

## Electronic Supplementary Information

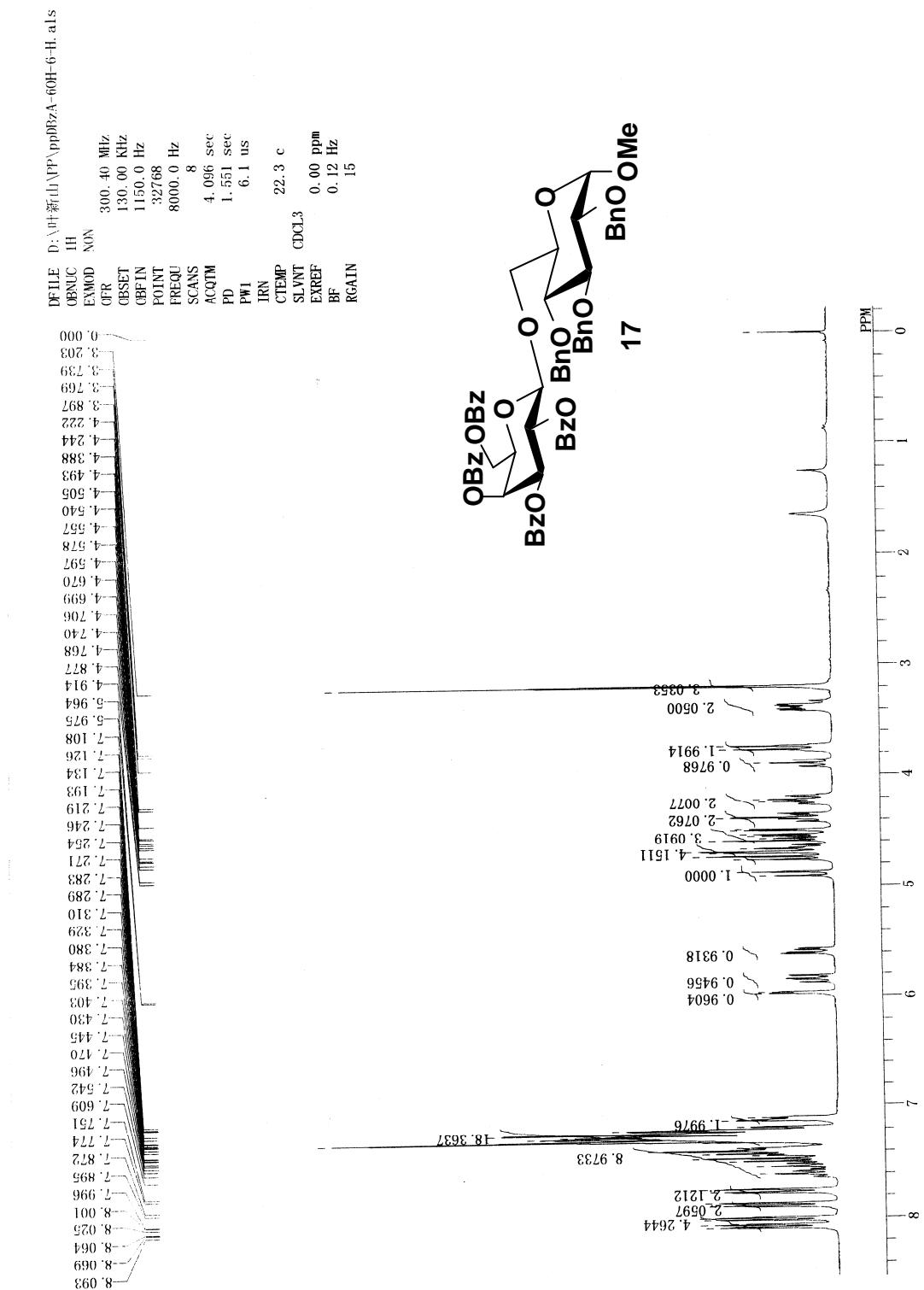
### ***O,O-Dimethylthiophosphonosulfenyl Bromide-Silver Triflate: A New Powerful Promoter System for the Preactivation of Thioglycosides***

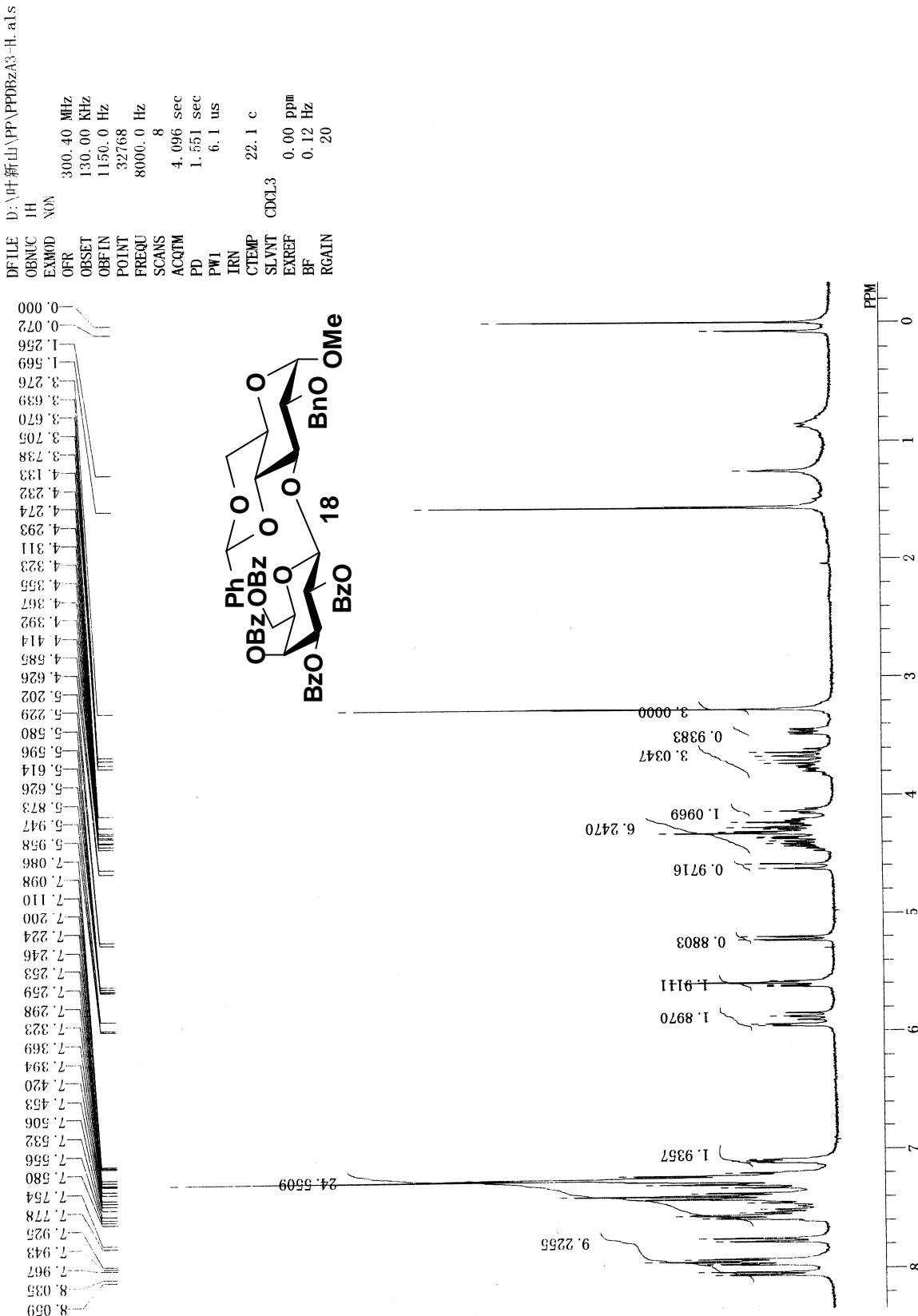
Peng Peng and Xin-Shan Ye\*

*The State Key Laboratory of Natural and Biomimetic Drugs, Peking University, and School of Pharmaceutical Sciences, Peking University, Xue Yuan Rd No. 38, Beijing 100191, People's Republic of China*  
e-mail: [xinshan@bjmu.edu.cn](mailto:xinshan@bjmu.edu.cn)

