

Electronic Supplementary Information

Design, Synthesis and Biological evaluation of Bicyclic Iminosugar Hybrids: Conformational constraint as an effective tool for tailoring the selectivity of α -glucosidase inhibitors

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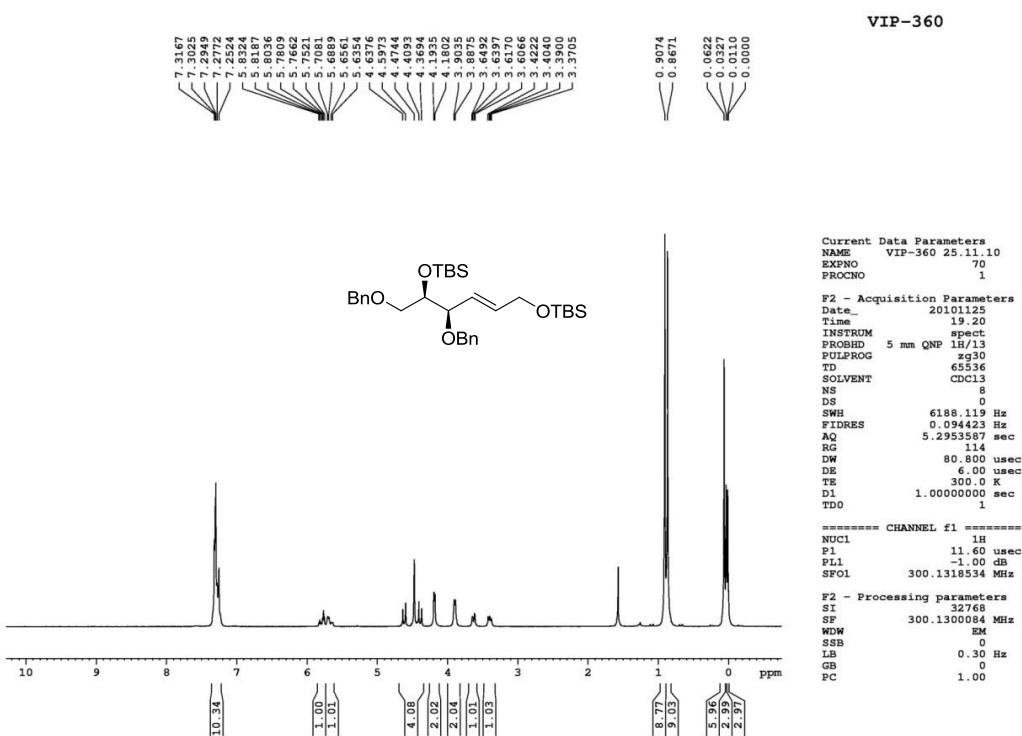
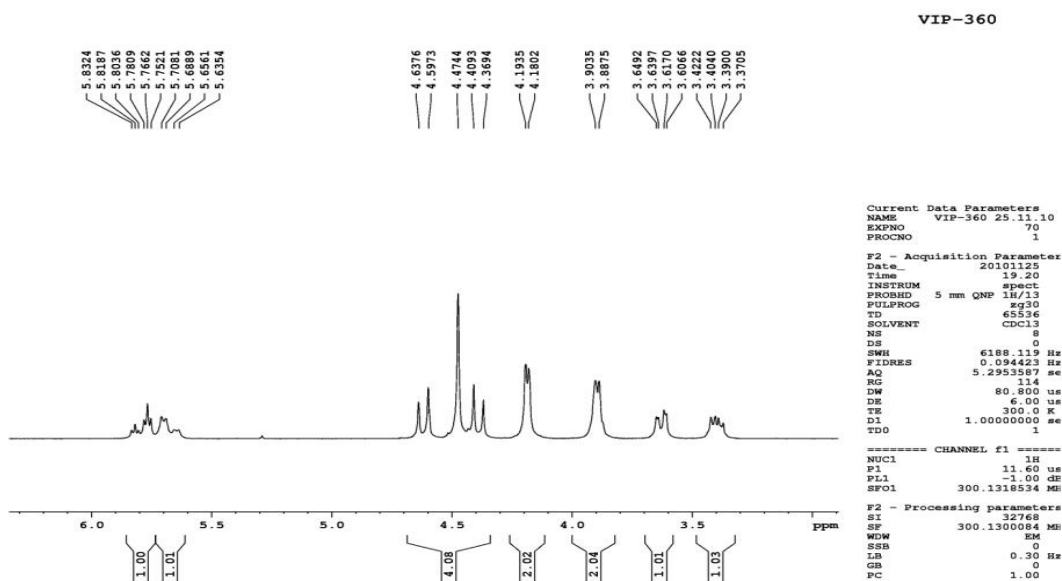
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Lucknow 226031, India

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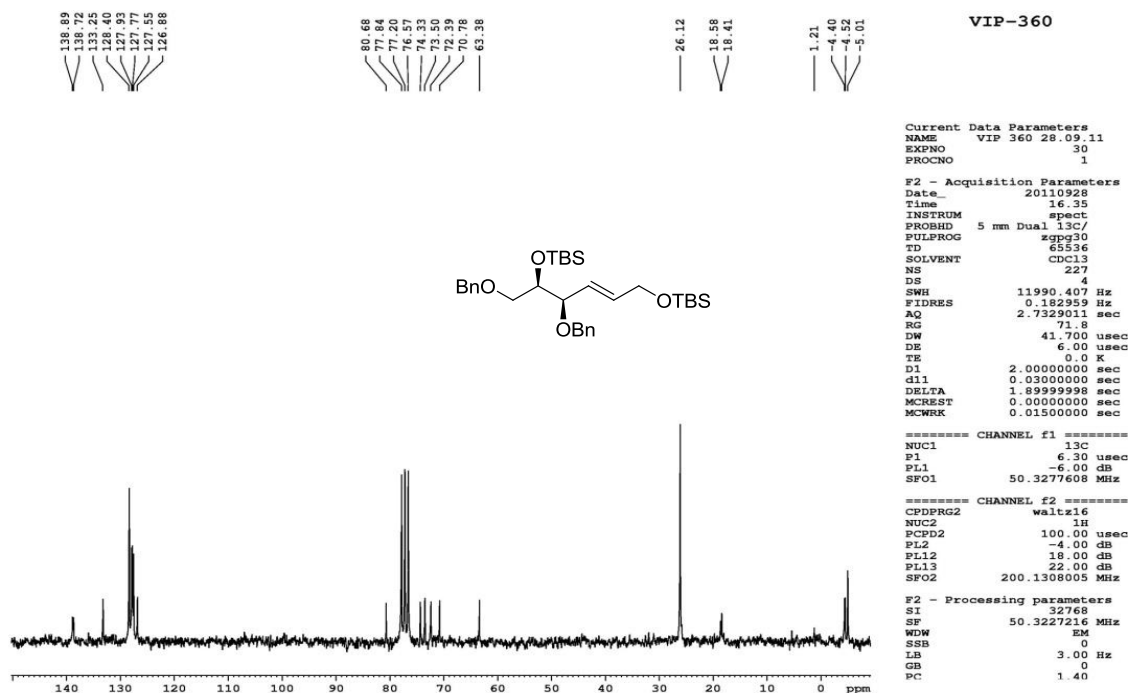
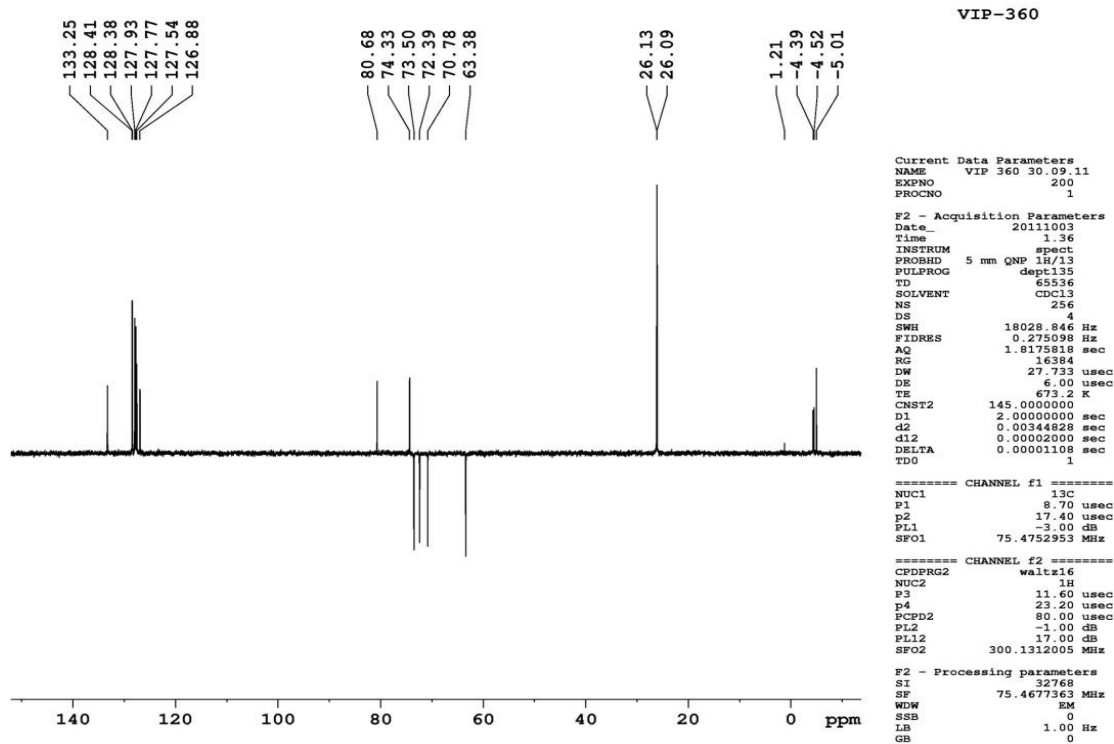
Contents	Page no.
1 Abbreviations	2
2 NMR Spectra of compounds 3-18	3-95
3 General methods for glycosidase inhibition studies	96
4 Table 1: List of enzymes, respective	96
substrates, buffers and optimum incubation temperatures.	
5 Table 2. Percentage of inhibition of 11 glycosidase by compounds 17a, 17b, 18a and 18b at 400 μ M and 1000 μ M	97
6 Michaelis-Menten plot of Activity versus Substrate concentration for inhibition of various enzymes	98-102
7. References	103

Abbreviations

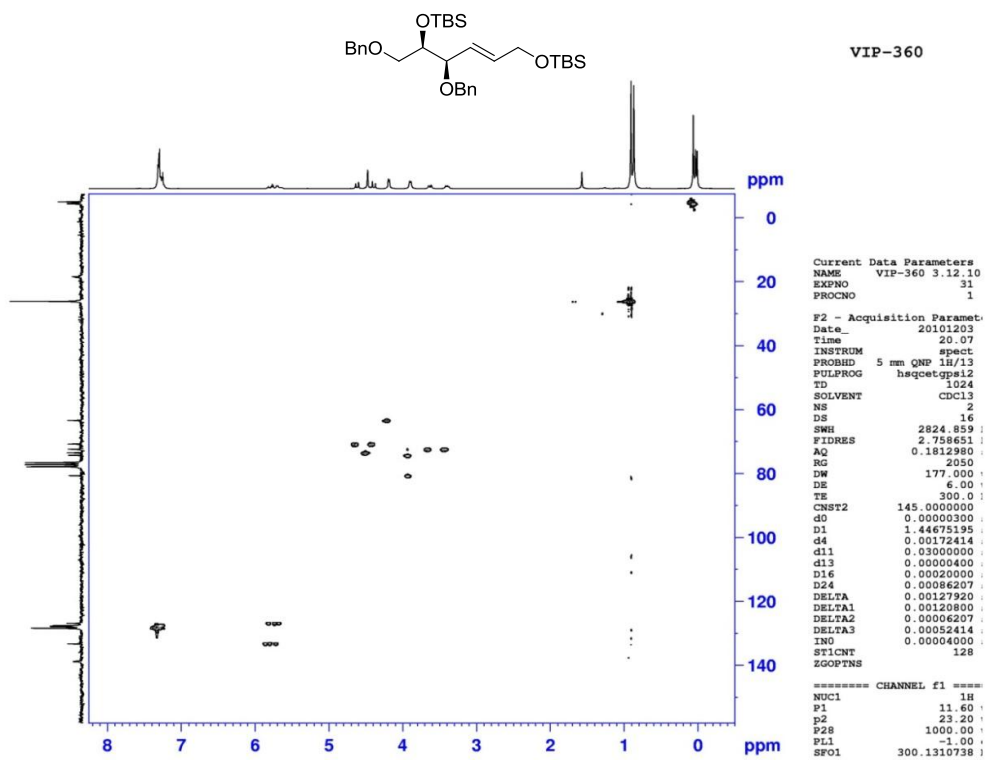
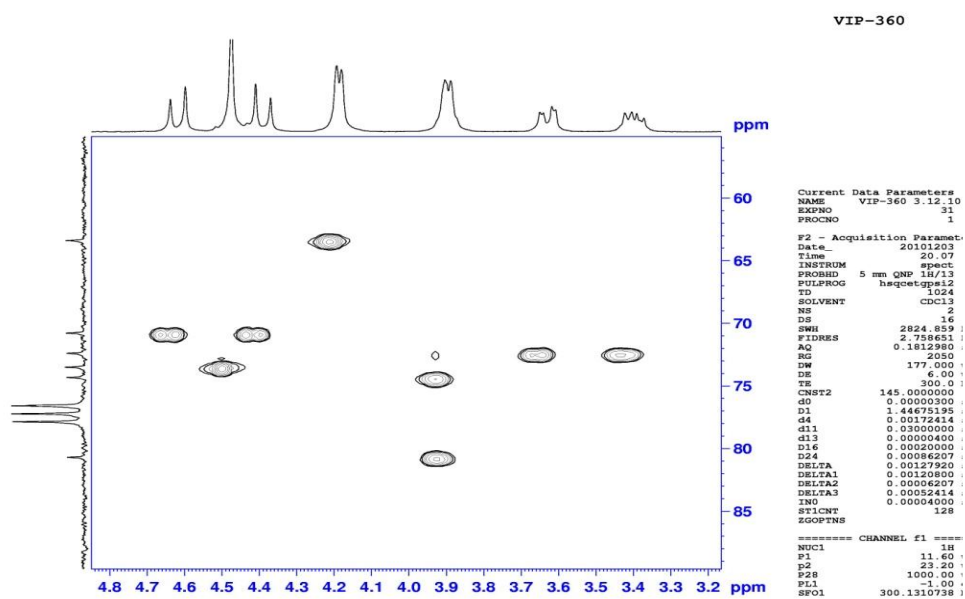
Bn	Benzyl
DCM	Dichloromethane
DIAD	Diisopropylazodicarboxylate
DPPA	Diphenylphosphoryl azide
DMF	Dimethylformamide
EtOAc	Ethyl acetate
EtOH	Ethanol
Eq	Molar equivalent(s)
NMR	Nuclear magnetic resonance
TBAF	Tetrabutylammonium fluoride
<i>t</i> -BuOOH	<i>tert</i> -Butyl hydroperoxide
TBSOTf	<i>tert</i> -Butyldimethylsilyl trifluoromethanesulfonate
THF	Tetrahydrofuran
TLC	Thin Layer chromatogram
TMS	Tetra methyl silane
<i>n</i> -BuLi	<i>n</i> -butyl lithium



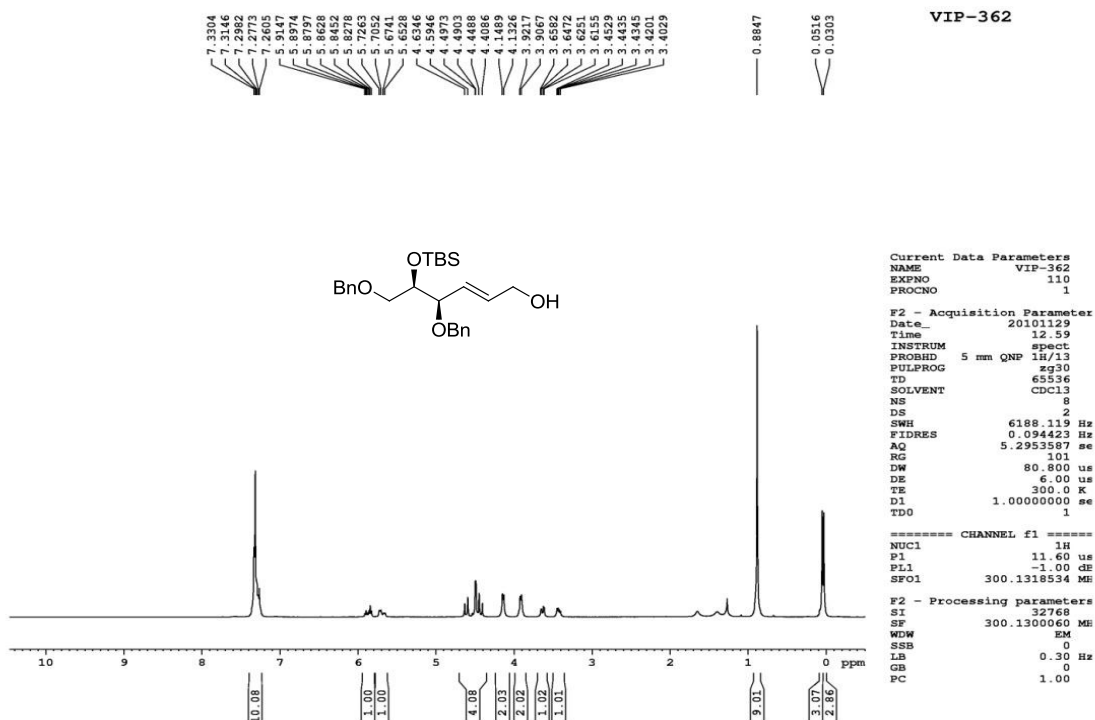
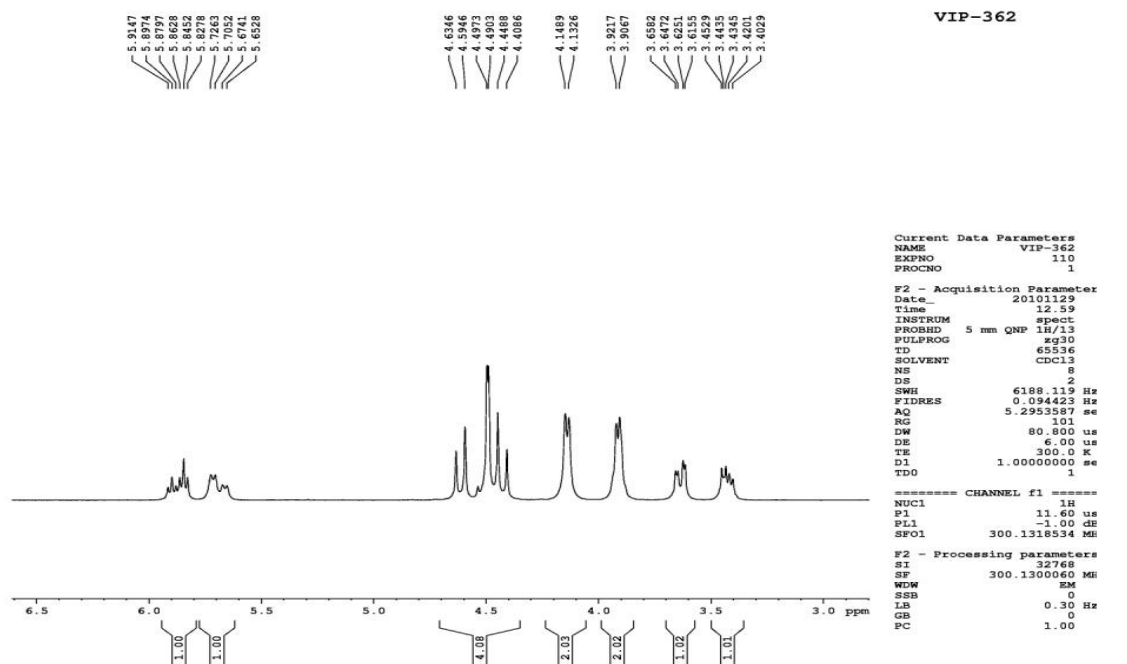
¹H NMR Spectrum of 3a



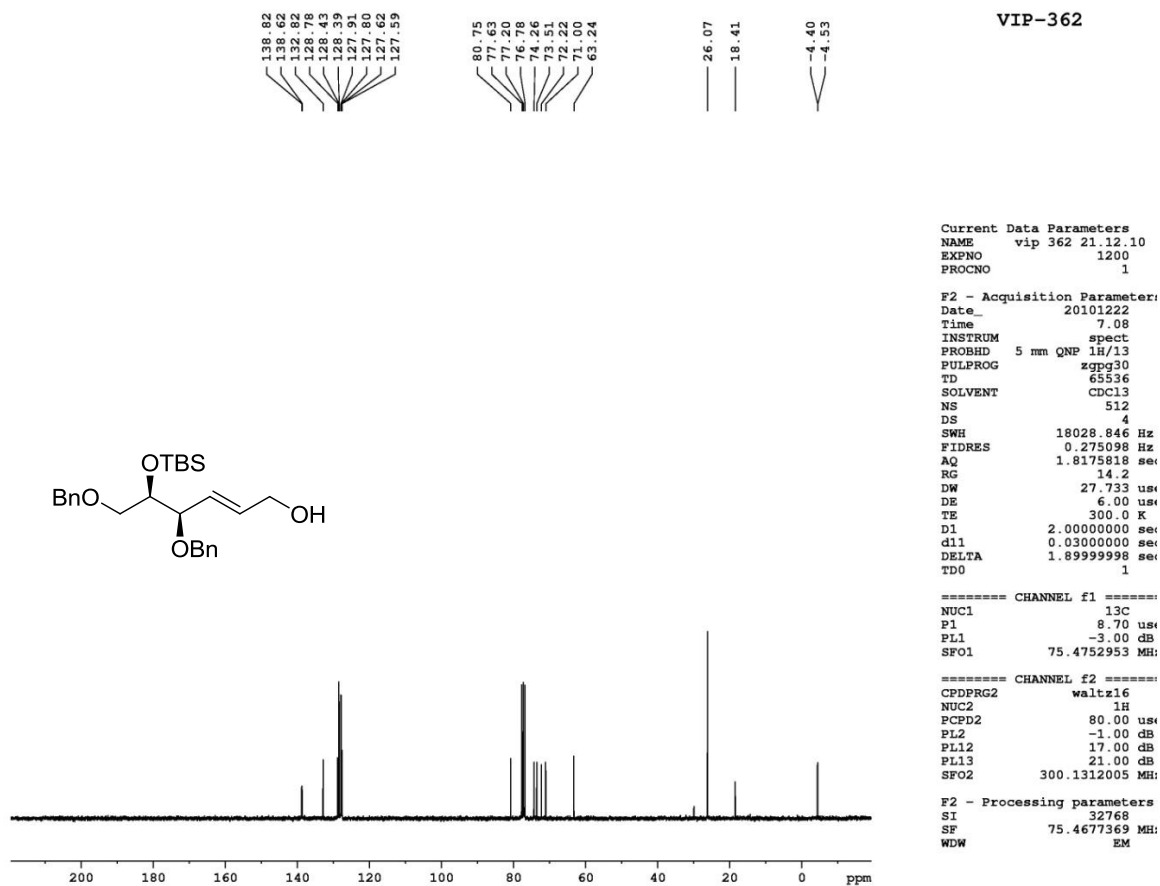
¹³C NMR Spectrum of 3a



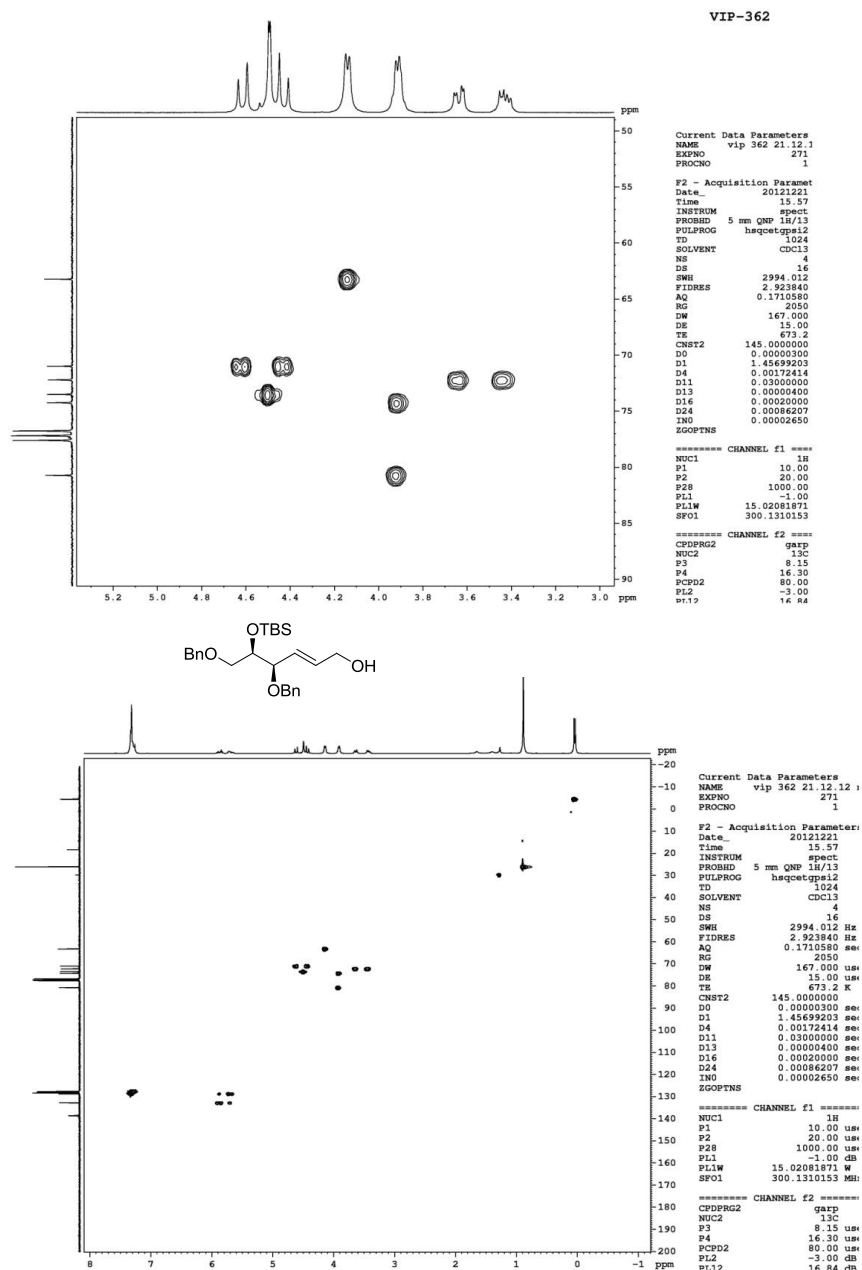
HSQC Spectra of 3a



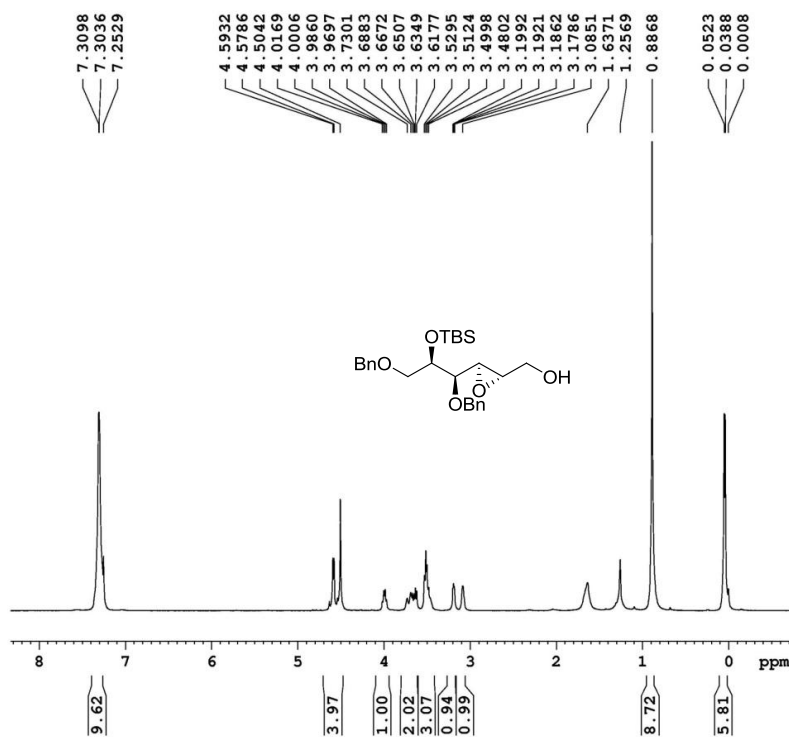
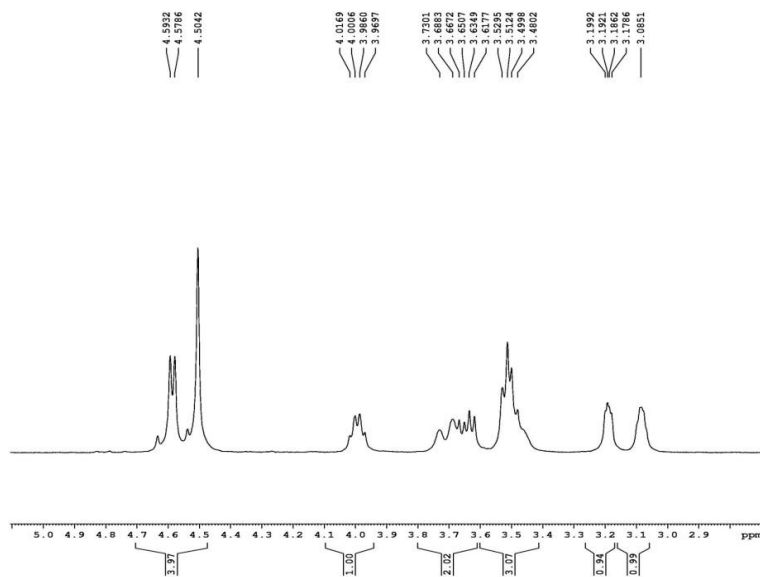
¹H NMR Spectrum of 4a



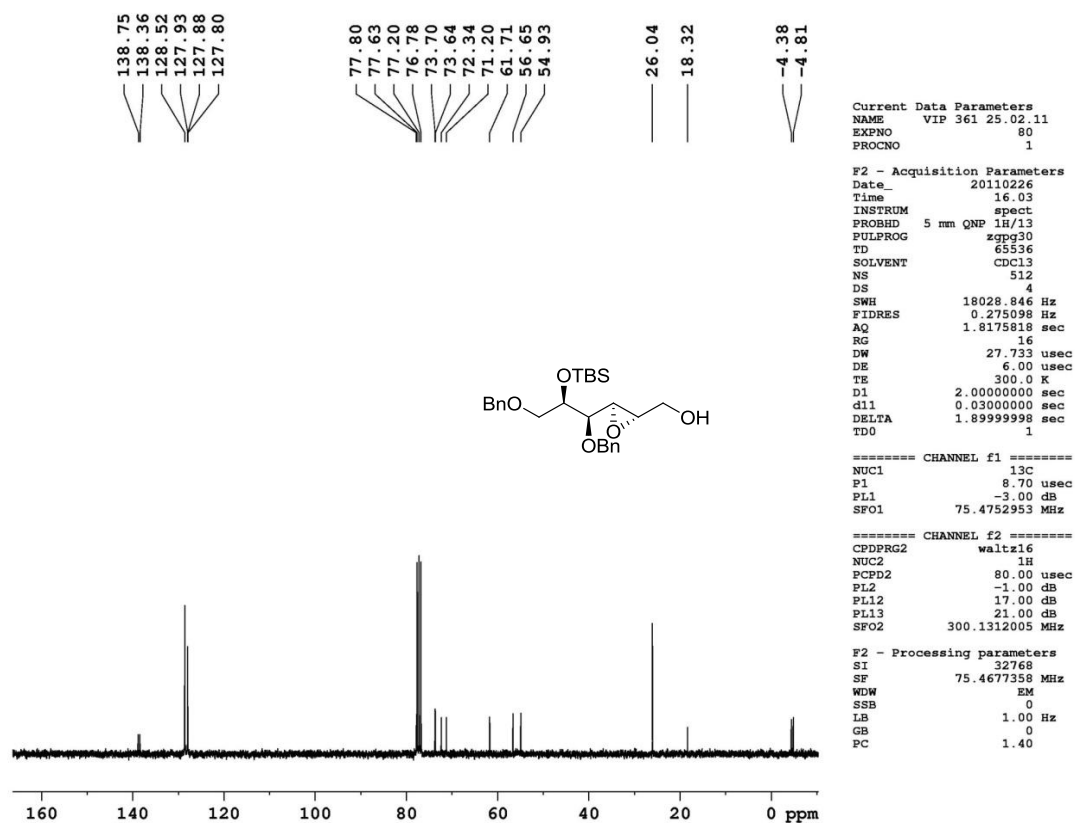
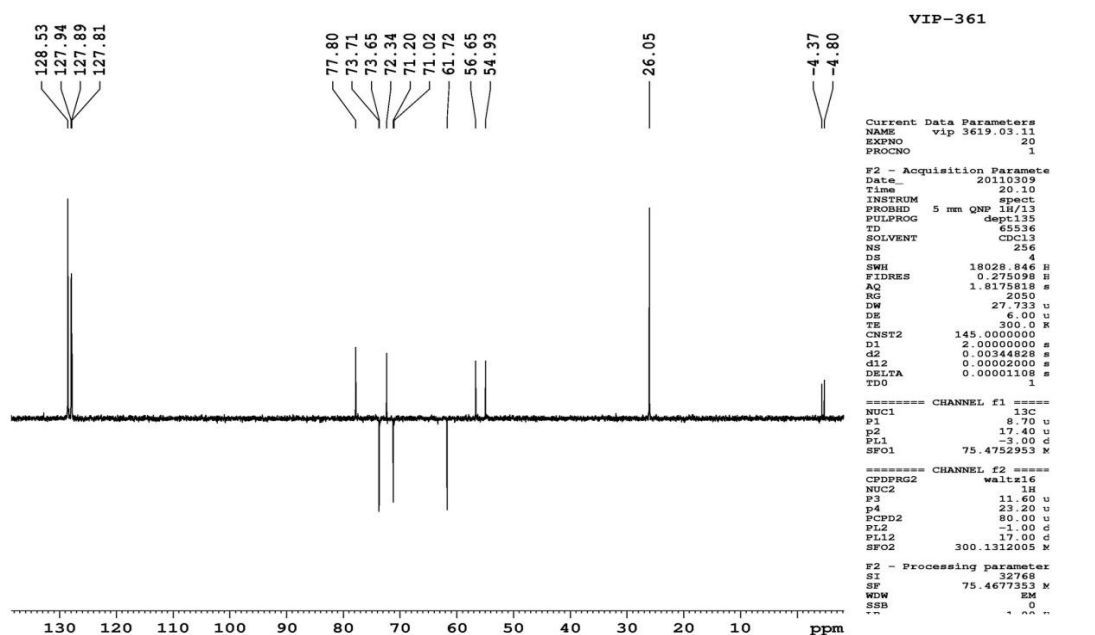
^{13}C NMR Spectrum of 4a



HSQC Spectra of 4a



¹H NMR Spectrum of 5a



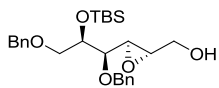
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OBn

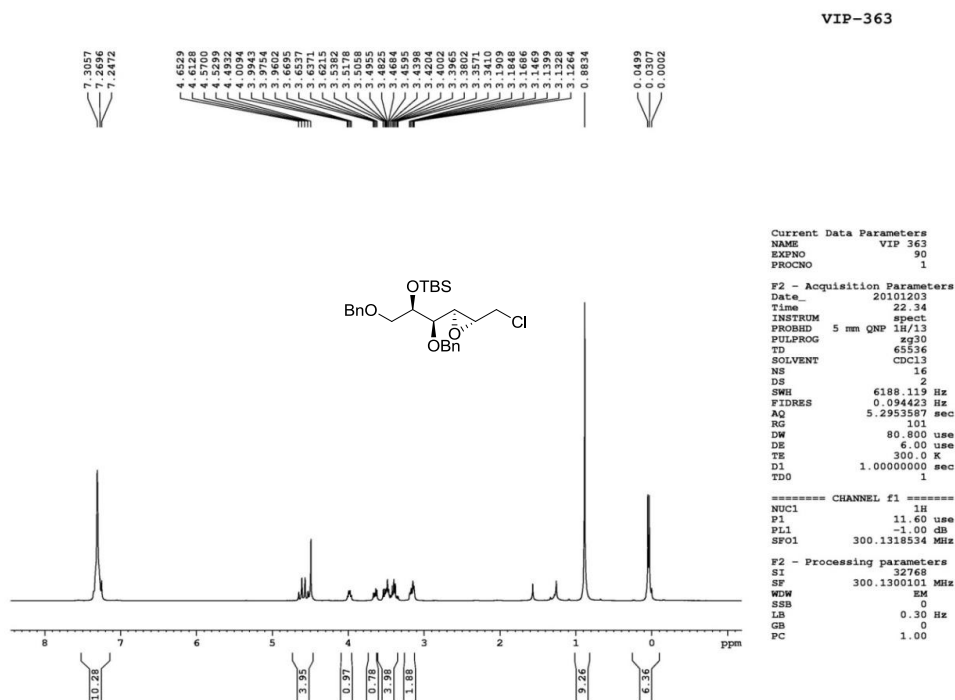
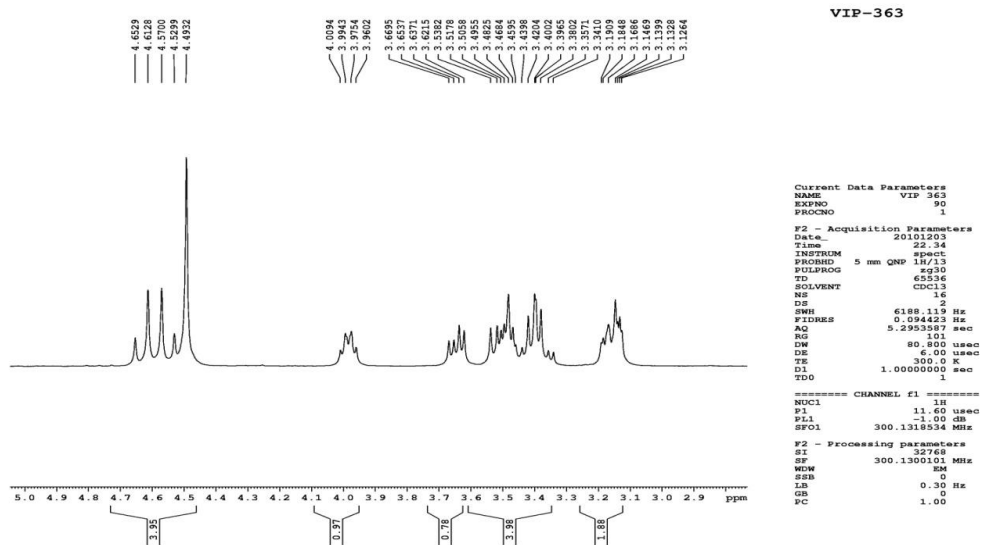
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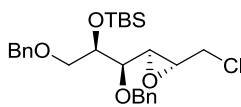
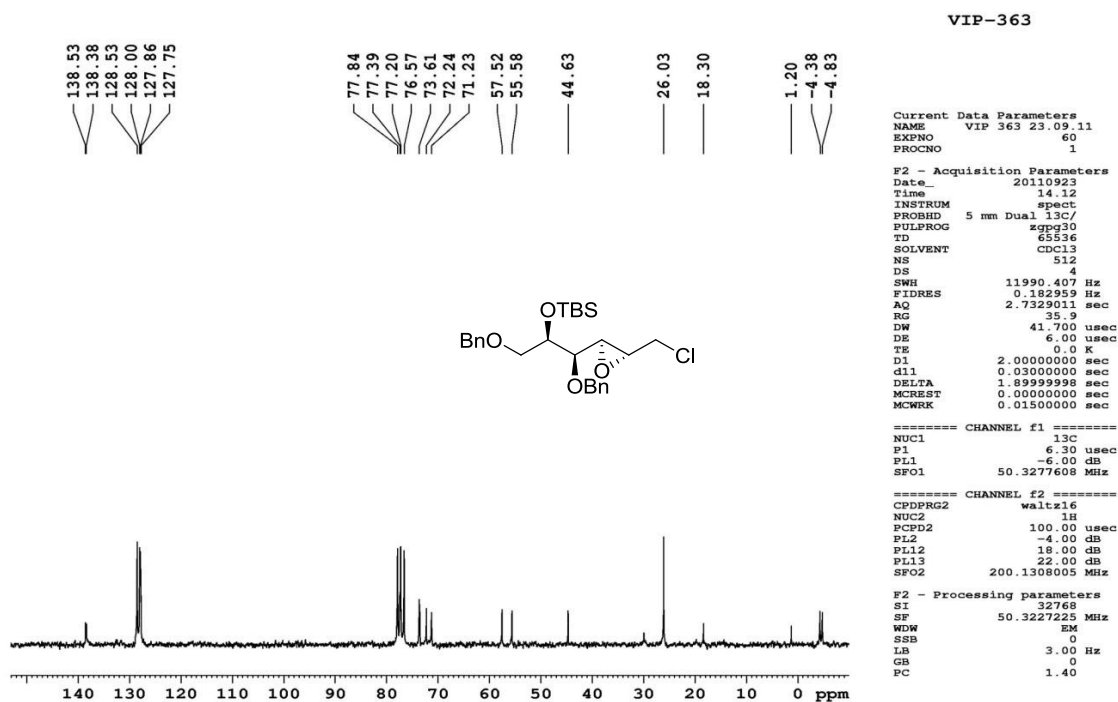
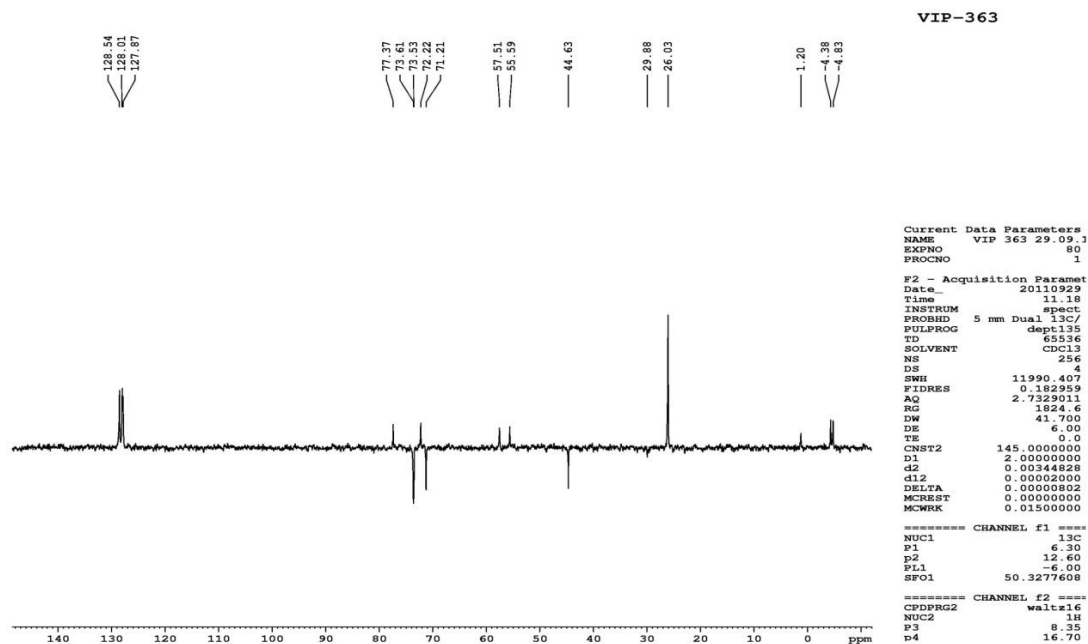
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 F2 24.70 usec
 F28 1000.00 usec

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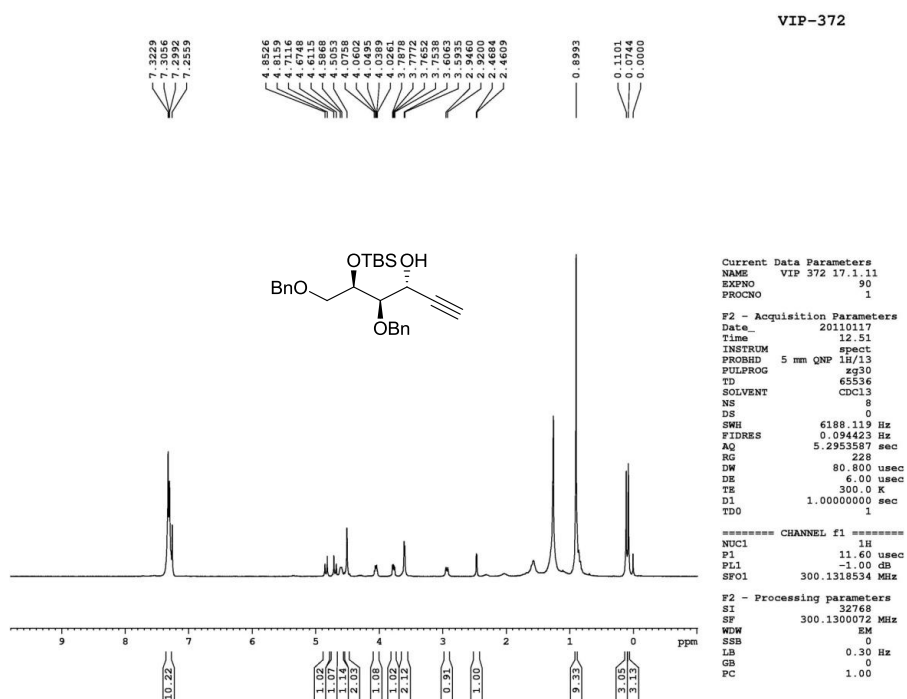
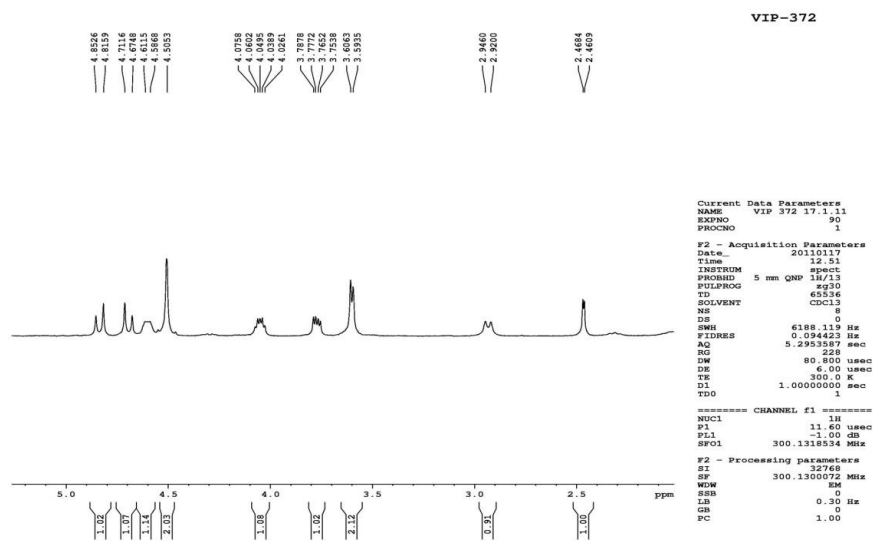
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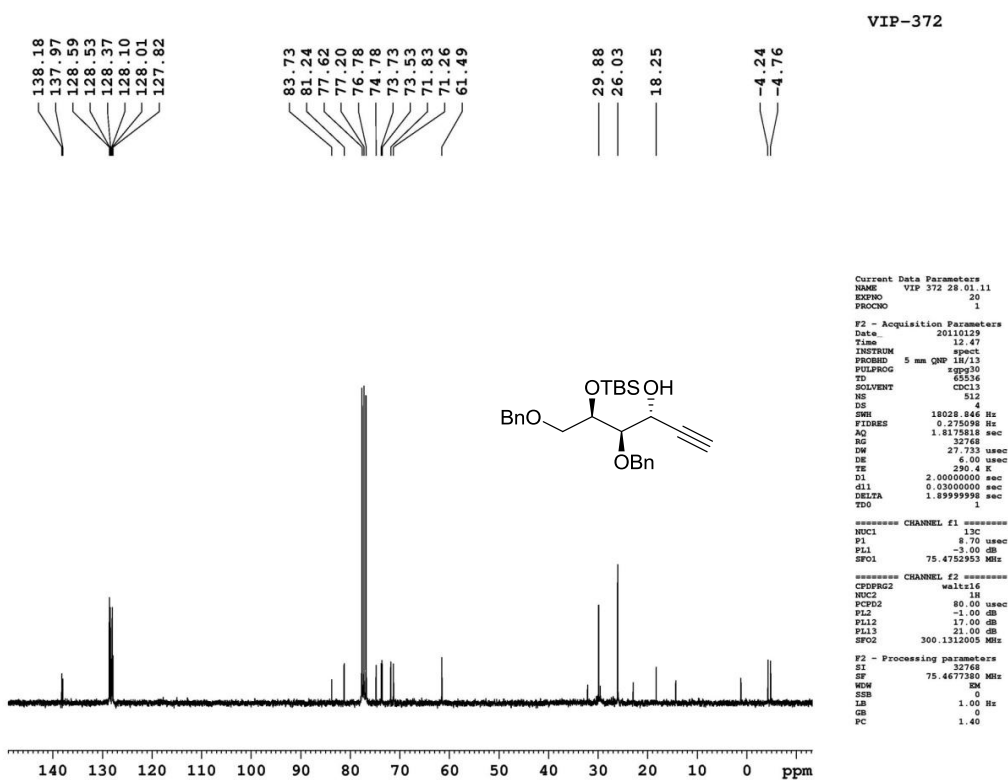
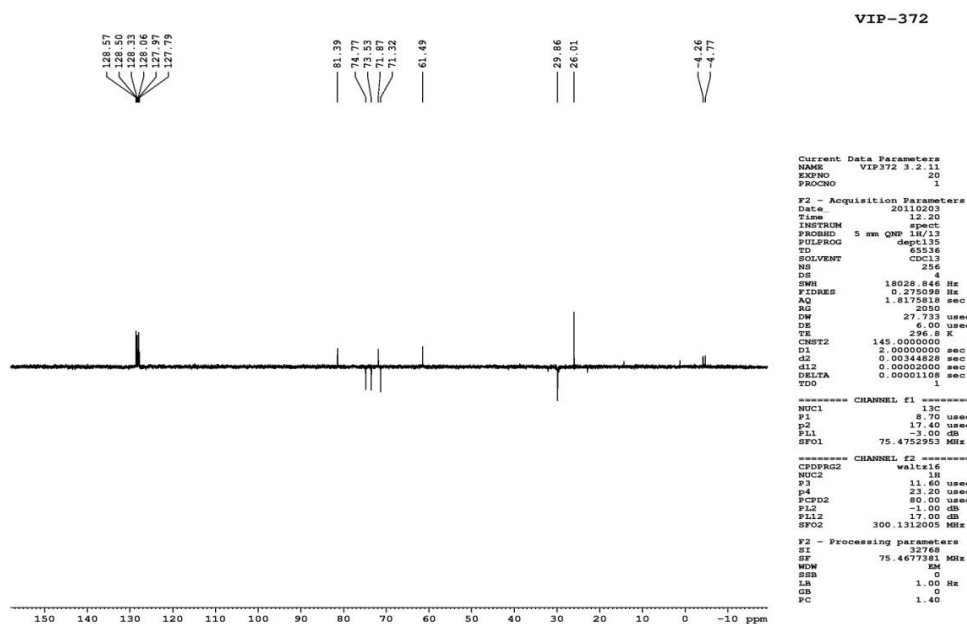
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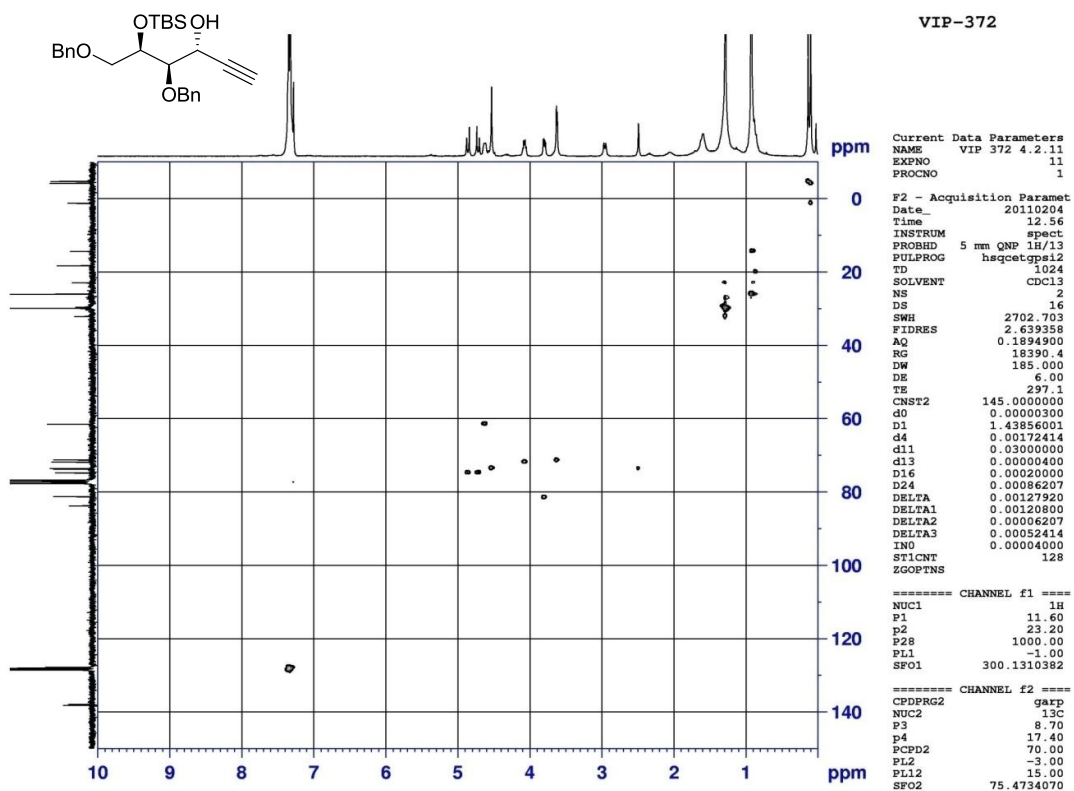
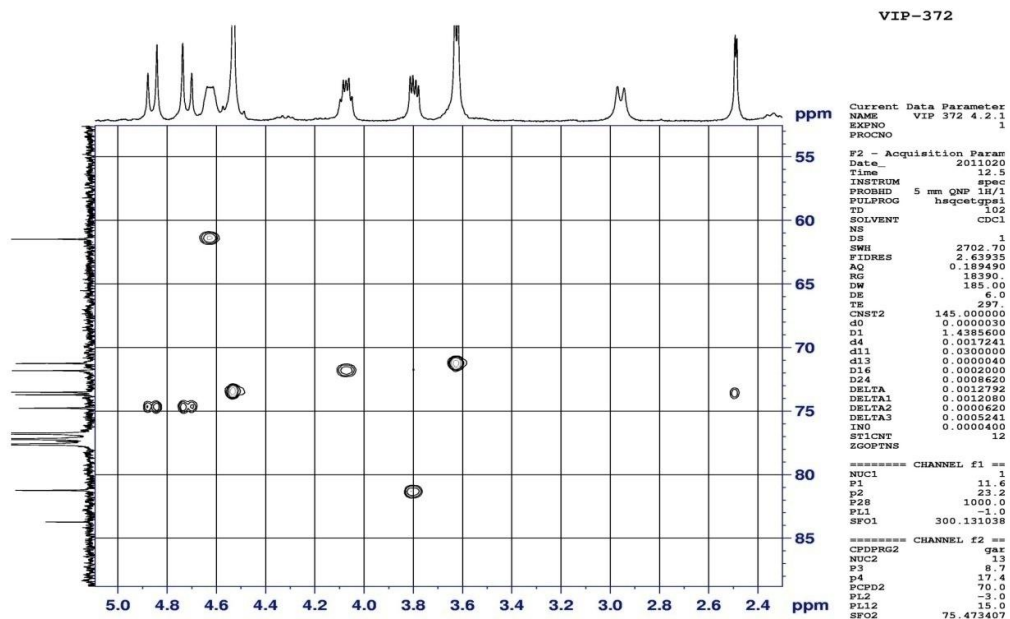
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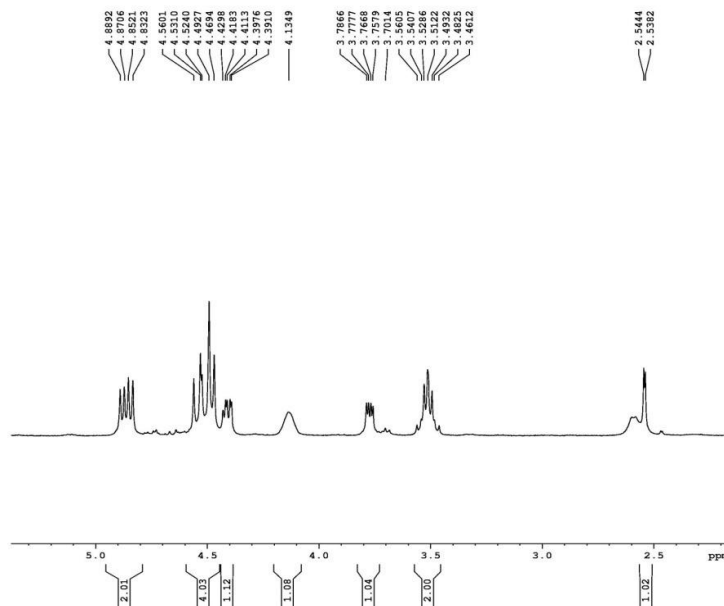
¹H NMR Spectrum of 7a



