

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

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Appendices

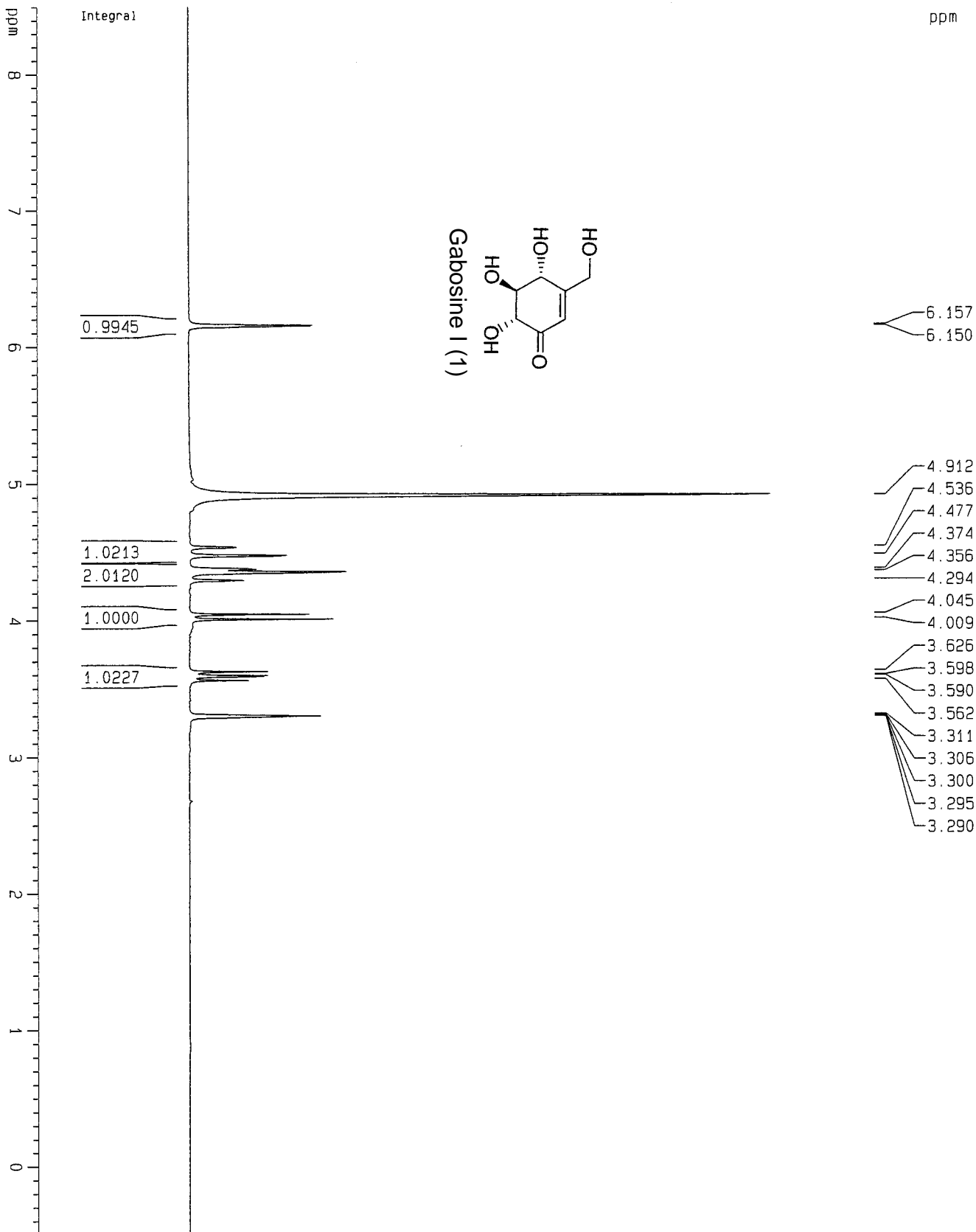
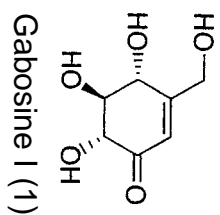
S1 List of contents

