

# Concise Synthesis of Calystegine B<sub>2</sub> and B<sub>3</sub> via Intramolecular Nozaki–Hiyama–Kishi Reaction

Hong-Yao Wang,<sup>a,e</sup> Atsushi Kato,<sup>\*b</sup> Kyoko Kinami,<sup>b</sup> Yi-Xian Li,<sup>a</sup> George W. J. Fleet,<sup>c,d</sup> and Chu-Yi Yu<sup>\*a,d</sup>

---

<sup>a</sup>Beijing National Laboratory for Molecular Science (BNLMS), CAS Key Laboratory of Molecular Recognition and Function, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, China. Email: yucy@iccas.ac.cn.

<sup>b</sup>Department of Hospital Pharmacy, University of Toyama, 2630 Sugitani, Toyama 930-0194, Japan. E-mail: kato@med.u-toyama.ac.jp.

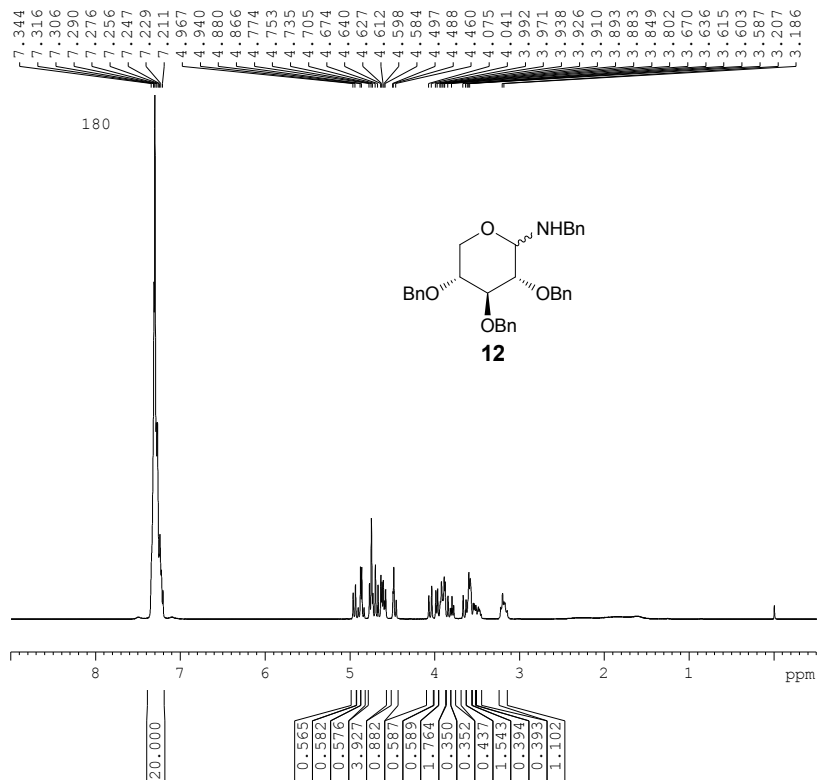
<sup>c</sup>Chemistry Research Laboratory, Department of Chemistry, University of Oxford, Mansfield Road, Oxford, OX1 3TA, UK.

<sup>d</sup>National Engineering Research Center for Carbohydrate Synthesis, Jiangxi Normal University, Nanchang 330022, PR China

<sup>e</sup>University of Chinese Academy of Sciences, Beijing 100049, China.

<b>1. Copies of <sup>1</sup>H, <sup>13</sup>C and 2D NMR spectra .....</b>	<b>2</b>
<b>2. Results of the bioassay .....</b>	<b>32</b>
<b>3. Comparison of <sup>13</sup>C NMR of synthetic calystegine B<sub>2</sub> (3) and B<sub>3</sub> (4) with natural products.....</b>	<b>33</b>

# 1. Copies of $^1\text{H}$ , $^{13}\text{C}$ and 2D NMR spectra

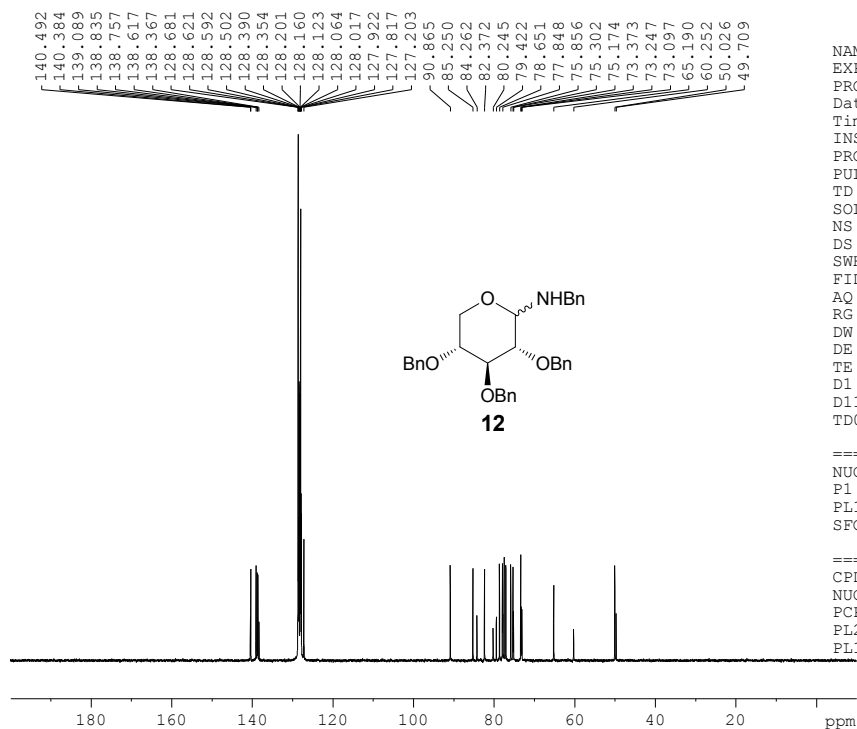


```

NAME          400MHZ
EXPNO         28
PROCNO        1
Date_         20140708
Time_         9.31
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            55.55
DW            62.400 usec
DE            6.50 usec
TE            300.3 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
SFO1          400.2424716 MHz
NUC1           1H
P1            14.80 usec
SI            65536
SF            400.2400291 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



```

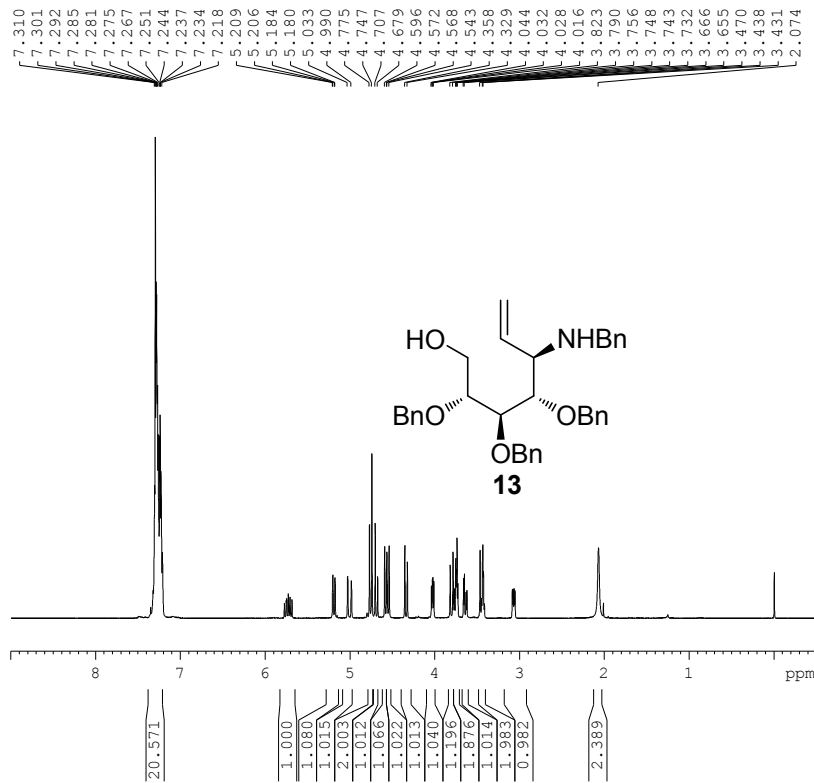
NAME          why-180
EXPNO         10
PROCNO        1
Date_         20140708
Time_         9.23
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            269
DS            4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG            1024
DW            27.800 usec
DE            6.50 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
  
```

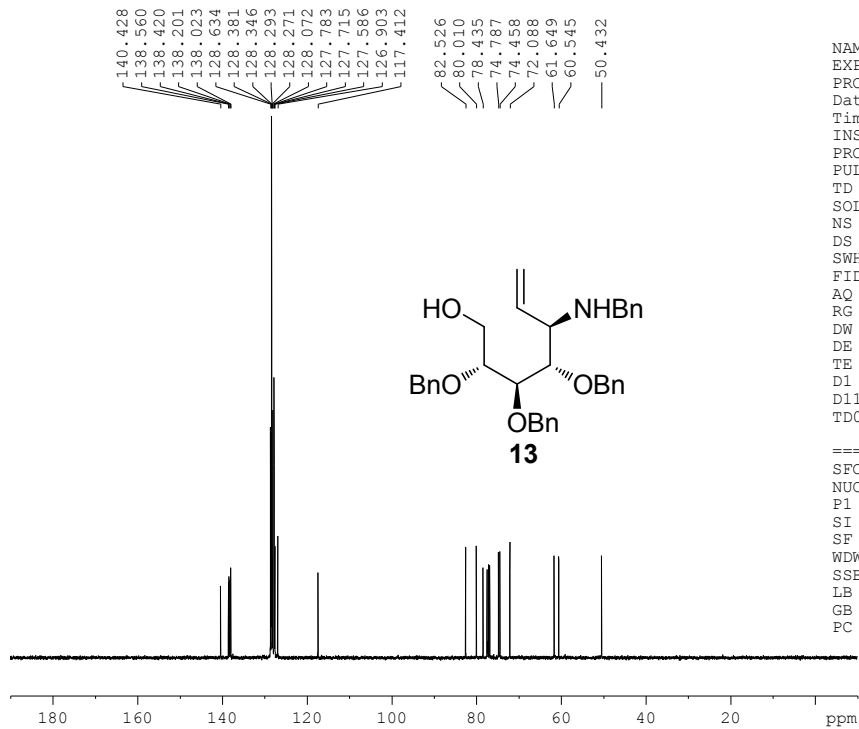
```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         100.00 usec
PL2           3.00 dB
PL12          22.74 dB
  
```



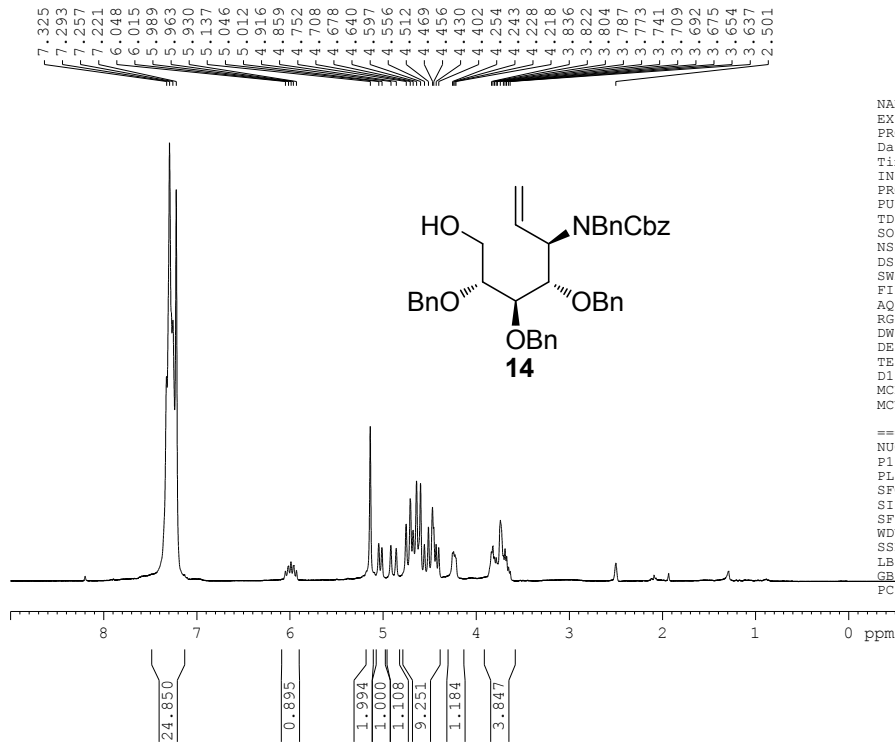
NAME 400MHZ  
EXPNO 449  
PROCNO 1  
Date\_ 20150416  
Time\_ 9.20  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8012.820 Hz  
FIDRES 0.244532 Hz  
AQ 2.0447731 sec  
RG 102.73  
DW 62.400 usec  
DE 6.50 usec  
TE 298.1 K  
D1 2.00000000 sec  
D10 1

===== CHANNEL f1 =====  
SFO1 400.2424716 MHz  
NUC1 1H  
P1 14.80 usec  
SI 65536  
SF 400.2400162 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



NAME 400MHZ  
EXPNO 450  
PROCNO 1  
Date\_ 20150416  
Time\_ 9.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 50  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 206.33  
DW 20.800 usec  
DE 6.50 usec  
TE 298.1 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D10 1

===== CHANNEL f1 =====  
SFO1 100.6504916 MHz  
NUC1 13C  
P1 10.00 usec  
SI 32768  
SF 100.6404406 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

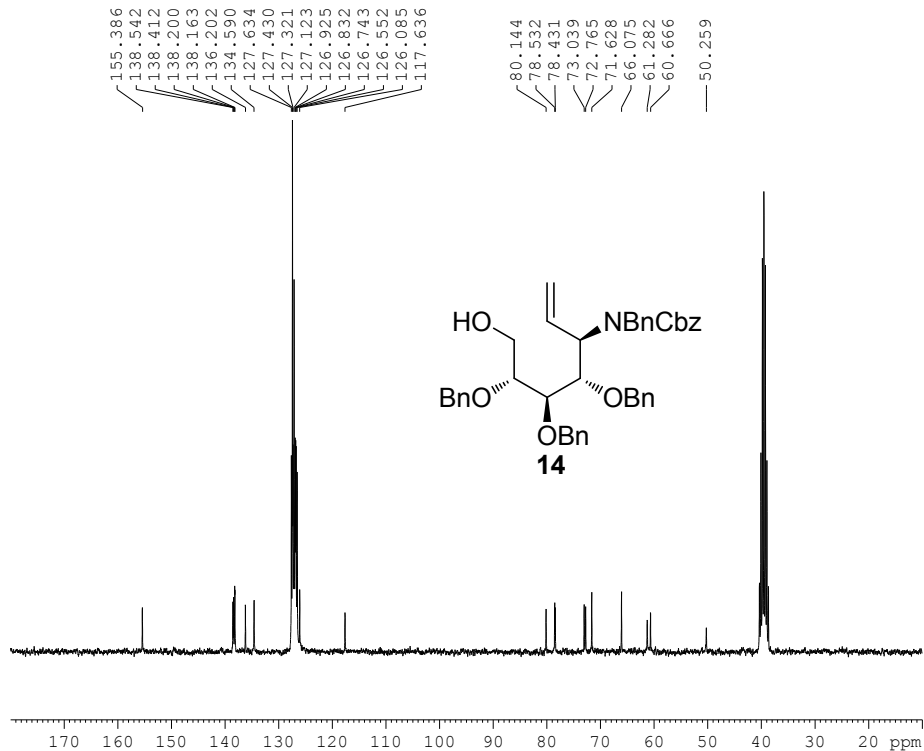


```

NAME          400MHZ
EXPNO         3015
PROCNO        1
Date_         20150320
Time          17.15
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            32
DS            0
SWH           6613.757 Hz
FIDRES        0.201836 Hz
AQ            2.4773109 sec
RG            1024
DW            75.600 usec
DE            6.00 usec
TE            370.0 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1           1H
P1             13.80 usec
PL1            3.00 dB
SFO1          300.1304800 MHz
SI            65536
SF            300.1300019 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             0.20
  
```



```

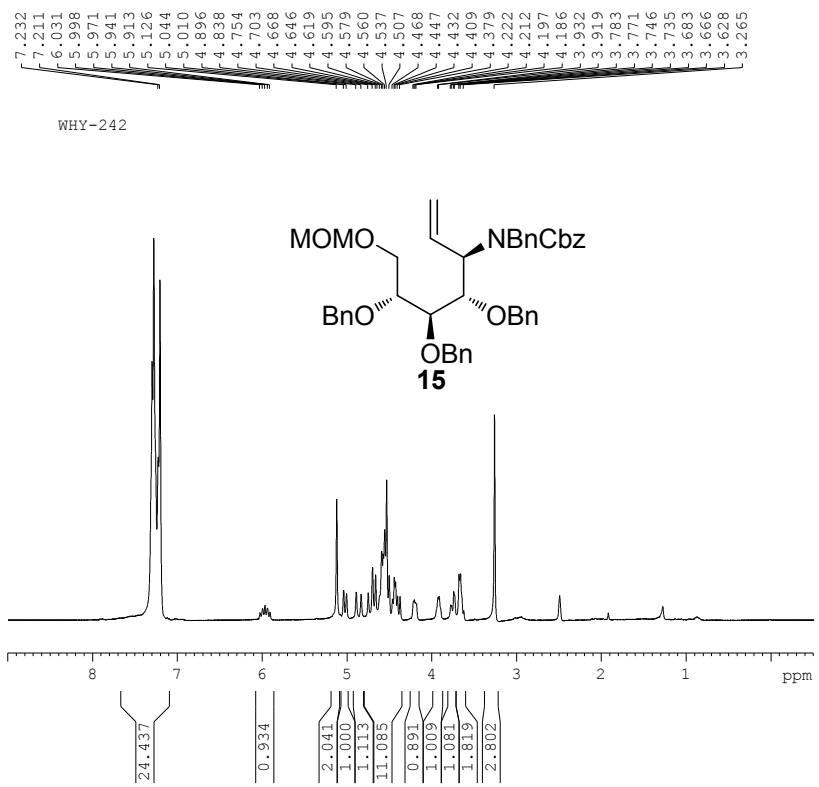
NAME          400MHZ
EXPNO         745
PROCNO        1
Date_         20150320
Time          16.29
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            1156
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ            0.7209460 sec
RG            32768
DW            22.000 usec
DE            6.00 usec
TE            370.3 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1           13C
P1             9.30 usec
PL1            1.00 dB
SFO1          75.4737678 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          18.00 dB
SFO2          300.1312005 MHz
SI            32768
SF            75.4678267 MHz
WDW            EM
SSB            0
LB             3.00 Hz
GB             0
PC             0.10
  
```

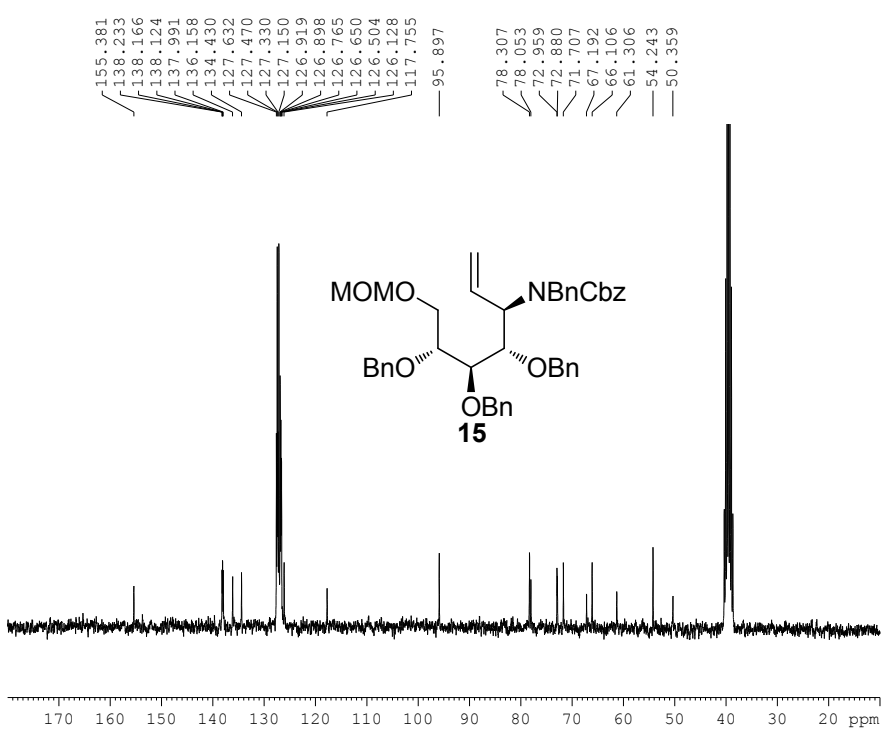


