

Electronic Supplementary Information (Part 2)

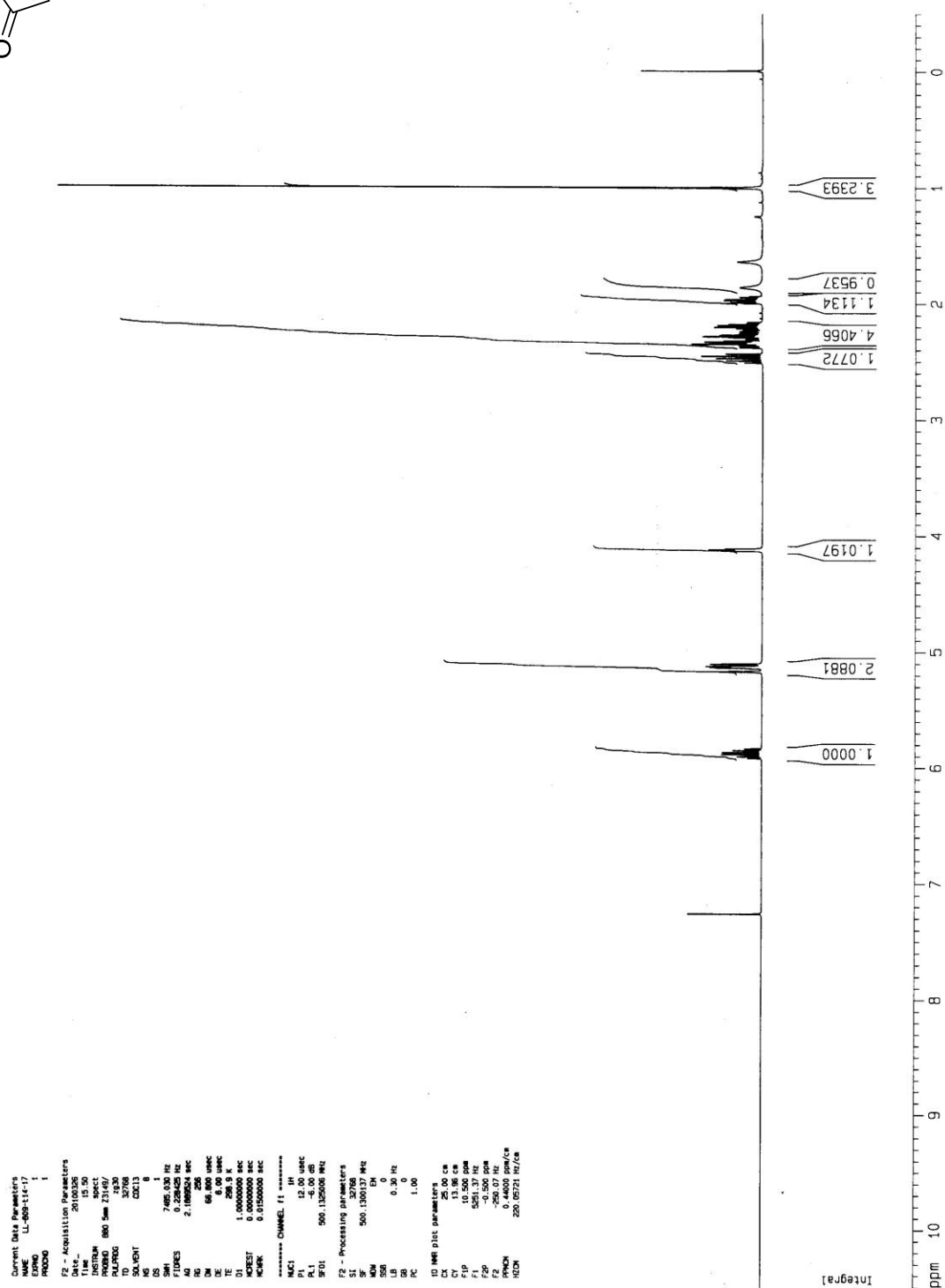
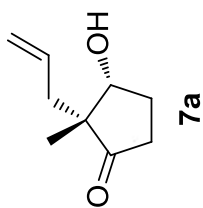
An Expeditious Asymmetric Synthesis of the Pentacyclic Core of the Cortistatins by an Intramolecular (4+3) Cycloaddition

Lok Lok Liu, Pauline Chiu*

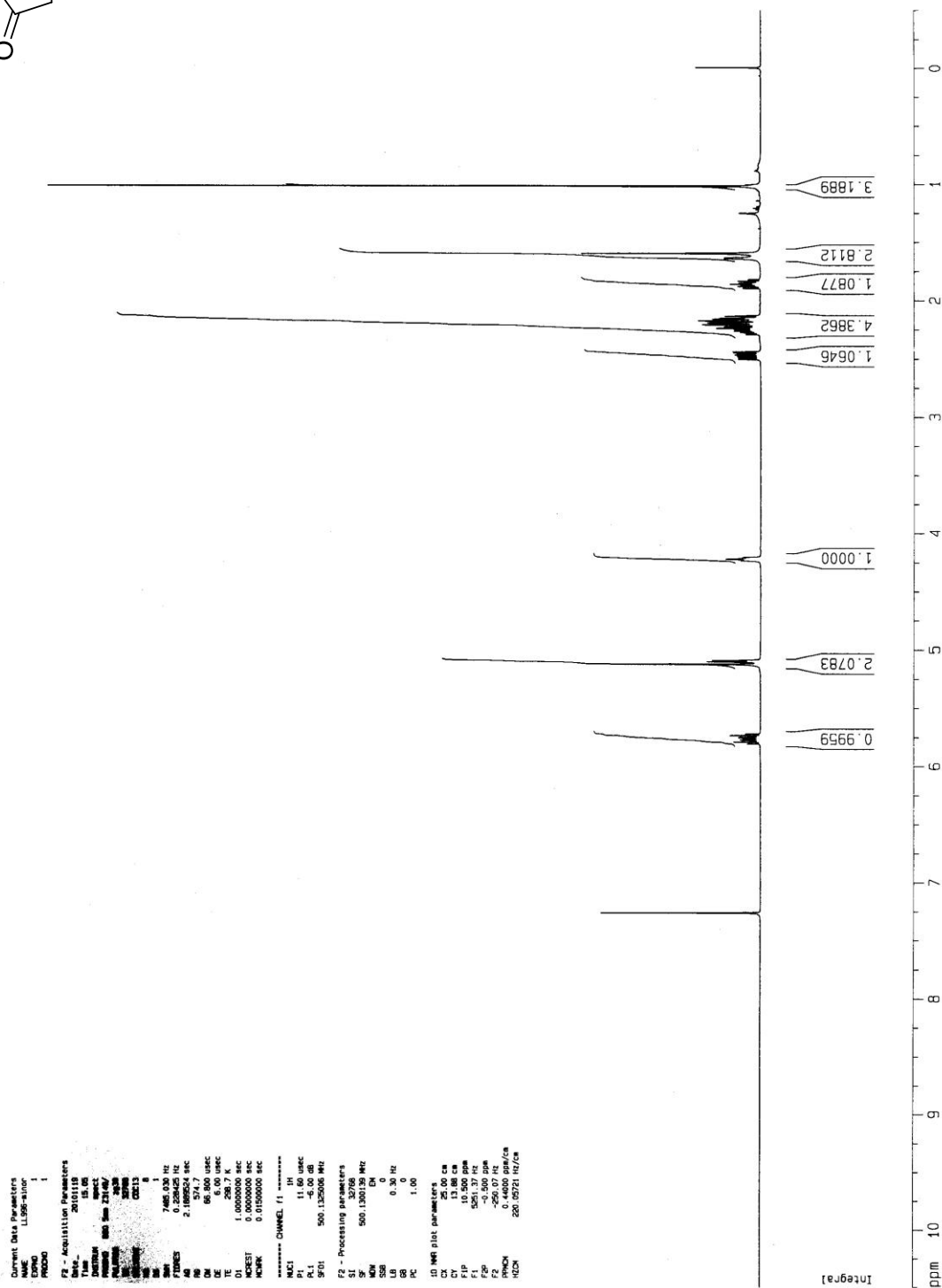
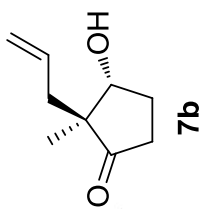
Department of Chemistry, The University of Hong Kong, Pokfulam Road, Hong Kong, P. R. China
pchiu@hku.hk

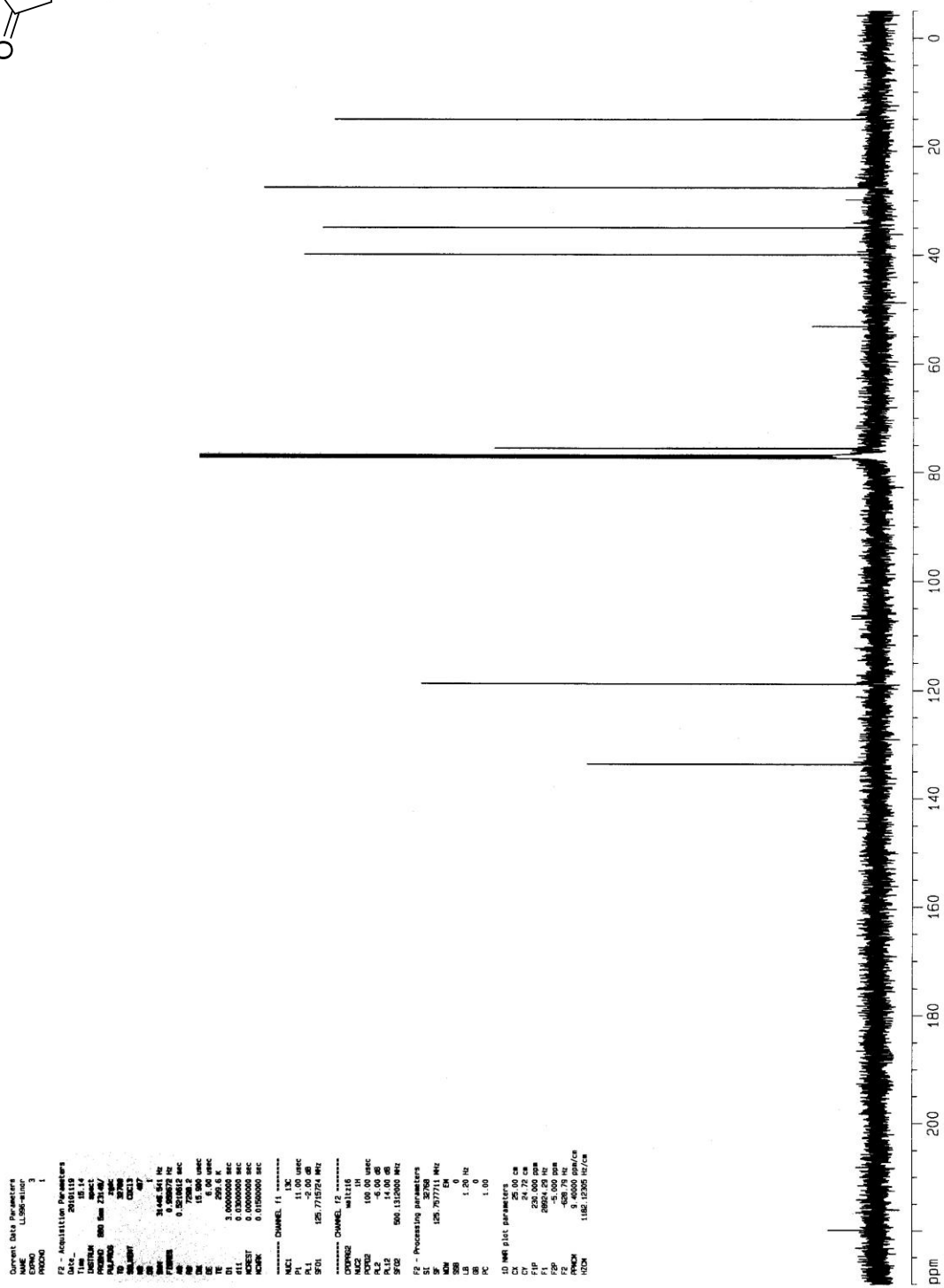
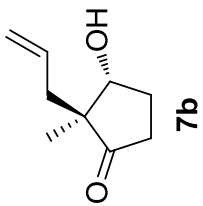
Contents

