

Design and Synthesis of Labystegines, Hybrid Iminosugars from LAB and Calystegine, as Inhibitors of Intestinal α -Glucosidases: Binding Conformation and Interaction for ntSI

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W. J. Fleet,^{□,‡} Jun Koseki,[#] Izumi Nakagome,[‡] Shuichi Hirono *

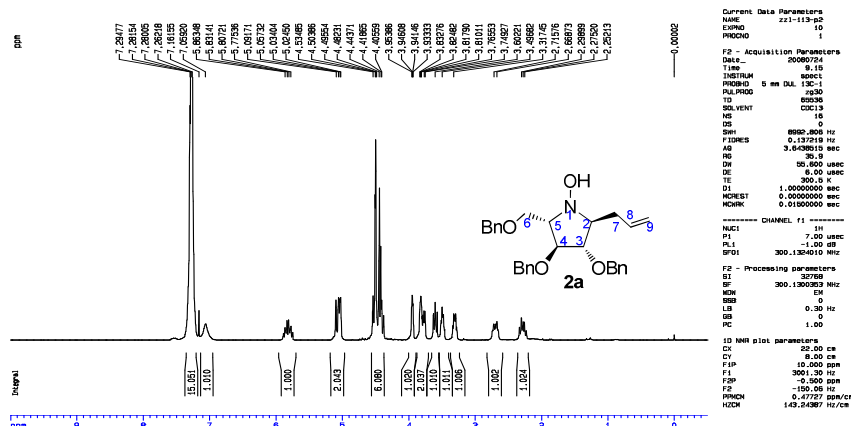
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SUPPORTING INFORMATION

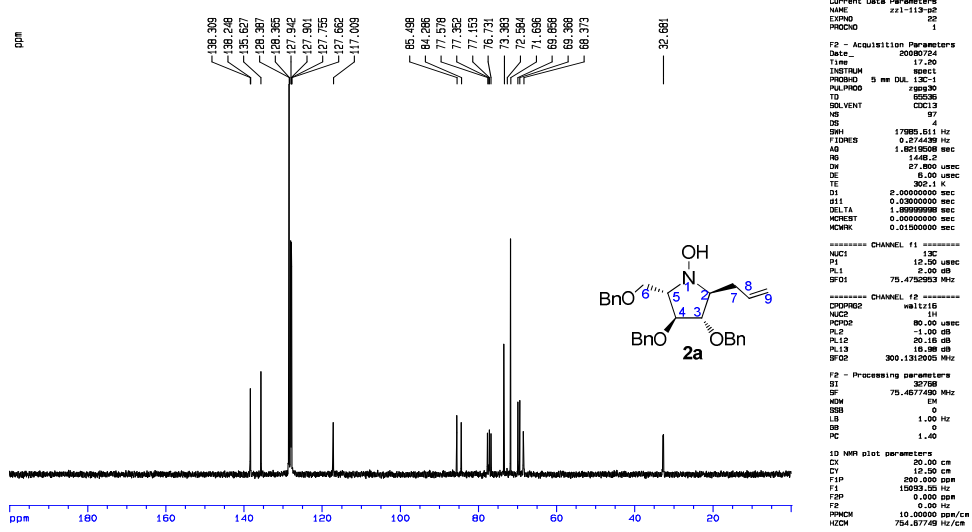
Section A: ^1H , ^{13}C and 2D-NMR Spectrum.....1

Section A: ^1H , ^{13}C and 2D-NMR Spectrum

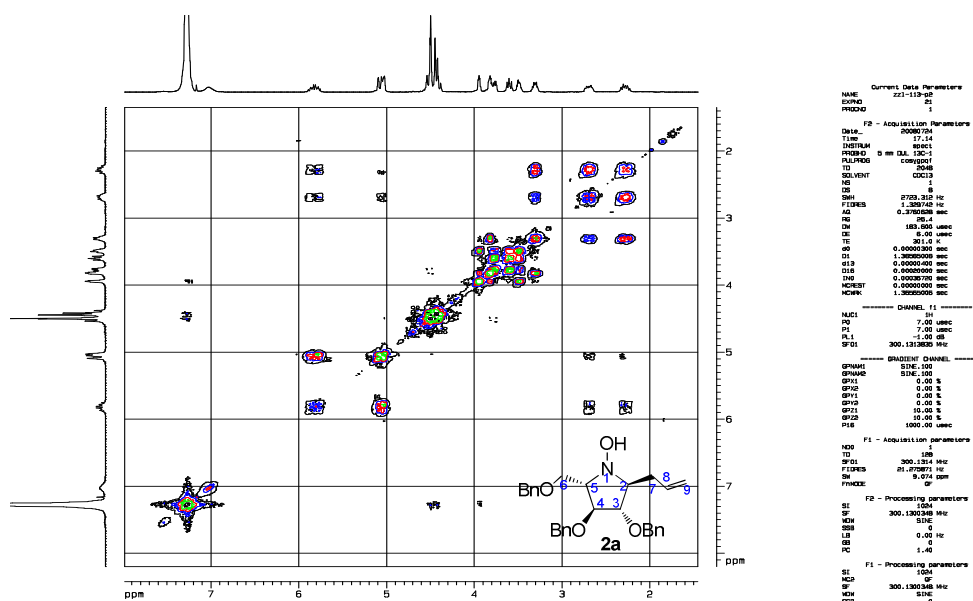
^1H NMR of compound 2a



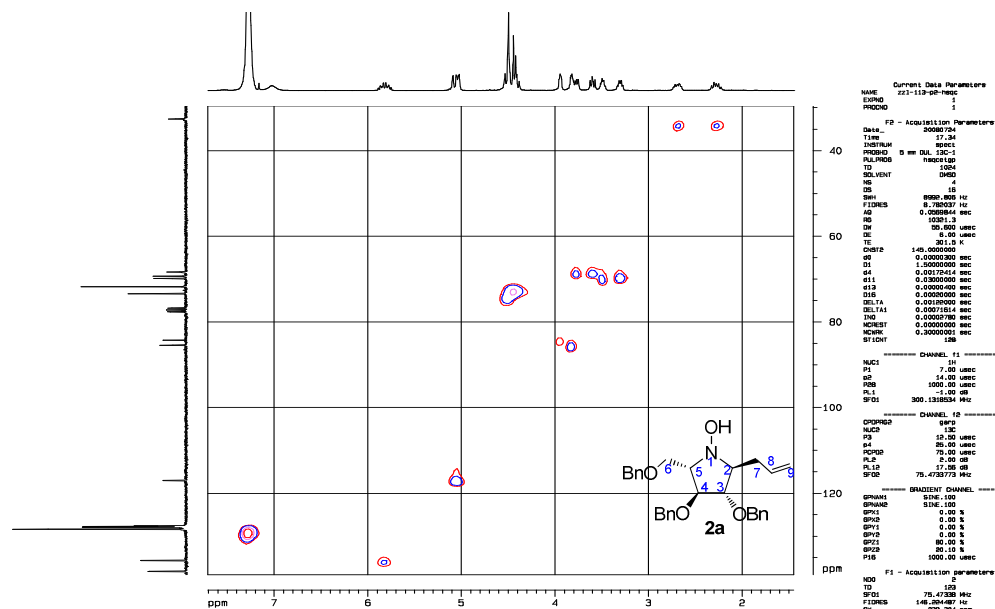
¹³C NMR of compound **2a**



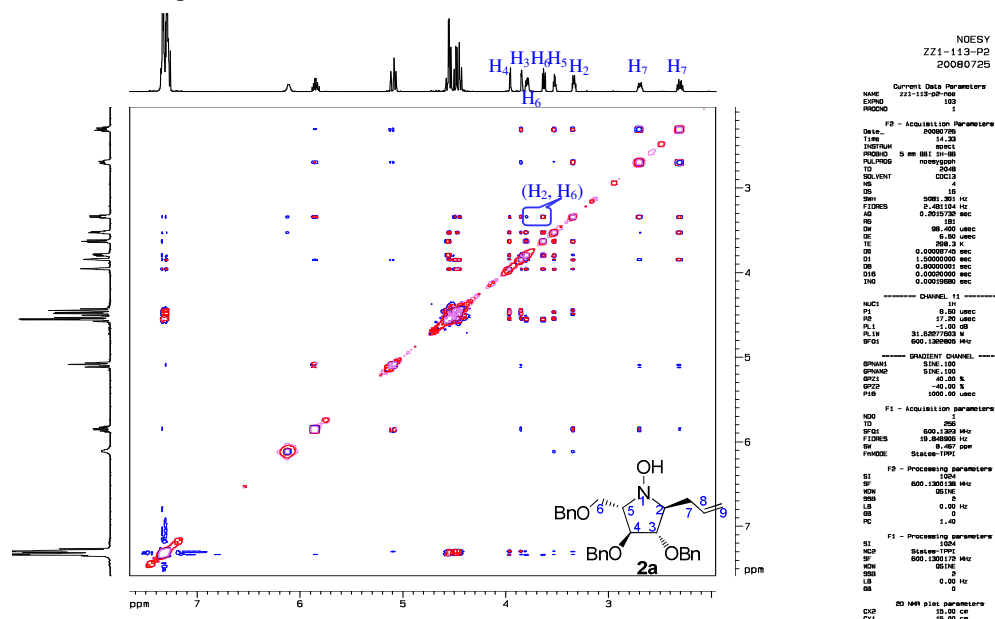
COSY of compound **2a**



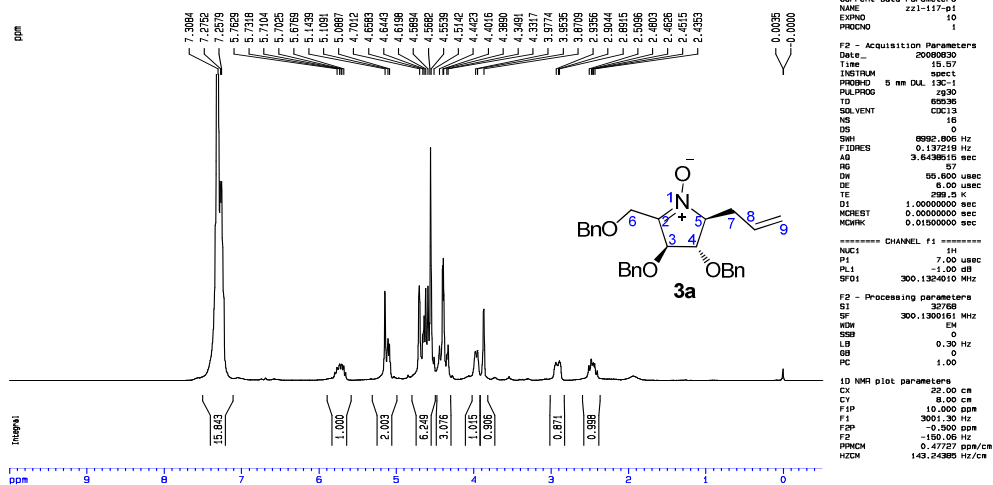
HSQC of compound **2a**



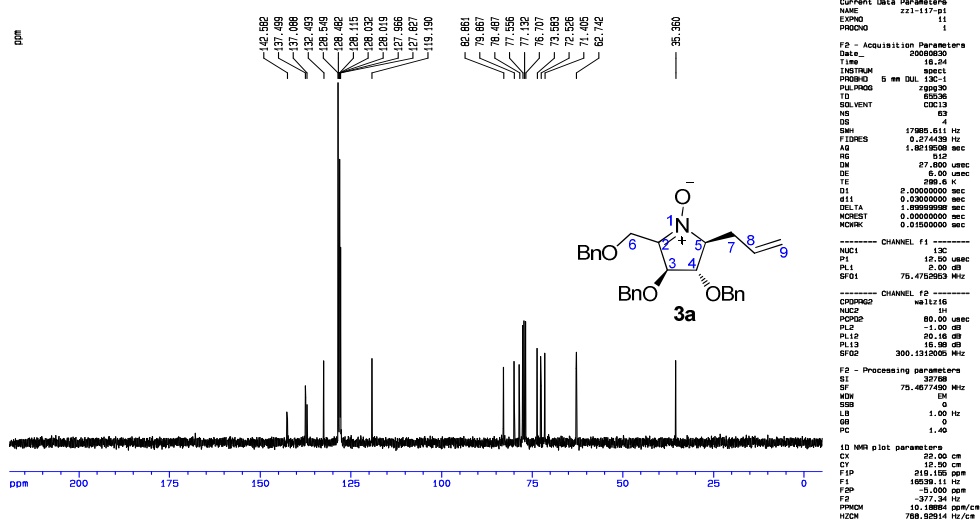
NOESY of compound **2a**



¹H NMR of compound **3a**



¹³C NMR of compound **3a**



Current Data Parameters

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PROCNO: 20

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RG: 40.3
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DE: 6.00 usec
TE: 298.1 K
D1: 1.00000000 sec
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PL1: -1.00 dB
SFO1: 300.1300410 MHz

F2 - Processing parameters

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F2 NMR plot parameters

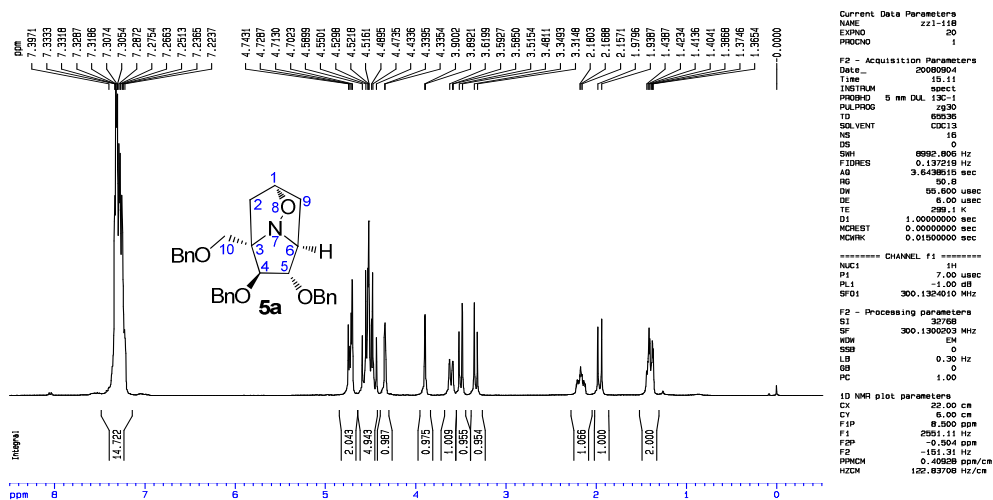
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HZCM: 129.60159 Hz/cm

Chemical structure 4a: CCOC(=O)C1(C)C(COC(=O)C)N(C1)C=C

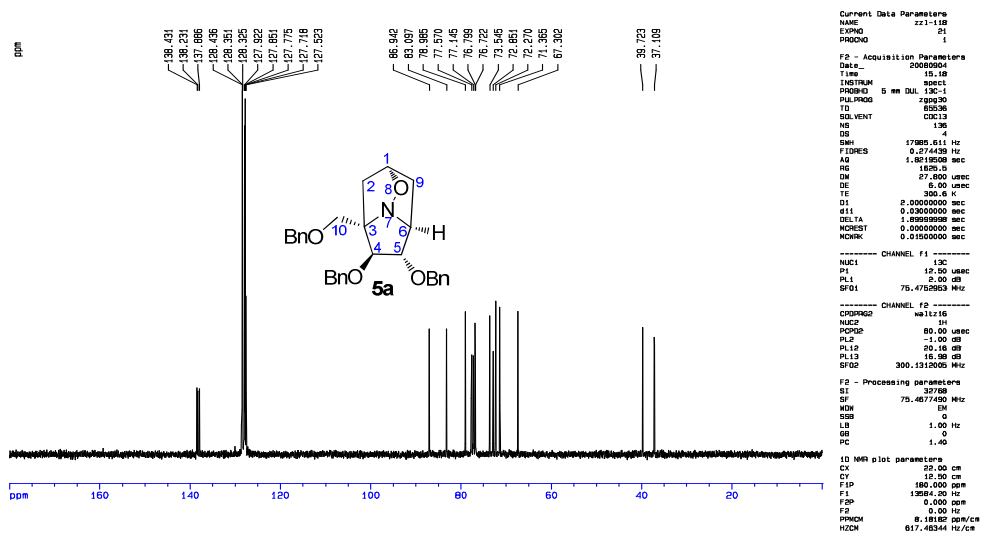
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K2VENT 1
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RG 874.7
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DE 6.00 usec
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WDEXT 0.00000000 sec
NOHST 0.01500000 sec
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SFO1 75.4750553 Mhz
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¹H NMR of compound **5a**



¹³C NMR of compound **5a**



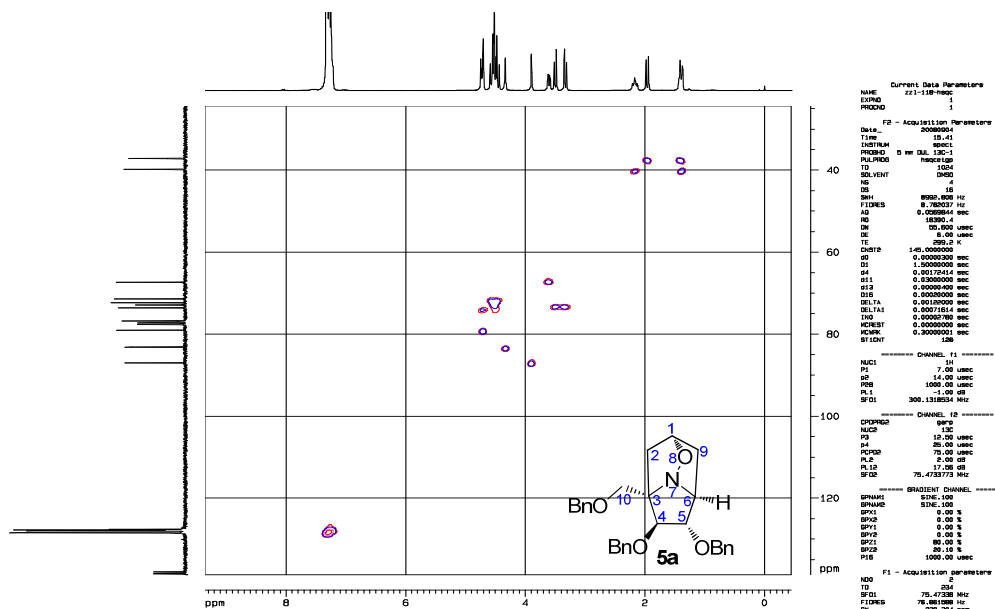
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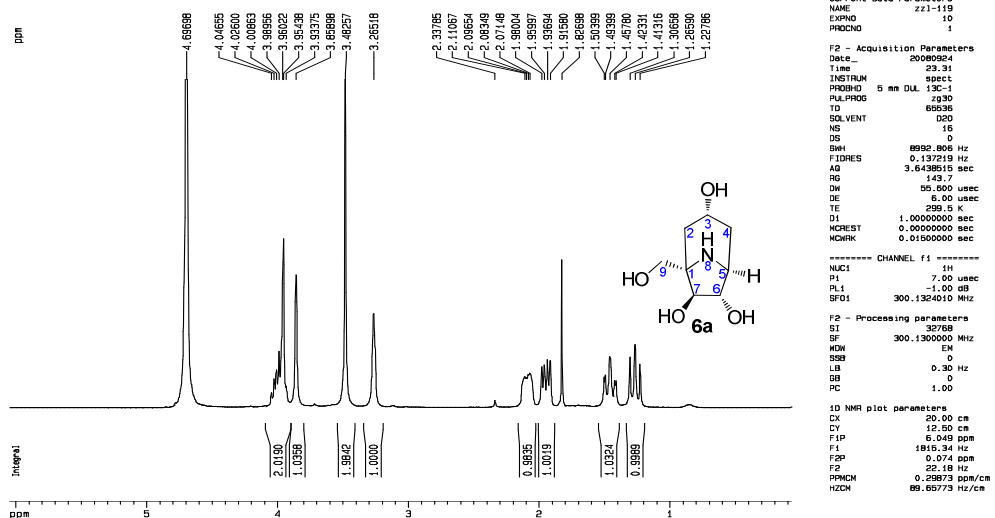
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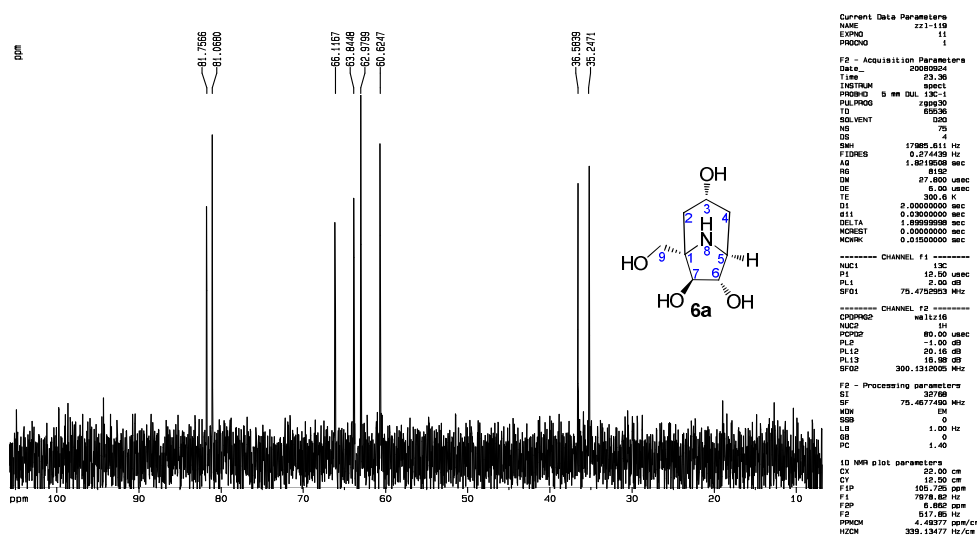
HSQC of compound **5a**