```

NAME          400MHZ
EXPNO         3016
PROCNO        1
Date_         20150320
Time_         17.57
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            20
DS            0
SWH           6613.757 Hz
FIDRES        0.201836 Hz
AQ           2.4773109 sec
RG           1024
DW           75.600 usec
DE           6.00 usec
TE           370.0 K
D1           2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.80 usec
PL1           3.00 dB
SFO1         300.1304800 MHz
SI           65536
SF           300.1300035 MHz
WDW           EM
SSB           0
LB           0.30 Hz
GB           0
  
```



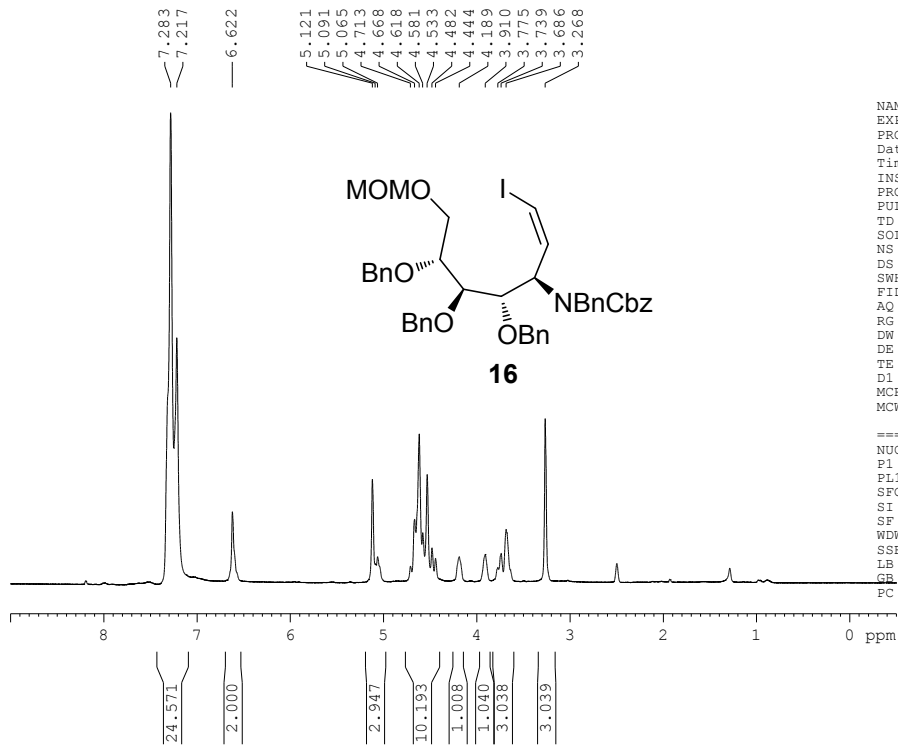
```

NAME          400MHZ
EXPNO         746
PROCNO        1
Date_         20150320
Time_         17.25
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            644
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ           0.7209460 sec
RG           32768
DW           22.000 usec
DE           6.00 usec
TE           376.6 K
D1           2.00000000 sec
d11          0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            9.30 usec
PL1           1.00 dB
SFO1         75.4737678 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          18.00 dB
SFO2         300.1312005 MHz
SI           32768
SF           75.4678267 MHz
WDW           EM
SSB           0
LB           3.00 Hz
GB           0
PC           0.10
  
```

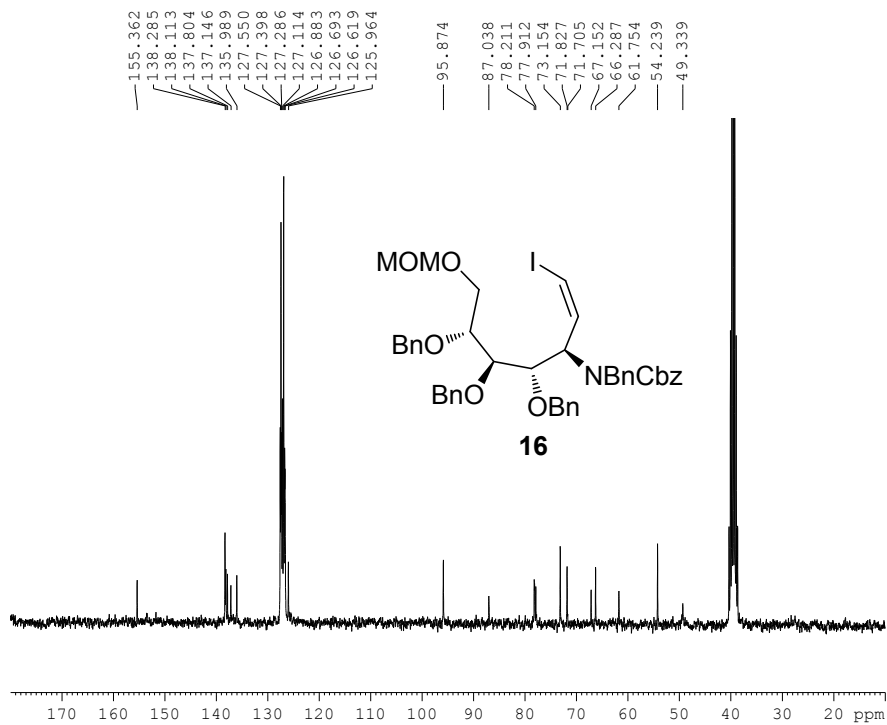


```

NAME          400MHZ
EXPNO         3017
PROCNO        1
Date_         20150324
Time_         10.11
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            16
DS            0
SWH           6613.757 Hz
FIDRES        0.201836 Hz
AQ            2.4773109 sec
RG            1024
DW            75.600 usec
DE            6.00 usec
TE            370.0 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.80 usec
PL1           3.00 dB
SFO1          300.1304800 MHz
SI            65536
SF            300.1298031 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            0.20
  
```



```

NAME          400MHZ
EXPNO         747
PROCNO        1
Date_         20150324
Time_         9.36
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            741
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ            0.7209460 sec
RG            32768
DW            22.000 usec
DE            6.00 usec
TE            373.8 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

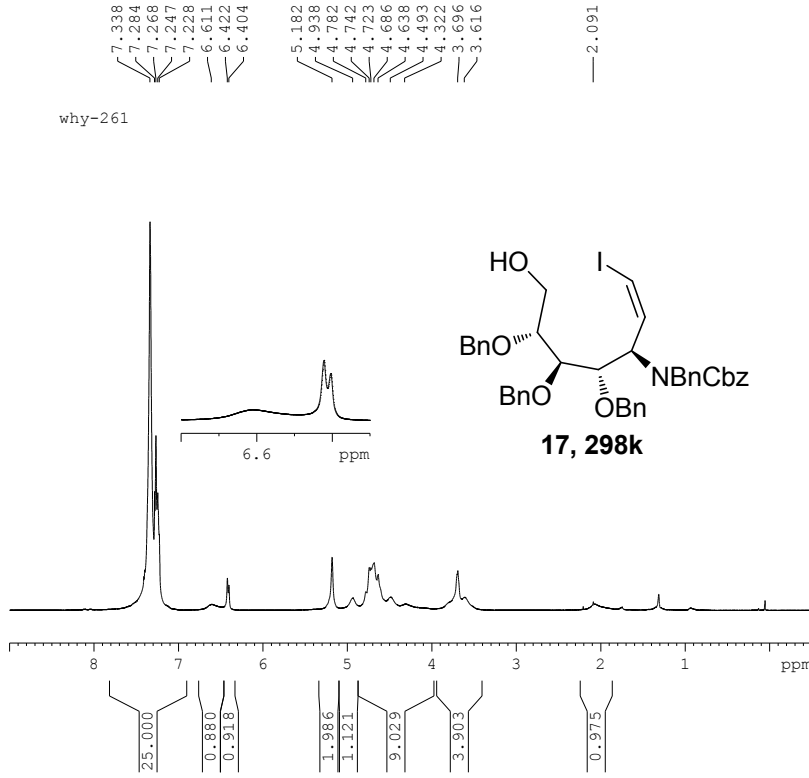
```

===== CHANNEL f1 =====
NUC1          13C
P1            9.30 usec
PL1           1.00 dB
SFO1          75.4737678 MHz
  
```

```

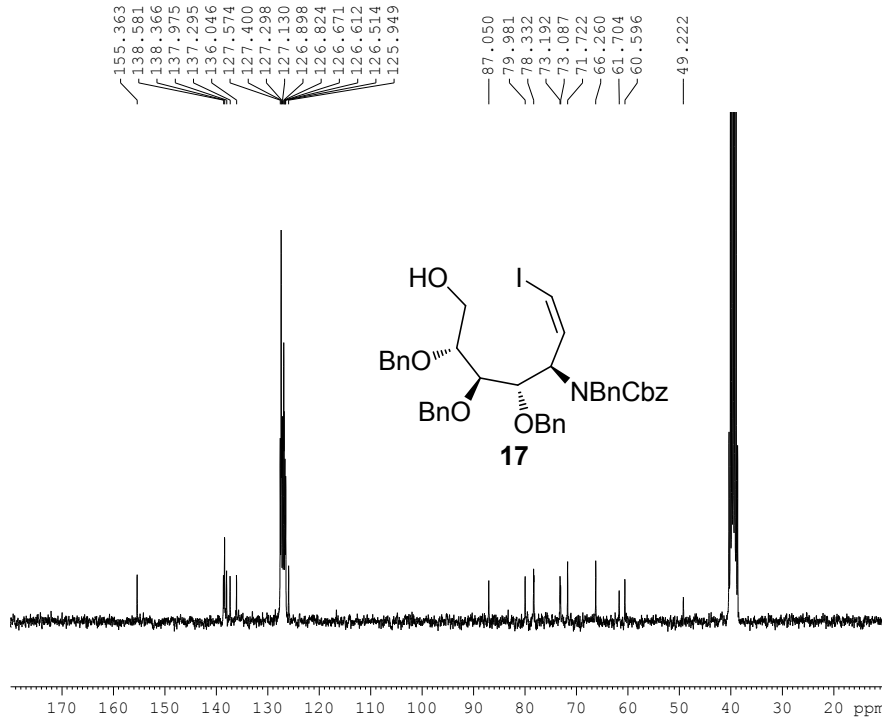
===== CHANNEL f2 =====
CPDPRG2       wallz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          18.00 dB
SFO2          300.1312005 MHz
SI            32768
SF            75.4677786 MHz
WDW           EM
SSB           0
LB            3.00 Hz
GB            0
PC            0.10
  
```

why-261



NAME 400MHZ  
EXPNO 318  
PROCNO 1  
Date\_ 20150109  
Time\_ 12.16  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8012.820 Hz  
FIDRES 0.244532 Hz  
AQ 2.0447731 sec  
RG 34.76  
DW 62.400 usec  
DE 6.50 usec  
TE 295.6 K  
D1 2.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 400.2424716 MHz  
NUC1 1H  
P1 14.80 usec  
SI 65536  
SF 400.2400000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

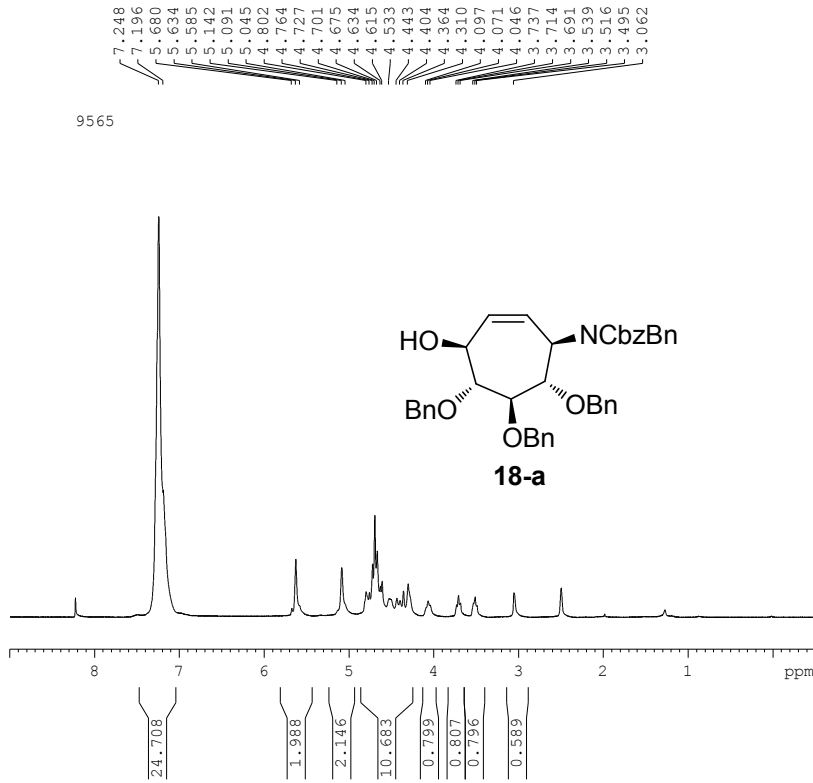


NAME 400MHZ  
EXPNO 748  
PROCNO 1  
Date\_ 20150324  
Time\_ 10.25  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgig30  
TD 32768  
SOLVENT DMSO  
NS 2306  
DS 0  
SWH 22727.273 Hz  
FIDRES 0.693581 Hz  
AQ 0.7209460 sec  
RG 32768  
DW 22.000 usec  
DE 6.00 usec  
TE 370.2 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
MCREST 0.00000000 sec  
MCWRK 0.01500000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.30 usec  
PL1 1.00 dB  
SFO1 75.4737678 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 2.50 dB  
PL12 18.00 dB  
SFO2 300.1312005 MHz  
SI 32768  
SF 75.4677815 MHz  
WDW EM  
SSB 0  
LB 3.00 Hz  
GB 0  
PC 0.10

9565

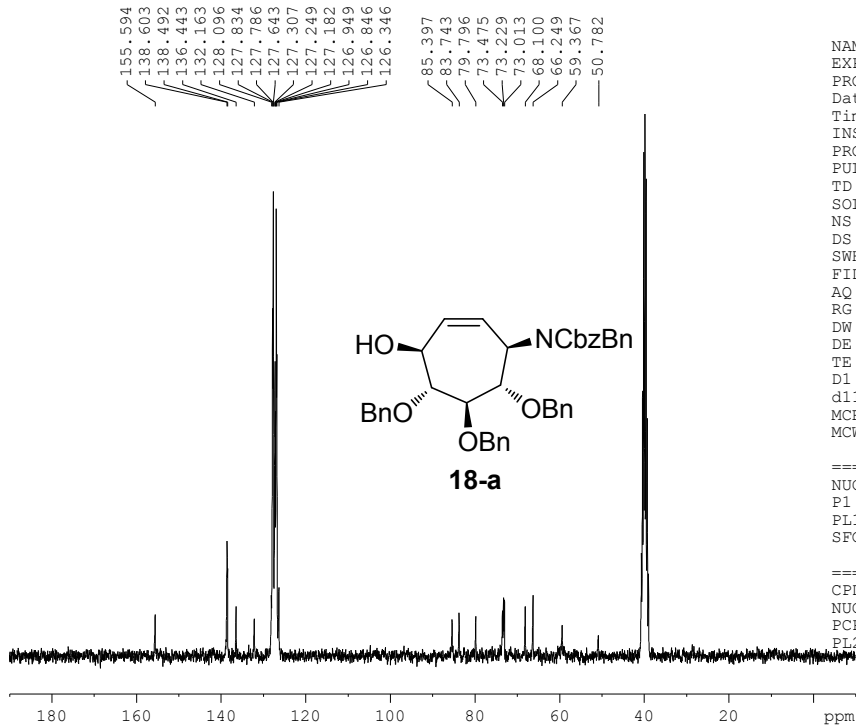


```

NAME          400MHZ
EXPNO         9
PROCNO        1
Date_         20151102
Time_         18.04
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            16
DS            0
SWH           8992.806 Hz
FIDRES        0.274439 Hz
AQ           1.8219508 sec
RG            11.3
DW           55.600 usec
DE            6.50 usec
TE            368.9 K
D1           2.00000000 sec
D10           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            10.50 usec
PL1           0.99 dB
PL1W         19.99861908 W
SFO1         300.1314106 MHz
SI           65536
SF           300.1300016 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
  
```



```

NAME          400MHZ
EXPNO         761
PROCNO        1
Date_         20150605
Time_         9.08
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            951
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ           0.7209460 sec
RG            32768
DW           22.000 usec
DE            6.00 usec
TE            371.0 K
D1           2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

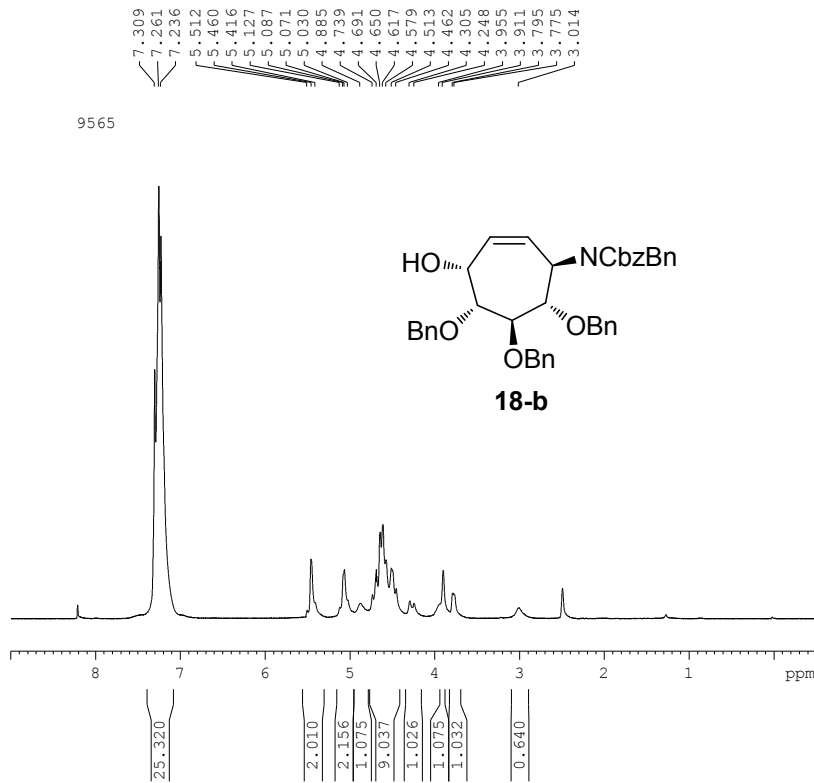
```

===== CHANNEL f1 =====
NUC1          13C
P1            9.30 usec
PL1           1.00 dB
SFO1         75.4737678 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
  
```





