

Short Syntheses of Gabosine I and G from δ -D-Gluconolactone

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Appendices

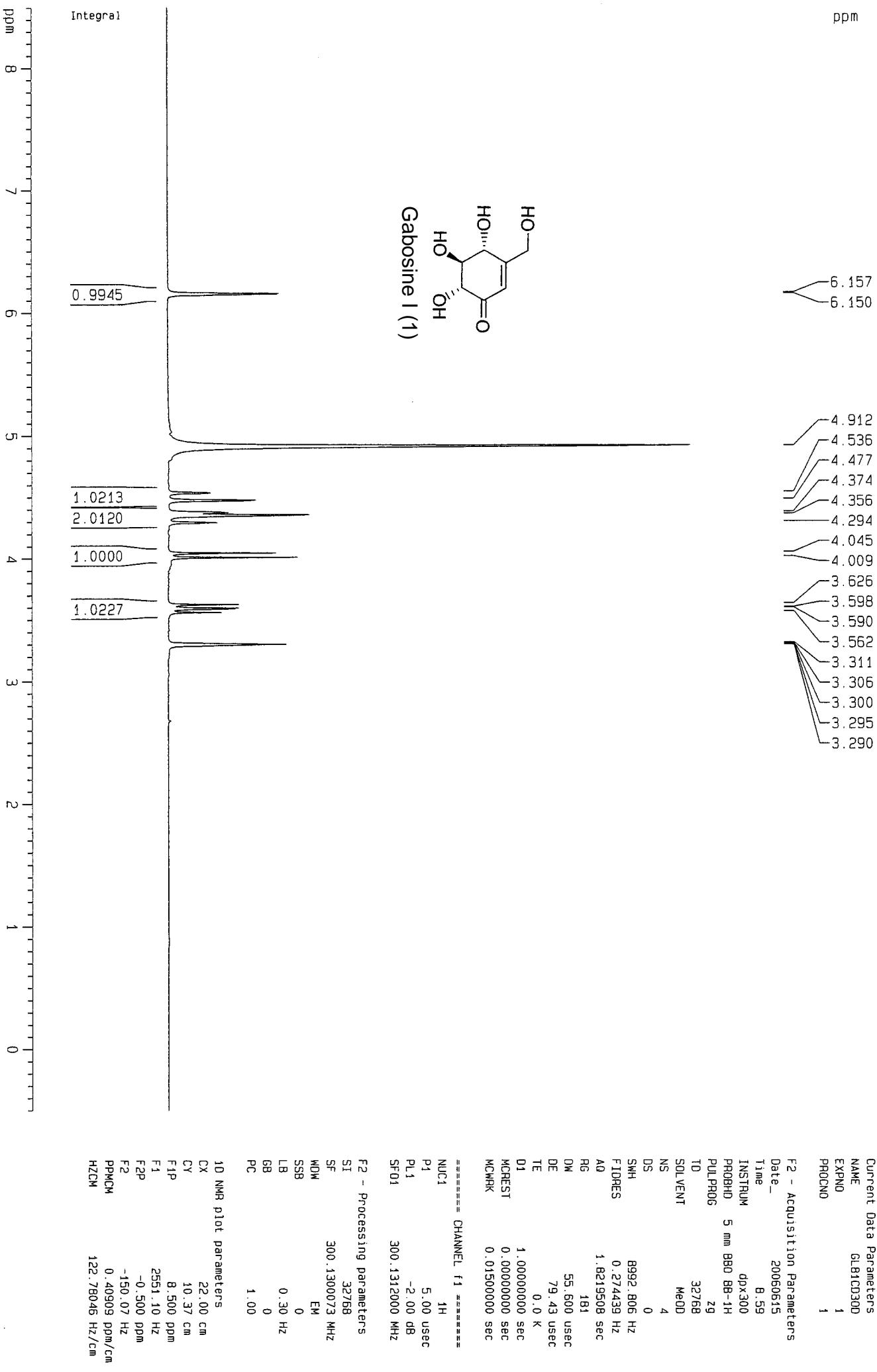
S1 List of contents

S2 General procedures

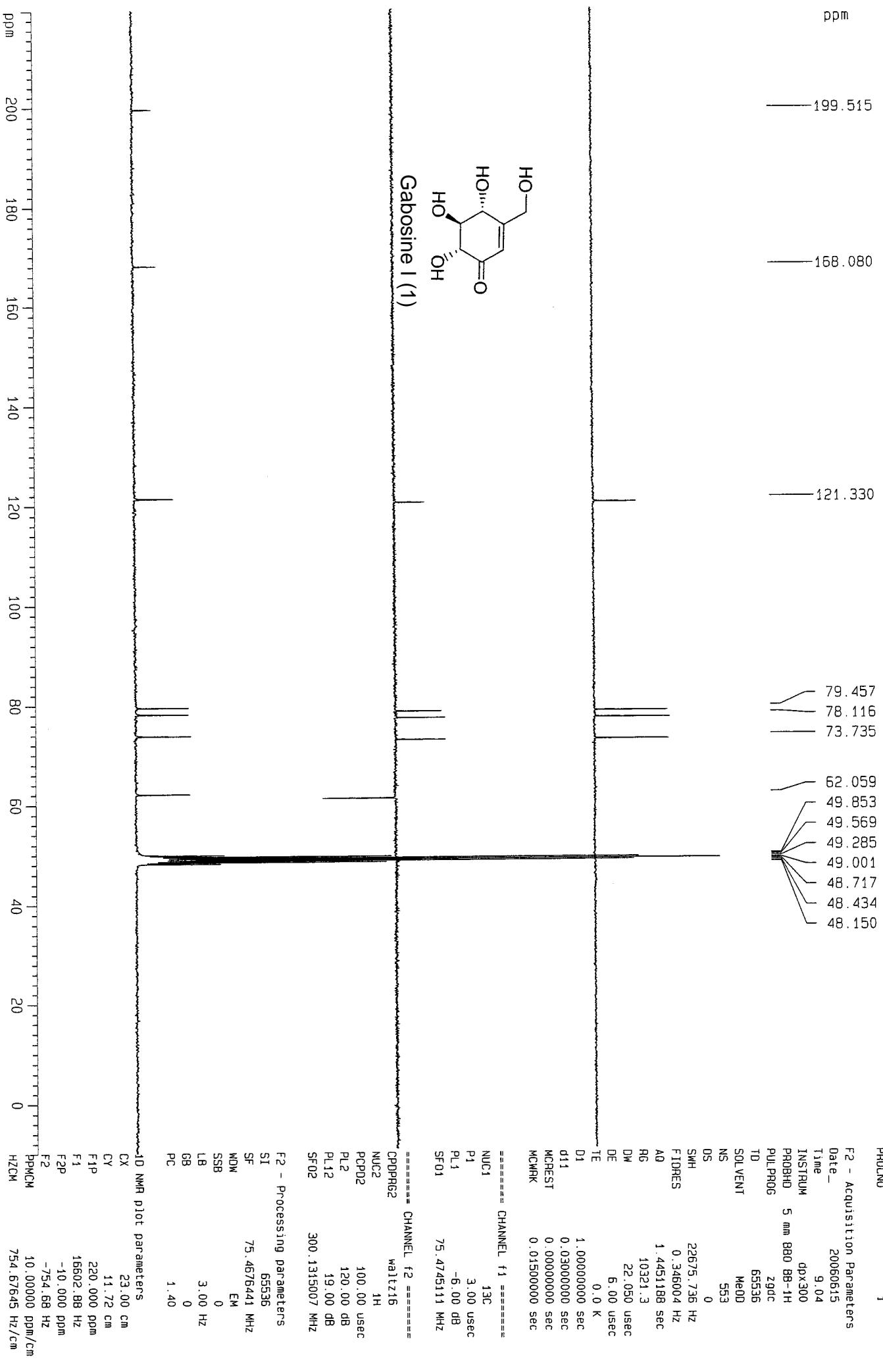
S3-S14 ^1H , ^{13}C and DEPT NMR Spectra of compound **1**, **2**, **4**, **5** and **7**

Melting points were measured with in Celsius degrees and were uncorrected. Optical rotations were operating at 589nm. Infrared spectra (IR) were recorded as thin film on potassium bromide discs. Nuclear magnetic resonance (NMR) spectra were measured at 300.13 MHz (^1H) or at 75.47 MHz (^{13}C). All chemical shifts were recorded in ppm relative to tetramethylsilane ($\delta = 0.0$). Spin-spin coupling constants (J value) recorded in Hz were measured directly from the spectra. All