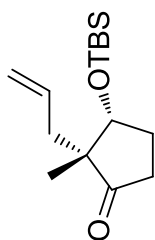
¹ H and ¹³ C NMR spectra of all new compounds.....	S16-45
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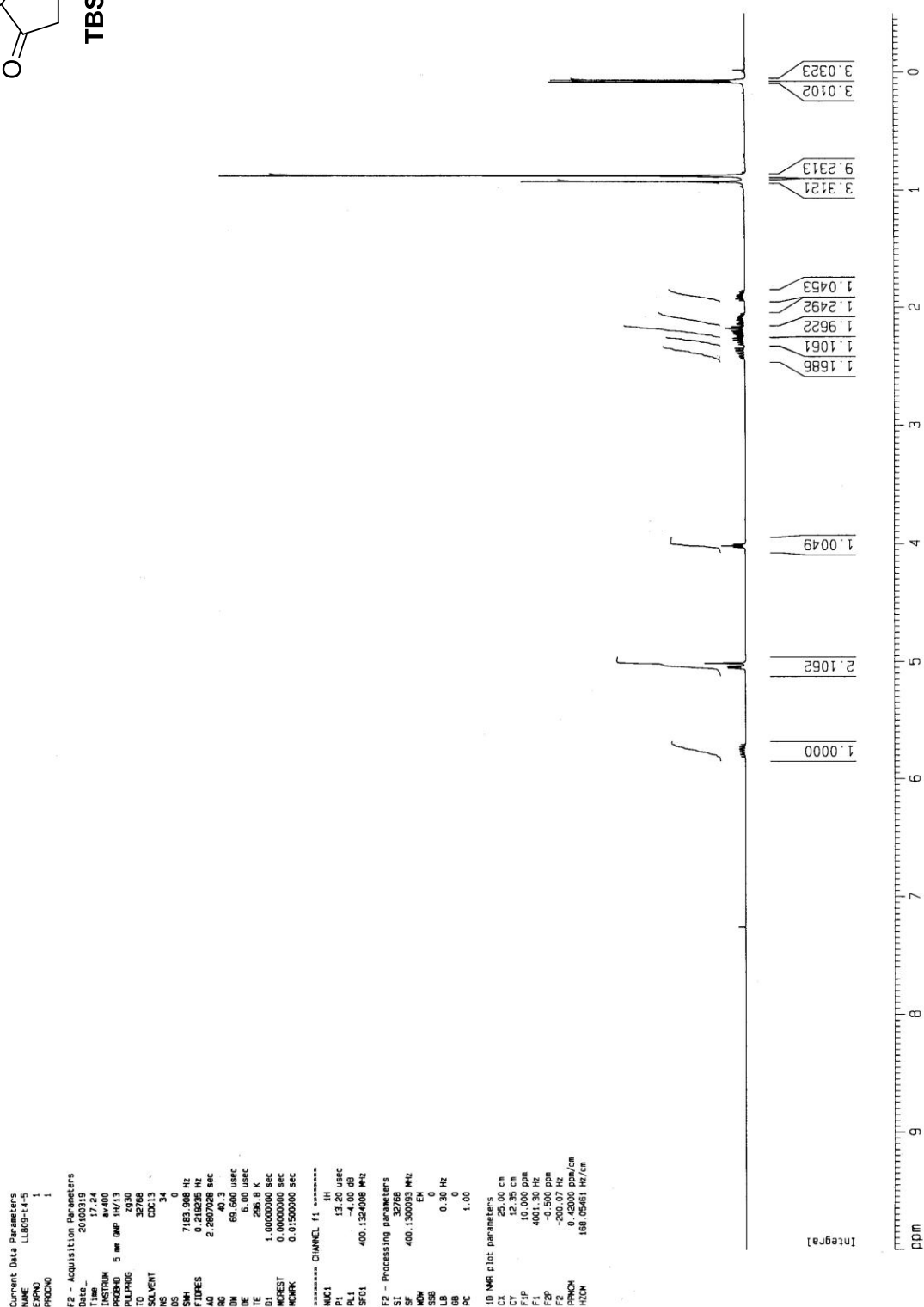


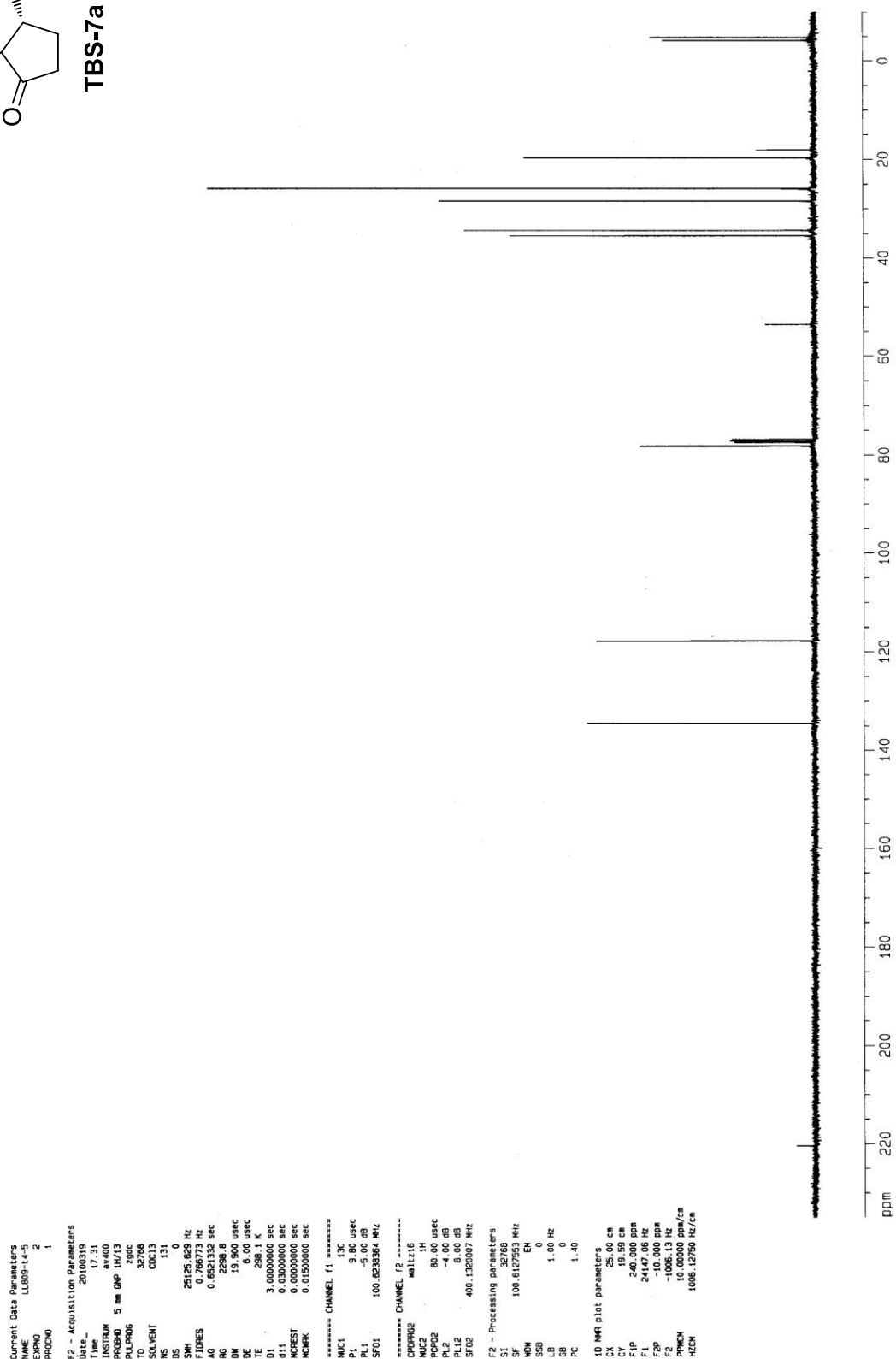
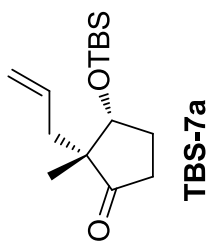




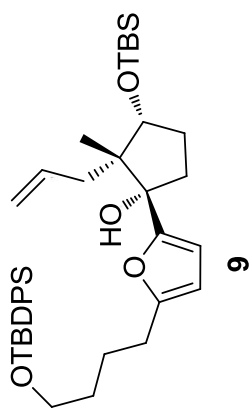


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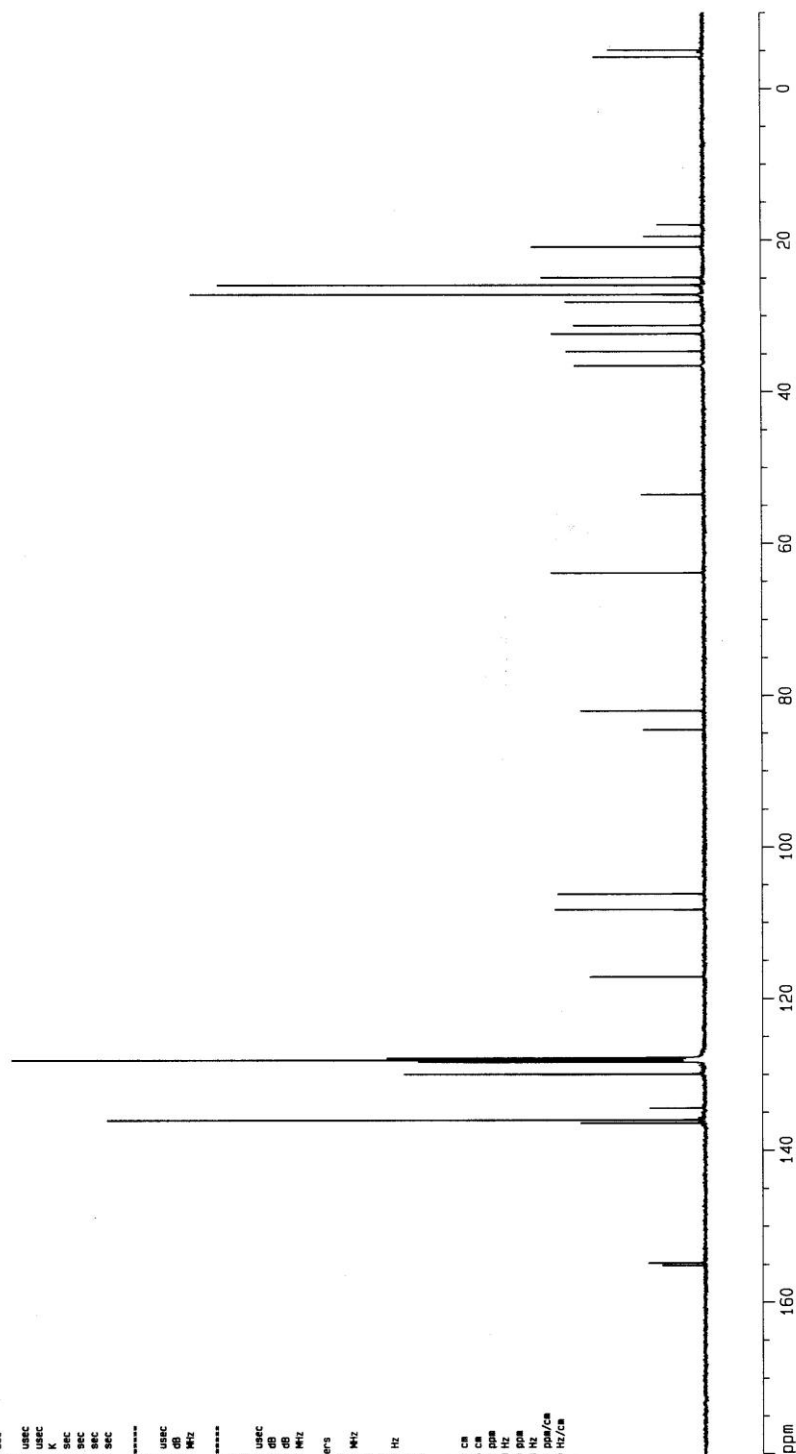


