

Supporting Information

Facile syntheses of (+)-gabosines A, D, and E

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Appendices

S1 List of contents

S2 General procedures

S3-S10 ¹H, ¹³C and DEPT NMR Spectra of compounds **4**, **5**, **12–15** and **17–22**.

General procedures

Melting points were measured 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 (230–400 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. 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.

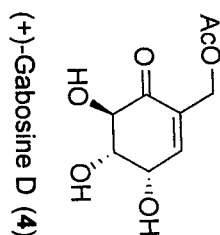
Solvent: CD₃OD

ppm

6.935
6.930
6.926
6.917
6.913
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4.878
4.737
4.734
4.499
4.482
4.468
4.338
4.306
3.811
3.798
3.779
3.766
3.305
3.300
3.295

2.061



Current Data Parameters
NAME GL145-h1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070530

Time 21.18

INSTRUM dpx300

PROBHD 5 mm BB0 BB-1H

PULPROG zg

TD 32768

SOLVENT MeOD

NS 8

DS 0

SWH 5402.485 Hz

FIDRES 0.164871 Hz

AQ 3.0327284 sec

RG 228.1

DW 92.550 usec

DE 132.21 usec

TE 0.0 K

D1 1.00000000 sec

MCREST 0.00000000 sec

MCWRR 0.01500000 sec

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

NUC1 1H

P1 5.00 usec

PL1 -2.00 dB

SFO1 300.1318000 MHz

F2 - Processing parameters

SI 32768

SF 300.1300076 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 22.00 cm

CY 10.38 cm

F1P 8.000 ppm

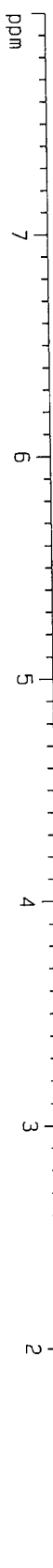
F1 2401.04 Hz

F2P 1.000 ppm

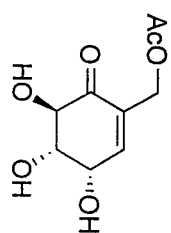
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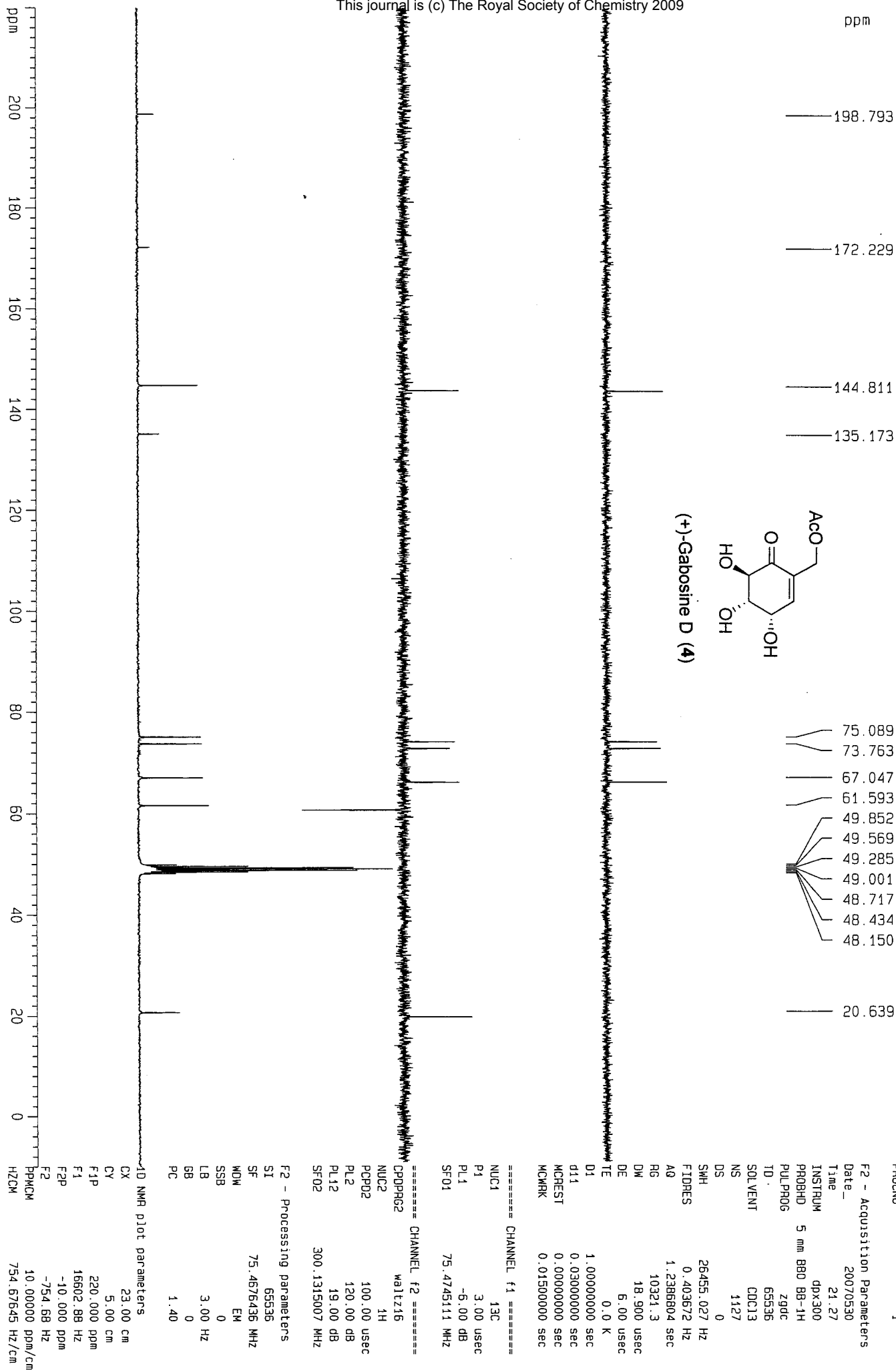
HZCM 95.49591 Hz/cm



