

Enantioselective Pd-Catalyzed α -Arylation of *N*-Boc-Pyrrolidine: The Key to an Efficient and Practical Synthesis of a Glucokinase Activator

Artis Klapars,* Kevin R. Campos,* Jacob H. Waldman, Daniel Zewge, Peter G. Dormer, Cheng-yi Chen

Department of Process Research, Merck Research Laboratories, Rahway, New Jersey 07065

artis_klapars@merck.com

Supporting Information

Table of Contents:

NMR Spectra.....	2
------------------	---

F2 - Acquisition Parameters

Date_ 20051117
 Time 14.10
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 4
 AS 6578.947 Hz
 FIDRES 0.200774 Hz
 AQ 2.4904180 sec
 RG 35.9
 DW 76.000 usec
 DE 6.00 usec
 TE 300.0 K
 DL 0.10000000 sec
 MUREST 0.00000000 sec
 MURK 0.01500000 sec

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

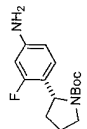
NUC1 1H
 P1 10.50 usec
 PL1 -3.00 dB
 SFO1 400.2522014 MHz

F2 - Processing parameters

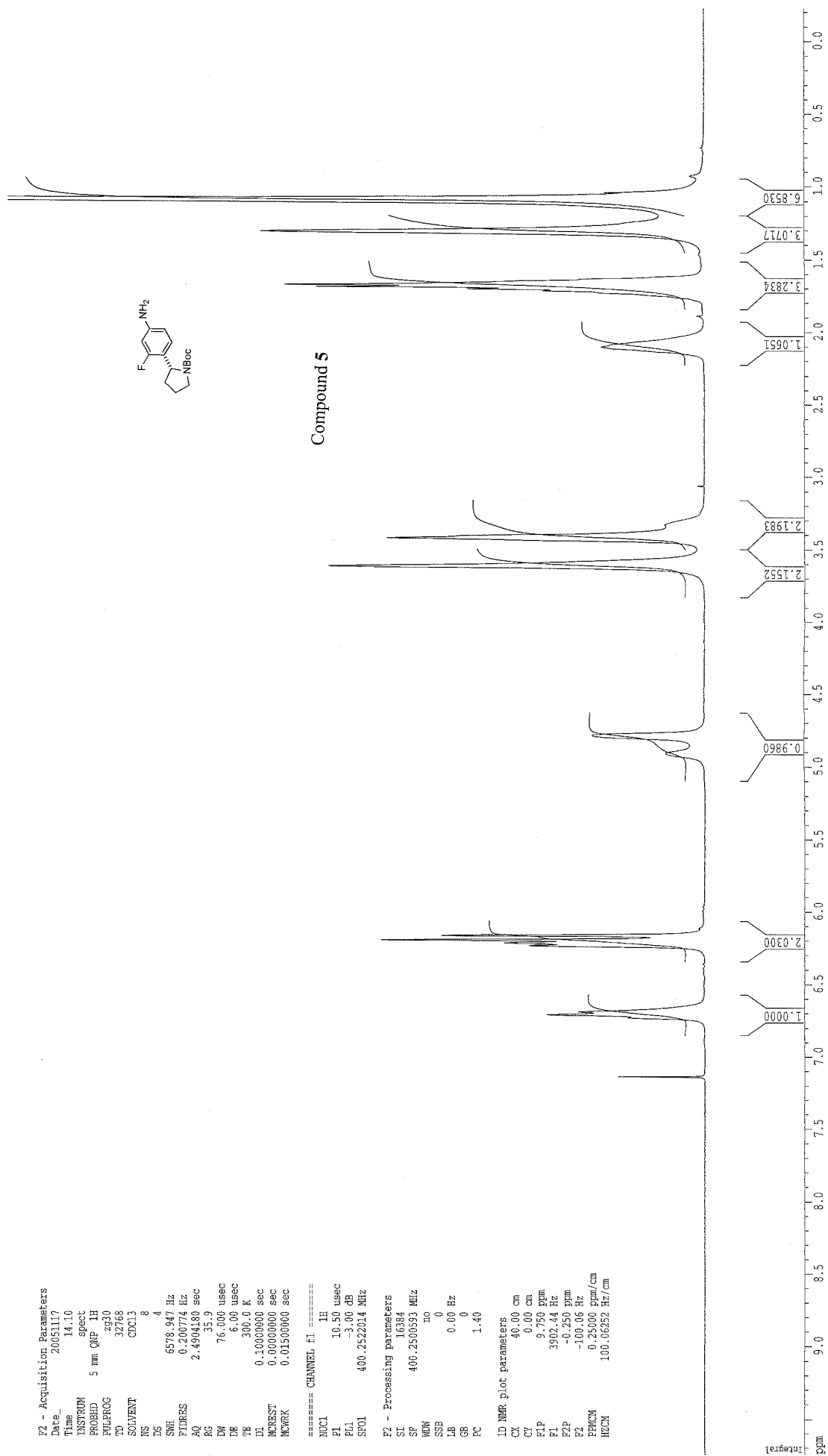
SI 16384
 SF 400.2500593 MHz
 MDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

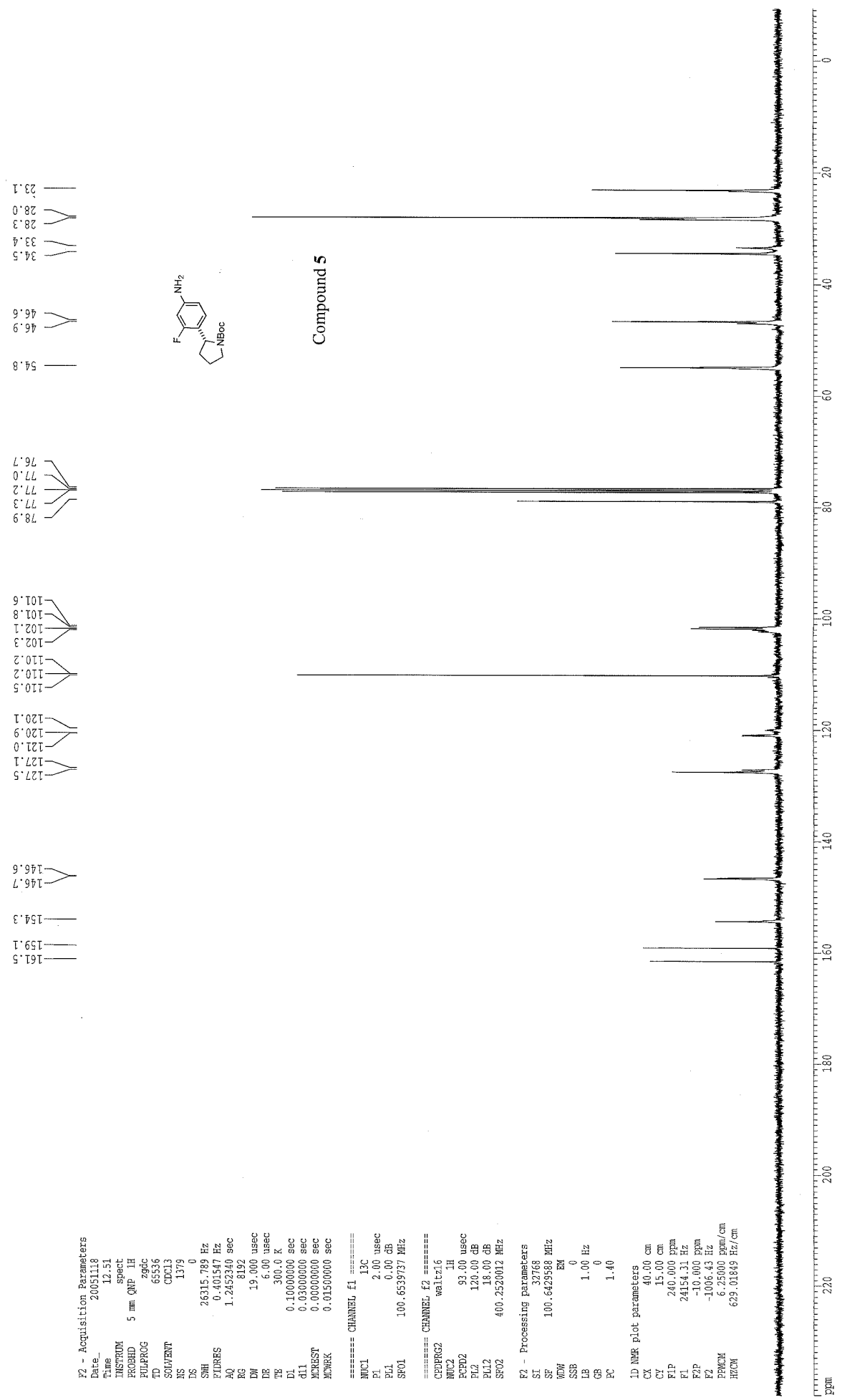
CA 40.00 cm
 CF 0.00 cm
 FIP 9.750 ppm
 FI 3902.44 Hz
 F2P -0.250 ppm
 F2 -100.06 Hz
 PPMCM 0.25000 ppm/cm
 HZCM 100.06252 Hz/cm



Compound 5



nmr400a h-1 decoupled c-13



F2 - Acquisition Parameters
 Date_ 20051118
 Time 12.51
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 65536
 CQ13
 SOLVENT CDCl3
 NS 1379
 DS 0
 SWH 26315.789 Hz
 FIDRES 0.401547 Hz
 AQ 1.2452340 sec
 RG 8192
 LW 19.000 usec
 DE 6.00 usec
 TE 300.0 K
 DL 0.10000000 sec
 d11 0.03000000 sec
 MCHST 0.00000000 sec
 MONK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.00 usec
 PL1 0.00 dB
 SFO1 100.6259737 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCDP2 93.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.2520012 MHz

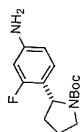
F2 - Processing parameters
 SI 32768
 SF 100.6259588 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 CY 15.00 cm
 P1P 240.000 ppm
 F1 24154.31 Hz
 F2 -10.000 ppm
 F2 -1006.43 Hz
 PPMCM 6.25000 ppm/cm
 HZCM 629.01849 Hz/cm

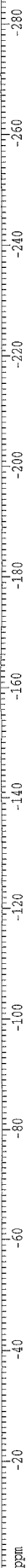
,nmr400a h-1 decoupled f-19

118.93
120.06

F2 - Acquisition Parameters
Date_ 20051117
Time 14.16
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30m
TD 131072
SOLVENT CD3CN
NS 18
DS 4
SWH 112994.352 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 256
IN 4.425 usec
DE 6.32 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
===== CHANNEL f1 =====
NUC1 19F
P1 4.00 usec
PL1 -6.00 dB
SFO1 376.5547800 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz
F2 - Processing parameters
SI 65536
SF 376.6114470 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 40.00 cm
CY 15.00 cm
PTP 0.000 ppm
PI 0.00 Hz
F1 -300.000 ppm
F2 -112983.42 Hz
PPMXX 7.50000 ppm/cm
HZCM 2824.58569 Hz/cm



Compound 5

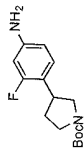


F2 - Acquisition Parameters

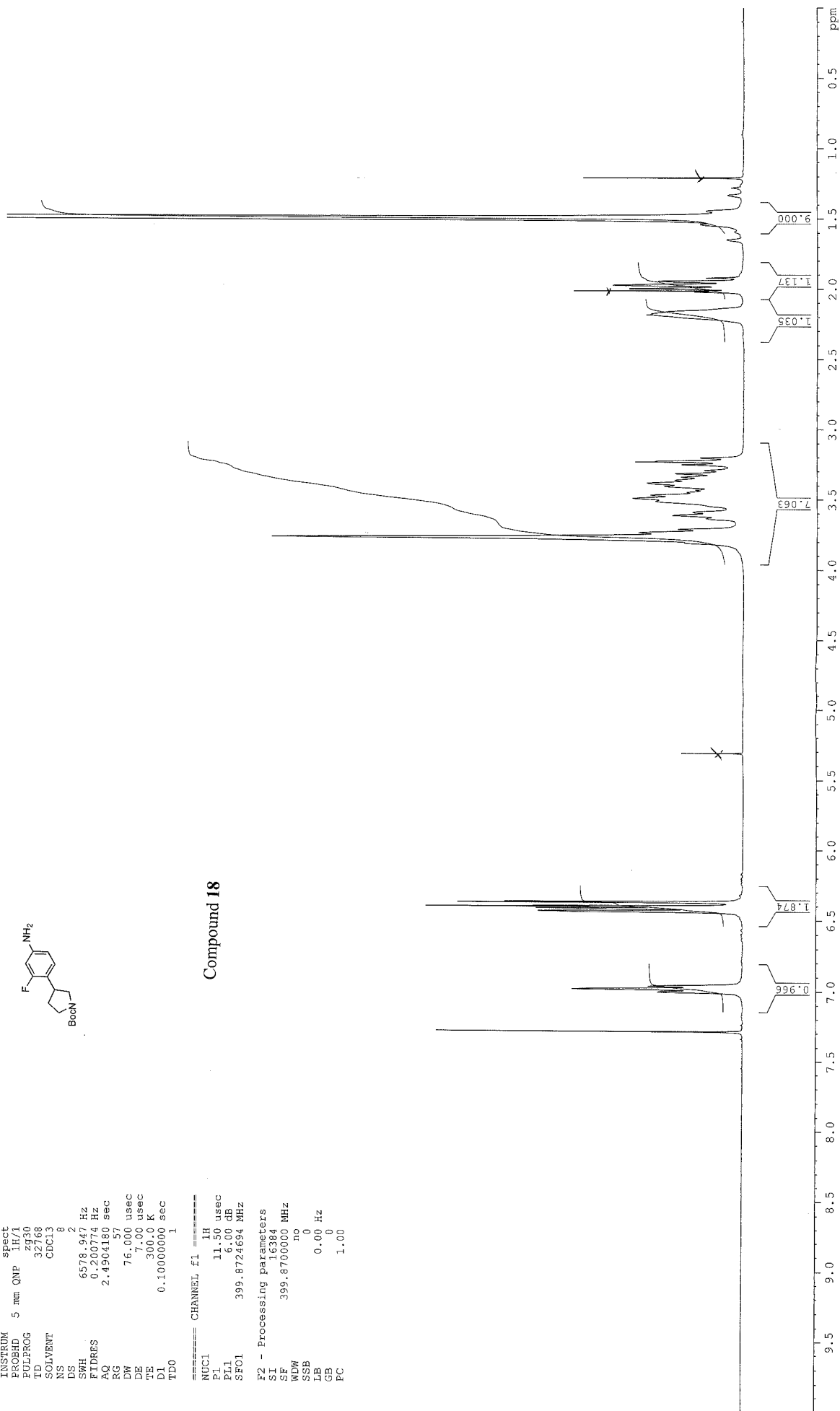
Date_ 20080317
Time_ 11.01
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904190 sec
RG 57
LW 76.000 usec
DE 7.00 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

