Investigation of the Regioselectivity for Staudinger Reaction and Its Application for the Synthesis of Aminoglycosides with N-1 Modification

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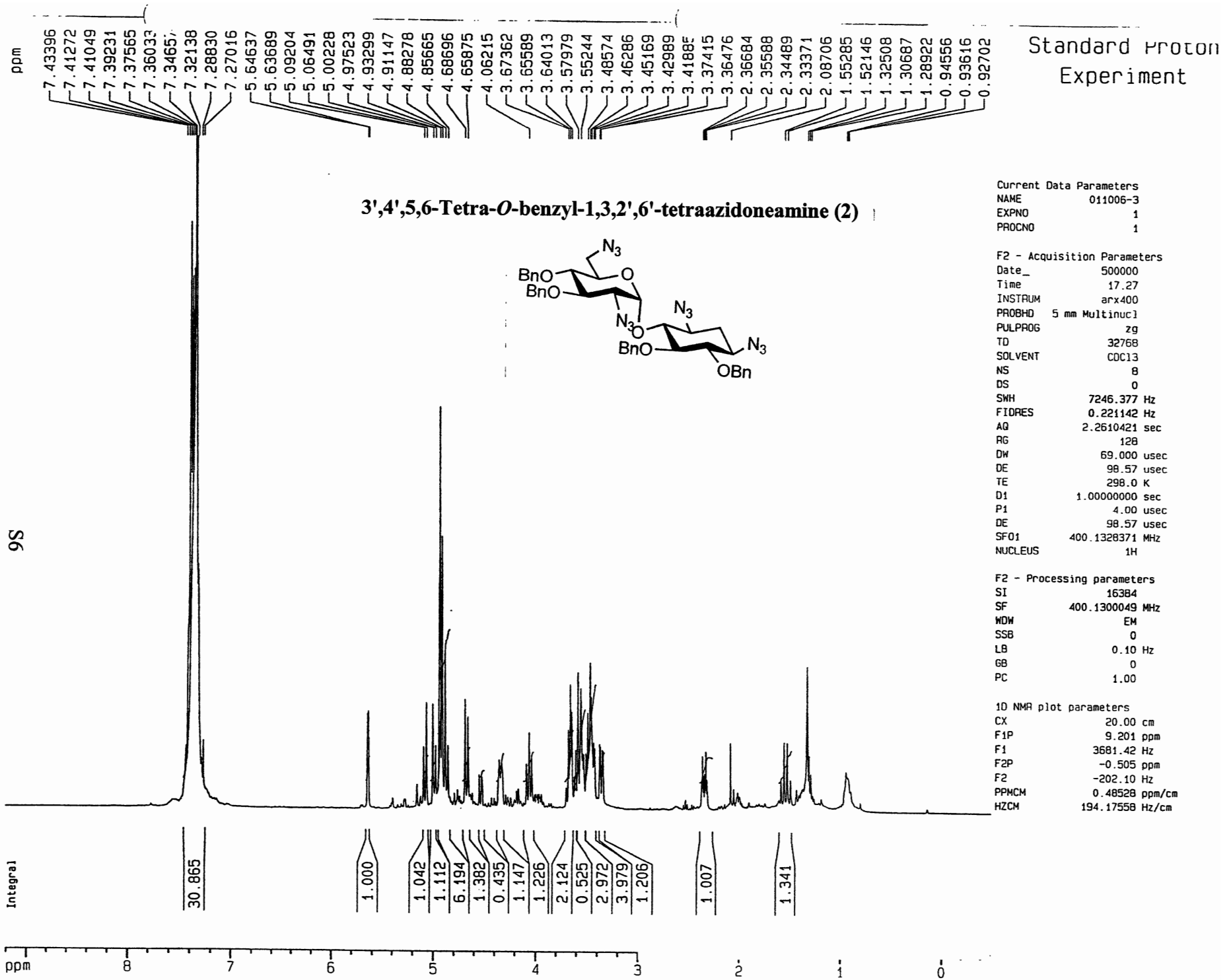
Supporting Information

I. General Experimental Procedures

General Procedure for Selective Staudinger Reaction. To a solution of polyazido starting material and BOC-ON ([2-(tert-butoxycarbonyloxyimino)-2-phenylacetonitrile]) (2.4 equiv.) in anhydrous toluene at -78^{o}C under atmospheric nitrogen, trimethylphosphine (1.0 M solution in toluene, 1.1 equiv.) was added. The reaction was kept at -78^{o}C for 30 minutes, then was stirred overnight allowing the temperature to warm up to room temperature. The reaction was quenched with phosphate buffer (*p*H = 7) and then was concentrated. The residue was re-dissolved with EtOAc. The organic solution was washed with saturated $NaHCO_{3(aq)}$, water and brine, then dried over anhydrous Na_2SO_4. Removal of the solvent followed by purification with a gradient column chromatography (Hexanes:EtOAc = 100:0 to 40:60) usually afforded multiple components, which were characterized by ^{1}H, ^{13}C and ^{1}H-^{1}H COSY NMR.

General Procedure for Glycosylation and Hydrolysis: A solution of phenylthioglycosyl donor (1 equiv.), neamine derivative (1.2 equiv.), and activated powder 4A^{o} molecular sieve was mixed together in anhydrous CH_2Cl_2 at room temperature first. N-iodosuccinimide (1.2 equiv.) was quickly added into above solution and the reaction mixture was stirred for 3 hrs at room temperature. Trimethylsilyl triflate (0.15 equiv.) was added. The reaction solution was stirred till the consumption of the glycosyl donor (ca. 48 hrs, monitored by TLC, Hexane: EtOAc = 65: 35). The reaction mixture was quenched by the addition of Na_2SO_4-$10H_2O_{(s)}$, $NaHCO_{3(s)}$, $Na_2S_2O_{3(s)}$. After being stirred for 5 mins, the reaction mixture changed color from dark brown to colorless. Then the reaction mixture was filtered through Celite and the solvent was removed. The crude product was extracted with EtOAc, washed with H_2O, brine and dried over anhydrous Na_2SO_4. After removal of the solvents, the crude product was purified with column chromatography. The glycosylated compounds were often mixed with inseparable impurities, and were fully characterized after hydrolysis. The glycosylated product was dissolved in tetrahydrofuran (3 mL) and H_2O (1 mL) and LiOH-H_2O was added. The reaction mixture was stirred at room temperature till the completion of the reaction (ca. 24 hrs, monitored by TLC, EtOAc: Hexane = 65: 35). The solvent was removed and the residue was purified via column chromatography to provide the product as colorless oil.

II. ^{1}H NMR and ^{13}C NMR Spectra for the Synthesized Compounds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3',4',5,6-Tetra-O-benzyl-1,3,2',6'-tetraazidoneamine (2)
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PROCNO 1
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Date_ 500000
Time 17.27
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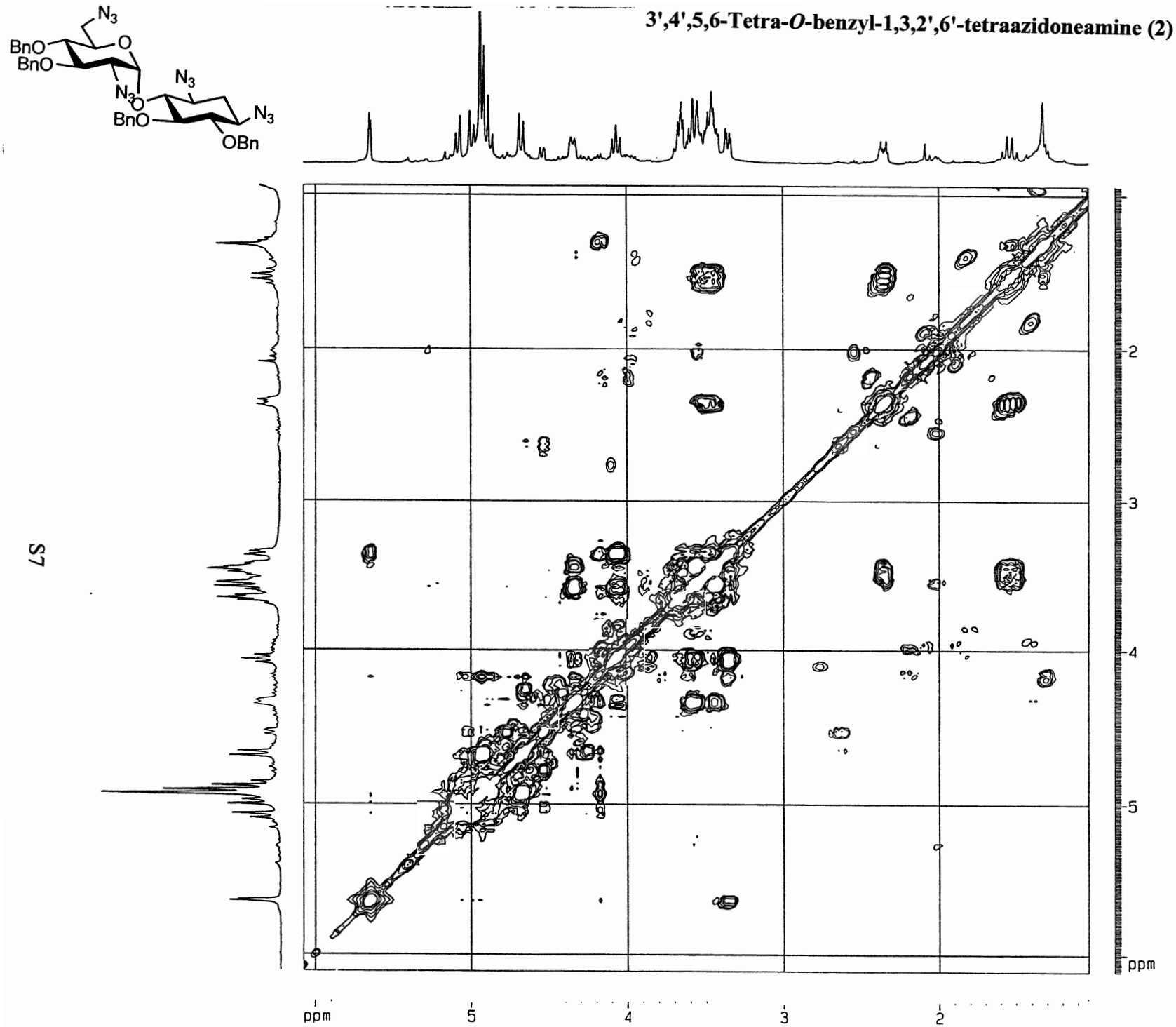
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 128
DW 69.000 usec
DE 98.57 usec
TE 298.0 K
D1 1.00000000 sec
P1 4.00 usec
DE 98.57 usec
SFO1 400.1328371 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 16384
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 9.201 ppm
F1 3681.42 Hz
F2P -0.505 ppm
F2 -202.10 Hz
PPMCM 0.48528 ppm/cm
HZCM 194.17558 Hz/cm
ppm
7.43396
7.41272
7.41049
7.39231
7.37565
7.36033
7.34657
7.32138
7.28830
7.27016
5.64637
5.63689
5.09204
5.06491
5.00228
4.97523
4.93299
4.91147
4.88278
4.85665
4.68696
4.65875
4.06215
3.67362
3.65589
3.64013
3.57979
3.55244
3.48574
3.46286
3.45169
3.42989
3.37415
3.36476
2.36684
2.35588
2.34489
2.33371
2.08706
1.55285
1.52146
1.32508
1.30687
1.28922
0.94556
0.93616
0.92702
Integral
30.865
1.000
1.042
1.112
6.194
1.382
0.435
1.147
1.226
2.124
0.525
2.972
3.979
1.206
1.007
1.341
ppm
8
7
6
5
4
3
2
1
0


3',4',5,6-Tetra-O-benzyl-1,3,2',6'-tetraazidoneamine (2)
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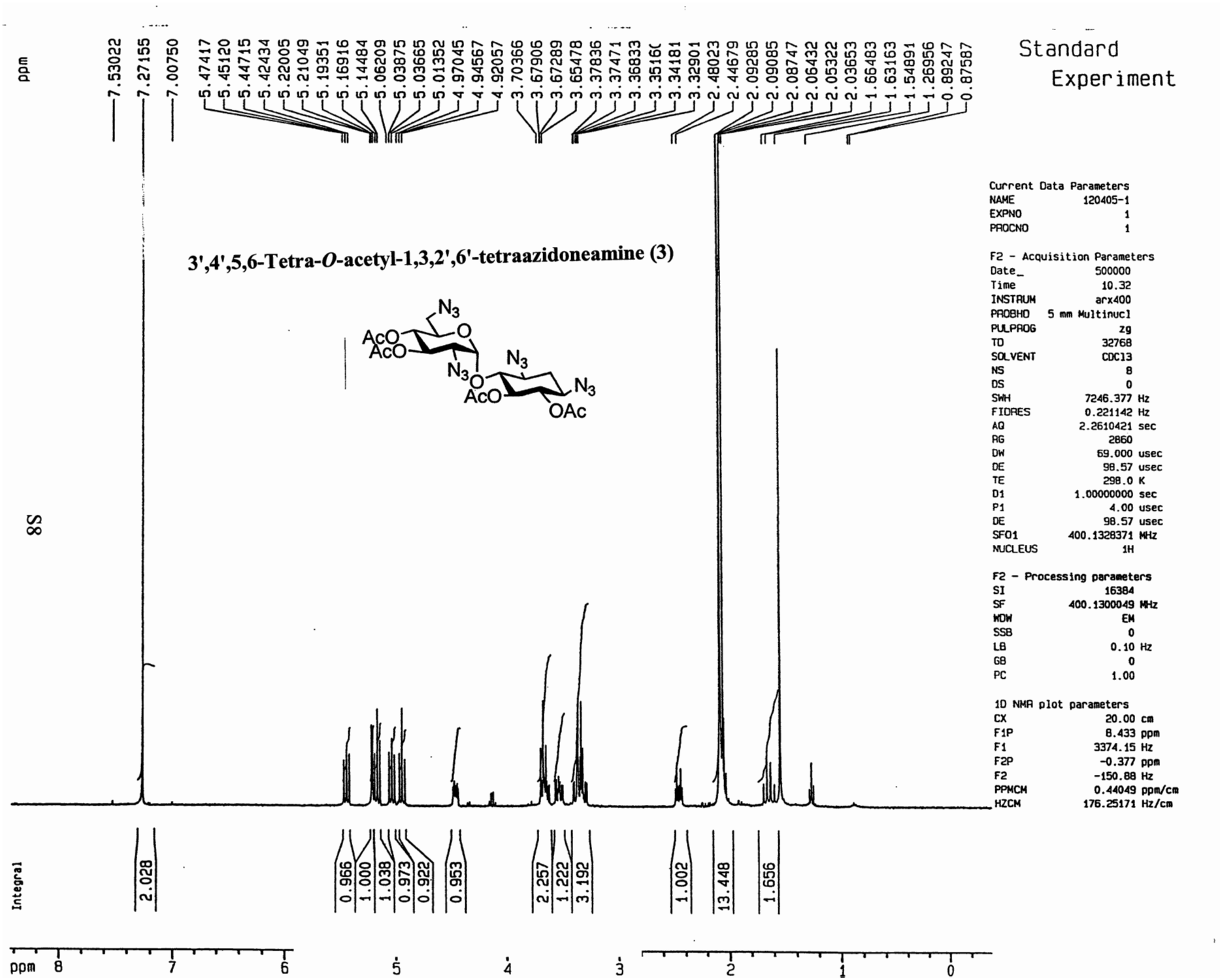
BnO
BnO
O
N3
O
N3
BnO
N3
OBn
ppm
5
4
3
2
ppm


Standard
Experiment
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PROCNO 1
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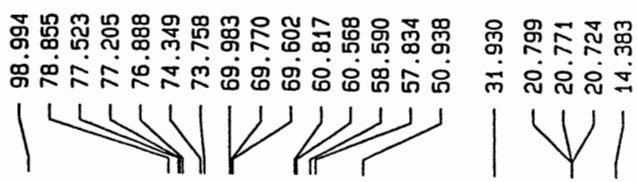
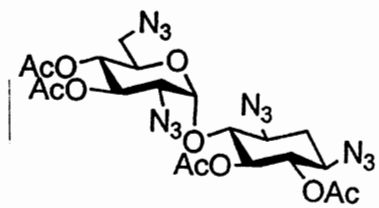
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 2860
DW 69.000 usec
DE 98.57 usec
TE 298.0 K
D1 1.00000000 sec
P1 4.00 usec
DE 98.57 usec
SFO1 400.1328371 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 16384
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 8.433 ppm
F1 3374.15 Hz
F2P -0.377 ppm
F2 -150.88 Hz
PPMCM 0.44049 ppm/cm
HZCM 176.25171 Hz/cm
ppm
Integral
2.028
0.966
1.000
1.038
0.973
0.922
0.953
2.257
1.222
3.192
1.002
13.448
1.656
ppm

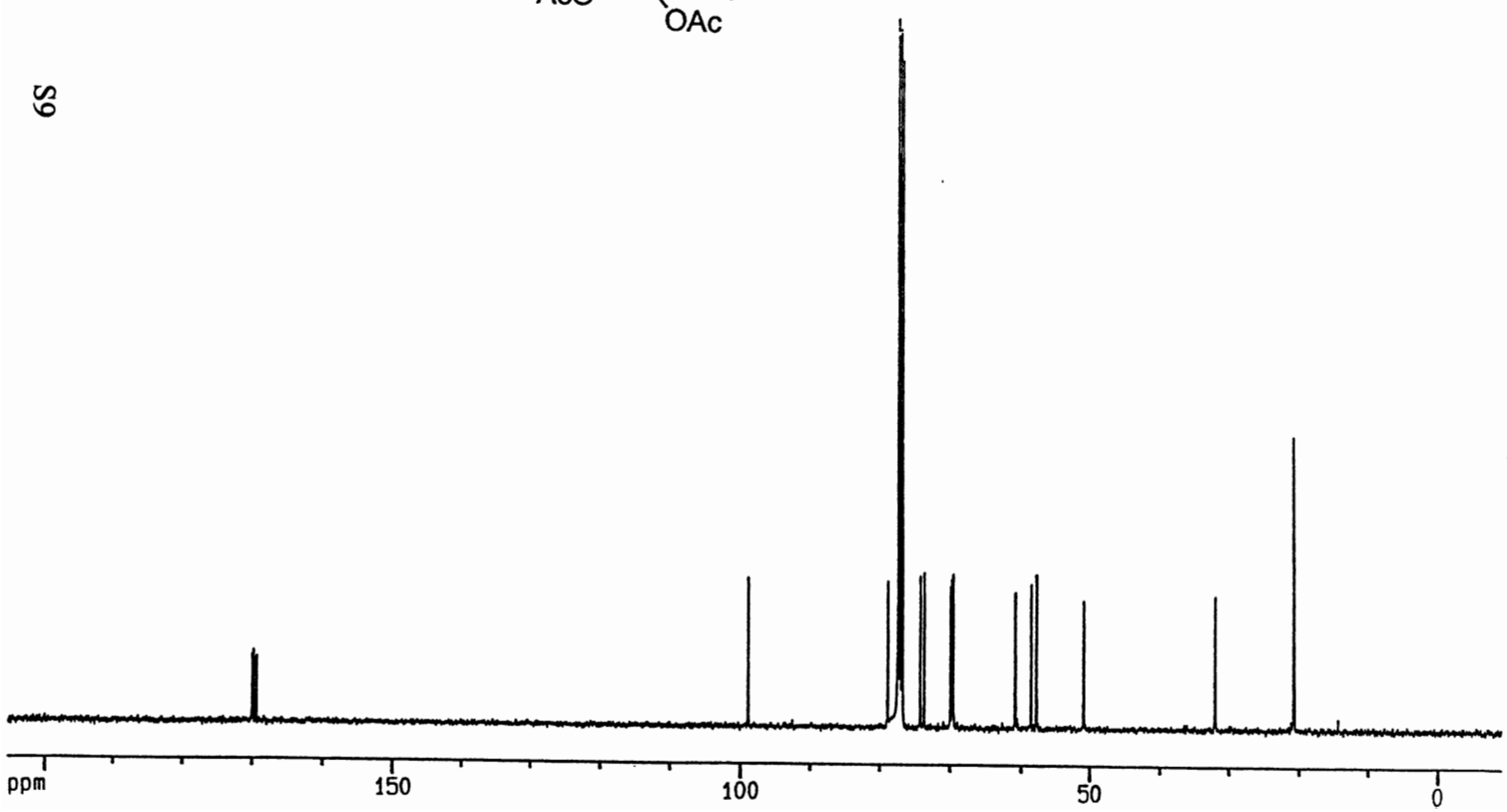
3',4',5,6-Tetra-*O*-acetyl-1,3,2',6'-tetraazidoneamine (3)

Standard 13C
Experiment





S9

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PROCNO                       1

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PULPROG                 zgdc30
TD                       32768
SOLVENT                  CDCl3
NS                       20000
DS                           2
SWH                25000.000 Hz
FIDRES             0.762939 Hz
AQ                 0.6554100 sec
RG                       45500
DW                    20.000 usec
DE                    27.14 usec
TE                    300.0 K
D12                0.00002000 sec
DL5                   20.00 dB
CPDPRG                waltz16
P31                   100.00 usec
D1                 0.40000001 sec
P1                     6.75 usec
DE                    27.14 usec
SFO1              100.6231179 MHz
NUCLEUS                  13C
D11                0.03000000 sec

F2 - Processing parameters
SI                       16384
SF                100.6127490 MHz
WDW                        EM
SSB                         0
LB                     2.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                  205.346 ppm
F1                 20660.42 Hz
F2P                   -9.474 ppm
F2                  -953.25 Hz
PPMCM               10.74102 ppm/cm
HZCM              1080.68311 Hz/cm
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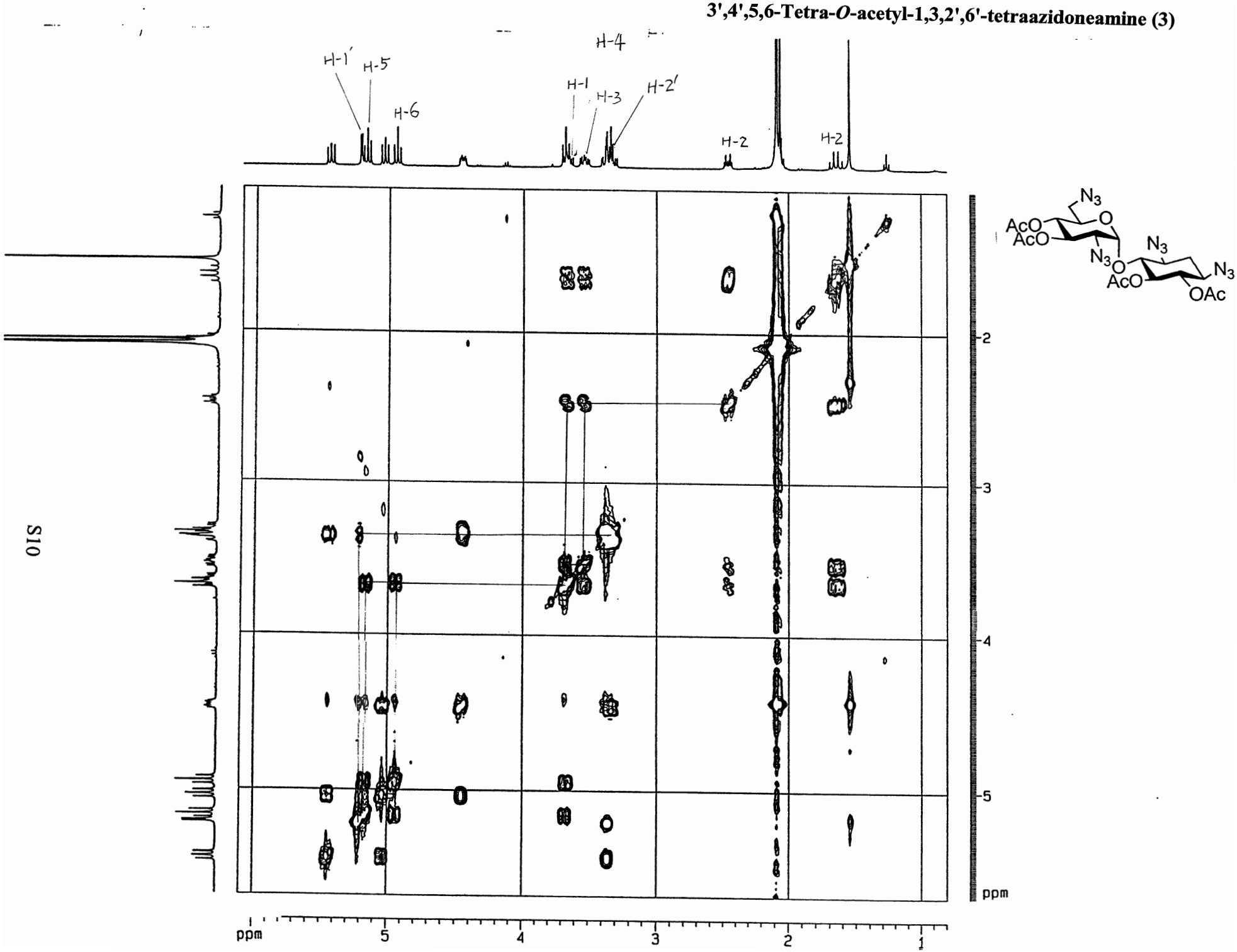
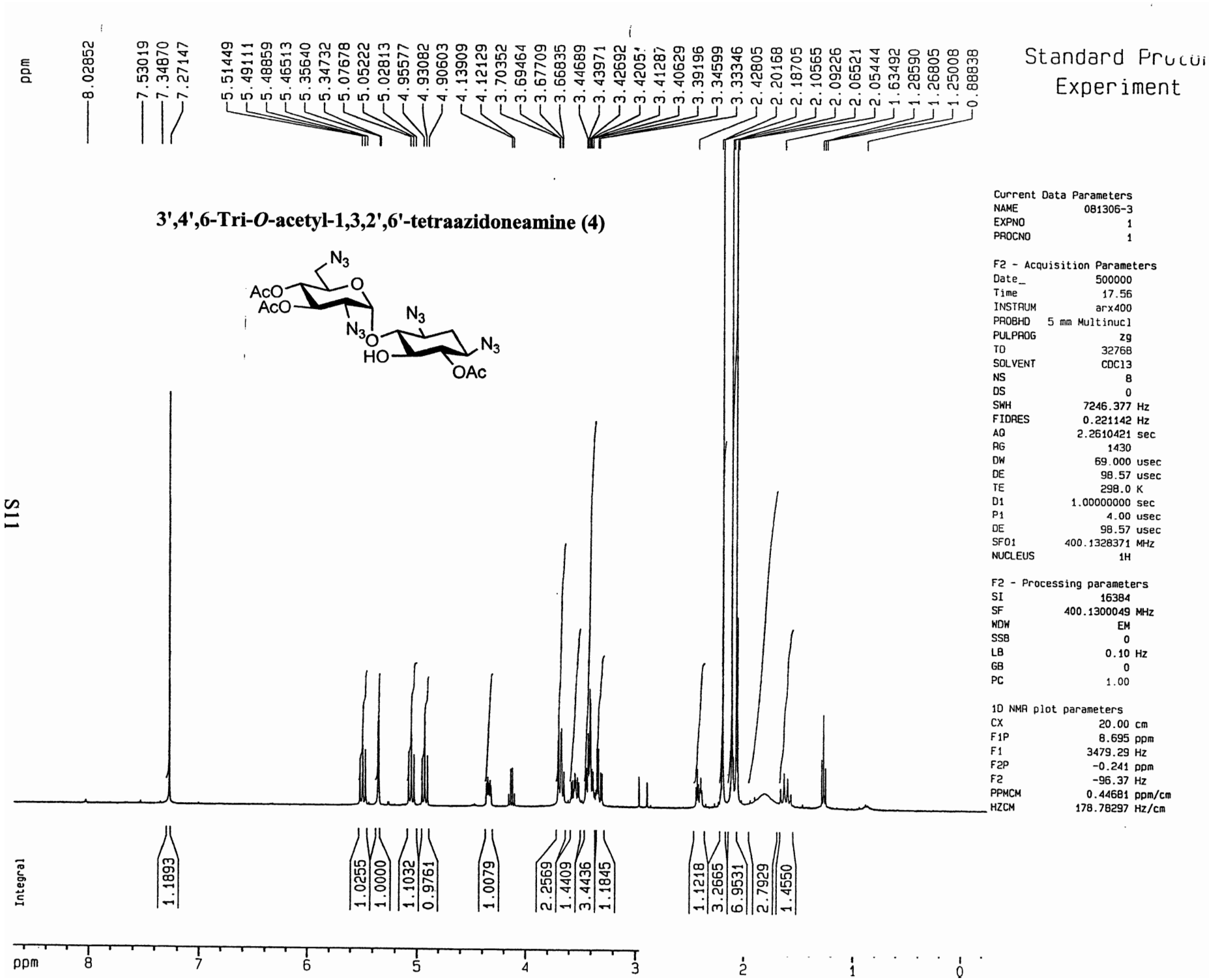
3',4',5,6-Tetra-*O*-acetyl-1,3,2',6'-tetraazidoneamine (3)