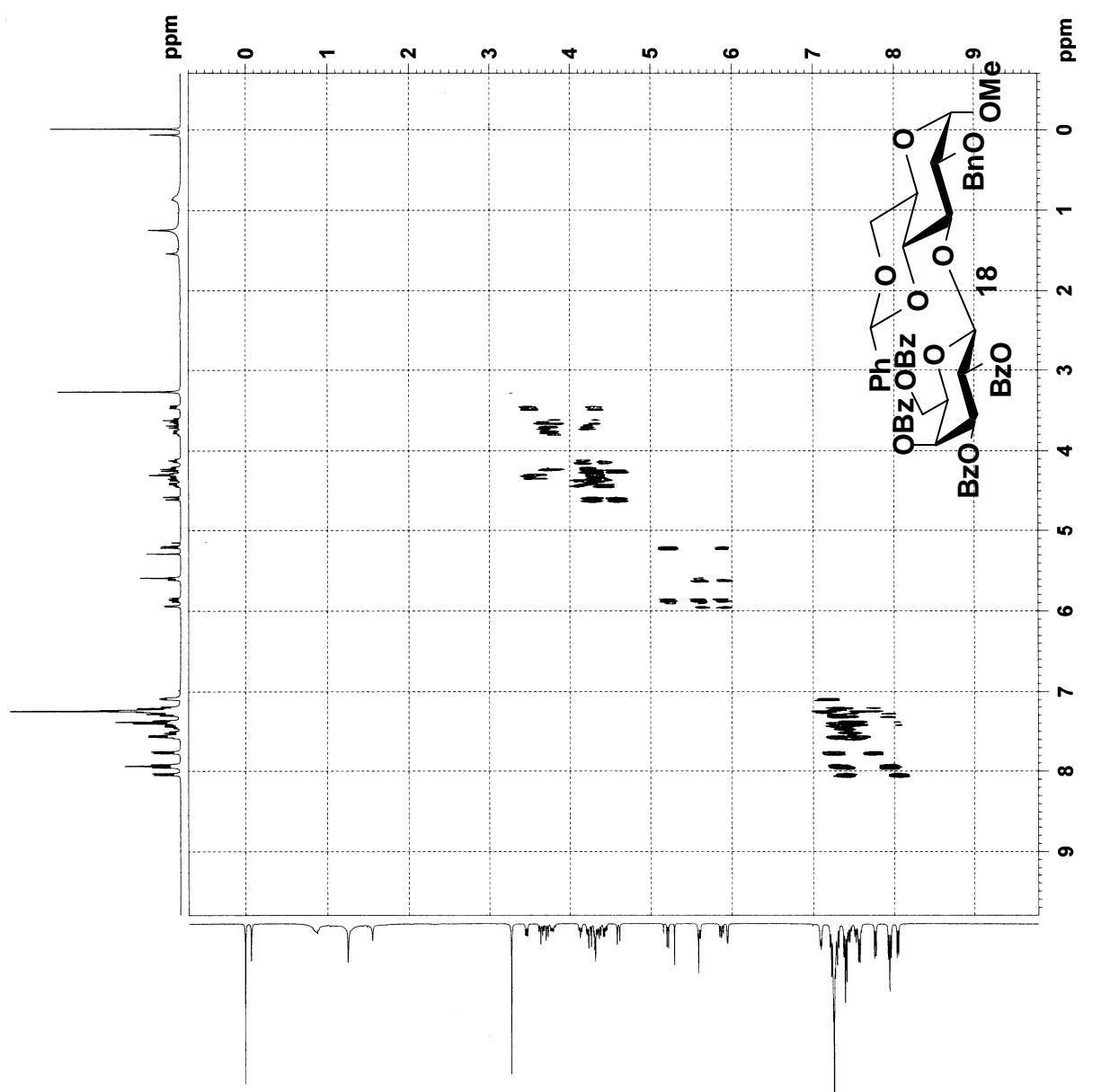
#### **Table of Contents:**

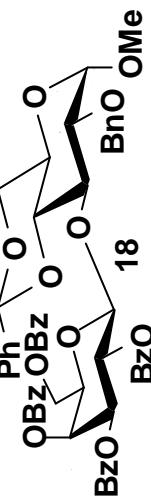
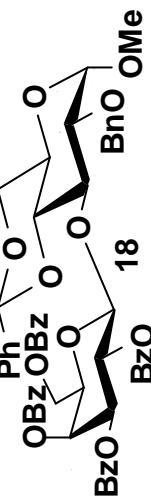
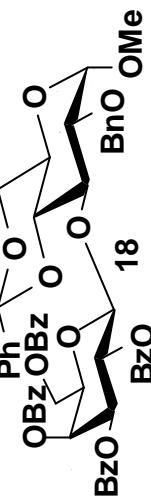
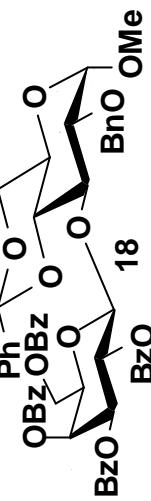
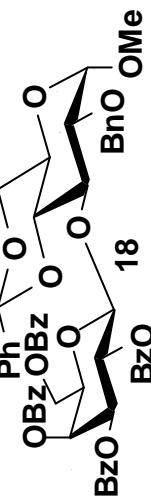
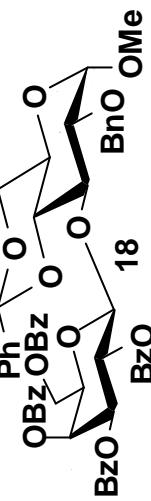
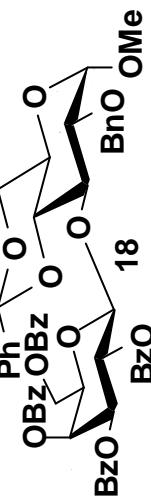
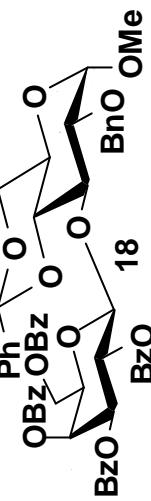
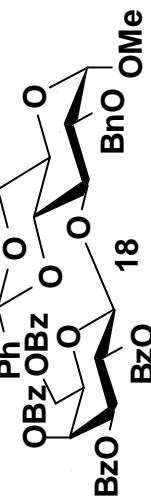
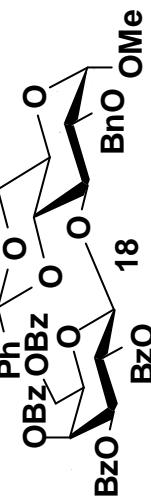
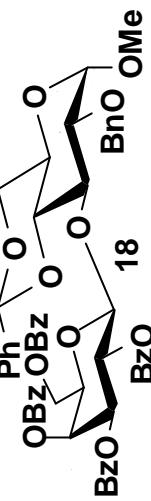
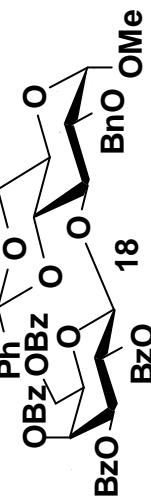
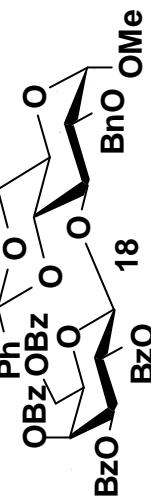
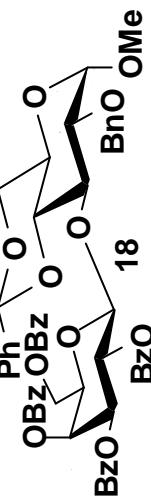
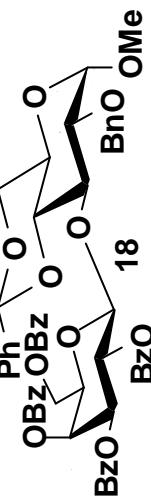
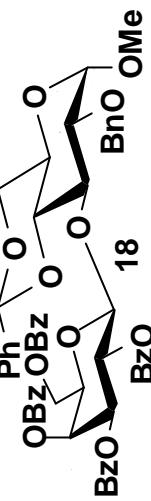
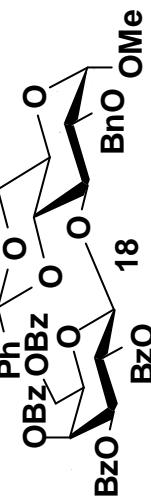
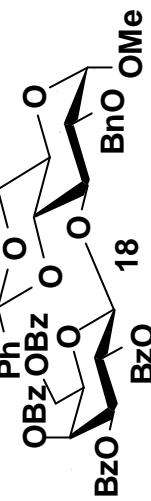
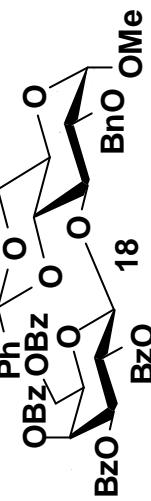
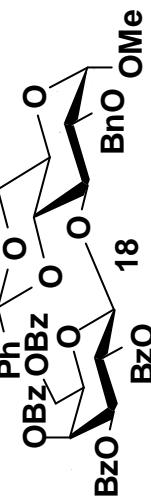
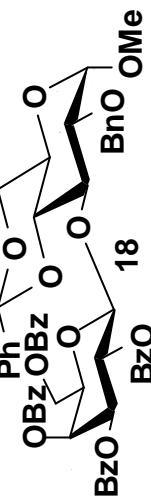
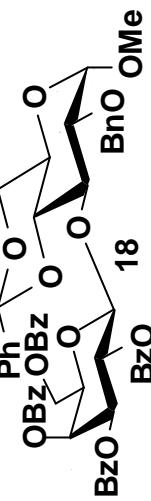
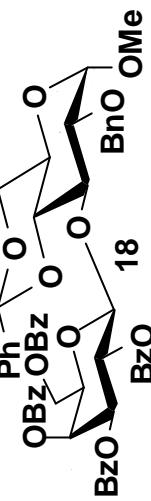
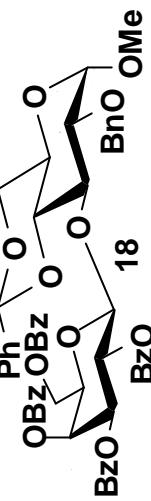
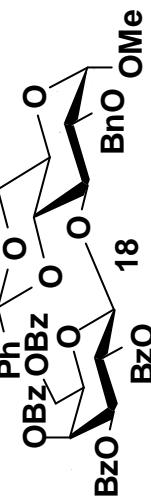
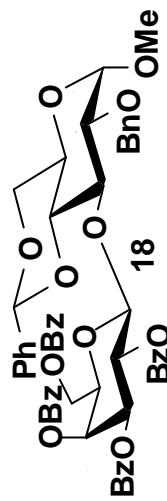
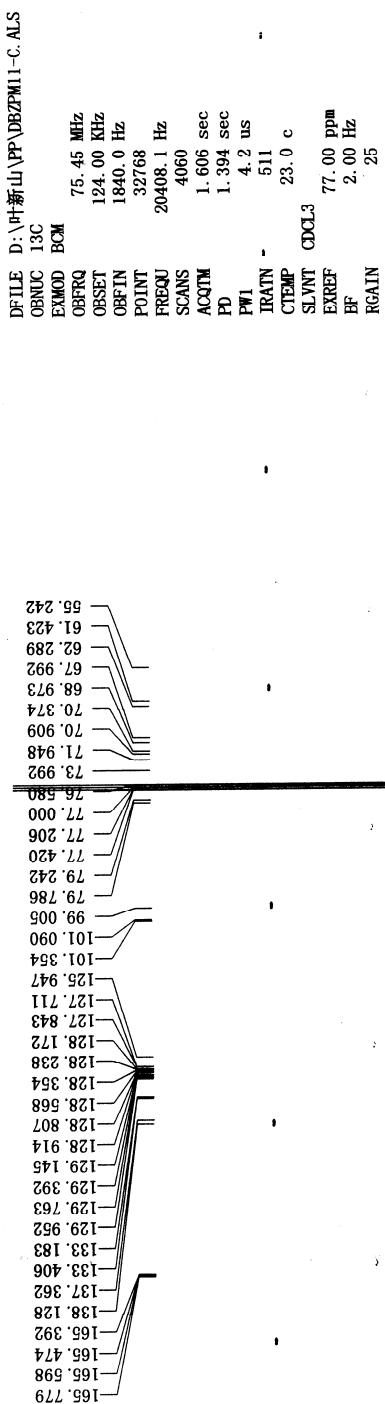
<sup>1</sup> H NMR Spectrum of Compound 17.....	S2
<sup>1</sup> H, COSY, <sup>13</sup> C, and HSQC NMR Spectra of Compound 18.....	S3-S6
<sup>1</sup> H NMR Spectrum of Compound 19.....	S7
<sup>1</sup> H NMR Spectrum of Compound 20.....	S8
<sup>1</sup> H NMR Spectrum of Compound 21.....	S9
<sup>1</sup> H NMR Spectrum of Compound 22.....	S10
<sup>1</sup> H NMR Spectrum of Compound 23.....	S11
<sup>1</sup> H NMR Spectra of Compounds 24 $\alpha$ and 24 $\beta$ .....	S12-S13
<sup>1</sup> H, COSY, <sup>13</sup> C, and HSQC NMR Spectra of Compound 25.....	S14-S17
<sup>1</sup> H NMR Spectrum of Compound 26.....	S18
<sup>1</sup> H, COSY, <sup>13</sup> C, and HSQC NMR Spectra of Compound 27.....	S19-S22
<sup>1</sup> H, COSY, <sup>13</sup> C, and HSQC NMR Spectra of Compound 28.....	S23-S26
<sup>1</sup> H, COSY, <sup>13</sup> C, and HSQC NMR Spectra of Compound 29.....	S27-S30
<sup>1</sup> H, COSY, <sup>13</sup> C, and HSQC NMR Spectra of Compound 30.....	S31-S34

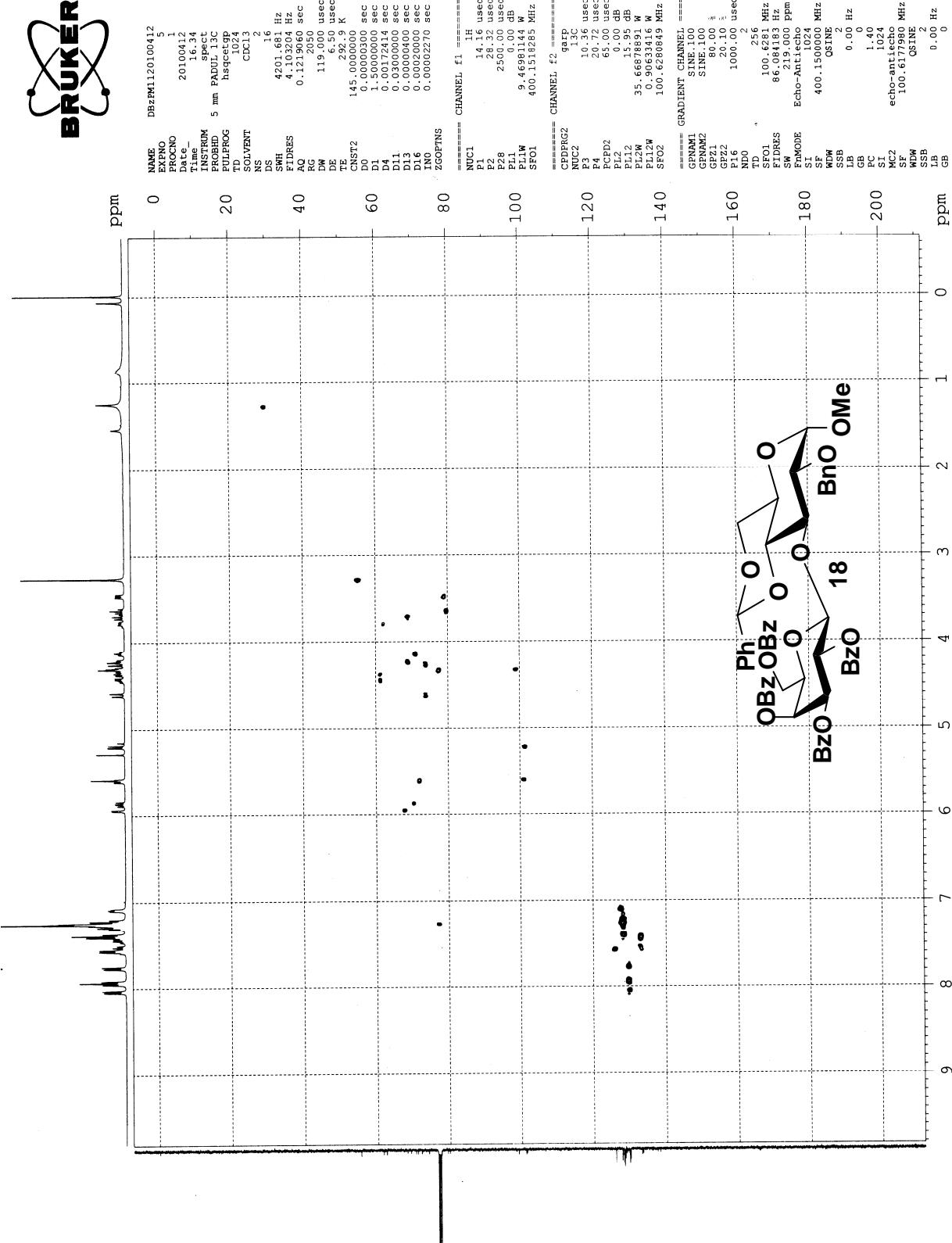




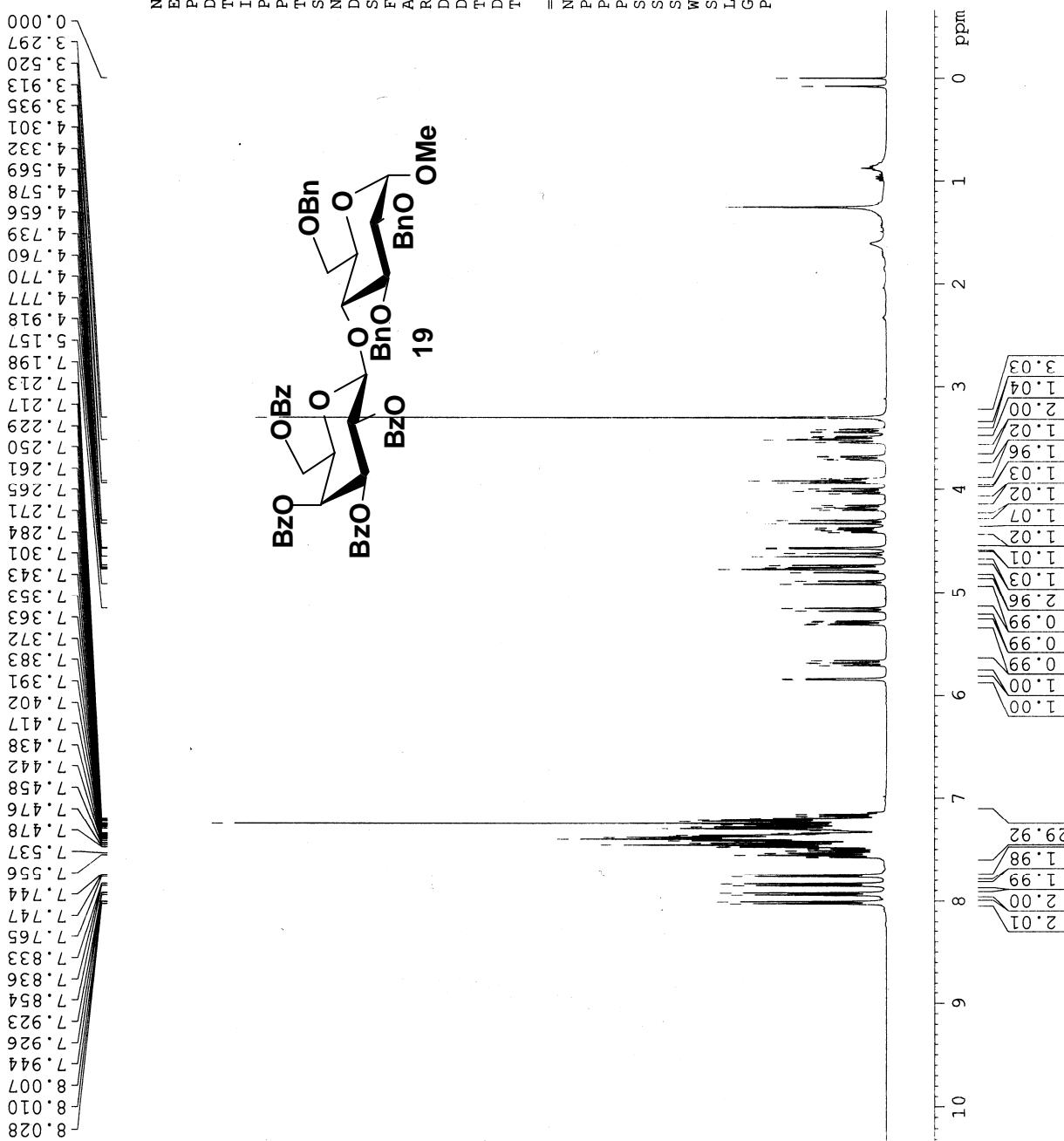
The Bruker logo consists of the word "BRUKER" in a bold, black, sans-serif font. The letters are oriented vertically, with the "B" at the bottom and the "R" at the top. Two thick, black, curved lines resembling orbits or magnetic field lines are positioned behind the letters, intersecting at the top and bottom.