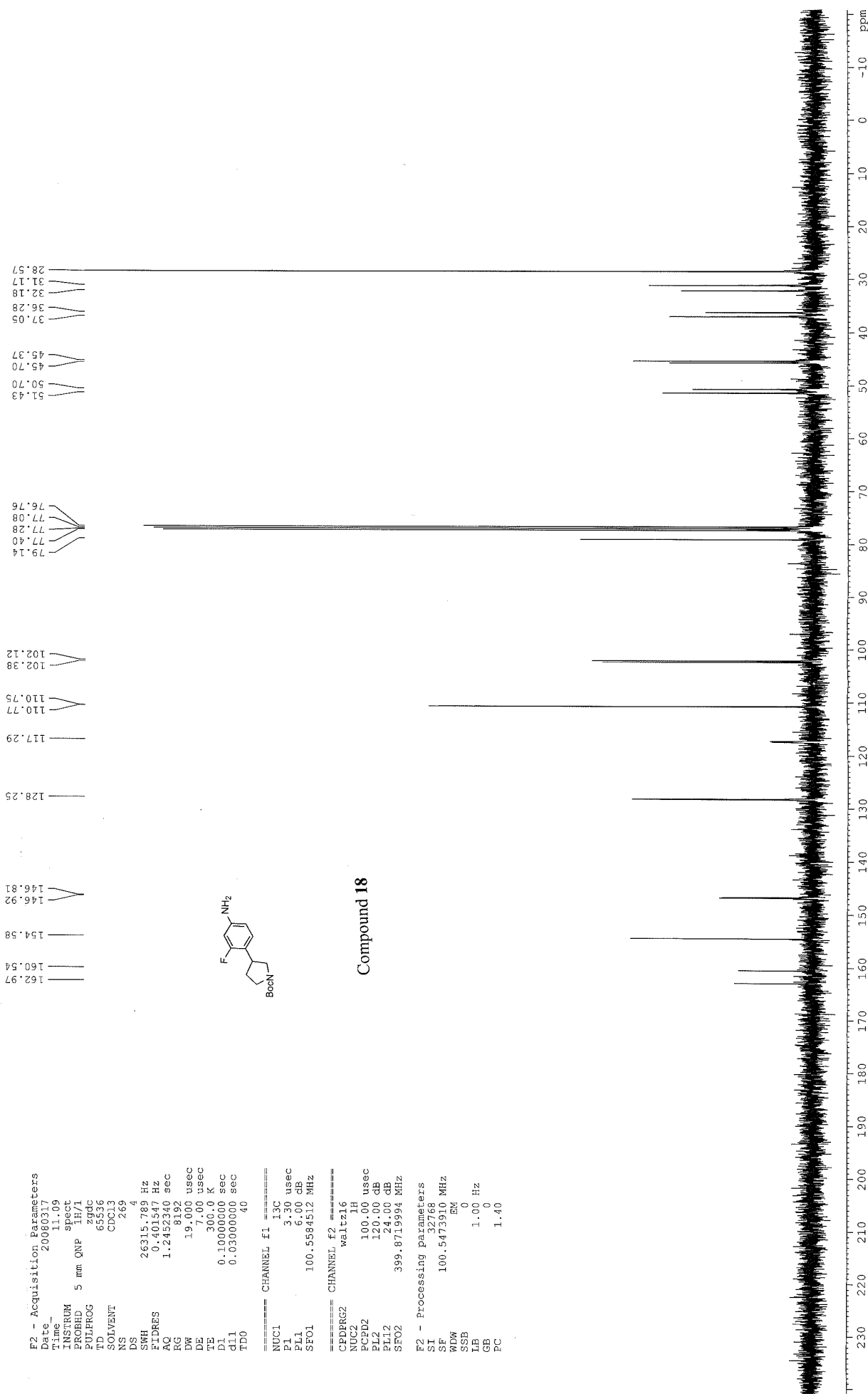
===== CHANNEL f1 =====

NUC1 1H
P1 11.50 usec
PL1 6.00 dB
SFO1 399.8724694 MHz
F2 - Processing parameters
SI 16384
SF 399.8700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



Compound 18





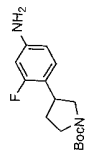
-117.28
-117.14

F2 - Acquisition Parameters
 Date_ 20080317
 Time_ 11.12
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 21
 DS 4
 SWH 112994.352 Hz
 FIDRES 0.862078 Hz
 AQ 0.5800436 sec
 RG 4096
 DW 4.425 usec
 DE 7.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.05000000 sec
 d12 0.0002000 sec
 TDO 1

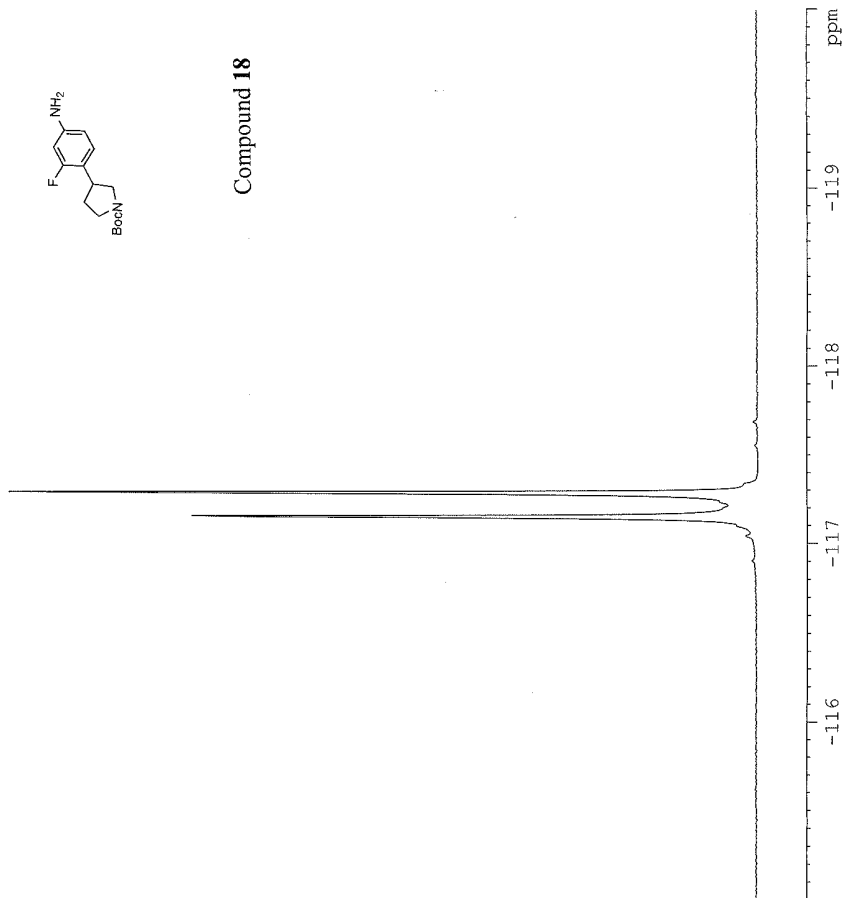
===== CHANNEL f1 =====
 NUC1 19F
 P1 3.00 usec
 PL1 -6.00 dB
 SFO1 376.1972840 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 6.00 dB
 PL12 24.00 dB
 SFO2 399.8719990 MHz

F2 - Processing parameters
 SI 65536
 SF 376.2537220 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.00



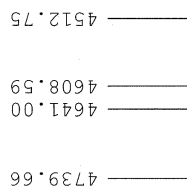
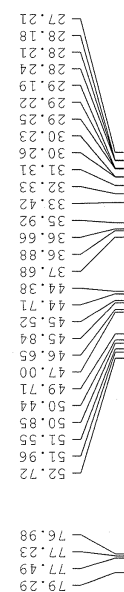
Compound 18



-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250 -260 -270 -280 -290 ppm

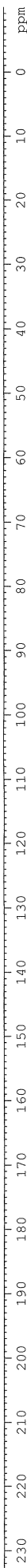
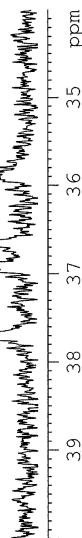
klapars 0280287-0001 cdcl3
nmr500b coupled c-13

Current Data Parameters
NAME Klab0279539-0010
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060317
Time 17.30
INSTRUM spect
PROBHD 5 mm BBI IH-BB
PULPROG zgpg30
TD 65536
FIDRES 0.307637 Hz
AQ 1.625352 sec
RG 8192
DW 12.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 5.00 usec
PL1 0.00 dB
SFO1 125.7049302 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
SFO2 499.8730867 MHz
F2 - Processing parameters
SI 65536
SF 125.6923932 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40

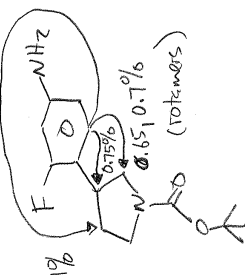


128 Hz

131 Hz



klapars 0280287-0001 cdcl3
nmr500b gradient noe



```

Current Data Parameters
NAME      Klab027959-0010
EXPNO     7
PROCNO    1

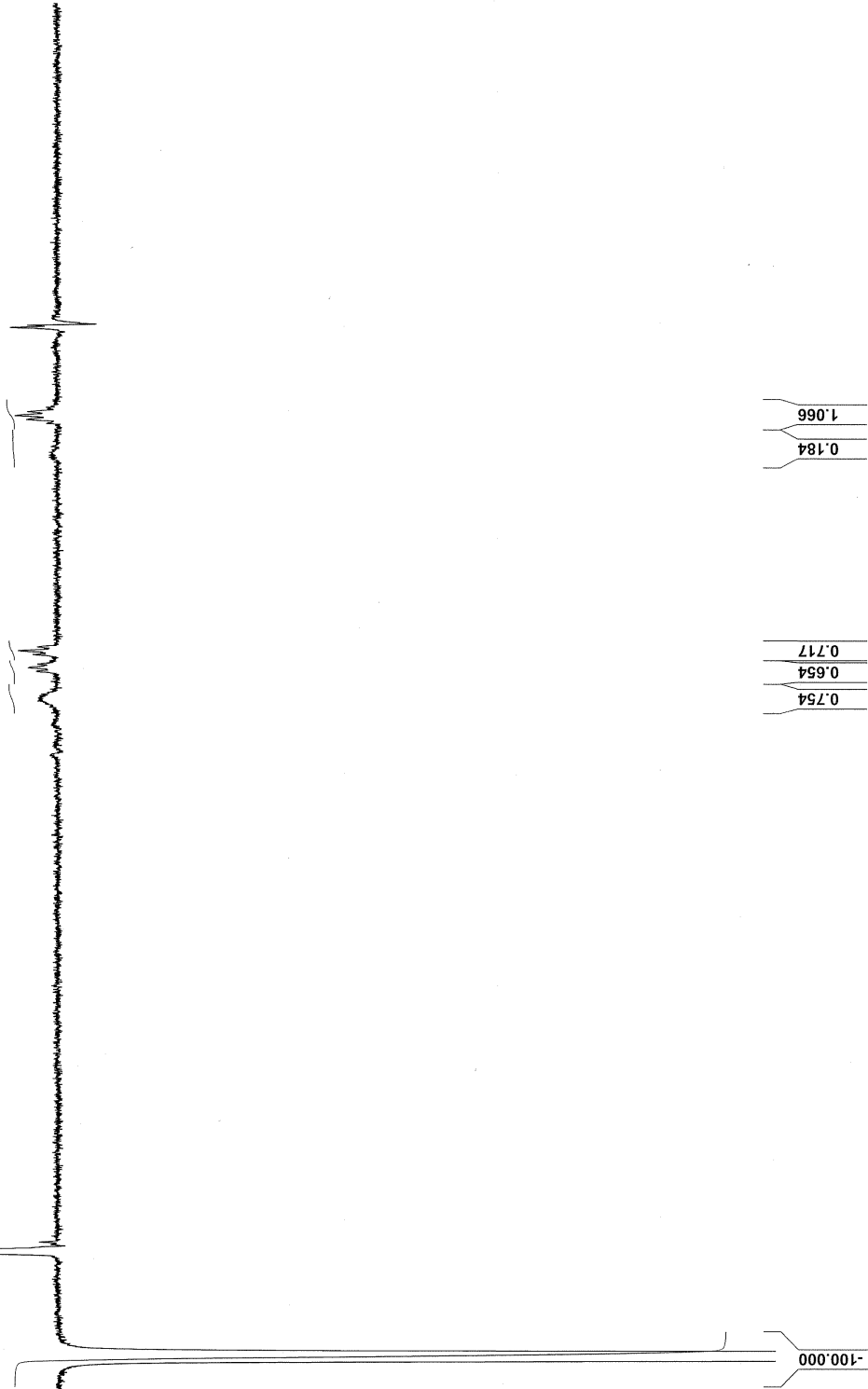
F2 - Acquisition Parameters
Date_     20080317
Time      14.28
INSTRUM   spect
PROBHD    5 mm BBI 1H-BB
PULPROG   selmgp.z2
TD         65536
SOLVENT   CDCl3
NS         128
DS         4
SWH        12376.237 Hz
FIDRES     0.188846 Hz
AQ         2.6477449 sec
RG         16
DW         40.400 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec
D8         0.60000002 sec
D9         0.00020000 sec
D16        0.29860002 sec
GZ0        0.29860002 sec

===== CHANNEL f1 =====
NUC1       1H
P1         9.82 usec
PL1        19.82
P2         19.82 usec
PL2        19.82
P3         125000.00 usec
PL3        125000.00
P4         12.00 dB
PL4        12.00
P5         499.8734922 MHz
PL5        499.8734922
SF          Gauss.100
SFOAL2     0.500 Hz
SFOFFS2    0.00 Hz

===== GRADIENT CHANNEL =====
GENAM1     SINE.100
GENAM2     SINE.100
GENAM3     SINE.100
GEX1       0.00 %
GEX2       0.00 %
GEX3       0.00 %
GEX4       0.00 %
GEX5       0.00 %
GEX6       0.00 %
GEX7       15.00 %
GEX8       40.00 %
GEX9       40.00 %
GEX10      -40.00 %
GEX11      -40.00 %
P16        1000.00 usec

F2 - Processing parameters
SI         32768
SF         499.8700177 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

5-coupled



9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

klapars 0280287-0001 cdcl3
nmr500b gradient noe