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7.27147
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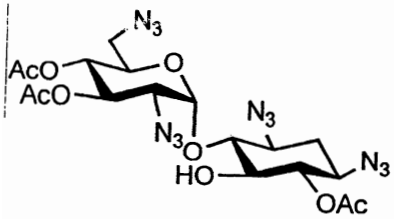
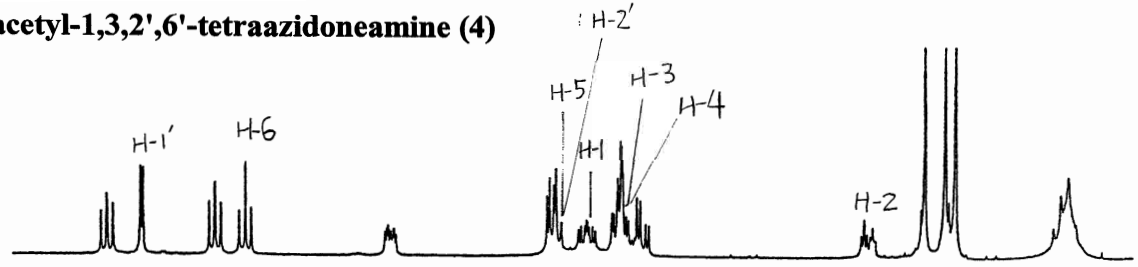
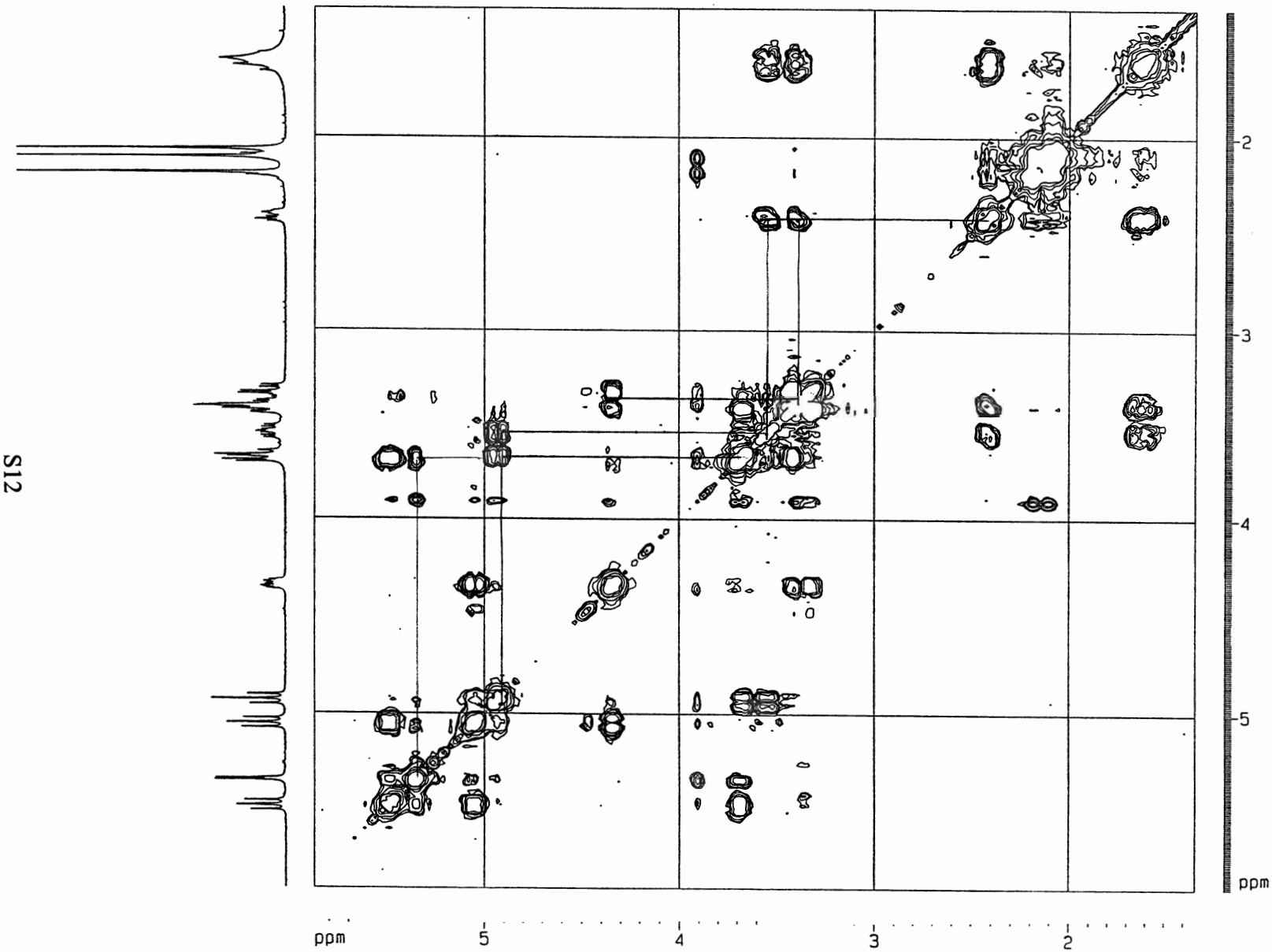
5.07678
5.05222
5.02813
4.95577
4.93082
4.90603
4.13909
4.12129
3.70352
3.69464
3.67709
3.66835
3.44689
3.43971
3.42692
3.42051
3.41287
3.40629
3.39196
3.34599
3.33346
2.42805
2.20168
2.18705
2.10565
2.09226
2.06521
2.05444
1.63492
1.28590
1.26805
1.25008
0.88838
Standard Proton Experiment
3',4',6-Tri-O-acetyl-1,3,2',6'-tetraazidoneamine (4)
N3
AcO
AcO
O
N3
N3
O
HO
N3
OAc
Current Data Parameters
NAME 081306-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 17.56
INSTRUM arx400
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 1430
DW 69.000 usec
DE 98.57 usec
TE 298.0 K
D1 1.00000000 sec
P1 4.00 usec
DE 98.57 usec
SFO1 400.1328371 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 16384
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 8.695 ppm
F1 3479.29 Hz
F2P -0.241 ppm
F2 -96.37 Hz
PPMCM 0.44681 ppm/cm
HZCM 178.78297 Hz/cm
Integral
1.1893
1.0255
1.0000
1.1032
0.9761
1.0079
2.2569
1.4409
3.4436
1.1845
1.1218
3.2665
6.9531
2.7929
1.4550
ppm
8
7
6
5
4
3
2
1
0

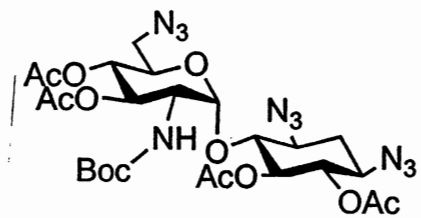
3',4',6-Tri-*O*-acetyl-1,3,2',6'-tetraazidoneamine (4)







3',4',5,6-Tetra-*O*-acetyl-2'-*N*-*tert*-butoxycarbonyl-1,3,6'-triazidoneamine (8)



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PROCNO	1
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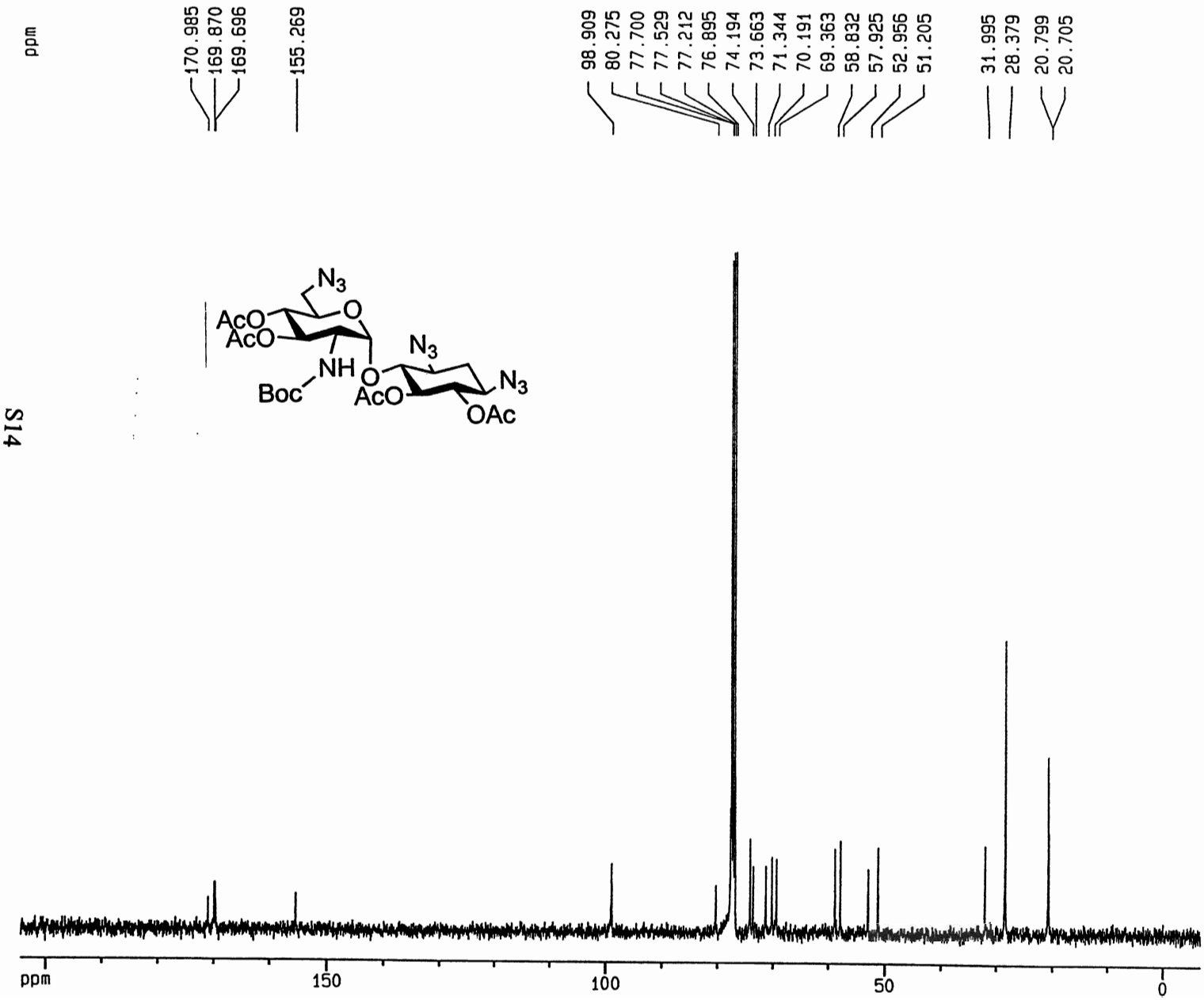
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	512
DW	69.000 usec
DE	98.57 usec
TE	298.0 K
D1	1.00000000 sec
P1	4.00 usec
DE	98.57 usec
SFO1	400.1328371 MHz
NUCLEUS	1H
F2 - Processing parameters	
SI	16384
SF	400.1300049 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00
1D NMR plot parameters	
CX	20.00 cm
F1P	8.411 ppm
F1	3365.61 Hz
F2P	-0.441 ppm
F2	-176.49 Hz
PPMCM	0.44262 ppm/cm
HZCM	177.10519 Hz/cm

Standard 13C
Experiment

3',4',5,6-Tetra-*O*-acetyl-2'-*N*-*tert*-butoxycarbonyl-1,3,6'-triazidoneamine (8)



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EXPNO 1
PROCNO 1

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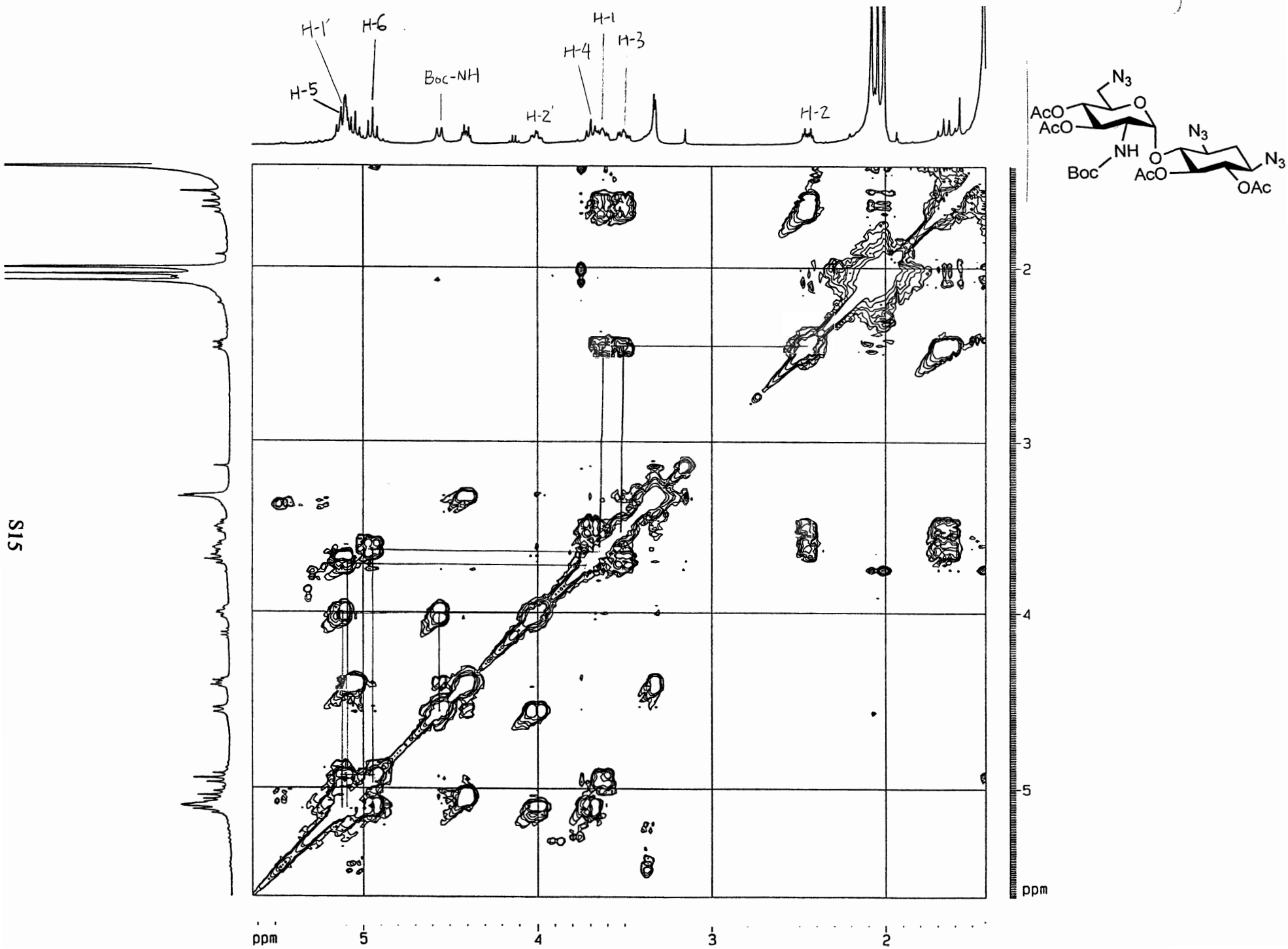
PULPROG zgdc30
TD 32768
SOLVENT CDCl3
NS 3782
DS 2
SWH 25000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554100 sec
RG 45500
DW 20.000 usec
DE 27.14 usec
TE 300.0 K
D12 0.00002000 sec
DL5 20.00 dB
CPDPRG waltz16
P31 100.00 usec
D1 0.40000001 sec
P1 6.75 usec
DE 27.14 usec
SF01 100.6231179 MHz
NUCLEUS 13C
D11 0.03000000 sec

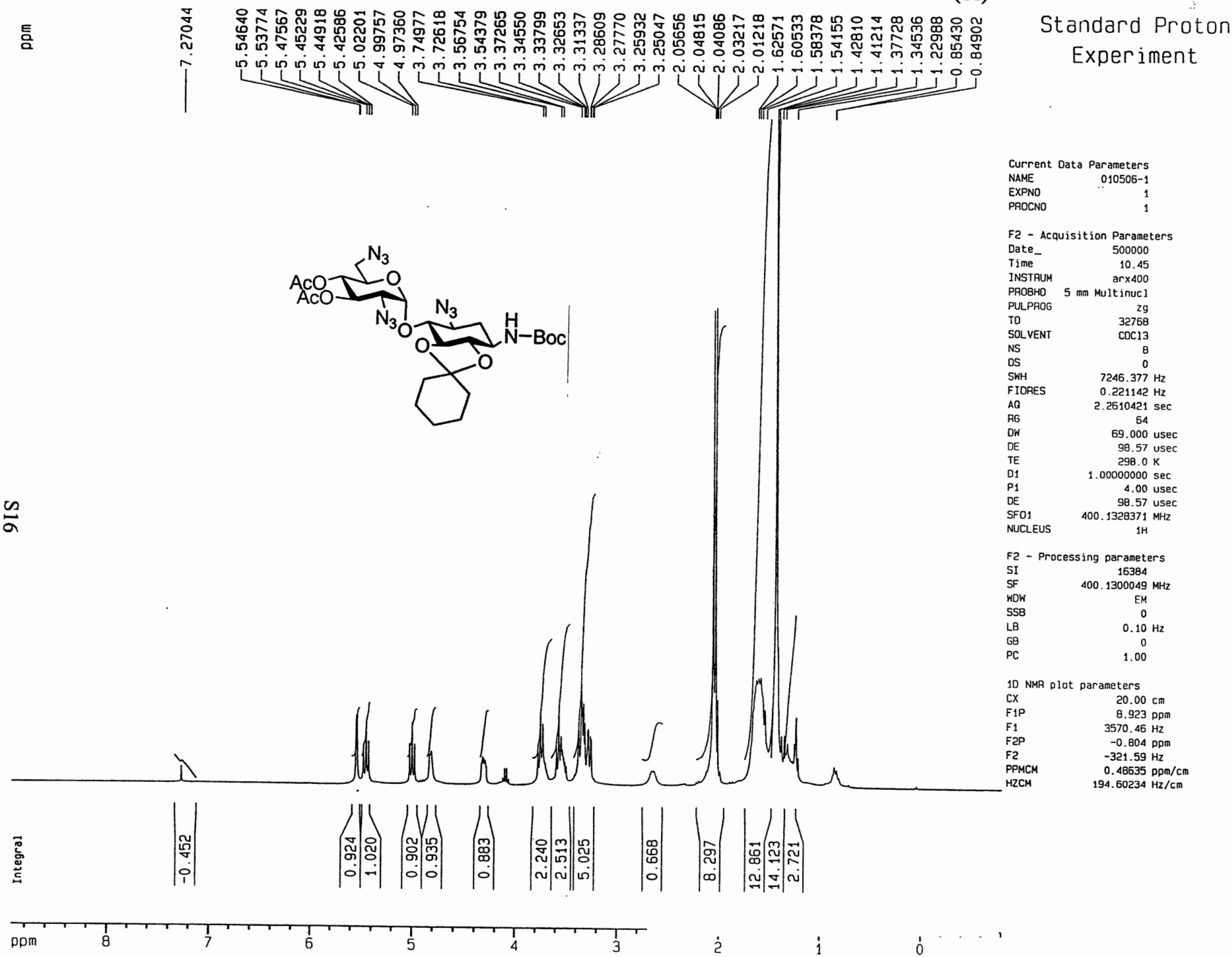
F2 - Processing parameters
SI 16384
SF 100.6127490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 204.468 ppm
F1 20572.08 Hz
F2P -6.840 ppm
F2 -688.23 Hz
PPMCM 10.56541 ppm/cm
HZCM 1063.01526 Hz/cm

3',4',5,6-Tetra-*O*-acetyl-2'-*N*-*tert*-butoxycarbonyl-1,3,6'-triazidoneamine (8)

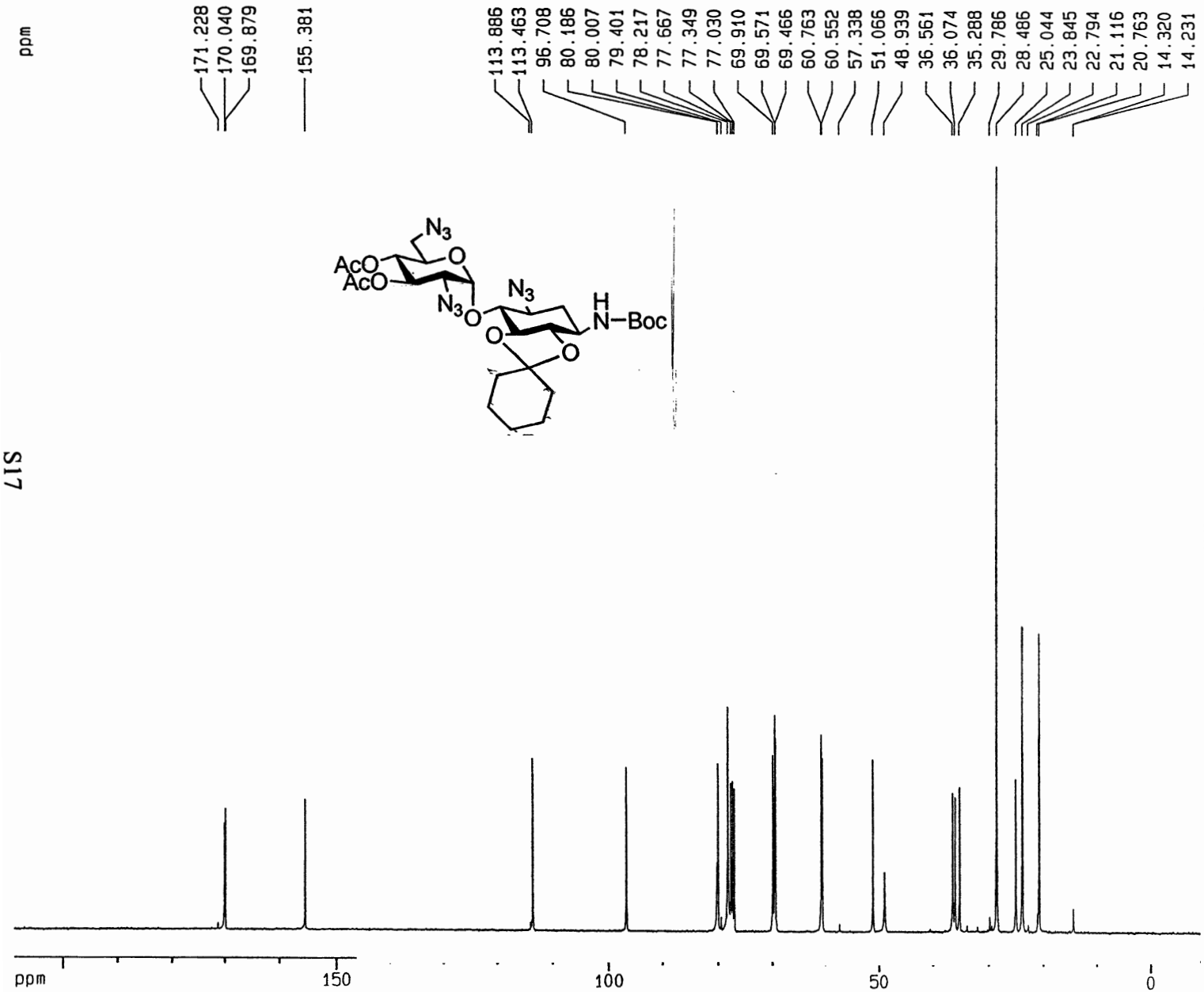


3',4'-Di-*O*-acetyl-5,6-*O*-Cyclohexylidene-1-*N*-*tert*-butoxycarbonyl-3,2',6'-triazidoneamine (11)



Standard 13C
Experiment

3',4'-Di-*O*-acetyl-5,6-*O*-Cyclohexylidene-1-*N*-*tert*-butoxycarbonyl-3,2',6'-triazidoneamine (11)



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PROCNO	1

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TD	32768
SOLVENT	CDCl3
NS	6867
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

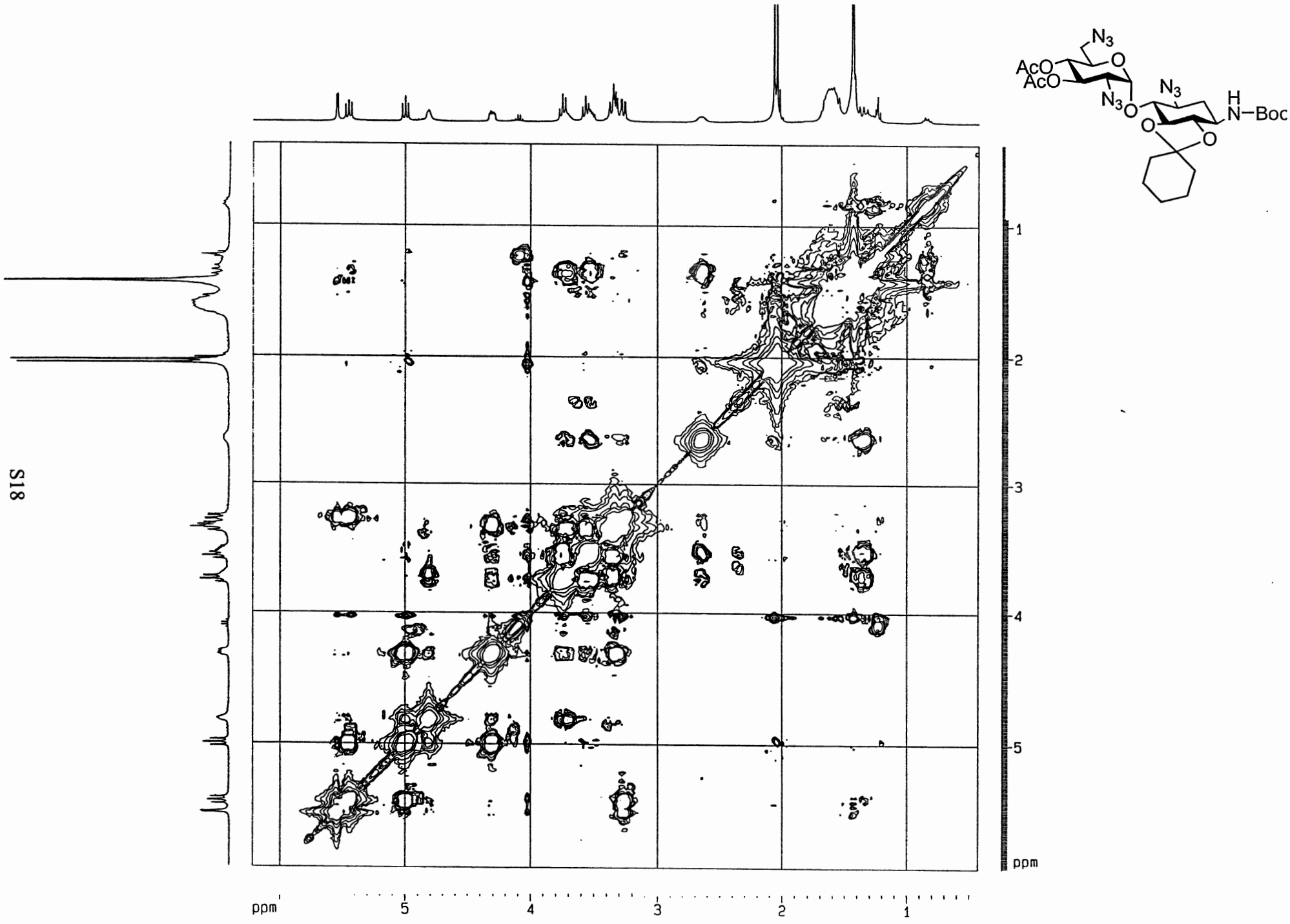
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

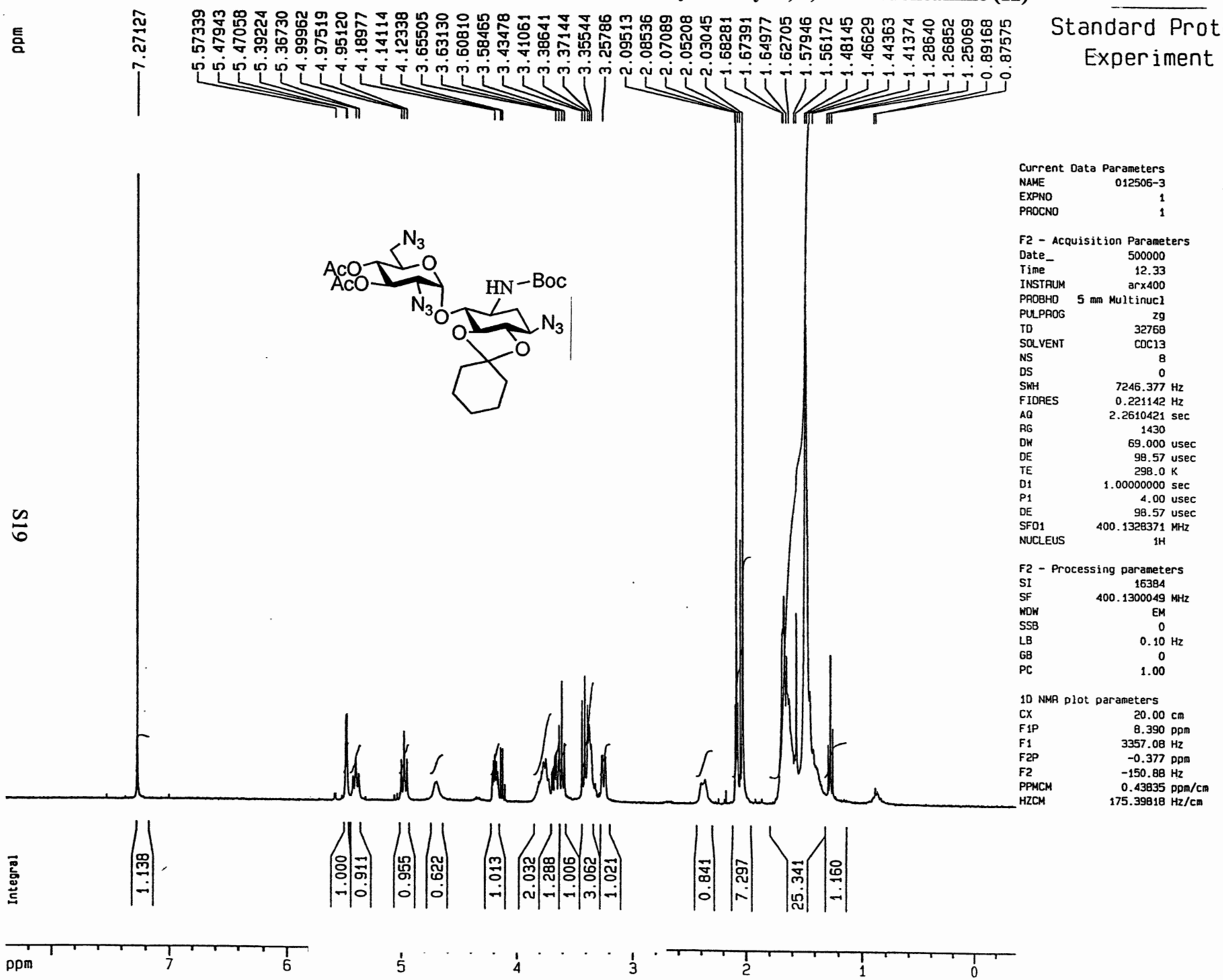
1D NMR plot parameters

CX	20.00 cm
F1P	208.565 ppm
F1	20984.33 Hz
F2P	-9.182 ppm
F2	-923.80 Hz
PPMCM	10.88735 ppm/cm
HZCM	1095.40625 Hz/cm

3',4'-Di-*O*-acetyl-5,6-*O*-Cyclohexylidene-1-*N*-*tert*-butoxycarbonyl-3,2',6'-triazidoneamine (11)

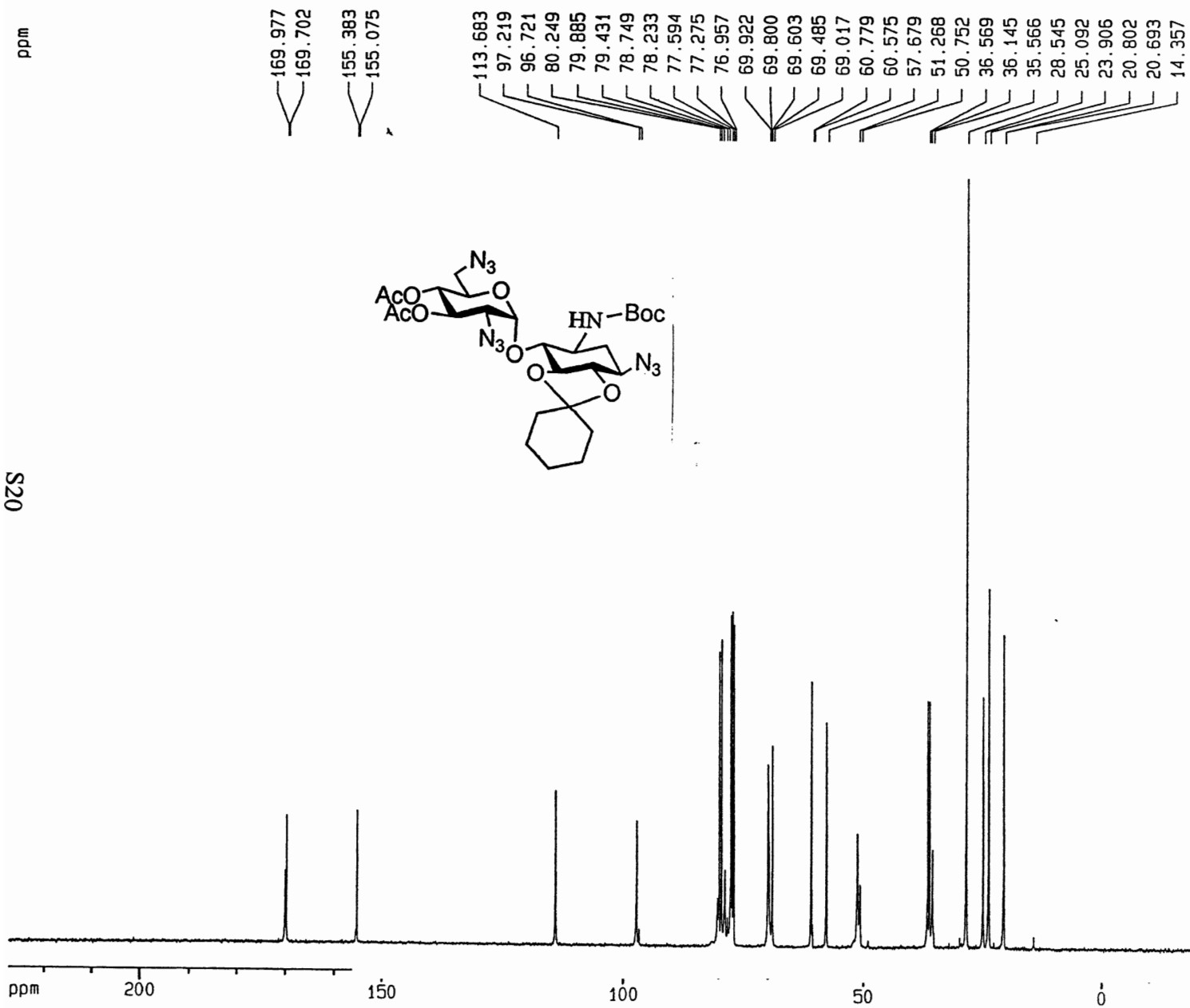


3',4'-Di-*O*-acetyl-5,6-*O*-Cyclohexylidene-3-*N*-*tert*-butoxycarbonyl-1,2',6'-triazidoneamine (12)



