

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.

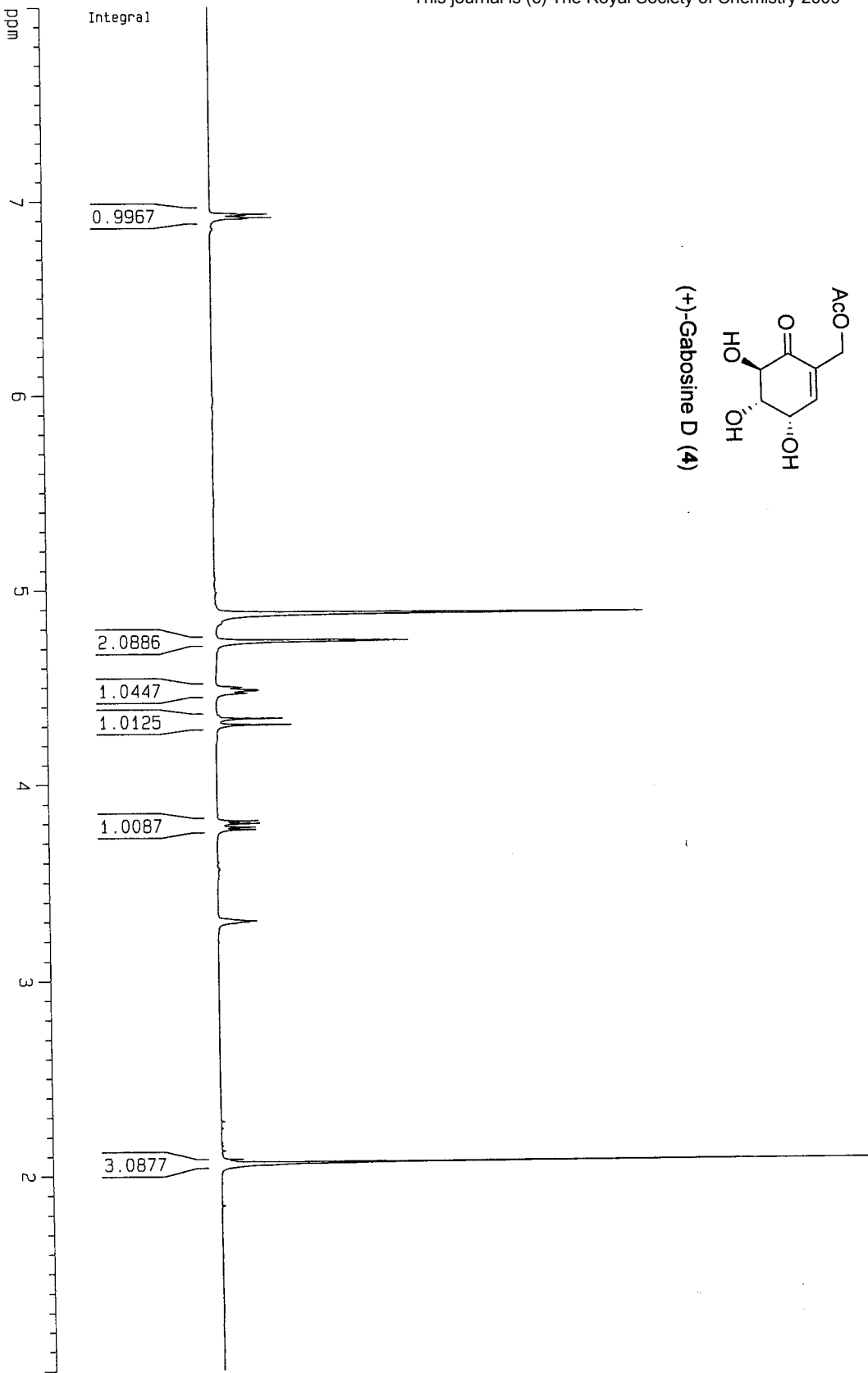
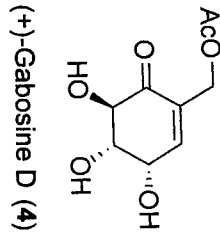
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ppm

6.935
6.930
6.926
6.917
6.913
6.908

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
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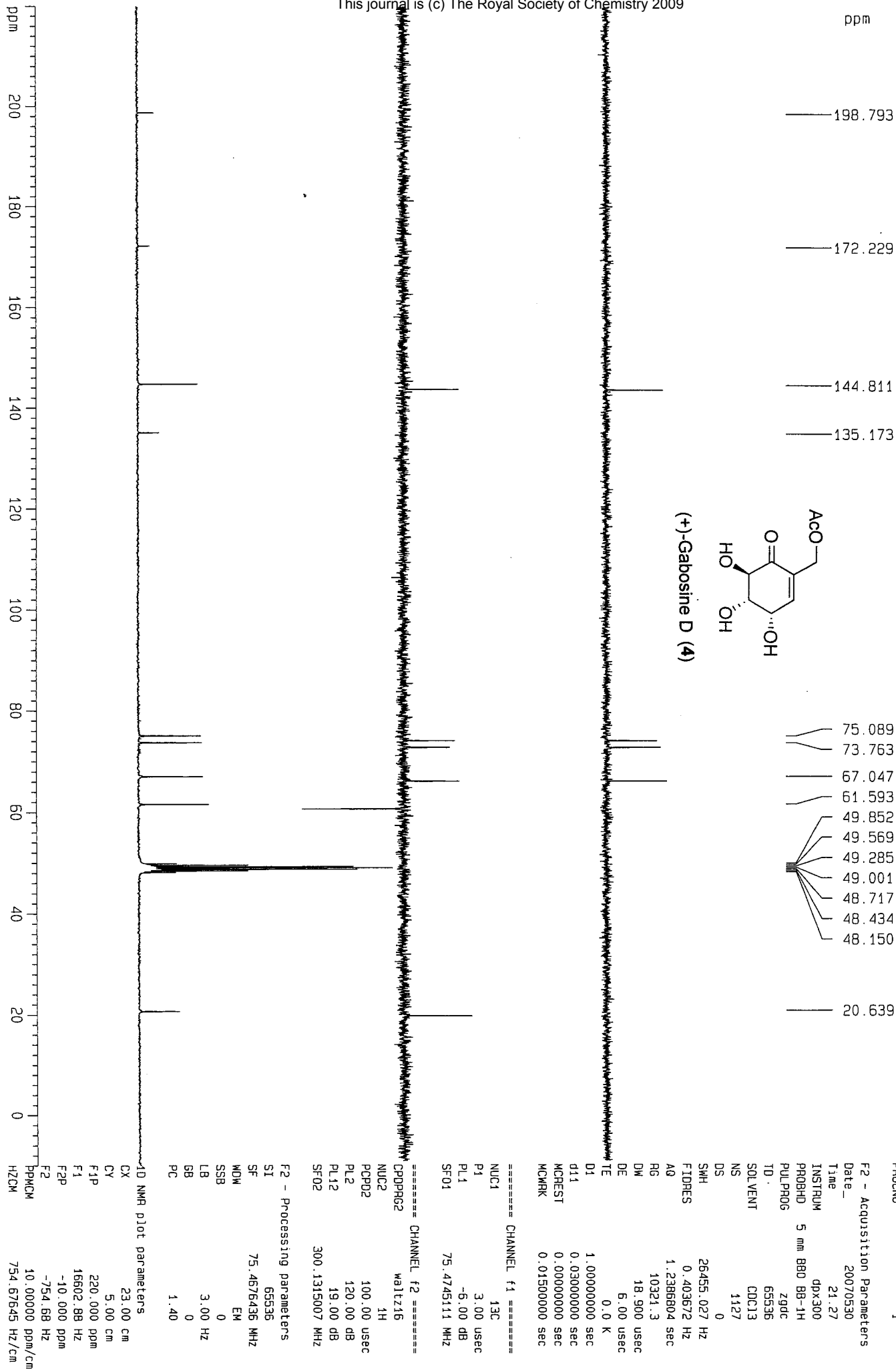
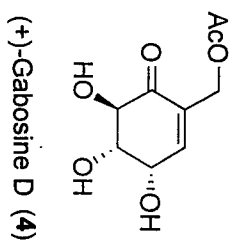
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INSTRUM dpx300
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PULPROG zg
TD 32768
SOLVENT MeOD
NS 8
DS 0
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FIDRES 0.164871 Hz
AQ 3.0327284 sec
RG 228.1
DW 92.550 usec
DE 132.21 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWPK 0.01500000 sec