```

Current Data Parameters
NAME      Klab0279559-0010
EXNO      2
PROCNO    1

F2 - Acquisition Parameters
Date_     20080317
Time      12.34
INSTRUM    Spect
PROBHD     5 mm BBI 1H-BB
PULPROG    selnpgp2
TD         65536
SOLVENT    CDCl3
NS         128
DS         4
SWH         12376.237 Hz
FIDRES     0.188846 Hz
AQ         2.6477442 sec
RG         16
DE         40.400 usec
TE         300.0 K
DT         6.00 usec
D1         1.00000000 sec
D2         0.60000002 sec
D3         0.00200006 sec
D4         0.29880002 sec
===== CHANNEL f1 =====
NUC1       1H
P1         9.82 usec
P2         18.85 usec
P12        125000.00 usec
PL1        120.00 dB
PL2        120.00 dB
SFO1       499.8732134 MHz
SF02       71.100 dB
SFOAL2     Gauss, 1000
SFOFFS2    0.500
SFOFFS2    0.00 Hz

===== GRADIENT CHANNEL =====
GNUM1      SINE, 100
GNUM2      SINE, 100
GNUM3      SINE, 100
GFX1       0.00 %
GFX2       0.00 %
GFX3       0.00 %
GFX4       0.00 %
GFX5       0.00 %
GFX6       0.00 %
GFX7       15.00 %
GFX8       40.00 %
GFX9       -40.00 %
GFX10      1000.00 usec

F2 - Processing parameters
SI          32768
SF          499.8700112 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00

```

5-coupled

~NH2

1.882

2.668

-100.000



klapars 0280287-0001 cdcl3
nmr500b gradient cosy-45

```

Current Data Parameters
NAME      klab0279559-0010
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20080317
Time      13.04
INSTRUM   spect
PROBHD    5 mm BBI-HR-BB
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         2
DS         8
SWH        3501.401 Hz
FIDRES     1.709668 Hz
AQ         0.2926472 sec
RG         256
DE         142.800 usec
TE         300.0 K
d0         0.0000300 sec
d1         1.0000000 sec
d13        0.0000400 sec
d16        0.0002000 sec
IN0        0.00028560 sec

===== CHANNEL f1 =====
NUC1       1H
P1         12.00 usec
PL1        0.00 dB
SFO1       499.8721494 MHz

===== GRADIENT CHANNEL =====
GENDAM1    SINE.100
GENDAM2    SINE.100
GEX1       0.00 %
GEX2       0.00 %
GEZ1       0.00 %
GEZ2       0.00 %
GPR1       10.00 %
GPR2       10.00 %
P16        1000.00 usec

F1 - Acquisition Parameters
ND0        1
TD         256
SFO1       499.8721 MHz
FIDRES     13.677346 Hz
SWH        7.005 ppm
RG         256
DE         142.800 usec
TE         300.0 K
d0         0.0000300 sec
d1         1.0000000 sec
d13        0.0000400 sec
d16        0.0002000 sec
IN0        0.00028560 sec

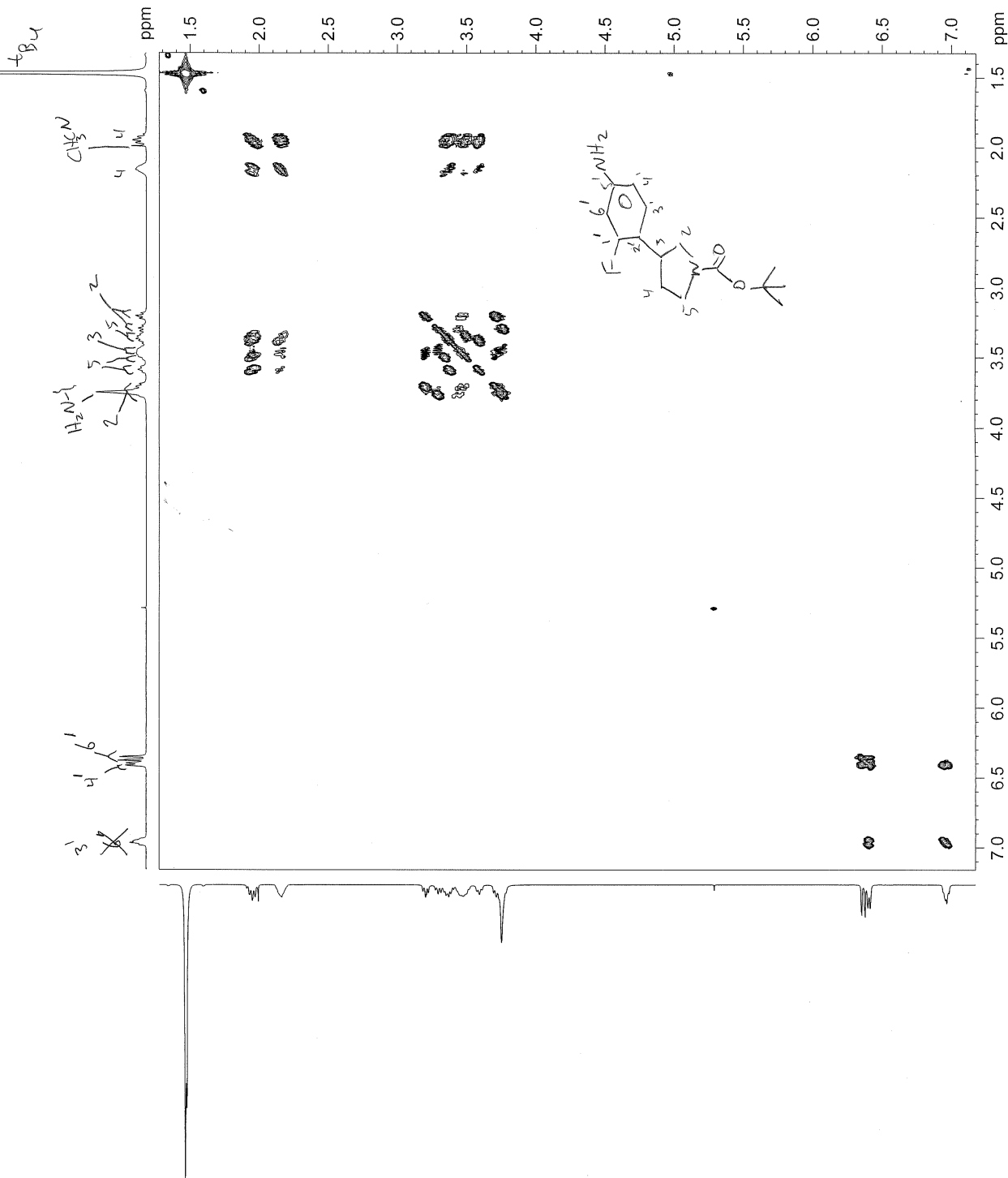
===== CHANNEL f2 =====
NUC2       13C
P2         12.00 usec
PL2        0.00 dB
SFO2       100.6261200 MHz

===== GRADIENT CHANNEL =====
GENDAM1    SINE.100
GENDAM2    SINE.100
GEX1       0.00 %
GEX2       0.00 %
GEZ1       0.00 %
GEZ2       0.00 %
GPR1       10.00 %
GPR2       10.00 %
P16        1000.00 usec