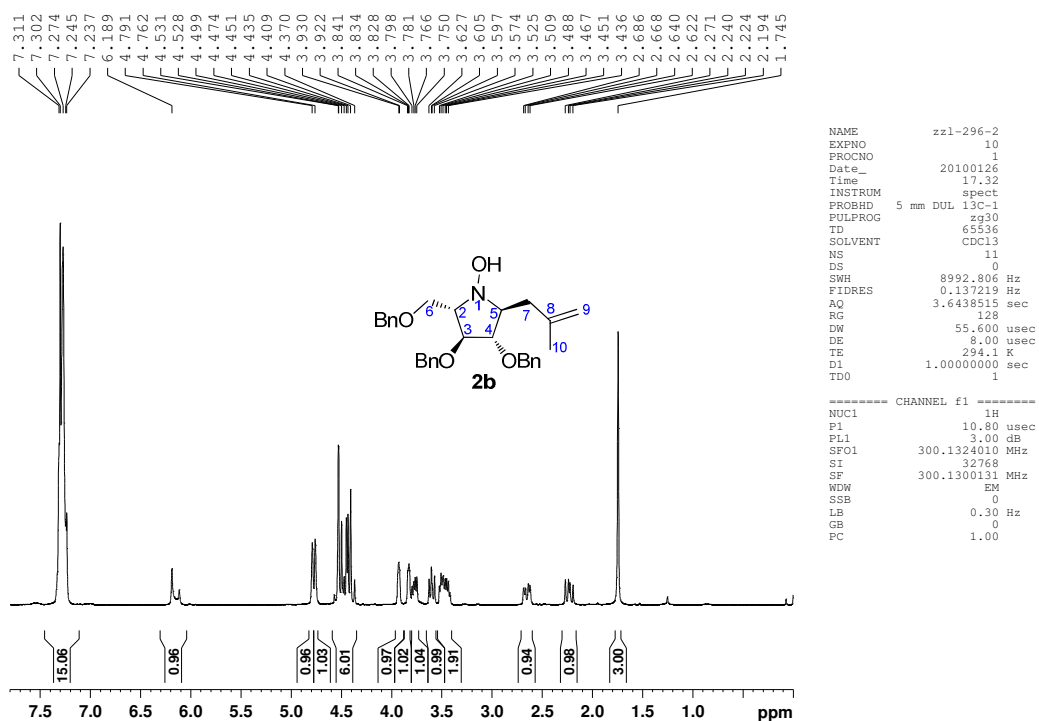
¹H NMR of compound **6a**



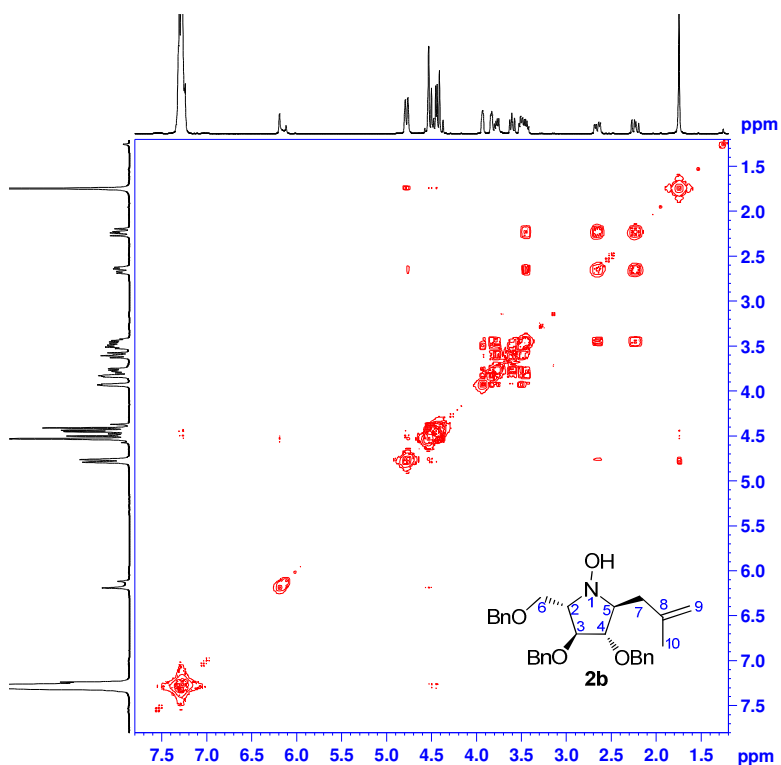
¹³C NMR of compound **6a**



¹H NMR of compound **2b**



COSY of compound **2b**

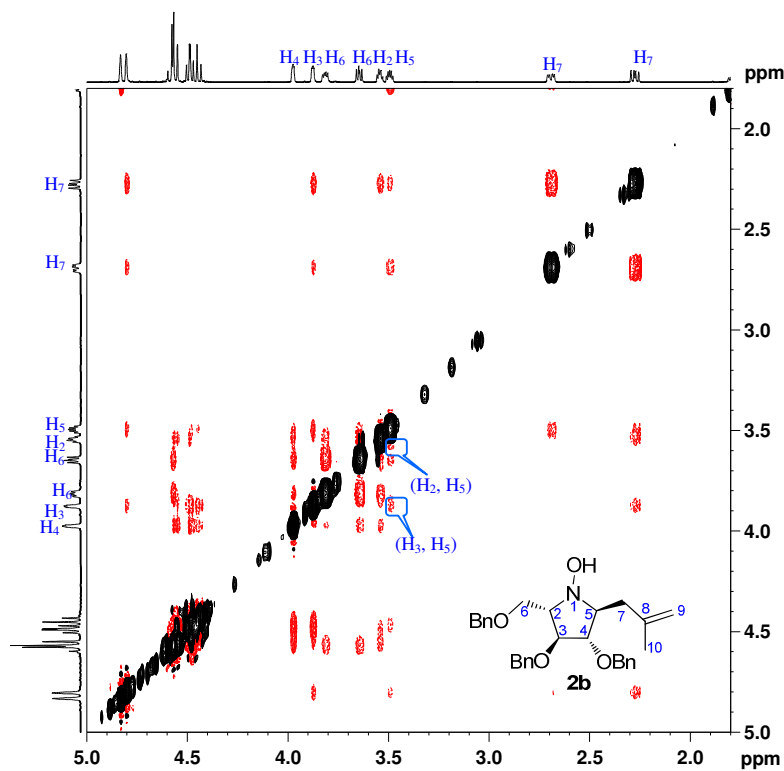


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FIDRES        1.147277 Hz
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DW            212.800 usec
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TE            294.1 K
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D1            1.30625795 sec
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IN0           0.00042560 sec

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NUC1          1H
P0            10.80 usec
P1            10.80 usec
PL1           3.00 dB
SFO1          300.1314022 MHz

===== GRADIENT CHANNEL =====
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GPZ1          10.00 %
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FIDRES        34.052521 Hz
SW            7.829 ppm
FnmODE        QF
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PROCNO        1

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SOLVENT       CDC13
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DS            16
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FIDRES        2.630826 Hz
AQ            0.1901044 sec
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DW            92.800 usec
DE            6.50 usec
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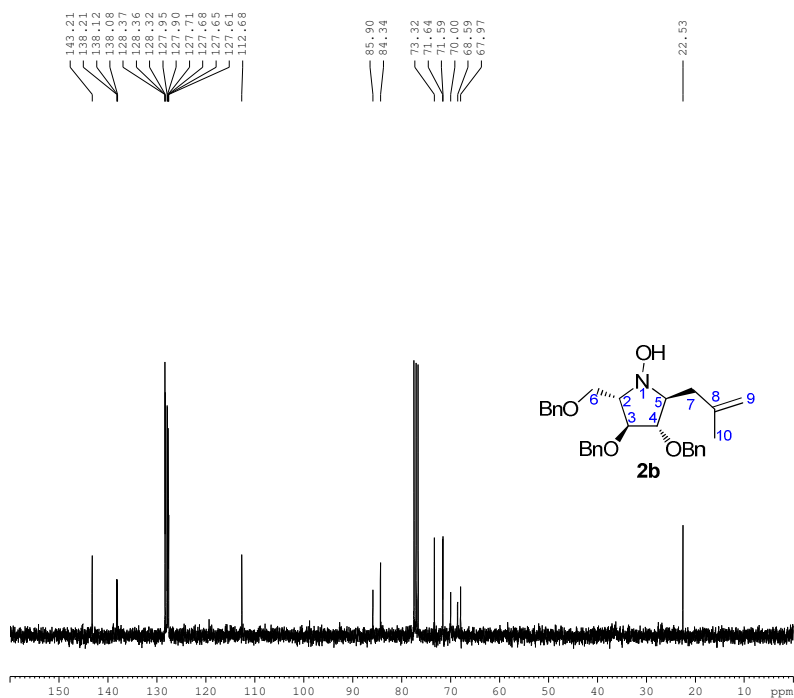
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SFO1          600.1324005 MHz

===== GRADIENT CHANNEL =====
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GPNAM2        SINE.100
GPZ1          40.00 %
GPZ2          -40.00 %
P16           1000.00 usec

F1 - Acquisition parameters
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TD            256
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FIDRES        21.046597 Hz
SW            8.978 ppm
FnmODE        States-TPPI

F2 - Processing parameters
SI            1024
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¹³C NMR of compound **2b**



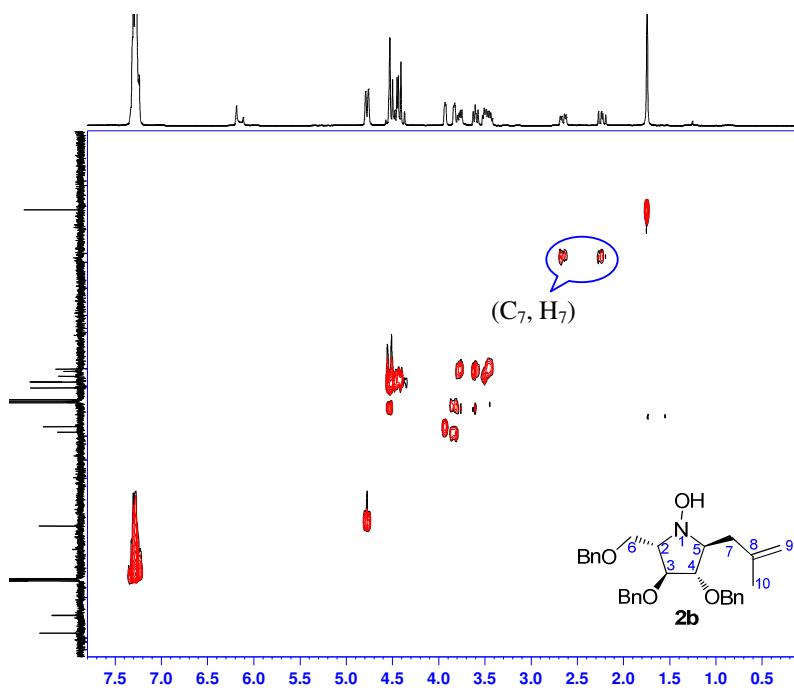
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PROCNO     1
Date_      20100126
Time       17.37
INSTRUM    spect
PROBHD     5 mm DUL 13C-1
PULPROG    zgpg30
TD          65536
SOLVENT     CDCl3
NS          53
DS          4
SWH         17985.611 Hz
FIDRES     0.274439 Hz
AQ          1.8219508 sec
RG          18390.4
DW          27.800 usec
DE          8.00 usec
TE          294.4 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1        13C
P1          12.50 usec
PL1         2.00 dB
SFO1        75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2     waltz16
NUC2         1H
PCPD2       100.00 usec
PL2          3.00 dB
PL12        22.33 dB
PL13        23.00 dB
SFO2        300.1312005 MHz
SI          32768
SF          75.4677490 MHz
WDW          EM
SSB          0
LE           1.00 Hz
GB           0
PC           1.40
    
```

HSQC of compound **2b**



```

Current Data Parameters
NAME      zzi-296-2-hsqc
EXPNO     1
PROCNO     1

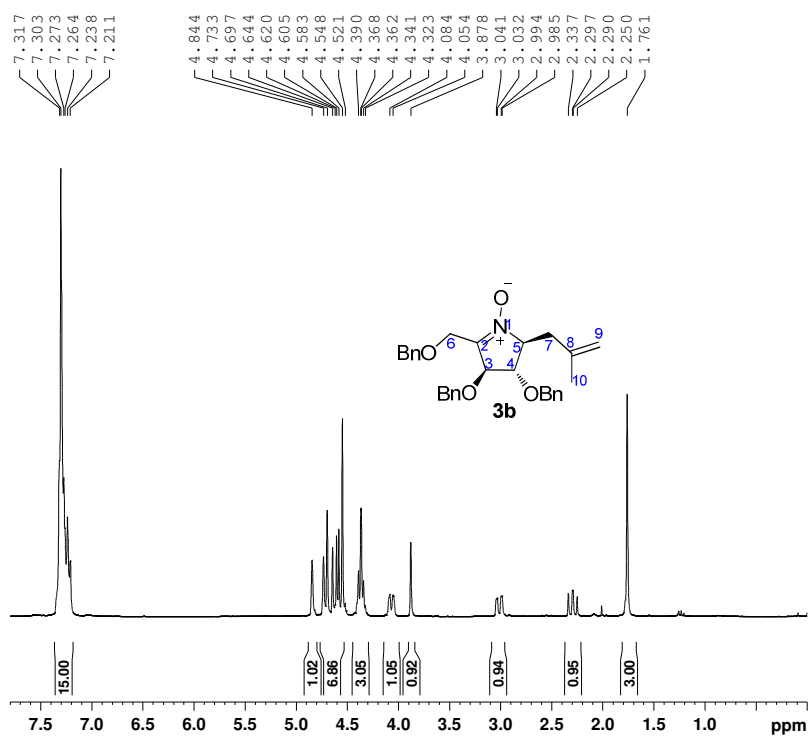
F2 - Acquisition Parameters
Date_      20100126
Time       17.47
INSTRUM    spect
PROBHD     5 mm DUL 13C-1
PULPROG    hsqcetp
TD          2048
SOLVENT     CDCl3
NS          4
DS          16
SWH         2394.636 Hz
FIDRES     1.169256 Hz
AQ          0.4276724 sec
RG          256
DW          208.800 usec
DE          8.00 usec
TE          294.5 K
CNST2      145.0000000
D0          0.00000300 sec
D1          1.50000000 sec
D4          0.00172414 sec
D11         0.03000000 sec
D13         0.00000400 sec
D16         0.00020000 sec
INO         0.00004140 sec
ZGPGTNS

===== CHANNEL f1 =====
NUC1        1H
P1          10.80 usec
P2          21.60 usec
P23         1000.00 usec
PL1          3.00 dB
SFO1        300.1312005 MHz

===== CHANNEL f2 =====
CPDPRG2     gacp
NUC2        13C
P3          12.50 usec
P4          25.00 usec
PCPD2       90.00 usec
PL2          2.00 dB
PL12        19.15 dB
SFO2        75.4737864 MHz

===== GRADIENT CHANNEL =====
GPNAM1      SINE.100
GPNAM2      RTNR.100
    
```

¹H NMR of compound **3b**



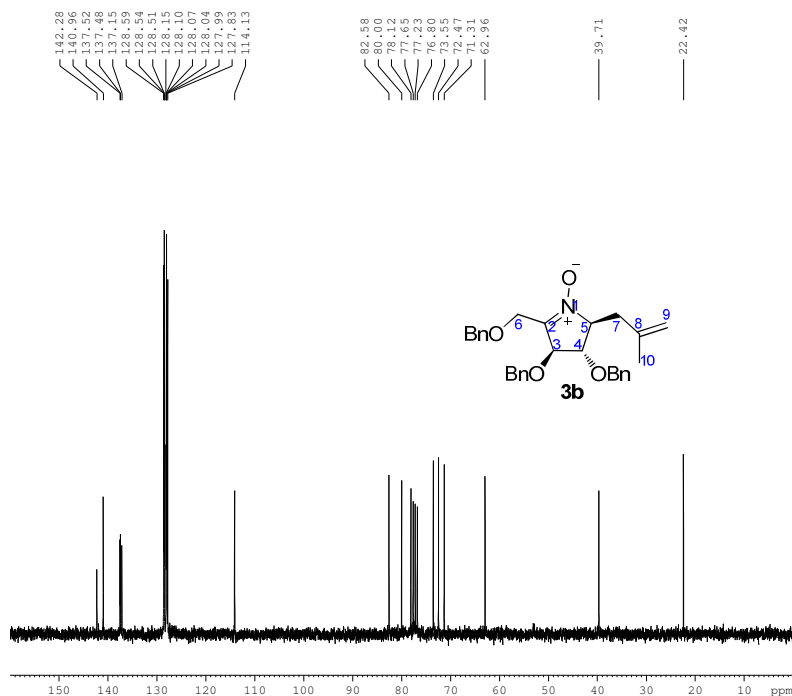
Current Data Parameters
NAME zz1-299-p1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091124
Time 17.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 35.9
DW 81.000 usec
DE 8.00 usec
TE 297.0 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR of compound **3b**



Current Data Parameters
NAME zz1-299-p1
EXPNO 12
PROCNO 1

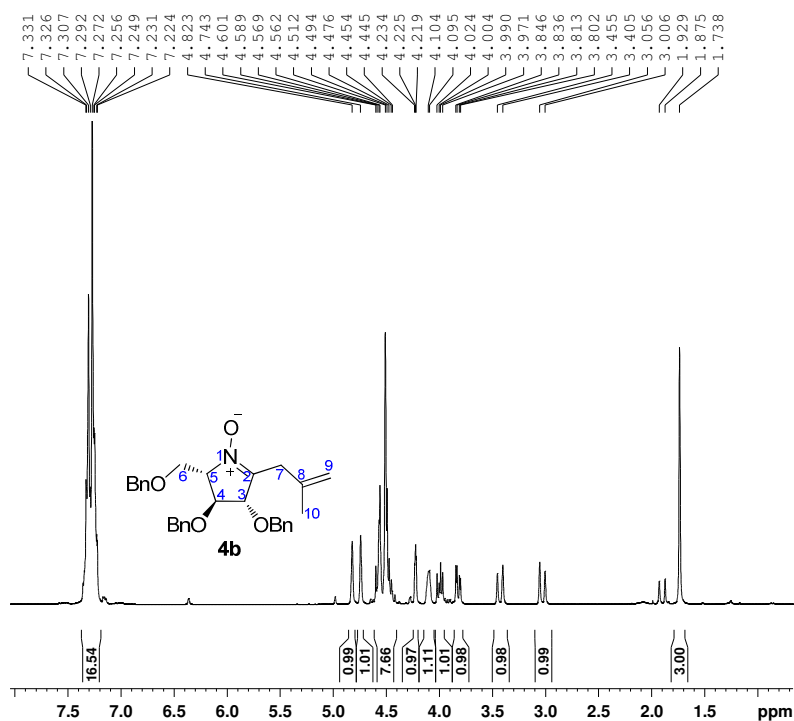
F2 - Acquisition Parameters
Date_ 20091124
Time 17.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 14596.5
DW 27.800 usec
DE 8.00 usec
TE 297.2 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 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

¹H NMR of compound **4b**



