

Supporting Information File 2

for

Intramolecular carbolithiation of *N*-allyl-ynamides: an efficient entry to 1,4-dihydropyridines and pyridines – application to a formal synthesis of sarizotan

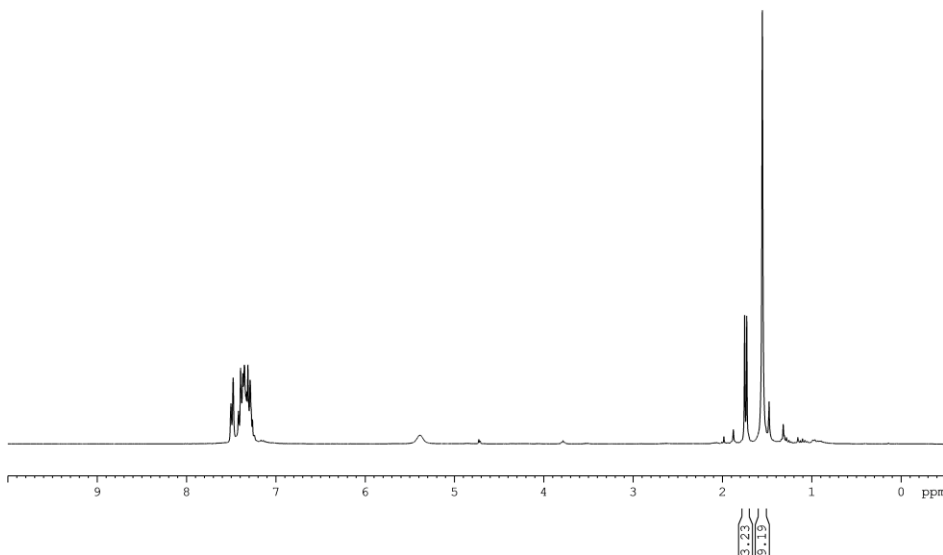
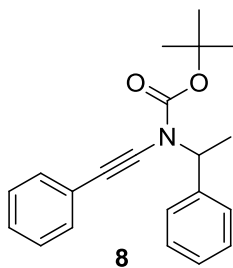
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* Corresponding author

Copies of ¹H and ¹³C NMR spectra for new compounds



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Current Data Parameters
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EXPNO     2
PROCNO    1

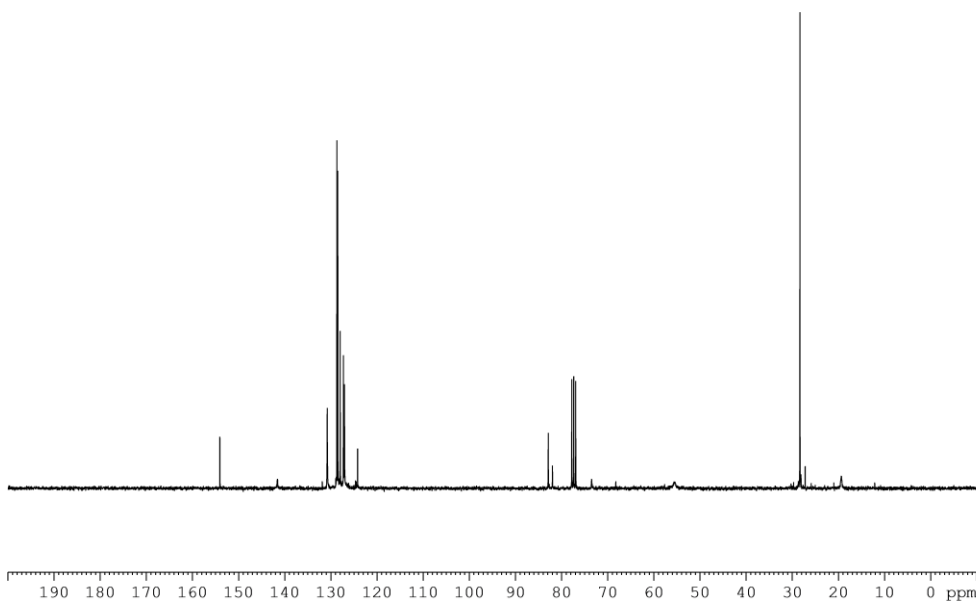
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SF         300.1300016 MHz
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1D NMR plot parameters
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F1         3001.30 Hz
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HZCM       157.56825 Hz/cm