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



Current Data Parameters

NAME	GLBICD300
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20060615
Time	8.59

INSTRUM

PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768

SOLVENT

NS	4
DS	0

SMH

FIDRES	0.274439 Hz
AQ	1.8219508 sec

RG

DW	55.600 usec
DE	79.43 usec
TE	0.0 K

D1

MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1

P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1312000 MHz

F2 - Processing parameters

SI

SF	300.1300073 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	10.37 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.40909 ppm/cm
HZCM	122.78046 Hz/cm

Solvent: CD3OD

Current Data Parameters
NAME GLB1c13
EXPNO 1
PROCNO 1

ppm

199.515

168.080

121.330

79.457

78.116

73.735

62.059

49.853

49.569

49.285

49.001

48.717

48.434

48.150

F2 - Acquisition Parameters

Date_ 20060615

Time 9.04

INSTRUM dpx300

PROBHD 5 mm BBO BB-1H

PULPROG zgpg30

TD 65536

SOLVENT MeOD

NS 553

DS 0

SWH 22675.736 Hz

FIDRES 0.346004 Hz

AQ 1.4451188 sec

RG 10321.3

DW 22.050 usec

DE 6.00 usec

TE 0.0 K

D1 1.00000000 sec

d11 0.03000000 sec

MCREST 0.00000000 sec

MCMRK 0.01500000 sec

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

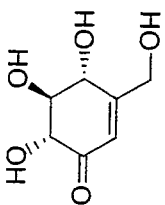
NUC1 13C

P1 3.00 usec

PL1 -6.00 dB

SF01 75.4745111 MHz

Gabosine I (1)



===== CHANNEL f2 =====

CPDPRG2 wa1tz16

NUC2 1H

PCPD2 100.00 usec

PL2 120.00 dB

PL12 19.00 dB

SF02 300.1345007 MHz

F2 - Processing parameters

SI 65536

SF 75.4676441 MHz

WDW EM

SSB 0

LB 3.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 23.00 cm

CY 11.72 cm

F1P 220.000 ppm

F1 16502.88 Hz

F2P -10.000 ppm

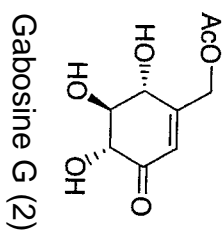
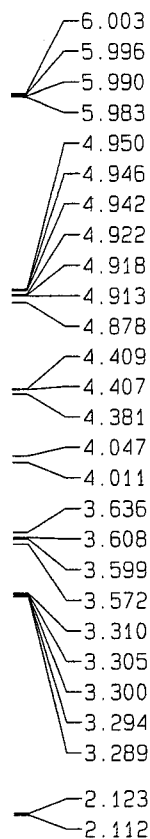
F2 -754.68 Hz

PCMC 10.00000 ppm/cm

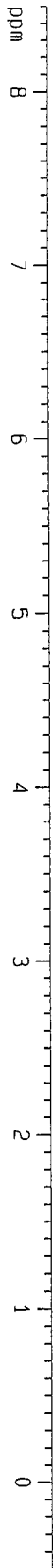
H2CM 754.67645 Hz/cm

Solvent: CD3OD

ppm



Integral



Current Data Parameters
 NAME GL82CD30D
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060621
 Time 15.07

INSTRUM dpx300
 PROBD 5 mm BBO BB-1H
 PULPROG zg

TD 32768
 SOLVENT MeOD
 NS 4

DS 0
 SMH 8992.806 Hz
 FIDRES 0.274439 Hz

AQ 1.8219508 sec
 RG 181

DW 55.600 usec
 DE 79.43 usec
 TE 0.0 K

D1 1.00000000 sec
 MCOREST 0.00000000 sec
 MCMRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 5.00 usec
 PL1 -2.00 dB