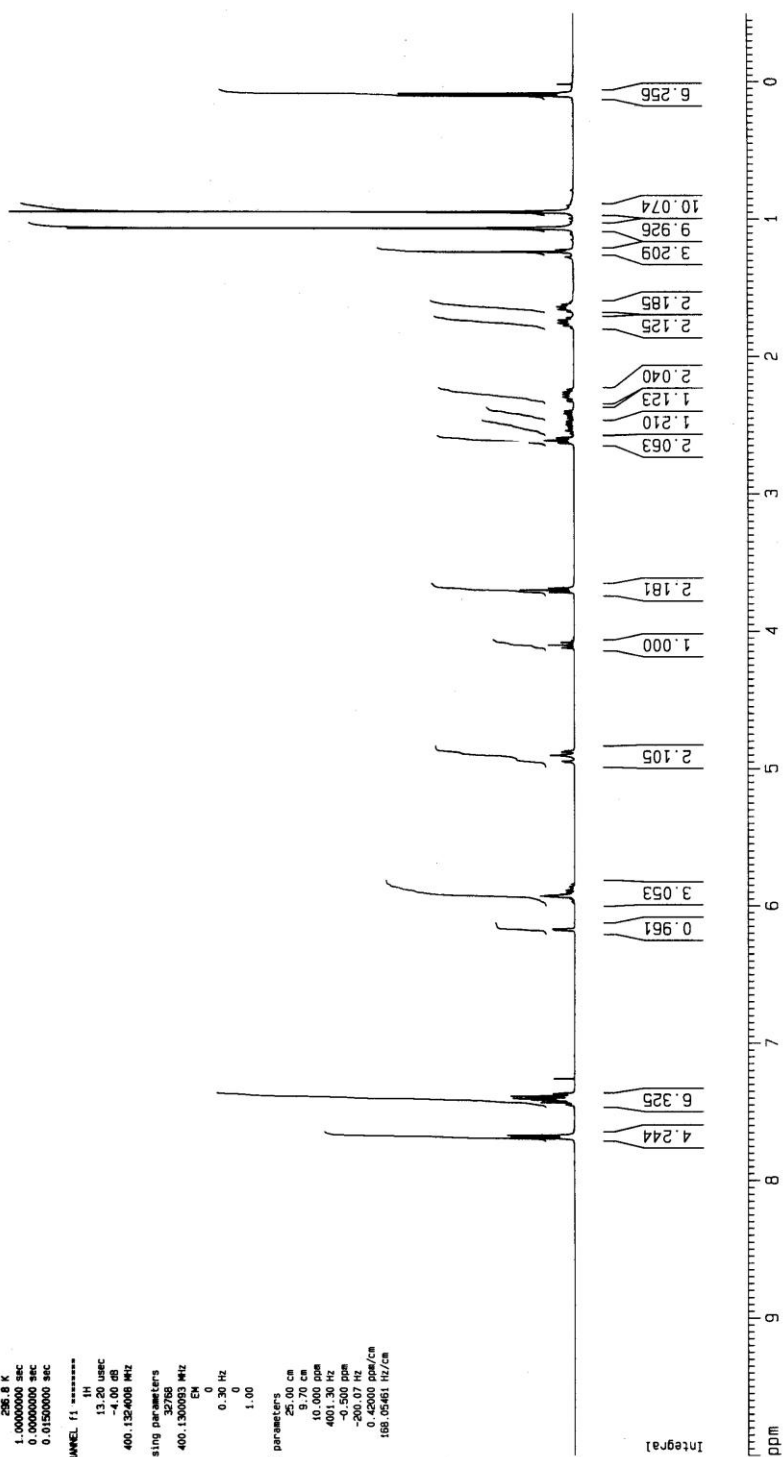


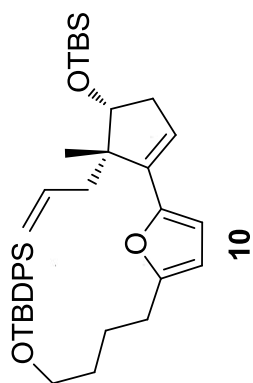
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EXPNO 2
PROCNO 1

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Time 15.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
FIDRES 0.32768
SOLVENT CDCl₃
NS 136
DS 0
SWH 25125.629 Hz
FIDRES 0.32768 Hz
AQ 0.0521132 sec
RG 2298.8
DM 19.900 usec
DE 6.00 usec
TE 297.3 K
D1 3.00000000 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
NUC1 13C
NUC2 1H
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -5.00 dB
SFO1 100.625000 MHz
===== CHANNEL f2 =====
CPDPRG2 zgpg30
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
SFO2 400.132007 MHz
F2 - Processing parameters
SI 32768
SF 100.6127431 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 25.00 cm
CY 11.93 cm
CZ 18.00 cm
F1 1810.20 Hz
F2 -10.000 ppm
F2 -1006.13 Hz
PPM0 7.60000 ppm/cm
HZ0 784.65690 Hz/cm







Current Data Parameters
NAME LL1014
EXPNO 2
PROCNO 1

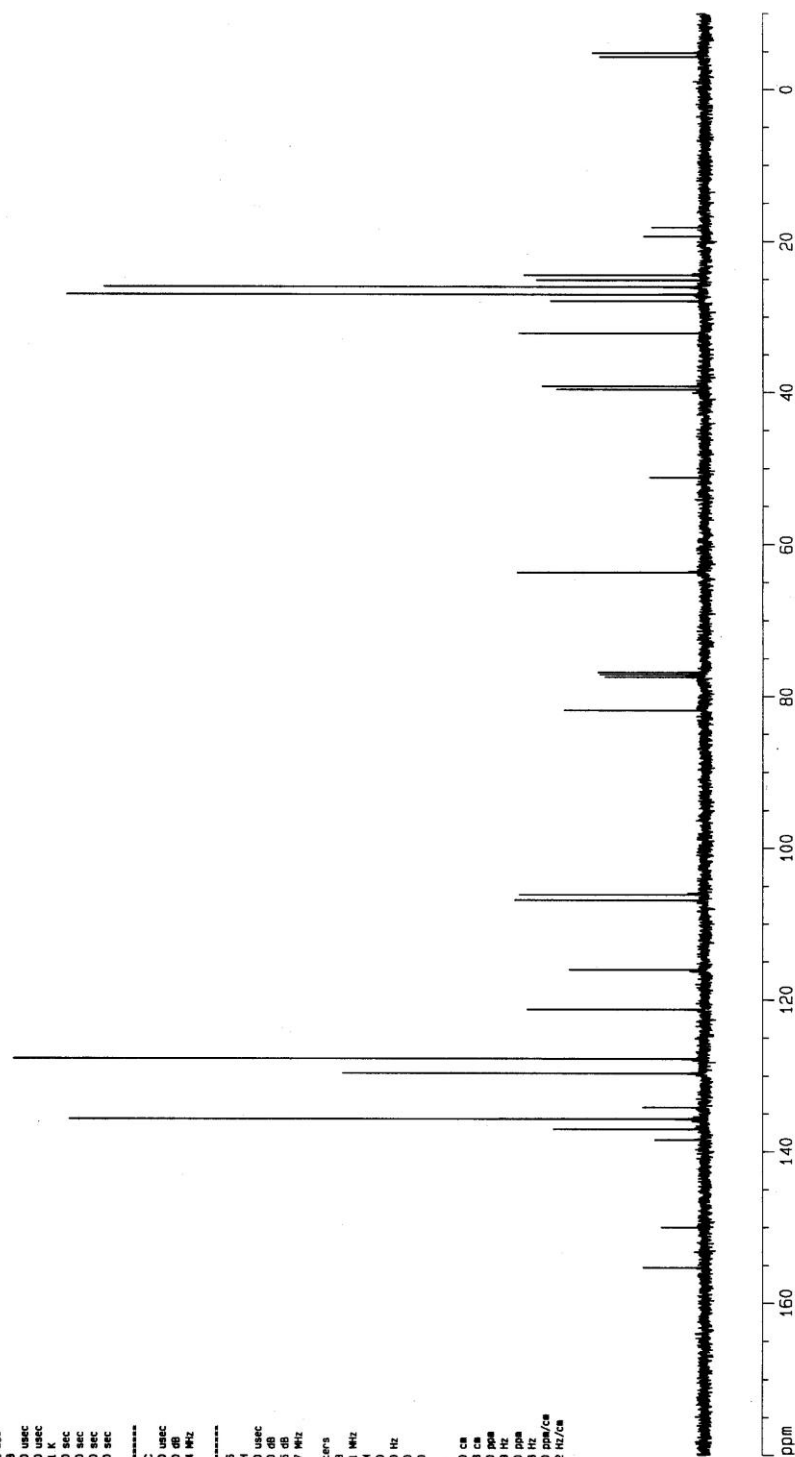
F2 - Acquisition Parameters
Date_ 20101109
Time 19.14
INSTRUM av400
PROBHD 5 mm QNP
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 80
DS 0
SWH 20126.859 Hz
FIDRES 0.000775 Hz
AQ 0.001512 sec
RG 2098.8
DM 19.900 usec
DE 6.00 usec
TE 297.1 K
D1 3.00000000 sec
d11 0.00000000 sec
DELTA 0.00000000 sec
NUC1 13C
NUC2 1H

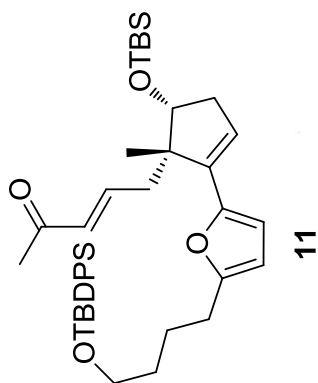
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NUC1 13C
P1 10.00 usec
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SFO1 100.6253504 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 -4.00 dB
SFO2 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127561 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 25.00 cm
CY 11.33 cm
FID 180
F1 1810.20 Hz
F2 -10.000 ppm
F3 -1006.13 Hz
PPMCH 7.50000 ppm/cm
HZCH 764.95692 Hz/cm





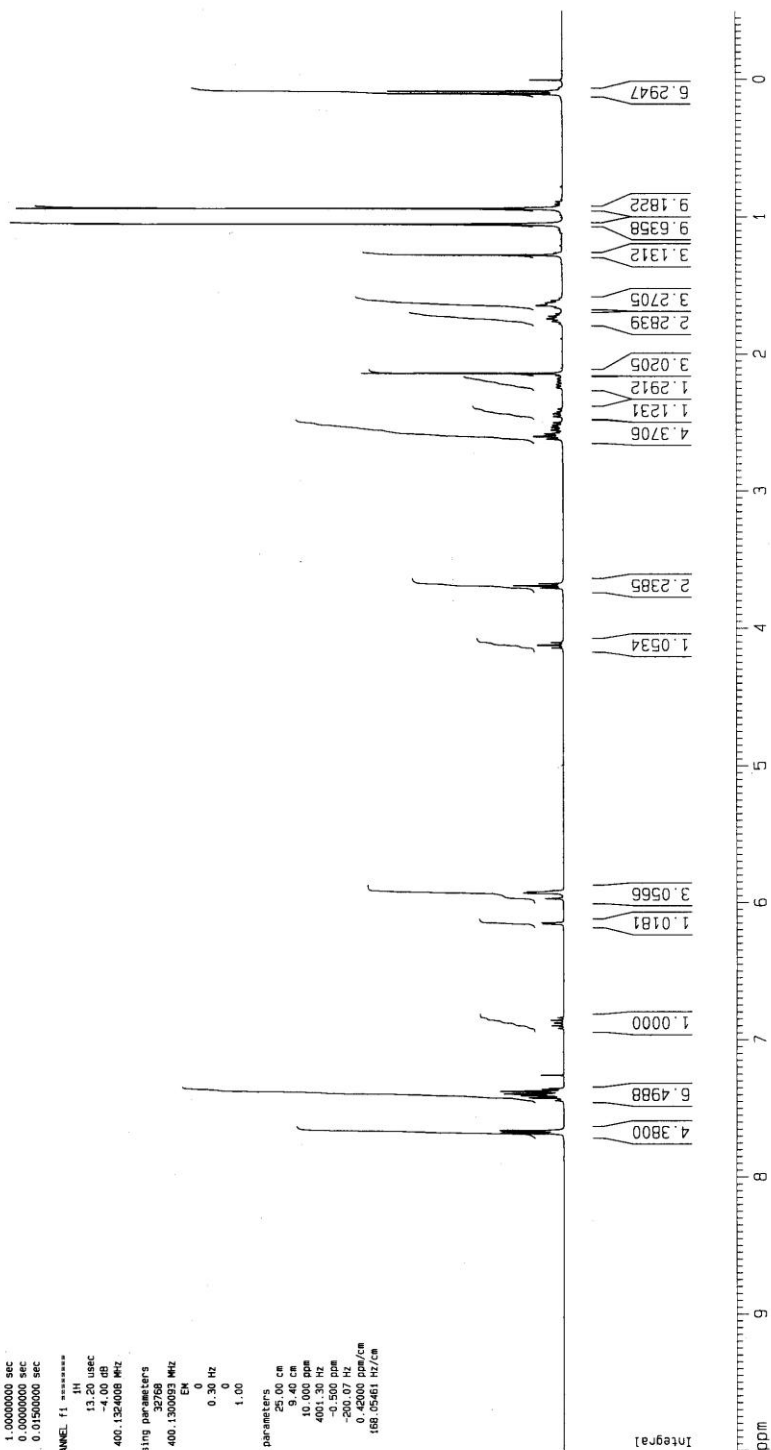
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NAME: 11-17
EXPNO: 1
PROCNO: 1

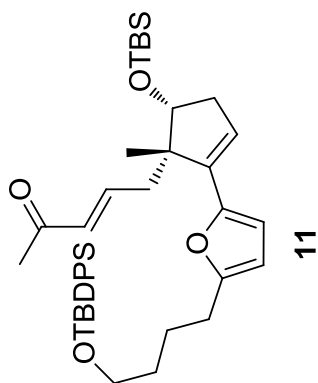
F2 - Acquisition Parameters
Date_: 20100405
Time: 17:05
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 15
DS: 4
SWH: 7163.908 Hz
FIDRES: 0.218295 Hz
AQ: 2.2867028 sec
RG: 90.5
DM: 69.600 usec
DE: 6.00 usec
TE: 300.2 K
D1: 1.0000000 sec
d11: 0.0000000 sec
d12: 0.0000000 sec
d13: 0.0150000 sec
MCPRK: 0.0150000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 13.20 usec
PL1: -2.00 dB
SFO1: 400.1324008 MHz