```

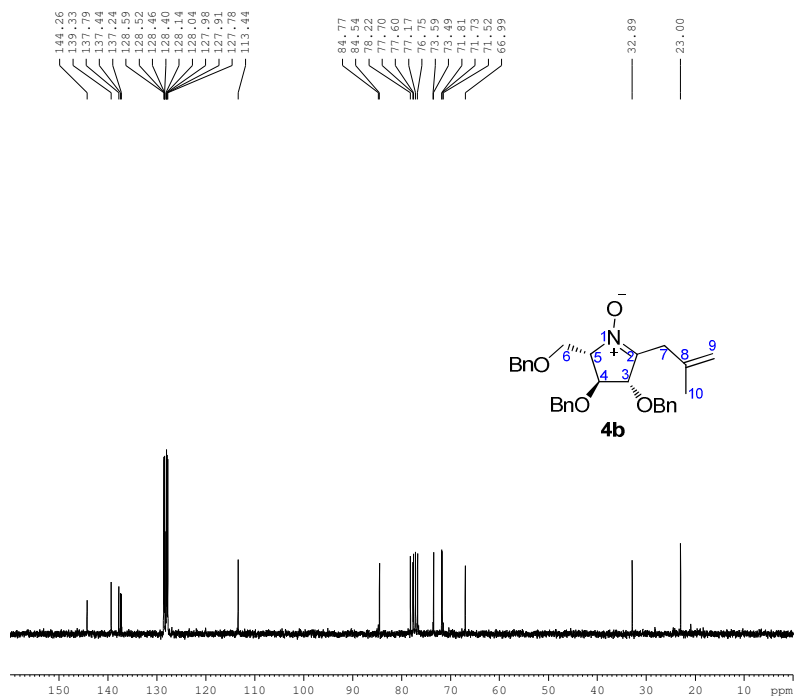
Current Data Parameters
NAME          zzi-299-p2
EXPNO         10
PROCNO        1

F2 - Acquisition Parameters
Date_         20091125
Time          9.24
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG            45.3
DW            81.000 usec
DE            8.00 usec
TE            295.5 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1           1H
P1            10.80 usec
PL1           3.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI            32768
SF            300.1300168 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

¹³C NMR of compound **6a**



```

Current Data Parameters
NAME          zzi-299-p2
EXPNO         12
PROCNO        1

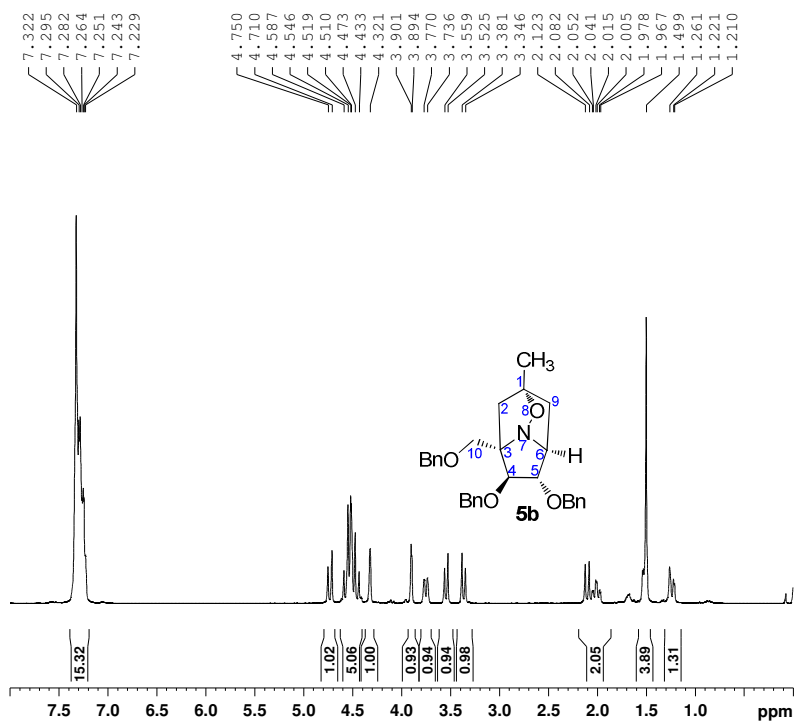
F2 - Acquisition Parameters
Date_         20091125
Time          9.31
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            27
DS            4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG            13004
DW            27.800 usec
DE            8.00 usec
TE            295.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1           13C
P1            12.50 usec
PL1           2.00 dB
SFO1          75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         100.00 usec
PL2           3.00 dB
PL12          22.33 dB
PL13          23.00 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
  
```

¹H NMR of compound **5b**



```

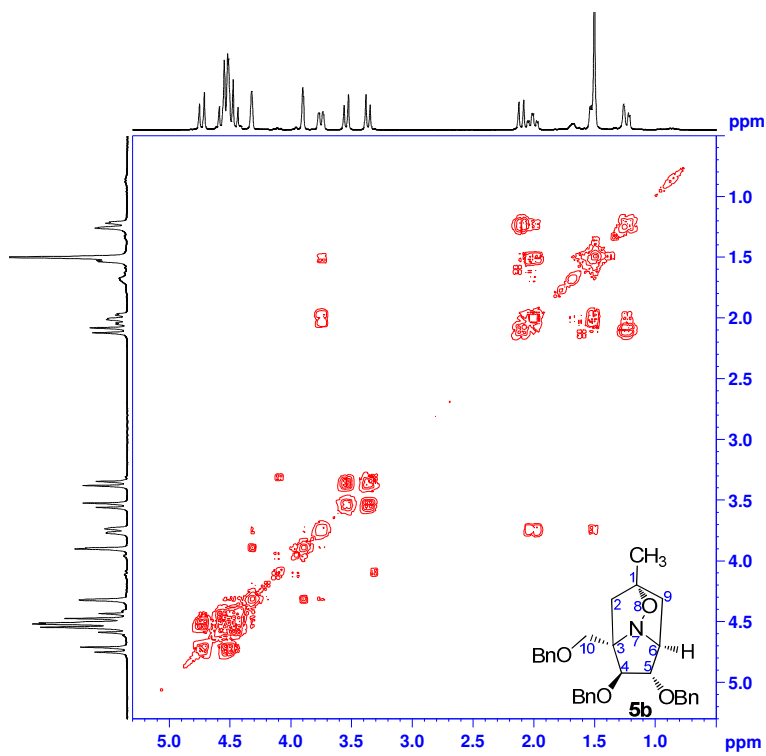
Current Data Parameters
NAME      zz1-302
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20091203
Time      15.27
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       6172.839 Hz
FIDRES    0.094190 Hz
AQ        5.3084660 sec
RG        128
DW        81.000 usec
DE        8.00 usec
TE        298.5 K
D1        1.00000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      1H
P1        10.80 usec
PL1       3.00 dB
SFO1      300.1318534 MHz

F2 - Processing parameters
SI        32768
SF        300.1300115 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```

COSY of compound **5b**



```

Current Data Parameters
NAME      zz1-302
EXPNO     11
PROCNO    1

F2 - Acquisition Parameters
Date_     20091203
Time      15.28
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   cosygpqf2
TD        2048
SOLVENT   CDCl3
NS        1
DS        8
SWH       2796.421 Hz
FIDRES    1.365440 Hz
AQ        0.3662324 sec
RG        64
DW        178.800 usec
DE        8.00 usec
TE        298.5 K
D0        0.00000300 sec
D1        1.37589002 sec
D13       0.00000400 sec
D16       0.00020000 sec
IN0       0.00035760 sec

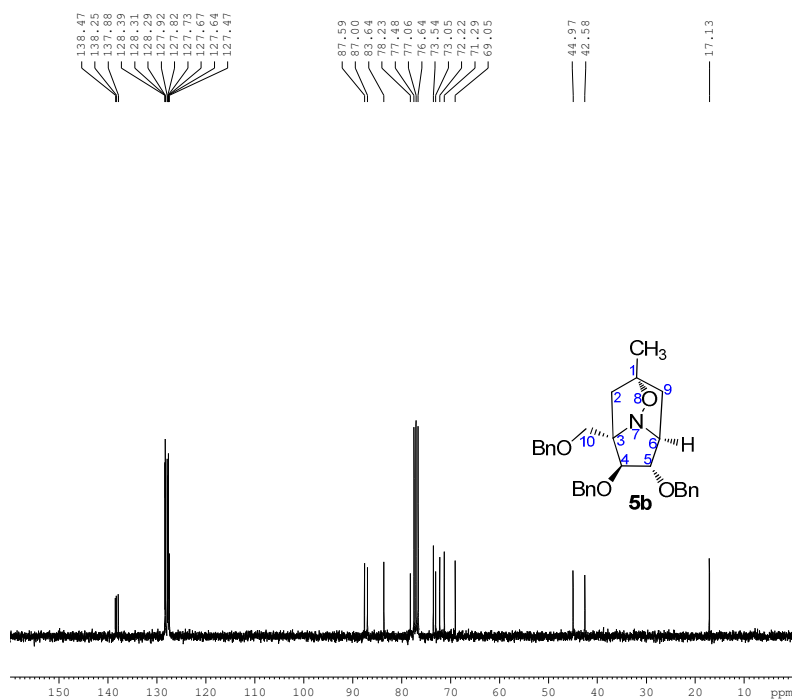
===== CHANNEL f1 =====
NUC1      1H
P0        10.80 usec
P1        10.80 usec
PL1       3.00 dB
SFO1      300.1312302 MHz

===== GRADIENT CHANNEL =====
GPNAM1    SINE.100
GP21      10.00 %
P16       1000.00 usec

F1 - Acquisition parameters
ND0       1
TD        128
SFO1      300.1312 MHz
FIDRES    21.847036 Hz
SW        9.317 ppm
FNAME     QF

F2 - Processing parameters
SI        1024
SF        300.1300115 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.40
    
```

¹³C NMR of compound **5b**



```

Current Data Parameters
NAME          zz1-302
EXPNO         12
PROCNO        1

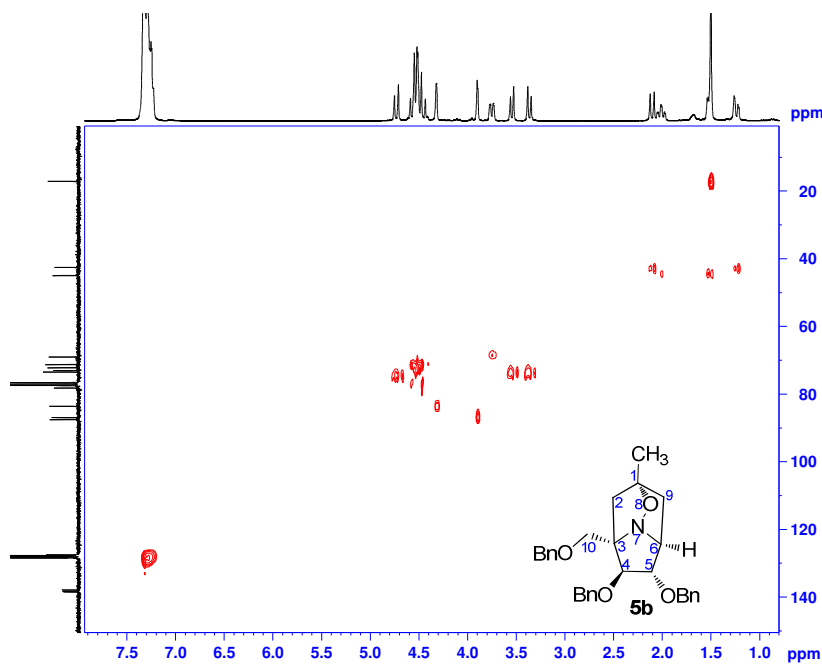
F2 - Acquisition Parameters
Date_         20091203
Time          15.38
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            100
DS            4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG            18390.4
DW            27.800 usec
DE            8.00 usec
TE            299.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            12.50 usec
PL1           2.00 dB
SFO1          75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         100.00 usec
PL2           3.00 dB
PL12          22.33 dB
PL13          23.00 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
  
```

HSQC of compound **5b**



```

Current Data Parameters
NAME          zz1-302-hsqc
EXPNO         1
PROCNO        1

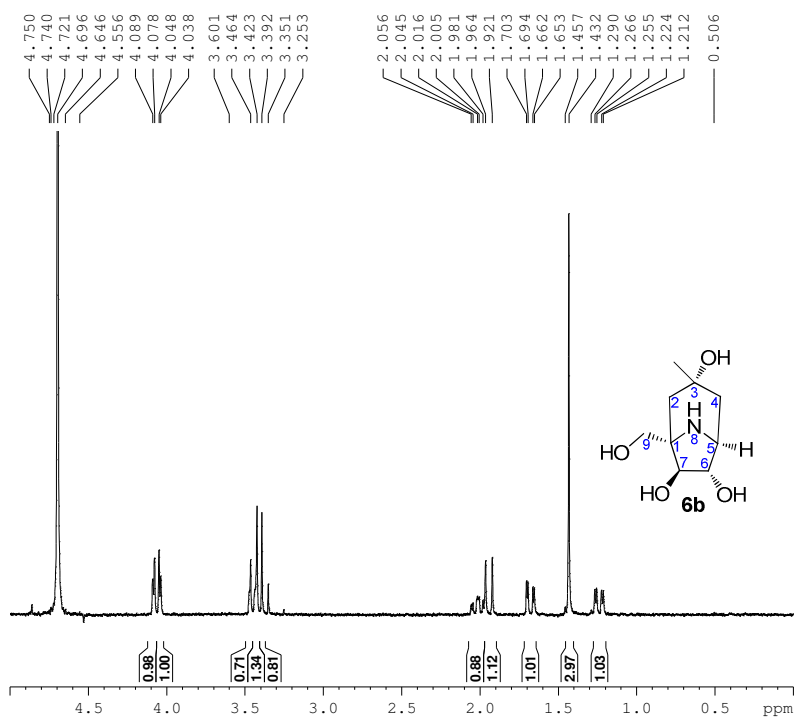
F2 - Acquisition Parameters
Date_         20091203
Time          15.47
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       hsqcetop
TD            2048
SOLVENT       CDCl3
NS            4
DS            16
SWH           2394.636 Hz
FIDRES        1.169256 Hz
AQ            0.4276724 sec
RG            256
DW            208.800 usec
DE            8.00 usec
TE            299.1 K
D1            0.00000300 sec
D11           1.50000000 sec
D4            0.00172414 sec
D111          0.03000000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00004415 sec
ZGOFPTS

===== CHANNEL f1 =====
NUC1          1H
P1            10.80 usec
P2            21.60 usec
P28           1000.00 usec
PL1           3.00 dB
SFO1          300.1312005 MHz

===== CHANNEL f2 =====
CPDPRG2       garp
NUC2          13C
P3            12.50 usec
P4            25.00 usec
PCPD2         90.00 usec
PL2           2.00 dB
PL12          19.15 dB
SFO2          75.4734091 MHz

===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPZ1          80.00 %
GPZ2          20.10 %
P16           1000.00 usec
  
```

¹H NMR of compound **6b**



```

NAME          zz1-352
EXPNO         40
PROCNO        1
Date_         20100310
Time          9.35
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg30
TD            65536
SOLVENT       D2O
NS            6
DS            0
SWH           8992.806 Hz
FIDRES        0.137219 Hz
AQ            3.6438515 sec
RG            645.1
DW            55.600 usec
DE            8.00 usec
TE            297.2 K
D1            1.00000000 sec
D11           1
D10           1

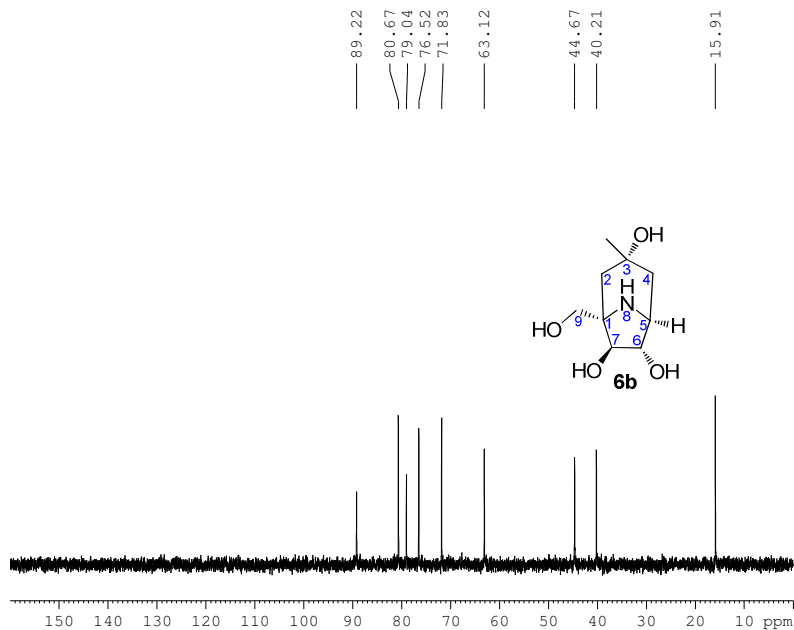
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            10.80 usec
PL1           3.00 dB
SFO1          300.1324010 MHz
SI            32768
SF            300.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```

¹³C NMR of compound **6b**



```

NAME          zz1-352
EXPNO         12
PROCNO        1
Date_         20100303
Time          17.39
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT       D2O
NS            50
DS            4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG            16384
DW            27.800 usec
DE            8.00 usec
TE            295.3 K
D1            2.00000000 sec
D11           0.03000000 sec
D10           1