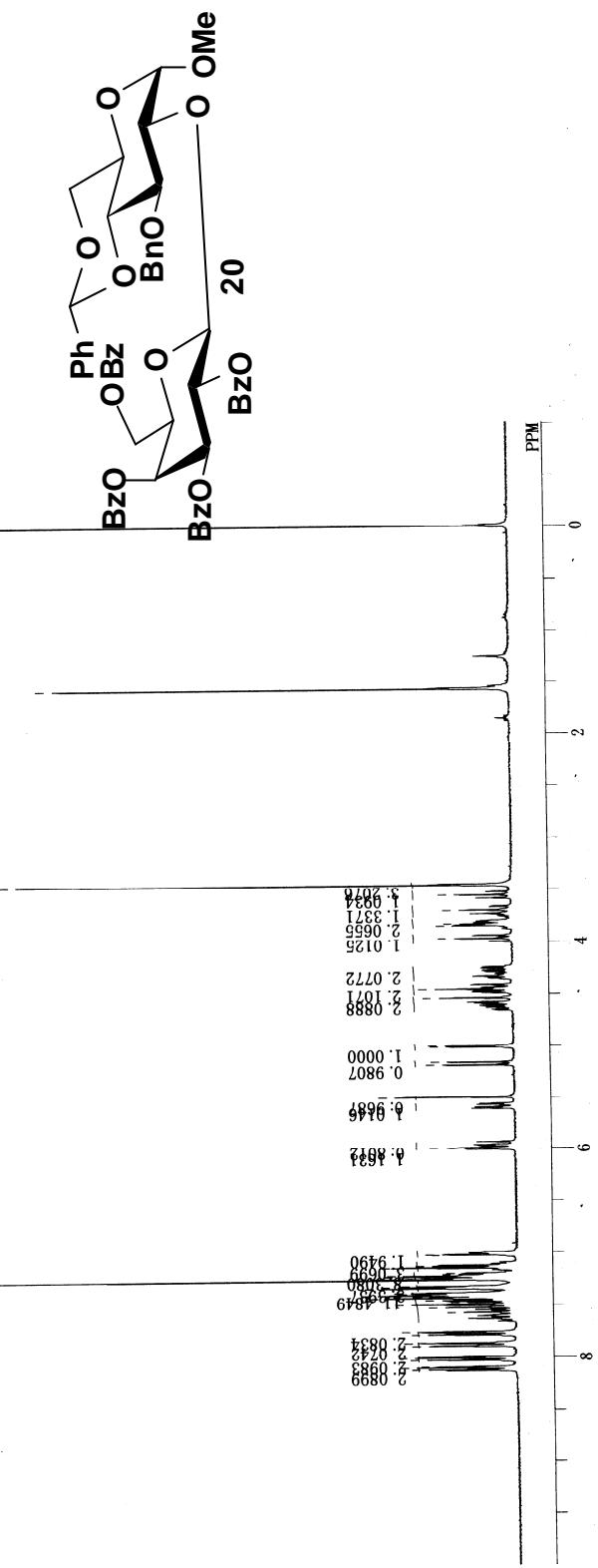


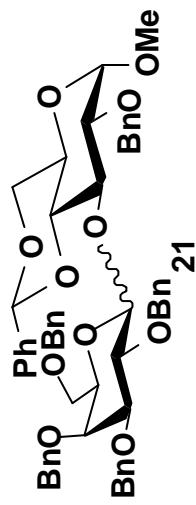
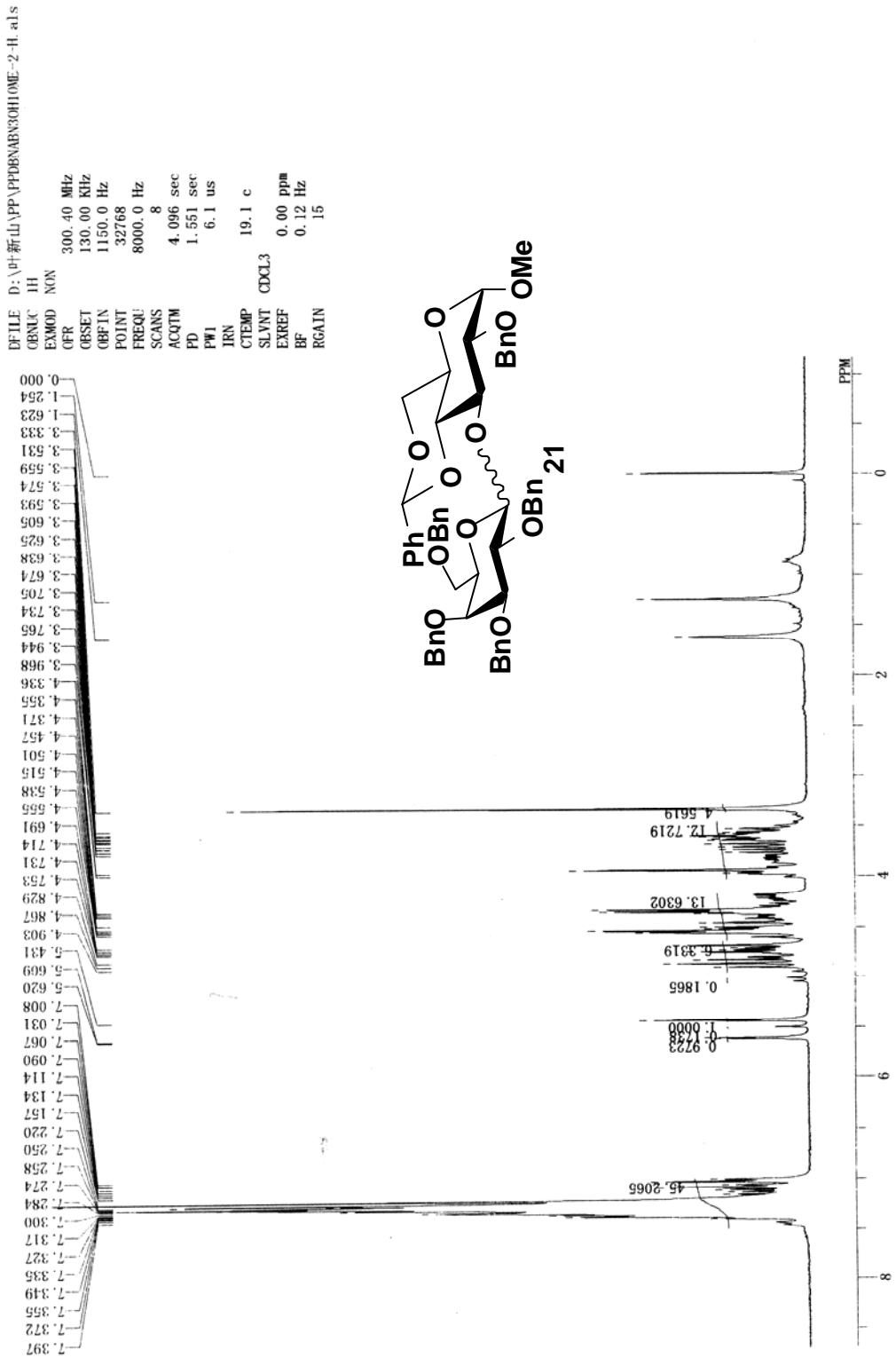
The Bruker logo consists of the word "BRUKER" in a bold, sans-serif font, oriented vertically. The letters are partially obscured by two stylized, intersecting elliptical arcs that resemble atomic orbits or magnetic field lines.



DFILE D:\叶\新山\PP\PPBZ2-0H--2Hals.als

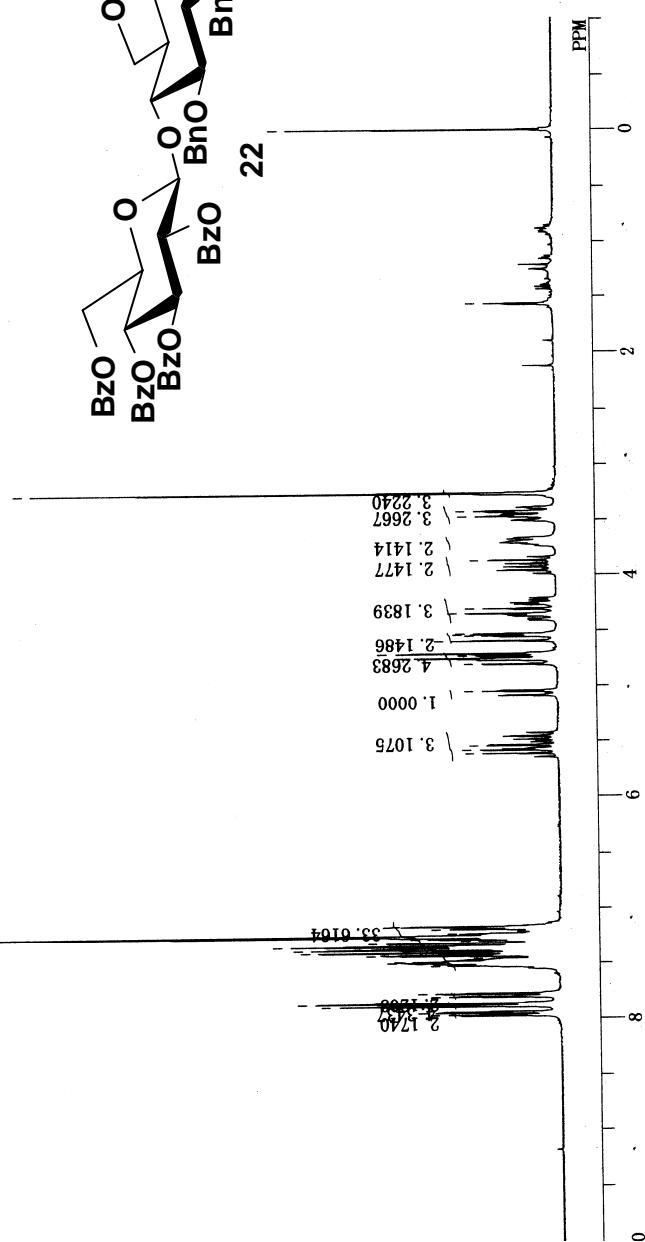
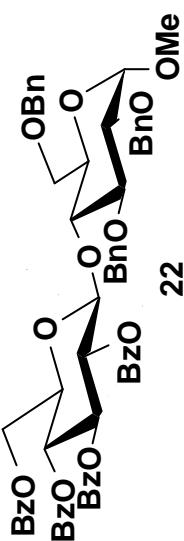
Parameter	Value
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OSETRQ	130.00 kHz
OSSET	1150.0 Hz
POINT	32768
FREQU	8000.0 Hz
SCANS	16
ACQTM	4.096 sec
PD	1.551 sec
PW1	6.1 us
IRATN	511
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	

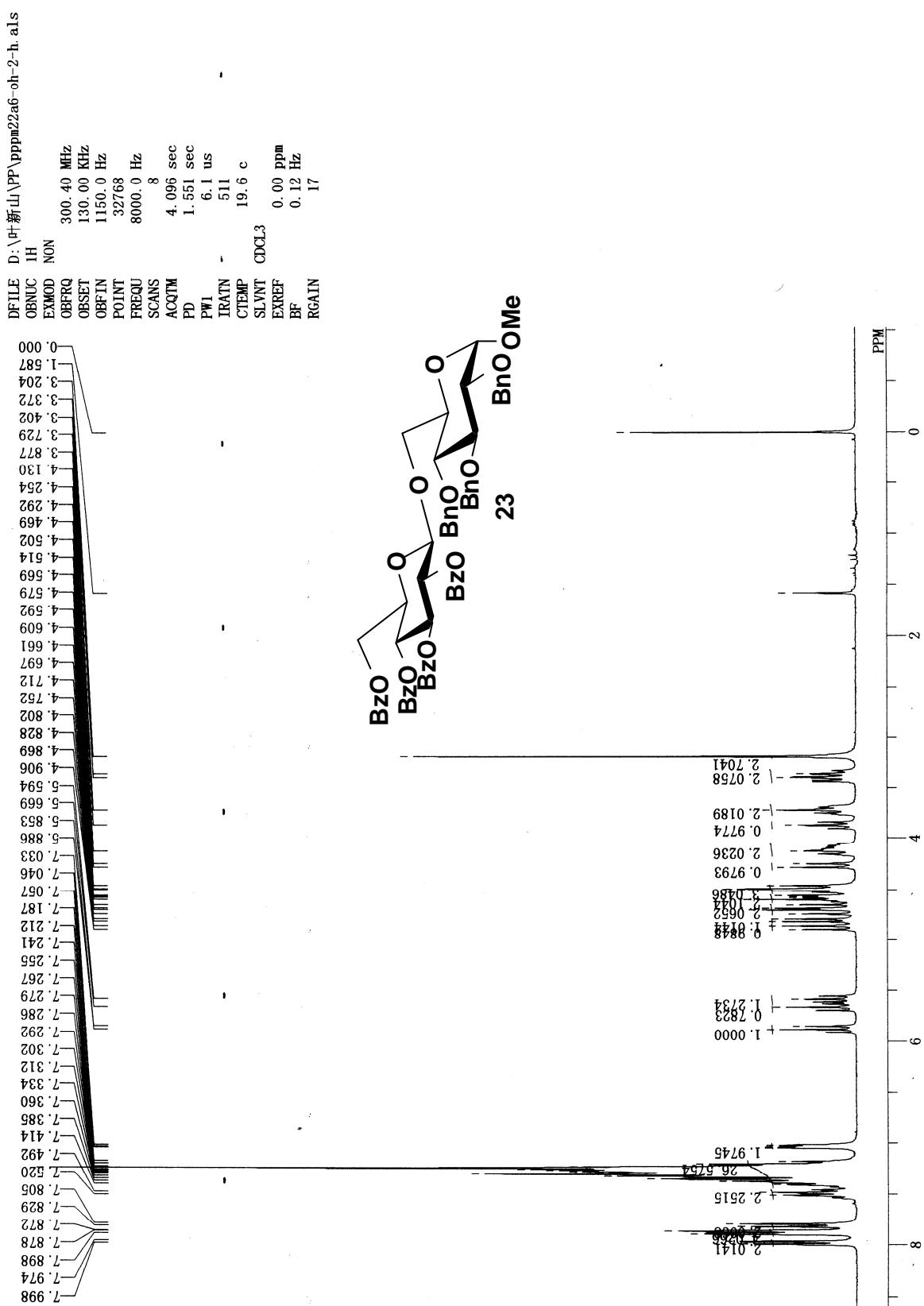




D:\1H\新山\PPV\PPV22PM13-2-H.ALS

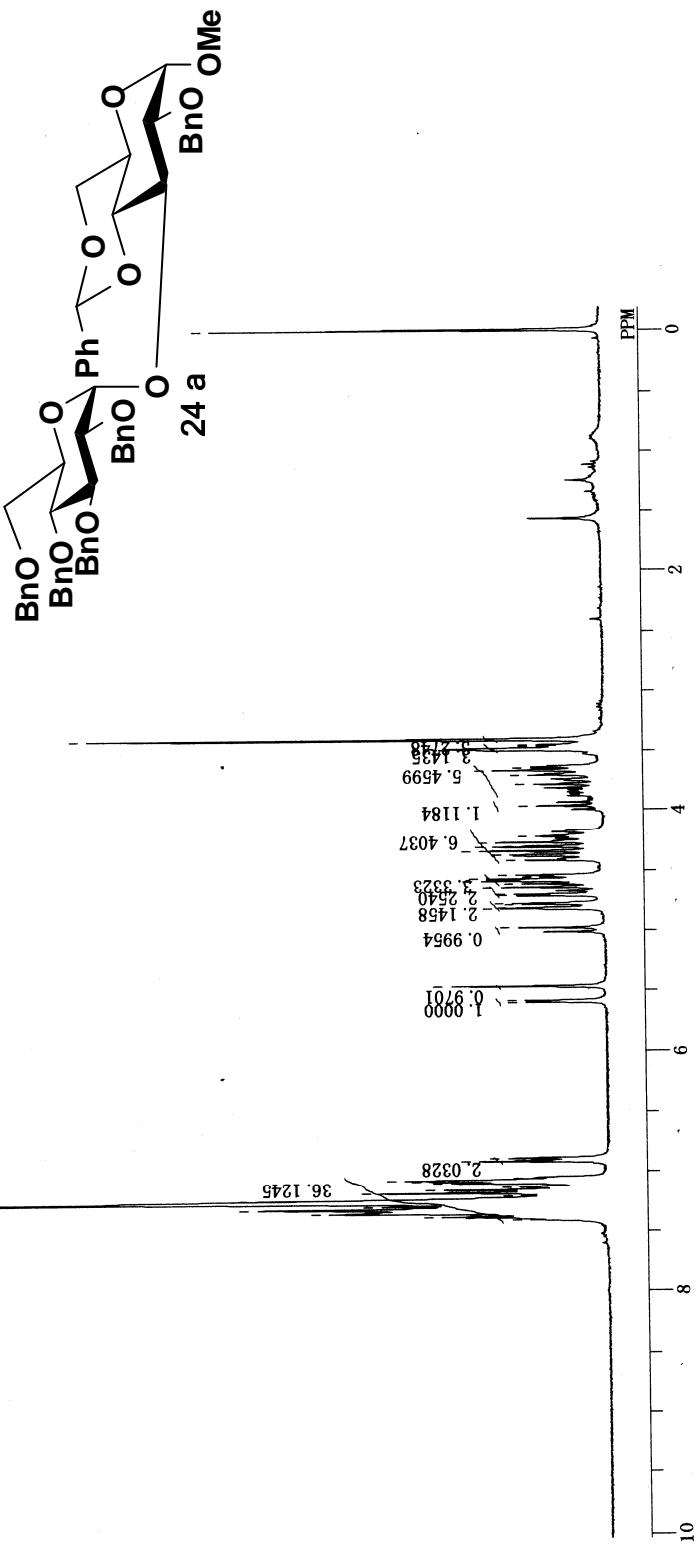
Chemical Shift (ppm)	Peak Description
0.00	TMS reference
1.571	sharp peak
2.273	sharp peak
3.431	sharp peak
3.474	sharp peak
3.871	sharp peak
4.135	sharp peak
4.356	sharp peak
4.536	sharp peak
4.548	sharp peak
4.560	sharp peak
4.600	sharp peak
4.722	sharp peak
4.743	sharp peak
4.764	sharp peak
4.776	sharp peak
4.814	sharp peak
5.049	sharp peak
5.538	sharp peak
5.582	sharp peak
5.613	sharp peak
7.171	sharp peak
7.175	sharp peak
7.195	sharp peak
7.243	sharp peak
7.267	sharp peak
7.287	sharp peak
7.301	sharp peak
7.326	sharp peak
7.352	sharp peak
7.374	sharp peak
7.383	sharp peak
7.409	sharp peak
7.430	sharp peak
7.436	sharp peak
7.463	sharp peak
7.472	sharp peak
7.479	sharp peak
7.489	sharp peak
7.497	sharp peak
7.503	sharp peak
7.514	sharp peak
7.521	sharp peak
7.527	sharp peak
7.569	sharp peak
7.714	sharp peak
7.797	sharp peak
7.857	sharp peak
7.862	sharp peak
7.886	sharp peak
7.941	sharp peak
7.945	sharp peak

