Catalytic Asymmetric Synthesis of Allylic Thiol Derivatives

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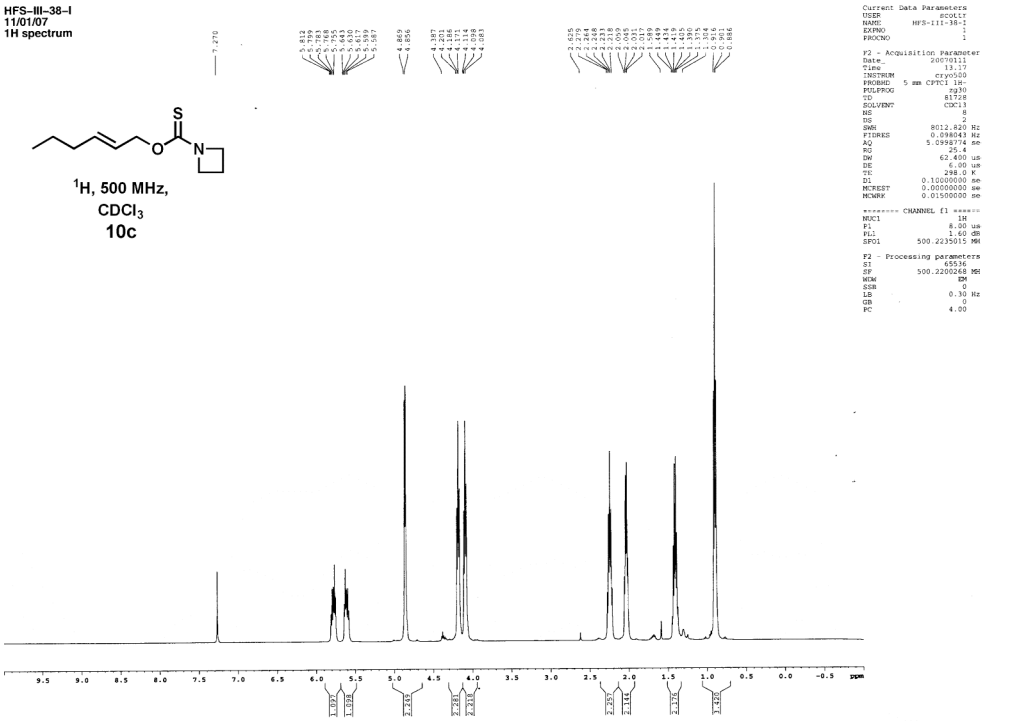
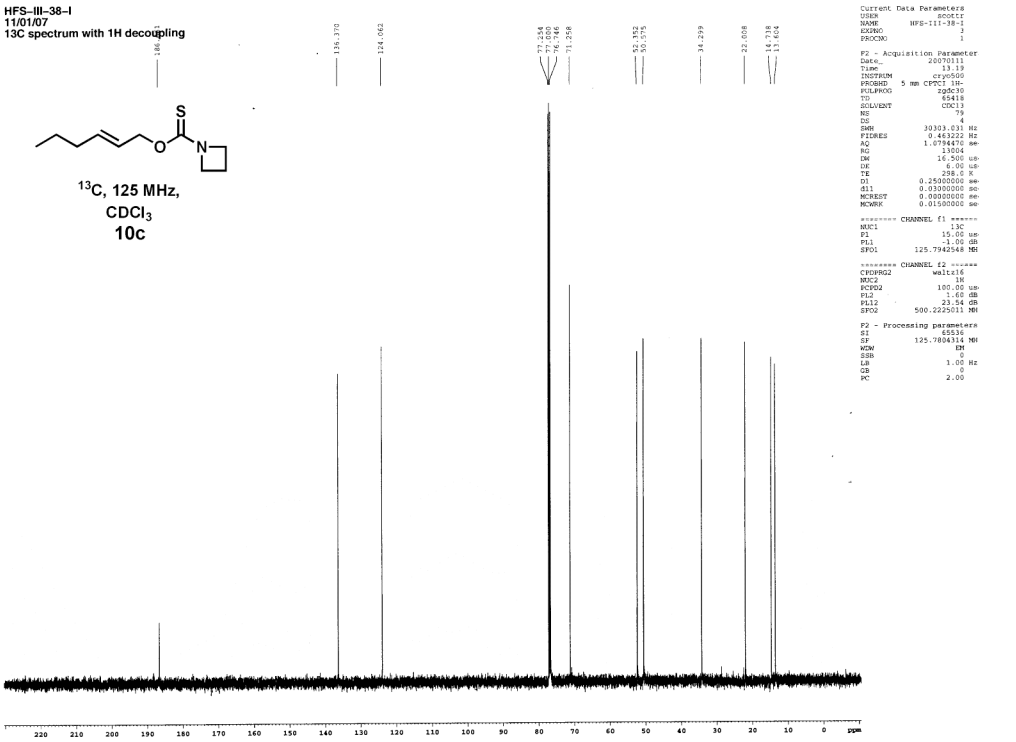
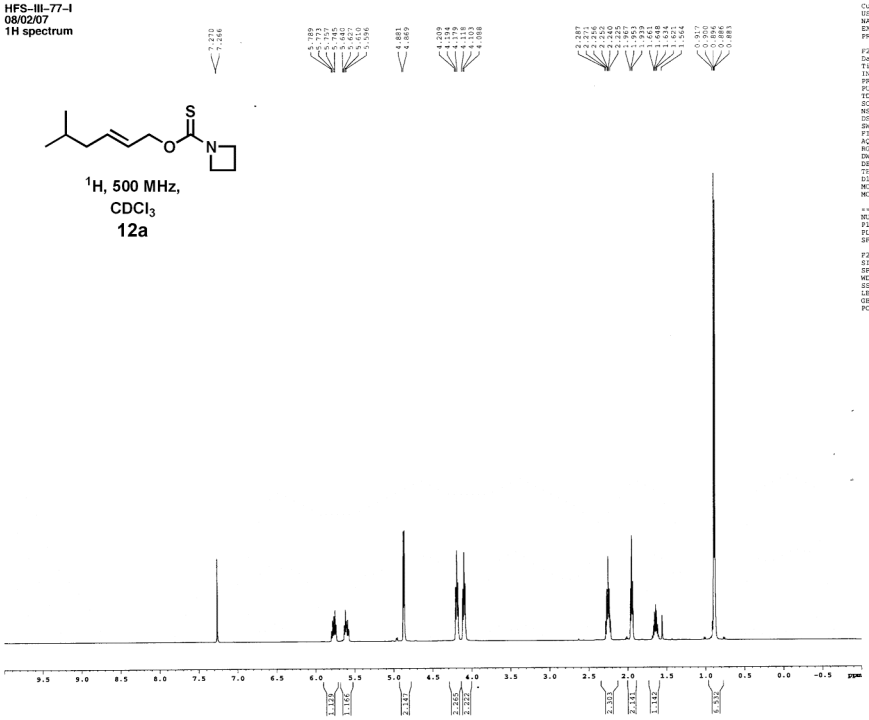
NMR Spectra.


HFS–III–38–I
11/01/07
1H spectrum
^{1}H, 500 MHz,
$CDCl_3$
10c
Current Data Parameters
USER scottr
NAME HFS-III-38-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20070111
Time 13.17
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 se
RG 25.4
DW 62.400 us
DE 6.00 us
TE 298.0 K
D1 0.10000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se
======== CHANNEL f1 ======
NUC1 1H
P1 8.00 us
PL1 1.60 dB
SFO1 500.2235015 MH
F2 - Processing parameters
SI 65536
SF 500.2200268 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00


HFS–III–38–I
11/01/07
13C spectrum with 1H decoupling
^{13}C, 125 MHz,
$CDCl_3$
10c
Current Data Parameters
USER scottr
NAME HFS-III-38-1
EXPNO 3
PROCNO 1
F2 - Acquisition Parameter
Date_ 20070111
Time 13.19
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 79
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794476 se
RG 13004
DW 16.500 us
DE 6.00 us
TE 298.6 K
D1 0.25000000 se
d11 0.03000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se
======== CHANNEL f1 ======
NUC1 13C
P1 15.00 us
PL1 -1.00 dB
SFO1 125.7942548 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MH
F2 - Processing parameters
SI 65536
SF 125.7804314 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