F2 - Processing parameters
SI: 32768
SF: 400.1350093 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
RG: 256.00 cm
CY: 9.40 cm
FIP: 10.000 ppm
F1: 4001.30 Hz
F2: -0.500 ppm
F2: -200.07 Hz
NUC2: 13C
NUC2: 100.6260000 MHz





Current Data Parameters
NAME: LUG2317
EXPNO: 2
PROCNO: 1

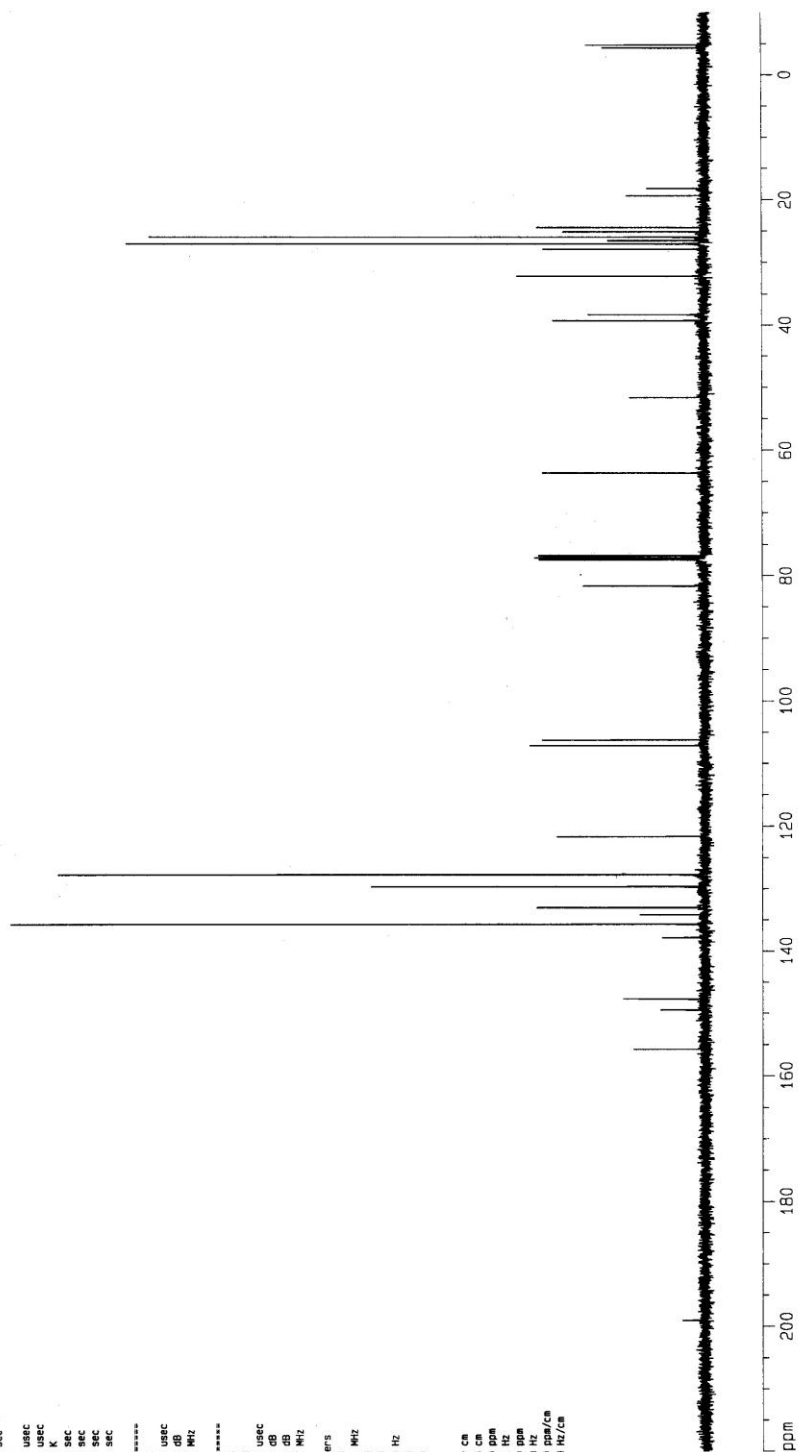
F2 - Acquisition Parameters
Date_: 20100405
Time: 17.13
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 146
DS: 0
SWH: 25125.658 Hz
FIDRES: 0.166773 Hz
AQ: 0.6521332 sec
RG: 2296.8
DM: 19.900 usec
DE: 6.00 usec
TE: 297.9 K
D1: 3.0000000 sec
d11: 0.0500000 sec
d12: 0.0000000 sec
d13: 0.0000000 sec
d14: 0.0000000 sec
d15: 0.0150000 sec
d16: 0.0150000 sec

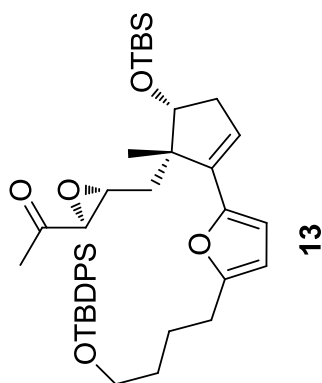
===== CHANNEL f1 =====
NUC1: 13C
P1: 9.80 usec
PL1: -2.50 dB
SF01: 100.628354 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: -1.50 dB
PL12: 8.00 dB
SF02: 400.132007 MHz

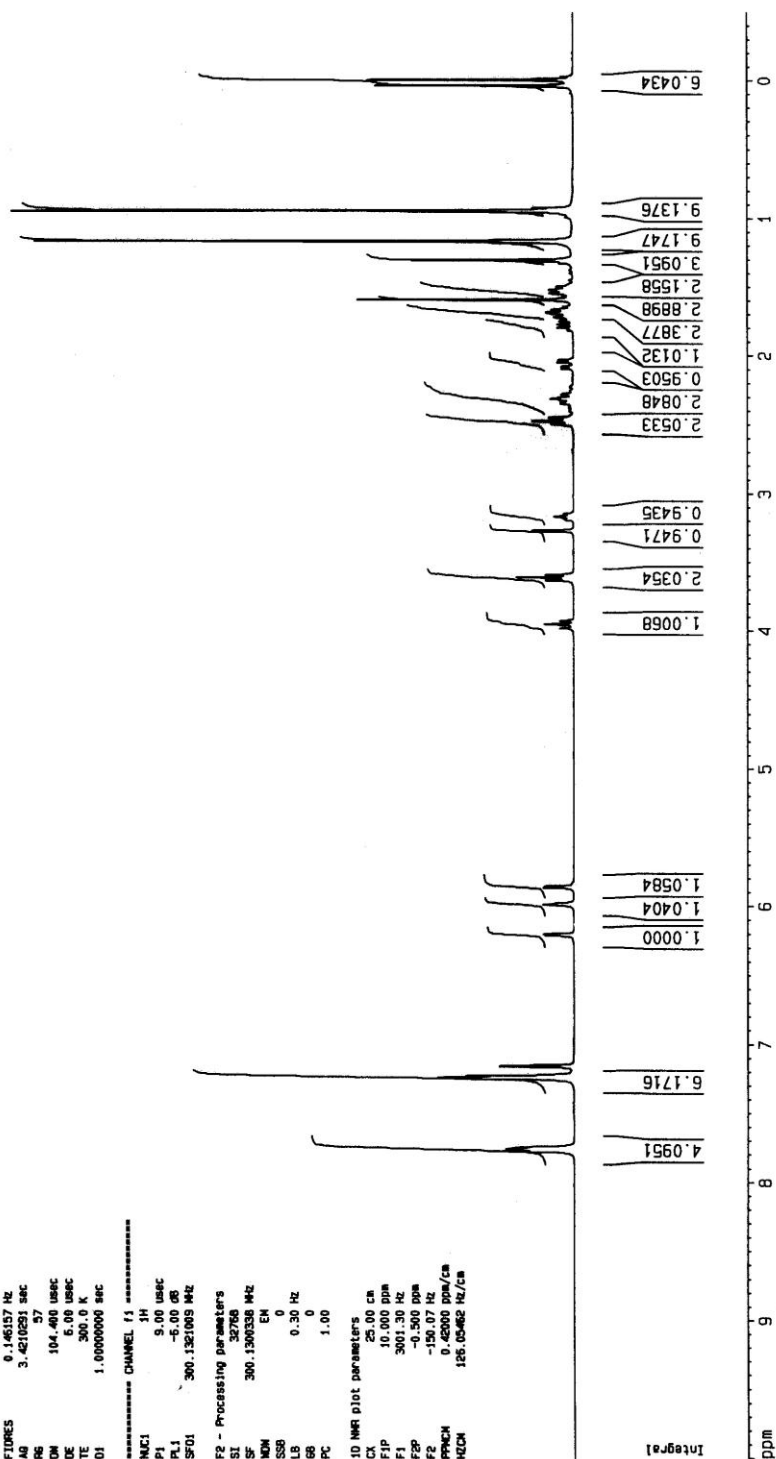
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SI: 32768
SF: 100.6127561 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

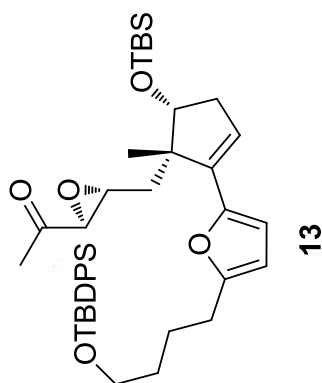
1D NMR plot parameters
CX: 25.00 cm
CY: 25.00 cm
FID: 220.000 ppm
F1: 23134.81 Hz
F2: -10.000 ppm
F2: -1005.13 Hz
P1: 9.20000 ppm/cm
H2O: 925.63735 Hz/cm





Current Data Parameters
NAME LLJ026
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101207
Time 16.35
INSTRUM spect
PROBHD QNP300
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 400
DS 4
SWH 4789.275 Hz
FIDRES 0.146157 Hz
AQ 3.4210201 sec
RG 57
DM 104.400 usec
DE 5.00 usec
TE 300.0 K
D1 1.00000000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -5.00 dB
SFO1 300.1361009 MHz
F2 - Processing parameters
SI 32768
SF 300.1360338 MHz
WDW EN
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ID NMR plot parameters
CX 25.00 cm
FID 10.000 ppa
F1 3001.30 Hz
F2 -0.500 ppa
F3 -150.07 Hz
PPHCH 0.42000 ppa/cm
HZCH 125.05462 Hz/cm





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Current Data Parameters
NAME      LL1026
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20101207
Time      16.45
INSTRUM   dx300
PROBHD    DUAL 5mm

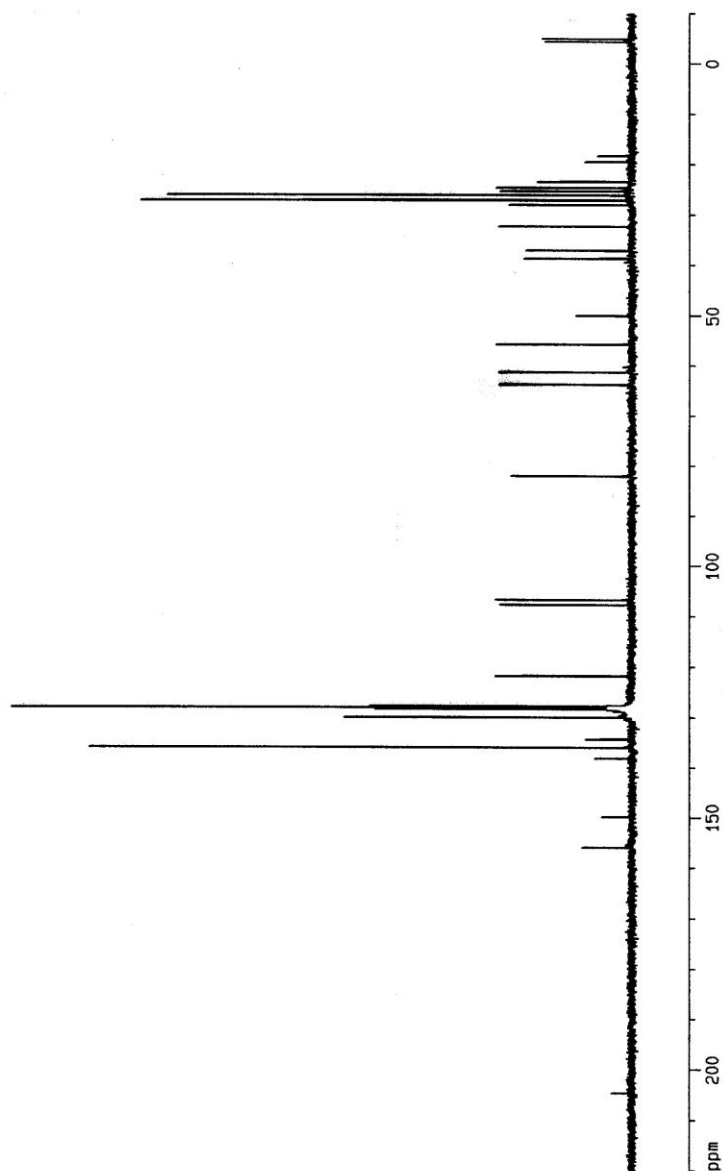
PULPROG   zgpg
TD         32768
SOLVENT    CDCl3
NS         128
DS         1
SWH        18832.363 Hz
FIDRES     0.574719 Hz
AQ         0.8700404 sec
RG         6192
DM         26.550 usec
DE         6.00 usec
TE         300.0 K
D1         2.50000000 sec
d11        0.03000000 sec