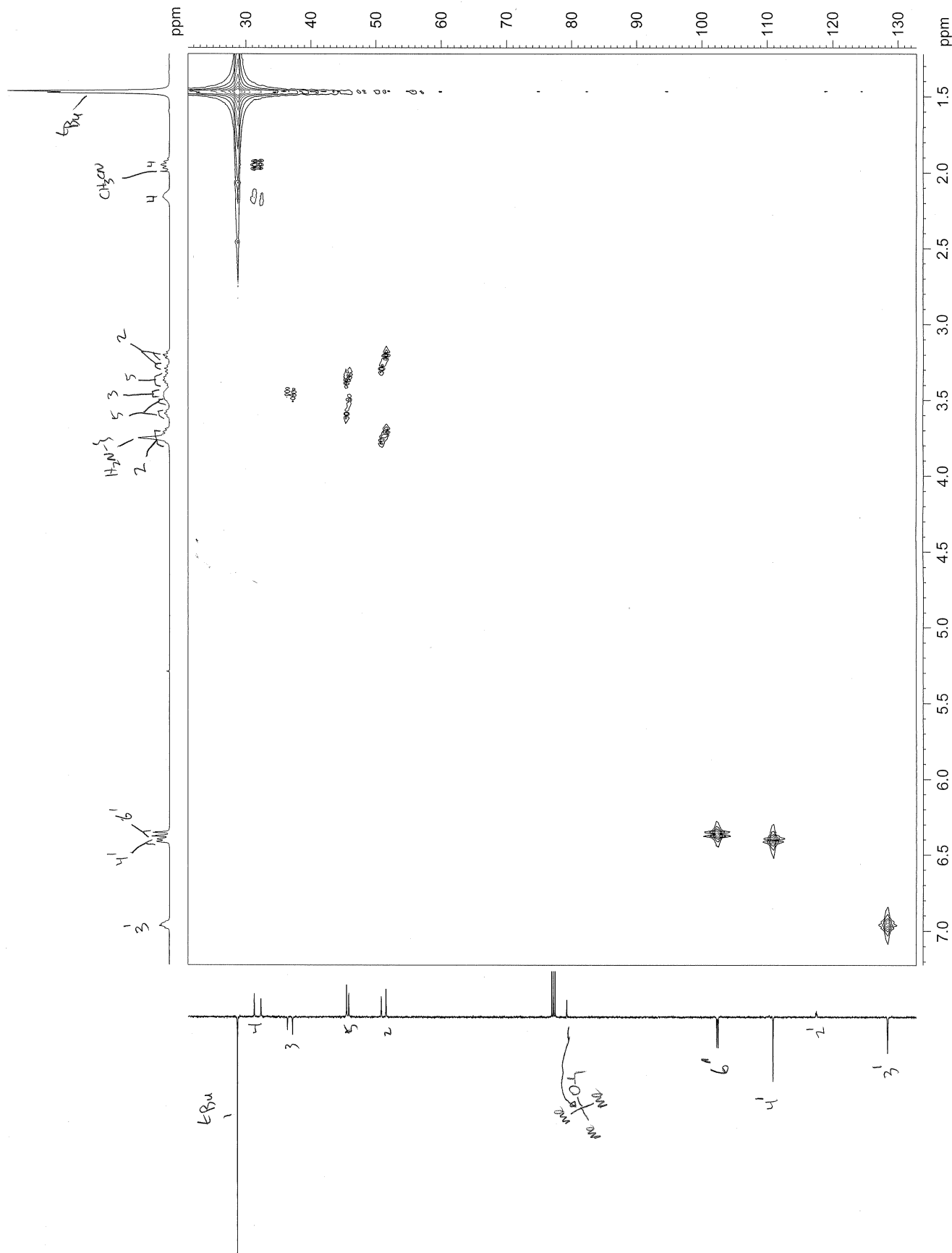
F2 - Processing Parameters
SI         32768
SF         100.6261200 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing Parameters
SI         32768
SF         499.8700099 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

```



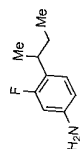
klapars 0280287-0001 cdcl3
nmr500b gradient hmqc



Current Data Parameters
NAME K1ab0279559-0010
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080317
Time 13.16
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG hmqcpar
TD 4096
SOLVENT CDCl3
NS 16
DS 2
SWH 3501.401 Hz
FIDRES 0.854834 Hz
AQ 0.5851016 sec
RG 327.500
DE 142.800 usec
TE 300.1 K
CNS2 145.0000000
DO 0.0000300 sec
DZ 1.0000000 sec
D1 0.0002000 sec
d12 0.0002000 sec
d13 0.0000400 sec
d16 0.0001000 sec
DELTA1 0.0023248 sec
IN0 0.0000248 sec
===== CHANNEL f1 =====
NUC1 1H
P1 9.82 usec
P2 19.65 usec
PL1 1.00 dB
SFO1 499.8721494 MHz
===== CHANNEL f2 =====
CDEPRG2 garp
NUC2 13C
P3 14.50 usec
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.00 dB
SFO2 125.7024154 MHz
===== GRADIENT CHANNEL =====
GPM1 SINE.100
GPM2 SINE.100
GPM3 SINE.100
GFX1 0.00 %
GFX2 0.00 %
GFX3 0.00 %
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPZ1 0.00 %
GPZ2 0.00 %
GPZ3 0.00 %
P16 40.10 %
P16 1000.00 usec
F1 - Acquisition Parameters
NUC1 1H
TD 256
SFO1 125.7024 MHz
FIDRES 78.596581 Hz
SW 160.066 ppm
FMODE QF
F2 - Processing Parameters
SI 2048
SF 499.8700105 MHz
WDW QSI
LB 2
GB 0.00 Hz
PC 1.40
F1 - Processing Parameters
SI 512
MC2 0F
SF 125.6923758 MHz
WDW QSI
LB 2
GB 0.00 Hz

2587.
2585.
2579.
2576.
2562.
2560.
2550.
2548.

Compound 19



2807
2798
2790

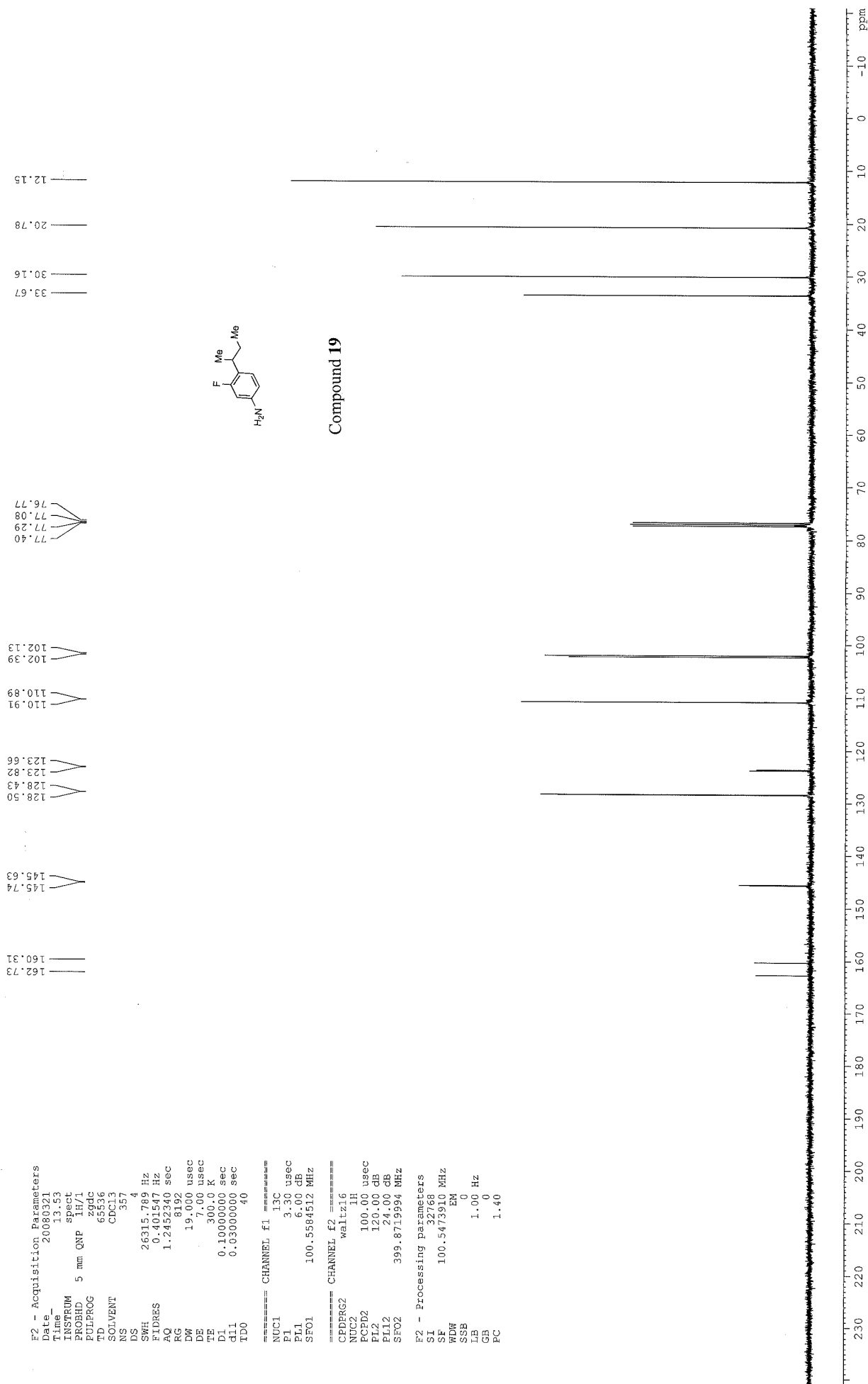
F2 - Acquisition Parameters
Date_ 20080321
Time 13.43
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.450418 sec
RG 222
DE 76.000 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 11.50 usec