HFS–III–77–I
08/02/07
1H spectrum

^{1}H, 500 MHz, $CDCl_3$
12a



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PROCNO                  1

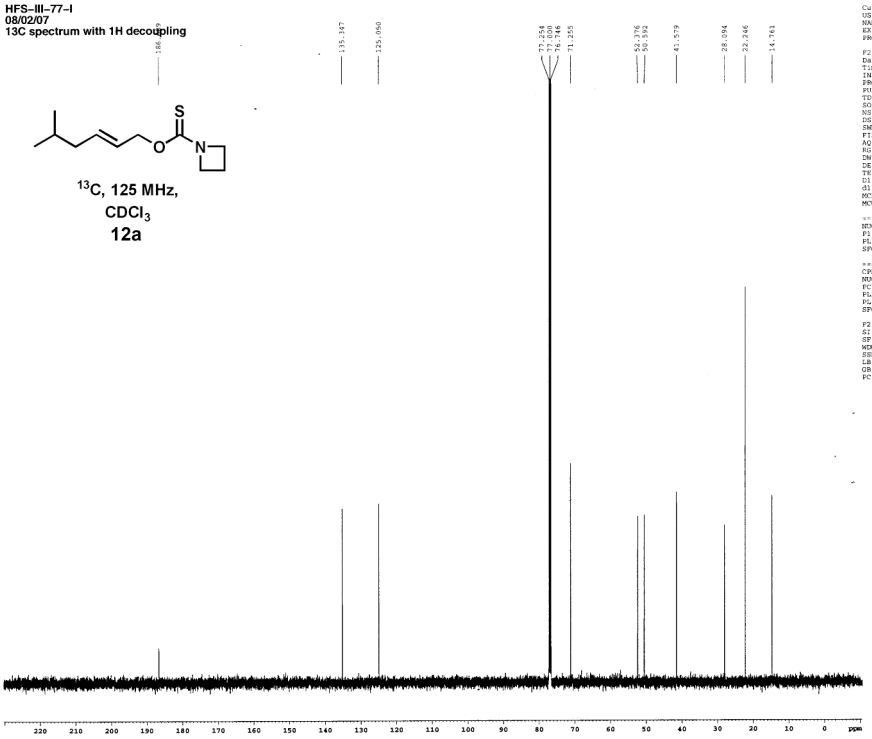
F2 - Acquisition Parameter
Date_           20070208
Time              21.17
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG           zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ             5.0998774 sec
RG                 71.8
DW               62.400 usec
DE                6.00 usec
TE              298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ======
NUC1                 1H
P1                 8.00 us
PL1               1.60 dB
SFO1        500.2235015 MHz

F2 - Processing parameters
SI                65536
SF          500.2200287 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 4.00
```

HFS–III–77–I
08/02/07
13C spectrum with 1H decoupling

^{13}C, 125 MHz, $CDCl_3$
12a



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Current Data Parameters
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NAME          HFS-III-77-I
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameter
Date_           20070208
Time              21.23
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG         zgdc30
TD                65418
SOLVENT           CDCl3
NS                  160
DS                    4
SWH           30303.031 Hz
FIDRES         0.463222 Hz
AQ             1.0794470 sec
RG                13004
DW               16.500 usec
DE                6.00 usec
TE              300.0 K
D1           0.25000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ======
NUC1                 13C
P1                15.00 us
PL1               -1.00 dB
SFO1        125.7942548 MHz

======== CHANNEL f2 ======
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 us
PL2               1.60 dB
PL12             23.54 dB
SFO2        500.2225011 MHz

F2 - Processing parameters
SI                65536
SF          125.7804291 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00
```
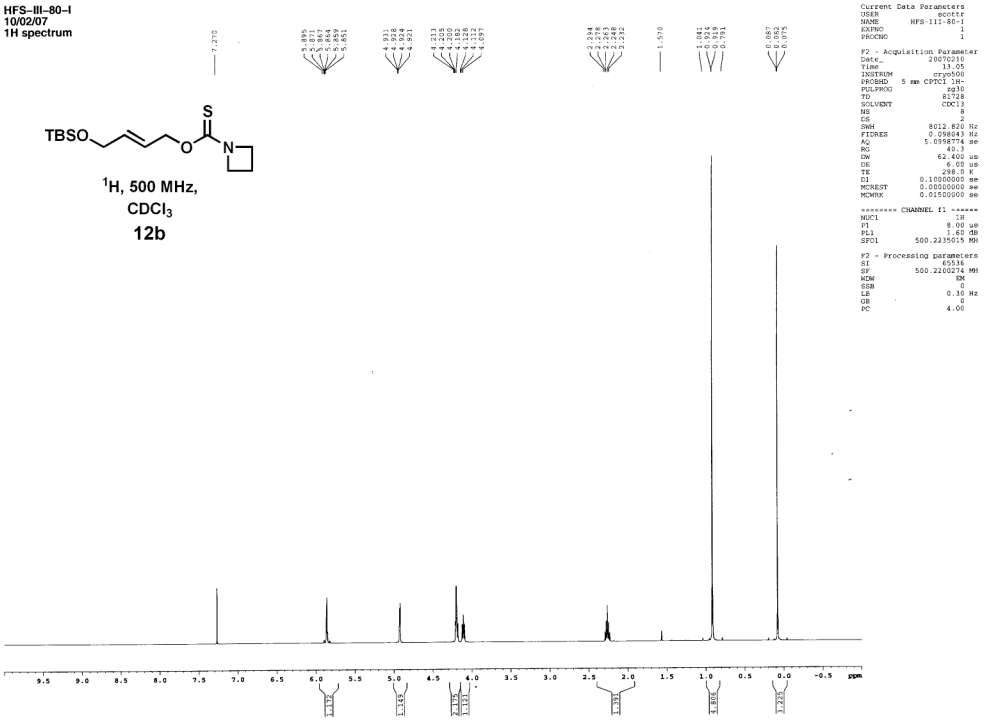
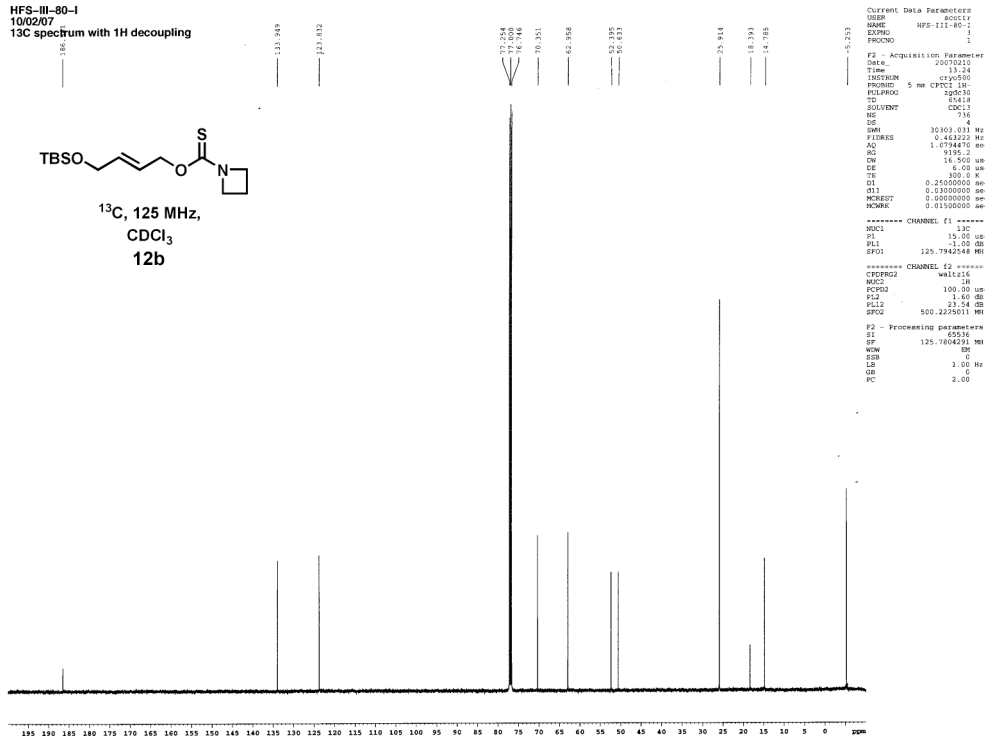
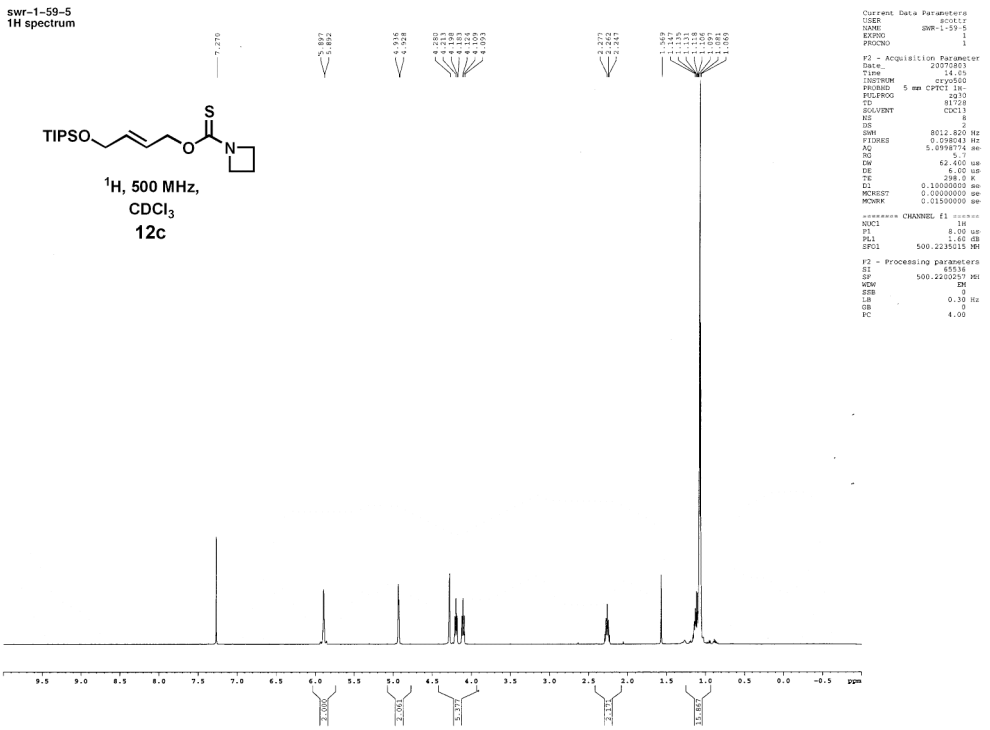
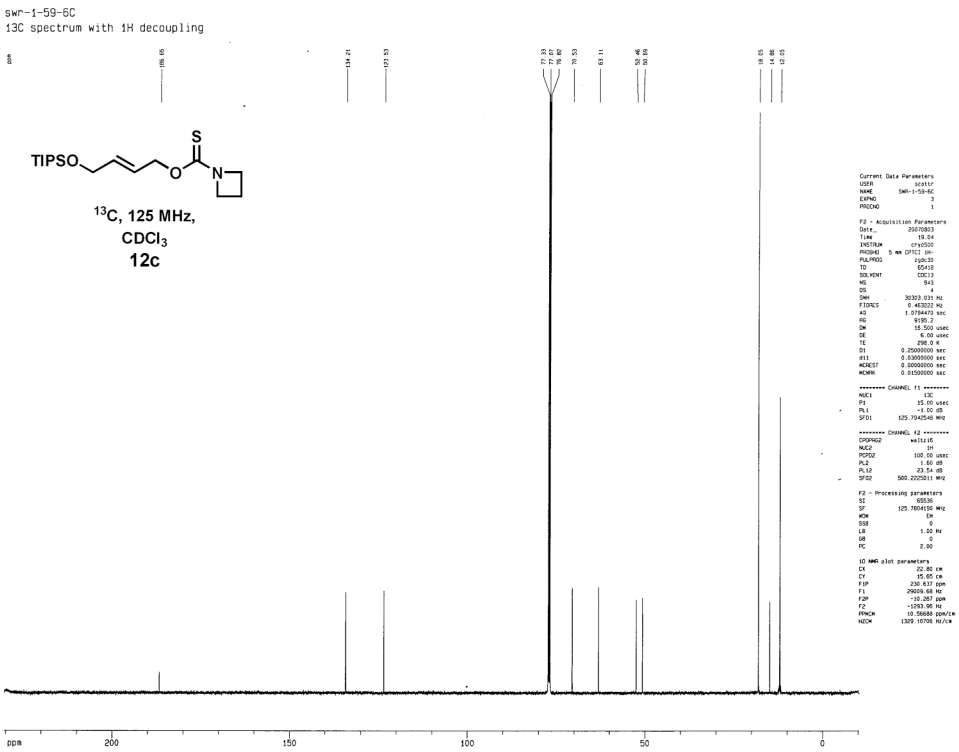
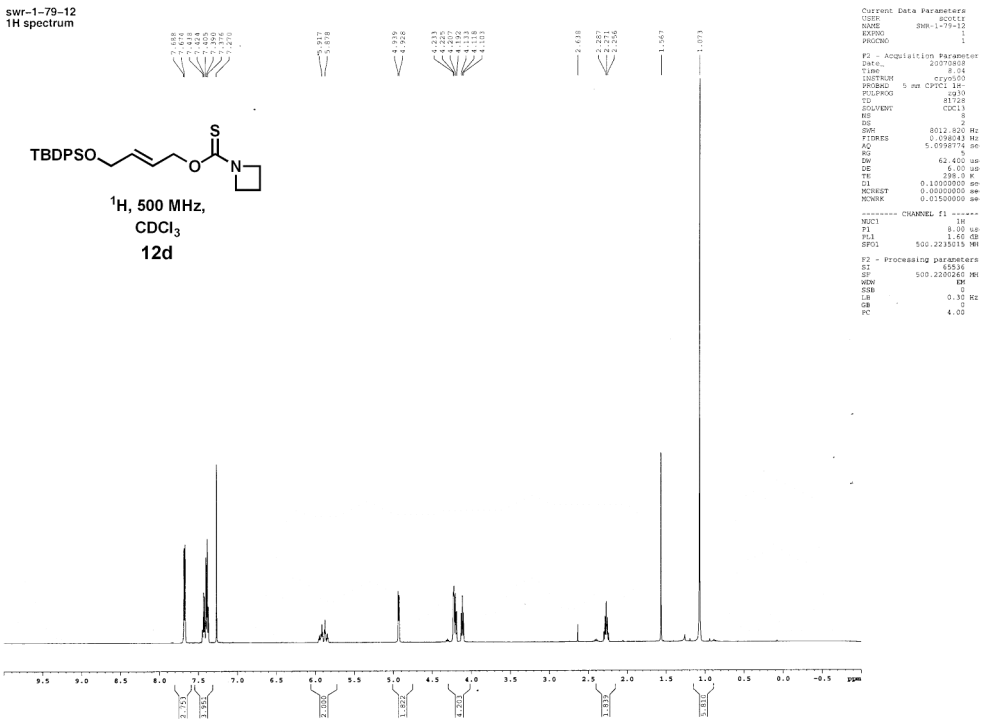
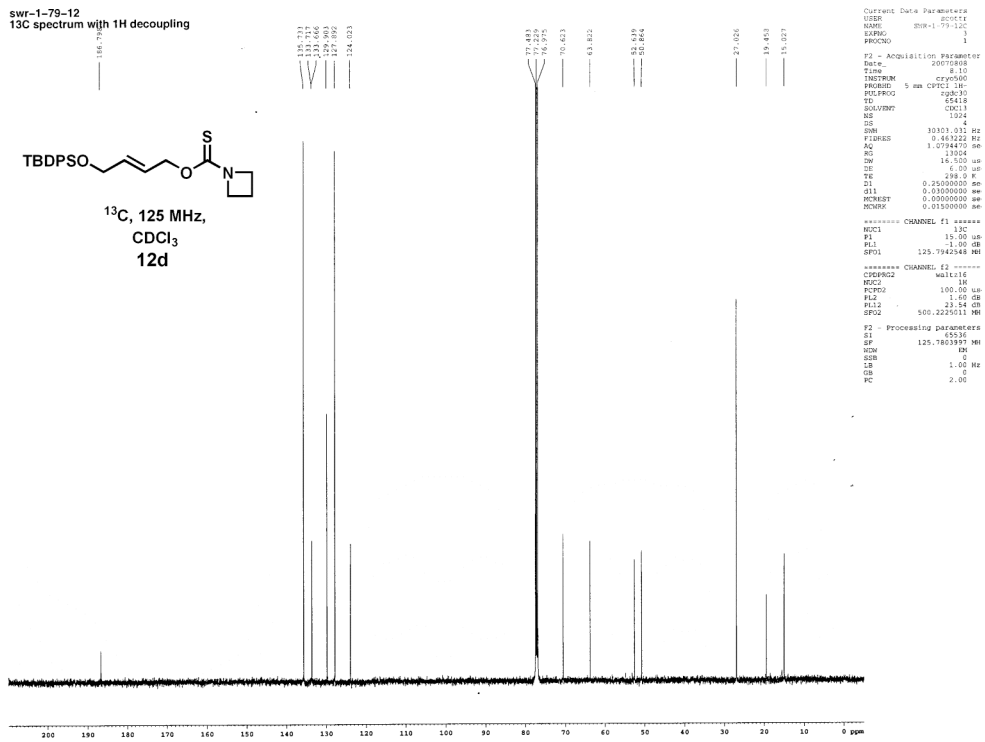