reactions were monitored by analytical thin-layer chromatography (TLC) on aluminium-precoated plates of silica gel with detection by spraying with 5% (w/v) dodecamolybdophosphoric acid in ethanol. Silica gel 60 (2306400 mesh) was used for flash chromatography. All reagents and solvents were general reagent grade unless otherwise stated. DMF was dried by magnesium sulfate and filtered. It was then freshly distilled under reduced pressure. Acetonitrile was freshly distilled from P_2O_5 under nitrogen. THF was freshly distilled from Na/benzophenone ketyl under nitrogen. Dichloromethane was freshly distilled from P_2O_5 under nitrogen. Other reagents were purchased from commercial suppliers and were used without purification.

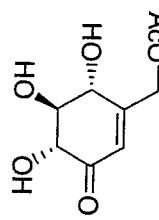
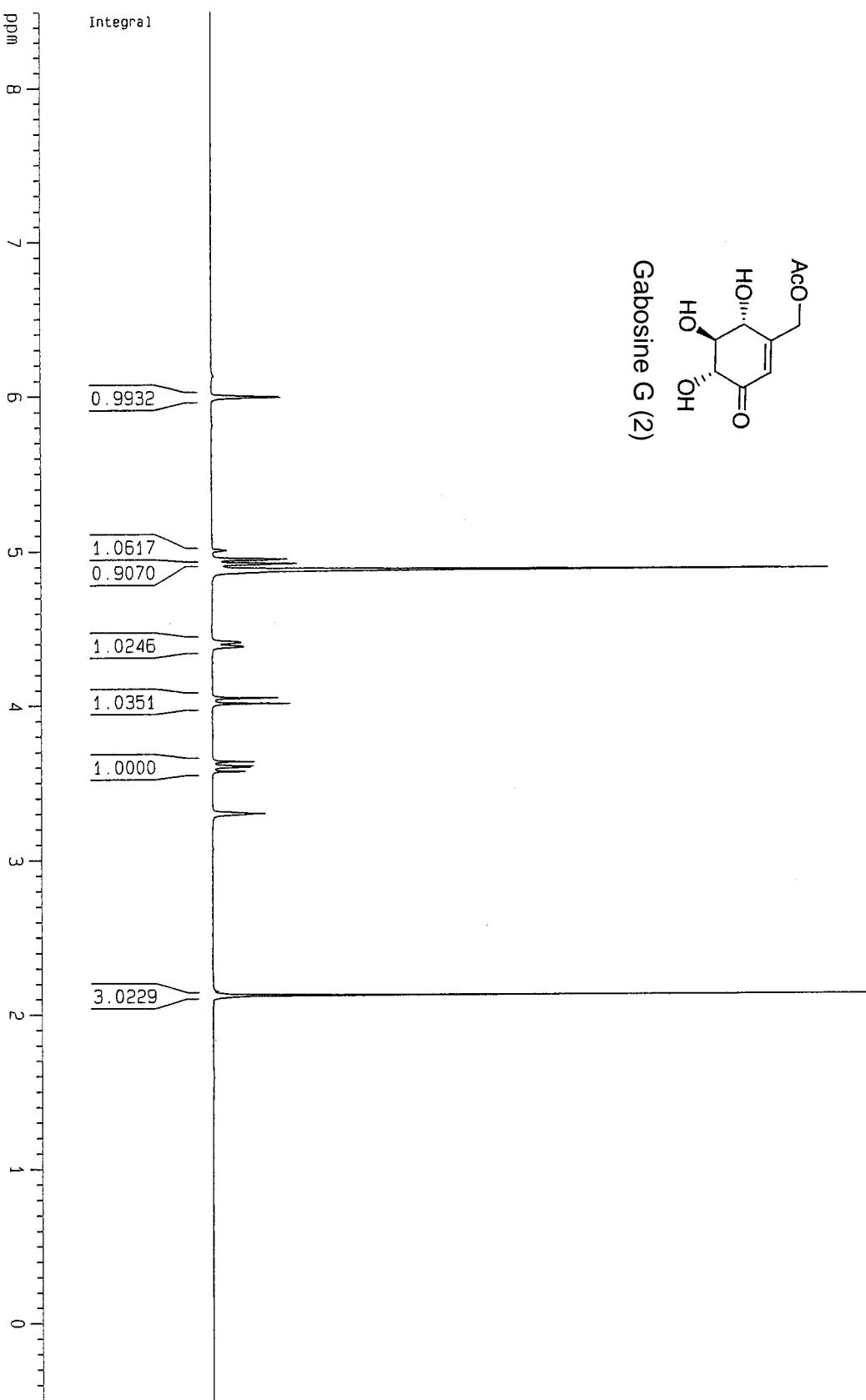
Solvent: CD3OD