```

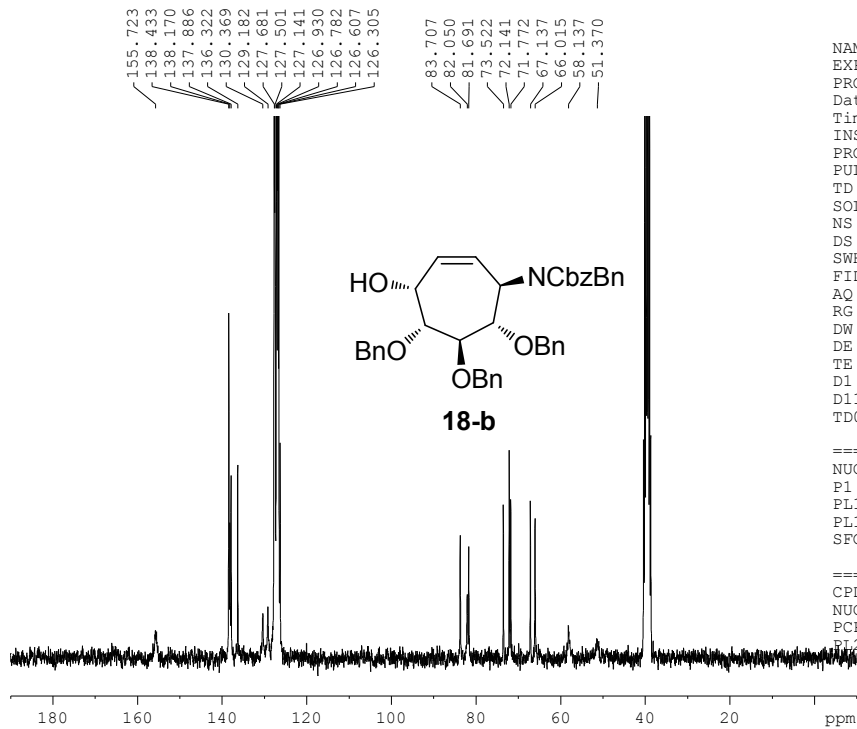
NAME          400MHZ
EXPNO         10
PROCNO        1
Date_         20151102
Time_         18.28
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            16
DS            0
SWH           8992.806 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG            11.3
DW            55.600 usec
DE            6.50 usec
TE            371.0 K
D1            2.00000000 sec
D11           1
TD0           1

```

```

===== CHANNEL f1 =====
NUC1          1H
P1            10.50 usec
PL1           0.99 dB
PL1W         19.99861908 W
SFO1         300.1314106 MHz
SI           65536
SF           300.1300025 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0

```



```

NAME          400MHZ
EXPNO         11
PROCNO        1
Date_         20151109
Time_         14.28
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgig30
TD            32768
SOLVENT       oC6D4Cl2
NS            2427
DS            0
SWH           18832.393 Hz
FIDRES        0.574719 Hz
AQ            0.8700404 sec
RG            16384
DW            26.550 usec
DE            6.50 usec
TE            360.7 K
D1            1.50000000 sec
D11           0.03000000 sec
TD0           1

```

```

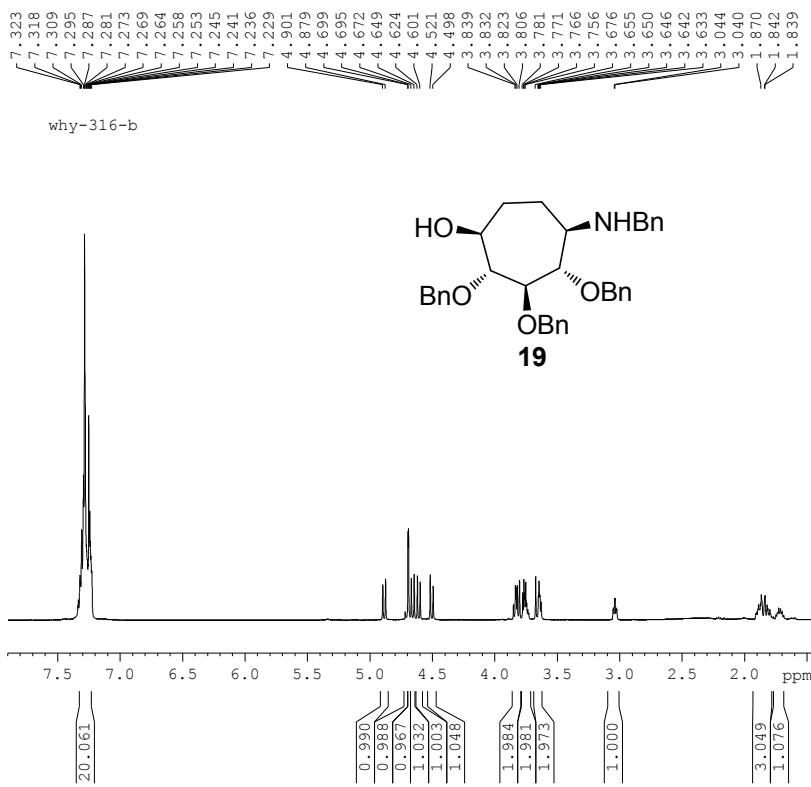
===== CHANNEL f1 =====
NUC1          13C
P1            12.00 usec
PL1           4.00 dB
PL1W         35.00000000 W
SFO1         75.4752953 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.99 dB

```



```

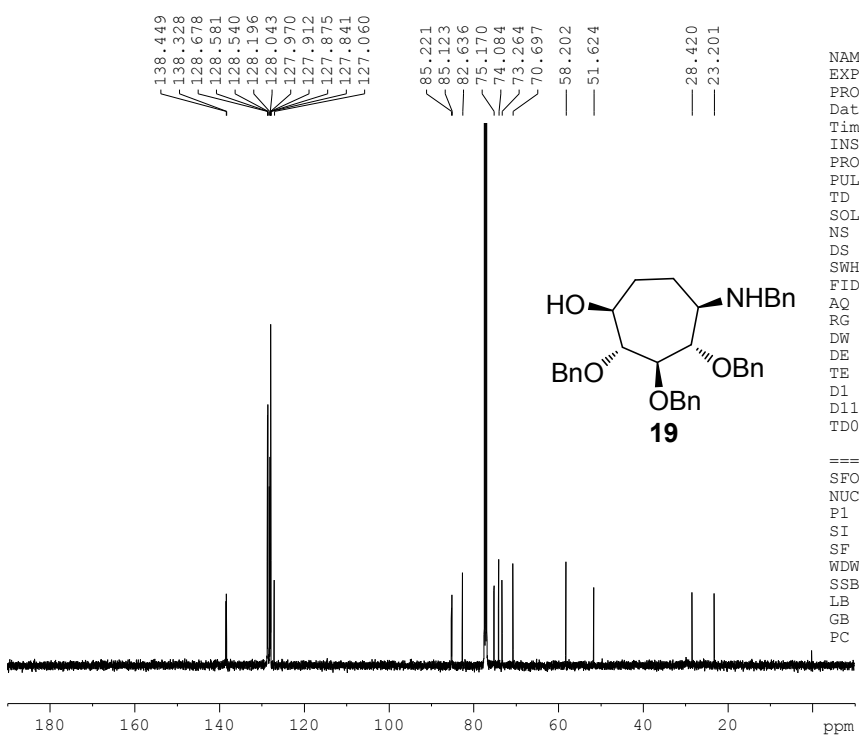
NAME          500m 2016
EXPNO         4
PROCNO        1
Date_         20160116
Time_         19.14
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10000.000 Hz
FIDRES        0.152588 Hz
AQ            3.2768500 sec
RG            55.37
DW            50.000 usec
DE            6.50 usec
TE            298.0 K
D1            1.00000000 sec
D11
D10           1

```

```

===== CHANNEL f1 =====
SFO1          500.1330885 MHz
NUC1          1H
P1            10.60 usec
SI            65536
SF            500.1300158 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```



```

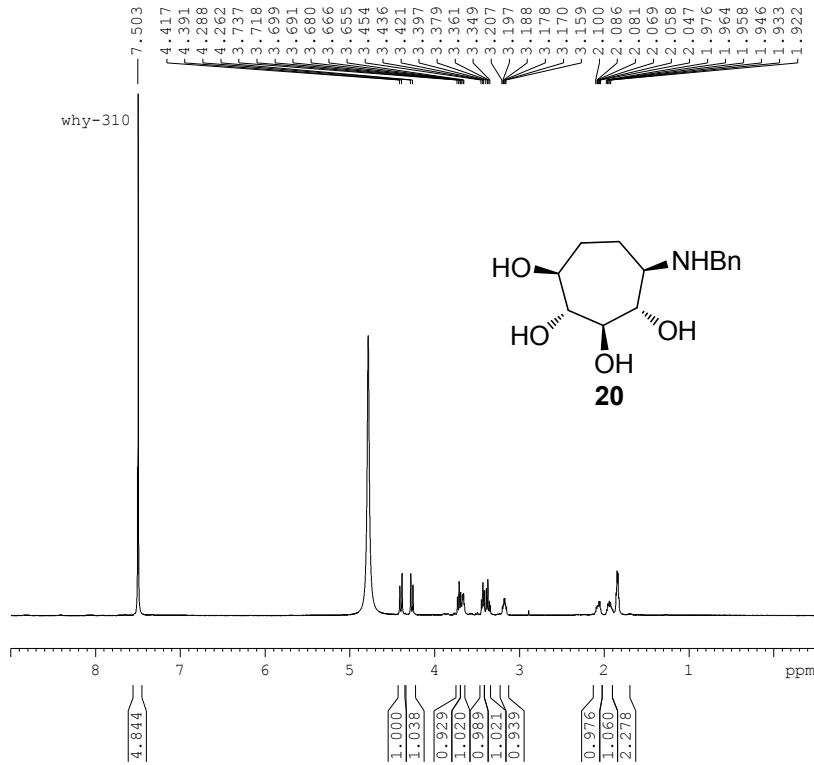
NAME          500m 2016
EXPNO         5
PROCNO        1
Date_         20160116
Time_         19.21
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            180
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            192.89
DW            16.800 usec
DE            18.00 usec
TE            298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D10           1

```

```

===== CHANNEL f1 =====
SFO1          125.7703637 MHz
NUC1          13C
P1            9.80 usec
SI            32768
SF            125.7577724 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

```



```

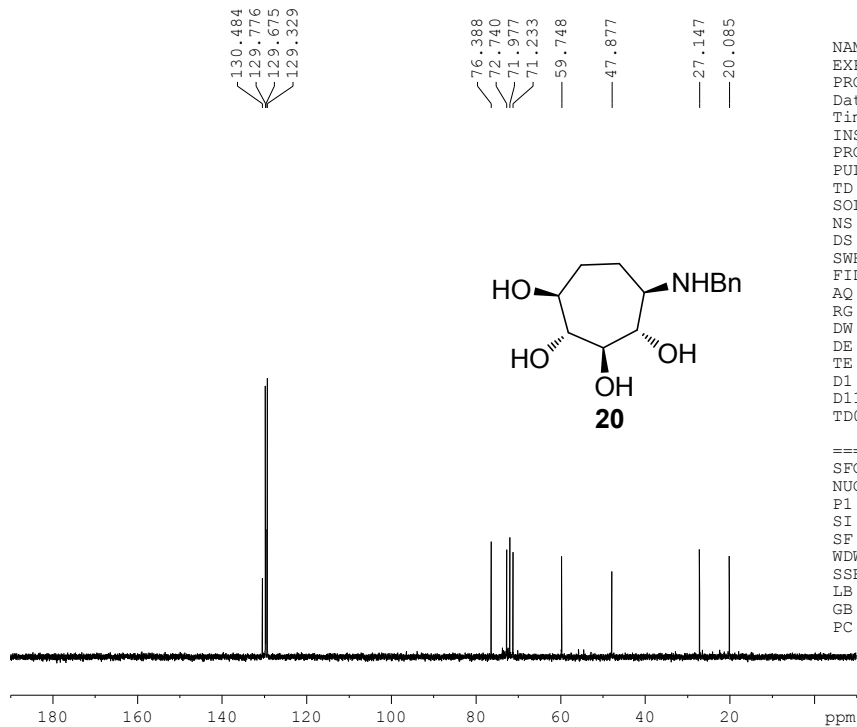
NAME          500M
EXPNO         52
PROCNO        1
Date_         20150914
Time_         10.37
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zg30
TD            65536
SOLVENT       D2O
NS            16
DS            2
SWH           10000.000 Hz
FIDRES        0.152588 Hz
AQ            3.2768500 sec
RG            55.37
DW            50.000 usec
DE            6.50 usec
TE            298.0 K
D1            1.00000000 sec
D10           1
TD0           1

```

```

===== CHANNEL f1 =====
SFO1          500.1330885 MHz
NUC1          1H
P1            10.60 usec
SI            65536
SF            500.1299526 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```



```

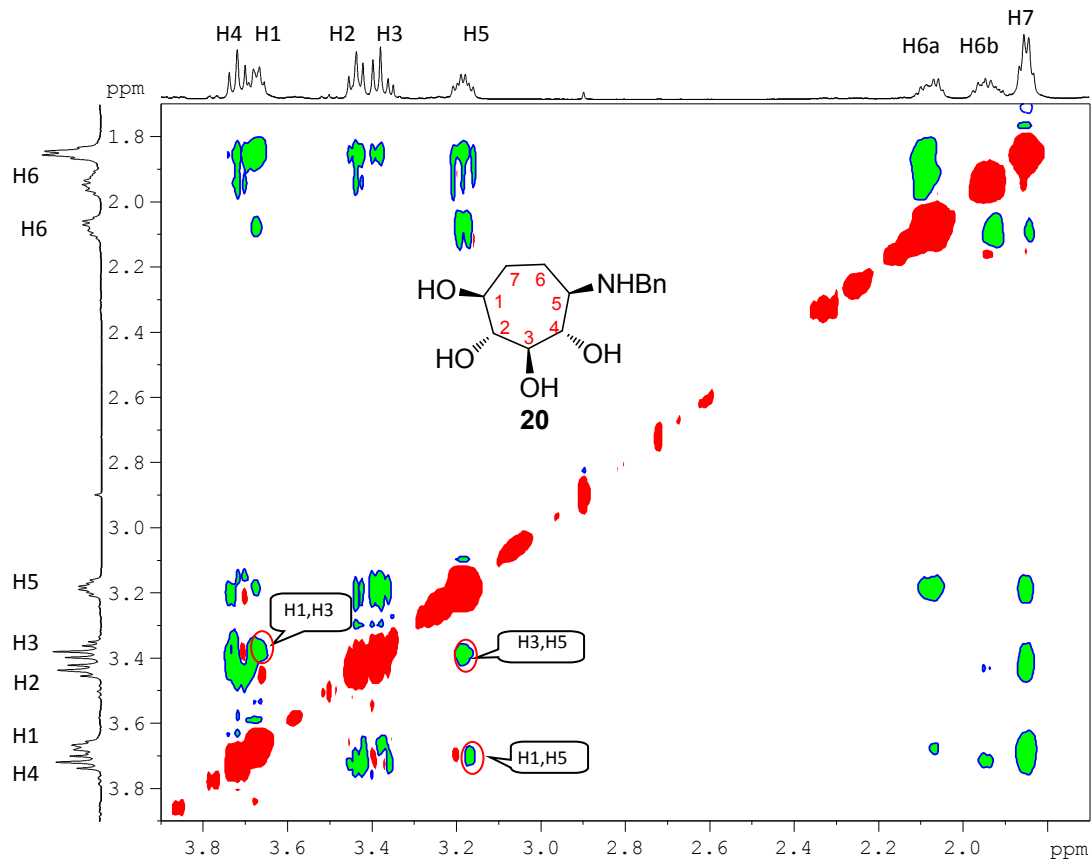
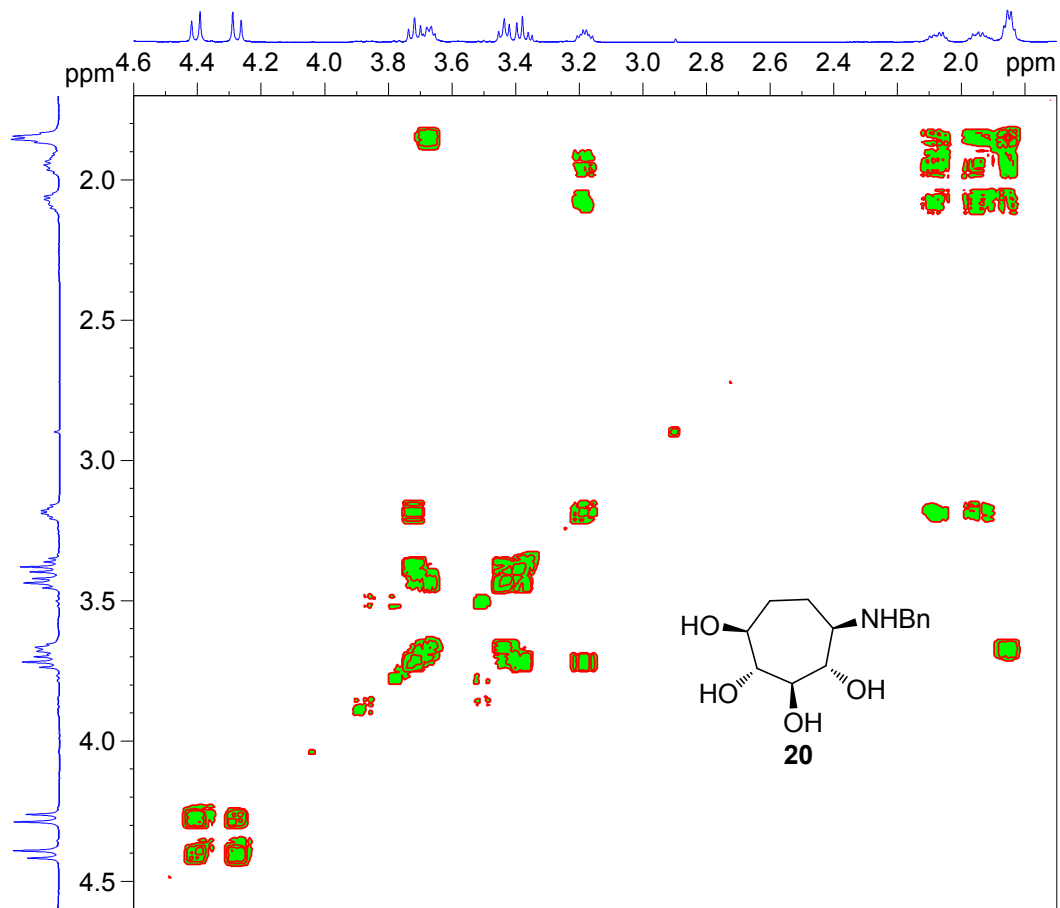
NAME          500M
EXPNO         53
PROCNO        1
Date_         20150914
Time_         10.40
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zgpg30
TD            65536
SOLVENT       D2O
NS            135
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            192.89
DW            16.800 usec
DE            18.00 usec
TE            298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