HFS–III–80–I
10/02/07
1H spectrum
TBSO
S
O
N
^{1}H, 500 MHz,
$CDCl_3$
12b
Current Data Parameters
USER scottr
NAME HFS-III-80-I
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20070210
Time 13.05
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 se
RG 40.3
DW 62.400 us
DE 6.00 us
TE 298.0 K
D1 0.10000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se
======== CHANNEL f1 ======
NUC1 1H
P1 8.00 us
PL1 1.60 dB
SFO1 500.2235015 MH
F2 - Processing parameters
SI 65536
SF 500.2200274 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00


HFS–III–80–I
10/02/07
13C spectrum with 1H decoupling
TBSO
S
O
N
^{13}C, 125 MHz,
$CDCl_3$
12b
Current Data Parameters
USER scottr
NAME HFS-III-80-I
EXPNO 3
PROCNO 1
F2 - Acquisition Parameter
Date_ 20070210
Time 13.24
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 736
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 se
RG 9195.2
DW 16.500 us
DE 6.00 us
TE 300.0 K
D1 0.25000000 se
d11 0.03000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se
======== CHANNEL f1 ======
NUC1 13C
P1 15.00 us
PL1 -1.00 dB
SFO1 125.7942548 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2235011 MH
F2 - Processing parameters
SI 65536
SF 125.7804291 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00