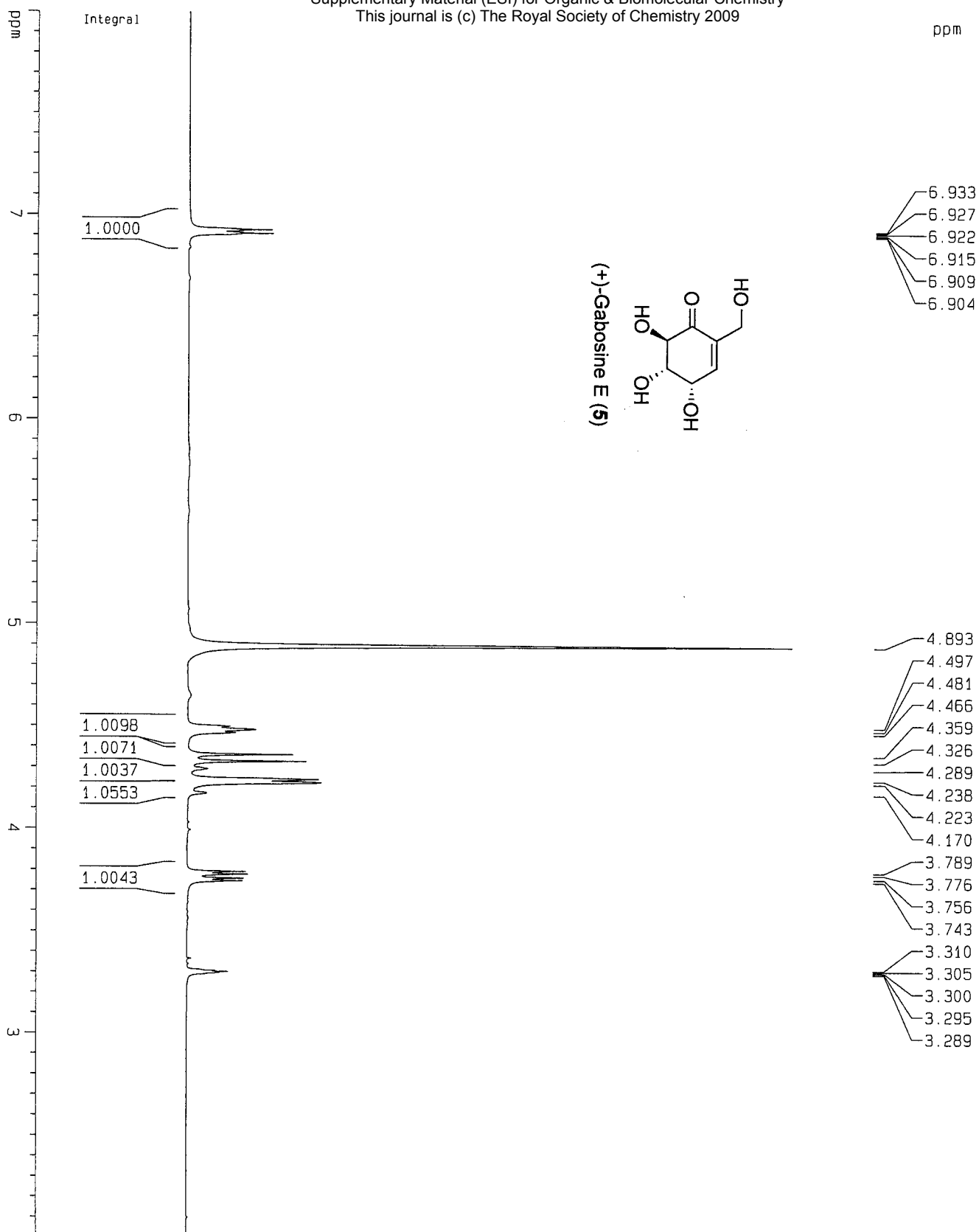
Solvent: CD₃OD



(+)-Gabosine D (4)



Solvent: CD₃OD



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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070606
Time 18.45
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT MeOD
NS 8
DS 0
SWH 5402.485 Hz
FIDRES 0.164871 Hz
AQ 3.0327284 sec
RG 181
DW 92.550 usec
DE 132.21 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

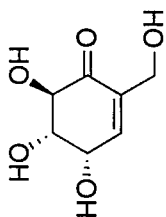
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P1 5.00 usec
PL1 -2.00 dB
SFO1 300.1318000 MHz

F2 - Processing parameters
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WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 10.75 cm
F1P 8.000 ppm
F1 2401.04 Hz
F2P 2.000 ppm
F2 600.26 Hz
PPMCM 0.27273 ppm/cm
HZCM 81.85364 Hz/cm

Solvent: CD₃OD

(+)-Gabosine E (5)



ppm

199.676

141.835

139.931

75.145

73.894

67.090

59.472

49.852

49.568

49.284

49.000

48.716

48.432

48.148

Current Data Parameters
NAME GL148-c13
EXPNO 1
PROCNO 1

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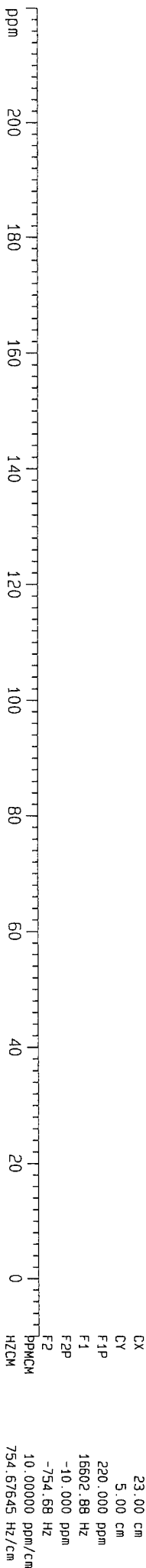
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 140
DS 0
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FIDRES 0.403672 Hz
AQ 1.236804 sec
RG 5792.6
DW 18.900 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWAK 0.01500000 sec

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

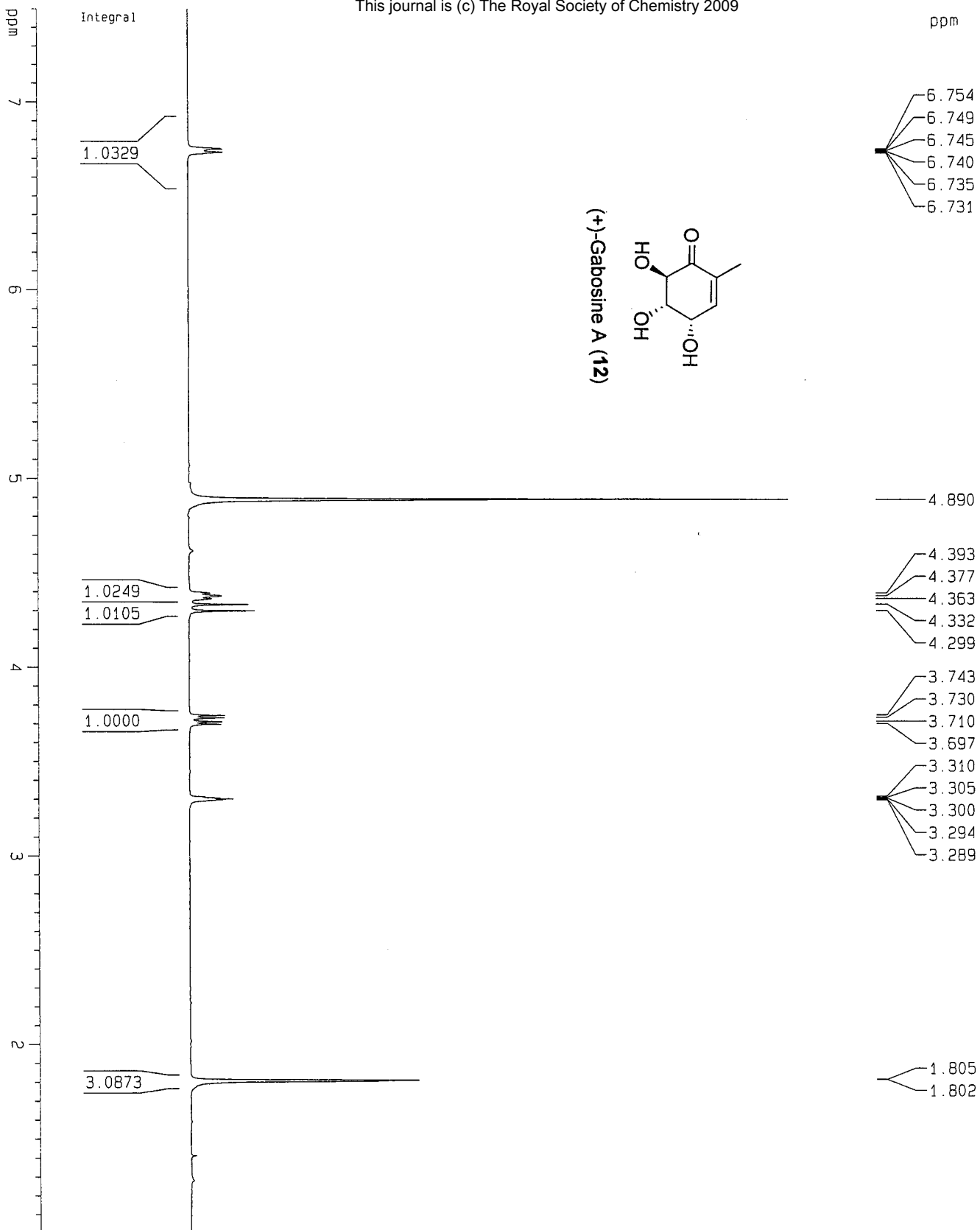
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CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4676448 MHz
WDW EM
SSB 0
LB 2.50 Hz
GB 0
PC 1.40

1D NMR plot parameters



Solvent: CD₃OD



Current Data Parameters
NAME GL154-h1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070709
Time 10.17