SFO1 300.1312000 MHz

F2 - Processing parameters
 SI 32768
 SF 300.130079 MHz

WDW EM
 SSB 0
 LB 0.30 Hz

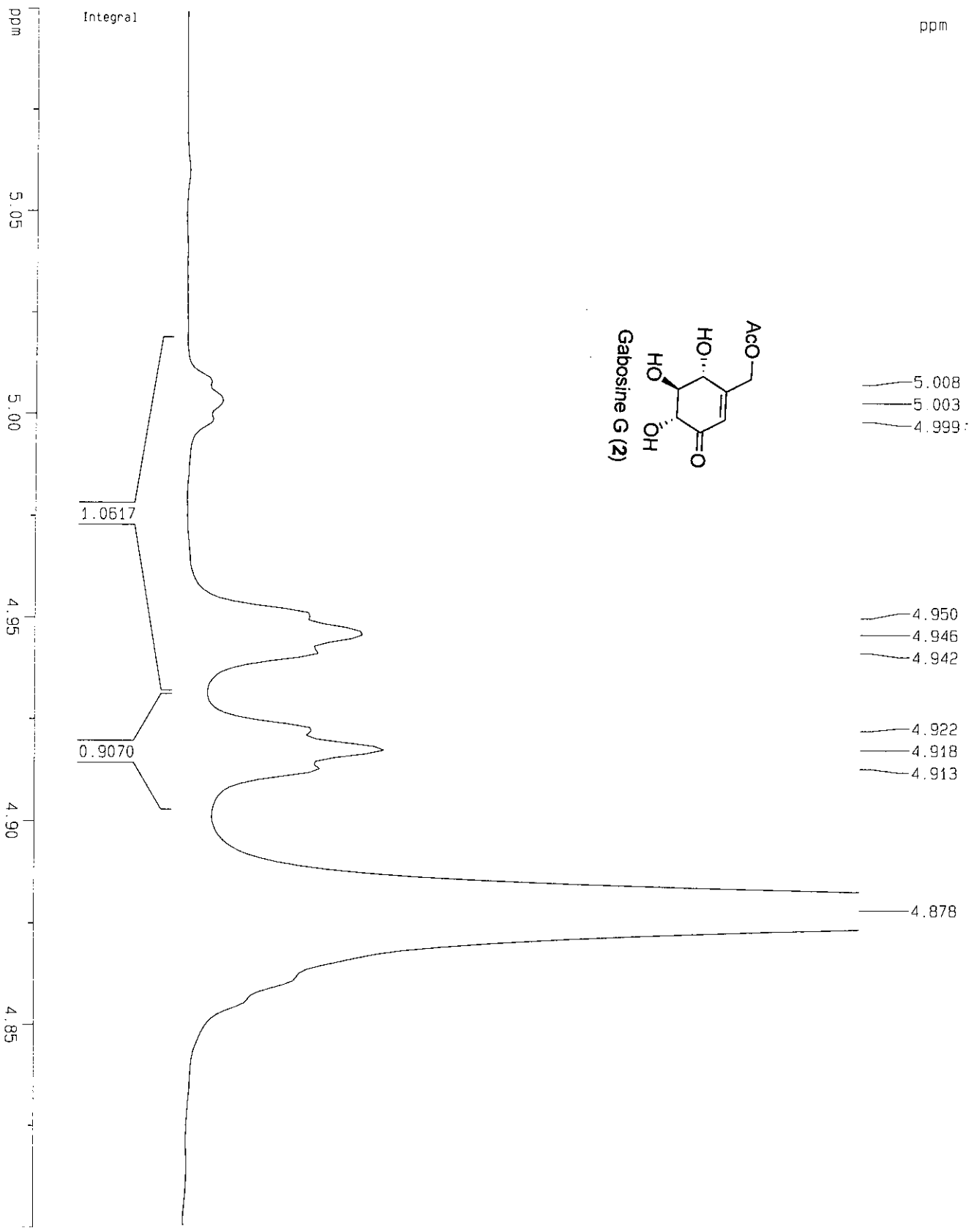
GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 11.24 cm