¹³C NMR Spectrum of 7a



HSQC Spectra of 7a



VIP-383

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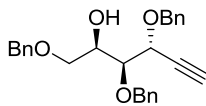
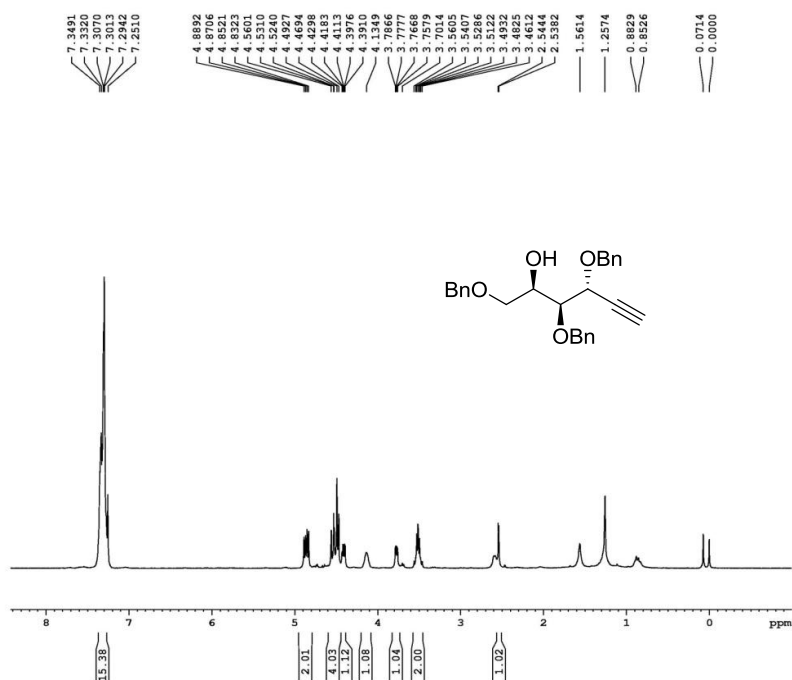
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RG         228
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VIP-383

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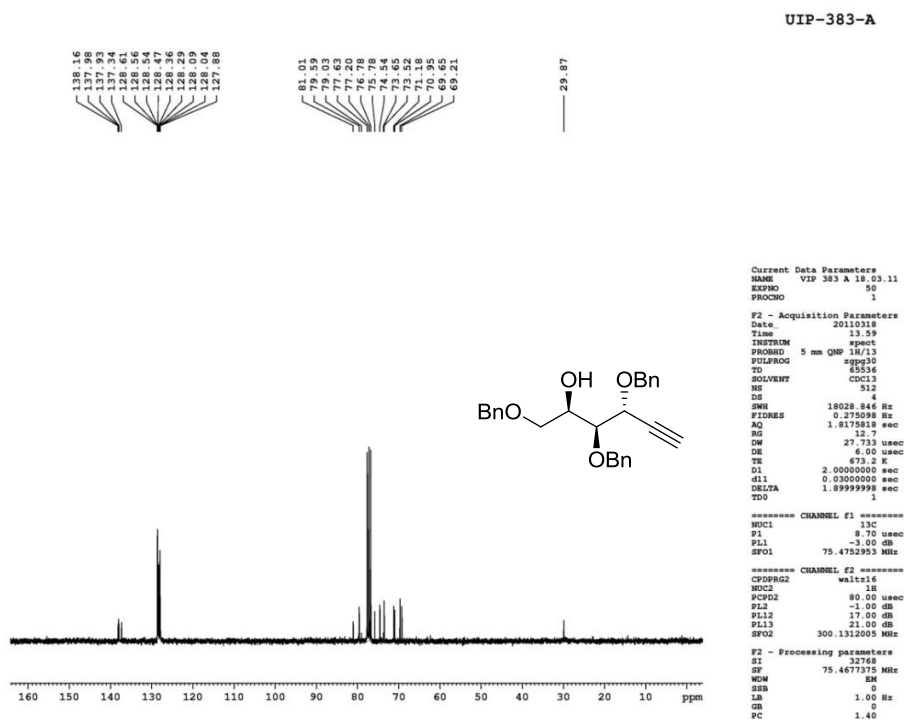
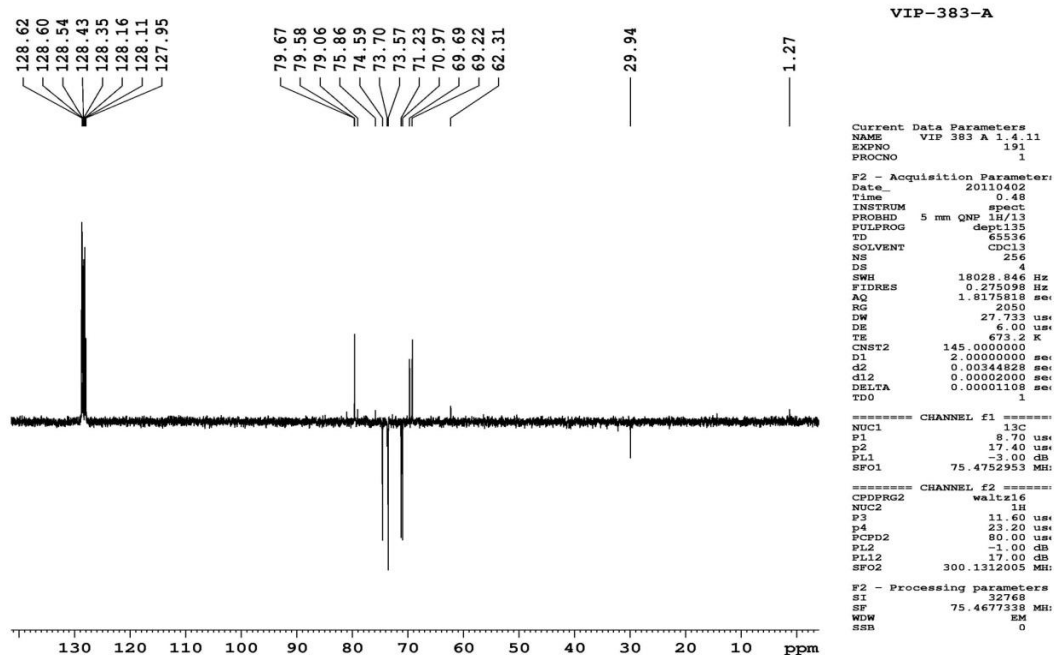
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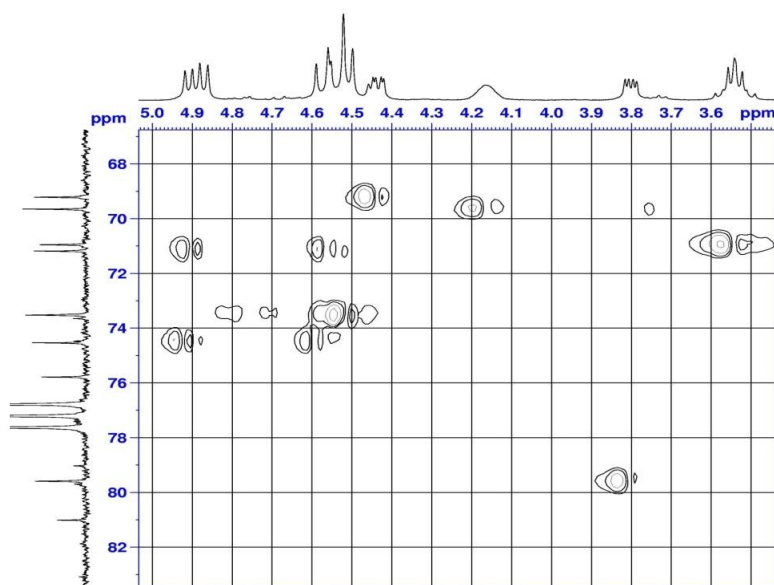
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¹H NMR Spectrum of 8a



¹³C NMR Spectrum of 8a



VIP-383-A

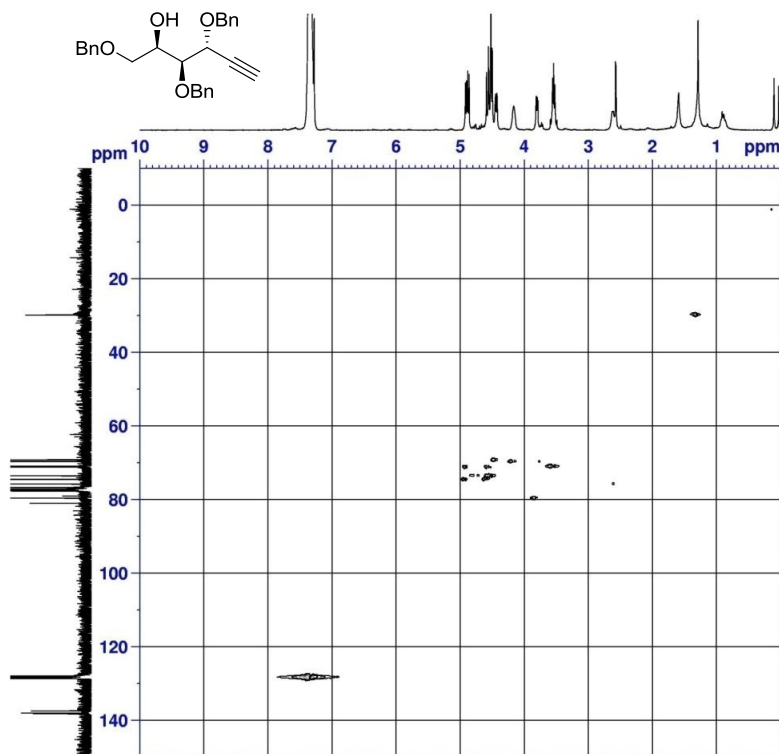
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d16 0.00020000 sec
D24 0.00086207 sec
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DELTA1 0.00120800 sec
DELTA2 0.0006207 sec
DELTA3 0.00052414 sec
IN0 0.00040000 sec
STICNT 128
ZGPTNS

===== CHANNEL f1 =====
NUC1 1H
P1 11.60 usec
P2 23.20 usec
P28 1000.00 usec
PL1 -1.00 dB
SFO1 300.1311387 MHz

===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P3 8.70 usec
P4 17.40 usec
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 15.00 dB
SFO2 75.4734070 MHz

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VIP-383-A

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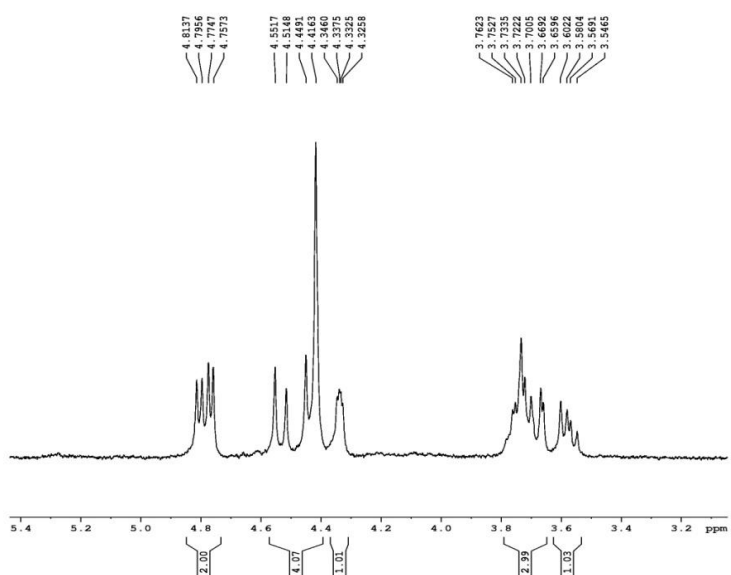
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NS 2
DS 16
SWH 2631.579 Hz
FIDRES 2.569901 Hz
AQ 0.1946100 sec
RG 2050
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DE 6.00 usec
TE 300.0 K
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ZGPTNS

===== CHANNEL f1 =====
NUC1 1H
P1 11.60 usec
P2 23.20 usec
P28 1000.00 usec
PL1 -1.00 dB
SFO1 300.1311387 MHz

===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P3 8.70 usec
P4 17.40 usec
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 15.00 dB
SFO2 75.4734070 MHz

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GPNAM2 SINE 100
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HSQC Spectra of 8a



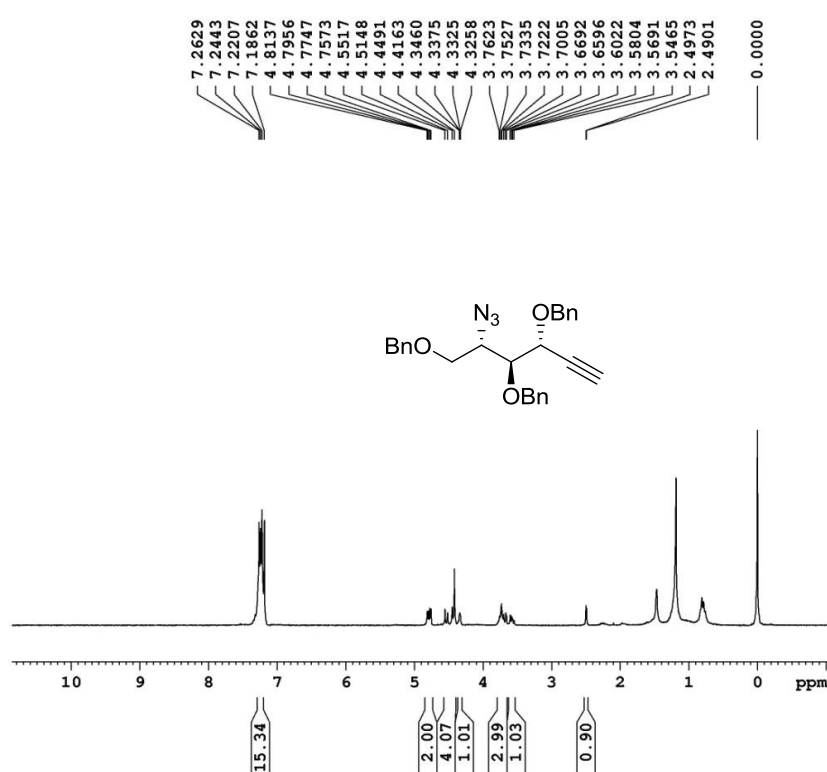
VIP-384

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SWH        6188.1
FIDRES     0.0944
AQ          5.29535
RG          322
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DE          6.0
TE          300
D1          1.000000
TDO         0

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VIP-384

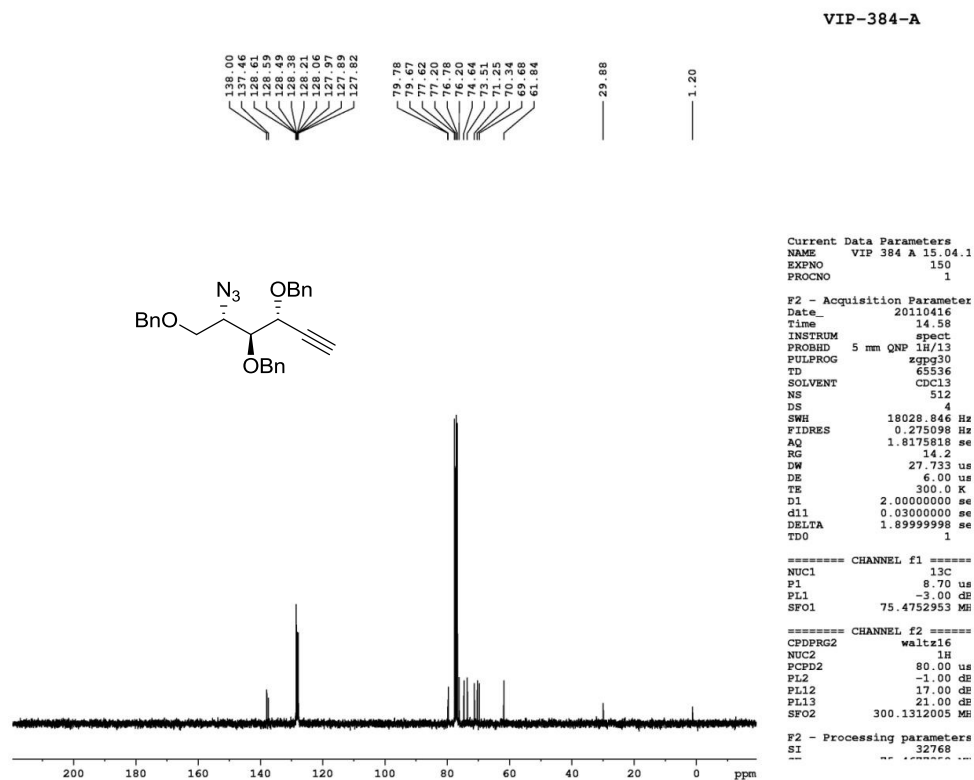
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FIDRES     0.094423 Hz
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RG          322
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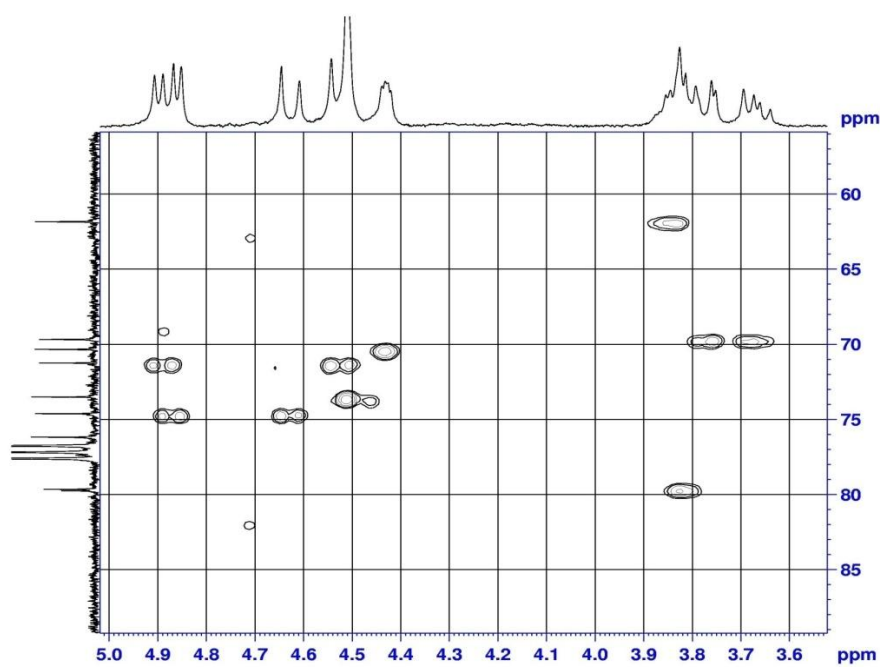
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¹H NMR Spectrum of 9a

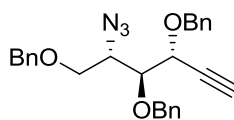


^{13}C NMR Spectrum of 9a

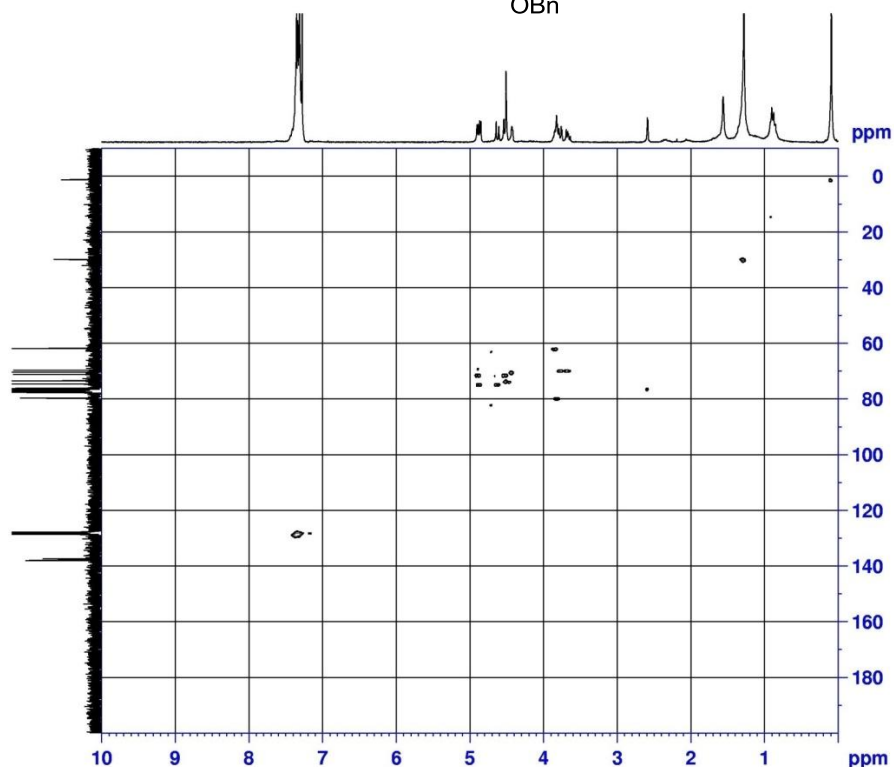
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 TD 65536
 SOLVENT cdcl3
 NS 1024
 DS 4
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 FIDRES 2.631
 AQ 0.1894
 RG 183
 DW 185
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 TE 300.2
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 P1 12.00
 P2 2.00
 P28 100.00
 PL1 -1.00
 SFO1 300.131
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 NUC2 1H
 P3 1.00
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 PCPD2 7.00
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 PL12 1.00
 SFO2 75.4734
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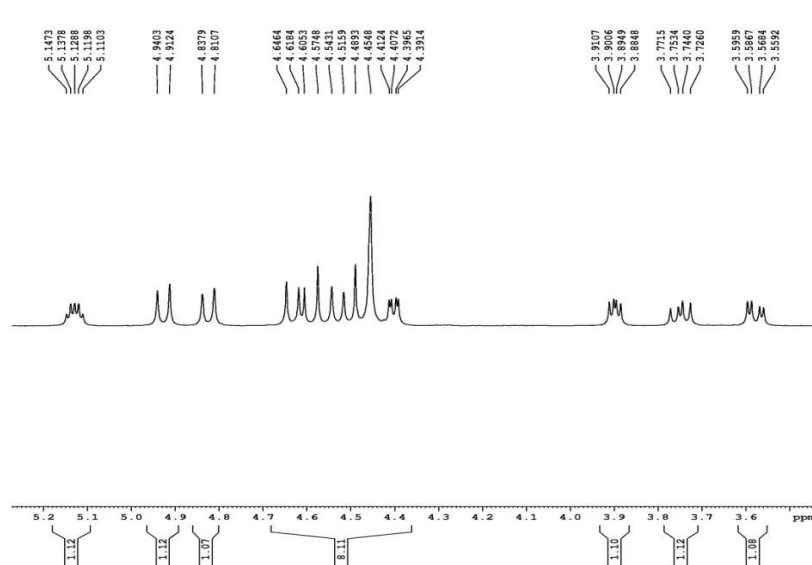


VIP-384-A



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 SWH 2702.143
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 AQ 0.1894
 RG 183
 DW 185
 DE 1.00
 TE 300.2
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 d0 0.0000
 d1 1.43854
 d4 0.00172
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 D16 0.00020
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 DELTA2 0.00004
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 ZGPGTNS 1
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 P2 2.00
 P28 100.00
 PL1 -1.00
 SFO1 300.131
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 NUC2 1H
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 P4 1.00
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 PL12 1.00
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 ===== GRADIENT CHANNELS
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HSQC Spectra of 9a



VIP-9C

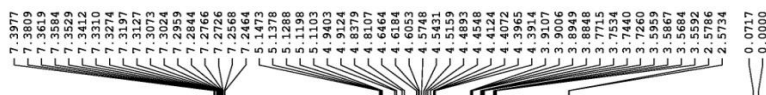
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PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         89.7
DW         62.400 usec
DE         6.50 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      400.1629712 MHz
NUC1       1H
P1         12.35 usec

F2 - Processing parameters
SI         65536
SF         400.1605153 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



VIP-9C

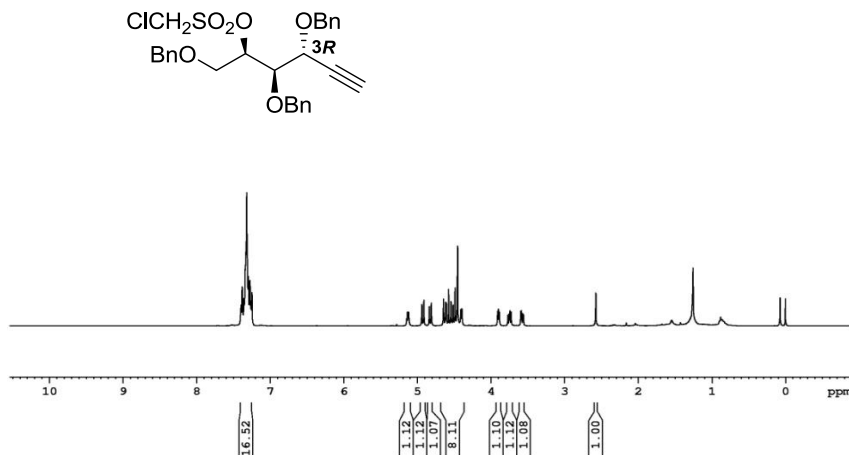
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Current Data Parameters
NAME      vip 9c hsqc
EXPNO     100
PROCNO    1

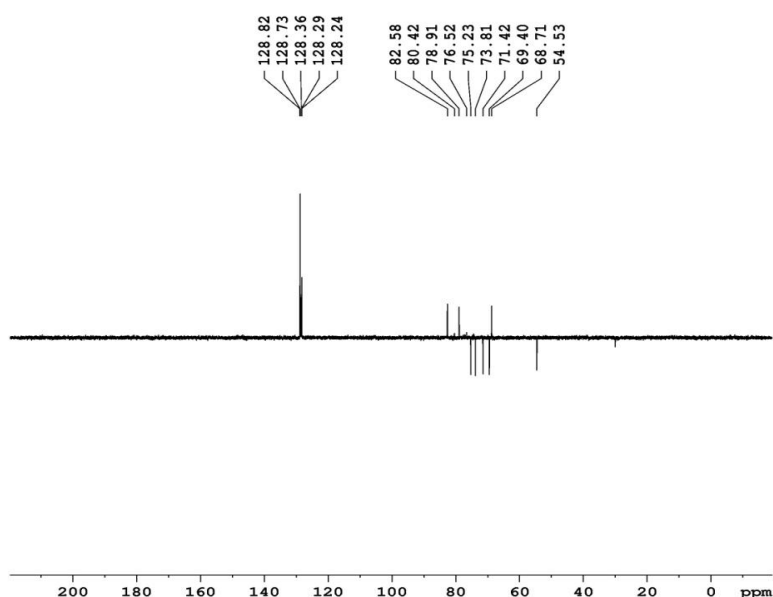
F2 - Acquisition Parameters
Date_     20130130
Time      21.31
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         89.7
DW         62.400 usec
DE         6.50 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      400.1629712 MHz
NUC1       1H
P1         12.35 usec

F2 - Processing parameters
SI         65536
SF         400.1605153 MHz
WDW        EM
SSB        0
LB         0.30 Hz
  
```



¹H NMR Spectrum of 9c



VIP-9C

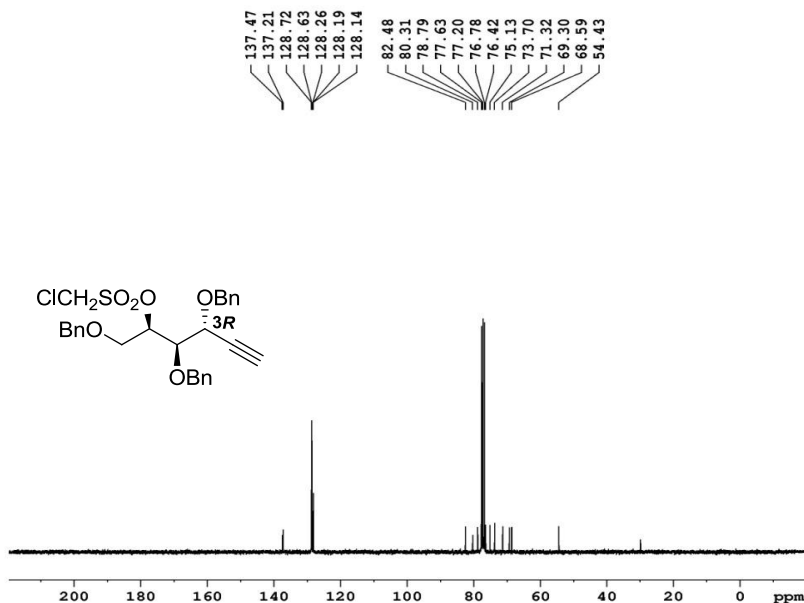
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Current Data Parameters
NAME    VIP 9c 21.12.12
EXPNO   240
PROCNO   1

F2 - Acquisition Parameters
Date_    20121221
Time     15.15
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        256
DS        4
SWH       18028.846 Hz
FIDRES    0.275098 Hz
AQ         1.8175818 sec
RG         2050
DW         27.733 usec
DE         15.00 usec
TE         673.2 K
CNET2     145.000000 sec
D1         2.0000000 sec
D2         0.0344828 sec
D12        0.0002000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1         8.15 usec
P2         16.30 usec
PL1        -3.00 dB
PL1W       55.13059616 W
SFO1       75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
P3         10.00 usec
P4         20.00 usec
PCPD2      80.00 usec
PL2        -1.00 dB
PL12       17.06 dB
PL2W       15.02081871 W
PL12W      0.23479761 W
SFO2       300.1312005 MHz

F2 - Processing parameters
SI         32768
SF         75.4677281 MHz
WDW         RM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
```



VIP-9C

```
Current Data Parameters
NAME    VIP 9c 20.12.12
EXPNO   40
PROCNO   1

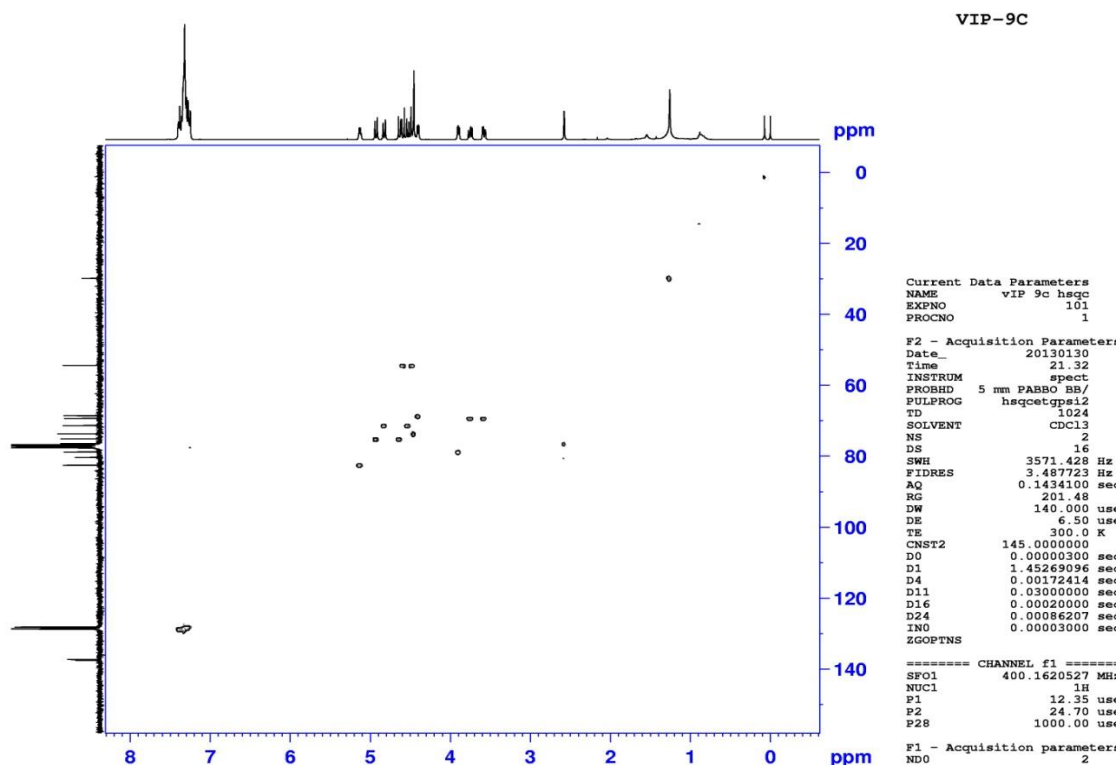
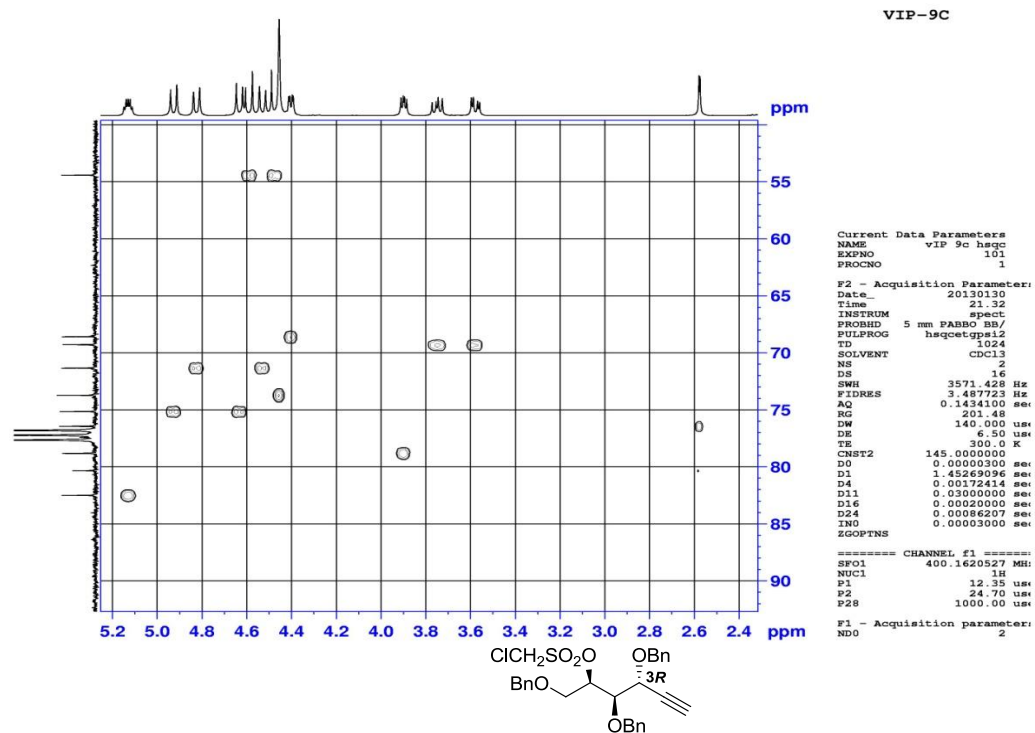
F2 - Acquisition Parameters
Date_    20121221
Time     4.29
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        512
DS        4
SWH       18028.846 Hz
FIDRES    0.275098 Hz
AQ         1.8175818 sec
RG         2050
DW         27.733 usec
DE         15.00 usec
TE         673.2 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1         8.15 usec
P2         16.30 usec
PL1        -3.00 dB
PL1W       55.13059616 W
SFO1       75.4752953 MHz

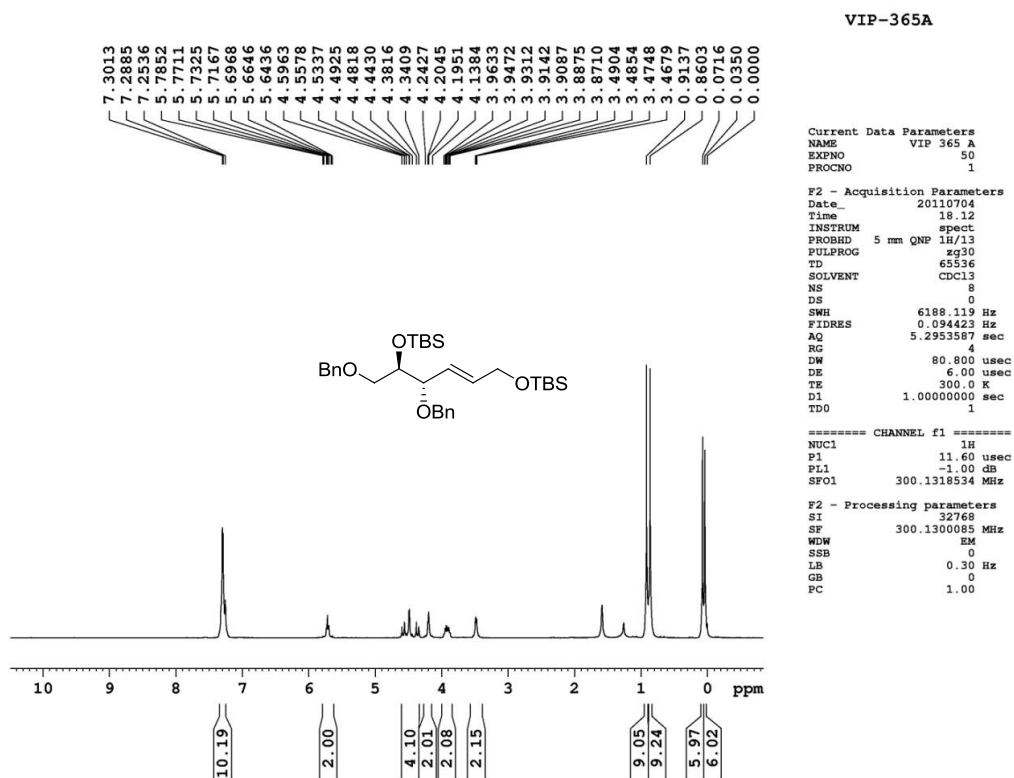
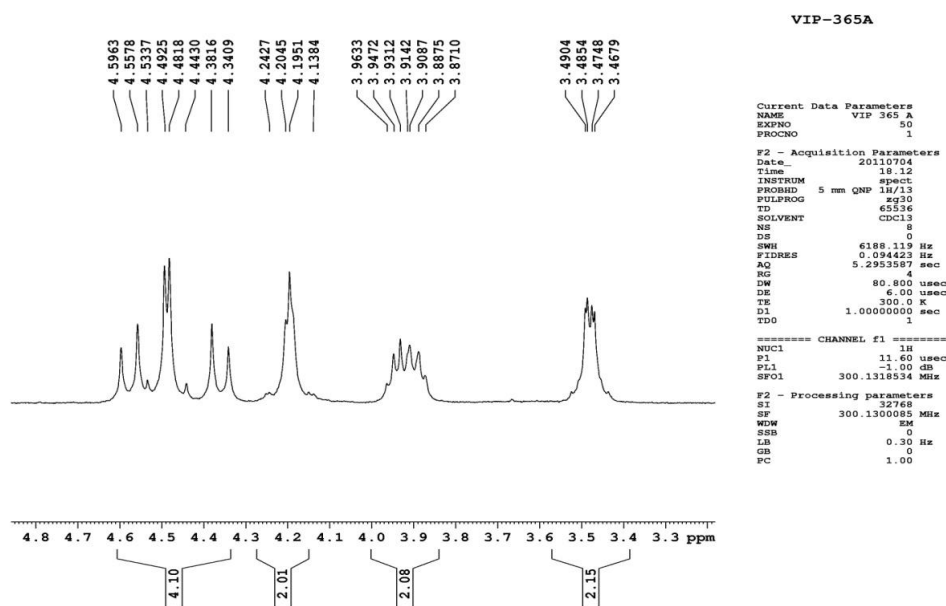
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -1.00 dB
PL12       17.06 dB
PL13       21.00 dB
PL2W       15.02081871 W
PL12W      0.23479761 W
PL13W      0.09477496 W
SFO2       300.1312005 MHz

F2 - Processing parameters
SI         32768
SF         75.4677360 MHz
WDW         RM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
```

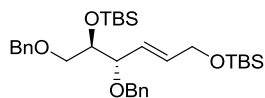
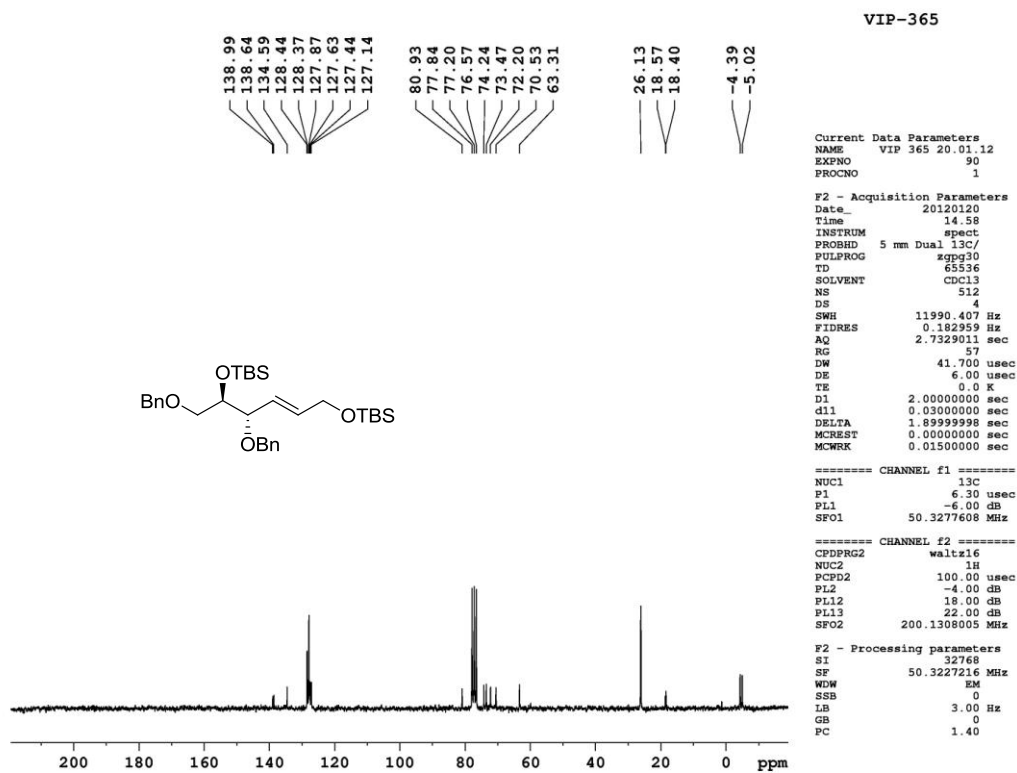
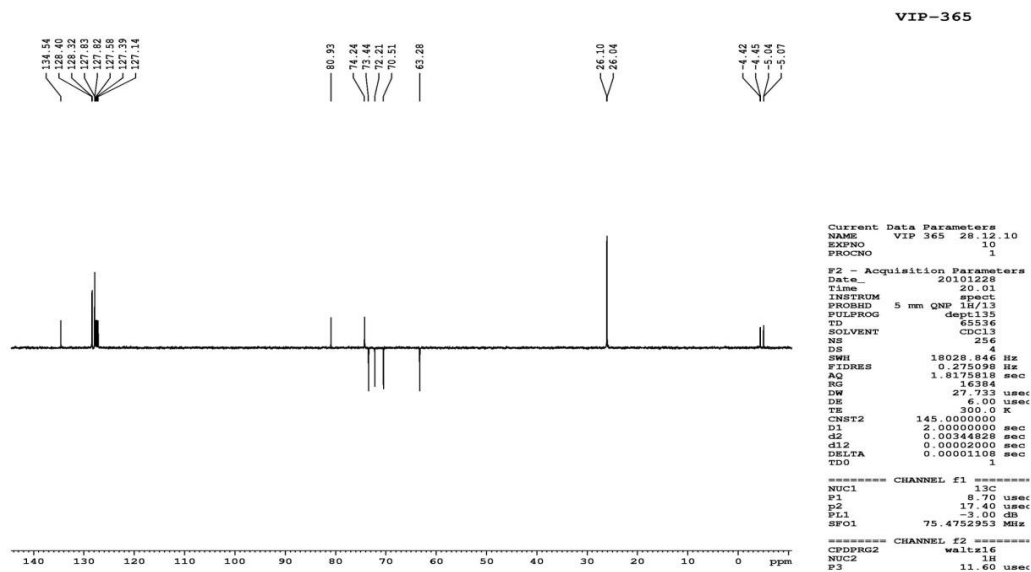
¹³C NMR Spectrum of 9c