==== CHANNEL f1 =====
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SF01 300.1318000 MHz

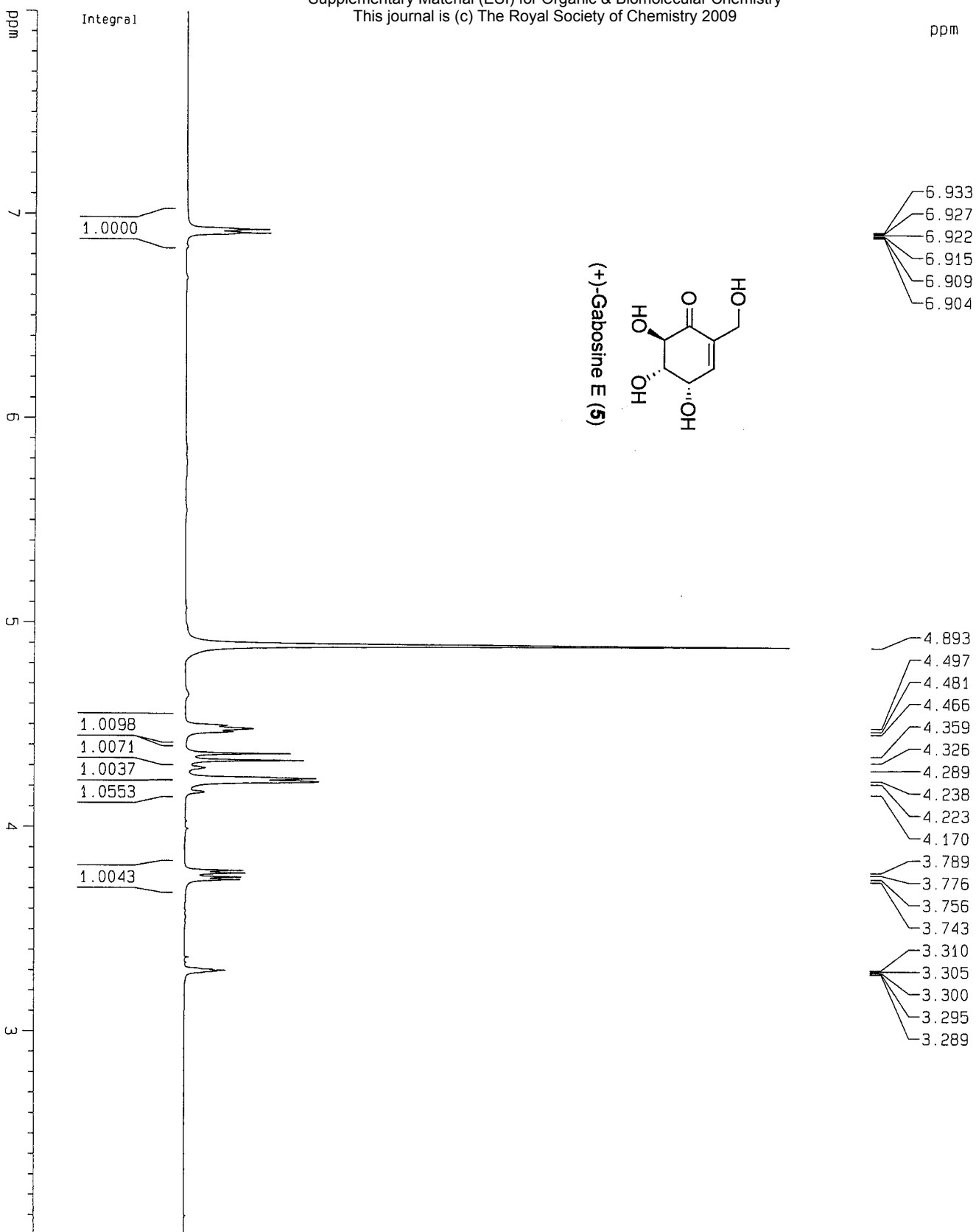
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PC 1.00

1D NMR plot parameters
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CY 10.38 cm
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F1 2401.04 Hz
F2P 1.000 ppm
F2 300.13 Hz
PPMCM 0.31818 ppm/cm
HZCM 95.49591 Hz/cm

Solvent: CD₃OD



Solvent: CD₃OD



Current Data Parameters
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 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 Time 18.45

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

TD 32768
 SOLVENT MeOD
 NS 8

DS 0
 SMH 5402.485 Hz
 FIDRES 0.164871 Hz

RG 181
 DE 132.21 usec
 TE 0.0 K

D1 1.00000000 sec
 ACREST 0.00000000 sec
 MCWRR 0.01500000 sec

===== CHANNEL f1 =====
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 P1 5.00 usec
 PL1 -2.00 dB
 SFO1 300.1318000 MHz

F2 - Processing parameters
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 SF 300.1300076 MHz
 WDM EM

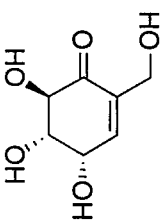
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1D NMR plot parameters
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 CY 10.75 cm
 F1P 8.000 ppm

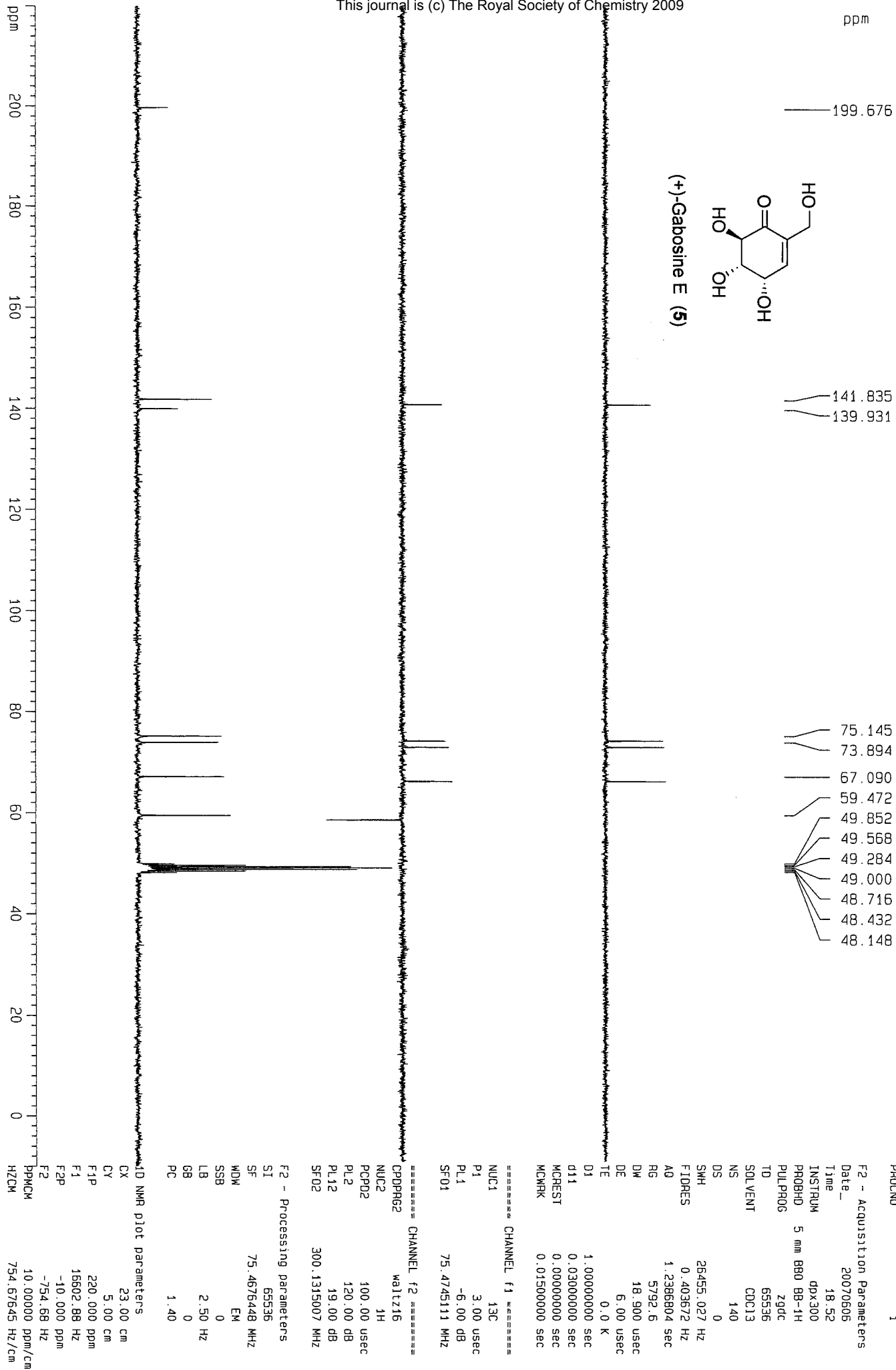
F1 2401.04 Hz
 F2P 2.000 ppm
 F2 600.26 Hz