INSTRUM dpX300
PROBHD 5 mm BBO BB-1H
PULPROG zg

TD 32768
SOLVENT MeOD

NS 8
DS 0

SWH 5402.485 Hz
FIDRES 0.164871 Hz
AQ 3.0327284 sec

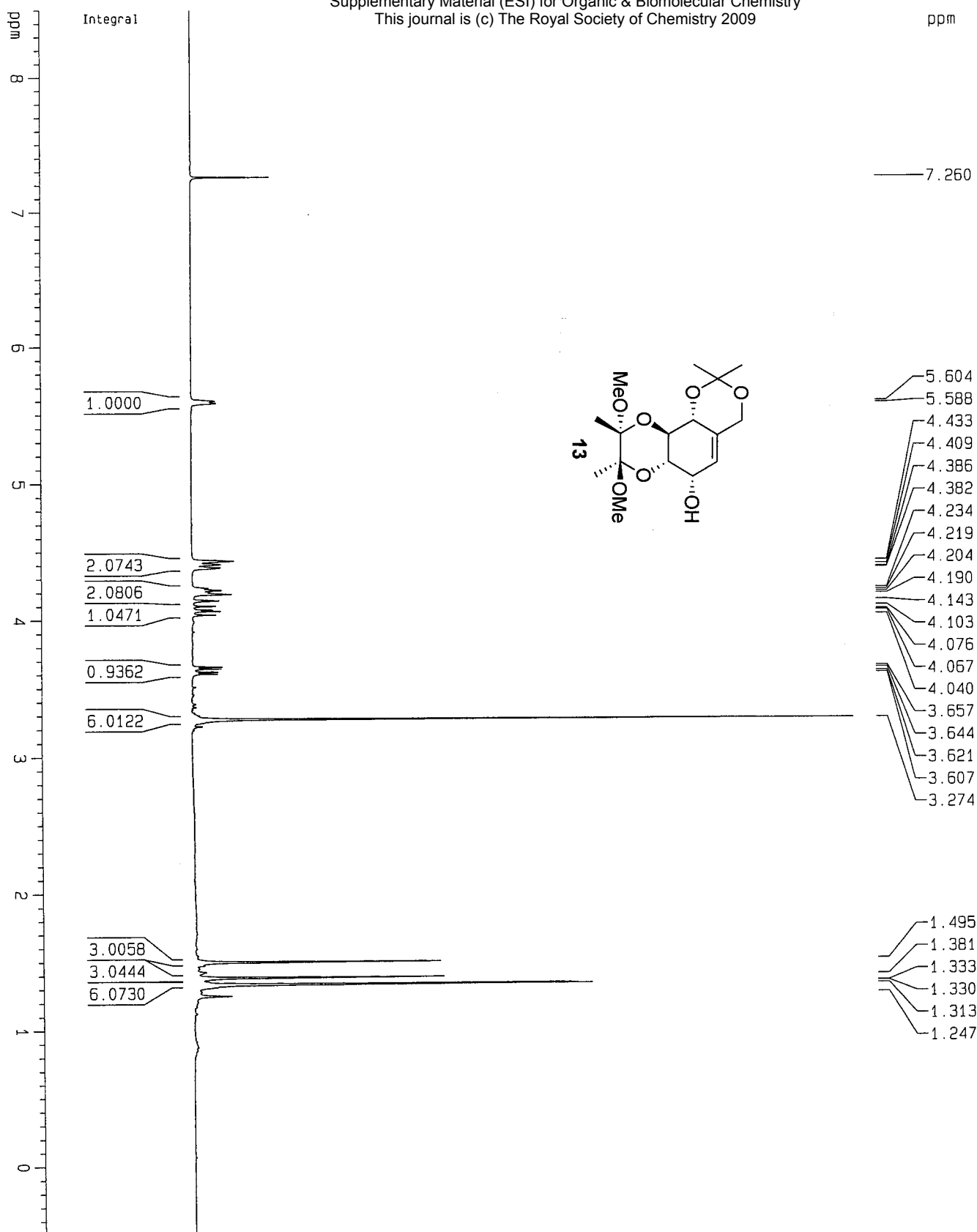
RG 228.1
DE 92.550 usec
TE 0.0 K

D1 1.00000000 sec
MCREST 0.00000000 sec
MCNMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 5.00 usec
PL1 -2.00 dB
SF01 300.1318000 MHz

F2 - Processing Parameters
SI 32768
SF 300.1300078 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 10.66 cm
F1P 7.500 ppm
F1 2250.98 Hz
F2P 1.000 ppm
F2 300.13 Hz
PPMCM 0.29545 ppm/cm
HZCM 88.67478 Hz/cm