Standard 13C
Experiment

3',4'-Di-*O*-acetyl-5,6-*O*-Cyclohexylidene-3-*N*-*tert*-butoxycarbonyl-1,2',6'-triazidoneamine (12)



Current Data Parameters

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EXPNO	1
PROCNO	1

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Time	0.44
INSTRUM	arx400
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PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	20000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

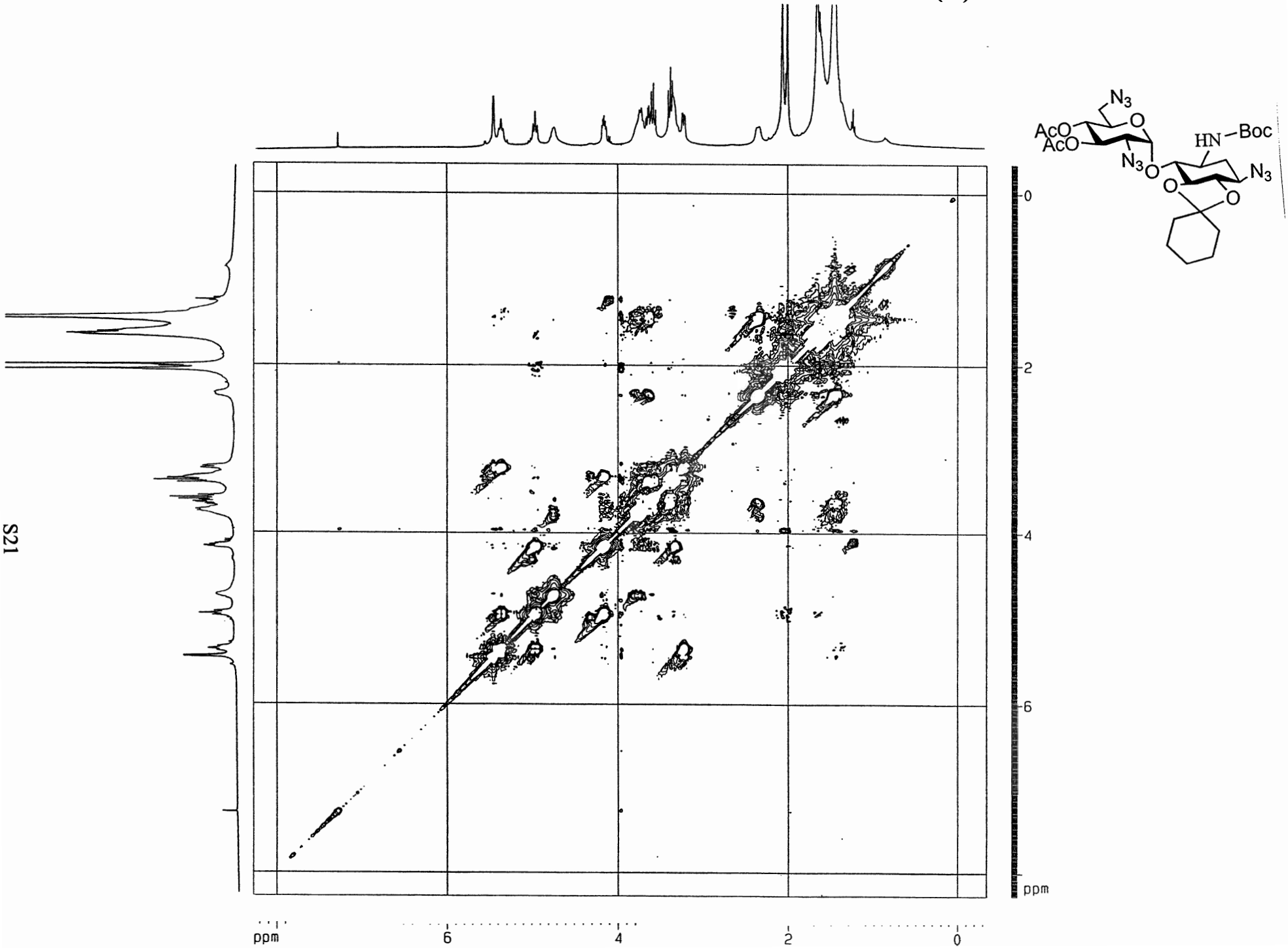
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	227.296 ppm
F1	22868.89 Hz
F2P	-21.181 ppm
F2	-2131.10 Hz
PPMCM	12.42387 ppm/cm
HZCM	1249.99988 Hz/cm

3',4'-Di-*O*-acetyl-5,6-*O*-Cyclohexylidene-3-*N*-*tert*-butoxycarbonyl-1,2',6'-triazidoneamine (12)



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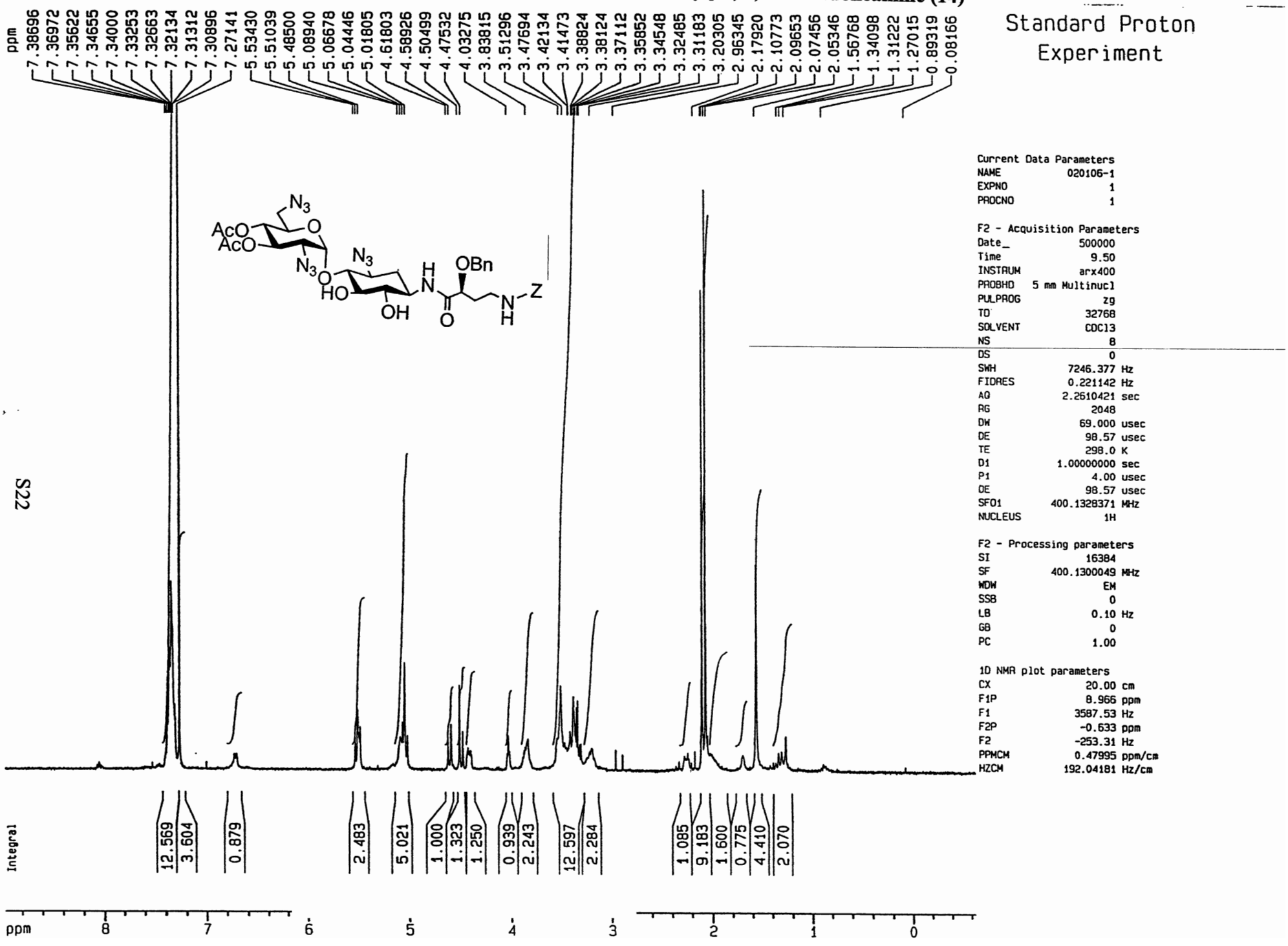
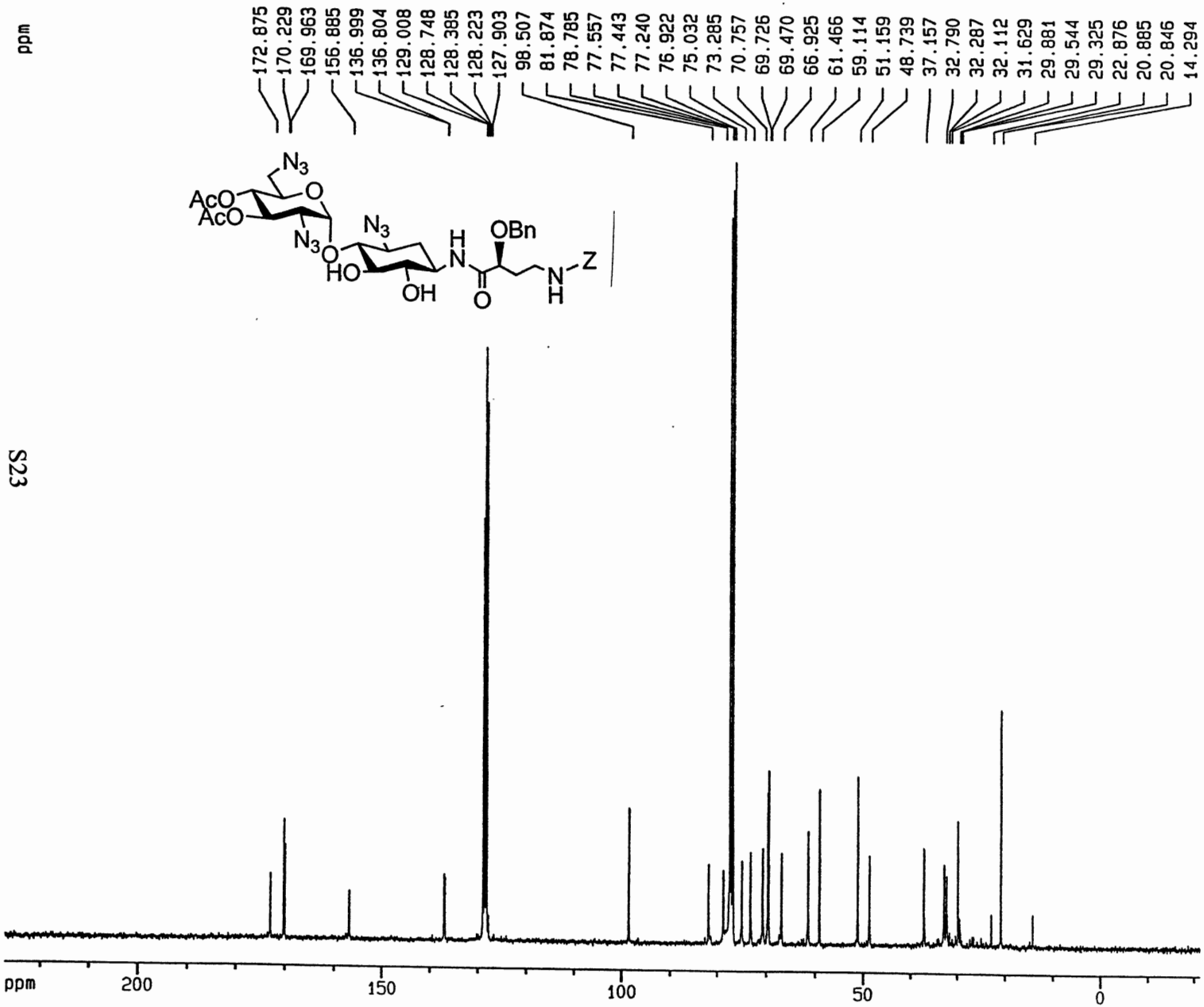


Standard 13C
Experiment

3',4'-Di-*O*-acetyl-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2',6'-triazidoneamine (14)

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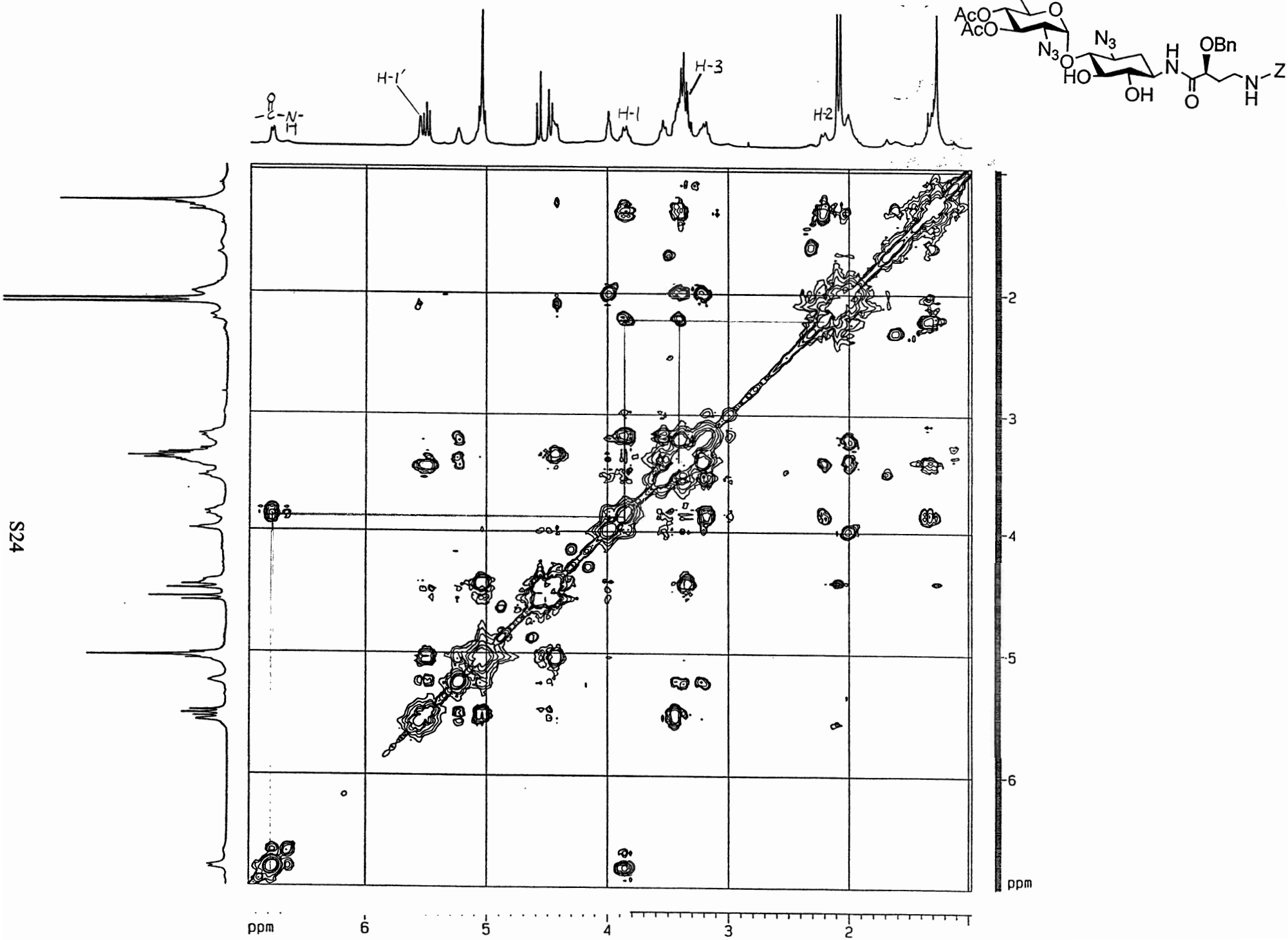
Current Data Parameters
NAME                  011306-5
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                   500000
Time                     22.28
INSTRUM                  arx400
PROBHD   5 mm Multinucl
PULPROG                 zgdc30
TD                       32768
SOLVENT                  CDCl3
NS                       30000
DS                           2
SWH                25000.000 Hz
FIDRES             0.762939 Hz
AQ                 0.6554100 sec
RG                       45500
DW                    20.000 usec
DE                     27.14 usec
TE                     300.0 K
D12               0.00002000 sec
DL5                    20.00 dB
CPDPRG                waltz16
P31                   100.00 usec
D1                0.40000001 sec
P1                      6.75 usec
DE                     27.14 usec
SFO1             100.6231179 MHz
NUCLEUS                  13C
D11               0.03000000 sec