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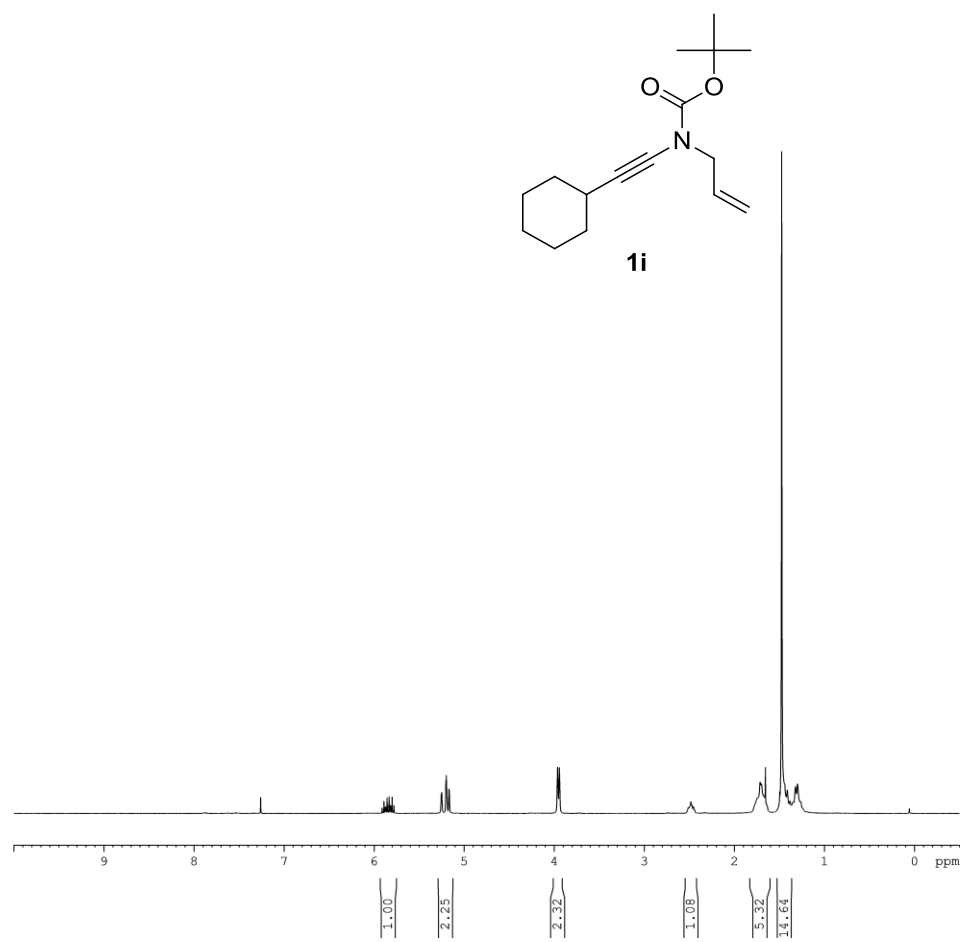
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SOLVENT   CDC13
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RG         2048
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DE         10.00 usec
TE         294.8 K
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NUC1       13C
P1         8.20 usec
PL1        -6.00 dB
SFO1       75.4756731 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        0.00 dB
PL12       21.54 dB
PL13       21.54 dB
SFO2       300.1312005 MHz

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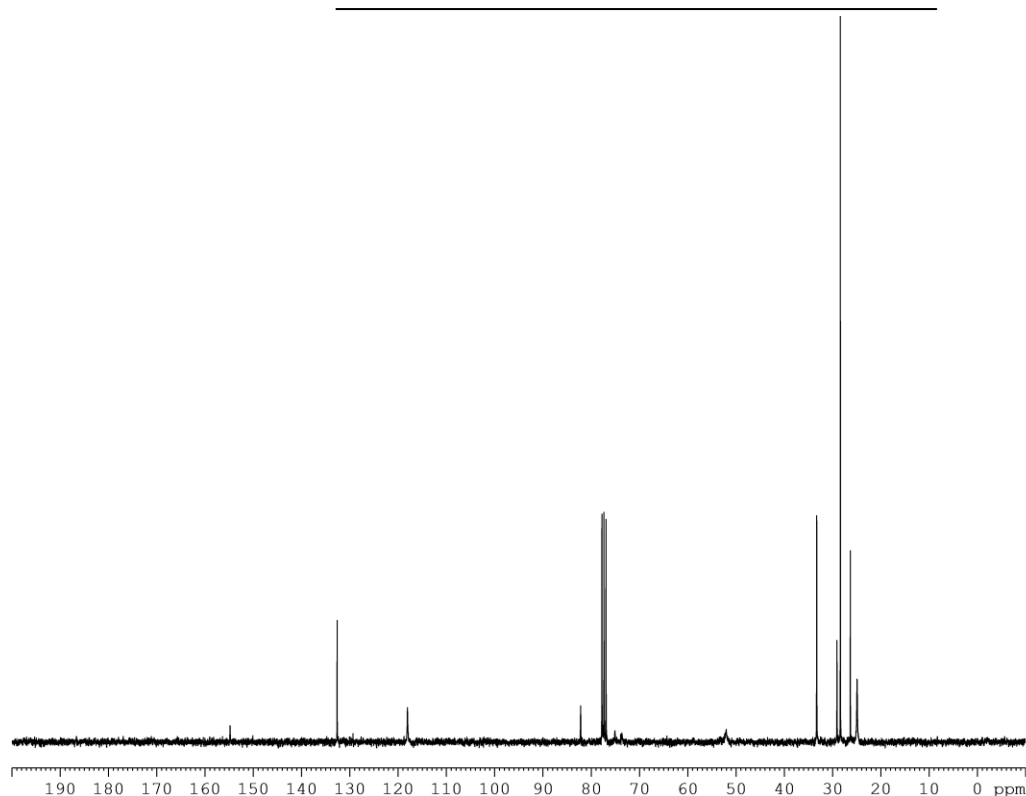
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 TD 32768
 SOLVENT C6D6
 NS 16
 DS 0
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 FIDRES 0.192661 Hz
 AQ 2.5952756 sec
 RG 80.6
 DW 79.200 usec
 DE 6.50 usec
 TE 293.8 K
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===== CHANNEL f1 =====
 NUC1 1H
 P1 6.20 usec
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 SFO1 300.1328512 MHz