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F2 - Acquisition Parameters
Date_      20050521

```

INSTIRUM 0px300

PULPHUS	29
ID	33768

CONV. NS

SMH	B592.806
CIDRRC	0 37443D

RG	181
EL	200

TE 296.2

2000

NOCl	17
PA	500

32/bi
300 470055

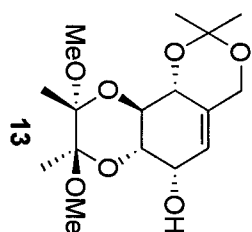
MDM

68

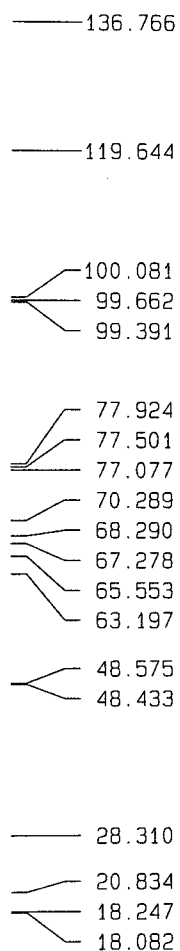
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F.1p 8.20

F2 -150.0



ppm



Current Data Parameters
NAME GL44c13
EXPNO 1
PROCNO 2

F2 - Acquisition Parameters
Date_ 20050520

Time 11.31
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 882
DS 0
SWH 26455.027 Hz
FIDRES 0.403672 Hz
AQ 1.2386804 sec
RG 2896.3
DW 18.900 usec
DE 6.00 usec
TE 297.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

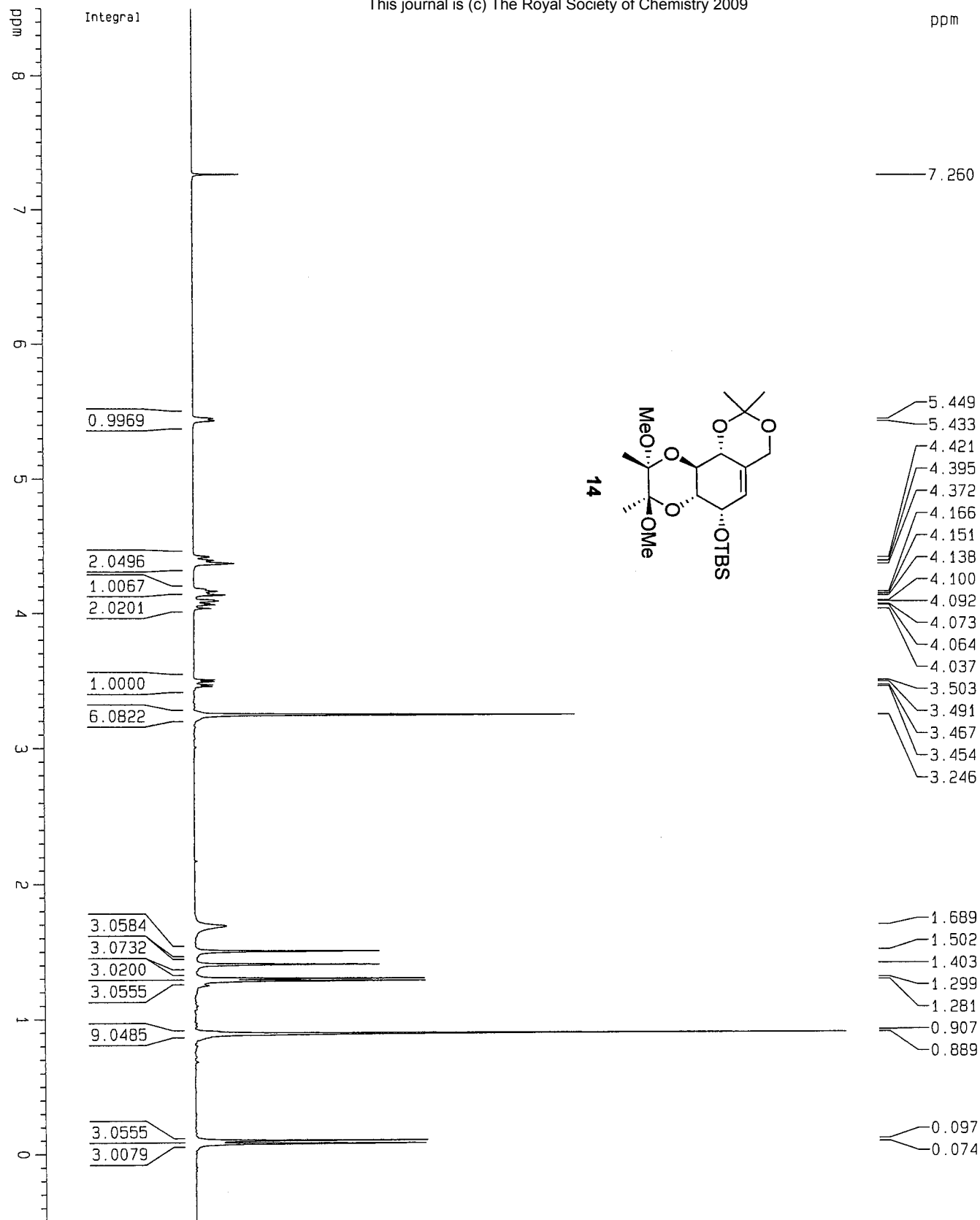
===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SFO1 75.4745111 MHz

===== CHANNEL f2 =====
CQPPRG2 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 75.4677141 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 23.00 cm
CY 4.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67712 Hz/cm



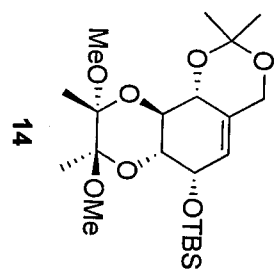
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EXPNO 1
PROCNO 1

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Time 11.14
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4803.074 Hz
FIDRES 0.146578 Hz
AQ 3.4111989 sec
RG 128
DM 104.100 usec
DE 148.71 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWPK 0.01500000 sec

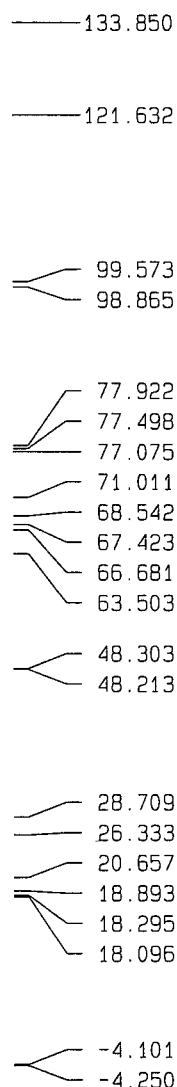
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PL1 -2.00 dB
SF01 300.1318000 MHz

F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.50 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