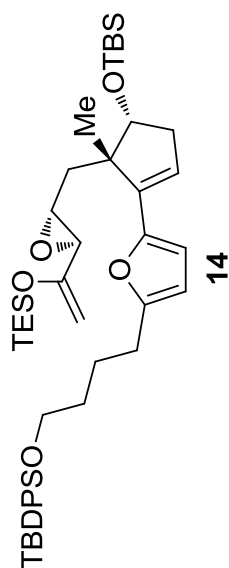
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NUC1       13C
P1         7.00 usec
PL1        -6.00 dB
SFO1       75.4760204 MHz

===== CHANNEL f2 =====
CPOPRG2    waltz16
NUC2        1H
PCPD2       100.00 usec
PL2         120.00 dB
PL12        18.00 dB
SFO2       300.1312005 MHz

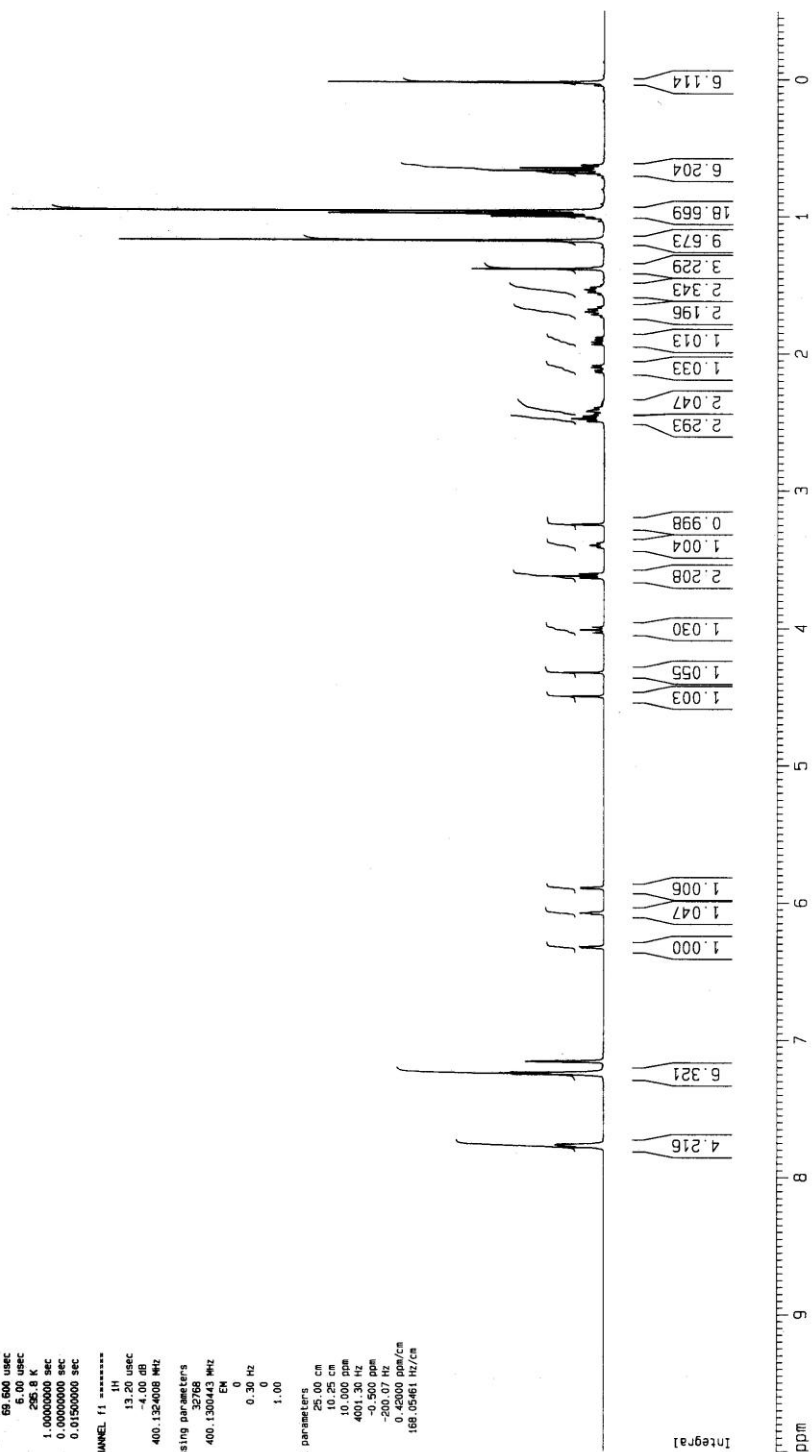
F2 - Processing parameters
SI         32768
SF         75.4677296 MHz
NM         EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

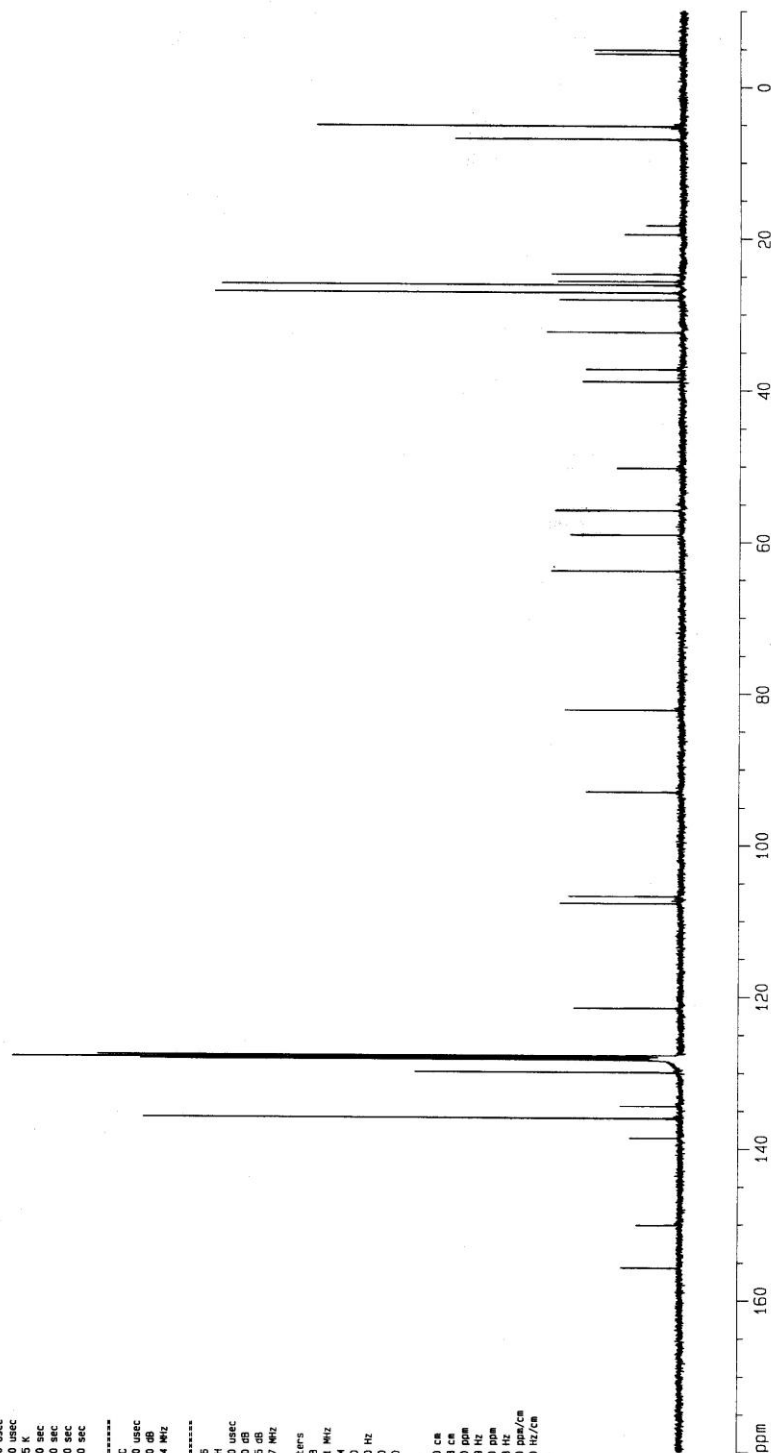
1D NMR plot parameters
CX         20.00 cm
F1P        220.000 ppm
F1         16602.90 Hz
F2P        -10.000 ppm
F2         -754.68 Hz
PPHCH      11.50000 ppm/cm
HZCH       867.87885 Hz/cm
  
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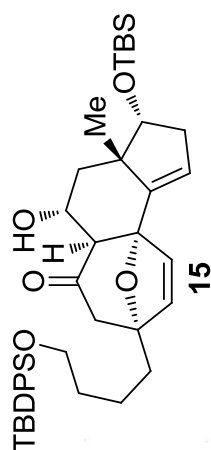


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NAME: 11027
EXPNO: 1
PROCNO: 1
F2 - Acquisition Parameters
Date_: 20101209
Time: 14.11
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 32768
SOLVENT: DMSO
NS: 512
DS: 4
SWH: 7163.500 Hz
FIDRES: 0.214226 Hz
AQ: 2.2807028 sec
RG: 50.8
DM: 69.600 usec
DE: 6.00 usec
TE: 295.2 K
D1: 1.00000000 sec
d11: 0.00000000 sec
d12: 0.00000000 sec
d13: 0.00000000 sec
d14: 0.01500000 sec
===== CHANNEL f1 =====
NUC1: 1H
P1: 13.20 usec
PL1: 0.00 dB
SFO1: 400.1324000 MHz
F2 - Processing parameters
SI: 32768
SF: 400.1300443 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
ID: NMR plot parameters
CY: 256.00 cm
CX: 10.25 cm
CY: 10.000 ppm
F1P: 4001.30 Hz
F2P: -0.500 ppm
F2: -200.07 Hz
PQCH: 0.42500 ppm/cm
HLCN: 188.05461 Hz/cm