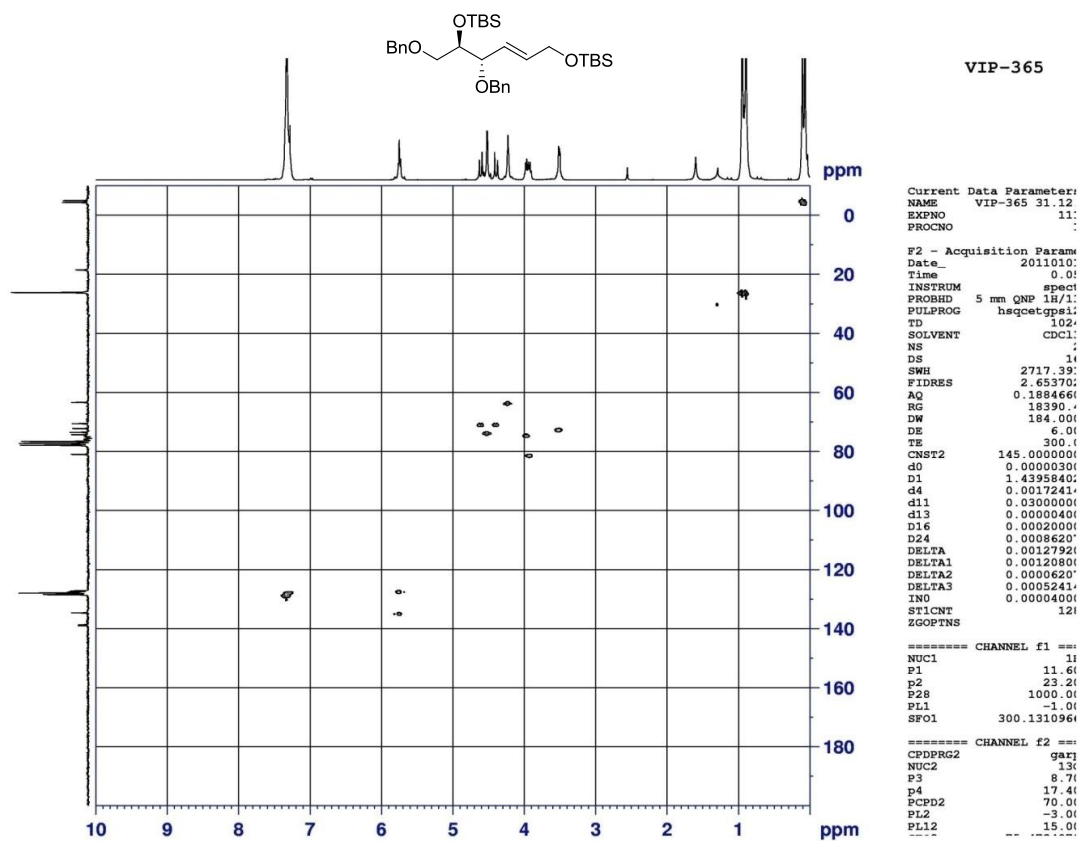
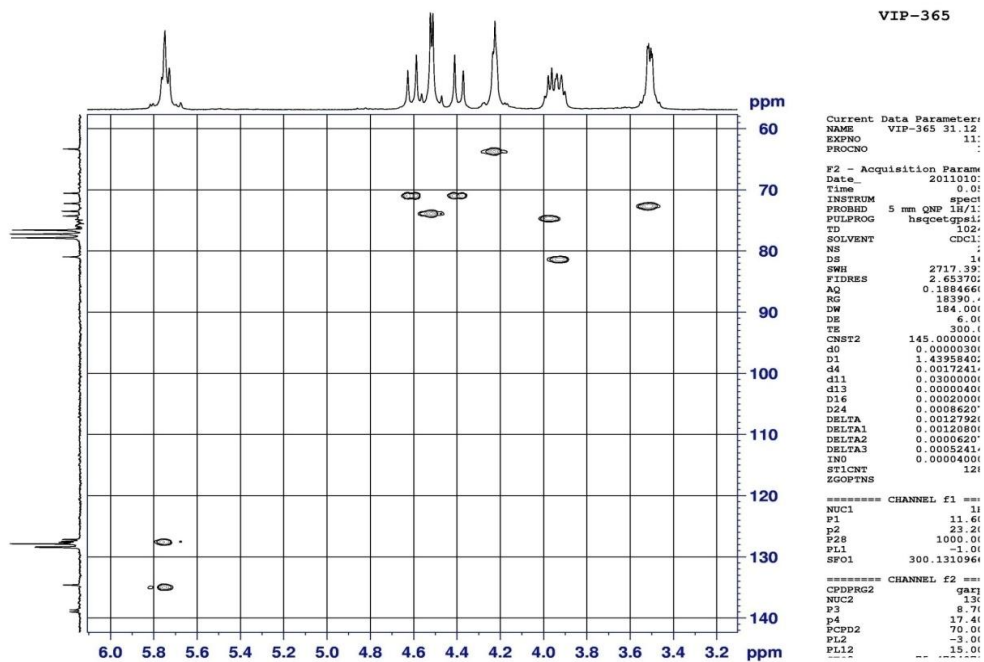
HSQC Spectra of 9c



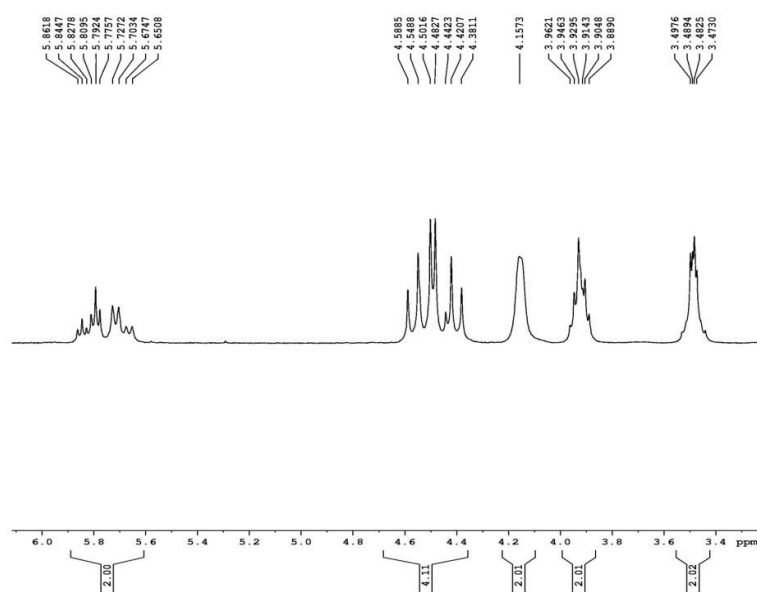
¹H NMR Spectrum of 3b



¹³C NMR Spectrum of 3b



HSQC Spectra of 3b



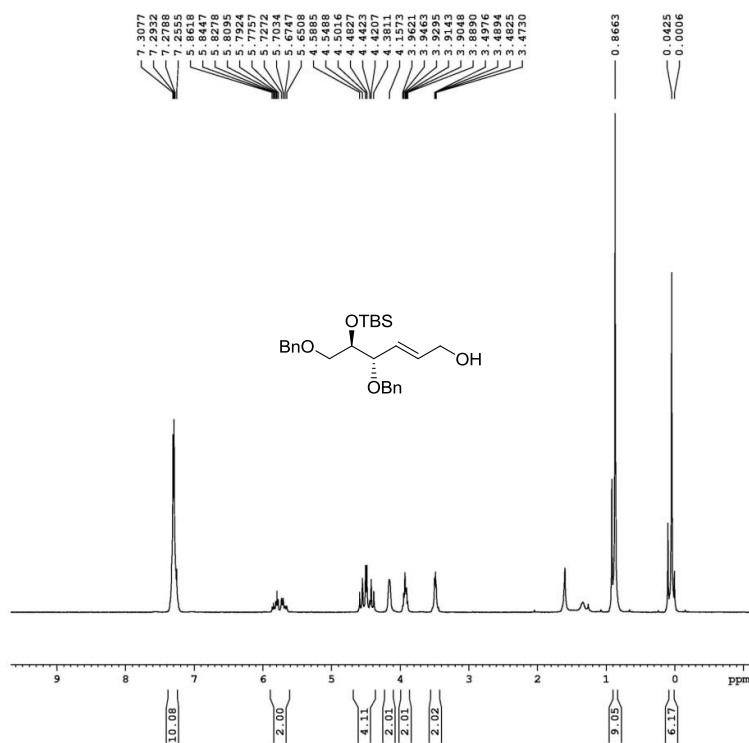
VIP-366

```
Current Data Parameters
NAME      vip 366 13.07.11
EXPNO     200
PROCNO    1

F2 - Acquisition Parameters
Date_     20110713
Time      18.37
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        6188.119 Hz
FIDRES     0.094423 Hz
AQ         5.2953587 sec
RG         181
DW         80.800 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         11.60 usec
PL1        -1.00 dB
SFO1       300.1318534 MHz

F2 - Processing Parameters
SI         32768
SF         300.1300079 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```



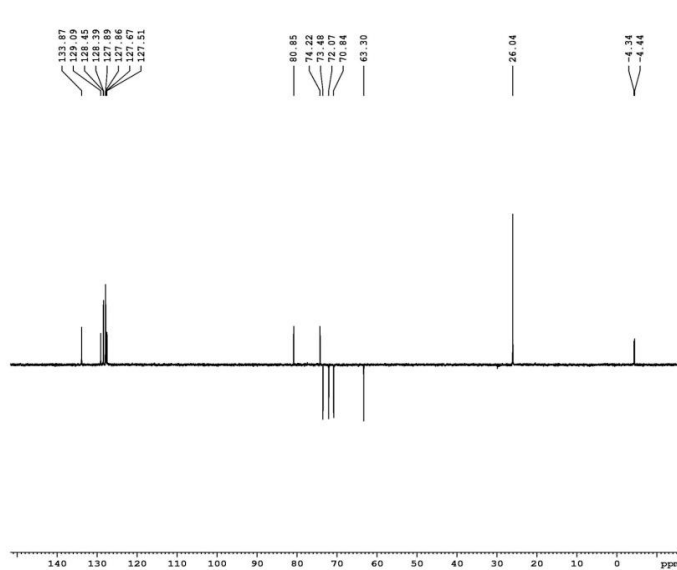
```
Current Data Parameters
NAME      vip 366 13.07.11
EXPNO     200
PROCNO    1

F2 - Acquisition Parameters
Date_     20110713
Time      18.37
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        6188.119 Hz
FIDRES     0.094423 Hz
AQ         5.2953587 sec
RG         181
DW         80.800 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         11.60 usec
PL1        -1.00 dB
SFO1       300.1318534 MHz

F2 - Processing parameters
SI         32768
SF         300.1300079 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```

¹H NMR Spectrum of 4b

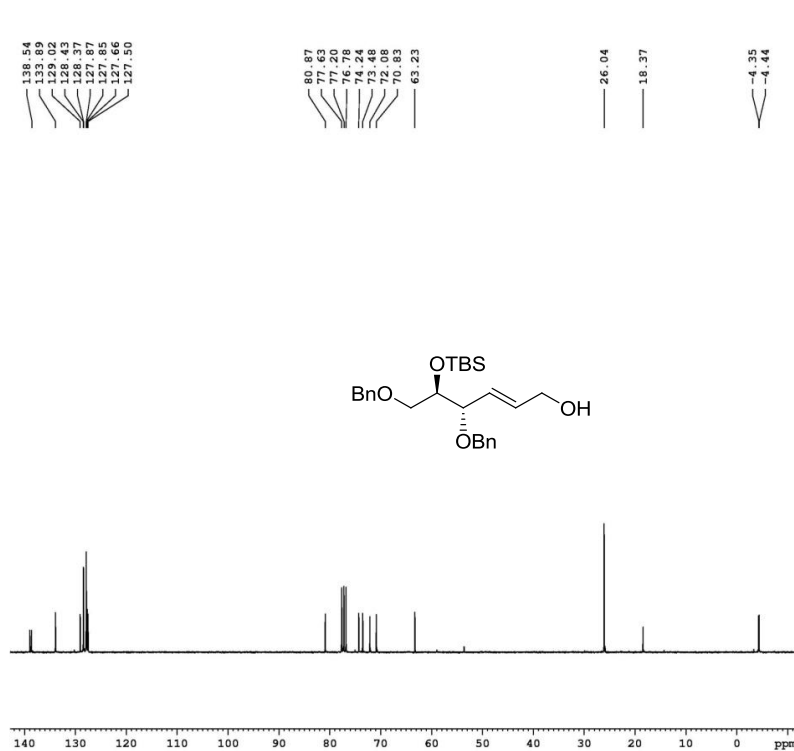


VIP-366

Current Data Parameters
NAME VIP 366 30.12.10
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101231
Time 2.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 18028.846 H
FIDRES 0.275098 H
AQ 1.8175818 s
RG 16384
DW 27.733 u
DE 6.00 u
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 s
d2 0.00344828 s
d12 0.00002000 s
DELTA 0.00001108 s
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.70 u
P2 17.40 u
PL1 -3.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 11.60 u
P4 23.20 u
PCPD2 80.00 u
PL2 -1.00 dB
PL3 17.00 dB
SFO2 300.1312005 MHz



VIP-366

Current Data Parameters
NAME VIP 366 30.12.11
EXPNO 130
PROCNO 1

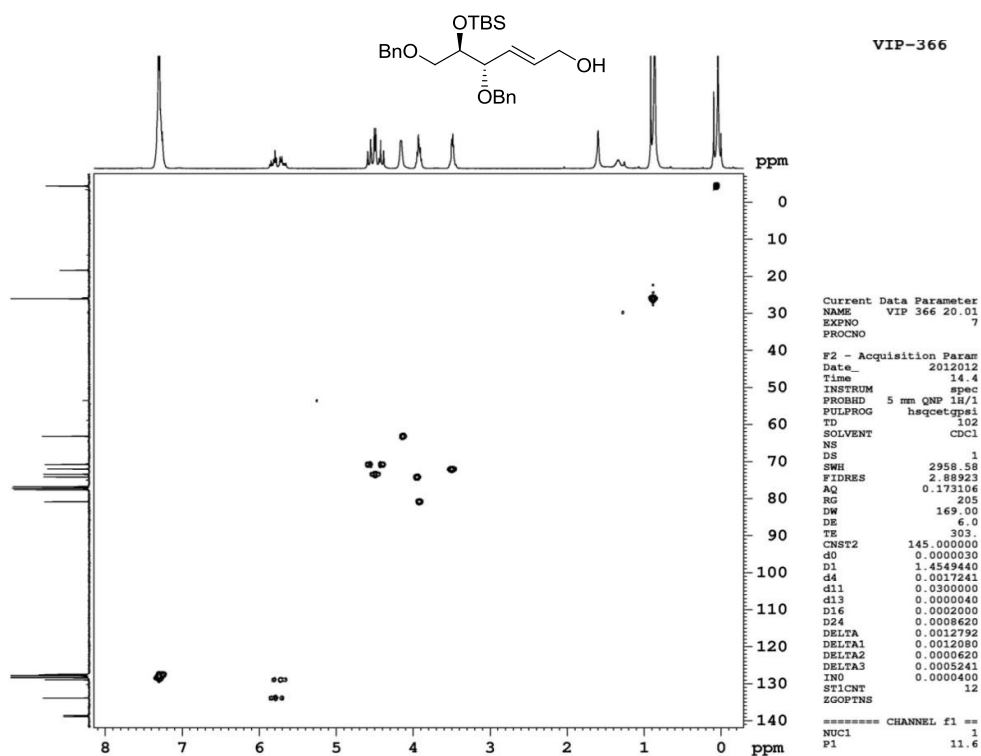
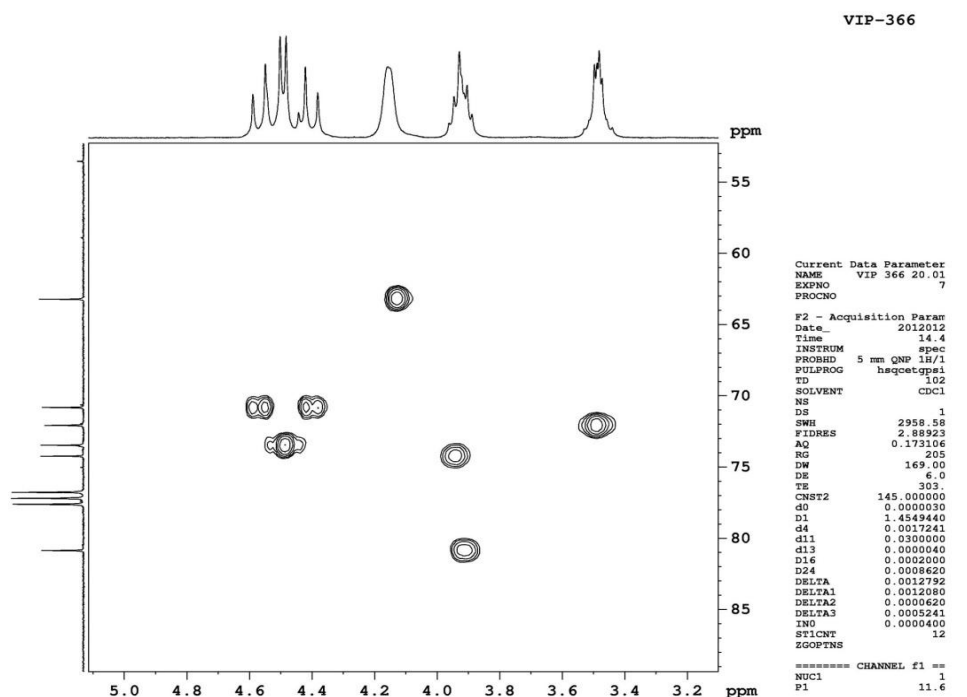
F2 - Acquisition Parameters
Date_ 20111231
Time 3.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 32768
DW 27.733 use
DE 6.00 use
TE 303.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.70 use
PL1 -3.00 dB
SFO1 75.4752953 MHz

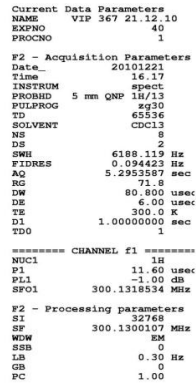
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768

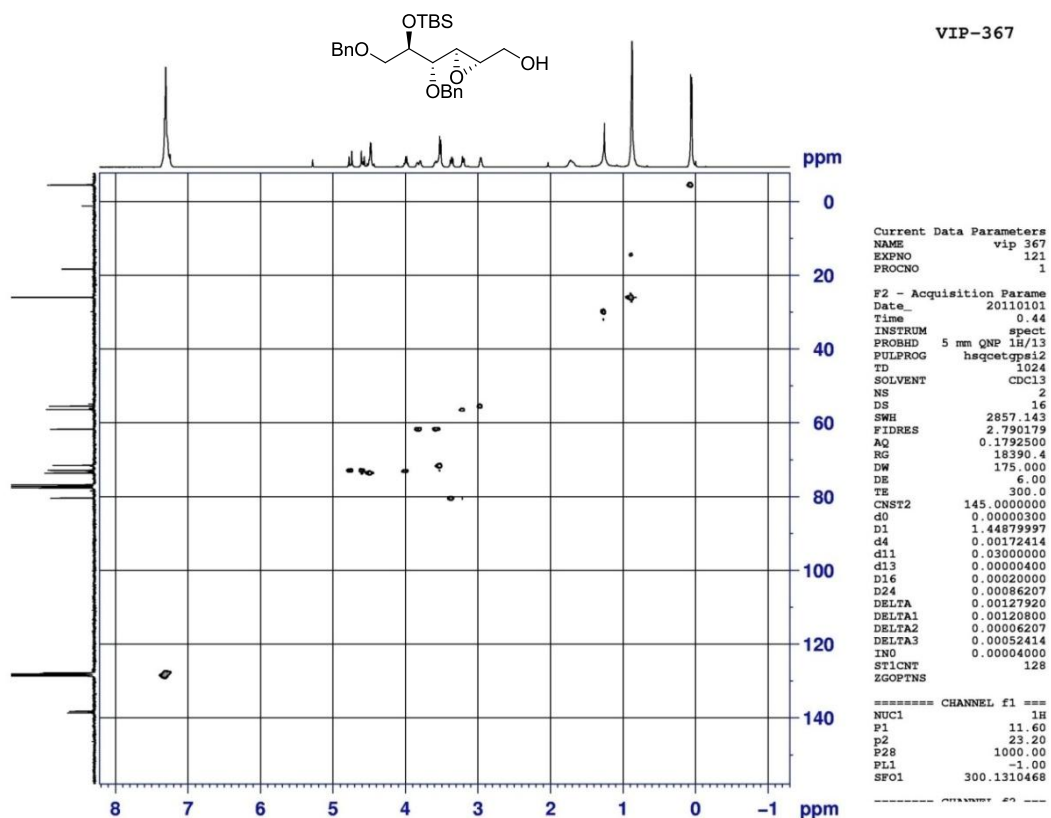
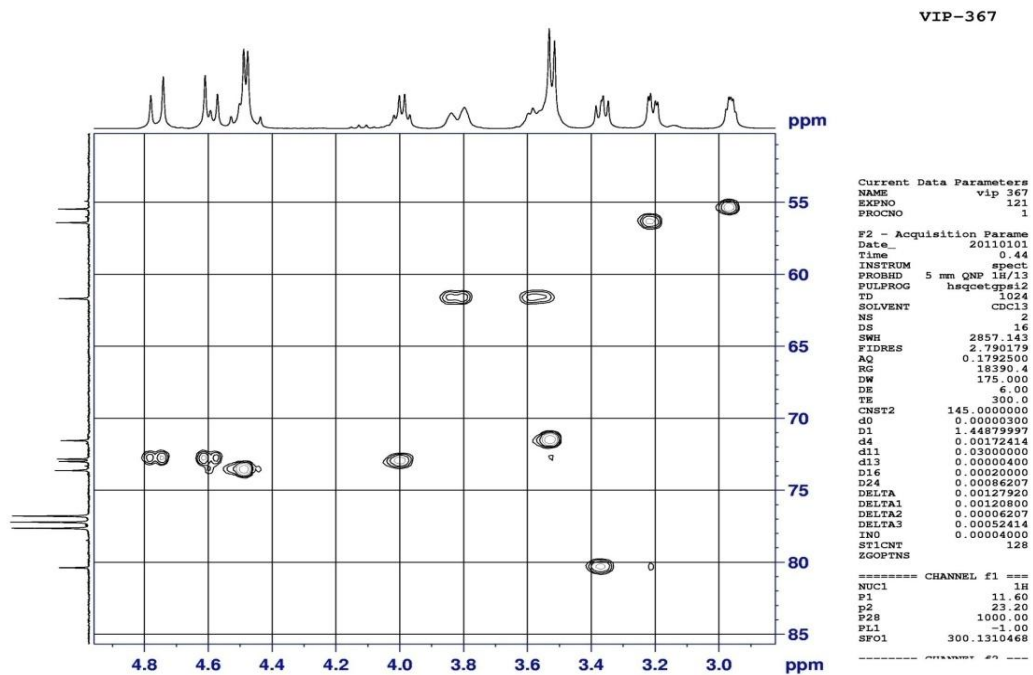
¹³C NMR Spectrum of 4b



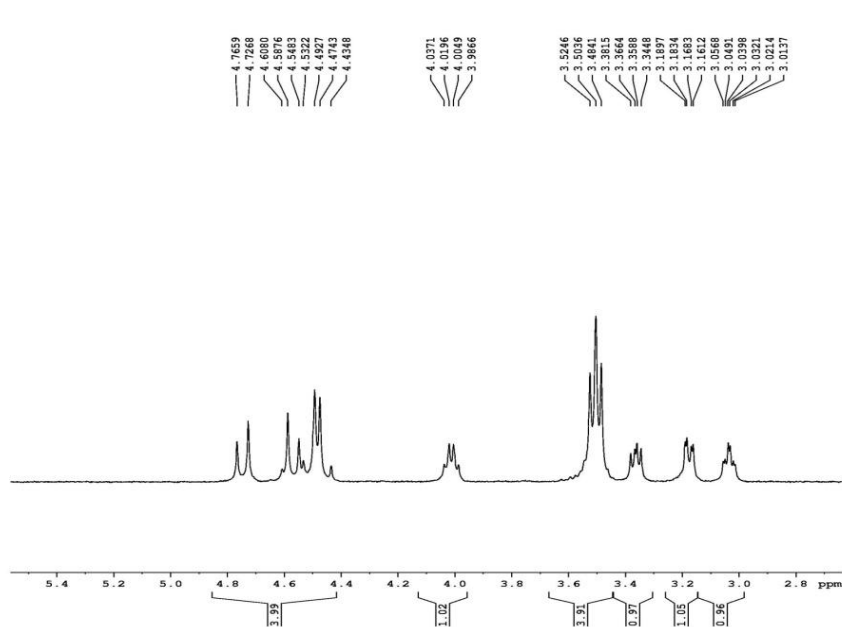
HSQC Spectra of 4b







HSQC Spectra of 5b

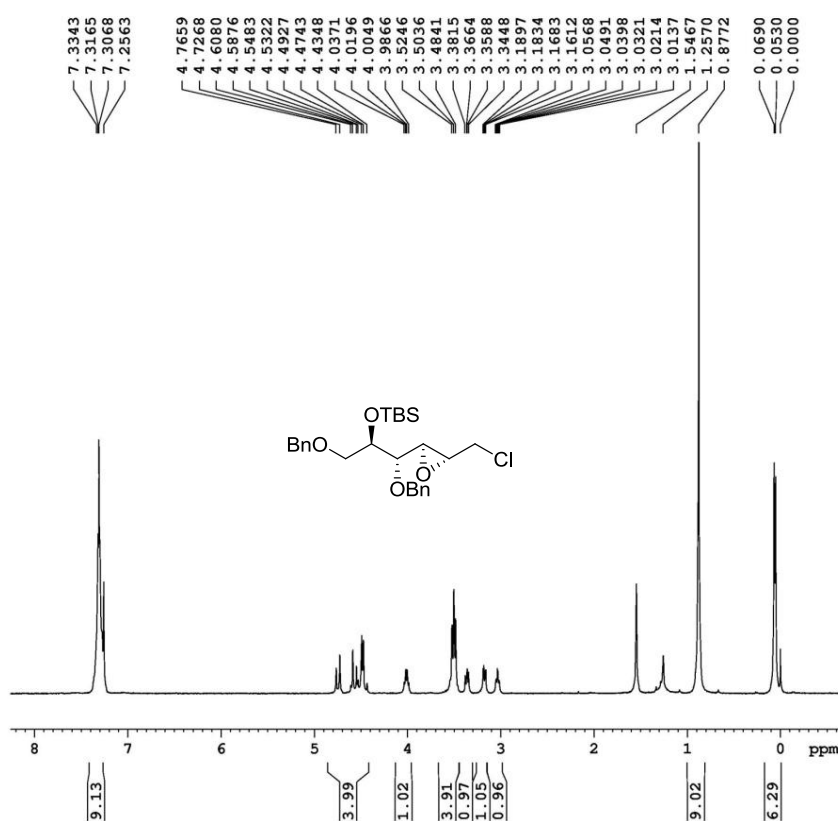


Current Data Parameters
NAME VIP-368 28.12.10
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101228
Time 23.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 4
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



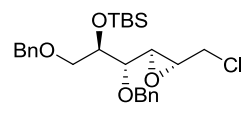
Current Data Parameters
NAME VIP-368 28.12.10
EXPNO 70
PROCNO 1

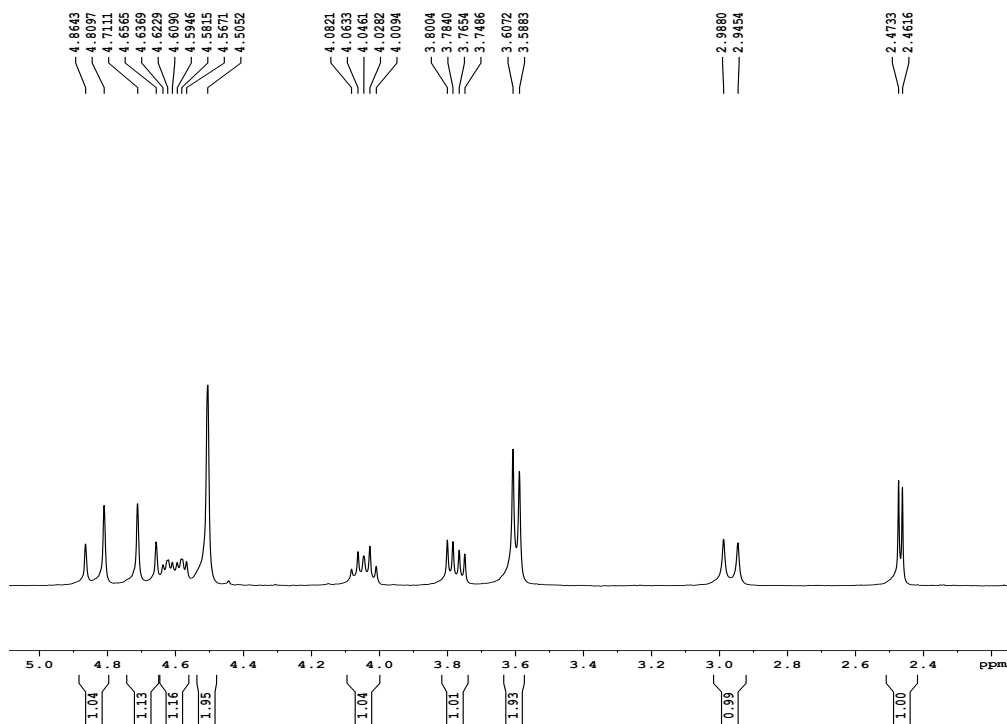
F2 - Acquisition Parameters
Date_ 20101228
Time 23.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 4
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H NMR Spectrum of 6b





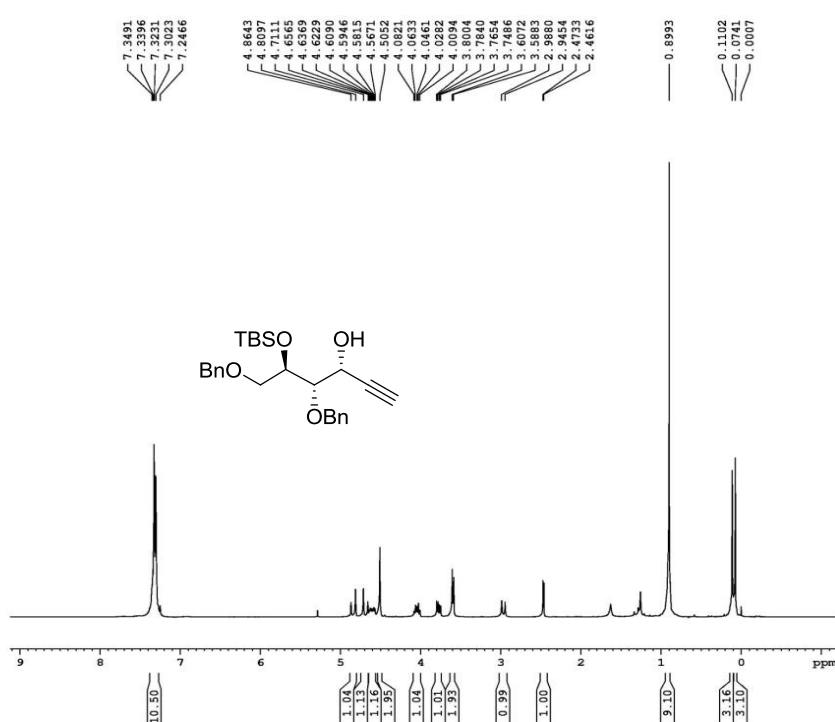
VIP-394-H

Current Data Parameters
NAME VIP 394 H
EXPNO 1060
PROCNO 1

F2 - Acquisition Parameter
Date_ 20110729
Time 16.04
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 4139.073 Hz
FIDRES 0.063157 Hz
AQ 7.9167986 se
RG 181
DW 120.800 us
DE 6.00 us
TE 0.0 K
D1 1.00000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se

===== CHANNEL f1 =====
NUC1 1H
P1 8.35 us
PL1 -4.00 dB
SFO1 200.1312359 MH

F2 - Processing parameters
SI 32768
SF 200.1300050 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



VIP-394-H

Current Data Parameters
NAME VIP 394 H
EXPNO 1060
PROCNO 1

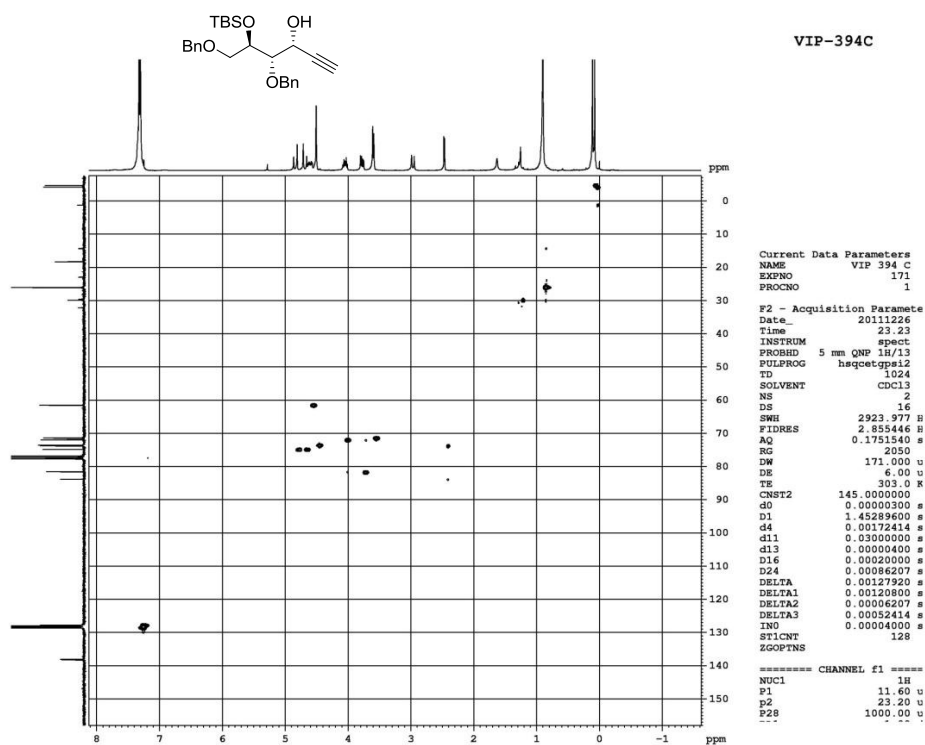
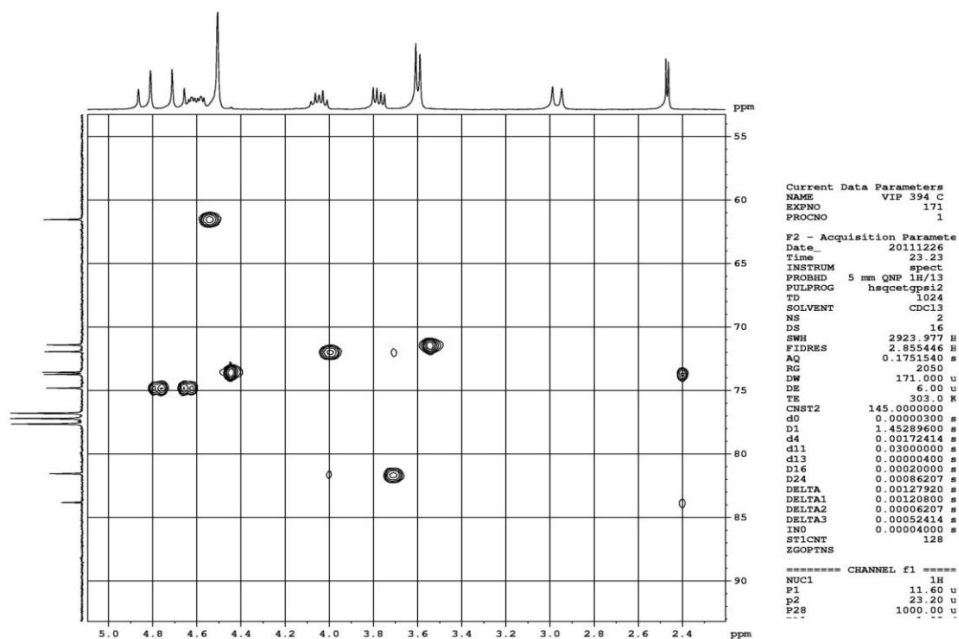
F2 - Acquisition Parameter
Date_ 20110729
Time 16.04
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 4139.073 Hz
FIDRES 0.063157 Hz
AQ 7.9167986 se
RG 181
DW 120.800 us
DE 6.00 us
TE 0.0 K
D1 1.00000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se

===== CHANNEL f1 =====
NUC1 1H
P1 8.35 us
PL1 -4.00 dB
SFO1 200.1312359 MH

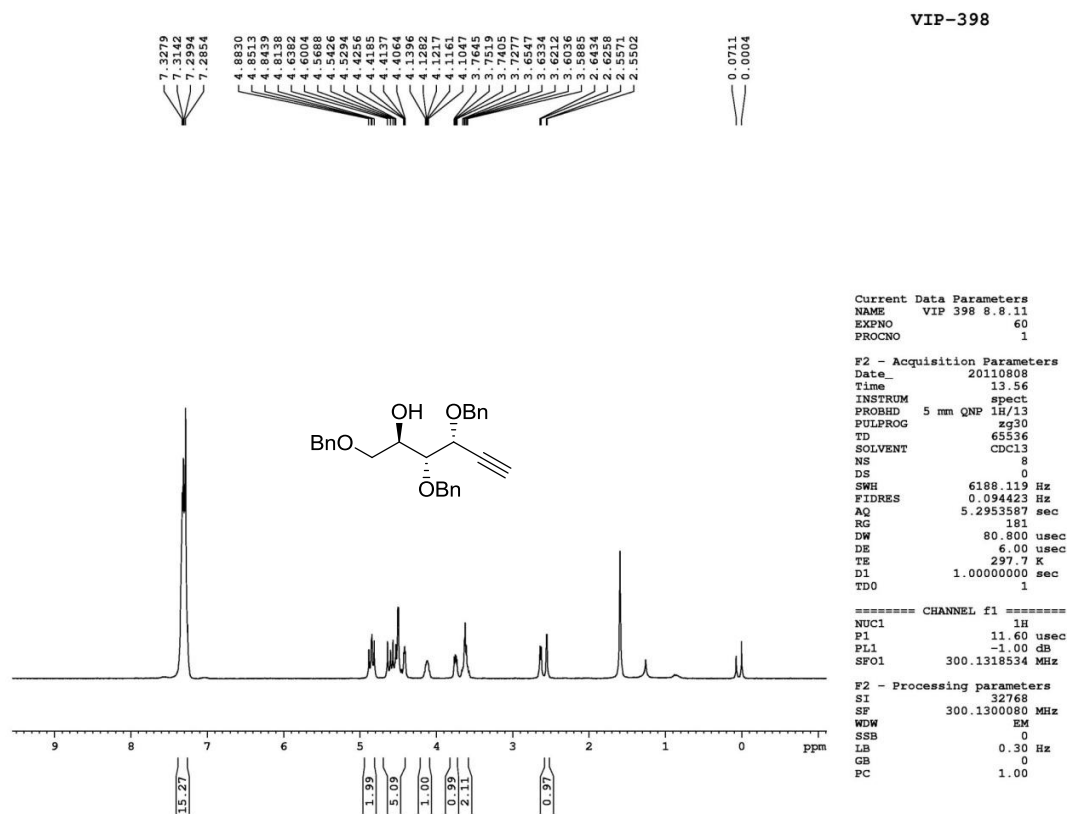
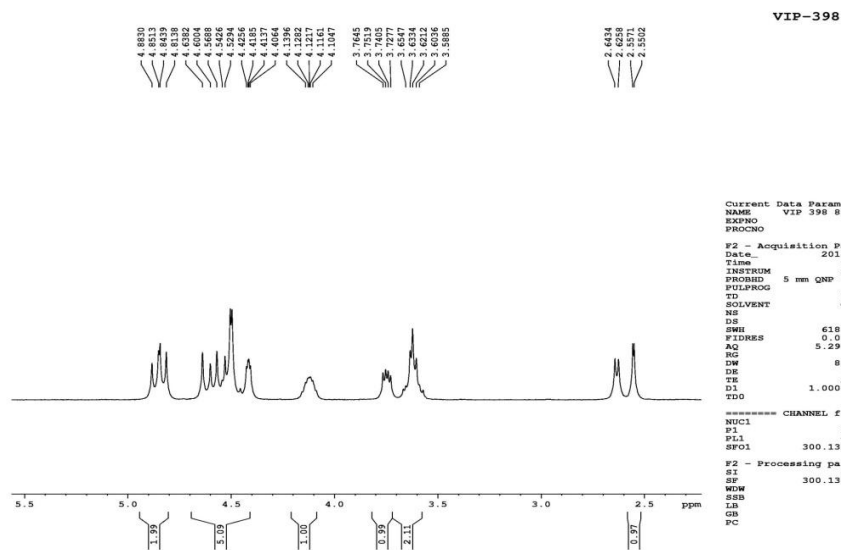
F2 - Processing parameters
SI 32768
SF 200.1300050 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H NMR Spectrum of 7b

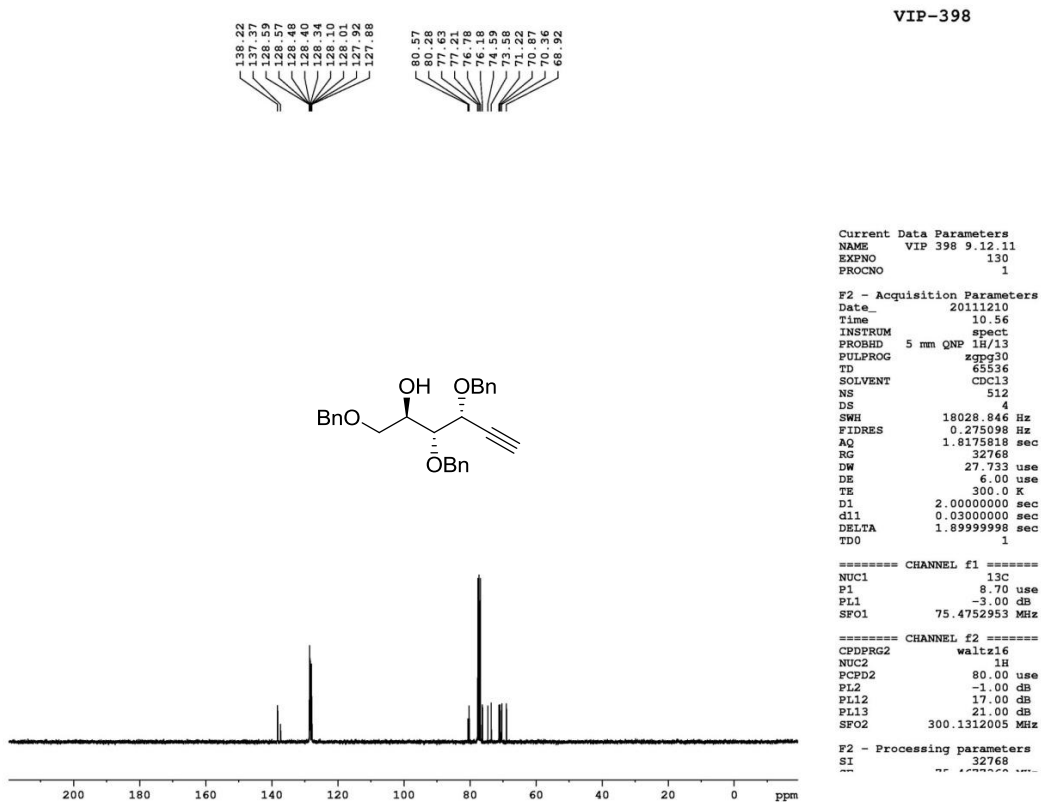
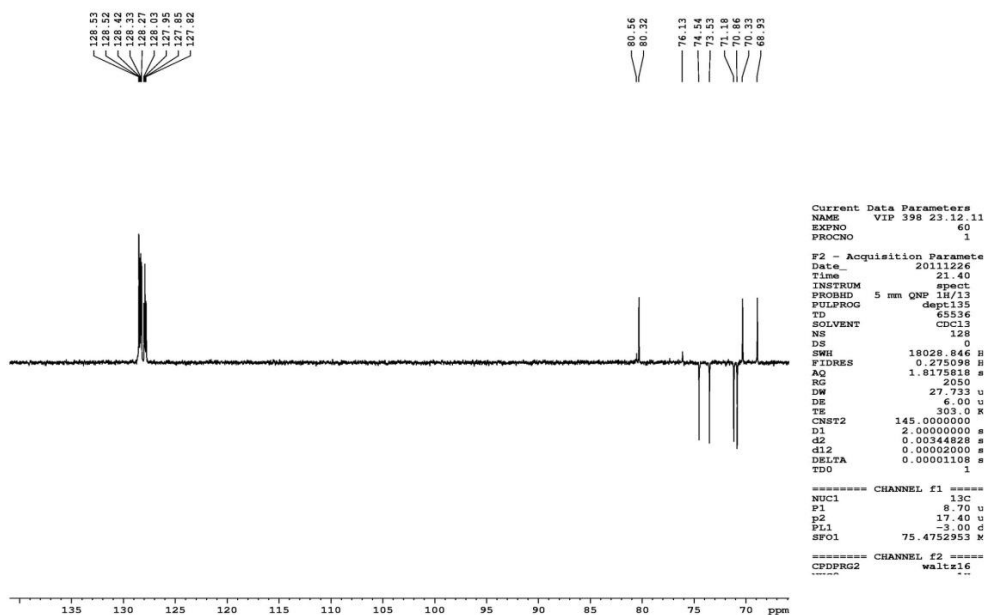
VIP-394C



HSQC Spectra of 7b

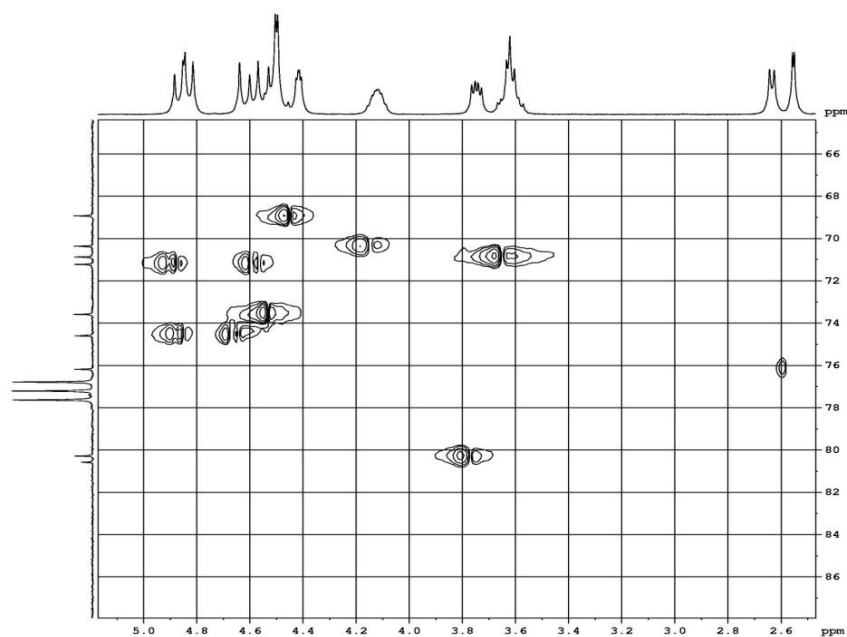


¹H NMR Spectrum of 8b



^{13}C NMR Spectrum of 8b

VIP-398

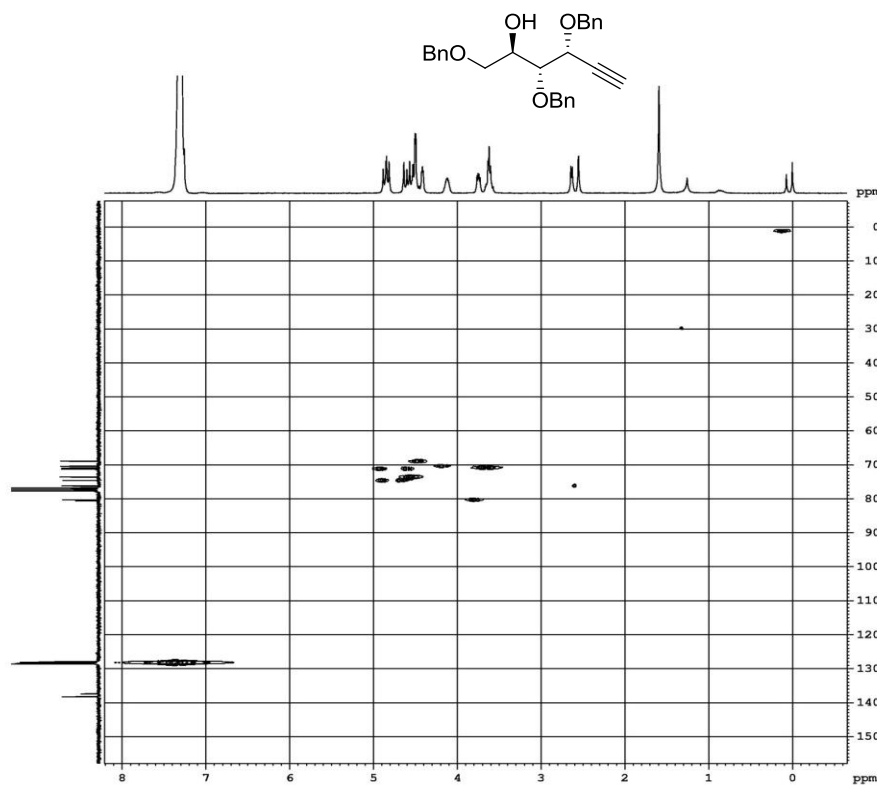


Current Data Parameters
 NAME VIP 398 30.12.11
 EXPNO 221
 PROCNO 1

F2 - Acquisition Parameters
 Date 20111231
 Time 9.00
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG hsqcetgps12
 TD 1024
 SOLVENT CDCl3
 NS 2
 DS 16
 SWH 2659.574 H
 FIDRES 2.597241 H
 AQ 0.1925620 s
 RG 2050
 DW 188.000 u
 DE 6.00 u
 TE 303.0 K
 CNST2 145.0000000
 d0 0.00000300 s
 D1 1.43548799 s
 d4 0.00172414 s
 d11 0.03000000 s
 d13 0.00000400 s
 D16 0.00020000 s
 D24 0.00086207 s
 DELTA 0.00127920 s
 DELTA1 0.00120800 s
 DELTA2 0.00086207 s
 DELTA3 0.00052414 s
 INO 0.00004000 s
 STCNT 128
 ZGPTNS