PPMCM 0.40909 ppm/cm
HZCM 122.78046 Hz/cm



ppm



Current Data Parameters
NAME GL141-c13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 11:21

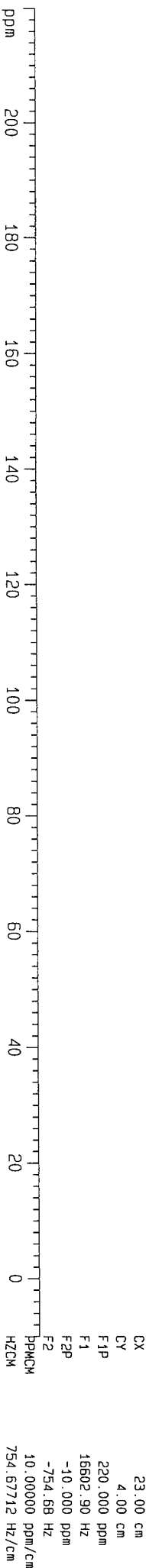
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PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 335
DS 0
SWH 26455.027 Hz
FIDRES 0.403672 Hz
AQ 1.2386804 sec
RG 5792.6
DM 18.900 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWPK 0.01500000 sec

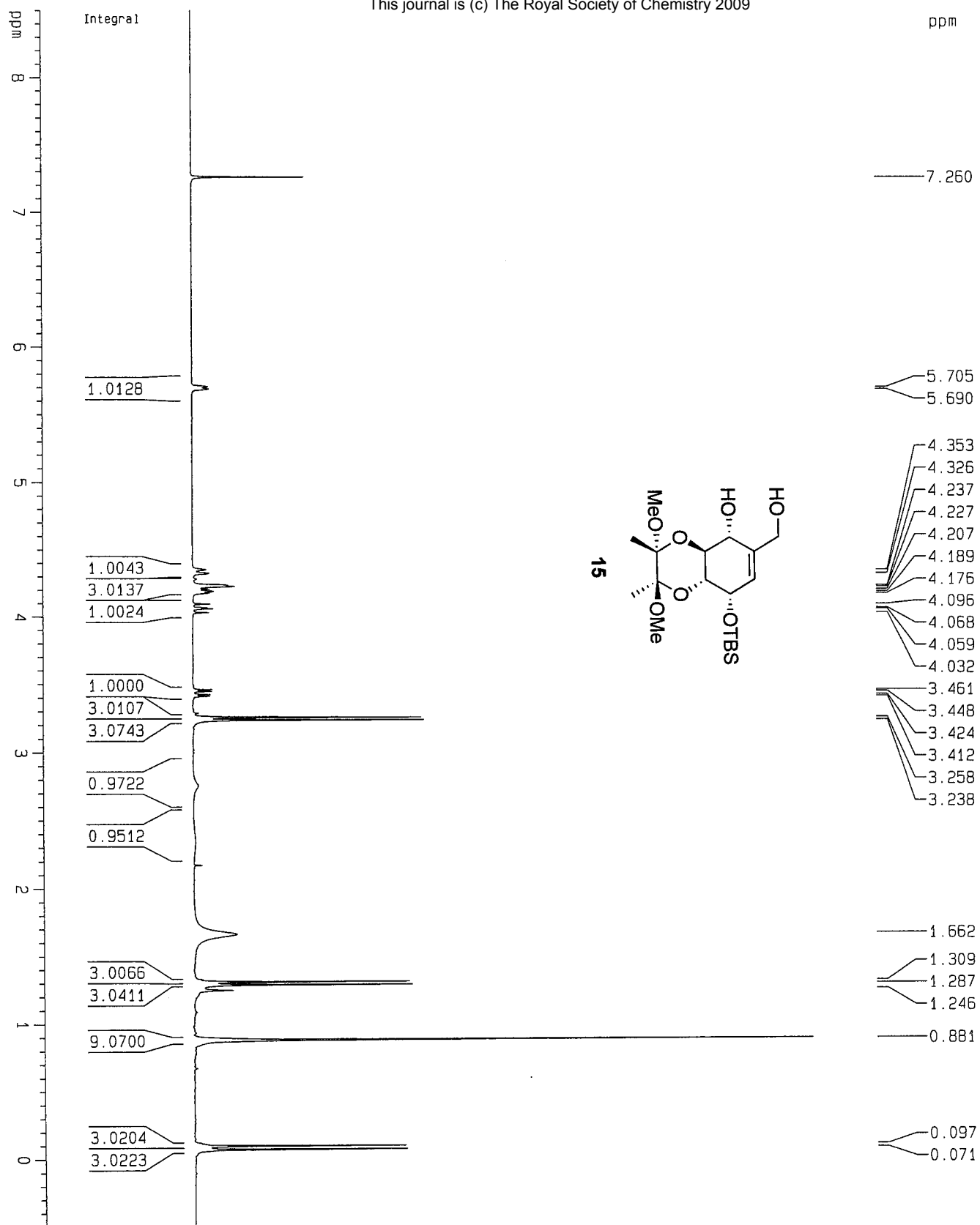
===== CHANNEL f1 =====
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SF01 75.474511 MHz

===== CHANNEL f2 =====
CQPRG2 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 75.4677129 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

ID NMR plot parameters





Current Data Parameters

NAME	GL142-h1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20070523
Time	16.02
INSTRUM	dpX300
PROBHD	5 mm BB0 BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4803.074 Hz
FIDRES	0.146578 Hz
AQ	3.411989 sec
RG	362
DW	104.100 usec
DE	148.71 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

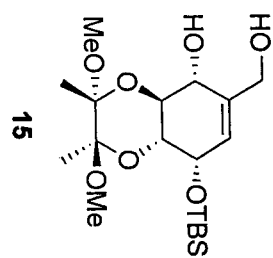
NUC1	¹ H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1318000 MHz

F2 - Processing parameters

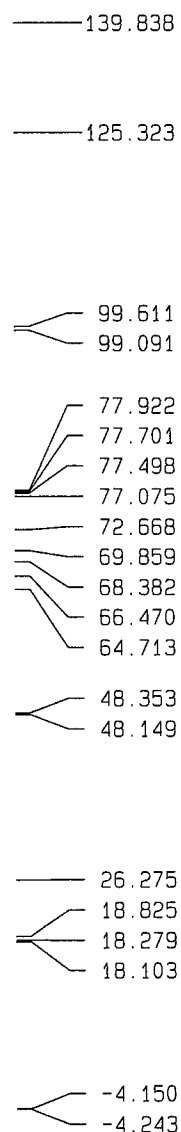
SI	32768
SF	300.130063 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.09 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



Current Data Parameters
NAME GL142-c13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070523

Time 16.50
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1230
DS 0
SWH 26455.027 Hz
FIDRES 0.403672 Hz
AQ 1.2386804 sec
RG 5792.6
DM 18.900 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