PPMCM 0.27273 ppm/cm
 HZCM 81.85364 Hz/cm

Solvent: CD₃OD



(+)-Gabosine E (5)



Current Data Parameters
 NAME GL148-c13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070606
 Time 18.52

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

TD 65536
 SOLVENT CDCl3
 NS 140

DS 0
 SWH 26455.027 Hz
 FIDRES 0.403672 Hz

AQ 1.2386804 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
 MCWRR 0.01500000 sec

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 NUC1 13C
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 PL1 -6.00 dB
 SFO1 75.4745111 MHz

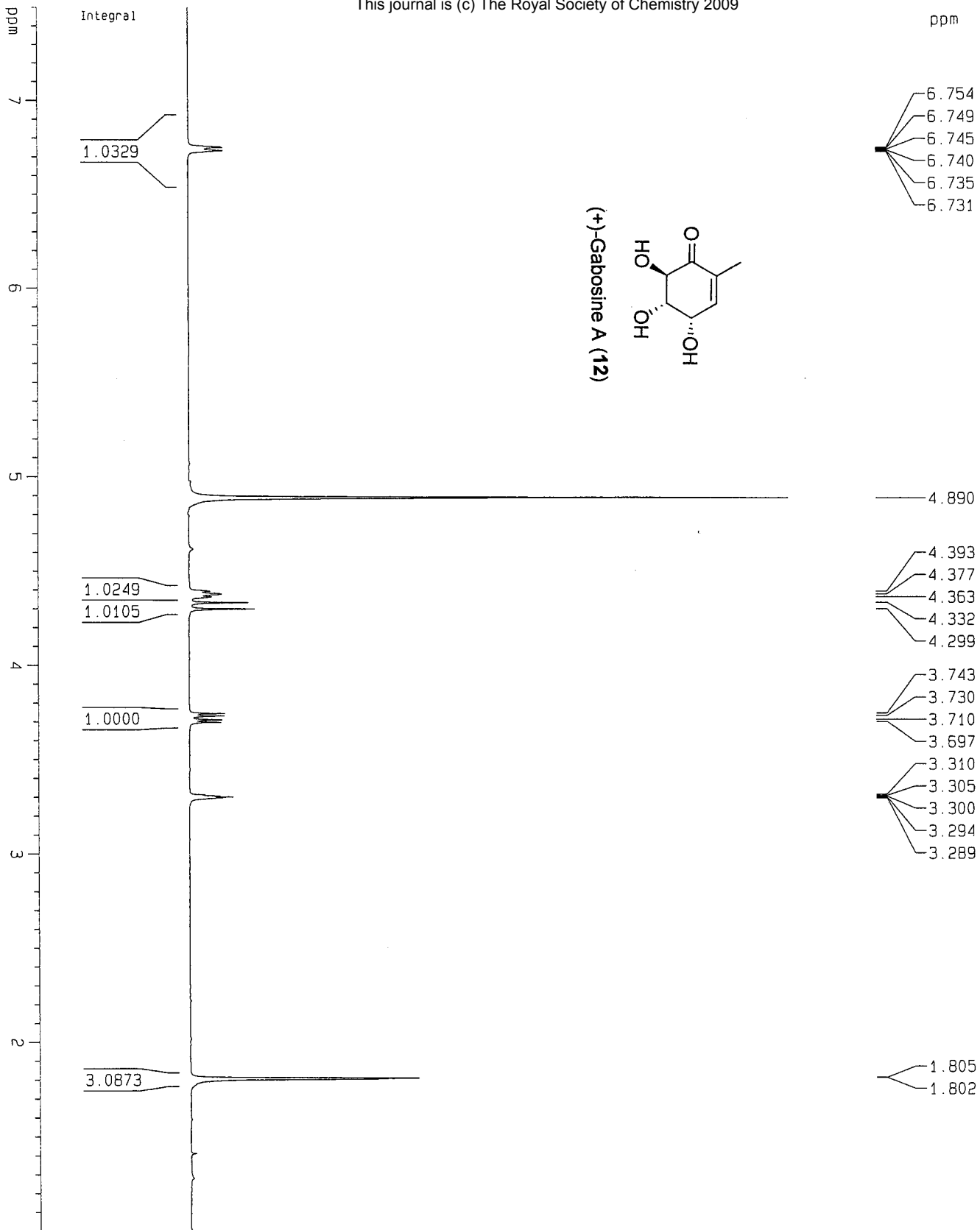
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 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SFO2 300.1315007 MHz

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
 CX 23.00 cm
 CY 5.00 cm
 F1P 220.000 ppm
 F1 16502.88 Hz
 F2P -10.000 ppm
 F2 -754.68 Hz

==== 1D NMR Plot parameters
 PPMCM 10.00000 ppm/cm
 HZCM 754.67645 Hz/cm

Solvent: CD₃OD



Current Data Parameters
 NAME GL154-h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070709
 Time 10.17

INSTRUM ddx300
 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
 DW 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
 SFO1 300.1318000 MHz

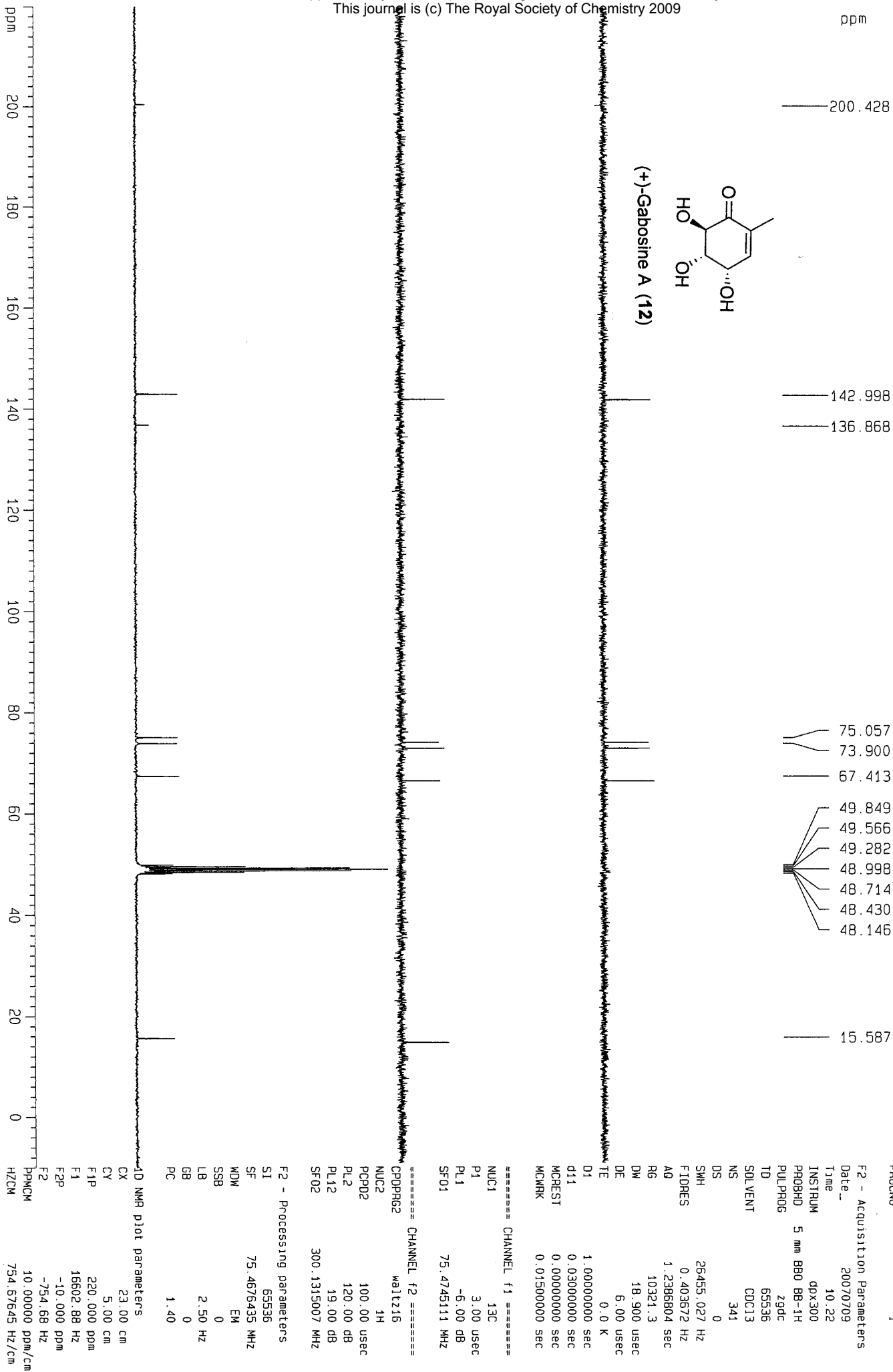
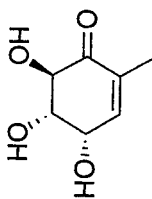
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 WDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