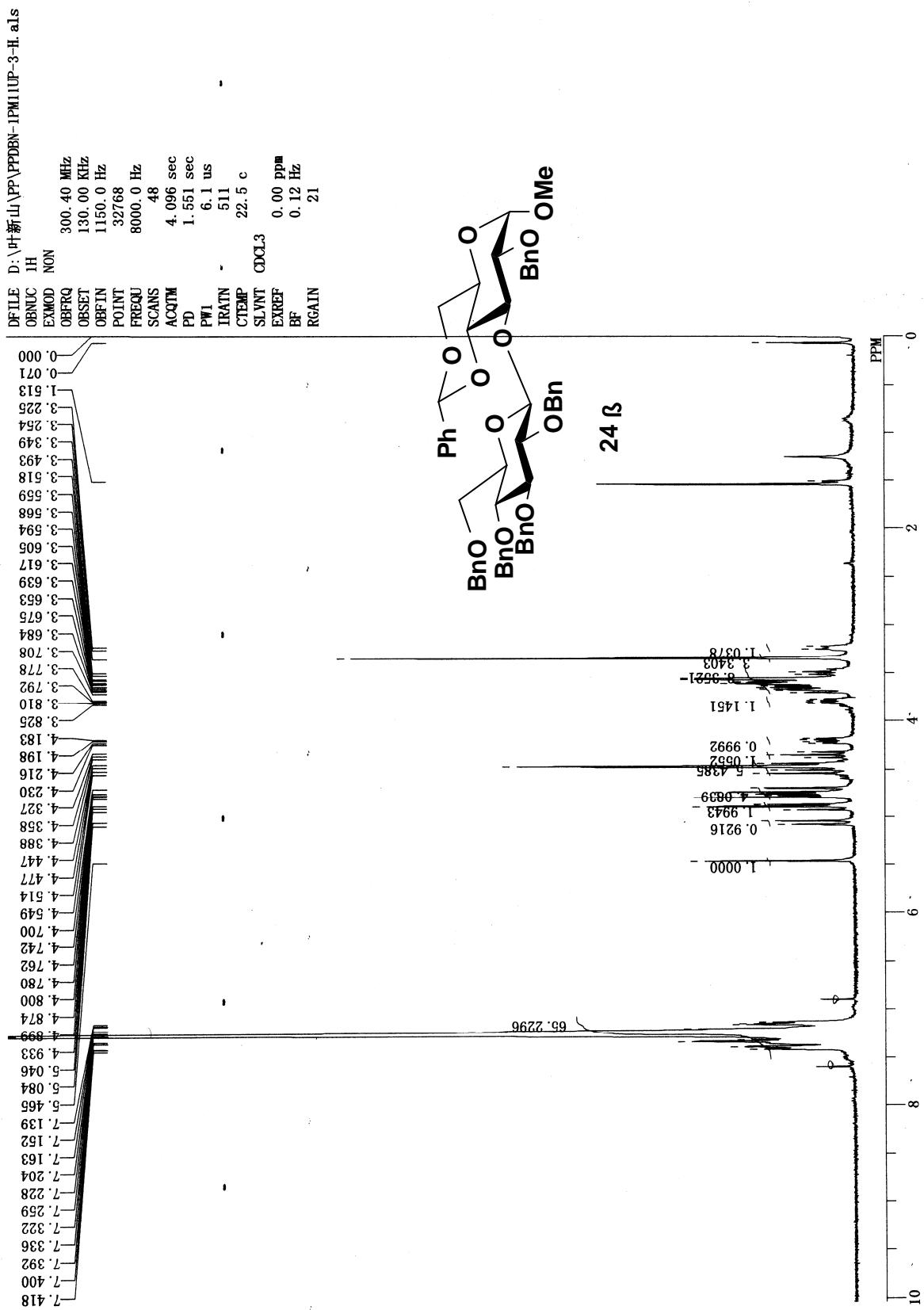


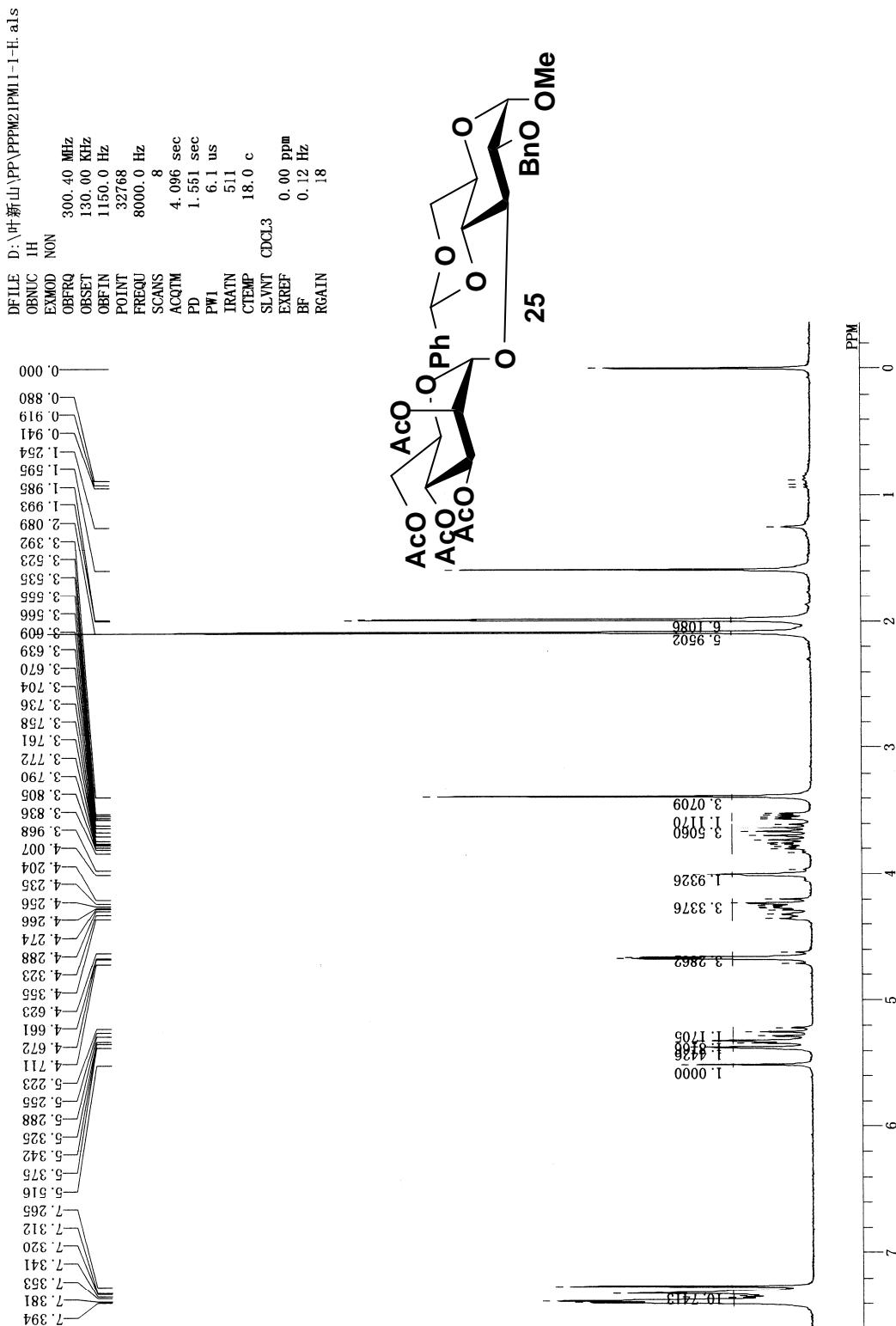


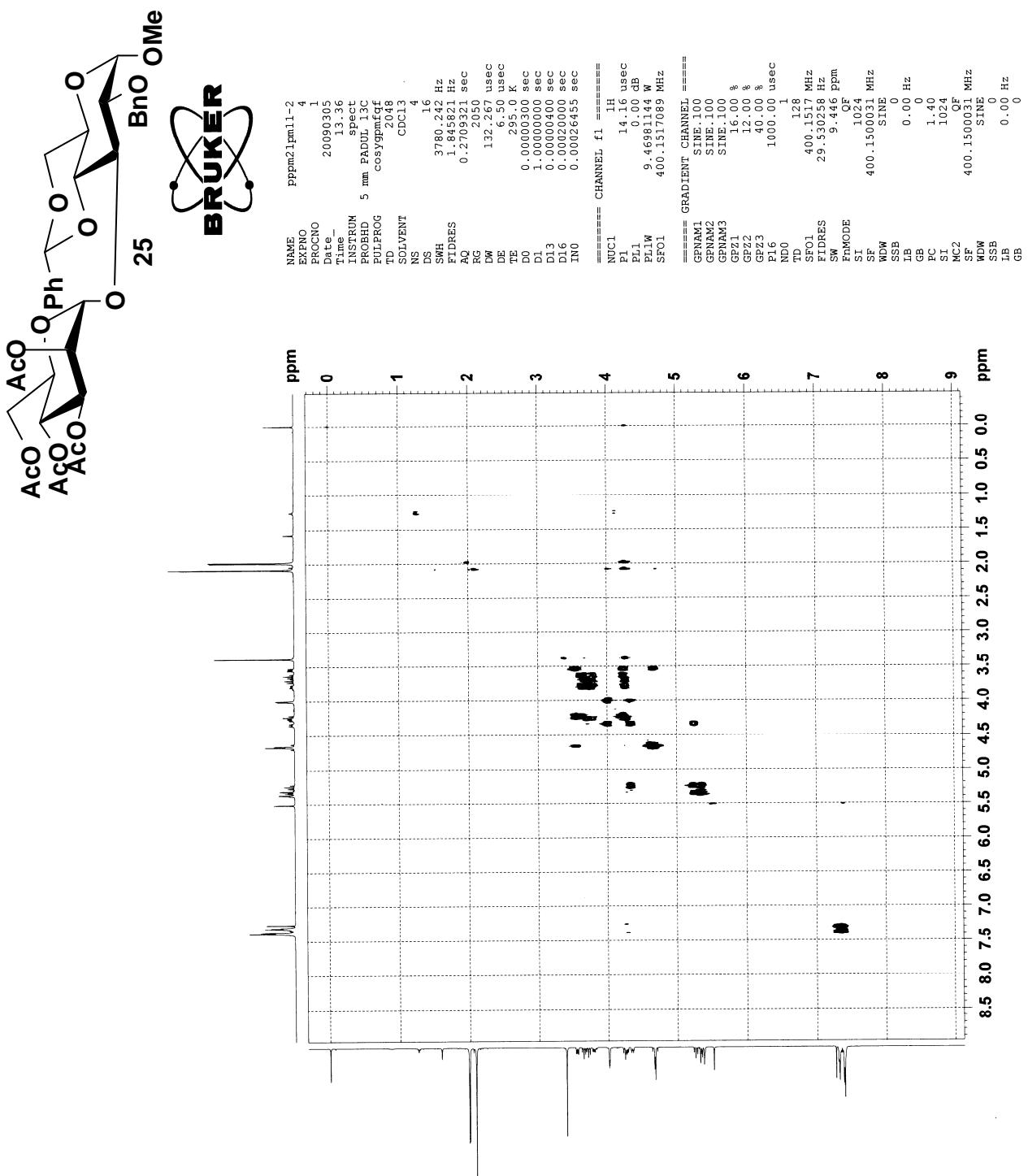
D:\叶新山\PP\PPdBm-1pM1XIA.als

Frequency (Hz)	Power (dBm)
0.000	0.000
0.412	3.412
0.824	3.400
1.236	3.389
1.406	-4.000
1.718	3.367
2.130	3.352
2.542	3.328
2.954	3.313
3.366	3.295
3.778	3.258
4.190	3.239
4.592	3.207
4.994	3.184
5.396	3.163
5.798	3.141
6.200	3.119
6.592	3.081
6.994	3.057
7.396	3.023
7.798	3.000
8.199	2.967
8.599	2.934
8.999	2.896
9.399	2.857
9.799	2.818
10.199	2.779
10.599	2.741
10.999	2.699
11.399	2.657
11.799	2.614
12.199	2.566
12.599	2.518
12.999	2.466
13.399	2.414
13.799	2.361
14.199	2.309
14.599	2.258
14.999	2.207
15.399	2.164
15.799	2.134
16.199	2.091
16.599	2.057
16.999	2.023
17.399	1.986
17.799	1.953
18.199	1.919
18.599	1.884

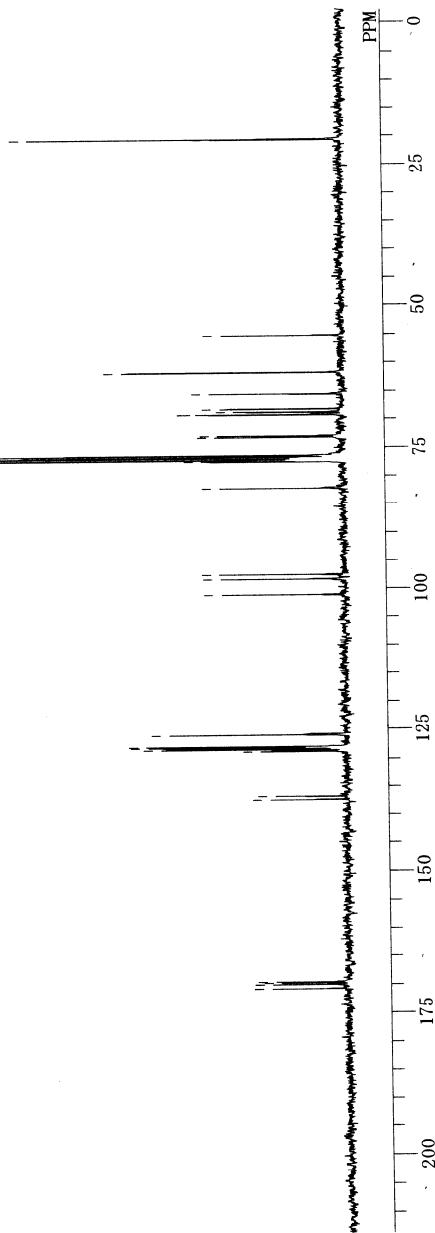
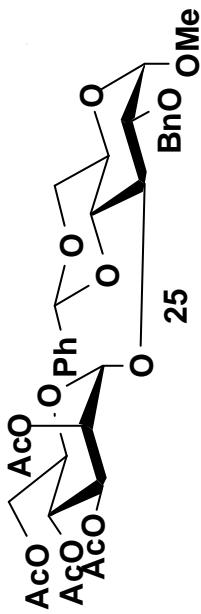