Solvent: CD3OD



Solvent: CD3OD



Gabosine G (2)

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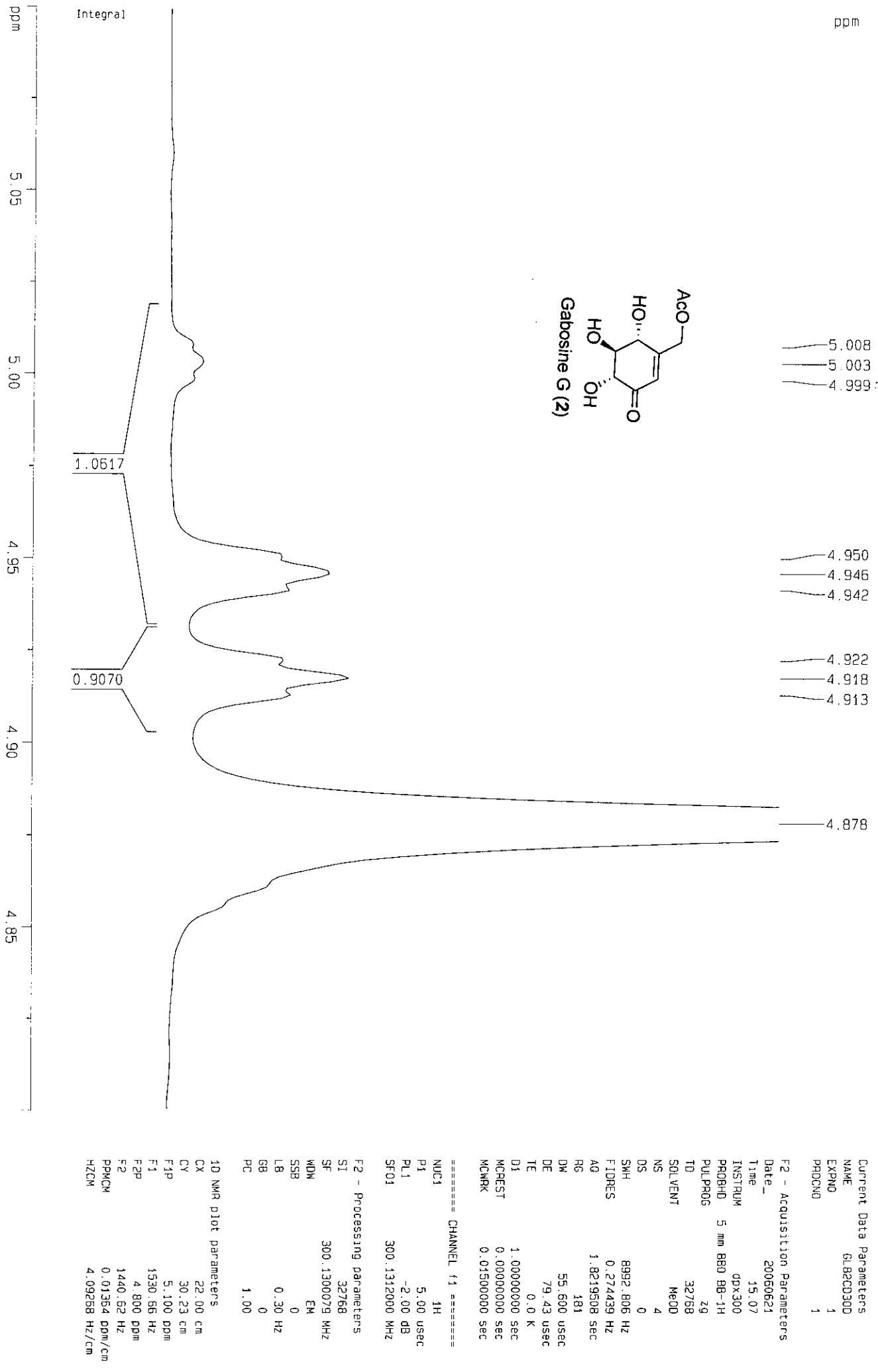
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 CHANNEL f1 =====
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PROCNO 1

