sec
MCWRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 0.00 usec
PL1 0.00 dB
SFO1 100.6297864 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 19.00 dB
PL12 18.00 dB
SFO2 400.1300007 MHz

F2 - Processing parameters
SI 32768
SF 100.6197786 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 200.000 ppm
F1 20254.31 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHCH 9.55170 mm/cm

Compound 3h: Isolated as a solid in 70% yield from aryl precursor after SiO₂ chromatography from 3:1 (Hex:EtOAc): ¹H NMR δ 7.61 (dd, 1H, *J* = 8.8, 6.8 Hz), 7.42-7.37 (m, 4H), 7.33 (m, 1H), 7.26 (dd, 1H, *J* = 8.3, 1.6 Hz), 4.71 (s, 2H), 4.68 (d, 2H, *J* = 3.6 Hz), 3.98 (d, 3H, *J* = 1.2 Hz) ppm; ¹³C NMR δ 61.7 (d, *J* = 4.8 Hz), 73.4, 75.6 (d, *J* = 10.4 Hz), 123.4 (d, *J* = 14.4 Hz), 124.4 (d, *J* = 4.0 Hz), 125.7 (d, *J* = 3.2 Hz), 127.9 (3C), 128.4, 134.2 (d, *J* = 4.0 Hz), 137.1, 144.8 (d, *J* = 14.4 Hz), 156.0 (d, *J* = 257.1 Hz), 193.6 (d, *J* = 5.6 Hz); ¹⁹F NMR δ -124.6. Anal. Calcd for C₁₆H₁₄ClFO₃: C, 62.25; H, 4.57. Found: C, 62.10; H, 4.35.

```

Current Data Parameters
NAME      224662-41g3
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20061205
Time      15.26
INSTRUM   spect
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD        32768
SOLVENT   CDCl3
NS         32
DS         2
SWH        6561.680 Hz
FIDRES     0.200247 Hz
AQ         2.4969716 sec
RG         181
DM         75.200 usec
DE         6.00 usec
TE         300.2 K
D1         0.10000000 sec
dCREST    0.00000000 sec
dCMR      0.01500000 sec

***** CHANNEL f1 *****
NUC1       1H
P1         10.00 usec
PL1        -5.00 dB
SF01       400.1322007 MHz

F2 - Processing parameters
SI         16384
SF         400.1300056 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

ID NMR plot parameters
CX         23.50 cm
CY         15.00 cm
F1P        9.750 ppm
F1         3901.27 Hz
F2P        -0.250 ppm
F2         -100.33 Hz
PPHQM      0.42953 ppm/cm
HZQM       170.26808 Hz/cm

```

Current Data Parameters
 NAME 224682-44q3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080808
 Time 1:02
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 0
 DS 0
 SWH 200.40710 Hz
 FIDRES 0.400493 Hz
 AQ 1.2486510 sec
 RG 6192
 IN 10.000 usec
 DE 5.00 usec
 TE 300.2 K
 D1 0.10000000 sec
 d11 0.03000000 sec
 ACQRES 0.0000000 sec
 FWHM 0.0100000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.50 usec
 PL1 0.00 dB
 SFO1 100.627766 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1363007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.627766 MHz
 CH 1H
 SSF 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 FIDP 200.000 ppm
 F1 200.000 Hz
 F2 400.000 Hz
 F3 0.00 Hz
 FWH 9.36170 nm/cm

Current Data Parameters
 NAME 224682-44q3
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080808
 Time 10:36
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 4
 DS 4
 SWH 1120.04282 Hz
 FIDRES 0.186070 Hz
 AQ 0.380258 sec
 RG 1824.6
 IN 4.400 usec
 DE 6.30 usec
 TE 300.2 K
 D1 0.10000000 sec
 d11 0.03000000 sec
 d12 0.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 5.30 usec
 PL1 -1.00 dB
 SFO1 376.4600000 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 13C
 P2 80.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1363007 MHz

F2 - Processing parameters
 SI 65536
 SF 376.4600000 MHz
 CH 1H
 SSF 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 FIDP 0.000 ppm
 F1 0.00 Hz
 F2 -300.000 ppm
 F3 -1120.04282 Hz
 FWH 12.76596 nm/cm

Compound 3i: Isolated as a solid in x % yield from aryl precursor after SiO₂ chromatography from 3:1 (Hex:EtOAc): ¹H NMR δ 7.34 (m, 3H), 7.24 (m, 2H), 6.89 (m, 2H), 5.02 (s, 2H) ppm; ¹³C NMR δ 72.9, 116.3, 126.8, 128.1, 129.4, 131.2, 131.4, 136.3, 156.3, 197.1; Anal. Calcd for C₁₄H₉Cl₃O₂: C, 53.28; H, 2.87. Found: C, 53.18; H, 2.59.

```

Current Data Parameters
NAME      C295-405-18c
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20060908
Time      10.33
INSTRUM   spect
PROBHD    5 mm QNP
PULPROG   zgpg30
TD         32768
SOLVENT    CDCl3
NS         32
DS         2
SWH         6561.680 Hz
FIDRES     0.200247 Hz
AQ         2.4986716 sec
RG         101.6
DN         76.200 usec
DE         6.00 usec
TE         300.2 K
D1         0.10000000 sec
dCHEST     0.00000000 sec
dCMFR      0.01500000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         10.00 usec
PL1        -5.00 dB
SFO1       400.132007 MHz

F2 - Processing parameters
SI         16384
SF         400.130006 MHz
WDW         no
SSB         0
LB         0.00 Hz
GB         0
PC         1.00

ID NMR plot parameters
CX         23.50 cm
CY         18.00 cm
*F1        5.750 ppm
F1         3901.27 Hz
F2P        -0.250 ppm
F2         -100.03 Hz
PPMCM      0.42553 ppm/cm
HZCM       170.26800 Hz/cm

```

Current Data Parameters
NAME CDE445-16
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090808
Time 1:48
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 26000
DS 0
SWH 20040.710 Hz
FIDRES 0.400493 Hz
AQ 1.24860100 sec
RG 8192
DM 32.000 usec
DE 8.00 usec
TE 300.2 K
SI 0.10000000 sec
SFO 0.03000000 sec
WDWESS 0.00000000 sec
WDW 0.01000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.627766 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 88.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.146007 MHz

F2 - Processing parameters
SI 32768
SF 100.627766 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FID 200.000 ppm
F1 200.000 Hz
F2 0.000 ppm
F3 0.00 Hz
PPMCH 9.36170 ppm/cm

c-13 nmr400c