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1D NMR plot parameters
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 CY 149.34 cm
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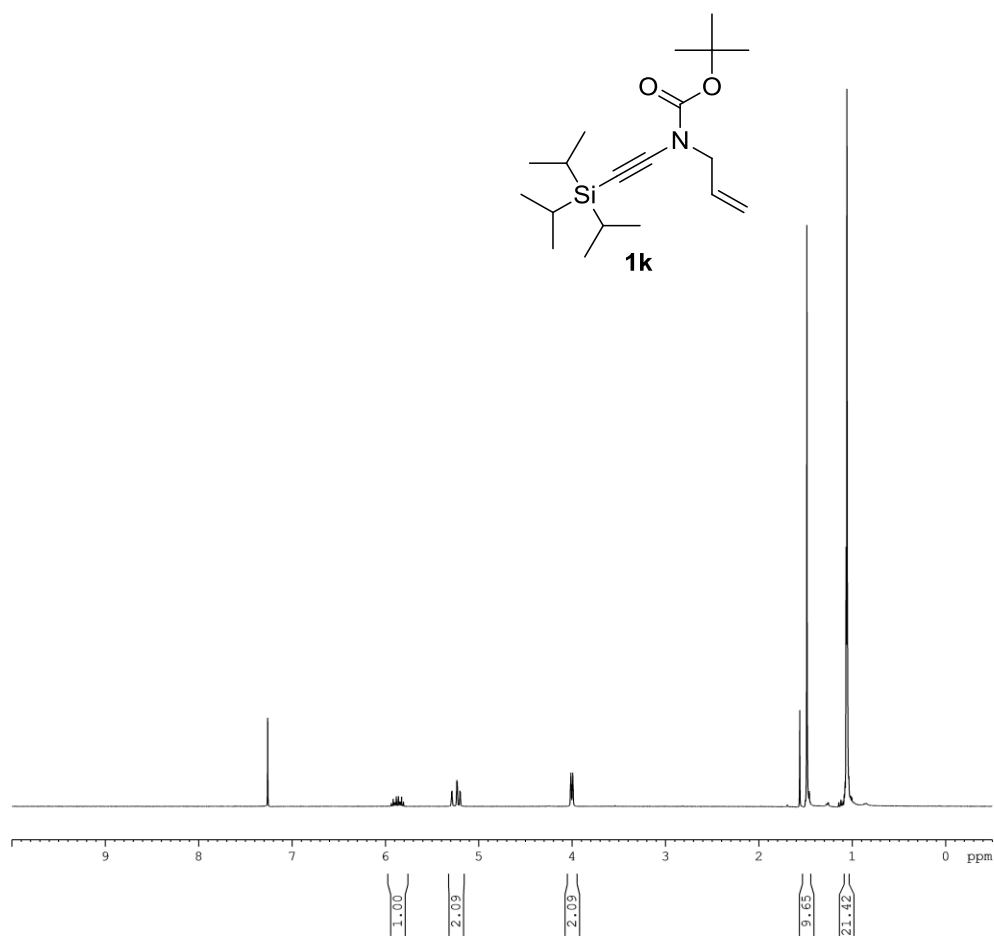
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 SOLVENT C6D6
 NS 1024
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 1625.5
 DW 27.800 usec
 DE 10.00 usec
 TE 294.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 -6.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 22.00 dB
 PL13 22.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
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 LB 1.00 Hz
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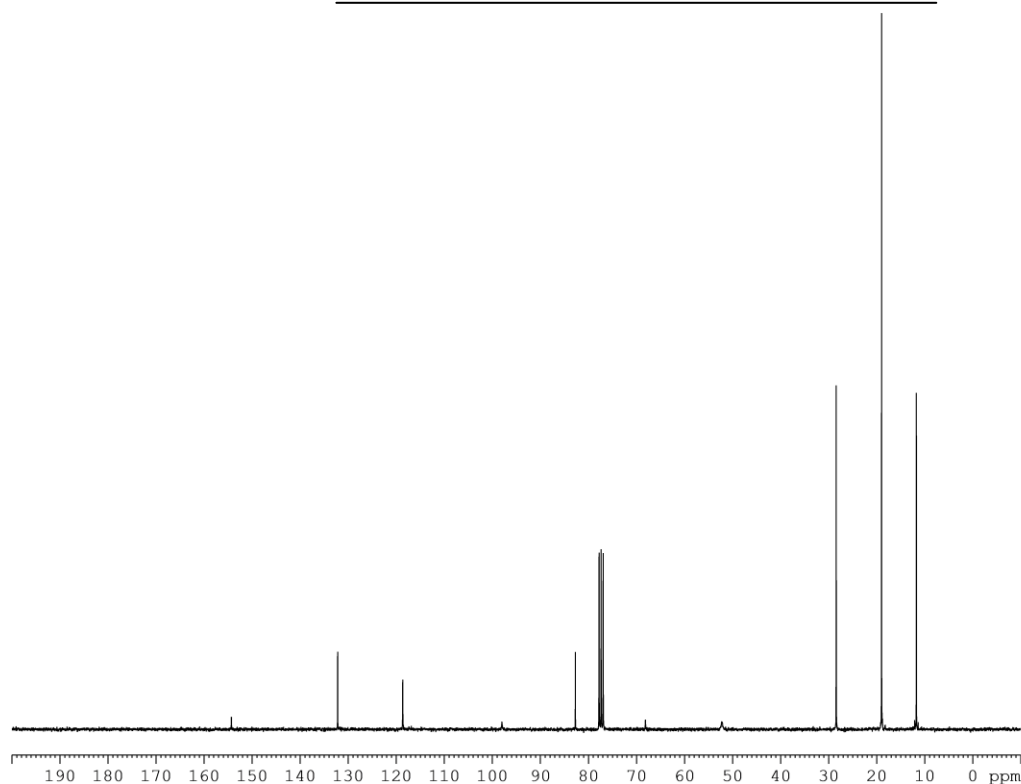


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PROCNO 1

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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
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SWH 6313.131 Hz
FIDRES 0.192661 Hz
AQ 2.5952756 sec
RG 456.1
DW 79.200 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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NUC1 1H
P1 6.20 usec
PL1 0.00 dB
SFO1 300.1328512 MHz

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LB 0.10 Hz
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PC 1.00