===== CHANNEL f1 =====
 NUC1 1H
 P1 1.00

VIP-398

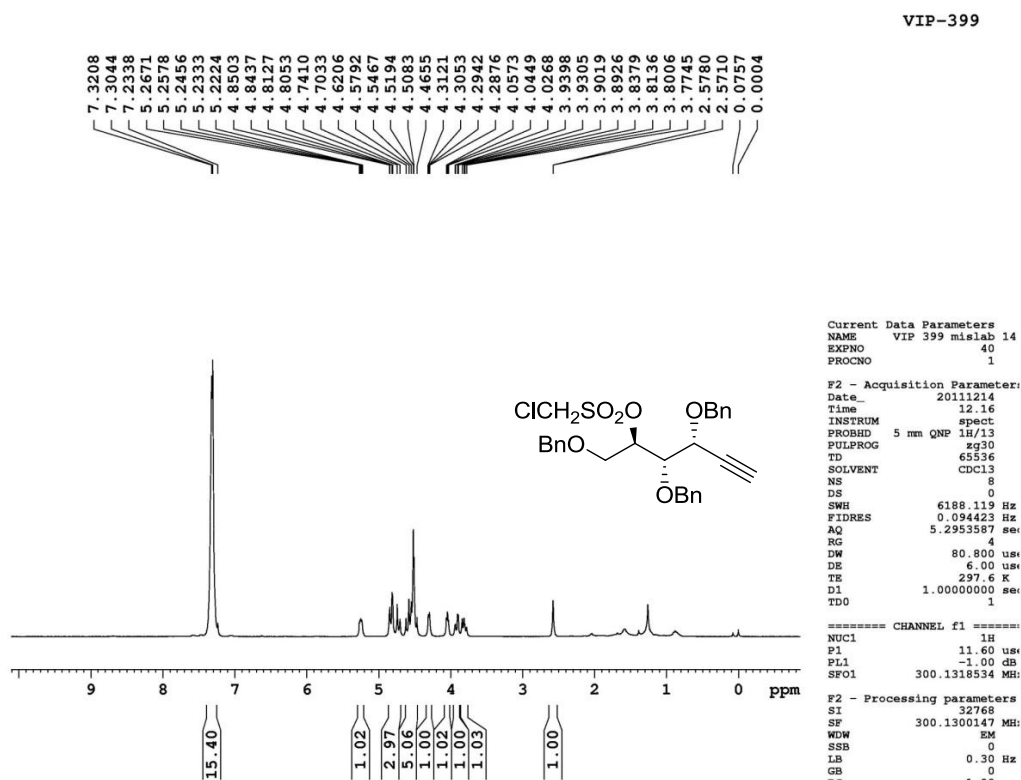
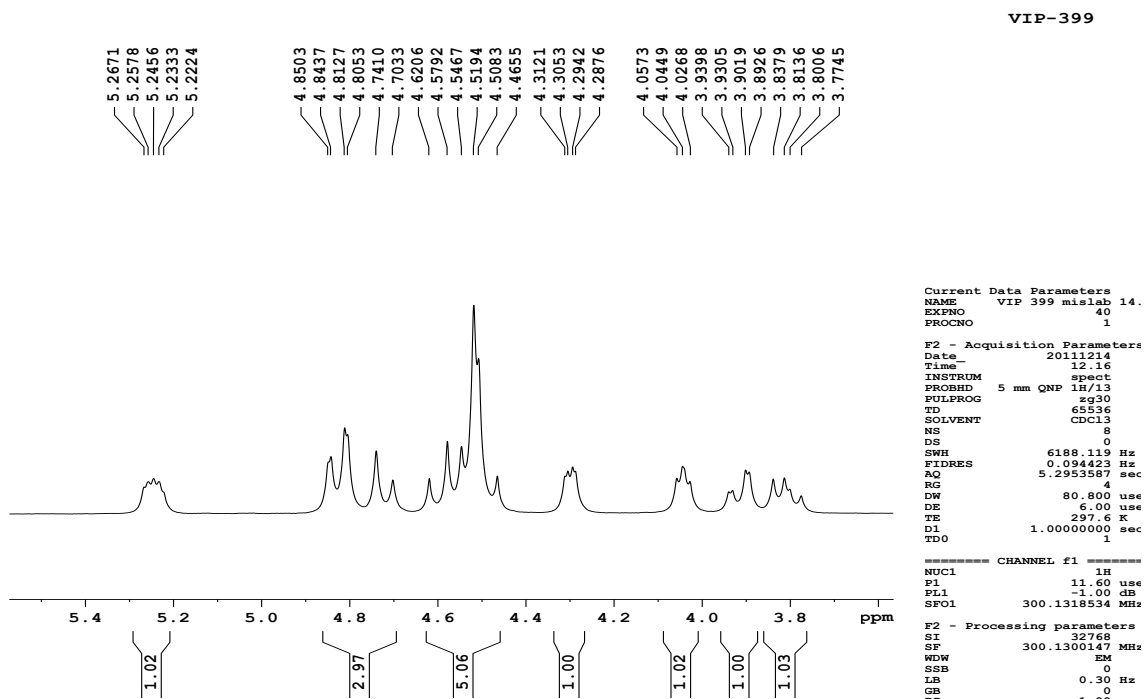


Current Data Parameters
 NAME VIP 398 30.12.11
 EXPNO 221
 PROCNO 1

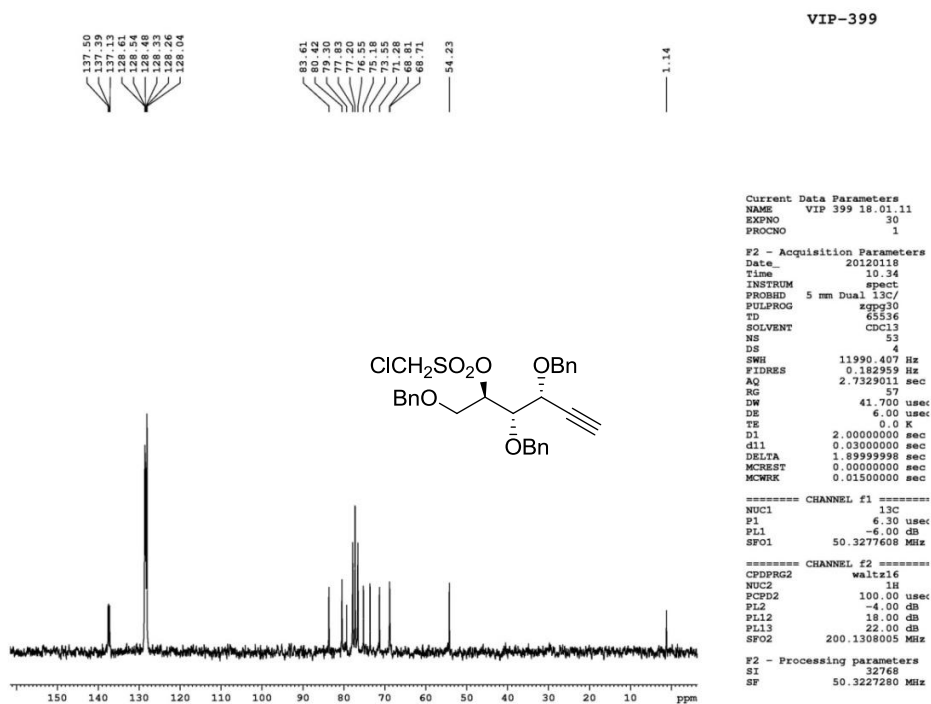
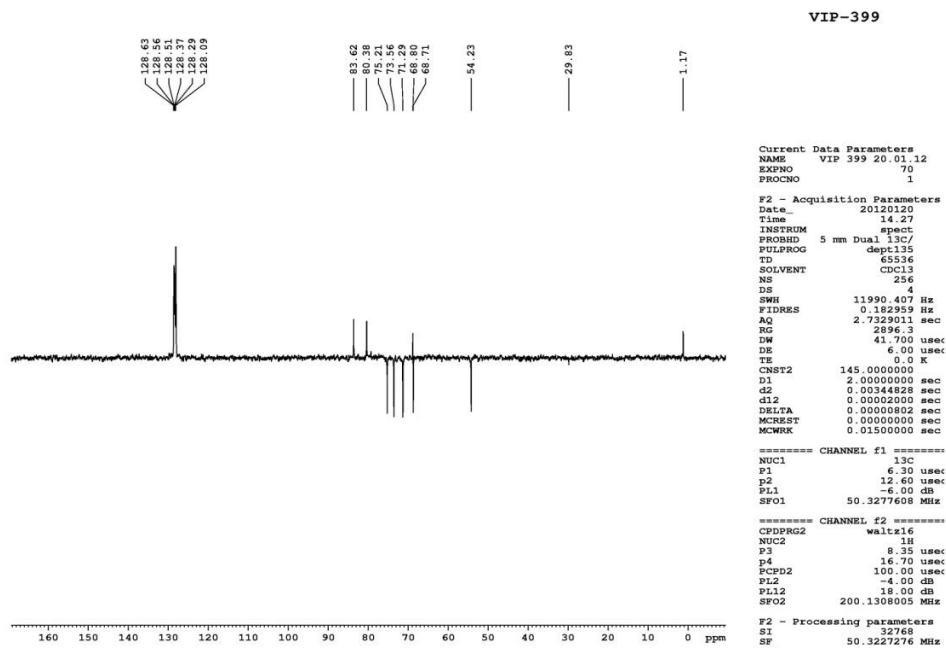
F2 - Acquisition Parameters
 Date 20111231
 Time 9.00
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG hsqcetgps12
 TD 1024
 SOLVENT CDCl3
 NS 2
 DS 16
 SWH 2659.574 H
 FIDRES 2.597241 H
 AQ 0.1925620 s
 RG 2050
 DW 188.000 u
 DE 6.00 u
 TE 303.0 K
 CNST2 145.0000000
 d0 0.00000300 s
 D1 1.43548799 s
 d4 0.00172414 s
 d11 0.03000000 s
 d13 0.00000400 s
 D16 0.00020000 s
 D24 0.00086207 s
 DELTA 0.00127920 s
 DELTA1 0.00120800 s
 DELTA2 0.00086207 s
 DELTA3 0.00052414 s
 INO 0.00004000 s
 STCNT 128
 ZGPTNS

===== CHANNEL f1 =====
 NUC1 1H
 P1 1.00

HSQC Spectra of 8b

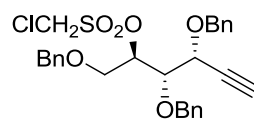
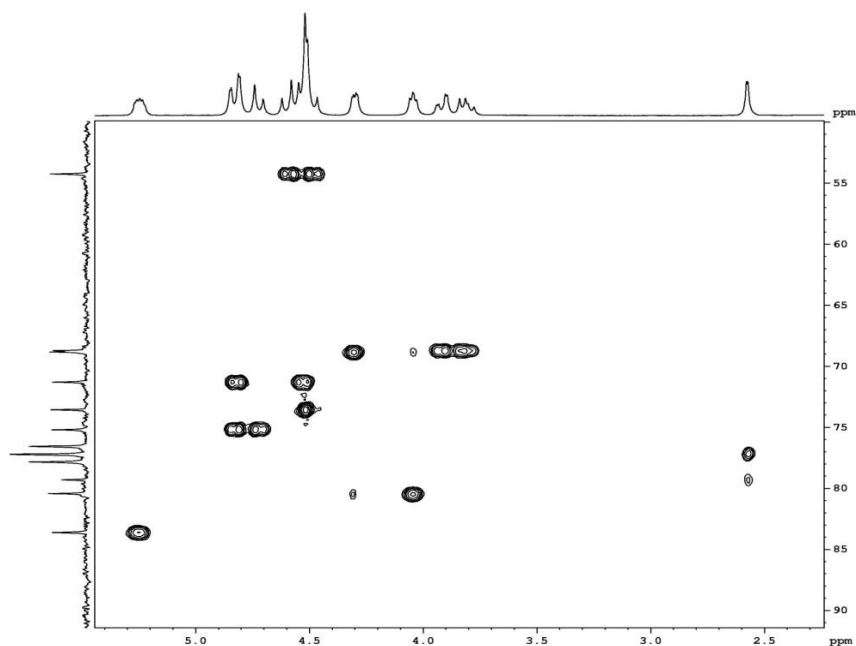


¹H NMR Spectrum of 9b

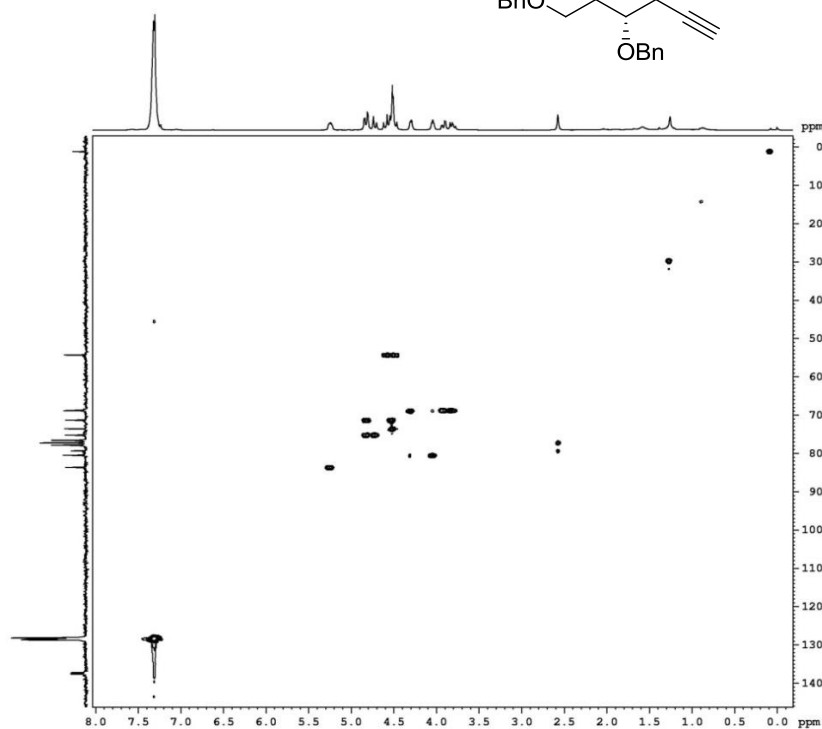


¹³C NMR Spectrum of 9b

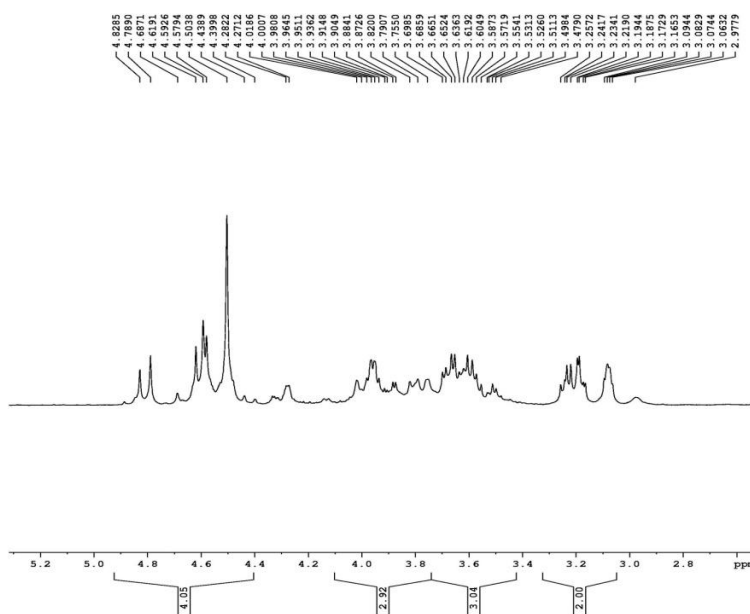
VIP-399



VIP-399



HSQC Spectra of 9b



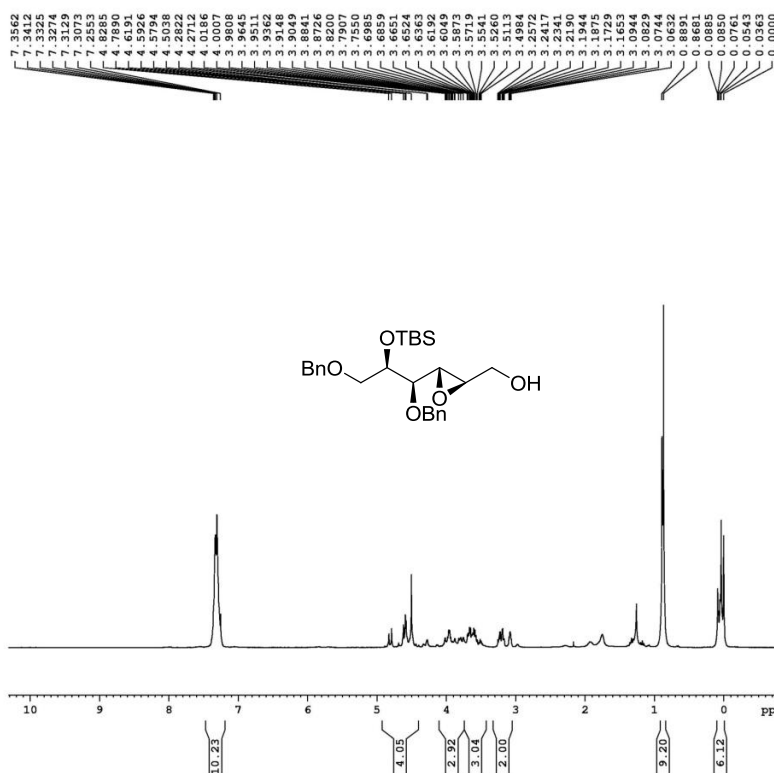
VIP-389

Current Data Parameters
NAME VIP 389 17.06.11
EXPNO 150
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110618
Time 2.42
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 8
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 90.5
DW 80.800 use
DE 6.00 use
TE 297.3 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.60 use
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



VIP-389

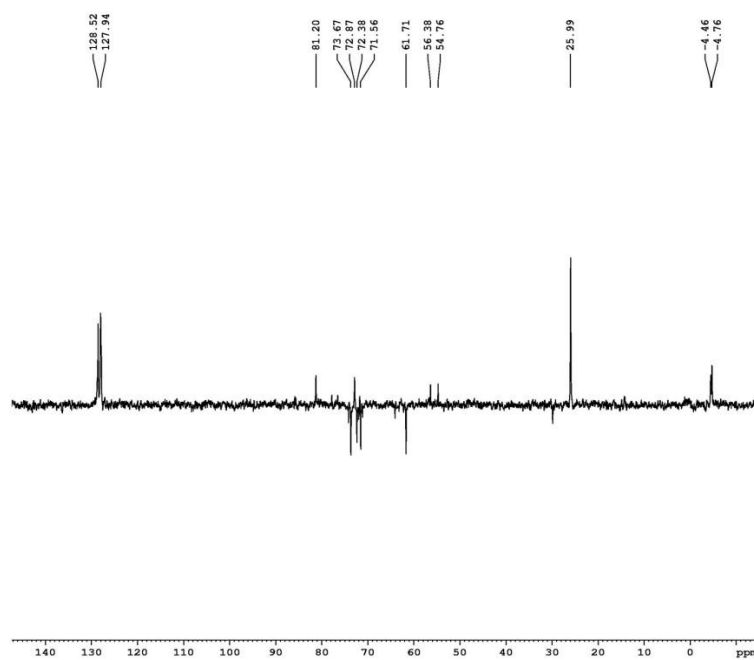
Current Data Parameters
NAME VIP 389 17.06.11
EXPNO 150
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110618
Time 2.42
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 8
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 90.5
DW 80.800 use
DE 6.00 use
TE 297.3 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.60 use
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H NMR Spectrum of 10a



vip-389

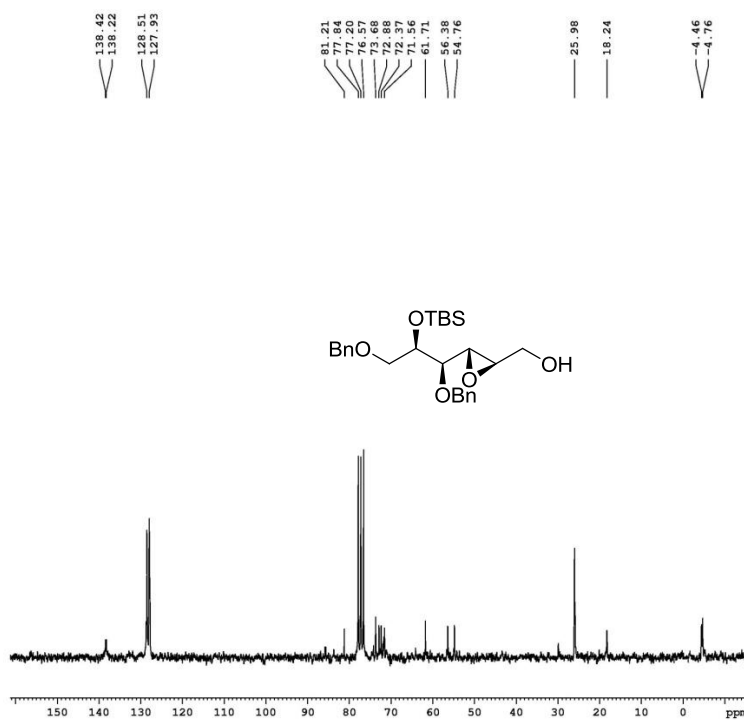
Current Data Parameters
NAME VIP 389 06.07.11
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters:
Date_ 20110706
Time 13.09
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1625.5
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
CNST2 145.000000 sec
D1 2.00000000 sec
d12 0.00344828 sec
d12 0.00020000 sec
DELTA 0.00000802 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.30 usec
P2 12.60 usec
PL1 -6.00 dB
SFO1 50.3277608 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 8.35 usec
P4 16.70 usec
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.00 dB
SFO2 200.1308005 MHz

F2 - Processing parameters



vip-389

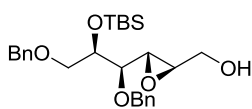
Current Data Parameters
NAME VIP 389 08.07.11
EXPNO 130
PROCNO 1

F2 - Acquisition Parameters:
Date_ 20110708
Time 11.28
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 64
DW 41.700 usec
DE 6.00 usec
TE 0.0 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 6.30 usec
PL1 -6.00 dB
SFO1 50.3277608 MHz

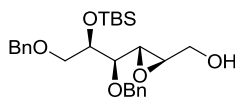
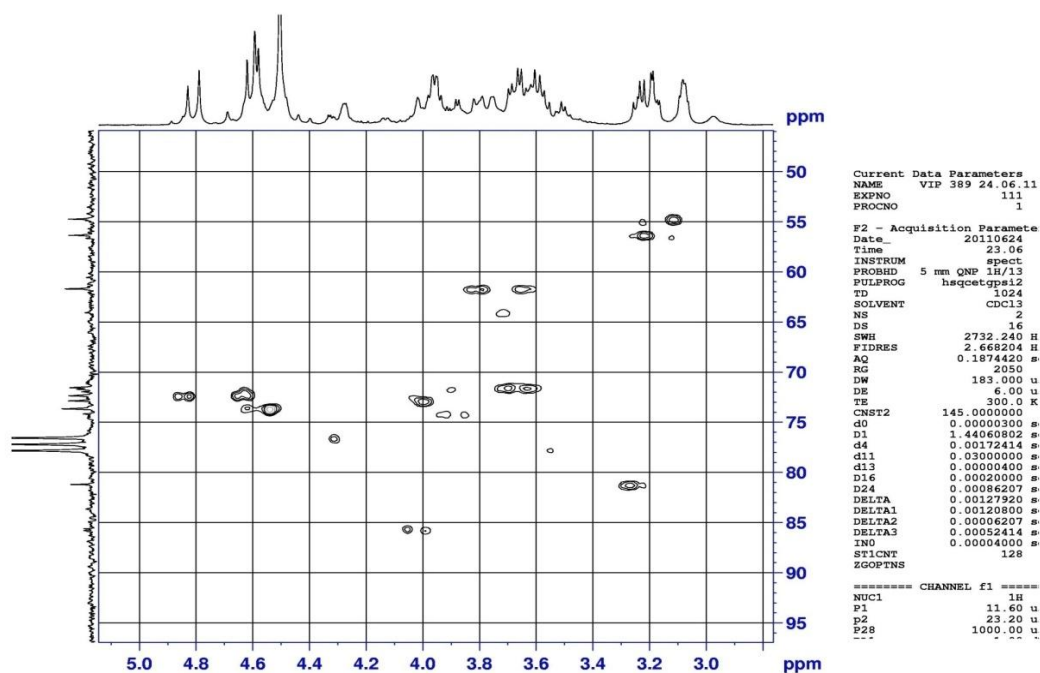
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.1308005 MHz

F2 - Processing parameters
SI 32768
SF 50.3227204 MHz

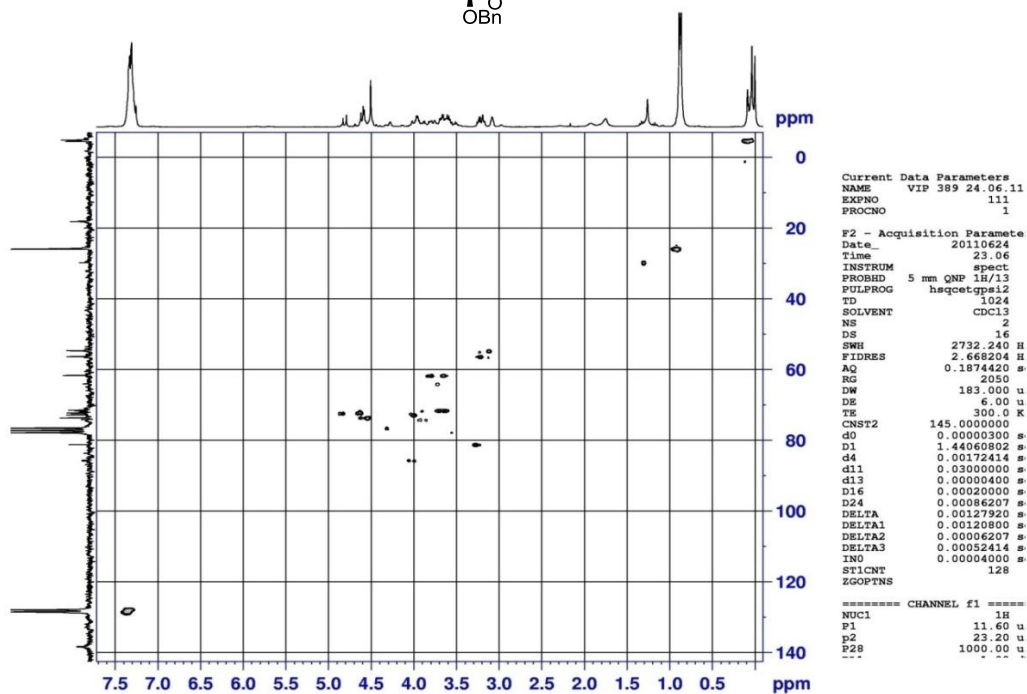


¹³C NMR Spectrum of 10a

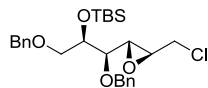
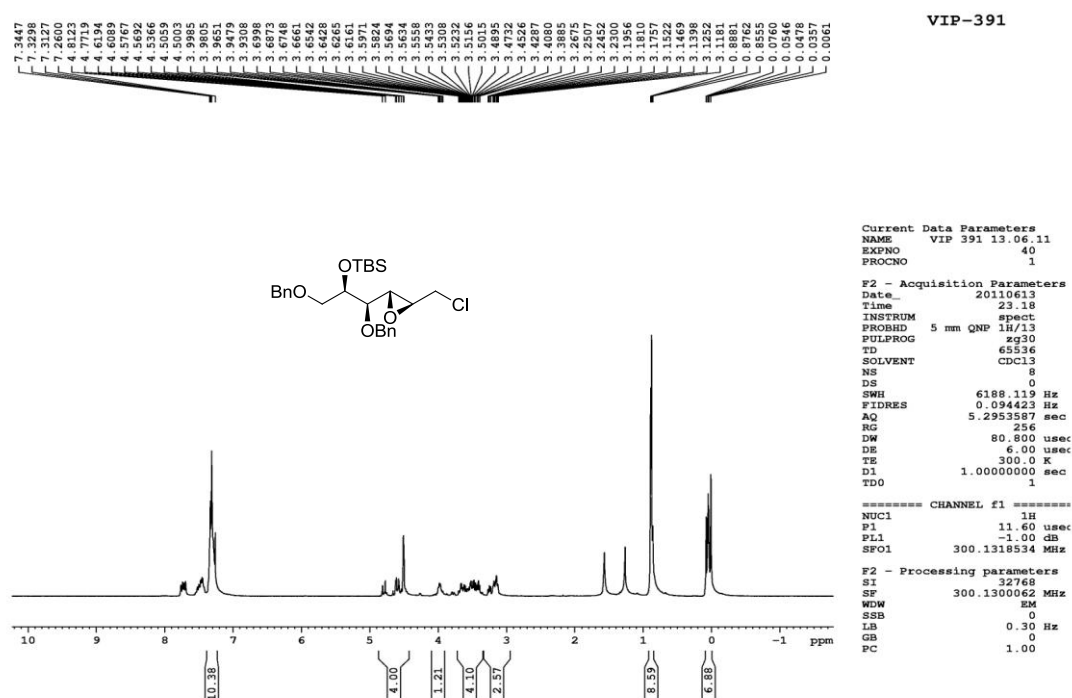
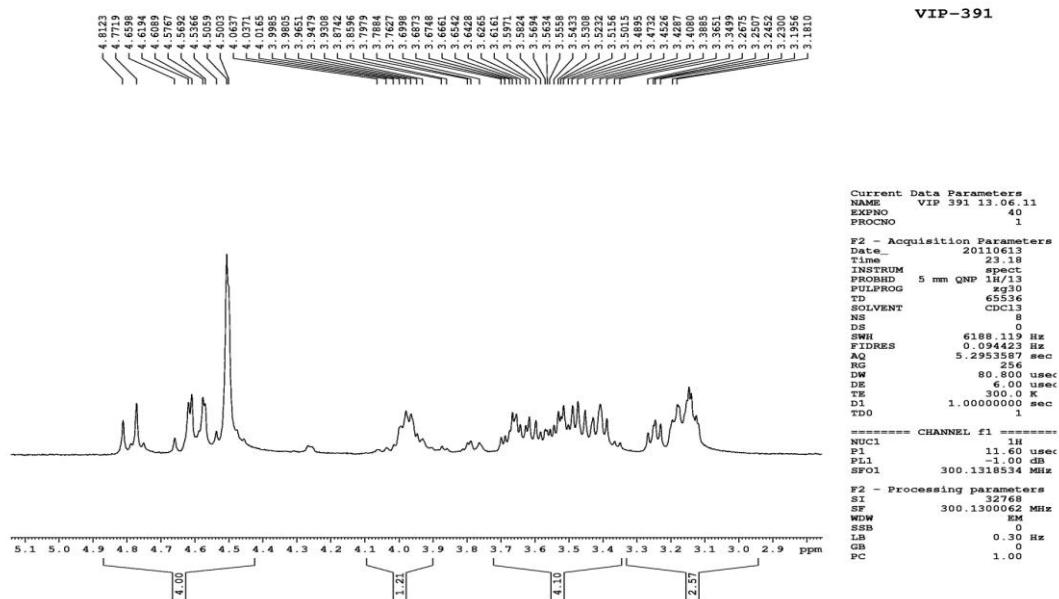
VIP-389



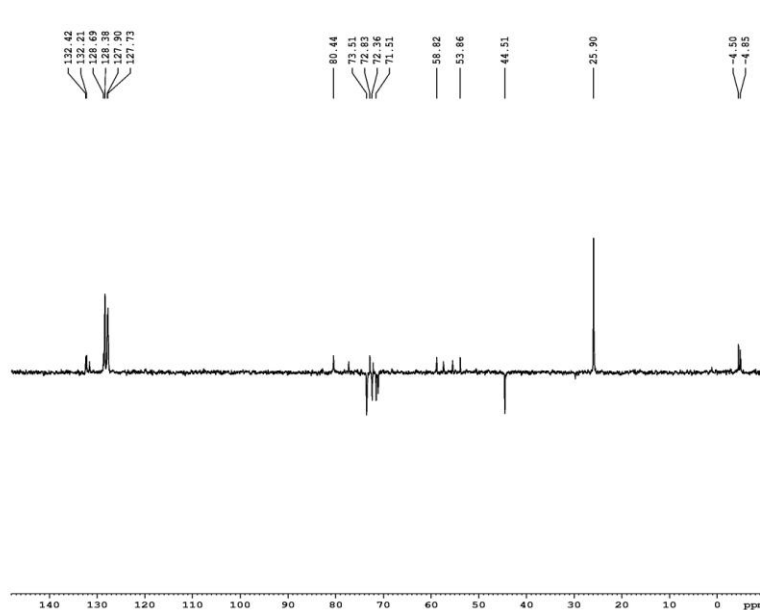
VIP-389



HSQC Spectra of 10a



¹H NMR Spectrum of 11a



VIP-391

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Current Data Parameters
NAME      VIP 391 17.04.12
EXPNO     30
PROCNO    1

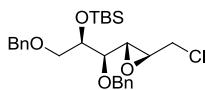
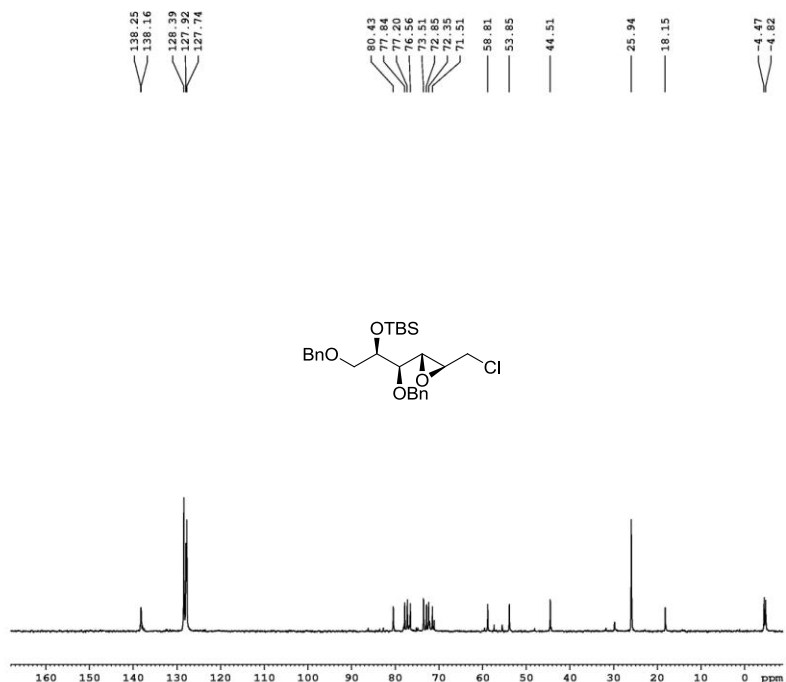
F2 - Acquisition Parameters
Date_     20121016
Time      12.25
INSTRUM   spect
PROBHD    5 mm Dual 13C/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         174
DS         4
SWH        11990.407 Hz
FIDRES     0.182959 Hz
AQ         2.7329011 sec
RG         1290.2
DW         41.700 usec
DE         6.00 usec
TE         0.0 K
CNST2     145.0000000 sec
D1         2.00000000 sec
d2         0.00344828 sec
d12        0.00002000 sec
DELTA      0.00000802 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         6.30 usec
p2         12.60 usec
PL1        -6.00 dB
SFO1       50.3277608 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P3         8.35 usec
p4         16.70 usec
PCPD2      100.00 usec
PL2        -4.00 dB
PL12       18.00 dB
SFO2       200.1308005 MHz

F2 - Processing parameters
SI         32768
SF         50.3277273 MHz
WDW        EM
SSB         0
LB         3.00 Hz
GB         0
PC         1.40

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VIP-391

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Current Data Parameters
NAME      VIP 391 27.03.12
EXPNO     30
PROCNO    1

F2 - Acquisition Parameters
Date_     20120925
Time      10.27
INSTRUM   spect
PROBHD    5 mm Dual 13C/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         4
SWH        11990.407 Hz
FIDRES     0.182959 Hz
AQ         2.7329011 sec
RG         57
DW         41.700 usec
DE         6.00 usec
TE         0.0 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

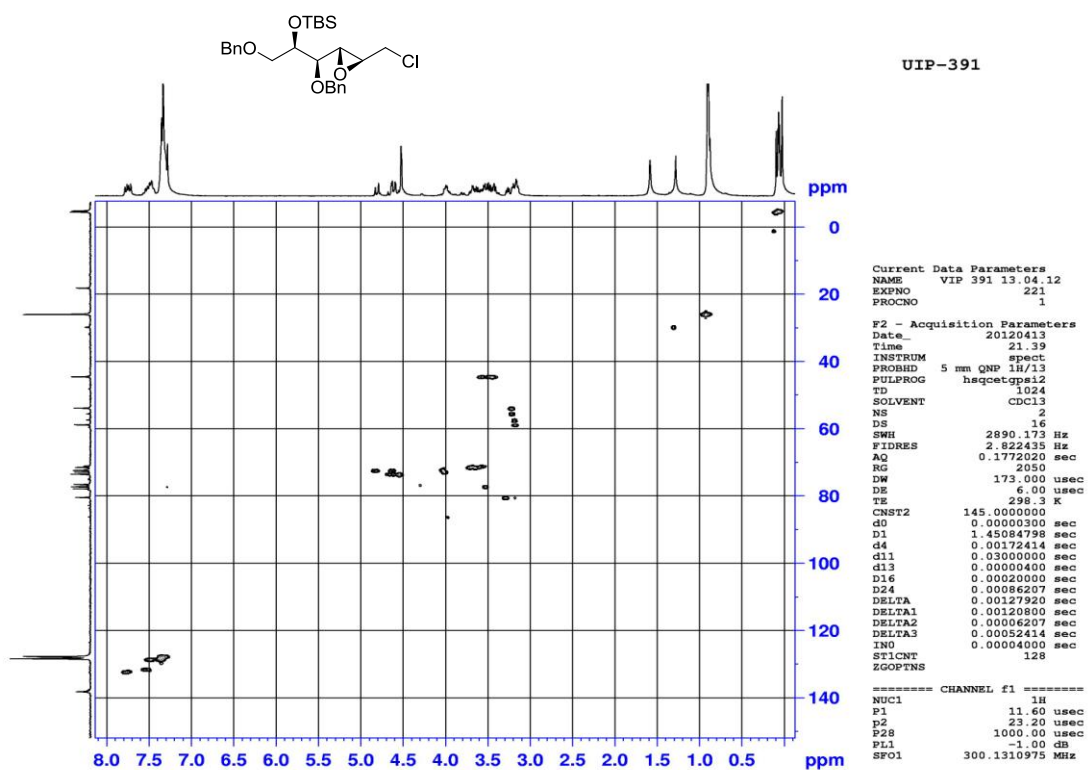
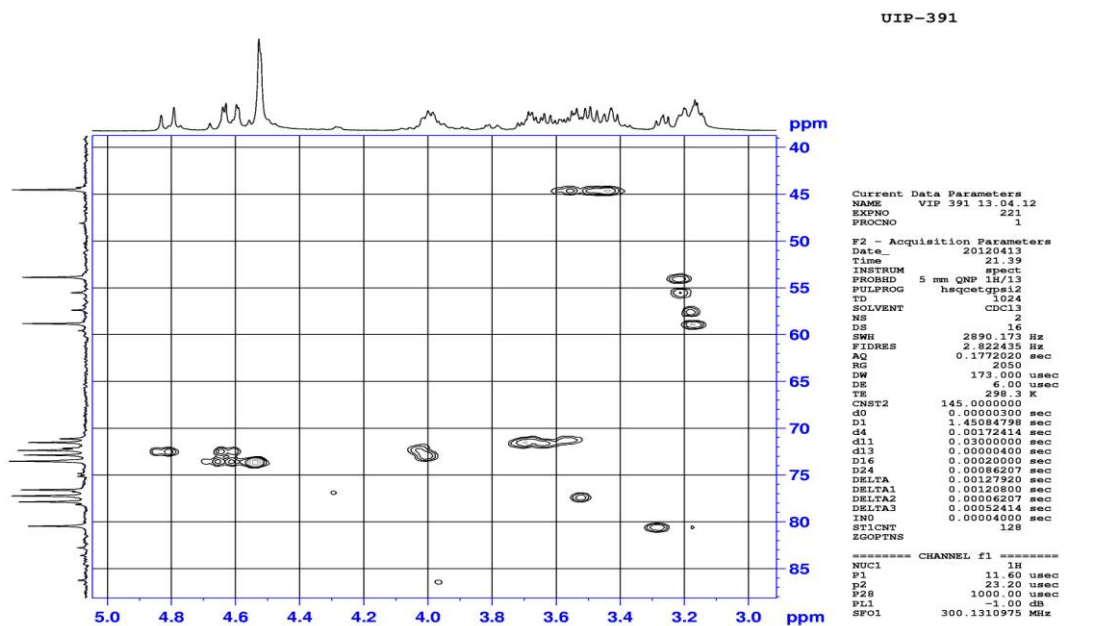
===== CHANNEL f1 =====
NUC1       13C
P1         6.30 usec
PL1        -6.00 dB
SFO1       50.3277608 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      100.00 usec
PL2        -4.00 dB
PL12       18.00 dB
PL13       22.00 dB
SFO2       200.1308005 MHz

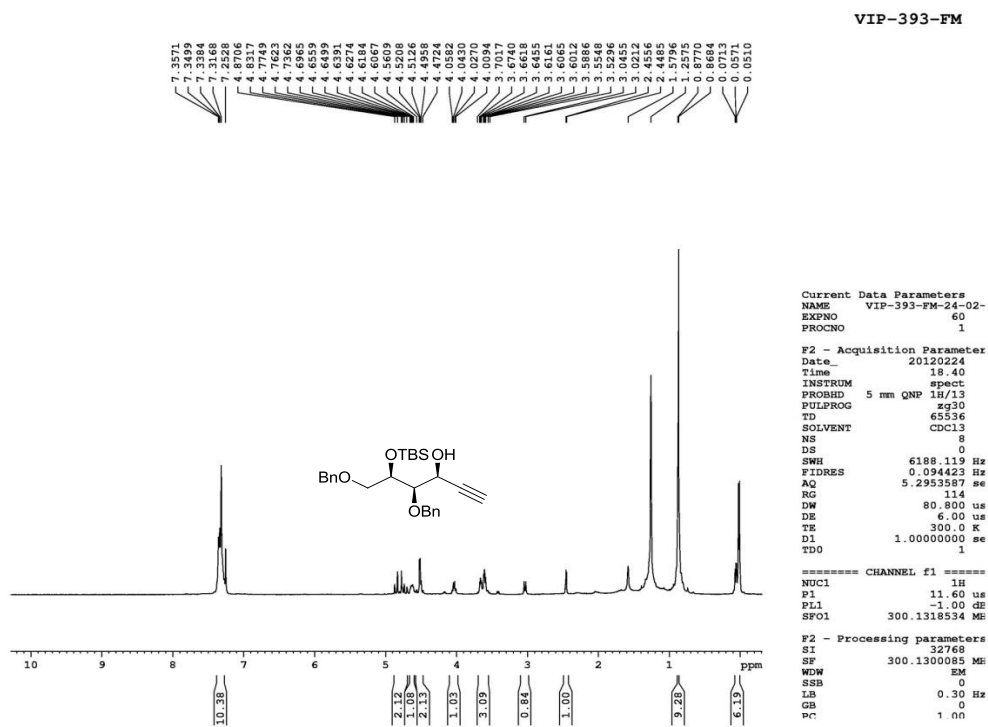
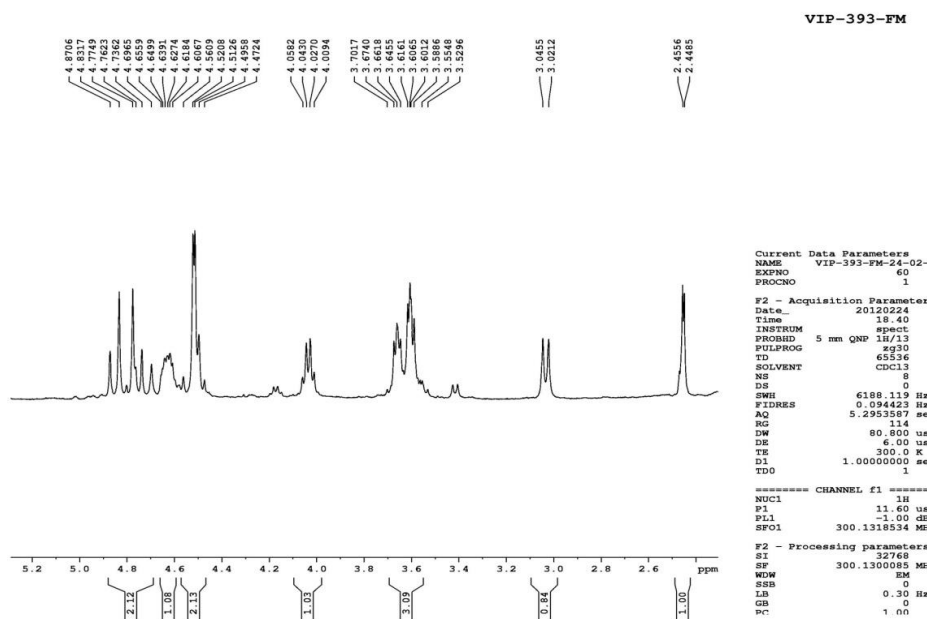
F2 - Processing parameters
SI         32768
SF         50.3277299 MHz
WDW        EM
SSB         0
LB         3.00 Hz
GB         0
PC         1.40

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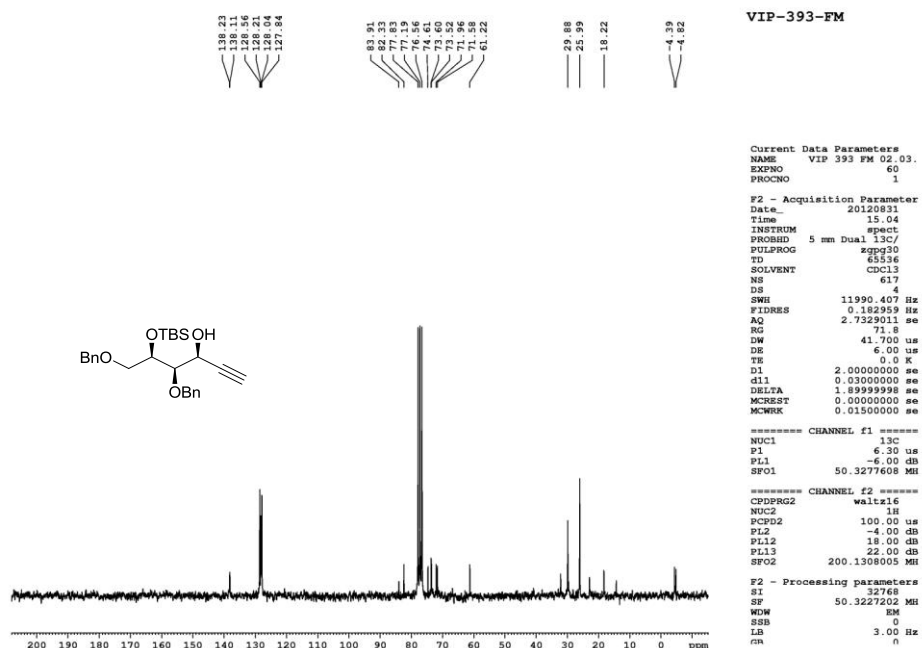
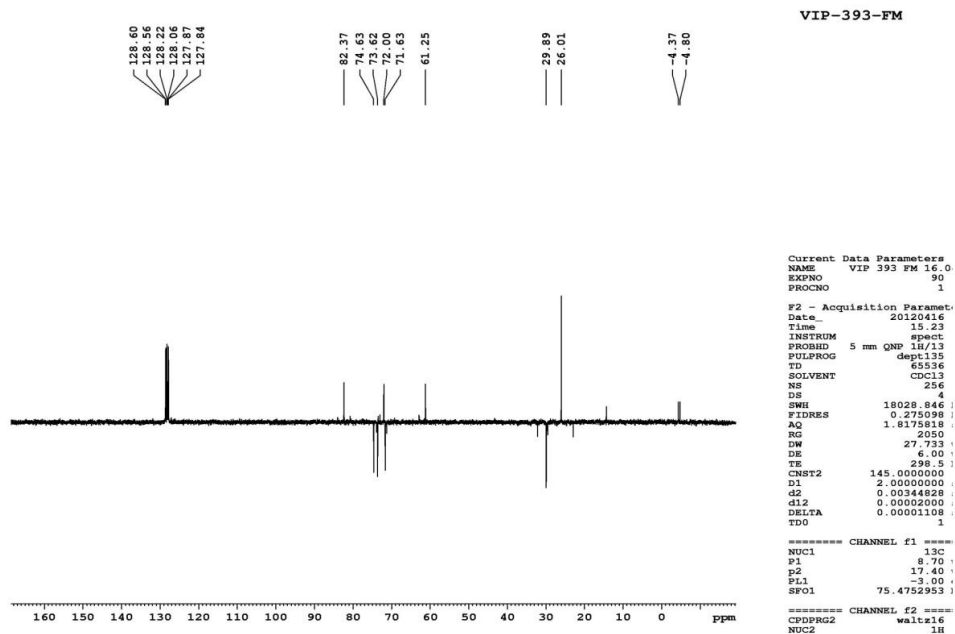
¹³C NMR Spectrum of 11a