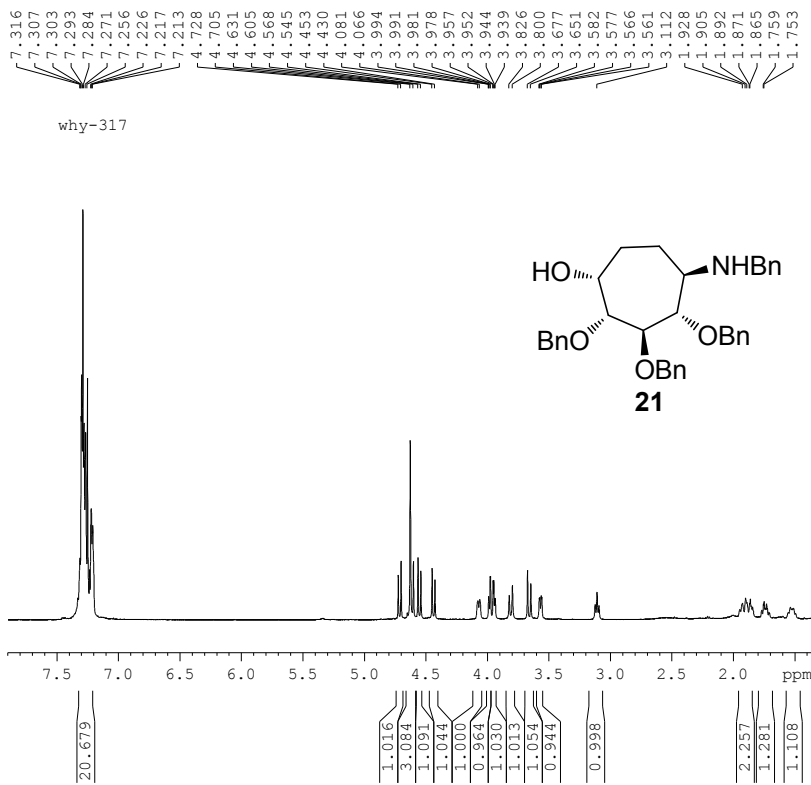
```

```

===== CHANNEL f1 =====
SFO1          125.7703637 MHz
NUC1          13C
P1            9.80 usec
SI            32768
SF            125.7577885 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

```



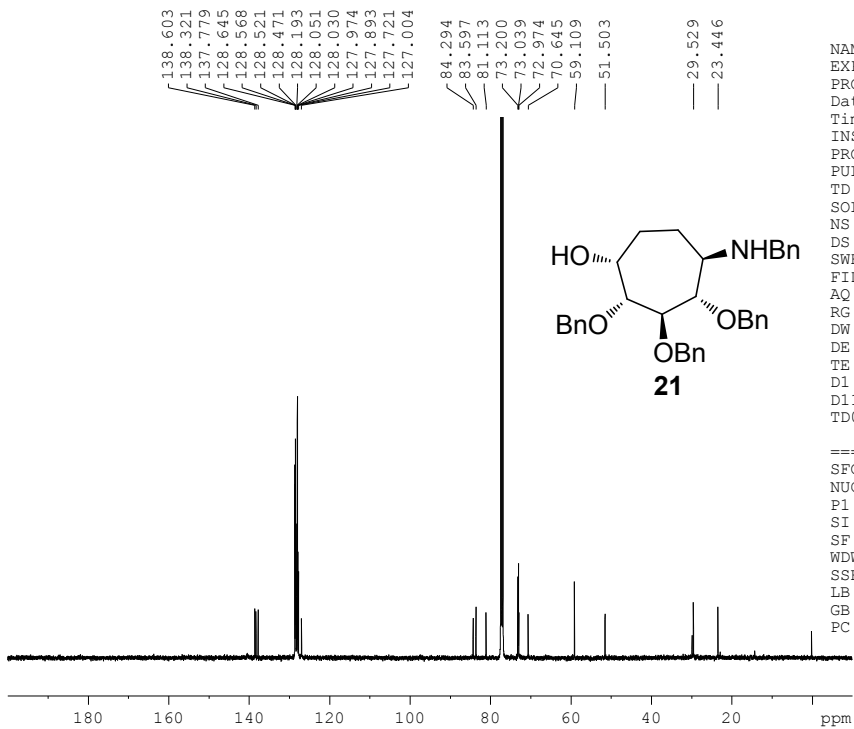


```

NAME          500m 2016
EXPNO         9
PROCNO        1
Date_         20160116
Time_         21.02
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10000.000 Hz
FIDRES        0.152588 Hz
AQ            3.2768500 sec
RG            55.37
DW            50.000 usec
DE            6.50 usec
TE            298.0 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
SFO1          500.1330885 MHz
NUC1          1H
P1            10.60 usec
SI            65536
SF            500.1300147 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

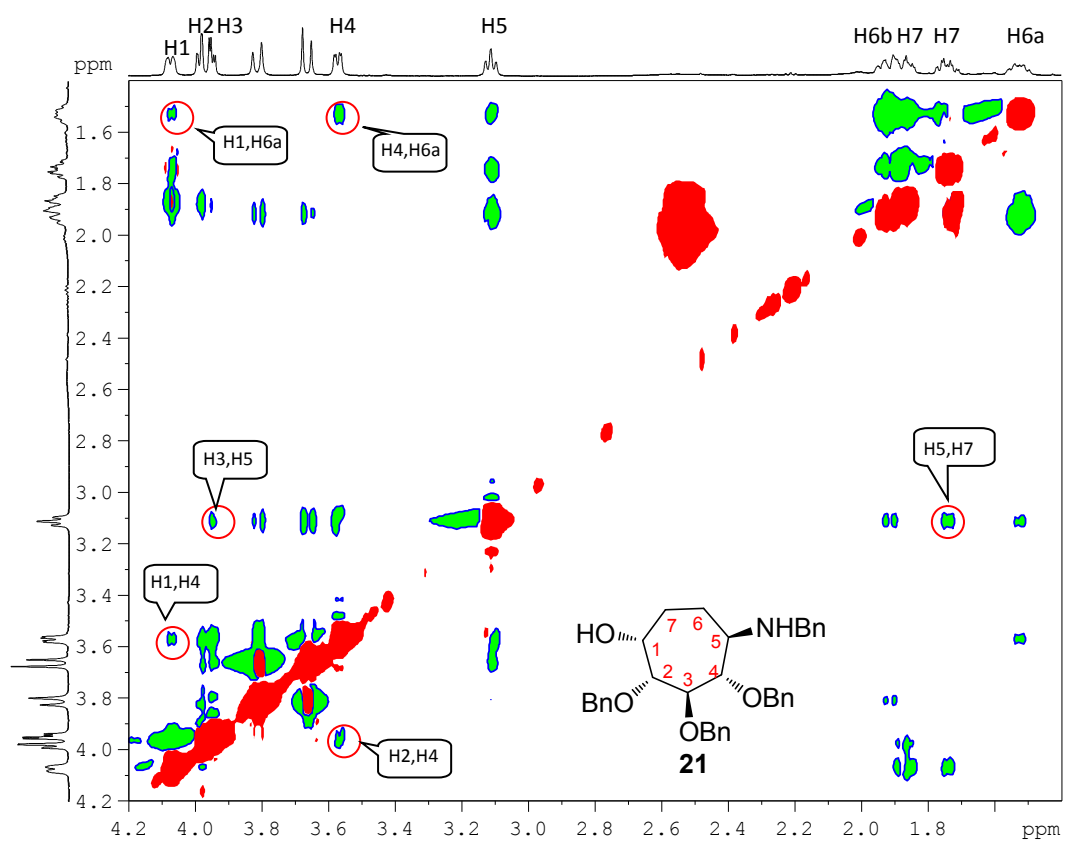
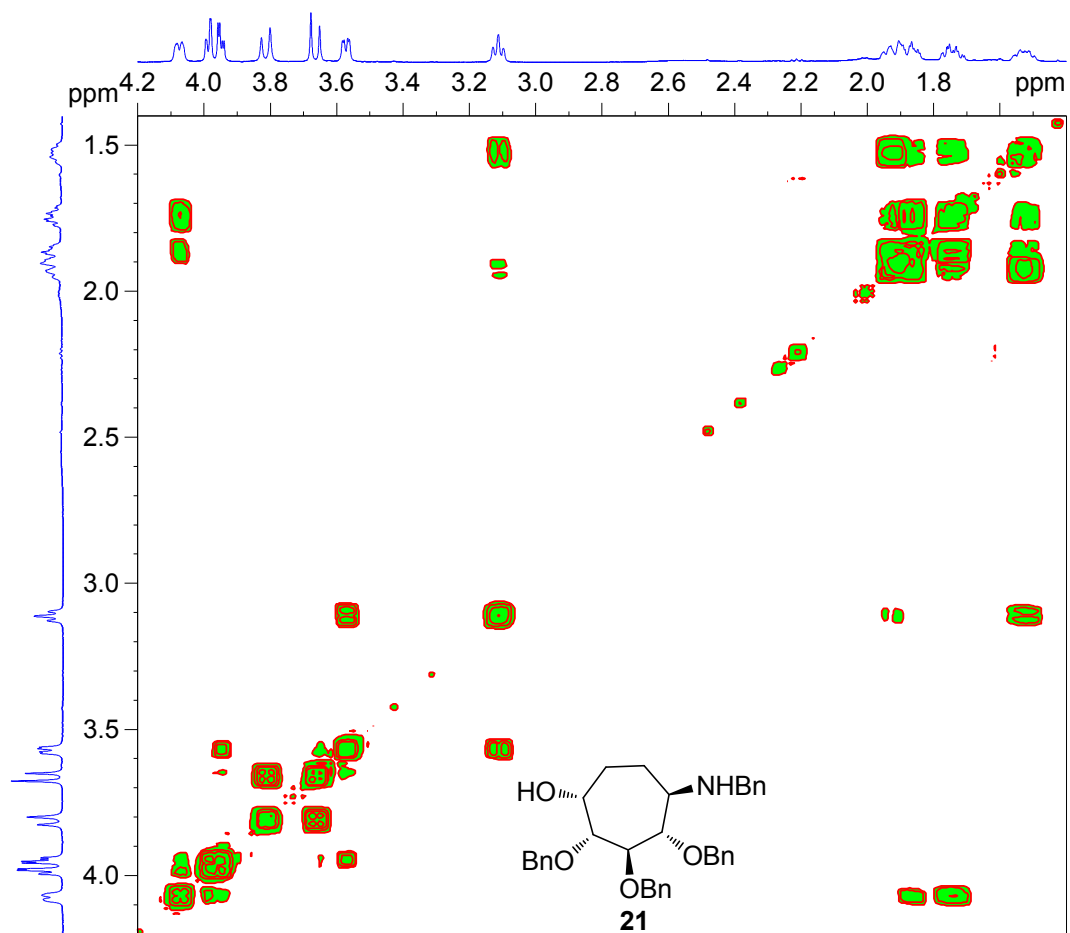


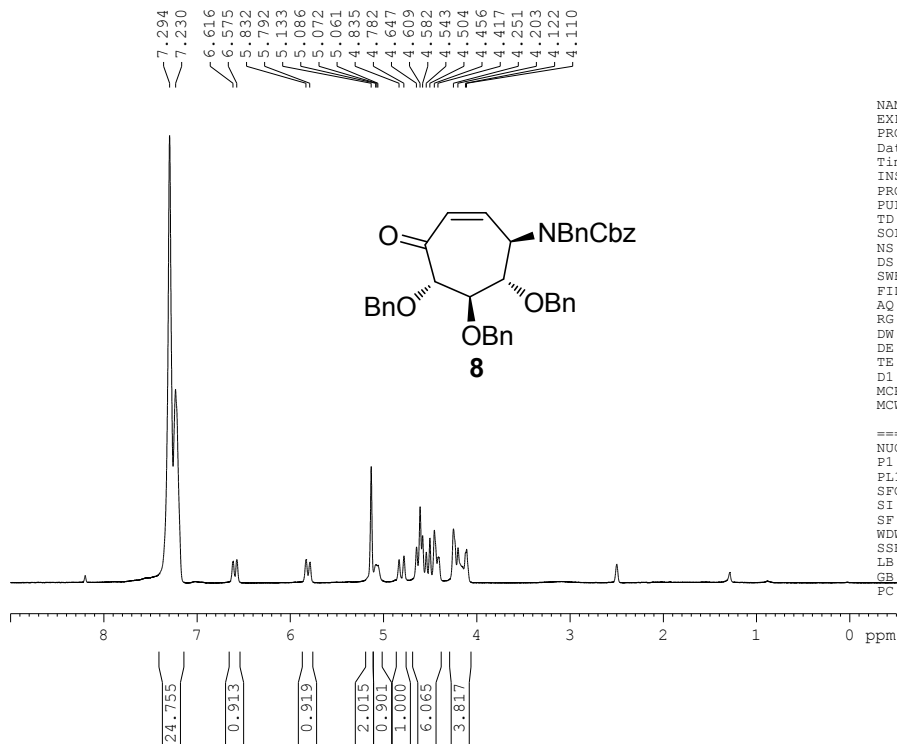
```

NAME          500m 2016
EXPNO         10
PROCNO        1
Date_         20160116
Time_         21.08
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            192.89
DW            16.800 usec
DE            18.00 usec
TE            298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
SFO1          125.7703637 MHz
NUC1          13C
P1            9.80 usec
SI            32768
SF            125.7577721 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



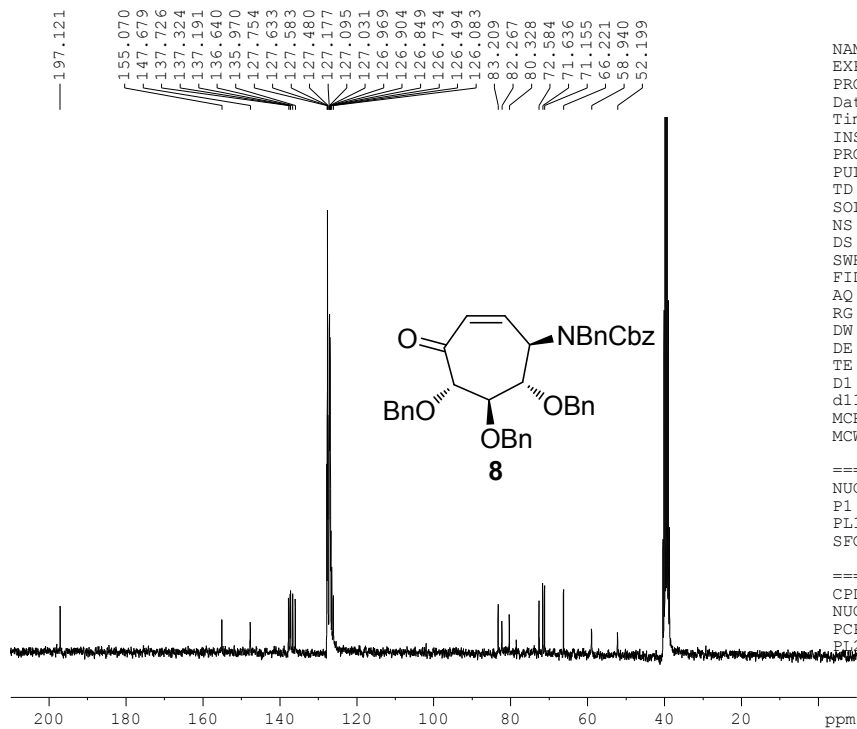


```

NAME          400MHZ
EXPNO         3026
PROCNO        1
Date_         20150421
Time_         10.10
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            25
DS            0
SWH           6613.757 Hz
FIDRES        0.201836 Hz
AQ            2.4773109 sec
RG            8192
DW            75.600 usec
DE            6.00 usec
TE            370.0 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.80 usec
PL1           3.00 dB
SFO1          300.1304800 MHz
SI            65536
SF            300.1299047 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            0.20
  
```



```

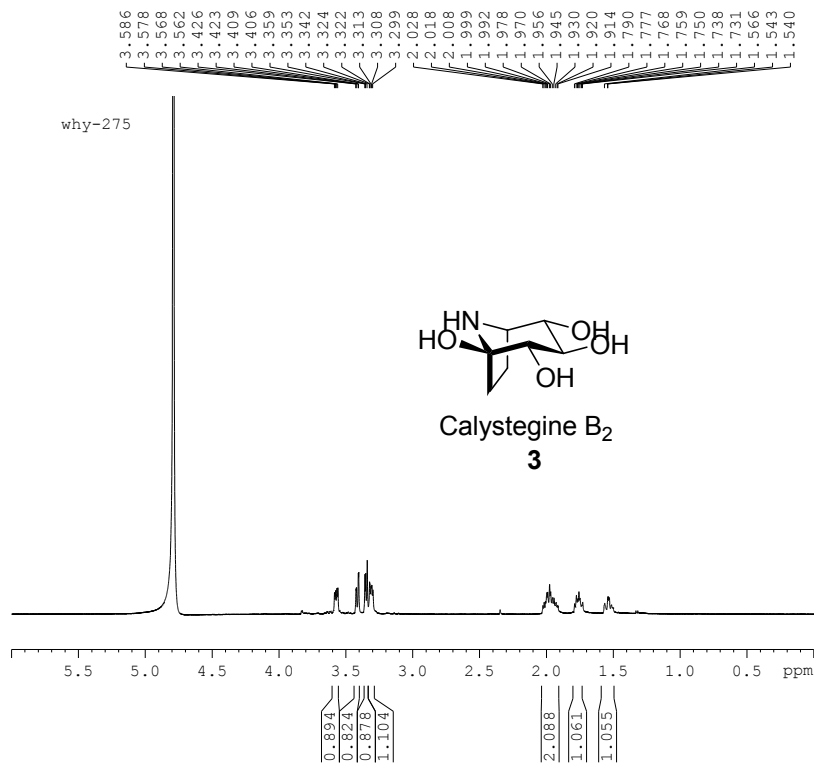
NAME          400MHZ
EXPNO         752
PROCNO        1
Date_         20150421
Time_         9.26
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            1012
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ            0.7209460 sec
RG            32768
DW            22.000 usec
DE            6.00 usec
TE            370.7 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            9.30 usec
PL1           1.00 dB
SFO1          75.4737678 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PT2           2.50 dB
  
```

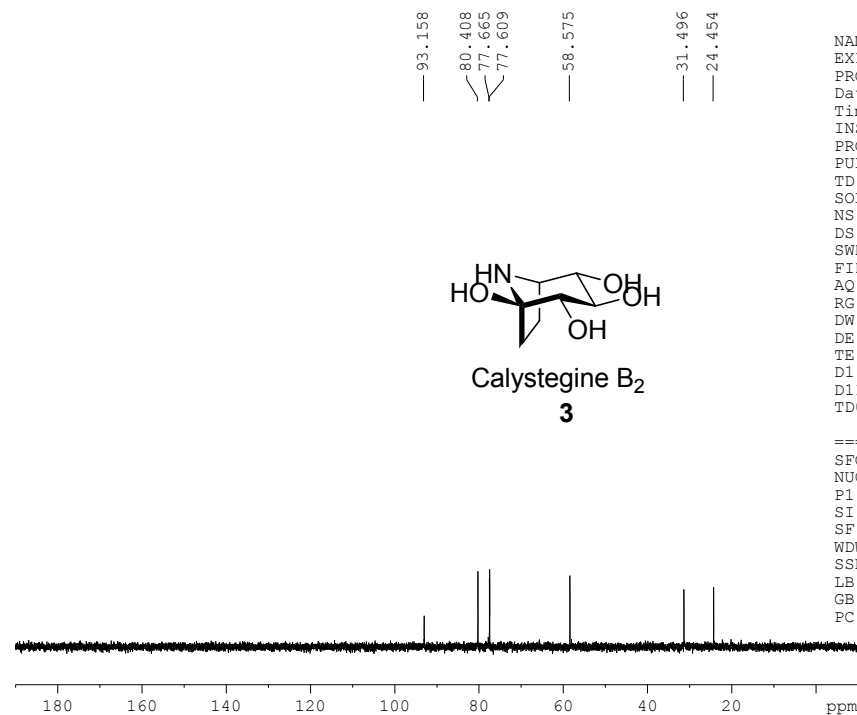


```

NAME          500m 2016
EXPNO         24
PROCNO        1
Date_         20160122
Time          16.47
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zg30
TD            65536
SOLVENT       D2O
NS            68
DS            2
SWH           10000.000 Hz
FIDRES        0.152588 Hz
AQ            3.2768500 sec
RG            87.79
DW            50.000 usec
DE            6.50 usec
TE            298.0 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
SFO1          500.1330885 MHz
NUC1          1H
P1            10.60 usec
SI            32768
SF            500.1299560 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