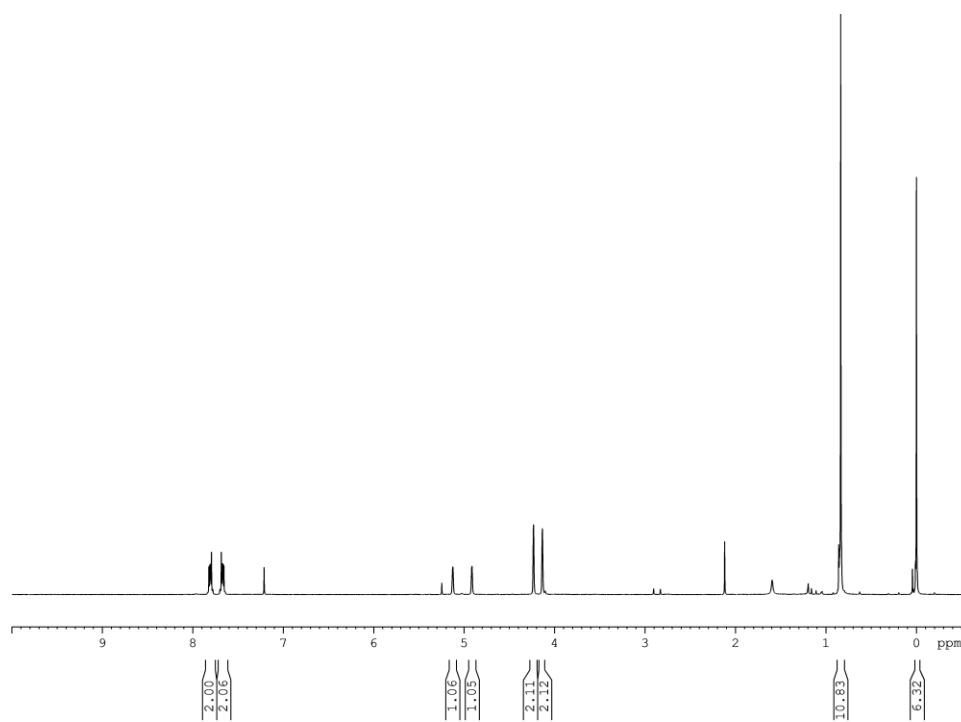
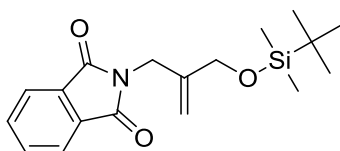
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NS 2000
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FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 10.00 usec
TE 297.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999999 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 -6.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
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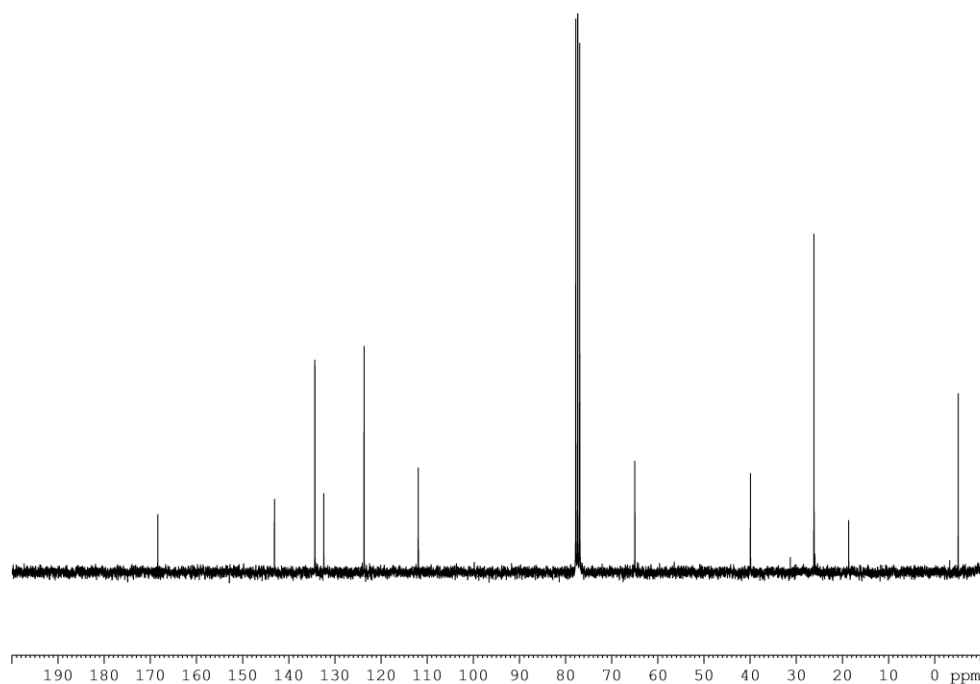


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EXPNO 10
PROCNO 1

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PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4194.631 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 287.4
DW 119.200 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
SFO1 300.1319508 MHz

F2 - Processing parameters
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SSB 0
LB 0.30 Hz
GB 0
PC 1.00



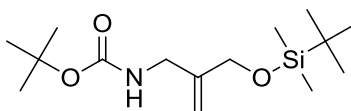
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EXPNO 12
PROCNO 1

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SOLVENT CDCl3
NS 1024
DS 4
SWH 18115.941 Hz
FIDRES 0.532855 Hz
AQ 0.9044468 sec
RG 2048
DW 27.600 usec
DE 10.00 usec
TE 292.4 K
D1 2.00000000 sec
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DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

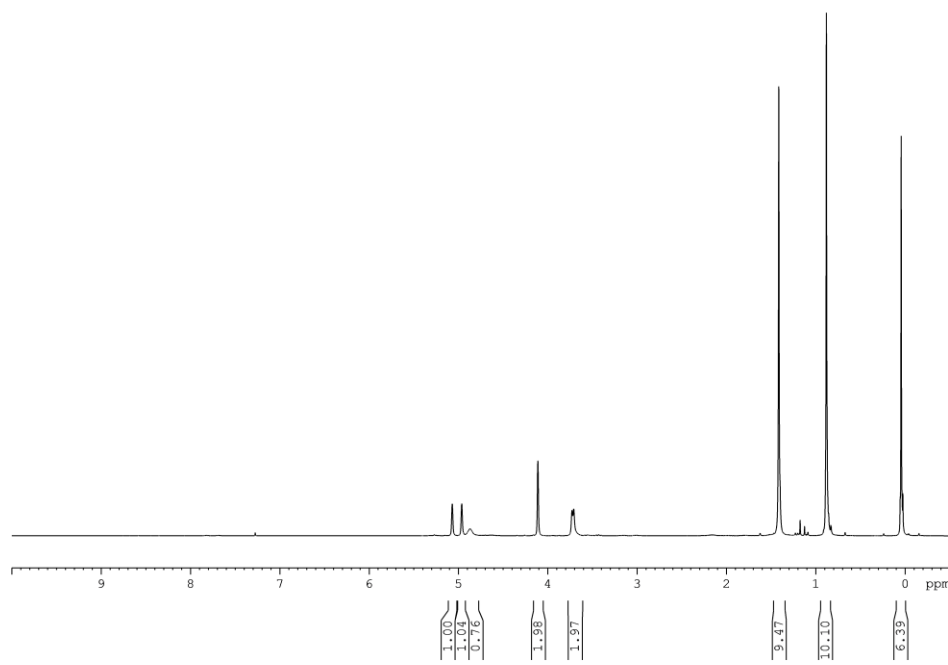
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PL1 -6.00 dB
SFO1 75.4756731 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 21.54 dB
PL13 21.54 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