swr-1-59-5
1H spectrum
TIPSO
S
O
N
^{1}H, 500 MHz,
$CDCl_3$
12c
ppm

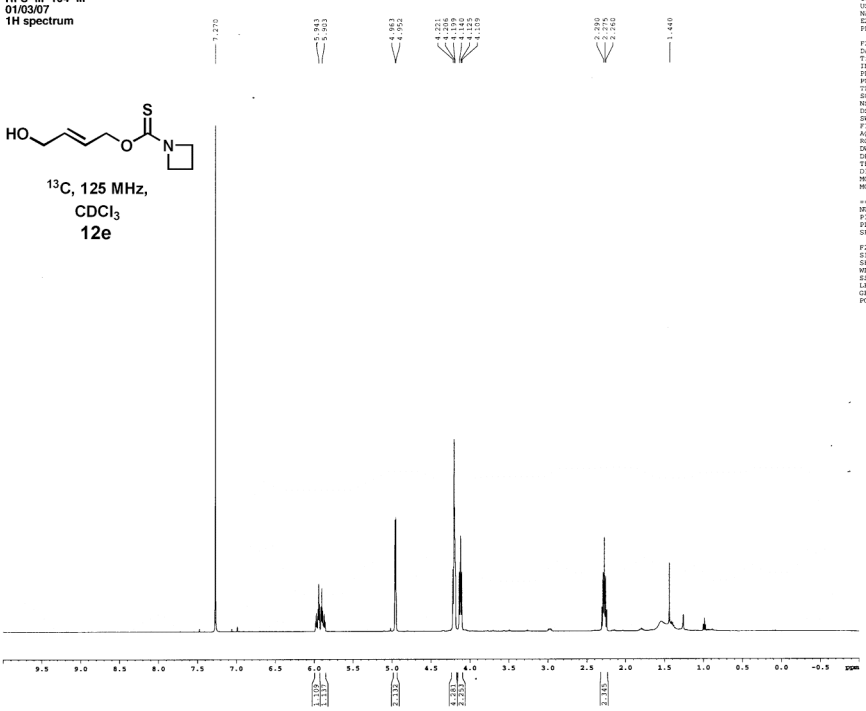

swr-1-59-6C
13C spectrum with 1H decoupling
TIPSO
S
O
N
^{13}C, 125 MHz,
$CDCl_3$
12c
ppm


swr-1-79-12
1H spectrum
TBDPSO
S
O
N
^{1}H, 500 MHz,
$CDCl_3$
12d
Current Data Parameters
USER scott
NAME SWR-1-79-12
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20070808
Time 8.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 se
RG 5
DW 62.400 us
DE 6.00 us
TE 298.0 K
D1 0.10000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se
-------- CHANNEL f1 ------
NUC1 1H
P1 8.00 us
PL1 1.60 dB
SFO1 500.2235015 MH
F2 - Processing parameters
SI 65536
SF 500.2200260 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00


swr-1-79-12
13C spectrum with 1H decoupling
TBDPSO
S
O
N
^{13}C, 125 MHz,
$CDCl_3$
12d
Current Data Parameters
USER scott
NAME SWR-1-79-12C
EXPNO 3
PROCNO 1
F2 - Acquisition Parameter
Date_ 20070808
Time 8.10
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 1024
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 se
RG 13004
DW 16.500 us
DE 6.00 us
TE 298.0 K
D1 0.25000000 se
d11 0.03000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se
-------- CHANNEL f1 ------
NUC1 13C
P1 15.00 us
PL1 1.00 dB
SFO1 125.7942548 MH
-------- CHANNEL f2 ------
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MH
F2 - Processing parameters
SI 65536
SF 125.7803997 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

HFS–III–104–III
01/03/07
1H spectrum

^{13}C, 125 MHz,
$CDCl_3$
12e



Current Data Parameters
USER scottr
NAME HFS-III-104-III
EXPNO 4
PROCNO 1

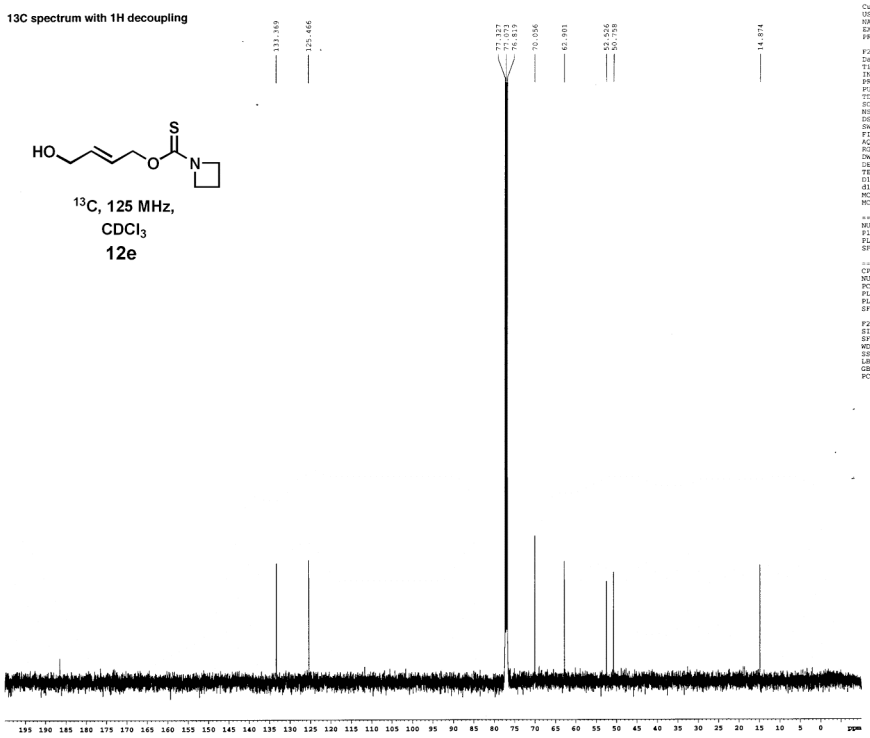
F2 - Acquisition Parameter
Date_ 20070301
Time 8.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 se
RG 406.4
DW 62.400 us
DE 6.00 us
TE 298.0 K
D1 0.10000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se

======== CHANNEL f1 ======
NUC1 1H
P1 8.00 us
PL1 1.60 dB
SFO1 500.2235015 MH

F2 - Processing parameters
SI 65536
SF 500.2200263 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

13C spectrum with 1H decoupling

^{13}C, 125 MHz,
$CDCl_3$
12e



Current Data Parameters
USER scottr
NAME HFS-III-104-III
EXPNO 6
PROCNO 1

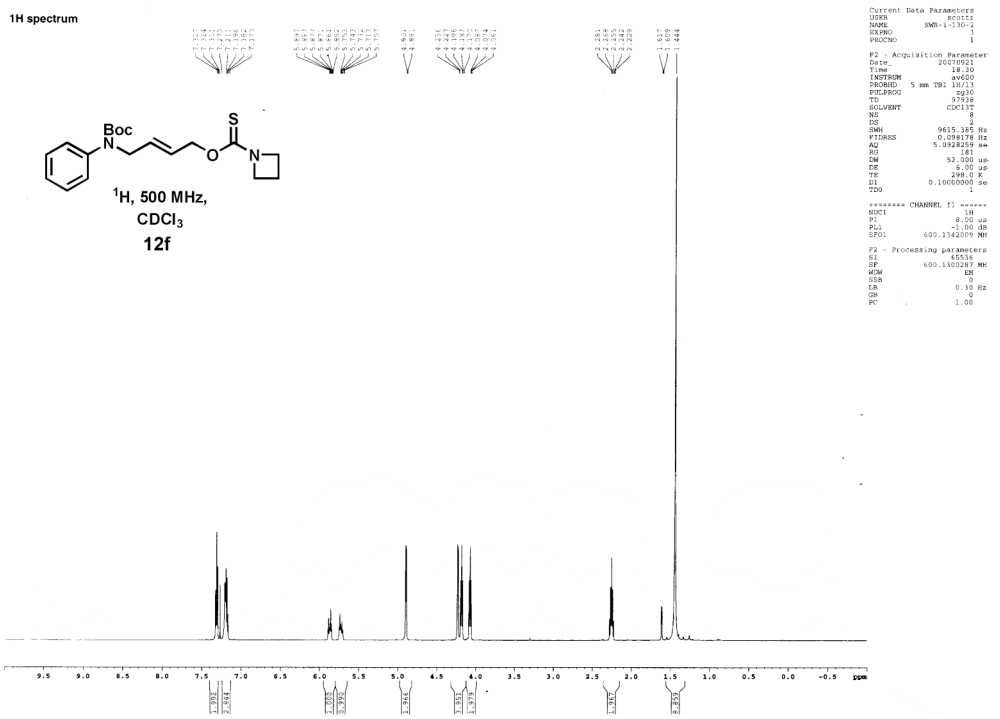
F2 - Acquisition Parameter
Date_ 20070301
Time 8.37
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 608
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 se
RG 8192
DW 16.500 us
DE 6.00 us
TE 298.0 K
D1 0.25000000 se
d11 0.03000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se