```

```

===== CHANNEL f1 =====
NUC1          13C
P1            12.50 usec
PL1           2.00 dB
SFO1          75.4752953 MHz

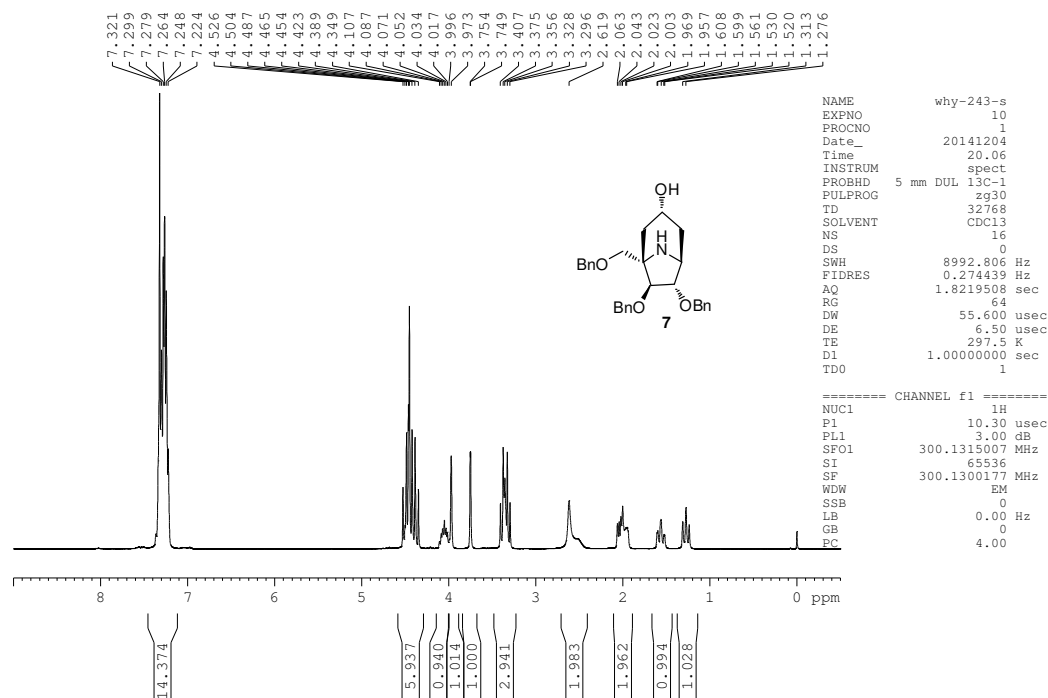
```

```

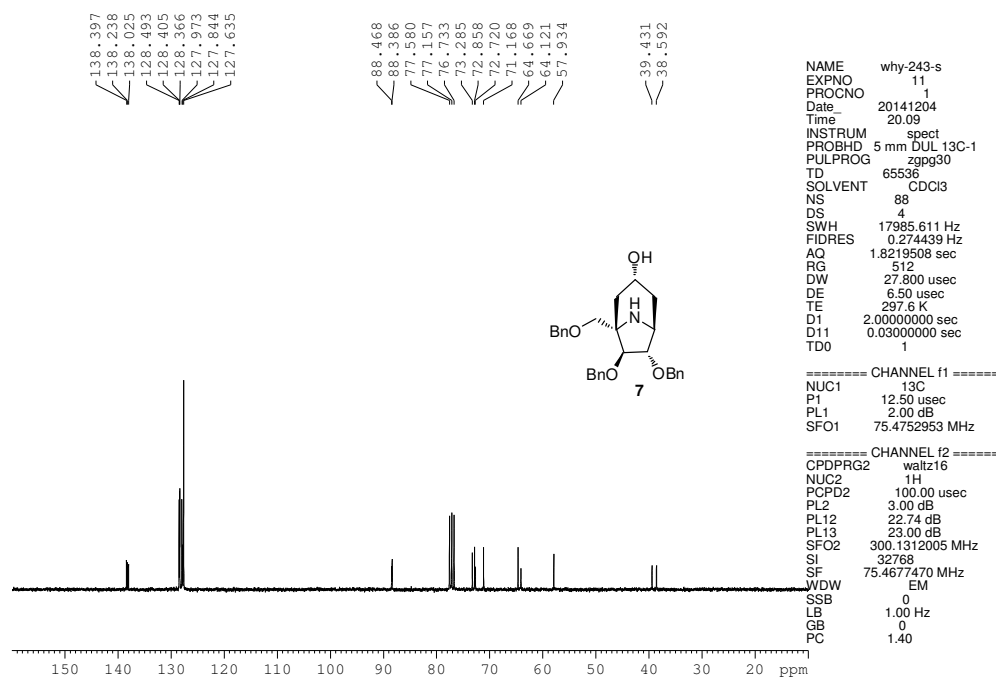
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         100.00 usec
PL2           3.00 dB
PL12          22.33 dB
PL13          23.00 dB
SFO2          300.1312005 MHz
SI            32768
SF            75.4677490 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

```

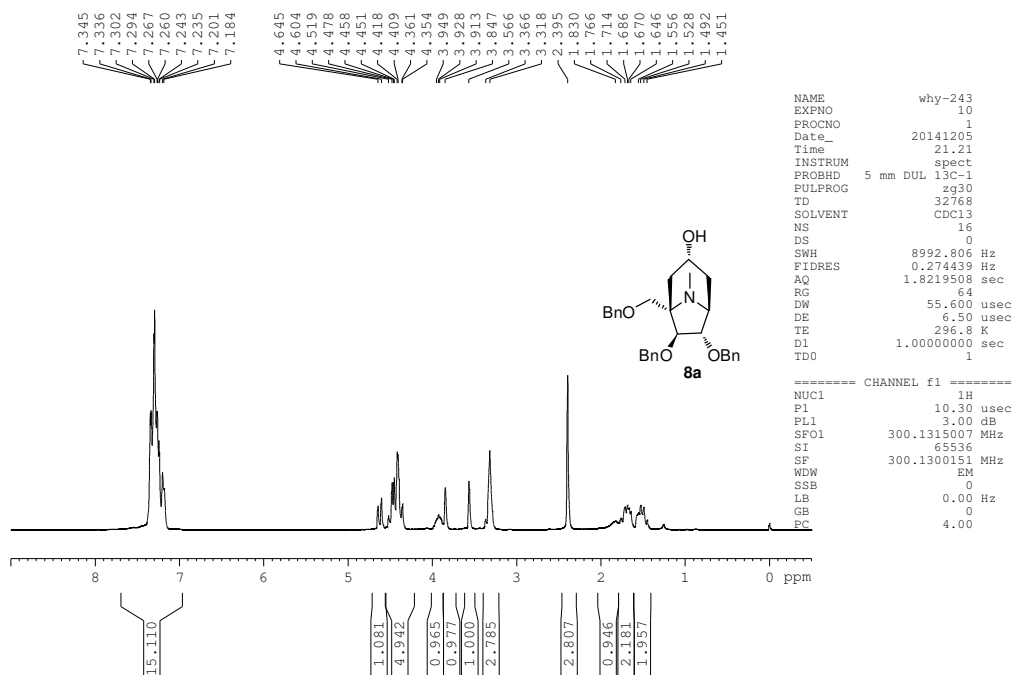
¹H NMR of compound 7



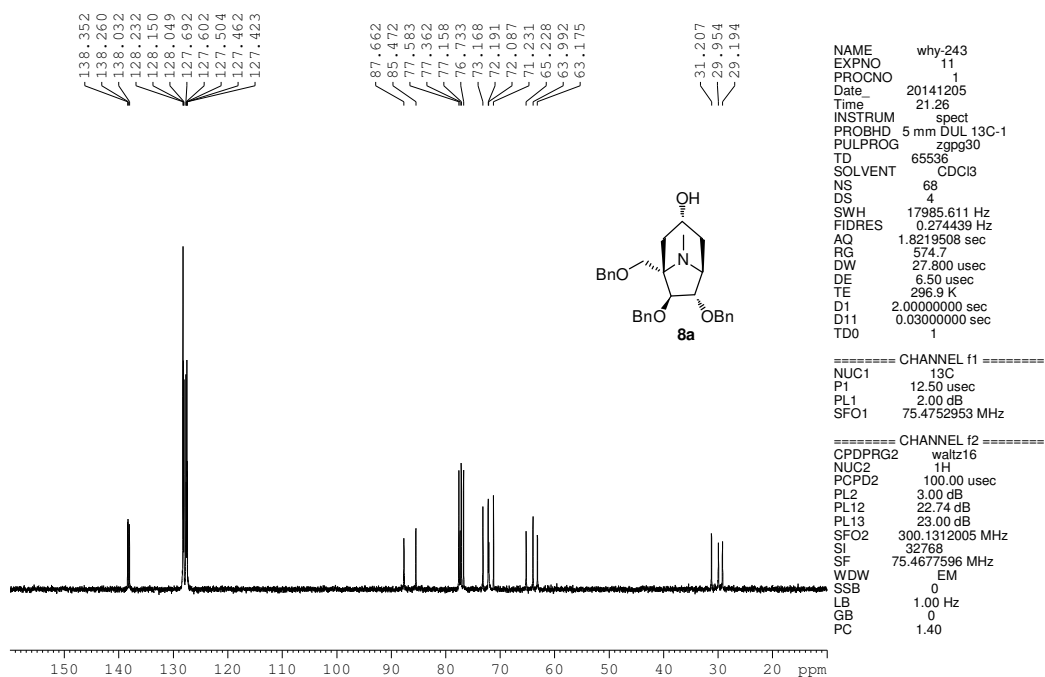
¹³C NMR of compound 7



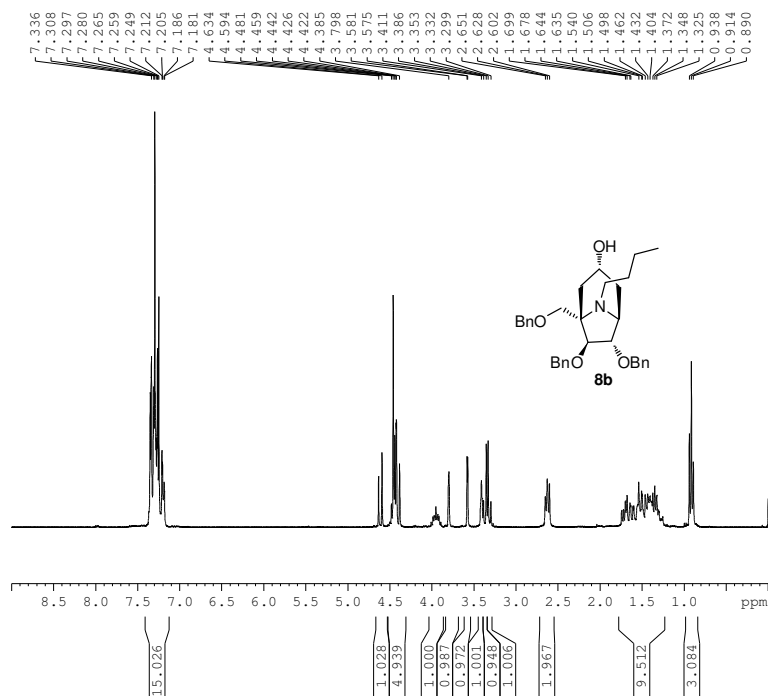
¹H NMR of compound **8a**



¹³C NMR of compound **8a**



¹H NMR of compound **8b**

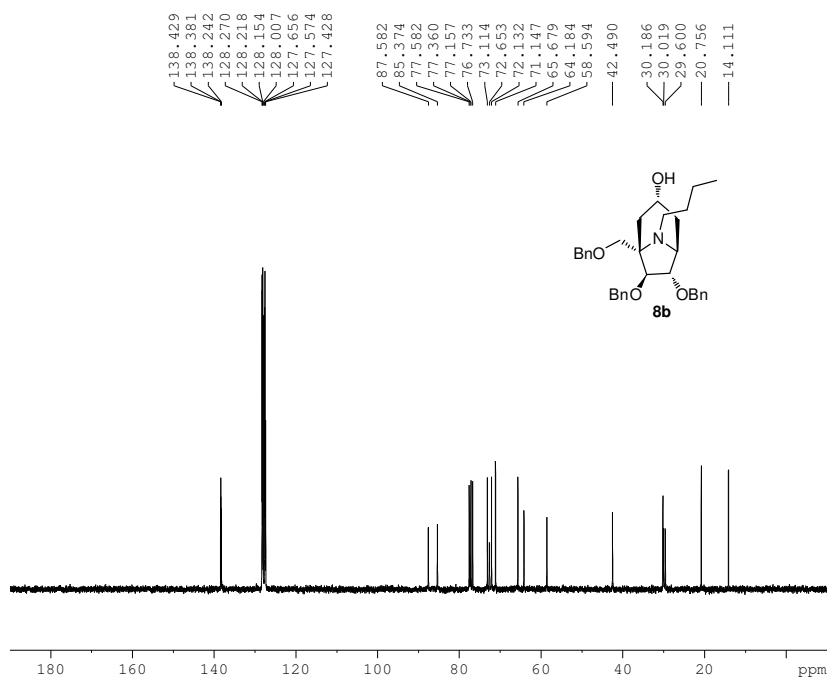


```

NAME      why-244
EXPNO     10
PROCNO    1
Date_     20141205
Time      21.35
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zg30
TD         32768
SOLVENT   CDCl3
NS         16
DS         0
SWH        8992.806 Hz
FIDRES     0.274439 Hz
AQ         1.8219508 sec
RG         181
DW         55.600 usec
DE         6.50 usec
TE         297.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         10.30 usec
PL1        3.00 dB
SFO1       300.1315007 MHz
SI         65536
SF         300.1300101 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0
PC         4.00
  