13



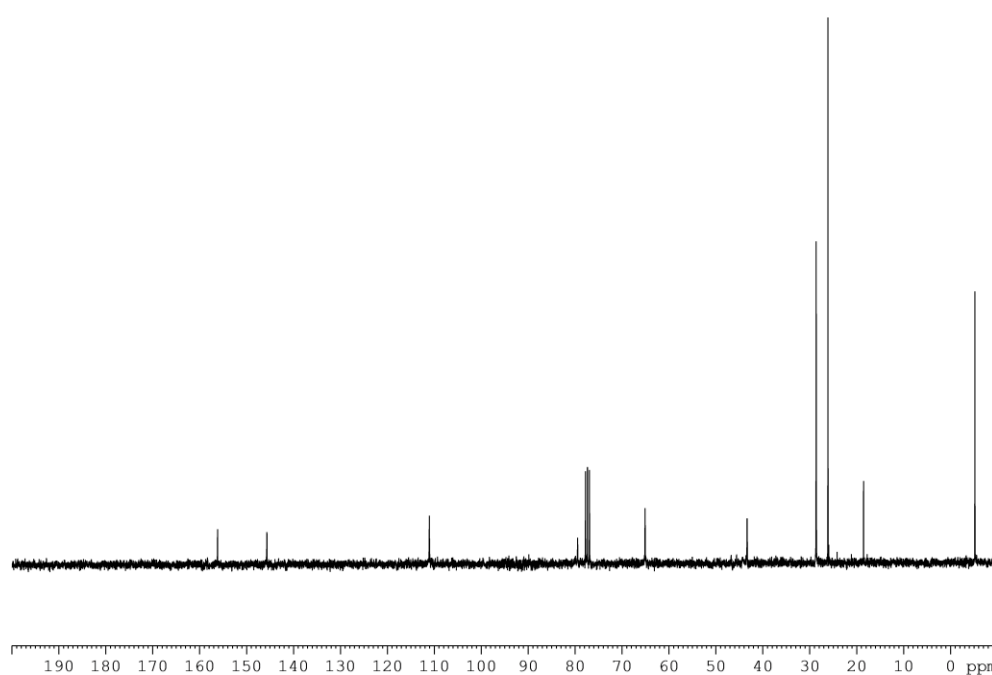
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EXPNO 1
PROCNO 1

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PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4194.631 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 35.9
DW 119.200 usec
DE 6.50 usec
TE 293.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
SFO1 300.1319508 MHz

F2 - Processing parameters
SI 32768
SF 300.1300016 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
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EXPNO 2
PROCNO 1