======== CHANNEL f1 ======
NUC1 13C
P1 15.00 us
PL1 -1.00 dB
SFO1 125.7942548 MH

======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MH

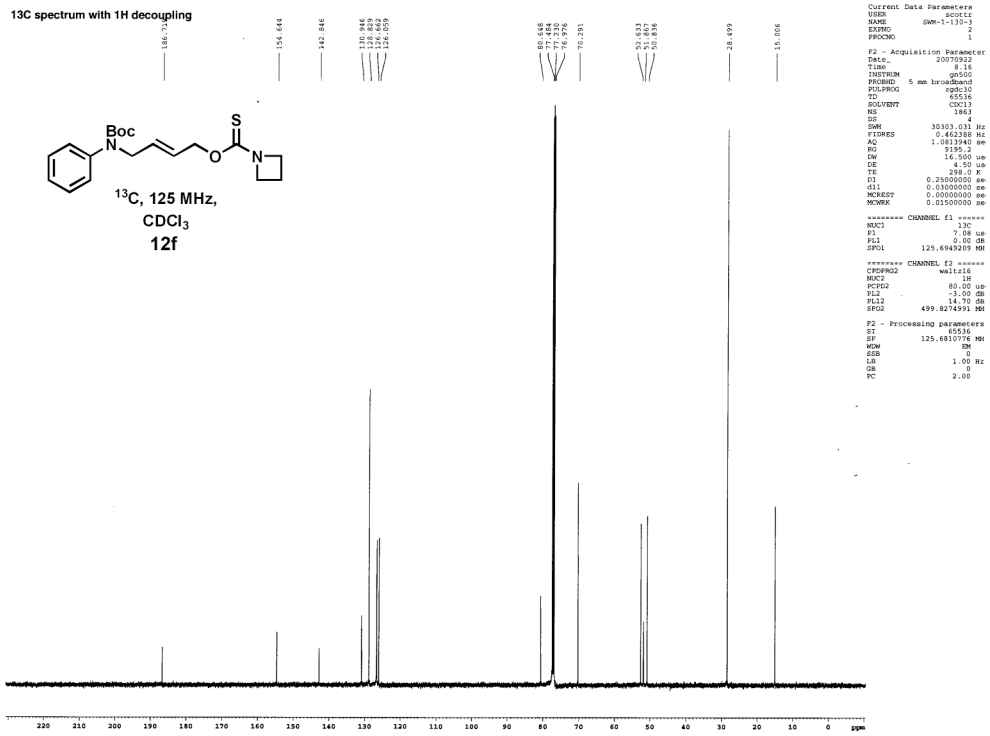
F2 - Processing parameters
SI 65536
SF 125.7804190 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1H spectrum




```
Current Data Parameters
USER              scottr
NAME          SWR-1-130-2
EXPNO               1
PROCNO              1

F2 - Acquisition Parameter
Date_          20070921
Time             18.30
INSTRUM          av600
PROBHD   5 mm TBI 1H/13
PULPROG          zg30
TD               97938
SOLVENT          CDCl3
NS                  8
DS                  2
SWH            9615.385 Hz
FIDRES        0.098178 Hz
AQ            5.0928259 sec
RG                181
DW             52.000 usec
DE              6.00 usec
TE            298.0 K
D1          0.10000000 sec
TD0                 1

======== CHANNEL f1 ======
NUC1               1H
P1              8.00 usec
PL1            -1.00 dB
SFO1       600.1342009 MHz

F2 - Processing parameters
SI              65536
SF         600.1300287 MHz
WDW               EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```

13C spectrum with 1H decoupling

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Current Data Parameters
USER              scottr
NAME          SWR-1-130-3
EXPNO               2
PROCNO              1

F2 - Acquisition Parameter
Date_          20070922
Time              8.16
INSTRUM         qnt500
PROBHD   5 mm broadband
PULPROG        zgdc30
TD              65536
SOLVENT         CDCl3
NS               1863
DS                  4
SWH           30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813940 sec
RG              9195.2
DW             16.500 usec
DE              4.50 usec