```

¹³C NMR of compound **8b**



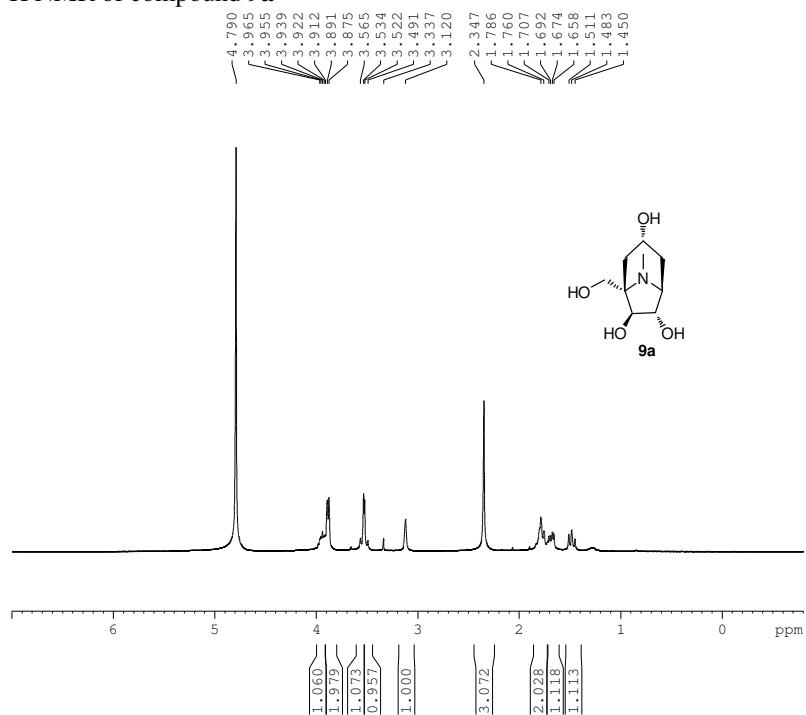
```

NAME      why-244
EXPNO     20
PROCNO    1
Date_     20141205
Time      21.40
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         79
DS         4
SWH        17985.611 Hz
FIDRES     0.274439 Hz
AQ         1.8219508 sec
RG         574.7
DW         27.800 usec
DE         6.50 usec
TE         297.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         12.50 usec
PL1        2.00 dB
SFO1       75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     100.00 usec
PL2        3.00 dB
PL12       22.74 dB
PL13       23.00 dB
SFO2       300.1312005 MHz
SI         32768
SF         75.4677586 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

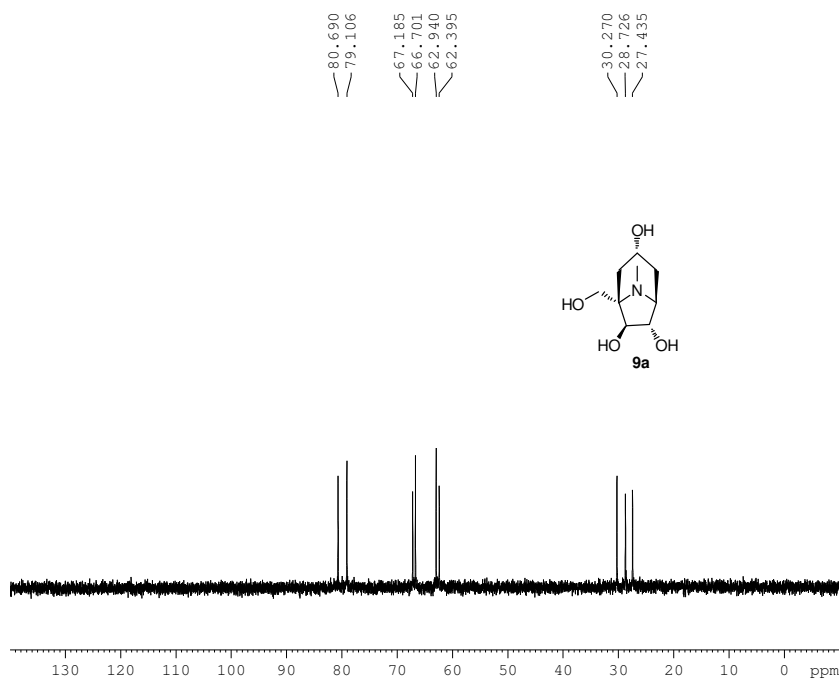
¹H NMR of compound **9a**



NAME 400MHZ
EXPNO 282
PROCNO 1
Date_ 20141207
Time 17.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT D2O
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 140.59
DW 62.400 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.2424716 MHz
NUC1 1H
P1 14.80 usec
SI 65536
SF 400.2399651 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

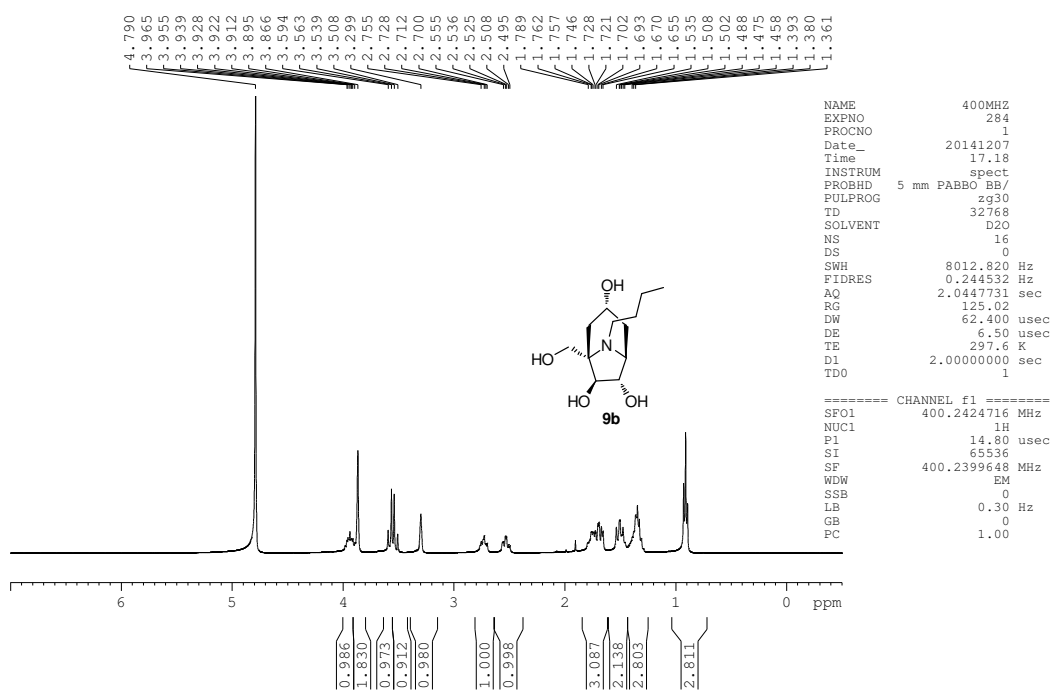
¹³C NMR of compound **9a**



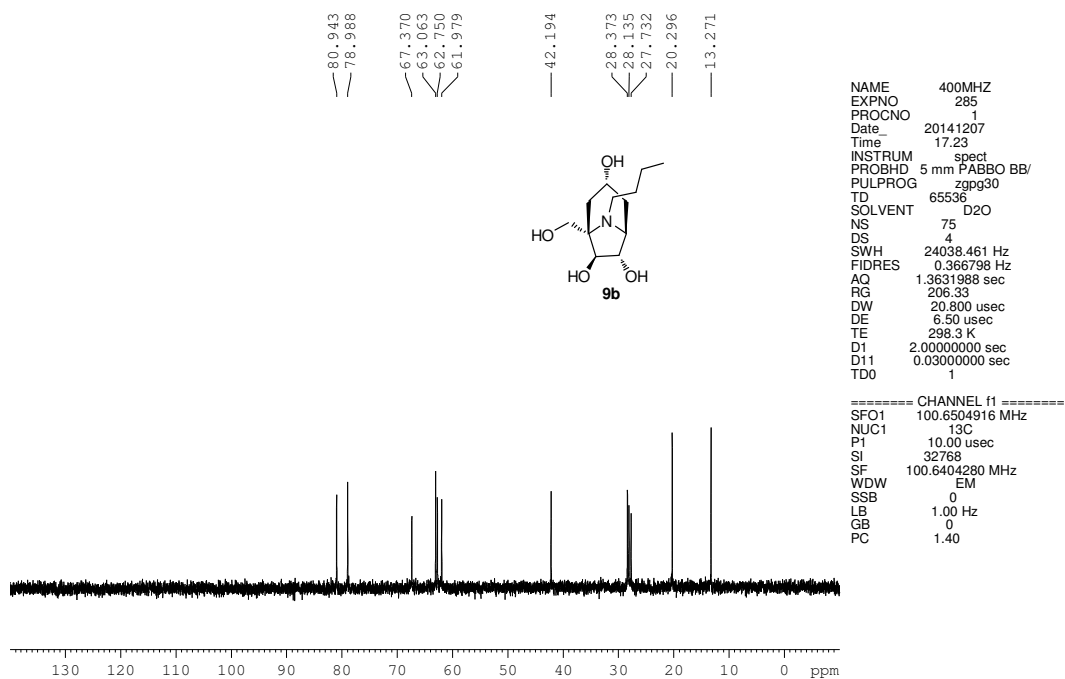
NAME 400MHZ
EXPNO 283
PROCNO 1
Date_ 20141207
Time 17.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 78
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 206.33
DW 20.300 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6504916 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6404280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

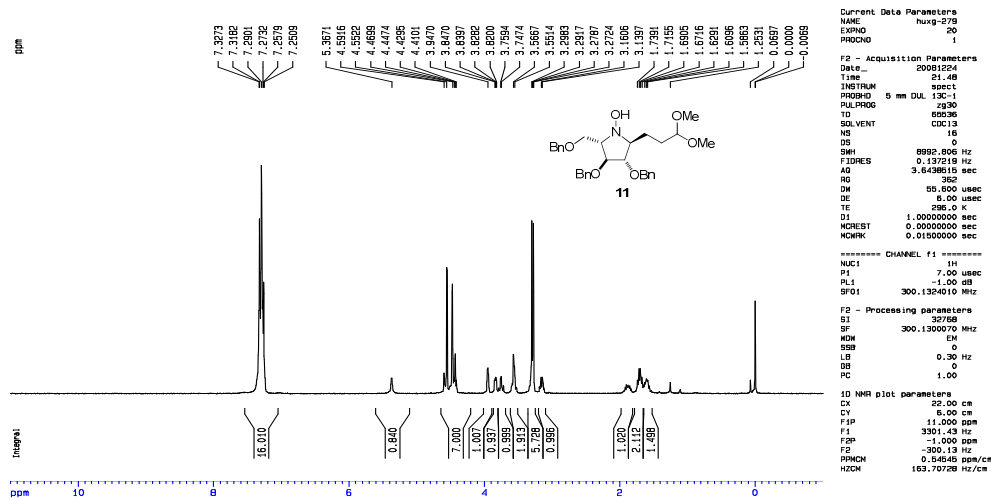
¹H NMR of compound **9b**



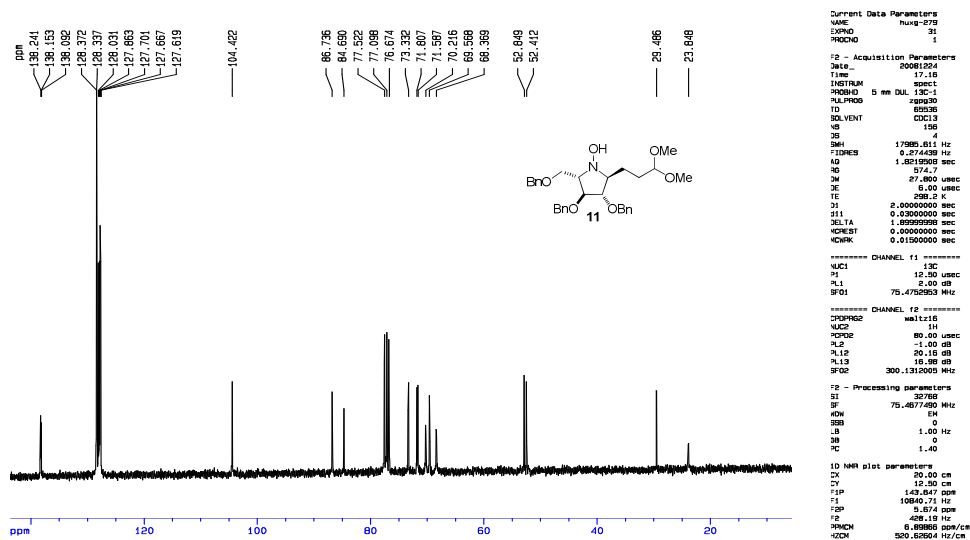
¹³C NMR of compound **9b**



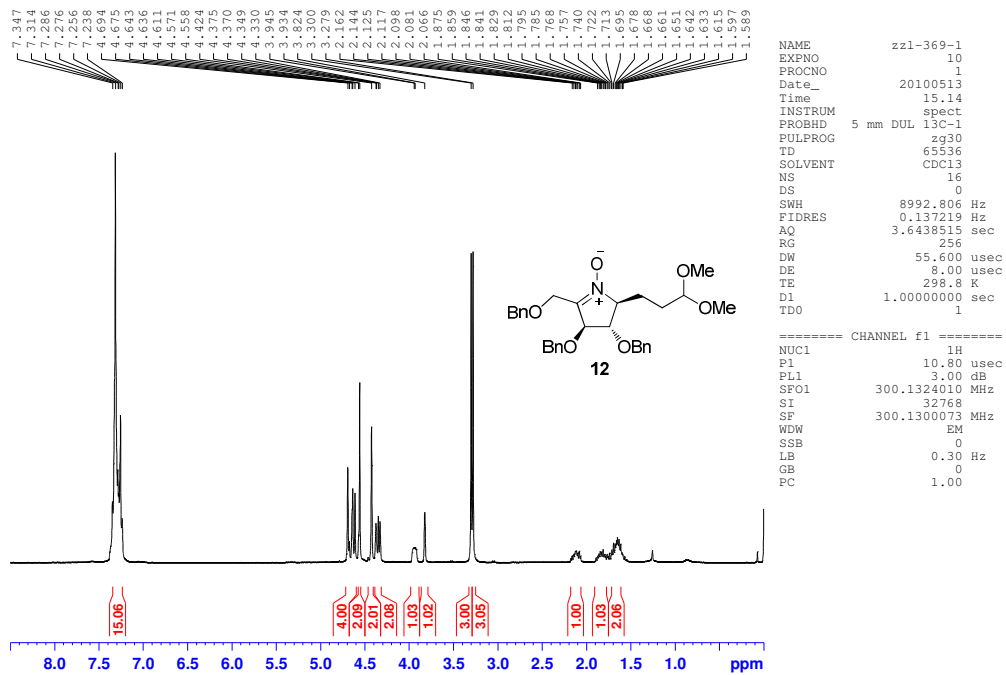
¹H NMR of compound 11



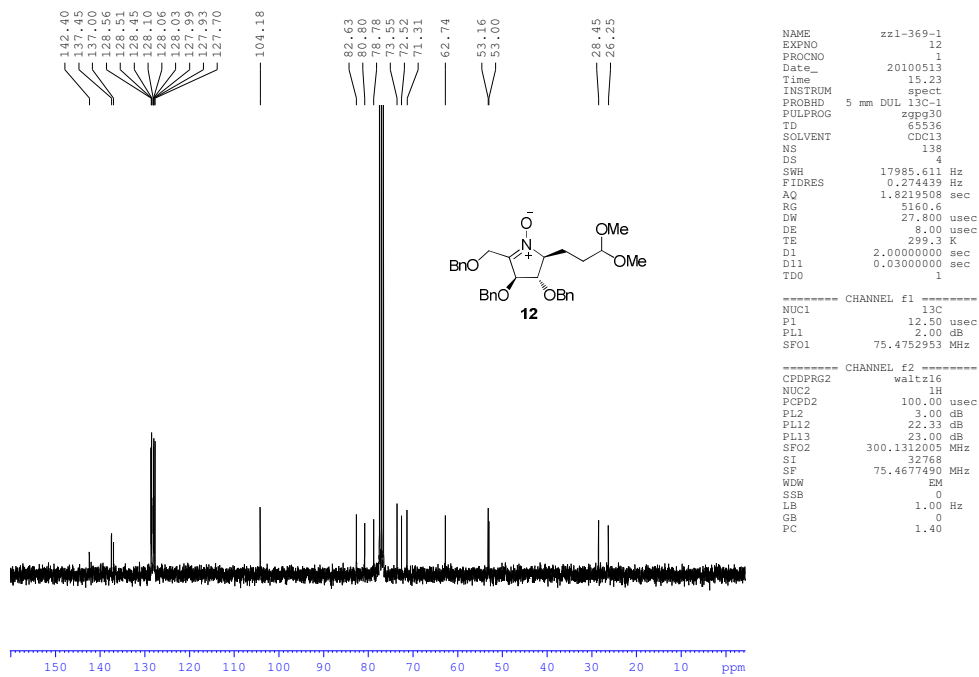
¹³C NMR of compound 11



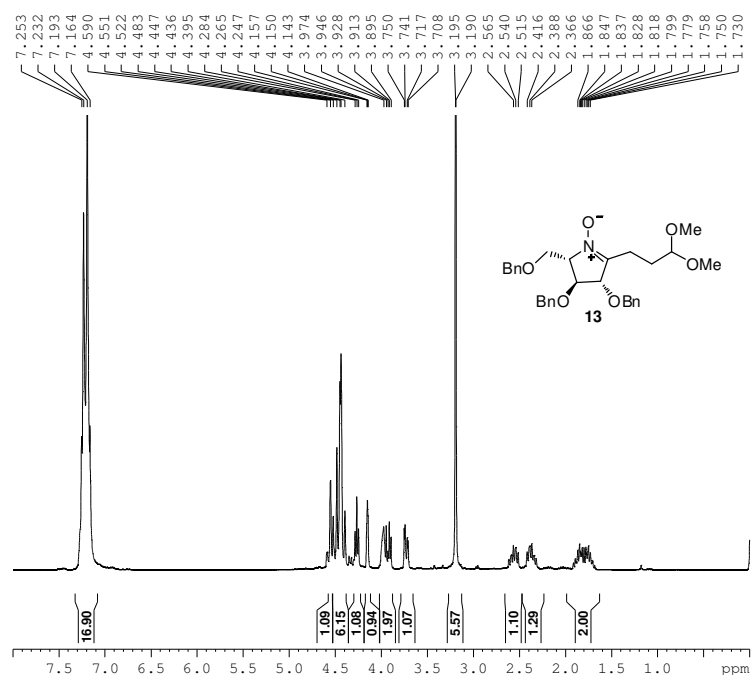
¹H NMR of compound **12**



¹³C NMR of compound **12**



¹H NMR of compound **13**

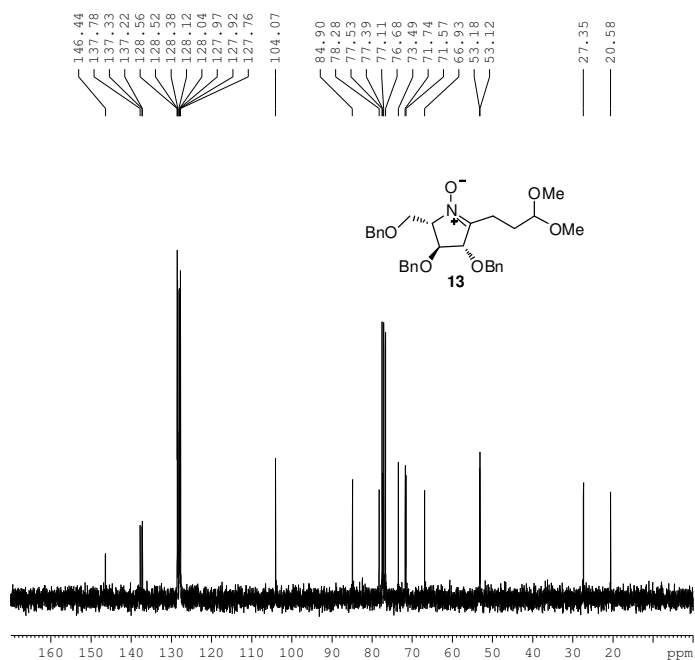


```

NAME      zzl-368-2
EXPNO     10
PROCNO    1
Date_     20100518
Time      9.16
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         10
DS         0
SWH        8992.806 Hz
FIDRES     0.137219 Hz
AQ         3.6438515 sec
RG         64
DW         55.600 usec
DE         8.00 usec
TE         299.1 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         10.80 usec
PL1        3.00 dB
SFO1       300.1324010 MHz
SI         32768
SF         300.1300347 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

¹³C NMR of compound **13**



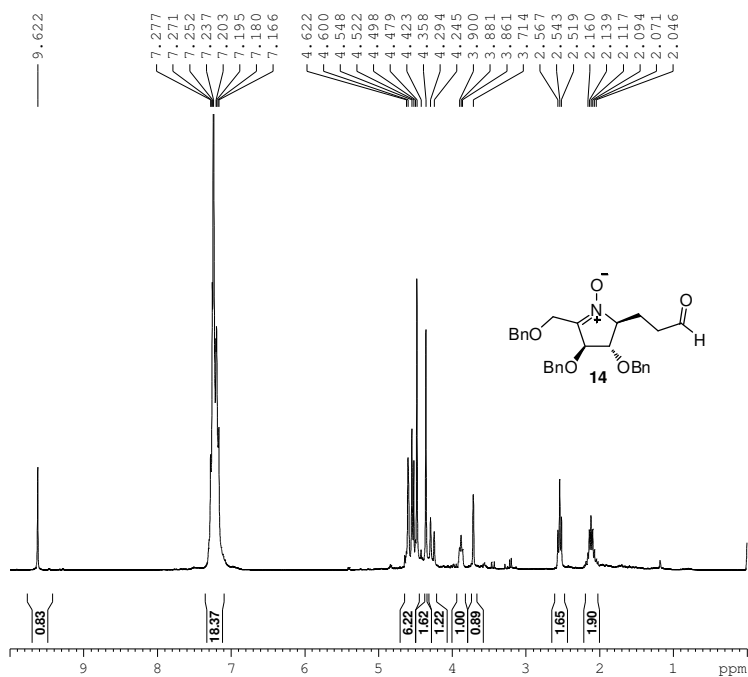
```

NAME      zzl-368-2
EXPNO     12
PROCNO    1
Date_     20100518
Time      9.23
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         21
DS         4
SWH        17995.611 Hz
FIDRES     0.274439 Hz
AQ         1.6219508 sec
RG         13004
DW         27.800 usec
DE         8.00 usec
TE         299.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         12.50 usec
PL1        2.00 dB
SFO1       75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     100.00 usec
PL2        3.00 dB
PL12       22.33 dB
PL13       23.00 dB
SFO2       300.1312005 MHz
SI         32768
SF         75.4677490 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

¹H NMR of compound **14**



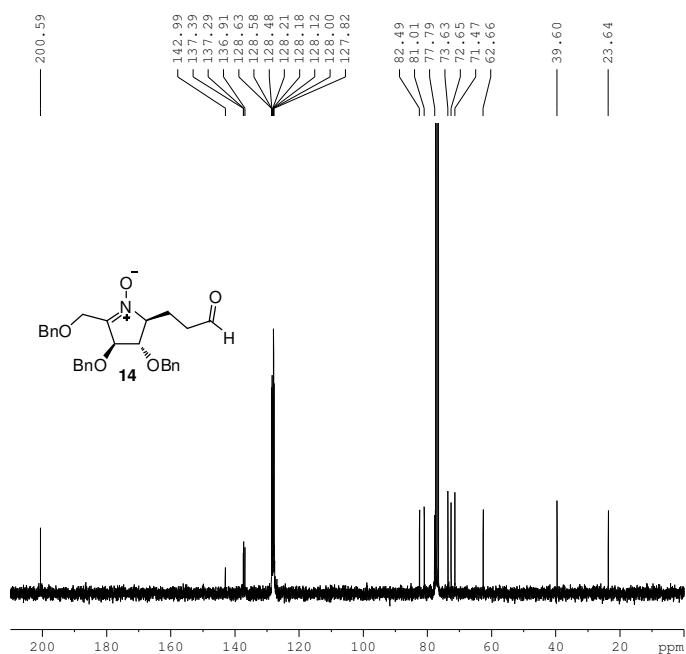
```

NAME      zz1-370
EXPNO     10
PROCNO    1
Date_     20100520
Time      22.22
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        8992.806 Hz
FIDRES     0.137219 Hz
AQ         3.6438515 sec
RG         128
DW         55.600 usec
DE         8.00 usec
TE         299.3 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         10.80 usec
PL1        3.00 dB
SFO1      300.1324010 MHz
SI         32768
SF         300.1300342 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

¹³C NMR of compound **14**



```

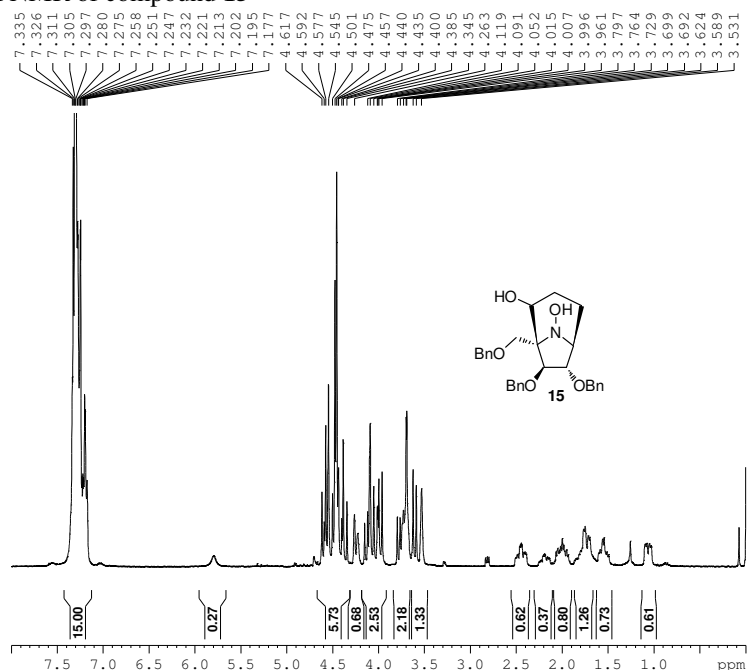
NAME      zz1-370
EXPNO     11
PROCNO    1
Date_     20100520
Time      22.28
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         94
DS         4
SWH        17985.611 Hz
FIDRES     0.274439 Hz
AQ         1.8219508 sec
RG         13004
DW         27.800 usec
DE         8.00 usec
TE         300.6 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         12.50 usec
PL1        2.00 dB
SFO1      75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     100.00 usec
PL2        3.00 dB
PL12      22.33 dB
PL13      23.00 dB
SFO2      300.1312005 MHz
SI         32768
SF         75.4677490 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

¹H NMR of compound **15**

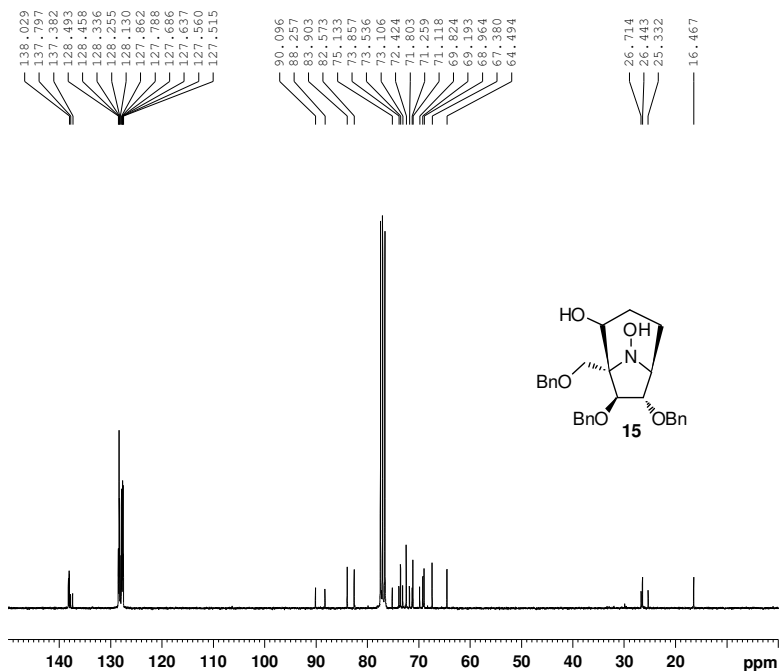