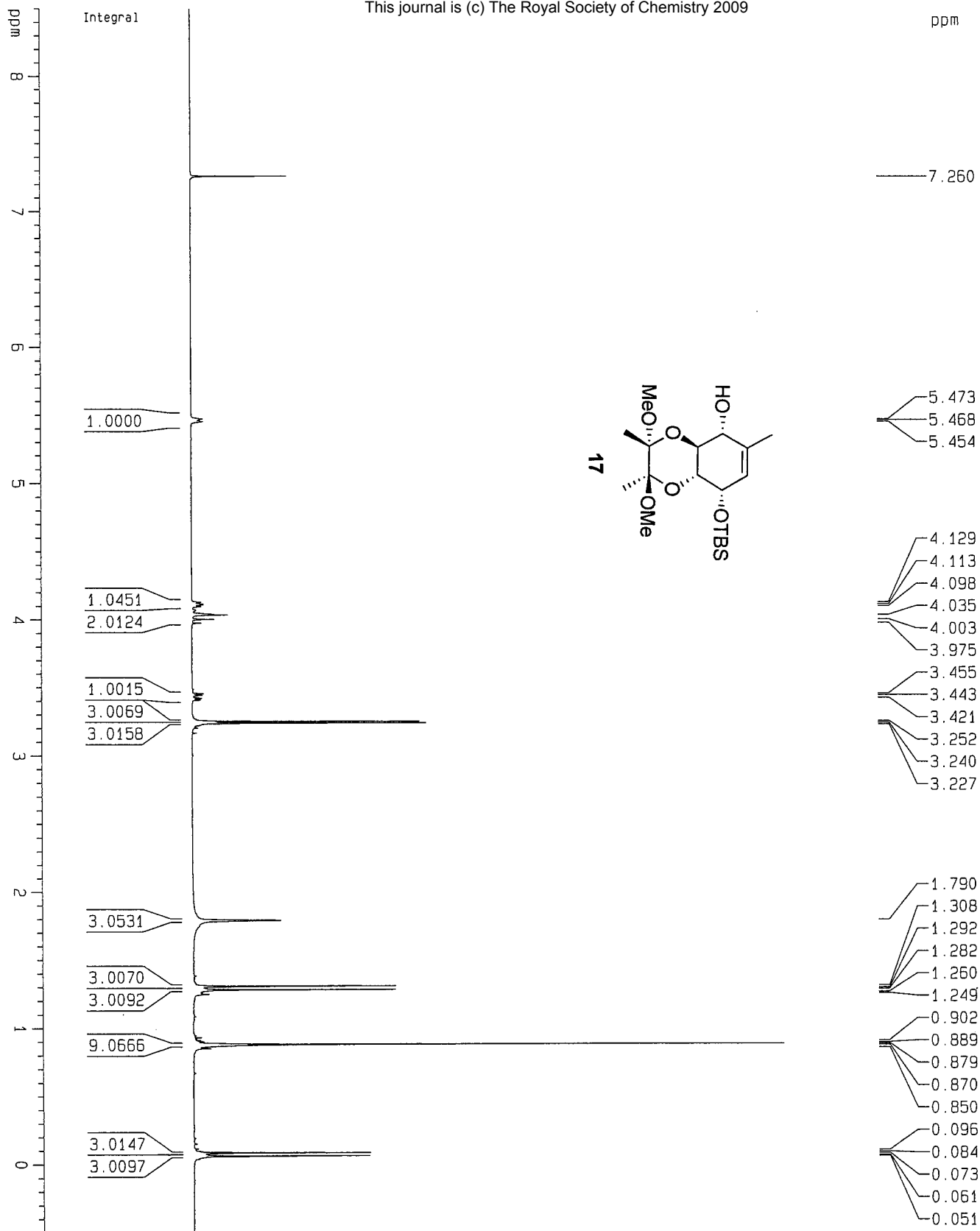
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NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SF01 75.4745111 MHz

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

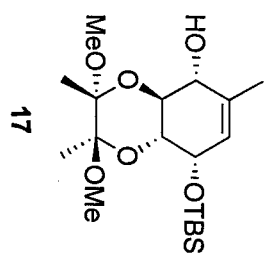
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GB 0
PC 1.40

1D NMR plot parameters

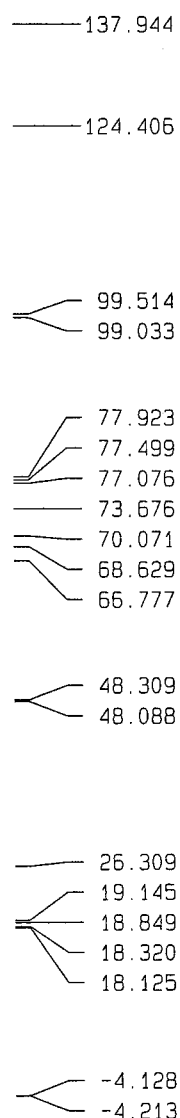
CX 23.00 cm
CY 5.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67712 Hz/cm



Current Data Parameters	
NAME	GL1521-h1
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	20070608
Time	18.12
INSTRUM	dpX300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDC13
NS	32
DS	0
SWH	5402.485 Hz
FIDRES	0.164871 Hz
AQ	3.0327284 sec
RG	362
DM	92.550 usec
DE	132.21 usec
TE	0.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWK	0.01500000 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1318000 MHz
F2 - Processing parameters	
SI	32768
SF	300.1300064 MHz
MWM	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00
1D NMR plot parameters	
CX	22.00 cm
CY	10.62 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



Current Data Parameters
NAME GL152-c13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070604
Time 19.09

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

TD 65536
SOLVENT CDCl3
NS 880

DS 0
SWH 26455.027 Hz
FIDRES 0.403672 Hz

AQ 1.2386804 sec
RG 2560.3
DM 18.900 usec

DE 5.00 usec
TE 0.0 K

D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec

MCNMRK 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 75.4677125 MHz

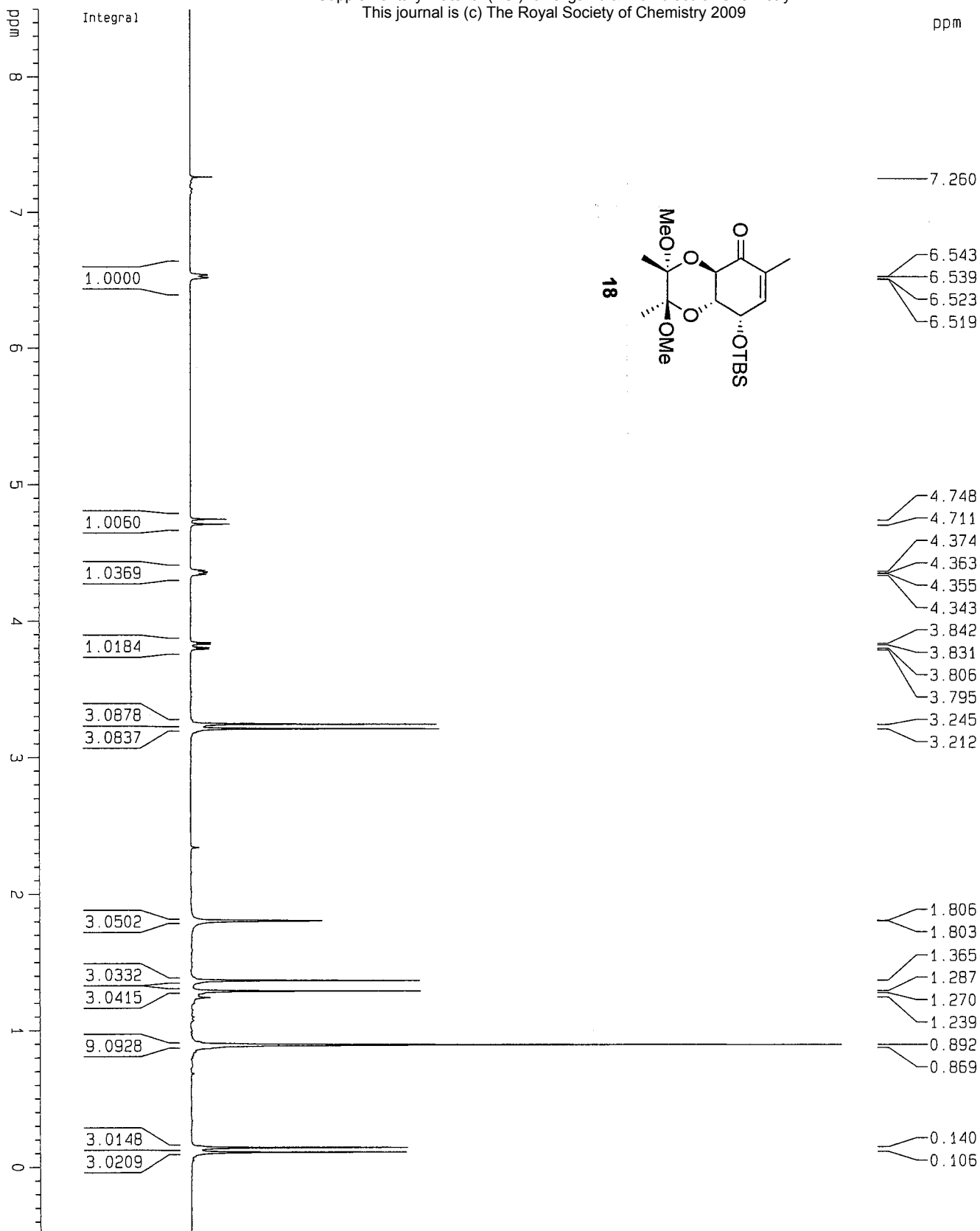
WDW EM
SSB 0
LB 2.50 Hz

GB 0
PC 1.40

===== 1D NMR plot parameters =====
CX 23.00 cm
CY 3.50 cm