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INSTRUM op3300
PROBHD 5 mm BBO BB-1H
PULPROG 29
TD 32768
SOLEX 4
NS 0
DS 0
SWH 8992.806 Hz
FIDRES 0.27439 Hz
AQ 1.8219500 sec
RG 181
DW 55.500 usec
DE 79.43 usec
TE 0.0 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

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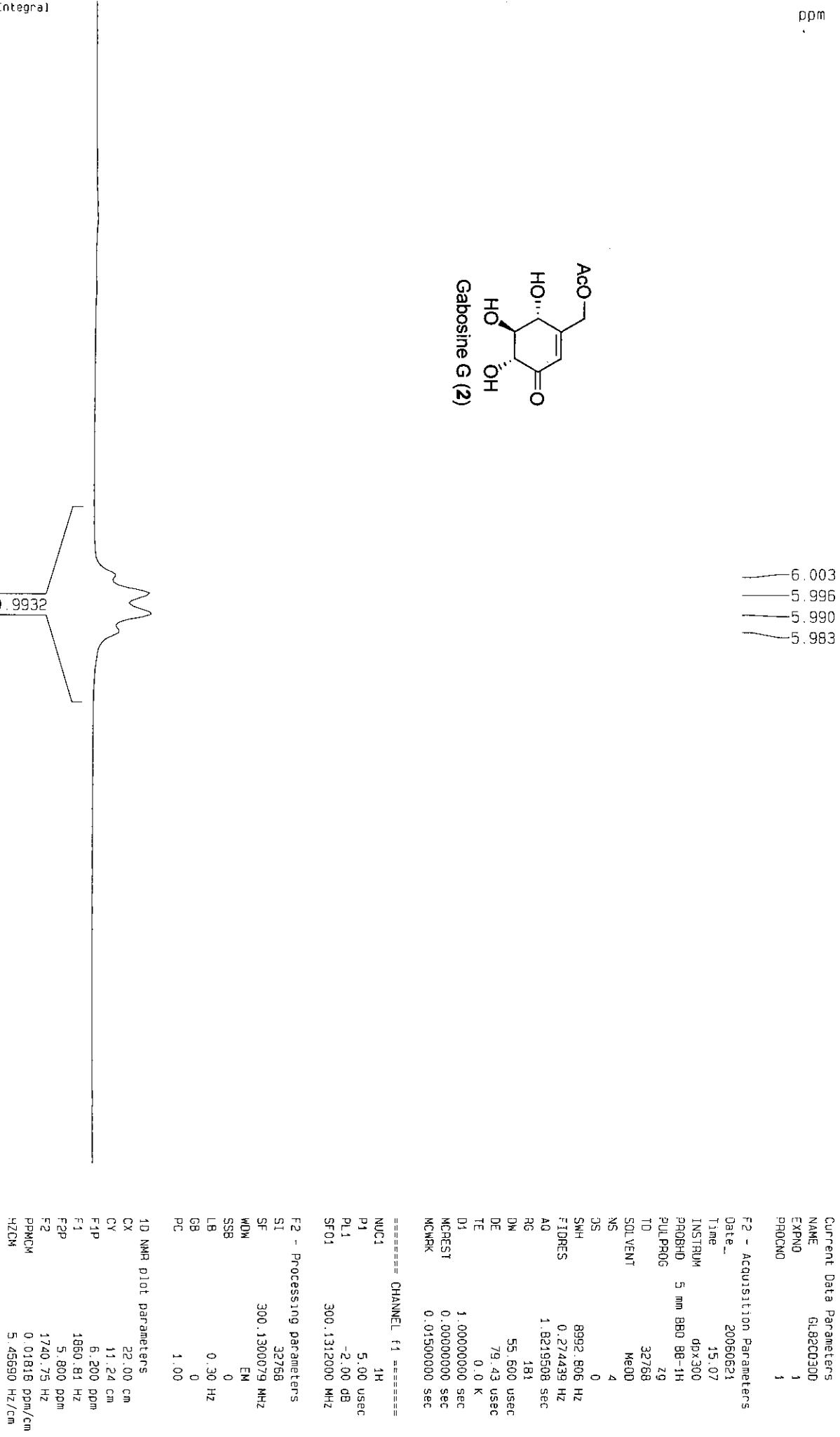
Expanded spectrum of Gabosine G(2)