```

NAME          zz1-372
EXPNO         20
PROCNO        1
Date_         20100525
Time          0.48
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            0
SWH           8992.806 Hz
FIDRES        0.137219 Hz
AQ            3.6438515 sec
RG            228.1
DW            55.600 usec
DE            8.00 usec
TE            304.2 K
D1            1.00000000 sec
D11           1
D10           1

===== CHANNEL f1 =====
NUC1          1H
P1            10.80 usec
PL1           3.00 dB
SFO1          300.1324010 MHz
SI            32768
SF            300.1300101 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

¹³C NMR of compound **15**



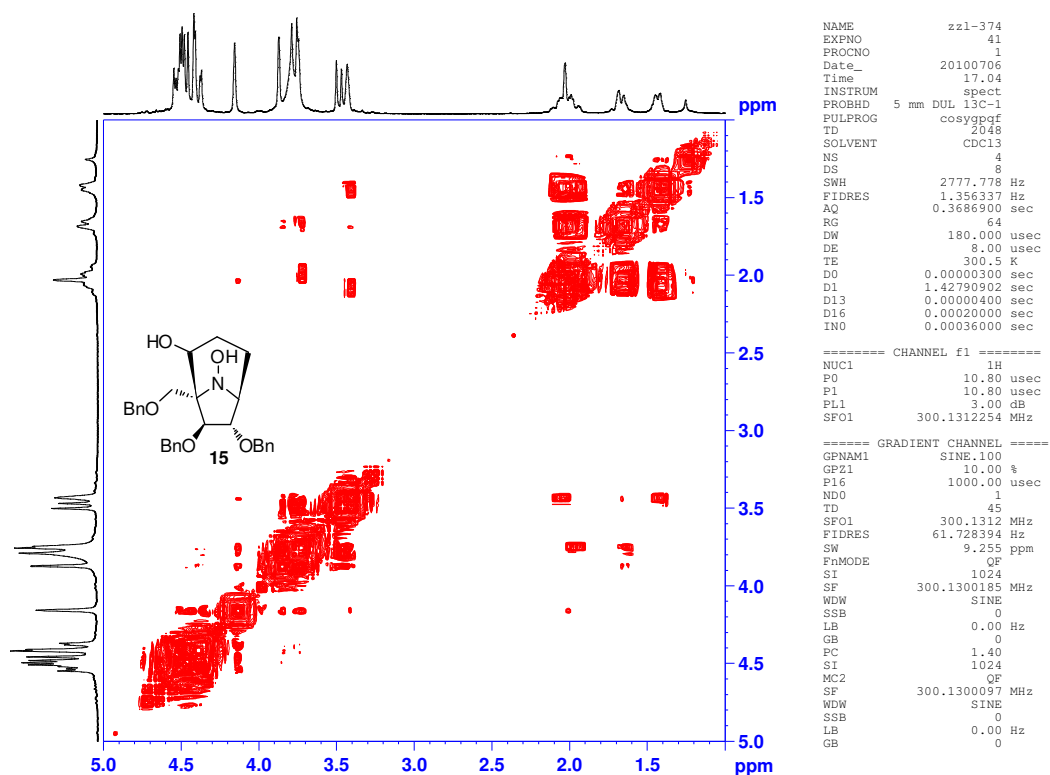
```

NAME          zz1-372
EXPNO         22
PROCNO        1
Date_         20100525
Time          7.40
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            4
DS            4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG            9195.2
DW            27.800 usec
DE            8.00 usec
TE            304.3 K
D1            2.00000000 sec
D11           0.03000000 sec
D10           1

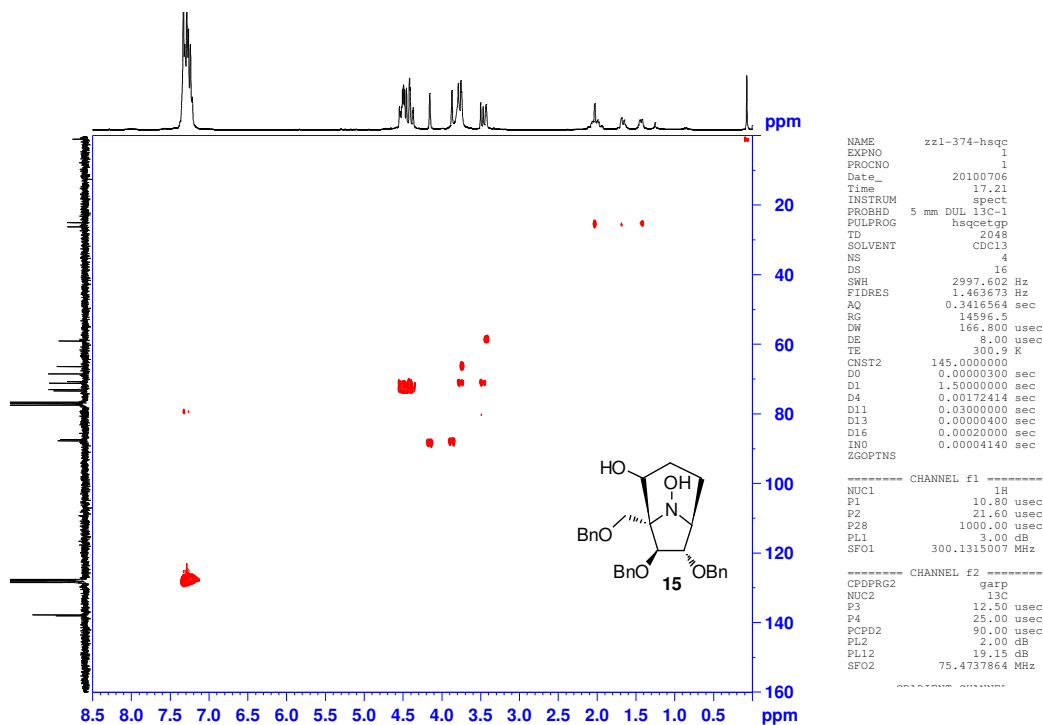
===== CHANNEL f1 =====
NUC1          13C
P1            12.50 usec
PL1           2.00 dB
SFO1          75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         100.00 usec
PL2           3.00 dB
PL12          22.33 dB
PL13          23.00 dB
SFO2          300.1312005 MHz
SI            32768
SF            75.4677490 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

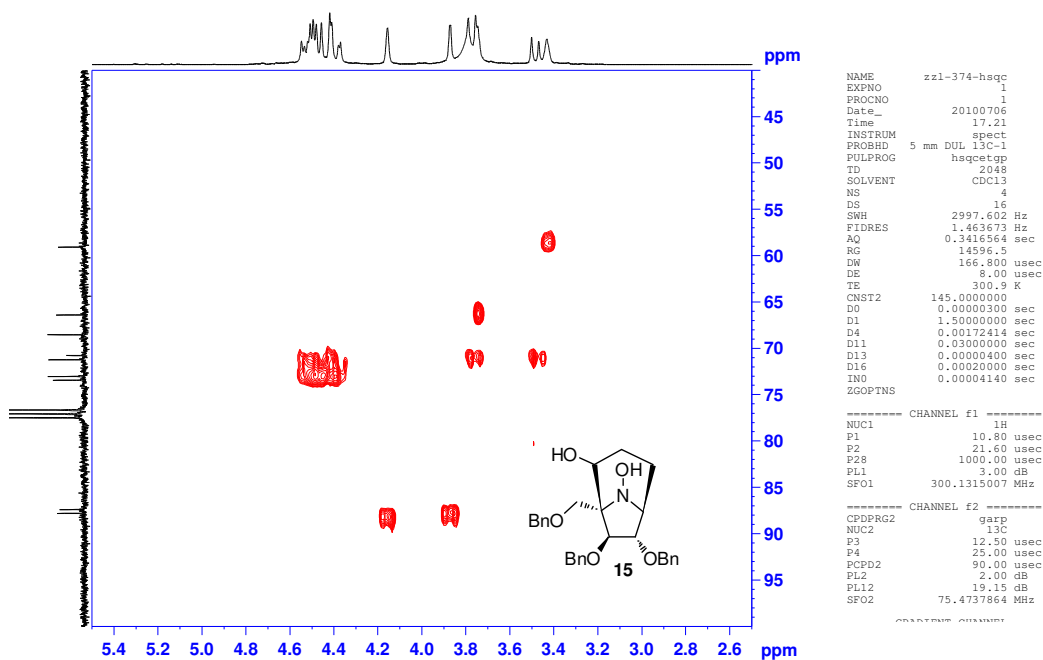
COSY of compound **15**



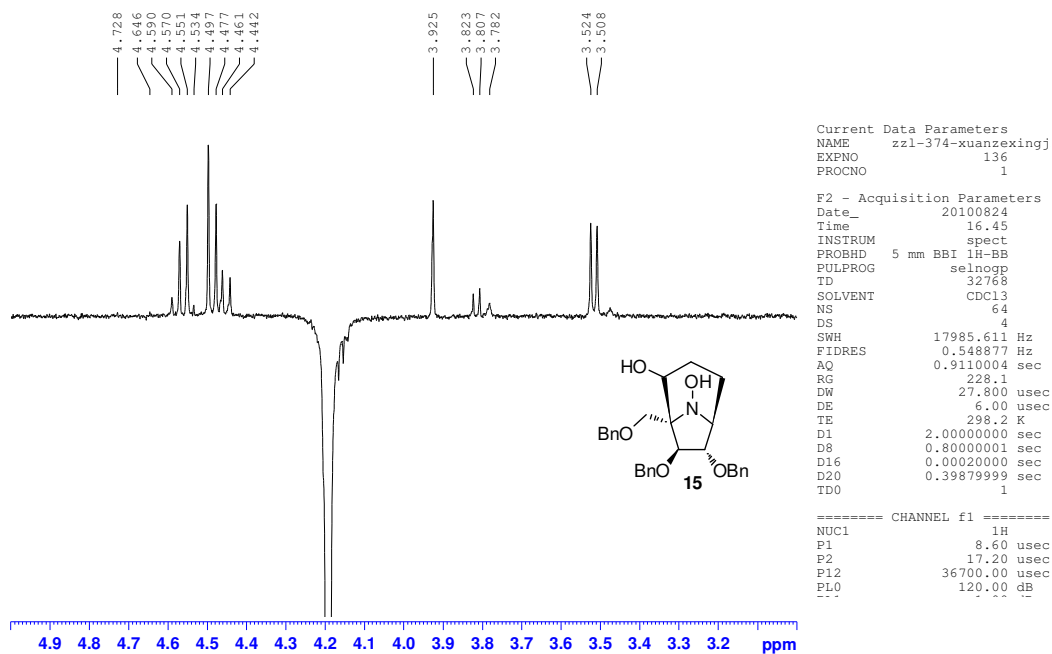
HSQC of compound **15**



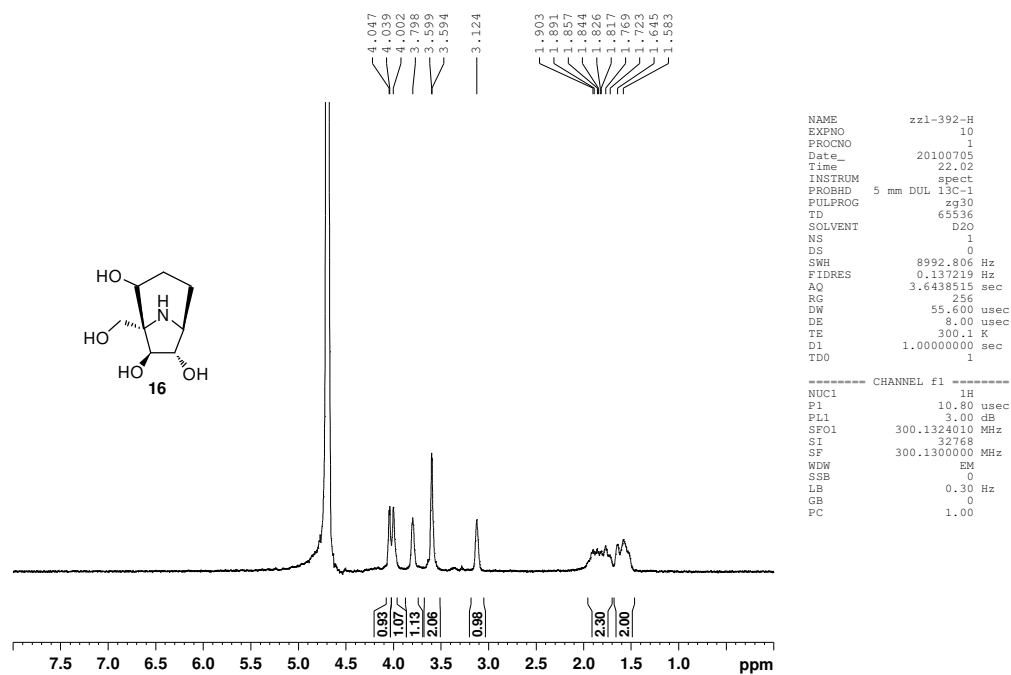
HSQC of compound **15**



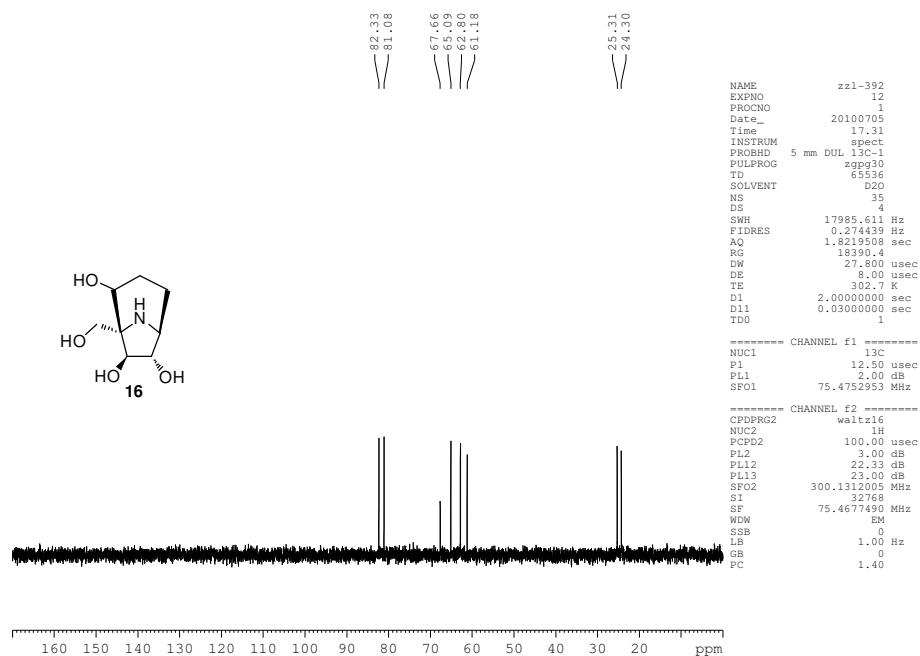
NOE of compound **15**



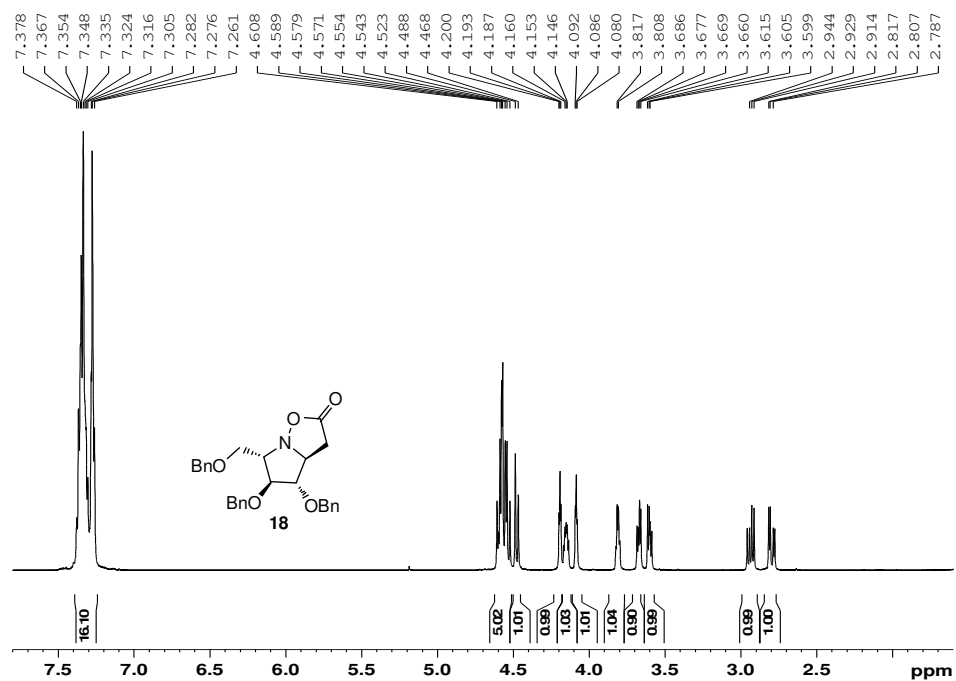
¹H NMR of compound **16**



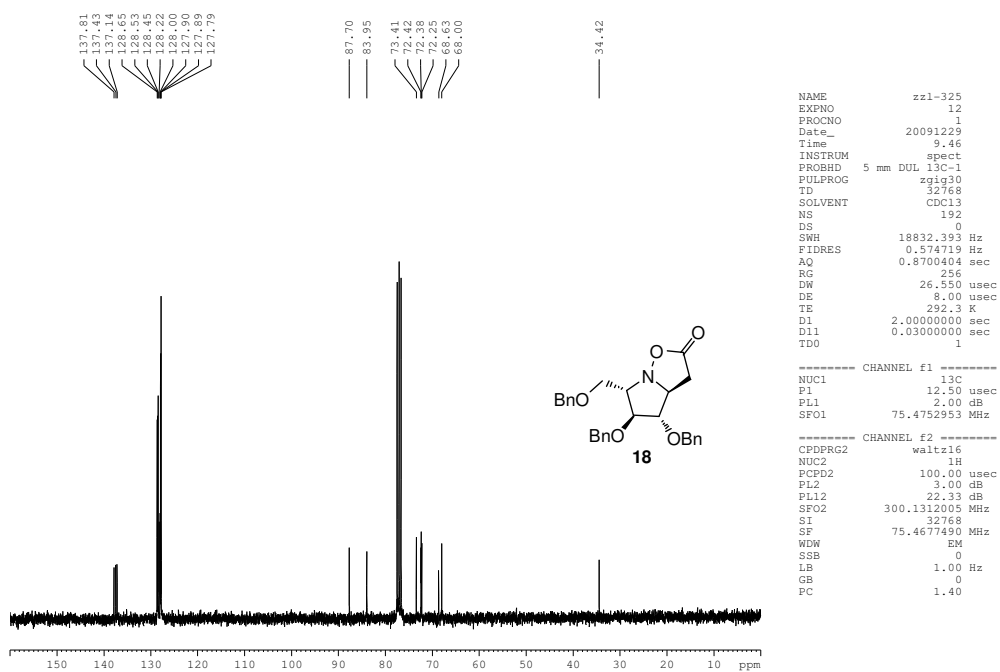
¹³C NMR of compound **16**



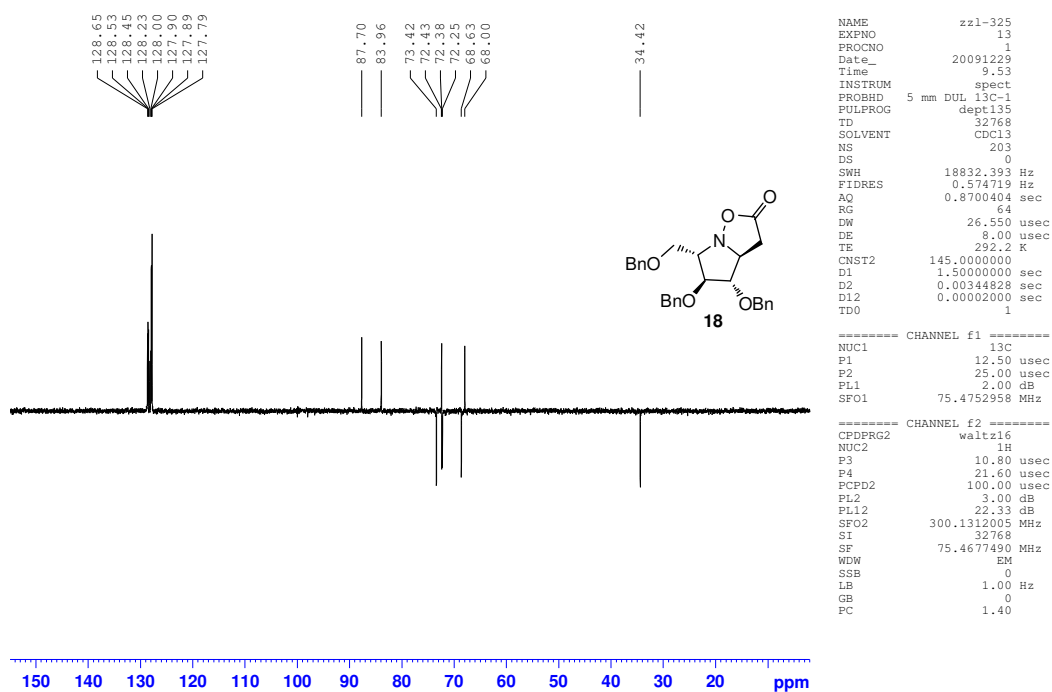
¹H NMR of compound **18**



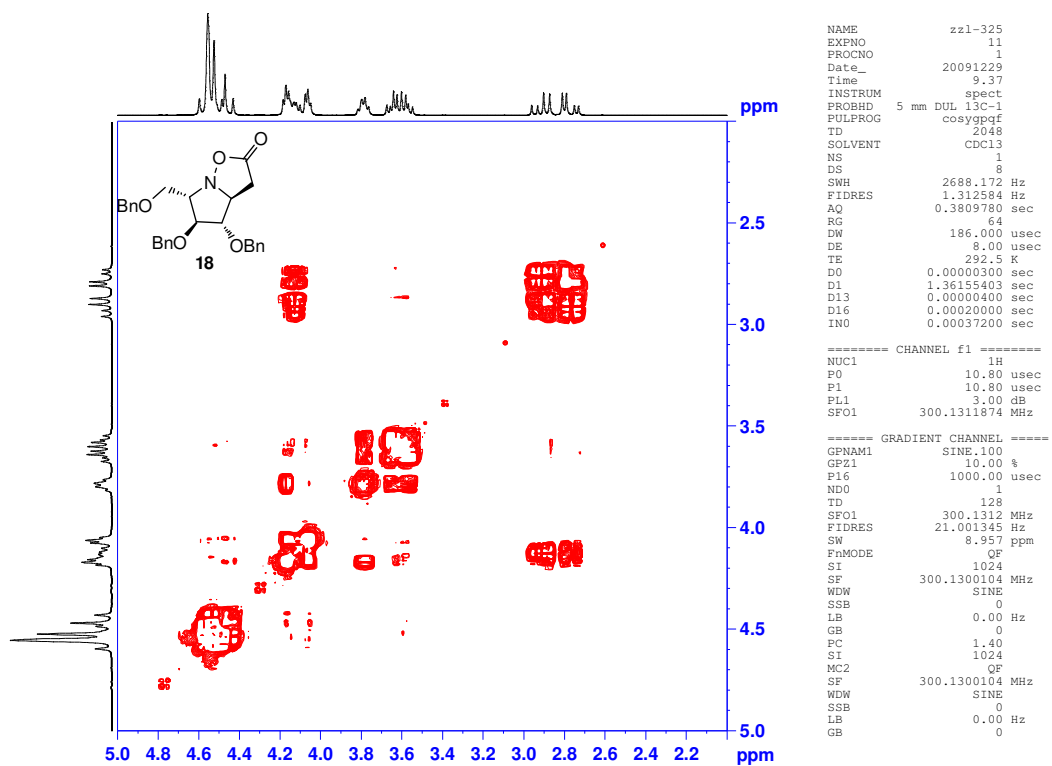
¹³C NMR of compound **18**



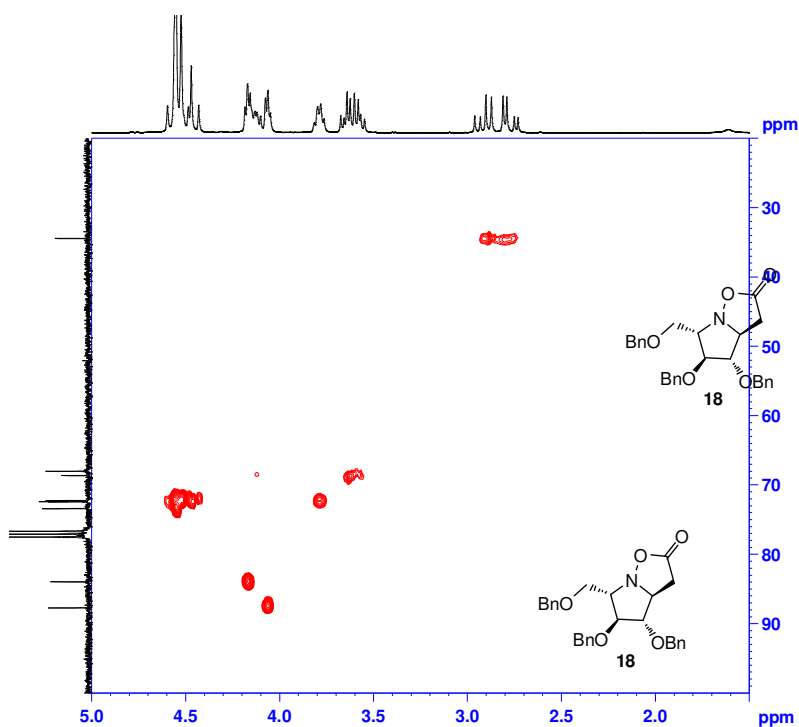
DEPT135 of compound 18



COSY of compound 18



HSQC of compound **18**