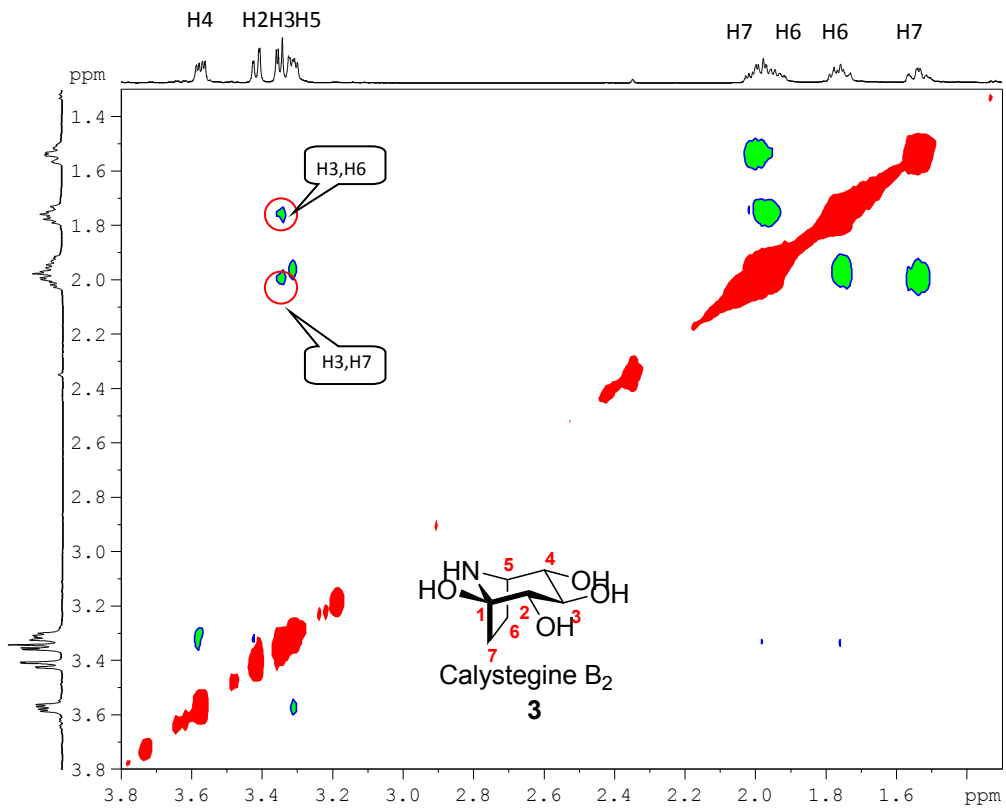
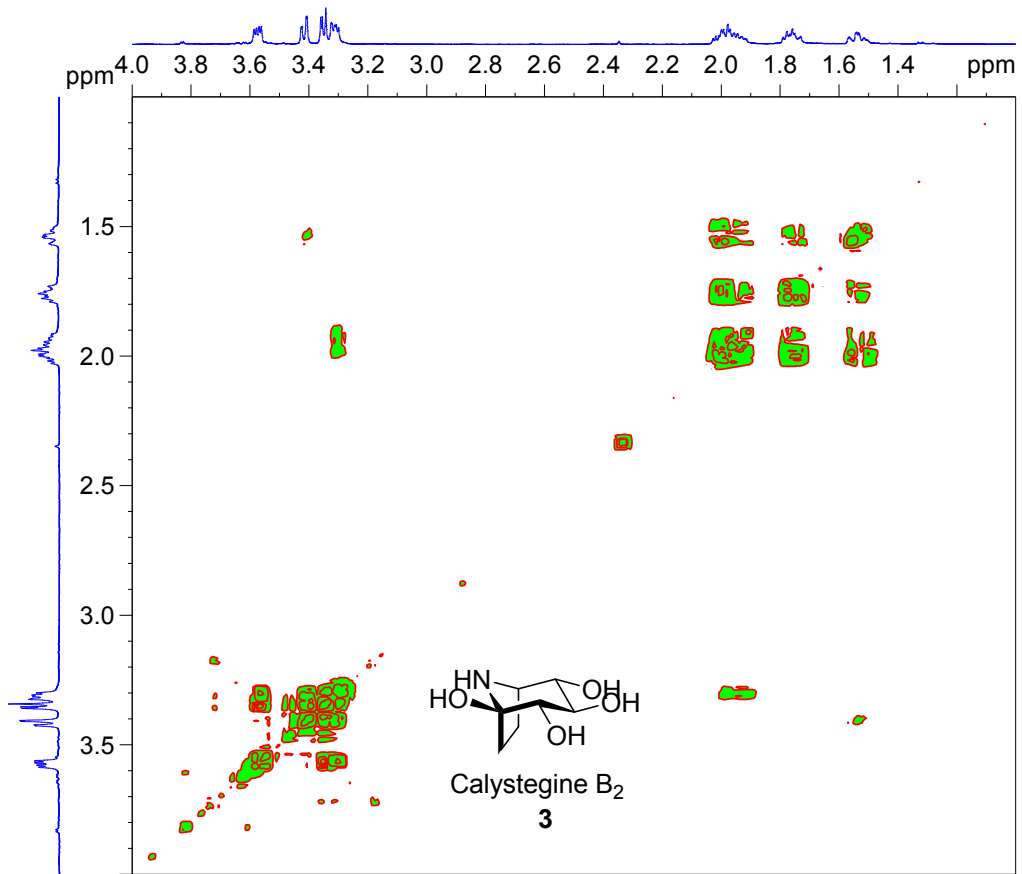
```

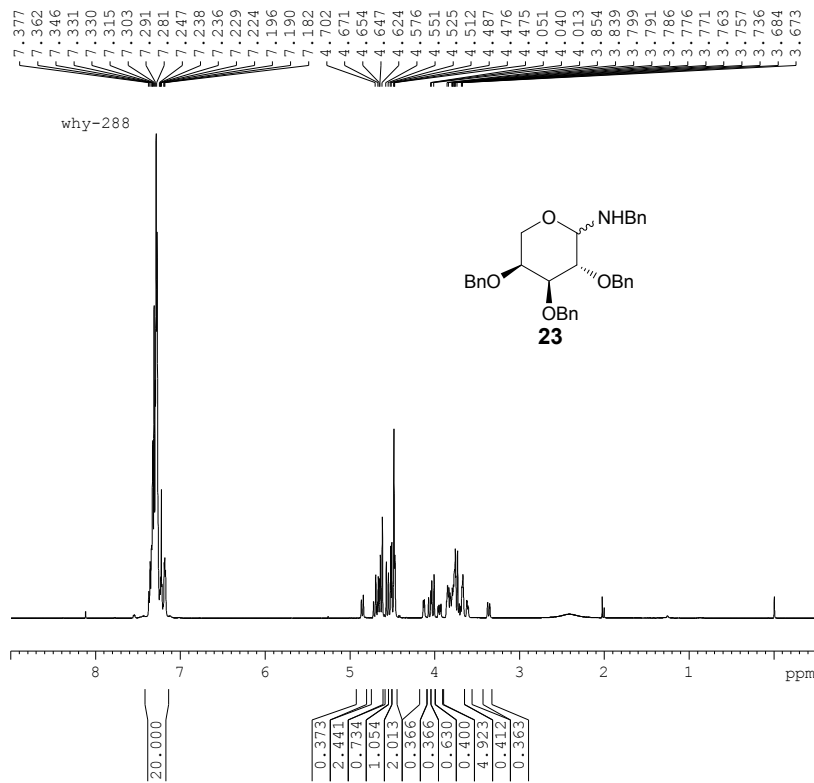
NAME          400MHZ
EXPNO         413
PROCNO        1
Date_         20150203
Time          15.41
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       D2O
NS            217
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            206.33
DW            20.800 usec
DE            6.50 usec
TE            298.7 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            100.6401583 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```







```

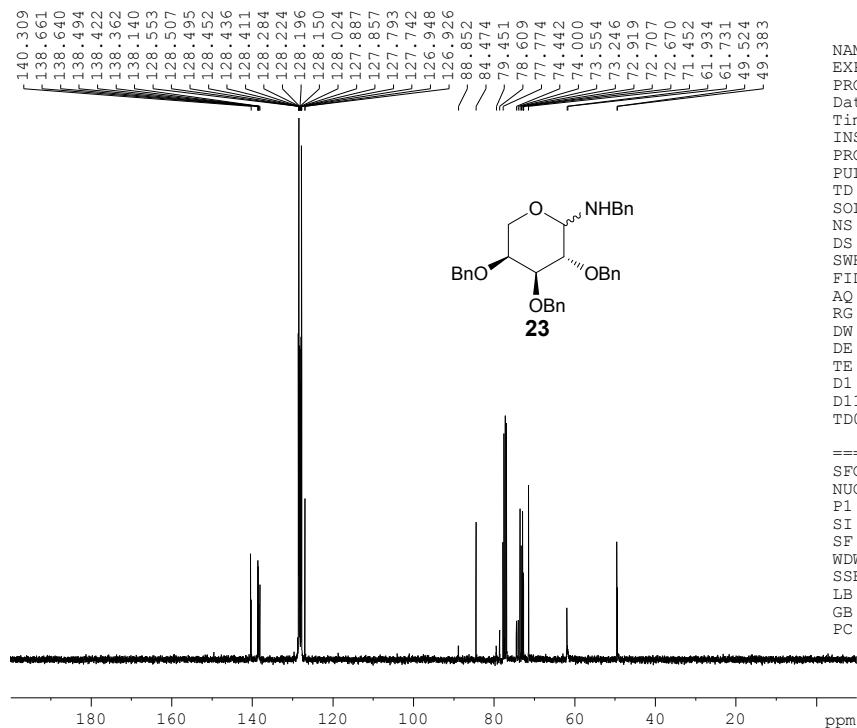
NAME          500M
EXPNO         19
PROCNO        1
Date_         20150422
Time_         18.28
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10000.000 Hz
FIDRES        0.152588 Hz
AQ            3.2768500 sec
RG            31.72
DW            50.000 usec
DE            6.50 usec
TE            298.0 K
D1            1.00000000 sec
D10           1

```

```

===== CHANNEL f1 =====
SFO1          500.1330885 MHz
NUC1           1H
P1            10.60 usec
SI            65536
SF            500.1330288 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```



```

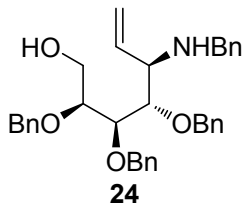
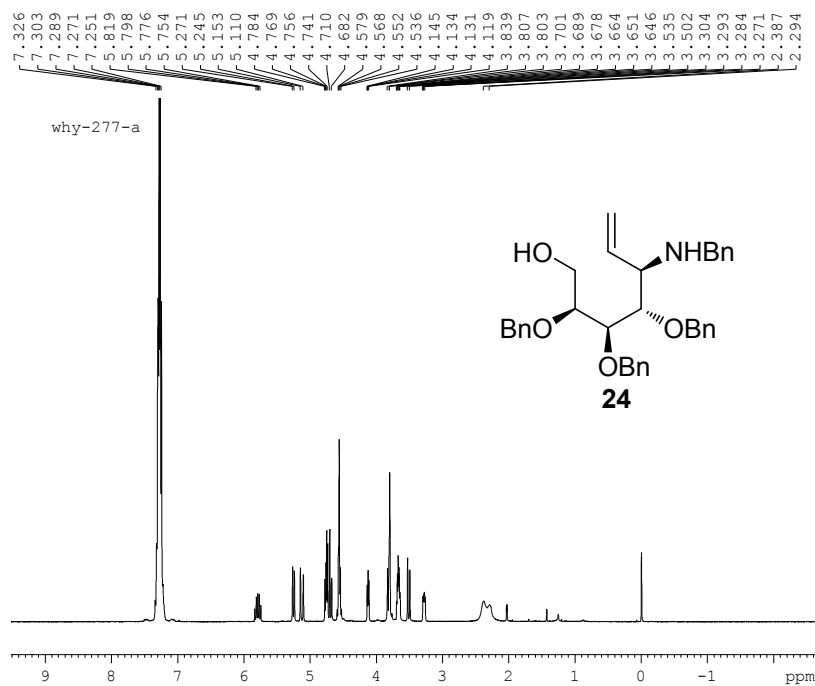
NAME          400MHZ
EXPNO         459
PROCNO        1
Date_         20150422
Time_         18.44
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            59
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            206.33
DW            20.800 usec
DE            6.50 usec
TE            298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
D10           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

```

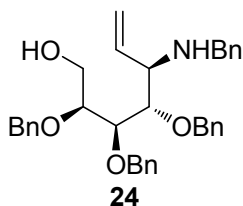
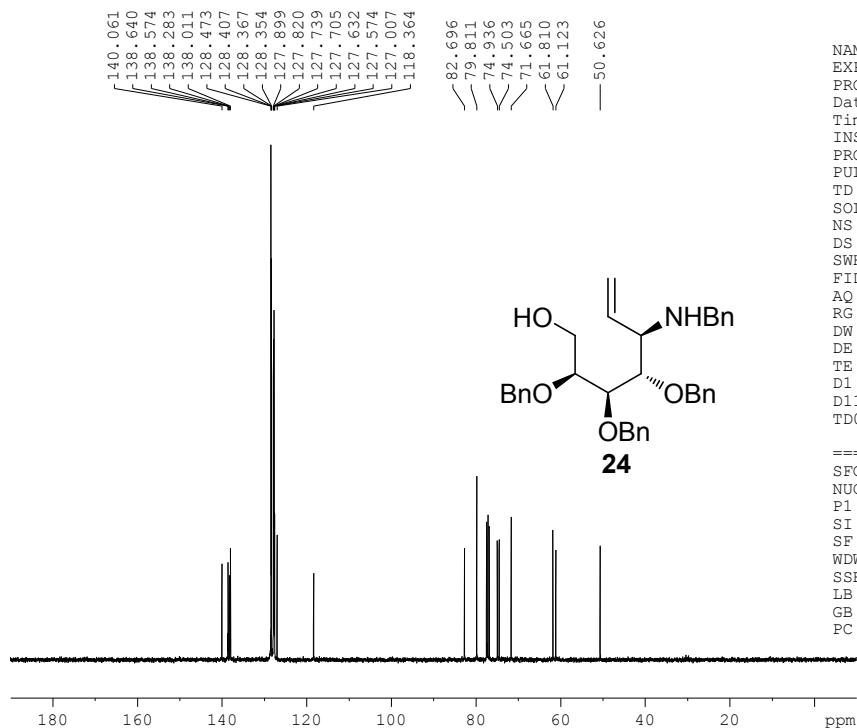


```

NAME          400MHZ
EXPNO         427
PROCNO        1
Date_         20150331
Time_         19.45
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
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            298.1 K
D1            2.00000000 sec
D11           1
TD0           1
  
```

```

===== CHANNEL f1 =====
SFO1          400.2424716 MHz
NUC1          1H
P1            14.80 usec
SI            65536
SF            400.2400131 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

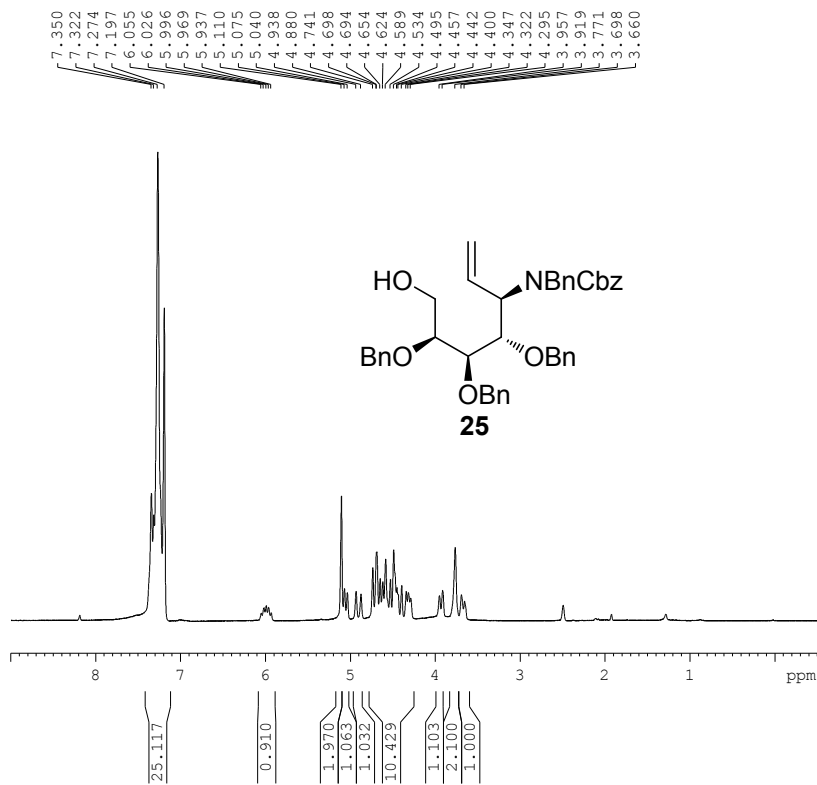


```

NAME          400MHZ
EXPNO         428
PROCNO        1
Date_         20150331
Time_         19.52
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            58
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            206.33
DW            20.800 usec
DE            6.50 usec
TE            298.3 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            100.6404369 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

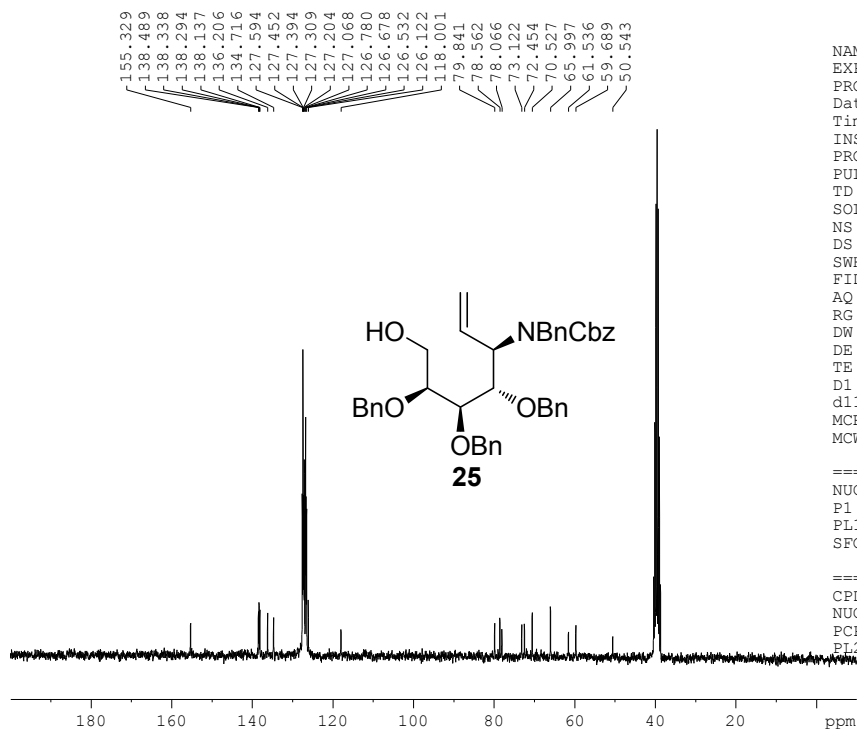


```

NAME          400MHZ
EXPNO         3027
PROCNO        1
Date_         20150421
Time_         10.57
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            23
DS            0
SWH           6613.757 Hz
FIDRES        0.201836 Hz
AQ           2.4773109 sec
RG            8192
DW           75.600 usec
DE            6.00 usec
TE            370.0 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1           1H
P1             13.80 usec
PL1            3.00 dB
SFO1          300.1304800 MHz
SI            65536
SF            300.1299084 MHz
WDW            EM
SSB            0
LB            0.30 Hz
GB            0
  
```



```

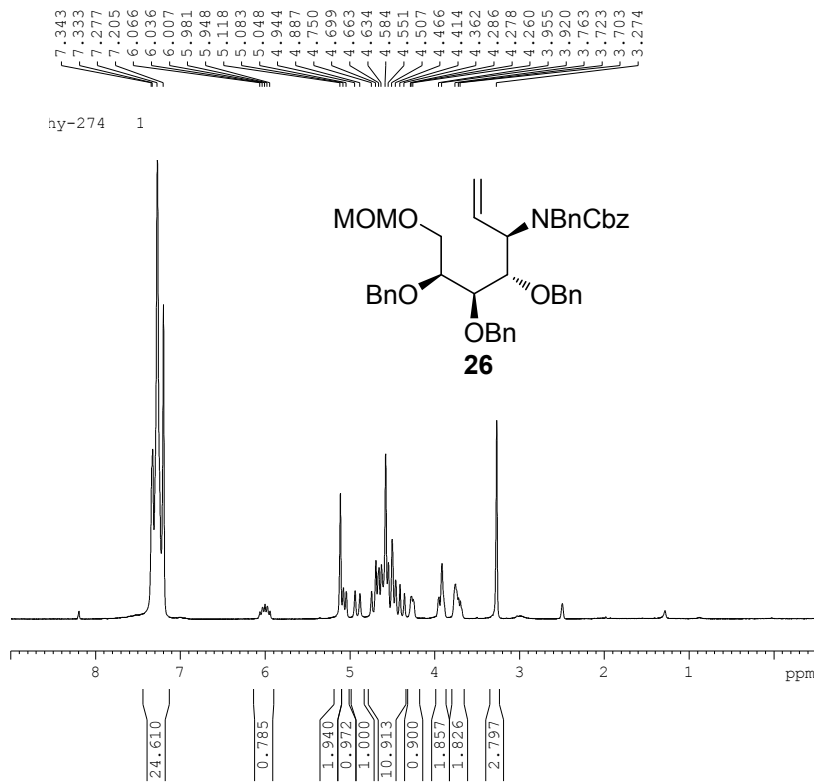
NAME          400MHZ
EXPNO         753
PROCNO        1
Date_         20150421
Time_         10.32
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            526
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ           0.7209460 sec
RG            32768
DW           22.000 usec
DE            6.00 usec
TE            371.5 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1           13C
P1             9.30 usec
PL1            1.00 dB
SFO1          75.4737678 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2            2.50 dB
  