S6

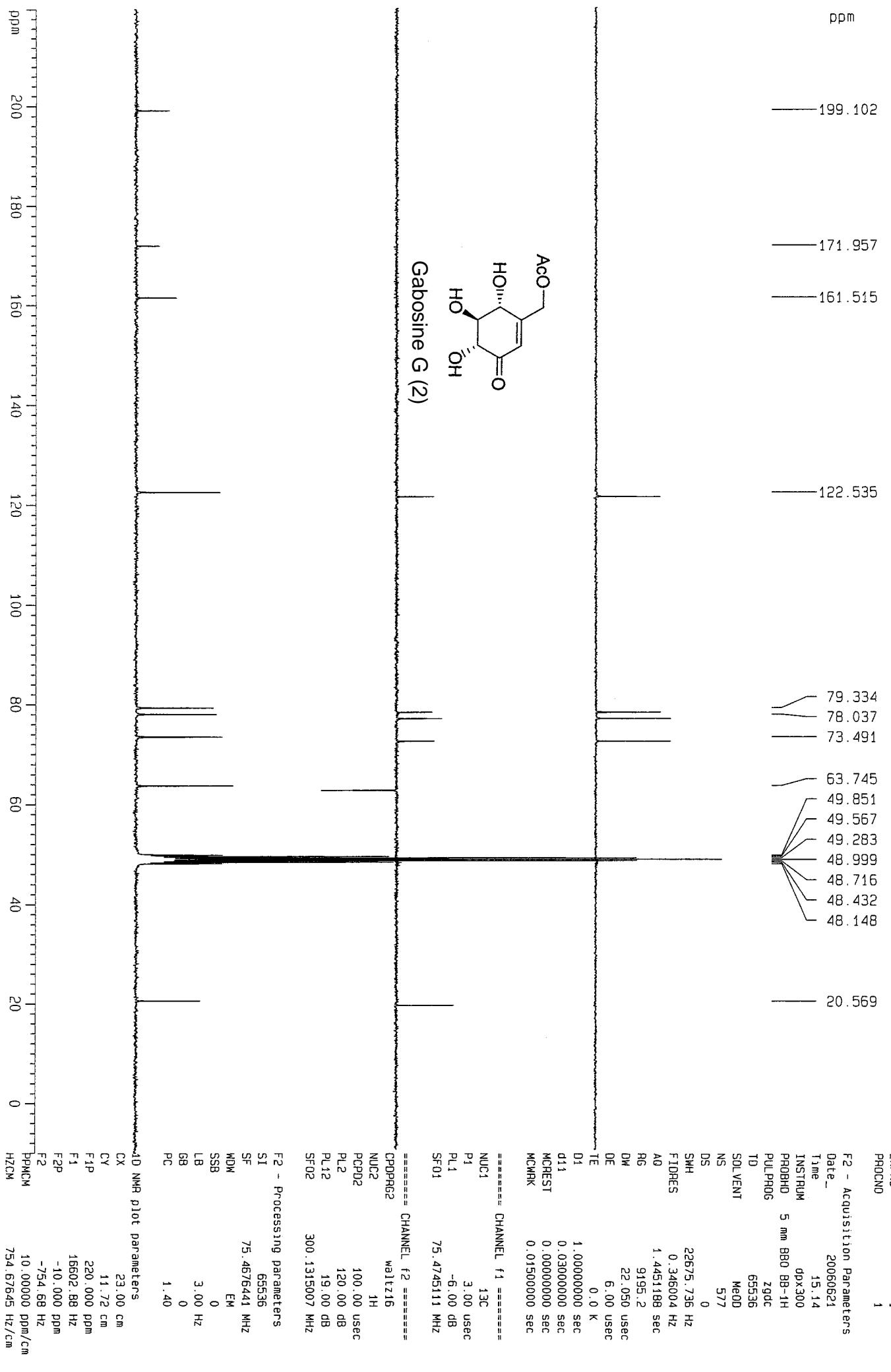


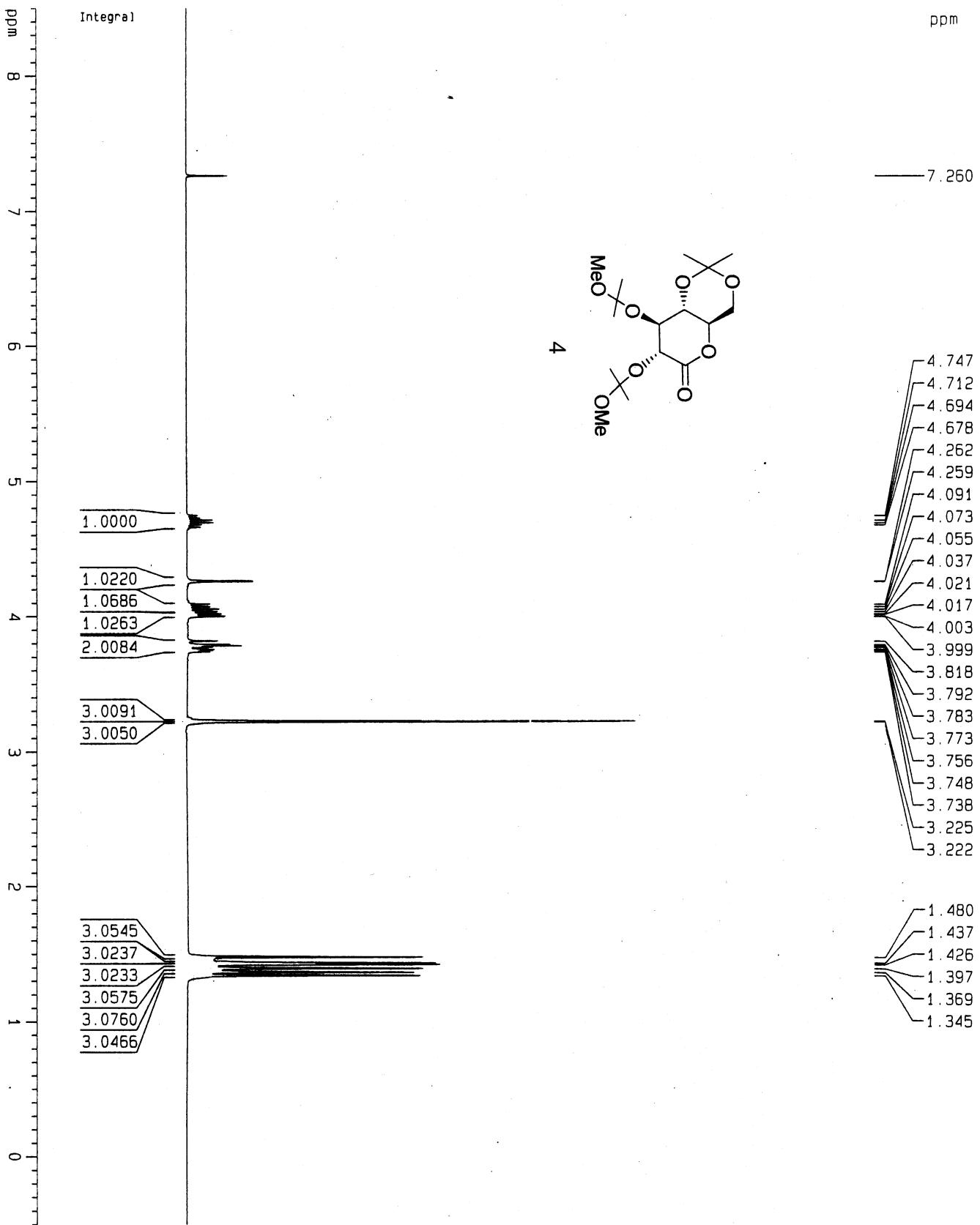
Expanded spectrum of Gabosine G(2)