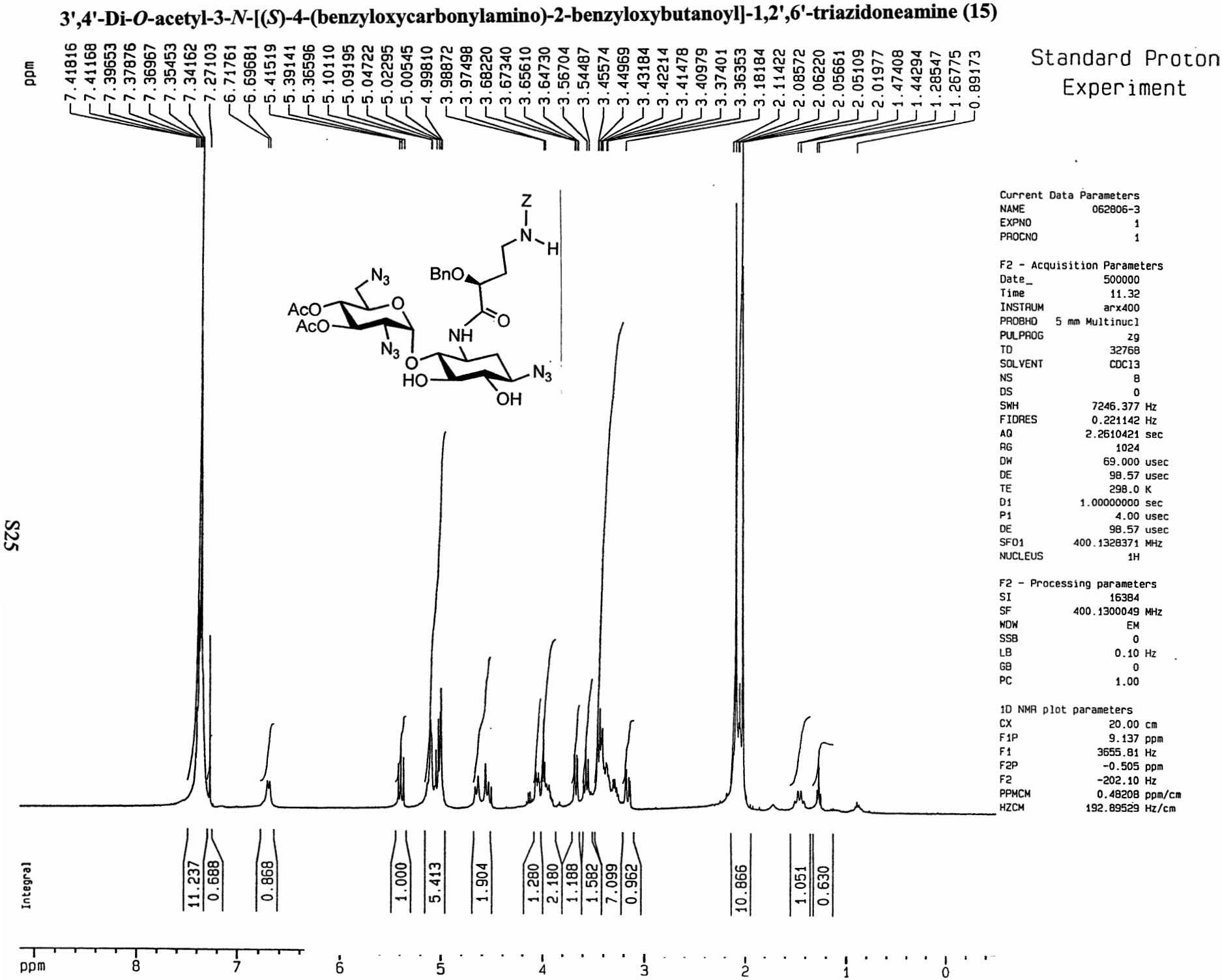
F2 - Processing parameters
SI                       16384
SF               100.6127490 MHz
WDW                         EM
SSB                          0
LB                        2.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     227.296 ppm
F1                    22868.89 Hz
F2P                    -21.181 ppm
F2                   -2131.10 Hz
PPMCM                 12.42387 ppm/cm
HZCM               1249.99988 Hz/cm
```

3',4'-Di-*O*-acetyl-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2',6'-triazidoneamine (14)

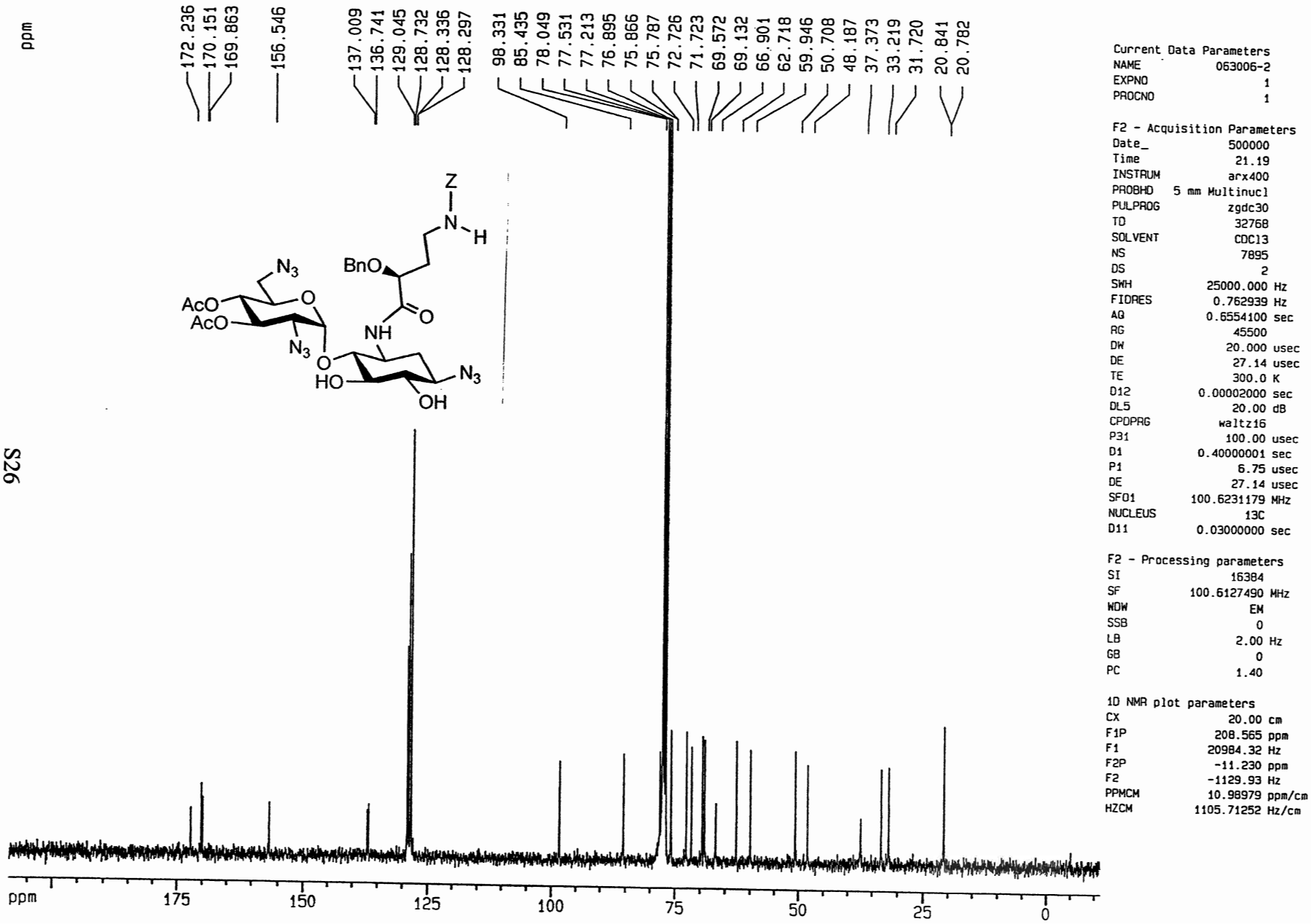


3',4'-Di-*O*-acetyl-3-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-1,2',6'-triazidoneamine (15)



Standard 13C
Experiment

3',4'-Di-*O*-acetyl-3-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-1,2',6'-triazidoneamine (15)



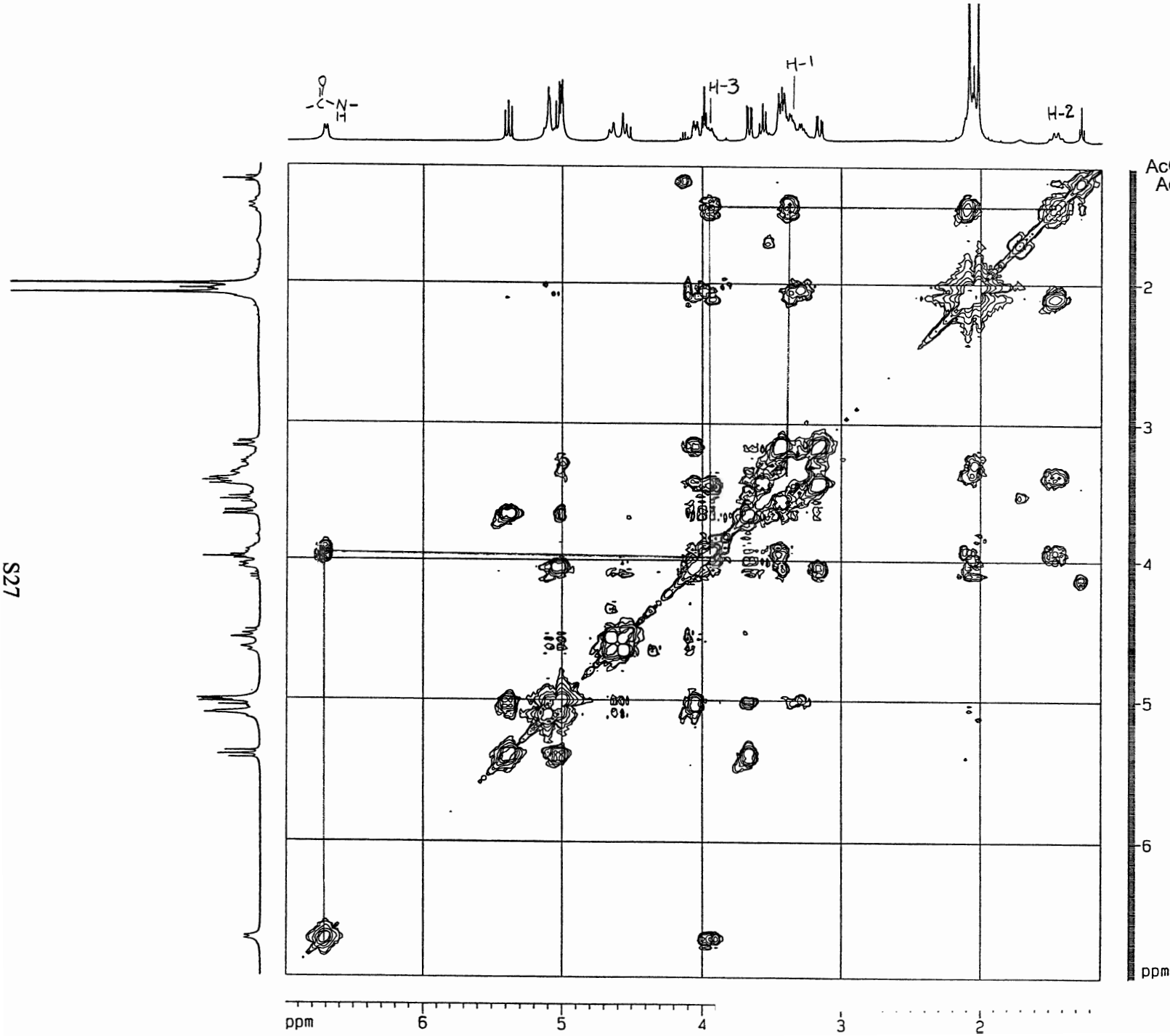
Current Data Parameters	
NAME	063006-2
EXPNO	1
PROCNO	1

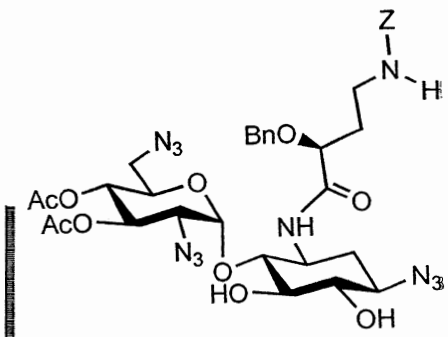
F2 - Acquisition Parameters	
Date_	500000
Time	21.19
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	7895
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters	
SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

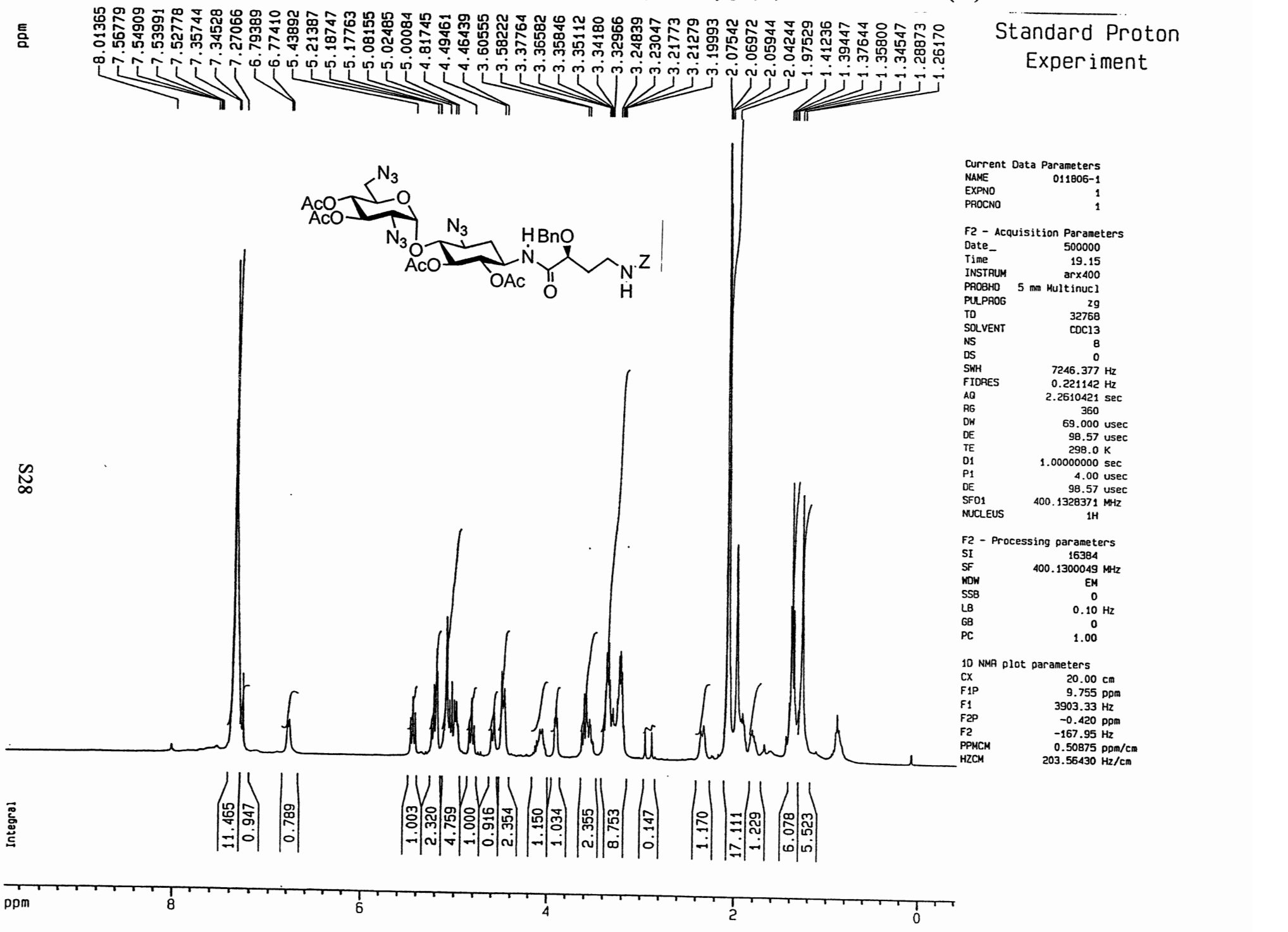
1D NMR plot parameters	
CX	20.00 cm
F1P	208.565 ppm
F1	20984.32 Hz
F2P	-11.230 ppm
F2	-1129.93 Hz
PPMCM	10.98979 ppm/cm
HZCM	1105.71252 Hz/cm

3',4'-Di-*O*-acetyl-3-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-1,2',6'-triazidoneamine (15)



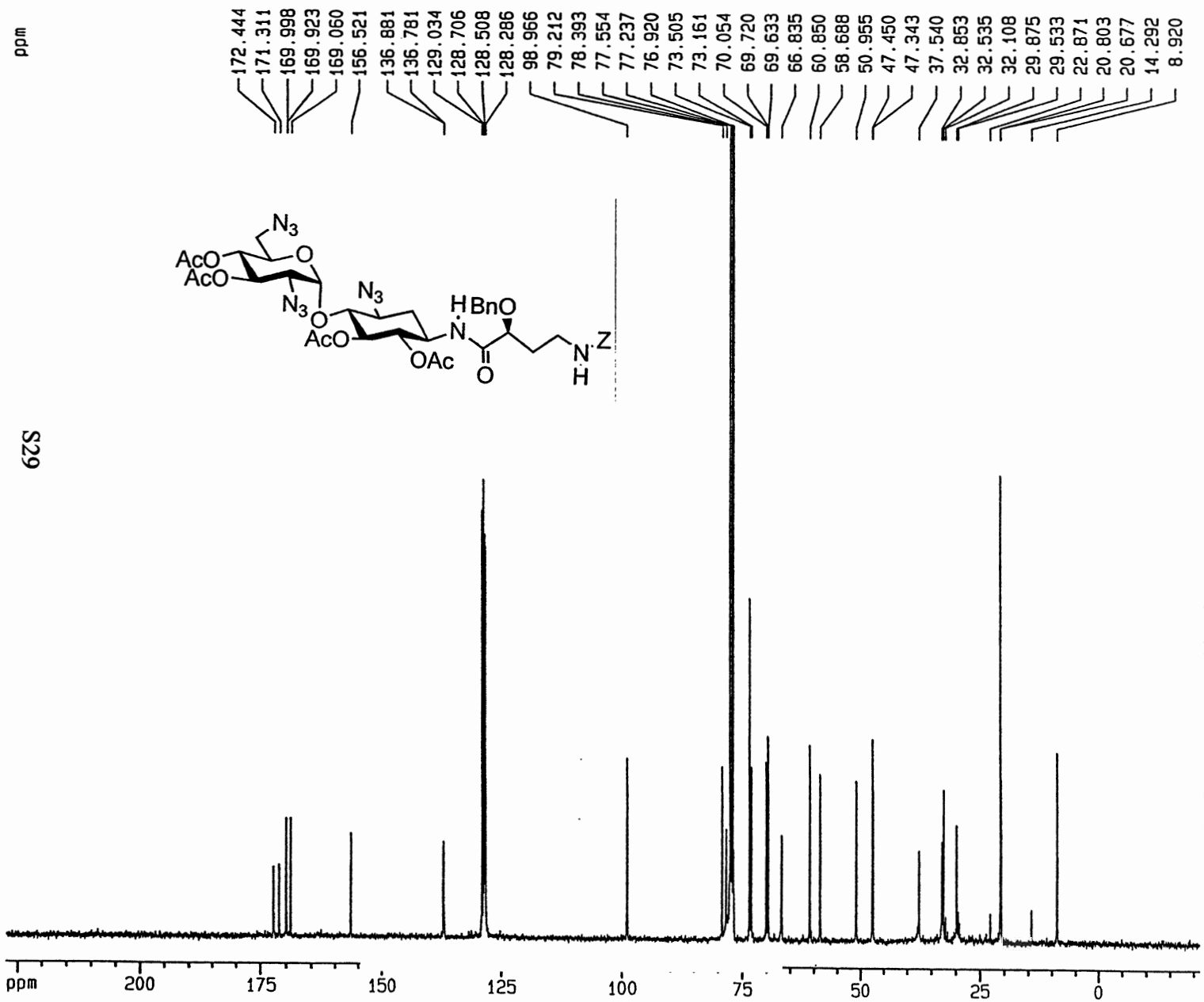


3',4',5,6-Tetra-*O*-acetyl-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2',6'-triazidoneamine (16)



Standard 13C
Experiment

3',4',5,6-Tetra-*O*-acetyl-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2',6'-triazidoneamine (16)



Current Data Parameters

NAME	011806-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	20.37
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	30000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

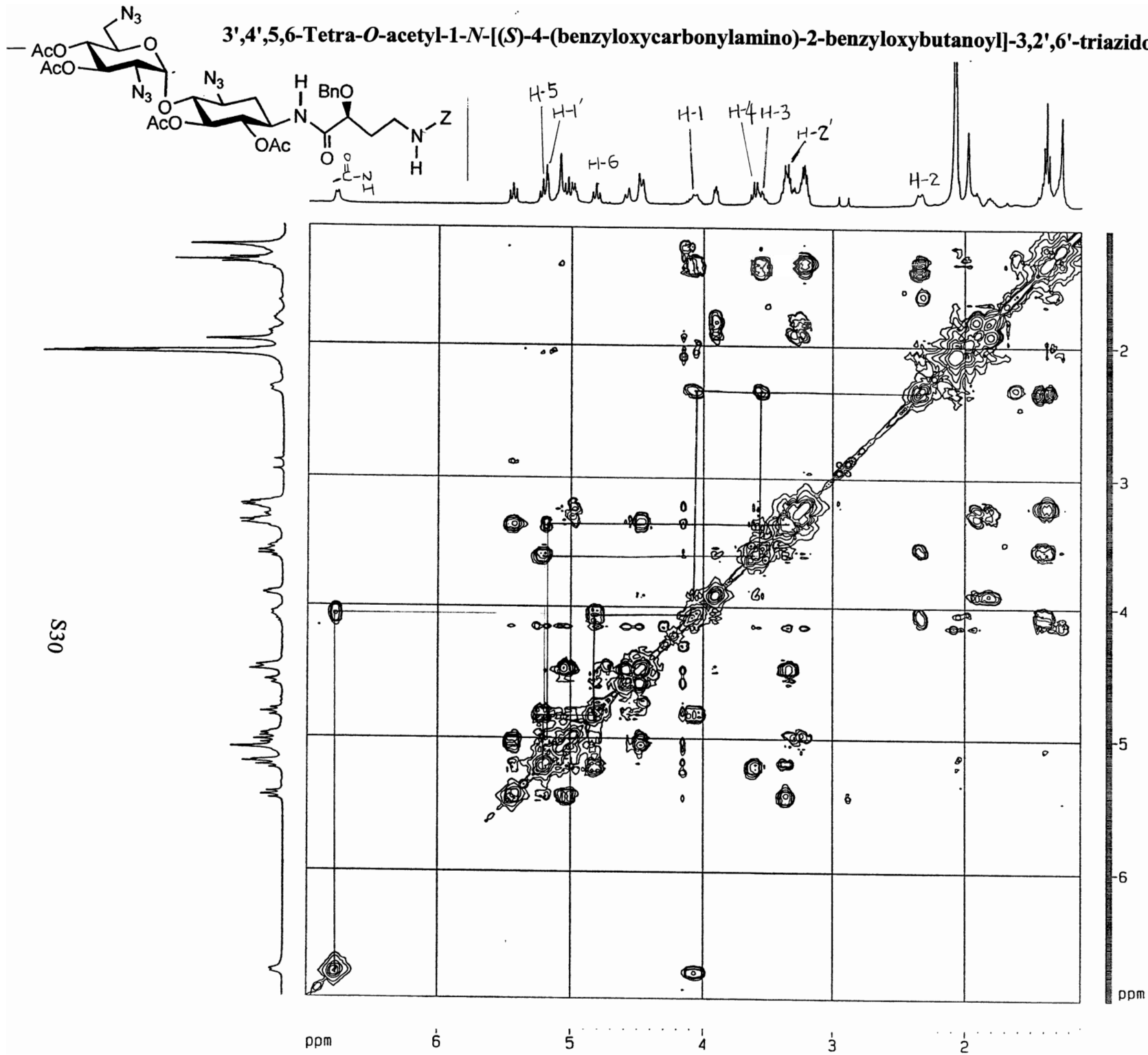
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

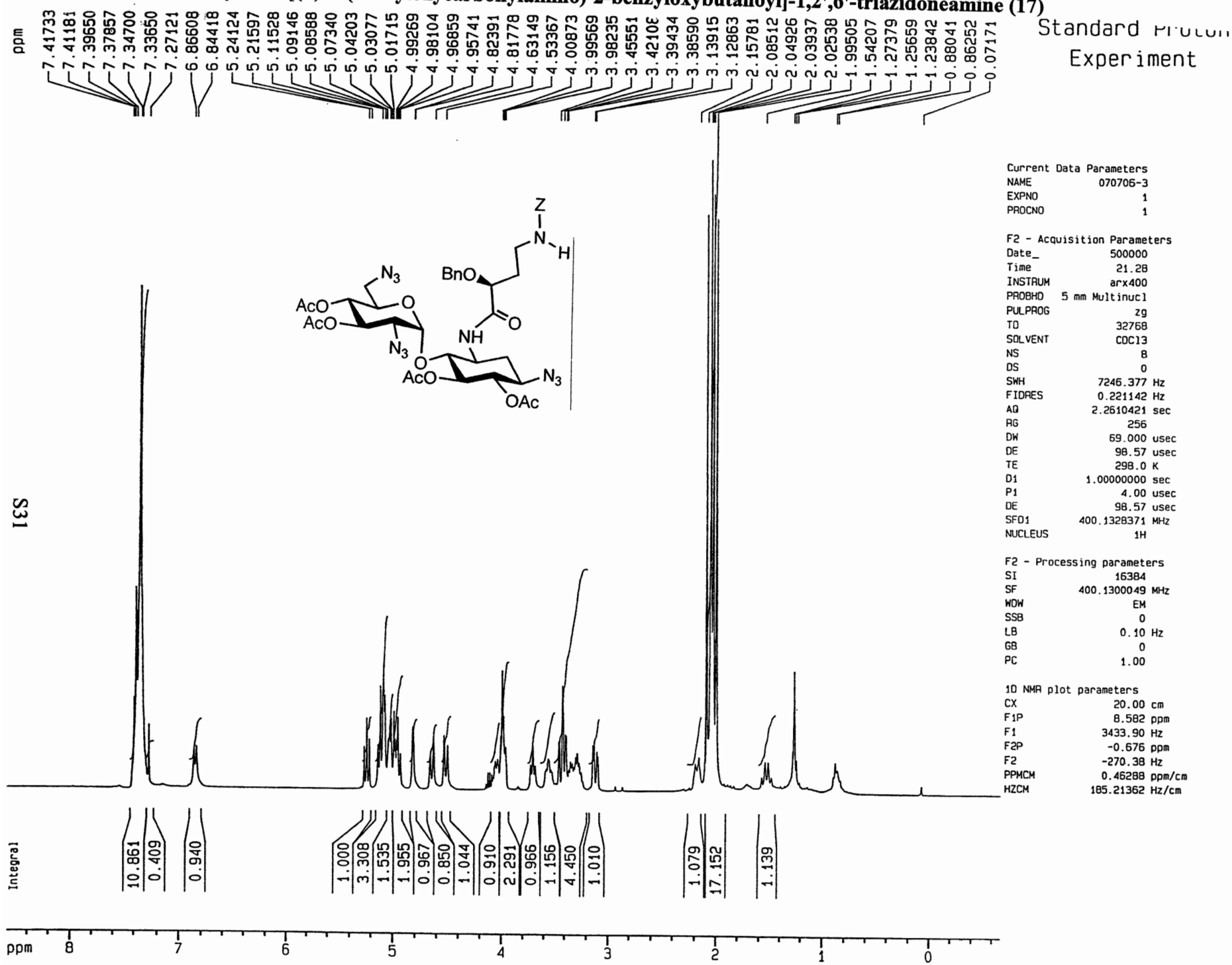
1D NMR plot parameters

CX	20.00 cm
F1P	227.296 ppm
F1	22868.89 Hz
F2P	-21.181 ppm
F2	-2131.10 Hz
PPMCM	12.42387 ppm/cm
HZCM	1249.99988 Hz/cm

3',4',5,6-Tetra-*O*-acetyl-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2',6'-triazidoneamine (16)

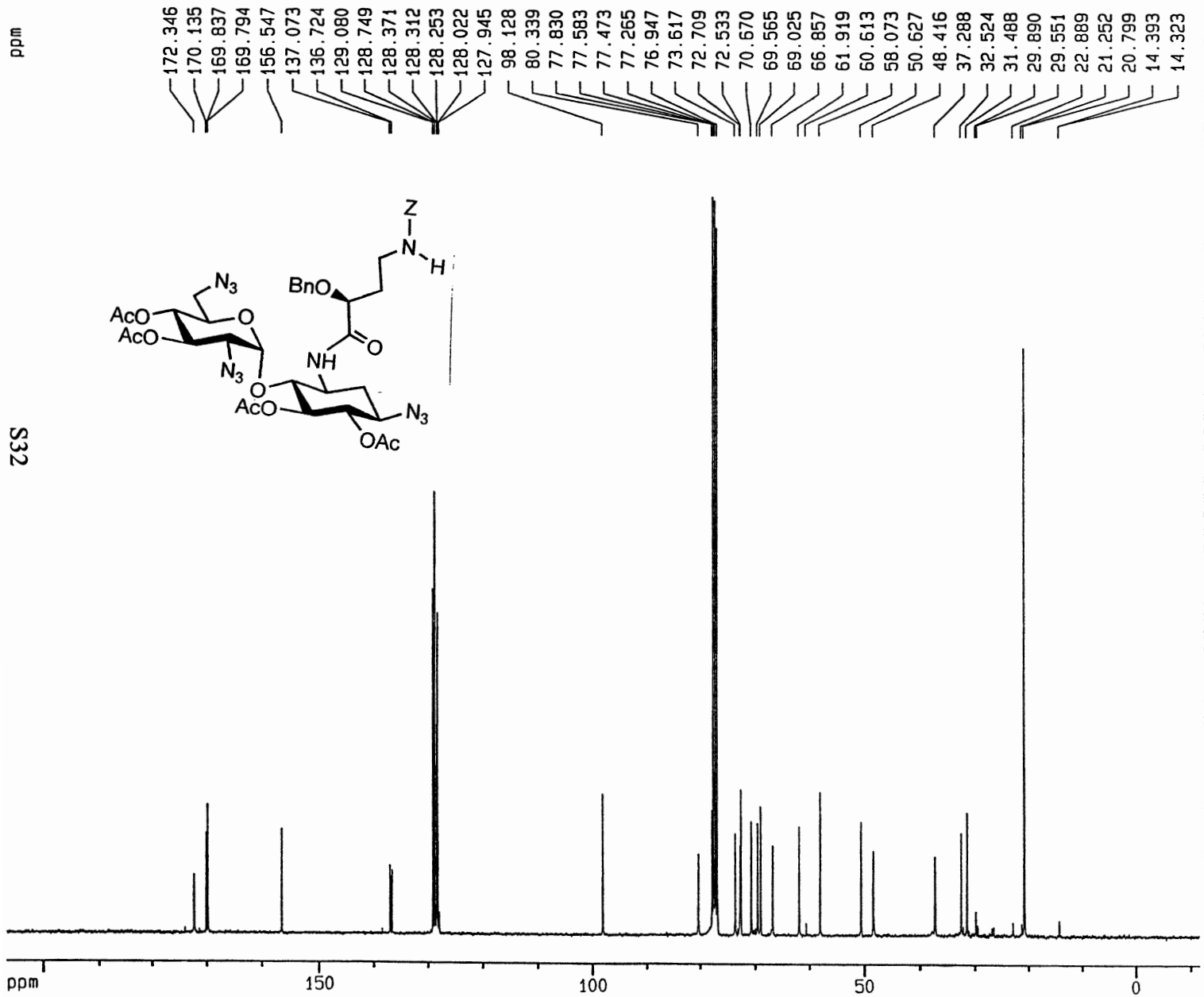


3',4',5,6-Tetra-*O*-acetyl-3-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-1,2',6'-triazidoneamine (17)



Standard 13C
Experiment

3',4',5,6-Tetra-*O*-acetyl-3-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-1,2',6'-triazidoneamine (17)



Current Data Parameters

NAME	070706-4
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	23.02
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	20000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

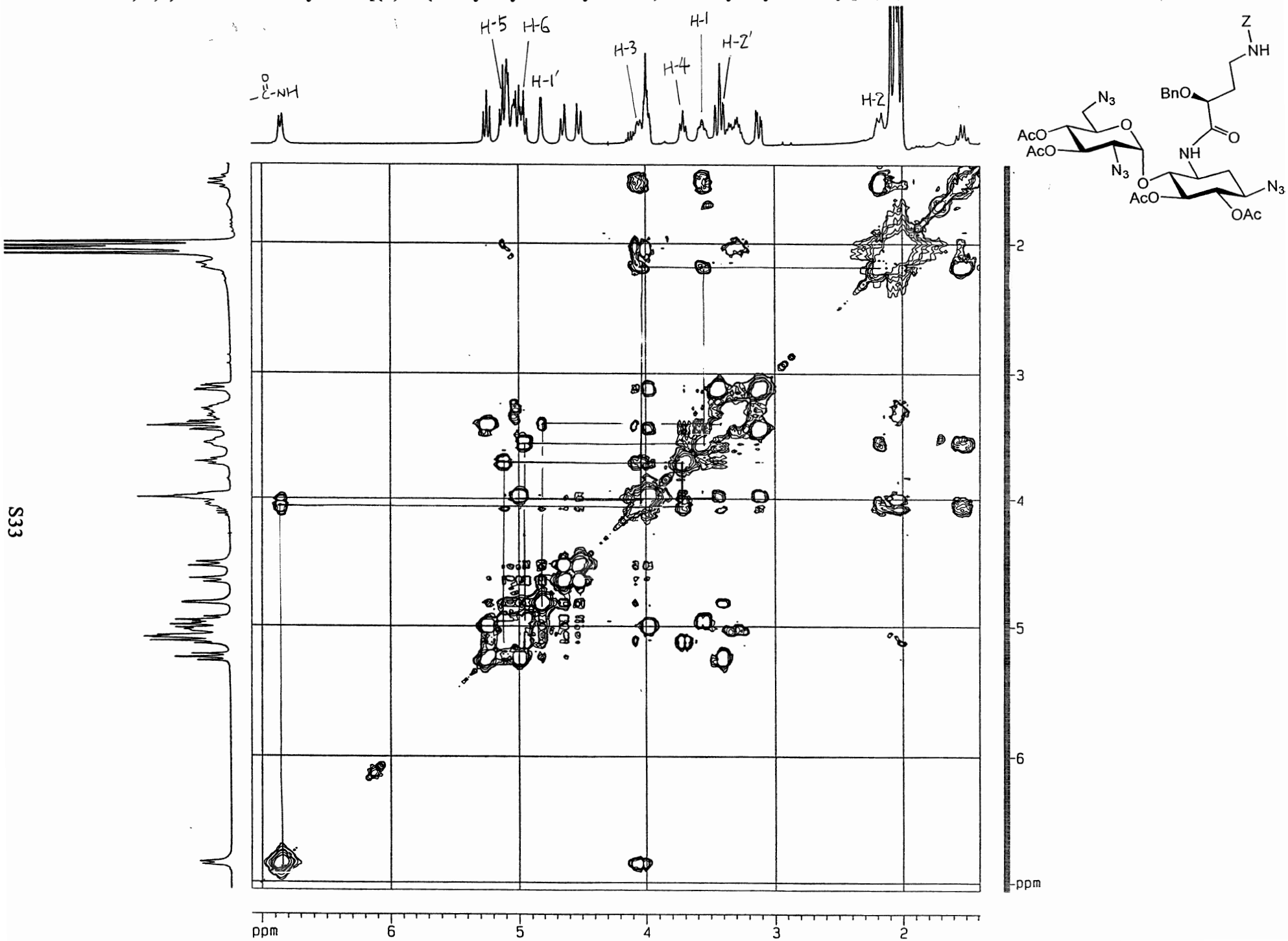
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

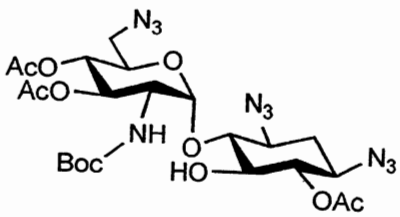
1D NMR plot parameters

CX	20.00 cm
F1P	206.517 ppm
F1	20778.20 Hz
F2P	-11.523 ppm
F2	-1159.37 Hz
PPMCM	10.90198 ppm/cm
HZCM	1096.87854 Hz/cm

3',4',5,6-Tetra-*O*-acetyl-3-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-1,2',6'-triazidoneamine (17)



3',4',6-Tri-*O*-acetyl-2'-*N*-*tert*-butoxycarbonyl-1,3,6'-triazidoneamine (18)



Standard Proton Experiment

ppm: 7.96551, 7.31696, 7.27068, 5.68232, 5.45044, 5.43804, 5.17133, 5.14598, 5.05406, 5.02930, 5.00523, 4.89120, 4.86666, 4.84212, 4.10042, 4.08262, 3.37484, 3.34340, 3.33683, 3.32151, 3.30790, 2.94089, 2.85096, 2.14149, 2.06798, 2.05577, 2.04100, 2.03686, 2.03103, 2.01924, 2.00491, 1.98819, 1.97948, 1.64913, 1.61685, 1.52183, 1.42873, 1.39092, 1.25023, 1.23222, 1.21442, 0.85300

Current Data Parameters
NAME 090506-3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 17.49
INSTRUM arx400
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 128
DW 69.000 usec
DE 98.57 usec
TE 298.0 K
D1 1.00000000 sec
P1 4.00 usec
DE 98.57 usec
SFO1 400.1328371 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 16384
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.097 ppm
F1 3640.16 Hz
F2P -0.405 ppm
F2 -162.17 Hz
PPMCM 0.47514 ppm/cm
HZCM 190.11685 Hz/cm

Integral: -0.021, 0.546, 1.844, 1.236, 1.000, 0.812, 0.854, 1.365, 2.947, 4.235, 1.084, 3.253, 8.629, 1.212, 11.025

ppm 8 7 6 5 4 3 2 1 0

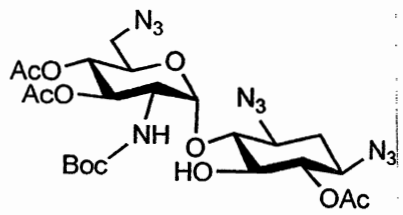
Standard 13C
Experiment