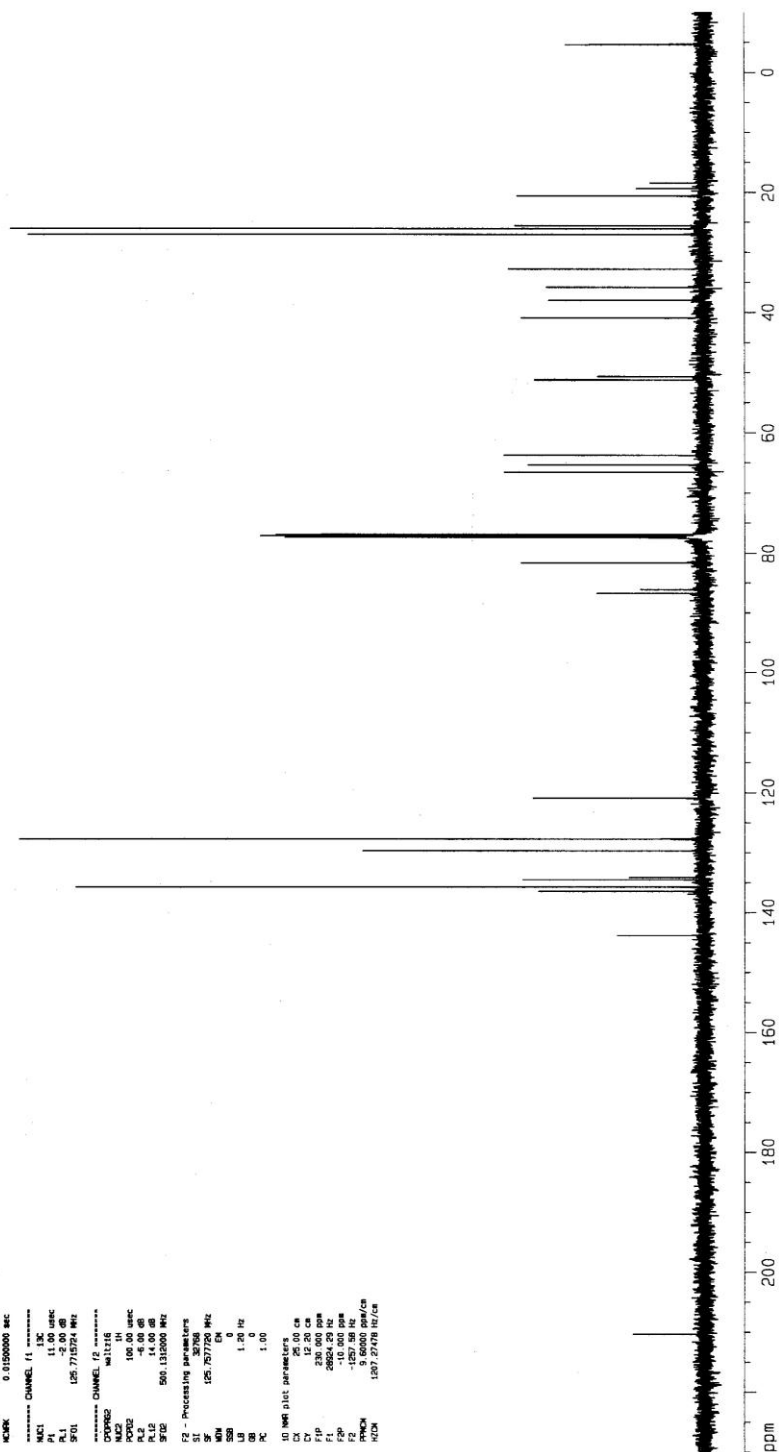


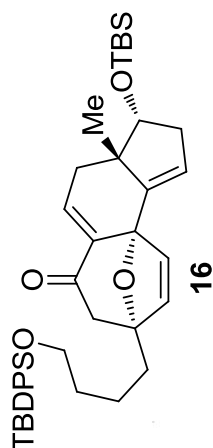






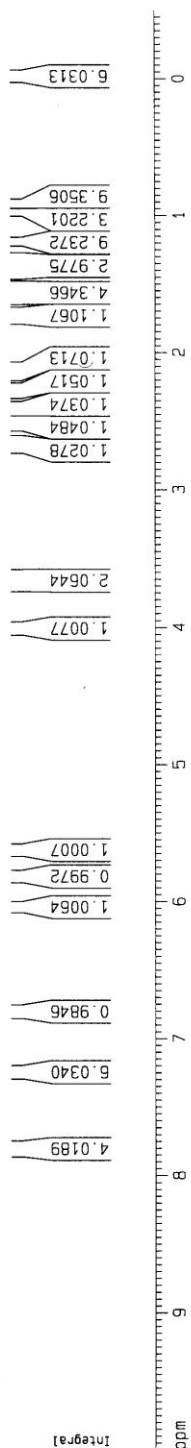
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PROCNO 1
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Time 16:16
INSTRUM spect
PROBHD BBO Sm 231d/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
DS 1
OS 1
SH 31446.541 Hz
FIDRES 0.5200000 Hz
AQ 0.5200000 Hz
RG 7298.2
RW 15.900 Hz
OR 15.900 Hz
TE 298.15 K
D1 3.0000000 sec
D11 11.0000000 sec
D2 0.5000000 sec
D21 0.5000000 sec
D3 0.0150000 sec
D31 0.0150000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 11.00 usec
PL 0.00 dB
PC 1.00
SFO1 125.761254 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 100.00 usec
PL2 0.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 500.1310000 MHz
F2 - Processing parameters
SI 32768
SF 125.757720 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00
LO WALTZ parameters
CY 25.00 Hz
CY 12.20 Hz
F1 200.1310000 MHz
F2 -10.000 Hz
F3 -125.758 Hz
F4 125.757720 MHz
H2CN 125.757720 MHz

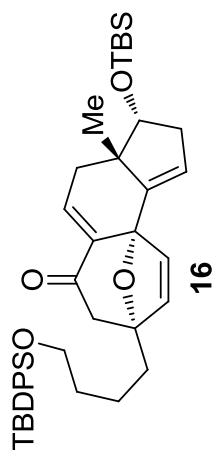




Current Data Parameters
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PROCNO 1
F2 - Acquisition Parameters
Date_ 20100519
Time 14.54
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 12
DS 4
SWH 7183.908 Hz
FIDRES 0.218235 Hz
AQ 2.2807028 sec
RG 161.3
DM 69.600 uM
DE 6.00 uM
TE 300.2 K
D1 1.0000000 sec
d11 0.0000000 sec
d12 0.0000000 sec
d13 0.0150000 sec
d14 0.0150000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 13.00 uM
PL1 -4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 32768
SF 400.1300443 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
===== CHANNEL f2 =====
NUC2 13C
P2 12.50 uM
PL2 -4.00 dB
SFO2 100.6261250 MHz
F2 - Processing parameters
SI 32768
SF 100.6261250 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
===== CHANNEL f3 =====
NUC3 13C
P3 12.50 uM
PL3 -4.00 dB
SFO3 100.6261250 MHz
F3 - Processing parameters
SI 32768
SF 100.6261250 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Integral





Current Data Parameters
Name: 16
EXPNO: 2
PROCNO: 1

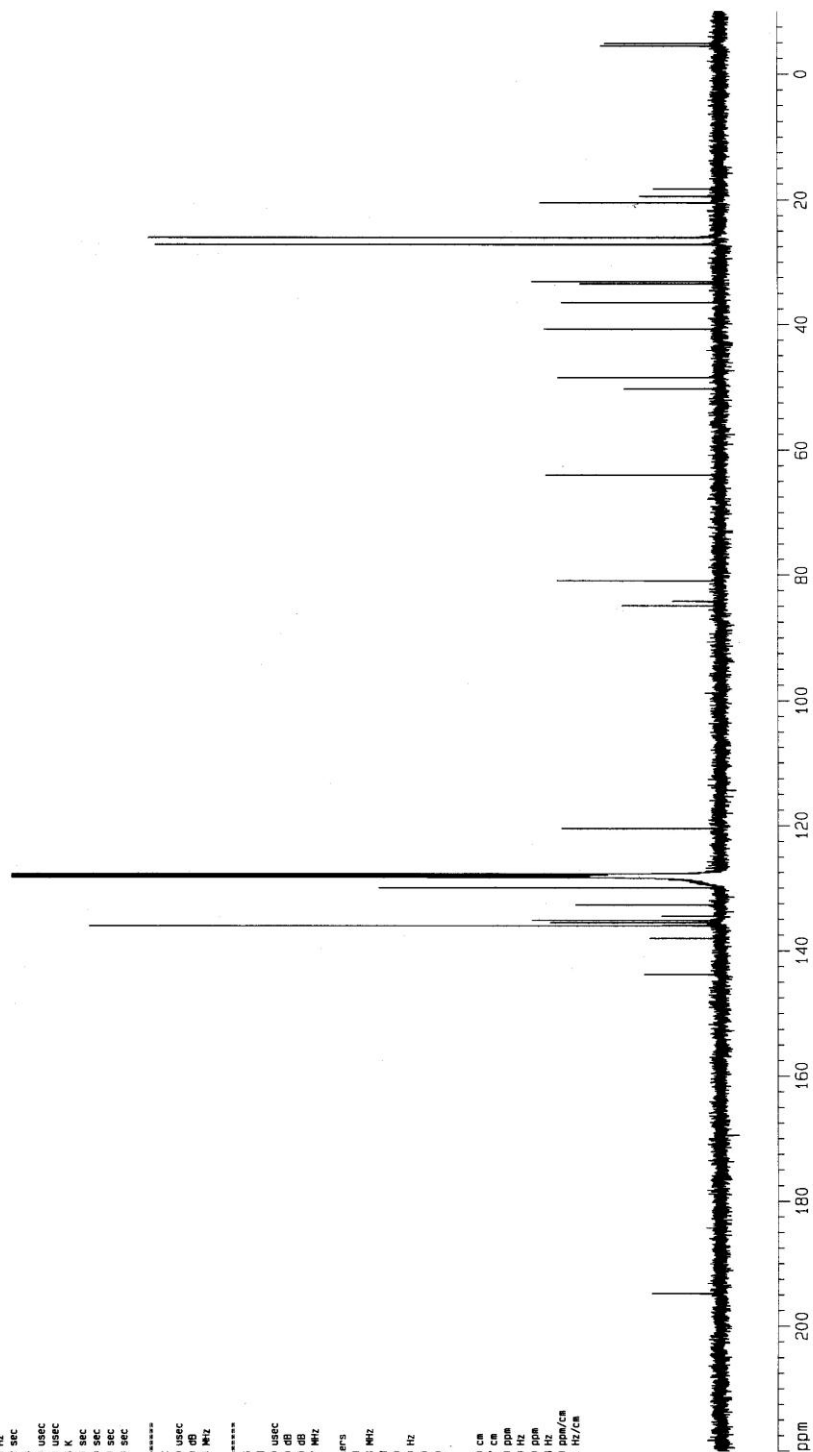
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Time: 15.02
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PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TO: 32768
SOLVENT: CDCl3
NS: 276
DS: 0
SWH: 25125.620 Hz
FIDRES: 0.166771 Hz
AQ: 0.652132 sec
RG: 2298.8
DM: 19.800 usec
DE: 6.00 usec
TE: 298.2 K
T1: 3.0000000 sec
T1RHO: 0.0000000 sec
T2: 0.0000000 sec
T2RHO: 0.0000000 sec
MORPH: 0.0150000 sec
MORPH: 0.0150000 sec

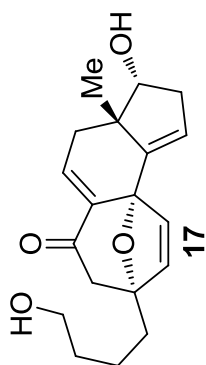
===== CHANNEL f1 =====
NUC1: 13C
P1: 6.00 usec
PL1: 0.00 dB
SFO1: 100.628354 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
P2: 80.00 usec
PL2: 0.00 dB
PL12: 8.00 dB
SFO2: 400.132007 MHz

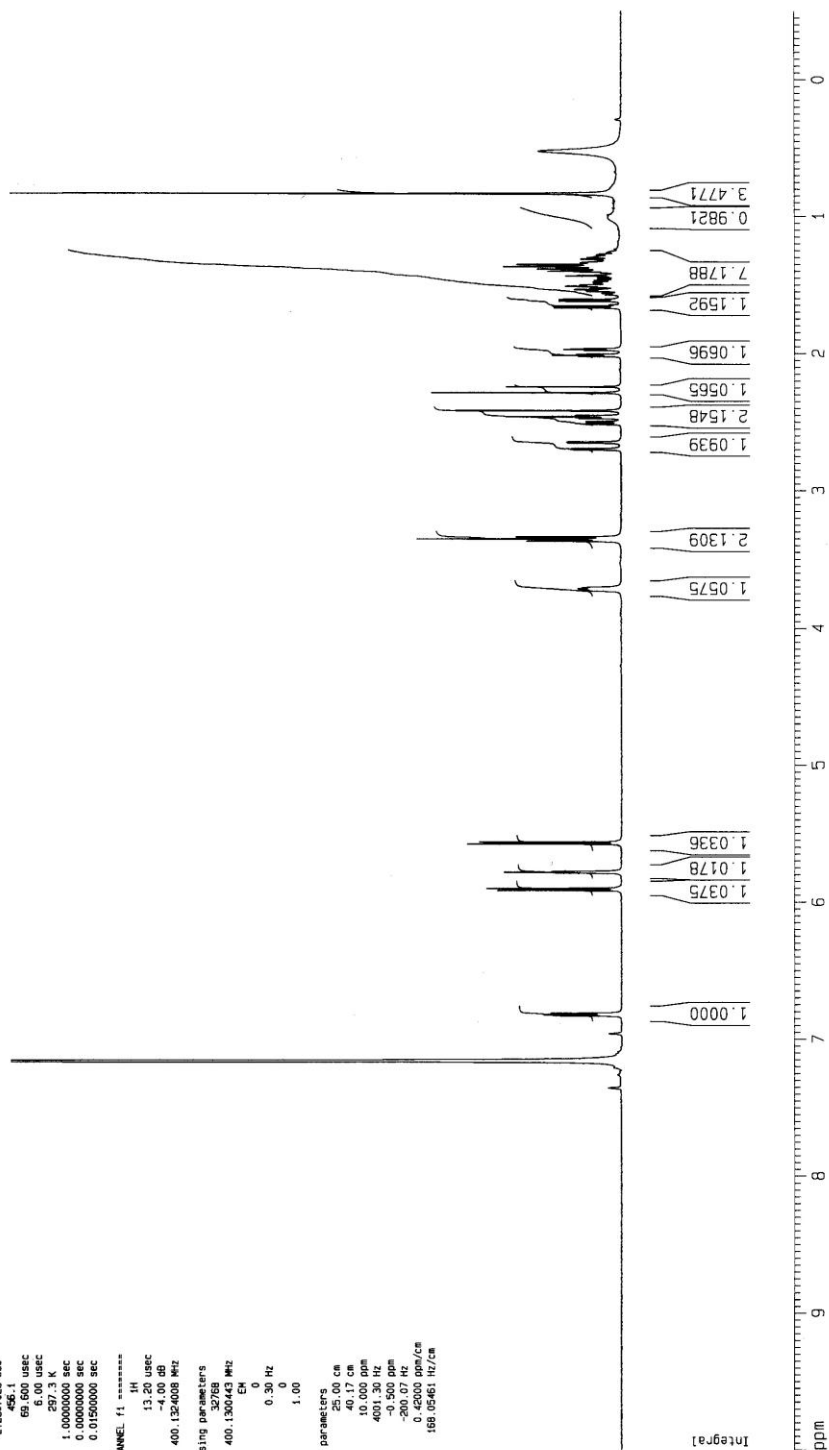
F2 - Processing parameters
SI: 32768
SF: 100.6127424 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