PL1 6.00 dB
SFO1 399.8724694 MHz
F2 - Processing parameters
SI 16384
SF 399.8700000 MHz
WDW no
SSB 0
LB 0
GB 0
PC 1.00

7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5 3.4 3.3 3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8 0.7 0.6 0.5 0.4 0.3 0.2 0.1 0.0

1.003
0.975
0.960

2.111
2.173
3.249
3.168

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm



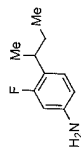
-118.78

```
F2 - Acquisition Parameters
Date_ 20080321
Time 13.55
INSTRUM 5 mm QNP spect
PROBHD IH/1
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 13
DS 4
SH 112994.352 Hz
AQ 0.862076 Hz
RG 0.3900436 sec
EG 4096
DE 4.425 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec
TD0 1

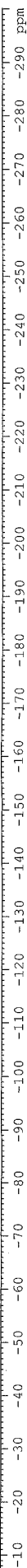
===== CHANNEL f1 =====
NUC1 19F
P1 3.00 usec
PL1 -6.00 dB
SFO1 376.1972840 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 6.00 dB
PL12 24.00 dB
SFO2 399.8719990 MHz

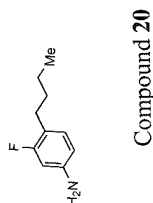
F2 - Processing parameters
SI 65536
SF 376.2537220 MHz
WDW EM
SSB 0
GB 0.20 Hz
PC 1.40
```



Compound 19



nmr400b h-1



Compound 20

F2 - Acquisition Parameters

Date_ 20070430
Time 9.09
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 30.6
IN 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MTEK 0.01500000 sec

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

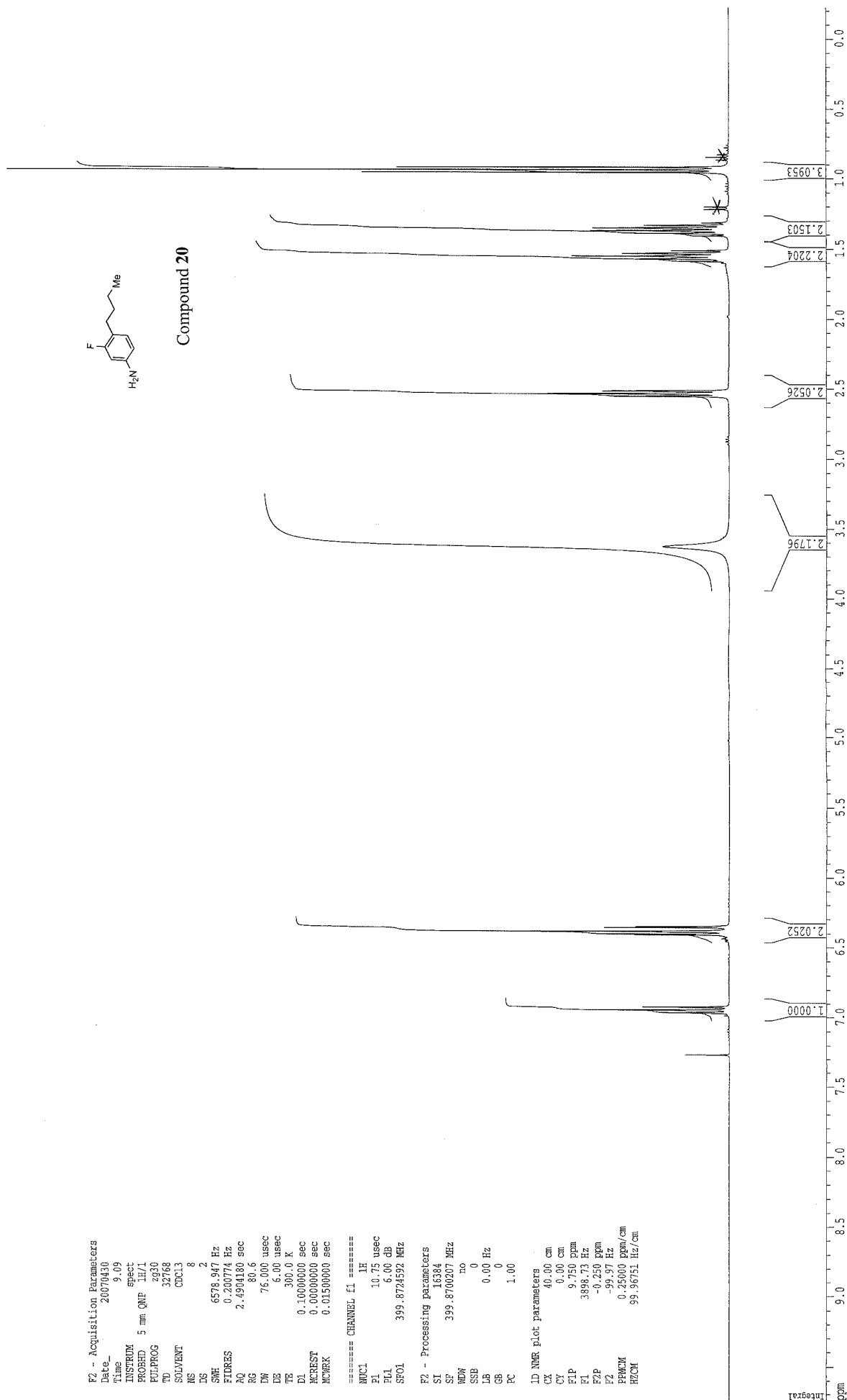
MUCL 1H
P1 10.75 usec
PL1 6.00 dB
SF01 399.874592 MHz

F2 - Processing parameters

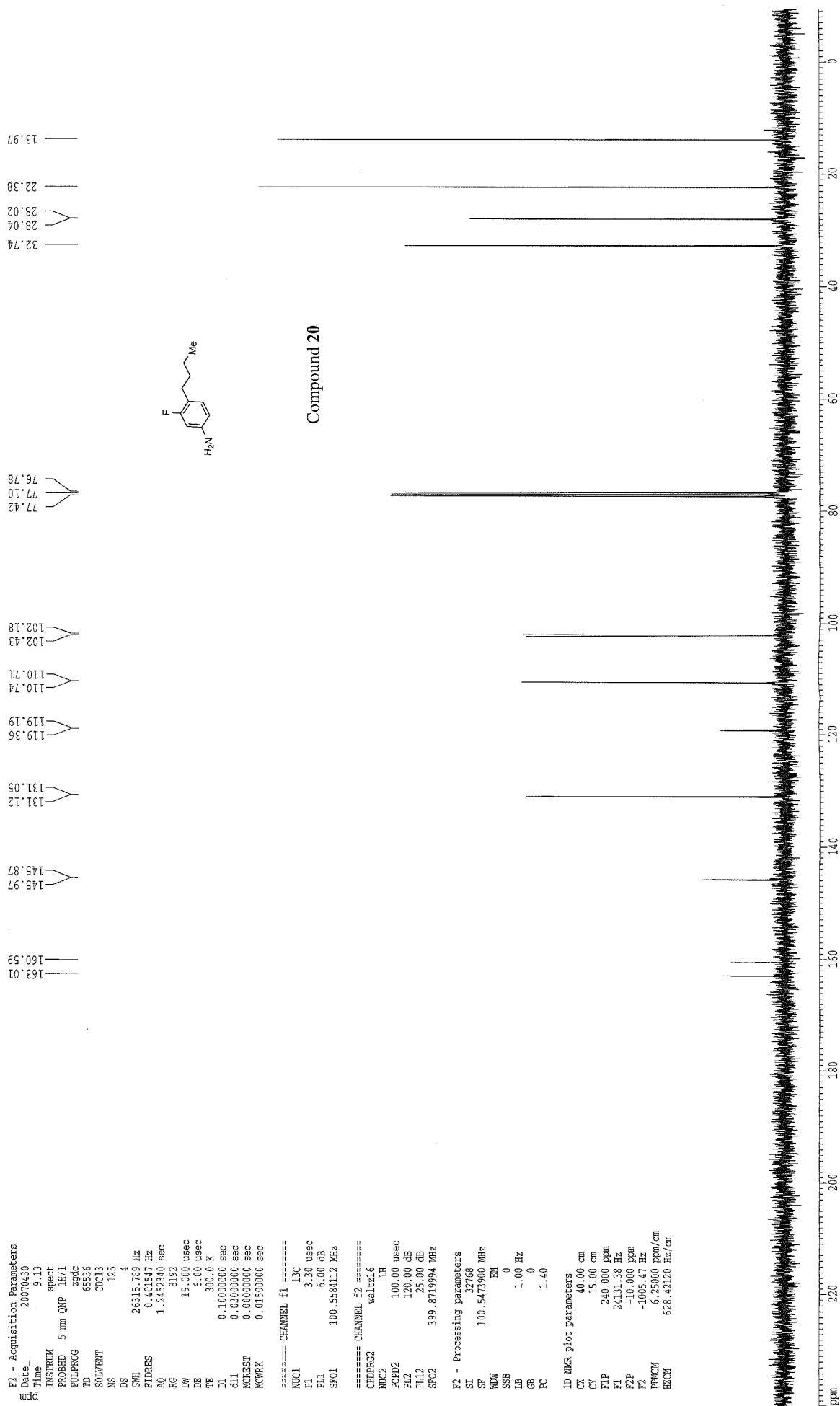
SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

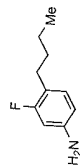
CX 40.00 cm
CY 0.00 cm
FLP 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
FNUC 0.25000 ppm/cm
HZCM 99.96751 Hz/cm



nmr400b c-13 (h-1 decoupled)



nmr400b f-19 (h-1 decoupled)



Compound 20

-119.1