TE            298.0 K
D1          0.25000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               13C
P1              7.08 usec
PL1             0.00 dB
SFO1       125.6949209 MHz

======== CHANNEL f2 ======
CPDPRG2         waltz16
NUC2               1H
PCPD2          80.00 usec
PL2            -3.00 dB
PL12           14.70 dB
SFO2       499.8274991 MHz

F2 - Processing parameters
SI              65536
SF         125.6810776 MHz
WDW               EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               2.00
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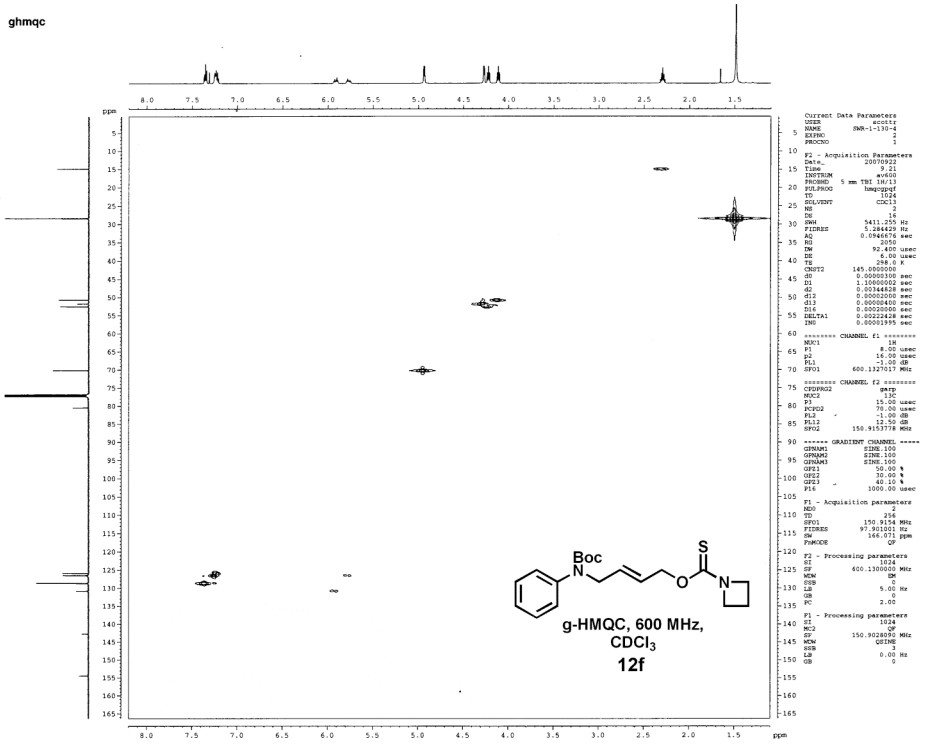
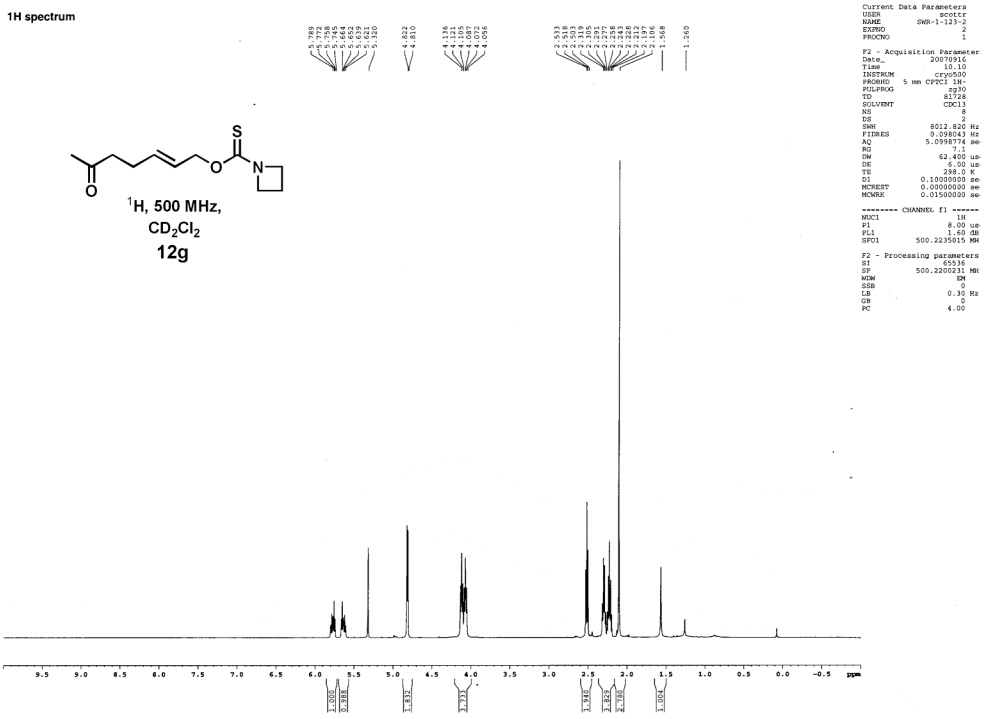
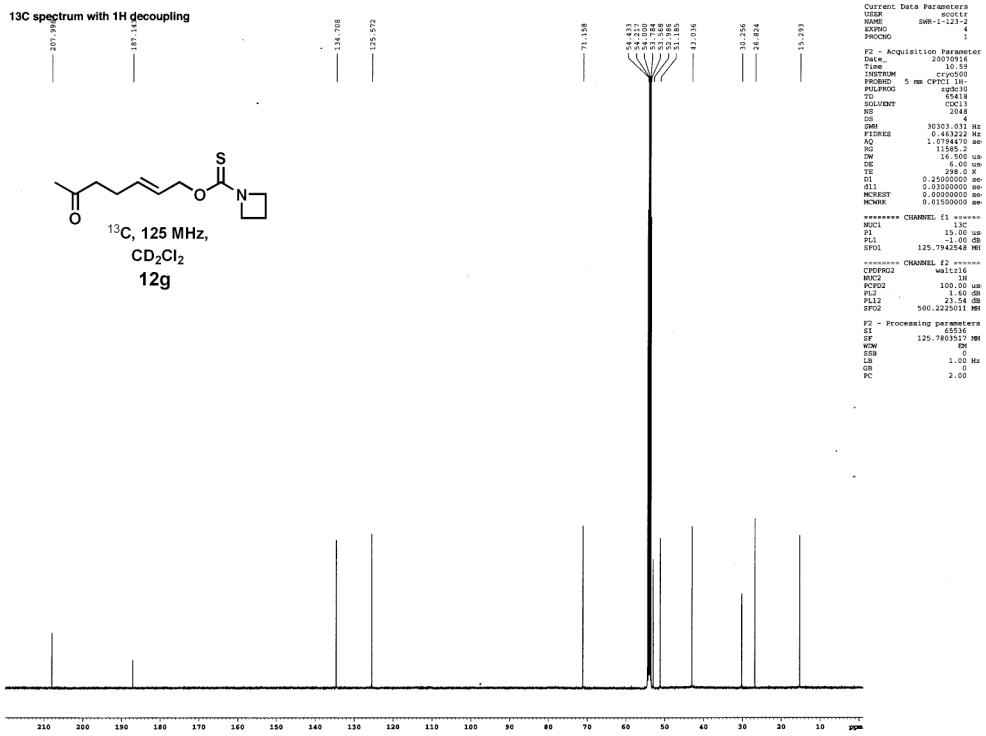
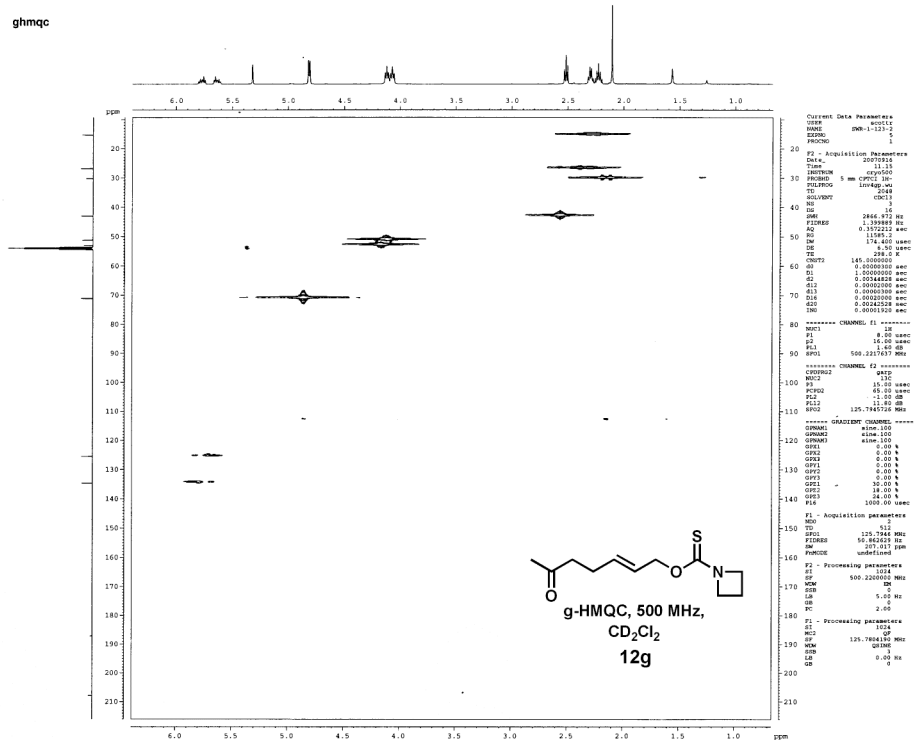
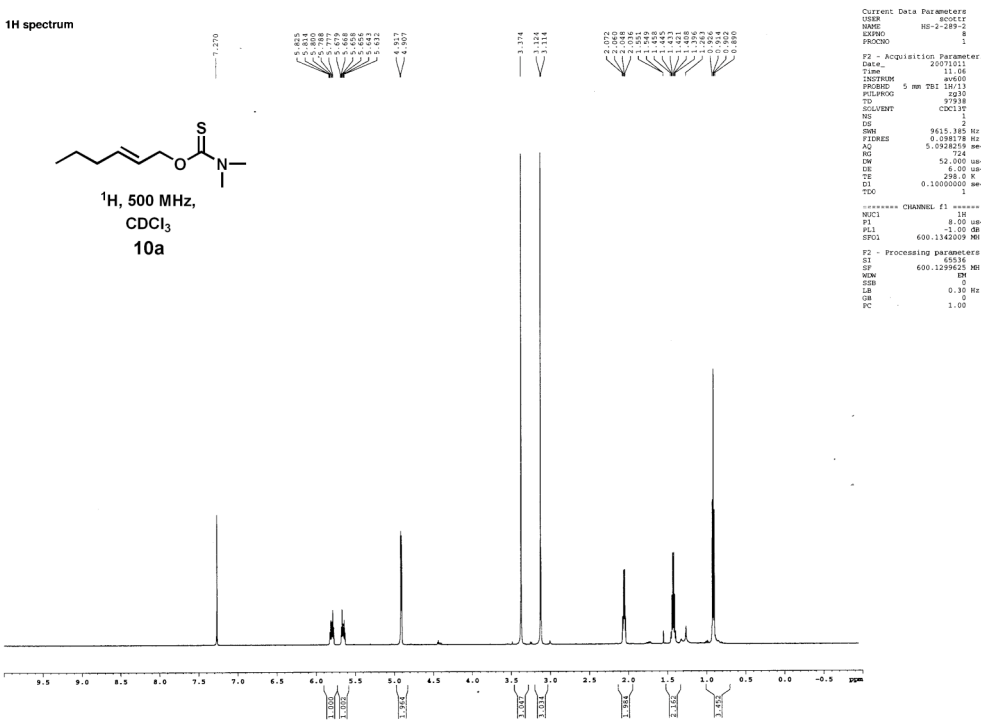

ghmqc
Boc
N
S
O
N
g-HMQC, 600 MHz,
CDCl3
12f


1H spectrum
S
O
N
O
1H, 500 MHz,
CD2Cl2
12g

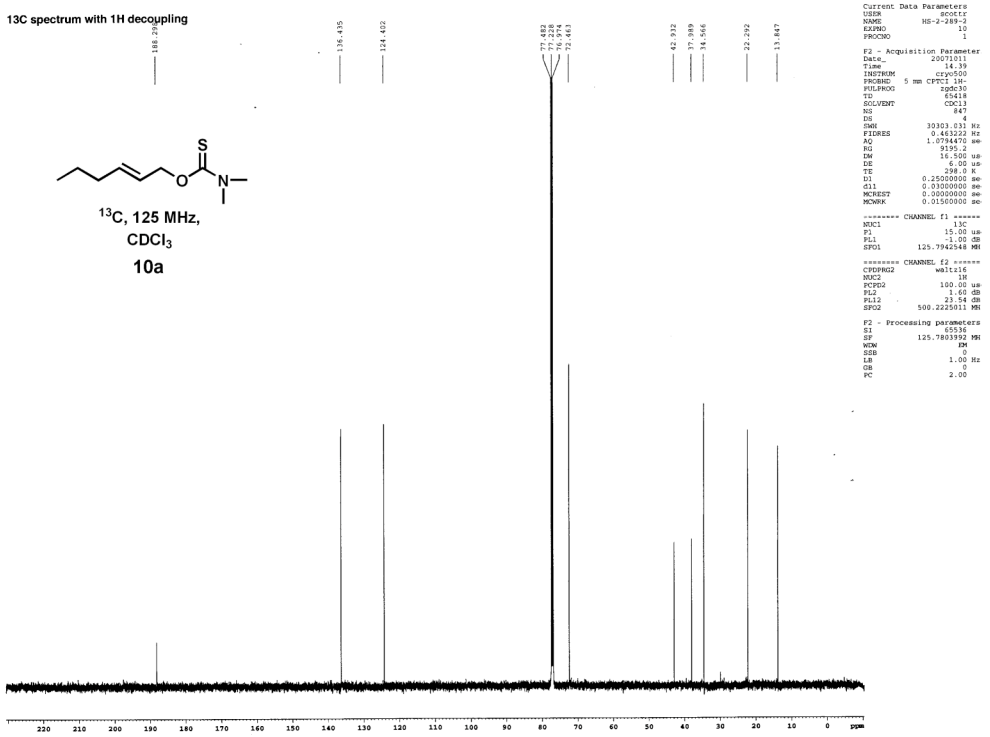

13C spectrum with 1H decoupling
207.999
187.144
134.708
125.572
71.158
54.433
54.217
54.000
53.784
53.568
53.352
51.185
43.036
30.256
26.824
15.293
S
O
N
^{13}C, 125 MHz,
CD_2Cl_2
12g
Current Data Parameters
USER scottr
NAME SWR-1-123-2
EXPNO 4
PROCNO 1
F2 - Acquisition Parameter
Date_ 20070916
Time 10.59
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 2048
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 se
RG 11585.2
DW 16.500 us
DE 6.00 us
TE 298.0 K
D1 0.25000000 se
d11 0.03000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se
======== CHANNEL f1 ======
NUC1 13C
P1 15.00 us
PL1 -1.00 dB
SFO1 125.7942548 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MH
F2 - Processing parameters
SI 65536
SF 125.7803517 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