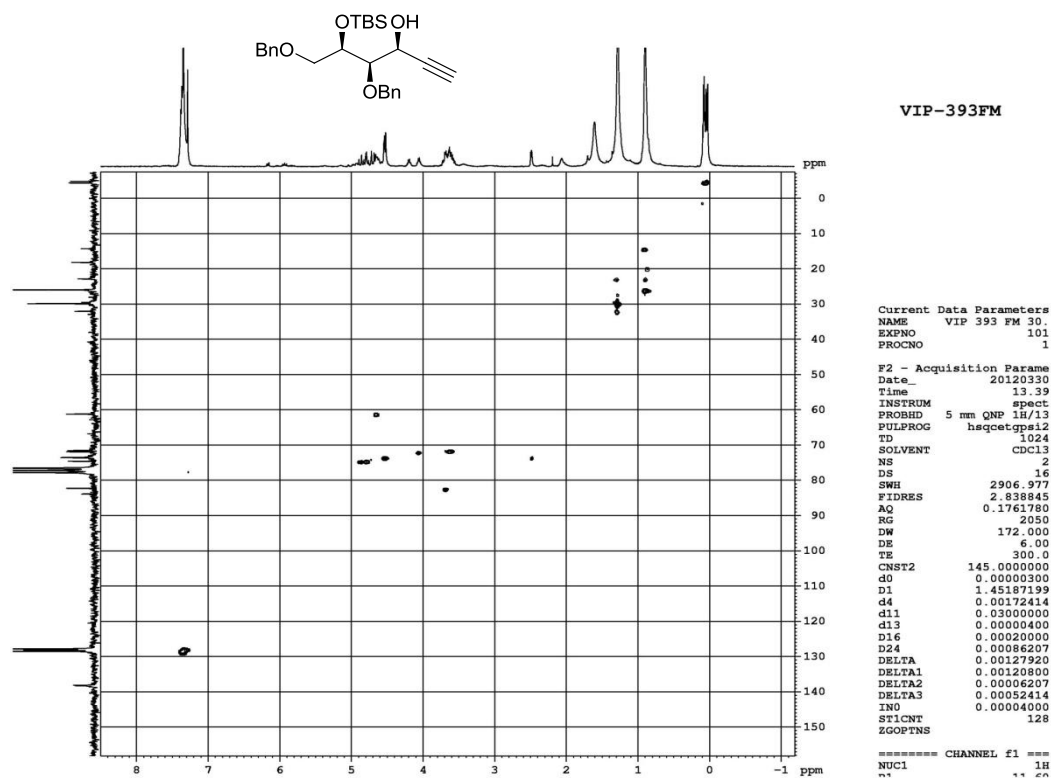
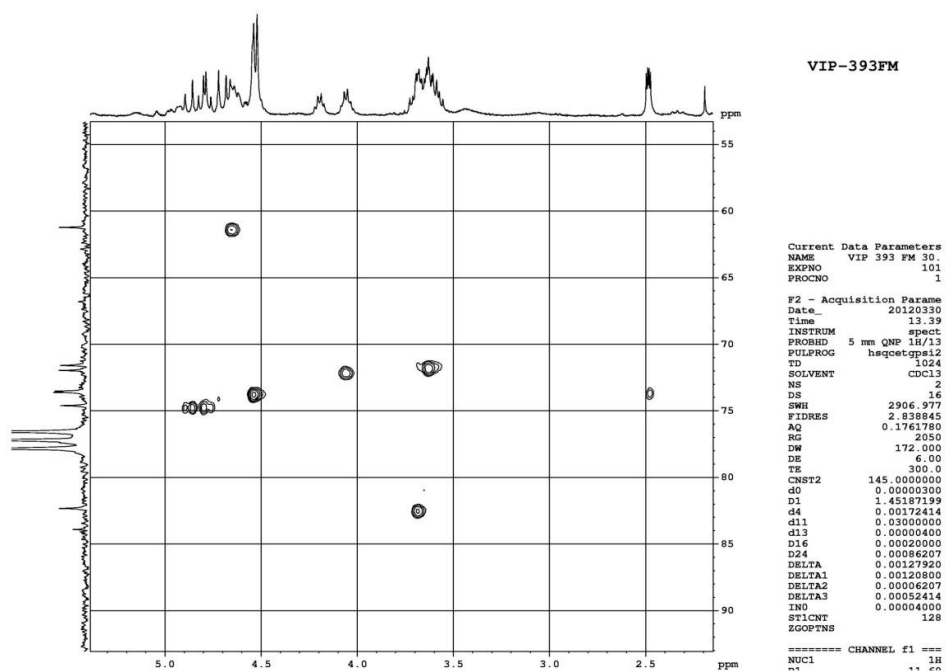
HSQC Spectra of 11a



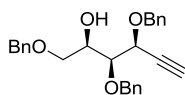
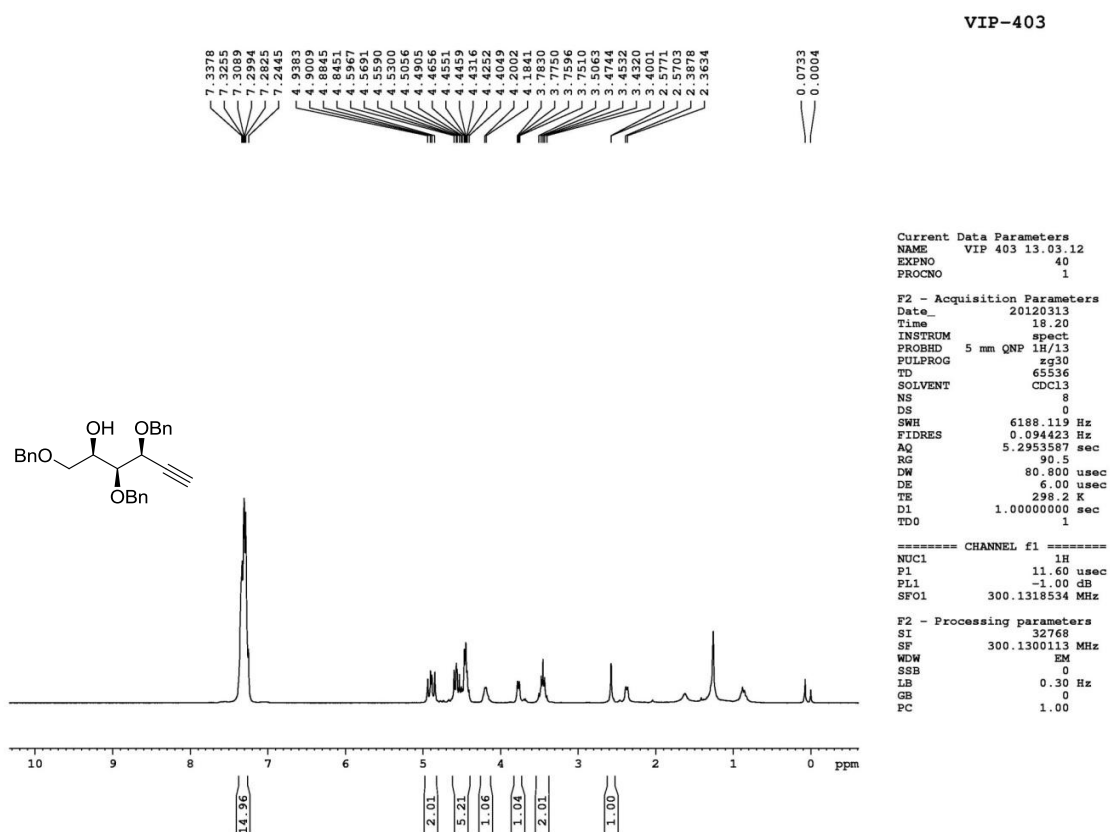
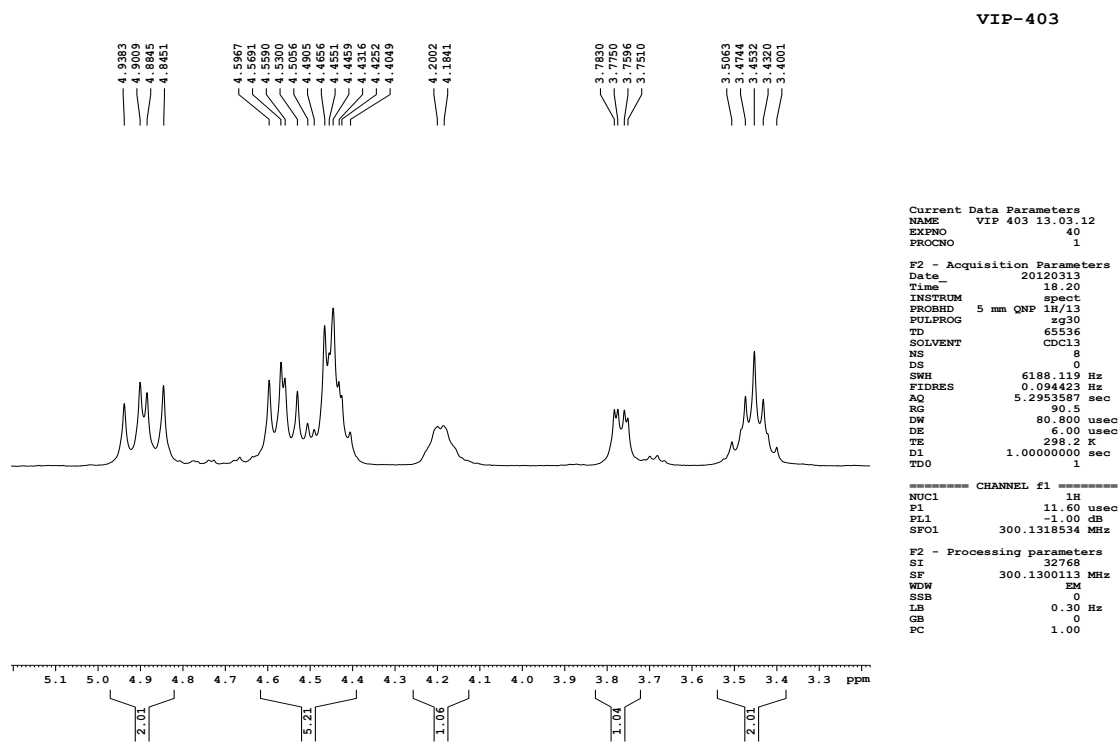
¹H NMR Spectrum of 12a



¹³C NMR Spectrum of 12a

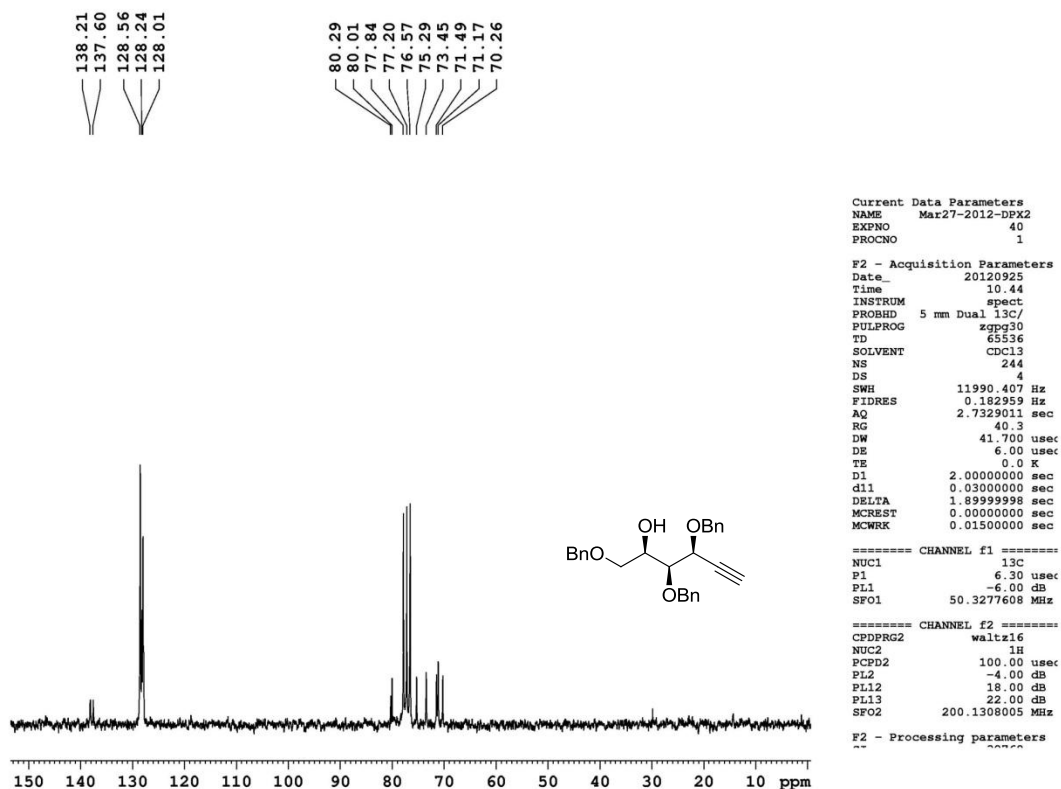


HSQC Spectra of 12a

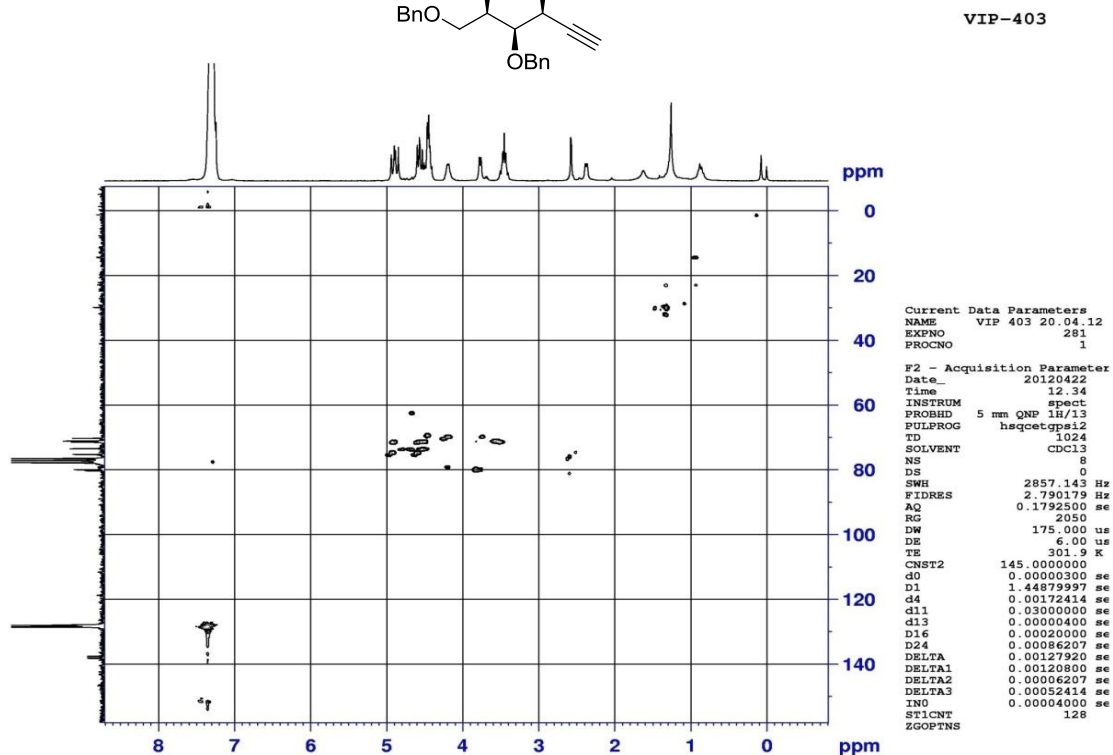
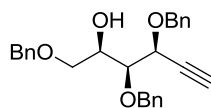
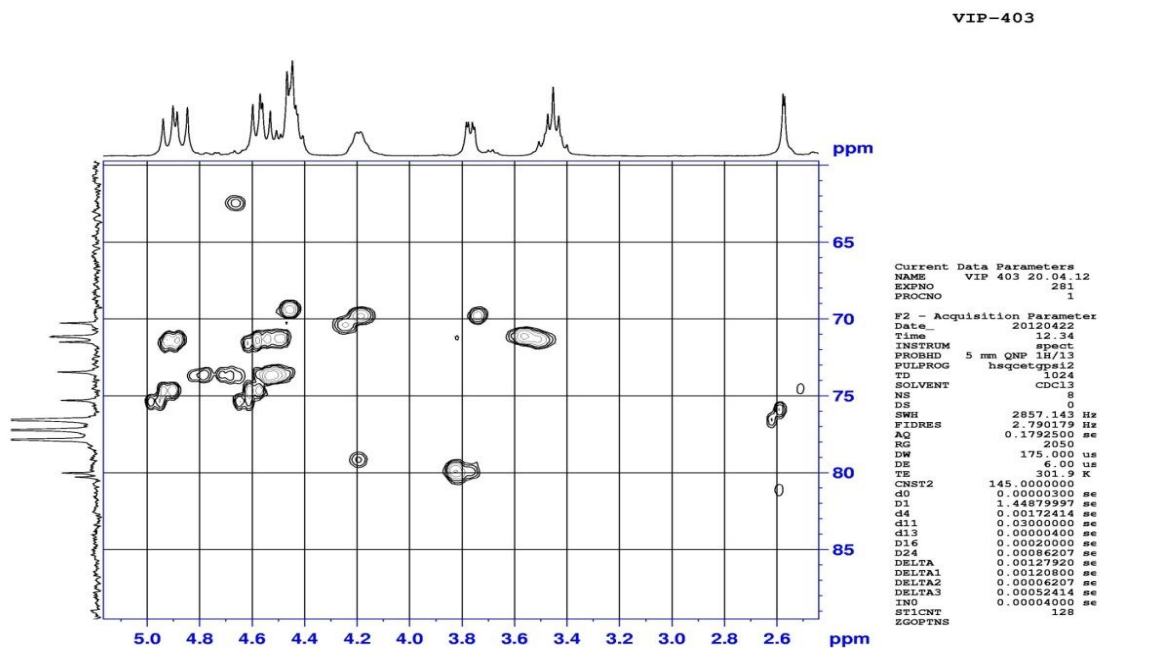


¹H NMR Spectrum of 13a

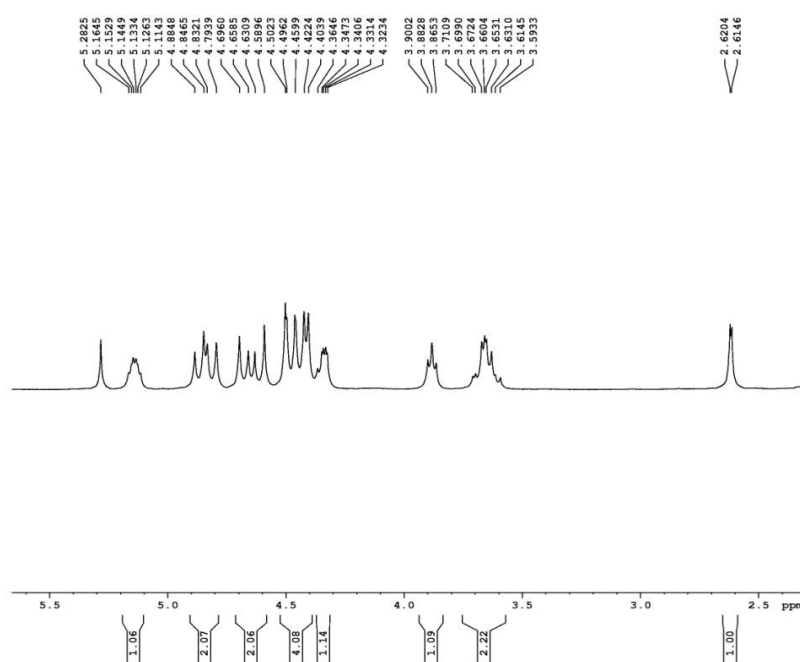
VIP-403



^{13}C NMR Spectrum of 13a



HSQC Spectra of 13a



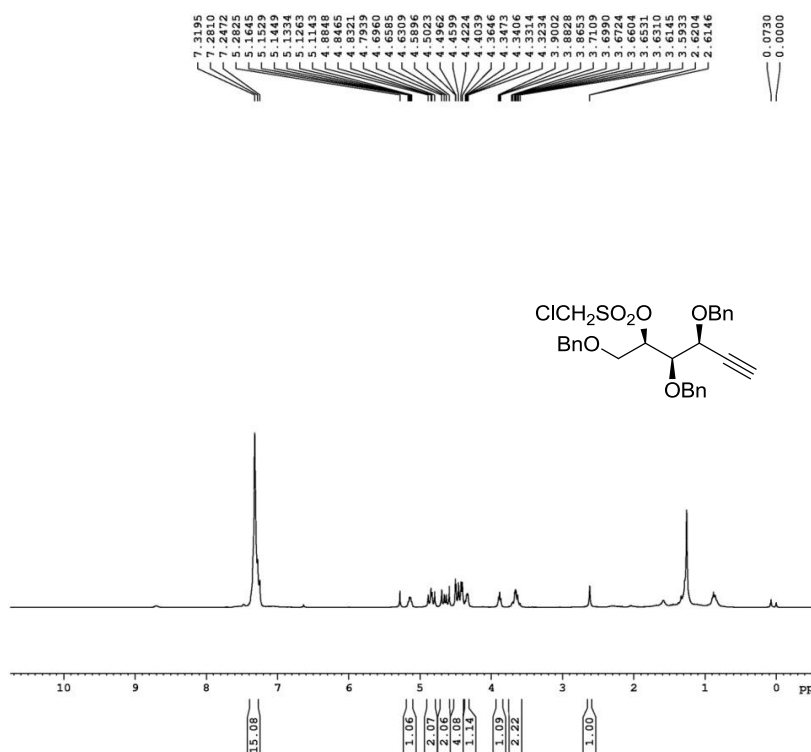
VIP 406

Current Data Parameters
NAME vip 406 02.0
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 201204
Time 17:
INSTRUM spect
PROBHD 5 mm QNP 1H/
PULPROG zg
TD 655
SOLVENT CDC
NS
DS
SWH 6188.1
FIDRES 0.0944
AQ 5.29535
RG 1
DW 80.8
DE 6
TE 297
D1 1.000000
TDO

===== CHANNEL f1 =====
NUC1
P1 11
PL1 -1
SFO1 300.13185

F2 - Processing parameters
SI 327
SF 300.13001
WDW
SSB
LB 0
GB
PC 1



VIP 406

Current Data Parameters
NAME vip 406 02.0
EXPNO 1
PROCNO 1

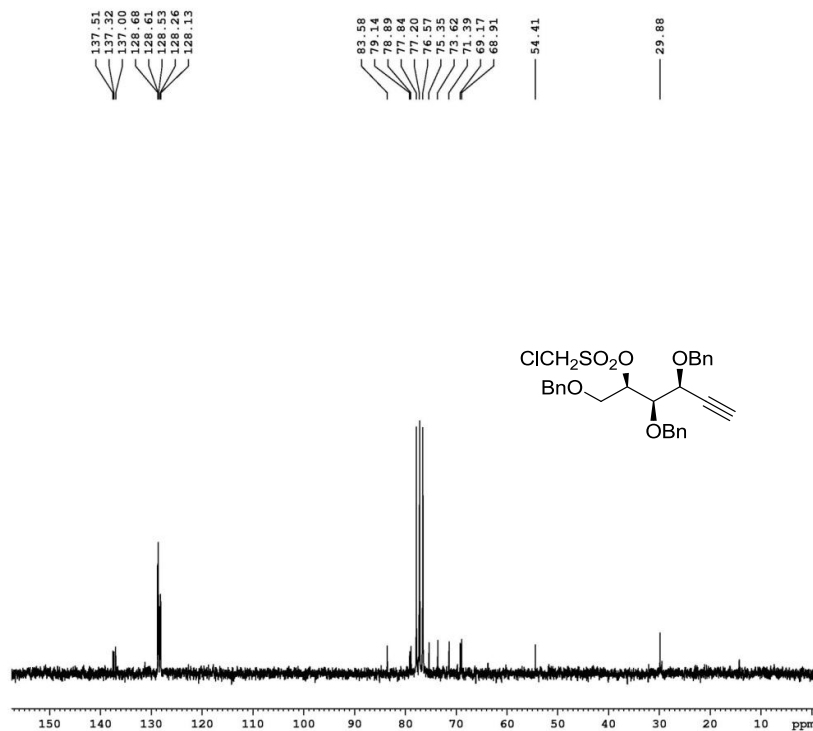
F2 - Acquisition Parameters
Date_ 201204
Time 17:
INSTRUM spect
PROBHD 5 mm QNP 1H/
PULPROG zg
TD 655
SOLVENT CDC
NS
DS
SWH 6188.1
FIDRES 0.0944
AQ 5.29535
RG 1
DW 80.8
DE 6
TE 297
D1 1.000000
TDO

===== CHANNEL f1 =====
NUC1
P1 11
PL1 -1
SFO1 300.13185

F2 - Processing parameters
SI 327
SF 300.13001
WDW
SSB
LB 0
GB
PC 1

¹H NMR Spectrum of 14a

VIP-406



Current Data Parameters
NAME VIP 406 10.04.12
EXPNO 30
PROCNO 1

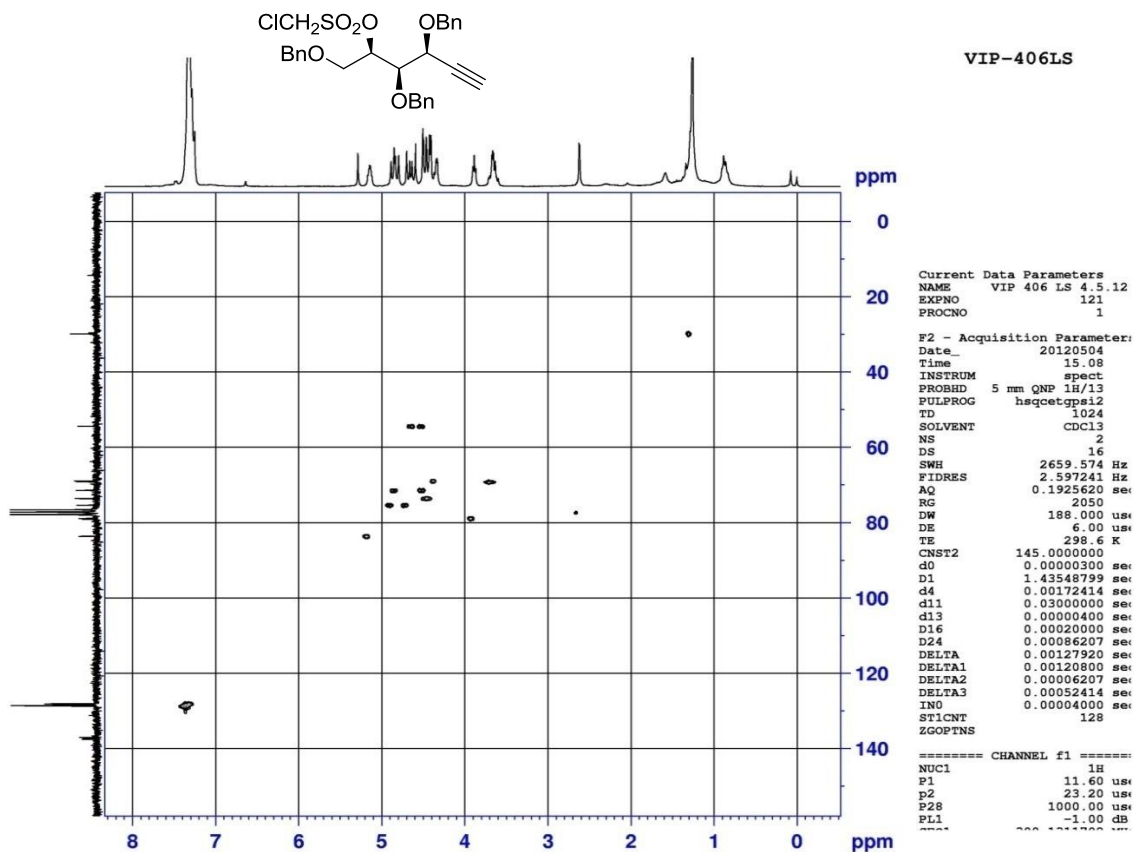
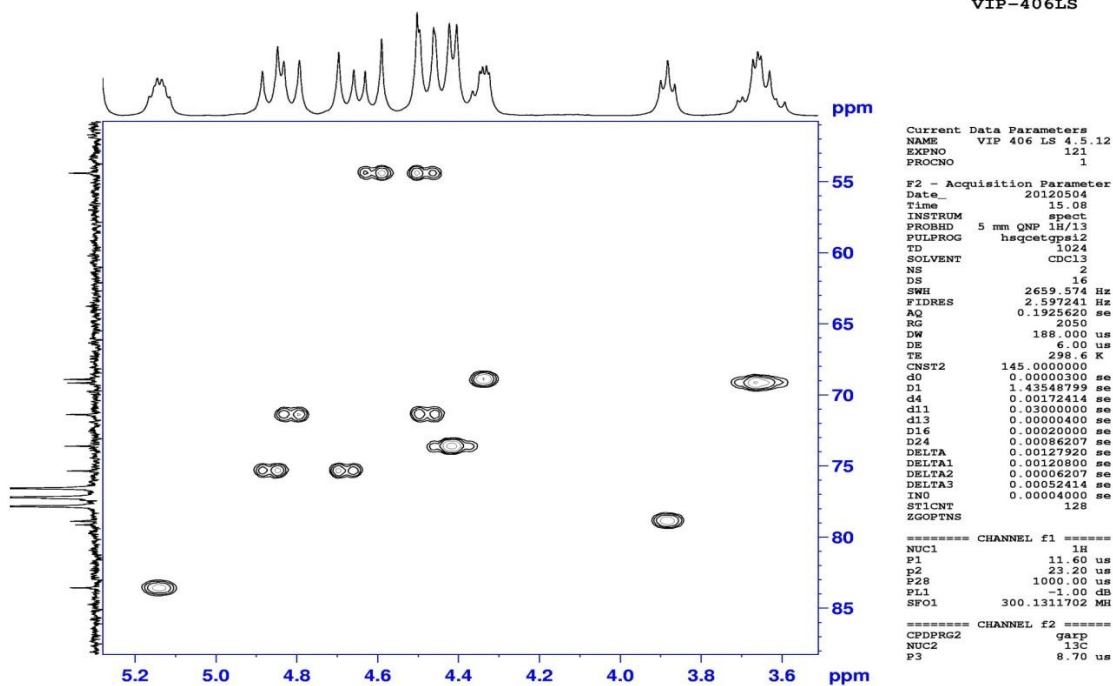
F2 - Acquisition Parameters
Date_ 20121009
Time 16.51
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 319
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 se
RG 50.8
DW 41.700 us
DE 6.00 us
TE 0.0 K
D1 2.00000000 se
d11 0.03000000 se
DELTA 1.89999998 se
MCREST 0.00000000 se
MCWRR 0.01500000 se

===== CHANNEL f1 =====
NUC1 13C
P1 6.30 us
PL1 -6.00 dB
SFO1 50.3277608 MHz

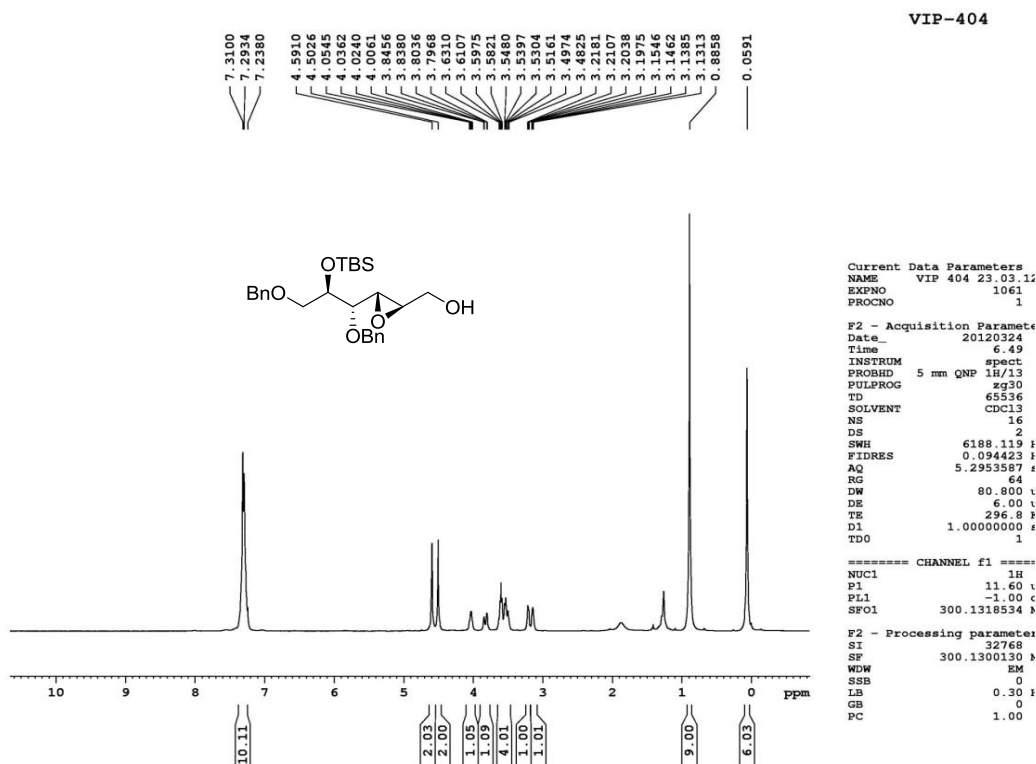
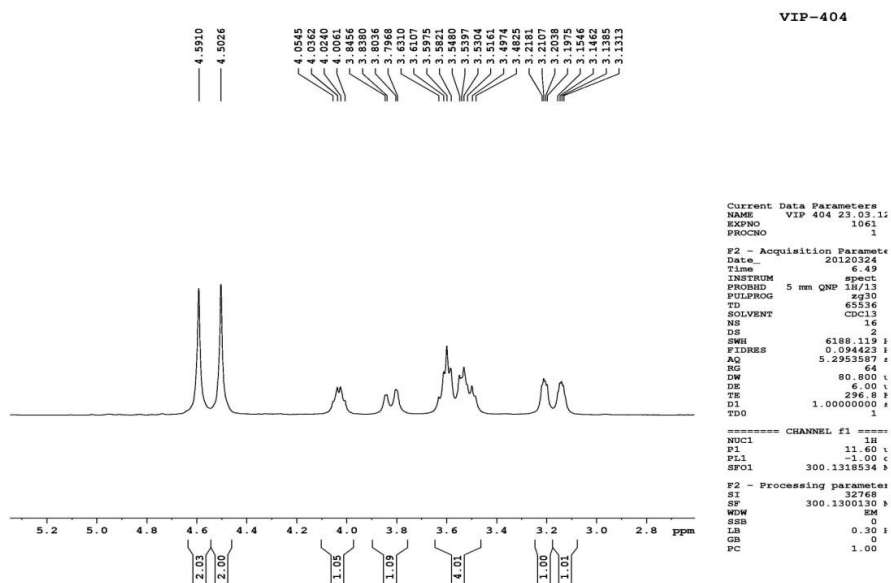
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.1308005 MHz

^{13}C NMR Spectrum of 14a

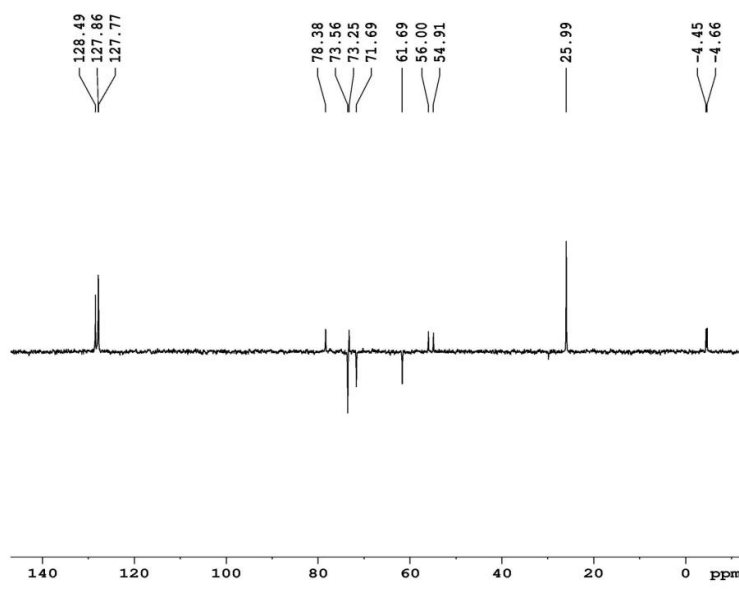
VIP-406LS



HSQC Spectra of 14a



¹H NMR Spectrum of 10b



VIP-404

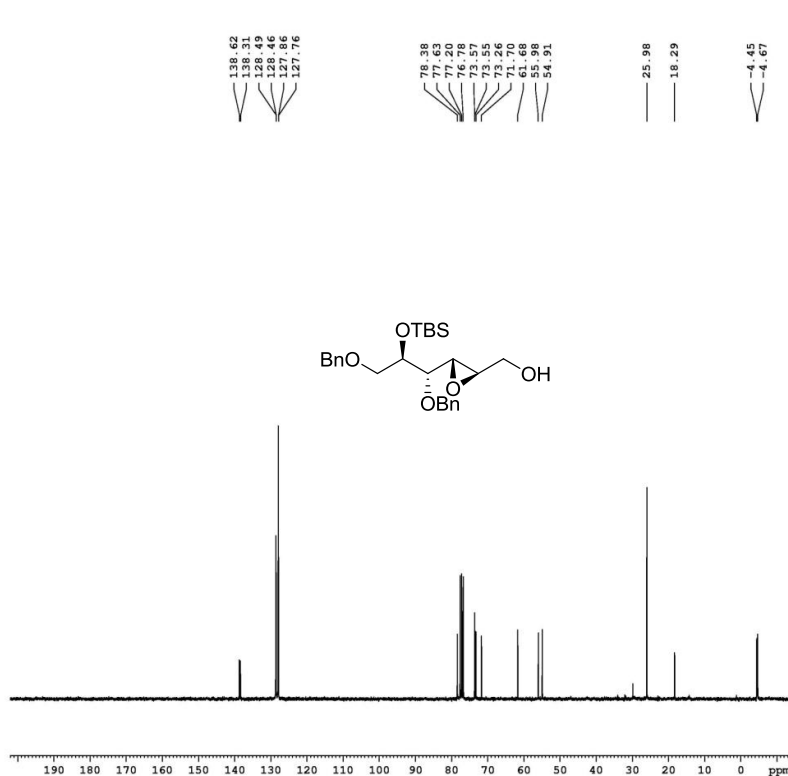
Current Data Parameters
NAME VIP 404 28.03.12
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120926
Time 12.33
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1824.6
DW 41.700 usec
DE 6.00 usec
TE 300.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00008002 sec
MCREST 0.00000000 sec
MCNRC 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.30 usec
P2 12.60 usec
PL1 -6.00 dB
SFO1 50.3277608 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 8.35 usec
p4 16.70 usec
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 0.00 dB
SFO2 200.1308005 MHz

F2 - Processing parameters
SI 22768
SF 50.327219 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



VIP-404

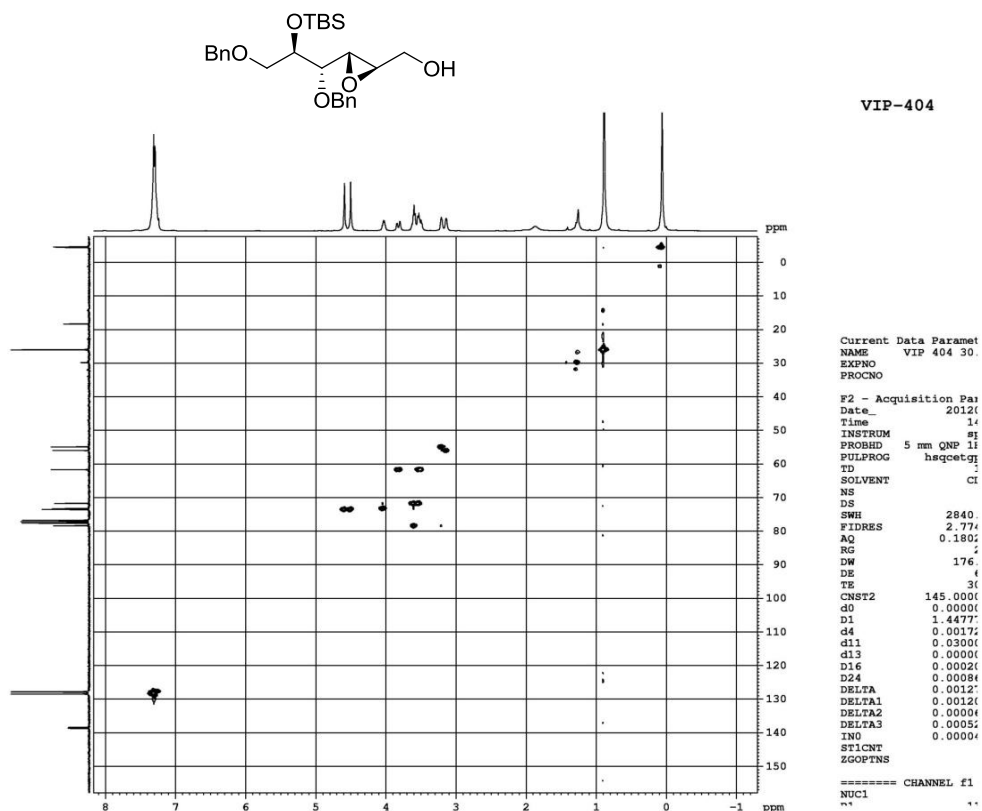
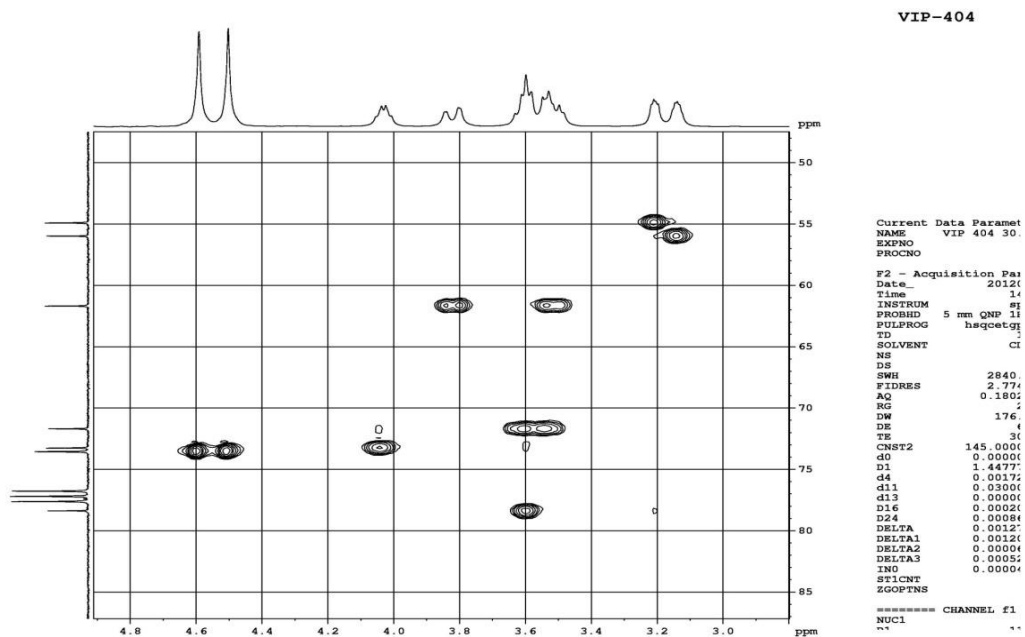
Current Data Parameters
NAME VIP 404 23.0.
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 201203
Time 6.
INSTRUM spe
PROBHD 5 mm QNP 1H/
PULPROG zgpg
TD 655
SOLVENT CDCl3
NS 10
DS 18028.8
FIDRES 0.2750
AQ 1.81758
RG 12
DW 27.7
DE 6.
TE 297
D1 2.000000
d11 0.030000
DELTA 1.899999
TD0

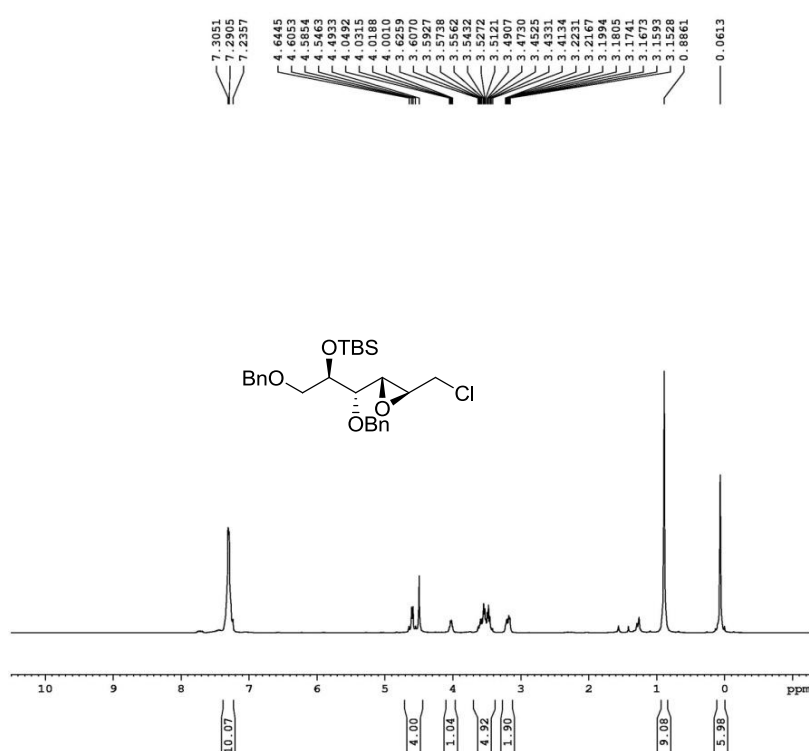
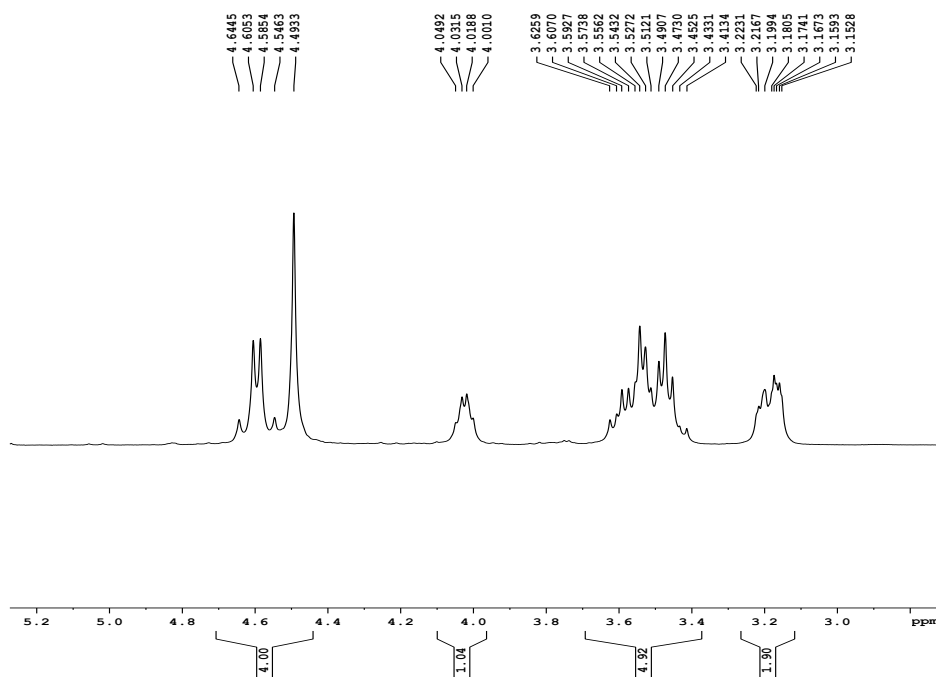
===== CHANNEL f1 =====
NUC1 13C
P1 8.
PL1 -3.
SFO1 75.47529

===== CHANNEL f2 =====
CPDPRG2 waltz
NUC2 1H
PCPD2 80.
PL2 -1.
PL12 17

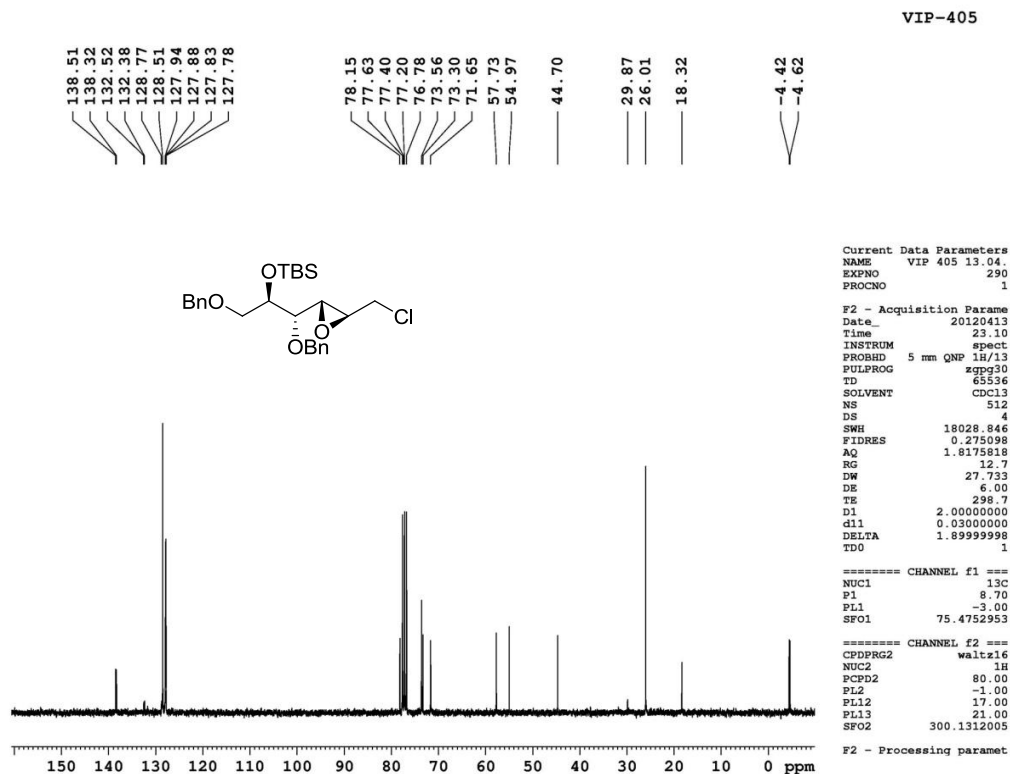
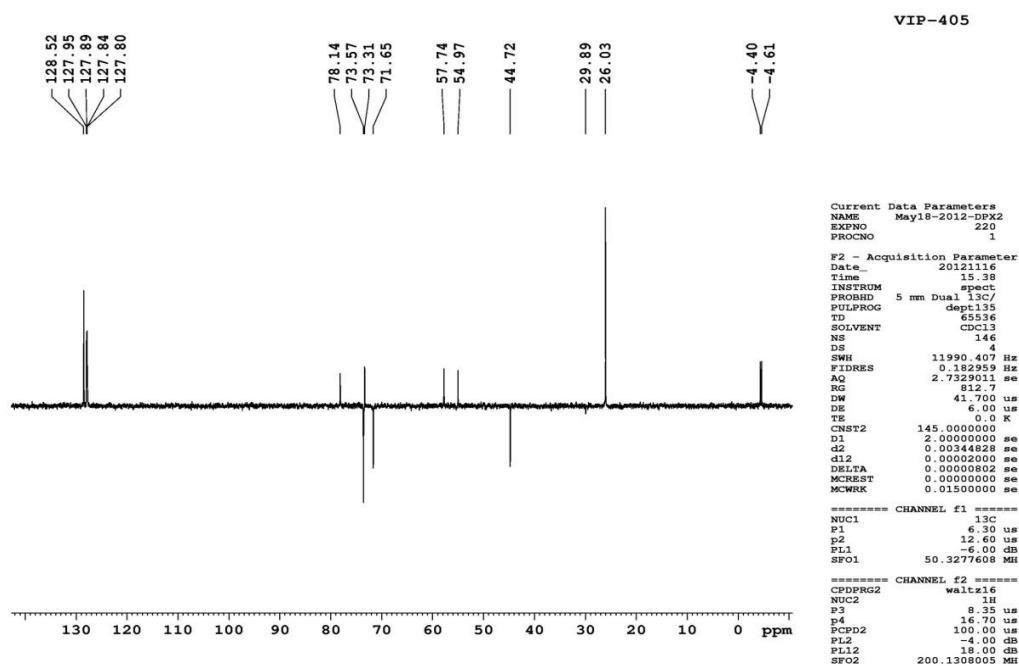
¹³C NMR Spectrum of 10b