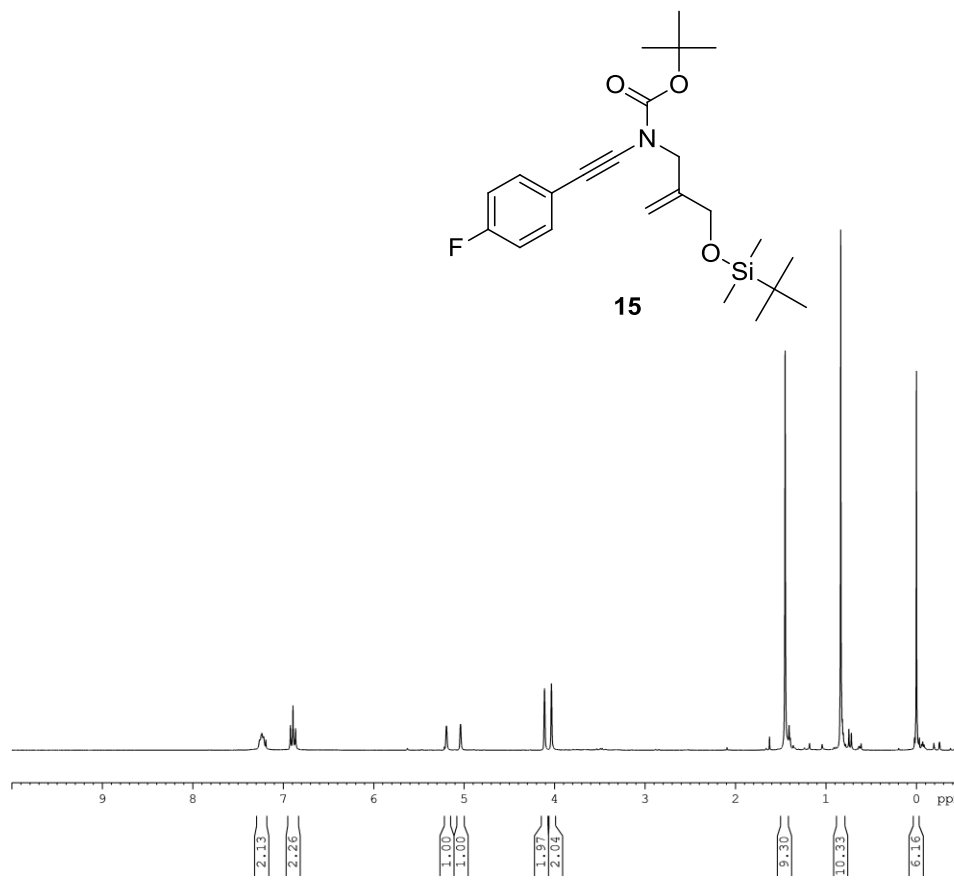
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TD 32768
SOLVENT CDCl3
NS 55
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 2048
DW 27.600 usec
DE 10.00 usec
TE 293.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 -6.00 dB
SFO1 75.4756731 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 21.54 dB
PL13 21.54 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 9.41 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
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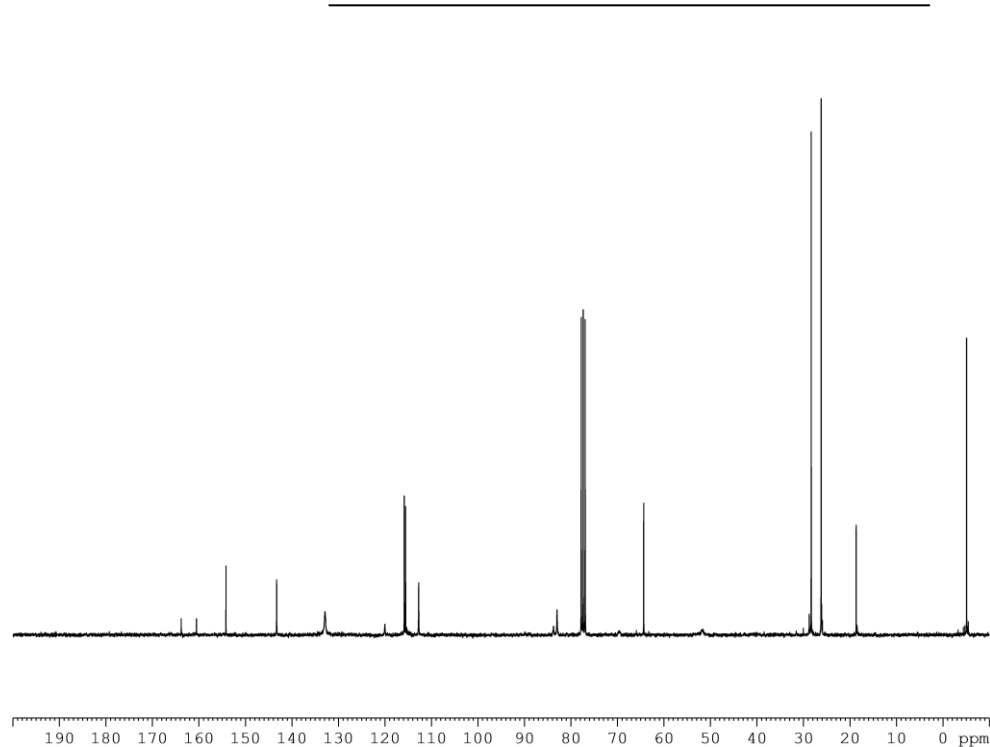


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PROCNO 1

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PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4194.631 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 64
DW 119.200 usec
DE 6.50 usec
TE 292.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
SFO1 300.1319508 MHz

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PC 1.00



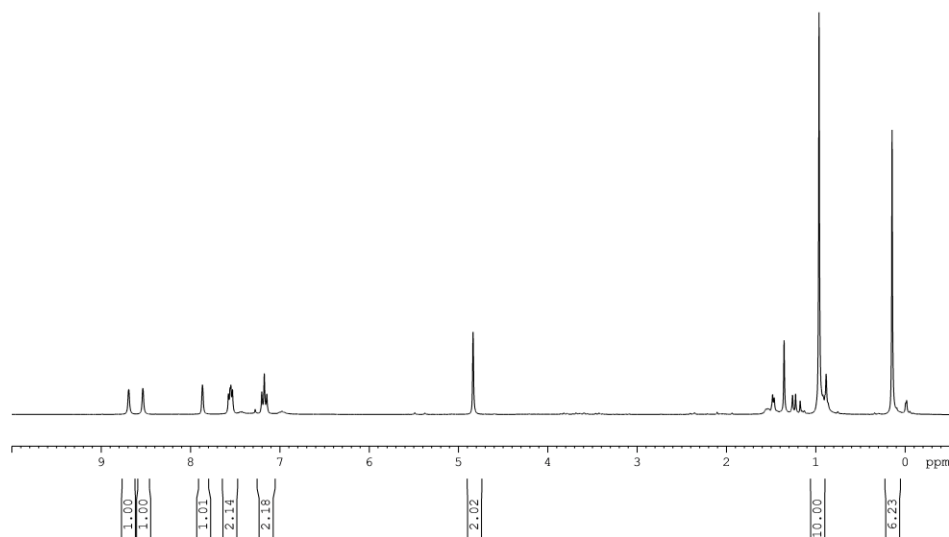
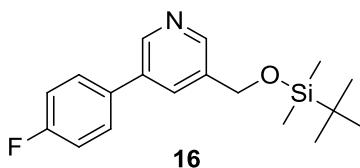
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TD 32768
SOLVENT CDCl3
NS 2000
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 2048
DW 27.600 usec
DE 10.00 usec
TE 292.1 K
D1 4.00000000 sec
d11 0.03000000 sec
DELTA 3.90000010 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 -6.00 dB
SFO1 75.4756731 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 21.54 dB
PL13 21.54 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