```

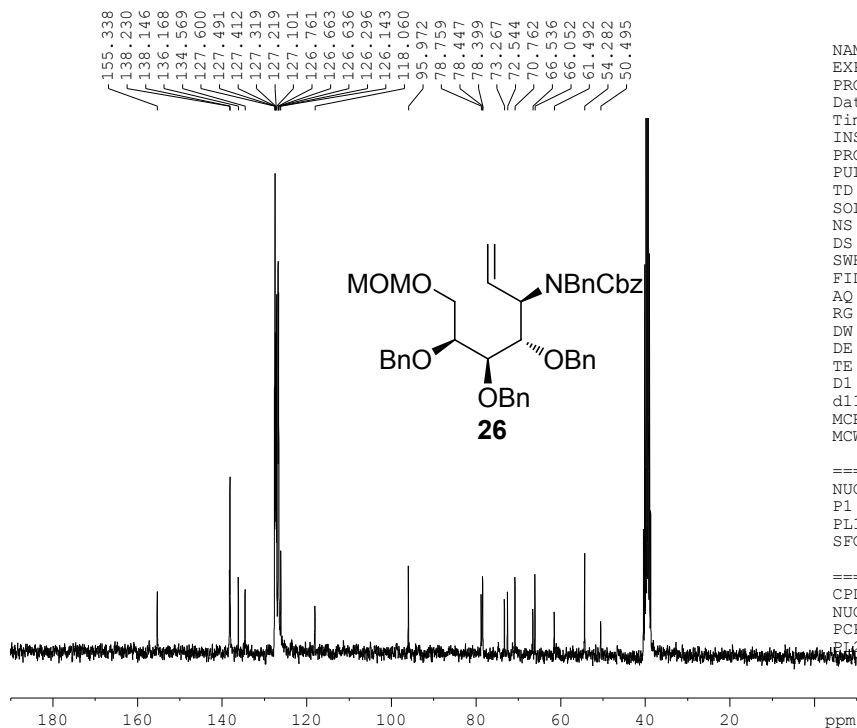


```

NAME          400MHZ
EXPNO         3028
PROCNO        1
Date_         20150421
Time_         11.45
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            19
DS            0
SWH           6613.757 Hz
FIDRES        0.201836 Hz
AQ            2.4773109 sec
RG            8192
DW            75.600 usec
DE            6.00 usec
TE            370.0 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.80 usec
PL1           3.00 dB
SFO1          300.1304800 MHz
SI            65536
SF            300.1299051 MHz
WDB           EM
SSB           0
LB            0.30 Hz
GB            0
  
```



```

NAME          400MHZ
EXPNO         754
PROCNO        1
Date_         20150421
Time_         11.18
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            557
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ            0.7209460 sec
RG            32768
DW            22.000 usec
DE            6.00 usec
TE            369.9 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

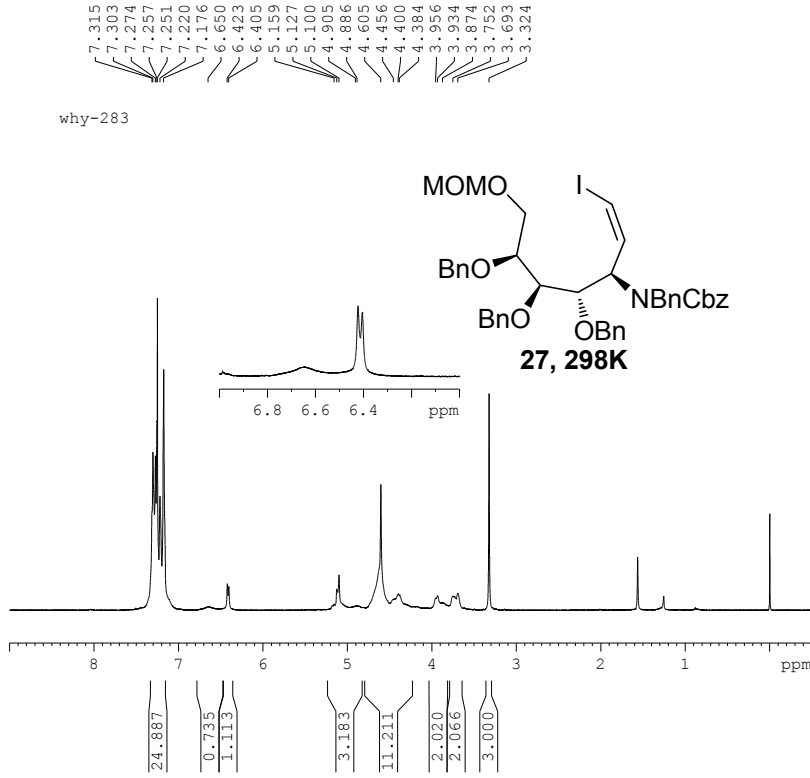
```

===== CHANNEL f1 =====
NUC1          13C
P1            9.30 usec
PL1           1.00 dB
SFO1          75.4737678 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
  
```

why-283

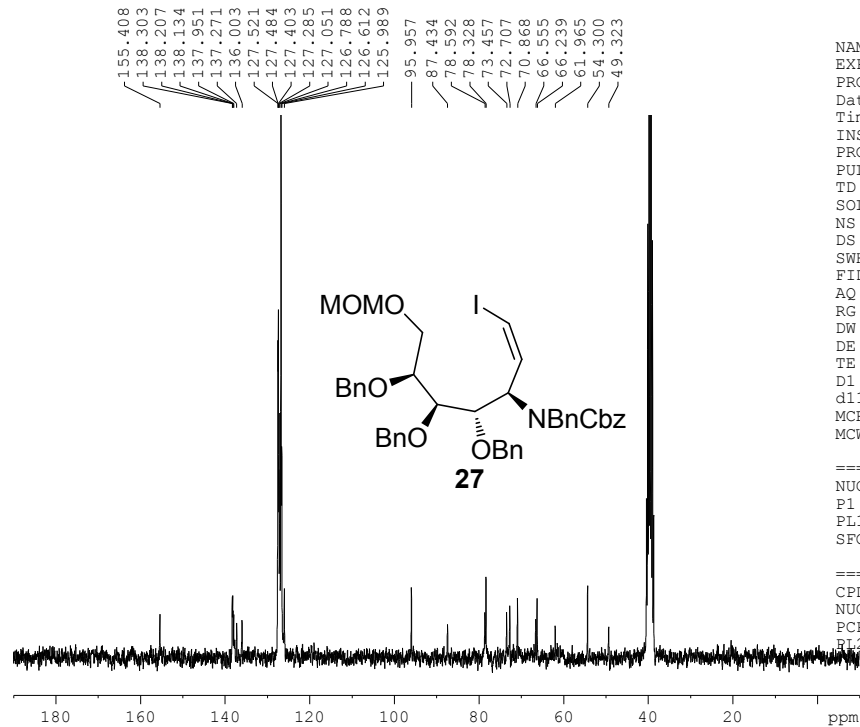


```

NAME          400MHZ
EXPNO         451
PROCNO        1
Date_         20150416
Time_         9.32
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            0
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            2.0447731 sec
RG            125.02
DW            62.400 usec
DE            6.50 usec
TE            298.2 K
D1            2.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
SFO1          400.2424716 MHz
NUC1           1H
P1            14.80 usec
SI            65536
SF            400.2400133 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



```

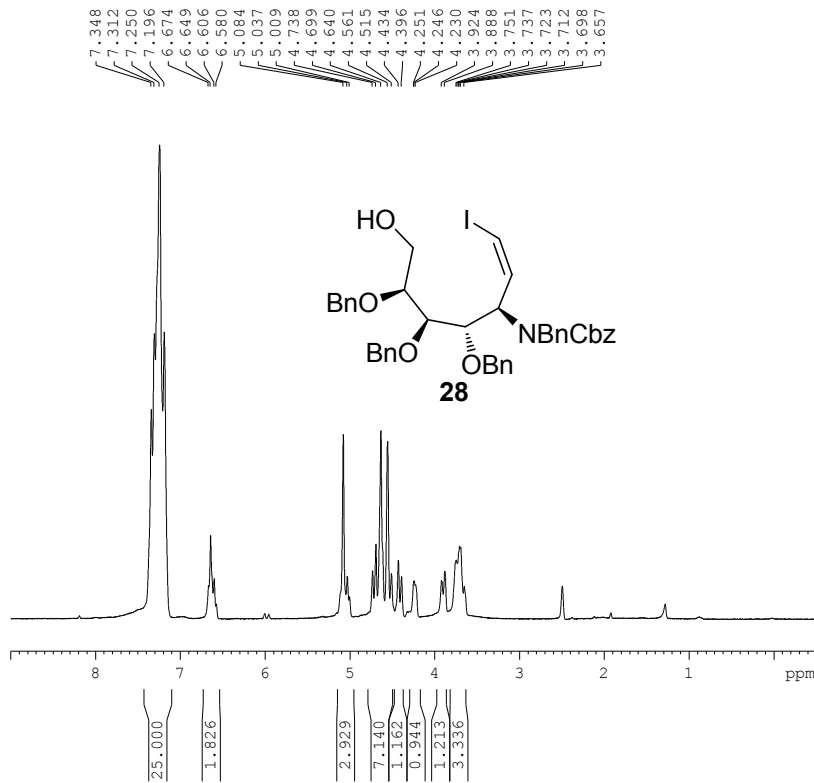
NAME          400MHZ
EXPNO         758
PROCNO        1
Date_         20150507
Time_         8.34
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            1002
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ            0.7209460 sec
RG            32768
DW            22.000 usec
DE            6.00 usec
TE            370.4 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1           13C
P1             9.30 usec
PL1            1.00 dB
SFO1          75.4737678 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PT2           2.50 dB
  
```



```

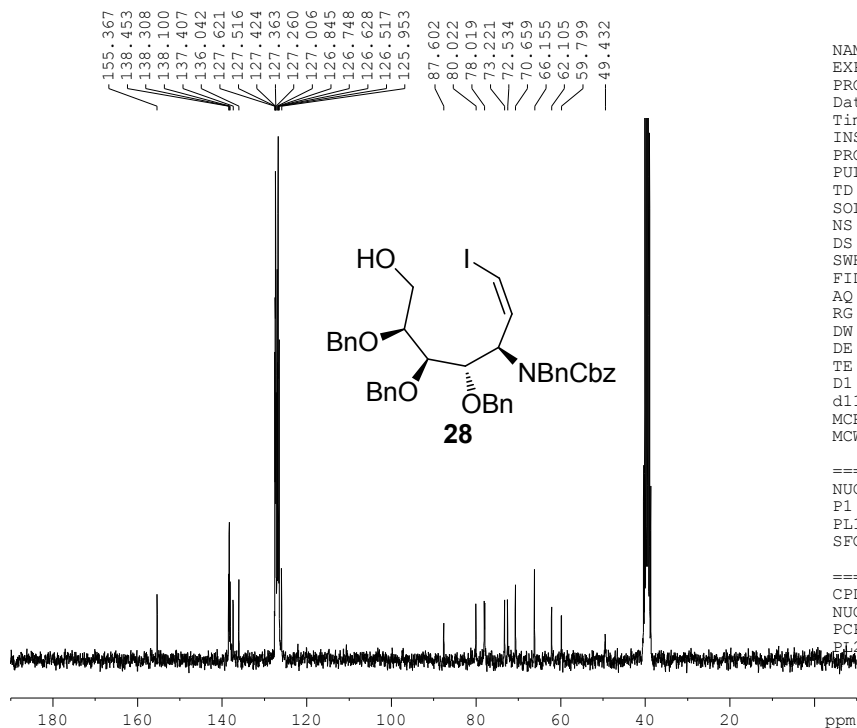
NAME          400MHZ
EXPNO         3029
PROCNO        1
Date_         20150421
Time_         14.48
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            21
DS            0
SWH           6613.757 Hz
FIDRES        0.201836 Hz
AQ            2.4773109 sec
RG            8192
DW            75.600 usec
DE            6.00 usec
TE            369.9 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.80 usec
PL1           3.00 dB
SFO1         300.1304800 MHz
SI            65536
SF           300.1299091 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0

```



```

NAME          400MHZ
EXPNO         755
PROCNO        1
Date_         20150421
Time_         13.55
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            1145
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ            0.7209460 sec
RG            32768
DW            22.000 usec
DE            6.00 usec
TE            371.2 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

```

```

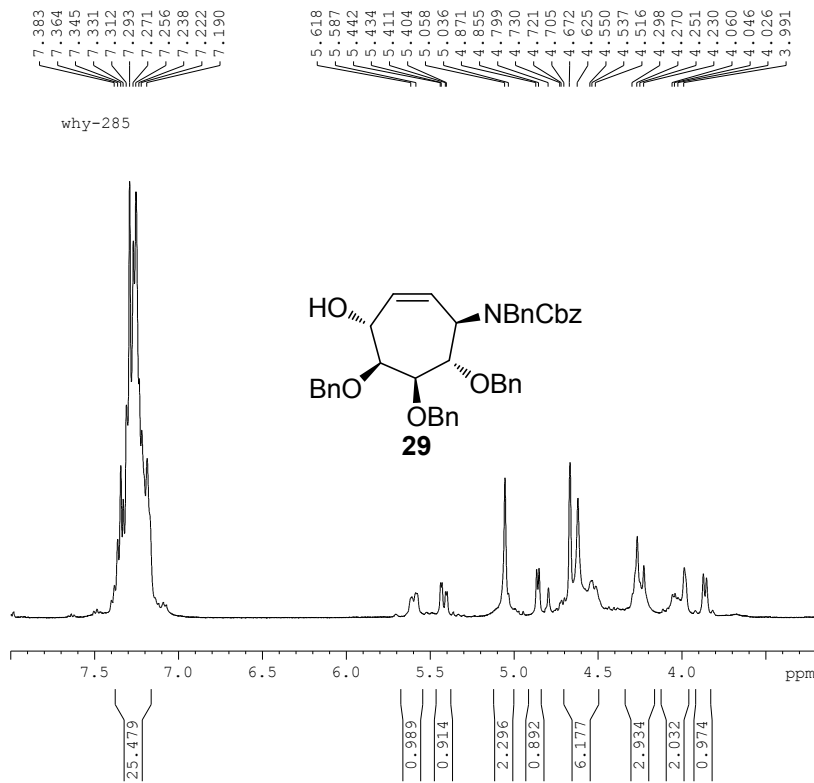
===== CHANNEL f1 =====
NUC1          13C
P1            9.30 usec
PL1           1.00 dB
SFO1         75.4737678 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB

```

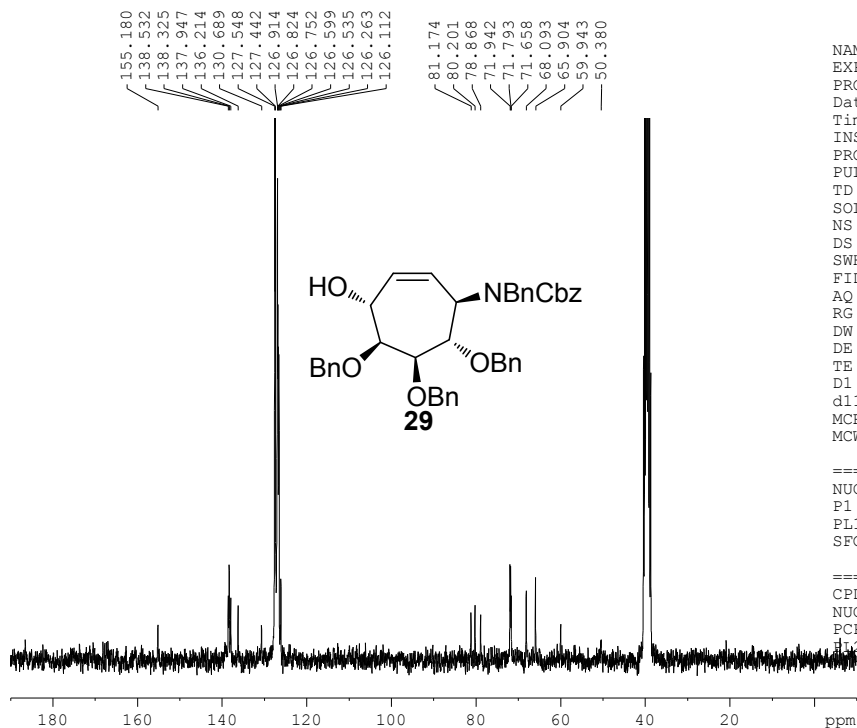


```

NAME          400M-2016
EXPNO         13
PROCNO        4
Date_         20160316
Time_         20.13
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            16
DS            0
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            2.0447731 sec
RG            125.02
DW            62.400 usec
DE            6.50 usec
TE            342.8 K
D1            2.00000000 sec
D11           1
TD0           1
  
```

```

===== CHANNEL f1 =====
SFO1          400.2424716 MHz
NUC1           1H
P1            14.80 usec
SI            65536
SF            400.2400034 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



```

NAME          400MHZ
EXPNO         759
PROCNO        1
Date_         20150507
Time_         9.38
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            2036
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ            0.7209460 sec
RG            32768
DW            22.000 usec
DE            6.00 usec
TE            370.9 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

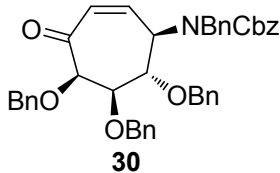
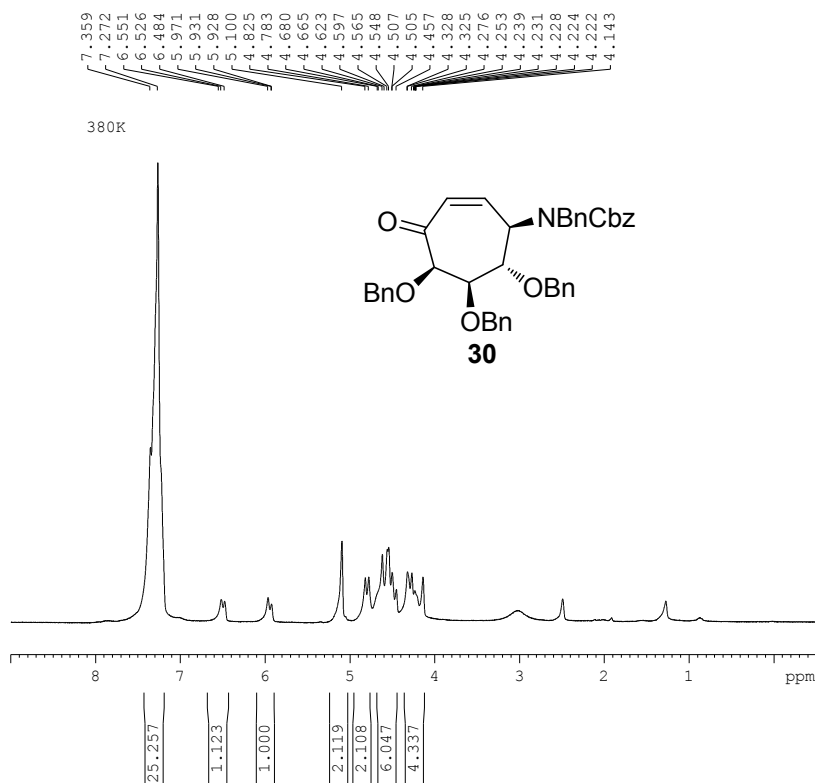
```

===== CHANNEL f1 =====
NUC1          13C
P1            9.30 usec
PL1           1.00 dB
SFO1          75.4737678 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2           2.50 dB
  