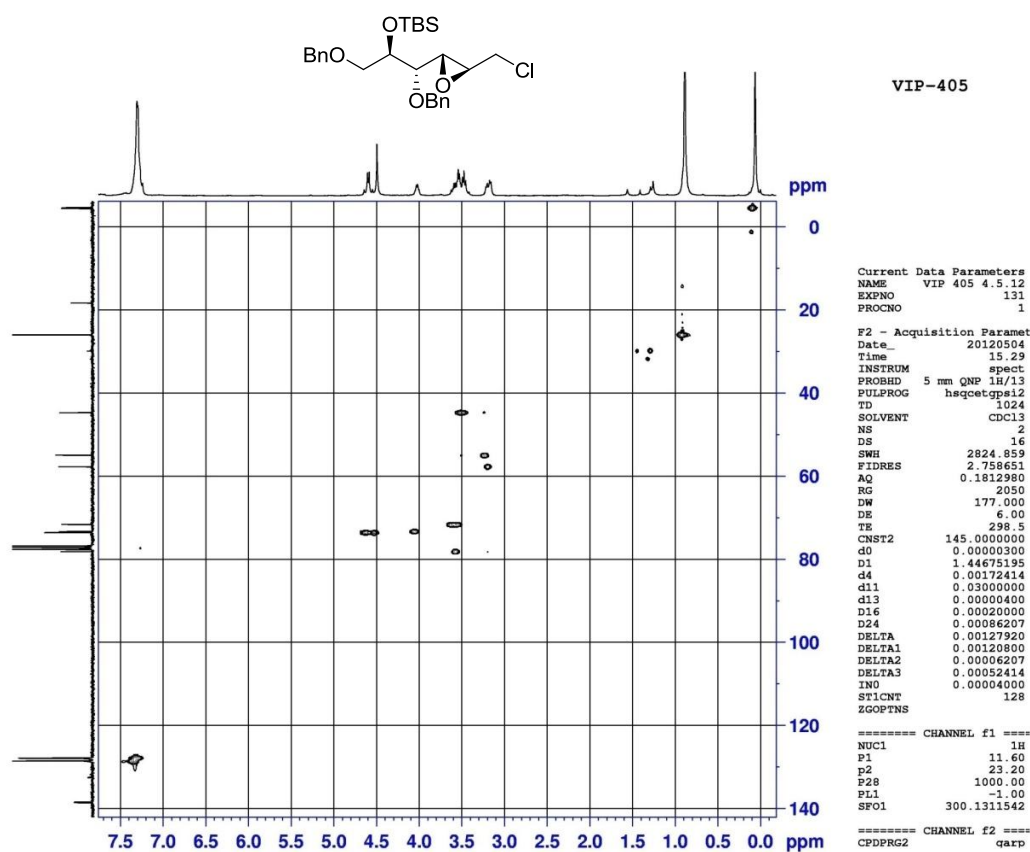
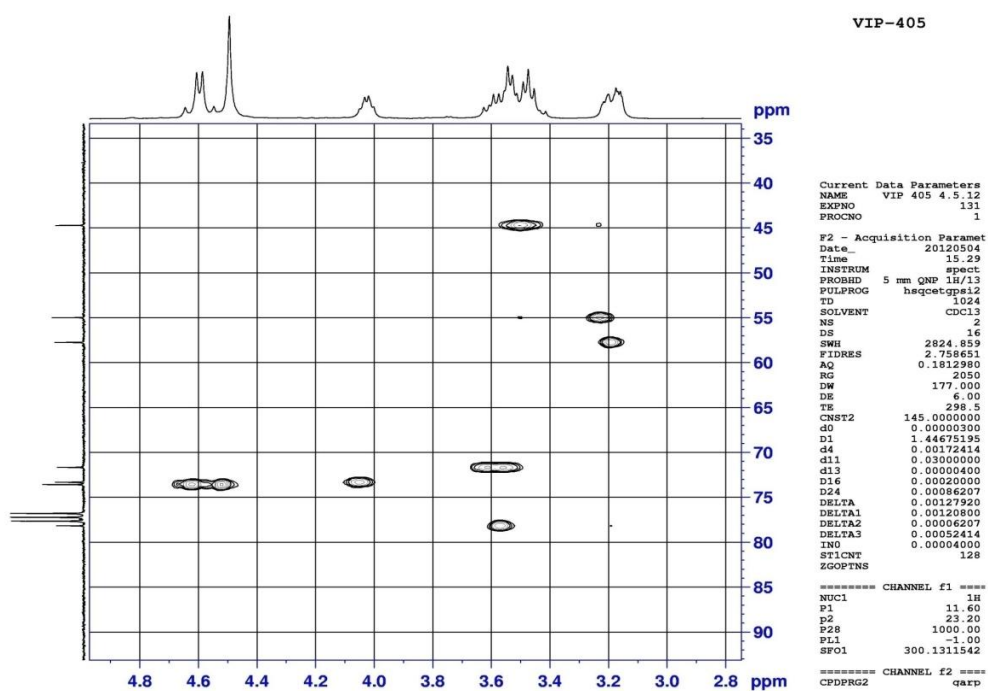
HSQC Spectra of 10b



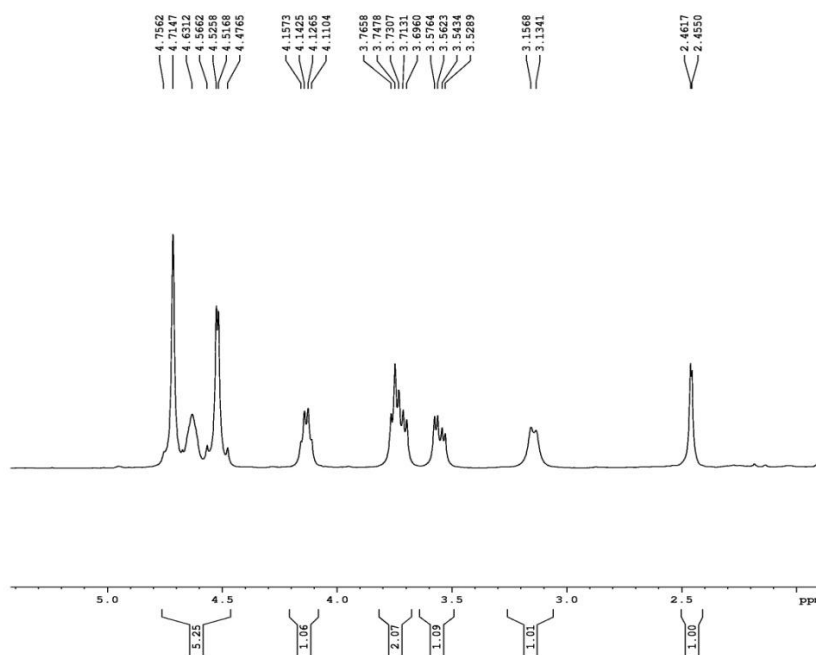
¹H NMR Spectrum of 11b



¹³C NMR Spectrum of 11b



HSQC Spectra of 11b

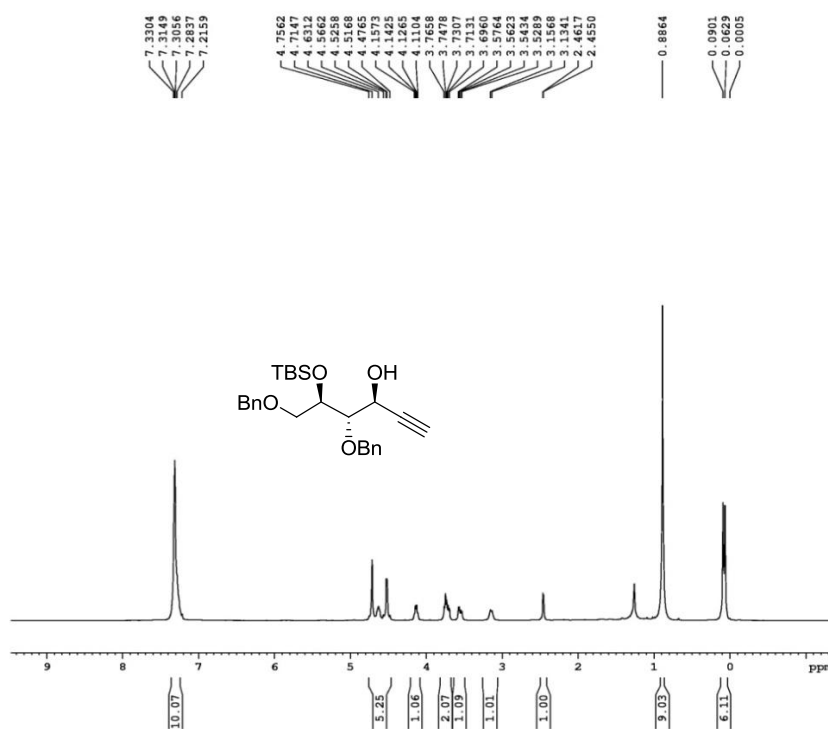


Current Data Parameters
NAME VIP 407 01.06.11
EXPNO 110
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120601
Time 16.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.113 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 32
DW 80.800 use
DE 6.00 use
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.60 use
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300198 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



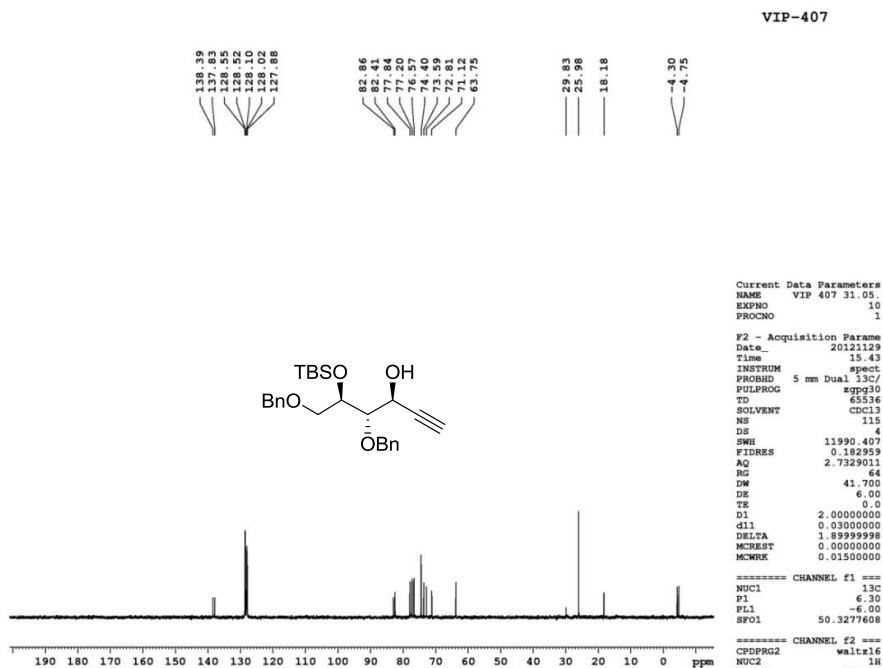
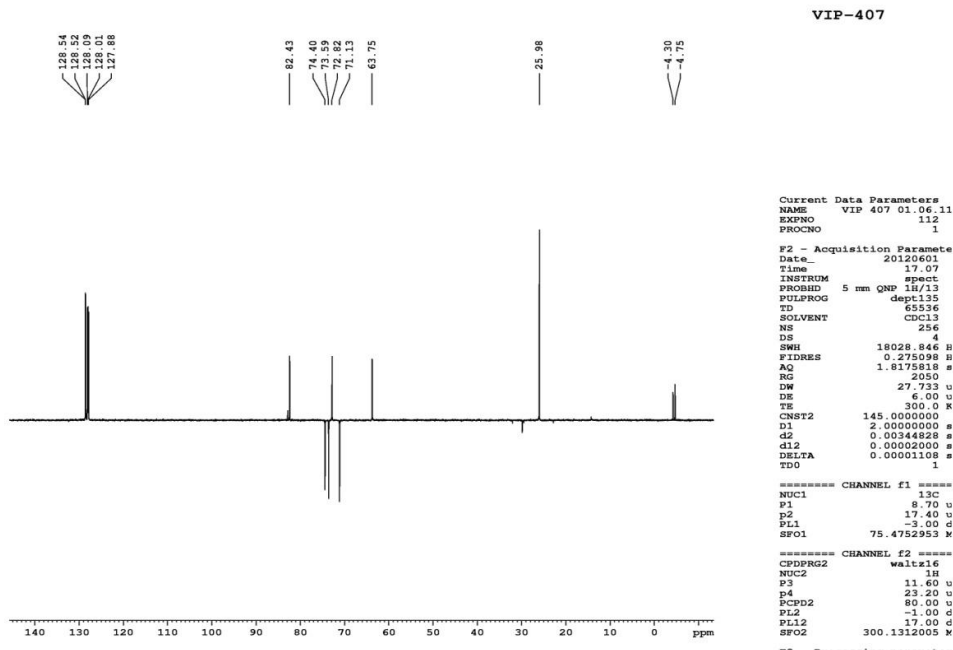
Current Data Parameters
NAME VIP 407 01.06.11
EXPNO 110
PROCNO 1

F2 - Acquisition Parameters
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Time 16.34
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.113 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 32
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D1 1.00000000 sec
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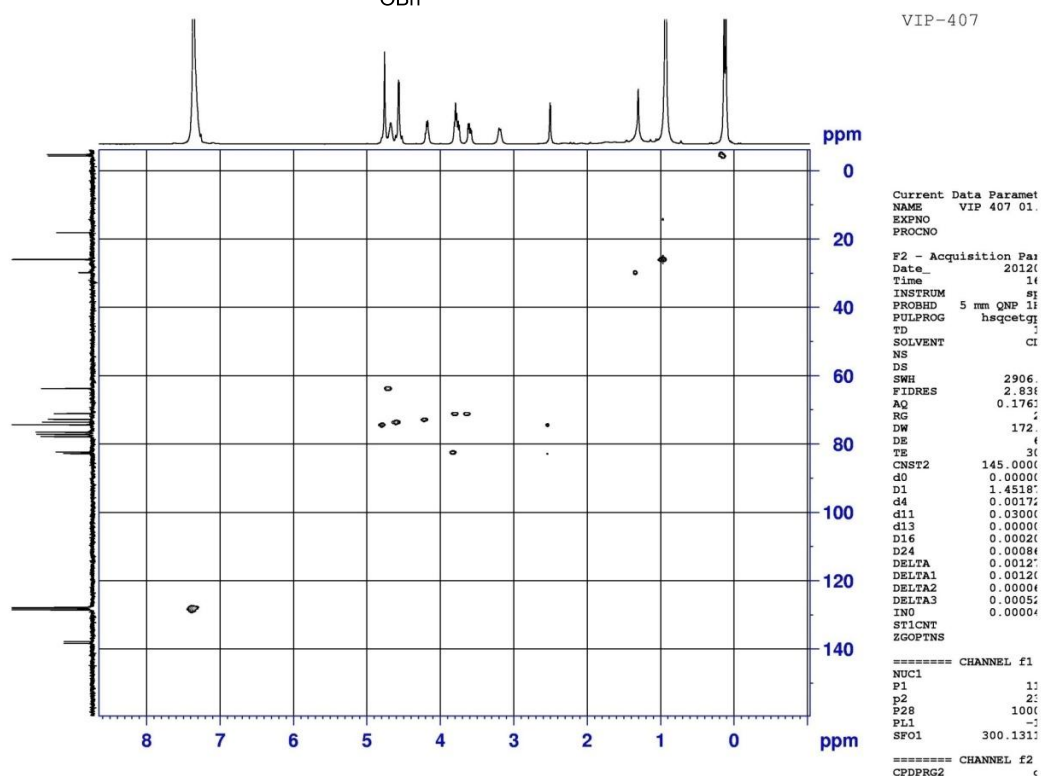
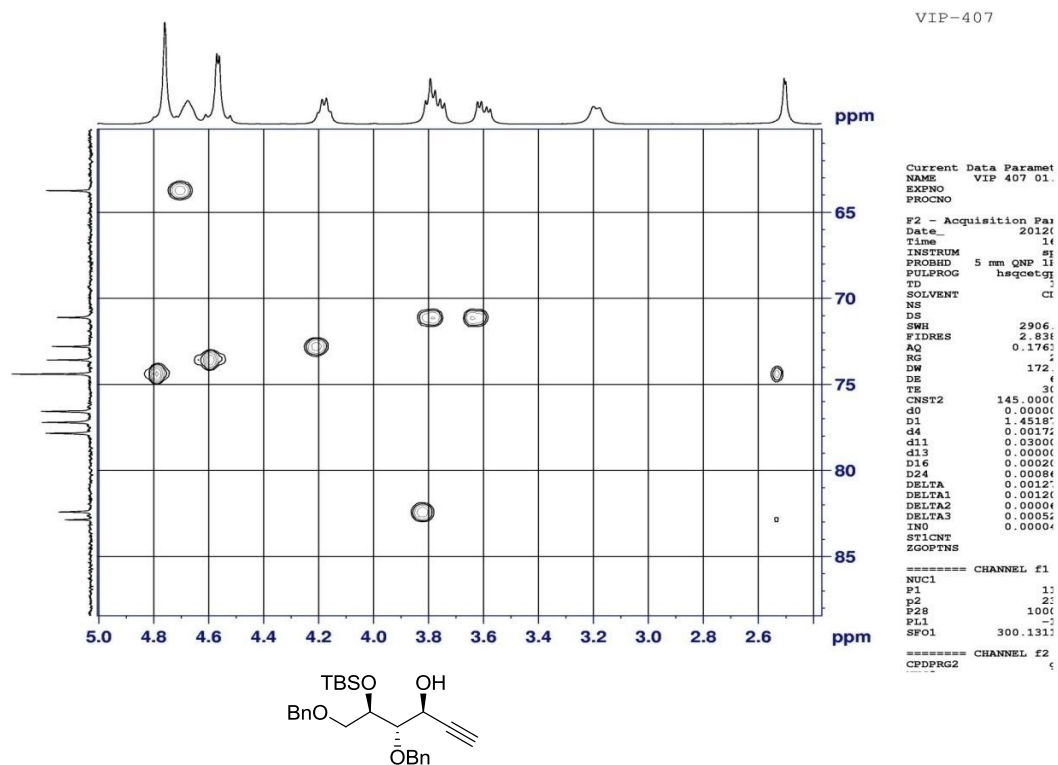
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P1 11.60 use
PL1 -1.00 dB
SFO1 300.1318534 MHz

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SSB 0
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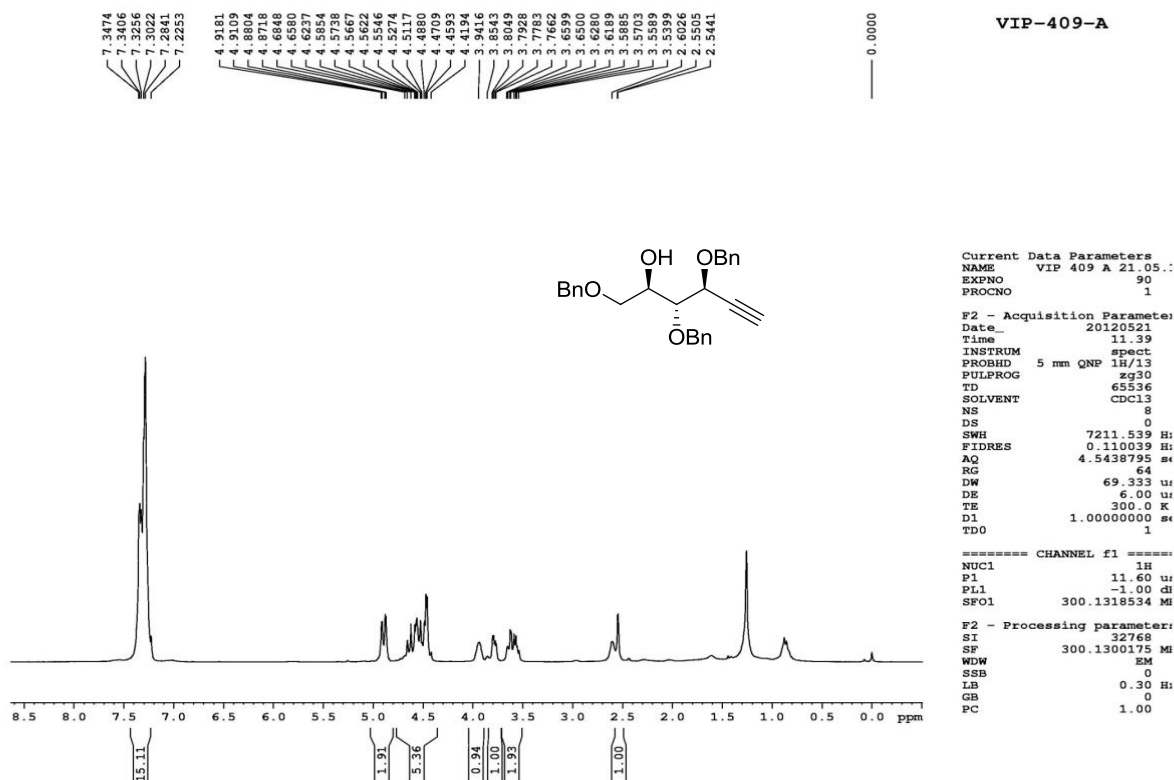
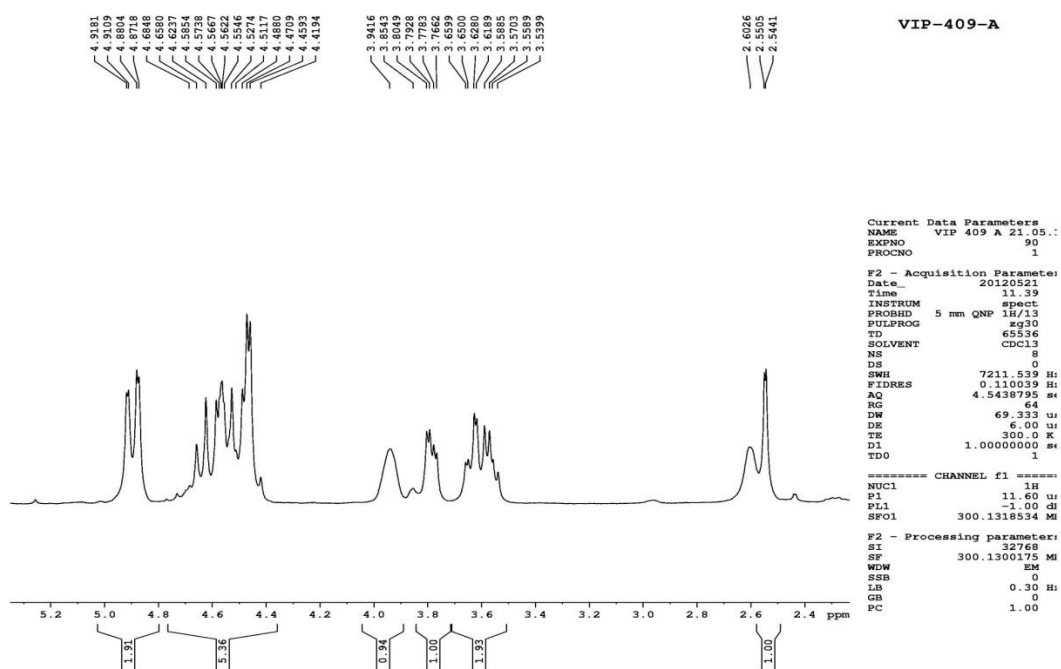
¹H NMR Spectrum of 12b



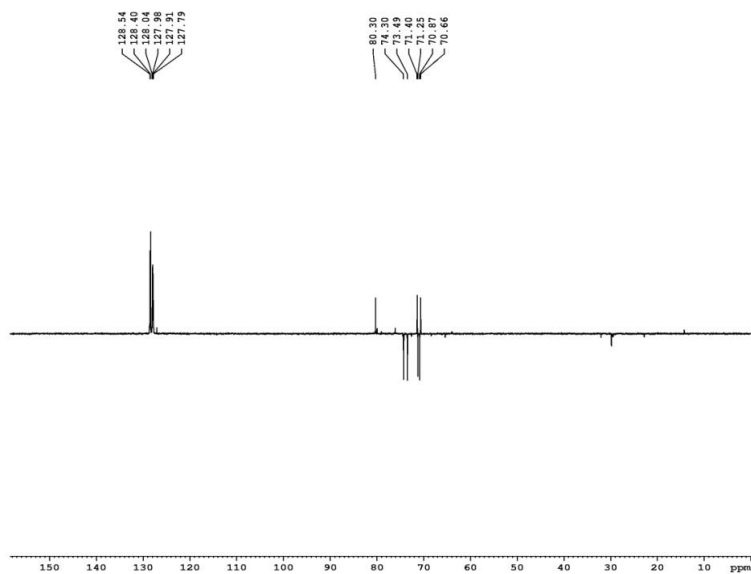
¹³C NMR Spectrum of 12b



HSQC Spectra of 12b



¹H NMR Spectrum of 13b



VIP-409-A

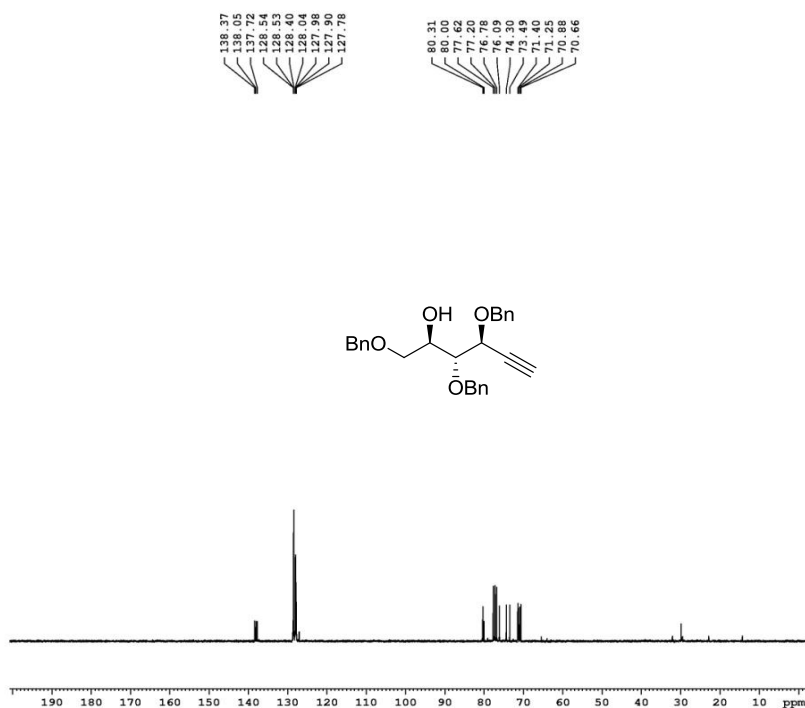
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Current Data Parameters:
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EXPNO     990
PROCNO    1

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SOLVENT   CDCl3
NS         250
DS         4
SWH        18028.846
FIDRES     0.275098
AQ         1.8175811
RG         2050
DE         27.73
TE         299.2
CNET2     145.000000
D1         2.0000000
d2         0.00344824
d12        0.0000200
DELTA     0.0000110
TDO       1

===== CHANNEL f1 =====
NUC1       13C
P1         8.70
PL1        -3.00
SFO1       75.4752951

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P2         11.40
PL2        -1.00
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VIP-409-A

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EXPNO     960
PROCNO    1

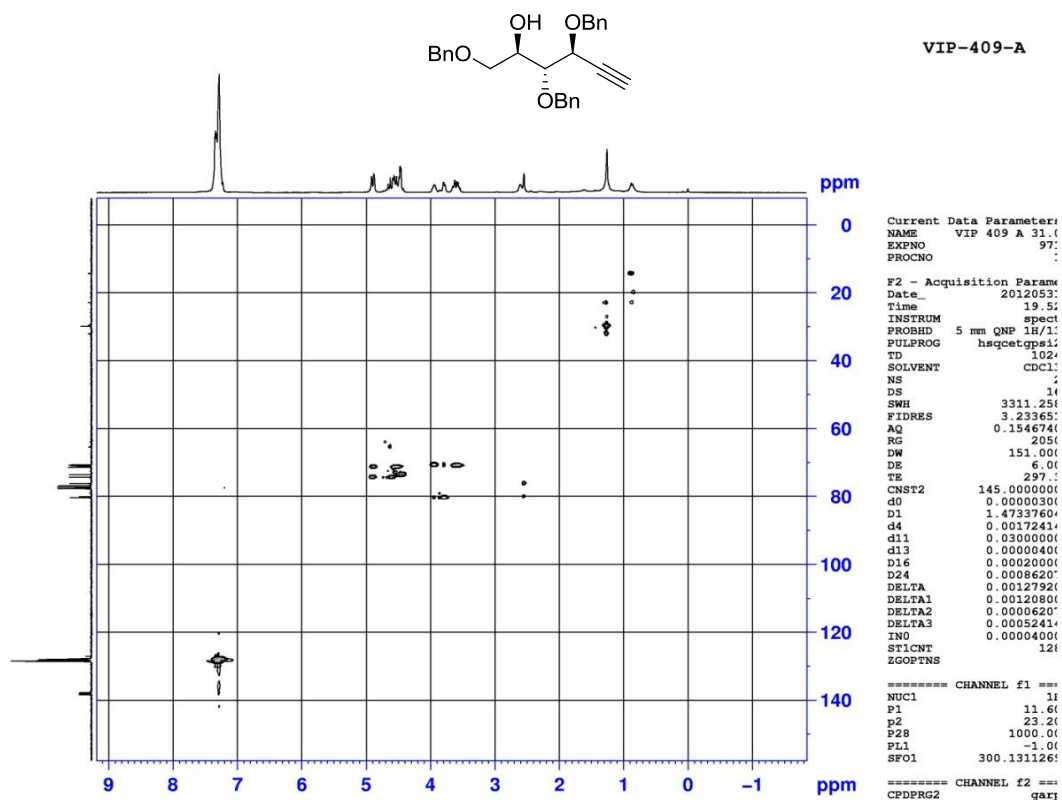
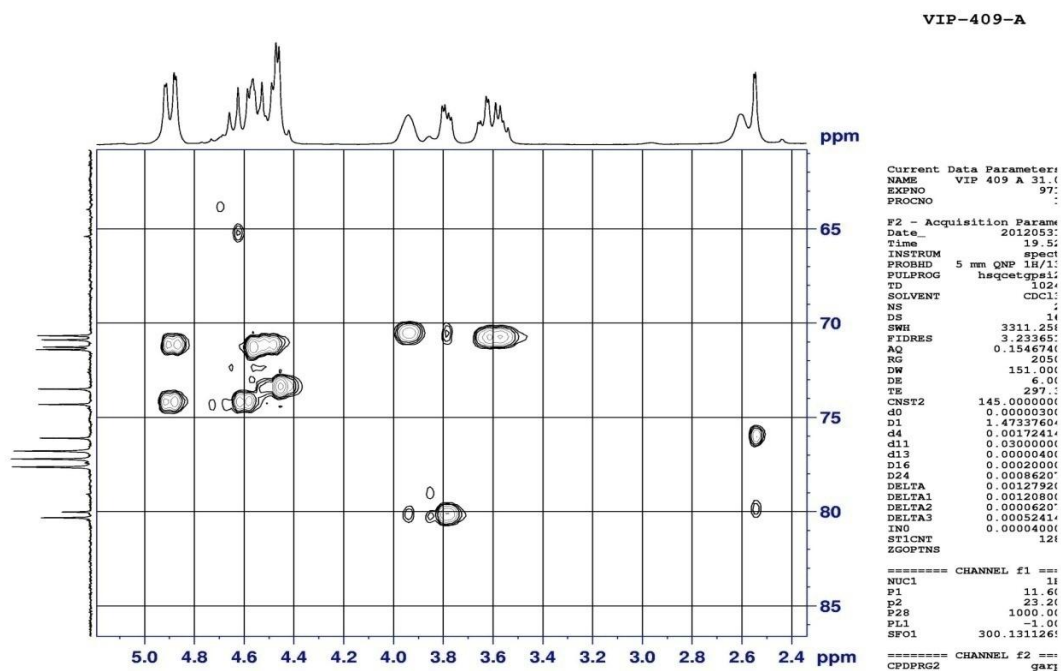
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DS         4
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AQ         1.8175818 sec
RG         16
DE         27.733 usec
TE         299.6 K
D1         2.0000000 sec
d11        0.0300000 sec
DELTA     1.8999998 sec
TDO       1

===== CHANNEL f1 =====
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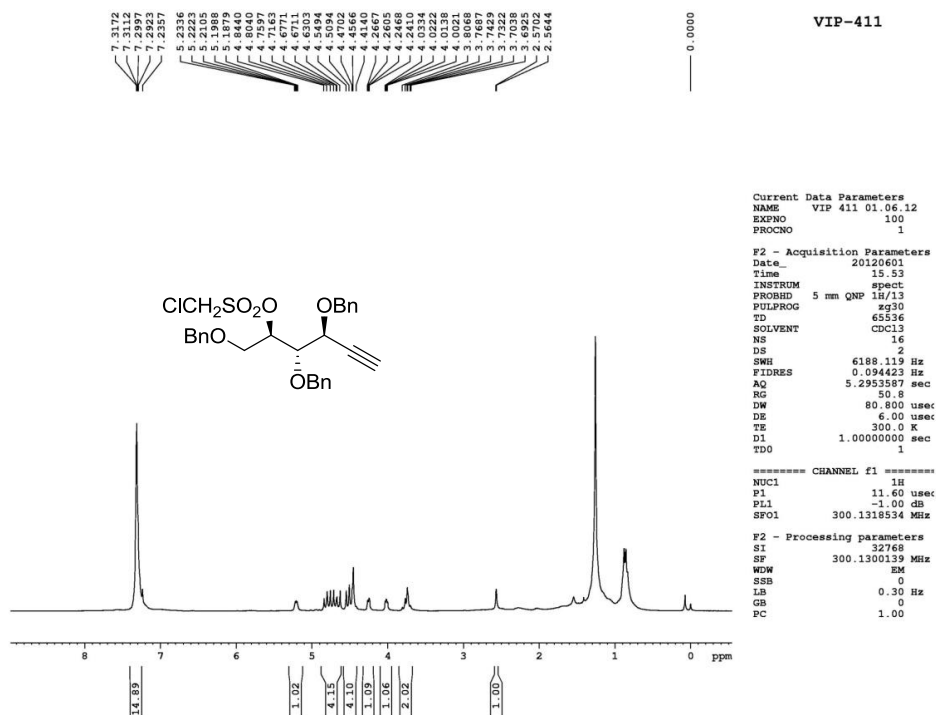
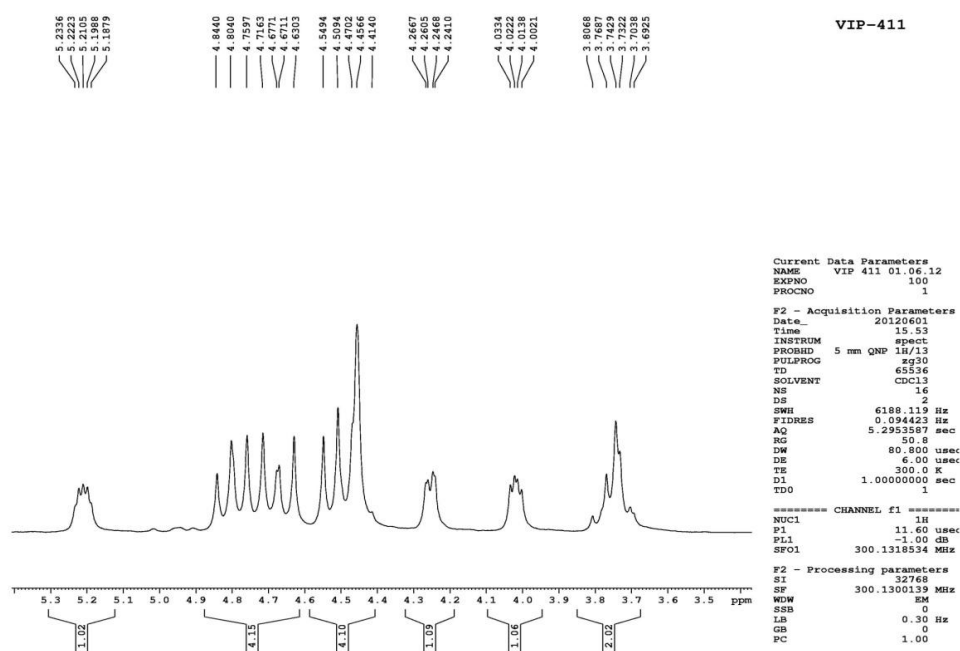
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NUC2       1H
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PL12       17.00 dB
PL13       21.00 dB
SFO2       400.1312005 MHz

F2 - Processing parameters:
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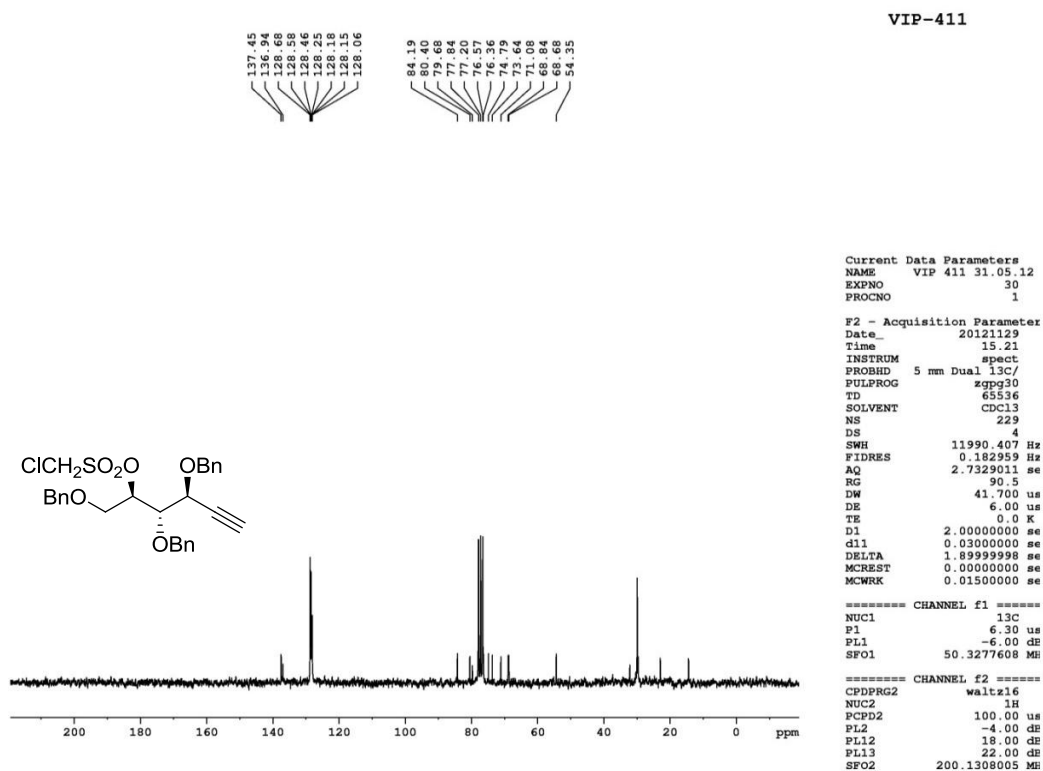
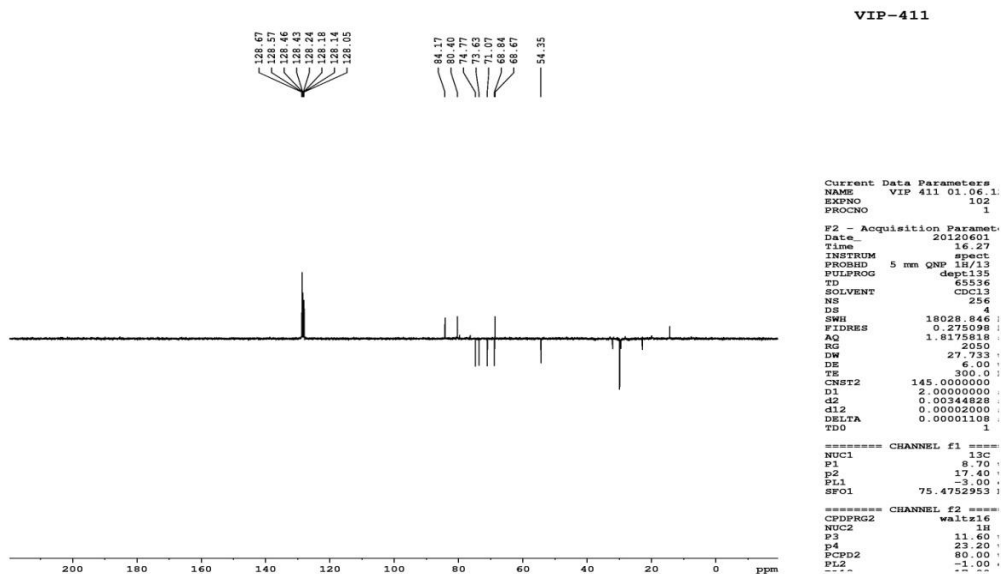
¹³C NMR Spectrum of 13b



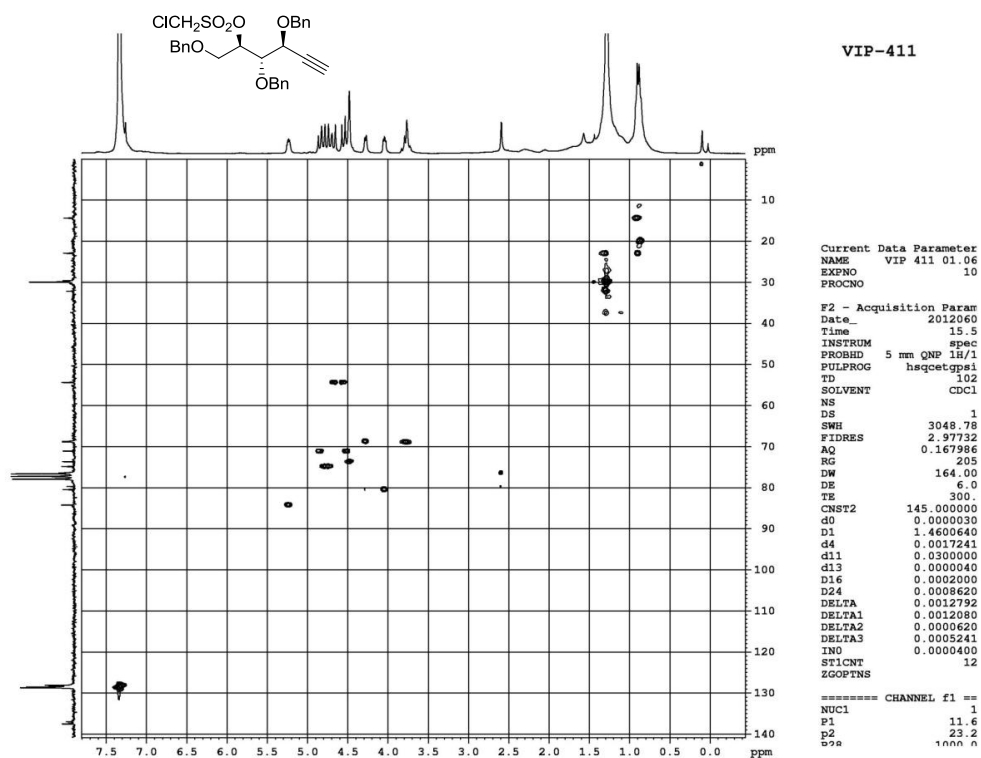
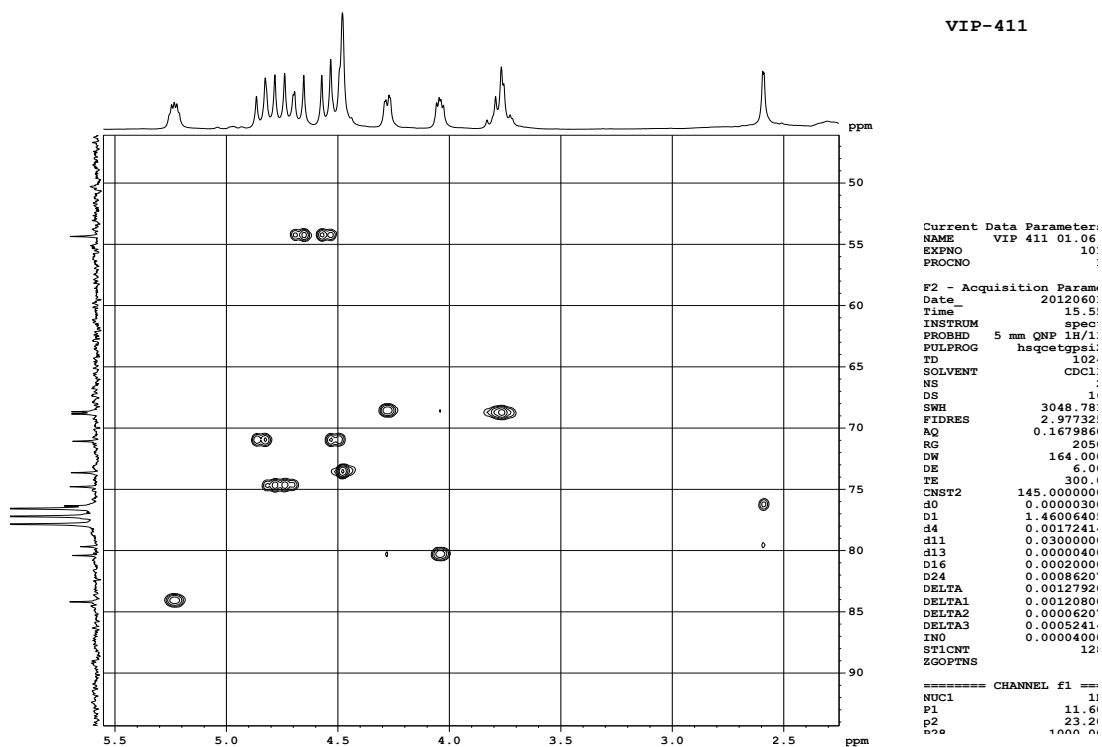
HSQC Spectra of 13b



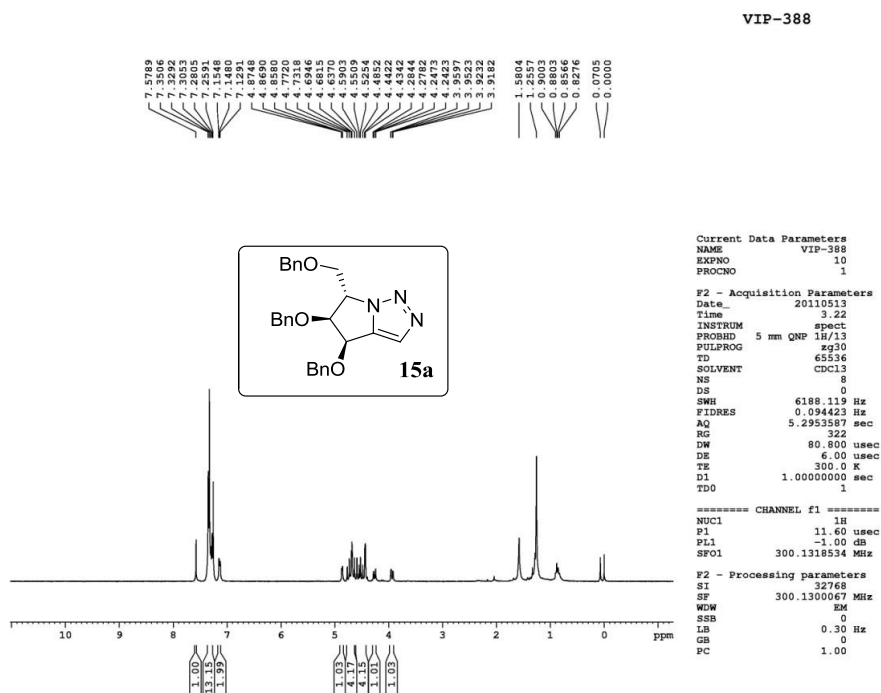
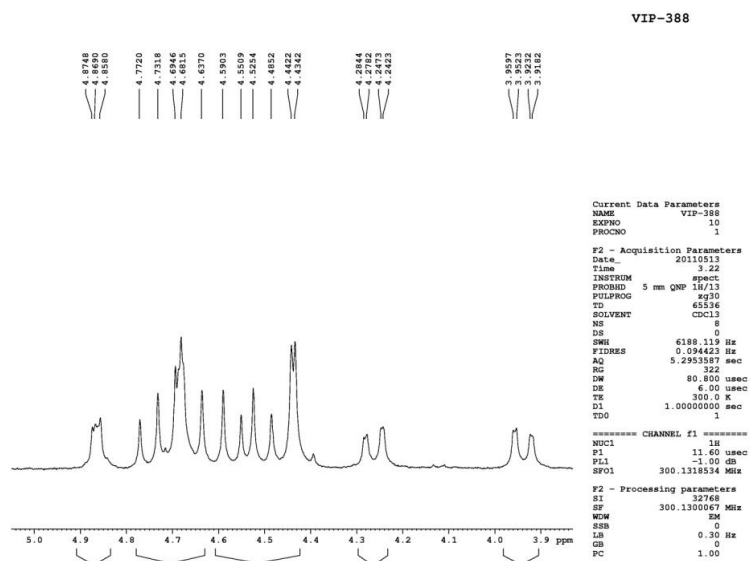
¹H NMR Spectrum of 14b



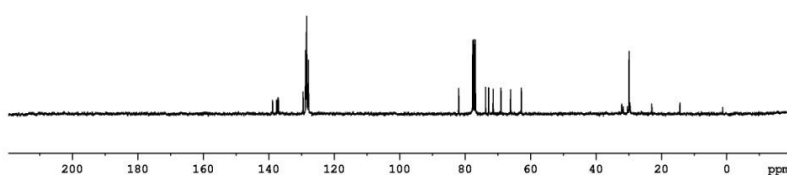
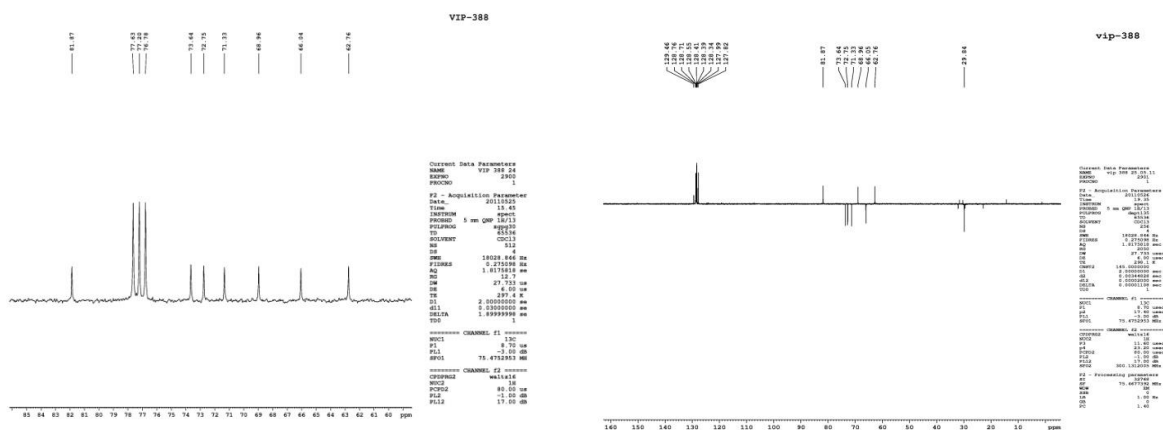
¹³C NMR Spectrum of 14b