3',4',6-Tri-*O*-acetyl-2'-*N*-*tert*-butoxycarbonyl-1,3,6'-triazidoneamine (18)

ppm

171.303
169.829
163.084
155.688

98.800
98.413
80.837
80.201
77.641
77.323
77.005
76.384
75.364
74.790
74.272
71.446
69.759
69.507
60.633
58.909
58.746
58.062
53.629
51.249
31.953
29.836
28.412
28.288
21.219
21.041
20.931
20.857
14.348



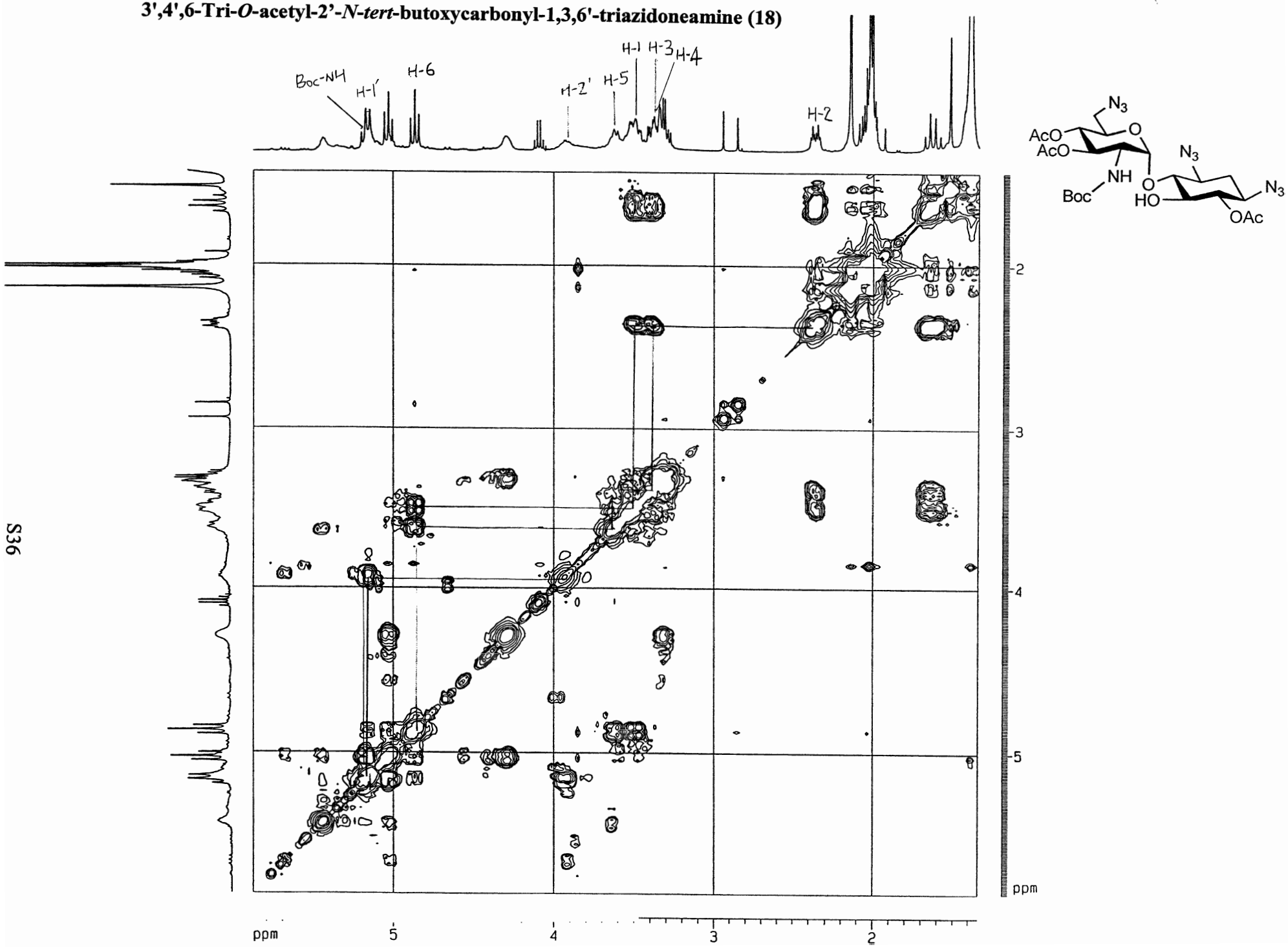
Current Data Parameters
NAME 091906-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 22.21
INSTRUM arx400
PROBHD 5 mm Multinucl
PULPROG zgdc30
TD 32768
SOLVENT CDCl3
NS 2694
DS 2
SWH 25000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554100 sec
RG 45500
DW 20.000 usec
DE 27.14 usec
TE 300.0 K
D12 0.00002000 sec
DL5 20.00 dB
CPDPRG waltz16
P31 100.00 usec
D1 0.40000001 sec
P1 6.75 usec
DE 27.14 usec
SFO1 100.6231179 MHz
NUCLEUS 13C
D11 0.03000000 sec

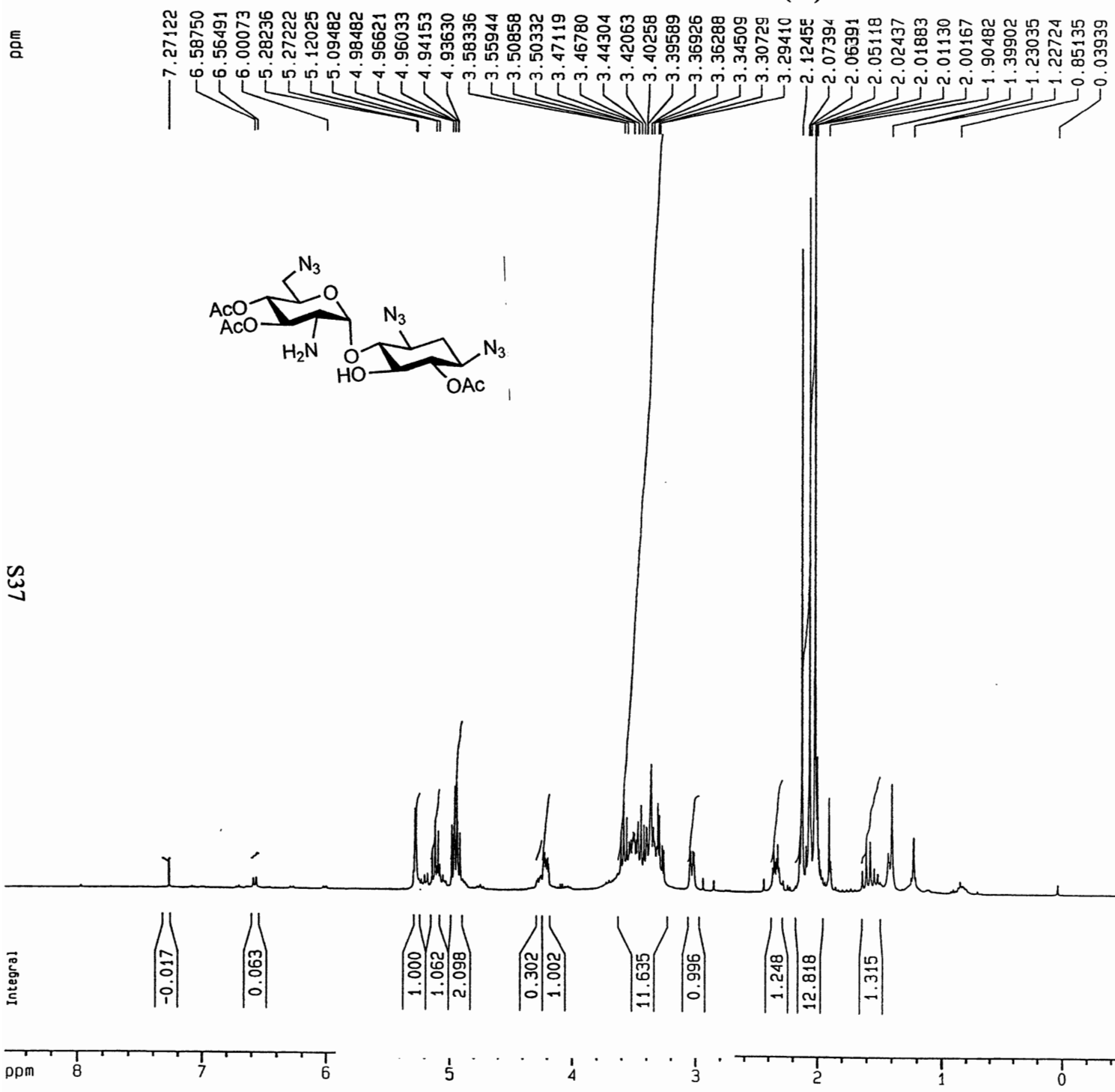
F2 - Processing parameters
SI 16384
SF 100.6127490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 207.488 ppm
F1 20875.96 Hz
F2P -7.140 ppm
F2 -718.39 Hz
PPMCM 10.73142 ppm/cm
HZCM 1079.71753 Hz/cm

3',4',6-Tri-*O*-acetyl-2'-*N*-*tert*-butoxycarbonyl-1,3,6'-triazidoneamine (18)



3',4',6-Tri-*O*-acetyl-1,3,6'-triazidoneamine (19)



Current Data Parameters	
NAME	090406-2
EXPNO	1
PROCNO	1

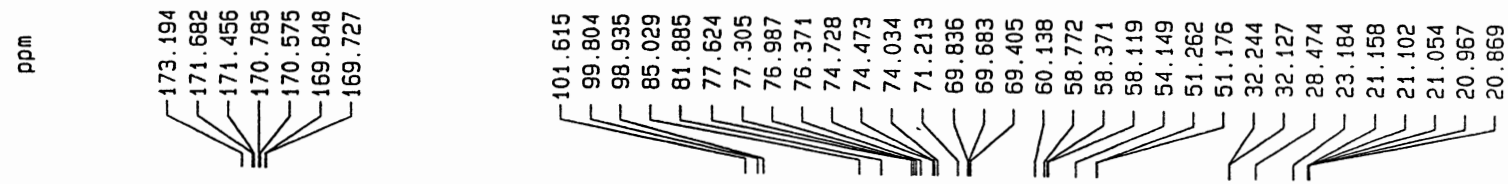
F2 - Acquisition Parameters	
Date_	500000
Time	11.55
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	90
DW	69.000 usec
DE	98.57 usec
TE	298.0 K
D1	1.00000000 sec
P1	4.00 usec
DE	98.57 usec
SFO1	400.1328371 MHz
NUCLEUS	1H

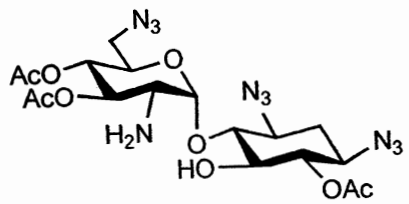
F2 - Processing parameters	
SI	16384
SF	400.1300049 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters	
CX	20.00 cm
F1P	8.567 ppm
F1	3428.11 Hz
F2P	-0.552 ppm
F2	-220.67 Hz
PPMCM	0.45595 ppm/cm
HZCM	182.43906 Hz/cm

Standard 13C
Experiment

3',4',6-Tri-*O*-acetyl-1,3,6'-triazidoneamine (19)





S38

Current Data Parameters

NAME	091906-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	20.35
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	6441
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

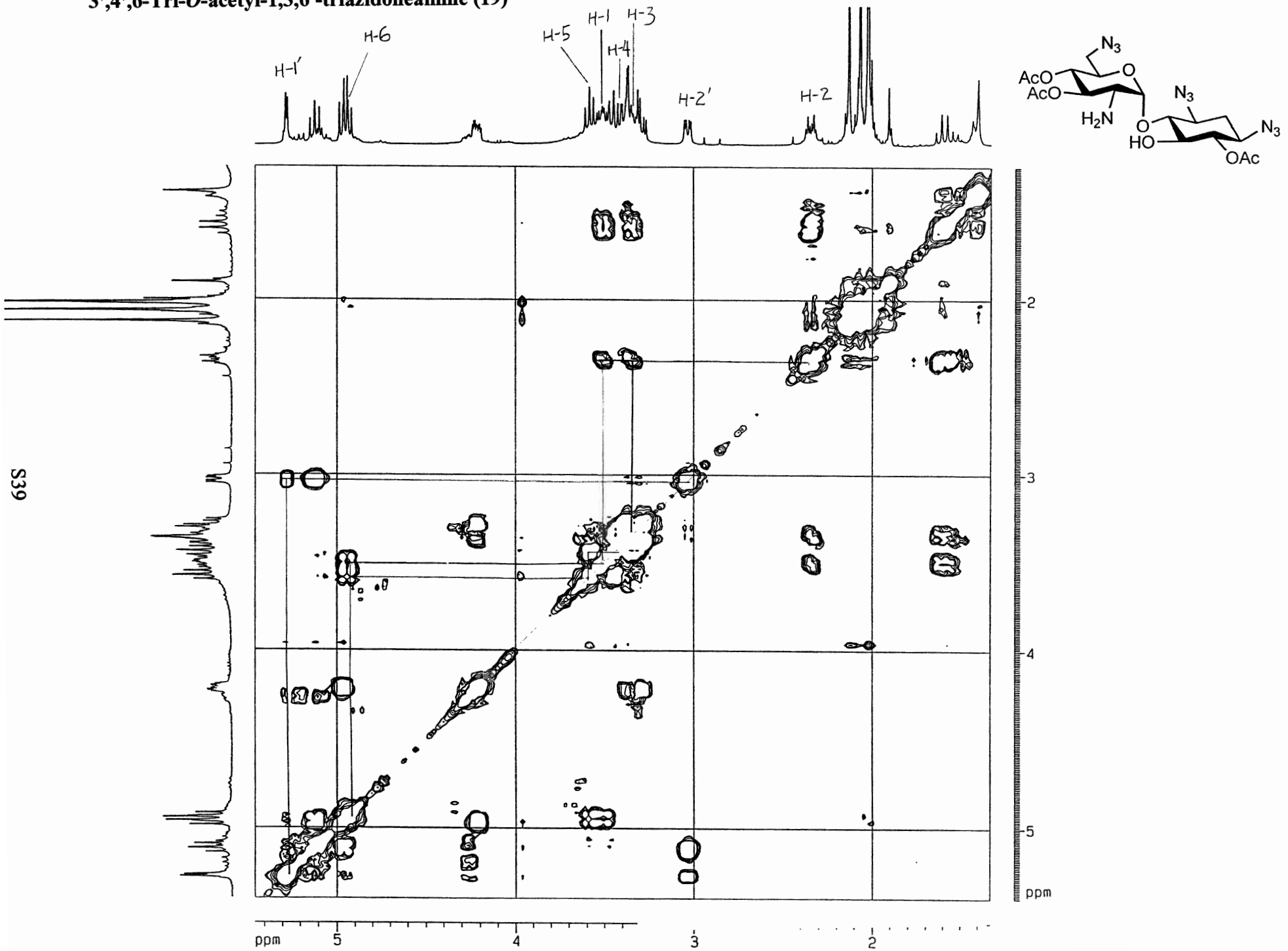
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

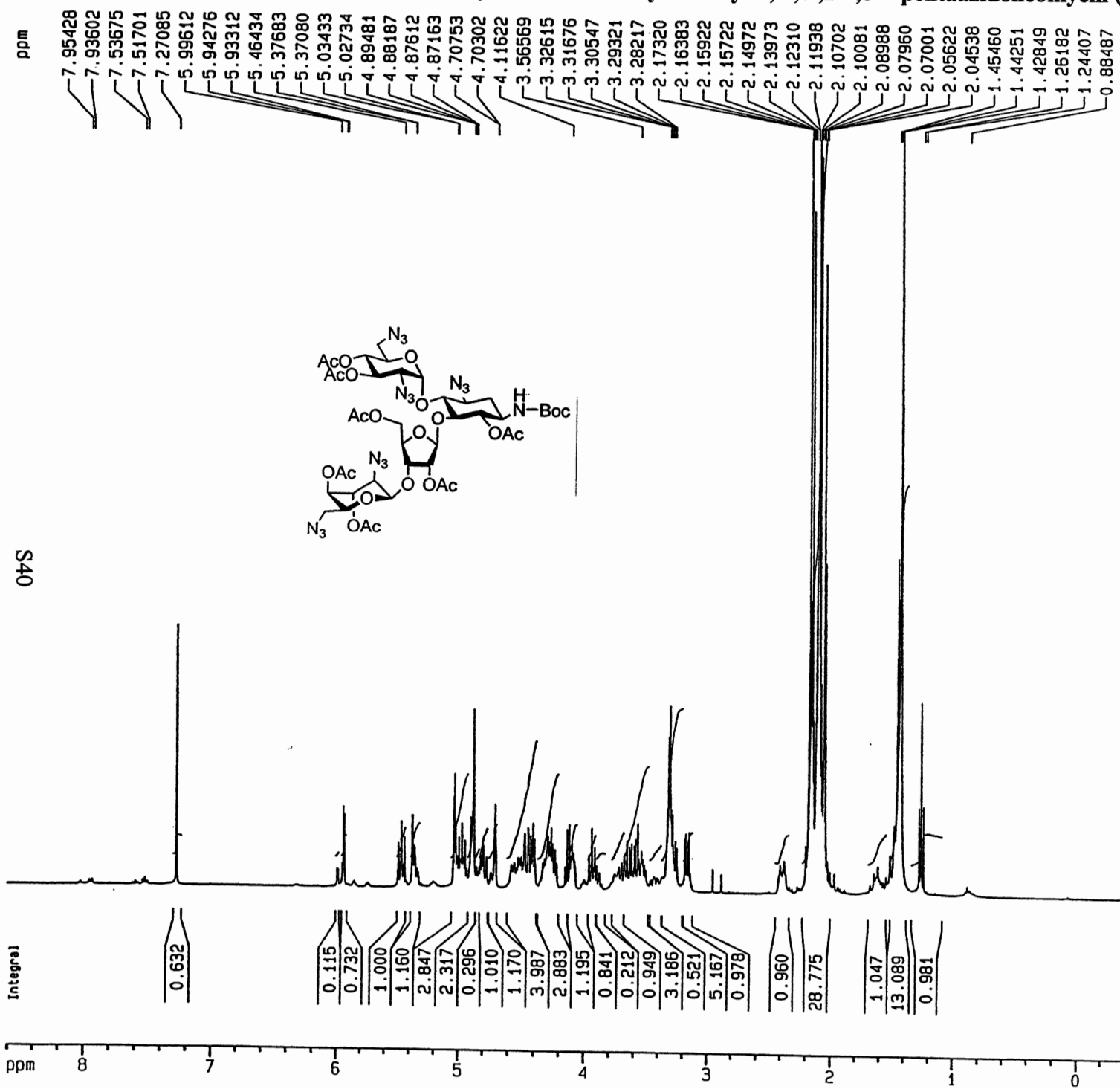
1D NMR plot parameters

CX	20.00 cm
F1P	205.733 ppm
F1	20699.37 Hz
F2P	-8.143 ppm
F2	-819.30 Hz
PPMCM	10.69381 ppm/cm
HZCM	1075.93335 Hz/cm

3',4',6-Tri-*O*-acetyl-1,3,6'-triazidoneamine (19)

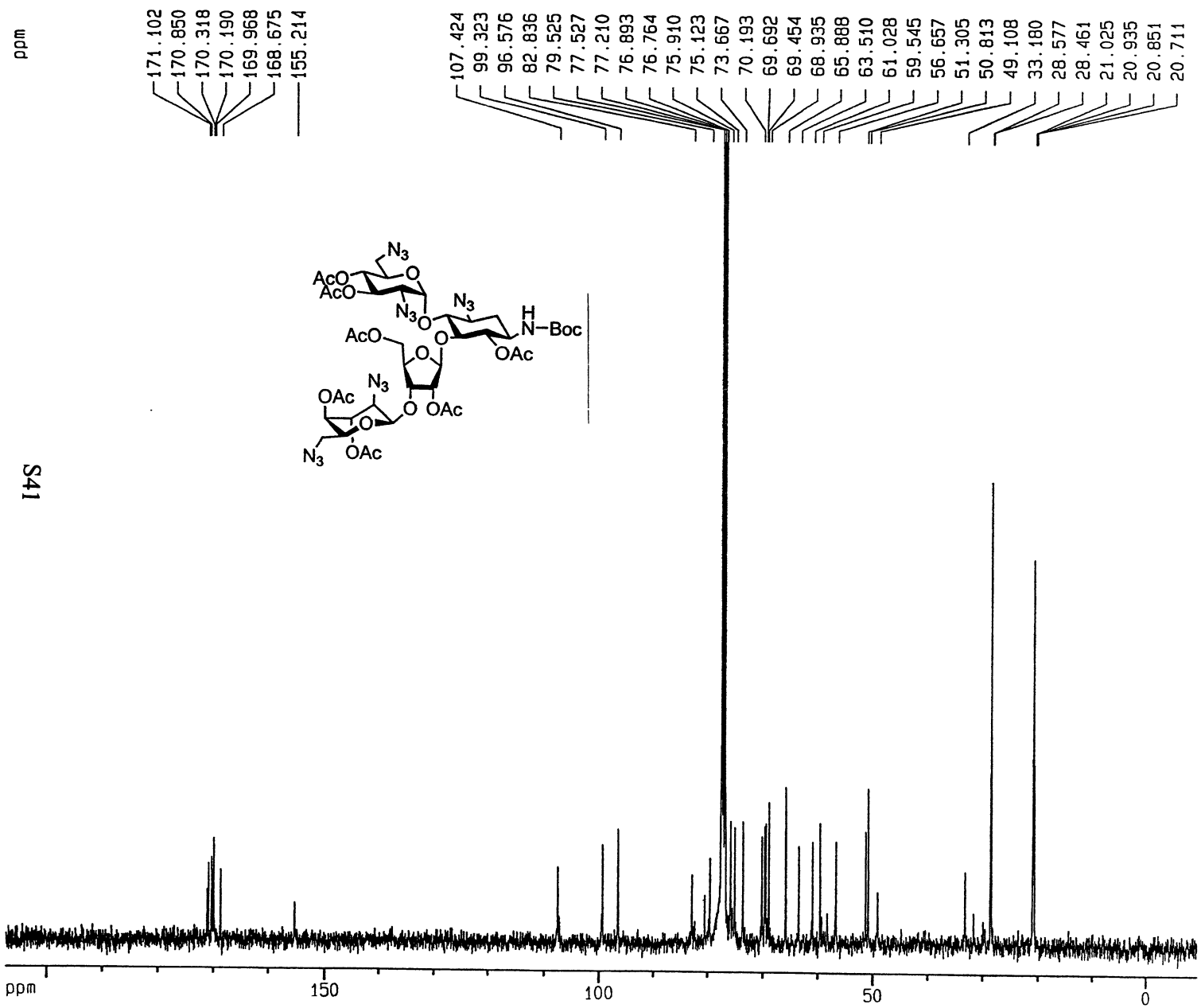


3',4',6,2'',5'',3''',4'''-Hepta-*O*-acetyl-1-*N*-*tert*-butoxycarbonyl-3,2',6',2''',6'''-pentaazidoneomycin (21)



Standard 13C
Experiment

3',4',6,2'',5'',3''',4'''-Hepta-*O*-acetyl-1-*N*-*tert*-butoxycarbonyl-3,2',6',2''',6'''-pentaazidoneomycin (21)



Current Data Parameters

NAME	062206-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	21.07
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	14708
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

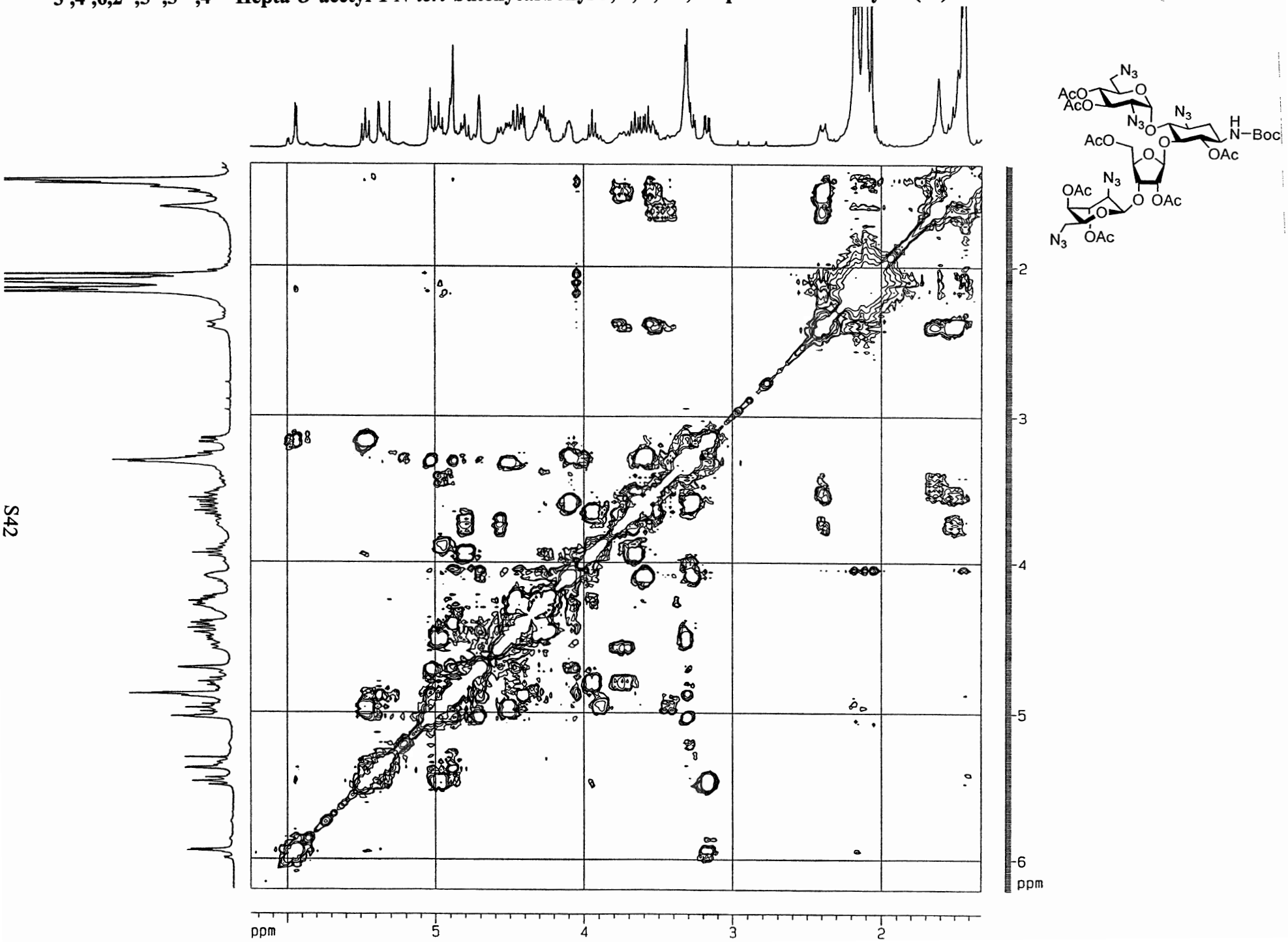
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

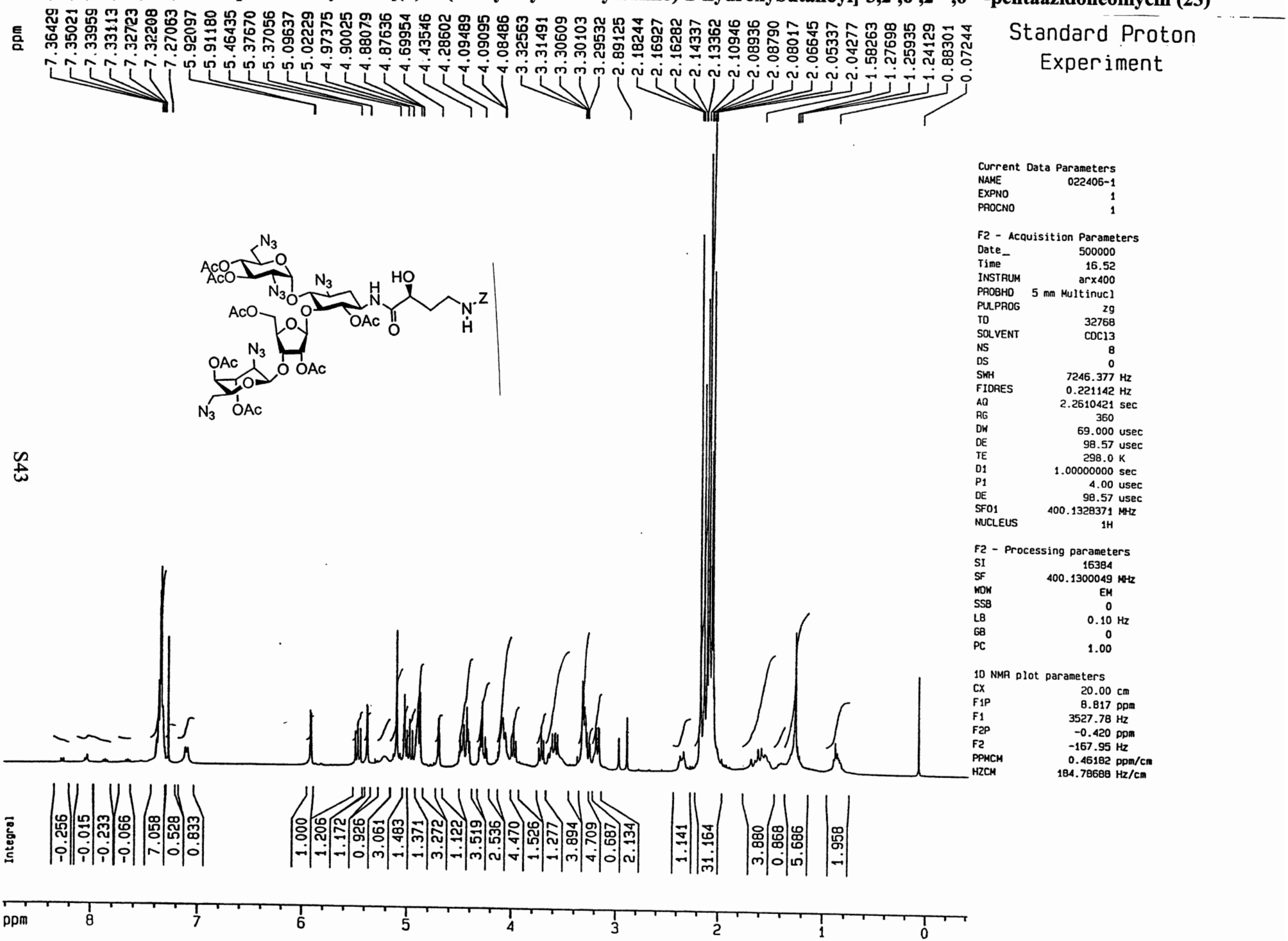
CX	20.00 cm
F1P	207.395 ppm
F1	20866.54 Hz
F2P	-9.474 ppm
F2	-953.25 Hz
PPMCM	10.84345 ppm/cm
HZCM	1090.98926 Hz/cm

3',4',6,2'',5'',3''',4'''-Hepta-*O*-acetyl-1-*N*-*tert*-butoxycarbonyl-3,2',6',2''',6'''-pentaazidoneomycin (21)



3',4',6,2'',5'',3''',4'''-Hepta-*O*-acetyl-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-hydroxybutanoyl]-3,2',6',2''',6'''-pentaazidoneomycin (23)

Standard Proton Experiment



Current Data Parameters

NAME	022406-1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	16.52
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	360
DW	69.000 usec
DE	98.57 usec
TE	298.0 K
D1	1.00000000 sec
P1	4.00 usec
DE	98.57 usec
SFO1	400.1328371 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	400.1300049 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

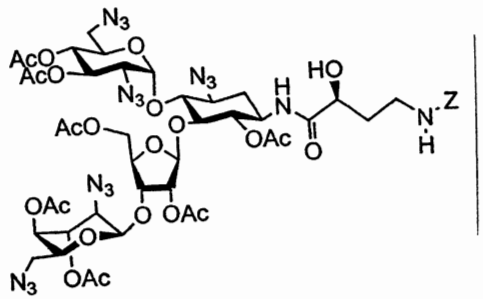
CX	20.00 cm
F1P	8.817 ppm
F1	3527.78 Hz
F2P	-0.420 ppm
F2	-167.95 Hz
PPMCM	0.46182 ppm/cm
HZCM	184.78688 Hz/cm

Standard 13C
Experiment

3',4',6,2'',5'',3''',4'''-Hepta-*O*-acetyl-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-hydroxybutanoyl]-3,2',6',2''',6'''-pentaazidoneomycin (23)

ppm

173.575 170.866 170.304 170.204 169.970 169.909 168.696 158.440 136.243 128.809 128.692 128.549 128.406 128.268 107.253 99.325 96.673 82.311 79.655 77.548 77.230 76.912 76.719 75.977 75.215 75.111 73.603 70.160 69.693 69.498 68.933 67.478 65.887 63.609 61.002 59.359 56.678 51.310 50.836 47.201 32.175 21.009 20.932 20.847 20.700