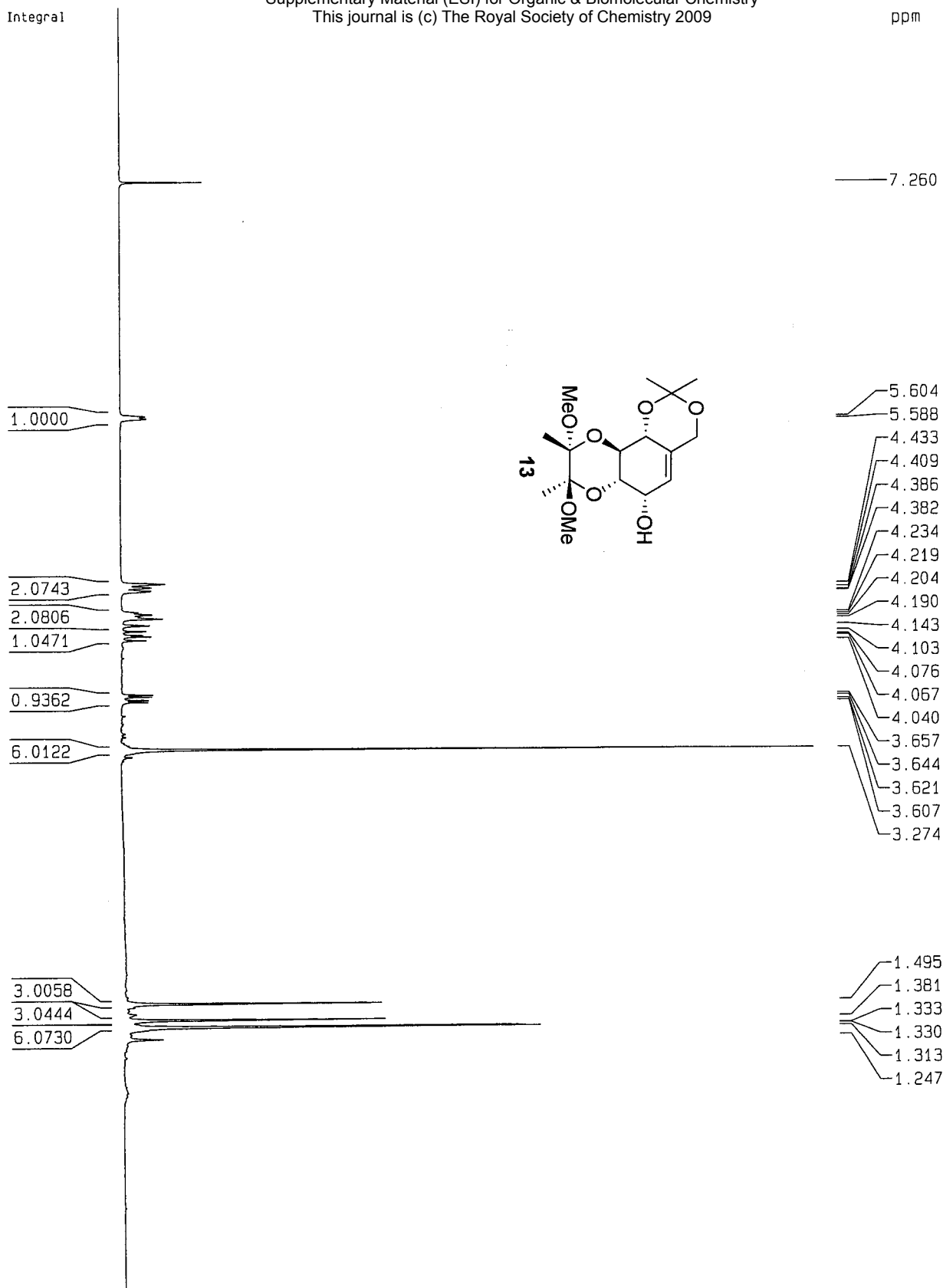
CX 22.00 cm
 CY 10.66 cm
 F1P 7.500 ppm
 F1 2250.98 Hz
 F2P 1.000 ppm
 F2 300.13 Hz
 PPMCM 0.29545 ppm/cm
 HZCM 88.67478 Hz/cm

Solvent: CD₃OD



ppm

Integral



Current Data Parameters
 NAME GL44data
 EXPNO 1
 PROCNO 2

F2 - Acquisition Parameters
 Date_ 20050521
 Time 13.29

INSTRUM ddx300
 PROBHD 5 mm BBO BB-1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 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 296.2 K
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 MCREST 0.00000000 sec
 MCMRK 0.01500000 sec

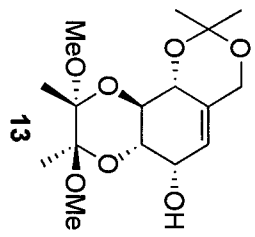
==== CHANNEL f1 =====
 NUC1 1H

P1 5.00 usec
 PL1 -2.00 dB
 SF01 300.1312000 MHz

F2 - Processing parameters
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 SF 300.1300063 MHz
 WDM EM