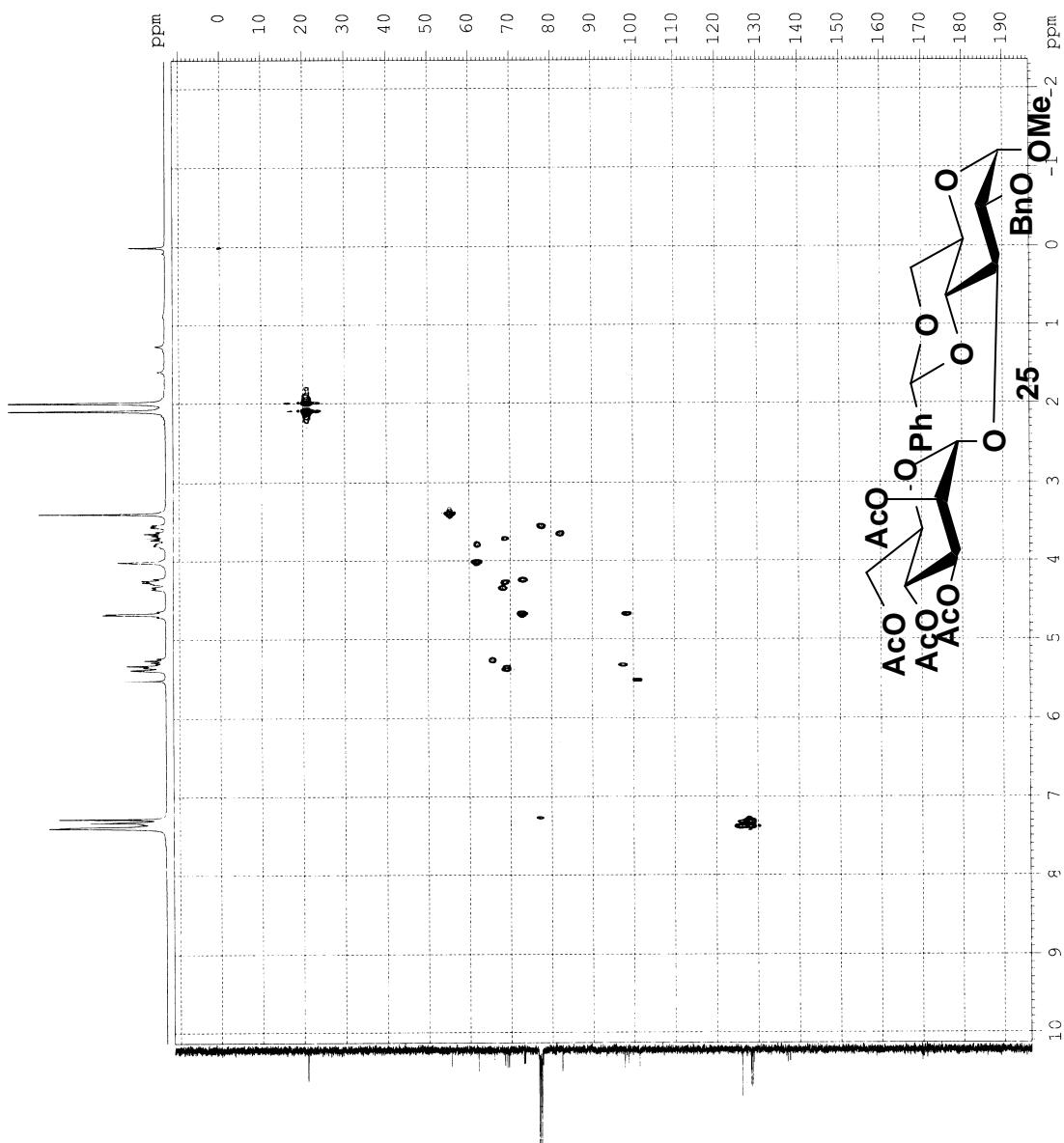


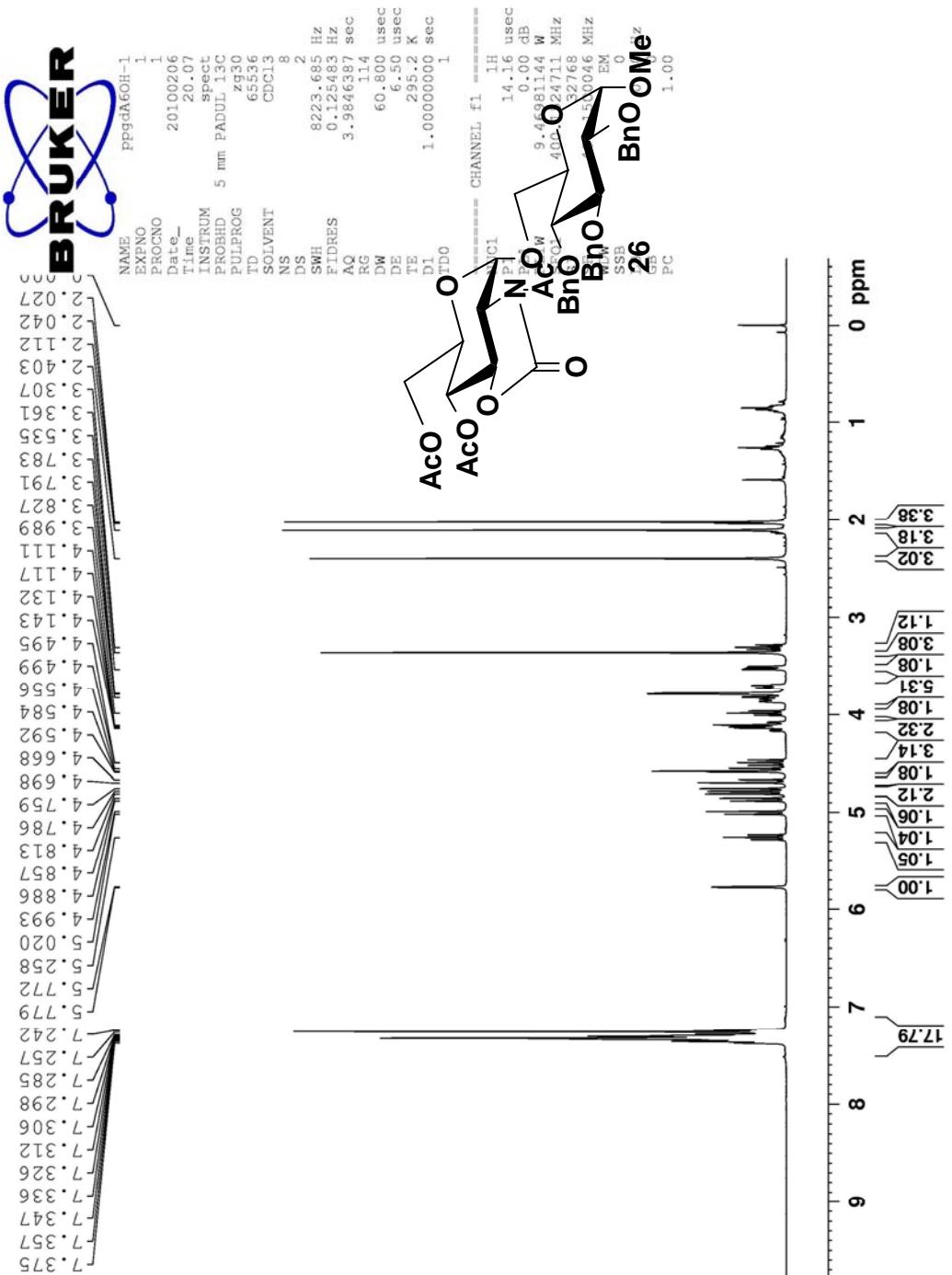


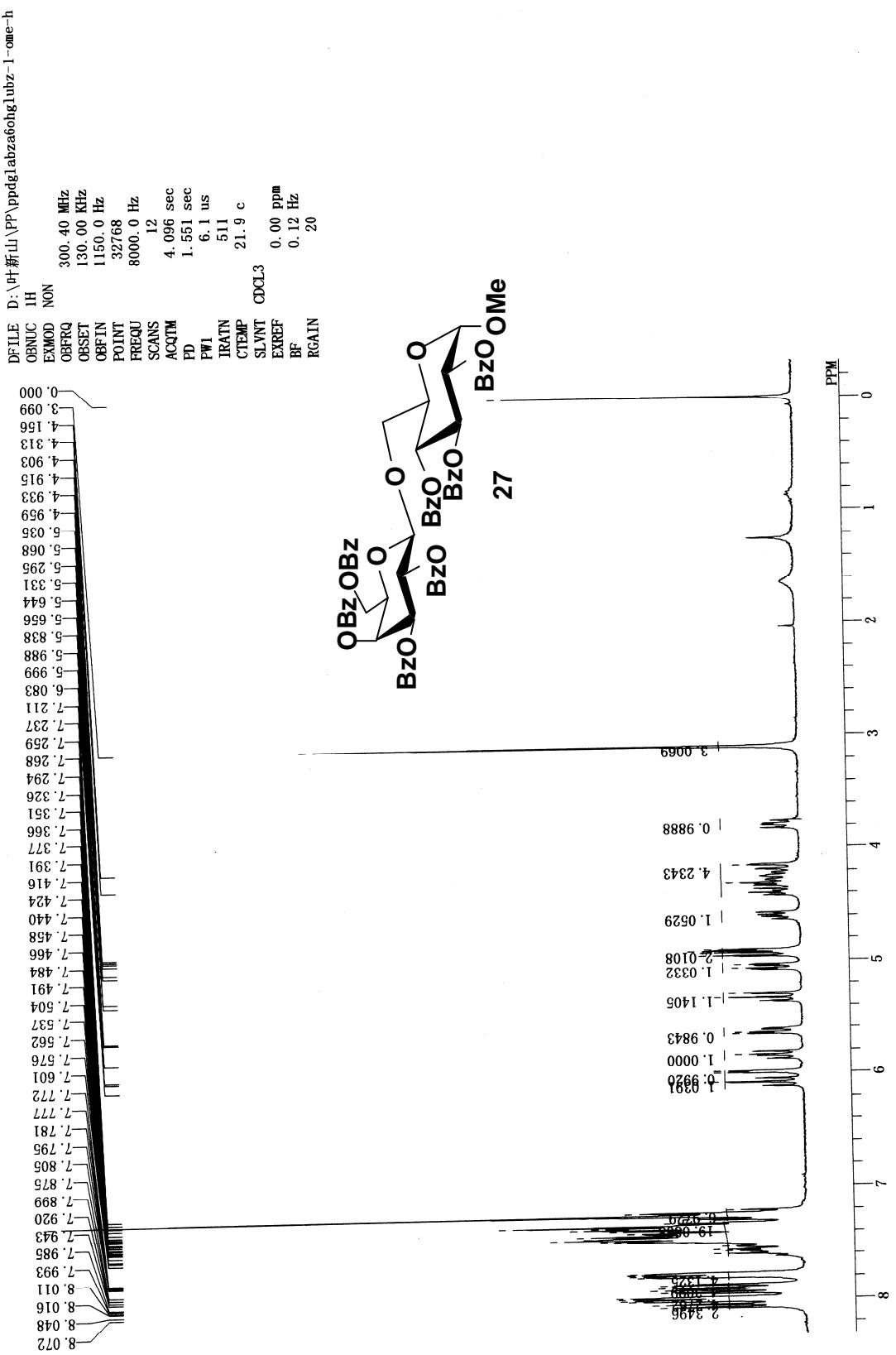
D:\1\H\新山\PP\PM2\PM11-C.als

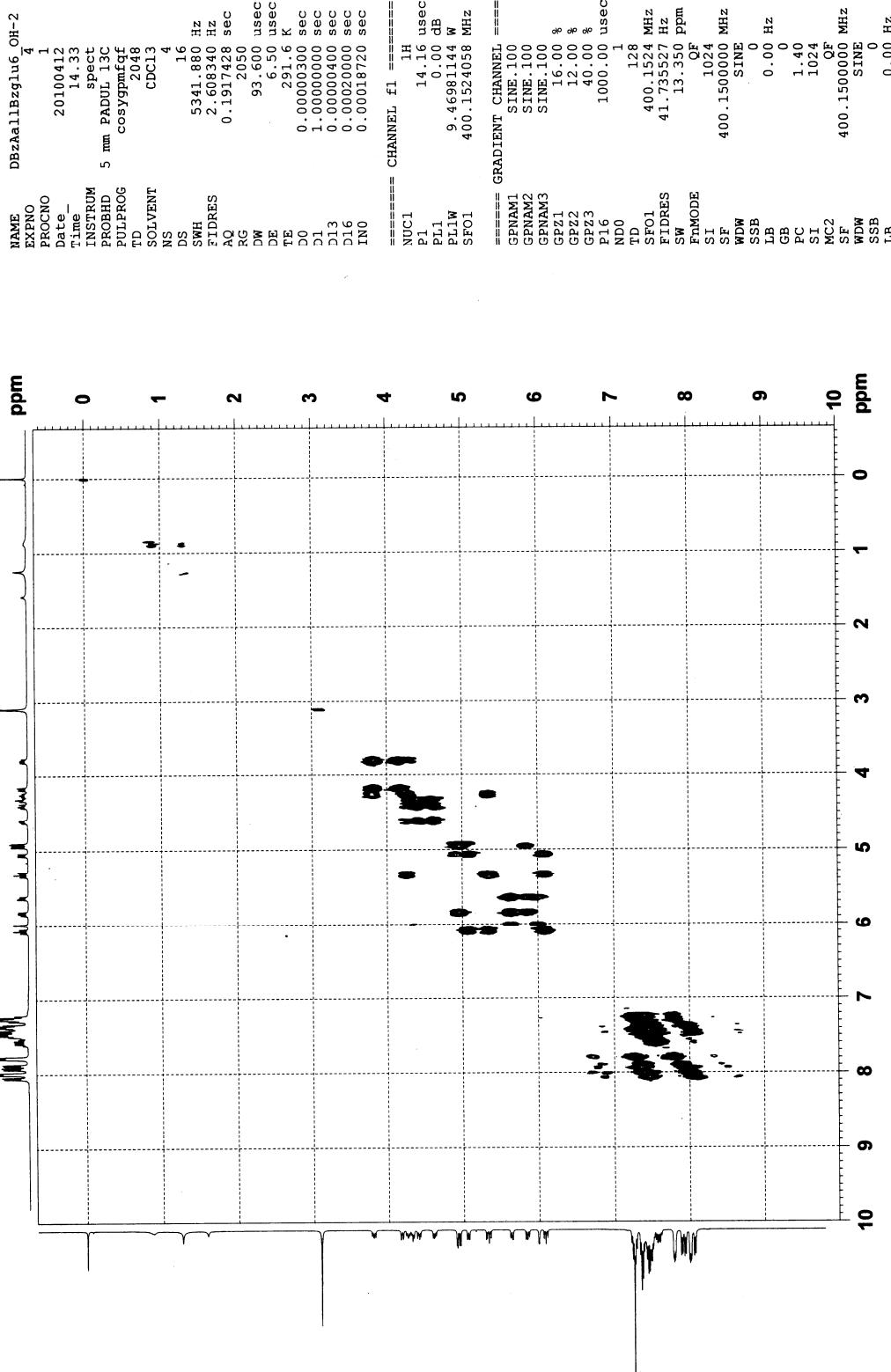
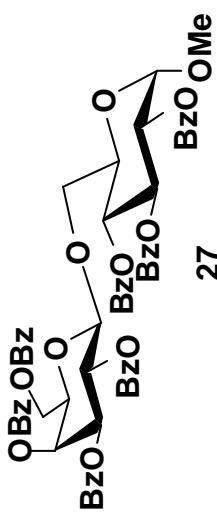