```
=====
KJ - Acquisition Parameters
=====
Date_      20070430
Time       9.15
INSTRUM    spect
PROBHD     5 mm QNP 1H/1
PULPROG    zgpg30
TD          131072
SOLVENT    CDCl3
NS          16
DS          4
SWH         112994.352 Hz
FIDRES      0.862078 Hz
AQ          0.5890436 sec
RG          512
RG          4.425 usec
DE          5.42 usec
TE          300.1 K
D1          0.1000000 sec
d11         0.0300000 sec
d12         0.0002000 sec

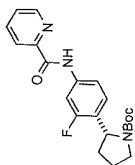
===== CHANNEL f1 =====
NUC1        19F
P1          3.00 usec
PL1         -6.00 dB
SFO1        376.1974519 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       100.00 usec
PL2         120.00 dB
PL12        18.00 dB
SFO2        399.8719994 MHz

F2 - Processing parameters
SI          65536
SF          376.2539310 MHz
WDM         EN
SSB         0
LB          0.20 Hz
GB          0
PC          1.00

1D NMR plot parameters
CX          40.00 cm
CY          15.00 cm
FIP         0.000 ppm
F1          0.00 Hz
F2P         -300.000 ppm
F2          -112876.17 Hz
PPMCM       7.50000 ppm/cm
HZCM        2821.90430 Hz/cm
```





Compound 21

F2 - Acquisition Parameters

Date_ 20051108
Time 14.26
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SHF 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 80.6
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

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

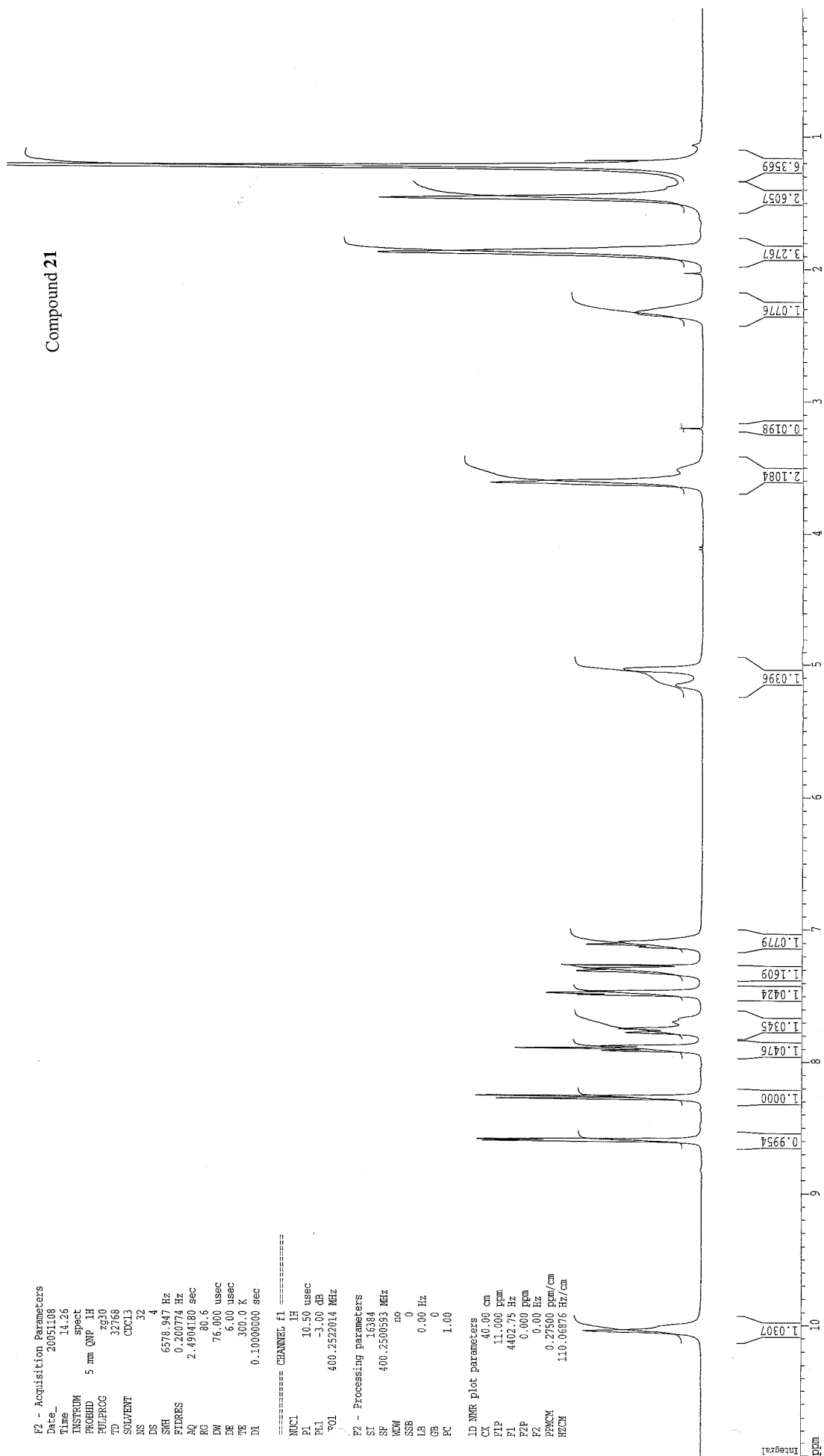
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
FO1 400.252014 MHz

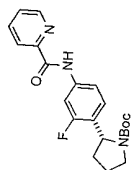
F2 - Processing parameters

SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 40.00 cm
P1P 11.000 ppm
F1 4402.75 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.27500 ppm/cm
HZCM 110.06876 Hz/cm





Compound 21



F2 - Acquisition Parameters

Date_ 20051108
Time 17.28
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 20000
DS 0
SS 26315.789 Hz
SMH 0.401547 Hz
FIDRES 1.2452340 sec
AQ 8192
RG 19.000 usec
LW 6.00 usec
DE 300.0 K
TE 0.10000000 sec
D1 0.03000000 sec
d11 0.03000000 sec

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

NUC1 13C
P1 2.00 usec
PL 0.00 dB
F01 100.6539737 MHz

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

CPDPRG2 waltz16
NUC2 1H
P2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.2520012 MHz

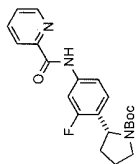
F2 - Processing parameters

SI 32768
SF 100.6429588 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 40.00 cm
F1P 240.000 ppm
F1 24154.31 Hz
P2P -10.000 ppm
F2 -1006.43 Hz
P2MCM 6.25000 ppm/cm
HZCM 629.01849 Hz/cm





Compound 21

116.90
117.96

F2 - Acquisition Parameters

Date_ 20051108
Time 14.56
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl₃
NS 143
DS 4
F2 112994.352 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 5792.6
DW 4.425 usec
DE 6.32 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

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

NUC1 15P
P1 197
PL1 4.00 usec
PL1 -6.00 dB
SFO1 376.5547800 MHz

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

CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

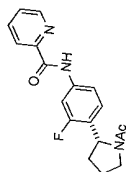
F2 - Processing parameters

SI 6556
SF 376.6114470 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 40.00 cm
CY 15.00 cm
F1P 0.000 32mm
F1 0.00 Hz
F2P -300.000 ppm
F2 -112983.42 Hz
PPMCM 7.50000 ppm/cm
HZCM 2824.58569 Hz/cm

ppm -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280



F2 - Acquisition Parameters

Date 20051111

Time 11.51

INSTRUM spect

PROBHD 5 mm QNP 1H

PULPROG zgpg30

TD 32768

SOLVENT CDCl3

NS 32

DS 4

SS 6578.947 Hz

SFRS 0.200774 Hz

AQ 2.4904180 sec

RG 71.8

DW 76.000 usec

DE 6.00 usec

TE 300.0 K

D1 0.10000000 sec

MCREST 0.00000000 sec

MORPH 0.01500000 sec

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

NUC1 1H

AQ 10.50 usec

RG -3.00 dB

SF01 400.2522014 MHz

F2 - Processing parameters

SI 16384

SF 400.2500593 MHz

WDW no

SSB 0

LB 0.00 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 40.00 cm

CY 0.00 cm

FLP 11.000 ppm

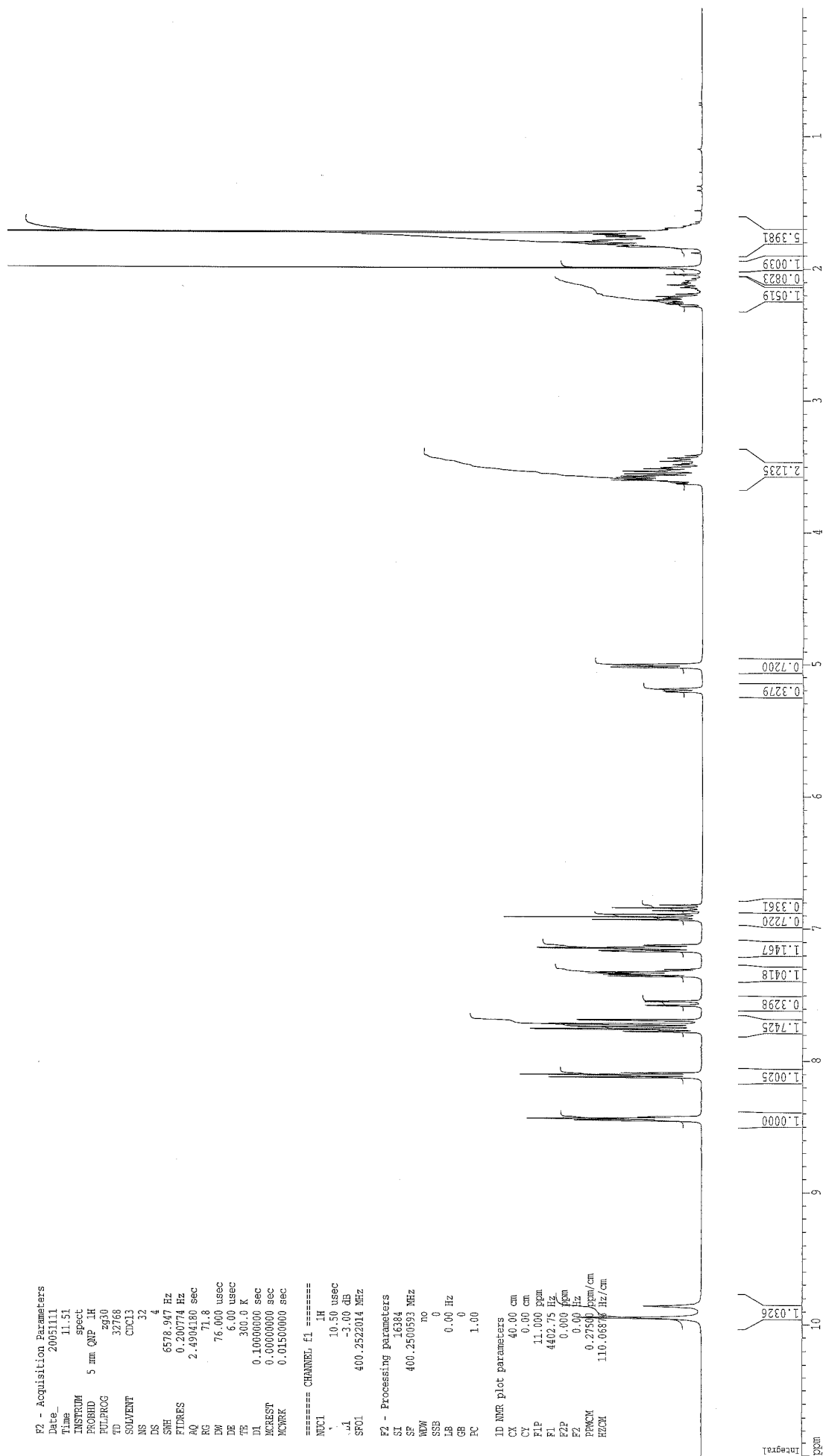
F1 4402.75 Hz

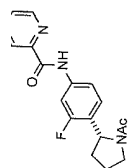
F2P 0.000 ppm

F2 0.00 Hz

PPMCM 0.27500 ppm/cm

HZCM 110.06876 Hz/cm





Current Data Parameters
NAME 224457-39s
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters

Date_ 20051114