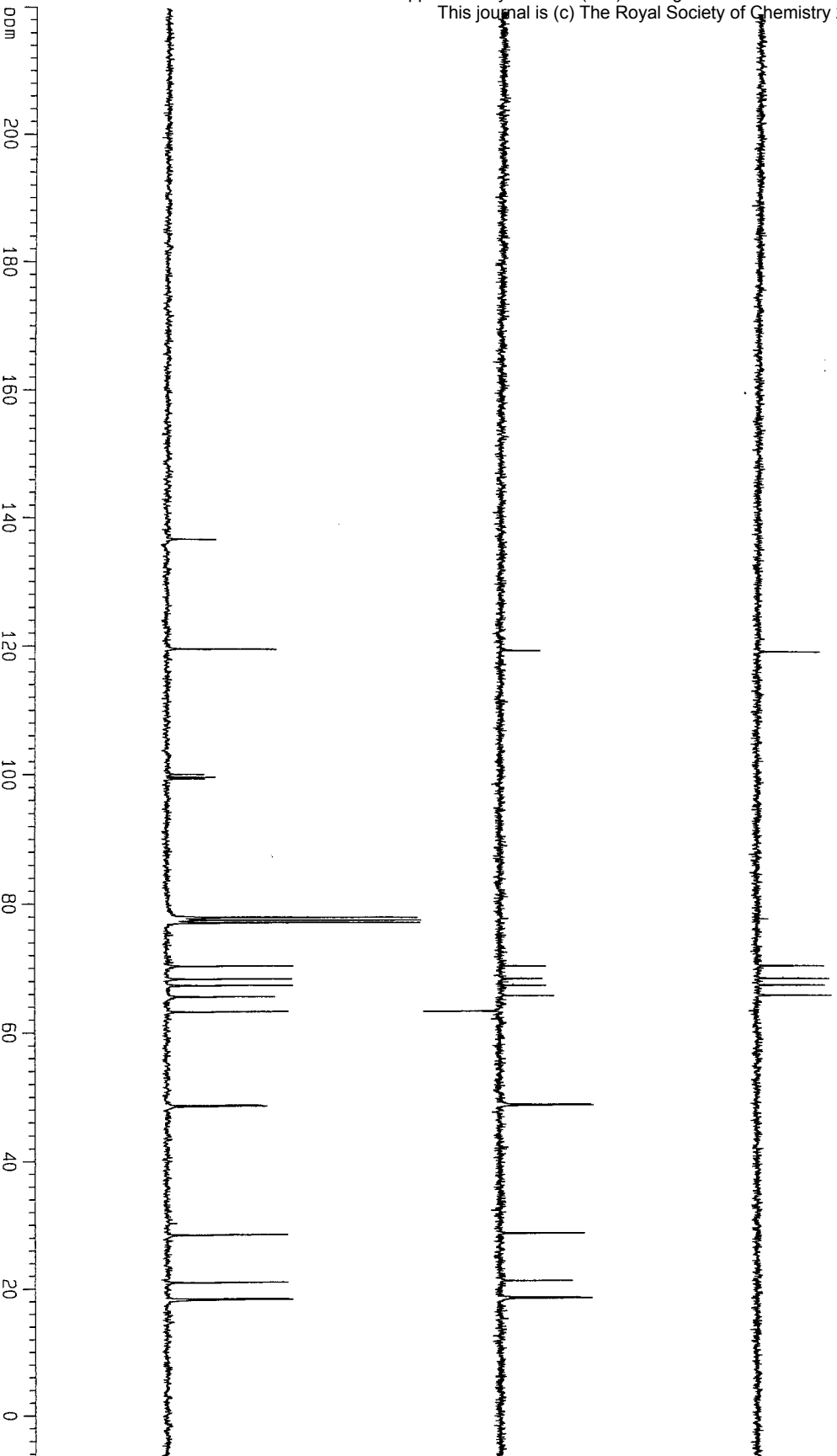
SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 11.81 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

- 136.766
- 119.644
- 100.081
- 99.662
- 99.391
- 77.924
- 77.501
- 77.077
- 70.289
- 68.290
- 67.278
- 65.553
- 63.197
- 48.575
- 48.433
- 28.310
- 20.834
- 18.247
- 18.082



Current Data Parameters
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 EXPNO 1
 PROCNO 2

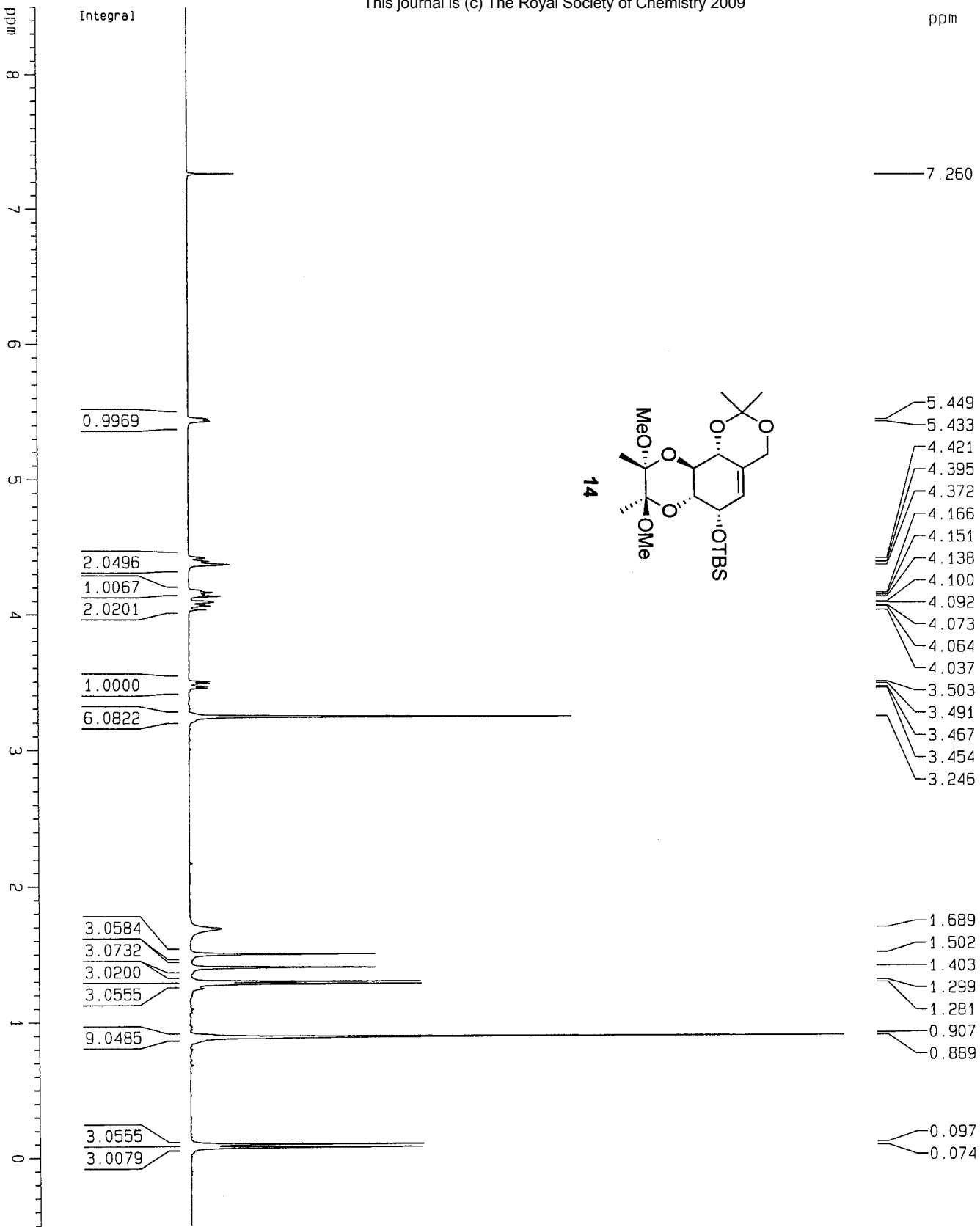
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 INSTRUM dpx300
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 882
 DS 0
 SMH 26455.027 Hz
 FIDRES 0.403672 Hz
 AQ 1.2386804 sec
 RG 2896.3
 DW 18.900 usec
 DE 6.00 usec
 TE 297.2 K

===== CHANNEL f1 =====
 NUC1 13C
 P1 3.00 usec
 PL1 -6.00 dB
 SFO1 75.4745111 MHz
 MCREST 0.0000000 sec
 MCMRK 0.01500000 sec

===== 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
 WDM 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
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EXPNO 1
PROCNO 1