**BRUKER**



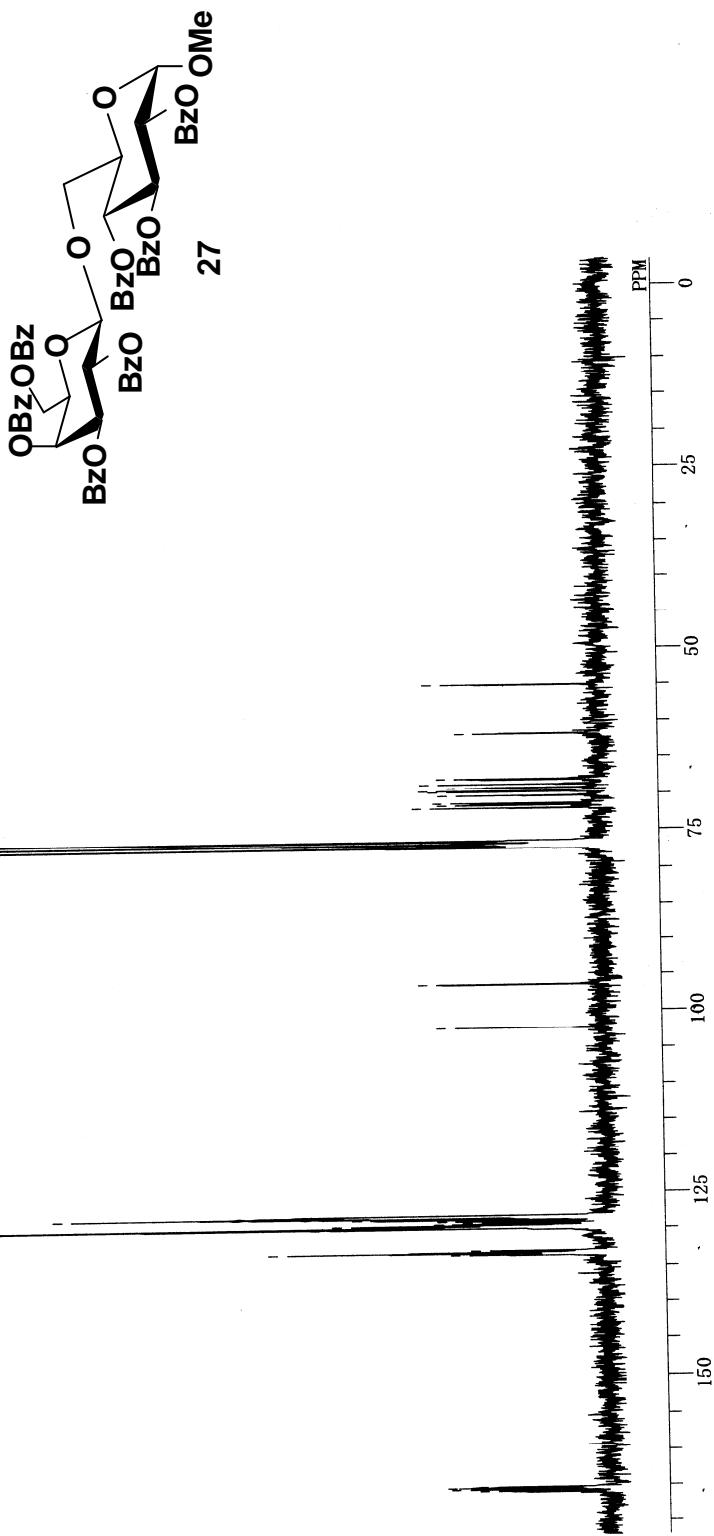


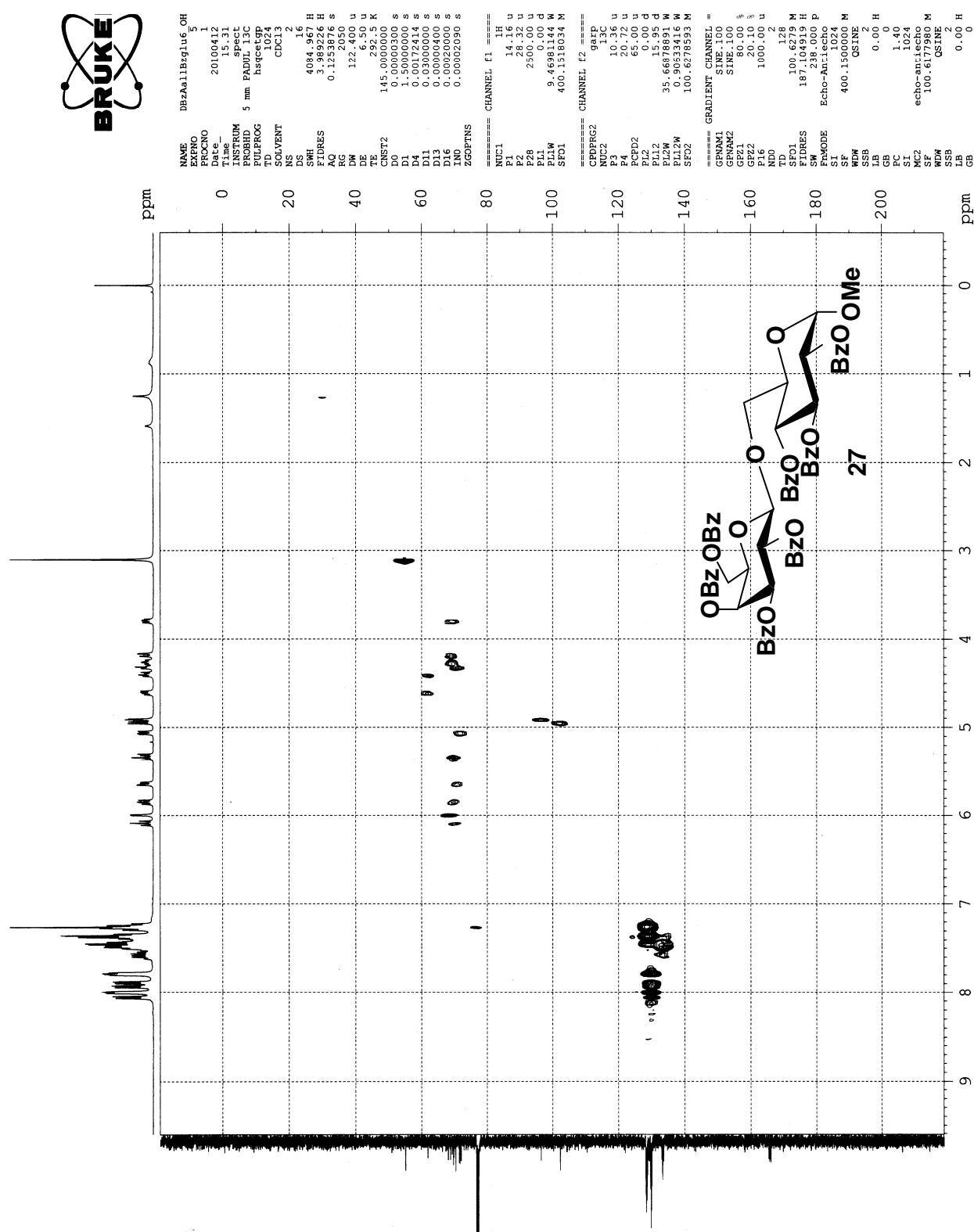




DFILE D:\VH\新山\PP\PPDBEA17-C.als  
 13C  
 EXMOD BGM  
 OBNUC 13C  
 OBFRQ 75.45 MHz  
 OBSET 124.00 kHz  
 OBFIN 1840.0 Hz  
 POINT 32768  
 FREQU 20408.1 Hz  
 SCANS 1577  
 ACQTM 1.606 sec  
 PD 1.394 sec  
 PW1 4.2 us  
 IRATN 511  
 CTEMP 23.2 c  
 SLVNT CDCL<sub>3</sub>  
 EXREF 77.00 ppm  
 BF 2.00 Hz  
 RGAIN 25

27

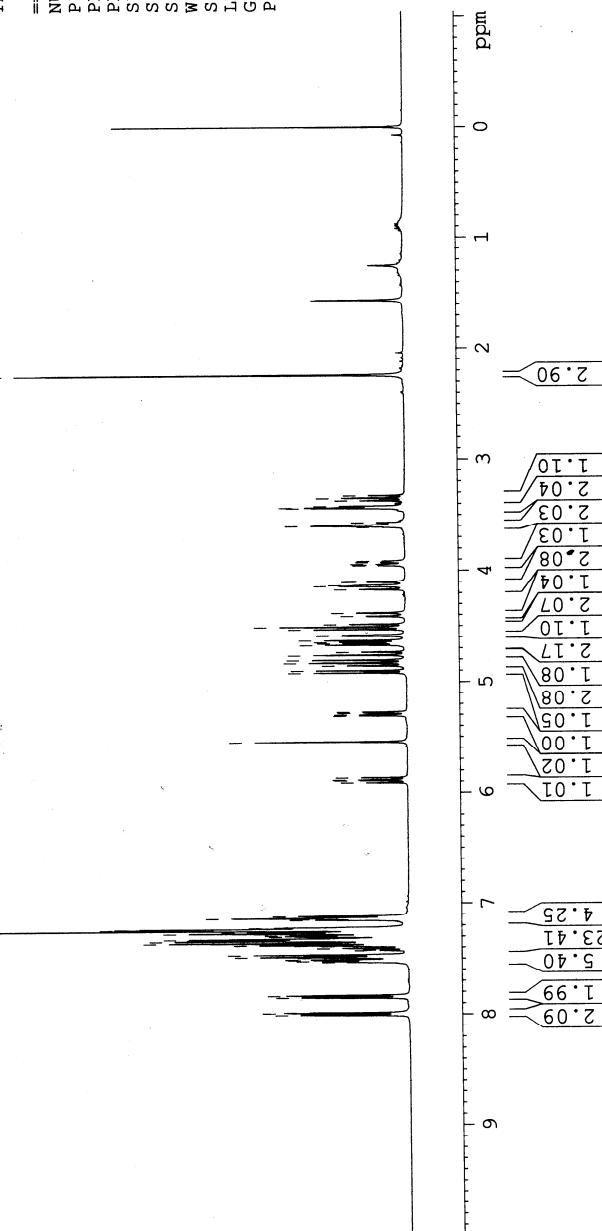
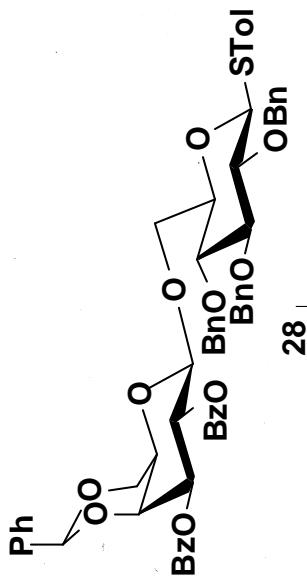


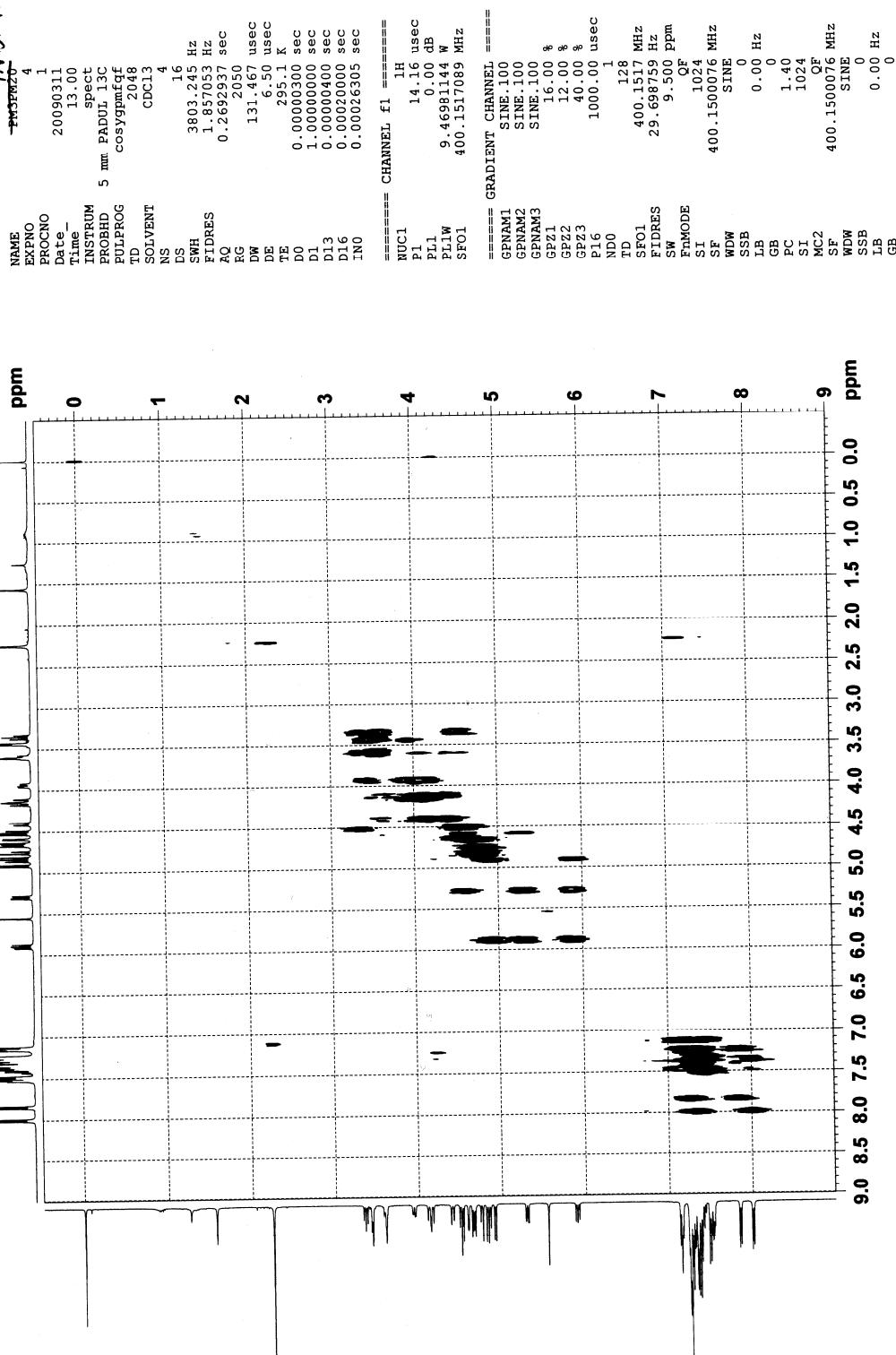
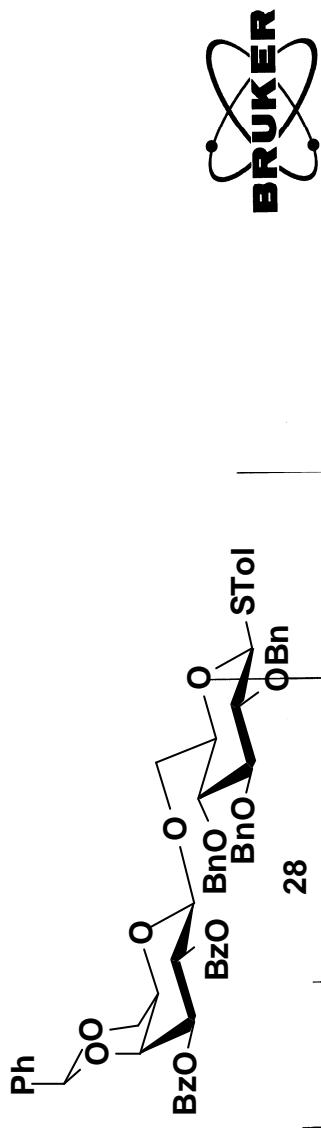


**BRUKER**

PM3A001  
PM3A002  
2  
1  
20090311  
11.59  
spect  
5 mm PADUL 13C  
2930  
65536  
CDCl<sub>3</sub>  
8  
0  
8223.685 Hz  
0.125453 Hz  
3.9846387 sec  
161  
60.800 usec  
6.50 usec  
295.5 K  
1.0000000 sec  
TDO

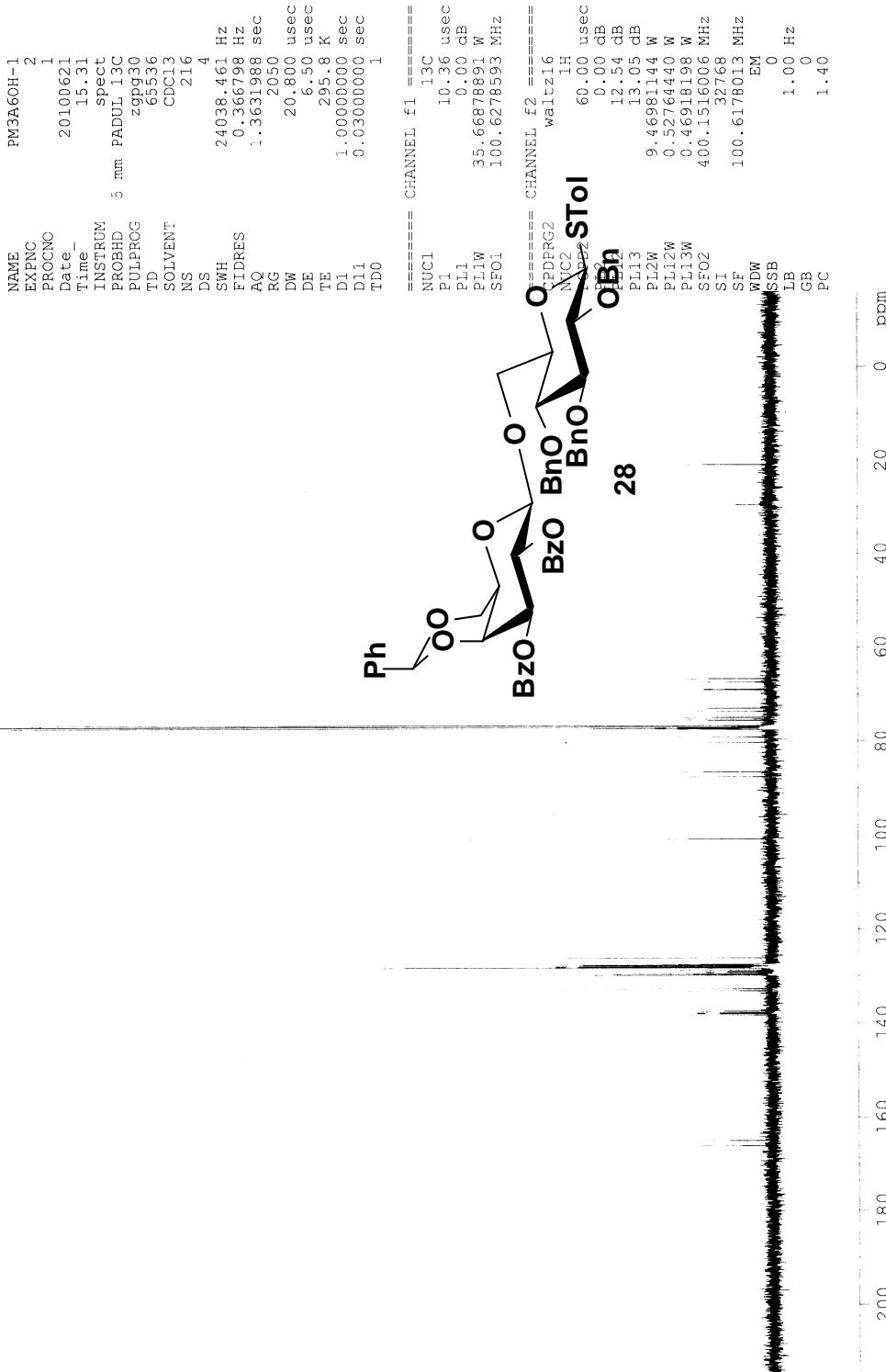
===== CHANNEL f1 =====  
NUC1 1H  
P1 14.16 usec  
PL1 0.00 dB  
PL1W 9.46981144 W  
SFO1 400.1524711 MHz  
SI 32768  
SF 400.1500076 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