F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm

F2 -754.68 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67712 Hz/cm



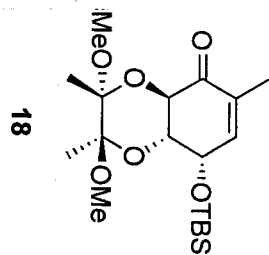
Current Data Parameters
NAME GL153-h1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070606
Time 18.23
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SMH 5402.485 Hz
FIDRES 0.164871 Hz
AQ 3.0327284 sec
RG 71.8
DM 92.550 usec
DE 132.21 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
SF01 300.1318000 MHz

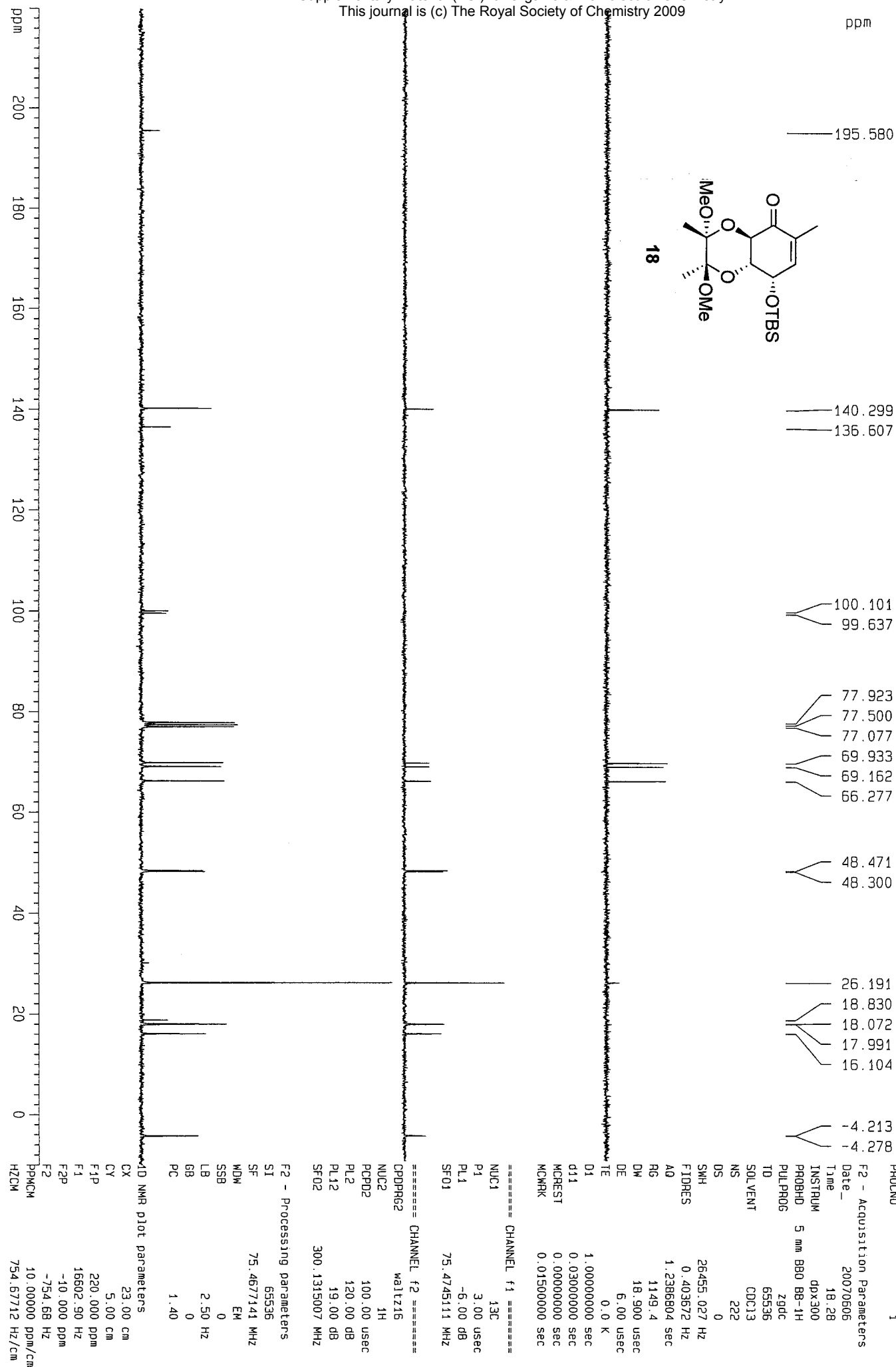
F2 - Processing parameters
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SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

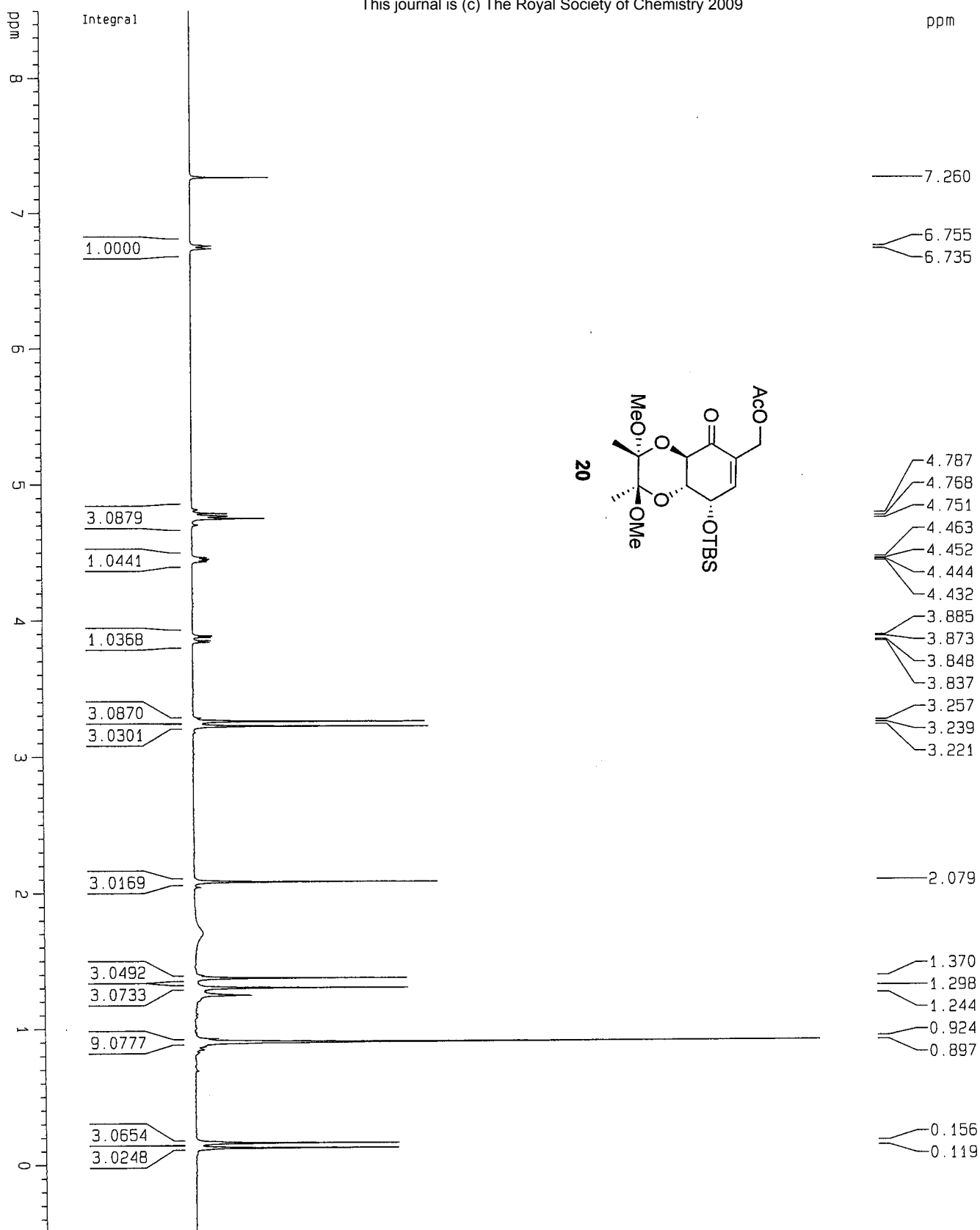
1D NMR plot parameters
CX 22.00 cm
CY 11.67 cm
F1P 8.500 ppm
F1 2551.11 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.40909 ppm/cm
HZCM 122.78047 Hz/cm



18

ppm





Current Data Parameters

NAME	GL144-h1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20070530
Time	20.16
INSTRUM	dot300
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	5402.485 Hz
FIDRES	0.164871 Hz
AQ	3.0327284 sec
R6	228.1
DW	92.550 usec
DE	132.21 usec
TE	0.0 K
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MCREST	0.00000000 sec
MCNRC	0.01500000 sec

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

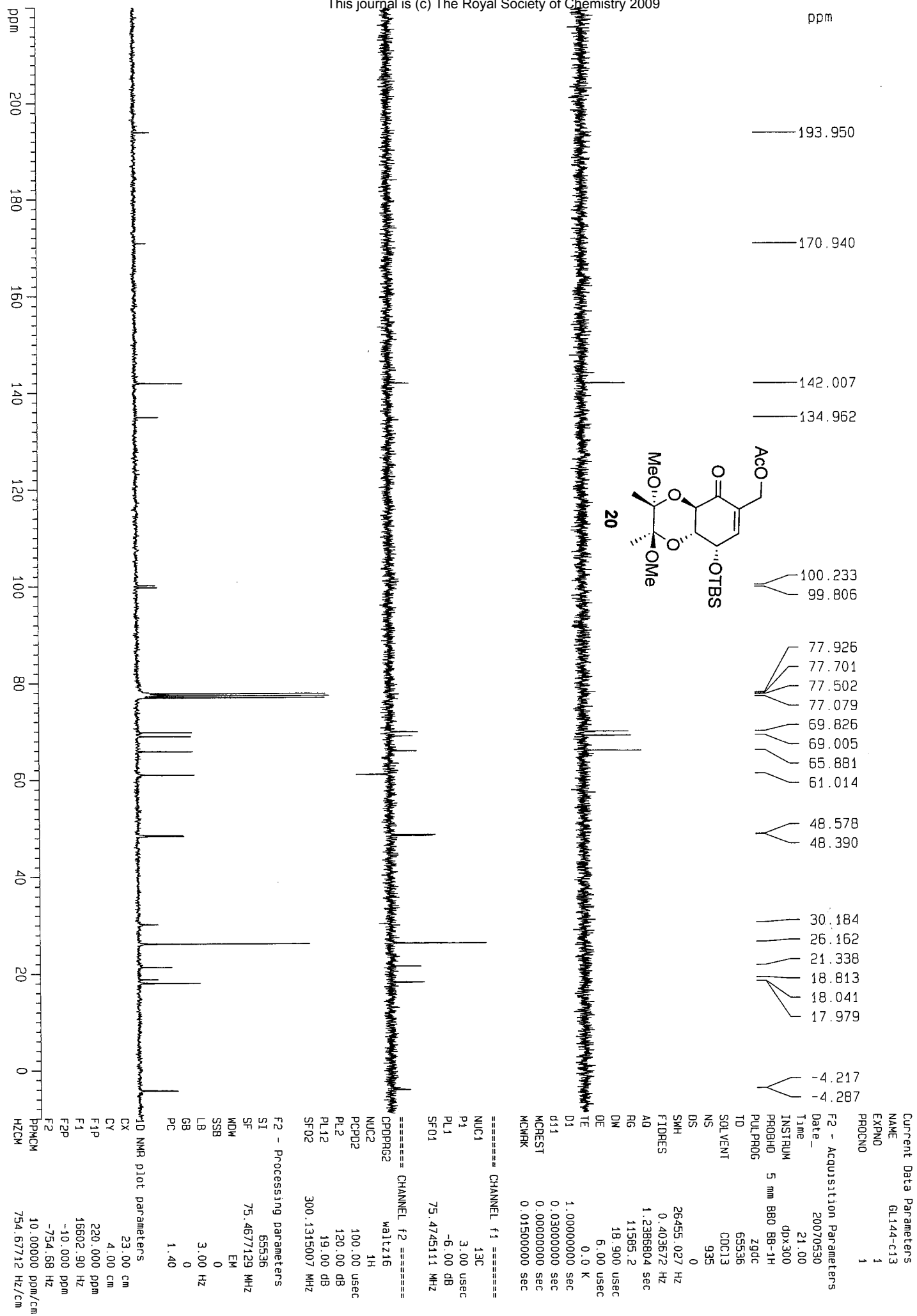
NUC1	1H
P1	5.00 usec
PL1	-2.00 dB
SFO1	300.1318000 MHz

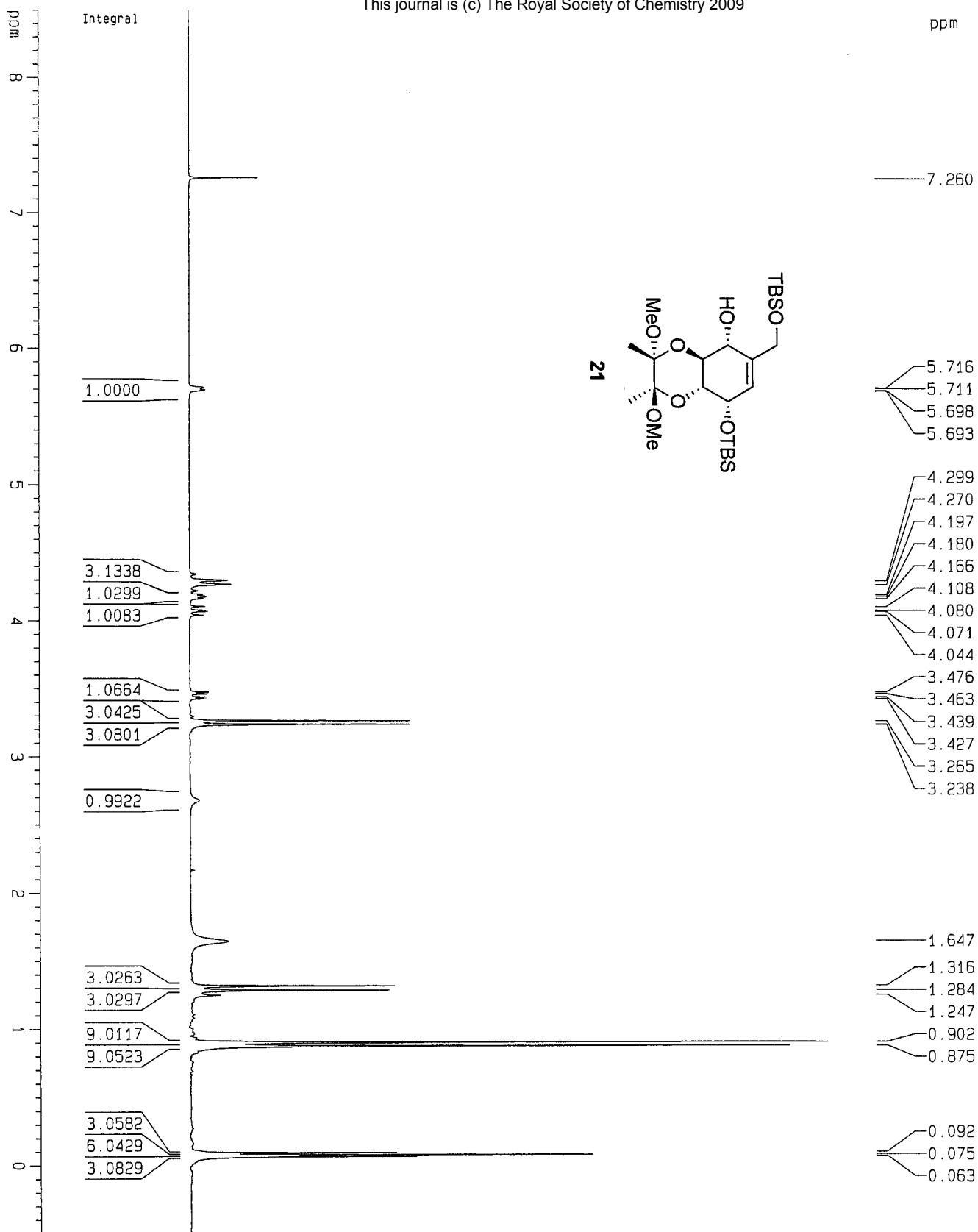
F2 - Processing parameters

SF	300.1300064 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	11.33 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





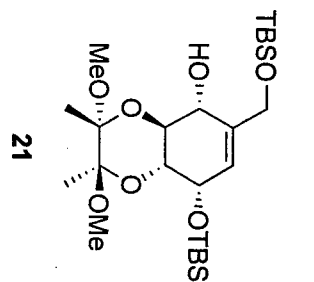
Current Data Parameters
NAME GL146-h1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070530