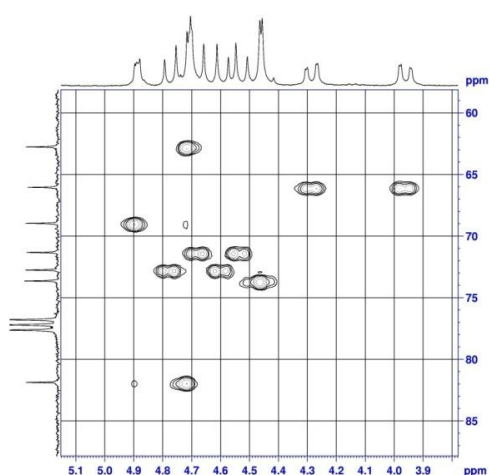
HSQC Spectra of 14b



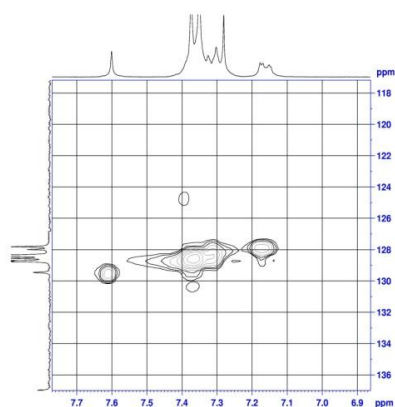
¹H NMR Spectrum of 15a



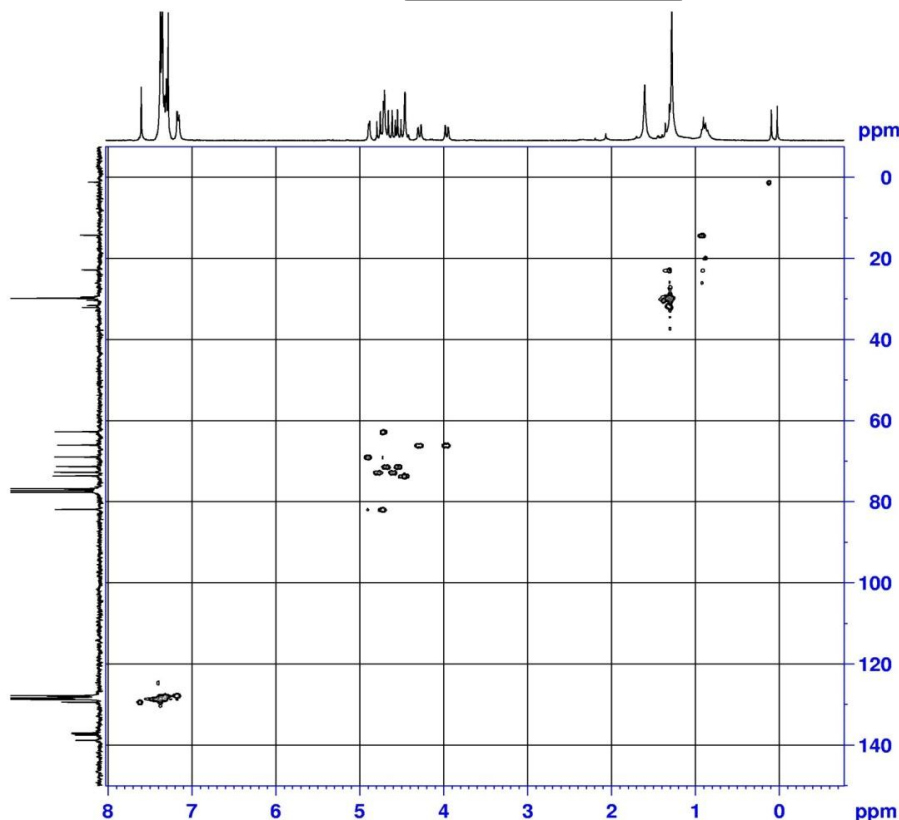
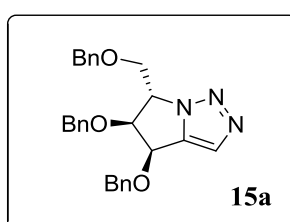
¹³C NMR Spectrum of 15a



VIP-388



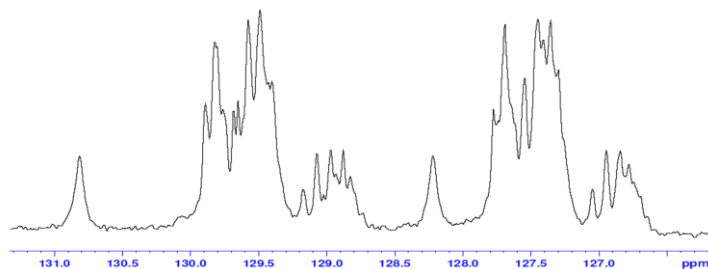
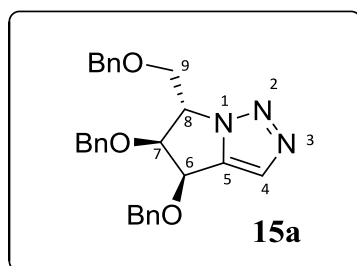
VIP-388



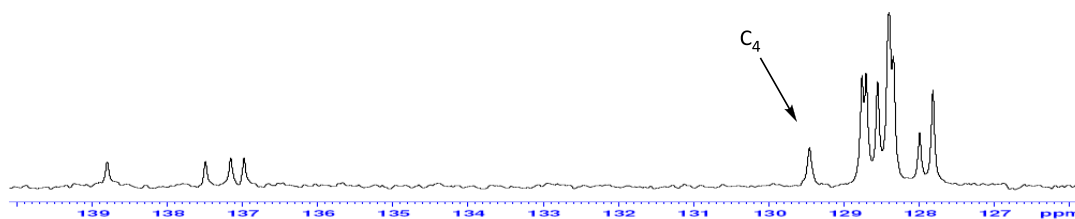
VIP-388

HSQC Spectra of 15a

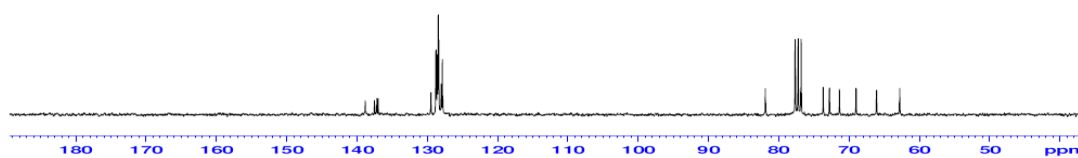
← $J = 195 \text{ Hz}$ →



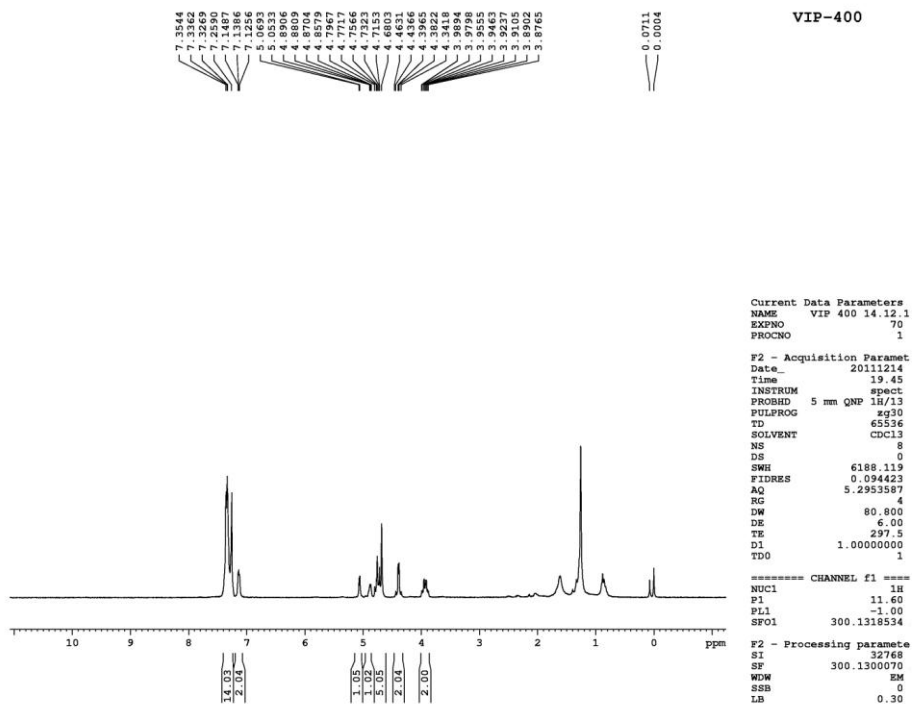
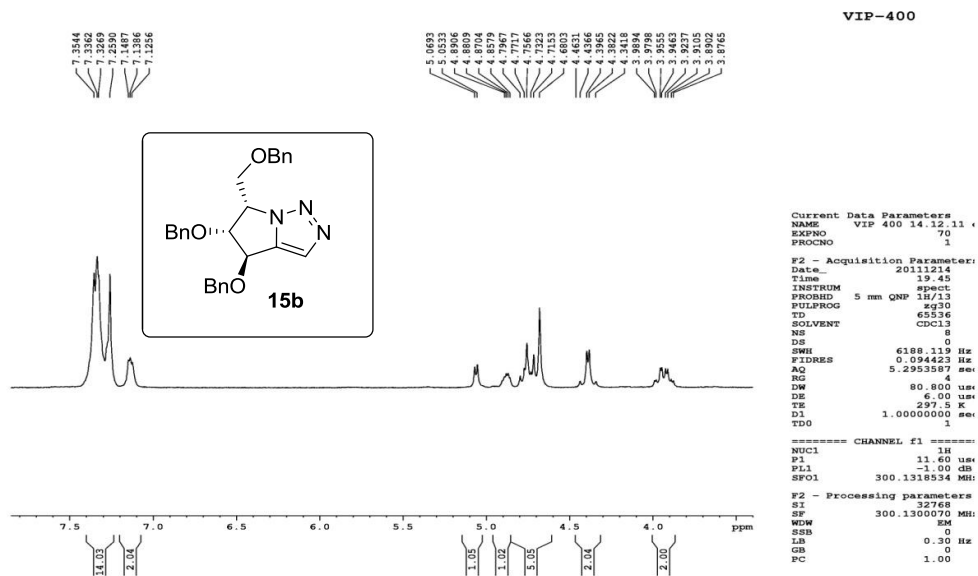
Proton coupled ^{13}C Spectra of 15a



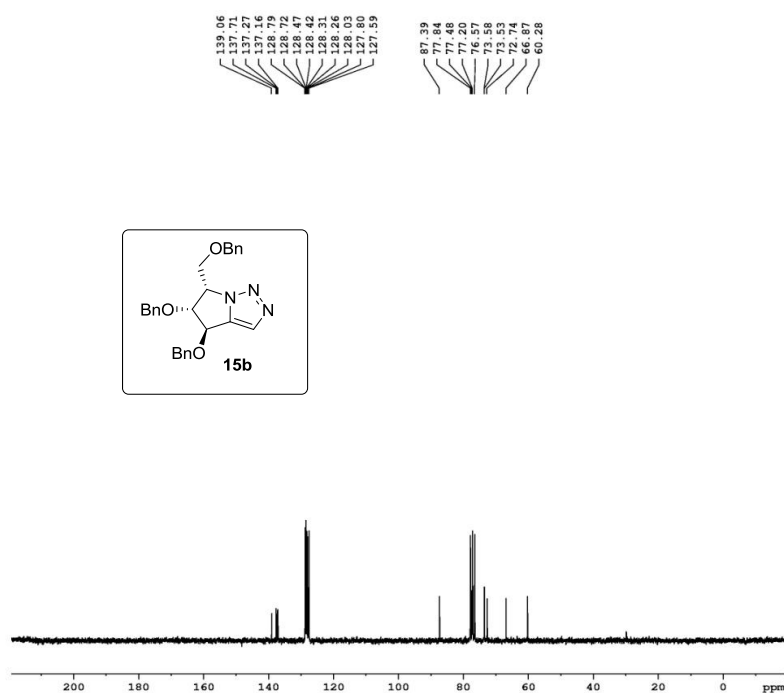
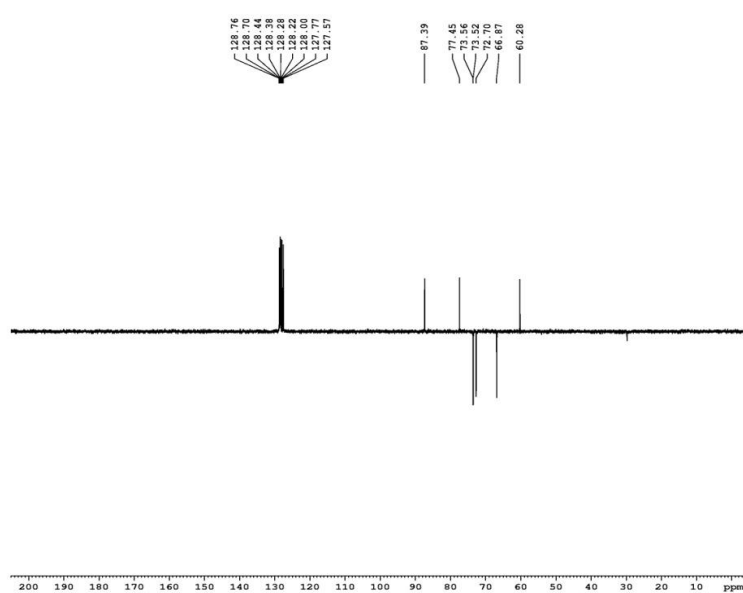
Expanded ^{13}C Spectra of 15a



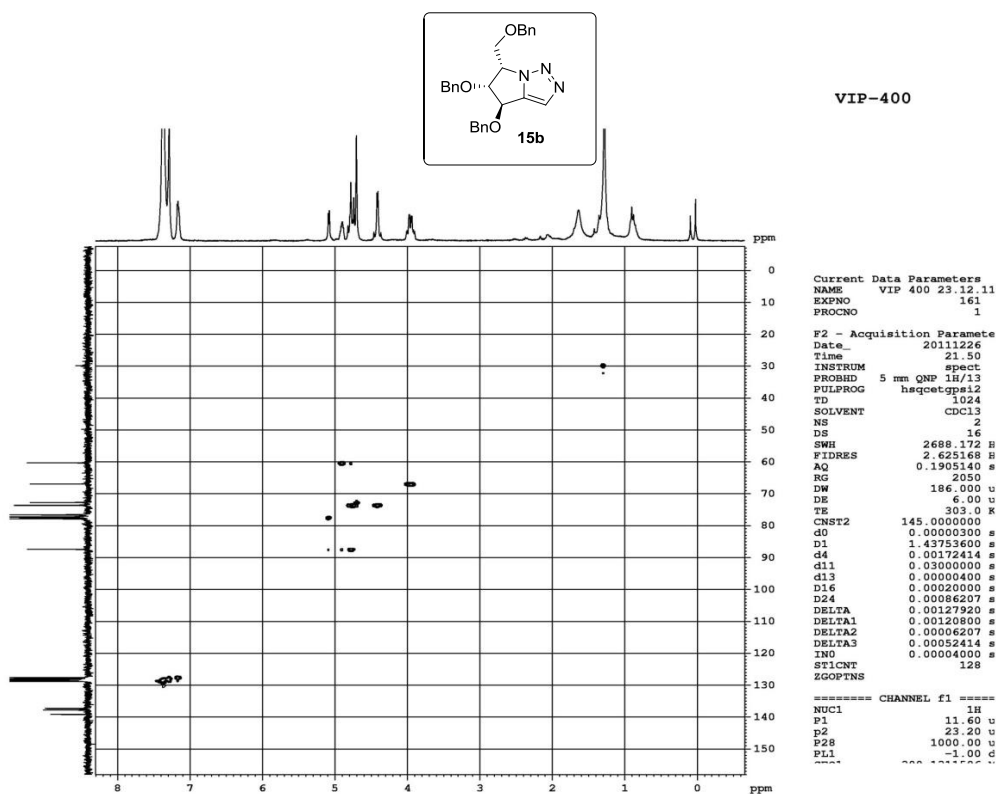
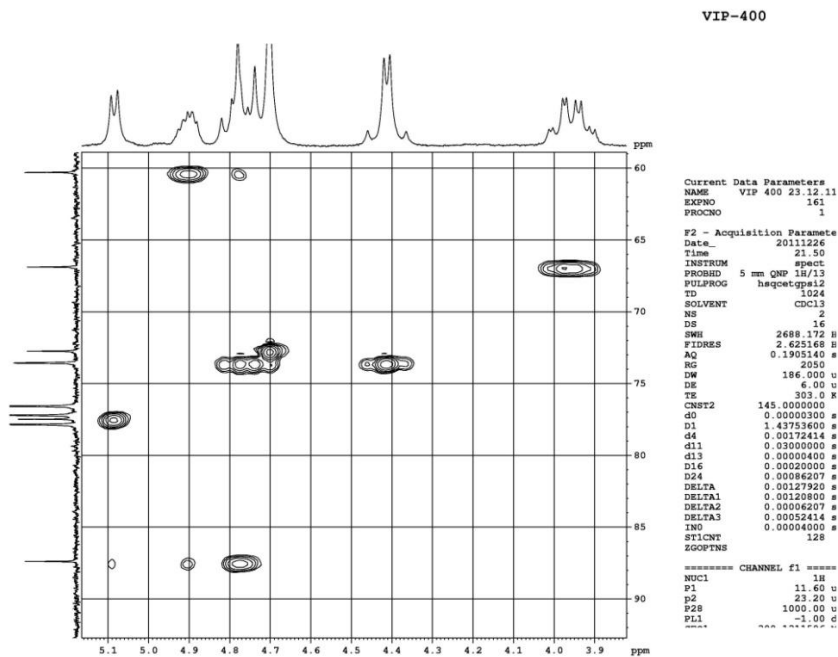
^{13}C Spectra of 15a



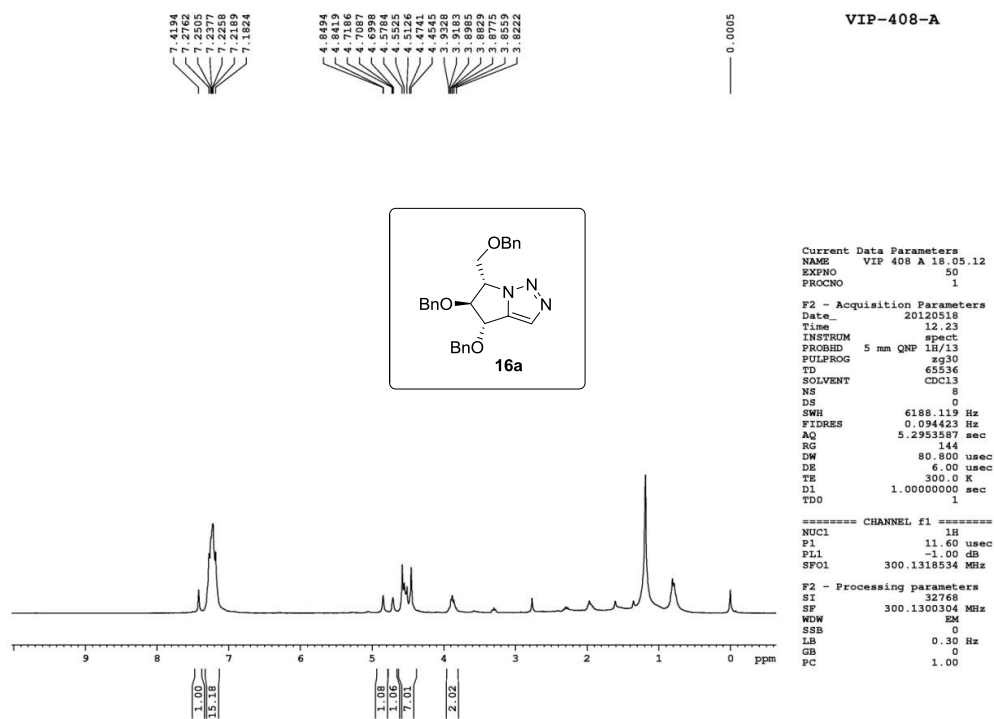
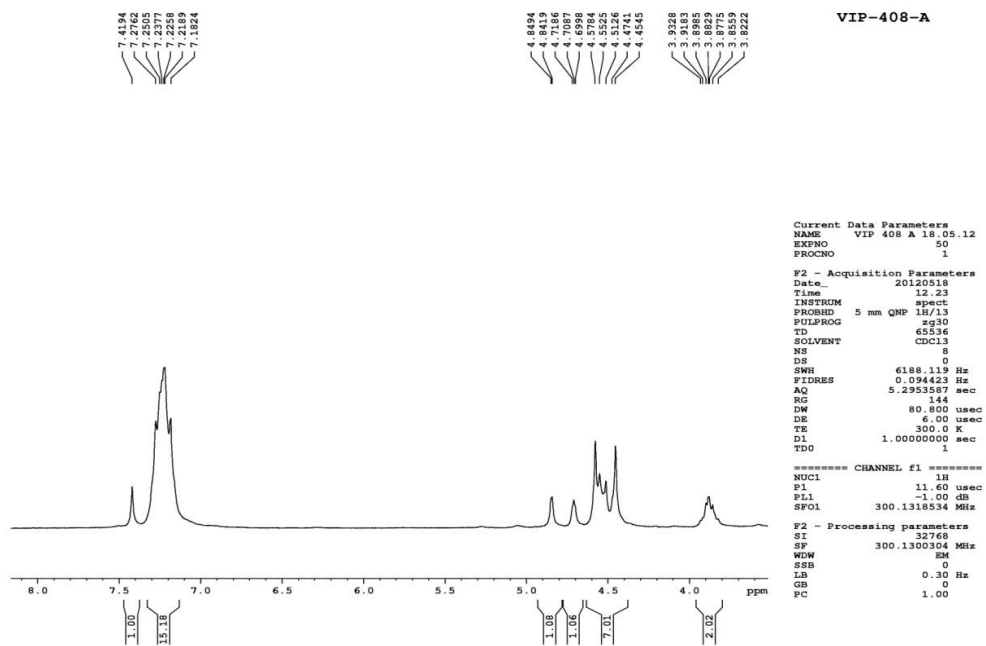
^1H NMR Spectrum of 15b



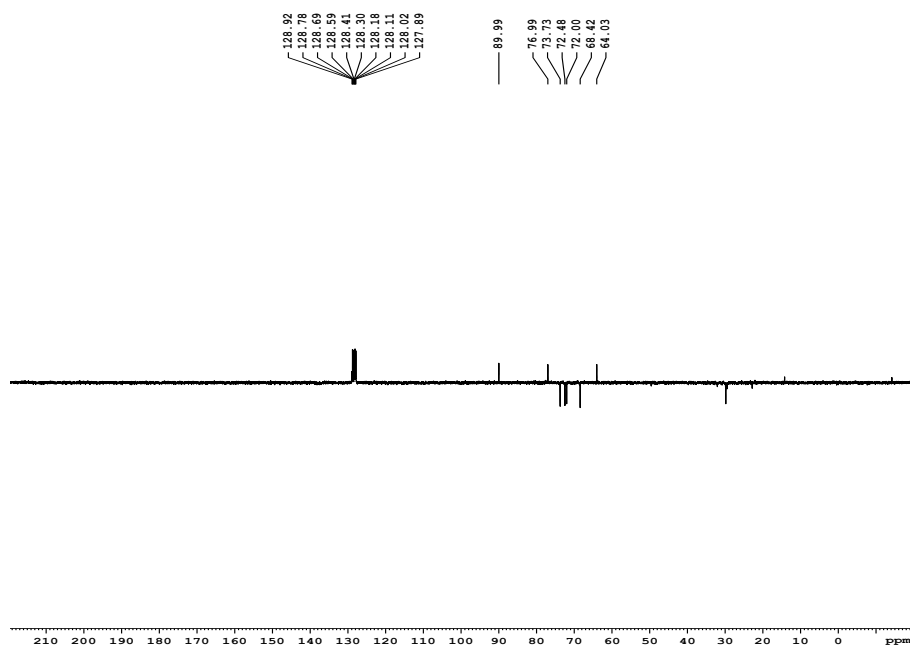
¹³C NMR Spectrum of 15b



HSQC Spectra of 15b



¹H NMR Spectrum of 16a



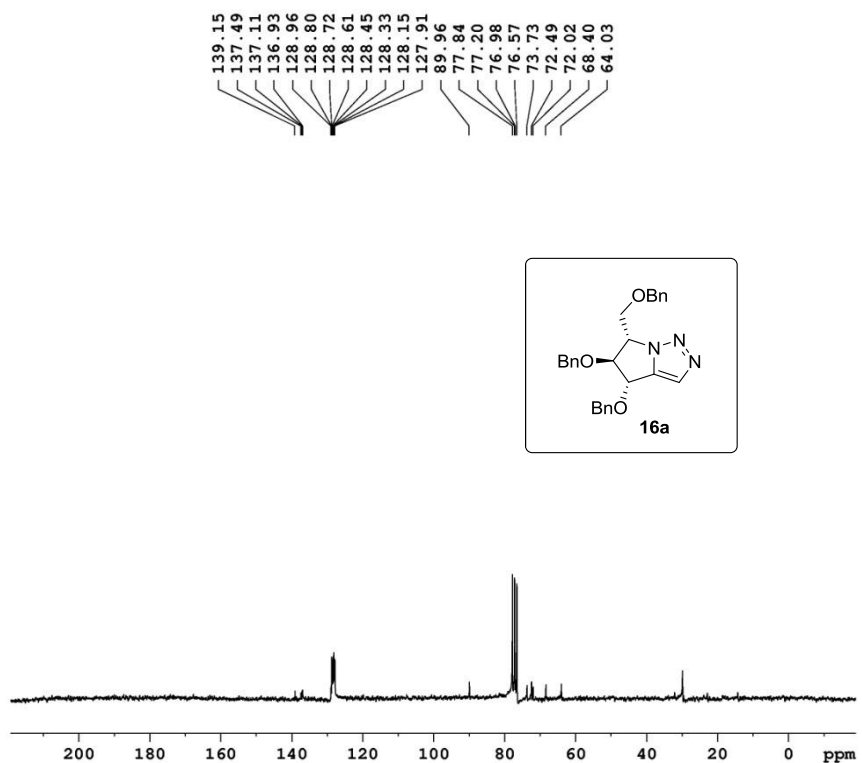
VIP-408-A

Current Data Parameters
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PROCNO 1
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Time 18.29
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PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 2050
DW 27.733 us
DE 6.00 us
TE 303.0 K
CNST2 145.0000000
d1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001108 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.70 us
PL1 17.40 us
SFO1 75.4752353 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 11.60 us
PL2 23.20 us
PCPD2 80.00 us
PL12 -1.00 dB
PL12 17.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
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SF 75.4677385 MHz
WDW EM
GB



VIP-408-A

Current Data Parameters
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EXPNO 190
PROCNO 1

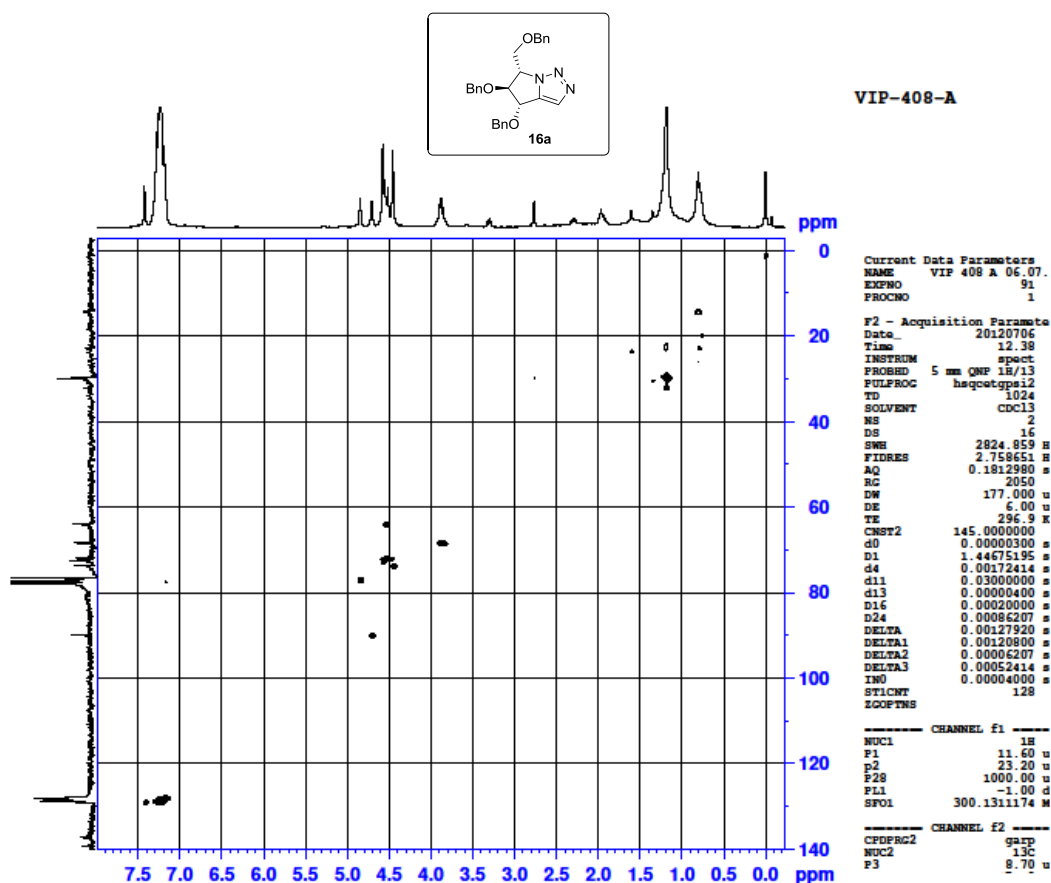
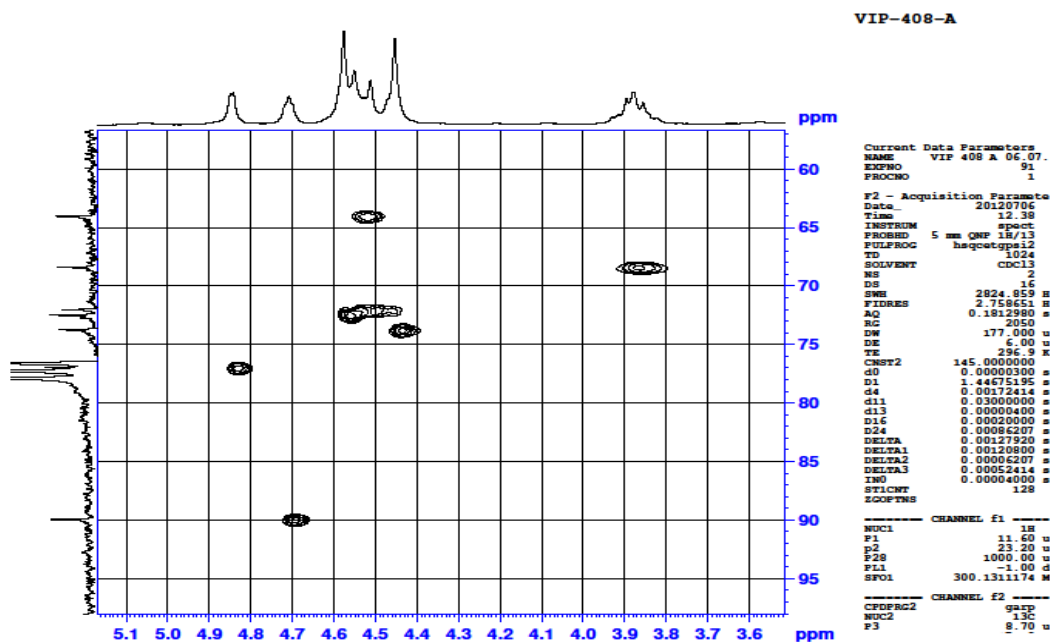
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TD 65536
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DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 64
DW 41.700 us
DE 6.00 us
TE 0.0 K
d1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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SFO1 50.3277608 MHz

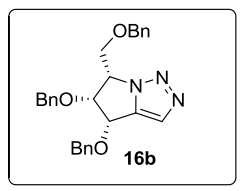
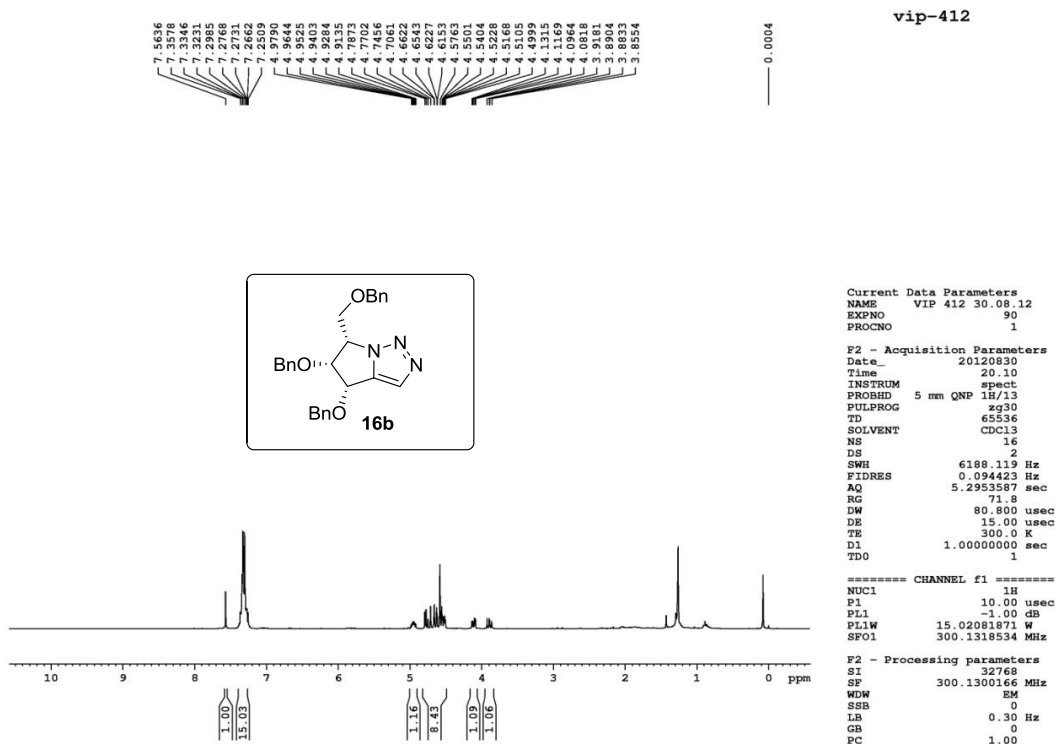
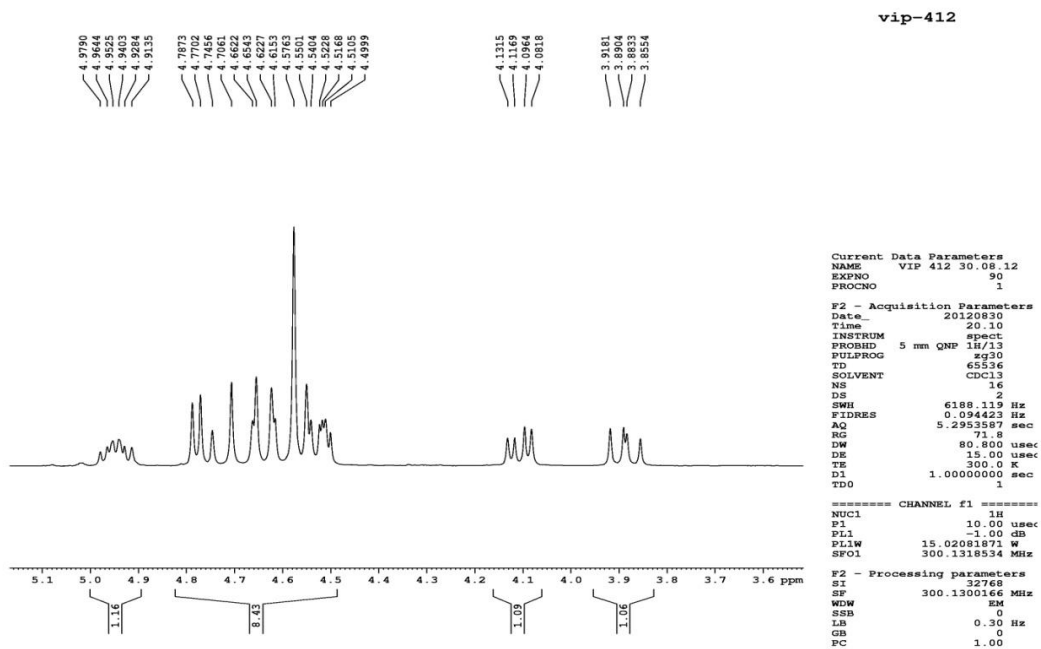
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NUC2 1H
PCPD2 100.00 us
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.1308005 MHz

F2 - Processing parameters
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WDW EM
SSB 0
LB 3.00 Hz
GB 0

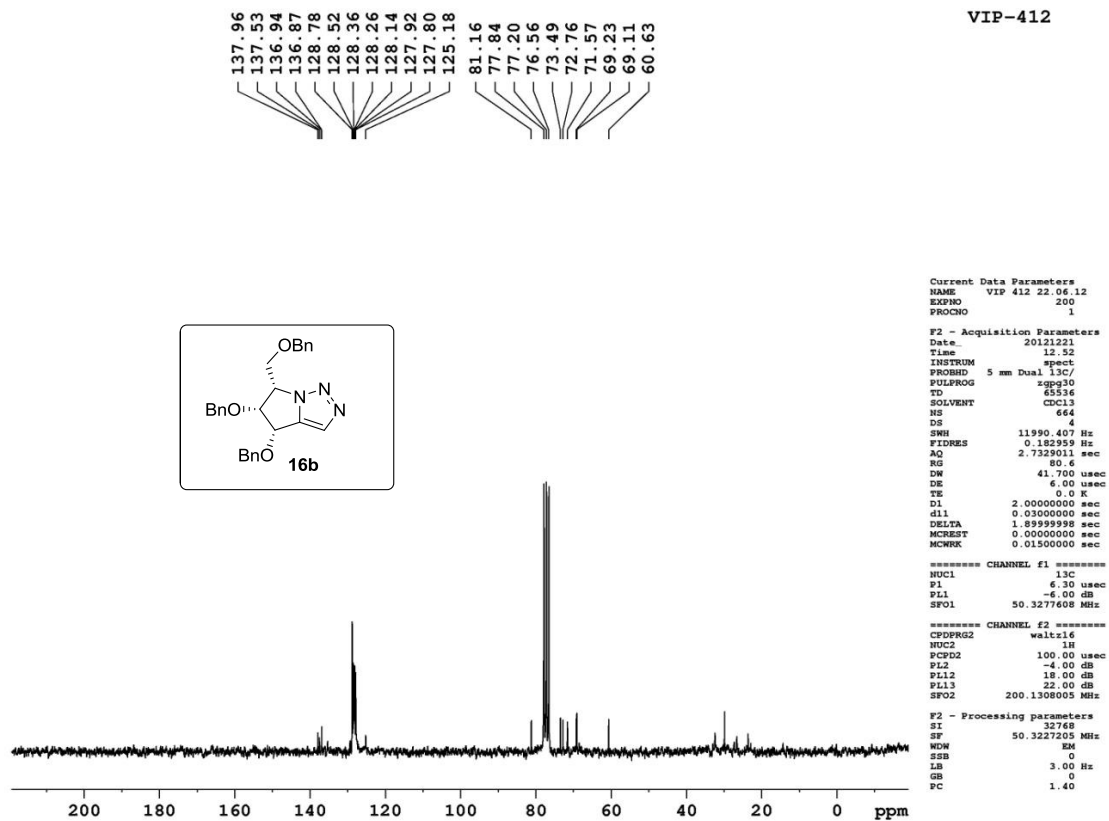
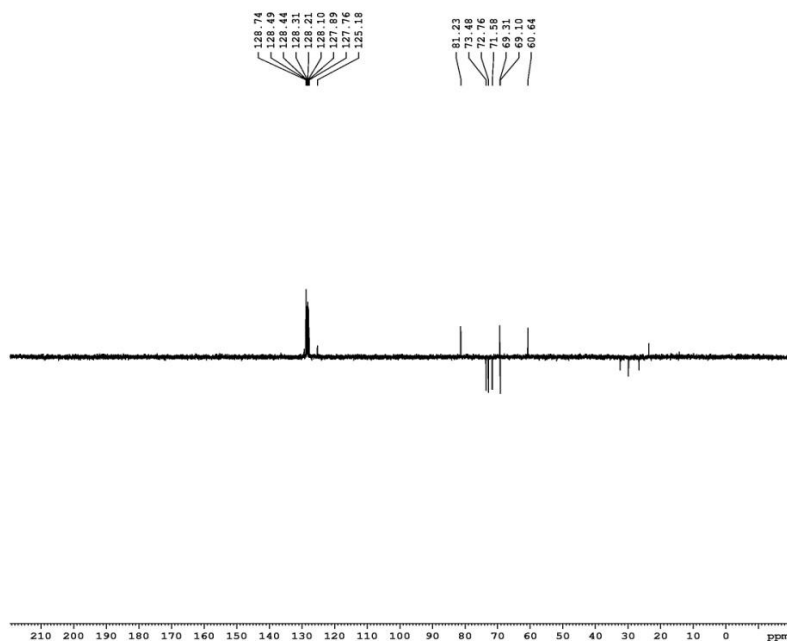
¹³C NMR Spectrum of 16 a



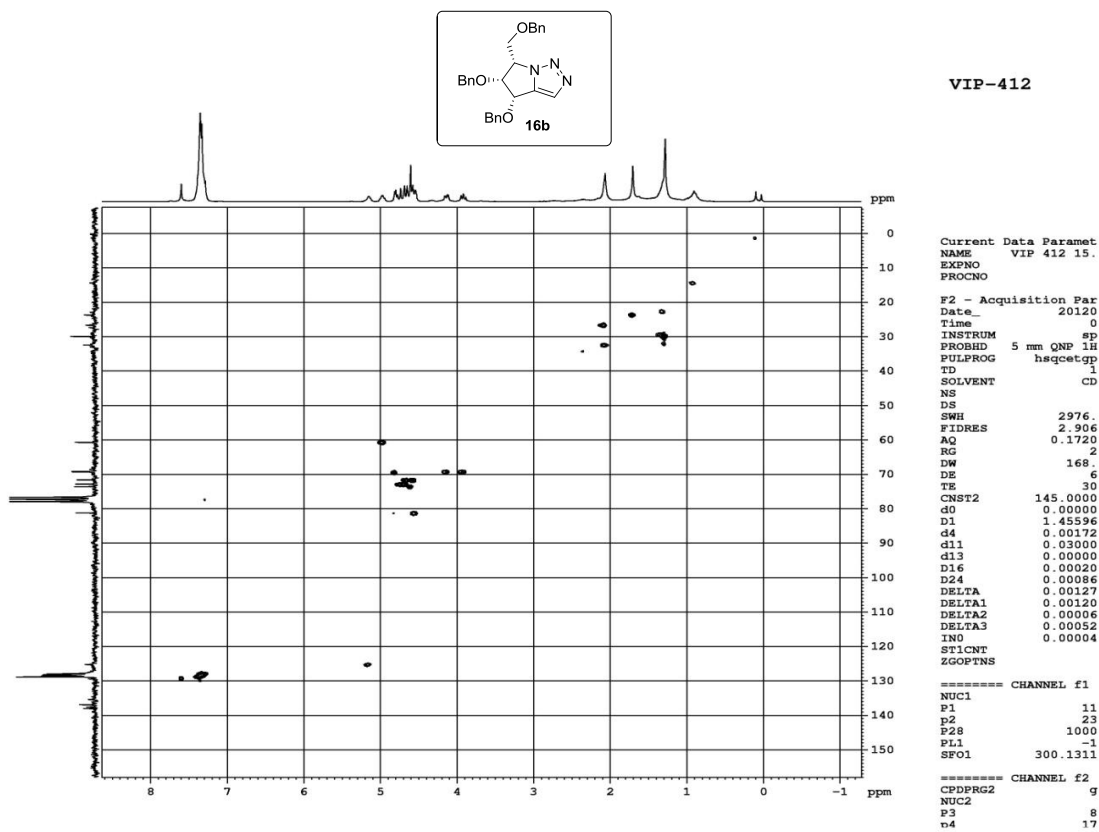
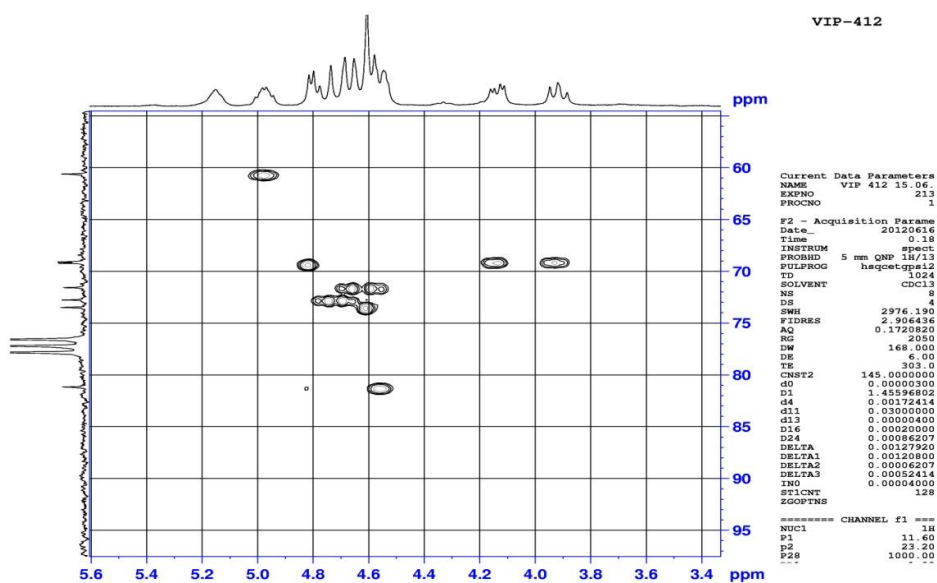
HSQC Spectra of 16a



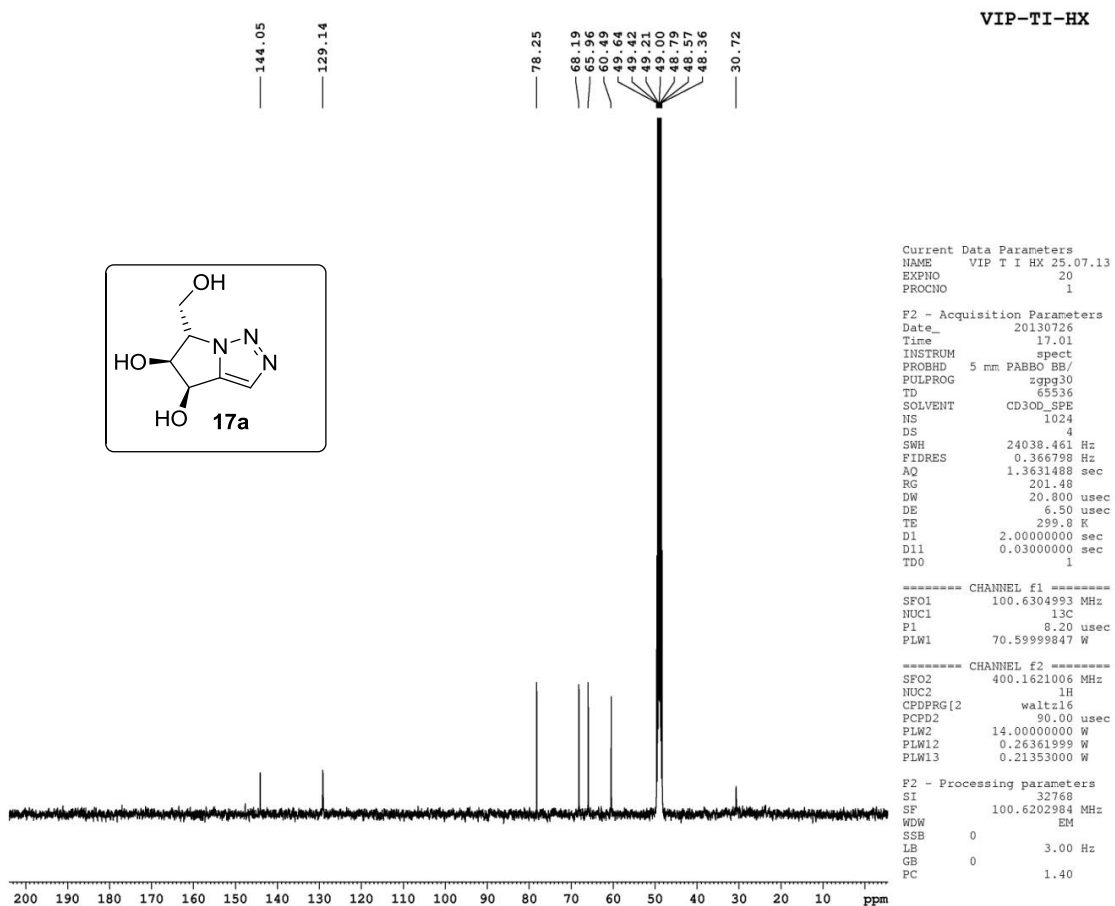
¹H NMR Spectrum of 16b



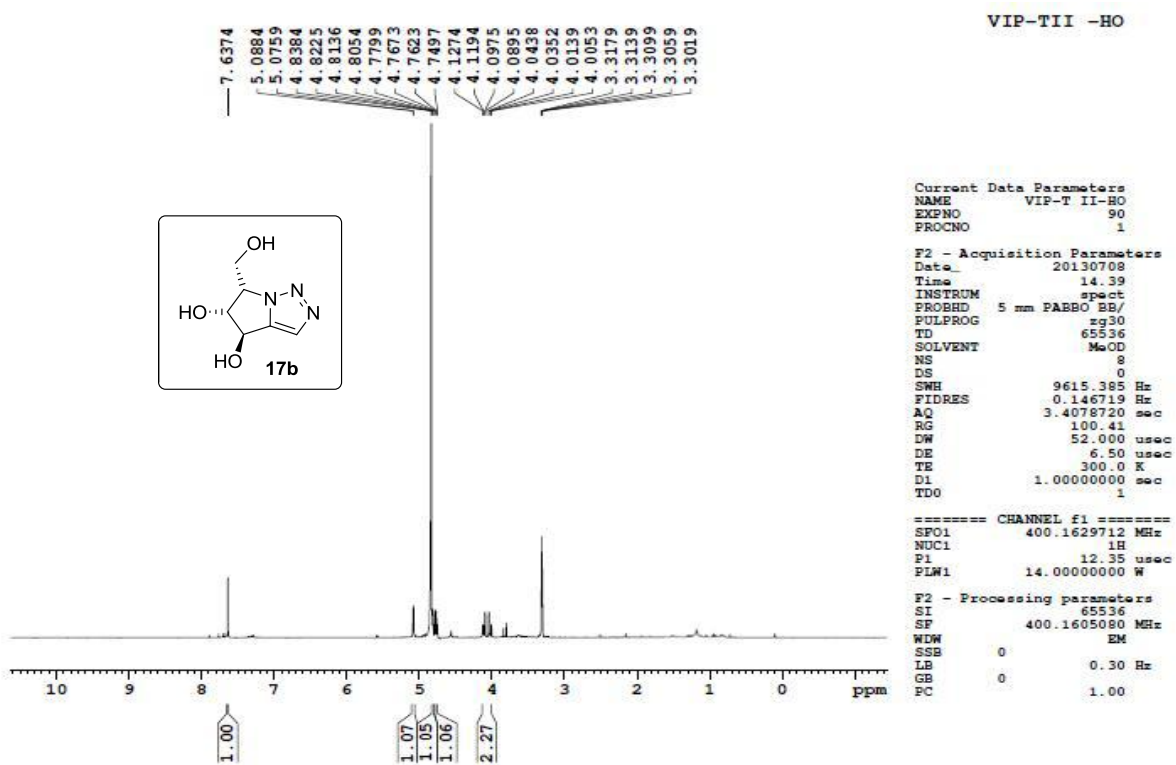
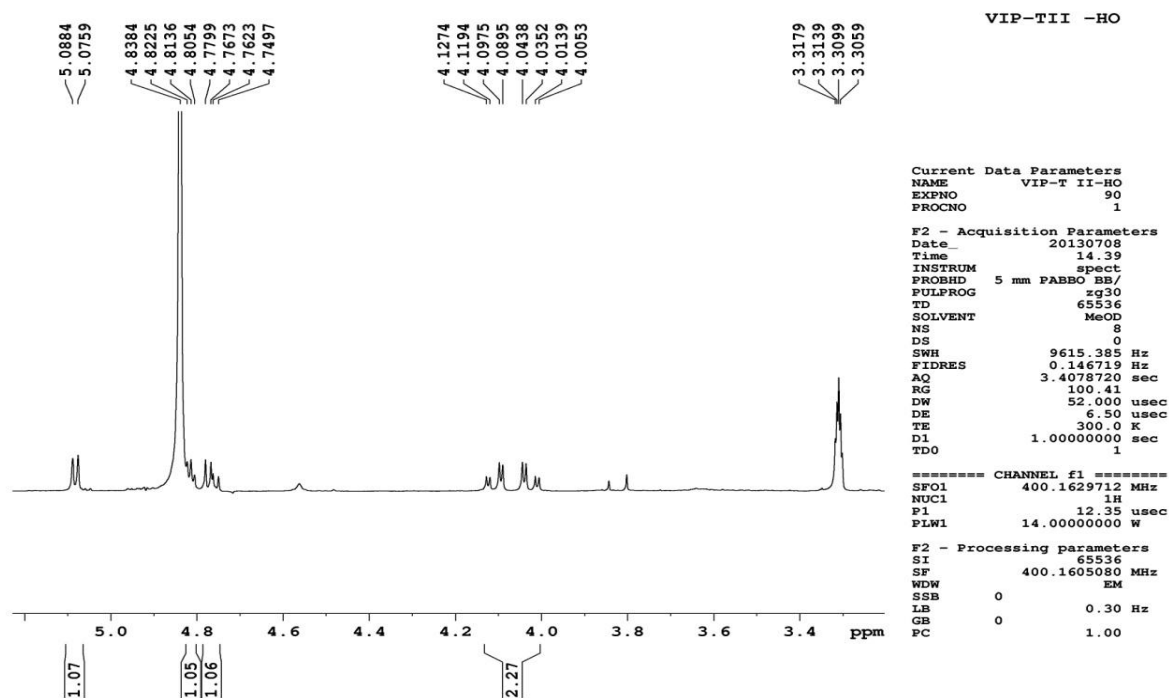
^{13}C NMR Spectrum of 16b