S7



Solvent: CD3OD





===== CHANNEL f1 =====

NUC1 1H
 P1 5.00 usec
 PL1 -2.00 dB
 SFO1 300.1312000 MHz
 SI 32768
 SF 300.1300063 MHz
 MDW 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

F2 - Processing parameters

SI 32768
 EM 0
 F2 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 22.00 cm
 CY 8.29 cm
 F1P 8.500 ppm
 F1 2551.10 Hz
 F2P -0.500 ppm
 F2 -150.07 Hz
 PRF 0.40909 ppm/cm
 HZCM 122.78046 Hz/cm

Current Data Parameters
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 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date 2004/11/20
 Time 18:58
 INSTRUM dpx300
 PROBHD 5 mm BBO BB-1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8992.806 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 143.7
 DW 55.600 usec
 DE 79.43 usec
 TE 0.0 K
 D1 1.0000000 sec
 MCREST 0.0000000 sec
 MCWRK 0.01500000 sec



Current Data Parameters

NAME ^13C -1207-c13

TPMO 1

PROCNO 1

F2 - Acquisition Parameters

Date 20051207

TIME 9.44

INSTRUM dpx300

PROBHD 5 mm B80 BB-1H

ZULPROG zgpc

TD 65536

SOLVENT CDCl3

NS 234

DS 0

SWH 26455.037 Hz

ETR 0 403672 Hz

AQ 1 238680.0 sec

RG 18.900 usec

TE 6.00 usec

TM 472.2 K

TDZ 1 00000000 sec

D1 0 03000000 sec

MC 0 00000000 sec

MCR 0 01500000 sec

==== CHANNEL f1 =====

NUC1 ^{13}C

P1 3.00 usec

PL1 -6.00 dB

SF01 75.4745111 MHz

==== CHANNEL f2 =====

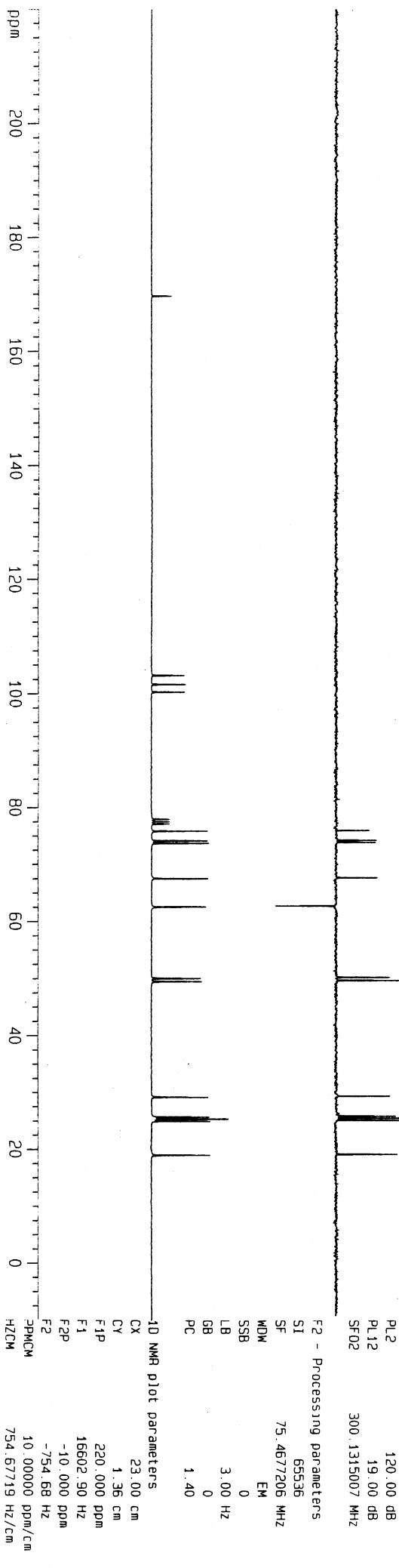
NUC2 ^1H

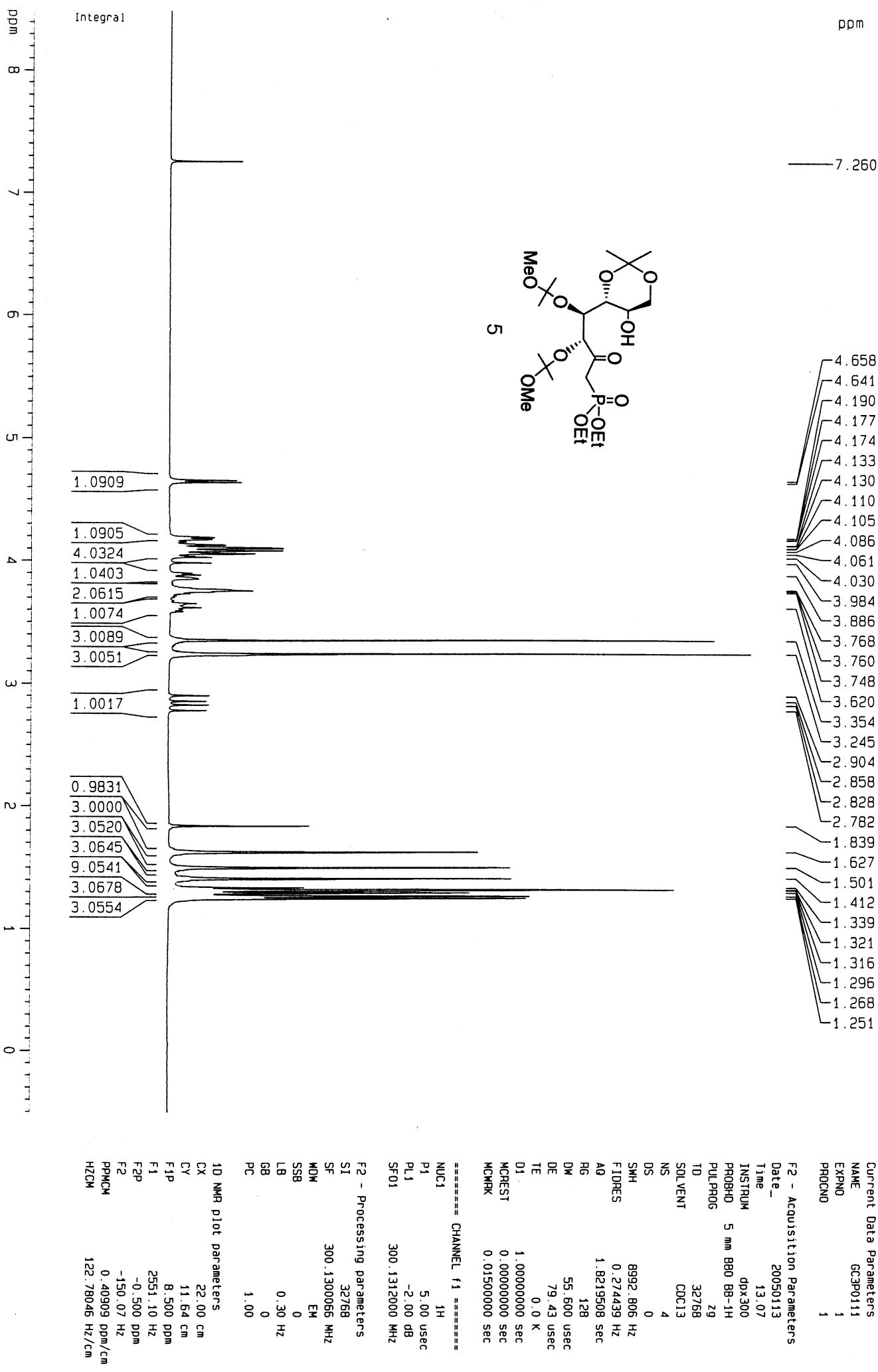
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PL2 120.00 dB

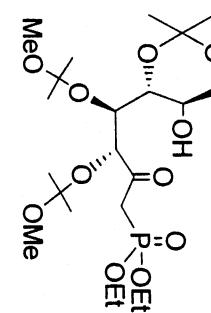
PL12 19.00 dB

SF02 300.1315007 MHz





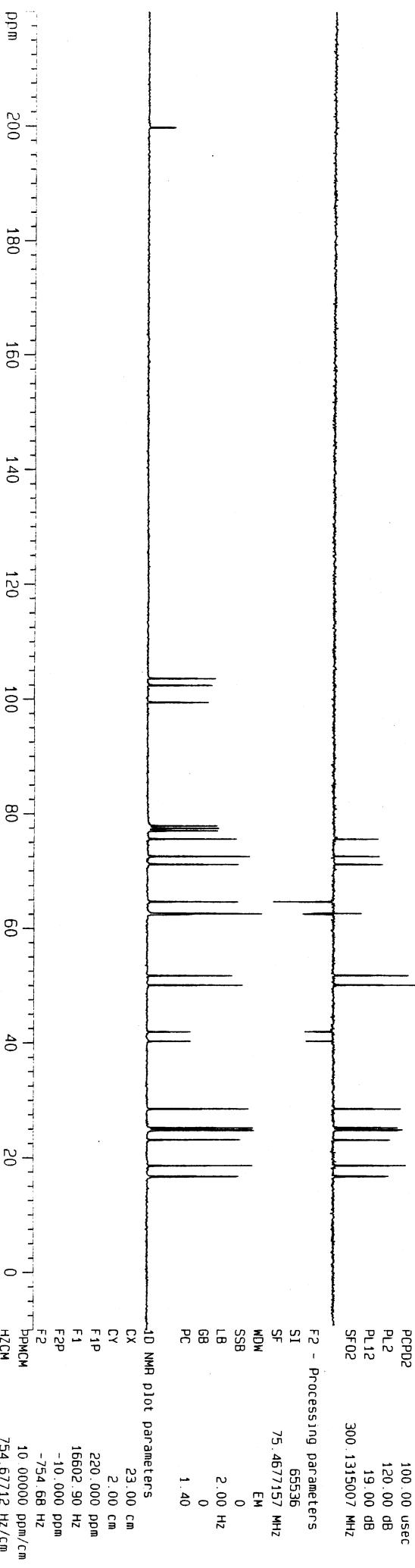
199.753
199.680



103.603
102.422
99.441
77.927
77.503
77.079
75.644
72.612
71.225
64.702
62.627
62.538
62.453
51.761
50.131
41.988
40.331
28.578
25.263
24.941
24.826
23.168
18.597
16.763
16.680