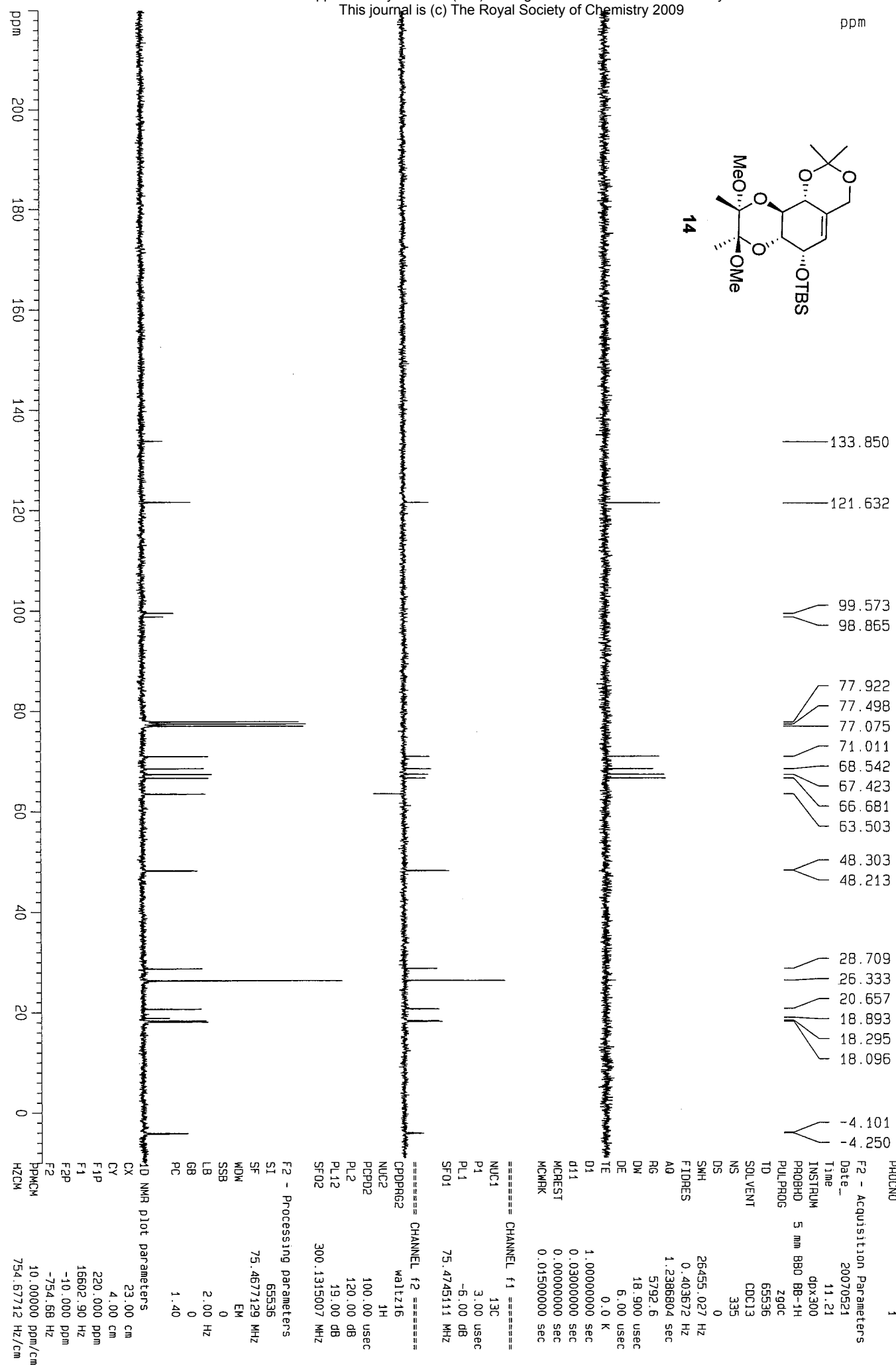
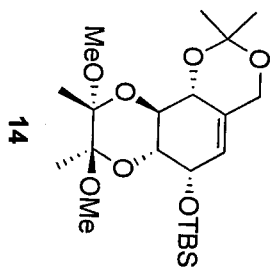
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Time 11.14

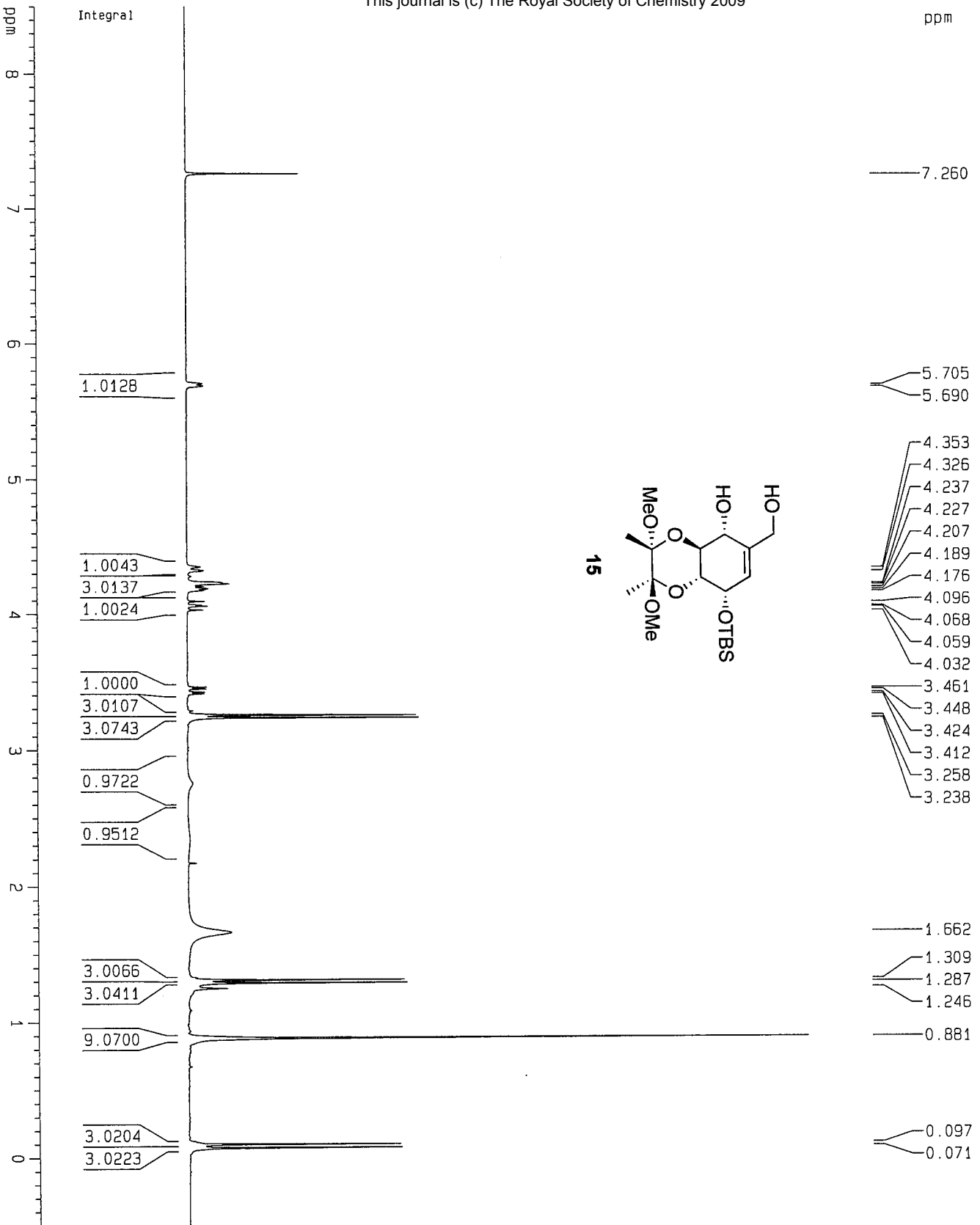
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SMH 4803.074 Hz
FIDRES 0.146578 Hz
AQ 3.4111989 sec
RG 128
DW 104.100 usec
DE 148.71 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWPK 0.01500000 sec

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

F2 - Processing parameters
SI 32768
SF 300.1300050 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 -150.07 Hz
PPMCM 0.40909 ppm/cm
HZCM 122.78046 Hz/cm