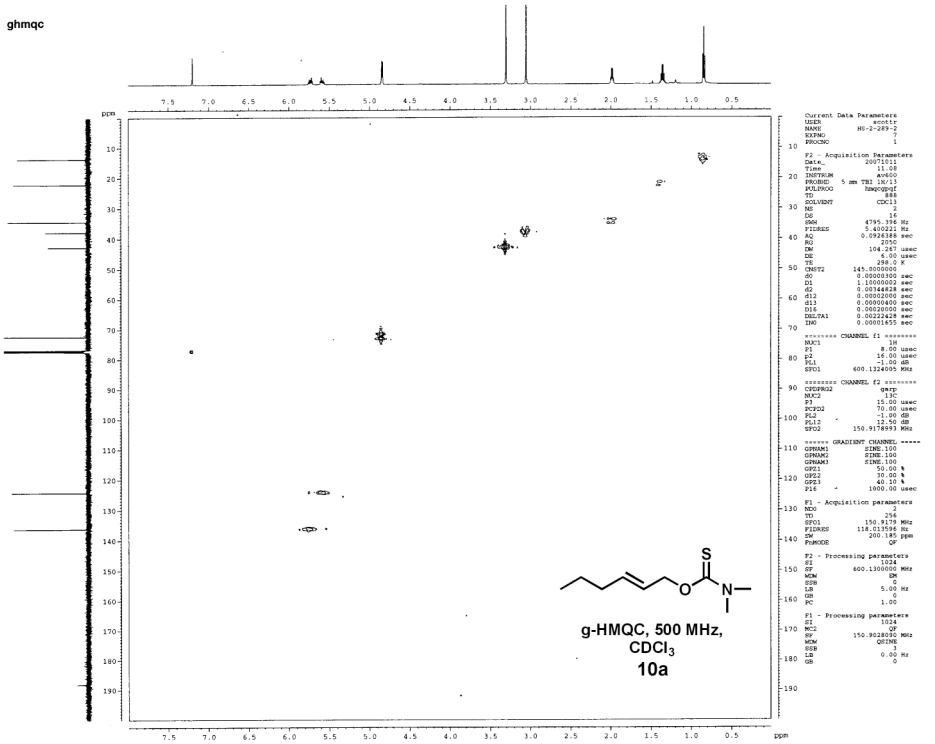

ghmqc
S
O
N
g-HMQC, 500 MHz,
CD_2Cl_2
12g
Current Data Parameters
USER scottr
NAME SWR-1-123-2
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070916
Time 11.15
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG inv4gp.wu
TD 2048
SOLVENT CDCl3
NS 2
DS 16
SWH 2866.972 Hz
FIDRES 1.399889 Hz
AQ 0.3572212 sec
RG 11585.2
DW 174.400 usec
DE 6.50 usec
TE 298.0 K
CNST2 145.0000000
d1 1.00000000 sec
d11 0.03000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
d13 0.00000300 sec
D16 0.00020000 sec
d20 0.00242528 sec
IN0 0.00001920 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
p2 16.00 usec
PL1 1.60 dB
SFO1 500.2217037 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 15.00 usec
PCPD2 65.00 usec
PL2 -1.00 dB
PL12 11.80 dB
SFO2 125.7945726 MHz
====== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPNAM3 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 30.00 %
GPZ2 16.00 %
GPZ3 0.00 %
PLW 1000.00 usec
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 125.7946 MHz
FIDRES 50.862629 Hz
SW 207.017 ppm
FnMODE undefined
F2 - Processing parameters
SI 1024
SF 500.2200002 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 2.00
F1 - Processing parameters
SI 1024
MC2 QF
SF 125.7804190 MHz
WDW QSINE
SSB 0
LB 0.00 Hz
GB 0

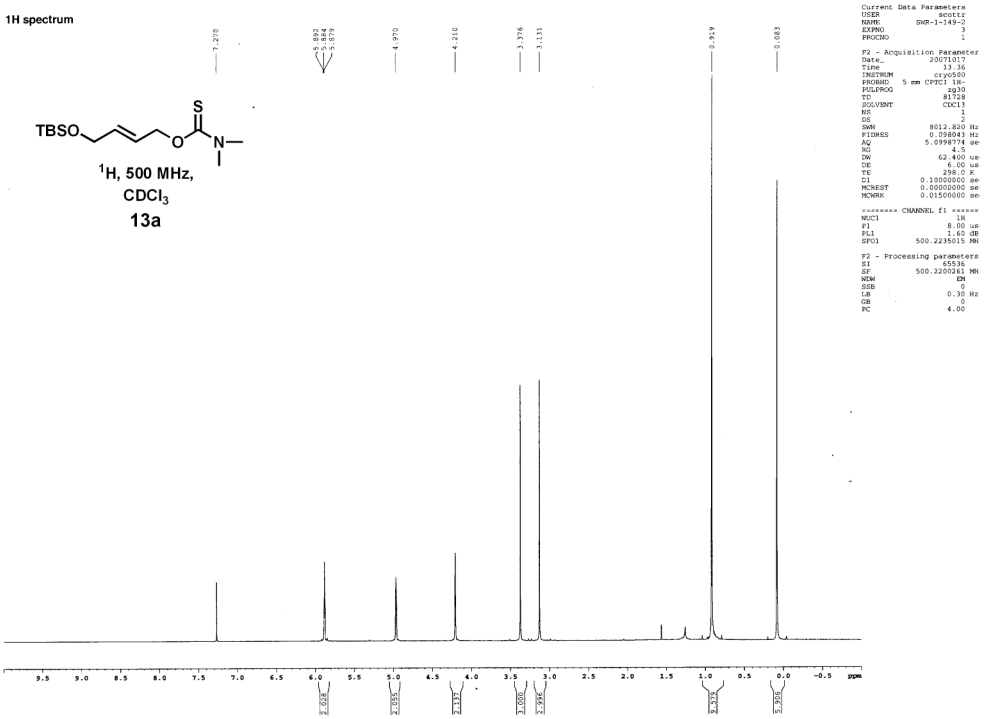
1H spectrum

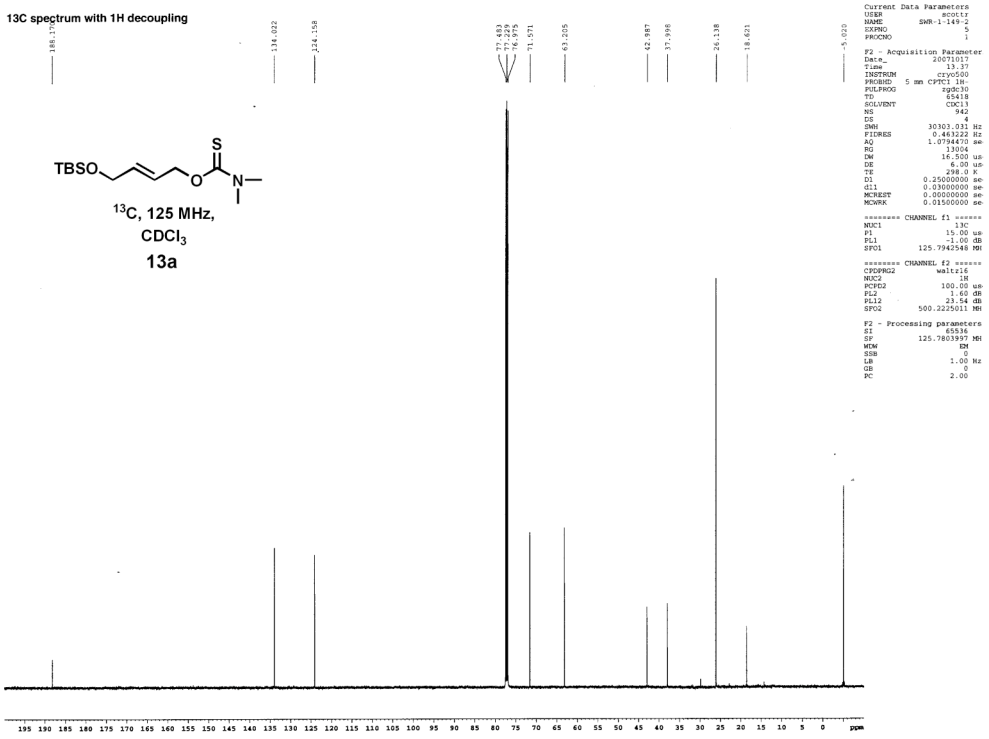


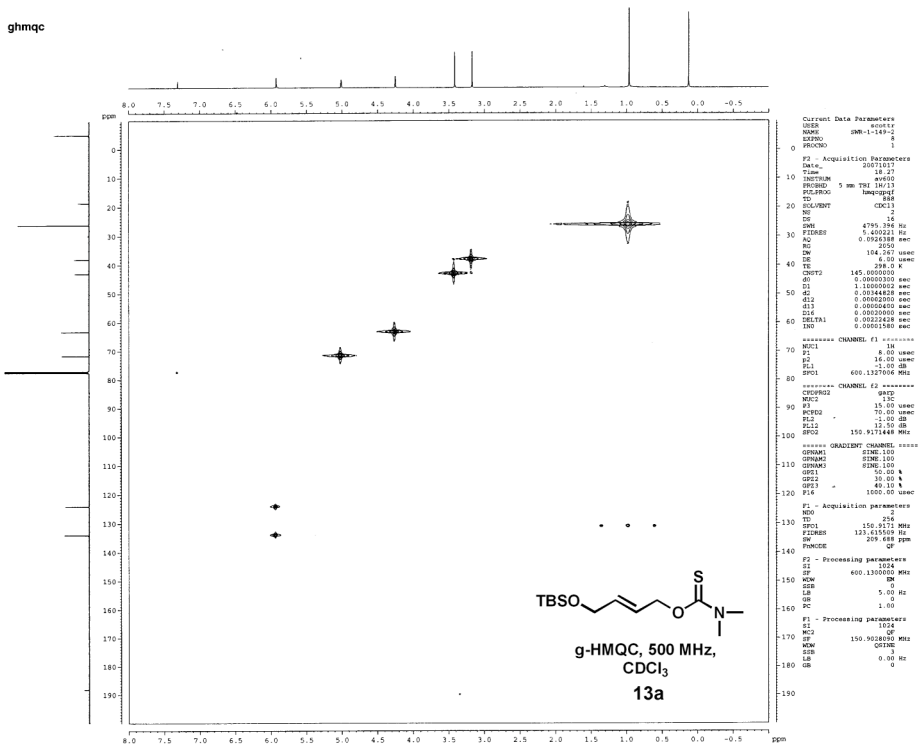
13C spectrum with 1H decoupling



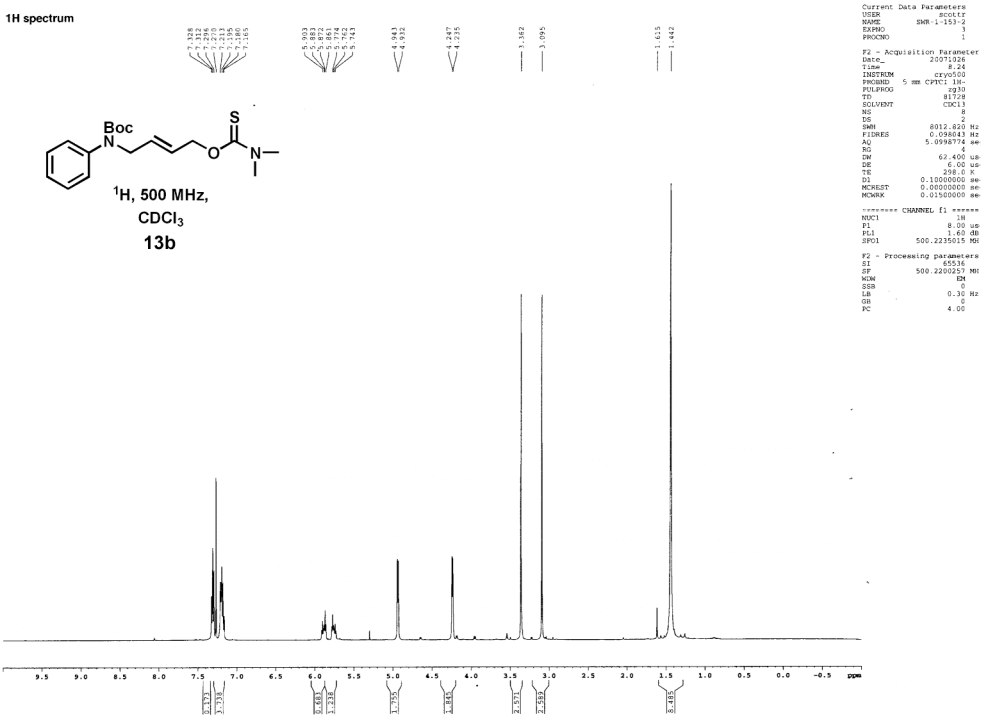

ghmqc
g-HMQC, 500 MHz,
CDCl3
10a


1H spectrum
TBSO
1H, 500 MHz,
CDCl3
13a


13C spectrum with 1H decoupling
TBSO
S
O
N
^{13}C, 125 MHz,
$CDCl_3$
13a


ghmqc
TBSO
S
O
N
g-HMQC, 500 MHz,
$CDCl_3$
13a

1H spectrum



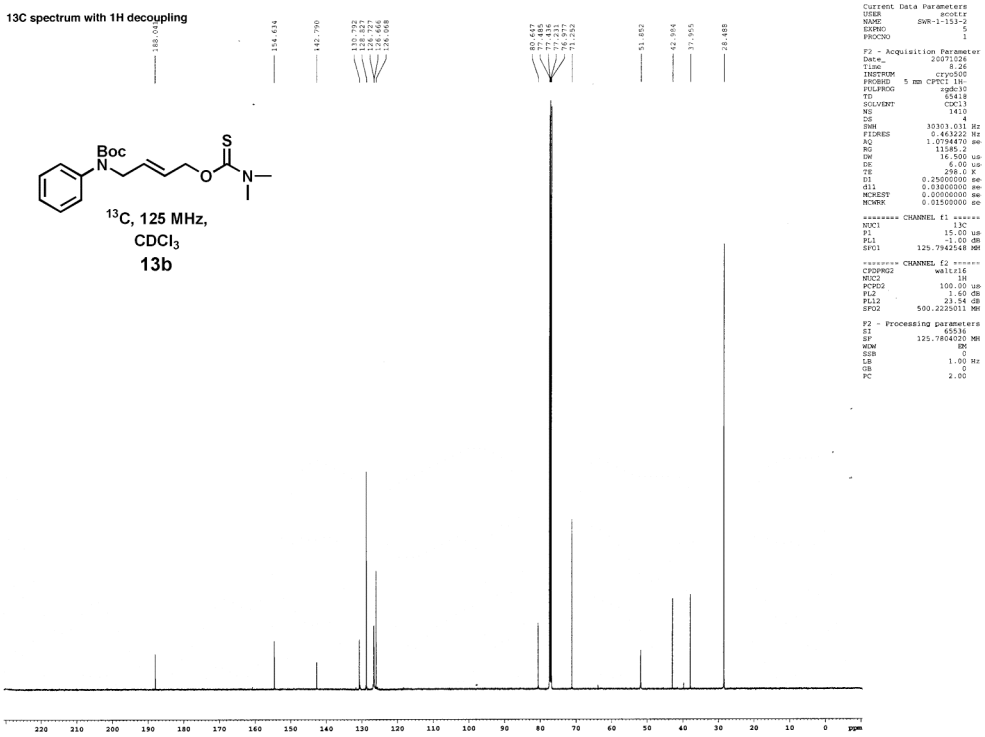