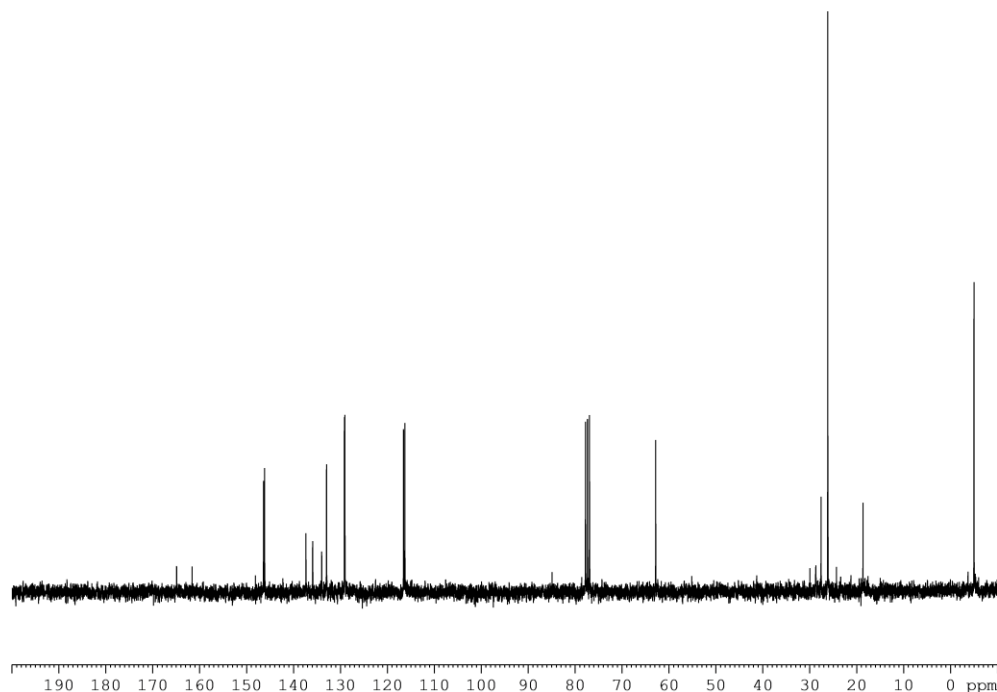
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 PULPROG zg30
 TD 16384
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.256020 Hz
 AQ 1.9530228 sec
 RG 57
 DW 119.200 usec
 DE 6.50 usec
 TE 295.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.70 usec
 PL1 0.00 dB
 SFO1 300.1319508 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300016 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 9.55 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm



Current Data Parameters
 NAME fc44wa535
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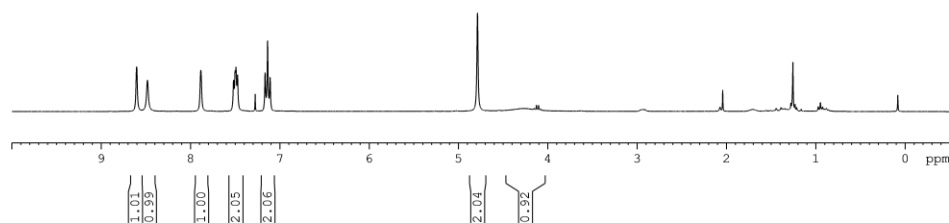
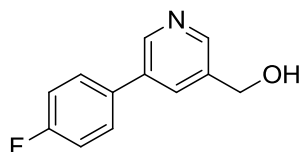
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 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 100
 DS 4
 SWH 18115.941 Hz
 FIDRES 0.552855 Hz
 AQ 0.9044468 sec
 RG 2048
 DW 27.600 usec
 DE 10.00 usec
 TE 295.1 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 -6.00 dB
 SFO1 75.4756731 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.54 dB
 PL13 21.54 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 6.89 cm
 F1P 200.000 ppm
 F1 15093.55 Hz
 F2P -10.000 ppm
 F2 -754.68 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 792.41138 Hz/cm

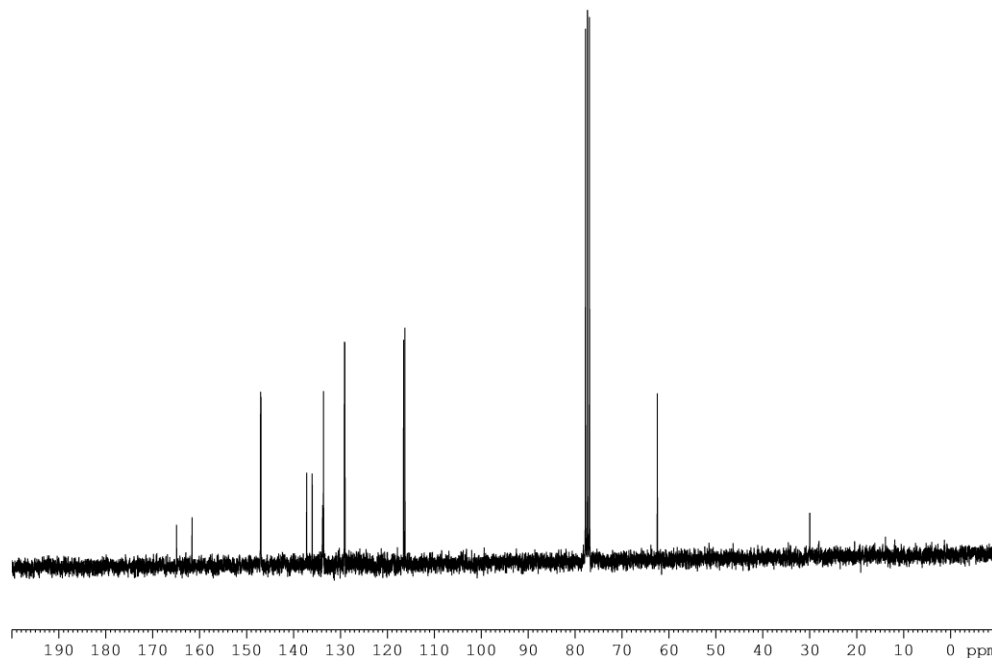


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 PROCNO 1
 F2 - Acquisition Parameters
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 Time_ 10.12
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 PULPROG zg30
 TD 16384
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.256020 Hz
 AQ 1.9530228 sec
 RG 161.3
 DW 119.200 usec
 DE 6.50 usec
 TE 293.9 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.70 usec
 PL1 0.00 dB
 SFO1 300.1319508 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300016 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 5.98 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm



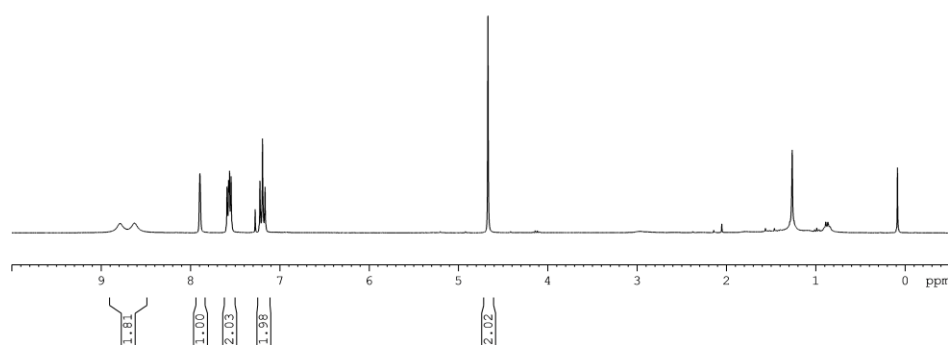
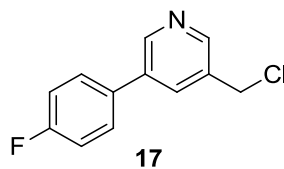
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 Time_ 10.15
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 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 250
 DS 4
 SWH 18115.941 Hz
 FIDRES 0.552855 Hz
 AQ 0.9044468 sec
 RG 2048
 DW 27.600 usec
 DE 10.00 usec
 TE 294.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 -6.00 dB
 SFO1 75.4756731 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.54 dB
 PL13 21.54 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 13.90 cm
 F1P 170.234 ppm
 F1 12847.15 Hz
 F2P 108.119 ppm
 F2 8159.52 Hz
 PPMCM 3.10572 ppm/cm
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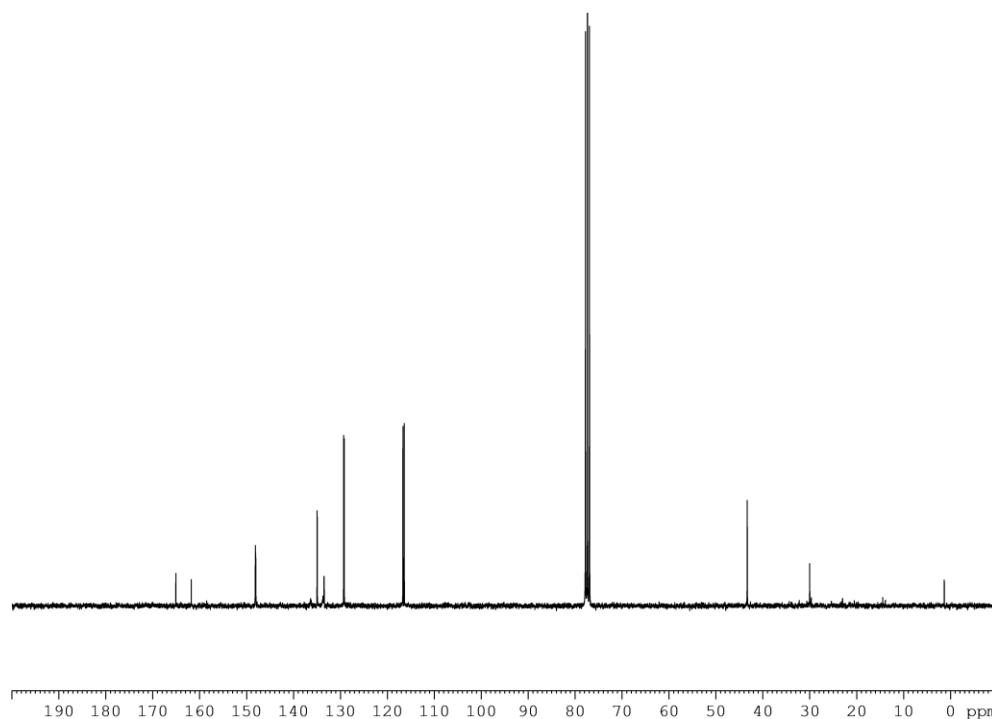
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 NAME fc45wa540
 EXPNO 300
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121107
 Time 8.49
 INSTRUM spect
 PROBHD 5 mm BBI 1H-BB
 PULPROG zg30
 TD 16384
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 4194.631 Hz
 FIDRES 0.256020 Hz
 AQ 1.9530228 sec
 RG 203.2
 DW 119.200 usec
 DE 6.50 usec
 TE 296.9 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.70 usec
 PL1 0.00 dB
 SFO1 300.1319508 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300016 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 5.64 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm



Current Data Parameters
 NAME fc46wa540
 EXPNO 14
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121114
 Time 20.47
 INSTRUM spect
 PROBHD 5 mm BBI 1H-BB
 PULPROG zgpg30
 TD 32768
 SOLVENT CDC13
 NS 2048
 DS 4
 SWH 18115.941 Hz
 FIDRES 0.552855 Hz
 AQ 0.9044468 sec
 RG 2048
 DW 27.600 usec
 DE 10.00 usec
 TE 295.1 K
 D1 4.00000000 sec
 d11 0.03000000 sec
 DELTA 3.90000010 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 -6.00 dB
 SFO1 75.4756731 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.54 dB
 PL13 21.54 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677258 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 10.79 cm
 F1P 200.000 ppm
 F1 15093.55 Hz
 F2P -10.000 ppm
 F2 -754.68 Hz
 PPMCM 10.50000 ppm/cm
 HZCM 792.41113 Hz/cm