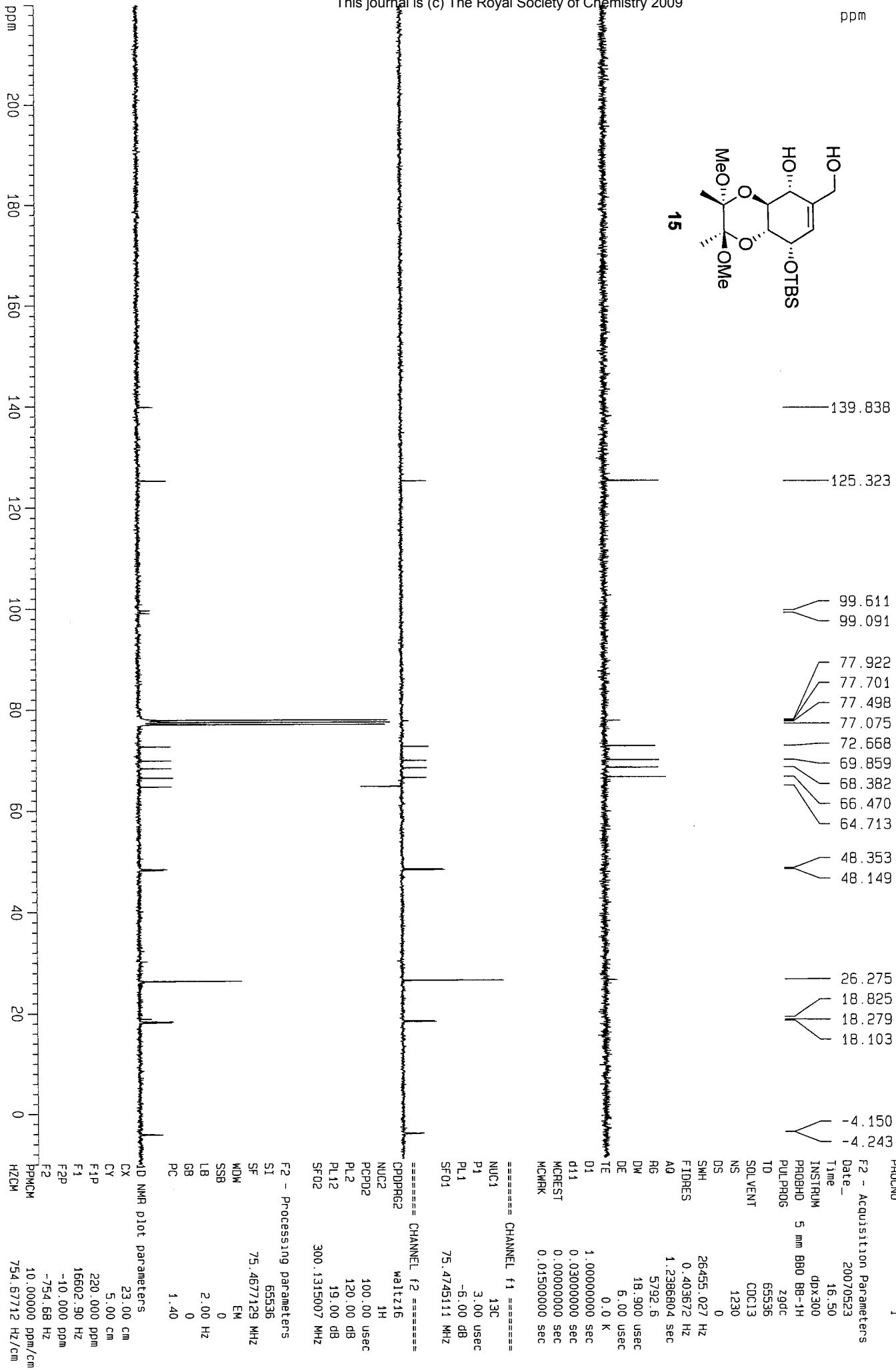
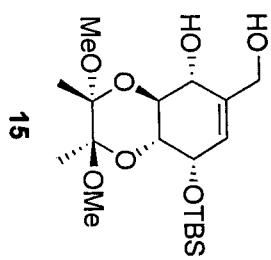
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 NAME GL142-h1
 EXPNO 1
 PROCNO 1

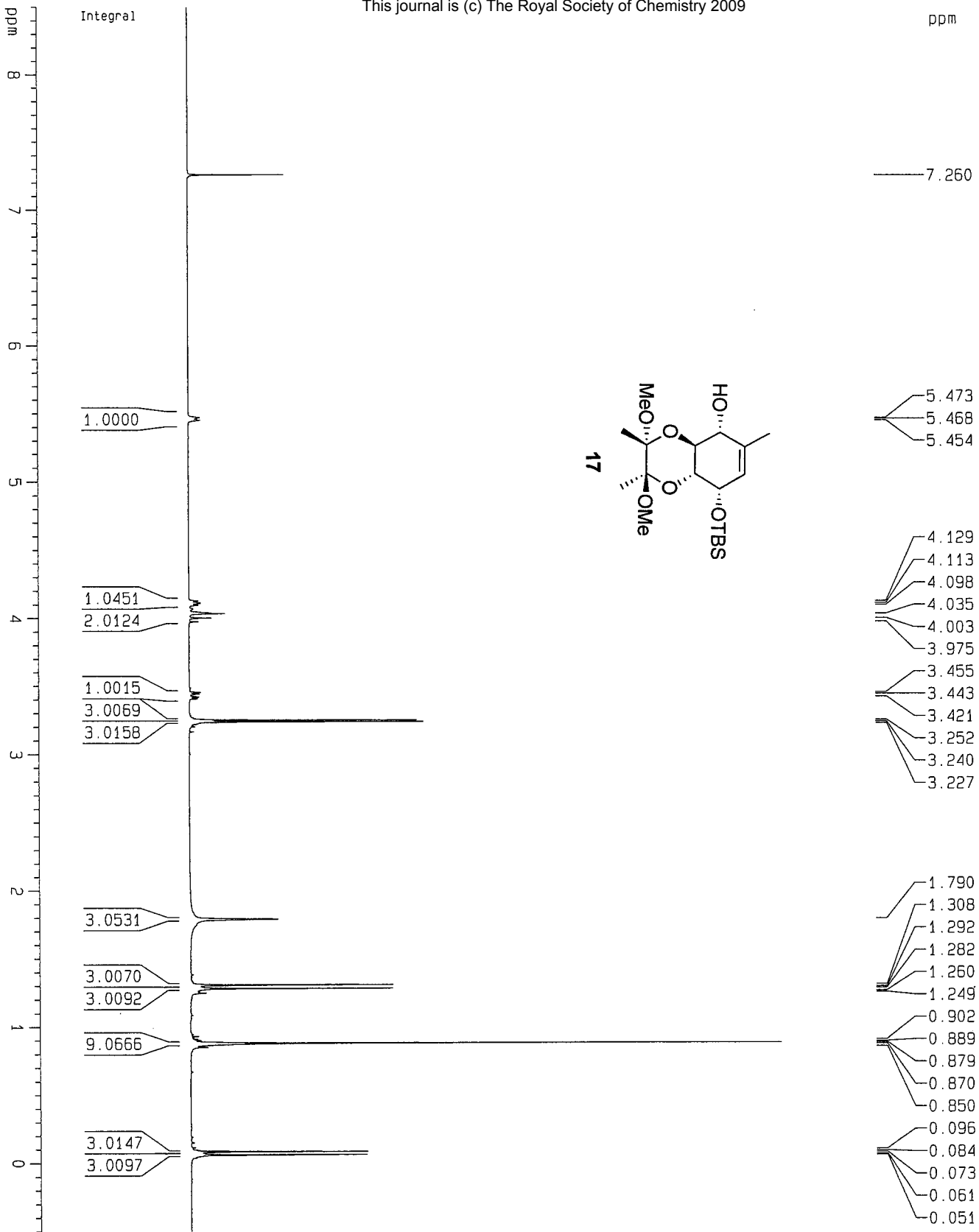
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 Time 16.02
 INSTRUM dpX300
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 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 4803.074 Hz
 FIDRES 0.146578 Hz
 AQ 3.4111989 sec
 RG 362
 DW 104.100 usec
 DE 148.71 usec
 TE 0.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

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

F2 - Processing parameters
 SI 32768
 SF 300.130063 MHz
 MDW 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





Current Data Parameters
 NAME 6L152j-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 CDCl3
 NS 32
 DS 0

SWH 5402.485 Hz
 FIDRES 0.164871 Hz
 AQ 3.0327284 sec
 RG 362

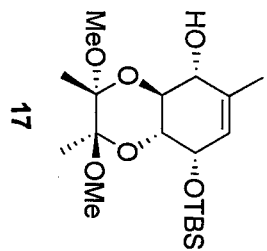
DW 92.550 usec
 DE 132.21 usec
 TE 0.0 K