```

NAME          zzl-325-hsqc
EXPNO         20
PROCNO        1
Date_         20091229
Time          17.04
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       hsqcetgp
TD            2048
SOLVENT       CDCl3
NS            4
DS            16
SWH           2997.602
FIDRES        1.463673
AQ            0.3416564
RG            18390.4
DW            166.800
DE            8.00
TE            292.8
CNST2         145.0000000
D0            0.00000300
D1            1.50000000
D4            0.00172414
D11           0.03000000
D13           0.00000400
D16           0.00020000
IN0           0.00003310
ZGPTNS
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            10.80
P2            21.60
P28           1000.00
PL1           3.00
SFO1          300.1315007
  
```

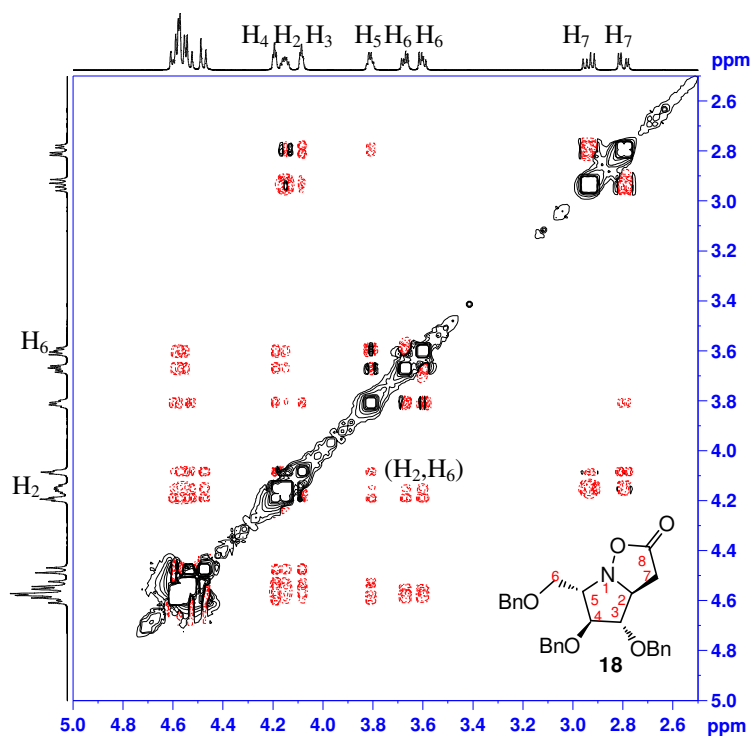
```

===== CHANNEL f2 =====
CPDPRG2       garp
NUC2          13C
P3            12.50
P4            25.00
PCPD2         90.00
PL2           2.00
PL12          19.15
SFO2          75.4752958
  
```

```

===== GRADIENT CHANNEL =====
  
```

NOESY of compound **18**



```

Current Data Parameters
NAME          zzl-325-NOE
EXPNO         9
PROCNO        1
  
```

```

F2 - Acquisition Parameters
Date_         20091230
Time          9.23
INSTRUM       spect
PROBHD        5 mm BBI 1H-BB
PULPROG       noesygpphph
TD            2048
SOLVENT       CDCl3
NS            4
DS            16
SWH           5387.931 Hz
FIDRES        2.630826 Hz
AQ            0.1901044 sec
RG            181
DW            92.800 usec
DE            6.50 usec
TE            298.1 K
D0            0.00008185 sec
D1            1.50000000 sec
D8            0.80000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00018560 sec
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            8.60 usec
P2            17.20 usec
P17           0.10 usec
PL1           -1.00 dB
PL10          9.85 dB
PL1W          31.62277603 W
PL10W         2.60015941 W
SFO1          600.1324005 MHz
  
```

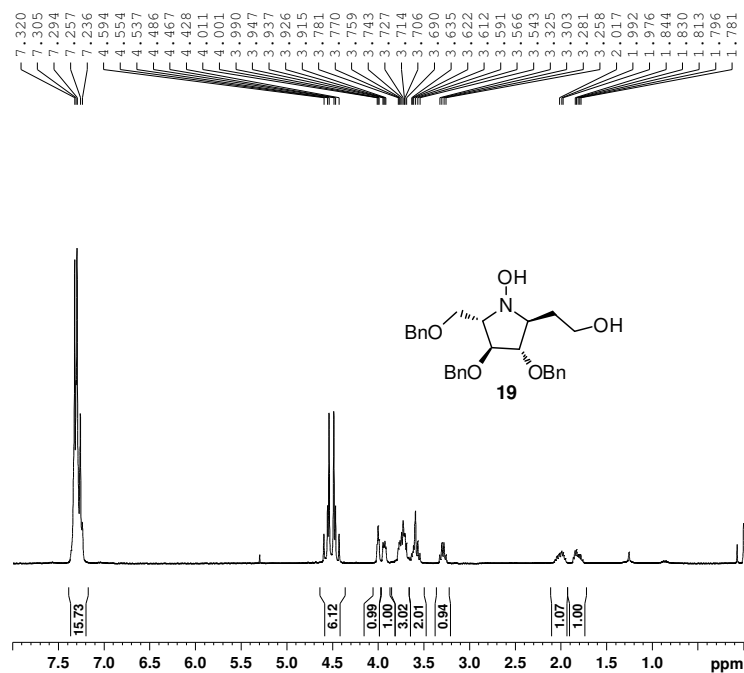
```

===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPZ1          40.00 %
GPZ2          -40.00 %
P16           1000.00 usec
  
```

```

F1 - Acquisition Parameters
  
```

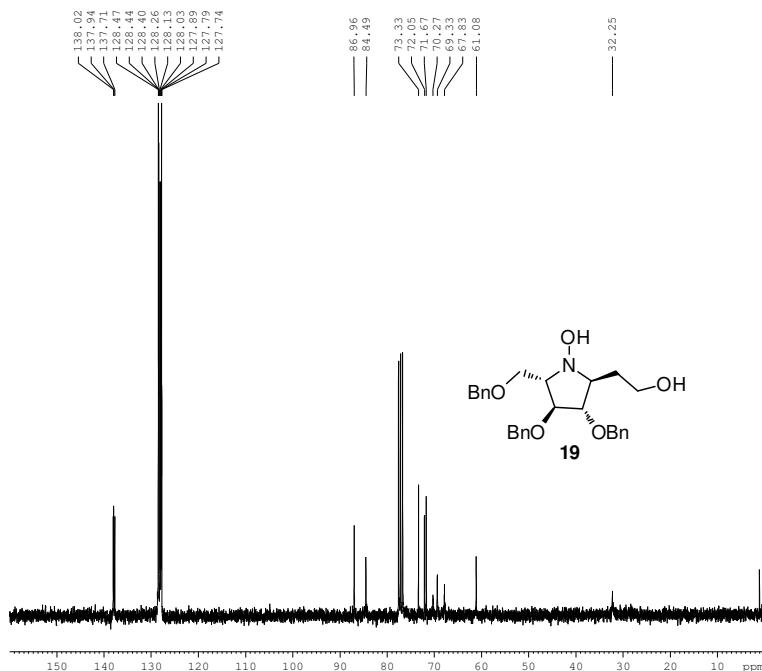
¹H NMR of compound **19**



```

NAME          zz1-326
EXPNO         10
PROCNO        1
Date_         20091230
Time          17.14
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG            228.1
DW            81.000 usec
DE            8.00 usec
TE            293.6 K
D1            1.00000000 sec
D10           1
===== CHANNEL f1 =====
NUC1          1H
P1            10.80 usec
PL1           3.00 dB
SFO1          300.1318534 MHz
SI            32768
SF            300.1300067 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