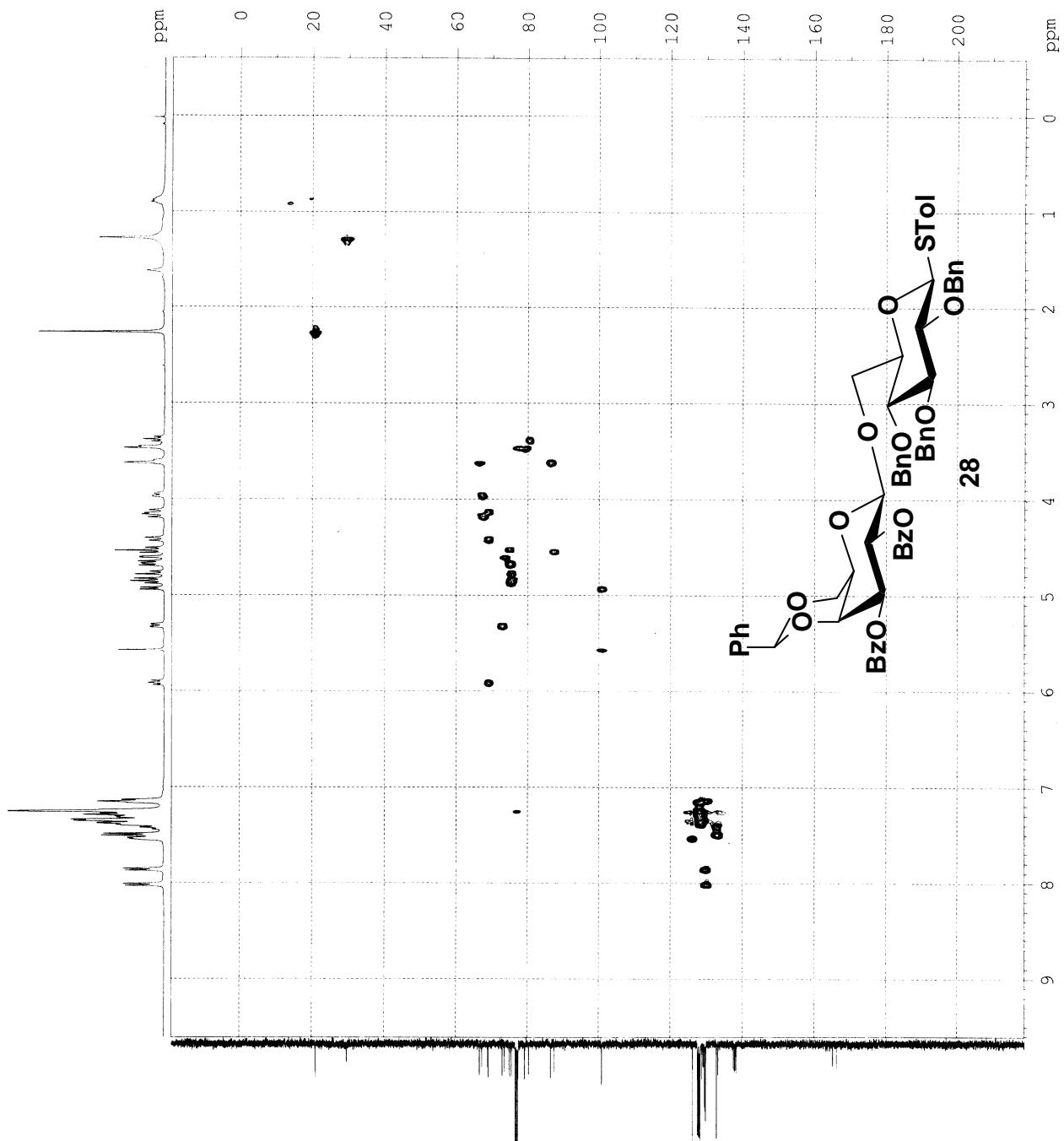




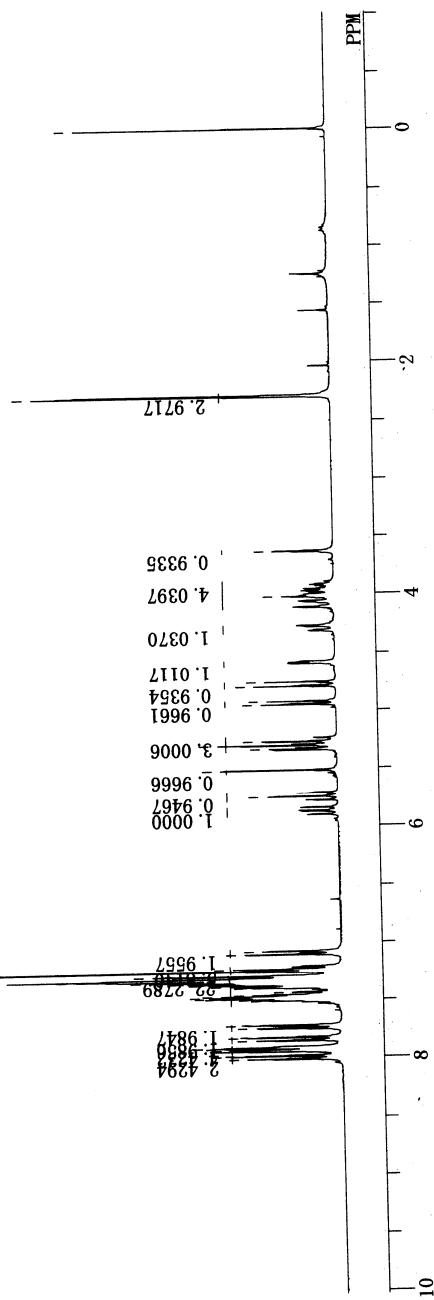
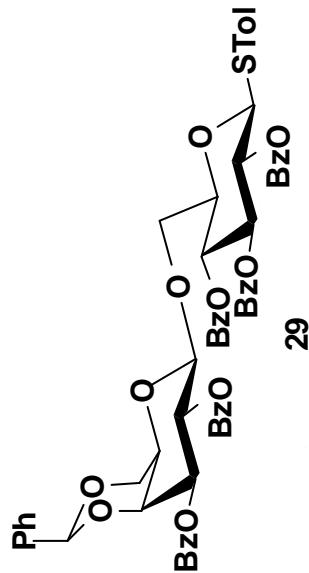
**BRUKER**

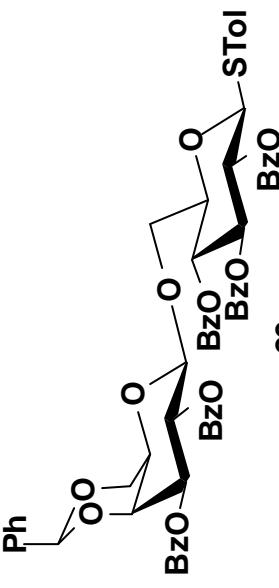
21.02  
66.58  
67.28  
68.92  
69.05  
72.98  
73.60  
74.90  
75.28  
75.61  
76.68  
77.00  
77.20  
77.32  
77.48  
79.26  
80.40  
86.55  
87.55  
100.88  
126.29  
127.60  
127.68  
127.73  
127.79  
127.86  
128.08  
128.18  
128.23  
128.37  
128.87  
129.16  
129.33  
129.64  
129.87  
129.93  
132.90  
133.31  
137.55  
137.84  
138.09  
138.37  
165.14  
166.20