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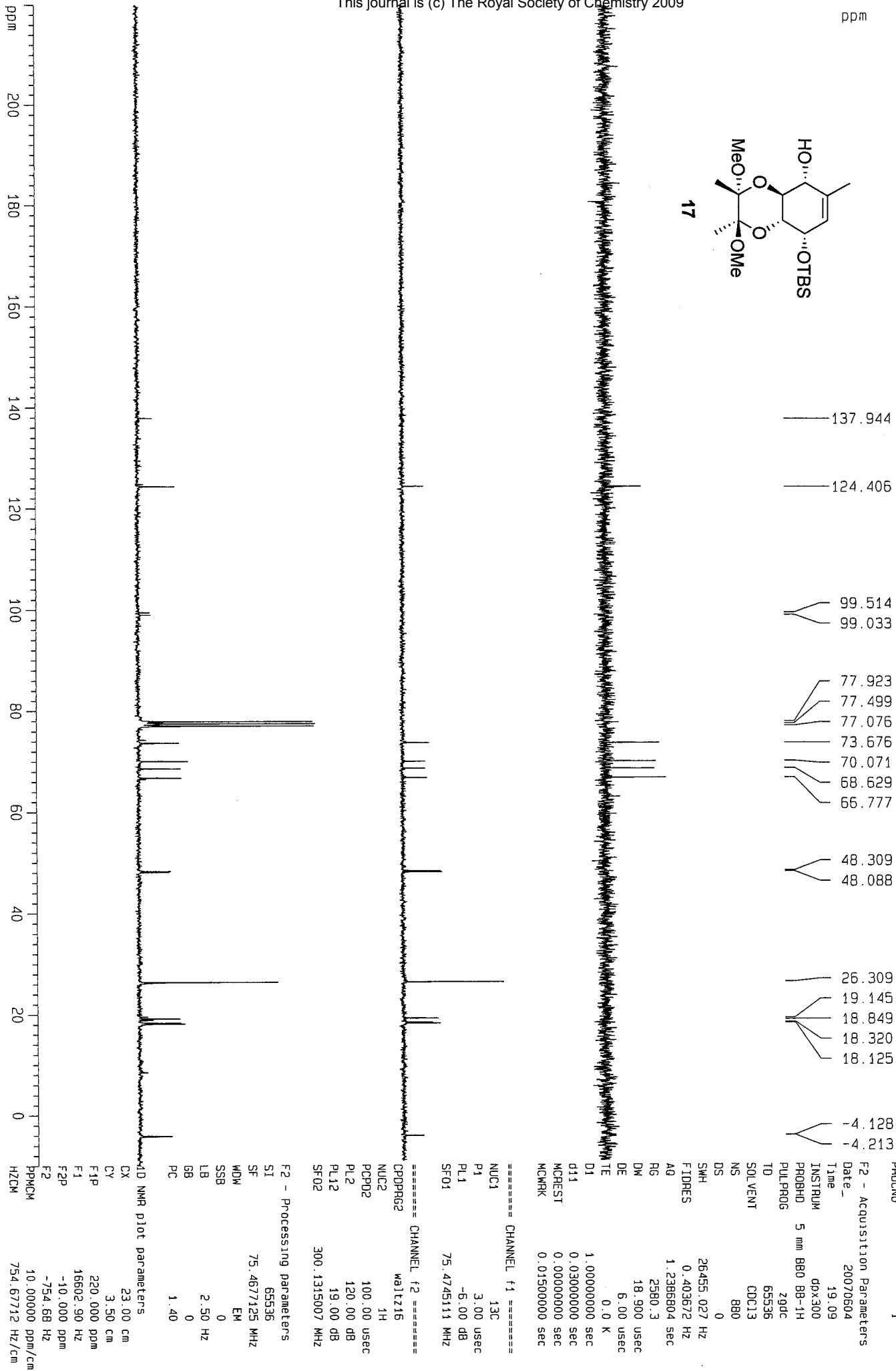
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 SFO1 300.1318000 MHz

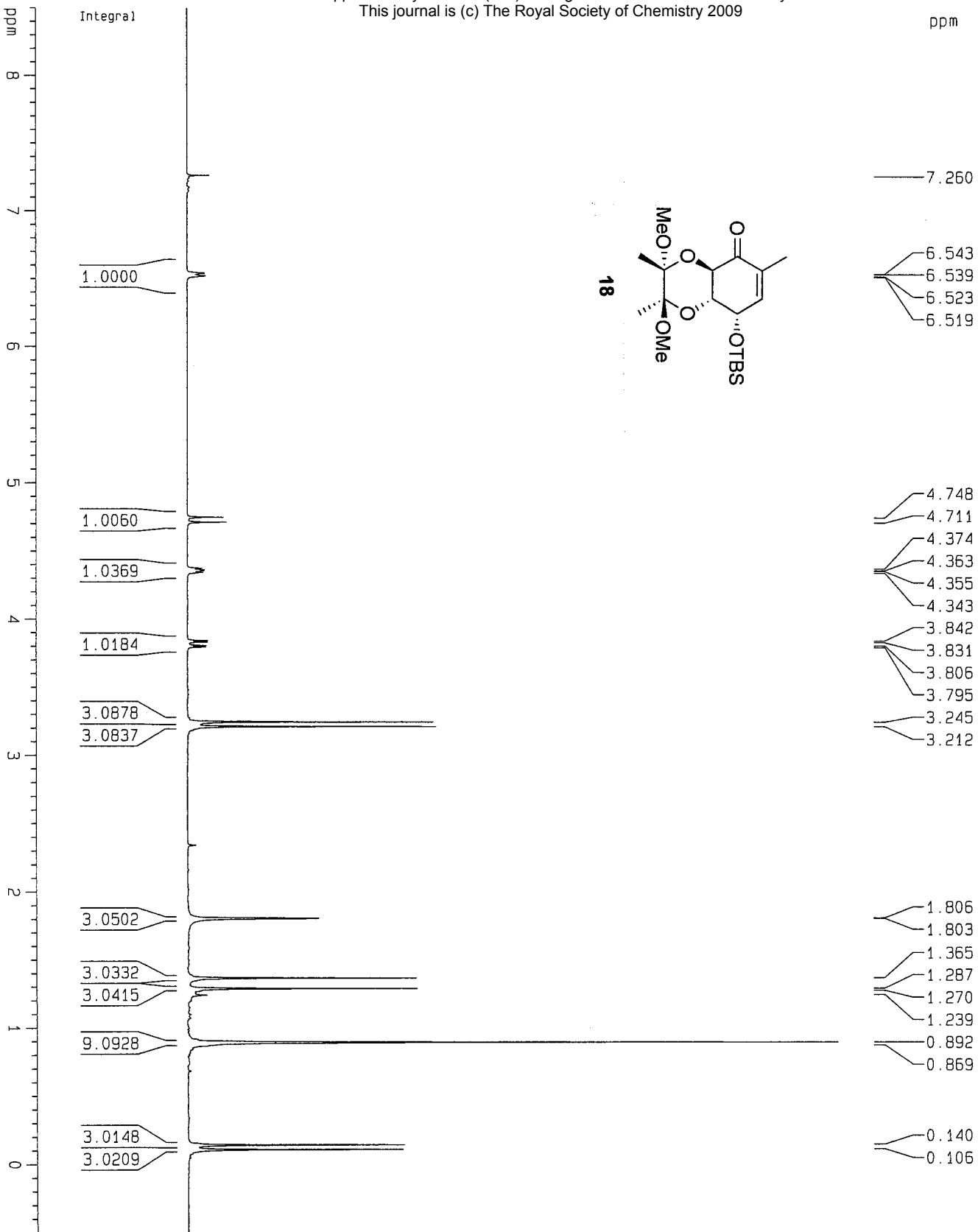
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 LB 0.30 Hz
 GB 0
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1D NMR plot parameters
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 CY 10.62 cm
 F1p 8.500 ppm
 F1 2551.10 Hz
 F2p -0.500 ppm
 F2 -150.07 Hz
 PPMCM 0.40909 ppm/cm
 HZCM 122.78046 Hz/cm



ppm





Current Data Parameters
 NAME G1153-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
 MCNPK 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
 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