Time 19.22
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PROBHD 5 mm BB0 BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5402.485 Hz
FIDRES 0.164871 Hz
AQ 3.0327284 sec
RG 228.1
DE 132.21 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

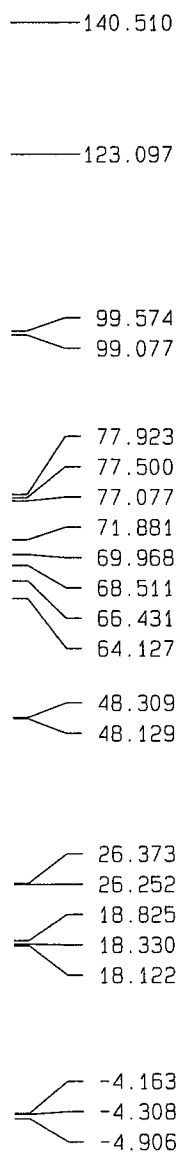
===== CHANNEL f1 =====
NUC1 1H
P1 5.00 usec
PL1 -2.00 dB
SF01 300.1318000 MHz

F2 - Processing parameters
SI 32768
SF 300.130062 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 11.33 cm
F1P 8.500 ppm
F1 2551.10 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.40509 ppm/cm
HZCM 122.78046 Hz/cm



ppm



Current Data Parameters
NAME GL146-c13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070530
Time 19.49

INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT COC13
NS 921
DS 0
SWH 26455.027 Hz
FIDRES 0.403672 Hz
AQ 1.2366804 sec
RG 4597.6
DM 18.900 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

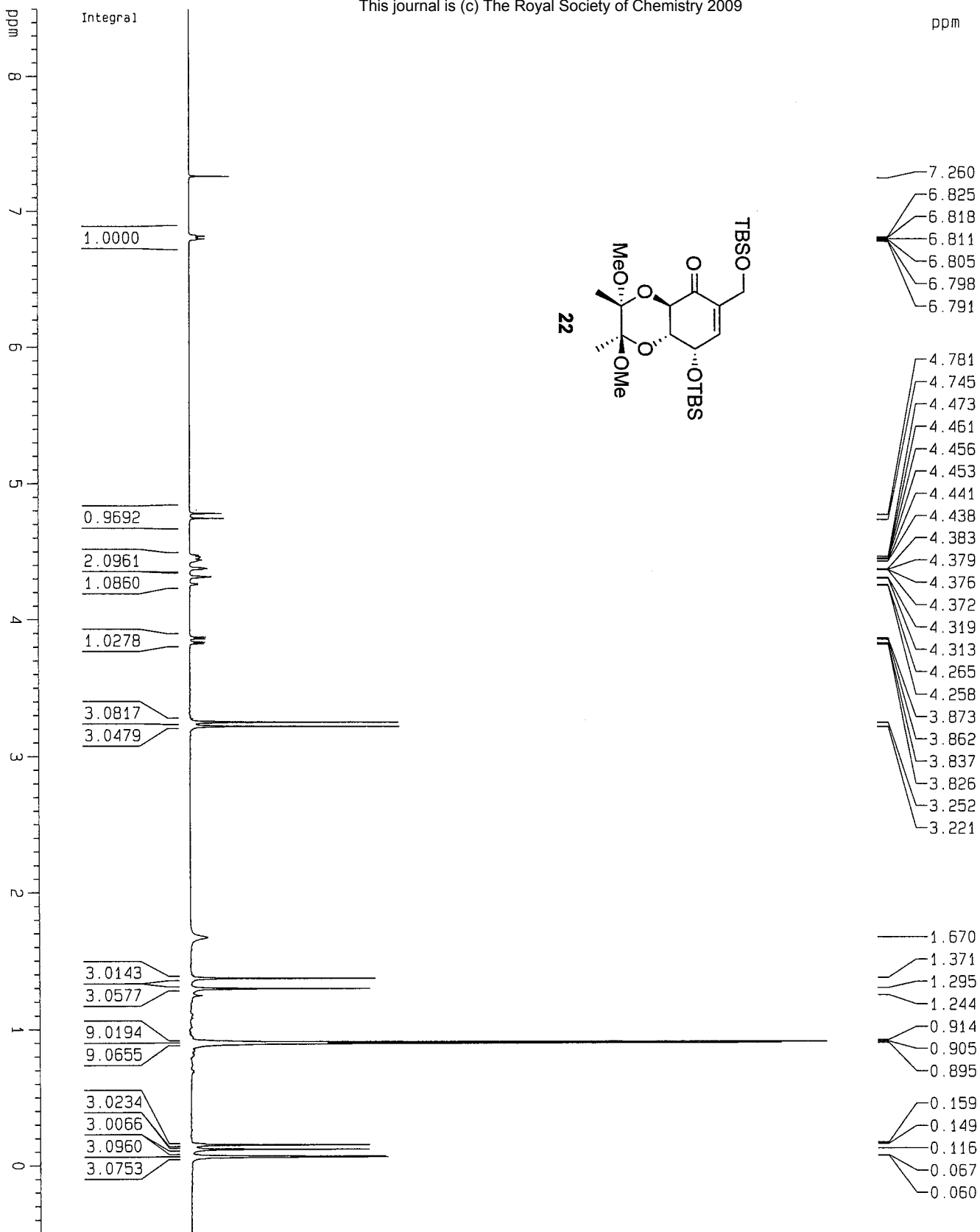
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4677125 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

ID NMR plot parameters

CX 23.00 cm
CY 4.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67712 Hz/cm





Current Data Parameters
NAME 6L147-h1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070530
Time 18.29
INSTRUM dpx300
PROBHD 5 mm BB0 BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SMH 5402.485 Hz
FIDRES 0.164871 Hz
AQ 3.0327284 sec
RG 114
DW 92.550 usec
DE 132.21 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
SF01 300.1318000 MHz

F2 - Processing parameters
SI 32768
SF 300.130062 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 11.48 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