1D NMR plot parameters
X: 250.00
Y: 250.00
F1: 220.000 ppm
F2: 22134.80 Hz
F2P: -10.000 ppm
F2: -1005.13 Hz
PCHN: 9.20000 ppm/cm
HZCM: 925.6371 Hz/cm

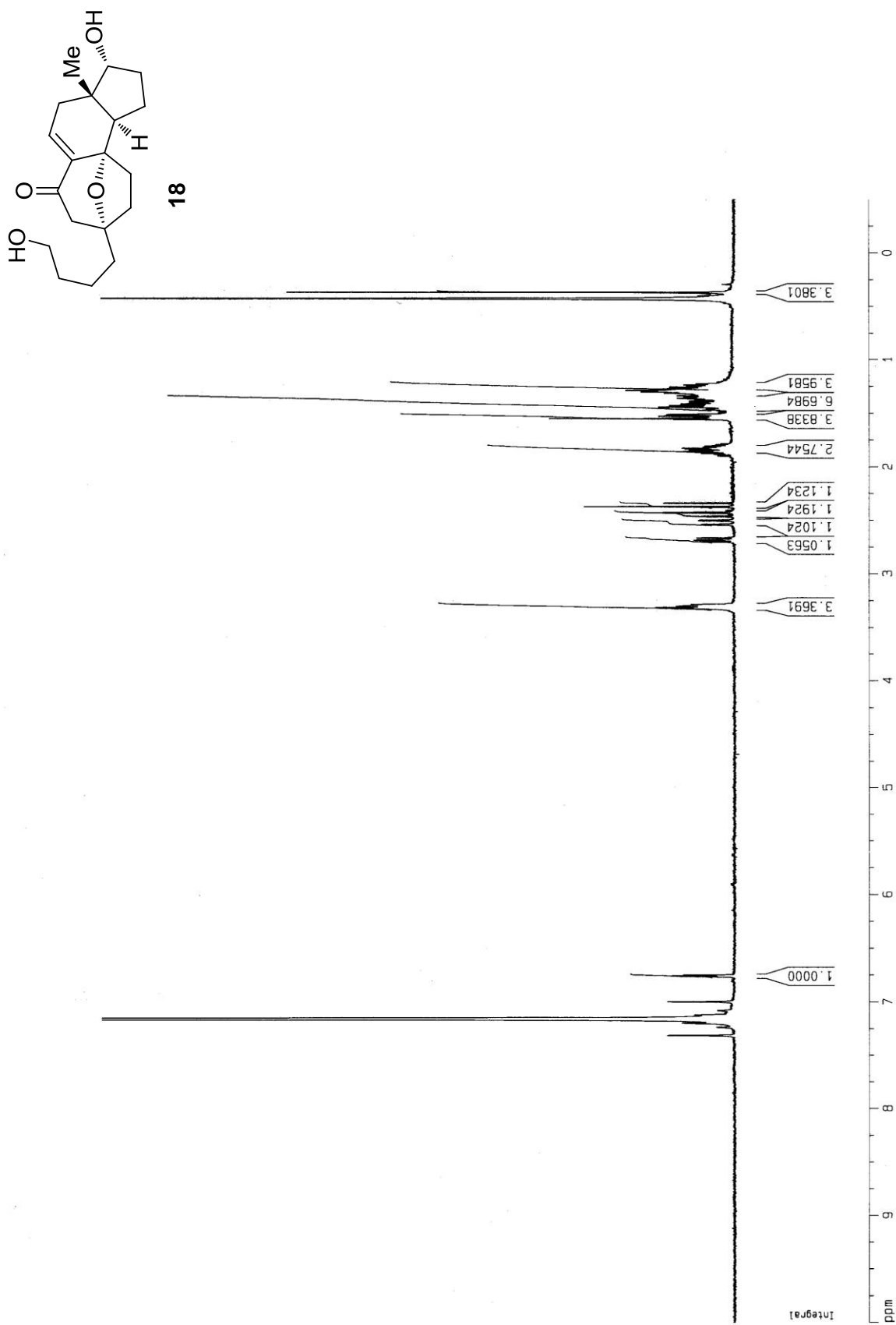




Current Data Parameters
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100524
Time 17:16:50
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 20
DS 4
SWH 7183.908 Hz
FIDRES 0.218235 Hz
AQ 2.2867028 sec
RG 456.1
DM 69.600 uSec
DE 6.00 uSec
TE 300.2 K
D1 1.00000000 sec
MCHRG 0.00000000 sec
MCHRG 0.01500000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 13.00 uSec
PL1 -4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 32768
SF 400.1350443 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
D0 NMR file parameters
CY 25.00 cm
C4 40.17 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
NUC2 13C
P2 12.00 uSec
PL2 0.00 dB
SFO2 100.6261197 MHz

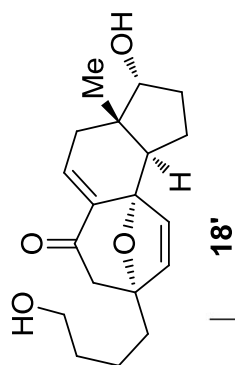




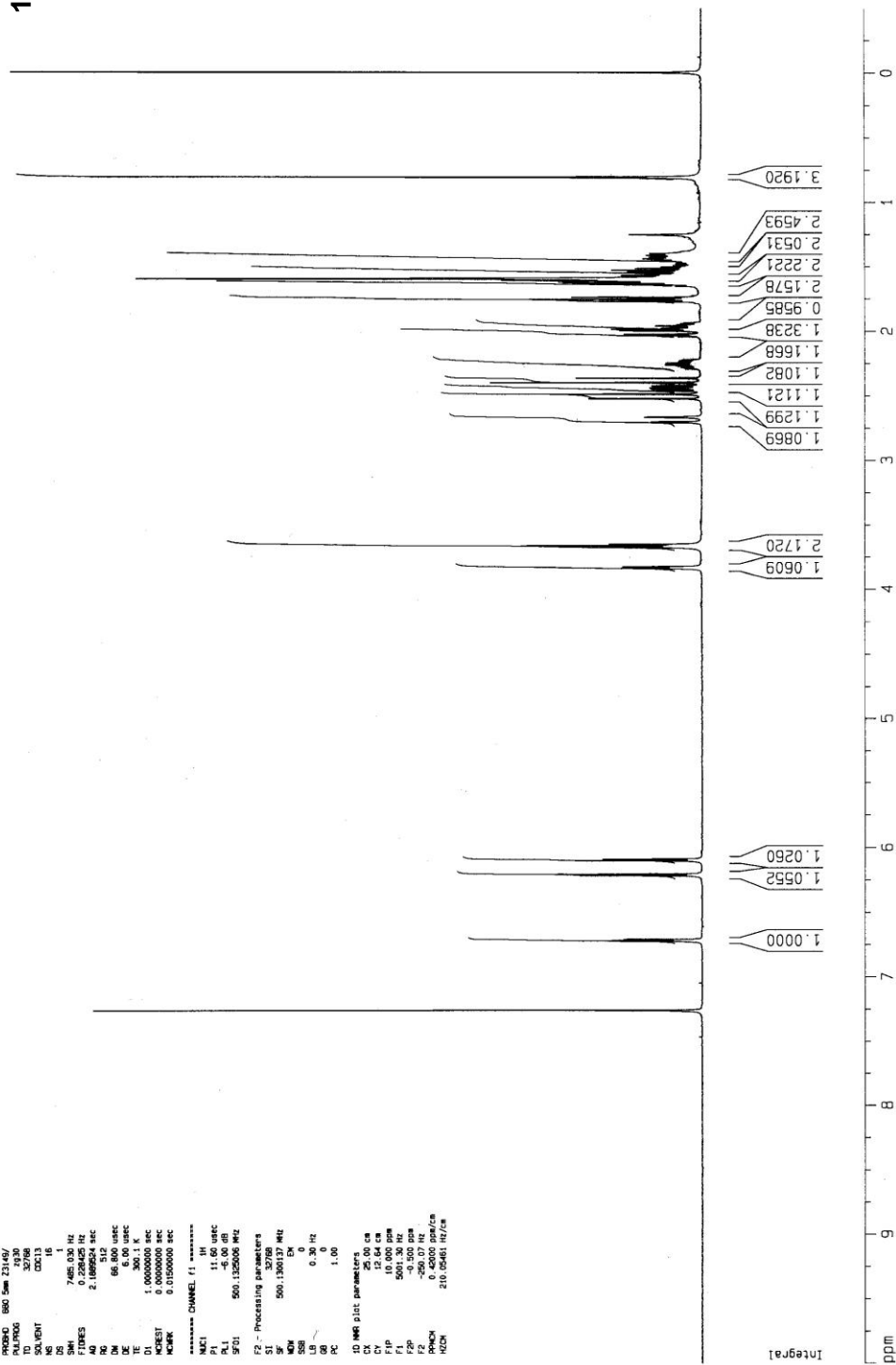


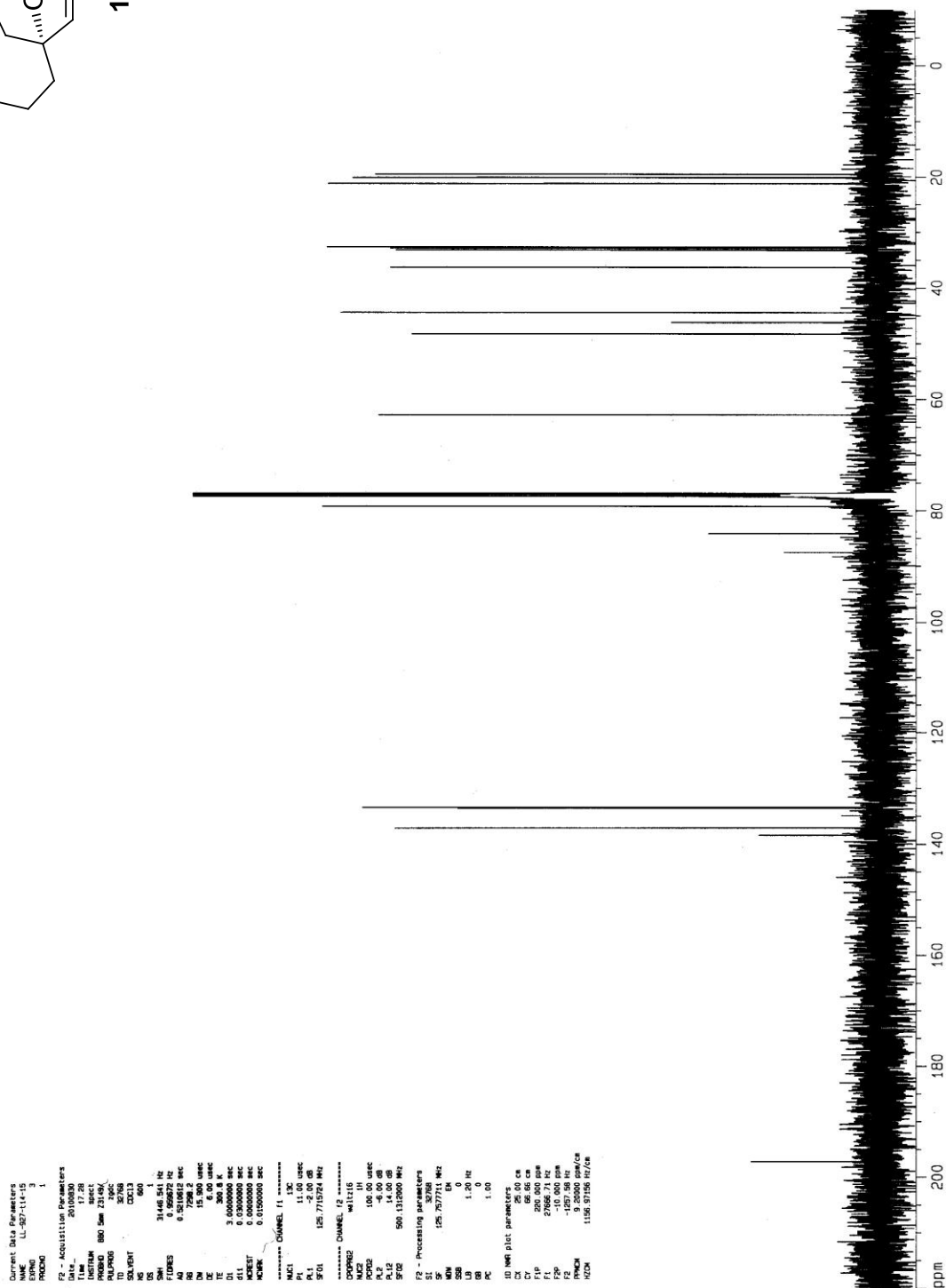
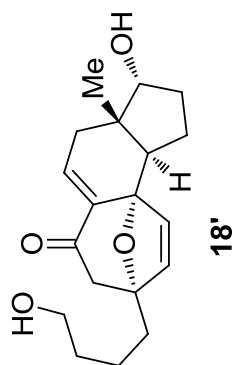
18