```



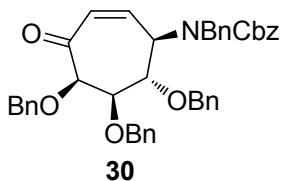
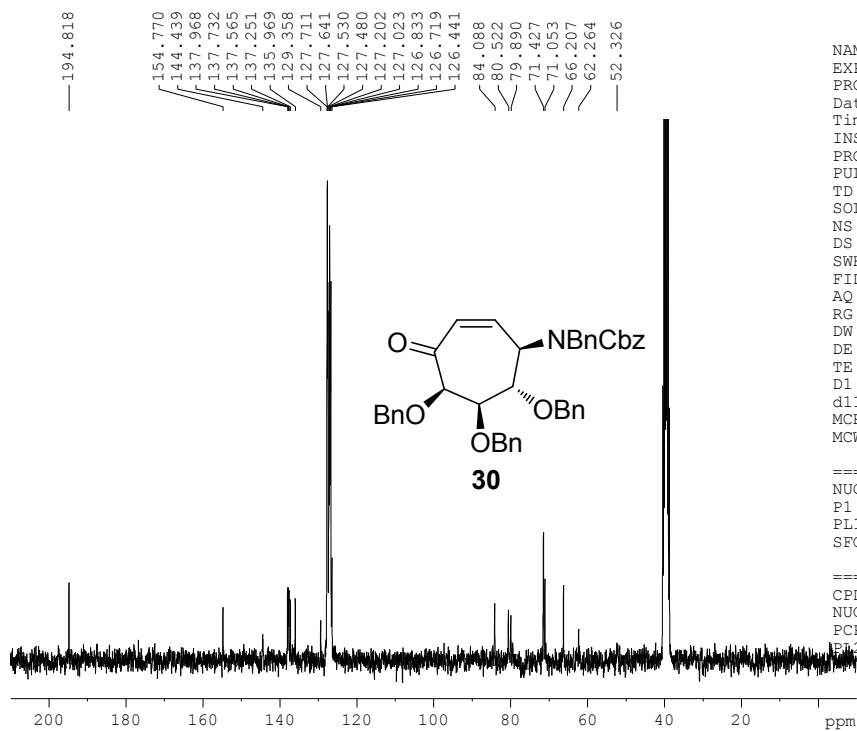


```

NAME          400MHZ
EXPNO         3062
PROCNO        1
Date_         20150507
Time_         12.19
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            32768
SOLVENT       CDC13
NS            19
DS            0
SWH           8992.806 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG            8192
DW            55.600 usec
DE            6.00 usec
TE            369.9 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.80 usec
PL1           3.00 dB
SFO1          300.1304800 MHz
SI            65536
SF            300.1304844 MHz
WDW           EM
SSB           0
LB            0.00 Hz
GB            0
  
```



```

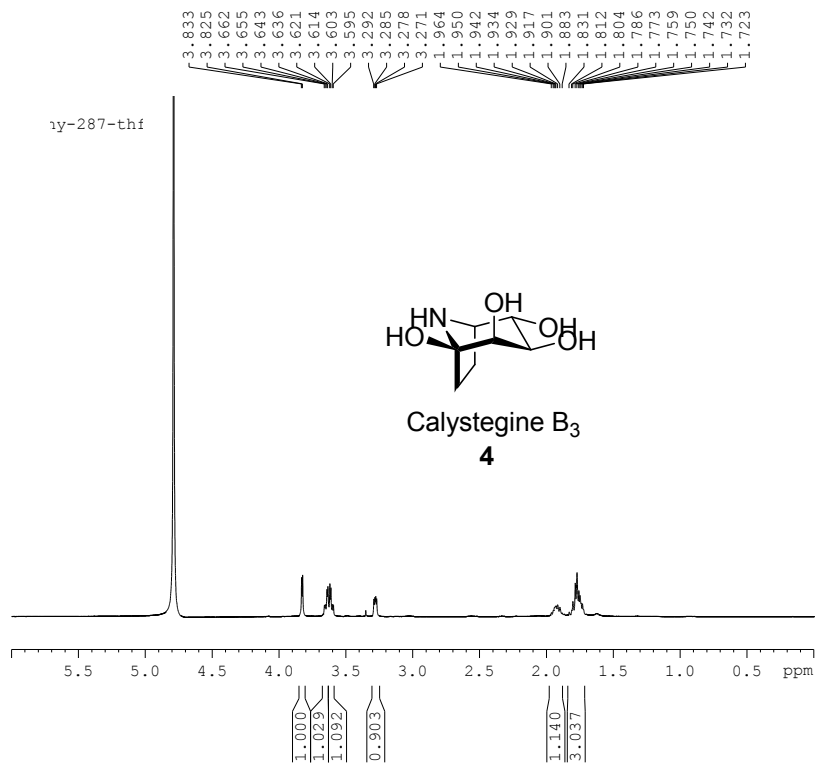
NAME          400MHZ
EXPNO         760
PROCNO        1
Date_         20150507
Time_         11.28
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig30
TD            32768
SOLVENT       DMSO
NS            1086
DS            0
SWH           22727.273 Hz
FIDRES        0.693581 Hz
AQ            0.7209460 sec
RG            32768
DW            22.000 usec
DE            6.00 usec
TE            371.1 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            9.30 usec
PL1           1.00 dB
SFO1          75.4737678 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
P2           2.50 dB
  
```

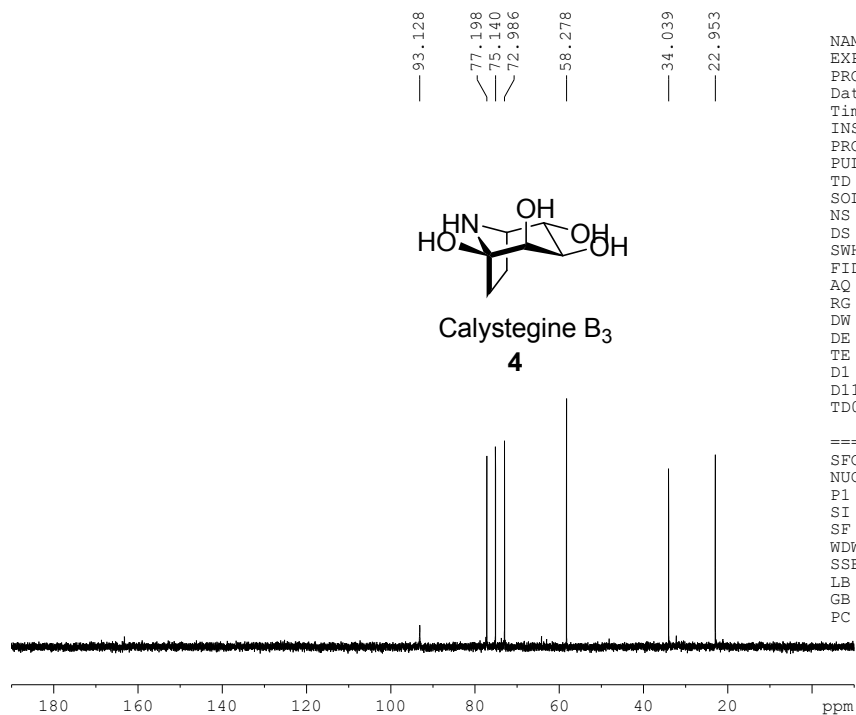


```

NAME          500m 2016
EXPNO         39
PROCNO        1
Date_         20160202
Time          15.31
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zg30
TD            65536
SOLVENT       D2O
NS            16
DS            2
SWH           10000.000 Hz
FIDRES        0.152588 Hz
AQ            3.2768500 sec
RG            55.37
DW            50.000 usec
DE            6.50 usec
TE            298.0 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
SFO1          500.1330885 MHz
NUC1          1H
P1            10.60 usec
SI            65536
SF            500.1299563 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

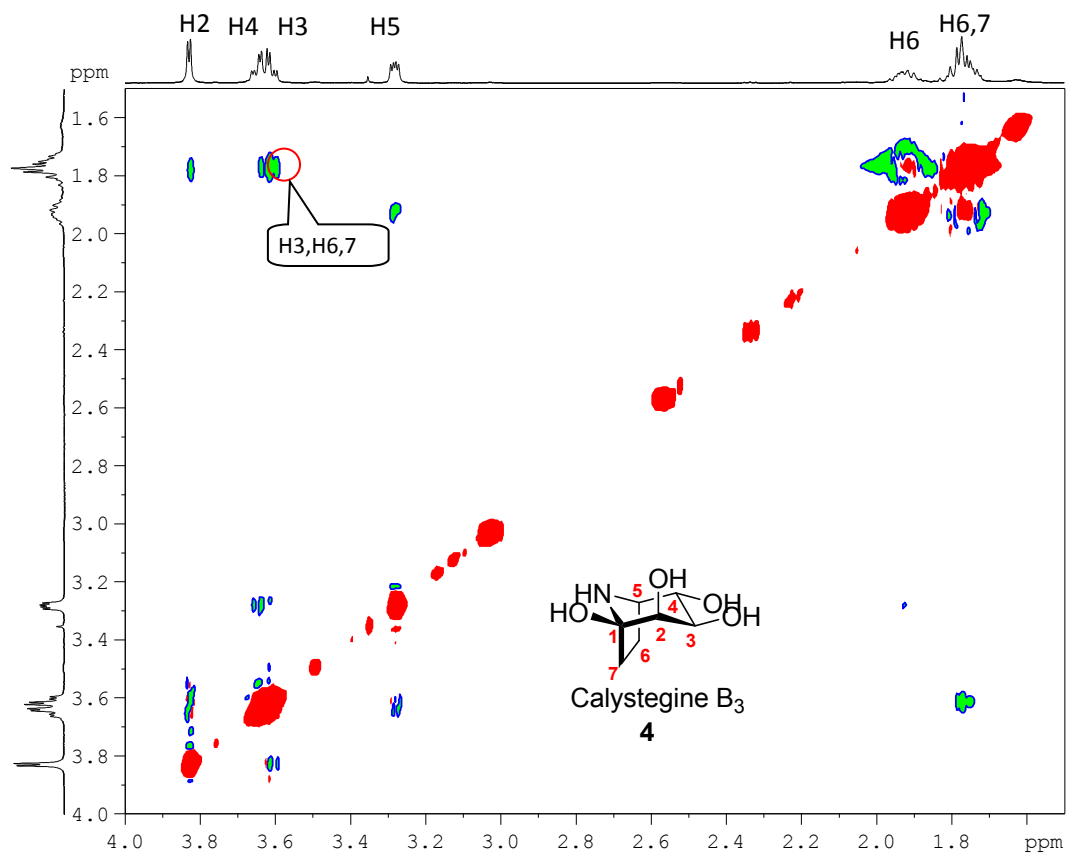
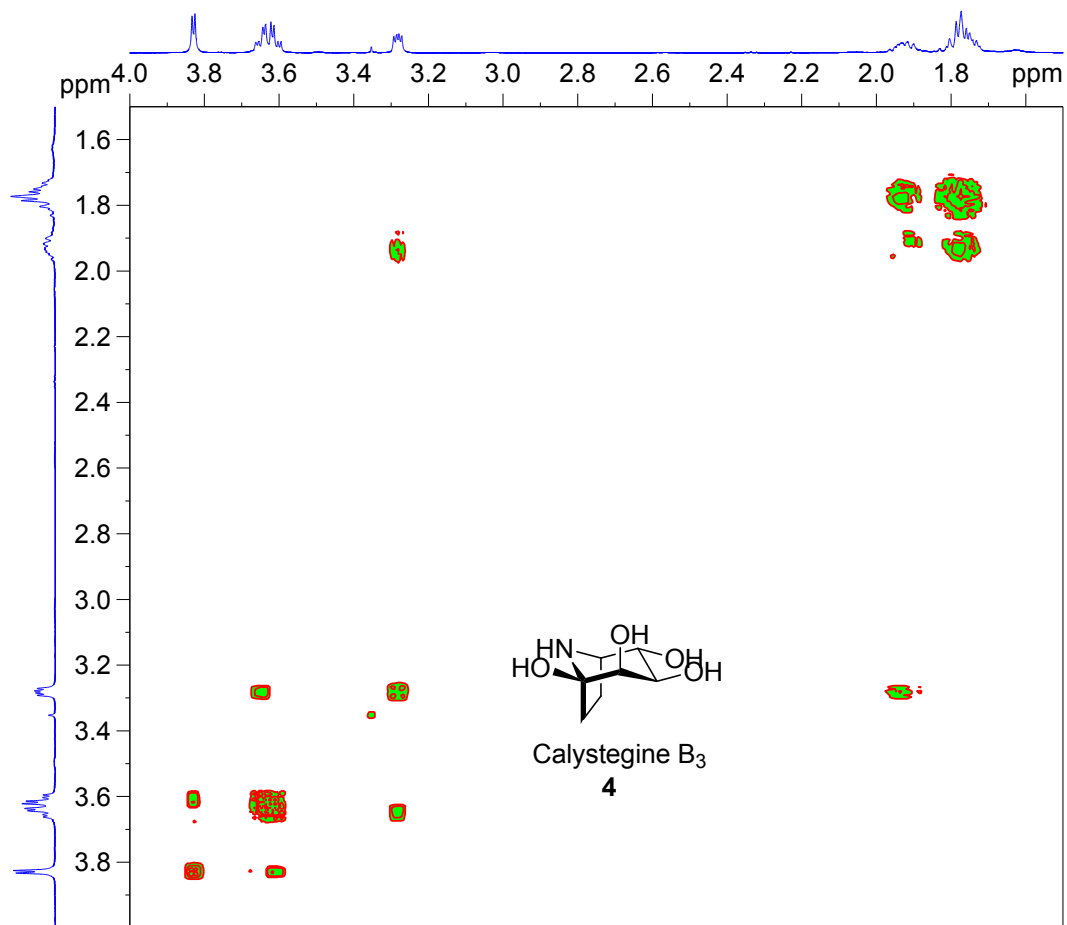


```

NAME          500m 2016
EXPNO         40
PROCNO        1
Date_         20160202
Time          16.25
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zgpg30
TD            65536
SOLVENT       D2O
NS            1024
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            192.89
DW            16.800 usec
DE            18.00 usec
TE            298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