F1P 8.500 ppm
 F1 2551.10 Hz
 F2P -0.500 ppm

F2 -150.07 Hz
 PPMCM 0.40909 ppm/cm
 HZCM 122.78046 Hz/cm

Expanded spectrum of Gabosine G(2)



Current Data Parameters
NAME GL82CD30D
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060621
Time 15.07

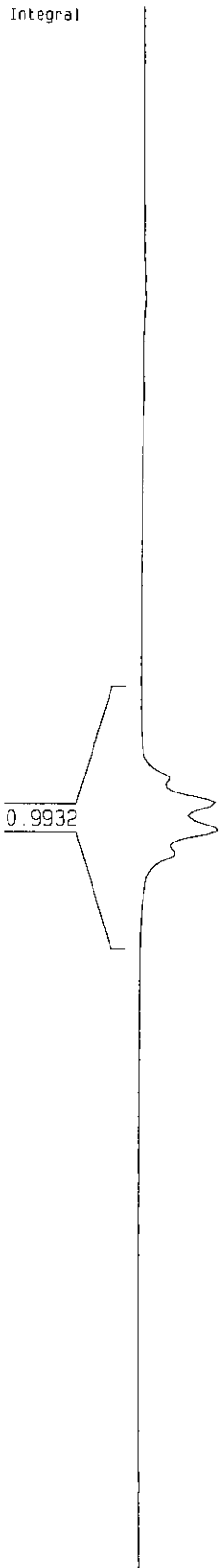
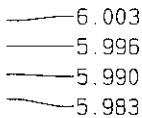
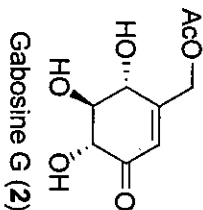
INSTRUM dpx300
PROBHD 5 mm BB0 BB-1H
PULPROG zg
TD 32768
SOLVENT MeOD
NS 4
DS 0
SWH 8992.806 Hz
FIDRES 0.27439 Hz
AQ 1.8219508 sec
RG 181
DM 55.600 usec
DE 79.43 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCNMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 5.00 usec
PL1 -2.00 dB
SFO1 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 300.1300079 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 30.23 cm
F1P 5.100 ppm
F1 1530.66 Hz
F2P 4.800 ppm
F2 1440.62 Hz
PPMCM 0.01354 ppm/cm
HZCM 4.09268 Hz/cm

Expanded spectrum of Gabosine G(2)



Current Data Parameters
NAME GL82CD30D
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060621
Time 15.07
INSTRUM dpx300
PROBHD 5 mm BB0 BB-1H
PULPROG zg
TD 32768
SOLVENT MeOD
NS 4
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 181
DM 55.600 usec
DE 79.43 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 5.00 usec
PL1 -2.00 dB
SFO1 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 300.1300079 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 11.24 cm
F1P 6.200 ppm
F1 1860.81 Hz
F2P 5.800 ppm
F2 1740.75 Hz
PPMCM 0.01818 ppm/cm
HZCM 5.45690 Hz/cm

Solvent: CD3OD

ppm

199.102

171.957

161.515

122.535

79.334

78.037

73.491

63.745

49.851

49.567

49.283

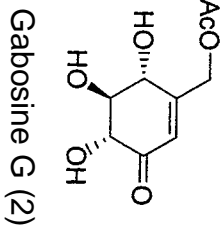
48.999

48.716

48.432

48.148

20.569



Current Data Parameters
NAME GLB2c13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060521
Time 15.14

INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgpgc

TD 65536
SOLVENT MeOD

NS 577
DS 0

SWH 22675.736 Hz
FIDRES 0.346004 Hz

AQ 1.4451188 sec
RG 9195.2

DM 22.050 usec
DE 6.00 usec

TE 0.0 K
D1 1.00000000 sec

d11 0.03000000 sec
MCREST 0.00000000 sec

MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SF01 75.4745111 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4576441 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