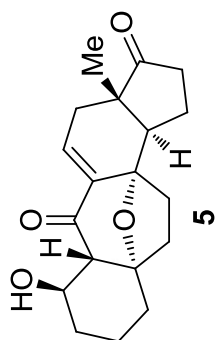


Current Data Parameters
NAME 18-27-14-15
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100930
Time 17:44
INSTRUM spect
PROBHD 5mm Z146/
PULPROG zgpg30
SOLVENT DMSO
SOLVENT DMSO
NS 16
DS 4
SWH 7465.030 Hz
FIDRES 0.228425 Hz
AQ 2.1889924 sec
RG 327.500
IN 66.800 usec
DE 6.00 usec
TE 300.1 K
TD 65536
SFO 500.136037 MHz
KRGST 0.0000000 sec
MCURK 0.0150000 sec
***** CHANNEL f1 *****
NUC1 1H
P1 11.60 usec
PL1 -5.00 dB
SFO1 500.136037 MHz
F2 - Processing parameters
SI 32768
SF 500.136037 MHz
WDW EN
SSB 0
GB 0
PC 1.00
DO 4096 points
CX 25.00 cm
CY 12.64 cm
F1 10.135300 MHz
F2 -0.500000 ppm
F2 250.07 Hz
PCPN 0.0000000 sec
PCPN 210.05461 Hz/sec



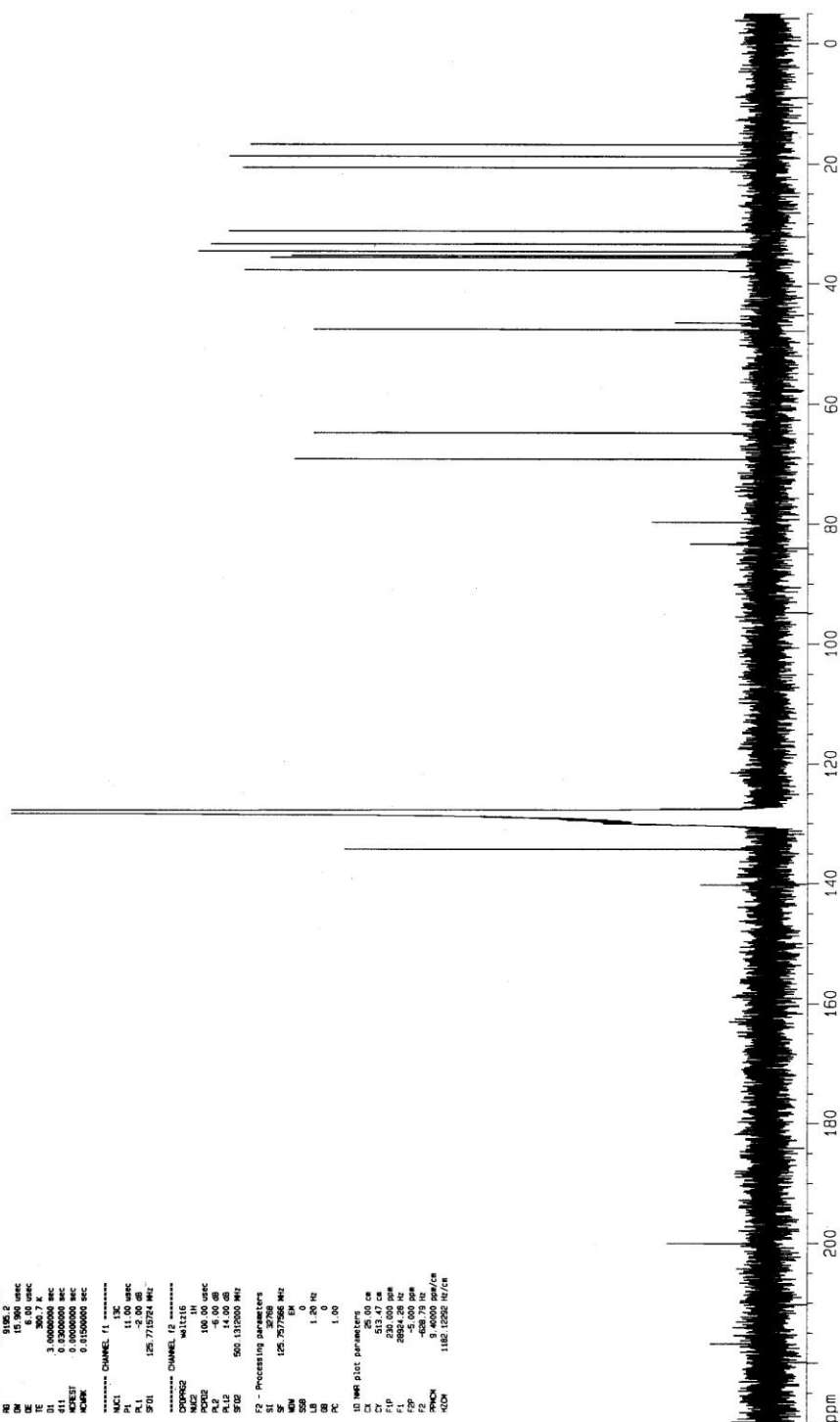


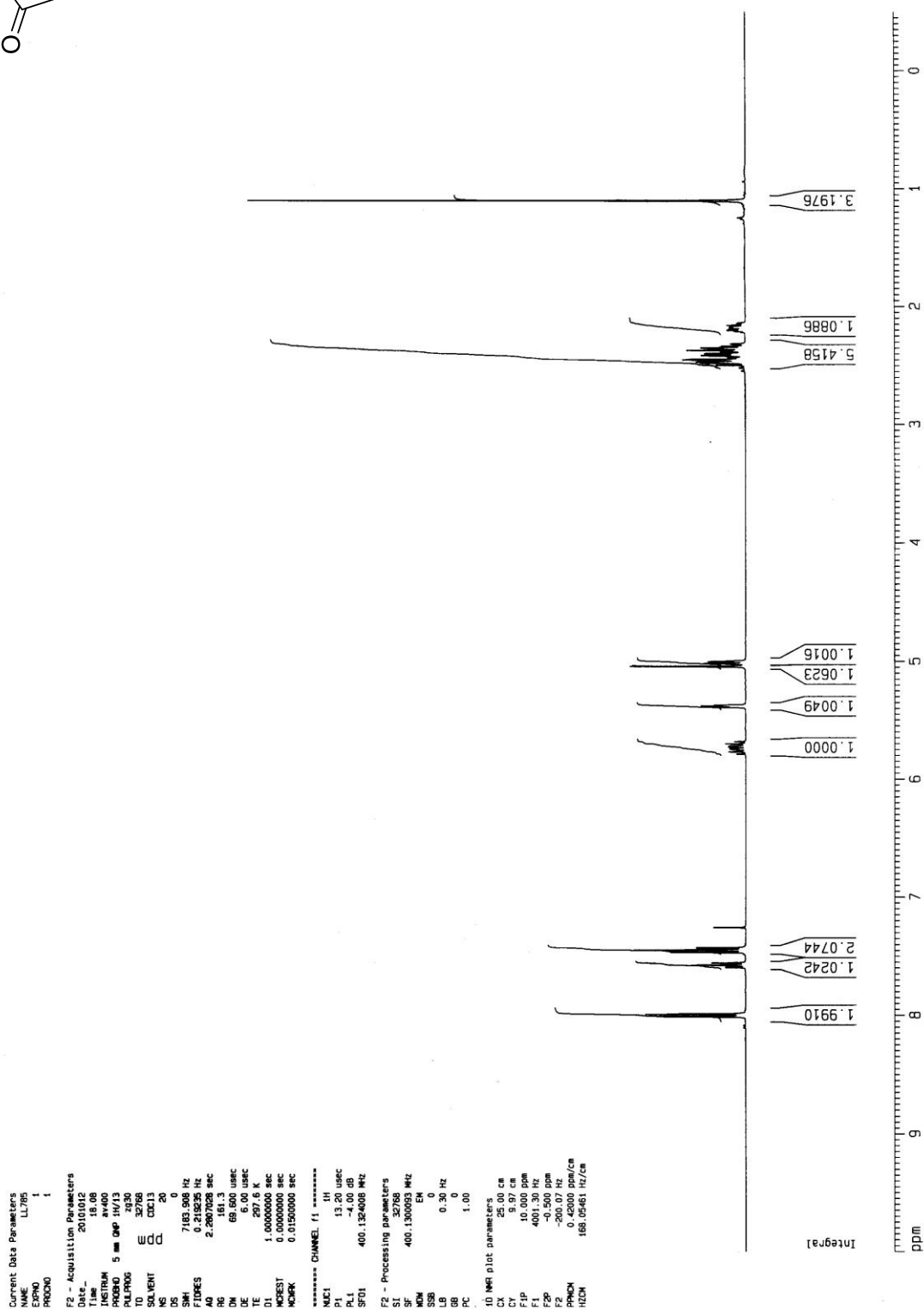
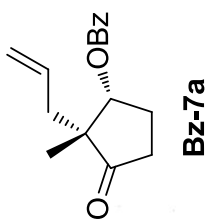


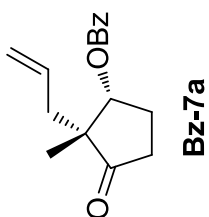


```

Current Data Parameters
NAME          LL-997
EXPNO         3
PROCNO        1
F2 - Acquisition Parameters
Date_         20100505
Time          15.58
INSTRUM       spect
PROBHD        BBO 5mm 23146/
PULPROG       zgpg30
TD             32768
SOLVENT       CDCl3
NS            1511
DS            1
SWH            31446.541 Hz
FIDRES         0.520072 Hz
AQ             0.5200512 sec
RG             9195.2
AQ             15.960 usec
DE            3.00 usec
TE             300.2 K
D1             3.00000000 sec
d11            0.00000000 sec
DELTA          0.00000000 sec
ACQRES        0.01500000 sec
***** CHANNEL f1 *****
NUC1           13C
P1             11.00 usec
PL             0.00 dB
SFO1          125.767576 MHz
***** CHANNEL f2 *****
CPDPRG2       waltz16
NUC2           1H
P2             100.00 usec
PL2            19.00 dB
PL12           14.00 dB
SFO2          500.1310000 MHz
F2 - Processing parameters
SI             32768
SF            125.757568 MHz
WDW            0
SSB            0
LB             1.20 Hz
GB             0
PC             1.00
LS WFT plot parameters
CH            26.00 cm
CY            513.47 cm
F1P           228.000 MHz
F2P           -5.000 MHz
FE            408.70 Hz
WDW            0
SSB            0
LB             1.000 Hz
GB             0
PC            1180.0000 Hz/Hz
  
```







Current Data Parameters
NAME LL765
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101012
Time 18.14
NORM 18.14
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 108
DS 0
SWH 25125.629 Hz
FIDRES 0.000132 Hz
AQ 0.052132 sec
RG 4096
DM 19.900 usec
DE 6.00 usec
TE 298.3 K
D1 3.00000000 sec
DELTA 0.03000000 sec
WALTZ16 0.01500000 sec
WALTZ16 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -5.00 dB
SFO1 100.6253504 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 -4.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127568 MHz
WDW EN
SSB 0
GB 1.00 Hz
PC 1.40

10 MHz plot parameters
CX 25.00 cm
CY 11.93 cm
FID 240.00 Hz
F1 24147.06 Hz
F2 0.000 Hz
PPHCH 9.60000 ppm/cm
HZCH 965.98257 Hz/cm