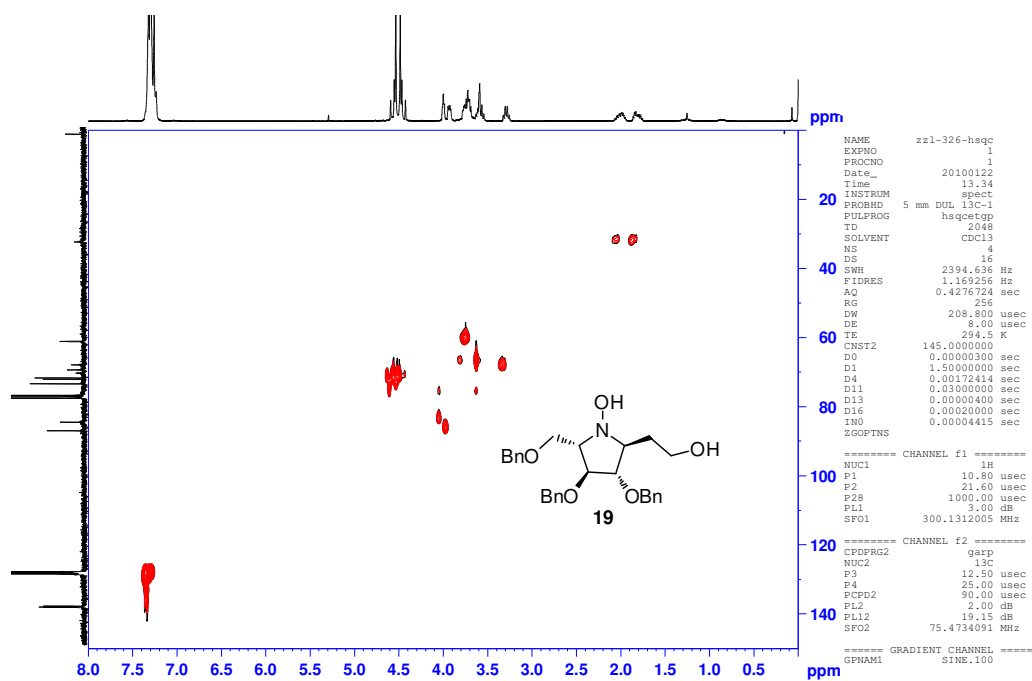
¹³C NMR of compound **19**



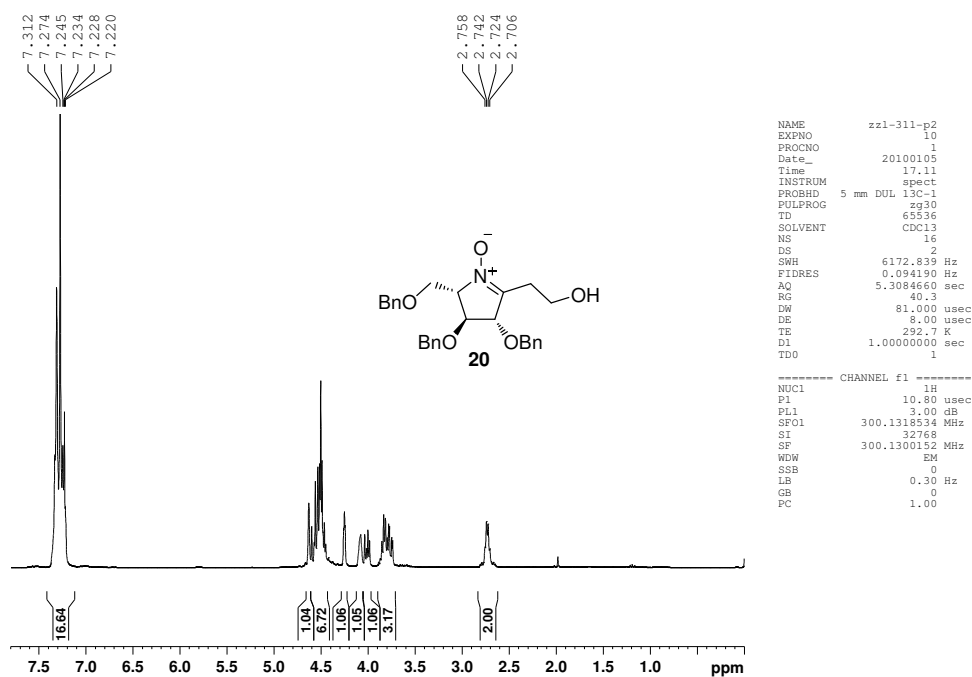
```

NAME          zz1-326
EXPNO         12
PROCNO        1
Date_         20100122
Time          13.16
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg1g30
TD            32768
SOLVENT       CDCl3
NS            256
DS            0
SWH           18832.393 Hz
FIDRES        0.574719 Hz
AQ            0.8700404 sec
RG            5160.6
DW            26.550 usec
DE            8.00 usec
TE            294.2 K
D1            2.00000000 sec
D11           0.03000000 sec
D10           1
===== CHANNEL f1 =====
NUC1          13C
P1            12.50 usec
PL1           2.00 dB
SFO1          75.4752953 MHz
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         100.00 usec
PL2           3.00 dB
PL12          22.33 dB
SFO2          300.1312005 MHz
SI            32768
SF            75.4677490 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

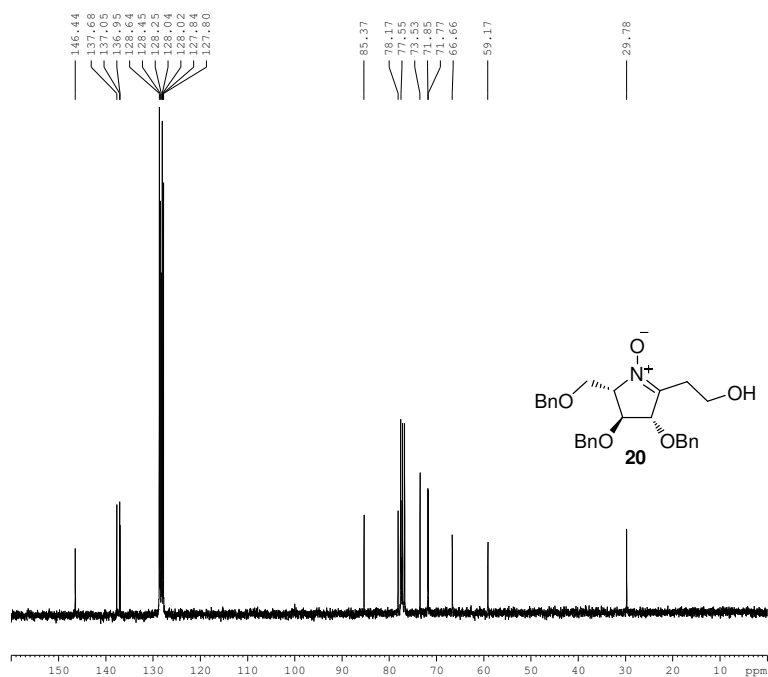
HSQC of compound **19**



¹H NMR of compound **20**



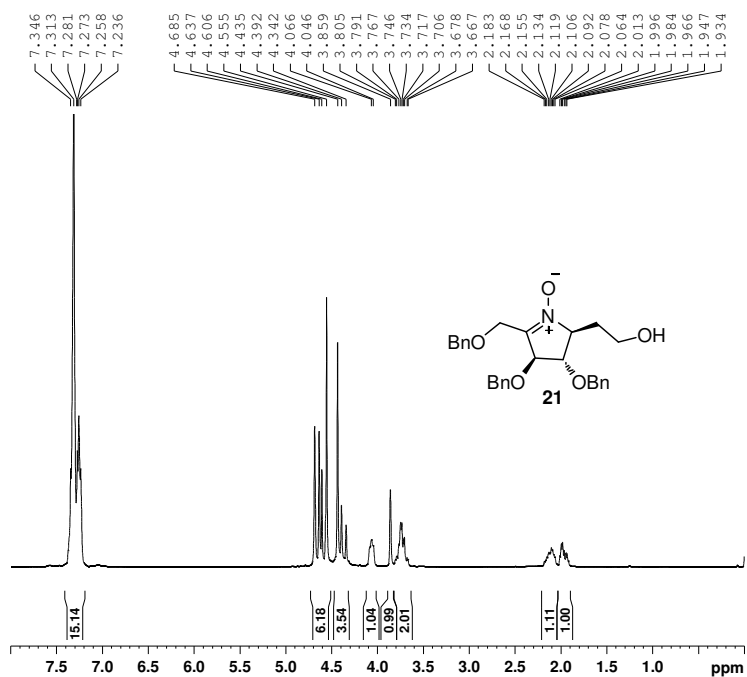
¹³C NMR of compound **20**



NAME zsl-311-p2
EXPNO 12
PROCNO 1
Date_ 20100105
Time 17.19
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 152
DS 0
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 256
DW 26.550 usec
DE 8.00 usec
TE 293.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz
----- CHANNEL f2 -----
CPDPRG2 waitz16
NUC2 1H
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

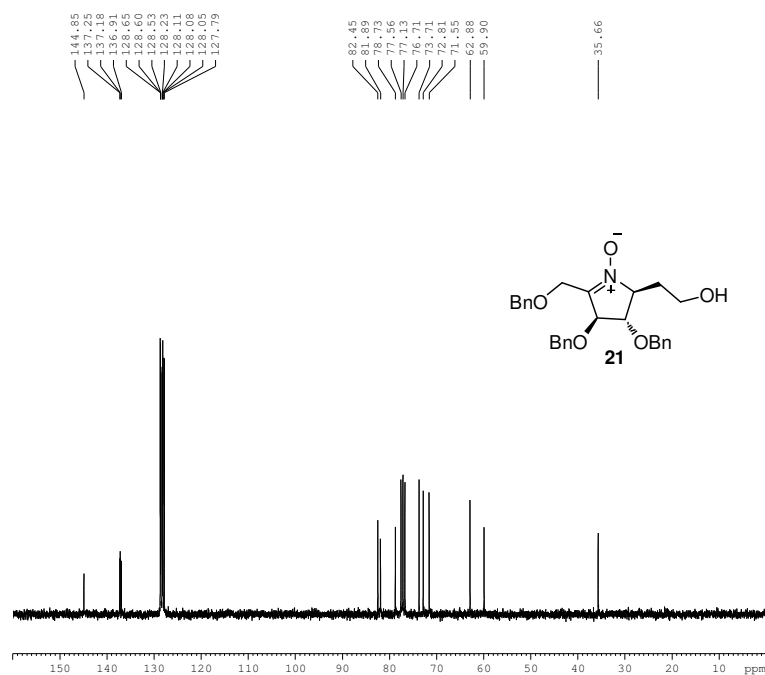
¹H NMR of compound **21**



NAME zsl-311-p1
EXPNO 10
PROCNO 1
Date_ 20091214
Time 9.41
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 71.8
DW 81.000 usec
DE 8.00 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300131 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR of compound **21**



```

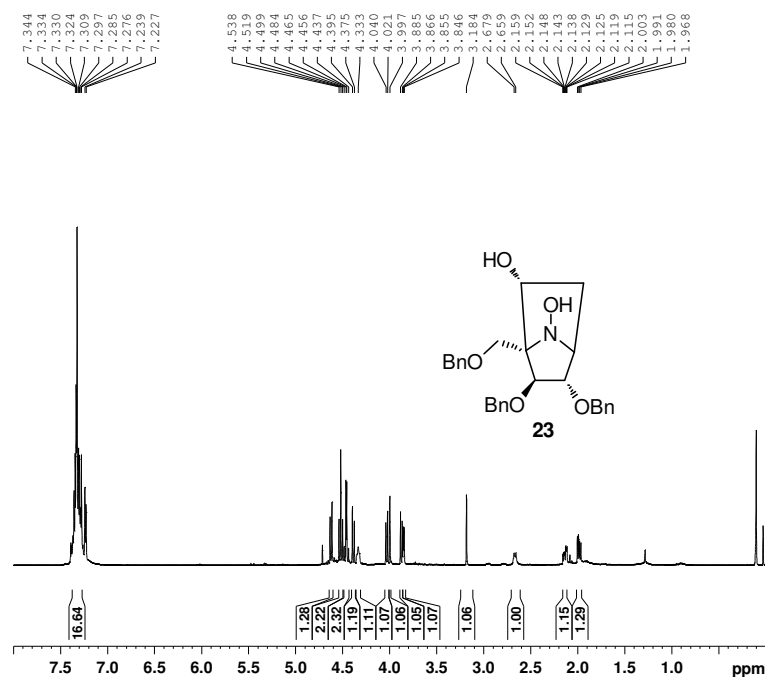
Name      zzi-311-p1
EXPNO     12
PROCNO    1
Date_     20091214
Time      9.48
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         61
DS         4
SWH        17985.611 Hz
FIDRES     0.274439 Hz
AQ         1.8219508 sec
RG         16384
DW         27.800 usec
DE         8.00 usec
TE         295.4 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        12.50 usec
PL1       2.00 dB
SFO1      75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPDZ     100.00 usec
PL2       3.00 dB
PL12      22.33 dB
PL13      23.00 dB
SFO2      300.1312005 MHz
SI        32768
SF         75.4677490 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

¹H NMR of compound **23**



```

Current Data Parameters
NAME      zzi-367-noe
EXPNO     571
PROCNO    1

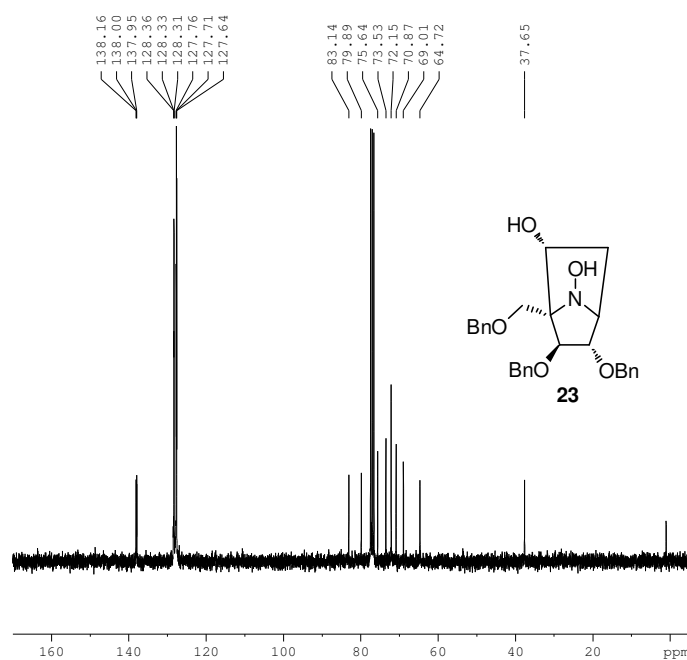
F2 - Acquisition Parameters
Date_     20100520
Time      8.06
INSTRUM   spect
PROBHD    5 mm BBI 1H-BB
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         0
SWH        17985.611 Hz
FIDRES     0.274439 Hz
AQ         1.8219508 sec
RG         57
DW         27.800 usec
DE         6.00 usec
TE         298.2 K
D1         2.0000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        8.60 usec
PL1       -1.00 dB
PL1W      31.62277603 W
SFO1      600.1348010 MHz

F2 - Processing parameters
SI        32768
SF         600.1300000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

¹³C NMR of compound **23**



```

NAME      zz1-367
EXPNO     42
PROCNO    1
Date_     20100517
Time      17.11
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         134
DS         4
SWH        17985.614 Hz
FIDRES     0.274439 Hz
AQ         1.8219508 sec
RG         5792.6
DM         27.800 usec
DE         8.00 usec
TE         299.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

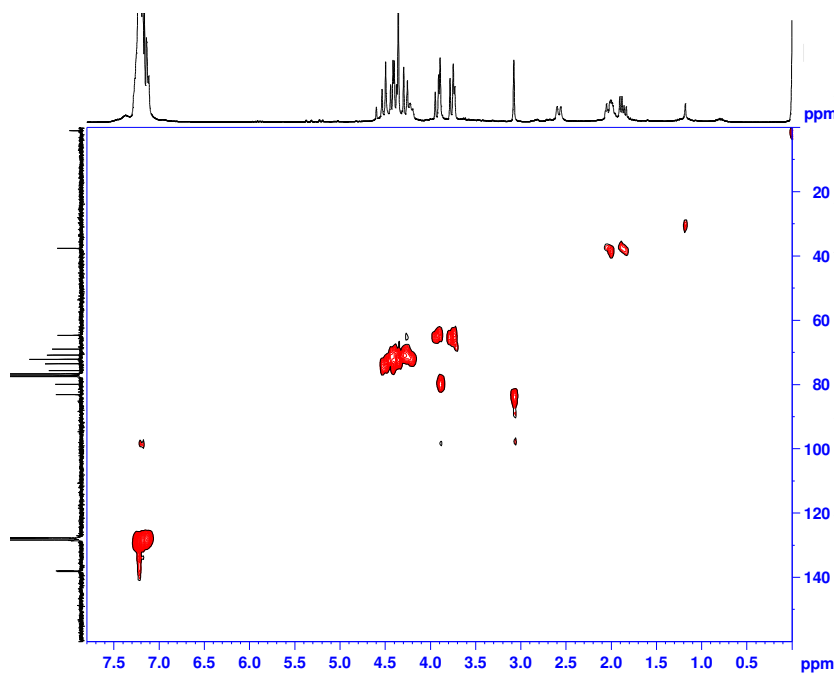
```

===== CHANNEL f1 =====
NUC1      13C
P1        12.50 usec
PL1       2.00 dB
SFO1      75.4752953 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     100.00 usec
PL2        3.00 dB
PL12       22.33 dB
PL13       23.00 dB
SFO2      300.1312005 MHz
SI         32768
SF        75.4677490 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

HSQC of compound **23**



```

NAME      zz1-367-hsqc
EXPNO     1
PROCNO    1
Date_     20100518
Time      8.54
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   hsqcetgpg
TD         2048
SOLVENT   CDCl3
NS         4
DS         16
SWH        5995.204 Hz
FIDRES     2.927346 Hz
AQ         0.1708532 sec
RG         18390.4
DM         83.400 usec
DE         8.00 usec
TE         299.1 K
CNST2     145.0000000
D0         0.00000300 sec
D1         1.50000000 sec
D4         0.00172414 sec
D11        0.03000000 sec
D13        0.00000400 sec
D16        0.00020000 sec
INO        0.00002650 sec
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        10.80 usec
P2        21.60 usec
P28       1000.00 usec
PL1        3.00 dB
SFO1      300.1324010 MHz
  
```

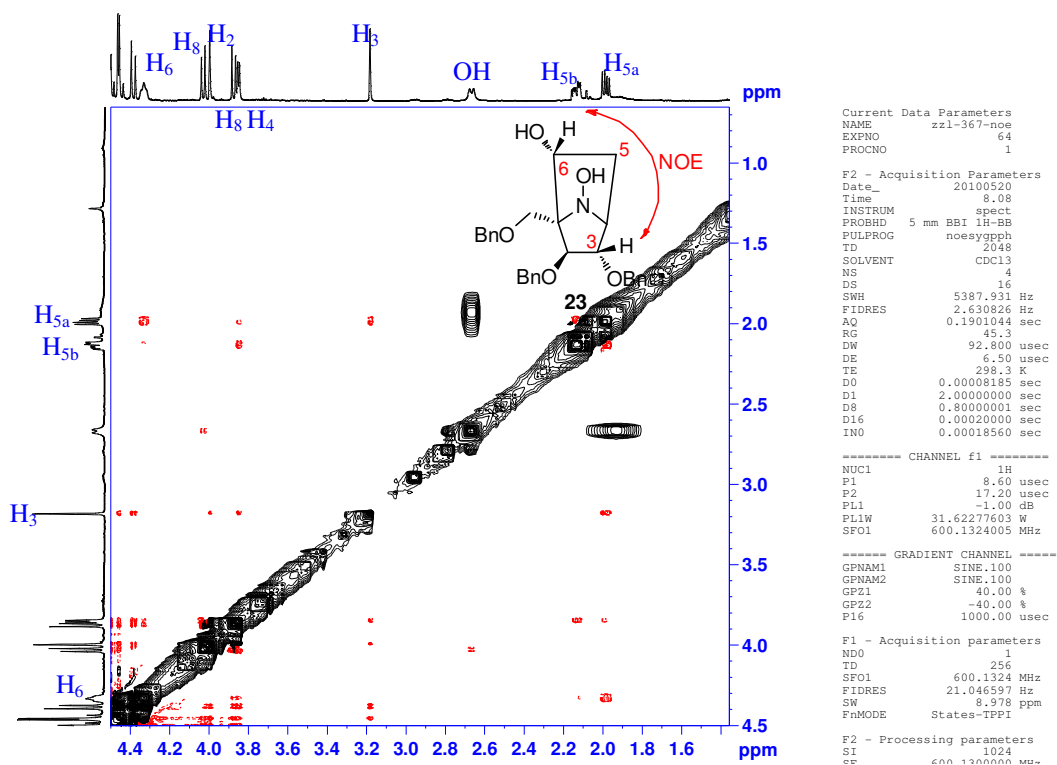
```

===== CHANNEL f2 =====
CPDPRG2   garp
NUC2      13C
P3        12.50 usec
P4        25.00 usec
PCPD2     90.00 usec
PL2        2.00 dB
PL12       19.15 dB
SFO2      75.4752958 MHz
  
```

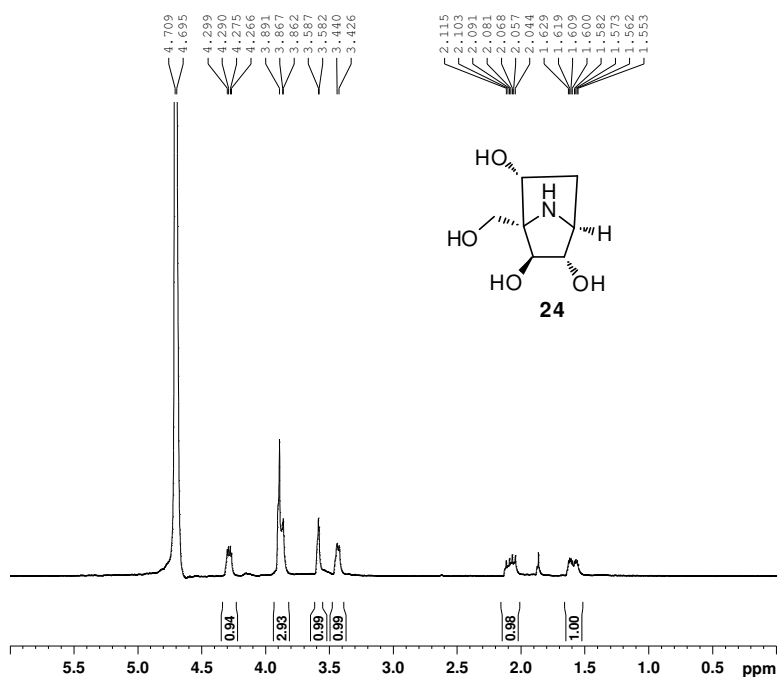
```

===== GRADIENT CHANNEL =====
GPNAM1    SINE.100
GPNAM2    SINE.100
GP21      80.00 %
GP22      20.10 %
P16       1000.00 usec
ND0        2
  
```

NOESY of compound 23



¹H NMR of compound **24**

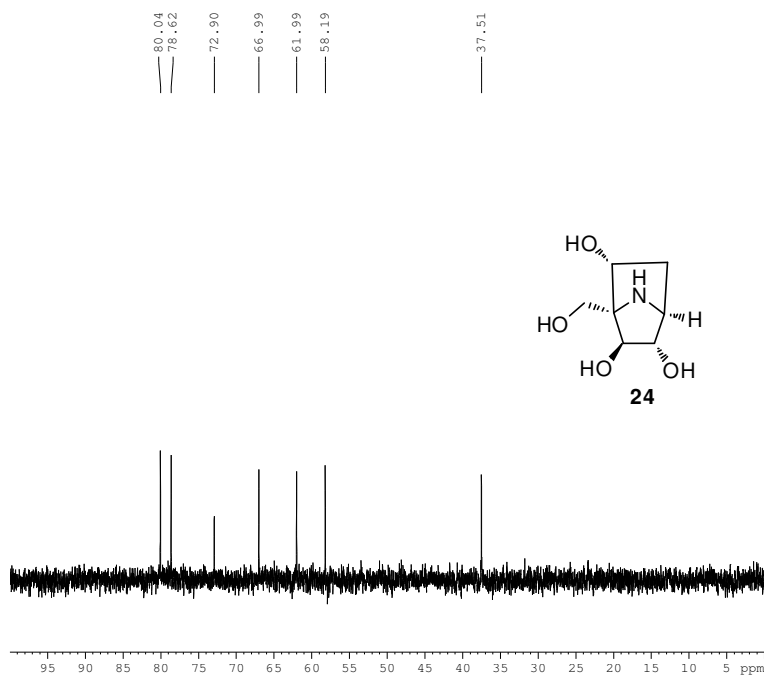


```

NAME      zz1-394-freebase
EXPNO     1
PROCNO    1
Date_     20100909
Time      15.23
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zg30
TD        65536
SOLVENT   D2O
NS         2
DS         0
SWH        8992.806 Hz
FIDRES     0.137219 Hz
AQ         3.6438515 sec
RG         228.1
DW         55.600 usec
DE         8.00 usec
TE         301.4 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         10.80 usec
PL1        3.00 dB
SFO1       300.1324010 MHz
SI         32768
SF         300.1300000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

¹³C NMR of compound **24**



```

NAME      zz1-394-freebase
EXPNO     10
PROCNO    1
Date_     20100909
Time      15.14
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zgpg30
TD        65536
SOLVENT   D2O
NS         144
DS         4
SWH        17985.611 Hz
FIDRES     0.274439 Hz
AQ         1.8219508 sec
RG         16384
DW         27.800 usec
DE         8.00 usec
TE         301.3 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         12.50 usec
PL1        2.00 dB
SFO1       75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2      100.00 usec
PL2         3.00 dB
PL12       22.33 dB
PL13       23.00 dB
SFO2       300.1312005 MHz
SI         32768
SF         75.4677490 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```