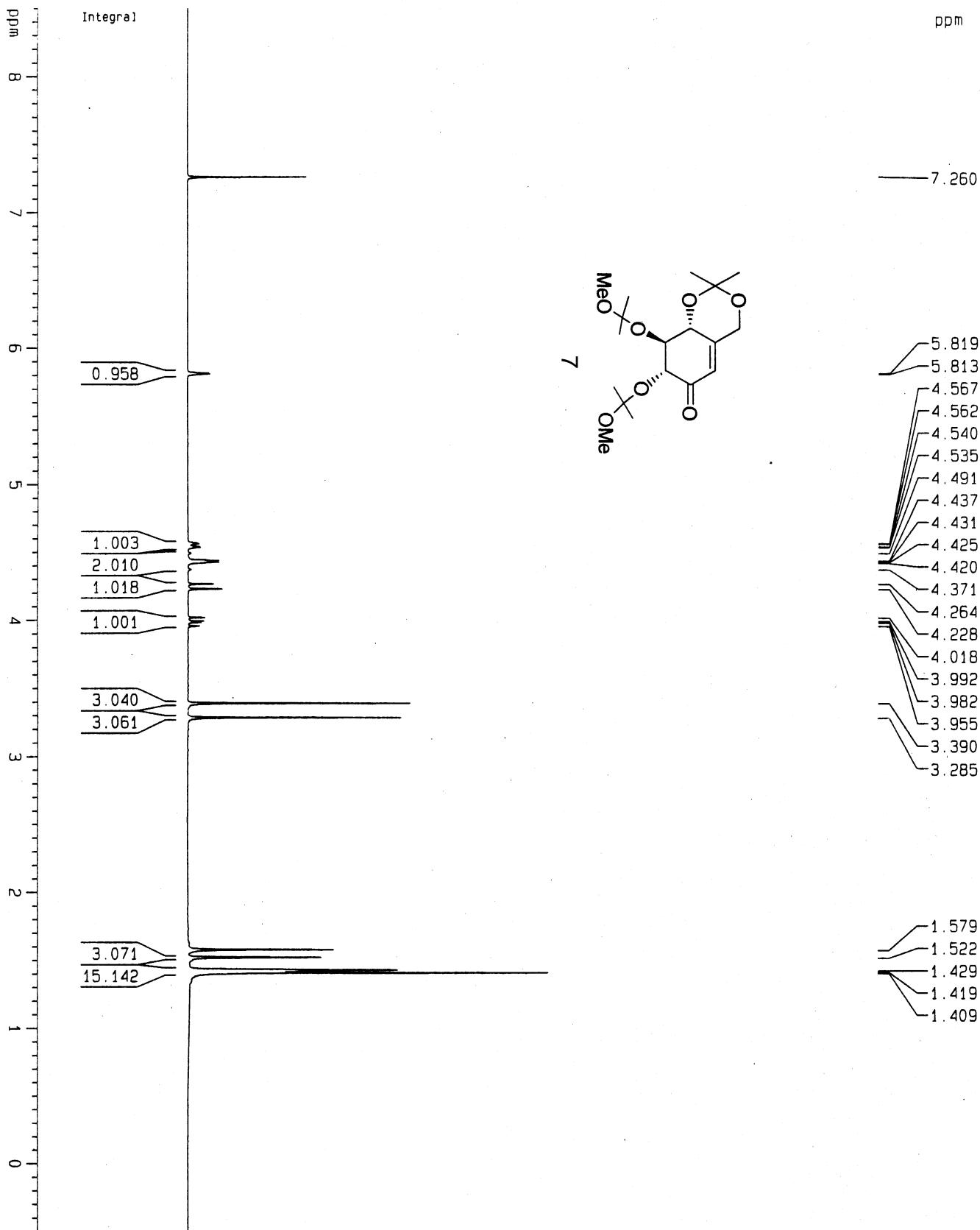
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PULPROG zgpc
TD 65536
SOLVENT CDCl3
NS 586
DS 0
SWH 26455.027 Hz
ETR 0 403672 Hz
AQ 1 2386804 sec
RG 2048
DW 18.900 usec
DE 6.00 usec
TE 297.2 K
D1 1.0000000 sec
d1 0.0300000 sec
MC 0 0000000 sec
MCREST 0 0000000 sec
MCWRK 0 01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
PL2 120.00 dB
PL12 19.00 dB
SF01 75.4745111 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCP02 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 300.1315007 MHz



ppm 200 180 160 140 120 100 80 60 40 20 0

Hz/cm 754.67712 Hz/cm



Current Data Parameters

NAME	GC5-1120-h1
EXPNO	1
PROCNO	2

F2 - Acquisition Parameters

TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8992.806 Hz
FORES	0.274439 Hz
AQ	1.8319508 sec
RG	645.1
DW	55.600 usec
DE	79.43 usec
TE	0.0000000 sec
D1	0.0000000 sec
MOREST	0.0150000 sec
MCMRK	

===== CHANNEL f1 =====

NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SF01	300.1312000 MHz

F2 - Processing parameters

SI	32768
SF	300.1300066 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	5.38 cm
FI _P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
Hz/cm	122.78046 Hz/cm