Time 11.03

INSTRUM spect

PROBHD 5 mm QNP 1H

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 715

DS 0

SWH 26315.789 Hz

FIDRES 0.401547 Hz

AQ 1.2452340 sec

RG 3251

DM 19.000 usec

DE 6.00 usec

TE 300.0 K

TD 0.1000000 sec

DI 0.0300000 sec

DEL 0.0000000 sec

ACQRES 0.0150000 sec

PC 0.0150000 sec

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

NUC1 13C

PC1 2.00 usec

PL1 0.00 dB

SFO1 100.6539737 MHz

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

CPDPRG2 waltz16

NUC2 1H

PCPD2 93.00 usec

PL2 120.00 dB

SFO2 400.2520012 MHz

F2 - Processing parameters

SI 32768

SF 100.6429583 MHz

WDW no

SSB 0

LB 0.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 40.00 cm

CY 15.00 cm

F1P 240.000 ppm

F1 24154.31 Hz

F2P -10.000 ppm

F2 -1006.43 Hz

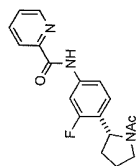
PPMCM 6.25000 ppm/cm

WCM 629.01849 Hz/cm

77.3
77.0
76.6
56.1
56.3
55.2
48.2
46.6
34.6
32.7
23.8
22.6
22.2
21.9

169.9
169.8
169.0
167.0
160.9
160.6
158.4
158.2
149.3
149.1
147.9
147.8
138.4
138.3
137.7
137.6
137.5
127.0
126.9
126.8
126.7
126.6
126.4
125.5
125.4
125.4
122.3
122.3
115.1
115.1
114.6
114.6
107.5
107.4
107.2
107.2

ppm



-116.19
-116.78

F2 - Acquisition Parameters

Date_ 20051114
Time 11.00
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 169
DS 4
SS 112994.352 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 2896.3
LW 4.425 usec
DE 6.32 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

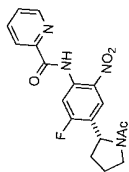
===== CHANNEL f1 =====
NUC1 13C
P1 4.00 usec
PL1 -6.00 dB
SFO1 376.5547800 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 65536
SF 376.6114470 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
CY 15.00 cm
FLP 0.000 ppm
F1 0.00 Hz
F2P -300.000 ppm
F2 -112983.42 Hz
PFGM 7.50000 ppm/cm
HSCN 2824.58569 Hz/cm





Compound 11

F2 - Acquisition Parameters

Date_ 20051104
Time 13.27
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 71.8
LW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

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

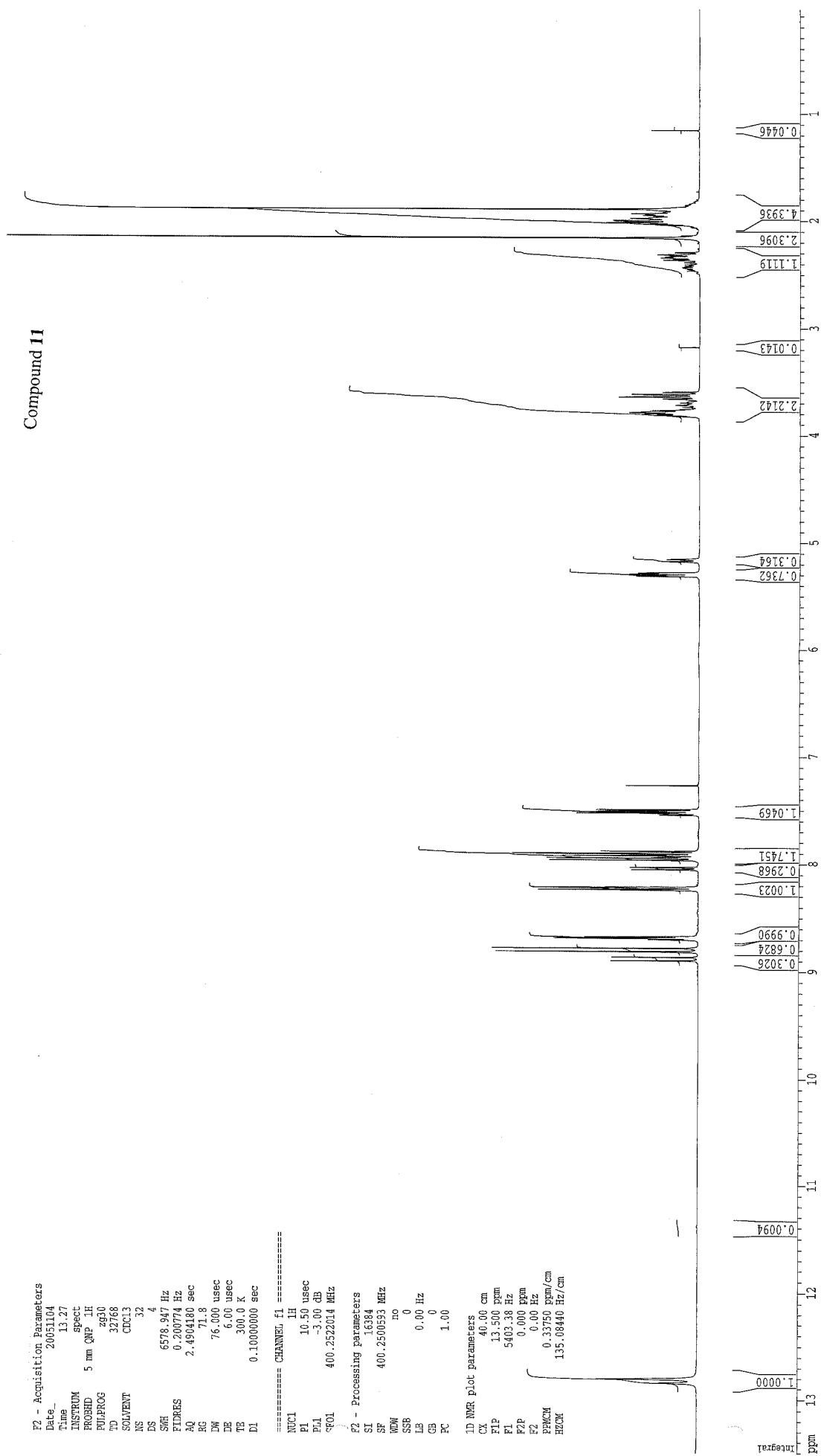
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters

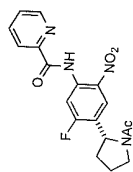
SI 16384
SF 400.2500553 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

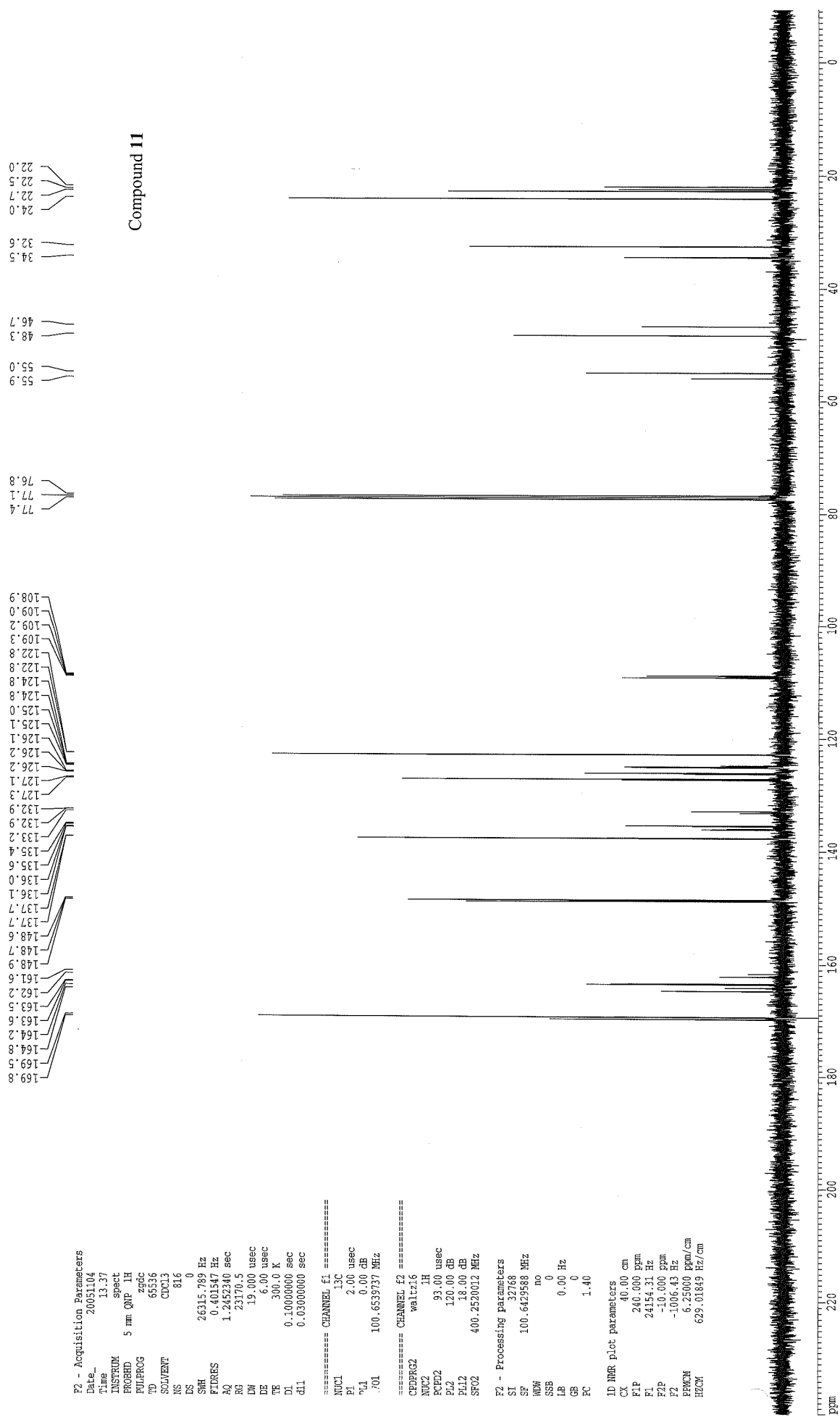
CX 40.00 cm
F1P 13.500 ppm
F1 5403.38 Hz
F2P 0.000 ppm
F2 0.00 Hz
FPCW 0.33750 ppm/cm
HZCN 135.08440 Hz/cm

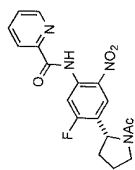


Integral



Compound 11





Compound 11

102.97
103.80

F2 - Acquisition Parameters

Date_ 20051104
Time 13.52
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30m
TD 131072
SOLVENT CDCl3
NS 103
DS 4
SWH 112994.352 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 2896.3
RW 4.425 usec
DE 6.32 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

NUC1 13F
P1 4.00 usec
PA 6.00 dB
SFO1 376.5547800 MHz

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

CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters

SI 65536
SF 376.6114470 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 40.00 cm
F1P 0.000 ppm
F1 0.00 Hz
F2P -300.000 ppm
F2 -112983.42 Hz
FREQM 7.50000 ppm/cm
HZCM 2824.58569 Hz/cm

ppm

-20

-40

-60

-80

-100

-120

-140

-160

-180

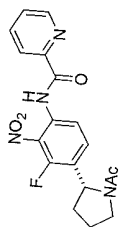
-200

-220

-240

-260

-280



Compound 23

F2 - Acquisition Parameters

Date 20051116
Time 9.40
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SS 6578.947 Hz
SMH 0.200774 Hz
FIDRES 2.4904180 sec
AQ 143.7
RG 76.000 usec
LW 6.00 usec
DE 300.0 K
TE 0.10000000 sec
D1 0.00000000 sec
MCREST 0.01500000 sec
MORCK 0.01500000 sec

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

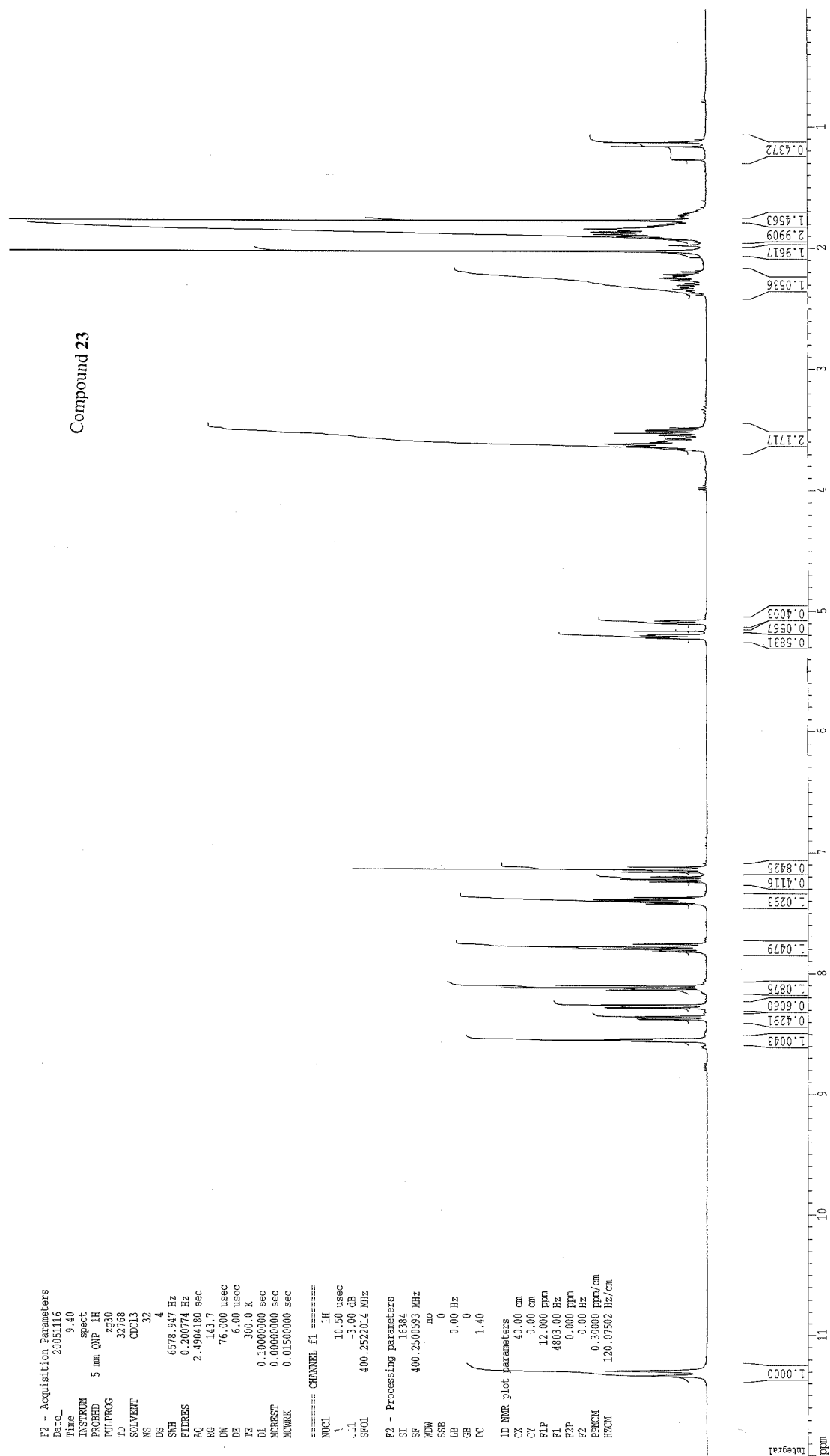
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SF01 400.2522014 MHz

F2 - Processing parameters

SI 16384
SF 400.250593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

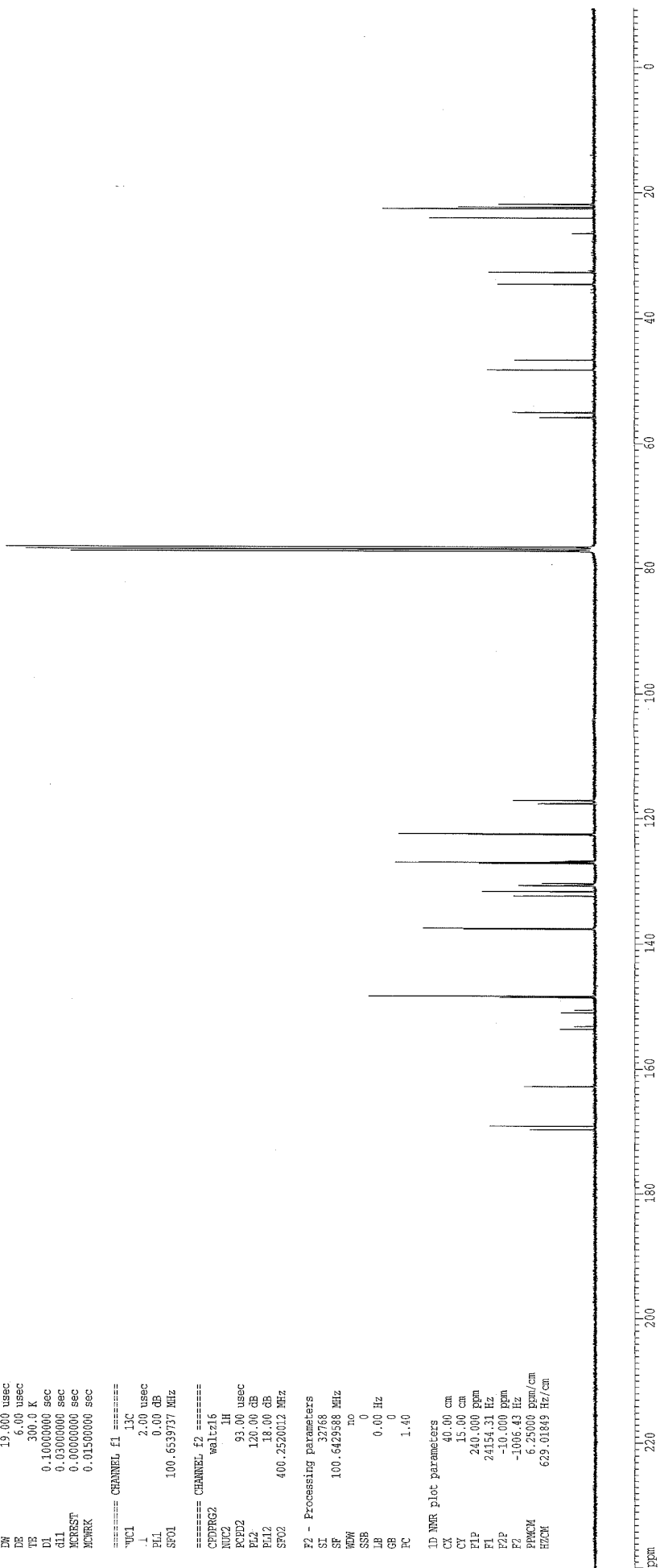
CX 40.00 cm
CY 0.00 cm
FIP 12.000 ppm
F1 4803.00 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.30000 ppm/cm
HCM 120.07502 Hz/cm

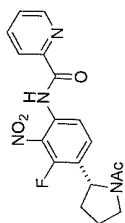


Integral

Age Group	1990-1994 (%)	1995-1999 (%)
18-29	21.9	26.5
30-49	22.3	24.0
50-69	22.6	24.0
70+	32.7	34.6

Year	Population (millions)
1980	46.7
1985	48.2
1990	55.0
1995	55.1
2000	55.8
2005	55.9





Compound 23

124.26
124.60