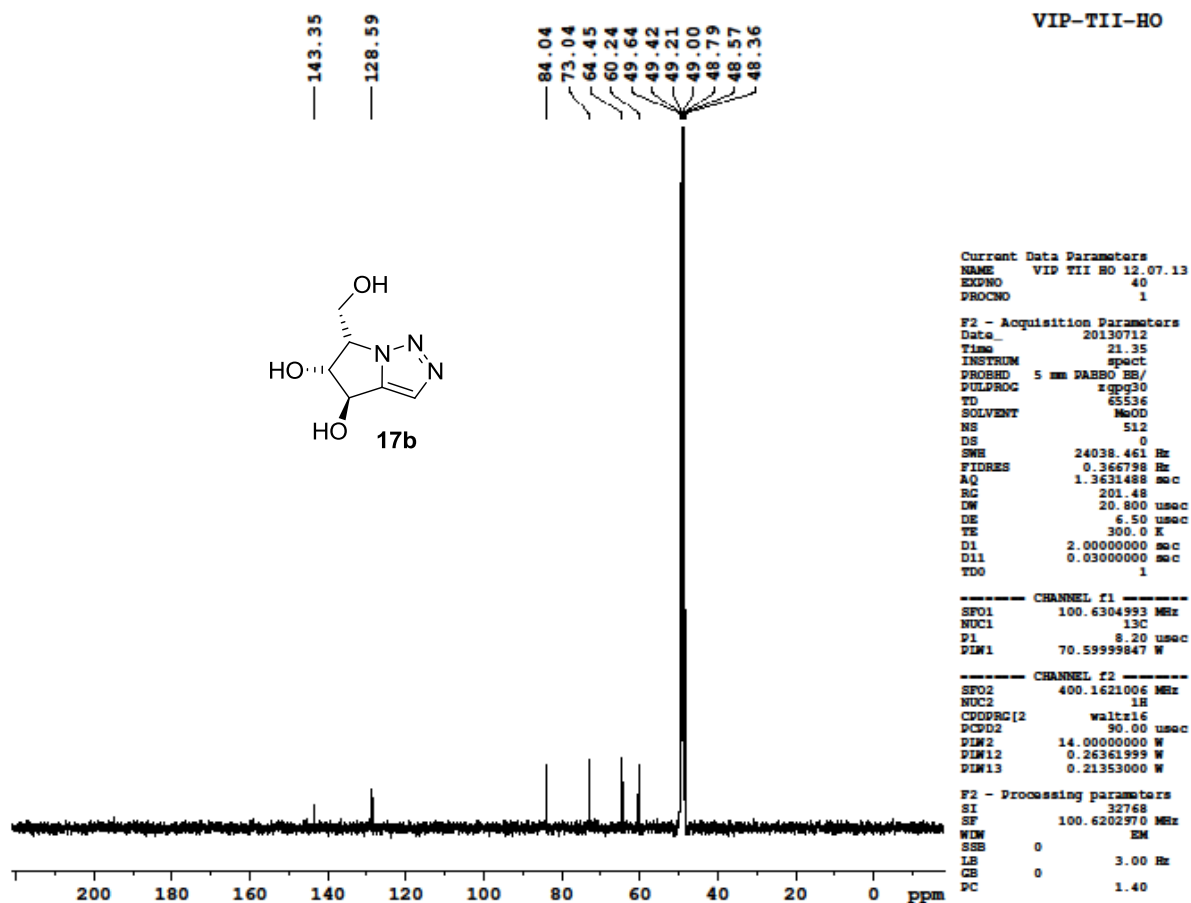
HSQC Spectra of 16b



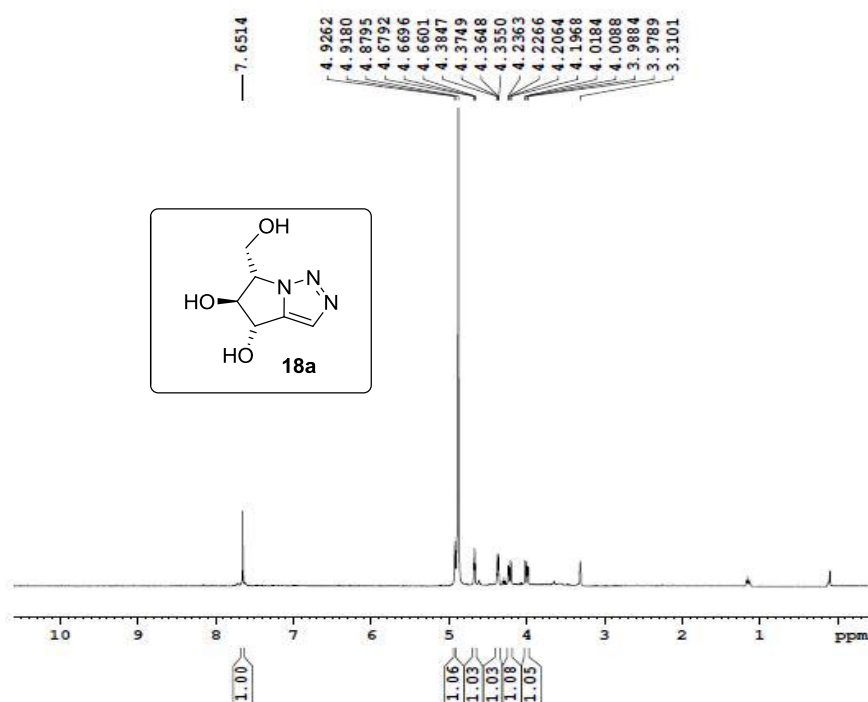
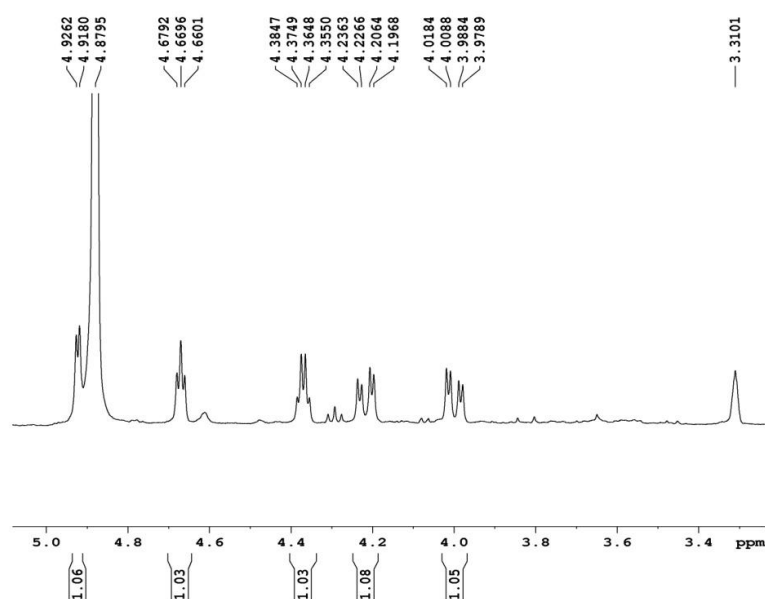
¹³ C NMR Spectrum of 17a



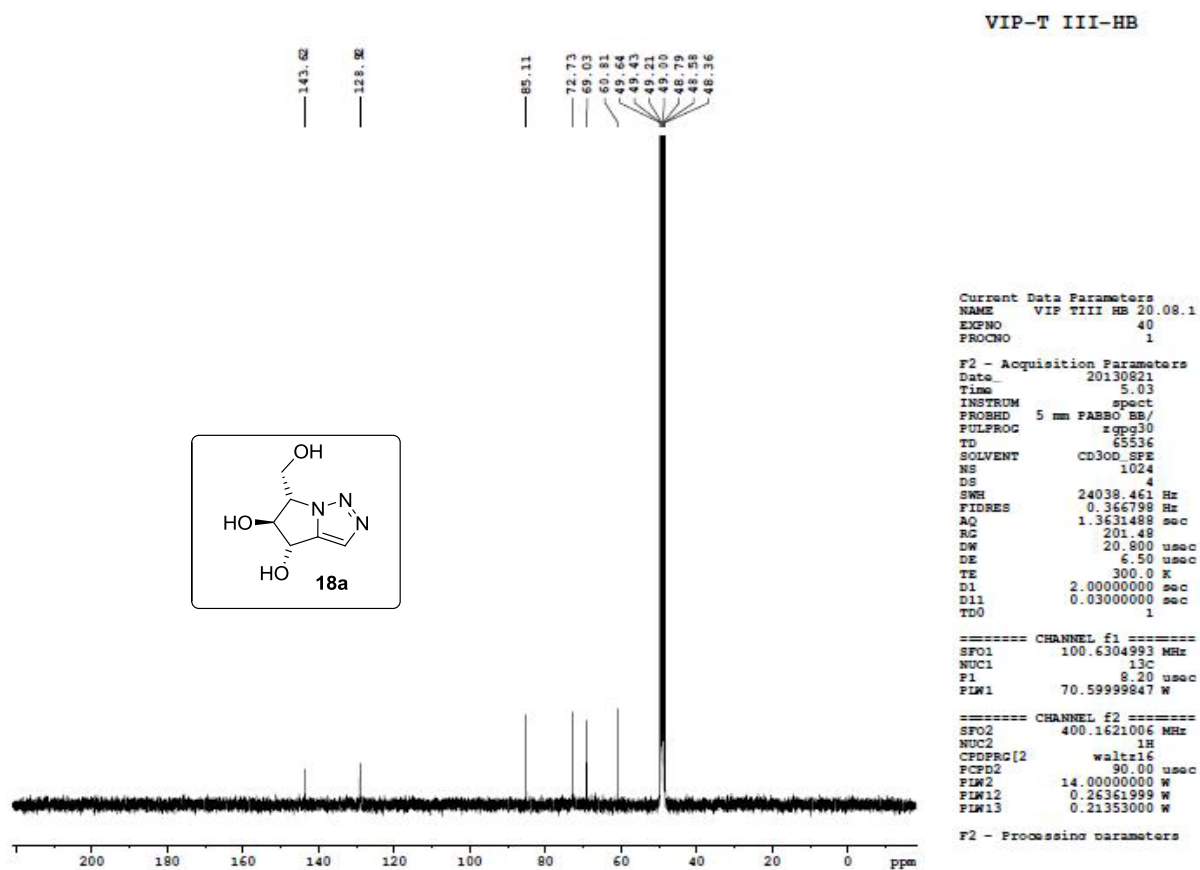
¹H NMR Spectrum of 17b



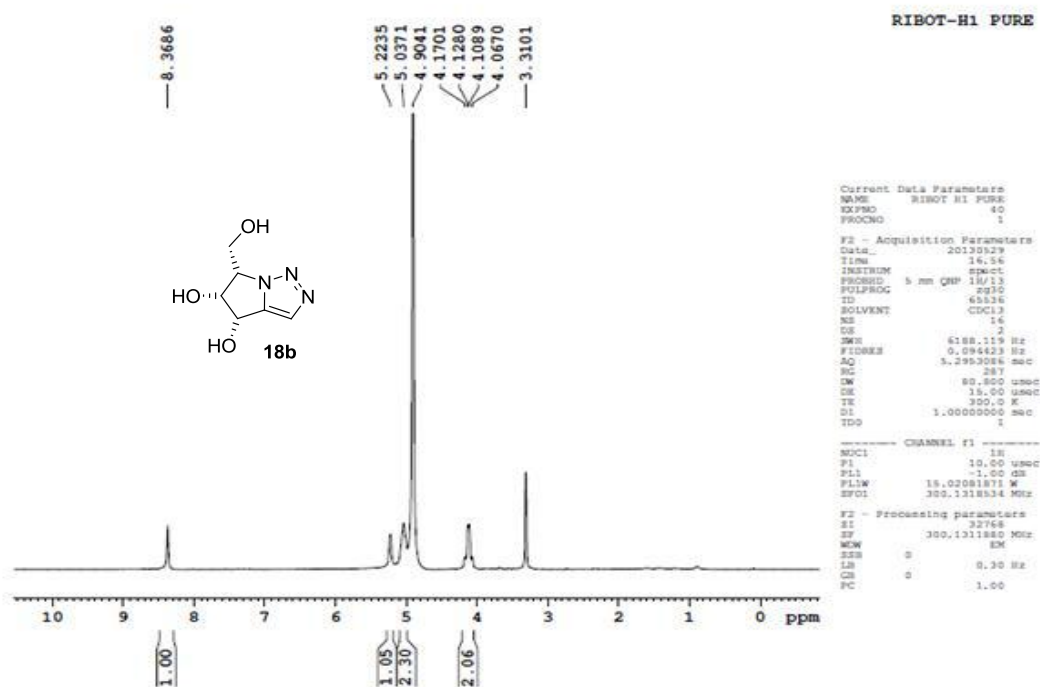
^{13}C NMR Spectrum of 17b



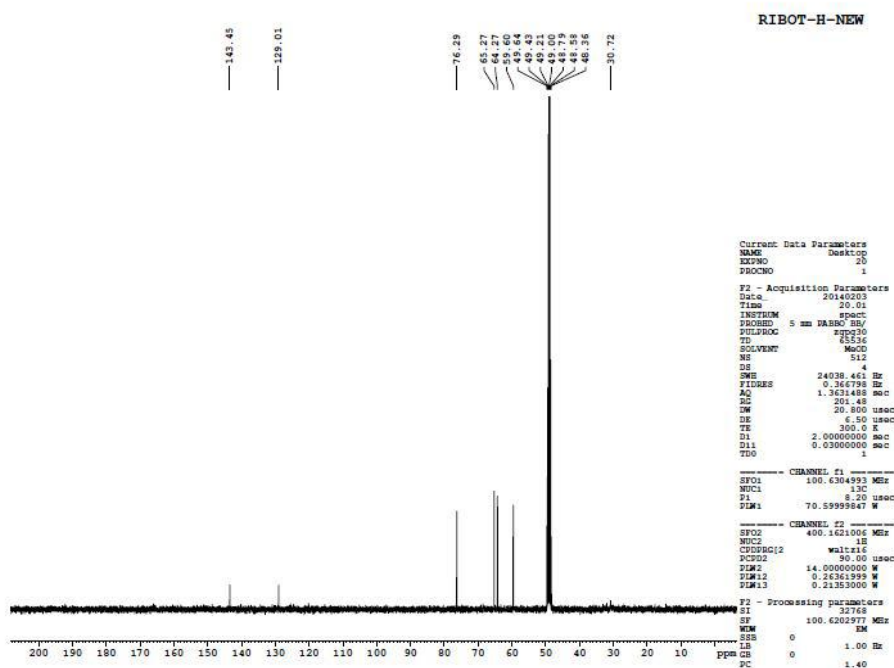
¹H NMR Spectrum of 18a



¹³ C NMR Spectrum of 18a



¹H NMR Spectrum of 18b



¹³C NMR Spectrum of 18b

Glycosidase Inhibition

General Methods are mentioned in the manuscript text. Inhibition potencies of compounds were determined according to Gunter and Stefan (1986), Li *et al* (2011) by minute modifications, measuring the residual hydrolytic activities of glycosidase of the corresponding p-nitrophenyl glycosides in presences of compounds spectrophotometrically. Michaelis-Menten plot of Activity versus Substrate concentration for inhibition and K_i were determined by nonlinear regression using data to a competitive inhibition model using Graph Pad Prism (version 6.01 for Windows, Graph Pad Software, San Diego California (USA)).¹⁻³

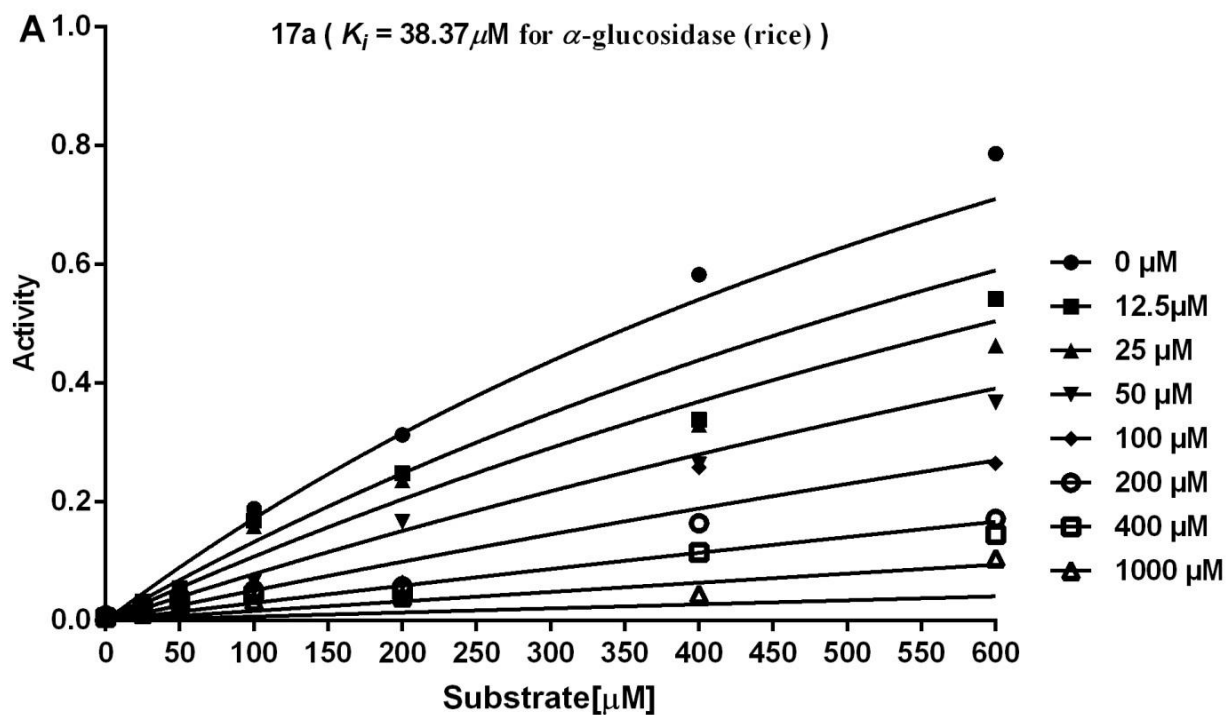
Table 1: List of Enzyme respective substrates, buffer and optimum incubation temperature

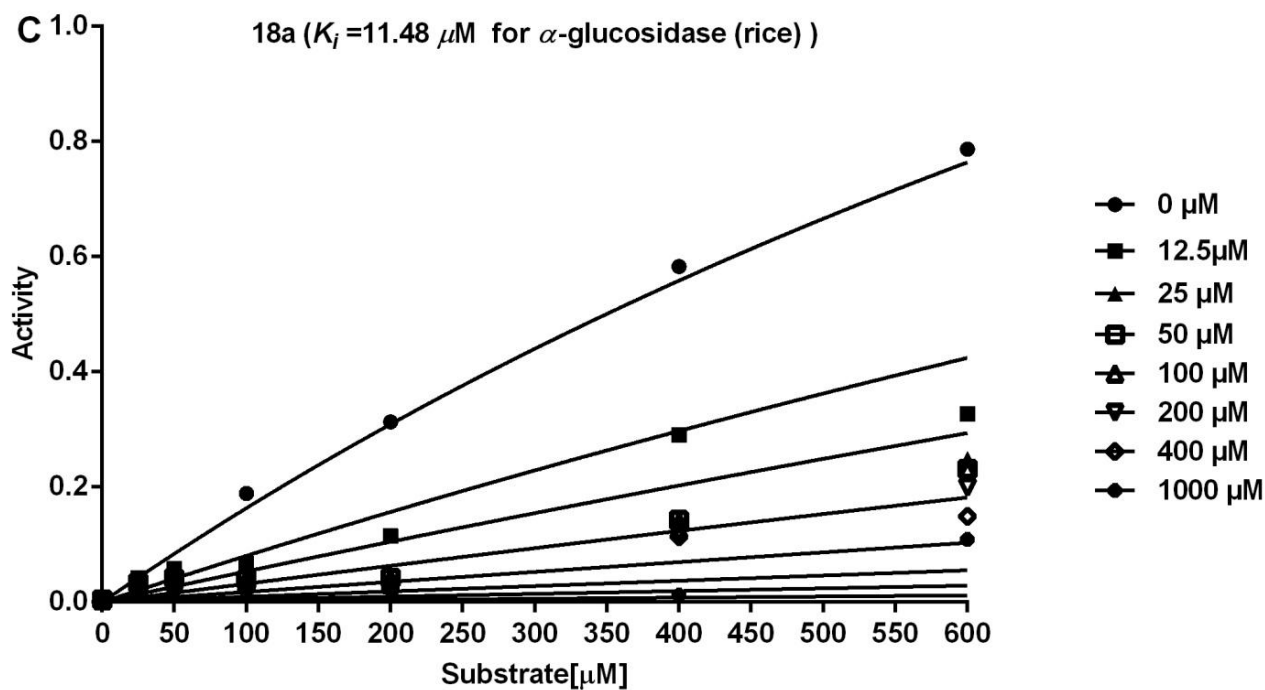
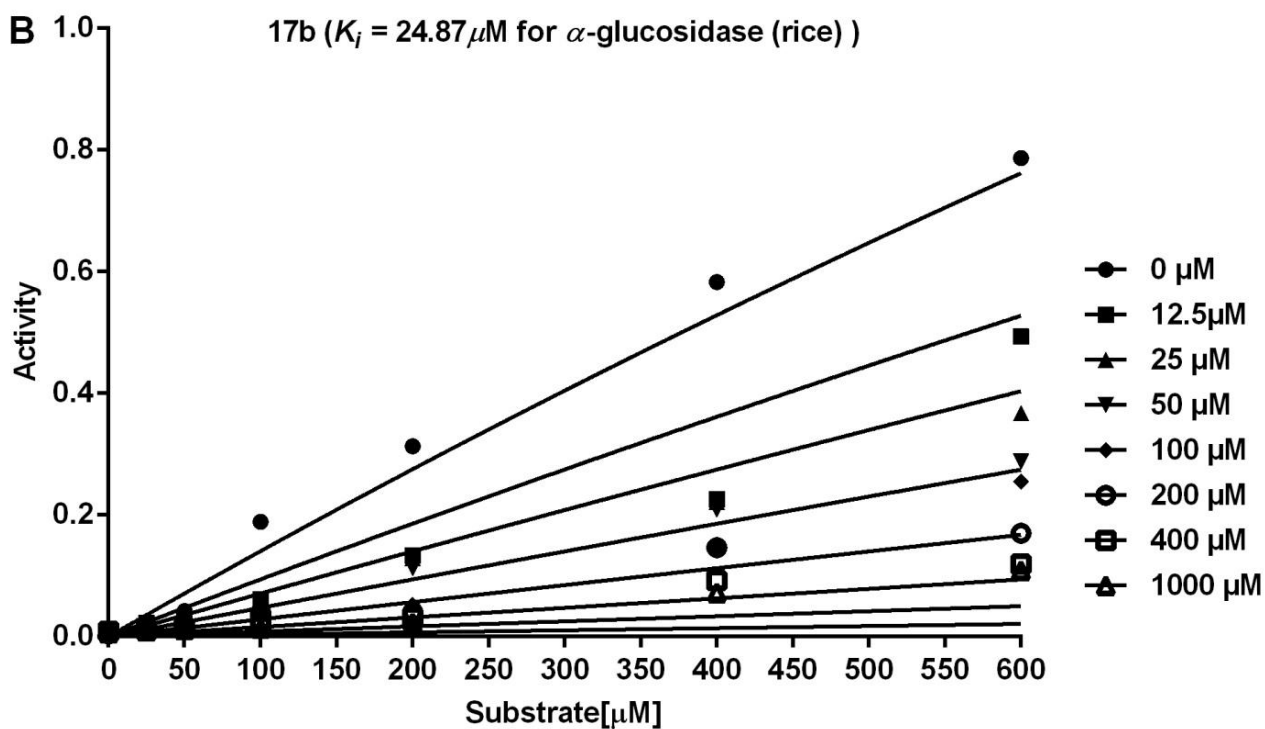
S.No	Enzyme	Substrate	Buffer	Incubation temperature.
1	α -galactosidase (green coffee bean)	<i>p</i> -nitrophenyl α -D-galactopyranoside	Citrate phosphate buffer (50 mM, pH 6.5)	25°C
2	β -galactosidase (bovine liver)	<i>p</i> -nitrophenyl β -D-galactosidase	Citrate phosphate buffer (50 mM, pH 4.5)	30°C
3	Trehalase (porcine kidney)	D-trehalose dihydrate	Sodium acetate buffer (50 mM, pH 6.5)	25 °C
4	Amyloglucosidase (<i>A. niger</i>)	Starch wheat	Citrate phosphate buffer (50 mM, pH 5.5)	45 °C
5	α -mannosidase (jack bean)	<i>p</i> -nitrophenyl α -D-mannopyranoside	Acetate buffer (50 mM, pH 4.5)	25 °C
6	β -glucosidase (almond)	<i>p</i> -nitrophenyl β -D-glucopyranoside	Citrate phosphate buffer (50 mM, pH 5.5)	37 °C
7	α -glucosidase (yeast)	<i>p</i> -nitrophenyl α -D-glucopyranoside	Citrate phosphate buffer (50 mM, pH 6.8)	37 °C
8	Glucosidase (<i>A. niger</i>)	<i>p</i> -nitrophenyl α -D-glucopyranoside	Citrate phosphate buffer (50 mM, pH 6.8)	37 °C
9	α -glucosidase (rice)	<i>p</i> -nitrophenyl α -D-glucopyranoside	Citrate phosphate buffer (50 mM, pH 6.8)	37 °C
10	β -N-acetyl glucosaminidase (jack bean)	4-nitrophenyl <i>N</i> -Acetyl β -D-glucosaminide	Citrate buffer (50 mM, pH 4)	37°C
11	α -L-fucosidase (bovine kidney)	4-nitrophenyl α -L-fucopyranoside	Citrate phosphate buffer (50 mM, pH 5)	30 °C

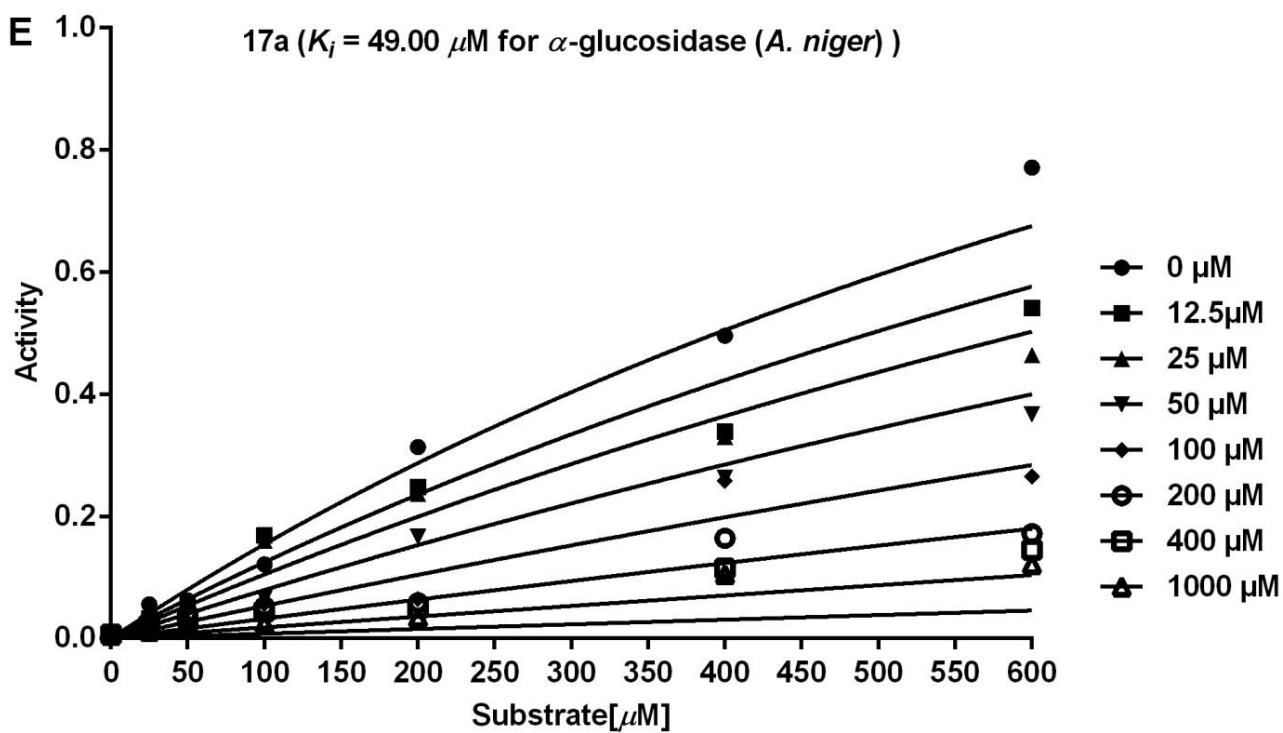
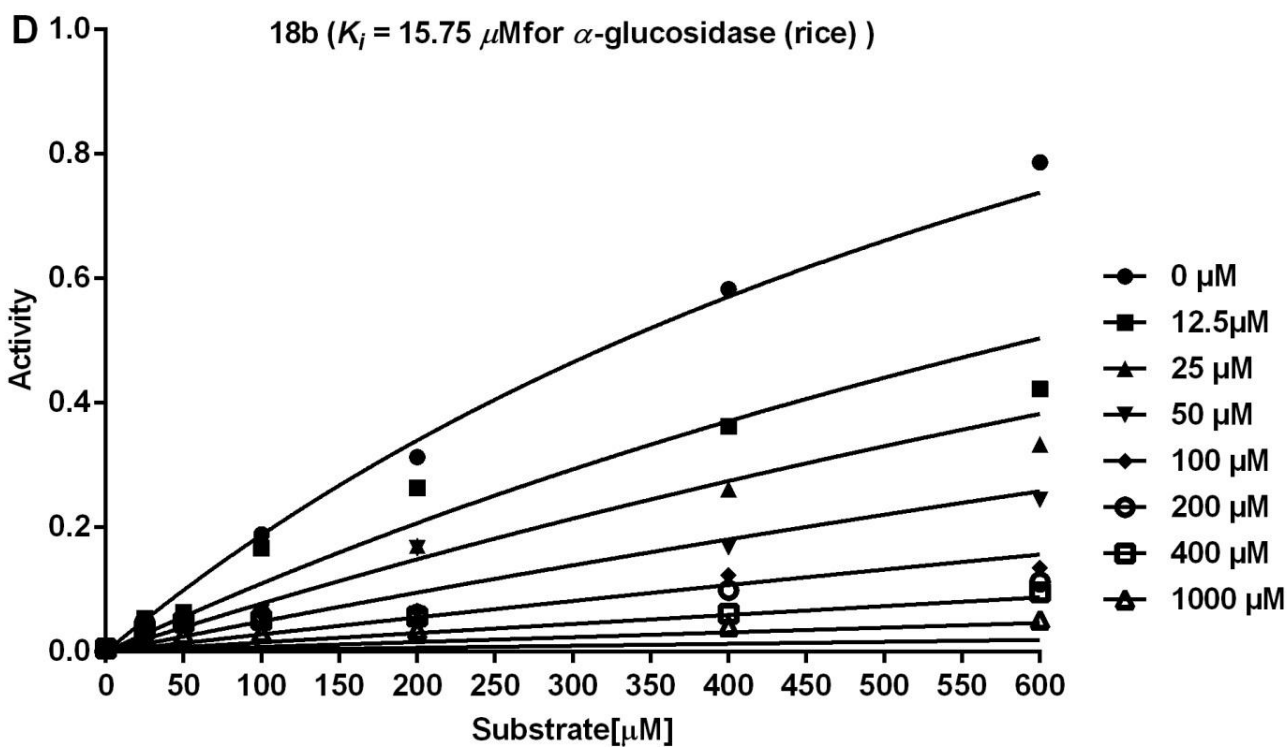
Table 2: % of inhibition of 11 glycosidase by all 4 compounds (17a, 17b, 18a and 18b) at 400 μ M and 1000 μ M

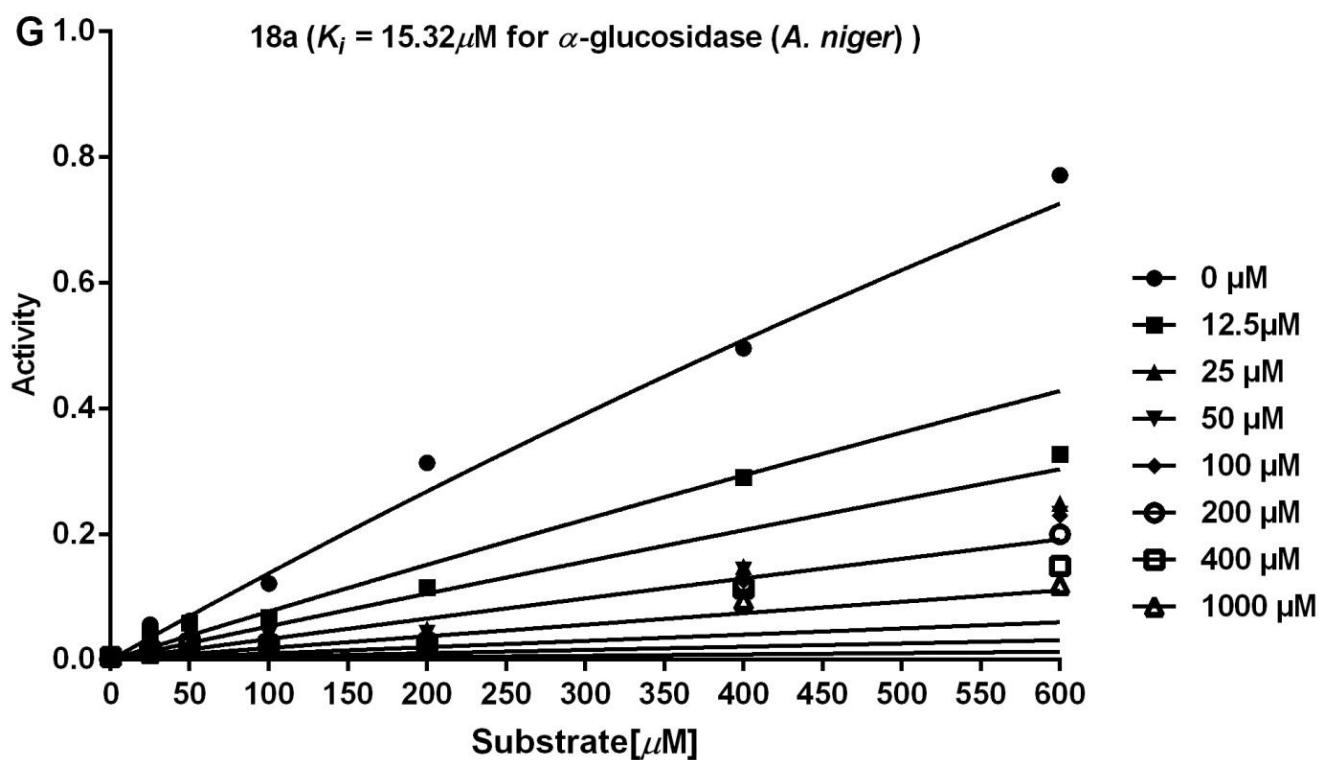
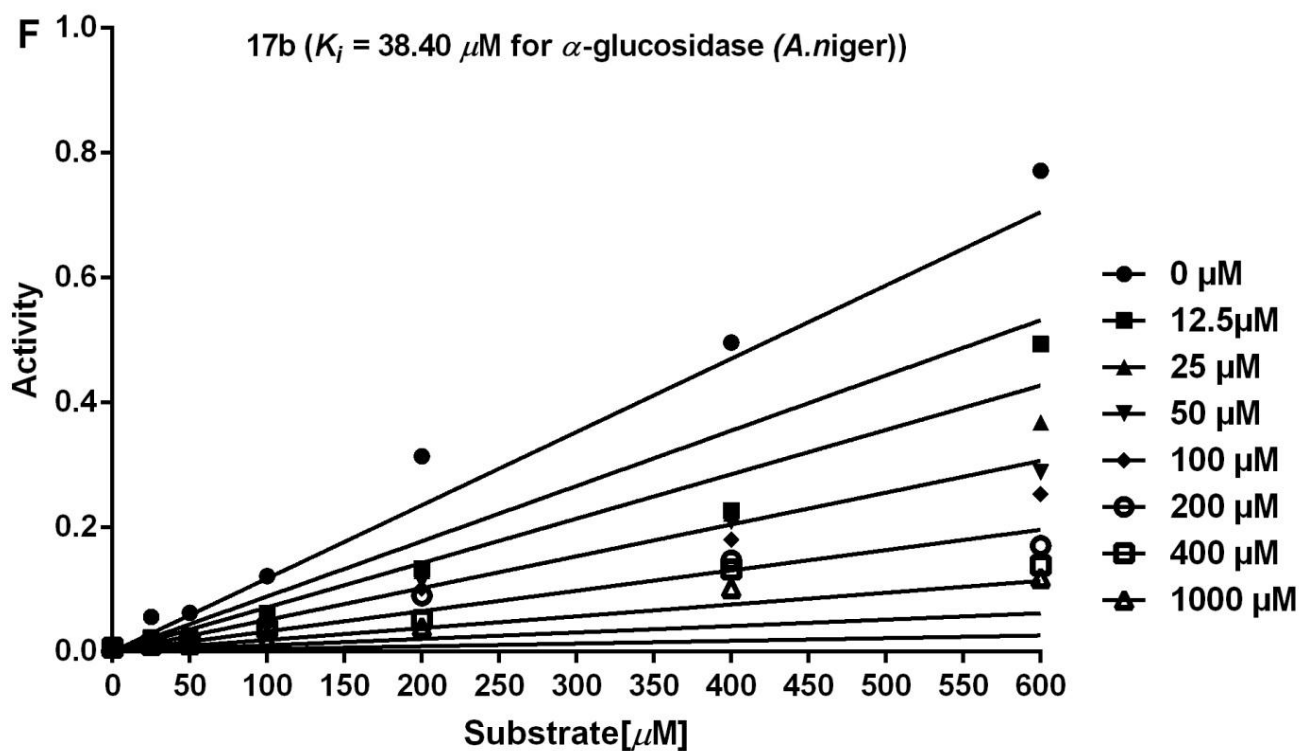
	(% of inhibition at 400 μ M)				(% of inhibition at 1000 μ M)			
ENZYME	17a	17b	18a	18b	17a	17b	18a	18b
α -galactosidase (green coffee bean)	NI (2.78)	NI (1.7)	NI (0.66)	NI (4.44)	NI (6.55)	NI (6.5)	NI (4.76)	NI (5.88)
β -galactosidase (bovine liver)	NI (6.58)	NI (6.16)	NI (6.66)	NI (1.43)	NI (6.75)	NI (8.05)	NI (5.56)	NI (3.49)
α -glucosidase (yeast)	NI (1.31)	NI (1.18)	NI (0.64)	NI (0.8)	NI (3.26)	NI (1.66)	NI (0.98)	NI (0.97)
α -glucosidase (rice)	(81.53)	(84.86)	(81.04)	(87.78)	(86.62)	(86.13)	(86.12)	(93.45)
β -glucosidase (almond)	NI (3.44)	NI (3.18)	NI (3.8)	NI (1.81)	NI (1.48)	NI(13.23)	NI (2.7)	NI (2.2)
α -glucosidase (<i>A. niger</i>)	(81.16)	(81.96)	(80.66)	(81.46)	(84.26)	(84.63)	(84.85)	(85.47)
α -mannosidase (jack bean)	NI (2.07)	NI (2.72)	NI (3.51)	NI (3.01)	NI (1.40)	NI (5.95)	NI (4.38)	NI (1.4)
Trehalase (porcine kidney)	NI (0.58)	NI (0.54)	NI (0.85)	NI (1.23)	NI (5.05)	NI (4)	NI (1.14)	NI (2.39)
Amyloglucosidase (<i>A. niger</i>)	NI (0.82)	NI (1.17)	NI (0.35)	NI (2.7)	NI (3.41)	NI (2.74)	NI (3.53)	NI (3.97)
β -N- acetyl glucosaminidase (jack bean)	NI (1.57)	NI (2.53)	NI (1.32)	NI (3.83)	NI (2.09)	NI (6.76)	NI (2.14)	NI((1.04)
α -L-fucosidase (Bovine Kidney)	NI (0.55)	NI (0.05)	NI (0.04)	(86.15)	NI (0.63)	NI (0.04)	NI (0.98)	(91.78)

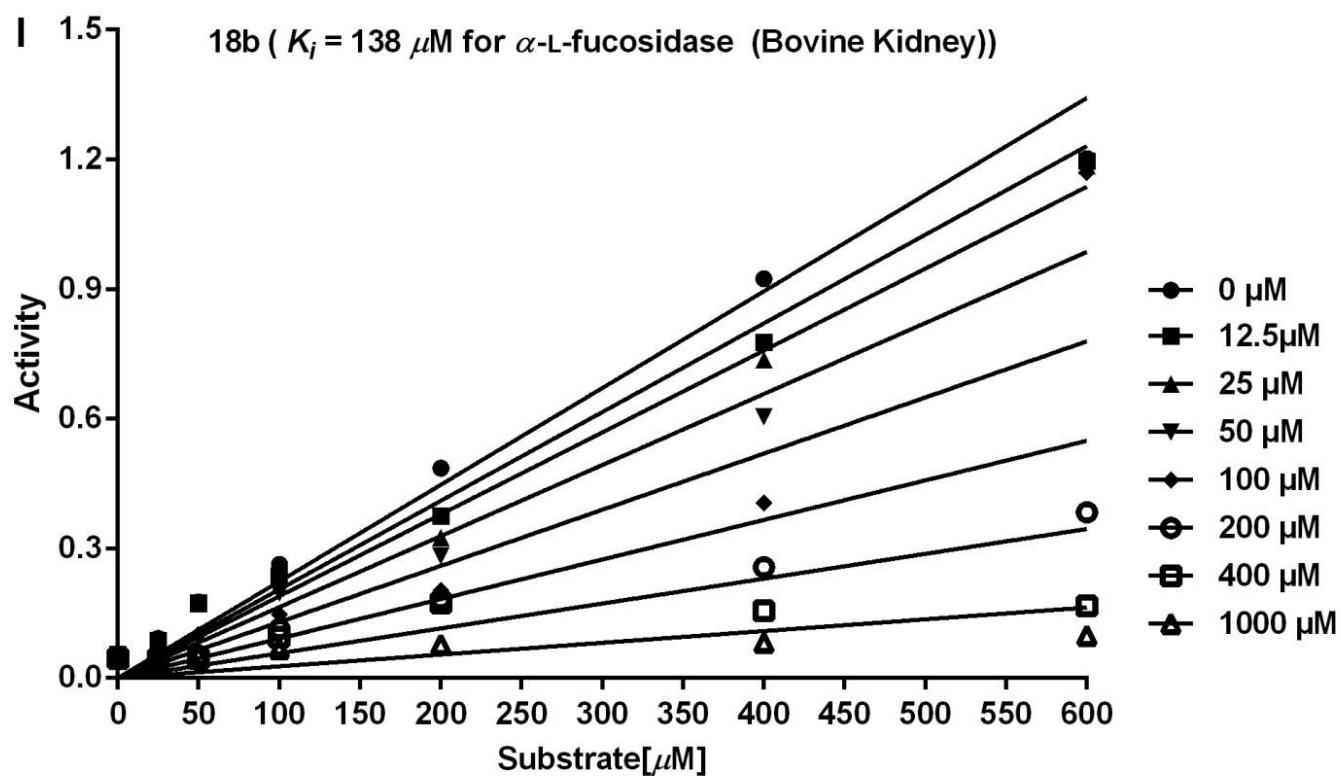
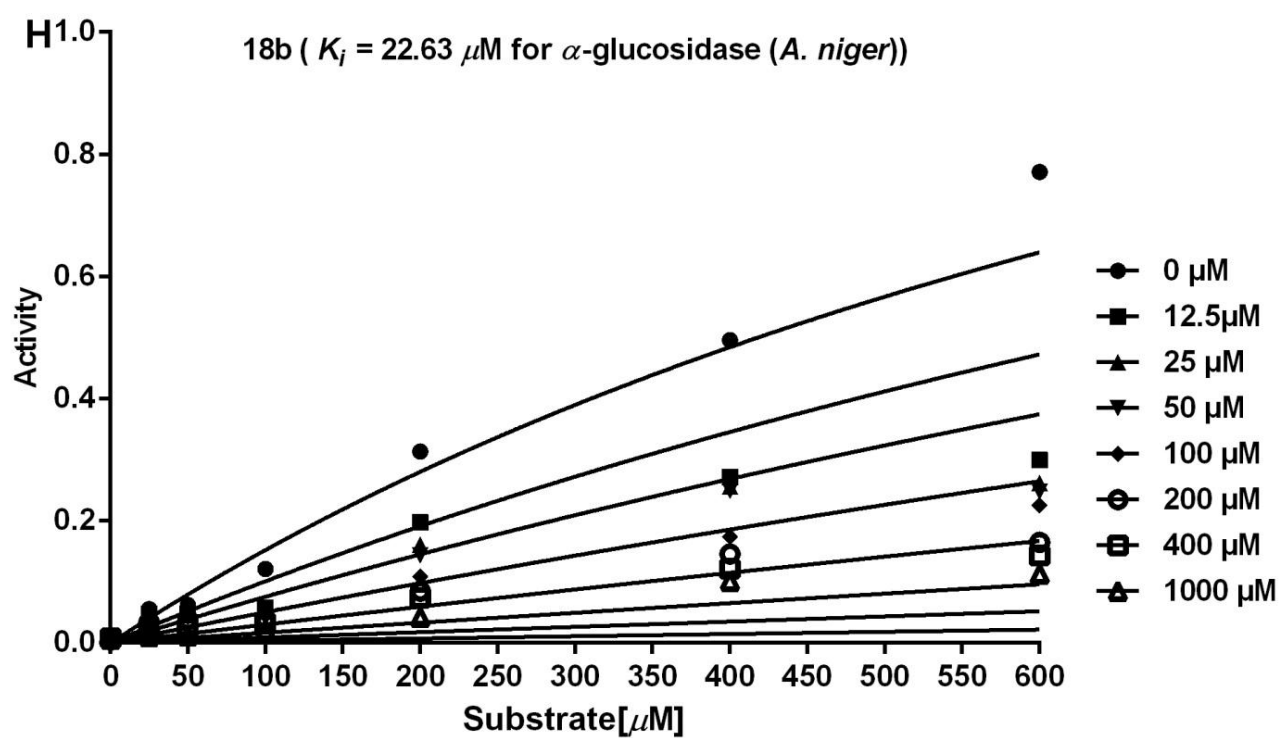
Figures A, B, C, D, E, F, G, H and I represent Michaelis-Menten plot of Activity versus Substrate concentration for inhibition of various enzymes(see methods) by compounds 17a, 17b, 18a and 18b. The K_i were determined by nonlinear regression using Graph Pad Prism (version 6.01 for Windows, Graph Pad Software, San Diego California USA) These curves show that the inhibition is competitive. Activity represented absorbance of liberated *p*-nitrophenol measured at 405 nm.











References

- (1) L. Gunter and P. Stefan, *Carbohydr. Res.* 1986, **155**, 119–129.
- (2) Y. X. Li, M. H. Huang, Y. Yamashita, A. Kato, Y. M. Jia, W. B. Wang, G. W. J. Fleet, R. J. Nash and C. Yu, *Org. Biomol. Chem.* 2011, **9**, 3405-3414.
- (3) K. Wagschal, D. Franqui-Espiet, C. C Lee, R. E. Kibblewhite-Accinelli, G. H. Robertson and D. W. S. Wong, *Enzyme Microb. Technol.* 2007, **4**, 747–753.