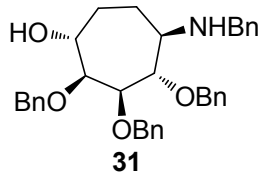
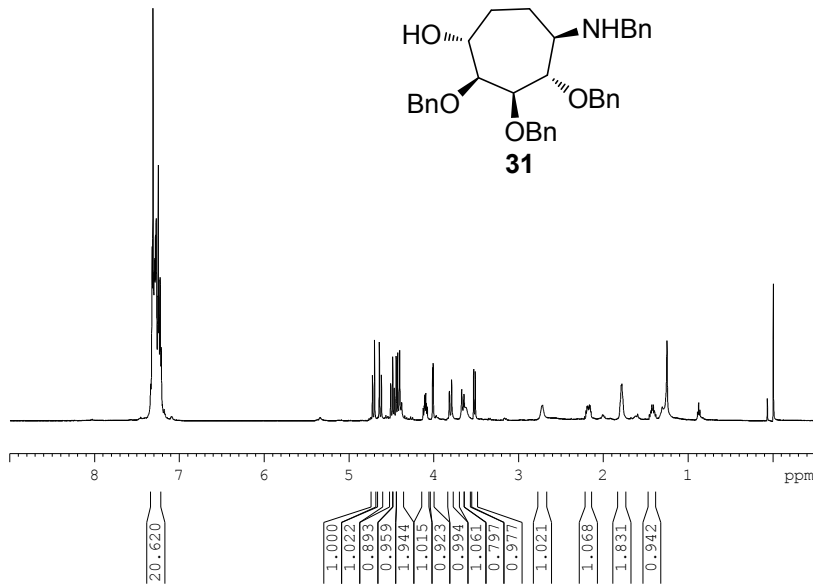
```

===== CHANNEL f1 =====
SFO1          125.7703637 MHz
NUC1          13C
P1            9.80 usec
SI            32768
SF            125.7574222 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



7.330  
7.326  
7.317  
7.308  
7.304  
7.302  
7.297  
7.294  
7.287  
7.282  
7.279  
7.259  
7.254  
7.247  
7.232  
7.219  
4.728  
4.704  
4.647  
4.623  
4.513  
4.490  
4.472  
4.448  
4.430  
4.407  
4.384  
4.111  
4.100  
4.093  
4.018  
4.012  
3.821  
3.795  
3.675  
3.649  
3.534  
3.516  
2.168  
2.161  
1.794  
1.787  
1.433  
1.416

why-312

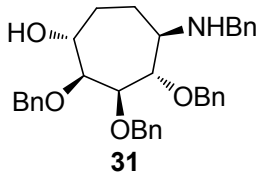
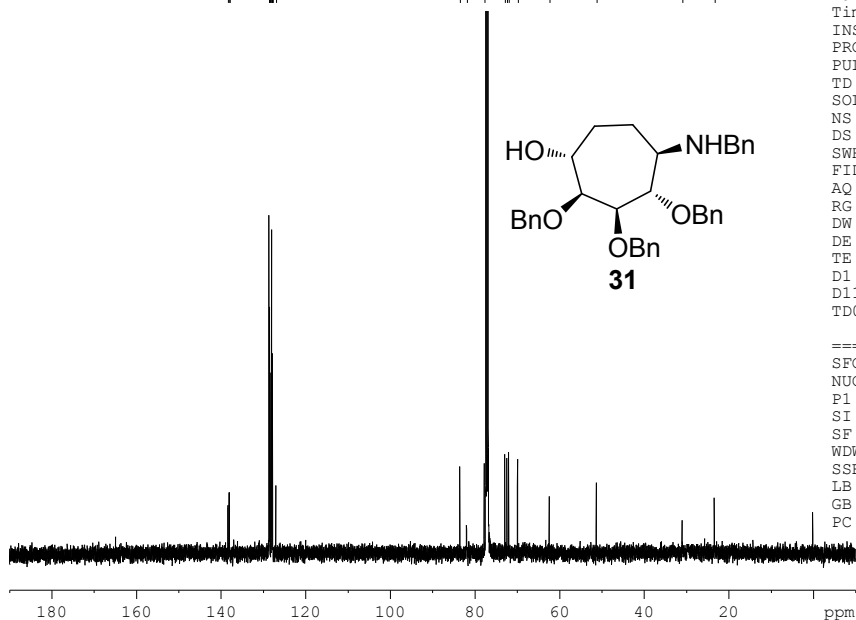


NAME 500m 2016  
EXPNO 14  
PROCNO 1  
Date\_ 20160117  
Time\_ 19.29  
INSTRUM spect  
PROBHD 5 mm CPPBBO BB  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2768500 sec  
RG 55.37  
DW 50.000 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 10.60 usec  
SI 65536  
SF 500.1300154 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

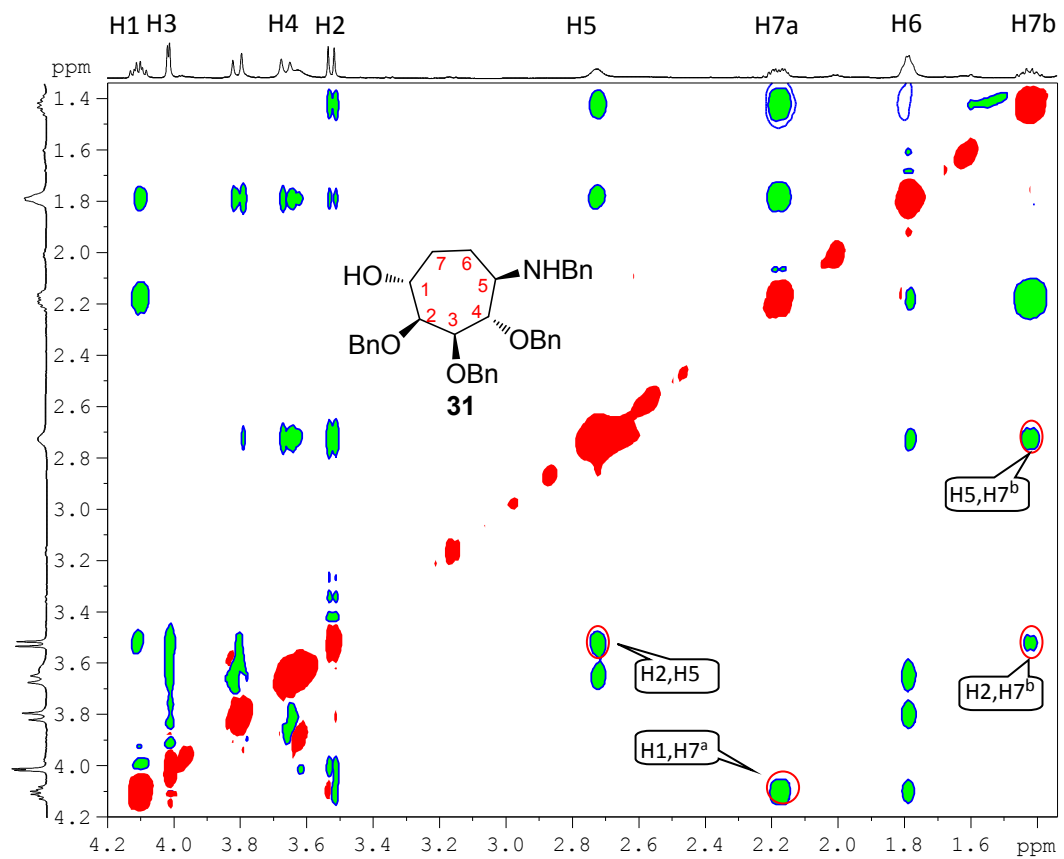
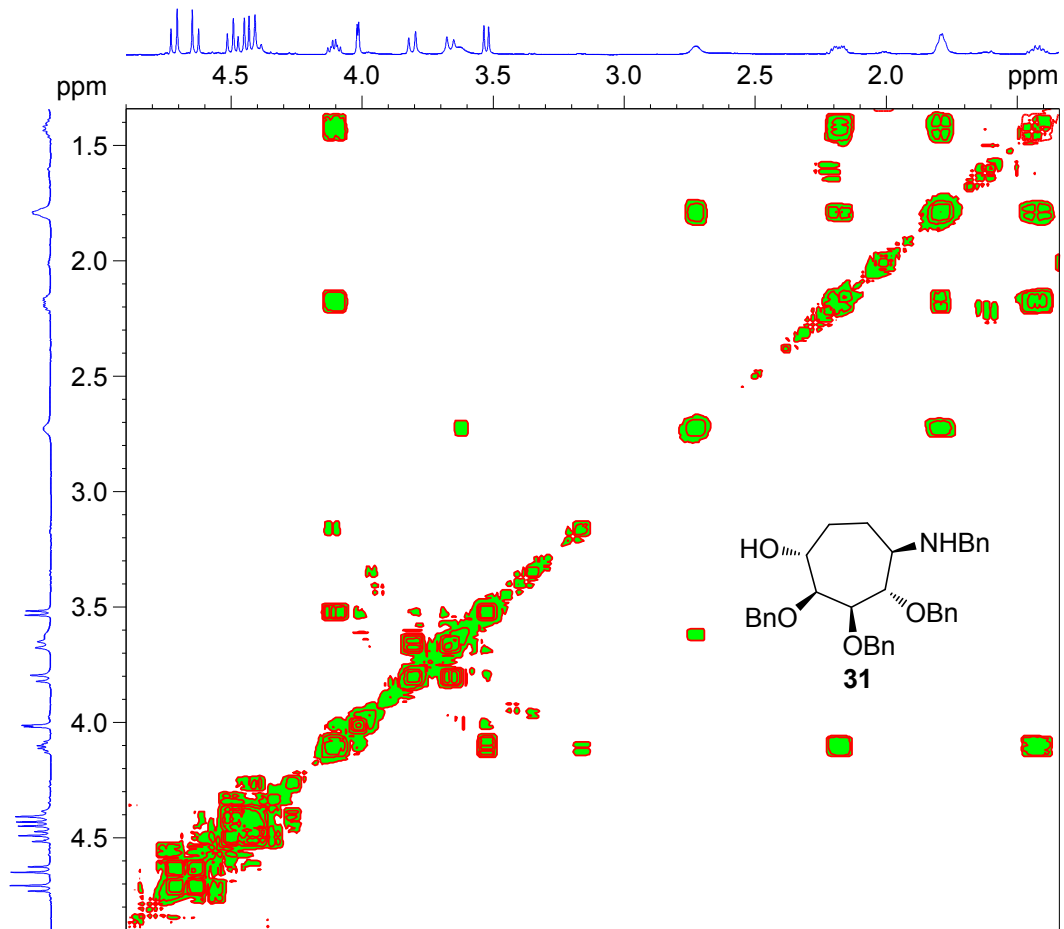
138.266  
137.995  
137.894  
128.567  
128.426  
128.403  
128.128  
127.951  
127.928  
127.906  
127.763  
127.718  
126.932

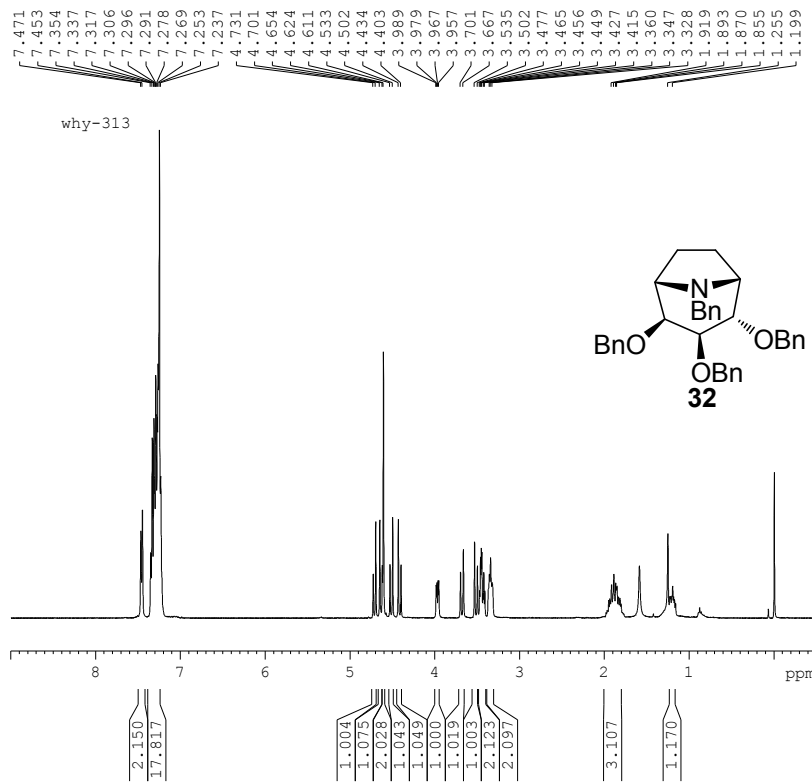
83.441  
81.851  
77.673  
72.837  
72.303  
71.891  
69.796  
62.298  
51.170  
30.878  
23.280



NAME 500M  
EXPNO 77  
PROCNO 1  
Date\_ 20150923  
Time\_ 9.34  
INSTRUM spect  
PROBHD 5 mm CPPBBO BB  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 417  
DS 4  
SWH 29761.904 Hz  
FIDRES 0.454131 Hz  
AQ 1.1010548 sec  
RG 192.89  
DW 16.800 usec  
DE 18.00 usec  
TE 298.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 125.7703637 MHz  
NUC1 13C  
P1 9.80 usec  
SI 32768  
SF 125.7577717 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40





```

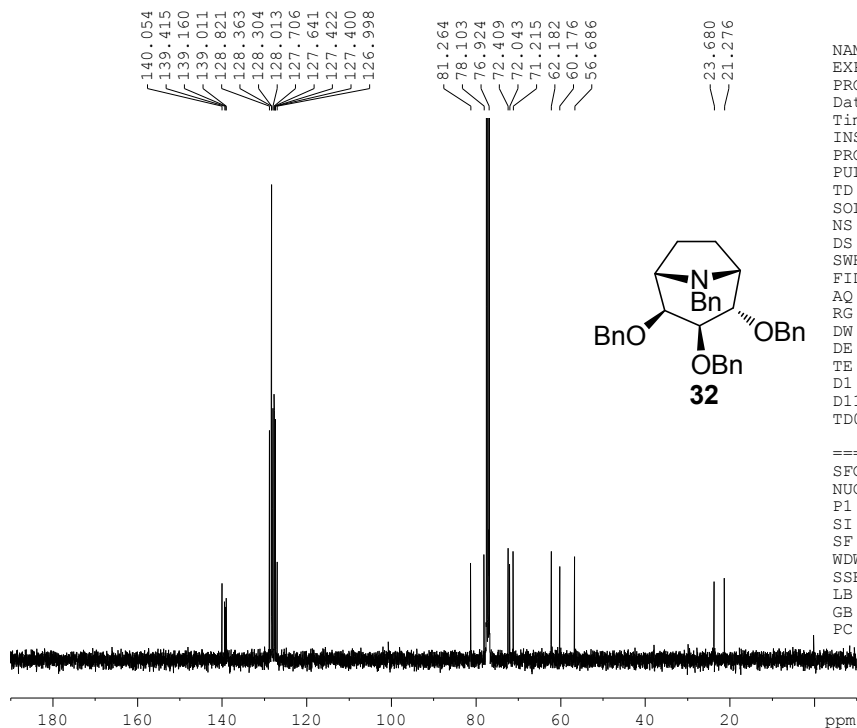
NAME          400MHZ
EXPNO         518
PROCNO        1
Date_         20151016
Time_         17.59
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            0
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            2.0447731 sec
RG            206.33
DW            62.400 usec
DE            6.50 usec
TE            298.7 K
D1            2.00000000 sec
D11           1
TD0           1

```

```

===== CHANNEL f1 =====
SFO1          400.2424716 MHz
NUC1           1H
P1            14.80 usec
SI            65536
SF            400.2400126 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```



```

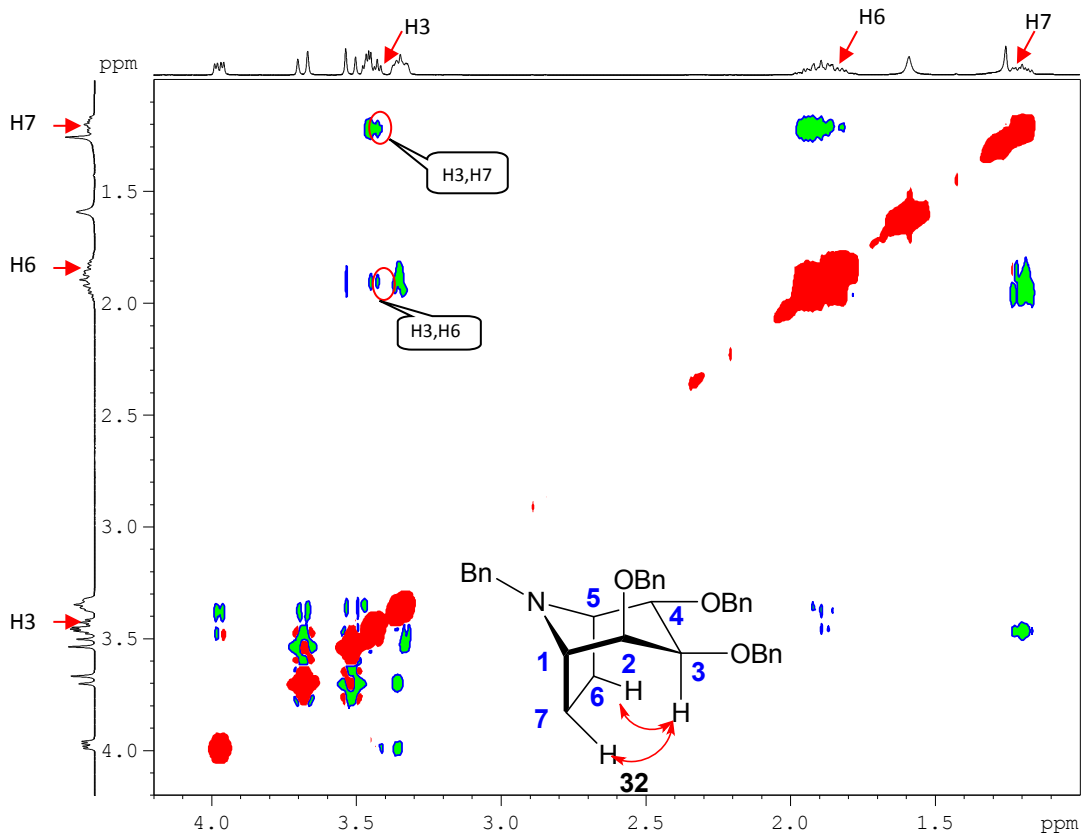
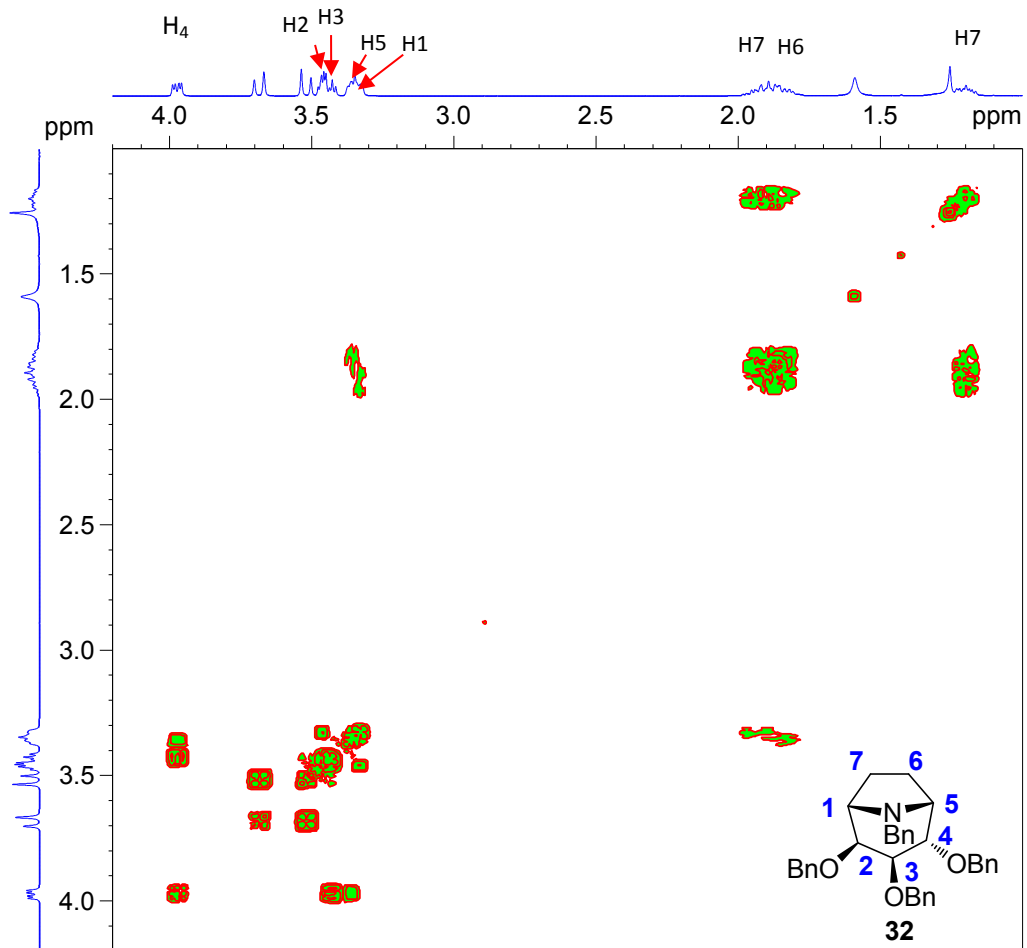
NAME          400MHZ
EXPNO         519
PROCNO        1
Date_         20151016
Time_         18.03
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            477
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            206.33
DW            20.800 usec
DE            6.50 usec
TE            299.7 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            100.6404148 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

```



## 2. Results of the bioassay

The enzymes  $\alpha$ -glucosidases (from yeast, rice and rat intestinal maltase),  $\beta$ -glucosidases (from almond and Human lysosome),  $\alpha$ -galactosidase (from coffee beans),  $\beta$ -galactosidase (from bovine liver),  $\alpha$ -mannosidase (from jack beans),  $\beta$ -mannosidase (from snail),  $\alpha$ -L-fucosidase (from bovine kidney),  $\alpha,\alpha$ -trehalase (from porcine kidney), amyloglucosidase (from *Aspergillus niger*),  $\alpha$ -L-rhamnosidase (from *Penicillium decumbens*) and  $\beta$ -glucuronidase (from *E. coli* and Bovine liver) were purchased from Sigma-Aldrich Chemical Co. (St. Louis, Mo. USA). Brush border membranes prepared from rat small intestine according to the method of Kessler et al.<sup>1</sup> were assayed at pH 5.8 for rat intestinal maltase using maltose. The released D-glucose was determined colorimetrically using the Glucose CII-test Wako (Wako Pure Chemical Ind.; Osaka, Japan). Other glycosidase activities were determined using an appropriate p-nitrophenyl glycoside as substrate. The reaction was stopped by adding 400 mM Na<sub>2</sub>CO<sub>3</sub>. The released p-nitrophenol was measured spectrometrically at 400 nm. The assay results are summarized in the following Table.

**Table 1.** Glycosidase inhibition of Calystegine B<sub>2</sub> (**3**) and Calystegine B<sub>3</sub> (**4**)

Enzyme	IC <sub>50</sub> ( $\mu$ M)	
	calystegine B <sub>2</sub> ( <b>3</b> )	calystegine B <sub>3</sub> ( <b>4</b> )
$\alpha$ -glucosidase		
Yeast	NI <sup>a</sup> (3.2%) <sup>b</sup>	NI (14.6%)
Rice	NI (33.5%)	154
Rat intestinal	NI (25.1%)	55
$\beta$ -glucosidase		
Almond	5.3	84
Human lysosome	5.1	616
$\alpha$ -Galactosidase		
Coffee beans	3.2	73
$\beta$ -Galactosidase		
Bovine liver	383	71
$\alpha$ -Mannosidase		
Jack bean	NI (0%)	NI (6.6%)
$\beta$ -Mannosidase		
Snail	NI (0%)	NI (2.0%)
$\alpha$ -L-Fucosidase		
Bovine kidney	NI (0.1%)	NI (2.7%)
$\alpha,\alpha$ -Trehalase		
Porcine kidney	19	384
Amyloglucosidase		
<i>Aspergillus niger</i>	NI (0%)	NI (8.2%)
$\alpha$ -L-Rhamnosidase		
<i>Penicillium</i>	NI (47.7%)	NI (13.4%)
$\beta$ -glucuronidase		
<i>E. coli</i>	NI (6.9%)	NI (8.8%)
Bovine liver	NI (0%)	NI (4.1%)

<sup>a</sup>NI : no inhibition (less than 50% inhibition at 1000  $\mu$ M).

<sup>b</sup>( ) : inhibition at 1000  $\mu$ M.

(1) Kessler, M.; Acuto, O.; Storelli, C.; Murer, H.; Semenza, G. A. *Biochim. Biophys. Acta*, **1978**, *506*, 136.



### 3. Comparison of $^{13}\text{C}$ NMR of synthetic calystegine **B<sub>2</sub> (3)** and **B<sub>3</sub> (4)** with natural products

**Table 1** Comparison of the synthetic calystegine **B<sub>2</sub> (3)** with natural products

calystegine <b>B<sub>2</sub></b> (lit.) <sup>a</sup>	93.2	80.4	77.7	77.6	58.6	31.5	24.5
Calystegine <b>B<sub>2</sub></b> (synthetic) <sup>b</sup>	93.2	80.4	77.7	77.6	58.6	31.5	24.5
$\Delta\delta$	0.0	0.0	0.0	0.0	0.0	0.0	0.0

a)  $\delta$  in  $\text{D}_2\text{O}$  at 100MHz. b)  $\delta$  in  $\text{D}_2\text{O}$  at 100MHz.

**Table 2** Comparison of the synthetic calystegine **B<sub>3</sub> (4)** with natural products

calystegine <b>B<sub>3</sub></b> (lit.) <sup>a</sup>	93.0	77.2	75.2	73.0	58.3	34.1	23.0
Calystegine <b>B<sub>3</sub></b> (synthetic) <sup>b</sup>	93.1	77.2	75.1	73.0	58.3	34.0	23.0
$\Delta\delta$	0.1	0.0	-0.1	0.0	0.0	-0.1	0.0

a)  $\delta$  in  $\text{D}_2\text{O}$  at 100MHz. b)  $\delta$  in  $\text{D}_2\text{O}$  at 125MHz.