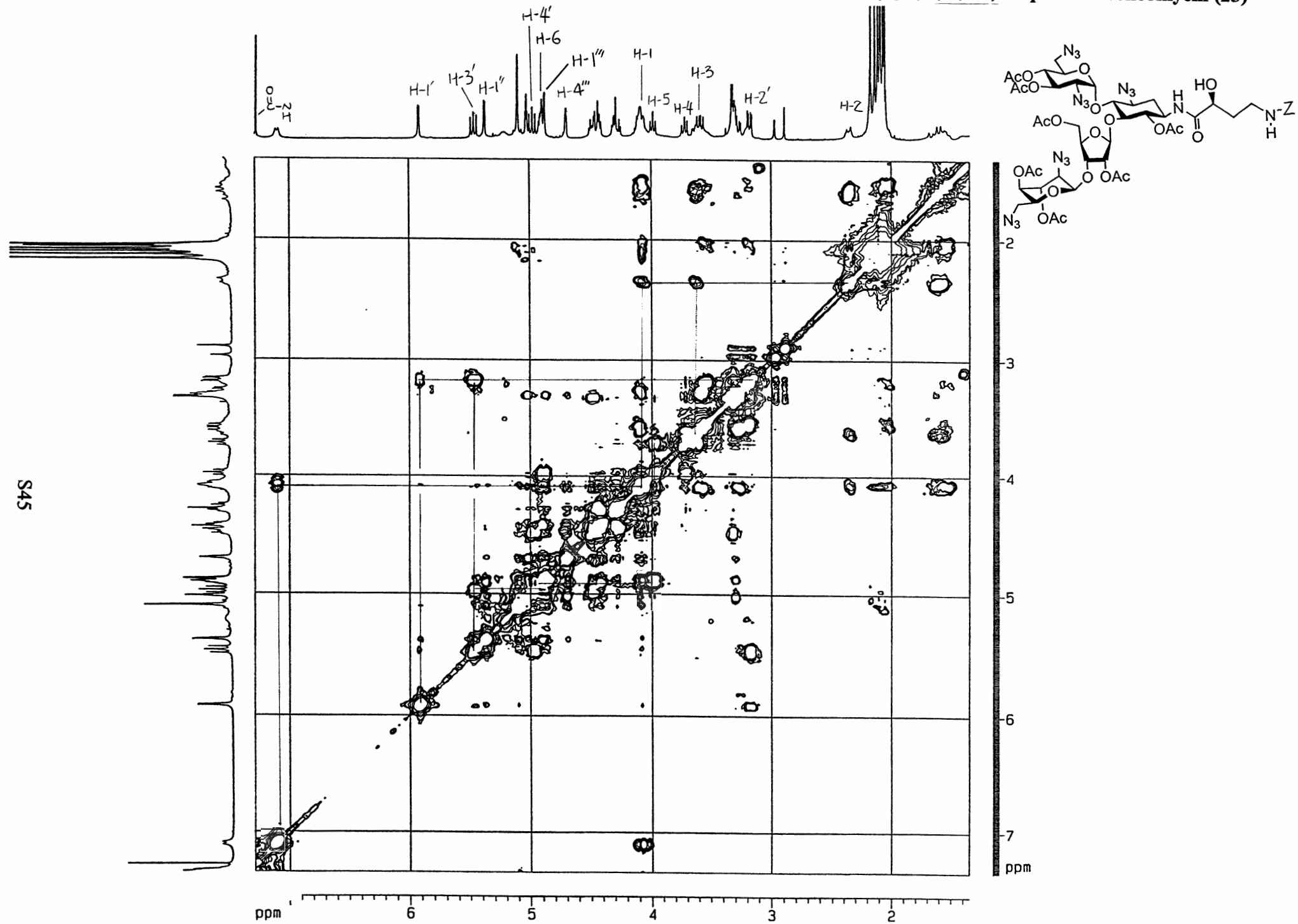
Current Data Parameters
NAME 022406-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 19.39
INSTRUM arx400
PROBHD 5 mm Multinucl
PULPROG zgdc30
TD 32768
SOLVENT CDCl3
NS 13526
DS 2
SWH 25000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554100 sec
RG 45500
DW 20.000 usec
DE 27.14 usec
TE 300.0 K
D12 0.00002000 sec
DL5 20.00 dB
CPDPRG waltz16
P31 100.00 usec
D1 0.40000001 sec
P1 6.75 usec
DE 27.14 usec
SFO1 100.6231179 MHz
NUCLEUS 13C
D11 0.03000000 sec

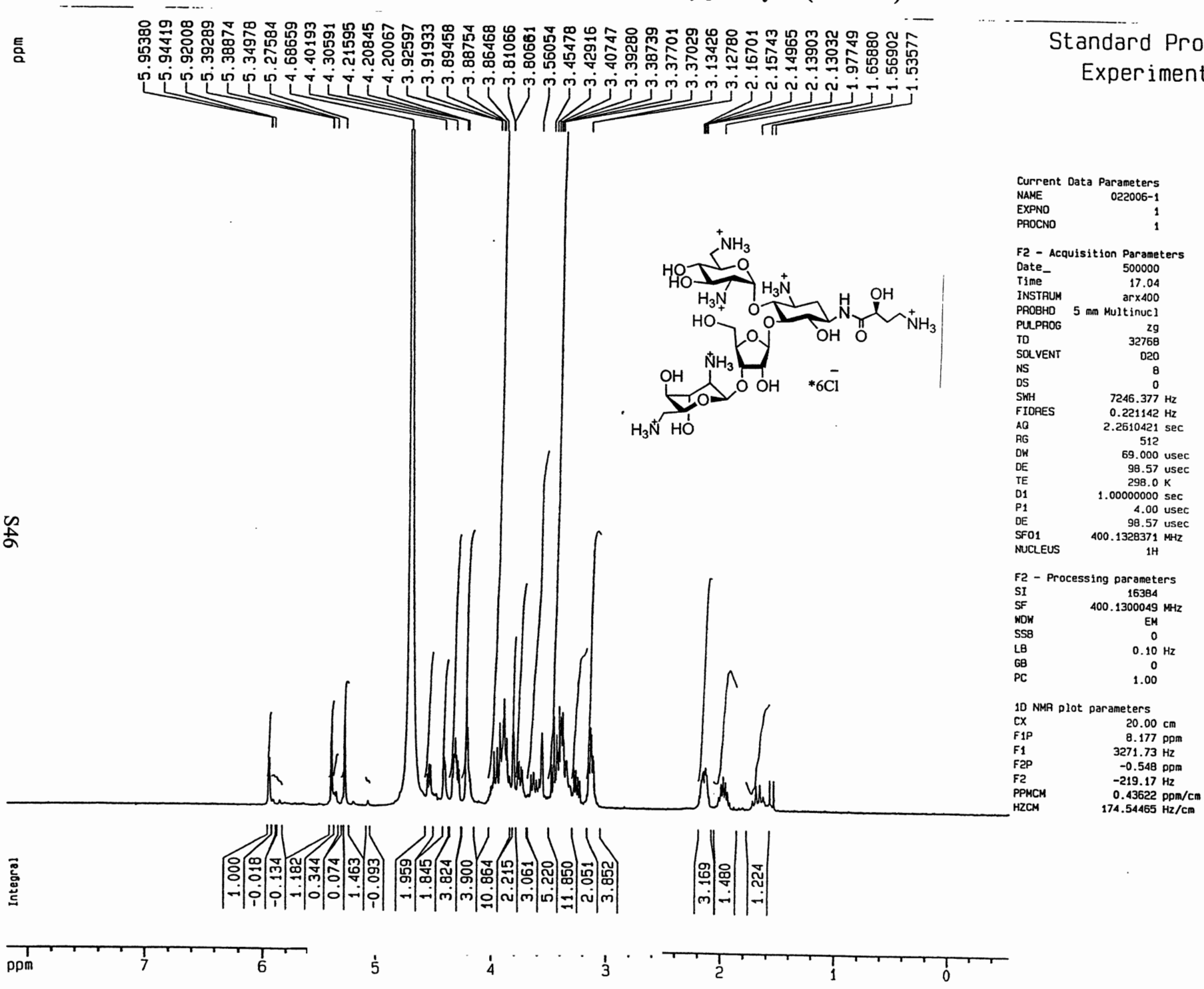
F2 - Processing parameters
SI 16384
SF 100.6127490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 205.346 ppm
F1 20660.41 Hz
F2P -7.426 ppm
F2 -747.12 Hz
PPMCM 10.63858 ppm/cm
HZCM 1070.37683 Hz/cm

3',4',6,2'',5'',3''',4'''-Hepta-*O*-acetyl-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-hydroxybutanoyl]-3,2',6',2''',6'''-pentaazidoneomycin (23)



1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neomycin (neokacin)



Standard Proton Experiment

Current Data Parameters	
NAME	022006-1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters	
Date_	500000
Time	17.04
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	D2O
NS	8
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	512
DW	69.000 usec
DE	98.57 usec
TE	298.0 K
D1	1.00000000 sec
P1	4.00 usec
DE	98.57 usec
SFO1	400.1328371 MHz
NUCLEUS	1H

F2 - Processing parameters	
SI	16384
SF	400.1300049 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters	
CX	20.00 cm
F1P	8.177 ppm
F1	3271.73 Hz
F2P	-0.548 ppm
F2	-219.17 Hz
PPMCM	0.43622 ppm/cm
HZCM	174.54465 Hz/cm

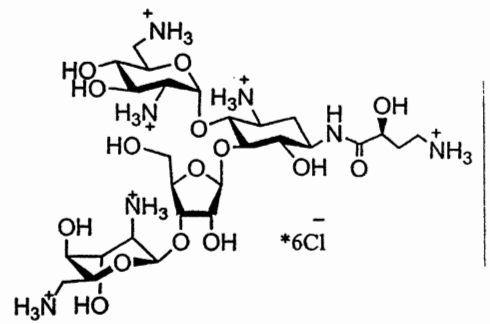
Standard 13C
Experiment

1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neomycin (neokacin)

ppm

175.809

110.457
95.840
95.570
86.030
81.394
77.719
75.376
74.178
73.815
71.045
70.423
69.813
69.392
68.739
67.963
67.600
60.417
53.970
51.161
49.288
49.199
40.739
40.474
36.927
31.426
31.142



S47

Current Data Parameters

NAME	022006-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

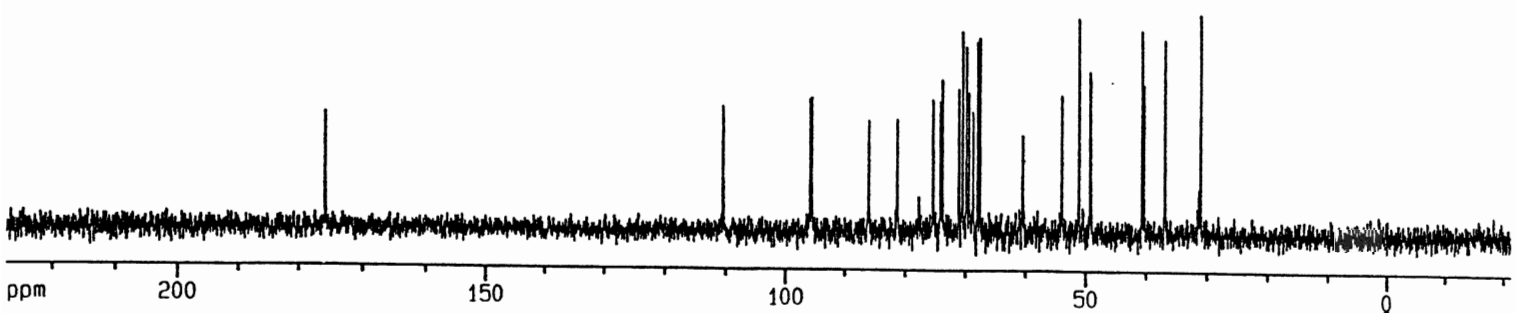
Date_	500000
Time	0.01
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	D2O
NS	40000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

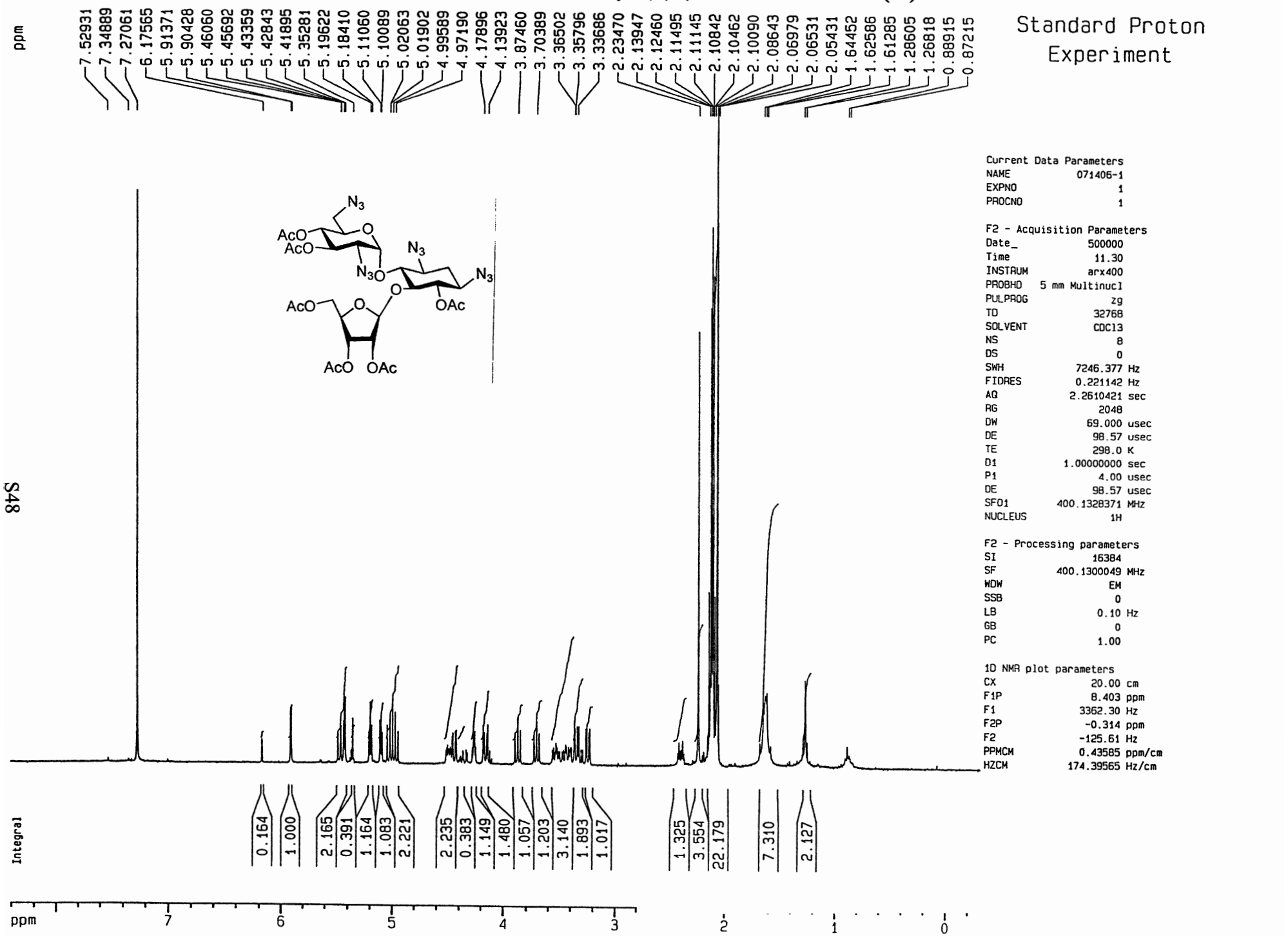
SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	227.296 ppm
F1	22868.89 Hz
F2P	-21.181 ppm
F2	-2131.10 Hz
PPMCM	12.42387 ppm/cm
HZCM	1249.99988 Hz/cm



5-*O*-(2,3,5-Tri-*O*-acetyl-β-D-ribofuranosyl)-6,3',4'-Tri-*O*-acetyl-1,3,2',6'-tetraazidoneamine (25)



Current Data Parameters

NAME	071406-1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	11.30
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	2048
DW	69.000 usec
DE	98.57 usec
TE	298.0 K
D1	1.00000000 sec
P1	4.00 usec
DE	98.57 usec
SFO1	400.1328371 MHz
NUCLEUS	1H

F2 - Processing parameters

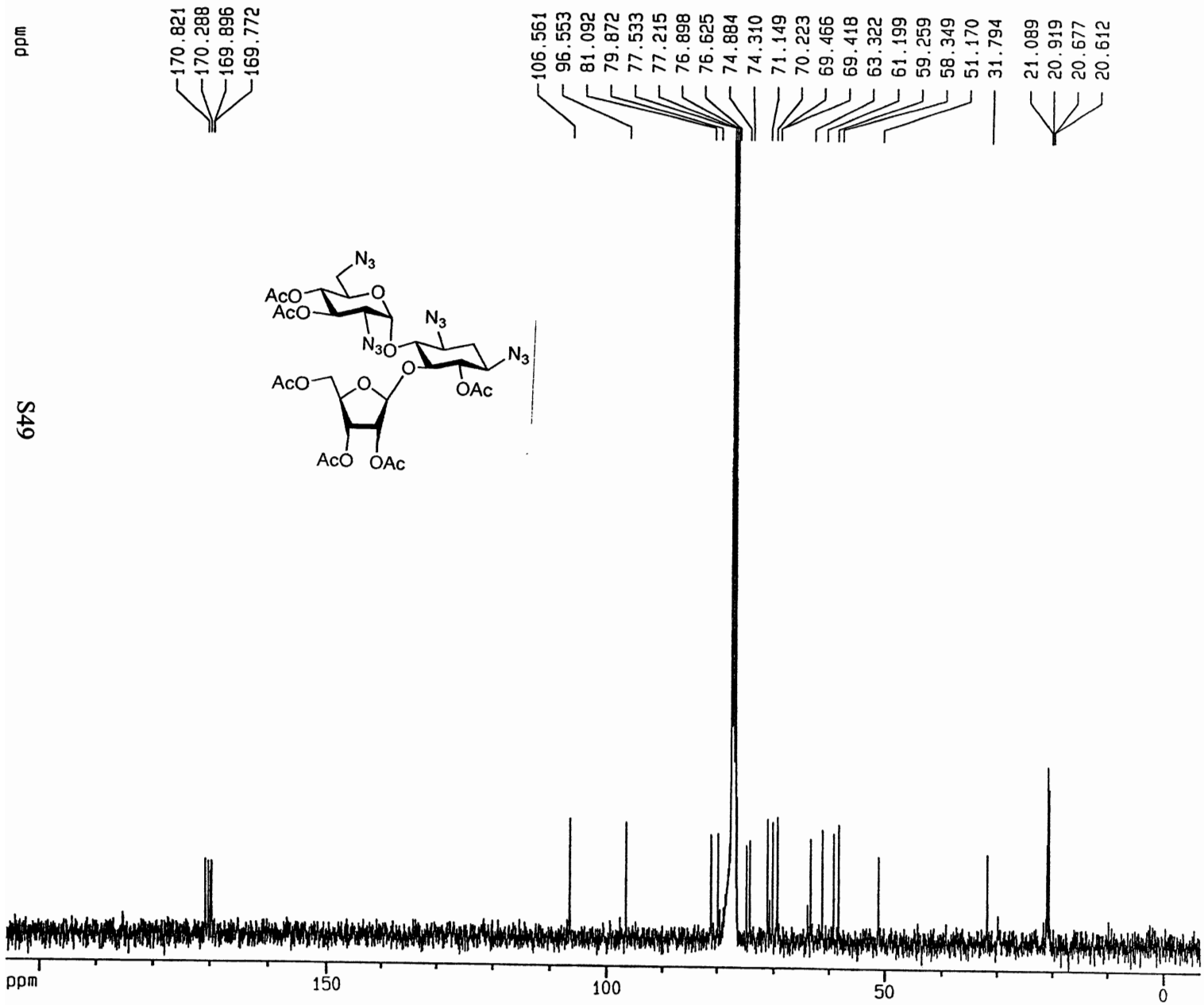
SI	16384
SF	400.1300049 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.403 ppm
F1	3362.30 Hz
F2P	-0.314 ppm
F2	-125.61 Hz
PPMCM	0.43585 ppm/cm
HZCM	174.39565 Hz/cm

Standard 13C
Experiment

5-*O*-(2,3,5-Tri-*O*-acetyl-β-D-ribofuranosyl)-6,3',4'-Tri-*O*-acetyl-1,3,2',6'-tetraazidoneamine (25)



Current Data Parameters

NAME	071906-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	0.58
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	30000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

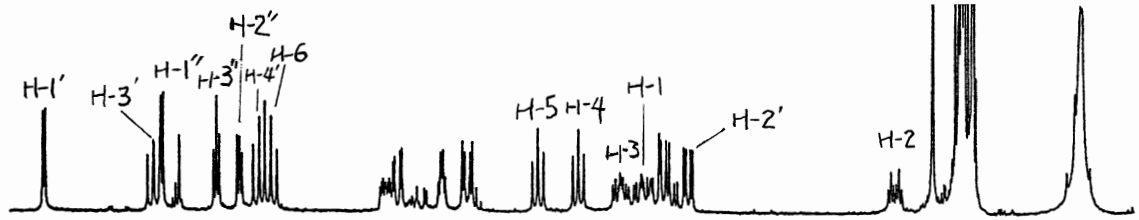
F2 - Processing parameters

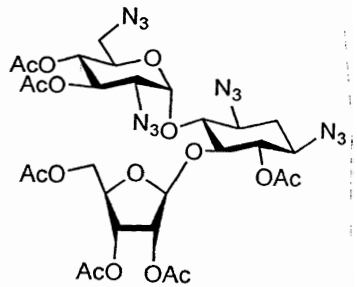
SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

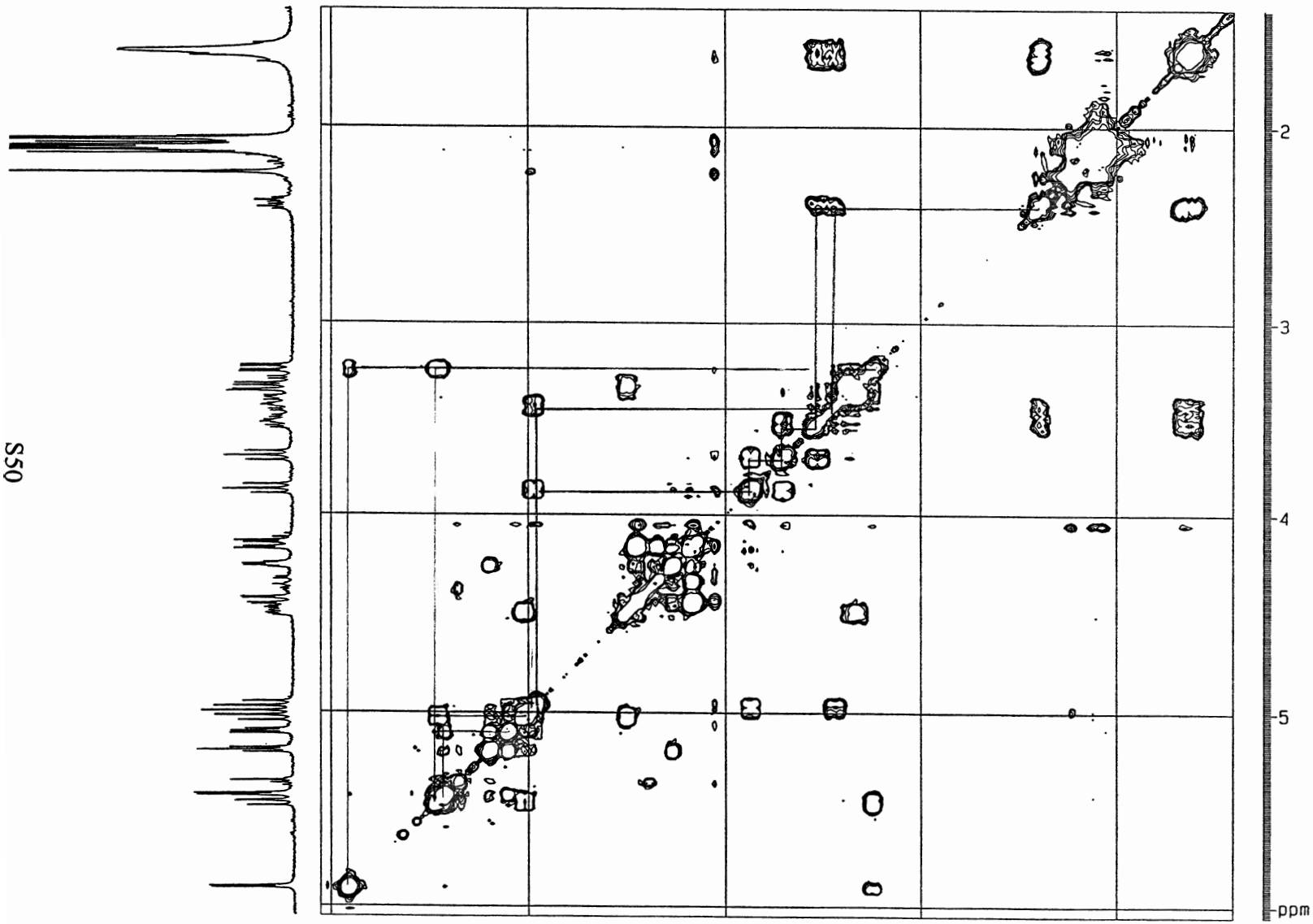
1D NMR plot parameters

CX	20.00 cm
F1P	205.733 ppm
F1	20699.37 Hz
F2P	-6.639 ppm
F2	-667.93 Hz
PPMCM	10.61859 ppm/cm
HZCM	1068.36536 Hz/cm

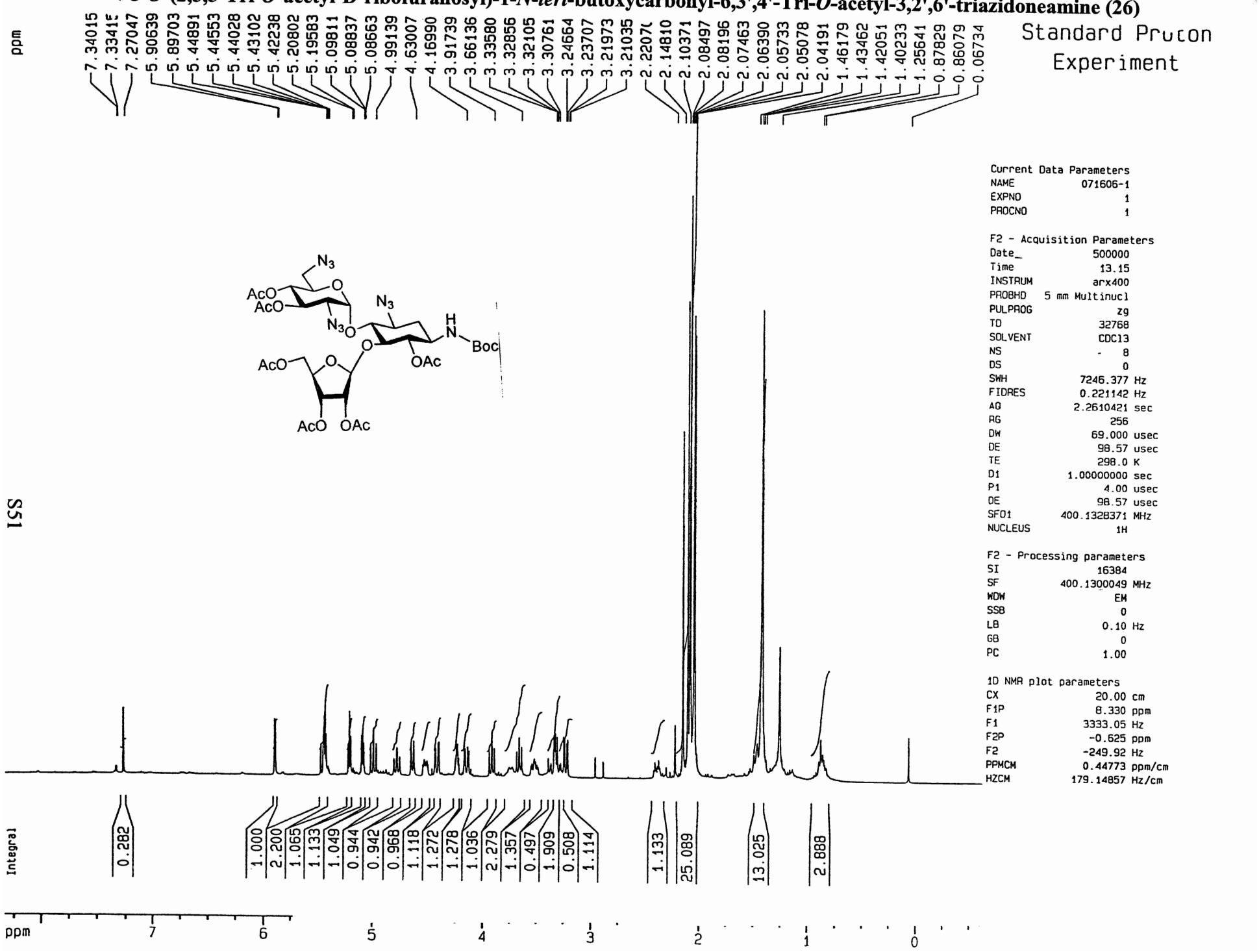
5-*O*-(2,3,5-Tri-*O*-acetyl-β-D-ribofuranosyl)-6,3',4'-Tri-*O*-acetyl-1,3,2',6'-tetraazidoneamine (25)





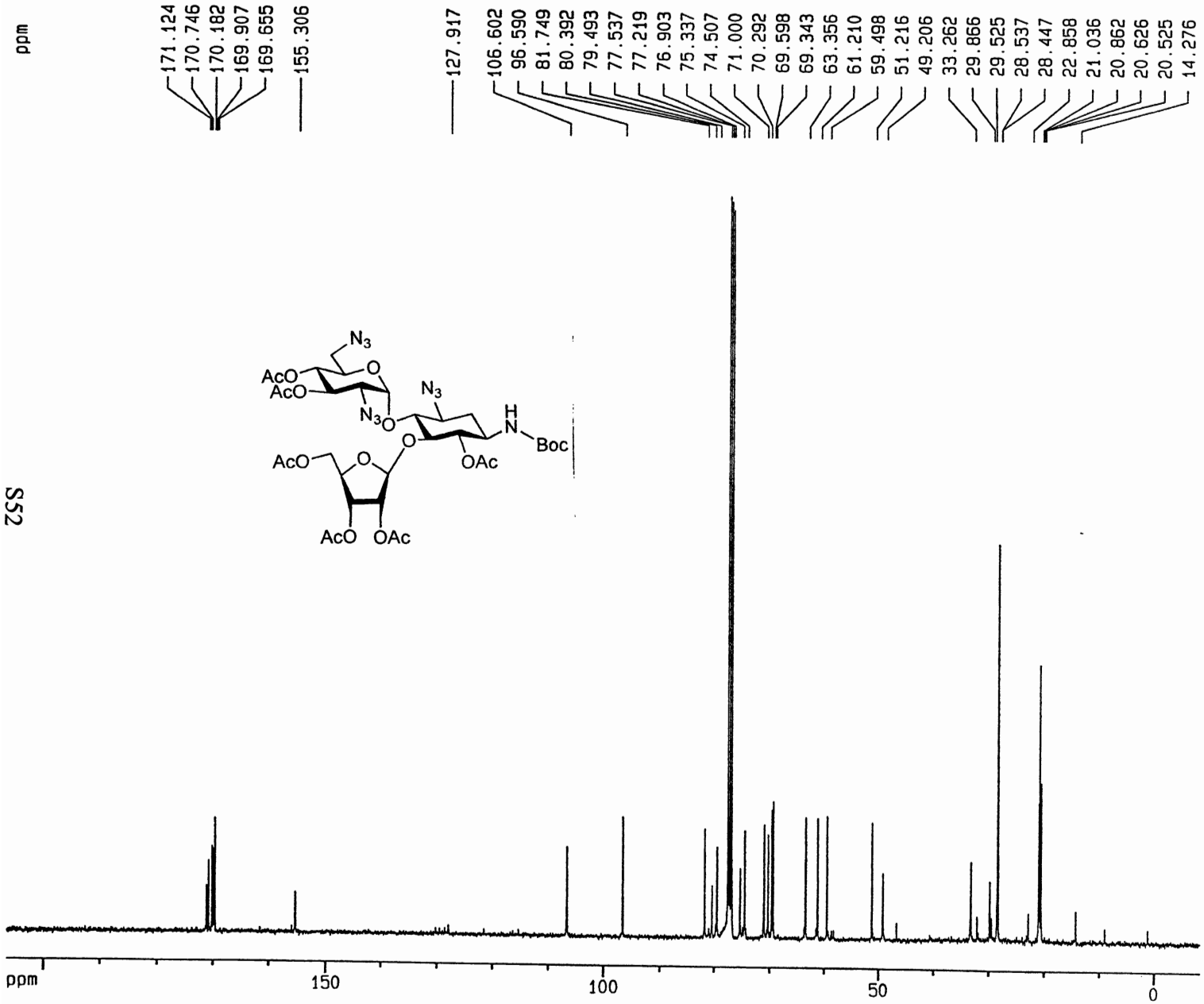


5-*O*-(2,3,5-Tri-*O*-acetyl-D-ribofuranosyl)-1-*N*-*tert*-butoxycarbonyl-6,3',4'-Tri-*O*-acetyl-3,2',6'-triazidoneamine (26)



Standard 13C
Experiment

5-*O*-(2,3,5-Tri-*O*-acetyl-D-ribofuranosyl)-1-*N*-*tert*-butoxycarbonyl-6,3',4'-Tri-*O*-acetyl-3,2',6'-triazidoneamine (26)



Current Data Parameters

NAME	071606-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	15.42
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	20000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

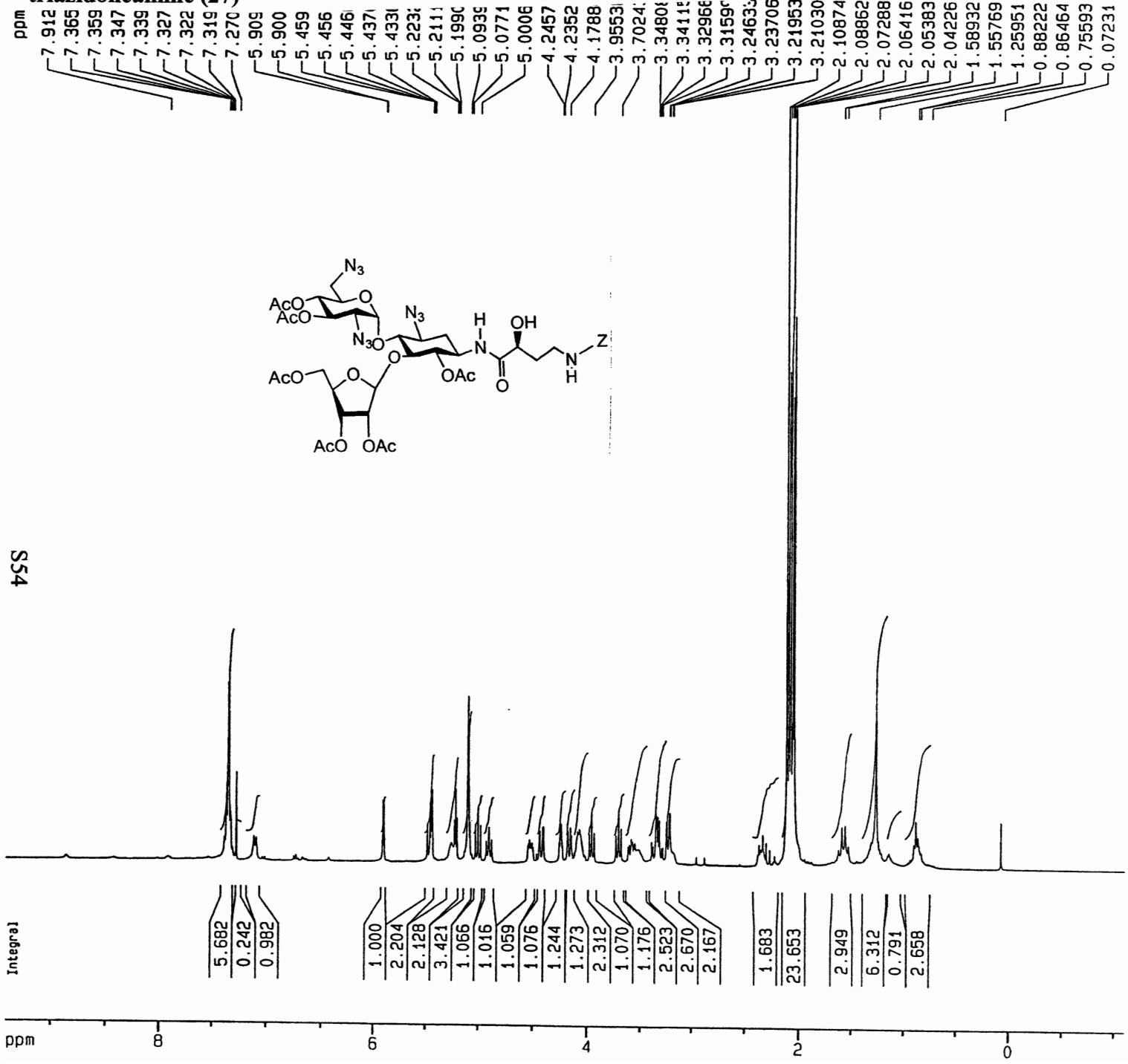
SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	206.485 ppm
F1	20775.05 Hz
F2P	-8.394 ppm
F2	-844.53 Hz
PPMCM	10.74396 ppm/cm
HZCM	1080.97888 Hz/cm

5-*O*-(2,3,5-Tri-*O*-acetyl-D-ribofuranosyl)-1-*N*-*tert*-butoxycarbonyl-6,3',4'-Tri-*O*-acetyl-3,2',6'-triazidoneamine (26)

5-*O*-(2,3,5-Tri-*O*-acetyl-β-D-ribofuranosyl)-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-hydroxybutanoyl]-6,3',4'-Tri-*O*-acetyl-3,2',6'-triazidoneamine (27)

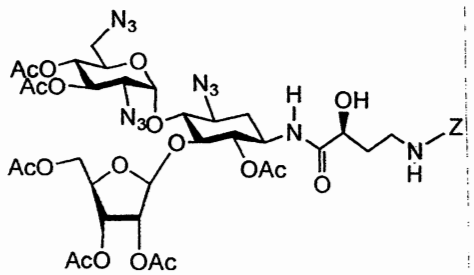


Standard 13C
Experiment

5-*O*-(2,3,5-Tri-*O*-acetyl-β-D-ribofuranosyl)-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-hydroxybutanoyl]-6,3',4'-Tri-*O*-acetyl-3,2',6'-triazidoneamine (27)

ppm

173.740 170.999 170.788 170.221 169.924 169.698 158.301 136.311 130.206 128.792 128.506 128.287 127.256 121.468 115.349 112.548 106.498 96.633 81.579 79.595 77.548 77.433 77.230 76.912 75.030 74.502 71.057 70.255 69.604 69.369 69.040 67.417 63.402 61.174 59.409 51.236 47.456 37.201 35.322 32.503 29.870 22.863 21.024 20.883 20.646 20.525



SSS

Current Data Parameters

NAME	080306-5
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	23.10
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	30000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SF01	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

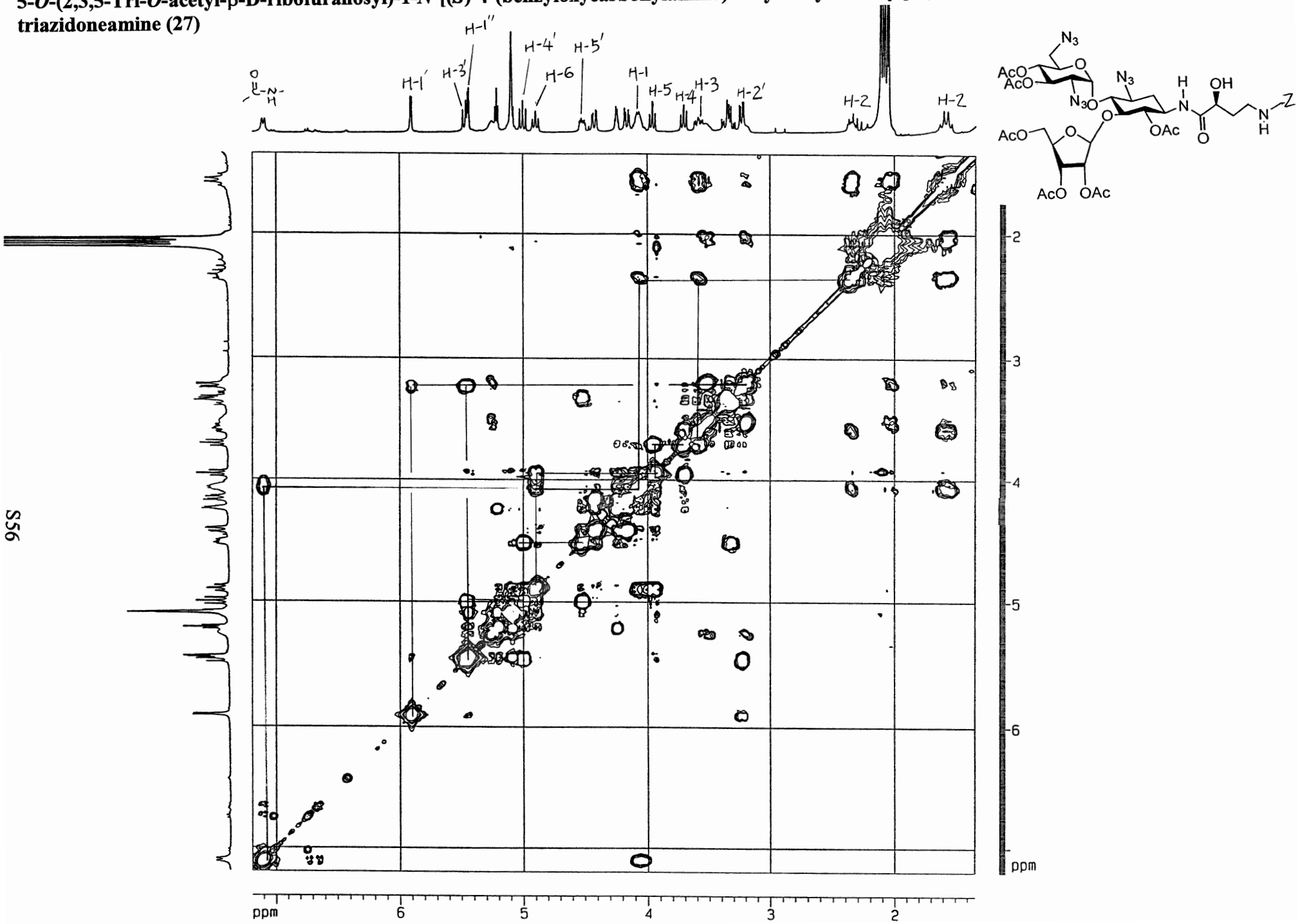
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

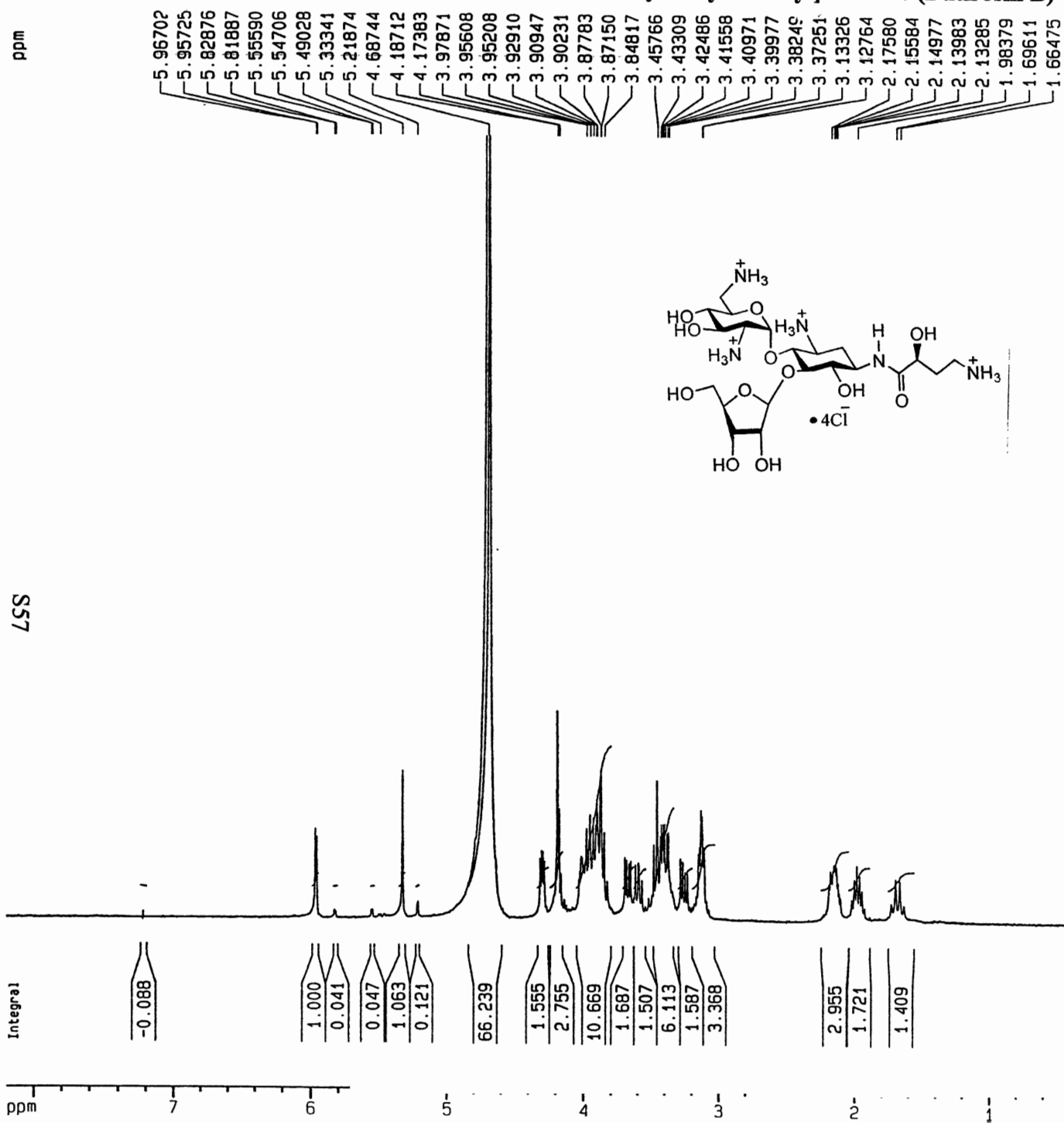
1D NMR plot parameters

CX	20.00 cm
F1P	209.243 ppm
F1	21052.55 Hz
F2P	-10.901 ppm
F2	-1096.80 Hz
PPMCM	11.00723 ppm/cm
HZCM	1107.46729 Hz/cm

5-*O*-(2,3,5-Tri-*O*-acetyl-β-D-ribofuranosyl)-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-hydroxybutanoyl]-6,3',4'-Tri-*O*-acetyl-3,2',6'-triazidoneamine (27)



5-*O*-(β-D-ribofuranosyl)-1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neamine (Butirosin B)



Standard Proton Experiment

SS7

Current Data Parameters

NAME	122005-1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	19.32
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	D2O
NS	8
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	512
DW	69.000 usec
DE	98.57 usec
TE	298.0 K
D1	1.00000000 sec
P1	4.00 usec
DE	98.57 usec
SFO1	400.1328371 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	400.1300049 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.198 ppm
F1	3280.26 Hz
F2P	-0.356 ppm
F2	-142.35 Hz
PPMCM	0.42769 ppm/cm
HZCM	171.13057 Hz/cm

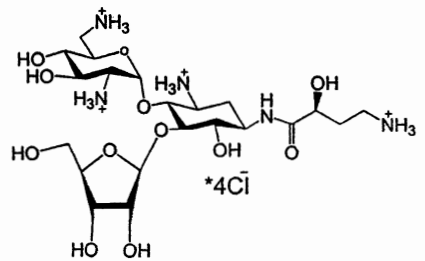
Standard 13C
Experiment

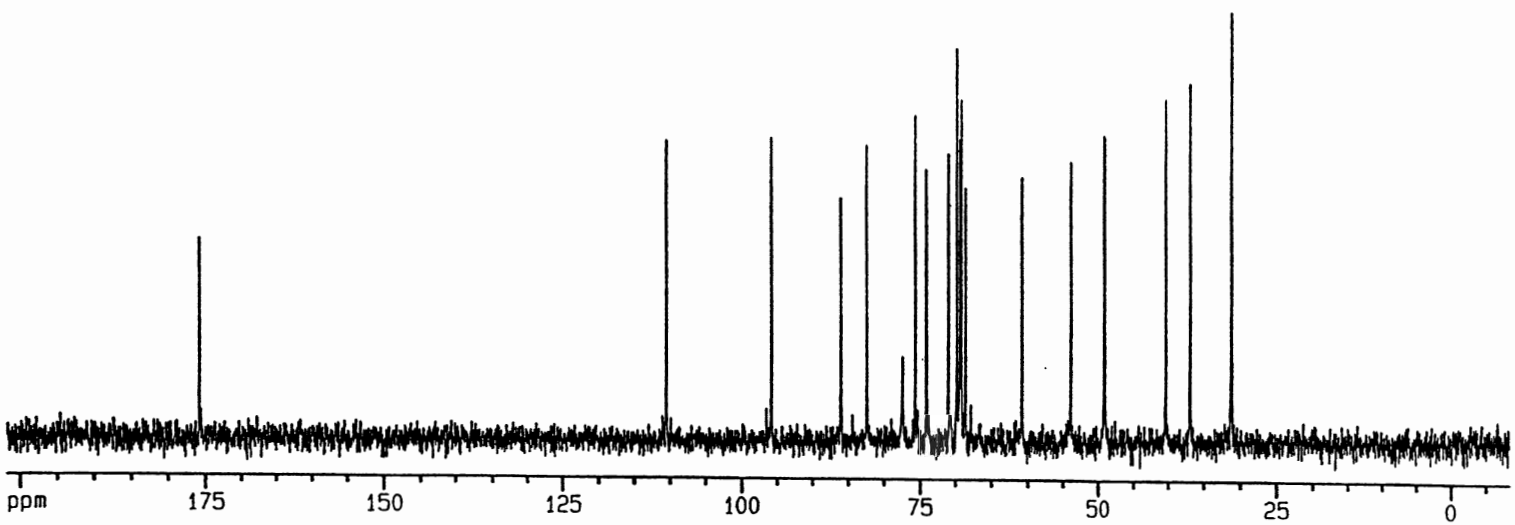
5-*O*-(β-D-ribofuranosyl)-1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neamine (Butirosin B)

ppm

175.821

110.537
95.824
86.036
82.482
77.445
75.670
74.165
70.970
69.818
69.388
69.193
68.606
60.869
53.944
49.230
49.131
40.404
36.888
31.147





Current Data Parameters

NAME	122005-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	23.57
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	D20
NS	32768
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

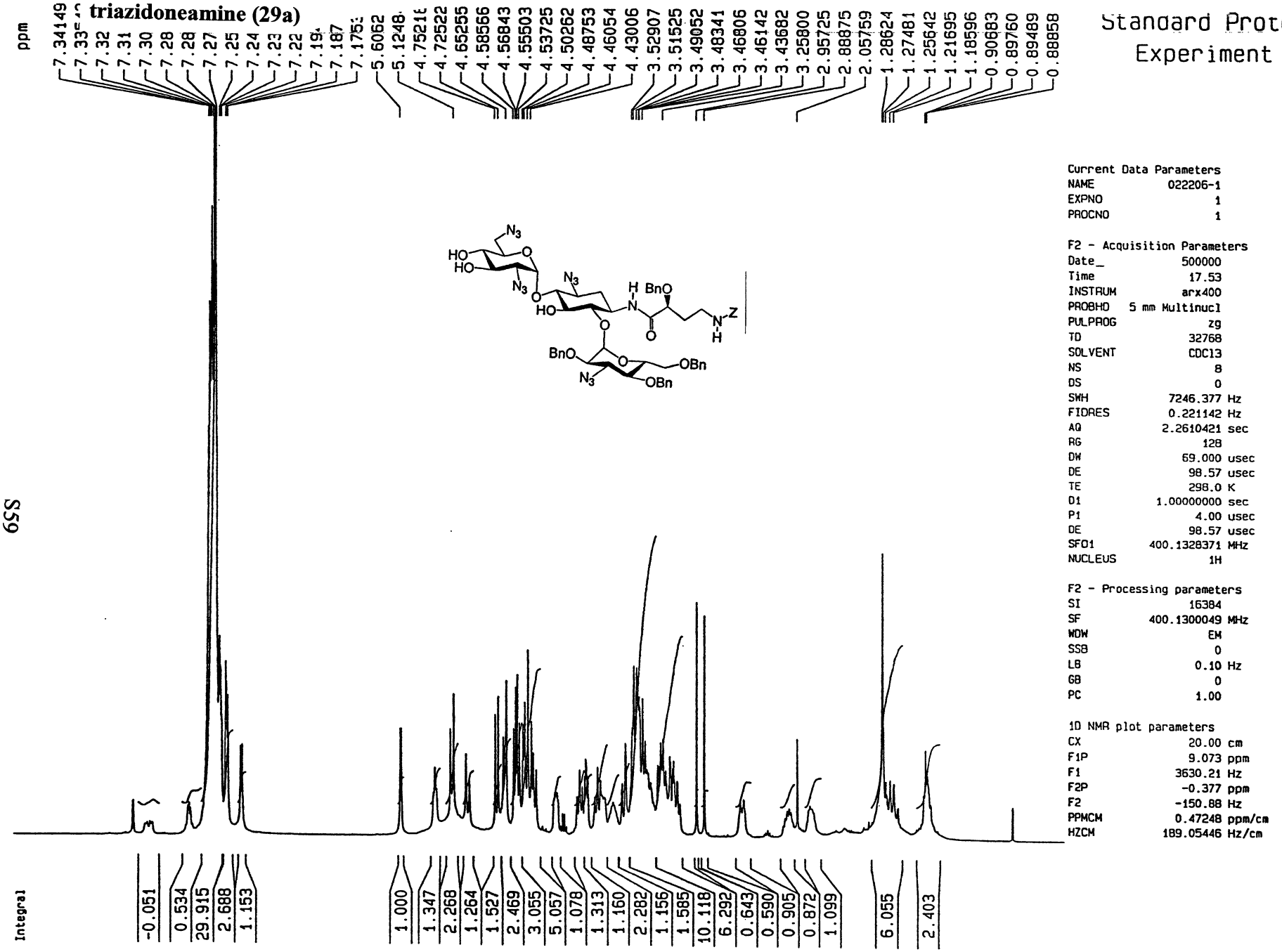
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

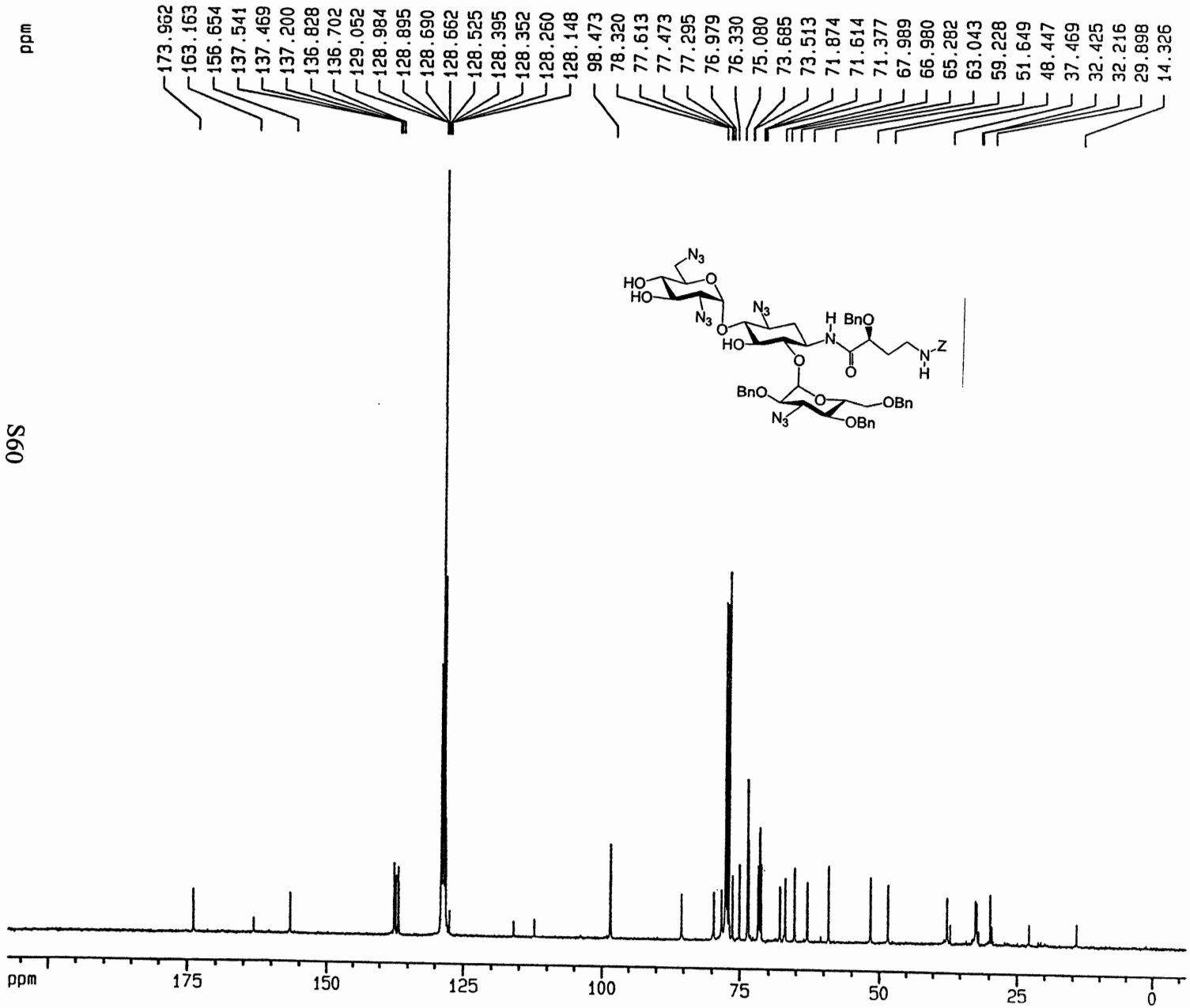
CX	20.00 cm
F1P	201.834 ppm
F1	20307.06 Hz
F2P	-8.304 ppm
F2	-835.46 Hz
PPMCM	10.50688 ppm/cm
HZCM	1057.12598 Hz/cm

6-*O*-(3-Azido-2,4,6-tri-*O*-benzyl-3-deoxy-α-D-glucopyranosyl)-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2'6'-triazidoneamine (29a)



Standard 13C
Experiment

6-*O*-(3-Azido-2,4,6-tri-*O*-benzyl-3-deoxy-α-D-glucopyranosyl)-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2'6'-triazidoneamine (29a)



S60

Current Data Parameters

NAME	022206-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	18.34
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	15220
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

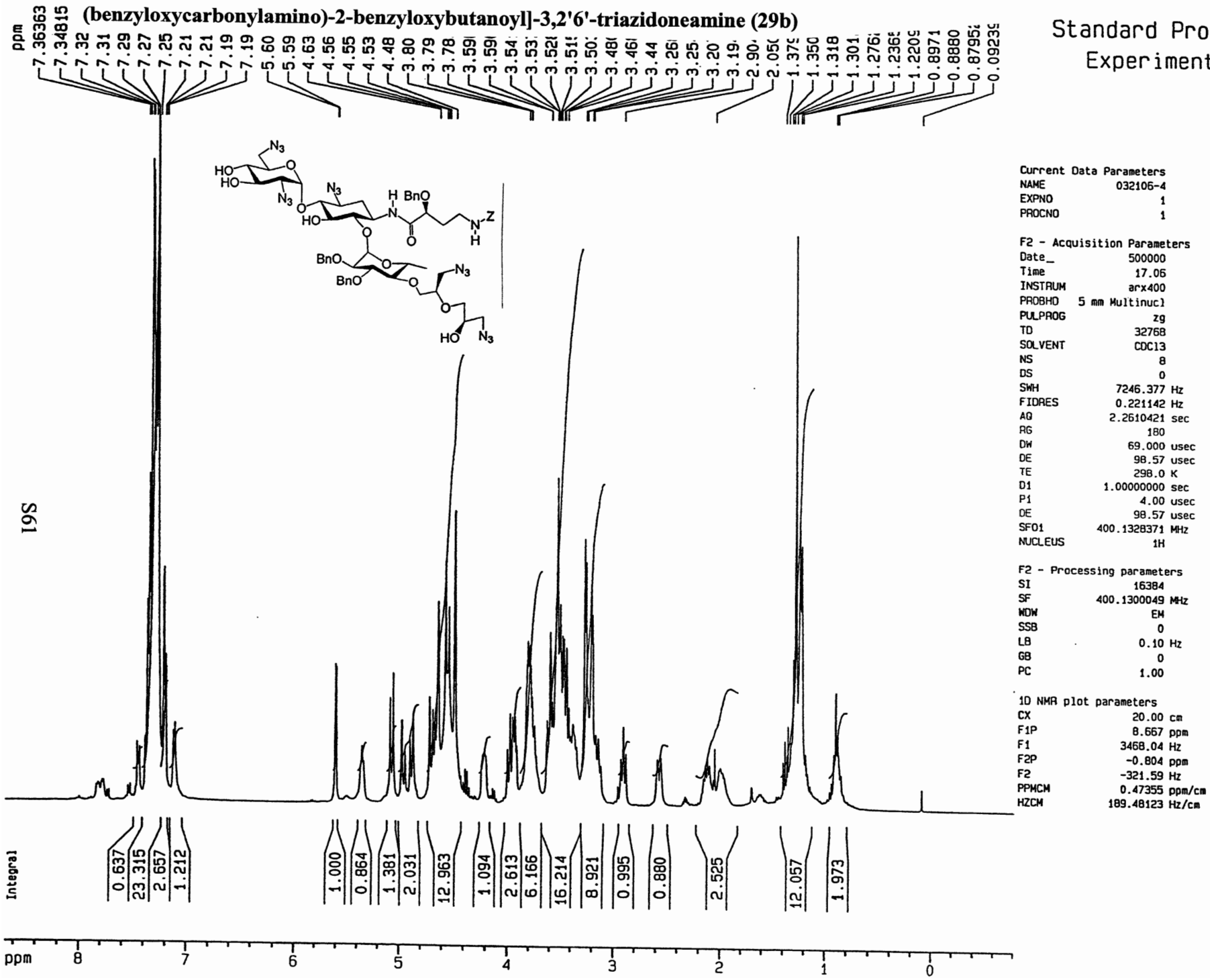
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

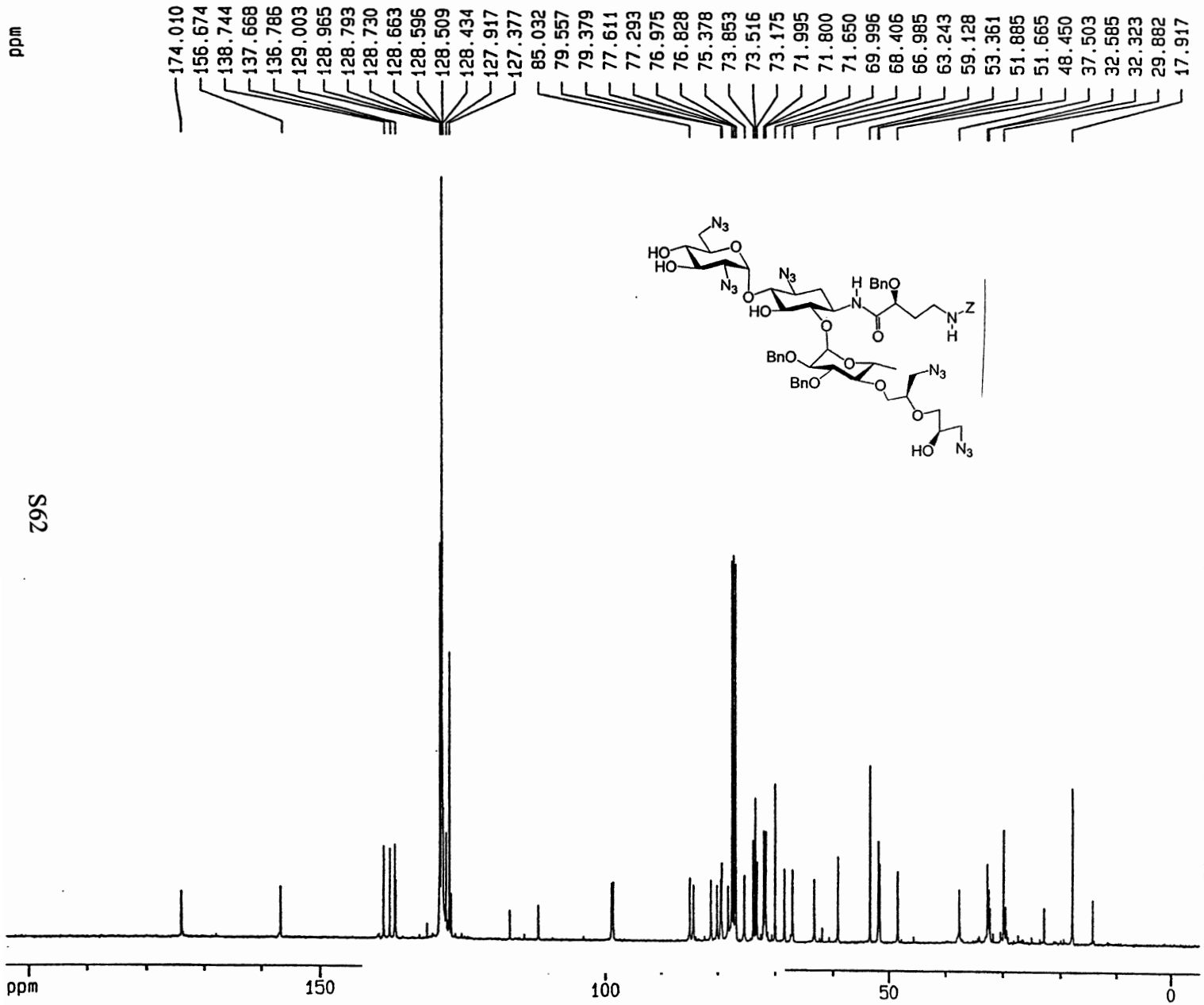
1D NMR plot parameters

CX	20.00 cm
F1P	207.395 ppm
F1	20866.54 Hz
F2P	-6.255 ppm
F2	-629.34 Hz
PPMCM	10.68248 ppm/cm
HZCM	1074.79382 Hz/cm

6-*O*-(4-*O*-((*R*)-3-Azido-2-((*R*)-3-azido-2-benzyloxypropoxy)propyl)-2,3-di-*O*-benzyl-6-deoxy-α-D-glucopyranosyl)-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2'6'-triazidoneamine (29b)



6-*O*-(4-*O*-((*R*)-3-Azido-2-((*R*)-3-azido-2-benzyloxypropoxy)propyl)-2,3-di-*O*-benzyl-6-deoxy-α-D-glucopyranosyl)-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2'6'-triazidoneamine (29b)



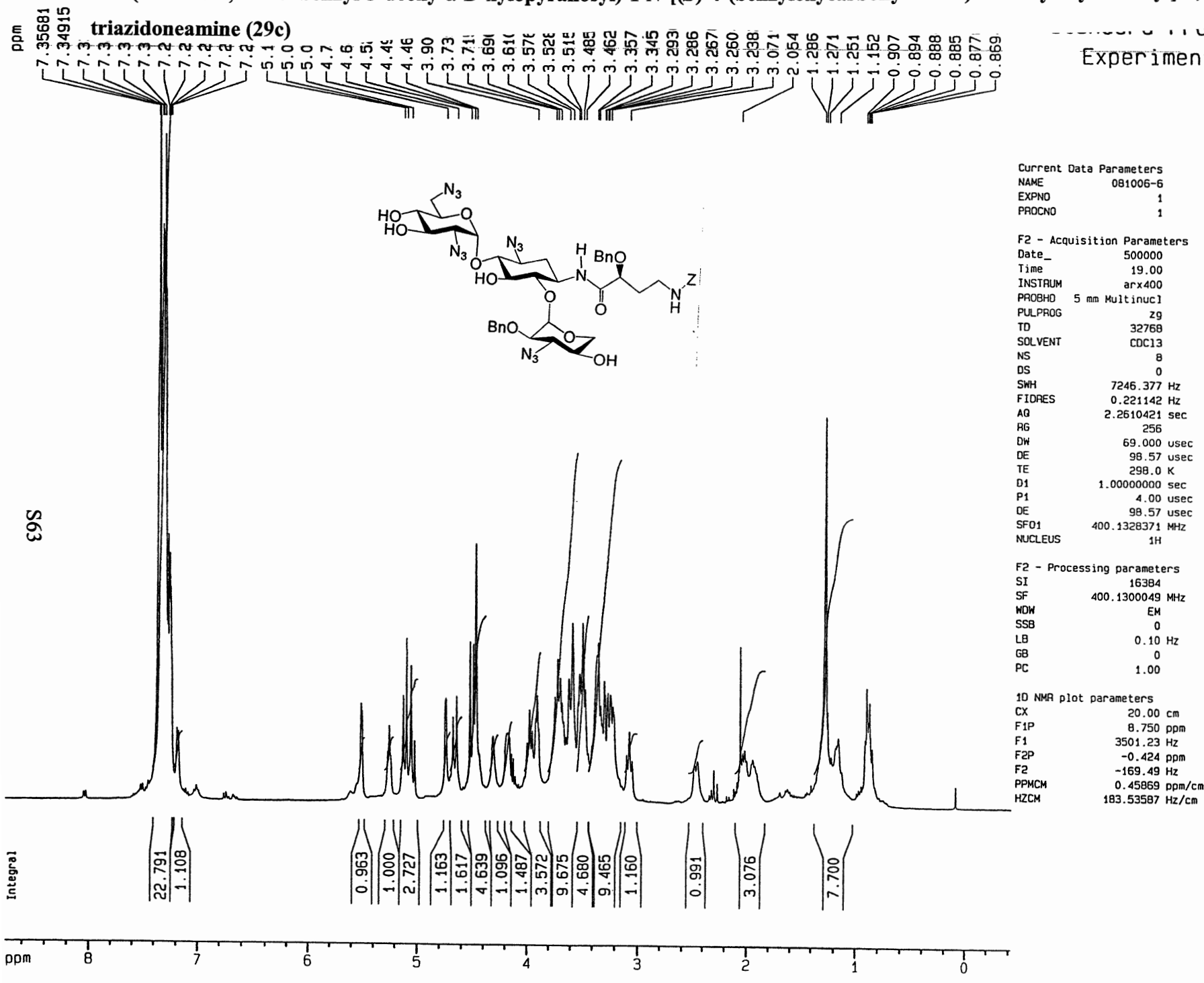