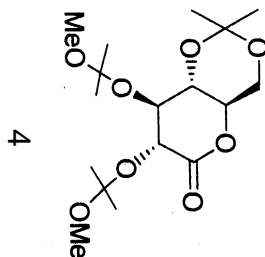
1D NMR plot parameters
CX 23.00 cm
CY 11.72 cm
F1P 220.000 ppm
F1 16602.88 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67645 Hz/cm

ppm

7.260

4.747
4.712
4.694
4.678
4.262
4.259
4.091
4.073
4.055
4.037
4.021
4.017
4.003
3.999
3.818
3.792
3.783
3.773
3.756
3.748
3.738
3.225
3.222

1.480
1.437
1.426
1.397
1.369
1.345



Integral

1.0000

1.0220

1.0686

1.0263

2.0084

3.0091

3.0050

3.0545

3.0237

3.0233

3.0575

3.0760

3.0466

Current Data Parameters
NAME GC2-1120-n1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041120
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 0.27439 Hz
AQ 1.8219508 sec
RG 143.7
DM 55.600 usec
DE 79.43 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWPK 0.01500000 sec

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

NUC1 1H
P1 5.00 usec
PL1 -2.00 dB
SFO1 300.1312000 MHz

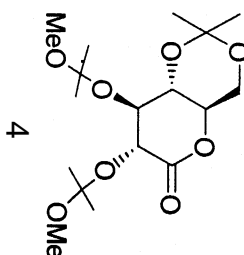
F2 - Processing parameters
SI 32768
SF 300.130063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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
PPMCM 0.40909 ppm/cm
HZCM 122.78046 Hz/cm

ppm 8 7 6 5 4 3 2 1 0

ppm

169.574

103.141
101.571
100.22177.924
77.499
77.074
75.816
74.105
73.704
67.474
62.50150.036
49.45429.218
25.710
25.371
25.343
24.989
18.978

Current Data Parameters
NAME GC2-1207-C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051207

Time 9.44
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 234
DS 0
SWH 26455.027 Hz
FIDRES 0.403672 Hz
AQ 1.2386804 sec
RG 2048
DM 18.900 usec
DE 6.00 usec
TE 472.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

NUC1 ¹³C
P1 3.00 usec
PL1 -6.00 dB
SFO1 75.474511 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 ¹H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters

SI 65536
SF 75.4677206 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 23.00 cm
CY 1.36 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67719 Hz/cm

ppm

200

180

160

140

120

100

80

60

40

20

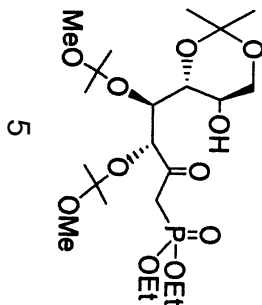
0

PPMCM

HZCM

ppm

7.260



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4.641
4.190
4.177
4.174
4.133
4.130
4.110
4.105
4.086
4.061
4.030
3.984
3.886
3.768
3.760
3.748
3.620
3.354
3.245
2.904
2.858
2.828
2.782
1.839
1.627
1.501
1.412
1.339
1.321
1.316
1.296
1.268
1.251

1.0909
1.0905
4.0324
1.0403
2.0615
1.0074
3.0089
3.0051
1.0017
0.9831
3.0000
3.0520
3.0645
9.0541
3.0678
3.0554

ppm

8

7

6

5

4

3

2

1

0

Integral

Current Data Parameters
NAME GC3P0111
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050113
Time 13.07