```

F2 - Acquisition Parameters
Date_      20051116
Time       11.55
INSTRUM    spect
PROBHD     5 mm QNP 1H
PULPROG    zgpg30
TD         131072
SOLVENT    CDCl3
NS         152
DS         4
SWH         112994.352 Hz
FIDRES     0.862078 Hz
AQ         0.5800436 sec
RG         2580.3
DN         4.425 usec
DE         6.32 usec
TE         300.0 K
D1         0.10000000 sec
d11        0.03000000 sec
d12        0.00002000 sec

===== CHANNEL f1 =====
NUC1       13F
P1         4.00 usec
PL1        -6.00 dB
SFO1       376.5547800 MHz

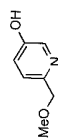
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      93.00 usec
PL2        120.00 dB
PL12       18.00 dB
SFO2       400.2520012 MHz

F2 - Processing parameters
SI         65536
SF         376.6114470 MHz
WDW        ho
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

1D NMR plot parameters
CX         40.00 cm
CY         15.00 cm
FLP        0.000 KHz
FI         0.00 Hz
F2P        -300.000 ppm
F2         -112983.42 Hz
PPHXX      7.50000 ppm/cm
HZCX       2824.58569 Hz/cm
  
```

ppm -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280

nmr400b h-1



Compound 9

F2 - Acquisition Parameters

Date_ 20070502

Time 9:31

INSTRUM spect

PROBHD 5 mm QNP 1H/1

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 8

DS 2

SWH 6578.947 Hz

FIDRES 0.200774 Hz

AQ 2.4904180 sec

RG 181

RW 76.600 usec

DS 8.00 usec

TE 300.0 K

D1 0.10000000 sec

MCREST 0.00000000 sec

MCWRR 0.01500000 sec

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

NUC1 1H

P1 10.75 usec

PL1 6.00 dB

SFO1 399.8724592 MHz

F2 - Processing parameters

SI 16384

SF 399.8700207 MHz

WDW no

SSB 0

LB 0.00 Hz

GB 0

PC 1.00

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

F2 - Processing parameters

SI 16384

SF 399.8700207 MHz

WDW no

SSB 0

LB 0.00 Hz

GB 0

PC 1.00

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

F2 - Processing parameters

SI 16384

SF 399.8700207 MHz

WDW no

SSB 0

LB 0.00 Hz

GB 0

PC 1.00

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

F2 - Processing parameters

SI 16384

SF 399.8700207 MHz

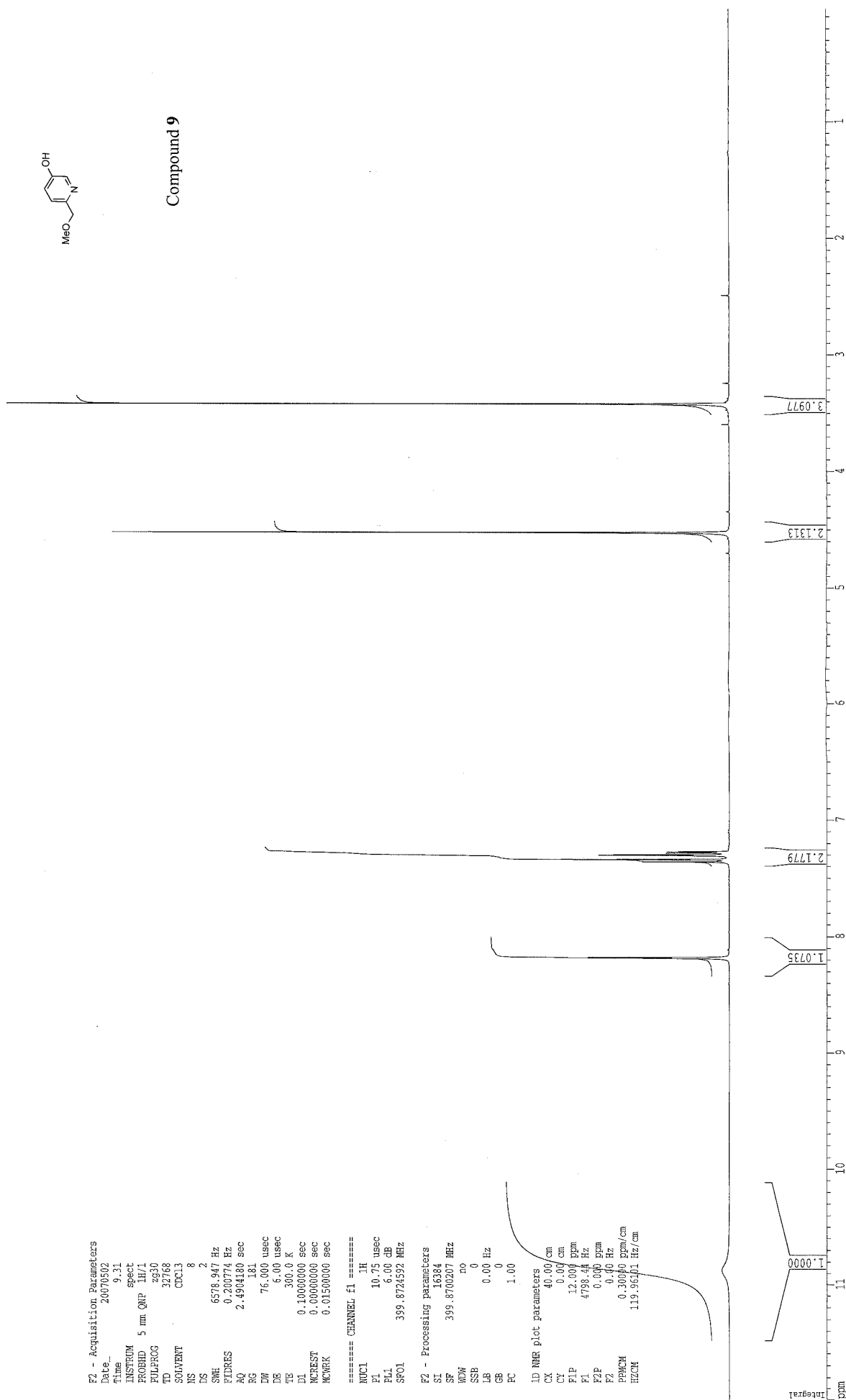
WDW no

SSB 0

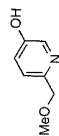
LB 0.00 Hz

GB 0

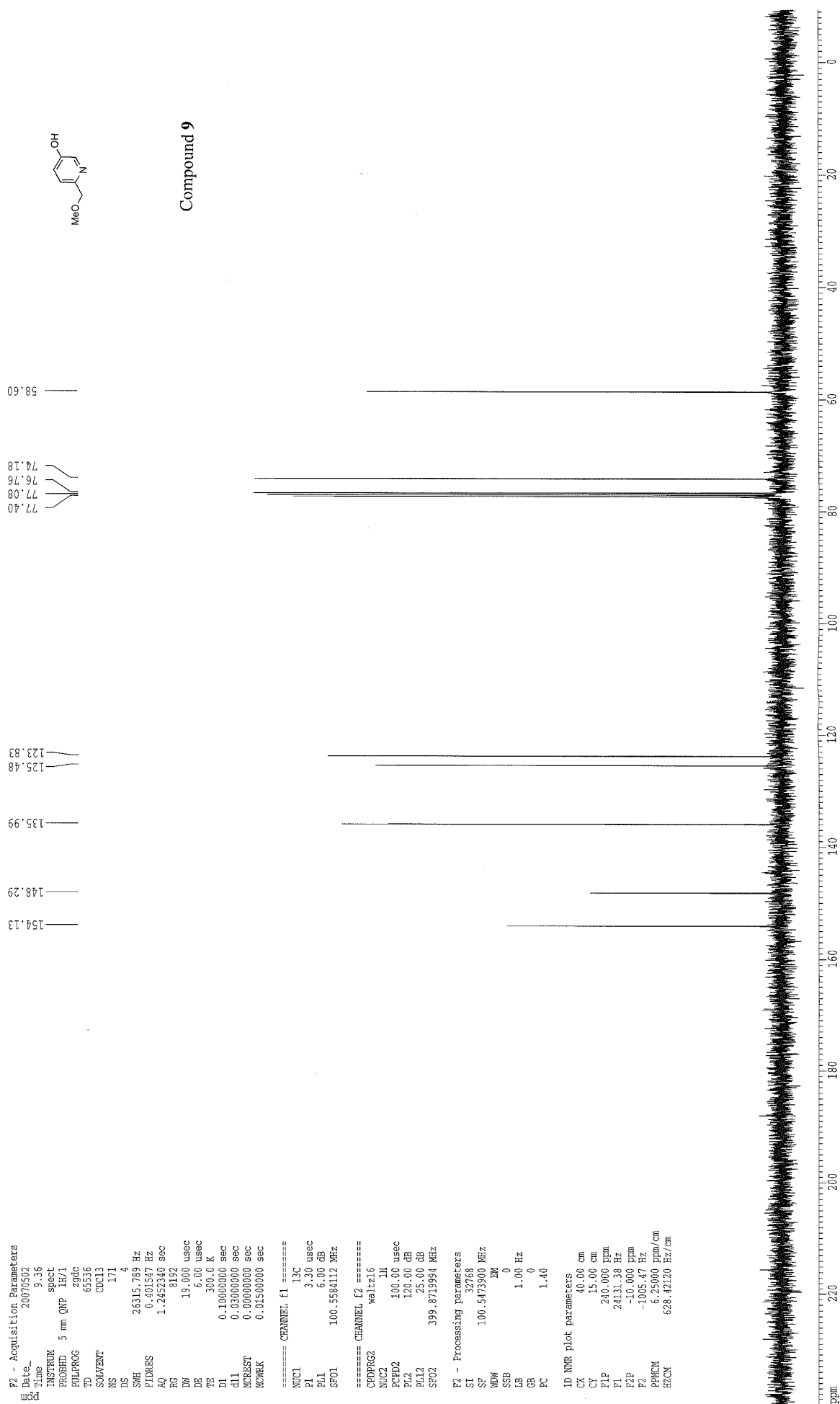
PC 1.00



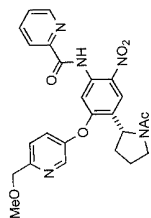
nmr400b c-13 (h-1 decoupled)



Compound 9



nmr400b h-1



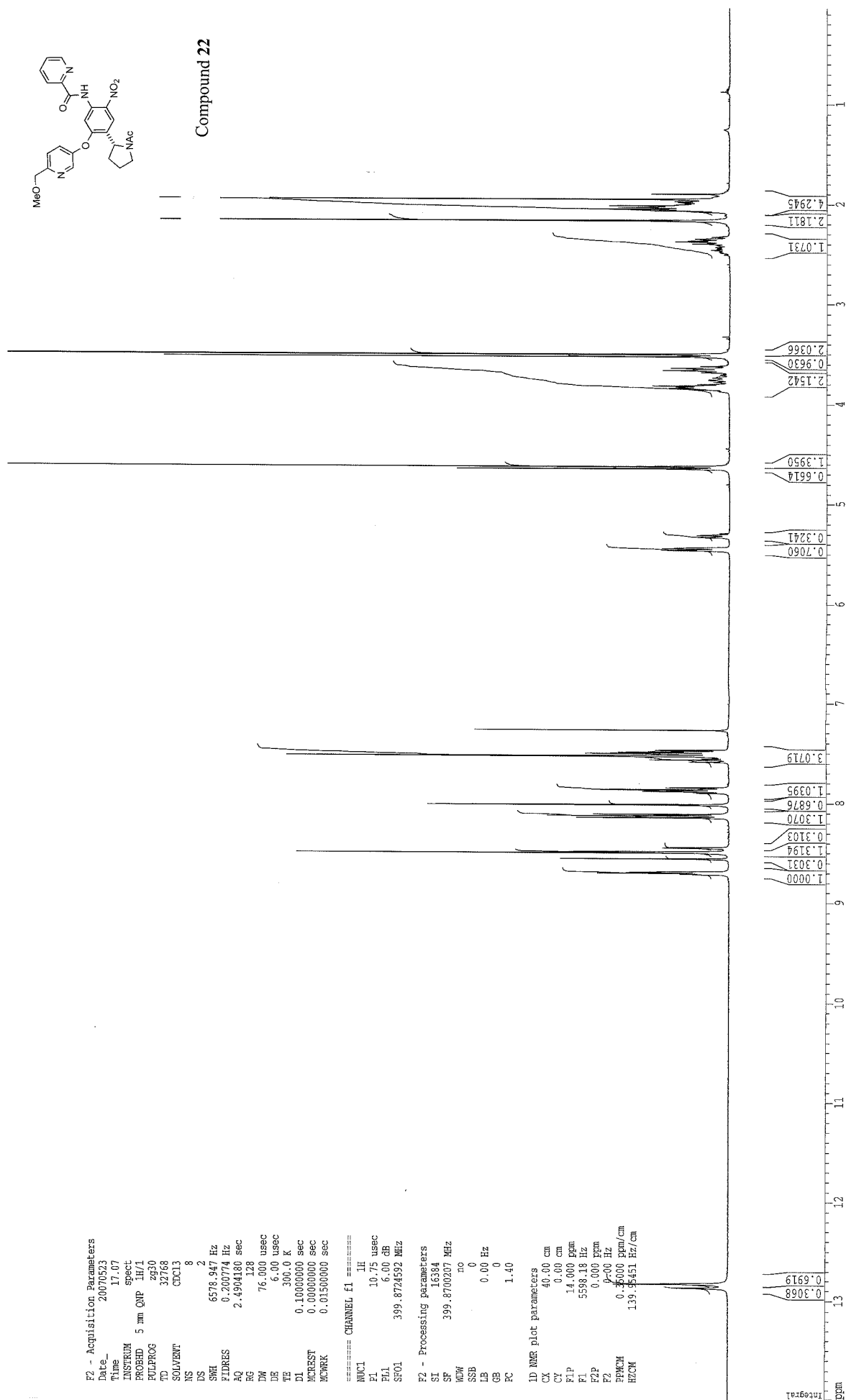
Compound 22

F2 - Acquisition Parameters
 Date_ 20070523
 Time 17.07
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6578.947 Hz
 FIDRES 0.200774 Hz
 AQ 2.4904180 sec
 RG 128
 IN 76.000 usec
 DE 6.00 usec
 TE 300.0 K
 DI 0.10000000 sec
 ACQRES 0.00000000 sec
 PCYCLE 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.75 usec
 PL1 6.00 dB
 SFO1 399.8724592 MHz

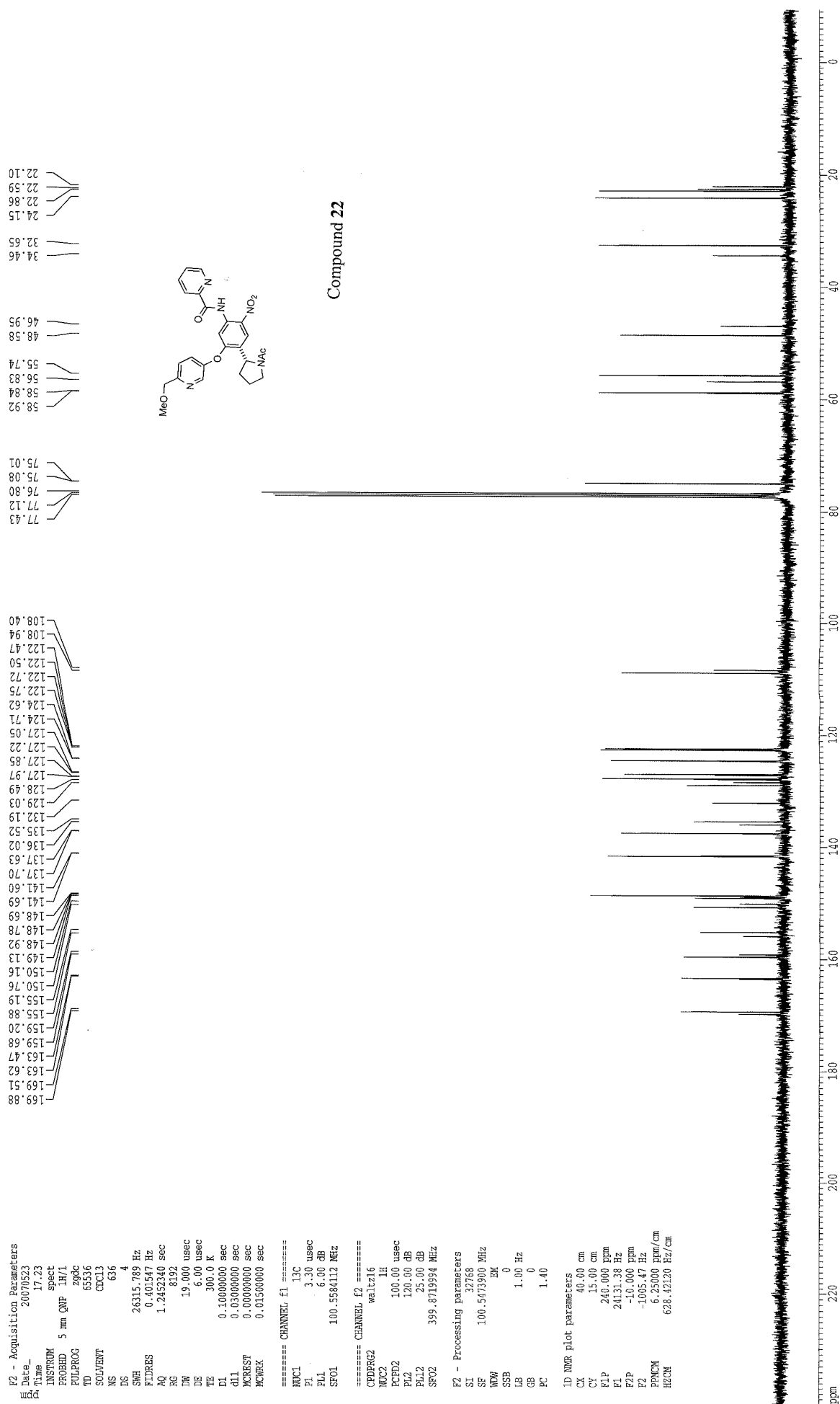
F2 - Processing parameters
 SI 16384
 SF 399.8700207 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 CY 0.00 cm
 F1P 14.900 ppm
 F1 5598.18 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 DPMCM 0.0000 ppm/cm
 HZCM 139.35451 Hz/cm



Integral

nmr400b c-13 (h-1 decoupled)



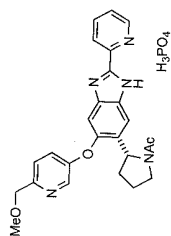
F2 - Acquisition Parameters

Date_ 20080321
 Time_ 16.52
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT DMSO
 NS 8
 DS 2
 SWH 6578.947 Hz
 FIDRES 0.200774 Hz
 AQ 2.4904180 sec
 RG 71.8
 DW 76.000 usec
 DE 7.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL 0.00 dB
 SF01 399.8724694 MHz

F2 - Processing parameters

SI 16384
 SF 399.8700000 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00



Compound 1