```
Current Data Parameters
NAME                032106-7
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_                 500000
Time                   19.26
INSTRUM               arx400
PROBHD   5 mm Multinucl
PULPROG               zgdc30
TD                     32768
SOLVENT                CDCl3
NS                     40000
DS                         2
SWH               25000.000 Hz
FIDRES            0.762939 Hz
AQ                0.6554100 sec
RG                     45500
DW                  20.000 usec
DE                  27.14 usec
TE                  300.0 K
D12               0.00002000 sec
DL5                   20.00 dB
CPDPRG               waltz16
P31                 100.00 usec
D1                0.40000001 sec
P1                    6.75 usec
DE                  27.14 usec
SFO1             100.6231179 MHz
NUCLEUS                 13C
D11               0.03000000 sec

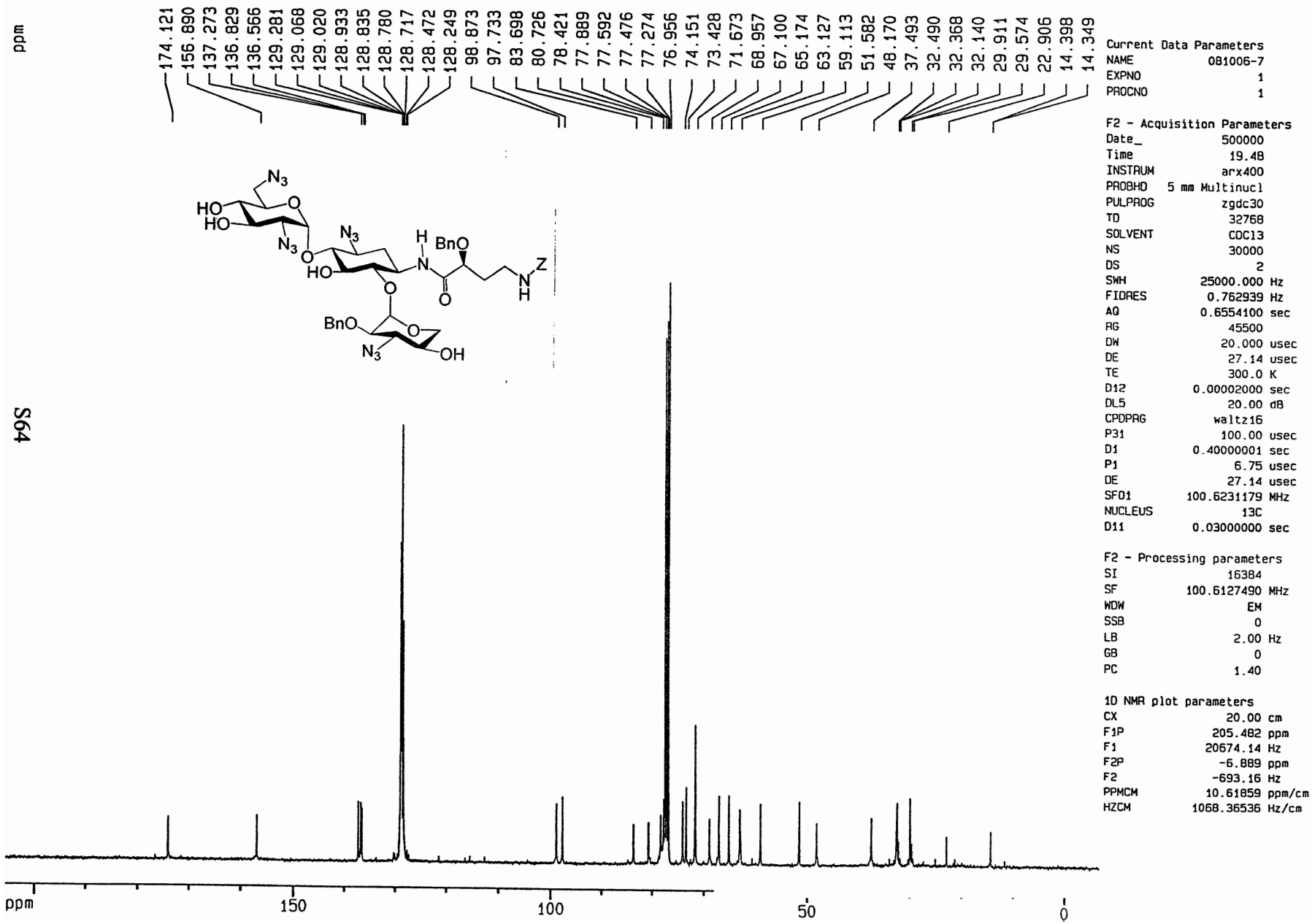
F2 - Processing parameters
SI                     16384
SF               100.6127490 MHz
WDW                       EM
SSB                        0
LB                    2.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                 203.883 ppm
F1                20513.18 Hz
F2P                 -5.084 ppm
F2                 -511.55 Hz
PPMCM              10.44835 ppm/cm
HZCM             1051.23669 Hz/cm
```

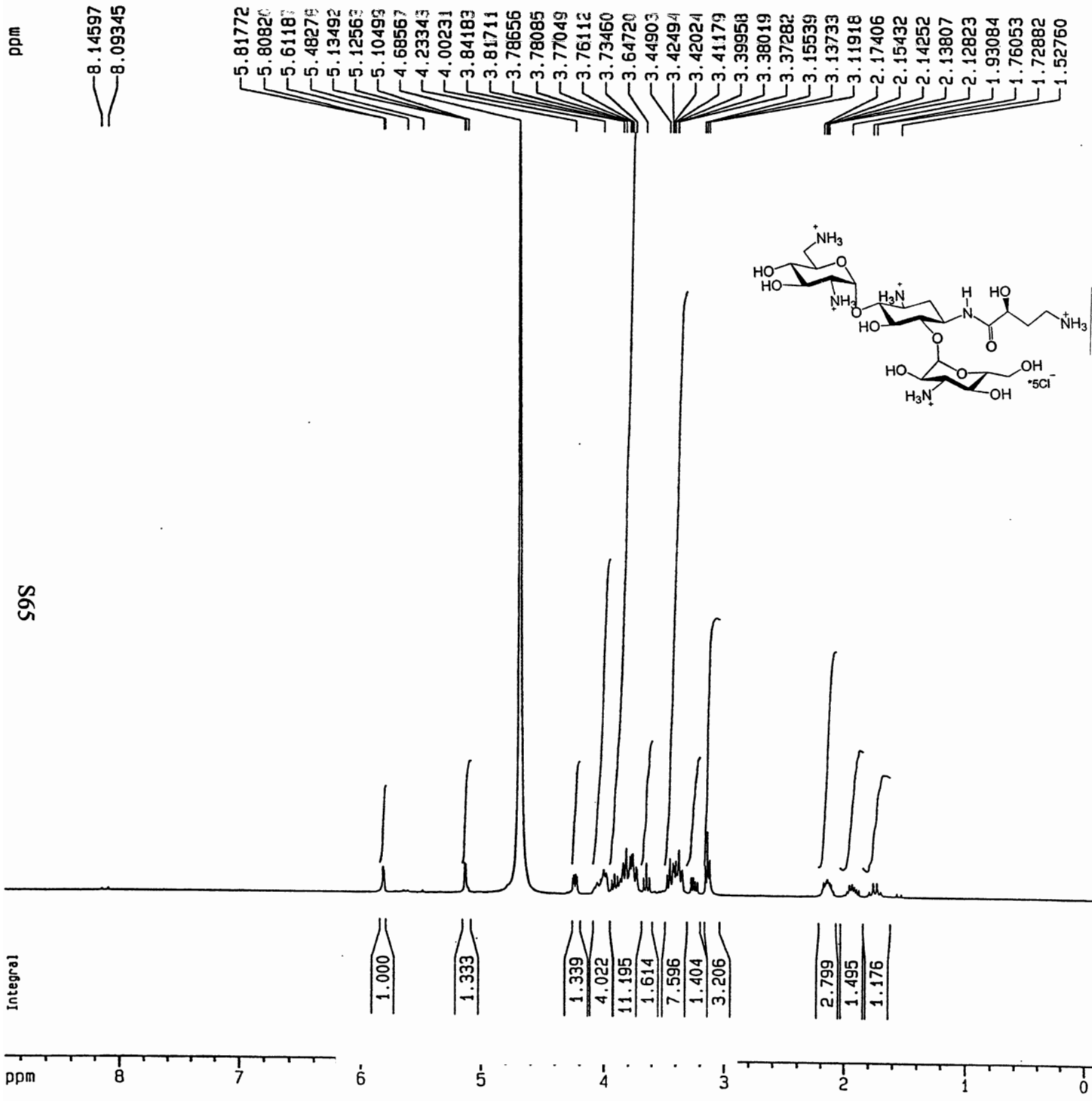
6-*O*-(3-Azido-2,4-di-*O*-benzyl-3-deoxy-α-D-xylopyranosyl)-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2'6'-triazidoneamine (29c)



6-*O*-(3-Azido-2,4-di-*O*-benzyl-3-deoxy-α-D-xylopyranosyl)-1-*N*-[(*S*)-4-(benzyloxycarbonylamino)-2-benzyloxybutanoyl]-3,2'6'-triazidoneamine (29c)



6-*O*-(3-Amino-3-deoxy-β-D-glucopyranosyl)-1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neamine (JLN007)

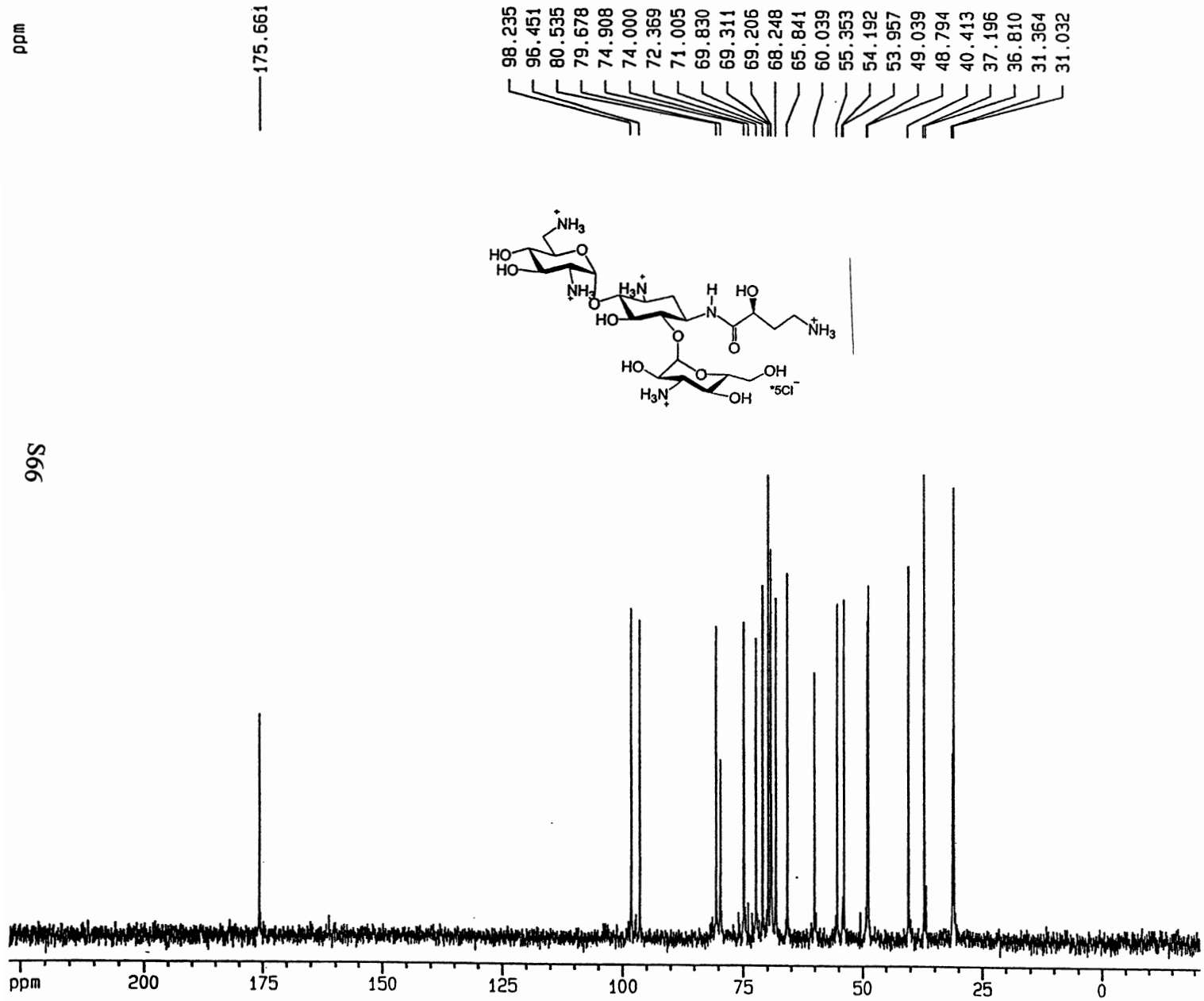


Standard Proton Experiment

Current Data Parameters	
NAME	030406-2
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	500000
Time	18.22
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	D2O
NS	8
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	256
DW	69.000 usec
DE	98.57 usec
TE	298.0 K
D1	1.00000000 sec
P1	4.00 usec
DE	98.57 usec
SFO1	400.1328371 MHz
NUCLEUS	1H
F2 - Processing parameters	
SI	16384
SF	400.1300049 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00
1D NMR plot parameters	
CX	20.00 cm
F1P	8.923 ppm
F1	3570.46 Hz
F2P	-0.420 ppm
F2	-167.96 Hz
PPMCM	0.46715 ppm/cm
HZCM	186.92070 Hz/cm

6-*O*-(3-Amino-3-deoxy-β-D-glucopyranosyl)-1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neamine (JLN007)

Standard 13C Experiment



Current Data Parameters

NAME	030406-3
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	21.36
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	D2O
NS	40000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

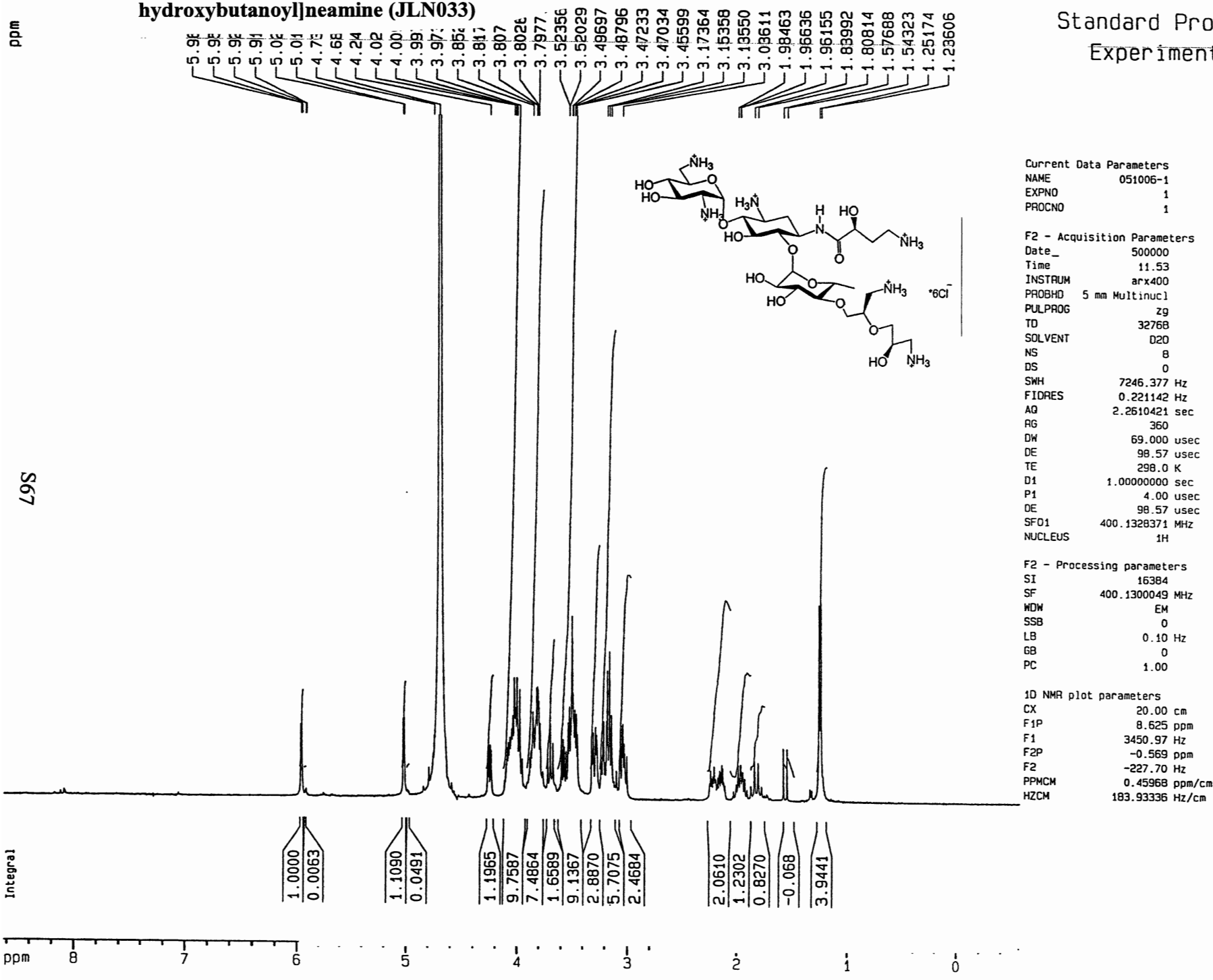
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	227.296 ppm
F1	22868.89 Hz
F2P	-21.181 ppm
F2	-2131.10 Hz
PPMCM	12.42387 ppm/cm
HZCM	1249.99988 Hz/cm

6-*O*-(4-*O*-((*R*)-3-Amino-2-((*R*)-3-Amino-2-hydroxypropoxy)propyl)-6-deoxy-α-D-glucopyranosyl)-1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neamine (JLN033)



Current Data Parameters

NAME	051006-1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	11.53
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zg
TD	32768
SOLVENT	D2O
NS	8
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	360
DW	69.000 usec
DE	98.57 usec
TE	298.0 K
D1	1.00000000 sec
P1	4.00 usec
DE	98.57 usec
SFO1	400.1328371 MHz
NUCLEUS	1H

F2 - Processing parameters

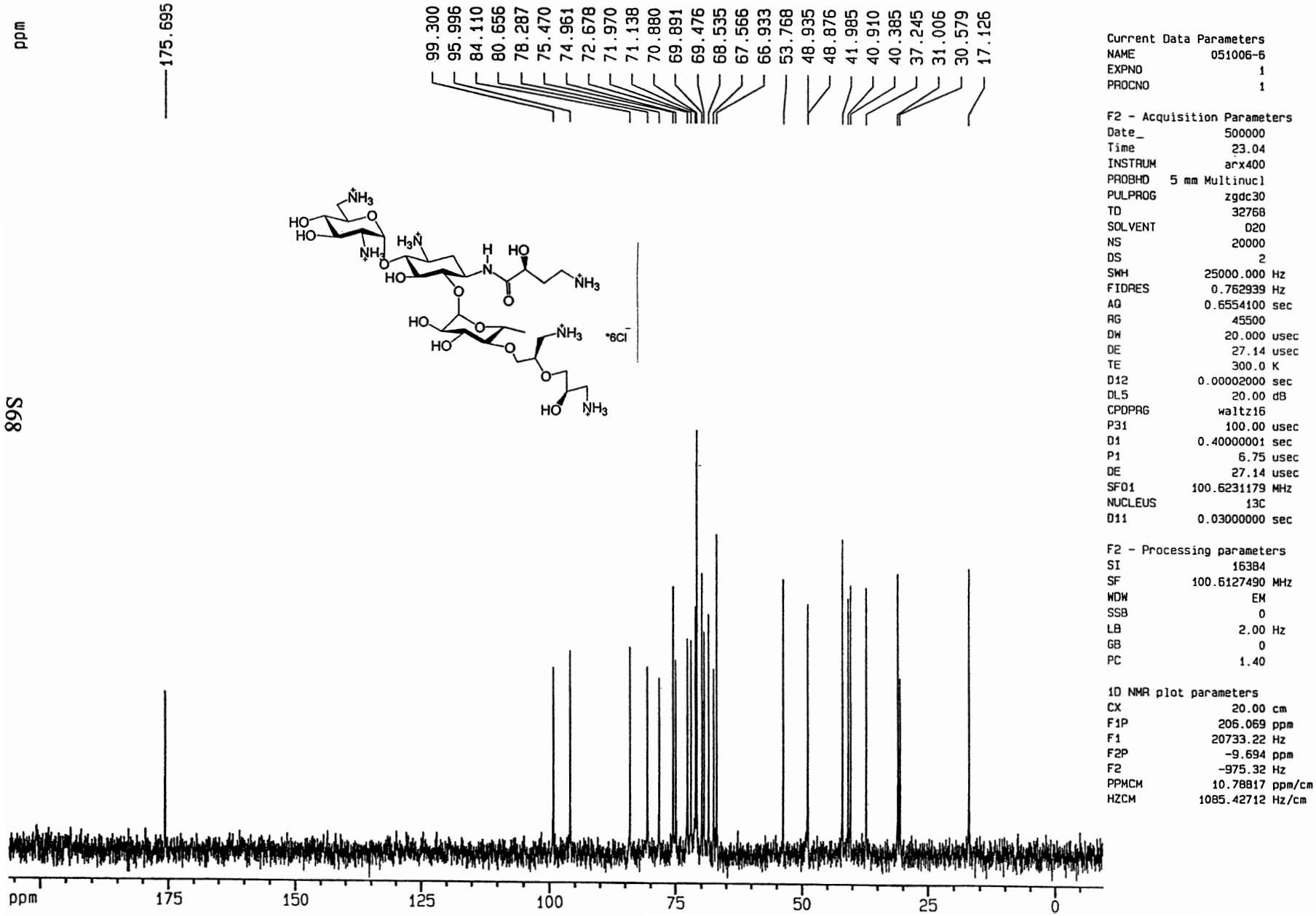
SI	16384
SF	400.1300049 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.625 ppm
F1	3450.97 Hz
F2P	-0.569 ppm
F2	-227.70 Hz
PPMCM	0.45968 ppm/cm
HZCM	183.93336 Hz/cm

6-*O*-(4-*O*-((*R*)-3-Amino-2-((*R*)-3-Amino-2-hydroxypropoxy)propyl)-6-deoxy-α-D-glucopyranosyl)-1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neamine (JLN033)

Standard 13C Experiment



Current Data Parameters

NAME	051006-6
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	23.04
INSTRUM	arx400
PROBHD	5 mm Multinucl
PULPROG	zgdc30
TD	32768
SOLVENT	D20
NS	20000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	45500
DW	20.000 usec
DE	27.14 usec
TE	300.0 K
D12	0.00002000 sec
DL5	20.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	0.40000001 sec
P1	6.75 usec
DE	27.14 usec
SFO1	100.6231179 MHz
NUCLEUS	13C
D11	0.03000000 sec

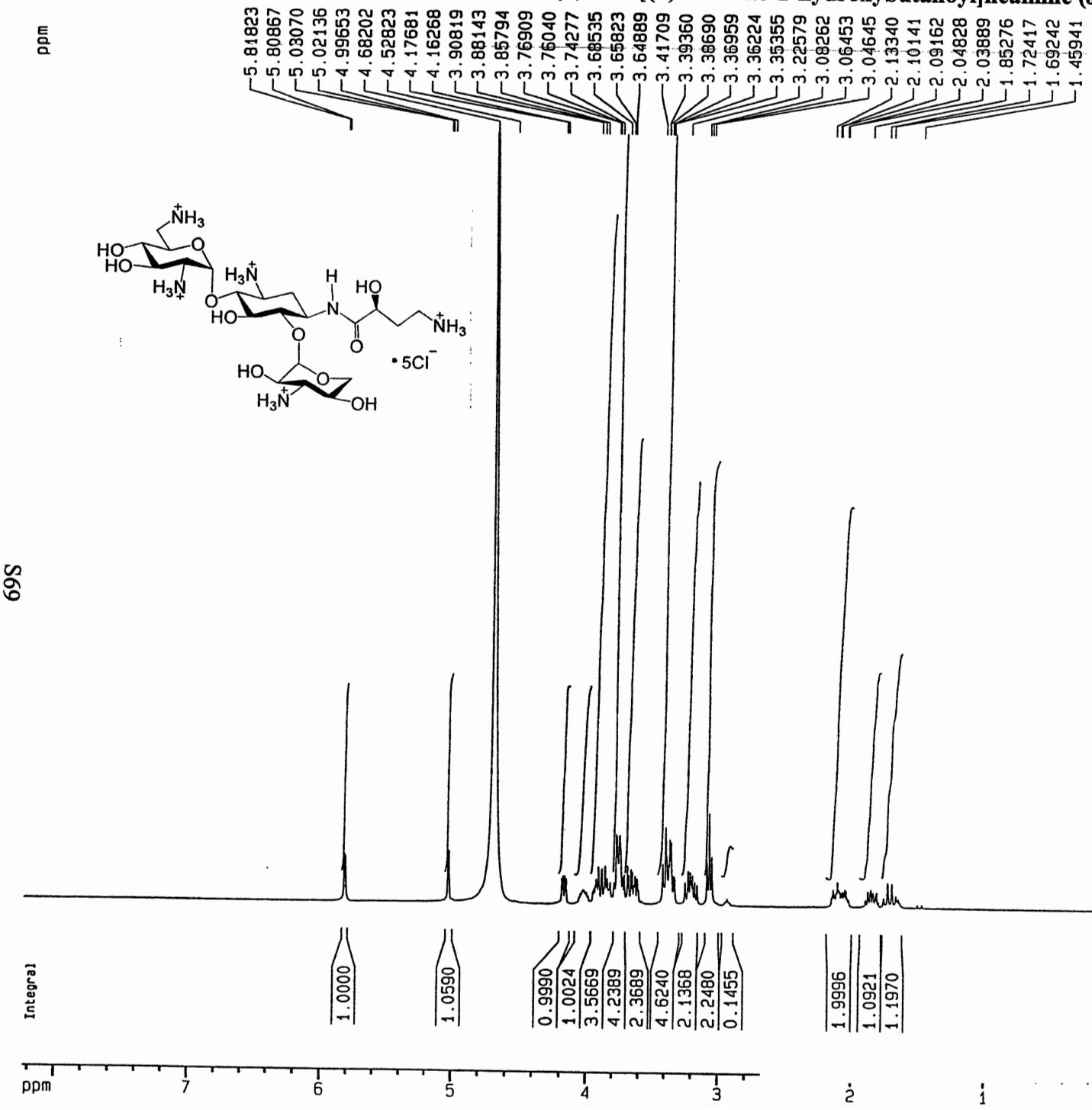
F2 - Processing parameters

SI	16384
SF	100.6127490 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

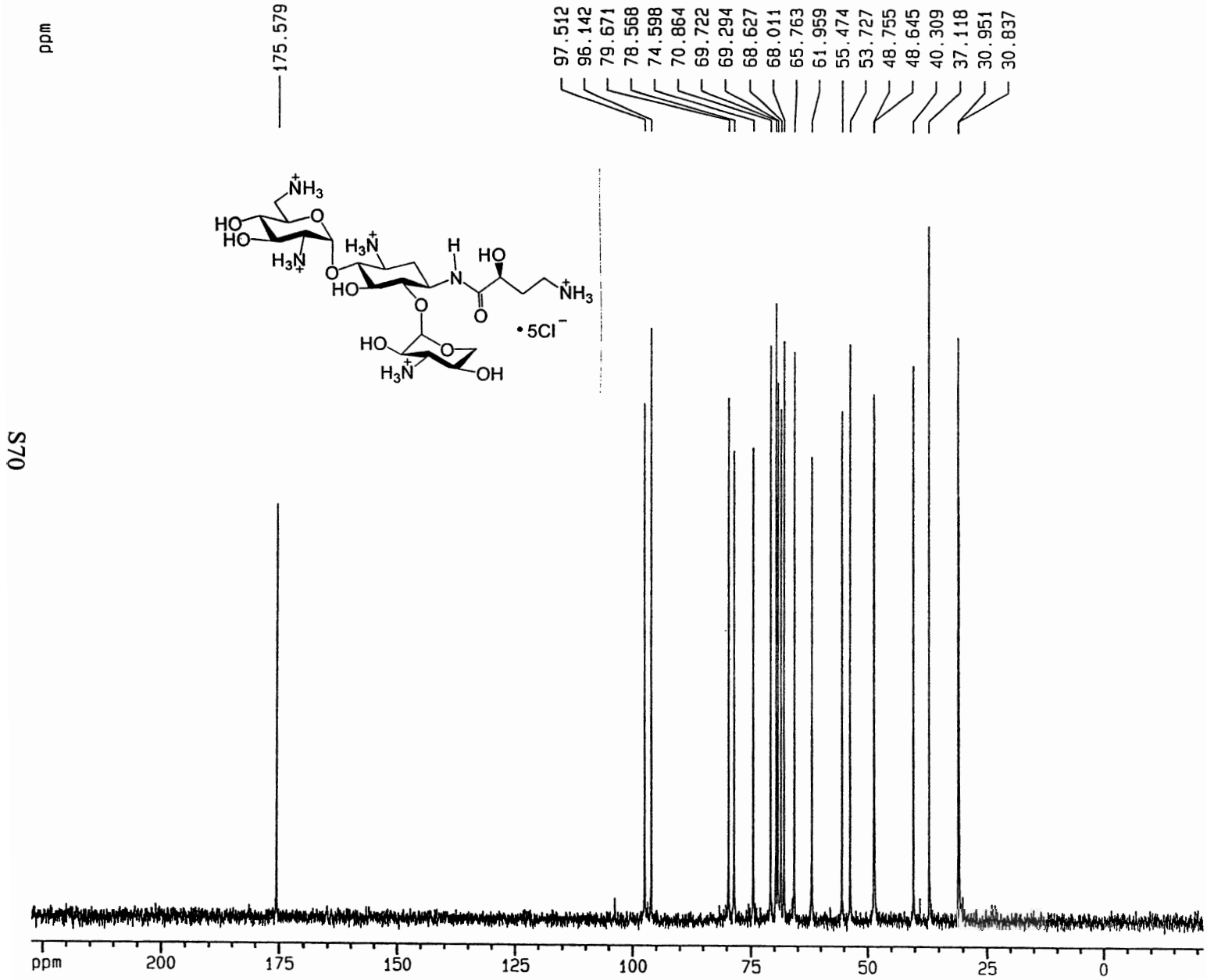
CX	20.00 cm
F1P	205.069 ppm
F1	20733.22 Hz
F2P	-9.694 ppm
F2	-975.32 Hz
PPMCM	10.78817 ppm/cm
HZCM	1085.42712 Hz/cm

6-*O*-(3-Amino-3-deoxy-α-D-xylopyranosyl)-1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neamine (JLN040)



6-*O*-(3-Amino-3-deoxy-α-D-xylopyranosyl)-1-*N*-[(*S*)-4-amino-2-hydroxybutanoyl]neamine (JLN040)

Standard 13C
Experiment



S70

Current Data Parameters
NAME 092306-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 22.54
INSTRUM arx400
PROBHD 5 mm Multinucl
PULPROG zgdc30
TD 32768
SOLVENT D20
NS 30000
DS 2
SWH 25000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554100 sec
RG 45500
DW 20.000 usec
DE 27.14 usec
TE 300.0 K
D12 0.00002000 sec
DL5 20.00 dB
CPDPRG waltz16
P31 100.00 usec
D1 0.40000001 sec
P1 6.75 usec
DE 27.14 usec
SFO1 100.6231179 MHz
NUCLEUS 13C
D11 0.03000000 sec

F2 - Processing parameters
SI 16384
SF 100.6127490 MHz
WDW EM
SSB 0
LB. 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 227.296 ppm
F1 22868.89 Hz
F2P -21.181 ppm
F2 -2131.11 Hz
PPMCM 12.42387 ppm/cm
HZCM 1250.00012 Hz/cm