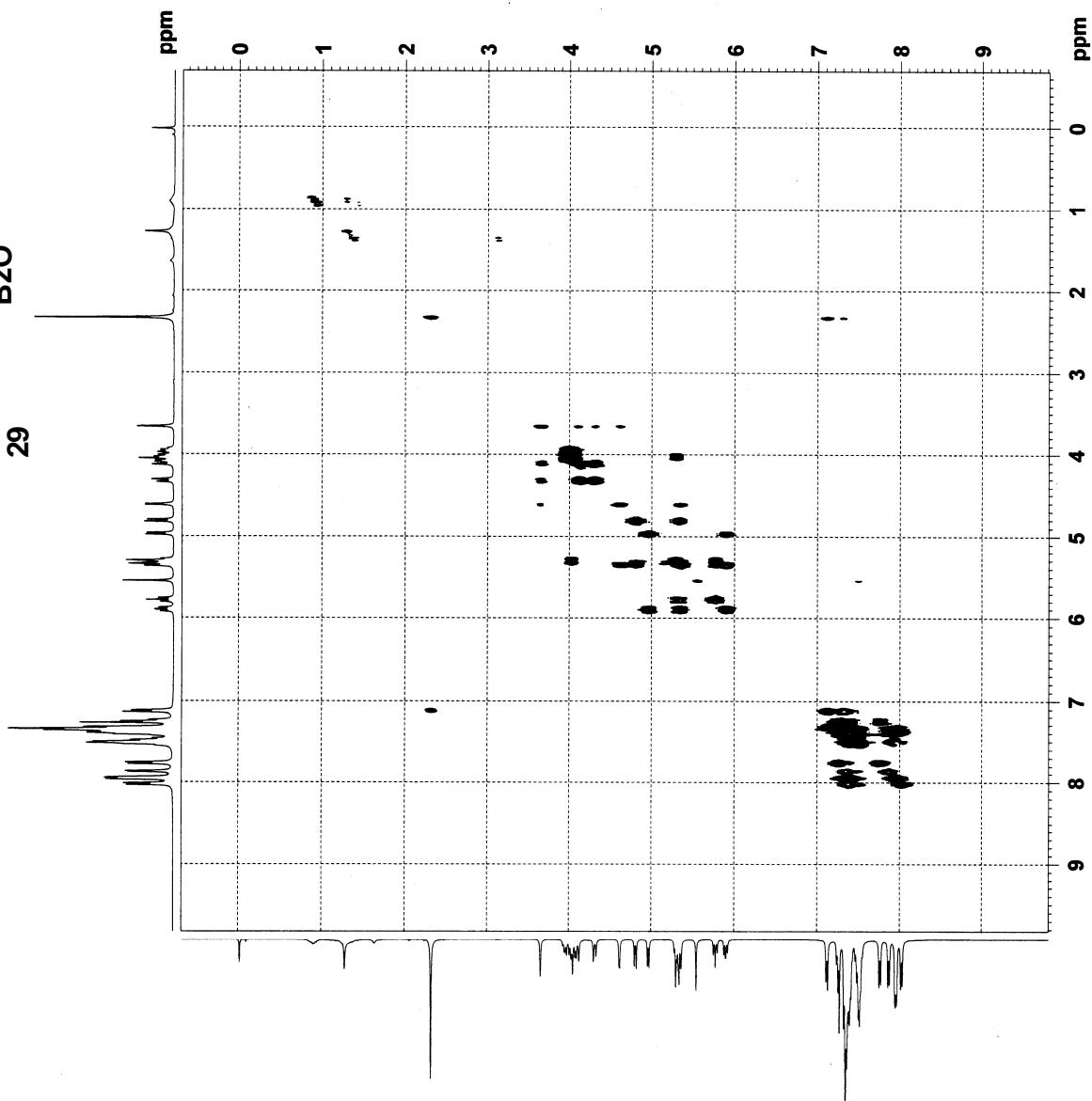


FILE D:\H\新山\PPM3.21-H.als





**BRUKER**

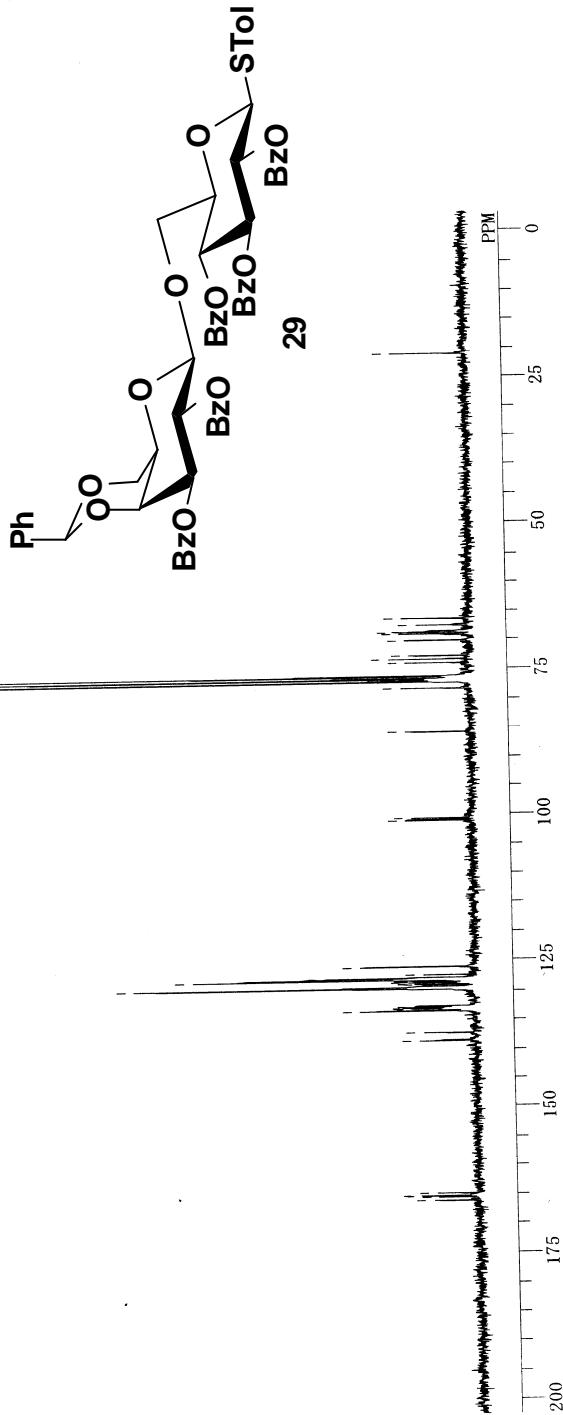


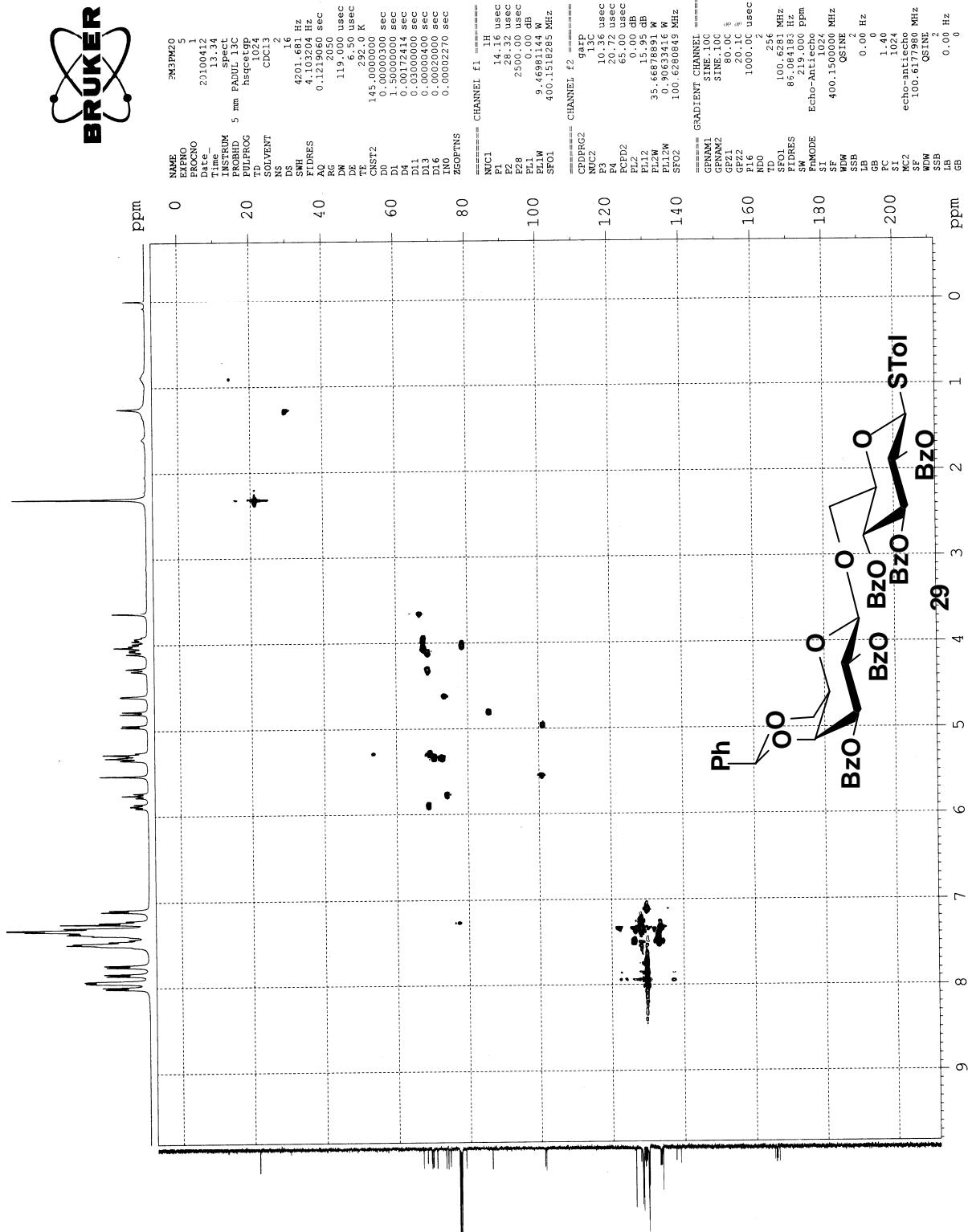
```

NAME          PM3DM20
EXPNO        4
PROCNO       1
Date         20100412
Time         12.44
INSTRUM     spect
PROBHD      5 mm PADUL 13C
PULPROG     cosyppmfgf
TD          2048
SOLVENT      CDCl3
NS           4
DS           16
SWH          4201.681 Hz
FIDRES      2.051602 Hz
AQ           0.2437620 sec
RG           64
DW           119.000 usec
DE           6.500 usec
TE           291.8 K
DO           0.00000300 sec
D1           2.0000000 sec
D13          0.0000400 sec
D16          0.0002000 sec
T1           0.00023800 sec
                ===== CHANNEL f1 =====
NUC1         1H
P1           14.16 usec
PL1          0.00 dB
PL1W         9.46991144 W
SF01         400.1518285 MHz
                ===== GRADIENT CHANNEL =====
GPNAME1    SINE.100
GPNAME2    SINE.100
GPNAME3    SINE.100
GPZ1        16.00 %
GPZ2        12.00 %
GPZ3        40.00 %
P16          1000.00 usec
ND0          1
TD           128
SF01         400.1518 MHz
FDRES       32.824955 Hz
SW           10.500 ppm
FMODE        QF
SI           1024
SF           400.1500000 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0
PC           1.40
SI           1024
MC2          QF
SF           400.1500000 MHz
WDW          SINE
SSB          0

```

D:\VH\新山\PP\ppm3pm20-2-C.als  
 QBNUC 13C  
 EXMOD BOM 75.45 MHz  
 OBRQ 124.00 kHz  
 OFFSET 1840.0 Hz  
 POINT 32768  
 FREQU 20408.1 Hz  
 SCANS 1408  
 ACQTM 1.606 sec  
 PD 1.394 sec  
 PWI 4.2 us  
 TRATN 511  
 CTMP 19.2 c  
 SLINT 0DCL3  
 EXPRF 77.00 ppm  
 BF 2.00 Hz  
 RGAIN 25



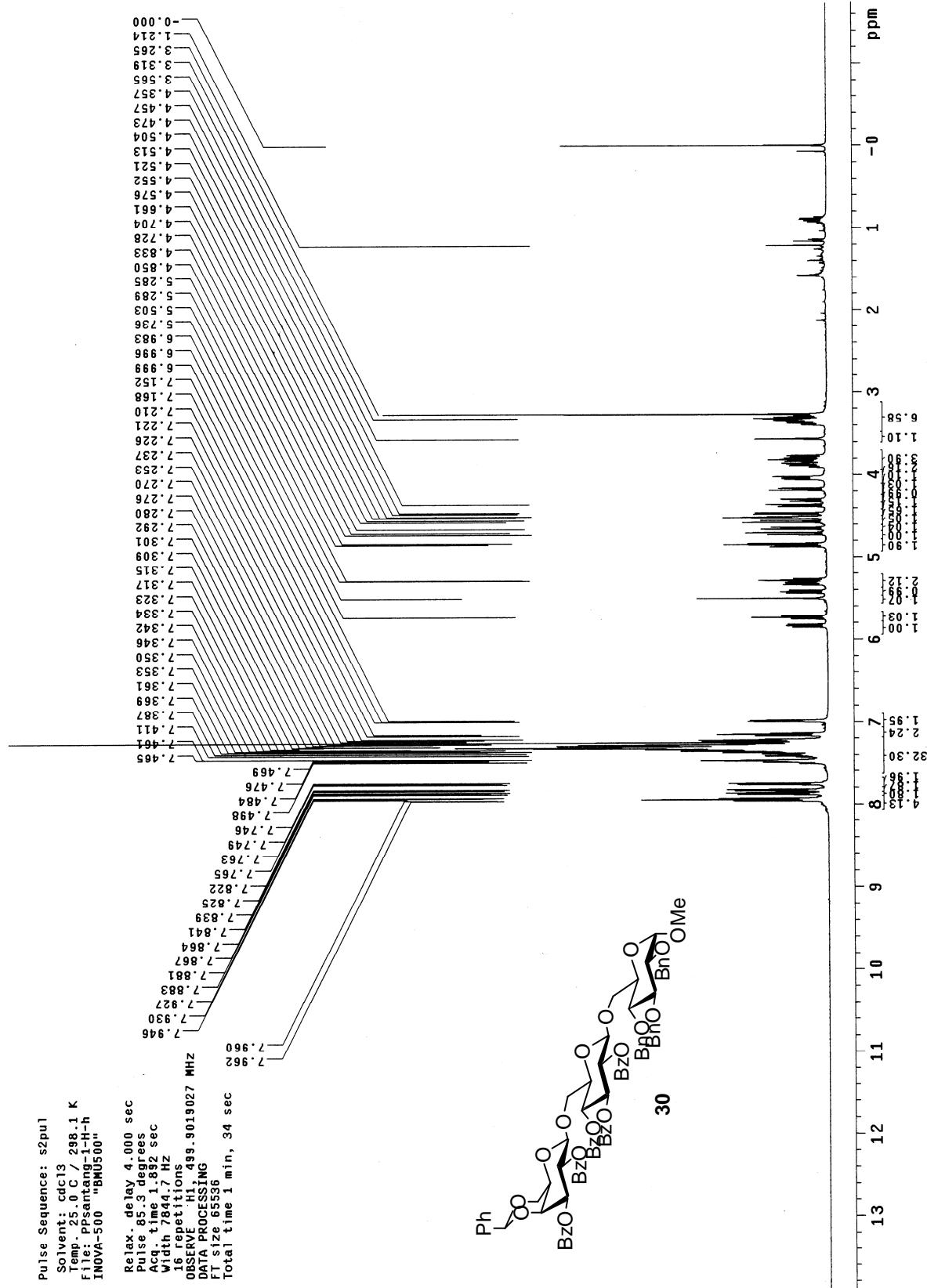


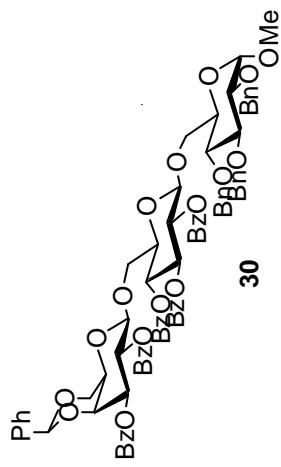
PPsantang-1-H

Pulse Sequence: s2pu1

Solvent: cdc13  
Temp: 25 °C / 298.1 K  
File: PPsantang-1-H-h  
INOVA-500 "BH500"

Relax, delay 4.000 sec  
Pulse 85.3 degrees  
Acq. time 1.812 sec  
Width 7844.7 Hz  
16 repetitions  
OBSERVE H1, 499.9019027 MHz  
DATA PROCESSING  
FT size 65536  
Total time 1 min, 34 sec





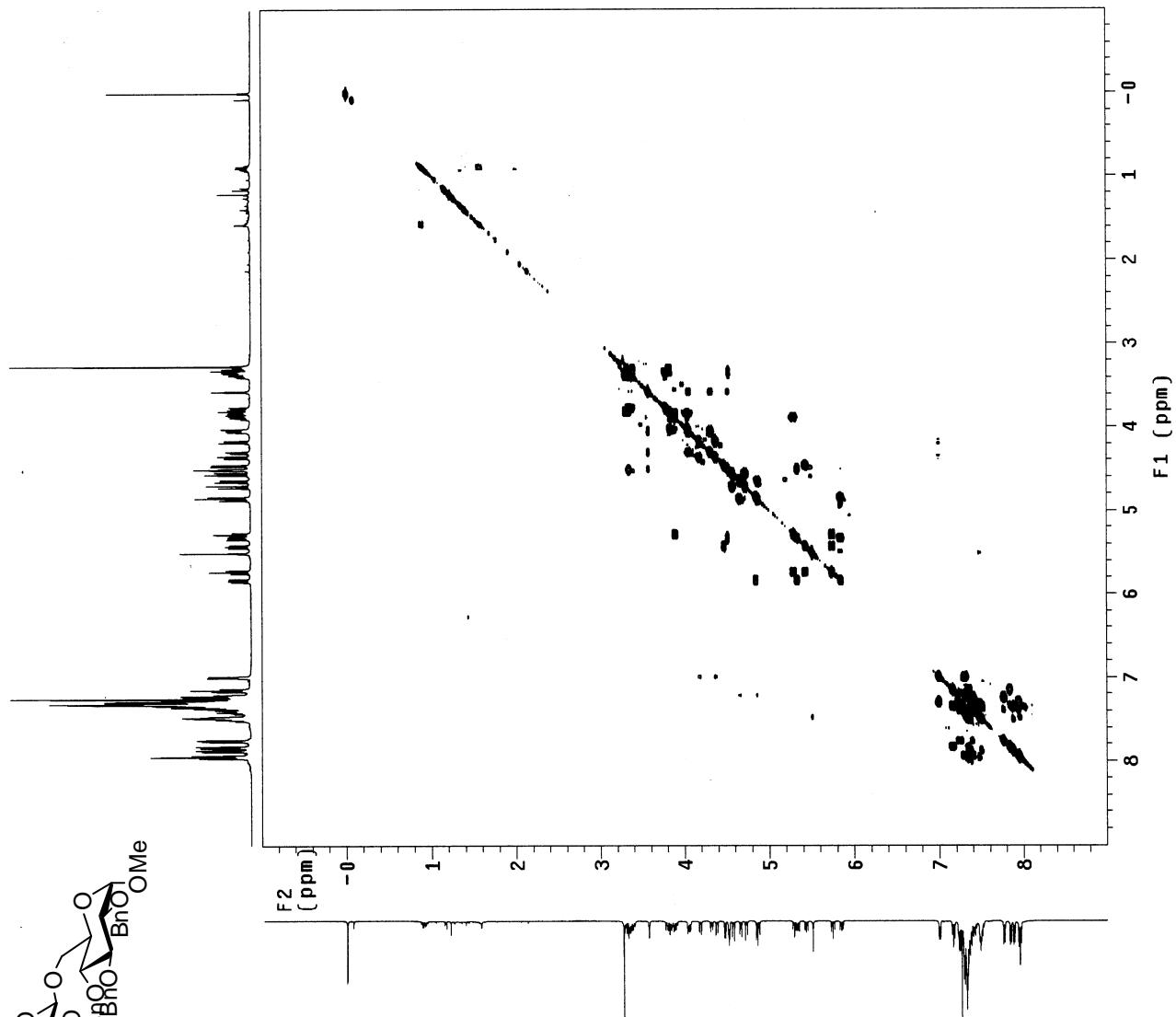
File: PROTON

Pulse Sequence: gCOSY  
 Solvent: cdc13  
 Temp. 25.0 C / 298.1 K  
 NOVA-500 "RM1500"

```

Relax. delay 1.000 sec
Aq. time 0.25 sec
Width 4996.6 Hz
120 Width 4996.6 Hz
120 repetitions
400 increments
OBSERVE H1, 499.01035 MHz
DATA PROCESSING
Sr. sine be1 0.102 sec
1.0 DATA PROCESSING
Sq. sine be1 0.004 sec
T size 4096 x 4096
T 2 hr, 48 min, 1 sec

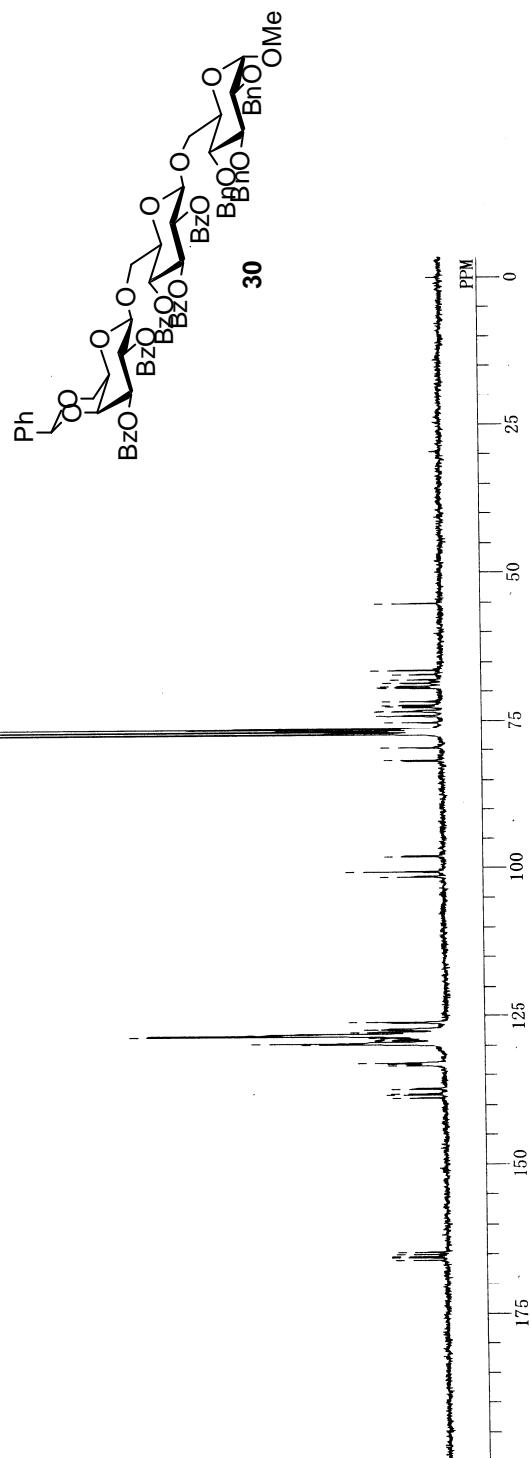
```



DFILE C:\WINNMR95\COMMON\\_DEFAULT.ALS

13C

OBNUC BCM  
EXMOD 75.45 MHz  
OBFRQ 124.00 kHz  
OBFIT 1840.0 Hz  
POINT 32768  
FREQU 20408.1 Hz  
SCANS 16576  
ACQTM 1.606 sec  
PD 1.394 sec  
PWL 4.2 us  
TRATN 511  
CTEMP 22.5 c  
SLVNT CDCL<sub>3</sub>  
EXREF 77.00 ppm  
BF 2.00 Hz  
RGAIN 25



**BRÜKER**