INSTRUM dpx300

PROBHD 5 mm BBO BB-1H

PULPROG zg

TD 32768

SOLVENT CDCl3

NS 4

DS 0

SMH 8992.806 Hz

FIDRES 0.274439 Hz

AQ 1.8219508 sec

RG 128

DW 55.600 usec

DE 79.43 usec

TE 0.0 K

D1 1.00000000 sec

MCHEST 0.00000000 sec

MCNKR 0.01500000 sec

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

NUC1 1H

P1 5.00 usec

PL1 -2.00 dB

SFO1 300.1312000 MHz

F2 - Processing parameters

SI 32768

SF 300.1300066 MHz

MDM EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 22.00 cm

CY 11.64 cm

F1P 8.500 ppm

F1 2551.10 Hz

F2P -0.500 ppm

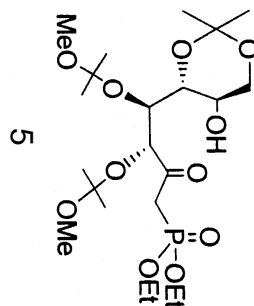
F2 -150.07 Hz

PPMCH 0.40909 ppm/cm

HZCH 122.78046 Hz/cm

ppm

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

Current Data Parameters
NAME GC3P-C13-1208
EXPNO 1
PROCNO 2

F2 - Acquisition Parameters

Date_ 20051209
Time 13.04
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 65536
SOLVENT CDCl3
VS 586
DS 0
SWH 26455.027 Hz
FIDRES 0.403672 Hz
AQ 1.2386804 sec
RG 2048
DM 18.900 usec
DE 6.00 usec
TE 297.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

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

NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SFO1 75.4745111 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters

SI 65536
SF 75.4677157 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 23.00 cm
CY 2.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm
F2 -754.68 Hz
DPMCM 10.00000 ppm/cm
HZCM 754.67712 Hz/cm

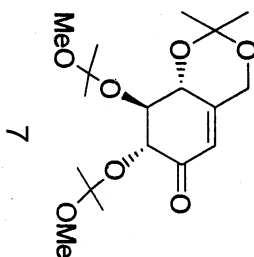
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180
160
140
120
100
80
60
40
20
0

ppm

7.260

5.819
5.813
4.567
4.562
4.540
4.535
4.491
4.437
4.431
4.425
4.420
4.371
4.264
4.228
4.018
3.992
3.982
3.955
3.390
3.285

1.579
1.522
1.429
1.419
1.409



ppm 8 7 6 5 4 3 2 1 0

Integral

0.958

1.003

2.010

1.018

1.001

3.040

3.061

3.071

15.142

Current Data Parameters
NAME GC5-1120-n1
EXPNO 1
PROCNO 2

F2 - Acquisition Parameters
Date_ 20041120
Time 19.12

INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg

TD 32768
SOLVENT CDCl3

NS 32
DS 0

SMH 8992.806 Hz
FIDRES 0.27439 Hz

AQ 1.8219508 sec
RG 645.1

DW 55.600 usec
DE 79.43 usec

TE 0.0 K
D1 1.00000000 sec

MCREST 0.00000000 sec
MCMRK 0.01500000 sec

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

NUC1 1H

P1 5.00 usec

PL1 -2.00 dB

SFO1 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 6.38 cm

F1P 8.500 ppm

F1 2551.10 Hz

F2P -0.500 ppm

F2 -150.07 Hz

PPMCM 0.40909 ppm/cm

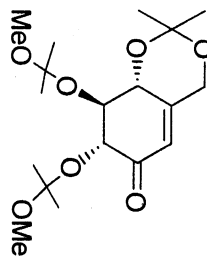
HZCM 122.78046 Hz/cm

ppm

196.947

157.480

121.296

102.637
102.389
100.78577.927
77.502
77.077
76.095
74.646
72.768
61.81850.491
50.43426.376
25.926
25.766
25.744
25.634
22.597

7

Current Data Parameters
NAME GC5C13060107
EXPNO 1
PROCNO 2

F2 - Acquisition Parameters
Date_ 20060107

Time 10.02
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 581
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.445188 sec
RG 8192
DM 22.050 usec
DE 6.00 usec
TE 297.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SF01 75.4745111 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4677205 MHz
WDW EM
SSB 0
LB 2.50 Hz
GB 0
PC 1.40

10 NMR plot parameters

CX 23.00 cm
CY 2.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67719 Hz/cm

ppm

200

180

160

140

120

100

80

60

40

20

0

PPMCM
HZCM