```
Current Data Parameters
USER              scottr
NAME          SWR-1-153-2
EXPNO               3
PROCNO              1

F2 - Acquisition Parameter
Date_          20071026
Time              8.24
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG           zg30
TD               81728
SOLVENT          CDCl3
NS                  8
DS                  2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ             5.0998774 se
RG                  4
DW              62.400 us
DE               6.00 us
TE              298.0 K
D1          0.10000000 se
MCREST      0.00000000 se
MCWRK       0.01500000 se

======== CHANNEL f1 ======
NUC1               1H
P1               8.00 us
PL1              1.60 dB
SFO1       500.2235015 MH

F2 - Processing parameters
SI               65536
SF         500.2200257 MH
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               4.00
```

13C spectrum with 1H decoupling



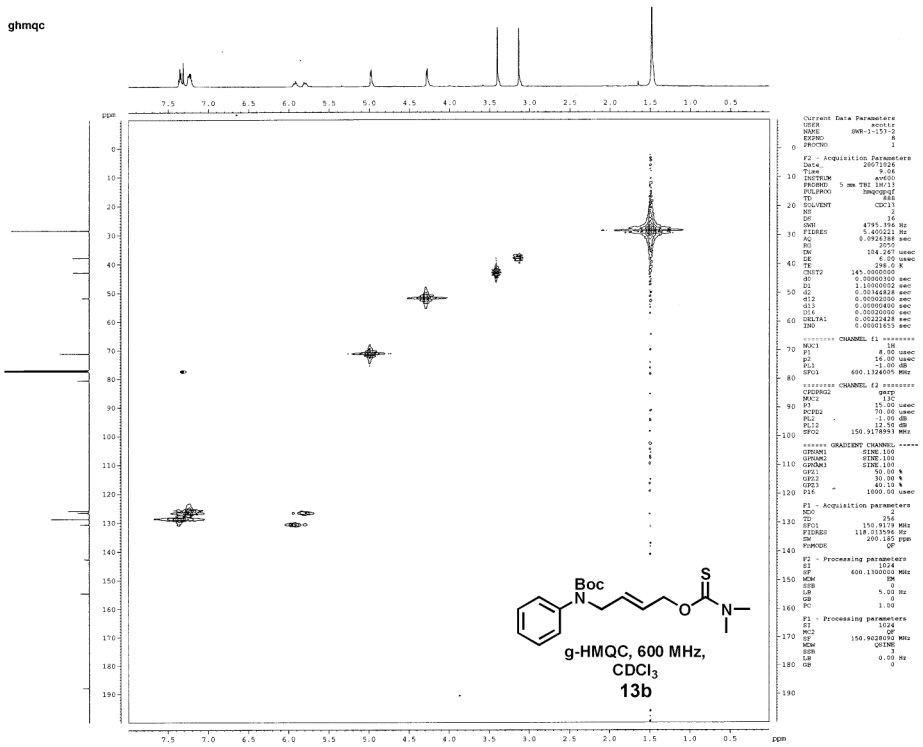

```
Current Data Parameters
USER              scottr
NAME          SWR-1-153-2
EXPNO               5
PROCNO              1

F2 - Acquisition Parameter
Date_          20071026
Time              8.26
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG         zgdc30
TD               65418
SOLVENT          CDCl3
NS                1410
DS                  4
SWH           30303.031 Hz
FIDRES         0.463222 Hz
AQ             1.0794470 se
RG             11585.2
DW              16.500 us
DE               6.00 us
TE              298.0 K
D1          0.25000000 se
d11         0.03000000 se
MCREST      0.00000000 se
MCWRK       0.01500000 se

======== CHANNEL f1 ======
NUC1               13C
P1              15.00 us
PL1             -1.00 dB
SFO1       125.7942548 MH

======== CHANNEL f2 ======
CPDPRG2         waltz16
NUC2               1H
PCPD2          100.00 us
PL2              1.60 dB
PL12            23.54 dB
SFO2       500.2225011 MH

F2 - Processing parameters
SI               65536
SF         125.7804020 MH
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               2.00
```


ghmqc
Boc
N
S
O
N
g-HMQC, 600 MHz,
CDCl3
13b